

Handbook of Thermal Spray Technology

Edited by
J.R. Davis
Davis & Associates

Prepared under the direction of the
Thermal Spray Society Training Committee



Copyright © 2004
by
ASM International®
All rights reserved

No part of this book may be reproduced, stored in a retrieval system, or transmitted, in any form or by any means, electronic, mechanical, photocopying, recording, or otherwise, without the written permission of the copyright owner.

First printing, September 2004

Great care is taken in the compilation and production of this book, but it should be made clear that NO WARRANTIES, EXPRESS OR IMPLIED, INCLUDING, WITHOUT LIMITATION, WARRANTIES OF MERCHANTABILITY OR FITNESS FOR A PARTICULAR PURPOSE, ARE GIVEN IN CONNECTION WITH THIS PUBLICATION. Although this information is believed to be accurate by ASM, ASM cannot guarantee that favorable results will be obtained from the use of this publication alone. This publication is intended for use by persons having technical skill, at their sole discretion and risk. Since the conditions of product or material use are outside of ASM's control, ASM assumes no liability or obligation in connection with any use of this information. No claim of any kind, whether as to products or information in this publication, and whether or not based on negligence, shall be greater in amount than the purchase price of this product or publication in respect of which damages are claimed. THE REMEDY HEREBY PROVIDED SHALL BE THE EXCLUSIVE AND SOLE REMEDY OF BUYER, AND IN NO EVENT SHALL EITHER PARTY BE LIABLE FOR SPECIAL, INDIRECT OR CONSEQUENTIAL DAMAGES WHETHER OR NOT CAUSED BY OR RESULTING FROM THE NEGLIGENCE OF SUCH PARTY. As with any material, evaluation of the material under end-use conditions prior to specification is essential. Therefore, specific testing under actual conditions is recommended.

Nothing contained in this book shall be construed as a grant of any right of manufacture, sale, use, or reproduction, in connection with any method, process, apparatus, product, composition, or system, whether or not covered by letters patent, copyright, or trademark, and nothing contained in this book shall be construed as a defense against any alleged infringement of letters patent, copyright, or trademark, or as a defense against liability for such infringement.

Comments, criticisms, and suggestions are invited and should be forwarded to ASM International.

ASM International staff who worked on this project include Scott Henry, Senior Manager of Product and Service Development, Publications; Bonnie Sanders, Manager of Production; Carol Polakowski and Joanne Miller, Production Supervisors; and Jill Kinson and Pattie Pace, Production Coordinators.

Library of Congress Cataloging-in-Publication Data

Handbook of thermal technology / edited by J.R. Davis ; prepared under the direction of the Thermal Spray Society Training Committee.

p. cm.

Includes bibliographical references and index.

ISBN 0-87170-795-0

1. Metal spraying—Handbooks, manuals, etc. 2. Plasma spraying—Handbooks, manuals, etc. I. Davis, J. R. (Joseph R.) II. ASM International. Thermal Spray Society Training Committee.

TS655.H24 2004

671.7'34—dc22

2004046412

SAN: 204-7586

ASM International®
Materials Park, OH 44073-0002
www.asminternational.org

Printed in the United States of America

Multiple copy reprints of individual articles are available from Technical Department, ASM International.

The cover photograph shows the four electric arc gun arrangement used by Sprayform Holdings Limited (Staffordshire, UK) to make steel tooling. The arc guns are mounted and manipulated by a robot. The tooling is used for production of a large number of parts, by plastic injection molding or press forming, for example. Photograph provided courtesy of Patrick Grant, Department of Materials, Oxford University. The photograph originally appeared on the cover of the Journal of Thermal Spray Technology, Vol 9 (No. 2), June 2000.

Preface

Thermal spray technology encompasses a group of coating processes that provide functional surfaces to protect or improve the performance of a substrate or component. Many types and forms of materials can be thermal sprayed—which is why thermal spray is used worldwide to provide protection from corrosion, wear, and heat; to restore and repair components; and for a variety of other applications.

This handbook is intended to be an extensive reference guide to thermal spray technology. It covers principles, processes, types of coatings, applications, performance, and testing/analysis. It will serve as an excellent introduction and guidebook for those who are new to thermal spray. The handbook provides in-depth coverage and data that will be of great value to specifiers and users of thermal spray coatings, as well as to thermal spray experts who need additional background in certain areas. This handbook also was tailored with undergraduate and graduate students in mind so that they too can be exposed to the excitement of thermal spray technology.

The Thermal Spray Society (TSS) is pleased to serve as copublisher with ASM International for this important work. The genesis for this project was the plan to revise the ASM International homestudy course on thermal spray, first published in 1992. That endeavor was well underway in 2002 when the Reference Publications Department at ASM International developed a proposal for a handbook on thermal spray technology. At the October 2002 meeting of the Thermal Spray Society Board, the decision was made to merge the two projects. The lesson materials were revised extensively and then augmented with additional information to round out the coverage and create a comprehensive reference handbook.

This project truly has been a team effort. The main driver and guiding force has been the TSS Training Committee, which has overseen the volunteer contributions to the handbook and otherwise contributed mightily to make the handbook a reality. Special recognition is due to Richard Knight, former chair of the committee, for his detailed technical review of the revised lesson materials. Several other committee members have served as authors and reviewers for the handbook. For ASM International, Joanne Miller initially served as the staff project coordinator for the revision of the homestudy course. The project was transferred to Scott Henry when it was decided to transform the narrow scope of the content into a more all-encompassing and comprehensive reference handbook. We were fortunate in attracting Joseph R. Davis to act as Editor of the project in 2002. Joe has excelled in tying the materials together and adding additional key reference content to complete the coverage of the handbook, as well as dealing with many pragmatic issues such as meeting tight deadlines. Of course, the most important involvement in the project has been provided by the authors, reviewers, and other contributors who have created the content contained in these pages.

Christopher C. Berndt
Stony Brook University
President, Thermal Spray Society (2002–2004)

William J. Lenling
Thermal Spray Technologies Inc.
Chair, TSS Training Committee (2002–)

Dedication

This handbook is dedicated to the memory of Douglas H. Harris (1936–2001), who made enormous contributions to the practice and implementation of thermal spray over a period of more than 35 years. Doug wrote the section on thermal spray applications for the 1992 ASM homestudy course, *Thermal Spray Technology*. He contributed to the revision and updating of the applications section for this handbook before he passed away in 2001.

Thermal Spray Society Board (2003–2004)

Christopher C. Berndt
President
Stony Brook University

Richard Knight
Vice President
Drexel University

Peter Hanneforth
Secretary/Treasurer
Sulzer Metco (U.S.) Inc.

Albert Kay
Past President
ASB Industries Inc.

Mitchell R. Dorfman
Sulzer Metco (U.S.) Inc.

Pierre Fauchais
University of Limoges

Edmund F. Rybicki
University of Tulsa

Roland D. Seals
Oak Ridge National Laboratory

Raymond J. Sinatra
Rolls Royce Corporation

Mark F. Smith
Sandia National Laboratories

Joseph P. Stricker
St. Louis Metallizing Company

Robert H. Unger
Consultant

Thermal Spray Society Training Committee (2003–2004)

William J. Lenling
Chair
Thermal Spray Technologies Inc.

Richard Bajan
Goodrich Corporation

Graeme Herbert
G&M Thermal Coatings

J. Karthikeyan
ASB Industries Inc.

Albert Kay
ASB Industries Inc.

Richard Knight
Drexel University

Howard M. Kopech
Praxair TAFE Inc.

Elaine Motyka
Praxair Surface Technologies TAFE Inc.

Irene Nava
Solar Turbines Inc.

Joseph P. Stricker
St. Louis Metallizing Company

Authors, Reviewers, and Other Contributors

Christopher C. Berndt
Stony Brook University

Thomas Bernecki
BIRL, Northwestern University

Bruce Bodger
Hitemco

X. Chen
Solar Turbines Inc.

Kenneth Couch
Protech Lab Corporation

Daryl E. Crawmer
Thermal Spray Technologies

Deborah K. Curtis
ASB Industries Inc.

Mitchell R. Dorfman
Sulzer Metco (U.S.) Inc.

Kathy M. Dusa
International Thermal Spray Association

Leo Glass
APS-Materials Inc.

Glen Hannah
Stryker Orthopaedics

Doug H. Harris
APS-Materials Inc.

Graeme Herbert
G&M Thermal Coatings

Frank Hermanek
Praxair Surface Technologies/TAFA (retired)

Stephen M. Jaffe
Material Methods

J. Karthikeyan
ASB Industries Inc.

Albert Kay
ASB Industries Inc.

Charles M. Kay
ASB Industries Inc.

Richard Knight
Drexel University

Kristen Larson
Hitemco

D. Lee
Deloro Stellite Company Inc.

William J. Lenling
Thermal Spray Technologies Inc.

Frank N. Longo
Longo Associates

Robert C. McCune
Ford Motor Company

Z. Mutasim
Solar Turbines Inc.

Irene Nava
Solar Turbines Inc.

Larry Pollard
Progressive Technologies Inc.

Walter L. Riggs II
TubalCain Company Inc.

Bruce D. Sartwell
Naval Research Laboratory

Mark F. Smith
Sandia National Laboratories

Ronald W. Smith
Materials Resources Inc.

Joseph P. Stricker
St. Louis Metallizing Company

Robert A. Sulit
Sulit Engineering

P. Tommer
Sulzer Metco AG

Robert H. Unger
Praxair TAFA Inc.

Contents

Introduction to Thermal Spray Technology and Surface Science	1
Introduction to Thermal Spray Processing	3
Characteristics of Thermal Spray Coatings	3
Thermal Spray Processes and Techniques	5
Materials for Thermal Spray	7
Markets and Applications	8
Appendix: Historical Development of Thermal Spray	9
Processing and Equipment	9
Developments after World War II	10
Surface Science	14
What Is a Surface?	14
How Surfaces Interact with Their Environments	14
Understanding Thermal and Thermochemical Processes	18
Surface Hardening	18
Thermochemical Treatments	20
Electrochemical Treatments	22
Chemical Treatments	24
Hot Dip Coating	24
Chemical Vapor Deposition (CVD)	25
Physical Vapor Deposition (PVD)	26
Ion Implantation	29
Thermal Spray Processes	29
Hardfacing	30
Comparison of Major Coating Methods	32
Guide to General Information Sources	36
ASM International and the Thermal Spray Society	36
Specifications, Standards, and Quality Control for Coatings	36
Further Literature Sources for Thermal Spray	39
Coatings, Equipment, and Theory	41
Introduction to Coatings, Equipment, and Theory	43
Thermal Spray versus Other Coating Processes	44
Thermal Spray Process Classification	44
Processing Characteristics	45
Coating Structures, Properties, and Materials	47
Splat	47
Oxide Stringers	48
Porosity	49
Thermal Spray Coating Properties	51
Thermal Spray Materials	52
Thermal Spray Processes	54
Flame Spray	54
Detonation Gun	55
High-Velocity Oxyfuel Spray (HVOF)	56
Electric Arc Spray	60
Plasma Spray	62
Controlled-Environment Spraying	70
Other Significant Plasma Spray Processes	72
Emerging Developments in Thermal Spray Devices	73
Cold Spray Process	77
Cold Spray Equipment and Process Parameters	78
Deposition Efficiency and Coating Properties	78
Coating Materials and Applications	79
Conclusions	82
Process Control Equipment	85
Process Parameters	85
Material Feed	86
Powder Characteristics	88
Powder Pulsing	90
Gas Flow Measurement and Control	91
Related Measurement Issues	93
Gas Quality Issues	93
Thermal Spray Controls	94
Plasma Arc Starting Problems	95
Ancillary Equipment	96
Health and Safety Equipment	99
Gas Handling	99
Ventilation and Heat Exhaust	99
Particulates	100
Noise	100
Radiation	101
Other Potential Safety Concerns	101
Processing and Design	103
Introduction to Processing and Design	105
Advantages and Disadvantages of Thermal Spraying	105
Coating Processing	108
Substrate Metallurgy and Thermal History	108
Part Configuration and Coating Location	108
Surface Preparation	110
Masking	117
Preheating	117
Coating Operations	120
Spray Pattern	120
Coating Buildup	121
Process Variations	123
Substrate/Coating Temperature Control	124
Torch and Substrate Motion	124
Deposition Efficiency	126
Statistical Design Techniques	126
Information Management	127
Postcoating Operations	128
Finishing	128
Densification	129
Inspection	130
Materials Production for Thermal Spray Processes	133
Introduction to Materials Production for Thermal Spray Processes	135
Terminology	135
Summary	136
Feedstock Material Considerations	137
The Powder Feeding Problem	137
Powder Feeding Systems	138
Feedstock/TC Heat Source Interactions	140
Process Parameter Problems and Health Concerns	141
Material Categories for Thermal Sprayed Coatings	142
Classification According to Thermal Spray Process	142
Classification According to Material Morphology	143

Classification According to Chemical Nature of the Material	145	Testing and Characterization	215
Classification According to Applications	146	Introduction to Testing and Characterization	217
Material Production Processes	147	Metallurgical and Materials Nature of Thermal Spray Coatings Compared to Wrought, Forged, and Cast Materials	217
Machining Techniques	147	Testing and Characterization Methods as Response Generators	221
Crushing and Milling	147	Testing and Characterization as a Diverse Materials Engineering Opportunity	222
Atomization	150	Metallography and Image Analysis	224
Chemical Techniques	153	Sectioning Techniques	226
Particle Characterization	159	Mounting Materials and Techniques	227
Particle Morphology and Porosity	159	Grinding	231
Particle Classification Methods	161	Polishing	239
Quality Control and Feedstock Material Specification	164	Recommended Procedures	246
Applications for Thermal Spray Processing	169	Image Analysis	252
Introduction to Applications for Thermal Spray Processing	171	Etchants	259
Coatings for Wear Resistance	171	Testing of Coatings	260
Coatings for Corrosion Resistance	173	Hardness Testing	260
Selected Applications	174	Tensile Adhesion Testing	261
Thermal Spray Processes Described in the Article	174	Corrosion Testing and Monitoring	266
Aerospace Applications	175	Testing of Thermal Barrier Coatings	268
Thermal Spray Coatings for Land-Based Turbine Applications	181	Residual-Stress Determination in Thermal Spray Coatings ...	270
Automotive Applications	184	Research in Nondestructive Testing	271
Biomedical Coatings for Orthopedic and Dental Implants	189	Powder Testing and Characterization	273
Glass Contact Coatings	191	Particle Size	273
Corrosion Applications for Marine and Land-Based Infrastructure	192	Powder and Coating Stoichiometry	275
Processing Electronic Materials and Devices	198	Particle Chemistry	275
Printing Industry Applications	200	Phase Analysis by X-ray Diffraction	276
Processing Industries and Corrosion Control	201	Reference Information	279
Metals Processing Industries	206	Glossary of Terms	281
Electrical Utilities	207	Abbreviations and Symbols	324
The Textile and Plastics Industries	208	Metric Conversion Guide	329
Recent Developments in Thermal Spray Application Technology	208	Index	332



ASM International is the society for materials engineers and scientists, a worldwide network dedicated to advancing industry, technology, and applications of metals and materials.

ASM International, Materials Park, Ohio, USA
www.asminternational.org

This publication is copyright © ASM International®. All rights reserved.

Publication title	Product code
Handbook of Thermal Spray Technology	#06994G

To order products from ASM International:

Online Visit www.asminternational.org/bookstore

Telephone 1-800-336-5152 (US) or 1-440-338-5151 (Outside US)

Fax 1-440-338-4634

Mail Customer Service, ASM International
9639 Kinsman Rd, Materials Park, Ohio 44073-0002, USA

Email CustomerService@asminternational.org

In Europe American Technical Publishers Ltd.
27-29 Knowl Piece, Wilbury Way, Hitchin Hertfordshire SG4 0SX,
United Kingdom
Telephone: 01462 437933 (account holders), 01462 431525 (credit card)
www.ameritech.co.uk

In Japan Neutrino Inc.
Takahashi Bldg., 44-3 Fuda 1-chome, Chofu-Shi, Tokyo 182 Japan
Telephone: 81 (0) 424 84 5550

Terms of Use. This publication is being made available in PDF format as a benefit to members and customers of ASM International. You may download and print a copy of this publication for your personal use only. Other use and distribution is prohibited without the express written permission of ASM International.

No warranties, express or implied, including, without limitation, warranties of merchantability or fitness for a particular purpose, are given in connection with this publication. Although this information is believed to be accurate by ASM, ASM cannot guarantee that favorable results will be obtained from the use of this publication alone. This publication is intended for use by persons having technical skill, at their sole discretion and risk. Since the conditions of product or material use are outside of ASM's control, ASM assumes no liability or obligation in connection with any use of this information. As with any material, evaluation of the material under end-use conditions prior to specification is essential. Therefore, specific testing under actual conditions is recommended.

Nothing contained in this publication shall be construed as a grant of any right of manufacture, sale, use, or reproduction, in connection with any method, process, apparatus, product, composition, or system, whether or not covered by letters patent, copyright, or trademark, and nothing contained in this publication shall be construed as a defense against any alleged infringement of letters patent, copyright, or trademark, or as a defense against liability for such infringement.

Introduction to Thermal Spray Processing

THERMAL SPRAY is a generic term for a group of coating processes used to apply metallic or nonmetallic coatings. These processes are grouped into three major categories: flame spray, electric arc spray, and plasma arc spray. These energy sources are used to heat the coating material (in powder, wire, or rod form) to a molten or semimolten state. The resultant heated particles are accelerated and propelled toward a prepared surface by either process gases or atomization jets. Upon impact, a bond forms with the surface, with subsequent particles causing thickness buildup and forming a lamellar structure (Fig. 1). The thin “splats” undergo very high cooling rates, typically in excess of 10^6 K/s for metals (Ref 1).

A major advantage of thermal spray processes is the extremely wide variety of materials that can be used to produce coatings (Ref 2). Virtually any material that melts without decomposing can be used. A second major advantage is the ability of most thermal spray processes to apply coatings to substrates without significant heat input. Thus, materials with very high melting points, such as tungsten, can be applied to finely machined, fully heat-treated parts without changing the properties of the part and without excessive thermal distortion of the part. A third advantage is the ability, in most cases, to strip off and recoat worn or damaged coatings without changing part properties or dimensions. A disadvantage is the line-of-sight nature of these deposition processes. They can only coat what the torch or gun can “see.” Of course, there are also size limitations. It is impossible to coat small, deep cavities into which a torch or gun will not fit. The article “Introduction to Processing and Design” in this Handbook provides a more complete discussion of the advantages and disadvantages of thermal spray processes.

Characteristics of Thermal Spray Coatings (Ref 1)

Microstructural Characteristics. The term “thermal spray” describes a family of processes that use the thermal energy generated by chemical (combustion) or electrical (plasma or arc) methods to melt, or soften, and accelerate fine dispersions of particles or droplets to speeds in the range of 50 to >1000 m/s (165 to >3300 ft/s). The high particle temperatures and speeds achieved result in significant droplet deformation on impact at a surface, producing thin layers or lamellae, often called “splats,” that conform and adhere to the substrate surface. Solidified droplets build up rapidly, particle by particle, as a continuous stream of droplets impact to form continuous rapidly solidified layers. Individual splats are generally thin (~ 1 to $20\text{ }\mu\text{m}$), and each droplet cools at very high rates ($>10^6$ K/s for metals) to form uniform, very fine-grained, polycrystalline coatings or deposits. Figure 2 shows a schematic

of a generic thermal spray powder consolidation process, illustrating the key features and a typical deposit microstructure.

Sprayed deposits usually contain some level of porosity, typically between 0 and $\sim 10\%$, some unmelted or partially melted particles, fully melted and deformed “splats,” metastable phases, and oxidation from entrained air. Thermal spray process jets or plumes

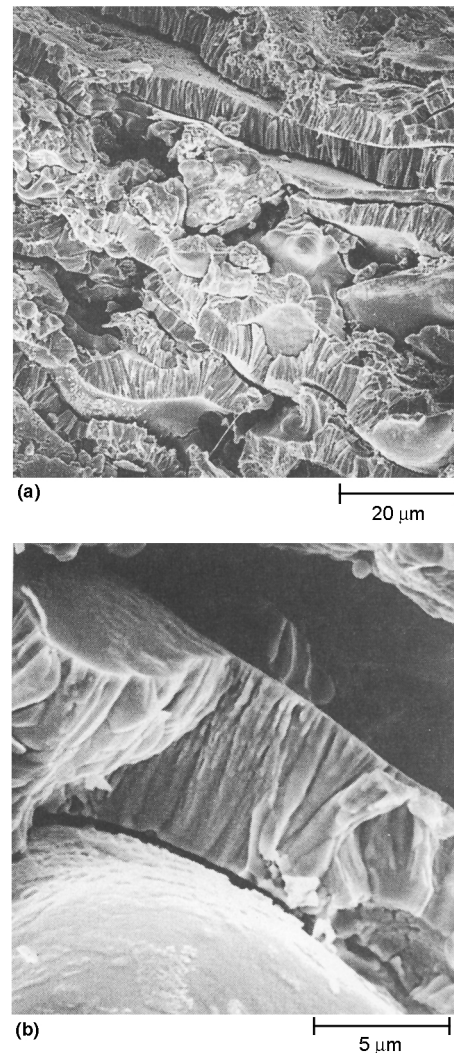


Fig. 1 Scanning electron micrographs of fracture cross sections of an air plasma-sprayed tungsten coating. (a) Lamellar microstructure. (b) Presence of a columnar grain structure within the splats. Source: S.J. Bull, AEA Technology

are characterized by large gradients of both temperature and velocity. Feedstock is usually in powdered form with a distribution of particle sizes. When these powdered materials are fed into the plume, portions of the powder distribution take preferred paths according to their inertia. As a result, some particles may be completely unmelted and can create porosity or become trapped as

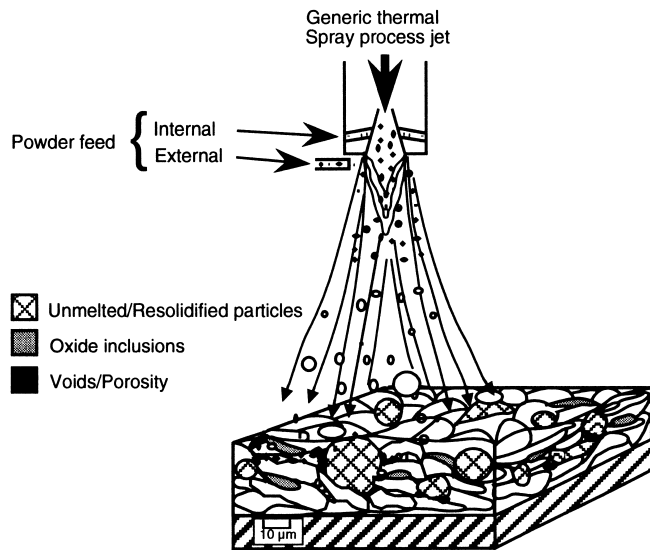


Fig. 2 Schematic of a typical thermal spray powder process. Source: Ref 1

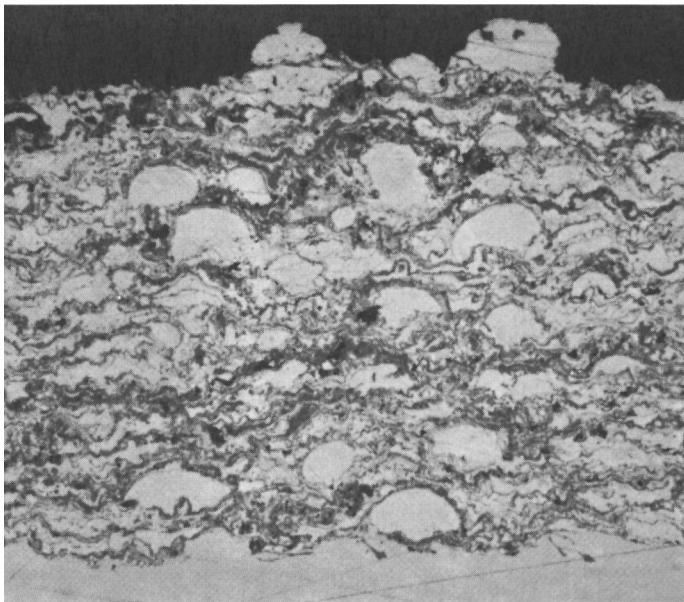


Fig. 3 Photomicrograph showing the microstructure of an HVOF-sprayed 80Ni-20Cr alloy. Source: Ref 1

“unmelts” in the coating. Use of wire and rod feedstock materials produces particle size distributions because of nonuniform heating and unpredictable drag forces, which shear molten material from the parent wire or rod. The level of these coating defects varies depending on the particular thermal spray process used, the operating conditions selected, and the material being sprayed, as described later.

Figure 3 is a photomicrograph of a thermal-sprayed 80Ni-20Cr alloy coating applied via the high-velocity oxyfuel (HVOF) process showing the characteristic lamellar splat structure. The microstructure shown in Fig. 3 includes partially melted particles and dark oxide inclusions that are characteristic of many metallic coatings sprayed in air. Such coatings exhibit characteristic lamellar microstructures, with the long axis of the impacted splats oriented parallel to the substrate surface, together with a distribution of similarly oriented oxides. Coating oxide content varies with the process—wire arc, plasma, or HVOF. The progressive increases in particle speed of these processes leads to differing levels of oxide and differing degrees of oxide breakup on impact at the surface. Oxides may increase coating hardness and wear resistance and may provide lubricity. Conversely, excessive and continuous oxide networks can lead to cohesive failure of a coating and contribute to excessive wear debris. Oxides can also reduce corrosion resistance. It is important to select materials, coating processes, and processing parameters that allow control of oxide content and structure to acceptable levels for a given application.

Thermal spray coatings may contain varying levels of porosity, depending on the spray process, particle speed and size distribution, and spray distance. Porosity may be beneficial in tribological applications through retention of lubricating oil films. Porosity also is beneficial in coatings on biomedical implants. Lamellar oxide layers can also lead to lower wear and friction due to the lubricity of some oxides. The porosity of thermal spray coatings is typically <5% by volume. The retention of some unmelted and/or resolidified particles can lead to lower deposit cohesive strengths, especially in the case of “as-sprayed” materials with no postdeposition heat treatment or fusion.

Other key features of thermal spray deposits are their generally very fine grain structures and columnar orientation (Fig. 1b). Thermal-sprayed metals, for example, have reported grain sizes of <1 μm prior to postdeposition heat treatment. Grain structure across an individual splat normally ranges from 10 to 50 μm, with typical grain diameters of 0.25 to 0.5 μm, owing to the high cooling rates achieved (~10⁶ K/s).

The tensile strengths of as-sprayed deposits can range from 10 to 60% of those of cast or wrought materials, depending on the spray process used. Spray conditions leading to higher oxide levels and lower deposit densities result in the lowest strengths. Controlled-atmosphere spraying leads to ~60% strength, but requires postdeposition heat treatment to achieve near 100% values. Low as-sprayed strengths are related somewhat to limited intersplat diffusion and limited grain recrystallization during the rapid solidification characteristic of thermal spray processes. The primary factor limiting adhesion and cohesion is residual stress resulting from rapid solidification of the splats. Accumulated residual stress also limits thickness buildup.

Thermal Spray Processes and Techniques

Members of the thermal spray family of processes are typically grouped into three major categories: flame spray, electric arc spray, and plasma arc spray, with a number of subsets falling under each category. (Cold spray is a recent addition to the family of thermal spray processes. This process typically uses some modest preheating, but is largely a kinetic energy process. The unique characteristics of cold spray are discussed in the article “Cold Spray Process” in this Handbook.) A brief review of some of the more commercially important thermal spray processes is given below. Table 1 compares important process characteristics associated with these techniques. Selection of the appropriate thermal spray method is typically determined by:

- Desired coating material
- Coating performance requirements
- Economics
- Part size and portability

More detailed information on thermal spray processes can be found in the article “Introduction to Coatings, Equipment, and Theory” (see, in particular, Fig. 2 in the aforementioned article, which illustrates the three major coating categories and their subsets) and in the article “Thermal Spray Processes” in this Handbook.

Flame Spray Processes (Ref 3)

Flame spraying includes low-velocity powder flame, rod flame, and wire flame processes and high-velocity processes such as HVOF and the detonation gun (D-Gun) process (D-Gun is a registered trademark of Praxair Surface Technologies Inc.).

Flame Powder. In the flame powder process, powdered feedstock is aspirated into the oxyfuel flame, melted, and carried by the flame and air jets to the workpiece. Particle speed is relatively low (<100 m/s), and bond strength of the deposits is generally lower than the higher velocity processes. Porosity can be high and cohesive strength is also generally lower. Spray rates are usually in the 0.5 to 9 kg/h (1 to 20 lb/h) range for all but the lower melting point materials, which spray at significantly higher rates. Substrate surface temperatures can run quite high because of flame impingement.

Wire Flame. In wire flame spraying, the primary function of the flame is to melt the feedstock material. A stream of air then atomizes the molten material and propels it toward the workpiece. Spray rates for materials such as stainless steel are in the range of 0.5 to 9 kg/h (1 to 20 lb/h). Again, lower melting point materials such as zinc and tin alloys spray at much higher rates. Substrate temperatures often range from 95 to 205 °C (200 to 400 °F) because of the excess energy input required for flame melting. In most thermal spray processes, less than 10% of the input energy is actually used to melt the feedstock material.

High-Velocity Oxyfuel. In HVOF, a fuel gas (such as hydrogen, propane, or propylene) and oxygen are used to create a combustion jet at temperatures of 2500 to 3100 °C (4500 to 5600 °F). The combustion takes place internally at very high chamber pressures, exiting through a small-diameter (typically 8 to 9 mm, or

0.31 to 0.35 in.) barrel to generate a supersonic gas jet with very high particle speeds. The process results in extremely dense, well-bonded coatings, making it attractive for many applications. Either powder or wire feedstock can be sprayed, at typical rates of 2.3 to 14 kg/h (5 to 30 lb/h).

Detonation Gun. In the detonation gun process, pre-encapsulated “shots” of feedstock powder are fed into a 1 m (3 ft) long barrel along with oxygen and a fuel gas, typically acetylene. A spark ignites the mixture and produces a controlled explosion that propagates down the length of the barrel. The high temperatures and pressures (1 MPa, or 150 psi) that are generated blast the particles out of the end of the barrel toward the substrate. Very high bond strengths and densities as well as low oxide contents can be achieved using this process.

Electric Arc Processes (Ref 3)

Electric Arc. In the electric arc spray process (also known as the wire arc process), two consumable wire electrodes connected to a high-current direct-current (dc) power source are fed into the gun and meet, establishing an arc between them that melts the tips of the wires. The molten metal is then atomized and propelled toward the substrate by a stream of air. The process is energy efficient because all of the input energy is used to melt the metal. Spray rates are driven primarily by operating current and vary as a function of both melting point and conductivity. Generally materials such as copper-base and iron-base alloys spray at 4.5 kg (10 lb)/100 A/h. Zinc sprays at 11 kg (25 lb)/100 A/h. Substrate temperatures can be very low, because no hot jet of gas is directed toward the substrate. Electric arc spraying also can be carried out using inert gases or in a controlled-atmosphere chamber (Ref 1).

Plasma Arc Processes (Ref 3)

Conventional Plasma. The conventional plasma spray process is commonly referred to as air or atmospheric plasma spray (APS). Plasma temperatures in the powder heating region range from about 6000 to 15,000 °C (11,000 to 27,000 °F), significantly above the melting point of any known material. To generate the plasma, an inert gas—typically argon or an argon-hydrogen mixture—is superheated by a dc arc. Powder feedstock is introduced via an inert carrier gas and is accelerated toward the workpiece by the plasma jet. Provisions for cooling or regulating the spray rate may be required to maintain substrate temperatures in the 95 to 205 °C (200 to 400 °F) range. Commercial plasma spray guns operate in the range of 20 to 200 kW. Accordingly, spray rates greatly depend on gun design, plasma gases, powder injection schemes, and materials properties, particularly particle characteristics such as size, distribution, melting point, morphology, and apparent density.

Vacuum Plasma. Vacuum plasma spraying (VPS), also commonly referred to as low-pressure plasma spraying (LPPS, a registered trademark of Sulzer Metco), uses modified plasma spray torches in a chamber at pressures in the range of 10 to 50 kPa (0.1 to 0.5 atm). At low pressures the plasma becomes larger in diameter and length, and, through the use of convergent/divergent nozzles, has a higher gas speed. The absence of oxygen and the abil-

Table 1 Comparison of thermal spray processes

Process	Gas flow		Flame or exit plasma temperature		Particle impact velocity		Relative adhesive strength(a)	Cohesive strength	Oxide content, %	Relative process cost(a)	Maximum spray rate		Power		Energy required to melt	
	m ³ /h	ft ³ /h	°C	°F	m/s	ft/s					kg/h	lb/h	kW	hp	kW/kg	kW/lb
Flame powder	11	400	2200	4000	30	100	3	Low	6	3	7	15	25–75	34–100	11–22	5–10
Flame wire	71	2500	2800	5000	180	600	4	Medium	4	3	9	20	50–100	70–135	11–22	5–10
High-velocity oxyfuel	28–57	1000–2000	3100	5600	610–1060	2000–3500	8	Very high	0.2	5	14	30	100–270	135–360	22–200	10–90
Detonation gun	11	400	3900	7000	910	3000	8	Very high	0.1	10	1	2	100–270	135–360	220	100
Wire arc	71	2500	5500	10,000	240	800	6	High	0.5–3	1	16	35	4–6	5–8	0.2–0.4	0.1–0.2
Conventional plasma	4.2	150	5500	10,000	240	800	6	High	0.5–1	5	5	10	30–80	40–110	13–22	6–10
High-energy plasma	17–28	600–1000	8300	15,000	240–1220	800–4000	8	Very high	0.1	4	23	50	100–250	135–335	9–13	4–6
Vacuum plasma	8.4	300	8300	15,000	240–610	800–2000	9	Very high	(b)	10	10	24	50–100	70–135	11–22	5–10

(a) 1 (low) to 10 (high). (b) ppm levels. Source: Ref 3

ity to operate with higher substrate temperatures produce denser, more adherent coatings with much lower oxide contents.

Kinetic Energy Processes

Kinetics has been an important factor in thermal spray processing from the beginning. With the introduction of detonation gun, HVOF, and high-energy plasma spraying, the kinetic-energy component of thermal spraying became even more important. The latest advance in kinetic spraying is known as “cold spray.”

Cold spray is a material deposition process in which coatings are applied by accelerating powdered feedstocks of ductile metals to speeds of 300 to 1200 m/s (985 to 3940 ft/s) using gas-dynamic techniques with nitrogen or helium as the process gas. The process is commonly referred to as “cold gas-dynamic spraying” because of the relatively low temperatures (0 to 800 °C, or 32 to 1470 °F) of the expanded gas and particle stream that emanates from the nozzle. Powder feed rates of up to 14 kg/h (30 lb/h) are possible. More details are provided in the article “Cold Spray Process” in this Handbook.

Materials for Thermal Spray (Ref 1)

Three basic types of deposits can be thermal sprayed:

- Single-phase materials, such as metals, alloys, intermetallics, ceramics, and polymers
- Composite materials, such as cermets (WC/Co, $\text{Cr}_3\text{C}_2/\text{NiCr}$, $\text{NiCrAlY}/\text{Al}_2\text{O}_3$, etc.), reinforced metals, and reinforced polymers
- Layered or graded materials, referred to as functionally gradient materials (FGMs)

Examples of these, along with their particular advantages and applications, are described below.

Single-Phase Materials

Metals. Most pure metals and metal alloys have been thermal sprayed, including tungsten, molybdenum, rhenium, niobium, superalloys, zinc, aluminum, bronze, mild and stainless steels, NiCr alloys, cobalt-base Stellites, cobalt/nickel-base Triballoys, and NiCrBSi “self-fluxing” alloys. Sprayed alloys have advantages due to their similarity to many base metals requiring repair, their high strength, and their corrosion, wear, and/or oxidation resistance. Applications include automotive/diesel engine cylinder coatings; piston rings or valve stems; turbine engine blades, vanes, and combustors; protection of bridges and other corrosion-prone infrastructure; petrochemical pumps and valves; and mining and agricultural equipment.

Ceramics. Most forms of ceramics can be thermal sprayed, including metallic oxides such as Al_2O_3 , stabilized ZrO_2 , TiO_2 , Cr_2O_3 , and MgO; carbides such as Cr_3C_2 , TiC, Mo_2C , and SiC (generally in a more ductile supporting metal matrix such as cobalt or NiCr); nitrides such as TiN and Si_3N_4 ; and spinels or per-

ovskites such as mullite and 1-2-3-type superconducting oxides. Sprayed deposits of these materials are used to provide wear resistance (Al_2O_3 , Cr_2O_3 , TiO_2 , Cr_3C_2 , TiC, Mo_2C , and TiN), thermal protection (Al_2O_3 , ZrO_2 , and MgO), electrical insulation (Al_2O_3 , TiO_2 , and MgO), and corrosion resistance. Ceramics are particularly suited to thermal spraying, with plasma spraying being the most suitable process due to its high jet temperatures.

Intermetallics such as TiAl, Ti_3Al , Ni_3Al , NiAl, and MoSi_2 have all been thermal sprayed. Most intermetallics are very reactive at high temperatures and very sensitive to oxidation; hence, inert atmospheres must be used during plasma spraying. Research has also been conducted on thermal spray forming/consolidation of bulk intermetallic deposits (Ref 1).

Polymers also can be thermal sprayed successfully, provided they are available in particulate form. Thermal spraying of polymers has been practiced commercially since the 1980s, and a growing number of thermoplastic and thermosetting polymers and copolymers have now been sprayed, including urethanes, ethylene vinyl alcohols (EVAs), nylon 11, polytetrafluoroethylene (PTFE), ethylene tetrafluoroethylene (ETFE), polyetheretherketone (PEEK), polymethylmethacrylate (PMMA), polyimide, polycarbonate, and copolymers such as polyimide/polyamide, Surllyn (DuPont), and polyvinylidene fluoride (PVDF). Conventional flame spray and HVOF are the most widely used thermal spray methods for applying polymers.

Composite and Cermet Materials

Particulate-, fiber-, and whisker-reinforced composites have all been produced and used in various applications. Particulate-reinforced wear-resistant cermet coatings such as WC/Co, $\text{Cr}_3\text{C}_2/\text{NiCr}$, and TiC/NiCr are the most common applications and constitute one of the largest single thermal spray application areas; cermet coatings are discussed extensively throughout this Handbook. Thermal spray composite materials can have reinforcing-phase contents ranging from 10 to 90% by volume, where the ductile metal matrix acts as a binder, supporting the brittle reinforcing phase.

Functionally Gradient Materials

Developed in the early 1970s, FGMs are a growing application area with significant promise for the future production of (a) improved materials and devices for use in applications subject to large thermal gradients, (b) lower-cost clad materials for combinations of corrosion and strength or wear resistance, and (c) improved electronic material structures for batteries, fuel cells, and thermoelectric energy conversion devices. The most immediate application for FGMs is thermal barrier coatings (TBCs), where large thermal stresses are minimized. Component lifetimes are improved by tailoring the coefficients of thermal expansion, thermal conductivity, and oxidation resistance. These FGMs are finding use in turbine components, rocket nozzles, chemical reactor tubes, incinerator burner nozzles, and other critical furnace components. Figure 4 illustrates an example of a thermal-sprayed FGM proposed for the protection of copper using a layered FGM ceramic structure. Successful fabrication of this structure would

have application for improved burner nozzles, molds, and furnace walls.

Other FGMs are being developed for:

- Thermal protection of lightweight polymeric insulating materials in aircraft components
- Production of graded metallic/oxide/intermetallic advanced batteries and solid oxide fuel cells
- Production of oxide/metal/air-type electrode/electrolyte systems
- Forming of composite gun barrels
- Biomedical implant devices for enhanced bone-tissue attachment
- Ceramic outer air seals in aircraft gas turbines and other “clearance-control” coatings in rotating machinery
- Thick, multilayer TBCs for heavy-duty diesel engine pistons
- High-performance dielectric coatings for electronic devices
- Wear-resistant coatings for diesel engine piston rings
- Oxidation-resistant coatings for high-temperature conditions

Future growth of FGM applications will ensure that thermal spray processes, particularly HVOF and plasma spray processes, will increase and develop, provided that material properties can be controlled and processing costs optimized.

Markets and Applications

As shown in Fig. 5, the thermal spray industry underwent unprecedented growth during the period between 1960 and the late 1990s. Major contributors to this growth include the commercial

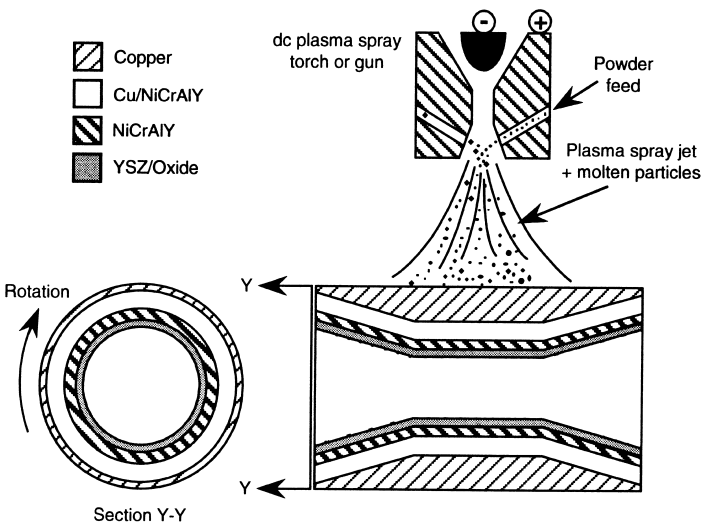


Fig. 4 Schematic of a thermal-sprayed FGM for a burner nozzle application in a pyrometallurgical operation. Source: Ref 1

introduction of plasma, electric arc, and HVOF, improved process control equipment, and the introduction of new materials and original equipment manufacturer (OEM) applications. Figure 5 also shows the influence that aircraft engine applications have had on the growth of the industry. Thermal spray coatings have been used for advanced gas turbine components such as compressor blades, compressor stator vanes, bearing housings, and labyrinth seals since the early 1960s. Common coating materials include cobalt-base (Co-Mo-Cr-Si) Laves-phase alloys such as Tribaloy T-400 and T-800, WC/Co materials, Cr₃C₂/20-30NiCr cermets, and MCrAlY coatings. Figure 6 shows current and potential gas turbine component applications for thermal spray coatings.

The global thermal spray market consists of:

Market segment	Market value (in U.S. dollars)
OEM/end users	1,400,000,000
Large coating service companies	800,000,000
Small coating service companies	600,000,000
Powder/equipment sales	700,000,000
Estimated total market	3,500,000,000

Source: Ref 5

According to Business Communications Company, Inc. (Norwalk, CT), the average annual growth rate of the thermal spray market in North America was +6.1% from 1997 through 2002.

As shown in Tables 2 and 3, a wide variety of industrial sectors rely on thermal spray processes and coatings. As summarized in

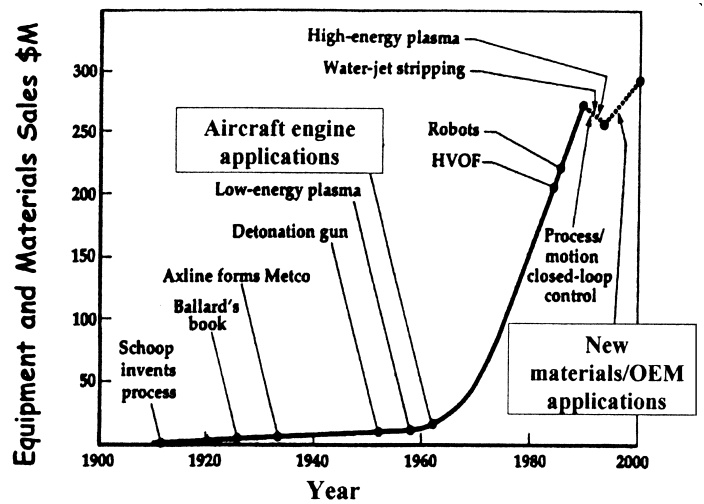


Fig. 5 Timeline of significant developments during the growth of the thermal spray industry. Source: C.C. Berndt, State University of New York at Stony Brook

Table 4, these industries use thermal spray coatings because they offer improved:

- Wear resistance
- Heat resistance (thermal barrier coatings)
- Clearance and dimensional control
- Corrosion and oxidation resistance
- Electrical properties (resistance and conductivity)

Additional information on thermal spray application areas can be found in the articles “Material Categories for Thermal Spray Coatings” and “Selected Applications” in this Handbook.

Future Areas of Growth. As discussed earlier, FGMs offer the thermal spray industry a number of opportunities for growth. Other important areas that will determine future growth include (Ref 1 and 4):

- Continued advances in process control equipment (robotics, motion control, gas pressure technology, real-time sensors, etc.)
- Improved methods for nondestructive testing and evaluation of coatings
- Improved understanding and optimization of the cold spray process
- Thermal spray forming of near-net-shape parts
- Spray forming of high critical temperature (T_c) superconducting oxide ceramics
- Computer-aided design (CAD)/rapid prototyping techniques such as stereolithography

- Diamond synthesis and deposition
- Thin-film deposition via LPPS
- Improved feedstock production techniques and quality control
- New materials (e.g., composites, nanophase materials, perovskites, and zirconates)

Appendix: Historical Development of Thermal Spray Processing and Equipment

Ronald W. Smith, Materials Resources, Inc.

THE EARLIEST RECORDS OF THERMAL SPRAY originate in the patents of M.U. Schoop (Zurich, Switzerland), dating from 1882 to 1889. These patents describe a process that fed lead and tin wires into a modified oxyacetylene welding torch. Later torches were modified to accept powdered materials. The powders were caught up in the hot expanding jet flow where the particles were heated while being accelerated toward the surface to impact, spread (if molten), and solidify. The results were coatings that were incrementally formed from impacting droplets.

Electric arc spray was also patented by Schoop around 1908, which enabled more metals to be sprayed. Steel, stainless steel, and zinc by wire-arc metallizing advanced through improvements in equipment and process control as well as through promotion of

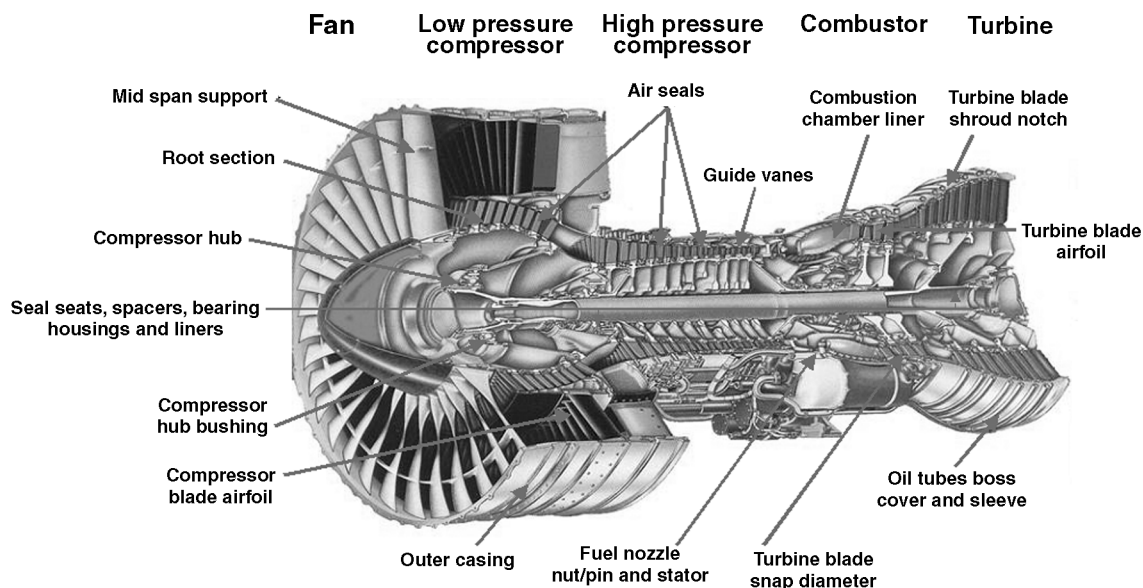


Fig. 6 Current and potential thermal spray coating applications for aircraft turbine engine parts. Source: Ref 4

process applications for applying zinc as a protective coating, primarily to prevent corrosion on structures. The “metallizing” industry thus began.

Developments after World War II

Thermal Spray Process Developments. Significant expansion of the technology did not occur, however, until after World War II, when powder spraying and plasma spray were developed and introduced. Many improvements in these processes have been made since then, but the basic operating principles remain unchanged. Powders are now fed more directly into the flames of combustion devices, which have been modified to generate higher-velocity jets. In addition, feedstock materials have improved significantly, to the point where they are now tailored for the process. Figure 7 summarizes the thermal spray process, applications, and materials growth, highlighting important milestones in its development history.

Application interest grew in these two thermal spray processes, with the first plasma spray coating process introduced by Reinecke in 1939. As welding technology developed, the plasma spray process also grew. Just prior to Reinecke, a confined arc gas heater was developed to assist in cutting and joining. Reinecke was the first to demonstrate that powders injected into a plasma arc gas heater could create molten particles, which could be accelerated toward a surface to form a coating. One advantage of the plasma spray process was significantly higher process temperatures compared to the combustion spray jet. Another advantage was the independence of the material feed from the heat source, compared to the electric current-carrying wires of the wire arc spray process. Plasma processes—with their inert plasma-forming gases, greater heating potential (due to the high process temperatures), and higher particle speeds—were largely responsible for the increased coating quality and subsequent growth of high-technology thermal spray applications through the 1980s.

Thermal spray equipment technology saw much of its progress in the late 1950s, with flame or combustion spray, electric arc, and

Table 2 Thermal spray processes used in various industrial sectors

Industry sector	Oxyfuel	Spray/fuse	HVOF	D-Gun	Air plasma	Vacuum plasma	Shroud plasma
Aero gas turbine	X		X	X	X	X	X
Agriculture	X	X			X		
Architectural	X	X			X		
Automotive engines	X		X		X		
Business equipment			X	X	X		
Cement and structural clays	X	X	X				
Chemical processing	X	X	X	X	X		
Copper and brass mills	X						
Defense and aerospace	X		X	X	X	X	
Diesel engines	X		X		X		
Electrical and electronic			X	X	X	X	
Electric utilities			X		X		
Food processing	X	X	X	X	X		
Forging	X		X				
Glass manufacture		X		X	X		
Hydro-steam turbines	X		X	X	X	X	X
Iron and steel casting			X		X		
Iron and steel manufacture	X	X			X		
Marine manufacture and repair	X						
Medical			X	X	X	X	X
Mining, construction, and dredging	X	X	X		X		
Nuclear			X	X	X		
Oil and gas exploration	X	X	X	X	X		
Printing equipment			X	X	X		
Pulp and paper	X		X		X		
Railroad	X		X		X		
Rock products	X	X	X		X		
Rubber and plastics manufacture	X	X	X		X		
Screening							
Ship and boat manufacture and repair	X						
Land-based gas turbine	X		X	X	X	X	
Steel rolling mills	X	X	X		X		
Textile	X		X	X	X		
Transportation, non-engine	X			X	X		

Source: Frank Hermanek

plasma spray all making parallel but separate advances. The detonation gun (D-Gun) process and further improved arc gas heater technology for powder spray applications were introduced. At its inception, the D-Gun proved capable of producing the highest bond strength and the most adherent, dense, and reliable wear-resistant coatings that thermal spray technology could offer. From 1960 to today, no equivalent process has emerged for producing D-Gun-type coatings. However, HVOF coatings are now challenging this position. Prior to the inception of the D-Gun process, aircraft engine manufacturers did not specify thermal spray coating methods, due to relatively low coating adherence and high coating porosity levels. Plasma spray, with its higher level of materials flexibility, had experienced similar high application growth for many noncarbide and carbide coatings and oxides for thermal protection. This was partially due to the acceptance of the process by aircraft engine manufacturers.

Driven by aerospace industry needs, many companies modified plasma arc heaters to improve coatings and advance application development. Hence, plasma spray emerged in the early 1970s as the most widely used high-tech thermal spray coating technology. Many equipment and materials developments were aimed at this growing market. Plasma spray guns, initially with 72 to 145 MJ (20

to 40 kW) input powers, now exceed 900 MJ (250 kW). Continuous system operation was enabled by improvements in water cooling and electrodes and by the use of higher arc voltages. Gas flows have concurrently increased, with plasma guns evolving from subsonic to supersonic gas-exit speeds. Subsequent increases in particle speeds have increased coating densities and bond strengths to the point where today thermal spray (especially plasma spray) is widely used in critical aircraft and even in biomedical coating applications. Although spray device developments have slowed, many thermal spray advances now focus on improved process control, targeting computer-controlled consoles, robotics, real-time sensors, and automated handling systems.

Combustion spray technology, with the exception of D-Gun, has also seen advances due to the development of HVOF spray systems. The HVOF spray has improved the combustion spray jet by increasing particle temperatures and speeds through confinement of combusting gases and particles. The resulting higher particle temperatures and speeds have significantly increased coating densities and bond strengths compared to conventional flame spray. The HVOF processes are seriously challenging the D-Gun and plasma coating market. It has been reported that higher particle speeds reduce particle overheating, thus preventing the oxida-

Table 3 Thermal spray coatings used in various industrial sectors

Industry sector	Carbides	Self-fluxing	Iron and steel	Nickel alloys	Superalloys	MCrAlY	Cobalt alloys	Nonferrous
Aero gas turbine	X		X	X	X	X	X	X
Agriculture		X	X	X				X
Architectural	X							X
Automotive engines	X			X	X	X	X	
Business equipment								X
Cement and structural clays		X	X					X
Chemical processing			X	X	X		X	
Copper and brass mills							X	
Defense and aerospace	X	X	X	X	X	X	X	X
Diesel engines	X		X	X	X	X	X	
Electrical and electronic								X
Electric utilities		X	X	X			X	X
Food processing		X	X					
Forging		X	X	X	X		X	
Glass manufacture	X	X	X					
Hydro-steam turbine	X	X	X	X	X		X	X
Iron and steel casting		X	X					X
Iron and steel manufacture		X	X	X	X		X	X
Medical								X
Mining, construction, and dredging		X	X					X
Nuclear								
Oil and gas exploration		X	X	X			X	X
Printing equipment								X
Pulp and paper		X	X	X				X
Railroad		X	X	X				X
Rock products		X	X					X
Rubber and plastics manufacture		X	X	X			X	
Screening			X					
Ship and boat manufacture/repair			X	X				X
Land-based gas turbine	X		X	X	X	X	X	X
Steel rolling mills		X	X	X	X		X	X
Textile			X					
Transportation, non-engine			X	X				X

Source: Frank Hermanek

tion and decarburization of carbides sometimes seen in plasma-sprayed coatings.

Advances in feedstock materials (consumables) led to growth in thermal spray technology applications. Metals, ceramics, and now composite feedstock materials are all being developed for specific thermal spray processes. The manufacture of wire by extrusion and drawing was limited to materials that could be drawn to 0.8 mm (0.032 in.) diameter; the introduction of cored wires enabled electric wire-arc spray to produce wear-resistant cermet (composites of ceramics and metals) coatings. Powders, the original sprayed materials, were first produced by crushing and sieving to size. Powder and particulate spray method develop-

ments provided a much broader range of materials that could be made into coatings. To support the need for particulate materials, powder-atomization techniques have advanced, yielding a wide range of high-quality materials for powder spray processes.

Other powder production methods are now available for making thermal spray feedstock, such as those for ceramic and ceramic-alloy powders. For example, chemical methods can form particulates from solutions by sol-gel processing and/or by fusing and crushing. Agglomeration by spray drying, sintering, and even plasma spray densification are recent advances used to make metal, ceramic, and composite cermet powders, as shown in Fig. 8. Powder production technology now produces powder sizes and

Table 4 Thermal spray coating properties of importance for various industrial applications

Industry sector	Wear						Thermal barrier	Clearance control			Corrosion/oxidation	Electrical	
	Abrasive	Adhesive	Fretting	Erosion	Cavitation	Impact		Abradable	Abrasive	Restoration		Resistance	Conductivity
Aero gas turbines	X	X	X	X			X	X	X	X	X		
Agriculture	X			X		X				X			
Architectural	X					X							
Automotive engines	X	X		X		X	X	X	X	X	X	X	
Business equipment	X	X	X										
Cement and structural clays	X					X				X	X		
Chemical processing	X			X						X	X		
Copper and brass mills	X									X	X		
Defense and aerospace	X	X	X	X	X	X	X			X			
Diesel engines	X	X		X		X	X			X	X		
Electrical and electronic													
Electric utilities	X	X		X	X	X				X	X		
Food processing	X									X			
Forging	X	X				X				X	X		
Glass manufacture	X	X								X	X		
Hydro-steam turbines	X	X	X	X	X					X	X		
Iron and steel casting	X			X		X				X	X		
Iron and steel manufacture	X			X		X				X	X		
Medical	X		X								X		
Mining, construction, and dredging	X			X	X	X				X	X		
Nuclear											X		
Oil and gas exploration	X	X		X		X				X	X		
Printing equipment	X	X								X			
Pulp and paper	X				X	X				X	X		
Railroad	X	X				X				X	X		X
Rock products	X					X				X	X		
Rubber and plastics manufacture	X			X		X				X	X		
Screening	X					X				X	X		
Ship and boat manufacture/repair	X			X						X	X		
Land-based gas turbines	X	X	X	X			X	X	X	X	X		
Steel rolling mills	X	X				X				X	X		
Textile	X									X			
Transportation, non-engine	X	X					X			X	X	X	

Source: Frank Hermanek

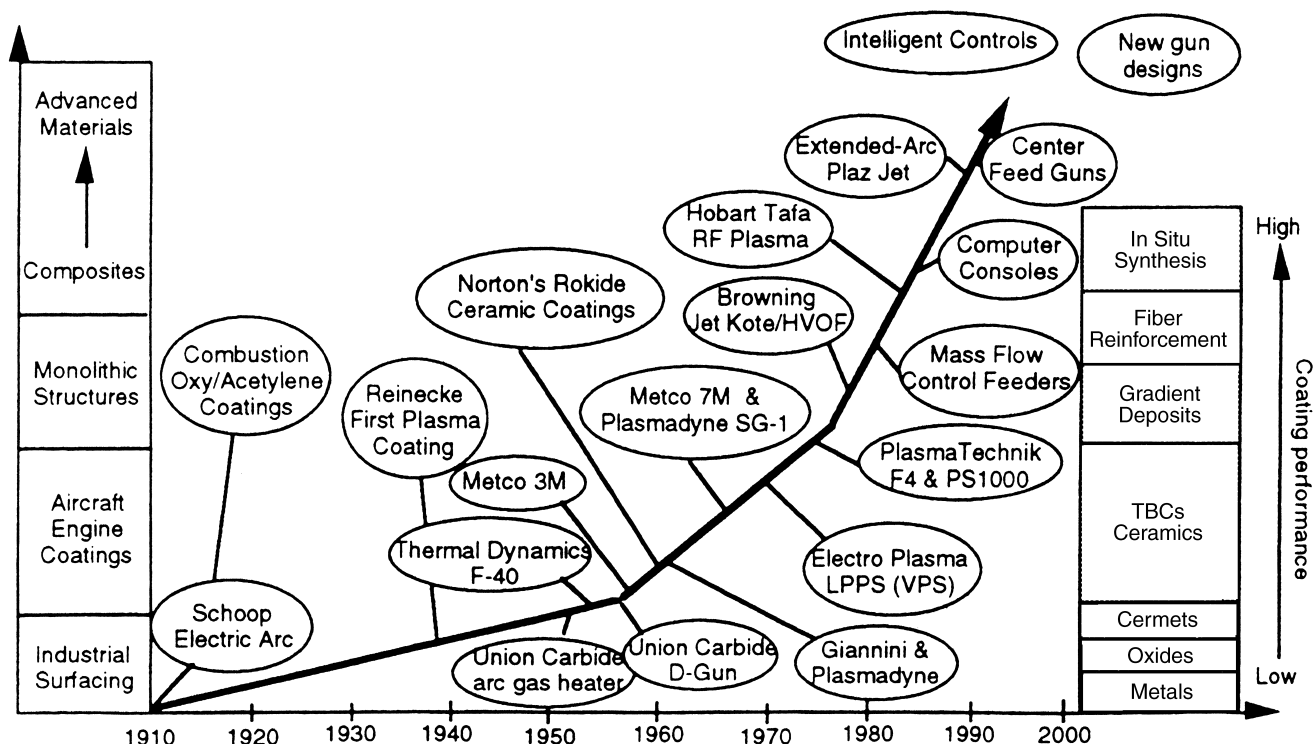


Fig. 7 Timeline of thermal spray developments, equipment, processes, and materials

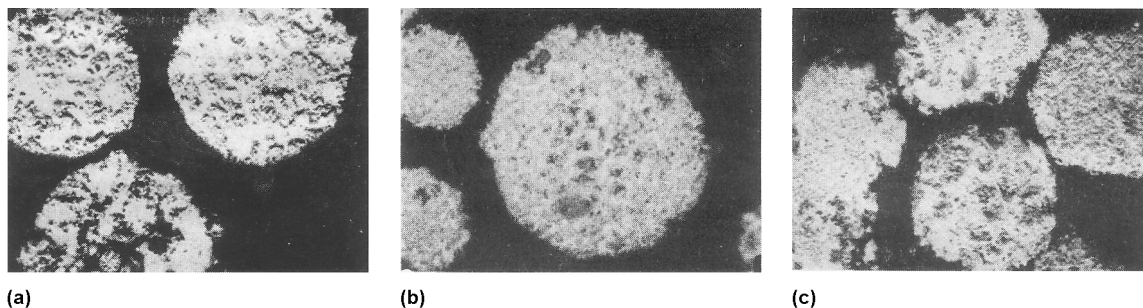


Fig. 8 Recent advances in thermal spray applications. Optical micrographs of powder cross sections. (a) Fe/TiC. (b) Tool steel/TiC agglomerated, metal/TiC plasma densified. (c) NiCr/TiC. Courtesy of Alloy Technology International

distributions tailored to fit particular thermal spray devices. The variation and supply of materials that can now be thermal sprayed is unlimited, with even cermet electric arc spray coatings available via cored wires. More detailed information on advances in feed-stock materials can be found in the Section "Materials Production for Thermal Spray Processes" in this Handbook.

REFERENCES

1. R. Knight and R.W. Smith, Thermal Spray Forming of Materials, *Powder Metal Technologies and Applications*, Vol 7, ASM Handbook, ASM International, 1998, p 408–419
2. R.C. Tucker, Jr., Thermal Spray Coatings, *Surface Engineering*, Vol 5, ASM Handbook, ASM International, 1994, p 497–509
3. M.L. Thorpe, Thermal Spray: Industry in Transition, *Adv. Mater. Process.*, Vol 143 (No. 5), 1993, p 50–56
4. B. Walser, The Importance of Thermal Spray for Current and Future Applications in Key Industries, *SPRAYTIME*, Vol 10 (No. 4), 2004, p 1–7
5. J. Read, International Thermal Spray Association, Keynote address, China International Thermal Spray Conference and the 16th National Thermal Spraying Seminar, Dalian, China, 22–25 Sept 2003

Surface Science

Thomas Bernecki, BIRL, Northwestern University

COATINGS AND SURFACE MODIFICATION TECHNOLOGIES allow the engineer to improve the performance, extend the life, and enhance the appearance of materials used for engineering components. These technologies have been developed because the interactions of manufactured components with other components, liquid, and/or gaseous environments can result in component degradation and failure. Additionally, coating technologies enhance the performance of components by selectively applying coatings that perform specific tasks without compromising the benefits of the substrate material. Two examples of the diversity of coating applications can underscore the values of coating technologies. The application of abrasive-resistant optical coatings does not adversely affect the optical quality of the substrate, while providing a wear resistance not obtainable by the substrate. Secondly, the application of bearing material to a shaft allows the shaft to maintain its desired performance without the need to fasten additional components composed of bearing material, thereby reducing weight and cost and improving efficiency.

Furthermore, coatings are no longer limited to the traditional applications associated with wear and corrosion. The use of coatings for decorative effects, implant prostheses, and electrical isolation/conduction is increasing daily. As the technological needs of a society become more complex, the implementation of surface modification and coatings will assume an increasing role in meeting those needs.

The article provides an understanding of the various wear and corrosion phenomena. Additionally, it provides an introduction to various coating processes, their advantages, and their weaknesses. In particular, this article reviews:

- The various types of wear and the associated mechanisms of surface deterioration
- The two major forms of corrosion and how they affect surfaces through chemical activity
- Chemical, thermal, and thermochemical processes for coating applications and how they differ
- Concepts associated with the application of coatings from the vapor state by chemical vapor deposition and physical vapor deposition
- Concepts associated with weld overlays, and an understanding of where thermal spray stands in the scheme of coating technologies

What Is a Surface?

Webster's dictionary defines a surface as "the exterior face of an object, or a material layer constituting such an exterior face." For the purposes of this article, this concept needs to be extended, and a surface is defined as an interface between a solid object and its

surroundings. The object is limited to a solid, because coatings are applied to surfaces to protect them when they interface with other solids, liquids, or gases, or to enhance the surface for aesthetic purposes. In the performance of the designed task of a component, these interfaces encounter physical, chemical, electrical, and other forces. These forces can degrade the performance of these components and can even cause them to fail. By applying the best coating to the component surface, the effects of these forces may be mitigated. The key word in the previous sentence is *best*. The purpose of this article and this Handbook is to inform the reader how materials fail and what coatings and processes may provide the best solution to the problem.

How Surfaces Interact with Their Environments

While the mechanisms of wear and corrosion are used daily to sand, grind, polish, and etch materials, the annual cost associated with unwanted wear and corrosion in the United States is estimated to be in the hundreds of billion dollar range. A comprehensive understanding of the complex phenomena associated with wear and corrosion is beyond the scope of this article. It is the intent of this section to provide the vocabulary and general understanding of wear and corrosion processes.

Wear

Wear involves the physical removal of material from a solid surface by another surface or material. The principal reason wear occurs at a surface is the presence of asperities at a microscopic level. Under a microscope, these asperities resemble a grit-blasted surface. The degree to which these asperities exist can be represented as a surface roughness measured as the root mean square value of the asperity height. One of the first attempts to reduce friction and wear was to polish the surface. By using the mechanisms of wear in a positive manner, abrasive grits in decreasing sizes are used to minimize the height of the asperities and thereby reduce the surface roughness. It should be noted that despite advancements in the art of polishing, the asperities remain, being measured in the nanometer range, for example, on mirrors. In addition to surface roughness, wear requires an interaction with another object or material(s) under a load moving with relative motion to the surface. The relative motion need not be continuous or in plane with the surface. The degree of interaction of these factors—and therefore wear—can be categorized by the surface material, the interacting material(s), the surface loading, and the nature and speed of the relative motion. These variables can be used to characterize wear mechanisms as adhesive, abrasive, and/or fatigue.

Adhesive Wear. When two metal surfaces are in relative motion with each other and under load, the points of contact deform

and can weld together locally due to interatomic forces. If the resulting bond is stronger than the underlying material, the bonded material shears or plastically deforms, and work hardening occurs.

When material is transferred from one surface to another, the process is termed adhesive wear. (Note: The abrading material may not be metal or an alloy but a ceramic.) Adhesive wear can be further divided into mild and severe. Under normal conditions, all metallic surfaces are covered with a layer of adsorbed gases and/or chemical reaction products. The predominant chemical reaction products for metals exposed to air are oxides. The oxide layers are generally thin, and contact with opposing surface roughness can expose the underlying unoxidized metal. This newly exposed surface will have a strong tendency to adhere to the opposing surface, particularly if it too has regions that are oxide-free. Reoxidation of the exposed surfaces then begins anew.

When the oxide layer regrows faster than it can be removed, it is termed mild adhesive or oxidation wear. Mild wear also occurs when the materials in contact with each other have low adhesion characteristics, that is, hardened steels, nonmetallic materials, and thermochemically treated surfaces.

When the oxide layer cannot grow faster than it is removed, the process is termed severe adhesive wear. For similar materials that exhibit mild adhesive wear, this type of wear is usually the result of increased surface interactions at higher speeds and/or loads. A familiar form of severe adhesive wear is scuffing. Scuffing occurs when a liquid or solid lubricant that separates sliding or rolling metallic surfaces breaks down at high temperature, such as at the piston ring/cylinder wall of an internal combustion engine. The wear that results is characterized by tearing and smearing of the surface. It is mainly a problem associated with gears, cams, valves, piston rings, and cylinders.

Abrasive wear is the removal of material from a surface by harder material impinging on the surface (i.e., grit blasting) or moving along the surface under load (i.e., grinding). The material need not be a solid or of a single phase, but the interaction with the surface does result in indentation of the surface and may remove material by cutting, plowing, chipping, or fatigue cracking.

When a hard surface with asperities slides on a softer surface and removes material by gouging or plowing, the process is called machining wear. Alternatively, another form of machining wear occurs when hard abrasive wear debris becomes embedded in one or both interacting surfaces to form sharp edges. If the two surfaces are of different degrees of hardness, the particles will tend to embed themselves in the softer surface, causing wear of the harder surface.

When an abrasive material slides over a surface at relatively light loads without impact, the wear is called low-stress abrasion. Hardness is the most important property in resisting low-stress abrasion. For pure and annealed metals, the abrasive wear resistance is the inverse of the wear rate and increases linearly with metal hardness, as shown in Fig. 1. For hardened steels, the abrasion resistance is lower than for pure or annealed metals of equal hardness. Prior work hardening of the material does not significantly influence the resistance to low-stress abrasive wear.

When loose abrasive particles are present between surfaces in relative motion, the wear mechanisms are more complex and depend on many factors. Among these factors are:

- The hardness, size, and fracture strength of the abrasives
- The hardness and degree of roughness of the surfaces
- The load and relative motion of the surfaces

Gouging abrasion occurs when relatively large portions of a surface are removed by interacting with abrasive materials that may not necessarily be metals. The mechanisms of metal removal can include deformation, cutting, and chipping on a scale larger than the microstructure of the abraded material.

Impact wear can occur by the impingement of particulate materials onto a surface, by the impact of two surfaces, or through cavitation.

Particle erosion occurs when a liquid or gaseous stream containing particles impinges or impacts onto a surface. At low angles of impingement, the particles remove material through a micro-cutting action, and the surface is scratched in the direction of fluid flow. High surface hardness is desirable to resist abrasion due to low-angle impingement, and unless the particle velocity is very high, even brittle materials such as ceramics can be used. At high angles of impingement, wear is generated by deformation and fatigue in ductile metals and by micro/macrocrazing in ceramics. The ability of any material to resist wear due to high-angle impingement is related to the amount of energy it can absorb prior to fracture. This value can be determined by the measure of the area under the material stress-strain curve. The influence of impact angle on wear rate is shown in Fig. 2. This is typical of erosive wear from hard particles.

When two surfaces impact each other and one or both surfaces are stressed by the impact, percussive wear results. Several modes of wear/failure are possible, depending on the impact stress. If the impact stress exceeds the ultimate strength of the material, cracking or spallation of the material may occur after one or more impacts.

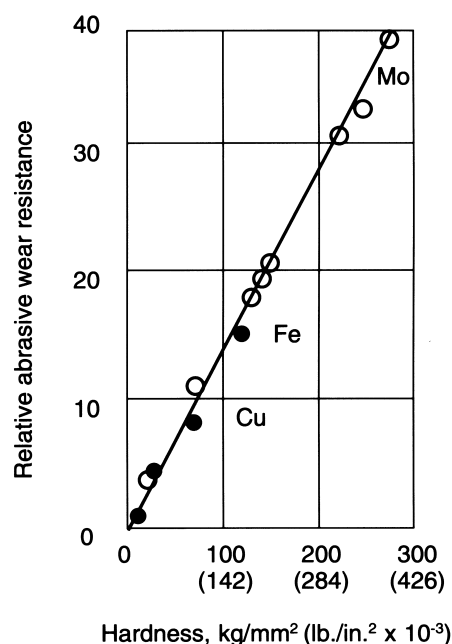


Fig. 1 Wear resistance of pure metals. Source: Ref 1

If, however, the impact stress is only higher than the yield strength of the material, successive impacts may plastically deform the material until the plastic limit of the material is exceeded and cracking results. When the impact stresses are below the yield strength of the surface material, fatigue may occur. It should be noted that peening and shot peening are processes that use repeated impact with a surface and, when used properly, can lead to increased fatigue resistance.

Cavitation is a phenomenon associated with hydraulic applications, particularly with pump rotors and ship propellers. As these components rotate, they generate regions of high and low pressure on their surfaces. Cavitation is the formation of bubbles that adhere to the surface, where the pressure falls below its vapor pressure, and boiling occurs. When the bubbles enter regions of high pressure, they collapse. When they collapse, those in contact with a surface can generate shock waves with sufficient force (as high as 60 ksi, or 4.2×10^7 kg/m²) to remove protective surface films and cause plastic deformation in many metals. The resulting area of deformation acts as a nucleation site for future bubbles, thus accelerating the process.

Fatigue Wear. Cracking, and ultimately, spallation, may occur from a surface subjected to cyclic or repetitive loading at levels below the yield strength of the material. This can be the result when the surface is exposed to low-frequency or high-frequency loading. The resulting failures are termed low-cycle fatigue and high-cycle fatigue, respectively. Cracking occurs at or below the surface and propagates in a direction dependent on the internal stress distribution, as well as the presence of discontinuities or voids within the component. Additionally, materials subjected to stresses related to the expansions and contractions associated with thermal cycling can exhibit surface fatigue. This form of wear is termed thermal fatigue, with the resulting surfaces resembling dried-up river beds and called mud-flat-type cracks.

Multiple-Wear Mechanisms. In real-life wear situations, there is a great probability that more than one wear mechanism will be present. The most familiar multiple-wear mechanism is fretting. Fretting occurs when two contacting surfaces undergo very small amplitude oscillations (as low as 10^{-7} mm, or 10^{-9} in.) and is characterized by the production of finely oxidized debris that is trapped

between the surfaces, resulting in accelerated abrasive wear. There is still some discussion as to the mechanisms associated with fretting. It is possible that oxidation can occur on asperities present on the surface. These asperities can then be removed from the surface by impact with a second surface, thus producing abrasive debris. Alternately, the material can be removed from the surface and then oxidized to produce the abrasive debris. Because oxidation is important in the generation of abrasive debris, fretting is often considered a form of corrosion. A classic case of fretting occurred in the early days of transporting automobiles by train in the United States. Because the rails did not have welded joints, the movement of the train over the rails created an oscillating motion that resulted in fretting of the automobile wheel bearings.

Corrosion

Corrosion is the result of the undesirable chemical interaction of a surface with its environment. As with all chemical reactions, the reaction rates and chemical kinetics (which are a function of concentration and temperature) determine the severity of surface degradation due to corrosion. In general, corrosion may be classified as oxidation, aqueous corrosion, dry corrosion, and stress-enhanced corrosion.

Oxidation is a process that occurs when a metal loses electrons as a result of interaction with either an atom or a molecule. The oxidation process can also occur when a metal changes valency by loss of electrons. While all too often oxidation is associated with the reaction of a metal in the presence of oxygen, the oxidation process is associated with the loss of electrons and, as often as not, includes reactions that do not involve oxygen.

Aqueous corrosion occurs in the presence of a liquid, usually an aqueous solution or an electrolyte. When two dissimilar metals are connected by an electrically conductive path and are immersed in a conductive aqueous solution, an electrical potential difference exists, and a current will flow between them. This is also called galvanic corrosion (Fig. 3) and is the mechanism by which non-rechargeable batteries operate.

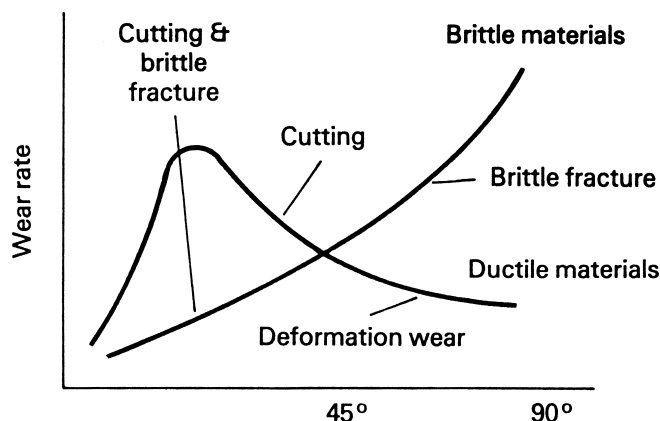


Fig. 2 Effect of impact angle on erosive wear. Source: Ref 2

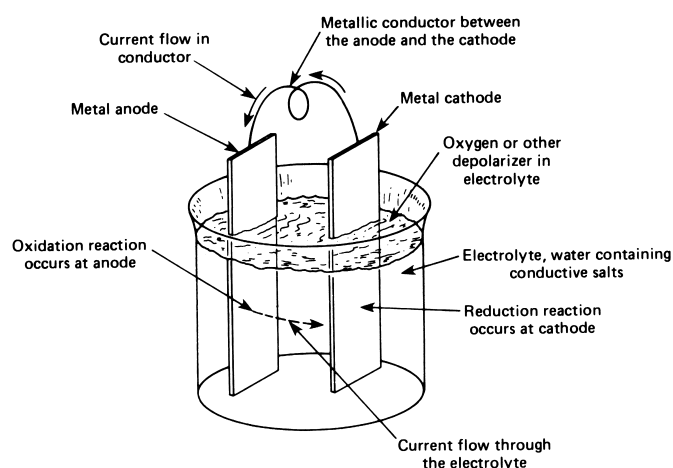


Fig. 3 Simple cell showing components necessary for corrosion

Of these two metals (the electrodes), the one having the highest electrode potential (cathode) will corrode less than it would if the two metals were not in electrical contact. While galvanic corrosion is one of the most widespread forms of corrosion, the process can be used to protect large structures, by making them the cathode of a galvanic cell and through the use of sacrificial anodes. By making the structure to be protected the cathode and by choosing an appropriate anode material, usually as a coating or in billet form, the rate of corrosion of the structure can be reduced.

A classic example is the application of zinc coatings to steel structures. The zinc is applied not because it is corrosion resistant but because it is not. Zinc provides long-term corrosion protection to steel through galvanic action at the zinc/steel surface as well as by its ability to protect itself with its own corrosion by-products. Zinc has a lower oxidation potential than iron and will therefore preferentially corrode, preventing the steel from rusting. Additionally, this property provides cathodic protection to any small discontinuities or damage done to the zinc coating that may expose the steel component. Being a reactive metal, zinc readily forms a protective corrosion product film. When exposed to air, a very thin layer of zinc oxide forms. When exposed to moisture in the atmosphere, the zinc oxide reacts with the moisture to form zinc hydroxide. During the drying process, the zinc hydroxide reacts with carbon dioxide to form an insoluble zinc carbonate layer on the surface, providing excellent protection to the underlying zinc. Figure 4 illustrates how the zinc corrodes differentially to protect the steel.

Dry corrosion is a chemical process that usually involves the interaction of a gaseous phase with a surface. An elevated-temperature environment usually accelerates the process. A classic example involves thermal barrier coatings. Thermal barrier coatings are used to protect surfaces from exposure to high temperatures. This is accomplished by applying an oxide ceramic coating with a low coefficient of heat transfer to the surface. This ceramic has a lower coefficient of thermal expansion (CTE) than the surface being protected. In order to minimize the stresses associated with this mismatch, an intermediate layer or bond coat is used. For certain bond-coat/ceramic systems operating at elevated temperatures, elements of the bond coat, mainly aluminum, diffuse to the bond-coat/ceramic interface and oxidize (dry corrosion). The by-product(s) of oxidation, mainly alumina, have a different CTE, which increases the potential for coating failure through induced stresses. Figure 5 illustrates this process for a commercially applied two-layer thermal barrier coating.

Another example of hot gas corrosion is associated with the combustion by-products of sulfur-containing gases condensing on cooler surfaces. The reaction with the substrate can often result in catastrophic failures due to rapid intergranular penetration of a liquid sulfide product and subsequent disintegration of the substrate.

Stress-Enhanced Corrosion. Surface damage is intensified whenever corrosion is concentrated in localized areas. Applied stresses and fatigue can enhance and promote corrosion in otherwise noncorroding conditions, and the mechanisms by which this enhancement takes place are still not well understood. What is known is that when certain surfaces are exposed to a corrosive environment and stresses are applied to the region where depletion has occurred, intergranular and intragranular cracking and failure

may occur. This phenomenon is called stress-corrosion cracking. Stress-corrosion cracking is generally associated with alloyed materials but may also occur in “pure” metals that have grain-boundary impurities.

Hydrogen Embrittlement. Alloys may fail under conditions of stress in a corrosive environment if hydrogen atoms have entered the alloy. This phenomenon is called hydrogen embrittlement. Hydrogen can be retained at levels in excess of its normal solubility limit. The excess hydrogen is retained and concentrated at interfaces between solid nonmetallic inclusions and the metal. The hydrogen also accumulates at voids. When an external stress is applied, the hydrogen concentration becomes localized, and intergranular cracks begin on the surface, in the region of maximum tensile stress.

Liquid Metal Embrittlement. Additional forms of embrittlement and corrosion occur when metals and alloys are exposed to

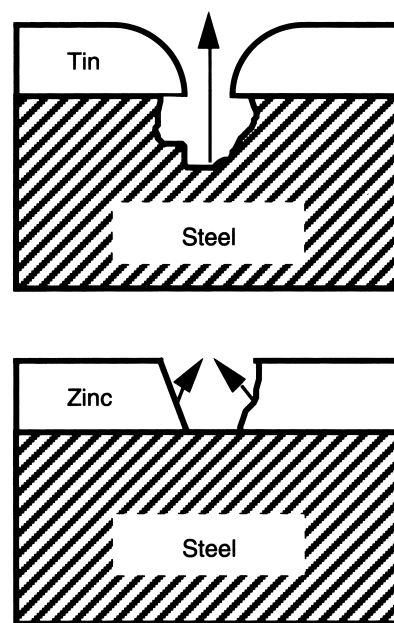


Fig. 4 Cathodic protection of steel by zinc

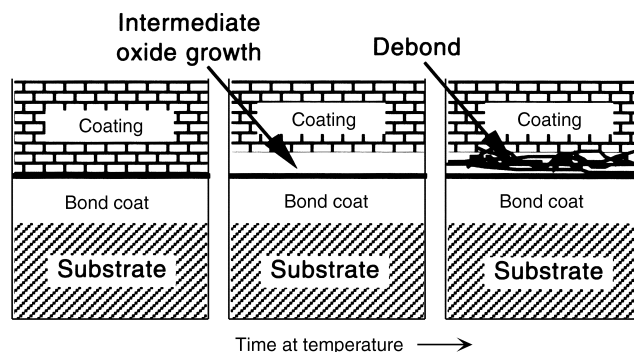


Fig. 5 Failure of a two-layer thermal barrier coating due to dry corrosion

metals in liquid form. Generally, for embrittlement to occur, a tensile stress and/or the existence of a crack or discontinuity are necessary, as well as adsorption of the embrittling species. Liquid metal embrittlement and corrosion is a complex process that is temperature dependent. Metal transfer can occur from hotter regions to cooler regions and also along activity gradients. The effects of relative solubility of the metals, impurities, species types, and diffusion rates on the formation of intermetallic compounds are factors influencing the type and extent of corrosion.

Electrical Isolation/Conduction

Oftentimes, it is necessary and desirable to electrically isolate a component from its environment. The ability to coat the component with polymers or oxide ceramics is a major application of many of the coating technologies discussed in this Handbook. Conversely, the ability to enhance the electrical conductivity by supplementing the substrate with a highly conductive coating is of equal importance in the implementation of coatings technology. The ability to enhance the electrical isolation/conduction by coatings is the basis of the semiconductor industry.

Biocompatibility

Within the medical community there is a great interest in coatings, particularly for use on medical and dental implants. The potential exists that 40 million people can be candidates for implants. Two types of materials are commonly used. Bioinert materials are desirable because, when used *in vivo*, they will not degrade in the human body, nor would they tend to be included in biological reactions. Bioactive materials are desirable because they elicit a host response on implantation.

Two bioinert metals, commercially pure titanium and the Ti-6Al-4V alloy, are used in coating form and are designed to maximize high fatigue life and to produce very high surface roughness with open porosity. The pores are designed to be of a size between 50 and 100 μm , which has been shown to encourage capillary growth, thereby providing blood to sustain bone tissue development and growth. Bioactive materials typically are ceramics coatings, such as hydroxyapatite, which has a chemical composition similar to bone. The desired host response is to encourage normal bone tissue to grow as close to the implant surface as possible.

Understanding Thermal and Thermochemical Processes

From the earliest attempts to work metals, four major factors have had to be addressed: the material being worked, heat, the reaction environment, and quenching of the surface. The same four factors are addressed today but with the help of roadmaps developed through years of research. These roadmaps, called phase diagrams, allow the engineering of the final properties of the surface. Figure 6 is a phase diagram for steel and iron. As can be seen from the *x*-axis, steel is classified based on its carbon content. General steel (wrought and cast) has carbon content between 0.05 and 2%, while ductile iron has carbon content between 3.5 and 4.5%. Hypoeutectoid steels are those with carbon content below 0.8%. The *y*-axis is

temperature, and the diagram indicates the phases present at a given temperature for a given percentage of carbon in steel/iron. The phase diagram indicates the physical form(s) the atoms assume under those conditions and the related physical properties of hardness and degree of brittleness that the surface would have if those phases could be “frozen” by the proper quench. Low-carbon steels are important because of their high ductility, which allows easy fabrication while maintaining toughness and strength. At room temperature, a body-centered cubic phase is present. On heating to an elevated temperature, a face-centered cubic phase (austenite) is formed, which is more densely packed. By rapidly quenching to below 215 °C (420 °F), a martensite phase is formed, which is the hardest structure formed from austenite.

Surface Hardening

Surface hardening, otherwise known as case hardening, is a thermal process by which a material, usually a low-alloy steel with between 0.35 and 0.60% C or a cast iron, is austenitized and quenched to form a martensitic. This process produces hardnesses of 700 DPH. The surfaces are usually tempered at 200 °C (390 °F), resulting in hardnesses of 600 DPH. The depth of hardening is normally in the range of 0.5 to 5 mm (0.02 to 0.19 in.).

Flame Hardening

Flame hardening is a combustion process employing fuel gases such as acetylene, propane, methyl-acetylene propadiene, or natural gas burning in air or oxygen. For one-of-a-kind components or for localized regions on numerous components, such as rolls, it is still an energy-efficient method for case hardening. The choice of fuel and oxidant is based on the desired speed of application of the coating and the desired depth of hardening as well as the associated cost.

Induction Hardening

By far, the most widely used method for case hardening components having a circular or near-circular cross section is induction heating of the surface. Typically, an alternating current flows through a work coil (Fig. 7), causing an eddy current to be induced in the component, thereby generating heat. The skin depth or depth of penetration is a function of frequency (Table 1) and other properties of the material.

For steels to be sufficiently hardened, it is necessary that carbides dissolve when the steel is heated; therefore, steels possessing smaller carbide particles are more responsive to induction heating. Additionally, the induction-hardening process introduces compressive stresses into the surface of the component, thereby improving its fatigue properties.

High-Frequency Resistance Heating

In this process (Fig. 8) for noncircular components, a water-cooled conductor is placed in close proximity to the surface to be heated and connected to the workpiece through a pair of contacts at the outer edges and to a power source. When the high-frequency current is applied, heating occurs across the work surface to a

depth dependent on the frequency used, the duration of heating, and the power level, normally in the range of 0.37 to 0.75 mm (0.014 to 0.030 in.). Once the heated strip reaches the hardening temperature, the power is switched off, and self-quenching occurs by heat dissipation to the bulk of the component. A significant advantage of high-frequency resistance heating is that it does not require a closed loop of current but can heat a path between two points, with the hardness between two points being dictated by the shape of the conductor.

Plasma Heating

An inexpensive method for the hardening of cast iron involves the use of a tungsten inert gas torch to rapidly heat portions of the surface of a component. The use of an inert gas minimizes the oxidation effects that can occur through use of a high-temperature energy source in air. The inert gas also aids in the rapid quenching necessary for the hardening process.

Laser Heating

When component distortion due to overheating is a concern, solid-state transformation hardening by lasers is employed. The

laser interacts with the surface of a material to a depth dependent on the power density and interaction time (Fig. 9). The surface of a ferrous material can reach austenitization temperatures after very short interaction times. Because the hardening occurs within the narrow beam of the laser, the bulk of the material remains unaffected by the heat. After the laser heating is removed, the bulk of the component acts as a heat sink, and therefore, no external quenching is necessary to bring about the martensitic transformation.

A carbon dioxide (CO_2) laser operating at a wavelength of 10.6 μm is the most widely used laser in the heat treating industry, because of the relatively high power densities available. Because clean metallic surfaces can reflect up to 95% of the incident radiation at this wavelength, the surfaces to be hardened are coated with a material that absorbs at this wavelength. One such material is colloidal graphite.

Electron Beam Heating

Electron beam hardening, while not as widespread as laser hardening, is also a solid-state transformational hardening process that uses direct impingement of a highly focused electron beam to provide surface heating. This process requires a vacuum system,

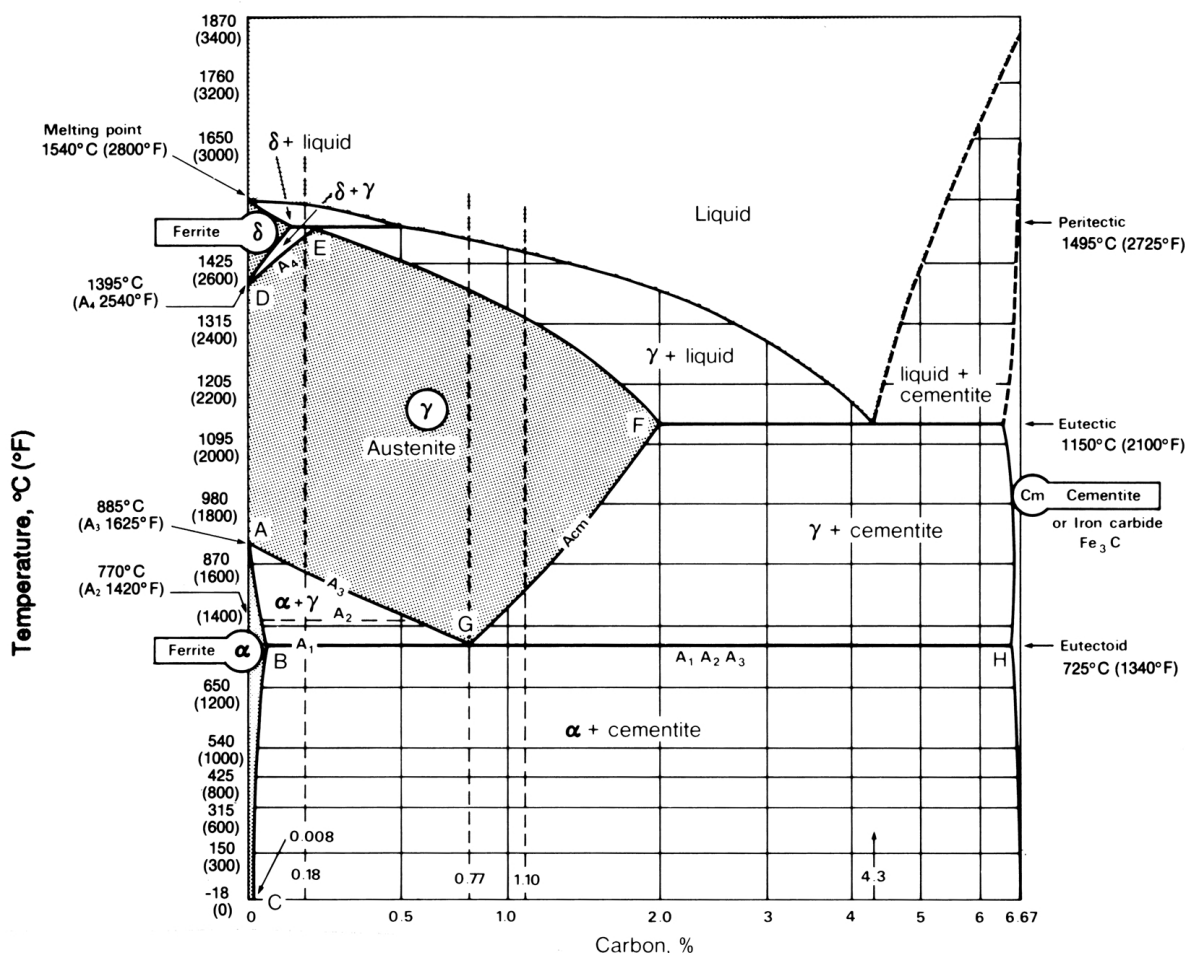


Fig. 6 Phase diagram for the iron-carbon system

adding capital expense while providing an environment conducive to protecting the heat treated component. Table 2 provides a comparison of laser and electron beam hardening processes.

Thermochemical Treatments

Thermochemical processes provide environments that introduce chemical elements into surfaces at elevated temperatures. The elements of interest are primarily carbon and nitrogen, although niobium and vanadium are often diffused into steel to form very hard carbide layers. The elements can be transferred from solid, liquid, gaseous, or plasma media. Like thermal processes, thermochemical processes rely on a surface reaction and transformation to a martensitic phase to produce a wear-resistant surface. Nitriding and nitrocarburizing are processes performed at temperatures below the formation of the austenite phase, and hardening occurs by means of a fine-scale dispersion of nitrides and carbonitrides in the surface layer of the matrix material.

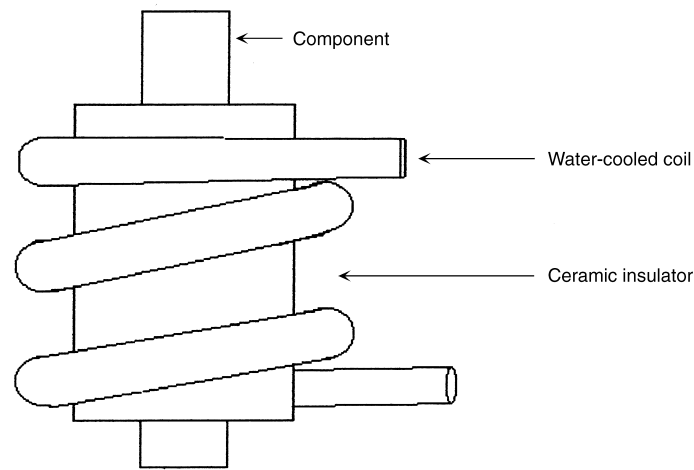


Fig. 7 Induction heating setup

Table 1 Conditions for induction heating

Depth of hardening		Frequency, kHz	Power input	
mm	in.		W/mm ²	kW/in. ²
0.5–1.1	0.020–0.045	450	15–19	10–12
1.1–2.3	0.045–0.090	450	8–12	5–8
1.5–2.3	0.06–0.09	10	15–25	10–16
2.3–3.0	0.09–0.12	10	15–23	10–15
3.0–4.0	0.12–0.16	10	15–22	10–14
2.3–3.0	0.09–0.12	3	23–26	15–17
3.0–4.0	0.12–0.16	3	22–25	14–16
4.0–5.0	0.16–0.20	3	15–22	10–14

Source: Ref 3

Carburizing

Carburizing involves the heating of steel above its ferrite/ austenite transformation temperature (815 to 1090 °C, or 1500 to 2000 °F) to a region where the carbon solubility is high. The parts are then quenched to develop a martensitic structure in the surface layer. The hardness increases with carbon content to a maximum at the eutectoid level (Fig. 6), depending on the steel composition and the process used. The carburized surface may have a hardness of up to 900 DPH and a depth of up to 4 mm (0.15 in.). The depth of carburization can be expressed in either of two ways: actual or effective. The actual depth is measured by removing a cross section for chemical and material analysis. The effective depth is determined by a specific lower limit of carbon content or hardness. Carburizing can be performed in a solid, liquid, gas, or plasma media.

Pack carburization involves placing components into a steel container with a carbon source such as coke and an energizer such as barium carbonate. A furnace is used to heat the container to approximately 900 °C (1650 °F). Quenching directly from the carburizing temperature is not possible and thus requires reaustenitization and quenching.

Salt bath carburizing is carried out in a bath containing a mixture of sodium cyanide and an alkaline earth salt. The most likely reaction sequence is the oxidation of the cyanide into a cyanate that dissociates at the steel surface:

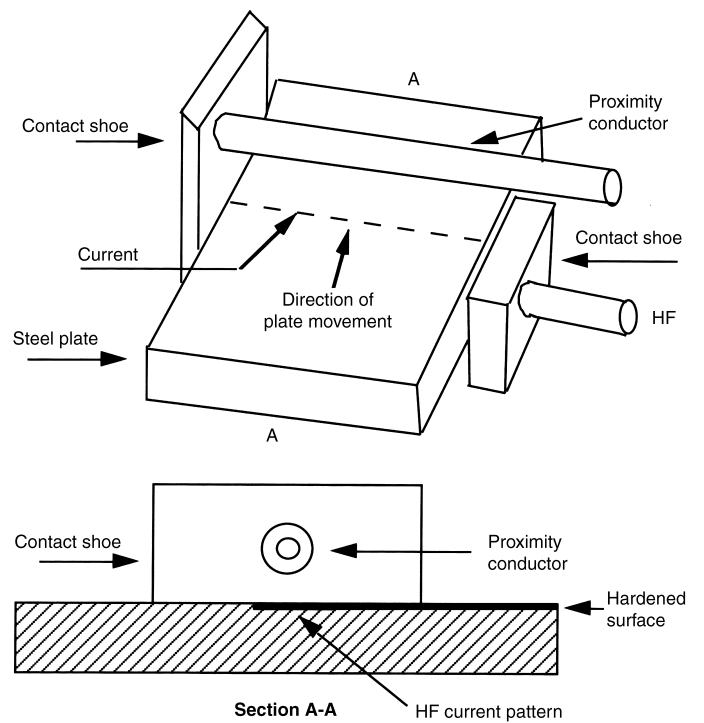
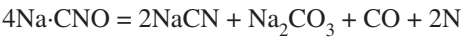
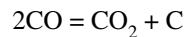


Fig. 8 Basic elements of high-frequency (HF) resistance surface hardening

where the nitrogen is solutioned in the iron. The carbon monoxide, in turn, reacts with the steel:



where the carbon is solutioned in the iron. Salt baths are normally operated at 850 °C (1560 °F) and usually are used for depths less than 0.5 mm (0.02 in.).

Gaseous techniques make up the bulk of commercial carburization. This is due largely to the ability to accurately control the surface carbon levels. Carburizing atmospheres can be generated in many ways; the basic reaction, however, is the same. Initially, a flow of carrier gas of low carbon potential is established, either inside or outside the furnace. The carbon potential is defined as the surface carbon level achieved on a steel surface in a particu-

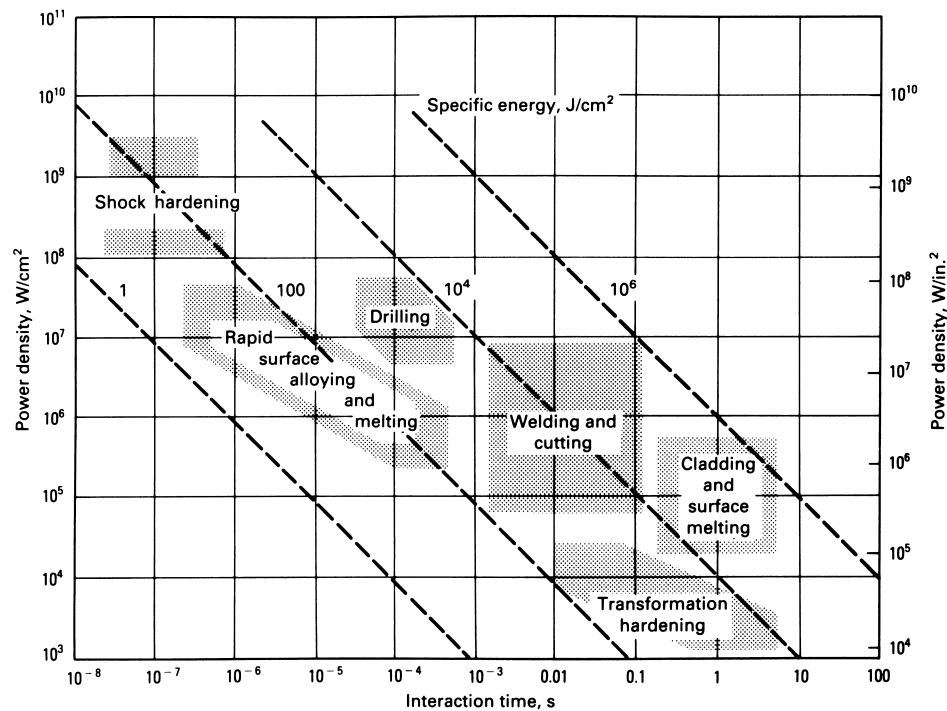


Fig. 9 Operational regimes for laser surface processing

Table 2 Comparison of laser and electron beam hardening

Item	Electron beam	Laser
Bore size limitation	Line of sight at 35% impingement	25.4 mm (1 in.) diam and up; no limit 6.3–25.4 mm (0.25–1 in.); using small spot size
Part contamination	Parts should be clean	Parts should be clean at reaction area
Focused spot size	Hard vacuum, 0.5 mm (0.02 in.) diam	Varied; depends on system and optics Soft, 0.75 mm (0.030 in.) diam
Heat treat pattern(a)	Shape and density controllable by computer or function generator	Shape limited to mirror deflection capabilities Density averaged
Beam deflection	Electromagnetically 30° included; two axes	Water-cooled reflecting optics—mechanical or integrating
Effect of vacuum	Production systems prepumped; no effect on cycle time. Low-volume systems, minimum 5 s pumpdown but depends on chamber size	No effect
Part size limitations	Limited to chamber size and motion required	No limit
Power available	100+ kW	15 kW max (commercial), 100+ kW (experimental)
Conversion efficiency	>90%	>10%
Operating costs	Low	High, due to cost of gases and low efficiency
Surface preparation	None; can be done on finish-ground surfaces	Can be done on finish-ground surfaces but requires an absorptive coating that may need cleaning
Investment	Lowest on high-power applications	Lowest on low-power applications

(a) Pattern size is controlled by the available power of the equipment. Nominally, 15–30 W/mm² (10–20 kW/in.²) is used. Source: Ref 3

lar atmosphere at equilibrium conditions. Hydrocarbons are then added to increase the carbon potential to the desired level.

Carbonitriding

Carbonitriding is a process used to increase hardenability and provide a more uniform depth of hardening on plain carbon or very low-alloy steels. This is achieved by introducing 0.5% of nitrogen, in addition to carbon, into the surface of the steel. In salt bath carbonitriding, the amount of oxygen supplied to the bath is increased, which increases the amount of sodium cyanate in the bath, thereby producing more nitrogen. In the gaseous carbonitriding process, ammonia (NH₃) gas is introduced into the carburizing atmosphere as a source of nitrogen.

Nitriding

Nitriding is a process in which nitrogen is diffused into a steel surface at temperatures of 495 to 565 °C (925 to 1050 °F). Atomic nitrogen is formed at the surface of the steel and then diffuses inward. The atomic nitrogen can be generated by catalytic dissociation of ammonia or by a nitrogen-hydrogen plasma. During the process, a superficial layer is developed on the surface. As the layer increases in depth, the nitrogen diffuses from it into the surface and reacts with the steel to form well-dispersed nitrides. This layer, being very brittle, is removed by grinding, resulting in a layer measuring 25 µm to 0.7 mm (0.001 to 0.030 in.), with a hardness of 900 to 1100 DPH.

Nitrocarburizing

Nitrocarburizing involves the diffusion of nitrogen and carbon into a surface to form an ~20 µm (~7 × 10⁻⁴ in.) adherent compound layer over a diffusion zone of well-dispersed nitrides. Nitrocarburizing, done either by molten cyanide/cyanate baths or by gaseous means, is primarily used in the automotive industry for camshafts, crankshafts, rocker arms, rocker shafts, and so on, where an inexpensive method for improving the performance of low-carbon and low-alloy steels is desirable.

Boriding

Boriding is a diffusional technique by which boron is diffused into a metal substrate to form a metal-boride layer approximately 50 to 150 µm (0.002 to 0.006 in.) thick and having hardnesses of 1500 to 3000 DPH. Boride layered surfaces exhibit good wear and corrosion characteristics as well as resistance to attack by molten metals. The majority of boriding processes are performed at temperatures between 700 and 1000 °C (1300 and 1830 °F). Pack boriding involves the use of boron carbide as a boron source with an activator compound such as BaF₂, NH₄Cl, or K₂BF₄. Paste boriding requires the application of a commercially available paste supplied in a viscous state. Water is added, and several layers of the paste are applied. The components are then either inductively heated in a protective atmosphere or placed in a controlled atmosphere furnace. Salt bath boriding involves either thermochemical or electrolytic techniques. Gas boriding is carried out using a 75/25 nitrogen/hydrogen carrier gas containing 0.5% BCl₃.

Comparing Thermochemical Treatments

The various thermochemical surface treatments are compared and summarized in Table 3, and their respective surface hardness and processing temperature ranges are shown. Figure 10 shows the wear performance that may be expected for three of the thermochemical treatment processes described previously.

Electrochemical Treatments

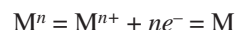
In the 1990s, many of the processes under discussion, as well as the thermochemical processes previously discussed, particularly their associated waste streams, received increased regulatory scrutiny from the United States Environmental Protection Agency (EPA). This resulted in research to clean up these processes or to find environmentally friendly replacements for them. Because the existing and proposed regulations are ever-changing, the latest information can be accessed through the EPA Web site (Ref 5). On the home page, enter the process or material of interest into the search box.

Electroplating

Electroplating is defined by ASTM B 374 as being the “electrodeposition of an adherent metallic coating upon an electrode for the purposes of securing a surface with properties or dimensions different from those of the base metal.” Figure 11 shows a schematic of a typical electroplating process.

In the aforementioned ASTM definition, the electrode on which the coating is to be applied is the cathode or work electrode. The anodes can be made of the material to be plated but can also be of a material that is only needed to complete the circuit, in which case they are called inert. The purpose of using electroplating is to apply coatings for decorative purposes, protection, special surface properties, or mechanical properties.

In its simplest form, metal deposition may be represented as:



Even when the metal atom M forms part of a negative complex ion, rather than existing as a simple (or hydrated) positive ion, it is still reduction by electrons that produces the metallic phase. If the potential at which the metal is deposited is more negative than that at which hydrogen ions can be reduced on that surface, then metal deposition and hydrogen evolution occur simultaneously. Thus, nickel is deposited at a cathodic current efficiency of approximately 95%, while chromium generally deposits at efficiencies below 20%. Metals with electrode potentials more than approximately 1 V more negative than the standard hydrogen potential, for example, titanium and aluminum, cannot be electrodeposited from aqueous solution. Metals and alloys most commonly deposited by electroplating include:

- Copper and copper alloys
- Chromium and chromium alloys
- Nickel and nickel alloys
- Iron

- Cadmium
- Zinc
- Indium
- Tin
- Lead
- Silver
- Gold
- Platinum-group metals

Brush Plating

Brush plating is an electroplating process that is performed with a handheld or portable plating tool rather than in a bath. The brush-plating process is also called contact plating, selective plating, or swab plating. In its simplest form, the brush-plating process is similar to a painting operation. The operator soaks the plating tool in the plating solution and then deposits the plating material by

brushing the plating tool against the component to be plated. Plating solution is delivered to the (cathode) work area by a porous, absorbent cover wrapped over the anode of the plating tool. This process is especially useful for treating components too large to immerse in a plating bath or belonging to equipment that is too expensive to dismantle. Table 4 lists several materials applied by either brush plating or by standard electroplating, and their degrees of hardness.

Anodizing

In brush plating, the cathode is, in general, the work electrode on which the coating is deposited. One major exception is the anodizing of aluminum and its alloys, where the work electrode is the anode. Aluminum as the anode in an aqueous electrolytic cell develops an oxide layer. If the electrolyte is a boric acid solution, the coating will grow until the resistance of the coating prevents

Table 3 Typical characteristics of diffusion treatments

Process	Nature of case	Process temperature		Typical case depth	Case hardness, HRC	Typical base metals	Process characteristics
		°C	°F				
Carburizing							
Pack	Diffused carbon	815–1090	1500–2000	125 μm–1.5 mm (5–60 mils)	50–63(a)	Low-carbon steels, low-carbon alloy steels	Low equipment costs; difficult to control case depth accurately
Gas	Diffused carbon	815–980	1500–1800	75 μm–1.5 mm (3–60 mils)	50–63(a)	Low-carbon steels, low-carbon alloy steels	Good control of case depth; suitable for continuous operation; good gas controls required; can be dangerous
Liquid	Diffused carbon and possibly nitrogen	815–980	1500–1800	50 μm–1.5 mm (2–60 mils)	50–65(a)	Low-carbon steels, low-carbon alloy steels	Faster than pack and gas processes; can pose salt disposal problem; salt baths require frequent maintenance
Vacuum	Diffused carbon	815–1090	1500–2000	75 μm–1.5 mm (3–60 mils)	50–63(a)	Low-carbon steels, low-carbon alloy steels	Excellent process control; bright parts; faster than gas carburizing; high equipment costs
Nitriding							
Gas	Diffused nitrogen, nitrogen compounds	480–590	900–1090	125 μm–0.75 mm (5–30 mils)	50–70	Alloy steels, nitriding steels, stainless steels	Hardest cases from nitriding steels; quenching not required; low distortion; process is slow; is usually a batch process
Salt	Diffused nitrogen, nitrogen compounds	510–565	950–1050	2.5 μm–0.75 mm (0.1–30 mils)	50–70	Most ferrous metals, including cast irons	Usually used for thin, hard cases <25 μm (1 mil); no white layer; most are proprietary processes
Ion	Diffused nitrogen, nitrogen compounds	340–565	650–1050	75 μm–0.75 mm (3–30 mils)	50–70	Alloy steels, nitriding steels, stainless steels	Faster than gas nitriding; no white layer; high equipment costs; close case control
Carbonitriding							
Gas	Diffused carbon and nitrogen	760–870	1400–1600	75 μm–0.75 mm (3–30 mils)	50–65(a)	Low-carbon steels, low-carbon alloy steels, stainless steel	Lower temperature than carburizing (less distortion); slightly harder case than carburizing; gas control critical
Liquid (cyaniding)	Diffused carbon and nitrogen	760–870	1400–1600	2.5–125 μm (0.1–5 mils)	50–65(a)	Low-carbon steels	Good for thin cases on noncritical parts; batch process; salt disposal problems
Ferritic nitrocarburizing	Diffused carbon and nitrogen	480–590	900–1090	2.5–25 μm (0.1–1 mil)	40–60(a)	Low-carbon steels	Low-distortion process for thin case on low-carbon steel; most processes are proprietary

(a) Requires quench from austenitizing temperature. Source: Adapted from Ref 4

current flow. In sulfuric, oxalic, or chromic acid solutions, the oxide coating is slightly soluble, and a porous coating will be produced. As the coating grows, it also dissolves, generating pores in the coating that provide a path for continued current flow. When the rate at which the coating dissolves equals the rate of coating

growth, the coating thickness will reach a limiting value based on the current density.

Chemical Treatments

Electroless plating is a process in which the deposited material is reduced from its ionic state in solution by means of a chemical reducing agent, rather than by an electric current. The base component to be coated must be catalytic in nature. When the component is immersed in the electroless solution, a uniform deposition begins. Minute quantities of the electroless metal itself will catalyze the reaction, and therefore, the process is autocatalytic in nature. The process will continue until either the metal ions are depleted or the reducing agent is depleted. Table 5 lists typical electroless plating baths and their deposition rates.

Phosphating is a chemical conversion process in which a metal surface reacts with an aqueous solution of a heavy metal, primarily phosphate plus free phosphoric acid, to produce an adherent layer of insoluble complex phosphates. In general, it is used mainly on mild or low-alloy steels. The principal phosphates involved are those of iron, zinc, and manganese. The biggest application is the pretreatment of mild steel prior to painting, where coatings improve adhesion and corrosion resistance. Coatings can be applied by immersion or alternatively by spraying or brushing. Additional uses of phosphating are corrosion resistance, electrical insulation, and improvement of lubricating properties.

Hot Dip Coating

One of the most widely used methods for protecting steel components from corrosion is hot dip coating. These coatings are applied either by a batch process or by a continuous process. In the batch process, fabricated steel parts ranging in size from nuts/bolts to light poles are dipped into a molten pool of coating material.

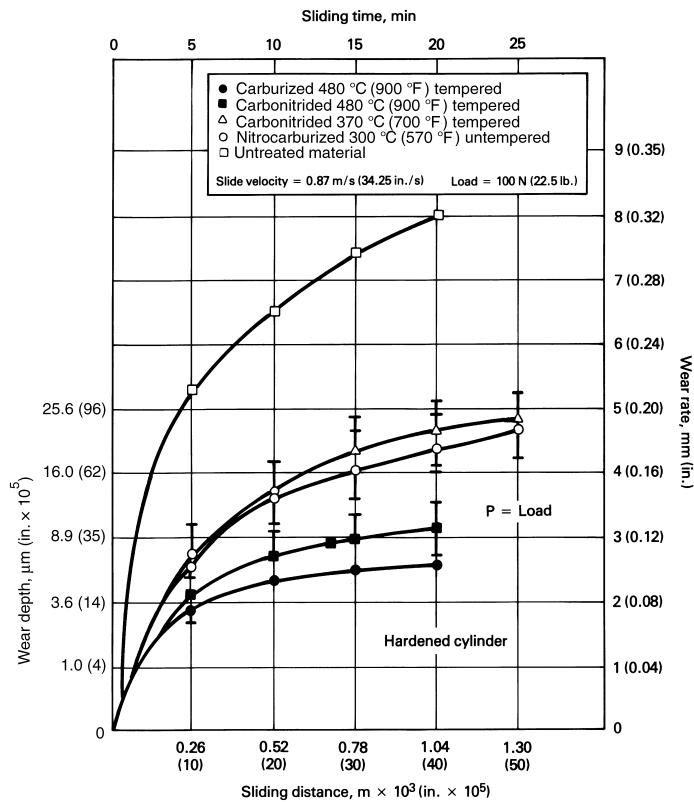


Fig. 10 Wear characteristics of 0.2% C steel. Source: Ref 2

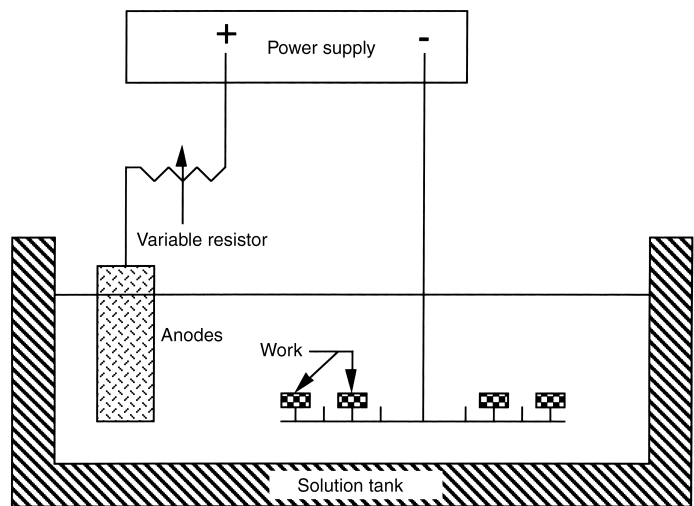


Fig. 11 Schematic of a typical electroplating process

Table 4 Electroplated materials and their relative hardnesses

Metal	Microhardness, DPH	
	Bath plated	Brush plated
Cadmium	30–50	20–27
Chromium	280–1200	580–780
Cobalt	180–440	510
Copper	53–350	140–210
Gold	40–100	140–150
Lead	4–20	7
Nickel	150–760	280–580
Palladium	85–450	375
Rhodium	550–1000	800
Silver	42–190	70–140
Tin	4–15	7
Zinc	35–125	40–54

Source: Ref 6

This process is only limited by the size of the component and of the molten pool. In the continuous process, rolls of steel sheet are continuously fed into a molten pool. Limitations to this process require that the steel sheet be flexible and the coating material be of a low melting temperature. The latter is critical to prevent deformation or tearing as the steel proceeds through a series of rollers present in the process. Materials typically used for the hot dipping process are zinc, zinc-iron alloy, aluminum, aluminum-silicon alloy, Zn-5Al alloy, 55Al-Zn alloy, and lead-tin alloys. Table 6 lists the alloys and their intended uses. It should be noted that the zinc coating process, more commonly known as galvanizing,

accounts for 86% of the hot-dip-coated steel produced in the United States.

Chemical Vapor Deposition (CVD)

Chemical vapor deposition is a generic term for a family of processes used for depositing coatings from the gaseous or vapor state (Table 7). Although considered a thin-film deposition process, coatings as thick as 25 mm (1 in.) have been successfully deposited by CVD. These processes incorporate the thermal decomposition of fluorides, chlorides, bromides, iodides, organometallics, hydrocarbons, phosphorus trifluorides, and ammonia complexes to deposit coatings of metals, alloys, intermetallics, boron, silicon, carbon, borides, silicides, carbides, oxides, and sulfides onto surfaces.

The CVD process is an omnidirectional process. Coatings produced by CVD processes produce theoretically dense, uniform, and well-adhered coatings with controlled microstructures. In this process, a gaseous precursor is thermally decomposed to produce a coating on the component surface. In general, CVD is a high-temperature process, thus limiting the choice of substrate material. An exception to this would be exothermic chemical reactions, where it is possible to obtain coatings at substrate temperatures lower than those associated with the decomposition of the precursor material. Because CVD is an omnidirectional process, all surfaces exposed to the gas are coated, including blind holes and the internal surfaces of porous bodies (Fig. 12).

Microstructures of the coatings can be controlled by temperature and supersaturation, which is the ratio of the local effective concentration of the material to be deposited, relative to its equilibrium concentration (Fig. 13).

Microstructural control is critical for optimizing the mechanical properties of the coatings. The high temperatures associated with the CVD process result in coatings that exhibit diffusion bonding of the coating to the substrate, resulting in well-adhered coatings with reduced interfacial stresses at the coating/substrate interface. In conventional CVD, the heating is done either by resistance or induction heating. The desire to overcome the limitation of substrate temperature while not losing the advantage of diffusion bonding has led to the use of alternate energy sources. Electric discharges, radio frequency, and microwave plasmas have been used to lower substrate temperatures to below 350°C (660°F) while maintaining diffusion bonding.

Plasma-Assisted CVD (PACVD) or Plasma-Enhanced CVD (PECVD)

For the purpose of this discussion, plasma is defined as a gas that has absorbed sufficient energy to strip the atoms of their electrons. This plasma is composed of ions, atoms, molecules, and free high-energy electrons. Therefore, a plasma can decompose reactant gases that flow through it, even at low temperatures where they normally would be stable. This is accomplished through the collision of the high-energy electrons with molecules of the reactants. The molecules dissociate:

Table 5 Plating baths and their deposition rates

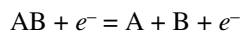
Electroless method	Temperature		pH	Deposition rate	
	°C	°F		μm/h	mil/h
Gold	65–88	149–190	10.0–13.0	2–5	0.08–0.2
Palladium	45–73	113–165	8.0–12.0	2–5	0.08–0.2
Cobalt	85–95	185–203	9.0–11.0	2.5–10	0.1–0.4
Acid nickel	77–93	170–200	4.4–5.2	12.7–25.4	0.5–1
Alkaline nickel	26–95	79–205	8.5–14.0	10–12.7	0.4–0.5
Copper	26–70	79–158	9.0–13.0	1–7.5	0.04–0.3

Source: Ref 7

Table 6 Typical applications of continuous hot-dip-coated steel sheet, wire, and tubing

Coating	Product		
	Sheet	Wire	Tubing
Zinc and Zn-5Al	Roofing, siding, doors, culvert, ductwork, housings, appliances, autobody panels and structural components	Nails, staples, guy wires, strand, tension members, rope, utility wire, fencing	Electrical conduit
Zinc-iron	Autobody panels and structural components
Aluminum type 1	Mufflers, tailpipes, heat shields, ovens, toasters, flues, chimneys	Guy wires, strand, rope, utility wire, fencing	...
Aluminum type 2	Roofing, siding, ductwork, culvert, silo roofs	Guy wires, strand, rope, utility wire, fencing	...
Zn-55Al	Roofing, siding, ductwork, culvert, mufflers, tailpipes, heat shields, ovens, toasters, flues, chimneys, silo roofs
Lead-tin	Fuel tanks, radiator components, valve covers, air-filter housings	...	Fuel lines, hydraulic lines

Source: Ref 8



resulting in the ability of these dissociated elements to become coatings. Because of the small mass of the electron and its high mobility, the reaction rates of PACVD processes can be greater than for conventional CVD.

Laser-Assisted CVD

Although omnidirectional coatings are desirable for many applications, it is oftentimes necessary to mask segments of a component, resulting in complexity and greater expense. With the use of a laser, it becomes possible to control the area of the surface where the coating is applied. Laser sources, operating in either the photolytic or direct pyrolytic mode, can limit the heating needed for reaction to a few microns in depth and can confine the coating area to that area that can be either optically or physically rastered by the laser beam. Laser sources are chosen based on the substrate material, desired coating, wavelength, and energy required to obtain an optimal coating. Traditionally, CO₂ and neodymium-doped yttrium-aluminum-garnet (Nd-YAG) have been extensively used, but increasingly, excimer lasers such as argon-fluoride (Ar-F) as well as combinations of several lasers operating at different wavelengths simultaneously have been used. Bonding of the deposited material is diffusional in nature and similar to conventional CVD processes.

Fluidized-Bed CVD (FBCVD)

Fluidized-bed CVD has been practiced for over 45 years and first reached prominence with the deposition of pyrolytic carbon, niobium, tungsten, aluminum oxide (Al₂O₃) and beryllium oxide (BeO) onto fissionable fuel particles of radioactive uranium and uranium compounds. This process uses an enclosed high-temperature, fluidized-bed coating vessel similar to the fluidized-bed vessels used for heat treating but modified to accommodate expand-

ing exhaust gases at both atmospheric and reduced pressures. A schematic of a FBCVD vessel is shown in Fig. 14.

There are several significant advantages in using FBCVD as a coating method. Components such as bolts, fasteners, bearings, and other items can be coated by FBCVD by immersing them in a powder bed whose bulk density is close to that of the components. The bulk density of the bed material can be adjusted across an extreme range (1.8 to 19.4 g/cm³, or 112 to 1210 lb/ft³). Therefore, large quantities of components can circulate through the fluidized bed, with sizes that range from 25 µm to 3 mm (1 × 10⁻³ to 0.125 in.). The lower limit is related to the ability of the system to separate particles from the gas stream, so that the powder is maintained in the coating vessel. The upper limit is more dependent on the fluidization gas velocity required to suspend larger components.

By injecting reactive gases into the carrier-gas stream, coating occurs on all heated surfaces exposed to the flowing gases within the fluidized-bed vessel. In this manner, all the particles contained within the moving bed are individually encapsulated with a coating of uniform thickness that conforms to the contours of each component. Controlled uniform compositions are formed in a reproducible manner. Multiple-layer coatings can be deposited, with each layer composed of a different material.

Physical Vapor Deposition (PVD)

Physical vapor deposition technology includes techniques such as evaporation, ion plating, and sputtering. It is used to deposit thin films as well as thick films and even freestanding components. With these processes, virtually any solid material, with the exception of certain polymers, may be deposited onto any other solid surface. The PVD methods are line-of-sight, vacuum-coating processes operating at pressures ranging from 13.33 to 1.33 × 10⁻⁶ Pa (10⁻¹ to 10⁻⁸ torr). Both amorphous and crystalline microstructures can be produced in a controlled manner at temperatures low

Table 7 Chemical vapor deposition (CVD) methods and characteristics

CVD method	Most distinguishing characteristic	Range of typical substrate temperatures		Range of typical deposition pressures
		°C	°F	
Conventional CVD	Omnidirectional coating of a wide variety of shapes and sizes	350–1600	660–2910	100 torr–1 atm
Metal-organic CVD	Lowered deposition temperatures using metal-organic precursor	200–800	390–1470	10 ⁻³ torr–1 atm
Plasma-assisted CVD	Reduced substrate temperature	200–800	390–1470	10 ⁻³ –10 torr
Laser CVD	Coat localized areas on a surface without bulk heating	200–1000	390–1830	100 torr–1 atm
Packed-bed CVD	Alloy/conversion coatings in powder packed-bed environment	500–1200	930–2190	100 torr–1 atm
Fluidized-bed CVD	Coat powders and small components	350–1600	660–2910	100 torr–1 atm
Continuous fiber coating	Deposit coatings on mono-filament and multifilament tows	350–1600	660–2910	100 torr–1 atm
Chemical vapor infiltration	Coat internal surfaces of porous media	350–1600	660–2910	5 torr–1 atm

enough to prevent distortion or microstructural changes during the coating process.

Evaporation

In the evaporation process (Fig. 15, 16), vapors are produced by heating either by direct resistance, radiation, eddy current, electron beam, laser beam, or an arc discharge. The evaporated atoms undergo an essentially collisionless line-of-sight transport prior to condensing on the surface of the component.

The rate of evaporation is a function of the vapor pressure of a material and the ambient pressure. Table 8 indicates the various temperatures at which certain metals have a vapor pressure of 1.33 Pa (10^{-2} torr).

Direct evaporation of ceramic materials is difficult because of their low vapor pressure and their tendency to dissociate. Reactive

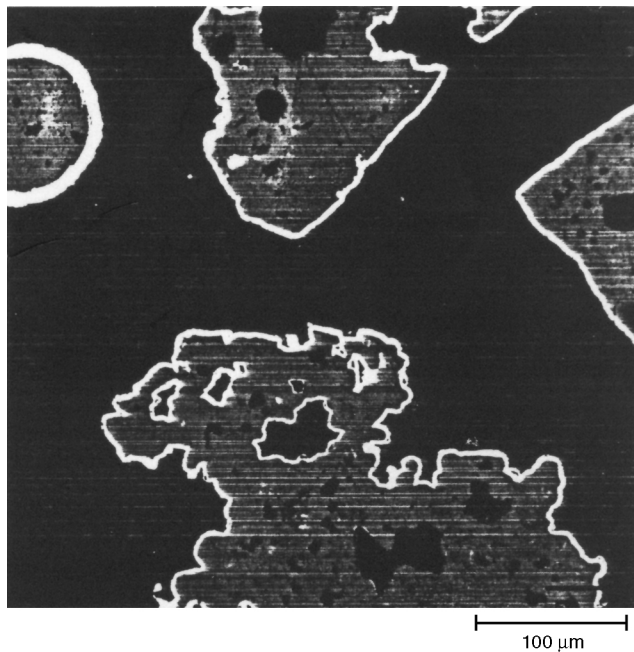


Fig. 12 A chemical vapor deposition coating on a porous surface. 200×

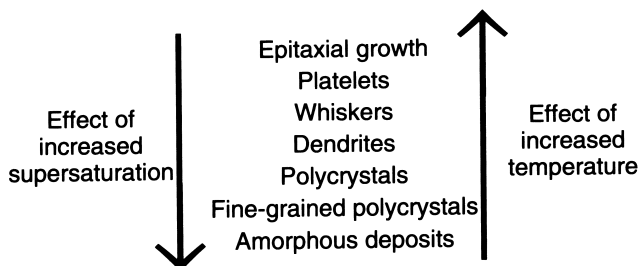


Fig. 13 General effect of temperature and supersaturation on coating microstructure. Source: Adapted from Ref 9

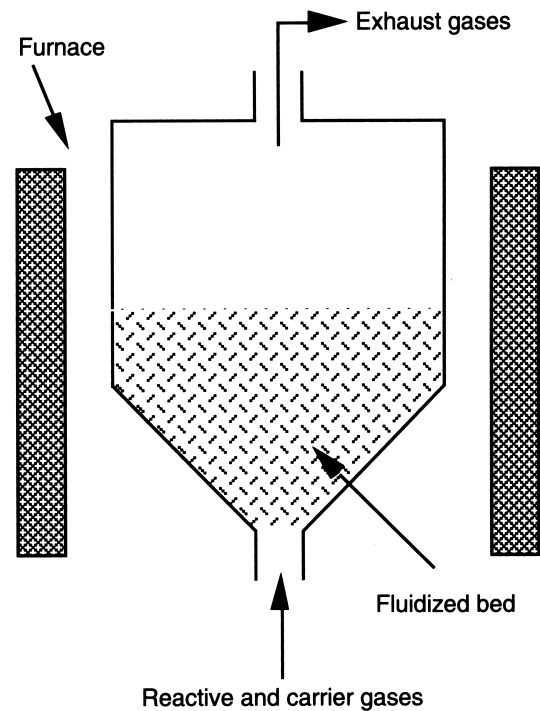


Fig. 14 Example of a fluidized bed chemical vapor deposition apparatus

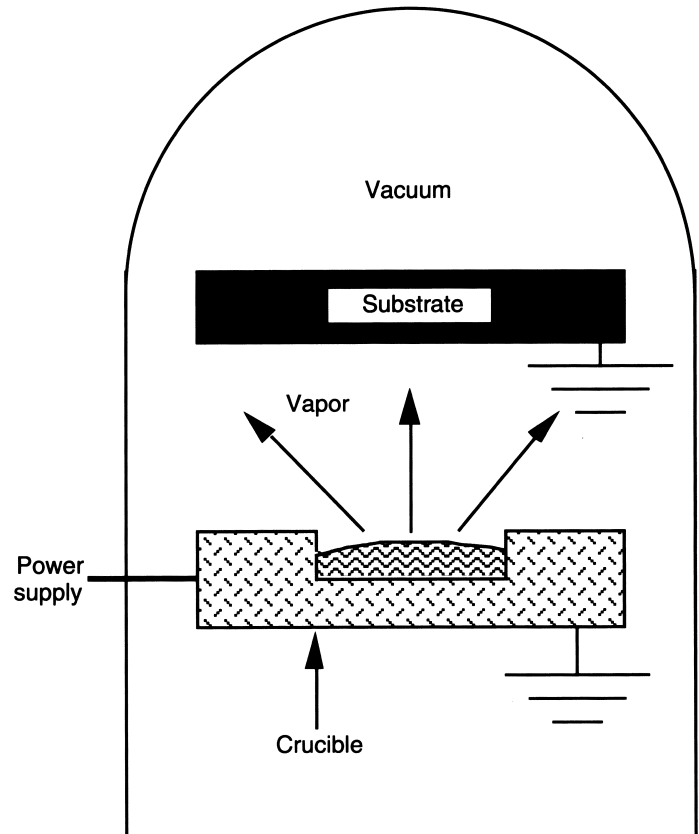


Fig. 15 Resistance-heated evaporation process

methods are therefore employed to produce coatings of carbides, nitrides, or oxides. In this process, an appropriate gas such as oxygen or nitrogen is introduced into the region where the metal is being evaporated. The reaction to produce the desired coating occurs on the surface of the component. Nonuniform thicknesses can be a concern with evaporative processes and generally require movement of the surface with respect to the source. Applications of evaporative coatings include optical coatings for abrasion resistance, reduction of glare, and decorative purposes. Additionally, MCrAlY coatings are applied to gas turbine blades and vanes to increase resistance to sulfidization and oxidation.

Sputtering

Sputtering is a process in which material is removed from the surface of a material through momentum exchange associated with the surface being bombarded by energetic particles (Fig. 17). The bombarding species are generally ions of a heavy inert gas such as argon, generated by a glow discharge with a pressure between 0.133 and 13.33 Pa (10^{-3} and 10^{-1} torr) and an applied

voltage of 500 V direct current (dc). Because the coating material is passed into the vapor phase by mechanical rather than chemical or thermal processes, virtually any material can be used for sputtered coatings.

As with evaporative methods, ceramics can be deposited by sputtering techniques, with the addition of reactive gases and close control of the process. Variations on the aforementioned basic process can result in coatings formed by:

- Employing a target that is a mosaic of several materials
- Employing multiple targets simultaneously, of identical or different materials
- Employing multiple targets sequentially to create a layered coating
- Biasing the substrate as an electrode to cause surface ion bombardment during deposition, in order to remove loosely bonded contamination or to modify the structure of the resulting coating. This is known as bias sputtering.
- Employing a gas to introduce one of the coating materials into the chamber. This process is known as reactive sputtering. It

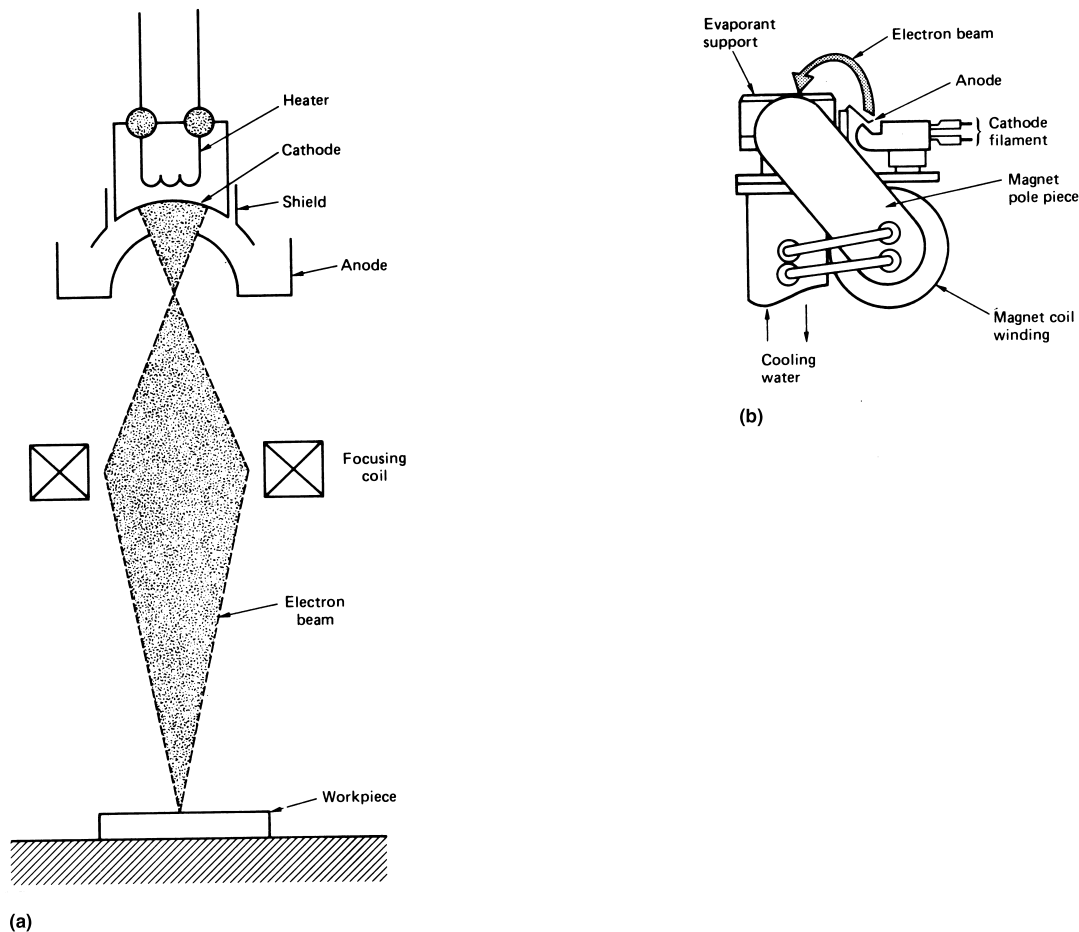


Fig. 16 Electron-beam-heated evaporation process. (a) Linear focusing gun. (b) Bent-beam electron gun with water-cooled evaporant support

permits metal targets and dc power supplies to be used for preparing coatings of nonconducting compounds.

- The use of balanced or unbalanced magnetrons

Ion Plating

Ion plating is the generic term applied to atomistic film deposition processes in which the substrate surface and/or the depositing film are subjected to a flux of high-energy particles sufficient to cause changes in the interfacial region or film properties, compared to the non-bombarded deposition. Such changes may be in the adhesion of the film to the substrate, film morphology, film density, film stress, or the coverage of the surface by the deposit-

ing film material. Ion plating (Fig. 18) is usually performed in argon at a pressure of approximately 1.33 Pa (10^{-2} torr). A negative potential in the range of 2 to 5 kV is applied to the workpiece so that it becomes the cathode of a glow discharge formed between it and the grounded parts of the apparatus. The resulting argon ion bombardment initially cleans the surface and continues while the coating material is evaporated into the plasma and deposited onto the substrate. This continuing bombardment increases nucleation and improves adhesion. Gas scattering is the major factor in ensuring uniform coating distribution. Applications of ion plating include optical coatings on glass and plastic, coating of electrical contacts, and lubricious metals.

Ion Implantation

Ion implantation is a process conducted in a vacuum (133×10^{-6} to 1.33×10^{-3} Pa, or 10^{-6} to 10^{-5} torr), wherein a beam of ions is directed and accelerated toward the substrate. Typically, the ions are accelerated to an energy of 100 keV. The ions penetrate to an average depth of 0.1 μm (4×10^{-6} in.) and a maximum depth of 0.25 μm (1×10^{-4} in.). Table 9 lists applications of ion implantation.

Thermal Spray Processes

Thermal spray is one of many methods of applying coatings for the protection of materials in harsh environments. Thermal spraying has historically been the process associated with the application of thick coatings whose bulk material properties allow the

Table 8 Temperature at which selected metals exhibit a vapor pressure of 1.33 Pa (10^{-2} torr)

Metal	Temperature	
	$^{\circ}\text{C}$	$^{\circ}\text{F}$
Cadmium	265	509
Zinc	345	653
Lead	715	1319
Silver	1030	1886
Aluminum	1220	2228
Copper	1260	2300
Gold	1400	2552
Chromium	1400	2552
Iron	1480	2696
Cobalt	1520	2768
Nickel	1530	2786
Titanium	1740	3164
Hafnium	2400	4352
Molybdenum	2530	4586
Tungsten	3230	5846

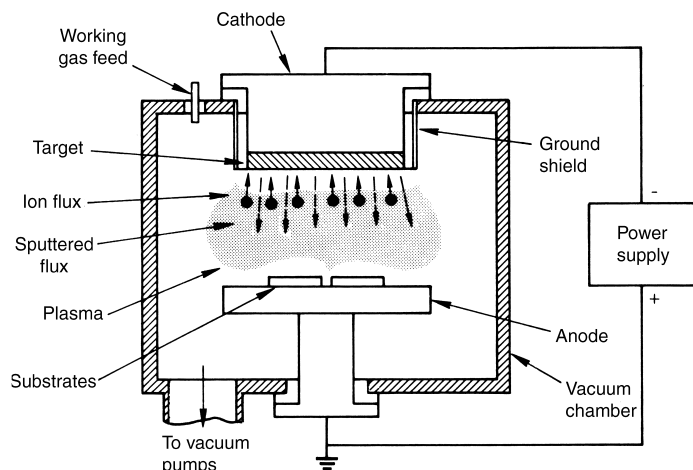


Fig. 17 Typical sputter coating process

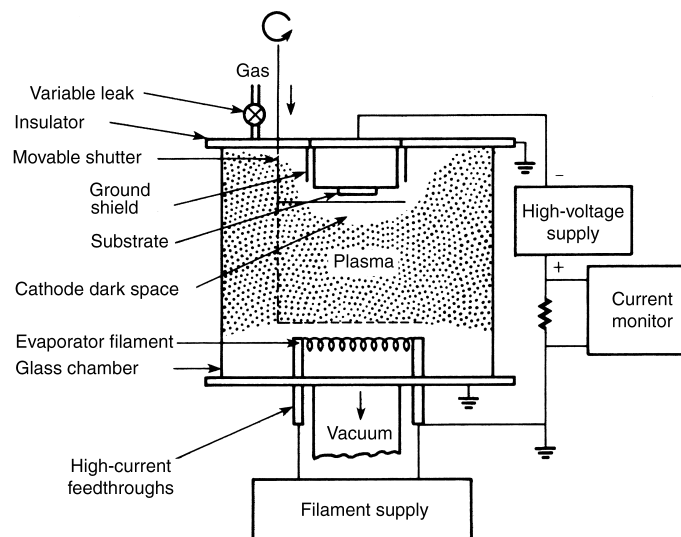


Fig. 18 Schematic representation of an ion plating process

substrate to perform in a manner beyond the capabilities of its uncoated state.

Typical coatings developed for wear and corrosion applications include metals, oxides, ceramics, cermets, nitrides, and carbides. The desire to apply coatings with diverse thermophysical properties has resulted in a diverse family of processes sharing the same basic elements.

Figure 19 illustrates a generic source of heat used to generate a region of high enthalpy (a measure of heat). Material, in powder, wire, or rod form, is injected into this region, and droplets form in either a molten or plastic state. These droplets are accelerated toward a prepared substrate, where they impact and bond to form a protective coating.

The application of thermal spray coatings is a line-of-sight process, and therefore, there is some restriction on the shape of the component being coated, and the impact of the sprayed material should be as close to 90° as possible.

Some of the limitations of the thermal spray process, in general, are the relatively low bond strengths, the amount of porosity within the coatings, the thickness of the coatings, as well as its inability to coat blind holes.

The structure of thermal sprayed coatings is lamellar, consisting of “splats,” which are particles flattened by impact with the surface and then rapidly quenched. Sprayed metals are usually harder than the corresponding wrought metal, due mainly to the inclusion of dispersed oxides formed during the spraying process. They are also less ductile, because of porosity and hardness associated with the spray process. Figure 20 represents the hierarchy of processes associated with thermal spray and is grouped by the method of heat generation, that is, combustion, electric arc, or plasma. A

more detailed discussion of thermal spray processes is presented in subsequent articles in this Handbook.

Hardfacing

Hardfacing, also known as weld overlay, is a family of processes that weld the protective coating to the surface to be protected. Unlike thermal spray processes, hardfacing requires that the surface be melted to form a physical, chemical, and metallurgical interface. As a result, hardfacing is done on larger components that are not deformed by the heat of application. Furthermore, the interface exhibits a heat-affected zone in which the properties of the surface are altered and also includes regions where the surface material and the applied material are both present. The principal application of hardfacing is for the rebuilding of worn components or in the manufacturing of new equipment where large amounts of wear need to be tolerated. The materials to be applied are either in powder or wire form, and the welding is carried out using either combustion or electric-arc processes. Table 10 lists hardfacing coating characteristics.

Oxyacetylene Weld Overlays

This technique, derived from the welding industry, is similar to brazing in that a flux is used to ensure adhesion for ferrous materials. The surface to be coated is heated, and a metallic filler rod is melted by an oxygen/acetylene flame (Fig. 21a). Where alloys are not available in rod or wire form but are available in powder form, a variation of the oxyacetylene process, called the powder welding

Table 9 Applications of ion implantation in the tooling industry

Tool	Material	Implanted species	Factor of improvement
Injection molding tool for plastics	Chromium-plated steel	Nitrogen	10
Injection screw for plastics	Nitrided steel	Nitrogen	10–15
Feed wear pads and sprue bushes machinery	Steel	Nitrogen	18
Punch/die for motor laminations	Steel and cemented carbide	Nitrogen	6
Drills for foil-coated laminate	Cemented carbide	Nitrogen	2–3
Tools for metal extrusion	Steel, stellite, cemented carbide	Nitrogen	3
Ring press tool for steel	Steel	Nitrogen	10
Forming tool for steel	Carburized mild steel	Nitrogen	2–3
Hot rolls for nonferrous rod	H13 steel	Nitrogen	5
Dies for copper rod	Cemented carbide	Carbon	5
Ring cutter for tinplate	Steel	Nitrogen	3
Gear cutter	Steel	Nitrogen	3
Thread-cutting dies	M2 steel	Nitrogen	5
Swaging dies for steel	Cemented carbide	Nitrogen	2
Wire-drawing dies	Cemented carbide	Carbon	3
Slitters for synthetic rubber	Cemented carbide	Nitrogen	12
Slitters for paper	1.6% Cr, 1% C steel	Nitrogen	2

Source: Ref 10

method, is used. In this process, the powder is aspirated into the fuel gas (Fig. 21b).

Tungsten Inert Gas (TIG) Weld Overlays

Figure 22 illustrates the TIG process. An electric arc is struck between the workpiece and a nonconsumable tungsten electrode. To prevent oxidation of the workpiece and coating, an inert gas is used to shield the area of coating application. Because this is an electric arc process, the available temperatures are higher than those of oxyacetylene combustion, and therefore, a larger selection of alloys is available for hardfacing.

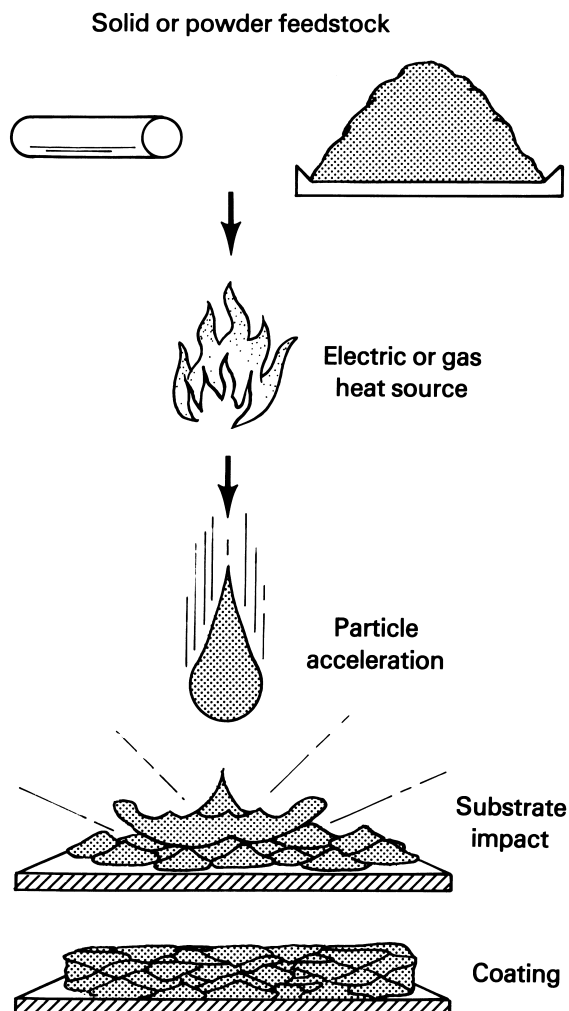


Fig. 19 Generic thermal spray process. Source: Adapted from TAFA, a division of Praxair Surface Technologies Inc.

Shielded Metal Arc Welding (SMAW)

The SMAW method (Fig. 23) is a manual arc-welding process in which the heat necessary to form the weld overlay is generated by an electric arc struck between a consumable rod electrode and the workpiece. The electrode is covered with a flux that undergoes combustion and decomposition, generating a gas shield that surrounds the electrode tip, molten weld pool, arc, and adjacent area of the workpiece. This gas shield provides protection from the effects of the surrounding atmosphere. Additional protection is provided by a film of flux floating on the surface of the weld pool. Filler metal is supplied by the core of the consumable electrode and from metal powder mixed with the covering of some electrodes.

Flux-Cored Arc Welding (FCAW)

In the FCAW process, heat necessary for the weld overlay is generated by an electric arc struck between a tubular, consumable rod electrode and the workpiece (Fig. 24). Shielding of the electrode tip, molten weld pool, arc, and adjacent areas is provided by the combustion and decomposition of a flux contained in the tubular electrode. A second form of FCAW incorporates an auxiliary shielding gas, such as argon or carbon dioxide, to provide additional protection from the surrounding environment.

Metal Inert Gas (MIG) Weld Overlays

In the MIG process, the heat necessary for the weld overlay is generated by an electric arc struck between a consumable electrode and the workpiece (Fig. 25). Shielding of the surface from the surrounding environment is provided by a jet of inert gases fed through the welding gun. The consumable electrode is made of the desired filler material and forms the weld overlay as it is consumed. Unlike SMAW and FCAW, a continuously fed metal wire can be used. This process is basically the FCAW process with the addition of a shielding gas such as argon or carbon dioxide.

Submerged Arc Welding

In this process (Fig. 26), the protective gas is replaced by a granular flux that surrounds the arc and covers the molten pool. In addition to providing protection from oxidation, the flux, after solidification, thermally insulates the coating and thus allows it to cool more slowly. The alloy content of the deposit is controlled by the composition of the filler rod and by metallic additions made to the flux. The filler material may be in the form of wires, rods, tubes, or strip. The need to keep the powdered flux in place means that the process must be operated in the gravity-fed mode onto flat or rotated cylindrical surfaces.

Plasma-Transferred Arc (PTA) Weld Overlays

The PTA process is often considered to be a thermal spray process, but historically, it is a weld overlay process. In this process (Fig. 27), an electric arc is struck between the workpiece

and a tungsten cathode, creating a dc electric arc. This arc is confined in a restricting orifice, and a plasma is formed as the gas flowing through an annular orifice is ionized. The material to be coated is in powder form and is fed into the ionized gas stream. Because the arc is rooted on the work, a diffusion bond layer is generated at the interfaces that consists of both the substrate material and the coating material. A protective auxiliary shielding gas can be used if needed. A second dc power supply is connected between the workpiece and the restricting orifice to help ignite the initial arc and add additional energy to the electric arc.

Comparison of Major Coating Methods

Table 11 summarizes the characteristics of four major coating methods: electro/electroless plating (EP/ElsP), chemical vapor deposition (CVD), physical vapor deposition (PVD), and thermal spray (TS). Both the initial equipment costs and the operating costs for EP/ElsP coatings are relatively low. However, the by-products are considered highly toxic and are subject to increasingly stricter government regulation, thereby making storage, reclamation, and disposal major economic concerns. Thermal

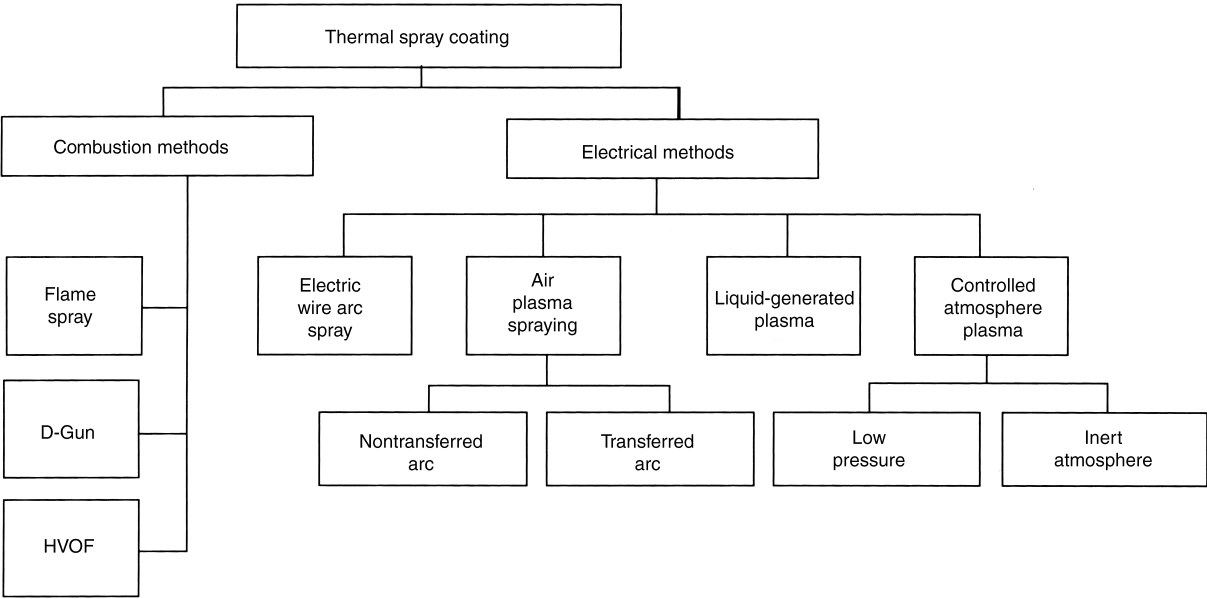


Fig. 20 Hierarchy of thermal spray processes. D-Gun, detonation gun (Praxair Surface Technologies); HVOF, high-velocity oxyfuel

Table 10 Hardfacing materials in order of increasing abrasion resistance and decreasing toughness

Material type	Properties of deposits	Type of duty for which deposits are most appropriate
Austenitic manganese steels Martensitic and high-speed steels	Tough, crack resistant, and soft. Ability to work harden Good combination of abrasion and impact resistance. Abrasion resistance increases with carbon and chromium content at expense of impact resistance.	High stress, heavy impact Nonlubricated metal-to-metal wear
Nickel- and cobalt-base alloys	Wear, corrosion, and heat resistant, with good all-around strength but low ductility	Abrasive conditions accompanied by high temperature and/or corrosion
Martensitic and high-chromium iron Tungsten-carbide composites	Excellent resistance to wear by most minerals. Brittle Extreme resistance to sliding abrasion by hard minerals. Worn surfaces become rough.	Highly abrasive conditions Extremely abrasive conditions where extra cost is warranted

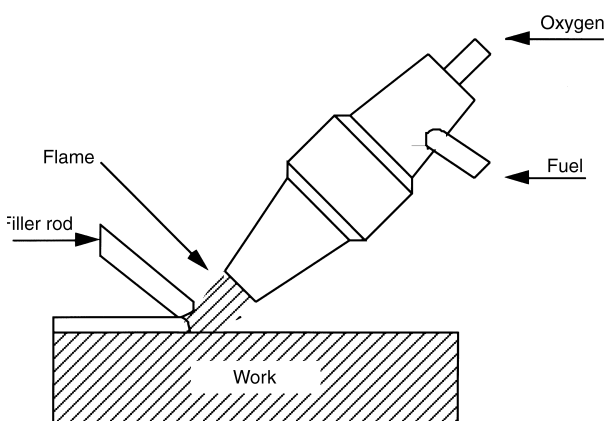
Source: Ref 2

spray equipment, and therefore its cost, varies widely from simple combustion devices to computer-controlled low-pressure plasma spray systems.

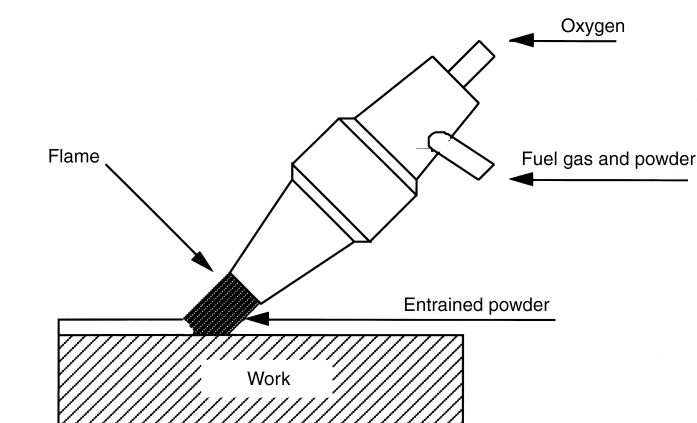
The operating costs are very much dependent on the cost of consumables such as powder, wire, or rod materials as well as the quantities and types of gases used. Equipment costs for CVD are moderate, with the understanding that neutralization of the output gases is an integral part of any system. The operating costs of CVD are dominated by precursor gas costs and the frequent need to clean the systems. The cost of PVD coating equipment is very high, due to the need to maintain high vacuums in chambers of

sufficient volume to make the process cost-effective. Operating costs are associated with the degree of surface cleanliness necessary for coating adhesion, as well as the target costs.

The EP/EIcP and CVD are omnidirectional coating processes, while TS and PVD are line-of-sight processes. Adherence for TS coatings, in general, is provided by mechanical bonding.



(a)



(b)

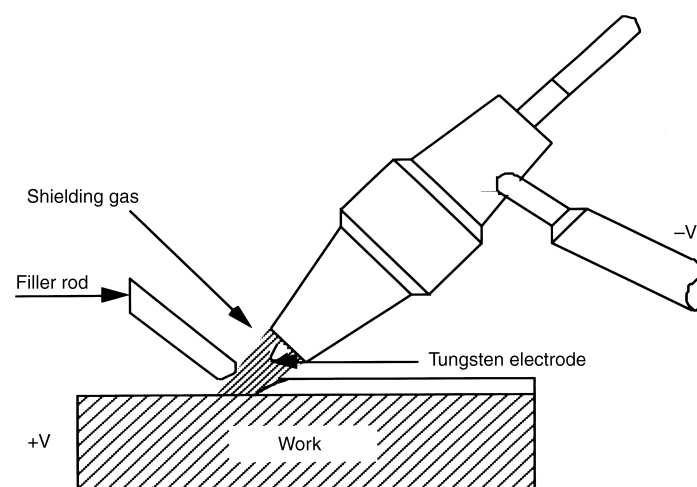


Fig. 22 Tungsten inert gas weld overlay process

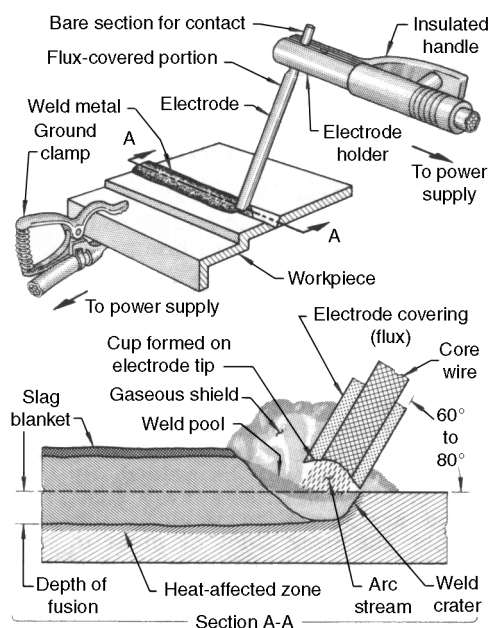


Fig. 23 Shielded metal arc welding process

Fig. 21 Oxyacetylene weld overlay processes. (a) Rod or wire. (b) Powder

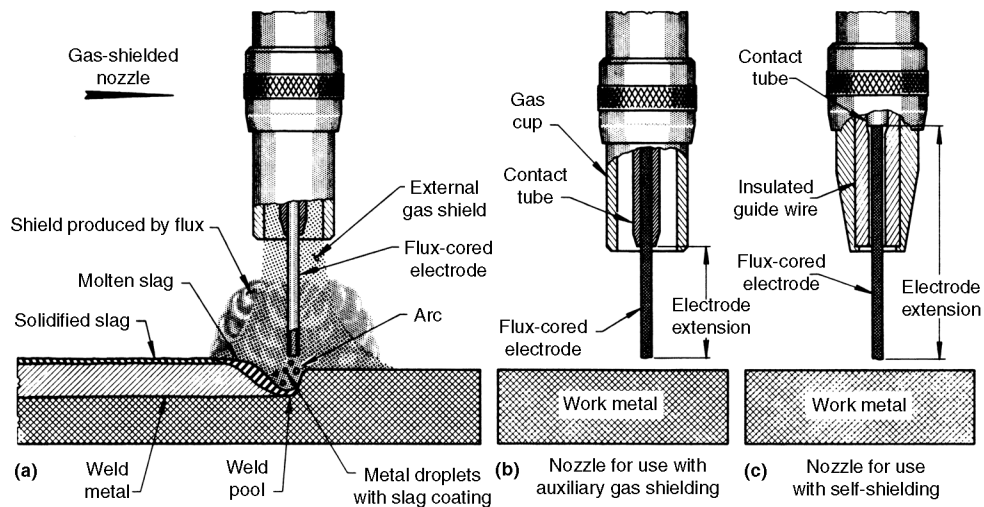


Fig. 24 Flux-cored arc welding process

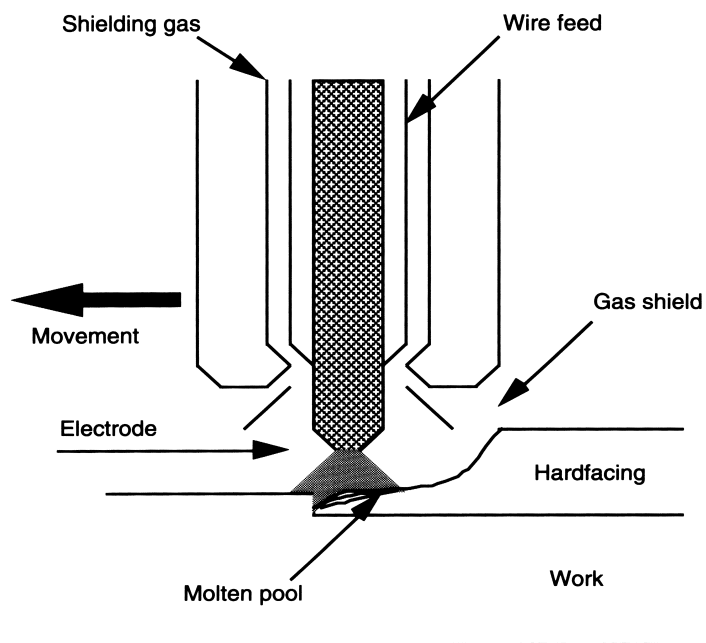


Fig. 25 Metal inert gas weld overlay process

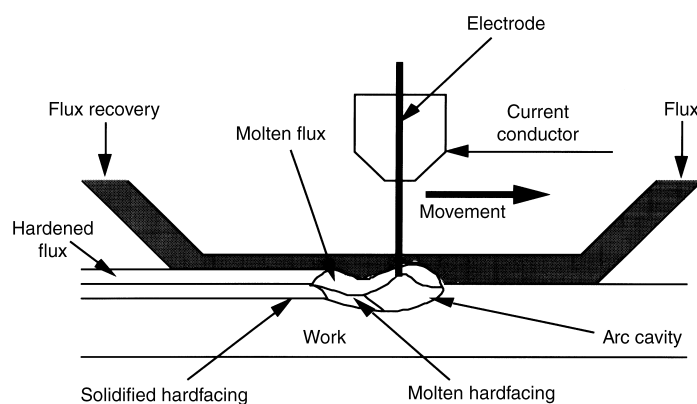


Fig. 26 Submerged arc welding process

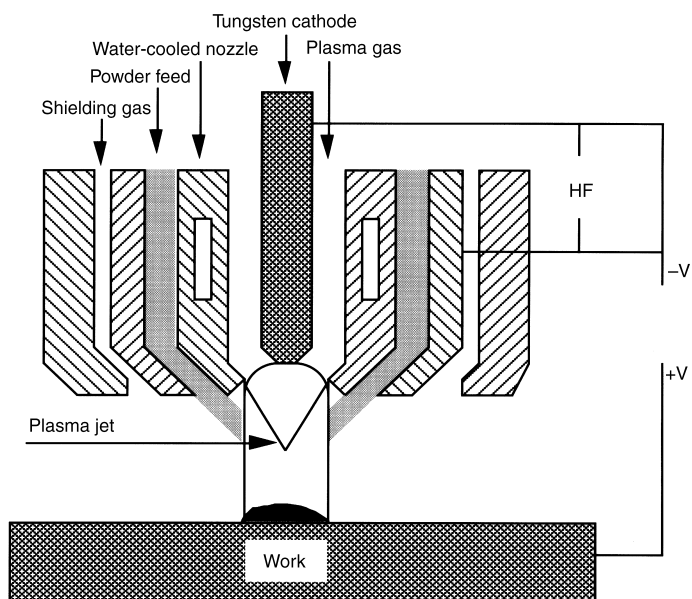


Fig. 27 Plasma-transferred arc weld overlay process

Table 11 General characteristics of major coating methods

Characteristic	Electro/electroless plating	Thermal spray	Chemical vapor deposition	Physical vapor deposition
Equipment cost	Low	Low to moderate	Moderate	Moderate to high
Operating cost	Low	Low to moderate	Low to moderate	Moderate to high
Process environment	Aqueous solution	Atmospheric to soft vacuum	Atmospheric to medium vacuum	Hard vacuum
Coating geometry	Omnidirectional	Line of sight	Omnidirectional	Line of sight
Coating thickness	Moderate to thick, 10 μm –mm	Thick, 50 μm –cm	Thin to thick, 0.1 μm –mm	Very thin to moderate,
Substrate temperature	Low	Low to moderate	Moderate to high	Low
Adherence	Moderate mechanical bond to very good chemical bond	Good mechanical bond	Very good chemical bond to excellent diffusion bond	Moderate mechanical bond to good chemical bond
Surface finish	Moderately coarse to glossy	Coarse to smooth	Smooth to glossy	Smooth to high gloss
Coating materials	Metals	Powder/wire, polymers, metals/ceramics	Metals, ceramics, polymers	Metals, ceramics, polymers

Adherence of EP/ElsP and PVD coatings is, in general, provided by mechanical and/or chemical bonding. Adherence for CVD coatings is provided by either chemical and/or diffusion bonding.

With the exception of EP/ElsP, which is limited to metals and some alloys, the TS, CVD, and PVD coating processes can apply a variety of metals, ceramics, cermets, and polymers.

REFERENCES

1. M.N. Kruschov, "Resistance of Metals to Wear by Abrasion as Related to Hardness," Conference on Lubrication and Wear, Institution of Mechanical Engineers, London, 1957
2. *Wear Resistant Surfaces in Engineering*, Her Britannic Majesty's Stationery Office, London, 1986
3. *Heat Treatment of Metals*, Wolfson Heat Treatment Centre, Aston University, England
4. S. Lampman, Introduction to Surface Hardening of Steels, *Heat Treating*, Vol 4, *ASM Handbook*, ASM International, 1991, p 259–267
5. U.S. Environmental Protection Agency, <http://www.epa.gov> (accessed 4/9/04)
6. H. Geduld, Zinc Plating, *Metal Finishing Guidebook and Directory*, Elsevier Science Publishing, 1990, p 326
7. J. Henry, Electroless (Autocatalytic, Chemical) Plating, *Metal Finishing Guidebook and Directory*, Elsevier Science Publishing, 1990, p 368–370
8. H.E. Townsend, Continuous Hot Dip Coatings, *Surface Engineering*, Vol 5, *ASM Handbook*, ASM International, 1994, p 340
9. F. Bunshah, *Deposition Techniques for Film and Coatings*, Noyes Publications, 1982, p 357
10. G. Dearnaley, Practical Applications of Ion Implantation, *J. Met.*, Vol 34 (No. 9), Sept 1982, p 18–28

SELECTED REFERENCES

- C.C. Berndt, Ed., *Thermal Spray: Surface Engineering via Applied Research, Proceedings of the First International Thermal Spray Conference*, 9–11 May 2000 (Montreal, Quebec, Canada), ASM International, 2000
- C.C. Berndt, K.A. Khor, and E.F. Lugscheider, Ed., *Thermal Spray 2001: New Surfaces for a New Millennium, Proceedings of the ITSC 2001*, ASM International, 2001
- R.F. Bunshah et al., *Deposition Technologies for Films and Coatings*, Noyes Publications, 1982
- J.R. Davis, Ed., *Surface Engineering for Corrosion and Wear Resistance*, ASM International, 2001
- M.G. Fontana, *Corrosion Engineering*, 3rd ed., McGraw-Hill, 1986
- S. Grainger and J. Blunt, *Engineering Coatings: Design and Application*, 2nd ed., Woodhead Publishing Ltd., England, 1999
- F.A. Lowenheim, *Electroplating*, McGraw-Hill, 1978
- *Materials Characterization*, Vol 10, *ASM Handbook*, American Society for Metals, 1986
- C.F. Powell, J.H. Oxley, and J.M. Blocher, Ed., *Vapor Deposition*, John Wiley and Sons, 1966
- E.E. Stansbury and R.A. Buchanan, *Fundamentals of Electrochemical Corrosion*, ASM International, 2000
- *Surface Engineering*, Vol 5, *ASM Handbook*, ASM International, 1994
- *Welding, Brazing, and Soldering*, Vol 6, *ASM Handbook*, ASM International, 1993

Guide to General Information Sources

THIS ARTICLE is a brief guide to information sources on thermal spray technology. It is by no means an all-inclusive review of information sources; rather, it is intended to serve as a starting point for readers interested in sources of information that may fall outside the scope of this Handbook. Emphasis has been placed on specifications and standards for thermal spray processing, materials, testing, and characterization.

ASM International and the Thermal Spray Society

ASM International and its affiliate society, the Thermal Spray Society (TSS), offer a wide range of products and services to the thermal spray community. These include magazines and journals; reference books, including the *ASM Handbook* series; conference proceedings; newsletters; education courses; and videos. Some prominent examples of these are discussed subsequently. A complete listing of all thermal spray products and services can be found at the TSS Web site (Ref 1).

The TSS was formed in June of 1994. It was previously a technical division of ASM International. Memberships in the TSS are individual memberships versus corporate or company memberships.

Journal of Thermal Spray Technology (JTST). The *JTST* began publication in March 1992. A quarterly publication, it features critically reviewed scientific papers and engineering articles that cover all fundamental research and practical aspects of thermal spray science, including equipment, processing, feedstock manufacture, and testing and characterization.

SPRAYTIME is a quarterly newsletter published jointly by the TSS and the International Thermal Spray Association (ITSA), an independent trade association (membership to ITSA is a corporate or company membership). The newsletter provides news of interest to the thermal spray community.

Advanced Materials & Processes (AM&P). The monthly flagship publication of ASM International, *AM&P* routinely publishes newsbriefs and articles related to thermal spray technology. It also features a thermal spray “focus” issue once a year. A *Thermal Spray Buyers Guide* has also been published in *AM&P* (see the subsequent discussion).

Reference Books. ASM International has published many books over the years that deal with surface engineering and that contain information related to thermal spray technology. Several recently published books in this area are listed in Ref 2 to 4.

ASM Handbook Series. A number of excellent reviews of thermal spray processing and thermal spray materials and their properties have been published in *ASM Handbook*. These can be found in Volumes 5, 6, 7, 13A, and 18 of *ASM Handbook* (Ref 5–9).

Safety Guidelines. As of 2004, two safety guidelines have been prepared by the TSS Safety Guidelines Committee and are available as Adobe portable document format (PDF) files at the

TSS Web site. “Safety Guidelines for Risk Assessments” describes the necessary steps involved in identifying, documenting, and implementing the necessary controls to minimize or eliminate the potential of risk in the workplace. “Safety Guidelines for the Handling and Use of Gases in Thermal Spraying” provides practical recommendations for the safe installation, operation, and maintenance of gas equipment used in thermal spraying.

Accepted Practices. As of 2004, the TSS Accepted Practice Committee on Evaluation of Mechanical Properties of Thermal Spray Coatings has published one accepted practice, “Modified Layer Removal Method for Evaluating Residual Stresses in Thermal Spray Coatings.” This document is also available as a PDF file at the TSS Web site. It describes specimen preparation and specimen dimensions, the equipment needed, strain gage application, the coating layer removal procedure, and the method for interpreting the data to evaluate residual stresses.

Terminology and Company Origins. The TSS published *Thermal Spray Terminology and Company Origins* (Ref 10) in 2001. The comprehensive set of terms and definitions in this document is the basis for the “Glossary of Terms” in this Handbook. The publication also features a section that traces the origins of thermal spray manufacturers to their beginnings. Acquisitions, amalgamations, and mergers for several major companies are laid out in a “family tree” arrangement.

Thermal Spray Buyers Guide. Available online at the TSS Web site, the buyers guide lists hundreds of companies in the thermal spray industry. It is searchable by category (for example, ceramic rods, powders, wires, equipment, services, masking, and testing) or by company name. The *Thermal Spray Buyers Guide* has also been published in printed form (see the earlier discussion on *AM&P* magazine).

Specifications, Standards, and Quality Control for Coatings

Specifications and Guidelines for Thermal Spray Applications. Several professional and industrial organizations in the United States have developed guidelines with the intent of assisting those people who need to specify coatings for a particular application as well as helping practical thermal spray operators accomplish a specific task. The listing that follows indicates the organization that generated the particular document as well as a very brief description of the content of the document. The organizations discussed include the American Welding Society (AWS); The Society for Protective Coatings (SSPC), formerly the Steel Structures Painting Council; and NACE International, formerly the National Association of Corrosion Engineers:

- AWS C2.16-92, “Guide for Thermal Spray Operator Qualification.” This guide recommends thermal spray operator quali-

fication procedures. It covers applicable documents relating to thermal spray equipment, consumables, and safety. It also contains operator qualification and coating system analysis forms.

- AWS C2.18-93, "Guide for the Protection of Steel with Thermal Sprayed Coatings of Aluminum and Zinc and Their Alloys and Composites" (30 pages). This guide sets forth recommended thermal spray operator qualification procedures. It covers applicable documents relating to thermal spray equipment consumables and safety. It also contains operator qualification and coating analysis forms.
- AWS C2.23, SSPC-CS 23.00, NACE No. 12, "Specification for the Application of Thermal Spray Coatings (Metallizing) of Aluminum, Zinc, and Their Alloys and Composites for the Corrosion Protection of Steel" (41 pages). This 2004 standard states requirements for equipment, application procedures, and in-process quality-control checkpoints for thermal spray coatings. It is meant to be used by facility owners, fabricators, engineers, and others who specify or contract the application of thermal spray coatings for the preservation and maintenance of steel structures. It presents a fill-in-the-blanks model procurement specification and provides an illustrated overview of the thermal spray coating process. It also explains and illustrates nine key production and quality-control checkpoints for applying thermal spray coatings. This standard may be used as a procurement document and may also be used for thermal spray coating inspectors and applicators to develop and maintain applications procedures, equipment inventory, and operator training programs.
- AWS C2.20-2003, "Specification for Thermal Spraying Zinc Anodes on Steel-Reinforced Concrete" (40 pages). This AWS standard is a specification for thermal spraying zinc anodes on

steel-reinforced concrete. The scope includes job description, safety, pass/fail job reference standards, feedstock materials, equipment, a step-by-step process instruction for surface preparation, thermal spraying, and quality control. There are three annexes: job control record, operator qualification, and portable adhesion testing.

- AWS C2.21-2003, "Specification for Thermal Spray Equipment Acceptance Inspection" (25 pages). This standard specifies the thermal spray equipment acceptance requirements for plasma, arc wire, flame powder, wire, rod, cord, and high-velocity oxyfuel equipment. The equipment supplier shall provide proof of suitability. Example inspection reports are provided in four appendixes.
- AWS C2.5-2003, "Specification for Solid and Composite Wires and Ceramic Rods for Thermal Spraying" (26 pages). This specification defines the as-manufactured, chemical-composition classification requirements for solid and composite wires and ceramic rods for thermal spraying. Requirements for standard sizes, marking, manufacturing, and packaging are included. The specification lists 100 materials.

Standards for Coatings. A great challenge for thermal spray coatings is their specification and standardization. Much of the history of thermal spray coatings has relied on internal company specifications so that reproducible coatings might be produced, and the outgrowth of such documentation now constitutes accepted practice (Ref 8). With regard to testing practices, the standard methods developed by ASTM International (or equivalent organizations) are a good basis on which to recommend procedures that rank coatings or different materials. Table 1 lists ASTM standards for wear and corrosion testing that are appropri-

Table 1 ASTM International wear and corrosion test standards applicable to thermal spray coating

ASTM designation	Title
Corrosion	
B 117-95	Practice for Operating Salt Spray (Fog) Testing Apparatus
G 3-89 (1994)	Practice for Conventions Applicable to Electrochemical Measurements in Corrosion Testing
G 5-94	Reference Test Method for Making Potentiostatic and Potentiodynamic Anodic Polarization Measurements
G 15-97	Terminology Relating to Corrosion and Corrosion Testing
G 78-95	Guide for Crevice Corrosion Testing of Iron-Base and Nickel-Base Stainless Alloys in Seawater and Other Chloride-Containing Aqueous Environments
G 85-94	Practice for Modified Salt Spray (Fog) Testing
Wear	
G 40-96	Terminology Relating to Wear and Erosion
G 65-94	Test Method for Measuring Abrasion Using the Dry Sand/Rubber Wheel Apparatus
G 73-93	Practice for Liquid Impingement Erosion Testing
G 76-95	Test Method for Conducting Erosion Tests by Solid Particle Impingement Using Gas Jets
G 77-93	Test Method for Ranking Resistance of Materials to Sliding Wear Using Block-on-Ring Wear Test
G 81-97	Practice for Jaw Crusher Gouging Abrasion Test
G 98-91 (1996)	Test Method for Galling Resistance of Materials
G 99-95	Test Method for Wear Testing with a Pin-on-Disk Apparatus
G 105-89	Test Method for Conducting Wet Sand/Rubber Wheel Abrasion Tests
G 115-93	Guide for Measuring and Reporting Friction Coefficients
G 119-93	Guide for Determining Synergism Between Wear and Corrosion
G 132-96	Test Method for Pin Abrasion Testing
G 133-95	Test Method for Linearly Reciprocating Ball-on-Flat Sliding Wear
G 134-95	Test Method for Erosion of Solid Materials by a Cavitating Liquid Jet

ate to thermal spray coatings. It should be noted that these tests are not specific to thermal spray coatings; that is, they can be used for a variety of alloy/coating materials. There are, however, ASTM standards that have been developed specifically for thermal spray coatings. They are listed as follows:

- ASTM B 833-01a, "Standard Specification for Zinc and Zinc Alloy Wire for Thermal Spraying (Metallizing)." This specification covers zinc and zinc alloy wire for use in oxyfuel and electric arc thermal spray protection of iron and steel. It does not cover zinc alloy compositions used in thermal spraying for electronic applications.
- ASTM C 633-01, "Standard Test Method for Adhesion or Cohesion Strength of Thermal Spray Coatings." This test method covers the determination of the degree of adhesion (bonding strength) of a thermal spray coating to a substrate or the cohesion strength of the coating in tension. The test consists of coating one face of a substrate fixture, bonding this coating to the face of a loading fixture, and subjecting this coating/fixture assembly to a tensile load normal to the plane of the coating. Usually, this test is performed at ambient temperature. Higher-temperature testing is restricted by the need

for a suitable adhesive bonding agent. For certain investigations, very low (cryogenic) temperatures may be used. The test is limited to thermal spray coatings that can be applied in thicknesses greater than 0.38 mm (0.015 in.). This limitation is imposed because the bonding agents developed to date for this method tend to penetrate the coatings and may invalidate results, unless the coatings are thick enough to prevent penetration through the coating. More detailed information on this test method can be found in the article "Testing of Coatings" in this Handbook.

- ASTM E 1920-97, "Standard Guide for Metallographic Preparation of Thermal Sprayed Coatings." This guide covers recommendations for sectioning, cleaning, mounting, grinding, and polishing to reveal the microstructural features of thermal spray coatings and the substrates to which they are applied when examined microscopically. See the article "Metallography and Image Analysis" in this Handbook for further details.
- ASTM F 1978-00e1, "Standard Test Method for Measuring Abrasion Resistance of Metallic Thermal Spray Coatings by Using the Taber Abraser." This test method quantifies the abrasion resistance of metallic thermal spray coatings deposited on flat metallic surfaces. It is intended as a means of

Table 2 National and international thermal spray conferences held between 1987 and 2003

1st National Thermal Spray Conference, 14–17 Sept 1987 (Orlando, FL)	D.L. Houck, Ed., <i>Thermal Spray: Advances in Coatings Technology</i> , ASM International, 1988, 426+ pages. ISBN: 0-87170-320-3; SAN: 204-7586. 59 papers published and 730 attendees
2nd National Thermal Spray Conference, 24–28 Oct 1988 (Cincinnati, OH)	D.L. Houck, Ed., <i>Thermal Spray Technology: New Ideas and Processes</i> , ASM International, 1989, 469+ pages. ISBN: 0-87170-335-1; SAN: 204-7586. 61 papers published and 1300 attendees
3rd National Thermal Spray Conference, 20–25 May 1990 (Long Beach, CA)	T.F. Bernecki, Ed., <i>Thermal Spray Research and Applications</i> , ASM International, 1991, 792+ pages. ISBN: 0-87170-392-0; SAN: 204-7586. 110 papers published and 1600 attendees
4th National Thermal Spray Conference, 4–10 May 1991 (Pittsburgh, PA)	T.F. Bernecki, Ed., <i>Thermal Spray Coatings: Properties, Processes and Applications</i> , ASM International, 1991, 556+ pages. ISBN: 0-87170-437-4; SAN: 204-7586. 79 papers published and 1300 attendees
13th International Thermal Spray Conference, 5th National Thermal Spray Conference, 28 May–5 June 1992 (Orlando, FL)	C.C. Berndt, Ed., <i>Thermal Spray: International Advances in Coatings Technology</i> , ASM International, 1992, 1044+ pages. ISBN: 0-87170-443-9; SAN: 204-7586. 161 papers published and 1600 attendees
6th National Thermal Spray Conference, 7–11 June 1993 (Anaheim, CA)	C.C. Berndt and T.F. Bernecki, Ed., <i>Thermal Spray Coatings: Research, Design and Applications</i> , ASM International, 1993, 691+ pages. ISBN: 0-87170-470-6; SAN: 204-7586. 105 papers published and 1250 attendees
7th National Thermal Spray Conference, 20–24 June 1994 (Boston, MA)	C.C. Berndt and S. Sampath, Ed., <i>Thermal Spray Industrial Applications</i> , ASM International, 1994, 800+ pages. ISBN: 0-87170-509-5; SAN: 204-7586. 126 papers published and 1500 attendees
8th National Thermal Spray Conference, 11–15 Sept 1995 (Houston, TX)	C.C. Berndt and S. Sampath, Ed., <i>Thermal Spray Science and Technology</i> , ASM International, 1995, 774+ pages. ISBN: 0-87170-541-9; SAN: 204-7586. 126 papers published and 1200 attendees
9th National Thermal Spray Conference, 7–11 Oct 1996 (Cincinnati, OH)	C.C. Berndt, Ed., <i>Thermal Spray: Practical Solutions for Engineering Problems</i> , ASM International, 1996, 992+ pages. ISBN: 0-87170-583-4; SAN: 204-7586. 131 papers published and 1350 attendees
1st United Thermal Spray Conference, 15–18 Sept 1997 (Indianapolis, IN)	C.C. Berndt, Ed., <i>Thermal Spray: A United Forum for Scientific and Technological Advances</i> , ASM International, 1997, 1020+ pages. ISBN: 0-87170-618-0; SAN: 204-7586. 137 papers published and 950 attendees
15th International Thermal Spray Conference, 25–29 May 1998 (Nice, France)	C. Coddet, Ed., <i>Thermal Spray: Meeting the Challenges of the 21st Century</i> , ASM International, 1998, 1693+ pages. ISBN: 0-87170-659-8; SAN: 204-7586. 272 papers published
2nd United Thermal Spray Conference, 17–19 March 1999 (Dusseldorf, Germany)	E. Lugscheider and P.A. Kammer, Ed., <i>Tagungsband Conference Proceedings</i> , Deutscher Verband für Schweißen, Germany, 1999, 879+ pages. ISBN 3-87155-653-X. 170 papers published
1st International Thermal Spray Conference, 8–11 May 2000 (Montréal, Québec, Canada) (The first ITSC sponsored by the ASM Thermal Spray Society, the German Welding Society, and the International Institute of Welding)	C.C. Berndt, Ed., <i>Thermal Spray: Surface Engineering via Applied Research</i> , ASM International, 2000, 1383+ pages. ISBN 0-87170-680-6; SAN 204-7586. 199 papers published and 1068 attendees
International Thermal Spray Conference, 28–30 May 2001 (Singapore)	C.C. Berndt, K.A. Khor, and E. Lugscheider, Ed., <i>Thermal Spray 2001: New Surfaces for a New Millennium</i> , ASM International, 2001, 1361+ pages. 185 papers published
International Thermal Spray Conference, 4–6 March 2002 (Essen, Germany)	E. Lugscheider, Ed., <i>Tagungsband Conference Proceedings</i> , Deutscher Verband für Schweißen, Germany, 2002, 1051+ pages. ISBN 3-87155-783-8. 202 papers published
International Thermal Spray Conference, 5–8 May 2003 (Orlando, FL)	B.R. Marple and C. Moreau, Ed., <i>Thermal Spray 2003: Advancing the Science and Applying the Technology</i> , ASM International, 2003, 1708+ pages. ISBN 0-87170-785-3. 244 papers published

characterizing coatings used on surgical implants (for example, plasma sprayed titanium coatings on hip implant devices). The Taber abraser generates a combination of rolling and rubbing to cause wear to the coating surface. Wear is quantified as cumulative mass loss. The test is limited to flat, rigid specimens that do not react significantly with water and do not undergo a phase transformation or chemical reaction between room temperature and 100 °C (212 °F).

- ASTM E 2109-01, "Test Methods for Determining Area Percentage in Thermal Spray Coatings." These test methods cover procedures to perform porosity ratings on thermal spray metallographic specimens prepared in accordance with ASTM guide E 1920 by direct comparison to standard images and through the use of automatic image analysis equipment. See the article "Metallography and Image Analysis" in this Handbook for further details.

Further Literature Sources for Thermal Spray

The major forum for thermal sprayers and technologists to meet since 1956 has been the International Thermal Spray Conferences (ITSC) (Ref 8). The first seven of these meetings up to 1973 were known as the International Metal Spraying Conferences. Since the mid-1980s, several other meetings have been convened: The National Thermal Spraying Conferences (organized by ASM International), the Plasma-Technik (Ref 11) meeting (a former Swiss company), and the Advanced Thermal Spraying and Allied Coatings Symposium (Ref 12) (organized by the High Temperature Society of Japan). The ITSC-2002 was convened in Essen, Ger-

many, in March 2002 (organized by Deutscher Verband für Schweißen, or DVS, the German Welding Society), ITSC-2003 was held in Orlando, Fla., in May 2003 (organized by the TSS), and ITSC-2004 was held in Osaka, Japan (organized by TSS, DVS, and the International Institute of Welding). Table 2 lists information on various thermal spray conferences held worldwide since 1987.

Other organizations, such as NACE International and AWS, have also convened focused conference sessions that specifically address thermal spraying. The NACE sessions emphasize the behavior of thermal spray coatings under corrosive environments. The SSPC often documents thermal spray coatings with their publication *The Journal of Protective Coatings and Linings (JPCL)*.

Online Resources. Table 3 lists examples of Web sites that can be accessed for thermal spray information. Again, it should be emphasized that this list is not intended to be all-inclusive. Many more thermal spray Web sites can be found in *Thermal Spray Buyers Guide*, described earlier.

REFERENCES

1. ASM International Thermal Spray Society, <http://www.asminternational.org/tss>, March 2004
2. J.R. Davis, Ed., *Surface Engineering for Corrosion and Wear Resistance*, ASM International and IOM Communications, 2001
3. R. Chattopadhyay, *Surface Wear: Analysis, Treatment, and Prevention*, ASM International, 2001
4. Y. Tamarin, *Protective Coatings for Turbine Blades*, ASM International, 2002

Table 3 Selected Internet resources for thermal spray information

Site name or sponsor	Internet address(a)
Professional societies and trade associations	
ASM International	www.asminternational.org
Thermal Spray Society (TSS)	www.asminternational.org/ss
International Thermal Spray Association (ITSA)	www.thermalspray.org
American Welding Society (AWS)	www.aws.org
ASTM International	www.astm.org
NACE International	www.nace.org
The American Ceramic Society (ACerS)	www.acers.org
The Society for Protective Coatings (SSPC)	www.sspc.org
Materials Research Society (MRS)	www.mrs.org
Metal Powder Industries Federation (MPIF)	www.mpiif.org
Deutscher Verband für Schweißen (DVS), The German Welding Society	www.dvs-ev.de/en
International Institute of Welding (IIW)	www.iiw-iis.org
Thermal Spraying and Surface Engineering Association (TSSEA)	www.tssea.co.uk
Surface Engineering Association (SEA)	www.sea.org.uk
Research organizations	
Los Alamos National Laboratory	www.lanl.gov
Oak Ridge National Laboratory (ORNL)	www.ornl.gov
Sandia National Laboratories	www.sandia.gov
Idaho National Engineering and Environmental Laboratory (INEEL)	www.inel.gov
The Center for Thermal Spray Research, State University of New York at Stony Brook	www.matscieng.sunysb.edu/tsl/ctr
Center for the Plasma Processing of Materials (CPPM), Drexel University	www.materials.drexel.edu/CPPM
Hard Chrome Alternatives Team (HCAT)	www.hcat.org
Commonwealth Scientific and Industrial Research Organization (CSIRO), Manufacturing and Infrastructure Technology	www.cmit.csiro.au

(a) URL as accessed March 2004

5. R.C. Tucker, Jr., Thermal Spray Coatings, *Surface Engineering*, Vol 5, *ASM Handbook*, ASM International, 1994, p 497–509
6. H. Herman and R.A. Sulit, Thermal Spray Coatings, *Welding, Brazing, and Soldering*, Vol 6, *ASM Handbook*, ASM International, 1993, p 1004–1009
7. R. Knight and R.W. Smith, Thermal Spray Forming of Materials, *Powder Metal Technologies and Applications*, Vol 7, *ASM Handbook*, ASM International, 1998, p 408–419
8. M.L. Berndt and C.C. Berndt, Thermal Spray Coatings, *Corrosion: Fundamentals, Testing, and Protection*, Vol 13A, *ASM Handbook*, ASM International, 2003, p 803–813
9. B. Kushner and E.R. Novinski, Thermal Spray Coatings, *Friction, Lubrication, and Wear Technology*, Vol 18, *ASM Handbook*, ASM International, 1992, p 829–833
10. F.J. Hermanek, Ed., *Thermal Spray Terminology and Company Origins*, Thermal Spray Society, 2001
11. A.R. Nicoll, Ed., *Proc. First Plasma-Technik Symposium*, 18–20 May 1988 (Lucerne, Switzerland), Plasma-Technik, 1988 (2 volumes)
12. V. Arata, Ed., *Proc. of International Symposium on Advanced Thermal Spraying Technology and Allied Coatings*, 13–15 May 1988 (Osaka, Japan), The High Temperature Society of Japan, 1988

SELECTED REFERENCES

- W.E. Ballard, *Metal Spraying and Flame Deposition of Ceramics and Plastics*, 4th ed., Griffin, London, U.K., 1963
- M.I. Boulos, P. Fauchais, and E. Pfender, *Thermal Plasmas: Fundamentals and Applications*, Vol 1, Plenum Publishing, 1994
- R.B. Heimann, *Plasma-Spray Coating*, VCH, 1996
- L. Pawlowski, *The Science and Engineering of Thermal Spray Coatings*, John Wiley & Sons, 1995
- R.W. Smith, P. Mathur, and D. Springs, Ed., *Thermal Spray Industry Environmental Guideline*, ASM International, 1994
- *Thermal Spraying: Practice, Theory, and Application*, American Welding Society, 1985
- *Thermal Spray Manual*, American Welding Society, 1996

Introduction to Coatings, Equipment, and Theory

Revised by Daryl E. Crawmer, Thermal Spray Technologies Inc.

COATINGS can be thought of as engineering solutions to enhance surfaces against wear, corrosion, thermal degradation, and other surface phenomena (refer to the article “Surface Science” in this Handbook for details). Acceptable coatings are generally characterized by good adhesion, substrate compatibility, and low porosity. An acceptable coating process must also be compatible with physical substrate constraints such as temperature and geometry. These coating/substrate attributes include coefficient of thermal expansion (CTE) matching, appropriate edge radii, substrate melting point, and chemical compatibility during deposition and service. Thermal spray can be tailored to meet many of these requirements. Key aspects of the process are illustrated in Fig. 1, which shows particles impacting a prepared surface and building up a lamellar structure characteristic of thermal spray processes.

Flame spray, the first thermal spray process, was invented nearly 100 years ago. Early applications of thermal spray were for simple shaft repair and roll restoration and surfacing; relatively simple metals and alloys were employed for these applications. Thermal spray usage and applications advanced significantly during the mid-1950s with the invention of plasma arc spray and detonation spray (D-Gun) by Union Carbide (now Praxair Surface Technologies, Inc.). Thermal spray coatings have found their way

into most industries, with widespread commercialization of plasma arc spray and electric arc spray in the early 1960s.

Thermal spray has grown from simple iron and steel coatings to encompass most metals, alloys, ceramics, cermets (ceramic-metal composites), polymers, advanced composites, and nanomaterials. Thermal spray coatings can be found in applications ranging from oil fields to aerospace. Applications include surgical instruments, medical implant prosthetics, medical diagnostics, turbine engine components, papermaking machines, printing rolls, petrochemical pumping and valving, communications, electronics, cookware, and countless others.

Historically, thermal spray was practiced in specialty service and job shops. Advances in thermal spray processes and materials—coupled with advances in process control, instrumentation, and automation—have taken thermal spray into high-volume production applications. Thermal spray processes are commonplace in the manufacture of aircraft engines, automobiles, communication electronics, agricultural products, food processing equipment, and other high-volume products. Advanced applications continue to emerge as breakthroughs develop in process techniques, materials, and controls.

The articles in this Section of the Handbook introduce coatings, thermal spray processes, and equipment. The major processes that constitute the technology—flame, electric arc, and plasma arc spray—are presented together with their processing characteristics. Coatings are presented with respect to structure and performance, while process equipment is presented with respect to operation, coating effects, and health and safety. In summary, this Section is intended to enable the reader to:

- Differentiate among the major processes—flame, electric arc, and plasma arc spray—and many subsets of these three processes
- Understand basic process equipment components
- Understand the characteristic thermal spray coating microstructure and its origin
- Understand how processing and coating microstructure relate
- Know the important controlling parameters for coating quality and understand how these parameters determine coating microstructure
- Become familiar with some of the latest spray coating technology advances
- Understand essential thermal spray health and safety issues

The only absolute in thermal spray is that there are no absolutes. The information given here is offered as generalities, for the most

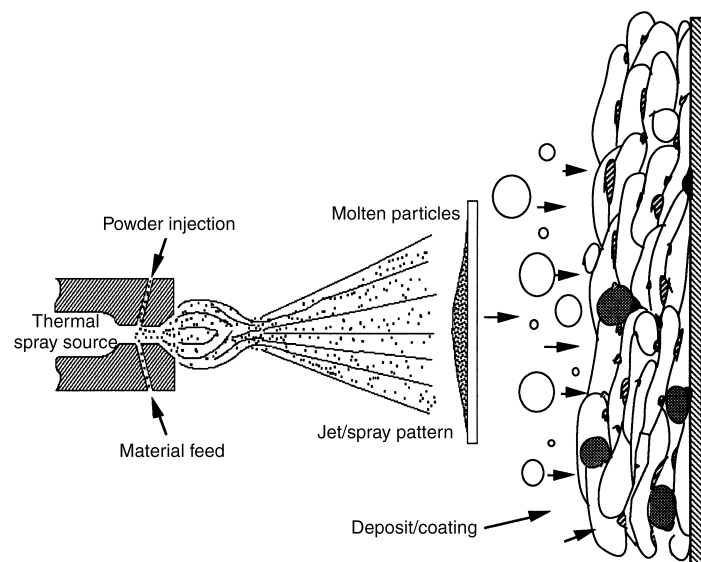


Fig. 1 Typical thermal spray process and coating

part. In the end, the skill and education of the practitioner will determine what can be accomplished with this versatile and powerful technology.

Thermal Spray versus Other Coating Processes

Thermal spray processes differ from other coating processes in that they are nonatomistic; that is, they do not deposit material onto surfaces as individual ions, atoms, or molecules. Instead, relatively massive particulates are deposited onto a surface in the form of liquid droplets or semimolten or solid particles. Coating feedstock materials usually come in the form of powder, wires, or rods. Feedstocks are generally heated to their melting point by a plasma jet, electric arc, or flame. The molten material is then atomized and propelled toward the substrate by process gases or atomizing jets formed through nozzles. Thermal spray is also a “line-of-sight” process, where the projected stream of droplets deposits only onto surfaces that are directly in line with the spray stream.

Because thermal spray processes are high-enthalpy (high energy density) processes, they are characterized as having high coating rates relative to other coating processes—for example, chemical vapor deposition (CVD), physical vapor deposition (PVD), and electroplating. Additionally, thermal spray processes are capable of operating over a broad range of temperature, velocity, and atmospheric conditions, enabling them to apply the great-

est variety of materials. Other advantages of thermal spray processes include a simplified waste-disposal stream and the ability to deposit thick coating sections.

Thermal spray coatings are considered to be “overlay” coatings, which can be defined as materials added to an original surface (called the substrate) where there is little or no mixing or dilution between the coating and the substrate, thus preserving the composition of the base material. Some forms of surface treatments are entirely diffusional in nature. In these surface treatments, elemental materials are added to a base material through diffusional processes, which occur below the substrate surface and cause no thickness buildup. Alloying may occur with the base materials to form new protective compounds or phases.

Diffusion and alloying often occur with thermal spray coatings, but the reaction zone is extremely narrow due to the extremely rapid cooling rates of the individual molten droplets impacting the relatively massive and cold substrate.

Thermal Spray Process Classification

Figure 2 illustrates the three major coating processes: flame, plasma arc, and electric arc, with many of their subsets. (Kinetic-energy-driven spraying or “cold spray” is discussed in the article “Cold Spray Process” in this Section.) Each of these processes encompasses many more subsets, and each has its own character-

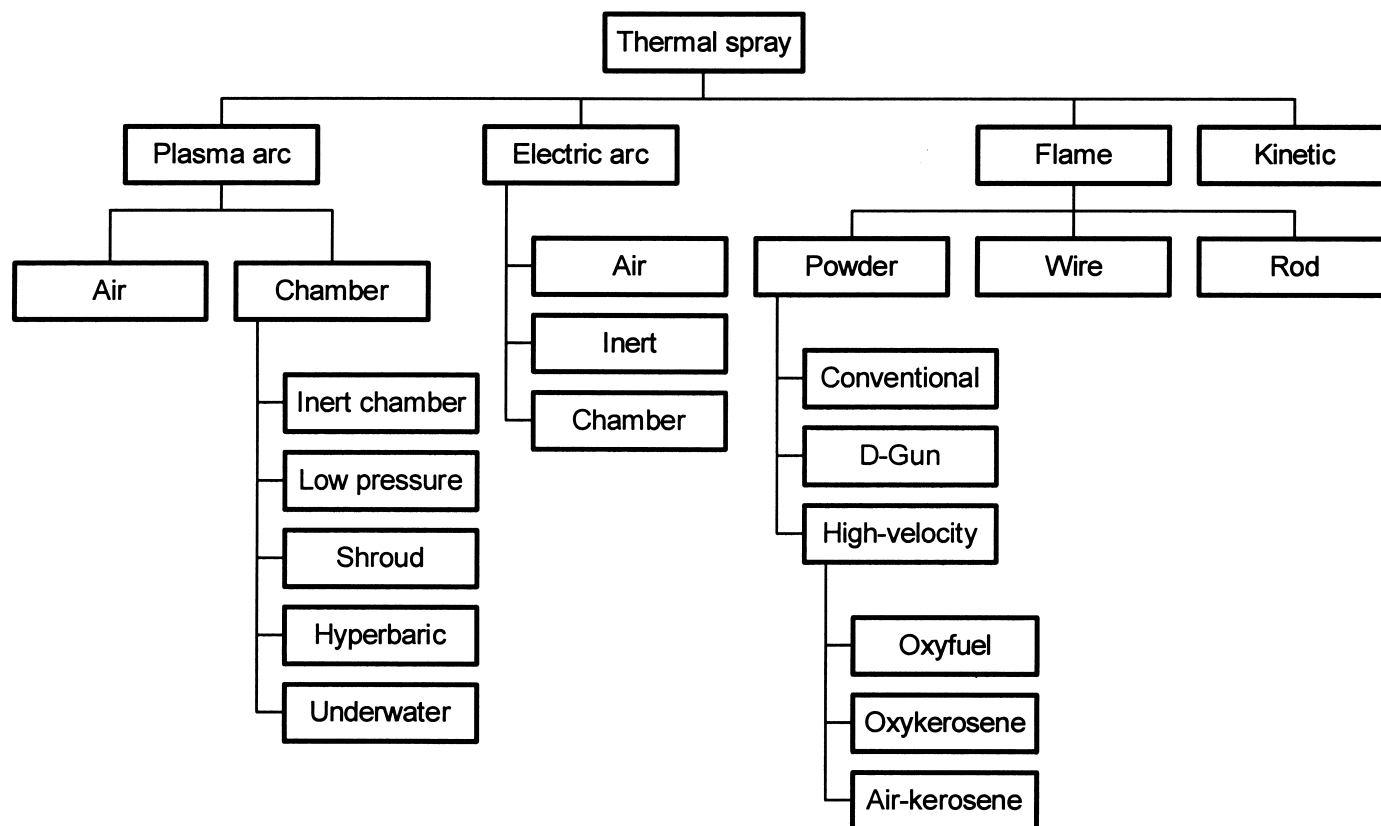


Fig. 2 Thermal spray processes and subsets

istic range of temperature, enthalpy, and velocity. These attributes in turn develop coating characteristics that are unique to each process; in the simplest terms these include coating bond strength, porosity, inclusions (usually oxides), and hardness.

Processing Characteristics

Figure 3 is a schematic representation of a powder spray process that highlights the major process characteristics affecting coating quality. From a simplistic point of view, the process can be viewed as high-speed heat treating (dealing with time, temperature, and mass) where the objective is to bring the powder mass to the desired temperature over a given time period. The time a particle spends in the process jet is called the dwell time; it is governed by gas velocity and powder particle characteristics. Gas velocity in turn is determined by the total gas flow through the nozzle, the gas characteristics, and the energy acting on or resulting from the process. Particle velocity is then a function of jet velocity coupled with particle characteristics—that is, size, morphology, and mass. The temperature of a given particle is thus a function of enthalpy, velocity, trajectory, and its own physical and thermal properties.

Gas temperatures in the spray stream vary greatly as a function of the process. In flame spray processes, temperature is determined in order by the fuel characteristics, oxygen- or air-to-fuel ratio, and the pressure in the combustion zone. Temperatures for common fuel gases range from 2540 to 3150 °C (4600 to 5700 °F). What is not commonly appreciated is the great heat generated by these processes. Standard oxyfuel spray torches operate at energies of 20 to 50 MJ (20,000 to 50,000 Btu). The high-velocity processes operate at energies between 500 MJ and 1 GJ (500,000 and 1 million Btu).

Plasma arc spray devices operate at energies of 72 to 720 MJ (20 to 200 kW), which is the electrical equivalent of 68,280 to 682,800 Btu. Plasma arc temperatures are typically 20,000 °C (36,000 °F), which is considerably higher than the melting point of any known substance. Electric arc spray guns operate at energies between 1.3 and 36 MJ (0.38 and 10 kW). High-spray-rate electric arc spray guns operate at energies up to 135 MJ (37.5 kW).

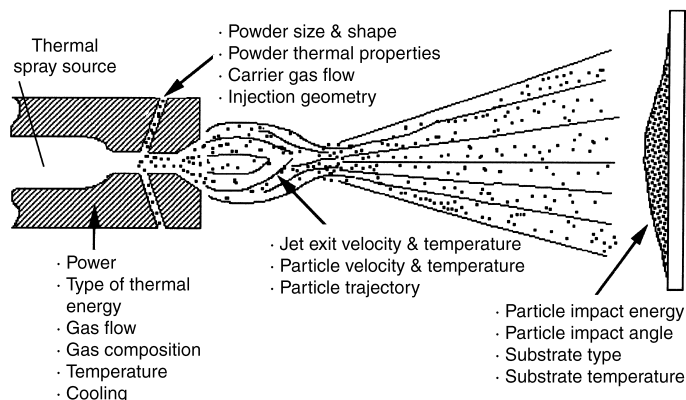


Fig. 3 Typical thermal spray process parameters and variables

Temperature and velocity vary considerably within the jet, because of several factors. The most significant of these is the difference in temperature between the hot core of the jet and its relatively cold surrounding environment. Plasma jets are confined by water-cooled anodes/nozzles. Flame-driven processes, with a few exceptions, are air-cooled devices operating at ambient conditions. The temperature drop from the jet core to the boundary between the nozzle and the environment is typically several thousand degrees over a few millimeters. This drop in temperature causes a corresponding drop in gas velocity and an increase in gas viscosity toward the outer boundaries. Temperature and velocity also decay as the jet exits the nozzle, so there are both radial and axial gradients of gas temperature, gas velocity, and gas viscosity. Into those complex distributions, a distribution of powder is injected. On the surface the process seems chaotic; however, these interactions are favorable, whether by design or chance.

Figure 4 schematically illustrates the dwell time of hypothetical particles, indicating how different particle trajectories lead to a range of dwell times and temperatures. A range of dwell times is characteristic of a spray process or a set of spray parameters. It should be emphasized that for thermal spray processes, single, specific dwell times and temperatures do not exist. Instead of absolute numbers, dwell times and temperatures are dealt with in terms of average values for the distributions.

Distributions of particles traveling in these thermal jets are accelerated by the gas-velocity distributions through a distribution of temperatures. The resulting, final particle velocities and temperatures depend on the trajectories of the particles and the jet/particle “drag forces.” It can be assumed that higher jet velocities generally lead to higher average particle velocities. It can also be assumed that higher enthalpies produce higher average particle temperatures. Particle velocities are important because it is generally accepted that higher particle velocities result in higher particle/droplet impact energy. This higher kinetic energy leads to a higher degree of particle deformation, producing better coating bonds and higher coating densities. It is also accepted that, on average, higher particle velocities result in shorter dwell times,

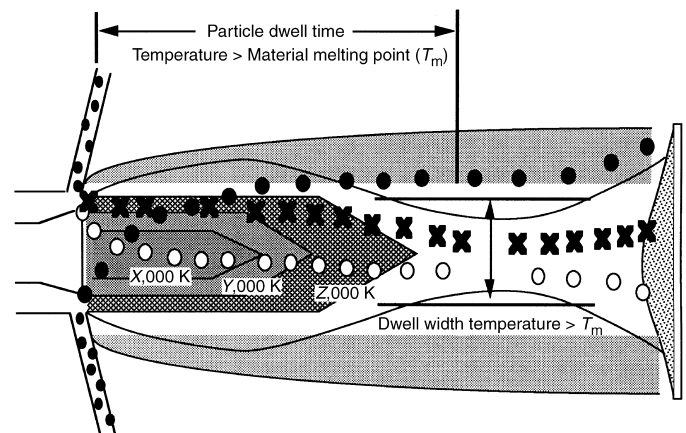


Fig. 4 Thermal spray jet temperature profiles and hypothetical particle trajectories

resulting in less time for particle heating. Lower particle temperatures may lead to lower deposit densities, since a larger percentage of unmelted or partially melted particles become included in the deposit. Increasing jet enthalpy can compensate for shorter dwell times. Lower particle temperatures may not always lower the coating quality, because less particle oxidation and less reduction of carbides, as seen in some plasma spray processes, may occur.

The average particle temperature distribution and velocity distribution determine bond strength, porosity, and coating inclusions such as oxides. This is particularly true when spraying under normal atmospheric (i.e., in air) conditions, where the time and temperature of the particles in the jet determine the level of interaction that the particles have with entrained air. In summary, the final temperature, velocity, and environmental interactions of the particle distribution are related to the particular jet velocity, temperature, and resultant particle dwell time, all of which determine the resulting coating microstructure and properties.

These observations only hold true if particles are being heated in a thermal spray jet. In the electric arc spray process, sometimes also called “wire arc” spray, the sprayed materials are wires that are melted into particulates by an electric arc formed between them. There are no hot gas jets associated with electric arc spray. Unlike other thermal spray processes, droplet/particle temperatures begin to decrease immediately after the particles are formed or atomized in the arc zone, leaving the wire tips as they melt. The dwell time that produces increased heating in other processes only serves to cool particles in electric arc spray, because the atomizing jets are only used to accelerate the particles toward the surface to be coated. However, since the particles are still hot from the melting process, interactions between air and the hot particle surfaces do still occur. The oxides disperse on impact and become included in the deposit, as with the other processes.

Despite the differences in their respective origins in plasma, combustion, or electric arc spray processes, once generated, the hot molten particles impact and deform on the substrate surface in similar ways. The physical differences between the thermal spray processes—that is, particle sizes, velocities, and temperatures—and the jet temperatures lead to large coating variations, as discussed in later articles in this Section.

ACKNOWLEDGMENT

This article has been revised and updated from a portion of “Equipment and Theory” by Ronald W. Smith, Lesson 2, *Thermal Spray Technology*, ASM International, 1992.

SELECTED REFERENCES

- Advances in Thermal Spraying, Proc. 11th International Thermal Spray Conf., 8–12 Sept 1986 (Montreal), Pergamon Press, 1986
- C.C. Berndt, Ed., *Thermal Spray: International Advances in Coatings Technology*, Proc. International Thermal Spray Conf., 29 May–5 June 1992 (Orlando, FL), ASM International, 1992

- C.C. Berndt, Ed., *Thermal Spray: Practical Solutions for Engineering Problems*, Proc. Ninth National Thermal Spray Conf., 7–11 Oct 1996 (Cincinnati), ASM International, 1996
- C.C. Berndt, Ed., *Thermal Spray: A United Forum for Scientific and Technological Advances*, Proc. First United Thermal Spray Conf., 15–18 Sept 1997 (Indianapolis), ASM International, 1998
- C.C. Berndt, Ed., *Thermal Spray: Surface Engineering via Applied Research*, Proc. First International Thermal Spray Conf., 8–11 May 2000 (Quebec), ASM International, 2000
- C.C. Berndt and T.F. Bernecki, Ed., *Thermal Spray Coatings: Research, Design, and Applications*, Proc. Fifth National Thermal Spray Conf., 7–11 June 1993 (Anaheim, CA), ASM International, 1993
- C.C. Berndt, K.A. Kohr, and E. Lugscheider, Ed., *Thermal Spray 2001: New Surfaces for a New Millennium*, Proc. International Thermal Spray Conf., 28–30 May 2001 (Singapore), ASM International, 2001
- C.C. Berndt and S. Sampath, Ed., *1994 Thermal Spray Industrial Applications*, Proc. Seventh National Thermal Spray Conf., 20–24 June 1994 (Boston), ASM International, 1994
- C.C. Berndt and S. Sampath, Ed., *Advances in Thermal Spray Technology*, Proc. Eighth National Thermal Spray Conf., 11–15 Sept 1995 (Houston), ASM International, 1995
- T.F. Bernecki, Ed., *Thermal Spray Research and Applications*, Proc. Third National Thermal Spray Conf., 20–25 May 1990 (Long Beach, CA), ASM International, 1991
- T.F. Bernecki, Ed., *Thermal Spray Coatings: Properties, Processes and Applications*, Proc. Fourth National Thermal Spray Conf., 4–10 May 1991 (Pittsburgh), ASM International, 1992
- C. Coddet, Ed., *Thermal Spray: Meeting the Challenges of the 21st Century*, Proc. 15th International Thermal Spray Conf., 25–29 May 1998 (Nice, France), ASM International, 1998
- D.L. Houck, Ed., *Thermal Spray: Advances in Coatings Technology*, Proc. National Thermal Spray Conf., 14–17 Sept 1987 (Orlando, FL), ASM International, 1988
- D.L. Houck, Ed., *Thermal Spray Technology: New Ideas and Processes*, Proc. National Thermal Spray Conf., 24–27 Oct 1988 (Cincinnati), ASM International, 1989
- F.N. Longo, Ed., *Thermal Spray Coatings: New Materials, Processes and Applications*, Proc. Second National Conf. on Thermal Spray, 31 Oct–2 Nov 1984 (Long Beach, CA), American Society for Metals, 1985
- E. Lugscheider and P.A. Kammer, Ed., *United Thermal Spray Conference*, Proc. United Thermal Spray Conf., 17–19 March 1999 (Düsseldorf) DVS-Verlag, 1999
- A. Ohmori, Ed., *Thermal Spraying: Current Status and Future Trends*, Proc. 14th International Thermal Spray Conf., 22–26 May 1995 (Kobe, Japan), High Temperature Society of Japan, 1995
- *Thermal Spraying: Practice, Theory and Application*, American Welding Society, 1985
- *12th International Conference Thermal Spraying*, 4–9 June 1989 (London), The Welding Institute, 1989

Coating Structures, Properties, and Materials

Revised by Daryl E. Crawmer, Thermal Spray Technologies Inc.

IMPORTANT THERMAL SPRAY COATING FEATURES, which combine to determine the properties of a coating, include the lamellar or layered splat structure, entrapped unmelted or resolidified particles, pores, oxide inclusions, grains, phases, cracks, and bond interfaces. Figure 1 schematically illustrates typical coating characteristics and a representative deposit/coating microstructure.

Splat

“Splat” is the term given to a single impacted droplet/particle, as illustrated in Fig. 2. Many overlapping splats (illustrated in cross section in Fig. 3) solidify and adhere to one another to form a continuous coating layer. Thus, the splat is the basic structural building block in thermal spray coatings. Splats are created when the accelerated, molten particles impact a prepared surface. The arriving molten droplets are generally spherical, and on impact with the substrate surface they spread over and fill the underlying interstices (spaces). The droplets become flattened, disklike structures which, when polished in cross section, look like the coating microstructures shown in Fig. 3 to 5. In these figures, the splats are seen on edge, revealing the characteristic lamellar structure of the thermal spray coating, shown schematically in Fig. 2.

Figures 4 and 5 show micrographs of a ceramic coating of 8 wt% yttria-stabilized zirconia (YSZ) and an electric arc sprayed low-carbon steel coating, respectively. Note that these thermal spray coatings both exhibit the characteristic lamellar structure. The splat structure in coating microstructures is indicative of the degree of particle melting achieved in flight. In electric arc sprayed coatings, the lamellar microstructures are also typical and indicate that the atomized droplets had not yet solidified prior to impact. The degree of “melting” in the coating structure, as indicated by these lamellae, largely determines the cohesion, porosity, and subsequent properties of the coating.

Thermal spray processes are also characterized by rapid solidification. As the relatively small individual particles impact the more massive substrate, their heat is liberated quickly. Solidification rates (for metals) are in the range of 10^5 to 10^8 °C/s. Such rapid cooling rates produce a wide range of material states, from amorphous to metastable. Two structures are generally present within a coating: splat structures and intrasplat structures. Within the splats, the rapid solidification of thermal spray creates grain sizes less than $5\text{ }\mu\text{m}$ (0.0002 in.) and prevents many materials from achieving their equilibrium phases, resulting in anisotropic coating properties. In most thermal spray coatings, the properties in the

x and y planes, that is, the planes parallel to the substrate surface, are the same. However, in the z plane, perpendicular to the substrate surface, properties vary significantly from those in the x and y planes. Despite the possibility of oxide inclusions in the coating, the rapid thermal processing and solidification in thermal spray

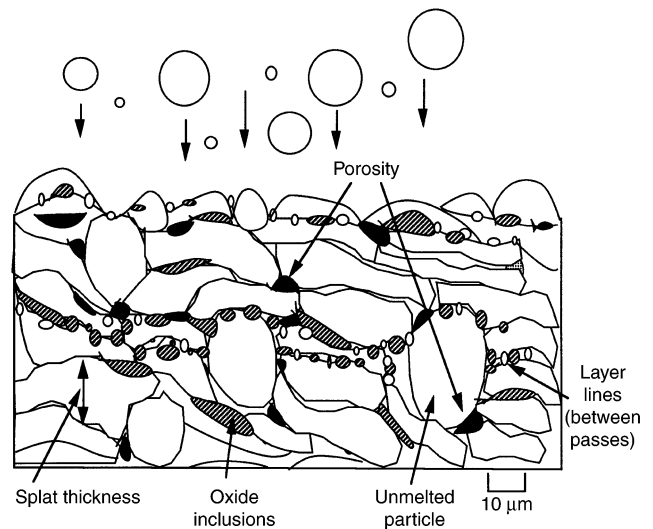


Fig. 1 Thermal spray coating microstructure showing common features

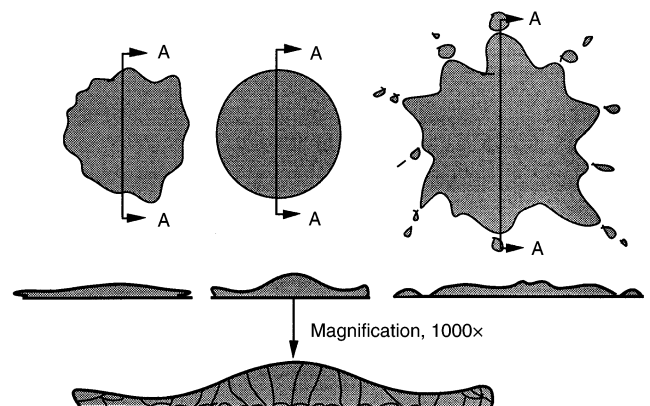


Fig. 2 Typical thermal spray splat structures

coatings lead to compositional homogeneity that is representative of the starting feedstock compositions.

Oxide Stringers

Oxide inclusions (stringers) in metallic coatings are generally seen as dark, elongated phases that appear as strings in the coating cross section, parallel to the substrate. Oxides are produced by particle/atmosphere interaction and/or heating of the coating surface during deposition. Interaction of the hot particles with their surrounding environment, usually air, leads to oxide and/or nitride films on particle surfaces. Longer dwell times and higher particle

temperatures increase the thickness of the oxide or nitride layers on the particles, producing higher concentrations of oxide stringers within the coating. As the particles spread on impact, surface films fracture with the flowing metal and become included in the deposit as the droplets solidify into splats, also shown in Fig. 2, 3, and 5.

It has been found that these oxide inclusions add to the hardness of the coating, since oxides have hardnesses exceeding 1000 DPH. Therefore, the composite mixture of metal and metal oxides becomes harder than the metal coating itself. This increased hardness can lead to brittle coatings, because oxides fracture easily. If too concentrated, oxides may interfere with splat-to-splat cohesion, leading to decreased cohesive strength of the coating. Oxide or nitride inclusions are often considered detrimental to coating properties. However, in some applications oxide inclusions are desired because they can increase wear resistance or lower thermal conductivity.

Minimizing Inclusions. Generally, however, inclusions should be minimized. Parameter trends that minimize inclusions are:

- Removing the reactive environment, through use of inert gas shrouds or chambers
- Reducing the average temperature of particles by lowering the heating capacity of the spray jets
- Reducing the dwell time of the particles by minimizing spray distances or increasing velocities
- Reducing substrate/coating surface temperature using cooling air jets or increasing the speed of the thermal spray device across the part surface (surface speed), thus minimizing oxidation at the coating surface
- Using the proper feedstock powder particle size. Larger particles have a lower surface area to volume ratio, which minimizes overall oxide content.

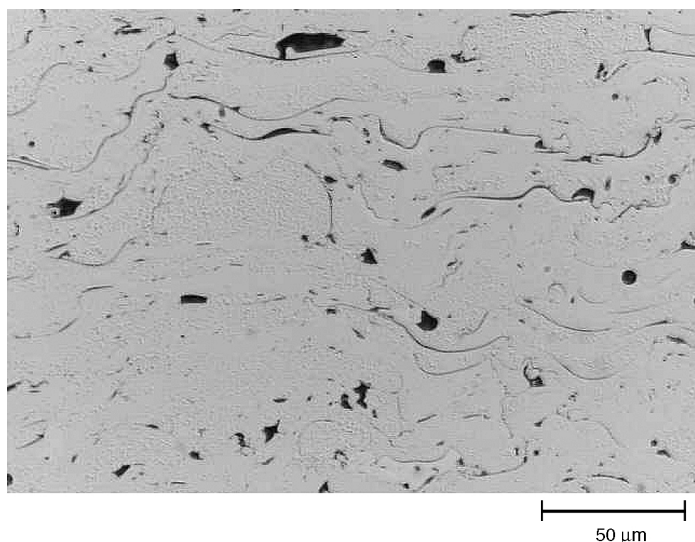


Fig. 3 Plasma-sprayed nickel-base alloy. Courtesy of Thermal Spray Technologies

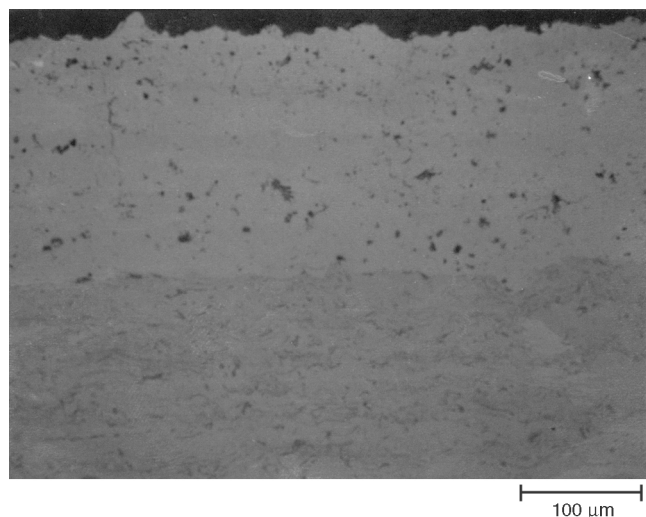


Fig. 4 Plasma-sprayed yttria-stabilized zirconia on vacuum plasma sprayed NiCrAlY. Courtesy of Drexel University

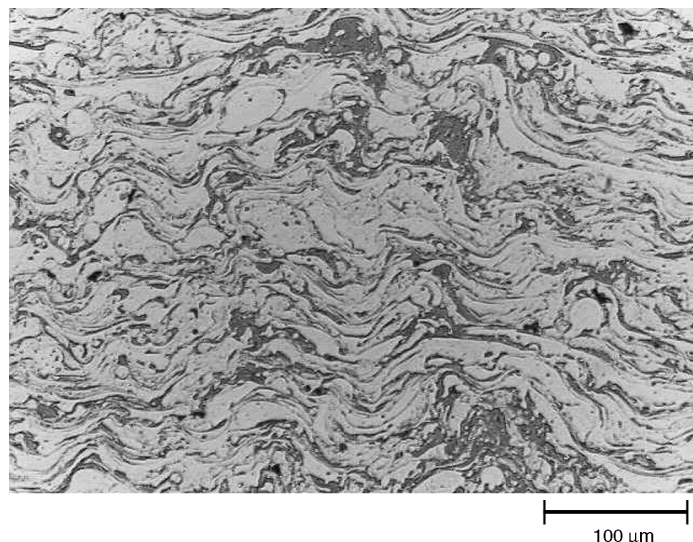


Fig. 5 Electric arc sprayed low-carbon steel. Courtesy of Thermal Spray Technologies

Porosity

Porosity is another important coating feature that strongly influences coating properties. As with oxide inclusions, porosity can be a desirable characteristic. This discussion takes the generally held position that porosity is undesirable. Porosity creates poor coating cohesion and allows for higher wear and corrosion rates. Porosity is generally associated with a high number of unmelted or resolidified particles that become trapped in the coating, as shown in Fig. 1 and 6. Poor splat or particle cohesion leads to premature coating cracking, delamination, or spalling. Open porosity can interconnect to the coating interface, enabling corroding or oxidizing elements to attack the base material. Porosity can thus “short-circuit” the inherent corrosion resistance of a coating. For hardface or wear-resistant coatings, porosity lowers coating hardness and contributes to poor surface finishes, thus decreasing wear resistance.

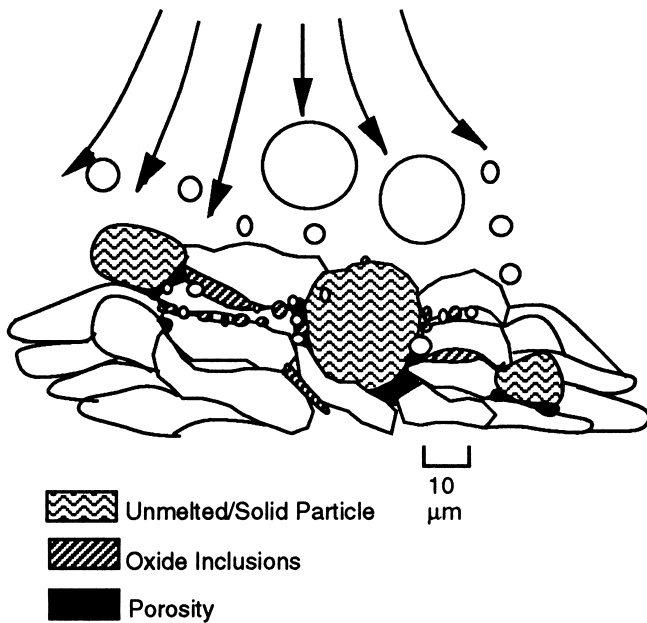


Fig. 6 Typical thermal spray coating defects

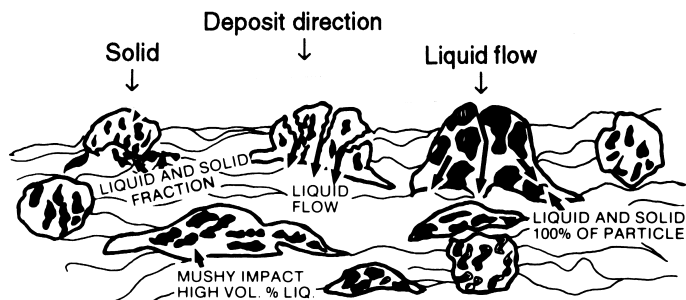


Fig. 7 Particle impact states that create structural features

Porosity in wear coatings can also lead to the generation of coating fragments that break away and become abrasive cutting agents, increasing coating wear rates.

In many applications the rough, as-sprayed surface finish typical of thermal spray coatings must be machined before the coating is put into service. Grinding often produces finishes below $1\text{ }\mu\text{m}$ ($40\text{ }\mu\text{in.}$). On some bearing surfaces, coatings must be honed to below $0.1\text{ }\mu\text{m}$ ($4.0\text{ }\mu\text{in.}$), producing mirrorlike finishes. High coating porosity prevents such finishes from being achieved and results in surface pits and nonuniform, poorly machined finishes.

Despite lowering coating cohesion and/or corrosion protection, coating porosity may not always be detrimental. Oil/grease-impregnated or solid-lubricant bearings can be produced by creating 10 to 30% coating porosity into which lubricants can infiltrate. Porosity is also usually desirable in ceramic thermal barrier coatings (TBCs), widely used in aircraft and land-based turbines and diesel engines. Thermal barrier coatings are generally composed of oxide ceramics, which by themselves are inherently good thermal insulators; the inclusion of 8 to 15% porosity further increases their insulating properties. Porosity also increases thermal shock and thermal cycling (fatigue) resistance. Medical implant prostheses are another useful application of porous coatings, where as much as 40% porosity is intentionally included. Porosity is used in implant coatings to allow bone matter to grow into the coating, which accelerates patient healing and shortens recovery time.

Sources of Coating Porosity. Because porosity is such an important microstructural feature, its origin must be understood and controlled. Porosity can originate from the following:

- Material shrinkage on cooling from the liquid state
- Entrapped unmelted, partially melted, or resolidified particles, which lead to voids
- Shadowing from these “unmelts,” creating line-of-sight voids that are not filled by the spreading droplets
- Poor intrasplat cohesion, leading to separation of splats
- Poor wetting onto adjacent surfaces, or splats that can result from cool or slow-moving particles or surface tension phenomena
- Intersplat or intrasplat cracking
- High deposition angles that lead to shadowing or “snow drifting”
- Ricochet of particles from masking and tooling that allows particles to cool before impacting with the coating surface
- Shadowing from adjacent surfaces (masks)
- Porosity inherent to the feedstock powder manufacturing process

From this list, the most common source of coating porosity is trapped, unmelted, or resolidified particles. Depending on the particle temperatures, the arriving droplets may cover the full range of liquid to solid states. The liquid-state particles flow easily and fill most voids, as illustrated in Fig. 7. Solid particles, some of which are reflected from the solid surface, may adhere locally to, or become trapped in, the rough finish of the coatings. These “overspray” particles are not well bonded, nor are they in intimate contact with the underlying splat, which creates voids that are not open to direct line-of-sight. As stated before, thermal spray is a

line-of-sight process; hence, the next arriving particles cannot fill voids adjacent to trapped solid particles. This is shown in Fig. 1, 6, and 7. In other cases, even in the same instant, some solid + liquid (i.e., partially molten) particles may deform and be completely densified, provided sufficient liquid is available to fill any voids that form around the small amount of existing solid core. It is also possible that partially melted particles act similarly to solid particles, leaving trapped voids around their solid portions.

The angle of impingement of the spray stream is another source of porosity, because of “shadowing.” Figure 8 illustrates the type of shadowing generally associated with coatings sprayed at angles below 45° from the optimal “normal” angle of incidence. Coating porosity decreases (i.e., density increases) as the angle of spray approaches 90°, that is, normal to the surface being coated. It follows that more advanced coating systems are sprayed with tighter tolerances on fixture alignment. Plasma coatings may be sprayed at $\pm 15^\circ$, whereas low-end coatings may be sprayed at $\pm 30^\circ$. Figure 8 shows how surface protrusions build up and then shadow interstices or voids adjacent to, and behind, the protrusions. These protrusions produce even more shadowing, particularly as the angle of incidence is lower relative to the surface, that is, $<90^\circ$.

Masking, tooling or edges, and corner radii may also contribute to localized porosity, as shown in Fig. 9. Colder, solid particles that may normally be blown from the surface by the process or

cooling air jets may ricochet off tooling and masking back into the deposit. The trapped solid particles lead to high, but localized, porosity. One notable exception to this rule is the high kinetic energy spray processes, for example, high-velocity oxyfuel (HVOF). In these cases, although deposition efficiency (DE) decreases as the spray stream is moved off-axis, the coating quality is maintained over a wider range of impingement angles. With HVOF, good coatings can be produced at angles up to 45° off-axis.

Porosity may also be caused by thermal stresses that can result in localized coating shrinkage, or by intersplat oxidation layers that pull themselves apart during cooling. In these cases, the next liquid droplets that impact cannot fill the voids, leading to porosity. Oxides can cause porosity due to poor wetting during splat spreading, which leads to lifting of the splat edges, producing voids. Residual stresses, caused by shrinkage or differences in coefficient of thermal expansion, can add to these local splat effects or act on a larger scale, leading to macrocracking or intersplat cracking of the coating.

Finally, metallographic preparation significantly influences the interpretation of coating quality. The heterogeneous nature of most coatings makes the polishing of them very difficult. Pullout or fracturing of hard phases and poorly adhered splat sections is a common source of porosity in both metallographic samples and in ground and machined coating surfaces. See the article “Metallography and Image Analysis” for more detailed information.

Porosity control is largely a function of particle size distribution, the method of powder manufacture, the degree of melting of the sprayed droplets, and their angle of impact. With these factors controlled, coating porosity can be managed. Particle melting is controlled by:

- Jet temperature and enthalpy distribution
- Jet/particle heat-transfer effectiveness, jet/gas properties
- Particle size and size distribution
- Particle morphology
- Particle thermal properties
- Particle dwell time
- Particle trajectory distributions
- In the case of electric arc spray, voltage, current, and atomization gas flow

These factors largely determine the molten state of particles on impact and the degree of particle oxidation prior to the impact of the next particle, which, as discussed previously, controls the final coating porosity levels.

Summary. This section has briefly introduced the thermal spray process and focused on the microstructural aspects of coatings. These microstructural features include splat formation, porosity, and inclusions.

Arriving particle/droplet velocity distributions strongly influence porosity formation. The droplet impact energy, as determined by the particle velocity and mass, is used to deform each particle or droplet. In the case of liquid droplets, the impact energy spreads the liquid interface into surface voids and around surface irregularities, further enhancing deposit cohesion. On the other hand, solid or partially melted particles are plastically deformed

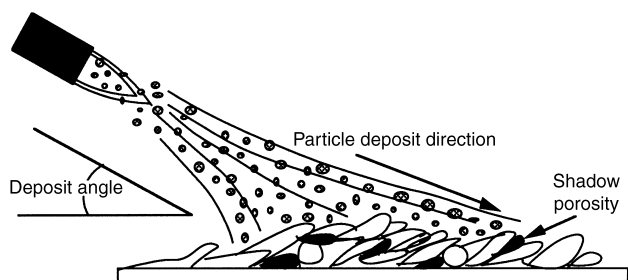


Fig. 8 Porosity created by shadowing resulting from off-axis spraying

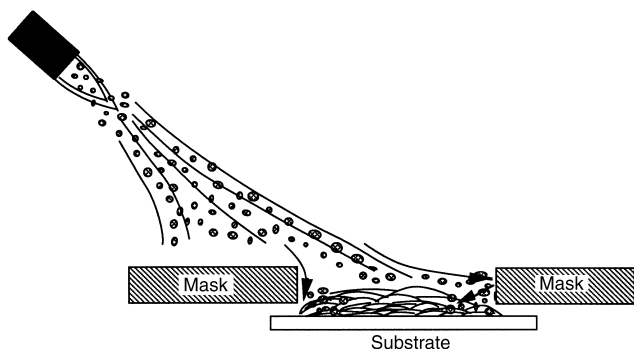


Fig. 9 Porosity created by masking interference

on impact. Thus, it can be seen that higher particle velocities lead to greater particle deformation and therefore better closing of intersplat voids. The high-velocity, higher-impact energies also break up oxide inclusions, leading to improved intersplat cohesion and lower oxide-related porosity. Therefore, depending on the desired coating structure, higher particle velocities—produced by higher jet velocities—improve coating density by mechanical (kinetic) energy, not thermal energy, by forcing porosity closure.

Finally, porosity attributed to line-of-sight limitations, the inclusion of solid particles, and/or deposit incidence angle effects can be managed by:

- Maintaining close-to-normal deposit angles, staying above 30° relative to the surface
- Using higher surface speeds to minimize localized substrate overheating
- Using properly designed masking and substrates to minimize inclusion of reflected particles

Thermal Spray Coating Properties

Thermal spray coatings are used to address an ever-increasing variety of surfacing needs. Their properties are usually expressed in terms of:

- Bond strength
- Hardness
- Corrosion/oxidation resistance
- Thermal properties
- Electrical properties, such as conductivity, resistivity, and dielectric strength
- Magneto-optical properties, such as absorptivity and reflectivity
- Machinability for finish

The relative importance of these properties is based on the intended coating function. As reviewed above, coating characteristics such as porosity, splat cohesion, and oxide content all have significant bearing on these properties.

Bonding of the coating to the substrate and cohesion between consecutive splats is affected, in rough order, by:

- Residual stresses within the coating
- Melting and localized alloying at the contact surfaces between particles and between the substrate and adjoining particles
- Diffusion of elemental species across splat boundaries
- Atomic-level attractive forces (van der Waals forces)
- Mechanical interlocking

Measurable bond strength is controlled by residual stress in the coatings. Typically, even where coating failure appears to occur at the coating/substrate interface, residual coating material is found adhered to the substrate. Coating adhesive and cohesive failures are usually attributable to stresses within the coating structure. Most coating failures occur at the coating/substrate interface, because that is where the greatest discontinuity exists in the mate-

rials system. Where the materials are compatible and the surfaces properly prepared, adhesion between the first splat layer and the substrate is extraordinary, due to the phenomena listed previously. Surface roughening is generally used to increase surface area, which increases the probability that these favorable interactions will occur.

Mechanical interlocking, as illustrated in Fig. 10, has been viewed historically as the main mechanism of thermal spray coating adhesion. Mechanical interlocking can play a part in coating adhesion and cohesion when the surfaces being coated have features that allow molten material to flow into and fill negative relief or where the part has negative relief, as with shafts. In this case, the bond between the impacting particles and the surface is established largely through the impact of particles that flow and solidify around the substrate surface asperities. Substrate asperities with negative relief can be formed prior to coating by grit blasting and other mechanical surface preparation techniques, or by process-induced irregularities on the actual coating surface.

Oxides, dirt, and/or oils on the substrate usually interfere with splat formation and will reduce or prevent local bonding. The degree of coating bonding determines its reliability under mechanical or thermal loading. Hence, surface preparation, cleaning, and roughening are very important steps prior to thermal spraying. Coating bonding continues to be one of the largest sources of coating failure. Many failures result from overlooking the substrate design, incorrect material selection, and improper design of the processing steps.

After deposition of the initial coating layer, which is in direct contact with the base material, splats of the next coating layer bond through what is termed “cohesion” as new droplets impact on previously solidified particles. Cohesion is simply adhesion between splat particles. The coating strength is largely determined by its cohesive strength (controlled by many of the same bonding factors as for base material/coating), but is also influenced by porosity, oxide inclusions, and unmelted particles within the deposit. Unmelted particles lower the splat-contact area, which reduces particle cohesion and locally decreases heat transfer, leading to local coating inhomogeneities. Localized cohesive failures, caused by poor bonding, thermal cracking, and poor wetting

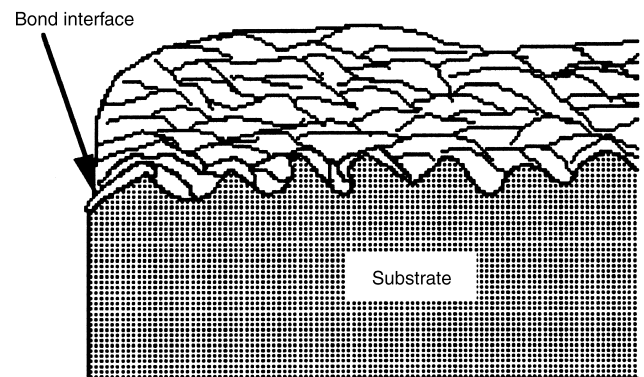


Fig. 10 Interlocking of coating particles

between particles, ultimately determine the net strength and physical properties of a coating. Although thermal spray coatings exhibit limited ductility or toughness, they have intrinsically high strengths and often exhibit high hardnesses.

Bond strength is typically measured in accordance with ASTM C 633-01 ("Standard Test Method for Adhesion or Cohesion"). This technique is limited by both technique and the strength of the adhesive used for the test. The structural adhesives used are limited to ~83 MPa (~12 ksi). Thermal spray coatings have measured bond strengths ranging from 7 to 83 MPa (1 to 12 ksi) using ASTM C 633. In some cases, the actual bond strengths may exceed the strength of the base metal. As a general rule of thumb, the values indicated by ASTM C 633 are an order of magnitude lower than the actual coating bond strength.

Coating behavior is ultimately the average of all the localized structural features and properties. Porosity, oxide inclusions, splat cohesive contact, and various phase inclusions all contribute to coating macroproperties and thus define its suitability for a given application.

Thermal Spray Materials

Generally, materials that are suitable for thermal spray processing are stable at elevated temperatures. Materials that dissociate, decompose, or sublime tend to be poor candidates for coatings. Thus, most metals, intermetallics, alloys, all forms of ceramics (including oxides, borides, silicides, etc.), cermets, and some polymers are sprayable by one or more of the thermal spray processes. Again, these materials are usually supplied in powder, wire, or rod form.

Coating Functions

The flexibility of thermal spray processing offers the possibility of applying coatings of almost any material onto almost any substrate to address a wide range of applications. The following is a brief list of common applications. This list is in no way complete. Once the attributes of the various thermal spray methods are known and their effects on coating or materials structures are understood, then only one's imagination limits the use of thermal spray.

Wear Coatings. Tribology, the scientific study of wear phenomena, is a complex discipline in its own right, and there are myriad wear mechanisms. Thermal spray coatings have been employed to address many of these wear mechanisms, including abrasion, erosion, cavitation, galling, fretting, and more. Because thermal spray can deposit unconventional materials, more than one aspect of wear can be addressed by a single coating since other physical properties can be included in the coating. Cermets can be used to combine wear resistance with thermal conductivity. Nonstick and low coefficient of friction materials can be combined with hard coatings. Self-lubricating materials can also be deposited. Corrosion- and wear-resistant materials can be combined. Coating hardnesses can be engineered, ranging from the low 20s through the low 70s HRC. Oxides and carbides are common wear-resistant materi-

als, with chromium oxide and tungsten-carbide/cobalt (WC/Co) cermets being the two most common coatings.

Thermal Insulation. Thermal barrier coatings are usually made from low-thermal-conductivity zirconium and/or aluminum oxides, which are deposited to reduce heat conduction to base materials. These insulating oxide coatings can also reduce heat loss, thermal transient effects, and high-temperature oxidation/corrosion effects by lowering the temperature of the coated base metal. Thermal spray processes usually introduce controlled levels of porosity and microcracking, which further enhance the insulation of the coating and increase its thermal shock resistance. The most common of these is YSZ, which is actually a partially stabilized zirconia (PSZ) form of the material usually containing 8 wt% yttria.

Corrosion Resistance. Corrosion protection is usually provided by more noble metals, chemically inert ceramics, or plastics, all of which can be thermally sprayed onto parts to protect against environmental, atmospheric, acidic, or caustic corrosion. Porosity is of more concern in corrosion coatings than most applications. Interconnected porosity can short-circuit the corrosion protection, which requires thermally sprayed coatings to be impervious. High-energy thermal spray coatings are particularly well suited for corrosion applications because they characteristically exhibit small, isolated porosity, usually <1%. Common materials used for corrosion control include zinc, pure aluminum, nickel-base alloys, and copper-nickel alloys.

Abradables and Abrasives. Abradable coatings are most often applied in gas turbine engines for clearance control at the tips of rotating turbine blades. Blade tips are designed so that they are able to cut grooves in the relatively soft coating. The blade/seal labyrinth creates a gas-path seal that increases engine performance by preventing gases from bypassing the blades. These coatings are typically nickel/graphite, nickel/bentonite, aluminum/polyester, or other hard/soft material combinations. Abrasion-resistant materials, such as ceramics and carbides, may be applied to the blade tips directly or in a matrix to reduce wear as the tips rub against the abradable coating.

Electrically Conductive Coatings. Thermal spray is often used to create conductors for contacts, electrical connections, heating elements, electromagnetic and radio frequency interference (EMI/RFI) shielding, and so on. Conductive materials include silver, copper, aluminum, tin alloys, and bronze alloys. Typical conductivities of these sprayed metals are reported to be 40 to 90% of their wrought material counterparts, depending on the spray coating method used. Silicon bronze, zinc, and babbitt are used for interference shielding to protect sensitive electronic devices from EMI/RFI. Unusual conductors sometimes sprayed include aluminum titanate, barium titanate, molybdenum disilicide, and other ceramic compounds. Thermal spray has seen limited use for the deposition of superconducting materials. These are materials that have zero resistance at low to intermediate temperatures of 77 K (−320 °F), for example, 1-2-3 YBaCuO, Cu/Nb₃Sn, and Nb₃Sn.

Electrically Resistive/Insulating Coatings. The most common electrically insulating materials used in thermal spray are alumina compounds, including pure alumina, alumina/titania, mul-

lite, aluminum silicate, and so forth. These coatings can be found in communications devices and surgical instruments, among many other applications.

Dimensional Restoration Coatings. Thermal spray had its beginnings in shaft and roll coatings, which were mostly restorative (repair) applications. Simple iron and steel coatings are used to restore worn shafts and journals and roll surfaces. Using electric arc spray techniques, coating thicknesses of up to several millimeters can be applied.

One unusual application is the repair of aircraft skins using plasma arc sprayed pure aluminum as a filler for scratches and dents. This simple, but effective repair technique saves the great expense of removing and replacing aircraft panels, which are often structural members.

Medical coatings are divided into two categories: bioactive and biocompatible. Both coating types are used for implant prostheses. Biocompatible coatings are typically porous titanium applied to titanium alloys; new bone tissue grows into the porosity and secures the implant. Bioactive coatings are typically some form of calcium phosphate, including tricalcium phosphate and hydroxyapatite. These coatings emulate the characteristics of natural bone such that new bone growth attaches to the implant and eventually resorbs—that is, replaces—the coating. Both types of coatings are intended to speed patient recovery and extend usable implant life. Dental implants and other orthopedic devices are also coated in this way.

Polymer Coatings. Many polymers can be thermally sprayed for use as protection against chemical attack, corrosion, or abrasion. Unlike inorganic coatings, many polymer coatings exhibit properties equal to, or greater than, their cast or molded counterparts. Powder flame spray is the most common technique used to produce polymer coatings. Low-melting (glass transition temperature, T_g) polymeric materials such as polyvinyl chloride, ethylene vinyl acetate, and nylon coatings are the most easily sprayed.

In summary, thermal spray coatings have become critical to enhancing the performance of many base materials used in spacecraft, aircraft engines, gas turbines, chemical reactors, metalworking mills, textile guides, bridges, pumps, compressors, medical prostheses, and even household items such as frying pans and toilet seats. An important surfacing method, the technology is heavily relied upon by manufacturers to provide wear, corrosion,

and/or thermal protection; to improve electrical properties; and/or to refurbish and maintain many products. Historically, thermal spray has been used as a coating technology. However, recent process developments now extend thermal spray to the production of freestanding structures, composite metal structures, metal/ceramic-matrix materials, superconducting oxides, and near-net-shape parts. Thus, thermal spray has become more widely used as a materials processing technology for a broad range of manufacturing applications.

The next article in this Section of the Handbook reviews different deposition processes, operating parameters, and the influence of process parameters on coating microstructures. Improved thermal energy transfer to particles, increased particle dwell times, properly selected particle sizes for a particular process, and controlled particle/environment interactions all lead to lower coating porosity and improved coating mechanical properties.

ACKNOWLEDGMENT

This article has been revised and updated from a portion of “Equipment and Theory” by Ronald W. Smith, Lesson 2, *Thermal Spray Technology*, ASM International, 1992.

SELECTED REFERENCES

- M.L. Berndt and C.C. Berndt, Thermal Spray Coatings, *Corrosion: Fundamentals, Testing, and Protection*, Vol 13A, *ASM Handbook*, ASM International, 2003, p 803–813
- S. Granger and J. Blunt, Ed., Thermal Spray Processes, *Engineering Coatings: Design and Application*, 2nd ed., William Andrew Publishing, 1998, p 119–166
- H. Herman and R.A. Sulit, Thermal Spray Coatings, *Welding, Brazing, and Soldering*, Vol 6, *ASM Handbook*, ASM International, 1993, p 1004–1009
- B.A. Kushner and E.R. Novinski, Thermal Spray Coatings, *Friction, Lubrication, and Wear Technology*, Vol 18, *ASM Handbook*, ASM International, 1992, p 829–833
- R.C. Tucker, Thermal Spray Coatings, *Surface Engineering*, Vol 5, *ASM Handbook*, ASM International, 1994, p 497–509

Thermal Spray Processes

Revised by Daryl E. Crawmer, Thermal Spray Technologies Inc.

THERMAL SPRAY is a group or family of processes that uses a concentrated heat source to melt feedstock materials while imparting kinetic energy, using process jets to propel the molten particulates toward a prepared surface. When the hot energetic particles impact the surface of the part being coated, they rapidly solidify. As subsequent particles impact the surface, a deposit thickness is built up. Central to the process are the heat source and means used to accelerate the particulates. Thermal spray devices used to accomplish this work are commonly called guns or torches.

Thermal energy for spraying may be generated chemically, through the combustion of fuels with oxygen or air, or through the electrical heating of industrial gases. These are the most common heat sources. Radiofrequency (RF), or induction-coupled plasma, torches have been used since the early 1960s. In these devices, the plasma is generated by imposing an RF field onto the gas stream.

The most practical means of producing the kinetic energy needed to propel molten particles is to constrict or focus the process gas stream through a nozzle. Gas jets are able to heat and/or accelerate the droplets by confining the particulates to the jet where they can be heated and accelerated. Feedstock sources for the coating can be gases, liquids, or solids; however, solids such as powders, wires, or rods are most compatible with both the ranges of materials used and the processes used in thermal spraying. Energy can be applied either directly to the materials, as in

electric arc spraying, or indirectly, as in flame or plasma jets. This article presents the major thermal spray processes and their subsets, presenting each of the commercially significant processes together with some of their important variations. Each process is presented along with the attributes that influence coating structure and performance. The essential equipment components and the necessary controls are also summarized. New and emerging thermal spray processes are introduced at the end of this article. Table 1 summarizes the thermal spray processes and lists some of the specific important process attributes and the resulting coating characteristics. A classification scheme for thermal spray processes can be found in the article "Introduction to Coatings, Equipment, and Theory" in this Section of the Handbook (refer to Fig. 2 in the aforementioned article).

Flame Spray

Process Characteristics. Conventional flame spray was the first thermal spray process developed (~1910) and is still in common use. Modern torches have changed little since the 1950s. Flame spray uses the chemical energy of combusting fuel gases to generate heat. Oxyacetylene torches are the most common, using acetylene as the main fuel in combination with oxygen to generate

Table 1 Thermal spray process comparisons

Attribute	Flame spray	High-velocity oxyfuel	Detonation gun	Wire arc	Air plasma	Vacuum plasma	Radiofrequency plasma
Jet							
Jet temperature, K	3500	5500	5500	>25,000	15,000	12,000	10,000
Jet velocities, m/s (ft/s)	50–100 (160–300)	500–1200 (1600–4000)	>1000 (>3300)	50–100 (160–300)	300–1000 (1000–3300)	200–600 (700–2000)	20–80 (70–300)
Gas flow, sLm	100–200	400–1100	N/A	500–3000	100–200	150–250	75–150
Gas types	O ₂ , acetylene	CH ₄ , C ₂ H ₆ , H ₂ , O ₂	O ₂ , acetylene	Air, N ₂ , Ar	Ar, He, H ₂ , N ₂	Ar, He, H ₂	Ar, He, H ₂
Power input, kW equiv.	20	150–300	N/A	2–5	40–200	40–120	40–200 (plate)
Particle feed							
Particle temperature (max), °C (°F)	2500 (4500)	3300 (6000)	N/A	>3800 (>6900)	>3800 (>6900)	>3800 (>6900)	>3800 (>6900)
Particle velocities, m/s (ft/s)	50–100 (160–300)	200–1000 (700–3300)	N/A	50–100 (160–300)	200–800 (700–2600)	200–600 (700–2000)	20–50 (70–160)
Material feed rate, g/min	30–50	15–50	N/A	150–2000	50–150	25–150	20–50
Deposit/coating							
Density range (%)	85–90	>95	>95	80–95	90–95	90–99	95–99
Bond strength, MPa (ksi)	7–18 (1–3)	68 (10)	82 (12)	10–40 (1.5–6)	<68 (<10)	>68 (>10)	>68 (>10)
Oxides	High	Moderate to dispersed	Small	Moderate to high	Moderate to coarse	None	None

the highest combustion temperatures. Powders, wires, or rods are introduced axially through the rear of the nozzle into the flame at the nozzle exit. The feedstock materials are melted and the particles/droplets accelerated toward the substrate surface by the expanding gas flow and air jets.

Figure 1 illustrates a typical powder flame spray gun. In conventional flame spray torches, a jet of fuel and oxygen is combusted at the front of the torch, external to the nozzle. In some designs, a constricting flow of air is fed through a concentric set of holes at the nozzle exit and is used to pinch the expanding gases, generating a higher-temperature combustion zone. The flame spray torch consists of gas distribution passages through the nozzle and mixing orifices at the nozzle end. Nozzles are used to shape the spray pattern. Air caps are added to wire- and rod-fed torches, producing a concentrated air jet to atomize the molten wire or rod tip. Wires or rods are mechanically fed through the center of the nozzle into the heating zone. Oxide and metal rods and wires can be used as coating feedstock. Figure 1 also illustrates this design. One significant advantage of wires and rods over powders is that the degree of melting is significantly higher, producing denser coatings. In addition, the atomizing air produces finer droplets, which in turn produce finer, smoother coatings.

In flame spray processes, fuel/oxygen ratio and total gas flow rates are adjusted to produce the desired thermal output. Optional air jets, downstream of the combustion zone, may further adjust the thermal profile of the flame. Jet gas speeds typically are below 100 m/s (330 ft/s), generating particle speeds up to 80 m/s (260 ft/s) before impact. Externally combusted, or open-flame, jet temperatures are generally above 2600 °C (4700 °F) and are controlled by the combustion temperatures of the fuel/oxygen mixtures and the mixing patterns of the combustion gases with the surrounding air. The temperature curves for fuel gases are parabolic in shape and centered roughly around the stoichiometric ratio with oxygen. Adjusting the fuel/oxygen ratio, either side of stoichiometry, will cool the flame. The reason behind the adjustment is to make the flame either oxidizing or reducing, as the feedstock material and application dictate. Flames are set to be reduc-

ing (fuel-rich) for most metal spraying in order to minimize oxidation. Generally, flame spray torches are designed to scavenge part of the oxygen needed for combustion from the atomizing air or cooling air jets. This design further helps produce metal coatings with lower oxides.

Flame spray is capable of depositing a wide range of materials, ranging from polymers to ceramics and refractory metals.

Equipment. Powder is fed into these spray torches in one of two ways: by carrier gases or by gravity. Gravity-fed devices have powder canisters or bottles mounted directly to, and on top of, the torch. Powder feed rate is controlled by a pinch valve that meters powder into the body of the torch, where it is aspirated by the gases flowing through the torch. Carrier-gas-fed units use externally mounted powder feeders. Powder feeders (which are discussed later in this article), use a carrier gas stream to transport the powder from the feeder through a hose to the spray torch. Carrier gases used for flame spray are commonly air or nitrogen.

Wire- and rod-fed devices use air turbines built into the torch that power the drive rolls, which pull feedstock from the source and push it through the nozzle.

Components. The key components of a typical flame spray system are:

- Gas supply
- Air supply
- Gas hoses
- Gas regulators for oxygen, fuel, and air
- Rotameters for gas flow control
- Flashback arrestors at the gun and regulators
- A flame spray gun comprising a torch body, nozzle, and atomized air cap
- Feedstock delivery system comprising a powder feeder, powder hopper, and air turbine drive for wires and rods

Coating Characteristics. Flame spray processes typically yield coating densities ranging from 85 to 98%, depending on the coating material and particular technique. Lower jet temperature and velocities used in flame spray have been shown to contribute to these lower deposit densities. Figure 2 shows some typical microstructures of flame sprayed metal, ceramic, and polymer coatings. Note the relatively coarse microstructures of the splats and the relatively high porosity levels. Oxide inclusions in metallic deposits result from the oxidizing condition of the flame spray process, which causes a high degree of droplet/atmosphere interaction. The polymer coating structure shows a very dense, well-bonded surface layer. This structure is a result of fusing after, or coincidental with, deposition.

Detonation Gun

Process Characteristics and Equipment. The design of conventional flame spray processes limits their ability to produce dense (<2% porosity), well-bonded coatings. Detonation guns, however, produce higher thermal and kinetic energy jets by confining the combustion within a tube or barrel into which powders are introduced. This design produces greater heat and momentum

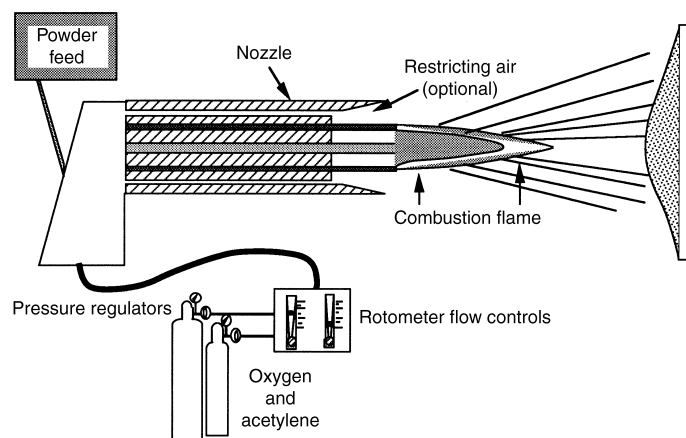


Fig. 1 Powder flame spray system

transfer to the powder particles. D-Gun (developed by Union Carbide, now Praxair Surface Technologies, Inc., Indianapolis, IN) was the first and most widely known form of detonation gun processing. D-Gun is a proprietary process; however, other forms of detonation spraying are now becoming commercially available. Detonation spray is illustrated schematically in Fig. 3.

In detonation spraying, an explosive mixture of fuel, oxygen, and powder is introduced into a long tube/barrel and ignited by a spark plug. The resulting detonation-pressure wave heats and accelerates the entrained powder particles, which travel down the water-cooled barrel toward the substrate. Nitrogen is used to purge the barrel, between detonations. The cycle of purging, injection, and detonation is repeated at a frequency of 3 to 6 Hz. Detonation spray processes are hotter and yield longer particle dwell times than conventional flame spray. The frequency and noise levels (145+ dBA) associated with detonation spraying require that they be confined to acoustical enclosures. High particle velocities, greater than 800 m/s (2625 ft/s), are generated. These high particle velocities have been shown to improve particle deformation and increase particle/coating bonding, eliminating much of the porosity typically found in conventional flame sprayed coatings.

Coating Characteristics. Figure 4 shows typical WC/Co cermet and oxide coating microstructures produced by D-Gun. Note the finer, thinner splat features compared to conventional flame spray; note also the lower oxide content of the cermet coating. Lower oxides are the result of the fine particles being protected by

the combustion gas environment of the extended barrel and the reduced dwell time. This protective environment prevents the sprayed particles from the oxidation typical in conventional flame spray in open atmosphere during heating and acceleration.

D-Gun versus Other Thermal Spray Processes. Although other processes, such as high-velocity oxyfuel and high-energy plasma, have been able to produce coatings comparable to detonation spray, D-Gun has been the standard for comparison since the late 1950s. When compared to flame and plasma sprayed coatings, it has been shown that detonation sprayed WC/Co coatings have a higher degree of retained carbides. The higher retained carbide is due to the reducing atmosphere of the confined combustion zone in the barrel and the shorter dwell time. Detonation sprayed coatings are among the densest and hardest of all thermally sprayed materials. For these reasons, many wear coatings in commercial aircraft engines are specified for the D-Gun process.

High-Velocity Oxyfuel Spray (HVOF)

Process Characteristics. The HVOF spray process was invented in 1958 by Union Carbide (now Praxair Surface Technologies, Inc.) but did not become commercially significant until the early 1980s when the JetKote (Deloro Stellite, Goshen, IN) system was introduced by James Browning. The HVOF spray process is similar, in some respects, to detonation spray. Like det-

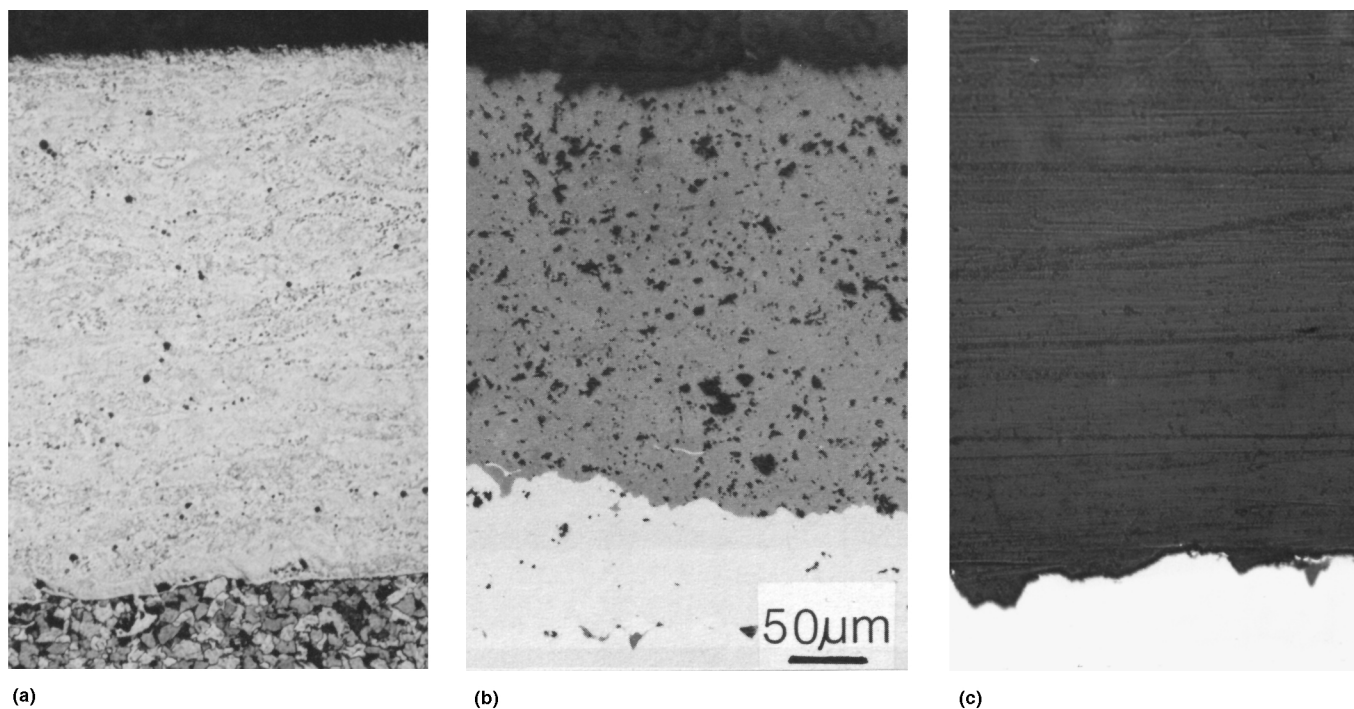


Fig. 2 Typical thermal spray microstructures, showing general coating structures. (a) NiCrBSiC/Colmonoy 72 spray and fused, flame spray coating. (b) Zirconia, plasma sprayed. (c) Polymer, flame sprayed. Courtesy of Wall Colmonoy, Plasma Technik, and UTP, respectively

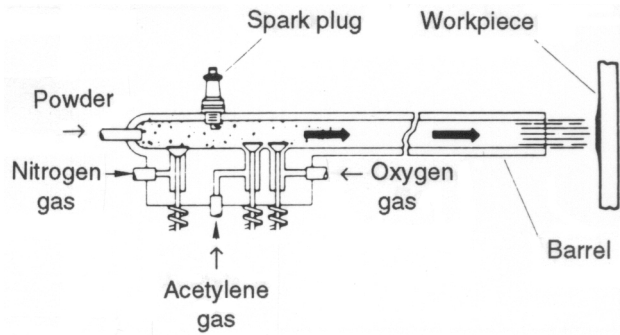
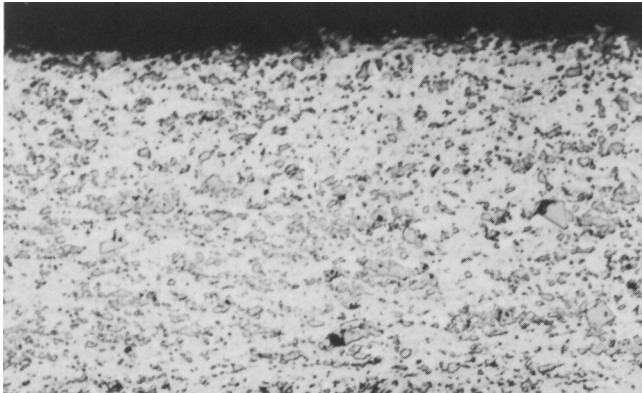
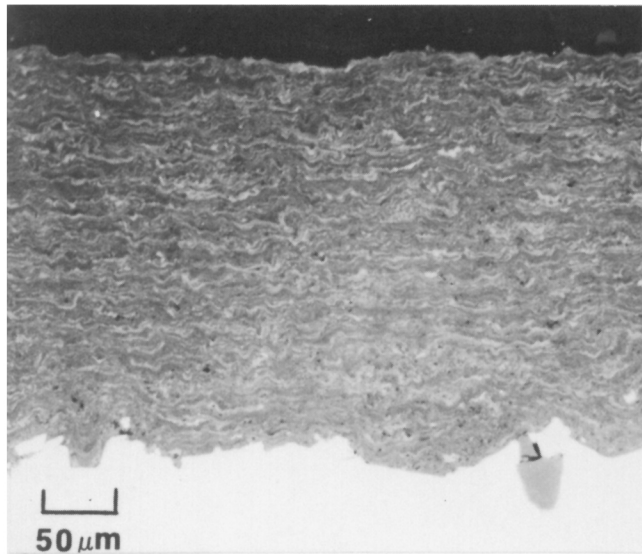


Fig. 3 Detonation gun system (D-Gun). Courtesy of Praxair Surface Technologies



(a)



(b)

Fig. 4 Typical D-Gun coating microstructures. (a) WC/Co. (b) $\text{Al}_2\text{O}_3/\text{TiO}_2$. Courtesy of Praxair Surface Technologies

onation spray, HVOF has an extended internal-confined combustion. Unlike detonation spray, however, HVOF operates on a continuous, steady-state basis.

In principle, high-volume combustible gases are fed into a combustion chamber, feeding into an 8 to 30 cm (3 to 12 in.) long confining nozzle (barrel) through which the combusting gases exit the device. The sheer volume of gas flow, coupled with the high temperature of combustion, creates gas velocities in the 1525 to 1825 m/s (5000 to 6000 ft/s) range at the nozzle exit. High-velocity oxy-fuel gases used include hydrogen, propylene, propane, acetylene, and kerosene; trademark gases such as MAPP (methylacetylene-propadiene-stabilized gas), Apache, and Crylene are also used.

It is important to note that there are two distinct classes of high-velocity combustion spray devices, divided according to their combustion chamber pressure. The first class, better fitting the term *high velocity*, is characterized by chamber pressures exceeding 241 kPa (35 psi) and heat inputs of nominally 527 MJ (500,000 Btu). The second class would be better termed hypervelocity, operating nominally at 620 to 827 kPa (90 to 120 psi) with ~1 GJ (~1 million Btu) input, and is typically fueled with kerosene. Kerosene-fueled guns use either air or oxygen to support combustion.

High-velocity oxyfuel guns have air- or water-cooled combustion chambers that combust the fuel/oxygen mixtures under pressure and accelerate the resulting flame/gas stream down a confined, cooled tube or nozzle. Powders, borne by a carrier gas, are fed into the nozzle, where particles become entrained into the confined, high-pressure flame/jet.

Figure 5 illustrates the basic features incorporated into an internal combustion HVOF design: water cooling, particle injection, and a nozzle to produce supersonic expansion of the combusting gases. Particle heat transfer and efficiency increase with dwell time by this design. The high gas velocity generated by HVOF, much higher than in conventional flame or plasma spray, has been shown to increase particle velocity, with a corresponding increase in coating density and coating adhesion. Lower average particle temperatures, compared to plasma spray, reduce the degree of par-

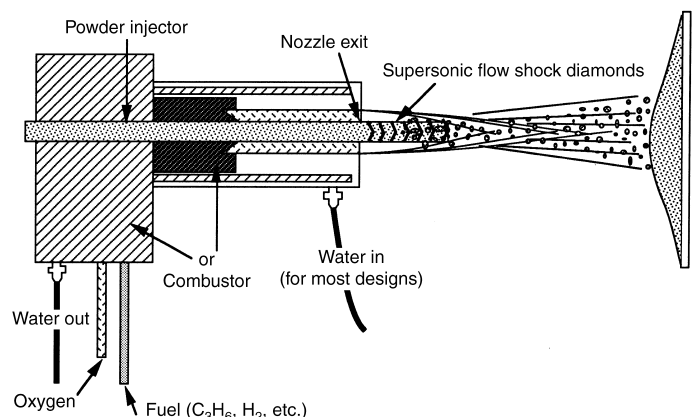


Fig. 5 High-velocity oxyfuel gun features

ticle melting and oxidation. Despite the lower average particle temperatures, high coating densities are still achieved through HVOF high particle impact velocities, which deform particles that may not have been well melted. Some particle heating occurs on impact from the conversion of kinetic energy into thermal energy, which further aids in producing dense coatings.

Generally, HVOF processes are used to deposit dense, hard cermet coatings of WC/Co or $\text{Cr}_2\text{C}_3/\text{NiCr}$. Many uses have been found, however, for HVOF sprayed metallic coatings. MCrAlY coatings are being applied to aircraft turbine blades by oxy-kerosene HVOF spray.

Equipment. Apart from the guns themselves, HVOF process equipment is similar to that used for conventional flame spray. However, HVOF guns that employ internal combustion require active cooling systems.

Components. Key HVOF components include:

- Oxygen, fuel gas, and powder carrier gas circuits consisting of a high-volume gas supply, high-pressure gas hoses, high flow gas regulators for oxygen and fuel, high flow gas controls, and flashback arrestors at the gun and regulators
- An HVOF spray gun comprising a torch body, combustion chamber, powder injector, and nozzle
- Water- or air-cooling circuits
- Feedstock delivery: high-pressure powder feeder
- Safety interlocks and console purging

Kerosene-fueled guns require additional controls, such as liquid fuel pumps and high-volume air, to support combustion and/or cooling for some gun designs.

There are several important differences between conventional flame spray and high-velocity guns. Cooling circuits are the most obvious difference. The high enthalpy of HVOF guns results in high thermal stresses and oxidation of gun components, particularly the combustion chamber and nozzle. Appropriate choice of component materials and attention to design are necessary to prevent water from boiling in the gun. Combustion chamber materials need to be resistant to oxidation, particularly in kerosene-fueled devices. Nozzles are typically oxygen-free, high-conductivity (OFHC) copper or high-copper alloys that approach OFHC properties. Because of the high enthalpy and velocity of the jet, the nozzles are typically polished internally to prevent powder buildup within the nozzle. Water-cooling passages are designed to minimize pressure drops, which would result in rapid boiling of the cooling water. (Boiling reduces heat transfer to the water, resulting in oxidation of the gun components, which further reduces heat transfer. Reduced heat transfer on the water side of the component usually results in powder buildup and nozzle melting on the flame side.) Powder feed rate control is also critical in HVOF processes, more so than in conventional flame spray. This is due primarily to the fact that HVOF devices are more sensitive to the volume of powder being injected into them. In high-velocity (low-pressure) designs, volumetric feed rate is limited to approximately 6 to 9 cm^3/min (0.37 to 0.55 $\text{in.}^3/\text{min}$).

High-velocity oxyfuel guns are separated into four major designs:

- Axially aligned combustion chambers and nozzles, where gases are supplied through a mixer into the combustion chamber. Carrier gas and powder particles are injected into the combustion chamber through a powder port in the center of the mixer face.
- Combustion chambers that feed hot gases through a right-angle connector into the nozzle. Powder is injected through a port in the rear face of the connector, which centers the powder stream within the nozzle.
- An adaptation of conventional flame gun design, where combustion may or may not be confined by a water-cooled nozzle into which a stream of powder with carrier gas is injected
- High-pressure, kerosene-fueled systems with radial injection of powders downstream of the combustor exit

Figure 6 illustrates these various designs, showing the major differences between them. (Compare these with the detonation process shown in Fig. 3.) Each design has both strengths and limitations. All of the designs produce high-quality WC/Co coatings. The axial design shown in Fig. 6(a) is particularly well suited for spraying ceramic materials and chromium carbide. The designs depicted in Fig. 6(b) to (d) are better suited for spraying metals.

As mentioned previously, gas flow rates are much higher in HVOF processes than in conventional flame spray. In low-pressure HVOF, hydrocarbon fuel gas flows are nominally 66 sLm (140 scfh), with oxygen flows of nominally 283 sLm (600 scfh). Hydrogen flow rates are typically in the range of 660 to 850 sLm (1400 to 1800 scfh). In high-pressure systems, kerosene is fed at nominally 23 to 26 L/h (6 to 7 gal/h), with oxygen at nominally 944 sLm (2000 scfh) or air at 4.2 m^3/min (150 scfm). The high-velocity gas flows, coupled with longer nozzles, accelerate the gases and powder particles to velocities much closer to the gas velocity than in other thermal spray processes.

The gas exit velocities are so high that most HVOF processes exhibit clearly noticeable “shock” diamond patterns in the gas jets, characteristic of the supersonic nature of the jet. These shock patterns are similar to those seen in high-performance jet-engine exhausts using afterburners. The number of shock diamonds, although associated with gas velocity, is not an accurate indication of the Mach number (the gas velocity relative to the local velocity of sound). These extreme velocities also generate extreme noise. Noise levels for low-pressure HVOF systems are ~125+ dBA (typical of high-energy plasma), whereas the high-pressure HVOF systems (most notably, kerosene-fueled guns) operate at 133+ dBA.

Coating Characteristics. High-velocity oxyfuel coating properties are reported to be comparable to those of detonation gun coatings, particularly for carbide and oxide coatings. High-velocity oxyfuel sprayed coating density, adhesion, and oxide contents also compare favorably with high-energy, plasma sprayed coatings. High-velocity oxyfuel coating bond strengths usually exceed ASTM C 633 (“Standard Test Method for Adhesion or Cohesion”), which is limited by the adhesive to ~83 MPa (~12 ksi). Porosity is typically reported at less than 1%. Compositional analysis of HVOF WC/Co coatings show that, compared to

plasma spray, only a small amount of WC decomposition (to W_2C) occurs, preserving the intrinsic high hardness values of the material. Table 1 summarizes the attributes of HVOF, including the higher particle velocities and the lower particle temperatures, compared to conventional flame spray and plasma spray. One characteristic of HVOF processes, particularly the high-pressure, kerosene-fueled guns, is some control of residual stress. Parameters that generate high combustion chamber pressures have a strong tendency to produce compressive stresses in the coatings, which are beneficial to the function of most coatings. However, high residual stress comes with a price. The deposition efficiency (DE) of kerosene-fueled HVOF guns is much lower than that of conventional HVOF guns. The DE for conventional HVOF guns range from 50 to 70%, whereas the DE for kerosene-fueled guns

ranges from 35 to 50%. It would appear that the upper (coarser) portion of the feedstock powder size distribution is lost. It is also apparent, from the DE and microstructure, that the larger particles play some part in peening the coating, which helps to produce the compressive residual stresses. Figure 7 shows some examples of HVOF coating microstructures, depicting the finer oxide dis-

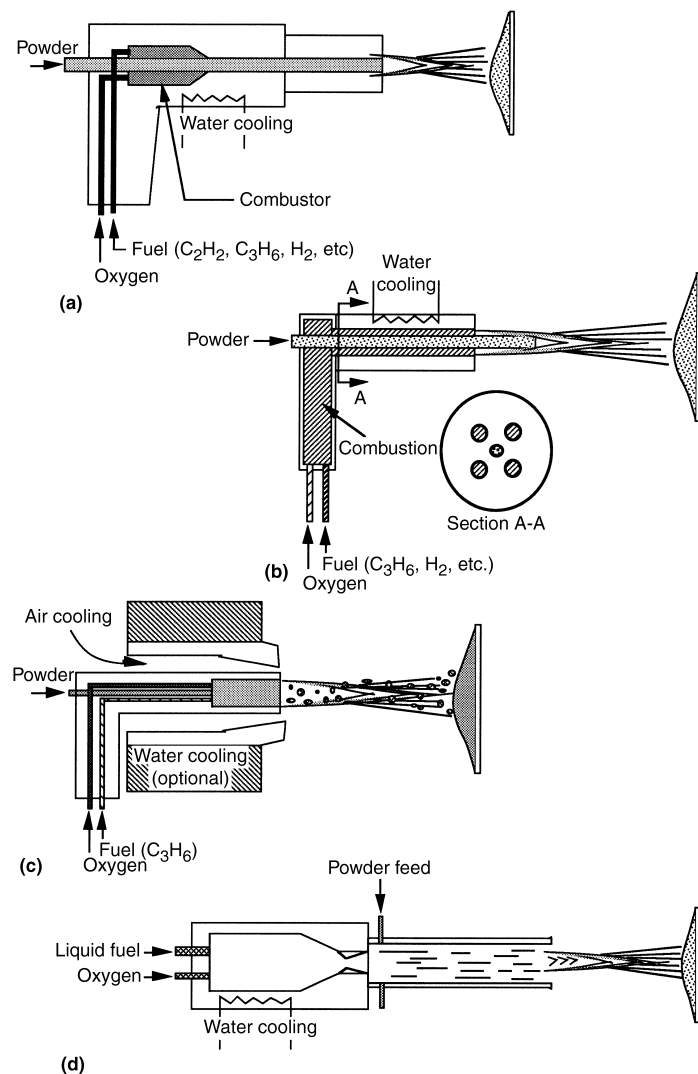


Fig. 6 Operational principles of several commercially available high-velocity oxyfuel thermal spray guns. (a) HV-2000. (b) JetKote. (c) Diamond Jet (D-J). (d) JP-5000

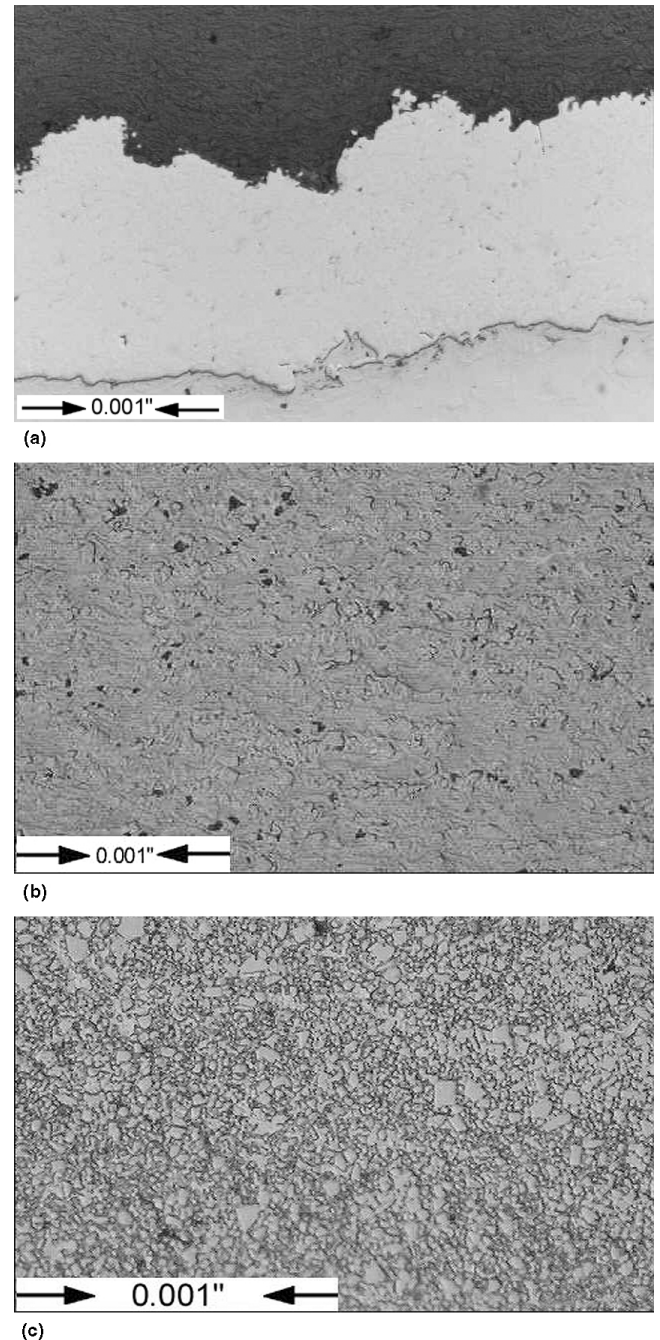


Fig. 7 Typical high-velocity oxyfuel coating microstructures. (a) Nickel. (b) Alumina. (c) WC/Co. Courtesy of Thermal Spray Technologies

persions, denser and finer splat structures, and the high carbide retention.

Electric Arc Spray

Process Characteristics. The consumable, two-wire electric arc spray (also called twin-wire arc, arc spray, or wire arc spray) method was originally developed by M.U. Schoop in approximately 1910, but the process did not gain wide commercial acceptance until the early 1960s. (The historical development of thermal spray processing is discussed in the article “Introduction to Thermal Spray Processing” in this Handbook.) Unlike the other thermal spray processes, which indirectly heat the particles using heated gas jets, electric arc spray processes use a direct-current (dc) electric arc, struck between two consumable electrode wires to effect direct melting. An electric arc is formed in the gap between the wire tips as the two wires are continuously fed together. The process is shown schematically in Fig. 8. Because the wires are melted directly by the arc, the thermal efficiency of the electric arc spray process is considerably higher than that of any other thermal spray process. A high-velocity air jet located behind the intersection of the wires shears away the molten metal that continuously forms as the wires are fed into the arc and melted. The high-velocity airflow ranges from 0.8 to 1.8 m³/min (30 to 65 scfm). As the molten metal shears from the wires, it atomizes, or breaks up, the material into finer particles, creating a fine distribution of molten metal droplets. The atomizing air is also used to accelerate the particles toward the substrate surface, where the molten particles impact, deform, and solidify to build up a coating. Unlike flame or plasma spray, however, the droplets are already molten when the material is picked up and entrained in the jet, and unlike other processes, the particles begin to cool immediately after leaving the arc zone. In order to minimize oxidation in the electric arc spray process, the dwell time needs to be shortened by using short standoff distances and high atomizing air flows. Figure 8 illustrates the continuous stream of molten metal leaving the wire tips that are broken up by the air jets.

Electric arc spray transfers less heat to the substrate per unit of material sprayed than any other conventional thermal spray process, owing to the absence of a flame or plasma jet. The only heat input to the part is the “sensible” heat borne by the individual particles in the spray stream.

Particles generated by electric arc spray are generally of similar size and distribution as the powder feedstocks used in flame and plasma spray but are biased or skewed toward the coarser end of the distribution. Particle size and distribution are, however, functions of the operating parameters used. Particle size increases as current increases, as voltage decreases, as air pressure/flow decreases, or as wire size increases. Conversely, particle size decreases as current decreases, as voltage increases, as air pressure/flow increases, or as wire size decreases. It has been observed that droplet (particle) sizes range from submicron (fume) to ~200 μm, depending on the process parameters; the droplets are usually spherical in shape. Atomizing irregularities are a function of wire feed uniformity, arc voltage and current stability, variability of the arc gap, and the length of wire that protrudes from the wire guides (contact tips). Variability in the arc gap at the wire tips can be caused by several factors, including permanent sets in the wires (cast and cant or helix), wear, and/or misalignment of the wire guides.

Material feed rates in electric arc spray are relatively high in comparison to other thermal spray processes. Table 2 gives typical feed rates for common spray wires.

Materials for electric arc spray are limited to conductive materials that can be formed into wires. The use of cored wires has, however, expanded the range of materials to include cermets and amorphous materials. Cored wires consist of a tubular metallic sheath with powder in the core. These powders are usually carbides or amorphous alloys. Cored wires can also be made by wrapping a smaller-gage wire with a sheath of a dissimilar alloy.

Equipment. A typical electric arc spray system consists of the following:

- Constant-voltage, dc rectifier/power supply
- Compressed air supply
- Wire feeder comprising an electric or air-driven motor, drive wheels, and drive wheel tensioning devices

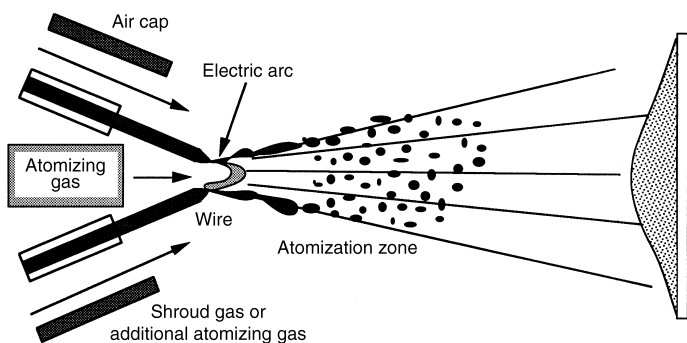


Fig. 8 Electric arc spray process

Table 2 Electric arc spray rates for various materials

Wire	g/min (lb/h)/100 A dc
Aluminum	45 (6)
Babbitt	379 (50)
Brass	83 (11)
Copper	83 (11)
Molybdenum	76 (10)
Steel	76 (10)
Stainless steel	76 (10)
Tin	341 (45)
Titanium	23 (3)
Zinc	182 (24)

- Electric arc spray gun comprising a gun body, air nozzle, wire guide/contact tips, air cap, and arc shield
- Hoses/cables

Arc stability is governed by many factors, including power supply design, wire straightness, wire guide tolerances, and constancy of wire feed. Power supply design limits most systems to operating above 50 A dc; very few designs permit operation below 25 A dc.

Wire straightness is expressed in terms of cast (a permanent set in the hoop direction) and cant or helix (a permanent set off-axis to the hoop direction). When the wire has too much set in either direction, the wire ends will rotate as they exit the wire guides. This rotation causes the wires to miss the intersection, causing an interruption in the arc, sometimes called “spitting.” Gun motion during spraying accentuates the problem of wire rotation. Quality-control checks for cast and cant are relatively simple. A 3 to 4 m (10 to 12 ft) length of wire is cut from the wire spool and allowed to free fall to the floor or work surface. The wire will naturally open as any tension is released. The diameter of the loop that the wire forms is an indication of the residual stress or cast in the hoop direction. The height above the floor of the loose end of the wire is an indication of the residual stress or cant off-axis to the hoop direction. Acceptable numbers for these dimensions have to be determined empirically or obtained from the supplier. Values vary greatly as a function of material, processing, and annealing.

Alignment and wear are the two most significant problems with wire guides. Misalignment or wear will also cause the wires to miss their intersection, causing an interruption in the arc and spitting.

Poor tension control in the wire feed mechanism causes one or both wires to slip in the drive rolls, resulting in arc outages and spitting. Several feed roll designs have been developed to better grip the wide variety of materials sprayed. Among the many designs are V-grooved/knurled rollers for soft and very hard wires and U-grooved rollers for common wires.

Wire feeding schemes include feeders that pull only, push only, and combination push/pull designs. Pull-type drives have electric or air turbine motors mounted in the gun. These units pull wires from the wire spools through conduits into the gun. The distance that wires can be pulled depends largely on the torque of the drive motor, the type of feed rolls, and the endurance of the operator. Push systems have lighter guns, because the drive unit is remote from the gun, coupled by the conduits. The distance from the feeder to the gun is determined by the column strength of the wire. Softer or smaller-diameter wires are more difficult to feed than larger or harder ones. Figure 9 shows a push-type system (a) and gun (b).

Combination push/pull systems can relieve some of the distance limitations of either push-only or pull-only devices. Push/pull designs make use of both a drive motor in the gun and a drive motor at the wire source. Generally, push-only and pull-only drives are limited to ~6 m (~20 ft), whereas push/pull designs enable wires to be fed more than 15 m (50 ft). One drawback of push/pull drives is the difficulty in synchronizing the two motors. Lack of synchronization leads to frequent failure of drive motors, which significantly affects coating costs. Figure 10 illustrates the gun part of a commercial push/pull device, where the wires are

pulled by an electric motor in the spray device. The other end of the conduit would be connected to a push motor drive unit.

The atomizing air flow also serves as cooling air for the wire guides or contact tips. Clean, dry, compressed air is required for prolonged life and continuous operation. Most systems require up to 1.8 m³/min (65 scfm) at up to 690 kPa (100 psi). Oil and moisture in the air lead to coating oxidation, substrate contamination, and coating inclusions.

Air caps and arc shields are typically fitted around the front of the gun to protect the operator from direct line of sight to the high-intensity visible and ultraviolet light of the arc. Air caps also provide shaping of the air jet to control the velocity and spray pattern of the particle stream.

Power supplies used for electric arc spray are modified rectifier-type constant-voltage power supplies used for welding. Typical operating ranges are 15 to 400 A dc and 50 to 250 A dc, with open-circuit voltages of ~40 V dc. Few units use inverter-type power supplies, because inverters require specialized circuitry to prevent shorting of the wires at the arc zone. High throughput systems used primarily for corrosion coatings operate between 400 and 1500 A dc. The larger of these types of units requires water cooling for the contact tips.

Coating Characteristics. The cooler deposit characteristic of electric arc spray minimizes the substrate heating associated with other thermal spray processes. This gives electric arc spray a major processing advantage for applications where coating/substrate temperatures need to be kept low. This enables the electric arc spray process to deposit coatings onto polymers, fiberglass, wood, and paper products, as well as metallic or glass substrates. Typical electric arc spray microstructures are shown in Fig. 11.

Electric arc spray coating microstructures can be varied widely by varying the operating parameters. The average electric arc spray coating is characterized by splats that are thicker and more varied in size than those seen in wire flame and plasma spray coatings, and with a higher oxide content. In powder-fed processes, unmelted material is often seen in the coatings. Similar-looking features are seen in electric arc spray microstructures, but in this case, they are resolidified particles, that is, particles that melted from the wires and resolidified before impact.

Electric arc spray microstructures can be improved by:

- Using smaller-diameter wires, deposited at lower feed rates (current)
- Using high-velocity air caps to reduce dwell time
- Using inert atomizing gases to reduce oxidation
- Reducing standoff distance to minimize oxidation
- Reducing the arc voltage to minimize overheating of the droplets

Figure 12 illustrates some of these effects. Note that the splats and oxide stringers are somewhat thinner and the porosity levels are lower than those typically seen in flame and some plasma sprayed coatings. These attributes can be associated in part with the higher droplet temperature and shorter dwell time. Inert-chamber electric arc spraying has been developed; spraying in an inert gas further reduces oxides. Spraying with inert gas, instead of compressed air, also reduces oxide content. Figure 13 depicts some of the

improvements in microstructure that have been made by using inert gas electric arc spray. Note that deposit oxide inclusions in these chamber or inert gas coatings are virtually eliminated.

Electric arc spray occupies an important niche in thermal spray technology and is particularly suited to high-rate deposition. Specific areas of interest for this unique attribute are municipal and civil structures such as steel structures, bridges, water and storage tanks, and marine coatings for ship stacks and decks. In the case of zinc coatings, the somewhat porous nature of this cathodic coating has led to excellent corrosion protection, particularly when impregnated with epoxy paints, which seal open porosity. Electric arc spray coatings can be made much rougher than the other thermal spray processes, which makes them well suited for use as non-skid surface coatings. As previously discussed, electric arc spray produces a relatively cool spray stream and has found application

as a coating process for low-temperature-capability substrates such as polymers and even paper.

Plasma Spray

Process Characteristics. Plasma, often called the fourth state of matter, normally consists of neutral atoms, positive ions, and free electrons. Plasma is produced by transferring energy into a gas until the energy level is sufficient to ionize the gas, allowing the electrons and ions to act independently of one another. The plasma state is achieved when, under an electric field, currents can be sustained as the free electrons move through the ionized gas. Once the energy input is removed, the electrons and ions recombine, releasing heat and light energy.



(a)



(b)

Fig. 9 Push-type electric arc spray components. (a) System with door open showing drive components. (b) Gun. Courtesy of Thermach, Inc.

It has been estimated that ~98% of the visible universe exists in the form of plasma. Natural plasmas occur on earth as the aurora borealis, aurora Australis, St. Elmo's fire, and lightning. Common man-made plasmas include neon and fluorescent tubes and bulbs. The plasmas under consideration in this article are less common and are part of a class called thermal plasmas or plasma arcs.

Thermal plasmas are the state where the electron population (very low mass) and the heavy particle (high mass) or ion population have similar energy levels. The higher-mass ions transfer significantly more kinetic heating energy in the gas than the electrons during collisions. High-electron-temperature plasmas, although energetic, do not impart much thermal energy transfer due to their lower-mass particles. Energy sources for the thermal plasmas used in thermal spray devices are usually dc, electric arcs, or RF (sometimes called induction-coupled plasma) discharges. Thermal plasmas can also be generated using microwaves, gamma radiation, and alternating current (ac) electric arcs.



Fig. 10 Commercial electric arc spray gun from a push/pull system. Courtesy of Praxair Surface Technologies

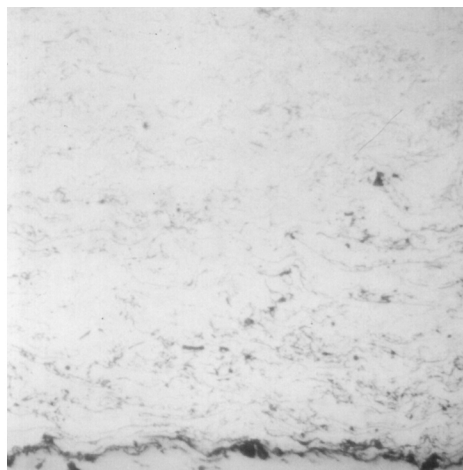
In a dc plasma arc, gas heating is sufficient to generate core plasma temperatures exceeding 20,000 °C (36,000 °F), depending on the gas properties and its electrical breakdown characteristics. Figure 14 graphs energy density, or enthalpy, values of several typical plasma spray gases. Gas enthalpy indicates the heating potential for a gas as it is heated to the plasma state. The monatomic gases, argon and helium, simply ionize, generating their respective ionic species, Ar^+ and He^+ , and their related free electrons. Figure 14 plots the energy content, or enthalpy, of the gases as a function of temperature. Note that the diatomic gases, nitrogen and hydrogen, have higher enthalpies for a given excitation temperature, which is related to the dissociation of their molecular structure, followed by ionization.

Direct-current plasma arc spray is the most flexible of the thermal spray processes with respect to materials that can be sprayed, because of the inert gases, high gas velocity, and extremely high temperatures. The basic components and design of a dc plasma arc gun are illustrated in Fig. 15 and 16. Several commercial versions, shown in Fig. 17 to 19, illustrate the different designs.

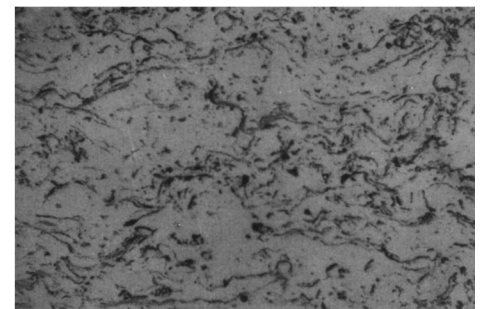
The dc plasma arc gun combines an axially aligned cathode (typically, 1, to 2% thoriated tungsten) and an anode/nozzle (typically, OFHC copper). Tungsten is used as a cathode for two reasons: first, it has a high melting point (>3500 K), and second, it is a good thermionic emitter, readily releasing electrons to maintain the arc discharge. Typically, the thoriated tungsten tip operates at or near its melting point, due to high electron current densities, which locally heat the cathode tip. A water-cooled copper anode/nozzle constricts and spatially stabilizes the arc and accelerates the expanding, heated gases. Cathodes rely on conduction and the high melting point of tungsten to survive the high-temperature arc, because they are not directly water-cooled. Direct-current plasma arc guns are intensely water cooled to prevent melting and to minimize cathode and nozzle erosion.



(a)



(b)



(c)

Fig. 11 Typical microstructures of electric arc sprayed coatings. (a) Aluminum. (b) Zinc. (c) NiAl. (a) and (b) courtesy of Miller Thermal; (c) courtesy of Hobart TAFE

Plasma forming gases are introduced through the rear of the gun. Typically, these gases enter the arc chamber through a gas ring (gas injector) that imparts a spin or vortex flow to the gas. The vortex stabilizes the arc at the cathode tip in the low-pressure

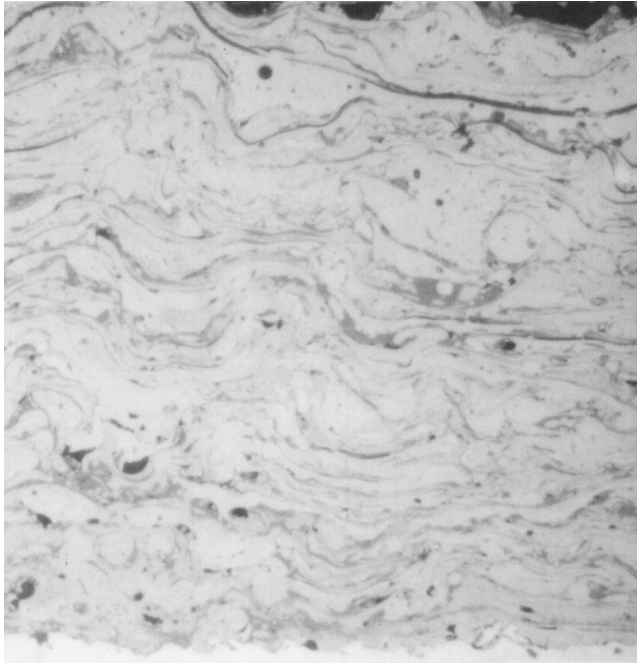
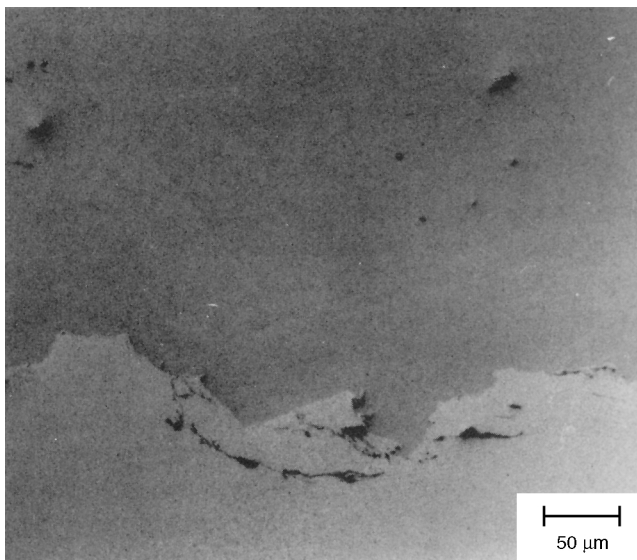


Fig. 12 Electric arc sprayed steel coating using smaller-diameter wires. Courtesy of Miller Thermal

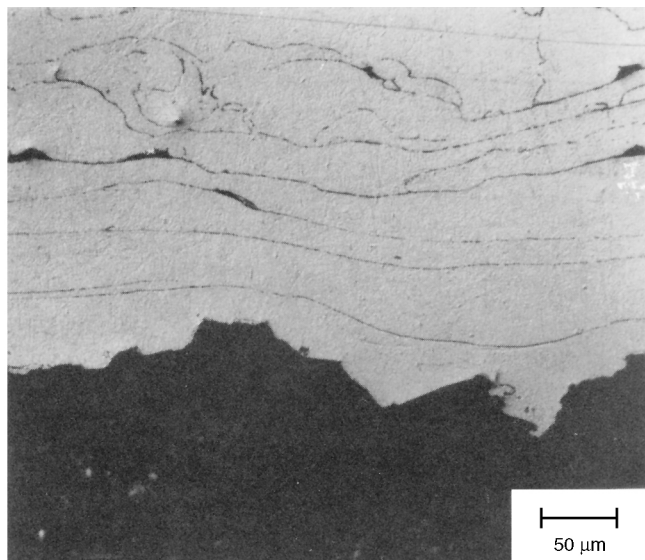
region of the vortex and rotates the arc attachment or arc root at the anode. This is called vortex stabilization. Rotation of the anode arc attachment minimizes arc erosion of the anode. The arc gases are heated as they pass around and through the arc. The heated gases expand radially and axially, accelerating as they expand and exit through the nozzle. Depending on the pressure ratio between the upstream arc region and the downstream nozzle exit of the gun, the gases (plasma jet) either expand subsonically or supersonically. Many gun designs use a converging/diverging (de Laval) nozzle to achieve supersonic expansion, which increases particle velocity. Other designs intentionally slow the plasma to subsonic velocities to increase dwell time.

Equipment. Plasma arc spray systems, illustrated in Fig. 20, consist of components similar to other thermal spray systems. The main components are:

- Gas supply
- Gas hoses
- Gas regulators for plasma and powder carrier gases
- Gas flow controls, usually rotameters or mass flowmeters/controllers
- Plasma arc spray gun comprising a torch body, anode/nozzle, cathode, gas ring (gas injector), and powder injector
- Electrical controls for arc current and voltage
- Direct-current power supply, typically a constant-current type
- High-frequency RF spark arc ignition source
- Water-cooling circuit
- Feedstock delivery: powder feeder
- Water-cooled power cables
- Hoses/cables
- Safety interlocks and console purging



(a)



(b)

Fig. 13 Titanium sprayed in inert chambers. (a) Etched. (b) Unetched

Direct-current plasma arc spray systems are the most complex and most flexible of all thermal spray processes. Plasma gun designs range from 144 to 720 MJ (40 to 200 kW) input power. These high power levels result in electrode erosion. As mentioned previously, water cooling is essential, because of the high temperatures and enthalpies. Typically, water pressures exceeding 1 MPa (150 psi) are needed to suppress localized film boiling in the gun, which would reduce component life and cause coating contamination. This necessitates the use of high-pressure O-ring seals and passageways inside the gun. Ceramic gas injectors and high-temperature insulators are required to separate the electrodes; these components require sealing from gas and water passages. The high power density of dc plasma arcs can produce relatively high erosion rates of the anodes and cathodes. Gun design is a significant factor in component life. Designs that use a closely coupled anode and cathode (typically, a welding-electrode-type design) experience shorter lives than those that use a wide spacing and a flow nozzle. The differences in usable life are tens of hours versus hundreds of hours. Operational procedures, particularly startup and shutdown sequences, significantly affect electrode life. The use of monatomic gases also extends electrode life, due to the lower enthalpy. Arc erosion of the anode and powder erosion of powder ports and powder feed tubes are factors in anode/nozzle replacement. The cathode is generally replaced after every two to five nozzle changes, depending on the cathode geometry and current density. Gas injectors, for introducing arc gases, generally fail from thermally induced cracking, due to arc heating.

Powder ports are a significant factor in coating consistency. Diameter, angle with respect to the plasma jet, axial position along the jet, radial distance from the jet centerline, and geometry all affect particle trajectories. Particle heating is determined by parti-

cle trajectory; slight changes in particle trajectory distributions can cause significant changes in the operation of the gun and in coating quality. Adjustable and removable powder ports need to be monitored and controlled. Alignment tools should be used to ensure precise and repeatable positioning when powder ports are installed, replaced, or adjusted. This is more of a problem with externally injected plasma guns than with internally injected guns. External-feed designs separate the functions of the nozzle from powder feeding; therefore, powder-port alignment is independent of the anode and must be maintained separately. Internally fed nozzles have integral powder ports, which can be eroded by the powder before the anode itself is spent. Internal powder-port erosion rates are a function of the materials being sprayed. Some anode/nozzles incorporate wear-resistant powder-port inserts to extend their lives and are used for long-life nozzles. Erosion of a powder-port bore by just 25 μm (0.001 in.) can reduce the powder-injection velocity by as much as 5%, which can significantly affect the operation of the gun and the quality of the coating.

Arc-gas flow control to the plasma torch is accomplished by rotameters, critical orifices, or mass flowmeters/controllers. Rotameters are rapidly falling into disuse in today's thermal spray environment due to their inherent inaccuracy. Critical orifices and mass flow devices are the most accurate and reliable and are used in the more advanced control systems. Carrier gas control, which affects particle trajectory, is equally critical. Gas flow control is discussed in the article "Process Control Equipment" in this Section of the Handbook.

Water-cooling circuits operate at pressures of 1.0 to 1.7 MPa (150 to 250 psi) and flow rates of 11 to 45 L/min (3 to 12 gal/min). Water is generally monitored only for minimum flow or pressure. As with HVOF guns, internal water passages are designed to min-

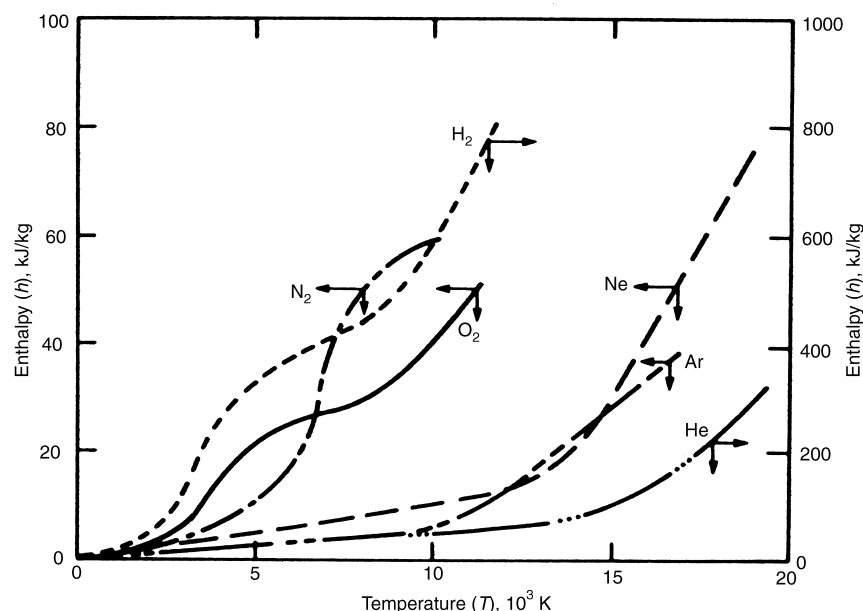


Fig. 14 Enthalpy values of typical plasma spray gases as a function of temperature. Source: Ref 1

imize pressure drops, which would result in rapid boiling of the cooling water. Boiling reduces heat transfer to the water, resulting in oxidation of the gun components, which further reduces heat transfer. Reduced heat transfer on the water side of the component usually results in melting of the anode on the plasma side. Heat transferred through electrode surfaces to the water circuit decreases the electrical efficiency of the process by reducing the power available for jet heating. The actual plasma stream energy is an extremely important parameter, which is monitored in advanced control systems. The electrical efficiency of commercial plasma guns is between 42 and 65%.

Constant-current, dc power supplies (rectifiers) have been specifically developed for producing low-ripple, constant-current power for plasma arc spraying. Older saturable-core-reactor-type power supplies with selenium or silicon-diode rectifiers have largely been replaced by silicon-controlled rectifier (SCR) power supplies. Plasma current would drift with process changes in the older units. The SCR-type power supplies produce less ac ripple and react faster to the dynamic changes in the arc. The newer designs have a higher power factor, a cost advantage in areas of the world where power consumption is an issue. The SCR-based designs are also more tolerant of line voltage variations. Power-supply ripple frequencies (360 to 720 Hz) can be on the order of the particle dwell time (1 to 3 ms). Thus, some particles may miss the peak of the plasma temperature profile by following the power nodes created by the ac ripple. This can potentially increase the number of unmelted particles in a coating. Inverter power supplies that operate at 30 to 45 kHz are being used on a limited basis, with some success. Voltage-stable, constant-current power supplies are needed to ensure stable plasma coating processes.

Plasma gun development has focused either on increasing power and gas/particle velocity or dwell time in order to increase particle heating and kinetic energy, such that particles impact, deform, and densify more completely. In addition, higher particle speeds have been found to minimize oxidation. With the advancement of technology, average coating densities and bond strengths are increasing, and average coating oxide contents are decreasing.

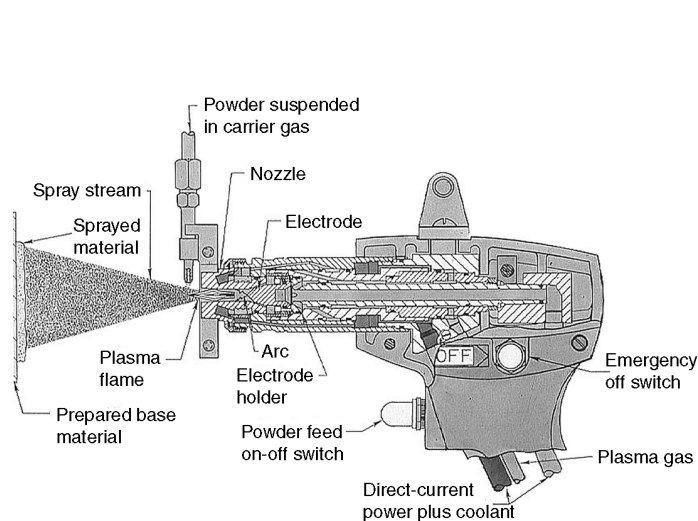


Fig. 15 Cross section of an external injection plasma spray gun. Courtesy of Sulzer Metco

Plasma spray gun designs vary widely. Some of the differentiating characteristics are:

- Plasma generator: dc versus RF
- Operating power levels: 20, 40, 80, 120, and >200 kW
- Gas velocity: subsonic versus supersonic
- Arc stabilization: gas or water, axial or vortex flow
- Powder injection: internal axial, internal radial, or external radial, as shown in Fig. 15, 16, and 21

Powder-injection geometry and conditions strongly influence the degree of particle heating/melting, thereby controlling coating porosity and oxide inclusion levels. The combination of carrier gas flow rate and powder-port diameter determine the carrier gas injection velocity, which determines the particle-injection velocity and, in part, the trajectory. Particle velocities combine with particle mass to directly control particle momentum; this determines the path, or trajectory, that an injected particle takes into and through the plasma jet. Powder-injection angles, both axial and circumferential, determine the initial particle-injection conditions, which, when acted on by the plasma flow, determine the final powder-particle trajectory. For example, upstream particle-injection angles increase particle dwell time and interaction with the plasma jet, yielding higher particle temperatures. Downstream injection angles yield the opposite effects.

Figure 21 illustrates the various trajectories that particle distributions may follow. Each trajectory shown represents extremes in particle heating, and this affects the average condition of the particles on impact (liquid, solid + liquid). A fortunate law of nature is that the smaller-diameter particles do not have enough momentum to penetrate the hottest portion of the jet core. Therefore, smaller particles are not heated as long as the larger ones; larger particles, with their higher momentum, penetrate to the jet core. High carrier gas flows (velocity) may force larger particles to follow a trajectory that passes through the jet to the opposite side of

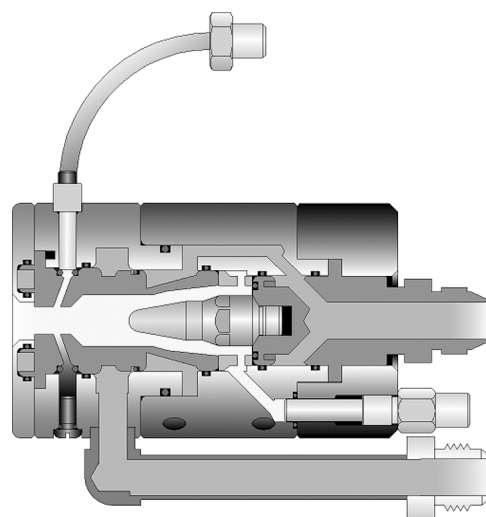


Fig. 16 Cross section of an internal injection plasma spray gun. Courtesy of Praxair Surface Technologies

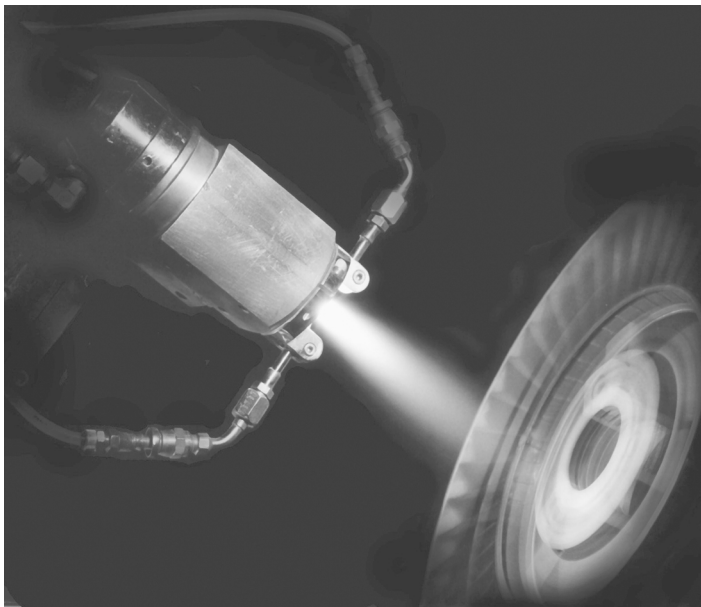
the plasma. This would result in decreased heating and possibly cause a buildup of powder on the nozzle bore.

Internally fed plasma spray guns provide better particle heating due to the particle trajectory, longer dwell time, and hotter point of injection. Internal injection has also been found to enhance particle-heating uniformity. One drawback to internal powder feed is an intolerance of poorly classified (sized) powder. Narrow powder particle size distributions are preferred for internal injection.

External powder feed, on the other hand, injects particles after the plasma jet exits the nozzle and begins to expand, so the particles do not have such long dwell times. Jet exit temperatures still

exceed 12,000 °C (21,600 °F); thus, if particle trajectories are optimized, there is sufficient heating to melt most materials and produce acceptable coatings. However, DEs are lower for external injection.

The most significant effect of the different powder-injection schemes is on particle dwell time. Reduced dwell times occur with external feed, resulting in higher powder-feed sensitivity. Overall, the powder sizes used for plasma spray fall between 10 and 100 μm (0.4 and 4 mils), with a preferred fraction generally between 10 and 45 μm (0.4 and 1.8 mils). Coarser particle sizes, particularly for higher-melting-point materials, do not experience suffi-



(a)



(b)



(c)



(d)

Fig. 17 Plasma spray devices developed by Praxair Surface Technologies. (a) PlazJet. (b) SG-100. (c) SG-200. (d) 2700 Mini-Gun III

cient dwell time to melt, while smaller particles with lower momentum cannot penetrate the jet.

The distributions of gas temperature and velocity in plasma jets range widely with anode/cathode design, power, arc-gas flow rate,

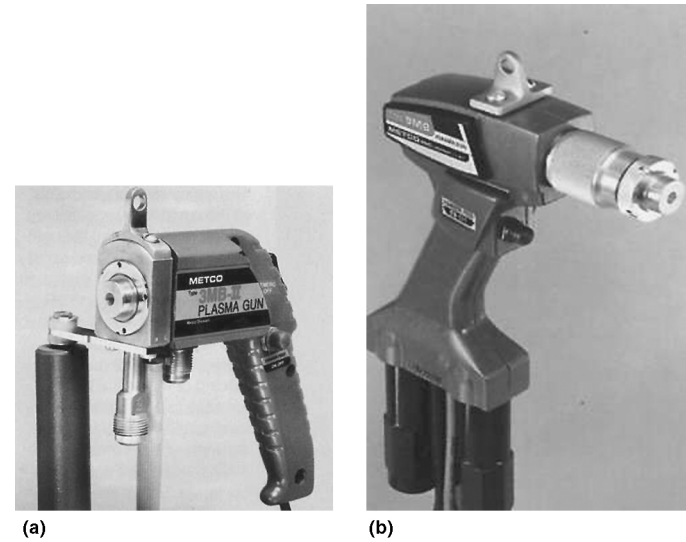


Fig. 18 Plasma spray devices developed by Sulzer Metco. (a) 3MB-II. (b) 9M



Fig. 19 Plasma spray device (model PG-100) developed by Baystate Surface Technologies

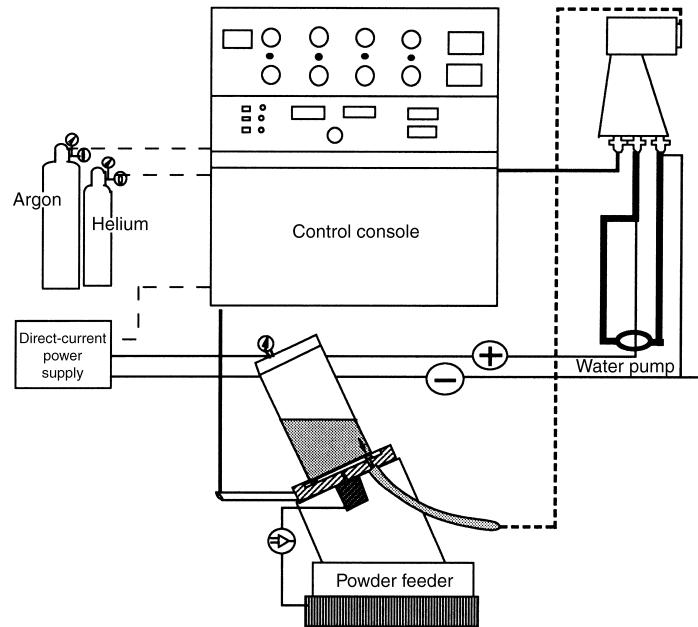


Fig. 20 Typical plasma spray system

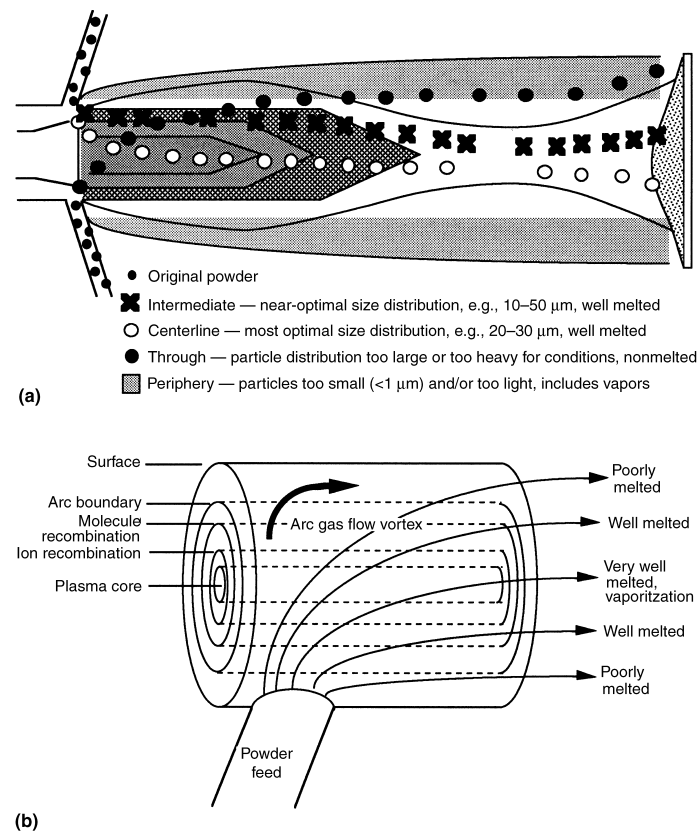


Fig. 21 (a) Plasma jet gradients and (b) particle trajectories across the jets

and gas composition. Typical plasma jets use argon together with another secondary (auxiliary) gas. Argon-only plasmas create a relatively low-energy plasma (as shown in Fig. 14), which is related to its ionization potential and thermal heat capacity. Nitrogen, on the other hand, is one of the hottest plasma gases; however, it is also a reactive gas and has some material limitations. Argon/helium mixtures are very often used, with 20 to 50 vol% He. Helium increases the thermal conductivity of the plasma stream, increasing the heating capability of the plasma. Argon/hydrogen mixtures are also commonly used, with hydrogen additions of approximately 5 to 15 vol%. Argon/hydrogen plasmas provide increased enthalpy over argon/helium due to the diatomic structure of hydrogen and its high collisional cross section related to its low mass. The trade-off with increased enthalpy is decreased anode/cathode life. The cost of consumable components can add significantly to the cost-per-unit of applied coatings. Consumable life with one common gun design using argon/hydrogen is 8 to 40 h where another design will be 100 to 160 h. Argon/helium gun designs have life expectancies of several hundred hours under some operating conditions, whereas other high-energy argon/helium anodes may last only 80 h. Common plasma gas combinations, in ascending order of enthalpy, are:

- Argon (Ar)
- Argon/helium (Ar/He)
- Argon/hydrogen (Ar/H₂)
- Nitrogen (N₂)
- Nitrogen/hydrogen (N₂/H₂)

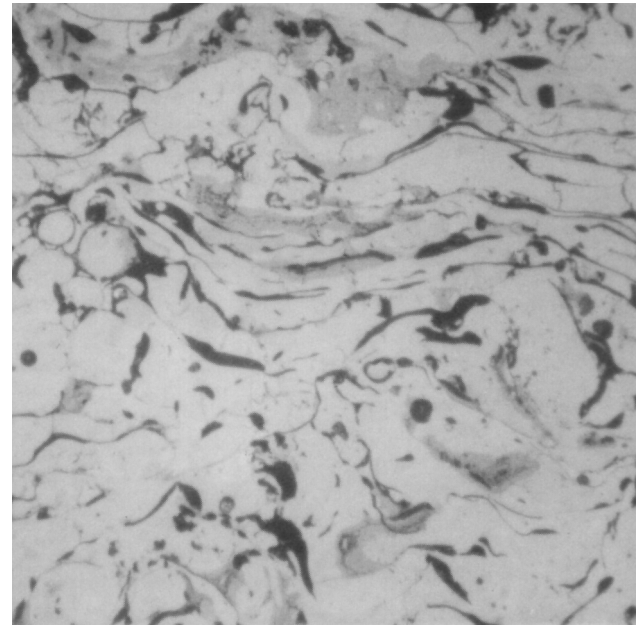
Usable anode life may be more a function of increasing copper contamination as the electrode wears. Those components with higher wear rates have yet shorter usable lives because of copper spitting.

Coating Characteristics. In plasma sprayed coatings, the hot gas jet created by a plasma arc expands, entrains powder particles, heats the particles, and accelerates them toward the substrate, where they impact, deform, and resolidify to form a coating. Refer again to Table 1, which compares the attributes of plasma spray to other thermal spray processes. The high degree of particle melting and relatively high particle velocity of plasma lead to higher deposit densities and bond strengths compared to most flame and electric arc spray coatings. The low porosity of plasma sprayed coatings can equal that of HVOF and detonation-gun-type coatings, exceeding 99%, depending, of course, on material properties, gun type, and configuration. In that porosity is a function of the characteristics of both the powder and the gun, porosity may range from 40 to <1%, as the application dictates. The high quality of a plasma spray coating microstructure results from the high particle/droplet kinetic energy, which effectively deforms the impacting particles, and the high degree of heating/melting that facilitates particle deformation and flow.

Plasma spray coating bond strengths typically exceed 34 MPa (5 ksi) and usually are more than 69 MPa (10 ksi) (ASTM C 633). The high droplet/substrate adhesion is achieved due to the high particle velocity and deformation that occur on impact. The inert gas plasma jet, ignoring ambient air mixing effects, contributes to a lower oxide content than other thermal spray processes. The

interaction of the turbulent plasma jet with the environment does, however, entrain air, which oxidizes the particle/droplet surfaces in flight, leading to oxide phases that become entrained as inter-splat oxide inclusions. Figure 22 depicts a typical plasma sprayed coating microstructure, illustrating coating oxidation, which can be detrimental to some coating functions.

Close control of standoff distance, particle heating, and particle velocity produces lower-oxide-content microstructures, as shown



(a)



(b)

Fig. 22 Air plasma spray metal coating microstructures showing oxidation levels. (a) High oxygen level in NiAl coating. (b) Typical gas level in 80Ni/20Cr coating

in Fig. 23. Some oxides will always be present in air plasma spray (APS) coatings. Environmentally controlled spraying, that is, spraying in an inert or low-pressure chamber or using an inert gas shroud, can eliminate the presence of discrete oxides in the coating microstructure. In these processes, air is excluded, eliminating coating oxide inclusions. These processes are discussed further in the following section of this article.



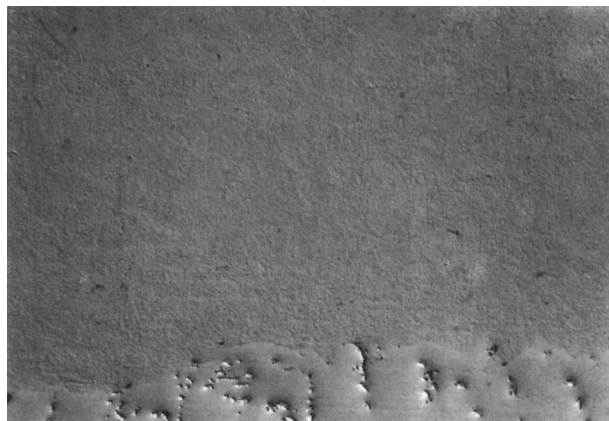
Fig. 23 Optimal air plasma sprayed metallic coating microstructure, Triballoy 400. Courtesy of Miller Thermal

Controlled-Environment Spraying

Previous sections in this article described the interaction of heated and molten particles with the environment during thermal spraying. These atmospheric interactions lead to the typical oxide inclusions (stringers) seen in air sprayed coating microstructures. Figures 12 and 22 illustrate the oxide inclusion content of the deposited materials caused by particle interactions with the air. (Similar examples can be found in the article “Coating Structures, Properties, and Materials”—see Fig. 3 and 5—in this Section of the Handbook.) The plasma process starts with inert gases for heating; thermal spray processes, in general, benefit most by controlled-atmosphere deposition methods. Beginning in the late 1960s and into the early 1970s, developments in plasma spray focused on controlled-atmosphere spraying. These developments were based on the concept that if inert gases could be substituted for air as the surrounding environment, which mixes into the plasma spray jets, then coating oxide inclusions could be reduced or eliminated.

Low-Pressure or Vacuum Plasma Spray (VPS). In 1974, E. Muehlberger (Electro Plasma, Inc., now Sulzer Metco, Inc.) published the first results on the use of low-pressure, or vacuum, plasma spray. The process demonstrated that extremely clean coatings with virtually no oxide inclusions could be produced. Figure 24 illustrates a typical VPS MCrAlY coating with <1% porosity and no oxide inclusions, with an extremely clean bond-line interface. The spray equipment consists of a conventional plasma spray gun (insulated to prevent low-pressure arc discharges) fitted with a nozzle modified for higher pressure-expansion ratios. These high ratios are achievable because the plasma jet exhausts into a chamber at below atmosphere pressure, typically in the range of 30 to 300 torr (0.6 to 5.8 psia).

Figure 25 represents a typical low-pressure or VPS system. The closed environment of the chambers necessitates remote manipulation of the spray guns and load-lock systems to transfer parts into



Unetched

30 μm



Etched

30 μm

Fig. 24 Typical microstructure of as-polished vacuum or low-pressure plasma sprayed MCrAlY coating, unetched and etched. Courtesy of Metco GmbH

the chamber. Other unique aspects of chamber spraying include vacuum pumping, exhaust cooling, filtration, and chamber-wall cooling.

Low-pressure plasma spraying results in the following process and coating characteristics relative to conventional APS:

- Broader and longer spray jets
- Cleaner interfaces, made possible by reverse transferred arc sputter cleaning
- Oxide-free coatings
- High coating densities, close to theoretical
- Low residual stresses
- Increased deposition thickness capability (>25 mm, or 1 in.)
- High substrate/deposit preheating capability: greater than 1000 °C (1832 °F) prior to, and during, coating

The primary reason for these beneficial characteristics is that the low pressure produces significant expansion of the isotherms and velocity contours of the plasma jet, due to the increased mean free path between the ions and electrons in the plasma. This leads to very long spray distances for metals. For example, spray distances for MCrAlYs via air plasma spray are 75 to 100 mm (3 to 4 in.), whereas in low-pressure plasma spray, these distances are extended to >405 mm (>16 in.). This jet expansion broadens the spray jet and produces a large, uniform spray pattern. Typical plasma plume diameters for VPS are less than 50 mm (2 in.) at a 300 mm (12 in.) standoff, compared to 10 mm (0.4 in.) at a 75 mm (3 in.) standoff for APS. The larger spray patterns result in more uniform and consistent coatings, which also tend to be more tolerant to process changes, such as variations in particle trajectories.

Low-pressure plasma spray has the added benefit of being able to operate in a transferred arc mode. Transferred arc operation is made possible by using an auxiliary power supply circuit. The transferred arc power supply is connected between the part being coated (the workpiece) and the anode/nozzle of the plasma gun. In transferred arcs, a secondary arc current couples the plasma jet to the part. Usually, the gun end connection is made to the anode.

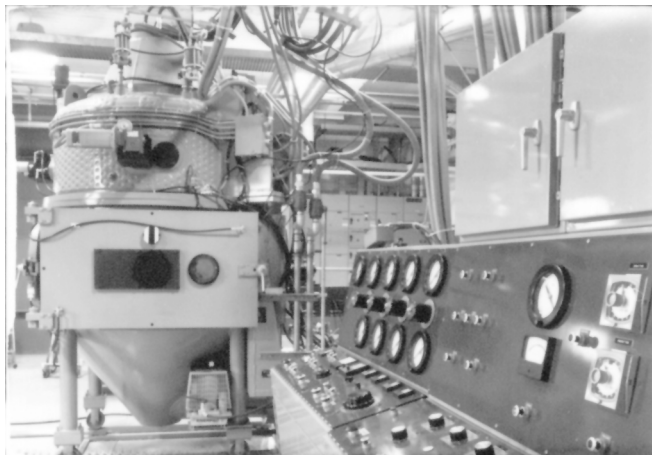


Fig. 25 Vacuum or low-pressure plasma spray system with remote plasma gun manipulation. Courtesy of ElectroPlasma Inc.

(Note: Normal APS uses a nontransferred arc, where the current flows only from the cathode, through the plasma, to the anode.) Two polarities are used in transferred arc spraying: forward and reverse bias. In forward bias, the part is electrically positive with respect to the plasma, such that current flows from the gun through the plasma to the part. In reverse bias, the part is electrically negative with respect to the plasma, such that current flows from the part, through the plasma, to the gun. Reverse bias is used to clean the substrate surface, by removing oxides and other contaminants from the surface through a process known as sputtering. Forward bias can be used to couple more of the plasma energy into the substrate, which can increase substrate heating.

The reversed-bias, transferred arc technique of VPS has been instrumental in achieving commercial success for turbine airfoil coatings. Figure 24 shows a typical bond line possible through VPS transferred arc cleaning.

Although VPS coatings are very dense, density is apparently derived from the longer dwell times and higher substrate temperatures used. Modeling, verified by testing, has shown that the mean free path (separation between the rarefied gas atoms and electrons at pressures of 10 to 100 torr, or 0.2 to 1.9 psia) in the low-pressure plasma jet approaches the size of many powder particles, approximately 25 μm (0.001 in.). This results in poor gas/particle coupling, which reduces convective heating and momentum transfer.

Oxygen contamination cannot, however, be completely eliminated in VPS coatings. With VPS, the oxygen content of the coating is more related to surface oxides on the feedstock powder particles than to the partial pressure of oxygen in the low-pressure environment of the chamber. For some applications, the oxides inherent to the powder are too high. To date, the only safe and practical method to alleviate this problem has been to increase the powder particle size, which reduces the surface-area-to-volume ratio of the particles. However, these larger particle sizes (>100 μm) are not easily melted in dc plasma jets. This need has forced the reintroduction of RF plasma spray torches. Radiofrequency torches have larger plasma zones, which produce longer particle dwell times. This is discussed in further detail later in this article.

Low-oxide-content powders have been produced and sprayed but at significantly higher cost and increased hazard. Powders produced and handled completely under an inert cover gas are pyrophoric and should be handled with extreme care. Using load locks, these powders can be moved from production to powder feeders (which must also be purged or pumped free of air) without being exposed to air.

High substrate preheating temperatures and the high-deposit-thickness capability of VPS have led to the emergence of VPS as a spray-forming tool, which is being promoted for forming free-standing shapes from superalloys, advanced composites, ceramics, and intermetallic materials. Vacuum plasma spray is inherently more expensive than other thermal spray techniques and is specified only when the deposited properties outweigh the price disadvantage. For example, VPS is cost-effective for the spray forming of specialized materials, refractory metals, and reactive materials (e.g., titanium).

Inert-Chamber Plasma Spray. Inert-chamber plasma arc spray has been used since the 1960s, combining many of the ben-

efits of VPS and APS. Inert-chamber spraying is done at atmospheric pressure but with an inert gas environment. Commonly, air is removed from the chamber with vacuum pumps, the chamber is then backfilled with an inert gas, usually argon, to atmospheric pressure. The oxygen content is typically <7 ppm when a liquid argon source is used to provide the backfill gas through a leaktight system. Simpler systems use gas purging with argon to displace the air inside the chamber. Although effective, purging takes considerably more time to remove the air and often does not achieve the same low level of oxygen as vacuum pumping.

Inert-chamber spraying offers the same thermal efficiency as APS but with the low coating oxide content of VPS. What inert-chamber spraying does not produce is the refined microstructure and clean bond lines characteristic of VPS. Inert-chamber spray lends itself well to handheld spraying through glove ports. Inert-chamber systems also cost only a fraction of the cost of a VPS system.

Shrouded Plasma Spray. As described earlier in this article, turbulent mixing of the plasma jet and the surrounding air causes oxidation of metallic powders during spraying. Significant entrainment of the air occurs, within the length of a few anode-bore diameters. A nonchamber approach to mitigating the problem of entrained air and oxidation is the use of inert gas shrouding. Shrouding provides an inert atmosphere surrounding the jet, to exclude the air, reducing the amount of oxygen available to oxidize the sprayed powder. It has also been found that argon mixed into the plasma jet cools the plasma less than entrained air, thus maintaining jet temperature over a longer jet length, effectively increasing particle dwell time at temperature.

Inert gas shrouds have been used in many forms since the 1960s. These devices usually attach to the front of a plasma gun and surround or shroud the plasma jet with a blanket of inert gas. The inert gas, usually argon or nitrogen, is used to displace air from the area surrounding the jet at the exit of the gun. The goal here is to prevent entrainment of air by the turbulent plasma jet.

There are two basic approaches to shrouding. The first type of shroud uses relatively high volumes of inert gas to flood or blanket the general area between the gun and the part being sprayed. Figure 26 shows one such device. Gas flows for one commercial shroud of this type are 280 to 510 sLm (10 to 18 scfm). Other designs require gas flows of 1.3 m³/min (45 scfm).



Fig. 26 Blanket-type plasma gun shroud

The second type of shroud attempts to control mixing at the boundary layer by supplying a concentric sheath of inert gas between the hot plasma jet and the cold ambient air. There are two basic schemes of boundary layer control. An example of the first is the Drexel shroud, developed by R.W. Smith and colleagues at Drexel University. This device attaches to the front of a plasma gun and provides a shielding gas flow concentric with the plasma jet, which replaces air with inert gas at the jet/air boundary. Gas flows for this type of device are in keeping with arc gas flows, 140 sLm (300 scfh).

An example of the second boundary layer control scheme was developed by J.V.R. Heberlein and colleagues at the University of Minnesota. This invention integrates boundary layer control into the plasma gun anode, using a porous insert to diffuse inert gas into the boundary layer between the hot plasma jet and the cold atmosphere. Gas usage for this design is minimal.

Other Significant Plasma Spray Processes

Gator-Gard. This atmospheric plasma spray process was developed in the early 1970s by Pratt and Whitney and is now assigned to Sermatech International. Gator-Gard is a modification of a standard plasma gun, operating on 100% He at flow rates of approximately 280 sLm (600 scfh). This process was designed to achieve extremely high-velocity powder particles that would deform on impact and form a dense coating, because of their high kinetic energy. Another goal was to minimize the decarburization of WC, typical in conventional atmospheric plasma sprayed WC/Co coatings. This is accomplished by lowering the plasma jet enthalpy with helium, while developing significant kinetic energy. The process has been shown to produce carbide coatings of equivalent or superior quality to D-Gun and is being used on many components for aircraft turbine engines and oil-field equipment.

Radiofrequency plasma, sometimes referred to as induction-coupled plasma spray, has been commercially available since the 1960s. Radiofrequency plasma did not see much commercial exposure until the late 1980s, when the need arose to spray larger-diameter powder particles. Figure 27 illustrates a RF plasma torch. Radiofrequency torches use an induction coil to produce an oscillating RF field (450 kHz to 4 MHz) in a reactor tube, which couples to and inductively heats the plasma arc gas stream. The RF energy initially creates a glow discharge within the coil, which quickly cascades to an elongated, toroidal (doughnut-shaped), electrodeless arc-type discharge. The arc is confined by a water-cooled quartz tube or reactor. Arc gases are fed into the reactor by several means to create a discharge that does not touch the quartz tube. The RF field and boundary layer also help to keep the arc from contacting and melting the quartz. Arc gases are usually one or more of the same four gases (argon, helium, nitrogen, hydrogen) used for other plasma arc spray devices; however, virtually any gas can be used. Water-cooled powder injector tubes are required to penetrate the plasma wall, because powder particles do not have enough momentum to penetrate the viscous plasma. Once injected, particles become entrained and heated in the plasma jet flow, which accelerates them toward the exit. Under atmospheric conditions, gas jet

and particle velocities are reported to be lower than 30 m/s (100 ft/s), although torch designs incorporating supersonic nozzles and gas flows have recently been reported. This results in increased particle dwell times in a larger, more uniform heating volume, enabling coarser powder sizes to be melted.

Continuous-fiber-reinforced metal-matrix composite prepreps that require low-oxygen-content coatings made from coarse particles are being fabricated using this technology. In this application, monofilament layers are coated with matrix materials using RF plasma spray. After coating, these prepreps are subsequently hot pressed into laminate sheets.

Radiofrequency plasmas are well suited to materials processing. The long dwell times make RF practical for consolidation, spheroidization, and chemical modification of powders. Radiofrequency plasma is also a noncontaminating process, because there are no electrodes. This feature allows the use of reactive gases, which would destroy the electrodes in conventional dc plasma spray guns. Reactive gases offer the ability to significantly alter powders during spraying by using oxygen-, nitrogen-, and carbon-rich environments. Radiofrequency plasma spraying is a niche technology, used where there is a need for advanced materials processing and composite forming.

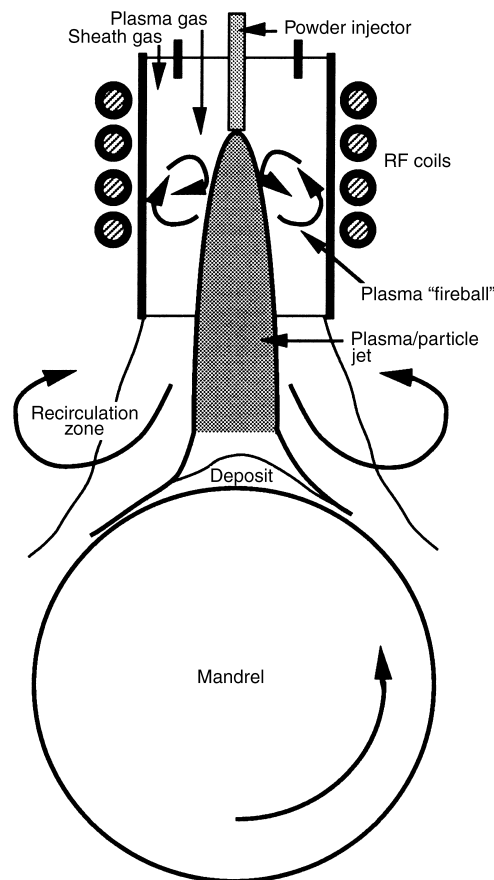


Fig. 27 Radiofrequency (RF) induction-coupled plasma spray device and process for spray forming

Emerging Developments in Thermal Spray Devices

This section covers plasma spray processes that are in development or in the early stages of commercialization. They are briefly introduced here to present their advantages and specialized applications. These methods, although currently limited in use, have the potential to become more commercially significant over the next decade.

Underwater plasma spray (UPS) was brought to the forefront through work done by E. Lugscheider and colleagues at RWTH Aachen in the late 1980s. Underwater plasma spray is a plasma spray technique that provides control of the gas atmosphere in the environment immediately surrounding the plasma jet by confining the plasma jet and its expanding gases with water. Underwater plasma spray can be used for underwater applications such as oil-field coatings and specialty materials production and synthesis. Underwater plasma spray is also useful for spraying materials that are expensive, reactive, or toxic. Collection and reclamation of overspray material is simplified by the water environment, making it useful for these applications.

Spraying underwater offers some unique technical benefits. The inert gas bubble that naturally forms around the plasma jet keeps the sprayed particles from oxidizing. Because of the hyperbaric conditions created by the hydrostatic water pressure, the enthalpy of the jet is higher, resulting in more effective heating of the particles. Another benefit of UPS is in keeping substrate temperatures cooler during spraying, which virtually eliminates surface oxidation. Thermal distortion of parts is also minimized because of the heat sink provided by the tank water, providing better dimensional control of critical parts. In UPS coatings, some coating-to-substrate fusion has been reported, which would further increase bond strength. Underwater spray distances are typically reduced to less than 30 mm (1.2 in.).

Unusual equipment needs for underwater plasma spraying include gun insulation and electrical isolation, a water tank, and changes to gun starting procedures. Apart from these unique differences, UPS process equipment is essentially the same as that for internal powder-injection APS.

Extended-arc plasma guns have been introduced for higher-rate deposition, particularly of ceramic materials. In these designs, the walls of the confining nozzle are insulated from the anode, which is positioned near the nozzle exit, as shown in Fig. 28. High gas flow rates, >300 sLm (>636 scfh), force the arc through the nozzle toward the exit where the arc root attaches to the anode. Arc lengths of >100 mm (>4 in.) have been achieved. High voltages (250 to 400 V dc) are necessary to maintain the arc at these great lengths. These higher arc voltages produce higher energy/power levels (up to 900 MJ, or 250 kW) at lower arc currents. Although more unstable than the shorter arcs used in conventional plasma spray guns, the long arc column increases jet enthalpy by producing more arc gas heating. Gas velocity is reported to approach 3 km/s (9842 ft/s). Powder injection into extended-arc gun designs is generally external to the nozzle, close to the exit. The arc jet traveling outside of the nozzle adds significantly to the melting effectiveness of these torches. Industrial acceptance and

use of these guns have been limited. One significant application has been in the area of segmented yttria-stabilized zirconia (YSZ) coatings for land-based turbines.

One variation of the extended-arc gun, the Sulzer Metco Triplex II, uses three separate arcs, operating at 180 MJ (50 kW) total power. In this design, three separate cathodes are used to strike three separate arcs, as shown in Fig. 29. (Conventional plasma guns generate an arc between a single cathode and the anode. In the more advanced conventional guns, the arc rotates, by design, in an annular position on the anode.) With the triple-arc approach, the arcs attach to separate locations on a common cascaded anode. Voltages are 100 to 120 V dc, and the total arc current is moderate at 300 to 600 A dc. Gas flows are also moderate at <100 sLm (<210 scfh) total flow. This design uses external powder injection, and the three injectors are aligned with the three-arc attachment point or arc roots. Jet velocity is lower in this design than in other extended-arc designs, which reduces noise (reportedly <110 dBA) and increases dwell time. Noise reduction is also due to the lack of longitudinal motion of the arc roots. These characteristics make the triple arc particularly well suited for spraying ceramics at higher powder feed rates and deposition efficiencies.

Water-stabilized plasmas (WSP) have been investigated since the early 1920s. It is reported that spraying was done using WSP in the early 1960s. Significant development of WSP began in the Czech Republic in the mid-1980s by P. Chraska (Czech Technical University in Prague) and was concluded in the late 1990s. Hardface Alloys, Inc. has secured the rights to produce

WSP for commercialization. In a WSP gun, the plasma is formed from a stream of water that flows from a hollow graphite cathode to an external rotating copper anode (powder is also externally injected). The water stream is dissociated by the arc, which also ionizes the oxygen and hydrogen, creating a high-enthalpy plasma jet. Power levels are typically 360 to 648 MJ (100 to 180 kW). Water-stabilized plasma operates at voltages of the order of 300 V dc and at moderate current levels. The high enthalpy, of what is essentially a hydrogen plasma, enables the spraying of metals and cermets at up to 136 kg/h (300 lb/h) and alumina-base ceramics at up to 68 kg/h (150 lb/h). Average particle velocity has been measured at 83 m/s (272 ft/s) by J.A. Brogan and colleagues at State University of New York at Stony Brook. Water-stabilized plasma is best suited for spraying large-scale oxide ceramic coatings and free-standing shapes. However, metals and cermets can also be sprayed by WSP.

Axial Powder-Feed Guns. Whether internal or external, radially fed plasma devices have inherent limitations in the efficiency of powder injection and, in the case of internal injection, with the quality of powder used. Axial powder-feed schemes for plasma spray have been investigated by many for decades, with little success. Feeding powder directly into the core of the plasma jet, where the temperature and velocity are highest, should theoretically provide more uniform heating of the powder distribution and should minimize the particle segregation common in radially fed plasma guns. Axial powder feeding is considerably more difficult with plasma than with flame or HVOF because of the high tem-

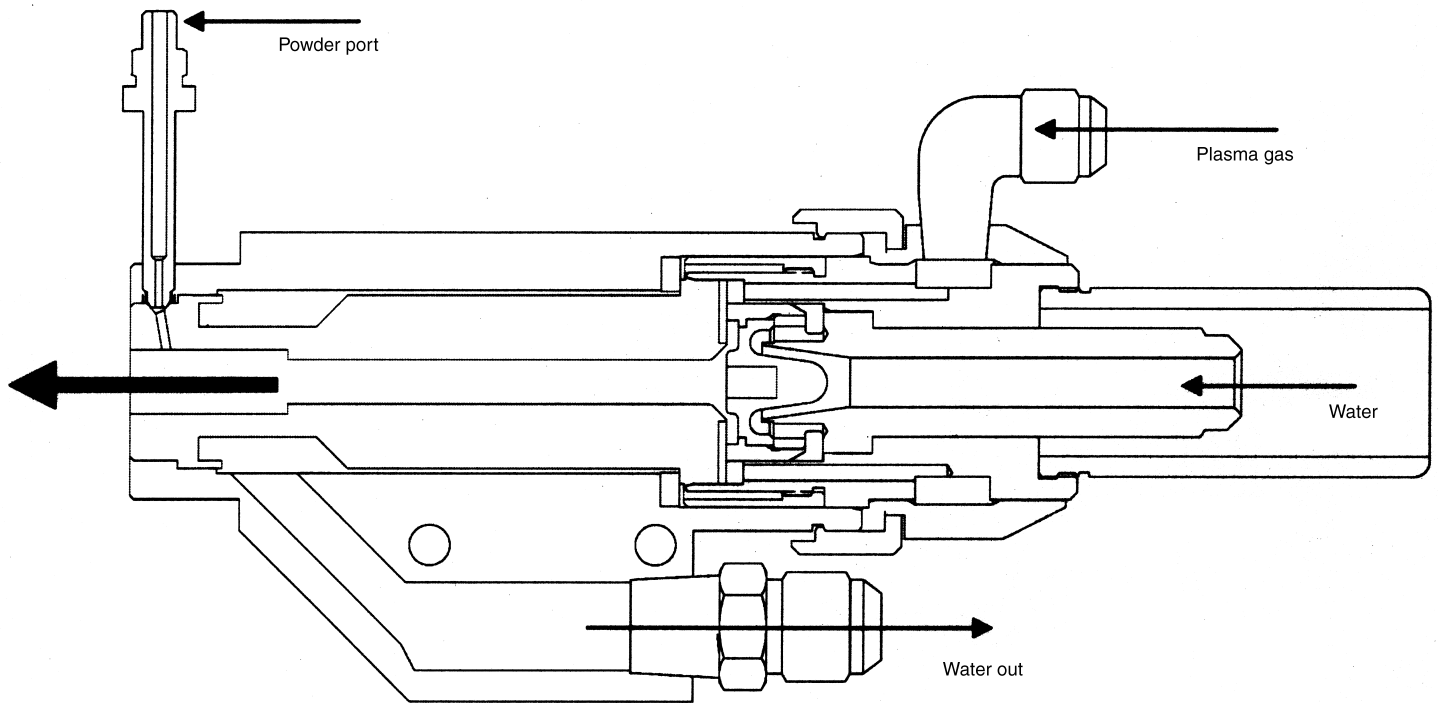


Fig. 28 Single-plasma, extended-arc gun. Courtesy of Praxair Surface Technologies

peratures of the arc. It was not until the early 1990s that commercial axial-feed plasma spray guns emerged. Although these guns have seen only limited commercialization to date, they hold promise for changing the face of plasma spraying. There have been several approaches to axial powder feeding. Commercial designs include multiple, radial cathode configurations and single cathode/anode designs with an injector located within the plasma stream. Some approaches employ extended-arc principles. Coating structures produced by axial-feed plasma guns are comparable to those from other high-end plasma spray processes.

The first of the current axial-feed plasma gun designs, developed at the University of Vancouver in the early 1990s, was commercialized by Northwest Mettech Corporation. This design uses three anodes and three cathodes. The individual plasma streams are oriented to converge within the nozzle. Powder is injected into the convergence of the three plasmas, as shown in Fig. 30 for the Axial III gun. As with the other designs, advantages include high powder-feed rates and high DEs. The increased dwell time and high power levels make this design well suited for spraying oxide ceramics. Feed rates for YSZ are reported to be up to 114 g/min (15 lb/h) at DEs approaching 80%. Copper is reportedly sprayed at 454 g/min (60 lb/h) at an 80% DE. This design uses lower currents and higher voltages to achieve power levels up to 360 MJ (100 kW).

Another commercial axial-feed design, marketed by Progressive Technologies, uses a single-anode/cathode design in an ex-

tended-arc configuration, with a coaxial plasma “splitter” through which the powder is injected, as shown in Fig. 31. The plasma splitter is a water-cooled powder injector positioned in the center of the nozzle bore, as shown in Fig. 32 for the 100HE. This aerodynamically shaped element causes the plasma stream to divide as it passes over the airfoil. Powder is injected at the trailing edge of the airfoil, where the plasma stream converges. This particular

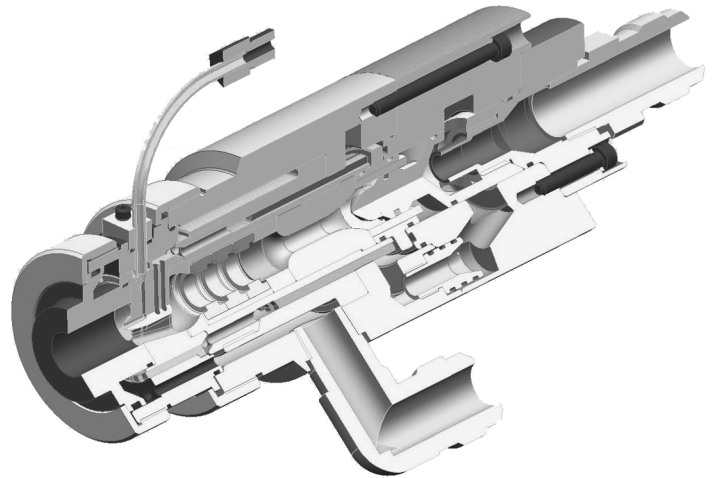


Fig. 31 Single-plasma, axial-feed plasma gun. Courtesy of Progressive Technologies

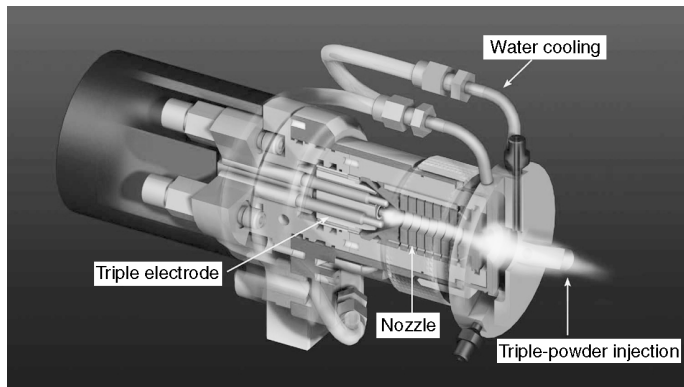


Fig. 29 Triple-plasma, extended-arc gun. Courtesy of Sulzer Metco

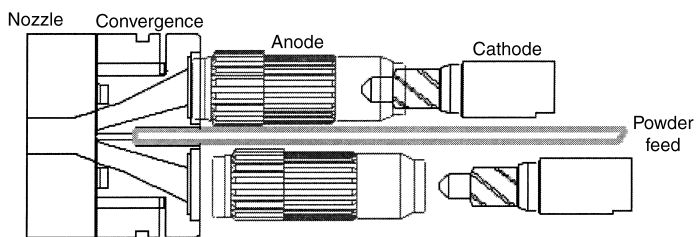


Fig. 30 Triple-plasma, axial-feed plasma gun. Courtesy of Northwest Mettech

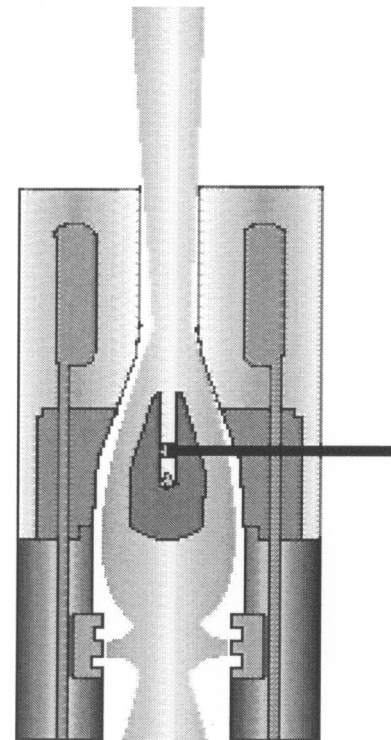


Fig. 32 Plasma splitter and powder injector. Courtesy of Progressive Technologies

device operates at power levels up to 360 MJ (100 kW), using a ternary gas mixture of argon, nitrogen, and hydrogen or helium. The combination of axial feed and the high enthalpy of the ternary gas mixture produces significantly higher spray rates and DEs. Spray rates of 100 g/min (13 lb/h) are reported for materials ranging from aluminum-polyester to YSZ, with DEs of 85 and 80%, respectively. This device is suitable for Anilox roll coatings, abradable coatings, thermal barrier coatings, and other high-rate, high-end applications.

ACKNOWLEDGMENT

This article has been revised and updated from a portion of "Equipment and Theory" by Ronald W. Smith, Lesson 2, *Thermal Spray Technology*, ASM International, 1992.

REFERENCE

1. K.S. Drellishak, Ph.D. dissertation, Northwestern University, 1963

SELECTED REFERENCES

- Advances in Thermal Spraying, Proc. 11th International Thermal Spray Conf., 8–12 Sept 1986 (Montreal), Pergamon Press, 1986
- C.C. Berndt, Ed., *Thermal Spray: International Advances in Coatings Technology*, Proc. International Thermal Spray Conf., 29 May–5 June 1992 (Orlando, FL), ASM International, 1992
- C.C. Berndt, Ed., *Thermal Spray: Practical Solutions for Engineering Problems*, Proc. Ninth National Thermal Spray Conf., 7–11 Oct 1996 (Cincinnati), ASM International, 1996
- C.C. Berndt, Ed., *Thermal Spray: A United Forum for Scientific and Technological Advances*, Proc. First United Thermal Spray Conf., 15–18 Sept 1997 (Indianapolis), ASM International, 1998
- C.C. Berndt, Ed., *Thermal Spray: Surface Engineering via Applied Research*, Proc. First International Thermal Spray Conf., 8–11 May 2000 (Quebec), ASM International, 2000
- C.C. Berndt and T.F. Bernecki, Ed., *Thermal Spray Coatings: Research, Design, and Applications*, Proc. Fifth National Thermal Spray Conf., 7–11 June 1993 (Anaheim, CA), ASM International, 1993
- C.C. Berndt, K.A. Kohr, and E. Lugscheider, Ed., *Thermal Spray 2001: New Surfaces for a New Millennium*, Proc. International Thermal Spray Conf., 28–30 May 2001 (Singapore), ASM International, 2001
- C.C. Berndt and S. Sampath, Ed., *1994 Thermal Spray Industrial Applications*, Proc. Seventh National Thermal Spray Conf., 20–24 June 1994 (Boston), ASM International, 1994
- C.C. Berndt and S. Sampath, Ed., *Advances in Thermal Spray Technology*, Proc. Eighth National Thermal Spray Conf., 11–15 Sept 1995 (Houston), ASM International, 1995
- T.F. Bernecki, Ed., *Thermal Spray Research and Applications*, Proc. Third National Thermal Spray Conf., 20–25 May 1990 (Long Beach, CA), ASM International, 1991
- T.F. Bernecki, Ed., *Thermal Spray Coatings: Properties, Processes and Applications*, Proc. Fourth National Thermal Spray Conf., 4–10 May 1991 (Pittsburgh), ASM International, 1992
- C. Coddet, Ed., *Thermal Spray: Meeting the Challenges of the 21st Century*, Proc. 15th International Thermal Spray Conf., 25–29 May 1998 (Nice, France), ASM International, 1998
- D.L. Houck, Ed., *Thermal Spray: Advances in Coatings Technology*, Proc. National Thermal Spray Conf., 14–17 Sept 1987 (Orlando, FL), ASM International, 1988
- D.L. Houck, Ed., *Thermal Spray Technology: New Ideas and Processes*, Proc. National Thermal Spray Conf., 24–27 Oct 1988 (Cincinnati), ASM International, 1989
- F.N. Longo, Ed., *Thermal Spray Coatings: New Materials, Processes and Applications*, Proc. Second National Conf. on Thermal Spray, 31 Oct–2 Nov 1984 (Long Beach, CA), American Society for Metals, 1985
- E. Lugscheider and P.A. Kammer, Ed., *United Thermal Spray Conference*, Proc. United Thermal Spray Conf., 17–19 March 1999 (Düsseldorf) DVS-Verlag, 1999
- A. Ohmori, Ed., *Thermal Spraying: Current Status and Future Trends*, Proc. 14th International Thermal Spray Conf., 22–26 May 1995 (Kobe, Japan), High Temperature Society of Japan, 1995
- *Thermal Spraying: Practice, Theory and Application*, American Welding Society, 1985
- *12th International Conference Thermal Spraying*, 4–9 June 1989 (London), The Welding Institute, 1989

Cold Spray Process

COLD SPRAYING has come to be understood, within the larger family of thermal spray processes, as a materials deposition process in which relatively small particles (ranging in size from approximately 1 to 50 μm in diameter, as shown in Fig. 1) in the solid state are accelerated to high velocities (typically 300 to 1200 m/s, or 980 to 3940 ft/s), and subsequently develop a coating or deposit on an appropriate substrate by an impactation process. Various terms—including “kinetic energy metallization,” “kinetic spraying,” “high-velocity powder deposition,” and “cold gas-dynamic spray method”—have been applied to the general form of this technique. In most instances, deformable powder particles are brought to high velocities through introduction into a nozzle, employing gas-dynamics principles of converging/diverging flows to develop high-velocity gas streams.

The mechanism by which the solid-state particles deform and bond, both to a substrate and to each other, is not well understood; however, it is thought that the high-velocity impact disrupts the oxide films on the particle and substrate surfaces, pressing their atomic structures into intimate contact with one another under momentarily high interfacial pressures and temperatures (Ref 1). This hypothesis is consistent with the fact that while various ductile materials, including metals and polymers, have been cold-spray deposited, attempts to deposit brittle or hard materials, such as ceramics, have not been successful unless they are codeposited with a ductile matrix material.

Process optimization is based on operational parameters of the particular application device (typically a gas nozzle) that permit a suitable distribution of particle velocities for producing the desired physical properties of the deposit for any given material. Typically these parameters include the gas preheat temperature, pressure, nozzle geometry, and throat size. A critical process parameter is the feedstock powder material itself—primarily particle size distribution and particle attributes such as oxide skins and mechanical properties, which influence the ability to form a compacted layer. Operational parameters are typically selected to achieve the most suitable coating for its intended application at the lowest operational temperatures and gas flows.

The distinguishing feature of the cold spray process compared with conventional thermal spray processes is its ability to produce coatings with preheated gas temperatures in the range of 0 to 700 $^{\circ}\text{C}$ (32 to 1290 $^{\circ}\text{F}$), a range that is generally lower than the melting temperature of the coating particle materials. The nozzle exit temperature is substantially lower than the gas preheat temperature, further lowering the temperature excursions experienced by the feedstock particles. Consequently, deleterious effects of high-temperature oxidation, evaporation, melting, recrystallization, residual stresses, debonding, gas release, and other concerns associated with thermal spray methods employing a liquefaction step are minimized or eliminated.

Figure 2 compares the particle velocity and gas temperature ranges of cold spray and other thermal spray processes (Ref 2).

When the particle velocity (V_p) is too low for a given coating/substrate combination, the feedstock particles are simply reflected from the surface and do not form a contiguous coating or deposit. At higher values of V_p solid particle erosion of the surface may occur, as with shot blasting of surfaces. When V_p exceeds a critical value V_{crit} (which varies with particle and substrate material),

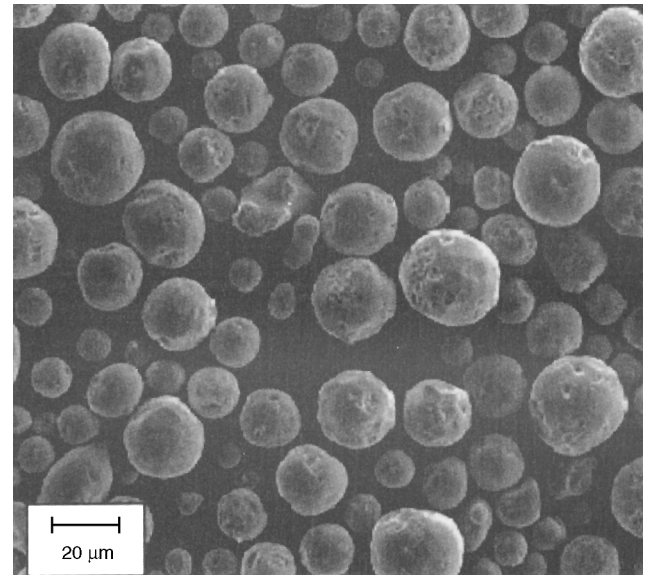


Fig. 1 Micron-sized copper powder used in cold spray experiments/applications. Source: Ref 1

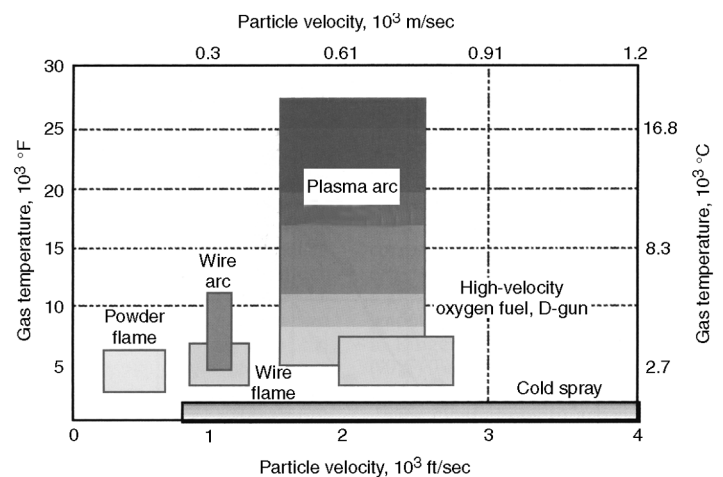


Fig. 2 Temperature/velocity regimes for common thermal spray processes compared to cold spray technology. Source: Ref 2

particles begin to plastically deform, adhere to the substrate, and form an overlay coating.

The cold spray process was originally developed in the mid-1980s at the Institute of Theoretical and Applied Mechanics of the Russian Academy of Science in Novosibirsk by A. Papyrin and colleagues (Ref 3–7). They deposited a wide range of pure metals, metal alloys, and composites onto substrate materials, and demonstrated the feasibility of cold spray for a number of applications. A U.S. patent was issued in 1994 (Ref 8), the European patent in 1995 (Ref 9). Currently, a variety of cold-spray research is being conducted at institutions in the United States, Russia, Germany, and Japan (Ref 10). These studies include gas dynamics, physics of high-speed particle impact, powder materials, novel application methods, and the development of specific applications.

Cold Spray Equipment and Process Parameters

Figure 3 is a schematic of a typical cold-gas spray system. The process gas is introduced through a gas control module to a manifold system containing a gas heater and powder-metering device. The pressurized gas is heated to a preset temperature, often using a coil of an electrically resistance-heated tube. The gas is heated *not* to heat or soften spray particles, but instead to achieve higher sonic flow velocities, which ultimately result in higher particle impact velocities. The high-pressure gas is introduced into the converging section of a de Laval-type nozzle (i.e., a converging/diverging nozzle), the gas accelerates to sonic velocity in the throat region of the nozzle, and the flow then becomes supersonic (Mach numbers ranging from 2 to 4) as it expands in the diverging section of the nozzle. The gas cools as it expands in the spray nozzle, often exiting the spray gun at below ambient temperature (hence the name “cold” spray). The powder feedstock is typically introduced into the high-pressure side of the nozzle in alignment with the throat orifice, and is delivered by a precision-metering device.

In a particularly novel adaptation of the technology, Kashirin and colleagues (Ref 11) have employed a downstream injection of the particles, thereby reducing the requirement of introduction into a high-pressure vessel, and also avoiding some of the potential issues of nozzle erosion and fouling associated with particle flows

occurring upstream of the nozzle orifice. Typical gas-jet parameters for cold spraying are summarized in Table 1.

As shown in Table 1, process gases include nitrogen, helium, air, and mixtures of these gases. Nitrogen is a favored process gas because it can be used to spray some materials without promoting oxidation (Ref 12) and because it is much less expensive than helium. The nitrogen can be supplied from liquid nitrogen tanks, but it must then be compressed to the proper working pressure. For some materials, the velocities produced with nitrogen are not sufficient to produce good deposition efficiencies or high-quality coatings. Helium is capable of providing the highest gas velocities, and therefore can be used in depositing the widest possible range of materials. Helium may also be diluted with nitrogen to improve the economics of the process, while providing particle velocities in excess of that achieved with nitrogen alone. In an effort to make helium more cost effective, gas-handling systems that permit the recycling of helium gas used in the cold spray process have been developed and demonstrated (Ref 13). Process economics for the recycling system strongly depend on production volume; that is, production volume must be sufficient to cover and provide a return on the cost of leasing or purchasing the recycling equipment.

Deposition Efficiency and Coating Properties

As with other thermal spray processes, cold-spray process parameters such as those listed in Table 1 affect both the process deposition efficiency (DE) and properties of the prepared coatings. Reference 14, for example, describes a series of spray experiments that were conducted on various materials in order to determine how process parameters influence DE and mechanical properties. Typical results from these experiments are given in Fig. 4, which presents the variation of the DE with the different spray parameters for titanium metal. It was observed that process parameters varied widely with the coating material. For example, aluminum could be sprayed at temperatures as low as 120 °C (250 °F), while MCrAlY required 650 °C (1200 °F). As indicated in Fig. 4, the gas composition, temperature, and pressure were found to have the strongest effects on the DE of the material. Typical DE values were found to vary between 20 and 80%. Process parameters were

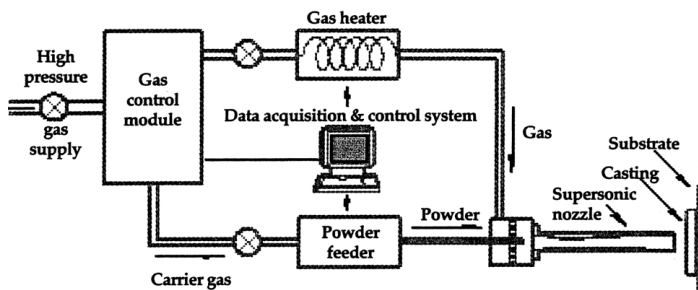


Fig. 3 Schematic diagram of a cold spray system. Source: Ref 10

Table 1 Typical range of gas-jet parameters for cold spray coating

Stagnation jet pressure, MPa (psi)	1–3 (145–435)
Stagnation jet temperature, °C (°F)	0–700 (32–1290)
Gas flow rate, m ³ /min (ft ³ /min)	1–2 (35–70)
Powder feed rate, kg/h (lb/h)	2–8 (4–18)
Spray distance, mm (in.)	10–50 (0.4–2)
Power consumption, kW (for heating gas)	5–25
Particle size, μm	1–50

Operating gases: air, nitrogen, helium, and their mixtures. Source: Adapted from Ref 10

found to affect the coating, microstructure resulting in variations in both mechanical and thermal properties. Figure 5, for example, shows the variation in hardness and elastic modulus of titanium coatings as a function of gas temperature.

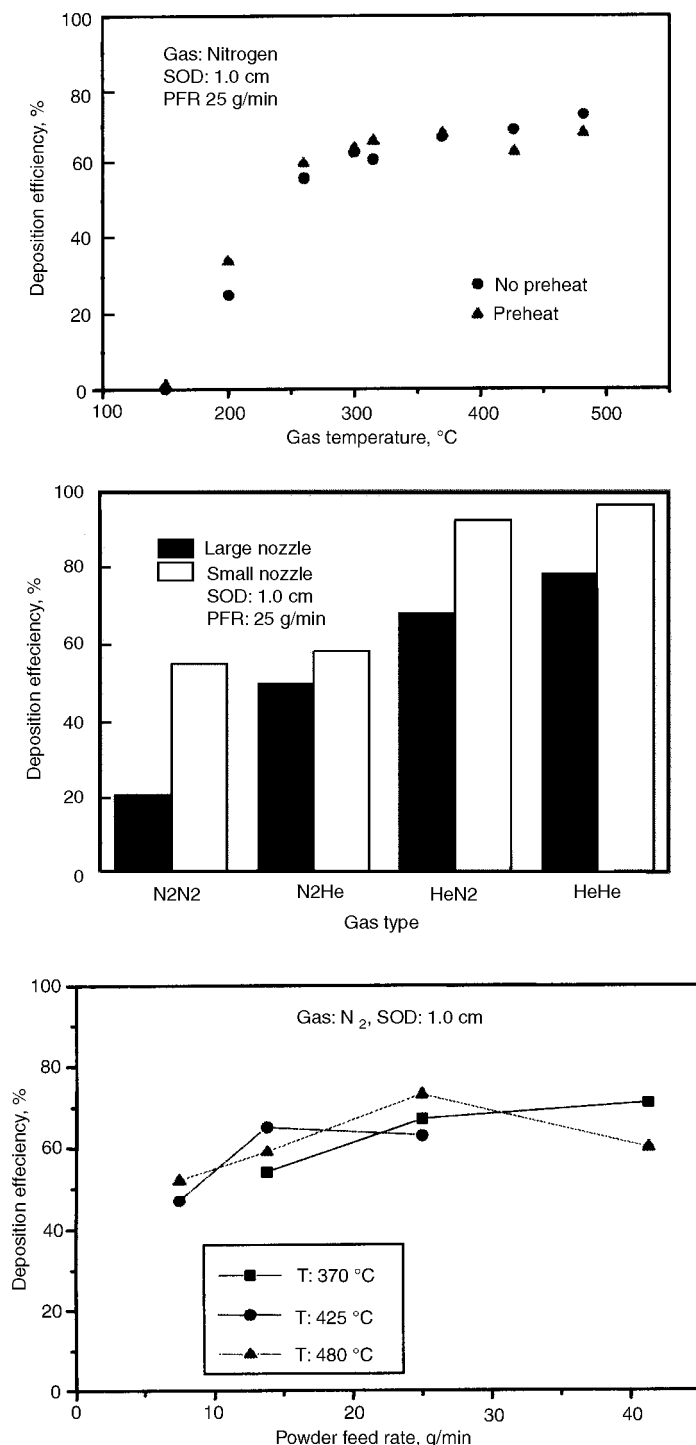


Fig. 4 Variation of cold spray deposition efficiency of titanium coatings with process parameters. SOD, standoff distance (nozzle); PFR, powder feed rate; T, temperature. Source: Ref 15

It has become increasingly clear to practitioners in the field that powder characteristics significantly affect both the ability to form deposits by cold spraying and the nature of those deposits. This was illustrated for the case of two distinctly different copper powders by McCune et al. (Ref 16).

The tensile properties of cold-sprayed coatings are typically much better than those obtained with other thermal spray processes. However, the ductility of cold-sprayed coatings in the as-sprayed condition is typically very low due to the extensive work hardening that is inherent in the deposition process. Table 2 gives the tensile properties of cold spray processed nickel and bulk nickel (Ref 17). As can be seen, cold-sprayed coatings have lower ductility and higher tensile strength in the as-sprayed condition, compared to bulk material. Heat treating the coatings at high temperature retrieves sufficient ductility.

Coating Materials and Applications

A variety of coatings have been applied by the cold spray process. These include pure metals, ferrous and nonferrous metal alloys, composites, and cermets. Table 3 lists examples of cold

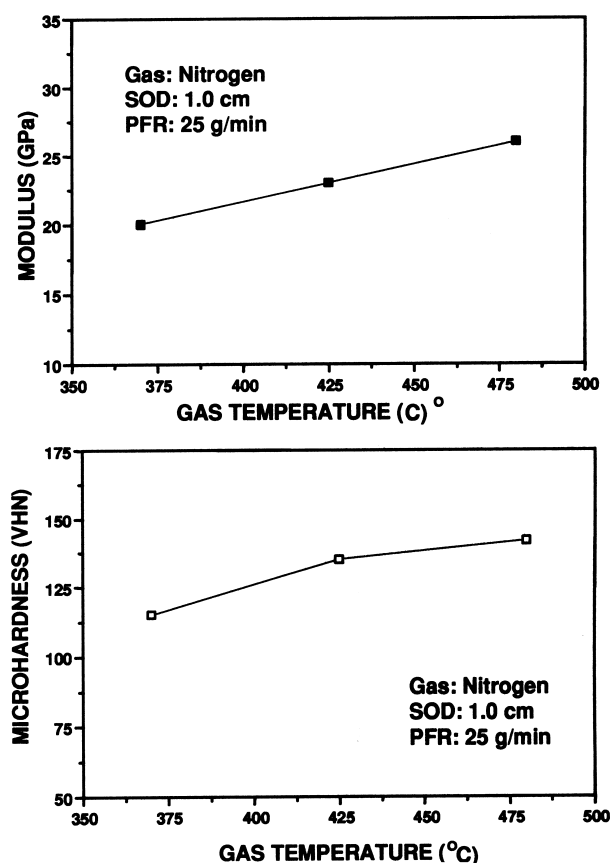


Fig. 5 Variation of mechanical properties of cold-sprayed titanium coatings with process parameters. SOD, standoff distance (nozzle); PFR, powder feed rate. Source: Ref 15

Table 2 Tensile properties of bulk nickel and electroplated nickel compared to cold-sprayed nickel in the as-sprayed and heat-treated conditions

Material	Young's modulus		Yield strength		Ultimate strength		Fracture strain
	GPa	10 ⁶ psi	MPa	ksi	MPa	ksi	
Cold-sprayed nickel, as sprayed(a)	158 ± 6	22.9 ± 0.9	300 ± 119	43.5 ± 17.3	300 ± 119	43.5 ± 17.3	0.0002
Cold-sprayed nickel, heat treated(a)	125 ± 31	18.1 ± 4.5	207 ± 57	30.0 ± 8.3	304 ± 101	44.1 ± 14.6	0.0956
Bulk nickel(b)	207	30.0	59	8.6	317	46.0	0.30
Electroplated nickel(b)	155 ± 11	22.5 ± 1.6	305 ± 12	44.2 ± 1.7	562 ± 9	81.5 ± 1.3	0.124 ± 0.005

(a) Average of data from four tests. (b) Handbook data. Source: Ref 17

Table 3 Examples of cold spray coating materials

Material	Ref.
Pure metals	
Copper	1, 2, 10, 12, 14, 16, 18, 19
Aluminum	2, 10, 14, 18
Titanium	14, 15, 18
Silver	14
Nickel	10, 14, 17
Zinc	19, 20
Molybdenum	20
Iron	19, 20
Low-alloy steels	
Ancorsteel 1000	2
Nickel-chromium alloys	
50Ni-50Cr	14
60Ni-40Cr	14
80Ni-20Cr	14
Nickel-base superalloys	
Alloy 625	14
Alloy 718	14
Hastelloy C	14
Stainless steels	
Type 316/316L	12, 14, 18
Type 420	14
Type 440	14
Zinc alloys	
Zn-20Al	19
Aluminum alloys	
1100	21
6061	14
Copper alloys	
C95800 (Ni-Al bronze)	22
MCrAlY	
	12, 14, 18
Composites/cermets	
WC-Co	14
Cr ₃ C ₂ -NiCr	2, 12
Fe-NdFeB	19
Ti-Al	12, 18
Al-Cu	18

spray coating materials. Figures 6 to 9 show typical microstructures of copper, stainless steel, titanium, and composite cold spray coatings, respectively.

Current and envisioned applications for cold spray coatings include electrical/electronic applications (copper and Fe-NdFeB),

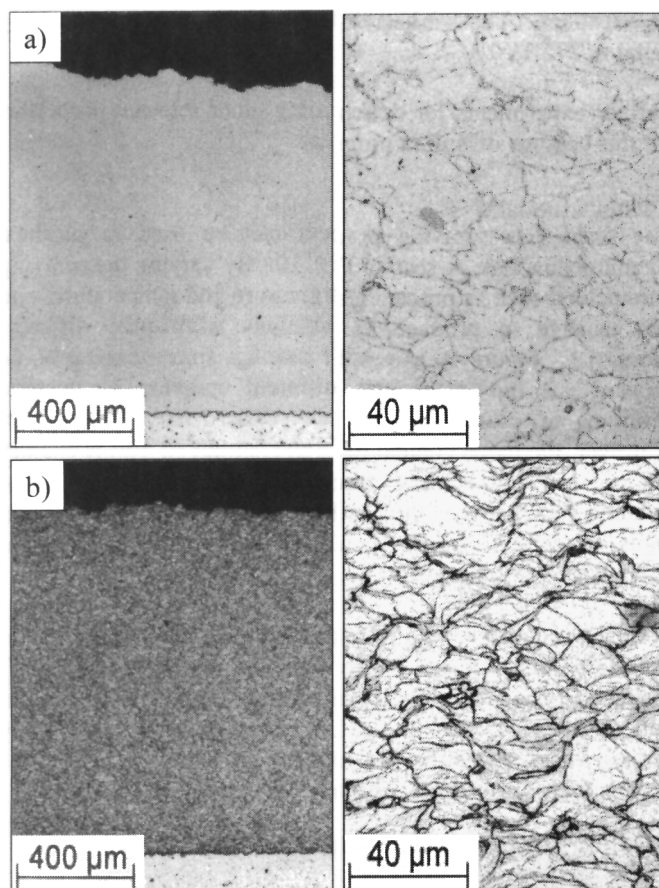


Fig. 6 Microstructure of a cold-sprayed copper coating on an aluminum substrate using nitrogen as the process gas. (a) As-polished. (b) Etched. Source: Ref 18

aerospace (MCrAlY), automotive (Al and Zn), chemical (Ti), freestanding structures for rapid prototyping, multilayered sleeves (Zn-Cu-Al), and conductive polymers. Several examples adapted from Ref 19 pertaining to the automotive industry are given below. Additional information on a broader range of cold spray applications can be found in the article “Selected Applications” in this Handbook.

Copper Metallization. Copper is perhaps the most studied feedstock material using the cold spray process. Figure 1 shows copper particles typical of those used in many studies of cold-sprayed copper coatings. Figure 6 shows a typical microstructure of a cold-sprayed copper coating on an aluminum substrate, in both the as-polished and etched states. This figure clearly shows that the coating has a very low porosity level. Furthermore, the oxygen content of the coating is equal to 0.1%, as measured with a Leco oxygen/nitrogen analyzer (Ref 18). The combination of both the low porosity level and low O_2 content contributes to the excellent electrical properties of copper cold spray coatings (Table 4). Cold-sprayed copper deposits also display excellent tensile properties. Direct-write electrical metallizations employing copper were one of the earliest envisioned applications for cold spray technology (Ref 19).

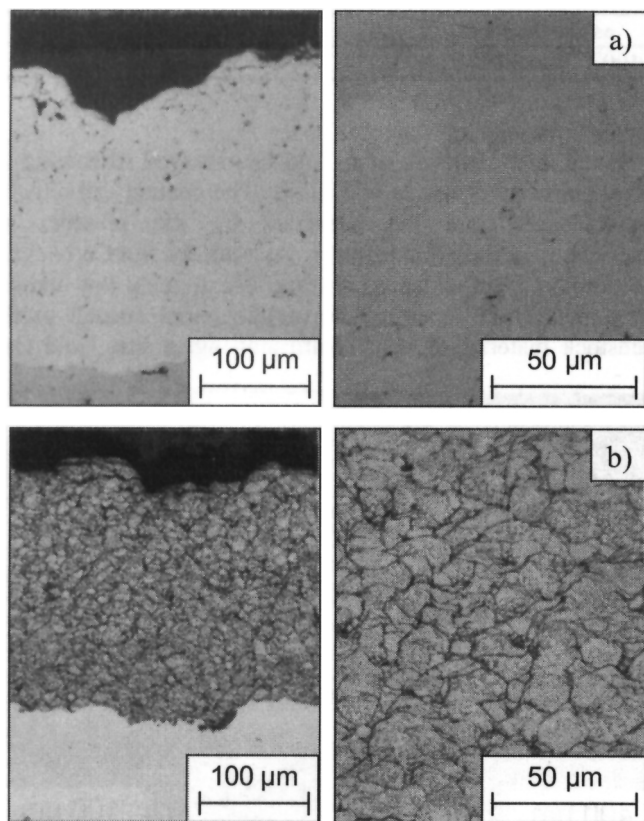


Fig. 7 Microstructure of a cold-sprayed type 316L stainless steel coating on an aluminum substrate using nitrogen as the process gas. (a) As-polished. (b) Etched. Source: Ref 18

Selective Galvanizing (Ref 19). Corrosion protection for automotive body and chassis structures relies primarily on the use of pre-coated (galvanized) steels and postmanufacturing processes

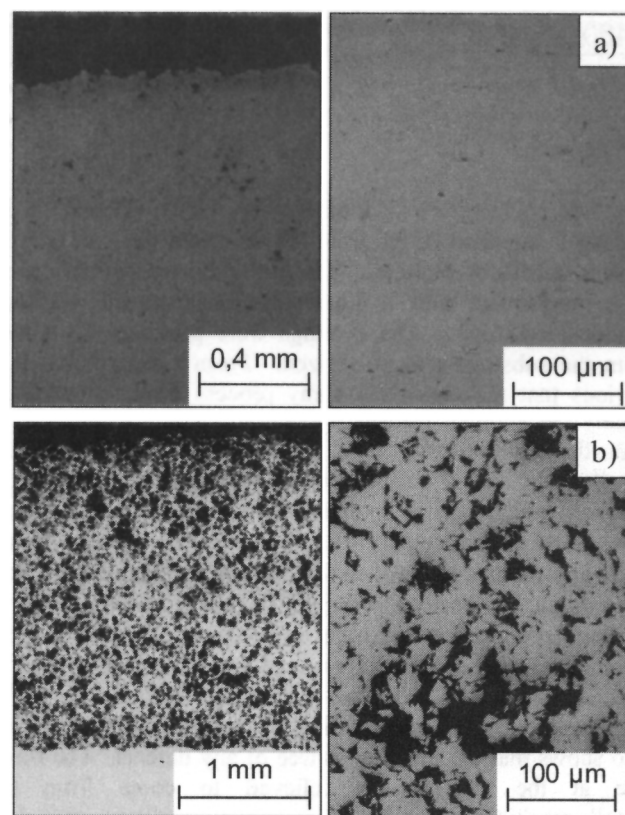


Fig. 8 Microstructure of a cold-sprayed titanium coating on an aluminum substrate using nitrogen as the process gas. (a) Dense titanium coating. (b) Porous titanium coating for biomedical (prosthesis) applications. Source: Ref 18

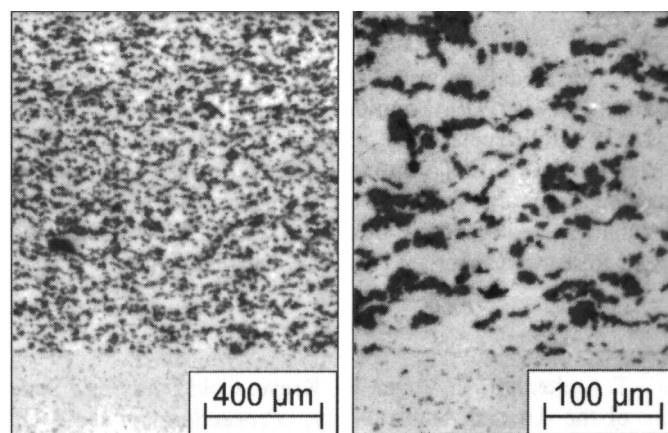


Fig. 9 Microstructure of a cold-sprayed titanium-aluminum composite coating on an aluminum substrate using nitrogen as the process gas. Source: Ref 18

such as phosphating and electrophoretic coating. However, details of construction (such as hems, weldments and other joints, extreme deformations, etc.) typically are the weakest links in total corrosion protection.

Cold spraying has been found to be a useful approach for the selective protection of localized areas that are the likely locations for corrosive attack. For example, folded or welded seams in galvanized sheet metal used for applications such as automotive door panels or stamped gas tanks often are susceptible to corrosion because the protective galvanizing layer is damaged or removed in the assembly process. Such areas can be easily protected by local application of an anticorrosion coating, such as zinc, to the affected areas.

Figure 10 shows schematically an example of a steel hem structure wherein an inner piece is secured to an outer panel by a metal fold or hem. The potential placement of supplemental zinc protection in the vicinity of the hem is illustrated. Since the duration of galvanic protection by a sacrificial metal such as zinc on steel is linearly proportional to the amount of sacrificial metal available, it then becomes possible to “program” the amount of sacrificial protection according to the severity of the specific geometry. In this manner, the galvanic protection can be strategically placed instead of relying on large expanses of precoated material to achieve protection goals in localized features. This would be similar in concept to fabrication of “tailored” panels of steel with differing strengths and thicknesses for metal forming. Potentially, both weight and cost savings could be realized through such an approach.

To date, the principal experimentation with selective galvanizing has been with steel fuel tank weld seams and hem flanges, as

illustrated in Fig. 10. The use of the pregalvanized sheet to accommodate a cold-sprayed layer of different composition can be seen in Fig. 11, where a Zn-20Al alloy powder was applied to an EG60 electrogalvanized steel panel. EG60 has a nominal zinc coating thickness of approximately 8 μm . Zinc-aluminum alloys are expected to show greater corrosion resistance in salt water than pure zinc; thus another dimension of corrosion protection is possible in local application of highly corrosion-resisting layers in specific locations.

Rapid Tooling Repair (Ref 19). An important recent development in the area of rapid tooling involves the use of massive thermal spray deposits of steel over ceramic molds to form tooling used in sheet-metal forming. Typically, the spray deposits used in this process are from twin-wire arc deposition of carbon steels and contain a relatively high fraction of oxide, as well as carbon, rendering the material difficult to repair or adjust by tungsten inert gas (TIG) or metal inert gas (MIG) welding, which is the usual industry practice. Cold spraying of high-purity iron as an intermediate layer was found to permit the use of more conventional welding processes for the repair and material buildup on dies formed by the thermal spraying process. By forming a very high purity “barrier” layer, the cold-spray iron deposit permitted attachment of weld filler material, while isolating the difficult-to-weld underlying tool material. Figure 12 illustrates the ability to form a weld bead of an iron-nickel filler wire in contact with a cold-sprayed iron substrate. Figure 13 shows an actual weld overlay on tooling material that had been previously coated with the cold-sprayed iron.

Conclusions

Cold spray deposition in its various forms is an emerging technology that addresses many classic shortcomings of more tradi-

Table 4 Comparison of electrical resistivities for copper produced by various thermal spray processes with those for bulk materials

Material	Resistivity, $\mu\Omega \cdot \text{cm}$
Bulk copper	1.68
Hard-drawn copper	1.77
Cold-spray (Acupowder 500) copper	2.39
Twin-wire arc copper	3.05–6.0
Plasma-arc copper	5.45

Source: Ref 19

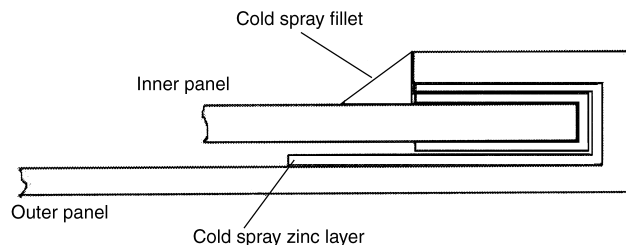


Fig. 10 Schematic of a galvanized steel hem structure with supplemental zinc applied by cold spray processing. Source: Ref 19

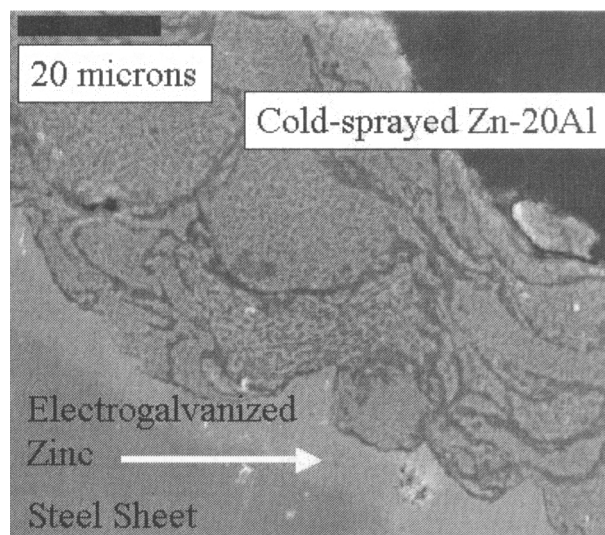


Fig. 11 Microstructure of a cold-sprayed Zn-20Al alloy powder layer on EG60 electrogalvanized steel sheet. Source: Ref 19

tional thermal spray processes employing liquefaction as the primary process. Efforts are under way as of this writing to develop a more comprehensive scientific basis for the process as well as a wider range of suitable materials, applications, and infrastructure. One of the most exciting prospects for cold spraying is its use as a selective technology for very controlled application of materials having very high purities and superior physical properties; as such it may serve the overall field of thermal spray technology as a specialized tool for novel applications and devices heretofore unachievable with existing materials and practices.

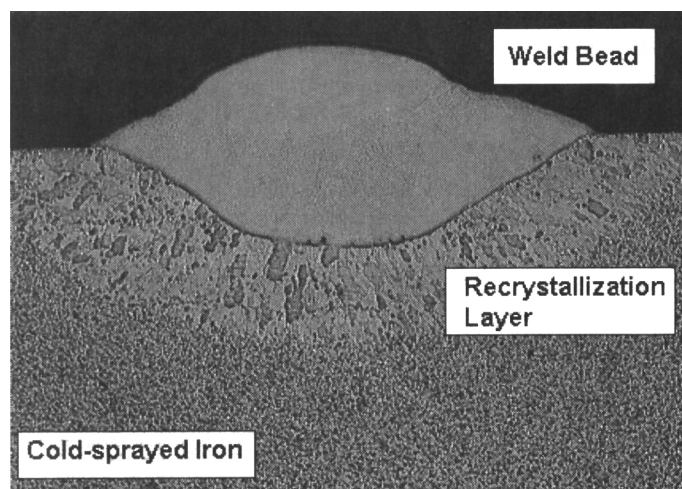


Fig. 12 Filler-metal weld bead formed on cold-sprayed iron structure, showing a recrystallized layer and grain growth below the bead. Source: Ref 19

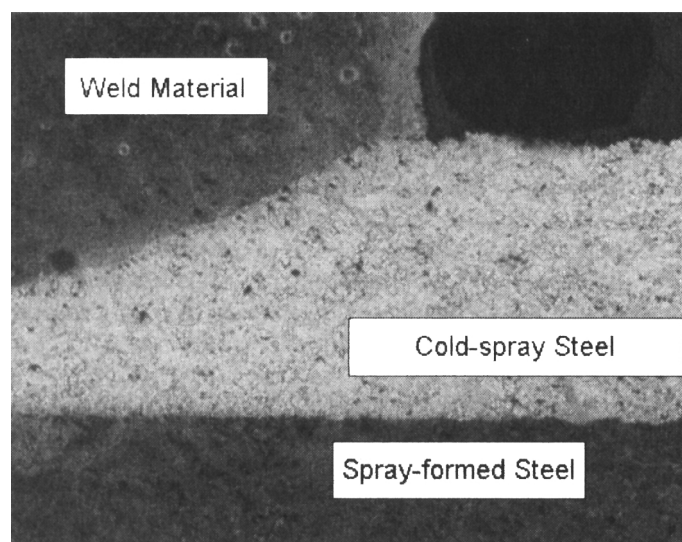


Fig. 13 Repair of thermal-sprayed steel tooling using a cold-sprayed steel intermediate layer on which a conventional weld bead was deposited. Source: Ref 19

ACKNOWLEDGMENT

Significant contributions were made to this article by J. Karthikeyan, ASB Industries Inc.; R.C. McCune, Ford Motor Company; and M.F. Smith, Sandia National Laboratories.

REFERENCES

1. R.C. Dykhuizen, M.F. Smith, D.L. Gilmore, R.A. Neiser, X. Jiang, and S. Sampath, Impact of High Velocity Cold Spray Particles, *J. Therm. Spray Technol.*, Vol 8 (No. 4), Dec 1999, p 559–564
2. R.C. McCune, A.N. Papyrin, J.N. Hall, W.L. Riggs, and P.H. Zajchowski, An Exploration of the Cold Gas-Dynamic Spray Method for Several Materials Systems, *Advances in Thermal Spray Technology* (Proc. of the Eighth National Thermal Spray Conf.), C.C. Berndt and S. Sampath, Ed., ASM International, 1995, p 1–6
3. A.P. Alkhimov, V.F. Kosarev, N.I. Nesterovich, and A.N. Papyrin, "Method of Applying Coatings," Russian Patent No. 1618778, 8 Sept 1990
4. A.P. Alkhimov, V.F. Kosarev, N.I. Nesterovich, A.N. Papyrin, and M.M. Shushpanov, "Device for Applying Coatings," Russian Patent No. 1618777, 8 Sept 1990
5. A.P. Alkhimov, V.F. Kosarev, N.I. Nesterovich, and A.N. Papyrin, "Method of Applying Metal Powder Coatings," Russian Patent No. 1773072, 1 July 1992
6. A.P. Alkhimov, V.F. Kosarev, and A.N. Papyrin, A Method of Cold Gas-Dynamic Deposition, *Sov. Phys. Dokl.*, Vol 35 (No. 12), 1990, p 1047–1049 (transl. American Institute of Physics, 1991)
7. A.N. Papyrin, A.P. Alkhimov, and V.F. Kosarev, Cold Gas-Dynamic Spray Method, *New Materials and Technologies: Theory and Practice of Hardening Materials in Extreme Processes*, Nauka, Novosibirsk, 1992, p 146–168
8. A.P. Alkhimov, A.N. Papyrin, V.F. Kosarev, N.I. Nesterovich, and M.M. Shushpanov, "Gas Dynamic Spraying Method for Applying a Coating," U.S. Patent No. 5,302,414, 12 Apr 1994, Re-examination Certificate, 25 Feb 1997
9. A.P. Alkhimov, A.N. Papyrin, V.F. Kosarev, N.I. Nesterovich, and M.M. Shushpanov, "Method and Device for Coating," European Patent No. 0484533, 25 Jan 1995
10. A. Papyrin, Cold Spray Technology, *Adv. Mater. Process.*, Sept 2001, p 49–51
11. A.I. Kashirin, O.F. Klyuev, and T.V. Buzdygar, "Apparatus for Gas-Dynamic Coating," U.S. Patent No. 6,402,050, 11 June 2002
12. T. Stoltenhoff, H. Kreye, and H.J. Richter, An Analysis of the Cold Spray Process and Its Coatings, *J. Therm. Spray Technol.*, Vol 11 (No. 4), Dec 2002, p 542–550
13. S. Jaynes and F. Lauricella, Helium Recovery: Design Considerations for Cold Spray Systems, *Thermal Spray 2003: Advancing the Science and Applying the Technology*, C. Moreau and B. Marple, Ed., ASM International, 2003, p 113–116

14. J. Karthikeyan and C.M. Kay, Cold Spray Technology: An Industrial Perspective, *Thermal Spray 2003: Advancing the Science and Applying the Technology*, C. Moreau and B. Marple, Ed., ASM International, 2003, p 117–121
15. R.S. Lima, A. Kucuk, C.C. Berndt, J. Karthikeyan, C.M. Kay, and J. Lindemann, Deposition Efficiency, Mechanical Properties and Coating Roughness in Cold-Sprayed Titanium, *J. Mater. Sci. Lett.*, Vol 21 (No. 21), Nov 2002, p 1687–1689
16. R.C. McCune, W.T. Donlon, O.O. Popoola, and E.L. Cartwright, Characterization of Copper Layers Produced by Cold Gas-Dynamic Spraying, *J. Therm. Spray Technol.*, Vol 9 (No. 1), 2000, p 73–82
17. M.K. Decker, R.A. Neiser, D. Gilmore, and H.D. Tran, Microstructure and Properties of Cold Spray Nickel, *Thermal Spray 2001: New Surfaces for a New Millennium*, C.C. Berndt, K.A. Khor, and E.F. Lugscheider, Ed., ASM International, 2001, p 433–439
18. J. Voyer, T. Stoltenhoff, and H. Kreye, Development of Cold Gas Sprayed Coatings, *Thermal Spray 2003: Advancing the Science and Applying the Technology*, C. Moreau and B. Marple, Ed., ASM International, 2003, p 71–78
19. R.C. McCune, Potential Applications of Cold-Spray Technology in Automotive Manufacturing, *Thermal Spray 2003: Advancing the Science and Applying the Technology*, C. Moreau and B. Marple, Ed., ASM International, 2003, p 63–70
20. “Cold Gas-Dynamic Spray Technology,” Product literature from Ktech Corporation, Albuquerque, 2004
21. J. Karthikeyan, T. Laha, and A. Agarwal, Microstructural and Electrochemical Characterization of Cold-Sprayed 1100 Aluminum Coating, to be presented at International Thermal Spray Conference, Osaka, Japan, May 2004
22. R.B. Bhagat, M.F. Amateau, A. Papyrin, J.C. Conway, Jr., B. Stutzman, and B. Jones, Deposition of Nickel Aluminum Bronze Powder by Cold Gas Dynamic Spray Method on 2618 for Developing Wear Resistant Coatings, *Thermal Spray: A United Forum for Scientific and Technological Advances*, Proc. 10th National Thermal Spray Conf. ASM International, 1997, p 361–367

Process Control Equipment

Revised by Daryl E. Crawmer, Thermal Spray Technologies Inc.

SUPPORT EQUIPMENT to control thermal spray processes can be categorized according to the following functions or sub-processes:

- Gun and part manipulation
- Feedstock control
- Gas pressure control
- Gas flow control
- Energy control

Equipment for these subprocess functions is critical to maintaining coating quality, as described in this article.

Process Parameters

There are several accounts of the number of variables in thermal spray processes, ranging from a few to several hundred. The possible combinations of parameters that can produce a “good” coating are staggering. Apart from manipulation, parameters that directly affect coating characteristics can be simplified into those that affect time, temperature, and mass. In order to stabilize a thermal spray process, manipulation, time, temperature, and mass must all be held constant.

Manipulation variables include:

- Standoff distance: the distance from the face of the gun to the part being coated
- Surface speed: the relative velocity between the gun and the part
- Pitch or increment: the distance that the gun moved with each subsequent stroke of the gun manipulation or revolution of the part
- Angle of impingement: the variation of the spray stream from normal or 90° to the surface of the part

The time variable is controlled in part by:

- Gun design (e.g., bore geometry and exit diameter)
- Total arc/flame gas flow, that is, primary plus secondary gas flows (fuel-to-oxygen ratio being a significant parameter in flame spray processes)
- Arc gas characteristics
- Available energy, electrical or chemical, acting upon the arc/flame gases

Temperature is controlled in part by:

- The same gas issues as for time, above
- The available energy acting upon the system

Other Important Variables. Mass is a complex variable, and perhaps the most significant one, in the sense that beyond specifi-

cations, it is the most difficult to control because feedstock is usually a purchased commodity. The mass of spray material supplied to the gun has several important characteristics. Variables common to all feedstocks include chemistry, melting point, thermo-physical properties, and coefficient of thermal expansion (CTE). Below are some of the unique variables for wire, rod, and powder feedstock.

Important wire characteristics include:

- Diameter
- Circularity
- Surface finish
- Lubrication
- Tensile strength
- Residual stress (cast and cant; see the article “Thermal Spray Processes” in this Section of the Handbook)

Important rod characteristics include:

- Diameter
- Circularity
- Straightness
- Porosity

Important powder characteristics include:

- Particle size
- Particle shape/morphology, which affects a powder’s feedability
- Particle size distribution
- Method of manufacture
- Apparent density

When these variables are controlled by job specifications, mass feed rate consistency is all that is under the practitioner’s direct control.

Flame spray processes use the energy available in the chemical bonds of fuel gases. In these combustion processes, the melting and expansion power of the flame jet is produced by breaking molecular bonds in the fuel gas and the number of bonds broken per unit time (higher fuel gas flow rates). Plasma, on the other hand, utilizes the energy of an electric arc, the power of which is directly related to the voltage drop across and the current flow through the arc. In most plasma systems, power is calculated as the product of the voltage and the current supplied by the system. Depending on losses to the water cooling, the electrical to thermal conversion efficiency of a plasma spray gun ranges from 42 to 65% of the power input to the gun. The heating power is measured by the specific gas enthalpy, which is the energy per unit mass of gas, for example, J/L or kW/scfh.

Electric arc spray exhibits the most efficient use of energy of any of the thermal spray processes. In electric arc spraying, dc

power is established by the arc gap between the advancing wires and the current in the arc. This product power ($V \cdot A = \text{kW}$) is the energy available for melting the wires, less the small amount of energy lost through radiation and heating of the atomizing air. In other thermal spray processes, it is the complexity of injecting powder into the jet that determines the effectiveness of particle heating. It can be shown that for plasma systems only about 1 to 3% of the total system energy is used for actually melting the powder particles. Powder flame spray processes are not much different. Wire and rod flame sprays are more efficient at 2.5 to 9%. Guns configured for larger-diameter rods and wires are even more efficient. Most of the flame energy is spent heating the atomizing air, input gases, combustion products, and the environment. The electric arc spray process differs in that about 70% of the available energy is usable for melting the feedstock.

Material Feed

Materials that can be thermally sprayed include polymers, metals, metal alloys, intermetallics, ceramics, and composites. The feedstock material can take the form of wire, rod, powder, or in rare cases, liquids. This leads to two basic types of feed systems: one that feeds wires or rods and another that feeds particulates.

Wire and rod feeders are simple mechanical devices consisting of electrical or air-driven motors, drive rolls, and associated speed controls. Wires and rods need to be fed continuously and uniformly into the flame or arc. The injection point of the wire/rod and feed rate are extremely important in controlling the coating quality. Uniformity of feed depends on motor drives having sufficient torque to overcome friction within the system, and in the case of wires, from the wire spools. Drive roll design is another important aspect of uniform feed. Drive rolls must grip the wire/rod sufficiently to prevent slipping while not deforming the wire or crushing the rod. Drive rolls vary according to the wire properties. Rolls are usually made from metal or fiber-reinforced phenolic. Phenolic rolls are used for ceramic rods, whereas metal rolls are used for wires. Metal rolls can be knurled for hard wires or grooved for softer wires; many variations exist. Figure 1 illustrates two concepts, V-groove and geared.

Rod flame spray is used chiefly for spraying ceramics. Rods are typically no more than 500 mm (20 in.) long and range from 4.75 to 7.94 mm ($\frac{3}{16}$ to $\frac{5}{16}$ in.) in diameter. The disadvantages of rod feeding are interruptions during rod changes and feeding rods end-to-end. Rod feed is used where materials are too difficult to form or too brittle, as in the case of ceramics, which cannot be formed into wires. Wire feed, on the other hand, is continuous and represents the preferred feed for many metallic coatings, provided the wire is available. Rod flame spray ceramic coatings exhibit very low residual stresses and can be applied in very thick sections compared to other thermal spray processes.

Wire feeding is more problematic than rod feed, being subject to more mechanical variables, because the wire load imposed on the drive system can vary greatly. Wire characteristics mentioned earlier in the section "Process Parameters" can significantly affect the uniformity of wire feeding. One source of problem is wire slip-

page in the drive rolls. Slippage can be caused by tension on the wire from drag in the dispensing units of the wire spools, coils, or drums. Slippage can also be caused by wire diameter and stiffness, which impose mechanical loads on the feeding system that interrupt continuous feeding. Friction in wire conduits between the source and the feed mechanism, or between the feed mechanism and the gun, can cause wires to slip in the drive rolls. Shorter conduits are recommended, wherever possible.

Kinks in wires and sharp bends in the feed conduits must be avoided. Wire diameters used in electric arc and flame wire spray vary from 0.5 to 5 mm (0.02 to 0.20 in.). Larger, stiffer wires, usually >2 mm (>0.08 in.) in diameter, become more difficult to feed as uncoiling requires more force and leads to larger wire tension variations. Large or stiff wires also change the electrode/guide contact forces, increasing the wear of the electrode surfaces, which affects the arc position between the wires and the particle size distributions generated at the wire tips.

Critical wire feed issues in electric arc and flame spray include wire tension, wire straightness, wire diameter consistency, wire surface characteristics, drive wheel geometry and material, and wire guide and electrode wear. For electric arc spray, consistent positioning accuracy of the wires as they form the arc gap is also an important variable.

Powder feeding is a common and important subprocess because it features in many high-end applications of thermal spray. Powder feeding also involves the most variables of any material feed system. The common feature in all powder feeders is the output, that is, a carrier gas stream in which the powder feedstock is entrained and delivered to the powder-injection port at the spray gun.

Powder feeders can be grouped into three basic categories: gravity-fed hoppers, volumetric powder feeders, and fluidized-bed powder feeders. The oldest of these is the gravity-fed hopper. These devices are simple containers, usually with funnel-shaped bottoms, that allow the powder to flow freely, assisted by gravity, from the bottom of the funnel. Feed rate in these devices is controlled by a pinch or a needle valve located in the funnel exit port. Variants of gravity-fed hoppers are still found in handheld flame powder spray devices. Although gravity-fed hoppers are not accu-

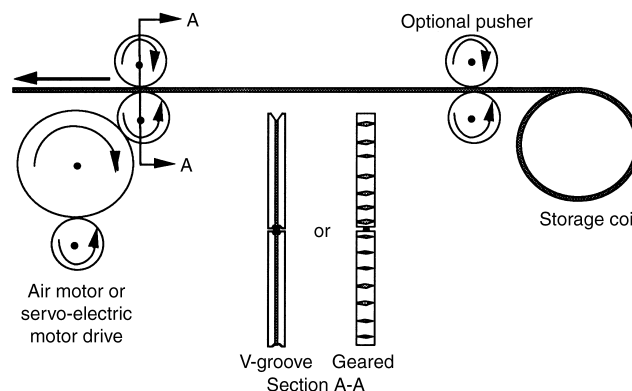


Fig. 1 Wire feeding principles and devices

rate, they are inexpensive and relatively trouble-free, provided the feedstock powder is free flowing.

Volumetric powder feeders utilize a positive-displacement mechanism to deliver packets of powder into a carburetor, where it is picked up by the carrier gas stream and delivered to the powder port in the gun. Volumetric mechanisms include screws, slotted wheels, or drilled wheels. As a screw or wheel rotates within a bed of powder, the powder is displaced by the screw or picked up by the wheel and taken to the carburetor, where it is suspended or fluidized in the carrier gas. Tampers are sometimes used to assist the packing of non-free-flowing powders into the wheel slots or holes. Vibrators are also used when powders do not flow well. Scrapers or scrapers are sometimes used to scrape excess powder from the tops of the slots/holes, so that a predetermined volume of material is delivered to the carburetor for each wheel revolution. In screw feeders, the end of the screw enters a tube, which serves the same function of controlling the volume per revolution of the screw, keeping excess powder from entering the carburetor.

In both feeder designs, powder enters the carrier gas stream by a positive pressure and, using gravity assist, moves the powder particles from the pickup area in the carburetor to the powder-feed hose leading to the gun. Feed rate is controlled by wheel or screw rotation speed. Most manufacturers offer screws with different pitches or wheels with different slot/hole sizes for feeding powders over a range of feed rates. Feed rate is also affected to a lesser extent by gravity, volume of powder in the feeder, powder characteristics, and carrier gas flow. Figure 2 schematically shows a common volumetric feeder design.

Figure 3 illustrates an aspirating-type volumetric feeder, which uses a continuous circumferential groove in a powder-feed wheel that fills with powder by gravity. Powder is packed into the groove by a counter-rotating, inverted conical stirrer. The conical stirrer helps to maintain a more consistent powder bed density above the feed slots, ensuring uniform filling of powder in the groove. Pow-

der is picked up in an aspirating venturi, through which the carrier gas flows, creating a vacuum that sucks the powder out of the wheel. The uniformity of volumetric feed for this type of feeder becomes a strong function of the packing efficiency and packing uniformity of the powder particles into the metering wheel.

Powder pulsing can occur in volumetric feeders, related to the holes, slots, and screw pitches. These devices all deliver "packets" of powder into the carburetor. The periodic interruption in powder flow is sometimes carried through to the powder-injection port at the gun. One method used to mitigate pulsing in screw feeders is a "cyclone mixer," a tangentially injected conical chamber with an axial exit. This arrangement effectively damps pulsations under conditions when it occurs. Other means of damping pulsations include changing the length and internal diameter of the powder-feed hose. Volumetric feeders are relatively insensitive to downstream pressure variations because of the positive, mechanical aspects of the particle metering.

Fluidized-bed powder feeders (Fig. 4) are also commonly used in thermal spray processes. In these units, a "bed" of powder is continuously agitated by a fluidizing gas, in this case the carrier gas. The intent by design is to suspend a controlled volume of powder in a bed using a controlled volume of gas such that a sample of powder can be drawn off at a uniform rate. To accomplish this, the bed must be in continuous motion—that is, fluidized—and the volumes of gas and powder being removed must be continuously replaced. Vibrators and/or gravity feed are used to assist and maintain flow from the hopper into the bed. Figure 4 also illustrates how the powder-laden carrier gas is aspirated through openings in the carrier-gas tube, using the venturi effect created by gas flowing through the tube.

Powder feed rate in fluidized-bed feeders is affected by powder characteristics and particle size distribution. The degree to which a particle size distribution can be suspended in the bed and carried away by the carrier gas flow is determined by the drag forces exerted by the carrier gas. Powder may fall out at a given carrier

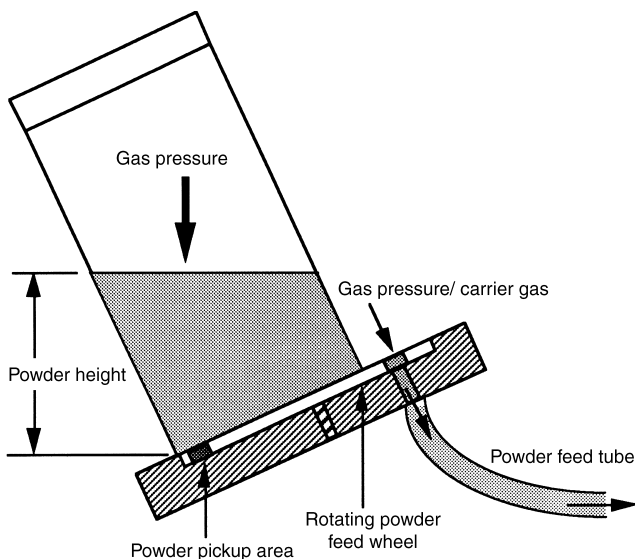


Fig. 2 Pressurized volumetric powder feeder

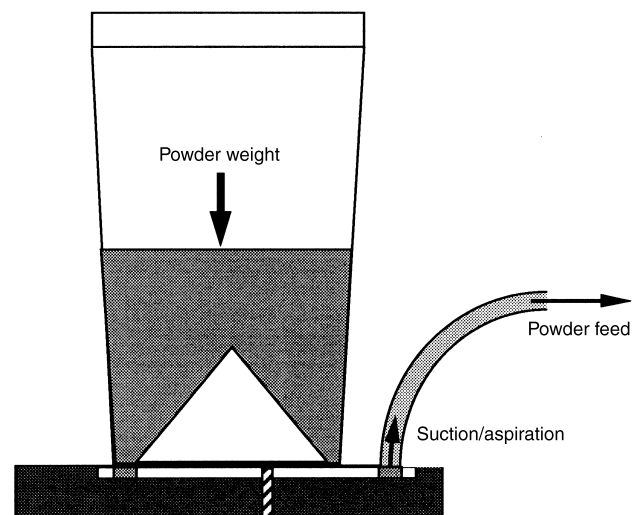


Fig. 3 Aspirating volumetric powder feeder

gas flow, if the particle size is too large or too dense. In addition, particle mass, size, and morphology determine how quickly powders can be aspirated and reach the carrier-gas flow velocity. Smaller, lighter, and/or irregularly shaped particles move faster and are carried out of the bed before larger, denser, or spherical particles. This classification of powders according to their physical characteristics is a particularly bothersome phenomenon in fluidized-bed feeders, which makes them very sensitive to powder size and morphology distributions and variations.

One advantage of fluidized-bed feeders is that they do not typically experience the pulsing sometimes exhibited by volumetric feeders. Fluidized-bed feeders do, however, experience powder mass flow variations as a function of variations in either carrier gas supply or gun back-pressures. Back-pressure variations can result from motion in the powder-feed hose, changes in plasma or flame conditions, and changes in atmospheric pressure.

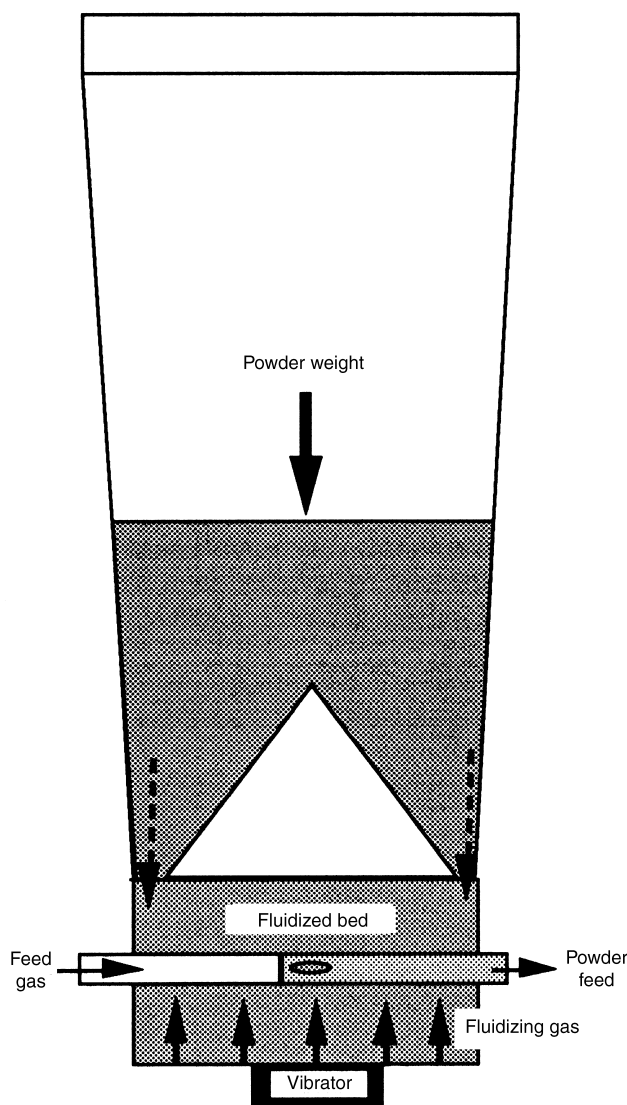


Fig. 4 Fluidized-bed powder feeder

Powder Characteristics

Several characteristics of powders make them suitable or unsuitable for uniform feeding. Ideally, powders should be free flowing, normally distributed, and free of unwanted fine particulates. Fines are often called “satellites,” because they adhere to larger particles. The ideal situation rarely exists, so it is necessary to understand the characteristics of powders that make them usable for feeding and spraying. Among the important powder characteristics are:

- Powder particle size
- Particle size distribution
- Morphology
- Method of manufacture
- Apparent density
- Moisture content

Particle size, commonly defined by the upper and lower limits of the powder size distribution, is often used for specifying and sourcing powder. It should be understood that these limits are normally established by the 90th and 10th percentiles of the distribution, respectively. For example, a typical plasma spray powder might be defined as -325 mesh, $+10\ \mu\text{m}$ (10 to $44\ \mu\text{m}$). This powder by normal definition would have a minimum of 80% of its particles between 10 and $44\ \mu\text{m}$, allowing for up to 10% below $10\ \mu\text{m}$ and 10% above $44\ \mu\text{m}$. The assumption is that the powder is normally distributed (Gaussian). Oftentimes this is not the case. When a powder sizing is skewed toward the fine side of the distribution, it will not feed well.

Size distribution further defines powder sizing. The average or mean particle size and the skew of the distribution are also critical parameters. Generally, good powders are monomodal (normally distributed, having only one peak in the curve) and are not skewed (the difference between the 50th percentile and the average or mean particle size) beyond reasonable limits. Some skew is unavoidable, particularly in particle size distributions where the upper limit is a screen size and the lower limit is not. For economic reasons, manufacturers use screens to classify powders whenever possible, and screens produce a sharper cutoff than air classification. Air classification is often used for distribution limits that fall below $44\ \mu\text{m}$ (325 mesh), because screens below $44\ \mu\text{m}$ are impractical for production processes.

Differences in the skew of the powder from one lot to another will affect coating properties. All spray parameters considered, if only the particle size changes, then the powder trajectory through the jet will also change, which in turn will change the time/temperature profile of the powder stream. Again, as the skew shifts toward the fine side of the size distribution, powder-feeding irregularities will occur.

Powder morphology, or shape, should be inextricably linked to the method of manufacture; it is not. Manufacturing process variations can effect changes in particle morphology. Morphology affects feedability, apparent density, sprayability, unmelted particles in the coating, porosity, vaporization, and so forth. Water-atomized powders are one example of this phenomenon, where the morphology may range from almost perfect spheres to highly

irregular, convoluted shapes, depending on the manufacturing process parameters. Irregularly shaped particles do not feed as easily as spherical or equiaxed (particles with the same dimension in every direction) powders, because they pack together more easily. Because a sphere has the lowest surface-area-to-volume ratio of any geometry, any nonspherical shape can adsorb more surface moisture than a sphere. Gas-atomized powders are usually more spherical and therefore typically flow very well. Figure 5 is a micrograph of water-atomized powder particles with high surface areas, typically leading to poor feeding. A spherical gas-atomized powder is shown in Fig. 6.

Manufacturing Method. A variety of manufacturing methods can be used to make a given type of powder. Each method produces unique powder characteristics in terms of morphology, feedability, apparent density, grain size, and so on. Each manufacturing method, for a given particle size and composition, will

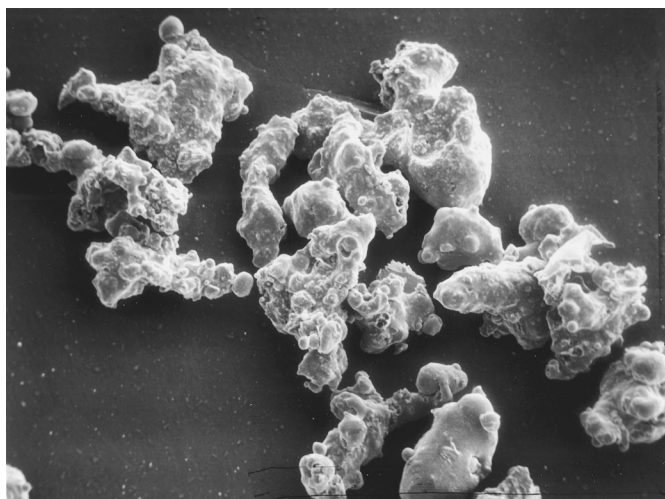


Fig. 5 Micrograph of irregularly shaped water-atomized powder

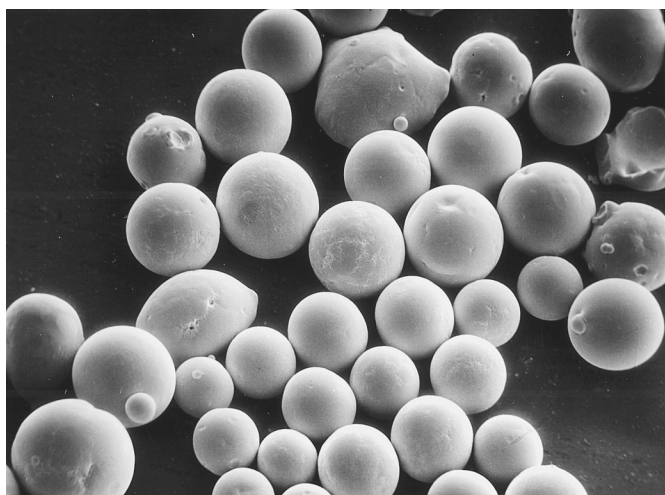


Fig. 6 Micrograph of spherical gas-atomized powder

also produce different coating characteristics. These differences may or may not be significant. Manufacturing methods are covered in the Section "Materials Production for Thermal Spray Processes" in this Handbook.

Powder manufacture plays a key role in high-velocity oxyfuel (HVOF) spraying. Cast and crushed WC/Co powders do not perform well in HVOF processes. This type of powder can shatter on impact due to the high kinetic energy of the jet and the brittle nature of the powder. However, WC/Co powders that have some inherent porosity, such as sintered and crushed or spray-dried, plasma-densified powders, work very well in HVOF guns. It is believed that these powders do not shatter on impact, because the energy is spent in crushing as the porous powder collapses on impact.

Thermal spray powders can also be engineered as mixtures of phases in the form of blended, agglomerated, or sintered particles. Agglomerated powders tend to be the most fragile, often fracturing under the stress of shipping, handling, feeding, and delivery to the jet. Fracturing of these particles increases the volume percent of fine powders, which reduces flowability and can cause spitting problems in the plasma gun and increased fume generation. It can also affect coating quality.

Density is a rather loosely used term in the thermal spray industry. Density is variously used to define the absence of porosity, the specific gravity of a material, and the apparent density of powder. Apparent density is the "specific gravity" of a powder, which includes all the spaces between particles and the pores within the powder particles themselves. Apparent density is related to powder packing density. As an example, wrought or forged steel might have a density of 7.8 g/cm^3 , whereas powdered steel may only be 3.1 to 4.7 g/cm^3 because of poor particle packing. Apparent density affects two important factors in spraying: feed rate and trajectory.

The apparent density of a bulk powder is controlled largely by the particle size and size distribution. In this sense, as apparent density changes, so does feed rate, particularly with volumetric feeders. Repeatability in a volumetric feeder, at a given feed rate, is determined by the constancy of the apparent density of the powder. Apparent density in the individual powder particle, related to porosity, will affect the trajectory of the powder particle through the jet.

Moisture Content. Adsorbed (surface) moisture on powders is one of the more significant and often overlooked aspects of powder feeding, but one of the simplest to control. Powdered materials have very high specific surface areas and can retain or adsorb an appreciable amount of surface moisture. Some materials are hygroscopic (water absorbent) and can hold even more water. To alleviate this problem and eliminate a significant source of inconsistent powder feed, it is strongly recommended that powder-drying procedures be implemented. Powders should be placed in a heated oven for some time prior to spraying.

General practice is to place powder containers in the oven with the lids loosened and any security plugs or covers removed. Time and temperature vary considerably within the industry, but all powders should be dried for at least 8 h prior to being sprayed, preferably at slightly above the boiling point of water, 100°C (212°F).

°F). However, temperatures must vary with powder material, container material, and particle size. Polymer powders are usually heated to 50 °C (125 °F), as are powders in plastic containers. Finer powders should be heated for longer periods because of their high surface-area-to-volume ratio. This ratio increases exponentially as the particle size decreases, such that finer powders can hold significantly more moisture than coarser powder. A vacuum drying oven is much more effective than heat alone.

Moisture poses such a significant problem that many powder feeders have heated canisters to keep powders from picking up moisture during spraying and between shift work. Powder moisture is a major cause of powder feed problems, but is easily rectified through proper control.

Powder Flowability Measurements. Apparent density and flowability can be measured quickly and inexpensively. Such tests are illustrated in Fig. 7, which includes a modified flow test that measures powder flow through a metering orifice as a function of time. Powder that fails to flow through the orifice signifies poor flowability. The angle of repose or slope of the mound of powder formed by powder dropping from a specified height through a flow orifice can also be used to assess relative flowability. Poor flowability would be indicated by steep slopes of the powder pile, while free-flowing powders would have little slope. High angles of repose alone would not exclude a powder from use, although caution in feeding and handling would be suggested.

Powder Feed Rate Control. Even with well-specified and well-characterized powders, minor variations occur in carrier gas pressure/flow rate and in powder characteristics. These variations affect feed rate. Closed-loop feed-rate controllers are used to provide consistent mass flow into the flame or plasma jet. Most of these controllers operate on a loss-in-weight principle. Feeders are placed on sensitive electronic scales that measure the weight loss as powder is dispensed from the feeder. The rate of weight change is compared electronically to a preset feed rate. Variations from the setpoint cause the motor speed or gas pressure to change to compensate for the variation, holding the mass feed rate constant. Accuracy for these units is typically better than 2%. The limitation of these closed-loop feed-rate controllers is that, although they deliver consistent powder feed rates, if the powder is out of specification, they will deliver bad powder consistently.

Powder Pulsing

Pulsations in powder feeding are a common occurrence. Pulsations cause nonuniformity in coating thickness and microstructure. The following paragraphs discuss some of the known causes of powder-feed pulsations.

Leaks. The leading cause of powder pulsing is gas leaks in the carrier-gas circuit. Leaks are common in powder hose fittings at

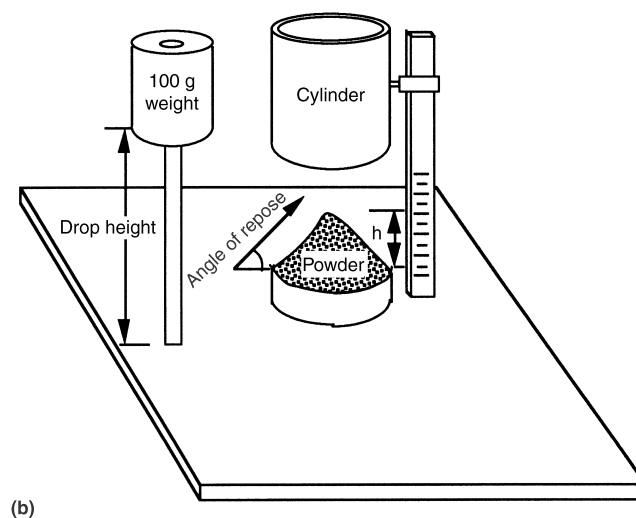
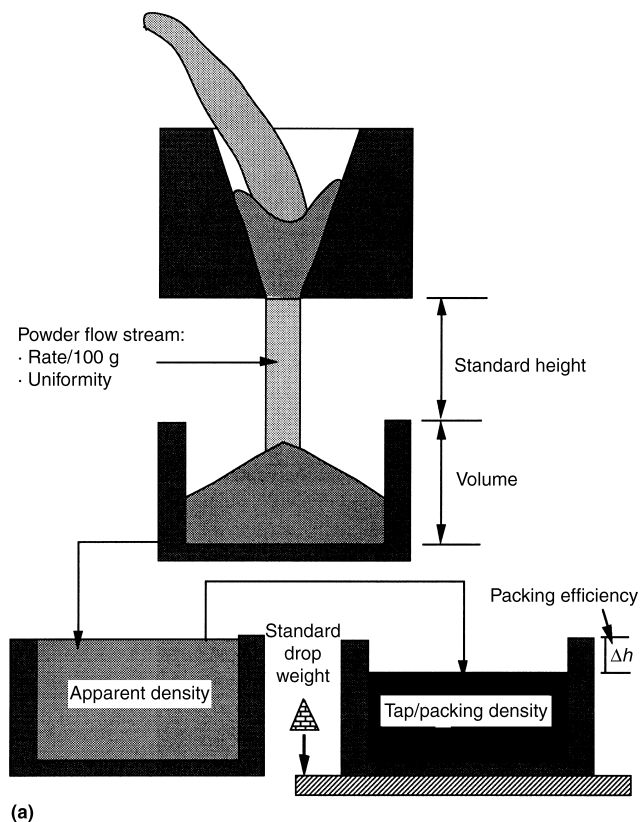


Fig. 7 Powder flowability measurements. (a) Hall flowability test. (b) Powder and angle of repose

both the gun and feeder ends. Loose hose fittings and damaged seals are usually the causes of gas leaks. Leaks are also common at the lid seals on powder canisters, mostly due to powder residue from loading the canister.

Powder Particle Size Distribution. Wide particle size distributions, distributions containing excessive fines, and bimodal distributions all affect flowability.

Moisture in powder affects flowability.

Static electricity is a problem primarily with ceramics and polymers. Static charges build up in powder-feed hoses as the insulating powders rub against the hose walls. These charges cause powder to stick to the inside surface of powder-feed tubes. Periodically, the powder buildup breaks free, causing pulsing. Static charges can build up to potentials sufficiently high to damage the electronic circuits in the feeder and control console. Using conductive hoses will work for flame spray, but is not recommended for plasma, because the powder ports of plasma spray guns are at the anode potential. Using a conductive powder hose can also direct the high-frequency starting pulse back into the powder feeder and control console, resulting in damage. Antistatic hoses with embedded grounding circuits are commercially available.

Hose dressing is another source of powder-pulsing problems. Powder feeding is a dynamic process, with the motion of the powder through the hose being acted upon by the motion of the hose and gun during spraying. It has been observed that unnecessarily long hoses aggravate the problem with excessive motion. Using the shortest possible hose length is one remedy for this problem. Another solution is tying up the hose so as to restrict its independent movement with respect to the gun.

Hose inside diameter (ID) can be a factor contributing to poor feeding. Hose IDs range from 4.8 to 6.35 mm (0.1875 to 0.25 in.). In some instances, these larger IDs create reduced powder velocities, causing the powder to fall out of suspension in the carrier gas stream. Reducing the hose ID to 3.2 mm (0.125 in.) will keep the powder moving through the hose without affecting powder injection.

Gas Flow Measurement and Control

All thermal spray devices use some type of gas. This section discusses the practical application of gas flow measurement techniques as they relate to thermal spray processes. The information given herein avoids esoteric, purist issues that relate to accuracies and effects below 2%. Gas flow is a much-disputed topic in many fields; thermal spray is no different. Issues with which industry wrestles include the type of measurement instrument, its calibration, accuracy, and so on. The three basic types of devices used in the thermal spray controls are rotameters, critical orifices, and thermal mass flowmeters. Each of these devices has strengths and weaknesses.

Rotameters, often called variable-area flowmeters or flow tubes, consist of a vertical glass or polymeric tube having a tapered or conical gas passage (hence, variable area) over the length of the tube. As shown in Fig. 8, a “float” located in the gas passage is used to indicate the gas flow.

Floats are either spherical or machined and are made from a variety of materials having different densities (specific gravities.) In order of increasing density, float materials are typically nylon, glass, sapphire, stainless steel, WC/Co, or tantalum. The choice of float material is made primarily based on the required gas flow. For a given tube diameter and length, increasing the float density increases the flow for a given position or reading on a scale. Readings are taken at the widest point of the float. The widest point of a spherical float is the equator; for a machined float it is usually a knife-edge near the top.

Scales located on, or next to, the rotameter tube are used to read the float position. Scale units are given in one of three ways:

- 0 to 100 for rotameters that read as a percentage of full scale
- 0 to X, where X equals the length of the tube in centimeters or millimeters, for example, 0 to 150 for a 150 mm tube
- 0 to X, where X equals the calibrated flow, for example, 0 to 1500 scfh or 0 to 750 sLm

Because rotameters are inherently inaccurate, measurements taken with them require some interpretation. In decreasing order of significance, pressure, temperature, and parallax (see below) have the greatest effect on accuracy. Other factors such as wear of the float and tube are longer-term problems. Rotameters are calibrated at specific gas pressures and temperatures. Generally, they are referenced to room temperature (20 °C, or 70 °F) and atmospheric pressure (100 kPa, 1 bar, or 14.5 psi absolute.) Any variation from the calibration conditions results in an error.

Gas pressure, in practice, is the pressure drop (ΔP) across the float. As ΔP increases, the susceptibility of the measurement to variations in the downstream or process back-pressure also increases. If, for example, the meter is calibrated at atmospheric pressure but used where the back-pressure is 10 psig (gauge pressure = 24.7 psia), the actual gas flow for a given float level will be considerably higher. It follows that when the meter is calibrated at a fixed set of upstream and downstream pressures, any variation in these will affect the actual flow. In practice, both pressures vary as

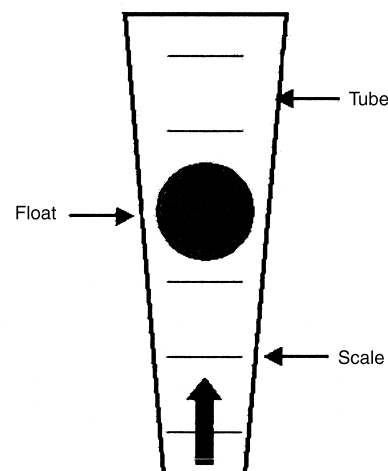


Fig. 8 Rotameter gas flow measurement gage

a function of flow. As the flow is increased, the supply pressure usually drops and must be adjusted. In any configuration other than a vent to atmosphere, back-pressure increases with flow. From a practical and economic perspective, these pressures cannot be simultaneously controlled.

Gas density, for a specific gas, is defined by both temperature and pressure. Gas temperature is nearly equal to pressure with regard to its effect on rotameter accuracy. Density and temperature are inversely proportional; that is, as temperature increases, density decreases. Since the buoyancy of a float varies with the density of the gas being measured, it follows that accuracy also varies; the position of the float will vary as a function of temperature, all else being equal. The general environment and gas withdrawal rates have the most significant effect on gas temperature. Where gas is stored outside, seasonal temperature variations can be greater than 90 °C (160 °F). Daily variations can be as high as 40 °C (70 °F). Extreme temperature variations are experienced even with gases stored inside, because gas withdrawal rate also affects gas temperature.

As gas is withdrawn from a supply, the bulk gas or liquid source cools because of adiabatic expansion. (When gas expands away from its source, it removes heat from the source.) To illustrate this problem, as the working day begins, the gas supply will be at room temperature. As gas is drawn, the temperature falls to some equilibrium point where the withdrawal rate and ambient temperature nullify each other. During this time, the rotameter float has been kept at the same level and supply pressures have been held constant. The density of the gas throughout the run has continued to increase until equilibrium has been achieved, where the density stabilizes with temperature. In this case, flow has been accurate at perhaps only one instant during the run. As a rule of thumb, gas density varies by approximately 1% per 5.5 °C (10 °F) temperature change.

Parallax is the apparent displacement of the rotameter float that occurs when the float is viewed from different vertical angles or heights. The position of the rotameter with respect to the eye of the person reading it results in errors from one operator to another. The problem is worse when the scale is located on the tube surface because the operator has to “sight” past the scale to read the float level. Scales mounted beside the rotameter tube help to minimize parallax errors.

All factors combined, under calibrated conditions, rotameter accuracy and resolution (the ability to read scale divisions accurately) vary as an inverse function of length. Depending on the rotameter manufacturer and materials, accuracies range from $\pm 2\%$ at 250 mm to $\pm 20\%$ for 50 mm long tubes. It has been observed in the field that errors of 2 to 30% exist in “calibrated” systems. Scale types and location also affect accuracy. While rotameters are inexpensive and compact, they are sensitive to pressure and temperature variations and parallax errors.

A critical orifice, sometimes called a sonic nozzle, is a disk-shaped element with an axial hole. The orifice is usually inserted into a fitting, tube, or pipe through which flows the gas to be controlled. As the upstream or supply pressure (P_1) increases, the velocity through the orifice reaches sonic velocity. Above sonic velocity, the flow through the hole becomes a linear function of

increasing supply pressure. As a rule of thumb, these conditions exist at P_1 -to- P_2 (upstream/downstream pressure) ratios higher than 2 to 1 (see Fig. 9). Where the downstream process pressures are higher than 1 atm, the upstream pressure has to be correspondingly higher. For example, in HVOF processes gun back-pressures may be 690 kPa (100 psi); in this case, to achieve critical flow, the minimum P_1 pressure will need to be 1380 kPa (200 psi); the supply pressure to P_1 must be higher yet to have a control range.

Critical orifices have two major advantages over rotameters: they exhibit essentially no parallax problems, and, provided the 2-to-1 pressure ratio is maintained, downstream pressure has no effect on flow. The control point for an orifice is either an analog pressure gage or an electronic transducer. Even with an analog pressure gage, parallax error is minimal. The basic accuracy of an orifice is determined by the precision of the hole and the accuracy of the P_1 pressure gage, usually $\pm 2\%$. Another advantage is lack of wear, particularly in jeweled orifices.

Critical orifices are smaller and less expensive than rotameters. They are not subject to excessive wear and are relatively insensitive to process pressure variations. However, orifices have the same sensitivity to temperature as rotameters.

Mass flowmeters (MFMs), sometimes called thermal mass flowmeters, are electronic devices that indicate flow by measuring the thermal properties of a given gas. In most commonly used MFMs, the gas flows through a precision tube that has a precise sampling or bypass capillary tube running in parallel with the main flow (see Fig. 10). The sampling tube samples or bypasses the primary flow at a ratio of approximately 100 to 1. The bypass

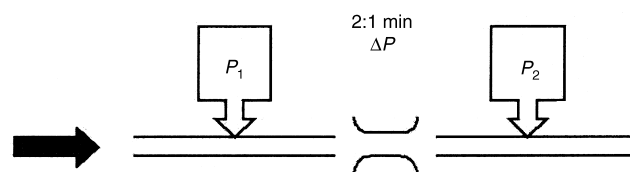


Fig. 9 Critical orifice gas flowmeters

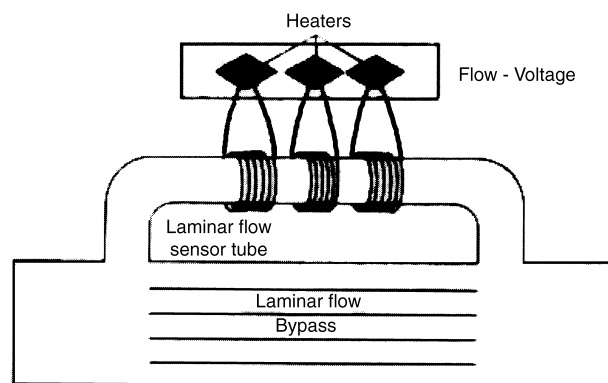


Fig. 10 Mass flow controller. Source: Courtesy of MKS Instruments

flow is passed through a controlled-heating element, which imparts a known amount of heat (i.e., energy) into the gas stream. Knowing the temperature of the gas, before and after the heating element, together with the thermophysical properties of the gas, the main flow through the meter can be accurately calculated. Temperature effects caused by supply parameters are eliminated because the temperatures are measured within the device. The effects of pressure variations on density are also eliminated because temperature changes, measured within the device, are based only on mass flow.

Mass flow controllers (MFCs) are identical to MFMs, with the exception of a control valve and an internal closed-loop control circuit to maintain the setpoint at the desired value. Upstream and downstream pressures must be defined in order to calibrate the valve for proper operation.

Mass flowmeters measure and compensate for temperature variations, have no parallax problems, and are relatively insensitive to pressure variations. Mass flowmeters and MFCs are, however, considerably more expensive than rotameters and critical orifices. Mass flow controllers can be controlled remotely and are ideal for intermediate and high-end thermal spray process controls.

Theoretically, rotameters, critical orifices, and mass flowmeters/controllers can all give accurate measurements, on the order of 2% error. Given the limitations and benefits described, the thermal spray application, its economics, and the need for process control drive the choice of gas flow measurement instrument.

Related Measurement Issues

Terms that define the ability of an instrument to provide useful information include resolution, accuracy, linearity, and repeatability.

Resolution relates to the smallest unit that is readable or resolvable. If a scale is divided into 100 units for 100% full scale, the smallest resolvable increment is 1%. Reading between the lines, so to speak (known as interpolation), does not increase the true resolution. For a variety of reasons, no two people will interpolate the same number. It thus follows that either larger-diameter analog gages or longer rotameter tubes, with finer divisions, provide higher resolution. Resolution in digital electronic devices is determined by the “bit” (binary digit) divisions of the scaled signal. For example, if a full-scale signal represents 100 psi, and the signal is processed through an 8-bit device, the signal will be divided by 2^8 (256). The internal electronic resolution becomes 0.391 psi.

Accuracy is a term used too loosely. In instrumentation, accuracy typically has two definitions: percent of full scale (FS) and percent of reading. Percent full scale defines limits related to the maximum measurable value of a device. As an example, if a 100 psi gage has a stated accuracy of $\pm 2\%$ FS, the gage would have a maximum error of ± 2 psi at any reading within its range. (Accuracy numbers are always plus/minus, \pm , whether stated or not.) Alternatively, a stated accuracy of 2% of reading, for a similar 100 psi gage, would yield a maximum error of 0.2 at 10 psi and 2 at 100 psi.

Linearity defines the deviation of the response of the measurement from a straight line, assuming that the device under consideration outputs a straight-line function. In reality, measurements taken with any device vary nonlinearly within stated accuracies. Errors may be positive over one or more portions of a flow curve and negative in others. The absolute variance at a given point over the reading gives percent linearity.

Repeatability mostly relates to hysteresis and is more noticeable in mechanical devices, although it is present in all processes and systems. When elements in analog gages, rotameters, and pressure transducers are cycled through their ranges, accuracy, linearity, and resolution trade off to give what should be a reasonable representation of the actual forces. Hysteresis comes into play when the forces on the elements are relaxed and the indication begins to reverse direction. Hysteresis is a term used to define the effects of losses, internal to the measurement device itself, related to friction, viscosity, magnetic fields, and other forces. For example, when a gage is pressurized from 0 to 100 psi, all of the forces acting on the internal elements have the same polarity; that is, they are going in the same direction. When the pressure is lowered to 50 psi, for example, not all of the stresses that were generated to produce the indication of pressure at 100 psi relax. The difference between the indicated pressure and the real pressure, at that indication, is the hysteresis, expressed in percent. An example of hysteresis could be shown in a gage that accurately reads 50 psi as pressure is increased. However, as pressure is increased past the desired setpoint and then lowered back to 50 psi, the indication might be 50 psi although the actual pressure may be 51 psi. The error would thus be 2% of the reading. Electronic circuits also exhibit hysteresis, but not usually to the same extent as mechanical devices.

If resolution, linearity, and repeatability fall within the limits of the stated accuracy, as they should, then accuracy can be used to define the operating limits of a piece of equipment. To ensure accuracy, all devices, whether analog or digital, should be calibrated at regular intervals, not to exceed one year.

Gas Quality Issues

As advancements are made in plasma gun designs, anodes and cathodes are becoming long-term consumable items. When anode life was measured in terms of a few hours, gas contamination was less significant. With the development of anode and gun designs that can last for several hundred hours, gas purity has become an issue. Impurities, usually oxygen, cause rapid deterioration of anodes and cathodes because oxidation affects both the thermal and electrical properties of tungsten and copper. Several other factors can influence the life of plasma gun hardware—for example, dimensional control, water pressure, and number of start/stop cycles. This section focuses on gas purity as it relates to hardware life.

Gas purity is one of the more important factors in determining the lifetime of plasma gun hardware. Purity is expressed in several ways. Often gases are purchased in terms of percent purity. For example, good commercial-grade gases for plasma spraying are

stated to be at least 99.995% (“four-nines-five” purity). Argon is the most commonly used primary plasma arc gas. From a liquid source, argon has a stated purity of 99.998% and is generally more reliable than high-pressure gas cylinders. An expressed purity of 99.995% is another way of stating that a gas has 50 ppm of impurities.

Oxygen is the most critical gas contaminant, followed by hydrocarbon impurities. It has been empirically determined that 50 ppm of oxygen is the upper limit for plasma gases. Other gases, including hydrocarbons, make up the balance of the impurities. Nitrogen as an impurity is of little concern, as are other inert gases. Hydrocarbon contaminants cause discoloration of the electrodes and increased resistance at the surface. Upper limits of contamination have been empirically established at 50 ppm. Welding-grade gases, typically 99.995% pure, are adequate. When oxygen levels in helium and argon exceed 50 ppm, a blue/black discoloration will occur on the cathode. Indigo coloration always indicates oxygen; black can be an indication of methane in the argon.

Another important contaminant is moisture—typically of concern in hydrogen, although sometimes also in nitrogen. The dew point of hydrogen and nitrogen for plasma spray should be below -46°C (-50°F). Water, when dissociated by the plasma, releases oxygen.

Gas-purity levels are critical to long electrode lifetimes, but the supply gas is not the only potential source of contamination. The gas-delivery system is the most common source of oxygen contamination, leading to early failures in plasma guns. Regulators at the supply end are often neglected. These devices should be inspected and calibrated at least yearly. The most common failure is the control diaphragm. Diaphragms are usually made of polymeric materials that age harden or fatigue in service. Typically, a diaphragm will develop significant leaks before it fails. High-purity regulators with stainless steel bellows are recommended.

A common misconception is that systems under pressure do not leak inward. However, under high flow conditions, these systems can aspirate or leak inward. The result is the same: electrode failure. Other common sources of leaks are hoses, fittings, and pipe threads. Again, yearly inspection is strongly recommended.

Close monitoring of spray conditions and hardware performance will help to establish reasonable limits for a given installation. After a repeatable condition has been established that reduces coating quality or powder sprayability, these conditions may be used as indicators for gas contamination levels and hardware replacement. When a computerized plasma spray system is used, gun efficiency can be set to alarm when conditions are deteriorating.

Visual inspection is acceptable for determining the condition of gun hardware. Cathodes should be replaced when the tips become blunted or mushroomed, or when cracks form at the tip. In most cases, anodes and cathodes can be polished or cleaned with emery paper or fine steel wool, being careful not to remove too much material. Hardware should be replaced if cleaning reveals more serious damage.

The use of an oxygen analyzer is recommended to monitor gases and delivery systems and as a troubleshooting tool. Bad gas is seldom a problem, so attention to gas-purchasing specifications

is usually sufficient. Occasionally, even the most conscientious supplier will deliver bad gases.

Thermal Spray Controls

Equipment for each thermal spray process is outlined in the article “Thermal Spray Processes” in this Handbook. This section focuses on specific controls and data acquisition, highlighting plasma spray, the most complex process.

Control schemes can range from simple to complex and can be categorized accordingly. Commercial thermal spray controls follow this order:

- Open-loop, input-based
- Open-loop, output-based
- Closed-loop, input-based
- Closed-loop, output-based or adaptive control

The terms “open-loop” and “closed-loop” refer to the absence or presence of circuitry that provides feedback to change a process parameter based on some predetermined reference or setpoint. Open-loop implies manual control of process inputs. An example of simple open-loop control is a rotameter, where the flow is set manually, but there is no means to correct for flow variations apart from manual readjustment. Closed-loop control implies control by circuitry that automatically corrects for deviations from the setpoint. An example of closed-loop control is a thermal MFC. Mass flow controllers have internal feedback circuitry that controls a flow valve to maintain the desired flow even when it begins to deviate from the setpoint.

The terms input- and output-based refer to the point of control. Input-based control is based solely on providing utilities (pressure, flow, voltage, current, and mass) to the process. There is no feedback to the operator or process control system that would indicate a change in parameters or their effect on the process. The examples in the previous paragraph are typical of input-based control. Output-based controls provide an indication to the operator or to the process control system of changes in parameters or in the process. Examples of output-based controls would be temperature-limit indicators, gun back-pressure gages, or particle temperature/velocity monitoring.

The vast majority of process controllers manufactured and used for thermal spray are open-loop, input-based controls. Examples of these controls are rod and wire flame spray systems where gas and air pressures, gas flows, and wire speed are all set and maintained by the operator. An example of this type of control for plasma spray would include voltage, current, gas pressure, and gas flow. With electric arc spray the controls are simply voltage, current, and atomizing air pressure. Open-loop controls are inexpensive in terms of capital investment, but may cost more in terms of poor coating quality, equipment downtime, and consumables. Open-loop controls are responsible, in part, for the coating repeatability and booth-to-booth equivalency. Open-loop systems have been used from the earliest days of thermal spray.

Closed-loop subprocesses are often used in thermal spray controls, but are not always apparent to the operator. Thermal mass

flow controllers, heat exchangers, water chillers, powder feed-rate controllers, and constant-current power supplies are all examples of closed-loop, output-based controls. In each of these subprocesses, a setpoint is specified and the subprocess is automatically maintained based on the output rate of deviation from the setpoint. The main shortcoming of these subprocess controls is that there is no direct interaction between them. These types of subsystem controls have been commercially available since the mid-1980s.

Advanced controls have been, and are being, developed around plasma arc spray, for two reasons: (1) Plasma is the most complex of the thermal spray processes, and (2) the aircraft industry, which in the past has driven much technology development, requires advanced controls. Advanced controls are typically based on computers or programmable logic controllers (PLCs). This class of control includes both closed-loop, output-based and adaptive controllers. Closed-loop subprocesses are used extensively for advanced controllers; these include MFCs, water chillers, powder mass flow controllers, motion controls, and constant-current power supplies. Setpoints for the subprocesses are established according to a "recipe" in the computer or PLC. A recipe is a set of retrievable data that includes all processing parameters for the gun and ancillary equipment.

Usually, these controllers have multiple high- and low-limit alarms that can warn the operator of potential problems or shut down the process. The alarms are separate from system fault limits, which are engineered into the process for safety. During processing, data are recorded for on-line control and analysis or off-line analysis and statistical process control (SPC). An example of on-line control in an output-based control scheme would be a running calculation of input power to the plasma gun. In this example, voltage and current data would be acquired and power would be calculated. The output of the calculation would be used as the feedback for comparison with a setpoint for power applied to the gun. If a deviation from the setpoint occurs, then the current is usually adjusted to compensate. This type of control has been commercially available since the mid-1980s.

The first adaptive control (developed by D.E. Crawmer and colleagues at Battelle Memorial Institute, circa 1988) used multiple data acquisition points to account for all significant energy losses in the plasma process. These data were manipulated to produce an accurate number for the effective energy in the plasma stream (a technique also used for HVOF processes). By creating an energy balance equation, the actual energy of the plasma stream became a control point. Using plasma energy as a control point in a closed-loop, output-based control scheme, compensation could be made for deterioration within the gun and water-cooled power cables. The premise for this control was that if the subprocess controllers delivered fixed quantities of powder, gas, and power to the gun, and compensation could be made for losses in the gun, then coating quality could be held constant. This control has been commercially available since the early 1990s. This approach to adaptive control is limited primarily by powder consistency.

The limitations of early adaptive control were obvious from the beginning. Out-of-tolerance powders make bad coatings, even

when jet temperature and velocity are held constant. The problem of powder-quality variations has been addressed, beginning in the mid-1990s. Diagnostic tools of the type developed in the mid-1980s by J. Fincke and colleagues at the Idaho National Engineering and Environmental Laboratory became commercially viable a decade later. By the mid-1990s commercial devices were available to measure particle temperature and velocity in real time. These tools have been successfully integrated into a control scheme that enables arc gas flow and energy to be manipulated simultaneously to effectively change the particle temperature and velocity in-flight. The advantage of this approach is that the controller can compensate for variations in powder particle size and distribution by continuously modifying the jet characteristics. The hope is that this type of control will become affordable and therefore broadly available.

Plasma Arc Starting Problems

This section discusses common causes and practical solutions for arc starting problems. The arc starting method most commonly employed in thermal spray is to induce a high-voltage (10 to 50 kV), radio-frequency (RF) pulse onto the negative power cable that feeds the cathode in the plasma gun. The RF pulse is generated by a spark-gap oscillator similar to radio transmitters used in the early days of Morse code transmission. This RF "signal" is typically 10 kV at ~1 MHz. When the RF pulse reaches the cathode and charges the tip, it breaks down across the narrow cathode/anode gap and discharges to the anode. The conducting spark generated between the cathode tip and the anode creates a current path for the open-circuit voltage of the dc plasma power supply. In a well-maintained plasma system, an RF pulse of 0.1 s duration is sufficient to initiate the plasma arc. Many factors can cause this energy to dissipate before it reaches the cathode tip. Among the potential problems are defective power cables, water purity, plasma gun hardware, and arc starter hardware. Radiofrequency energy will always find the shortest path to ground.

Conductors in the water-cooled power cables that supply power to the gun tend to break down because of repetitive motion during spraying. Because of the direction of current flow and the mechanical configuration of the electrical connections, the negative power cable, at the gun end, tends to exhibit a higher failure rate than the positive cable. The inner electrical cable suffers fatigue, resulting in work hardening, and begins to break down, one strand at a time. At some point, before complete failure of the cable, the RF energy is dissipated and does not reach the cathode or simply becomes too weak to break down the gap to the anode. A simple measurement of the voltage drop across the cables will help to establish a reference point for the replacement of worn cables. When new cables are installed on a plasma system, a reference voltage drop (at a fixed current) should be noted. Periodic inspection of the cables, by comparing the present voltage drop (at a specified current) to the initial drop, will help to monitor cable deterioration. Advanced controllers measure individual cable voltage drop and electrical resistance in process.

Water-cooled power cables are jacketed by high-pressure water hoses. These hoses are made of materials that have electrical properties suited for plasma spray applications. However, after extended use, the hoses can begin to break down electrically and lose their resistance to RF. The dielectric properties of the hose material can be affected by abrasion, repetitive arc starts, and impregnation by powders, among other things. The polymeric hose materials break down after repeated arc starts, allowing the RF to dissipate to a ground point, through the walls of the hose. These ground points include adjacent fixtures, tooling, decking, and other conductive surfaces. It is good practice to route cables away from, and off, metal walls, conduits, raceways, and decking. Even new cables can dissipate RF over long runs when in contact with conductive surfaces. Where it is necessary to run cables near metallic surfaces, adding extra insulation will help avoid problems. Polymeric sheeting, such as neoprene or silicone rubber, is generally acceptable. Using insulated standoff hardware also works well. Structural features such as sharp edges and corners should be avoided when routing cables. These points of contact tend to be sites where fretting can occur. As hoses wear, the RF will pass to ground through the weakened wall. Again, adding extra insulation in the form of polymeric sheeting will relieve these stress points.

Robotic manipulation of the plasma gun is a significant cause of rapid cable deterioration. High velocities and exaggerated movements associated with robot motion have a tendency to cause accelerated cable wear. This problem can be mitigated by securing the cables at the joints of the robotic arm, allowing enough strain relief for unrestricted motion.

The electrical conductivity of the cooling water in the cables can affect arc starting, due to RF dissipation. It has been documented that good-quality tap water and distilled water measure about 400 micro-mhos and normally do not affect arc starting. It has also been observed that water with a conductivity of 800 micro-mhos can significantly affect arc starting. In closed-circuit water-cooling systems, the simplest fix for this problem is to change the water periodically. If an open-loop system is being used and the water is too conductive, water treatment may be necessary. The use of particulate filters and/or deionizers will usually lower conductivity. Maintenance performed on water-distribution systems can also temporarily affect water purity/conductivity.

Mineral deposits and corrosion resulting from poor-quality water can inhibit arc starting. Oxidation of the electrodes (anodes and cathodes), usually the result of gas supply leaks or contaminated gases, will affect the ability of the RF to initiate the plasma. Inspect and clean anodes, cathodes, housings, and other metallic components when maintenance is performed on guns. It is necessary to keep plasma-gas distribution systems free of leaks. This is best accomplished through scheduled inspection and maintenance. Generally, plasma gases should contain less than 50 ppm oxygen.

The high-frequency (HF) unit is a broadband spark-gap generator that produces "white" RF energy. Dirt and oils, which can contaminate the inside surfaces of the HF unit, will tend to dissipate the RF energy within the unit. The HF unit should be inspected and cleaned periodically. Condensing moisture on or in the HF unit can also dissipate the RF energy. This usually occurs as strong weather fronts move through a region. Again, compressed air may relieve the problem. Drying the unit with heated air can also be effective.

Placing the HF unit in an area that is less susceptible to condensation is preferred. Arcing inside the HF unit can result from dirt, oil, and moisture as described previously. Arcing usually occurs on or around the high-voltage components, creating arc tracks. Arc tracks are black, carbonaceous lines leading from the conductors to a ground point.

The points that form the spark gap in the RF generator need to be kept clean, flat, and polished. Points should be inspected regularly, cleaned, and set to the appropriate spacing.

Finally, water condensation on and in the plasma gun can dissipate the RF pulse. Refer to the "Water Cooling" discussion in the next section for the causes of condensation.

Ancillary Equipment

Motion control is necessary to ensure operator safety and coating quality. Although handheld spraying has been carried out for nearly a century, it is not the preferred or safest means of spraying. The thermal spray environment is harsh and not suited for handheld operations. Safety has become more of an issue as HVOF and plasma energies and velocities have continued to increase. Operator fatigue (typical surface speeds are 30 m/min, or 1200 in./min) and inconsistency also affect coating uniformity. Part- and gun-handling equipment is required to eliminate the operator from the process. Commercially available, off-the-shelf motion-control equipment usually takes the form of turntables, *x-y* manipulators, and six-axis robots. Where the production volume of a specific part warrants the investment, specialized tooling is often used. The disadvantage of using manipulators is in spraying small numbers of complex parts. In limited production runs, programming the manipulation equipment can be more expensive and time consuming than spraying the part. There are many applications where handheld spraying seems to be the only practical approach to the problem—for example, bridges, boilers, architecture, artwork, and so forth.

High-volume production spraying dictates that ancillary processes such as surface cleaning, masking, and grit blasting also be automated. Coating process cells, which represent the highest level of automation, may include pick-and-place robots to handle part loading and unloading. Figure 11 shows an automated ther-

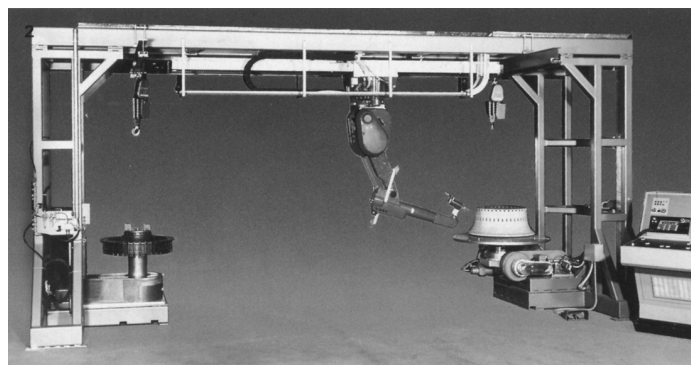


Fig. 11 Advanced thermal spray cell. Source: Courtesy of Sulzer Metco

mal spray cell for coating aircraft engine components. In this plasma spray cell, the operator loads and unloads parts, and the spray cell does all of the operations, including process control, statistical process control, quality inspection for thickness, and final processing. Although expensive, such equipment is justified for coating applications where consistency is critical.

Water Cooling. Plasma, HVOF, detonation spray, and some electric arc spray systems require either water or air cooling to maintain gun stability or effectiveness. The HVOF processes have input energy levels of 528 to 1055 MJ (500 to 1000 kBtu). Input energy to plasma guns range from 72 to 720 MJ (20 to 200 kW, or 68 to 683 kBtu). One commercial electric arc spray gun operates up to 1500 A dc, requiring water cooling. Water cooling is necessary for these high-heat flux spray guns to prevent the anodes and nozzles from melting.

Internal water pressure is more important than water flow. However, internal pressure is developed and more easily controlled by flow. Water pressure is required to suppress nucleate or film boiling in the gun. It has been observed in some areas of the world that plant and city water-supply temperatures are too high such that, for normal water pressures, boiling still occurs. Increasing water flow/pressure can again suppress boiling. However, most thermal spray cooling loops are limited by the hoses to pressures of ~1.7 MPa (~250 psi). As mentioned in the section "Plasma Arc Starting Problems," heat transfer to the water is greatly diminished when the water boils and the anodes/nozzles become oxidized. The thermal properties of water are, for all practical purposes, constant between freezing and boiling. Temperature control of the cooling water is important for both processes, but for different reasons.

It is recommended that the inlet water temperature to plasma guns be kept above the dew point of the environment. This measure will prevent condensation from collecting on and in the gun. Condensation can have two deleterious effects on the gun itself. First, condensation can cause arc-starting problems by dissipating the RF pulse. Second, condensation in the arc chamber will dissociate when the plasma is initiated, momentarily forming an oxygen-hydrogen plasma that is very damaging to the anode, significantly shortening anode life.

Water temperature in the plasma gun has a direct effect on boundary layer thickness, which in turn will affect the trajectory of internally, radially injected powder. Therefore, cooling-water temperature for a plasma gun should be kept at a constant temperature, above the dew point. To avoid seasonal coating variations induced by dew-point variations, the inlet temperature can be set for the yearly worst-case condition for condensation.

In HVOF, cooling water has the same effect on boundary layer thickness. However, the powder-injection schemes in most HVOF guns are such that boundary layer thickness does not affect the operation. Water cooling in combustion processes can cause combustion products (carbon) to deposit on the nozzle walls, especially when the inlet temperature is too cold.

Condensation also affects HVOF guns. Because HVOF guns are internally injected, condensation inside the gun can cause powder plugging. Generally, inlet water temperatures are kept above 20 °C (70 °F). In some HVOF gun designs, inlet water directly impinges the nozzle wall. In these designs, hotter water is

required to keep a cold spot from developing on the nozzle, which results in powder buildup inside the nozzle. It has been determined that an inlet temperature of 30 °C (85 °F) is sufficient to prevent buildup.

Water-cooling schemes are divided into three categories: open-loop (where the water is not recycled), water-to-water heat exchangers, and refrigerated systems. Utility or city water cooling is not recommended for many reasons. The most obvious is waste. However, there are several technical considerations. Untreated city water causes corrosion in gun water passages, which affects cooling efficiency and leads to shortened electrode life. In addition, city water is frequently too conductive, causing arc-starting problems. Water-temperature variations in city water systems are a significant problem. Temperature variations are caused by seasonal effects on the water supply and by temperature variations imposed by the environment and demands of the plant or factory. Seasonal variations in water temperature greater than 30 °C (55 °F) have been reported. Temperature variations in plant water cooling loops can be as high as the seasonal variations.

Closed-loop cooling systems are preferred, whether water-to-water or refrigerated. Such systems are capable of providing constant-temperature water input, within a few degrees. The cooling capacity of closed-loop systems should be matched to the load imposed by the process to prevent the control circuit from oscillating. Water conditioning, deionization/demineralization, and buffering should be applied with some caution in trying to prevent nozzle corrosion or freezing. Any water treatment affects the thermal and electrical properties of the water in some way. Deionizers are used in some systems to minimize corrosion in the gun and control conductivity. Pure deionized water, however, is corrosive, and shortened anode lifetimes have been observed when deionizer parameters have been set too high. One way to avoid the expense of water treatment is to use distilled water in the gun circuit of closed-loop systems. Monitoring water conductivity weekly or monthly and changing the water when it becomes too conductive is all that is necessary. Plant-side circuits of water-to-water systems are usually plumbed into either city water or the plant's closed-loop cooling circuit. Cost and availability of water determine the choice of cooling system.

ACKNOWLEDGMENT

This article has been revised and updated from a portion of "Equipment and Theory" by Ronald W. Smith, Lesson 2, *Thermal Spray Technology*, ASM International, 1992.

SELECTED REFERENCES

- Advances in Thermal Spraying, Proc. 11th International Thermal Spray Conf., 8–12 Sept 1986 (Montreal), Pergamon Press, 1986
- C.C. Berndt, Ed., *Thermal Spray: International Advances in Coatings Technology*, Proc. International Thermal Spray Conf., 29 May–5 June 1992 (Orlando, FL), ASM International, 1992

- C.C. Berndt, Ed., *Thermal Spray: Practical Solutions for Engineering Problems*, Proc. Ninth National Thermal Spray Conf., 7–11 Oct 1996 (Cincinnati), ASM International, 1996
- C.C. Berndt, Ed., *Thermal Spray: A United Forum for Scientific and Technological Advances*, Proc. First United Thermal Spray Conf., 15–18 Sept 1997 (Indianapolis), ASM International, 1998
- C.C. Berndt, Ed., *Thermal Spray: Surface Engineering via Applied Research*, Proc. First International Thermal Spray Conf., 8–11 May 2000 (Quebec), ASM International, 2000
- C.C. Berndt and T.F. Bernecki, Ed., *Thermal Spray Coatings: Research, Design, and Applications*, Proc. Fifth National Thermal Spray Conf., 7–11 June 1993 (Anaheim, CA), ASM International, 1993
- C.C. Berndt, K.A. Kohr, and E. Lugscheider, Ed., *Thermal Spray 2001: New Surfaces for a New Millennium*, Proc. International Thermal Spray Conf., 28–30 May 2001 (Singapore), ASM International, 2001
- C.C. Berndt and S. Sampath, Ed., *1994 Thermal Spray Industrial Applications*, Proc. Seventh National Thermal Spray Conf., 20–24 June 1994 (Boston), ASM International, 1994
- C.C. Berndt and S. Sampath, Ed., *Advances in Thermal Spray Technology*, Proc. Eighth National Thermal Spray Conf., 11–15 Sept 1995 (Houston), ASM International, 1995
- T.F. Bernecki, Ed., *Thermal Spray Research and Applications*, Proc. Third National Thermal Spray Conf., 20–25 May 1990 (Long Beach, CA), ASM International, 1991
- T.F. Bernecki, Ed., *Thermal Spray Coatings: Properties, Processes and Applications*, Proc. Fourth National Thermal Spray Conf., 4–10 May 1991 (Pittsburgh), ASM International, 1992
- C. Coddet, Ed., *Thermal Spray: Meeting the Challenges of the 21st Century*, Proc. 15th International Thermal Spray Conf., 25–29 May 1998 (Nice, France), ASM International, 1998
- D.L. Houck, Ed., *Thermal Spray: Advances in Coatings Technology*, Proc. National Thermal Spray Conf., 14–17 Sept 1987 (Orlando, FL), ASM International, 1988
- D.L. Houck, Ed., *Thermal Spray Technology: New Ideas and Processes*, Proc. National Thermal Spray Conf., 24–27 Oct 1988 (Cincinnati), ASM International, 1989
- F.N. Longo, Ed., *Thermal Spray Coatings: New Materials, Processes and Applications*, Proc. Second National Conf. on Thermal Spray, 31 Oct–2 Nov 1984 (Long Beach, CA), American Society for Metals, 1985
- E. Lugscheider and P.A. Kammer, Ed., *United Thermal Spray Conference*, Proc. United Thermal Spray Conf., 17–19 March 1999 (Düsseldorf) DVS-Verlag, 1999
- A. Ohmori, Ed., *Thermal Spraying: Current Status and Future Trends*, Proc. 14th International Thermal Spray Conf., 22–26 May 1995 (Kobe, Japan), High Temperature Society of Japan, 1995
- *Thermal Spraying: Practice, Theory and Application*, American Welding Society, 1985
- *12th International Conference Thermal Spraying*, 4–9 June 1989 (London), The Welding Institute, 1989

Introduction to Processing and Design

Frank N. Longo, Longo Associates

A NUMBER OF KEY PROCESSING STEPS are required in order to produce optimized thermal spray coatings. For example, to ensure adequate bonding of a coating, it is critical that the substrate be properly prepared. The substrate surface must be clean, and usually must be roughened after cleaning by grit blasting or some other means. Masking and preheating of the substrate also must be carried out prior to the application of thermal spray coatings; see the article "Coating Processing" in this Section of the Handbook for more detailed information.

Coating quality also depends on spray process variables/parameters such as spray pattern, coating buildup, process variations, temperature control, torch and substrate motion, and deposition efficiency. These important parameters, which can introduce some of the limits controlling designed usefulness of sprayed coatings, are described in the article "Coating Operations."

Postcoating operations further enhance the quality of thermal spray coatings. As discussed in the article "Postcoating Operations," these operations include finishing treatments (such as grinding and polishing), densification treatments (fusion, hot isostatic pressing, and heat treating), sealing, and testing and inspection.

In addition to these processing steps and parameters, it is important to understand how to design coatings for optimal performance. Thermal spray coatings are fabricated by combining equipment (spray guns or torches, manipulators, etc.), feedstock materials (powders, wires, or rods), and process know-how (experience). Only when all components of the spray process are used correctly does a well-designed coating result.

This introductory article reviews the advantages and disadvantages associated with thermal spray processing. Good processing practice allows the advantages of thermal spray coatings to be realized, while minimizing some of the disadvantages.

Advantages and Disadvantages of Thermal Spraying

Thermal spray coatings exhibit a unique microstructural architecture. Coatings are formed as melted and partially melted particles of different sizes impact substrates at a rate of perhaps one million particles per second, and build up one upon the other, as shown in Fig. 1. Figure 2 shows the typical "lamellar" (meaning layered) thermal spray coating microstructure that results. This unique morphological architecture imparts several advantages and disadvantages.

Advantages

Many of the defects identified in Fig. 2 are controllable through appropriate equipment and feedstock selection, but by far the

greatest influence on coating structure comes from the actual processing step. Properties of a sprayed coating stemming from the lamellar shape of the flattened particles or "splats" and residual porosity can be overcome only by postdeposition treatments. Porosity, however, can sometimes be a benefit. Surface porosity can retain lubricating oils and thus replenish a bearing surface with lubrication during periods when the oil supply is diminished. The rapid particle cooling rate and resulting lamellar splat shapes are perhaps the two features that distinguish thermal spray coatings from other coatings. Brittleness, hardness, anisotropic properties, and high internal (residual) stresses are all the result of very rapidly cooled and flattened particles. Properly applied thermal spray coatings have many uses and offer several advantages:

A wide range of materials can be deposited as coatings, including metals, alloys, carbides, oxide and nonoxide ceramics, refractory metals, plastics, cermets, and special composite structures comprising metals, ceramics, and plastics, as well as combinations of these. Competitive coating processes do not afford this versatility.

Low Processing Costs. Rapid rates of deposition—from 1 to 45 kg/h (2 to 100 lb/h) or more—can be achieved. Typically, 2 to 7 kg/h (5 to 15 lb/h) is normal practice. Rapid spray rates and coating deposition result in relatively low processing costs.

Wide Range of Coating Thicknesses. Coating thicknesses from 50 μm to 6.5 mm (0.002 to 0.250 in.) are used today. Competitive coating capabilities on the basis of thickness are as follows:

- *Little to no dimensional change.* A surface is modified without a finite coating being applied. Examples: diffusion processes, nitriding, carburizing, and ion implantation
- *Thin films.* Finite coatings up to 0.01 μm (0.4 $\mu\text{in.}$) thick. Examples: chemical vapor deposition (CVD), physical vapor deposition (PVD), sol-gel, diamondlike films, and ion plating
- *Intermediate coatings.* Coatings from 0.01 to 0.05 μm (0.4 to 2 $\mu\text{in.}$) thick. Examples: PVD, plating processes, organometallic coatings
- *Thick coatings.* Coatings from 50 μm to 6.5 mm (0.002 to 0.250 in.) or more in thickness. Examples: thermal spraying, welding processes, cladding, some plating processes, and some PVD or electron beam physical vapor deposition (EB-PVD) up to 0.254 mm (0.010 in.) thick

Wide Application Range. Thermal spray coatings function effectively for a broad range of applications, including:

- Wear resistance—abrasive, adhesive, galling, antifretting, cavitation, and erosion
- Multilayered thermal barrier coatings (TBCs) comprising metallic bond coats and oxide ceramic topcoats

- Abradable and abrasive coatings for gas-turbine engines
- Atmospheric and aqueous corrosion control
- High-temperature oxidation resistance and corrosion control
- Electrical resistance and conductivity
- Net- and near-net-shape component manufacturing
- Metal and ceramic-matrix composite structures

Equipment Costs and Portability. Basic thermal spray equipment is relatively low cost (ranging from \$7000 to \$10,000 for low-velocity combustion or electric arc torches to \$75,000 for air plasma torches), is small and portable, and allows on-site in situ spraying. The exceptions are vacuum plasma spraying (VPS) and fully integrated plasma systems or high-velocity oxyfuel (HVOF) systems for in-house original equipment manufacturing. Most hardfacing processes are portable, although some are not. Vapor deposition processes, on the other hand, are larger, more sophisticated, and complex and costly.

Minimal Thermal Degradation to Substrate. With proper control, there is little risk of thermally degrading the substrate during spraying. Thermal spraying is a relatively “cold” process, and substrates usually are kept below 150 °C (300 °F). Higher temperatures are achievable if desired and can be used if provisions are made to control oxidation.

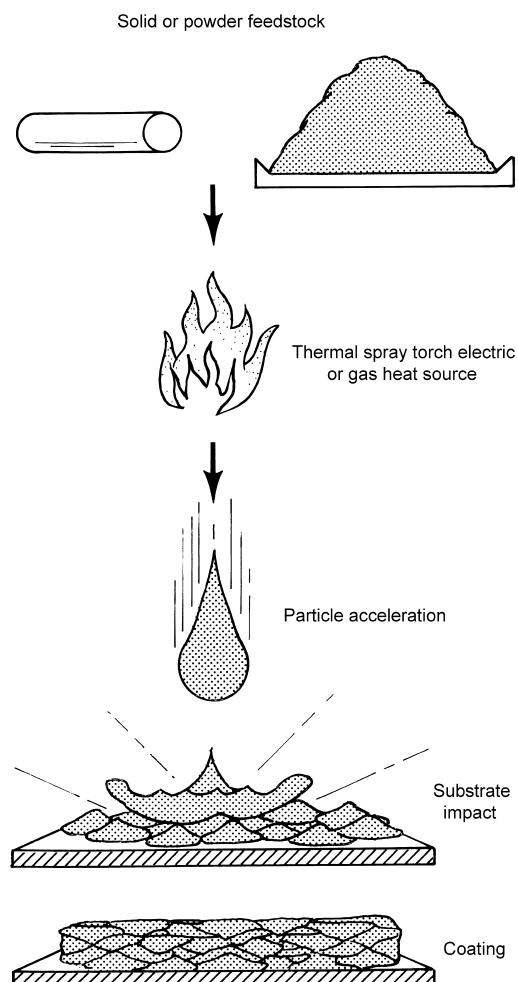


Fig. 1 Schematic of the thermal spray process

Disadvantages

Thermal spray coatings also have shortcomings, and these are important to understand so that designers can avoid placing thermal spray coatings in situations where satisfactory performance is unlikely. Often, failure occurs because coating limitations were never considered or perhaps poorly understood.

Low Bond Strength. The tensile bond strength achieved via thermal spraying is relatively low compared with other coating processes. Using ASTM Standard C 633, tensile adhesion is measured at between 41 and 83 MPa (6 and 12 ksi). Plating, welding, and vapor deposition processes usually have minimums at 103 MPa (15 ksi), depending on the coating and substrate chemistry. Welding adhesion is of course substantially higher than 103 MPa (15 ksi).

Porosity. Coatings are generally somewhat porous, allowing the passage of gases and/or liquids through to the coating/substrate interface. Porosity is controllable down to levels below approximately 1%, and while some porosity is sometimes beneficial—for example, in TBCs—achievement of a truly pore-free coating requires postdeposition treatments such as fusion or hot isostatic pressing. Through-porosity can be minimized using HVOF combustion and VPS processes. Surface porosity, however, also can be minimized through the application of liquid organic sealers, as discussed in the article “Postcoating Operations” in this Section of

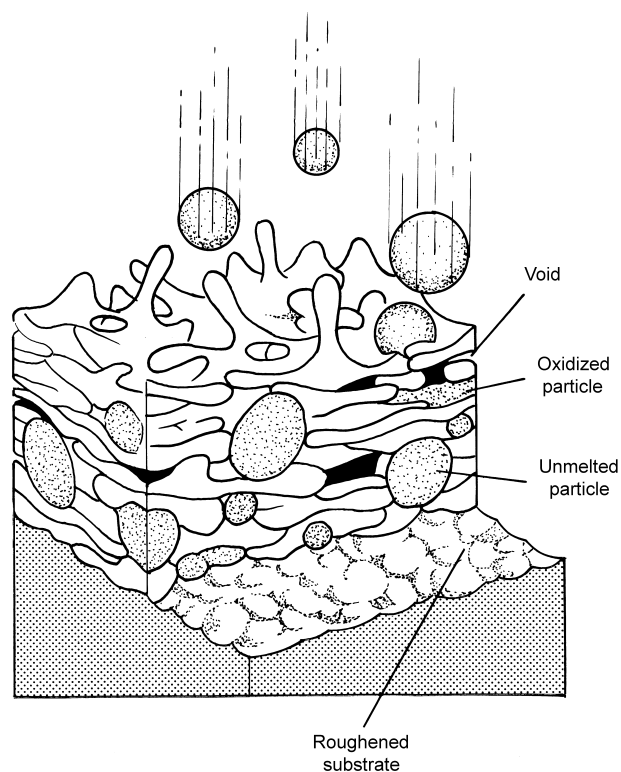


Fig. 2 Thermal spray coating. Buildup of a thermal spray coating is a chaotic process. Molten particles spread out and deform (splat) as they strike the substrate, at first keying onto asperities on the substrate surface, then interlocking to one another. Voids can occur if the growing deposit traps air. Particles overheated in the spray jet can become oxidized. Unmelted particles may simply be embedded in the accumulating deposit.

the Handbook. Competing processes such as vapor deposition, plating, and hardfacing are normally pore-free.

Anisotropic Properties. Thermal spray coatings are anisotropic; that is, they tend to have up to 10 times the tensile strength in the longitudinal direction than in the direction parallel to the spray. They are generally more brittle than corresponding wrought or cast materials and do not resist impact loading unless postdeposition heat treated. Weld overlay processes, on the other hand, provide the best bulk coatings for impact resistance.

Low Loading Capacity. Thermal spray coatings are not generally used as structural members and do not perform adequately under point or line loadings. Technologies emerging from HVOF and VPS studies, however, have demonstrated coatings with potential for use as structural materials, with impact and line-loading capability. Typically, thermal spray coatings perform best when loaded in compression, explaining their widespread use for sliding wear resistance. Point and line loadings cause problems because underlying porosity fails to support the concentrated loads and the coating collapses, resulting in permanent surface deformation.

Line-of-Sight Process. Thermal spray deposition of coatings on simple geometrical configurations is relatively straightforward, but complex shapes or contours, as in the case of gas-turbine airfoils, require expensive, automated, computer-controlled, multi-axis manipulators or robots. Deviation from spraying normal to the surface ($90^\circ \pm 20^\circ$) will often compromise coating properties. Porosity will likely increase and coating integrity (cohesion) will decrease. Competing processes that are also limited by line-of-sight restrictions include ion implantation and PVD.

SELECTED REFERENCES

- C.C. Berndt, Ed., *Thermal Spray: International Advances in Coatings Technology*, Proc. International Thermal Spray Conf., 29 May–5 June 1992 (Orlando, FL), ASM International, 1992
- T.F. Bernecki, Ed., *Thermal Spray Research and Applications*, Proc. Third National Thermal Spray Conf., 20–25 May 1990 (Long Beach, CA), ASM International, 1991
- T.F. Bernecki, Ed., *Thermal Spray Coatings: Properties, Processes and Applications*, Proc. Fourth National Thermal Spray Conf., 4–10 May 1991 (Pittsburgh, PA), ASM International, 1992
- P. Fauchais, State of the Art for the Understanding of the Physical Phenomena Involved in Plasma Spraying at Atmospheric Pressure, *Proc. National Thermal Spray Conf.*, 14–17 Sept 1987 (Orlando, FL), ASM International
- D.L. Houck, Ed., *Thermal Spray: Advances in Coatings Technology*, Proc. First National Thermal Spray Conf., 14–17 Sept 1987 (Orlando, FL), ASM International, 1988
- D.L. Houck, Ed., *Thermal Spray Technology: New Ideas and Processes*, Proc. Second National Thermal Spray Conf., 24–27 Oct 1988 (Cincinnati, OH), ASM International, 1989
- H.S. Ingham and A.P. Shepard, Ed., *Metallizing Handbook*, 7th ed., Vol 1–3, Metallizing Engineering Co., Inc. (Metco), 1959
- E.J. Kubel, Thermal Spraying Technology: From Art to Science, *Adv. Mater. Proc.*, Vol 132 (No. 6), Dec 1987, p 69–80
- F.N. Longo, Ed., *Thermal Spray Coatings: New Materials, Processes and Applications*, Proc. National Thermal Spray Conf., 31 Oct–2 Nov 1984 (Long Beach, CA), American Society for Metals, 1985
- E. Pfender, Fundamental Studies Associated with the Plasma Spray Process, *Proc. National Thermal Spray Conf.*, 14–17 Sept 1987, (Orlando, FL), ASM International
- T.J. Steeper, D.J. Varacalle, Jr., G.C. Wilson, and V.T. Berta, Use of Thermal Spray Processes to Fabricate Heater Tubes for Use in Thermal-Hydraulic Experiments, *Thermal Spray Coating: Properties, Processes, and Applications*, Proc. National Thermal Spray Conf., 4–10 May 1991 (Pittsburgh, PA), ASM International, 1992
- *Thermal Spraying: Practice, Theory and Application*, American Welding Society, 1985

Coating Processing

Frank N. Longo, Longo Associates

PROCESSING OF THERMAL SPRAY COATINGS may involve some or all of the steps outlined in this article. Each step represents an essential part of the planning process that must be considered before and during the coating process. For instance, a first-time repair application will require some attention to all of the steps shown, whereas some of the steps can be omitted for an original-equipment-manufactured (OEM) component that needs to be coated.

Thermal spraying begins with proper surface preparation: cleaning and roughening by grit blasting, macromachining, the application of sprayed bond coats, or a combination of these procedures. Repairing worn parts sometimes requires undercutting to remove damaged surface material and squaring of the surface for dimensional restoration.

After surface preparation, masking protects selected component areas next to the zone being coated from unwanted abrasive and/or spray particle impact. Common masking methods include metal shadow masks, tough high-temperature tapes, and paint-on compounds. Some tapes offer protection from both abrasive grit and sprayed particles without damage; others do not.

Preheating, the next step before spraying, is used to drive off moisture and present a warm, dry surface to the first impacting particles. Aluminum, copper, and titanium substrates normally are not preheated. Excess preheating should be avoided, as it may oxidize the surface and impair bonding.

Substrate Metallurgy and Thermal History

The substrate material type, hardness, and toughness, as well as prior heat treatment, must be determined in order to evaluate how to proceed. This simple analysis quickly tells the user which substrate preparation to use and which to avoid. Metal type and thermal history also help the user determine the correct preheating temperatures to use during spraying.

Thermal spray coatings can be applied to virtually all metallic surfaces, and to wood, plastics, glass, paper, and so forth. The most frequently coated substrate materials are steels, iron, nickel alloys, titanium, aluminum, copper, and magnesium.

Carbon and alloy steels generally come in a heat-treated condition, with hardness as the major property indicator. Substrates with hardness under 40 HRC can be roughened and sprayed using most standard techniques. When the hardness exceeds 40 HRC, surface roughening with abrasive grit is difficult to achieve; as a result, coating adhesive strength is often adversely affected. The section "Roughening" in this article describes procedures for handling hardened substrates. There will be little change to the heat-treated condition of steel substrates provided that both the preheat

and operating temperatures are kept below ~150 °C (~300 °F). Steel substrates that go above 150 °C (300 °F), even for short periods of time, may begin to exhibit property changes that could manifest themselves as softening and/or warping.

Stainless steels and nickel alloys should be handled carefully. Preheating even to 100 °C (210 °F) will accelerate thickening of the transparent and tenacious oxide film on stainless steel surfaces, preventing good adhesion of sprayed coatings. Limit preheat temperatures to 65 °C (150 °F) and begin spraying immediately when that temperature is reached.

Aluminum, copper, magnesium, and titanium alloys require special handling when using abrasive grit for roughening. It is also advisable to avoid preheating before spray. A small amount of heat applied to the surface rapidly converts surface layers to oxides (i.e., TiO₂, Al₂O₃, etc.), which prevents good adhesion and may lead to complete failure of the mechanical bonding.

Casting or powder metallurgical (P/M) components may require special cleaning. See the section "Cleaning" in this article for details.

Part Configuration and Coating Location

Once the area where the coating is to be applied has been identified, determine whether the part is symmetrical about the coating location, which would allow fixturing for rotation and/or translation about an axis perpendicular to the spray torch. The stream of spray particles should impact the target surface as close to normal (90°) as possible (Fig. 1a and b). The minimum acceptable impact angle is 45° to the target area (Fig. 1c). A 45° spray angle impact should be used only as a last resort, bearing in mind that some coating property deviation from the optimum can be expected. Both bond strength and coating cohesion will be compromised.

Porosity increases dramatically as the angle of the spray torch moves from normal to 45° (Fig. 1d). If spraying must be done at angles between normal and 45°, first consider whether the compromised coating will be able to perform as required. To obtain this information, spray a sample coating at the impact angle expected and test its integrity before committing to spray the part.

The substrate should be inspected for defects and flaws, especially cracks. Structural flaws will be reflected in the sprayed coating. Such flaws cannot be repaired using a sprayed coating, nor does a sprayed coating add strength to the substrate. Sprayed coatings only enhance the substrate surface and add dimension, but do not act as structural elements providing improved tensile strength, fatigue resistance, or fracture toughness.

In addition to assessing the implications of adverse spray angles, when examining the substrate target area the following

acceptable practices and design considerations should be weighed for compliance with the target area (Fig. 2):

- When the substrate target area is undercut to accept a coating (Fig. 2a), always chamfer or radius the corners before spraying

ing. Sharp 90° corners capture loose spray particles, dust, and debris and leave large porous areas. Sprayed particles are unlikely to bridge the corner. See the discussions on masking and undercutting later in this article and the discussion on spray patterns in the article “Coating Operations.”

- Likewise, the edge of a sprayed coating applied across the full face of the substrate as shown in Fig. 2(b) should be chamfered or radiused. Typically, 0.75 mm (0.030 in.) radii or 45° chamfers are recommended. Loads applied to the edges of coatings that have not been chamfered or radiused will cause them to crack and break off, perhaps leading to total coating failure. If the substrate permits, it is also good practice to radius or chamfer the substrate *before* spraying. The coating will then conform to the edge profile, effectively forming the required chamfer or radius.

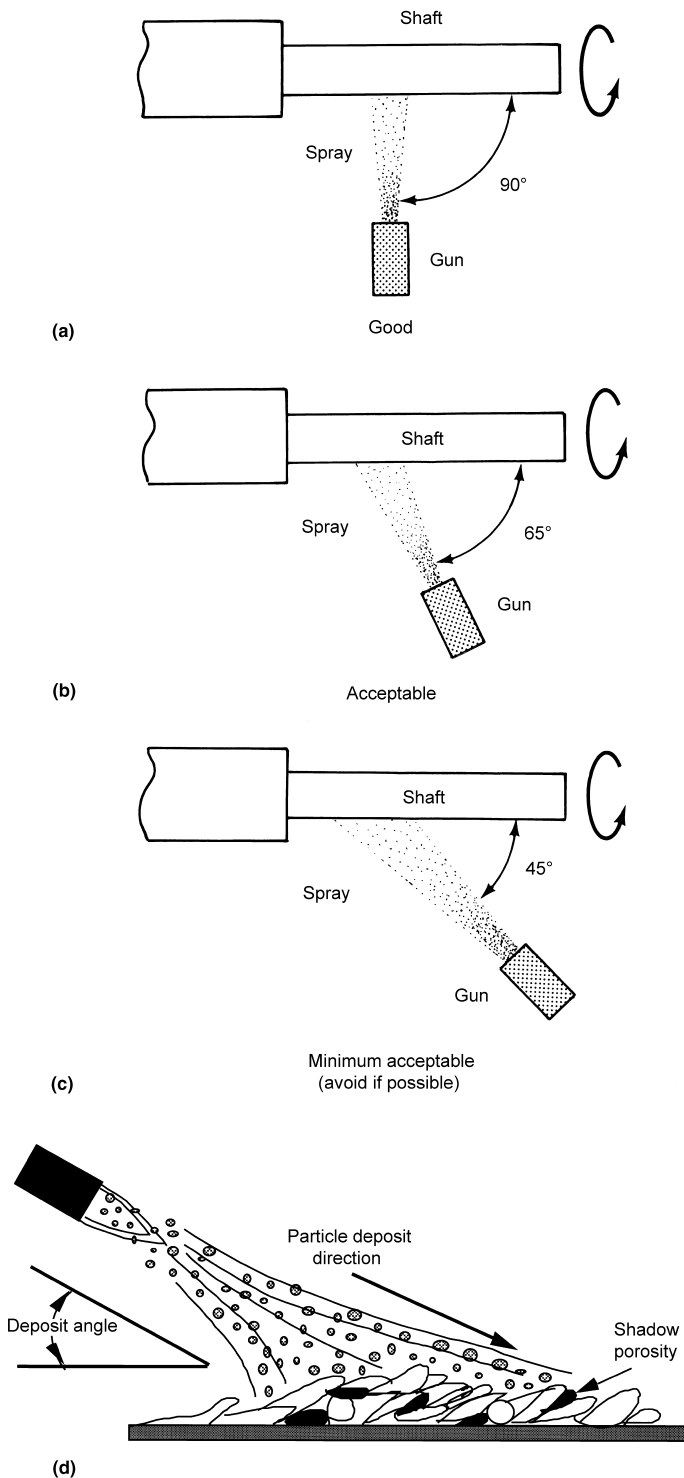


Fig. 1 Thermal spray stream positions. (a) Good. (b) Acceptable. (c) Minimum acceptable. (d) Formation of porosity in sprayed coatings when the spray angle is reduced to approximately 45°. Particles impacting at angles of less than 90° create a shadowing effect that results in increased coating porosity.

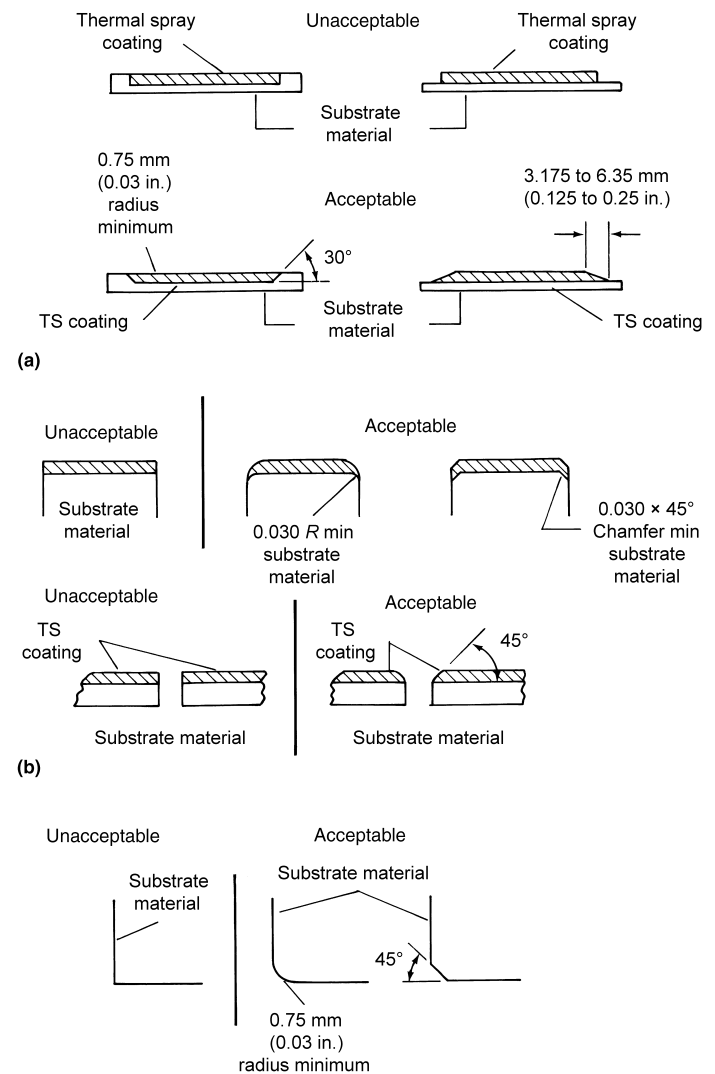


Fig. 2 Design of substrate geometry for thermal spray coating processes

- Figure 2(c) shows the acceptable practices for depositing a coating onto a substrate and up against a shoulder. Using procedures similar to undercutting, corners must be radiused to approximately 0.75 mm (0.030 in.) or chamfered. Sharp corners against a shoulder are likely to result in porous, weakly bonded coatings and act as sites for premature coating failure. See the discussions on masking and spray patterns for further insight into problems associated with spraying against corners.

Surface Preparation

Proper substrate surface preparation prior to thermal spraying is absolutely essential. Steps must be undertaken correctly in order for the coating to perform to design expectations. At worst, total failure could occur. For a one-of-a-kind part in for repair, the time for surface preparation and finishing may account for 50 to 75% of the total time required to complete the job. This represents a substantial investment, considering labor and overhead expenses, and should be done correctly the first time.

Cleaning

The first step in preparing a substrate surface for spraying is to remove contaminants such as oil, grease, paint, rust, scale, and moisture. Contaminants remaining between the sprayed coating and substrate will serve to minimize metal-to-metal contact at the interface and reduce adhesion (adhesive strength).

Vapor degreasing is commonly used to remove oils, greases, and other solvent-soluble organic compounds. It is fast, economical, and efficient. Hot vapor degreasing is a variation that is even faster and more efficient. Porous materials such as castings, P/M parts, or cast irons should be soaked for 15 to 30 min to dissolve hidden contaminants that may have penetrated the substrate. Large parts can be steam cleaned or submerged in solvents. Manual cleaning with solvents is often all that is necessary to remove surface greases and oils. Common solvents include:

- Methyl ethyl ketone (MEK)
- Acetone
- Aqueous washer solutions containing acetic acid

Chlorinated solvents such as trichloroethylene and 1,1,1-trichloroethane, while very effective, must no longer be used due to environmental restrictions.

To avoid the possibility of corrosive cracking, chlorinated solvents must be avoided when cleaning titanium and titanium alloys. Instead, use steam, acetic, or alkaline washes.

Baking. When solvent soaking does not work for porous materials such as castings or P/M parts, baking at 315 to 345 °C (600 to 650 °F) for a few hours will vaporize and remove deeply penetrated oils. Residual oils will “bleed” out of the substrate onto the surface during preheating and/or after spraying. The result is disastrous, as major coating debonding is likely to occur.

Ultrasonic cleaning can also be used to dislodge some contaminants in confined or hidden areas. Commercial equipment is available, together with the manufacturer’s recommended cleaning solutions. Check to be sure that the solution chosen is compatible with the substrate material. Ultrasonic cleaning can also be

used to remove residual entrapped grit after grit blasting. The substrate must be oriented, however, so that dislodged grit can fall free of the substrate.

Vapor honing/vapor blasting/wet abrasive blasting/liquid honing use a water-based slurry containing an abrasive medium that is projected by air jets toward the surface being prepared. The abrasive material will vary, but –200 mesh (–74 µm) aluminum oxide is preferred. Rust inhibitors and antisolidsifying compounds usually are added. Since these processes rely on an abrasive for cleaning, they can be used to remove light burrs, corrosion products, and paint as well as greases and oils.

Dry abrasive blasting rigorously removes surface contaminants. More aggressive than vapor honing, this process is used to remove old sprayed coatings, mill scale, paint, burrs, corrosion products, and oxides. Abrasive media are carried by high-pressure compressed air jets and directed toward the substrate surface. See the section “Dry Abrasive Grit Blasting” for further details. Never use the same abrasive equipment for both cleaning and roughening since contaminants from cleaning could spoil the surface.

Roughening

After cleaning, roughening is the most important step in preparing the surface to accept the sprayed coating. It is perhaps the single most critical step to ensure good coating adhesion. The principal methods used to roughen surfaces are:

- Dry abrasive grit blasting
- Machining or macroroughening
- Application of a bond coat

Combinations of these methods are often used—for example, bond coats applied over grit-blasted surfaces to provide the highest bond strength or grit blasting over macroroughened surfaces. Properly cleaned and roughened surfaces provide the critical interface upon which the first layer of molten or softened coating particles impact. A properly prepared surface has the following attributes:

- Cleanliness provides white metal-to-metal contact for interatomic and metallurgical interactions between the substrate and sprayed particles.
- Increased surface area. Roughening provides significantly increased surface area for particle-to-substrate contact, and increasing atomic and metallurgical interaction.
- Surface asperities and irregularities. Roughening provides many opportunities for mechanical interlocking.
- Macroroughening reduces in-plane (longitudinal) stresses by inducing folds in the coating. See the section “Machining and Macroroughening.”

Dry Abrasive Grit Blasting

Dry abrasive grit blasting is the most commonly used surface-roughening technique. Dry abrasive particles are propelled toward the substrate at relatively high speeds. On impact with the substrate, the sharp, angular particles act like chisels, cutting small irregularities into the surface.

The amount of substrate (plastic) deformation is a function of the angularity, size, density, and hardness of the particles and the

speed and angle at which the particles are directed toward the substrate. The action of the grit and/or shot causes irreversible plastic deformation of the surface material. The material beneath the deformed surface material remains elastic and tries to return the deformed surface material to its original (shorter) length. The resulting balance of forces places the outer layer of material in residual compressive stress, while the underlying material is in tension. To avoid reduction of fatigue life, titanium substrates are sometimes shot peened before dry abrasive grit blasting to reduce the underlying tensile stresses, effectively balancing the compressive/tensile forces.

Irregularities on a perfectly grit-blasted surface will have a large number of reverse draft pits, providing a partially hidden space into which sprayed molten particles can flow. Coatings sprayed to this surface are said to be positively keyed, creating what is commonly referred to as a mechanical interlocking bond. Figure 3 illustrates interfacial keying for mechanical interlocking.

Abrasive Grit Blasting Variables

Variables that determine the size and geometry of the surface irregularities include the size, shape, hardness, specific gravity, and chemistry of the abrasive particles, as well as their speed and angle of impingement. Hardness of the base material and thoroughness (coverage) of the blasting operation are other important variables. The significance of each is discussed briefly.

Abrasive Grain Size. Because the mass of an abrasive grain varies as the cube of its diameter, the force of the impact of a 20 mesh (840 μm) abrasive particle will, for example, be eight times that of a 40 mesh (420 μm) particle (only if the particles are spherical). Consequently, the larger particle will cut much deeper, everything else being equal. In preparing surfaces of machine elements where relatively thick coatings of hard metals are to be applied, abrasive particle sizes in the 8 to 20 mesh (2380 to 840 μm) range are generally required. For zinc and aluminum coatings, 12 to 50 mesh (1680 to 297 μm) abrasives are used.

Surface roughness values are generally expressed in microinch ($\mu\text{in.}$) or micron (μm) units. The American National Standard for surface roughness measurements calls for arithmetic average (AA) deviation from the mean (ANSI B46.1-1978, "Surface Texture," ASME). Root mean square (RMS) values are also com-

monly used. Surface roughness is easily measured using commercially available surface profilometers that average the heights (h) of all the peaks and valleys from the centerline traced by a diamond stylus and divides by the number of measurements (N). The AA can be expressed as:

$$AA = \frac{h_1 + h_2 + h_3 + \dots h_N}{N}$$

Particle Shape (or Morphology). The angularity of the abrasive particles profoundly affects the depth and shape of the pits cut into the substrate surface. Rounded shot will make smooth, rounded indents in the surface, providing little or no bonding. Rounded beach sand also affords very little bonding. Angular grit crushed from chilled iron shot provides a much stronger bond when new and sharp than when dulled due to prolonged use. In general, abrasive particles with many sharp, angular cutting edges are required.

Particle Hardness. Blasting particles should be hard enough to cut efficiently, but not so brittle as to shatter excessively. A certain amount of toughness (ductility) is desirable to avoid rapid breakdown.

Specific Gravity (Density) of Particles. Angular chilled iron grit has nearly twice the density of the heaviest nonmetallic abrasive and so will have nearly twice the impact energy at the same velocity, for equal-size particles. This will result in deeper pitting, and many users consider steel grit to be a suitable blast abrasive for machine element work.

Blast Speed. With any type of abrasive, the force of the impact varies directly with the impact speed. Hence, higher pressures result in deeper pitting. With nonmetallic abrasives, high pressures result in rapid shattering of grit particles, and for this reason blast pressures often are reduced to save on grit. In other cases, the maximum available pressure is used and the abrasive is considered to be expendable. As a rule, blast machines of the suction-feed type provide only one-third to one-half the speed possible with the force-fed pressure-generator type of blast machines.

Angle of Impact. It has been shown that if a surface is grit blasted at an angle from one direction and then sprayed at an angle from the opposite direction, a very weak bond will result. This clearly demonstrates that the sprayed metal particles must penetrate into the surface asperities or pits in order to mechanically key the coating to the surface. It is generally considered essential that the angle of impingement of the blast be similar to that of the spraying and be maintained as close to 90° to the surface as possible if maximum bonding is to be achieved. If substrate geometry requires a spray angle at less than normal, then it is advisable that grit blasting be done at the same angle to take advantage of re-entrant asperities providing better keying.

Substrate Material Hardness. There are two reasons why it is not possible to obtain a strong bond to hard metals through grit blasting. First, it is difficult or impossible to effectively "cut" the surface to the required depth. Second, and more important, hard materials lack ductility and surface metal cannot be displaced in a manner that provides rough asperities. When soft steel is grit

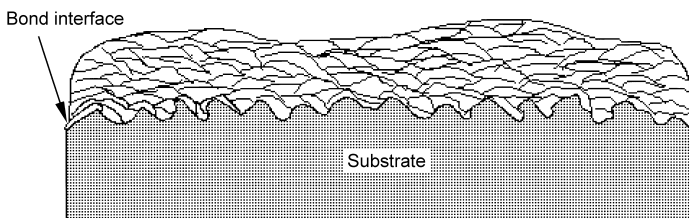


Fig. 3 Mechanical bonding, showing a properly grit-blasted surface with a large number of reverse draft pits into which sprayed particles flow to achieve positively keyed mechanical interlocking

blasted, an abrasive particle striking the surface adjacent to a previously cut pit will plastically displace the metal on the side of the pit to form a key or dovetail. On hardened steel, the displaced metal continually breaks away (brittle fracture) instead of deforming to produce a key. Blasting soft steel very thoroughly will raise the surface by 0.025 to 0.075 mm (0.001 to 0.003 in.). The same degree of grit blasting on hardened steel will actually cut away some of the surface.

Thoroughness of the Grit-Blasting Operation. It is not merely sufficient to grit blast until the surface is reasonably well pitted. If keys or anchors are to be formed, it is necessary to continue the blasting somewhat beyond the point at which the surface appears to be completely covered. This will ensure that a large number of the pits have been deformed sufficiently to provide reverse draft and positive keying. When using alumina grit, check to ensure that alumina is not embedded in the substrate, as this condition will substantially lower adhesion. Ultrasonic cleaning after grit blasting can help in this respect.

Surface Detail Size. The importance of the size of the surface detail of the prepared surface must be emphasized. High tensile residual stresses develop parallel to the surface in sprayed metals. Sprayed metals possess limited ductility; therefore, very slight lateral displacement of any part of the coating with respect to the substrate surface will result in shearing off the sprayed metal keys. Pits produced by heavy grit blasting—for example, by using G-16 angular chilled iron grit on mild steel—will be 5 to 10 mils (125 to 240 μm) in depth and up to 30 or 40 mils (750 to 960 μm) in width. Irregularities of this order of magnitude provide substantial resistance to shearing. It is readily apparent, however, that a more effective shear resistance can be obtained by macroroughening of the substrate surface, such as produced by grooving or threading prior to blasting. See Fig. 4 for a guide to surface roughnesses expected from alumina of various grit size on typical substrates.

Abrasive Grit Blasting Equipment

There are three basic types of grit-blasting equipment:

- Pressure machines or “blast generators”
- Blast cabinets with suction-type nozzles
- Centrifugal or “airless” blasting machines

Pressure machines are generally used for outside work, since they provide higher abrasive speeds and use air more efficiently than suction-feed types (see Fig. 5a). For inside work on large pieces, or for production blasting, pressure generators are used in blasting rooms. Production applications of this type sometimes include an abrasive elevator to convey the grit from a pit beneath the floor grating to a hopper above the blast generator. The system often includes a means for removing dust and ultrafine particles from the abrasive as it is transferred from the conveyor to the hopper.

Blasting speed with pressure-type machines depends on the nozzle size, among other variables. Other factors being equal, blasting rate is a direct function of the nozzle orifice area, or the

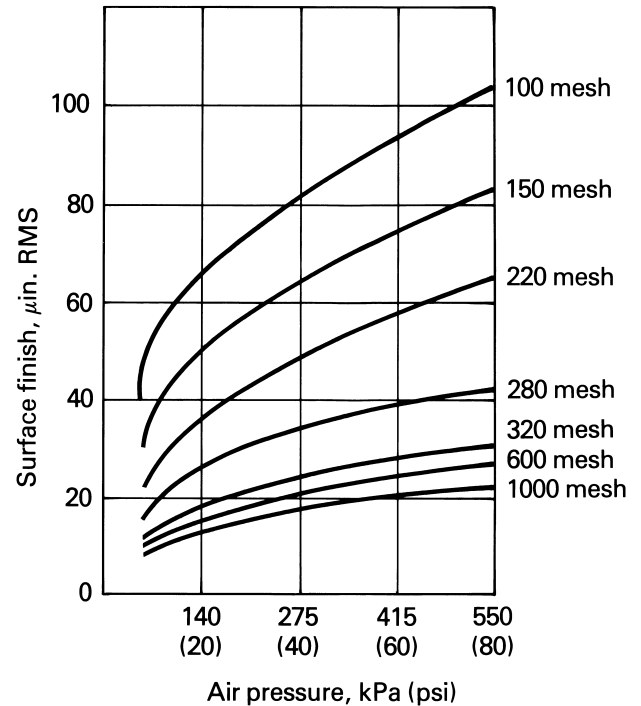


Fig. 4 Typical surface finish obtained using aluminum oxide abrasive. Curves illustrate the range of results obtainable by pressure and abrasive size variations. The exact results will be influenced by material differences, nozzle selection, and other variables.

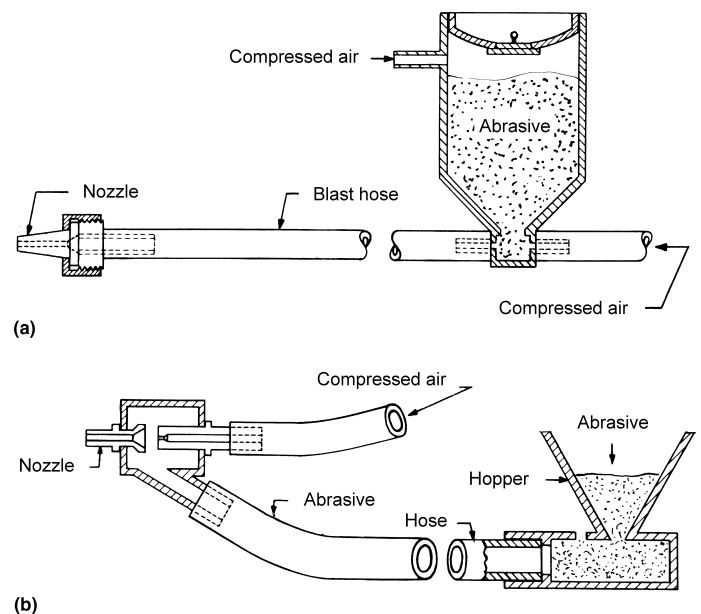


Fig. 5 Pressure-type (a) and suction-type (b) grit-blasting machines. Adapted from Ref 1

square of the diameter. Table 1 shows the consumption of air, sand, and chilled iron grit for various nozzle sizes and air pressures, and the horsepower needed to drive them. Actual compressor ratings should exceed the horsepower shown by 20 to 25% to reduce compressor wear.

Blasting cabinets usually employ suction-type nozzles (Fig. 5b). These do not provide as high an abrasive speed as pressure generators, and they are less efficient with respect to use of air; however, they are very convenient for small work. The abrasive is recycled so that periodic reloading is not needed. The operator remains outside the blasting area, so there is no need for helmets or specialized protective clothing.

Centrifugal blasting machines are ideally suited for production blasting. With this equipment, grit is thrown in a fan-shaped pattern against the substrate by a rapidly revolving paddle wheel. In some cases, the work is also rotated to expose all surfaces to the blast. Other machines may use two or more wheels that can be positioned to suit the substrate geometry. This kind of equipment is much more efficient than air-type blast generators in terms of power used. It is claimed that structural steel shapes or large-

diameter pipes can be blasted thoroughly at a cost of less than one cent per square foot.

Blasting Abrasives

This section discusses commonly used abrasives, along with some caveats regarding their use.

Angular chilled iron grit is the only practical material for use with centrifugal blasting equipment in preparing surfaces for flame spraying. It is also the preferred abrasive for use with pressure machines in blasting rooms. Chilled iron grit has a hardness of 60 to 62 HRC, and can be used on substrate materials as hard as 40 to 45 HRC. It is often erroneously called steel grit, but true steel grit has a hardness of 43 to 48 HRC and is too soft. Steel grit becomes dull and does not produce the sharply defined surface details needed for proper bonding of sprayed coatings. Chilled iron grit stays sharp much longer, and some fracturing occurs during use to produce new, sharp cutting edges.

On most work, a blend of grit sizes is more efficient than a single size. Maintaining the right grit size distribution requires that

Table 1 Consumption of air, horsepower, sand, and grit for various nozzle sizes and air pressures

Consumable	Air pressure, psig					
	50	60	70	80	90	100
0.19 in. (4.75 mm) nozzle size						
Air, cfm	26	30	33	38	41	45
Horsepower	3.6	4.6	5.5	6.9	7.9	9.3
Sand/h	188	214	245	270	298	330
Grit/h	470	532	612	674	748	824
0.25 in. (6.40 mm) nozzle size						
Air, cfm	47	54	61	68	74	81
Horsepower	6.4	8.3	10.2	12.4	14.2	16.8
Sand/h	336	390	443	510	560	618
Grit/h	840	974	1110	1275	1400	1545
0.31 in. (7.90 mm) nozzle size						
Air, cfm	77	89	101	113	126	137
Horsepower	10.5	13.6	16.9	20.6	24.2	28.4
Sand/h	585	668	755	840	925	1015
Grit/h	1460	1670	1885	2100	2310	2535
0.38 in (9.50 mm) nozzle size						
Air, cfm	108	126	143	161	173	196
Horsepower	14.8	19.3	23.9	29.3	33.2	40.6
Sand/h	835	955	1080	1200	1315	1440
Grit/h	2080	2390	2700	3000	3290	3600
0.44 in. (11 mm) nozzle size						
Air, cfm	147	170	194	217	240	254
Horsepower	20.1	26.0	32.4	39.5	46.1	52.6
Sand/h	1120	1290	1470	1640	1810	1980
Grit/h	2800	3220	3670	4100	4530	4950
0.50 in. (12.70 mm) nozzle size						
Air, cfm	195	224	252	280	309	338
Horsepower	26.7	34.3	42.1	51.0	59.0	70.0
Sand/h	1450	1670	1890	2100	2300	2530
Grit/h	3620	4170	4720	5250	5800	6320

cfm, cubic feet per minute

frequent small additions of the coarsest size in the mix be made. Normal breakdown will usually keep the grit size distribution in fairly good balance. Excessive fine particles should be removed periodically by screening, unless the installation is designed to remove these fines by air cleaning.

In any case, the aggregate grit size should be the smallest that will produce the desired surface characteristics. A blasting machine throws a certain number of pounds of abrasive particles per minute, regardless of particle size. A pound of G-18 grit contains 38,000 particles, whereas a pound of G-25 grit contains 150,000, and a pound of G-40 grit contains 520,000, so a change of only one mesh number results in a nearly fourfold increase in the number of particle impacts and, consequently, an increase in the area covered per unit of blasting time. There is an added advantage: The grit breakdown rate will be lower with smaller particles.

The coarser grades of chilled iron grit (G-16 or G-18) do not work too well in blasting cabinets. Mixed G-25 and G-40 grits, or anything finer, are quite satisfactory.

Alumina (Al_2O_3) grit works very well in blasting cabinets. The specific gravity of alumina is just over half that of chilled iron, so that this type of abrasive is readily picked up by the suction feed and is effectively accelerated through the blasting nozzle. The rate of breakdown is not excessive, except when very high air pressures are used to blast hardened steel or other very hard substrate materials.

Alumina, fused and pure, is extremely hard (>9 on the Mohs scale) and when properly crushed has sharp cutting edges. There are two grades: C (coarse) and F (fine). A good all-purpose grit for general use in a blasting cabinet would be to start with a 50/50 mixture of C and F, with periodic small additions of the C grade to maintain a balanced grit range of the mix. If the -50 mesh fraction is occasionally removed by screening, it can be set aside and used where a fine, light grit blast is needed. Alumina grains of 240 mesh and 150 mesh size are shown in Fig. 6.

Alumina grit should also be used on very hard surfaces (>45 HRC) and on nonferrous materials where embedded steel grit could result in corrosion problems.

Silica sand is widely used for outside work, and a good grade of clean, properly sized sand will do a first-class job. Sand offers the advantages of low cost and wide availability. The principal disadvantage is the hazard of silicosis, so blast operators and others working in the immediate vicinity need proper respiratory protection. Some U.S. states restrict the use of sand, and in England a special permit is required for each individual job.

Some types of sand (e.g., Ottawa sand) have smooth, rounded grains and are not recommended or suitable for thermal spray substrate preparation.

Crushed flint for blasting is available in all mesh sizes. Some users feel that crushed flint is superior to ordinary silica sand. It is very clean, and the particles are extremely angular and sharp. Flint is, of course, a form of silica, and proper precautions against silicosis must be taken.

Crushed garnet is available in several areas. Probably the finest grade for blasting comes from Idaho in the United States. The composition of garnet varies widely, however, and some

grades may contain free silica and thus mandate respiratory protection. As might be expected, the breakdown rate also varies with garnets from different sources. Some grades are almost dust-free when blasting, while others produce nearly as much dust as silica sand. Crushed garnet does an excellent job.



(a)



(b)

Fig. 6 Aluminum oxide (Al_2O_3) particles. 130x. (a) 240 mesh. (b) 150 mesh

Silicon carbide (SiC) is also available in many mesh sizes, but for coating applications where high temperatures are encountered it is not recommended because of its reactivity with various substrates and coating materials.

Crushed slag is a black, glassy grit made from the slag produced by certain types of furnaces. Ordinarily it contains no free silica, but this point should be checked with the supplier. Crushed slag is usually very angular and well graded with respect to mesh size.

Machining and Macroroughening

Macroroughening is usually accomplished by machining grooves or threads into the surface to be sprayed (Fig. 7). Typically, rough machined surfaces are also grit blasted prior to spraying. Surfaces roughened to this magnitude are often used for thick coatings to restrict shrinkage stresses and to disrupt the lamellar pattern of particle deposition in order to break up the shear stresses parallel to the substrate surface (Fig. 8).

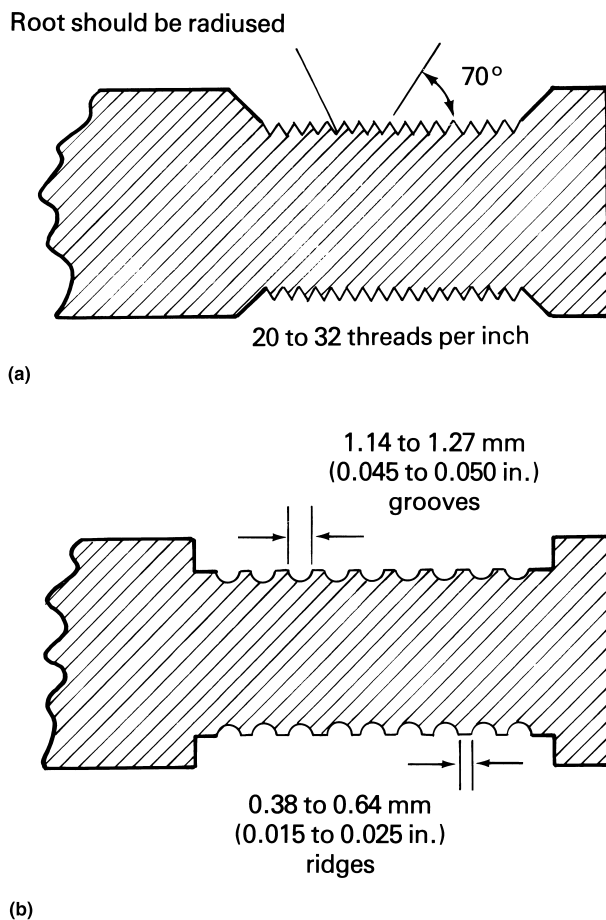


Fig. 7 Examples of surface preparation by threading or grooving. Adapted from Ref 2

Groove threads should be considered for coatings used on machine-element repairs for thicknesses greater than 1.3 mm (0.050 in.) on the radius, for high-shrinkage coatings, and for coatings that may be subject to high shear stresses (i.e., loads parallel to the interface).

When to Start Spraying

It is always good practice to begin thermal spraying of a component as soon as possible after completion of surface preparation. Freshly exposed clean metal is metallurgically active and readily susceptible to contamination. Fingerprints or contact with foreign materials may severely affect coating adhesion. If a freshly prepared surface must be stored before spraying, it should be kept covered and stored in a clean, dry area with the temperature maintained at about 40 °C (100 °F).

Application of a Bond Coat

Using a bond coat is another method of obtaining a roughened, textured surface. Specially designed bond coatings have several attributes that make them ideally suited for surface preparation:

- They often are *self-bonding*; that is, they adhere to smooth surfaces by reacting with the substrate to form microscopic alloy layers that do not depend on mechanical interlocking normally achieved from surface roughening. Coatings that are not self-bonding require at least a finely grit-blasted surface or in rare cases a high preheat temperature in order to adhere. See the

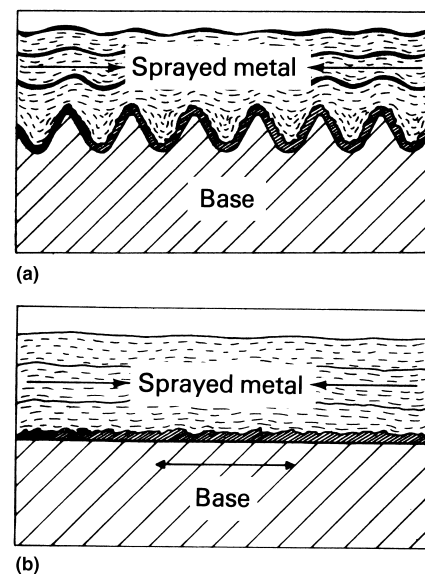


Fig. 8 Comparison of thermal spray coatings deposited on macroroughened and smooth surfaces. (a) Sprayed metal over grooves; shrinkage constrained by grooves. (b) Sprayed metal on smooth surface; effect of shear stress on bond due to shrinkage. Adapted from Ref 3

next subsection for a list of materials to which bond coats are self-bonding.

- They can accept machining or grinding to feather an edge without lifting.
- They have a sprayed surface roughness of 400 to 600 AA, which is ideally suited (the surface area is greatly increased) for reception of sprayed topcoats. The need for the proper as-sprayed roughness eliminates many fine coatings from consideration as bond coats, although they may be self-bonding.

Materials to Which Bond Coats Are Self-Bonding:

- All common steels
- Stainless steels, 300 and 400 series
- Hardened alloy steels
- Monel
- Nickel and nickel-base alloys
- Cobalt and cobalt-base alloys
- Chromium-nickel alloys
- Cast iron
- Cast steel
- Titanium and titanium alloys

Materials to Which Bond Coats Do Not Reliably Self-Bond:

- Aluminum and aluminum alloys
- Copper and some high-copper alloys
- Molybdenum
- Tantalum
- Niobium (also known as columbium)
- Tungsten
- Polymers
- Paper
- Wood
- Glass
- Ceramics

Bonding Mechanism. The mechanism by which thermal spray coatings bond to metallic substrates has not been extensively studied. Nevertheless, of the many mechanisms proposed, only fusion-diffusion has been demonstrated as repeatedly operating for self-bonding coatings. Hot particles first striking a substrate will locally raise the temperature of the metal immediately beneath the impact zone of the particle to a point where the particle and substrate interact. If the heat available is high enough to melt the substrate, then fusion with the particle will occur to form microscopic alloy layers. If no melting occurs, interaction may be characterized by high-temperature solid-state diffusion, again resulting in an alloy bond layer.

The degree to which fusion-diffusion takes place depends partly on the coating and substrate metallurgy and partly on the preheat temperature. Heating the substrate to a high temperature prior to spraying increases the probability of microscopic fusion and/or diffusion occurring. Conversely, little to no preheating reduces particle-to-substrate interaction. The use of high preheat temperatures to assist bonding must be weighed against the formation of

oxides, which are generally detrimental to coating adhesion. See the section "Preheating" in this article.

Why Use a Bond Coat? The following guidelines should be applied to determine when to use bond coats:

- A minimum of surface preparation is needed. Machining or grinding after cleaning is all that is necessary. This is especially useful on thin substrates where blasting may cause warpage or distortion.
- For added insurance against bonding failures, use a bond coat over a grit-blasted surface. Metallurgical bonding, combined with mechanical keying, provides the best of both worlds.
- Use as a convenience when handling large parts.
- Use for hard substrates that cannot be effectively roughened by grit blasting.
- Use for protection of the substrate.
- Use to help accommodate differences in coefficient of thermal expansion (CTE) between ceramic topcoats and metallic substrates.
- When the topcoat has a thickness limitation, bond coatings may be applied to build up extra thickness.

Bond coat thicknesses for all except the last situation in the preceding list should be between 75 and 125 μm (0.003 and 0.005 in.). There is no need to use more than this to achieve the roughness needed. Many bond coat materials can be applied thicker and are used as one-coating solutions for dimensional restoration.

Bond Coats Available. Bond coats can be applied by wire and powder combustion, plasma, high-velocity oxyfuel (HVOF), and twin-wire arc spray. Most materials are available in the form of powders or wires. Some typical materials, their chemistry, and service temperatures are listed in Table 2.

Undercutting (During Repair and Dimensional Restoration)

When parts come in for repair, the surface is usually damaged due to wear and/or corrosion. Normal repair procedures call for removal of the damaged surface material by undercutting into the substrate. Undercutting removes some good material as well as the damaged surface in order to provide a uniform flat surface to accept the new coating. It is important to remember that undercut-

Table 2 Common bond coat materials

Coating	Wire	Powder	Service temperature	
			$^{\circ}\text{C}$	$^{\circ}\text{F}$
Molybdenum	Yes	Yes	315	600
80Ni-20Al	...	Yes	845	1550
90Ni-5Mo-5Al	...	Yes
95Ni-5Al	Yes	Yes	845	1550
95NiCr-5Al	Yes	Yes	980	1800
95FeNi-5Al	Yes	Yes	1200	1650
90FeNi-5Mo-5Al	...	Yes	650	1200

ting reduces the cross-sectional area and correspondingly reduces the tensile and ultimate strength. Sprayed coatings will not restore the original tensile strength, even though the cross-sectional area will have been restored. The undercut surface area can be prepared by grit blasting, macroroughening, or bond coating, depending on the depth of undercut and the coating material chosen for spraying.

Masking

Various masking techniques have been developed to protect component areas next to the target zone from impact by overspray particles. Methods include:

- Metal “shadow” masks
- Tough, high-temperature tapes, including silicone with two sides sticky, resist blasting and plasma spray, 3M -33 black vinyl for blasting only, 3M stencil tape for blasting and spray resistance, and glass-filled tape for both blast and spray resistance
- Paint-on masking compounds

Metal shadow masks are placed approximately two to three times the total coating thickness away from the part to be sprayed and in front of the spray torch, thus shielding selected areas from the spray deposit (Fig. 9). The target spray area may be stationary or rotating. The mask is stationary and securely affixed. As the spray stream is traversed across the target area and onto the mask, the unwanted spray collects on the mask and is prevented from reaching the substrate. The coating will end in a narrow feathered band rather than as a sharply defined line (see Fig. 9b). In contrast to the feathered edge, note that a contact mask (see Fig. 9a), similar to the tape that is discussed in this section, leaves a sharp edge, which may act as a stress raiser that leads to debonding of the coating. Metal masks can be used over and over again, subject only to the amount of spray buildup and/or distortion due to heat from the spray process.

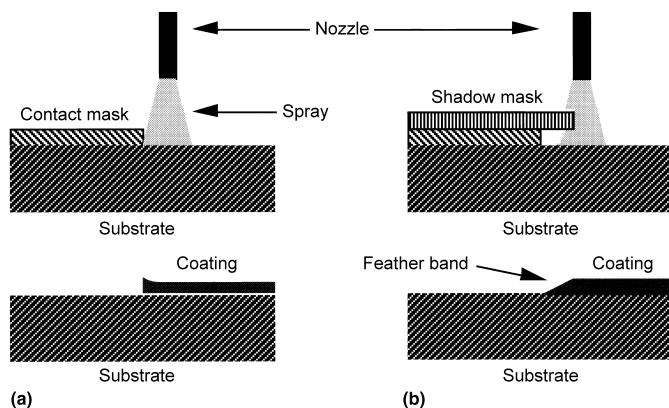


Fig. 9 Coatings resulting from contact masks (a) versus shadow masks (b)

Sheet metal masks are preferred for HVOF spraying, because tapes do not stand up to high-velocity particle impact.

Tape masks are applied by hand wrapping areas that do not require a coating. After spraying, the tape is easily removed, along with accumulated overspray, leaving the protected areas clean. Each part needs to have tape applied. Many different tapes are available commercially, some for resistance to grit blasting and others for thermal spraying. The best tapes, of course, have sufficient toughness to resist both grit blasting as well as heat resistance to withstand the hot gas and particle impact during spraying. The coating line when the tape is removed after the application of thin coatings leaves a neat and clean edge, but if the coating is thick, chipping is likely to result.

Masking Special Configurations. Further masking techniques are shown in Fig. 10. Radiused corners are protected using curved masks to keep unmelted particles and other debris from contaminating the coating. Dowel holes can be masked using simple plugs whose top surfaces are above the surface being coated as well as extending over the edge, so tapered, low-stressed edges will result.

Masking compounds are useful for providing short-term protection from overspray, but are not really useful for protection from grit blasting. Commercially available compounds comprise refractory particles, such as graphite, carried in water- or solvent-soluble binders. These can be applied by brushing or spraying. When dry, the film prevents overspray from sticking, while the refractory particles provide short-term heat protection. Excessive exposure to the spray stream, however, will cause these compounds to break down, after which spray particles may stick. Residual compound is easily removed by rinsing with water or solvents depending on the carrier solubility. Masking compounds are easy and convenient to use in spite of their shortcomings.

Preheating

Preheating of substrates prior to thermal spraying is a normal and acceptable practice. To avoid problems, however, several precautions and limitations must be observed. Preheating drives moisture from the target area and presents a warm, dry surface to the first particles impacting. Preheating also helps by pre-expanding the substrate so that it subsequently shrinks with the coating, reducing residual stresses created in the coating during cooling.

Preheating Temperatures. As a general rule, preheating to temperatures of 100 to 150 °C (210 to 300 °F) for approximately 60 s is sufficient to remove moisture. Hot molten or semimolten particles quench less rapidly and flow and adhere better when impacting a warm, dry surface than a cold one. As soon as the pre-heat temperature is reached, spraying must begin. Never preheat to 150 °C (300 °F) and hold the substrate at temperature for any significant length of time. Always spray immediately.

Equipment. A gas torch gently played over the substrate surface is commonly used for preheating. Some applicators may use the spray torch itself, fired but without spraying material, as the heat source. When little to no preheating is recommended, parts should be stored at room temperature in a dry box or some other area (e.g., a desiccator) with moisture content below ~30%.

Materials That Should Not Be Preheated. Although preheating is sound practice, with some materials the damage created by the preheating temperature will more than offset the good intended. These materials include aluminum and aluminum alloys, copper and copper alloys, titanium, and manganese alloys. Freshly prepared nascent surfaces act as oxygen “getters” and rapidly grow oxide films that then serve to inhibit metal-to-metal interaction. Preheating accelerates oxide-film growth, further interfering with coating adhesion. No preheat should be used when coating these materials; instead, start with clean, dry, room-temperature parts.

Stainless steels, superalloys, and nickel-base alloys are best preheated to around 100 °C (210 °F), quickly followed by appli-

cation of the first pass of sprayed material. This precaution is recommended because these alloys also form refractory oxide scales which, if allowed to grow into thick films, will interfere with coating adhesion mechanisms.

Inert Atmospheres. Spraying done under inert cover, either by inert gas shrouding or in low-pressure chambers (e.g., vacuum plasma spraying or low-pressure plasma spraying, VPS/LPPS), can take advantage of very high preheating because the deleterious effects of oxygen are minimized. For example, gas-turbine blades that are to be plasma sprayed with NiCoCrAlY coatings in low-pressure inert gas chambers are first preheated to 815 °C (1500 °F).

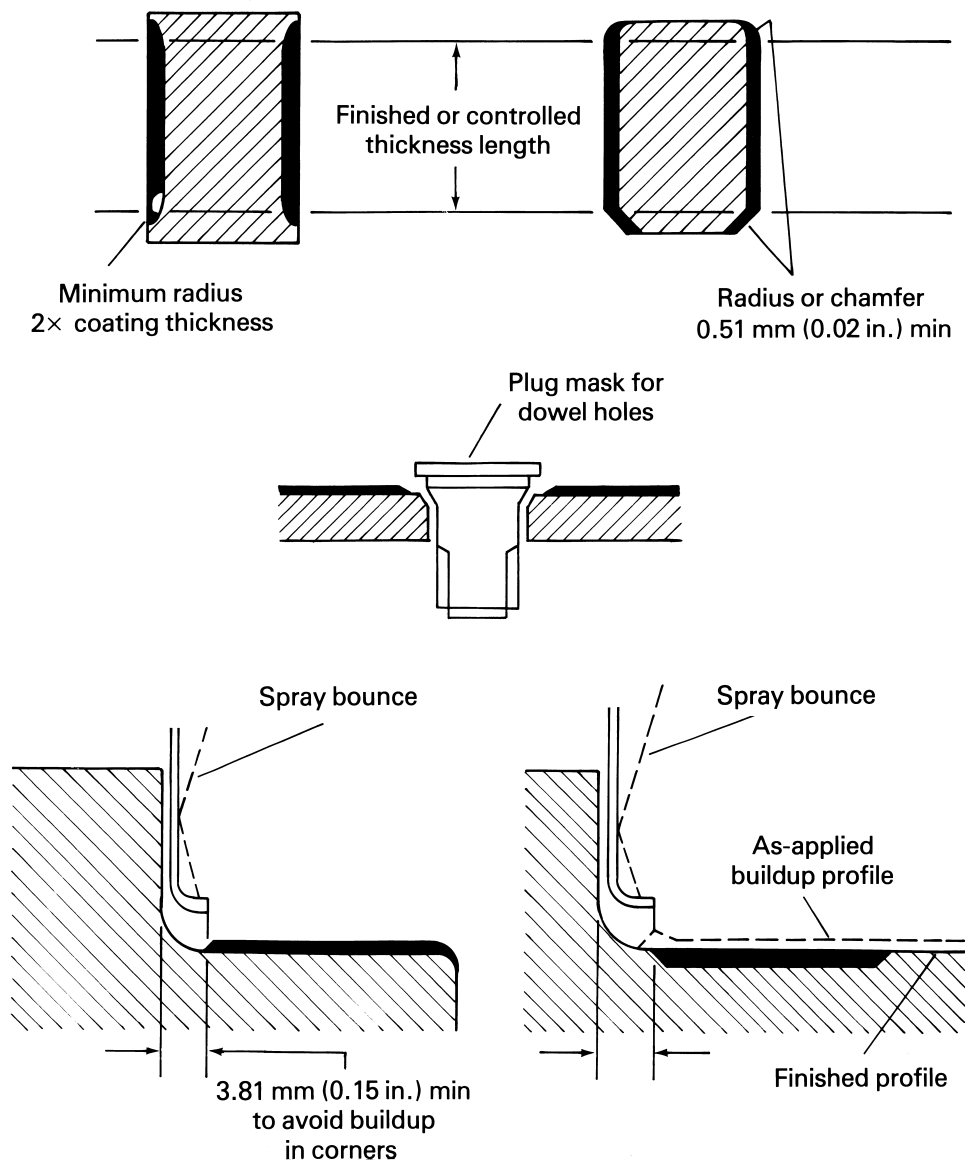


Fig. 10 Examples of how to use masking techniques for special configurations

REFERENCES

1. H.S. Ingham, and A.P. Shepard, Ed., *Metallizing Handbook*, 7th ed., Metallizing Engineering Co., Inc. (Metco), 1959, p A-136, 137
2. H.S. Ingham, and A.P. Shepard, Ed., *Metallizing Handbook*, 7th ed., Metallizing Engineering Co., Inc. (Metco), 1959, p A-28
3. H.S. Ingham, and A.P. Shepard, Ed., *Metallizing Handbook*, 7th ed., Metallizing Engineering Co., Inc. (Metco), 1959, p A-10

SELECTED REFERENCES

- J. Hancock, Ultrasonic Cleaning, *Surface Engineering*, Vol 5, *ASM Handbook*, ASM International, 1994, p 44–47
- T. Kostilnik, Mechanical Cleaning Systems, *Surface Engineering*, Vol 5, *ASM Handbook*, ASM International, 1994, p 55–66
- V.L. Rupp and K. Surprenant, Solvent Cold Cleaning and Vapor Degreasing, *Surface Engineering*, Vol 5, *ASM Handbook*, ASM International, 1994, p 21–32
- *Thermal Spraying: Practice, Theory and Application*, American Welding Society, 1985

Coating Operations

Frank N. Longo, Longo Associates

THE COATING OPERATIONS and process parameters/variables described in this article include:

- Spray pattern
- Coating buildup
- Process variations
- Substrate/coating temperature control
- Torch and substrate motion
- Deposition efficiency

Statistical design techniques used to optimize thermal coatings through a better understanding of the operating variables also are covered, along with a brief discussion of “information management”—the documentation of all process and design data.

Spray Pattern

Factors Influencing Spray Pattern Formation. The spray pattern, how it develops, and its composition, configuration, and variations are fundamental to understanding thermal spray coatings. Regardless of the feedstock material or equipment used, all spray processes involve molten or semimolten droplets or parti-

cles traveling at some velocity in a gas stream and impacting onto a substrate to form a coating. Particle trajectory, size, temperature, and the degree of melting at any moment in time in the gas stream constitute the spray pattern.

To understand what is happening in a hot jet stream refer to Fig. 1, which shows particle trajectories in a plasma jet. While the figure shows particles entering the gas stream radially, they could just as well be introduced along the centerline of the jet or generated by atomization, as in combustion-wire or twin-wire arc spray. Some particles travel along the jet centerline, others take an intermediate position, and still others ride along the periphery of the jet. Fine particles riding the periphery may, in fact, never enter the main jet, and coarse particles may flow through the jet and take an outside position on the opposite side to their injection point. On impact at the substrate, particles from all positions in the jet stream deposit virtually simultaneously, making up what is called the spray pattern.

Figure 2 depicts the spray pattern in cross section. In a planar view, parallel to the substrate, the spray pattern is circular or oval in shape. The fastest and densest deposits will build up at the center of the jet, where most particles are entrained and where the highest degree of melting occurs. Moving radially out from the

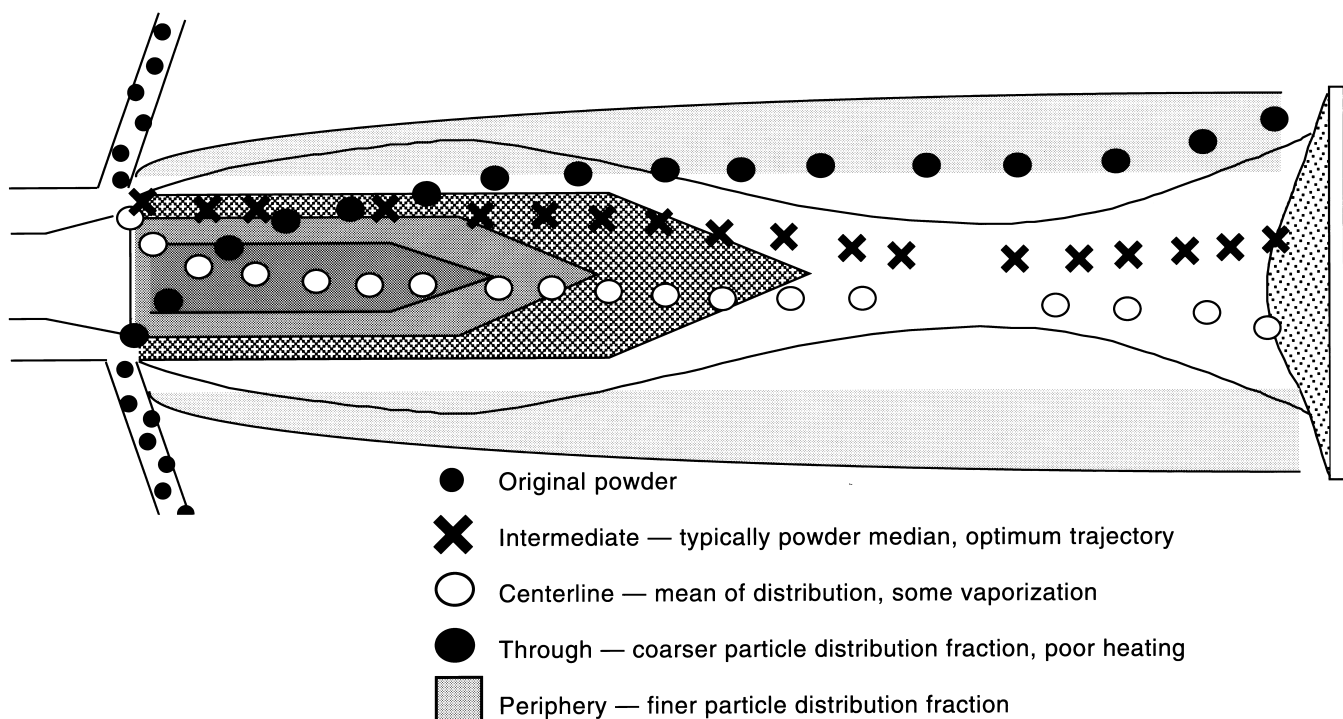


Fig. 1 Particle trajectories in a typical plasma spray stream

center (where fewer particles are entrained and where they tend to be coarser and perhaps semimolten), combined with impact at angles of less than 90° , results in increased porosity. At the jet periphery, fine particles oxidize readily due to entrained air from the surrounding atmosphere and deposit as debris. Small particles oxidize rapidly, sometimes completely, to form the major source of oxide inclusions in sprayed coatings.

The spray-pattern phenomena described in this section and shown in Fig. 1 and 2 apply to all thermal spray processes. Input

variables common to each process—such as gas type, density and velocity, heat energy, particle size distribution, specific gravity, and powder-carrier gas flows—may, however, combine to shift the distribution of porosity, unmelted particles, and oxides quantitatively. The overall spray pattern size and geometry also vary between processes.

Coating Buildup

Typical Coating Microstructure. As particles within a typical spray pattern traverse across the substrate being coated, both good and dense coating areas as well as the debris typically found in the periphery of the spray pattern are deposited during each pass. After many passes, a coating such as that shown in Fig. 3 results. This contains each of the particle types and other phenomena normally found in the spray pattern: porosity, unmelted particles, oxides, debris, and fine peripheral particles distributed from the substrate interface throughout the coating breadth and thickness.

Effect of Part Geometry on Coating Buildup. Debris and peripheral oxides are reduced when spraying takes place on small target areas. This effect is illustrated in Fig. 4(a). The shaft diameter is smaller than the spray pattern diameter; consequently, one-half of the peripheral debris actually misses the substrate and is never deposited. The larger target area shown in Fig. 4(b), however, captures the entire spray pattern on the substrate when traversed across and/or up and down.

Air-Cooling Devices for Minimizing Debris. To spray large target areas, an ancillary air-cooling device can be attached to the

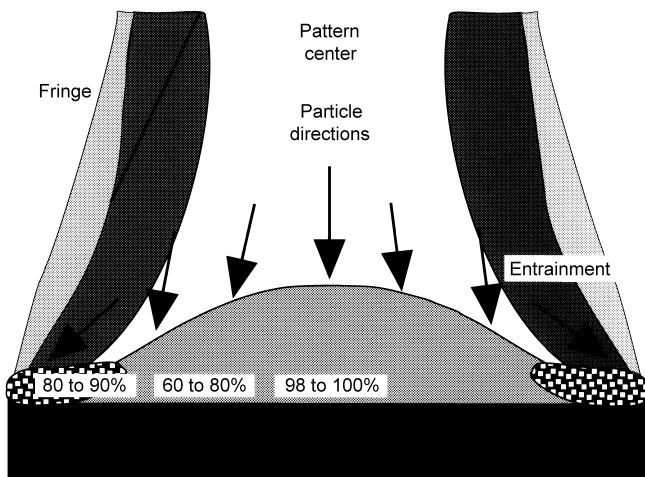


Fig. 2 The spray pattern, illustrating particle deposition and the effect of size and debris on thickness and porosity in cross section

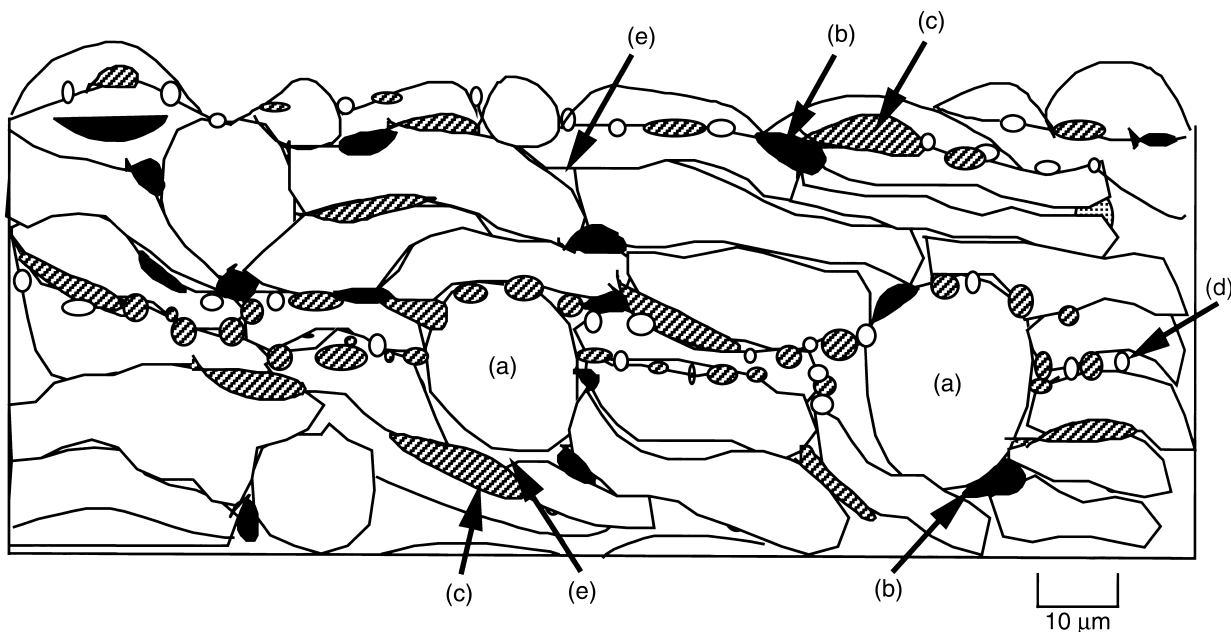


Fig. 3 Typical coating microstructure, containing unmelted particles, oxides, porosity, and debris. (a) Unmelted particles. (b) Oxides. (c) Debris. (d) Fine particles. (e) Porosity

spray torch, such that a stream of compressed air blows peripheral debris from the substrate surface in advance of the traversing spray stream. Figure 5 shows an air device positioned for spraying a large shaft. Debris is blown off the surface before it sticks and passes under “good” spray material to become permanently incorporated into the coating. When spraying flat surfaces, as shown in Fig. 4(b), air coolers should be positioned on both sides of the spray stream to help remove debris. Air coolers are usually

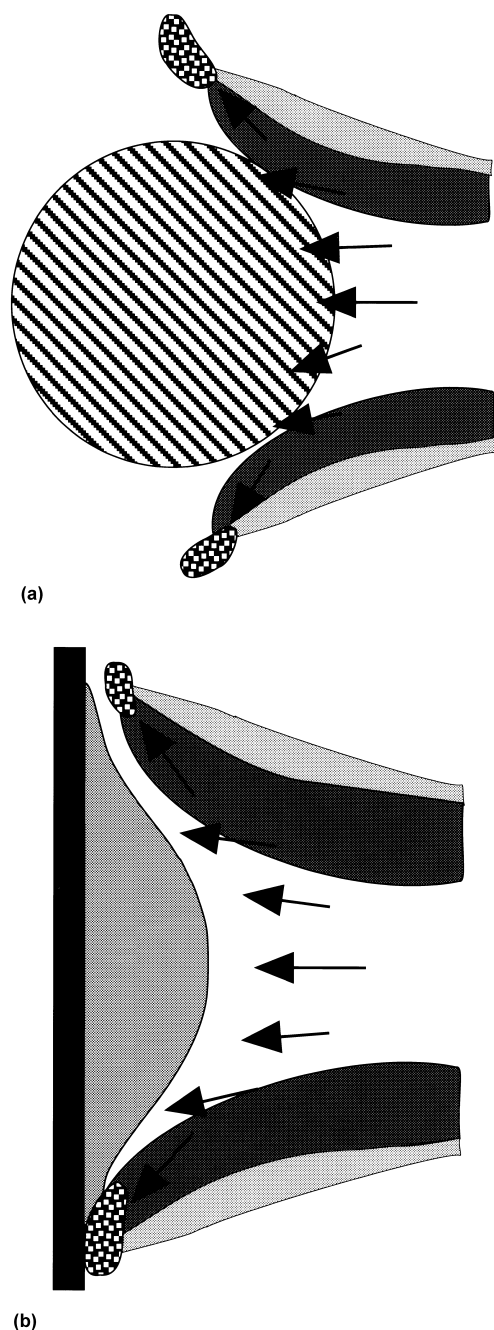


Fig. 4 How part geometry affects coating deposition. A small target (a) has peripheral debris missing the substrate, while a larger target (b) collects all the peripheral debris and oxides.

adjusted to precede and follow the spray torch along the traverse direction. While air is used to blow loose debris off the surface, it also helps to cool the surface, as the name implies. The need for cooling is covered in the section “Substrate/Coating Temperature Control” in this article.

Relationship Between Particle Melting and Coating Structure. In addition to the effects of peripheral particles stemming from the spray pattern, consideration must be given to center-stream particles and how variations in the degree of melting affect coating structure. Particles in the center of the spray pattern may include one or all of the following states on impact:

- Fully molten, or at just above the material melting temperature
- Superheated, well above the melting temperature and perhaps close to the vaporization point
- Semimolten, with the outside liquid but the core still solid
- Molten then resolidified while in-flight before impact

Fully melted particles at or just above the material melting point arrive at the substrate, impact, flow, and flatten (Fig. 6). Particle material spreads and cools rapidly as heat is conducted into the substrate. The classic lamellar splat morphology is thus achieved (Fig. 6b). Lamellar particle thickness depends on particle velocity, substrate surface tension, starting particle size, and substrate temperature. Superheated particles may splatter on impact, sending out radial splashes of fine droplets—generally spherical—that end up in the coating as “satellites” or debris (Fig. 6c). Debris formed from splatter differs from debris produced in the jet in that splatter lodges in the coating, building up at the first ridge that stops its radial travel. Air blasting does not remove splatter debris. Superheating is a condition that generally should be avoided. Particles that resolidify in flight after having been melted may not deposit; if they do, they may appear as unmelted particles but with clearly discernible oxide layers on the particle surfaces (Fig. 6a).

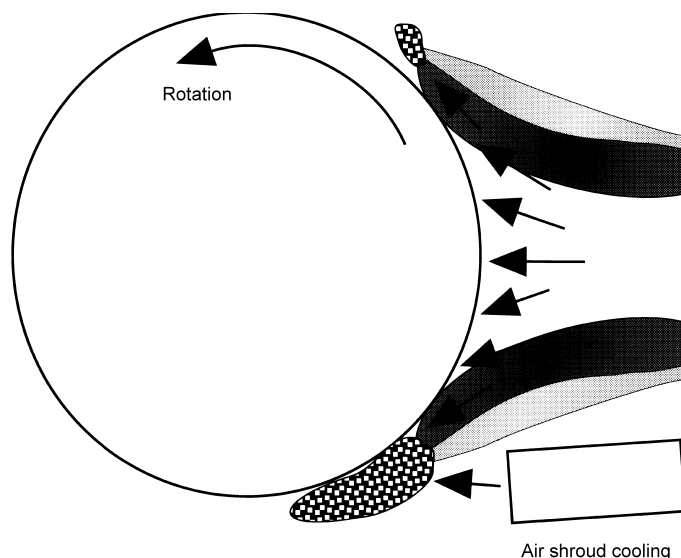


Fig. 5 Positioning of an air-cooling device to blow debris off the surface before it rotates into the center spray region and is incorporated into the coating

Sprayed coatings must be examined metallographically in cross section to see these effects. It is highly advisable to know the original particle morphology of powders before spraying in order to more accurately assess what is or is not an unmelted particle. Particle microstructures are usually quite unique and reflect their thermal processing histories. Additional information on spray processing and microstructures can be found in Ref 1 and the articles “Coating Structures, Properties, and Materials” and “Metallography and Image Analysis” in this Handbook.

Process Variations

The controllable parameters for each of the major thermal process types are different, but all share some common fundamentals. Equipment and theory are covered extensively in the article “Thermal Spray Processes” in this Handbook. It is important that the reader is familiar with various equipment theory and parameter variations in order to understand the cause-and-effect relationship between input parameters and sprayed coatings, as discussed in this section.

Converting feedstock material (whether powders, wires or rods) into a coating requires:

- Heat energy for melting
- A gas flow for atomization and/or particle acceleration to propel the particles toward the substrate

When the input heat energy, minus cooling gases (such as powder carrier gases or atomizing gases), is sufficient to melt the feedstock material and the gas flow is sufficient to propel the molten and semimolten particles toward the substrate, then a proper balance is achieved to create a thermally sprayed coating. Input heat energy controls directly affect these output variables:

- Flame/plasma/jet temperature
- Flame/plasma/jet velocity
- Particle temperature
- Particle speed
- Particle trajectory
- Deposit temperature

Each variable affects spray-pattern formation and particle heating differently. Table 1 shows a simple overview comparing heat energy and particle velocity for each process. In general, powder, combustion-wire, and twin-wire arc sprayed coatings are generated from high heat input plus low particle velocity. These coatings tend to exhibit high oxide contents, a large number of splat-

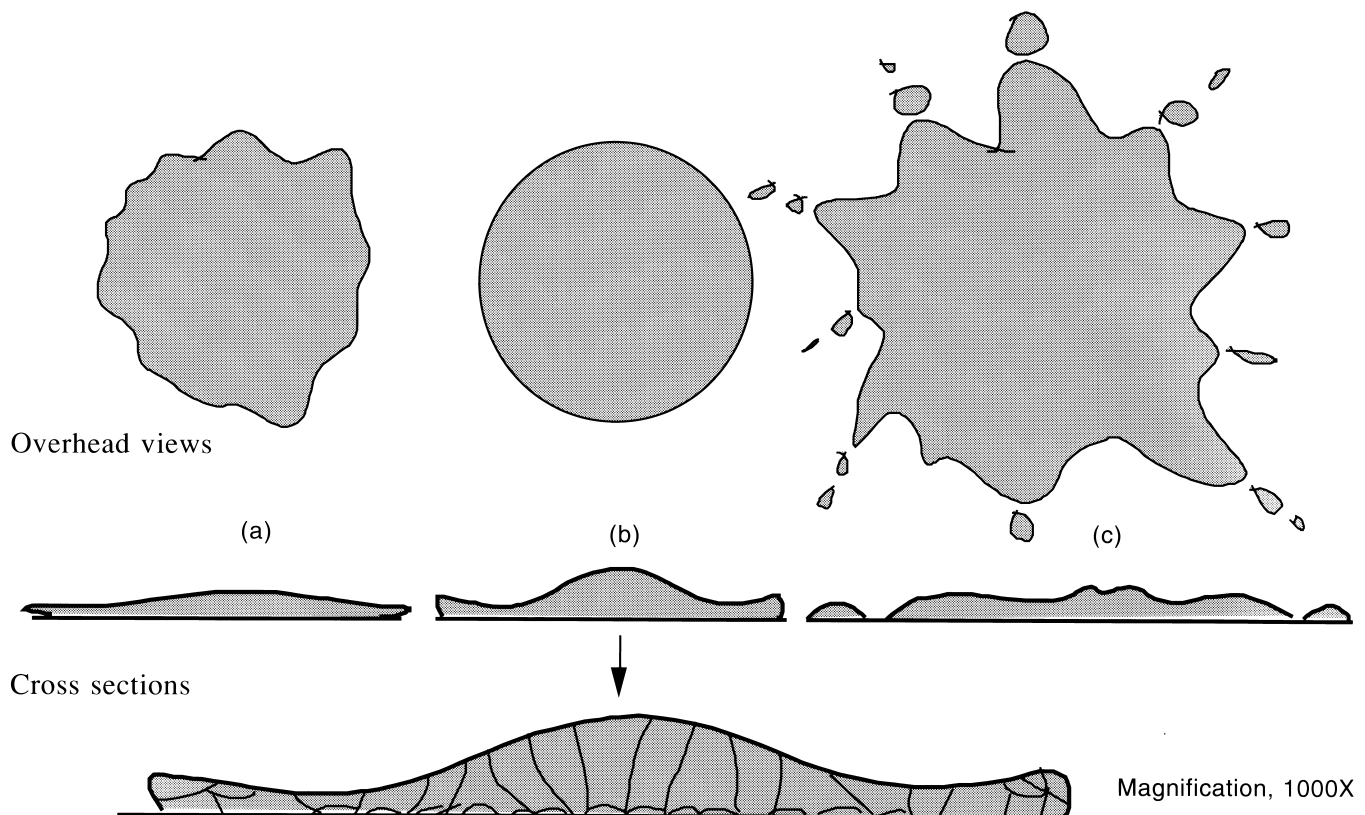


Fig. 6 The effect of the degree of particle melting at or just before impact on shape and coating structure. (a) Particle is heat softened or beginning to resolidify. At impact, it does not flow out and begins to lift at the edges. (b) Properly melted particle impacts and flows to form a well bonded classical lamellar splat shape. (c) Superheated particle impacts and splashes, creating debris, satellites, and dusting.

tered and rounded particles, and relatively high porosity. Plasma spraying tends to superheat many particles, and the splashing effect is always evident. As a result of the higher particle velocity, particles present in plasma sprayed coatings are flattened more than in combustion sprayed coatings. The resulting coatings are also denser, with finer porosity. Very-high-velocity systems (such as detonation gun and high-velocity oxyfuel, or HVOF), combined with lower heat inputs, will typically produce very dense and clean (no splashes) coatings not achievable by the other processes. In addition to the input spray parameters that control the spray pattern, particle heating and velocity, substrate temperature control, and torch-to-substrate relative motion are critical for producing desired coatings.

Substrate/Coating Temperature Control

When substrate preheating has been completed and spraying has begun, it is necessary to control the temperature of both the sprayed coating and the substrate to avoid substrate degradation, oxidation, and shrinkage/expansion.

Substrate Degradation. For those substrate materials susceptible to physical changes at high temperatures, irreparable damage can be inflicted when temperature is not controlled. Physical properties (e.g., hardness) established by heat treating are most likely to change.

Oxidation. Coating layers deposited and allowed to reach high temperatures during spraying will oxidize and appear to darken. Surface oxides so formed leave a plane of weakness in the coating and could lead to delamination under applied stresses. The critical oxidizing temperature for each material varies; nevertheless, keeping surface temperatures below 150 to 205 °C (300 to 400 °F) is a good general rule when spraying under air conditions.

Shrinkage/Expansion. Sprayed coatings shrink as they cool, exerting considerable shear stress on the substrate and leading to warpage and possible separation (Fig. 7). Substrate materials having a high coefficient of thermal expansion that are allowed to expand because of high temperatures during the spray process will experience major debonding at the substrate/coating interface.

Solving Substrate/Coating Overheating. The normal technique for solving substrate/coating overheating is to use air coolers. When properly positioned, with cool air directed so as not to disturb the centerline-sprayed material, substrate and deposit temperature are easily controlled (Fig. 5). Cool air directed at the substrate during spraying not only removes some surface debris but can also be used to control temperature. Liquid carbon dioxide or even liquid nitrogen can be used when extreme cooling is required.

When spraying under inert gas environments (either shrouded or in a chamber), inert gas, rather than air, should be used to remove debris and control temperature.

Torch and Substrate Motion

Thermal spraying is a line-of-sight process. Therefore, to deposit optimal coatings, the substrate target area should be normal (at 90°) to the spray stream. The fundamental objective of torch and substrate motion during spraying is to present the target area to the spray stream in a steady, consistent, and repeatable manner, always maintaining the same torch-to-substrate spray angle, standoff (spray distance), and relative motion. This could be as simple as a part rotating and the torch remaining stationary, or perhaps moving horizontally at a steady speed, or may involve more complex arrangements whereby relative motion must be adjusted to spray asymmetrical turbine blades or conical surfaces, for example.

Motion Control Variables

Relative movement of the torch (spray pattern) and target area (substrate) is critical for continuous and even deposition of the spray passes into cumulative coating layers. The critical variables in motion control are described in this section.

Torch-to-Substrate Standoff Distance. Once the spray distance is established, it must be kept constant for the entire operation regardless of part or torch movement. Spray distance control ensures that essentially the same coating will be deposited across the entire target area and throughout the total thickness.

Table 1 Heat energy input and particle velocity for common thermal spray processes

Process	Input heat energy to particle		Output particle velocity(a)				
	High	Low	Highest	High	Medium high	Medium	Low
Combustion wire	X					X	
Combustion powder	X						X
Standard plasma	X				X		
High-velocity plasma	X			X			
Vacuum plasma	X			X			
Standard wire arc	X					X	
Vacuum arc	X				X		
High-velocity oxyfuel		X	X				
Detonation gun		X	X				

(a) Particle speed ranges from a high of approximately 1000 m/s (3000 ft/s) to a low of 25 m/s (80 ft/s). Further variations within each process depend on the particle size, material type, and gas velocity.

Spray Pattern Normal to Substrate. Spraying normal to the substrate is desirable, but regardless of the angle chosen, it should be maintained throughout the entire spray operation regardless of part or torch motion. Typically, $90^\circ \pm 20^\circ$ is an acceptable variation if necessary.

Feedstock consumption (spray rate) establishes the rate of coating deposition as the spray pattern passes over the substrate. If the spray rate changes during spraying, the coating thickness will be uneven and coating properties will vary. The spray rate (feedstock input) must be balanced against the input energy to achieve optimal coating properties. A change in spray rate while energy input remains constant could affect particle melting and splatting, oxide content, porosity, bond strength, and deposition efficiency.

Part and Torch (Spray Stream) Motion. Simultaneous movement of the part and torch relative to each other should be managed so that an even and consistent coating thickness is maintained after each layer is applied to the target substrate. As a general rule, $25\text{ }\mu\text{m}$ (0.001 in.) of coating thickness should be applied per pass; therefore, a $250\text{ }\mu\text{m}$ (0.010 in.) thick coating will require 10 passes. The $25\text{ }\mu\text{m}$ (0.001 in.) thick layer rule is not hard and fast; it is, however, a useful first approximation for beginners. Layer thickness chosen can vary and often does so to accommodate powder type, component size, and the spray process used. For instance, less than $12.5\text{ }\mu\text{m}$ (0.0005 in.) per pass is used for wear-resistant coatings and up to $125\text{ }\mu\text{m}$ (0.005 in.) per pass is used for porous abrasable coatings and twin-wire arc sprayed zinc.

Considerations for HVOF Processing. High-velocity oxy-fuel spraying requires some special considerations. Part and torch surface speed typically range between 45 and 122 m/min (150 and 400 ft/min), whereas plasma spraying requires between 23 and 76 m/min (75 and 250 ft/min). High-velocity oxyfuel deposition of carbides is generally restricted to $12\text{ }\mu\text{m}$ (0.0005 in.) per pass. These guidelines are necessary to prevent substrate overheating during spraying.

To determine the speed of part movement relative to torch motion, consider that each pass of the torch over the surface leaves a band of deposited coating. Consider the spray pattern

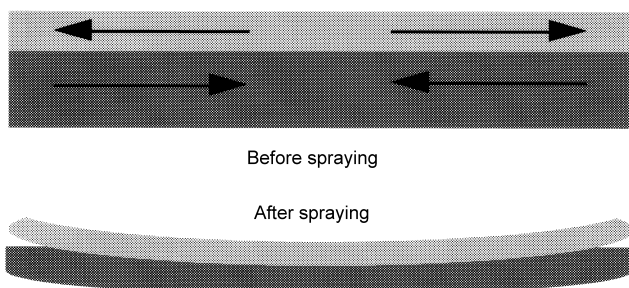


Fig. 7 Effect of coating shrinkage on interfacial shear stresses. The sprayed metal cools, creating a tensile stress in the coating and a compressive stress in the underlying substrate material. These stresses may deform the substrate or weaken the bond between the sprayed coating and the substrate, leading to debonding of the coating. The bottom illustration shows deformation of a thin-section substrate and debonding of the coating due to stresses generated by cooling of the sprayed metal. Source: Adapted from Ref 2

shown in Fig. 2 and then refer to Fig. 8. An even layer results when the spray pattern is deposited evenly across the target surface. Adjusting the relative motion of the torch and substrate so that the spray pattern is indexed approximately one-half the spray pattern diameter for each pass over the target area will overlap the spray patterns to produce a smooth, continuous layer.

One cycle across the substrate surface yields one complete layer, and multiple layers yield the desired thickness. On a flat surface, this can be accomplished in an x - y ladder or “rastered” mode by moving the torch across a stationary substrate. After the torch is translated in the x -direction across the surface, it is indexed in the y -direction for the next adjacent pass, and so on to cover the substrate area. For a rotating target (for instance, when a shaft is sprayed), the torch should be moved continuously in either an x - or y -direction along the axis such that for each substrate revolution the torch has been indexed one-half the spray pattern diameter. If the substrate rotation rate and torch traverse rates are improperly set, the coating may deposit either as a “barber pole” striping or in very thick layers after each pass.

Controlling Motion Control Variables Through Automation. Proper execution of torch and part manipulation and accurate control of these critical variables generally require automatic handling equipment and robotics, computer controlled and programmable. Automation is the only reliable method for ensuring part-to-part consistency. No matter how well trained an operator may be, the individual cannot be expected to provide the same accuracy time and time again. In the article “Process Control Equipment” in this Handbook, special attention is paid to multi-

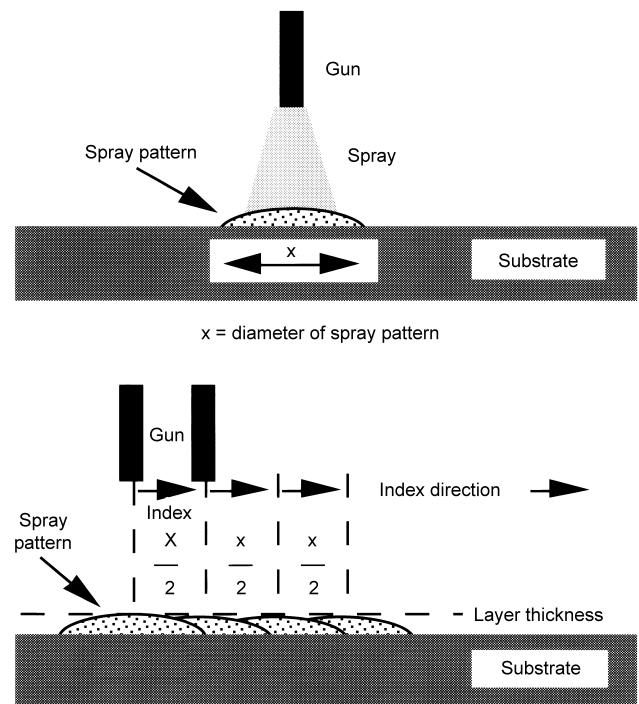


Fig. 8 How to obtain an even coating layer by indexing the torch after each pass. See text for details.

axis handling equipment, computer-controlled robotics, and integrated process control systems.

Spray rate control is also important. Powder feed rate is a key parameter that can be adjusted to affect deposition rate per pass. Today's powder feeders provide closed-loop spray rate control of process parameters, allowing continuous monitoring and real-time correction of powder flow.

Deposition Efficiency

Each of the variables discussed in the previous section—torch and substrate motion, standoff distance, spray angle, and spray rate—when combined with thermal spray process parameters and substrate geometry, directly influence deposition rate and coating quality. Deposition efficiency (DE) is an idealized measure of the percentage of particles introduced into the spray jet that actually deposit onto a flat substrate without overspray considerations. Overspray is eliminated by keeping the spray torch normal to the substrate and moving in a circular motion, never allowing the spray pattern to move off an edge.

Deposition efficiency is useful only to the extent that it provides a measure for optimizing torch spray parameters. A high DE is critical and desirable, because this ensures that the coating deposited under ideal conditions reflects the design intent of the starting powder. Low DEs suggest that some portion of the starting powder is not being deposited. This portion may consist of coarse particles, fine particles, or perhaps a blend. Low DEs are also less economical, incurring lengthy equipment operation times and high labor costs to spray thick coatings.

When substrate geometry, size, and overspray are taken into consideration, the ideal DE declines substantially, yielding the real deposition efficiency. This real efficiency is properly termed

target efficiency (TE). The TE should be used for estimating cost and powder consumption requirements.

Statistical Design Techniques

As noted earlier in this article, many variables/parameters require study before a coating can be optimized. Statistical design techniques reduce the number of experimental runs or parameter iterations needed for coating optimization. The results obtained are often unambiguous, at a minimum cost. Statistical design methods encourage the systematic study of the many variables that confront coatings engineers. Furthermore, these studies provide data on how variables interact and how they influence coating properties, as well as a plan for constructively changing input parameters to effect the most desirable properties.

Design of experiments (DOE) (Ref 3) and Taguchi factorial experiments (Ref 4) are two commonly used statistical techniques. The fundamental approach for each is the same: A matrix of selected variables versus a series of experimental runs is established. Table 2 is a Taguchi L-8 fractional factorial matrix taken from Ref 5. Nerz et al. (Ref 5) chose six variables represented by high and low values. The number of variables chosen determines the matrix size and number of runs. Typically, a fractional factorial matrix is constructed with variables deemed most likely to influence coating results, rather than a complete factorial of all variables.

The matrix shown in Table 2 includes a maximum of six variables and eight experimental runs. Selected coating properties are measured for each run. To understand the magnitude of variable influence on coating properties, an analysis of variance (ANOVA) is performed. ANOVA is a Taguchi analysis of results showing how each variable, both high and low, influences results. Another

Table 2 Taguchi matrix variables and constants

Experiment	Spray distance, mm	Spray rate, g/min	Carrier flow, scfh	Application temperature, °C	Particle size, μm	Power, kW
1	76	30	12	177	−100	40
2	76	30	15	343	−53	50
3	76	76	12	177	−53	50
4	76	76	15	343	−100	40
5	152	30	12	343	−100	50
6	152	30	15	177	−53	40
7	152	76	12	343	−53	40
8	152	76	15	177	−100	50

		Levels		
Constants	Variables	Low (1)	High (2)	
Gun	Metco 3 APG	Spray distance, mm	76	152
Nozzle	Metco 315	Spray rate, g/min	30	76
Powder port	No. 2	Carrier flow, scfh	12	15
Plasma gas	Argon	Application temperature, °C	177	343
Pressure	75 psi	Particle size, μm	−100	−53
Flows	60 scfh	Power level, kW	40	50

scfh, surface cubic feet per hour

method for evaluating factorial experiments is to use “analysis of means,” developed by Ott using the Null hypothesis (Ref 6). This method is a statistical approach employing graphics and statistical theory.

Information Management

It may seem obvious that all process and design data should be recorded. Yet many shops maintain no formal written records of what was done, by whom or where. Because thermal spray processing involves so many variables, it is extremely important that they be accurately and thoroughly documented.

Each organization is different; documentation may be handwritten or entered into a computer. Nevertheless, a system of information management should become a regular habit. For instance, a run sheet for each iteration should be integrated into all laboratory procedures to record, for example:

- Parameters, planned and actual
- Equipment used and supplier
- Powder type, lot number, and supplier

- Date, operator, and purpose of run
- Coating tests and results

Run sheets then become a permanent record, useful as a base for further work or as reference in a final report.

REFERENCES

1. D.A. Gerdeman and N.L. Hecht, *Arc Plasma Technology in Materials Science*, Springer Verlag, 1972
2. H.S. Ingham and A.P. Shepard, Ed., *Metallizing Handbook*, 7th ed., Metallizing Engineering Co., Inc. (Metco), 1959, p A-65
3. R.A. Fisher, *The Design of Experiments*, Oliver and Boyd, 1935
4. G. Taguchi and S. Konishi, *Taguchi Methods: Orthogonal Arrays and Linear Graphics*, ASI Press, 1987
5. J.E. Nerz et al., Taguchi Analysis of Thick Barrier Coatings, *Proc. NTSC* (Long Beach, CA), May 1990, p 670
6. T.E. Shahnazarian, “Statistical Methods for Quality Control,” Part 2, Technical Seminars, New Canaan, CT

Postcoating Operations

Frank N. Longo, Longo Associates

COATINGS OFTEN ARE TREATED AFTER SPRAYING to establish final dimensions or to improve or change the coating microstructure, thus avoiding some or all of the shortcomings of sprayed coatings. Expected improvements include:

- Changed microstructure
- Enhanced bond strength
- Relaxation of internal (residual) stresses
- Closure and reduction of porosity
- Improved homogeneity
- Reduced oxide content
- Hard phase precipitation
- Chemical modification

Methods used to obtain these results include:

- Chemical/physical treatments, such as organic sealing, aluminizing, and chromizing (chrome plating)
- Thermal treatments, such as fusion, heat treatment, diffusion, recrystallization, laser glazing, and hot isostatic pressing (HIP)
- Mechanical treatments, including machining, grinding, shot peening, HIP, and polishing

Finishing

The most common postspray operation is finishing by single-point turning or abrasive wheel grinding. Selection is usually dictated by the coating hardness, finish, and dimensional accuracy required.

Turning

Metal coatings are easily turned to remove as-deposited roughness. Because the structures of sprayed metals are granular rather than homogeneous, crumbly chips similar to those from cast iron will result. The surface will appear dull, with fine porosity apparent. Turning speeds, feeds, and tool bit shape can be extracted from wrought metallurgy standards for particular materials. Cutting oils, lubricants, and coolants or dry turning can all be used. Remember, however, that oils and coolants will penetrate and remain in the coating after machining. Carbide tools are recommended because sprayed coatings are very abrasive and tools wear rapidly. Recommended tool angles for turning with carbide tools are shown in Fig. 1.

Grinding

For dimensional accuracy and better surface finish, grinding is preferred. Dry or wet grinding can be used. Wet grinding is preferred to dry grinding for sprayed coatings because of greater lat-

itude in abrasive and wheel selection. Although grinding is an established industrywide process, grinding sprayed coatings involves the following special considerations:

- Sprayed coatings tend to “load” wheels rapidly; to control wheel wear, use relatively coarse grains with low binder contents.

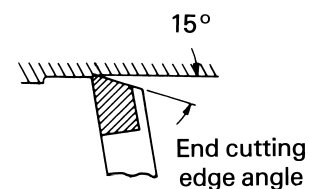
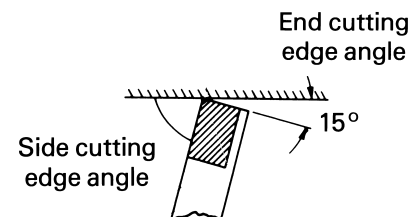
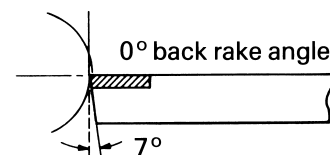
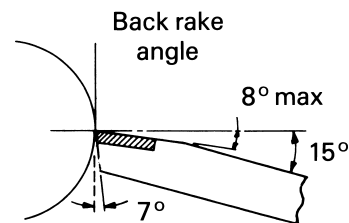
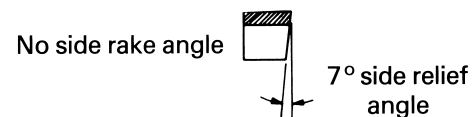


Fig. 1 Tool angles for machining cemented carbide coatings. Source: Adapted from Ref 1

- Use light cuts because excessive pressure can cause crack formation, delamination, or excessive coating fracture and pull-out.
- Do not “spark out” on the final pass; this tends to glaze the coating.
- Keep wheels freshly dressed.
- Alumina, silicon carbide, and diamond wheels can be used. Selection is based on required coating hardness and finish. Tungsten carbide coatings always require diamond wheels for grinding.

Polishing

Sprayed coatings turned or ground can be further finished by polishing to achieve very high mirror finishes down to 1 to 2 μm . Such finishes generally are possible only with very dense coatings. Polishing can be accomplished by lapping, tumbling, honing, and/or burnishing.

Densification

Coating densification is a commonly practiced postspray treatment. The objective is to reduce interconnected porosity and obtain:

- Higher hardness and wear resistance
- Improved particle cohesion
- A barrier to corrosive products

Fusion

Fully dense, totally homogeneous coatings are produced by melting selected coatings at approximately 1010 °C (1850 °F). Both coating and substrate are treated by torch or furnace heating. Coatings from the family of self-fluxing alloys are first applied by combustion-powder spraying. Self-fluxing alloys contain elements that react with oxygen or oxides to form low-density oxides that float to the surface, thus improving density, bonding, and so forth. In general, these are nickel- or cobalt-base alloys that contain boron or silicon, either singly or in combination, in excess of 1.5%, as well as carbon and iron. Chromium is added to many alloys for added wear and corrosion resistance. Table 1 lists compositions of some spray-and-fuse self-fluxing alloys.

Boron and silicon serve several functions. Boron is a potent hardener for both nickel- and cobalt-base materials and combined with silicon lowers the melting point by several hundred degrees, due to the formation of eutectic alloy compositions. Boron and silicon also promote wetting by reducing oxides of nickel, cobalt, chromium, and iron and control surface tension and fluidity. During fusing, a borosilicate glass is formed that is believed to further reduce oxidation. Boron and silicon are fluxing agents, hence the term *self-fluxing*.

Fused coatings exhibit these properties:

- Nearly 100% dense, impermeable, and metallurgically bonded to the substrate

- High hardness ranging from 20 to 70 HRC (depending on boron and carbon content)
- Slag inclusions in flame spray and fused coatings. Less slag is evident when the material is first plasma sprayed and then fused.
- Good impact resistance, attributed to exceptional coating integrity and metallurgical bonding
- Good corrosion resistance and high hot hardness, permitting service temperatures up to 650 °C (1200 °F)
- Coating shrinkage of 10 to 20% on fusion as the porosity closes during melting. Plasma sprayed coatings are denser before fusion, resulting in less shrinkage.
- Very fine ground finishes, often to less than 1 μm arithmetic average (AA)

Laser glazing or induction heating can also be used to fuse coatings.

Sealing

Thermal spray coatings, being somewhat porous by nature, are often sealed with organic materials that penetrate and fill the pores. Coatings sealed and cured in this fashion have their interconnected pores filled, which effectively creates a barrier to penetration of unwanted materials. For example, sealed coatings:

- Prevent corrosive environments—liquids or gases—from penetrating the coating and attacking the coating/substrate interface
- Keep grinding debris from lodging in the coating (assuming the coating was sealed prior to grinding)
- Enhance interparticle cohesion
- Extend the life of aluminum and zinc coatings on steel to prevent corrosion
- Provide special surface properties such as nonstick and release

Table 2 lists some common organic sealants. The choice of sealer dictates the curing temperature and time. When selecting a sealant, (1) be sure that its viscosity is low, allowing it to penetrate the coating to a satisfactory depth, and (2) determine its chemical corrosion resistance and service temperature after curing. Both factors will guide the use and limitations of sealants. Sealants can

Table 1 Nominal spray-and-fuse powder alloy compositions

Alloy designation	Composition, wt%							
	Ni	Co	Cr	C	Si	B	Fe	W
Deloro Alloy 40	bal	...	7.5	0.35	3.5	1.7	1.5	...
Deloro Alloy 50	bal	...	11.0	0.45	4.0	2.4	3.0	...
Deloro Alloy 60	bal	...	15.5	0.8	4.3	3.5	4.0	...
Stellite SF6	13	bal	19	0.7	2.3	1.7	3.0	7.5
Stellite Alloy 157	2 max	bal	22	0.1	1.6	2.4	2 max	4.5

bal, balance. max, maximum

be applied by brushing, spraying, or dipping. The method used depends on sealant properties and size of the treated and coated area.

Peening

Shot peening and glass-bead peening can be used to plastically deform the surface layers of sprayed coatings, placing them in a state of compressive residual stress to improve their fatigue properties. While surface porosity may be closed off, it is unlikely that penetration of liquids and gases will be prevented.

Hot Isostatic Pressing

Simultaneous exposure of coatings to high temperatures—typically below the solidus, for steels approximately 1100 °C (2000 °F) and for nickel alloys approximately 975 °C (1800 °F)—and high static pressures of 10 MPa (1400 psi) promotes densification and enhances the physical properties of sprayed coatings. The heat encourages diffusion at the coating/substrate interface, enhancing bond strength, while the pressure compresses the coating, collapsing and shrinking pores. The combined effects also relieve stresses, increase fracture toughness, and produce near-wrought-metal properties. Hot isostatic pressing is commonly used in the processing of near-net-shape components fabricated from thermal spray deposits.

Heat Treating

Heat treating takes place at high temperatures but well below the coating melting point. In contrast to fusion, no melting occurs. In contrast to HIP treatments, heat treatment takes place at atmospheric pressure or sometimes under vacuum.

- Reasons for coating heat treatment include:
- Diffusion occurs between the substrate and sprayed coating, enhancing the bond strength. Diffusion is a mixing of atoms that takes place at high temperatures but well below the melting point. Diffusion creates metallurgical bonding and higher strength.
 - Residual stresses are relaxed and interparticle cohesion is increased, analogous to annealing, only more pronounced.

Table 2 Common sealants used with thermal spray coatings

Organic material	Characteristic
Paints	Water and solvent soluble
Waxes	Low-temperature melt
Phenolics	Air dry or heat cure
Epoxy phenolics	Air dry or heat cure
Epoxy resins	One-part catalyst
Polyesters	Air or heat cure or one-part catalyst
Silicones	Heat cure
Polyurethanes	Air dry or one-part catalyst
Linseed oil	Air dry
Polyimides	Heat cure
Coal tars	Air dry
Anaerobics	Cure in absence of air

- Hardness may be reduced, and the coatings show improved ductility and impact resistance as well as increased fracture toughness and modulus of elasticity.
- Recrystallization and grain growth occur via sintering, effacing laminar particle shapes and creating new microstructural textures, further improving mechanical properties.
 - Diffusion may also serve to reduce porosity. This is a particularly time-dependent phenomenon and perhaps too slow to be considered a reliable practice.
 - Phase and structural changes occur. Oxides are reduced when heated under hydrogen (H₂) or in vacuum environments. Similarly, carbides and nitrides can be changed by selective treatment. Carbides are typically precipitated from solid solution with cobalt to enhance toughness.

Methods of heat treating include:

- Furnace heat treating in air or under inert or hydrogen atmospheres
- Vacuum or low-pressure furnace, using inert gas cover
- Induction heating
- Laser processing (glazing)

Laser glazing is unique in that the thermal energy applied can be controlled precisely in terms of both position and intensity. The laser beam is actually a stream of coherent (in-phase), monochromatic (single-color or wavelength) photons that can be focused and directed through the air over relatively long distances without loss of coherence or appreciable loss of power. Continuous-wave (CW) and pulsed carbon dioxide (CO₂) and neodymium-doped yttrium-aluminum-garnet (Nd:YAG) lasers are the most common. One of the great advantages of lasers for surfacing operations is that an intense amount of energy can be concentrated and delivered to a very shallow zone—in other words, a thin liquid phase—on the coating surface. This feature may establish ideal conditions for a broad range of surface treatments and coatings, allowing the formation of a metallurgical bond with a minimum of dilution of the coating alloy by the substrate.

Aside from the widely practiced transformation hardening of steels in its early stages of utilization, the process is now being used as a replacement for some conventional hardfacing processes and more importantly as a means of surface glazing and surface alloying. Unlike transformation hardening, which is a solid-state phenomenon akin to flame or induction hardening, laser glazing brings the coating surface to the liquid state and then allows it to solidify very rapidly. Rapid-rate solidification causes microstructural refinements and other metallurgical changes that profoundly affect the properties of metal, including tribological characteristics and corrosion resistance.

Inspection

Once a coating has been sprayed and post-treatments, including finishing, have been completed, it is ready for inspection. Two options are available: destructive and nondestructive tests.

Destructive Testing—Indirect

In destructive testing, as the name implies, finished parts are destroyed and a sample of the coating is removed and prepared for metallographic or scanning electron microscope (SEM) analysis. This provides the most accurate assessment of coating quality possible, but is often somewhat impractical when the coated parts are large, costly, or limited in number.

When destructive testing is required and parts themselves cannot be destroyed, a sample of the coating should be sprayed along with the actual part and used for evaluation. Sample test coupons so prepared are designed to provide information on tensile bond strength, microstructure, bend strength, hardness, and so forth. Information gathered on separate sample coupons, however, will always be somewhat subjective because it is indirect; that is, the data may not represent the coating as it was applied to the actual part if spraying was done at a different time or more likely onto a different substrate shape or substrate material. If possible, the sample should be sprayed simultaneously with actual parts, using the same substrate material and surface preparation.

Nondestructive Testing (NDT/NDE)—Direct

Such testing/examination is done directly on the finished part without damaging its appearance or functional capability. It is possible to test every part and achieve 100% quality assurance. Nondestructive testing technology does not always provide quantitative information normally gathered by metallography or tensile bond testing. One must know the limitations for detecting each important characteristic, such as debond size or debonds along edges. Calibration standards for each material/geometry are required.

Further discussion on the two inspection techniques follows. Additional information on testing and analysis can be found in the articles “Testing of Coatings” and “Metallography and Image Analysis” in this Handbook.

Current Destructive Tests

Tensile Adhesion Strength. Using ASTM Standard C 633, pull-test coupons are sprayed and the coating is loaded in tension until failure occurs. Bond tensile strengths in psi, ksi, or MPa are recorded. An absolute minimum of three tests should be carried out, although five or more are better from a statistical standpoint. The bond strength of the adhesive used must also be measured as part of each test batch. After failure, the sample surfaces must be examined to study the locus of failure. This can provide insight into the nature of the failure—within the adhesive, at the coating/substrate interface (adhesive failure), or within the coating (a mixed-mode failure).

Metallography. Samples of the coating/substrate are sectioned, mounted, polished, and studied using a light optical microscope, generally at magnifications ranging from 50 to 100 \times . From analysis of a polished coating cross section, density, melting efficiency (unmelted particles), oxide content and distribution, thickness, grain size (which may require etching to reveal the grain structure), bond-line integrity, and microhardness can be determined.

Bend Testing. Coatings sprayed onto 7.87 mm (0.31 in.) thick coupons are bent over a 25.4 mm (1 in.) shaft to $\sim 90^\circ$ and exam-

ined for cracking, chipping, and peeling as indicators of bond integrity and coating ductility.

Thermal Shock Tests. On prescribed samples, ceramic thermal barrier coatings (TBCs) usually sprayed over a bond coat are tested for thermal shock resistance by alternate high-temperature exposure and cooling cycles until failure.

Current Nondestructive Tests

Thickness. When the substrate size and shape permit, simple calipers or micrometers can be used. Eddy-current sensors and ultrasonic, capacitance, and optical coating thickness measuring devices can be used on more complex geometries.

Surface Texture. Visual inspection, stylus-tracing profilometers, and capacitance and optical measurements can be used. Newer techniques include white light and laser interferometric optical systems and conformal microscopes.

Hardness. Some argue as to whether a Rockwell hardness indentation is really nondestructive. Nevertheless, if taken on a nonworking area of a coating, valuable direct data can be collected. Microhardness (Vickers, 300 gf load) coating data are also commonly measured and reported.

Adherence. Acoustic emissions testing has the potential for measuring bond integrity, including delamination, cohesive weakness, and adhesive weakness. Ultrasonic waves are displaced/attenuated because of bond imperfections, providing a good indicator of bond integrity.

Density. Various techniques including Archimedean/displacement, x-ray, eddy current, and thermal measurements can be used to measure coating density.

Microstructure. Acoustic emissions (ultrasonic wave attenuation) is a sensitive indicator of microstructural features. Some recent ultrasonic studies have provided information on stress mapping and structural anisotropy.

Defects. Acoustic emission, x-ray, eddy current, and thermal techniques are used to detect defects.

REFERENCES

1. H.S. Ingham and A.P. Shepard, Ed., *Metallizing Handbook*, 7th ed., Metallizing Engineering Co., Inc. (Metco), 1959, p A-93

SELECTED REFERENCES

- K.P. Cooper, *Laser Surface Processing, Friction, Lubrication, and Wear Technology*, Vol 18, *ASM Handbook*, ASM International, 1992, p 861–872
- K. Subramanian, *Finishing Methods Using Multipoint or Random Cutting Edges*, *Surface Engineering*, Vol 5, *ASM Handbook*, ASM International, 1994, p 90–109
- *Thermal Spraying: Practice, Theory and Application*, American Welding Society, 1985
- R.C. Tucker, *Thermal Spray Coatings*, *Surface Engineering*, Vol 5, *ASM Handbook*, ASM International, 1994, p 497–509
- V.C. Venkatesh and S. Enomoto, *Finishing Methods Using Defined Cutting Edges*, *Surface Engineering*, Vol 5, *ASM Handbook*, ASM International, 1994, p 84–89

Introduction to Materials Production for Thermal Spray Processes

Christopher C. Berndt, Stony Brook University

THE MANUFACTURE AND PROCESSING of materials commonly used in thermal spray (TS) coating processes are described in this Section of the Handbook. Powder manufacturing processes directly reflect quality and economics. A TS material is not defined solely by its chemistry and particle size distribution. Thermal spray materials with nominally identical specifications may:

- Exhibit substantially different TS characteristics (i.e., different deposition efficiencies, spray rates, etc.)
- Have large purchase cost differentials
- Produce acceptable versus unacceptable coatings

Materials from different manufacturers with the same nominal chemical composition and physical shape (whether powder, rod, wire, or composite form) may exhibit quite different material and physical properties, and different service performances, even when deposited under identical conditions. Knowing how such differences can arise enables the overall technological limits of a process to be defined.

Thermal sprayed coatings cover a range of applications: for example, to combat corrosive environments, to prevent high-temperature oxidation, to build up and reclaim worn components, to minimize wear, or to confer specific electrical insulation and conduction characteristics on a bulk material. As discussed in the article “Material Categories for Thermal Sprayed Coatings” in this Section, metals and alloys, ceramics, intermetallics, composites, and polymers are used for these various application areas. The definition of *ceramic* will be taken in its broadest context and may include classical ceramics such as alumina, high-tech materials such as zirconia alloys, and the refractory materials based on carbides.

Coating applications are a direct consequence of their ability to retain some intrinsic characteristics of the feedstock material. (The term “feedstock” is used throughout the articles in this Section to indicate any material injected into the apparatus that is used to project the material onto a substrate.) The feedstock may be in any one of a variety of physical states. For example, aluminum, zinc, and aluminum-zinc alloys are used for corrosion protection, and tungsten carbide and cobalt mixtures are used for wear resistance applications. Since the 1990s, thermally sensitive polymeric materials have been increasingly used for coatings and composites.

Coating properties depend on the feedstocks used in the TS process. However, a TS coating need not necessarily retain the intrinsic characteristics of the feedstock because the TS process quite often modifies the properties of the original material. Thus, the structure of a coating is quite dissimilar to that of a material produced by a bulk fabrication process. The utility of a coating

may be limited by its mechanical, physical, thermal, and/or electrical properties. Adhesion of the coating to the substrate is also important, because it is crucial that the coating remain attached throughout its service life.

Differences in feedstock materials may include factors such as the material production route and the particle size distribution or particle morphology. These characteristics influence not only the TS parameters but also the coating performance. Among the industries that presently take advantage of TS coatings are aerospace, automotive, biomedical, the military sector, powder production, steelmaking, offshore engineering, nuclear and power generation, papermaking, mining, textiles, printing, and engineering maintenance.

The articles “Feedstock Material Considerations” and “Material Production Processes” describe the manufacturing methods for feedstocks used in TS processes. Of primary interest is *powdered materials* since these have wide diversity and utility. Feedstocks in the form of wires or rods, although of value throughout TS, are not mentioned since their morphology is not highly variable and the production route is not particularly unique to TS technology. An important aspect highlighted in these articles is how various powder characteristics can be controlled in order to retain some specific material property. Thus, to a certain degree, the powder processor has the ability to tailor feedstocks to produce a desired coating property.

The article “Particle Characterization” emphasizes particle size determination. Testing procedures that enable determination of the feedstock particle size are essential, because such measurements influence the classifications and utility of the initial process material.

Terminology

The generic term for the starting material in a TS process is *feedstock*. The processing of raw materials into feedstock powders that may be suitable for TS involves a wide range of manufacturing and classification procedures. The technical terms that are used in the powder processing industry are listed in the “Glossary of Terms” in this Handbook. Note that there may be some slight differences between North American and European terminologies, especially with respect to TS terminology.

It is important that the reader of this Section become familiar with what may be new terms. Those who are familiar with this area are also advised to reinforce their knowledge by reading through the glossary, because specialized definitions are used in

the articles that follow. For example, the term “alloy” is used to describe combinations of metallic materials *or* combinations of ceramic materials.

Summary

The articles presented in this Section enable the reader to:

- Understand the basic processes by which powders for TS are produced
- Learn that the various production routes for powders may have attributes and deficiencies that are, in turn, related to individual powder chemistries and TS processes
- Appreciate that quality control and specifications for powders are essential
- Comprehend that the term *particle size* should always be related to the technique by which the measurement was performed
- Recognize that particle morphology and particle size distribution are important quality control indicators of the feedstock
- Realize that the *powder feeding problem* is the root cause for coating variability and poor material properties

SELECTED REFERENCES

- C.C. Berndt, Ed., *Thermal Spray: Practical Solutions for Engineering Problems*, ASM International, 1996
- C.C. Berndt, Ed., *Thermal Spray: A United Forum for Scientific and Technological Advances*, ASM International, 1997
- C.C. Berndt, Ed., *Thermal Spray: Surface Engineering via Applied Research*, ASM International, 2000
- C.C. Berndt and S. Sampath, Ed., *Thermal Spray Science and Technology*, ASM International, 1995
- C. Coddet, Ed., *Thermal Spray: Meeting the Challenges of the 21st Century*, ASM International, 1998
- E. Lugscheider and P. A. Kammer, Ed., *Tagungsband Conference Proceedings*, Deutscher Verband für Schweissen (DVS), 1999
- A.R. Nicoll, Ed., *First Plasma-Technik Symposium*, Lucerne, Switzerland, 1988
- A. Ohmori, Ed., *Thermal Spraying: Current Status and Future Trends*, High Temperature Society of Japan, 1995
- L. Pawlowski, *The Science and Engineering of Thermal Spray Coatings*, John Wiley & Sons, 1995
- *Thermal Spraying: Practice, Theory, and Application*, American Welding Society, 1985

Feedstock Material Considerations

Christopher C. Berndt, Stony Brook University

THE QUALITY OF ANY THERMAL SPRAY (TS) COATING depends on the initial feedstock material. No technical procedure can correct for a poor-quality powder.

Coatings are formed from the repeated deposition of molten and semimolten feedstock particles. The resulting lamellar dimensions are approximately 60 μm in diameter and from 1 to 4 μm thick. Metallographic characterization of coatings is often used as a basis for determining the suitability of the initial feedstock. Advanced analytical techniques involving image analysis and statistical quantification of the coating microstructure are also used to ensure quality control (see the article “Metallography and Image Analysis” in this Handbook for details).

Feedstock materials should be considered as an integral part of a coating system that includes the substrate, possibly an intermediate layer, and finally the bulk of the coating. The coating structure is highly oriented, with lamellae parallel to the substrate surface. The real contact area of lamellae is approximately 30% of the available boundary area. Unmelted particles may become incorporated into the coating, and these, along with interlamellar oxide boundaries and porosity, constitute regions of poor bonding that ultimately may lead to failure of the coating system. On the other hand, melting of a material does not always mean good adhesion; substrate properties and material/substrate interactions are critical. The overall coating manufacturing schedule consists of production of the feedstock, then production of the coating so that a material-specific structure is formed that, in turn, exhibits distinct materials properties for specialized applications.

Some feedstock materials—for instance, alumina and stabilized zirconia—are manufactured by a wide range of production processes. It is not surprising that each feedstock material must be optimized with regard to the TS parameters of particular spray devices and that the so-formed coatings exhibit variable material properties that depend on specific attributes of the feedstock. The material properties of coatings are generally independent of specific equipment if the powder is optimized with respect to each type of equipment. Material properties such as hardness, wear resistance, and tensile adhesion strength generally correlate well with coating density. It is, therefore, essential that the TS applicator appreciate that feedstock materials are not designated solely by their chemistry and particle size. A simple substitution of one powder for another (because of cost, availability, etc.) does not necessarily mean that the performance characteristics of the original coating will be maintained.

This article focuses on the *powder feeding problem*, which is the main reason for coating variability and poor material properties. Commonly used powder feeding systems are also described and compared. Additional information on manufacturing methods for TS feedstock materials and feedstock/coating relationships can

be found in the article “Material Production Processes” in this Section of the Handbook.

The Powder Feeding Problem

A fundamental requirement of any TS process is to transport the feedstock to the heat source and then further direct it toward the substrate. A phenomenon commonly known as the “powder feeding problem” is a technological aspect of TS related to the “sprayability” of the feedstock.

Powder feeding often refers only to the initial stage of powder transportation—that is, from the powder feeder/hopper to the TS device. However, technical difficulties may result in coatings of poor quality, regardless of the fact that powder seems to be flowing into the heat source. This aspect of TS technology is the root cause for examining the production and physical aspects of powder preparation.

The normal route of the powder commences within a powder feeder hopper, which may hold up to 10 kg (22 lb) of material. From there the powder flows through some orifice or rate-controlling restriction and is mixed with a carrier gas. The carrier gas acts as a fluidizing agent and transports the powder through a flexible powder delivery tube with an internal diameter of up to 6 mm (~1/4 in.). The inside diameter of this tube can have a Teflon lining, which decreases powder friction within the confines of the tube. The mass fraction of the carrier gas to the powder mass is about 25%. Often the powder feed tube is oriented to take advantage of the natural tendency for powder to flow downward; that is, the powder hopper is placed above the TS torch.

Powder delivery tubes are typically approximately 3 m (10 ft) long and end in an approximately 25 mm (1 in.) long metal termination called a powder injection port. This port is either (a) attached directly to the anode (or nozzle) so that powder flows into its throat or (b) directed into the tail flame of the heat source. In some instances (e.g., detonation guns, flame spray devices, high-velocity oxyfuel [HVOF] devices, and some plasma torch devices), the powder is entrained into the gas and injected into the bore of the TS torch so that it flows coaxially along its longitudinal axis. This method of powder injection is claimed to provide consistent, “well-mannered” particle trajectories and, thus, optimum heat transfer to the particles.

One particularly common phenomenon that is eventually experienced by every TS operator is that of powder overloading in the powder feed tube. The technical term for this behavior is *saltation*, and it is physically manifested when powder does not flow evenly but appears to stop and start at irregular intervals every few seconds. Increasing the carrier gas flow rate may smooth out this

erratic, irreproducible flow behavior, but, in general, this solution has the undesirable effect of causing the particles to have too high a momentum so that they do not pass through the optimum part of the thermal spray source.

It is thus valuable to examine the types of powder feed delivery systems used and the requirements that constitute good powder feeding. A specific powder may not have identical feeding characteristics with different powder feeding devices.

Powder Feeding Systems

Three types of powder feeder systems are most commonly used throughout the TS industry:

- Gravity-based devices
- Rotating wheel devices
- Fluidized-bed systems

Each is described here, along with a brief discussion of other types that have been used in the past, find limited usage, or are still in the experimental stage.

Gravity-Based Devices

Figure 1 illustrates a gravity-based device whereby the powder falls into the thermal spray source. Some vibratory action, normally achieved by using compressed air to drive a ball bearing

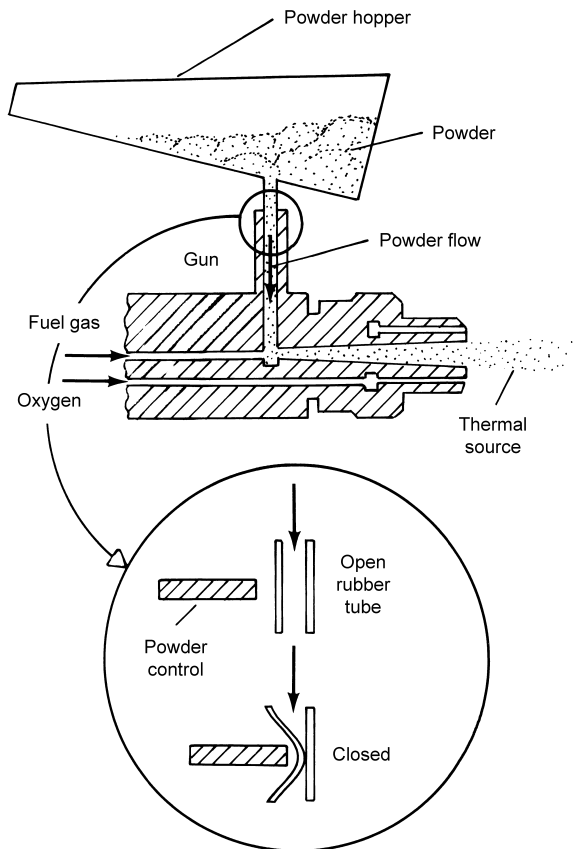


Fig. 1 Gravity- and vibration-based powder feeding system

around a bearing race, may be supplied to agitate the powder into the gas stream and prevent blockages. In the figure shown, the throttle control for the powder flow is a compressible rubber grommet that can be pinched to either allow or stop powder flowing into the carrier gas stream. This device relies on a full canister of powder so that the gravity head of material is approximately constant. There are no powder delivery tubes connecting the powder supply to the thermal spray source.

Uneven and somewhat sporadic powder flow often occurs when the canister nears an empty condition. The powder flow rate is controlled by altering the inside diameter of the rubber grommet. This type of powder feeding device is simple and robust, but not particularly accurate or reproducible with respect to powder flow rate.

Rotating Wheel Devices

Figure 2 shows a rotating wheel that delivers precise parcels of powder to the powder feed delivery tube. The wheel can be mounted either vertically or horizontally. When oriented in the vertical direction (in the geometry of a simple water wheel, as shown in the figure), the powder is dropped into the carrier gas stream. A gas tube is connected between both sides of the rotating wheel in order to equalize gas pressures on both sides of the wheel; otherwise, the powder would be blown off the pockets that are machined into the outside rim of the wheel.

The horizontally oriented wheel (or disk) has slots that pass over the gas delivery system. In this fashion, powder is taken out of the slots and enters the carrier gas stream.

In both cases, the powder delivery rate is controlled by (a) the physical dimensions of the slots or pockets that hold the powder and (b) the rotational speed of the wheel. In either geometry, it is easy to overload the carrier gas with solids and either block the powder feed tube or cause the saltation effect by using too high a powder feed rate or an insufficient carrier gas flow rate. Of course, the dimensions of the powder feed tube and the powder injection port, as well as the pressure conditions into which the powder and carrier gas are injected, also influence the powder delivery.

Fluidized-Bed Systems

Figure 3 depicts a fluidized-bed system whereby the feedstock is gas-suspended within the hopper. The powder feed rate is determined by a venturi effect that is created when the carrier gas passes across an aperture within a powder port that is placed in the hopper. The variables that control the powder feed rate are (a) carrier gas flow rate and (b) orifice size. A perceived advantage of such equipment is that it does not rely on mechanical systems and their associated instrumentation.

Other Devices Used to Feed Powders

Although the three powder feeder devices described earlier are the most commonly used in the TS industry, other systems are currently in limited use or have been used in the past. Several of these are described in this section.

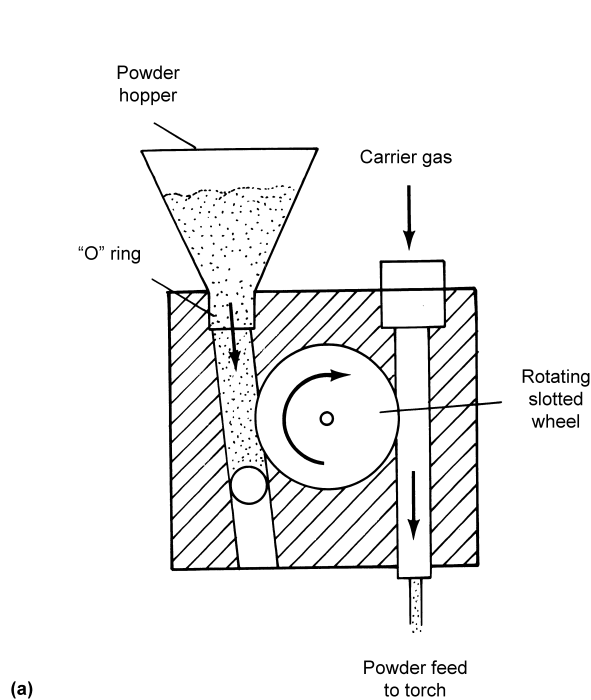
Vibratory Powder-Feed Device. With this system, the powder mass is vibrated and passes up a spiral channel that ends with

the powder moving into the feed gas stream (Fig. 4). Such devices are commonly used in material transport systems. Their adoption to powder transportation for TS has been extensive in some service shops; however, these designs are not available for the general TS practitioner.

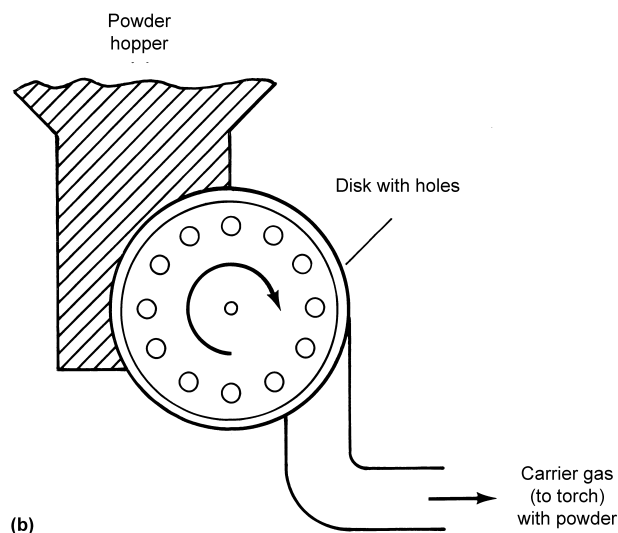
Archimedes Spiral Feeder. A delivery device based on an Archimedean spiral arrangement, which shifts the powder into the gas transport stream, has also been used (Fig. 5). This older method of material transportation was used in the early days of TS but has been superseded by modern and more reliable technology.

Encapsulated Shots. Powder may be encapsulated in well-defined mass lots (or *shots*) within a roll of paper tape (Fig. 6). The

tape can be wound onto a roll and then passed through a pressure chamber that connects directly to the spray torch. The operational concept is that a spike pierces each packet of powder so that its contents are then punched into the thermal source by a stream of compressed gas. Various valves and interconnected chambers are then synchronized so that three shots of material are expelled into the thermal spray stream per second. This method has in fact only been used for one TS system (detonation gun, or D-Gun) and is considered awkward in operation.



(a)



(b)

Fig. 2 Rotating wheel (a) and rotating disk (b) powder feeding systems

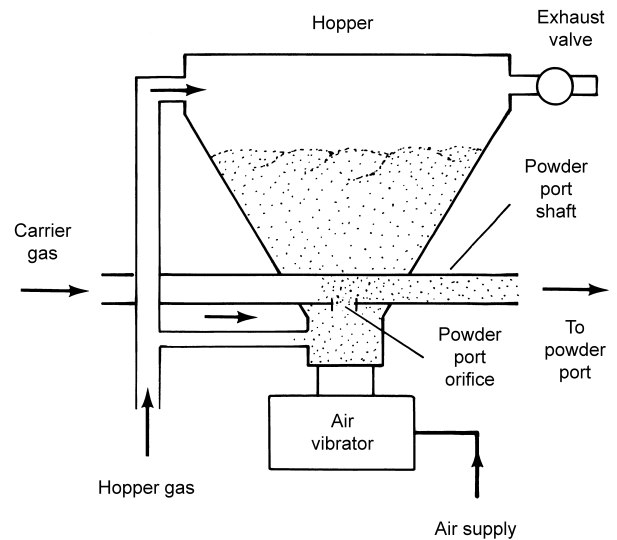


Fig. 3 Fluidized-bed powder feeding system

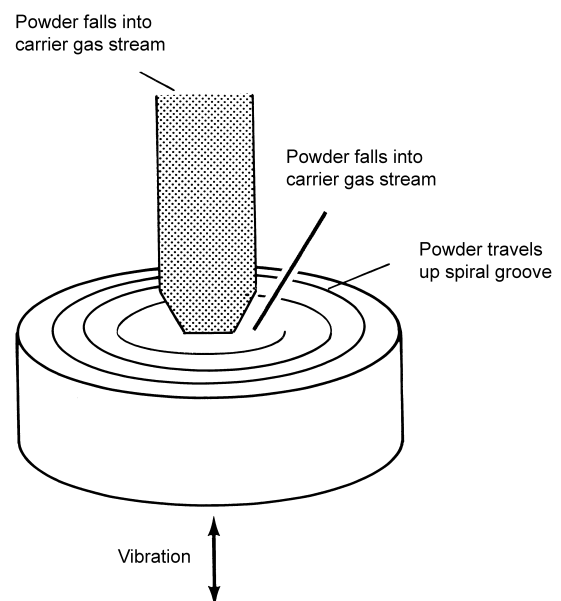


Fig. 4 Vibrating spiral powder feeding system

Experimental Devices

A more recent method of powder transportation involves feeding finely divided ($<10\text{ }\mu\text{m}$) materials into TS devices. Several experimental devices have been reported in the open literature; however, these are not commercially available and often suffer from very low powder feed rates, on the order of several grams per minute. Other experimental devices for the transport of fine materials rely on feeding slurries via peristaltic pumps or infusion injection devices.

Powder Feeding Variables

Regardless of the powder feed system used, the net effect of the powder feed device should be to carry feedstock at a consistent, smooth, nonpulsating, and reproducible rate into a gas stream that, in turn, transports the powder via a tube to the thermal spray source. The prime variables that influence the quality of powder feeding are powder density, particle morphology/size, gas density and flow rate, and whether the powder feed tube is introduced (a) directly into the heat source (such as tangentially to the plasma torch anode through the anode throat), (b) coaxially with respect to the anode axis, or (c) tangentially to the plasma torch anode external to the torch.

Avoiding Particle Agglomeration

As particles are fed into the TS gun, they must not clump together, or agglomerate, within the powder delivery system. To accomplish this, the powders must be adequately dried prior to the spray process. It is not uncommon for spray installations to have vacuum drying ovens for powder storage. This is particularly important in high-humidity environments and during seasonal variations.

Feed System Comparisons

Each of the feed systems described has attributes that may make it most suitable for a particular application. For example, the gravity-based method is usually integrated into the torch device, while rotating wheel and fluidized-bed systems are separate, stand-alone feeders. In terms of relative cost, the gravity-fed system (Fig. 1) generally is least expensive. Costs for fluidized-bed systems (Fig. 3) are approximately three to four times higher, and costs for wheeled delivery systems (Fig. 2) are approximately three to six times higher.

Feedstock/TS Heat Source Interactions

The key aim of this initial stage of the TS process is to introduce each particle of the feedstock powder into the thermal spray source so that it can be heated and then accelerated toward the substrate under sufficient thermokinetic conditions to enable spreading of the particles on impact against the substrate. The possible particle trajectories within the thermal spray source are shown schematically in Fig. 7 and 8. The essence of the powder feeding problem can now be described in terms of each particle following the correct path, as indicated in Fig. 7. Figure 8 is a more realistic

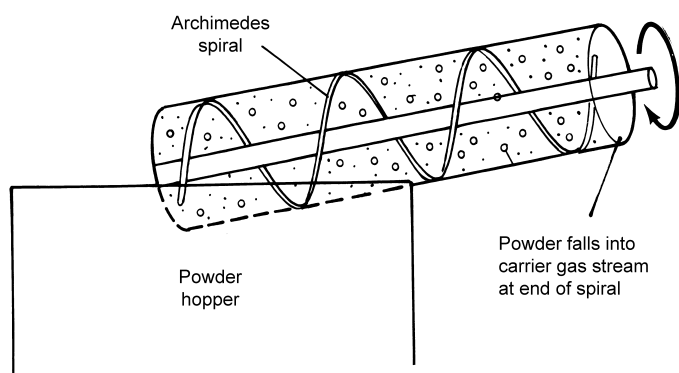


Fig. 5 Archimedes spiral powder feeding system

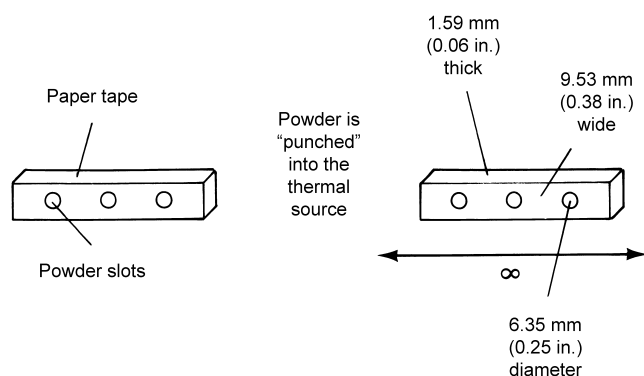


Fig. 6 Encapsulated shot powder feeding system

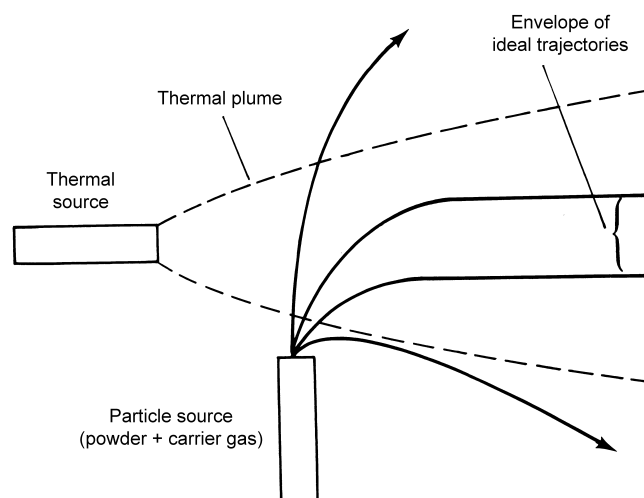


Fig. 7 Schematic of the particle trajectories within the thermal source

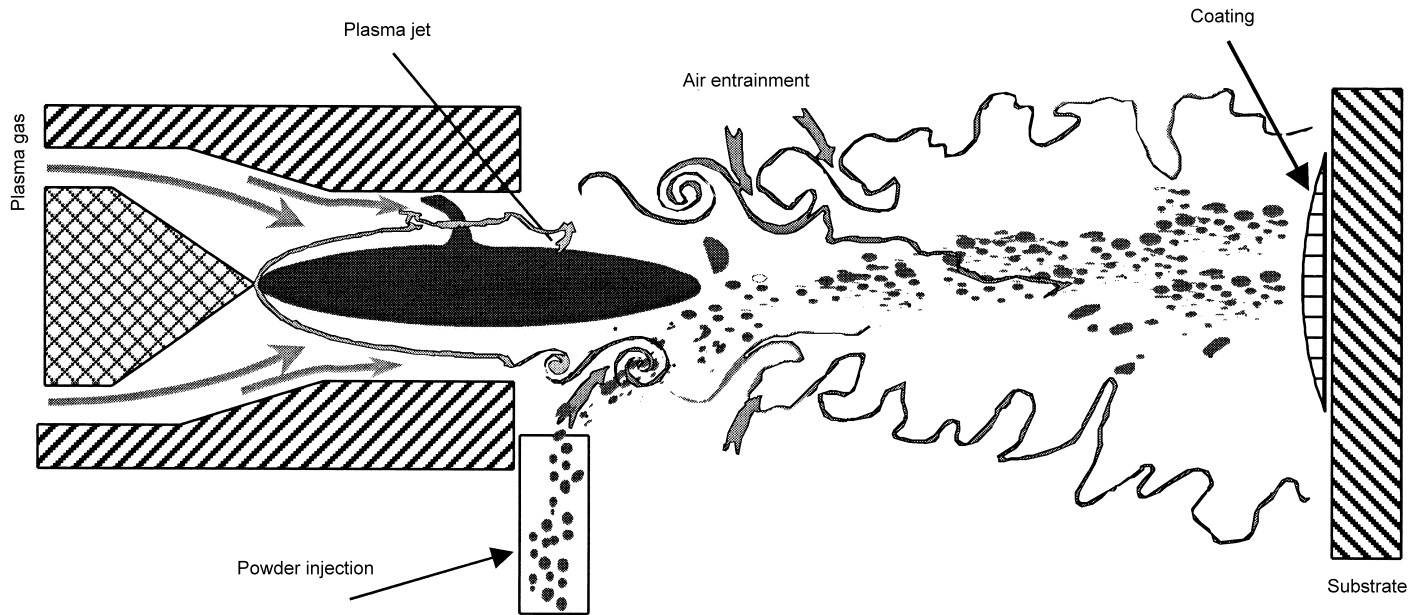


Fig. 8 Schematic of the physical interactions that occur between feedstock particles and the TS heat source. See text for details. Adapted from K.A. Gross and E. Pfender

schematic of the physical interactions that occur between feedstock particles and the TS heat source. In this particular model of the atmospheric plasma spray (APS) process, it can be observed that the plasma plume does not exhibit laminar flow. As a result, there is significant air entrainment (or “air engulfment”); consequently, oxygen/particle interactions are highly likely to occur.

Process Parameter Problems and Health Concerns

Process Parameter Problems. The impact of poor powder feeding cannot be underestimated, since it limits the utility, productivity, and efficiency of the TS process. Observations such as irregular or pulsating feed rates, powders that do not flow within their container, agglomerates of melted powder in the anode throat, and low deposition efficiencies all indicate process parameter problems that may be traced back to the feedstock. The ideal powder is thought to be mono-sized, with spherical particles that follow an ideal trajectory through the thermal spray source. Each particle becomes fully melted or attains a plastic condition so that it flows on the substrate surface and forms a coherent, dense deposit. Such powders are not commercially available for all materials.

Health Concerns. Several potential health concerns must be considered when dealing with powders. In addition to chemical toxicity, other associated dangers include:

- Inhalation of fine materials, which may cause breathing difficulties, skin and eye irritation, or a carcinogenic response
- Spontaneous combustibility of some fine materials (e.g., titanium and aluminum)
- Gases that may be combustible and asphyxiants
- High-pressure environments
- Electrical hazards associated with conducting powders in closed environments

Proper care must be taken when dealing with TS devices and materials.

SELECTED REFERENCES

- L. Pawlowski, *The Science and Engineering of Thermal Spray Coatings*, John Wiley & Sons, 1995
- *Thermal Spraying: Practice, Theory, and Application*, American Welding Society, 1985

Material Categories for Thermal Sprayed Coatings

Christopher C. Berndt, Stony Brook University

THERMAL SPRAY (TS) COATINGS can be categorized or classified according to their material chemistry and type, material morphology, particle size distribution, spray process, and/or application. The relationships among these coating categories are represented in Fig. 1, which shows that the initial chemistry and form of the feedstock is of utmost importance with regard to the engineering application (see the dashed line). An understanding of these relationships is critical when choosing particular feedstocks.

Classification According to Thermal Spray Process

Selection of the TS process often preselects the preferred feedstock. Examples of process/feedstock couples include:

- Arc metallization and wire-plasma arc process: wire feedstock
- Atmospheric plasma spraying (APS): powder feedstock
- Ceramic rod spraying (or the “Rokide” process): rod feedstock

- Cold spraying processes: powder feedstock
- Detonation gun spraying (D-Gun): powder feedstock
- High-velocity oxyfuel processes (HVOF, Jet Kote, D-J Gun, and others): powder feedstock, although wires have also been employed
- Induction plasma spraying (also called the radio frequency, or RF, induction torch): powder or sol-gel feedstock
- Liquid-stabilized plasma torches: powder feedstock
- Low-pressure plasma spraying (LPPS, VPS): powder feedstock
- Oxyacetylene gas flame spraying (flame spraying, combustion spraying): powder feedstock

Combinations of these processes may be used in tandem or simultaneously.

Considerations for Arc Metallization. The nature of a particular TS process is directly related to the types of materials that can be sprayed by it. For example, arc metallization requires that the feedstock be in the form of electrically conducting wires. It is

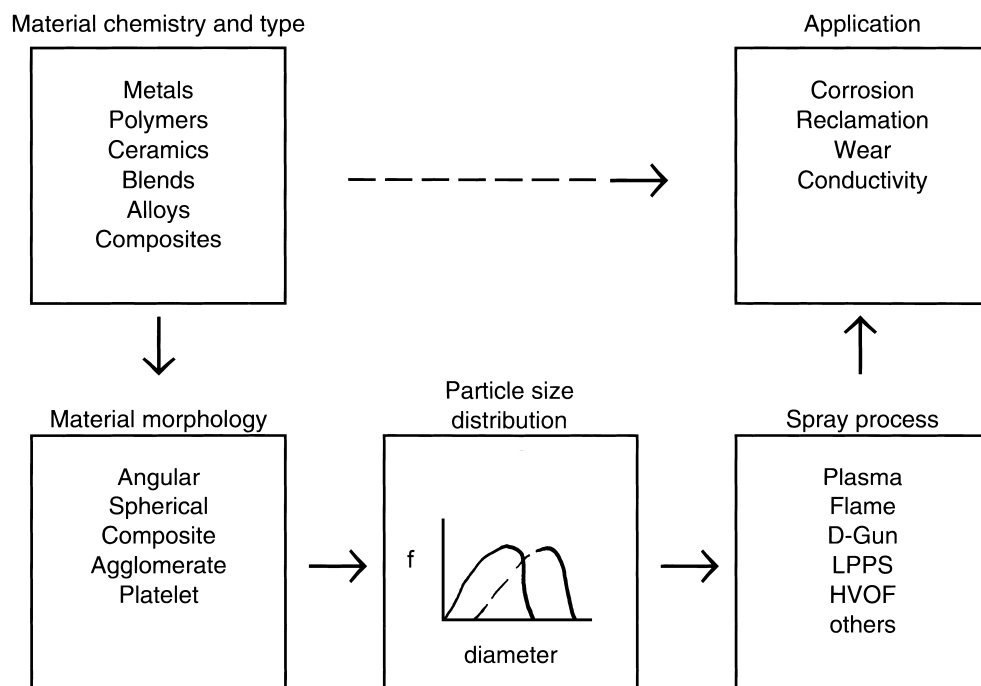


Fig. 1 Relationships between material chemistry and the intended application. The letter “ f ” on the vertical axis of the particle size distribution curve denotes frequency of number (in %) of particle diameter.

clear that the material cannot be an insulating ceramic or a polymer. However, it may not be as obvious that nonconducting materials can be inserted into conducting tubes so that arc metallization procedures can be utilized.

Effects of Process Temperature and Velocity on Feedstock Selection. Process temperature and velocity are also matched to feedstock characteristics. Processes such as flame spraying have relatively low temperatures and velocities and therefore may not be able to melt high-melting-point ceramics. One way of overcoming this type of problem is to decrease the average particle size; however, this creates another range of difficulties associated with the reliable feeding of such fine materials. This conflict between feedstock melting ability and process conditions can be resolved by changing to a TS technique that employs a more intense heat source, such as plasma spraying. Thus, a measure of the characteristics of a particular feedstock can be gaged by examining its spraying performance under certain processes.

Considerations for LPPS. Low-pressure plasma spraying allows reactive metals such as titanium and nickel alloys to be processed in an inert environment. These metal alloy materials oxidize under ambient spraying conditions, and the oxide boundaries formed result in poor high-temperature performance of such coatings. A special market for LPPS coatings involves use of NiCrAlY as bondcoats in thermal barrier coating (TBC) systems, which require dense, oxygen-free coatings.

On the other hand, the LPPS process offers limited advantages for the spraying of ceramic materials, because these feedstocks do not chemically degrade during spraying. The only perceived advantage for the LPPS processing of ceramics is that the particles attain a higher velocity and may be better able to form splats. This comes, however, at the expense of a shorter in-flight residence time; consequently, particle sizes must be smaller than those used for the APS process in order for the feedstock particles to reach their softening point. The upshot of this argument is that special particle size distributions (or “cuts”) of powder can be manufactured for LPPS processes, but these are more expensive than the usual cuts designed for the APS processing of ceramics.

Controlling Thermal Interactions. Certain behaviors of materials during thermal spraying can be controlled by altering the TS process. For example, the thermal interactions of superheated feedstock particles with the environment can be controlled by flooding the work area with an inert gas such as argon. This more costly method of obtaining a “clean” TS microstructure is termed a *shrouded gas environment*.

Thermal Spray Parameters. Thermal spray processes also involve a set of variables that are collectively known as spray parameters. These parameters include:

- Equipment specifications (i.e., torch type and model)
- Equipment operating parameters (i.e., voltage, amperage, power level, etc.)
- Gas pressures and gas consumption rates
- Torch-to-substrate distance (also called the standoff distance)
- Powder specifications (i.e., chemistry, size distribution, morphology, and density)
- Powder feed rate
- Substrate surface preparation

- Substrate handling parameters
- Substrate cooling requirements
- Safety and handling requirements
- Coating thickness(es)
- Deposition efficiency (DE) and, in some cases, spray efficiency.

With respect to the terms used in the last bullet, DE is defined as the amount of material deposited relative to the amount of material fed to the TS equipment. This calculation assumes a substrate of infinite size. The deposition rate, or DR, is the number of kilograms deposited per hour. The spray efficiency, or target efficiency, is the amount of feedstock that is actually deposited on the component relative to the amount of material fed to the TS equipment.

Spray Tables for TS Parameters. Table 1 presents a sample checklist employed for a plasma spray operation.

Table 1 Checklist for a plasma spray process

Plasma processing equipment
• Torch type (anode, cathode, injector ring), mm
• Volts and amps = power, kW
• Primary gas and flow rate, slpm
• Secondary gas and flow rate, slpm
• Feed gas and flow rate, slpm
• Standoff distance, mm
• Powder injection, mm
• Powder feed rate, g/min
Hardware
• Traverse speed of torch, m/s
• Substrate cooling, m ³ /s
• Spray footprint, mm ²

Checklists of this nature are often referred to as “spray tables” and can be defined as “a list of primary TS parameters that need to be specified and controlled during a specific coating operation.” All these parameters influence process economics and coating quality. In many instances, manufacturers will indicate the physical properties that can be expected if coatings are produced using their recommended spray parameters. Properties such as surface roughness, microstructure porosity, thickness per pass, hardness, and adhesion may be indicated. These values should be used only as a rough guideline for coating properties. The physical properties of the substrate are also very important, being essential to the production of an appropriate surface profile. For example, a hard tool steel substrate requires different grit blast parameters than a softer material in order to achieve the best surface profile.

Even with similar TS processes, the TS parameters are likely to change from process to process and between different equipment. Powders that are presumably identical may exhibit different spray parameters for each set of equipment.

Classification According to Material Morphology

Particle Morphology and TS Process Relationship. Figure 2 shows the morphologies of various feedstock materials. The material morphology refers to the shape of the individual particles; this

characteristic is commonly determined via optical (400× magnification) or scanning electron microscopy (SEM) at magnifications of less than 1000×. (Numerous particle morphologies examined by SEM are shown in the article “Powder Characterization” in this Section of the Handbook.) The morphology is directly related to the TS process (as indicated in Fig. 2), since this is the prime feedstock characteristic that controls its ability to be transported and to interact with the TS heat source in a consistent and reproducible fashion.

Internal Porosity. Figure 3 shows that the observed surface morphology may be deceptive, due to hidden internal features. This internal porosity greatly influences powder flow characteristics from the hopper to the TS device, as well as the physical interactions between the powder and the TS source.

Tailoring Feedstock Morphology and Properties. In many instances, powders of equivalent chemistry may be produced by

different routes. For example, Table 2 presents a case history for yttria-stabilized zirconia (YSZ) where five different feedstock manufacturing methods give rise to a wide range of macro- and microscopic morphologies and physical properties. Thus, to a certain extent the feedstock morphology and properties can be tailored.

Particle Shape and Size Distribution. Particle shape is unerringly controlled by the production process. It is incorrect to assume that the powder surface is smooth or that each particle is chemically homogeneous and 100% dense. The particle shape determines the material transport from the powder hopper to the spray torch. Thus, determination of this characteristic is essential. Particle size distribution is also important, because it is optimized with regard to coating quality. It is generally agreed that a closely sized distribution (i.e., a “narrow cut”) is required to take advan-

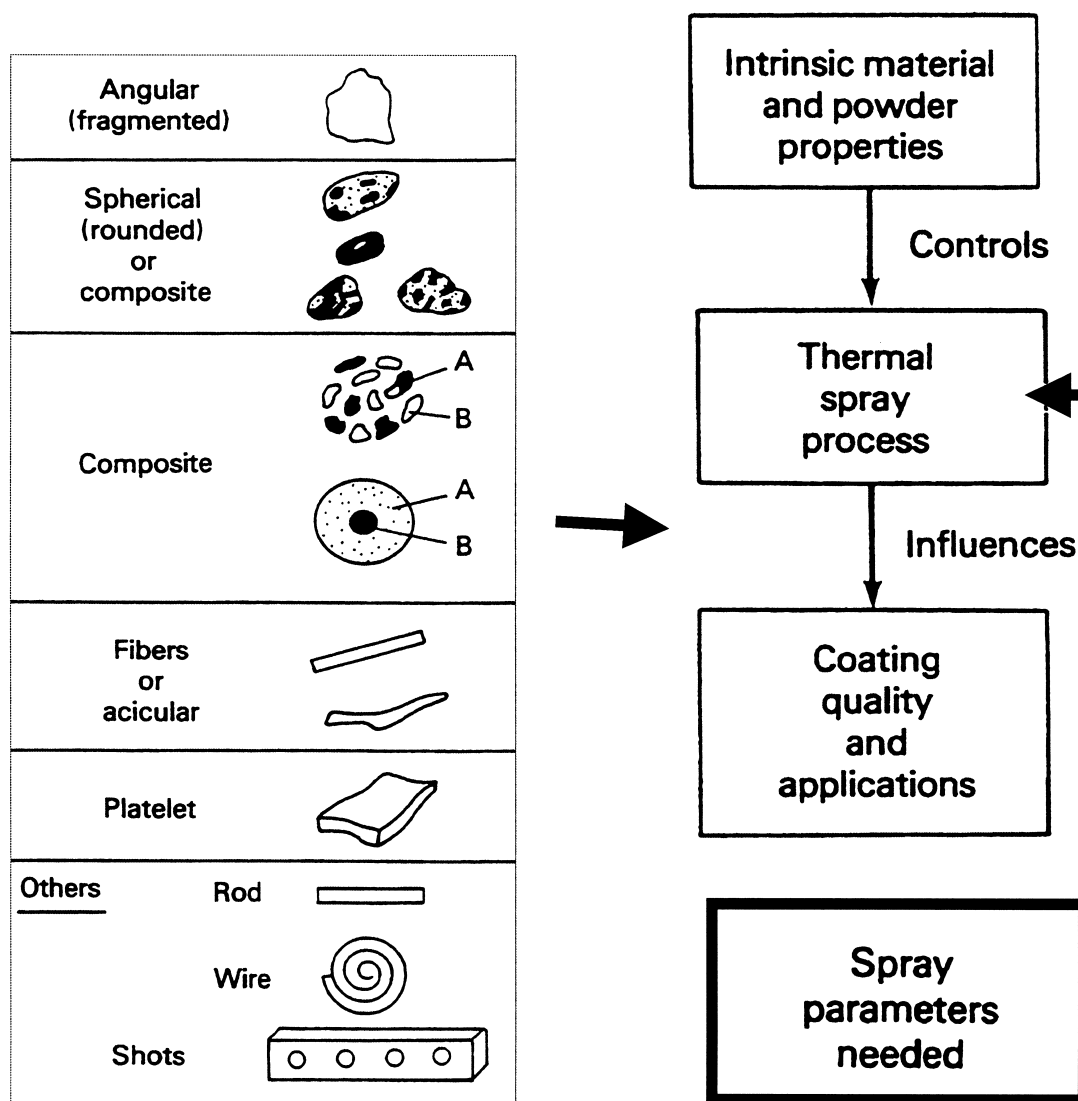


Fig. 2 Range of feedstock morphologies. The accompanying flowchart indicates how these properties may influence the thermal spray process.

tage of the optimum particle trajectories through the TS heat source so that a dense coating can be manufactured.

The ideal particle morphology is spherical, although needlelike and platelet forms are of research interest since they may confer special properties to the coating. For example, Fig. 4 depicts a

research method intended to produce fibers which may be transported into the melting zone of a TS torch in order to manufacture a composite coating where the original needle structure is retained.

Classification According to Chemical Nature of the Material

The broad classes of TS materials used are:

- *Metals and alloys:* for example, aluminum, aluminum-zinc, copper, molybdenum, nickel-aluminum alloys, and nickel-chromium alloys (NiCrAlY, Ni-Al)
- *Ceramics:* for example, aluminum oxide (alumina, Al₂O₃), chromium carbides, chromium oxide (chromia, Cr₂O₃), hydroxyapatite (HA, 10Ca-4[OH]·6[PO₄]), mullite (3 alumina + 2 silica, SiO₂), spinel (magnesia, MgO + alumina), titanium oxide (either as titania or rutile polymorphs, TiO₂), tungsten carbide-cobalt (WC-Co), YSZ (Y₂O₃-ZrO₂), and zirconium oxide (zirconia)
- *Intermetallics:* for example, NiAl, Ni₃Al, and NiAl₃
- *Composites and blends:* for example, aluminum/silicon/polyester, molybdenum-nickel-chromium-boron-silicon, nickel-graphite, and bentonite-NiCrAl
- *Polymers:* for example, nylon, polyesters, polyamides, and polyethylenes

Combinations of metals and ceramics, called cermets, can be manufactured as composites or blends. The powder is classified as

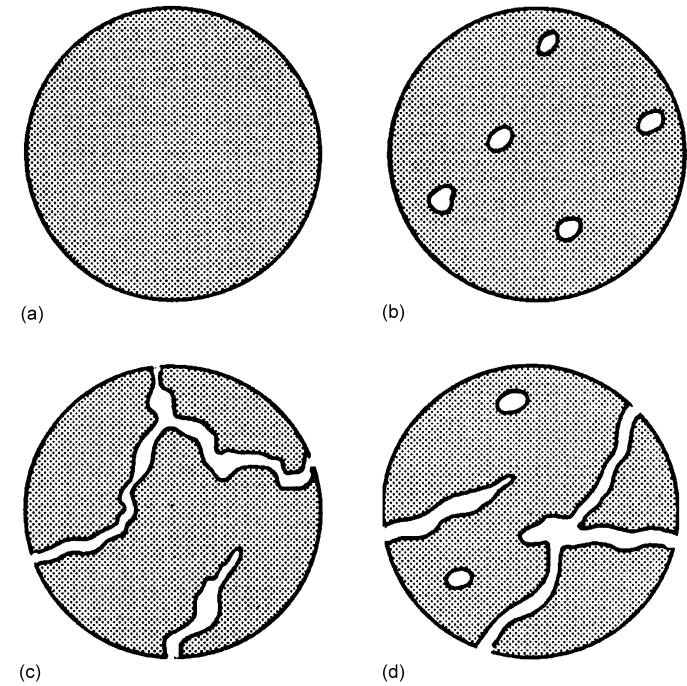







Fig. 3 Schematic comparing a fully dense powder with powders containing different types of internal porosity. (a) Fully dense. (b) Closed pores. (c) Interconnected pores. (d) Closed and interconnected pores

Table 2 Various forms of particle morphologies and corresponding physical features for YSZ

Feature	Powder type				
	Fused + crushed	Sintered + crushed	Spray dried	Plasma fused	Sol-gel
					
Particle shape	Blocky-angular	Blocky-angular	Spherical	Spherical	Spherical
Microstructure and porosity	Dense	Dense-porous	Porous	Dense-hollow	Dense
Grain size	Coarse	Coarse	Medium fine	Medium fine	Fine
Bulk density, g/cm ³	2.7	2.1	1.8	2.3	2.6
Hall-flow(a), s	32	40	40	34	22

(a) Flow rate as determined using a Hall flowmeter (ASTM B 213). Source: Courtesy of K.A. Gross, Monash University, Melbourne, Australia

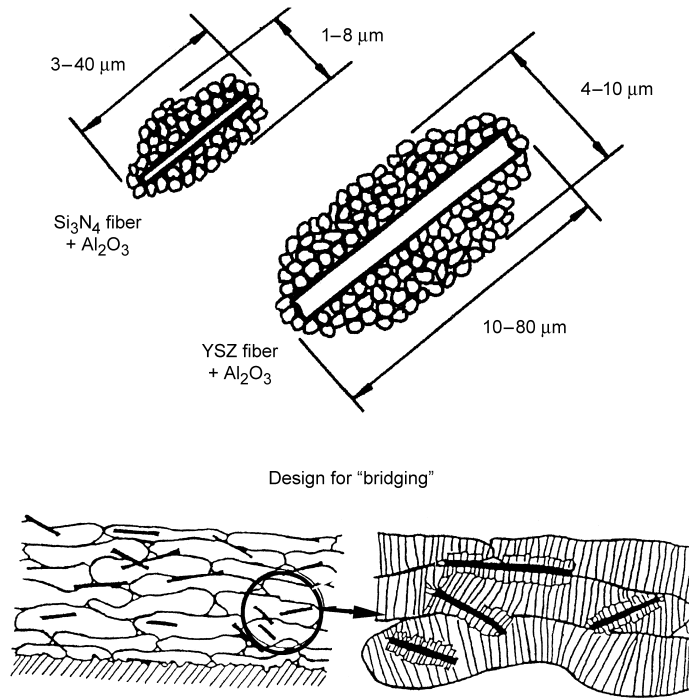


Fig. 4 A specialized “research quality” feedstock showing how particle design can be used to incorporate functional geometries

a composite when the two or more constituents are in close contact with one another, often under the strong forces of chemical interactions. (This definition of “composite” is special to the thermal spray field and is quite different from the definition most often used in materials technologies.) For example, as described in the article “Material Production Processes” in this Section, composites are formed during cladding and spray drying processes. Blends such as some powders of aluminum/silicon/polyester are created by the simple mechanical combination of the constituent materials so that they are more likely to interact with one another during the coating process. One upshot of these different processing methods is that composite powders will exhibit greater durability under mechanical forces than blended powders.

The characteristic of material chemistry is often related directly to the coating application. Knowledge of the chemistry allows close matching to specific properties of the substrate. For example, reclamation coatings may require feedstocks of similar chemistry so that the initial component characteristics are retained. On the other hand, protective coatings can be manufactured that consist of a material dissimilar to the base material. Thus, materials are often classified as ceramics when an abrasive or thermal barrier coating is required, or as a polymer for chemical resistance or low friction.

The classification of composites (see Fig. 2) has a special meaning in TS technology since it refers to powders that consist of two or more distinct chemical species. The composite structure can be in the form of an aggregate of two (or more) distinctly dual-phase regions. Every particle of the material exhibits this inhomogeneous structure. The mechanical combination of different powders is referred to as a *blended* material; that is, the two materials are not physically or chemically joined together.

The terms *composite structure* or *composite coating* are reserved for coatings that consist of two or more distinct morphological features, such as spherical particles in a fully melted coating or needle-shaped artifacts in a coating. *Graded coatings* are also often described as being composites, although such a description is misleading. Graded coatings consist of two (or more) different materials that have been blended together in a graduated fashion so that the composition changes from a majority of one species at the substrate to a majority of the other species at the outer part of the coating. The material structure is probably more correctly described as being a cermet, since the two prime blending constituents are often a metal and a ceramic. This is one unambiguous example where the inhomogeneous structure of TS coatings can be employed to confirm enhanced functionality in terms of a composite structure.

Classification According to Applications

The preceding sections of this article reveal that coating technology often incorporates a coating system that consists of a number of separate constituents. For example, a bondcoat is often

deposited prior to a ceramic overlay. The utility of many materials could be quite limited without this coating design, and the classification according to applications assumes that an appropriate coating *system* has been developed. It is often only the outermost layer that confers the intrinsic material property of interest to the component. Application classifications include:

- *Abrasive wear*: buffing and polishing machines, polishing rod liners
- *Adhesive wear*: piston guides, thrust bearing shoes, bronze and babbitt bearings
- *Atmospheric and immersion corrosion*: electrical conduits, bridges, transformer cases, steam cleaning equipment, ship superstructures, ship holds and tanks, storage tanks for oils, fuels, and solvents, power-line hardware, heat exchangers
- *Cavitation*: water turbine buckets, wear rings for hydraulic turbines, impeller pump housings
- *Electrical conductivity and resistivity*: ground connectors, lightning arresters, solder iron tips, ground coating for locomotive axles
- *Fretting*: cylinder liners, automotive valves, rocker arms, lathe and grinder dead centers
- *Hard bearings*: fuel pump rotors, impeller shafts, piston rings, armature shaft journals
- *Hard surfaces*: sizing punches, extrusion dies, pump seals, hot crushing rolls, hot forming guides
- *Heat and oxidation*: tuyeres for liquid metal resistance, continuous casting molds, heat-treating fixtures and brazing jigs, exhaust mufflers
- *Machine element clearance control*: air seals (to replace silver coatings), compressor seals (to replace rubber), aircraft engine components
- *Particle erosion*: exhaust fans, cyclone dust collectors, exhaust valve seats
- *Power generation*: manufacture of solid oxide fuel cells
- *Repair and reclamation*: machine components, shafts, bearings
- *Thermal control*: thermal barrier coatings, insulating coatings

More detailed information on applications for thermal spray coatings can be found in the article “Selected Applications” in this Handbook.

SELECTED REFERENCES

- B.A. Kushner and E.R. Novinski, *Thermal Spray Coatings, Friction, Lubrication, and Wear Technology*, Vol 18, *ASM Handbook*, ASM International, 1992, p 829–833
- L. Pawlowski, *The Science and Engineering of Thermal Spray Coatings*, John Wiley & Sons, 1995
- *Thermal Spraying: Practice, Theory, and Application*, American Welding Society, 1985
- R.H. Unger, *Thermal Spray Coatings, Corrosion*, Vol 13, 9th ed., *Metals Handbook*, ASM International, 1987, p 459–462

Material Production Processes

Christopher C. Berndt, Stony Brook University

THERMAL SPRAY (TS) POWDERS have evolved principally from powder metallurgy procedures. The most recent developments have arisen from ceramics and composites processing techniques. The production of polymeric powders that are suitable for TS is an emerging engineering field. The powder production technique has a marked influence on the nature of the powder that is produced. This article focuses on TS-related technology and process principles. The generic terms that describe the manufacture of powders by mechanical processes are *comminution* and *attrition* and denote the breaking up and size reduction of solid materials. The normal range of particle sizes required for TS, from 5 μm to about 120 μm , is shown in Fig. 1 relative to other manufacturing needs.

A general overview of a specific powder production process is shown in Fig. 2. In this example, which depicts a spray drying process, there are some 13 major steps between starting with raw materials and finishing with a product that can be shipped to a customer. It should be noted that mechanical and chemical methods can be used in combination to produce a single product.

Table 1 presents the general relationships between the mechanical and chemical routes of feedstock production. Some conventional comminution processes will be described first, followed by discussions of atomization and chemical preparation methods. (Atomization can also be classified as a mechanical powder production method. It differs from conventional comminution in that it involves the dispersion of melts rather than the dispersion of solids.) The powder preparation subcategory that is used extensively for the production of ceramic feedstocks lies within the solution methods listed in Table 1 and is termed the “sol-gel” method.

Machining Techniques

These are expensive processes, employed for relatively difficult-to-work materials such as magnesium, copper, and aluminum alloys, and noble materials such as gold, silver, and platinum. A rod of the cast material is typically lathe cut and the machine fragments collected. Often the process is performed under an inert atmosphere to mitigate the likelihood of spontaneous combustion of the product (e.g., magnesium). There is little call for this mechanical method in TS powder production.

Crushing and Milling

Crushing. The purpose of crushing is to break up large particles into smaller size fractions using mechanical energy. These processes are used mainly for brittle ceramics, since metals would be plastically deformed rather than broken up. High-purity feedstock can be made by melting the raw materials in an electric furnace. These so-called “cast” materials can then be crushed to form “cast-and-crushed” powders.

Table 1 Advantages and disadvantages of selected powder preparation techniques

Powder preparation technique	Advantages	Disadvantages
Conventional method		
Conventional comminution	Inexpensive Unaggregated powders Wide applicability	Limited purity Limited homogeneity Large particles
Chemical preparation		
Solution methods	High purity	Expensive
Solvent vaporization	Small particles	Aggregation of particles
Simple evaporation	Compositional control	Poor for nonoxides
Spray drying	Chemical homogeneity	
Spray roasting		
Fluid bed drying		
Emulsion drying		
Sol-gel or glass drying		
Freeze drying		
Precipitation or coprecipitation		
Vapor-phase methods	High purity	Expensive
Vaporization-condensation	Very small particles	Difficult for multi-component materials
Vapor decomposition	Low aggregation	
Vapor-vapor reaction	Good for nonoxides	
Salt decomposition	Used for solution techniques Simple apparatus	Aggregation of particles

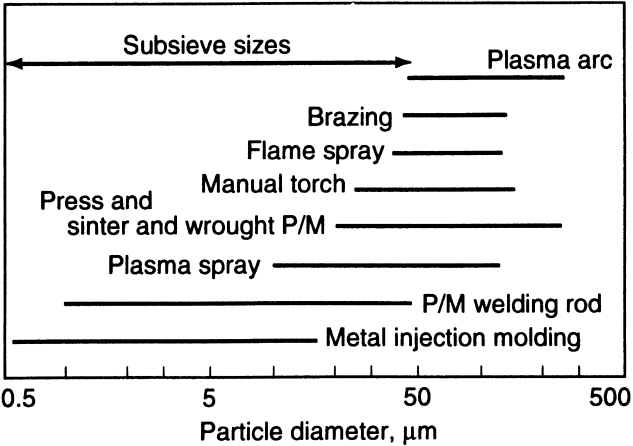


Fig. 1 Particle size ranges for common industrial processes, including flame spray and plasma spray TS powders. Source: Ref 1

A variation of cast-and-crushed powders is to form the powders via a process known as self-propagating high-temperature synthesis (SHS). The SHS method relies on the ability of high exothermic reactions to be self-sustaining and thus energetically efficient. The exothermic reaction is initiated and generates heat to volatilize low-boiling-point impurities, resulting in purer products than those produced by more conventional techniques. Powders produced by SHS can then be crushed into appropriately sized powders.

Crushing Followed by Milling. Ceramics and some metals can be reduced in size by mechanical impact and deformation in *attrition* processes. Crushing processes employ equipment such as hammer mills, stamping mills, jaw crushers, and gyratory crushers. The crushing stage is often followed by a more refined technique known as *milling*, which enables control of particle size. Milling involves the disintegration of brittle, friable materials

(some metals and most ceramics) or the pulverization of malleable metals. The milled particles are often irregularly shaped and of variable size (sometimes smaller than 5 μm). All of these powders are classified so that any fines can be postprocessed by agglomeration to form a suitable TS powder.

Milling machines include rod mills and ball mills. Care must be taken to ensure that the grinding medium does not unduly contaminate the material that is being reduced in size. The milling medium is usually of high specific gravity. For example, alumina, steel, zirconia, and mullite are common, although cemented carbides are sometimes used where contamination must be kept to a minimum. The use of higher-density grinding media gives rise to higher grinding rates because the impact during tumbling is greater. The mill liners are generally vulcanized rubber, polyurethane, high-density alumina, porcelain, tungsten carbide (for laboratory mills), or stainless steel.

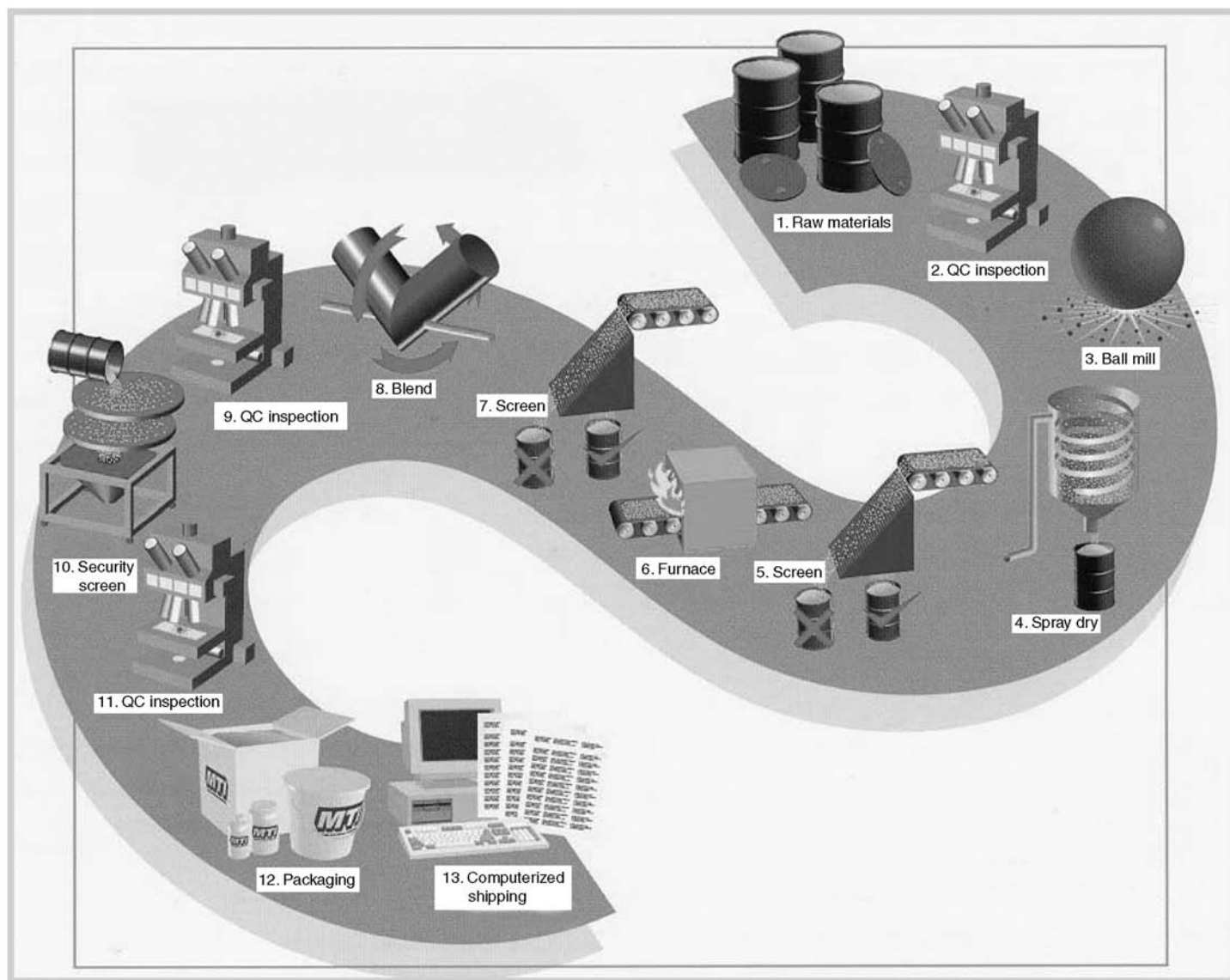


Fig. 2 A generalized powder production schematic indicating 13 steps in the manufacture of feedstock. Source: Courtesy of Praxair TAFE, Inc., Indianapolis, IN

Rod mills grind large particles (greater than $15\ \mu\text{m}$) more efficiently than ball mills. Ball mills grind all particles to the same degree, resulting in more variable size distribution. Ball mills are filled with the grinding media to approximately 50% of the total volume. The powder charge constitutes 25% of the total mill volume for dry ball milling and is generally 30 to 40% of the total volume for wet ball milling operations. A small amount ($\sim 1\ \text{wt}\%$) of a grinding aid such as stearic acid or folic acid is added to prevent caking of the powder charge. In wet ball milling, the charge is suspended in an inert liquid such as alcohol, acetone, or water. High-viscosity suspensions usually give rise to low grinding rates, while the other extreme of low solids content increases the wear rate on the mill lining and grinding media.

The speed of the rod or ball mill is quite critical with regard to equipment life and process efficiency (see Fig. 3). The balls (or rods) must drop from the top of the mill onto the material that is being ground (Fig. 3b). If the mill speed is too high, the grinding media will either not fall at all due to centrifugal forces or will fall

directly onto the media near the bottom of the mill and accelerate media wear due to chipping (Fig. 3c). At low speeds the media does not drop at all, but merely rolls around the bottom of the mill (Fig. 3a). At optimum speed the media continuously cascades onto the material that is being crushed (Fig. 3b).

Another common device is the high-energy attritor-type ball mill (Fig. 4). In this mill, an impeller with a Christmas-tree geometry rotates at about 250 rpm and causes ball media contained within a stationary tank (having a volume of ~ 0.004 to $0.4\ \text{m}^3$, or 1 to 100 gal) to impact and collide against the ceramic material being pulverized. Because heat is generated, the container is water cooled. This ball mill attritor controls particle size much the same as a conventional ball mill (Fig. 3), since it is the kinetic energy of the balls that imparts the fracture force for the feedstock material breakup.

The newly formed surfaces of pulverized material are highly active; therefore, particles may tend to cluster into agglomerates. This decreases attrition process efficiency. Organic additives such as alcohols are added in amounts of less than $0.1\ \text{wt}\%$ to reduce agglomeration and thereby improve grinding efficiency. The active surfaces of newly formed particles may also cause enhanced reactions with the environment or activate any post-processing operations such as sintering. A critical minimum particle size is eventually attained during ball milling, whereby the stress increase due to the reduced particle size is sufficient to cause particle welding rather than further particle fragmentation. This is the stage where further milling will not take place and usually limits ball milling operations to particle sizes of approximately $5\ \mu\text{m}$.

Problems of mill wear have been partially overcome with fluid energy and shear mills. The operating principle of these devices is essentially the same. The material is incorporated into either gas or fluid streams, and the two streams of fluidized material are

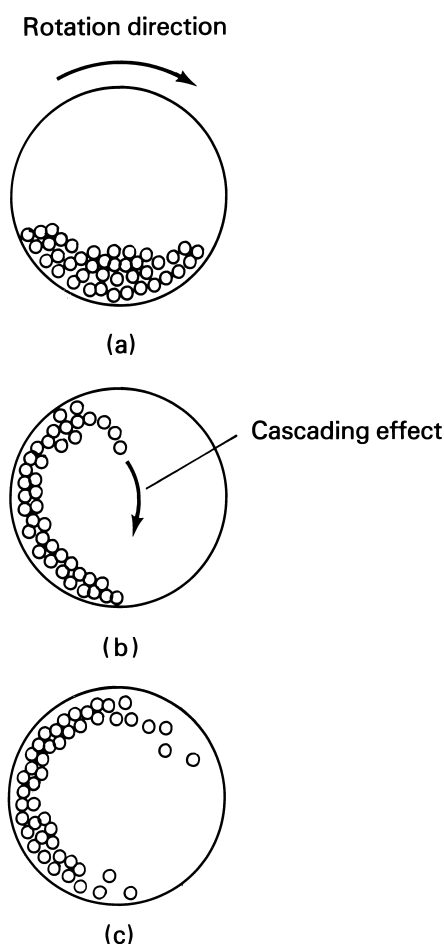


Fig. 3 Comminution variables that control particle grinding and efficiency. (a) Low speed. (b) Optimum speed. (c) High speed. The balls or rods within the mill are indicated.

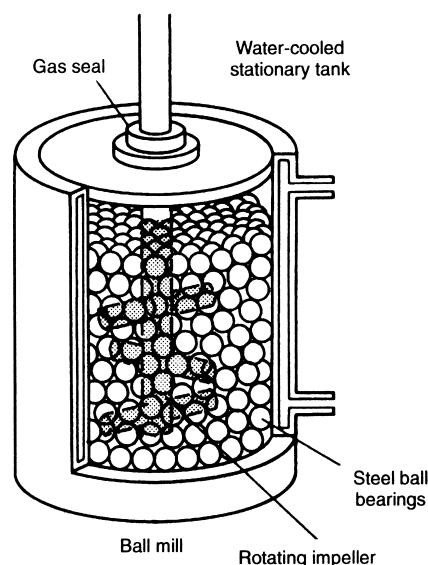


Fig. 4 Schematic of a high-energy attritor-type ball mill

forced to be coincident (Fig. 5). The individual particles collide and abrade one another. There is minimal product contamination in these mills since there is little high-velocity contact with the mill walls. The major problem involves separation of fine particles from the fluidizing gas or liquid. The required filtering system adds to the complexity of the milling operation.

The primary purposes of milling are to:

- Reduce the size of particles or agglomerates
- Eliminate particle segregation or preferred orientations in single-phase particles
- Homogeneously disperse the many components in multicomponent powder systems

Three basic stages occur during the milling process: an initial rapid reduction in the size of aggregates, fracture of individual particles, and, eventually, reagglomeration of fine particles after extended milling. The initial aggregates may be approximately 10 to 15 μm in size and will be reduced to fines on the order of 0.1 μm .

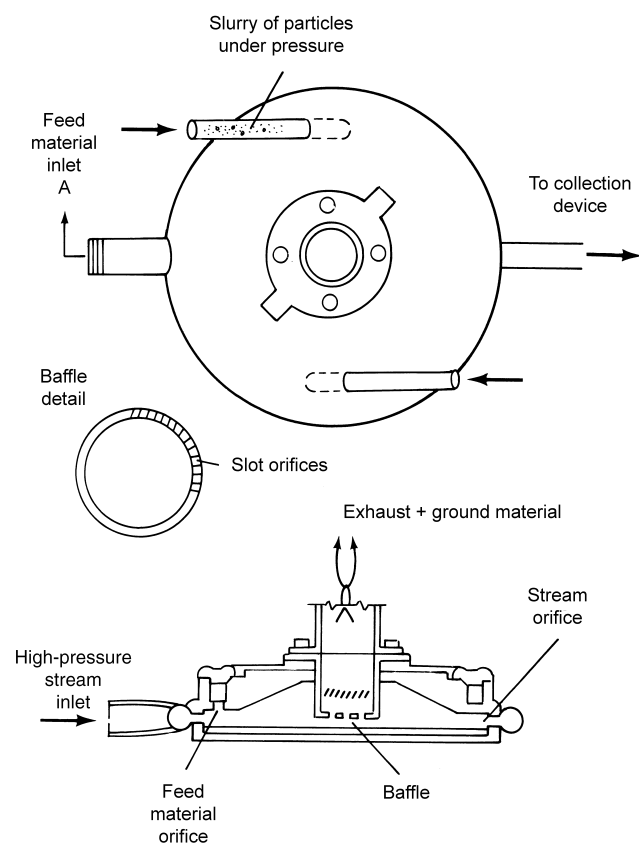


Fig. 5 Key components of a fluid energy mill

Atomization

An overall schematic of an atomizing plant is shown in Fig. 6. Eight basic steps can be identified, although some of these may be consolidated under various plant configurations. Atomization includes the four process categories of gas, water, centrifugal, and mechanical. Only the gas and water techniques are extensively used for TS powder production, especially for the production of metallic powders. Particle sizes vary between 10 and 250 μm . The principles of the gas and water processes are quite similar in that a continuous stream of liquid metal is broken down into droplets by the impingement of a gas or water stream. A variety of process parameters allow the particle morphology and size distribution to be varied.

One advantage of atomization facilities is that they can produce small experimental lots of several kilograms in tabletop configurations or be several stories in height and capable of producing several hundred kilograms in a single heat. They can be configured in a vertical arrangement or, for gas atomization, horizontally to conserve vertical height. The principal atomization components of a facility are contained primarily within the vertical section (parts 4, 5, and 6 of Fig. 6) and include:

- Melting and dispensing crucible (or *tundish*)
- Atomizer and associated control system
- Water-cooled spray chamber
- Cyclone separator or settling tank
- System for the storage and supply of gas or water
- Cooling section for the effluent
- Powder collection devices

Water Atomization

Figure 7 shows a water atomization process that produces irregularly shaped particles, although more spherical powders can be obtained by giving the melt a large degree of superheat (i.e., a temperature significantly above the material melting point). Water atomization constitutes a large portion of powder production techniques, primarily because it lends itself to high production rates. These powders usually require dewatering, drying, and milling after production. If the high oxygen content of these powders is unacceptable, then a reduction stage is necessary. It should be noted that the high oxygen content of nickel- and cobalt-base powders produced by water atomization is not overly detrimental for TS applications. Water-atomized powders generally have mean particle sizes in the range of 150 to 400 μm , although this range can be extended down to approximately 10 μm by altering the atomization process variables. For example, the water pressure, the configuration of the water nozzles relative to the metal stream, and the geometry of the liquid metal tundish all control the final powder cut.

Particle Formation. Two mechanisms have been proposed with regard to particle formation: the “splash” and “scrape” disintegration models (Fig. 8). In the splash model, a water droplet collides with the metal stream, causing a tongue of liquid metal to eject from the stream. This liquid tongue then reconstitutes itself

into metal droplets in response to minimizing surface tension. In the scrape model, a droplet of water skims across the liquid metal stream and shears off liquid metal, which then consolidates into spheres.

Gas Atomization

Gas/Metal Interactions. Gas atomization can be carried out using air, steam, nitrogen, argon, or helium (Fig. 9). In many ways the process is analogous to that of water atomization in that an impacting media interrupts a flowing stream of liquid metal. The model for the gas/metal interaction shown in Fig. 10 demonstrates the various stages where the breakup of the metal stream occurs. Essentially, the initially stable stream of liquid metal is broken up into waves, ligaments, and then droplets as its flow becomes more chaotic.

Operating Variables. A number of principal operating variables influence the powder quality produced by atomization techniques. The features of an atomizer that control product size and quality are shown in Fig. 11. These include:

- Atomizing jet distance, geometry, and pressure
- Nozzle geometry
- Velocity of the atomizing media and the metal
- Melt superheat (i.e., the temperature above the melting point of the metal)
- Gas purity. This controls the purity of the atomized product; usually inert gases of the 99.99% purity level are used.

Powder Characteristics. The powders produced can be extremely clean (i.e., about 100 ppm of oxygen), especially when produced under inert gas conditions. Atomization is a viable pro-

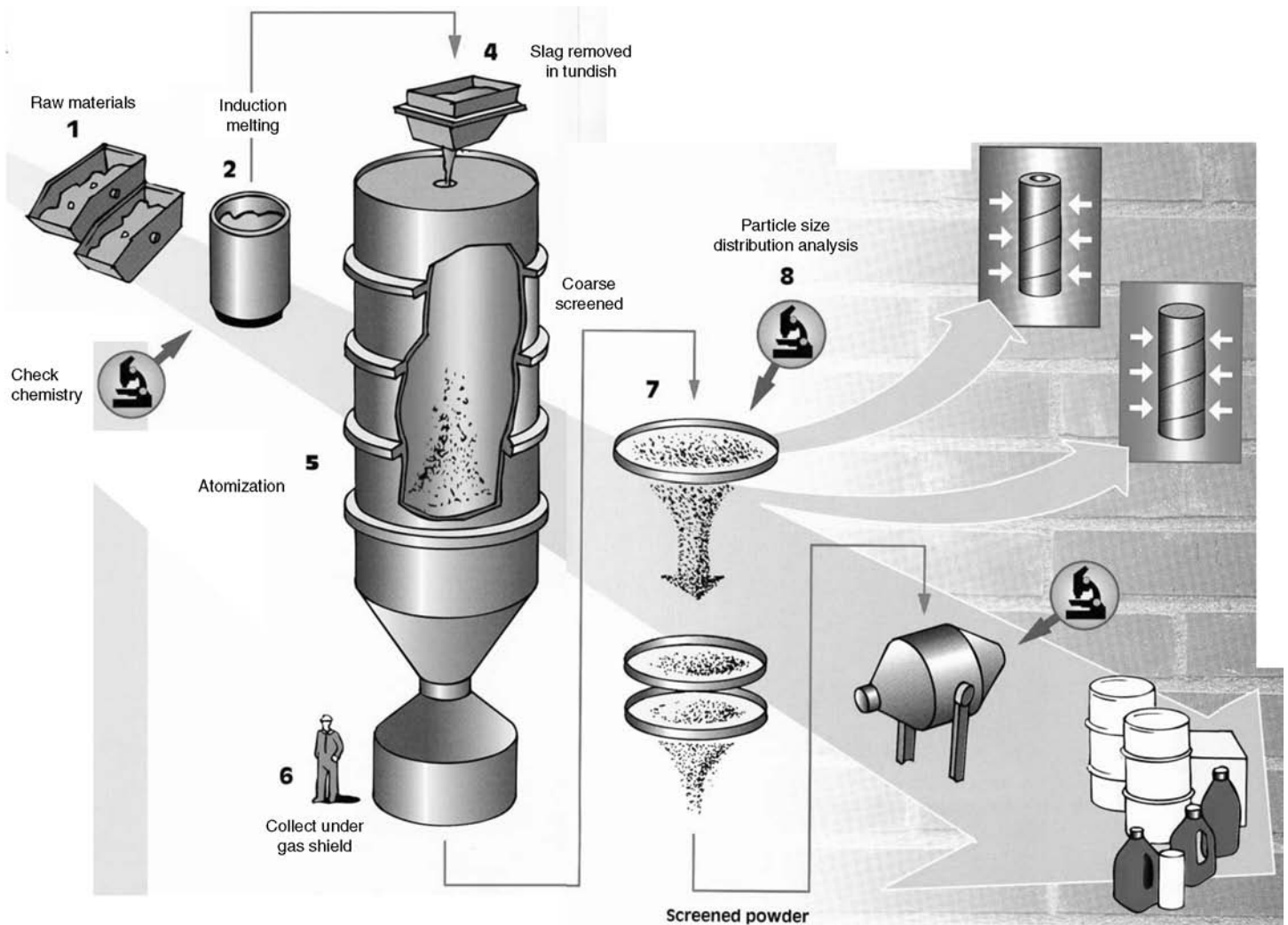


Fig. 6 Overall view of an atomization facility. Source: Courtesy of Anval, Inc., Rutherford, NJ

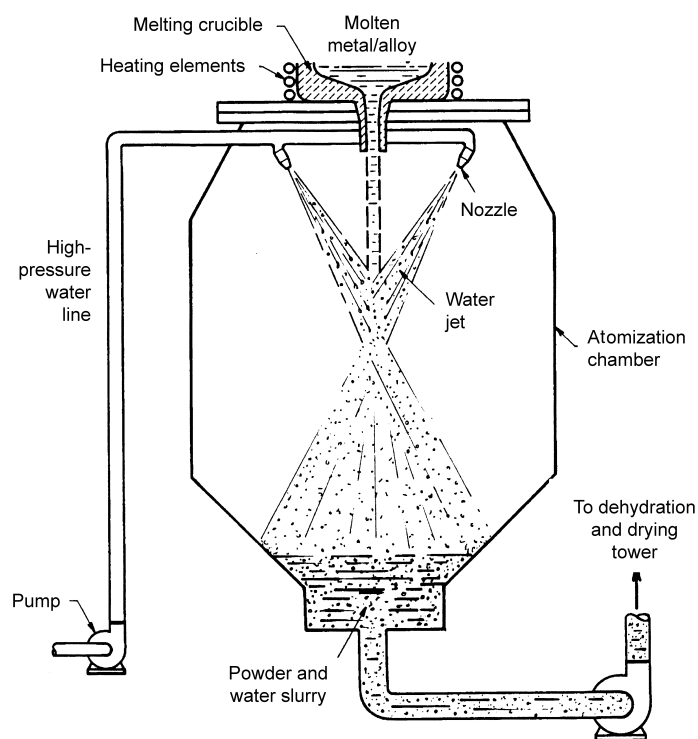


Fig. 7 Schematic of the water atomization process. Source: Ref 2

duction method for reactive materials such as titanium or titanium alloys and other reactive alloy systems. Gas-atomized products are typically spherical and have smaller-size particles than those produced by water atomization (typically in the range of 25 to 100 μm). Powders produced by this technique include cobalt-chromium-base alloys, nickel-chromium-base alloys, nickel-base alloys, copper-base alloys, zinc, aluminum, stainless steels (all from 38 to 150 μm in size), and nickel-base superalloys and (M)CrAlYs (in sizes less than 75 μm).

Comparison of Atomization Processes

Particle Size Distributions. The gas and water atomization processes can produce different particle size distributions through control of the atomization variables. For instance, Fig. 12 demonstrates how the atomization pressure enables production of coarse or fine particles by water and gas atomization processes, respectively. In addition, the particle size distribution can be altered, as shown in Fig. 13 for gas atomization, where the distribution changes from fine and monomodal to coarse and bimodal as the gas pressure is varied.

Operating Conditions. Many atomizer operating conditions are empirically based. The essential principle is that less coarse particles are obtained as the metallic stream is highly disrupted. Water atomization is performed at water pressures of 30 kPa to 65 MPa (4.4 psi to 9.4 ksi). The corresponding water flow rate may be near 90 kg/min (198 lb/min), corresponding to a water velocity of 40 to 150 m/s (130 to 490 ft/s). An important operating param-

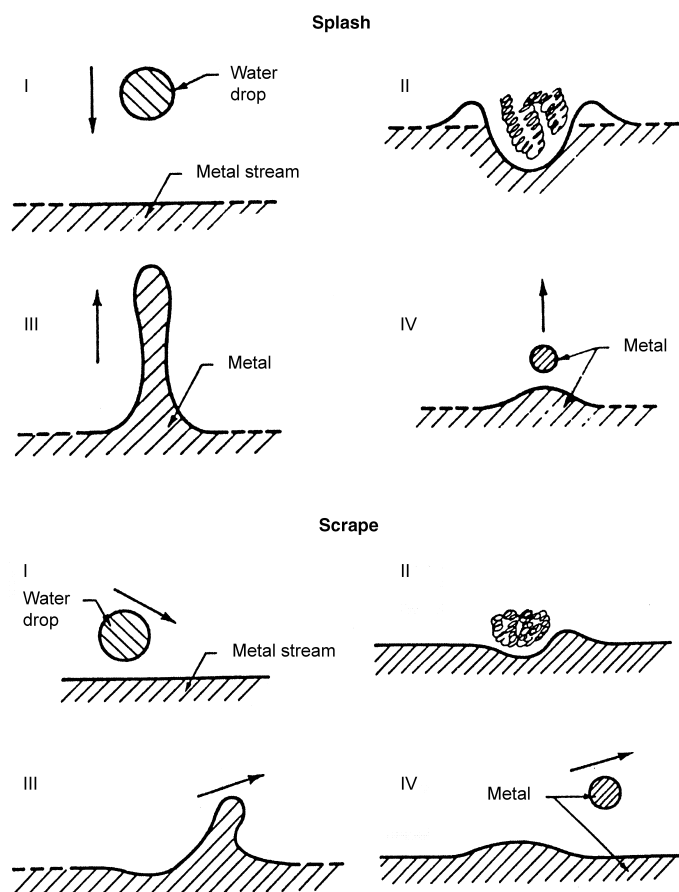


Fig. 8 "Splash" and "scrape" particle formation mechanisms for water atomization processes

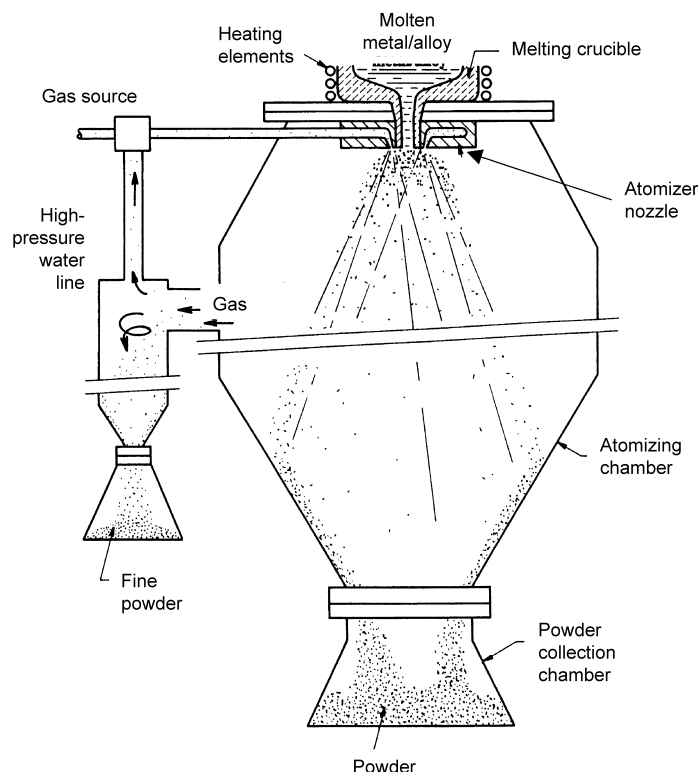


Fig. 9 Schematic of the gas atomization process. Source: Ref 2

eter is the water/metal ratio since there must be enough water to break the metal apart. The normal ratio is 4 to 10 L/kg (2.3 to 6 gal/lb) of metal, which provides sufficient cooling of the metal to avoid boiling and allows a range of metal flow rates to be accommodated. In gas atomization the typical gas usage rates are 0.5 to 2 m³/kg (39 to 155 ft³/lb) for nitrogen or air at pressures of 1.4 to 4.2 MPa (0.2 to 0.6 ksi). The gas velocity is 50 to 150 m/s (165 to 490 ft/s).

The prime advantages of gas atomization over water atomization are the enhancement of a spherical morphology and the ability to produce a clean structure; the lamellae of coatings subsequently produced from these powders exhibit oxide-free surfaces and low oxide contents and gas contamination. These two factors influence powder transport and flow characteristics, respectively, and the formation of oxide-free metallic bondcoats. The material structures formed via these techniques are typical of those produced by rapid solidification processing. Water atomization results in particles that are cooled at a rate of 10^4 to 10^6 °C/s,

whereas gas atomization cooling rates are an order of magnitude lower, i.e., 10^3 to 10^5 °C/s. Both processes rely on forced convective cooling in the liquid or gas, and the cooling rate is therefore dependent on the individual particle size. Thus, cooling rates can be as high as 10^7 °C/s for 10 µm particles that are cooled by high thermal conductivity gases such as helium. This range of cooling rates (from 10^3 to 10^7 °C/s) is within the same range for the TS process, so the coating crystal structure may be quite similar to that of the as-received powders. The crystal structure for milled and crushed powders is crystalline; these feedstock materials exhibit a marked structural change during TS since they melt and then are subjected to rapid solidification.

The melt size of each metal heat may vary from 20 to 500 kg (44 to 1100 lb). The ability to rapidly induction heat the melting crucible either outside or within the atomizer ensures high purity of the metal or alloy. The typical flow rate of the metal through the atomizing process is from 20 to 60 kg/min (44 to 132 lb/min); the total atomization time for the smaller specialized or laboratory runs may be less than 30 s. The metal runs directly from the ladle to the tundish of the atomizing unit. A gas atomizing unit that processes a 300 kg (135 lb) batch of metal is typically 4.5 m (14.8 ft) in height and 1.25 m (4.1 ft) in diameter. The two basic atomizer designs are termed as *confined* or *open* (or *closed* or *free-fall*, respectively). In a closed system the gas travels 1 to 10 mm (0.04 to 0.40 in.) before hitting the metal stream, whereas this distance is 30 to 150 mm (1.2 to 6 in.) for an open system. This distance greatly influences the manner in which the metal stream is fragmented.

Powder Morphology. Figure 14 illustrates the key morphological features of gas- and water-atomized powders; note that the gas-atomized materials are much more spherical (Fig. 14a). Porous coated powders formed by an agglomeration process are depicted in Fig. 14(c).

Chemical Techniques

Table 1 lists chemical powder preparation methods. Those most important for producing TS powders include:

- Sol-gel processing
- Spray drying and spray roasting
- Freeze drying
- Agglomeration and sintering

Sol-Gel

Sol-gel processing is a chemical engineering technique used to manufacture ceramic powders, especially oxides. The term *sol* refers to the initial solution of the chemical components from which the final powder is eventually derived. *Gel* describes the final product of the ceramic material. The method is based on the mixing of solutions that enable reactions for the formation of distinct particles. It is important to distinguish that the particles are not precipitated from solution; rather, the mechanism of particle production is based on colloidal science in which the particles are suspended in the liquid. Typical ceramic powders produced by this technique include chromia, alumina, and stabilized zirconia.

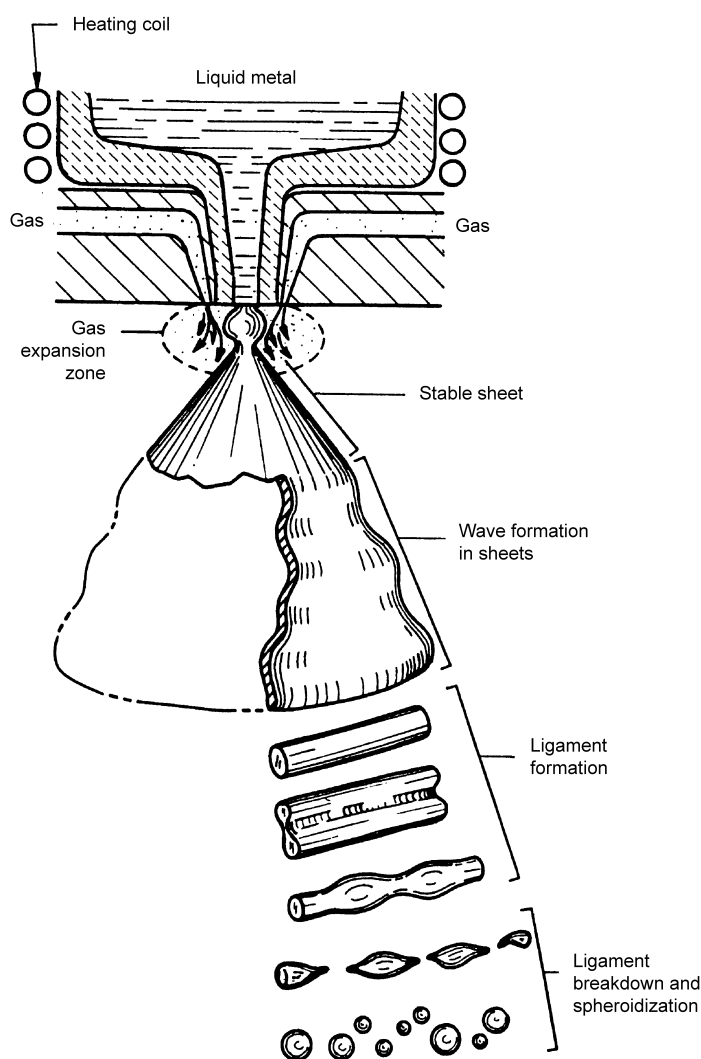


Fig. 10 Mechanisms of particulate formation in the gas atomization process. Source: Ref 2

The sol-gel technique and powders formed by this technique have the following attributes:

- Processing is performed at low temperatures, making it more energy efficient than high-temperature methods that may also require particle attrition.
- Powders of a predetermined size and morphology can be produced. The technique enables the production of high yields of a required size fraction. With reference to TS powders it is important to state that spherical powders are routinely produced, since this morphological attribute facilitates the manufacture of free-flowing feedstock.
- The powder composition can be closely controlled; for example, multicomponent powders can be produced. Also, the degree of particle aggregation can be used to control the pore structure of the final product.
- The process is performed by solution chemistry methods. The technique is quite efficient compared to powder blending. Another benefit is that there is no dust hazard, thus minimizing or completely eliminating pyrophoric, toxic, and environmental problems.

The steps that are followed to produce a powder via the sol-gel route are shown in Fig. 15, and are listed and discussed here.

Stage 1. The components are placed into solution. The liquid component is usually water or an alcohol. The solute is either an inorganic nitrate or chloride, or based on organometallic compounds. These components allow thorough mixing of the species on the atomic scale, explaining the extremely homogeneous compositions that are obtained. This solution is often referred to as the “precursor” since it is the basis of the subsequent steps that lead to the final powder.

Stage 2. The solution, if it is not already a sol, is converted to a sol. A sol is a colloidal dispersion (or a dispersed solid phase) that contains particles smaller than about 150 nm (0.15 μm , or 5.9×10^{-8} in.). The sol can be made stable by appropriate adjustment of the pH; otherwise, the particles will tend to grow into agglomerates that, in turn, may precipitate and thereby prohibit attainment of the unique powder chemistries.

Stage 3. The gelation step occurs next. This is essentially a step to remove most of the solvent (water or alcohol) so that a rigid body of well-defined chemistry is formed. The gel is still quite plastic or highly viscous. The key processing variables are pH, temperature, and time.

Stage 4. The gel is then shaped to the required morphology. This may be as spheres, fibers, or coatings. Thermal spray powders of spherical morphology are produced by a process called

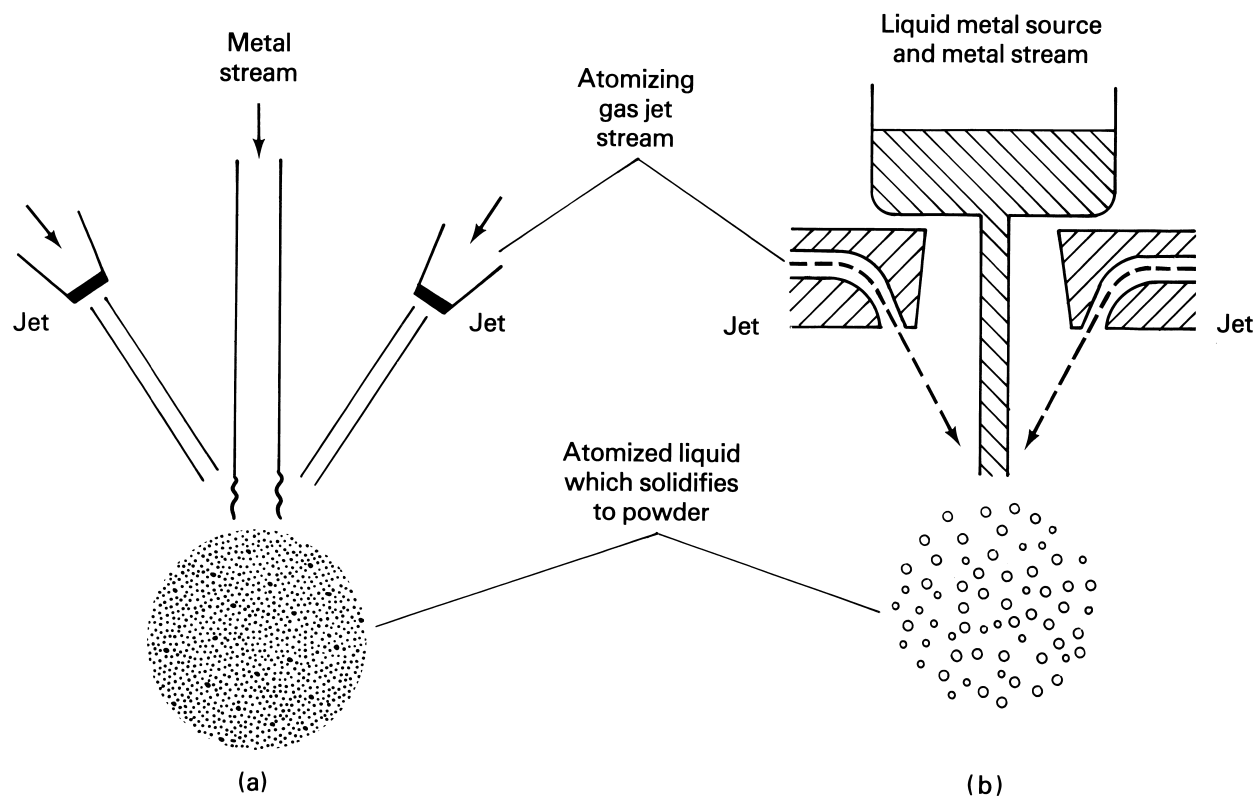


Fig. 11 Primary variables that control the gas atomization process. (a) Twin jet configuration. (b) Annular ring configuration

spray drying. These morphologies may also be produced by controlling the gelation stage in the conventional process.

Stage 5. The remaining solvent is removed and the gel is calcined (heated) to form the final powder. This final temperature is significantly lower than the temperatures of conventional process-

ing methods. For example, silica can be calcined at temperatures from 600 to 1000 °C (1110 to 1830 °F).

Sol Structure/Density. The sol structure can be either an aggregated or a dispersed structure so that varying degrees of particle alignment are obtained when conversion to a gel takes place. Figure 15 is a ball-model of the structures that may be established. The density of the product can be altered from low (i.e., an aggregated sol) to high (i.e., a nonaggregated, or dispersed, sol).

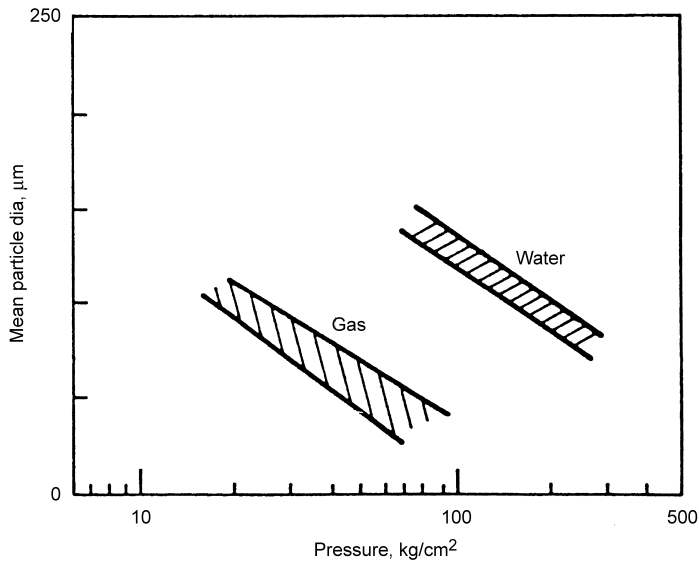


Fig. 12 Typical particle size ranges manufactured by the gas and water atomization processes

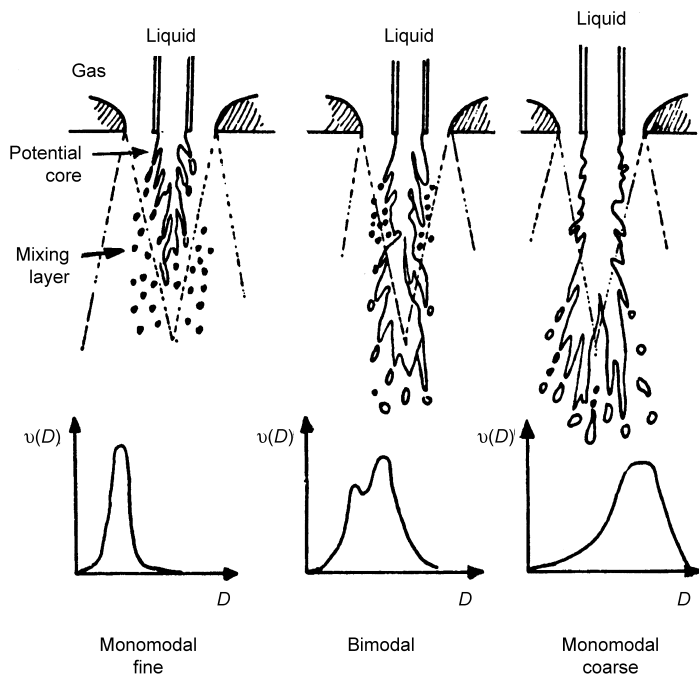


Fig. 13 Influence of fluidizing media variables on particle size distribution for gas atomization. $v(D)$, frequency of particle diameter

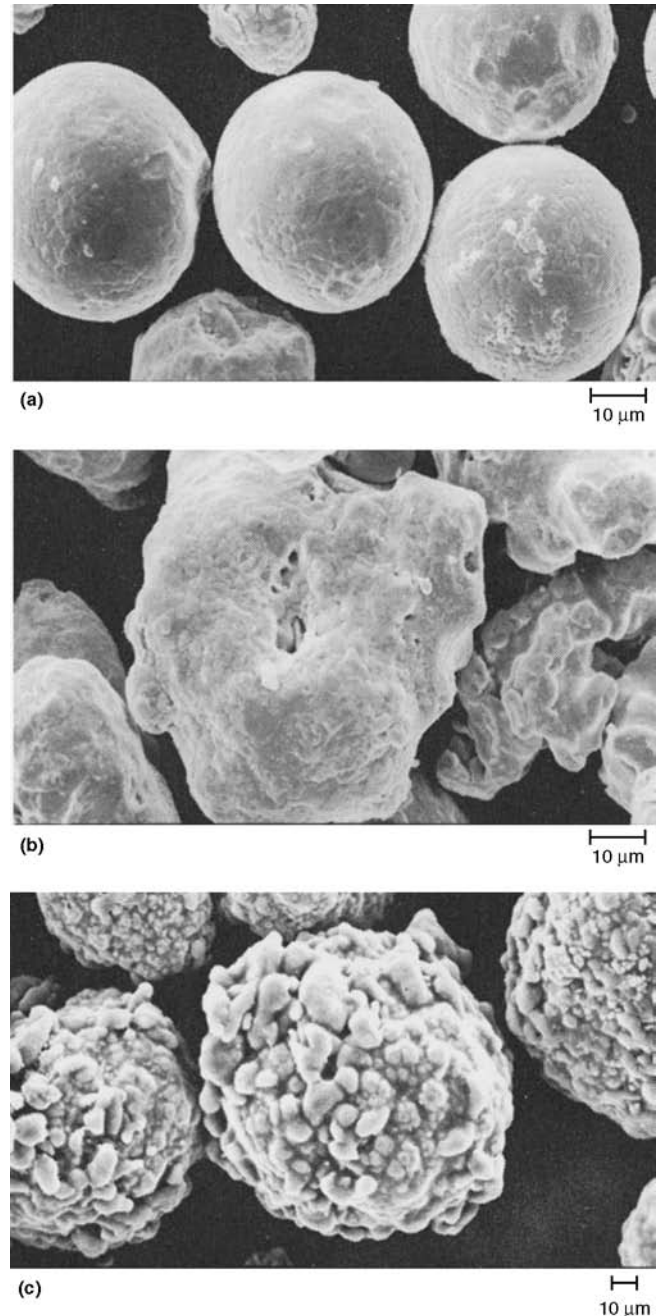


Fig. 14 Particle morphologies. (a) Gas atomization, 1000 \times . (b) Water atomization, 1000 \times . (c) Porous coated particles, 500 \times

Other Chemical Powder Production Methods

Spray Drying and Spray Roasting. In this process the sol is normally concentrated by partial evaporation of the solvent to form an aero-gel. The aero-gel is then fed into an enclosed chamber heated by hot air. The process chamber is similar to that used for atomization. The aero-gel is still quite fluid and forms a spray so that each droplet will, after evaporation, constitute the gel of the powder particles. The shrinkage of the individual droplets may be quite high—on the order of 50%—and particles in the size range of 5 to 250 μm may be produced. These gel particles can now be calcined to form the feedstock for TS. The hot air is usually at 400 $^{\circ}\text{C}$ (750 $^{\circ}\text{F}$) and the air leaving the spray drying device is at 200 $^{\circ}\text{C}$ (390 $^{\circ}\text{F}$), so the process is clearly not very energy efficient for small batches of material. Spray drying of powders is also referred to as spray *roasting*, as the process temperature may be quite high.

A spray drying facility can also be used to agglomerate fine powders, presently in the 1 to 10 μm range. The particles are mixed into a *slurry* that contains a binder. The slurry is then atomized at elevated temperatures and the various constituents form clusters of particles with the chemistry of the powders in the orig-

inal slurry. The powder size is controlled by altering the nozzle geometry and the atomizing pressure. This process is not a true sol-gel method (in the scientific sense) since the chemistry of the product is inhomogeneous. The method is attractive, however, because of its flexibility in utilizing raw materials that are generally inexpensive to produce free-flowing particles of a spherical morphology, as shown in Fig. 14(c).

Freeze drying also uses a sol or another type of emulsion that contains the various components of the powder alloy being manufactured. The droplets of material are rapidly frozen by stirring into hexane at $-30\text{ }^{\circ}\text{C}$ ($-86\text{ }^{\circ}\text{F}$). The material is then formed into solid phase that can be filtered from the system. The final step is to evaporate any solvent by causing it to sublime (i.e., a solid-to-gas transformation which bypasses the liquid state) at low pressures and ambient temperatures.

Agglomeration and Sintering. Agglomeration refers to the binding together of particles to form coherent bodies. Two methods are used: those that use a binder and those that rely on *sintering* operations. These methods also overlap with the sol-gel techniques described in the previous section.

The term sintering refers to the joining of particles through the combined actions of pressure and heating. A pressed material is heated to a temperature below its melting point, and binding between particles occurs due to chemical diffusion between the particles. The particles become joined to each other, and further comminution and sieving are necessary to obtain the required particle size distribution. Figure 16(a) shows coherently joined individual particles. There is also some residual porosity within the material (Fig. 16b). Porosity can be reduced by employing higher compaction pressures, higher sintering temperatures, and longer sintering times, but because the strength of the product is greatly increased, such changes may be counterproductive with regard to necessary milling operations. Many WC-Co materials are produced by these methods.

The process of binding particles together also enables the combining of different materials (e.g., alumina and titania, yttria and zirconia, etc.) and the recycling of fines from some other process. For example, particles produced via the sol-gel process may in turn be agglomerated. The binding material used is generally an organic base, such as polyvinyl alcohol or carboxy-methyl cellulose. The use of a binder is not mandatory since the fines can be

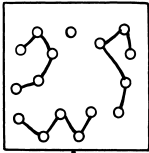
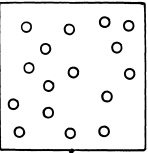
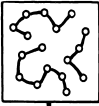
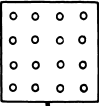


Powder attributes	Porous powder	Dense powder
Original dilute sol		
After drying, sol is concentrated		
After solvent is removed, gel is formed		
Change in relative volumes	Less dense	More dense

Fig. 15 Production steps for a sol-gel processing route, indicating the formation of variable densities during the process

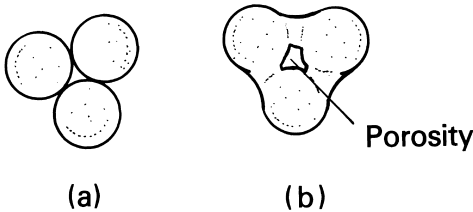


Fig. 16 Physical processes that occur during particle sintering. (a) Individual particles become coherently joined. (b) Residual porosity

consolidated by pressing and sintering; however, a further comminution and sieving procedure is always required for these materials. The agglomerated particles must be resieved regardless of whether any sintering is performed so that the new particle size distribution can be determined. The nature of the sieving operation is quite crucial since it may, and usually does, lead to a further breakdown of any loosely agglomerated particles and thus to a false representation of the as-received feedstock.

An agglomerated morphology is not spherical; it can be described as being globular in appearance, with many protuberances from the surface. Therefore, it might be expected that such powders would be difficult to feed. This is generally true, but the TS parameters for such materials have been optimized so these powders are viable from a technological viewpoint. A more important aspect is that the theoretical density of sintered powders is usually lower than powders obtained from the fused and crushed routes. Because there is an empirical relationship that relates low coating densities to low tensile adhesion strengths, the agglomerated powders (or spray-dried and sintered powders) may exhibit lower strengths. Such powders are still acceptable for their intended applications and may have the additional benefit of being lower-cost feedstock materials.

Material Production Techniques for Producing Unique Geometries or Compositions

Mechanical Cladding. Bondcoat materials that are based on nickel and aluminum are called *composites* and are produced by two main methods. One process is the same as the agglomeration techniques described earlier, but is referred to as *cladding* within the TS field. Particles of one component are coated onto a core of the other using a binder. For example, 5 wt% Al can be clad onto a nickel core, or 80 wt% Ni can be clad onto an aluminum core. The second type of Ni-Al composite materials is termed *prealloyed*. These are produced using water or gas atomization methods; for example, a 5Al-95Ni alloy is manufactured by this method.

Chemical Cladding. An important class of materials is used for high-temperature abrasible seals in gas turbines. These materials operate at 650 to 850 °C (1200 to 1560 °F) and consist, in some instances, of NiCrAl/bentonite composites. (Bentonite is a silica-based clay.) The feedstock material is a composite of a non-metallic core coated with a layer of metal. The bentonite core powder is suspended by mechanical agitation in a nickel amine sulfate solution. The nickel can be reduced and allowed to deposit onto the bentonite by bubbling hydrogen through the solution at 180 °C (355 °F) and 2.4 MPa (35 psi) in a high-pressure autoclave. Bentonite particles of 1 to 200 µm in size are used as cores, and the nickel coating thickness is typically greater than 2 µm. The nickel coating can be further alloyed with chromium and/or aluminum by pack diffusion. The average diameter of the core particles is about 85 µm and the alloy coating thickness is about 6 µm. The overall powder composition is typically 20 wt% bentonite, 5% Cr, 3% Al, and 72% Ni. The core material, which may also be graphite or a silicate, is abrasible when thermally sprayed and allows the rotating components to cut and form a close-clearance seal. The metal-

lic components of these coating systems have good high-temperature resistance and permit satisfactory bonded porous structures to be formed for special-purpose abrasible seals.

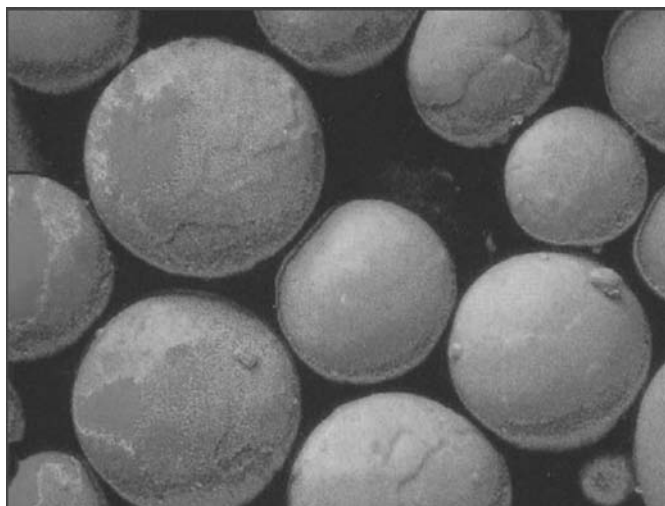
Plasma Fusion and Densification. A representative process for a plasma fusion method is the proprietary technique used to manufacture hollow-oxide spherical powders (HOSP). The HOSP process involves the TS processing of spray-dried materials. The powders are melted in a heat source to form spherical, hollow particles. The particles are collected and then classified by the usual processes. The technique is applied to ceramics such as alumina, zirconia, and chromia-base materials to form powders with good flow characteristics.

Manufacturers of such feedstocks claim that the good powder feeding properties of these materials, together with their chemical homogeneity and the ability of the HOSP process to melt high-melting-point materials within the TS stream, result in coatings superior to those obtained from powders produced from fused and crushed feedstocks. The basis of the higher-productivity argument is that hollow spheres allow more uniform melting behavior. In addition, caution should be observed since the bulk density of these materials is significantly lower than that of spray-dried, fused and crushed, and other “dense” feedstocks. Therefore, the powder feeding characteristics and plasma spray parameters for these different feedstocks should not be expected to be similar. The morphology of a plasma-fused powder is contrasted to that of conventional sol-gel derived powder in Fig. 17.

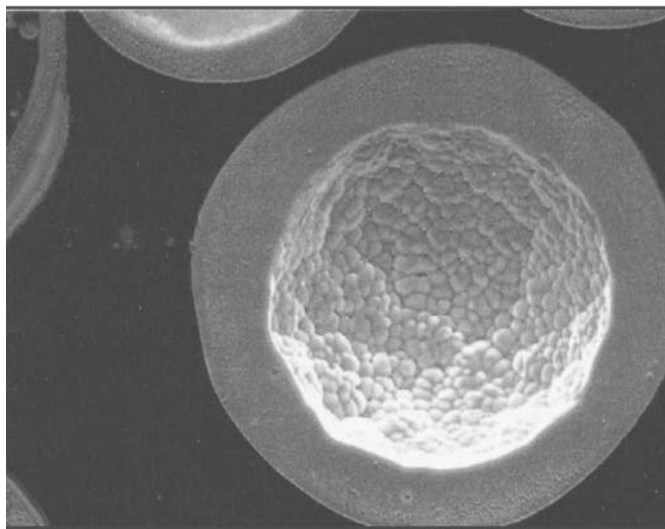
The plasma heat source can also be employed to “plasma densify” composites of material so that the individual constituents are in close contact. For example, some WC-Co powders are agglomerated, sintered, and then plasma-densified to increase their strength and density before spraying. Therefore, such materials are less likely to break up during their transport through the flame and often retain their initial chemistry within the coating.

Agglutination. A new laboratory technique for producing ceramic/metal composite powders is the agglutination process. This is a low-temperature process, on the order of 50 to 85 °C (120 to 185 °F). The ceramic cores are first tumbled in a binder phase of polyethylene glycol to form a uniform coating. The binder phase is about 4 wt% of the powder. The second step is to cool the mixture and break it up by conventional milling. The coated cores and fine metal powder (5 to 10 µm) are then charged into a tumbling furnace, where they combine to form a metal-coated core. The advantage of this process, other than the technical simplicity, is that it is very energy efficient and virtually unlimited with regard to the combinations of materials that can be formed into a composite.

Blends and Cermets. A final aspect of composite powder feedstock preparation is that of blended powders and codeposited powders. Blended powders are mechanical mixtures (or blends) of separate components; that is, the blended powder is fed through only one powder port. Care must be taken that the constituents of the powder blend have the ability to be sprayed with identical TS parameters; otherwise, no coherent deposit will form and the relative deposit efficiencies for each feedstock will be different. Thus, these powders require adjustment of the appropriate particle size



(a)



(b)

Fig. 17 Scanning electron micrographs of partially stabilized zirconia (PSZ). (a) Dense spherical PSZ. (b) Hollow particles of PSZ. Note: those particles are approximately 40–60 μm in diameter. Source: Courtesy of Saint-Gobain Ceramic Materials, Worcester, MA

distribution with respect to their weight loading in the composite coating. Thermal spray process jets are an excellent “classifying” medium and may result in significant segregation of blended powders having widely dissimilar particle sizes and/or densities, leading to inhomogeneous coatings.

Codeposited materials have very similar structures to blended materials but are formed from a dual powder-feeding arrangement. The powder ports may enter the TS jet at either the same location (if they require the same thermal trajectory) or at different locations (if they need different thermal treatments). One advantage of the dual-port system over the blended powder technique is that a continuous adjustment of the composite chemistry can be achieved. Such coatings are referred to as “graded.”

REFERENCES

1. A. Lawley, *Atomization*, Metal Powder Industries Federation, 1991
2. A. Bose, *Advances in Particulate Materials*, Butterworth-Heinemann, 1995

SELECTED REFERENCES

- A. Bose, *Advances in Particulate Materials*, Butterworth-Heinemann, 1995
- J.J. Dunkley, *Atomization, Powder Metal Technologies and Applications*, Vol 7, *ASM Handbook*, ASM International, 1998, p 35–52
- L.C. Klein, Sol-Gel Process, *Ceramics and Glasses*, Vol 4, *Engineered Materials Handbook*, ASM International, 1991, p 209–214
- C.C. Koch, Milling of Brittle and Ductile Materials, *Powder Metal Technologies and Applications*, Vol 7, *ASM Handbook*, ASM International, 1998, p 53–66
- A. Lawley, Preparation of Metal Powders, *Annual Review of Materials Science*, Vol 8, 1978
- Spray Drying and Granulation, *Powder Metal Technologies and Applications*, Vol 7, *ASM Handbook*, ASM International, 1998, p 91–96

Particle Characterization

Christopher C. Berndt, Stony Brook University

RELIABLE TECHNIQUES FOR PARTICLE CHARACTERIZATION and quality control testing are required to evaluate the physical properties of thermal spray (TS) powders. This article describes a variety of such procedures. Included are discussions of particle morphology, particle classification methods, representative sampling, Hall flow and density measurements, particle size measurements, the wipe test, and quality control procedures for ensuring proper powder feeding.

Particle Morphology and Porosity

Prior articles in this Section of the Handbook have explained that particle morphology not only is an important part of the powder feeding stage but can also be controlled. The range of particle morphologies that can be produced is shown in Fig. 1. Particle morphology is most commonly ascertained with either scanning electron or optical (light) microscopy. An electron microscope can

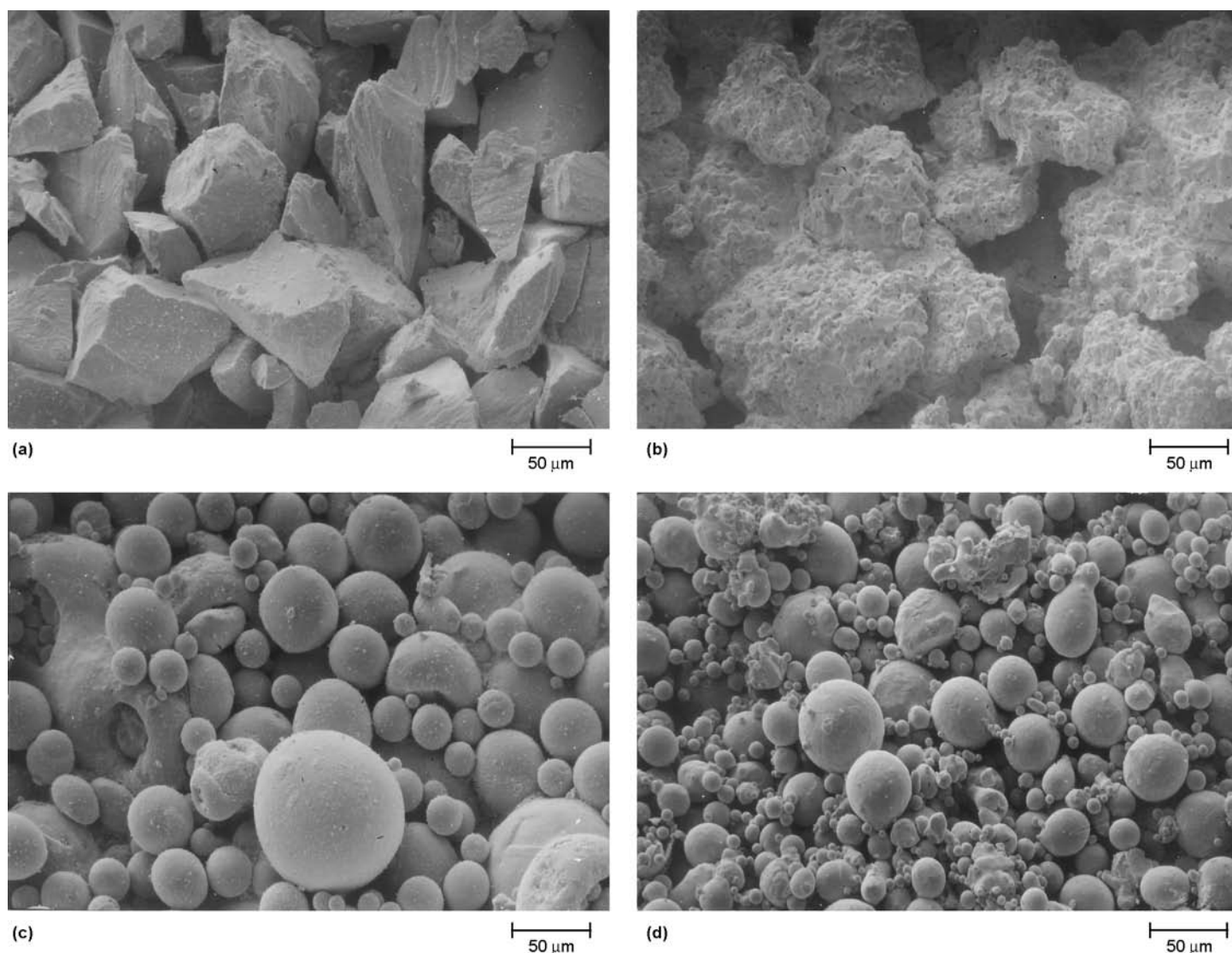


Fig. 1 Scanning electron micrographs of different TS powders. (a) 250 °C (480 °F) fused and crushed yttria-stabilized zirconia (YSZ). (b) Spray-dried YSZ. (c) Plasma-fused YSZ. (d) Gas-atomized Ni-Al. (e) Water-atomized Ni-Al. (f) 5 wt% Al clad onto nickel. (g) 80 wt% Ni clad onto aluminum. (h) Agglomerated materials

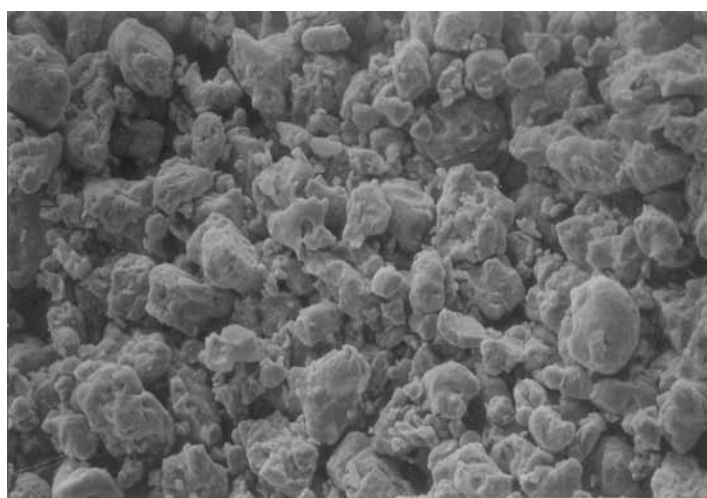
also check the chemical analysis of the material if equipped with an analytical facility such as energy-dispersive spectroscopy (EDS or EDAX).

Particle shape is the most important indication of the material processing methods since particle surface textures have quite different characteristics, depending on the production method. These morphological features also allow qualitative ascertainment of flow capability, if it can be assumed that perfectly flowing material would be monosized and have smooth surfaces.

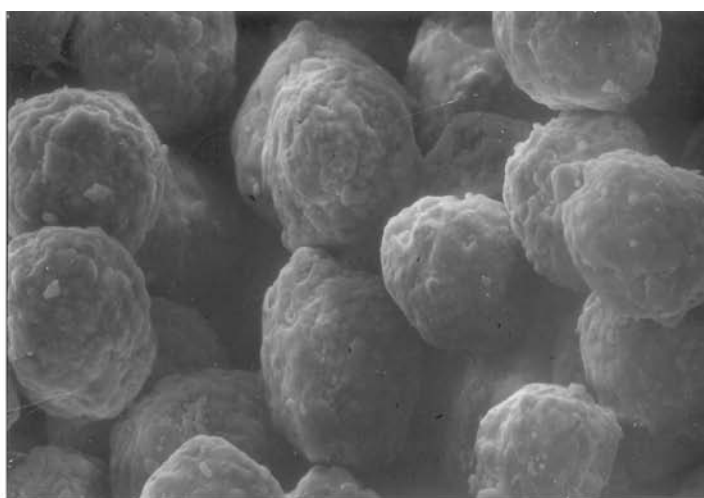
Spray-dried, sol-gel manufactured composites and agglomerated and sintered feedstock materials have characteristic globular features on their surfaces. The scale of these features is typically less than about 5 μm ; however, they do not disrupt the overall spherical nature of individual particles, and thus the flow charac-

teristics of these materials are good. Fused and crushed materials are clearly distinguished by smooth fracture surfaces indicative of their manufacturing method. Their shape is not spherical, but very irregular. These powders are quite dense and are sensitive to flow characteristics due to their nonideal geometry.

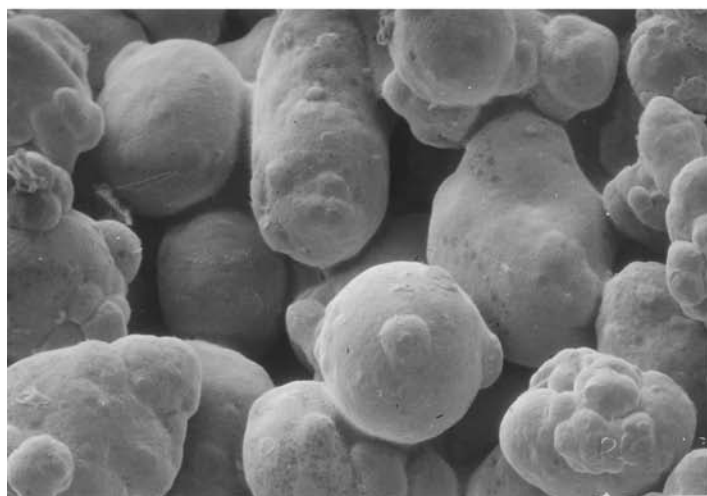
Individual feedstock particles may also exhibit very fine particles (less than 1 μm) on their surfaces. These fines are sometimes referred to as "satellites" and indicate either strong electrostatic forces within the body of the powder or moisture contamination. The overall result is that such particles may be indicative of either poor powder classification procedures (as described in the next section of this article) or a potential powder feeding problem. A wide range of particle sizes will result in irregular deposition due to inconsistent particle trajectories.



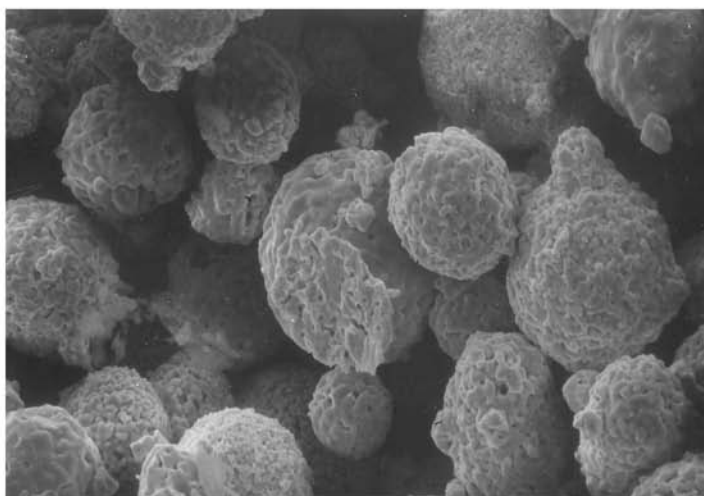
(e)

50 μm 

(f)

50 μm 

(g)

50 μm 

(h)

50 μm

Fig. 1 (continued) Scanning electron micrographs of different TS powders. (a) 250 °C (480 °F) fused and crushed yttria-stabilized zirconia (YSZ). (b) Spray-dried YSZ. (c) Plasma-fused YSZ. (d) Gas-atomized Ni-Al. (e) Water-atomized Ni-Al. (f) 5 wt% Al clad onto nickel. (g) 80 wt% Ni clad onto aluminum. (h) Agglomerated materials

Figure 2 compares the external appearance of powders with their internal structure. The micrographs on the left-hand side of Fig. 2 were obtained by embedding the feedstocks into an epoxy mount and then carrying out conventional metallographic procedures. It is apparent that the internal porosity, and hence the bulk density, of each material is quite different and depends on the manufacturing method.

Particle Classification Methods

Particle size distribution is an important part of powder manufacture. This aspect of powder technology is often referred to as *classification*, since powders are broken up into various groupings.

Sieving is the most common method of separating powders

into their size fractions. Sieving can be used either as a diagnostic method to determine the size distribution of a powder or as a manufacturing method to create powders of a specific size distribution.

As shown in Fig. 3(a), the sieve with the largest holes (or largest mesh opening) is placed on the top of a sieve stack. Figure 3(b) demonstrates a common variation of the sieving operation termed “scalping,” in which large particles are removed from a powder by passing it through just one sieve. The order of the sieves in the sieve stack should follow the ASTM specification: The mesh openings of two consecutive sieves must have a 1.414 (the square root of 2) ratio. Figure 4 shows other geometries for the industrial sieving of powders. In Fig. 4(a), a scalping operation is performed with a vibratory sieve, and Fig. 4(b) shows an oscillatory arrangement that uses a nest of three sieves to prepare various particle fractions (or “cuts”) of the input feedstock.

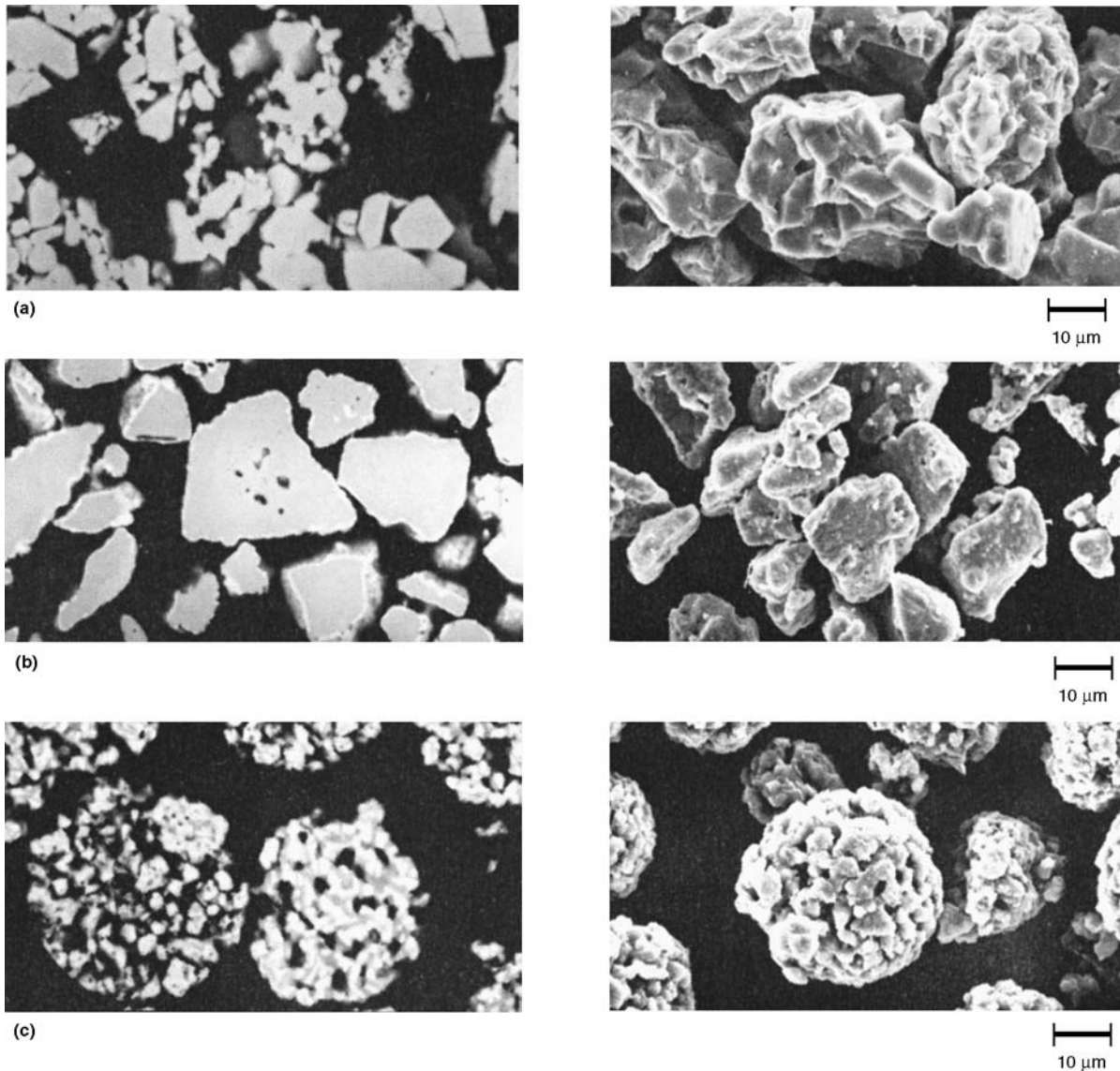


Fig. 2 Particle cross sections (left-hand side) and scanning electron micrographs of particle surfaces (right-hand side) of (a) a sintered powder, (b) an agglomerated and sintered powder, and (c) a dense coated powder. Source: Courtesy of H.C. Starck, Inc., Newton, MA

Table 1 lists the standard sieves generally used for TS powders. Note that the size of the mesh openings *decreases* as the sieve number (also called the “mesh”) increases. This reciprocal relationship can be understood if it is realized that the mesh number is related to the number of divisions per inch; i.e., more divisions per inch translates into smaller openings. There is also a physical limit for mesh construction since the wires used in their fabrication have a finite size. Therefore, fine particles ($<20\text{ }\mu\text{m}$) do not have an equivalent mesh size since they are sorted by either (1) electroformed sieves or (2) air classification methods.

A typical sieve stack might classify particles according to the following six micron ranges: +300 (oversize), -300 +150, -150 +106, -106 +74, -74 +45, -45 (fines or undersize). The particle fractions (or cuts) are, for example, stated as “-300, +150,” etc.,

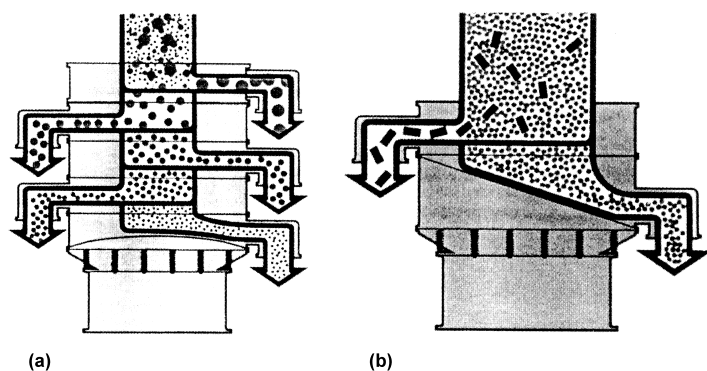


Fig. 3 Particle classification methods. (a) Sieving. (b) Scalping

which indicates that the powder passed through the (300 μm) mesh but was retained by the 150 μm mesh sieve. Powder is fed into the top sieve of the stack and the entire assembly is vibrated or shaken (Fig. 3 and 4). The particle size distribution is determined by weighing the amounts of powder in each sieve. The result is usually stated as a percent weight fraction, although some scientific studies find that number fractions are more useful. Some common particle cuts for a variety of processes and material classifications are listed in Table 2.

Air classification, another common method, employs cyclones (Fig. 5). The operating principle of a cyclone relies on the momentum discrimination of particles as they are forced to follow a circular trajectory. Heavier particles will be more inclined to settle out and therefore be separated from the fluidizing gas. Control of the gas pressure and dimensions of the cyclone allows various sized particles to be drawn off.

Particle Size Distribution Plots. A particle size distribution can be graphically represented as a cumulative plot (Fig. 6a) or a frequency plot (Fig. 6b). These graphs are essentially identical since they can be derived from each other; the summation of the individual frequencies at each size enables the cumulative plot to be developed. Here are the key features conveyed from particle-size distribution data:

- The most commonly occurring particle size (i.e., the modal value) is indicated by the peak in the frequency plot. The mean value can be determined from the diameter at the 50 wt% value of the cumulative graph.
- The sharpness of the plot gives a qualitative indication of the spread in particle size. A sharp peak (e.g., powder A in Fig. 6b) indicates that the particles are of similar size, whereas a flat or broad curve (powder B in Fig. 6a) indicates that the particle sizes are spread over a wide range of values.

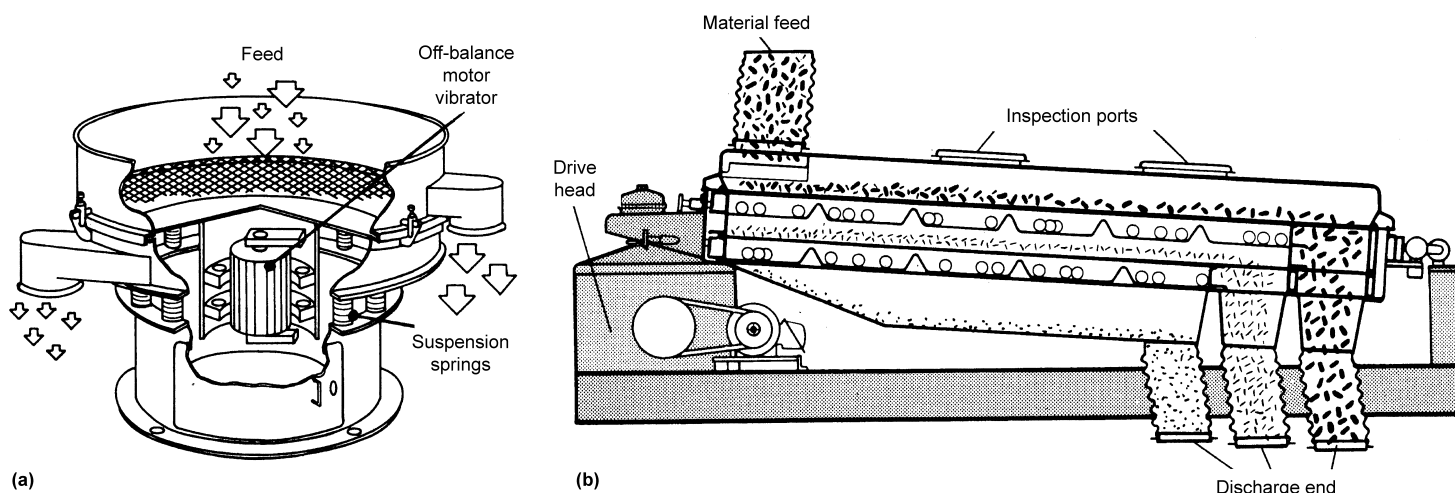


Fig. 4 Sieving of TS powder. (a) Vibratory sieve (1400 rpm). (b) Oscillatory sieve (200 rpm)

- The maximum and minimum values in measurable diameters allow the particle size range to be determined.
- It is important to look for a distribution that does not fluctuate, because that indicates a bimodal distribution of particle sizes. Such powders (e.g., powder B in Fig. 6b) do not feed reliably.

Difficulties Associated with Sieving. Because sieves are prone to abuse and general misuse, a major problem involves their maintenance and overall upkeep. The sieve wires may deform, rupture, or corrode, resulting in changes in the particle sieving process that render it unreliable and useless. Individual particles may also become embedded in the sieve grids, which can decrease sieving efficiency (since the total sieve area will be reduced) and may also contaminate any powder batches that are passed through the same set of sieves.

Table 1 Relationship between sieve (or mesh) number and mesh opening size

Sieve No.	Mesh opening	
	μm	in.
100	149	0.0059
120	125	0.0049
140	105	0.0041
170	88	0.0035
200	74	0.0029
230	62	0.0024
270	53	0.0021
325	44	0.0017
400	37	0.0015
500	31	0.0014

Table 2 Common particle cuts for a variety of TS processes and materials

Process	Particle cuts, μm		
	Metals and alloys	Carbides	Oxides
Flame spraying	75/20	90/45	...
Atmospheric plasma spraying	53/20	45/10	
	90/45	90/45	45/10
	45/10	45/10	45/10
Jet Kote		25/10	25/10
	45/10	45/22	...
Diamond Jet hybrid	45/15	45/15	
	38/10	38/10	15/5
JP-5000	38/15	38/15	12/2
	45/22	45/15	...
Top-Gun(a)	45/15	45/15	15/5
	45/10	45/10	12/2
	38/10	38/10	

(a) Not currently available. Source: H.C. Starck, Inc., Newton, MA

Wet Versus Dry Sieving. Sieving can be performed under either wet or dry conditions. Dry sieving is usually preferred for materials that are sensitive to corrosion or for materials that may tend to reaggregate in the sieving media. The advantage of wet sieving is that high throughputs can be attained under high-pressure conditions. Also, in many cases, the powder may already be in solution, making wet sieving a convenient and logical process for particle classification. A wet processing stage is usually encountered during chemical production processes such as precipitation and sol-gel methods. In such cases, wet classification offers the added benefit of washing away any residual reactants from the powder. Very slight milling will separate any agglomerated particles, with minimum particle breakup. Wet sieving is the preferred classification method when there is concern over environmental or toxicity issues.

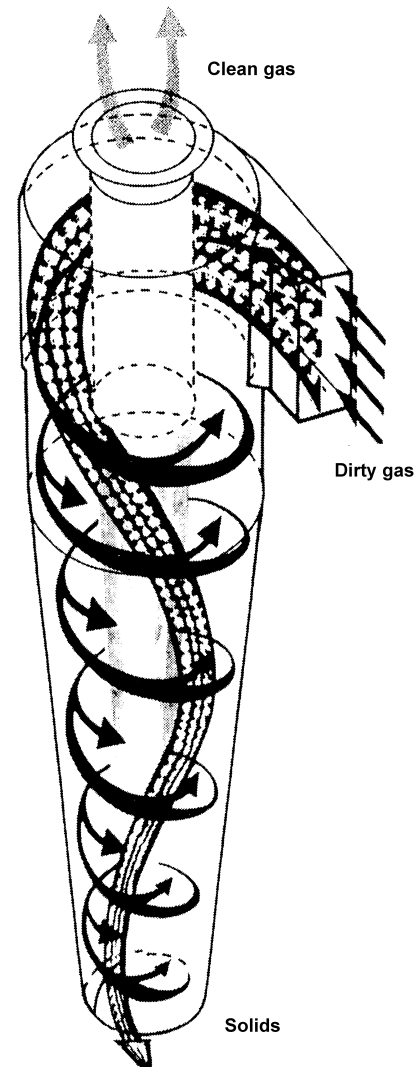


Fig. 5 Schematic of a cyclone used for air classification

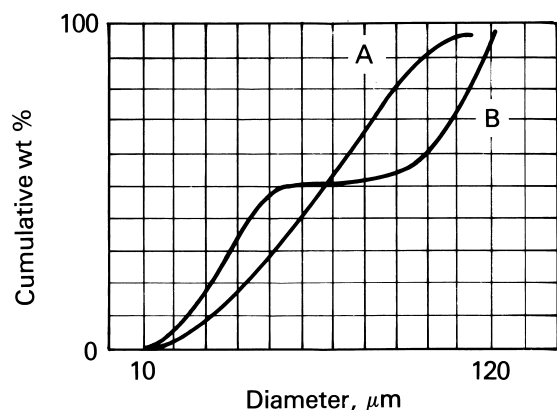
Quality Control and Feedstock Material Specification

Material specifications often establish utility. Successfully comparing materials requires a thorough knowledge of the appropriate standards and testing procedures. Another important factor is the TS process, which also influences coating quality.

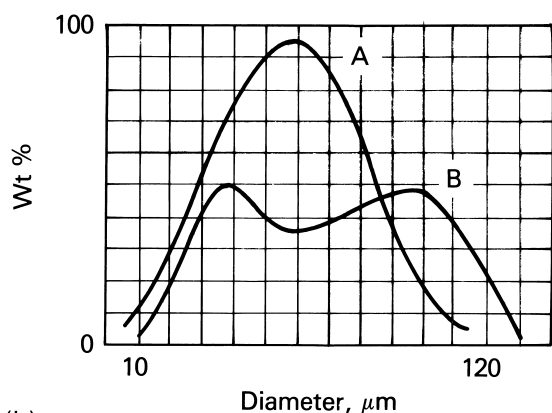
Representative Sampling. It is important to obtain a powder sample that is statistically representative of the entire powder batch. A sampling “thief,” as shown in Fig. 7, allows a small portion of the bulk powder to be removed from the container. The thief consists of two concentric cylinders that can be twisted relative to each other, thereby capturing powder from specific locations within a container. Samples thus obtained from various positions of the lot are then combined (Fig. 8), blended, and divided (Fig. 9) so that a fair sample is acquired for subsequent analyses and physical measurements. After a thorough analysis it may be determined that powder lots can be combined by a blending operation, such as that shown in Fig. 10. (Note that this is a macroscopic process of blending hundreds of kilograms of powder,

while the blending operation described in Fig. 8 involves only several kilograms.)

Hall Flow and Density Measurements. Quality control tests involve powder manufacture, particle size, powder porosity, material chemistry, Hall flow rate, spray tables, safety, and handling characteristics. The Hall flow test, as described by ASTM Standard B 213, is a simple means of measuring particle flowability. It uses an approved cone into which a standard 25 mL quantity of material is placed and gravity fed through an orifice at the bottom (Fig. 11). A low Hall flow value (e.g., <30 s) indicates that the powder has good flow characteristics. However, it is well known that powders with apparently poor Hall flow values can be reliably fed into TS torches; therefore, this property must be treated with caution.



(a)



(b)

Fig. 6 Particle classification curves. (a) Cumulative plot. (b) Frequency plot

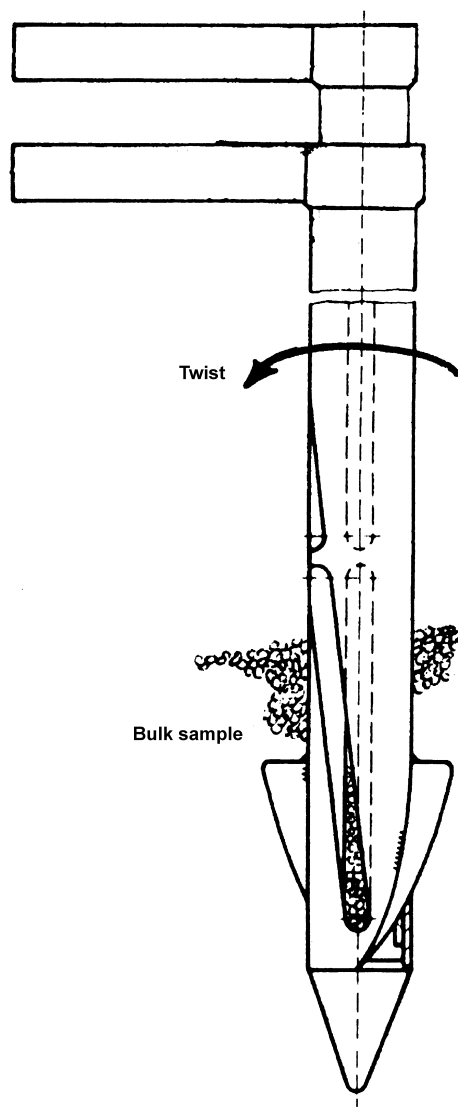


Fig. 7 Sampling thief used to obtain representative samples from a powder batch

The Hall flowmeter can also be used to measure powder density. Definitions for various terms that incorporate the word *density* include:

- **True density:** The ratio of the mass to the volume occupied by the mass. The contribution to the volume made by pores must be neglected. If the powder has no porosity, then the true density can be measured by displacement of any fluid in which the solid remains inert. However, many solid particles contain pores, cracks, or crevices that would cause errors in measurement. In such instances, a gas can be used as the displacement fluid.
- **Apparent density:** When a fluid displaced by a powder does not penetrate all of the pores, the measured density will be less than the true density. When densities are determined by liquid displacement, an apparent density is obtained. Because various liquids have different capacities to penetrate small pores, apparent density measurements can depend on the particular liquid used.
- **Bulk density:** The volume occupied by the solid plus the volume of voids when divided into the powder mass. When a powder is poured into a graduated container, the bulk density is the mass divided by the volume of the powder bed.
- **Tap density:** Another form of bulk density obtained by tapping or vibrating the container in a specified manner to achieve more efficient particle packing. The tap density is usually greater than the bulk density.
- **Effective density:** A particle may contain embedded foreign bodies that can alter its density. Blind pores that are totally encapsulated by the particle will also effectively reduce particle density. In this case, the measured quantity is the effective density.

Table 3 presents an example of feedstock measurements for thermal barrier coating (TBC) materials. In this table, 15 lots of TBCs are contrasted with respect to their Hall flow and apparent density. The primary conclusion is that there is no clear, unam-

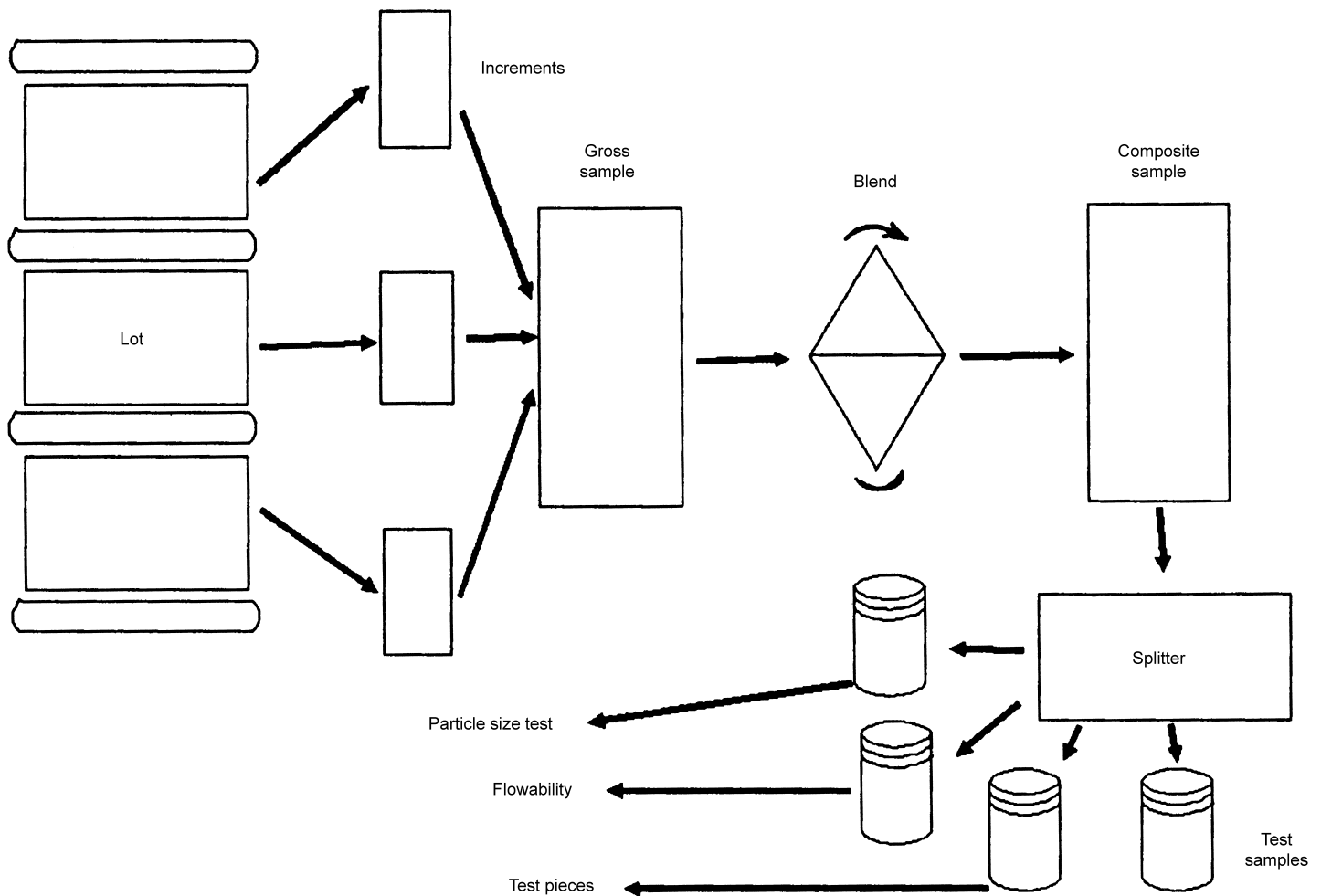


Fig. 8 Flow chart showing the procedures necessary to obtain a representative sample from a larger powder lot

biguous correlation between these physical properties, because the powder characteristics are controlled by manufacturing method: hollow-oxide spherical powder (HOSP, plasma fused), S/D (spray dried), S/D-S (spray dried and sintered), F/C (fused and crushed), and the sol-gel method.

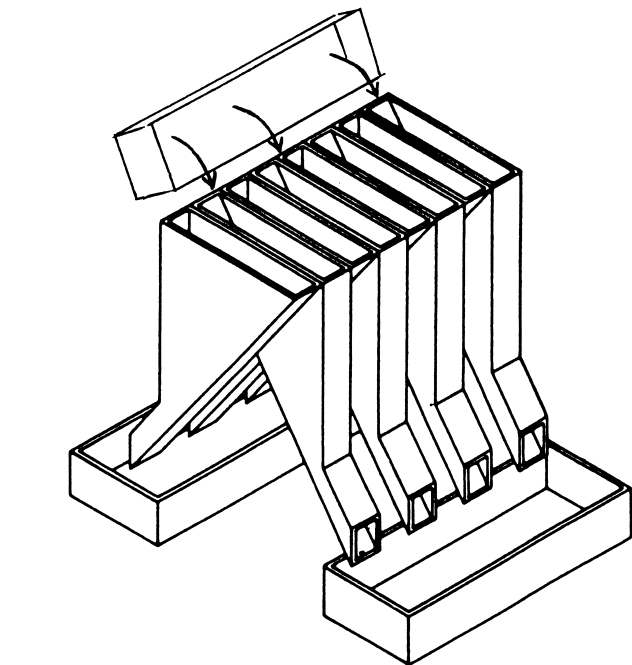


Fig. 9 Powder divider used to obtain many samples from one powder lot

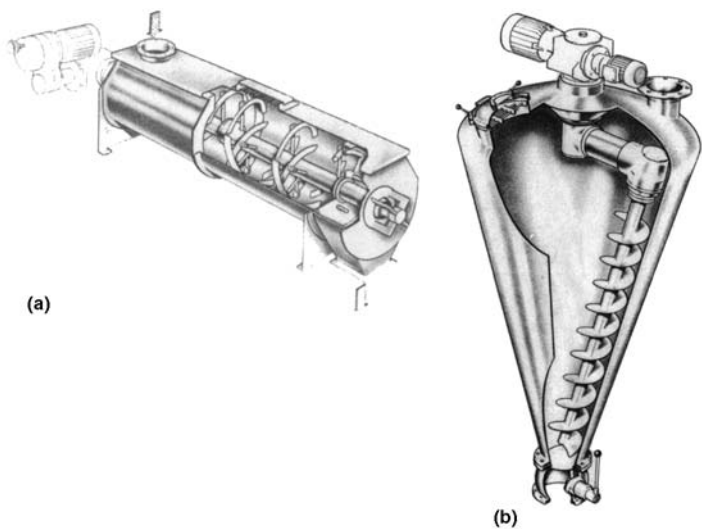


Fig. 10 Blenders used to combine, mix, and homogenize powders. (a) Ribbon blender. (b) Processing screw blender

Existing standards or potential standards for powder quality control should be implemented with respect to the intended use of the coating/substrate system. It is important that a coating not only be subject to some specific standard but that the standard be relevant to the particular feedstock material as well. The issue of which standards are appropriate for the TS industry has not been completely resolved.

Particle Size Measurement. There are two major methods of measuring particle size (other than the sieving technique described



Fig. 11 Hall flow apparatus used to measure feedstock flowability

Table 3 Hall flow and apparent densities of 15 lots of ceramic feedstocks

Material(a)	Hall flow, s	Apparent density, g/cm ³
8YSZ-HOSP	77.9	2.27
20YSZ-S/D	47.3	1.52
24CSZ-HOSP	34.1	2.40
CaTiO ₅	117.4	1.05
Mullite	71.6	1.16
8YSZ-S/D	52.2	1.44
8YSZ-S/D-S	(b)	1.10
8YSZ-F/C	45.1	2.55
8YSZ/sol-gel	39.2	1.72
8YSZ-S/D-S	(a)	1.84
8YSZ-S/D-S	40.3	2.00
8YSZ-HOSP	51.3	2.27
8YSZ-HOSP	81.7	2.26
8YSZ-S/D-S	(b)	2.26
8YSZ-S/D-S	46.3	1.76

(a) See text for explanation of abbreviations. (b) Material did not flow.

earlier): an x-ray method and a laser-scattering technique. However, the two methods have been found to produce quite different particle size distribution results for batches of the same material. There are technical reasons for this discrepancy, but it is disturbing that particle size depends on the measuring technique used. Because the TS applicator needs this data in order to optimize spraying parameters, the measuring technique for the mean particle size or particle size distribution should always be referenced so that the spraying parameters can be adjusted accordingly.

Many powder manufacturers and quality control establishments have shifted toward the laser-scattering technique. In this method, a small (~1 g) representative powder sample is fluidized in either air or water media. This fluid is then passed through a glass sample cell that is subjected to a beam of laser radiation. The laser beam diffracts off the feedstock particles contained within the fluid. The scattered laser beam is then measured via an array of detectors placed around the sample cell. The intensity of these responses can be converted into a direct measure of particle sizes and their relative proportions. Prime advantages of this equipment include simple maintenance and operation, and quick results generated within several minutes. However, the results must always be considered with care, because the scientific method employed to calculate the particle size assumes that the particles are perfect spheres.

For further details on particle size measurement, see the article "Powder Testing and Characterization" in this Handbook.

Variations in TS Deposition Properties. Table 4 presents a data compilation for some common TS ceramics, including chromium oxide (Cr_2O_3), aluminum oxide (Al_2O_3), and aluminum oxide combined with titanium dioxide ($\text{Al}_2\text{O}_3\text{-TiO}_2$). This data set shows the wide range of physical characteristics and properties of these materials and thus the corresponding variability in deposition properties in terms of deposit efficiency (DE), surface roughness, and hardness. There is, surprisingly, no unambiguous sci-

ence that has been able to correlate such powder specifications to coating properties.

Wipe Test. A powder-related quality control issue concerns the examination of deposits. A simple method for determining whether the material or TS parameters are suitable is the "wiper blade" test, also referred to as the "swipe test." This involves passing a series of glass microscope slides (or metallic substrates) in front of the TS torch during a trial feedstock spray operation. A series of glass slides usually are tested simultaneously by offsetting them on a rotor-type device so that a range of standoff distances can be examined in one TS run. The degree of particle melting and the optimum TS deposition parameters can be quickly ascertained by optical microscopy. The best feedstock and related spray conditions will be obtained when a well-formed, coherent splot can be produced.

Powder-Feeding Difficulties and General Discussion. Problems related to powder feeding have been discussed in the article "Feedstock Material Considerations" earlier in this Section of the Handbook. There are three common observations that imply when a quality control procedure is necessary:

- The powder has irregular flow, seemingly "turning on and off."
- The powder can only be fed at high carrier gas flow rates. These flow rates are high enough to carry the powder straight through the thermal source.
- The deposit efficiency (DE) is low.

Possible operator solutions to these problems include (a) adjusting the powder port and delivery tube sizes, (b) drying the powder in an oven, and/or (c) homogenizing the powder by rotating the powder container for a few minutes. If the powder-feed problem persists, then the TS equipment (particularly the powder feeder) may require maintenance, or modification of the powder specifications may be necessary.

Table 4 Compilation of TS characteristics and physical properties for chromia- and alumina-base ceramics

Material	Ratio	Type of powder	DE, %	Roughness R_a , μm	Hardness, $\text{HV}_{0.2}$	Apparent density, g/cm^3	Microtrac mean diameter, μm	Primary grain size diameter, μm
Cr_2O_3	n/a	Agglomerated and sintered	53	2.04	1287	1.8	26	5
	n/a	Fused	53	1.85	1156	2.1	22	20
Al_2O_3	n/a	Fused	50	3.75	1060	1.8	36	30
$\text{Al}_2\text{O}_3\text{-TiO}_2$	97/3	Fused	61	3.22	1069	1.7	38	30
	87/13	Blended	74	3.15	949	1.8	35	30
	87/13	Agglomerated and sintered	78	3.56	934	1.5	36	5
	87/13	Agglomerated	71	3.09	1012	1.1	...	3
	70/30	Agglomerated and sintered	75	3.90	887	1.4	40	5
	60/40	Blended	71	3.43	813	1.8	35	30
	60/40	Agglomerated and sintered	77	2.70	702	1.0	...	3
	60/40	Agglomerated and sintered	65	4.33	762	1.4	42	5
	30/70	Agglomerated and sintered	68	3.77	704	1.4	43	5
	n/a	Agglomerated and sintered	77	3.22	694	1.1	36	5
Al_2TiO_5	n/a	Fused	63	4.31	807	2.0	33	25

Note that the deposit efficiency (DE) depends on many variables, including the process and equipment used. The Microtrac method relies on the principle of laser-scattering. Source: H.C. Starck, Inc., Newton, MA

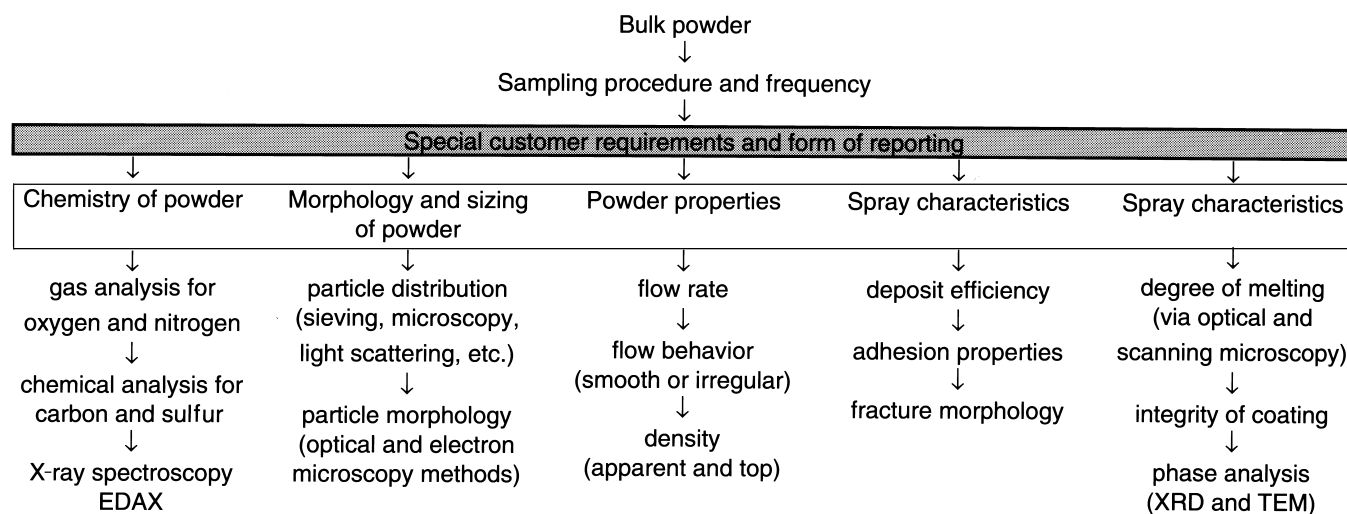


Fig. 12 Quality control procedures for powders

Powder testing and associated quality control must be ongoing. A suggested schedule is shown in Fig. 12, which emphasizes that it is essential to test coatings as well as to specify only high-quality powders. Very few of the determinations have standards that are followed throughout the TS community. Often any standards or specifications are supplied by the consumer of the feedstock, and different consumers may have different requirements for exactly the same powder under different applications. These lack of standards issues need to be resolved.

Figure 13 shows an experimental approach to resolving powder-feeding difficulties in a cost effective manner. The concept is to inject a sol-gel directly into the heat source and then allow thermal interactions of liquid evaporation, precursor condensation, and eventual in-flight particle formation and consolidation during transport toward the substrate. This approach, although not commercially available at present, is attracting much interest.

SELECTED REFERENCES

- M.B. Beardsley, Thick Thermal Barrier Coatings for Diesel Engines, *JTST*, Vol 6 (No. 2), 1997, p 181–186
- C. Bernhardt, *Particle Size Analysis*, Chapman & Hall, 1994
- J. Karthikeyan, C.C. Berndt, S. Reddy, J.-Y. Wang, A.H. King, and H. Herman, Nanomaterial Deposits Formed by DC Plasma Spraying of Liquid Feedstocks, *J. Am. Ceram. Soc.*, Vol 81 (No. 1), 1998, p 121–128
- S. Lowell and J.E. Shields, *Powder Surface Area and Porosity*, Chapman & Hall, 1991
- A.R. Nicoll and H. Eschnauer, Retesting Thermal Spray Powders, *Surf. Coat. Technol.*, Vol 30, 1987
- R. Roy, Ceramics by the Solution-Sol-Gel Route, *Science*, Vol 238, 1987
- “Standard Test Method for Flow Rate of Metal Powders,” B 213, *Annual Book of ASTM Standards*, ASTM

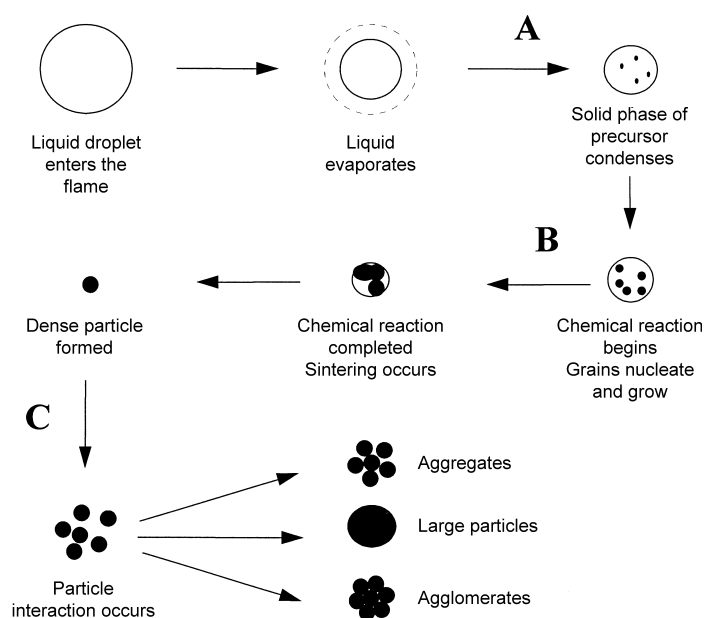


Fig. 13 Research method for obtaining deposits directly from a sol-gel without conventional powder manufacture

- T.A. Taylor et al., Plasma-Sprayed Yttria-Stabilized Zirconia Coatings: Structure-Property Relationships, *Surf. Coat. Technol.*, Vol 43/44, 1990
- “Test Method for Apparent Density of Free-Flowing Metal Powders,” B 212, *Annual Book of ASTM Standards*, ASTM
- F.Y. Wang, *Ceramic Fabrication Processes*, Academic Press, 1976

Introduction to Applications for Thermal Spray Processing

HISTORICALLY, THERMAL SPRAY PROCESSES have been used as a method of repairing, rebuilding, and retrofitting machine components, restoring original dimensions, or applying corrosion-resistant metals to various items of infrastructure, such as bridges, pilings, and lock gates. The expression *spray metallizing* thus became a routine term for most thermal spray processes. However, many of the restored components performed better than their new counterparts, a fact that should have led to their use on original equipment. A lack of understanding of these surfacing processes, however, resulted in industry considering their use largely as a last resort. Very few machine elements were actually designed for thermal spray coatings in the early history of this technology.

Because of growing interest and continued scientific pursuit among the materials community, thermal spray processes are now widely accepted by many industrial sectors. The aerospace industry was the first manufacturing segment that recognized the great utility and value of thermal spray coatings and was largely responsible for driving the development and confidence in thermal spray processes and coatings. Thermal spraying and the many materials applied as coatings have grown to include numerous applications. A partial list of applications includes coatings used for:

- Wear prevention
- Dimensional restoration and repair
- Thermal insulation and control
- Corrosion resistance
- Oxidation resistance
- Lubricity and low-friction surfaces
- Abrasive activity
- Abradable seals
- Biomedical compatibility
- Electromagnetic shielding

Most of these applications are found in a substantial cross section of world industry and are described in depth in the article “Selected Applications” that immediately follows in this Section of the Handbook. In this introductory article, coatings for wear and corrosion resistance are emphasized. These two areas offer the greatest number of applications for thermal spray coatings.

Coatings for Wear Resistance (Ref 1)

Selection of a thermal spray coating for a wear-resistant application is based on the type of wear mode that is anticipated. Applications and recommended materials for coatings resistant to various forms of wear are summarized as follows.

Adhesive Wear

Adhesive wear occurs when two surfaces slide against each other with intended motion, producing fragments from one surface that adhere to the other. It arises from the strong adhesive forces set up when two materials come into intimate contact. It generally occurs when lubrication is inadequate and results in metal transfer, usually called galling.

Soft bearing coatings allow the embedding of abrasive particles and also permit deformation for alignment of bearing surfaces. Adequate lubrication is required. Coatings of this type are generally low in cost, because they wear in preference to the mating surface.

The following performance factors apply to soft bearing coatings:

- Good lubrication must be provided, or the wear rate will be excessive.
- The coating must be soft enough to trap the many abrasive particles that will be carried by the lubricant.
- These coatings generally have poor abrasive wear resistance.
- The inherent nature of thermal spray coatings enhances their usefulness as bearing coatings. Pores act as reservoirs for lubricant; with reduced particle junctions, there is less tendency for adhesive wear.

Applications and recommended materials for soft bearing coatings are listed in Table 1.

Hard bearing coatings are highly resistant to adhesive wear. They are used where embeddability and self-alignment are not important and where lubrication is marginal.

The following performance factors apply to hard bearing coatings:

- Lubrication should be good but is not as important as for soft bearing coatings, because the high wear resistance of these materials allows them to withstand momentary unlubricated service.
- Applications that require hard bearing coatings are usually characterized by high load and low speed.
- Surfaces should generally be of equal hardness.
- Although like coatings can be used for sliding against each other, unlike combinations are frequently used, for example, a coating running against a wrought metal. This reduces seizing and scuffing.
- Wear rate generally increases with temperature.

Applications and recommended materials for hard bearing coatings are listed in Table 1.

Table 1 Thermal spray coatings for friction and wear applications

Type of wear	Coating material	Coating process(a)	Applications	
Adhesive and abrasive wear				
Adhesive wear	Soft bearing coatings:			
	Aluminum bronze	OFW, EAW, OFP, PA, HVOF	Babbitt bearings, hydraulic press sleeves, thrust bearing shoes, piston guides, compressor crosshead slippers	
	Tobin bronze	OFW, EAW		
	Babbitt	OFW, EAW, OFP		
	Tin	OFW, EAW, OFP		
	Hard bearing coatings:			
	Mo/Ni-Cr-B-Si blend	PA	Bumper crankshafts for punch press, sugar cane grinding roll journals, antigalling sleeves, rudder bearings, impeller shafts, pinion gear journals, piston rings (internal combustion), fuel pump rotors	
	Molybdenum	OFW, EAW, PA		
	High-carbon steel	OFW, EAW		
	Alumina/titania	OFP, PA		
	Tungsten carbide	OFP, PA, HVOF		
	Co-Mo-Cr-Si	PA, HVOF		
Fe-Mo-C	PA			
Abrasive wear	Aluminum oxide	PA	Slush-pump piston rods, polish rod liners, and sucker rod couplings (oil industry); concrete mixer screw conveyors; grinding hammers (tobacco industry); core mandrels (dry-cell batteries); buffing and polishing fixtures; fuel-rod mandrels	
	Chromium oxide	PA		
	Tungsten carbide	PA, HVOF		
	Chromium carbide	PA, HVOF		
	Ni-Cr-B-SiC/WC (fused)	OFP, HVOF		
	Ni-Cr-B-SiC (fused)	OFP, HVOF		
	Ni-Cr-B-SiC (unfused)	HVOF		
	Surface fatigue wear			
	Fretting: intended motion applications	Molybdenum	OFW, PA	Servomotor shafts, lathe and grinder dead centers, cam followers, rocker arms, piston rings (internal combustion), cylinder liners
		Mo/Ni-Cr-B-SiC	PA	
	Fretting: small-amplitude oscillatory displacement applications at low temperature (<540 °C, or 1000 °F)	Co-Mo-Cr-Si	PA, HVOF	Aircraft flat tracks (airframe component); expansion joints and midspan supports (jet engine components)
		Aluminum bronze	OFW, EAW, PA, HVOF	
Fretting: small-amplitude oscillatory displacement applications at high temperature (>540 °C, or 1000 °F)	Cu-Ni-In	PA, HVOF	Compressor air seals, compressor stators, fan duct segments and stiffeners (all jet engine components)	
	Cu-Ni	PA, HVOF		
	Co-Cr-Ni-W	PA, HVOF		
Erosion	Chromium carbide	PA, HVOF	Exhaust fans, hydroelectric valves, cyclone dust collectors, dump valve plugs and seats, exhaust valve seats	
	Chromium carbide	PA, HVOF		
Cavitation	Tungsten carbide	PA, HVOF	Wear rings (hydraulic turbines), water turbine buckets, water turbine nozzles, diesel engine cylinder liners, pumps	
	WC/Ni-Cr-B-SiC (fused)	OFP, HVOF		
	WC/Ni-Cr-B-SiC (unfused)	OFP, HVOF		
	Chromium oxide	PA		
	Ni-Cr-B-SiC-Al-Mo	PA		
	Ni-Al/Ni-Cr-B-SiC	PA		
	Type 316 stainless steel	PA		
	Ni-Cr-B-SiC (fused)	OFP, HVOF		
	Ni-Cr-B-SiC (unfused)	HVOF		
	Aluminum bronze	PA, HVOF		
	Cu-Ni	PA, HVOF		

(a) OFW, oxyfuel wire spray; EAW, electric arc wire spray; OFP, oxyfuel powder spray; PA, plasma arc spray; HVOF, high-velocity oxyfuel powder spray. Source: Ref 1

Abrasive Wear

Abrasive wear occurs when hard foreign particles, such as metal debris, metallic oxides, and dust from the environment, are present between rubbing surfaces. These particles abrade material off both surfaces. The selection of coating materials for this application should generally be based on operating temperature and surface finish requirements. The following performance factors must be considered:

- The coating must be hard. In particular, surface hardness should exceed the hardness of the abrasive grains present.
- The most common abrasive is silica (sand), with a hardness of approximately 820 HK. (For comparison, WC-Co cermet is 1400 to 1800 HK; Al_2O_3 is approximately 2100 HK.)
- Information about the abrasive—how often it is replenished, whether it is sharp and brittle, how it breaks down—is important in selecting the coating and estimating its performance.
- If the system is closed, debris created by the wear process will also contribute to the wear rate and thus must also be considered.
- The coating must exhibit oxidation resistance at the service temperature.

Applications and recommended materials for coatings resistant to abrasive grains at low and high temperatures are listed in Table 1.

Surface Fatigue Wear

Repeated loading and unloading cause cyclic stress on a surface, eventually resulting in the formation of surface or subsurface cracks. The surface ultimately fractures, and large fragments are lost, leaving pits. This phenomenon can occur only in systems where abrasive and adhesive wear are not present, for example, in systems with high surface contact loads. An area of surface must be stressed repeatedly, without constant removal of particles, to fail in fatigue. Fretting, erosion, and cavitation are typical examples of this type of wear.

Fretting. Some fretting-resistant coatings resist wear caused by repeated sliding, rolling, or impacting over a track. The repeated loading and unloading cause cyclic stresses, inducing surface or subsurface cracks. Other coatings resist wear caused when contacting surfaces undergo oscillatory displacement of small amplitude. This type of wear is difficult to anticipate, because no intended motion is designed into the system. Vibration is a common cause of fretting. The following performance factors apply to coatings for fretting resistance:

- The coating must be resistant to oxidation at the service temperature. If an oxide forms, it must be tough and tenacious; a loosely adherent oxide will cause severe abrasive wear.
- A surface that is free of stress, particularly tensile stress, is desirable. High-shrink coatings tend to have high surface stress and do not perform as well as low-stress coatings.
- Brittle coatings fail rapidly. Tough coatings tend to perform better.
- Coatings with hard particles distributed in a soft matrix are generally the most durable.

Applications and recommended materials for fretting-resistant coatings are listed in Table 1.

Erosion is caused when a gas or liquid that ordinarily carries entrained particles impinges on a surface with velocity. When the angle of impingement is small, the wear-producing mechanism is closely analogous to abrasion. When the angle of impingement is normal to the surface, material is displaced by plastic flow or is dislodged by brittle failure. The following performance factors apply to erosion-resistant coatings:

- If the angle of particle impact is less than 45° , the coating selected should be harder and more abrasion resistant.
- If the angle of particle impact is greater than 45° , the coating should be softer and tougher.
- At high service temperatures, coatings should have high hot hardness and oxidation resistance at temperatures and environments ranging from 540 to 815°C (1000 to 1500°F).
- When the carrier is liquid, the corrosion resistance of the coating must be considered.

Applications and recommended materials for coatings used to resist particle erosion are listed in Table 1.

Cavitation is caused by mechanical shock that is induced by bubble collapse in liquid flow. Materials that resist fretting-type surface fatigue are resistant to cavitation. The most effective coating properties are toughness, high wear resistance, and corrosion resistance. The following performance factors apply to cavitation-resistant coatings:

- Relative motion between a liquid and metal surface, including bubble generation and bubble collapse, must exist for cavitation to occur.
- Liquids will penetrate sprayed coatings unless fused; therefore, all coatings should be sealed.
- Selection of a coating must be influenced by its resistance to the liquid used in a particular application.
- Hardness is an important factor, but coatings must also be tough. Brittle coatings fail quickly.
- Coatings that work harden are especially resistant to the repeated pounding of cavitation.

Applications and recommended materials for coatings resistant to cavitation are listed in Table 1.

Coatings for Corrosion Resistance (Ref 2)

Thermal spray coatings, primarily zinc, aluminum, zinc-aluminum alloys (85Zn-15Al), and aluminum metal-matrix composites (Al-10 vol% Al_2O_3), have been successfully used to combat corrosion in a wide range of applications. Steel structures and components that have been zinc or aluminum sprayed include television towers, antennae, radar, bridges, light poles, girders, ski lifts, and countless other similar structures. In addition, thermal spray coatings, primarily aluminum, offer years of protection in marine applications, such as buoys and pylons. Aluminum spraying has been used in offshore oil rigs for well head assemblies, flare stacks, walkways, and other structural steel components.

Shipboard components, both above and below deck, commonly use aluminum spraying. The United States Navy, in particular, uses aluminum spraying extensively to combat corrosion. There are countless approved applications for sprayed metal coatings aboard Navy ships.

Numerous immersion applications have also employed zinc or aluminum spraying, for example, dams and sluice gates. The inte-

riors of potable water storage tanks are also sprayed with zinc to provide corrosion protection of the steel without the threat of contaminating the water with a solvent that may be present in an epoxy coating system.

Aluminum has also been used to control chemical corrosion in such applications as storage tanks for fuels or other liquids. The interiors of railroad hopper cars are often sprayed to protect them against sulfuric acid (H_2SO_4) corrosion when hauling coal.

Stainless steels (e.g., 316L), Hastelloy alloys (e.g., Hastelloy C-276), and other alloys can also be effective against chemical corrosion for storage vessels, rolls, pumps, and other structures. The coatings must be properly sealed when using these alloys.

Thermal spray coatings are also being used increasingly in highly demanding petrochemical environments. As shown in Table 2, such coatings must be resistant to corrosion, wear, or the combination of corrosion and wear.

Common applications involving high-temperature corrosion include coating exhaust stacks, chimneys, flues, rotary kilns and dryers, catalytic crackers, and furnace parts. These generally involve the use of an aluminum or nickel-chromium alloy coating such as those listed in Table 3.

One specific high-temperature corrosion application that has met with great success in recent years is the coating of boilers in paper mills, power plants, and chemical plants. Water-wall tubes in these applications suffer severe corrosion because of the high sulfur content of the burning fuel, the high operating pressures, and abrasion. A 375 μm (15 mil) coating of Ni-43Cr-2Ti alloy offers extremely good protection against this very severe corrosion at temperatures up to 550 °C (1020 °F).

Table 2 Petrochemical applications of thermal spray coatings

Component	Coating use	Process	Coating
Ball valves	Wear/corrosion	HVOF	Tungsten carbide
Gate valves	Wear/corrosion	HVOF	Tungsten carbide
Piston rods	Wear	Flame spray	Chrome oxide
Offshore oil rigs	Corrosion	Flame spray	Aluminum
Pump housings	Restore dimension	Arc spray	Aluminum bronze
Compressor cylinders	Restore dimension	Arc spray	420 stainless steel
Processing tanks	Corrosion	Flame spray	Aluminum

HVOF, high-velocity oxyfuel. Source: Ref 3

Table 3 Thermal spray coatings for elevated-temperature service

Service temperature, °C (°F)	Coating metal or alloy	Coating thickness	
		μm	mils
Up to 550 (1020)	Aluminum	175	7
Up to 550 (1020) in the presence of sulfurous gases	Ni-43Cr-2Ti	375	15
550–900 (1020–1650)	Aluminum or aluminum-iron	175	7
900–1000 (1650–1830)	Nickel-chromium or MCrAlY	375	15
900–1000 (1650–1830) in the presence of sulfurous gases	Nickel-chromium(a) Aluminum(a)	375 100	15 4

(a) Coating system consists of an aluminum layer deposited on top of a nickel-chromium layer. Source: Ref 3

REFERENCES

1. B.A. Kushner and E.R. Novinski, *Thermal Spray Coatings, Friction, Lubrication, and Wear Technology*, Vol 18, *ASM Handbook*, ASM International, 1992, p 829–833
2. R.H. Unger, *Thermal Spray Coatings, Corrosion*, Vol 13, *Metals Handbook*, ASM International, 1987, p 459–462
3. M.L. Berndt and C.C. Berndt, *Thermal Spray Coatings, Corrosion: Fundamentals, Testing, and Protection*, Vol 13A, *ASM Handbook*, ASM International, 2003, p 803–813

Selected Applications

THE THERMAL SPRAY APPLICATION TECHNOLOGY described in this article is approached in three ways:

- An industry-by-industry review of thermal spray applications. Areas of interest/industries discussed include aerospace, automotive, and orthopedic and dental applications, ceramics and glass manufacture, corrosion of marine and land-based infrastructure, electronic materials and devices, printing industry components, processing industries (e.g., chemical and pulp and paper) and corrosion control, metal processing, electrical utilities, and textile and plastics industries.
- An examination of the state-of-the-art developments in thermal spray coatings for land-based turbine applications. As is seen, some of the developments in this area parallel similar research activities in the aerospace industry.
- A review of recently emerging thermal spray application technologies. These include the use of thermal spray coatings as replacements for electroplated hard chromium coatings, the use of thermal spray polymer coatings as replacements for organic coatings (paints), the use of thermal spray processing to remove lead-base paints, the use of thermal spray forming of free-standing structures (for example, tooling manufacture), and cold spray process applications.

The purpose of this article is to enable readers to:

- Understand the reasoning behind process selection for various applications
- Define the nature of the problems encountered by various industries and identify the thermal spray process and material that best solves the problem
- Comprehend the various limitations of thermal spray coatings and understand how to combat them as necessary
- Appreciate the versatility of these processes and use the knowledge gained from other industry settings to the benefit of their applications

Thermal Spray Processes Described in this Article

Previous articles in this Handbook have shown that thermal spraying is a “family” of materials consolidation/deposition processes (see also the section “Thermal Spray Process Categories” in this article). Each process has certain unique characteristics that make it attractive for specific applications. Some of these processes, however, are so versatile that applications are possible over a very wide industrial spectrum. This section is offered as a brief review of the thermal spray processes described in earlier articles but with an emphasis on their specific abilities within the family of thermal spraying methods.

Thermal Spray Process Categories

The thermal spray family of processes is typically grouped into three major categories:

- Combustion spray (flame-wire/rod/powder and high-velocity oxyfuel)
- Wire arc or arc spray (air or inert gas)
- Plasma (air, vacuum, or inert atmosphere)

Combustion-wire flame spray is the practice of using combusting oxygen and fuels to melt a single wire fed into the flame by either a compressed gas or an electric motor. Compressed air or inert gas is used to atomize, or break up, the molten droplets and accelerate them onto an appropriate substrate. This process can be used for all elemental metals and those alloys that can be drawn into wire.

Wires ranging in diameter from 3 to >9 mm (0.12 to >0.35 in.) are typical for this process. The finer wires result in less heat input to the substrate. Oxides are almost always generated during this process; yet, in many cases, oxides are beneficial to the applications, imparting additional hardness and lubricity (depending on the material sprayed).

Metallic compositions usually come to mind when considering this process, but cored wires containing ceramic powders have been used, so that the deposit becomes a coating equivalent of a metal-matrix composite. Ceramic wires have also been produced using organic binders.

Combustion-rod spraying is a variation of the combustion-wire spray process and features short lengths of ceramic rods that are fed into the flame. The coatings produced via this process are usually quite porous but have excellent wear properties. It is a common practice to infuse fluorocarbons into the coating to add to the lubricity. The Rokide process (St. Gobain Advanced Ceramics) is a commercial example of a flame/rod combustion spray process.

Combustion-powder flame spraying also uses oxygen/fuel combustion as the heat source but feeds metal, ceramic, some cermets, and certain organic (polymer) powders to produce coatings of the same. As in wire-arc flame spray, some oxidation of the coatings occurs during processing. One common family of coatings consists of nickel brazing material containing boron and silicon along with some refractory metals. Postprocessing after the application of this coating requires a heat treating process at temperatures of approximately 1030 °C (1890 °F), which forms a hard, diffusion-bonded surface. This is commonly known as spray and fuse.

High-velocity oxyfuel (HVOF) is also a combustion-driven process but uses advanced technology and internal combustion to produce very high-velocity gas streams. Particle velocities are also high. Temperatures are not as high as those in plasma spraying, but the high kinetic energy of the particles in HVOF processes compensate for the lower temperatures. While most HVOF processes involve continuous combustion, a variant of the process—the detonation gun, or D-gun, process—uses a controlled series of explosions (pulsed combustion) to blast the coating onto the surface of the workpiece.

Wire electric arc spraying uses two electrically conducting wires as feedstock. A continuous direct current arc melts the tips of the advancing wires, and a high-flow gas jet atomizes the molten material and accelerates the resulting small droplets toward the surface being coated. This process is one of the most economical thermal spray processes but is limited to conductive materials that can be made in wire form. Some nonconductive materials can be deposited with this technology by using cored wires made with conductive wire sheaths. Coatings produced with this process have relatively high oxide contents, but this can be minimized by using an inert atomizing gas.

Air, or atmospheric, plasma spraying (APS) uses an electric arc as the heat source to generate a very high-temperature inert gas plasma jet. Plasma spraying often produces coatings of higher density and higher bond strengths from powdered feedstocks. Oxygen from the surrounding air is entrained into the plasma, so some oxides are typically formed in the coating.

Inert chamber plasma spraying locates the entire plasma spray process in a controlled inert atmosphere chamber. Such chambers are often equipped with pressure controls that permit the use of load locks for semicontinuous operation. With such equipment, oxidation is at a minimum. It is also an excellent facility for processing high-cost materials, where recovery of overspray is important.

Vacuum plasma spraying, or low-pressure plasma spraying (LPPS), is similar to inert chamber processing but with greatly reduced atmospheric pressure. The plasma jet broadens and extends, the distance to the substrate is greater, and coatings with a degree of true metallurgical bonding can be produced.

Aerospace Applications

Possibly, the most predominant uses for thermal spray coatings today are in the aircraft and land-based turbine engines (see the section "Thermal Spray Coatings for Land-Based Turbine Applications" in this article). Combustion-wire spraying of aluminum on the exposed cylinders (jugs) of radial piston engines was used on World War II aircraft. Reportedly, the coatings prevented oxidation of the ferrous jugs, which would have been detrimental to the air cooling of the engine.

Today, both military and industrial turbine engine units worldwide contain hundreds of components with thermal spray coatings serving as a surface defense mechanism to promote life extension. On older engines, such coatings were often retrofitted on components exhibiting higher wear. Today, industry accepts and designs components to incorporate particular coatings. In fact, some estimates claim that as high as 75% of the components in modern aircraft engines are coated in some fashion.

Because of its versatility, APS is used for the majority of thermal spray coating work, with some limited applications of combustion-powder spraying still found. Secondary processes, such as thermal diffusion and other heat treatments, are also added to plasma coatings.

The aerospace industry has been relatively quick to adapt advanced thermal spray technology such as LPPS and HVOF. Presently, new twin-wire arc spray processes are being explored.

Use of these newer technologies is expected to increase. High-velocity oxyfuel, in particular, is being adapted to coatings previously applied by APS.

Fretting Wear

Fretting in turbine engines occurs in two forms, intended motion and unintended motion, and both are found in the lower- and higher-temperature sections of engines. Fretting occurs between two metal (or ceramic) components with surfaces in contact. In addition to intended relative motion (if any), centrifugal forces, coefficient of thermal expansion (CTE) differences, and engine vibration can cause oscillatory relative motion between the two surfaces. The rubbing action causes a microwelding phenomenon recognized as galling, which breaks down the component surfaces. Generally, the wear generates particulate debris; additional particulate debris from other sources may be added, resulting in three-body abrasion.

A number of thermal coatings are used to overcome this problem. These are discussed in terms of material systems.

Metallic carbides (or cermets) are common material systems used in both hot and cold sections of engines. Chromium carbides in a ductile matrix of nickel-chromium are commonly used in hot sections (Fig. 1). Tungsten carbides, in matrices of cobalt ranging from 6 to 12%, with possible additions of chromium (4 to 12%), are used at lower temperatures (below 540 °C, or 1000 °F), because the hot hardness and oxidation resistance of tungsten carbide drops rapidly as temperatures increase. There is growing interest in the use of titanium carbide (TiC), which also retains its hardness at higher temperatures and is also capable of exhibiting low friction coefficients.

From the 1960s through the 1980s, most tungsten carbide (WC) coatings were specified for plasma spray methods; however, compositional changes occur as some of the carbide dissolves into the cobalt matrix at the higher processing temperatures. Decarburization of WC to the more friable, less wear-resistant W_2C can also occur in plasma sprayed WC/Co coatings. Today, the newer HVOF processes retain the carbide phase more readily.

Refractory metals are particularly good for applications in both the cold and hot sections of engines where low-friction

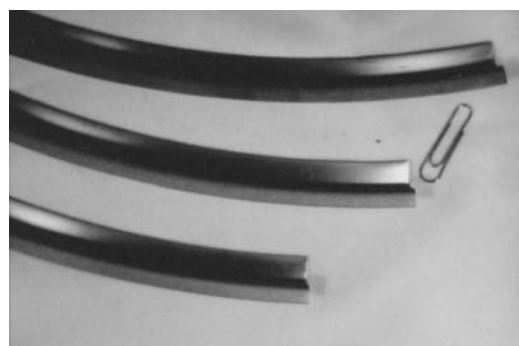


Fig. 1 NiCr-Cr₃C₂ coating applied to seal segments

behavior is desired. These coatings are usually formed from a single refractory metal (molybdenum, tungsten, rhenium), with small additions of cobalt, chromium, nickel, and silicon. Molybdenum is favored for coatings applied to both component interfaces. Molybdenum does not weld to itself readily and forms molybdenum trioxide at higher temperatures—an oxide that has high lubricity. Such coatings offer a secondary benefit by facilitating disassembly of the component group at rebuilding time.

Vacuum plasma sprayed rhenium and molybdenum coatings are used as ablation protection on graphite and carbon-carbon composite rocket/missile nozzle throat inserts.

The cobalt-base Tribaloy thermal spray (plasma) coatings are good examples of molybdenum-containing wear-resistant materials. In these materials, molybdenum (29%) and silicon (~3%) are added at levels in excess of their solubility limit, with the objective of inducing the precipitation of the hard (and corrosion-resistant) Laves phase (CoMoSi or $\text{Co}_3\text{Mo}_2\text{Si}$). An application to turbine vane bases is shown in Fig. 2.

Refractory metal hardfacings have historically been applied by APS, but more recently, HVOF has been used to achieve certain desirable coating morphologies.

Antifretting and clearance control coatings include CuNiIn, one of the oldest coatings used in turbine engines for fretting and wear applications. Some of these coatings are applied by combustion flame spray with a bond coat interface (usually, a nickel-aluminum alloy such as Ni-5Al). Air plasma spray may also be used without a bond coat. This coating allows easy disassembly of components where tight fits, compounded with high pressures and fretting, might otherwise create a weld during engine operation. CuNiIn coatings are found on all manner of brackets, pipe straps, and fuel-delivery tubes. It is also common to apply molybdenum disulfide (MoS_2) or similar solid-lubricant coatings on CuNiIn, using its porous surface to entrap lubricious material.

Nickel-aluminum-base coatings are also commonly used. Several versions of these materials, some with additions of chromium, are often used as bond coats for other hardfacings. They are good performers at both cold- and hot-section temperatures.

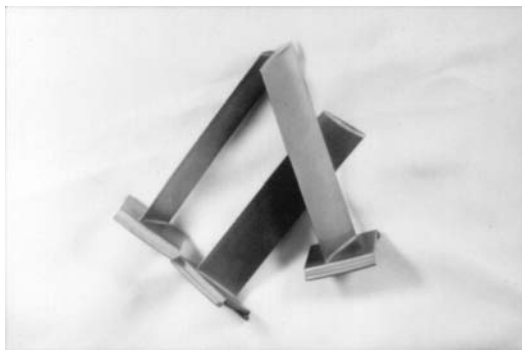


Fig. 2 Turbine vanes with the bases coated with cobalt-base Tribaloy (T-800) alloy. The coating aids in blade removal during rebuilds.

High-Temperature Protection

There are several specific applications in the hot sections of engines where corrosion and high-temperature metal fatigue occur together. If substrate temperatures can be held down at critical moments of engine activity, there is an opportunity to raise combustion temperatures, with resulting improvements in engine performance (thermal efficiency).

Metallic alloys used to inhibit corrosion focus on the family of materials known as MCrAlYs, where M is nickel, molybdenum, cobalt, iron, or alloys of these elements, such as NiCo, CoNi, and so on. The rapidly solidified microstructure of coatings of these materials created by thermal spraying can benefit their performance under high-temperature corrosive conditions. A common application of these materials is by LPPS on stator segments. Coatings over 3 mm (0.12 in.) thick are heat treated after spraying, which results in increased bond strength, grain growth, and improved density. They are subsequently machined to final dimension and become the effective lining that defends against the oxidative environment.

The LPPS process is greatly favored for such applications, because it is the one process that produces the lowest oxide levels in coatings and the highest densities and a degree of metallurgical bonding by permitting deposition on high-temperature substrates.

The CTE differences are important in the selection of the MCrAlY composition, for obvious reasons. Optimal (minimal) stresses suggest that expansion differences should be less than 1×10^{-6} m/m/K (0.5×10^{-6} in./in./°F), but data on thermal expansion coefficients of thermal spray coatings are limited. Handbook data for bulk materials should be used only as a guide, because little information is available on the effects of rapid solidification processes on CTE values. Additionally, heat treatments including the use of these coatings at operating temperature may alter expansion characteristics as grain growth occurs.

Thermal barrier coatings (TBCs) consist of a low-thermal-conductivity ceramic layer deposited over a MCrAlY bond coat. Such coating systems have been the subject of extensive study since the early 1960s, and the evolution of TBCs has been steady since then.

The ceramic of choice for TBCs is zirconia (ZrO_2), but pure zirconia exhibits a phase change as the temperature approaches 425 °C (800 °F), resulting in a substantial volume change that can subsequently generate internal stresses and lead to premature coating failure. Oxide stabilizers are therefore added to the zirconia to prevent this mode of failure, common examples being yttria, calcia, magnesia, or ceria. Yttria (Y_2O_3) is the most widely used stabilizer for TBCs at the present time, and the material is commonly known as yttria-stabilized zirconia (YSZ).

The amount of stabilizer is also important. Initially, fully stabilized zirconia containing 12 to 20% Y_2O_3 was used, but recently it has been demonstrated that partially stabilized material improves TBC performance, as shown in Fig. 3 and discussed in Ref 1. It was reasoned that allowing a controlled amount of the ceramic to undergo a phase change created microcracks that relieved high-temperature stresses and promoted longer coating life. However, operating temperatures are not generally high enough for this phase change activity to contribute significantly to life extension.

The stress-relief devices are the microcracks and 8 to 15% porosity, which initially occur during the plasma spraying of the deposit.

Thermal barrier coating failure stems from one or more of the following mechanisms:

- Attack of the MCrAlY bond coat by O_2 (the ZrO_2 is transparent to O_2), resulting in swelling and ceramic spalling
- Sintering of the ZrO_2 , which reduces the strain-relief devices
- Improper ZrO_2 powder or spraying conditions that result in a higher-than-desired monoclinic phase content
- An abrupt vertical wall of ceramic at the coating peripheral edge, which acts as a stress raiser for coating failure when exposed to deep thermal cycles
- Corrosive attack of the ceramic by impurities in fuels
- Instability and degradation of the thermally grown oxide layer of Al_2O_3 at the bond coat/ceramic interface

The state-of-the-art thermal spray TBC of today is a 6 to 8 wt% Y_2O_3 - ZrO_2 ceramic deposited at a thickness of 0.25 to 1 mm (0.010 to 0.040 in.), with 10 to 15% porosity. A bond coat of NiCrAlY or CoNiCrAlY is used at a thickness of 0.125 mm (0.005 in.). The YSZ topcoat is usually applied by APS; the bond coat is applied by LPPS or controlled atmosphere plasma spray. In recognition of greater economy, HVOF bond coats may be substituted. More recently, electroplated bond coats of platinum aluminides (Pt-Al) and electron beam-physical vapor deposition YSZ topcoats with strain-tolerant columnar grain structures have been developed (Ref 2).

Thermal barrier coatings are applied on vane bases, burner cans, afterburners, and a variety of other small engine components. Technologists envision that all hot gas flow path surfaces be so treated, although fulfillment is still in the future.

Certain deviations from the previous discussion are important to note. Magnesia-stabilized material is still used for a variety of applications. There is also a growing trend toward the use of thicker ceramics where service conditions are severe. In order to compensate for the additional stresses of thicker coatings, graded

interfaces between the bond coat and topcoat are frequently employed. This interface is formed by using two powder feeders and gradually increasing the amount of ceramic injected into the plasma while reducing the amount of bond coat material. Alternatively, several mixtures of ceramic and bond coat can be sprayed, both approaches yielding a diffuse bond line. Theoretically, stresses should be reduced, but optimizing powder and plasma spray parameters to accommodate mixtures of powders having widely different melting points and thermal characteristics is difficult.

Seals*

Rotating vane assemblies in aircraft turbines often require some method of sealing to reduce bypass flow of hot combustion gases or cold compressor gases through the spaces between the blade tips and the stationary housing (stator). Good seals greatly improve engine efficiency. Mechanical seals, such as those found in piston engines and compressors, are not practical because of the thermal expansion and high centrifugal forces acting on the rotating members. When the seal is applied by thermal spraying, it is called an abradable or rub-tolerant coating.

Abradable seals are coatings that are machined in situ by the rotating components, such as blades, so that very close tolerances result. This process provides effective sealing of the gas path. Regardless of whether the abradable is used for hot turbine or cold compressor sections, material compositions form a delicate balance of proper hardness to withstand gas and solid particle erosion, abradability, and functionality for the designed life span. The rotating parts must not be damaged when rubbing against the abradable coating. Typical damage caused by improper abradable compositions includes blade-tip wear, reduced blade height, and reduced turbine or compressor efficiency.

A variety of coating materials are used for this purpose; most notable for cold sections are combinations of various polymers and soft metals, such as aluminum, bronze, or Babbitt. These are generally applied using APS and arc spray systems, although HVOF is now also being investigated for this application. Hot-section abradables include combustion flame sprayed nickel-graphite; however, proper consideration should be given to the potential of autocatalytic galvanic corrosion of the alloy coating. Careful attention to the hardness of the coating assures the best performance. The combustion flame process is less likely to create carbides or ablate the graphite. Other turbine abradables include plasma sprayed composites consisting of MCrAlYs, polyester, and hexagonal boron nitride as well as NiAl with polyester. Figure 4 compares the suggested service temperature range of various turbine compressor abradables. Table 1 lists powder chemistries of selected thermal spray abradable seal coatings.

Dense HVOF and plasma sprayed MCrAlY alloys have also been applied on turbine sections as rub-tolerant materials to avoid tip loss during high-temperature rubs and to prevent oxidation and corrosion of the tip shoes. MCrAlYs are not intrinsically abradable and could cause tip damage on the blades.

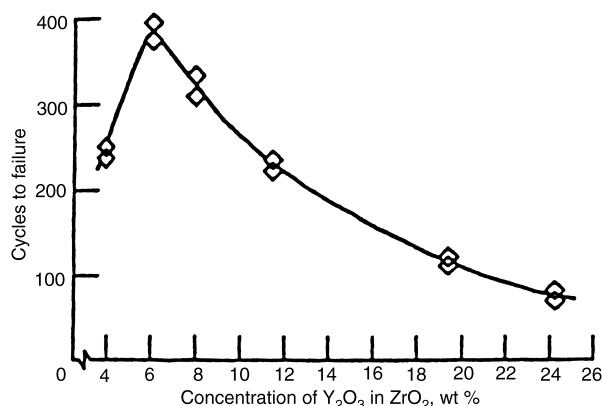


Fig. 3 Effect of yttria content on the performance of zirconia-base thermal barrier coatings. Source: Ref 1

*This section was revised by Irene Nava, Solar Turbines Inc.

More recently, variations of 7 wt% Y_2O_3 - ZrO_2 , sprayed by itself or combined with polyester and/or boron nitride particulates, have been used for thick abrasives. These coatings are made machinable by grading the porosity, with the greatest amount in the outer layers. Being a ceramic material, it is generally recommended to use abrasive or wear-resistant blade tips to avoid blade height loss.

Labyrinth seals consist of matching stator and rotor components that have an intermeshing sawtooth geometry (see Fig. 7 in the section "Thermal Spray Coatings for Land-Based Turbine Applications" in this article). This geometry increases the path

length for the bypass gas leak and, with all the turns that the gas flow encounters, effectively raises the pressure drop and reduces the bypass flow. Such seals, however, experience three-body abrasion as the result of debris ingested into the intakes. Hardfacings are often applied by thermal spray processes to combat wear of the labyrinth teeth. Carbides and alumina-base ceramics have been widely used for preventing wear. The mating stator is usually a softer, abrasible material, based on aluminum, copper, or lead alloys. For instance, bearing and bushing alloys including bronze and Babbitt (Ref 5) are thermal sprayed onto the ring seal to form a thick layer, up to ~2.5 mm (~0.10 in.) thick, that will tightly follow the sawtooth geometry to avoid gas or oil leakage.

High-Velocity Oxyfuel Thermal Spray Coatings as a Replacement for Hard Chrome Plating on Aircraft Landing Gear*

Electrolytic hard chrome (EHC) plating is a critical process that is used both for applying hard coatings to a variety of aircraft components in manufacturing operations and for general re-build of worn or corroded components that have been removed from aircraft during overhaul. In particular, chrome plating is used extensively on landing gear components such as axles, hydraulic cylinders, pins, and journals. Chromium plating baths contain chromic acid, in which the chromium is in the hexavalent state, with hexavalent chromium (hex-Cr) being a known carcinogen. During operation, chrome plating tanks emit a hex-Cr mist into the air, which must be ducted away and removed by scrubbers. Wastes generated from plating operations must be disposed of as hazardous waste, and plating operations must abide by U.S. Environmental Protection Agency (EPA) emissions standards and Occupational Safety and Health Administration (OSHA) permissible exposure limits (PEL). Recent studies have clearly shown that there are a significant number of excess deaths at the current PEL of $100 \mu\text{g}/\text{m}^3$, which is causing OSHA to explore significantly reducing the hex-Cr PEL. A Navy/industry task group concluded that the cost of compliance for all Navy operations that use hex-Cr (i.e., not just plating) would be in excess of \$10 million if the PEL was reduced to less than $5 \mu\text{g}/\text{m}^3$.

Research and development efforts conducted in the early 1990s had established that HVOF thermal spray coatings are the leading candidates for replacement of hard chrome. High-velocity oxyfuel thermal spraying can be used to deposit both metal alloy and cermet coatings that are dense and highly adherent to the base material. They also can be applied to thicknesses in the same range as what is currently being used for EHC. Currently, there are a number of HVOF thermal spray systems commercially available. Although a wide range of applications exists for these coatings, their qualification as an acceptable replacement for hard chrome plating has not been adequately demonstrated, particularly for fatigue-sensitive aircraft components. The Hard Chrome Alternatives Team (HCAT) was formed by the U.S. Department of Defense in 1996 to perform the demonstration/validation for the

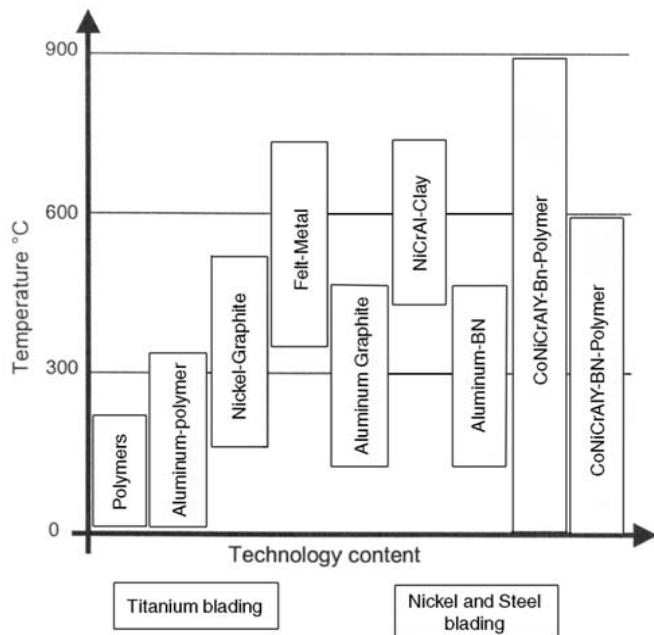


Fig. 4 Suggested temperature range for turbine compressor abrasives. Those shown to be compatible with titanium blading will also function against steel and superalloy blades. However, the reverse is not true. Source: Ref 3

Table 1 Compositions of thermal spray abrasible seal coatings

Material system	Metal matrix, wt%			Solid lubricant, wt%		Other, wt%	
	CoNiCrAl	NiCrAl	Ni	BN	Graphite	PE	Bentonite
CoNiCrAlY + BN + 15%PE	bal	4	...	15	...
CoNiCrAlY + BN + 17%PE	bal	4	...	17	...
CoNiCrAlY + BN + 18%PE	bal	4	...	20	...
Ni25C	bal	...	25
NiCrAl + bentonite	...	bal	21

PE, polyester. Source: Ref 4

*This section was contributed by Bruce D. Sartwell, Naval Research Laboratory.

HVOF coatings and to help with the implementation, both in manufacturing and repair/overhaul operations.

The objectives of the HCAT project on landing gear were to demonstrate through materials and component testing that the performance of HVOF WC/17Co and WC/10Co4Cr coatings on landing gear components was equal or superior to that of EHC coatings. Materials testing included axial fatigue, salt fog and cyclic corrosion, sliding wear, impact, and hydrogen embrittlement (HE). The HE testing had three components: verifying that the application of HVOF coatings did not cause HE in high-strength steels; verifying that hydrogen present in a high-strength steel specimen could diffuse through a HVOF coating during baking; and determining the relative susceptibility of HVOF-coated specimens to re-embrittlement as compared to EHC-coated specimens.

The results of the materials and component testing, plus a cost/benefit analysis, can be summarized as follows:

- **Fatigue:** Cycles to failure at different stress levels were measured for fatigue specimens fabricated from 4340, 300M, and Aermet 100 ultrahigh-strength steels coated with either EHC or HVOF WC/17Co to thicknesses of either 0.075 or 0.25 mm (0.003 or 0.010 in.). In general, the average number of cycles to failure at any stress level for the HVOF-coated specimens was greater than for EHC-coated specimens, and therefore, the HVOF coatings demonstrated superior performance.
- **Corrosion:** ASTM B 117 salt fog exposure tests were conducted on 4340, 300M, and Aermet 100 rod specimens coated with EHC or HVOF WC/17Co or WC/10Co4Cr to thicknesses of either 0.075 or 0.25 mm (0.003 or 0.010 in.). After 1000 h exposure, the average appearance rankings for the HVOF coatings were lower than for the EHC coatings. Thus, for this test, the HVOF coatings did not pass the acceptance criteria. However, in three-year atmospheric saltwater beach corrosion studies, the performance of HVOF WC/17Co coatings on 4340 steel was significantly better than that of EHC coatings. Based on these results and those from tests conducted outside the HCAT program, it was generally concluded that the corrosion performance of the WC/17Co and WC/10Co4Cr coatings was equivalent to that of EHC coatings.
- **Wear:** Wear tests involving a 4340 steel piston coated with 0.075 mm (0.003 in.) thick EHC or HVOF WC/17Co or WC/10Co4Cr sliding against bushings fabricated from 4340 steel, aluminum-nickel bronze, anodized 2024 aluminum alloy, a Nitrile rubber seal, or a Karon B seal generally showed that the average wear rate on the piston was less for the HVOF coatings than for the EHC coatings but that the wear on the mating surface was slightly higher.
- **Impact:** Both gravel impingement and ball impact tests were conducted against 4340 steel specimens coated with EHC or WC/17Co to thicknesses of either 0.075 or 0.25 mm (0.003 or 0.010 in.). In general, the extent of surface damage and cracking was less for the HVOF coatings, and therefore, they demonstrated superior performance.
- **Hydrogen embrittlement:** Testing verified that application of HVOF coatings does not cause HE in high-strength steels, that hydrogen can diffuse through WC/17Co coatings during nor-

mal HE relief baking, and that re-embrittlement is less likely to occur with HVOF coatings than with EHC coatings.

- **Rig and flight testing:** High-velocity oxyfuel WC/17Co coatings (0.075 mm, or 0.003 in., thick) were evaluated on a main landing gear (MLG) piston in a P3 rig test conducted at Lockheed Martin in Marietta, Georgia; WC/10Co4Cr coatings (0.075 mm, or 0.003 in., thick) were evaluated on several components on a nose landing gear in a F/A-18 E/F rig test at Messier-Dowty in Canada; and WC/17Co coatings (0.125 mm, or 0.005 in., thick) were evaluated on a MLG piston and axle journals in a three-year P3 flight test. Figure 5 shows the application of WC/Co coatings onto the P3 MLG piston for the rig test. In each case, the HVOF coatings showed no evidence of wear or delamination and passed the tests.
- **Cost assessment:** A detailed cost/benefit analysis was conducted at a landing gear overhaul facility that processes more than 1000 components per year. The results showed an annual cost avoidance of approximately \$200,000 and a 15 year net present value of approximately \$1,800,000. The payback period on the \$700,000 initial capital investment was 3 to 5 years.

The Air Force is proceeding with implementation of HVOF coatings on landing gear components at its Ogden Air Logistics Center. They have approved the application of WC/17Co coatings up to a thickness of 0.25 mm (0.010 in.) on 12 components from seven different aircraft. The Navy has approved the application of WC/17Co coatings onto P3 landing gear components. The HCAT worked with a Society of Automotive and Aerospace Engineers aerospace committee to develop and issue specifications for the WC/17Co and WC/10Co4Cr powder, the application of the coatings, and the grinding of the coatings. These specifications can

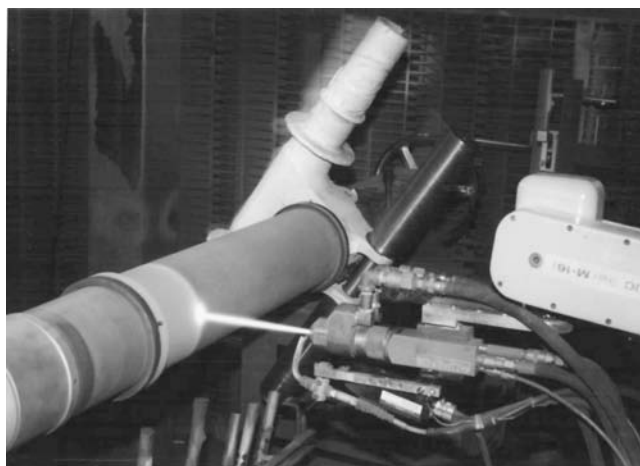


Fig. 5 Application of high-velocity oxyfuel WC/17Co coating to a P3 main landing gear cylinder. Courtesy of B.D. Sartwell, Naval Research Laboratory

now be used by any overhaul depot, and their use will result in consistency between facilities with respect to coating properties.

Thermal Spray Coatings for Land-Based Turbine Applications*

This section intends to illustrate the most commonly employed thermal spray coatings in land-based engine applications. Today's turbine engines are designed around the use of high-performance materials that rely heavily on thermal sprayed coatings to increase life span and operation efficiency. The engine materials range from aluminum alloys, cast iron, high-strength steels, stainless steels, nickel-base alloys, and superalloys to single-crystal materials. The external exposure temperature of the engine might range from cold environments of -40°C (-40°F) to extreme heats of 55°C (130°F). The internal sections of the compressor vary from the atmospheric suction air temperature to combustion temperatures in the fuel-fired combustion liner or cans and the turbine section. Additionally, contaminants inherent in the environment and fuel play a role in the life of turbine components. The coating material and method of application are usually designed according to the

particular service and the expected life of the coating, which is defined by a time between major inspections. The coating can be applied onto newly manufactured or overhauled bearing journals, bearing seals, stub shaft journals, labyrinth seals, blades, nozzles, tip seals, inlets and exhausts, and housings. Figure 6 illustrates an engine cutaway and locations where thermal spray coatings are applied.

Wear Resistance

A wide array of solid-surface mechanical interaction mechanisms occurs in turbine systems, causing wear processes such as abrasion, erosion, fretting, and galling. The wear effect in many areas is enhanced by the synergistic effect of low- or high-temperature oxidation and corrosion. Many of the wear interactions are mitigated with lubricant oils. Components exposed to elevated temperatures might face severe wear problems due to inability to provide lubrication. Wear-resistant coatings are widely applied on turbine and compressor shafts, bearing journals, thrust collar diameters and seats, and labyrinth seals (Fig. 7).

Some of the most popular coatings to provide wear resistance are tungsten carbide/cobalt (WC/Co), tungsten carbide/nickel (WC/Ni), tungsten carbide/cobalt/chromium (WC/Co/Cr), chromium carbide/nickel chromium (CrC/NiCr), and Co-Cr-Si-Mo alloys known as Triballoys. Usually, the tungsten carbides are used

*This section was contributed by Irene Nava, Solar Turbines Inc.

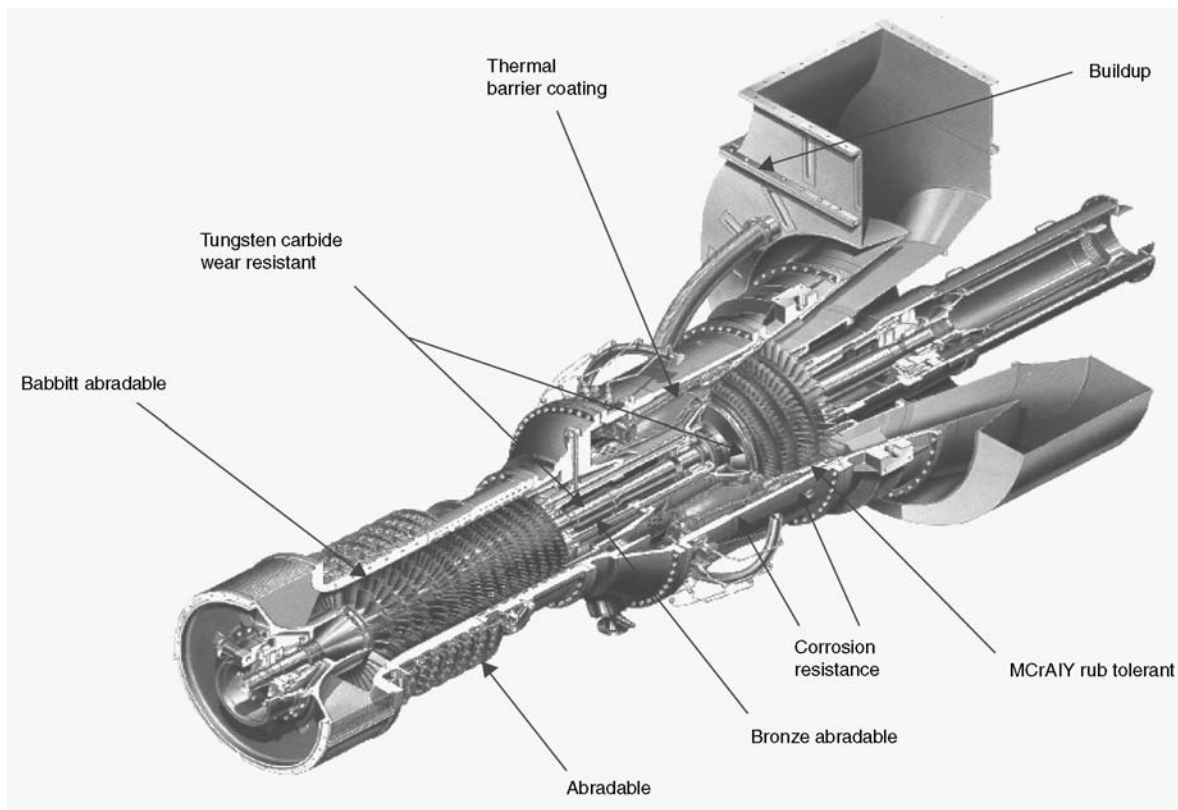


Fig. 6 Locations where thermal spray coatings are applied on industrial land-based gas turbines. Courtesy of I. Nava, Solar Turbines Inc.

for lower temperatures, below 480 °C (900 °F), such as in the compressor section of the gas turbine, while chromium carbides are used for, but not restricted to, applications up to 815 °C (1500 °F). Above these temperatures, the respective carbides might start decomposing. Concentrations of metallic content in the tungsten and chromium carbides vary and are tailored according to the application. The cobalt amount in WC/Co is typically 11 to 18 wt%, while the amount of NiCr (usually Ni-20Cr) in CrC/NiCr coatings is 7 to 50 wt%. The high-chromium-containing coatings are used for both wear and corrosion resistance and for higher-temperature applications found in the turbine section. Variations of the carbide coatings to add oxidation or corrosion resistance, such as WC/Co/4Cr, WC/CrC/NiCr, and WC/NiCrMo, are also available. Wear-resistant coatings are applied by conventional HVOF and plasma spray, and in a growing number of applications, they are applied by cold spray HVOF or kinetic metallization. A few core wires with attractive wear-resistant materials also allow application of thick, wear-resistant coatings by twin-wire arc spray. Nanocomposite carbides and nanostructured steels are relatively new compositions that offer impact toughness, wear and corrosion resistance, and other potential benefits.

The thermal spray coatings for wear resistance are usually applied to a thickness of 75 to 380 μm (0.003 to 0.015 in.) and often require a surface finish between 0.1 and 1.6 μm (4 and 64 $\mu\text{in.}$) roughness average. When thicker coatings are needed to achieve design dimensions, the part is first coated with nickel-base (i.e., alloys 718, 625, NiCrMo, or NiCrAl) or iron-base (i.e., stainless steels) coatings for buildup purposes.

It is known that a wear-resistant coating can extend component life. For instance, a WC/Co coating on a rotating shaft can increase the shaft life from a couple of months to a couple of years. Specifically, the life of a carbide-coated gas compressor shaft can be extended from six months to more than five years. The coatings

can be applied on the newly manufactured part to prevent wear. A wear coating can be applied to a serviced part to bring it to dimensional tolerances. When a nominal thickness is required, it is on the order of 254 μm (0.010 in.). Applications become more complicated for thick buildup coatings, because the performance decreases for thick and dual-layered coatings. In general, the composition of the hard phases, such as carbides in the coating material, can alter the coating ductile behavior. Therefore, the material needs to be selected according to the application requirements. For instance, a higher content of ductile material is recommended for low-angle blast erosion, that is, increased NiCr content in CrC/NiCr, rather than high amounts of CrC in the composite. For high-angle wear situations, the opposite concept applies.

Oxidation and Corrosion Resistance

Turbines can be located in harsh, corrosive environments, including high-temperature deserts, offshore platforms, and humid rural and city areas. The whole unit is exposed to environmental weathering. The exterior is usually protected with paints. Aluminum and zinc-base paints are used for oxidation and corrosion resistance. The compressor section is exposed to the suction air, which could carry excessive amounts of moisture and chlorides, depending on the location of the unit. The turbine is operated with a wide range of fuels, from clean gases to low-grade fuels containing sulfur and vanadium. To provide corrosion and oxidation resistance, nickel- and/or cobalt-base alloyed coatings are used. These materials typically are high-chromium-containing alloys, namely, NiCrMo (Hastelloy or Nistelle), CoCrSiMo (Triballoy), and MCrAlYs. Iron-base alloys could also be used for corrosion resistance. Most of these coatings are applied by HVOF or plasma spray. Most TBCs require a high-oxidation, corrosion-protective bond coating. The MCrAlY coatings offer these superior properties. Often, the MCrAlY coatings are post-heat treated to increase bond strength via a diffusion bonding mechanism, which improves the performance of the coating significantly. Oxidation- and corrosion-resistant coatings are applied on air inlets, combustor liners, injectors, turbine tip shoes and nozzles, and exhausts.

Abradables and Seals

To maintain tight blade-tip clearances, it is necessary to apply abrasible coating materials to the engine turbine shrouds and compressor case. Clearances between industrial gas turbine rotating systems (blades) and static components in the compressor and turbine sections of the engine have strong impact on the efficiency and fuel consumption of the gas turbine. Additionally, the abrasible coating offers wear protection to the shroud material and rubbing blades. Turbine seals are designed to accommodate high-speed blade-tip rubs induced during engine start-up and, to a lesser extent, at steady-state operating conditions. A good abrasible coating should exhibit a compromise among abrasibility, erosion resistance, and strength. It should be soft enough to prevent blade damage and strong enough to resist abrasion and spallation during rubbing. It must also be oxidation and corrosion resistant in the service environment. Blade damage is detrimental to the overall



Fig. 7 Application of high-velocity oxyfuel coarse-grain WC/Co coating onto labyrinth seal knife edges of an industrial gas turbine compressor rotor shaft. The coating is applied to 130 μm (5 mils) thickness and provides a smooth cutting action into the mating abrasible seal coating. Courtesy of Z.Z. Mutasim, Solar Turbines Inc.

engine, compressor, and turbine efficiency. When damage to the blade and/or shroud occurs, it is often due to the blades rubbing the shrouds, causing adhesive material transfer (similar to galling), melting, cutting, smearing, and tribooxidation by increased heat due to friction. According to the exposure temperature, abrasable coatings could be broadly classified into low-temperature abrasables, typically below 540 °C (1000 °F), and mid-to-high-temperature abrasables, from 540 to 980 °C (1000 to 1800 °F).

Typical low-temperature abrasables consist of porous, aluminum-base coatings (e.g., aluminum-silicon alloys) containing polyester, polyimide, or boron nitride, and nickel-graphite coatings (Ref 3, 6). Due to the high risk of moisture in the cold compressor section, aluminum-base rather than nickel-base composites are perhaps more suitable to avoid damaging the steel case by galvanic coupling. At the high end of the temperature range, above 450 °C (840 °F), compressor abrasables might also include MCrAlY/polyester/BN (Ref 6). Other low-temperature seals that are not interacting with the blades are also part of the engine system. In these sealing areas, coatings typically run against labyrinth seals along the engine shafts in the compressor and turbine, sealing either gas or oil paths, and are usually composed of Babbitt, bronzes, and AlSi/polyimide. The seals and compressor abrasables are usually deposited by plasma, low-velocity combustion flame, or twin-wire arc spray.

Nickel-base and MCrAlY-type coatings are typically applied onto mid-turbine hot-section tip seals or shroud surfaces using thermal spray techniques. These coatings may contain softer agglomerates, such as polyester, boron nitride, hollow ceramic particles, bentonite, or graphite. Caution is recommended in the use of graphite-containing nickel-base coatings due to the risk of coating autocatalytic galvanic oxidation, which significantly reduces coating life. In some application cases, these coatings consist of a MCrAlY alloy (no addition of fugitive compounds), which is not, by definition, an abrasable. They are designated rub-tolerant coatings, offer a relatively softer rubbing surface at high temperature, and are oxidation and corrosion resistant. High-temperature abrasables and rub-tolerant coatings are applied through HVOF, plasma spray, or low-velocity combustion spray.

In an increased effort to augment turbine efficiency through higher operation temperatures, turbine manufacturers have introduced ceramic abrasable coatings into the hot section of the turbine shroud. As the industrial turbine firing temperatures are increased to augment power output and engine efficiency, recent investigations have centered on the use of thick ceramic coatings for turbine applications, due to their higher temperature resistance and thermal barrier properties. Ceramic systems with sufficient substrate backside cooling would provide a thermal barrier effect between the hot gas path and the metallic shroud surface. Considering that there is a temperature drop of 3 to 4 °C (5 to 7 °F) per 25 µm (0.001 in.) for a typical YSZ thermal barrier coating (Ref 7), the top surface temperature of a 2 mm (0.080 in.) thick ceramic abrasable coating at the flow path might reach up to 1200 °C (2190 °F), while the superalloy would remain at an acceptable lower temperature. For comparison, the maximum service temperature of MCrAlY coatings is approximately 980 °C (1800 °F), which is

on the order of the tip seal surface temperature for industrial gas turbines. Ceramic abrasable coatings are, however, hard on the blade tip; hence, it is mixed with softer materials (e.g., boron nitride, polyester) to reduce hardness and increase abrasability (Ref 8). The manufacturer can also choose to use abrasive blade tips to rub against a TBC.

Thermal Barrier Coatings

Plasma spray TBCs are of great use in the hot section of turbines, such as the combustor liner (cans), transition ring, splash plate, and fuel injector. These coatings help to increase thermal efficiency and reduce exposure temperature, thereby extending component life and generally mitigating oxidation and corrosion. In general, the bond coat must offer high-temperature protection to oxidants and contaminants that might infiltrate through the ceramic coating to the bond coating. The most frequently used ceramic coatings are zirconia-base ceramics and include:

- Yttria-stabilized zirconia or $\text{ZrO}_2\text{-Y}_2\text{O}_3$ (6 to 8 wt%)
- Ceria-stabilized zirconia ($\text{CeO}_2\text{-YSZ}$)
- Ceria- and yttria-stabilized zirconia ($\text{ZrO}_2\text{-25CeO}_2\text{-Y}_2\text{O}_3$) (2 to 3 wt%)

Calcium titanate (CaTiO_3) is also used in some applications. Tables 2 and 3 list the thermal conductivity and thermal expansion coefficient of these coatings (Ref 9).

Common bond coatings are applied by plasma spray; however, there is an increased use of HVOF processes to apply the coatings. These coatings are typically 100 to 200 µm (0.004 to 0.008 in.)

Table 2 Thermal conductivity of selected plasma sprayed ceramic coatings

Coating	Thermal conductivity, W/m · K (Btu · in./h · ft ² · °F)
$\text{ZrO}_2\text{-7Y}_2\text{O}_3$	0.79–1.30 (5.48–9.01) 0.82 (5.69) at 400 °C (750 °F)
$\text{ZrO}_2\text{-25CeO}_2\text{-2Y}_2\text{O}_3$	0.4–0.90 (2.77–6.24) 0.43 (2.98) at 400 °C (750 °F)
CaTiO_3	1.63 (11.3) at 400 °C (750 °F)

Table 3 Coefficients of thermal expansion for plasma sprayed yttria-stabilized zirconia and CaTiO_3 coatings measured at specified temperatures

Coating	Coefficient of thermal expansion, m/m · °C × 10 ⁻⁶		
	At 100 °C (212 °F)	At 500 °C (930 °F)	At 1000 °C (1830 °F)
$\text{ZrO}_2\text{-7Y}_2\text{O}_3$	7.9	8.4	9.6
CaTiO_3	10.2	11.4	12.4

thick MCrAlY coatings. The TBC of choice seems to be YSZ, due to its acceptable resistance to thermal shock and its overall durability. A combustor liner with a 380 μm (0.015 in.) thick TBC may last more than 30,000 h in a turbine at full load with metal temperature exposures up to 870 $^{\circ}\text{C}$ (1600 $^{\circ}\text{F}$).

In searching for higher-thermal-resistant coatings, the tendency in the industry for thermal sprayed ceramics is to deposit thicker coatings to increase their thermal barrier capabilities. Due to the demands for higher temperatures in gas turbine hot sections, components have increased thickness requirements of YSZ coatings to 635 to 1000 μm (0.025 to 0.040 in.), in spite of the eventual reduction of their thermal cyclic lifetime. For coating thickness increments from 300 to 635 μm (0.012 to 0.025 in.), the thermal cyclic life is decreased by approximately 20% between 1000 and 1100 $^{\circ}\text{C}$ (1830 and 2010 $^{\circ}\text{F}$) (Ref 7). It is well known that as the thickness of the ceramic coating increases, its thermal cyclic resistance decreases, due to reduced cohesion and higher stress levels within the coating. Attempts have been made to replace YSZ with newer materials with lower thermal conductivity. Alternatives of material with lower-thermal-conductivity coatings are gadolinia-yttria-zirconate, samarium- and neodymium-containing zirconates, perovskite, and pyrochlore materials. However, these might be considered at the developmental stage compared to the YSZ coatings with more than 25 years of acceptable performance in industrial applications.

The use of certain low-grade fuels leads to the condensation of vanadium and sulfate compounds, and their fused salts are extremely corrosive to high-temperature materials for which CeO_2 -YSZ coatings are more suitable. Therefore, these coatings are mostly used in aggressive corrosive environments. The cerium oxide content contributes to the coating hot-corrosion resistance, when compared to yttrium oxide, expressed as a lower solubility to molten sulfate-vanadate salts. The CaTiO_3 ceramic coatings are also used where corrosion resistance is needed, such as in fuel injector tips. The CaTiO_3 coatings show a better thermal cyclic life, in particular, at greater thickness, due to their higher thermal conductivity and higher CTE (Ref 8). It is noteworthy that the maximum exposure temperature for CaTiO_3 is approximately 870 $^{\circ}\text{C}$ (1600 $^{\circ}\text{F}$), while YSZ and CeO_2 -YSZ can be exposed to approximately 980 $^{\circ}\text{C}$ (1800 $^{\circ}\text{F}$). The sintering of the zirconia-base TBCs is a big concern when the application temperature approaches 1150 $^{\circ}\text{C}$ (2100 $^{\circ}\text{F}$). Extensive literature has been written on the sintering of the zirconia-base TBCs at temperatures of approximately 1093 $^{\circ}\text{C}$ (2000 $^{\circ}\text{F}$).

Buildup and Repair Coatings

Sometimes, it is necessary to build up a worn, damaged, or mis-machined component. The coating thickness required may vary between 0.075 and 2.54 mm (0.003 and 0.100 in.) and usually needs finishing to correct dimensional tolerances and to comply with surface smoothness requirements. The rule of thumb is to use a buildup material similar to the base material. If dissimilar materials are used, the properties of the coating and part should have a similar thermal expansion and resistance to galvanic coupling.

Buildup coatings have a wide variety of compositions, such as iron-base (stainless steel) and nickel-base (NiCrMo, NiMoAl, alloys 718 and 625, NiAl, and NiCr) materials. The preferred deposition methods are HVOF and plasma spray, due to the higher bonding strength. Applications by twin-wire arc spray are common on nonrotating components, such as ball bearing seats and shroud flanges, between stages. Some large-power-size turbines also have Babbitt bearings restored using the arc spray process.

Other Nonthermal Spray Coatings Used in the Industry

Low-pressure plasma spray MCrAlY coatings are used on blades and vanes for oxidation and corrosion protection and as a bond coating for TBCs. Simple aluminides, platinum-aluminides, or MCrAlYs deposited by physical vapor deposition or chemical vapor deposition are used on blades and nozzles for protection against oxidation and corrosion. Also, physical vapor deposition TBCs are applied to reduce part-temperature exposure. On cold sections, aluminum, aluminum-phosphates, or organic paints are used against oxidation. Honeycomb abradable seals could be brazed onto the sealing rings. Overlays, such as nickel plating, cadmium plating, and hard chrome plating, are generally used for buildup and resistance for oxidation, corrosion, and wear. There is a significant effort being made to provide alternatives to cadmium and chrome plating, due to environmental concerns regarding exposure of personnel to carcinogenic materials, such as hexavalent chromium, associated with these processes. (See the section “High-Velocity Oxyfuel Thermal Spray Coatings as a Replacement for Hard Chrome Plating on Aircraft Landing Gear” in this article for further details.)

Automotive Applications

Thermal spray coatings have been used in automotive production throughout Europe and Japan for some time and are also being used increasingly on U.S. automotive production lines. The performance of these coatings has been excellent, and applications will increase in both spark ignition (SI) gasoline-powered vehicles and diesel-powered vehicles. Figure 8 illustrates current and potential application areas for thermal spray coatings in the automotive industry.

Powertrain Components*

Hot corrosion affects various portions of exhaust systems on both diesel- and gasoline-powered vehicles. Various exhaust system components are protected from hot corrosion by the use of thermal sprayed aluminum via the twin-wire arc spray process or the wire combustion process. Figure 9 shows the application of an

*The discussion “Plasma Coating of Aluminum Alloy Cylinder Bores” in this section was contributed by P. Tommer, Sulzer Metco AG.

aluminum thermal spray coating on exhaust headers. These headers will produce a 6 to 10% increase in power on stock engines and much more on high-performance engines. On stock engines, customers have reported over a 5% improvement in gas mileage.

“Rain-drop erosion” has been known to occur on the tops of pistons due to the impingement of fuel from the injectors. Aluminum pistons in diesel engines are frequently coated with a thin, plasma sprayed molybdenum coating to circumvent this problem.

In a related phenomenon, splash-back effects from fuel impingement (on the piston) may eventually erode the periphery of the delivery ports in the injector nozzle. A frequent defense is the same molybdenum coating applied to the tips of the fuel injectors.

Galling of aluminum piston skirts results in the transfer of aluminum to the cast iron cylinder liners. If the piston rings pass over such areas, serious ring wear, or even breakage, may occur. Piston skirts are also coated with a thin molybdenum layer to prevent this.

Piston ring coatings commonly consist of molybdenum and molybdenum-bearing compounds. Coatings are usually inlaid into the ring surface so that no exposed coating edges encounter the cylinder wall. (The piston ring cants slightly within the ring glands

as it reciprocates.) In Europe, combustion wire spray is frequently used and preferred over plasma, because the added oxides play a major role in coating performance. Molybdenum trioxide has a lubricious quality that is valued in such coatings.

In the United States, other hardening agents, such as silicon, nickel, and chromium, are often added to the molybdenum, and powders are applied by APS, flame spray, or HVOF. The open microstructure, consisting of many small pores at the surface of these coatings, is of value for oil retention.

An interesting variation is the inclusion of small percentages of MoS_2 solid lubricant within the coating matrix. Such material has been added mechanically to the spray powders and offers improved performance where engine loads or oil deprivation tend to cause galling. However, MoS_2 is known to break down at temperatures above 420°C (800°F) and could form sulfurous acids in the engine oil; care must be taken to prevent this.

Matching cylinder liner and piston ring coatings requires rigorous study. Variations of engine design and operating conditions can influence the coating selection. In general, it is desirable that the rings exhibit greater wear, because it is more cost-effective to replace rings than wet liners.

Cylinder Bore Coatings in Diesel Engines. Cylinder bore coatings applied by thermal spray processes are now replacing hard chromium plating on the larger (wet) liners used in diesel engines. Initially, thermal spray coatings were used to salvage these liners during overhaul, when severe wear made electroplating impractical. Again, molybdenum coatings similar to those used for piston rings are used for this application. More recently, however, HVOF-sprayed WC/Co and WC/Co/Cr are being explored because of their increased performance over electroplated hard chrome.

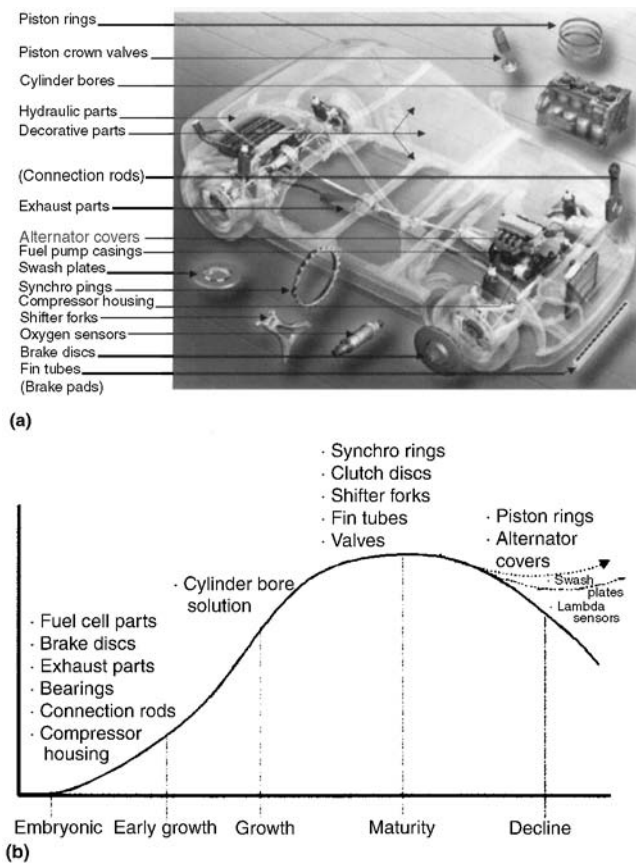


Fig. 8 Automotive applications for thermal spray coatings. (a) Locations where thermal spray coatings are applied in automobiles. (b) Product life cycles for thermal spray coatings in the automotive industry. Source: Ref 10

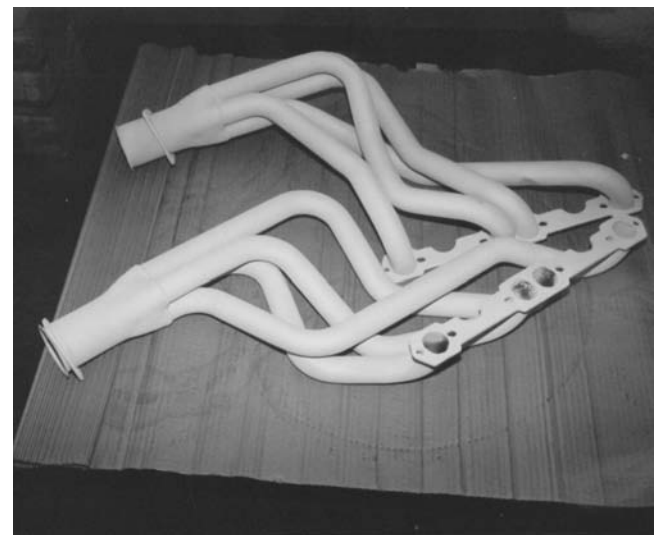


Fig. 9 Exhaust headers metallized with aluminum using the wire combustion process. The aluminum provides protection from heat and corrosion. Courtesy of ASB Industries Inc.

The inherent porosity of thermal sprayed coatings brings an advantage over chrome plating and other hardfacings, providing greater oil retention. Engine life tests have demonstrated longer service, and the worn liners (diesel) are easier to rebuild. These coatings are not generally inlaid but extend over the full length of the cylinder.

Plasma Coating of Aluminum Alloy Cylinder Bores. Cast aluminum-silicon cylinder bores are being specified increasingly for automobile internal combustion engines to reduce vehicle weight, thereby increasing fuel efficiency and reducing emissions. Aluminum-silicon cylinder bores must be treated to provide sur-

faces with sufficient wear resistance. A plasma coating process called Rotaplasma has been developed that offers economic, technological, and environmental advantages for this application (Fig. 10). The process was introduced in 2000 for automobile production in Europe. As of 2004, the process has been qualified for production of several diesel and gasoline engines (Table 4).

The plasma coating deposit produced using the Rotaplasma technology provides several advantages:

- Reduction in the coefficient of friction against the piston ring by as much as 30% versus that of cast iron cylinder bores with

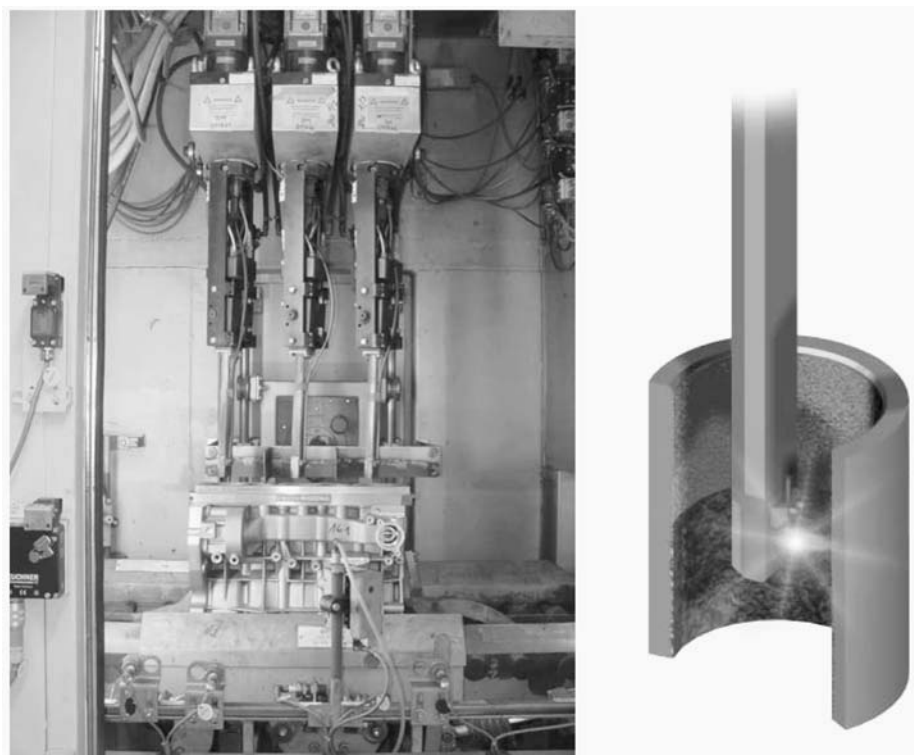


Fig. 10 Setup for Rotaplasma processing of aluminum-silicon cylinder bores. The plasma gun is rotated up to 200 rpm. Coating thicknesses range between 150 and 200 μm . Courtesy of P. Tommer, Sulzer Metco AG

Table 4 Automobile engines currently using the Rotaplasma process for coating of cylinder bores

Engine	Engine size		Power		Torque		Automobile or engine model
	L	in. ³	kW	hp	N · m	lbf · in.	
Diesel							
V 10 TDI VW	5	300	230	310	750	6640	Volkswagen Touareg
EA 115 VW	2.5	150	130	170	400	3540	Volkswagen Touareg, Van T 5
Gasoline							
EA 111 VW	1.4	85	77	100	Volkswagen Lupo
Go-Kart racing	0.25	15	20	30	Racing Biland
V 10 racing engine	>600	>800	Formula 1
Bugatti W 16	>700	>940	Custom

lamellar graphite surface structure. The reduced friction enables a significant decrease in engine fuel consumption—between 2 and 4%, depending on the type of engine.

- Very low levels of oil consumption by the engine
- A very low surface wear rate (approximately 5 nm/h of service)
- Smaller and lighter engine blocks are possible, due to the elimination of cast iron liners.

These characteristics are obtained with a particular topography of the cylinder surface that is created by diamond honing of the surface after spraying (Fig. 11).

At present, different spray materials are being used for application. The most common are iron/molybdenum composites and iron/iron oxide composites. The spray time for a cylinder bore in a conventional car engine (80 mm, or 3.2 in., in diameter; 120 mm, or 4.8 in., in length) is only approximately 1 min.

The cost of coating for a cylinder bore depends on the production volume; the greatest cost efficiency can be obtained at a product volume of at least 8000 bores/day. At this production level, the cost (in 2004) is between \$2 and \$3 (U.S.)/bore.

Exhaust Valves. The operating environment is particularly severe for exhaust valves, which is why metallurgically bonded structures are usually required. However, combustion flame sprayed and fused coatings of self-fluxing nickel-chrome-silicon-boron (AWS 4745 and related compounds) have been used by

Japanese automobile companies on valve seats and valve facings. These materials are classified as brazing compounds but are sold for spray-and-fuse applications too, where they retain excellent hot-corrosion resistance and wear properties. The same coatings have been used on camshafts in the United States to provide wear resistance during dimensional restoration.

A more recent innovation is the flame or plasma spray application of iron coatings onto aluminum valve lifters to improve their sliding wear performance. The advantage offered is the requirement of less energy by the lighter system through the use of aluminum alloys and the thermal spray wear-resistant surface.

Crankshaft coatings have been largely limited to dimensional restoration of worn parts. Rebuilding of crankshafts for off-road equipment has also been performed with combustion flame spray plus fusing of the nickel-chrome-boron-type materials mentioned earlier. Some rework with air plasma spray applications of molybdenum-nickel-chrome systems has also been performed.

Transmission and rear end gear clusters, as used on a number of passenger cars, frequently receive thin APS molybdenum coatings on the gear faces, lessening the friction component and producing smoother and quieter movement.

Other powertrain components are often rebuilt using thermal spray technologies. Clutches have been refaced with both plasma and combustion-wire-sprayed bronze. Transmission shafts have been rebuilt with both stainless and molybdenum coating systems deposited either by combustion powder or wire and air



Fig. 11 Rotaplasma processing. The cylinder bores in the engine block (top figure) are processed in the following stages (bottom figure, from left to right): premachined, surface activated prior to coating, Rotaplasma coated, and diamond honed. Courtesy of P. Tommer, Sulzer Metco AG

plasma spray. Turbocharger housings have been coated with aluminum-silicon-graphite by plasma spraying or HVOF. Abradable coatings derived from aerospace and industrial turbine technology have been applied to turbochargers, pumps, and compressors to provide clearance control. Materials used include aluminum-polymer, nickel-graphite, and CoNiCrAlY/BN/polymer composites.

Thermal Barrier Coatings

Thermal barrier coatings technology has been largely borrowed from aerospace and land-based turbine engine applications described earlier in this article. Until very recently, interest in TBCs has been on behalf of high-performance SI engines as used in motor sports or in low-heat rejection engines (diesel) for military applications. There seems to be conflicting evidence as to the benefits provided by TBCs on gasoline-engine passenger cars. If TBCs are ever to be used in high-volume production of gasoline-powered vehicles, the economics will have to be addressed.

Partially insulated diesels make use of less drastic applications of TBCs that have originated from laboratory and theoretical studies. These studies suggest that use of thinner TBCs plus water or air cooling will yield a performance gain. Figure 12 illustrates this concept, based on increased temperatures (and gas pressures) at the moment of combustion, but with lower average cylinder temperatures.

Coatings on such diesels are usually from 0.5 to 1 mm (0.02 to 0.04 in.) in thickness and are confined to surfaces that are not subjected to other metal contact or sliding wear. The economics for commercial use are attractive, because there is evidence that such coatings bring substantial gains in engine performance and lifetime between rebuilds. There is also much evidence that supports

the reduction of pollution (hydrocarbons, NO_x) by diesels so treated.

Another use of TBCs on diesels is for components subjected to high-temperature fatigue. Piston crowns are often coated, not for purposes of engine performance but to extend component life.

High-performance engines have used TBCs on piston crowns, cylinder heads, valves, exhaust ports, and, if naturally aspirated, intake ports too. Such coatings are usually less than 0.2 mm (0.008 in.) thick and are frequently subjected to stresses from flexing components. Thermal barrier coatings that are too thick may raise the average cylinder temperature, causing early detonation.

A factor in the TBC life cycle concerns the large valves (up to five in number) used in these engines. Engine geometry can result in very narrow regions of exposed head casting between intake and exhaust valves. This narrow zone is subject to extreme thermal gradients that vary laterally within the coating. Use of thicker bond coats (0.125 to 0.150 mm, or 0.005 to 0.006 in.) with thinner ceramic topcoats can aid in reducing this problem.

Aside from increased performance (horsepower and torque), TBC-insulated SI engines generally operate with lower oil temperatures that result from lower component temperatures, which means longer component life. In addition, radiator sizes can be reduced, and the effective cross section may improve the aerodynamics of the vehicle.

Manifolds internally coated with TBCs have been shown to produce significant reductions in catalyst light-off time. The problem is obtaining such coatings in currently manufactured manifolds—virtually an impractical issue. Such components have to be carefully designed for stamping operations, which fashion the component as two clamshell halves. After installation of the TBC, these halves must be welded together (gastight) by laser. Additionally, there is a fear of long-term performance. Should the TBC spall or delaminate, there is a possibility that the debris would be injected back into the engine, causing wear damage.

Heat management in engine compartments and elsewhere can be solved by proper use of TBCs. Modern engines are usually crowded into their space, and airflows may be inadequate under certain conditions. Thermal barrier coatings applied externally to manifolds and other exhaust system components of racing machines have been found to aid in temperature reduction of body and chassis components and engine mounts as well as provide improved driver comfort (Fig. 13).

Thermal barrier coatings have also been applied to brake pads to insulate them from the hydraulics, thereby improving brake fade characteristics. At least one production motorcycle has employed this technique.

Turbochargers offer significant opportunities for TBCs. These units usually closely couple the exhaust gases with the incoming air charge, adding heat to that already developed by compressing the air. The use of TBCs benefits the turbocharger performance.

Disc Brakes. The use of TBCs on disc brakes eliminates brake fade in high-performance applications. Frequent and severe braking adds heat through the caliper piston (usually aluminum) to the brake fluid, which results in the fluid boiling and in “spongy” brak-

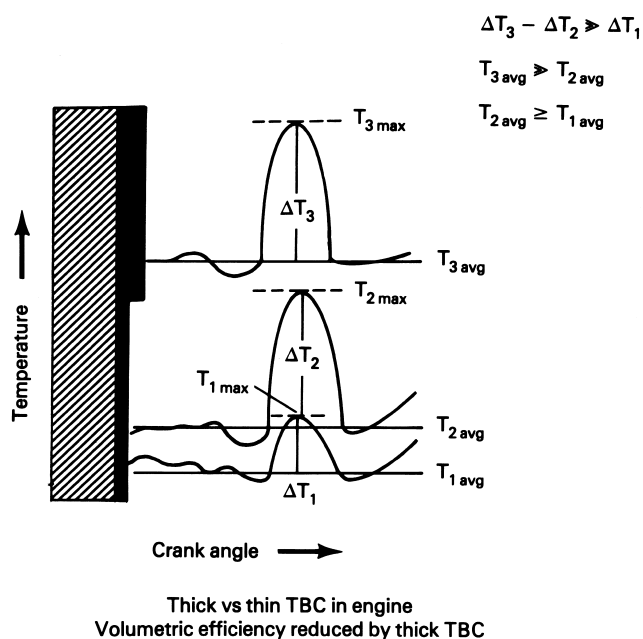


Fig. 12 The effects of thermal barrier coating (TBC) thickness on average and peak cylinder temperatures

ing response. Several automotive original equipment manufacturers have used TBCs on either the piston tops or the backing plates of the brake pads to interrupt the heat flow and eliminate the fade problem.

Aluminum Metal-Matrix Composite (MMC) Brake Discs

Aluminum-base MMCs have been developed for brake system applications (Ref 11). The weight and inertia of brake discs are cut in half when aluminum MMCs such as AlSi-20 vol% SiC are used instead of cast irons. However, the necessary high coefficient of friction cannot be achieved with an uncoated aluminum MMC disc. Initially, metal alloy coatings that would conduct heat quickly were sought. However, these actually promoted melting of the underlying aluminum alloy matrix substrate. The result was the development of a modified powder-based alumina coating that optimizes thermal conductivity and friction properties. These plasma sprayed powders have been thoroughly tested in motor sport competition and are used extensively in Grand Prix race cars.

Body and Chassis Applications*

Arc Spray for Automotive Weld Seams. Wire arc spray is an excellent process for applying metallic coatings for dimensional restoration. The high spray rates, good bond strength, and ease of automation make it ideal for automated production applications.

The spraying of automotive weld seams is one such highly successful application. In manufacturing an automobile, sheet metal is stamped into the side panels and roof panel. These are then joined together using a spot weld or other welding technique. The weld seam is then usually covered with a plastic molded strip along the roof panel perimeter, or, in the case of luxury model cars, a manual braze filler is applied to fill the joint and then ground smooth and flat. While the smooth metal joint is much

more aesthetically pleasing than the plastic strip, the manual brazing step is very slow and labor-intensive, making it too costly for most car models.

The wire arc spray process was adapted very successfully to solve this problem and to provide a low-cost, automated filler for the weld seam. The automotive people refer to it as spray brazing.

The weld seam is spot welded in the normal fashion. It then advances into the spray booth, which is part of the production line. The seam is robotically grit blasted, then four to six arc spray guns, mounted to robots on either side of the car, fill in the seam with a silicon bronze or aluminum bronze coating. The seam is then ground finished and ultimately painted. Figure 14 shows a body panel seam after postcoating grinding and buffing. The end result is a high-quality, low-cost, and very attractive weld joint that has now been incorporated into numerous assembly production lines of Chrysler and other automotive manufacturers.

Steering-stop brackets are welded to front wheel “A” frames and provide the steering radius limits for vehicles with rack-and-pinion steering. The brackets contact a rounded projection mounted to the frame (at the steering limits), preventing the wheels from turning into the car body. In many cars, maximum turning results in unacceptable shuddering and noise. Lubrication of the brackets, because they are unprotected on the “A” frame, is impractical, and the continual operation of the steering to the limits actually erodes and corrodes the bracket, limiting its useful life. Twin-wire arc spray is used to apply thin layers of zinc to the contact points of the brackets. The zinc, when wet, provides a lubricated surface for the contact and galvanically protects the bracket from further corrosion.

Biomedical Coatings for Orthopedic and Dental Implants

This is a relatively new class of applications for thermal spray coatings, making use of inert chamber plasma and inert chamber twin-wire arc spray to produce functional coatings. Both orthopedic and dental implants receive these coatings that are designed to

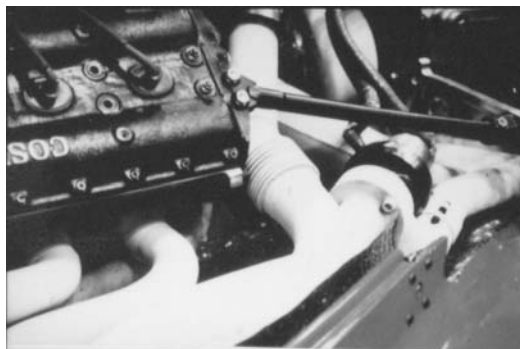


Fig. 13 High-performance race car manifold coated with a thermal barrier coating for heat management



Fig. 14 Body seam panel that was coated by the wire arc process after postcoat grinding and buffing. Courtesy of W.J. Lenling, Thermal Spray Technologies Inc.

*The discussion “Arc Spray for Automotive Weld Seams” in this section was contributed by Robert H. Unger, Praxair TAFA Inc.

aid fixation of the devices in bone tissue. Many of these implants are “fixed” without any adhesive, allowing bone tissue to make contact with porous inert or bioactive surfaces.

Orthopedic and Dental Community Issues

Orthopedic and dental markets have exhibited rapid growth due to new implant technologies, coupled with an aging population. These implants are direct replacements for various bone joints and teeth, and records point to acceptable service life, with excellent patient acceptance. Material and design advances account for much of the life extension obtained.

Two types of metal families are recognized as being compatible with human tissue: a Co-Cr-Mo alloy available in cast or wrought forms, and titanium materials, specifically, commercial-purity titanium (CP-Ti), the alpha-beta alloy Ti-6Al-4V, and more recently introduced beta alloys such as Ti-6Al-7Nb, Ti-13Nb-13Zr, and Ti-12Mo-6Zr-2Fe. Porous coatings deposited on load-bearing implant devices made from these alloys facilitate implant fixation

and bone ingrowth and replace the use of bone cements and screws for implant fixation. Figures 15 and 16 show a variety of implant devices that have been coated with biomedical coatings.

Biomedical coatings are subject to rigid standards and practices. All materials must be certified as to their exact nature and impurity content. Care must be taken with devices and coatings during application, so that contamination with masking material, cleaning agents, and other matter does not occur.

Bioinert Coatings

These coatings are benign in nature; that is, there is no activity between them and bone or soft tissue. Titanium-base coatings predominate, perhaps because of an allergic response by approximately 18% of the population to Co-Cr-Mo.

CP-Ti and Ti-6Al-4V are commonly sprayed onto both titanium and cobalt alloy substrates for bone-bonding mechanisms. They are deposited with a high-porosity content (approximately 30%) and with pore sizes upward of 50 μm (0.002 in.). Bond coats



(a)



(b)

Fig. 15 Examples of coated titanium alloy implant devices. (a) Acetabular shell featuring a machined Ti-6Al-4V substrate with a commercial-purity titanium (CP-Ti) coating deposited by inert gas (argon) wire arc thermal spraying. (b) Near-net forged and final machined hip stem made from Ti-6Al-4V and coated with CP-Ti by inert gas (argon) wire arc thermal spraying. The rough coatings on both devices have an average thickness of 0.75 mm (0.030 in.). The desirably rough coating allows for good mechanical locking of the device with bone ingrowth. Courtesy of G. Hannah, Stryker Orthopaedics

with different characteristics may also be used. The desired coating characteristics are obtained by careful design of the process parameters and powder size control.

Air plasma spray has been used to create oxide-containing coatings, which are subsequently vacuum heat treated to remove the unwanted oxygen. There may be some reduction in the fatigue strength of the titanium alloy component and, in the worst case, the development of some unwanted beta phase in the metallurgy. Care must be taken when using this method on Co-Cr-Mo alloys, because higher-temperature heat treatments can develop metal-oids whose behavior in the human body are unknown.

The preferred method of spraying these materials is by use of inert gas chambers or LPPS. This avoids the need for subsequent postdeposition heat treatments. In general, coatings up to 1.25 mm (0.050 in.) thick can be applied without significant impact on fatigue properties.

A finer cut of powder is generally used for dental implants, because a less aggressive morphology is desired. In some cases, this layer is used as a bond coat for subsequently applied ceramic coatings.

The Co-Cr-Mo alloy has been used in bead geometry to provide sintered porous coatings on like substrates. However, the same material can be sprayed as a porous coating by plasma. The same porous morphology found in the titanium coatings is desired.

Bioactive Coatings

These are ceramic coatings with compositions similar to that of bone tissue (calcium phosphates). The material interacts with bone to promote bonding interfaces. These ceramics are applied by air plasma, HVOF, and LPPS, usually at thicknesses of 40 to 80 μm (0.0016 to 0.0032 in.).

Hydroxyapatite (HA) is a calcium phosphate ceramic $[\text{Ca}_{10}(\text{PO}_4)_6(\text{OH})_2]$ that exhibits strong *activity* for joining to bone

tissue. As a coating, it is perceived as a dynamic interface between surgically implanted orthopedic and dental devices and bone. The Food and Drug Administration (FDA) is active in developing certain requirements for coating characteristics. The situation is still quite fluid, and the technology is advancing in understanding both the benefits and the methods of HA application.

Plasma spraying (both air plasma and LPPS) is the current primary method of application, although HVOF is also being researched. Problems exist, however, in obtaining pure stoichiometric powders and deposits. Contributing to these problems is the potential existence of vacancy states, which are not easily detected in HA. An important requirement is that of whiteness. There is a perception in the biomedical community that whiteness denotes purity. Trace elements in amounts that do not affect performance can color both the feedstock powder and the resulting deposits. These are unacceptable in the market.

In vivo testing has now shown that a high degree of crystallinity (as determined by x-ray diffraction) must be present in the plasma sprayed deposits, which contrasts sharply with the realities of a rapid solidification process. Careful development of coating parameters can produce high crystallinity contents (~95% for LPPS, ~80% for HVOF, and ~75% for air plasma), but this may be at the expense of reduced bond strength or coating hardness.

The HA coatings used in the orthopedic community can be applied directly to component surfaces (Fig. 16), over a textured bond coat, or over the highly porous metallic coatings made by both plasma spray processes or sintered beads. At present, only the highly crystalline coatings are desired for maximum coating effectiveness.

There are, however, implant designs in the dental market that are thought to be more effective as the coating resorbs. Hydroxyapatite, therefore, may be sprayed so as to have a higher amorphous phase content. Hydroxyapatite coatings used in dental applications may be used directly on the implant or over plasma sprayed textured coatings.

Tri-calcium phosphate is a compound known to resorb in vivo and is therefore used for appliances designed for these actions. With the advent of HA, its use has greatly declined.

Ceramic and Glass Manufacture

Three areas of importance for thermal spray coatings in ceramic and glass manufacture are glass contact coatings, mold coatings, and the use of TBCs for asbestos replacement.

Glass Contact Coatings

Molten glass is one of the most corrosive media the materials community can experience. Only a few materials are capable of contact for extended periods in many common glass composition melts. Thermal spray coatings have applications in several of the areas associated with production and transfer of molten glass.

Precious metals such as platinum and certain of their alloys are used extensively for long-term contact with molten glass. The industry is possibly the single largest consumer of platinum and is accustomed to measuring the total cost of new plant construction

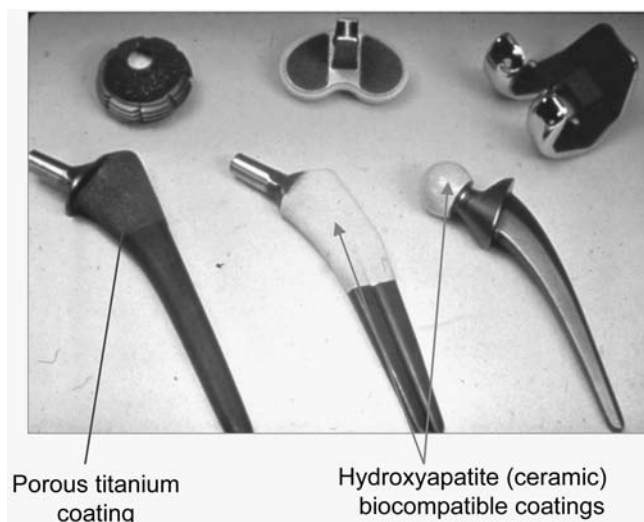


Fig. 16 A variety of implant devices for biomedical/biocompatible applications. Hip implants with bioinert (porous commercial-purity titanium) and bioactive (hydroxyapatite) plasma spray coatings are shown in the foreground. Courtesy of R. Knight, Drexel University

by the cost of the precious metals consumed. These metals also exhibit high vapor pressures, so significant percentages of the materials are actually lost during the course of glass production. Thermal spray ceramics deposited over the metal surfaces retard this action. Although the ceramic overlays may be damaged over a period of time, requiring reapplication, the value represented by the saved precious metals makes it worthwhile to treat these expensive tools.

Plasma sprayed coatings have been used in lieu of heavier platinum and platinum alloy sheets. Such coatings are generally produced in inert atmosphere chambers, where oversprayed powder can be retained and salvaged. Usually, the coated items are small in size but most critical to the glass processing. Among the examples are stirrer paddles, agitator shafts, thermocouple wells, and nozzles.

Molybdenum is a second material system that performs well in contact with certain molten glass compositions. Stirrers, mixing paddles, and some mold surfaces have been coated with plasma sprayed molybdenum or molybdenum alloy compositions.

Glass transfer troughs are used to handle “gobs” of the product in molten form, passing them to the mold. These troughs are generally made of steel or aluminum, with water-cooled walls to aid in handling the product. Owing to the high glass temperatures and its corrosive nature, high wear occurs in glass transfer troughs. The Mo-Ni-Cr coatings, with trace additions of MoS₂, are used to minimize this wear and reduce the rolling friction of the glass as it transfers to the mold. Water cooling of these channels keeps the coating below 800 °C (1470 °F), where breakdown of the disulfide is apt to occur. These coatings are applied by APS.

Mold Coatings

A variety of tools are used to shape glass into final products. Glass composition affects the choices of coating material, but high-temperature alloys with good oxidation resistance predominate.

There are situations in glass molding that are similar to those experienced in processing molten metals. Abrasive wear, thermal fatigue, inappropriate chilling of the melt, and corrosive action at grain boundaries in the molds occur, and many of these troubles can be reduced or eliminated through the use of coatings.

Flame-spray-and-fused NiCrBSi coatings, with possible additions of metallic carbides, have a good history of high-temperature wear protection in this industry. Air plasma sprayed Inconel (625 and 718) has been used to restore dimensions to a variety of glass-molding hardware. Thermal barrier coatings have also been used to improve thermal conditions for better molding and equipment life extension.

Some of these molds are in the form of pocket rollers, which entrap and squeeze high-viscosity melts into appropriate shapes. Such rollers thermally tax the bearings as well as see high thermal stresses. Coatings of NiCr containing higher-temperature carbides, such as Cr₃C₂ and TiC, have proved beneficial.

Asbestos Replacement

Asbestos has been used very extensively in the glassware industry in the past as a contact medium for hot product. Setter plates are trays used for heat treating glassware or for fire polishing. His-

torically, asbestos was used as an insulating blanket on these and other hot glassware carriers. Asbestos can be replaced with TBCs (magnesia-stabilized zirconia) that minimize the thermal gradients that would otherwise cause damage to the end product.

Corrosion Applications for Marine- and Land-Based Infrastructure

Any discussion on coatings ultimately focuses on their use as barriers to corrosion. Such coatings are usually considered as having two key attributes: ease of application with no through porosity or voids, and composed of a material that is totally inert in the environment to which it is exposed. These attributes are viewed as essential.

Historically, the maintenance of metallic infrastructure in the United States has focused on organic coatings, that is, various epoxies, polyurethanes, paints, and zinc silicate coatings that can be applied onto steel structures by dipping, brushing, and spraying. These treatments are usually viewed as impervious when properly applied, but they all are subject to the rigors of exposure to the weather and the corrosive environment. Thus, they still require frequent replacement. In the past, little consideration was given to coating infrastructure with metallic thermal spray coatings because the through porosity necessitates the use of some organic material as a sealer, and the overall application costs were considered substantially much higher.

There are exceptions, however. For example, zinc coatings applied by hot dip galvanizing or electrogalvanizing are commonly found on exposed infrastructure. These coatings work, not because they are impervious, but because they are sacrificial to the substrate, as discussed subsequently. Both thermal spray zinc and aluminum coatings are now commonly applied to infrastructure, and, as is discussed as follows, they are considered cost-competitive with other coating systems.

Other thermal spray materials commonly applied to infrastructure are polymer coatings. These coatings are increasingly replacing organic coatings (paints). Applications for polymer thermal spray coatings are described in the section “Recent Developments in Thermal Spray Application Technology” in this article.

Noble versus Sacrificial Coatings

Metallic coatings can be classified as being either noble or sacrificial, depending on their relationship to the substrate in the galvanic series (Table 5). Considering ferrous substrates, the term *noble* implies that the coating is nobler than iron in the galvanic series. Examples include nickel, stainless steels, and copper. If a galvanic couple is formed in an electrolyte, a voltage potential will be developed between the two metals. Completion of the electrical circuit effectively creates a battery, and current will then flow, which accelerates the corrosive attack (dissolution) of the less noble ferrous substrate, eventually undermining the coating (Fig. 17a). Noble coatings must thus be sealed in some manner to prevent the formation of a galvanic cell, if the goal is to protect the ferrous (or less noble) substrate.

It is inevitable, regardless of the thermal spray process used, that some level of porosity will exist in sprayed coatings. These pores present many opportunities for galvanic cell formation with respect to the substrate. If a noble coating is applied (e.g., a stainless steel), these pores and any exposed edges (areas where the substrate is not coated) will be the determining factor in coating performance. At all exposed interfaces (exposed to the electrolyte), a galvanic cell will immediately hasten the corrosion of the ferrous substrate. The corrosion rate is usually enhanced by the depletion of oxygen at the interface. Local corrosion will cause a gap between the two metals, and O_2 will be released, resulting in a preferential corrosion known as crevice corrosion. Its effect will be to debond the noble coating in large sheets. This is why sealers are so important. However, it is very difficult to seal all of the porosity and exposed edges (another topic) on ferrous infrastructure incorporating welds, eyebolts, rivets, and angular geometries that hinder application.

Sacrificial coatings, on the other hand, make use of materials that are less noble than the substrate; thus, the direction of galvanic

current is reversed, so that attack is accelerated in the coating and not the substrate (Fig. 17b). Under these conditions, the substrate is said to be *cathodically protected* by a coating that is *sacrificial*. Common examples of such coatings are zinc, cadmium, and aluminum. Provided that adequate current flows and the coating remains in good electrical contact with the substrate, protection of the base metal occurs for the life of the coating. Thicker coatings provide longer life, based on their sacrificial wear rates.

The degree of porosity in sacrificial coatings is no longer as critical an issue as it was for the noble coating, although many applications still call for the use of a sealer. In practice, even incomplete coverage still benefits the substrate. The geometric area of the sacrificial deposit relative to substrate geometry plays an important role. With zinc coatings on steel exposed to low-conductivity fresh water, for example, a coating defect (exposing the steel surface) measuring 3.2 mm (0.125 in.) in diameter will show some rust in the center of the defect. In seawater (a very good conductor), however, the zinc will protect the steel to a distance of several decimeters (or feet) from the deposit. The difference in behavior is the result of adequate current densities in the higher-conductivity fluid, which extends protection over much greater distances. In either electrolyte, minor pinholes are not significant in the overall scheme of things, and exposed edges are well protected.

An appropriate example of the “throwing power” of zinc is found in the zinc coatings applied to the steering-stop brackets discussed in the “Automotive Applications” section in this article. A very small patch with an area $<13 \text{ cm}^2$ ($<2 \text{ in.}^2$) sacrificially protects more than 0.09 m^2 (1 ft^2) of exposed steel on the “A” frame. The water to which the units are exposed contains salt from the roads and is thus more highly conductive than fresh water.

Aluminum coatings are frequently used in industrial and seawater applications, where its wear rates may exceed zinc but its higher hardness is beneficial in the erosion caused by salt spray.

Table 5 Galvanic series of selected commercial metals and alloys in seawater

Noble or cathodic end of series

Platinum
Gold
Graphite
Titanium
Silver
Chlorimet 3 (62Ni-18Cr-18Mo)
Hastelloy C (62Ni-17Cr-15Mo)
18-8Mo stainless steel (passive)
18-8 stainless steel (passive)
Chromium stainless steel 11–30% Cr (passive)
Inconel (passive) (80Ni-13Cr-7Fe)
Nickel (passive)
Silver solder
Monel (70Ni-30Cu)
Cupronickels (60–90Cu, 40–10Ni)
Bronzes (Cu-Sn)
Copper
Brasses (Cu-Zn)
Chlorimet 2 (66Ni-32Mo-1Fe)
Hastelloy B (60Ni-30Mo-6Fe-1Mn)
Inconel (active)
Nickel (active)
Tin
Lead
Lead-tin solders
18-8Mo stainless steel (active)
18-8 stainless steel (active)
Ni-Resist (high-nickel cast iron)
Chromium stainless steel, 13% Cr (active)
Cast iron
Steel or iron
Aluminum alloy 2024
Cadmium
Aluminum alloy 1100
Zinc
Magnesium and magnesium alloys

Active or anodic end of series

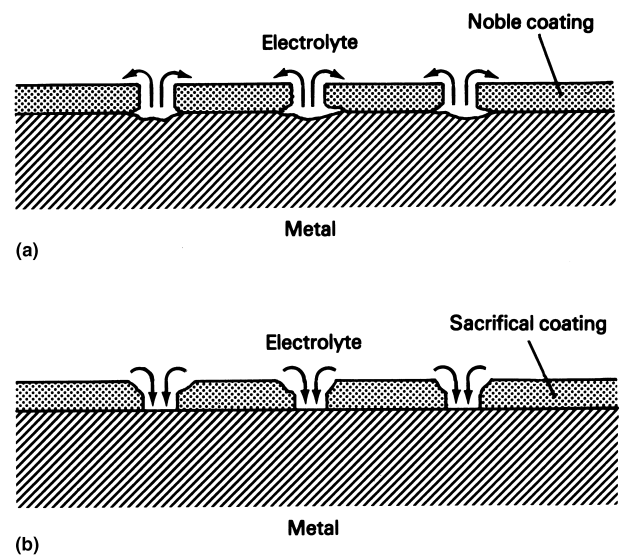


Fig. 17 Schematic of current flow at defects in noble (a) and sacrificial (b) coatings

An example of this is ship decks to create nonslip anticorrosion coatings.

Sacrificial Coatings for Infrastructure and Industry (Ref 12)

Experience with sacrificial thermal spray coatings for infrastructure applications extends back to the 1930s. Sluice gates and canal lock gates that have been zinc coated have remained in perfect condition with virtually no maintenance for decades; the St. Denis Canal Lock Gates in France, coated in the early 1930s, are an outstanding example. The locks of the Panama Canal have also been zinc sprayed. In the United Kingdom, the suspension chains and other components of the Menai Straits Bridge were zinc sprayed just before World War II. During the war, the bridge received no maintenance. When it was inspected after the end of the war, the sprayed areas of the bridge were found to be in excellent condition. The steel deck structure, on the other hand, which had been painted but not sprayed, was rusting. The obvious reduction in required maintenance of the bridge prompted the British to apply sprayed metal coatings to numerous other road and railway bridges over the past 40 years. In the United States, metal sprayed coatings are presently being specified and used by departments of transportation in Florida, Connecticut, California, Oregon, and Ohio.

Examples of some of the steel structures that are protected from corrosion by sacrificial coatings include:

- Buildings
- Bridges (Fig. 18)
- Towers
- Radio and television antenna masts
- Steel gantry structures
- High-power search radar aerials
- Overhead walkways
- Railroad overhead line support columns
- Electrification masts
- Tower cranes
- Traffic island posts
- Street and bridge railings

In addition, these coatings (most notably, aluminum) are also used extensively in ship applications, such as topside weather equipment, machinery spaces, and interior wet spaces.

The use of thermal spray metallization makes it possible to achieve fully effective, maintenance-free protection against corrosion and corrosion-enhanced mechanical failure of steel structures for more than 30 years. Fifty-year protection effectiveness has been documented in rural environments and more than 20 years in harsh industrial, urban, and coastal regions. On an overall cost basis that includes maintenance, thermal spray metallization has long been competitive with comparable corrosion-protection systems. Recent studies indicate that thermal spraying is now competitive on an initial cost basis alone. The conservative lifetime for a 255 μm (10 mil) thickness of zinc (or zinc-aluminum alloy) is approximately 25 years, which can be extended 15 years by the application of a vinyl paint as a topcoat to seal any porosity. Ini-

tial application costs of thermal spray zinc (in 1998 U.S. dollars) are \$10/ft or \$0.40/ft² per year for 25 years.

Cathodic Protection. In the case of cathodic protection of steel reinforcement in concrete, the thermal spray coating is used as an anode. Zinc is the most common material for this purpose, although titanium has also been used. The coating is applied directly to the concrete substrate. The type of connection to the reinforcement depends on the cathodic protection system. It is



Fig. 18 Three views of a bridge that was thermal spray coated with a Zn-15Al alloy. Courtesy of ASB Industries Inc.

more common to use impressed-current systems, although galvanic systems with zinc may also be effective in situations where the concrete exhibits low resistivity. Advantages of thermal sprayed anodes include ability to cover complex surfaces, uniform current distribution, and redundancy of an overlay as required with alternative mesh systems. Methods used to thermal spray zinc onto concrete include oxyacetylene wire flame spraying, twin-wire arc spray, and single-wire arc plasma spraying. These methods may form coatings that display different properties and process economics. Plasma and arc spraying have been used to produce titanium anodes.

Performance of Sacrificial Coatings in Atmospheric and Immersion Environments (Ref 13, 14)

Zinc and aluminum coatings both provide excellent protection in a wide variety of marine and industrial environments. In general, aluminum corrodes less rapidly than zinc in highly acidic conditions, but zinc performs better than aluminum in alkaline conditions. Aluminum is chosen for the protection of steel in chemical plants or in other applications in which the temperature is likely to exceed 120 °C (250 °F). Sprayed zinc coatings are used in coal mines because of the possibility of impact spark generation between aluminum and rusty steel (see the discussion of thermite sparking hazard in the section “Problems to Consider in Sacrificial Coatings” in this article). Zinc is normally the preferred metal for the protection of steel in fresh, cold waters; aluminum is used in aqueous solutions above 65 °C (150 °F).

Thermal spray zinc and aluminum coatings are progressively supplanting both painting and hot dip galvanizing for reasons of effectiveness and economics. Thermal spray coatings have a predictable life, require a single application, protect damaged areas cathodically, have good abrasion resistance, and do not require drying.

The size or shape of the structure to be protected is not a limiting factor. Components can be metal sprayed in-plant or on-site, and automation is possible for long runs of identical work. The

thickness of the metal coating can be controlled according to the degree of protection required. The life of the metal coating is proportional to the coating weight per unit area. For a very long service life or in highly corrosive conditions, it is possible to increase the coating thickness to enhance corrosion protection.

Sprayed zinc or aluminum coatings can be applied in thicknesses ranging from 50 to 500 µm (2 to 20 mils). Furthermore, parts that are not to be coated can be masked during spraying, and the coating thicknesses can be varied in different areas of the same structure. Areas of coating discontinuity or insufficient thickness can easily be rectified by additional spraying.

Both aluminum and zinc coatings have good adhesion to grit-blasted steel. Spraying does not cause excessive heating of the substrate; therefore, there is no distortion or effect on the mechanical properties of the steel. All grades of steel can be sprayed.

A limited amount of porosity (usually, <15%) is an inherent feature of thermal spray coatings. In the case of active metal coatings, such as zinc or aluminum, porosity is not a deficiency. Pores will not result in substrate attack, because of the protection afforded by galvanic action. In fact, the natural surface porosity provides an excellent base for sealers or topcoats, where these are desired for decorative purposes or to extend the life of the coating.

The selection of a zinc or aluminum coating system depends on the service environment and the desired life. Table 6 lists such coatings for atmospheric and immersion service but is intended to serve only as a guide in choosing a coating system.

Zinc-Aluminum Alloys and Aluminum MMCs. Corrosion protection can also be provided by the use of zinc-aluminum alloys, such as Zn-15Al. This alloy does not have the performance record of pure zinc or aluminum, but studies in Europe and Japan and, more recently, in the United States suggest that such alloys combine the advantages of zinc and aluminum and may outperform both in certain environments. The Zn-15Al alloy is now available in either wire or powder form. Installed costs are comparable to either pure zinc or aluminum.

Where resistance to wear, abrasion, or both is required in addition to corrosion protection, an aluminum MMC should be con-

Table 6 Thermal spray coatings for atmospheric and immersion service

Exposure environment	Thickness of coating systems for 20-year expected life							
	Unsealed zinc		Sealed zinc		Unsealed aluminum		Sealed aluminum	
	µm	mils	µm	mils	µm	mils	µm	mils
Atmospheric								
Inland (nonpolluted)	150	6	150	6	150	6	100	4
Inland (polluted)	150	6	150	6	150	6	150	6
Coastal (nonpolluted)	250	10	150	6	150	6	150	6
Coastal (polluted)	350	14	250	10	250	10	150	6
Immersion								
Seawater splash zone	250	10	150	6
Seawater immersion	250	10	150	6

Source: Ref 13

sidered. This material consists of an aluminum matrix filled with 10 vol% Al_2O_3 particles (8 to 10 μm in diameter).

Tables 7 and 8 give current service life information on zinc-aluminum alloys and aluminum MMCs. Figures 19 and 20 plot the required thickness specifications given in Tables 7 and 8. The service life estimates for Zn-15Al and Al-10 vol% Al_2O_3 , which were introduced in the 1970s and 1980s, respectively, are based on accelerated laboratory tests and service applications through 1992.

Problems to Consider in Sacrificial Coatings

Environmental concerns greatly influence the cost of applying such coatings on steel structures, either for atmospheric or marine corrosion control. In most areas, the Environmental Protection Agency (EPA) is very concerned about the types of debris generated during the surface preparation phase of the project. Because the coatings will be replacing existing paint and other organic overlays, concern over lead-bearing materials plus organic debris falling into flowing waters or onto the ground may require complete enclosure of the project in plastic sheeting and collection and disposal of all grit-blast material and debris. How-

ever, new technology, such as the thermal spray vitrification process, is making the removal of lead-base paints more environmentally friendly (see the section "Use of Thermal Spray Process to Remove Lead-Base Paint" in this article).

Zinc fumes generated during spraying can cause illness in exposed persons. Sprayers and other exposed parties are required to use appropriate breathing protection.

Thermite Sparking Hazard (Ref 14). Thermite sparking is caused by the reaction of rusted steel and aluminum (in the form of a finely divided smear) when this combustible mix is ignited by an impact. Aluminum smears may be generated on rusting steel by striking or dragging steel components and tools over bare aluminum surfaces or vice versa. Bare aluminum and bare aluminum thermal spray coatings should be avoided whenever there is a thermite sparking hazard. Situations where bare aluminum or bare aluminum thermal spray coatings and rusted steel will be near a combustion or explosion source can occur on a regular basis and should be avoided. There appears to be little risk of thermite sparking as a result of the impact of rusted steel on a sealed or painted aluminum, aluminum alloy, or aluminum composite thermal spray coating surface.

Table 7 Service life estimates of 85Zn-15Al thermal spray coatings in selected corrosive environments

See Fig. 19 for effect of coating thickness on service life.

Type of exposure	Coating thickness required for indicated service life							
	5–10 years		10–20 years		20–40 years		>40 years	
	μm	in.	μm	in.	μm	in.	μm	in.
Rural atmosphere	75–125	0.003–0.005	125–175	0.005–0.007	250–300	0.010–0.012
Industrial atmosphere	150–200	0.006–0.008	300–375	0.012–0.015	350–400	0.014–0.016
Marine atmosphere	250–300	0.010–0.012	300–375	0.012–0.015	350–400	0.014–0.016
Freshwater immersion	150–200	0.006–0.008	250–350	0.010–0.014	300–375	0.012–0.015
Saltwater immersion	250–300	0.010–0.012	350–400	0.014–0.016

Source: Ref 14

Table 8 Service life estimates of Al-10 vol% Al_2O_3 metal-matrix composite (MMC) in selected corrosive environments

See Fig. 20 for effect of coating thickness on service life.

Type of exposure	Coating thickness required for indicated service life(a)							
	5–10 years		10–20 years		20–40 years		>40 years	
	μm	in.	μm	in.	μm	in.	μm	in.
Rural atmosphere	150–200	0.006–0.008
Industrial atmosphere	150–200	0.006–0.008	250–300	0.010–0.012	250–375	0.010–0.015
Marine atmosphere	150–200	0.006–0.008	200–250	0.008–0.010	250–300	0.010–0.012	250–375	0.010–0.015
Freshwater immersion	150–200	0.006–0.008	200–250	0.008–0.010	250–300	0.010–0.012
Saltwater immersion	200–250	0.008–0.010	250–300	0.010–0.012	300–350	0.012–0.014
High-temperature (100–540 °C, or 210–1000 °F)	150–200	0.006–0.008	200–250	0.008–0.010	250–300	0.010–0.012
Wear, abrasion, erosion, and impact (90/10 MMC preferred)	150–200	0.006–0.008	250–300	0.010–0.012

(a) With exception of wear, abrasion, erosion, and impact properties, data for aluminum and 90/10 aluminum MMC are identical. Source: Ref 14

Weather concerns also influence these projects, because moisture and cold conditions can affect the quality of the coating as well as the project schedules. Fog and condensation usually hinder the application of coatings near rivers or on marine locations. Projects directly on water, such as the coating of dams or lock-gates, are frequently subject to high-water delays.

Nonsacrificial Thermal Spray Coatings (Ref 13)

Austenitic stainless steels, aluminum bronze (Fig. 21), nickel-base alloys, and MCrAlY, among other materials, are also thermal sprayed to combat corrosion in certain applications. Selection of a specific alloy depends on the particular environment. When using these alloys, it must be understood that these coatings will not galvanically protect the underlying steel. This can be a particular problem due to the porosity of thermal spray coatings. Great care must be taken to ensure that these coatings are properly sealed in order to prevent penetration of the corrosive medium to the underlying steel and subsequent corrosion at the coating/substrate interface.

Cathodic Protection

In addition to cathodic protection by sacrificial zinc thermal spray anodes described earlier, cathodic protection can also be

provided by anodes electrically polarized (positive) with respect to the metal structures they protect. Such anodes are commonly fabricated from cast iron alloys, graphite, precious metal plating, and precious metal/noble metal oxide mixtures. Some of the newer technologies use thermal spray deposits of semiconducting ceramics. These ceramic coatings are infiltrated with precious metal oxides, such as iridium or ruthenium oxide, that give them exceptionally high corrosion rates under high-current conditions. Impressed current anodes are immersed in both fresh and saltwater or buried underground in deep wells to protect structures, piping, tank bottoms, underground tanks, and even underground electrical cabinets. Some types of anodes are used in heated tanks for treating stored petroleum products. Other versions are buried in concrete to protect rebar.

Ferrite coatings have been found useful as anodic surfaces in several special cases. Compositions include nickel, cobalt, and MgMn ferrites plus several forms of iron oxide. The magnetic properties of these materials are of no value for this application; their value lies in the suboxides created during plasma spraying that cause the coating to be highly conductive.

These coatings are deposited on valve metals such as titanium, niobium, or tantalum. Their corrosion rates depend on the electrolyte and the exposed surface area they are required to protect. These rates are too high for general use in cathodic protection, but

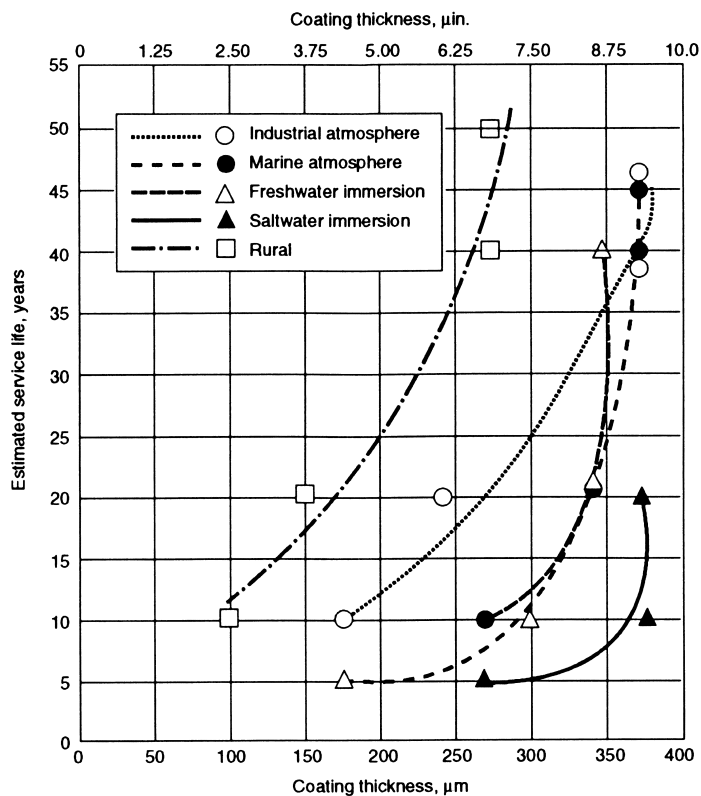


Fig. 19 Plot of service life versus coating thickness as a function of selected environments for a 85Zn-15Al thermal spray coating. See also Table 7. Source: Ref 14

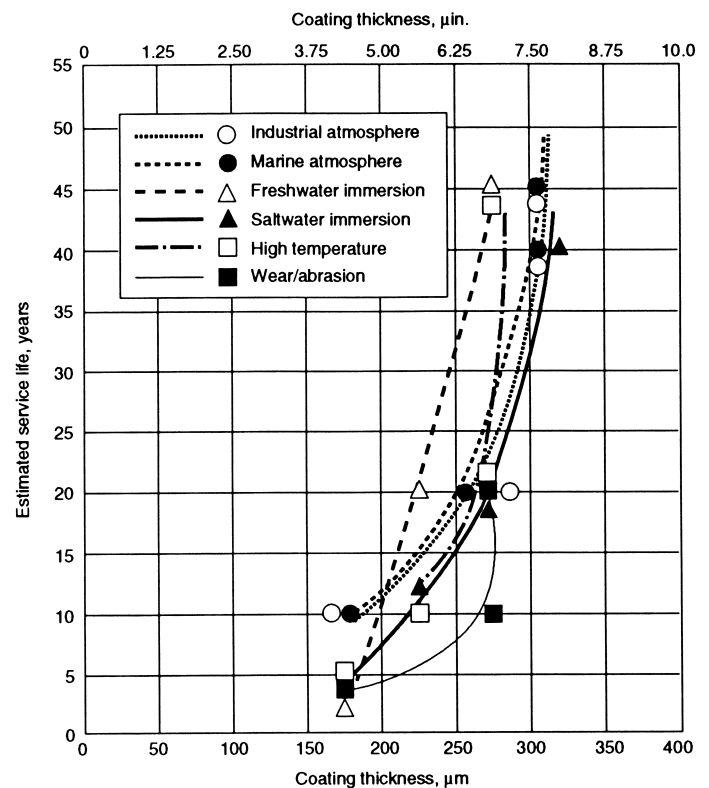


Fig. 20 Plot of service life versus coating thickness as a function of selected environments for an aluminum metal-matrix composite (Al-10 vol% Al₂O₃) thermal spray coating. See also Table 8. Source: Ref 14

special applications, including heat treater anodes (oil storage), precious metal recovery, and the protection of certain chemical process equipment, lend themselves to anode construction using sprayed ferrites.

Processing Electronic Materials and Devices

Conventional thermal spray processes present an image of being too coarse and uncontrolled for electronic applications. The chief problem would seem to be the splat interfaces, which dominate the microstructure of the deposited material, and both metals and ceramics are affected. For some applications at lower electromagnetic frequencies, this is not a problem, but these splats and their interfaces, plus stoichiometric differences, create problems for devices operating at higher frequencies.

Splat boundaries can be eradicated by using very high-temperature substrate preheats. Depending on the material and the desired properties, these temperatures may range from 700 to $>1200^{\circ}\text{C}$ (1290 to $>2190^{\circ}\text{F}$). These substrate temperatures must be maintained in the presence of O_2 for most ceramics and in inert or reducing atmospheres for most metals and semiconductors. This temperature requirement (plus some atmosphere requirements mentioned later) limits the choice of substrates. Designs must consider the differences in the CTE and possible reactions with the atmosphere.

One of the unusual factors that contributes to the electromagnetic performance of a material is the rapid solidification that yields hyperfine grain sizes, even at these very high deposition

temperatures. This phenomenon is both a blessing and a curse, because it permits the development of optimal microstructures for electromagnetic performance; however, it necessitates thermal annealing in many instances, which further complicates matters in the production of devices.

Thermal spray coatings cannot compete in the area of thin-film technology, where processes such as sputtering, ion plating, or chemical vapor deposition predominate. Thermal spray is a thick-film process and is therefore more useful as an alternative to screen printing plus firing of ceramics and metals. The advantages are found in the wide range of materials that can be processed and the many improvements obtained in performance.

The ability to deposit much thicker coatings also plays a role in applications. Many discrete electronic devices, such as capacitors, resistors, and inductors, can be manufactured using thermal spray processes.

Dielectrics

There are many applications for the spraying of oxide ceramics where electrical insulation is the desired property. Many of the alumina-base ceramics will serve in this capacity when processed by combustion flame, APS, and even HVOF. These deposits generally contain major phases of gamma alumina or wustite (FeO), which negate effective performance at higher frequencies. Other ceramics, such as titanates, steatites, and forsterites, suffer from some form of degradation during thermal spraying that renders them unsuitable for many dielectric applications. Some of these as-deposited properties, however, provide the way for new applications, and proper treatment of these dielectrics yields properties similar to those of bulk dielectrics or properties that may be superior. High-temperature substrates are used (requiring CTE matching plus annealing) for optimal properties.

Alumina ceramics that are conventionally thermal sprayed Al_2O_3 exhibit a high content of gamma alumina (due to the rapid solidification on the alumina splats) and little of the desired alpha phase, which is desirable for higher-frequency applications. *Conventional* refers to standard processing practices where substrates are not heated to high temperatures and O_2 is not added via shroud or powder gas.

Alpha alumina deposits are possible in as-sprayed deposits processed by combustion or plasma processes, provided the substrate temperatures are held above 1100°C (2010°F). (Note: Heating gamma alumina deposits to these temperatures to convert them usually results in cracking, because there is a substantial change in density as the phase change occurs.)

The difference in electrical performance between gamma and alpha alumina deposits shows in the dielectric constant and loss tangents at higher frequencies. This difference is also dependent, however, on density and morphology. Spraying alumina onto substrates heated to 1100°C (2010°F) results in deposits of essentially 100% theoretical density, provided CTE-matching substrate materials are used. These alpha deposits exhibit dielectric properties matching those reported for bulk materials at 9.6 GHz. There does not appear to be a measurable change in dielectric data as a function of grain size, so annealing after deposition is not necessary.

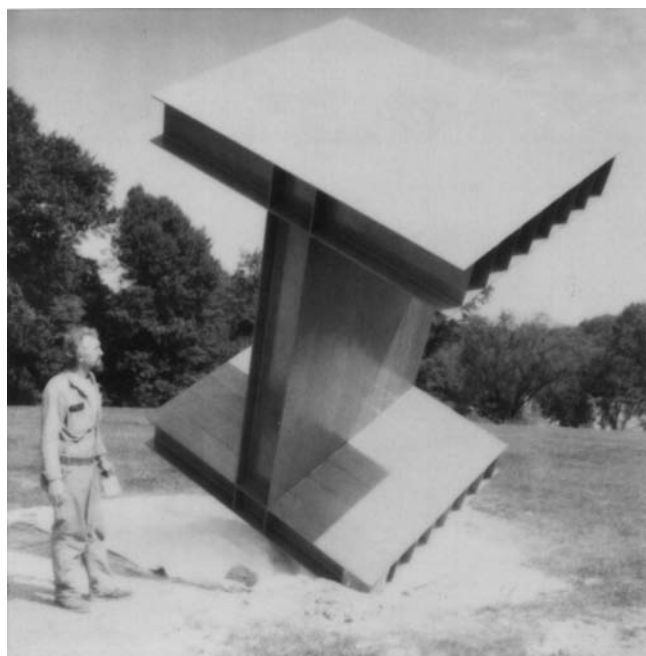


Fig. 21 Sculpture with an aluminum bronze thermal spray coating. Such nonsacrificial coatings must be sealed. The sealer fills the pores of the thermal spray coating, smoothes the sprayed surface, and improves appearance and service life. Courtesy of ASB Industries Inc.

There is little difference in voltage breakdown between the two phases. Both deposits reportedly achieve between 500 to 600 V/mil (1 mil = 25 μm , or 0.001 in.) when correctly processed. The 98% deposit density and associated splat morphology for gamma alumina products reduces its performance in high-moisture environments, whereas the denser alpha phase will sustain performance in such adverse conditions.

Some alumina-bearing ceramics resist the trend toward gamma phase formation during conventional deposition practices, the alumina being stabilized by other elements. Examples include $\text{MgO}\cdot\text{Al}_2\text{O}_3$ (a 1:1 spinel) and 20 wt% $\text{Cr}_2\text{O}_3\cdot\text{Al}_2\text{O}_3$. The spinel has microwave properties identical to alpha alumina when plasma sprayed, regardless of substrate temperature and so on. The chromia-alumina is not a microwave material but is a very effective insulator at temperatures above 1000 $^{\circ}\text{C}$ (1830 $^{\circ}\text{F}$). Both compositions can be altered through additions of magnesium or chromium to control CTEs.

Titanate ceramics are used in a variety of applications in the electronics industry. As dielectrics, it is essential that precise stoichiometries be obtained. Again, conventional thermal spray processing alters the composition by partially reducing the oxides to a semiconducting state, but process parameter modifications (e.g., much higher substrate temperature) yield rapidly solidified deposits that can be further tailored through control of the grain growth. Barium, strontium, lead, magnesium, and niobium titanates have been processed in this manner, with properties that match or exceed their bulk counterparts.

Beryllia

Spraying dense, pure beryllia (BeO) also requires higher substrate temperatures and very hot plasmas. Occupational Safety and Health Administration (OSHA) requirements for handling this toxic, disease-causing material are stringent and usually economically prohibitive. Excellent thermal and dielectric properties have been developed in plasma sprayed products, and structures have been produced for microwave and nuclear fusion applications. The device shown in Fig. 22 was formed by milling the meander-

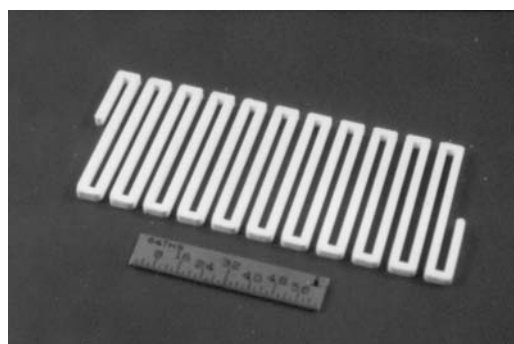


Fig. 22 A BeO meanderline substrate produced in near-net shape by inert chamber plasma spraying. Cross-sectional dimensions are $1.016 \times 1.016 \text{ mm}$ ($0.040 \times 0.040 \text{ in.}$).

line pattern in high-quality graphite. Inert chamber plasma spraying with a helium atmosphere was used to fill the milled pattern, the ceramic flashing being ground away. The net shape was completed by firing the structure at $>800^{\circ}\text{C}$ ($>1470^{\circ}\text{F}$), which removed the graphite, leaving the freestanding ceramic. Very accurate shapes can be manufactured this way.

Metallization

Every electronic device has to have current-carrying metals. Thick-film technology, as used for even moderate frequencies, has drawbacks. Such methods require bonding agents to be added to metal/organic inks that are screen printed onto ceramic substrates and fired at high temperatures to drive off the organic binders and sinter/bond the pattern. Glass is a common bonding agent but reduces the conductivity of the deposit from bulk values. Some refractory metals, such as molybdenum and tantalum, can be processed with reduced amounts of glass as a bonding agent for copper and other conductors, but their higher resistance plus poorer thermal properties further degrade device performance. (These same printing methods are also used for resistors and capacitors.)

Spraying of copper and other elemental metals for current-carrying tracks operating at moderate frequencies is not complex. Etched metal or tape masking can be used to define the geometry. In fact, etched metal masks have been used to produce parallel 10 μm (0.0004 in.) thick conductors 50 μm (0.002 in.) wide on 100 μm (0.004 in.) centers. Alternatively, the deposit can be photoetched. Both twin-wire arc and plasma spraying have been used, and properly designed and constructed masks exhibit reasonable life.

More problems are encountered, however, when it is desired to perform quality metallizations on highly polished ceramic substrates (or single-crystal surfaces) for use at microwave or millimeter wave frequencies. Splat interfaces will predominate unless these deposits are made at high temperatures. Most of these metallizations are approximately 5 μm (0.0002 in.) thick, and this can only be achieved in plasma spraying by using very fine powders (mean particle sizes of 10 μm , or 0.0004 in., or less) and powder feeders specially designed to handle these fine materials. Splats at these higher substrate temperatures are much thinner than at room temperature, which assists thickness control.

Oxide contents in feedstock powders result in reduction of the electrical conductivity. Furthermore, powders such as copper will getter, or absorb, oxygen during spraying. Steps must be taken to acquire clean feedstock powders and store them under inert (nonoxidizing) atmospheres. In most instances, spraying is done using shrouds or inert chamber plasma spraying, where O_2 pickup is minimal.

Bonding to ceramic substrate is accomplished by heating the substrate to temperatures several hundred degrees below the melting point of the deposit. All elemental metals have a eutectic formation with oxygen at temperatures a few degrees below their melting temperature. This can make for very high bond strengths when using carefully controlled processes including trace amounts of O_2 in the atmosphere. Copper, for instance, sprayed directly

onto a 0.254×10^{-4} mm (1×10^{-6} in., or 1 μ m.) polished alumina substrate held at 800 °C (1470 °F) develops an adhesive bond that is stronger than the intrinsic tensile strength of the alumina. Gold deposits directly onto polycrystalline alumina or sapphire or quartz with equal bond strengths, and a gold oxide is found diffused into the interface.

Electronic Device Applications

The needs for achieving desired properties from sprayed electronic materials have been described in the previous section. It now remains to describe how thermal spray processes can be used in component or system manufacture. The following is a summary of such applications but is not exhaustive or fully comprehensive.

Electromagnetic field/radio-frequency interference (EMF/RFI) shielding is perhaps the least technically demanding application for thermal spray coatings in the electronics industry. Various plastic- and foam-layered instrument and computer equipment cases require shielding against stray (EMF/RFI) radiation. Combustion-wire and twin-wire arc sprayed zinc can be applied to such cases. Higher-frequency shielding, however, places a demand on higher-conductivity coatings. Plasma sprayed aluminum may be of benefit, provided the cases will withstand the higher substrate temperatures. Twin-wire arc spray equipment using very small-diameter wires has also been used for the application of aluminum. Electrical conductivity is the most important property for such coatings. Because the substrate cannot be heated or otherwise affect the splat morphology, high-density and low-O₂ contents are required for best shielding performance. Powder spray processes, because of the higher surface area, usually have greater difficulty in processing low-conductivity coatings, whereas wire feedstock processes, particularly twin-wire arc, offer lower O₂ contents to begin with. Nitrogen (N₂) or other inert gases can be used for atomization to further improve conductivity.

A particular need exists in military applications, where secrecy is involved and computer centers might be compromised by emitted electromagnetic radiation. The conventional way of shielding such rooms was through the use of copper screening installed over the walls. Newer approaches involve the use of twin-wire arc sprayed zinc and aluminum coatings deposited directly onto cement block surfaces. A number of military installations have been treated this way.

Automotive electronics now make use of air plasma sprayed alumina for insulators on diode and other electronic package mounts. The application has replaced discrete BeO chips, which are used for high-power devices such as the final drive on stereo amplifiers or automatic door and window controls. High-volume production of plasma sprayed dielectric coatings on large-frame alternators is also being carried out by a major automaker (Ref 15).

Planar microwave devices, referred to as microstrip components, have been fabricated by plasma spraying dielectrics and ferrites in layers and combining these with appropriate metallization and sprayed rare earth magnets when required. The operating frequency dictates the material properties, but there is usually some variety of compositions from which to select CTE-matching com-

positions. Annealing of the ferrites is accomplished before metallization, but inert atmosphere annealing of magnets and metallizations does not damage the ceramic structure. Such devices include phase shifters, isolators, various filters, and circulators (Fig. 23).

One feature of this process is that magnetic materials and metallizations can be inlaid into dielectric structures, which results in superior electrical performance. Furthermore, at microwave frequencies, the absence of a refractory metal bond layer beneath the copper or gold metallization contributes to reduced device losses.

Waveguide devices for microwave applications have been the focus of several companies and U.S. Department of Defense agencies. The spraying of ferrites and dielectrics offers the opportunity to fabricate near-net shapes of high-performance ceramic structures. S-, C-, and X-band toroidal phase shifters have been so constructed. Stripline devices have also been made with metallizations added by plasma spraying. The performance of these devices generally exceeds that of conventionally made counterparts.

For a short period of time, there was an interest in solid ceramic waveguides made from alumina and other ceramics, with dielectric constants in the range of 6 to 9. However, metallization forming the outer periphery of the waveguide exhibited high losses due to the presence of refractory/metal interfaces. The problem was solved by APS of aluminum on all walls. Even without inert atmospheres surrounding the parts, losses were significantly lower than sputtered chromium-gold coatings.

Printing Industry Applications

The thermal spray community serves the printing industry with a variety of coatings applied on behalf of original equipment makers and end users. The wear and corrosion problems are enhanced by both the growing amount of printed matter and the variety of inks used, some of which are quite acidic. High-speed printers require closer machine tolerances and coatings that promote longer component life. These coatings, applied to paper-contact surfaces such

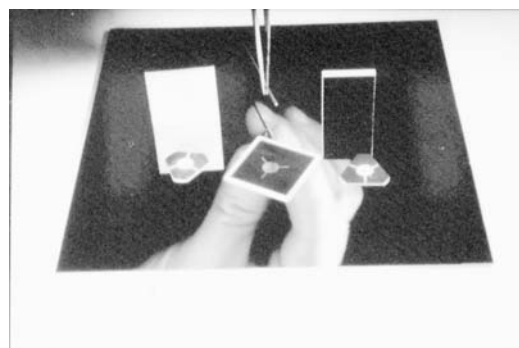


Fig. 23 An array of planar microwave devices and substrate fabricated by air plasma spraying

as rollers and doctor bars, protect against increased wear in these machines. Specific applications are described as follows.

Engraver Rolls

Engraver rolls are used to transfer ink to paper and historically have used finely scribed lines or engravings to provide a surface that is readily wetted by the inks. These rolls have very tight dimensional tolerance requirements, and the surface must remain clear of any defects. In recent years, plasma sprayed ceramic-coated ink transfer rolls have found greater acceptance. The ceramic is tougher, provides increased surface area for improved wetting, and offers additional corrosion resistance. The rolls are produced with as-sprayed surfaces of ceramic, generally, a 3 or 13% $\text{TiO}_2\text{-Al}_2\text{O}_3$ or chromium oxide (Cr_2O_3). Very fine powders are used, and the coatings must be applied uniformly. Some rolls may have engraver lines prescribed in the roll, and the coating must conform to those lines. Others may use only the texture of the coating. Still another approach uses laser engraving of the lines after the ceramic coating has been applied. This is typical for thermal spray coated anilox rolls. Anilox rolls are used in the printing industry to transport a precisely determined quantity of ink in flexographic printing machines. The rolls, which are manufactured from stainless steels, feature an APS bond coat (Ni, NiCr, or Ni-Al) and a topcoat of Cr_2O_3 alloyed with titanium oxide and silicon oxide. The topcoats are also applied by APS. The large size of the anilox rolls makes it difficult to process in a chamber, such as LPPS or inert plasma spraying. The topcoating is ground and polished, engraved with a laser, and polished prior to service. A thorough review of surface engineering of anilox rolls can be found in Ref 16.

Plate and Blanket Cylinders

These items are used on web presses and frequently suffer attack from corrosive inks. Historically, these rolls have been fabricated from solid stainless steel or with stainless steel weld overlays or electroless nickel. Recently, though, inks have become more acidic, and improved corrosion resistance has been desired. At one time, combustion-wire sprayed molybdenum coatings were used, but the visible porosity plus the lack of a cosmetic appearance curtailed its use.

More recently, Hastelloy C alloys have been applied to these rolls by either APS or HVOF. This material meets both the appearance and corrosion-defense criteria.

These cylinders have a slot in their surface parallel to the axis in which the blankets are fixed. Coatings must conform to that slot yet maintain their integrity. The cylinder is ground to final dimension after coating, but the slot may be simply stoned to remove the rough surface.

Draw Rolls

A number of rolls used in printing presses are textured to grip the paper and draw the sheets through the press. These varied-size rolls are fabricated from hardened steel and grit blasted to provide

the necessary “tooth.” Such rolls wear, oxidize, and go out of specified dimensions, however, necessitating repair and restoration.

Plasma sprayed coatings of nickel-chromium hardfacings have improved their performance. These coatings are available in a range of controllable textures by carefully selecting the feedstock powder size and process parameters used, and they exhibit much longer service lifetimes. Very close tolerances in finished rolls are a must; it is possible to remove taper in the roll itself by control of the deposition process.

Processing Industries and Corrosion Control

This section focuses on a rather large group of industries involved in the production of materials, ranging from organic to inorganic, as used by consumers or by various industrial customers. This includes mining, logging, and drilling (oil and gas)—the industries that provide basic material resources. It also includes the refining of these resources (ore extraction; refining of coal, oil, and gas; pulp making; and chemical processing) and the manufacture of the final products, such as paper, basic metals and metal alloys, organic and inorganic chemicals, and plastics.

These industries all have similar needs and often make use of the same equipment, that is, tanks, pressure vessels, pumps, valves, centrifuges, mills, and related peripheral equipment. Therefore, much of the maintenance needs and the coatings that can be used to protect the equipment against harsh environments is often similar. For instance, Stellite No. 6 and Colmonoy (a spray-and-fuse-type coating) are frequent requests, because their performance has been often proven, and they are well known in all these industries. A list of applications includes:

- Wear prevention
- Dimensional restoration
- Thermal insulation and control
- Corrosion resistance
- Lubricious films
- Seals

Among these materials processing industries, there has been a steady growth of new products, made possible by gains in materials science and related processes. As a result, their market vision is constantly changing, along with their traditional ways of conducting business! Whereas plants were formerly constructed for long-term (perhaps 30 years) life with a minimum of modification, today's rapidly changing technology no longer makes such planning economically attractive. It now makes more sense to design for ongoing modifications, and coatings are being viewed as one means of rapidly and economically adapting existing plant equipment to new processes, thereby reducing capital costs. By way of example, quotations in 1986 (approximately the beginning of the new vision) showed that a 7600 L (2000 gal) processing vessel constructed from Hastelloy C-276 would cost approximately \$1.2 million and require up to 1 year lead time. Replacing bulk material with explosion-clad material would reduce both the price and

lead time to \$750,000 and 18 months, respectively. A plasma sprayed application of C-276 on a ferrous-constructed vessel of the same size could, however, reduce the total cost to less than \$100,000 and delivery to less than 3 months. Factors likely to favor the decision to use a thermal spray coating include:

- Rapid entry into the product market
- The likely short product life
- Reduced capital expenses improving the margins
- The ability to reapply the selected coating or another coating to the substrate

The corrosive environments encountered by such equipment require the utmost care in application of the coating and the proper selection of the sealing agents used. In fact, the sealer will make the apparatus very product-specific, because, unlike the bulk construction, the range of pH, the amount and nature of organic solvents, and the phase of the corrosive environment encountered will all be sealer-dependent.

Coatings for Use in a Corrosive Environment

Splat boundaries often dominate the corrosion properties of thermal spray coatings. Effectively, such interfaces leak; that is, there are clear, albeit tortuous, paths through these interfaces to the substrate that cannot be entirely eliminated, regardless of coating thickness, without some additional treatment. Some types of materials may also exhibit a shift in corrosion potential as a function of these interfaces, regardless of how tight they may be. Several solutions are discussed as follows.

The issue of galvanic cells was discussed in an earlier section, "Noble versus Sacrificial Coatings," in this article. Coatings of interest to these process industries are noble relative to the substrate. The galvanic couple formed by the contact of both the ferrous substrate and the noble coating with an electrolyte will result in a rapid attack of the ferrous metal, that is, the substrate. Unsealed areas of the coating, stress fractures, or other inadvertently exposed edges will, unfortunately, ensure such exposure. Generally, the removal of the coating by such corrosion is rapid, because gases will build up beneath the coating, causing it to lift away (debonding or spalling) from the substrate, and these bubbles will eventually crack and spall away from the component they are supposed to protect. It is even possible that with an improperly chosen, applied, and treated coating, a galvanic cell may be established that actually hastens the demise of a piece of equipment more rapidly than if it had been left uncoated. Therefore, some discussion is essential on how best to implement these coatings for the processing industries.

Substrate surfaces must be well prepared for the application of any thermal spray coating. Among the first things to be considered is the geometry of the surfaces to which the coating must be applied. Much of today's equipment used in the processing industries is crudely manufactured or certainly not intended for use with coatings or linings. Such equipment includes sharp edges, reduced nozzles, rough welds, and other sites that make for difficult application of thermal spray coatings. Frequently, therefore, equipment modifications are essential. At the very least, all edges must be

well rounded, because these act as stress raisers during production cycles, and such stresses may increase the possibility of coating cracking and corrosion.

Substrates that have been previously attacked by any corrosive media must be thoroughly cleaned, and fresh (white metal) surfaces must be exposed. Frequently, the corrosive attack may be at grain boundaries, where residues of the media are still lodged. Unless this material is removed to uninvolved structure, the applied coating will be subject to attack from above and below, with little hope of long-term survival.

A special condition known as hydrogenation (or hydrogen embrittlement) is caused by corrosion that generates molecular hydrogen that resides within the microstructure of the attacked metal. The metal is usually of a different texture when ground, often exhibiting a brittle nature, with microfractures occurring as a result of the collection of nascent hydrogen in any porosity. If thermal spray coatings are applied over this substrate, the same nascent hydrogen will debond the coating after a short period of time.

Frequently, it is necessary to remove sections of the substrate that have been chemically attacked in the manner discussed previously. New material is then added by welding, and welds must be smoothed to provide proper coating geometry. It is important to note that welding itself can induce hydrogen penetration into certain substrates, cast iron and titanium being among them. Welding technologies and materials that eliminate this danger do, however, exist and should be used in these cases.

Proper substrate preparation includes aggressive grit blasting to remove all corrosion product residuals and includes cleaning and degreasing with proper agents to assure the absence of oils and grease.

Coating selection should begin with the review of corrosion data and tables as found in various handbooks, manufacturer's literature, and in the customer's library of experience. However, it should not be limited to these resources, because aside from the issues of through porosity in thermal spray coatings via the microstructure, the rapidly solidified anisotropic deposits often behave differently from reported data, which are often for bulk or monolithic materials. The latter is usually taken on fully developed (forged, heat treated, hot rolled, etc.) microstructures.

Coupons that incorporate the various coating candidates and candidates for sealers are frequently tested under laboratory conditions simulating production conditions. In the absence of that capability, samples may be inserted with wands or wired into locations within the piping, vessels, or even pumps for timed periods and periodically evaluated for wear and/or corrosion. Knowing the wear rates (millimeters/year or inches/year) is important, because they determine how thick the coating needs to be in order to provide the desired equipment life.

Sealing is the traditional method of handling through porosity (or "holidays") in coatings. All manner of sealer systems have been used, including carnauba wax, epoxies, polyesters, furans, anaerobics (which harden in the absence of air), sodium or potassium silicates, phosphates, and even sugar (heated to turn it into carbon).

The nature of coating morphology assures a very high length-to-diameter ratio for the interlinking of the splat interfaces. This is

both a curse and a blessing for applications in corrosive environments. On the positive side, sealers can be very well anchored if they penetrate into the coating. Penetration can be aided by establishing a vacuum around the coated part, allowing the sealer to enter the coating structure, and even adding some overpressure to force it into the pores—a method often used to seal castings. Preheating the coating also aids sealing by forcing some of the air out of the pores and perhaps reducing the viscosity of the sealer. The use of sealers with solvent agents to lower the viscosity is also common. The nature of splat interfaces causes the holidays to have very high length-to-diameter ratios, so sealers will tend to lock in place.

The negative side concerns the corrosion activity of the sealer. The sealer material must be chosen with care, taking into consideration the environment it will likely encounter. Moreover, if the sealer is likely to be attacked, then it can be chosen so that the corrosion products still act as an effective sealer, an attribute that is enhanced by the high length-to-diameter ratios of the coating splats. Furan resins, for example, are often capable of being converted to carbon during chemical attack and will remain locked into the interstitial spaces as an effective sealer.

Sealers also have temperature limitations, because most are organic. Sealer selection must be based on the manufacturer's data, including corrosion behavior at elevated temperatures, if that will be the exposure.

Large surface areas that cannot be easily evacuated or heated intensify the problem of sealers. The only approach then is multiple applications of a product that is chemically compatible, endeavoring to use it in the lowest viscosity form. The sealer cannot depend on heating for its curing; therefore, a two-part sealer, such as an epoxy, is necessary.

Post-heat treats are applicable to smaller-sized pieces of equipment. Atmospheric or vacuum heat treatments will alter the coating splat morphology and can improve bonding of the coating to the substrate. This makes the choice of coating material more difficult, because the CTE must more closely match the substrate as well as have favorable corrosion characteristics. Graded interfaces, however, have been used to overcome the CTE problems encountered during such heat treatments.

Not all coating systems effectively sinter to reduce porosity. It may be necessary to add sintering aids to the coating system, and these may impair corrosion resistance.

Laser glazing is a technology in its infancy, yet the U.S. Navy and private industry have successfully used it on large process equipment. Both high-power (kilowatt range) continuous-wave (CO_2) and pulsed lasers have been successfully used. The advantage is that CTE mismatches are not as critical, and the average temperature encountered by the substrate will not have adverse effects on structural welds or material properties.

Laser glazing has limitations, however, because it fails to work on some metals systems where the thin glaze has substantially different properties from the base metal, resulting in "mud cracks" in the surface.

The LPPS and HVOF processes are being researched to produce dense, corrosion-resistant overlays for these industries. While it is true that many of the coatings required involve very large surface areas ($>46 \text{ m}^2$, or $>500 \text{ ft}^2$), the technology is available to produce certain coatings. However, to be successful, these

processes must yield holiday-free coatings. This has been demonstrated in the laboratory, and there are encouraging reports from field operations involving HVOF equipment modified with inert gas shrouds.

Process Industry Maintenance Applications

Thermal spray coatings have been associated with the maintenance and repair sectors of various processing industries. Classic (and historical) applications are pump seals (shafts and sleeves), compressor rods, rotary airlocks and feeders, conveyor screws, and a variety of other components. Materials employed range from tungsten carbides (plasma sprayed), chromium oxides or alumina (plasma, combustion flame, or rod sprayed), to MoNiCr (plasma sprayed), and NiCrBSi (combustion flame spray and fuse).

Dimensional restoration of a number of components, including pump housings, back plates, compressor bores, compressor pistons, and centrifuges, can be carried out by thermal spray processes that include twin-wire arc, flame, plasma, and HVOF spraying. Materials used are generally of the NiAlCr variety or sometimes bronze, if corrosion is not a particular problem. Machining to final dimensions can be performed with these coatings.

A particular example of the success of thermal spray is the restoration of very large (and expensive) pump housings using twin-wire arc. Bronze and other materials have been used to fill cavitation damage 20 mm (0.75 in.) deep. The restoration is completed by the use of HVOF to apply carbide surfaces.

Chemical Process Industries

Coatings for pressure and storage vessels can provide major savings in process industries. Linings of interest include Hastelloy (B, C, D, and G), various Monels, Inconel (600, 625, 718), and Nickel 200. To a lesser extent, linings of tantalum and molybdenum are also of interest.

These are all costly alloys to use, requiring special forming and welding techniques. Most of these materials are available as claddings, but ordering materials for construction requires very long lead times.

Low-carbon steel fabrications with corner radii large enough to allow thermal spraying are readily fabricated. Air plasma sprayed coatings are being successfully employed as corrosion barriers for a variety of chemical processing components. Even ceramic linings such as alumina or chromium oxide are of interest, particularly where chemical attack is combined with aggressive abrasion.

While it is desirable to coat large items of chemical process equipment in a controlled thermal spray facility, this is usually not practical. Therefore, on-site application is frequently necessary. Every effort should be made to automate the coating process as much as possible, but hand-spray operations may be required for certain pieces of equipment.

In Europe, inert atmospheres have been established for large pieces of chemical apparatuses using inflatable warehouse structures inflated with inert atmospheres such as argon. Under these conditions, some automation and robotic work has been demonstrated, with plasma sprayed tantalum being the coating of choice.

Pressure vessels of up to 7600 L (2000 gal) capacity have been APS coated with Hastelloy C, Hastelloy B, and Inconel 600, and,

in all cases, the lifetime of these coated units has greatly exceeded the design life. Similar linings have been applied to larger pumps, centrifuge housings, and filter housings.

Coatings for heat-affected zones (HAZ) offer a solution to a critical problem in the chemical process industries. Many of the process vessels require water jacket construction about the lower tank head and up the sidewalls to control the temperature of the process inside the vessel, either through the use of steam heating or cold-water cooling. Higher temperatures in a vessel, particularly at localized sites, can cause accelerated corrosion that may not have been predicted by pilot-plant or laboratory-scale observations. Furthermore, many alloy systems exhibit problems with the metallurgy at welded seams, in which certain species of carbides or other unwanted elements segregate at grain boundaries. This is evidenced by accelerated corrosion attack on both sides of the weld and, in some cases, removal of several inches from the seam.

Thermal spray deposits, especially plasma and HVOF, are rapidly solidified, which means that the homogeneity and the grain development are such that carbides and unwanted species will not segregate at the grain boundaries. Such coatings can be applied over metals in the HAZ, thereby preventing the advanced corrosion attack.

A special case exists for 6061 and 3003 aluminum alloys as used in very large nitric acid tanks. These welds corrode at an accelerated rate, making it necessary that such tanks be frequently reworked. Plasma sprayed niobium, doped with small percentages of palladium to prevent a galvanic cell problem, has been found to reduce or eliminate this weld corrosion. Spraying of either of the aluminum alloys, however, offers no benefit, because it would require heat treating of the deposits to render them resistant to the acid.

Glass-lined equipment, found in some chemical reactors, is another specialized niche for APS. Much of the chemical process industry uses such vessels and storage tanks for their corrosion resistance against strong acids in combination with organic solvents or their release properties for various polymers and compounds being produced.

This equipment is highly susceptible to glass breakage caused by localized thermal shock (during acid neutralization, for example), foreign objects being dropped into the reactor, or accidental chipping during cleaning of the vessel.

The standard repair procedure for damaged linings is the installation of a tantalum plug gasketed with a synthetic fluorine-containing resin. For larger areas, tantalum sheets are used with tantalum bolts and capnuts. Large areas and geometric contours, however, render this repair method unsuitable for a sizable number of damages.

On-site APS has been used to repair damaged glass linings with tantalum deposits, followed by an overlay of chromium oxide ceramic. The tantalum, being foremost among the corrosion-resistant materials, bonds directly to the glass. Great care has to be used, however, because the glass is highly stressed, and interparticle bonding of the inner glass layers is rather low. Air plasma spray has worked best in this application, because it offers minimal thermal shock, and the deposition rates and particle velocity can be kept

quite low. The exposed steel substrate is coated first with NiCrMo, which is a low-stress deposit that can fill in corrosion damage, especially in seal areas. The ceramic topcoat protects the edges of the tantalum/glass attachment and generally strengthens the patch against abrasion, cavitation, and other types of erosion. Incidentally, chromium oxide is an equally corrosion-resistant material; picric acid is the only acid reported to corrode it.

Tantalum repairs of any kind are subject to gross failure if chromium- or nickel-bearing alloys are placed into the vessel with some form of electrical connection to the tantalum. Under low-pH conditions, tantalum will become sacrificial to those elements; however, a very small addition of platinum, mechanically blended with the tantalum powder during spraying operations, maintains the noble aspect of the patch under these conditions.

Pulp and Paper*

There are many opportunities in this industry for thermal spray coatings to extend the service life of the equipment used during the manufacture of paper and cardboard. Aside from the common items such as pumps and rolls (Fig. 24), there are usually areas where special handling occurs that leads to unusual wear and corrosion problems. In the analysis of such opportunities, coating practitioners should be aware of the closed water systems now used in the pulp-making process. In these systems, microbial corrosion can occur, because the continual reuse of the water supply without proper treatment can cause a buildup of microorganisms. Even with treated water, regions may exist in certain pieces of equipment or piping where the water stagnates and does not receive the required amounts of antibacterial chemicals.

The decaying bodies of bacteria that become embedded in the grain boundaries of various metals cause bacterial corrosion. Commonly found in situations where there is a great deal of energy expended within a small area, these decaying bodies (carbon) create a galvanic cell that weakens the microstructure of the metals involved. Subsequent mild erosion, cavitation, or abrasion attacks the weakened microstructure, and rapid wear results. On inspection, abrasion/erosion/cavitation appears to be the root cause, but all attempts at providing corrosion-resistant hardfacings fail. A positive test (of the water) for the type of bacteria known in these processes usually reveals the real cause of such failures.

Machine knives, such as slitters and guillotines, benefit from the application of WC/Co coatings. Such knives work against a second blade, usually a blade having a 90° geometry, trapping the fiber between the rotating or shearing point and the fixed bed knife. The dulling action occurs when sheared material trapped between the two blades causes abrasive wear of the slitter or guillotine, the wear occurring just behind the contact point. As the metal thins, support for the sharpened edge gives away, and it simply rolls over (Fig. 25). High-velocity oxyfuel or even air plasma sprayed carbide applied to the eroding edge, usually the flat side, protects against this activity. The deposits do not have to be thick;

*The discussion "Coating of Large Center Press Rolls for Paper Machines" was contributed by William J. Lenling, Thermal Spray Technologies Inc.

coating thicknesses of $13\text{ }\mu\text{m}$ (0.0005 in.) have been shown to be effective. Blades are then resharpened by grinding the opposite side only.

Coating of Large Center Press Rolls for Paper Machines.

Center press rolls (Fig. 26) are at the heart of many papermaking machines. The center press roll is the last in a series of rolls that is used to mechanically remove water from the paper as it is being made. It is very important to remove as much water from the sheet using mechanical forces, because it is more economical than using thermal energy. Thermal energy is used after the center roll position to remove the remainder of the water from the sheet of paper.

Paper has a tendency to stick to the roll after coming out of the nip, due to the high solids content. When the material sticks to the roll, the sheet tears, and production time is lost. Equipment damage can also occur, because the material buildup can deflect or damage mechanical parts. Rolls made from more common materials, such as steel and cast iron, have major sticking problems and cannot be used in the center press position where they might directly contact the paper. For many years, the standard in the

paper industry for center press rolls was granite rock. Granite press rolls, however, have their disadvantages. The first issue was that making a precision roll from solid granite was very expensive. Some center press rolls are 1 m (3 ft) in diameter and 10 m (30 ft) long. Another disadvantage was that as paper machines advanced in design, the production speeds became faster and faster. The higher machine speeds put more stress on the granite rolls. Eventually, rolls exploded on some machines, causing thousands of dollars in damage and downtime. In a few cases, there was loss of life. Because of these problems, granite roll substitutes needed to be developed.

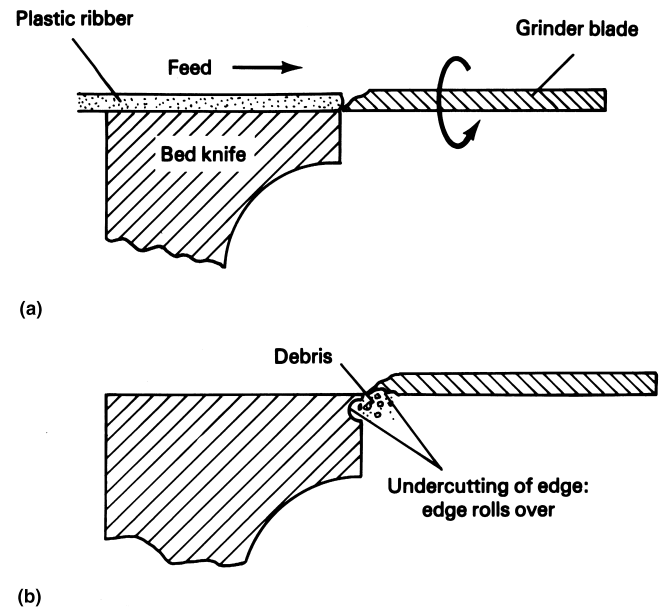
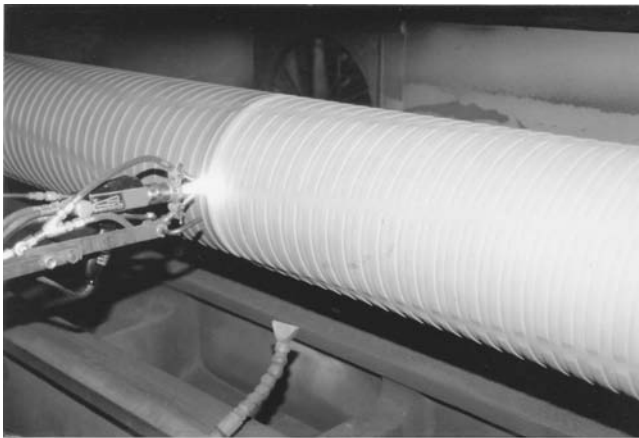
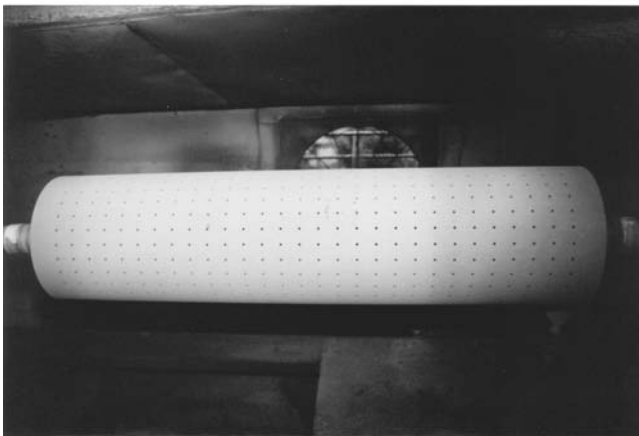


Fig. 25 Illustration of wear on a machine knife edge



(a)



(b)

Fig. 24 Examples of thermal spray coatings deposited on pulp and paper processing components. (a) Roll used in the paper industry being coated with tungsten carbide to provide a traction coating. (b) Suction roll that has been coated with tungsten carbide. Courtesy of ASB Industries Inc.



Fig. 26 Center press roll used in papermaking machines. These rolls are coated with nonstick ceramic coatings by plasma spraying.

Today's state-of-the-art center press roll is made with the help of thermal spraying. Cast iron or steel roll bodies are coated first with a corrosion-resistant bond coating approximately 40 μm thick, and then a ceramic coating of alumina-titania or chromium oxide is applied. The ceramic coating is approximately 80 μm thick after the coating is ground to achieve a desired surface finish. Typically, the bond coat is deposited by the HVOF process, and the ceramic coating is plasma sprayed.

Both alumina-titania and chromium oxide were found to have excellent paper release properties (as good as and, in many cases, better than granite). The thermal spray coating properties have allowed for improved production on some machines. The use of thermal spray, along with a gray iron or steel roll bodies, provides a much safer design to handle the higher running speeds of today's paper machines.

Yankee drier rolls used in the manufacture of tissue paper can be resurfaced on-site using thermal spray processes. Such rolls are usually hollow castings operating with high-temperature steam to provide a drying action to the wet fiber. The ideal situation is to be able to match the performance (thermal conductivity) of the cast iron but eliminate the rusting, which requires continual machining of the surface and eventual roll replacement. Stainless steels can obviously aid the corrosion problem, but their reduced conductivity also reduces productivity of the drying process. Molybdenum offers better thermal performance, and so, stainless steel coatings with high molybdenum contents are often used, applied by HVOF for maximum density. Approximately 34,000 kg (75,000 lb) of material is used annually for this application. Reportedly, the savings to the plant site is in the six figures.

The operation requires on-site grinding or other machining to prepare the cast iron surface and to finish the coating surface. Custom-designed equipment must be constructed that is capable of holding exacting tolerance and providing excellent finishes.

Blow tanks are large storage bins that receive high quantities of wet pulp at various stages of the papermaking process. The abrasive wet mixture, often containing sand and other particulate debris, is blown into the vessel at high rates, which results in a short bin life unless the walls are suitably protected. One method of protection has been the welding of ceramic tiles over the walls, but these have been known to break readily and further contribute to the damage. Recently, it has been determined that on-site applications of HVOF sprayed WC/Co coatings work well as a wear-resistant surface. Related equipment, including large pipe elbows, could benefit from the same treatment.

Metals Processing Industries

Mold coatings for foundries offer significant benefits to cast metal products by serving as thermal barriers. Thermal barrier coatings can control the flow of heat within a permanent mold and retard rapid chilling, which gives rise to defective microstructures. Also, with TBCs, initial casting runs to preheat the mold are no longer necessary. Therefore, productivity for the mold is improved.

The most common solution within the industry is to use ladle washes, which are aqueous suspensions of various ceramic parti-

cles that are applied by brushing or spraying and are often preheated to consolidate the film. Wash coatings are never long-lived and require frequent replacement, with accompanying losses of production and labor costs. In addition, they are not all that effective as thermal barriers.

In the absence of O_2 , most molten metals respond well to coatings of ZrO_2 -7 wt% Y_2O_3 or even $\text{MgO}\cdot\text{ZrO}_2$, but if O_2 is present, a slag forms, and very aggressive corrosion processes fail most ceramics. Yttria proves, however, to be resistant to slag attack and can be used as a very effective barrier against corrosive melts that might otherwise attack molds. A three-layer system ($\text{NiCrAlY}/7\text{ wt}\%\text{Y}_2\text{O}_3\text{-ZrO}_2/\text{Y}_2\text{O}_3$) is successful in withstanding the slagging corrosion and offers the slight retardation of freezing that is desired. This coating functions successfully for ferrous, bronze, aluminum, and several noble metal cast products.

High-temperature wear coatings are also of interest in the foundry environment, because some molds have areas of small cross section or delivery channels that are prone to hot-metal erosion. Other molds have protruding pins and risers that also suffer from hot-metal erosion. Thermal spray hardfacings that retain toughness while remaining hard at higher temperatures can effect a longer life for such components. Many of the same coatings used in the aerospace industry for turbine engine wear, such as nickel-chromium/chromium carbide, have proven beneficial in such applications.

Ancillary equipment used in foundry operations can also be improved through the use of high-temperature wear and corrosion-resistant coatings. Examples include conveyor rollers, bearing boxes, and ladle pivots.

Casting salvage is another area in which thermal spray operations contribute to the foundry industry. Large castings, sometimes weighing several tons, are expensive items. Subsequent machining of surfaces may expose porosity that effectively compromises the casting utility. Localized grinding of the porosity to open all cavities and expose possible reentrant geometries is followed by repair of the damage. Both twin-wire arc and plasma spray techniques have been successfully used to fill in these voids with appropriate metal. Re-machining of the surface completes the salvage. This technique has been used on large machine tool castings, diesel engine block castings, large castings for the process industries, and the permanent molds for tires, shown in Fig. 27.

Molten metal containment and delivery constitutes a major difficulty for many of the newer processes. Rapid solidification processes, such as spin melting, ribbon casting, and powder production facilities using spray atomization, have serious problems. New techniques of injection molding and pressure casting also have similar needs. The remarks made earlier regarding the ceramic compositions are pertinent for these applications. Crucibles and ladles are routinely coated with Y_2O_3 and hold molten metals as diverse as uranium and titanium + titanium diboride. Many container materials are suitable for the application of ZrO_2 or Y_2O_3 coatings. Alumina, zirconia (for yttria lining), platinum, graphite, molybdenum, and other refractories have been used.

Steel mill roll coating services are conducted on-site with thermal spray coatings designed specifically for both wet and dry mill environments. Although these coatings will protect the cold mill from deterioration (giving up to three years of service life),

their main benefit is to the strip product. Production rates are greatly enhanced by the reduction of sheet slippage. Rolls are generally coated with tungsten carbides, with choices of several alloy matrices (cobalt, nickel, cobalt-chromium), depending on the nature of the mill corrosion. Coated rolls must be finish ground on-site.

Within the purview of strip mills (steel), there are applications for rolls that contact molten aluminum or molten zinc, which are used for aluminizing or galvanizing the sheet steel. In these applications, ceramic coatings—some multilayered for better performance—are used. The proprietary materials are chosen to reduce adherence of dross while permitting even dispersal (wetting) of the molten metal over the surfaces of the strip.

Uncoated rolls in this application may experience failure within three days of production time. Life extension averages more than one month are achieved for coated rolls.

Hearth rolls, also devices for galvanizing and aluminizing, can make use of special-purpose ceramic coatings as well. The coatings resist wear, slippage, and withstand the thermal shock that would cause checking or cracking in an uncoated roll. Dross also does not accumulate on the roll to such a point as to become embedded into the surface of the strip.

Other rolls often coated include stabilizer rolls, sink rolls, guide rolls, and tower rolls. The preferred coating is HVOF tungsten carbide, because it greatly minimizes wear and promotes higher-quality strip.

Electrical Utilities

Coal-Fired Magnetohydrodynamic (MHD) Generators.

The MHD generators have demonstrated approximately a 15% gain for coal-fired electrical power plants when used as the initial stage in coal combustion. The MHD is essentially a gigantic Hall-effect cell using seeded coal gas as the doped semiconductor. A magnetic field applied orthogonally causes the generation of an electric field in the remaining rotational direction. Direct current is extracted from the electrodes that form the channel for the hot plasma gases.

Two approaches are common: a cold-wall version using water-cooled copper electrodes, and a hot-wall system that requires con-

ductive ceramics to withstand the 2000 °C (3630 °F) temperature. The cold-wall version suffers from intense electrical discharges, which will ultimately cause failure of the electrodes. Plasma sprayed platinum up to 1 mm (0.04 in.) thick has been used to reduce such damage. The cold-wall approach, however, while being less sensitive to material problems, is made economically unattractive by the extensive use of platinum, and theoretical projections indicate that the hot-wall method has efficiency advantages.

The early hot-wall MHD electrodes were constructed from a variety of conducting ceramics, including lanthanum chromate, magnesium-manganese spinel, and magnesium-manganese ferrous ferrites. The electrodes were formed using conventional press-and-sinter ceramic processing techniques but suffered failure due to thermal shock and delamination from the copper bases, caused by CTE mismatches.

Air plasma spray proved to be very successful, because its attributes permitted the correction of CTE problems through the use of graded interfaces, the control of morphology to improve the thermal shock resistance, and the use of stoichiometry control to adjust thermal conductivity and electrical parameters during electrode construction. Many electrodes were fabricated in several layers, with some plasma sprayed deposits being 7 mm (0.28 in.) thick (Fig. 28). These devices withstood coal gas containing slag and potassium carbonate seeding at 2000 °C (3630 °F), with velocities of the effluent exceeding Mach 2.

Dielectric spinels (1:1) were used as an electrical insulator to separate the electrodes in the array. These coatings ranged from 1 to 2 mm (0.04 to 0.08 in.) thick and withstood the rigors of the environment quite successfully.

Fluidized-Bed Coal-Fired Boilers. Such units burn a finely divided mixture of coal and limestone particulates. In recent years, they have found application as small electricity-generation facilities on industrial sites.

Vertical-mounted steam pipes and boiler walls are eroded at high temperatures by the coal and limestone particulates. The 20 wt% $\text{Cr}_3\text{O}_2\text{-Al}_2\text{O}_3$ (solid solution) ceramic coatings have been found to be excellent coatings (with a NiCrAlY bond coat) to withstand this activity at higher temperatures, with little impact on thermal efficiency. Coatings are usually applied on-site.



Fig. 27 A large tire mold cast in an aluminum alloy that has been reclaimed by filling in the porosity

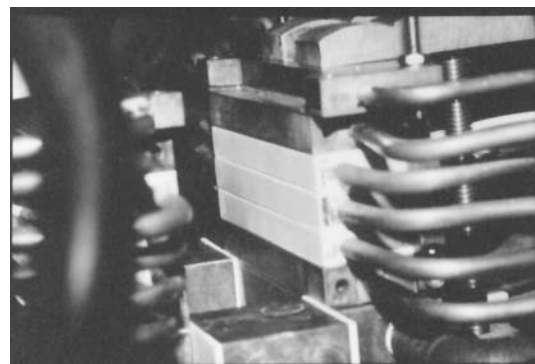


Fig. 28 A magnetohydrodynamic electrode set. These items were constructed as a series of layers deposited on copper. In order of deposit: Hastelloy B-4 (graded to lanthanum chromate), platinum (which also was applied to the electrode sides), and topcoated with CeO

Conventional Coal-Fired Boilers. Utility boilers suffer from high wear and corrosion caused by coal slagging. In recent years, there has been substantial research showing extended life for such boilers when corrosion/wear zones are coated either by plasma spray, twin-wire arc (Fig. 29), or HVOF with Inconel systems or other proprietary alloys. Most are now coated with wire-arc applications of high-chromium alloys or chromium-nickel alloys that effectively protect them from high-sulfur corrosion for periods of up to five years. It is estimated that 4650 m² (50,000 ft²) of new surfaces are coated each year.

The Textile and Plastics Industries

There are similarities in the textile and plastics industries, because many of the textile products contain organic fibers. For the purpose of brevity, thermal spray coating applications common to both are discussed.



(a)



(b)

Fig. 29 Economizer tube bends being coated with a high-chromium steel alloy using the twin-wire electric arc process (a). The tubes after coating are shown in (b). Courtesy of ASB Industries Inc.

Knives. A variety of cutting edges is used for slitting, chopping, pelletizing, and grinding fibers and extruded plastic shapes. These blades have single edges; that is, the taper is ground only on one side, and the other side remains flat. Some of these knives operate against opposing edges (Cumberland grinders, pelletizers, and shears), while others may cut against a rubber or paper roll. Extremely corrosive environments or the high mineral contents in the fibers or plastics may dictate that blades are fabricated out of solid carbides, although metal blades can exhibit significant life extension with properly applied tungsten carbide coatings. The nature of knife blade wear was discussed in an earlier section (see also Fig. 25), noting that this wear is caused by lack of point support.

This same type of wear can occur on the anvil knife (or bed knife) that operates in conjunction with the shear/grinder/pelletizer blades. Once again, the applications of tungsten carbide coatings to the surfaces normal to the opposing blade provide longer service lives between sharpening and replacement.

High-energy air plasma systems or HVOF spraying are generally used to apply the more successful coatings.

Extruder Dies. A common method of forming plastic pellets and chopped fibers is to extrude molten material through a die plate that has carbide-lined holes of appropriate size. Rotating blades operating against this die plate cut the product to the desired length. This operation is often performed under water.

A common wear problem exists, with the blades and debris eroding the stainless steel surfaces of the die, leaving raised edges on the tungsten carbide inserts. Thermal spray WC/Co coatings similar to those applied to knife blades greatly retard this action and extend the life of the die plates.

Textile Rolls. A number of roll variants are used in these industries to produce the proper fiber characteristics. Chromium oxide ceramics and tungsten carbides seem to predominate as coatings, but white alumina and the various $\text{TiO}_{2-x}\text{-Al}_2\text{O}_3$ materials are also used. Air plasma spraying has been the process of choice, although the use of HVOF is growing. The carbide rolls are generally ground smooth, but very special surface treatments are applied to the oxide coatings to generate appropriate finishes that impart certain characteristics to the fibers. These treatments may include using abrasive flap brushes, grit blasting, or combinations of these.

Recent Developments in Thermal Spray Application Technology

There are a number of key areas of industrial research and development that are contributing to the increased use of thermal spray processes and coatings. Examples of some of these are:

- The use of thermal spray coatings as replacements for electroplated hard chrome coatings
- The use of thermal spray polymer coatings as replacements for paints
- The use of the thermal spray process to remove lead-base paints

- New applications resulting from the development of the thermal spray forming process
- New applications resulting from the development of the cold spray process

Each of these, with the exception of chrome coating replacement, is discussed in this section. The section “Aerospace Applications” in this article describes the use of thermal spray coatings as replacements for hard chrome deposits. Of course, there are many other new applications that are being developed on an almost daily basis. These new applications areas, which are the result of improvements in both processing and feedstock materials, are described in numerous articles in the literature (refer in particular to papers published in the annual *International Thermal Spray Conference*, as discussed in the article “Guide to General Information Sources” in this Handbook).

Use of Thermal Spray Polymer Coatings as a Replacement for Paints (Ref 12)

Polymer spraying has gained significant commercial attention within the United States since the mid-1990s. Its acceptance as a replacement for paint is due to:

- No need for a primer
- A one-step process with no need for multiple passes
- No need for a topcoat
- Virtually zero volatile organic compounds
- Improved weathering resistance
- Excellent impact resistance

Thermal spray technology is also well suited for automation and can be adapted to existing robotic and automatic applicators. Polymer spraying is a one-coat process that acts as both the primer and the sealer, with no additional cure times, unlike the traditional multicoat painting processes. Thermal spraying is ideally suited for large structures that otherwise could not be dipped in a polymer suspension. Thermoplastic coatings can be repaired by simply remelting or applying extra material to the desired location. In addition, reports have shown that functionalized polyethylene polymers, such as ethylene methacrylic acid copolymer and ethylene acrylic acid, can be applied in high humidity as well as at temperatures below freezing.

Polymer Powders and Process Parameters. Polymer powders are specified by their chemistry, morphology, molecular weight distribution or melt-flow index, and particle size distribution. Spray parameters must be selected to accommodate each particular polymer formulation. A narrow particle size distribution is preferred to produce a homogeneous coating structure. A larger thermal input is required to melt larger particles and higher-molecular-weight organics. A large particle size or molecular weight distribution will facilitate the formation of numerous heterogeneities within the coating microstructure, creating voids, a range of splat aspect ratios, and degraded material. A processing window therefore exists for each polymer, where poor particle coalescence defines the lower limit and pyrolyzation defines the upper limit. It is also important to note that the polymer may also cross link, undergo chain scission, and/or oxidize during processing.

Polymer powder is axially fed into the combustion zone via a carrier gas. The polymer particulates are propelled through the flame where, on melting, they become droplets and are transported to the preheated substrate. As the molten particles impinge on the substrate, they deform and solidify, forming an interlaced network of splats. Porosity within the coating depends on the nature of the substrate surface, polymer melt viscosity, and decomposition products. The thickness of the coating is governed by the number of repeated passes of the spray gun across the substrate.

The heat source of the thermal spray torch can be a plasma, combustion flame, or combustion exhaust, as in the HVOF process. The technique is chosen on the basis of the polymer-melting characteristics. A simple combustion torch is well suited for low-melting-point polymers with a large processing window, such as polyethylene. High-melting-point polymers may require plasma deposition for maximum quality to melt the polymer without causing oxidation. Therefore, processing parameters must be selected for each polymer chemistry and deposition technique.

Two sets of distinct processes illustrate two fundamentally different spheres of polymer coatings. Both processes can generically be referred to as thermal spray; however, one process relies on a propane torch, and the other class of processes uses the heat source of either a direct current plasma torch or a HVOF gun. Thermal spraying of polymer powder is a 100% solids process. Polymers that can be applied using this process are listed in Table 9 and also include polyaryletherketone, fluoropolymers, polyesters, and others.

Intrinsic properties of the polymers of interest include melt processability, high chemical and impact resistance, electrical insulation, low coefficient of friction, ductility, and high toughness. Certain polymers also exhibit stability at elevated temperatures up to 300 °C (570 °F).

Applications. Thermal spraying of polymers is receiving increased attention in military and civil engineering sectors. The ability to apply thin (approximately 0.13 mm, or 0.005 in.) and thick (up to 6.4 mm, or 0.25 in.) coatings of polymers onto a variety of metals, ceramics, and composites of complex geometry provides solutions to component manufacture and on-site or factory

Table 9 Selection of polymers applied as coatings via thermal spray processes

Polymer	Maximum temperature resistance	
	°C	°F
Ethylene methacrylic acid copolymer (EMAA)	40–60	105–140
Polyethylene (PE)	40–80	105–175
Polypropylene (PP)	70	160
Nylon 6,6 (PA)	65	150
Polyphenylene sulfide (PPS)	110	230
Polyethylene-tetrafluoroethylene copolymer (PE-TFE)	160	320
Polyetheretherketone (PEEK)	125	255
Liquid-crystal polymers (LCPs)	250	480
Phenolic epoxy	130	265
Polyimide (PI)	300	570

Source: Ref 12

rehabilitation. The development of complex coating chemistries then may be dual phase; for example, composites of plastics and metals or plastics and ceramics is an area that needs further research.

Applications of polymer coatings currently include snowplow blades, pump impellers, tank linings, external pipe coatings, structural steel coatings, transfer chutes, and vacuum systems. Here, the intrinsic properties of polymers—such as high chemical resistance, high impact resistance, and high abrasion resistance—are used to advantage.

Use of Thermal Spray Process to Remove Lead-Base Paint (Ref 12)

An alternative means of surface preparation, patented by the U.S. Army Corps of Engineers, is on-site vitrification of hazardous waste. Thermal spraying of specially developed molten glass is used to encapsulate and chemically stabilize lead-base paint and rust. The encapsulant is then removed by chipping off relatively large chunks, which are readily removed for disposal. The objective has been to develop technology and guidance for maintenance where work will involve the removal of a coating material containing lead-base paint or other hazardous waste. Spray techniques, glass deposit chemistries, and encapsulation effectiveness are being tested and optimized.

Thermal Spray Vitrification Process. The U.S. Environmental Protection Agency (EPA) regulations have necessitated the removal and containment of toxic lead from lead-oxide-containing organic paints. Traditional grit blasting of hydraulic structures and highway bridges potentially involves the wide distribution of lead contaminant some distance from work sites and inflicts unacceptable health risks. The thermal spray vitrification process (TSVP) is an elegant technique that has been proposed to alleviate this problem. The TSVP is a “green technology” that seeks to provide an economic solution to the maintenance of existing structures, for example, bridges and other infrastructure. In this process, glass powder is melted in a high-temperature flame and sprayed onto the lead-painted surface. On impact of the molten glass droplet, lead diffuses and dissolves from the paint into the glass and forms a stable glass network that is resistant to lead leaching by acid attack.

Principles of the TSVP. When a molten droplet of glass strikes a lead-painted surface, the following phenomena occur. First, the droplet will spread, splatter, and deform to form an intimate contact layer between the glass and the painted surface. The molten glass, will penetrate and fill the pores and cavities in the surface by capillary transport. Mass transport rates and extent of reaction will depend on the properties of the molten glass, such as surface tension, kinematic viscosity, and heat-transfer coefficient, and on the properties of the coated surface. Next, there is diffusion, dissolution, and natural and forced convection of lead into the molten glass. The kinetics of this reaction will be controlled by the diffusivity of lead in the paint. The temperature and vapor pressure of the lead, as well as the pore structure of the glass, are the deciding factors for this phenomenon. There may also be solid-state interdiffusion of cationic species at the interface between the glass and painted surface, and the reaction layer will grow into the

glass and the substrate surface. The cation with the lowest diffusivity will be the rate-limiting specie for this reaction. In summary, the lead can be absorbed into the glass by gas-phase transport, dissolution into liquid slag, and solid-state interdiffusion of cationic species.

Dissolution and diffusion of lead into the glass are determined by the time duration for which the glass particles remain molten and at high temperature, respectively. In thermal spray processes, the coating material is melted in a flame and then quenched on impact onto a cold underlayer. The heating and cooling of the particles depend on the thermophysical properties of the particles in their liquid and solid states and their processing environments.

The composition of the glass basically decides its thermophysical properties. Selection of the glass composition for the lead-vitrification application should follow the following three criteria:

- Lead dissolution into the glass should follow the congruent path of the phase equilibrium diagram.
- The kinematic viscosity should be low.
- The glass should be stable in the desired range of lead concentrations.

Glass modifiers, such as alkali oxides, break the glass network and thereby reduce the melt viscosity. However, breaking of the network also weakens the glass structure and affects the stability of the glass. Intermediate oxides, such as alumina, titania, and lead oxide, for example, increase the stability of the glass, but because they increase the surface energy of the melt, the kinematic viscosity of the glass melt also increases with the addition of the intermediate oxides. Thus, the composition of the glass should be optimized so that it will have high lead absorptivity and lead-leaching resistivity.

The processing environment that decides the rate at which the lead and momentum transfer to and from the particle is controlled by a large number of spray-process parameters, such as the operating power level, stand-off distance, powder feed rate, gas flow rates, carrier gas flow rate, and gun traverse. A considerable amount of information already exists on the effect of spray variables on the properties of the coatings.

Applications of the Thermal Spray Forming Process (Ref 17)

While the thermal spray technology described in this article and throughout this Handbook originated primarily as a coating or surfacing process, thermal spray processing can also be used for forming freestanding materials/structures. Early applications included zirconia λ -oxygen sensors and bulk ceramic materials for crucibles. More recent research has dealt with spray forming of superconducting (high-critical-temperature) oxide ceramics and the synthesis of diamonds. Two growing and related areas for thermal spray forming of materials are the direct production of solid free-forms and tooling manufacture and repair.

Tooling Applications. Solid free-form fabrication has been demonstrated using a computer-aided design (CAD) model in conjunction with thermal spray deposition to fabricate freestanding solid parts. Successive planar layers have been deposited using

the wire arc spray process to incrementally build up three-dimensional shapes without the need for preformed mandrels. The wire arc spray process has also been used to replicate metallic, wooden, or polymeric tool and die patterns produced using CAD/rapid part prototyping techniques such as stereolithography (SLA). Materials sprayed for this application include zinc and aluminum alloys. Zinc is particularly attractive because of its low shrinkage on solidification, but it is limited by compatibility of the final application with relatively soft tooling. More demanding applications require the spraying of tool steel, which can damage thermally sensitive polymeric SLA models. Wire arc sprayed Kirksite-type alloys as the tool surface have also been proposed as a means of reducing tooling costs for many plastics molding processes, including structural foam, injection molding, compression molding, and vacuum forming. This approach is attractive for short production runs in which the cost of traditionally machined tooling can be very high. Coating thicknesses of 1.5 to 2 mm (60 to 80 mil) are typical, with reported cost savings ranging from a half to a twentieth of that of conventionally produced tooling.

A recent development in this area was the unique combination of spray forming of tool steel using nitrogen as the atomizing gas, with in situ shot peening to incrementally eliminate distortion due to stresses in the sprayed deposit. Tools and dies have been produced from 0.8% C steel, 13% Cr steel, M-2 tool steel, and 18/8 stainless steel using this technique.

Thermal spray forming of materials is also being used for the repair of tooling subsequently used in other metalforming processes. Air plasma sprayed coatings consisting of 70 vol% YSZ + 30 vol% CoNiCrAlY have been successfully applied to the surfaces of cast centrifugal casting molds. Service life was reportedly extended from 20 to 200 runs, with reduced product rejection rate.

Applications of the Cold Spray Process*

The cold spray process produces a well-defined and high-particle-density spray footprint. This has been exploited for producing near-net shapes and parts of various materials such as aluminum alloys. In general, it is difficult to produce uniform metal-matrix composites (MMCs) when the matrix and dispersant materials have widely varying characteristics. Cold spray can be used to produce any MMC coating and bulk form of these MMCs by simply blending the matrix and dispersant material and using the blend as spray feedstock. Apart from these noncoating applications, cold spray produces ultrapure coatings with unique characteristics that can be used for specific industrial applications, some of which are discussed as follows. Additional information on this process can be found in the article "Cold Spray Process" in this Handbook.

Brazing/Metal Joining Applications. Cold sprayed pure metal coatings have been studied for applications in brazing/metal joining processes. Two avenues have been studied. In one, a pure metal coating such as aluminum is cold sprayed onto the mating surfaces (e.g., steel), and then brazing flux is applied and brazed using conventional techniques. In the other technique, the brazing

flux itself is cold sprayed onto the mating surfaces, followed by heat treatment to join the parts.

High-Performance Applications. In the cold spray process, powder particles do not get heated to high temperature, and hence, deleterious high-temperature reactions such as oxidation, decomposition, and so on are avoided. Cold-spray-produced inclusion-free metal and alloy coatings have been studied for many high-performance applications, such as electrical/thermal conductivity, high-temperature oxidation protection, and corrosion resistance (Ref 18). Aluminum and oxygen-free high-conductivity copper coatings have been used as electrical conducting layers. The MCrAlY coatings are studied for oxidation protection in turbine and other industries. Ultrapure metal coatings, such as aluminum, titanium, and zinc, and alloy coatings, such as stainless steel, Inconel 625, nickel-chromium alloys, and others, are being researched for corrosion-resistant applications in many industries, including petrochemicals and electric utilities. Moreover, noble metal coatings such as niobium are used as diffusion barriers. Cold spray produces high-thermal conductivity layers, and this has been exploited in the commercial production of cold-spray-coated heat sinks, as shown in Fig. 30. Researchers have developed a nickel-aluminum bronze coating for tribological applications in corrosive wear environments of naval ships (Ref 19).

In the automobile industry, cold sprayed coatings are studied for various applications, such as copper coatings as current-carrying thick films, preparation of linear electrical machines by sequential spraying of metallization, soft and hard magnets and leads, zinc coatings for selective galvanizing to protect weld seams and hems, production and repair of rapid tooling, and thermal management layers (Ref 20).

Aerospace Applications. Because cold spray produces high-performance coatings, aerospace industry is keenly interested in adopting this technology (Ref 21). Typical applications studied

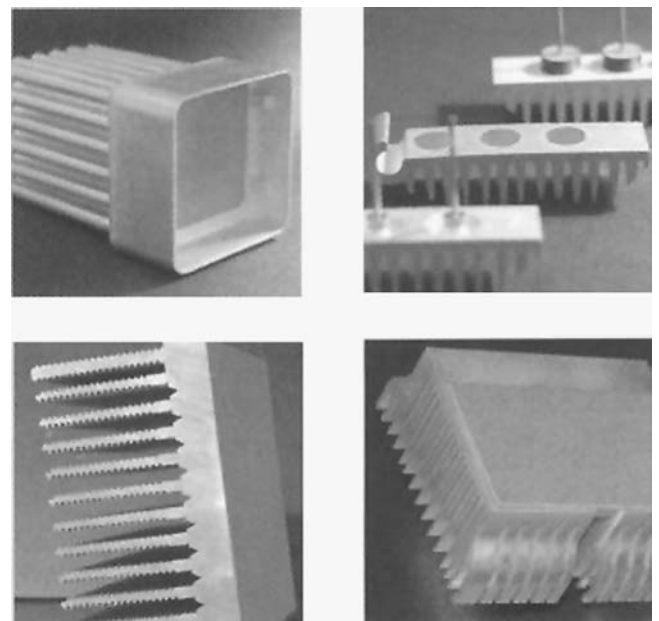


Fig. 30 Cold sprayed heat sinks

*This section was contributed by J. Karthikeyan, ASB Industries Inc.

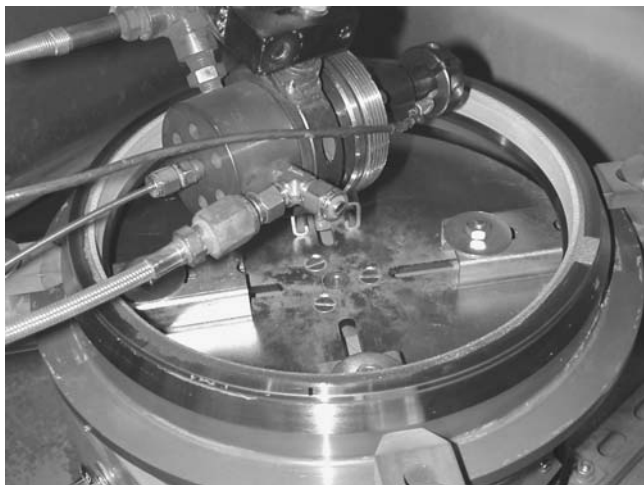


Fig. 31 Cold spray processing of a rocket engine manifold

include oxidation-resistant MCrAlY bond coats; high-thermal-conductivity coatings for thermal management; and production, refurbishment, and repair of critical components and parts. Figure 31 shows cold spray processing of a rocket engine manifold. This high-thermal-conductivity layer has resulted in protection of the manifold from the heat flux received from the combustion product by transferring the heat to the liquid hydrogen and oxygen.

ACKNOWLEDGMENTS

This article has been revised and updated from “Selected Applications” by Doug H. Harris, Lesson 5, *Thermal Spray Technology*, ASM International, 1992. Contributions to the revision were provided by Daryl E. Crawmer and William J. Lenling, Thermal Spray Technologies Inc.; Joseph P. Stricker, St. Louis Metallizing Company; Leo Glass, APS Materials Inc.; Richard Knight, Drexel University; D. Lee, Deloro Stellite Co. Inc., Stellite Coatings; and X. Chen and Z. Mutasim, Solar Turbines Inc. Additional credits are provided in footnotes within the article.

REFERENCES

1. R.A. Miller, Thermal Barrier Coatings for Aircraft Engines—History and Directions, *Thermal Barrier Coating Workshop*, March 1995 (Cleveland, OH), NASA Center for Aerospace Information, 1995, p 17–34
2. R.L. Jones, Thermal Barrier Coatings, *Metallurgical and Ceramic Protective Coatings*, K.H. Stern, Ed., Chapman & Hall, 1996, p 194–235
3. R.K. Schmid, F. Ghasripor, M. Dorfman, and X. Wei, An Overview of Compressor Abradables, *ITSC 2000, First International Thermal Spray Conference*, May 2000 (Montreal, Quebec, Canada), ASM International, p 1087–1093
4. F. Ghasripor, R.K. Schmid, and M.R. Dorfman, Development of Advanced Materials for Clearance Control Applications up to 850 °C, *Ninth Cimtech World Forum on New Materials, Symposium III, Surface Engineering*, P. Vincenzini, Ed., Techna Srl, 1999, p 641–650
5. “Bearing and Bushing Alloys—Chemical Composition of SAE Bearing and Bushing Alloys,” SAE J460, Society of Automotive Engineers International
6. Y. Nava, Z. Mutasim, and M. Coe, Abradable Coatings for Low-Temperature Applications, *Thermal Spray 2001: New Surfaces for a New Millennium (Proceedings of the International Thermal Spray Conference)*, C.C. Berndt, K.A. Khor, and E. Lugscheider, Ed., May 2001 (Singapore), ASM International, 2001, p 263–268
7. Z. Mutasim and Y. Nava, Development and Performance Evaluation of Thick Air Plasma Sprayed Thermal Barrier Coatings, *Thermal Spray: Surface Engineering via Applied Research (Proceedings of ITSC 2000)*, C.C. Berndt, Ed., May 2000 (Quebec, Canada), ASM International, 2000, p 1325–1330
8. Y. Nava, Z. Mutasim, and M. Coe, Ceramic Abradable Coatings for Applications up to 1100 °C, *Thermal Spray 2001: New Surfaces for a New Millennium (Proceedings of the International Thermal Spray Conference)*, C.C. Berndt, K.A. Khor, and E. Lugscheider, Ed., May 2001 (Singapore), ASM International, 2001, p 119–126
9. Y. Nava, “Flame Impingement Tests at 982 °C of YSZ, CaTiO₃ and CeO₂-TSZ Ceramic Coatings,” International Thermal Spray Conference (Orlando, FL), ASM International, May 2003
10. B. Walser, The Importance of Thermal Spray for Current and Future Applications in Key Industries, *SPRAYTIME*, Vol 10 (No. 4), fourth quarter, 2003, p 1–7
11. W.H. Hunt and D.B. Miracle, Automotive Applications of Metal-Matrix Composites, *Composites*, Vol 21, ASM Handbook, ASM International, 2001, p 1029–1032
12. M.L. Berndt and C.C. Berndt, Thermal Spray Coatings, *Corrosion: Fundamentals, Testing, and Protection*, Vol 13A, ASM Handbook, ASM International, 2003, p 803–813
13. R.H. Unger, Thermal Spray Coatings, *Corrosion*, Vol 13, *Metals Handbook*, 9th ed., ASM International, 1987, p 459–462
14. H. Herman and R.A. Sulit, Thermal Spray Coatings, *Welding, Brazing, and Soldering*, Vol 6, ASM Handbook, ASM International, 1993, p 1004–1009
15. T. Peterman, The High Volume Production of Plasma Sprayed Dielectric Coatings, *International Thermal Spray Conference 2003, Thermal Spray: International Advances in Coatings Technology*, C.C. Berndt, Ed., ASM International, 2003, p 309–320
16. L. Pawlowski, Technology of Thermally Sprayed Anilox Rolls: State of the Art, Problems, and Perspectives, *J. Therm. Spray Technol.*, Vol 5 (No. 3), Sept 1996, p 317–334
17. R. Knight and R.W. Smith, Thermal Spray Forming of Materials, *Powder Metal Technologies and Applications*, Vol 7, ASM Handbook, ASM International, 1998, p 408–419
18. J. Karthikeyan, T. Laha, and A. Agarwal, Microstructural and Electrochemical Characterization of Cold-Sprayed 1100 Alu-

- minum Coating, to be presented at International Thermal Spray Conference 2004, May 2004 (Osaka, Japan)
19. R.B. Bhagat, M.F. Amateau, A. Papyrin, J.C. Conway, Jr., B. Stutzman, and B. Jones, Deposition of Nickel Aluminum Bronze Powder by Cold Gas Dynamic Spray Method on 2618 Al for Developing Wear Resistant Coatings, *Thermal Spray: A United Forum for Scientific and Technological Advances*, ASM International, 1997, p 361–367
 20. R. McCune, Potential Applications of Cold-Spray Technology in Automotive Manufacturing, *Thermal Spray: Advancing the Science and Applying the Technology (Proc. ITSC 2003)*, (Orlando, FL), ASM International, 2003, p 63–70
 21. J. Haynes and J. Karthikeyan, Cold Spray Copper Application for Upper Stage Rocket Engine Design, *Thermal Spray: Advancing the Science and Applying the Technology (Proc. ITSC 2003)*, (Orlando, FL), ASM International, 2003, p 117–122
- ### SELECTED REFERENCES
- G. Barbezat, S. Keller, and K.H. Wegner, Rotaplasma Applications in the Automotive Industry, *Thermal Spraying—Current Status and Future Trends*, Vol 1, High Temperature Society of Japan and Japan Welding Research Institute, 1995, p 9–13
 - R.A. Carello, D.M. Parks, and J.A. Apostolos, “Development, Testing and Field Application of Metallized Cathodic Protection Coatings on Reinforced Concrete Substructures,” Report FHWA/CA/TL-89/04, State of California, DOT, Division of Construction, Office of Transportation Lab, May 1989
 - J.R. Davis, Ed., *Handbook of Materials for Medical Devices*, ASM International, 2003
 - D.H. Harris, “Tailored Dielectrics,” final report, U.S. Army-ECOM R&D, TR-ECOM-0201, U.S. Department of Defense, April 1974
 - D.H. Harris, Practical Aspects of Ultra-Thick Thermal Barrier Coatings, *Surface Modifications and Coatings (Proceedings of the International Thermal Spray Conference, 1985)*, R.D. Sisson, Jr., Ed., American Society for Metals, 1986, p 91–96
 - D.H. Harris, “Bio-Inert CP-Ti and Ti-6Al-4V Coatings by the Arc-Plasma Spray Process,” Titanium Development Assoc., Dayton, OH, 1990
 - D.H. Harris, Overview of Problems Surrounding the Plasma Spraying of Hydroxyapatite Coatings, *Thermal Spray Research and Applications (Proceedings of the Third National Thermal Spray Conference, 1990)*, T.F. Bernecki, Ed., ASM International, 1991 p 419–423
 - D.H. Harris and F. Rosenbaum, “Ferrite/Dielectric High Power Phase Shifter Development, Part IV: Arc-Plasma Spray Fabrications and Material Studies,” final report, Contract F33615-72-C-1034, AFAL-TR-73-250, Department of Defense, Aug 1973
 - R. Hillary and B. Pilsner, “Thermal Barrier Coating Life Prediction Model,” final report, Contract NAS3-2343, NASA, 1988
 - A.V. Levy, The Performance of Ceramic Coatings on Diesel Engine Combustion Zone Components, *Thermal Spray Technology: New Ideas and Processes (Proceedings of the Second National Thermal Spray Conference, 1988)*, D.L. Houck, Ed., ASM International, 1989, p 263–272
 - M. Naylor, “Development of Wear Resistant Ceramic Coatings for Diesel Engine Components,” final report, Contract 86X-SA581C, U.S. Department of Energy, Aug 1991
 - M.C. Nestler, G. Prenzel, and T. Seitz, HVOF Spraying vs. Hard Chrome Plating—Coating Characteristics and Aircraft Applications, *Proceedings of the 15th International Thermal Spray Conference*, May 1998 (Nice, France), ASM International, 1998, p 1073–1078
 - R.C. Novak, A.P. Matarese, and R.P. Huston, Development of Thermal Barrier Coatings for Diesel Applications, *Thermal Spray Technology: New Ideas and Processes (Proceedings of the Second National Thermal Spray Conference, 1988)*, D.L. Houck, Ed., ASM International, 1989, p 273–281
 - A. Ohmori, K.C. Park, and Y. Arata, Photoelectrochemical Properties of Plasma Sprayed TiO₂ Coatings, *Thermal Spray Research and Applications (Proceedings of the Third National Thermal Spray Conference, 1990)*, T.F. Bernecki, Ed., ASM International, 1991, p 509–515
 - A.R. Parks, Aluminum Sprayed Coatings onboard U.S. Navy Ships: A Ten Year Overview, *Thermal Spray: Advances in Coating Technology (Proceedings of the First National Thermal Spray Conference, 1987)*, D.L. Houck, Ed., ASM International, 1988, p 389–392
 - W.J. Pejryd, Thermal Barrier Coatings—Why, How, Where and Where to, *Proceedings of the 15th International Thermal Spray Conference*, May 1998 (Nice, France), ASM International, 1998, p 1531–1542
 - P.W. Schilke, A.D. Foster, J.J. Pepe, and A.M. Beltran, Advanced Materials Propel Progress in Land-Based Gas Turbines, *Adv. Mater. Process.*, Vol 141 (No. 4), April 1992, p 22–30
 - R.H. Unger, Thermal Spraying of Bridges, *Thermal Spray: Advances in Coating Technology (Proceedings of the First National Thermal Spray Conference, 1987)*, D.L. Houck, Ed., ASM International, 1988, p 399–406

Introduction to Testing and Characterization

Walter Riggs, TubalCain Company Inc.

ASSESSING THE USEFULNESS OF THERMAL SPRAY COATINGS requires understanding, developing, and utilizing appropriate testing and characterization methods. While the materials resulting from thermal spray processes are often very different from their wrought, forged, and cast counterparts, standard testing and characterization methods used to test them have been largely borrowed from the other materials science disciplines.

At the time of the original writing of this Section of the Handbook (as a lesson in the 1992 ASM education course *Thermal Spray Technology*), a limited amount of work had been carried out to understand how these “borrowed” tests interacted with thermal spray coatings, and how the coatings responded to the tests. Little data were available to understand how sensitive the coatings were to variations in test parameters (e.g., how much pressure must be applied when grinding and polishing coatings metallographically). Since the original publication, much has been accomplished in the field of metallography, both by way of experimentation and by way of round-robin comparative testing. As a result, a consensus has been emerging regarding the true microstructure of coatings.

In the article “Metallography and Image Analysis” in this Section, examples are given to show the response of coatings to the metallographic methods used; specific procedures are recommended for achieving good metallographic results. (Note that more than one metallographic procedure can be used to produce the desired results; the authors are merely providing recommended procedures.) For metallurgical and materials engineers, understanding the true nature of coating microstructures is essential and a prerequisite to formulating material processing/microstructure/property relationships, which are critical to the mechanical engineering community and applications designers. It is imperative that applications designers be provided with materials with consistent properties and the necessary tests to evaluate those properties. Hence, it is hoped that these updated articles will help the thermal spray community to gain further insight into these materials.

This Section of the Handbook is intended to help the reader to:

- Understand the more common test methods applied to thermal spray coatings
- Identify the critical attributes in the tests
- Explain how the materials themselves respond to the various test methods

Other Sections in this Handbook discuss the raw materials (powders) and the use of various thermal spray systems to process those powders. This Section focuses on the test methods them-

selves, including those test parameters that can be varied and the influence of each on the results obtained. While some recommendations will be given for common coatings of interest (to consistently and correctly reveal the measured attributes), it must be understood that these are suggestions or recommendations only, and that governing authorities’ requirements for their specific test methods for coatings must be adhered to.

This Section deals with the more commonly used test and characterization methods: metallography, image analysis, hardness (superficial hardness and microhardness), tensile adhesion testing, x-ray diffraction, nondestructive testing, and powder characterization (particle size distribution and chemistry). Some advanced test methods, such as erosion, abrasability, and wear, are not covered; references, however, are provided so that the reader can independently investigate those methods.

Finally, it is important to understand that this Section does not present “recipes” for each test method. Rather, the intent is to initiate an understanding of the test methods themselves, to introduce readers to the arena of testing and characterization in general as response-generating methods that must be as well understood as the results themselves, and to provide, where appropriate, fundamental laboratory guidelines.

Metallurgical and Materials Nature of Thermal Spray Coatings Compared to Wrought, Forged, and Cast Materials

Thermal Spray Coating Formation. Thermal spray coatings are produced by the injection of powders, usually in the range of 5 to 50 μm (0.0002 to 0.0020 in.) diameter, and/or wires and rod feedstocks into a hot gas jet, through which they travel and are subsequently deposited onto a relatively cool substrate. The total dwell time for particles, which travel at speeds ranging from approximately 75 m/s (250 ft/s) to almost 1000 m/s (3000 ft/s) for the higher-velocity processes, is on the order of 1 ms (1/1000 s) or less. During this time, the particles must undergo a change from ambient, or room, temperature to slightly above their melting temperature (at least on their periphery) and, upon impact with the substrate (which is usually below 150 °C, or 300 °F), cool quickly. The particle heating/melting/resolidification/cooling steps must take place at rates near or exceeding 5×10^5 °C/s (10^6 °F/s). As such, thermal spray processes are usually considered to be rapid solidification processes. In the case of metals, the ability of the resolidifying material to first form grains, then undergo grain

growth, is suppressed. As such, grains present in thermal spray metallic coatings are typically 1 μm or less.

Formation of Wrought, Cast, and Forged Materials. By comparison, wrought, cast, and forged materials remain at elevated temperatures for considerable periods of time. Some steel castings, for example, are known to take longer than 1 h to complete the solidification process; thereafter, they are cooled slowly to room temperature. Nickel-base superalloys are subjected to complex heat treatments to produce “gamma prime” and other precipitates of the correct size and shape, and these heat treatments can take longer than 24 h. Considerable energy and time are invested to bring materials to the proper bulk temperature prior to forging, and many forging temperatures are above 1095 °C (2000 °F).

Grain Sizes. The result of these comparatively long solidification times and exposures to high temperatures is that grain sizes are much larger than those found in thermal spray coatings. This conclusion is obvious in that the grains produced are usually observable at relatively low magnifications via light optical microscopy, whereas electron microscopy is usually necessary to see grains in thermal spray coatings; for example, a 0.5 μm diameter grain would have to be magnified 25,000 times to be seen as a 12.5 mm, or 0.5 in., grain on a Polaroid micrograph. Such observations are usually the province of scanning electron microscopy (SEM).

The ASTM grain size chart is shown in Table 1. Notice that a grain size of 12, considered extremely small, is on the order of 6 μm , which is still 10 to 100 times larger than most grains formed during thermal spray processing. This significant difference in grain size also has consequences when the materials are deformed. (Most testing methods discussed herein will necessarily involve coating deformation.) Figures 1 to 3 show typical grain sizes for wrought, cast, and forged materials, and Fig. 4 shows grain size typical of plasma spray processing.

Interfacial Strengths. The differences between thermal spray materials and wrought, cast, and forged metals goes far beyond their grain sizes, however. The latter material types usually solidify from the molten state by a competitive process, wherein clusters of atoms either exceed a certain critical radius by gaining additional atoms (exceeding the critical radius is necessary to

lower the total free energy of the cluster) and nucleate new grains, or the cluster loses atoms to the surrounding liquid and thus disappears. As solidification continues, the surviving grains grow into the surrounding molten metal until they encounter another growing grain, where they stop. There is usually a difference in orientation of the crystalline structures between adjacent grains. The strength of these materials is lower than that of a single, defect-free crystal (i.e., a single grain), which has been estimated for some materials to be in excess of 70,000 MPa (10,000 ksi). However, their strengths are still quite high. High-strength steel castings can have tensile strengths exceeding 1,700 MPa (250 ksi),

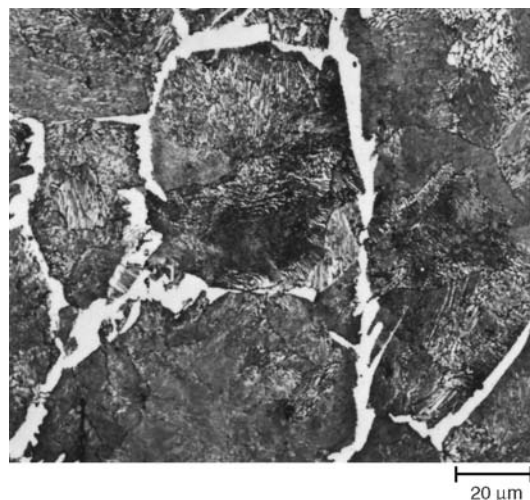


Fig. 1 1045 steel bar normalized by austenitizing at 1095 °C (2000 °F) and cooling in air. Structure is pearlite (gray) with a network of grain-boundary ferrite (white) and a few side plates of ferrite. Picral etch. Magnification, 500 \times

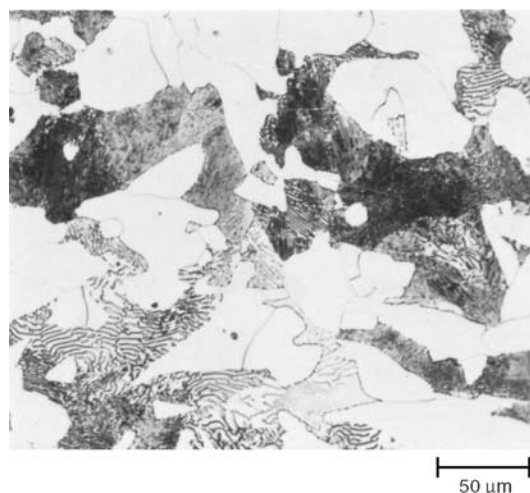


Fig. 2 ASTM A 27 steel, grade 70-36 (0.26 C, 0.71 Mn), 150 mm (6 in.) thick, normalized by austenitizing at 900 °C (1650 °F) for 6 h and air cooling. The microstructure consists of lamellar pearlite (gray and black) and ferrite (white). Nital etch. Magnification, 250 \times

Table 1 Comparison of grain-size measuring systems

ASTM No.	Grains per mm^2 at 1 \times (per in.^2 at 100 \times)	Average grain diameter, mm
-3	1 (0.06)	1.000
-2	2 (0.12)	0.750
-1	4 (0.25)	0.500
0	8 (0.5)	0.350
1	16 (1)	0.250
2	32 (2)	0.180
3	64 (4)	0.125
4	128 (8)	0.091
5	256 (16)	0.062
6	512 (32)	0.044
7	1024 (64)	0.032
8	2048 (128)	0.022
9	4096 (256)	0.016
10	8200 (512)	0.011
11	16,400 (1024)	0.008
12	32,800 (2048)	0.006

the tensile strengths of superalloy forgings are typically above 1,200 MPa (175 ksi), and even high-strength low-alloy (HSLA) steels—which are low-carbon steels containing the least amount of alloying element that still provides proper yield strength without heat treatment—have tensile strengths in the range of 340 to 550 MPa (50 to 80 ksi).

In comparison, thermal spray coatings generally have tensile strengths in the range of 27.5 to 55 MPa (4 to 8 ksi), with techno-

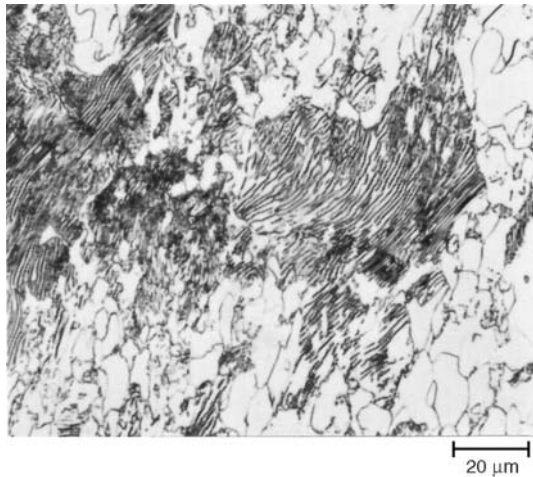


Fig. 3 4047 steel forging (longitudinal section, 16 mm, or 0.625 in., thick) austenitized at 830 °C (1525 °F), cooled to 665 °C (1225 °F) and held 6 h, furnace cooled to 540 °C (1000 °F), then air cooled. Ferrite (white) and lamellar pearlite (dark). 2% nital etch. Magnification, 500×

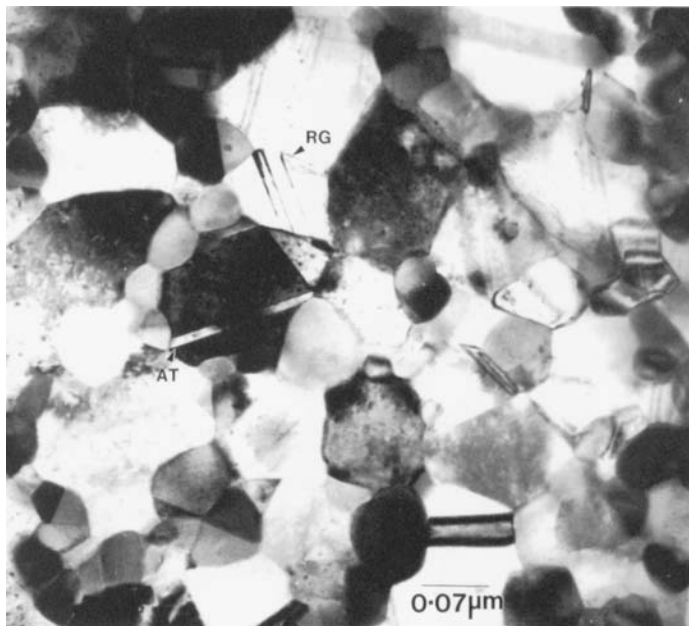


Fig. 4 Thin section of a vacuum plasma-sprayed nickel-base alloy coating (Metco 700) as observed by transmission electron microscopy. The microstructure displays fine, equiaxed, recrystallized grains. RG, recrystallized grains. AT, annealing twins

logical developments bringing an incremental evolution to slightly higher strengths (nearer to, or slightly exceeding, 83 MPa, or 12 ksi). It should be noted that these strengths are determined by testing a sample of the coating epoxied to test bars, as will be discussed in the article “Testing of Coatings” in this Section; the coating is not a freestanding shape, and as such does not afford gripping directly, as do wrought, cast, and forged materials.

Bonding of Thermal Spray Materials. Coatings owe what strength they have to mixed-mode bonding between the splats (i.e., a mixture of the three types of bonding, explained in the following). Bonding of thermal spray coatings is distributed among three types:

- True metallurgical bonding, where heat from an arriving depositing particle is sufficient to cause a local rise in the temperature of the substrate material or previously deposited particle, allowing remelting and/or diffusion/mixing, or simply diffusion
- Mechanical interlocking of arriving particles with either a previously roughened substrate or the rough topography of previously deposited particles
- Weak physical bonding, via van der Waals forces, between particles

Various estimates have been made of the degree of each type of bonding; it can be concluded that the degree of true metallurgical bonding is typically low; otherwise, the tensile strengths would approach those of the other materials discussed previously. When fractured and observed via SEM, most splats appear separated from their neighbors (Fig. 5), evidence of a lack of metallurgical bonding between them. The buildup of a thermal spray deposit is

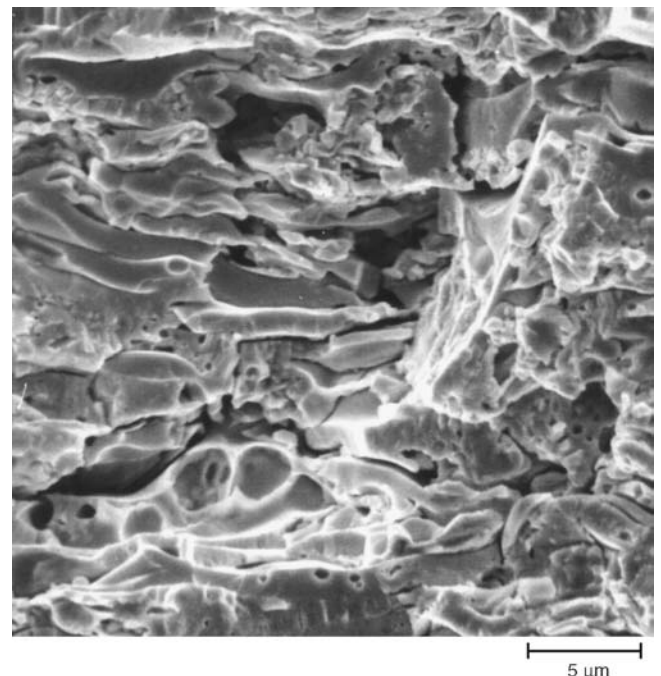


Fig. 5 Fracture surface of a plasma spray coating. Magnification, 3000×

illustrated in Fig. 6. Depending on their size, chemistry, phase, and trajectory in the spray jet, arriving particles will be either fully or partially molten or unmelted. (Evidence has been presented and defended that some powders even evaporate, so that vapor “clouds” arrive at the substrate and form a part of the buildup.) The resulting network is a heterogeneous patchwork of deposited particles, or splats, somewhat akin to a stone wall built using different size stones, the mortar being deposited haphazardly and sparingly. Various types of microstructure exist within each splat, as shown by Fig. 7. The flat, outer (“pancaked”) perimeter, having been thoroughly melted, spreads out quickly to the surrounding topography, while the cooler core, if molten, is much more viscous and resists spreading/wetting. (If not molten, then the core is possibly heated to a more ductile state than those particles that remain totally unmelted, and therefore undergo some deformation upon impact.) The theory of partially molten particles arriving at the substrate is defended by Fig. 8, which illustrates a particle captured midstream at the spray distance. The columnar structure suggested by Fig. 7 in the outer, pancaked regions is supported by Fig. 9, which shows the columnar structure within a fractured surface, as observed by SEM.

Interfacial Chemistry of Thermal Spray Coatings. The final difference between thermal spray coatings and their wrought, cast,

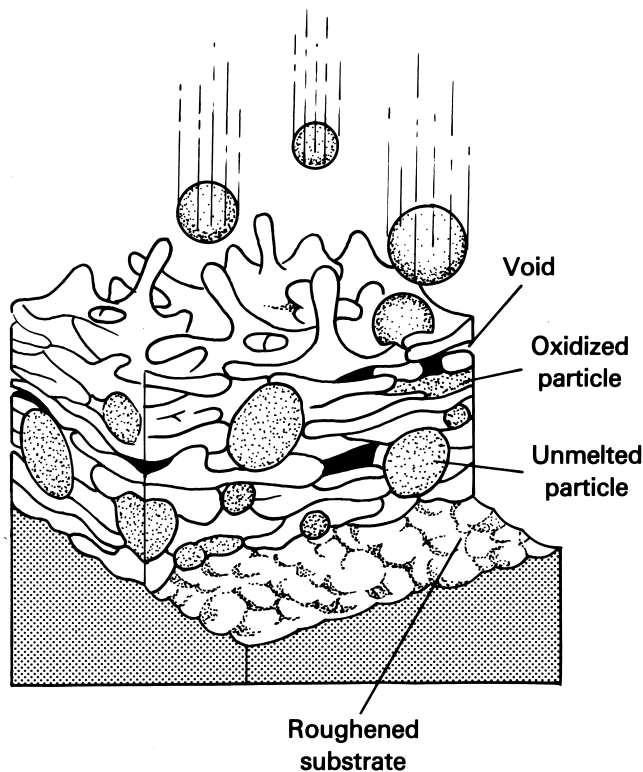


Fig. 6 Schematic showing the buildup of a plasma spray coating. Molten particles spread out and splatter as they strike the target, at first locking onto the irregularities of the roughened surface and then interlocking with one another. Voids result as the growing deposit traps air. In some cases, particles overheat in the flame and become oxidized; other particles may not melt and simply get embedded in the deposit.

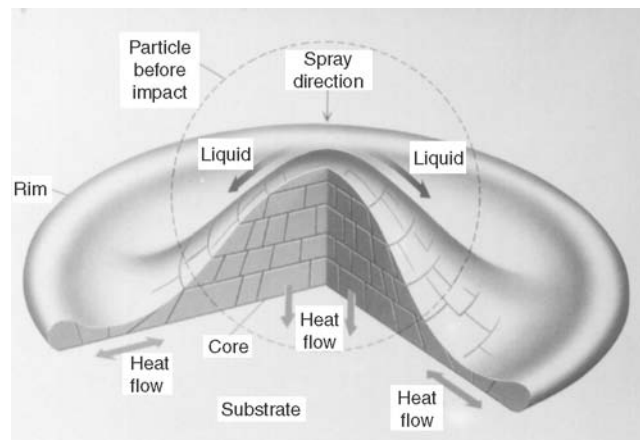


Fig. 7 Splat results when a microscopic droplet of molten coating material strikes a surface, flattens out, and solidifies. As a splat solidifies, heat is lost to the substrate beginning at the center, which strikes the surface first. A solidified core forms, and remaining melt spills off it and hardens into a raised rim.

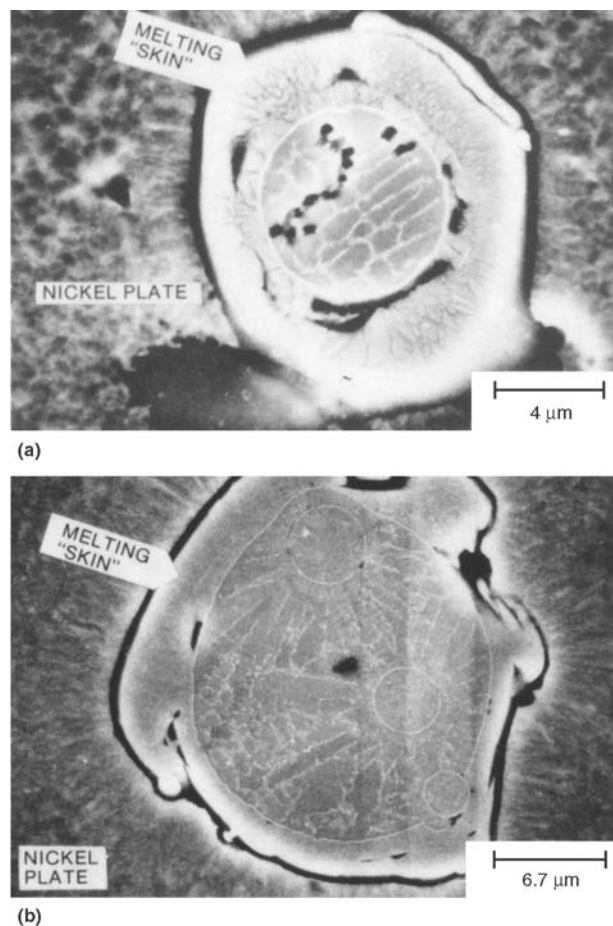


Fig. 8 Powder particle captured midstream in plasma plume illustrating the outer molten layer, in addition to preferential intercellular melting. Both (a) and (b) are SEMs of René 80 collected powder particles, sandwiched in nickel plate.

and forged counterparts is the interfacial chemistry between splats. As a result of spray jet fluctuations brought about by power supply fluctuations (among other sources), air, containing oxygen, nitrogen, and traces of other gaseous molecules, is entrained into the spray jet. Since the outer surfaces of molten metallic particles are at a high temperature, depending on the free energies of formation and the kinetics of reaction, oxides and nitrides may form. While x-ray diffraction, a bulk material evaluation method, will easily detect bulk phases in the coating, detection of periodic surface oxides and nitrides is more difficult. Oxides and nitrides may, however, significantly affect coating properties. They may impede or stop the diffusion of metal atoms from the impacting particle into the substrate or previously deposited particle, greatly influencing coating strength and performance.

Summary. The methods of formation, grain sizes, and interfacial strengths and chemistries of thermal spray coatings are remarkably different from their wrought, cast, and forged counterparts. That these differences should have a significant impact on properties as measured by various test methods is no surprise. It would be expected, for example, that if the degree of metallurgical bonding could be improved, or if chemical purity could be obtained/maintained at particle surfaces, strengths would improve significantly. Indeed, they do, as evidenced by plasma spraying in a low-pressure controlled-atmosphere chamber: Oxide and nitride formation is significantly reduced, allowing very clean interfaces. If the substrate is sufficiently preheated prior to coating, room-temperature strengths approaching 70% of wrought materials can be obtained. If these materials are then post-spray heat treated, strengths equaling or exceeding wrought materials can be obtained. The impact of the differences described above for air

plasma spray coatings (not low-pressure, chamber-sprayed coatings) on testing and characterization methods will become apparent in the following discussions.

Testing and Characterization Methods as Response Generators

Having demonstrated that the nature of thermal spray coatings is very different from other types of materials, the nature of the test methods themselves must be discussed. Test methods are response generators. This means that the test method uses loads or applies some signal, stress, or other stimulus to the coating. The material responds to the load, signal, or stimulus, such as heat, in a way dictated by its physical, chemical, and structural properties. A tool steel specimen, for example, responds to the load of a hardness indenter much differently than a bronze specimen. A solid bronze specimen responds to the indenter much differently than a porous bronze specimen. A porous polymer, such as a styrofoam coffee cup, responds to the application of heat much differently than a metal, such as aluminum; the styrofoam cup keeps the coffee hot and your hand cool, while the aluminum cup does the opposite. A sample of pure copper will have different ultrasonic wave velocity and attenuation than a block of wood.

Test methods, then, are processes that load the material being tested, or apply some signal or stimulus to it, *and* have the capability of measuring the material's response. For example, a hardness testing machine applies a load via an indenter and measures the distance traveled by the indenter after application of the load, giving a measure of relative hardness. An ultrasonic transducer both sends and receives signals. A tensile test machine loads the specimen and records the pressure required to cause failure. In this way, the test methods are known as "response generators."

The result of practically every test method is a number: hardness, tensile strength, percent porosity, dielectric strength, ultrasonic wave velocity, electric potential with respect to some reference, and so on. The numbers generated from tests are presumed to be representative of some useful property, such as resistance to buckling load for a beam in a structure, resistance to corrosion for a buried coated pipeline, or resistance to wear. In order for the numbers, and thus the tests and materials being tested, to be useful, some degree of confidence is required. The usual measure of confidence is expressed statistically as the results of a significant number of tests; for a high degree of confidence, minimal variance or standard deviation from the average value should be exhibited.

For example, from typical numbers for the tensile strength of high-strength tool steels (more than 1700 MPa, or 250 ksi), a design engineer might be encouraged by the high average tensile strengths, but might be disheartened if the range of the tests were from 340 to 5100 MPa (50 to 750 ksi), with no explanation for the variance. Fortunately, the grouping of test responses for wrought, cast, and forged materials is much tighter. The spread of values for thermal spray coatings can be much different: Some recent tensile tests for one coating system sprayed three times using the same set of parameters varied from approximately 27.5 MPa (4 ksi) to more than twice that.

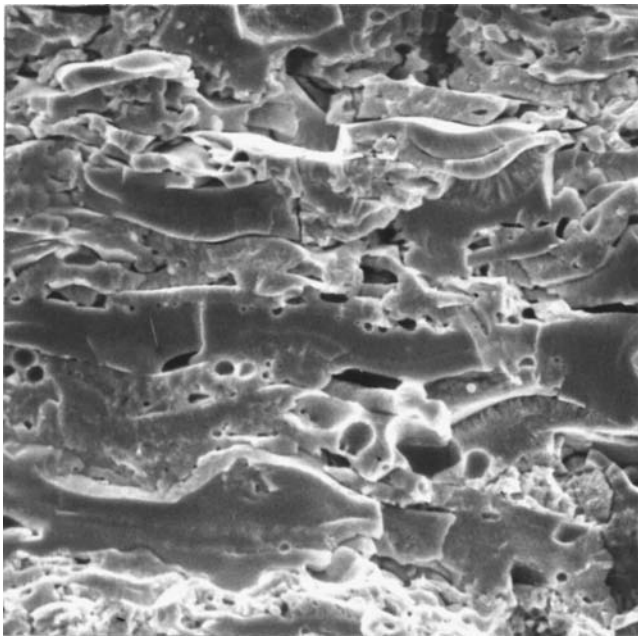


Fig. 9 SEM of fractured surface of plasma spray coating showing columnar grains

Effect of Metallurgical Bonding and Interfacial Chemistry on Test Results. As discussed, thermal spray coatings likely contain varying degrees of metallurgical bonding, as well as differences in interfacial chemistries if oxidation and nitridation occur. It can be expected that the numbers generated by tests will vary as a function of these factors. If some unknown parameter, such as the radial location of the powder injector port, is both uncontrolled and significant, then the degree of metallurgical bonding and interfacial chemistry can vary considerably, and the numerical results of tests applied to such coatings could also be expected to vary. In addition to process parameter variations, the initial powder size distribution, chemistry, and phase composition may also vary from time to time during the process. It is also to be expected that a region of the coating free of oxide clusters, unmelted particles, and porosity will respond differently than an adjacent area containing such features.

With such low percentages of true metallurgical bonding, thermal spray coatings should, from a first principle standpoint, be considered from an *individual splat response*. Each splat and its associated metallurgical/mechanical/physical bonding are loaded either by the test (for splats near the surface) or by adjoining splats (for splats in the center of the coating volume or away from the surface being loaded). Depending on their degree of bonding and their local neighborhood splat network, splats might be expected to respond similar to a homogeneous wrought material if movement were prohibited. As an example, consider microhardness testing in a metallurgical mount: If the coating were very dense, those particles under the load of the indenter would be constrained to move, resisting the movement of the indenter and giving an indication of high hardness. If the material were less dense, however, some of the splats could move under the applied load, allowing the indenter to penetrate further, giving an indication of lower hardness.

It is to be expected that individual splats are able to respond to loads such as those applied during metallographic polishing in a variety of ways:

- By resisting the load and not moving (high degree of metallurgical bonding or constraint of the surrounding splat network)
- By rotating about the metallurgical bond as the surrounding splat network allows (lower-density coatings, lower degree of metallurgical bonding)
- By deforming plastically and being translated by the test load (i.e., along the polished surface in metallography)
- By being totally removed from the coating (little or no metallurgical bond)

In addition, it should be noted that ceramic constituents within coatings, such as oxides, would not be expected to be plastically deformable, but in fact are subject to fracture, owing to very low fracture toughness, and upon fracture, leave behind a void or pore.

Summary. Testing and characterization schemes as applied to thermal spray coatings are very complex. The tests used are response generators that both impart loads, signals, or other stimuli to materials, and allow for the measurement of the material's response; the coatings themselves respond to the tests as individual splats (and as cumulative individual splats across the entire splat network). Compared to wrought, cast, and forged materials, the testing of thermal spray coatings is very complex, and both the test

method and the nature of the coating material must be well understood.

Testing and Characterization as a Diverse Materials Engineering Opportunity

The testing and characterization of thermal spray coatings is a diverse field of study covering all types of materials: metals, ceramics, polymers, composites, cermets, and so forth. In addition, a variety of scientific and engineering disciplines are involved, including metallurgical engineering and materials science, mechanical engineering for the design of loading devices, and electrical engineering for the design of electrical test methods.

Regarding metallurgical and materials science, as an example, in the metallographic preparation of coatings for microscopic observation, the coating and the substrate onto which it is deposited must first be prepared for sectioning; this may involve encapsulating it in a suitable protective film of epoxy (polymer). The prepared sample is then subjected to sectioning using abrasive wheels made of aluminum oxide or silicon carbide (ceramics) bonded to a rubberlike or polymeric backing material or diamond blades comprising synthetic diamonds cemented to a metallic blade. The sectioned sample is then mounted either in a thermosetting or thermoplastic hot mounting material, or in a room-temperature curing epoxy, where polymer chain formation and cross-linking are important.

Following this procedure, the cured mount is then subjected to abrasion using silicon carbide or aluminum oxide papers or proprietary lapping disks with rolling abrasives, followed by polishing using aluminum oxide, diamond, colloidal silica, or other materials embedded or deposited on various polishing cloths with a wide range of naps, resiliencies, etc. In the process of metallographic preparation, one must consider material deformation processes, including metallic slip, twinning, shearing, ceramic fracture modes, and polymeric responses to the abrading and polishing mechanisms.

The tensile adhesion test offers another example. Usually, an uncured epoxy is applied to the coating and adjoining test bars at room temperature. The assembly is cured at an elevated temperature, and then tested in a standard tensile test machine. The curing cycle (temperature, time, and possibly rate of heating and cooling) is critical to ensuring correct polymeric chain formation and cross-linking, just as in the preparation of metallurgical mounts.

Many engineers and scientists devote their entire careers to test methods development. An understanding of the nature of the materials being tested, the nature of the test method as a response generator, the accuracy of the response measuring/recording device, and some knowledge of the expected range of measured responses (i.e., a statistical understanding of the process) must all be developed.

SELECTED REFERENCES

- C.C. Berndt and S. Sampath, Ed., *Thermal Spray Science and Technology*, Proc. Eighth National Thermal Spray Conf., 11–15 Sept 1995 (Houston), ASM International, 1995

- C.C. Berndt and S. Sampath, Ed., *Thermal Spray Industrial Applications*, Proc. Seventh National Thermal Spray Conf., 20–24 June 1994 (Boston), ASM International, 1994
- C.C. Berndt, Ed., *Thermal Spray: Research, Design, and Applications*, Proc. Sixth National Thermal Spray Conf., 7–11 June 1993 (Anaheim, CA), ASM International, 1993
- C.C. Berndt, Ed., *Thermal Spray: International Advances in Coatings Technology*, Proc. 13th International Thermal Spray Conf., 29 May–5 June 1992 (Orlando, FL), ASM International, 1992
- T.F. Bernecki, Ed., *Thermal Spray Coatings: Properties, Processes and Applications*, Proc. Fourth National Thermal Spray Conf. 4–10 May 1991 (Pittsburgh), ASM International, 1992
- T.F. Bernecki, Ed., *Thermal Spray Research and Applications*, Proc. Third National Thermal Spray Conf., 20–25 May 1990 (Long Beach, CA), ASM International, 1991
- D.L. Houck, Ed., *Thermal Spray Technology: New Ideas and Processes*, Proc. Second National Thermal Spray Conf., 24–27 Oct 1988 (Cincinnati), ASM International, 1989
- D.L. Houck, Ed., *Thermal Spray: Advances in Coatings Technology*, Proc. First National Thermal Spray Conf., 14–17 Sept 1987 (Orlando, FL), ASM International, 1988
- F.N. Longo, Ed., *Thermal Spray Coatings: New Materials, Processes and Applications*, National Thermal Spray Conf. Proc., 31 Oct–2 Nov 1984 (Long Beach, CA), American Society for Metals, 1985

Metallography and Image Analysis

Walter Riggs, TubalCain Company Inc., Ken Couch, Protech Lab Corporation, and Kristen Larson, Hitemco

METALLOGRAPHIC EXAMINATION is a critical step in the assessment of thermal spray coating characteristics; yet, the heterogeneous or composite nature of the thermal spray deposit can make choosing a metallographic preparation process very difficult. Hard versus soft phases, brittle ceramics, and mixtures of many phases can create a variety of challenges for the metallographer.

Prior to starting a discussion of metallographic methods as applied to thermal sprayed coatings, it is important to give credit where it is due. While the authors have spent several years testing and characterizing these specific materials, two particular references are used extensively in this article. Readers are encouraged to consult these references for a more in-depth review of metallographic methods, including equipment, consumable materials, response of the material being prepared, and so on. Each has a comprehensive bibliography that serves to expand even further in particular areas. The references are:

- L.E. Samuels, *Metallographic Polishing by Mechanical Methods*, 4th ed. (Ref 1)
- G.F. Vander Voort, *Metallography: Principles and Practice* (Ref 2)

This article draws from these standard references, supplementing them with practical experience with thermal sprayed coatings.

Having previously alluded to the sensitivity of thermal sprayed materials to metallographic procedures, it is now necessary to undertake a thorough discussion of the metallographic process, the materials and equipment used as part of the process, the variables encountered (i.e., parameters that may be varied), and the responses of thermal sprayed coatings to the polishing materials, equipment, and variables. Samuels (Ref 1) correctly states in his book that "Any experimenter who wishes to obtain reliable information by the use of optical metallography must first become proficient at specimen preparation."

The aim of metallographic preparation of thermal sprayed coatings is to present the true, original, undisturbed microstructure for observation. As Samuels (Ref 1) puts it, "All the structural features characteristic of this plane must be detectable, and no false structures must be observable." Even Henry Clifton Sorby, one of the pioneers in this field, recognized as early as 1887 that materials showed sensitivity to preparation technique:

"It was, however, found that in many cases no good results could be obtained by polishing directly after using fine emery paper, because the surface was so much modified by the scratching. This exterior surface was, therefore, ground off by using fine-grained water-of-Ayr stone and water, until all trace of the scratches was removed. It was then polished on wet cloth stretched flat on a piece of wood, using in the first place the finest-grained crocus, and lastly the very best and finest-washed rouge, so as to obtain a beautiful polish, almost or altogether free from microscopic scratches, without any of such surface disturbance as is caused by a forced polish. This latter

usually looks far better, because the minute cavities and other irregularities are hidden."

A current example of the previous discussion, over 100 years later, is the following. A previously prepared metallurgical mount of a tungsten carbide/cobalt plasma sprayed coating was taken to a leading laboratory and photographed (Fig. 1). The coatings engineer remarked that the microstructure was not typical for that coatings facility, and that he thought that the mount had been improperly prepared. The laboratory supervisor agreed, whereupon the engineer asked the supervisor to reprepare the mount (without remounting), using the laboratory's best procedure. The supervisor complied and a few minutes later photographed the coating again (Fig. 2). The engineer this time remarked that the new microstructural appearance, while indeed interesting, was also not typical; the typical microstructure was much more dense than either of the two representations already seen.

The supervisor, then aware of what the engineer wanted to see, took the mount again and reprepared it using a quite different procedure. He presented the micrograph shown in Fig. 3, much to the delight of the engineer. Having demonstrated the sensitivity of the coating system to metallographic procedure variance, the engineer, now being curious, subjected tungsten carbide/cobalt coatings from another high-energy plasma spray process and from a high-velocity oxyfuel (HVOF) process to the same procedures. It

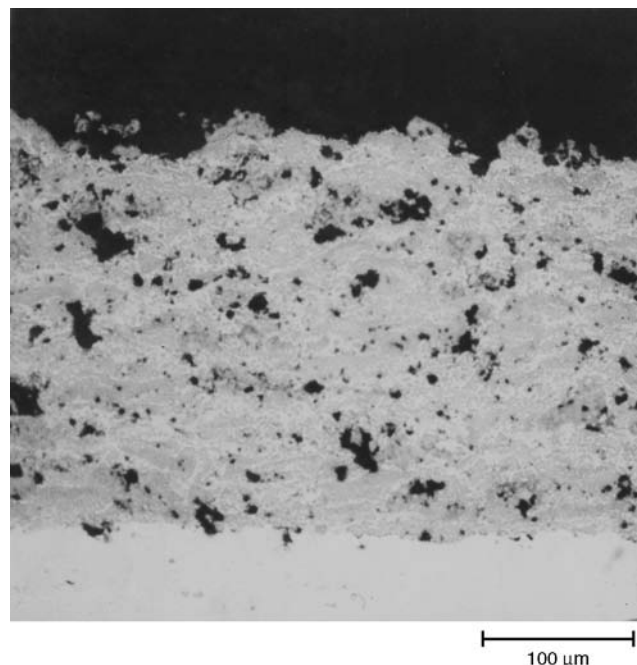


Fig. 1 Plasma sprayed tungsten carbide/cobalt coating in as-received condition. Original magnification 200x

was found that neither of these coating types exhibited the sensitivity of the first type of material.

Figures 4 and 5 show the other plasma sprayed coating (Fig. 4 corresponds to the metallographic procedure that generated the

more porous structure for the coating first shown in Fig. 2, while Fig. 5 corresponds to the procedure shown in Fig. 3). Figures 6 and 7 show the corresponding extremes for the HVOF coating system. The engineer concluded that while some coating systems exhibit

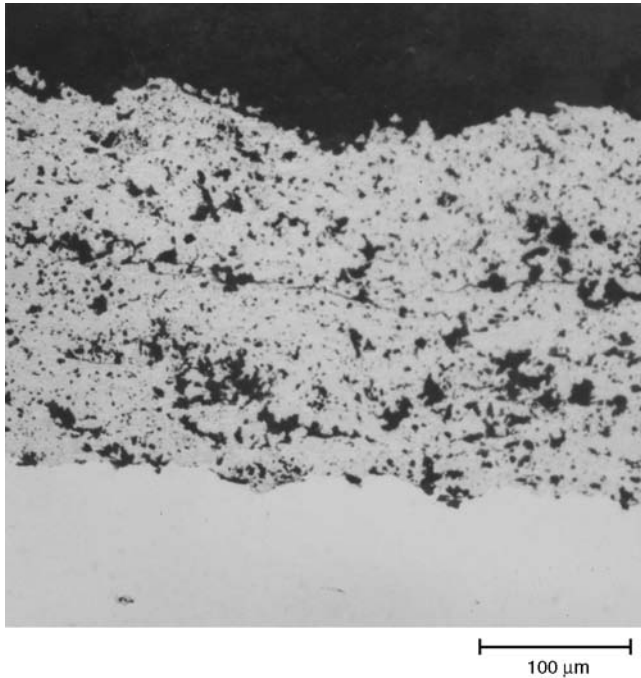


Fig. 2 Plasma sprayed tungsten carbide/cobalt coating shown in Fig. 1 after repreparing (regrinding and repolishing). Original magnification 200×

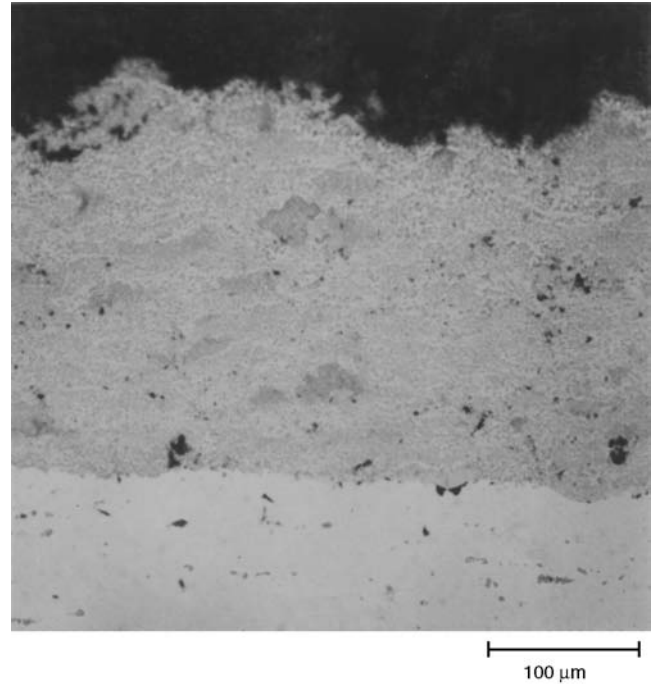


Fig. 3 Plasma sprayed tungsten carbide/cobalt coating shown in Fig. 2 after second regrind/repolish. Original magnification 200×

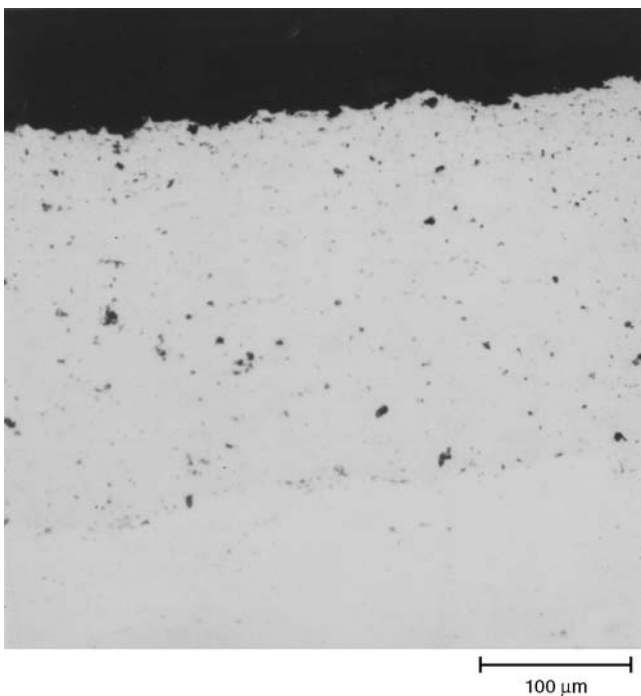


Fig. 4 High-energy plasma sprayed tungsten carbide/cobalt coating prepared similarly to the coating shown in Fig. 2. Original magnification 2. 200×

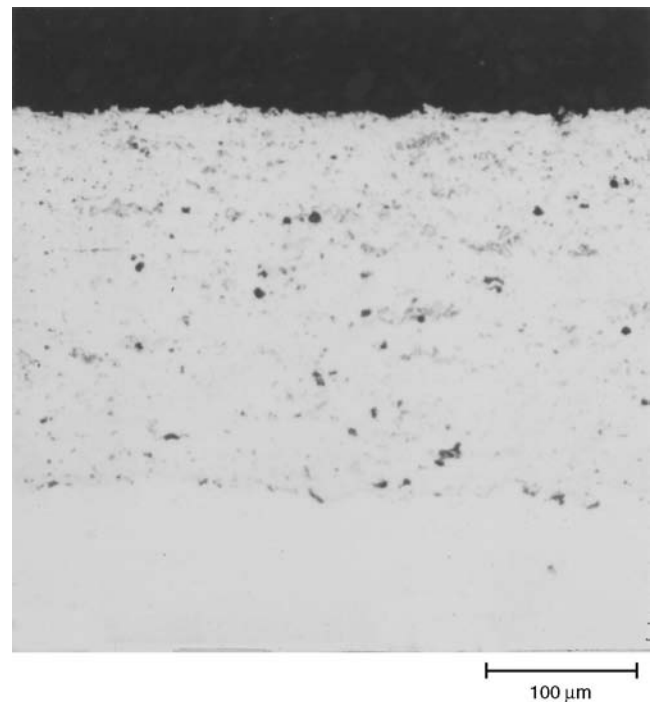


Fig. 5 High-energy plasma sprayed tungsten carbide/cobalt coating prepared similarly to the coating shown in Fig. 3. Original magnification 3. 200×

extreme sensitivity to the metallographic procedure used, others appear relatively immune; the probability, then, was either that the first coating was not dense to begin with, or that the particle splats for the first coating were very loosely bonded.

It is seen in the following discussion that many parameters are involved in the metallographic processing of coatings. As discussed in the introductory article "Introduction to Testing and Characterization" in this Section of the Handbook, thermal sprayed coatings should not be expected to respond similarly to wrought, cast, and forged materials. As the reader progresses through this article, an appreciation for the inherent sensitivities of various types of coating materials should be gained.

All the procedures discussed in this article are intended to be performed using automatic grinding and polishing equipment. Manual grinding and polishing methods should be avoided, simply because pressures and speeds are not repeatable and/or cannot be calibrated. While some metallographers have demonstrated the ability to prepare specimens that are very appealing (i.e., very dense microstructures, minimal disturbance, etc.), the fact remains that individual techniques may indeed not be consistent, and their methods are not readily transferable.

The following sections are considered important as topics within themselves: sectioning, mounting, grinding, polishing, optical microscopy, and image analysis. Following these sections, a brief discussion of etching to reveal grain structure is presented, and recommendations for some standard coatings are given.

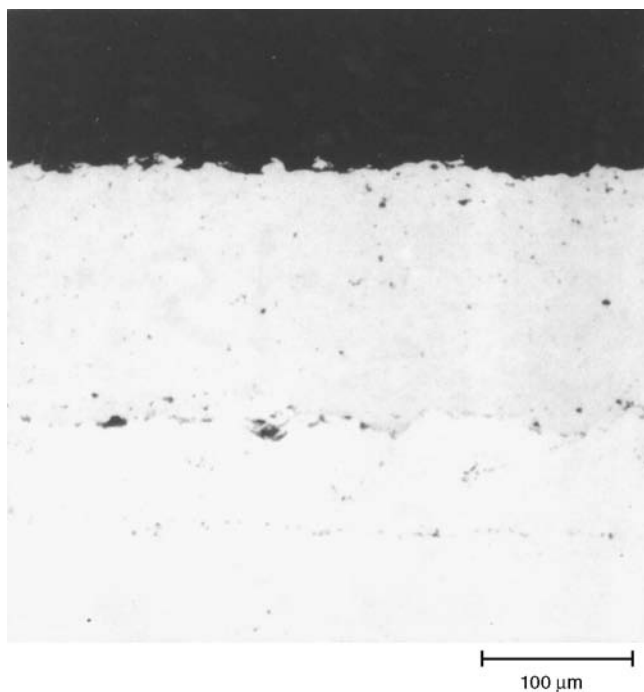


Fig. 6 A high-velocity oxyfuel sprayed tungsten carbide/cobalt coating prepared similarly to the coating shown in Fig. Original magnification 2. 200x

Sectioning Techniques

Prior to encapsulating the specimen in some form of mounting material, it is usually necessary to reduce the size of the specimen; this is accomplished by sectioning. Abrasive cutting using rotating wheels or discs is now almost universally the standard method of sectioning.

Figure 8 shows an abrasive cutoff machine. The specimen to be cut or sectioned is held firmly by some form of grip, the wheel or disc is rotated by an electric motor at either a fixed or variable speed, and either the wheel or the specimen is translated to per-

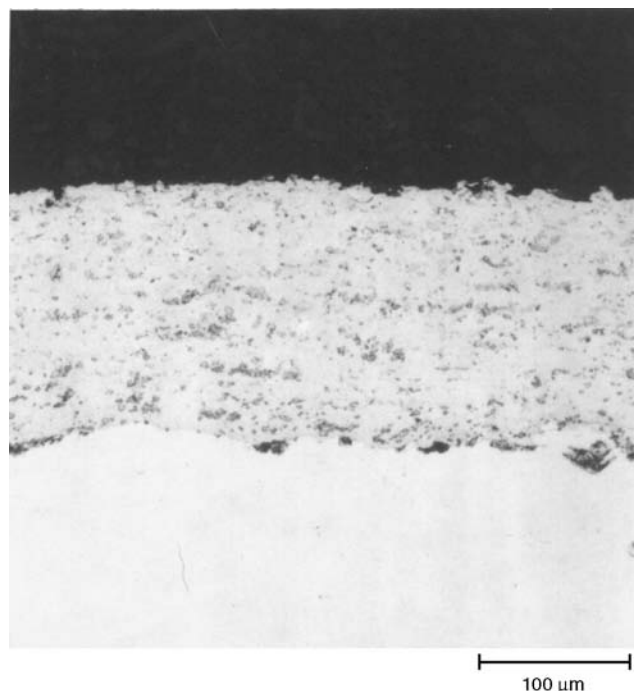


Fig. 7 A high-velocity oxyfuel sprayed tungsten carbide/cobalt coating prepared similarly to the coating shown in Fig. Original magnification 3. 200x



Fig. 8 High-speed cutoff machine. The specimen is fed into the wafering blade at a constant rate and force that are controlled electronically. Courtesy of Struers, Inc.

form the cut. Translation may be either manual or automatic. The wheels themselves, either silicon carbide or aluminum oxide, are available in a variety of sizes (up to 300 mm, or 12 in., in diameter) and thicknesses (up to 15 mm, or 0.6 in.). The abrasive wheels use either resin or rubber for bonding. Resin-bonded wheels are used for dry cutting, while rubber-bonded wheels are used for wet cutting and are therefore preferred for metallographic work.

These consumable abrasive cutoff wheels are available with different levels of bonding; that is, the amount of porosity is varied to control hardness. A soft wheel has a relatively porous rubber bond that allows the wheel to be consumed at a fast rate. A soft wheel is desirable for cutting hard materials, because breakdown of the wheel exposes fresh cutting abrasive more frequently. Hard wheels contain little porosity, break down at a slower rate, and thus are preferred for cutting soft materials. Therefore, for optimal cutting action, the wheel and its cutting rate must be matched to the hardness of the material being cut.

Sectioning Damage

With regard to the sectioning of thermal sprayed coatings, it should be remembered that these coatings consist of a heterogeneous splat network with varying degrees of metallurgical bonding that may deform via a variety of modes under an applied load. Care must be taken to avoid damaging the coatings to significant depths. Table 1 shows that the damaged or deformed layer for abrasive cutoff is 700 μm , or 0.0276 in., for a 70:30 brass alloy. The table also contains information for other processes, including abrasive grinding using silicon carbide papers and water, a commonly used technique. The deformed layer in 70:30 brass is 77 μm (0.003 in.) for 220-grit paper and still ~22 μm (0.00087 in.) for 600-grit paper. This final deformed zone must be removed by polishing to reveal the true microstructure.

One method sometimes used to minimize sectioning damage is a low-speed diamond saw, as shown in Fig. 9. The abrasives used in this sectioning process are synthetic diamonds attached to a thin metallic disc. Rotational speeds are significantly lower than those of the abrasive cutoff machines. The pressure used for the diamond saw is measured in grams of force, and wheel thicknesses are much less than typical abrasive wheels; thicknesses of 0.15 to 0.38 mm (0.006 to 0.015 in.) and diameters of 75 to 125 mm (3 to 5 in.) are common.

Sectioning of the coating must begin through the outermost layer of the coating, inward and through the substrate, rather than the opposite. Sectioning beginning at the outermost layer of the coating will produce a compressive, rather than a tensile, stress within the coating, and the compressive stress capability of coatings is generally much greater than their tensile stress capability.

In the most critical applications, to avoid damage to the coating specimen, it can first be encapsulated in a cold-mount-type epoxy prior to sectioning. Such encapsulation virtually guarantees minimal damage to the specimen but is very time-consuming and usually found to be unnecessary.

A combination of the aforementioned techniques will minimize coating damage due to sectioning: presectioning encapsulation in a protective epoxy, using a low-speed diamond saw, and cutting through the coating compressively. The damage depths as listed in Table 1 can be minimized by following these practices.

Mounting Materials and Techniques

Prepared specimens, in order to be metallographically processed, are usually encapsulated in cylindrical polymeric mounts. Mounts allow gripping either by hand or in a holder for

Table 1 Depth of the plastically deformed layer produced on annealed polycrystalline 30% Zn brass by abrasive machining processes

Process	Abrasive		Conditions(a)	Cutting fluid	Depth, μm		
	Material	Grade			Scratches	Significant deformation (D_s)(b)	Deformed layer (D_d)(b)
Cutoff wheels	Aluminum oxide	60 mesh	...	Proprietary oil-water emulsion	4	16	700
Belt surfacing	Diamond wheel	(c)	...	Proprietary fluid	1	14	...
	Aluminum oxide	100 mesh	Specimen hand held	None	15	35	250
	Silicon carbide	80 grade	Specimen hand held	Water	10	45	240
		120 grade	Specimen hand held	Water	5.5	25	190
		240 grade	Specimen hand held	Water	3.5	15	95
		400 grade	Specimen hand held	Water	1.2	5	60
Abrasion on abrasive papers	Emery	1/0 grade	Hand abrasion	Kerosene	1.8	7.5	45
		2/0 grade	Hand abrasion	Kerosene	1.0	7.0	38
		3/0 grade	Hand abrasion	Kerosene	0.4	4.0	30
		4/0 grade	Hand abrasion	Kerosene	0.3–1.0(d)	3–10(d)	20–50(d)
	Silicon carbide	220 grade	Hand abrasion	Water	2.0	7.5	77
		400 grade	Hand abrasion	Water	1.5	6.5	43
		600 grade	Hand abrasion	Water	0.8	5.0	22
Abrasion on fixed-abrasive laps	Aluminum oxide	10–20 μm grade	Hand abrasion	None	0.3	3	16

(a) Specimen pressure, approximately 20 kPa (3 ksi). (b) Maximum depth beneath the roots of the surface scratches or machining marks. (c) Low-speed metal-bonded disc. (d) The higher values result when the paper is loaded with abrasion debris. Source: Ref 1

use in automatic grinding/polishing devices. Many materials and associated processes are available for mounting; a review of the requirements of the mount material is given as follows, prior to discussing the materials themselves.

Desired Characteristics of Mounting Materials

The first requirement of mounting has already been mentioned: to allow gripping of the specimen by hand or automatic devices. This can be subdivided into two basic requirements: The mount itself must be capable of being held, and the mount itself must firmly hold the specimen. Castable plastics or polymers have been found to fulfill these requirements and are very easy to work with (i.e., they are not skill-intensive).

A second requirement of mounting materials is that they should not respond in a significantly different manner than the specimen during grinding and polishing; otherwise, in a relatively short time, the specimen will protrude beyond the mount surface. Table 2 lists abrasion rates for common metals and alloys, while Table 3 lists the abrasion rates for commonly used mounting materials. The abrasion rates for the common mount materials—phenolic, acrylic, and epoxy—are very similar and are an order of magnitude higher than those for metals; consequently, the mount will recede during abrasion and polish more quickly than the specimen. Figure 10 illustrates this effect for different mount materials, abrasive papers versus laps, and types of specimen being processed. Note that aluminum is less affected than chromium. Table 2 indicates that the abrasion rate for aluminum is ten times higher than that for chromium. The higher abrasion rate for aluminum results in less of a difference between mount and specimen. The consequence of the mount material responding differently than the specimen is shown in Fig. 10 as rounding of the specimen at its outermost edge. It would be expected from Table 3 that phenolic mounts would abrade and polish faster than polyvinyl chloride, and, as shown in Fig. 10(c), that expectation

holds true. The phenolic material has a more protruding specimen; the edge of the protruding specimen interacts with the abrading and polishing devices, causing rounding of the outermost edge. Edge rounding is very important, especially when microstructural evaluation of the outermost layers of a coating material is needed (usually at 50 to 500× magnification).

Figure 11 illustrates the effect of edge rounding for a plasma sprayed coating as viewed through a microscope. Referring again to Table 3, it would be expected that the addition of alumina, especially in sintered pellet form, would significantly decrease the rate at which the mount material abrades and polishes, resulting in less edge rounding. Figure 11 shows the cumulative effects of a mount material (softer than the coating), use of a high-napped cloth, and alumina (the softest abrasive polishing material used). Significant edge rounding occurs.

A third requirement is that the mounting material and the process used to form the mount must not physically damage the specimen or cause the specimen to be heated significantly. This requirement is very crucial for thermal sprayed coatings and is the

Table 2 Abrasion rates obtained with various metals that cause little deterioration of abrasive papers

Metal or alloy	Abrasion rate(a), μm/m			
Description	Hardness, HV	Silicon carbide, P240	Aluminum oxide, P240	Diamond 220
Aluminum:				
High purity, annealed	24	2.61	1.93	1.76
Alloy, heat treated	105	1.29	0.85	0.65
Cadmium: commercial purity, annealed	21	2.4	2.0	...
Chromium: high purity, annealed	200	0.25	0.20	0.16
Copper:				
High purity, annealed	50	0.61	0.28	0.19
Brass 30% Zn, annealed	45	0.81	0.72	0.40
Brass 40% Zn, leaded	155	2.06	1.48	0.77
Aluminum bronze	200	0.78	0.66	0.18
Gold: high purity, annealed	22	0.26	0.16	0.08
Lead: commercial purity	4.2	1.3	1.3	...
Nickel:				
Commercial purity, annealed	130	0.08	0.17	0.14
Alloy (Nimonic 100), heat treated	260	0.10	0.21	0.06
Steel: austenitic, type 304	155	0.09	0.36	...
Silver: high purity, annealed	35	1.17	0.41	0.21
Tin: high purity	9	3.5	3.45	...
Titanium:				
Commercial purity, annealed	200	0.25	0.15	0.11
Alloy (Ti-6Al-4V), heat treated	295	0.15	0.07	0.07
Zinc: commercial purity, annealed	35	1.24	1.22	...

(a) Abrasion rates obtained after approximately 500 traverses on a track of paper. Pressure applied to specimen, 38.7 kPa (395 g/cm²). Abrasion rates in $\mu\text{m}/\text{min}$ for a specimen abraded on a track 16 cm (6 in.) in diameter on a wheel rotating at 200 rpm can be obtained by multiplying these figures by 100. Source: Ref 1



Fig. 9 Low-speed cutoff machine. The specimen is fed into the diamond wafering blade by the force exerted by a deadweight. The blade rotation is fixed at 500 rpm. Courtesy of Buehler Ltd.

subject of much controversy. Many coatings engineers are concerned about the potential damage to coatings caused by the high pressures (above 27.5 kPa, or 4 ksi) used in hot-mounting processes.

The fourth requirement is that metallographic mounting materials should also have the ability to penetrate and fill nonoccluded (surface-connected) pores. The filling of pores by mounting material will preserve their original size and shape and possibly minimize pull-out-type damage during grinding and polishing. Epoxy-

type mounts, which are usually poured around the specimen, are better able to fill pores, due to their lower viscosity as compared to thermosetting plastics. Specific recommendations for performing cold mounting are discussed later.

Mount Preparation

Having discussed the main requirements of mounting materials, it remains to discuss the method of preparation of the mounts themselves.

Cleaning. Two methods of cleaning are worthy of note here; the two may be used individually or in combination. The first cleaning method is old-fashioned soap and water. Scrubbing of the sample with soap and water using cotton swabs is a simple method used to remove oils, dirt, and so on. Adequate rinsing and drying are necessary prior to mounting; use of an alcohol, such as isopropyl alcohol, after rinsing will hasten drying. Drying can also be facilitated by placing sectioned, washed, and rinsed samples on a hot plate or in an oven at ~65 to 95 °C (150 to 200 °F). Ultrasonic cleaning is a second method sometimes used to clean materials prior to mounting. This method works by an induced ultrasonic shear wave that, on rebound from the surface of the material, creates cavitation (the collapse of gaseous bubbles at the surface), a by-product of the cavitation being the cleaning of the surface. Some materials, especially soft materials, are very sensitive to ultrasonic cleaning, and caution should be exercised when using this technique.

Mounting Processes

There are two basic mounting processes: hot and cold. Hot mounting uses considerable pressure, while cold mounting uses no heat or pressure.

Typical hot-mounting presses are shown in Fig. 12. A ram inside the central cylinder is activated, bringing a platen or pedestal base to the top; the specimen is placed face down (the face being the surface to be ground, polished, and viewed) on the platen; if necessary, the specimen is held upright by some device, such as a spring clip. The ram is then deactivated (i.e., depressurized), whereupon the platen descends to the bottom of the cylindrical chamber. The mounting material, either as a powder or preform cylinder, is then placed in the cylinder over the specimen. The threaded cap is then assembled to the cylinder, the ram reactivated (raising the platen until a prescribed force is obtained), and heat applied. The thermosetting plastic is softened and maintained in this condition under pressure and with heat applied for approximately 5 to 10 min, during which time polymeric chain growth and cross linking occur. The cylinder, ram, and mount are then allowed to cool (using either fins or forced cooling), and the finished mount is ejected from the plastic cavity. Silicone mold-release agents allow easy removal of the mount from the press.

The cold-mounting process, by its name, indicates that heat is not used nor is pressure applied. The method typically employed to make cold mounts is to place the sample to be encapsulated into a plastic or rubber cup, some of which have detachable bottoms for easy removal of the mount after it has cured. The cold-mount material consists of a polymeric resin and hardener and sometimes

Table 3 Abrasion and polishing rates of common specimen-mounting plastics

Mounting plastic			Abrasion rate,	Maximum thickness removable,
Type	Filler	Abrasive	µm/m	µm
Abrasion rates of plastics that cause only minor deterioration of papers coated with conventional abrasives				
Phenolic	Cellulose(a)	Silicon carbide	11.0	...
		Alumina	11.6	...
	Mineral(b), 7 wt%	Silicon carbide	8.0	...
Acrylic	...	Alumina	8.5	...
		Silicon carbide	10.5	...
		Alumina	11.5	...
Epoxy, casting	...	Silicon carbide	20	...
Formvar	...	Alumina	20.5	...
		Silicon carbide	5.0	...
		Alumina	5.0	...
Polyvinyl	...	Alumina	3.0	...
Abrasion rates of plastics that cause severe deterioration of papers coated with conventional abrasives				
Epoxy, casting	Alumina(c), 20 wt%	Silicon carbide, P240 grade(d)	15(e) 1.5(f)	2500
Epoxy, molding	Mineral(g), 70 wt%	Silicon carbide, P240 grade(d)	3.5(e) 0.2(f)	450
Allyl	Mineral(h), 55 wt%	Silicon carbide, P240 grade(d)	11(e) 2.5(f)	2500
Mounting plastic		Polishing rate, µm/100 m(i)		
Type	Filler	Monocrystalline diamond	Polycrystalline diamond	
Polishing rates				
Phenolic	Cellulose(a)	14	19	
	Mineral(b), 7 wt%	8.8	14	
Acrylic	...	11	19	
Epoxy, casting	...	4.1	11	
	Alumina(j), 20 wt%	2.6	4.2	
Epoxy, molding	Mineral(g), 70 wt%	0.8	0.5	
Formvar	...	2.0	3.0	
Polyvinyl	...	5.0	6.0	
Allyl	Mineral(k), 55 wt%	4.5	5.2	

(a) Wood flour. (b) Probably mica and asbestos. (c) Added to the polymer-hardener mixture before polymerization commenced. (d) The values for P240-grade alumina paper are not greatly different. (e) Average abrasion rate for the first 100 specimen traverses on a track of the paper. (f) Average abrasion rate after 1000 specimen traverses on a track of the paper. (g) Probably silica. (h) Probably a mixture of silica and glass. (i) Polished on a 4 to 6 µm grade of diamond abrasive. (j) Added as 600-mesh abrasive powder to the polymer-hardener mixture before polymerization commenced. (k) Probably mica and glass. Source: Ref 1

(a) Wood flour. (b) Probably mica and asbestos. (c) Added to the polymer-hardener mixture before polymerization commenced. (d) The values for P240-grade alumina paper are not greatly different. (e) Average abrasion rate for the first 100 specimen traverses on a track of the paper. (f) Average abrasion rate after 1000 specimen traverses on a track of the paper. (g) Probably silica. (h) Probably a mixture of silica and glass. (i) Polished on a 4 to 6 µm grade of diamond abrasive. (j) Added as 600-mesh abrasive powder to the polymer-hardener mixture before polymerization commenced. (k) Probably mica and glass. Source: Ref 1

a filler material such as alumina or glass beads, which helps nucleate uniform solidification and provides a more abrasion-resistant surface, thus improving edge retention. If a filler is used, it is usually measured and mixed with a specific amount of epoxy prior to adding the hardener. After the hardener is added, the mixture is stirred with a stirring stick for several seconds, and the mixture is then poured around the specimen in the cup. Depending on the epoxy resin/hardener system used, curing occurs in a few minutes to several hours. In the authors' laboratories, resins are heated to approximately 80 °C (180 °F), the filler (glass beads) is stirred into a measured amount of resin, then the hardener is added and stirred; the resulting mixture is then poured into the plastic or rubber cups containing the specimen. The mounts are typically cured and ready to grind and polish within 30 min.

Mounting Materials

A significant benefit gained by using cold-mount materials is that the epoxy can impregnate and fill nonoccluded (surface-connected) pores. In order for maximum filling to occur, the sample must be evacuated; that is, the gaseous spaces in the pores must be emptied by placing the prepared sample (specimen in freshly poured epoxy) into an evacuation or vacuum chamber, as shown in Fig. 13. As the pressure is lowered, the mount starts to froth as the gas escapes. More elaborate evacuation chambers allow samples to be placed in a ring form prior to evacuation. At the same time, a container of the mixed epoxy, also within the chamber, is evacuated. After the frothing of the epoxy stops, the epoxy is then poured, under vacuum, onto the specimen. Such a procedure

ensures maximum penetration and filling of the pores. It must be remembered that proper cleaning and drying of the specimen prior to performing the cold-mount procedure is essential.

Other mount materials, such as acrylic thermoplastics, are also available that cure at high temperatures (150 °C, or 300 °F) under high pressures (>27.5 kPa, or 4 ksi), similar to phenolics (Bakelite). Table 4 lists different mounting materials and their respective properties.

Mounting Defects

Occasionally, defects may occur in prepared mounts. These include cracking at the corners of an encapsulated specimen; bulging at the front or the back of phenolic mounts; soft mounts in the case of cold-setting materials; porous, friable areas in phenolic mounts; and bubbles in cold-setting materials. The typical causes of each of these anomalies are described as follows.

Cracking is caused by too large a specimen for the mount size or by corners that are possibly too sharp. To overcome this problem, either reduce the specimen size or increase the mount size. Also, eliminate sharp corners where possible.

Bulging in phenolics is caused by insufficient curing time or insufficient pressure while the specimen is at the curing temperature.

Soft mounts are usually caused either by incorrect mixing of resin and hardener or by incomplete mixing when the hardener is added to the resin. In addition, the authors found several years ago that some epoxy resin/hardener combinations required significant degassing when filler materials were used. The aforementioned

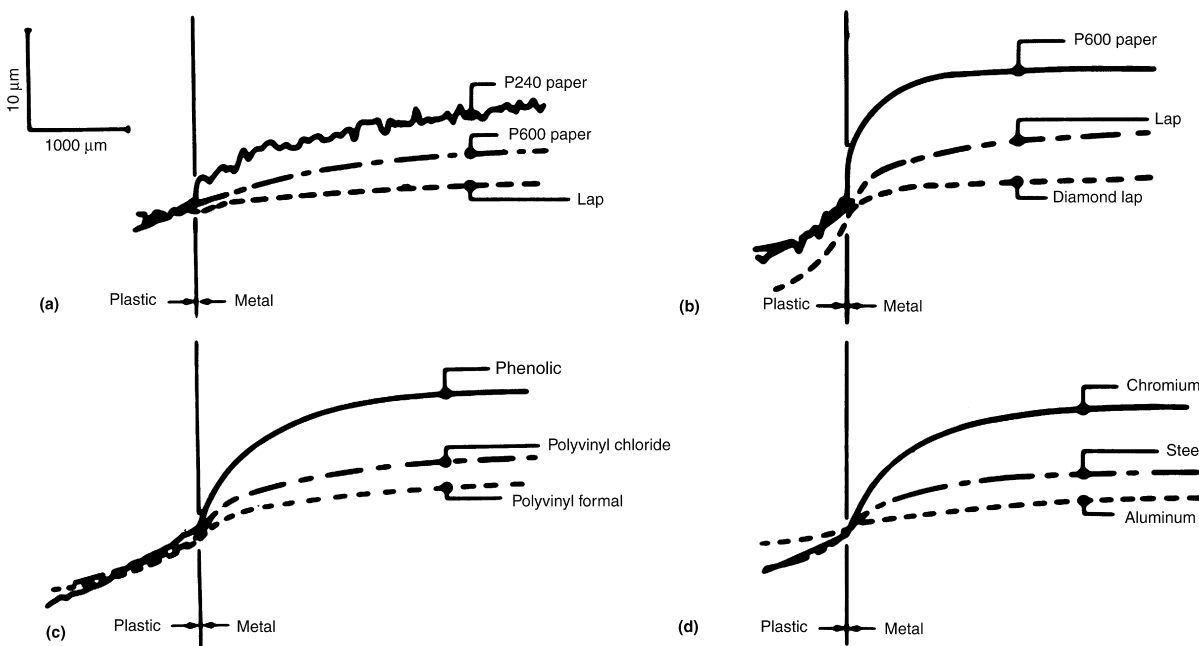


Fig. 10 Contours at the plastic/metal interface following abrasion. (a) Annealed low-carbon steel (130 HV) mounted in Formvar plastic; abraded as indicated. (b) Annealed low-carbon steel (130 HV) mounted in phenolic plastic; abraded as indicated. (c) Chromium mounted in plastics indicated; abraded on 15 μ m aluminum oxide wax laps. (d) Metals indicated mounted in phenolic plastic; abraded on 15 μ m aluminum oxide wax laps. For (a), (b) and (d), the steel specimen was chosen as being representative of metals with a polishing rate lower than that of the mounting plastic.

glass-bead-filled epoxy system with 80 °C (180 °F) heating of the resin has eliminated all soft mount problems.

Porous, friable areas (for hot mounts) can be caused by low molding pressure, short curing times, or by adding the powder to an excessively hot mold.

Bubbles (for cold mounts) can be caused either by too violent a mixing action during adding of hardener to resin or by failure to

completely outgas the specimen. Sometimes, bubbles are due to chemical contaminants on the surface of the specimen.

Occasionally, scanning electron microscopy (SEM) is used to evaluate metallographic mounts of thermal sprayed coatings. Energy-dispersive x-ray analysis (EDS) is used to determine the chemical constituents of selected areas being scanned. It is important to know the chemistry of the mounting material in order to ascertain whether the material being scanned is actually from the mount itself or from some other source. Table 5 lists the EDS analyses from several common mount materials.

In summary, it is recommended that cold-mounting materials and methods be used for thermal sprayed coatings to avoid damage to the specimen, to attach more securely (physically) to the coating, and, with suitable fillers, to minimize edge rounding. Only in the case of metallic coatings should hot-mounting practices be considered, and then only when absolutely necessary.

Grinding

The prepared metallurgical mount is next subjected to a series of grinding and polishing steps, with the aim of producing a surface suitable for observation at both low and high magnifications. This goal embodies several fundamental needs: that the microstructure revealed be true and undisturbed; that the largest abrasive or polishing artifact (i.e., scratch) be of smaller dimension than that observable at the particular magnification (e.g., a 10 μm scratch magnified 500 times would appear 5000 μm , or 5 mm [0.1969 in.], wide; a 1 μm scratch then would be 0.50013 mm [0.01969 in.] wide, which is more acceptable); and that the entire field of view be in focus. This latter requirement is related to specimen flatness and edge rounding.

Each of these needs is discussed in the next two sections. Before discussing these needs and their associated problems, however,

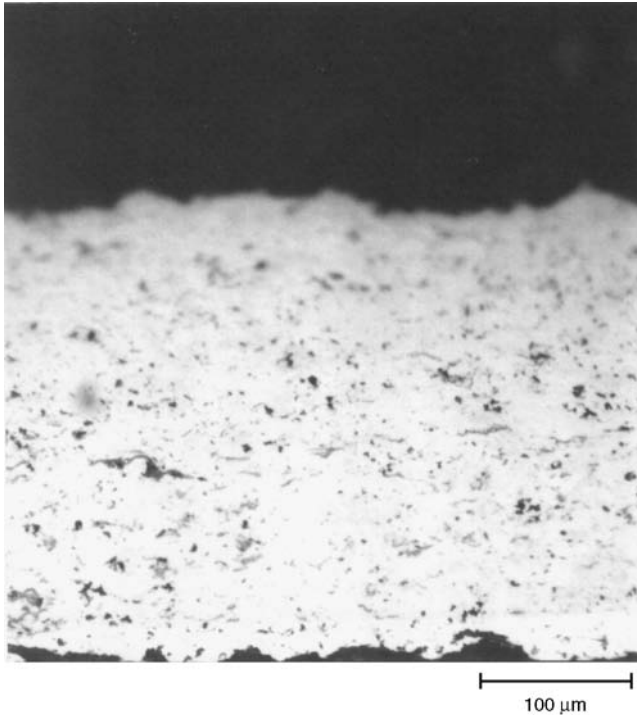


Fig. 11 Edge rounding of tungsten carbide/cobalt coating. Original magnification 200x



Fig. 12 Mounting presses. (a) Old-style mounting press. The dies are cooled by a manually applied metal heat sink. Courtesy of Meta-tech Industries. (b) Automatic mounting press. The pressure and heating and cooling times are controlled automatically. The dies are water cooled. Courtesy of Allied High Tech Products Inc.



Fig. 13 Vacuum degassing apparatus for cold-mount preparation. Courtesy of Struers, Inc.

Table 4 Typical properties of plastics suitable for metallographic mounts

Plastic	Type	Molding conditions			Heat-distortion temperature, °C (°F)(a)	Coefficient of thermal expansion, mm/mm/°C(b)	Transparency	Chemical resistance
		Temperature, °C (°F)	Pressure, MPa (psi)	Curing time				
Phenolic molding powder	Thermosetting(c)	170 (340)	28 (4000)	5 min	140 (285)	$3.0\text{--}4.5 \times 10^{-5}$	Opaque	Not resistant to strong acids or alkalis
Acrylic (polymethyl methacrylate) molding powder	Thermoplastic	150 (300)	28 (4000)	nil	65 (150)	$5\text{--}9 \times 10^{-5}$	Water white	Not resistant to strong acids
Epoxy casting resin	Thermosetting(d)	20–40 (70–105)	...	24 h	60 (140)(e)	$4\text{--}7 \times 10^{-5}$	Clear but light brown in color	Fair resistance to most acids and alkalis. Poor resistance to conc. nitric and glacial acetic acids
Allyl molding compound	Thermosetting(f)	160 (320)	17 (2500)	6 min	150 (300)	$3\text{--}5 \times 10^{-5}$	Opaque	Not resistant to strong acids and alkalis
Formvar (polyvinyl formal) molding compound	Thermoplastic	220 (430)	28 (4000)	nil	75 (170)	$6\text{--}8 \times 10^{-5}$	Clear but light brown in color	Not resistant to strong acids
Polyvinyl chloride molding compound	Thermoplastic(g)	160 (320)(h)	21 (3000)	nil	60 (140)	$5\text{--}18 \times 10^{-5}$	Opaque	Highly resistant to most acids and alkalis

(a) As determined by the method described in ASTM D 648, at a fiber stress of 1.8 MPa (264 psi). (b) The coefficient of thermal expansion in most metals is in the range $1\text{--}3 \times 10^{-5}$ mm/mm/°C. (c) Wood-filled grade, preferably with low filler content. (d) A liquid epoxy resin with an aliphatic amine hardener. (e) Depends on the curing schedule; can be as high as 110 °C (230 °F) with heat curing. (f) A diallyl phthalate polymer with a mineral filler. (g) A stabilized rigid polyvinyl chloride. For example, a mixture of 100 parts of a paste-making grade of polyvinyl chloride, 2 parts dibasic lead phosphate, and 2 parts tribasic lead sulfate. (h) Must not exceed 200 °C (390 °F). Source: Ref 1

Table 5 Energy-dispersive analysis of common mounting materials

Mounting material	EDS value(a)							
	Al	Si	Ca	Cl	Ti	Cu	Fe	Others
Thermosetting resins								
Bakelite(b)	...	200	375	150	Mn: 75
Bakelite(b)
Bakelite(b)	320	450	975	110	...	Pb: 610, Cr: 225
Diallyl phthalate								
Fiber-filled	710	1450	900	250	300
Mineral-filled	200	425	225	125	...	125	...	Mg: 200, Zn: 75
Thermoplastic resins								
Methyl methacrylate(c)
Thermosetting epoxies								
Plastimet	750	4000	300	...	150
Epomet	...	1600	175	350
Struers No. 5	225	475	850	50	...
Acrylic								
Kold Mount(c)
Epoxy								
Epoxide(c)	...	90	...	220	...	70	80	S: 225
Conductive mounts								
Cu-diallyl phthalate	1975	100	...
Al-phenolic	8000
Fe-Struers No. 1	...	200	2250	...
C-phenolic	90	360	375	80	60	40	60	K: 110, S: 70

Note: Energy-dispersive analysis does not detect elements lighter than sodium. (a) Values are counts in 30 s. (b) Bakelite is highly variable in composition, which is probably due to impurities in the wood flour. (c) These materials charge badly under the beam and must be coated with carbon (vacuum deposition). Source: Ref 2

the materials and basic concepts of grinding (this section) and polishing (following section) are discussed. The organization of each section is according to materials and concepts, followed by aims, needs, and related problems.

Grinding Materials

Several different types of abrasive are used in the grinding process: grinding stones, which are sometimes used for the initial coarse grinding to remove any sectioning damage; abrasive papers, made of either silicon carbide or aluminum oxide particles bonded to a paper backing; fixed-diamond discs, where the diamonds are held in place by some form of coating or plating mechanism; and laps, which initially contain no abrasive, only the proprietary polymeric or composite materials onto which the abrasive (usually synthetic diamond) is externally charged, typically by spraying. These four abrasive grinding devices are discussed as follows.

Grinding Stones. Coarse grinding stones or wheels are sometimes used to remove a significant amount of material during the initial grinding step to eliminate damage caused by sectioning. The stones or wheels consist of coarse (80- or 120-grit) abrasives sintered together with a binder to form a vitreous bond. They must be periodically leveled with a planing device to restore flatness. As much as 3.2 mm (0.125 in.) of mount material can be removed relatively quickly using these stones or wheels. Care must be exercised to minimize the damage caused during use of these wheels or stones, however. According to Table 1, damage depths resulting from use of these wheels can be 170 μm or greater.

Abrasive Papers. Abrasive metallographic grinding papers generally consist of particles of silicon carbide or aluminum oxide cemented to a paper backing and coated (other abrasives are available but are not in common use). Several grades of papers are available, ranging from coarse (60 grit) through fine (P1200), as shown in Table 6. Two systems for defining abrasive paper types are used: a premetric system and a metric system. The standard now accepted is the metric system. The median diameter refers to the sieve screen opening through which roughly one-half of the abrasive passes. This indicates that there is a distribution of sizes dispersed on each paper. Thus, a P1200-grade paper would be expected to contain abrasive particle sizes ranging above and below the median 15.3 μm. The same is also true for polishing abrasives.

Figures 14 to 16 show 240-, 320-, and 600-grit (premetric designations) silicon carbide abrasive papers from a commercial supplier, as viewed by SEM; Fig. 17 shows a 240-grit paper from a second supplier. Note that the appearance of the two 240-grit papers is remarkably different, hence it would be reasonable to expect that the two would not produce identical results when used for grinding metallographic samples. Figure 18 shows a cross section through a 240-grit silicon carbide paper. Note the very irregular shapes of the silicon carbide particles. Beneath the particles is the paper, and surrounding and covering each particle is a coating.

The shape of the abrasive particles and their angle of orientation are very important. The goal in abrasive grinding is to remove material by machining or cutting it away from the underlying material. Figure 19 shows the grooves formed when an abrasive

particle acts against the surface of a material being ground: either plowing or cutting occurs, depending on, among other factors, the abrading particle form factor (depth-to-width ratio, with long, thin particles behaving much differently than short, large ones) and rake angle (the angle between the particle and the surface being abraded). A good statistical estimate of particle size, shape, and

Table 6 Approximate median sizes of various grades of silicon carbide and aluminum oxide abrasives

Grit number		
Metricated	Premetrication	Median diameter, μm
Screen grit size range		
60	60	250
80	80	180
100	100	150
120	120	106
150	150	90
180	180	75
220	220	63
Microgrit range		
P240	240	58.5
P280	...	52.2
P320	280	46.2
P360	320	40.5
P400	...	35
P500	360	30.2
P600	400	25.75
P800	...	21.8
P1000	500	18.3
P1200	600	15.3
P2400	...	8

Source: Ref 1

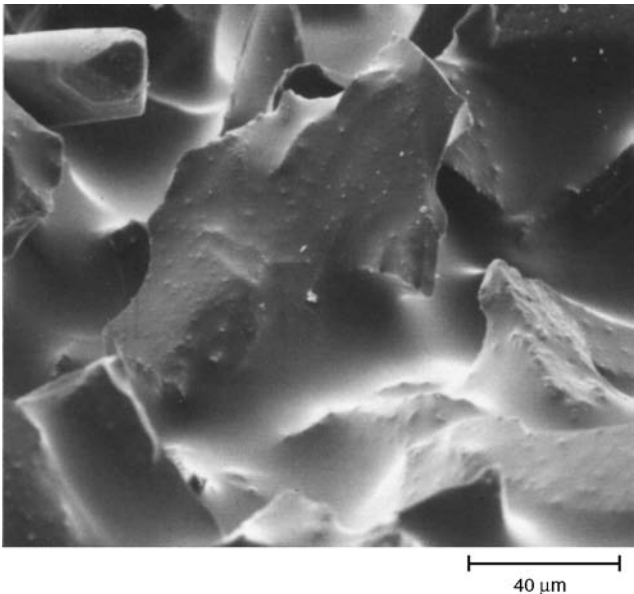


Fig. 14 SEM of 240-grit silicon carbide paper. Original magnification 500x

angle of orientation can be made from cross sections of metallographic papers (Fig. 18).

Studies conducted on wrought materials have shown that every material has its own unique critical angle at which plowing transitions to cutting. This transition depends on the hardness of the material being abraded, its ductility (as determined by crystal structure, defect structure, etc.), and other factors. The particles on the surface of an abrasive paper, as indicated by Fig. 14 to 17, vary

in height, so not all of the abrasive particles contact the surface being abraded at any given time. Of the fraction of contacting points (which can be as little as 0.5% of the total number of abrasive particles on the paper), as little as one-eighth (12.5%) actually

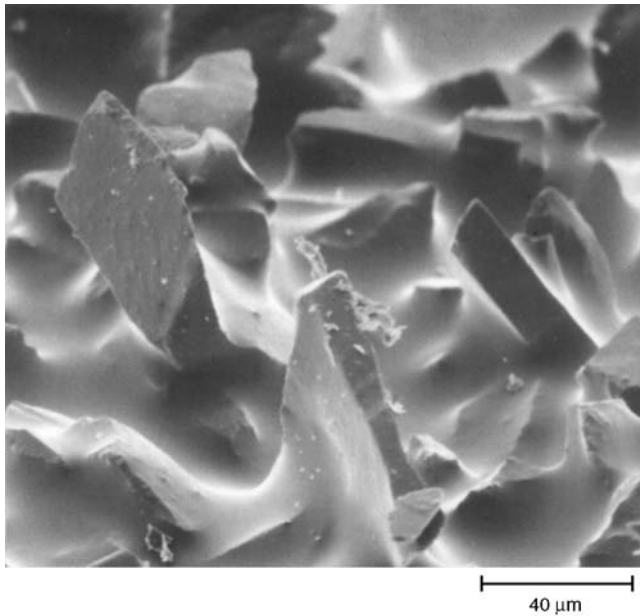


Fig. 15 SEM of 320-grit silicon carbide paper. Original magnification 500×

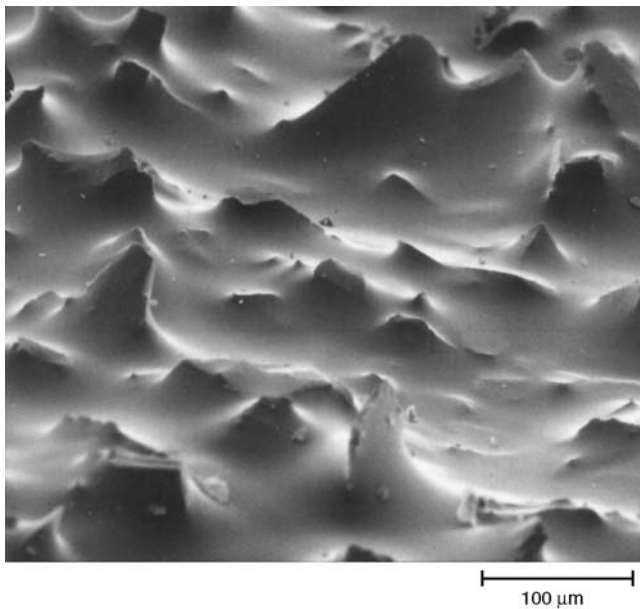


Fig. 17 SEM of 240-grit silicon carbide paper from second manufacturer. 200×

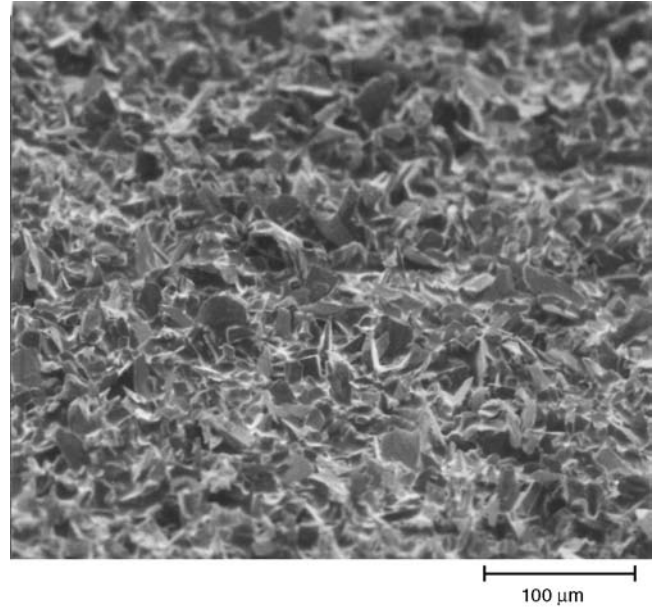


Fig. 16 SEM of 600-grit silicon carbide paper. Original magnification 200×

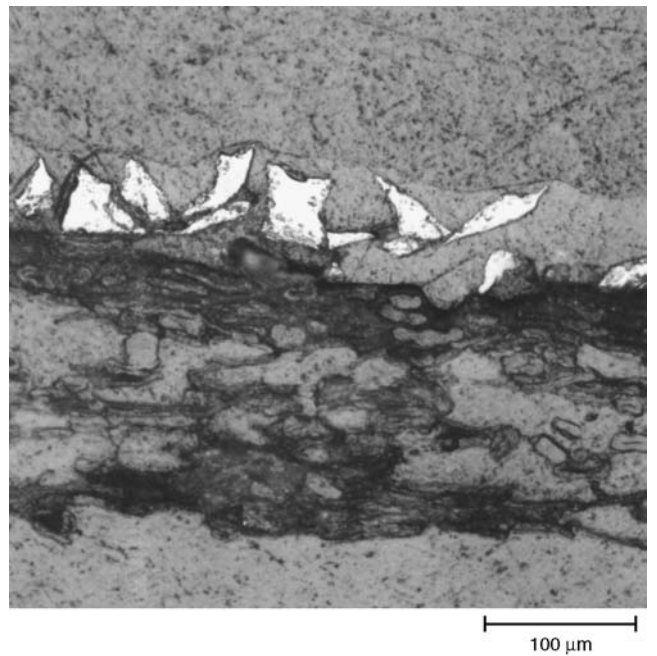


Fig. 18 Cross-sectional view of 240-grit silicon carbide paper. Original magnification 200×

perform cutting. Thus, for example, for a 600-grade silicon carbide paper with an approximate density of abrasive particles of 200,000/cm² (31,000/in.²), only 1000 actually contact the surface at any given time, and only 125 of those actually perform cutting (i.e., have the proper form factor and rake angle to perform cutting); after 400 traverses, the number of contacting points that cut is minimal. This implies a degradation of the abrasive paper; indeed, with use, the silicon carbide (or aluminum oxide) particles fracture, are torn from the paper, or are worn significantly to the point that they become ineffective at cutting.

Another requirement of abrasive particles is that they be at least 2.5 times as hard as the material they are being used to cut, if cutting is to occur. Silicon carbide particles have a Vickers microhardness (HV) of approximately 2500, aluminum oxide approximately 2000 HV, and synthetic diamond, also used as an abrasive, has a microhardness in excess of 8000 HV. This becomes important for thermal sprayed coating materials such as tungsten carbide/cobalt, where the tungsten carbide particles, with hardnesses in the range of HV₃₀₀ 2000, are embedded in a softer cobalt matrix, with a hardness of approximately HV₃₀₀ 600. The softer cobalt matrix abrades much more quickly than the tungsten carbide particles. When abrading tungsten carbide using either aluminum oxide or silicon carbide, the probable mechanism is not the clean cutting of the carbide particles but rather the abrading away of their surrounding matrix, resulting in them ultimately becoming dislodged and torn away. (As is shown later, the choice of abrasive becomes very important for the final polishing of tungsten car-

bide/cobalt coatings, in order to determine carbide volume fractions.)

One of the by-products of using abrasive papers is a somewhat uneven or undulating surface. This type of surface is produced because of the resilience of the paper backing—the paper “gives” under applied loads, as an abrading particle encounters the mount surface and begins to cut or plow. Also, because the silicon carbide or aluminum oxide particles may encounter phases in the coating that are harder than can be cut, some deflection of the abrading particles and backing naturally occur.

Fixed-Diamond Discs. Another type of abrasive that is not consumable (compared with papers) is the fixed-diamond disc, in which diamond abrasive particles are permanently embedded (and therefore not movable) with some form of coating into an underlying substrate. Figure 20 shows such a disc. This photo shows both particles that are embedded and holes where other particles used to be—clearly, the disc shown has been used! In the upper right-hand corner of this figure, an abrasive particle is covered with what appears to be debris from the grinding process. One problem with fixed-diamond discs is that they can load with abraded debris fairly quickly; the loaded disc then effectively becomes a rotating press that can significantly disturb the material being processed. Damage due to a loaded disc, as revealed by taper mount and etch studies, can be very severe (Ref 1).

Laps. The final abrasive type is relatively new within metallography, especially as used to prepare thermal sprayed coatings for polishing. Laps are somewhat proprietary; some manufacturers are still developing lap materials, and some of those already developed continue to undergo review and revision as they are used in varieties of applications. All laps, however, use the same

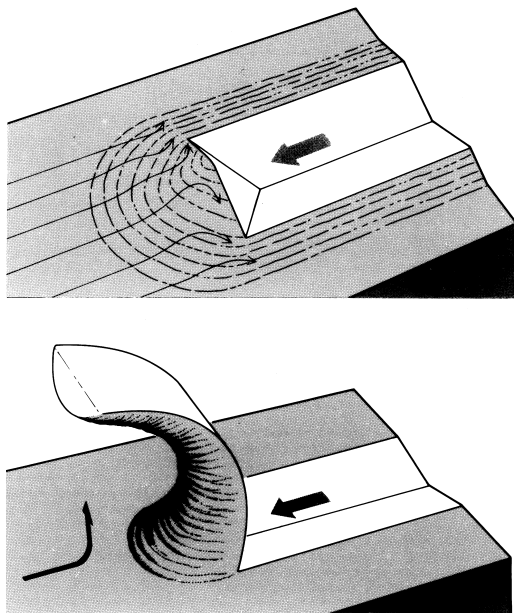


Fig. 19 Comparison of the plowing and cutting modes of a V-point tool. In plowing (top), material in the surface layers of the specimen first moves upward ahead of the rake face and then moves around it into side ridges. In cutting (bottom), a ribbon of material is separated from the specimen and moves upward past the rake face of the tool until it breaks off. (In normal machining with orthogonal tools, the workpiece material does not have the option of flowing around the tool; a chip virtually has to separate.)

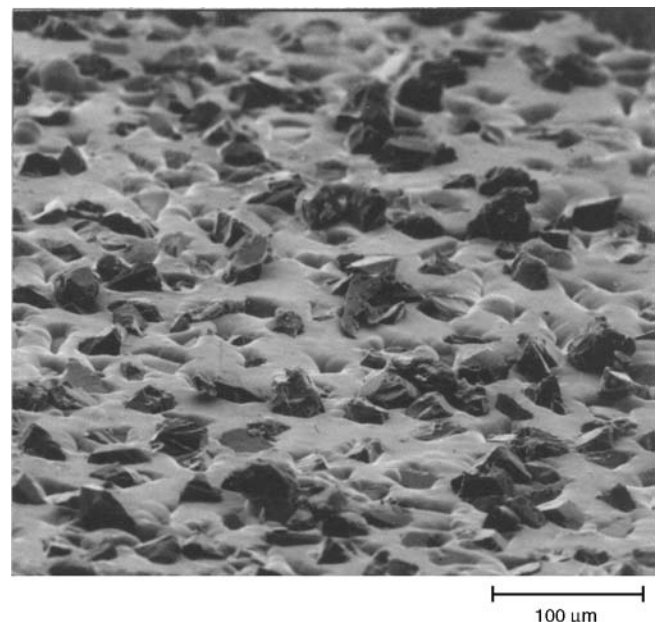


Fig. 20 Fixed-diamond abrasive disc. Original magnification 200×

basic mechanism: An externally charged abrasive, usually diamond, is deposited (usually by aerosol spraying) onto the lap surface; the abrasion mechanism is due to a combination of rolling abrasives that puncture the surface and fixed abrasives that plow the surface, as shown in Fig. 21. Rolling abrasion is relatively ineffective as a material removal mechanism; it is therefore desirable that fixed abrasion predominate. One of the key advantages of laps over abrasive papers is the flatness of the sample produced. Lap materials are much harder than papers and are not as resilient; that is, they do not give under applied loads. Rather, the abrading particles, acting in fixed abrasion, are allowed to transition to rolling abrasion if the force becomes too high, rolling along the lap surface until they again embed in the lap material. Because the abrasive used with laps is usually diamond, most, if not all, of the phases encountered in the coating can be cleanly cut (the diamond hardness of HV₃₀₀ 8000+ is more than adequate to cut the coating material). These two attributes of laps result in significantly flatter surfaces. Because lap technology is very closely guarded by manufacturers, micrographs are not shared in this article; the reader is advised to try those available and compare results to the use of abrasive papers and fixed-diamond discs in their own laboratory.

(Note: Some fixed-diamond discs load with abrasion debris quickly and easily; thereafter, they act effectively as rotating presses, distorting the underlying material. For thermal sprayed coatings, those materials that may not be fully dense can be made to appear fully dense, giving a false impression of a better polish. The purpose of this note is simply to bring to the attention of the reader the fact that some methods, such as fixed-diamond discs, may give false impressions of coating microstructure.)

Grinding Rates

A mechanistic model is discussed briefly for the purpose of acquainting the reader with the fact that a mathematical description of the process has been undertaken; those not inclined toward models can skip this portion of the article. Mulhearn and Samuels (Ref 3) have proposed that the mass of material removed during a particular traverse can be shown to be:

$$m_n = f_n \rho D L / (P \phi_m)$$

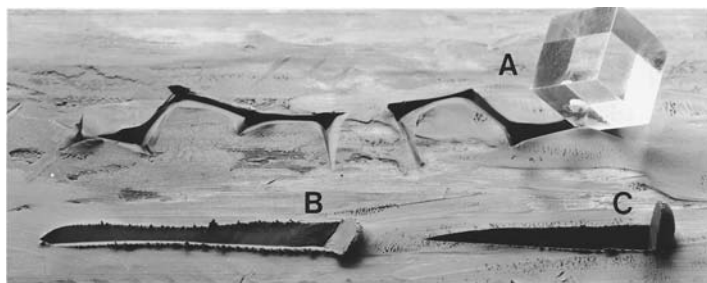


Fig. 21 Model illustrating possible modes of interaction between an abrasive particle and the surface of a specimen. A, a loose, tumbling particle; B, a fixed particle plowing a groove; and C, a fixed particle cutting a chip

where m_n is the mass removed during the n th traverse (grams), f_n is the fraction of cutting point during the n th traverse, ρ (rho) is the density of the specimen (g/cm^3), D is the distance traversed (cm), L is the load applied to the specimen, P is the indentation hardness of the specimen material (load/cm^2), and ϕ_m (phi) is the mean form factor for contacting points.

Concerning conservation of units, it can be shown that the aforementioned equation can be written as:

$$\text{grams, } g = [(\text{g/cm}^3) (\text{cm}) (\text{load})] / (\text{load/cm}^2)$$

which reduces to:

$$g = (\text{g/cm}^3)(\text{cm}^3)(\text{load/load})$$

which simplifies to:

$$g = g$$

The fraction of cutting points can be shown to be a function of a deterioration constant, β , so that:

$$f_n = f_0 e^{-\beta n}$$

where β is the deterioration constant of the particular device.

On integrating, the total mass removed up to the n th traverse is given by:

$$M_n = \rho D L / (P \phi_m \beta) f_0 (1 - e^{-\beta n})$$

Of interest here is the fact that the density of the particular material, ρ , occurs in the numerator, which indicates that denser materials will lose mass or material more quickly than less dense ones. Considering the metallography test as a form of wear test, it would seem logical that denser coatings (e.g., tungsten carbide/cobalt) would wear more slowly than less dense ones. This may be accounted for by the fact that denser coating materials often have higher indentation hardnesses than their less dense counterparts; because the term for microindentation hardness (P) occurs in the denominator in the previously mentioned equation, an increase would result in an overall decrease in mass removed.

Note that the form factor, ϕ , also occurs in the denominator, which indicates that particles with a large form factor (long, slender particles) will remove less material than those with a small form factor (short, wide particles). As form factor increases, material removal rate decreases. Load applied, being in the numerator, is obvious, with higher loads resulting in more material being removed. This is true up to the limiting point of the ability of an abrasive device to rid itself of grinding debris.

Finally, because the deterioration constant, β , is in the denominator, it is implied that a higher deterioration constant results in lower mass removal rates; thus, higher values of β are less desirable and indicate more rapid breakdown of the abrasive device. For a more complete discussion, readers should refer to Ref 1.

The speed of the abrasive particles with respect to the material being ground is of considerable importance. Studies in mechanics of materials and dynamics indicate that the speed of a moving object relates to its momentum, which relates to its force on impact

with another object. The relative speed of the abrading particles with metallurgical mounts is a complex function of the abrasive wheel and sample holder speeds (usually, multiple mounts are held in some form of sample holder, which itself rotates). The speed and also the direction of abrading is thus constantly changing. It has been found that some thermal sprayed coatings are significantly sensitive to the speed of grinding. Specifically, chrome carbide/nickel chromium coatings often exhibit near-interface separation (that is, near the interface between the coating and the substrate). An example of separation at the near-interface region in a chrome carbide/nickel chromium coating can be found later in this article (Fig. 47). It will most often be found that regrinding and repolishing with slower speeds will result in disappearance of the separation artifact. This suggests that the chrome carbide/nickel chromium coating system is inelastic, that is, that the momentum of the abrasive particles when grinding at higher speeds is sufficient to cause separation in the near-interfacial region.

Grinding Deformation

Damage due to grinding was briefly touched on earlier and is discussed in much greater detail here. Under the application of external loads, metals deform elastically (that is, they regain their original size, shape, and position when the load is removed) or, if the load is sufficient, the material will be stressed beyond its capability to respond elastically, and plastic deformation will occur. The point at which plastic deformation begins is known as the material yield point. Beyond the yield point, the applied stress is sufficient to begin to produce a sequence of deformation structures. The first deformation process to occur is the movement of internal defects called dislocations within the material; the dislocations glide along preferred crystallographic planes and directions. This glide of dislocations and subsequent pileup at boundaries (such as grain boundaries) is referred to as slip. Slip strain lines are observable in some materials that have been stressed slightly beyond their yield points, provided suitable etchants are used on well-prepared metallurgical mounts to reveal them. At stresses and corresponding strains beyond this, a second metallurgical phenomenon, known as twinning, occurs in some materials. A twin can be thought of as a mirror plane of atoms, with atoms on either side reflecting the atomic arrangement on the opposite side. Twins can be detected in some metals, such as titanium and brass, again, provided that adequate preparation and etchants are used. Further deformation causes the formation of distinct shear bands within the metal; for some materials, these slab-shaped cells can have lateral dimensions of 1 to 20 μm and thicknesses of 0.02 to 0.1 μm .

Figure 22 shows each of these structures in a 70:30 brass and discusses the strains applied to the specimen, as well as the etchants used. Because metallographic grinding and polishing exerts considerable pressure on the mount and performs material removal, itself a plastic deformation of the material beyond shear, at the surface of the specimen, it is to be expected that immediately beneath the surface that has been abraded or polished will be material that has been subjected to significant stress and corresponding strains (just short of the stress required to completely remove it from the underlying material). Stated differently, as the abrading

device applies a load to the material being prepared, sufficient stress is generated to remove some of the material—other material immediately beneath the removed material also undergoes significant applied stress, sufficient to generate the deformation structures referred to previously (slip, twinning, and shear bands).

The aforementioned deformation structures, which would be expected to be present in reverse order beneath any metallographically prepared surface, indeed have been revealed by taper-mount studies conducted on previously prepared metallographic specimens. The highest stress and strain occur at the surface, which would cause shear band formation; the next level of stress beneath it would create twinning, and finally, the lower stress experienced would cause slip. The region of slip is adjacent to material that responded elastically deeper within the volume of material, away from the ground surface. See Ref 1 for an extensive discussion of defect structures beneath metallographic surfaces.

In addition to the elastic and plastic responses discussed previously, material can also be plastically translated at the material surface. Some refer to this phenomenon as smearing. The evidence for such plastic translation at the surface of the specimen is seen in Fig. 23 (Ref 1). This figure shows ribbons of silver in a eutectic bismuth-silver alloy standing in relief after an etch to remove the bismuth matrix; note that in Fig. 23(a), the tops of the silver are plastically deformed in the direction of grinding, evidence of plastic material translation at the surface of the mount. Note also in Fig. 23(b) that the plastic deformation caused by grinding with 600-grit papers has been removed by subsequent polishing.

Plastic material translation at the mount surface is expected when very high loads (105 kPa, or 15 psi, per mount) are used for grinding and polishing. Whereas wrought, cast, and forged materials have grain sizes that are sufficiently large to reveal slip, twin, and shear band formations using conventional optical microscopy, thermal sprayed coatings have much finer grain sizes, and such deformation artifacts are not expected to be revealed, short of the use of electron microscopy (see, for example, Fig. 4 in the article “Introduction to Testing and Characterization” in this Section of the Handbook).

At the beginning of this section, the stated needs included: that the microstructure revealed be true and undisturbed, that the largest abrasive or polishing artifact be of smaller dimension than that observable at a particular magnification, and that the entire field of view be in focus. In addition, due to economic considerations, the material removal rate should be relatively high.

Table 7 compares the material removal rates, specimen flatness, scratch depths, and forces used for conventional abrasive papers versus lapping discs. These findings have been confirmed in numerous laboratories.

Material Response to Various Parameters

The final area to be discussed in grinding is the response of materials to the various parameters that can be changed as part of the abrasive grinding process. These parameters include pressure, abrasive type, abrasion fluid, and time.

Pressure. While a few grinding and polishing machines are capable of imparting loads up to 105 kPa (15 psi) per mount for up

to six mounts, most machines do not have this capability. This alone renders it obvious that metallographic procedures that require such high pressures are atypical within the metallographic community. Reference 1 refers to a typical load as being 40 kPa (5.5 psi) per mount. The 35 kPa (5 psi) per mount range is adequate to polish most metallographic mount types for most thermal sprayed coatings of interest. Higher pressures are usually only required for those materials that are very hard and resist grinding and polishing. Ceramic materials, which fracture easily during abrasion processes, would be expected to be more significantly damaged at higher grinding pressures.

Figure 24 shows the proposed development of cracks beneath the surface of a brittle material under the force of an indenter. Figure 25 shows the ground surface of a ceramic material as revealed by SEM.

Abrasive Type. Fixed-diamond discs, as discussed earlier, can load quickly, may cause significant deformation of the material being polished, and need to be dressed and cleaned regularly. Within a matter of seconds, they can cause a somewhat porous microstructure to appear fully dense. Abrasive papers, most notably, silicon carbide, are commonly used for plasma sprayed coatings.

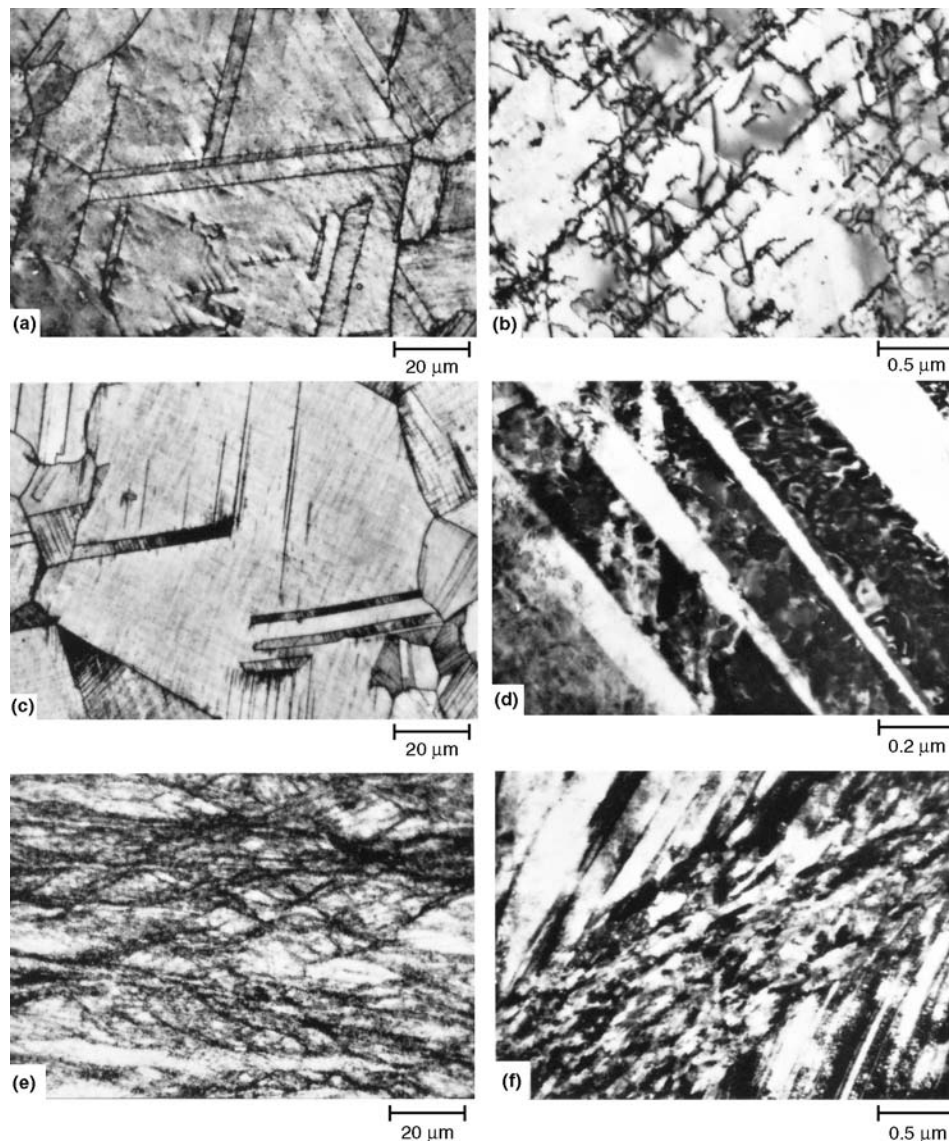


Fig. 22 Manifestations of plastic deformation observed in compressed 70:30 brass by optical and transmission electron microscopy (TEM). (a) Slip strain markings, revealed by the high-sensitivity sodium thiosulfate etch, in a specimen compressed just beyond yield. Optical micrograph. 500 \times . (b) Specimen similar to (a), exhibiting dislocation arrays on {111} planes. TEM. 20,000 \times . (c) Twin strain markings, among slip strain markings, revealed by the high-sensitivity sodium thiosulfate etch in a specimen compressed to 10% reduction. Optical micrograph. 500 \times . (d) Specimen similar to (c); electron diffraction reveals that the diagonal bands have a twin orientation with respect to the parent grain. TEM. 50,000 \times . (e) Shear-band strain markings revealed by an ammonium hydroxide/hydrogen peroxide etch in a specimen compressed to 80% reduction. Optical micrograph. 500 \times . (f) Specimen similar to (e); the diagonal band that contains elongated subgrains is a shear band. TEM. 20,000 \times . In all cases, the compression direction is vertical.

Abrasion Fluid. Most types of abrasive machining devices are flooded with a fluid during use, and this fluid behaves as a lubricant, as a coolant, and/or as a medium to flush abrasion debris away from the abrasion track. Water performs these functions well when applied in adequate doses (dry abrasive papers can severely damage underlying material, increasing total depths of deformation, as shown in Table 1).

Time. The requirement of each grinding or polishing step is to remove the volume of material that was damaged by the previous step. Suggested polishing procedures for thermal sprayed coatings with total times of as short as 20 s for each step are known. It is doubtful that damaged layers from preceding steps can be removed in such short times. Table 8 shows the time required to remove the deformed material from each preceding stage for an annealed 70:30 brass; the total time for all steps can be very long. The appropriate times for each stage of a grinding and polishing procedure need to be determined from the deformation structures for the given material, as ascertained by taper-mount and etch techniques discussed earlier. It is probable that such detailed investigations have not been completed anywhere for thermal sprayed coatings. Because deformation structures due to grinding and polishing are not yet fully understood for these coating types, it must be concluded that the grinding steps should be continued at least twice as long as it takes to remove scratches from the previous step. This will ensure that both the scratches and some volume of deformed material are removed.

For abrasive papers, grinding should be on the order of 2 min per size of abrasive (with a changing of the paper after 1 min); a series of six papers (i.e., 60, 120, 180, 240, 320, 400, and 600 grit) would thus take approximately 14 min.

From Table 6, it can be seen that the finest grade of abrasive paper recorded, 600 grit (premetrification), has a mean abrading particle diameter of approximately 15 μm . It would be expected that finer grades of paper will have mean diameters $<10 \mu\text{m}$, which is shown to be important with regard to interaction with carbides in some tungsten carbide/cobalt coatings (the finer grit sizes

approach the size of the individual carbide particles, causing dislodging or pull-out of the carbides and a higher apparent porosity). Generally, grinding papers finer than 600 grit are employed only for soft coatings, and then in order to reduce the scratches that remain after polishing (i.e., aluminum coatings appear less scratched after polishing if finer-grit papers are used prior to polishing).

In summary, it is recommended that abrasive papers be used for grinding. Pressures should be on the order of 35 kPa (5 psi) per 32 mm (1 $\frac{1}{4}$ in.) mount to avoid significant plastic material translation at the mount surface, which occurs at higher pressures. Water should be used as a lubricant, in sufficient quantities to flush the abrasion debris from the abrasion track. Several sizes of grit should be used, for a total of 2 min with a maximum of 60 s per paper, to ensure that previously damaged material is removed in each grinding step.

Polishing

The first and most important objective of polishing is to remove the abrasion-damage layer that was produced by the preceding grinding step. This damage layer includes both the plastically deformed material, seen as scratches, and the slip/twin/shear damage layer immediately beneath the surface. It has often been observed that, whereas a metallic specimen does not show evidence of scratches immediately after polishing and before etching, a significant network of scratches can appear after etching. This phenomenon is related to plastic deformation of the substrate and the generation of distinct shear and twin bands. Metallographic polishing must therefore remove not only the scratches from the previous step but also any substrate plastic deformation.

Of considerable importance in the development of an appropriate polishing procedure is a thorough understanding of the nature of the material being investigated, including knowledge of the size, shape, hardness, and ductility of the features of interest, and

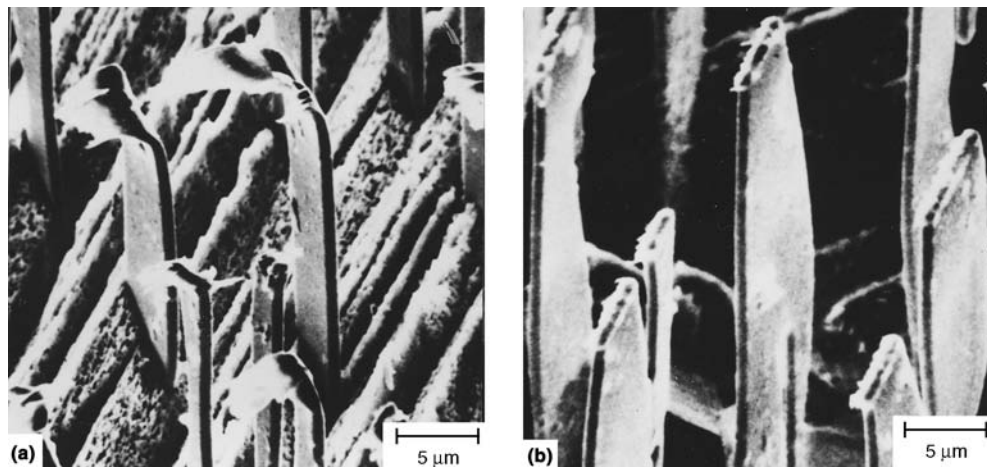


Fig. 23 Surfaces of a eutectic bismuth-silver alloy (a) abraded on 600-grade silicon carbide paper and (b) polished on 3 and 0.05 μm aluminum oxide abrasives. Original magnification 2200 \times

how different features may be dispersed throughout the matrix. For example, it should be instructive to realize that any pore present in the final ground-and-polished sample should not be larger than the largest powder particle; thus, if a pore of 50 μm in size appears when the largest powder particle was less than 30 μm , it likely suggests faulty preparation techniques. Another consideration is that tungsten carbide/cobalt coatings contain very hard tungsten carbide particles (in the range of 2000 HV) embedded in a softer cobalt matrix (approximately 600 HV). These two materials will grind and polish very differently. If the specimen is to be viewed at moderately high magnifications (1000 \times and higher), the largest scratch on the specimen should be less than 1 μm (which would appear 1 mm, or 0.4 in., wide at 1000 \times), necessitating polishing using 0.05 μm abrasives, for example.

Each of the requirements stated previously for grinding are also required for polishing; that is, the microstructure revealed should be true and undisturbed, the largest polishing artifact should be of smaller size than that observable at a particular magnification, and the entire field of view should be in focus.

Polishing Concepts/Mechanisms

Many of the same general concepts that are important in grinding are also applicable to polishing. Basically, there are considered to be two types of material removal mechanisms, similar to grind-

ing. These include either a rolling abrasive particle or a fixed abrasive particle that either plows a groove or cuts a chip (Fig. 21). The first mechanism is the rolling abrasive mechanism suggested in Fig. 21 and discussed in the section on grinding with laps. For polishing, a hard, napless cloth is considered to act with much the same mechanism, that is, with a rolling abrasive. The second mechanism is associated with napped cloths consisting of fibers, as is discussed shortly. Abrasive particles embed on the fibers. The embedded particles are held against the surface of the specimen by the elasticity of the fibers, which act as cantilever springs, as shown in Fig. 26—very light springs for high-napped cloths and somewhat heavier springs for medium-napped cloths. Thus, the two significantly different polishing mechanisms are shown in Fig. 26 for napped cloths: essentially an embedded abrasive particle on a nap or fiber that has spring force, with the action being a combined hammering and plowing, and, as illustrated in Fig. 21, a rolling abrasive on a hard surface. It is very important that metallographers understand these two basic mechanisms as they develop metallographic polishing procedures.

Abrasives

Material removal rates during metallographic polishing are an order of magnitude lower than those previously reported for grinding. This is due to the very low number of contacting points (10/cm²). Table 9 (Ref 1) shows the relative polishing rates for silicon carbide, aluminum oxide (alumina), and diamond abrasives, while Table 10 lists comparative polishing rates of several metals and alloys. The highest removal rates are achieved using the coarser grades of silicon carbide and aluminum oxide in the 15 μm range. (It is important to realize that every size has an associated range; the 15 μm size abrasive can have particles ranging from 5 to 40 μm .) Aluminum oxides and silicon carbides are usually deposited as a slurry that is mixed in the laboratory.

Diamond abrasives are applied either as a suspension, an aerosol, or a paste. Pastes can be color-coded for easy identification of the abrasive size. Diamonds, in addition to being significantly harder than most materials being abraded, also exhibit an unusually low coefficient of friction, which could result in lower critical rake angles necessary to achieve cutting.

Colloidal silica, which Ref 1 suggests to be in the hardness range of HV₃₀₀ 400, nevertheless has been shown to be very useful as the only abrasive used in conjunction with hard, napless cloths for polishing most coatings. A rolling mechanism, as previously discussed, is suspected.

The method of adding the abrasive is also relatively important. Aerosol cans are not regarded as being a highly controllable method of dispersing abrasives onto polishing cloths, because the amount of abrasive per unit time of spray is variable and not known. Pastes seem to generate higher abrasion rates, possibly due to more uniform dispersion on the polishing cloths and also possibly due to holding the abrasives in the upper regions of the nap on the cloth. In addition, the paste may provide an effective means of attaching abrasive particles to the fibers.

The maximum effective abrasive particle size for 70:30 brass has been shown to be of the order of 6 μm . Larger particle sizes tend to decrease the polishing rate. This is possibly a function of

Table 7 Material removal rates, flatness, and scratch depths for hard versus soft formats

Characteristic	Hard	Soft
Material removal	Lower	Higher
Flatness	Flatter	More rounded
Scratch depth	Most	Least

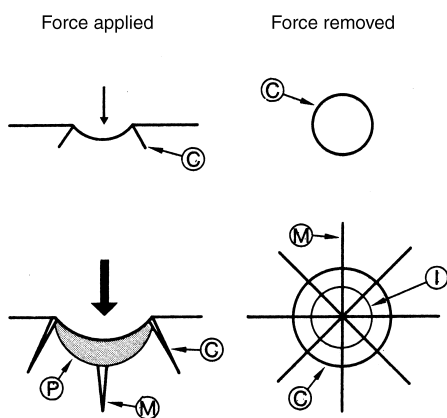


Fig. 24 Schematic illustrating the sequence of events initiated when a spherical indenter is forced into the surface of a brittle material under an increasing load (top to bottom), and the events that occur during unloading. Left, load applied; right, load removed. P, pseudoplastic zone; C, cone crack, also known as a ring crack when viewed on the surface; M, median vent crack; and I, permanent impression

the fiber diameter, the fiber being capable of retaining abrasive only up to a certain size. Removal rates increase with increasing amounts of abrasive up to approximately 1% by weight of abrasive in the paste.

Polycrystalline diamond has been shown to have significantly higher polishing rates than those obtained with monocrystals of the same nominal grade when applied with standard carrier paste. The reason for the higher rates is possibly due to more angular points being available for polycrystalline diamond; additionally, it is possible that the polycrystalline diamond more easily embeds in the fibers of the cloth.

Polishing Cloths

Numerous types of polishing cloths are available, but these can be subdivided into four distinct categories: papers, felts, woven cloths, and napped cloths. Figure 27 illustrates a hard-format paper cloth; Fig. 28 shows a variation on this, with more fibrous material added. Figure 29 shows a billiard cloth (felt). Nylon woven cloth is shown in Fig. 30, and finally, a high-napped cloth with long fibers is shown in Fig. 31.

With regard to the previous discussion of the spring-type action of an abrasive embedded in a fiber performing polishing (Fig. 26), it is to be expected that those polishing cloths that have low or no nap (i.e., papers) will have lower material removal rates. Also, as with hard-format grinding devices (discs), the tendency to form scratches will be higher, and the tendency to round edges will be

less (dramatically better edge retention is observed when using a combination of filled cold-mount material and hard, napless cloths for polishing). Of final importance for polishing cloths is that a reduction in polishing rate is sometimes observed as polishing debris accumulates in the fibers; this situation would be expected to be more critical for those cloths that have more closely packed fibers, because debris would not be able to escape and would accumulate more quickly, and also, more critical for the nylon-type cloths, because it would be very difficult for the polishing debris to travel between the woven fibers. Polishing cloths should therefore be changed after a few uses; the authors' laboratories prefer to change cloths after a maximum of five uses.

Polishing Lubricants

The choice of lubricant can be a very significant parameter in metallography. Historically, two types of lubricant have been used: water based and oil based (e.g., kerosene). Higher polishing rates are obtained when using oil-based lubricants in combination with napped cloths. Scanning electron microscopy work performed by Samuels (Ref 1) suggested that the main reason for this was that the oil-based lubricants maintain the abrasive higher on the nap than do water-based lubricants. When the abrasives are embedded higher on the fibers or nap, they can hammer with greater force. Unfortunately, for some coatings, this is accompanied by a corresponding increase in porosity, especially the generation of larger pores. Figure 32 shows a tungsten carbide/cobalt

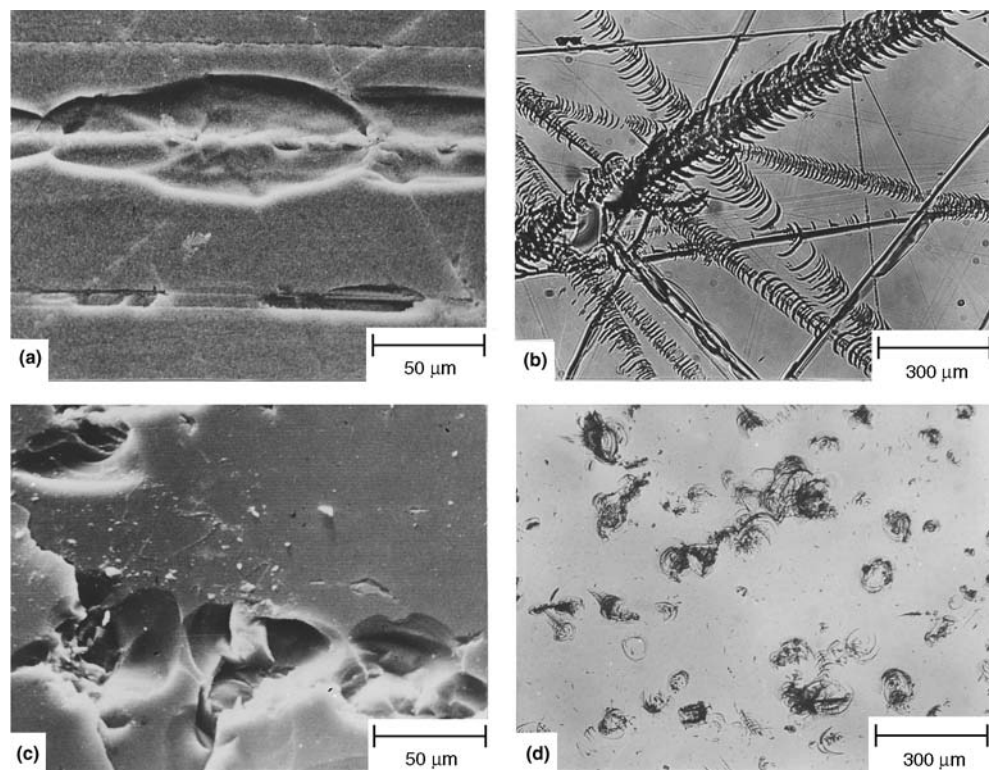


Fig. 25 Surface of fused silica abraded with 220-grade silicon carbide abrasive under constant load. (a) Two-body abrasion. Fresh, sharp abrasive particles. SEM. 300 \times . (b) Two-body abrasion. Worn, blunt abrasive particles. Optical micrograph after etching. 50 \times . (c) Three-body abrasion. Fresh, sharp abrasive particles. SEM. 300 \times . (d) Three-body abrasion. Worn, blunt abrasive particles. Optical micrograph after etching. Original magnification 50 \times

coating that has been prepared using water as the lubricant during polishing; Fig. 33 is the same mount after polishing using an oil-based lubricant.

Note: At the time of this writing, common practice for polishing thermal sprayed coatings is to avoid the use of high-napped cloths; further, lubricants are not used. Simple dosing of colloidal silica or diamond onto napless or low-napped cloths is standard procedure for most, if not all, coatings.

Table 8 Time required to remove the abrasion-deformed layer in annealed 30% Zn brass

Treatment stage	Preceding stage	Treatment time(a), min	
		D_{S1}	D_{S2}
P240 silicon carbide paper	Hacksaw; abrasive cutoff wheel	0.6	8.4
	Filed	0.5	3.8
	Ground; turned	0.4	0.9
P800 silicon carbide paper	P240 silicon carbide paper	0.01	0.4
P1200 silicon carbide paper	P800 silicon carbide paper	0.02	0.5
Aluminum oxide/wax lap	P800 silicon carbide paper	0.01	0.4
3 μm diamond, polish	P800 silicon carbide paper	0.6	4.1
	P1200 silicon carbide paper	0.5	2.1
	Aluminum oxide lap	0.4	2.0
1 μm diamond, polish	P1200 silicon carbide paper	2.0	8.8
1 μm aluminum oxide, polish	P1200 silicon carbide paper	2.0	8.8
0.3 μm aluminum oxide, polish	P1200 silicon carbide paper	5.0	22

(a) Time required after the preexisting scratches have been removed to reduce the depth of the deformed layer produced by the preceding stage to that of the deformed layer being produced by the new stage. D_{S1} and D_{S2} are the bases of the abrasion deformation layers in which two different etchants develop manifestations of deformation. Source: Ref 1

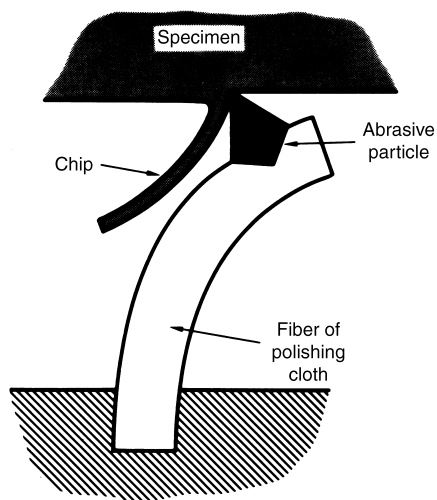


Fig. 26 Schematic illustration of a possible mechanical mechanism of polishing, showing how an abrasive particle could cut a chip when it became embedded in a fiber of the polishing cloth

Polishing Rate Formulas

As with the earlier section discussing grinding rates, those not inclined toward mathematical models can skip this portion of the discussion.

Table 9 Typical abrasives used in metallographic polishing and their polishing rates for 70:30 brass relative to diamond abrasive (6 μm grade)

Abrasive type	Abrasive size range, μm	Relative polishing rate
Silicon carbide	10–20	1.5
Aluminum oxide, α type	10–20	1.5
	0–1	0.3
	0–0.3	0.15
Aluminum oxide, γ type	0–0.1	0.1
Diamond	4–8	1.0
	0–1	0.3

Table 10 Maximum polishing rate obtained in polishing various metals and alloys with two grades of polycrystalline diamond (6 and 3 μm) on a synthetic suede cloth

Metal or alloy	Hardness, HV	Maximum polishing rate(a), $\mu\text{m}/\text{m}$	
		4–8 μm grade	2–4 μm grade
Aluminum:			
High purity	24	9.9	17.2
Alloy (4.5% Cu)	105	9.0	15.0
Chromium: high purity	200	0.42	0.1
Copper:			
Commercial purity	50	5.6	8.8
Brass (30% Zn)	45	8.5	12.5
Brass (40% Zn), leaded	155	10	14.5
Aluminum bronze (11% Al)	200	4.0	6.5
Gold: high purity	22	0.10	0.18
Lead:			
Commercial purity	4	4.0	4.9
Alloy (40% Sn)	13	5.6	6.5
Nickel:			
Commercial purity	130	2.8	3.8
Alloy	260	1.8	2.0
Platinum: high purity	40	1.9	1.9
Steel:			
0.15% C, annealed	130	2.2	3.1
0.75% C, heat treated	340	1.8	3.0
0.75% C, heat treated	800	1.4	2.0
1.4% C, heat treated	840	2.1	1.6
Austenitic, type 304	105	2.2	2.8
Silver: high purity	35	3.0	7.8
Tin: high purity	9	2.5	4.4
Titanium:			
Commercial purity	200	1.8	1.8
Alloy (6% Al, 4% V)	295	1.6	2.1
Tungsten carbide (12% Co)	1550	1.3	1.4
Zinc:			
Commercial purity	35	8.3	10.4
Alloy (4% Al, 1% Cu)	90	7.3	11.8

Note: The abrasive was applied in the reference carrier paste, and kerosene was used as the polishing fluid. Conditions otherwise optimized, as described elsewhere in this article. (a) Under the particular conditions used, this corresponds to depth removed per minute of polishing time. Source: Ref 1

Polishing rate increases in direct proportion to the load applied. For most polishing processes, including those referred to in Ref 1, 40 kPa (5.6 psi) per 32 mm (1.25 in.) mount is common.

Reference 2 presents a series of equations for polishing that are very useful. Some of those are given (but not defended) here. The number of polishing steps necessary to proceed from an initial roughness to a final roughness can be represented by:

$$f_{\min} = \ln (S_o/S_f)$$

where f_{\min} is the number of steps, S_o the initial roughness, and S_f the final roughness. The size of abrasive required is given by:

$$S_1 = (S_o^2 S_f)^{1/3}$$

$$S_2 = (S_o S_f^2)^{1/3}$$

where S_1 is the first abrasive and S_2 the second abrasive.

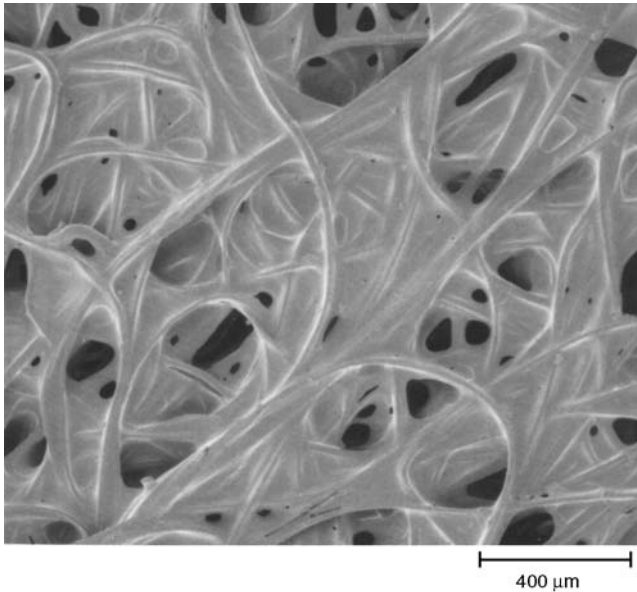


Fig. 27 SEM of hard-format paper cloth for polishing. Original magnification 50x

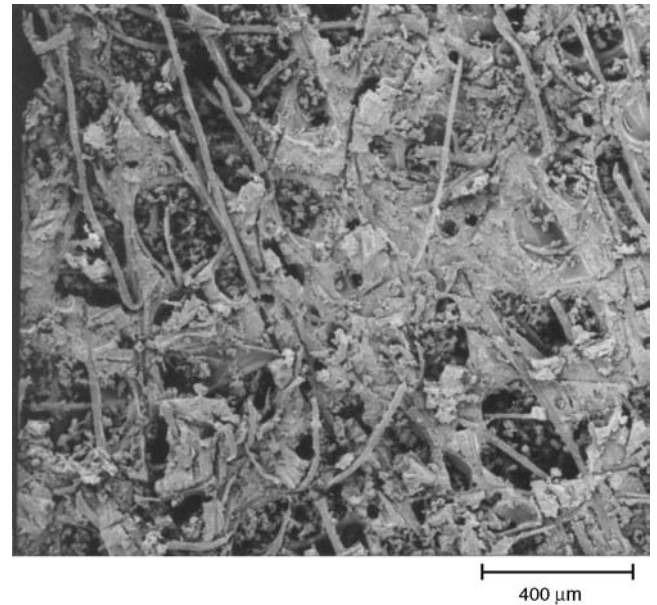


Fig. 28 SEM of hard-format paper cloth for polishing, similar to that shown in Fig. 27, with more fibrous filler. Original magnification 50x

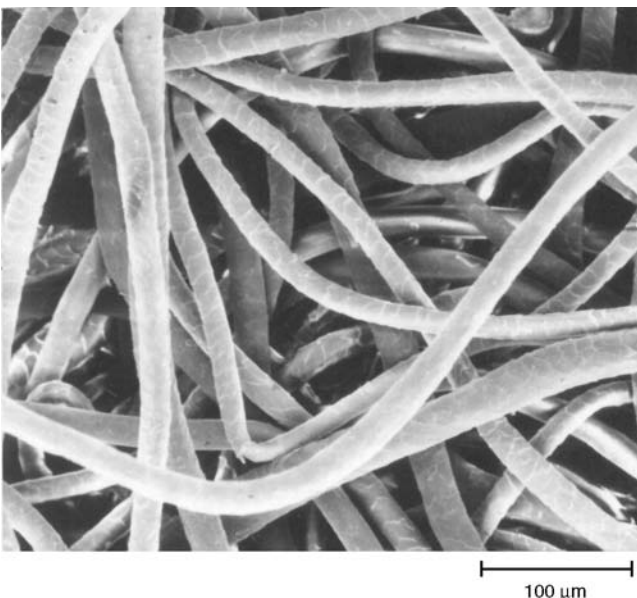


Fig. 29 SEM of billiard cloth for polishing. Original magnification 200x

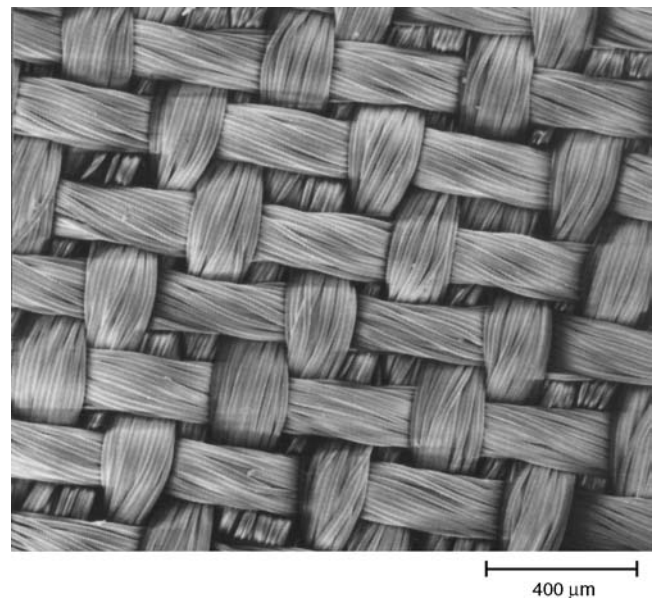


Fig. 30 SEM of nylon woven cloth for polishing. Original magnification 50x

Finally, the total time required can be estimated as:

$$T = (f_{\min} e) / \alpha$$

where α is a constant depending on the nature of the abrasive and the applied pressure, and $e = 2.72$.

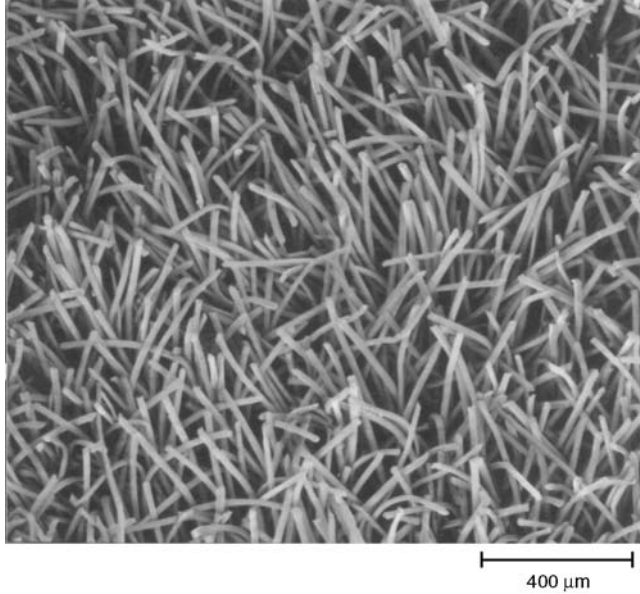


Fig. 31 SEM of high-napped cloth. Original magnification 50×

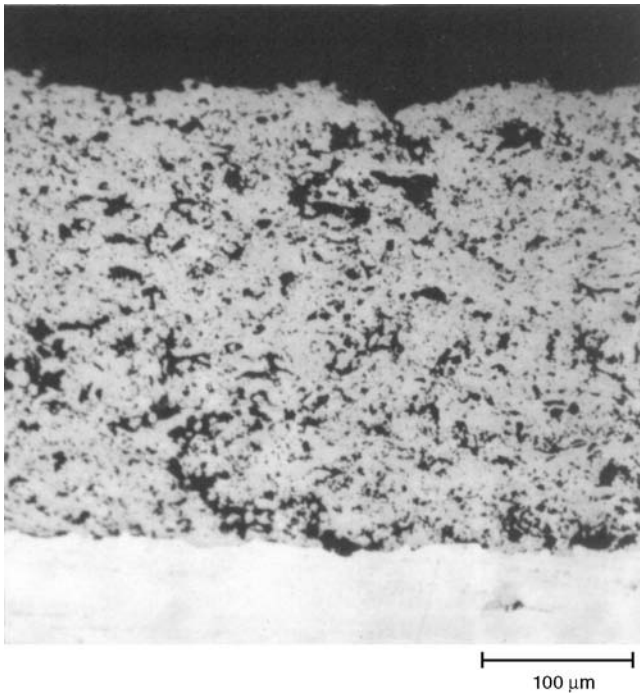


Fig. 32 Tungsten carbide/cobalt coating prepared with high-napped cloth, diamond abrasive, and water-based lubricant. Original magnification 200×

If the initial surface roughness is 30 μm , the final surface roughness is 1 μm , and α is a constant equal to 1.2, the following results:

$$f_{\min} = \ln (30/1) = 3.4, \text{ or three polishing steps}$$

$$T = 3(2.72)/1.2 = 6.8 \text{ min}$$

$$S_1 = [30^2 (1)]^{1/3} = 9.4 \mu\text{m}$$

$$S_2 = [30 (1^2)]^{1/3} = 3.1 \mu\text{m}$$

According to Ref 2, the aforementioned result was found to work in practice.

While the foregoing mathematical model may be applicable to wrought, cast, or forged materials, it has not been used, to the authors' knowledge, in the development of polishing procedures for thermal sprayed coatings. It is included here to share with those readers inclined to mathematical models in order to acquaint them with work that has been performed in this area.

One additional subject that must be dealt with is the occasional need to introduce some relief via the final polishing step in order to properly resolve coating microstructural constituents that are otherwise difficult to distinguish (such as carbide volume fraction assessment in tungsten carbide/cobalt coatings). Figure 34 shows a fine tungsten carbide/cobalt coating that has been polished using diamond; the diamond, being much harder than either of the coating constituents, cleanly cuts both the cobalt matrix (approximately HV₃₀₀ 600) and the tungsten carbide particles (approx-

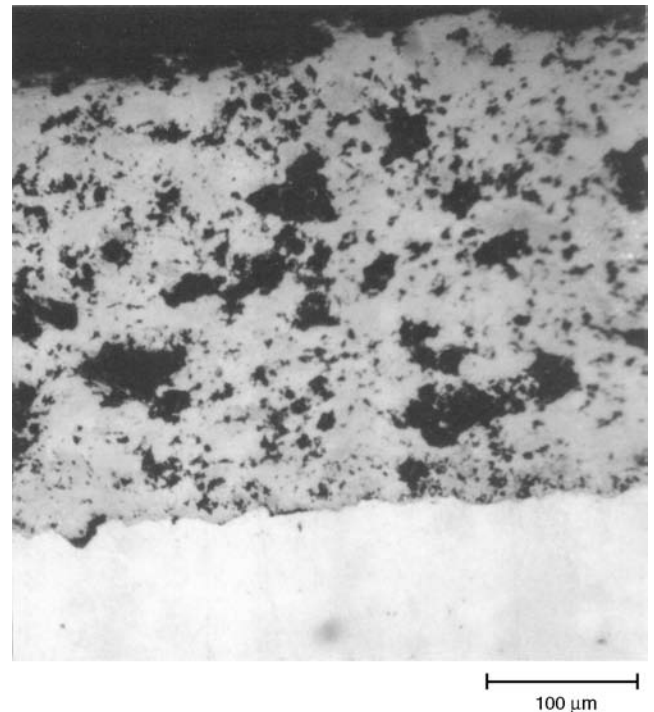


Fig. 33 Tungsten carbide/cobalt coating prepared with high-napped cloth, diamond abrasive, and oil-based lubricant. Original magnification 200×

mately HV_{300} 2000). The specimen is very flat, and very little relief of the carbide occurs. Comparatively, when colloidal silica was used as the polishing medium, its hardness was insufficient to cleanly cut the tungsten carbide particles. The cobalt matrix was therefore removed selectively, leaving the carbides standing in relief (Fig. 35). Carbide volume fraction is thus much easier to assess when using colloidal silica. While diamond cuts both the carbide particles and the surrounding cobalt matrix, and therefore produces a surface where the carbide particles are not in relief, it can be observed, by comparing Fig. 34 and 35, that the diamond polish is more porous. Both were polished on a napless cloth at 35 kPa (5 psi) per 32 mm (1.25 in.) mount at 150 rpm. The only difference was the abrasive.

It should be noted that coarse and fine tungsten carbide/cobalt coatings do not respond the same. The coarse tungsten carbide/cobalt coatings are somewhat resistant to polishing, thus requiring long polish times to reveal their proper coating microstructures. They respond very similarly to yttria-stabilized zirconia coatings, as is discussed subsequently. Figure 36 shows a coarse tungsten carbide/cobalt coating after long polish times. Figure 37, 38, and 39 show the probable cause of the increase in apparent porosity when using diamond instead of colloidal silica (Fig. 34 instead of Fig. 35) and suggest the mechanism. Figure 37 shows diamond polishing of a fine tungsten carbide/cobalt particle after 2 min of polishing. The high-magnification image shown in Fig. 37 reveals that the apparent pores are actually associated with carbide fracture. Figures 38 and 39 clearly reveal that continued polishing with diamond causes the microstructure to become progressively

more porous with time. It should also be noted that with increased polish time, increased rounding of the specimen occurs—note the significant out-of-focus areas in Fig. 39 after just a few minutes (i.e., 6 or less min) of polishing. The suggested cause for increase of porosity with time involves both an aggressive metallographic mechanism and a coating microstructure that is sensitized due to spraying:

- When diamond is used with a hard, napless cloth, the rolling abrasive action shown in Fig. 21 predominates; the diamond is much harder than either the tungsten carbide particles or the surrounding cobalt matrix. The metallographic mechanism, then, is a rolling, extremely hard abrasive on a hard, napless cloth.
- It is well established in the literature that plasma spraying of tungsten carbide/cobalt can cause solutioning of the carbides; the region around the dissolving carbides then becomes rich with three elements: cobalt, tungsten, and carbon. Ternary compounds known as eta phases can and do develop in the near-carbide regions. Finally, it is also known that the eta phases are brittle.

The resulting suggested combinatorial mechanism, then, is fracturing or puncturing of the near-carbide regions, often comprised of brittle eta phases, by the very aggressive rolling diamond abrasive. With increased polish time, more fracturing occurs, resulting in more apparent porosity. Thus, if polishing with diamond is essential for these coatings, minimization of polish time will help to reduce apparent porosity.

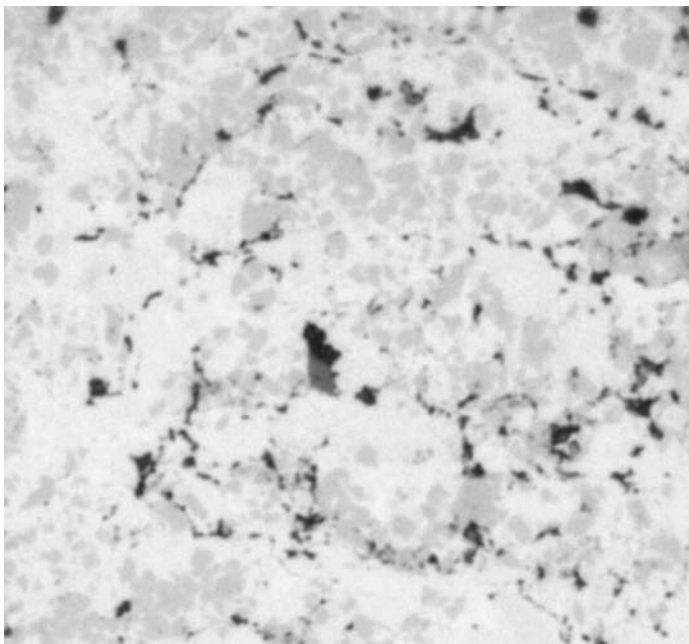


Fig. 34 Fine tungsten carbide/cobalt coating prepared using diamond. Original magnification 200×

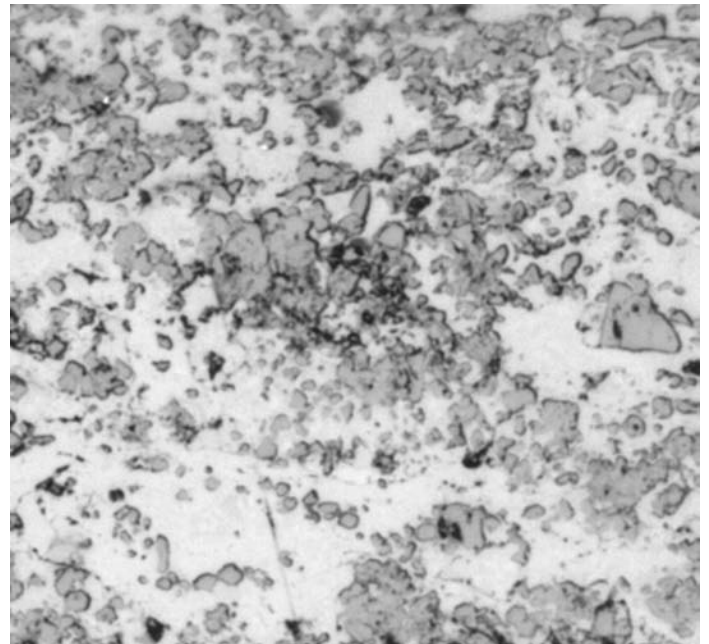


Fig. 35 Fine tungsten carbide/cobalt coating prepared using colloidal silica. Original magnification 200×

Recommended Procedures

The previous sections discussed and presented the various aspects of metallographic preparation, including pertinent examples from thermal sprayed coatings. The discussion includes significant extractions from Ref 1 and 2.

Specimen Sectioning

Care during sectioning of thermal sprayed coatings is extremely important. If sectioning is not performed properly, cracking, debonding, or excessive deformation of the coating and/or substrate may result in a false or misleading microstructure.

Damage due to sectioning can be avoided by using small coating coupons that do not require cutting prior to mounting. Another option is to section in a way that an uncut surface is exposed for grinding and polishing. If sectioning cannot be avoided, vacuum impregnation of porous coatings using a two-part epoxy resin

before sectioning has been shown to reduce coating damage. See *Metallography and Microstructures*, Volume 9 of *ASM Handbook* (Ref 4).

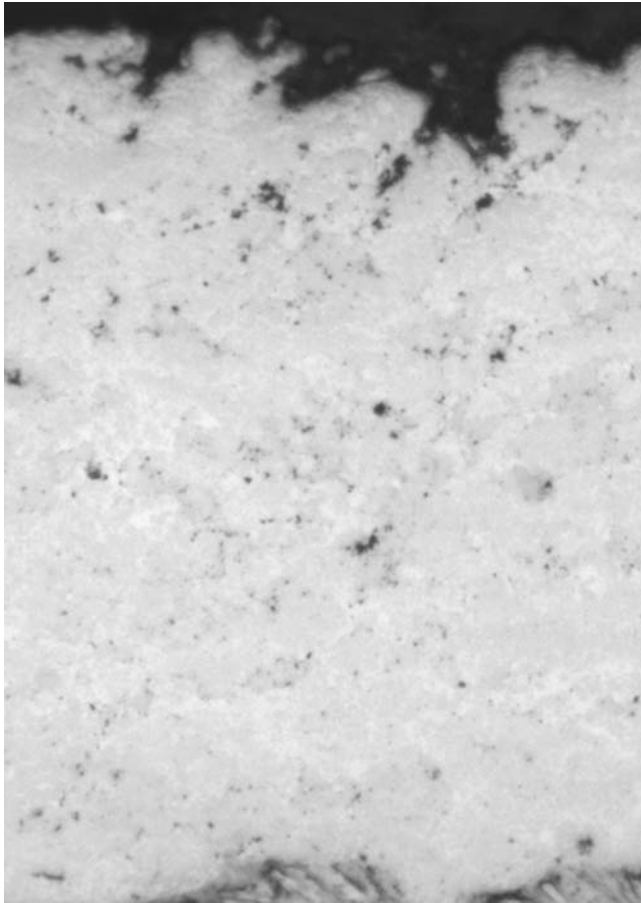


Fig. 36 Coarse tungsten carbide/cobalt coating polished with 1 μm polycrystalline water-based diamond suspension on a napless cloth for long times (>15 min) at 150 rpm, 35 kPa (5 psi) per 32 mm (1 $\frac{1}{4}$ in.) mount. Original magnification 200 \times

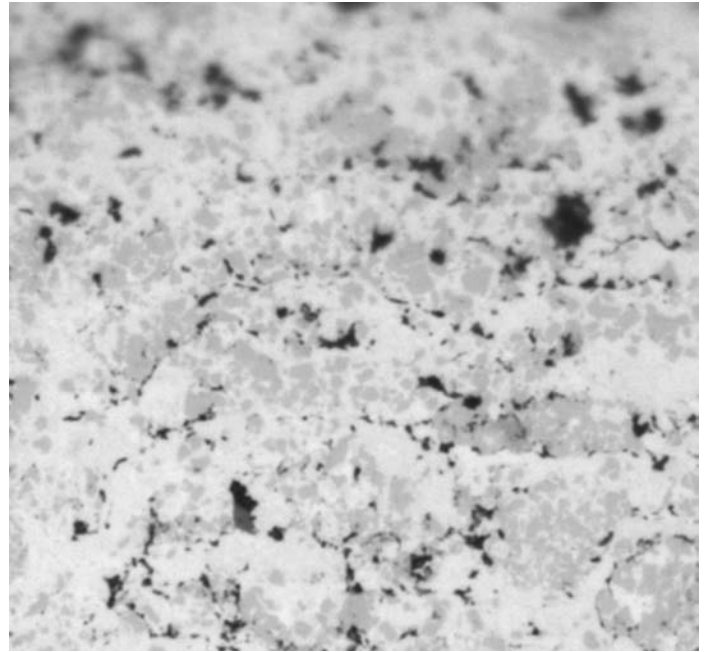


Fig. 37 Fine tungsten carbide/cobalt coating polished with 1 μm diamond suspension on a napless cloth at 150 rpm, 35 kPa (5 psi) per 32 mm (1 $\frac{1}{4}$ in.) mount for 2 min. Note that the apparent pores are associated with carbide or near-carbide fracturing. Original magnification 500 \times

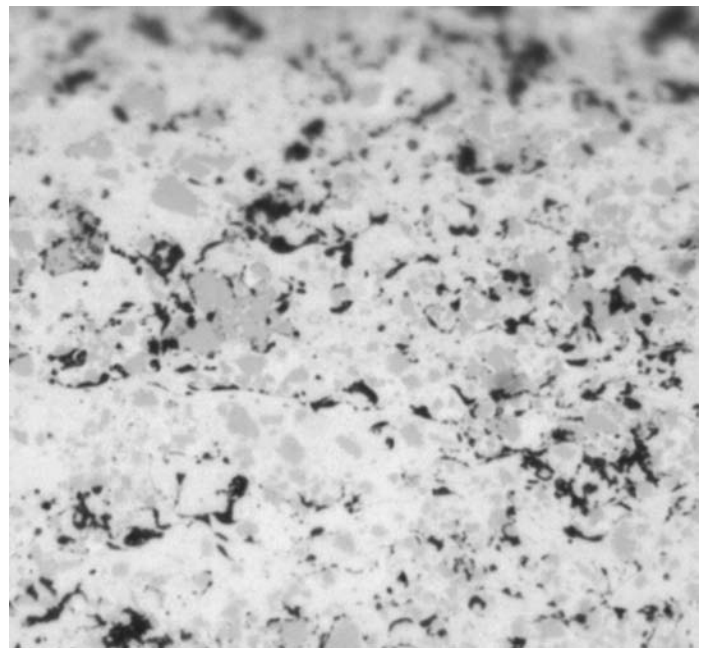


Fig. 38 Same sample as shown in Fig. 37, polished for a total of 4 min. Note increased apparent porosity due to continued/increased carbide fracturing. Original magnification 500 \times

Cleaning

After sectioning and during specimen preparation, cleaning is important to obtain high-quality metallographic mounts. A preferred practice is to clean the specimen with soap and water or other cleaning solvents. The use of an ultrasonic cleaner to remove entrapped abrasive from cutting and from preparation is not recommended, because ultrasonic action could weaken inter-particle boundaries, possibly causing separation during sample preparation.

Drying of the specimen after sectioning is important to ensure that mounting media will adhere to the specimen surface during mounting. Hot plates or ovens are typically used for this purpose, at temperatures in the range of 65 to 95 °C (150 to 200 °F).

Specimen Mounting

A common and preferred practice of mounting sectioned thermal sprayed coating specimens is the use of a two-part epoxy that cures at room temperature. Some epoxy systems require vacuum degassing, some do not.

A problematic method commonly employed with thermal sprayed coatings is hot mounting. Hot mounting uses both heat and pressure to cure a thermosetting resin (phenolic). This method has been shown to cause cracking and separation of the coating from the substrate. Compression of the lamellar structures associated with thermal sprayed coatings could result in densifying structures containing porosity. This could lead to inaccurate assessment of the true microstructure of the coating.

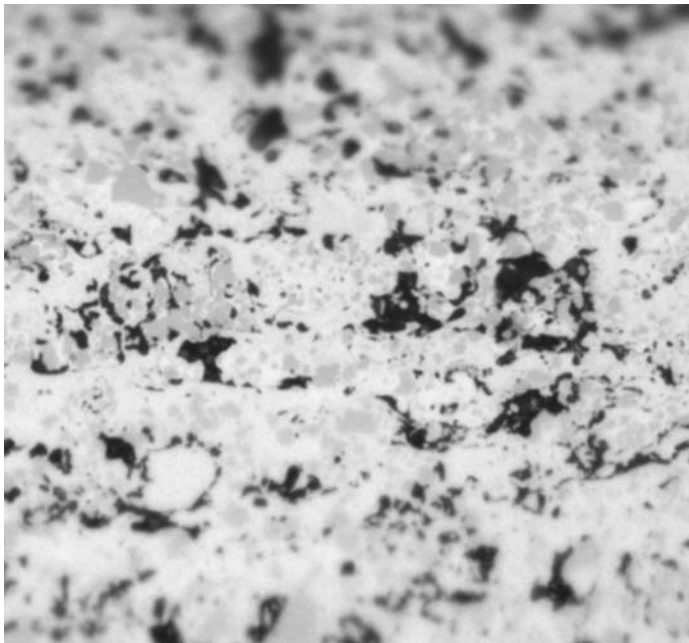


Fig. 39 Same sample as shown in Fig. 37, polished for a total of 6 min. Note progressive increase in apparent porosity due to continued/increased carbide fracturing. Also, note increased specimen rounding due to longer polishing times. Original magnification 500×

Planar Grinding

Planar grinding is used to produce flat specimens in multiple-specimen racks, with all specimen surfaces being on one plane prior to subsequent grinding, hence the term *planar grinding*. All the grinding and polishing steps discussed earlier use automated preparation equipment. The initial grinding step should use an abrasive that is coarse enough to remove all damage due to sectioning without creating new defects in the coating. The options to remove material include: coarse alumina stone, which can be trued (leveled), and coarse silicon carbide abrasive paper.

Intermediate Grinding

The intermediate grinding steps involve the use of sequentially smaller abrasive sizes to remove damage created by the previous grinding step. In the majority of coating applications, silicon carbide papers are the abrasive of choice. A common sequence of papers is 60, 120, 180, 240, 320, 400, 600 grit, with a preferred cutting time of 30 s and a maximum time of 90 s, depending on the quality of the abrasive and the relative hardness of the specimens. Two papers per step should be employed to guarantee removal of the deformation artifacts discussed earlier in this article.

Polishing

Prior to presenting some preferred polishing procedures, it is useful to discuss the response of the coating materials to grinding and polishing. Very few polishing procedures are needed to polish virtually all coatings of interest, a significant benefit.

Yttria-Stabilized Zirconia. Perhaps one of the more controversial coatings to be prepared metallographically is yttria-stabilized zirconia. The controversy ensues over disagreement between laboratories regarding the amount of apparent porosity in the coatings. Since the inception of the use of these materials, there has been a misunderstanding of the true microstructural nature of the coatings; a lack of understanding of the microstructures has caused coatings engineers to postulate incorrectly how the materials perform their functions in their applications. Evidence is presented as follows to suggest that the estimates of porosity in these coatings has been historically too high, and that the coatings are not as porous as previously expected.

Figure 40 is a schematic showing the proposed material response. The grinding papers, in effect, have caused a significant amount of damage to the ceramic material; the abrasive grit gouges divots into the zirconia. The zirconia, under the action of the abrasive grit, does not smear but rather fractures, leaving the proposed divot (Fig. 25a). Polishing for short times, as most laboratories have historically done, leaves much of the divot or fractured area unpolished. As additional polishing time is added, more material is removed, and the divots, which are seen as pores under the microscope, gradually recede and close. After several minutes, the apparent porosity in the coatings asymptotically approaches the true porosity, as suggested by the percent porosity versus polish time graph in the figure. The mechanism postulated is supported by Fig. 41 to 46. These figures show a sample after grinding (Fig. 41) and after polishing for 3 min (Fig. 42), 6 min (Fig.

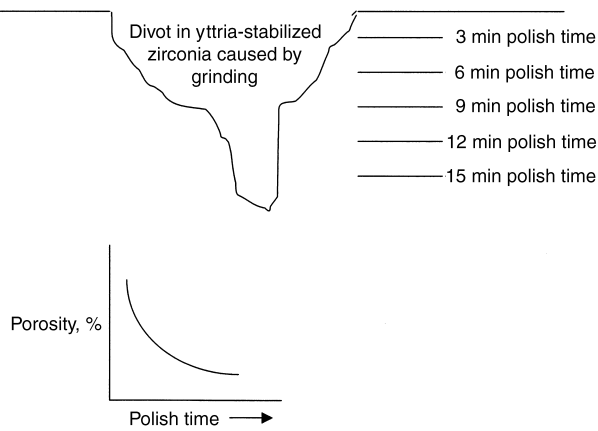


Fig. 40 Schematic of ceramic divot caused by grinding and porosity as a function of polish time

43), 9 min (Fig. 44), 12 min (Fig. 45), and 15 min (Fig. 46). The polishing cloth used was napless—a hard paper, essentially—and the abrasive was 0.05 μm colloidal silica. No water was used. It has been found that this paper is useful for two to three racks of samples. The series of figures is conclusive that perceived porosity, or apparent porosity, for yttria-stabilized zirconia is very clearly a function of polishing time and asymptotically approaches true porosity with increased polishing time.

The recommended procedure for yttria-stabilized zirconia, then, is grinding through 600-grit silicon carbide papers, followed by longer grinding (12 to 15 min) on napless cloths using colloidal silica. Speeds of 150 rpm and loads of 35 kPa (5 psi) per 32 mm ($1\frac{1}{4}$ in.) mount are adequate.

Tungsten Carbide/Cobalt. Another coating material that has been shown to be difficult to prepare is tungsten carbide/cobalt. There are two basic varieties of this powder in use in most thermal

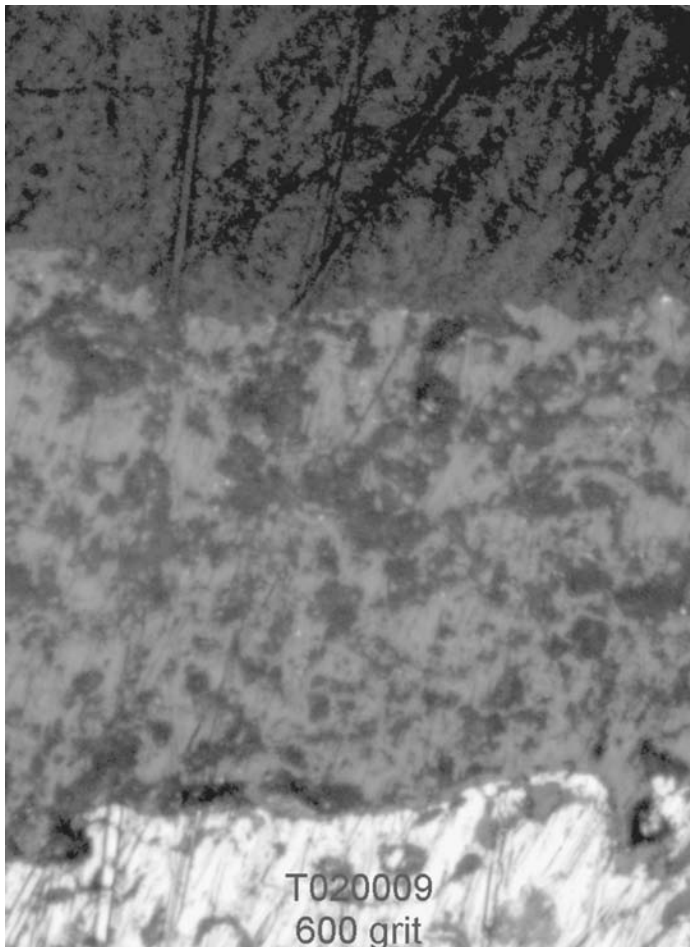


Fig. 41 Yttria-stabilized zirconia after grinding through 600-grit papers. Original magnification 200 \times . See examples of this specimen in the ground-and-polished condition (varying polishing times) in Fig. 42 to 46.

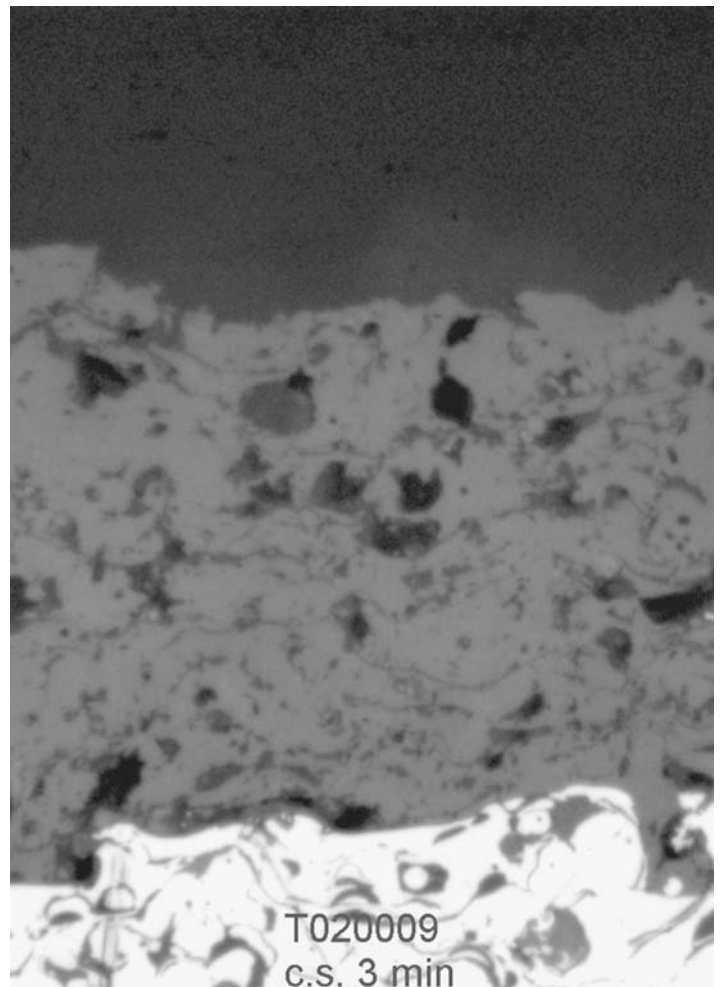


Fig. 42 Yttria-stabilized zirconia after 3 min polish using colloidal silica on a napless cloth at 150 rpm, 35 kPa (5 psi) per 32 mm ($1\frac{1}{4}$ in.) mount. Original magnification 200 \times . Compare with Fig. 43 to 46.

spray shops: the finer AMS 7879 material and the coarser varieties, such as Sulzer Metco's 72F-NS. Each has its own response to the metallographic process.

The finer-grade materials, AMS 7879, have been shown to interact with the abrasives on papers of grit finer than 600. That is, if grinding is carried out using papers finer than 600 grit, for example, 800 grit, the apparent porosity will increase fairly dramatically. From Table 6 it was shown that 600-grit papers have grit sizes of the order of 15 μm . It would thus be expected that the 800-grit papers would have grit sizes approaching 5 μm . The proposed mechanism for the increase in apparent porosity when using 800-grit papers is an interaction between the grit particles and the primary carbide particles, causing dislodging of the carbide particles. The dislodging of the particles will then subject the area surrounding the particles to further damage. It is recommended to stop grinding at 600-grit papers for the finer tungsten carbide materials.

As discussed previously, another sensitivity of tungsten carbide/cobalt materials is due to the formation of so-called eta phases around dissolving carbides. Eta phases are ternary cobalt/tung-

sten/carbon compounds and have been shown in many publications to be of very low fracture toughness. The use of diamond abrasives has been shown to cause a significant amount of fracturing in the near-carbide regions (the exact location of the eta phases). Use of colloidal silica has been shown to have two beneficial effects: no near-carbide fracturing, and the colloidal silica cuts the cobalt matrix while not cutting the tungsten carbide particles, causing the carbide particles to be seen in relief, with much easier assessment of the carbide percentages. Indeed, in recent years, some major thermal spray coatings suppliers have been forced to change their processing parameters, owing to the use of colloidal silica in polishing. When using diamond polishing, there was difficulty in discerning carbide percentages. When the customers switched to colloidal silica, it was discovered that the coatings were virtually carbide-free; they had been dissolving all of the carbides. Figures 34 to 39 and the associated discussion illustrate the use of diamond and colloidal silica.

It is to be noted here that the divot phenomenon associated with the grinding of yttria-stabilized zirconia material does not occur for the fine tungsten carbide/cobalt, owing to the more malleable

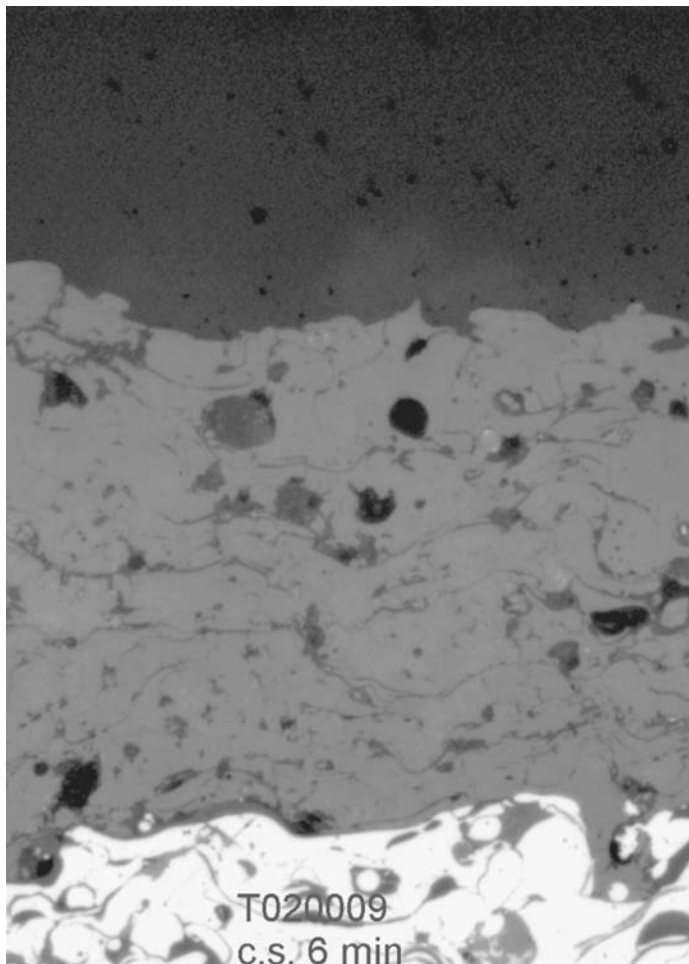


Fig. 43 Same area as shown in Fig. 42 after 6 min total polish. Original magnification 200 \times

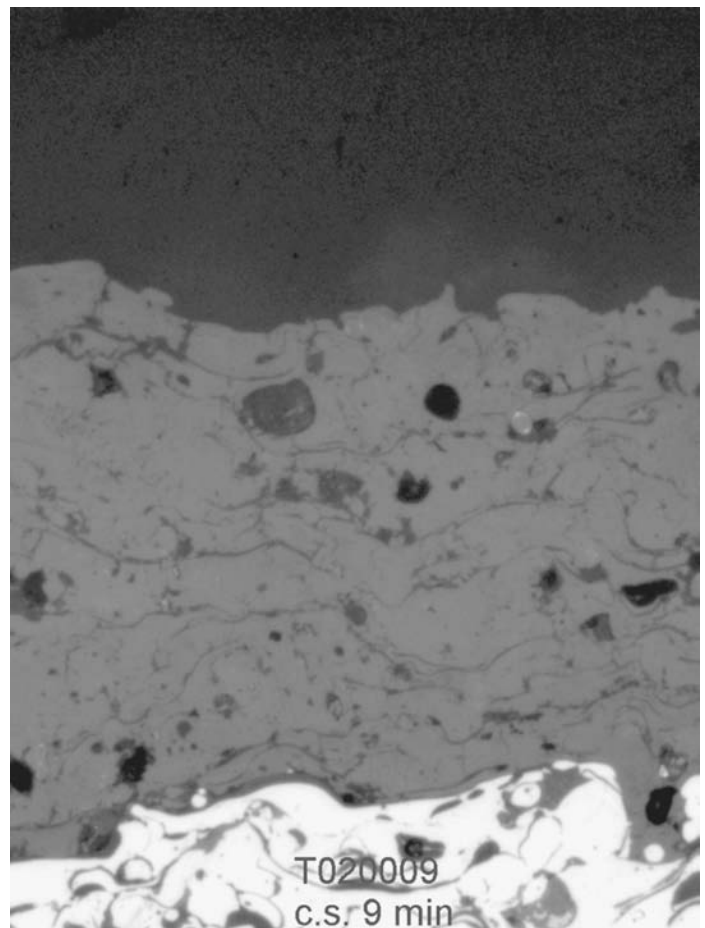


Fig. 44 Same area as shown in Fig. 42 after 9 min total polish. Original magnification 200 \times

cobalt matrix, but does in fact occur for the coarse tungsten carbide/cobalt coatings such as Metco 72-FNS.

The recommended procedure for fine tungsten carbide/cobalt, then, is to grind through 600-grit papers, stopping before going to finer-grit papers to avoid interaction of the silicon carbide particles with the tungsten carbide particles (resulting in higher apparent porosity), and to polish with either colloidal silica or diamond on a napless cloth. Colloidal silica is recommended. However, if diamond is required to be used (i.e., by end-user specifications), polish times must be short to minimize carbide fracture and specimen rounding. Speeds of 150 rpm and loads of 35 kPa (5 psi) per 32 mm (1 $\frac{1}{4}$ in.) mount have been found to be adequate.

Thus, the percent porosity versus polishing time response of yttria-stabilized zirconia and fine tungsten carbide/cobalt are opposite—the yttria-stabilized zirconia requiring extended polishing times to remove grinding-related divots, and the fine tungsten carbide/cobalt requiring short polishing times to minimize carbide fracturing. Coarse tungsten carbide/cobalt coatings follow the yttria-stabilized zirconia mechanism; however, it will be found that long (indeed, very long) polish times are required to obtain a true assessment of porosity.

Chrome Carbide/Nickel Chromium. The chrome carbide/nickel chromium materials contain a somewhat heterogeneous microstructure composed of periodic unmelted particles, some porosity, and oxides that are very prone to fracture. When the oxides fracture, they appear under the microscope as pores, when in fact they are not. Figure 47 shows such a heterogeneous microstructure. The oxides in this coating have survived the grinding/polishing steps, and there are some fine pores and small, unmelted particles. Also readily apparent in this photograph is a separation immediately above the coating/substrate interface. Chrome carbide/nickel chromium materials have been found to exhibit near-interfacial separation sensitivity. This is believed to be due to a lack of elastic resilience of the substrate/coating interface; stated otherwise, the interface gives when too high pressures or too high speeds are used. It will be found that subsequent polishing of the mount represented in Fig. 47 with lower pressures and speeds will eliminate the near-interfacial separation phenomenon.

The recommended procedure for chrome carbide/nickel chromium materials is to grind through 600-grit silicon carbide papers, then polish using napless cloths and colloidal silica (to avoid fracture of the sensitive oxides) with lower than normal

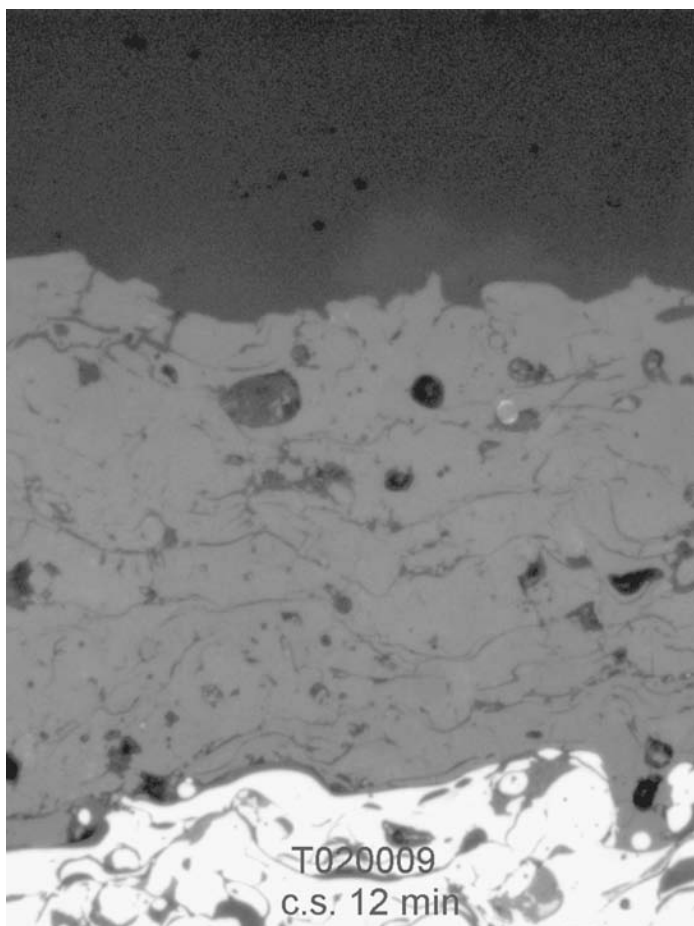


Fig. 45 Same area as shown in Fig. 42 after 12 min total polish. Original magnification 200×

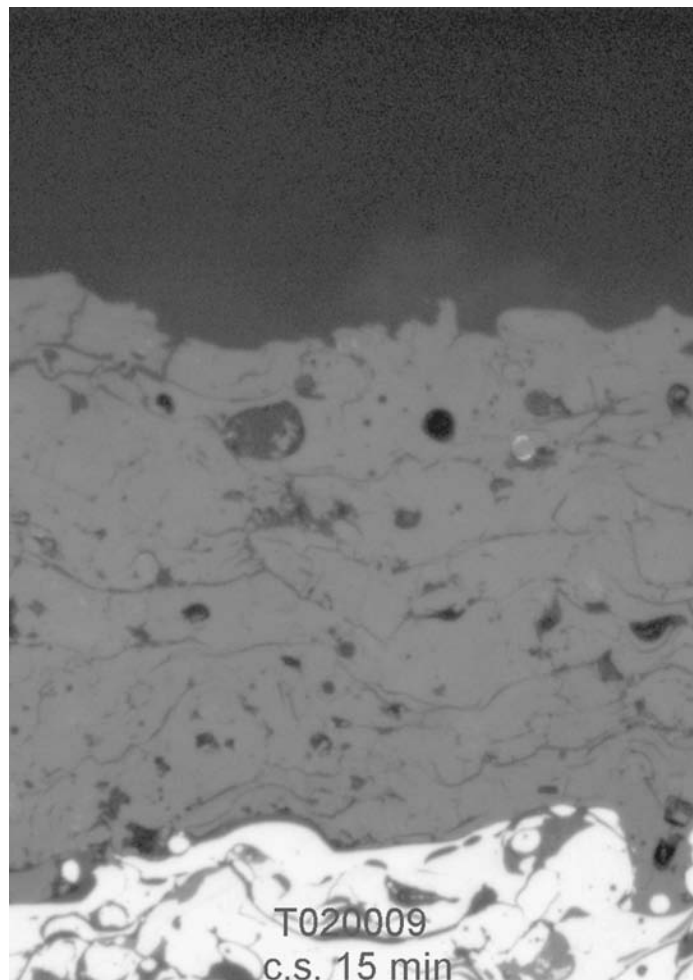


Fig. 46 Same area as shown in Fig. 42 after 15 min total polish. Original magnification 200×

speeds and pressures. It has been found that speeds of approximately 100 rpm and pressures of 14 to 21 kPa (2 to 3 psi) per 32 mm ($1\frac{1}{4}$ in.) mount preserve the oxides intact and do not cause the interfacial separation anomaly.

Soft coatings such as aluminum can also be very difficult to prepare metallographically. It is usually found that finer-grit papers must be used to eliminate scratches, and that fresh polishing cloths also must be used. Furthermore, it is more likely that smearing, or plastic deformation, of the coating will occur, even

during polishing, and as such, the use of etchants is recommended. Figure 48 shows an etched aluminum microstructure after the preceding recommendations (finer-grit grinding papers and fresh polishing cloth) were used.

Polymeric/Metallic Coatings. It has not been found that polymeric/metallic materials, such as aluminum-silicon/polyester, pose significant problems to grinding and polishing.

One Basic Procedure. It is likely to be of interest to the user community that one basic procedure can be developed for virtually all coatings, and that only slight variations in the procedure are necessary for even the most difficult of coatings. Here is one example:

- Sectioning using an abrasive cutoff saw
- Mounting using a standard epoxy system with glass bead filler; cure time, 30 min
- Grinding: 6 mounts maximum, 35 kPa (5 psi) per mount, 32 mm ($1\frac{1}{4}$ in.) mounts, 150 rpm, 60 s, two papers per grit size, 60-, 120-, 180-, 240-, 320-, 400-, and 600-grit papers
- Polishing: napless cloth (for example, Strayers Pan-W, Buehler Tex-Met), blue colloidal silica, 35 kPa (5 psi) per

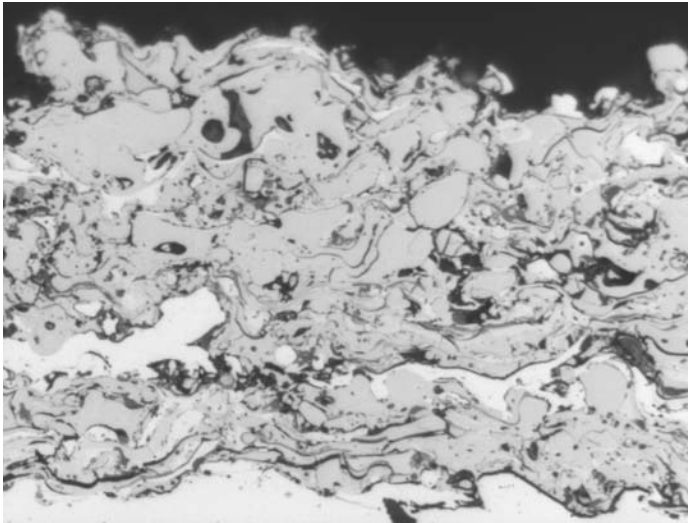


Fig. 47 Chrome carbide/nickel chromium showing separation at the near-interface region due to too high of a combination of speed and pressure, suggesting inelastic material response. This condition is remedied by repolishing at slower speeds and lower pressures. Original magnification 500x



Fig. 48 Etched aluminum microstructure (any of several caustic etchants will work) showing splat structure and porosity. Original magnification 200x

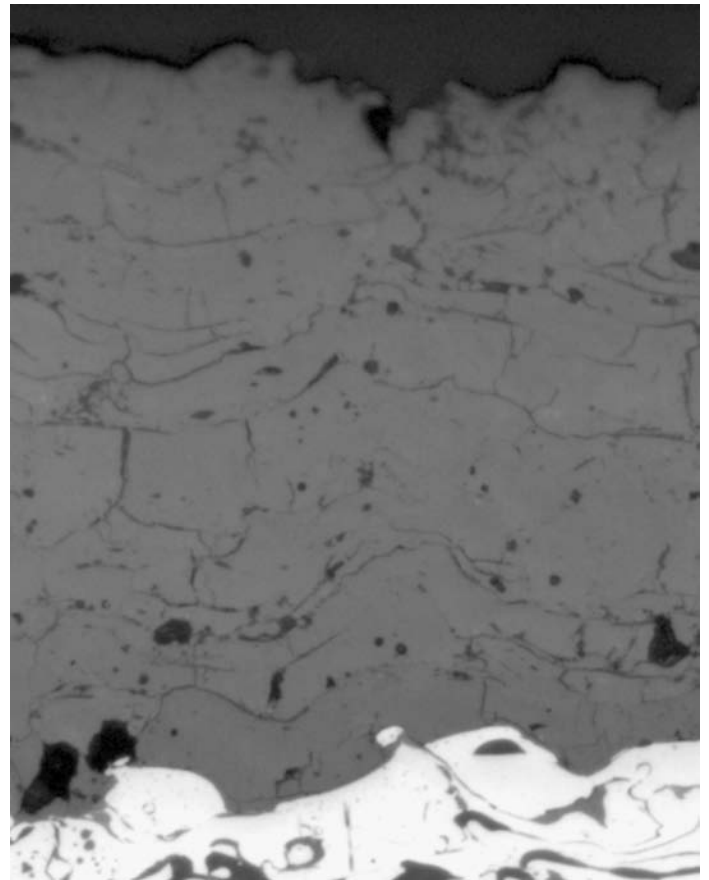


Fig. 49 First of eight different coating types prepared using one standard procedure described in text. Yttria-stabilized zirconia. Original magnification 200x

mount, 32 mm ($1\frac{1}{4}$ in.) mounts, 150 rpm, for 3 to 15 min, depending on the coating material

Figures 49 to 56 show microstructures of yttria-stabilized zirconia, aluminum oxide, tungsten carbide/cobalt (fine), tungsten carbide/cobalt (coarse), chrome carbide/nickel chromium, aluminum-silicon/polyester, MCrAlY bond coats, and aluminum, all prepared using the previously mentioned procedure, the only variations being time (yttria-stabilized zirconia and coarse tungsten carbide/cobalt require longer times), number of grinding steps (more for the aluminum), and pressure (higher for the tungsten carbide/cobalt and lower for the chrome carbide/nickel chromium material). Otherwise, the same basic procedure was used.

Image Analysis

An area with some level of controversy is the use of image analysis to assess the area percentages of some attribute such as porosity. Perhaps the most fair defense of the choice of the term *controversy* is to point out that, while there have been a multitude of papers and publications in the areas of image analysis and its

related discipline, stereology, the institutions generating the work are almost always either academic or commercial, and the authors of the publications are not the typical laboratory technicians and engineers who must use the systems day-to-day. While the academicians and commercial scientists and engineers devote considerable time and effort to understanding the application of image analysis to the measurement of attributes such as porosity from images of coatings, it will be found that typical production laboratories have not. The learning curve to obtain reliable information from image analysis of materials is not short; issues such as proper specimen preparation (i.e., metallography—consider the entire foregoing discussion), illumination, image capture (including selecting the right camera and frame-grabber board), background correction (for uneven illumination due to microscope optics, dust on the lenses, etc.), gray-image processing, thresholding to select features of interest, binary-image processing, and selection/sorting of the final selected features need to be addressed. Further, it must be stated that a review of several of the publications describing the use of image analysis to assess thermal sprayed coating attributes has shown that they ignore the most sensitive issues in the extraction of information from images and proceed instead to analyze data voluminously, with application of advanced statisti-

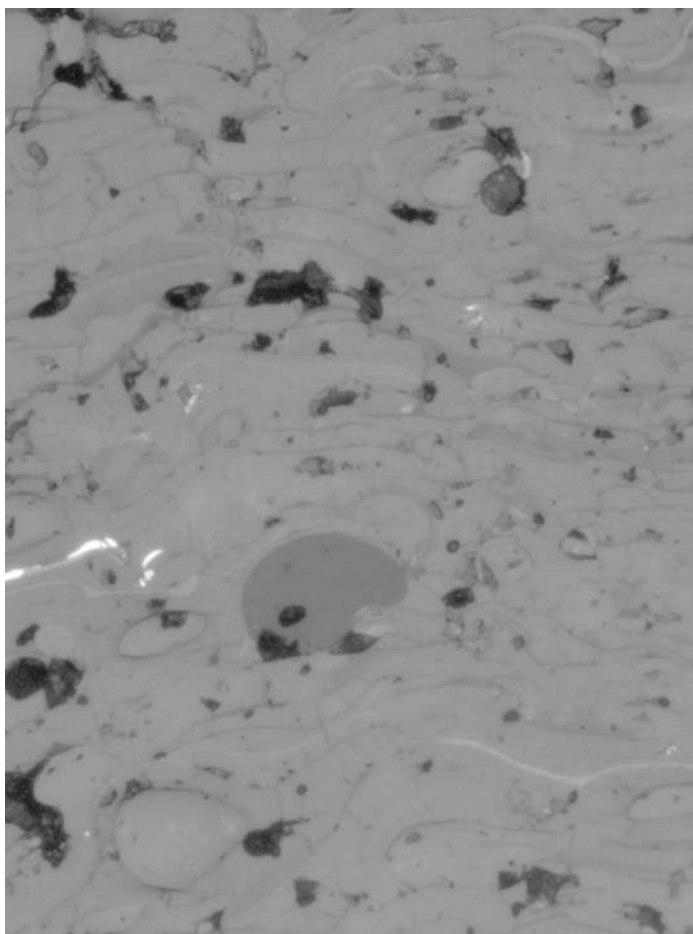


Fig. 50 Second of eight different coating types prepared using one standard procedure. Aluminum oxide. Original magnification 200×

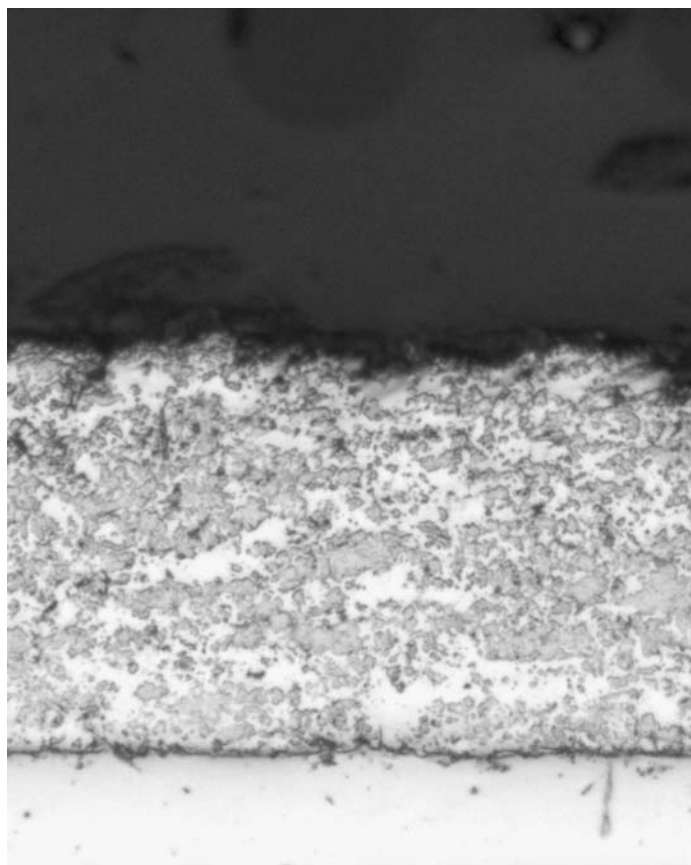


Fig. 51 Third of eight different coating types prepared using one standard procedure. Fine tungsten carbide/cobalt. Original magnification 200×

cal methods to assert that something of significance has been learned. In this section, the more rudimentary issues of concern to the using laboratory are discussed, with the hope that the reader is enabled to assess microstructural attributes, such as porosity, with greater confidence.

The Microscope. Image analysis, it must be recalled, is computer-assisted digital microscopy that is, a camera and frame-grabber board are used to digitize images for subsequent analysis using a computer. A somewhat trite phrase years ago was “garbage in/garbage out.” That is nowhere more true than image analysis. The ability to extract information from images with accuracy and confidence is inextricably dependent on the quality of the microscope. Some low-cost microscopes use 25 W tungsten filament bulbs. Such low-wattage illumination sources have “hot spots” in the center of the image. No amount of gray-image processing, including background subtraction and/or mathematical schemes, will be able to overcome this problem. Halogen light sources of 100 W have been found to overcome the lower-wattage hot spot phenomenon.

Image Analysis versus Reflected Light Microscopy of Metallographically Polished Thermal Sprayed Coatings. The microscopes used to assess thermal sprayed coatings are reflected light. In reflected light microscopy, incident light beams strike a target, such as a polished metallic surface, and bounce off or reflect. The angle of reflection is dependent on the incident angle, the smoothness or roughness of the surface, and the wavelength of the incident light. Regarding the smoothness or roughness of the surface, it will be appreciated that a rough surface will tend to scatter the incident beam in many directions; as such, the rough-surface features will not be perceived sharply, either to the human eye or to the digitized image. Features such as coating pores may have sloping sides rather than sharp sides and, as such, partially reflect and partially scatter the incident light. The accumulation of effects from a somewhat undulating surface (i.e., splat-to-splat polishing undulations), relief between phases, and porosity is to yield a significant amount of scatter in the color or gray-scale images; that is, the peaks in the associated gray-scale histograms are not sharp, such as would be the case if the pores had sharp

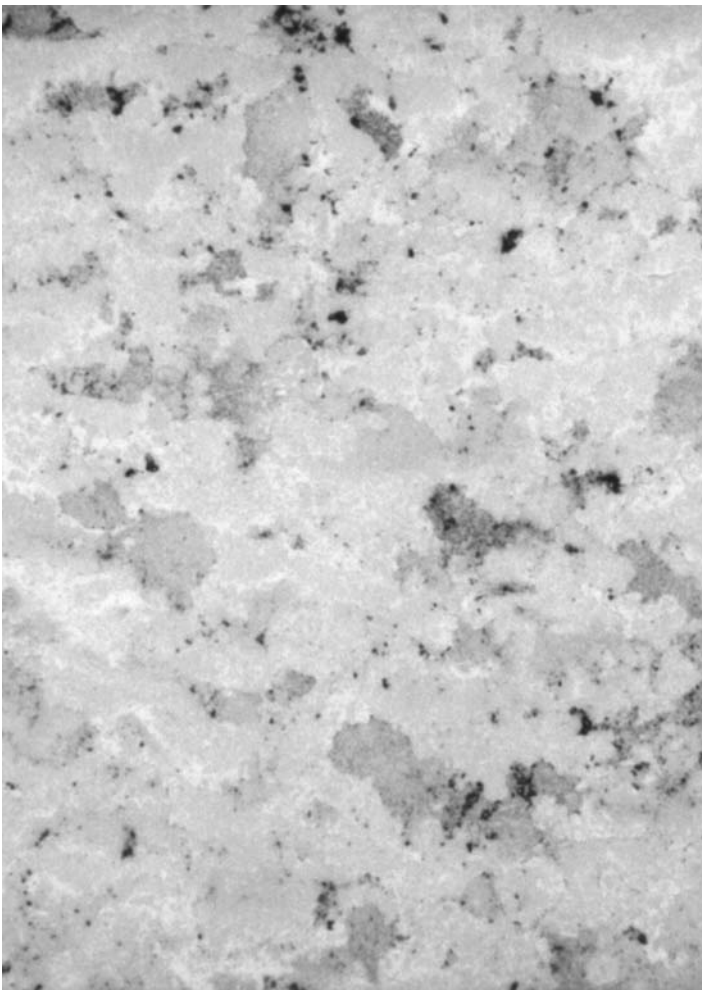


Fig. 52 Fourth of eight different coating types prepared using one standard procedure. Coarse tungsten carbide/cobalt. Original magnification 200×

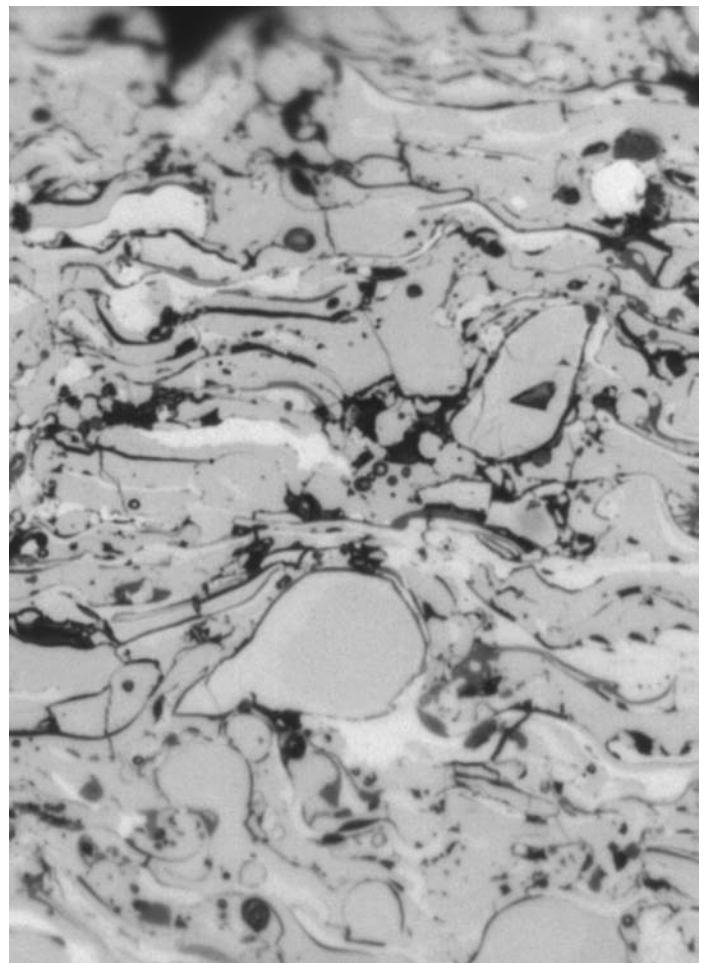


Fig. 53 Fifth of eight different coating types prepared using one standard procedure. Chrome carbide/nickel chromium. Original magnification 200×

edges and were all black and the coatings perfectly flat, perfectly reflective, and all white, and the incident light all of one wavelength (monochromatic): They (the peaks) are instead broad and gradually blend into the rest of the histogram. Regarding lighting, unfortunately, even halogen lighting sources produce white light, that is, a light beam composed of many wavelengths of light. Monochromatic (i.e., one wavelength) or near-monochromatic lighting is preferred. Some better microscopes provide filters intended to minimize light beam wavelength size distributions; more expensive ones have xenon light sources. Microscope optics have also improved. It is to be expected that with improved optics and improved light sources comes improved reliability of the method of image analysis to extract feature-specific information from coatings.

The Camera. In the late 1980s/early 1990s, the typical camera and frame-grabber board system was 512 by 512 pixels and gray scale only. The pixels on the charge-coupled device cameras were relatively large, and as a result, images were not sharp or crisp. Currently, color cameras and frame-grabber boards are readily available with more than 1000 by 1000 pixel resolution. The individual pixel sizes are less than $5\text{ }\mu\text{m}^2$; the resulting images are

much sharper (indeed, many state that they can see more microstructural features now on the computer screen than they can looking through the microscope).

The Computer. The reader will certainly understand the improvements in personal computers (PCs) since the late 1980s. Whereas many processing speeds were on the order of 30 MHz at the time, processors now commonly approach 2 GHz. In addition, whereas the image analyst in 1990 had to add random access memory (RAM) in order to perform rudimentary image analysis operations (a PC with as much as 16 megabytes of RAM was very unusual, and the owner had to buy new RAM chips to install), current PCs commonly have sufficient RAM for image analysis operations. The net result is that image processing routines, such as computer-aided thresholding analysis, which then took 15 min per image, can now be accomplished in a few seconds per image for the same basic computer algorithm. Indeed, image processing on laptop PCs has become rather routine.

Shade Correction. All microscopes have some level of shading associated with the delivery of the image to the camera. These can be due to the illumination source, the bending of light as it traverses the various lenses of the microscope, bifurcation at

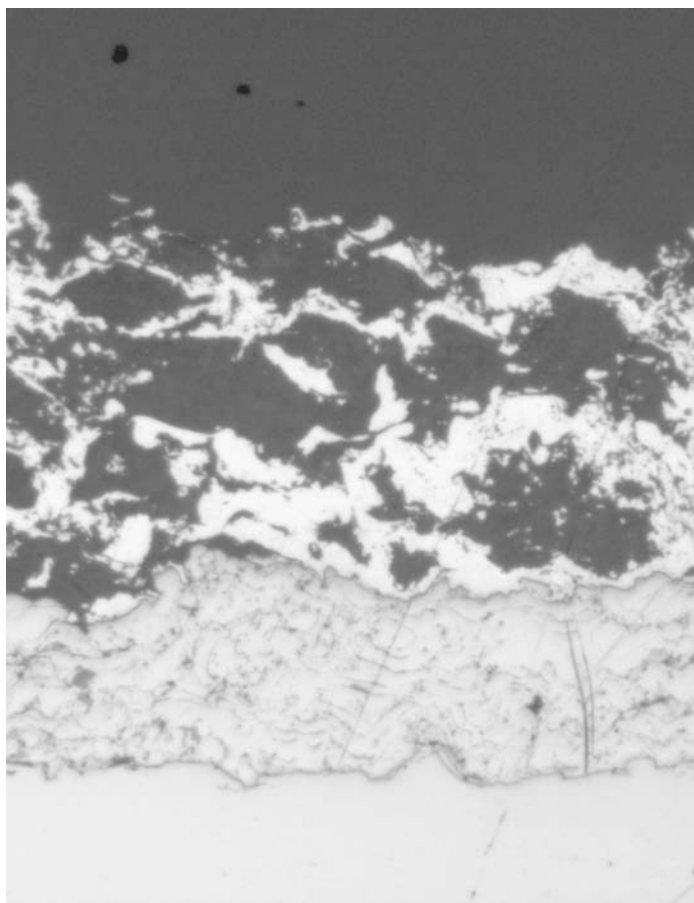


Fig. 54 Sixth of eight different coating types prepared using one standard procedure. Aluminum-silicon/polyester. Original magnification 200 \times

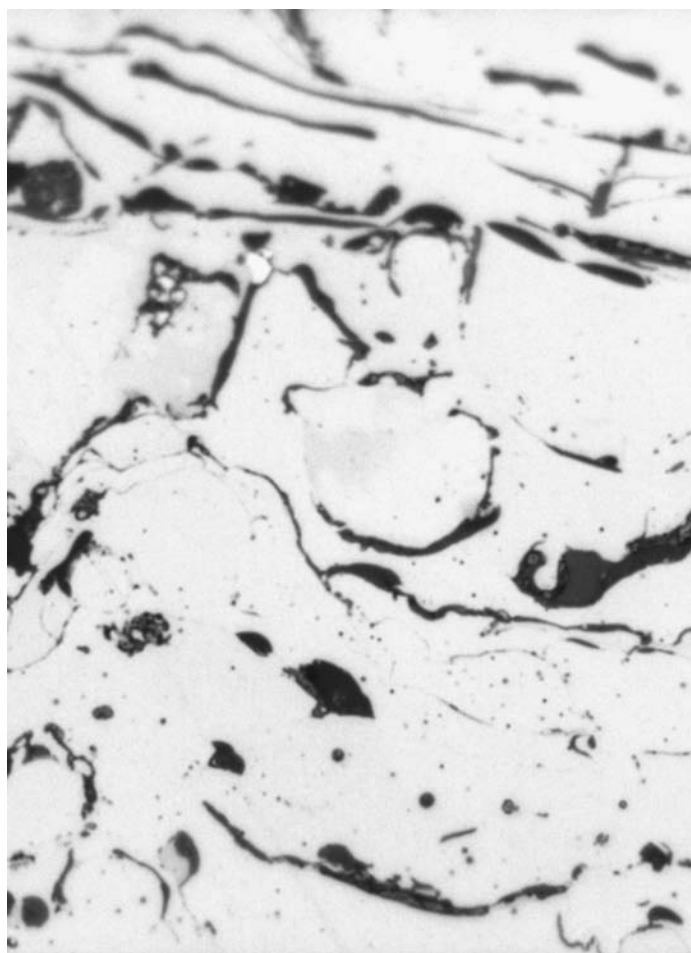


Fig. 55 Seventh of eight different coating types prepared using one standard procedure. MCrAlY bond coat. Original magnification 200 \times

edges, dust on lenses (microscopes should be cleaned by a qualified technician at least once yearly), and so on. The result of these problems can be spots on the image, regions of the image being darker, and so on.

While mathematical models are available to construct a background image that is subtracted from the primary image to correct for shading problems, it has been found that simply capturing an image on the specimen (i.e., the substrate beneath the coating) after defocusing and using the illumination intensity that will be used for imaging the coating works sufficiently well. Any dust on the microscope lenses will be properly dealt with, as well as the various shading anomalies of the particular microscope at the set illumination. The background image is subtracted from the primary images prior to subsequent processing (i.e., gray-scale processing).

Gray-Image Processing—Getting Further from the Truth.

Gray-image processing is performed on images for the sole purpose of making them easier to analyze, that is, making the features of interest more distinct (in gray scale) from the rest of the image, in order to more easily separate or detect those features. Upon digitization, the camera/frame-grabber board system assigns to each pixel in a digitized image a numerical value relating to its brightness (gray-scale images) or values relating to its color, intensity,

and hue (color images). For the present discussion, it is noteworthy that the color images acquired using current systems are very often converted to a gray-scale image for subsequent processing; therein, the following discussion is applicable. Historically, gray scales numerically have ranged from 0 to 255 for 8-bit images and an integer multiple of that for higher-bit images. It is instructive herein to think of an image not as perceived by the human being but as perceived by the computer, that is, not as an image but as a large spreadsheet or two-dimensional matrix with numbers in every cell, the numbers being representative of the gray scale assigned to it by the digitizing camera and frame-grabber board. The gray-image processing routines that the computer can perform (on the numerical values of the pixels) are virtually unlimited and are classified as three different types: point, line, and neighborhood operators. Point operators are very simple to understand and can be thought of as acting on a cell (i.e., a pixel) based on the contents of that cell alone; that is, adding a 25 gray-scale value to each pixel in the image would cause it to become uniformly lighter (or darker, depending on the particular imaging device). Line operators would act on the image in such a way as to accentuate edges of features. Neighborhood operators take into account the entire neighborhood of a pixel when calculating a new value for the pixel. A standard neighborhood operator is a median filter. The median filter ranks the pixels in the entire surrounding neighborhood (i.e., a 3 by 3 neighborhood) around the central pixel from darkest to brightest, then reassigns the median value to the central pixel. The median filter is used to get rid of extraneous “noise” in images, such as inadvertent bright spots or dark spots caused by electrical pulses during acquisition. Single bright pixels would thus disappear when median filters are used, as would single dark pixels. The sequential application of median filters will cause dark areas in images (i.e., pores in coatings) to become more uniformly dark, whereas light areas (i.e., the matrix in metallic coatings) would become more uniformly light.

Unfortunately, as more and more gray-scale processing operations are used, the image departs further and further from the original, hence the title of this section, “Gray-Image Processing—Getting Further from the Truth.”

As was previously stated, the sole purpose of gray-image processing is to make it easier to detect the features of interest via thresholding.

Thresholding—The Big Controversy. Hopefully, the net result of everything that has happened up to this point will have been successful—specimen preparation for the microscopic analysis, illumination adjustments, focus, image capture, background shading corrections, and gray-scale image processing—to make the selection of features of interest simple and accurate. Unfortunately, however, the thresholding step remains the step of controversy in image analysis. Why is this? Simply because no two operators agree exactly on what to set the limits at to detect the features of interest. Pores, for example, could have a threshold region of gray scales 175 to 255 for one operator, 185 to 255 for another, and 195 to 255 for a third. The controversy ensues because there is no way to prove which one is right, other than the observations and comparisons of several referees. Further, while 175 to 255 may be good for one image, it may not be good for

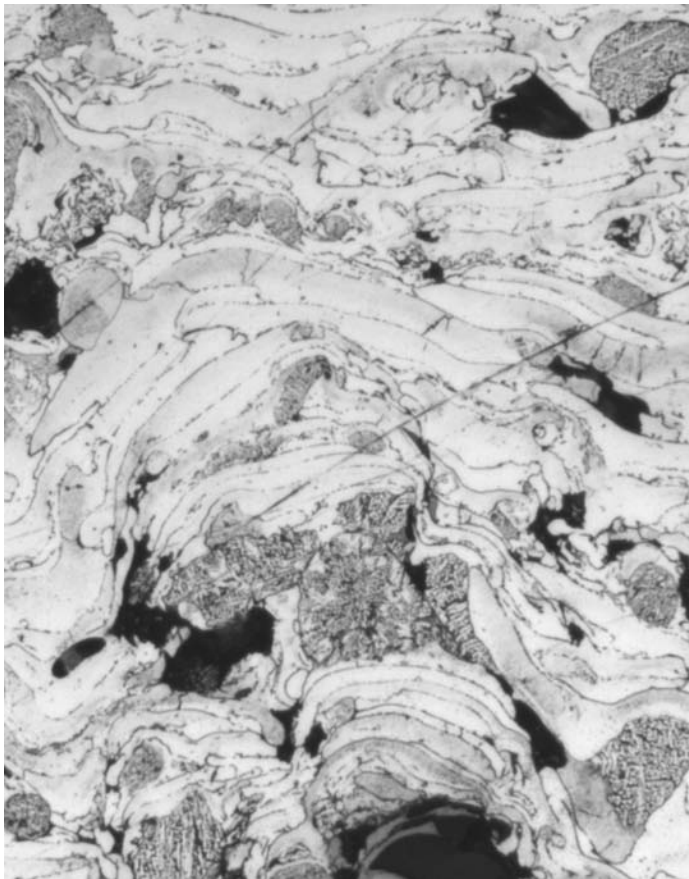


Fig. 56 Eighth of eight different coating types prepared using one standard procedure. Aluminum. Original magnification 200x

another image (depending on the overall image brightness, cumulative reflections, etc.). The following is a discussion that illustrates the thresholding controversy.

Figure 57 shows a ceramic coating and Fig. 58 its associated gray-scale histogram, with 8-bit imaging selected, such that gray scales range from 0 to 255. Note that the histogram peaks are not sharp and that they gradually blend into the surrounding histogram. If one were to start at zero gray scale and accumulate pixels by gray scale, one would start with no pixels and end

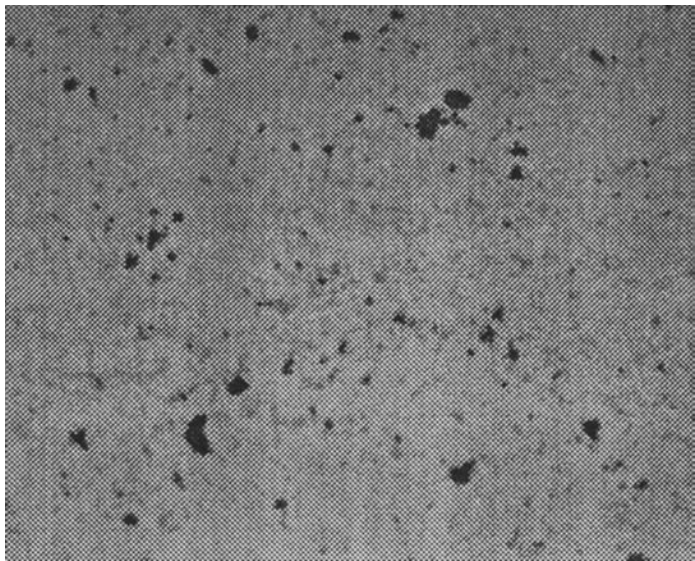


Fig. 57 Image of chrome oxide ceramic coating to be processed for determination of percentage porosity

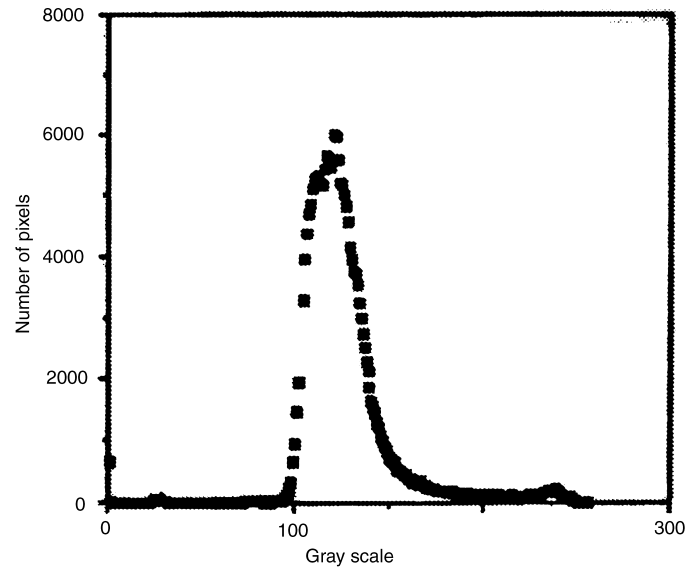


Fig. 58 Gray-scale histogram of chrome oxide coating shown in Fig. 57

up with all of the pixels by the time gray scale 255 was reached, that is, a cumulative number of pixels versus gray-scale histogram, as shown in Fig. 59. Figure 59 represents the histogram more as the computer sees it. For example, if one decides to count the number of pixels from gray scale 175 to 255, the computer would simply select those values above gray scale 174. The cumulative number of pixels versus gray-scale histogram is presented to illustrate a very critical point in image analysis by reflected light microscopy from undulating surfaces with pores, phases in relief,

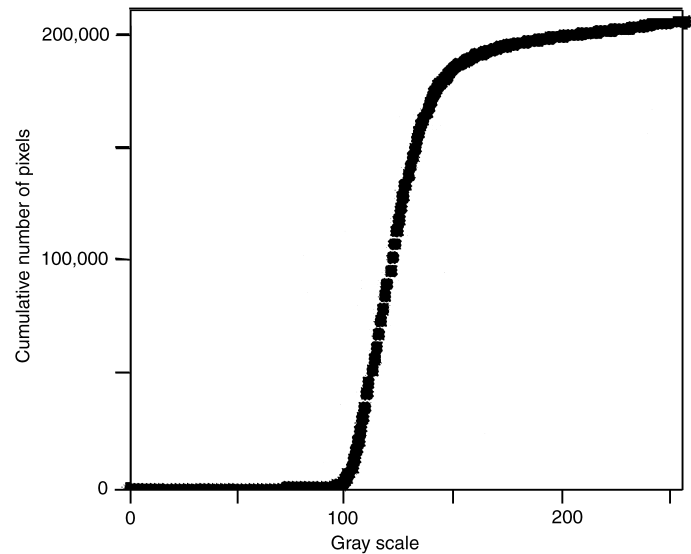


Fig. 59 Cumulative number of pixels versus gray scale for coating shown in Fig. 57 and 58. Accumulation of pixels from zero to gray scale 255, where, for example, the value plotted for gray scale 5 would be the summation of pixels from gray scales 1, 2, 3, 4, and 5

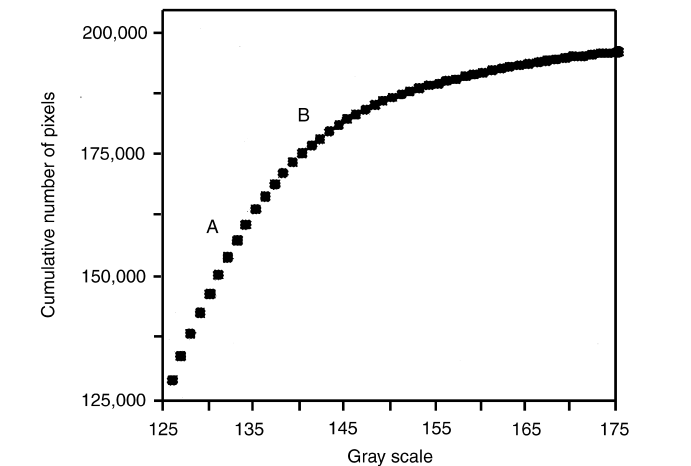


Fig. 60 Zoomed region in upper portion of curve in Fig. 59, showing no bending point of the histogram, suggesting the onset of porosity, and showing that image analysis operator A and B disagree as to where to set the threshold

and so on. Because of the very significant scattering of the incident light, there are no sharp bends or fulcrums in the histogram that represent, for instance, the onset of porosity gray-scale range. Referring to Fig. 60, operator A may select the starting point for selection of porosity as shown, while operator B selects a different point. The very critical point is that both would be equally correct, because there is no fulcrum or bending point in the histogram, so there is no mathematical reason to favor operator A over operator B, and therein lies the subjectivity of image analysis by reflected light microscopy—no one is right mathematically! Operator A may say that the porosity percentage is 5% and operator B, 3%. And again, both would be right, neither would be wrong!

Computer-automated thresholding research and development needs to be conducted in the application of image analysis to thermal sprayed coatings in order to separate the engineer or technician from the threshold decision-making process so that comparative and accurate assessment of coatings attributes can be made. One such computer-automated thresholding routine for thermal sprayed coatings is presented here for consideration/use by the reader.

Referring to Fig. 57 to 59 and the discussion of “Image Analysis versus Reflected Light Microscopy of Metallographically Polished Thermal Sprayed Coatings” earlier in this section, it is seen that the peaks in the histogram shown in Fig. 58 are indeed very broad and gradually blend into the rest of the histogram. This is due to significant light scattering, as previously discussed. In the section “Gray-Image Processing—Getting Further from the Truth,” median filters were discussed. It was stated in that section that sequential median filters applied to pores would make them uniformly darker, while sequential median filters would make the

matrix uniformly lighter. The net effect is seen in Fig. 61: The cumulative number of pixels versus gray-scale histogram bends, with intrapore pixels becoming darker and intramatrix pixels becoming lighter. Postulating that the point of crossing of the two histograms could in fact be the proper threshold to detect pores in coatings, a computer algorithm was developed. The original image was first duplicated, the duplicate image subjected to several median filters, and the cumulative number of pixels versus

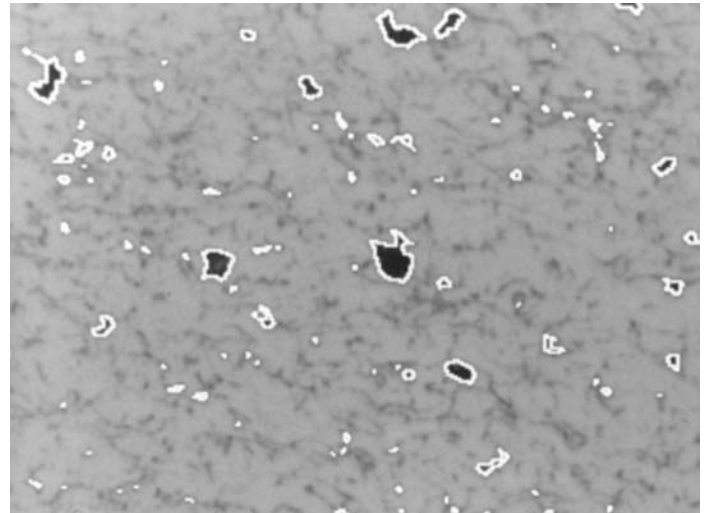


Fig. 62 Application of the histogram crossing procedure to an image of low porosity, showing computer-selected pores

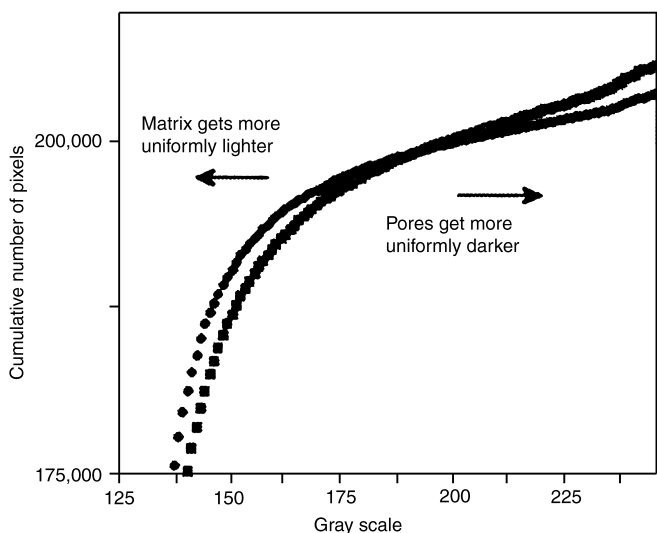


Fig. 61 Bending of the cumulative number of pixels versus gray-scale histogram due to successive application of median filters, causing the pore structures to become more uniformly dark and the coatings to become more uniformly light

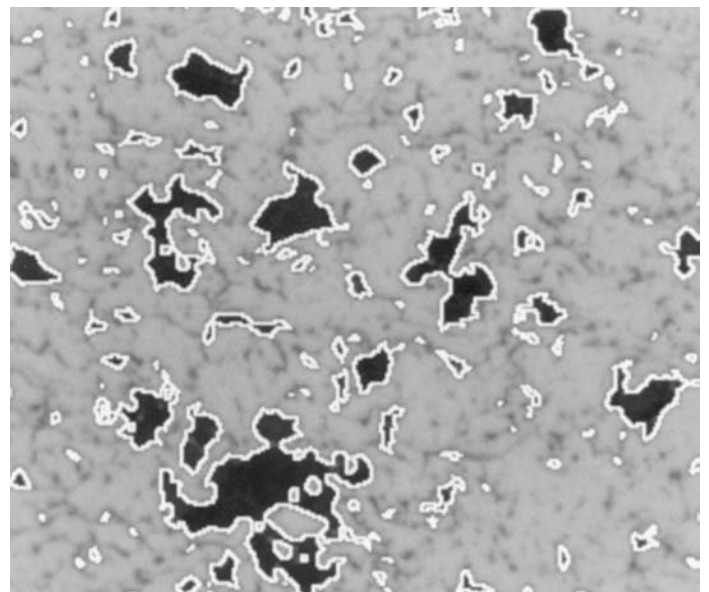


Fig. 63 Application of the histogram crossing procedure to an image of higher porosity, showing computer-selected pores

gray-scale histograms developed. The point of crossing of the two histograms was chosen as the threshold for porosity. As is seen in Fig. 62 and 63, the method works for the selection of pores in areas of both low porosity (Fig. 62) and high porosity (Fig. 63). Figures 64 to 66 show that the procedure is insensitive to illumination: One frame imaged at normal illumination, then very dark (difficult to see on the computer screen), then very bright was nevertheless treated equally by the algorithm. The total porosity area percent between the three images was only 0.09% in a field of view with several percent porosity. The algorithm can be termed the histogram crossing procedure.

Binary-Image Processing. Once the gray-scale range for selection of features of interest has been determined, the selected features are turned one color and the unselected features another (usually black and white for ease of subsequent analysis). It is useful again to consider the image not as an image but as a spreadsheet or matrix with numbers assigned to each cell, but now there are only two numbers, that is, 0 corresponding to black and 255 corresponding to white. Usually, for thermal sprayed coatings, there is no need to perform any binary operations if the information is simple, such as area percentage. However, some coating information, such as a coating surface roughness or a surface profile total line length per unit length (e.g., how much total surface is there in an image digitized at 200 \times ?), does require binary-image processing.

Binary operations usually act on a neighborhood principle. For example, if there were a multitude of long, thin features that were erroneously selected during thresholding, and the features were

only one pixel wide with touching neighbors on either side, the operator could remove them by stipulating that those pixels that had only two neighbors would be eroded, while those having more would not. The aforementioned surface profile line length per unit length assessment could be performed by selecting (thresholding on) the entire coating, leaving the mount material unselected. The selected coating would then be turned into a binary, or black and white, image; the binary image duplicated; the duplicate image eroded by one pixel along its entire periphery, regardless of number of nearest-neighbor pixels; and the resulting duplicate image subtracted from the initial binary image, leaving a one-pixel-wide profile of the surface of the coating. The calculation is then straightforward: simply count the number of pixels and divide by the unit length.

What Data? At this point, the ability of the computer to provide data based on the selected features is limitless and very powerful. For example, if the operator wanted to know the distribution of size of pores as a function of distance from the surface, all of the information is available in the image, and sort routines are simple to the computer. Feature aspect ratio, position, degree of clustering, texture, and so on are readily available once the foregoing operations have been completed.

How Many Frames? A final consideration for image analysis is how many frames to analyze in order to properly represent the feature of interest, such as percentage porosity in a given speci-

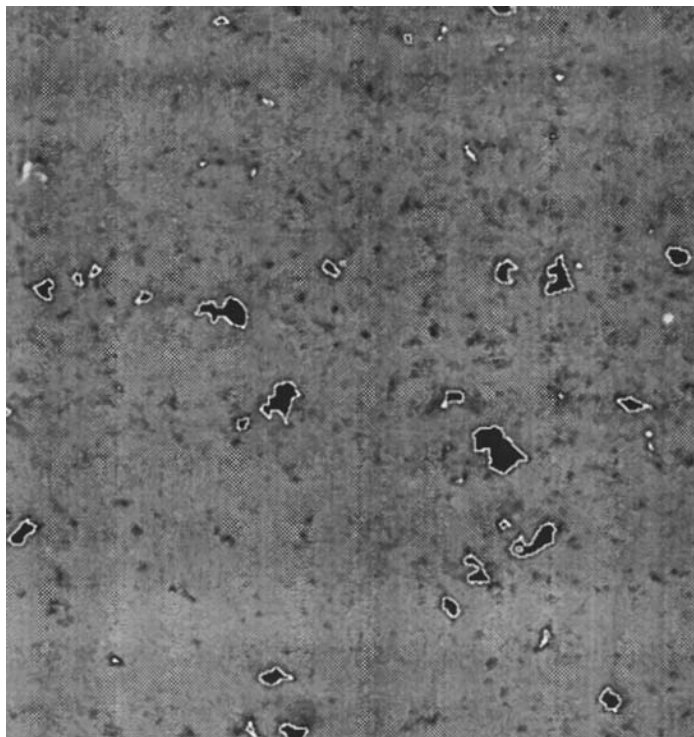


Fig. 64 Application of the histogram crossing procedure to an image at normal illumination. Determined porosity, 3.34%

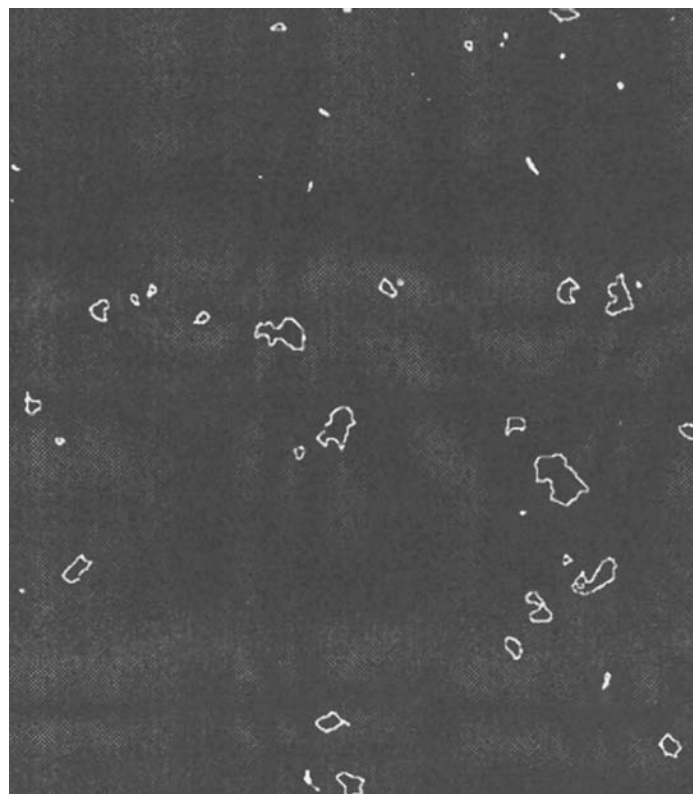


Fig. 65 Application of the histogram crossing procedure to the same area of the coating shown in Fig. 64, without moving the microscope stage and simply turning down the illumination. Determined porosity, 3.25%

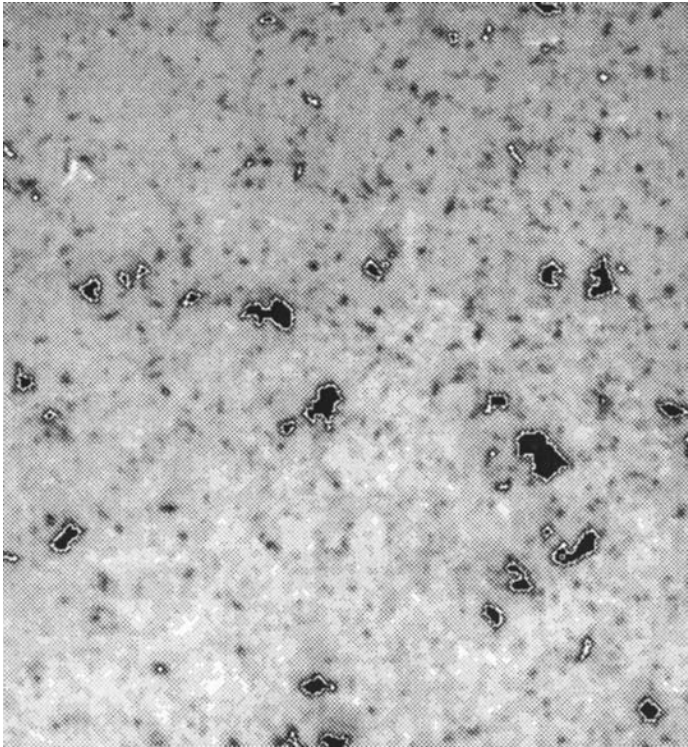


Fig. 66 Application of the histogram crossing procedure to the same area of the coating shown in Fig. 64, without moving the microscope stage and simply turning up the illumination. Determined porosity, 3.33%

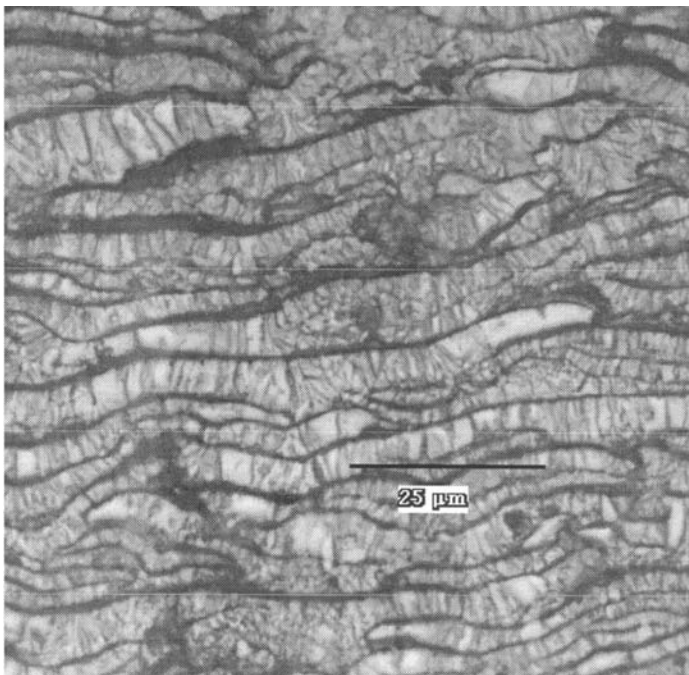


Fig. 67 Pure tungsten coating processed with chemical/mechanical polishing to reveal splat network, porosity, and intrasplat structure. Original magnification 500×

men. A simple answer is to continue until the coefficient of variance from frame to frame of the given attribute ceases to change, with ten frames being considered minimum.

Summary. While many of the problems, such as illumination, shade correction, gray-image processing, and thresholding, have been presented in some detail, each of which will cause its own contribution to error in image attribute measurements, a successful computer-automated thresholding routine has also been presented. It is hoped that the reader will appreciate the need for thorough operator/technician training and detailed procedures development and gain confidence that the extraction of feature-specific information from coatings microstructures via image analysis can be accomplished.

Etchants

Thermal sprayed coatings are typically not etched to reveal microstructural features, because the grain sizes are so small and solidification rates so rapid. The consequent distribution of phases within the coating splats (which is the object of any etching study) is not as dramatic as in wrought, cast, or forged materials. Nevertheless, it may be instructive to ascertain the degree of melting of metallic coatings. Etchants have been used to determine the extent of melting in aluminum-silicon/polyester coatings. Figure 48 shows an aluminum coating after etching to reveal splat morphology and porosity. Figure 67 shows a pure tungsten coating following a chemical/mechanical polish to reveal splat morphology and pore structure.

REFERENCES

1. L.E. Samuels, *Metallographic Polishing by Mechanical Methods*, 4th ed., ASM International, 2003
2. G.F. Vander Voort, *Metallography: Principles and Practice*, McGraw-Hill, 1984 (now published by ASM International)
3. T.O. Mulhearn and L.E. Samuels, *Wear*, Vol 5, 1962, p 478–498
4. *Metallography and Microstructures*, Vol 9, *ASM Handbook*, American Society for Metals, (revised edition to be published in 2004) 1985

SELECTED REFERENCES

- G.A. Blann, Metallographic Specimen Preparation of Thermally Sprayed Coatings for Microstructural Analysis, *Microstructural Science*, Vol 17, ASM International, 1989, p 139–151
- D.B. Fowler, Metallographic Evaluation of Thermally Sprayed Coatings, *ASTM Standardization News*, May 1991, p 54–57
- J.P. Sauer, Metallographic Preparation of Thermal Spray Coatings: Coating Sensitivity and the Effect of Polishing Intangibles, *Thermal Spray, Practical Solutions for Engineering Problems*, C.C. Berndt, Ed., ASM International, 1996, p 777–78

Testing of Coatings

Walter Riggs, TubalCain Company Inc., Ken Couch, Protech Lab Corporation

TESTING OF THERMAL SPRAY COATINGS, as with other engineered materials, involves a wide range of tests for determining mechanical properties, hardness, wear and corrosion resistance, coating bond strength, and high-temperature properties. In addition, both destructive and nondestructive tests have been developed to evaluate residual stresses in thermal spray coatings.

This article describes two of the more commonly used standardized tests for determining the properties of thermal spray coatings: hardness testing and tensile adhesion testing. Emphasis is on the tensile adhesion test. Adhesion is a property of major concern for thermal spray coatings, because it is necessary for the coating to adhere to the substrate throughout the design life of the coating system. The adhesion strength can be influenced by many factors (Ref 1). Some of them are intrinsic, that is, related to the spray variables, such as powder characteristics, spray parameters, substrate preparation, and so forth. The others are extrinsic, which include posttreatment and service conditions.

In addition to hardness and tensile adhesion testing, recently developed electrochemical methods for determining the corrosion performance of thermal spray coatings are described. Additional corrosion tests applicable to thermal spray coatings are summarized in the article "Guide to General Information Sources" in this Handbook.

A brief review of tests for determining the thermomechanical and environmental stability of thermal barrier coatings is also provided. Examples of such tests include thermal fatigue testing and bond coat oxidation tests.

Tests for determining residual stresses in thermal spray coatings are also addressed. Excessively high residual stresses in coatings may cause either coating separation from the substrate or cracking of the coating (Ref 2).

Hardness Testing

Hardness measurement of thermal spray coatings is a straightforward process and is not as difficult or controversial as metallographic practices for these materials (see the article "Metallography and Image Analysis" in this Section of the Handbook). The general nature of the hardness tests is discussed here, with the necessary attention being given to using the tests for the measurement of the hardness of coatings.

The Vickers hardness test is the standard microhardness test employed for thermal spray coatings (Knoop microindentation hardness testing is less widely used). As shown in Fig. 1, the Vickers hardness test uses a square-based diamond pyramid as an indenter, with an included angle between opposite faces of 136° (the test is also sometimes referred to as the diamond pyramid hardness, or DPH, test). The number generated from the test, the Vickers hardness number (HV) or DPH number, is defined as the

load divided by the surface area of the indentation. The HV may be determined from the following equation:

$$HV = [2P \sin(\theta)]/L^2$$

where P is the applied load in kilograms, L is the average length of diagonals in millimeters, and θ is the angle between opposite faces of the diamond (136°).

The units, then, are kg/mm^2 . Loads of various magnitudes are applied, the most common being 300 g. Because the impressions made by the pyramid indenter are geometrically similar no matter what their size, the HV should be independent of load—that is, readings taken at 300, 200, and 100 g applied load on a given specimen should be very similar; however, it has been found that lower loads are often affected more by localized microstructural variations than are higher loads, and as such, lower loads are sometimes not used.

The microhardness test can be sensitive to metallographic polishing. Microhardness values can vary by over 100 units when a specimen is prepared using two different metallographic procedures (refer to the article "Metallography and Image Analysis" in this Handbook for details on polishing).

The Rockwell superficial hardness test (R15N) is the most commonly used macrohardness test for coating hardness acceptance, due in part to its comparative ease of use and minimal surface preparation. It uses the depth of indentation, under a constant load, as a measure of hardness. A minor load is first applied, which

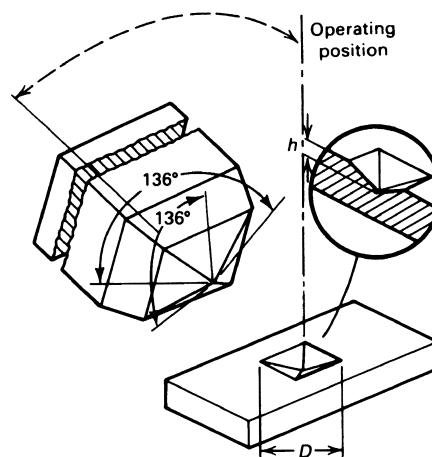


Fig. 1 Schematic of the square-based diamond pyramid indenter used for the Vickers hardness test, and an example of the indentation it produces

tends to set the indenter into the specimen material without rising or sinking. The major load is then applied, and the depth of indentation is automatically recorded on a dial or digital gage in terms of hardness numbers. (For the general Rockwell hardness test, the dial contains 100 divisions, each division representing a penetration of 0.002 mm, or 0.00008 in.) The dial is reversed so that a high hardness, which corresponds to a small penetration, results in a high hardness number.

Thermal spray coatings have somewhat rough surfaces compared to wrought, cast, and forged materials. The roughness needs to be minimized, via abrasion with metallographic grinding papers, or emery papers, prior to hardness testing. A second concern for the superficial hardness testing of coatings is thickness: Testing of thin coatings may yield some substrate influence in addition to the coating response to the load. Generally, it is felt that coating thicknesses in excess of 0.254 mm (0.010 in.) and, in some cases, 0.381 mm (0.015 in.) are necessary. The interested reader should review ASTM E 18, "Standard Test Methods for Rockwell Hardness and Rockwell Superficial Hardness of Metallic Materials," for recommended coating thickness, based on the hardness of the material being tested.

Usually, multiple readings are taken and averaged for hardness measurements. Ten readings should provide adequate assurance that the hardness of a given specimen has been appropriately measured. Obvious coating defects and edges should be avoided during hardness readings.

Because hardness measurement of thermal spray coatings has not been controversial, publications verifying the use of superficial or microhardness test methods on the coatings have not been forthcoming.

Tensile Adhesion Testing

Another very controversial test method used to characterize thermal spray coatings is tensile adhesion testing. The controversy ensues from the very large variance in test results from laboratory to laboratory and, occasionally, from test to test within one laboratory. Because the coatings cannot be directly gripped, other methods must be derived to ascertain their adhesive and cohesive strengths. The most common method employed is that of applying simple epoxy glue to the coating and to grippable bars, curing the epoxy at some elevated temperature to facilitate cross-linking of polymeric chains, then pulling apart the assembly, with applied load and failure location being recorded. The sensitivities of this type of test to its various parameters are numerous:

- Proper alignment is a critical issue. Bending moments that cause nonuniform loading of the coating-substrate system can result in substantial errors.
- If the coating is porous and the adhesive sufficiently fluid, the adhesive may penetrate the coating to the substrate to yield a false result.
- Values obtained in the test depend on the thickness of the adhesive film and its rheology.
- Residual stresses can be set up in the coating-substrate system prior to testing as a consequence of thermal history and thermal expansion mismatches between the coating and the substrate.

- Externally generated vibration, often random, transmitted into the specimen at the time of testing can affect the results adversely.
- Loading rate will have an important effect on results.
- The character and distribution of flaws at which fracture can initiate can have an important effect on results.
- Nonuniformity of coating thickness can cause variance.
- Uncontrolled testing environments, such as temperature and/or humidity, may impact results.
- Undetermined variations in the amount of coating removed in the test are important.
- Complex failure patterns introduce scatter in the resulting data.

Reference 3 discusses the results themselves, including numerical strengths and failure locations. As with any engineering process that generates numbers, the results of tensile adhesion tests need to be analyzed with a variety of methods in order to distinguish true differences between the original tested materials; for example, looking only at the range of results may not reveal mechanistic differences in the failure modes, which, from an engineering design standpoint, is the most critical information. Other mathematical and statistical methods may reveal a distinct difference in the results that can be attributed to fine microstructural attributes.

The common statistical tests of mean, standard deviation, and signal-to-noise ratio should be considered, but the analysis should not stop there. A variety of data transformation methods are available to extract features or relationships that relate the measurements to some property of interest, such as performance. Multiple regression analysis, which develops a best-fit polynomial from a database of measurements of the desired property, is becoming more popular (i.e., relating a combination of factors, such as a coating pore size distribution, location within the coating, shape of pores, fractal dimension, etc., to wear). In Ref 3, Weibull statistics were used to examine results from other researchers who had examined results only using mean and standard deviation. According to the results published in Ref 3, Weibull analysis clearly reveals a bimodal distribution of data, the lower-tensile-strength coatings supposedly failing from a totally different failure mechanism and mode than the higher-strength coatings.

Figure 2 illustrates the standard coating/fixture assembly used for tensile adhesion testing of thermal spray coatings. This figure is adapted from ASTM C 633-01, "Standard Test Method for Adhesion or Cohesion Strength of Thermal Spray Coatings." The coating is applied to a 25 mm (1 in.) diameter cylindrical bar that is threaded for gripping at its end. A somewhat viscous adhesive bonding agent is then applied to the surface of the coating, and the coating/bonding agent is applied downward in a suitable alignment fixture onto another bar, as is discussed subsequently. The entire assembly is then placed into an oven for curing at moderate temperatures (120 to 175 °C, or 250 to 350 °F). Following the cure cycle, the assembly is then pulled apart to determine its strength. The following paragraphs detail those process parameters that may significantly impact test results.

Surface Preparation

A variety of techniques are used to prepare the surfaces for the tensile test. Certainly, the surface must be clean; applicable meth-

ods are those described following the sectioning of coated panels prior to metallographic mounting. Some laboratories prefer to grind the surface of the coating prior to applying the adhesive. While this minimizes the mechanical interlocking, it may tend to activate the surface for chemical reaction with the epoxy. Other laboratories prefer to grit blast the surface of the specimen; this could both activate the surface chemically, as with grinding, and provide for mechanical interlocking of the epoxy with the coating

(recall that mechanical interlocking is responsible for most of the strength of thermal spray coatings).

Adhesive Properties

There are two main categories of adhesives commonly employed for testing the strength of coatings. The first category is a structural adhesive. Structural adhesives are typically one-part,

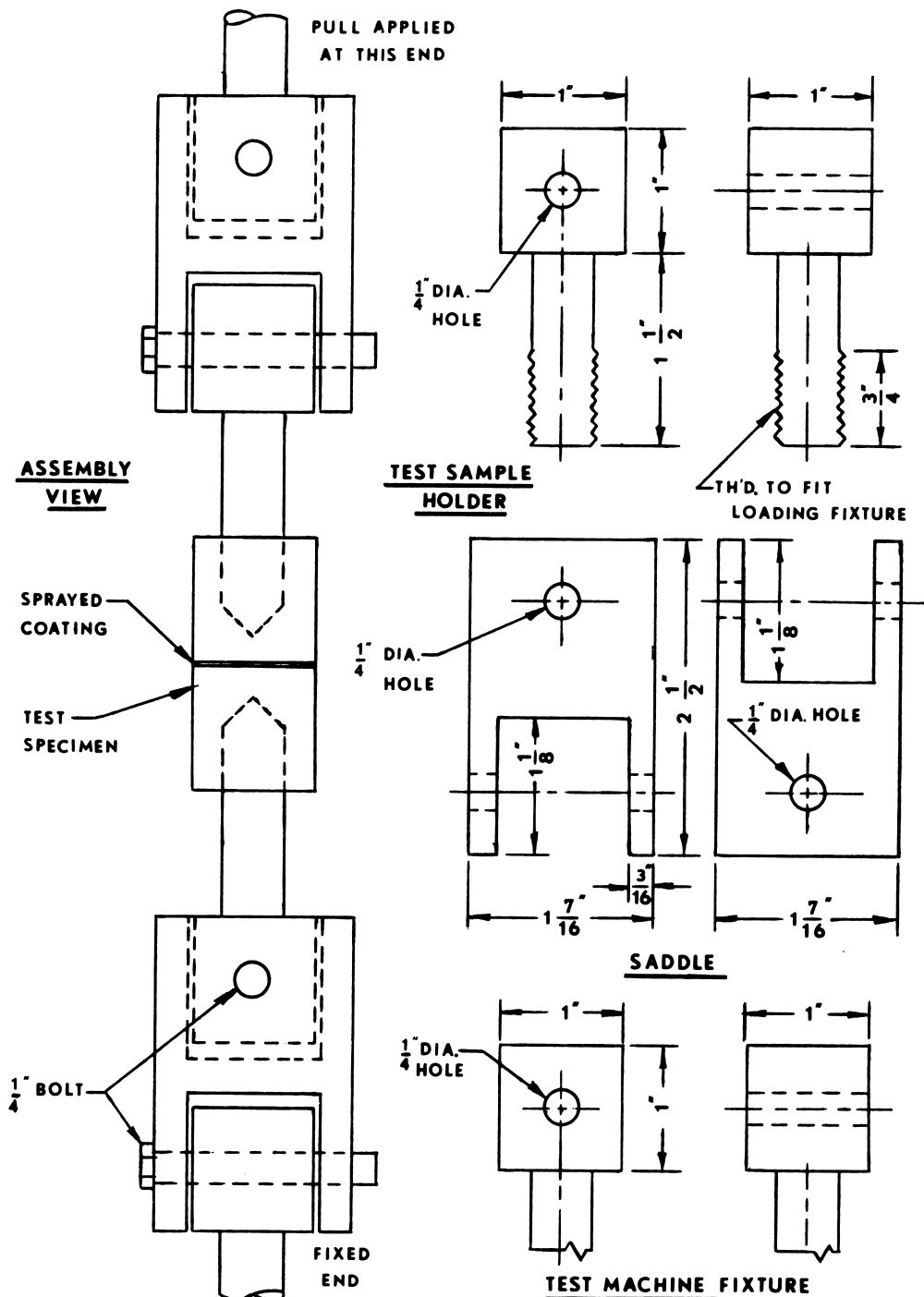


Fig. 2 The coating/fixture assembly used for tensile adhesion testing of thermal spray coatings. Dimensions are in inches (1 in. = 25.4 mm). Source: ASTM C 633

heat activated; two-part, heat activated; or room-temperature cured. Experience has shown that heat-activated adhesives possess significantly higher ultimate tensile strengths (UTS) than those with room-temperature cures. The second category is an elastomeric adhesive; it is also heat activated.

While either type of adhesive, structural or elastomeric, will yield consistent results, there can be significant differences in the average or mean UTS and mode of failure (i.e., failure locus), depending on the coating being tested. To demonstrate the effect of adhesive on tensile testing of thermal spray coatings, a study was performed by one of the authors using plasma sprayed Ni-Cr-Al, a common coating used in the industry. For the test, thirty tensile specimens were coated at the same time, along with a flat panel for microstructural observation. Fifteen of the specimens were bonded with an elastomeric film adhesive and fifteen with a one-part, heat-activated structural adhesive. The cure cycles were typical for each of the two adhesives. The metallographically polished coating, shown in Fig. 3, is typical for this material; porosity is <5%. All thirty specimens were tested in the tensile adhesion test apparatus during one session to ensure equivalent treatment of the samples. The results are presented in Table 1.

The test results demonstrate that, given the same testing parameters (i.e., rate of pull, the same universal fixturing, etc.), the structural adhesive yields higher average tensile strength than the elastomeric adhesive. For this particular coating, the difference in averages was approximately 16%. This difference in averages may be a function of such material properties as ductility and fracture toughness and may be significantly different for materials that exhibit more brittle properties, such as ceramics and cermet coatings. Further, curing conditions of the adhesive were performed under the optimal conditions subsequent to testing. Results could vary depending on cure cycle optimization in a given laboratory.

It is important to understand that the tensile adhesion test for thermal spray coatings is not a simple uniaxial test, as is performed

on wrought, cast, or forged materials, where a machined specimen or prepared-from-bulk material is essentially one continuous material piece and is gripped at its ends and pulled uniaxially. Indeed, the term *elastomeric* suggests a rubber-band-type effect: stretch the rubber band, and as it is pulled, the diameter reduces. The system for testing thermal spray coatings is a multipart system, consisting of two pull bars, a coating, the adhesive, and sometimes a disk with a second adhesive layer. As the bars, which are gripped by the tensile test apparatus, are pulled, a load is imparted to the cured epoxy, which in turn applies a load to the coating (refer to the discussion on testing devices that impart loads and measure responses in the article "Introduction to Testing and Characterization" in this Section of the Handbook).

An area of metallurgical and materials engineering that rightly receives a significant amount of research and development effort is theoretical and applied mechanics. These departments in indus-

Table 1 Results of tensile adhesion tests of thermal spray coatings bonded with elastomeric adhesives and structural adhesives

Sample identification	MPa	psi
Elastomeric adhesive(a)		
EL-1	56.01	8129
EL-2	63.07	9154
EL-3	56.90	8259
EL-4	56.53	8204
EL-5	55.63	8074
EL-6(b)	56.59	8213
EL-7	57.01	8274
EL-8(c)	49.42	7173
EL-9(b)	57.64	8366
EL-10	55.88	8111
EL-11	53.87	7819
EL-12	57.46	8340
EL-13	54.84	7959
EL-14	57.06	8282
EL-15	55.64	8076
Average(d)	56.72	8233
Structural adhesive(e)		
ST-1	64.36	9341
ST-2	65.84	9556
ST-3	65.00	9434
ST-4(b)	67.73	9830
ST-5	65.29	9476
ST-6	65.12	9451
ST-7	64.52	9364
ST-8	64.13	9307
ST-9	65.96	9574
ST-10	65.80	9550
ST-11	64.64	9381
ST-12	64.50	9361
ST-13	66.43	9641
ST-14	65.28	9474
ST-15(b)	68.58	9954
Average	65.54	9513

(a) Spread between high result (8366 psi) and low result (7819 psi) is 547 psi (6.6% of the average). (b) Specimens out of flat by greater than the 0.003 permitted by ASTM C 633. However, each was tested and counted with the group and coincidentally resulted in higher strength than the other specimens in the same group. (c) Sample eliminated as "no test" due to partial adhesive deterioration. (d) Average of 14 results; EL-8 eliminated as noted in footnote (c). (e) Spread between high result (9954 psi) and low result (9307 psi) is 647 psi (6.8% of the average).

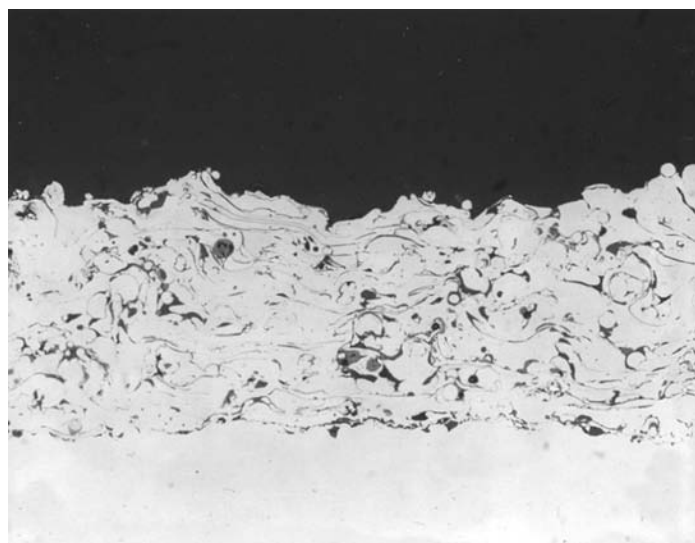


Fig. 3 Nickel-chromium-aluminum coating used to study the tensile adhesion test as listed in Table 1

try and at academic institutions are concerned with static loading, dynamic loading, and the response of materials to various types of loading, including the establishment of triaxial states of stress and consequent strain in materials, elastic/plastic zone creation and nature, crack tip propagation under applied loads (leading, of course, to fracture/failure), and so on. While highly funded industrial and academic consortia have existed for decades and have published a large volume of research material that has helped to better understand component failures, no such effort has occurred in the thermal spray coatings tensile adhesion test area.

It is to be expected that the cured epoxies in tensile adhesion testing of thermal spray coatings do not behave as monolithic slabs. That is, being polymeric in nature, it is anticipated that a triaxial state of stress develops within the epoxy under the applied load. That triaxial state of stress would result in a triaxial state of strain, which, in turn, would impart a load to the coating. It is further anticipated that the magnitude, and perhaps direction (i.e., resultant load vector), of the states of triaxial stress and strain would be different for different epoxies. The elastomeric film epoxy will most probably impart a different load magnitude and direction than the structural adhesive, hence the different averages in the two sets of tests seen in Table 1.

As evidenced from the previous discussion, adhesives can play a very important role in the final result of coating tensile strength. It is important to note that neither single set of results is to be considered the correct strength of the coating. It is imperative to understand the adhesive used for testing the strength of the coating. Further, unless extensive studies have been performed to determine comparative results (and make adjustments to the minimum allowable strengths), it is highly recommended to remain with a comparable adhesive. Development of coating tensile requirements for specifications with one type of epoxy, then using a different type of epoxy, does not take into consideration the foregoing applied mechanics discussions. This becomes especially critical when statistical process control is being used.

Adhesive Curing Cycles

Time and temperature play a critical role in adhesive curing. Ovens used for curing adhesives are very diverse in internal compartment size and mechanical operation. Calibration and oven temperature surveys alone are not sufficient to determine the optimal process parameters for adhesive cure.

All adhesives require activation of some type to create cross-linking of polymeric macromolecules, resulting in curing of the adhesive. For two-part adhesives, this may require certain ratios of resin to hardener, which may be quite critical. Additionally, heat may be required to optimize the cross-linking. One-part adhesives always require a temperature change to activate the curing process. Most metallic and ceramic coatings can easily withstand the oven temperatures for the adhesive cross-linkage. It is preferred to use the one-part adhesives to eliminate potential ratio error factors among operators.

The cross-linking of adhesives depends on a given temperature range to activate the curing process. If too low a temperature is used, incomplete molecular cross-linking will occur, resulting in reduced strength of the cured adhesive. Likewise, too high a temperature can cause decomposition of the adhesive. Therefore, it is

very important to completely understand the curing system for the adhesive being used. When determining adhesive properties, it is good practice to begin with the manufacturer's recommended curing time and temperature. However, the recommended parameters are often not optimized for the particular equipment being used. Test runs should be performed to determine the optimal parameters for individual ovens. These test runs may be performed on non-coated fixtures, because this is a test of adhesive properties only.

While calibration and oven temperature uniformity surveys are essential, they are not sufficient to optimize cure cycles. Internal compartment sizes, power availability, and mass to be cured in the oven are critical. In general, more rapid ramp times to temperature result in optimization of adhesive cures. For this reason, single-phase and 120 V ovens are not recommended, because they tend to heat very slowly, resulting in incomplete cross-linking of the adhesive.

Another issue of concern for the curing process is the cooldown rate subsequent to oven cure. As a general rule, it is advisable to allow the specimens to slow cool in the oven to below 95 °C (200 °F), and preferably overnight when feasible, before removing them from the oven. This will prevent potential thermal shock to the bonded coating system.

Pull Bars and Alignment Apparatus for Bonding of Test Specimens

The topics addressed in this section are:

- Specimens for bonding to the coating surface (pull bars)
- Type of alignment apparatus (pressure or gravity bond)

As stated in ASTM C 633-01, it is preferable to apply the coating to the same (or similar) substrate material (i.e., pull bar) as the part being coated. The pull bar being used to bond the adhesive to the coating surface does not require the same material, because it is not part of the coating system being tested. In fact, for purposes of using the gravity bonding techniques discussed subsequently, it is preferred to use the same material and dimensions for all pull bars to allow for equivalent masses for the top bars. With such equal masses, gravity will produce an equivalent force, allowing for even adhesive thickness. When testing with pull bars coated directly onto the bars and using bars of different materials, it is advisable to retain small discs to apply to the top of the coating system. The extra discs will make up the difference in mass when coatings are applied to lightweight substrate materials. The primary concept is to maintain the same approximate downward pressure on each sample, thus allowing gravity to produce consistent adhesive thickness.

Most specifications permit either gravity- or pressure-bonding techniques. Depending on the type of alignment apparatus employed, there are some basic rules that apply. When using a pressure-bonding fixture (alignment apparatus), the type of adhesive can be critical. For very low-viscosity (thin) adhesives, pressure bonding can enhance the probability of adhesive penetration into the coating. As the viscosity increases (becomes thicker), the probability of penetration decreases. Additionally, the use of too-high pressures can result in insufficient adhesive retained for bonding. Another consideration when using pressure bonding is that during the heating cycle, the torque will change from the orig-

inal pressure applied to the system, due to expansion of the materials. Therefore, if pressure bonding is the method selected, it is highly recommended that spring-loaded fixtures be used to maintain equal pressure throughout the bonding process.

However, it has been found that gravity techniques are adequate when using certain guidelines to minimize the possibility of penetration. Gravity-bonded specimens require a V-block alignment apparatus placed on a set angle, typically 60 to 75°. Such angles allow the entire test system to rest directly against the V-block, providing proper alignment (Fig. 4). All coating pull bars (or, alternatively, the coated sides of disks) are placed in the fixture in a downward position. Gravity will minimize or prevent the possibility of adhesive infiltration into the coating. The use of high-viscosity (thick) adhesives will further minimize the possibility of infiltration due to potential wicking. Further, the use of adhesives containing metallic fillers will create a natural barrier against infiltration of the adhesive. (Note: A good way to assess the probability of infiltration is to observe the excess bead surrounding the specimen in the cured condition. If the excess bead has not run down the pull bar due to natural gravity, the possibility of adhesive infiltration in an upward direction into the coating will be minimal. Metallographic analysis of tested specimens has demonstrated this to be the case on many occasions.)

Coated Specimen Preparation

ASTM C 633-01 requires flatness of the entire coating-substrate system to be within 0.075 mm (0.003 in.) across the entire specimen. This dimension can be easily measured. Additionally, the specification requires that the final specimen coating thickness be at least 0.38 mm (0.015 in.). Many individual company specifications override this requirement, noting that the actual part condition, or some specified thickness, such as 0.25 mm (0.010 in.), is

to be used. Any of the techniques are appropriate, provided that they are consistent. However, in instances where the coating is out of flat by greater than the 0.075 mm (0.003 in.) requirement, great care must be taken when removing the coating prior to bonding to retain a coating thickness that is acceptable to the requirement specified. The condition of the as-received specimen must be noted on any report.

Due to high spots on the surface of the coated specimen, it is recommended that a minimal amount of coating (<0.05 mm, or 0.002 in., if possible) be removed, because there can be weak areas at the top of the coating. Additionally, many parts require some form of finishing following spraying; tensile testing without removing the looser outer layers will not accurately represent the coating strength.

Coating overspray onto the sides of the sample is a great problem with regards to proper alignment. It is necessary to remove all overspray from the sides of the specimen. However, during removal of the excess coating (e.g., by a rotating wire brush), the coating must be maintained in a compressive state to avoid potential damage at the edge of the coating.

There are many companies that diverge from the ASTM specification concerning substrate size. The most common divergence is the use of 6.4 mm (0.250 in.) thick tensile discs, coated on one of the flat surfaces. This is an acceptable method for coated specimens, provided that flatness dimensions are maintained. However, it should be noted that this method requires two bond joints, which increases the possibility of misalignment. Extra care should be taken to match the diameters of the pull bars to the specimen.

Test Conditions

Prior to testing, the bead of excess adhesive around the bond joint must be removed. Great care must be taken to perform this process slowly, while keeping the coating edge in a compressive state to protect it from damage.

It is a typical practice to test the samples at 0.013 to 0.021 mm/s (0.03 to 0.05 in./min) crosshead travel speed. This rate is common throughout the industry and should always be maintained, unless a specific deviation is indicated.

The use of self-aligning fixtures is essential during the testing process. Due to the nature of the thermal spray coating sample preparation (bonding two or three individual pieces together), there is an inherent possibility for slight misalignment. Universal grips similar to those in the ASTM specification (Fig. 2) will work very well to compensate for slight misalignment of the bonded samples.

Understanding of Failure Modes

Care should be taken when evaluating failure modes. It is highly recommended to use a stereomicroscope to evaluate fractures. The ASTM specification details the following different types of failures:

- Cohesive coating failure (within the coating) and approximate location
- Adhesive coating failure (at the coating-substrate interface)
- Cohesive failure of the adhesive itself (within the adhesive)

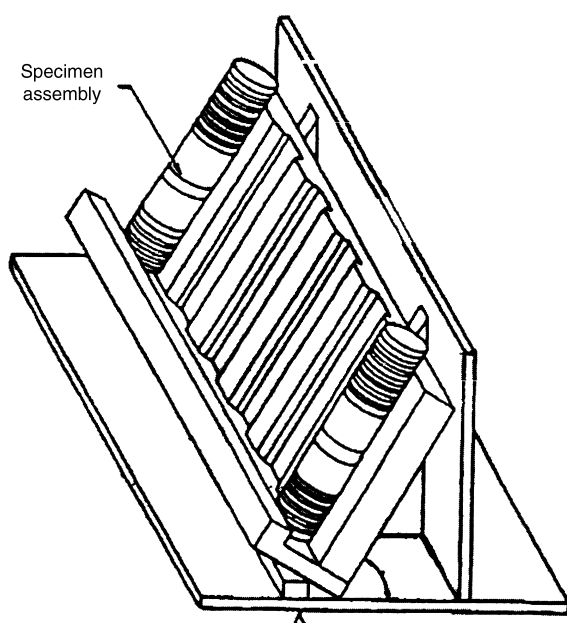


Fig. 4 Standard V-block set at an incline for gravity bonding of tensile specimens

- Adhesive failure of the adhesive itself (at the adhesive-coating interface)
- Combinations of the aforementioned

When dual-coating systems are tested, any of the previously mentioned failure locations can occur. However, determination of which coating location should be provided. Additionally, for dual systems, there is one additional type of failure (adhesive failure between coatings).

Summary of Recommended Procedures for Tensile Adhesion Testing

The recommended procedure for tensile adhesion testing of thermal spray coatings, then, is as follows: (1) adequately clean the surfaces to be tested; (2) remove loosely bonded outer coating layers, and activate the surfaces by lightly grinding; (3) apply the selected epoxy evenly and consistently (same mass); (4) align the assembly in a suitable alignment apparatus with coated side down; (5) cure for adequate time at temperature (having optimized the particular oven for ramp up, mass, etc.); (6) cool slowly to avoid introduction of cracks; (7) if flash has to be removed, do so without the introduction of notches into the epoxy/coating joint, keeping the coating always in compression; (8) use self-aligning grips; (9) use a crosshead travel speed between 0.013 and 0.021 mm/s (0.03 and 0.05 in./min); and (10) evaluate failure locus in accordance with ASTM C 633-01.

Corrosion Testing and Monitoring

Another testing methodology to be considered has only recently been applied to thermal spray coatings (the last 5 to 12 years) but is gaining in popularity, certainly among academic institutions and research laboratories. Two distinctly different methods are used: direct current (dc) electrochemical techniques and alternating current (ac) impedance. The dc methodologies are only briefly discussed, because the ac methods are considered somewhat more capable and are thus receiving more attention.

Direct Current Methods. The primary dc method in use is potentiodynamic polarization. Essentially, a sample, such as a coating, is exposed to an electrolyte (in this case, a liquid), and the system is allowed to equilibrate (reach a state of near-electrochemical equilibrium). The equilibrium corroding potential (E_{corr}) with respect to a reference electrode is measured. The sample is then driven electrically to a more negative potential of some 50 to 100 mV and slowly allowed to return to the equilibrium potential, then driven positively, often to a potential of several hundred millivolts with respect to E_{corr} . The test determines the susceptibility of the material to corrode in the given electrolytic environment. Figure 5 shows a typical potentiodynamic scan for a stainless steel that has passivated, or become resistant to corrosion, at potential values slightly above E_{corr} . At much higher values, the material again begins to corrode. Tests such as this may be recommended to determine the corrosion protection conferred by a thin coating.

Alternating Current Methods. Although the authors are familiar with many sources in the literature regarding the use of ac methods to investigate painted and coated metals, much of this

section has been derived from Ref 4. The discussions on ac methods are dispersed throughout Ref 4; it will here be attempted to gather and simplify the presentation, as well as to embellish from numerous other articles available in the open literature and from the authors' own experience.

The primary consideration for the application of ac methods to coatings is that in the presence of an electrolyte, the corroding system can be thought to have a double-layer capacitance, as seen in Fig. 6. The equivalent electrical circuit is shown in Fig. 7 (a resis-

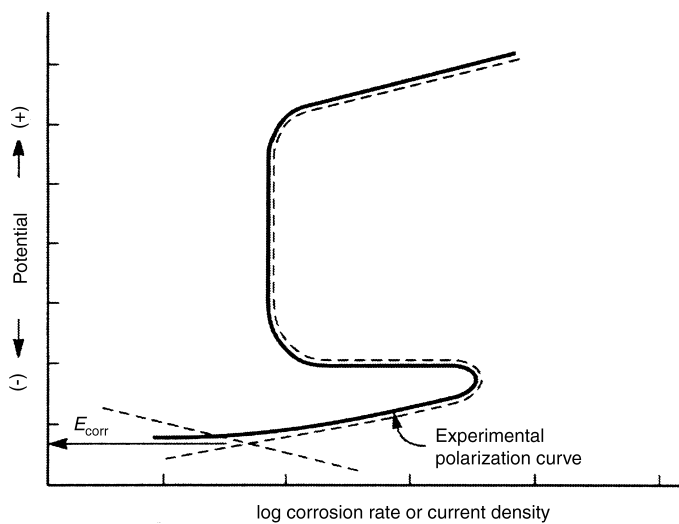


Fig. 5 Electrochemical potentiodynamic scan of passivating stainless steel. E_{corr} , corrosion potential

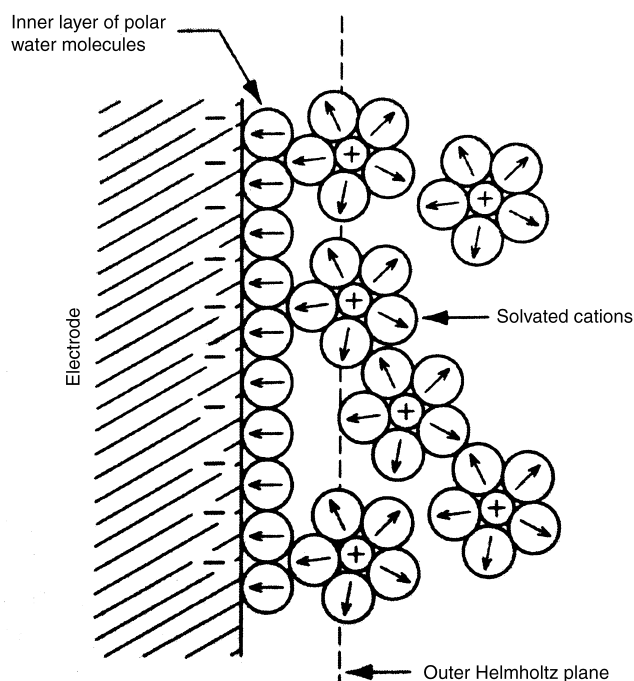


Fig. 6 Corroding interface of a material immersed in an electrolyte showing the establishment of a double-layer capacitance

tive/capacitive or RC, parallel circuit) in conjunction with an electrochemical impedance spectroscopy (EIS) diagram, which is discussed in the following paragraphs.

Simple Oscilloscope Measurements. When an alternating voltage is applied to a corroding system (or a RC circuit, as shown in Fig. 7), the current response will lag, as shown in Fig. 8. Traces, such as seen in Fig. 8, are detected on the oscilloscope by selecting the "A and B" signal setting. To determine the exact phase shift, the oscilloscope is turned to "A versus B;" a pattern such as that shown in Fig. 9 appears. Figure 9 is known as a Lissajous pattern. Conveniently, $A/B = \sin \theta$. Another oscilloscope measurement needs to be taken. Figure 10 shows the determination of the time constant, τ , which is the time for current to decay to 0.37 of its original value, or for a charge across the capacitor to increase to 0.63 of its final value.

Having made these measurements, the following equations will reveal the values of resistance and capacitance:

$$\text{Impedance, } Z = V/I$$

$$\text{Resistance, } R = Z \cos \theta$$

$$1/\omega C = Z \sin \theta$$

Thus, knowing V , I , θ , and ω (the operator presets the frequency), the values of R and C are determined.

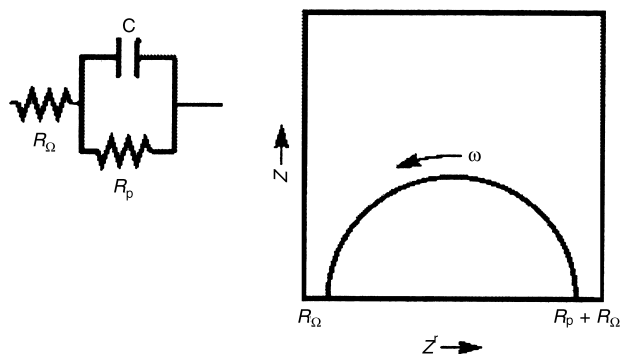


Fig. 7 Equivalent circuit of the corroding system shown in Fig. 6 with an electrochemical impedance spectroscopy Nyquist plot

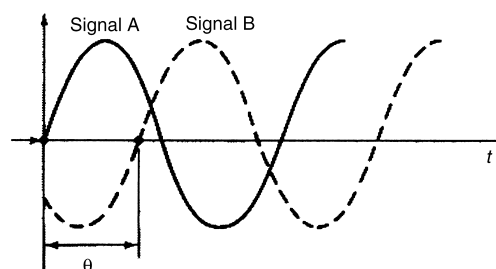


Fig. 8 Phase shift between voltage (signal A) and current (signal B) wave forms in a resistive/capacitive circuit

As a check, because τ has been determined, the following equations can be used:

$$\tau = RC$$

$$\theta = 1/\omega RC$$

$$I(t) = (V_0/R)e^{-t/RC}$$

If these measurements can be made with simple oscilloscopes and "dummy" RC circuits, then why bother with sophisticated (and very expensive) computer-driven ac impedance (or EIS) devices? Precisely because the cumulative errors from the oscilloscope measurements can accumulate—in one of the authors' experience, errors of 15% from calculations based on oscilloscope measurements were considered normal.

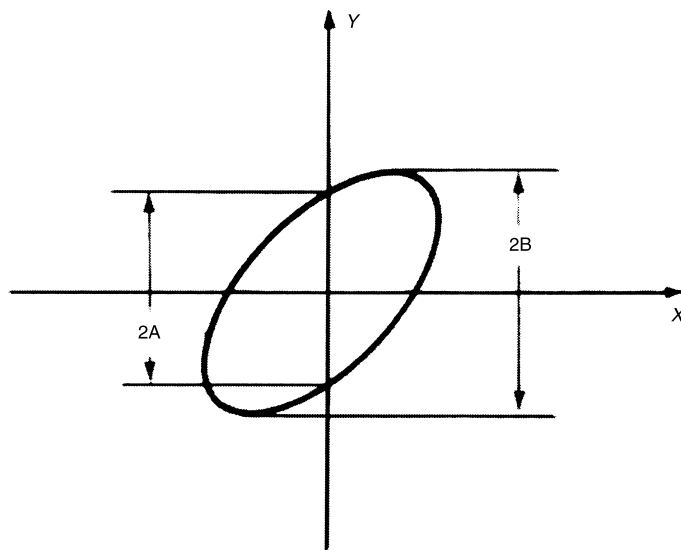


Fig. 9 Lissajous pattern of voltage versus current from a resistive/capacitive circuit

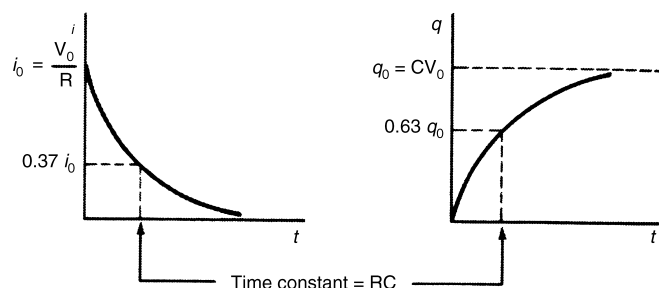


Fig. 10 Time constant of a resistive/capacitive (RC) circuit

For the RC circuit shown in Fig. 7, if the system is driven from extremely low frequencies to very high frequencies (i.e., 10^{-2} to 10^5 Hz), a plot such as that shown in Fig. 11 results. It is called a Nyquist plot of the real versus the imaginary components of impedance, which is beyond the scope of this discussion. A sometimes more useful plot is the Bode log impedance versus log frequency, which does not require an understanding of real and imaginary components. However, the Nyquist is perhaps easier to explain in terms of the information for this discussion. Figure 11 shows a semicircle (of a dummy RC cell), with values of R_ϕ , $R_\phi + R_p$, and ω_{\max} . The semicircle represents literally thousands of data points and is therefore exact, by comparison to the 15% error for manual oscilloscope measurements, as mentioned earlier.

The value R_ϕ represents the resistance of the solution or electrolyte, R_p is the resistance of the material (in this case the coating), and C is the capacitance of the coating. The data point to the far left on the semicircle is R_ϕ . It is recorded at the highest frequencies, near 10^5 Hz. At very high frequencies, capacitors act as conductors; there is no impediment or impedance to the flow of current, and the RC circuit shown in Fig. 7 will simply become an “R” circuit—all of the current will flow through the capacitor, bypassing the resistance R_p . The second data point, to the far right on the semicircle, is $R_\phi + R_p$. At very low frequencies (i.e., 10^{-2} Hz, or 1 cycle/100 s), the capacitor portion of the corroding system acts like a capacitor—it effectively blocks the current. In this case, the current is forced to flow through both the R_ϕ and R_p resistances, hence the value $R_\phi + R_p$. R_ϕ includes the summation of resistances from the electrolyte, surface films, circuit leads, and so on, while R_p is termed the polarization resistance of the coating.

It has been suggested that failed or failing coatings can be described by the equivalent electrical circuit shown in Fig. 12. Such a circuit may be representative of a coating with significant porosity, especially if the pores are interconnected to the base metal.

In one of the authors' laboratories, it was found that a yttria-stabilized zirconia (YSZ) coating that had been sprayed at low amperage and long spray distances and that metallographically showed a high percentage of large porosity indeed did show a double-semicircle EIS graph. This suggests two different corrosion mechanisms (i.e., coating and pores) at different excitation frequencies. Figure 13 shows the results. The coating used for the double-semicircle plot was termed “ugly,” while the ones for the single-semicircle plots were termed “good.” The good coatings evidenced low porosity metallographically.

Testing of Thermal Barrier Coatings (Ref 6)

Thermal barrier coatings (TBCs) are applied to metallic components of advanced heat engines to reduce the metal temperature, increase the environmental resistance and life of the component, and, in some cases, reduce noxious exhaust emissions. This thin coating usually consists of a metallic bond coat applied to the metal component, followed by a layer of magnesia- or yttria-stabilized zirconia. If the TBC is on nickel- or cobalt-base superalloy components intended for high-temperature operation, it is advisable to heat treat the TBC to achieve bond coat densification

and diffusion bonding to the substrate. The selection of the TBC system involves many considerations, principally, knowledge of the intended service conditions, temperature reduction expectations, component life estimates, cost, and, in some cases, the external surface finish requirements. It is implicit that a successful TBC will also survive the rigors of service without spalling or eroding. This section discusses some of the tests for determining the thermomechanical and environmental stability of TBCs. Additional information can be found in Ref 6.

Thermomechanical Stability

The TBC system is expected to survive the thermal shock of rapid cycle heating and cooling, as experienced in gas turbine or diesel engines, without spallation. Spallation is the result of the thermal stress pattern created when the thermal cycle repeatedly exceeds the stress for crack growth within the zirconia layer. This results in a time-dependent extension of preexisting cracks within

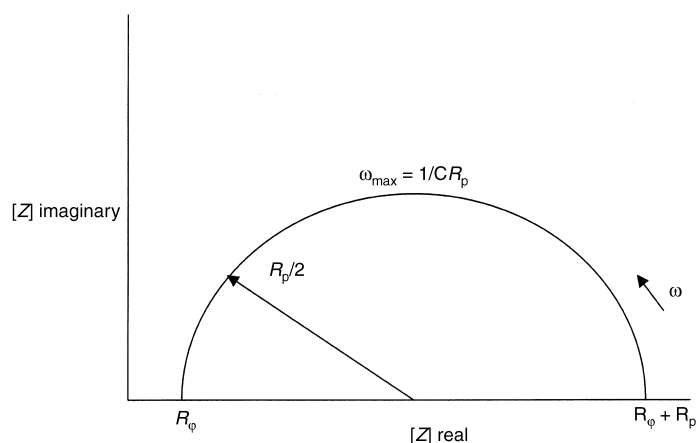


Fig. 11 Standard Nyquist plot of a resistive/capacitive circuit. Z , impedance; R_ϕ , resistance of the electrolyte; R_p , resistance of the coating; C , capacitance of the coating

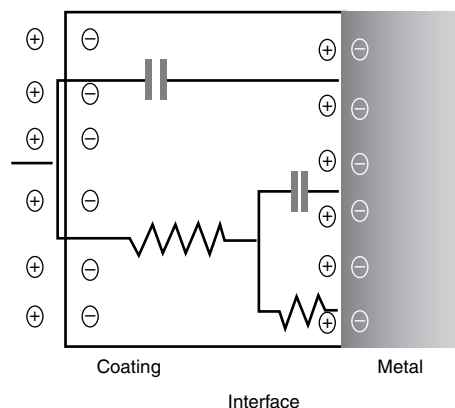


Fig. 12 Complex equivalent electrical circuit of a defective coating in a corroding system. Source: Ref 5

the plane of the coating until critical linkup and spallation. In many tests, the location of the spall line is in the zirconia layer but near the bond coat interface.

Two strategies to improve the resistance to in-plane crack growth in thermal sprayed zirconias are to use a low-density coating, with the pores acting to blunt growing cracks, or to increase the cohesive strength between splats, which requires high-density coating conditions.

Thermal Fatigue Testing. Laboratory testing has been used, particularly in the coating development stage, to thermally cycle a TBC specimen, followed by posttest microscopic examination for spallation-type cracks. The thermal cycle for evaluation of thermal shock resistance employs rapid heating and cooling rates, using direct impingement flames or heating jets on the oxide face of the specimen, with little hold time at the maximum temperature. This test principally challenges the oxide layer, because the bond coat remains at relatively low temperatures due to the insulating nature of the zirconia layer and the short time at high temperatures.

Figure 14 illustrates a cycle that simulates the thermal shock of first-stage gas turbine outer airseals with a zirconia layer 1.27 mm (0.050 in.) thick. The test sample is a 25 mm (1 in.) diameter button, 3.2 mm (0.125 in.) thick, made from the substrate alloy of interest. After the button is coated with the TBC system, the edges are ground and polished to produce a square edge to allow examination of the coating layers.

The test rig consists of an oxygen-propane or propylene burner, a fixture arm to hold the button sample, a stepping motor system to advance quickly between stations and to hold for prescribed times in the heating and cooling positions, and a means to record

at least the front oxide face temperature during the heating cycle. Two-color infrared optical pyrometers are widely used for the latter task, with the output connected to a chart recorder or computer. One word of caution concerning the temperature measured: If the zirconia coating is not an ideal gray body, that is, the emissivity is not the same at the two wavelengths used by the pyrometer, the reading may be in error. This issue is under study at several test facilities, and it could affect burner rig tests as well. If the samples being tested are all the same material, such as plasma sprayed 8% YSZ coatings of common thickness, the measured temperatures should be relatively correct.

In one test configuration, the heating cycle is 20 s, followed by a 20 s air blast and two 20 s periods of natural convection cooling. The burner-to-specimen standoff, burner size, and gas flows are set to heat the oxide face of a standard specimen rapidly to 1400 °C (2550 °F) in the first 20 s. The air blast then drops the front face temperature to approximately 815 °C (1500 °F), and it finally reaches approximately 454 °C (850 °F) after 40 s of natural cooling. After the cycle is repeated 2000 times, the edge of the coated button is examined at 10 to 30× for evidence of separation-type cracking in the zirconia layer. The specimen edge should also be inspected before the test, with few to no starting cracks expected.

The posttest cracking of good TBC systems having zirconia thicknesses of approximately 1.27 mm (0.050 in.) should be less than 15% of the circumference and typically much less. If a tested specimen is mounted in cross section and serially polished, it will be seen that the crack is indeed at the edge of the coating and extends inward toward the center of the button. The cracks will continue to extend around the circumference and grow inward

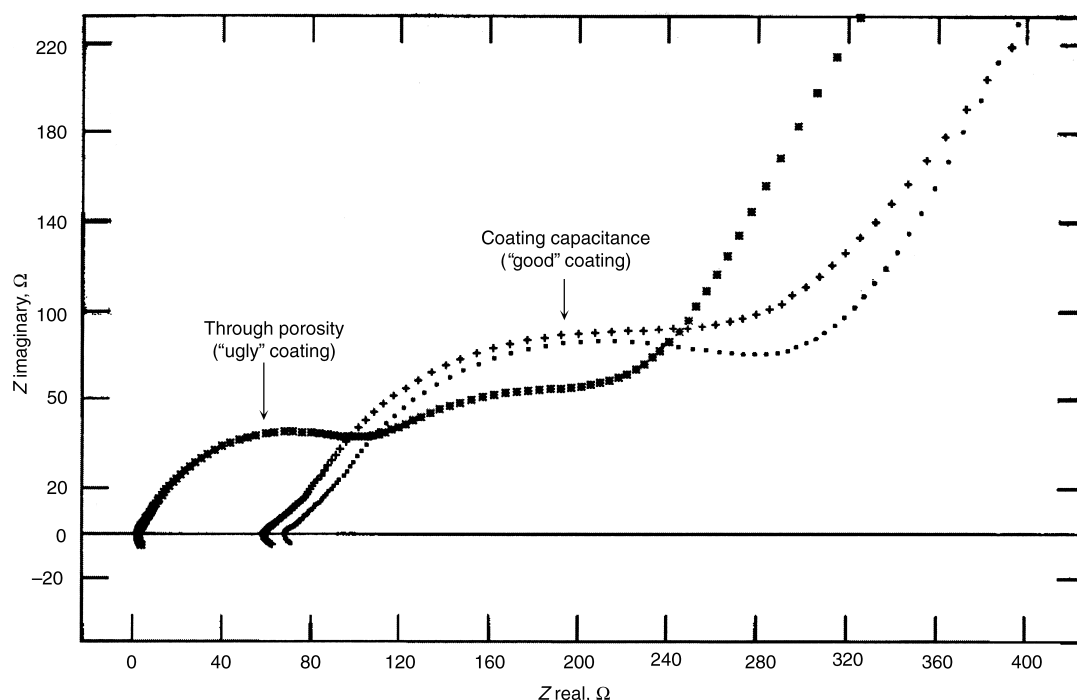


Fig. 13 Nyquist plot of "good" and "ugly" yttria-stabilized zirconia coatings showing the onset of a second semi-circle for high-porosity coatings. Z , impedance

until eventual spallation, for the case of a TBC with poor thermal fatigue resistance. In good TBC systems, additional sets of 2000 cycles will show little crack growth, if any, and typically at a lower rate than in the first test period.

Environmental Stability

A TBC must also resist environmental degradation. This may occur as oxidation of the bond coat, phase transformation stress deterioration of the zirconia layer, or chemical destabilization of the oxide layer. The severity of these effects depends on the oxidation resistance of the bond coat, the initial crystalline phase mixture of the zirconia coating, the service temperature, and the environmental impurities present. Emphasis in this section is on bond coat oxidation tests. Zirconium oxide phase stability and chemical effects on phase stability are described in Ref 6.

Bond Coat Oxidation. To test the oxidation resistance of the bond coat and its effect on the spallation of the TBC, a thermal cycle needs to combine extended soak time at high temperatures, with periodic heating and cooling cycling to induce thermal stress. One such cycle used to test TBCs for aircraft gas turbine applications is a simple tube furnace that operates at a constant temperature of 1121 °C (2050 °F), with the sample set pneumatically moved in and out of the hot zone. The TBC samples rest on an alumina firebrick boat, with dense alumina pushrods made from thermocouple insulator ceramics, for an all-oxide hot-zone support system. The samples heat to furnace temperature in approximately 5 min, soak for 50 min, and then are quickly pulled out for a 5 min cooling period. At the end of every 25 cycles, the samples are held out and visually examined at room temperature. After 200 cycles, the samples are inspected for spallation or edge cracking, then are mounted in plastic for evaluation in polished cross section. There, the extent of bond coat oxidation and interface cracking can be measured. The test temperature is as much as 110 °C (200 °F) higher than expected for the component in service and is thus an accelerated test for oxidation of the bond coat. To make this comparison test valid, care should be taken that the furnace tempera-

ture profile is uniform and covers the full zone occupied by the TBC samples.

A cycle for testing coatings for diesel applications can be similarly designed by setting the soak temperature approximately 110 °C (200 °F) higher than the measured steady-state temperature of the substrate metal of the component in question.

Residual-Stress Determination in Thermal Spray Coatings

In most combinations of coating material and application process, there is a temperature difference between the coating and the substrate caused by heating the coating and having the substrate at a different temperature than the applied coating. As the two materials cool to room temperature, they shrink by different amounts, causing residual stresses in the coating and the substrate. Other factors, such as particle impact velocity and splat cooling rate, influence coating residual stresses. The result is that there are residual stresses in the coating and substrate, and these stresses can be tensile or compressive. Excessive residual stresses can have a deleterious effect on coating performance, fatigue life, and bond strength.

The most popular methods of residual-stress measurement in thermal spray coatings can be roughly divided into three groups (Ref 7):

- Material removal methods, which include the hole-drilling methods and the coating layer removal method
- Substrate curvature or deflection measurement
- X-ray diffraction method

The hole-drilling method for measuring residual stresses involves drilling a shallow hole in the test specimen to a depth approximately equal to the hole diameter (Ref 2). Typical hole diameters range from 0.8 to 5.0 mm (0.03 to 0.20 in.). The creation of the hole redistributes the stresses in the material surrounding the hole. A specially designed three-element strain-gage rosette, such as that shown in Fig. 15, measures the associated partial strain relief. The in-plane residual stresses that originally existed at the hole location can then be calculated from the measured strain reliefs using the method described in ASTM E 837, "Measurements of Residual Stresses by Hole-Drilling Strain-Gage Method." The ASTM standard also gives details of practical drilling procedures.

The modified layer removal method for evaluating residual stresses involves spraying a 25.4 by 25.4 by 6.35 mm (1 by 1 by 1/4 in.) specimen and carrying out strain measurements before, during, and after the coating has been removed by polishing. Details on the test specimen, the equipment needed, the procedure for applying strain gages, the procedure for removing coating layers, and the method for interpreting the data to evaluate residual stresses can be found in Ref 8.

Substrate Curvature Measurement. Another very common method of stress determination is based on optical or mechanical measurement of substrate curvature, induced by stress in the coating (Ref 7). This method, in its simplest form, can determine only

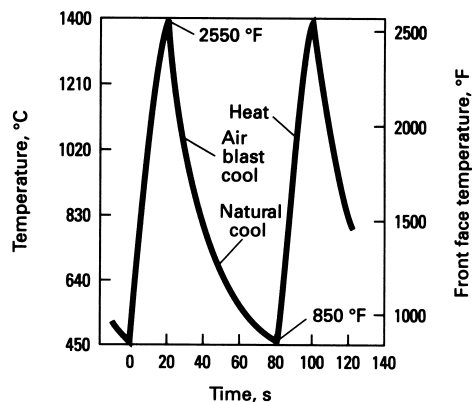


Fig. 14 Schematic illustration of oxide surface temperature cycle used for thermal fatigue testing of ZrO_2 -coated test buttons 1.27 mm (0.050 in.) thick, simulating a first-stage gas turbine outer airseal application. Source: Ref 6

average stress value in the coating and has severe limitations on specimen shape and dimensions. More information about stress distribution can be obtained if dimensional changes of both the coating and the substrate are measured after detachment. The analysis becomes quite complex if thicker coatings or multilayers are studied, and especially if plastic deformation of the substrate occurs. Among the most important advantages of this approach are relatively simple experimental setup, direct determination of coating stress (most of the other methods measure strain), ability to observe stress changes in situ during deposition, and separation of the contributions of quenching and thermal mismatch stresses.

X-Ray Diffraction (XRD) Stress Measurement Method. The diffraction method of stress determination is based on the measurement of changes in crystal plane spacing in different directions with respect to the specimen surface, which exhibit themselves as shifts in angular positions of respective diffraction peaks (Ref 7). From this strain determination, stress can be calculated with the use of appropriate elastic constants.

Among the various methods for residual-stress measurements, XRD holds a specific position due to the following characteristics (Ref 7):

- It is nondestructive; therefore, the same specimens can be used for other investigations, or repeated measurements can be performed on specimens undergoing various treatments, fatigue, and so forth.
- It is phase distinctive, that is, capable of stress determination in each phase.
- It has moderate restrictions on specimen dimensions and shape; therefore, it can be used for measurements on various specimen sizes and components.
- Due to the low penetration of x-rays, the investigated volume is limited to a thin surface layer (on the order of tens of micrometers) comprising a few splat thicknesses. This feature represents certain advantages and limitations at the same time.

More detailed information on the XRD stress measurement method can be found in Ref 2, 7, and 9.

Research in Nondestructive Testing

Nondestructive testing (NDT) techniques for evaluating and characterizing coatings have been extensively demanded by the thermal spray community; nonetheless, few results have been produced in practice due to difficulties in analyzing the complex structure of thermal spray coatings (Ref 7). Perhaps the NDT technique that is most important to date, both technically and commercially, is XRD for residual-stress determination, as discussed earlier in this article. Several other NDT methods are, however, being considered for thermal spray coating analysis. Some of these are briefly summarized as follows. Additional information on NDT techniques can be found in the article “Postcoating Operations” in this Handbook.

Acoustic emissions are stress waves produced by sudden movement in stressed materials. The classic sources of acoustic emissions are defect-related deformation processes, such as crack growth and plastic deformation. The process of generation and detection is illustrated in Fig. 16. Sudden movement at the source produces a stress wave, which radiates out into the structure and excites a sensitive piezoelectric transducer. As the stress in the material is raised, many of these emissions are generated. The sig-

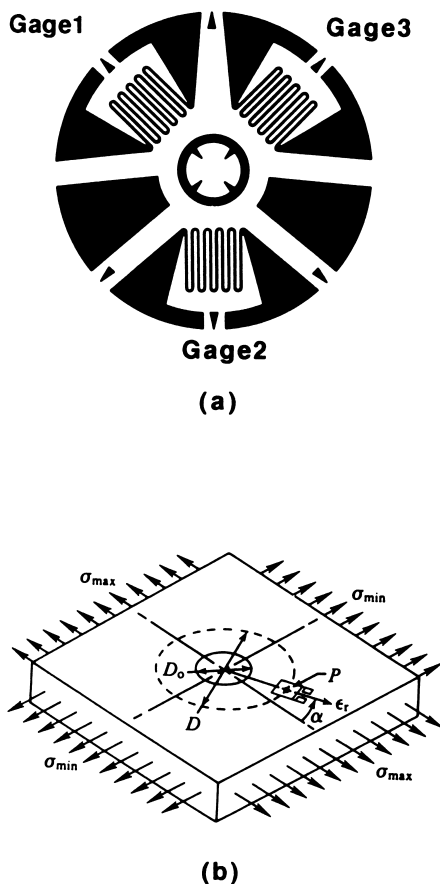


Fig. 15 Hole-drilling method for measuring residual stresses. (a) Typical three-element strain-gage rosette. (b) In-plane strain components caused by release of residual stresses through the introduction of a hole. Source: ASTM E 837-92

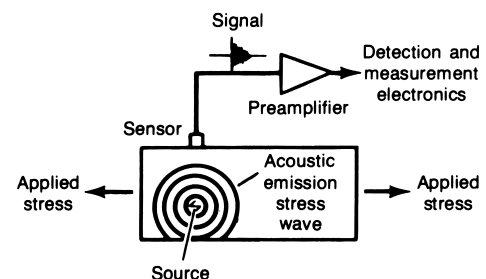


Fig. 16 Basic principle of the acoustic emission method

nals from one or more sensors are amplified and measured to produce data for display and interpretation.

The source of the acoustic emission energy is the elastic stress field in the material. Without stress, there is no emission. Therefore, an acoustic emission (AE) inspection is usually carried out during a controlled loading of the material. For thermal spray coatings, the AE technique has been used for the determination of the onset of cracking during thermal fatigue testing of TBCs (Ref 10, 11) and four-point bend testing of various coating materials (Ref 12, 13).

The laser-ultrasonics technique has been used to determine the elastic constants (elastic modulus and Poisson's ratio) of thermal spray coatings (Ref 14). Knowledge of these properties is very important when seeking to understand and/or model the mechanical behavior or to develop life prediction models of thermal spray coatings employed in various applications (e.g., wear, fatigue, and high-temperature). The laser-ultrasonics technique uses surface acoustic waves or Rayleigh waves to obtain information about a material. Conventional ultrasonic techniques cannot be used due to the porosity and extensive microcracking of thermal spray coatings. These features may attenuate and scatter the ultrasonic waves, increasing the background noise and making it difficult to interpret the ultrasonic signals and obtain reliable data.

Computed microtomography (CMT) can be used as a NDT tool for three-dimensional imaging of microstructural features in thermal spray coatings. The CMT results reveal the differences in pore morphology arising from differences in thermal spray processes, that is, predominantly globular pores in plasma sprayed deposits, while the porosity is highly layered in high-velocity oxy-fuel deposits. Reference 15 describes the use of this technique for characterizing microstructure-property correlations in thermal spray coatings.

Small-Angle Neutron Scattering (SANS). Microstructure and defect (porosity) characterization can also be accomplished using the SANS technique described in Ref 16 and 17. Processing-microstructure-property correlations in both ceramic (e.g., YSZ) and metallic (e.g., NiCr) deposits have been studied using SANS, sometimes in conjunction with other characterization techniques such as scanning electron microscopy and image analysis. Image analysis is discussed in the article "Metallography and Image Analysis" in this Handbook.

REFERENCES

1. M.L. Berndt and C.C. Berndt, Thermal Spray Coatings, *Corrosion: Fundamentals, Testing, and Protection*, Vol 13A, ASM Handbook, ASM International, 2003, p 803–813
2. J.A. Sue and G.S. Schajer, Stress Determination for Coatings, *Surface Engineering*, Vol 5, ASM Handbook, 1994, p 647–653
3. C.C. Berndt, Tensile Adhesion Testing Methodology for Thermally Sprayed Coatings, *J. Mater. Eng.*, Vol 12 (No. 2), June 1990, p 151–158
4. D.A. Jones, *Principles and Prevention of Corrosion*, 2nd ed., Prentice Hall, 1995
5. G.D. Davis, C.M. Dacres, and L.A. Krebs, Electrochemical Techniques for In-Service Corrosion Monitoring, *Corrosion: Fundamentals, Testing, and Protection*, Vol 13A, ASM Handbook, 2003, p 509–513
6. T.T. Taylor and R.E. Taylor, Testing of Stability and Thermal Properties of Thermal Barrier Coatings, *Surface Engineering*, Vol 5, ASM Handbook, ASM International, 1994, p 654–659
7. J. Matejcek, S. Sampath, and J. Dubsky, X-Ray Residual Stress Measurement in Metallic and Ceramic Plasma Sprayed Coatings, *J. Therm. Spray Technol.*, Vol 7 (No. 4), Dec 1998, p 489–496
8. "Modified Layer Removal Method for Evaluating Residual Stresses in Thermal Spray Coatings," Accepted Practice for Mechanical Properties #1, ASM International/Thermal Spray Society, Jan 2002. Available as an Adobe Portable Document Format (PDF) file on the Thermal Spray Society Web site, www.asminternational.org/tss
9. P.S. Prevey, X-Ray Diffraction Residual Stress Techniques, *Materials Characterization*, Vol 10, ASM Handbook, American Society for Metals, 1986, p 380–392
10. D.J. Andrews and J.A.T. Taylor, Quality Control of Thermal Barrier Coatings Using Acoustic Emission, *J. Therm. Spray Technol.*, Vol 9 (No. 2), June 2000, p 181–190
11. J. Voyer, F. Gitzhofer, and M.I. Boulos, Study of the Performance of TBC under Thermal Cycling Conditions Using an Acoustic Emission Rig, *J. Therm. Spray Technol.*, Vol 7 (No. 2), June 1998, p 181–190
12. J. Voyer and H. Kreye, Determination of Cracking Resistance of Thermal Spray Coatings during Four-Point Bend Testing Using an Acoustic Emission Technique, *J. Therm. Spray Technol.*, Vol 12 (No. 3), Sept 2003, p 416–426
13. A. Kucuk, C.G. Dambra, C.C. Berndt, U. Senturk, and R.S. Lima, Cracking Behavior of NiCrAlY/YSZ Thermal Barrier Coatings under Four-Point Bend Loads, *Ceram. Trans.*, Vol 114, 2001, p 177–186
14. R.S. Lima, S.E. Kruger, G. Lamouche, and B.R. Marple, Elastic Modulus Measurements via Laser-Ultrasonic and Knoop Indentation Techniques, International Thermal Spray Conference 2003: *Advancing the Science and Applying the Technology*, Vol 2, ASM International, 2003, p 1369–1378
15. A. Kulkarni, S. Sampath, A. Goland, H. Herman, and B. Dowd, Computed Microtomography Studies to Characterize Microstructure-Property Correlations in Thermal Sprayed Alumina Deposits, *Scr. Mater.*, Vol 43 (No. 5), Aug 2000, p 471–476
16. D.G. LeGrand, Small-Angle X-Ray and Neutron Scattering, *Materials Characterization*, Vol 10, ASM Handbook, American Society for Metals, 1986, p 402–406
17. Z. Wang, A. Kulkarni, S. Deshpande, T. Nakamura, and H. Herman, Effects of Pores and Interfaces on Effective Properties of Plasma Sprayed Zirconia Coatings, *Acta Mater.*, Vol 51 (No. 18) Oct 2003, p 5319–5334

Powder Testing and Characterization

Walter Riggs, TubalCain Company Inc.

THE RAW MATERIALS used in thermal spray processes are a critical parameter in the finished coating. Lot-to-lot variations in powder or wire with respect to size, morphology, chemistry, and phase composition can significantly impact coating properties. This article discusses various characterization methods for powders and, in some cases, the resultant coating. Among the topics addressed are:

- Methods for determining particle size and/or size distribution
- Powder and coating stoichiometry
- Particle chemistry
- Phase analysis by x-ray diffraction

Additional information can be found in the article "Particle Characterization" in this Handbook.

Particle Size

Powder size and size distribution determination methods used for thermal spray powders include:

- Sieving
- Electrical resistance
- Sedimentation
- Centrifugation
- Image analysis
- Light scattering

Of the various methods available to determine particle size, sieving is still the most widely used, with light scattering and diffraction methods gaining in acceptance and popularity.

Sieving. Standard sieves traceable to the National Institute of Standards and Technology (NIST) can be obtained for determining powder size distributions. The sieves are usually stacked one on top of the other, with the larger sizes being on the top. The powder to be sized is poured into the top sieve and the entire unit is then shaken and tapped for several minutes. The shaking and tapping are intended to continually mix, reorient, and therefore introduce new material to the screen in order that particles smaller than the sieve opening can pass through to the next sieve. The results are expressed as percentages of the original material retained on each sieve. Standard sieve sizes and their respective micron opening sizes are:

Sieve	Opening, μm
400	37
325	44
270	53
230	62
200	74
170	88
150	105
140	105
120	125
100	149

The sieve method is perhaps the most commonly used, though its accuracy is somewhat controversial. An area of concern stems from the tacit assumption that the particles being screened are spherical. In practice, errors are introduced if particles with high length-to-width aspect ratios are sized using standard sieves. In addition, sieves can (a) wear, with the mesh openings becoming enlarged, and (b) load, resulting in particles of certain sizes to preferentially pass through.

Electrical Resistance. The standard electrical resistance particle size method is called the Coulter Counter. In particle size analysis using the Coulter effect, if a nonconducting particle suspended in a conducting medium is placed within a small aperture, an increase in the resistance across the orifice, relative to that of the medium alone, is produced when the particle passes the critical aperture. The magnitude of this increase in resistance, dR , for a spherical particle of diameter d suspended in an aperture of diameter D , is:

$$dR = [(8\rho d)/3\pi D^4][1 + 4/5(d/D)^2 + 24/35(d/D)^4 + \dots]$$

where ρ is the resistivity of the conducting medium. With suitable external circuitry, this resistance pulse dR results in a voltage pulse idR for a sphere of diameter d , where I is the current across the aperture. The dynamic range of a single aperture is 20 \times , and with a series of apertures the overall applicable size ranges from approximately 0.5 to 1000 μm .

Gravitational Sedimentation. The steady-state speed of a particle suspended in a viscous medium, settling in the gravitational field, is given by:

$$Mg = f(dx/dt)$$

where M is the particle effective mass, f the frictional coefficient, dx/dt the particle velocity, and g the acceleration caused by gravity (9.80 m/s^2). Steady-state speeds are customarily expressed as the sedimentation coefficient, S , the particle speed per unit field. Letting S be the sedimentation coefficient, and setting it equal to:

$$S = 1dx/gdt$$

it can be shown that:

$$S = M/f$$

At the outset, the results from a sedimentation experiment can be given as a distribution of the sedimentation coefficient S . If the densities of the particle, ρ , and the medium, ρ_0 , are known, then S can be expressed as:

$$S = [V(r - r_0)]/f$$

where V is the particle volume. Finally, if the particles are spherical, with the frictional coefficient given by Stokes' law as

$f = 3\pi\eta_0 d$ for a particle of diameter d suspended in a medium with viscosity η_0 , the sedimentation coefficient becomes:

$$S = [(\rho - \rho_0)d^2]/18\eta_0$$

from which particle diameter d may be calculated. Note that sedimentation is directly proportional to the particle diameter d , so that larger particles settle faster.

A popular device utilizing this method is the Micromeritics Sedigraph. This device uses a selection of fluids or dispersants of known viscosity. The powder sample is magnetically stirred in a beaker containing the dispersant; the mixture is simultaneously circulated through a length of plastic tubing into a control cell of known volume, then back to the beaker again with plastic tubing. The control cell has see-through glass windows on each side. The control cell is placed in a location such that a beam of x-rays passes through the windows; a detector on the opposite side senses how much of the x-rays pass. In practice, the mixing in the beaker and flowing through the cell continue for about 2 to 3 min, until steady-state conditions prevail—that is, uniform distribution of powder sizes throughout the loop.

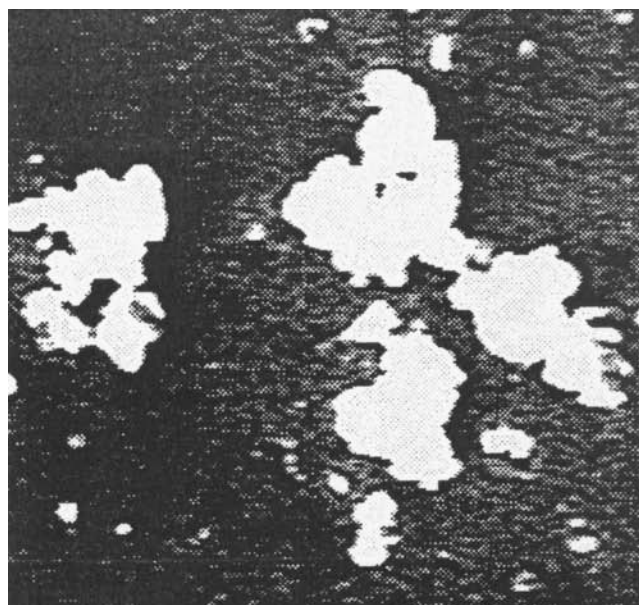
After the steady-state conditions have developed, the “start” switch is enabled. The flow immediately stops, essentially “freezing” the control cell. Sedimentation begins instantaneously. With the understanding from the final equation above stipulating that larger particles settle out (or “sediment”) faster, coupled with the mechanism of detection of quantity of x-rays passing through the control cell, mechanism can be thought of as dispersant/powder mixture turbidity versus time. That is, as the powder particles settle out in the control cell, the mixture becomes clearer with time, allowing more x-rays to pass through. The result is plotted as per-

centage of x-rays detected versus time, and proceeds from 0 to 100%. The dispersant is selected such that the entire test can be conducted in a reasonable amount of time of time (e.g., 1 h). The first 85 to 90% of the particle sizes settle out quickly (generally in a few minutes), whereas the final 10 to 15% settle out very slowly. Checking the device versus a garnet standard with a known particle size provides great accuracy—within 1 μm for an 11.5 μm reference standard.

Sedimentation methods generally suffer from the fact that few thermal spray powders are spherical (or even nearly spherical); departure from sphericity causes problems. In addition, it is suggested that significantly magnetic powders not be evaluated with sedimentation methods. This is most probably due to the magnetic stirring requirement for the acquisition of steady-state dispersion and distribution of powder particle sizes.

Centrifugal Sedimentation. Instrumentation for particle size analysis using gravitational sedimentation is of limited flexibility. The only means of varying particle speed is by selecting a medium with different viscosity and specific gravity. By using the centrifugal field, a substantially lower size limit is achieved, along with a considerable decrease in the time required for analysis.

Image Analysis. Figure 1 illustrates an optical means of determining particle size and shape. Image analysis techniques are used to digitize an image of the powder particles and, using image enhancement, thresholding, and binary processing routines, yield a size and shape distribution. While simple in theory, this method is difficult in practice due to the need to separate touching powder particles and to accumulate a significant number of images to properly statistically represent the specimen. Provided these issues can be overcome, image analysis is a precise method of measuring particles size and shape. More detailed information on



(a)



(b)

Fig. 1 Image analysis method of determining powder size and shape. (a) Original image. (b) Processed image, providing shapes, that are less ambiguous and easier to measure

image analysis can be found in the article “Metallography and Image Analysis” in this Section of the Handbook.

Laser Light Scattering and Diffraction. The increasingly popular laser light-scattering method can be described by two theories based on particle size: relatively small ($\leq 6 \mu\text{m}$) particles and larger particles. Brief discussion of these methods follows. For a more thorough treatment, refer to Chapter 6 in *Modern Methods of Particle Size Analysis* (Ref 1).

In the early 1900s, Gustav Mie formulated equations to account for the complete light-scattering pattern in a particulate system by considering the electrical field inside and outside a single particle (Ref 2). The scattering intensity was calculated as a series of expressions, each a function of various scattering angles, the difference between the refractive indices of the particle sphere and the continuum, the wavelength of the incident radiation, and the diameter of the sphere. As sphere or particle size increases from 1/10 to 10 times the size of incident light ($0.63 \mu\text{m}$ for a helium-neon laser), scattering from different portions of a single particle is out of phase, causing interference and reduced intensity. Phase differences are small at small scattering angles, resulting in greater intensity of scattered light at low angles. The overall effect gives rise to an angular distortion of scattered light (depending on the refractive index and particle size), in which most scattering (i.e., the highest intensity) is in the forward direction. Thus, light collection in a plane intersecting the forward scattered light is sufficiently intense and contains appropriate information for the determination of particle size.

For applications involving particles with a diameter much larger than the wavelength of light, there is little effect of the refractive index, and a special case of Mie scattering theory, Fraunhofer diffraction, is invoked. This theory explains that the intensity of light scattered by particles is proportional to particle size, and the size of the diffraction pattern (scattering angle) is inversely proportional to particle size.

The results of the light-scattering test are the percentages of a sample volume that exist in 13 different size “bins.” The threshold of the bins can be adjusted by changing apertures in the system.

Powder and Coating Stoichiometry

In certain applications of thermal spray coatings, properties other than hardness, wear rates, and corrosion resistance are important. Examples of such coatings are (a) biomedical, where compatibility with bone tissue is essential, and (b) electromagnetic, or electronic applications, where the coatings may serve as a ferro- or ferrimagnetic material, a dielectric, a resistance, or even a conductor. In such applications the coating stoichiometry is the prime interest. Without it, the coating cannot fulfill its function. Stoichiometry refers to the compositions, the nature of the crystal lattice, and the crystal structure or phase(s) of the material.

Stoichiometry is of interest in mechanical applications as well. For instance, 88WC/12Co forms a “collage” of carbide compositions when sprayed in air as a result of reaction kinetics at higher temperatures. The same composition sprayed with high-velocity oxyfuel (HVOF) processes exhibits chemistry and structure properties much closer to the powder.

Many technologists assume that the coating material will be identical in composition and crystal phase to the injected powder. This is untrue. Some factors that may influence compositional differences are discussed below, along with examples.

Preferential volatilization may occur in some metals and ceramics due to higher vapor pressures and reaction kinetics within the plasma or thermal spray plume. As an example, the common ferrimagnetic NiZn ferrite will lose zinc when sprayed in air because of the zinc volatility.

Rapid solidification (RS) may cause a compositional shift, particularly if the material desired is close to a eutectic formation. By way of example, RCO_5 (where R is a rare-earth element such as Sm, Y, La, Ce, or Pr) may shift to a mixture of several compounds or phases.

The spraying of α -alumina results in γ -alumina coatings. Heating the deposit to $>1100^\circ\text{C}$ (1830°F) causes the γ -alumina to revert to the α -phase, but at the cost of a dimensional (volume) change that crazes the deposit. Alternatively, the coating may be deposited on a substrate that is heated to the conversion temperature. The result will be α -alumina, but other factors such as coefficients of thermal expansion and substrate oxidation may restrict this application.

Rutile (TiO_2 , white in color, high dielectric properties) deposits as an incomplete anatase phase (TiO_{2-x}) that is almost black in color and exhibits semiconducting properties. Conversion to rutile takes place at temperatures of approximately 750°C (1380°F), with a potential 7% dimensional change. As with alumina, the rutile phase may be maintained if substrate temperatures are held above the critical temperature.

Rapid solidification is responsible for the ultrafine grain sizes of deposited coatings. Metals usually exhibit grain sizes of $<0.1 \mu\text{m}$. Plasma-deposited ceramics are $<0.05 \mu\text{m}$. At such small sizes, electromagnetic properties are greatly altered. Ferro- and ferrimagnetic materials exhibit superparamagnetic properties, dielectric ceramics have greatly lowered dielectric constants, and normally electrically insulative materials such as ferrites and garnets are semiconductors or conductors.

Therefore, optimization of a coating process is not simply a matter of adjusting parameters for maximum bond strength or density. In the section that follows, a number of methods for determining the stoichiometry of the initial spray powder and the resulting coatings are described. Some of these analytical methods can make use of a coating on an appropriately sized substrate. Others require “powdering” of the coating so that it will compact in a sample holder. Such powders can also be milled from small “free-stands” made by removing the substrate.

Particle Chemistry

This section draws heavily upon a standard reference for atomic absorption, atomic emission, and x-ray fluorescence methods: H.H. Willard’s *Instrumental Methods of Analysis* (Ref 3). In addition to wet chemical methods, adsorption and emission spectroscopy are used to determine the chemistry of thermal spray feedstock materials. Spectroscopy is the measurement and interpretation of electromagnetic radiation absorbed or emitted when

molecules, atoms, or ions of a sample move from one energy state to another. A characteristic of a chemical element is that the energy required to raise it to the next higher energy level is specific and repeatable; this "identification" of differences of energy levels can be used to determine the presence or absence of given chemicals in a material, in addition to the relative quantity, or percent, of the total powder.

Wet chemical methods involve dissolving the powder material into a liquid solvent. The solution is then processed with additional chemicals and treatments to cause precipitation of the element of interest. The precipitate is thoroughly dried and weighed, the mass being proportional to the original sample. (This brief discussion is a dramatic oversimplification of wet chemical methods, but captures the basics of the approach.) Wet chemical methods, while accurate, are not generally used in the production release of materials used for thermal spray coatings; rather, atomic emission and absorption, and x-ray fluorescence and diffraction techniques are favored.

Atomic Absorption Spectrometry. Combustion flames provide a remarkably simple means for converting inorganic materials in solution into free atoms. It is only necessary to introduce an aerosol of the sample solution into an appropriate flame, and a fraction or all of the metallic ions present in the aerosol droplets will eventually be converted into free atoms. Once the free atoms are formed, they can be detected and quantitatively determined at trace levels by atomic flame emission, atomic absorption, or atomic fluorescence spectrometry.

Atomic absorption in flames is carried out using a principle that entails determination of the absorption at the line center using a narrow-line source emitting a given resonance line of the element, whose emission line profile is less than the absorption line profile of the material in the flame. The gases are treated as a medium containing free, unexcited atoms capable of absorbing radiation from an external source when the radiation corresponds exactly to the energy required for a transition of the test element from the ground electronic state to an upper excited electronic state. Unabsorbed radiation passes through a monochromator that isolates the exciting spectral line and into a photodetector. Absorption is measured by the difference in transmitted signal in the presence and absence of the test element.

Atomic emission spectroscopy has long been the standard for routine metal analysis in many applications. The basic principle underlying the method can be described as follows: A minute part of the sample is vaporized and excited to the point of light emission. This may be done using an electric arc or spark, a direct-current (dc) argon plasma, an inductively coupled argon plasma (ICAP), or a laser. Having selected the source, the emission spectrum is focused onto the spectrometer entrance slit. The light derived from the vaporized, excited material is dispersed into its component parts in the spectrometer. At the exit aperture this light is either photographed on a plate or film, or recorded by a photodetector. Since each element produces a series of spectral lines of specific wavelengths, characteristic of itself, the identification of an element is possible by studying the lines according to their respective locations. Determination of quantity is made according to the intensity of the lines in almost any matrix, ranging from alloys to ores, or from ashes of organic materials to atmospheric dusts.

The ICAP is a special type of plasma that derives its sustaining power by electromagnetic induction from a high-frequency magnetic field. A similar technology is sometimes used to deposit coatings and is called radio frequency (RF) induction plasma. Essentially, a gas flowing through a tube is ionized in a strong induced magnetic field; the magnetic field is induced by a high-frequency (450 kHz to several MHz) current flowing in a coil surrounding the flowing gas. For the inductively coupled plasma (ICP) atomic emission test, a tube is inserted beneath the glowing ionized argon cloud or annulus; the material is injected into the plasma via a carrier gas. The powder is then ionized, producing spectral lines characteristic of the elements it contains. There are no electrodes in ICP sources. Therefore, excitation and emission zones are resolved spatially, producing a relatively clean background spectrum that consists of argon lines and some weak band emission from OH, NO, NH, and CN molecules. This low background, combined with a high signal-to-noise ratio of sample emission, produces low detection limits, typically in the parts-per-billion range.

X-Ray Fluorescence. Characteristic x-ray spectra are excited when a specimen is irradiated with a beam of sufficiently short-wavelength x-ray radiation. Intensities of the resulting fluorescent x-rays are smaller by a factor of roughly 1000 than an x-ray beam obtained by direct excitation from a beam of electrons. Only availability of high-intensity x-ray tubes, very sensitive detectors, and suitable x-ray optics render the fluorescent method feasible. A certain number of quanta have to be accumulated at the detector in order to sufficiently reduce the statistical error of the measurement. The sensitivity of the analysis—that is, the lowest detectable concentration of a particular element in a specimen—will depend on the peak-to-background ratio of spectral lines.

Phase Analysis by X-ray Diffraction

Increasingly, phases (crystal structure) in thermal spray coatings are being analyzed by x-ray diffraction. Only a brief discussion of this method will be provided here. The interested reader should refer to *Elements of X-ray Diffraction* by B.D. Cullity for a more in-depth treatment (Ref 4). Figure 2 shows a diagram typically used to

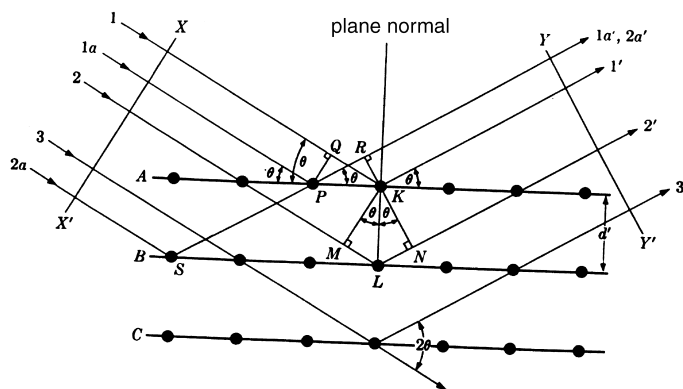


Fig. 2 Schematic of Bragg diffraction of a crystal

discuss Bragg's law. Specifically, incident x-ray radiation of a prescribed wavelength (1.541 \AA for copper $K\alpha$) is directed at the surface of a specimen at a given angle. In order for the diffracted rays to be detected, they must be in phase with each other. The second wave travels through the additional distance $ML + LN$ (refer to Fig. 2); this total length must then be equal to some integer number of wavelengths λ , or $n\lambda$. The angles are equal to the incident angles θ . In addition, $ML = LN = KL \sin \theta$. Since $KL = d$, the overall equation becomes $n\lambda = 2d \sin \theta$. Thus, by rotating an analyzing crystal mounted on a goniometer through a scan of θ values, since the incident wavelength λ is known, the interplanar spacings, or d , can be determined. These interplanar spacings are unique for each crystalline material. Figure 3 shows an x-ray diffraction 2θ scan for a WC/Co coating, with most of the peaks identified for phase and type of crystallographic plane. The highest peaks are threefold and appear at 2θ angles of approximately 31° , 35.5° , and 48.5° in the figure. The peaks are labeled WC(001), WC(100), and WC(101). They are the reflecting peaks for primary tungsten carbide. The scan was taken from a sprayed coating. The primary carbide peaks in the diffraction scan reveal that the carbides present in the powder were not dissolved during spraying.

The individual peak heights for a given set of planes for a given phase are a function of:

- The accumulation of reflections of x-rays from individual atoms spaced throughout the crystal lattice, which depends on the crystal structure, type of atom, and its position within the lattice (structure factor, F)
- The total number of planes available within that particular phase, of that particular type, for reflecting the x-rays. For example, in simple cubic materials there are four planes of the type $\{111\}$, but only three planes of the type $\{100\}$; thus, the total volume of reflected x-rays will be greater for the $\{111\}$ planes (multiplicity factor, p).
- The spacing between planes of atoms, which changes the angle at which a given set of planes reflect. Angles of low spacing have low reflection angles, and vice versa; as a diffraction trace proceeds from left to right, or from low angles to high angles, the total reflection ability of a given set of planes decreases (Lorentz polarization factor, L).
- To a lesser extent, the ability of the materials to absorb, rather than reflect, the incident beam of x-rays (absorption factor, A)
- The effect of temperature. Atoms in a crystal lattice vibrate about their mean positions, even at absolute zero temperature; the vibration is thermally induced and, as such, increases with temperature. The result is that, at higher temperatures, the atoms can be regarded as being not at exact fixed positions, but rather within "bands"; the lattice expands and the spacing between planes of atoms changes, thus affecting angles of reflection (temperature factor, $e^{-2\theta}$).
- The volume of the unit cell. If one phase has a smaller unit cell than the other, its ability to reflect increases. This can be envisioned as growing or shrinking of a cube with atoms at the corners—the smaller cube, of course, being more densely occupied with atoms while maintaining the same size of incident beam. The beam is less likely to "pass through" the denser, or smaller cube (unit cell volume, v).

Thus, for any given set of planes for any given phase, the necessary calculations can be regarded as:

$$\text{Intensity } I = F^2 p L A e^{-2\theta}$$

For comparative quantitative phase analysis, the following equation is used:

$$I_\gamma / I_\alpha = R_\gamma C_\gamma / R_\alpha C_\alpha$$

where $R_\gamma = (1/v_2)F^2 p L A e^{-2\theta}$ and C_γ = concentration of phase γ .

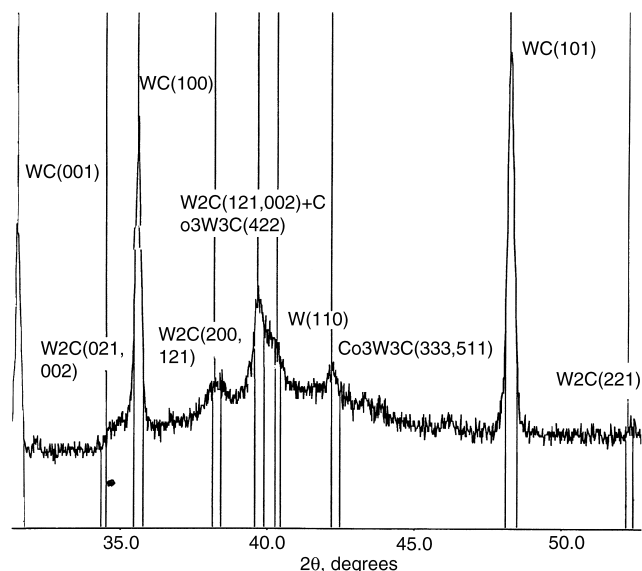


Fig. 3 X-ray diffraction 2θ scan for WC/Co coating sprayed at moderate parameter levels. Compare with Fig. 4.

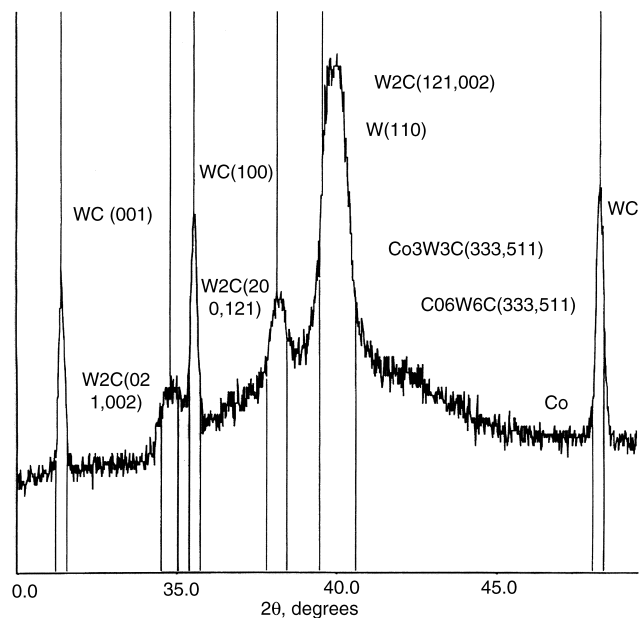


Fig. 4 X-ray diffraction 2θ scan for WC/Co coating sprayed at higher energy or "hotter" parameter levels than the coating in Fig. 3. Note the reduction in size of the WC peaks as compared with Fig. 3.

Use of the foregoing methods include the quantitative determination of phases—for example, the monoclinic phase in yttria-stabilized zirconia, primary tungsten carbide retention in WC/Co coatings, and calcium phosphate phases in hydroxyapatite medical/dental coatings. Figure 4 shows another diffraction scan from a sprayed WC/Co coating. Comparing it to the scan shown in Fig. 3 reveals that the primary WC peaks have “shrunk” and the peak at approximately 40° , representing the subcarbide W_2C , has grown significantly. This is evidence of the dissolving of the primary tungsten carbide particles, which was expected, since the spray process for this coating was “hotter” than for the one shown in Fig. 3.

In addition to phase analysis, efforts have been made to use

x-ray diffraction to assess texture (preferred orientation or solidification direction) and residual stresses.

REFERENCES

1. H. Barth, Ed., *Modern Methods of Particle Size Analysis*, John Wiley & Sons, 1984
2. G. Mie, *Ann. Phys.*, Vol 25, 1908, p 377
3. H.H. Willard, Ed., *Instrumental Methods of Analysis*, 7th ed., Wadsworth Publishing, 1988
4. B.D. Cullity, *Elements of X-ray Diffraction*, 2nd ed., Addison Wesley, 1978

Glossary of Terms*

A

adhesive. A material that resists adhesion. A film or coating applied to surfaces to prevent sticking, such as a parting or mold-release agent.

ablation. A self-regulating heat- and mass-transfer process in which incident thermal energy is expended by a loss of material.

abradable coating. The sacrificial member of a two-component clearance control (gas path seal) system, usually applied to a static component.

abrasion. (1) A roughening or scratching. (2) Grinding or wearing away using abrasives.

abrasion rate. The rate at which material is removed from a surface during abrasion. Usually expressed in terms of (1) thickness removed per unit of time or traversed distance, and (2) mass removed per unit of time or distance traveled.

abrasion resistance. The ability of a material to resist surface wear.

abrasive. (1) A hard substance used for grinding, honing, lapping, superfinishing, polishing, pressure blasting, or barrel finishing. Typical materials include alumina, silicon carbide, boron carbide, diamond, cubic boron nitride, garnet, and quartz. (2) Hard particles that wear away a surface when moved across it.

abrasive belt. A coated abrasive product, in the form of a belt, used for grinding and/or polishing.

abrasive blasting. A process for cleaning, finishing, or roughening with an abrasive under high pressure. See preferred term *blasting or blast cleaning*.

abrasive erosion. Erosive wear caused by fluidized solid particles moving across a surface. See also *erosion*.

abrasive wear. Removal of material by particles moving across a surface. The particles may be loose or part of another surface in contact with the surface being worn. Compare with *adhesive wear*.

abrasivity. The extent to which a surface, particle, or particles will cause *abrasive wear* when forced against a solid surface under relative motion and certain conditions.

absorb. To take in.

absorption. (1) The taking up of a liquid or gas by capillary, osmotic, or solvent action. (2) The ability to receive and retain a substance, usually a liquid or gas. (3) The process by which a liquid is drawn into a porous substance.

accelerated corrosion test. Test method designed to approxi-

mate, in a short time, the deteriorating effect under normal long-term service conditions.

accelerated-life test. See preferred term *accelerated testing*.

accelerated testing. A test meant to produce failures caused by the same mechanism as expected in field operation but in a significantly shorter time.

acceptable quality level. (1) The lowest acceptable quality level permitted. (2) The maximum percentage of defects considered to be an acceptable average for a given process.

acceptance. A decision that the process is operating properly with respect to the statistical measures being followed.

acceptance test. Tests conducted on receipt by, or for, the procuring agency to (1) determine if an individual lot of product conforms to the contract or to (2) determine the degree of uniformity of the item supplied. Compare with *preproduction test* and *qualification test*.

accepted reference value. A value that serves as an agreed-on reference for comparison and that is derived as: (1) a theoretical or established value, based on scientific principles, (2) an assigned value, based on experimental work, or (3) a consensus value, based on collaborative experimental work. When the accepted reference value is the theoretical value, it is sometimes referred to as the “true” value.

accuracy. The closeness of agreement between a test result and a fact or value.

acetylene (C₂H₂). Unsaturated aliphatic hydrocarbon gas used as a fuel in combustion gas flame spraying.

acicular powder. A powder composed of needlelike or sliverlike particles.

acid. A substance having a pH below 7.0. Compare with *base*.

acid embrittlement. A form of *hydrogen embrittlement* that may be induced in some metals by acid.

acoustical room. A soundproof enclosure containing thermal spraying and sometimes auxiliary equipment. Its purpose is to reduce or isolate unacceptable operating noises from the surrounding environment.

acoustic emission. A measure of the integrity of a material as determined by sound emission when that material is stressed.

acrylics. A broad array of polymers and copolymers in which the major constituents belong to two families of esters-acrylates and methacrylates.

activation. The changing of a passive surface of a metal to a chemically active state. Contrast with *passivation*.

*This glossary was adapted from *Thermal Spray Terminology and Company Origins*, F.J. Hermanek, Ed., ASM Thermal Spray Society, 2001.

active metal. A metal receptive to chemical reaction as corroding or being corroded.

adhere. To cause two surfaces to be held together by adhesion.

adherence. The attachment of a material or coating to a surface (either by adhesion or other means). See also *adhesion*.

adherent. A body held to another body by an adhesive force. See also *substrate*.

adhesion. A binding force that holds together bodies or materials that are normally separated.

adhesive. A substance used to hold bodies or materials together. The word is a general term used to include the following: cement, glue, mucilage, and paste. The terms are often used interchangeably.

adhesive bond. Attractive forces between a coating and the substrate.

adhesive joint. Location at which two adherents are held together with an adhesive. See also *bond*.

adhesive strength. The magnitude of attractive forces between a coating and substrate. Interactions contributing to adhesion are *van der Waals* forces, permanent dipole bonds, and mechanical interlocking. Generally expressed in kilopascals (kPa) or pounds per square inch (psi).

adhesive wear. Wear by transference of material from one surface to another due to solid-phase welding. Particles removed from one surface are permanently or temporarily attached to the other surface. Compare with *abrasive wear*.

admixture. A blend of two or more chemically different powders.

adsorb. To take in.

advanced ceramics. Chemical compounds that exhibit superior properties. Includes many monolithic ceramics and particulate, whisker-, and fiber-reinforced glass, glass-ceramics, and ceramic-matrix composites. Materials can be oxides, carbides, nitrides, borides, and so on.

advanced composites. Materials reinforced with continuous fibers having a modulus higher than that of fiberglass fibers. Includes metal-matrix, ceramic-matrix, and carbon-carbon composites.

aerate. To fill with air or gas; to fluff up a powder or grit.

aeration. Exposing to the action of air or a gas.

age hardening. Hardening a metal by *aging* (heat treatment), usually after rapid cooling or cold working.

age softening. A decrease of strength and hardness over time and some temperature. It can occur at room temperature in certain strain-hardened alloys, especially those of aluminum.

agglomerate. The clustering together of particles used to form a composite powder. Examples include Ni+Al and NiCr+Al.

aggregate. A mass of particles held together by adhesive or interparticle forces.

aging. Denotes a change in the properties of certain metals that occurs at ambient or moderately elevated temperatures after hot working or a heat treatment (quench aging in ferrous alloys, natural or artificial aging in ferrous and nonferrous alloys) or after

a cold working operation (strain aging). The change is often due to a phase change (precipitation) but never a change in chemical composition. See also *age hardening*.

agitator. A device used to intensify mixing. Example: a high-speed stirrer or paddle in a blender.

air cap. A device for forming, shaping, and directing an air pattern for the atomization of wire or ceramic rod during spray.

air classification. The separation of powder into particle-sized fractions by means of an air stream of controlled velocity.

air cooler. See preferred term *auxiliary cooler*.

air dried. Dried without the application of heat.

air feed. A process variation in which an air stream conveys the material to be sprayed through the gun and into the heat source.

air filter. Mechanism for cleaning air of such contaminants as water, oil, and solid matter.

air furnace. Reverberatory-type furnace in which heat is obtained by burning fuel.

air-hardening steel. A steel containing sufficient carbon and other alloying elements to harden fully during cooling in air from a temperature above its transformation range. Same as self-hardening steel.

air roller analyzer. An instrument used for air classification of thermal spray powder. Based on Stokes' law.

alclad. Composite wrought product consisting of an aluminum alloy core with one or both surfaces overlaid with a metallurgically bonded aluminum or aluminum alloy.

alcohols. A series of hydroxyl liquid compounds that are colorless, volatile, and flammable. They can be manufactured by the fermentation of sugars, starches, or petroleum. Often used as cleaning agents and in the manufacture of synthetic resins, rubbers, and plasticizers.

alkali metal. A metal in group IA of the periodic system. Included are lithium, sodium, potassium, rubidium, cesium, and francium.

alkaline. Having a *pH* greater than 7.0.

alkaline cleaner. A material blended from alkali hydroxides and alkaline salts such as borates, carbonates, phosphates, or silicates, usually with water.

alkyd. Reaction products of polyhydric alcohols and polybasic acids.

alligatoring. Pronounced cracking on the surface of a coating. It has the general appearance of an alligator hide or skin.

alligator skin. See *alligatoring* and *orange peel*.

allotropy. (1) Generalized synonym for *polymorphism*. (2) The existence of a substance, especially an element, in two or more physical states (for example, crystals). See also *graphite*.

allowance. The specified difference in limiting size or range from the specified dimension and/or tolerance.

alloy. (1) A substance having metallic properties and being composed of two or more chemicals, at least one of which is a metal. (2) To make or melt an alloy.

alloying element. An element added to and remaining in a metal that changes structure and properties.

alloy plating. The codeposition of two or more metallic elements.

alloy powder, alloyed powder. A metal powder consisting of at least two constituents that are partially or completely alloyed with each other.

alloy steel. A steel containing specified quantities of alloying elements (other than carbon and manganese) added to effect changes in properties.

alloy system. A complete series of compositions produced by mixing in many proportions any group of two or more components, at least one of which is a metal.

Alumel. A nickel-base alloy containing approximately 2.5% Mn, 2% Al, and 1% Si and used chiefly as a component of pyrometric thermocouples.

alumina. A ceramic aluminum oxide (Al_2O_3). The natural crystalline mineral is called *corundum*. Uses include: abrasives, structural ceramics, refractories, pigments, and chemicals.

aluminizing. Thermally treating an aluminum coating, causing it to react and diffuse into its substrate.

aluminum. A white metal obtained from the mineral bauxite. The second most abundant metallic element on earth.

amalgam. An alloy produced by combining mercury with silver, tin, copper, and sometimes zinc.

ambient. The environment that surrounds and contacts a system or component.

American wire gage (AWG). A standard used in the determination of the physical size of a metallic thread. Determined by its circular-mil area.

ammeter. An instrument for measuring the magnitude of electric current flow (amperes).

amorphous. Not having a crystalline structure; noncrystalline.

amorphous solid. A rigid material whose structure lacks crystalline periodicity. See also *metallic glass*.

ampere. The unit used for measuring the quantity of an electric current flow.

amplifier. A lens used in place of an eyepiece to project the magnified image formed by an objective. It is designed for flatness of field and should be used with an apochromatic objective.

analysis. Identification and/or concentration determination of the constituents or components of a sample. See also *determination*.

analyzer. Any device used for determining properties.

anchorage. Keying action by which coatings are attached to their substrates.

anchoring. The attachment or locking of a coating to its substrate by providing a roughened surface. Methods include grit blasting, knurling, threading, and inserting screw heads, studs, and so on.

anchor pattern. A pattern made by threading, knurling, abrasive blasting, and so on a surface in preparation for spraying. Pattern is examined in profile.

angstrom unit (Å). A unit of linear measure equal to 10^{-10} m, or 0.1 nm.

angularity. Being angular. Generally referred to as the spray angle or degrees off perpendicular from the surface being sprayed.

anhydride. A compound from which water has been extracted.

anion. A negatively charged *ion*.

anisotropic. Exhibiting different properties when examined along axes in different directions.

anisotropy. The characteristic of exhibiting different values of a property in different directions.

annealed powder. A metallic powder that is heat treated to render it softer.

annealing. A generic term denoting a treatment used primarily to soften metals.

anneal to temper. A final partial anneal that softens a cold-worked nonferrous alloy to a specified level of hardness or tensile strength.

anode. The positive electrode in a thermal spray device. In a plasma thermal spray gun, it is the front electrode, often the nozzle. In electric arc spray guns, one feed wire guide is the positive electrode.

anodic protection. Imposing an external electrical potential to protect a metal from corrosive attack. Contrast with *cathodic protection*.

anodizing. Forming a *conversion coating* on a metal surface by anodic oxidation; most frequently applied to aluminum.

antibond. Any liquid substance applied to surfaces adjacent to those to being sprayed and on which coating is not desired.

anticorrosive additive. A lubricant additive used to reduce corrosion.

antifouling coating. A coating material applied to prevent fouling, by living organisms, of underwater structures such as boat/ship hulls.

antifriction material. Any material that exhibits low-friction or self-lubricating properties. Examples: CuNiIn and aluminum-bronze.

antiseizure property. The ability of a bearing material to resist seizure during momentary lubrication failure.

antiwear additive. A lubricant additive used to reduce wear. Examples: graphite and molybdenum disulfide.

aperiodic. Irregular; occurring without a recognized format or set of rules. Used to describe the atomic/crystalline arrangement of quasi-crystalline alloys.

aperture. The working diameter of a lens or mirror in a light microscope.

aperture size. The opening of a mesh, as in a sieve. See also *sieve analysis*.

apparent density. The weight per unit volume of a powder, in contrast to the weight per unit volume of the individual particles. Apparent density is always less than the true density of a solid, homogeneous material. See also *density*.

apparent hardness. The value obtained when testing a coating with standard indentation hardness equipment. Because the reading is a composite of pores and solids, it is usually lower

than that of a wrought or cast material of the same composition and condition. Not to be confused with *particle hardness*.

apparent pore volume. The total pore volume of a loose powder mass. Calculated by subtracting the apparent density from the theoretical density of the substance.

apparent porosity. The relation of the open pore space to the bulk volume, expressed in percent.

arc. A luminous discharge of electrical current crossing the gap between two electrodes.

arc chamber. The confined space within the plasma thermal spray gun enclosing the anode and cathode in which the arc is struck.

arc force. The axial force developed by a plasma.

arc gas. The gas or gases introduced into the arc chamber and ionized to form a plasma.

arc gas secondary. See preferred term *primary gas*.

architectural masking. Dedicated masking, usually reusable, designed for a specific configuration or geometry (hard tooling/masking).

arc plasma. See *plasma*.

arc voltage. The voltage across the arc.

arc wire spraying. See preferred term *electric arc spraying*.

aromatic. An unsaturated hydrocarbon with one or more benzene ring structures in the molecule.

aromatic polyester. A polyester derived from monomers in which all the hydroxyl and carboxyl groups are directly linked to an aromatic nuclei.

artifact. A feature of artificial character, such as a scratch or lint, on a metallographic sample that can be erroneously interpreted as a true feature. See also *mounting artifact* and *polishing artifact*.

as-brazed. The condition of brazements after *brazing* and without subsequent thermal, mechanical, or chemical processing.

asperity. A peak, protuberance, or elevation in the small-scale topographical irregularities of a solid surface. Typically used to describe the higher features (peaks) on a grit-blasted surface.

assay. The determination of how much of a sample is the material indicated by name.

assembly. A number of parts or subassemblies combined or joined together.

as-sprayed. The condition of the thermal sprayed coating and without subsequent thermal, mechanical, or chemical processing.

athermal. Not isothermal. Changing rather than constant temperature conditions.

atom. The smallest particle of an element that retains the characteristic properties and behavior of the element. See also *atomic structure* and *isotope*.

atomic number. The number of protons in the nucleus of an atom.

atomic percent. The number of atoms of an element in a total of 100 representative atoms of a substance.

atomic structure. The arrangement of the parts of an atom. Also used to describe the atomic alignment of a phase or compound.

atomic weight. (1) A number assigned to each chemical element that specifies the average mass of its atoms. (2) The mean weight of the atom of an element in relation to $^{12}\text{C} = 12.000$.

atomization (powder metallurgy). The disintegration of a molten metal into particles by a rapidly moving gas or liquid stream or by other means.

atomization (thermal spraying). The division of molten material at the end of the wire or rod into fine particles.

atomizer. A device that atomizes a sample. See also *atomization*.

attrition. A gradual wearing away; the removal of minute surface fragments during sliding contact.

attritor. A high-intensity ball mill wherein the drum is stationary and whose balls are agitated by rotating baffles, paddles, or rods at a right angle to the drum axis.

austenite. A structure or phase found in iron alloys. It is a solid solution of one or more elements in face-centered cubic iron (gamma iron).

austenitic corrosion-resistant stainless steel. A highly alloyed corrosion-resistant stainless steel containing 16% or more chromium and austenite-stabilizing elements such as nickel (up to approximately 35%), manganese, and nitrogen. Typical austenitic stainless steels are those of the 300 series. See also *stainless steels*.

austenitic grain size. The size attained by the grains in steel when heated to the austenitic region. It may be revealed by appropriate etching of metallographic cross sections.

austenitic steel. An alloy steel whose structure is normally austenitic at room temperature.

autoclave. A closed, pressured vessel for conducting chemical reactions including sterilization. Widely used for bonding and curing reinforced plastic laminates.

automatic spraying. Spraying without adjustment of the controls by an operator. The equipment may or may not perform the loading and unloading of the work.

auxiliary cooler. A device used to direct compressed air or gas onto the vicinity being sprayed to prevent overheating of the deposit or the substrate.

average density. The *density* measured on an entire body/coating or on a large number of its parts whose values are then averaged.

average erosion rate. The total erosion divided by the corresponding total exposure time.

average particle size. A single value representing the entire particle size distribution.

average run length. The average number of times/units that a process/parts must be sampled and evaluated before a change in processing is observed.

Avogadro's number. The number of molecules (6.0225×10^{23}) in a mole of any substance. Also see *molecule*.

axial. Longitudinal, or parallel to the spray direction, axis, or centerline of a part or thermal spray gun.

axial injection. Injecting or feeding the spray stock down the axial length of the thermal spray device.

axial loading. The application of pressure in the direction of the press axis.

axis. A straight line about which a body or object rotates or may be conceived to rotate.

B

Babbitt metal. Nonferrous bearing alloys consisting mainly of various amounts of copper, antimony, tin, and lead. Lead-base Babbitt metals are also used.

backfire. The momentary recession of the flame into the flame spray gun tip, followed by immediate reappearance or complete extinction of the flame.

baffle. A device used to restrict or divert passage.

baghouse. A chamber containing bags for filtering solids out of gases.

bainite. A metastable aggregate of *ferrite* and *cementite* resulting from the transformation of *austenite* at temperatures below the *pearlite* range but above M_s , the martensite start temperature. Upper bainite is an aggregate containing parallel lath-shaped units of ferrite, exhibiting a “feathery” appearance. It is formed above 350 °C (660 °F). Lower bainite has an acicular appearance similar to tempered martensite. It is formed below 350 °C (660 °F).

bake. Low heating in an oven at a controlled temperature.

Bakelite. (Georgia-Pacific Corporation) A proprietary name for a phenolic thermosetting resin used for mounting metallographic samples.

baking. (1) Heating to a low temperature in order to remove gases. (2) Heat curing or hardening surface coatings such as sealers. (3) Heating to drive off moisture or oils. (4) Preheating a part prior to spray.

ball bearing. A rolling bearing in which the elements are spherical.

ball mill. A machine consisting of a rotating hollow cylinder partly filled with grinding media (usually hardened steel, white cast iron, or ceramic material) used to pulverize a product or object.

ball milling. A method of grinding a material in a rotating cylinder or conical mill partially filled with grinding media. Used to reduce particle size and/or cause agglomeration.

bar. (1) A section hot rolled from a *billet* to a form a round, hexagonal, octagonal, square, or rectangular shape with sharp or rounded corners or edges and a cross-sectional area of less than 105 cm² (16 in.²). (2) An obsolete unit of pressure equal to 100 kPa.

barstock. Same as *bar*.

base. (1) The substrate or host surface. (2) A chemical substance having a *pH* higher than 7.0. (3) The surface on which a single-point tool rests when held in a tool post; also known as the heel.

base material. The material to be coated, welded, brazed, soldered, or cut. See also *base metal* and *substrate*.

base metal. (1) The metal to be coated, brazed, cut, soldered, or welded. (2) The metal present in the largest proportion in an alloy. (3) A metal that readily oxidizes or that dissolves to form ions. Contrast with *noble metal*. See preferred term *substrate*.

base metal test specimen. A test sample having properties and composition representative of the base metal.

basis metal. The original metal to which one or more coatings are applied. See preferred term *substrate*.

basketweave. A spray pattern in which the passes resemble a woven pattern.

batch. A quantity of parts or materials processed together and having identical characteristics throughout. See also *lot*.

bauxite. A whitish to reddish mineral, $Al_2O_3 \cdot 2H_2O$; the most important ore (source) of aluminum.

bearing area. The projected bearing or load-carrying area when viewed in the direction of the load. Often used as a synonym for real area of contact.

bearing bronzes. Bronzes used for bearing applications. Included are copper alloys containing 5 to 20 wt% Sn and a small amount of phosphorus (phosphor bronzes) and those containing up to 10 wt% Sn and up to 30 wt% Pb (lead bronzes). See also *bronze*.

bearing strength. The maximum *bearing stress* that can be sustained.

bearing stress. The *shear* load on a mechanical joint divided by the effective bearing area.

bed. For machine tools, the portion of the main frame that supports the tool, the work, or both.

bell. A jarlike enclosure for containing a vacuum or a controlled atmosphere.

bellows seal. A type of mechanical seal that uses a bellows for providing secondary sealing.

bell-type furnace. A type of controlled atmosphere furnace having a belllike shape.

belt furnace. A continuous-type furnace that uses a mesh-type or cast-link belt to convey parts through the furnace.

belt grinding. Grinding with an *abrasive belt*.

bench press. Any small press that can be mounted on a bench or table.

bend angle. The angle through which a bending operation is performed.

bending. The forming of material by moving it around a straight axis lying in the neutral plane.

bending strength. See preferred term *bending stress*.

bending stress. Either uniformly or nonuniformly distributed stress involving a combination of both tensile and compressive forces. Resistance to bending can be termed stiffness.

bend or twist. Distortion similar to warpage generally caused during forging or trimming operations. When the distortion is along the length of the part, it is referred to as bend; across the width, it is twist.

bend radius. (1) The inside radius of a bend section. (2) The radius of a tool around which metal is bent during fabrication.

bend test. Mechanical test for adhesion determination in which a flat sprayed panel (25 mm by 150 mm, or 1 in. by 6 in.) is bent over a prescribed radius. The coating is always to the outside of the bending mandrel.

bentonite. A colloidal claylike substance derived from decomposed volcanic ash. The main components include minerals of the montmorillonite family. Often used as a component in an abradable coating.

berry formation. See preferred term *nozzle accumulation*.

beryllia. Beryllium oxide (BeO). A colorless to white powder used in the manufacture of ceramic parts, including basic refractories and electronic substrates (heat sinks).

beryllium. A steel-gray, lightweight, hard metallic element (Be). Inhalation of powdered beryllium and its compounds should be avoided. Users should comply with occupational safety and health standards.

beryllium-copper. Copper-base alloys containing not more than 3% Be. These alloys rank high among copper alloys in attainable strength, while retaining useful levels of electrical and thermal conductivity.

beryllium oxide (BeO). See *beryllia*.

bias. The difference between the population mean of the test results and an accepted reference value.

biaxiality. Having two axes.

biaxial load. A loading condition in which a specimen is stressed in two directions.

biaxial stress. A state of stress in which only one of the principal stresses is zero, the other two being in tension.

billet. A semifinished section that is hot rolled from a metal *ingot*, with a rectangular cross section.

binary alloy. An alloy containing only two elemental components.

binary system. The complete series of compositions produced by mixing two components in all proportions.

binder. In powder technology, a cementing or gluing medium added to a powder mixture for the specific purpose of binding together powder particles.

binder metal. A metal used as a binder. An example would be cobalt in cemented carbides.

binder phase. The soft metallic phase that cements the carbide particles in cemented carbides. Also see *matrix*.

biocompatible. The ability of a material to be implanted into living tissue without being rejected.

biomaterials. A group of materials suited for implantation into living tissue. The materials may be either organic or inorganic.

bitumen. Various hydrocarbons and other substances occurring naturally or obtained by distillation from coal or petroleum. Found in asphalt and tar.

bituminous coating. Coal tar or asphalt-based coating.

black liquor. The liquid material remaining after pulpwood is cooked in the soda or sulfate papermaking process.

black oxide. A black metallic finish produced by immersion in hot oxidizing salts or salt solutions.

blasting or blast cleaning. A process for cleaning or roughening metal objects with an air blast or centrifugal wheel that forcibly directs abrasive particles against the workpiece.

blend (noun). Thoroughly intermingled powders of the same nominal chemical composition.

blend (verb). To mix. See *blending*.

blending. The thorough intermingling of powders of the same nominal chemical composition (not to be confused with *mixing*).

blind sample. A sample submitted for analysis whose composition is known to the submitter but unknown to the analyst; used to test the accuracy of measurement.

blister. A raised area, often dome shaped, resulting from loss of adhesion between the coating and the substrate.

block-on-ring. A specific type of wear test.

blow hole. A void produced by the outgassing of air during solidification of a fused, self-fluxing thermal spray alloy coating.

BN material. A material containing some form of boron nitride (BN). It may be among several crystallographic varieties, including hexagonal and cubic.

boat. A box or container used to hold powder during furnace passage.

body-centered. An atomic structure wherein a singular atom is at the center of the cell structure.

body stress. Residual stress within an individual sprayed particle.

bond. The junction between the coating and the substrate, or its strength. See also *adhesive bond*, *mechanical bond*, and *metallic bond*.

bond cap (bond bar). The test specimen on which a sprayed coating is applied for the purpose of determining adhesive-cohesive strength.

bond coat. A preliminary (or prime) coat of material that improves adherence of the subsequently sprayed deposit.

bonding. The joining together of two materials.

bond line. In a cross-sectional view, the demarcation at the interface between a thermal spray deposit and its substrate.

bond strength. The force required to pull a coating free of its substrate. Usually expressed in megapascals (MPa) or pounds per square inch (psi).

bore. A hole or cylindrical cavity produced by a single-point or multipoint tool other than a drill.

boron carbide. A black crystalline powder of high hardness having a composition of either B₆C or B₄C. The latter is a composite of B₄C and carbon in graphitic form.

boss. A relatively short protrusion or projection from the surface of a forging or casting, often cylindrical in shape. Usually intended for drilling and tapping for attaching other parts.

bottle. See preferred term *cylinder*.

bow. A condition of longitudinal curvature.

bowing. Deviation from flatness.

brake. A device for bending sheet metal to a desired angle.

brale indenter. A conical 120° diamond indenter used in certain types of Rockwell and scratch hardness tests.

brass. A copper alloy containing up to 40% Zn. See also *copper* and *copper alloys*.

brazing. A group of welding processes for joining materials by heating them to a suitable temperature and using a filler metal with a liquidus above 450 °C (840 °F) and below the solidus of the base materials. The filler metal is distributed between the surfaces of the joint by capillary attraction.

break in. To operate a newly installed component so as to condition its functional operation.

breaking load. The maximum load (or force) applied to a component and resulting in rupture or failure.

breaking stress. Same as *fracture stress*.

bridging. The unwanted adhesion of spray particles between mating surfaces of the substrate and masking.

bright-field illumination. In light microscopy, the form of illumination that causes spectrally reflected surfaces normal to the axis of the microscope to appear bright. For transmission electron microscopy, the illumination of an object so that it appears on a bright background. Compare with *dark-field illumination*.

brine. Water containing a higher concentration of dissolved salt than that of the ordinary seawater.

Brinell hardness number (HB). A number related to the applied load and to the surface area of the permanent impression made by a ball indenter.

Brinell hardness test. A test for determining the hardness of a material by forcing a hard steel or carbide ball of specified diameter (typically, 10 mm, or 0.4 in.) into it under a specified load. The result is expressed as the *Brinell hardness number*.

Brinelling. Indentation of the surface of a solid body by repeated local impact or impacts, or static overload.

British thermal unit (Btu). A unit of measure for heat (equal to 1055 J).

brittle. Permitting little or no plastic (permanent) deformation prior to fracture.

brittle fracture. Separation of a solid accompanied by little or no macroscopic plastic deformation.

brittleness. The tendency of a material to fracture without first undergoing significant plastic deformation. Contrast with *ductility*.

bronze. A copper-tin alloy with or without small proportions of other elements such as zinc and phosphorus. Also, other binary copper-base alloys containing no tin, such as aluminum bronze (copper-aluminum), silicon bronze (copper-silicon), and beryllium bronze (copper-beryllium). See also *copper* and *copper alloys*.

buildup. The construction or fabrication of a coating.

buildup sequence. The order in which the passes of a multiple-pass spray process are deposited. See also *spraying sequence*.

bulk density. Powder in a container or bin, expressed in mass per unit volume.

burning. (1) Permanently damaging a metal or alloy by overheating during spraying. (2) In grinding, when the work becomes so hot as to cause discoloration or to change its microstructure.

burn-in pass. The initial “hot” pass of an electric arc spray sequence where the gun is moved close to the part, depositing superheated, self-bonding particles.

burr. A thin, sharp ridge or roughness left on a workpiece resulting from cutting, punching, or grinding.

bushing. A bearing or guide.

C

calcination. Heating ores, concentrates, precipitates, or residues to decompose carbonates, hydrates, or other compounds.

calcine. (1) A ceramic material or mixture fired to less than fusion for use as a constituent in a ceramic composition. (2) Refractory material, often fireclay, that has been heated to eliminate volatile constituents and to produce desired physical changes.

calibrate. To determine, by measurement or comparison with a standard, the correct value of each scale reading on a measuring (test) instrument.

calibration. To *calibrate*.

camber. To deviate from straightness; usually refers to ceramic rods.

cap. See preferred terms *air cap* or *nozzle*.

carbide. A chemical compound formed between carbon and a metal or metals. Examples are tungsten carbide, tantalum carbide, titanium carbide, and chromium carbide.

carbonizing flame. See preferred term *reducing flame*.

carbon steel. Steel having no specified minimum for any alloying element—other than accepted amounts of manganese, silicon, and copper—and containing only slight amounts of any element other than carbon.

carburize. To diffuse carbon into a low-carbon steel. Usually followed by heat treating to harden the surface.

carburizing flame. A gas flame that will introduce carbon into some heated metals. A carburizing flame is a *reducing flame*, but a reducing flame is not necessarily a carburizing flame.

carrier gas. The gas used to convey powdered materials from the powder feeder to the spray gun.

case harden. To harden the surface of a low-carbon steel.

cast. The twist wrap or curvature of a flame spray wire.

cast iron. A generic term that identifies a family of cast ferrous alloys in which the carbon content exceeds 2% and contains from 1 to 3% Si.

catalyst. (1) A substance capable of changing the rate of a reaction without itself undergoing any net change. (2) A substance that markedly speeds up the *cure* of a plastic compound.

catastrophic failure. Sudden failure of a component or assembly.

cathode. The negative electrode of an electrolytic cell. In a plasma generator, it is the rear electrode.

cathodic protection. Partial or complete protection of a metal from corrosion by making it a cathode, using either a galvanic or an impressed current. Contrast with *anodic protection*.

cation. A positively charged *ion*.

caustic. (1) Burning or corrosive. (2) A hydroxide of a light metal, such as sodium hydroxide or potassium hydroxide.

cavitation. (1) The formation and collapse of cavities or bubbles in a liquid that contain vapor or gas or both. (2) The formation of cavities on a surface.

cavitation damage. The degradation of a solid body or coating resulting from its exposure to cavitation. This includes material lost, surface deformation, or changes in properties or appearance.

cavitation erosion. Progressive loss of material from a solid surface due to continuing exposure to *cavitation*.

cell. In a honeycomb core, a cell is a single honeycomb unit, usually in a hexagonal shape.

cell growth. See preferred term *crystallization*.

cell size. The diameter of an inscribed circle within a cell of *honeycomb* core.

cemented carbides. A class of wear-resistant, refractory materials in which the hard carbide particles are bound together, or cemented, by a soft and ductile metal binder, such as with tungsten carbide crystallites bonded with cobalt.

cementite. A hard (~800 HV), brittle compound of iron and carbon, known chemically as iron carbide and having the approximate chemical formula Fe_3C .

centerless grinding. Grinding the outside or inside diameter of a cylindrical piece that is supported on a work support blade instead of being held between centers and that is rotated by a regulating feed wheel.

ceramic (adjective). The ability to tolerate high temperature. See also *refractory*.

ceramics (noun). Any of a class of inorganic nonmetallic products that are subjected to a high-temperature exposure during manufacture or use (approximately 540 °C, or 1000 °F). Typically, but not exclusively, a ceramic is a metallic oxide, boride, carbide, or nitride, or a mixture of such materials. See also *advanced ceramics* and *refractory materials*.

ceramic-matrix composites. Candidate materials for high-performance engines and wear-resistant parts. Compositions vary with the application requirement; typically, they are oxides or carbides with reinforcing fibers or particles of similar composition.

ceramic-metal coating. A mixture of a ceramic material with a metallic phase.

ceramic rod flame spray gun. A flame spraying device wherein an oxyfuel gas flame provides the heat, and the surfacing material to be sprayed is in ceramic rod form.

ceramic rod flame spraying. A spraying process in which the material to be sprayed is in ceramic rod form. See also *flame spraying*.

ceramic rod speed. The length of ceramic rod sprayed in a unit of time.

cermet. An acronym used to designate a heterogeneous combination of metal(s) with a ceramic phase(s). Generally, there is little solubility between the metallic and ceramic phases during spraying.

chamfer. A beveled surface to eliminate an otherwise sharp corner.

chamfering. Making a sloping surface on the edge of a member. Also called *beveling*.

characteristic. A distinctive property, condition, or quality of an item that helps to distinguish between items.

Charpy test. A pendulum-type, single-blow impact test in which the specimen, usually notched, is supported at both ends as a simple beam and broken by the falling pendulum. The energy absorbed, as determined by the subsequent rise of the pendulum, is a measure of impact strength or notch toughness. Contrast with *Izod test*.

charring. The scorching of a plastic or rubber.

chatter. In machining or grinding, (1) a vibration of the tool, wheel, or workpiece, producing a wavy surface on the work, and (2) the finish produced by such vibration.

checks. (1) Very fine cracks often associated with thermal treatment or thermal cycling. May also result from poor grinding techniques that burn the coating. Also called check marks, checking, or *heat checks*. (2) A series of small cracks resulting from thermal fatigue.

chemical decomposition. The separation of a compound into its constituents.

chemically precipitated powder. Thermal spray powder produced as a precipitate by chemical displacement. See also *precipitation*.

chemical machining. Removing metal stock by controlled selective chemical dissolution.

chemical milling. A process in which metal is fashioned to shape by masking certain portions and then dissolving away the exposed (unwanted) metal.

chemical vapor deposition (CVD). (1) A coating process in which a reactant gas is fed into a processing chamber, where it decomposes on the workpiece, liberating material for absorption by, or accumulation on, the workpiece. A second gaseous material is liberated and removed from the chamber, along with excess starting gas. (2) Process in which a material is deposited from vapor phase onto a continuous core.

chevron blender. A V-shaped device for blending and/or mixing powders.

chilled iron. Cast iron for grit blasting.

chipping. Minor edge spalling of a sprayed coating.

chips. (1) Pieces of material removed from a coating by cutting tools or by an abrasive medium. (2) Small coating flakes spalled off either by external damage or poor application techniques.

chlorinated hydrocarbon. An organic compound having chlorine atoms in its chemical structure. Trichloroethylene, methyl chloroform, and methylene chloride are chlorinated hydrocarbon solvents; polyvinyl chloride is a plastic.

chromate treatment. A treatment of metal in a solution of a hexavalent chromium compound to produce a *conversion coating*.

Chromel. (1) A 90Ni10Cr alloy used in thermocouples. (2) A series of nickel-chromium alloys, some with iron, used for heat-resistant applications.

chrome plating. The electrodeposition of chromium metal from an aqueous solution containing chromic acid. Deposits may be used for wear resistance (hard chrome), corrosion resistance, or decorative purposes.

chromia. Also referred to as chromium oxide (Cr_2O_3), a compound having many properties and derivatives similar to those of *alumina*. Useful in both basic and high-alumina refractories.

chuck. A device for holding work or tools on a machine so that the part can be held or rotated during machining or grinding.

circuit breaker. A device that, when properly applied within its rating, is designed to open and close a circuit by nonautomatic means and to open the circuit automatically on a predetermined overload of current, without injury to itself.

cladding. The spraying process or the coating.

clad metal. Metal substrates with a thermal sprayed overlay.

clad powder. See preferred term *powder clad*.

classification. Separation of a powder into fractions according to particle size.

clay. A natural mineral aggregate consisting of hydrous aluminum silicates. It is plastic when sufficiently wetted, rigid when dried, and vitreous when fired at a sufficiently high temperature.

clean surface. A surface that is free of foreign material, both visible and invisible.

cleanup allowance. See preferred term *finish allowance*.

clearance. The gap or space between two mating parts.

clearance control coating system. A two-component thermal spray coating system consisting of (1) an abradable sealant and (2) an abrasive. The abradable is usually applied onto a static component, where it is rubbed or abraded by the moving abrasive-coated component.

clearance fit. Any of various classes of fit between mating parts where there is a positive allowance (gap) between the parts, even when they are made to the respective extremes of individual tolerances that ensure the tightest fit between the parts. Contrast with *interference fit*.

closed-loop control. Computerization that continuously monitors and controls application parameters to assure process repeatability.

closed porosity. Coating pores that are closed off by surrounding material and are inaccessible to each other and to the external surface. They are not detectable by gas or liquid penetration. Contrast with *interconnected porosity*.

cloth. Metallic or nonmetallic screen or fabric used for screening or classifying powders.

coalesce. To grow or come together; fuse; unite.

coalescence. The fusing together of sprayed particles to form a singular, homogeneous deposit; usually effected at temperatures above the melting range.

coarse fraction. The large particles in a powder.

coating. A layer of material applied by thermal spraying for the purpose of corrosion prevention, resistance to high-temperature scaling, wear resistance, lubrication, or other purposes.

coating density. The ratio of the determined density of a coating to the theoretical density of the material used in the coating process. Usually expressed as percent of *theoretical density*.

coating strength. A measure of cohesive bonding within a coating, as opposed to coating-to-substrate bonding (adhesive strength); coating tensile strength is usually expressed in kilopascals (kPa) or pounds per square inch (psi).

coating stress. The stresses in a coating resulting from rapid cooling and solidification of molten material or semimolten particles as they impact the substrate. Coating stresses are a combination of body and textural stresses.

cobalt and cobalt alloys. Tough, silver-gray magnetic metal that resembles iron and nickel in appearance and in some properties. Cobalt is useful in applications that use its magnetic properties, corrosion resistance, wear resistance, biocompatibility, and/or strength at elevated temperatures.

coefficient of elasticity. The reciprocal of Young's modulus in a tension test. See also *Young's modulus*.

coefficient of expansion. A measure of the change in length or volume of an object, specifically, a change measured by the increase in length or volume of an object per unit length or volume.

coefficient of friction. The dimensionless ratio of the friction force (F) between two bodies to the normal force (N) pressing these bodies together: μ (or f) = (F/N).

coefficient of thermal expansion (CTE). (1) Change in unit of length (or volume) accompanying a unit change of temperature, at a specified temperature. (2) The linear or volume expansion of a given material per degree rise of temperature, expressed at an arbitrary base temperature or as a more complicated equation applicable to a wide range of temperatures.

cohesion. The state in which the particles of a thermal spray coating are bound together either mechanically or metallurgically.

cohesive failure. Failure wholly within the coating material.

cohesive strength. Intrinsic strength of a thermal spray coating.

coil. Rolled metal sheet, strip, or wire.

cold gas dynamic spray method (CGSM). See preferred term *cold spray*.

cold shut. A lap on the surface of a wire or wrought product that was closed without fusion during deformation.

cold spray. A kinetic spray process using supersonic jets of compressed gas to accelerate near-room-temperature powder particles at ultrahigh velocities. The unmelted particles, traveling at speeds between 500 to 1500 m/s (1600 to 5000 ft/s), plastically deform and consolidate on impact with their substrate to create a coating.

cold work. Forming or rolling performed at low temperatures (less than 200 °C, or 390 °F), resulting in permanent strain to a metal, accompanied by strain hardening.

cold working. Deforming metal plastically under conditions of temperature and strain rate that induce hardening. Usually, but not necessarily, conducted at room temperature. Contrast with *hot working*.

collaring. Adding or cutting a shoulder onto a shaft or similar component to provide a confining wall for the spray deposit.

collet. A split sleeve used to hold work or tools during machining or grinding.

colloidal. A state of suspension in a liquid medium in which extremely small particles are suspended and dispersed but not dissolved.

columnar structure. Often observed in castings, it is a coarse structure of parallel elongated grains formed by unidirectional growth.

comminution. Reducing to powder by mechanical means. See also *pulverization*.

compaction. The settling of a spray powder either in its storage container or feeder to form a relatively dense nonflowable mass.

companion panel. A small tab coated concurrently with the workpiece and used for inspection.

complete fusion. Fusion that has occurred within the entire deposit.

component. (1) One of the elements or compounds used to define a chemical (or alloy powder) system. (2) A mechanical part, assembly, or total system.

composite. See *composite material*.

composite coating. (1) A coating that consists of two or more components blended or fully integrated with each other. (2) A coating system consisting of many distinguishable layers. Sometimes referred to as a multilayer coating.

composite material. A combination of two or more materials in intimate contact with each other but differing in form or composition on a macroscale. Constituents retain their identities and do not dissolve or completely merge into one another, although they act in concert. Normally, the components can be physically identified and exhibit an interface at their juncture.

composite powder. A powder in which each particle consists of two or more different materials. See preferred term *powder composite*.

composite structure. A coating that is built up by codepositing together two or more distinct components, each of which may be made of a metal, alloy, nonmetal, or *composite material*.

compound. A chemical substance of relatively fixed composition and properties, whose ultimate structural unit (molecule or repeat unit) is comprised of atoms of two or more elements.

compressed-air mask. A force-feed type of face mask, with a suitable regulator, worn by a spray operator to provide a fresh air supply.

compression test. A method for assessing the ability of a material to withstand compressive loads.

compressive. Pertaining to forces on a body, or part of a body, that tend to crush or compress the body.

compressive strength. The maximum compressive stress that a material is capable of supporting, based on original area of cross section.

compressive stress. A stress that causes an elastic body to deform (shorten) in the direction of the applied load. Contrast with *tensile stress*.

concentration. The amount of a specific substance in a unit amount of another substance.

cone. The conical part of an oxyfuel gas flame next to the orifice of the tip.

conformal coating. A coating that covers and exactly fits the shape of the coated object.

connected porosity. The pores, voids, and channels within a coating that are interconnected with each other and communicate with the external surface and are measurable by gas or liquid penetration. Contrast with *closed porosity*.

constituent. (1) Any of the ingredients that make up an alloy or chemical system. (2) A phase or a combination of phases occurring in a coating. (3) In powder mixtures, constituents are the various components.

container. The vessel into which thermal spray powder is offered, delivered, and/or stored.

continuous phase. In a coating or in an alloy that contains more than one phase, it is that portion that forms the matrix in which the other phase or phases are dispersed.

continuous sequence. The uninterrupted application of a coating onto a part. Spraying is continuous across the part until the total required thickness is achieved.

contraction. The volume change that occurs in metals and alloys on solidification and cooling to room temperature.

control console. The instrumented unit from which the spray gun is operated and the operating variables are monitored and controlled.

controlled atmosphere chamber. A gas-filled (usually inert) enclosure or cabinet in which plasma or electric arc spraying can be performed to minimize (or prevent) oxidation of the coating or substrate.

controlled cooling. Cooling substrate or coating in a predetermined manner to avoid internal damage or spalling of the coating.

conversion coating. A coating consisting of a compound of the surface metal, produced by chemical or electrochemical treatments of the metal. See also *chromate treatment* and *phosphating*.

convoluted. The rolling over of sprayed particles and oxides around themselves and adjacent particles. See also *undulating*.

coolant. The liquid used to cool the workpiece during machining or grinding and to prevent it from rusting. It also lubricates, washes away chips and grits, and aids in obtaining a finer finish. In metal cutting, the preferred term is cutting fluid.

cooler. See preferred term *auxiliary cooler*.

cooling stresses. Residual stresses in coatings resulting from nonuniform distribution of temperature during cooling or from different coefficients of thermal expansion.

copper and copper alloys. A reddish-brown metal that is soft, ductile, and malleable, exhibiting excellent thermal and electrical conductivity. Sprayed coatings are generally used for bearing and antifretting applications.

copper plating. To clad with copper by electrolytic deposition.

cord. A plastic tube filled with powder and extruded to form a compact, flexible, level-wound, wirelike cord.

cored wires or rods. Multicomponent composite wire consisting of an outer sheath surrounding one or more powdered metal/ceramic/plastic elements.

coring. A condition of variable chemical composition within a microstructure (such as a dendrite, grain, carbide particle); results from nonequilibrium solidification. Often found in fused coatings.

corona. The edge or ring surrounding a flattened splat when viewed in the normal direction.

corrosion. The chemical or electrochemical reaction between a material, usually a metal, and its environment that produces a deterioration of the material and its properties.

corrosion effect. A change in any part of the *corrosion system* caused by *corrosion*.

corrosion-erosion. See *erosion-corrosion*.

corrosion fatigue. The fracture of a metal under simultaneous conditions of corrosion and cyclic loading. Normally at lower stress levels and/or cycles than would be anticipated in the absence of the corrosive medium.

corrosion inhibitor. See *inhibitor*.

corrosion product. Substance formed as a result of *corrosion*.

corrosion protection. Modification of a *corrosion system* so that corrosion damage is avoided or lessened.

corrosion rate. *Corrosion effect* on a metal per unit of time. It may be expressed as corrosion depth per unit of time (penetration rate, for example, mils/yr) or the mass of metal turned into corrosion products per unit area of surface per unit of time (weight loss, for example, g/m²/yr). Corrosion rate reports should contain information on the type, time dependency, and location of the corrosion effect.

corrosion resistance. The ability of a material to withstand contact with ambient natural factors or those of a particular, artificially created atmosphere, without degradation or change in properties. For metals, this could be pitting or rusting; for organic materials, it could be crazing.

corrosion system. A system consisting of one or more metals and all parts of the environment that influence *corrosion*.

corrosive wear. *Wear* in which chemical or electrochemical reaction with the environment is significant. See also *oxidative wear*.

corrosivity. Tendency of an environment to cause *corrosion* in a given *corrosion system*.

corundum. A naturally occurring *alumina* usually of relatively high purity. Corundum and its artificial counterparts are abrasives especially suited to the grinding and blasting of metals.

cosmetic pass. A spray pass made primarily for the purpose of enhancing appearance.

cospray. Thermal spraying of two or more dissimilar materials through a single gun using multiple insertion points.

Coulter counter. An apparatus for measuring the number and size of powder particles.

countersinking. Beveling or tapering material around the periphery of a hole, creating a concentric surface at an angle less than

90° with the centerline of the hole for the purpose of chamfering holes or recessing screw and rivet heads.

couple. See *galvanic corrosion*.

coupon. A piece of material from which a test specimen is to be prepared. A coupon will experience the same processing steps as the part it represents. See also *companion panel*.

crack. (1) A fracture-type discontinuity characterized by a sharp tip and high ratio of length to width. (2) A line of fracture without complete separation.

crack extension. An increase in crack size.

crack growth. Rate of propagation of a crack through a coating due to an applied stress.

crack size. A lineal measure of a crack.

craze cracking. Irregular surface cracking of a metal associated with thermal cycling.

crazing. The cracking that occurs in metallic or ceramic coatings due to critical tensile stresses.

creep. Time-dependent strain occurring under stress.

critical dimension. A dimension on a part that must be held within the specified tolerance for the part to function in its application.

crown. In an as-sprayed coating, the thickest portion, usually the centermost section of a single pass.

crushing. A process of comminuting large pieces of a material into rough-sized fractions prior to grinding into powder.

crystal. A three-dimensional atomic, ionic, or molecular structure consisting of one specific orderly geometrical array, periodically repeated and termed lattice or unit cell.

crystal growth. See preferred term *crystallization*.

crystalline. That form of a substance that is predominantly comprised of crystals, as opposed to glassy or amorphous.

crystallite. A crystalline grain not bounded by habit planes.

crystallization. (1) The precipitation, usually from a liquid phase on cooling, to a solid crystalline phase. (2) The progressive process in which crystals are first nucleated (started) and then grown in size within a host medium that supplies their atoms.

cubic. Having three mutually perpendicular axes of equal length.

cubic boron nitride (CBN). An extremely hard ceramic material synthesized by high-pressure sintering of hexagonal boron nitride. CBN is used in the machining and grinding of hardened materials. See also *superabrasives*.

cubicle. See preferred term *acoustical room*.

cup test. An adherence test wherein a test coupon is laid on a cupped die and a 22 mm (7/8 in.) ball depressed on it. A cup shall be drawn so that the coating is on the outside of the cup. The coating should not exhibit chipping or spalling to be accepted.

cure. (1) To change the physical properties of a material by chemical reaction or by the action of heat and catalysts, alone or in combination, with or without pressure. (2) To irreversibly change, usually at elevated temperatures, the properties of a thermosetting resin by chemical reaction.

curing temperature. The temperature to which an adhesive or an assembly is subjected to cure the adhesive.

curing time. In adhesive bonding, the time at temperature or the duration required to cure an adhesive.

current. The net transfer of electric charge per unit time. Also called electric current. See also *current density*.

current density. The current flowing to or from a unit area of an electrode surface.

cutoff wheel. A thin abrasive wheel for severing or slotting a material or a part.

cycle. The time required to achieve a final coating. Includes cleaning, masking, roughening, positioning, spraying, clean up, and finishing.

cyclone. In powder metallurgy, a collector of fractions of a fluidized powder; the device operates with the aid of centrifugal force that acts on the powder suspension in the fluid.

cylinder. A portable container used for the transportation and storage of a compressed gas. Used in spraying, welding, and cutting operations.

cylindrical grinding. Grinding the outer cylindrical surface of a rotating part.

D

D-gun. (Praxair Surface Technologies). Trade name of a proprietary coating device and process. Uses controlled detonations (hence, D-gun) to apply coatings. Formerly referred to as flame plating.

dark-field illumination. In light microscopy, the illumination of an object so it appears bright and the surrounding field dark. This results from illuminating the object with rays of sufficient obliquity so that none can enter the objective directly. In electron microscopy, the image is formed using only electrons scattered by the object. Contrast with *bright-field illumination*.

debond. An interface separation of a coating from its substrate. Also, an unbonded or nonadhered region; a separation due to strain incompatibility. See also *disbond* and *delamination*.

debris. See *wear debris*.

deburring. Removing burrs, sharp edges, or fins from metal parts by filing, grinding, or tumbling. Sometimes called *burring*.

decagonal. A ten-sided crystal structure.

decarburization. Loss of carbon from the surface of a carbon-containing alloy or carbide coating due to overheating or chemical interaction.

decibel (dB). Unit that expresses differences in the level of noise.

decomposition. Breakdown or separation of a compound into its chemical elements or components.

defect. A condition or variance from a standard or accepted norm. Defects may include cracks, delamination, alligatoring, brittleness, bubbles, burrs, checks, chipping, debonding, and so on. This term does not designate rejectability but rather an imperfection. See also *discontinuity* and *flaw*.

defective. A quality-control term describing a unit of product or service containing at least one defect, or having several lesser imperfections that, in combination, cause the unit not to fulfill its anticipated function. The term “defective” is not synony-

mous with nonconforming (or rejectable) and should be applied only to those units incapable of performing their anticipated functions.

deformation. A change in the form of a body due to stress, thermal change, change in moisture, or other causes. Measured in units of length.

degradation. A deleterious change in the chemical structure, physical properties, or appearance of a material.

degrease. To remove oil or grease from adherent surfaces. See also *solvent cleaning/degreasing*.

degreasing. Removing oil or grease from a surface. See also *vapor degreasing*.

delamination. The separation of a coating either within itself or from its substrate.

delamination wear. A wear process in which thin layers of material are formed and removed from the wear surface.

deliquescent. The process of melting or becoming liquid by absorbing moisture from the air.

demixing. The undesirable separation of one or more constituents of a powder mixture; sometimes due to overmixing.

dendrite. A crystal that has a treelike branching pattern, being most evident in cast metals slowly cooled through the solidification range.

dendritic powder. Particles usually of electrolytic origin typically having the appearance of a pine tree.

densify. To make dense as by *sintering* or *plasma densification*.

density. The mass of a substance per unit volume, expressed as grams per cubic centimeter (g/cm^3) or pounds per cubic inch (lb/in.^3).

depletion. Selective removal of one component of an alloy, usually from the surface or preferentially from grain-boundary regions.

deposit. See preferred term *spray deposit*.

deposition. The process of applying a sprayed material to a substrate.

deposition efficiency (DE). The ratio, usually expressed in percent, of the weight of spray deposit to the weight of the material sprayed.

deposition rate. The weight of material deposited in a unit of time. It is usually expressed as kilograms per hour (kg/h) or pounds per hour (lb/h).

deposition sequence. The order in which the increments of sprayed material are deposited. See also *buildup sequence*.

deposit sequence. See preferred term *deposition sequence*.

depth of field. The depth in the subject over which features can be seen to be acceptably in focus in the final image produced by a microscope.

descaling. Removing the oxide layers (scale) found on some metals; removal may be by chemical or mechanical means.

desiccant. A chemical, with an affinity for water, used to attract and remove moisture from air or gas.

design of experiment (DOE). A statistical technique using various levels (versions) of critical operating factors (parameters)

- or combinations of factors to optimize or produce a desired end result.
- detergent.** A chemical substance, generally used in aqueous solution, that removes surface contamination.
- determination.** The ascertainment or defining of the quality, quantity, position, or characteristics of a process, coating, or raw material. See also *analysis*.
- detonation flame spraying.** A thermal spraying process variation in which the controlled explosion of a mixture of fuel gas, oxygen, and powdered coating material is used to melt and propel the material to the workpiece.
- detritus.** See *wear debris*.
- deviation.** The difference between a measurement and its intended value.
- dewpoint.** Temperature at which liquid will condense from vapors.
- dezincification.** Commonly found in copper-zinc alloys (brasses) containing less than 85% Cu. A form of corrosion in which zinc is selectively leached out, leaving a relatively weak layer of copper and copper oxide.
- diagnostic.** The determination or identification of problems or faults related to a coating or its application.
- diamond pyramid hardness test.** See *Vickers hardness test*.
- diamond tool.** An insert, made from polycrystalline diamond compacts, shaped or formed to the contour of a single-point cutting tool for use in precision machining.
- diamond wheels.** A grinding wheel in which crushed and sized industrial diamonds are held in a resinoid, metal, or vitrified bond.
- diaphragm.** (1) A fixed or adjustable aperture in an optical system used to intercept scattered light, limit field angles, or limit image-forming bundles or rays. (2) A component of a gas regulator for controlling the pressure and quantities of released gases.
- die.** A tool, usually containing a cavity, that imparts shape to solid, molten, or powdered metal. Used in many press operations, in die casting, and in forming green powder metallurgy compacts. Die casting and powder metallurgy dies are sometimes referred to as molds.
- dielectric.** (1) A nonconductor of electricity. (2) The ability of a material to resist the flow of an electrical current.
- dielectric shield.** In a cathodic protection system, an electrically nonconductive material, such as a coating, that is placed between an anode and an adjacent cathode to avoid current wastage and to improve current distribution, usually on the cathode.
- dielectric strength.** (1) The maximum voltage that a dielectric can withstand without resulting in a voltage breakdown. (2) A measure of the ability of a dielectric (insulator) to withstand a potential difference across it without electric discharge.
- differential thermal analysis (DTA).** A technique in which a specimen and a control are heated simultaneously, and the difference in their temperatures is monitored. The difference in temperatures provides information on relative heat capacities (melting ranges), changes in structure, and chemical reactions.
- diffusion.** The spontaneous movement of atoms or molecules to new sites within a material.
- diffusion coating.** (1) A coating that is thermally treated, usually in a controlled atmosphere, to cause a diffusion between it and its substrate. Diffusion occurs at the interface. (2) Immersing a metal in a heated gaseous or liquid medium, containing another metal, to simultaneously apply a coating and diffuse it into the base metal.
- dilatometer.** An instrument for measuring the linear movement in a metal resulting from changes such as temperature and allotropy.
- dimensional stability.** A measure of dimensional change caused by factors such as temperature, humidity, chemical treatment, age, or stress.
- dimension change.** Shrinkage or growth resulting from changes in temperature or stress.
- directionally solidified castings.** Castings produced by *directional solidification* that exhibit an oriented columnar structure.
- directional solidification.** Controlled solidification of molten metal in a casting to provide feed metal to the solidifying front of the casting.
- disbond.** The loosening of a coating from its substrate.
- discontinuity.** (1) An interruption in the typical structure of a coating, such as a lack of homogeneity in the mechanical, metallurgical, or physical characteristics of the coating. A coating discontinuity is not necessarily a defect. (2) Any interruption in the normal structure or configuration of a part, such as cracks, laps, seams, inclusions, or porosity. A discontinuity may or may not affect the utility of the part. See also *defect* and *flaw*.
- distortion.** Any deviation from an original size, shape, or contour that occurs due to the application of stress or the release of residual stress.
- distribution.** (1) The dispersion of phases or components within a powder or coating. (2) Particle size or range in a powder. (3) In statistics, a set of numbers collected from a well-defined universe of possible measurements arising from a property.
- dovetailing.** A method of surface preparation involving angular undercutting to interlock the spray deposit.
- dress.** Cutting, breaking down, or crushing the surface of a grinding wheel to improve or restore its cutting quality.
- drill.** A rotary end-cutting tool used for making holes.
- drilling.** Holmaking using a rotary end-cutting tool.
- dross.** Slag that forms on the surface of molten metal due to oxidation and/or the surfacing of impurities.
- drum.** An enclosed cylindrical container into which a continuous length of wound or coiled thermal spray wire is packaged.
- dry.** To store a powder at elevated temperature; to drive off water moisture for the purpose of enhancing flow.
- drying.** Removal, by evaporation, of uncombined water or other volatile substance from a powdered material. Usually expedited by low-temperature heating.

dry lubricant. See preferred term *solid lubricant*.

dry objective. A microscope objective lens designed for use without liquid between the specimen and the objective lens.

ductile iron. A *cast iron* that, while molten, has been doped with another element to induce the formation of graphite nodules or spherulites, which impart a measurable degree of ductility to the cast metal. They typically contain 3.0 to 4.0% C and 1.8 to 2.8% Si. Also called nodular cast iron.

ductility. The ability of a material to deform plastically without fracturing.

dullness. A lack of surface gloss or shine.

duplex alloys. Bearing alloys consisting of two phases, one much softer than the other.

duplex coating. A coating consisting of two layers of two dissimilar materials.

duplicate sample. A second sample, randomly selected from a population, to assist in evaluating sample variance.

durometer reading. Index used to rank the relative hardness of elastomers. The test forces a 30° tapered indenter into the specimen surface using calibrated loading springs. A dial indicates the depth of penetration in durometer numbers.

dust. A superfine spray powder having predominantly submicron-sized particles.

dwell time. The length of time in which the sprayed particles remain in an optimized heat zone.

E

ear protection. A device approved by the U.S. Occupational Safety and Health Administration (OSHA) or other safety agency for the reduction of sound audible to the outer ear.

eccentric. The offset portion of a driveshaft that can impart a reciprocating motion.

edge effect. Adhesional bond loss between the spray deposit and the substrate along the workpiece edges. Mainly due to cooling stresses.

edge loss. Spray material lost as overspray resulting from spraying beyond the workpiece edge.

effector. The integrating device or attachment surface for mating a thermal spray gun or grit-blast nozzle to a manipulation unit such as an articulated robot.

elasticity. Property of certain materials enabling them to return to their original dimensions after the removal of an applied stress.

elastic modulus. The ratio of stress, within the proportional limit, to the corresponding strain for uniaxial loading.

elastomer. Any elastic, rubberlike substance, such as natural or synthetic rubber.

electrical conductivity. The reciprocal of volume resistivity. The electrical or thermal conductance of a unit cube of any material (conductivity per unit volume).

electrical discharge grinding (EDG). Grinding by spark discharges between a negative electrode grinding wheel and a positive workpiece separated by a small gap containing a dielectric fluid such as petroleum oil.

electrical discharge machining (EDM). Metal removed by a rapid spark discharge between different polarity electrodes, one the workpiece, the other the tool. They are separated by a narrow gap, 0.013 to 0.9 mm (0.0005 to 0.035 in.), which is filled with dielectric fluid.

electrical insulator. That component of a thermal spray device that inhibits electrical contact or flow.

electric arc spraying. A thermal spraying process in which an electric arc is struck between two consumable electrodes of a coating material. Compressed gas is used to atomize and propel the material to the substrate.

electric bonding. See preferred term *surfacing*.

electrochemical corrosion. Corrosion that is accompanied by a flow of electrons between cathodic and anodic areas on metallic surfaces.

electrochemical machining (ECM). Controlled metal removal by anodic dissolution. Direct current passes through a flowing film of conductive solution that separates the workpiece from the electrode-tool. The workpiece is the *anode*, and the tool is the *cathode*.

electrochemical reaction. A reaction caused by passage of an electric current through a medium that contains mobile ions (as in electrolysis); or, a spontaneous reaction made to cause current to flow in a conductor external to this medium (as in a galvanic cell). In either event, electrical connection is made to the external portion of the circuit via a pair of electrodes.

electrocorrosive wear. Wear of a solid surface that is accelerated by the presence of a corrosion-inducing electrical potential across the contact interface. Moisture from the air can facilitate this type of corrosion wear when a galvanic couple exists and the contacting materials are sufficiently reactive.

electrode. In arc and plasma spraying, the current-carrying components that support the arc. See also *anode* and *cathode*.

electrode cable. Same as *electrode lead*.

electrode holder. A device used for mechanically holding the electrode while conducting current to it.

electrode lead. The electrical conductor between the electrical source and the electrode holder.

electroless plating. The deposition of a conductive material from an autocatalytic plating solution without the application of electrical current.

electrolysis. Chemical change resulting from the passage of an electric current through an *electrolyte*.

electrolyte. A conducting medium in which the flow of current is accompanied by the movement of matter.

electrolytic grinding. A combination of grinding and machining where the *cathode*, a metal-bonded abrasive wheel, is in physical contact with the anodic workpiece; contact is made beneath the surface of an electrolyte. The abrasive particles of the wheel perform grinding and act as nonconducting spacers, permitting simultaneous machining through electrolysis.

electrolytic machining. Controlled removal of metal by use of an applied potential and a suitable electrolyte.

electrolytic powder. Powder produced by electrolytic deposition or by pulverizing of an electrodeposit.

electromagnetism. Magnetism caused by the flow of an electric current.

electromotive force (EMF). (1) The force that determines the flow of electricity; a difference of electric potential. (2) Electrical potential; voltage.

electron. An elementary particle that is the negatively charged constituent of ordinary matter.

electron beam (EB). A stream of electrons in an electron-optical system.

electron microscope. An electron-optical device that produces a highly magnified image of an object. See also *scanning electron microscope* and *transmission electron microscopy*.

electron microscopy. The study of materials by means of an electron microscope.

electrophoresis. Transport of charged colloidal or macromolecular materials in an electric field.

electroplate. The application of a metallic coating on a surface by means of electrolytic action.

electroplating. The electrodeposition of an adherent metallic coating for the purpose of securing a surface with properties or dimensions different from those of the base metal.

electropolishing. A metallographic technique used to prepare specimens, in which a high polish is produced by making the specimen the anode of an electrolytic cell, where preferential dissolution at high points smoothes the surface. Also referred to as electrolytic polishing.

elongation. (1) A mechanical testing term describing the amount of testpiece extension when it is stressed. (2) In tensile testing, the increase in specimen gage length, measured after fracture, and usually expressed as a percentage of the original gage length. See also *elongation, percent*.

elongation, percent. The extension of a uniform section of a uniaxial loaded specimen expressed as a percentage of the original gage length:

$$\text{Elongation, \%} = \frac{(L_x - L_o)}{L_o} \times 100$$

where L_o is the original gage length, and L_x is the final gage length.

embrittlement. The loss of ductility and/or toughness.

emery. A naturally occurring, impure form of *alumina*, generally less pure than *corundum*.

encapsulation. To enclose an item in another substance. Sometimes used specifically in reference to the enclosure of powder particles with a metal.

enclosure. See preferred term *acoustical room*.

end milling. Machining with a rotating cutting tool with cutting edges on both the face end and the periphery. See also *face milling* and *milling*.

endothermic atmosphere. A gas mixture produced by the partial combustion of a hydrocarbon gas with air in an endothermic reaction.

endothermic compounds. A substance that absorbs moisture from the air and is consumed in the process. The action is termed deliquescence. Typical compounds are potassium carbonate and sodium chloride.

endothermic reaction. Designating or pertaining to a reaction that involves the absorption of heat. See also *exothermic reaction*.

endurance limit. The maximum stress that a material can withstand for an infinitely large number of fatigue cycles. See also *fatigue limit* and *fatigue strength*.

energy-dispersive spectroscopy (EDS). A method of x-ray analysis that discriminates by energy levels the characteristic x-rays emitted from the sample.

engineering adhesive. An ultrahigh-strength bonding agent. The term differentiates such bonding agents from glues used to join paper and other nondurables.

engineering ceramics. Same as *advanced ceramics*.

engineering plastics. A general term covering all plastics, with or without fillers or reinforcements, that have properties suitable for use as construction materials, machine components, and chemical processing equipment components.

environment. The aggregate of all conditions (such as contamination, temperature, humidity, radiation, magnetic and electric fields, shock, and vibration) that externally influence the performance of a material or component.

epoxy plastic. A thermoset polymer containing one or more epoxide groups and curable by reaction with amines, alcohols, phenols, carboxylic acids, acid anhydrides, and mercaptans.

epoxy resin. A viscous liquid or the brittle, solid-containing epoxide groups that can be cross linked into final form by means of a chemical reaction with a variety of setting agents.

equiaxed grain structure. A microstructural characteristic wherein the grains have approximately the same dimensions in all directions.

equilibrium. The dynamic condition of physical, chemical, mechanical, or atomic balance that appears to be a condition of steady state rather than one of chemical change or mechanical acceleration.

equilibrium diagram. A graph of the temperature, pressure, and composition limits of phase fields in an alloy system as they exist under conditions of thermodynamical equilibrium. Compare with *phase diagram*.

erosion. (1) Loss of material from a solid surface due to relative motion in contact with a fluid that contains solid particles. (2) Progressive loss of material from a solid surface due to mechanical interaction between that surface and a fluid, a multicomponent fluid, an impinging liquid, or solid particles. (3) Loss of material from an electrical contact surface due to an electrical discharge (arcing). See also *cavitation erosion* and *erosion-corrosion*.

erosion-corrosion. A joint action involving corrosion and erosion in the presence of a moving corrosive fluid, leading to the accelerated loss of material.

erosion rate. Any determination of the rate of loss of material (erosion) with exposure duration. In certain contexts, it is given by the slope of the cumulative erosion-time curve.

erosive wear. See *erosion*.

erosivity. The characteristic of a collection of particles, liquid stream, or a slurry that expresses its tendency to cause erosive wear when forced against a solid surface under relative motion.

error. Deviation from the correct value.

estimate. An approximated calculation.

estimation. Making a statistical approximation about a numerical value.

etchant. (1) A chemical solution used to etch a metal to reveal structural details. (2) A solution used to remove, by chemical reaction, the unwanted portion of material from a surface.

etching. Subjecting the surface of a metal to preferential chemical or electrolytic attack; to etch.

eutectic. (1) An isothermal reversible reaction in which a liquid solution is converted into two or more intimately mixed solids on cooling, the number of solids formed being the same as the number of components in the system. (2) An alloy having the composition indicated by the eutectic point on a phase diagram. (3) An alloy structure of intermixed solid constituents formed by a eutectic reaction, often in the form of regular arrays of lamellae or rods.

eutectic melting. Melting of localized microscopic areas whose composition corresponds to that of the eutectic in the system.

eutectoid. (1) An isothermal reversible reaction in which a solid solution is converted into two or more intimately mixed solids on cooling, the number of solids formed being the same as the number of components in the system. (2) An alloy having the composition indicated by the eutectoid point on a phase diagram. (3) An alloy structure of intermixed solid constituents formed by a eutectoid reaction.

eutectoid point. The composition of a solid phase that undergoes univariant transformation into two or more other solid phases on cooling.

evaluation. An examination or test to determine conditions or properties.

evaporation. The vaporization of a material by heating. It may be accomplished in a vacuum.

evaporative deposition. The techniques of condensing a thin film of material on a substrate while in vacuum. The source material may be heated by bombardment with electrons (electron beam) or by thermal-conduction techniques.

exfoliation. The lifting or separation of a coating from its substrate.

exhaust booth. A system of individual units that function collectively to gather expended spray gases and airborne oversprayed (nonadherent) material(s) while simultaneously flushing and renewing the air in the working environment.

exotherm. The temperature/time curve of a chemical reaction or a phase change giving off heat. The amount of heat given off.

exothermic. Characterized by the liberation of heat.

exothermic reaction. A reaction that liberates heat, such as the spraying of NiAl.

extractive metallurgy. The branch of *process metallurgy* dealing with the separation of metals from their ores.

extrusion. The conversion of an ingot or billet into lengths of uniform cross section by forcing metal to flow plastically through a die orifice.

eyepiece. A lens or system of lenses for increasing magnification in a microscope by magnifying the image formed by the objective.

eye protection. Proper helmets, face masks, or goggles required to protect the eyes from ultraviolet and infrared radiation during thermal spraying or welding.

F

face milling. Milling a surface that is perpendicular to the cutter axis. See also *milling*.

face shield. A device positioned in front of the eyes and a portion of, or all of, the face. Its predominant function is to protect the eyes and face during spraying or welding.

facing. See preferred term *hardfacing*.

failure. A general term used to imply that a part in service (a) has become completely inoperable, (b) is still operable but incapable of satisfactorily performing its intended function, or (c) has deteriorated seriously, to the point that it has become unreliable or unsafe for continued use.

failure criteria. The limiting conditions relating to deviation from the characteristic value due to changes resulting from stress or environmental conditions.

failure mechanism. A structural or chemical process, such as corrosion or fatigue, that causes failure.

fatigue. The phenomenon leading to crack initiation, crack growth, or fracture under repeated or fluctuating stresses having a maximum value less than the ultimate tensile strength of the material. Fatigue cracks leading to fractures are progressive, starting as minute cracks that grow under the action of the fluctuating stress.

fatigue failure. Failure that occurs when a component undergoing fatigue completely fractures into two parts, has softened, or been otherwise significantly reduced in stiffness by thermal heating or cracking.

fatigue life (N_f). (1) The number of loading cycles of stress, strain, or temperature of a specified character that a given component sustains before failure of a specified nature occurs. (2) The number of deformation cycles required to bring about failure under a given set of oscillating conditions (stresses, strains, or temperatures).

fatigue limit. The maximum stress that presumably leads to fatigue fracture in a specified number of stress cycles. See also *endurance limit*.

fatigue strength. The maximum cyclical stress a material can withstand for a given number of cycles before failure occurs.

fatigue test. A method for determining the range of alternating (fluctuating) stresses a material can withstand without failing.

feathering. Tapering or blending the edges of a coating to near-zero thickness.

feedability. The ability of a powder to flow.

feeder. See *feed hopper*.

feed hopper. A container used for holding and metering powder for delivery to the spray device.

feeding. Conveying powder or wire to the thermal spray gun.

feed lines. Hoses that convey powder to the spray device.

feed rate. The rate at which material passes through the spray device in a given length of time. A synonym for spray rate.

feedstock. Any consumable form of spray material used in the application of a coating.

ferrite. Essentially, a carbon-free solid solution in which alpha iron is the solvent and that is characterized by a body-centered cubic crystal structure.

ferrites. A term referring to magnetic oxides in general, and especially to material having the formula MFe_2O_3 , where M is a divalent metal ion or a combination of such ions.

ferroalloy. An alloy of iron that contains a sufficient amount of one or more other chemical elements to be useful as an agent for introducing these elements into molten metal, especially into steel or cast iron.

ferrous. Metallic materials in which the principal component is iron.

fiber. A general term used to refer to filamentary materials.

fiberglass. An individual filament made by drawing molten glass.

fillet. A concave radius (curvature) imparted to inside meeting surfaces.

finer. (1) The product that passes through the finest screen in sorting crushed or ground material. (2) The portion of a powder composed of particles smaller than a specified size, usually 44 μm (–325 mesh).

finish. Surface condition, quality, or appearance of a coating or a surface.

finish allowance. Amount of excess stock left on the surface of a coating for machining.

finish grinding. The final grinding action on a workpiece, of which the objectives are surface finish and dimensional accuracy.

finish machining. A machining process analogous to finish grinding.

firebrick. A *refractory* brick, often made from fireclay, that is able to withstand high temperature (1500 to 1600 °C, or 2700 to 2900 °F) and is used to line furnaces, ladles, or other molten metal containment components.

Fisher subsieve sizer. Air permeability method of measuring average powder particle size.

fixture. A device designed to hold parts to be coated or joined in proper relation to each other.

fixturing. The placing of parts in a constraining or semiconstraining apparatus.

flake powder. Flat or scalelike particles whose thickness is small compared to the other dimensions.

flaking. The removal of material from a coating in the form of flakes or scalelike particles.

flame hardening. Localized heating of a medium carbon steel surface to above 900 °C (1650 °F), using a gas flame, then quenching it to produce a hard martensitic structure.

flame plating. Early term used to identify the *D-gun* thermal spray coating process.

flame spraying. Thermal spraying in which a coating material is fed into an oxyfuel gas flame, where it is melted. Compressed gas may or may not be used to atomize the coating material and propel it onto the substrate. The sprayed material is originally in the form of wire or powder. The term “flame spraying” is usually used when referring to a combustion-spraying process, as differentiated from *plasma spraying*.

flammability. Measure of the extent to which a material will support combustion.

flashback. A recession of a flame into or back of the mixing chamber of the spray gun or torch.

flashback arrestor. A device incorporated into an oxyfuel torch or spray gun to limit damage from a *flashback* by preventing propagation of the flame front beyond the point at which the arrestor is installed.

flash coat. A thin metallic coating usually less than 0.05 mm (0.002 in.) in thickness.

flash point. The temperature to which a material must be heated to give off sufficient vapor to form a flammable mixture.

flaw. A general term often used to imply unmelts, cracks, discontinuities, or gross porosity in a coating.

flexibility. The quality or state of a material that allows it to be repeatedly flexed or bent without undergoing rupture. See also *flexure*.

flexure. A term used in the study of strength of materials to indicate the property of a body, usually a rod or beam, to bend without fracture. See also *flexibility*.

flocculate. A grouping of primary particles, aggregates, or agglomerates having weaker bonding than either the aggregate or agglomerate structures. Flocculates can generally be broken up by gentle shaking or stirring.

flocculation. Agglomeration of particles.

flow. The volume or weight of a gas or spray material in a given length of time. Generally expressed as cubic feet per hour (CFH), liters per minute (L/min), and pounds per hour (lb/h).

flow meter. A device for indicating the rate of gas flow in a system.

flow rate. The time required for a powdered material sample of standard weight to flow through an orifice in a standard instrument according to a specified procedure.

flow test. A standardized test to measure how readily a metal powder flows. See also *flow rate*.

fluidized bed. A contained mass of a finely divided solid that behaves like a fluid when brought into suspension in a moving gas or liquid.

fluidized-bed coating. A method of applying a thermoplastic resin coating onto a heated article. The article is immersed in a dense, fluidized bed of powdered resin, coated, and thereafter heated in an oven to provide a smooth, pinhole-free coating.

fluorescent penetrant inspection (FPI). Inspection using a fluorescent liquid that will penetrate any surface opening; after the surface has been wiped clean, the location of any surface flaws may be detected by the fluorescence, under ultraviolet light, of back-seepage of the fluid. This is not a suitable test for sprayed coatings.

fluorocarbons. The family of synthetic fluorine-containing resin plastics that are characterized by good thermal and chemical resistance, nonadhesiveness, low dissipation factor, and low dielectric constant.

fluoroscopy. An inspection procedure in which the radiographic image of an object is viewed on a fluorescent screen; limited to low-density materials or thin metallic sections because of the low light output of the fluorescent screen at safe levels of radiation.

flux. Certain elements and/or alloys that act as temperature suppressants and prevent the formation of, or act to dissolve and facilitate removal of, oxides and other undesirable substances.

fly ash. A finely divided siliceous material formed during the combustion of coal, coke, or other solid fuels.

foil. Metal in sheet form less than 0.15 mm (0.006 in.) thick.

forging. The process of working metal to a desired shape by impact or pressure. While some metals can be forged at room temperature, most are made more plastic by heating.

formability. The ease with which a metal can be shaped through plastic deformation.

forming. Making a change in the shape or contour of a part or object without intentionally altering its thickness.

fouling. An accumulation of deposits, including the growth of marine organisms on a submerged metal surface and/or heat-exchanger tubing.

foundry. A commercial establishment or building where metal castings are produced.

fraction. In powder metallurgy, the portion of a powder sample that lies between two stated particle sizes.

fractional factorial design. An experiment in which only an adequately chosen fraction of the treatments required for the complete factorial experiment are run.

fracture. A crack or separation in a material. The material may be in one piece and contain a fracture in two or more pieces.

fracture stress. The true, normal stress on the minimum cross-sectional area at the beginning of *fracture*. The term usually applies to tension tests of unnotched specimens.

fragmentation. The subdivision of a powder particle into smaller discrete particles.

fragmented powder. A powder obtained by fragmentation and mechanical comminution into fine particles.

free carbon. The part of the total carbon in metals or carbides that is present in elemental form as graphite or temper carbon.

free machining. Pertains to the machining characteristics of an alloy to which one or more ingredients have been introduced to

produce small broken chips, lower power consumption, better surface finish, and lengthen tool life. Among such additions are sulfur or lead to steel, lead to brass, lead and bismuth to aluminum, and sulfur or selenium to stainless steel.

free standing. A self-supporting coating with a definite shape and without attachment to a substrate.

fretting. A type of wear that occurs between tight-fitting surfaces subjected to cyclic, relatively slight motions of extremely small amplitude.

fretting wear. Wear arising as a result of fretting.

friction. The resisting force tangential to the common boundary between two bodies when, under the action of an external force, one body moves or tends to move relative to the surface of the other.

friction material. Any material exhibiting a high coefficient of friction designed for use where rubbing or frictional wear is encountered.

frit. A glass produced by fritting. It contains fluxing materials and is employed as a constituent in a glaze, body, or other ceramic composition.

fritting. The rapid chilling of the molten glassy material to produce frit.

fuel gases. Gases usually used with oxygen to produce a flame. Included are acetylene, natural gas, hydrogen, propane, methylacetylene propadiene stabilized, and other synthetic fuels and hydrocarbons.

full hard. A temper of nonferrous alloys and some ferrous alloys corresponding approximately to a cold-worked state beyond which the material can no longer be formed by bending.

furnace fusing. The melting together, in a furnace, of the spray deposit and the substrate, which results in coalescence. Furnacing offers the advantages of controlled heating, cooling, and often a protective atmosphere.

fused coating. A metallic coating (usually nickel or cobalt base) that has been melted and solidified, forming a metallurgical bond to the substrate.

fused/crushed powder. A powder resulting from the commutation of a fused solid. Generally a ceramic.

fused spray deposit. A self-fluxing spray deposit that is deposited by conventional thermal spraying and is subsequently fused using either a heating torch or a furnace. The coatings are usually nickel and cobalt alloys to which hard particles, such as tungsten carbide, may be added for increased wear resistance.

fusible alloys. A group of alloys containing temperature-suppressing elements such as bismuth, tin, indium, and cadmium. The term "fusible alloy" refers to any alloy that melts at relatively low temperatures, that is, below 185 °C (360 °F).

fusing. The melting of a metallic coating by means of a heat-transfer medium, followed by solidification.

fusion. The melting together of sprayed metal and base metal (substrate), or of sprayed metal only.

fusion temperature. During the fusing of self-fluxing coatings, the narrow temperature range within which the coating surface exhibits a glassy or highly reflective appearance.

fusion zone. The area of base metal melted as determined on the cross section of the coating.

G

gage. (1) The thickness of sheet or the diameter of wire. The various standards are arbitrary and differ with regard to ferrous and nonferrous products as well as sheet and wire. (2) An instrument used to measure thickness, length, or strain.

gall. To damage the surface by friction (adhesive wear).

galling. (1) A condition whereby excessive friction between surface high points results in localized welding, with subsequent spalling and a further roughening of the rubbing surfaces of one or both of two mating parts. (2) A severe form of scuffing associated with gross damage to the surfaces or failure. See also *scoring*.

galvanic cell. (1) A cell in which chemical change is the source of electrical energy. (2) A cell or system in which a spontaneous oxidation-reduction reaction occurs, the resulting flow of electrons being conducted in an external part of the circuit.

galvanic corrosion. Corrosion associated with the current of a *galvanic cell* consisting of two dissimilar conductors in an electrolyte or two similar conductors in dissimilar electrolytes.

galvanic series. A list of metals and alloys arranged according to their relative corrosion potentials in a given environment.

galvanize. To coat a metal surface with zinc using any of various processes (for example, hot dipping, electrogalvanizing, etc.)

gas atomization. An atomization process whereby molten metal is broken up into particles by a rapidly moving gas stream. The resulting particles are nearly spherical, some with attached satellites.

gas classification. Separation of powder into its particle size fractions by means of a controlled velocity gas stream flowing counterstream to the gravity-induced fall of the particles. The method is used to classify submesh-sized particles.

gas classifier. A device for gas classification; size will vary with end applications.

gas flow rate. The quantity of gas delivered or used during a thermal spray or grit-blasting operation.

gas holes. Voids in castings and fused coatings formed by outgassing of the molten metal as it solidifies. They may occur individually, in clusters, or throughout the solidified metal.

gasket. A sealing device, usually made of a deformable material, used between matched parts to prevent leakage of either a gas or fluid.

gas mass spectrometry. An analytical technique that provides quantitative analysis of gases through the complete range of elemental and molecular gases.

gas metal arc welding (GMAW). An arc welding process that produces coalescence of metals by heating them with an arc between a continuous filler metal (consumable) electrode and the work. Formerly referred to as metal inert gas (MIG) welding.

gas path seal. A clearance control coating system consisting of a sacrificial abradable component and a hard, abrasive component. The former is generally applied to a static member, while the latter is found on rotating parts.

gas pocket. A cavity caused by entrapped gas.

gas porosity. Voids or pores within a casting or fused coating caused by entrapped gas or by the evolution of gas during solidification.

gas tungsten arc welding (GTAW). An arc welding process that produces coalescence of metals by heating them with an arc between a tungsten (nonconsumable) electrode and the work. Formerly referred to as tungsten inert gas (TIG) welding.

general corrosion. Corrosion identified by uniform deterioration, proceeding uniformly and without appreciable localized attack. See also *uniform corrosion*.

glass. An amorphous solid made by fusing silica (SiO_2) with a basic oxide. Characteristic properties are transparency, hardness, and rigidity at ordinary temperatures.

glass transition temperature (T_g). The temperature at which an amorphous polymer changes from a hard and relatively brittle condition to a viscous or rubbery condition. In this temperature region, physical properties, such as hardness, brittleness, thermal expansion, and specific heat, may undergo significant, rapid changes.

glue. A hard gelatin obtained from various animal parts; may also be an adhesive prepared by combining it with heated water. Now, however, the word is synonymous with bond and adhesive.

graded coating. See also preferred term *graded coating*.

grade. In powder metallurgy, a specific, nominal chemical analysis powder identified by a code number. Example: cemented carbide manufacturers grade 74M 60 FWC (74 is usage; M is equipment manufacturer; 60 is nominal HRC; FWC is fine cut tungsten carbide).

graded coating. A thermal spray coating consisting of several successive layers of different materials; for example, starting with 100% metal, followed by one or more layers of metal-ceramic mixtures, and finishing with 100% ceramic. Also known as gradient coating.

gradient coating. See *graded coating*.

grain. An individual crystal in a polycrystalline material.

grain boundary. A narrow zone in a metal or ceramic corresponding to the transition from one crystallographic orientation to another, thus separating one *grain* from another.

grain growth. In polycrystalline materials, a phenomenon occurring slightly below the material melting point in which the large grains grow at the expense of the smaller ones. See also *recrystallization*.

granular powder. A powder having equidimensional but non-spherical, irregular-shaped particles.

graphite. (1) A crystalline allotropic form of carbon. (2) Uncombined carbon in cast irons.

gravity feed. A process by which powder is fed into a thermal spray gun by gravity.

grease. A lubricant composed of an oil thickened with a soap or other substance to a semisolid or solid consistency. Greases may contain various additives.

green. A coating in the as-sprayed condition.

grindability. Relative ease of grinding, analogous to *machinability*.

grinding. Removing material from a workpiece with a grinding wheel or abrasive belt.

grinding cracks. Shallow cracks formed on the surfaces of hard materials because of either overheating during grinding or the sensitivity of the material.

grinding fluid. An oil- or water-based fluid introduced into grinding operations to (1) reduce and transfer grinding heat, (2) lubricate during chip formation, (3) wash loose chips away, and (4) chemically aid the grinding action.

grinding oil. An oil-type grinding fluid that may contain additives but not water.

grinding wheel. A cutting tool of circular shape made of abrasive grains bonded together.

grit. Crushed ferrous or nonmetallic abrasive material in various mesh sizes used in abrasive blasting to clean and/or roughen surfaces.

grit blasting. Abrasive blasting with small, irregular pieces of steel, malleable cast iron, or hard, nonmetallic materials.

grit size. Nominal size of abrasive particles in blasting media and/or a grinding wheel, corresponding to the number of openings per linear inch in a screen through which the particles can pass. Usually expressed by Society of Automotive Engineers numbers, such as SAE G25.

gross porosity. In a sprayed coating, pores, gas holes, or voids that are larger and in greater numbers than those obtained in good practice.

guard. A device, often made of sheet metal or wire screening, that prevents accidental contact with moving parts of machinery.

gun. See preferred term *thermal spray gun*.

gun extension. Tubes of various lengths containing the nozzle and attached to the front of a thermal spraying device to permit spraying within confined areas or deep recesses.

H

half hard. A temper of nonferrous alloys and some ferrous alloys characterized by tensile strength approximately midway between those of dead soft and full hard tempers.

Hall flow. A standardized test method (ASTM B 13) used to determine the time required for 50 g (1.8 oz) of powder to flow through a fixed orifice.

halogen. Any of the elements of the halogen family, consisting of fluorine, chlorine, bromine, iodine, and astatine.

hard chromium. Chromium electrodeposited for engineering purposes rather than as a decorative coating. It is customarily thicker ($>1.2\ \mu\text{m}$, or 0.05 mils) than a decorative deposit but not necessarily harder.

hard face. (1) A coating of high hardness that is applied to a softer material. (2) To overlay with a coating for the purpose of promoting surface hardness.

hardfacing. The application of a hard, wear-resistant material to the surface of a component by thermal spraying or welding.

hardfacing alloys. A wear-resistant material. Available types include many metallic alloys, ceramics, and combinations thereof that may be supplied in powder or wire form.

hardness. A measure of the resistance of a material to surface indentation or abrasion. There is no absolute scale for hardness; therefore, to express hardness quantitatively, each type of test has its own scale of arbitrarily defined hardness. Indentation hardness can be measured by *Brinell*, *Rockwell*, *Vickers*, *Knoop*, and *Scleroscope hardness tests*.

hard surfacing. See preferred terms *hardfacing* or *surfacing*.

hard temper. Same as *full hard temper*.

heat-affected zone (HAZ). That portion of the base metal that was not melted during the fusing of a coating but whose microstructure and mechanical properties were altered by the heat.

heat buildup. The rise in temperature in a part during spray coating application.

heat check. A surface crack pattern formed by alternate rapid heating and cooling of the uppermost surface metal. May also result by overheating during grinding.

heat checking. A process in which fine cracks are formed on the surface of a metal or a coating due to the buildup of excessive frictional heat. Often the result of poor grinding practices.

heat distortion. The change in geometric size or shape or the flow of a material or configuration due to the application of heat.

heat-resistant alloy. An alloy developed for very-high-temperature service where relatively high stresses are encountered and where oxidation resistance is frequently required. Examples include refractory metals, chromium-molybdenum steels, stainless steels, and superalloys.

heat treatable alloy. An alloy that can be hardened by heat treatment.

heat treatment. Heating and cooling a solid metal or alloy in such a way as to obtain desired conditions or properties.

heavy metal. Tungsten carbide with nickel, cobalt, copper, and/or iron, the tungsten content being at least 90 wt% and the density being at least $16.8\ \text{g/cm}^3$.

helium (He). A monatomic inert gas having the atomic number 2. Sometimes used as a plasma gas.

hertz. A designation of electrical frequency that denotes cycles per second. Abbreviated Hz.

heterogeneity. The degree of nonuniformity of composition or properties. Contrast with *homogeneity*.

heterogeneous. A material or matter comprised of more than one phase separated by boundaries. In a sprayed coating, this is usually metals, oxides, and voids.

high-velocity noncombustion spray (HVNC). See preferred term *cold spray*.

high-velocity oxyfuel (HVOF) process. A flame spray process that takes place at very high pressures and combustion jet temperatures.

histogram. A plot of frequency of occurrence versus the measured parameter.

holidays. Discontinuities in a coating (such as porosity, cracks, gaps, etc.).

homogeneity. The degree of uniformity of composition or properties. Contrast with *heterogeneity*.

homogeneous. A body of material or matter, alike throughout; hence, comprised of only one chemical composition and phase.

homogenizing. A heat treating practice whereby a metal object is held at high temperature to eliminate or decrease chemical segregation by diffusion.

honeycomb. Manufactured product of resin sheet or metal foil, formed into hexagonal-shaped cells. Used as a core material in composite sandwich constructions. See also *sandwich construction*.

honoring. A finishing process used to produce uniform, dimensionally accurate, ultrahigh fine finishes.

hoop stress. The stress in a material of cylindrical form subjected to internal or external pressure.

hot corrosion. Accelerated metal surface corrosion resulting from the combined effects of oxidation and reactions with contaminants to form a molten salt on the metal that fluxes, destroys, or disrupts the normal protective oxide.

hot dip. Covering a surface by dipping the surface to be coated into a molten bath of the coating material. See also *hot dip coating*.

hot dip coating. A metallic coating obtained by dipping the basis metal into a molten metal such as zinc or aluminum.

hot isostatic pressing (HIPing). High-temperature/high-pressure compaction of a coating or powder metal component to consolidate its structure and improve density.

hot working. (1) The plastic deformation of metal at such a temperature and strain rate that recrystallization takes place simultaneously with the deformation and avoids *strain hardening*. Also referred to as hot forging and hot forming. (2) Controlled mechanical operations for shaping a product at temperatures above the recrystallization temperature. Contrast with *cold working*.

hydride powder. A powder produced by removal of the hydrogen from a metal hydride.

hydrogen (H₂). A diatomic gas having the atomic number 1. Lightest element known; very reactive. Often used as a secondary gas in plasma spraying and/or as a fuel in either flame or high-velocity oxyfuel spraying.

hydrogen embrittlement. A decrease of the *toughness* or *ductility* of a metal due to the absorption of atomic hydrogen.

hydrogen-reduced powder. Metal powder produced by hydrogen reduction of a compound.

hydrophilic. Having an affinity for water; easily wetted by water. Contrast with *hydrophobic*.

hydrophobic. Lacking an affinity for, repelling, or failing to absorb water; poorly wetted by water. Contrast with *hydrophilic*.

hydroxyapatite (HA). A biomaterial often used as a bone fixate. Recognized by the chemical formula Ca₅(PO₄)₃(OH). Also referred to as hydroxylapatite.

hygroscopic. Capable of attracting, absorbing, and retaining atmospheric moisture.

hypereutectic alloy. Any alloy whose composition has an excess of alloying element, compared with the eutectic composition, and whose equilibrium microstructure contains some eutectic structure.

hypereutectoid alloy. Any alloy whose composition has an excess of base metal, compared with the eutectoid composition, and whose equilibrium microstructure contains some eutectoid structure.

I

icosahedral. Pertaining to a twenty-sided crystal. The psi (ψ) phase in a quasi-crystal alloy.

image analysis. Tool used to determine the size, shape, and distribution of microstructural features by electronic scanning methods. Data can include individual measurements on separate features (feature specific) or field totals (field specific).

immersion lens. See *immersion objective*.

immersion objective. In optical microscopy, an objective in which a high-refractive-index medium is used between the specimen and objective to increase the resolving power of the lens.

immiscible. Characterized by the inability of two or more liquids or substances to dissolve in one another to form a single solution; mutually insoluble; incapable of attaining homogeneity.

impact wear. Wear of a solid surface resulting from repeated collisions between that surface and another solid body.

impingement. A succession of impacts between liquid or solid particles and a solid surface.

impingement angle. See preferred term *spray angle*.

impregnate. To infiltrate or saturate a coating with a sealer such as a resin, wax, or oil.

impurities. Minor constituent(s) not intentionally included that are, at some level, undesirable.

inclusion. Foreign material or a physical/mechanical discontinuity occurring within a coating or material.

incomplete fusion. In a fusing operation, fusion that is less than complete.

indentation hardness. Type of hardness test in which a pointed or rounded indenter is pressed into a surface under a substantially static load. Numerical hardness values are not absolute physical quantities but depend on the hardness scale used to express hardness. See also *Brinell*, *Knoop*, *Rockwell*, and *Vickers hardness tests*.

indenter. In hardness testing, a solid body of prescribed geometry used to determine the resistance of a solid surface to penetration.

indicator. A substance or device that, through some visible or audible occurrence, indicates the status of a solution, material, or process.

individuals. Constituent parts of a *population*.

inert. Unreactive with other materials.

inert gas. A gas that is stable, does not support combustion, and does not form reaction products with other materials. Included are helium, argon, and sometimes nitrogen.

infiltrant. Material used to infiltrate a porous coating.

ingot. A casting of simple shape.

inhibitor. A process or substance that retards a specific reaction.

injection angle. Angle at which powder is injected into the thermal spray stream. Powder injected at 0° is injected perpendicular to the flame/plasma. Positive angles indicate injection in the direction of spraying; negative angles are toward the nozzle or in reverse of the direction of spray.

inorganic. Matter being composed of substances other than hydrocarbons and their derivatives, or not of plant or animal origin. Contrast with *organic*.

insert. A part fitted into a second part.

insulator. (1) A material of low electrical or thermal conductivity, such as that used to insulate structures. (2) A component of a plasma gun.

integrated circuit (IC). An interconnected array of active and passive elements capable of performing at least one complete electronic circuit function.

intensity. Energy per unit area per unit time incident on a surface.

interconnected porosity. A network of connecting pores in a sprayed coating that permits a fluid or gas to pass through. Also referred to as intercommunicating, interlocking, or open porosity.

intercrystalline. Between the crystals, or grains, of a polycrystalline material. See *intergranular*.

intercrystalline corrosion. See *intergranular corrosion*.

intercrystalline cracking. See *intergranular cracking*.

interface. The contact surface between the spray deposit and the substrate.

interference fit. A fit between mating parts where there is nominally a negative or zero allowance between the parts, and where there is either part interference or no gap when the mating parts are made to the respective extremes of individual tolerances that ensure the tightest fit between the parts. Contrast with *clearance fit*.

intergranular. Between crystals or grains; in the grain boundary. Also called intercrystalline. Contrast with *transgranular*.

intergranular corrosion. Corrosion occurring preferentially at grain boundaries. Also called intercrystalline corrosion.

intergranular cracking. Cracking or fracturing that occurs in grain boundaries in a polycrystalline aggregate. Also called intercrystalline cracking. Contrast with *transgranular cracking*.

interlaminar. Between two adjacent laminae or sprayed particles.

intermediate phase. A distinguishable homogeneous phase in an alloy or coating whose composition range does not extend to any of the pure components of the system.

intermetallic compound. An intermediate phase in an alloy system having a narrow range of homogeneity and relatively simple stoichiometric proportions.

intermetallic phases. Compounds, or solid solutions, containing two or more metals having compositions, properties, and crystal structures different from those of the pure components of the system.

internal grinding. Grinding an internal surface, such as inside a cylinder or hole.

internal shrinkage. A void or network of voids within a coating caused by rapid solidification.

internal shrinkage cracks. Cracks in and/or between individual splats. Cracking is caused by shrinkage strain induced during cooling from the molten state. When confined to an individual lamina, they are usually transverse rather than longitudinal.

internal stress. See preferred term *residual stress*.

interpass temperature. In a multipass coating, the temperature of the deposited material before the next pass is started.

intersplat oxidation. Oxides surrounding a sprayed particle and preventing metal-to-metal contact between splats.

intracrystalline. Within or across the crystals or grains of a metal; same as transcrystalline and transgranular.

intracrystalline cracking. See *transgranular cracking*.

intralaminar. Within a single lamina or lamella, for example, an object (a void) or event (a fracture).

inverted microscope. A microscope arranged with the line of sight directed upward through the objective to the object.

ion. Atom or groups of atoms carrying a positive or negative charge as a result of having lost or gained electrons.

ionization. (1) The process wherein neutral atoms become charged by gaining or losing an electron. (2) Ionization of a gas forms a plasma.

irregular powder. A powder in which the particles lack symmetry.

isometric. A crystal in which the unit dimension on all three axes is the same.

isomorphous. Having the same crystal structure. Generally refers to intermediate phases that form a continuous series of solid solutions.

isotope. One of two or more nuclidic species of an element having an identical number of protons (Z) in the nucleus but a different number of neutrons (N). Isotopes of a given element differ in mass but are the same chemically.

isotropic. Having uniform properties in all directions.

isotropy. The condition of having the same values of properties in all directions.

item. An object or quantity of material.

Izod test. An impact test in which a V-notched specimen, mounted vertically, is subjected to a sudden blow delivered by a weighted pendulum arm. The energy required to break the

specimen is a measure of the impact strength or toughness of the material. Contrast with *Charpy test*.

J

jet pulverizer. A machine that comminutes metal pieces, ores, or agglomerates.

jig. Mechanism or fixture for holding a part and/or guiding the tool during machining, spraying, or assembly operation.

joint. The location where two or more members meet, are to be met, or have been fastened together.

joint clearance. The distance between the faying surfaces of a joint.

joint design. Joint geometry, including the required dimensions of the welded/brazed/bonded joint.

joint geometry. The shape and dimensions of a joint in cross section.

journal. The part of a shaft or axle that rotates or oscillates relative to a radial bearing. A journal is part of a larger unit, such as a crankshaft. It is preferred that the term “shaft” be kept for the whole unit.

K

kelvin. A scale of absolute temperatures in which zero is approximately -273.16°C (-459.69°F).

kerosene. A liquid fuel used in certain high-velocity oxyfuel processes.

kiln. A large furnace used for sintering, baking, drying, or burning firebrick or refractories, or for calcining.

kinetic energy. The energy that a body possesses because of its motion.

kinetic spray. See preferred term *cold spray*.

Kirkendall voids. The formation of voids by diffusion across the interface between two different materials, with the material having the greater diffusion rate diffusing into the other.

Knoop hardness number (HK). A number related to the applied load and the area of the impression made by a rhomboidal-shaped indenter. Knoop hardnesses are reported with the test load indicated.

Knoop hardness test. An indentation hardness test using calibrated machines to force a rhombic-based diamond indenter into the surface of a test material and to measure the long diagonal after removal of the load.

knurling. Impressing a design into a metallic surface by small, hard rollers that carry the corresponding design on their surfaces.

L

laboratory sample. A small sample, intended for analysis, withdrawn from a larger sample; the laboratory sample must retain the composition of the larger sample.

lacquer. (1) A coating formulation based on thermoplastic film-forming material dissolved in an organic solvent. (2) In lubrication, a deposit resulting from the breakdown of fuels and lubricants when exposed to high temperatures. A softer deposit is called a *varnish*.

lamella. A thin layer, as in the overlaid particles in a thermal spray deposit.

lamina. A single flattened, sprayed particle. See also *splat*.

lamination. The buildup of sprayed particles (splats) to form a coating.

lap. A surface imperfection, or seam, caused by hot metal being folded over and then being drawn, rolled, or forged into the surface but without being welded.

lapping. A finishing operation using a fine abrasive loaded onto a lapping material. Lapping offers improvements to the workpiece, including better dimensional accuracy, correction of minor imperfections, better surface finishes, and a closer fit between mating surfaces.

laser. A device that emits a concentrated, coherent beam of light. Lasers are used in metalworking to melt, cut, engrave, or weld and sometimes to inspect parts.

laser cladding. Coating buildup using powder and directing a laser beam onto it, causing it to melt and fuse to the substrate.

laser glazing. Using a laser beam to melt a coating surface to form a fine-grained structure or glaze.

latent heat. Thermal energy absorbed or released by the coated part during/after spraying.

lateral crack. A crack produced beneath and generally paralleling the splats within a coating.

latex. A stable dispersion of a polymeric substance; included are natural or synthetic rubber or plastic.

Laves phase. A type of hard intermetallic compound found in cobalt-base hardfacing alloys containing molybdenum and silicon. Laves phase has the formula $\text{Co}_3\text{Mo}_2\text{Si}$ or CoMoSi .

layer. A stratum of surfacing material.

lead. A soft, malleable, ductile metallic element having atomic number 82; it is bluish-white in color. Major ore is galena (PbS). Primary uses include storage battery grids, radiation shielding, paint, type metal, and some solders.

leak testing. A nondestructive test for determining the escape or entry of liquids or gases from pressurized or into evacuated components or systems designed to hold these fluids.

lens. A transparent optical element, so constructed that it serves to change the degree of convergence or divergence of the transmitted rays.

lenticular. Plateletlike; to be flat. Used to describe a sprayed particle or splat.

levigation. (1) Separation of fine powder from coarser material by forming a suspension of the fine material in a liquid. (2) A means of classifying a material as to particle size by the rate of settling from a suspension.

light metal. One of the low-density metals, such as aluminum ($\sim 2.7 \text{ g/cm}^3$), magnesium ($\sim 1.7 \text{ g/cm}^3$), titanium ($\sim 4.4 \text{ g/cm}^3$), beryllium ($\sim 1.8 \text{ g/cm}^3$), or their alloys.

linear expansion. The increase of a given dimension measured by the expansion of a specimen or component subject to a temperature gradient. See also *coefficient of thermal expansion*.

linear shrinkage. The shrinkage in one dimension of a coating during cooling.

liquation. (1) Separation of a low-melting constituent of an alloy from the remaining constituents; most apparent in alloys having a wide melting range. (2) Partial melting of an alloy, often as a result of *coring* or other compositional heterogeneities.

liquation temperature. The lowest temperature at which partial melting can occur.

liquid honing. Directing an air-ejected aqueous solution, containing fine abrasives, against a surface to produce a finely polished finish.

liquid penetrant inspection (LPI). A type of nondestructive inspection that locates open discontinuities on the surface of a metal. First, a penetrating dye or fluorescent liquid is used to infiltrate the discontinuity; then, excess surface penetrant is removed. Penetrant within the discontinuities seeps out and registers as an indication. The test is not recommended for sprayed coatings.

liquidus. The lowest temperature at which a material is completely liquid. See also *solidus*.

load. (1) With testing machines, a force applied to a testpiece, measured in units such as pounds, Newtons, or kilograms. (2) In tribology, the force applied normal to the surface of one body by another contacting body or bodies.

loading. Filling the pores of a grinding wheel with material from the work, resulting in lost productivity and finish quality.

localized corrosion. Corrosion at discrete sites, for example, crevice corrosion, pitting, and stress-corrosion cracking. Contrast with *general corrosion* and *uniform corrosion*.

loose metal. A debonded area of coating.

lot. A specific amount of material produced at one time using one process, with constant manufacturing conditions, and offered as a unit quantity. Compare with *batch*.

low-alloy steels. A category of ferrous metals containing additive alloying elements that exhibit mechanical properties superior to plain carbon steels. The alloying elements optimize mechanical properties and, in some cases, increase corrosion resistance.

low-cycle fatigue (LCF). Fatigue that occurs at a relatively small number of cycles ($<10^4$ cycles).

low-expansion alloys. Materials whose dimensions do not appreciably change with temperature. Generally, the low-expansion alloys are identified by trade names, such as Invar (64Fe-36Ni), Kovar (54Fe-29Ni-17Co), Elinvar (52Fe-36Ni-12Cr), Super Invar (63Fe-32Ni-5Co), and Stainless Invar (37Fe-53Co-10Cr).

low-pressure plasma spray (LPPS). See preferred term *vacuum plasma deposition*.

lubricant. Any substance interposed between two surfaces for the purpose of reducing the friction or wear between them.

lubricity. The ability of a substance to reduce wear and friction, other than by its purely viscous properties.

M

machinability. The ability of a material to be machined with relative ease.

machining. Removing material from the surface of a part, usually accomplished using a power-driven cutting tool.

machining allowance. See preferred term *finish allowance*.

machining damage. Damage to a solid surface resulting from the machining process. Also see *surface integrity*.

machining stress. *Residual stress* caused by machining.

macro. The gross properties of a coating without consideration of the properties of its individual components.

macrograph. A graphic representation of a prepared specimen surface at a magnification not exceeding 25 \times . When photographed, the reproduction is known as a photomacrograph.

macrohardness test. Term applied to hardness testing procedures, such as Rockwell or Brinell, to distinguish them from microindentation hardness tests, such as the Knoop or Vickers tests. See also *microindentation* and *microindentation hardness number*.

macropore. Large pores visible to the naked eye.

macroscopic. Visible at magnifications at or below 25 \times .

macroshrinkage and cracking. Isolated, clustered, or interconnected voids in a coating that are detectable macroscopically.

macrostress. Same as macroscopic stress.

macrostructure. Coating structure revealed by macroscopic examination of the surface of a metallographically prepared specimen.

magnesia. Magnesium oxide (MgO); used principally in basic refractories.

magnesium and magnesium alloys. A group of silvery-white metals valued for their light weight (the density of magnesium is approximately 1.7 g/cm³, versus 2.7 g/cm³ for aluminum and 7.8 g/cm³ for steel) and high strength-to-weight ratio. Magnesium is produced either by electrolysis of fused chloride (from brine wells or seawater) or extracted from ore (generally dolomite).

magnetic-particle inspection. A nondestructive inspection technique that identifies variations in magnetic flux in ferromagnetic materials such as would be caused by discontinuities and variations in hardness. Variations are indicated by a pattern change seen on an oscilloscope screen. Not a valid test for as-sprayed coatings; however, it may be successfully employed with fused deposits.

magnification. A ratio of the size of an image to its corresponding object. Usually determined by linear measurement.

main bearing. A bearing supporting the main power-transmitting shaft.

malleability. A characteristic of metals that permits plastic deformation in compression without fracture. See also *ductility*.

mandrel. (1) A blunt-ended tool or rod used to retain the cavity in a hollow metal product during working. (2) A metal bar around which other metal may be cast, bent, formed, or shaped. (3) A shaft or bar for holding work to be machined.

manipulator. A mechanical device for moving and orienting a spray gun or part during coating application.

manual spraying. Spraying wherein the thermal spray device is hand held by the operator, who also inputs its operating parameters.

martensite. A term used for microstructures formed by diffusionless phase transformation in which the parent and product phases have a specific crystallographic relationship. Martensite is characterized by an acicular pattern in the microstructure in both ferrous and nonferrous alloys.

martensitic stainless steels. Essentially ferrous alloys of chromium and carbon that possess a body-centered tetragonal crystal structure (*martensite*) in the hardened condition. See also *stainless steels*.

martensitic transformation. A reaction that takes place in some metals on cooling, with the formation of an acicular structure called *martensite*.

mask. A device for protecting a surface from the effects of blasting and/or coating. Masks are generally either reusable (permanent) or disposable.

masking tape. A tape used as a stopoff or deterrent to grit-blast roughening and/or the adherence of thermal spray particles. Tapes are generally of a disposable nature.

mass flow controller. A device for metering and controlling the amount/mass (not volume) of gas fed to the spray gun or powder feed system.

master alloy. An alloy, rich in one or more desired addition elements, added to a metal melt to raise the percentage of a desired constituent.

master alloy powder. A prealloyed metal powder of specific alloy content, designed to be diluted by mixing with a base powder to produce the desired composition.

materials characterization. Using analytical methods (spectroscopy, microscopy, chromatography, etc.) to describe various features of composition (both bulk and surface) and structure that contribute to the identification of the properties or use of a material.

matrix. The major continuous substance of a coating, as opposed to inclusions or particles of materials having dissimilar characteristics.

matrix metal. The metallic constituent in which separate particles of one or more other constituents are embedded.

matte finish. (1) A dull texture on surfaces that have been roughened by blasting. (2) A dull finish characteristic of most thermal spray coatings.

maximum strength. See preferred term *ultimate strength*.

MCrAlY. An acronym for a metallic alloy suited for high-temperature use and resistance to oxidation and hot corrosion. The "M" represents the major metallic constituent, usually iron (FeCrAlY), nickel (NiCrAlY), or cobalt (CoCrAlY). Other constituents include chromium (Cr), aluminum (Al), and yttrium (Y).

mechanical alloying (MA). An alternate cold welding and shearing of particles of two or more species. Generally accomplished by ball milling.

mechanical bond. The adhesion of a thermal sprayed deposit to a roughened surface by the mechanism of particle interlocking. See also *adherence*.

mechanical disintegration. See preferred terms *comminution* and *pulverization*.

mechanical metallurgy. The science and technology dealing with the behavior of metals when subjected to applied forces; generally restricted to plastic working or shaping of metals.

mechanical properties. Properties of a material that reveal its elastic and inelastic behavior when force is applied. Included are modulus of elasticity, tensile strength, elongation, hardness, and fatigue limit. Compare with *physical properties*.

mechanical stage. In microscopy, a device for adjusting the position of a specimen, usually by translation in two directions at right angles to each other.

mechanical testing. Methods by which the mechanical properties of a coating or material are determined.

mechanical wear. Removal of material due to mechanical processes under conditions of sliding, rolling, or repeated impact. The term includes *adhesive wear* and *abrasive wear*. Compare with *corrosive wear* and *thermal wear*.

mechanical working. Subjecting metals to pressure exerted by rolls, hammers, or presses in order to change the shape or physical properties of the metal.

melt. (1) To change a solid to a liquid by the application of heat. (2) A charge of molten metal.

melting point. Temperature at which a pure metal, compound, or eutectic changes from solid to liquid; the point that the liquid and the solid are at equilibrium.

melting range. The range of temperatures over which an alloy other than a compound or eutectic changes from solid to liquid; the range of temperatures from solidus to liquidus of any given composition.

melting rate. In wire spraying, the weight or length of wire melted in a unit of time. Sometimes called melting-off rate, burn-off rate, or spray rate.

melting temperature. See *melting point*.

melt-off rate. See *melting rate*.

membrane. Any thin sheet or layer.

mesh. A screen or sieve used to determine powder particle size and/or distribution.

mesh size. (1) The opening(s) or size of opening(s) in a designated sieve or screen. Mesh sizes are given as the number of wires per inch of standard screen construction, for example, Tyler or U.S. standard. (2) The width of the aperture in a cloth or wire screen. See also *sieve analysis* and *sieve classification*.

metal. An opaque, lustrous, elemental chemical substance that is a good conductor of heat and electricity and, when polished, reflects light. Most elemental metals are malleable and ductile and are, in general, denser than other elemental substances. Metals may be either elemental, compounds, or alloys.

metal arc spraying. See preferred term *electric arc spraying*.

metal cored wire. A composite filler metal wire consisting of a metal tube or other hollow configuration containing alloying

ingredients. Minor amounts of additive ingredients facilitate arc stabilization and oxide fluxing.

metallic bond. That which exists between a self-bonding sprayed metal and its metallic substrate. See also *metallurgical bond*.

metallic glass. A noncrystalline (amorphous) metal or alloy commonly produced by drastic rapid supercooling of a molten alloy.

metallic wear. Wear due to rubbing or sliding contact between metals; exhibiting the characteristics of severe wear as: plastic deformation, material transfer, and indications that cold welding of asperities has taken place. See also *adhesive wear*.

metallic whisker. A fiber composed of a single crystal of metal. See also *whisker*.

metallizing. Forming a thermal spray coating using wire. See preferred terms *wire flame spraying* or *electric arc spraying*.

metallograph. An optical instrument designed for visual observation and photomicrography of prepared surfaces of opaque materials at magnifications of 25× to approximately 2000×.

metallography. Using microscopy, the study of metallic structures by various optical and electron methods.

metallurgical bond. Adherence of a coating to the base material, characterized by diffusion, alloying, or intermolecular or intergranular attraction at the interface between the coating and the base material.

metallurgy. The science and technology of metals and their alloys.

metal-matrix composite. Material with a nonmetallic reinforcement incorporated into a metallic matrix. The reinforcement may constitute from 10 to 60% of the composite. Reinforcements include graphite, silicon carbide, boron, Al_2O_3 , and refractory metals.

metal powder. Material consisting of metallic particles, usually in the size range of 0.1 to 1000 μm .

metal spraying. Fabrication by depositing plasticized and/or molten metal onto a suitable substrate. See also *thermal spraying* and *flame spraying*.

micro. Metallurgical term referring to a microspecimen or photomicrograph.

microanalysis. The analysis of samples smaller than 1 mg.

microcrack. A crack of microscopic proportions. Also termed microfissure.

microcracking. Intersplat and/or transsplat cracking.

microetching. Development of microstructure features for microscopic examination. The usual magnification exceeds 25× (50× in Europe).

microfissure. A crack of microscopic proportions.

micrograph. A reproduction of a minute image at a magnification greater than 25×. If produced by photographic means, it is called a photomicrograph (not a microphotograph).

microhardness. The hardness of a material determined by forcing a Vickers or Knoop diamond indenter into its surface under light loads. Typically, the resulting indentations are very small, requiring measurement with the use of a microscope. The pro-

cedure is capable of determining the hardnesses of different microconstituents within a structure, or of measuring steep hardness gradients, such as those encountered in case hardening or a diffusion zone. See also *microhardness test*.

microhardness number. A commonly used term for the more technically correct term *microindentation hardness number*.

microhardness test. A microindentation hardness test using a calibrated machine to force an indenter, under a test load of 1 to 1000 gram-force, into a test material and to measure the diagonal or diagonals optically. See also *Knoop hardness number*, *microindentation hardness number*, and *Vickers hardness number*.

microinch ($\mu\text{in.}$). One millionth of an inch (0.000001 in.).

microindentation. In hardness testing, the small residual impression left in a solid surface when an indenter, typically a diamond stylus, is withdrawn after penetrating the surface.

microindentation hardness number. A numerical quantity, stated in units of pressure (kg/mm^2), that expresses the resistance to penetration of a solid surface by a hard indenter under a specified, slowly applied force. The prefix “micro” indicates that the indentations produced are typically between 10.0 and 200.0 μm across. See also *Knoop hardness number* and *Vickers hardness number*.

micromesh. A sieve with precisely square openings in the range of 10 to 120 μm produced by electroforming.

micromesh sizing. The process of sizing ultrafine particles (*micromesh*) using an air or a liquid suspension process.

micrometer. (1) A dimension of 0.001 mm, written with the abbreviation μm . Also referred to as micron (not recommended). (2) A measuring device.

micropore. The pores in a coating that can only be detected under a microscope.

microporosity. Extremely fine coating porosity.

micropulverizer. A machine that disintegrates powder by strong impacts from a small hammer fastened to a solid disk that rotates at very high velocity.

microscope. An instrument capable of producing a magnified image of an object.

microscopic. Only visible at magnifications above 25×.

microscopy. The science of the interpretive use and applications of microscopes.

microsegregation. Minute segregation within a coating, grain, crystal, or particle. See also *coring*.

microshrinkage. A coating imperfection, only detectable microscopically, consisting of cracks and intersplat voids. Microshrinkage results from particle contraction during solidification.

microstress. *Residual stress* in a material within a distance comparable to the splat or grain size.

microstructure. The structure of an object, organism, or material as revealed by a microscope at magnifications greater than 25×.

microtrac. A laser-light-scattering device used to measure powder particle size and specific surface area.

migration. Movement of entities (such as electrons, ions, atoms, molecules, vacancies, and grain boundaries) from one site to

another under the influence of a driving force (such as an electrical potential, heat, or a concentration gradient).

MIG welding. See preferred term *gas metal arc welding*.

mil. An English measure of thickness or diameter equal to 0.0254 mm (0.001 in.). A common designation of wire diameter, coating thickness, or corrosion loss.

mild steel. Carbon steel with a maximum of 0.25% C and 0.4 to 0.7% Mn, 0.1 to 0.5% Si, and some residuals of sulfur, phosphorus, and/or other elements.

mill finish. A nonstandard (and typically nonuniform) surface finish on mill products that have not undergone a special surface treatment after the final working or heat treating step.

milling. Removing material from a workpiece as it moves past a toothed rotating cutter.

milling cutter. A rotary cutting tool provided with cutting elements (teeth) that intermittently engage the workpiece and remove material by relative movement of the workpiece and cutter.

mill product. Any commercial product of a mill.

mill scale. The heavy oxide layer that forms during hot fabrication or heat treatment of metals.

minus sieve. The portion of a powder sample that passes through a standard sieve of a specified number. See also *sieve analysis*.

mist lubrication. Lubrication by an oil mist produced by injecting oil into a gas stream.

mixing. In powder metallurgy, the thorough intermingling of powders of two or more different materials (not to be confused with *blending*).

mixing chamber. That element of a plasma or fuel gas gun in which gases are mixed.

modeling. In statistics, a theoretical procedure used to construct a relationship of experimental outputs with a reasonable number of inputs.

modulus. A constant or coefficient that represents the degree to which a material possesses some attribute.

modulus of elasticity. The measure of rigidity or stiffness of a material; the ratio of stress, below the proportional limit, to the corresponding strain.

moisture content. Proportion of moisture in a material, expressed as a mass percent of the moist specimen, that is the mass of the dry substance plus the moisture.

mold. (1) The form that contains the cavity into which molten metal is poured to produce a casting. (2) A die.

molecule. A structure, of definite geometrical configuration, built of atoms bound together by chemical forces.

molten metal flame spraying. A thermal spraying process variation in which the metallic material to be sprayed is in the molten condition. See also *flame spraying*.

monoclinic. A crystal structure having three axes of any length, with two included angles equal to 90° and one included angle not equal to 90°.

morphology. The characteristic shape, form, or surface texture or contours of coatings, crystals, grains, or particles of (or in) a material, generally on a microscopic scale.

mounting. Embedding or clamping a metallographic specimen during surface preparation to facilitate grinding, polishing, and observation.

mounting artifact. A false structure introduced during the mounting stages of metallographic preparation.

mounting resin. Thermosetting (e.g., Bakelite or diallyl phthalate) or thermoplastic (e.g., methyl methacrylate or polyvinyl chloride) resins used to mount metallographic specimens.

mullite. A mineral of theoretical composition $3\text{Al}_2\text{O}_3 \cdot 2\text{SiO}_2$. Includes kyanite, sillimanite, andalusite, and various clays. Used as a refractory material for firebrick and furnace linings.

multiple-pass coating. A coating made by depositing material with two or more successive passes.

N

napped cloth. A woven metallurgical polishing cloth in which some fibers are aligned approximately normal to one of its surfaces.

nascent surface. A freshly developed and completely uncontaminated surface.

neat. Not diluted or mixed with another substance.

needles. Elongated or rodlike powder.

net shape. The shape of a powder metallurgy part, casting, or forging that conforms closely to specified dimensions.

neutral flame. (1) A flame in which there is neither an excess of fuel nor oxygen in the inner flame. (2) An oxyfuel gas flame in which the portion used is neither oxidizing nor reducing. See also *carburizing flame*, *oxidizing flame*, and *reducing flame*.

nickel plating. Electrodeposition of nickel.

nitriding. Case hardening by diffusing nitrogen into an alloy steel surface to form hard nitrides.

noble metal. A metal with good resistance to chemical reaction, oxidation, and to solution by inorganic acids. The term is often used synonymously with precious metals (e.g., gold, platinum, palladium, etc.).

nodular powder. Irregular particles having knotted, rounded, or similar shapes. Not sharply irregular.

nominal dimension. The size of the dimension to which the tolerance is applied. For example, if a dimension is 50 mm \pm 0.5 mm (2.00 in. \pm 0.02 in.), the 50 mm (2.00 in.) is the nominal dimension, and the \pm 0.5 mm (\pm 0.02 in.) is the tolerance.

nominal value. A value assigned for the purpose of a convenient designation. Often, it is an average number with a tolerance to enable the fitting together of parts.

nonconforming. A quality-control term describing an item that does not meet normal acceptance criteria for the specific product or service. A nonconforming unit is not necessarily defective.

nondestructive evaluation (NDE). Broadly considered synonymous with *nondestructive inspection (NDI)*. Specifically, the quantitative analysis of NDI findings to ascertain if determinable defects are in or out of acceptable limits.

nondestructive inspection (NDI). A diagnostic tool, such as ultrasonic or radiographic inspection, for determining the quality or characteristics of an item without permanently altering the subject or its properties. Used to find internal anomalies in a structure without degrading or impairing either its properties or serviceability.

nondestructive testing (NDT). Broadly considered synonymous with *nondestructive inspection (NDI)*.

nonmetallic inclusions. See *inclusion*.

nontransferred arc. In plasma spraying, the arc established between the electrode (cathode) and the constricting nozzle (nozzle). The workpiece is not in the electrical circuit. Compare with *transferred arc*.

nonwetting. A lack of metallurgical wetting between a molten, fusible, self-fluxing sprayed coating and a metallic surface due to surface tension effects or a physical barrier on the metallic surface.

normal. (1) An imaginary line forming right angles with a surface or other lines. Sometimes called the perpendicular. (2) Usual and ordinary.

normal section. A metallographic section taken through the face (downward) of a thermal sprayed coating. Sprayed particles are observed as broad, spreading droplets. The view is in the direction of spraying or impact.

normal wear. Loss of material within the design limits for the specific intended application.

nozzle. That portion of a thermal spray device through which the flame or plasma stream exits. In a plasma gun, it is the anode.

nozzle accumulation. Surfacing material deposited on the inner surface and on the exit end of the nozzle.

nucleus. The heavy central core of an atom consisting of protons and neutrons.

nylon. The generic name for all synthetic polyamides.

O

objective. The primary magnifying system of a microscope.

oblique illumination. Illumination from light inclined at an oblique angle to the optical axis.

observation. Obtaining information regarding the attitude of a test specimen, or reading a characteristic of the specimen. See also *observed value*.

observed value. The attitude or measurement information obtained from a single observation of a test specimen.

ocular. See preferred term *eyepiece*.

oil. A liquid of vegetable, animal, mineral, or synthetic origin that feels slippery to the touch.

one-component adhesive. An adhesive material incorporating a latent hardener or catalyst that is activated by heat.

open-circuit voltage. The voltage between the output terminals of a plasma system when no current is flowing.

open pore. A pore open to the surface of a thermal spray coating.

open porosity. See preferred term *interconnected porosity*.

optical microscope. An instrument used to obtain an enlarged image of a small object, using visible light. Magnification capability ranges from 1 to 2000 \times .

optical pyrometer. An instrument for measuring the temperature of heated material by comparing the intensity of light emitted with a known intensity of an incandescent lamp filament.

orange peel. A surface roughening resembling a pebble-grained pattern. Also called *alligator skin*.

organic. Being or composed of hydrocarbons or their derivatives, or matter of plant or animal origin. Contrast with *inorganic*.

organic acid. A chemical compound with one or more carboxyl radicals (COOH) in its structure; examples are butyric acid, $\text{CH}_3(\text{CH}_2)_2\text{COOH}$; maleic acid, $\text{HOOCCH}:\text{CHCOOH}$; and benzoic acid, $\text{C}_6\text{H}_5\text{COOH}$.

outgassing. The release of adsorbed or occluded gases or water vapor, usually by heating.

overcoat. To apply subsequently more material over previously applied layers.

overheating. Heating a metal to such an excessively high temperature that its properties are impaired.

overlap. In continuous spraying, applying subsequent passes partly over those previously applied.

overlaying. See preferred term *surfacing*.

overmix. Mixing of a powder longer than necessary to produce adequate distribution of powder particles. Overmixing may cause particle size segregation.

oversize powder. Powder particles larger than the maximum permitted by a particle size specification.

overspray. The spray material that is not deposited on the part during coating but rather is captured by the dust collection system, drops to the floor, or is deposited in nonintended areas.

oxidation. A corrosion reaction in which the corroded metal forms an oxide; usually applied to reaction with a gas containing elemental oxygen, such as air. When occurring at high temperatures, it is referred to as *hot corrosion*.

oxidative wear. A *corrosive wear* process in which chemical reaction with oxygen results in a loss of material, or where an oxidizing environment predominates.

oxide. A chemical compound, the combination of oxygen with a metal, forming a ceramic; examples are aluminum oxide, zirconium oxide, and chromium oxide.

oxidized steel surface. Surface having a thin, tightly adhering oxidized skin (from straw to blue in color).

oxidizing flame. A gas flame produced with excess oxygen in the inner flame that has an oxidizing effect.

oxyfuel gas spraying. See preferred term *flame spraying*.

oxygen (O_2). Combustion-supporting gas used in both flame and high-velocity oxyfuel spraying. Very reactive.

P

packing density. See preferred term *apparent density*.

paddle mixer. A mixer that uses paddles mounted on a rotating shaft or disk to move and mix thermal spray powder.

parameter. Any one of a group of thermal spray process variables or operating conditions. Usually refers to spray gun settings and target manipulation.

parent metal. See preferred term *base metal*.

partially stabilized zirconia. Zirconia (ZrO_2) that contains a mixture of cubic and tetragonal and/or monoclinic phases produced by the addition of small amounts of stabilizing agents, such as magnesium oxide (MgO), calcium oxide (CaO), or yttrium oxide (Y_2O_3). See also *zirconia*.

particle. (1) A small subdivision of matter, such as a metal or ceramic powder particle or sprayed splat. Typically measured in micrometers. (2) Any small part of matter, such as an electron, atom, or molecule.

particle chemistry. Elements contained within individual particles.

particle hardness. The hardness of an individual splat as measured by a Knoop- or Vickers-type microhardness indentation test.

particle morphology. The form and structure of an individual metal or ceramic powder particle.

particle shape. The appearance of any thermal spray particle, such as spherical, rounded, angular, acicular, dendritic, irregular, porous, fragmented, blocky, rod, flake, nodular, or plate.

particle size. The lineal dimension of an individual particle as determined by analysis with screens or other suitable instruments. See also *sieve analysis* and *sieve classification*.

particle size analysis. See preferred term *sieve analysis*.

particle size classification. See preferred term *sieve classification*.

particle size distribution. Classification of powdered materials as determined by various testing methods defining the particle sizes and quantities in a given size range.

particle size range. The limits between which a variation in powder particle size is allowed; for example $-200 + 300$ mesh, the largest particles of which will pass through a 200-mesh sieve and the smallest of which will not pass through a 325-mesh sieve. See also *sieve analysis* and *sieve classification*.

particle sizing. Segregation or distribution of powder into specified particle size ranges.

parts per million (ppm). A measure of proportion by weight, equivalent to one unit weight of a material per million (10^6) unit weights of compound.

pass. A single progression of the thermal spray device across the surface of the substrate being coated.

passivation. The process in metal corrosion in which corrosion is precluded by the formation of a barrier layer.

passive. The state of a metallic surface characterized by low corrosion rates in a potential region that is strongly oxidizing for the metal.

patina. The coating, usually green, that forms on the surface of metals such as copper and its alloys when exposed to the atmosphere. Also used to describe the appearance of a weathered surface of any metal.

pearlite. A metastable lamellar aggregate of ferrite and cementite resulting from the transformation of austenite at temperatures above the bainite range.

pebbles. See *orange peel*.

peeling. The detaching of one layer of a coating from another, or from the base metal, because of poor adherence.

peening. Blasting with spherical beads or shot for surface cleaning or modification.

penetrant. A liquid with low surface tension used in liquid penetrant inspection to flow into surface openings of parts being inspected.

penetrant inspection. See preferred term *liquid penetrant inspection*.

penetration hardness. Same as *indentation hardness*.

penetration hardness number. Any number value expressing the resistance of a body to the penetration of a second, usually harder, body.

percent error. The ratio, expressed as a percentage, of the error to the correct value of the applied load.

periodic. Occurring at regular intervals, in a prescribed or predetermined, repeatable fashion. Used to describe the atomic or crystal array of most metals.

perpendicular section. A section cut perpendicular to a surface of interest in a metallographic specimen.

pewter. A tin-base white metal containing antimony and copper.

pH. The negative logarithm of the hydrogen-ion activity; it denotes the degree of acidity or basicity of a solution. At 25 °C (77 °F), 7.0 is the neutral value. Values below 7.0 indicate increasing acidity; those above 7.0, increasing basicity (pH values range from 0 to 14).

phase. A physically homogeneous and distinct portion of a material system; chemically identical.

phase change. The transition from one physical or chemical state to another, such as gas to liquid, liquid to solid, gas to solid, or vice versa, that is, WC to W_2C .

phase diagram. A graphical representation of the temperature and composition limits of phase fields in an alloy system as they actually exist under the specific conditions of heating or cooling. Synonymous with constitution diagram. Compare with *equilibrium diagram*.

phase separation. The formation of a second portion from a previously homogeneous substance. It is time-dependent.

phosphating. Forming an adherent phosphate coating on a metal by immersing it in an aqueous phosphate solution. Also called phosphatizing. See also *conversion coating*.

phosphor bronzes. See *bearing bronzes*.

photomacrograph. A photograph depicting an object or feature at a maximum magnification less than 25 \times .

photomicrograph. A photograph depicting an object or feature at magnifications greater than 25 \times .

physical bond. See preferred term *metallic bond*.

physical metallurgy. The science and technology dealing with the properties of metals and alloys, and of the effects of composition, processing, and environment on those properties.

physical properties. Properties of a material that are relatively insensitive to structure and can be measured without the application of force; for example, density, electrical conductivity, coefficient of thermal expansion, magnetic permeability, and lattice parameter. Does not include chemical reactivity. Compare with *mechanical properties*.

physical testing. Methods used to determine the entire range of the physical properties of a material.

physical vapor deposition (PVD). A coating process whereby the deposition species are transferred and deposited in the form of individual atoms or molecules. Sputtering and evaporation are typical PVD methods.

pickle. Chemical removal of surface oxides (scale) and other contaminants from iron and steel by immersion in an aqueous acid solution such as sulfuric and hydrochloric acids.

pickling. Removing surface oxides from metals by chemical or electrochemical reaction.

pimple. An imperfection, such as a small protuberance on the surface of a thermal spray coating.

pine-tree crystal. A type of *dendrite*.

pinhole porosity. Porosity consisting of numerous small holes distributed throughout a thermal spray coating.

pinholes. Very small holes that are sometimes found as a type of porosity in a coating because of the microshrinkage or gas evolution during solidification.

pin-on-disk machine. A mechanical test machine used to determine wear resistance in which a stylus or "pin" is loaded against the surface of a flat disk. Pin loading is parallel to the axis of rotation of either it or the disk. A circular wear path occurs due to the rotational motion of the pin.

pistol. See preferred term *thermal spray gun*.

pit. A small crater in the surface of a coating created by environmental exposure, for example, corrosion, wear, or thermal cycling. See also *pitting*.

pitting. Small, localized cavities caused by corrosion, wear, or other mechanically assisted degradation of a metallic surface.

planar. Lying essentially in a single plane.

plasma. A gas of sufficient energy so that a large fraction of the species present is ionized and will conduct electricity.

plasma arc. The electrically charged, ionized gas contained within the arc chamber.

plasma densification. To densify a powder by spraying it through a plasma generator/gun.

plasma-forming gas. The gas, in the plasma generator, that is heated to the high-temperature plasma state by the electric arc. See preferred term *arc gas*.

plasma metallizing. See preferred term *plasma spraying*.

plasma spraying. A thermal spraying process in which a non-transferred arc is a source of heat that ionizes a gas that melts the coating material and propels it to the workpiece.

plasma stream. The zone of intense heat and light issuing from the plasma nozzle bore. It results from energy liberated as the charged gas particles (ions) recombine with electrons.

plasma transfer arc (PTA). A welding process using a plasma. Instead of using a neutral plasma, the arc is transferred to the substrate (the anode). Powder is fed into the plasma, heated, and fused to the substrate. Coatings are similar to weldments.

plastic. A material that contains, as an essential ingredient, an organic polymer of large molecular weight, is solid in its finished state, and can be shaped by flow.

plastic deformation. The permanent (inelastic) distortion of materials under applied stresses that strain the material beyond its elastic limit.

plastic flow. Phenomenon that takes place when metals are stretched or compressed permanently without rupture.

plasticity. The property of a material that allows it to be repeatedly deformed without rupture and allows it to retain its shape after the applied force has been removed.

platelets. Flat powder or sprayed particles having considerable thickness. The thickness, however, is smaller when compared with the length and widths of the particles. See also *particle shape*.

plating. Forming an adherent layer of metal on an object; often used as a shop term for electroplating.

polarizer. In an optical microscope, a device into which normal light passes and from which polarized light emerges.

polished surface. A metallographically prepared surface that reflects a large proportion of the incident light.

polishing. A surface-finishing process for ceramics and metals using successive grades of abrasive papers and cloths.

polishing artifact. A false structure introduced during a polishing stage of metallographic surface preparation.

polyamide-imide (PAI) resins. A family of polymers based on the combination of trimellitic anhydride with aromatic diamines.

polyamide (PA) resins. The thermoplastic polymers that are most commonly regarded as being synonymous with nylons.

polycrystalline. A solid comprised of many crystals or crystallites, intimately bonded together.

polyesters. Plastics based on resins composed principally of polymeric esters.

polyimide (PI). A polymer produced by reacting an aromatic dianhydride with an aromatic diamine.

polymer. A high-molecular-weight organic compound, natural or synthetic. Examples include polyethylene, rubber, and cellulose.

polymorphism. The ability of a solid to exist in more than one form. In metals, alloys, and similar substances, this usually means the ability to exist in two or more crystal structures, or in an amorphous state and at least one crystal structure.

polyurethanes (PUR). A large family of polymers with widely varying properties and uses. They may be thermosetting or thermoplastic, rigid or soft and flexible, cellular or solid. Properties may be tailored to suit the desired application.

polyvinyl chlorides (PVC). The second most widely used plastic after polyethylene. It is manufactured from sodium chloride and natural gas, with the chloride from the salt comprising 57 wt%.

population. The total number of items or units of material under consideration.

pore. A small opening, void, or channel within a sprayed coating.

pore size. Width of a pore in a particle or coating.

pore size distribution. Indicates the volume fractions of different pore size categories, which are determined metallographically.

porosity. Fine holes or pores within a coating resulting from trapped air or shrinkage.

port. See preferred term *powder port*.

postheating. Heating a coating immediately after spraying or fusing for tempering, diffusion, stress relieving, or for providing a controlled rate of cooling to prevent formation of a hard or brittle structure.

posttreatment. Any processing performed on the coating after its application. This could include any of the many joining, cleaning, thermal, finishing, and other operations employed in industry.

potentiometer. An instrument that measures electromotive force by balancing against it an equal and opposite electromotive force across a calibrated resistance.

powder. Material manufactured into finely divided particles. When explicitly blended for thermal spraying, powder falls within a specific mesh range, usually finer than 120 mesh (125 μm).

powder alloy. Powder prepared from a homogeneous molten mixture of elements and sometimes entrapped carbides or metal oxides. All particles have approximately the same composition.

powder blend. A mixture of two or more alloy powders.

powder clad (wire clad). Powder or wire wherein one alloy is encapsulated in another; a composite.

powder composite. Two or more individual materials combined to form a single integrated unit. May be either chemically clad, mechanically agglomerated, or spray dried.

powder feeder. A mechanical device designed to introduce a controlled flow of powder into the spray device.

powder feed gas. see preferred term *carrier gas*.

powder feed rate. The quantity of powder introduced into the hot, gaseous stream per unit of time. Expressed in pounds/hour (lb/h) or grams/minute (g/min).

powder flame spraying. A thermal spraying process in which the material to be sprayed is in powder form. See also *flame spraying*.

powder injection. Feeding of a powder through a powder port into a thermal spray flame or plasma stream.

powder injection angle. Angle at which powder is introduced to the flame or plasma.

powder injector. See preferred term *powder port*.

powder metallizing. See preferred term *powder flame spraying*.

powder metallurgy (P/M). The use of metal powders for the production of shaped objects.

powder port. Internal or external device through which powder is injected into the flame or plasma stream.

powder production. The process by which a powder is produced, such as machining, milling, atomization, condensation, reduction, oxide decomposition, carbonyl decomposition, electrolytic deposition, or precipitation from a solution.

prealloyed powder. A metallic powder composed of two or more elements that are alloyed during the manufacturing process and in which the particles are of the same nominal composition throughout.

precoat. (1) A bond coat. (2) A thin coating. (3) An underlayment or undercoat.

precious metals. The eight precious metals, listed in order of their atomic number as found in periods 5 and 6 (groups VIII and Ib) of the periodic table, are ruthenium, rhodium, palladium, silver, osmium, iridium, platinum, and gold.

precipitation. Causing a solid substance to be separated from a liquid solution.

precision. The closeness of agreement between test results measured under a fixed set of conditions.

preheat. See *preheating* and *preheat temperature*.

preheating. Heating to an intermediate temperature for a short time immediately before thermal spraying.

preheat temperature. A specified temperature that the base metal must attain immediately before the coating operation is performed.

premix. A uniform mixture of components prepared by a powder producer. See also *mixing*.

preproduction test. Testing conducted to determine conformity to established production standards, or to determine product quality before parts are produced, or to determine conformance to specification requirements requiring qualification testing.

primary gas. The major constituent of the arc gas fed to the gun to produce the plasma.

process metallurgy. The science and technology of winning metals from their ores and purifying metals; its two major branches are *extractive metallurgy* and refining.

propane (C_3H_8). A colorless fuel gas used in flame spraying; it is found in natural gas.

propylene (C_3H_6). A fuel gas used in flame spraying, particularly high-velocity oxyfuel. It is derived from petroleum hydrocarbon cracking.

protective barriers. Curtains or portable fireproof canvas shields sometimes required to enclose work areas when there is a possibility of the spray stream being misdirected or where the glare of the arc or flame could injure unprotected eyes.

protective clothing. Leather or metal-coated articles designed to prevent burns from ultraviolet radiation or misdirected particles.

prototype. A model suitable for use in the complete evaluation of form, design, performance, and materials processing.

pull-out. Condition where particles are pulled from a coating surface during machining, grinding, or metallographic preparation.

pulverization. Process of reducing powder particle sizes by mechanical means; also called *comminution* or mechanical disintegration.

purge. The removal of air from gas lines or the spray gun and replacing it with a gas prior to spray initiation.

pyrometer. A device for measuring temperatures above the range of liquid thermometers.

pyrophoric powder. A powder whose particles self-ignite and burn on exposure to oxygen or air. Example: fine zirconium powder.

Q

qualification test. Testing to determine material or system conformance to specification requirements. Generally, specification qualification requires conformance to all tests in the specification, or it may be limited to conformance to a specific type and/or class under the specification. Compare with *acceptance test* and *preproduction test*.

qualitative analysis. An analysis in which some or all of the components of a sample are identified. Compare with *quantitative analysis*.

quality. The totality of features and characteristics of a product or service that relate to its ability to satisfy a given need (fitness-for-use concept of quality).

quality characteristics. Any dimension, mechanical property, physical property, functional characteristic, or appearance characteristic that can be used as a basis for measuring the quality of a unit, a product, or a service.

quality control. All aspects of the spraying process and the final product.

quantitative analysis. A measurement in which the amount of one or more components of a sample is determined. Contrast with *qualitative analysis*.

quartz. One of several crystalline forms of silica (SiO_2); others include cristobalite and tridymite.

quartz glass. See *quartz* and *glass*.

quasi-crystal. Alloys or intermetallic compounds whose atomic lattices exhibit a complicated, predictable pattern described as quasi-periodic. This attribute yields a low coefficient of friction, excellent lubricity, and a nonstick as-sprayed surface.

quench rate. The speed at which a sprayed particle cools on striking the surface of the substrate.

R

race (or raceway). The groove or path in which the rolling elements in a rolling-contact bearing operate.

radio frequency (RF) plasma. A system in which the torch is a water-cooled, high-frequency induction coil surrounding a gas stream. On ignition, a conductive load is produced within the induction coil, which couples to the gas, ionizing it to produce a plasma.

radiograph. An x-ray photograph. See also *radiography*.

radiography. Nondestructive inspection method in which an object is exposed to an x-ray beam and the resulting shadow

image is recorded on photographic film placed behind the object, or viewed on a screen (real-time radiography).

radiology. Material inspection methods based on the differential absorption of penetrating radiation.

radius. (1) The distance from the center of a circle to a point on the circumference or surface of the circle. (2) Removing sharp edges or corners.

radius of bend. The radius of the inside surface of the bend during bending.

randomize. Procedure used to allot treatments at random to experimental units to provide a high degree of independence in the contributions of experimental error to estimates of treatment effects.

range. A measurement equal to the difference or interval between the highest and lowest values of a set of quantities.

rapid solidification. The cooling or quenching of liquid (molten) metals at rates that range from 10^4 to 10^8 °C/s.

rare earth metals. Includes the 16 in group IIIA of the periodic table: scandium, yttrium, lanthanum, cerium, praseodymium, neodymium, promethium, samarium, europium, gadolinium, terbium, dysprosium, holmium, erbium, thulium, ytterbium, and lutetium. "Rare" implies that these elements are scarce; in fact, they are quite abundant. Rare earths, as a group, lie in the 50th percentile of elemental abundances.

rate of flame propagation. The speed at which a flame travels through a mixture of gases.

reactive metal. A metal that readily combines with oxygen to form stable oxides, for example, titanium, zirconium, and beryllium. They may also become embrittled by the absorption of some gases.

reagent. A substance, chemical, or solution used to detect, measure, or react with other substances, chemicals, or solutions. See also *reagent chemicals*.

reagent chemicals. High-purity chemicals used for analytical reactions where impurities must either be absent or at a known concentration.

recess. A groove or depression in a surface.

recrystallization. (1) The formation of a new, strain-free grain structure from that existing in cold-worked metal. (2) The change from one crystal structure to another, as occurs on heating or cooling through a critical temperature. (3) A process by which crystals are grown at the expense of others. See also *crystallization*.

rectifier. An electrical device for converting alternating current to direct current.

reducing flame. A combustion gas flame mixture containing excessive fuel or too little oxygen.

reel. A spool or hub for coiling or feeding wire.

refractory. The ability to resist the effects of high heat. The noun form usually refers to an inorganic, nonmetallic, or ceramic material of very high melting point with properties that make it useful in kiln construction and furnace hardware.

refractory alloy. A heat-resistant alloy, usually one with a very high melting range.

refractory materials. Traditionally, nonmetallics (ceramics) that resist degradation by corrosive gases, liquids, or solids at elevated temperatures. They must withstand thermal shock caused by rapid temperature changes, failure attributable to thermal stresses, and mechanical fatigue attributed to the high-temperature environment.

refractory metals. High-melting-point metals, including niobium, tantalum, molybdenum, tungsten, and rhenium. They have the lowest vapor pressures of all metals. However, they are readily degraded by oxidizing environments at relatively low temperatures, which restricts their use at elevated temperatures.

regulator. A device for controlling the delivery of gas at some substantially constant pressure.

relative density. See preferred term *apparent density*.

relay. An electrically controlled device that opens and closes electrical contacts to effect the operation of other devices in an electric circuit.

reliability. A quantitative measure of the ability of a product or service to fulfill its intended function for a specified period of time.

repeatability. A term used to refer to result variability associated with a limited set of specifically defined sources of variability at a single spray source.

reproducibility. Used to describe result variability associated with specifically defined components of variance obtained at two or more spray facilities.

residual elements. Small quantities of elements unintentionally present in an alloy.

residual strain. The strain associated with residual stress.

residual stress. The stress existing in a resting body, in equilibrium, and not subjected to external forces. Stress arises in sprayed coatings primarily because the sprayed particles contract on cooling from the liquid to solid state.

resin. A solid, semisolid, or pseudosolid organic material that has a high molecular weight, exhibits flow when subjected to stress, has a softening or melting range, and usually fractures conchoidally in a shelllike pattern.

resistance alloys. Electrical application alloys used to control equipment and to generate heat.

resonance time. See preferred term *dwell time*.

rheology. The science of deformation and the flow of matter.

riddle. A sieve used to separate granular materials into various particle-sized grades or to remove undesirable foreign matter from a powdered material.

robotic spraying. The use of a computerized, articulated, or multi-axis manipulation mechanism to traverse the spray gun and/or target during coating application.

robust. Insensitivity of a process to variances in working conditions and/or parameters.

Rockwell hardness number (HR). A number derived from the depth of impression of an indenter as the weight is increased from a fixed minor load to a major load and then returned to the minor load. Various Rockwell scales have been developed and are designated by alphabetic suffixes to the hardness designa-

tion (for example, Rockwell C-scale, HRC). See also *Rockwell superficial hardness number*.

Rockwell hardness test. An indentation hardness test using a calibrated machine that uses the depth of indentation, under constant load, as a measure of hardness. Either a 120° diamond cone or various-diameter steel balls may be used as the indenter. See also *brale indenter*.

Rockwell superficial hardness number. Values obtained using lighter loads to determine the hardness of thin samples. The superficial hardness is expressed by the symbol HR followed by a scale designation and quantity of weight used, for example, HR15N and HR30T.

Rockwell superficial hardness test. The same test used to determine the Rockwell hardness number except that smaller minor and major loads are used.

room temperature. A temperature in the range of ~20 to 30 °C (~68 to 85 °F).

root mean square (rms). (1) The square root of the arithmetic mean of the squares of the numbers in a given set of numbers. (2) A term describing the surface roughness of a machined surface. See also *surface roughness*.

rotating electrode process. A method for producing metal powders wherein a rapidly rotating, consumable metal electrode is arc melted. Centrifugal force causes the molten material to be slung off as droplets that become solidified in flight.

rough grinding. Grinding without regard to finish, usually to be followed by a subsequent operation.

rough machining. Machining without regard to finish, usually to be followed by a subsequent operation.

roughness. (1) Relatively finely spaced surface irregularities, the heights, widths, and directions of which establish the predominant surface pattern. (2) The microscopic peak-to-valley distances of surface protuberances and depressions. See also *surface roughness*.

rough threading. A surface-roughening technique consisting of cutting threads with their sides and tops jagged and torn.

routing. Cutting out and contouring various shapes using a small-diameter, high-speed rotating cutter.

run. A continuing or uninterrupted sequence of occurrences in the same parametric series.

rust. A visible corrosion product consisting of hydrated oxides of iron. Applied only to ferrous alloys.

rutile. A particular crystalline form of titanium dioxide (TiO₂) used as a pigment and as an additive or component in some types of ceramic, glass, and glaze manufacture.

S

sacrificial protection. Reducing the degree of corrosion to a metal by galvanically coupling it to a more anodic metal; a form of cathodic protection.

sagging. A condition observed during the fusing of self-fluxing alloys where the coating is overheated (melted) and begins to run or sag.

salt fog test. An accelerated corrosion test in which specimens are exposed to a fine mist of a corrosion-inducing solution (typically, 5% NaCl in water). Also known as salt spray test.

salt spray test. See *salt fog test*.

sample. (1) Units of a product withdrawn for testing purposes in order to provide information about the properties or characteristics of the product. (2) A portion of a material intended to be representative of the whole.

sample average. The sum of all the observed values in a sample divided by the sample size. It is a point estimate of the population mean. Also known as arithmetic mean.

sample size. The number of parts, units, or observations in a sample.

sample splitter. A device to divide a powder lot for sampling.

sample thief. A pointed, hollow, tubular device to withdraw a representative powder sample from a packaging unit.

sampling. Obtaining a representative portion of a material that is of adequate size for conducting any required tests or analyses.

sand. A granular material resulting from the disintegration or crushing of rocks or mineral deposits.

sandblasting. Abrasive blasting with sand. See also *blasting or blast cleaning*.

sandwich construction. A panel composed of a lightweight core material, such as honeycomb, cellular or foamed plastic, and so forth, to which two relatively thin, dense, high-strength/high-stiffness faces/skins are adhered.

satin finish. A diffusely reflecting surface finish on metals, lustrous but not mirrorlike.

sawing. Using a toothed blade or disc to sever parts or cut contours.

scale. Surface oxidation, consisting of loosely adherent layers of corrosion products, left on metals by heating or casting in an oxidizing atmosphere.

scaling. Forming a thick layer of oxidation products on metals at high temperature. Scaling should be distinguished from rusting, which involves the formation of hydrated iron oxides. See also *rust*.

scalping. In powder processing, removing either oversized or undersized particles.

scanning electron microscope (SEM). A high-power magnifying and imaging instrument that uses an accelerated electron beam as an optical device. It may use reflected (SEM) or transmitted (scanning transmission electron microscopy) electron optics. SEM offers capabilities unobtainable with the light microscope, including magnifications up to 60,000 \times and improved depth of field by as much as a factor of 300.

scanning electron microscopy (SEM). An analytical technique for the study of surfaces and objects at very high resolution and magnifications.

Scleroscope hardness number (HSc or HSd). A number related to the height of rebound of a hammer dropped on the material being tested. It is measured on a scale divided into 100 units; the higher the number, the higher the hardness.

Scleroscope hardness test. A dynamic indentation hardness test that drops a diamond-tipped hammer onto the surface being tested; the height of rebound is the hardness of the material being tested.

scoring. (1) Surface damage evidenced by severe scratches in the direction of sliding. *Scuffing* is sometimes used as a synonym for scoring. (2) In tribology, a severe form of wear characterized by the formation of extensive grooves and scratches in the direction of sliding.

scrap. Items discarded because they are defective or otherwise unsuitable for use.

scratch. A groove produced in a solid surface by the cutting and/or plowing action of a sharp particle or protuberance moving across it.

scratch hardness test. A test in which a stylus is traversed along a surface so as to determine the resistance of that surface to cutting or abrasion. Scratch hardness tests involve using specialized tips to produce a scratch or groove whose width and/or depth are measurable. However, these tests have not been standardized to the extent that indentation-type tests have been.

screen. The woven wire or fabric cloth, having square openings, used in a sieve for retaining particles greater than the particular mesh size. U.S. standard, ISO, or Tyler screen sizes are commonly used.

screen analysis. See *sieve analysis*.

screen classification. See *sieve classification*.

screening. Separation of a powder according to particle size by passing it through a screen having the desired mesh size.

scuffing. Localized damage caused by the occurrence of solid-phase welding between sliding surfaces, without local surface melting.

seal. A device designed to prevent the movement of gas or fluid from one chamber to another, or to exclude contaminants.

sealant. A material applied to a coating that hardens or cures in place, forming a seal against a gas or liquid entry. Principal function is as a barrier to water, air, atmospheric pollution, vibration, insects, dirt, and noise.

seal coat. Material applied to infiltrate the pores of a thermal spray deposit.

sealing. Closing pores in coatings to render them less absorbent.

secondary gas. The gas constituting the minor constituent of the arc gas fed to the gun to produce the plasma. Typical secondary gases include nitrogen, helium, and/or hydrogen; their introduction increases the temperature of the plasma.

secondary operation. Any operation performed on a coating, such as sizing, coining, repressing, impregnation, infiltration, heat or steam treatment, machining, joining, plating, or other surface treatment.

sectioning. The removal of a conveniently sized, representative specimen from a larger sample for metallographic inspection. Sectioning methods include shearing, sawing, and abrasive cutting.

seed. Small particles that serve as nuclei or centers for further attachment of material on their surfaces.

segment. A portion of the whole.

segregation. (1) Nonuniform distribution of alloying elements, impurities, or phases. (2) In powders, the separation either by size or constituents.

seize. To bind or prevent motion.

seizing. The stopping of a moving part by a mating surface as a result of excessive friction.

seizure. The stopping of relative motion as the result of interfacial friction.

selectivity. The ability to respond to a prescribed circumstance, substance, or constituent and not to others.

self-bonding materials. Those materials that form a metallurgical bond with the substrate in the as-sprayed condition.

self-fluxing alloys. Certain materials that “wet” the substrate and coalesce when heated to their melting point, without the addition of a fluxing agent. The alloys usually contain temperature suppressants such as boron and/or silicon.

semiautomatic spraying. Spray processing wherein the gun parameters are fixed and gun manipulation is programmed.

sensitivity. (1) The capability of a method or instrument to discriminate between samples having different concentrations or amounts of the analyte. (2) The smallest difference in values that can reliably be detected with a given measuring instrument.

setup. The positioning of the spray gun and/or component to be coated to a prescribed schedule.

shadow mask. A thermal spraying technique in which an area is partially shielded (by raising the masking slightly above the surface) during spraying, thus permitting overspray to produce a feathering at the coating edge.

shear. The type of force that causes or tends to cause two adjacent parts to slide relative to each other in a direction parallel to their plane of contact.

shear fracture. A fracture mode in crystalline materials resulting from translation along slip planes that are preferentially oriented in the direction of the shearing stress.

shear strength. The maximum shear stress that a material is capable of sustaining. It is calculated from the maximum load during shear testing using the original cross-sectional area.

sheath. The outermost covering of a cored wire.

sheet. A flat-rolled metal product of some maximum thickness and minimum width.

shelf life. Duration a substance or product can be stored under specified conditions and continue to meet all applicable requirements and/or remain suitable for its intended function.

shielding gas. During spray application, a stream of inert gas directed at the substrate and spray stream to provide an atmospheric barrier and minimize oxidation.

shim. A thin piece of material used between two surfaces to obtain a proper fit, adjustment, or alignment.

Shore hardness. A measure of the resistance of a material to indentation during Scleroscope hardness testing. The higher the number, the greater the resistance. Normally used for rubber materials. See also *Scleroscope hardness test*.

shot. Small, spherical particles of metal or glass.

shotblasting. (1) Blasting with round metal shot to remove mill scale more rapidly or more effectively than done by sandblasting. See also *shot peening*.

shot peening. Cold working of metals by the impingement of a stream of shot, directed at the metal surface at high velocity under controlled conditions. This produces compressive stresses in the metal surface, resulting in an increase in fatigue strength.

shrinkage. (1) The dimensional or volume reduction of a material when dried, calcined, or fired (sintered). (2) The contraction of metal during cooling.

shrinkage cracks. Cracks that form in a coating as a result of particle contraction prior to complete solidification.

shrinkage stress. Residual coating stresses caused by the shrinkage of individual particles and their interaction with adjacent particles.

shrink fit. An interference fit produced by heating the outer member of mating parts to a practical temperature for easy assembly. The inner member is usually maintained at or near room temperature. The inner component may be cooled to increase ease of assembly.

shroud. A device fitted to the front of a spray gun. It surrounds the exiting spray stream, inhibiting air from entering the stream, thereby reducing oxidation of the coating.

shunt. A device used to divert part of an electric current.

sieve. A standard wire mesh or screen used in graded sets to determine the mesh size or particle size distribution of granular solids. See also *sieve analysis*.

sieve analysis. A method of determining particle size distribution wherein sieves are stacked in order, with the largest mesh size at the top and a pan at the bottom. A weighed powder sample is spread on the top sieve, and the stack is agitated in a prescribed manner for a specified period. Powder fractions, remaining on each sieve and in the pan, are weighed separately and reported as percentages retained or passed by each sieve.

sieve classification. The separation of powder into particle size ranges by the use of a series of graded sieves. Also called screen analysis.

sieve fraction. That portion of a powder sample that passes through a sieve of specified number and is retained by some finer mesh sieve of specified number. See also *sieve analysis*.

sieve shaker. A device for shaking, knocking, or vibrating a single sieve or a stack of sieves. It consists of a frame; a motorized knocker, shaker, or vibrator; and fasteners for the sieve(s).

silica (SiO₂). The oxide of silicon, found naturally as quartz or in complex combination with other elements such as silicates.

silica gel. A precipitated colloidal mass or gel of indefinitely hydrated silica. Useful as a desiccant, scavenger, and catalyst substrate.

silicon carbide (SiC). A hard ceramic material. Often used as a blasting or grinding abrasive and as a refractory brick for furnace linings.

silicon nitride (Si_3N_4). A hard, wear-resistant ceramic compound. Major types are reaction-bonded Si_3N_4 (RBSN), hot-pressed Si_3N_4 (HPSN), sintered (pressureless) Si_3N_4 (SSN), sintered reaction-bonded Si_3N_4 (SRBSN), and hot isostatically pressed Si_3N_4 (HIPSIN).

single crystal. A material that is completely composed of a regular array of atoms; one grain.

single-crystal superalloys. Nickel-base alloys that contain a single crystal, or more accurately, a single grain or primary dendrite.

sinter. To densify, crystallize, bond together, and/or stabilize a particulate material, agglomerate, or product by heating close to but below the melting point of the material. May involve the melting of minor components or constituents, and/or a chemical reaction.

sintering. The bonding of adjacent surfaces of particles in a mass of powder or a coating by heating. Sintering strengthens and normally produces densification and recrystallization.

sintering atmospheres. The atmosphere in a sintering furnace designed to protect the parts or powder from contamination from the environment.

sintering cycle. A predetermined and closely controlled time-temperature regime, including heating and cooling cycles.

sintering temperature. The maximum temperature at which sintering is performed.

sintering time. The time period during which a product is at the sintering temperature.

size analysis. The determination of particle sizes within a given distribution. See also *sieve analysis*.

size fraction. A separated fraction of a powder whose particles lie between specified upper and lower size limits. See also *sieve analysis*.

slag. A nonmetallic product resulting from the mutual dissolution of flux and nonmetallic impurities in smelting and refining.

slick up. Point at which a self-fluxing alloy begins to shine during the fusing operation.

sliding wear. The motion of two moving bodies in which their surface velocities, at the point of contact, are different with regard to magnitude and/or direction.

slip fit. A loosely defined *clearance fit* between parts assembled by hand without force, but implying slipping contact.

slitting. Cutting or shearing along single lines to cut strips from a sheet or to cut along lines of a given length or contour.

slurry. A thick mixture of liquid and solids, the solids being in suspension in the liquid.

slurry erosion. Erosion produced by the movement of a slurry past a solid surface.

slurry wear test. Erosion testing wherein material is worn (eroded) away by the action of an erosive medium suspended in a liquid carrier.

smearing. A mechanical phenomenon involving plastic shear deformation, where material is flowed or carried over itself.

solidification. The change in state from liquid to solid on cooling.

solidification range. The temperature between the liquidus and the solidus.

solidification shrinkage. The reduction in volume of a material from beginning to end of solidification.

solid lubricant. Any solid used as a powder or thin film on a surface to provide protection from damage during relative movement and to reduce friction and wear. Examples include molybdenum disulfide, graphite, synthetic fluorine-containing resin, and mica.

solid solution. A single, solid, homogeneous crystalline phase containing two or more chemical species.

solidus. The highest temperature at which a metal or alloy is completely solid. See also *liquidus*.

solute. The component of either a liquid or solid solution that is dissolved in the solvent.

solution. In chemistry, a homogeneous dispersion of two or more types of molecular or ionic species. Solutions may be composed of any combination of liquids, solids, or gases, but they always consist of a single phase.

solvent. The component of either a liquid or solid solution that dissolves the solute.

solvent cleaning/degreasing. The removal of soluble contaminants from the workpiece surface by immersion in suitable cleaners.

spalling. The spontaneous chipping, fragmentation, or separation of a surface or coating.

specimen. A test object, often of standard dimensions and/or configuration, that is used for destructive or nondestructive testing.

specimen stage. The part of the microscope that supports the specimen holder and the specimen. It is movable in a plane perpendicular to the optic axis.

spectrograph. An optical instrument that uses photography to record a spectral range. Radiant power passing through the system is integrated over time, and the quantity recorded is a function of radiant energy.

spherical powder. A powder consisting of rounded or ball-shaped particles.

spinel. A group of compounds of the same crystal type and general formula as magnesium aluminate, MgAl_2O_4 or $\text{MgO} \cdot \text{Al}_2\text{O}_3$, that are refractory and chemically near neutral.

splashing. The impacting, sliding, and wetting of a sprayed particle on a substrate.

splat. A single, thin, flattened, sprayed particle.

splat cooling. A rapid, high rate of cooling in sprayed deposits, leading to the formation of metastable phases and sometimes an amorphous microstructure.

splat powder. A rapidly cooled or quenched powder whose particles are flattened.

splat quenching. The process of producing splats.

sponge. A form of metal characterized by a porous condition that is the result of decomposition or reduction of a compound with-

- out fusion. Often applied to forms of iron, titanium, zirconium, uranium, plutonium, and the platinum-group metals.
- spongy.** A porous condition in some types of powder particles, usually reduced oxides.
- spool.** Spray wire package consisting of a continuous length of metallic wire wound on a cylinder.
- spot weld.** A resistance weld made between overlapping members. Its cross section is approximately circular.
- spray.** A moving mass of dispersed liquid droplets or heat-softened particles.
- sprayability.** Material attribute enabling it to be readily passed through a spray device without degradation and be deposited onto a prepared substrate.
- spray angle.** The angle of particle impingement, measured from the surface of the substrate to the axis of the spraying nozzle.
- spray booth.** See preferred term *exhaust booth*.
- spray chamber.** An enclosure in which spraying is confined. It generally contains a protective atmosphere.
- spray deposit.** A coating applied by any of the thermal spray methods. See also *thermal spraying*.
- spray distance.** During spraying, the distance maintained between the spray gun nozzle tip and the workpiece surface.
- spray drier.** A vessel into which an aqueous powder slurry is sprayed through injection ports, atomized, and hurled into a heated gas stream in which it is dried. The dried droplets are typically tiny, spherical, free-flowing agglomerates.
- spray drying.** A powder-producing process in which a slurry of liquids and solids is atomized into a chamber through which heated gases are passed. The liquids evaporate, and the solids are collected. The finished product consists of free-flowing, spherical agglomerates.
- spray forming.** The fabrication of *free-standing* shapes by depositing a material onto a mandrel, or other host surfaces, and removing the spray deposit after achieving the desired thickness.
- spray fuse.** Processing in which a coating is deposited and subsequently fused by heating.
- spraying sequence.** The order in which different layers of material are applied in a planned relationship, such as overlapped, superimposed, or at certain angles.
- spray metallizing.** See preferred term *metallizing*.
- spray nozzle.** (1) In atomizing metal powders, an orifice through which molten metal passes, forming a stream that is disintegrated by a gas, a liquid, or by mechanical means. (2) In spray drying, the head in which the injector ports are located.
- sputtering.** A glow discharge coating process wherein bombardment of a cathode causes atoms to be stripped from its surface for deposition onto a nearby positioned target.
- stabilizing gas.** The ionized arc gas forming the plasma; it is usually introduced into the arc chamber tangentially. The cold gas chills the outer surface of the arc stream, tending to constrict the plasma, raise its temperature, and force it out of the anode (nozzle) in a steady, relatively unfluctuating stream.
- stage.** A device for holding a specimen in the desired position in the optical path of a microscope.
- stainless steels.** Iron-base alloys containing at least 10.5% Cr that achieve their stainless or corrosion-resistance characteristics through the formation of an adherent chromium-rich oxide surface film. The oxide is self-generating and self-renewing.
- standardization.** (1) Establishing, by common agreement, criteria, terms, principles, practices, materials, items, processes, and equipment parts and components. (2) The adoption of generally accepted uniform procedures that directly affect the design of a product or a facility. (3) In analytical chemistry, the assignment of a compositional value to one standard on the basis of another standard.
- standoff distance.** See preferred term *spray distance*.
- static.** Stationary or very slow. Contrast with dynamic.
- statistic.** A summary value calculated from the observed values in a sample.
- statistical process control (SPC).** To enhance, optimize, and control coating properties influenced by parameter interaction.
- statistical quality control.** The application of statistical techniques for measuring and improving the quality of processes and products (includes statistical process control, diagnostic tools, sampling plans, and other statistical techniques).
- steels.** Iron-base alloys that contain manganese, carbon, and, depending on the type of steel, other alloying elements.
- step mounting.** The intentional overlapping of workpieces in order that one protects or masks its neighbor during the blasting or spraying process.
- stoichiometric.** Having the precise weight relation of all the elements in a chemical compound.
- stopoff.** An antiadhesion substance applied to surfaces adjacent to areas to be coated to restrict the adhesion of sprayed particles.
- strain.** A unit of change in the size or shape, resulting from a deformation or flow process, of a body due to an applied force.
- strain gage.** Device for measuring small amounts of strain produced during tensile and similar tests on metal.
- strain rate.** The time rate of straining. Strain, as measured on the tensile specimen gage length, is used for determining strain rate. Because strain is dimensionless, the units of strain rate are the reciprocal of time.
- strength.** The maximum nominal stress a material can sustain. Always qualified by the type of stress (tensile, compressive, or shear).
- stress.** The intensity of the internal forces that resist a change in the shape of a material that is or has been subjected to external forces. It is expressed in force per unit area; it can be normal (tension or compression) or shear.
- stress corrosion.** Preferential attack of areas under stress in a corrosive environment, where such an environment alone would not have caused corrosion.
- stress raisers.** Design features (sharp corners) or defects (notches) that act to intensify the stress at these locations.

stress relieving. Heating to a suitable temperature, holding sufficiently long to reduce residual stresses, then minimizing the development of additional residual stresses by slow cooling.

strike. A thin, electrodeposited film of metal.

stringer. (1) Long, narrow oxides between sprayed platelets. (2) In wrought materials, an elongated configuration of microconstituents or foreign material aligned in the direction of working.

strip. A flat-rolled metal product of some maximum thickness and width arbitrarily dependent on the type of metal; narrower than sheet.

stripper. A chemical solvent or solution used to remove a sprayed coating.

stripping. Removing a coating from a substrate.

structure. As applied to microstructure, the size, shape, and arrangement of phases, constituents, and so on.

sublime. To volatilize from the solid state to a gas.

submicron powder. Any powder whose particles are smaller than $\sim 1\ \mu\text{m}$.

submicroscopic. Below the resolution of a microscope.

subsieve analysis. Size distribution of particles that will pass through a standard 325-mesh sieve having $44\ \mu\text{m}$ openings.

subsieve fraction. Particles that will pass through a $44\ \mu\text{m}$ (325-mesh) screen.

subsieve size. See preferred term *subsieve fraction*.

substrate. The material, workpiece, or substance on which the coating is deposited.

substrate preparation. The set of operations, including cleaning, degreasing, and roughening, applied to the base material prior to applying a coating; intended to ensure adequate bonding of the coating.

substrate temperature. The temperature attained by the base material as the coating is applied.

subsurface corrosion. Formation of corrosion products beneath a metal surface.

sulfidation. The reaction of a metal with a sulfur-containing species to produce a sulfur compound. It forms on or beneath the surface of the metal or alloy.

superabrasives. Refers to synthetically produced diamond and cubic boron nitride (CBN). Diamond is the hardest material known; CBN is second.

superalloys. Heat-resistant alloys based on nickel (Ni), iron-nickel (Fe-Ni), or cobalt (Co) that exhibit mechanical properties and resistance to surface degradation unmatched by other metallic alloys.

superconductivity. A property of many substances that, at temperatures near absolute zero, lose their electrical resistivity and become strongly diamagnetic. See also *superconductors*.

superconductors. Materials that exhibit a complete disappearance of electrical resistance on lowering the temperature below a critical temperature. The critical temperatures may approach absolute zero.

superficial hardness test. See *Rockwell superficial hardness test*.

superfines. The portion of a powder that is composed of particles smaller than a specified size.

superfinishing. An abrasive process used to remove surface fragmentation and to correct inequalities in geometry, such as grinding feed marks and chatter marks. Also known as microhoning.

superheating. Any increment of temperature above the melting point of a metal.

supersaturated. A solution in which the dissolved material exceeds the amount that the solvent can hold in normal equilibrium at the existing conditions.

supersonic. Speed that is faster than that of sound.

surface area. (1) The area of all surfaces of a granular or powdered solid that is accessible to a penetrating gas or liquid. (2) The actual area of the surface to be coated.

surface damage. Damage to a solid surface resulting from mechanical contact with another substance. In certain contexts, wear is a form of surface damage in which material is progressively removed.

surface feet per minute (sfm). Linear velocity of the thermal spray gun as it traverses the length of the workpiece. Also, the circumferential velocity of the substrate.

surface film. Any continuous layer on the surface of a powder particle or workpiece.

surface finish. Condition of a surface as a result of a final treatment.

surface grinding. Producing a plane surface by grinding.

surface integrity. Technology that involves the specification and manufacture of unimpaired or enhanced surfaces through the control of the manufacturing process.

surface modification. Any alteration of a surface.

surface preparation. The operations necessary to produce a desired or specified surface condition.

surface roughness. Fine irregularities in the surface texture of a coating or material. It is usually reported as the arithmetic roughness average, R_a , and is given in micrometers or microinches.

surface tension. Force acting on the surface of a liquid, tending to minimize the surface area.

surface texture. The roughness, waviness, lay, and flaws associated with a surface.

surface topography. The geometrical detail of a surface as it relates to microscopic changes in height. See also *surface roughness*.

surface void. A void that is located at the surface of a coating.

surfacing. The deposition of material on a substrate.

swabbing. Wiping the surface of a metallographic specimen with a cotton ball. The ball may be wetted with an etchant to reveal specific microstructural characteristics.

T

Taguchi analysis. A mathematical approach to design of experiment advocated by Genichi Taguchi.

talc. A whitish, greenish, or grayish hydrated magnesium silicate, $\text{Mg}_3\text{Si}_4\text{O}_{10}(\text{OH})_2$, mineral that is extremely soft (hardness is 1 on the Mohs scale). It has a characteristic soapy or greasy feel.

tap density. The apparent density of a powder, obtained when the volume receptacle is tapped or vibrated during loading.

target efficiency. Percentage of total feedstock sprayed versus the amount actually deposited onto the substrate. It is a function of part geometry relative to spray pattern (not to be confused with *deposition efficiency*).

tarnish. Surface discoloration of a metal caused by formation of a thin film of corrosion product.

temper. Reheating a hardened steel or cast iron to some temperature below the eutectoid temperature to decrease hardness and increase toughness.

temper color. A thin, tightly adhering oxide film (only a few molecules thick) that forms when steel is tempered in air at a low temperature or for a short time. Oxide film color may range from straw to blue depending on its thickness, which varies with time and temperature.

tempered martensite. Decomposition products resulting from heating martensite below the ferrite-austenite transformation temperature.

tempering. Reheating hardened steel to some temperature below the eutectoid temperature to decrease hardness and/or increase toughness.

template (templet). A guide or a pattern.

tenacity. Term generally used to denote strength, adherence, or endurance.

tensile strength. The measure of resistance that a material offers to tensile stress. Expressed as the force per unit cross-sectional area required for fracture.

tensile stress. A stress that causes two parts of an elastic body, on either side of a typical stress plane, to pull apart. Contrast with *compressive stress*.

tensile testing. See *tension testing*.

tension testing. A method of determining the behavior of materials subjected to uniaxial loading, which tends to stretch the material. A longitudinal specimen of known length and diameter is gripped at both ends and stretched at a slow, controlled rate until rupture occurs. Also known as tensile testing.

ternary alloy. An alloy that contains three principal elements.

ternary system. The complete series of compositions produced by mixing three components in all proportions.

test. An evaluation procedure to determine the presence or absence of an attribute.

test result. Value obtained in testing.

test specimen. See preferred term *specimen*.

tetragonal. A crystal structure wherein all angles are 90° and the four sides are of equal length and greater than either end.

textural stress. The accumulated stresses within an entire coating.

theoretical density. The density of a solid, homogeneous piece of material.

thermal analysis. A method for determining transformations in a metal by noting the temperatures at which thermal arrests occur.

thermal barrier coating (TBC). Two or more layers of sprayed coating in which the initial coating that is closest to the substrate is a metal, generally a *MCrAlY*, and provides hot corrosion/oxidation protection as well as performing the function of a bond coat. Its coefficient of thermal expansion is generally tailored to match that of the host metal. Subsequent layers may be wholly refractory oxides or mixes of the metal and the ceramic. A TBC is a thermally insulating coating system protecting the substrate from the higher temperatures and corrosive products of the surrounding environment.

thermal conductivity. The ability of a material to conduct heat.

thermal cycling. The cyclic change in thermal environment.

thermal decomposition. Decomposition of a compound into its elemental species at elevated temperatures.

thermal expansion. Change in length of a material with a change in temperature. See also *coefficient of thermal expansion*.

thermal fatigue. Fracture resulting from the presence of temperature gradients that vary with time in such a manner as to produce cyclic stresses.

thermal resistance. A measure of the ability of a body to prevent heat from flowing through it, equal to the difference between the temperatures of opposite faces of the body divided by the rate of heat flow. Also known as heat resistance.

thermal shock. Development of a steep temperature gradient and accompanying high stresses within a material or structure.

thermal spray gun. A device for heating, feeding, and directing the flow of a thermal spray material.

thermal spraying. A group of coating processes in which finely divided metallic or nonmetallic materials are deposited in a molten or semimolten condition to form a coating. The coating material may be in the form of powder, ceramic rod, wire, or molten materials.

thermal spraying deposit. The coating or layer of surfacing material applied by a thermal spraying process.

thermal spray powder. A metal, carbide, plastic, or ceramic powder designed for use in thermal spray operations.

thermal stresses. Stresses in a material resulting from nonuniform temperature distribution.

thermal wear. (1) Material removal by softening, melting, or evaporation during sliding or rolling. (2) Transfer of atoms from one body to another at elevated temperatures.

thermit mixture. A mixture of a metal and finely divided aluminum with or without the addition of alloying metals. NiAl , NiCrAl , NiCrMoAl , and so on are examples of thermit reacting metal powders.

thermit reactions. Strongly exothermic self-propagating reactions such as that where finely divided aluminum reacts with a metal.

thermocouple. A device for measuring temperatures that consists of two dissimilar metals electrically joined at one end and

connected to a voltage-measuring instrument at the other. When one junction is hotter than the other, a thermal electromotive force is produced that is roughly proportional to the difference in temperature between the hot and cold junctions.

thermoelectromotive force. Corrosion resulting from an electrochemical cell caused by a thermal gradient.

thermogravimetric analysis (TGA). The study of the change in mass of a material under various conditions of temperature and pressure.

thermoplastic. Capable of being repeatedly softened by an increase in temperature and hardened by a decrease in temperature.

thermoset. A resin that is cured, set, or hardened, usually by heating, into a permanent shape. The reaction is irreversible.

thinner. A volatile liquid added to modify the consistency or other properties.

threading. Producing external threads on a cylindrical surface.

threading and knurling. A method of surface roughening in which spiral threads are prepared, followed by upsetting with a knurling tool.

tin and tin alloys. A malleable, brilliant white, metallic element with a low melting point. It is used as a corrosion-preventative coating and is often alloyed with other metals to form solders, bronzes, and pewters.

titanium and titanium alloys. Titanium is the fourth most abundant metal in the earth's crust. Titanium alloys exhibit low densities (approximately 60% that of steel) while providing high structural efficiencies with a wide range of mechanical properties and excellent resistance to aggressive environments.

titanium carbide (TiC). Hard, heat-resistant ceramic used in cermets and tungsten carbide cutting tools. See also *cermet*.

titanium nitride (TiN). A hard, high-melting-point ceramic (2950 °C, or 5342 °F) used in wear-resistant coatings.

titration. A method of determining the composition of a sample by adding known volumes of a known solution until a reaction (precipitation or color/conductivity change) is produced.

tolerance. The permissible deviation from a specified nominal dimension, or the permissible variation in size or other quality characteristic of a part.

tolerance limits. The extreme values (upper and lower) that define the range of permissible variation in size or other quality characteristic of a part. See also *quality characteristics*.

tooling. Term denoting masking, fixturing, or related items used for preparing, protecting, and/or locating parts for spraying.

tooling marks. Indications imparted to the surface of a part. These are usually slight rises or depressions in the surface.

tool steels. Any steel used to make tools for cutting, forming, or otherwise shaping a material into a final part component. In addition to carbon, they may also contain chromium, tungsten, molybdenum, vanadium, and manganese.

tooth. A projection on a multipoint tool.

torch. A device used for fusing sprayed coatings; it mixes and controls the flow of gases.

torch fusing. Using a torch to heat and melt a fusible spraying deposit to produce coalescence.

torch tip. That part of an oxyfuel gas torch from which the gases exit.

torsion. (1) A twisting deformation of a body about an axis in which lines that were initially parallel to the axis become helices. (2) A twisting action resulting in shear stresses and strains.

torsional stress. The shear stress on a transverse cross section caused by a twisting action.

total carbon. The sum of both the free and combined carbon (including carbon in solution) in a ferrous alloy.

toughness. Ability of a material to absorb energy and deform plastically before fracturing.

transcrystalline. See *transgranular*.

transcrystalline cracking. Cracking or fracturing that occurs through or across a crystal. Also called *intracrystalline cracking*.

transferred arc. An arc established between the plasma torch and the workpiece. The workpiece is part of the electrical circuit. Can be reversed to preheat and clean a substrate surface.

transformation temperature. The temperature at which a change in phase occurs. It is sometimes used to denote the limiting temperature of a transformation range.

transgranular. Through or across crystals or grains. Also called *intracrystalline* or *transcrystalline*.

transgranular cracking. Cracking or fracturing that occurs through or across a crystal or grain.

transistor. An active semiconductor device capable of providing power amplification.

transmission electron microscopy (TEM). An analytical technique in which an image is formed on a cathode-ray tube whose raster is synchronized with the raster of an electron beam over an area of the sample surface. Image contrast is formed by the scattering of electrons out of the beam.

transverse direction. Literally, "across," usually signifying a direction or plane perpendicular to the direction of spraying.

transverse section. A metallographic section taken in the same plane as the direction of spray. Sprayed particles are observed as flattened, narrow, lenticular splats.

traverse speed. The linear velocity at which the spray gun traverses across the workpiece during the spraying operation.

tribology. The science concerned with the design, friction, lubrication, and wear of contacting surfaces that move relative to each other (as in bearings, cams, or gears, for example).

Troy ounce. A unit of weight for precious metals that is equal to 31.1034768 g (1.0971699 oz avoirdupois).

tryout. A preparatory run to check or test equipment, stock, tools, or methods prior to a production run.

tube stock. A semifinished tube suitable for subsequent finishing.

tumbling. Rotating workpieces in a barrel filled with abrasives to remove sand, scale, and so on. It may be done dry or with an aqueous solution added to the contents of the barrel.

tungsten carbide. See *cemented carbides*.

tungsten electrode. A non-filler metal electrode used in a plasma arc gun. The negative electrode, it is made principally of tungsten.

turning. Removing material by forcing a single-point cutting tool against the surface of a rotating workpiece.

twist. See *bend or twist*.

U

ultimate strength. The maximum stress (tensile, compressive, or shear) a material can sustain without fracture.

ultimate tensile strength. The ultimate or final (highest) stress sustained by a specimen in a tension test. Determined by dividing maximum load by the original cross-sectional area of the specimen.

ultramicroscopic. See *submicroscopic*.

ultrasonic cleaning. Immersion cleaning aided by ultrasonic waves that cause microagitation.

ultrasonic inspection. A nondestructive test method in which beams of high-frequency sound waves are introduced into materials to detect flaws.

ultraviolet (UV). Pertaining to the region of the electromagnetic spectrum from approximately 10 to 380 nm.

unbond. An area between the substrate and coating in which bonding has failed to occur.

undercoat. A deposited coat of material that acts as a substrate for a subsequent thermal spraying deposit. See also *bond coat*.

undercutting. A surface preparation step involving the removal of substrate material.

underlayment. The primary or initial coating. Typically, a bond coat.

undersize powder. Powder particles smaller than the minimum permitted by a particle size specification.

undulating. The wavelike rising, falling, and rolling of sprayed particles, oxides, and voids over and around themselves and neighboring particles.

uniform corrosion. (1) A type of corrosion (deterioration) uniformly distributed over a metal surface. (2) Corrosion that proceeds at approximately the same rate over a metal surface.

unit. An object or part on which a measurement or observation may be made.

V

vacuum plasma deposition (VPD). Plasma spraying under low pressure conditions, usually at approximately 7 kPa (50 torr).

vacuum plasma spray (VPS). See preferred term *vacuum plasma deposition (VPD)*.

van der Waals bond. A secondary bond arising from the fluctuating dipole nature of an atom with all occupied electron shells filled.

vapor. The gaseous form of substances normally in a solid or liquid state; they can revert to those states either by increasing the pressure or decreasing the temperature.

vapor degreasing. Degreasing of work in a vapor over a boiling solvent.

vapor deposition. See *chemical vapor deposition (CVD)*.

variance. A measure of the squared dispersion of observed values or measurements expressed as a function of the sum of the squared deviations from the population mean or sample average.

varnish. A transparent coating applied as a liquid that hardens to a clear film; varnishes are solutions of resinous materials in a solvent.

V-cone blender. A device for blending powders that has two cylindrical containers arranged in a V and open to each other at their junction point. See also *blending*.

verification. Checking or testing an instrument to ensure conformance with accepted standards.

vermiculite. A granular, clay mineral constituent used as a filler or insulating material.

vernier. A short auxiliary scale that slides along the main instrument scale to permit more accurate fractional reading of the least main division of the main scale.

vibration density. The apparent density of a powder mass when the volume receptacle is vibrated under specified conditions. Similar to *tap density*. See also *apparent density*.

vibratory finishing. Surface finishing in which the product and an abrasive mixture are placed in a container and vibrated.

vibratory polishing. Mechanical polishing in which a metallographic specimen rotates around the polishing cloth by imparting a suitable vibratory motion to the polishing system.

Vickers hardness number (HV). A number related to the applied load and the surface area of the permanent impression made by a diamond indenter having included face angles of 136°.

Vickers hardness test. A microindentation hardness test employing a 136° diamond pyramid indenter (Vickers) and variable loads. Also known as *diamond pyramid hardness test*. See also *microindentation* and *microindentation hardness number*.

virgin material. A material that has not been subjected to any processing other than its initial manufacture.

viscosity. The bulk property of a substance that causes it to resist flow.

viscous. Possessing viscosity; frequently used to imply high viscosity.

vitreous. Partially or completely comprised of a glass.

vitrification. The formation of a glassy or noncrystalline material.

void. A shrinkage cavity produced during spraying or solidification.

void content. Volume percentage of voids.

volatiles. Materials capable of being driven off as a vapor at room temperature or at slightly elevated temperature.

volatilization. The conversion of substance from a liquid or solid state to a gaseous or vapor state by the application of heat, reducing pressure, or a combination of these. Also known as vaporization.

volt. The unit of potential difference or electromotive force in the meter-kilogram-second system, equal to the potential difference between two points for which 1 coulomb of electricity will do 1 joule of work in going from one point to the other. Symbolized V.

voltage drop. The amount of voltage loss from original input in a conductor of given size and length.

voltage regulator. An automatic electrical control device for maintaining a constant voltage supply.

volume percent (vol %). A powder blend or mixture in which the components are measured by volume rather than weight.

vortex-stabilized plasma. A plasma device in which gas is fed tangentially into the chamber between the front and rear electrodes to produce an intense vortex (rotation) at the tip of the cathode, with a corresponding low pressure area at the center of the vortex.

vulcanization. Changing the physical properties of a rubber in the direction of decreased plastic flow, less surface tackiness, and increased tensile strength by reacting it with sulfur or other suitable agents in the presence of heat and pressure.

W

warp. A distortion or significant variation from the original true, or plane, surface or shape.

warpage. The distortion that can occur during high-temperature service or processing.

water absorption. The ratio of the weight of water absorbed by a material to the weight of the dry material.

wear. Surface damage involving a progressive loss of material due to relative motion between it and a contacting surface or substance.

wear debris. Particles that become detached in a wear process.

wear rate. Rate of material removal or dimensional change due to wear per unit of exposure, for example, quantity of material removed (mass, volume, thickness).

wear resistance. Resistance of a body to removal of material by a wear process. It is a function of the conditions under which wear takes place and should be specified.

wear scar. The portion of a surface that exhibits evidence that material has been removed from it due to wear.

weathering. The result of exposure of materials to the environment.

welding. Joining two or more pieces of metal by applying heat or pressure, or both, with or without filler metal, to produce a localized union through fusion or recrystallization across the interface.

wet, wettability. That property of a liquid to spread over a surface due to a low contact angle.

wet blasting. Process for cleaning or finishing by means of directing an abrasive slurry at high velocity against the workpieces.

wetting. The spreading, and sometimes absorption, of a fluid over or into a surface.

whisker. A short, single-crystal fiber or filament used as a reinforcement in a matrix.

white metal. Term describing the visual appearance of a clean metallic surface after grit blasting.

wire. A thin, flexible, continuous length of metal, usually of circular cross section, and usually produced by drawing through a die.

wire arc spraying. See preferred term *electric arc spraying*.

wire feed speed. Rate of speed in millimeters per second (mm/s) or inches per minute (in./min) in which a filler metal is consumed during spray.

wire flame spray gun. A flame spraying device using an oxyfuel flame to provide the heat; the spray material is in wire or rod form. See also *wire flame spraying*.

wire flame spraying. Spray process in which the feed stock is in wire or rod form. See also *flame spraying*.

wire metallizing. See preferred term *wire flame spraying*.

wire straightener. Device for controlling the cast of coiled metal wire, enabling it to be readily fed into the wire spray gun.

work distance. The defined distance between the substrate surface and the nozzle of the spray gun.

work hardening. An increase in hardness and strength of metals caused by plastic deformation at temperatures below the recrystallization range.

workpiece. The object or surface to be coated. See also *substrate*.

workpiece cooler. See preferred term *auxiliary cooler*.

X

x-ray. A penetrating electromagnetic radiation, usually generated by accelerating electrons to high velocity and suddenly stopping them by collision with a solid body.

x-ray diffraction (XRD). An analytical technique in which measurements are made of the angles at which x-rays are preferentially scattered from a sample in order to deduce information on the crystalline nature of the sample.

x-ray fluorescence. Emission by a substance of its characteristic x-ray line spectrum on exposure to x-rays.

Y

yield strength. Stress at which a material exhibits a specified deviation from proportionality of stress and strain. An offset of 0.2% is used for many materials, particularly metals.

Young's modulus. The ratio of tensile or compressive stresses to the resulting strain. See also *modulus of elasticity*.

yttria. A rare earth oxide of composition Y_2O_3 .

Z

zinc. An elemental metal. Its alloys and chemical compounds represent the fourth most industrially utilized metal. The largest single application for zinc is for the corrosion protection of iron and steel.

zirconia (ZrO_2). A white powder that naturally occurs as the mineral baddeleyite. However, most zirconia is produced from zircon, which is found in zirconium silicate sand ($\text{ZrO}_2 \cdot \text{SiO}_2$). Zirconia exhibits three polymorphs: monoclinic, tetragonal, and cubic. Monoclinic is stable up to approximately 1170 °C (2140 °F), where it transforms to tetragonal, which is stable up to 2370 °C (4300 °F), while the cubic phase exists up to the

melting point of 2680 °C (4855 °F). Of significance is the tetragonal-to-monoclinic transformation, unusual in that, on cooling through the transformation temperature (the monoclinic), there is a large volume change (3 to 5%). This is sufficient to exceed elastic and fracture limits and can only be accommodated by cracking. Additives such as calcia (CaO), magnesia (MgO), yttria (Y_2O_3), or ceria (CeO_2) must be alloyed with ZrO_2 to stabilize it in either the tetragonal or cubic phase.

zirconium. A lustrous, grayish-white metal obtained from zircon (ZrSiO_4), which occurs throughout the world in the form of beach sand. It is ranked 19th in abundance.

Abbreviations and Acronyms*

a acceleration	cal calorie
A amperage; ampere; area	CAPS controlled atmospheric plasma system
Å angstrom (10^{-10} m)	CASS copper-accelerated acetic acid-salt spray (test)
AA arithmetic average roughness; atomic absorption; Aluminum Association	CBN cubic boron nitride
ac alternating current; adaptive control; air cooled	CCD charge-coupled device
ACerS American Ceramic Society	CDS continuous detonation spray
ACGIH American Congress on Government and Industrial Hygiene	CEA Commissariat à l'Energie Atomique (French atomic energy center)
ACS American Chemical Society	CEAC Connecticut Engineering Associates Corporation
AESF The American Electroplaters and Surface Finishers Society, Inc.	CEREM Centre d'étude et de Recherche Matériaux
AGA American Gas Association	CFH cubic feet per hour
AISI American Iron and Steel Institute	cfm cubic feet per minute
AM&P <i>Advanced Materials and Processes</i> (magazine)	C(g) carbon in graphitic form
AMCA American Metallizing Contractors Association	CGA Compressed Gas Association
AMDRY atomized dry	ChamPro chamber processes
AMS Aerospace Material Specification; acoustic material signature	CIP cold isostatic pressing
ANSI American National Standards Institute	cm centimeter
API American Petroleum Institute	CNC computer numerical control
APMI American Powder Metallurgy Institute	CoCrAlY a cobalt (Co), chromium (Cr), aluminum (Al), yttrium (Y) alloy
APS air plasma spray	cos cosine of an angle
ASLE American Society of Lubrication Engineers (former name of Society of Tribologists and Lubrication Engineers, or STLE)	cot cotangent of an angle
ASM ASM International	CP commercially pure
ASME American Society of Mechanical Engineers	CRS cold rolled steel
ASTM American Society for Testing and Materials	CRT cathode ray tube
ATCS advanced thermal coating system	CTE coefficient of thermal expansion; <i>see also</i> α (alpha)
atm atmosphere (pressure)	CVD chemical vapor deposition
AVEM Association of Vacuum Equipment Manufacturers	d day
AVS American Vacuum Society	d depth; diameter
AWG American wire gage	DARPA Defense Advanced Research Projects Agency
AWS American Welding Society	dB decibel
BABS British Association for Brazing and Soldering	dc direct current
bcc body-centered cubic	DE deposit efficiency
BIL/IBS Belgisch Instituut voor Lastechiek/Institut Belge de la Soudure	d.f. degrees of freedom
BN boron nitride	D-gun detonation gun
BS British Standard	diam diameter
Btu British thermal unit	DIN Deutsche Industrie-Normen (German Industrial Standards)
CAD/CAM computer-aided design/computer-aided manufacturing	DJ diamond jet
	dm decimeter
	DOC Department of Commerce
	DoD Department of Defense
	DOE design of experiment; Department of Energy

*Adapted from *Thermal Spray Terminology and Company Origins*, F.J. Hermanek, Ed., ASM Thermal Spray Society, 2001

DOT Department of Transportation	h height; hour
DPH diamond pyramid hardness (Vickers hardness); <i>see also</i> HV and VHN	H_s substrate hardness; scratch hardness
DR deposition rate	HA hydroxyapatite
DSC differential scanning calorimetry	HAZ heat-affected zone
DTA differential thermal analysis	HB Brinell hardness
DVS Deutschen Verband für Schweiss-technik e.V. (German Welding Society)	HBN or hBN hexagonal boron nitride
EAW electric arc wire (spray)	HCAT Hard Chrome Alternatives Team
EB electron beam	HEM hydrogen embrittlement
EB-PVD electron beam-physical vapor deposition	hex-Cr hexavalent chromium (Cr)
ECM electrochemical machining	HFPD high-frequency pulse detonation
EDM electrical discharge machining	HIP hot isostatic pressing
EDS energy-dispersive spectroscopy of x-rays	HK Knoop hardness
EDXA energy-dispersive x-ray analysis (pronounced “edax”)	HOSP hollow oxide spherical particle
EEC European Economic Community	hp horsepower
EHC electrolytic hard chrome	HP high purity; hot pressed; high pressure
emf electromotive force	HPC high-pressure compressor
EPA Environmental Protection Agency	HPT high-pressure turbine
EPMA electron probe microanalysis	HR Rockwell hardness (requires scale designation, such as HRC for Rockwell C hardness)
ESD Engineering Society of Detroit	HRS hot rolled steel
et al. and others	HRTEM high-resolution transmission electron microscope
eV electron volt	HSc or HSd Scleroscope hardness number
F force	HSS high-speed steel(s)
FAA Federal Aviation Administration	HTSJ High Temperature Society of Japan
FBCVD fluidized-bed chemical vapor deposition	HV Vickers hardness
fcc face-centered cubic	HVAF high-velocity air fuel
FDA Food and Drug Administration	HVIF high-velocity impact fusion spraying
FeCrAlY an iron (Fe), chromium (Cr), aluminum (Al), yttrium (Y) alloy	HVOF high-velocity oxyfuel spraying
FGM functionally gradient material	HV_P Vickers hardness at applied force of <i>P</i>
FHC Federal Highway Commission	Hz hertz
Fig. figure	ICP inductively coupled plasma
FPI fluorescent particle inspection	ID inside diameter
fps feet per second; frames per second	IEEE Institute of Electrical and Electronics Engineers
FS flame spray	IGA intergranular attack
FSZ fully stabilized zirconia	IIW International Institute of Welding
ft foot	in. inch
FTIR Fourier transform infrared (spectroscopy)	INEEL Idaho Engineering and Environmental Laboratory
g gram	IP conventional ion plating
gal gallon	ipm inches per minute
GC gas chromatography	ips inches per second
GMAW gas metal arc welding	IR infrared
GPa gigapascal	IS Institut de Soudure
gr grain	ISO International Organization for Standardization
GTA gas tungsten arc	ITSA International Thermal Spray Association
GTAW gas tungsten arc welding	ITSC International Thermal Spray Conference
GTS Gemeinschaft Thermisches Spritzen e.V. (Association of Thermal Spraying [German])	IUPAC International Union of Pure and Applied Chemistry
	IUVSTA International Union for Vacuum Science, Technique, and Applications
	J joule

JIT just in time	min minute; minimum
JTSS Japanese Thermal Spray Society	MIT Massachusetts Institute of Technology
JTST <i>Journal of Thermal Spray Technology</i>	ml milliliter
K Kelvin	mL milliliter
K coefficient of thermal conductivity	MLG main landing gear
kbar kilobar	mm millimeter
kg kilogram	MMC metal-matrix composite
kgf kilogram force	MMI man machining interface
km kilometer	mo month
kPa kilopascal	MOCVD metal-organic chemical vapor deposition
ksi kips (1000 lb) per square inch	mPa millipascal
kV kilovolt	MPa megapascal
kVA kilovolt ampere	mpg miles per gallon
kW kilowatt	mph miles per hour
l length; liter	MPIF Metal Powder Industries Federation
L longitudinal; liter	ms millisecond
L_c characteristic length	MSA Microscopy Society of America
LACVD laser-assisted chemical vapor deposition	MSC Metallizing Service Contractors
lb pound	MSDS material safety data sheet
lbf pound force	mV millivolt
LCF low-cycle fatigue	MW megawatt; molecular weight
LCVD laser-induced chemical vapor deposition	N Newton
LED light-emitting diode	NA or na not applicable
L/h or l/h liter per hour	NACE National Association of Corrosion Engineers
LIF laser-induced fluorescence	NASA National Aeronautics and Space Administration
LinTrav linear traverse	NBS National Bureau of Standards (former name of National Institute of Standards and Technology, or NIST)
L/min or l/min liter per minute	NC numerical control
ln natural logarithm (base <i>e</i>)	NDE nondestructive evaluation
log common logarithm (base 10)	NDT nondestructive testing
LOX liquid oxygen	NEMA National Electrical Manufacturers Association
LPPS low-pressure plasma spray	NER erosion resistance number
LPT low-pressure turbine	NFPA National Fire Prevention Association
LT long transverse (direction)	NiCrAlY a nickel (Ni), chromium (Cr), aluminum (Al), yttrium (Y) alloy
m meter	NIL Nederlands Instituut voor Lastechniek (Netherlands Institute for Lasertechniek)
m mass	NIOSH National Institute of Occupational Safety and Health
MAPP methyl-acetylene-propadiene (stabilized gas)	NIST National Institute of Standards and Technology
max maximum	NLG nose landing gear
MB megabyte	NLPM normal liter per minute
MCrAlY a metal (M) alloy with chromium (Cr), aluminum (Al), and yttrium (Y)	nm nanometer
MEK methyl ethyl ketone	NMR nuclear magnetic resonance
MFC mass flow controller	n/n not named
MFM mass flowmeter	No. number
mg milligram (0.001 gram)	NRL Naval Research Laboratories
Mg megagram (10 ⁶ gram, metric tonne, or kg × 10 ³)	NR noise reduction rating
MHz megahertz	ns nanosecond
MIG metal inert gas (welding); <i>see also</i> GMAW	NSP not specified
MIL-STD military standard	
MIM metal injection molding	

NTSC National Thermal Spray Conference	Ref reference
OD outside diameter	rem remainder
ODS oxide dispersion strengthened	RF or rf radio frequency
OEM original equipment manufacturer	rms root mean square
OFD oxyfuel detonation (spray)	rpm revolutions per minute
OFHC oxygen-free high conductivity (copper)	RT room temperature
OFP oxyfuel powder (spray)	RTA reverse transferred arc
OFW oxyfuel wire (spray)	s second
OSHA Occupational Safety and Health Administration	S_u ultimate strength
oz ounce	S_y yield strength
p page	SA Société Anonyme
p pressure; hydrostatic pressure acting on the surface	SAE Society of Automotive Engineers
P specific load	SANS scanning-angle neutron scattering
Pa pascal	SCFH or scfh standard cubic feet per hour
PA plasma arc (spray); prealloyed; polyamide	scfm standard cubic feet per minute
PA-CVD plasma-assisted chemical vapor deposition	SCR silicon-controlled rectifier
PA-PVD plasma-assisted physical vapor deposition	SEM scanning electron microscope/microscopy
PE-CVD plasma-enhanced chemical vapor deposition	sfm surface feet per minute
PEL permissible exposure limits	SG spheroidal graphite
pH negative logarithm of hydrogen-ion activity	SHS self-propagating high-temperature synthesis
PH precipitation hardenable	sin sine of an angle
PI polyimide	sLm standard liters per minute
PLC programmable logic controller	slpm standard liters per minute
P/M powder metallurgy	SME Society of Manufacturing Engineers
PMMA polymethyl methacrylate	SNECMA Societe National d'Etude et de Construction de Moteurs d'Aviation (French)
POD pin on disk	SOFC solid oxide fuel cells
POF pin on flat	SPC statistical process control
ppb parts per billion	sp gr specific gravity
ppm parts per million	SS stainless steel
PROTAL projection thermique assistée par laser	SSPC Steel Structures Painting Council (now The Society for Protective Coatings)
PS plasma spray	std standard
psi pounds per square inch	STEM scanning transmission electron microscope/microscopy
psia pounds per square inch (absolute)	SVC Society of Vacuum Coaters
psig pounds per square inch, gage pressure (pressure relative to ambient pressure)	t thickness; time
PSZ partially stabilized zirconia	T temperature
PTA plasma transferred arc	T tesla
PUR polyurethane	tan tangent of an angle
PVC polyvinyl chloride	TBC thermal barrier coating
PVD physical vapor deposition	tc thermocouple
Q thermal energy generated per unit time	TE target efficiency
r or R radius; radial distance of receiver from source; resistivity	TEM transmission electron microscope/microscopy
R Rankine	TGA thermogravimetric analysis
R_a surface roughness in terms of arithmetic average	TIG tungsten inert gas (welding); <i>see also</i> GTAW
R_q root mean square roughness	TLV threshold limit value
R_z maximum peak-to-valley roughness	TMS The Minerals, Metals and Materials Society
Rad radians	TP thermoplastic
RAM random access memory	TS thermal spray or spraying
RE rare earth	

TSS ASM International Thermal Spray Society
TSS-HOF Thermal Spray Society-Hall of Fame
TSVP thermal spray vitrification process
TWA time-weighted average
TWI The Welding Institute
UL Underwriter's Laboratories
USAF United States Air Force
USDA United States Department of Agriculture
UTP Universal Tiefpunkt (Universal Welding-Penetration Welding)
UTS ultimate tensile strength
UTSC United Thermal Spray Conference
UV ultraviolet
v velocity
V volt
V_p particle velocity
VAMAS Versailles Advanced Materials and Standards
VHN Vickers microindenter hardness number
V/O volume percent
VOC volatile organic compound
vol volume
vol% or **v/o** volume percent
VPD vacuum plasma deposition
VPS vacuum plasma spray
W width; mass losses
W watt; load; bearing load-carrying parameter; weight of body; abrasive wear resistance; wear volume
WIC Welding Institute of Canada
wk week
WSP water-stabilized plasma
wt% or **w/o** weight percent
XRD x-ray diffraction
XRF x-ray fluorescence
yr year
YSZ yttria-stabilized zirconia
ZTA zirconia-toughened alumina
 ° degree; angular measurement and temperature

°C degrees Celsius; centigrade
 °F degrees Fahrenheit
 % percent
 √ surface roughness
 α coefficient of thermal expansion (alpha)
 Δ change in quantity
 μin. microinch
 μm micrometer; micron
 ρ density
 Ω ohm

Greek Alphabet

A, α alpha
B, β beta
Γ, γ gamma
Δ, δ delta
E, ε epsilon
Z, ζ zeta
H, η eta
Θ, θ theta
I, ι iota
K, κ kappa
Λ, λ Lambda
M, μ mu
N, ν nu
Ξ, ξ xi
O, ο omicron
Π, π pi
P, ρ rho
Σ, σ sigma
T, τ tau
Υ, υ upsilon
Φ, φ phi
X, χ chi
Ψ, ψ psi
Ω, ω omega

Metric Conversion Guide

This Section is intended as a guide for expressing weights and measures in the Système International d'Unités (SI). The purpose of SI units, developed and maintained by the General Conference of Weights and Measures, is to provide a basis for worldwide standardization of units and measure. For more information on metric conversions, the reader should consult the following references:

- *The International System of Units*, SP 330, 1991, National Institute of Standards and Technology. Order from Superintendent of Documents, U.S. Government Printing Office, Washington, DC 20402-9325
- *Metric Editorial Guide*, 5th ed. (revised), 1993, American National Metric Council, 4340 East West Highway, Suite 401, Bethesda, MD 20814-4411
- "Standard for Use of the International System of Units (SI): The Modern Metric System," IEEE/ASTM SI 10-1997, Institute of Electrical and Electronics Engineers, 345 East 47th Street, New York, NY 10017, USA
- *Guide for the Use of the International System of Units (SI)*, SP 811, 1995, National Institute of Standards and Technology, U.S. Government Printing Office, Washington, DC 20402

Base, supplementary, and derived SI units

Measure	Unit	Symbol	Measure	Unit	Symbol
Base units					
Amount of substance	mole	mol	Force	newton	N
Electric current	ampere	A	Frequency	hertz	Hz
Length	meter	m	Heat capacity	joule per kelvin	J/K
Luminous intensity	candela	cd	Heat flux density	watt per square meter	W/m ²
Mass	kilogram	kg	Illuminance	lux	lx
Thermodynamic temperature	kelvin	K	Inductance	henry	H
Time	second	s	Irradiance	watt per square meter	W/m ²
Supplementary units			Luminance	candela per square meter	cd/m ²
Plane angle	radian	rad	Luminous flux	lumen	lm
Solid angle	steradian	sr	Magnetic field strength	ampere per meter	A/m
Derived units			Magnetic flux	weber	Wb
Absorbed dose	gray	Gy	Magnetic flux density	tesla	T
Acceleration	meter per second squared	m/s ²	Molar energy	joule per mole	J/mol
Activity (of radionuclides)	becquerel	Bq	Molar entropy	joule per mole kelvin	J/mol · K
Angular acceleration	radian per second squared	rad/s ²	Molar heat capacity	joule per mole kelvin	J/mol · K
Angular velocity	radian per second	rad/s	Moment of force	newton meter	N · m
Area	square meter	m ²	Permeability	henry per meter	H/m
Capacitance	farad	F	Permittivity	farad per meter	F/m
Concentration (of amount of substance)	mole per cubic meter	mol/m ³	Power, radiant flux	watt	W
Current density	ampere per square meter	A/m ²	Pressure, stress	pascal	Pa
Density, mass	kilogram per cubic meter	kg/m ³	Quantity of electricity, electric charge	coulomb	C
Dose equivalent, dose equivalent index	sievert	Sv	Radiance	watt per square meter steradian	W/m ² · sr
Electric charge density	coulomb per cubic meter	C/m ³	Radiant intensity	watt per steradian	W/sr
Electric conductance	siemens	S	Specific heat capacity	joule per kilogram kelvin	J/kg · K
Electric field strength	volt per meter	V/m	Specific energy	joule per kilogram	J/kg
Electric flux density	coulomb per square meter	C/m ²	Specific entropy	joule per kilogram kelvin	J/kg · K
Electric potential, potential difference, electromotive force	volt	V	Specific volume	cubic meter per kilogram	m ³ /kg
Electric resistance	ohm	Ω	Surface tension	newton per meter	N/m
Energy, work, quantity of heat	joule	J	Thermal conductivity	watt per meter kelvin	W/m · K
Energy density	joule per cubic meter	J/m ³	Velocity	meter per second	m/s
Entropy	joule per kelvin	J/K	Viscosity, dynamic	pascal second	Pa · s
			Viscosity, kinematic	square meter per second	m ² /s
			Volume	cubic meter	m ³
			Wavenumber	1 per meter	1/m

Conversion factors

To convert from	to	multiply by	To convert from	to	multiply by	To convert from	to	multiply by
Angle			Heat input			Pressure (fluid)		
degree	rad	1.745 329 E - 02	J/in.	J/m	3.937 008 E + 01	atm (standard)	Pa	1.013 250 E + 05
Area			kJ/in.	kJ/m	3.937 008 E + 01	bar	Pa	1.000 000 E + 05
in. ²	mm ²	6.451 600 E + 02	Impact energy per unit area			in. Hg (32 °F)	Pa	3.386 380 E + 03
in. ²	cm ²	6.451 600 E + 00	Length			in. Hg (60 °F)	Pa	3.376 850 E + 03
in. ²	m ²	6.451 600 E - 04	ft · lbf/ft ²	J/m ²	1.459 002 E + 01	lbf/in. ² (psi)	Pa	6.894 757 E + 03
ft ²	m ²	9.290 304 E - 02	Mass			torr (mm Hg, 0 °C)	Pa	1.333 220 E + 02
Bending moment or torque			Å	nm	1.000 000 E - 01	Specific heat		
lbf · in.	N · m	1.129 848 E - 01	μin.	μm	2.540 000 E - 02	Btu/lb · °F	J/kg · K	4.186 800 E + 03
lbf · ft	N · m	1.355 818 E + 00	mil	μm	2.540 000 E + 01	cal/g · °C	J/kg · K	4.186 800 E + 03
kgf · m	N · m	9.806 650 E + 00	in.	mm	2.540 000 E + 01	Stress (force per unit area)		
ozf · in.	N · m	7.061 552 E - 03	in.	cm	2.540 000 E + 00	tonf/in. ² (tsi)	MPa	1.378 951 E + 01
Bending moment or torque per unit length			ft	m	3.048 000 E - 01	kgf/mm ²	MPa	9.806 650 E + 00
lbf · in./in.	N · m/m	4.448 222 E + 00	yd	m	9.144 000 E - 01	ksi	MPa	6.894 757 E + 00
lbf · ft/in.	N · m/m	5.337 866 E + 01	mile, international	km	1.609 344 E + 00	lbf/in. ² (psi)	MPa	6.894 757 E - 03
Current density			mile, nautical	km	1.852 000 E + 00	MN/m ²	MPa	1.000 000 E + 00
A/in. ²	A/cm ²	1.550 003 E - 01	mile, U.S. statute	km	1.609 347 E + 00	Temperature		
A/in. ²	A/mm ²	1.550 003 E - 03	Mass			°F	°C	5/9 · (°F - 32)
A/ft ²	A/m ²	1.076 400 E + 01	oz	kg	2.834 952 E - 02	°R	K	5/9
Electricity and magnetism			lb	kg	4.535 924 E - 01	K	°C	K - 273.15
gauss	T	1.000 000 E - 04	ton (short, 2000 lb)	kg	9.071 847 E + 02	Temperature interval		
maxwell	μWb	1.000 000 E - 02	ton (short, 2000 lb)	kg × 10 ³ (a)	9.071 847 E - 01	°F	°C	5/9
mho	S	1.000 000 E + 00	ton (long, 2240 lb)	kg	1.016 047 E + 03	Thermal conductivity		
Oersted	A/m	7.957 700 E + 01	Mass per unit area			Btu · in./s · ft ² · °F	W/m · K	5.192 204 E + 02
Ω · cm	Ω · m	1.000 000 E - 02	oz/in. ²	kg/m ²	4.395 000 E + 01	Btu/ft · h · °F	W/m · K	1.730 735 E + 00
Ω circular-mil/ft	μΩ · m	1.662 426 E - 03	oz/ft ²	kg/m ²	3.051 517 E - 01	Btu · in./h · ft ² · °F	W/m · K	1.442 279 E - 01
Energy (impact, other)			oz/yd ²	kg/m ²	3.390 575 E - 02	cal/cm · s · °C	W/m · K	4.184 000 E + 02
ft · lbf	J	1.355 818 E + 00	lb/ft ²	kg/m ²	4.882 428 E + 00	Thermal expansion		
Btu (thermochemical)	J	1.054 350 E + 03	Mass per unit length			in./in. · °C	m/m · K	1.000 000 E + 00
cal (thermochemical)	J	4.184 000 E + 00	lb/ft	kg/m	1.488 164 E + 00	in./in. · °F	m/m · K	1.800 000 E + 00
Cal (nutritional)	J	4.184 000 E + 03	lb/in.	kg/m	1.785 797 E + 01	Velocity		
kW · h	J	3.600 000 E + 06	Mass per unit time			ft/h	m/s	8.466 667 E - 05
W · h	J	3.600 000 E + 03	lb/h	kg/s	1.259 979 E - 04	ft/min	m/s	5.080 000 E - 03
Flow rate			lb/min	kg/s	7.559 873 E - 03	ft/s	m/s	3.048 000 E - 01
ft ³ /h	L/min	4.719 475 E - 01	lb/s	kg/s	4.535 924 E - 01	in./s	m/s	2.540 000 E - 02
ft ³ /min	L/min	2.831 000 E + 01	Mass per unit volume (includes density)			km/h	m/s	2.777 778 E - 01
gal/h	L/min	6.309 020 E - 02	g/cm ³	kg/m ³	1.000 000 E + 03	mph	km/h	1.609 344 E + 00
gal/min	L/min	3.785 412 E + 00	lb/ft ³	g/cm ³	1.601 846 E - 02	Velocity of rotation		
Force			lb/ft ³	kg/m ³	1.601 846 E + 01	rev/min (rpm)	rad/s	1.047 164 E - 01
lbf	N	4.448 222 E + 00	lb/in. ³	g/cm ³	2.767 990 E + 01	rev/s	rad/s	6.283 185 E + 00
kip (1000 lbf)	N	4.448 222 E + 03	lb/in. ³	kg/m ³	2.767 990 E + 04	Viscosity		
tonf	kN	8.896 443 E + 00	Power			poise	Pa · s	1.000 000 E - 01
kgf	N	9.806 650 E + 00	Btu/s	kW	1.055 056 E + 00	stokes	m ² /s	1.000 000 E - 04
Force per unit length			Btu/min	kW	1.758 426 E - 02	ft ² /s	m ² /s	9.290 304 E - 02
lbf/ft	N/m	1.459 390 E + 01	Btu/h	W	2.928 751 E - 01	in. ² /s	mm ² /s	6.451 600 E + 02
lbf/in.	N/m	1.751 268 E + 02	erg/s	W	1.000 000 E - 07	Volume		
Fracture toughness			ft · lbf/s	W	1.355 818 E + 00	in. ³	m ³	1.638 706 E - 05
ksi√in.	MPa√m	1.098 800 E + 00	ft · lbf/min	W	2.259 697 E - 02	ft ³	m ³	2.831 685 E - 02
Heat content			ft · lbf/h	W	3.766 161 E - 04	fluid oz	m ³	2.957 353 E - 05
Btu/lb	kJ/kg	2.326 000 E + 00	hp (550 ft · lbf/s)	kW	7.456 999 E - 01	gal (U.S. liquid)	m ³	3.785 412 E - 03
cal/g	kJ/kg	4.186 800 E + 00	hp (electric)	kW	7.460 000 E - 01	Volume per unit time		
Power density			Power density			ft ³ /min	m ³ /s	4.719 474 E - 04
W/in. ²	W/m ²	1.550 003 E + 03	W/in. ²	W/m ²	1.550 003 E + 03	ft ³ /s	m ³ /s	2.831 685 E - 02
						in. ³ /min	m ³ /s	2.731 177 E - 07

(a) kg × 10³ = 1 metric ton or 1 megagram (Mg)

SI prefixes—names and symbols

Exponential expression	Multiplication factor	Prefix	Symbol
10^{24}	1 000 000 000 000 000 000 000 000	yotta	Y
10^{21}	1 000 000 000 000 000 000 000	zetta	Z
10^{18}	1 000 000 000 000 000 000	exa	E
10^{15}	1 000 000 000 000 000	peta	P
10^{12}	1 000 000 000 000	tera	T
10^9	1 000 000 000	giga	G
10^6	1 000 000	mega	M
10^3	1 000	kilo	k
10^2	100	hecto(a)	h
10^1	10	deka(a)	da
10^0	1	BASE UNIT	
10^{-1}	0.1	deci(a)	d
10^{-2}	0.01	centi(a)	c
10^{-3}	0.001	milli	m
10^{-6}	0.000 001	micro	μ
10^{-9}	0.000 000 001	nano	n
10^{-12}	0.000 000 000 001	pico	p
10^{-15}	0.000 000 000 000 001	femto	f
10^{-18}	0.000 000 000 000 000 001	atto	a
10^{-21}	0.000 000 000 000 000 000 001	zepto	z
10^{-24}	0.000 000 000 000 000 000 000 001	yocto	y

(a) Nonpreferred. Prefixes should be selected in steps of 10^3 so that the resultant number before the prefix is between 0.1 and 1000. These prefixes should not be used for units of linear measurement, but may be used for higher order units. For example, the linear measurement, decimeter, is nonpreferred, but square decimeter is acceptable.

Index

A

Abbreviations and symbols 324–328
Abradable coatings 52, 178–179, 179(F)(T), 182–183, 182(F)
Abrasive cutting. *See* Cutoff machines; Sectioning.
Abrasive grinding. *See* Grinding.
Abrasive grit blasting
 blasting abrasives 113–115, 114(F)
 dry 110–111
 equipment 112–113, 112(F), 113(T)
 mechanical bonding 111(F)
 surface finish 112(F)
 variables 111–112
 wet 110
Abrasive wear
 applications 172(T), 173
 overview 15
Acoustic emission testing 271–272, 271(F)
Adhesion testing. *See* Tensile adhesion testing.
Adhesive curing 264
Adhesive wear
 applications 171, 172(T)
 overview 14–15
Aerospace applications 8, 8(F), 9(F), 176–181, 180(F), 211–212, 212(F)
Agglomeration 140, 156–157, 159(F), 160(F), 161(F)
Agglutination 157
Air classification 162, 163(F)
Air plasma spraying. *See also* Plasma arc spraying.
 compared with other processes 6(T), 54(T)
 microstructures 3(F), 69(F), 70(F)
 overview 5, 176
 particle cuts 163(T)
Aluminum and aluminum alloy coatings
 applications 193–194, 195–196, 195(T), 196(T), 197(F), 198(F)
 cold spray 80(T)
 metallographic preparation 251, 251(F), 255(F)
 microstructures 63(F), 251(F), 255(F)
American Congress on Government and Industrial Hygiene 100
American Gas Association 99
American Welding Society 36–37, 39, 39(T)
Anodizing 23–24
Applications
 aerospace 8, 8(F), 9(F), 176–181, 180(F), 211–212, 212(F)
 automotive 184–189, 185(F), 186(F)(T), 187(F), 188(F), 189(F),
 200, 211
 brazing/joining 211
 ceramic manufacturing 191–192
 chemical process industries 203–204
 and coating classification 146
 cold spray 80–81, 211–212, 211(F), 212(F)
 corrosion resistance 52, 173–174, 174(T), 184–185, 185(F), 192–198,
 193(F)(T), 194(F), 195(T), 196(T), 197(F), 198(F), 202–203
 diesel engines 185–186
 electrical utilities 207–208, 207(F), 208(F)
 electronic materials and devices 198–200, 199(F), 200(F)
 elevated-temperature 174(T), 177–178
 forming process 210–211
 functionally gradient materials 7–8, 8(F)
 glass manufacturing 191–192
 high-performance 211, 211(F)
 infrastructure 192–198, 194(F), 195(T), 196(T), 197(F)
 ion implantation 30(T)
 metal coatings 7
 metals processing industries 206–207, 207(F)
 microwave 200, 200(F)
 overview 171, 175

 oxidation resistance 182
 petrochemical 174(T)
 plastics industry 208
 printing industry 200–201
 processing industries 201–206
 pulp and paper industry 204–206, 205(F)
 recent developments 208–212
 seals 176(F), 178–179, 179(F)(T), 182–183, 182(F)
 textile industry 208
 timeline 8(F)
 tooling 210–211
 turbine engines 176–179, 177(F), 181–184, 181(F), 182(F)
 wear resistance 52, 171–173, 172(T), 181–182, 182(F)
APS. *See* Air plasma spraying.
Aqueous corrosion 16–17
Arc metallization 142–143
Asbestos, replacement by TBCs 192
ASM International 36, 39, 39(T), 99
ASTM International 37–39, 37(T), 39(T)
Atmospheric plasma spraying. *See* Air plasma spraying.
Atomic absorption spectrometry 276
Atomic emission spectroscopy 276
Atomization 150, 151(F). *See also* Gas atomization; Water
 atomization.
Automotive applications .. 184–189, 185(F), 186(F)(T), 187(F), 188(F),
 189(F), 200, 211

B

Baking 110
Bearing coatings 171, 172(T)
Bend testing 131
Beryllia 199, 199(F)
Binary-image processing 258
Biocompatibility 18
Biomedical coatings 53, 189–191, 190(F), 191(F), 275
Blasting. *See* Abrasive grit blasting.
Boilers, coal-fired 207–208, 208(F)
Bond coating 115–116, 116(T)
Bond strength 51–52, 51(F)
Bonding 219–220, 222
Boriding 22
Bragg diffraction 276(F), 277
Brazing/joining applications 211
Bridges. *See* Infrastructure applications.
Brush plating 23
Buildup (repair) coatings ... 121–123, 121(F), 122(F), 123(F), 184, 203

C

Cameras, for image analysis 254
Carbide coatings. *See* Cermet coatings.
Carbonitriding 22, 23(T), 24(F)
Carburizing
 gaseous techniques 21–22
 overview 20–22, 23(T), 24(F)
 pack 20
 salt bath 20–21
Casting, vs. thermal spraying 217–221
Cathodic protection 17, 17(F), 192–197, 194(F), 195(T), 196(T), 197(F)
Cavitation
 applications 172(T), 173
 overview 16
Ceramic coatings. *See also* Hydroxyapatite; Thermal barrier coatings;
 Yttria-stabilized zirconia (YSZ).

applications 198–199
deposition properties 167, 167(T)
overview 7, 145
properties 183(T)
sol-gel powder production 147(T), 153–155, 156(F)
Ceramic manufacturing applications 191–192
Cermets coatings
 applications 176, 176(F), 181–182, 182(F)
 blended 157–158
 cold spray 80(T)
 metallographic preparation 224–226, 224(F), 225(F), 226(F),
 244(F), 245(F), 246(F), 247(F), 248–251,
 251(F), 252(F), 253(F)
 microstructures 57(F), 59(F), 231(F), 244(F), 245(F), 246(F),
 247(F), 251(F), 252(F), 253(F)
 overview 7, 52, 145–146
 x-ray diffraction scans 277(F)
Characterization. *See* Testing and characterization.
Chemical powder preparation
 agglomeration 156–157
 freeze drying 147(T), 156
 sintering 156–157, 156(F)
 sol-gel 147(T), 153–155, 156(F)
 spray drying/roasting 147(T), 156
Chemical processing applications 203–204
Chemical treatments 24
Chemical vapor deposition
 fluidized-bed 26, 26(T), 27(F), 32, 33, 35, 35(T)
 laser-assisted 26, 26(T)
 microstructure 27(F)
 overview 25–26, 26(T), 27(F)
 plasma-assisted 25–26, 26(T)
Cladding
 chemical 157
 mechanical 157
Cleaning 110
Coating buildup. *See* Buildup (repair) coatings.
Coating materials. *See also* Particles; Powder feeders/feeding; Powders;
 various coating types.
 advances in 12–13, 13(F)
 applications 146
 classification 142–146, 142(F)
 overview 7–8, 52–53, 135–136, 137
 testing and characterization 217–278
Cobalt and cobalt alloy coatings 177, 177(F), 190–191
Cold spraying
 applications 80–82, 211–212, 211(F), 212(F)
 coating materials 79–80, 80(F)(T), 81(F)
 coating microstructures 77(F), 80(F), 81(F), 82(F), 83(F)
 coating properties 78–79, 79(F), 80(T)
 compared with other processes 77(F)
 copper metallization 77(F), 80(F), 81
 deposition efficiency 78–79, 79(F)
 equipment 78, 78(F)
 galvanizing, selective 81–82, 82(F)
 heat sinks 211(F)
 overview 7, 77–78
 process parameters 78, 78(T)
 rapid tooling repair 82, 83(F)
Combustion spraying. *See* Flame spraying.
Comminution 147, 147(T), 149(F). *See also* Crushing;
 Machining; Milling.
Composite coatings
 cold spray 80(T), 81(F)
 microstructures 81(F)
 overview 7, 144(F), 145, 146
Computed microtomography 272
Computers, for image analysis 254
Conferences 38(T), 39
Controlled-environment spraying 70–72
Conventional plasma spraying. *See* Air plasma spraying.
Copper and copper alloy coatings
 cold spray 77(F), 80(F)(T), 81, 82(T)
 microstructures 77(F), 80(F), 238(F)
Corrosion
 ASTM test standards 37(T)

galvanizing protection 81–82, 82(F)
hot 184–185, 185(F)
overview 16–18, 16(F)
resistance 52, 173–174, 174(T), 182, 184–185, 185(F), 192–198,
 193(F)(T), 194(F), 195(T), 196(T), 197(F),
 198(F), 202–203
 testing 266–268, 266(F), 267(F), 268(F), 269(F)
Costs 106, 140
Coulter effect 273
Critical orifices 92, 92(F)
Crushing 147–148
Cutoff machines 226–227, 226(F), 227(T), 228(F)
CVD. *See* Chemical vapor deposition.
Cyclones 162, 163(F)

D

D-Gun. *See* Detonation gun spraying.
Debris, minimizing 121–122, 122(F)
Densification 129–130
Density, measurements 164–166, 166(T)
Dental implants 189–191
Deposition efficiency 78–79, 79(F), 126
Design, statistical. *See* Statistical design.
Destructive testing 131
Detonation gun spraying
 coating characteristics 56
 compared with other processes 6(T), 54(T), 56
 equipment 55–56
 microstructures 57(F)
 overview 5, 175
 process characteristics 55–56
 system 57(F)
Diamond Jet, particle cuts 163(T)
Diamond pyramid hardness testing 260, 260(F)
Dielectrics 198–199
Diesel engine applications 185–186
Dimensional restoration 53, 116–117
Dry corrosion 17, 17(F)
Dwell time 45–46, 45(F)

E

Electric arc spraying
 applications 189, 189(F)
 coating characteristics 61–62
 compared with other processes 6(T), 54(T)
 equipment 60–61, 62(F), 63(F)
 microstructures 63(F), 64(F)
 overview 5, 44(F), 176
 process characteristics 60, 60(F)
 spray rates 60(T)
Electrical isolation/conduction 18, 52–53
Electrical utility applications 207–208, 207(F), 208(F)
Electrochemical treatments 22–24
Electroless plating 24, 25(T), 32, 33, 35, 35(T)
Electron beam heating 19–20, 21(T)
Electronic applications 198–200, 199(F), 200(F), 275
Electroplating 22–23, 24(F)(T), 32, 33, 35, 35(T)
Elevated-temperature applications 174(T), 177–178, 206
Engines. *See* Automotive applications; Diesel engine applications;
 Turbine engine applications.
Environmental Protection Agency (EPA) 196, 210
Equipment
 abrasive grit blasting 112–113, 112(F), 113(T)
 atomization 150, 151(F), 152(F)
 blenders 166(F)
 cold spraying 78, 78(F)
 costs 106
 cyclones 162, 163(F)
 detonation gun spraying 55–56
 electric arc spraying 60–61, 62(F), 63(F)
 flame spraying 55
 glass-lined 204
 grinding 233–236
 Hall flow apparatus 166(F)

334 / Handbook of Thermal Spray Technology

Equipment (continued)

health and safety	99–102
high-velocity oxyfuel spraying	58
milling	148–150, 149(F), 150(F)
motion control	96
mounting	231(F)
plasma arc spraying	64–69, 66(F), 67(F), 68(F), 72(F), 73–76, 74(F), 75(F)
preheating	117
process control	85–98, 86(F), 87(F), 88(F), 91(F), 92(F), 96(F)
pulp and paper	204–206, 205(F)
sampling thief	164(F)
sectioning	226–227, 226(F), 228(F)
sieves	161–162, 162(F), 273
vacuum plasma spraying	71(F)
Erosion	172(T), 173, 185
Etchants	251(F), 259, 259(F)
Evaporation	27–28, 27(F), 28(F), 29(T)
Eye protection	101, 101(T)

F

Fatigue wear	16
Feedstock materials. <i>See</i> Coating materials; Powder feeders/feeding; Powders.	
Ferrite coatings	197–198
FGM. <i>See</i> Functionally gradient materials.	
Finishing	128–129
Flame hardening	18
Flame powder spraying	
compared with other processes	6(T)
equipment	55
overview	5
system	55(F)
Flame spraying. <i>See also</i> Detonation gun spraying; Flame powder spraying; High-velocity oxyfuel spraying; Wire flame spraying.	
coating characteristics	55
combustion-powder/rod/wire	175
equipment	55
microstructures	56(F)
overview	5, 43, 44(F), 54–55, 54(T)
particle cuts	163(T)
process characteristics	54–55
Flame wire spraying. <i>See</i> Wire flame spraying.	
Flowmeters. <i>See</i> Critical orifices; Mass flowmeters; Rotameters.	
Forging, vs. thermal spraying	217–221, 219(F)
Forming process applications	210–211
Freeze drying	147(T), 156
Fretting	172(T), 173, 176–177
Functionally gradient materials	7–8, 8(F)
Fused coatings	129, 129(T)

G

Galling	186
Galvanic series	193(T)
Galvanizing	81–82, 82(F), 192–193
Gas	
flow measurement and control	91–93, 91(F), 92(F)
handling	99
quality	93–94
Gas atomization	
advantages	153
gas/metal interactions	151, 153(F)
operating conditions	152–153
operating variables	151, 154(F)
particle size distributions	152, 155(F)
powder characteristics	151–152
powder morphology	153, 155(F), 159(F), 160(F)
schematic	152(F)
Gator-Gard process. <i>See</i> Plasma arc spraying.	
General information sources, guide to	36–40
Generators, magnetohydrodynamic	207, 207(F)
Glass contact coatings	191–192
Glass manufacturing applications	191–192

Glossary of terms	281–323
Gouging abrasion	15
Grain size	218, 218(F)(T), 219(F)
Gray-image processing	255, 256–258, 256(F), 257(F), 258(F), 259(F)

Grinding

deformation	237, 238(F), 239(F), 240(T)
intermediate	247
materials	233–236, 233(F)(T), 234(F), 235(F), 236(F)
overview	128–129, 231, 233
parameters	237–239, 240(F), 241(F), 242(T)
planar	247
rates	236–237
Grit blasting. <i>See</i> Abrasive grit blasting.	

H

Hall flow	164–166, 166(F)(T)
Hardfacing	
flux-cored arc welding	31, 34(F)
metal inert gas welding	31, 34(F)
overview	30–32, 32(T)
oxyacetylene welding	30–31, 33(F)
plasma-transferred arc welding	31–32, 34(F)
shielded metal arc welding	31, 33(F)
submerged arc welding	31, 34(F)
tungsten inert gas welding	31, 33(F)
Hardness, of electroplated materials	24(T)
Hardness testing	260–261, 260(F)
Health and safety	
equipment	99–102
guidelines	36, 99
motion control	96
powder-related	141
Heat-affected zones	204
Heat exhaust	99–100
Heat sinks, cold sprayed	211(F)
Heat treating	130
High-energy plasma spraying	
compared with other processes	6(T)
microstructures	225(F)
High-frequency resistance heating	18–19, 20(F)
High-performance applications	211, 211(F)
High-temperature applications. <i>See</i> Elevated-temperature applications.	
High-velocity oxyfuel spraying	
applications	179–181, 180(F), 182(F), 203
coating characteristics	58–60
compared with other processes	6(T), 54(T)
components	58
equipment	58
gun features	57(F), 59(F)
microstructures	4(F), 59(F), 226(F)
motion control variables	125
overview	5, 175
process characteristics	56–58
Histograms	255–258, 256(F), 257(F), 258(F), 259(F)
Historical developments	9–13, 13(F)
Hollow-oxide spherical powders. <i>See</i> HOSP process.	
Hoses, and powder pulsing	90–91
HOSP process	157, 158(F)
Hot dip coating	24–25, 25(T)
Hot isostatic pressing	130
HVOF. <i>See</i> High-velocity oxyfuel spraying.	
Hydrogen embrittlement	17
Hydroxyapatite	191, 191(F)

I

Image analysis	
binary-image processing	258, 274, 274(F)
cameras	254
computers	254
gray-image processing	255, 256–258, 256(F), 257(F), 258(F), 259(F)
microscopes	253

overview 252–253, 274–275
shade correction 254–255
thresholding 255–258, 256(F), 257(F), 258(F), 259(F), 274
vs. reflected light microscopy 253–254
Impact wear 15–16, 16(F)
Inclusions
 minimizing 48
 overview 47(F), 48, 48(F), 49(F)
Induction hardening 18, 20(F)(T)
Inert chamber plasma spraying 71–72, 176
Information management 127
Information sources, guide to 36–40
Infrasound 101–102
Infrastructure applications 192–198, 194(F), 195(T), 196(T), 197(F)
Inspection 130–131
Interfacial chemistry 220–221, 222
Interfacial strength 218–219
Intermetallic coatings 7, 145
Internet resources 39(T)
Ion implantation 29, 30(T)
Ion plating 29, 29(F)
Iron coatings
 cold spray 80(T), 82, 83(F)
 microstructures 83(F)

J

Jet Kote, particle cuts 163(T)

K

Kinetic energy processes 7

L

Laser glazing 130, 203
Laser heating 19, 21(F)(T)
Laser light scattering 275
Laser-ultrasonics testing 272
Liquid metal embrittlement 17–18
Lissagous pattern 267(F)
Low-pressure plasma spraying. *See* Vacuum plasma spraying.

M

Machining. *See also* Turning.
 for powder production 147
 for surface macroroughening 115, 115(F)
Magnetic fields 101
Markets 8–9
Masking 117, 117(F), 118(F)
Mass flowmeters 92–93, 92(F)
Materials. *See* Coating materials.
Medical coatings. *See* Biomedical coatings.
Metal coatings. *See also* Refractory metal coatings; various metal
 coating types.
 applications 7, 177, 184
 cold spray 80(T)
 microstructures 70(F), 254(F)
 overview 7, 145
Metallic carbide coatings. *See* Cermet coatings.
Metallization 199–200
Metallography
 basic procedure 251–252
 cleaning 247
 etchants 251(F), 259, 259(F)
 grinding 231, 233–239, 233(F)(T), 234(F), 235(F), 236(F), 238(F),
 239(F), 240(F)(T), 241(F), 242(T), 247
 mounting 227–231, 228(T), 229(T), 230(F), 231(F), 232(T), 247
 overview 131, 224–226
 polishing 239–245, 242(F)(T), 243(F), 244(F), 245(F), 246(F),
 247–251, 247(F), 248(F), 249(F), 250(F), 251(F)
 sectioning 226–227, 226(F), 227(T), 228(F), 246
Metals processing applications 206–207, 207(F)

Metric conversion guide 329–331
Microscopes, for image analysis 253
Microwave applications 200, 200(F)
Milling 148–150, 149(F), 150(F)
Molybdenum and molybdenum alloy coatings
 applications 192
 cold spray 80(T)
Motion control
 equipment 96
 torch/substrate 124–126, 125(F)
 variables 124–126
Mounting
 abrasion rates 228(T), 229(T)
 cold mounting 229–230, 231(F)
 defects 230–231
 hot mounting 229, 231(F), 247
 materials 228–229, 228(T), 229(T), 230, 232(T)
 preparation 229
 presses 231(F)
 recommended practice 247

N

NACE International 36, 39, 39(T)
National Fire Protection Agency 99
National Institute of Occupational Safety and Health 100
Nickel and nickel alloy coatings
 cold spray 80(T)
 microstructures 4(F), 48(F), 56(F), 59(F), 63(F), 69(F), 159(F),
 160(F), 219(F), 263(F)
Nitriding 22, 23(T)
Nitrocarburizing 22, 24(F)
Noble coatings, vs. sacrificial coatings 192–194, 193(F)(T)
Noise levels 100–101, 100(T)
Nondestructive testing 131, 271–272
Nonsacrificial coatings 197, 198(F)
Nyquist plots 267(F), 268(F), 269(F)

O

Occupational Safety and Health Administration (OSHA) 100
Orifices, critical. *See* Critical orifices.
Orthopedic implants 189–191, 190(F), 191(F)
Oscilloscope measurements 267–268, 267(F), 268(F), 2693
Oxidation resistance 16, 124, 182
Oxide coatings, microstructures 57(F), 59(F), 252(F)
Oxide inclusions. *See* Inclusions.
Oxide stringers. *See* Inclusions.

P

Paints
 lead-base, removal of 210
 polymer coatings as replacement for 209–210, 209(T)
Part configuration. *See* Substrate, geometry.
Partially stabilized zirconia (PSZ) 158(F)
Particles. *See also* Coating materials; Powder feeders/feeding; Powders.
 agglomeration 140, 159(F), 160(F), 161(F)
 characterization 159–168, 273–278
 chemistry 275–276
 classification 161–163, 162(F), 164(F)
 formation 152(F), 153(F)
 morphology 143–145, 144(F), 145(F)(T), 155(F), 159–161,
 159(F), 160(F), 161(F)
 size distribution 144–145, 155(F), 162–163, 164(F), 273–275,
 274(F)
 size measurement 166–167
 testing 273–278
Particulates 100
Peening 130
Petrochemical applications 174(T)
Phase diagrams
 iron-carbon 19(F)
 overview 18
Phosphating 24

336 / Handbook of Thermal Spray Technology

Physical vapor deposition 26–29, 32, 33, 35, 35(T)
Plasma arc spraying. *See also* Air plasma spraying; High-energy plasma spraying; Inert chamber plasma spraying; Vacuum plasma spraying.
 arc starting problems 95–96
 axial powder-feed guns 74–76, 75(F)
 coating characteristics 69–70
 coating formation 220(F)
 controlled-environment 70–72
 enthalpy values 65(F)
 equipment . . . 64–69, 66(F), 67(F), 68(F), 72(F), 73–76, 74(F), 75(F)
 extended-arc guns 73–74, 74(F), 75(F)
 Gator-Gard 72
 microstructures 56(F), 69(F), 70(F), 219(F), 221(F), 224(F), 225(F)
 overview 5–7, 44(F), 45–46
 process characteristics 62–64
 radiofrequency 54(T), 72–73, 73(F)
 shrouded 72, 72(F)
 underwater 73
 water-stabilized 74
Plasma fusion and densification 157, 158(F)
Plasma heating 19
Plastics industry applications 208
Polishing
 abrasives 240–241, 242(T)
 of aluminum specimens 251, 251(F)
 of cermet coating specimens 244(F), 245(F), 246(F), 247(F), 248–251, 251(F)
 cloths 241, 243(F), 244(F)
 lubricants 241–242, 244(F)
 mechanisms 240, 242(F)
 overview 129, 239–240
 rates 242–245, 242(T)
 of soft coating specimens 251, 251(F)
 of YSZ specimens 247–248, 248(F), 249(F), 250(F)
Polymer coatings
 applications 209–210, 209(T)
 microstructures 56(F)
 overview 7, 53, 145
Porosity
 control of 50
 internal 144, 145(F), 161(F)
 overview 4, 47(F), 49, 49(F)
 and particle morphology 159–161, 161(F)
 sources of 49–50, 50(F)
 and spray angle 108, 109(F)
Postcoating operations 128–131, 203
Powder feeders/feeding. *See also* Coating materials; Particles; Powders.
 agglomeration 140
 Archimedes spiral 139, 140(F)
 encapsulated shot 139, 140(F)
 experimental 140
 fluidized-bed 87–88, 88(F), 138, 139(F)
 gravity-based 86–87, 138, 138(F)
 health concerns 141
 heat source interactions 140–141, 140(F), 141(F)
 problems 137–138, 141, 167–168, 168(F)
 rotating wheel 138, 139(F)
 systems compared 140
 variables 140
 vibratory 138–139, 139(F)
 volumetric 87, 87(F)
Powder flame spraying. *See* Flame powder spraying.
Powders. *See also* Coating materials; Particles; Powder feeders/feeding.
 blended 157–158
 characteristics 85, 88–90
 density 89
 feed rate control 90
 feeders 86–88, 87(F), 88(F)
 flowability measurements 90, 90(F)
 hollow-oxide spherical 157, 158(F)
 manufacturing/production 89, 147–158, 147(T), 148(F)
 moisture content 89–90, 91

 morphology 88–89, 89(F), 155(F)
 particle melting 122–123, 123(F)
 particle size and distribution 88, 91, 144–145, 147(F)
 pulsing 87, 90–91
 stoichiometry 275
 testing and characterization 273–278
Precious metals, in glass manufacture 191–192
Preheating, of substrates 117–118
Pressure vessels 203–204
Printing industry applications 200–201
Process control equipment . . . 85–98, 86(F), 87(F), 88(F), 91(F), 92(F), 96(F)
Processing industries applications 201–206
Pulp and paper applications 204–206, 205(F)

Q

Quality control
 and feedstock material specification 164–168, 168(F)
 motion control 96
 specifications and standards 36–39, 37(T)

R

Radiation 101
Radiofrequency plasma spraying
 compared with other processes 54(T)
 overview 72–73, 73(F)
Rapid solidification 275
Refractory metal coatings, applications 176–177
Repair coatings. *See* Buildup (repair) coatings.
Residual stresses 270–271, 271(F)
Rockwell superficial hardness testing 260–261
Rod
 characteristics 85
 for combustion spraying 175
 feeders 86
Rotameters 91–92, 91(F)
Rotaplasma 186–187, 186(F)(T), 187(F)
Roughening 110

S

Sacrificial coatings
 in atmospheric and immersion environments 195–196, 195(T), 196(T), 197(F)
 for infrastructure and industry 194–195, 194(F)
 problems 196–197
 vs. noble coatings 192–194, 193(F)(T)
Safety. *See* Health and safety.
Salvage, casting 206, 207(F)
Sampling
 representative 164, 164(F), 165(F), 166(F)
 thief 164(F)
Scalping 161, 162(F)
Sealants 129–130, 130(T), 202–203
Seals 176(F), 178–179, 179(F)(T), 182–183, 182(F)
Sectioning 226–227, 226(F), 227(T), 246
Sedimentation
 centrifugal 274
 gravitational 273–274
Self-fluxing alloys 129, 129(T)
Self-propagating high-temperature synthesis (SHS) 148
Sieving 161–162, 162(F), 163, 163(T), 273, 273(T)
Silver and silver alloy coatings, cold spray 80(T)
Single-phase materials. *See* Ceramic coatings; Intermetallic coatings; Metal coatings; Polymer coatings.
Sintering 156–157, 156(F), 161(F)
Small-angle neutron scattering 272
Society for Protective Coatings 36, 39, 39(T)
Sol-gel processing 147(T), 153–155, 156(F), 168(F)
Solidification rapid, 275
Specifications and standards 36–39, 37(T), 100, 261

Splats 3, 47–48, 47(F), 220(F)
Spray drying/roasting 147(T), 156
Spray pattern 120–121, 120(F), 121(F), 125, 125(F)
Spray rate 125
Sputtering 28–29, 29(F)
Stainless steel coatings
 cold spray 80(T), 81(F)
 microstructures 81(F)
Standards. *See* Specifications and standards.
Statistical design 126–127, 126(T)
Steel coatings. *See also* Stainless steel coatings.
 cold spray 80(T)
 microstructures 48(F), 64(F)
Steel mill applications 206–207
Stoichiometry 275
Storage tanks 203–204
Stress-enhanced corrosion 17
Stringers. *See* Inclusions.
Substrate
 degradation 124
 geometry 108–110, 109(F), 121, 122(F)
 masking 117, 117(F), 118(F)
 metallurgy 108
 motion 124–126
 preheating 117–118
 preparation 110–117
 shrinkage/expansion 124
 temperature control 124, 125(F)
Superalloy coatings, cold spray 80(T)
Surface fatigue wear, applications 172(T), 173
Surface hardening 18–20
Surface preparation
 abrasive grit blasting 110–115, 111(F), 112(F), 113(T), 114(F)
 bond coating 115–116, 116(T)
 cleaning 110
 machining and macroroughening 115, 115(F)
 roughening 110
 tensile adhesion testing 261–262
 undercutting 116–117
Surface science 14–35
Symbols and abbreviations 324–328

T

Taguchi matrix 126(T)
TBC. *See* Thermal barrier coatings.
Temperature control, of substrate/coating 124, 125(F)
Tensile adhesion testing 131, 261–266, 262(F), 263(F)(T), 265(F)
Tensile strength 4
Terms, glossary of 281–323
Testing and characterization. *See also* Image analysis; Metallography.
 acoustic emission 271–272, 271(F)
 atomic absorption spectrometry 276
 atomic emission spectroscopy 276
 of coatings 217–223, 260–272
 computed microtomography 272
 corrosion 266–268, 266(F), 267(F), 268(F), 269(F)
 Coulter effect 273
 destructive 131
 electrical resistance 273
 Hall flow 164–166, 166(F)(T)
 hardness 260–261, 260(F)
 laser light scattering 275
 laser-ultrasonics 272
 as materials engineering opportunity 222
 nondestructive 131, 271–272
 overview 217–223, 260
 of particles 159–168
 of powders 273–278
 residual stress 270–271, 271(F)
 as response generators 221–222
 sedimentation 273–274
 small-angle neutron scattering 272
 tensile adhesion 261–266, 262(F), 263(F)(T), 265(F)

 of thermal barrier coatings 268–270, 270(F)
 wet chemical methods 276
 wipe test 167
 x-ray diffraction 276–278, 276(F), 277(F)
 x-ray fluorescence 276
Textile industry applications 208
Thermal barrier coatings
 applications 177–178, 178(F), 183–184, 183(T), 184, 188–189, 188(F), 189(F), 192, 206
 for asbestos replacement 192
 dry corrosion 17(F)
 environmental stability 270
 and FGMS 7, 8
 overview 52
 testing 268–270, 270(F)
 thermomechanical stability 268–270, 270(F)
Thermal mass flowmeters. *See* Mass flowmeters.
Thermal shock testing 131
Thermal Spray Society 36, 39(T), 99
Thermal spray vitrification process 210
Thermal spraying
 advantages 3, 105–106
 applications 8–9, 8(F), 9(F), 10(T), 11(T), 12(T), 43, 80–81, 105–106, 171–213
 coating characteristics 3–4, 11(T), 12(T), 47–51
 coating formation 217–221
 coating materials 7–8, 52–53, 137–146
 coating operations 120–127
 coating processing 108–119
 coating properties 51–52
 coating testing 260–272
 conferences 38(T), 39
 controls 94–95
 disadvantages 106–107
 future growth 9
 general information sources 36–40
 glossary of terms 281–323
 historical developments 9–13, 13(F)
 image analysis 252–259
 metallography 224–252
 overview 3–13, 29–30, 31(F), 32, 32(F), 33, 35, 35(T), 43–46, 43(F), 44(F), 45(F), 54
 particle characterization 159–168, 273–278
 postcoating operations 128–131
 powder production 147–158
 powder testing and characterization 273–278
 process characteristics 45, 45(F)
 process classification 44–45, 44(F), 175–176
 process comparison 6(T), 54(T)
 process control equipment 85–98, 86(F), 87(F), 88(F), 91(F), 92(F), 96(F)
 process parameters 85–86, 120–127
 process variations 123–124, 124(T)
 processes and techniques 5–7, 6(T), 10(T), 54–76, 54(T), 77–84
 schematic 4(F)
 specifications and standards 36–39, 37(T)
 surface science 14–35
 testing and characterization 217–278
 vs. forging and casting 217–221
 vs. other coating processes 44
Thermochemical treatments 20–22
Thresholding 255–258, 256(F), 257(F), 258(F), 259(F)
Titanium and titanium alloy coatings
 applications 190–191, 190(F), 191(F)
 cold spray 79(F), 80(T), 81(F)
 microstructures 64(F), 81(F)
Tooling applications 210–211
Top-Gun, particle cuts 163(T)
Tri-calcium phosphate 191
TSVP. *See* Thermal spray vitrification process.
Tungsten and tungsten alloy coatings, microstructures 3(F), 259(F)
Turbine engine applications 176–179, 177(F), 181–184, 181(F), 182(F)
Turning 128, 128(F). *See also* Machining.

338 / Handbook of Thermal Spray Technology

U

Ultrasonic cleaning 110
Undercutting 116–117

V

Vacuum plasma spraying
 compared with other processes 6(T), 54(T)
 equipment 71(F)
 microstructures 70(F), 219(F)
 overview 5, 7, 70–71, 143, 176
 for process industry applications 203
Vapor degreasing 110
Ventilation 99–100
Vickers hardness testing 260, 260(F)
Volatilization, preferential 275
VPS. *See* Vacuum plasma spraying.

W

Water atomization
 operating conditions 152–153
 particle formation 150–151, 152(F)
 particle size distributions 152, 155(F)
 powder morphology 153, 155(F), 159(F), 160(F)
 schematic 152(F)
Water cooling 97
Wear. *See also* Abrasive wear; Adhesive wear; Surface fatigue wear.
 ASTM test standards 37(T)
 of carbon steel 24(F)
 overview 14–16, 15(F), 16(F)
 paper equipment 205(F)
 resistance 52, 171–173, 172(T), 181–182, 182(F), 206
Weld overlay. *See* Hardfacing.
Wipe test 167

Wire

 characteristics 85
 for combustion spraying 175
 feeders 86, 86(F)

Wire arc spraying. *See* Electric arc spraying.

Wire flame spraying

 compared with other processes 6(T)
 overview 5
Wrought materials, vs. thermal spraying 217–221

X

X-ray diffraction 276–278, 276(F), 277(F)
X-ray fluorescence 276

Y

Yttria-stabilized zirconia (YSZ)

 applications 177–178, 178(F), 183–184, 183(T)
 metallographic preparation 247–248, 248(F), 249(F), 250(F),
 251(F)
 microstructures 48(F), 159(F), 160(F), 248(F), 249(F), 250(F),
 251(F)
 morphology 145(T), 159(F), 160(F)
 physical properties 145(T)

Z

Zinc and zinc alloy coatings

 for cathodic protection 17, 17(F), 192–197, 194(F), 195(T),
 196(T), 197(F)
 cold spray 80(T), 81–82, 82(F)
 for galvanizing 81–82, 82(F)
 microstructures 63(F), 82(F)

Zirconia. *See* Partially stabilized zirconia (PSZ); Yttria-stabilized zirconia (YSZ).



ASM International is the society for materials engineers and scientists, a worldwide network dedicated to advancing industry, technology, and applications of metals and materials.

ASM International, Materials Park, Ohio, USA
www.asminternational.org

This publication is copyright © ASM International®. All rights reserved.

Publication title	Product code
Handbook of Thermal Spray Technology	#06994G

To order products from ASM International:

Online Visit www.asminternational.org/bookstore

Telephone 1-800-336-5152 (US) or 1-440-338-5151 (Outside US)

Fax 1-440-338-4634

Mail Customer Service, ASM International
9639 Kinsman Rd, Materials Park, Ohio 44073-0002, USA

Email CustomerService@asminternational.org

In Europe American Technical Publishers Ltd.
27-29 Knowl Piece, Wilbury Way, Hitchin Hertfordshire SG4 0SX,
United Kingdom
Telephone: 01462 437933 (account holders), 01462 431525 (credit card)
www.ameritech.co.uk

In Japan Neutrino Inc.
Takahashi Bldg., 44-3 Fuda 1-chome, Chofu-Shi, Tokyo 182 Japan
Telephone: 81 (0) 424 84 5550

Terms of Use. This publication is being made available in PDF format as a benefit to members and customers of ASM International. You may download and print a copy of this publication for your personal use only. Other use and distribution is prohibited without the express written permission of ASM International.

No warranties, express or implied, including, without limitation, warranties of merchantability or fitness for a particular purpose, are given in connection with this publication. Although this information is believed to be accurate by ASM, ASM cannot guarantee that favorable results will be obtained from the use of this publication alone. This publication is intended for use by persons having technical skill, at their sole discretion and risk. Since the conditions of product or material use are outside of ASM's control, ASM assumes no liability or obligation in connection with any use of this information. As with any material, evaluation of the material under end-use conditions prior to specification is essential. Therefore, specific testing under actual conditions is recommended.

Nothing contained in this publication shall be construed as a grant of any right of manufacture, sale, use, or reproduction, in connection with any method, process, apparatus, product, composition, or system, whether or not covered by letters patent, copyright, or trademark, and nothing contained in this publication shall be construed as a defense against any alleged infringement of letters patent, copyright, or trademark, or as a defense against liability for such infringement.