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Measurement Methods and Standards for Processing and Application of Thermal Barrier Coatings

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Application of thermal barrier coatings deposited by thermal spray, physical vapor and possibly other methods is expected to be extended from aircraft gas turbines to industrial and utility gas turbines as well as diesel engines. This increased usage implies the participation of greater numbers of processors and users, making the availability of standards for process control and property measurement more important. In this paper, available standards specifically for processing and evaluation of thermal barrier coatings are identified as well as those generally applicable to coatings and those needed but unavailable.

Keywords measurement methods, standards, thermal barrier coatings

1. Introduction*

THERMAL BARRIER COATINGS (TBCs) based on stabilized zirconia have been applied to aircraft gas turbines for several years, with early application on combustor components extended to airfoils. The increased system thermodynamic efficiency due to higher operating temperatures allowed by these coatings, as well as extended substrate lifetimes due to lower metal temperatures, has warranted consideration for non-aircraft turbine as well as diesel engine applications. The increased commerce in coating services and the number of coated parts customers expected for these new applications implies a greater need for standardized methods of describing and controlling processing conditions and measurement of coating properties.

Commerce between material or coating suppliers and component manufacturers is conducted by reference to specifications. Specifications are instituted to address two primary concerns: (a) cost, which is driven by process reproducibility (manifested as scrap) or reject rate and materials utilization (manifested as waste); and (b) performance, which is driven by predictable properties and optimized design. Standard test methods (such as for mechanical properties) and analytical techniques (such as for chemical or microstructural analysis) are, or can be, used for determining compliance to specifications. In applications where coatings are an integral design element of structural components, increased precision in control of the coating deposition process and more accurate measurement of coating properties are desirable, raising further the importance of standard test and analysis methods.

Standard test and analysis methods often rely on, or are based on, reference materials with well-characterized properties that are relevant to specific measurements. It is also important that standard test methods and standard reference materials be appropriate to the accuracy, speed requirements, instrumentation, and skill capabilities of the intended users. Therefore, development of standards and reference materials is best conducted through the consensus and participation of interested users. It is important that standards or reference materials be developed with a sufficient understanding of the phenomenon or property of interest to ensure that the limits of applicability be defined.

The value of standardized measurement methods for coatings has been recognized in the United States by the Department of Defense (DOD), primarily for plasma-sprayed or detonation gun coatings. In addition to military specifications, Society of Automotive Engineers (SAE) and American Society for Testing and Materials (ASTM) consensus standards have been developed, primarily for wear-resistant plasma-sprayed coatings. Standards developed in Great Britain (British Standards Institute, BSI), Germany (Deutsches Institut fur Normung, DIN), and Japan (Japanese Institute of Standards, JIS) primarily address thin coatings not intended for thermal barrier application. They largely focus on measurement of physical characteristics such as thickness and qualitative measures of adhesion. The increased interest in coatings of all types has fostered a Committee for European Normalization (CEN) effort to develop standard measurement methods for coatings: Working Group 5, Ceramic Coatings, under Technical Committee 184, Advanced Technical Ceramics. It may be expected that these national and regional standards will be incorporated into International Standards Organization standards eventually.

In the long term, the use of materials designed to take advantage of intrinsic microstructural refinements, such as functionally graded and nanolayered coatings, will require the development of characterization and measurement standards that are now experimental techniques.

2. Relevant Measurements

The efficacy of TBCs depends on two major factors: adhesion to metallic substrates and the thermal insulation provided by the ceramic. The primary means of TBC deposition, plasma spray and physical vapor deposition (PVD), create anisotropic

*Identification of trade names and products in this paper does not imply that the products are necessarily the best for the purpose or that they are recommended by the National Insitute of Standards and Technology (NIST).

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materials that have a low elastic modulus due to the presence of widely distributed microcracks or porosity. This low modulus allows compliance with the substrate during dimensional changes caused by mechanical or thermal loading. Long-term operability of TBCs depends on adherence of the stabilized zirconia topcoat to a metallic undercoat (bond coat) applied to the structural component. The importance of adhesion has encouraged researchers to identify the causes of spallation and to develop models using thermal, mechanical, and chemical properties to predict coating behavior. Studies (Ref 1-3) have identified or utilized several key properties that influence coating lifetime defined by spallation resistance: coefficient of thermal expansion, thermal conductivity, tensile strength, creep, elastic modulus, Poisson's ratio, and the growth and nature of the oxide layer on the undercoat at the top coat (zirconia) interface. In these studies, properties were generally measured on freestanding materials removed from substrates but processed to provide microstructures similar to those in relevant coatings.

Processing parameter measurement and control is critical to reproducible, cost-effective coating deposition. Typical process parameters controlled for both plasma spray and PVD include power supplied, feedstock chemistry and homogeneity, spacial relationship of feedstock source to substrate, feed rate of coating material, substrate temperature and, for PVD, relevant specie partial pressure in the deposition chamber. Control of these parameters influences deposition rate and efficiency as well as microstructural features such as porosity, microcracking, dendrite or splat size, chemistry and phase content, and thermal conductivity.

3. Standard Measurement Techniques

3.1 *Processing*

Procedures for the deposition of coatings are generally developed by coating purchasers in cooperation with coating processors and constitute valuable proprietary knowledge. Although not standards in themselves, these procedures can utilize standard analysis and measurement methods to ensure that both parties agree on the values of particular specified process parameters. An example might be a standard powder size distribution measurement method to ensure that commercial specifications for limits are not exceeded. The American Welding Society (AWS) and the American National Standards Institute (ANSI) have developed the "Guide for Thermal-Spray Operator Qualification," ANSI/AWS C2.16-92, which sets forth recommended thermal spray operator qualification procedures. It covers applicable documents relating to thermal spray equipment, consumables, and safety.

Standards for processing that focus on the characterization and measurement of properties appropriate for plasma spray feedstock have been developed through the ASTM and SAE. Standards and specifications from AWS, SAE, and DOD are listed in Table 1. An expanded description of the ASTM standards that can provide guidance for TBC processing and property measurement is provided in Table 2. ASTM standards that provide a useful basis for analysis of plasma spray feedstock, in particular, have been developed by the metals, ceramics, and petroleum industries and comprise the majority of standards listed in Table 2. Many of these standards address metals and may be applicable to plasma-sprayed bond coats. Standard procedures for chemical and phase content analysis for stabilized zirconia are lacking. NIST makes available standard reference materials (SRMs) used to calibrate instruments that measure feedstock powder size and size distribution. These are intended for use in sieving and in measuring light and electrical pore flow through counters, optical and electron microscopes, and sedigraphs. SRM 1982, yttria-stabilized, zirconia, is specifically intended for calibration of light scattering measurement of TBC feedstock particle size distribution. Table 3 identifies several SRMs suitable for the measurement of powder size distribution.

Standard procedures for the analysis of thermal spray, PVD, or chemical vapor deposition (CVD) process parameters are unavailable. However, commercial systems have been developed for monitoring of several thermal spray process parameters, such as particle velocity and spatial distribution (Ref 4, 5). These systems could conceivably be related to standard calibration procedures. Techniques for analysis of vapor composition in PVD and CVD processes could also be calibrated to known gaseous compositions via reference materials.

Table 1 Available standards for coatings

Processing	
American National Standards Institute/American Welding Society 550 N.W. LeJeune Rd. P.O. Box 351040 Miami, FL 33135 Tel: (305) 443-9353	
C 2.16-92	Guide for Thermal Spray Operator Qualification
Society of Automotive Engineers (SAE) 400 Commonwealth Dr. Warrendale, PA 15096-0001 Tel: (412) 776-4841 Fax: (412) 776-5760	
AMS 2435C	Detonation Process—Tungsten Carbide/Cobalt Coating
AMS 2436B	Coating, Aluminum Oxide—Detonation Deposition
AMS 2437B	Coating, Plasma Spray Deposition
AMS 5791	Powder, Plasma Spray, 56.5Co-25.5Cr-10.5Ni-7.5W
AMS 5792	Powder, Plasma Spray, 50(88W-12Co) + 35(70Ni-16.5Cr-4Fe-4Si-3.8B) + 15(80Ni-20Al)
AMS 5793	Powder, Plasma Spray (95Ni-5Al)
AMS 7875	Chromium Carbide Plus Nickel-Chromium Alloy Powder, $75Cr_2C_3$ + 25 (80Ni-20Cr Alloy)
AMS 7878	Tungsten Carbide Powder, Cobalt Coated
AMS 7879	Tungsten Carbide-Cobalt Powder, Cast and Crushed
AMS 7880	Tungsten Carbide-Cobalt Powder, Sintered and Crushed

(continued)

Table 1 Available standards for coatings (continued)

Department of Defense

Information Handling Services (IHS)
15 Inverness Way East
P.O. Box 1154
Englewood, CO 80150-1154
Tel: (800) 241-7824; (303) 790-0600
Fax: (303) 397-2717

MIL-C-52023	Coating: Ceramic, Refractory, for High Temperature Protection of Low Carbon Steel—1958
MIL-STD-1886	(AT) Tungsten Carbide-Cobalt Coating, Detonation Process For—1992
MIL-C-81751B	Coating, Metallic Ceramic
MIL-M-80141C	Metallizing Outfits, Powder Guns and Accessories—1987
MIL-STD-1884A	(AT) Coating, Plasma Spray Deposition—1991
MIL-Z-81572	(AS) Zirconium Oxide, Lime Stabilized, Powder and Rod, for Flame Spraying—1991

Properties

British Standards Institute (BSI)

Linford Wood
Milton Keynes MK 146LE
United Kingdom
Tel: 011-44-908-220022
Fax: 011-44-908-320856

BS5411	Methods of Test for Metallic and Related Coatings
Part 1	Definitions and Conventions Concerning the Measurements of Thickness
Part 2	Review of Methods for the Measurement of Thickness
Part 3	Eddy Current Method for Measurement of Thickness of Non-Conductive Coatings on Non-Magnetic Basis Materials
Part 4	Coulometric Method for the Measurement of Coating Thickness
Part 5	Measurement of Local Thickness
Part 6	Vickers and Knoop Microhardness Tests
Part 7	Profilometric Method for Measurements of Coating Thickness
Part 8	Measurement of Coating Thickness of Metallic Coatings: X-Ray Spectrometric Methods
Part 9	Measurement of Coating Thickness of Electrodeposited Nickel Coatings on Magnetic and Non-Magnetic Substrates-Magnetic Method
Part 10	1981/ISO 2819-1980 Review of methods for testing adhesion of electrodeposited and chemically deposited metallic coatings on metallic substrates (burnishing, ball burnishing, shot peening, peel [less than 125 microns], file, grinding and sawing, chisel [greater than 125 microns], scribe and grid, bending, twisting, tensile, thermal shock, drawing, cathodic; qualitative only)
Part 11	Measurement of Coating Thickness of Non-Magnetic Metallic and Vitreous or Porcelain Enamel Coatings on Magnetic Basis Metals: Magnetic Method
Part 12	Beta Backscatter Method for Measurement of Thickness
Part 13	Chromate Conversion Coatings on Zinc and Cadmium
Part 14	Gravimetric Method for Determination of Coating Mass per Unit Area of Conversion Coatings on Metallic Materials
Part 15	Review of Methods of Measurement of Ductility
Part 16	Scanning Electron Microscopy Method for Measurement of Local Thickness of Coatings by Examination of Cross Sections
BSI M. 40	Aerospace Series-Methods for Measuring Coating Thickness by Non-Destructive Testing

Deutsches Institut fur Normung (DIN)

(German Institute for Standardization)
Burggrafenstrasse 6
10787 Berlin
Germany
Tel: (4930) 2601-0
Fax: (4930) 2601-1231

50933-87	Measurement of Coating Thickness by Differential Measurement Using a Stylus
50949-84	Non-Destructive Testing of Anodic Oxidation Coatings on Pure Aluminum and Aluminum Alloys by Measurement of Admittance
50955-83	Measurement of Coating Thickness: Measurement of Thickness of Metallic Coatings by Local Anodic Dissolution: Coulometric Method
50976-89	Corrosion Protection: Hot Dip Batch Galvanizing: Requirements and Testing
50978-85	Testing of Metallic Coatings: Adherence of Hot Dip Zinc Coatings [up to 150 μm]
50982 PT	1-87 Principles of Coating Thickness Measurement: Terminology Associated with Coating Thickness and Measuring Areas
50982 PT	2-87 Principles of Coating Thickness Measurement: Review of Commonly Used Methods of Measurement
50982 PT	3-87 Principles of Coating Thickness Measurement: Selection Criteria and Basic Measurement Procedures
50987-87	Measurement of Coating Thickness by the X-Ray Spectrometric Method
50160-A	Tensile Adhesion

Japanese Institute of Standards (JIS)

1-24 Akaska 4, Minato-ku
Tokyo 107
Japan
Tel: 011-81-3-3588003
Fax: 011-81-3-35862029

H8666-90	Testing Method for Thermal Sprayed Ceramic Coatings
R4204	Method of Testing Ceramic Coating

Table 2 Expanded description of ASTM standards

ASTM designation	Title	Source(a)	Application	Sample description	Measurement	Comments
Processing						
B 212	Standard Test Method for Apparent Density of Free-Flowing Metal Powders	A	Die fill estimate	30-40 cm^3	Apparent density reported to 0.01 g/cm^3	Gravimetric technique using volume of specified cup; intended for powders which flow unaided through Hall flowmeter funnel
B 213	Standard Test Method for Flow Rate of Metal Powders	A	Die fill estimate	50 g	Results reported as flow time in seconds	Measure of time for powder sample to flow through 2.54 mm orifice calibrated using 150-mesh emery powder
B 214	Standard Test Method for Sieve Analysis of Granular Metal Powders	A	Quality control, specifications	100 g for apparent density >1.50 g/cm^3 measured by B 212; 50 g for <1.50 g/cm^3	45-80 μm	Analysis of size distribution using wire cloth sieves with mechanical shaking for 15 min
B 330	Standard Test Method for Average Particle Size of Powders of Refractory Metals and Their Compounds by the Fisher Sub-Sieve Sizer	A	Quality and process control	Sample mass in grams equal to true density of powder	0.2-50 μm	Based on air permeability to determine equivalent spherical particle diameter. Values determined not absolute, useful for purposes of comparison.
B 430	Standard Test Method for Particle Size Distribution of Refractory Metal Type Powders by Turbidity	A	Specifications, processing control	More than 30 g, varies with material, based on light transmission through suspension	1-12 μm	Based on change of intensity of light passing through a dispersion; weight percent of particles in a size range determined based on Stokes law; requires powder density
B 527	Standard Test Method for Determination of Tap Density of Metallic Powders and Compounds	A	Powder processing	25 cm^3	Varies with material	Measures packing of powders in a specified volume; correlation with quality of powders not fully determined
B 761	Standard Test Method for Particle Size Distribution of Refractory Metals and Their Compounds by X-Ray Monitoring of Gravity Sedimentation	A	Specifications, process control	<1 g	Average particle diameter of 0.1-100 μm; for carbides of Mo, Ta, W, 0.1-30 μm for Mo, Ta, W, less than 6 μm	Based on intensity of x-ray beam passing through suspension
B 822	Standard Test Method for Particle Size Distribution of Metal Powders and Related Compounds by Light Scattering	A	Specifications, process control	Liquid dispersion, <25 g; gaseous dispersion, <500 g	0.1-1000 μm	Calculated equivalent spherical particle diameter based on light scattering; reported as volume percent; results not absolute

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Table 2 **Expanded description of ASTM standards (continued)**

ASTM designation	Title	Source(a)	Application	Sample description	Measurement	Comments
C 135	Standard Test Method for True Specific Gravity of Refractory Materials by Water Immersion	F	Specifications, quality control	50 g of sub 150 μm powder	Material-specific	Water displacement method. Material must be nonhydrable or nonreactive with water and may not reflect presence of closed pores. Useful as a secondary standard.
C 690	Standard Test Method for Particle Size Distribution of Alumina or Quartz by Electric Zone Technique	G	Specifications, process control	0.7 g of sample in 200 mL of electrolyte solution	0.6-56.0 μm equivalent spherical diameter	Dilute suspension passed through restricting orifice modulates electric field. Comparable to other techniques for spherical particles.
C 721	Standard Test Method for Average Particle Size of Alumina and Silica Powders by Air Permeability	G	Guide to flow and packing properties, quality control	Sample mass in grams equal to true density of the powder	0.2-50 μm	Intended for alumina and quartz. Measures sample surface area by air permeability converted to average particle diameter. Calibrated with NIST SRM 114, Portland Cement.
C 925	Standard Test Method for Precision Electroformed Wet Sieve Analysis of Nonplastic Ceramic Powders	G	Specifications, process control	<1.0 g	5-45 μm	Intended for alumina and quartz. Vacuum- and vibration-assisted separation of dispersed powder suspension through precision electroformed sieves.
C 958	Standard Test Method for Particle Size Distribution of Alumina or Quartz by X-Ray Monitoring of Gravity Sedimentation	G	Specifications	2.5 g in 30 mL of dispersing liquid	0.5-50 μm with median particle diameter from 2.5-10 μm	Equivalent spherical diameter calculated from Stokes law. Setting measured by change in intensity of x-ray beam passing through aqueous dispersion.
C 1070	Standard Test Method for Determining Particle Size Distribution of Alumina or Quartz by Laser Light Scattering	G	Specifications, process control	Powder in 50 mL of dispersing liquid	1.9-170 μm	Calculated particle diameter based on light scattering in an aqueous dispersion. Based on Microtrac instrument. Average of three 100 s analyses.

(a) Source: 1996 Annual Book of ASTM Standards. A, Section 2—Nonferrous Metal Products, Volume 02.05—Metallic and Inorganic Coatings: Metal Powders, Sintered Plus Structural Parts. B, Section 4—Construction, Volume 04.06—Thermal Insulation; Environmental Acoustics. C, Volume 04.08—Soil and Rock (I): D 420 - D4914. D, Section 5—Petroleum Products, Lubricants and Fossil Fuels, Volume 05.03—Petroleum Products and Lubricants (III): D 4636 - Latest; Catalysts. E, Section 14—General Methods and Instrumentation, Volume 14.02—General Test Methods, Nonmetal: Chromatography; Durability of Nonmetallic Materials; Forensic Sciences; Laboratory Apparatus; Statistical Methods. F, Section 15—General Products, Chemical Specialties, and End Use Products, Volume 15.01—Refractories: Carbon and Graphite Products; Activated Carbon. G, Volume 15.02—Glass; Ceramic Whitewares

(continued)

Table 2 Expanded description of ASTM standards (continued)

ASTM designation	Title	Source(a)	Application	Sample description	Measurement	Comments
C 1182	Standard Test Method for Determining the Particle Size Distribution of Alumina by Photo Sedimentation	G	Specifications	1 g of sample in 200 mL of dispersing liquid	0.1-20 μm with median particle diameter from 0.5-5.0 μm	Based on Stokes law settling of aqueous powder dispersion as measured by light absorption in centrifuge. Requires powder density. Calculated as equivalent spherical diameter.
D 3663	Standard Test Method for Surface Area of Catalysts	D	Specifications, process control	<1 g	Surface areas > 1 m^2/g	Surface area determined by measuring the volume of nitrogen gas absorbed at various low pressure levels
D 4222	Standard Test Method for Determination of Nitrogen Adsorption and Desorption Isotherms of Catalysts by Static Volumetric Measurements	D	Analysis of catalysts	Mass with estimated total surface area of 20-100 m^2	See D 4641	See D 4641
D 4284	Standard Test Method for Determining Pore Volume Distribution of Catalysts by Mercury Intrusion Porosimetry	D	Analysis of pore volume distribution of catalysts and catalyst carriers	Instrument- and sample-specific	Apparent pore entrance diameter of 0.003-100 μm	Pore volume determined by measuring the volume of mercury forced into pores at various pressures. Measure of pore entrance size; does not measure closed pores. Requires knowledge of wetting contact angle.
D 4404	Standard Test Method for Determination of Pore Volume and Pore Volume Distribution of Soil and Rock by Mercury Intrusion Porosimetry	C	Analysis of soil and rock	Instrument-specific	Apparent pore entrance diameter of 100 μm and 2.5 nm	Similar to D 4284. Requires wetting contact angle and surface tension data.
D 4438	Standard Test Method for Particle Size Distribution of Catalytic Materials by Electronic Counting	D	Analysis of catalysts and catalyst carriers	250 mL of electrolyte with dilute suspension of sample	20-150 μm equivalent spherical diameter	Determines equivalent spherical diameter. Based on disruption of electric field across an orifice.
D 4641	Standard Practice for Calculation of Pore Size Distributions of Catalysts From Nitrogen Desorption Isotherms	D	Analysis of catalysts and catalyst carriers	...	15-1000 Å radius pores	Procedure for calculation of pore size distribution by analyzing desorption of layer of nitrogen on pore wells. Test method in D 4222.
E 831	Standard Test Method for Linear Thermal Expansion of Solid Materials by Thermomechanical Analysis	E	Data for design	2-10 mm long; lateral dimension <10 mm; ends flat/parallel ±25 μm	CTE > 5 μm/m · °C	Applicable from 120-600 °C; may be extended. Mechanical measurement external to test chamber.

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Table 2 Expanded description of ASTM standards (continued)

ASTM designation	Title	Source(a)	Application	Sample description	Measurement	Comments
Deposits						
B 487	Standard Test Method for Measurement of Metal and Oxide Coating Thickness by Microscopical Examination of a Cross Section	A	Specifications, coating performance	Variable thickness	Accuracy of 0.8 μm	General guidelines for metallographic preparation.
B 659	Guide for Measuring Thickness of Metallic and Inorganic Coatings	A	Specifications	Variable thickness	Varies with method	General summary of measurement methods. References specific ASTM and ISO standards.
C 177	Standard Test Method for Steady State Heat Flux Measurements and Thermal Transmission Properties by Means of the Guarded Hot Plate Apparatus	B	General property measurement	Variable	...	Absolute measure of thermal conductivity. Provides guidelines, not specifics on test methodology. Measures temperature different through specimen(s) heated on one side. Details on sources of uncertainties.
C 373	Standard Test Method for Water Absorption, Bulk Density, Apparent Porosity and Apparent Specific Gravity of Fired Whiteware Products	G	Determination of degree of maturation of a ceramic body	Five, 50 g each	...	Gravimetric method. Specimens boiled in water for 5 h plus 24 h soak.
C 577	Standard Test Method for Permeability of Refractories	F	Specifications, design	51 mm cubes	0-900 cm^3/min	Based on flow of air or nitrogen through cube.
C 633	Standard Test Method for Adhesion or Cohesion Strength of Flame-Sprayed Coatings	A	Acceptance testing, operator qualification	Substrate diameter is 2.54 cm, thickness > 0.38 mm	Varies with material and mode of failure	Deposit bonded with adhesive to test fixture. Ambient temperature use.
C 714	Standard Test Method for Thermal Diffusivity of Carbon and Graphite by a Thermal Pulse Method	F	To determine sulfur content in reactor graphite	6-12 mm diameter disks 2-4 mm thick	Diffusivities of 0.04-2.0 cm^2/s	Measurement to 500 °C, in furnace. Short pulse high intensity heating from flash lamp. Temperature change with time on back surface used to calculate diffusivity.

(a) Source: 1996 Annual Book of ASTM Standards. A, Section 2—Nonferrous Metal Products, Volume 02.05—Metallic and Inorganic Coatings: Metal Powders, Sintered Plus Structural Parts. B, Section 4—Construction, Volume 04.06—Thermal Insulation; Environmental Acoustics. C, Volume 04.08—Soil and Rock (I): D 420 - D4914. D, Section 5—Petroleum Products, Lubricants and Fossil Fuels, Volume 05.03—Petroleum Products and Lubricants (III): D 4636 - Latest; Catalysts. E, Section 14—General Methods and Instrumentation, Volume 14.02—General Test Methods, Nonmetal: Chromatography; Durability of Nonmetallic Materials; Forensic Sciences; Laboratory Apparatus; Statistical Methods. F, Section 15—General Products, Chemical Specialties, and End Use Products, Volume 15.01—Refractories: Carbon and Graphite Products; Activated Carbon. G, Volume 15.02—Glass; Ceramic Whitewares

(continued)

Table 2 Expanded description of ASTM standards (continued)

ASTM designation	Title	Source(a)	Application	Sample description	Measurement	Comments
E 228	Standard Test Method for Linear Thermal Expansion of Solid Materials with a Vitreous Silica Dilatometer	E	Design data, specifications	Variable such that $\Delta L/L$ is at least ±20 μm/m	Variable	Applicable from −180-900 °C. Specimen expansion measured external to furnace.
E 289	Standard Test Method for Linear Thermal Expansion of Rigid Solids with Interferometry	E	Design data, specifications	Cylinder dimensions vary with technique	CTE below 5 μm/m · K; precision better than ±40 nm/m · K	Optically measures dimensional change by observing change in fringe patterns. High precision techniques. SRMs available for calibration. Applicable for −150-700 °C.
E 1461	Standard Test Method for Thermal Diffusivity of Solids by the Flash Method	E	Design data	Disk, 6-12 mm diameter; 1.5-4 mm thick	Thermal diffusivity of 0.1-1000 mm^2/s	Laser flash heating in furnace 100-2500°F, vacuum or inert gas environment. More detailed form of C714.

(a) Source: 1996 Annual Book of ASTM Standards. A, Section 2—Nonferrous Metal Products, Volume 02.05—Metallic and Inorganic Coatings: Metal Powders, Sintered Plus Structural Parts. B, Section 4—Construction, Volume 04.06—Thermal Insulation; Environmental Acoustics. C, Volume 04.08—Soil and Rock (I): D 420 - D4914. D, Section 5—Petroleum Products, Lubricants and Fossil Fuels, Volume 05.03—Petroleum Products and Lubricants (III): D 4636 - Latest; Catalysts. E, Section 14—General Methods and Instrumentation, Volume 14.02—General Test Methods, Nonmetal: Chromatography; Durability of Nonmetallic Materials; Forensic Sciences; Laboratory Apparatus; Statistical Methods. F, Section 15—General Products, Chemical Specialties, and End Use Products, Volume 15.01—Refractories: Carbon and Graphite Products; Activated Carbon. G, Volume 15.02—Glass; Ceramic Whitewares

3.2 *Mechanical Properties*

The durability of TBCs depends on their mechanical properties. These properties, particularly as a function of temperature, are the subject of considerable research but their measurement has not been widely standardized. Coating adhesion to the substrate (e.g., zirconia adhesion to the metallic bond coat) is the primary measure of suitability for application. Cohesive strength is also important, but less investigated. Methods for the qualitative evaluation of adhesion, such as flexure, scratch, or impact testing, have been developed for less complex materials such as zinc coatings. These are of limited value but have been included in Tables 1 and 2. The most commonly used adhesion test is ASTM C 633, "Standard Test Method for Adhesion or Cohesive Strength of Flame-Sprayed Coatings (Ref 6, 7), which is similar to DIN 50 160-A and JIS H8666-80. This technique is limited by the use of epoxy adhesive grip attachment to test temperatures significantly lower than application temperatures.

Brown et al. (Ref 8) have reviewed methods used to measure adherence of coatings applied by thermal spray, including flexure and fracture mechanics techniques, and conclude that widely used tests do not provide information required and that simulation of service conditions is vital. Beardsley (Ref 9) has addressed the mechanical behavior of coatings intended for diesel applications and developed techniques for the evaluation of compression-tension fatigue on plasma-sprayed coatings removed from substrates. These techniques have not been developed as standards.

Recognition of the importance of more subtle properties, such as fracture toughness, thermal shock response, and thermomechanical fatigue, has been manifested in research on the modeling of coating behavior (Ref 10). However, standards for the measurement of these properties are not available. The availability of standard, broadly accepted methods for the measurement of coating properties would enhance the development of TBCs by allowing comparisons from one organization to another.

Hardness is commonly used as a process control measure. Its application to coatings has been recognized in the development of BS 5411-part 6, a standard on measuring Vickers and Knoop microhardness for metallic coatings. Fracture mechanics analysis can be combined with microindentation to measure the fracture behavior of ceramic coatings (Ref 11). However, this more sophisticated analytical technique has not been developed as a standard test method.

Significant research has been conducted on the use of scratch techniques to evaluate the adhesion of thin coatings (Ref 12). Although not particularly applicable for thick coatings, the ease of this technique to evaluate coating/substrate systems offers potential for study of the much thinner coatings now being developed.

3.3 *Oxidation and Hot Corrosion*

Significant effort has been directed to evaluating the high-temperature oxidation and hot-corrosion behavior of TBCs for gas turbine applications. Laboratory tests such as static furnace and high- and low-velocity burner rig exposure (Ref 13) have been related to coating behavior in specific engine environments by varying parameters, such as temperature, time, cycling rate, or corrodent levels. Specimen geometry and test conditions vary among manufacturers, but over time they have been used to compare material behavior, allowing a degree of performance prediction. Extensive databases on coating behavior have been developed by individual manufacturers. Because these databases represent large investments and because these evaluations are so closely related to applications in competitive markets, development of a standard test methodology is unlikely.

3.4 *Erosion Behavior*

The erosion resistance of coatings for gas turbine applications has been the topic of research for many years. Like corrosion evaluation, this testing is intended to simulate performance in an engine, and the development of standard test methodologies representative of gas turbine or diesel condition is unlikely. ASTM G 76, "Standard Practice for Conducting Erosion Tests by Solid Particle Impact Using Gas Jets," has been developed for low-velocity, low-temperature evaluation. It does not provide appropriate information for TBC operating conditions.

3.5 *Thermal Properties*

The measurement of thermal conductivity or diffusivity and the coefficient of thermal expansion are obviously important to TBC design and performance prediction. Measurement of these properties over the temperature range of application (900 to 1200 °C) is required, because extrapolation from room temperature introduces inaccuracies. Thermal diffusivity, which can be related to thermal conductivity by multiplying by the product of density and heat capacity per unit mass, is often measured for bulk materials and freestanding coatings by the laser flash technique. ASTM E 1461 addresses the use of flash methods for measurement of bulk materials to 223 °C and could be extended to coated materials. ASTM C 177 proscribes the more complex guarded hotplate method, which is generally regarded as the most accurate measure of conductivity and is used to 800 °C. Accurate calculation of conductivity from diffusivity requires the use of density and heat capacity values at the temperature of interest, which are not often available. ASTM C 177, like ASTM E 1461, may be extended to coatings. More importantly, the relationship of less expensive and less labor-intensive methods to the more sophisticated method is worth establishing.

Techniques such as the thermal comparator method (Ref 14, 15) for conductivity measurement were developed because of concerns about the apparently low thermal conductivity of thin films used in electronic and laser optic applications, as well as concerns about the adverse effects that poor heat dissipation would have on device performance. These methods have the potential to be developed into standard measurement techniques and may be suitable for thin TBCs in particular.

Thermal barrier coatings are anisotropic whether applied by thermal spray or PVD. Broadly accepted consistent descriptions are not available for microstructural features such as splat or PVD column dimension and size distributions. Accurate measurement of these microscopic features, as well as of macroscopic features such as thickness, is necessary for the specification of coatings. Optical metallographic preparation and analysis is skill dependent, and standard reference materials with known characteristics would aid in interpretation. The difficulty with consistent metallographic preparation has fostered

Table 3 Standard reference materials (SRMs) for powder size measurement

SRM	Material	Particle size, μm	Intended method
59	Silicon nitride	0.2-10	X-ray sedigraph
114	Portland cement	0.2-50(a)	Air permeability
1003b	Glass	10-60	Sieve
1004a	Glass	40-170	Sieve
1017b	Glass	100-310 nominal	Sieve
1018b	Glass	225-780 nominal	Sieve
1019a	Glass	760-2160	Sieve
1978	Zirconium oxide	0.33-2.19	X-ray sedigraph
1982	Zirconium oxide	10-150	Light scattering

(a) Specified for reference material in ASTM C 721 for conversion from specific surface area to particle size by air permeability analysis. Source: National Institute of Standards and Technology

the development of an industry-led program to identify appropriate specimen preparation techniques (Ref 16, 17). These studies should reduce the variability of observed microstructures and potentially lead to consensus-based standard preparation techniques.

Standard test methods have been developed for other applications and may be useful in characterizing TBCs. Currently, ASTM C 577, "Standard Test Method for Permeability of Refractories," is used to measure connected porosity by means of the Brunauer-Emmett-Teller gas absorption method. However, this method does not allow for size or orientation analysis, nor does it address the analysis of closed porosity.

Microstructural design to achieve specific thermal and mechanical properties is expected to gain interest as techniques of modeling coating behavior evolve. These models require measurement of properties at levels commensurate with elements in the models. Methods of measurement for this scale (less than 10 μm) are limited. Measurement by x-ray and neutron scattering of details such as porosity, crack size, crack distribution, and crack orientation provide valuable data for modeling. Measurement of elastic modulus and cohesive strength by the use of instrumented indentation offers similar promise, although microscopic measurements provide an intrinsic material property that may be unrepresentative of the coating system.

4. Summary

Increased commerce in TBCs is expected due to the improved performance and component life they provide to both gas turbines and diesel engines. This increased commerce, reflected in greater numbers of suppliers and users, will necessitate the use of broadly accepted methods for analysis and characterization of processing parameters and coatings. Standard measurement techniques and standard reference materials suitable for the analysis of properties are not available for TBCs specifically. Conventional TBCs processed by thermal spray and PVD, particularly low-conductivity thin film coatings, require examination as coated systems that include substrates in order to properly analyze critical interfacial strength and thermal effects. Available property measurement standards and reference materials are in some cases applicable to coatings or can be extended to coatings.

Improved methods of measuring adhesion, cohesion, and thermal properties for conventional coatings are necessary. In the long term, the development and application of nanolayered and functionally graded coatings will require techniques for measuring properties on scales characteristic of the microstructural features of these materials.

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