



Standard Guide for Testing the Thermal Properties of Advanced Ceramics¹

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1. Scope

1.1 This guide covers the thermal property testing of advanced ceramics, to include monolithic ceramics, particulate/whisker-reinforced ceramics, and continuous fiber-reinforced ceramic composites. It is intended to provide guidance and information to users on the special considerations involved in determining the thermal properties of these ceramic materials.

1.2 This guide is based on the use of current ASTM standards for thermal properties where appropriate and on the development of new test standards where necessary.

1.3 It is not the intent of this guide to rigidly specify particular thermal test methods for advanced ceramics. Guidance is provided on how to utilize the most commonly available ASTM thermal test methods, considering their capabilities and limitations.

1.4 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 ASTM Standards:²

2.1.1 Specific Heat:

C 351-92b Test Method for Mean Specific Heat of Thermal Insulation

D 2766-95 Test Method for Specific Heat of Liquids and Solids

E 1269-95 Test Method for Determining Specific Heat Capacity by Differential Scanning Calorimetry

2.1.2 Thermal Conductivity:

C 177 Test Method for Steady-State Heat Flux Measurements and Thermal Transmission Properties by Means of the Guarded-Hot-Plate Apparatus

C 182 Test Method for Thermal Conductivity of Insulating Firebrick

C 201 Test Method for Thermal Conductivity of Refractories

C 202 Test Method for Thermal Conductivity of Refractory Brick

C 408 Test Method for Thermal Conductivity of Whiteware Ceramics

C 518 Test Method for Steady-State Heat Flux Measurements and Thermal Transmission Properties by Means of the Heat Flow Meter Apparatus

C 767 Test Method for Thermal Conductivity of Carbon Refractories

C 1044 Practice for Using the Guarded-Hot-Plate Apparatus or Thin-Heater Apparatus in the Single-Side Mode

C 1045 Practice for Calculating Thermal Transmission Properties from Steady-State Heat Flux Measurements

C 1113 Test Method for Thermal Conductivity of Refractories by Hot Wire (Platinum Resistance Thermometer Technique)

C 1114 Test Method for Steady-State Thermal Transmission Properties by Means of the Thin-Heater Apparatus

C 1132 Practice for Calibration Practice for Heat Flow Meter Apparatus²

E 1225 Test Method for Thermal Conductivity of Solids by Means of the Guarded-Comparative-Longitudinal Heat Flow Technique

E 1530 Test Method for Evaluating the Resistance to Thermal Transmission of Thin Specimens of Materials by the Guarded Heat Flow Meter Technique

2.1.3 Thermal Expansion:

C 372 Test Method for Linear Thermal Expansion of Porcelain Enamel and Glaze Frits and Fired Ceramic Whiteware Products by the Dilatometer Method

C 1300 Test Method for Linear Thermal Expansion of Glaze Frits and Ceramic Whiteware Materials by the Interferometric Method

E 228 Test Method for Linear Thermal Expansion of Solid Materials With a Vitreous Silica Dilatometer

E 289 Test Method for Linear Thermal Expansion of Rigid Solids with Interferometry

E 831 Test Method for Linear Thermal Expansion of Solid Materials by Thermomechanical Analysis

2.1.4 Thermal Diffusivity:

C 714 Test Method for Thermal Diffusivity of Carbon and Graphite by a Thermal Pulse Method

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² For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

E 1461 Test Method for Thermal Diffusivity of Solids by the Flash Method

2.1.5 *Emittance/Emissivity*:

E 408 Test Methods for Total Normal Emittance of Surfaces Using Inspection Meter Techniques

E 423 Test Method for Normal Spectral Emittance at Elevated Temperatures of Nonconducting Specimens

2.1.6 *General Standards*:

C 373 Test Method for Water Absorption, Bulk Density, Apparent Porosity, and Apparent Specific Gravity of Fired Whiteware Products

C 1145 Terminology of Advanced Ceramics

E 122 Practice for Choice of Sample Size to Estimate a Measure of Quality for a Lot or Process

E 473 Terminology Relating to Thermal Analysis

E 1142 Terminology Relating to Thermophysical Properties

C 1045 Practice for Calculating Thermal Transmission Properties from Steady-State Heat Flux Measurements

3. Terminology

3.1 *Definitions*:

3.1.1 *advanced ceramic*, n —a highly engineered, high-performance, predominantly nonmetallic, inorganic, ceramic material having specific functional attributes. (C 1145)

3.1.2 *ceramic matrix composite*, n —a material consisting of two or more materials (insoluble in one another), in which the major continuous component (matrix component) is a ceramic, while the secondary component/s (reinforcing component) may be ceramic, glass-ceramic, glass, metal, or organic in nature. These components are combined on a macroscale to form a useful engineering material possessing certain properties or behavior not possessed by the individual constituents. (C 1145)

3.1.3 *coefficient of linear thermal expansion*, $\alpha [T^{-1}]$, n —the change in length, relative to the length of the specimen, accompanying a unit change of temperature, at a specified temperature. [This property can also be considered the instantaneous expansion coefficient or the slope of the tangent to the $\Delta L/L$ versus T curve at a given temperature.] (E 1142)

3.1.4 *continuous fiber-reinforced ceramic composite (CFCC)*, n —a ceramic matrix composite in which the reinforcing phase(s) consists of continuous filaments, fibers, yarns, or knitted or woven fabric. (C 1145)

3.1.5 *differential scanning calorimetry (DSC)*, n —a technique in which the difference in energy inputs into a specimen and a reference material is measured as a function of temperature while the substance and reference material are subjected to a controlled temperature program. (E 1269)

3.1.6 *discontinuous fiber-reinforced ceramic composite*, n —a ceramic matrix composite reinforced by chopped fibers. (C 1145)

3.1.7 *emittance (emissivity)*, $\epsilon (nd)$, n —the ratio of the radiant flux emitted by a specimen per unit area to the radiant flux emitted by a black body radiator at the same temperature and under the same conditions. Emittance ranges from 0 to 1, with a blackbody having an emittance of 1.00. (E 423)

3.1.8 *linear thermal expansion*, $[nd]$, n —the change in length per unit length resulting from a temperature change. Linear thermal expansion is symbolically represented by $\Delta L/L_0$, where ΔL is the observed change in length $\Delta L = L_2 - L_1$, and L_0 , L_1 , and L_2 are the lengths of the specimen at reference temperature T_0 and test temperatures T_1 and T_2 . (E 228)

3.1.9 *mean coefficient of linear thermal expansion*, $\alpha_L [T^{-1}]$, n —the change in length, relative to the length of the specimen, accompanying a unit change of temperature measured across a specified temperature range (T_1 to T_2). (C 372)

3.1.10 *particulate reinforced ceramic matrix composite*, n —a ceramic matrix composite reinforced by ceramic particulates. (C 1145)

3.1.11 *specific heat (specific heat capacity)*, $C [mL^{-1}T^{-2}\theta^{-1}]$, n —the quantity of heat required to provide a unit temperature increase to a unit mass of material. (E 1142)

3.1.12 *thermal conductivity*, $\lambda [mLT^{-1}\theta^{-1}]$, n —the time rate of heat flow, under steady conditions, through unit area, per unit temperature gradient in the direction perpendicular to the area. (E 1142)

3.1.13 *thermal diffusivity*, $[L^2T^{-1}]$, n —the property given by the thermal conductivity divided by the product of the bulk density and heat capacity per unit mass. (E 1461)

3.1.14 *thermodilatometry*, n —a technique in which a dimension of a substance under negligible load is measured as a function of temperature while the substance is subjected to a controlled temperature program in a specified atmosphere. (E 473)

3.2 Units for Thermal Properties:

Property	SI Units	Abbreviation
Specific heat capacity	joules/(gram-kelvin)	J/(g·K)
Thermal Conductivity	watts/(metre-kelvin)	W/(m·K)
Thermal diffusivity	metre/second ²	m/s ²
Thermal expansion	metre/(metre-kelvin)	K ⁻¹
Emittance/emissivity	no dimensions	—

4. Summary of Guide

4.1 Five thermal properties (specific heat capacity, thermal conductivity, thermal diffusivity, thermal expansion, and emittance/emissivity) are presented in terms of their definitions and general test methods. The relationship between thermal properties and the composition, microstructure, and processing of advanced ceramics is briefly outlined, providing guidance on which material characteristics have to be considered in evaluating the thermal properties. Additional sections describe sampling considerations, test specimen preparation, and reporting requirements.

4.2 Current ASTM test methods for thermal properties are tabulated in terms of test method concept, testing range, specimen requirements, standards/reference materials, capabilities, limitations, precision, and special instructions for monoliths and composites.

5. Significance and Use

5.1 The high-temperature capabilities of advanced ceramics are a key performance benefit for many demanding engineering applications. In many of those applications, advanced ceramics will have to perform across a broad temperature range. The thermal expansion, thermal diffusivity/conductivity, specific heat, and emittance/emissivity are crucial engineering factors in integrating ceramic components into aerospace, automotive, and industrial systems

5.2 This guide is intended to serve as a reference and information source for testing the thermal properties of advanced ceramics, based on an understanding of the relationships between the composition and microstructure of these materials and their thermal properties.

5.3 The use of this guide assists the testing community in correctly applying the ASTM thermal test methods to advanced ceramics to ensure that the thermal test results are properly measured, interpreted, and understood. This guide also assists the user in selecting the appropriate thermal test method to evaluate the particular thermal properties of the advanced ceramic of interest.

5.4 The thermal properties of advanced ceramics are critical data in the development of ceramic components for aerospace, automotive, and industrial applications. In addition, the effect of environmental exposure on thermal properties of the advanced ceramics must also be assessed.

6. Procedure

6.1 Review Sections 7-10 to become familiar with thermal property concepts and thermal testing issues for advanced ceramics, specimen preparation guidance, and reporting recommendations.

6.2 Review the test method text and tables in Section 11 for the property you need to determine. Use the text and tables to help select the most appropriate ASTM test method for evaluating the thermal property of interest for the specific advanced ceramic.

6.3 Perform the thermal property test in accordance with the selected ASTM test method, but refer back to the guide for directions and recommendations on material characterization, sampling procedures, test specimen preparation, and reporting results.

7. Thermal Properties and Their Measurement

7.1 Specific Heat Capacity:

7.1.1 Specific heat capacity is the amount of energy required to increase the temperature by one unit for a unit mass of material. It is a fundamental thermal property for engineers and scientists in determining the temperature response of materials to changes in heat flux and thermal conditions. The SI units for specific heat capacity are joules/(gram·K). Since the specific heat capacity changes with temperature, a specific heat capacity value must always be associated with a specific test temperature or temperature range.

7.1.2 Specific heat capacity is commonly measured by calorimetry in which changes in thermal energy are measured against changes in temperature. The two common calorimetry methods are differential scanning calorimetry and drop calorimetry.

7.1.3 Differential scanning calorimetry heats the test material at a controlled rate in a controlled atmosphere through the temperature region of interest. The heat flow into the test material is compared to the heat flow into a reference material to determine the energy changes in the test material as a function of temperature.

7.1.4 In drop calorimetry, the test sample is heated to the desired temperature and then immersed in an instrumented, liquid-filled container (calorimeter), which reaches thermal

equilibrium. The increase in temperature of the calorimeter liquid/container is a measure of the amount of heat in the test specimen.

7.1.5 In any calorimetry test, the experimenter must recognize that phase changes and other thermo-physical transformations in the material will produce exothermic and endothermic events which will be captured in the test data. The thermal events must be properly identified and understood within the context of the material properties, chemistry, and phase composition across the temperature range of interest.

7.2 Thermal Conductivity:

7.2.1 Thermal conductivity is a measurement of the rate of heat flow through a material for a given temperature gradient. It is normalized for thickness and cross-sectional area to give a material specific value. The thermal conductivity of a ceramic is used in determining the effectiveness of a ceramic either as a thermal insulator or as a thermal conductor. The SI units for thermal conductivity are watts/(metre·K). As with other thermal properties, thermal conductivity changes with temperature, so that a thermal conductivity value for a material must be associated with a specific test temperature.

7.2.2 In electrically nonconductive ceramics, thermal conductivity occurs by lattice vibration (phonon) conductivity and by radiation (photon) at higher temperatures (>500°C). Thermal conductivity decreases when the mean free path of the phonons and photons decreases. Lattice imperfections, differences in atomic weight between anions and cations, non-stoichiometric compositions, solid solutions, amorphous atomic structures, porosity, and grain boundaries all act as scattering sites for phonons and reduce the thermal conductivity of the material.

7.2.3 Thermal conductivity is commonly measured by steady-state methods, that is, heat flow techniques, heat flux meter techniques, guarded hot-plate techniques, and calorimetry techniques. It can also be determined by transient techniques (hot wire and flash diffusivity).

7.2.4 In comparative heat flow techniques, the test specimen is subjected to a known heat flow and the temperature differential is measured across the dimension of interest. The test system and the specimen are configured with insulation and heaters to minimize heat flow perpendicular to the direction of interest. The heat flow in the direction of interest is determined through the use of reference materials of known thermal conductivity. In the heat flux meter technique, the heat flux in the test is measured by a calibrated heat flux transducer. In both techniques, the thermal conductivity of the material is then calculated as follows:

$$\lambda = q \Delta L / (\Delta T) \quad (1)$$

where:

λ = thermal conductivity,

q = heat flow/unit area,

ΔL = distance across which the temperature difference is measured, and

ΔT = measured temperature difference.

Thermal conductivity can be calculated from thermal diffusivity measurements, using the specific heat and the material density as follows:

λ = thermal diffusivity * specific heat capacity * density (2)

7.3 Thermal Expansion (Therm dilatometry):

7.3.1 All materials expand or contract with changes in temperature, with most materials expanding with increasing temperature. Thermal expansion is often very important in engineering applications, because differences in thermal expansion between fitted or bonded components can produce thermal stresses, leading to component failure.

7.3.2 The thermal expansion for a given ceramic composition and phase is a function of crystal structure and of atomic bond strength. In ceramics with anisotropic crystal structures, the thermal expansion is different along the different crystal axes. (This is of particular concern for single crystal specimens and for specimens with oriented grain structures). For some specific ceramics (cordierite, aluminum titanate, and zirconium phosphate), the thermal expansion may be zero or negative in certain crystal axes in specific temperature regimes.

7.3.3 The quantitative determination of the change in dimension as a function of temperature is defined as the mean coefficient of linear thermal expansion – the ratio of a given change in length per unit length for a specimen for a specific change in temperature as follows:

$$\alpha_L = [(L_2 - L_1)/(T_2 - T_1)]/L_0 \quad (3)$$

where:

L_0 , L_1 , and L_2 = lengths of the specimen at reference temperature T_0 and test temperatures T_1 and T_2 , respectively.

7.3.4 The units for mean coefficient of linear thermal expansion are metres/(metre · K). The mean coefficient of linear thermal expansion for a material has to be defined for a given temperature range, for example, 7×10^{-6} m/m·K for 25 to 500°C.

7.3.5 The mean coefficient of linear thermal expansion across a temperature range is different than the instantaneous coefficient of thermal expansion, which is the tangent slope at a specific temperature for the expansion-temperature curve of the sample.

7.3.6 Thermal expansion is commonly measured by therm dilatometry, a technique in which a known dimension of a specimen under negligible load is measured as a function of temperature while the specimen is subjected to a controlled-temperature program in a specified atmosphere. The measurement of the dimensional change can be done by direct mechanical measurement or by optical techniques (interferometry and optical lever)

7.3.7 Different crystalline phases in ceramics have different thermal expansion characteristics. Major phase changes in ceramics can give abrupt or progressive changes in length with increasing or decreasing temperature and may cause confusion when included in overall expansion measurements. In a similar manner, crystallization and changes in amorphous, glassy phases in ceramics can produce marked changes in the thermal expansion over specific temperature regimes.

7.3.8 Thermal expansion measurements for ceramics often show heating/cooling hysteresis when microcracking or minor phase changes occur. There are also irreversible thermal expansion effects, based on annealing, heating rate, creep,

crystallization, or microcracking. Thermal expansion tests done only on heating can be misleading, and thermal expansion measurements should be done under both heating and cooling conditions.

7.4 Thermal Diffusivity:

7.4.1 Thermal diffusivity is a measurement of the rate of temperature change in a material measured under transient conditions. It is defined as the ratio of the thermal conductivity to the heat capacity per unit volume as follows:

$$\text{Diffusivity} = \lambda / (C_p \rho) \quad (4)$$

where:

λ = thermal conductivity,

C_p = specific heat, and

ρ = the bulk density,

all at the specific temperature of measurement.

The units of thermal diffusivity are metre/second².

7.4.2 Thermal diffusivity is important in characterizing the transient thermal response of ceramics that are used in heat transfer applications, either as insulators or as thermal conduction paths. As with the other thermal properties that change with temperature, a specific thermal diffusivity value must be defined for a specific temperature or a temperature range.

7.4.3 Thermal diffusivity is experimentally determined by measuring the temperature-time response of a material to a thermal event. The current method of choice is flash diffusivity, in which one side of a specimen of known thickness and temperature is subjected to a short duration thermal pulse. The energy of the pulse is absorbed on the front surface of the specimen and the resulting rear face temperature rise is measured. Thermal diffusivity is calculated from the specimen thickness and the time required for the rear face temperature to reach a specified percentage of its maximum value.

7.4.4 All of the microstructural variables that have an impact on thermal conductivity will have a similar effect on the thermal diffusivity.

7.5 Emittance/Emissivity:

7.5.1 All objects radiate energy depending on their temperature and their radiative characteristics. This radiation is called thermal radiation, because it is strongly dependent on temperature. A perfect emitter (a blackbody) emits radiation across a range of wavelengths according to its surface temperature through the Planck radiation equation. The amount of hemispherical radiated energy per unit surface area at a given temperature for a blackbody is calculated through the Stefan-Boltzmann's law as follows:

$$E_{b,\text{total}} = 5.670 \times 10^{-8} \text{ W}/(\text{m}^2 \cdot \text{K}^4) * T^4 \quad (5)$$

where:

T = specimen temperature for hemispherical emittance

7.5.2 At high temperatures, the emittance of radiation by a material has significant impact on its thermal condition, based on how the thermal radiation is emitted, absorbed, and reflected. For advanced ceramics operating at high temperatures (>600°C) where radiation is a major mode of heat transfer, the emittance properties of the ceramic are necessary to model the thermal conditions and to determine the heat transfer rates under heating and cooling conditions.

7.5.3 The measurement of emittance has a directional component based on the observation angle to the plane of the sample. Directional emittance is measured at a specific observation angle. Normal emittance is a special case of directional emittance, measured normal to the plane of the sample. Hemispherical emittance is measured by integration over the entire range of solid observation angles.

7.5.4 Emittance can also be characterized as either “total” or “spectral.” Total emittance is a measurement of the radiant energy across the entire range of thermal wavelengths and is commonly utilized for total radiation pyrometry and radiant heat transfer analysis. Spectral emittance is a measurement of the radiant energy at/across a particular portion of the wavelength/frequency spectrum. The emittance at a particular frequency is important in temperature measuring equipment, such as optical pyrometers.

7.5.5 Most materials are not perfect emitters. The emission of radiation from a surface depends on many factors: temperature, bulk composition, surface composition (impurities, coatings, and oxidation), optical transparency, surface profile/roughness, radiation wavelength, and observation angle. The relative radiant flux of a given material can be characterized by the term emittance (also called emissivity). Emittance is the ratio of the radiant flux emitted by a specimen per unit area to the radiant flux emitted by blackbody radiator at the same temperature and under the same conditions.

7.5.6 Emittance is determined by measuring the emitted thermal radiation at a given temperature and then comparing it to a reference standard of known emittance or by direct comparison to an experimental “blackbody” at the same temperature.

8. Test Specimen Characterization

8.1 Introduction:

8.1.1 Advanced ceramics, both monolithic and composite, offer a wide range of thermal properties, from thermal insulators to thermal conductors. Nominal thermal property values for a range of advanced monolithic ceramics are given in Table 1. Note the range of thermal properties listed in the table. Advanced ceramics such as aluminum nitride and beryllia with their high thermal conductivity are often used as thermal conductors.

8.1.2 Table 2 provides nominal thermal properties for different types of ceramic fibers used in continuous fiber-reinforced ceramic matrix composites. When these different

ceramic fibers are combined with different ceramic matrices, the resulting composites can contain constituents whose thermal properties are widely different.

8.1.3 The range of thermal properties for advanced ceramics and the complexity inherent in ceramic composites require a detailed understanding of the relationships between composition, processing, microstructure, and thermal properties in those ceramics. With that understanding, test operators will ensure that thermal test results for advanced ceramics are valid, useful, and reproducible.

8.2 Material Characteristics and Thermal Properties:

8.2.1 Advanced ceramics cover a broad range of compositions, microstructures, and physical properties. It is not possible to give specific guidance for every current or future advanced ceramic material, but general guidelines and information are provided on the effects of composition, microstructure, and processing on the thermal properties of advanced ceramics and on thermal property measurement.

8.2.2 The thermal properties of an advanced ceramic (monolithic or composite) are a function of the material composition and constituents, its microstructure, its processing history, and its environmental exposure. For example, porosity has a very strong effect on thermal conductivity and diffusivity. In a similar manner, composites containing high thermal conductivity components can be tailored to specific thermal property targets by changing the amount and the architecture of the reinforcing component. The advanced ceramic must be adequately characterized in terms of composition, constituents, microstructure, processing methods, and exposure history.

8.2.3 The sensitivity of the thermal properties of ceramics to such variations requires that the composition and constituents in advanced ceramics be sufficiently determined and documented to avoid misinterpretation of results and to permit adequate characterization of the test material. This characterization may be simple and straightforward for monolithic ceramics, but may require extensive microstructural, chemical, and physical analysis for complex systems such as whisker or fiber-reinforced composites. The degree of characterization required depends on the variation in the thermal properties produced by changes in composition, constituents, and microstructure. Spatial variations must be considered and adequately evaluated, particularly for whisker- and particle-reinforced composites, in which reinforcement concentrations may vary spatially through the test specimen.

TABLE 1 Nominal Thermal Properties for Monolithic Ceramics at Room Temperature

NOTE 1—Thermal property data obtained from reference books and producer specifications. Values are approximate for a given class of material and are provided for the sake of general comparison. Actual values in specific test specimens will depend on composition, microstructure, porosity, and other factors. Emittance/emissivity data from—Thermal Radiative Properties, Nonmetallic Solids, Touloukian, Y. S. and DeWitt, D.P., I.F. I. Plenum, 1972.

Property at Room Temperature	Alumina, 99.5 %	Silicon Nitride	Silicon Carbide	Aluminum Nitride	Boron Nitride	Zirconia	Mullite	Beryllia	Cordierite
Density, g/cm ³	3.85	3.31	3.10	3.25	2.10	6.02	2.80	2.90	2.30
Specific heat, [J/(g·K)]	0.92	0.69	0.67	0.78	0.79	0.45	0.77	1.04	0.74
Thermal conductivity, [W/(m·K)]	35.6	15	110	115	32.8	2.2	3.5	250	3.0
Thermal diffusivity, m ² /s	10.0	6.6	53.0	45.4	19.8	0.8	1.6	82.9	1.8
Coefficient of linear thermal expansion, 10 ⁻⁶ /K	8.0	3.0	4.4	5.7	10	10.3	5.3	8.7	1.7
	(25–1000°C)	(25–1000°C)	(25–1000°C)	(25–1000°C)	(25–1000°C)	(25–1000°C)	(25–1000°C)	(25–1000°C)	(25–1000°C)
Emittance/Emissivity, 1000°C	0.1-0.3	0.7-0.9	0.7-0.9	0.7-0.9	0.7-0.9	0.1-0.3	0.4	≈0.3	NA

TABLE 2 Nominal Thermal Properties of Ceramic Fibers at Room Temperature

NOTE 1—Thermal property data obtained from reference books and producer specifications. Values are approximate and are provided for the sake of general comparison. Actual values in specific test specimens will depend on composition, microstructure, porosity, architecture, and other factors.

Property at Room Temperature	Nicalon CG ^A	Hi-Nicalon ^A	Hi-Nicalon S ^A	Sylramic SiC ^A	Nextel 312 ^B	Nextel 610 ^B	Nextel 720 ^B	T-300 Carbon ^C	P-120 Carbon ^C
Nominal composition	Silicon Carbide	Silicon Carbide	Silicon Carbide	Silicon Carbide	Alumino-borosilicate	Alumina	Alumina-Mullite	Carbon	Graphite
Density, g/cm ³	2.55	2.74	3.10	3.20	2.70	3.88	3.40	1.76	2.17
Specific Heat, [J/(g·K)]	1.14	.067	NA	0.61	≈0.7	≈0.9	≈0.85	≈0.70	≈0.70
Thermal conductivity, [W/(m·K)]	2.97	7.77	18.4	40-45	≈3(est)	≈30 (est)	≈10 (est)	8.5	640
Coefficient of Linear Thermal Expansion, 10 ⁻⁶ /K	4.0	3.5	NA	5.4	3.0	7.9	6.0	-1.4 at (23°C)	-1.45 at (23°C)
	(0-900°C)	(0-500°C)		(20-1320°C)	(100-1100°C)	(100-1100°C)	(100-1100°C)		

^ACDI Ceramics, Inc., San Diego, CA.

^B3M Corp., St. Paul, MN.

^CAmoco Performance Products, Alpharetta, GA.

8.2.4 For advanced ceramic composites (whisker, chopped-fiber, and continuous-fiber reinforced) and for monolithics with oriented or textured grain growth, anisotropy effects must be carefully evaluated and characterized. Directional effects are pronounced in thermal conductivity/diffusivity and thermal expansion measurements. The character and degree of anisotropy in such composites must be adequately characterized and understood.

8.2.5 In a similar manner, the exposure history of a component can also affect the thermal properties through oxidation, phase changes, grain growth, high-temperature reactions, corrosion, and slow crack growth. Exposure history (time, temperature, and atmosphere) for test specimens has to be well-documented.

8.2.6 Exposure and thermal effects can also occur during the thermal testing, changing the composition and microstructure. It is imperative that the test operator be aware of potential reactions, oxidation, phase changes, and other thermal events that could occur across the test temperature range. This can occur if the test temperature exceeds the maximum processing temperature or if oxidation reactions (surface or bulk) occur at elevated temperatures. Oxidation-sensitive materials should be tested in inert atmospheres. Accelerated heating rates can also produce test anomalies, because of nonuniform temperatures within a test specimen. Thermal tests should always be done with the test specimen in relative steady-state thermal equilibrium, unless transient properties are specifically desired.

8.3 Monolithic Ceramics—Material Variables:

8.3.1 For monolithic ceramics, the following material characteristics should be carefully considered in terms of their expected and actual effect on the thermal properties. Evaluation of the material characteristics may be recommended, if the thermal properties are sufficiently impacted by the pertinent characteristics.

8.3.2 Porosity has a major effect on thermal conductivity/diffusivity. From one viewpoint, porosity can be considered an additional, low thermal conductivity/capacity phase in the test specimen in which the pore volume fraction, the porosity size (mean and distribution), and its geometric distribution can all have variable effects on the thermal properties, especially the thermal transport properties. The porosity in the test specimens should be adequately defined and characterized with regard to its effect on thermal conductivity/diffusivity.

8.3.3 Variations in stoichiometry, impurities, grain size, and grain boundary phases can have a large effect on the thermal conductivity and thermal diffusivity for ceramics with high intrinsic thermal conductivity (aluminum nitride, beryllia, silicon carbide, and so forth). The effect of such composition variations and phase distributions should be carefully considered and evaluated (if feasible) in thermal tests of high thermal conductivity ceramics.

8.3.4 Phase changes and devitrification of amorphous phases may occur during thermal tests, producing anomalies in the various thermal properties. Test operators should be informed of the potential for such transformations in specimens submitted for testing.

8.3.5 Thermal expansion measurements for ceramics often show heating/cooling hysteresis when microcracking or minor phase changes occur. Irreversible thermal expansion effects may also occur in certain materials, based on annealing, rapid heating rates, creep, crystallization, or microcracking. Thermal expansion tests done only on heating can be misleading, and thermal expansion measurements should be done under both heating and cooling conditions.

8.3.6 Oriented or textured grain structures in monolithic ceramics can introduce anisotropic effects in the thermal properties. Such grain structures are commonly developed during processing. If the thermal effects of such anisotropy are significant, the grain structure should be characterized by optical or SEM microscopy or by X-ray diffraction.

8.4 Particulate and Whisker-Reinforced Ceramic Composite—Material Variables:

8.4.1 Particulate and whisker reinforcement of advanced ceramics adds an additional degree of complexity to the composition and microstructure of the test specimen. It is critical that the particulate or whisker additions be adequately characterized in terms of composition, phase and crystal structure, particle/whisker morphology, and size distribution. It is also useful to have nominal thermal properties, that is, specific heat, thermal conductivity, and thermal expansion, for the reinforcement across the temperature range of interest. Those thermal property data will assist in interpreting the composite thermal test results.

8.4.2 As part of the composite specimen, the particulate/whisker reinforcement must be adequately defined in terms of the volume fraction, spatial distribution, and orientation/

anisotropy/texture. For example, reinforcements with high thermal conductivity relative to the matrix will have markedly different effects on the composite thermal properties, depending on how the reinforcements are distributed and oriented. For example, whiskers in a laminated, planar orientation will produce anisotropic thermal conductivity in the composite. The bulk thermal conductivity will markedly change if the particulate packing factor is high enough to produce significant particle-to-particle contact.

8.4.3 If there is a large difference in thermal expansion between the reinforcement and the matrix in the composite, residual stresses or microcracks, or both, can develop during processing. Such residual stresses and microcracks can produce anomalies in thermal expansion measurements. Microcracking can also have a direct effect on thermal conductivity/diffusivity.

8.5 *Continuous Fiber-Reinforced Ceramic Composites—Material Variables:*

8.5.1 The use of continuous fiber reinforcement in ceramic composites provides the ceramic engineer the greatest range of property control and tailoring, but also introduces the highest level of complexity into the composition and microstructure of advanced ceramics.

8.5.2 It is essential that the composition and architecture of the fiber reinforcement in the composite be adequately characterized and documented, to include the following:

8.5.2.1 Fiber composition, filament morphology (diameter and length), and fiber volume fraction.

8.5.2.2 Filament counts in tows, comprehensive description of the reinforcement architecture [one-dimensional (tows), two-dimensional (woven fabrics), and three-dimensional (weaves and braid)] to include tow count and repeat units.

8.5.2.3 The composition and morphology of any interface coatings on the fibers, used for process protection of the fibers or for development of crack deflection modes in the composite.

8.5.2.4 The composition and morphology (thickness, porosity, and grain structure) of any surface coatings on the composite component, used for surface sealing, oxidation/corrosion protection, or wear/abrasion resistance. Surface coatings may be considered as monolithic layers on the surface of the composite.

8.5.3 The matrix in the composite must also be adequately characterized in terms of composition, constituents, morphology, and grain structure. Porosity in the composite should also be characterized, considering volume fraction, pore size, shape factors, and distribution of porosity.

8.5.4 Fiber-reinforced ceramic composites often have strong anisotropic properties, determined by the reinforcement architecture with higher fiber volume loadings in specific directions. It is essential that the geometry and orientation of the reinforcement are fully documented and correlated with the thermal test results.

8.5.5 In many thermal tests (such as thermal dilatometry, diffusivity, and specific heat), the test specimens may have one or more dimensions which are small in comparison to the weave repeat elements. Experimenters should carefully consider the size of the test specimens for a specific test to ensure that an adequate number of weave repeat elements are included

in the specimen. Specimens of insufficient size may not be representative of the properties of the larger piece or may have exaggerated end or edge effects.

9. Test Specimen Sampling and Preparation

9.1 *Test Specimen Sources*—Test specimens for thermal evaluation can be taken from engineering components or fabricated test panels/billets. The selection of a particular source depends on the required test specimen geometry and the suitability of the test component for test specimen preparation. The primary objective is to select test specimens that are representative of the composition, processing, and properties of the final functional part.

9.2 *Specimen Sampling to Assess Variability:*

9.2.1 Depending on the degree of process control, variability may occur among test specimens within a batch and between different batches of test specimens. The variation may occur in the composition, microstructure, and porosity of advanced ceramics. The degree of variation will depend on the homogeneity within the batch and on reproducibility between batches from the producer. The test operator should select/prepare sufficient test specimens to provide a representative sample of the entire batch of specimens submitted for testing. In addition, batch/lot identification should be carefully noted for documentation purposes.

9.2.2 Practice E 122 provides guidance on calculating the number of units required for testing to obtain an estimate of certain precision for a given property.

9.3 *Orientation and Anisotropy Effects*—If the test material has significant anisotropy or orientation effects, it is imperative that test specimens be selected to adequately sample the anisotropy in the major directions of interest. A minimum of two directions should be selected for test specimens. The orientations should be selected to produce the maximum and minimum material properties. Each test specimen should be marked and identified so that its orientation in the original part/test piece can be identified.

9.4 *Location and Identification:*

9.4.1 Given the variability which may occur in developmental advanced ceramics and the anisotropy which may be an engineered characteristic of composite components, test specimen locations should be documented by drawing or photograph. Test specimens should be identified so that their location can be traced on the original part. Special identification should be used for test specimens taken from plate edges or from areas with local anomalies.

9.4.2 Nondestructive evaluation (NDE) methods may be of value in characterizing test plates prior to specimen preparation, particularly for fiber-reinforced ceramic composites. The NDE results will assist in locating material anomalies, such as excessive porosity or delaminations.

9.5 *Specimen Count and Repeat Tests*—Follow the instructions of the specific test method to determine the specimen count and repeat tests needed for the required precision and confidence.

9.6 *Surface Effects on Thermal Property Measurements*—Surface finish requirements are commonly detailed in thermal test methods to provide for minimal thermal resistance at contact surfaces or to ensure flat, smooth, and accurate surfaces

for measurement. Surface finish can have a strong effect on radiation absorption/emission with a direct effect on emittance measurements and on thermal diffusivity measurements by flash. Surface finish measurements may be advisable to ensure reproducibility and proper interpretation of specific thermal test results.

9.7 Test Specimen Preparation:

9.7.1 Careful machining of specimens is critical in all thermal measurements, primarily for accurate measurement of critical dimensions and for accurate fit of specimens into fixtures. Machining procedures should be sufficiently documented for reproducibility.

9.7.2 It is recommended that test specimens for thermal expansion have slightly rounded ends to prevent sideways levering. For thermal expansion measurements using optical methods, specimen ends need to be flat and parallel to a quite high precision to ensure stability and to define light paths.

9.7.3 There is no standard method for machining and finishing ceramic specimens for thermal testing. There are three general categories of machining which can be applied both to monolithic and composites ceramics

9.7.3.1 *Application-Matched Machining*—The machined surfaces of the thermal test specimen will have the same surface/edge preparation as that given to a service component.

9.7.3.2 *Customary Practices*—In instances where a customary machining procedure has been developed that is completely satisfactory for a class of materials (that is, it induces no unwanted surface/subsurface damage or residual stresses), this procedure may be used to produce the thermal specimens.

9.7.3.3 *Recommended Procedure*—In instances where Application-Matched Machining and Customary Practices are not pertinent, the following machining procedures are recommended as a method commonly used for ceramic test specimens for mechanical testing.

(1) Perform all grinding or cutting with ample supply of appropriate filtered coolant to keep the specimen and grinding wheel constantly flooded and particles flushed. Grinding can be done in two stages, ranging from coarse to fine rates of material removal. All cutting can be done in one stage appropriate for the depth of cut.

(2) The stock removal rate shall not exceed 0.03 mm per pass to the last 0.06 mm of material removed. Final finishing shall use diamond tools between 320 and 500 grit. No less than 0.06 mm shall be removed during the final finishing stage, and at a rate less than 0.002 mm per pass. Remove equal stock from opposite faces.

(3) Grinding is followed by either annealing or lapping, as deemed appropriate for a particular material and test. For silicon based ceramics (for example, silicon carbide or silicon nitride) or oxide ceramics containing a glassy phase (for example, aluminas with second phases) annealing at $\sim 1200^{\circ}\text{C}$ for ~ 2 h is generally sufficient to heal the grinding damage introduced during specimen preparation.

9.7.4 Specimens may require environmental or thermal conditioning, prior to testing, if there is a need to determine changes in thermal properties after such environmental or thermal exposure. Any conditioning procedures should be fully

documented with careful measurements of dimensions, weights, and appearance before and after conditioning.

9.7.5 Record dimensions and weights of all samples to the required tolerances or $\pm 1\%$, whichever is more restrictive. Dimensions and weights are important in calculating specimen densities. However, bulk density and apparent porosity should be calculated using Archimedes immersion (Test Method C 373) where necessary, especially for composites with significant porosity or surface roughness.

9.8 *Post Test Inspection*—In any thermal test involving high-temperature exposure, post-test examination of the tested specimen is of value in determining thermally induced changes, such as melting, sintering, phase changes, oxidation, or corrosion. Visual inspection should be done as a minimum. Dimensional inspection or microstructural and compositional analysis, or both, may be necessary if such thermal changes are suspected.

9.9 *Health and Safety*—Proper handling and safety procedures should be used for all ceramic materials in monolithic or composite forms. Refer to the Material Safety Data Sheets for the test materials for inhalation, handling, ingestion, reaction, fire, environmental, and disposal hazards.

10. Report

10.1 Follow all of the normal reporting requirements of the standard used for testing.

10.2 The accurate description of the advanced ceramic test specimens is essential for proper interpretation of the thermal test results. All ceramic test specimens should be described to the fullest extent possible, providing documentation (as applicable/available) on:

10.2.1 Material composition, chemistry, phase, and microstructure,

10.2.2 Composite composition and architecture,

10.2.3 Processing and fabrication method and exposure history,

10.2.4 Part description,

10.2.5 Sampling method and identification,

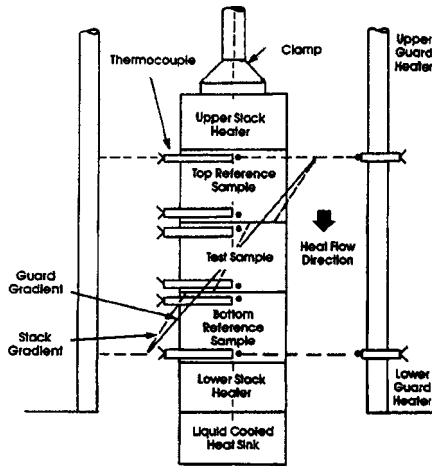
10.2.6 Specimen preparation, conditioning, and geometry, and

10.2.7 Equipment and test procedure description.

11. Test Method Comparison

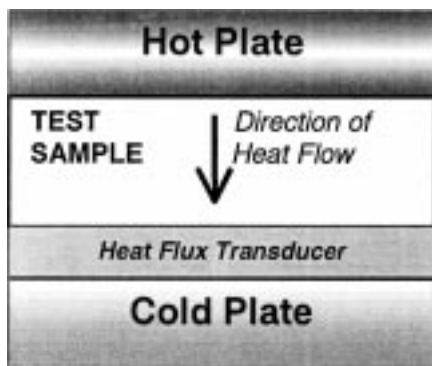
11.1 *Thermal Conductivity Test Methods*—There are five general types of thermal conductivity test methods: Comparative-Longitudinal Heat Flow, Guarded Heat Flow Meter, Guarded-Hot-Plate, Hot Wire, and Calorimetry Method for Refractories.

11.1.1 *Comparative-Longitudinal Heat Flow Technique*—A test specimen of measured cross section and thickness is sandwiched between two identical reference specimens of a material with known thermal conductivity. The test assembly (reference specimen/test specimen/reference specimen) is placed under load between two heating elements controlled at different temperatures to establish the desired thermal gradient along the length of the test assembly. The test assembly is designed (with heaters or insulation) to eliminate/minimize radial/lateral heat losses. The temperature gradient is measured by thermocouples at six known locations in the test assembly:



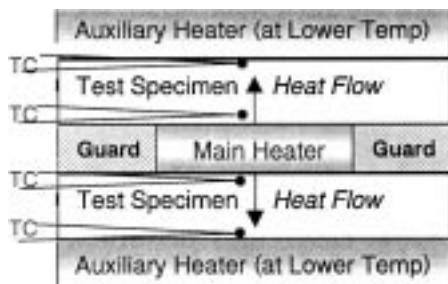
Schematic of Test Stack and Guard System for Comparative Longitudinal Heat Flow Technique

FIG. 1 Schematic of Test Stack and Guard System for Comparative Longitudinal Heat Flow Technique



Schematic of Guarded Heat Flow Meter Apparatus

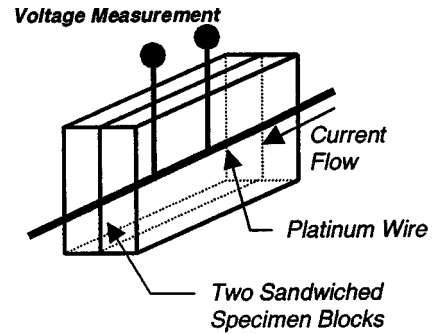
FIG. 2 Schematic of Guarded Heat Flow Meter Apparatus



Schematic of Guarded Hot Plate Apparatus

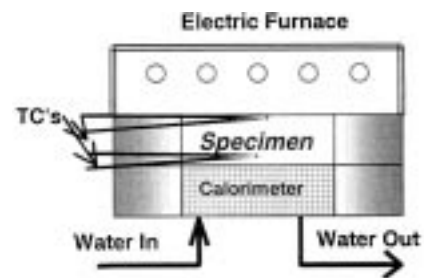
FIG. 3 Schematic of Guarded Hot Plate Apparatus

two locations in the upper reference specimen, two locations in the test specimen, and two locations in the lower reference specimen. Once thermal equilibrium is reached, the heat flux in the test assembly is calculated in the two reference specimens



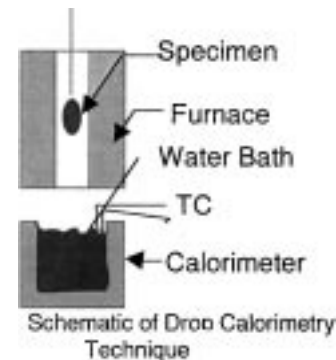
Schematic of Hot Wire Thermal Conductivity Technique

FIG. 4 Schematic of Hot Wire Thermal Conductivity Technique



Schematic of Calorimetry Technique

FIG. 5 Schematic of Calorimetry Technique



Schematic of Drop Calorimetry Technique

FIG. 6 Schematic of Drop Calorimetry Technique

based on the known thermal conductivity and the temperature gradients in the two references. Knowing the heat flux from the reference specimens and the thermal gradient in the test specimens, the thermal conductivity of the test specimen is calculated. (See Test Methods E 1225 and C 408.) See Fig. 1.

11.1.2 *Guarded Heat Flow Meter Technique*—The heat flow meter apparatus establishes a steady-state unidirectional heat flux through a test specimen between two parallel plates at constant but different temperatures. The heat flux is measured by a heat flux transducer (output voltage changes with heat flow through the sensor) in the instrument which is calibrated with a reference material of known thermal conductivity. At

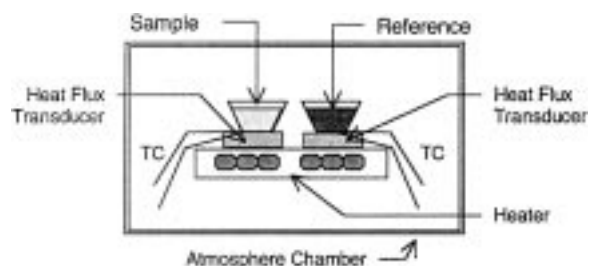
TABLE 3 Thermal Conductivity—Comparative and Guarded Heat Flow Meter Methods

ASTM Test Method	E 1225	C 408	C 518	E 1530
Measurement technique	Comparative	Comparative	Guarded Heat Flow Meter	Guarded Heat Flow Meter
Thermal conductivity range, λ	0.2-200 W/(m·K) depending on reference materials and instrument configuration	0.2-200 W/(m·K)	0.01-0.5 W/(m·K) depending on instrument configuration and meter range	0.01-5 W/(m·K) depending on instrument configuration and meter range
Test temperature range	–150 to 1000°C, depending on fixture material and thermal control range.	40 to 150°C	–150 to 300°C, depending on flux meter limit and thermal control range	–150 to 300°C, depending on flux meter limit and thermal control range.
Specimen environment	air, inert/reducing gas, vacuum depending on system design	vacuum	air	air
Specimen size and preparation	thin flat disk or rectangle—25-50-mm diameter and 10-40 mm thick depending on instrument design flat and parallel surfaces to ± 10 min and polished to 32 rms	thin flat disk or rectangle—6.5-13-mm diameter with thickness = diameter flat and parallel surfaces to ± 15 min and metallized and then soldered to the copper thermode references	thick flat slab—100-300 mm in length by 10-50 mm thick, depending on instrument configuration	thin flat disk or rectangle recommended—50-mm diameter by 0.5-12.7 mm thick, dependent on instrument configuration. flat and parallel faces with thickness ± 0.025 mm
Reference/standard materials	two reference specimens	copper reference specimens	calibration standard required	calibration standard required
Benefits and advantages	small sample size, high-temperature range, direct measurement against reference standards	small sample size	suitable for low thermal conductivity (<0.1 W/m·K) materials	smaller sample size than Test Method C 518
Test limitations and requirements	requires reference materials with suitable λ and stability at the test temperatures contact resistance should be minimized, especially for high λ materials.	limited temperature range contact resistance should be minimized, especially for high λ materials.	requires calibration with a reference standard of similar λ see Practice C 1132	requires calibration with a reference standard of similar λ . see Practice C 1132
Precision Data	± 7 % for 0-600°C	± 3 % for 0-150°C	± 5 %	± 5 %
Comments for advanced ceramic Monoliths	—	—	not suitable for high λ materials	not suitable for high λ materials
Comments for advanced ceramics composites	specimen dimensions should be 20× the dimensions of a “unit cell” or laminate thickness			

thermal equilibrium the measured heat flux, the temperature drop between the top and bottom faces of the specimen, and the specimen thickness are used to calculate the thermal conductivity of the test specimen. (See Test Methods C 518 and E 1530.) See Fig. 2.

11.1.3 Guarded-Hot-Plate Technique—Test specimens of fixed thickness are placed between a main heat source operating at a known power level and an auxiliary heater operating at a fixed temperature. Additional guard heaters are positioned to minimize lateral heat flow. Thermocouples measure the temperature differential across the test specimen thickness at thermal equilibrium. The hot-plate technique can be used in either a two-sided or a one-sided mode. The Thin-Heater Method is a geometrical variation of the guarded hot plate technique. (See Test Method C 177, Practice C 1044, and Test Method C 1114.) See Fig. 3.

11.1.4 Hot Wire Technique—In the hot-wire technique, a resistance heated platinum wire is sandwiched between two blocks of the test material in a temperature controlled chamber. A constant electrical current is applied to the wire. The rate at which the wire heats is dependent on how rapidly heat flows



Schematic of Differential Scanning Calorimeter
FIG. 7 Schematic of Differential Scanning Calorimeter

from the wire into the constant temperature mass of the test blocks. The rate of temperature increase of the wire is accurately determined by measuring its increase in resistance in the same way a platinum resistance thermocouple is used. A Fourier equation is used to calculate the thermal conductivity based on the rate of temperature increase of the hot wire and the power input. (See Test Method C 1113.) See Fig. 4.

11.1.5 Calorimetry Technique for Refractories—A refractory insulation specimen of known thickness is positioned in an

TABLE 4 Thermal Conductivity—Guarded Hot Plate, Hot Wire, and Calorimeter Methods

ASTM Test Method	C 177 and C 1044	C 1114	C 1113	C 182, C 201, C 202, C 767
Measurement technique	Guarded Hot Plate	Thin Heater	Hot Wire	Calorimeter
Thermal conductivity range, λ	0.01-2 W/(m·K)	0.01-2 W/(m·K)	0.5-15 W/(m·K)	<28 W/(m·K)
Test temperature range	–150 to 1000°C, depending on instrument configuration and thermal control range	–150 to 1000°C, depending on instrument configuration and thermal control range	40 to 1500°C, depending on instrument configuration and thermal control range and insulation range of test material	40 to 1500°C, depending on instrument configuration and furnace range
Specimen environment	commonly air, but inert/reducing gas, vacuum depending on system design			air
Specimen size and preparation	flat slab, generally 200-600-mm width and 10-150 mm thick depending on system configuration	flat slab, generally 200-600-mm width and 10-150 mm thick depending on system configuration	two bricks (9 by 4.5 by 3 in.) with a groove for the wire	six flat bricks – 228 by 64 by 57 mm flat and parallel to ± 0.3 mm
Reference/standard materials	none required	none required	none required	none required
Benefits and advantages	absolute measurement of thermal conductivity with no reference specimens	absolute measurement of thermal conductivity with no reference specimens	absolute measurement of thermal conductivity with no reference specimens	absolute measurement of thermal conductivity with no reference specimens
Test limitations and requirements	most suitable for low λ [<5 -W/(m·K)] insulators	most suitable for low λ [<5 -W/(m·K)] insulators	not applicable for electrically conductive ceramics	designed for refractory bricks and requires large specimens, not commonly used for advanced ceramic
Precision data	± 5 %	± 5 %	± 5 %	NA
Comments for advanced ceramic monoliths	limited application because of large sample size			very limited application because of large sample size
Comments for advanced ceramics composites	—	—	not recommended for anisotropic materials	very limited application because of large sample size

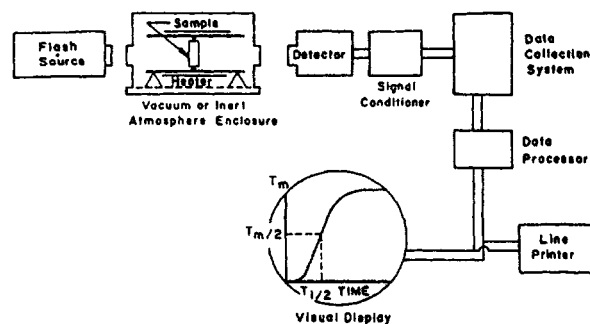
electrically heated furnace with the back side of the specimen positioned against a water-cooled calorimeter (a copper plate with flowing water). The inner and outer face of the refractory specimen are instrumented with thermocouples. The refractory test specimen is fixtured with insulating guard bricks to reduce lateral heat flow. With the furnace at the desired temperature and the calorimeter showing a steady-state heat flow, the heat flux flowing through the test specimen is determined from the measured heat flow and the specimen cross-sectional area. The thermal conductivity is then calculated from the heat flux and the temperature gradient across the specimen thickness. (See Test Methods C 182, C 201, C 202, and C 767.) See Fig. 5.

NOTE 1—The calorimetry technique has been superseded by simpler, less complex conductivity methods and is not commonly used for advanced ceramics, because of the large sample size and the mass and the cost of the test equipment.

11.1.6 General requirements and guidelines for determining the thermal transmission properties based upon heat flux measurements are given in Practice C 1045. Tables 3 and 4 give comparative information on the different ASTM test methods for thermal conductivity.

11.2 *Specific Heat Capacity Test Methods*—Specific heat capacity is measured in two ways in accordance with ASTM methods: drop calorimetry and scanning differential calorimetry.

11.2.1 *Drop Calorimetry*—A test specimen is heated to a specified temperature in a furnace and then rapidly inserted into an adiabatic calorimeter at a known temperature. When



Schematic of Flash Diffusivity Apparatus

FIG. 8 Schematic of Flash Diffusivity Apparatus

thermal equilibrium is reached between the test specimen and the calorimeter, the increase in thermal energy in the calorimeter is measured based on the temperature increase and the specific heat capacity of the calorimeter. The specific heat capacity of the sample is then calculated by dividing the measured increase in thermal energy of the calorimeter by the specimen temperature difference (initial specimen temperature – final specimen temperature) and the mass of the test specimen. (See Test Methods D 2766 and C 351.) See Fig. 6.

11.2.2 *Differential Scanning Calorimetry*—The test specimen is heated at a controlled rate in a controlled atmosphere through the temperature regime of interest. The heat flow into the test specimen is measured with a heat flux transducer and compared to the heat flow into a reference material or blank run

TABLE 5 Specific Heat—Scanning Differential and Drop Calorimetry Methods

ASTM Test Method	E 1269	D 2766	C 351
Measurement technique	Scanning Differential Calorimetry	Drop Calorimetry	Drop Calorimetry
Specific heat capacity, C_p , Range	no limit	no limit	no limit
Test temperature range	0 to 1000°C dependent on furnace range and specimen holder temperature limit	0 to 250°C depending on the furnace capability and specimen capsule limits	0 to 150°C depending on the boiling point of the calorimeter fluid and the furnace capability
Specimen environment	air, inert/reducing gas, vacuum depending on system design	air	air
Specimen size and preparation	1-500 mg, dependent on specimen holder volume samples must be dry, especially for powdered samples	dependent on instrument configuration and specimen capsule size	nominal—50 by 25 by 3 mm dependent on instrument configuration and test conditions
Reference/standard materials	reference specimen recommended	calibration standard required, copper or aluminum	calibration standard required, copper or aluminum
Benefits and advantages	rapid testing with data across a broad temperature range; easily identifies thermal transformations	convenient sample size for inhomogeneous materials	convenient sample size for inhomogeneous materials
Test limitations and requirements	small samples have to be homogeneous and representative	limited temperature range; determines linear mean value for C_p and requires multiple tests to determine C_p at different temperatures difficult to identify thermal transformations	limited temperature range; determines linear mean value for C_p and requires multiple tests to determine C_p at different temperatures difficult to identify thermal transformations
Precision data	± 2 %	NA	± 10 %
Comments for advanced ceramic monoliths	monitor samples for phase transformations and glass transitions		
Comments for advanced ceramics composites	small samples have to be homogeneous and representative		

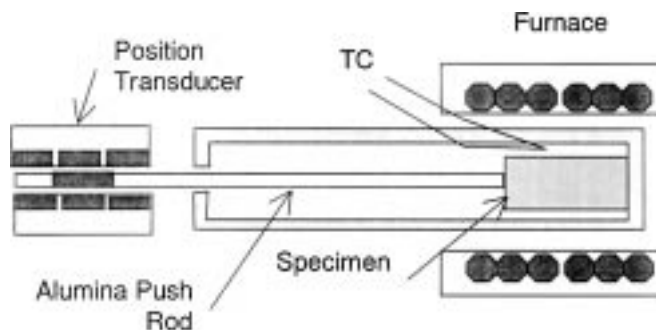
concurrently. The difference in heat flow is continually monitored and recorded as the test specimen is heated. The measured difference in heat flow across the temperature range of interest is used to calculate the specific heat capacity of the test specimen. (See Test Method E 1269.) See Fig. 7.

11.2.3 Table 5 gives comparative information on the different ASTM test methods for specific heat capacity.

11.3 Thermal Diffusivity Test Methods:

11.3.1 *Flash Diffusivity*—Thermal diffusivity is experimentally determined by measuring the temperature time response of a test specimen to a thermal event. The current method of choice is flash diffusivity, in which one side of a specimen of known thickness and temperature is subjected to a high-intensity short-duration thermal pulse. (The thermal pulse is introduced by a laser or flash lamps). The energy of the pulse is absorbed on the front surface of the specimen and the resulting rear face temperature rise is measured by a direct contact thermocouple or a noncontact pyrometer. The ambient temperature of the specimen is controlled by a furnace or cryostat.

11.3.2 Thermal diffusivity is calculated from the specimen thickness and the time required for the rear face temperature to reach a specified percentage of its maximum value. (See Test Methods C 714 and E 1461.) See Fig. 8.



Schematic of Thermodilatometry Method

FIG. 9 Schematic of Thermodilatometry Method

11.3.3 Table 6 gives comparative information on the different ASTM test methods for thermal diffusivity.

11.4 Thermal Expansion Test Methods:

11.4.1 Thermal expansion is commonly measured by thermodilatometry, a technique in which a specific, precisely measured dimension of a test specimen under negligible load is measured as a function of temperature while the substance is subjected to a controlled-temperature program in a specified atmosphere. The measurement of the dimensional change can

TABLE 6 Thermal Diffusivity by the Flash/Thermal Pulse Method

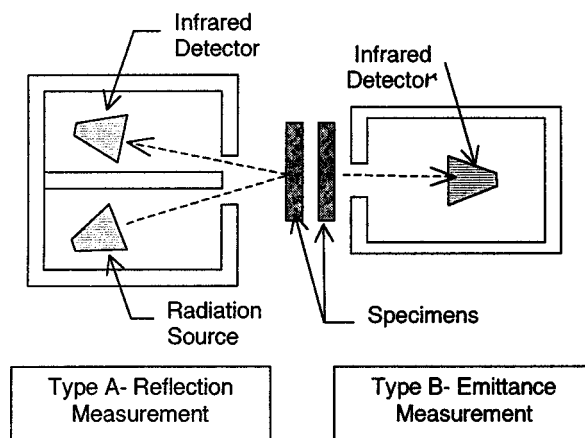
ASTM Test Method	E 1461	C 714
Measurement technique	Flash Method	Flash/Thermal Pulse Method
Thermal diffusivity range, m ² /s	0.1-1000 m ² /s	0.04-2.0 m ² /s
Test temperature range	–200 to 2000°C, depending on test chamber capability	20 to 500°C
Specimen environment	air, inert/reducing gas, vacuum depending on system design	air, inert or vacuum, depending on test chamber capability
Specimen size and preparation	thin circular disk, typically 6-18 mm in diameter with a thickness of 1.5-4 mm, depending on thermal properties of specimen thickness must be uniform to 0.5 %	thin circular disk, typically 6-12 mm in diameter with a thickness of 2-4 mm, thickness must be uniform to ±0.01 mm
Reference/standard materials	reference standards available, but not required for operation	
Benefits and advantages	small sample size and rapid testing across a range of temperatures	
Test limitations and requirements	<p>small samples have to be representative.</p> <p>Accurate thickness measurements are critical; surface finish may affect the absorption of the thermal radiation on the incident face.</p> <p>Calculation of thermal conductivity from thermal diffusivity requires specific heat and density at all the experimental temperatures.</p> <p>Translucency to laser radiation in the ceramic material will require a coating on one or both specimen surfaces with a calculation and adjustment for the effects of the coating on radiation absorption and thermal diffusivity.</p>	
Precision data	±5 %	±5 %
Comments for advanced ceramics monoliths	Translucency to laser radiation in the ceramic material will require a coating on one or both specimen surfaces with a calculation and adjustment for the effects of the coating on radiation absorption and thermal diffusivity.	
Comments for advanced ceramics composites	<p>Anisotropy effects must be carefully considered and analyzed to avoid misinterpretation of results.</p> <p>Surface skin effects (density and surface texture) may complicate the measurements.</p>	

be done by direct mechanical measurement or by optical measurement, using interferometers. See Fig. 9.

11.4.2 In mechanical measurement dilatometers, the specimen is positioned in a fixture with one end in contact with a rigid, spring-loaded push rod. The fixture and the push rod are made of materials with high-temperature stability and known thermal expansion. The opposite end of the push rod is fixtured into the position transducer (digital or linear variable differential transformer) which converts the linear motion of the push rod into a voltage signal. As the specimen in the fixture is heated in a controlled manner, the change in dimension of the sample is translated to the push rod and measured by the transducer as a function of time/temperature. A thermocouple is positioned close to the specimen to measure the specimen temperature.

11.4.3 Single-rod dilatometers require calibration against a standard specimen to account for the thermal expansion of the fixture. Dual-rod extensometers have two test positions; one position contains a known reference sample which acts as a direct standard for comparison to the specimen in the second position. Dilatometers can be designed with controlled atmosphere test chambers to operate under vacuum or inert gas atmospheres. (See Test Methods E 228, E 831, and C 372.)

11.4.4 With interferometric techniques, thermal expansion is measured by the use of laser or monochromatic light and a



Schematic of Reflectance and Emittance Methods

FIG. 10 Schematic of Reflectance and Emittance Methods

dual mirror system in which the specimen is positioned. The mirror system is designed so that fringe patterns are produced on a sensor by constructive interference of the light. Changes in the length of the specimen shorten or lengthen the light path and shift the fringe pattern. Fringe shifts are produced by sample dimension changes on the order of the wave length of

TABLE 7 Thermal Expansion by Therm dilatometry (Mechanical and Interferometry)

ASTM Test Method	E 228 and C 372	E 289 and C 1300	E 831
Measurement technique	Dilatometer	Interferometry	Dilatometer - Thermomechanical Analyzer
Mean coefficient of thermal expansion range, α_L	0.1 to 100 $10^{-6}/K$ depending on instrument sensitivity, specimen dimensions, and test temperature range	0.05 to 5 $10^{-6}/K$ depending on instrument sensitivity and specimen dimensions	5 to 100 $10^{-6}/K$ depending on instrument sensitivity and specimen dimensions
Test temperature range	–180 to 900°C for silica fixture; up to 2000°C for other fixture materials and depending on thermal chamber range	–150 to 1000°C depending on fixture materials and thermal chamber range	–120 to 600°C depending on fixture materials and thermal chamber range.
Specimen environment	air, inert/reducing gas, vacuum depending on system design	vacuum or low-pressure inert to avoid optical drift	air, inert/reducing gas, vacuum depending on system design
Specimen size and preparation	2-50 mm long cylinder or rod depending on instrument sensitivity, furnace uniformity, and specimen thermal expansion.	E 289—5-20-mm long cylinder or rod with 5-12-mm diameter with rounded ends.	2-10-mm long cylinder or rod depending on instrument sensitivity and specimen thermal expansion
	maximum diameter dependent on fixture size	C 1300—cones at least 6 mm high with a 3-mm base.	maximum diameter dependent on fixture size
	ends should have surface finish of 10 rms		ends flat and parallel to $\pm 25 \mu m$
	length measured to an accuracy of $\pm 0.002 \%$		
Reference/standard materials		calibration or reference standard required	
Benefits and advantages	small sample size and direct measurement	extra precision for specimens with very low thermal expansions.	smaller sample size than Test Method E 228
Test limitations	limited sensitivity for small specimens with $\alpha_L < 2 \times 10^{-6}/K$	not suitable for large specimens or materials with larger $\alpha_L (> 5 \times 10^{-6}/K)$ Specimens ends need to be precisely machined and flat.	limited high-temperature range; limited to specimens with α_L greater than 5 ppm/K
Precision	$\pm 1 \%$	NA	$\pm 2 \%$ for $\alpha_L > 20 \times 10^{-6}/K$ $\pm 15 \%$ for $\alpha_L < 5 \times 10^{-6}/K$
Comments for advanced ceramic monoliths	monitor samples for microcracking hysteresis, sintering, creep, and phase transformations.		
Comments for advanced ceramics composites	anisotropy effects must be carefully considered and analyzed to avoid misinterpretation of results.		

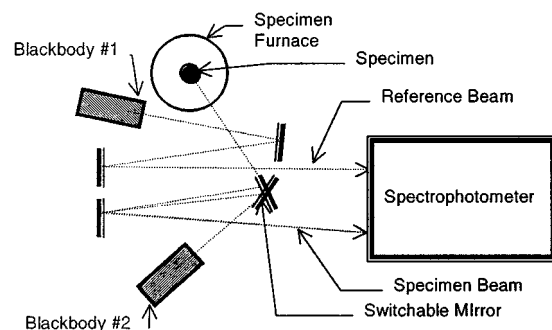
light (200 to 400 nm), depending on the specific wavelength of the light used. The specimen-mirror system is placed on a temperature-controlled furnace for high-temperature measurements. It is recommended that tests be done under vacuum or low-pressure helium to reduce atmospheric distortion along the light path. (See Test Methods C 1300 and E 289.)

11.4.5 Table 7 gives comparative information on the different ASTM test methods for thermal expansion

11.5 Emittance/Emissivity Test Methods:

11.5.1 There are two ASTM test methods used in the measurement of emittance of ceramics, E 408 and E 423. Both test methods measure the normal emittance, not the hemispherical emittance.

11.5.2 Test Methods E 408 describes two commercial systems for measuring radiant energy. Test Method A measures radiant energy reflected by the specimen from a radiant source in the test equipment. Test Method B measures radiant energy emitted by the specimen. The two systems are suitable for



Schematic of Spectrophotometer Emittance Method

FIG. 11 Schematic of Spectrophotometer Emittance Method

specimens at ambient temperatures and operate in the infrared

TABLE 8 Thermal Emittance/Emissivity

ASTM Test Method	E 408	E 423
Measurement technique	Test Method A—Reflected radiation measured by infrared detector and compared to a standard Test Method B—Emitted radiation measured by infrared detector and compared to a standard	spectrophotometer with high-temperature furnace and integral blackbody radiator
Emittance/emissivity range	0.05-1	0.05-1
Test temperature range	ambient temperature	1000 to 1800K depending on furnace capabilities
Specimen environment	ambient atmosphere; vacuum or inert atmosphere with special detector head and fittings to an atmosphere chamber	inert and air atmospheres, vacuum, depending on sample chamber
Specimen size and preparation	minimum size depends on the "spot size" of the optical system. No sample preparation required.	hollow cylinder 25 mm high and 25 mm in diameter with wall thickness sufficient to be opaque
Reference/standard materials	required for both reflection and emission methods	Blackbody standard is part of the test system.
Benefits and advantages	rapid testing; no special sample preparation	full-range capabilities across temperature and wavelength spectrum
Test limitations	limited temperature capability	complex furnace and measurement equipment
Precision	± 1 %	± 1 %
Comments for advanced ceramic monoliths	Surface texture may have a significant effect on emittance.	Surface texture may have a significant effect on emittance. Cylindrical test specimen is difficult to fabricate.
Comments for advanced ceramics composites	same as monoliths	same as monoliths

wavelength region of 4 to 50 μm , depending on the specific capabilities of the infrared detectors. Both systems require calibration against standards of known reflectance and emittance. See Fig. 10

11.5.3 Test Method E 423 describes a test method for measuring the emittance of ceramic specimens in a heated furnace across a range of temperatures with direct comparison to a blackbody reference at the same temperature. Special efforts are made to maintain the ceramic test specimen and the blackbody reference at the identical controlled and uniform

temperature. Radiant flux is measured with a spectrophotometer across the desired spectral range from 1 to 35 μm). See Fig. 11.

11.5.4 Table 8 gives comparative information on the different ASTM test methods for thermal emittance.

12. Keywords

12.1 advanced ceramic; ceramic composite; emissivity; emittance; specific heat capacity; thermal conductivity; thermal diffusivity; thermal expansion; thermal properties

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