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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 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 (monolithic and composite) is briefly outlined, providing guidance on which material and specimen characteristics have to be considered in evaluating the thermal properties of advanced ceramics. Additional sections describe sampling considerations, test specimen preparation, and reporting requirements.

1.3 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 monolithic and composite ceramics.

1.4 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. 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.

1.5 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard. See **IEEE/ASTM SI 10**.

1.6 *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, safety, health, and health environmental practices and determine the applicability of regulatory limitations prior to use.*

1.7 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 *ASTM Standards:*²

2.1.1 *Specific Heat:*

[C351 Test Method for Mean Specific Heat of Thermal Insulation](#) (Withdrawn 2008)³

[D2766 Test Method for Specific Heat of Liquids and Solids](#) (Withdrawn 2018)³

[E1269 Test Method for Determining Specific Heat Capacity by Differential Scanning Calorimetry](#)

[E2716 Test Method for Determining Specific Heat Capacity by Sinusoidal Modulated Temperature Differential Scanning Calorimetry](#)

2.1.2 *Thermal Conductivity:*

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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.

³ The last approved version of this historical standard is referenced on www.astm.org.

- C177 Test Method for Steady-State Heat Flux Measurements and Thermal Transmission Properties by Means of the Guarded-Hot-Plate Apparatus
- C182 Test Method for Thermal Conductivity of Insulating Firebrick
- C201 Test Method for Thermal Conductivity of Refractories
- C202 Test Method for Thermal Conductivity of Refractory Brick
- C408 Test Method for Thermal Conductivity of Whiteware Ceramics
- C518 Test Method for Steady-State Thermal Transmission Properties by Means of the Heat Flow Meter Apparatus
- C767 Test Method for Thermal Conductivity of Carbon Refractories
- C1044 Practice for Using a Guarded-Hot-Plate Apparatus or Thin-Heater Apparatus in the Single-Sided Mode
- C1045 Practice for Calculating Thermal Transmission Properties Under Steady-State Conditions
- ~~E1113~~C1113/C1113M Test Method for Thermal Conductivity of Refractories by Hot Wire (Platinum Resistance Thermometer Technique)
- C1114 Test Method for Steady-State Thermal Transmission Properties by Means of the Thin-Heater Apparatus
- C1130 Practice for Calibration of Thin Heat Flux Transducers
- E1225 Test Method for Thermal Conductivity of Solids Using the Guarded-Comparative-Longitudinal Heat Flow Technique
- E1530 Test Method for Evaluating the Resistance to Thermal Transmission by the Guarded Heat Flow Meter Technique
- 2.1.3 *Thermal Expansion:*
- C372 Test Method for Linear Thermal Expansion of Porcelain Enamel and Glaze Frits and Fired Ceramic Whiteware Products by the Dilatometer Method
- C1300 Test Method for Linear Thermal Expansion of Glaze Frits and Ceramic Whiteware Materials by the Interferometric Method
- E228 Test Method for Linear Thermal Expansion of Solid Materials With a Push-Rod Dilatometer
- E289 Test Method for Linear Thermal Expansion of Rigid Solids with Interferometry
- E831 Test Method for Linear Thermal Expansion of Solid Materials by Thermomechanical Analysis
- 2.1.4 *Thermal Diffusivity:*
- C714 Test Method for Thermal Diffusivity of Carbon and Graphite by Thermal Pulse Method
- D4612 Test Method for Calculating Thermal Diffusivity of Rock and Soil
- E1461 Test Method for Thermal Diffusivity by the Flash Method
- E2585 Practice for Thermal Diffusivity by the Flash Method
- 2.1.5 *Emittance/Emissivity:*
- E408 Test Methods for Total Normal Emittance of Surfaces Using Inspection-Meter Techniques
- E423 Test Method for Normal Spectral Emittance at Elevated Temperatures of Nonconducting Specimens
- 2.1.6 *General Standards:*
- C168 Terminology Relating to Thermal Insulation [ASTM C1470-20](#)
- C373 Test Methods for Determination of Water Absorption and Associated Properties by Vacuum Method for Pressed Ceramic Tiles and Glass Tiles and Boil Method for Extruded Ceramic Tiles and Non-tile Fired Ceramic Whiteware Products
- C1045 Practice for Calculating Thermal Transmission Properties Under Steady-State Conditions
- C1145 Terminology of Advanced Ceramics
- E122 Practice for Calculating Sample Size to Estimate, With Specified Precision, the Average for a Characteristic of a Lot or Process
- E473 Terminology Relating to Thermal Analysis and Rheology
- E1142 Terminology Relating to Thermophysical Properties
- ~~C1045 Practice for Calculating Thermal Transmission Properties Under Steady-State Conditions~~
- IEEE/ASTM SI 10 Standard for Use of the International System of Units (SI) (The Modern Metric System)American National Standard for Metric Practice

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. **(C1145)**

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. **(C1145)**

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 (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.}~~**(E1142)**

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. **(C1145)**

3.1.5 *differential scanning calorimetry (DSC)*, *n*—a technique in which the difference in energy inputs into a test specimen and a reference material is measured as a function of temperature while the test specimen and reference material are subjected to a controlled temperature program. **(E1269)**

3.1.6 *discontinuous fiber-reinforced ceramic composite*, *n*—a ceramic matrix composite reinforced by chopped fibers. **(C1145)**

3.1.7 *emittance (emissivity)*, ε (*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. **(E423)**

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 . **(E228)**

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). **(C372)**

3.1.10 ~~*particulate reinforced*~~ *particulate-reinforced ceramic matrix composite*, *n*—a ceramic matrix composite reinforced by ceramic particulates. **(C1145)**

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. **(E1142)**

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. **(EH42C168)**

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. **(E1461C168)**

3.1.14 *thermodilatometry*, *n*—a technique in which a dimension of a test specimen under negligible applied force is measured as a function of temperature while the test specimen is subjected to a controlled temperature program in a specified atmosphere. **(E473)**

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
Coefficient of 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.

[ASTM C1470-20](https://standards.iteh.ai/catalog/standards/sist/7bae6e5f-f763-476a-b4a1-c2b947874fa1/astm-c1470-20)

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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~~ 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-kelvin). As with other thermal properties, thermal conductivity changes with temperature, so that a specific 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~~ >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, cut bar comparative techniques, heat flow meter techniques, guarded hot-plate/heater/hot-wire techniques, and calorimetry techniques. It can also be determined by transient techniques (hot wire and flash diffusivity).

7.2.4 In cut bar comparative heat flow techniques, the test specimen is subjected to a known heat flow and the temperature differential/s are measured across the dimension/s of interest. The entire test system with the test specimen is configured with insulation and heaters to minimize heat flow perpendicular to the direction of interest. In the heat flowmeter technique, the heat flow in the test is measured by a calibrated heat flux transducer without the use of direct reference materials in the test system. 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 (λ) can be calculated from thermal diffusivity measurements, using the specific heat capacity and the material density as follows:

$$\lambda = \text{thermal diffusivity} \times \text{specific heat capacity} \times \text{density} \quad (2)$$

$$\lambda = \text{thermal diffusivity} \times \text{specific heat capacity} \times \text{density} \quad (2)$$

7.3 Thermal Expansion (Thermodilatometry):

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~~ 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:

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

where:

L_1 and L_2 = lengths of the test specimen at test temperatures T_1 and T_2 , respectively, where $T_2 > T_1$.

7.3.4 The units for mean coefficient of linear thermal expansion are ~~metres/(metre-K)~~ 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~~ 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 thermodilatometry, a technique in which a known dimension of a test specimen under negligible applied force 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 “specific heat capacity per unit volume” (which is the specific heat capacity ~~divided~~multiplied by the bulk density), as follows:

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

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

where:

λ = thermal conductivity,

C_p = specific heat capacity, 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~~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:

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

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

where:

F = ~~specimen temperature for hemispherical emittance~~

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)(>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~~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.

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.

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; Properties: Nonmetallic Solids*, Touloukian, Y. S. and DeWitt, D.P.; I.F. I. Plenum, D. P., IFI/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 capacity, [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 ⁻⁶	10.0	6.6	53.0	45.4	19.8	0.8	1.6	82.9	1.8
Thermal diffusivity, m ² /s × 10 ⁻⁶	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
Coefficient of linear thermal expansion, 10 ⁻⁶ /K	(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
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 (0–900 °C)	3.5 (0–500 °C)	NA	5.4 (20–1320 °C)	3.0 (100–1100 °C)	7.9 (100–1100 °C)	6.0 (100–1100 °C)	–1.4 at (23 °C)	–1.45 at (23 °C)
Coefficient of Linear Thermal Expansion, 10 ⁻⁶ /K	4.0 (0–900 °C)	3.5 (0–500 °C)	NA	5.4 (20–1320 °C)	3.0 (100–1100 °C)	7.9 (100–1100 °C)	6.0 (100–1100 °C)	–1.4 at (23 °C)	–1.45 at (23 °C)

^A CDI Ceramics, Inc., San Diego, CA.

^B 3M Corp., St. Paul, MN.

^C Amoco Performance Products, Alpharetta, GA.

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~~, 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~~(one-dimensional) (tows), two-dimensional (woven fabrics), and three-dimensional (weaves and ~~braid~~)](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.

ASTM C1470-20

<https://standards.iteh.ai/catalog/standards/sist/7bae6e5f-f763-476a-b4a1-c2b947874fa1/astm-c1470-20>

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 **E122** 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. Use Practice E122 as a guide, if specimen counts are not specified.

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 Per Test Method C372, test specimens for mechanical dilatometry should have ends that are cut/ground flat and perpendicular to the specimen axis. Dilatometer fixtures are commonly designed to provide point contact with specimens and to prevent sideways levering. For thermal expansion measurements using optical test 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~~ 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~~ silicon-based ceramics (for example, silicon carbide or silicon nitride) or oxide ceramics containing a glassy phase (for example, aluminas with second ~~phases~~ phases), annealing at $\sim 1200^{\circ}\text{C}$ $\sim 1200^{\circ}\text{C}$ 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 ~~Archimedes~~' immersion (Test Method C373) where necessary, especially for composites with significant porosity or surface roughness.

9.8 *Post-Test 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.