**Designation: C781 − 20**

# Standard Practice for
# Testing Graphite Materials for Gas-Cooled Nuclear Reactor Components[1]

This standard is issued under the fixed designation C781; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

## 1. Scope*

1.1 This practice covers the application and limitations of test methods for measuring the properties of graphite materials. These properties may be used for the design and evaluation of gas-cooled reactor components.

1.2 The test methods referenced herein are applicable to materials used for replaceable and permanent components as defined in Section 7 and includes fuel elements; removable reflector elements and blocks; permanent side reflector elements and blocks; core support pedestals and elements; control rod, reserve shutdown, and burnable poison compacts; and neutron shield material. Specific aspects with respect to testing of irradiated materials are addressed.

1.3 This practice includes test methods that have been selected from ASTM standards and guides that are specific to the testing of materials listed in 1.2. Comments on individual test methods for graphite components are given in Section 8. The test methods are summarized in Table 1.

1.4 The values stated in SI units are to be regarded as standard. The values given in parentheses after SI units are provided for information only and are not considered standard.

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

1.6 *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]

C559 Test Method for Bulk Density by Physical Measurements of Manufactured Carbon and Graphite Articles
C577 Test Method for Permeability of Refractories
C611 Test Method for Electrical Resistivity of Manufactured Carbon and Graphite Articles at Room Temperature
C625 Practice for Reporting Irradiation Results on Graphite
C651 Test Method for Flexural Strength of Manufactured Carbon and Graphite Articles Using Four-Point Loading at Room Temperature
C695 Test Method for Compressive Strength of Carbon and Graphite
C747 Test Method for Moduli of Elasticity and Fundamental Frequencies of Carbon and Graphite Materials by Sonic Resonance
C749 Test Method for Tensile Stress-Strain of Carbon and Graphite
C769 Test Method for Sonic Velocity in Manufactured Carbon and Graphite Materials for Use in Obtaining an Approximate Value of Young's Modulus
C816 Test Method for Sulfur Content in Graphite by Combustion-Iodometric Titration Method
C838 Test Method for Bulk Density of As-Manufactured Carbon and Graphite Shapes
C1039 Test Methods for Apparent Porosity, Apparent Specific Gravity, and Bulk Density of Graphite Electrodes
C1179 Test Method for Oxidation Mass Loss of Manufactured Carbon and Graphite Materials in Air
C1233 Practice for Determining Equivalent Boron Contents of Nuclear Materials
C1274 Test Method for Advanced Ceramic Specific Surface Area by Physical Adsorption
D346 Practice for Collection and Preparation of Coke Samples for Laboratory Analysis
D1193 Specification for Reagent Water
D2854 Test Method for Apparent Density of Activated Carbon

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[1] This practice is under the jurisdiction of ASTM Committee D02 on Petroleum Products, Liquid Fuels, and Lubricants and is the direct responsibility of Subcommittee D02.F0 on Manufactured Carbon and Graphite Products.

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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.

***A Summary of Changes section appears at the end of this standard**

TABLE 1 Summary of Test Methods for Graphite Components

NOTE 1—Graphite Components include: Fuel, Removable Reflector and Core Support Elements; Pebble Bed Reflector, Key and Sleeves and Dowel Pins, Permanent Side Reflector Elements and Dowel Pins, Core Support Pedestals and Dowels.

| | Test Method |
|---|---|
| Fabrication | |
| As Manufactured Bulk Density | C838 |
| Mechanical Properties | |
| Compressive Strength | C695 |
| Tensile Properties | C749 |
| Poisson's Ratio | E132, C747 |
| Flexural Strength | C651, D7972 |
| Fracture Toughness | D7779 |
| Modulus of Elasticity | C747, C769 |
| Physical Properties | |
| Bulk Density–Machined Specimens | C559 |
| Surface Area (BET) | C1274 |
| Permeability | C577[A,B] |
| Apparent Porosity | C1039 |
| Spectroscopic Analysis | [B] |
| Electrical Resistivity | C611 |
| Thermal Properties | |
| Linear Thermal Expansion | E228[A] |
| Thermal Conductivity | E1461[A] |
| Chemical Properties | |
| Oxidative Mass Loss | C1179, D7542 |
| Sulfur Concentration | C816 |
| Equivalent Boron Content[C] | C1233[A] |

[A] Modification of this test method is required. See Section 8 for details.
[B] New test methods are required. See Section 8 for details.
[C] There is no identified need for determining this property for core support pedestals and dowels.

D2862 Test Method for Particle Size Distribution of Granular Activated Carbon
D3104 Test Method for Softening Point of Pitches (Mettler Softening Point Method)
D4175 Terminology Relating to Petroleum Products, Liquid Fuels, and Lubricants
D4292 Test Method for Determination of Vibrated Bulk Density of Calcined Petroleum Coke
D5600 Test Method for Trace Metals in Petroleum Coke by Inductively Coupled Plasma Atomic Emission Spectrometry (ICP-AES)
D7219 Specification for Isotropic and Near-isotropic Nuclear Graphites
D7542 Test Method for Air Oxidation of Carbon and Graphite in the Kinetic Regime
D7775 Guide for Measurements on Small Graphite Specimens
D7779 Test Method for Determination of Fracture Toughness of Graphite at Ambient Temperature
D7846 Practice for Reporting Uniaxial Strength Data and Estimating Weibull Distribution Parameters for Advanced Graphites
D7972 Test Method for Flexural Strength of Manufactured Carbon and Graphite Articles Using Three-Point Loading at Room Temperature
D8186 Test Method for Measurement of Impurities in Graphite by Electrothermal Vaporization Inductively Coupled Plasma Optical Emission Spectrometry (ETV-ICP OES)
E11 Specification for Woven Wire Test Sieve Cloth and Test Sieves
E132 Test Method for Poisson's Ratio at Room Temperature
E228 Test Method for Linear Thermal Expansion of Solid Materials With a Push-Rod Dilatometer
E261 Practice for Determining Neutron Fluence, Fluence Rate, and Spectra by Radioactivation Techniques
E639 Test Method for Measuring Total-Radiance Temperature of Heated Surfaces Using a Radiation Pyrometer (Withdrawn 2011)[3]
E1461 Test Method for Thermal Diffusivity by the Flash Method
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

## 3. Terminology

3.1 *Definitions*—Terminology D4175 shall be considered as applying to the terms used in this practice.

## 4. Significance and Use

4.1 Property data obtained with the recommended test methods identified herein may be used for research and development, design, manufacturing control, specifications, performance evaluation, and regulatory statutes pertaining to nuclear reactors that utilize graphite.

4.2 The referenced test methods are applicable primarily to specimens in the non-irradiated and non-oxidized state. Testing irradiated specimens often requires specimen geometries that

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

do not meet the requirements of the standard. Specific instructions or recommendations with respect to testing nonconforming geometries can be found in STP 1578[4] and/or Guide D7775. When testing irradiated specimens at elevated temperatures, the effects of annealing should be considered (see Note 1).

NOTE 1—Exposure to fast neutron radiation will result in atomic and microstructural changes to graphite. This radiation damage occurs when energetic particles, such as fast neutrons, impinge on the crystal lattice and displace carbon atoms from their equilibrium positions, creating a lattice vacancy and an interstitial carbon atom. The lattice strain that results from displacement damage causes significant structural and property changes in the graphite and is a function of the irradiation temperature and dose. When the temperature of the graphite is brought above the temperature at which it was irradiated, enough energy is provided that the structure of the graphite will anneal back to its original condition. Therefore, measurement techniques that bring the specimen temperature above the irradiation temperature can result in property values that change during the measurement process. For this reason, measurements made on irradiated test specimens below the irradiation temperature will produce results that are representative of the irradiation damage. However, measurements made at temperatures above the irradiation temperature could include the effects of annealing.

4.3 Additional test methods are in preparation and will be incorporated. The user is cautioned to employ the latest revision.

## 5. Sample Selection

5.1 All test specimens should be selected from materials that are representative of those to be used in the intended application.

## 6. Test Reports

6.1 Test results should be reported in accordance with the reporting requirements included in the applicable test method. Where relevant, information on grade designation, lot number, billet number, orientation, and location (position of sample in the original billet) shall be provided.

6.2 Information on specimen irradiation conditions shall be reported in accordance with Practices C625 and E261 or referenced to source information of equivalent content.

## GRAPHITE COMPONENTS

## 7. Description and Function

7.1 The following are identified as typical components of a graphite moderated gas-cooled reactor based on historical designs. This list is not intended to be inclusive of all possible components, which will depend upon the particular reactor design.

7.2 *Fuel and Removable Reflector Elements:*

7.2.1 In manufactured carbons and graphites, a fuel element is a removable graphite element that contains channels for the passage of coolant gas, the fuel material (typically in the form of a compact containing coated particle fuel), the alignment dowel pins, and for the insertion of a handling machine pickup head. A fuel element may also contain channels for reactivity control material (control rods), reserve shutdown compacts, and burnable poison compacts, and nuclear instrumentation.

7.2.2 The fuel elements serve multiple functions, including *(1)* vertical and lateral mechanical support for the fuel elements and removable reflector elements above and adjacent to them, and for the fuel, reactivity control materials, and nuclear instrumentation within them, *(2)* moderation of fast neutrons within the core region, *(3)* a thermal reservoir and conductor for nuclear heat generated in the fuel, *(4)* a physical constraint for the flow of coolant gases, and *(5)* a guide for and containment of fuel material, reactivity control materials, and nuclear instrumentation.

7.2.3 A removable reflector element is a removable graphite element that contains channels for the alignment dowel pins and the insertion of a handling machine pickup head. A removable reflector element may also contain channels for the passage of coolant gas, reactivity control materials (control rods), neutron flux control materials (neutron shield materials), and nuclear instrumentation.

7.2.4 The primary function of the removable reflector elements that are located at the boundaries of the active reactor core (fuel elements) is to provide moderation of fast neutrons escaping from and reflection of thermal neutrons back into the active core region.

7.2.5 Except for support, guide, and containment of fuel material, removable reflector elements may also serve any of the functions listed in 7.2.2.

7.3 *Permanent Side Reflector Element:*

7.3.1 A permanent side reflector element is a graphite block that is designed to remain permanently in the core but may be removed for inspection and replacement, if necessary. A permanent side reflector element contains channels for alignment dowel pins. It may also contain channels for neutron flux control materials (boronated steel pins) and nuclear instrumentation, and recessed areas along its length on its outer periphery to provide channels for the passage of coolant gas between the element and the metallic lateral restraint for the reactor core.

7.3.2 The permanent side reflector elements encircle the active (fuel) elements and passive (removable reflector) elements of the reactor core and serve multiple functions, including *(1)* vertical and lateral mechanical support for the permanent side reflector elements above and beside them, *(2)* lateral mechanical support for the fuel, removable reflector, and core support elements, *(3)* moderation of fast neutrons within the reflector region, *(4)* reflection of thermal neutrons back into the core region, and *(5)* support, guide, and containment of nuclear instrumentation and neutron flux control materials (boronated steel pins) for reducing the neutron flux to metallic structures outside the permanent side reflector boundary.

7.4 *Core Support Pedestals and Elements:*

7.4.1 A core support pedestal is a graphite column that is designed to remain permanently in the core but can be removed for inspection and replacement, if necessary. A core support pedestal has a central reduced cross-section (dog bone shape) that at its upper end contains channels for the passage of

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[4] Tzelepi, N. and Carroll, M., Eds., *Graphite Testing for Nuclear Applications: The Significance of Test Specimen Volume and Geometry and the Statistical Significance of Test Specimen Population*, STP1578-EB, ASTM International, West Conshohocken, PA, 2014, https://doi.org/10.1520/STP1578-EB

coolant gas, alignment dowel pins, and for the insertion of a handling machine pickup head, and at its lower end contains a recessed region for locating it with respect to the metallic structure that supports the graphite core support assembly. A core support element is a graphite element that contains channels for alignment dowel pins and for the insertion of a handling machine pickup head. The core support elements may also contain channels for the passage of coolant gas, neutron flux control materials, and nuclear instrumentation.

7.4.2 The primary function of the core support pedestals is to provide for vertical mechanical support for core support elements and permanent side reflector elements above them. In addition, core support pedestals provide for lateral mechanical support for adjacent core support pedestals and permanent side reflector elements and physical constraint for the flow of coolant gases. The primary function of the core support elements is to provide for vertical mechanical support for core support, fuel, and removable reflector elements above them. In addition, core support elements provide for lateral mechanical support for adjacent core support and permanent side reflector elements and may provide for the physical constraint of coolant gases and for the support, guide, and containment of neutron flux control materials and nuclear instrumentation.

7.5 *Pebble Bed Modular Reactor Reflector Blocks:*

7.5.1 The fuel form of a pebble bed reactor is typically a 60 mm diameter sphere (pebble) containing graphite-carbon matrix and coated particle fuel.

7.5.2 The Pebble Bed reactor core structure consists of a graphite reflector supported and surrounded by a metallic core barrel. The graphite reflector is comprised of a large number of graphite blocks arranged in circular rings of separate columns. The graphite reflector can be subdivided into three subsystems, namely, the bottom, side, and top reflector. The side reflector may be split into an inner replaceable reflector and an outer permanent reflector. The graphite reflector blocks are interlinked within each circular ring by graphite keys set in machined channels in the reflector blocks. Certain Pebble Bed reactors designs have annular fuelled cores, and thus the reactor contains a central graphite column.

7.5.3 The primary function of the reflector blocks that are located at the boundary of the active reactor core (fuelled region) is to provide for moderation of fast neutrons escaping from, and reflection of thermal neutrons back into, the active core region.

7.5.4 Replaceable reflector blocks contain vertical channels for the reactivity control rods and reserve shutdown system. These channels contain graphite sleeves to eliminate cross flow of reactor coolant gas.

## 8. Test Methods

8.1 *Fabrication:*

8.1.1 *Coefficient of Thermal Expansion of Coke*—The method known as the flour-based graphitized rod CTE test is described in Annex A1.

8.1.2 *Bulk Density*—Determine bulk density on as-manufactured or machined specimens in accordance with Test Methods C838 and C559, respectively. Test Method C838 includes shaped articles other than right circular cylinders and rectangular parallelepipeds. Test Method C559 is used when a higher degree of accuracy is required. The procedures of Test Method C559 are modified in Annex A2 to provide for the measurement of bulk density of non-uniform specimens.

8.1.3 *Graphitization Temperature*—The graphitization temperature of a full-size billet is estimated from a laboratory correlation between Specific Electrical Resistivity (SER) (Test Method C611) and heat treatment temperature. The method is described in Annex A3.

8.2 *Mechanical Properties:*

8.2.1 *Compressive Strength*—Determine compressive strength in accordance with Test Method C695.

8.2.2 *Tensile Strength*—Determine tensile strength in accordance with Test Method C749 and Guide D7775.

8.2.3 *Flexural Strength*—Determine flexural strength in accordance with Test Method C651 or D7972.

8.2.4 *Fracture Toughness*—Determine fracture toughness in accordance with Test Method D7779.

8.2.5 *Modulus of Elasticity and Poisson's Ratio*—Determine modulus of elasticity in accordance with Test Method C747. Poisson's ratio can be determined using Test Method E132. Sonic velocity (Test Method C769) may be used to give an approximate Young's Modulus.

8.3 *Physical Properties:*

8.3.1 *Bulk Density*—See 8.1.2.

8.3.2 *Surface Area*—The determination of the specific surface area (BET) shall be in accordance with Test Method C1274.

8.3.3 *Gaseous Permeability*—Test Method C577 for measuring gaseous permeability must be modified to permit the additional use of helium as the permeating medium and the use of alternative geometries for specimens and specimen holders.

8.3.4 *Apparent Porosity*—The determination of the apparent porosity shall be in accordance with Test Method C1039.

8.4 *Thermal Properties:*

8.4.1 *Coefficient of Thermal Expansion of Graphite*—Determine the linear coefficient of thermal expansion (CTE) of graphite of all grain sizes in (general) accordance with Test Method E228. Test specimens of cylindrical or prismatic geometry shall be used. The diameter or transverse-edge length, respectively, shall be no less than five times the maximum grain size of the graphite, and in no case smaller than 4 mm. The length of the test specimen shall be at least 25 mm, preferably 50 mm to 125 mm. The report shall include the temperature range over which the CTE was measured.

8.4.2 *Thermal Conductivity*—Calculate the thermal conductivity from the thermal diffusivity as determined by Test Method E1461. The required calculation is described in Annex A4.

8.5 *Chemical Properties:*

8.5.1 *Oxidation*—Determine the oxidative mass loss in air in accordance with Test Method C1179. (A test method for the determination of oxidation rate in air is in preparation.)

8.5.2 *Chemical Impurities:*

8.5.2.1 The chemical impurities shall be measured in accordance with D5600 or D8186.

8.5.2.2 Determine sulfur concentration in accordance with Test Method C816.

8.5.2.3 A method for determining boron levels is described in Annex A5.

8.5.3 *Equivalent Boron Content*—Test Method C1233 shall be used to calculate equivalent boron content. The elements specified in D7219 shall be measured for the determination of the equivalent boron content.

## 9. Keywords

9.1 chemical properties; graphite; gas-cooled nuclear reactors; mechanical properties; neutronic properties; physical properties; thermal properties

# ANNEXES

### (Mandatory Information)

## A1. QUALIFICATION CTE TEST FOR CALCINED COKE

A1.1 *Scope*—This method is applicable to the manufacture of graphite test rods from calcined petroleum or coal tar pitch coke of any origin.

### A1.2 Sampling

A1.2.1 Coke samples that are submitted for testing shall properly represent those lots, barges, railcars, or trucks which are received by the manufacturing locations.

A1.2.2 The coke sample shall be collected in accordance with Practice D346.

A1.2.3 Approximately 0.5 kg of calcined coke shall be riffled from a larger sample.

### A1.3 Procedure

A1.3.1 *Preparation of Green Test Specimen*—The sample of calcined coke shall be split into equal parts and one half retained for possible recheck. The other half is dried at 110 °C for 2 h and then crushed in one cycle to pass through a U.S. Standard 6.35 mm screen. The crushed sample is milled to flour so that at least 95 % passes a U.S. Standard No. 40 screen, and 40 % to 60 % passes a U.S. Standard No. 200 screen. Then appropriate quantities of the flour and a suitable medium coal tar pitch binder (nominal softening point 110 °C according to Test Method D3104, crushed to pass a U.S. Standard No. 10 screen) are heated to about 150 °C in a suitable laboratory scale mixer with occasional stirring. An extrusion aid may be added and mixed thoroughly. The mixture is then cooled or placed directly into a suitable heated laboratory scale extrusion press and tamped prior to extrusion. The internal diameter of the laboratory press may be 38 mm to 50 mm. The quantity of mix is sufficient when extruded to produce three test specimens 12 mm to 20 mm in diameter and 100 mm to 150 mm in length. The first test specimen extruded is discarded.

A1.3.2 *Baking*—The duplicate green specimens are packed without touching in a suitable sagger in bed of graphite particles or bed of coke and sand mixture (the pack passes a U.S. Standard No. 10 screen) then covered with about 50 mm of the same packing media. The sagger is placed into a furnace at 100 °C and heated at about 90 °C ⁄h to 120 °C ⁄h to 850 °C to 900 °C and held for 1 h to 3 h. The sagger shall be furnace cooled to less than 300 °C before opening and unpacking the rods. The rods may be cleaned using coarse sandpaper if required.

A1.3.3 *Graphitizing*—The baked specimens are placed loosely in a graphite capsule and heated at approximately 15 °C ⁄min to a temperature above 2700 °C and held for 30 min. Graphitization shall be conducted under flowing argon and the capsule shall be cooled to less than 300 °C before removing the rods.

A1.4 *Measuring the Coefficient of Thermal Expansion*—The rods will be cleaned and machined so that the end faces are parallel and normal to the longitudinal axis. The bulk density shall be measured according to Test Method C559, and the resistivity measured according to Test Method C611, and the values recorded. The density and resistivity shall be evaluated to ensure proper processing was achieved. The coefficient of thermal expansion (CTE) of the test specimens shall be measured along the longitudinal axis of the rod between room temperature and 500 °C in accordance with Test Method E228 and 8.4.1. The average of a single determination on each of the two specimens shall be recorded. A variation of the method involves measurement of the CTE at the baked stage and the use of an empirical relationship to estimate the CTE of the graphite.

A1.5 *Report*—The average CTE of the individual rods shall be reported in µm/m·°C (25 °C to 500 °C). The averages of rod bulk density and resistivity shall also be reported.

A1.6 *Precision and Bias*—No precision statement has been determined for this test method.

## A2. BULK DENSITY OF NON-UNIFORM TEST SPECIMENS

A2.1 The bulk density of test specimens other than right circular cylinders or rectangular parallelepipeds may be determined using Test Method C559 provided the specimen volume can be determined within 0.15 %. (See 6.2 of Test Method C559.)

A2.2 The net volume of a nonuniform, axisymmetric test specimen can be calculated if the shape can be broken down into simple geometric elements. Element volumes can be calculated with the aid of mensuration tables generally found in math and engineering handbooks. Sum the element volumes to obtain the net volume of the test specimen. Calculate the bulk density as in 7.3 of Test Method C559.

## A3. ESTIMATION OF THE GRAPHITIZATION TEMPERATURE OF FULL-SIZE BILLETS FROM THE MEASURED SPECIFIC ELECTRICAL RESISTIVITY

A3.1 The estimate of the graphitization temperature of a full-size billet is determined from a laboratory correlation between heat treatment temperature and the specific electrical resistivity (SER) of small specimens.

### A3.2 Procedure

A3.2.1 Samples of appropriate dimensions as described in Test Method C611 shall be cut from a full sized billet that has completed all processing steps prior to graphitization. The SER and bulk density of the cut samples shall be measured, using Test Methods C611 and C559, respectively, and reported along with the sampling diagram. At least three samples at each temperature shall be heat treated under controlled laboratory conditions at a minimum of five temperatures covering the range 2400 °C to 3100 °C. A heating rate of 15 °C⁄min shall be used. The samples shall be held at the heat treatment temperature for a minimum of 30 min. The SER of the heat treated specimens shall be determined at room temperature in accordance with Test Method C611. An SER versus temperature correlation curve shall be developed and a value shall be established that represents the required full-size billet graphitization temperature, typically 2700 °C. Errors in measurement will be determined from the scatter in the SER versus temperature plot. Typically, the temperature is monitored using an optical pyrometer calibrated by Test Method E639. Note, a new SER value must be established if changes are made in raw materials, mix formulation, or processing procedures.

A3.2.2 The SER of each full-size graphite billet shall be measured at room temperature using a standard procedure developed by the supplier and approved by the purchaser. The value of SER determined on each billet shall be used to establish the graphitization temperature of that billet using the laboratory correlation.

A3.2.3 The SER derived graphitization temperature of each billet shall be reported to the purchaser.

A3.3 *Precision and Bias*—No precision statement has been determined for this test method.

## A4. SPECIFIC HEAT CAPACITY OF GRAPHITE AND THE CALCULATION OF THERMAL CONDUCTIVITY FROM MEASUREMENTS OF THERMAL DIFFUSIVITY

A4.1 The thermal conductivity may be calculated from the measurements of thermal diffusivity with the following equation:

$$\lambda = \alpha C_p \rho$$

where:

$\lambda$ = thermal conductivity, W/m·K,
$\alpha$ = thermal diffusivity,
$\rho$ = bulk density, and
$C_p$ = specific heat capacity.

A4.2 The bulk density of polycrystalline graphite at room temperature can be measured by applying 8.1.2 of this practice. If the thermal conductivity is desired at elevated temperatures, it is important to consider how both the density and the specific heat change with temperature. The thermal expansion of graphite can result in decreasing its density by as much as 5 % between 300 K and 3000 K. Thus, it may be necessary to predict density change as a function of temperature using measured values of the coefficient of thermal expansion (CTE). Refer to 8.4.1 of this practice for guidance on measuring CTE.

A4.3 Heat capacity is a measurable physical quantity equal to the ratio of the heat added to the resulting temperature change of a substance. By dividing the heat capacity by the mass of the substance, the resulting specific heat capacity is an intrinsic property of the substance and independent of its mass. In SI units, specific heat capacity is the amount of energy in Joules needed to raise the temperature of one kilogram of the substance one degree Kelvin.

A4.3.1 The energy absorbed by a substance as it increases in temperature is primarily stored in the movement of the atoms and molecules and depends on the number of degrees of freedom that are available to those atoms and molecules in the substance. The larger the number of degrees of freedom available to the particles of a substance, the more energy can be stored and the higher the specific heat capacity is for that substance. Translation, rotation, and vibration of atoms or molecules represent the degrees of freedom of motion which classically contribute to the heat capacity of gases, while only lattice vibrations are needed to describe the heat capacities of most solids.

A4.3.2 Graphite is the crystalline form of carbon. It has a layered, planar structure with the carbon atoms arranged in a hexagonal lattice. Energy storage in a single crystal of graphite is a combination of vibrations in the planer covalent hexagonal bonds and weaker interlayer van der Waals bonds. Nuclear grade graphite is a polycrystalline material, that is, it is made up of randomly oriented crystallites of various sizes. Energy storage in polycrystalline graphite is the same as in a single crystal with the addition of work done against frictional forces in the relative crystallite movement, and changes in surface energy. The contribution due to work done to overcome frictional forces is thought to be relatively small due to the low CTE of graphite. The changes in surface energy are also thought to be insignificant due to the ratio of surface atoms to bulk atoms.[5]

A4.3.3 Therefore, the energy storage of graphite is primarily the lattice vibrational modes of the covalent and van der Waals bonds. Because this is an atomic level mechanism, all graphite that has been "fully graphitized" or is fully crystalline will have the same specific heat capacity. The density, grain size, coke source, and so forth, will have no effect on the specific heat capacity.

A4.3.4 If the graphite is not fully crystalline for any reason, such as a low graphitization processing temperature or extreme irradiation damage, it is possible for the specific heat to deviate from that represented below. For graphites that are suspected of not being fully graphitized, measurements of the specific heat capacity should be made for use in the calculation of thermal conductivity from thermal diffusivity using Test Methods E1269 or E2716.

A4.3.4.1 There are three techniques used for determining specific heat capacity. The most basic technique consists of heating a well known quantity of graphite using an accurately determined amount of energy and measuring the temperature rise of the sample. Simple water calorimetry is also used. In this case, a known mass of graphite is heated and then submerged into a known mass of water. The change in temperature of both the graphite and water are measured. If it is assumed that 100 % of the energy from the graphite is transferred to the water, the specific heat of the graphite can be calculated. The most modern technique used is differential scanning calorimetry (DSC). In this technique, the difference in the amount of heat required to increase the temperature of a sample and a reference is measured as a function of temperature. If the heat capacity of the reference sample is well defined over the range of temperatures scanned, it is possible to calculate the heat capacity of the unknown sample, graphite in this case, as a function of temperature.

A4.4 Historical specific heat capacity data for poly crystalline synthetic graphite between the temperatures of 250 K and 3000 K are shown in Fig. A4.1. The most recently evaluated compilation of data is from NIST-JANAF Thermophysical Tables by Chase.[6] No polynomial fit was provided with this data. The data and polynomial labeled "NBS" are from an interlaboratory study performed by the National Bureau of Standards (NBS).[7] In this study, Hurst derived the following

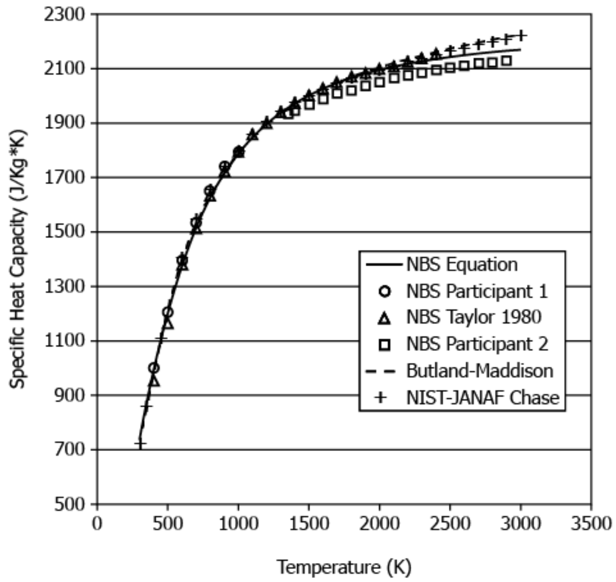

**FIG. A4.1 Specific Heat Capacity of Polycrystalline Graphite**

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[5] Butland, A. T. D. and Maddison, R. J., "The Specific Heat of Graphite: An Evaluation of Measurements," *J. of Nuc. Mat.*, Vol 49, North Holland Publishing Co., 1973/74.

[6] Chase, M. W., Jr., NIST-JANAF Thermochemical Tables, 4th ed., *J. Phys. Chem. Ref. Data*, Vol 9, 1998.

[7] Hust, J. G., Standard Reference Materials: A Fine-Grained, Isotropic Graphite for Use as NBS Thermophysical Property RM's from 5 to 2500K, *NBS Special Publication 260-89*, Boulder, CO, 1984.