

Standard Practice for Testing Graphite and Boronated Graphite Components for High-Temperature Gas-Cooled Nuclear Reactors¹

This standard is issued under the fixed designation C 781; 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 test methods for measuring those properties of graphite and boronated graphite materials that may be used for the design and evaluation of hightemperature gas-cooled reactors.

1.2 The test methods referenced herein are applicable to materials used for replaceable and permanent components as defined in Section 7 and include 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.

1.3 This practice includes test methods that have been selected from existing ASTM standards, ASTM standards that have been modified, and new ASTM standards that are specific to the testing of materials listed in 1.2. Comments on individual test methods for graphite and boronated graphite components are given in Sections 8 and 10, respectively. The test methods are summarized in Table 1 and Table 2.

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:

- C 559 Test Method for Bulk Density by Physical Measurements of Manufactured Carbon and Graphite Articles²
- C 560 Test Methods for Chemical Analysis of Graphite²
- C 561 Test Method for Ash in a Graphite Sample²
- C 577 Test Method for Permeability of Refractories³
- C 625 Practice for Reporting Irradiation Results on Graphite 2
- C 626 Methods for Estimating the Thermal Neutron Ab-

sorption Cross Section of Nuclear Graphite⁴

- C 651 Test Method for Flexural Strength of Manufactured Carbon and Graphite Articles Using Four-Point Loading at Room Temperature²
- C 695 Test Method for Compressive Strength of Carbon and Graphite²
- C 709 Terminology Relating to Manufactured Carbon and Graphite²
- C 747 Test Method for Moduli of Elasticity and Fundamental Frequencies of Carbon and Graphite Materials by Sonic Resonance²
- C 749 Test Method for Tensile Stress-Strain of Carbon and Graphite²
- C 816 Test Method for Sulfur in Graphite by Combustion-Iodometric Titration Method 2
- C 838 Test Method for Bulk Density of As-Manufactured Carbon and Graphite Shapes²
- C 1179 Test Method for Oxidative Mass Loss of Manufactured Carbon and Graphite Materials in Air²
- C 1251 Test Method for Determination of Specific Surface Areas of Advanced Ceramic Materials by Gas Adsorption²
- D 2854 Test Method for Apparent Density of Activated $\ensuremath{\mathsf{Carbon}}^2$
- D 2862 Test Method for Particle Size Distribution of Granular Activated Carbon²
- D 4292 Test Method for Vibrated Bulk Density of Calcined Petroleum Coke⁵
- E 132 Test Method for Poisson's Ratio at Room Temperature 6
- E 228 Test Method for Linear Thermal Expansion of Solid Materials with a Vitreous Silica Dilatometer⁷
- E 261 Practice for Determining Neutron Fluence Rate, Fluence, and Spectra by Radioactivation Techniques⁸
- E 1461 Test Method for Thermal Diffusivity of Solids by the Flash Method 7

3. Terminology

3.1 Definition:

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

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² Annual Book of ASTM Standards, Vol 05.05.

³ Annual Book of ASTM Standards, Vol 15.01.

⁴ Discontinued; see 1985 Annual Book of ASTM Standards, Vol 15.01.

⁵ Annual Book of ASTM Standards, Vol 05.02.

⁶ Annual Book of ASTM Standards, Vol 03.01.

⁷ Annual Book of ASTM Standards, Vol 14.02.

⁸ Annual Book of ASTM Standards, Vol 12.02.

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TABLE 1 Summary of Test Methods for Graphite Components

Note-Designations under preparation will be added as editorial changes when approved.

	Fuel, Removable Re- flector, and Core Sup- port Elements; PBMR Reflector, Keys and Sleeves; and Dowel Pins	Permanent Side Re- flector Elements and Dowel Pins	Core Support Pedes- tals and Dowel Pins
Bulk Density:			
As-Manufactured Shapes	C 838	C 838	C 838
Machined Specimens	C 559	C 559	C 559
Thermal Properties:			
Linear Thermal Expansion	E 228 ^A	E 228 ^A	E 228 ^A
Thermal Conductivity	E 1461 ^A	E 1461 ^A	E 1461 ^A
Mechanical Properties:			
Compressive Strength	C 695	C 695	C 695
Tensile Properties	C 749 ^A	C 749 ^A	C 749 ^A
Poisson's Ratio	E 132 ^A	E 132 ^A	E 132 ^A
Flexural Strength	C 651 ^A	C 651 ^A	C 651 ^A
Fracture Toughness	В	В	В
Modulus of Elasticity	C 747	C 747	C 747
Oxidation Related Proper-			
ties:			
Relative Oxidation Rate	C 1179 ^{<i>B</i>}	C 1179 ^{<i>B</i>}	C 1179 ^{<i>B</i>}
Surface Area	C 1251	C 1251	C 1251
Permeability	C 577 ^{AB}	C 577 ^{AB}	C 577 ^{AB}
Catalytic Impurities	C 560 ^B	C 560 ^B	C 560 ^B
Sulfur Concentration	C 816	C 816	C 816
Porosity	В	В	В
Neutronic Impurities:			
Ash	C 561 ^A	C 561 ^A	C 561 ^A
Spectroscopic Analysis	В	В	В
Thermal Absorption Cross Section	C 626 ^A	C 626 ^A	С

^A Modification of this test method is required. See Section 8 for details.

^B New test methods are required. See Section 8 for details.

 $^{\ensuremath{C}}$ There is no identified need for determining this property.

TABLE 2 Summary of Test Methods for Boronated Graphite Components

Note-Designations under preparation will be added as editorial changes when approved.

	Compacts			Neutron Chield
	Control Rod	Burnable Poison	Reserve Shutdown	- Neutron Shield Material
Bulk Density	C 838	C 838	C 838	D 4292
Linear Thermal Expansion	С	E 228'' C	E 228'' C	D 2862
Mechanical Strength:				D 2002
Compressive Strength Impact Performance	C 695 ^A B	C 695 ^A B	C 695 ^A B	B C
Chemical Properties:				
Catalytic Impurities	С	С	С	С
Sulfur Concentration	С	С	С	С
Hafnium Concentration	С	С	С	С
Relative Oxidation Rate	С	С	С	С
Boron Analysis:				
Total Boron	С	С	С	С
Boron as Oxide	С	С	С	С
B₄C Particle Size	D 2862 ^D	D 2862 ^D	D 2862 ^D	D 2862 ^D

^A Modification of this test method is required. See Section 10 for details.

^B There is no identified need for determining this property.

^C New test methods are required. See Section 10 for details

^D Additional test methods are required. See Section10 for details.

3.1.1 Terminology C 709 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 high temperature gas-cooled reactors.

4.2 The test methods are applicable primarily to specimens in the unirradiated and unoxidized state. Many are also applicable to specimens in the irradiated or oxidized state, or both, provided the specimens meet all requirements of the test method. The user is cautioned to consider the instructions given in the test methods.

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 C 625 and E 261 or referenced to source information of equivalent content.

GRAPHITE COMPONENTS

7. Description and Function

7.1 Fuel and Removable Reflector Elements:

7.1.1 A fuel element is a removable graphite element that contains channels for the passage of coolant gas, the fuel material, the alignment dowel pins, and 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.

7.1.2 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.

7.1.3 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.

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

7.1.5 Except for support, guide, and containment of fuel material, removable reflector elements may also serve any of the functions listed in 7.1.2.

7.2 Permanent Side Reflector Elements:

7.2.1 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.

7.2.2 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.

7.3 Core Support Pedestals and Elements:

7.3.1 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 coolant gas, alignment dowel pins, and 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 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.

7.3.2 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.

7.4 Pebble Bed Modular Reactor (PBMR) Reflector Blocks:

7.4.1 The PBMR 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 sub divided into three subsystems, namely, the bottom, side, and top reflector. The side reflector is 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.

7.4.2 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.

7.4.3 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 Bulk Density:

8.1.1 Determine bulk density on as-manufactured or machined specimens in accordance with Test Methods C 838 and



C 559, respectively. Test Method C 838 includes shaped articles other than right circular cylinders and rectangular parallelepipeds. Test Method C 559 is used when a higher degree of accuracy is required. The procedures of Test Method C 559 are modified in Annex A1 to provide for the measurement of bulk density of nonuniform specimens.

8.2 Thermal Properties:

8.2.1 Determine linear thermal expansion in general accordance with Test Method E 228. Modifications to Test Method E 228, which are in preparation and will be presented as an annex, are required to ensure the reliability of measurements for coarse-grained graphite and to permit more convenient sizes for irradiation test specimens and manufacturing control.

8.2.2 Calculate the thermal conductivity from the thermal diffusivity as determined by Test Method E 1461. The required calculation is described in Annex Annex A2.

8.3 Mechanical Properties:

8.3.1 Determine compressive strength in accordance with Test Method C 695.

8.3.2 A new tension test method is under preparation which will provide for testing both unirradiated and irradiated specimens. Determination of tensile properties may also be made in accordance with Test Methods C 749 and E 132. The procedures of Test Method C 749 are modified in Annex Annex A3 to provide for the measurement of the tensile stress-strain properties of specimens with glued ends, a convenient method that has been used in the past and verified for the testing of irradiated and unirradiated (control) graphite specimens. The procedures of Test Method E 132 are modified in Annex A4 to provide specimen geometries and measurements specifically adapted for measuring the Poisson's ratio of graphite.

8.3.3 Determine flexural strength in accordance with Test Method C 651.

8.3.4 A test method for determining fracture toughness is in preparation.

8.3.5 Determine modulus of elasticity in accordance with Test Method C 747.

8.4 Oxidation Related Properties:

8.4.1 Determine the relative rate of oxidation by mass loss in air in accordance with Test Method C 1179.

8.4.2 A test method for determining surface area is in preparation.

8.4.3 *Gaseous Permeability*—Test Method C 577 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. A second method is also in preparation to provide for materials with lower permeabilities than those covered by Test Method C 577.

8.4.4 *Catalytic Impurities*—Determine the concentration of iron, vanadium, and calcium in accordance with Test Methods C 560. New test methods for determining the concentrations of other catalytic impurities are in preparation.

8.4.5 *Sulfur*—Determine sulfur concentration in accordance with Test Method C 816.

8.4.6 A test method for determining porosity is in preparation.

8.5 Neutronic Impurities:

8.5.1 Determination of ash shall be in accordance with Test Method C 561. New test methods for determining the ash content of graphites with impurities that are lost during conventional ashing are in preparation.

8.5.2 A test method for determining impurity concentrations by spectroscopic techniques is in preparation.

8.5.3 Test Method C 626 is being revised to provide for calculation of both nonburnable and burnable boron-equivalent content.

BORONATED GRAPHITE COMPONENTS

9. Description and Function

9.1 Control Rod Compacts:

9.1.1 The control rod compacts are dispersions of approximately 40-weight % boron as boron carbide (B_4C) in a graphite matrix. The compacts are in the form of short, thick-walled tubular elements and are enclosed within the annuli of thin-walled metallic containers. These assemblies are connected to form sections of control rods.

9.1.2 The function of the control rod compacts is to absorb neutrons when inserted within the core, thereby providing a means for controlling the nuclear reactions.

9.2 Burnable Poison Compacts:

9.2.1 The burnable poison compacts are dispersions of approximately 1 weight % boron as boron carbide (B_4C) in a graphite matrix. The compacts are in the form of solid cylinders and are enclosed within channels in fuel elements.

9.2.2 The function of the burnable poison is to reduce the magnitude of the long-term reactivity changes that accompany fuel burnup.

9.3 Neutron Shield Material:

9.3.1 Neutron shield material consists of granules containing dispersions of approximately 25–weight % boron as boron carbide (B_4C) in a graphite matrix. These granules are enclosed within metallic containers located above the core.

9.3.2 The function of the neutron shield material is to reduce the neutron flux to adjacent metallic components.

9.4 Reserve Shutdown Compacts:

9.4.1 The reserve shutdown compacts are dispersions of approximately 40–weight % boron as boron carbide (B_4C) in a graphite matrix. These compacts are in the form of spherical elements or short cylindrical elements with rounded ends and are gravity fed from storage hoppers above the core into channels within fuel elements when an emergency shutdown of the reactor is required.

9.4.2 The function of the reserve shutdown compacts is to absorb neutrons thereby providing a means for rapidly stopping the nuclear reactions.

10. Test Methods

10.1 *Particle Size*—Determine particle size of neutron shield material in accordance with Test Method D 2862. A new test method may be required for determining particle size in as-manufactured compacts.

10.2 *Bulk Density*—Determine bulk density on asmanufactured or machined specimens in accordance with Test Method C 838. Determine apparent bulk density of neutron shield material in accordance with Test Method D 2854.

10.3 *Linear Thermal Expansion*—Determine linear thermal expansion in general accordance with Test Method E 228. Modifications to Test Method E 228, which are in preparation and will be presented as an annex, are required to permit specimen geometries consistent with as-manufactured shapes.

10.4 Mechanical Properties:

10.4.1 Determine compressive strength in general accordance with Test Method C 695. An exception is for control rod compacts, for which Test Method C 695 is modified in Annex A5 to conform to specimen machining requirements for boron carbide-containing composite materials.

10.4.2 A test method for determining the impact performance of reserve shutdown compacts is in preparation.

10.5 Chemical Properties:

10.5.1 A test method for determining the concentrations of catalytic impurities is in preparation.

10.5.2 A test method for determining the sulfur concentration is in preparation. 10.5.3 A test method for determining the hafnium concentration is in preparation.

10.5.4 A test method for determining the relative rates of oxidation by primary coolant impurities is in preparation.

10.6 Boron Analyses:

10.6.1 A test method for determining the total boron content is in preparation.

10.6.2 A test method for determining boron as boron oxide (moisture-leachable boron compound) is in preparation.

10.6.3 Determine B_4C particle size prior to manufacture of component shapes in accordance with Test Method D 2862. A new test method may be required for determining B_4C particle size in as-manufactured components.

11. Keywords

11.1 boronated graphite; chemical properties; graphite; high temperature gas-cooled nuclear reactor; mechanical properties; neutronic properties; physical properties; thermal properties

ANNEXES

(Mandatory Information)

A1. BULK DENSITY OF NONUNIFORM TEST SPECIMENS

A1.1 The bulk density of test specimens other than right circular cylinders or rectangular parallelepipeds may be determined using Test Method C 559 provided the specimen volume can be determined within 0.15 %. (See 6.2 of Test Method C 559.)

A1.2 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 C 559.

A2. DETERMINATION OF THERMAL CONDUCTIVITY FROM THERMAL DIFFUSIVITY

A2.1 The thermal conductivity may be calculated from the thermal diffusivity as determined by Test Method E 1461 from the following equation:

$$\lambda = \alpha C_{\rm p} \rho$$

where:

λ = thermal conductivity, W/m·K, α = diffusivity, m²/s, C_p = specific heat, J/kg·K, and ρ = density, kg/m³. The required values of C are given

The required values of C_p are given in Table A2.1. The following equation may be used in place of the table for temperatures between 300 and 3000 K:

$$C_{\rm p} = 8.1353 \times 10^{7} \,{\rm T}^{-2} - 6.2119 \times 10^{5} \,{\rm T}^{-1} + 1289.2 + 2.6326 \,{\rm T} \\ - 2.5559 \times 10^{-3} \,{\rm T}^{2} + 1.2376 \times 10^{-6} \,{\rm T}^{3} + 2.9593 \times 10^{-10} \,{\rm T}^{4} \\ + 2.7904 \times 10^{-14} \,{\rm T}^{5}$$

This equation reproduces the tabulated values within 0.1 % for the indicated range of temperatures.

TABLE A2.1	Recommended Values of the Specific Heat of				
Oneutite					

Graphite							
Т (К)	C _p (J/kg·K)	T (K)	C _p (J/kg⋅K)	T (K)	C _p (J/kg·K)		
300	713	1300	1940	2300	2142		
400	961	1400	1972	2400	2154		
500	1187	1500	2000	2500	2165		
600	1370	1600	2024	2600	2174		
700	1516	1700	2046	2700	2184		
800	1632	1800	2066	2800	2193		
900	1723	1900	2083	2900	2202		
1000	1796	2000	2100	3000	2213		
1100	1854	2100	2115				
1200	1901	2200	2129				

A2.2 (**Warning**—Calculation of thermal conductivity from thermal diffusivity is valid for most bulk graphites, but can lead to significant error for highly porous carbons and graphites where the initial heat pulse penetrates appreciably beyond the sample front face.)

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A3. MODIFICATIONS TO TEST METHOD C 749 FOR GLUED-END SPECIMENS

A3.1 The test specimen configurations referred to in Test Method C 749, Fig. 9, incorporate integral grooved heads for mounting the specimens in the gripping devices and reduced gage sections to control fracture location. However, test parameters for some studies (irradiation and oxidation studies and quality assurance tests for many manufactured carbon and graphite articles) may impose such stringent requirements on volume, diameter, and geometry that the resultant test specimen may be simply a right circular cylinder. This annex deals with bonding connectors to cylindrical specimens having plain ends to conduct tensile tests employing the load train and gripping devices detailed in Test Method C 749.

A3.2 *Test Specimen*—The test specimen shall be cylindrical with plain ends machined perpendicular to the longitudinal axis. (**Warning**—Experience has shown that when testing high strength graphite or graphites that have a large Poisson's ratio mismatch with that of the adhesive, specimens may fail at or very near the adhesive joint and yield invalid measurements. Under these circumstances, consideration should be given to the use of a specimen with a reduced gage section.)

A3.2.1 The recommended test specimen size is 0.256 in. (9.5 mm) diameter.

A3.2.2 The recommended height to diameter ratio for the specimen gage section is 4.

A3.2.3 The cylindrical surface shall be flat within 0.002 in. (0.03 mm), and the minimum diameter must not occur at either end of the specimen. The end faces of the specimen shall be perpendicular to the cylindrical surface to within 0.001 in./in. (0.02 mm/mm) of diameter total indicator reading. Reasonable care shall be exercised to assure that all edges are sharp and without chips or other flaws.

A3.3 *Specimen Connectors*—The specimen connectors that are bonded to the specimen ends shall be sized to fit the gripping devices. The recommended material for the specimen connectors is 6061-T6 aluminum alloy. The end (bond) face of

the connector shall be flat within 0.001 in. (0.02 mm) and perpendicular to the cylindrical axis of the connector within 0.001 in./in. (0.02 mm/mm) of diameter total indicator reading.

A3.4 Attachment of Test Specimens to Specimen Connectors—Specimen connectors shall be bonded to the test specimen with an epoxy or cyanoacrylate adhesive.

A3.4.1 The axial center lines of the test specimen and specimen connectors shall be aligned during bonding using an appropriate alignment fixture. The run out tolerance for the finished assembly shall be within 0.001 in. (0.02 mm) total indicator reading.

A3.4.2 An adhesive with a tensile shear strength (aluminum alloy to aluminum alloy) greater that 2500 psi (17 MPa) is recommended.

A3.4.3 The bond face of the specimen connector shall be approximately etched or grit blasted, washed, dried, and degreased to promote a strong adhesive bond.

A3.4.4 Dust shall be removed from the ends of the specimen by one of the following techniques: (1) rapid immersion in liquid nitrogen (preferred), (2) ultrasonic cleaning in an alcohol bath, or (3) taping and retaping ends.

A3.5 *Test Procedures*—Follow the test procedures given in 8.1 through 8.4.

A3.5.1 If the fracture occurs within a distance less than 10 times the measured thickness of the adhesive joint at either end of the specimen, the strength results shall be reported but not included in the calculation of the average strength value.

A3.6 *Tensile Property Calculations*—Calculate the strength, modulus of elasticity, and strain-to-failure as indicated in Section 9.

A3.7 *Precision*—A round-robin test is being planned to develop precision statements for this test method.

A3.8 Bias—Refer to 11.1.

A4. MODIFICATIONS TO TEST METHOD E 132

A4.1 The following modifications to Test Method E 132 are required to more clearly define the method for measuring Poisson's ratio of graphite. The comments are arranged to apply to the relevant sections by number as designated in Test Method E 132.

A4.2 *Note 3*—This discussion is to be disregarded. This annex does not require the measurement of the shear modulus *G*. The three independent Poisson's ratios will be determined by the requirements following below.

A4.2.1 It is recommended that at least three pairs of extensioneters be employed. The third pair shall be transverse to the direction of load and perpendicular to the first set of transverse extensioneters (that is, perpendicular to the plane of Fig. 1). Note 5 applies with the additional transverse pair added to the configurations.

A4.3 The following considerations shall be added to this paragraph: The parent block of material shall be assumed to have cylindrical symmetry and the same shall be assumed to apply to all samples abstracted from the block. The samples shall be classified into two groups—axial (wherein the axis of symmetry is assumed to be parallel to the direction of loading), and radial (wherein the axis of symmetry is assumed to be perpendicular to the direction of loading). This classification is recognized to be a simplification, but symmetries of lower order are beyond the scope of this method.

A4.4 This paragraph applies with the following two exceptions: first, the width and thickness shall be equal within practical limitations; second, these two dimensions should exceed the maximum particle size by a factor of 3, or the results may be atypical. It should be emphasized that the specimen size should be as large as possible to reduce experimental difficulty and uncertainty in obtaining representative measurements.

A4.5 The considerations of this paragraph must be modified for the assumed cylindrical symmetry of the specimens. Let the *z*-coordinate be the axis of symmetry and the *x*- and *y*-coordinates be two other mutually orthogonal coordinates. In general, the *z*-axis will correspond to either the molding or the extrusion direction. The approximation of uniform, cylindrical symmetry may not be valid and, if necessary, should be confirmed by appropriate sampling. (**Warning**—The failure of an extensometer pair to separately yield the same value for Poisson's ratio may not be an indication of misalignment, but rather of failure of the material to meet perfect cylindrical symmetry.)

The following two new subsections are added to 7.1:

A4.5.1 Specimens machined such that a uniform load, p, is applied along the *z*-axis yield the following Poisson's ratio:

$$\mu_1 = -\left(\frac{\partial \epsilon_x}{\partial p} \frac{\partial p}{\partial \epsilon_z}\right)_p = -\left(\frac{\partial \epsilon_y}{\partial p} \frac{\partial p}{\partial \epsilon_z}\right)_p$$

where ϵ_x and ϵ_y are the transverse strains, and ϵ_z is the longitudinal strain. Both of these ratios shall be measured to assure that the approximation of cylindrical symmetry is valid.

A4.5.2 Specimens machined such that a uniform load can be applied in a direction perpendicular to the *z*-axis of symmetry (call this the *x*-axis) yield the following Poisson's ratios:

$$\mu_{2} = -\left(\frac{\partial \epsilon_{y}}{\partial p}\frac{\partial p}{\partial \epsilon_{x}}\right)_{p}$$
$$\mu_{3} = -\left(\frac{\partial \epsilon_{z}}{\partial p}\frac{\partial p}{\partial \epsilon_{x}}\right)_{p}$$

where ϵ_y and ϵ_z are the two transverse strains oriented in the y and z directions, and ϵ_x is the longitudinal strain. While the testing in only two directions is a minimum requirement, it is desirable that additional test samples be oriented so that the uniform load applied along the y-axis also be evaluated.

A4.6 This paragraph will not apply. A normal regression analysis may be used to determine the instantaneous slope $d\epsilon/dp$ as appropriate, but no true proportional limit will usually exist. Each of the three μ_i can be calculated by the formula given in the method, except that the "lines" in general will not be linear and each μ_i will be a function of applied load *p*.

A5. MODIFICATIONS TO TEST METHOD C 695

A5.1 The following modifications to Test Method C 695 are designed to permit measurements of compressive strength of boron carbide/graphite composite materials for which, due to differences in hardness of the two components, normal machining tolerances cannot be maintained. Paragraph numbering below corresponds to pertinent section numbers of Test Method C 695.

A5.2 The cushion pad thickness shall be 0.015 to 0.025 in. (0.4 to 0.6 mm).

A5.3 This section shall apply up to the requirement on surface finish. It is recognized that tool drag will cause pullout of boron carbide particles; hence, all surfaces of the sample shall have a finish no rougher than a rating of 500 μ in. rms



(12.7 μ m). Furthermore, these surfaces shall show no obvious regions of segregation between carbide and graphite (carbon) and shall not exhibit visible spalling. Reasonable care should be exercised to assure that edges are sharp and without flaws and chipping.

A5.4 This section shall apply in its entirety with the exception that the minimum diameter shall be no less than five

times the maximum particle size in the carbide or the graphite (carbon), whichever is larger. In those cases where the formed part does not permit abstracting samples meeting this requirement, it may be waived; but in no case shall the diameter be less than 0.3 in. (7.5 mm).

A5.5 Add 6.1.7, Sample dimensions; and 6.1.8, Crushing strength.

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