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Standard Guide for Measurements on Small Graphite Specimens¹

This standard is issued under the fixed designation D7775; 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 guide covers best practice for properties measurements on small (nonstandard) graphite specimens and requirements for representing properties of the bulk material. This guide is aimed specifically at measurements required on graphites, where there may be constraints on the geometry or volume of the test specimen, or both. The objective of this guide is to provide advice on how the application of selected standards under noncompliant conditions can be tested for suitability.

1.2 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.3 *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.*

1.4 *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
- C565 Test Methods for Tension Testing of Carbon and Graphite Mechanical Materials
- C611 Test Method for Electrical Resistivity of Manufactured Carbon and Graphite Articles at Room Temperature

¹ This guide 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.

- 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
- C714 Test Method for Thermal Diffusivity of Carbon and Graphite by Thermal Pulse Method
- C747 Test Method for Moduli of Elasticity and Fundamental Frequencies of Carbon and Graphite Materials by Sonic Resonance
- C748 Test Method for Rockwell Hardness of Graphite Materials
- 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
- C886 Test Method for Scleroscope Hardness Testing of Carbon and Graphite Materials
- C1161 Test Method for Flexural Strength of Advanced Ceramics at Ambient Temperature
- C1259 Test Method for Dynamic Young's Modulus, Shear Modulus, and Poisson's Ratio for Advanced Ceramics by Impulse Excitation of Vibration
- D7779 Test Method for Determination of Fracture Toughness of Graphite at Ambient Temperature
- D7972 Test Method for Flexural Strength of Manufactured Carbon and Graphite Articles Using Three-Point Loading at Room Temperature
- D8289 Test Method for Tensile Strength Estimate by Disc Compression of Manufactured Graphite
- D8356 Test Method for Sonic Velocity in Manufactured Carbons and Graphite Materials for use in Obtaining Approximate Elastic Constants: Young's Modulus, Shear Modulus, and Poisson's Ratio
- E228 Test Method for Linear Thermal Expansion of Solid Materials With a Push-Rod Dilatometer
- E1461 Test Method for Thermal Diffusivity by the Flash Method

3. Summary of Guide

3.1 There is currently a suite of ASTM standards (see 2.1) that can be applied to graphite covering a range of physical, mechanical, electrical and thermal property measurements.

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

Each of these standards has been developed with the objective of optimizing the method of measurement in the absence of any constraints on test specimen production. Without exception, these standards specify limits on the ratio between test specimen dimensions and coke and filler grain sizes or prescribe test specimen geometries or size ranges, or both. The default position for any user should be to follow these standards exactly as described. However, in some applications, available test material or experiment design constraints on test specimen sizes may result in noncompliance. The objective of this guide is to provide advice on how the application of selected standards under noncompliant conditions can be tested for suitability. The ultimate objective is to provide guidance on the use of each of the ASTM standards listed. The 2021 issue of this guide addresses nine standards: Test Method **C559** for Bulk Density by Physical Measurement of Manufactured Carbon and Graphite Articles, Test Method **C611** for Electrical Resistivity of Manufactured Carbon and Graphite Articles at Room Temperature, Test Method **C747** for Moduli of Elasticity and Fundamental Frequencies of Carbon and Graphite Materials by Sonic Resonance, Test Method **C769** for Sonic Velocity in Manufactured Carbon and Graphite Materials for Use in Obtaining Young's Modulus, Test Method **C749** for Tensile Stress-Strain of Carbon and Graphite, Test Method **D7972** for Flexural Strength of Manufactured Carbon and Graphite Articles Using Three-Point Loading at Room Temperature, Test Method **E228** for Linear Thermal Expansion of Solid Materials with a Push-Rod Dilatometer, and Test Method **E1461** for Thermal Diffusivity by the Flash Method.

4. Significance and Use

4.1 The purpose of this guide is to report considerations, which should be included in testing nonstandard specimens that lie outside the constraints imposed on size/volume in existing ASTM standards for graphite (noting that there are some generic ASTM standards with no such constraints). These constraints may be real or may be an artifact of the round-robin test program that supported the standard. It is the responsibility of the user to demonstrate that the application of a standard outside any specified constraints is valid and reasonably provides properties of the bulk material from which the nonstandard specimen was extracted.

5. Test Specimen Volume/Size Constraints in Current Standards

5.1 *Test Method **C559***—Applies to test specimens with rectangular parallelepiped or right circular cylinder geometry. The minimum test volume is specified as 500 mm³. The minimum test specimen dimension should be 10 times the length of the largest visible grain.

5.2 *Test Methods **C565***—Applies to reduced diameter uniaxial tensile specimens. Grain size must be smaller than 0.79 mm; while not specified, it is assumed that this refers to average grain size. The acceptable fracture zone shall be 19 mm long with the centre of the zone at the point of minimum diameter. The ratio of specimen diameter to grain size or flaw size must be greater than 5.

5.3 *Test Method **C611***—Applies to strip, rod, bar or tube geometries. Specimen length to maximum cross-sectional dimension should be 6:1. No dimension should be smaller than 5 times the length of the largest visible grain.

5.4 *Test Method **C651***—Applies to rectangular parallelepiped geometries. The minimum dimension should be greater than 5 times the largest grain dimension. Test specimen length to thickness should be greater than 8. The ratio of test specimen width to thickness should be less than or equal to 2.

5.5 *Test Method **C695***—Applies to right cylinder geometry. The test specimen diameter should be greater than 10 times the maximum grain size. The test specimen height to diameter ratio should be in the range 1.9 to 2.1. The minimum test size is specified as 9.5 mm diameter and 19.1 mm height.

5.6 *Test Method **C714***—Applies to circular disks, 2 mm to 4 mm thick and 6 mm to 12 mm in diameter. The diameter must not be too large relative to the flash source as the front surface needs to be heated uniformly. The specimen thickness must be selected such that $\tau/t_{1/2}$ is smaller than 0.02, where τ is the pulse time and $t_{1/2}$ is the time for the rear surface temperature to rise to one half of its maximum value.

5.7 *Test Method **C747***—Applies to slender rod or bar geometries. The test specimen length to thickness ratio should lie in the range 5 to 20:1.

5.8 *Test Method **C748***—Applies to flat specimens of minimum thickness 6.35 mm. The grain size of the test material should be less than 0.8 mm, with a hardness range 0 to 120 Rockwell L.

5.9 *Test Method **C749***—Applies to reduced-diameter uniaxial tensile test geometries as defined in Fig. 9 of that standard. Gauge diameter must be greater than 3 to 5 times the maximum grain size.

5.10 *Test Method **C769***—Applies to right cylinder geometry. The user should minimize attenuation of the sonic pulse by selecting a wavelength appropriate to the grain size of the test material. If the test specimen is a few grains thick, acceptability of application should be tested over a range of lengths. Specimen should have a diameter of at least a factor of two and ideally a factor of five greater than the wavelength of sound within the material.

5.11 *Test Method **C886***—Can be applied to any convenient test specimen size, but test surfaces smaller than 5 mm by 5 mm are not recommended. The material must have a grain size less than 0.8 mm. The minimum specimen thickness is 5 mm.

5.12 *Test Method **C1161***—Applies to rectangular parallelepiped geometries and can be adapted for graphite. The average grain size should be less than 2 % of the beam thickness. For beam lengths of 25 mm, 45 mm, and 90 mm, specified widths are 2 mm, 4 mm, and 8 mm, respectively, and specified depths are 1.5 mm, 3 mm, and 6 mm, respectively.

5.13 *Test Method **C1259***—Can be applied to graphite test specimens with both round and rectangular cross sections. The ratio of test specimen length to minimal cross-sectional dimension should be greater than 10, and preferably greater than 20.