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An American National Standard

Standard Guide for Measurements on Small Graphite Specimens[1]

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.

~~ε[1] NOTE—Minor formatting changes made throughout editorially in July 2012.~~

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 nuclear graphites, where there may be constraints on the geometry or volume of the test specimen, or both.

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

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
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 Young's Modulus
C781 Practice for Testing Graphite and Boronated Graphite Materials for High-Temperature Gas-Cooled Nuclear Reactor Components
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
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. 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

[1] This guide is under the jurisdiction of ASTM Committee D02 on Petroleum ~~Products~~ 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.

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 2011 issue of this guide addresses eight 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 and Practice C781 for Testing Graphite and Boronated Graphite Materials for High-Temperature Gas-Cooled Nuclear Reactor Components, 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 mm~~500 mm^3. 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;~~ 0.79 mm; while not specified, it is assumed that this refers to average grain size. The acceptable fracture zone shall be ~~19 mm~~ 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~~ 9.5 mm diameter and ~~19.1 mm~~ 19.1 mm height.

5.6 *Test Method C714*—Applies to circular disks, 2 to 4 mm thick and 6 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.~~ 6.35 mm. The grain size of the test material should be less than ~~0.8 mm,~~ 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. Gage 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~~5 mm by ~~5 mm~~ 5 mm are not recommended. The material must have a grain size less than ~~0.8 mm.~~ 0.8 mm. The minimum specimen thickness is ~~5 mm.~~5 mm.

5.12 *Test Method E228*—Applies to right cylinder (preferable) or slab geometries. Ideally, test specimens should be ~~25~~25 mm to ~~60 mm~~ 60 mm long and ~~5~~5 mm to ~~10 mm~~ 10 mm in diameter or equivalent (although there is no fundamental limitation provided

the instrument controls the maximum thermal gradient to better than ~~±2°C/50~~±2 °C~~/ mm).~~ 50 mm). The specimen length should be such that the accuracy of determining the expansion $\Delta L/L_0$ is at least ~~±20~~ ±20 mm ~~mm/m.~~/m.

5.13 *Test Method E1461*—Applies to thin circular disk specimens with the front surface area less than that of the energy beam. Typically, test specimens should be ~~10~~10 mm to ~~12.5 mm~~ 12.5 mm in diameter and ~~1~~1 mm to ~~6 mm~~ 6 mm in thickness.

5.14 *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. For shear modulus measurements, the test specimen width to thickness ratio should be greater than 5.

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

6. General Principle for Measurements Outside Specified Specimen Volume/Size Constraints in Current Standards

6.1 The default position for any user should be to follow these standards exactly as described.

6.2 Specimen size and volume constraints may be set by a particular measurement technique and hence apply to any test material, but some may depend upon the microstructure and composition of the material. In such cases, it is preferable to provide technical data and basis to support the choice of the adapted measurement technique and test specimen dimensions used.

6.3 A simple, general principle should be applied to any proposed measurements that are noncompliant with respect to volume/size.

6.3.1 The user must first specify the level of accuracy required for the measurements together with tolerable repeatability, tolerance, and bias uncertainties associated with the measured properties. This may need to take into account the number of specimens used for the measurements.

6.3.2 These qualifying measurement criteria must be demonstrated using representative material in a manner compliant with the ASTM standard. The user should take account of in-service changes to test material (for example, irradiation, oxidation) when selecting representative material for such a demonstration; as-manufactured material may not be sufficiently representative for such purposes.

6.3.3 The measurements should then be repeated on the same material, progressively reducing the volume/size of the specimen and repeating the measurements. Ideally, this procedure would involve the successive re-sizing of the starting specimen. This would ensure that no specimen to specimen variability affected the results. Consideration should be given to within specimen variability and any potential effects of specimen preparation that might affect the property measurement. This process should be continued until there are sufficient compliant data to benchmark the measurement technique against the material; there should be sufficient data at and below the desired test specimen geometry to characterize the dependence of the measured property upon volume/size. It may be necessary to study more than one parameter and these should be varied singly in order not to confound the results.

6.3.4 The results should be analyzed to establish either the standard can be applied directly to an extended specimen volume/size range or it can be applied with volume/size corrections. In both cases, the accuracy and uncertainty of the measurement at the desired specimen volume/size should be evaluated and assessed for acceptability against the original specification.

6.3.5 It is good practice to retain the test specimens as checks or secondary standards in the subsequent measurement campaigns.

7. Bulk Density by Physical Measurement (Test Method C559)

7.1 Test Method C559 requires a mass measurement and a volume determination by mensuration on a test specimen with either a rectangular parallelepiped or right cylinder geometry. The standard specifies that the specimen volume should not be less than ~~500 mm~~500 mm^3 and the minimum dimension must be at least ten times the length of the largest visible grain. The minimum dimension should also be more than 2000 times the resolution of the measuring device. The volume determination involves four length measurements (longest dimension) either at the centre of each long face in the case of the rectangular parallelepiped or 90° apart on the periphery of the circular end faces in the case of the right cylinder. For the rectangular parallelepiped, width and thickness at each end and at two intermediate points along the length are required. For the right cylinder, two sets of diameter measurements are required, each set consisting of four measurements, one at each end and two at two intermediate points along an axial line.

7.2 The accuracy of contact measuring devices must be assessed in the context of point and flat contact options.

7.3 Principal sources of mensuration error will arise from geometry irregularity and from surface condition.

7.4 For specimens of regular geometry, mensuration could be carried out with automated multi-measurement contact devices that record and analyze results for prescribed measurement patterns.

7.5 Non-contact scanning devices can also be used to determine volumes of both regular and non-regular geometry specimens. Such devices need careful qualification before use to ensure the detectors respond consistently for graphite surfaces. The calibration and accuracy of the device must be tested on volume standards made from materials that respond to the scanning beam in a simple manner to graphite.

7.6 Bulk density can also be determined using Archimedes' Principle, as an alternative to mensuration techniques. The specimen immersed in a fluid is subject to an upwards buoyancy force equal to the weight of the fluid displaced by the specimen. By measuring the weight of the immersed specimen, the buoyancy force can be deduced, and by using the measured mass of the dry specimen the density can be calculated. This "immersion" method has the advantage of being applicable to non-regular specimen geometries. For a porous material, the method depends upon a constant level of penetration of the open pores by the fluid. The level of penetration is not important provided it is reproducible between repeat immersions.

7.7 An application of the immersion method is described as follows:

7.7.1 The dry test specimen is first weighed then immersed in water and reweighed while immersed; the specimen is removed from the water and the excess water removed by blotting on a damp chamois leather. The "blotted" specimen is reweighed and then immersed and weighed again while immersed. The difference between these two measurements is calculated and the cycle of measurements is repeated until three consecutive pairs of measurements are achieved with prescribed limits. Assuming a density of water of ρ_0 = 1000 kg m^{-3}, the density of the test specimen is:

$$\rho = \frac{W_1}{W_4}\rho_0 \tag{1}$$

where:

ρ = test specimen density, kg m^{-3},
ρ_0 = density of water, kg m^{-3},
W_1 = dry mass of test specimen, kg,
W_4 = mass difference from ($W_3 - W_2$), kg,
W_3 = dried mass, kg, and
W_2 = immersed mass, kg.

7.7.2 Corrections can be made to account for variations in the density of water due to temperature, dissolved air and purity. In practice, these effects are negligible compared to uncertainties in the overall method (and a water density of ~~1.0 g~~ 1.0 g cm^{-3} is normally assumed). Surface tension forces associated with the pan suspension wire and the water may need to be accounted for in the claimed level of accuracy.

7.7.3 In principle, there are no constraints on test specimen geometry. In practice, irregular geometries may trap air bubbles and are more difficult to dry between immersions.

7.7.4 In principle, there is no limit on test specimen size although the practical limit is set by the size of the pan on the balance. Also, as the test specimen volume decreases, uncertainties in density determinations increase due to the increasing significance of surface effects.

7.7.5 The test method as described requires the density of the test specimen to be greater than that of water. For low density material, a fixed weight kept immersed in the water reservoir is placed on the immersed specimen and its mass subtracted in the density evaluation.

7.7.6 Uncertainties in measurement may arise if the graphite specimen is friable. This can be quantified by ensuring that the oven-dried weight of the test specimen is known before and after measurement.

7.7.7 For highly porous materials, varying penetration of the water between immersions may lead to uncertainties in bulk density determinations. Water may "drain" out of the test specimen during surface drying and repenetrate the porosity to a varying degree during immersion. This can be addressed by waxing the test specimen, where the open porosity is partly filled with wax and the surface tension on the surface of the specimen is changed to prevent water penetration. This treatment is invasive and only applicable if no further measurements on the specimen are required. Care needs to be taken in the evaluation of the bulk density. The dry mass of the test specimen must be the mass measured before waxing, the difference in apparent weight between the test specimen immersed and the test specimen removed from the water being equal to the weight of the water being displaced by the removed specimen.

8. Electrical Resistivity (Test Method C611)

8.1 Test Method C611 applies to specimens in the form of a strip, rod, bar, or tube with a uniform cross-section. No dimension shall be smaller than five times the length of the largest visible grain.

8.2 The resistance of the material is measured by passing an electric current between two contact points on the specimen and measuring the potential drop. Numerous measurements are advised (16 is the recommended number) in order to minimize specimen or contact point artifacts which might lead to erroneous resistance values. The resistivity of the specimen is defined as ρ in the following relationship: