Designation: D7775 – 16

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.

## 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. 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 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 an Approximate Value of 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

D7972 Test Method for Flexural Strength of Manufactured Carbon and Graphite Articles Using Three-Point Loading at Room Temperature

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 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

[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.

Current edition approved Dec. 15, 2016. Published February 2017. Originally approved in 2011. Last previous edition approved in 2015 as D7775 – 11 (2015). DOI: 10.1520/D7775-16.

[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

suitability. The ultimate objective is to provide guidance on the use of each of the ASTM standards listed. The 2016 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 and 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 $mm^3$. 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; 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 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 $\tau$ 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. For shear modulus measurements, the test specimen width to thickness ratio should be greater than 5.

5.14 *Test Method D7972*—Applies only to those specimen sizes and geometries selected for the interlaboratory study that underwrites the standard. Reference should be made to the table of specimen sizes and testing configurations included in the standard. It is recommended that the size of the test specimen is selected such that the minimum dimension of the specimen is greater than 5 times the largest particle dimension. It is recommended that the test specimen has a length to thickness/diameter ratio of at least 6, and a width to thickness ratio not greater than 2.

5.15 *Test Method E228*—Applies to right cylinder (preferable) or slab geometries. Ideally, test specimens should be 25 mm to 60 mm long and 5 mm to 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 mm). The specimen length should be such that the accuracy of determining the expansion $\Delta L/L_0$ is at least ±20 mm/m.

5.16 *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 mm to 12.5 mm in diameter and 1 mm to 6 mm in thickness.

## 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 $mm^3$ 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 center 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 $\rho_0 = 1000$ kg $m^{-3}$, the density of the test specimen is:

$$\rho = \frac{W_1}{W_4}\rho_0 \quad (1)$$

where:

$\rho$ = test specimen density, kg $m^{-3}$,
$\rho_0$ = 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 $cm^{-3}$ 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 $\rho$ in the following relationship:

$$\rho = \frac{R \cdot A}{L} \quad (2)$$

where:

$\rho$ = resistivity in m$\Omega$ meters,
$R$ = resistance of the material,
$A$ = uniform cross section, $mm^2$, and
$L$ = distance between potential contacts, mm.

8.3 The specimen geometry requirement of a specimen length to maximum cross-sectional dimension of at least 6:1 may pose a challenge to test specimens with specific size constraints when coupled with the 5:1 ratio of smallest dimension to grain or visible particle size.

8.4 For specimens with length to cross-section dimensions of less than 6:1, the number of measurements taken and resulting spread of data will indicate whether the measured resistivity values are reliable. Care should be taken to ensure that the length measurement between contact points can still be measured to within $\pm 0.5$ %, as errors due to contact length variation will be magnified commensurate with the total length available for attachment of electrical contacts. Small specimen errors can again be "averaged" by multiple measurements, as required by the specification even for ideally sized test specimens.

8.5 Small specimens that will not meet the minimum required grain or particle size restriction should, where possible, be evaluated through a systematic set of resistivity measurements with progressively larger specimens. A measurable shift in resistivity as more grains are included in scoping specimen sets will provide the operator with an indication of the expected contribution of grain boundary effects in the electrical resistance of the cross-section of the material.

8.6 Reported results for resistivity measurements on specimens that do not meet the dimensional restrictions of Test Method C611, in addition to the resistivity value calculated from the average resistance measured, should also record each individual measurement so that an evaluation can be made with respect to the spread of values collected and precision of the measurement technique.

## 9. Moduli of Elasticity and Fundamental Frequencies by Sonic Resonance (Test Method C747)

9.1 Test Method C747 applies to specimen geometries that are straight and have uniform cross section. Accurate results will depend upon appropriate shape factors that rely on careful dimensional measurements, which are more prone to error or variation in smaller geometries.

9.2 Specimens having relatively small or large ratios of length to thickness may be difficult to excite in the fundamental frequency modes, and therefore a length to width ratio between 5 and 20 is required by this test method.

9.3 The elastic modulus or modulus of rigidity, as appropriate, is calculated in the transverse (or flexural) mode, the longitudinal mode, or the torsional mode based upon the use of the specimen‘s fundamental mode of vibration, appropriate to the calculated modulus.

9.4 Deviations from the recommended test specimen ratio range introduce an elevated level of difficulty in obtaining a measurable fundamental frequency. The modulus of the test material is first established from the measured frequency of a large test specimen and this modulus is used to estimate a frequency for the small test specimens. It is recommended that two additional guidelines be employed in order to increase the confidence in the recorded frequency:

9.4.1 Determine the fundamental frequency using specimens that are within the recommended length to width ratio of between 5 and 20, or use progressively larger specimens as necessary, in order to establish baseline frequency characteristics of the material being evaluated. The expected value for fundamental frequency of a nonstandard specimen can be calculated based upon the measured geometry and the known fundamental frequency of a standard specimen, and any deviation or shift can be appropriately noted.

9.4.2 In all cases, use the required procedural practices employed in Test Method C1259 for the number of readings taken. For small test specimens, there is a risk that the excited mode is not the intended mode and consequently the formula applied to calculate modulus from the frequency will be wrong. Spurious vibration modes are more easily discounted with the fundamental frequency of the required mode having been measured if the test is repeated on the same specimen until ten readings are within ±10 % of the mean. It is acknowledged that for less ideal specimen geometries, the frequency mean that is eventually used for the modulus calculation may require an extended number of measurements until an appropriate group of ten readings is obtained. In this case, the total number of measurements required to obtain the group of ten readings should be reported. It is good practice to confirm independently that the excited mode is the intended one by using an alternative experimental, numerical or analytical method.

9.5 The report shall contain additional information pertaining to the testing that may have been carried out per item 9.4.1, and the individual numerical values recorded and the mean value obtained per item 9.4.2.

## 10. Tensile Stress-Strain (Test Method C749)

10.1 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.

10.2 Practice C781 includes an annex describing modifications to Test Method C749 to extend its application to small test specimens. This annex does not address gauge diameter to grain size constraints but it does describe how bonding connectors to specimens can extend applicability of the method described in Test Method C749 from the standard reduced-diameter uniaxial test geometry to a simple right cylindrical geometry. In order for this guide to contain a complete compilation of methods for small graphite specimens, the procedure in Annex A4 of Practice C781 has been reproduced here.

10.2.1 *Test Specimen*—The test specimen shall be cylindrical with ends machined perpendicular to the longitudinal axis.

10.2.1.1 The recommended test specimen size is 6.5 mm diameter.

10.2.1.2 The recommended height to diameter ratio for the specimen gauge section is 4.

10.2.1.3 The cylindrical surface shall be flat within 0.05 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 0.025 mm⁄mm of diameter total indicator reading. Reasonable care shall be exercised to ensure that all edges are sharp and without chips or other flaws.

10.2.2 *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 0.025 mm and perpendicular to the cylindrical axis of the connector within 0.02 mm⁄mm of diameter total indicator reading.

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

10.2.3.1 The axial center line 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 0.025 mm total indicator reading.

10.2.3.2 An adhesive with a tensile shear strength (aluminum alloy to aluminum alloy) greater that 17 MPa is recommended.

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

10.2.3.4 The ends of the specimen shall be dust-, grease-, and moisture-free.

10.2.4 *Test Procedures*—Follow the test procedures given in Sections 8.1 through 8.4 of Test Method C749.

10.2.4.1 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.

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 gauge section.

10.2.5 *Tensile Property Calculations*—Calculate the strength, modulus of elasticity, and strain-to-failure as indicated in Section 9 of Test Method C749.

10.2.6 *Precision and Bias*—A round-robin test is being planned to develop precision and bias statements for this test method.

10.3 When applying bonded connectors to small cylindrical specimens in order to measure strength, modulus of elasticity and strain-to-failure, the user should take account of the gauge diameter to grain size constraint in Test Method C749 when assessing the validity and applicability of test results.

## 11. Sonic Velocity for Use in obtaining Young's Modulus (Test Method C769)

11.1 Test Method C769 applies to right cylinder geometries. If the test specimen is a few grains thick, acceptability of application (that is, the measurement has been made over a volume representative of the bulk material) should be tested over a range of lengths. Specimen should have a diameter at least a factor 2 and ideally a factor 5 greater than the wavelength of sound within the material.

11.2 The velocity of longitudinal sound waves passing through the test specimen is determined by measuring the distance through the specimen and dividing by the time elapsed, between the transmitted pulse and the received pulse (1, 2).[3] Provided the wavelength of the transmitted pulse is a sufficiently small fraction of the test specimen lateral dimensions, a value for Young's modulus for isotropic graphite can then be obtained using Eq 3 and 4.

$$E = C_v \rho V^2 \tag{3}$$

where:

$E$ = Young's modulus of elasticity, Pa,
$\rho$ = density, kg/m$^3$,
$V$ = longitudinal signal velocity, m/s, and
$C_v$ = Poisson's factor.

The Poisson's factor, $C_v$ is related to Poisson's ratio, ν, by the equation:

$$C_v = \frac{(1+v)(1-2v)}{1-v} \tag{4}$$

11.3 If the wavelength is not a small fraction of the test specimen lateral dimensions, and instead is much larger than the specimen lateral dimensions, then the Young's modulus, $E$, is given by Eq 3 with $C_v$ set to one.

11.4 If the grain size of the carbon or graphite is greater than or about equal to the wavelength of the sonic pulse, the method may not be providing a Young's modulus value representative of the bulk material. Therefore, it would be preferable to test at a lower frequency (longer wavelength) to demonstrate that the sonic velocity is independent of frequency. Significant signal attenuation should be expected (and usually observed) when the grain size of the material is greater than or about equal to the wavelength of the transmitted sonic pulse.

11.5 If the test specimen is only a few grains thick, the acceptability of the test method's application should be demonstrated by initially performing measurements on a series of tests covering a range of test specimen lengths between the proposed test length and a test length incorporating sufficient grains to adequately represent the bulk material. However, it should be noted that increasing test specimen length on test specimens can lead to attenuation and dispersion of the sonic pulse and such effects may be more significant than issues relating to test specimen thickness and how representative the material over that length is of the bulk matrix.

11.6 Limitations on dimensions cannot be precisely specified as they will depend upon the properties of the material being tested. In order to satisfy the theory that supports Eq 3, as a guide, the specimen should have a diameter that is at least a factor two, and ideally a factor five, greater than the wavelength of sound in the material under test. It is good practice to run a series of tests with varying diameter to wavelength ratio to confirm the suitability of the test geometry. In practice, the length of the specimen will be determined taking account of the comments provided in 11.4 and 11.5.

11.7 The test method is satisfactory for test specimens greater than 6 mm length providing that the specimen diameter is greater than two wavelengths. For short test specimens it very important to use a measure of time of flight that is reproducible. The onset of the pulse can be difficult to define giving poor repeatability. A number of other methods are available for estimating the time of flight from the received wave signal including: *(1)* measurement of the position of a number of the first peaks and troughs (for example, the first two) to form an average; *(2)* measurement of the zero positions in the signal to form an average; *(3)* determination of the onset of a peak or a trough by the moment when a fraction (for example, 5%) of its amplitude is reached. It is the responsibility of the user to choose a method for estimating the time of flight. Where the frequency of the transmitted signal has changed significantly due to the attenuation of high frequency components in the specimen, the user should check that the chosen method provides adequate timing accuracy, by carrying out tests on a range of materials standards covering the full range of measurements. The test method used to determine the time of flight should be recorded as part of the measurement data.

11.8 In all cases, it is incumbent upon the operator to appropriately couple the specimen to the transducers. Combinations of grain size, pulse wave characteristics, length and test specimen geometry constraints generally affect the measured wave speed values. Proper coupling minimizes signal measurement errors, which are more detrimental to the overall recorded values in reduced-size (relatively thin) or high modulus (for example, irradiated) specimens due to the shorter time of flight. Subsequent increases in signal gain could have the detrimental

[3] The boldface numbers in parentheses refer to a list of references at the end of this standard.

effect of amplifying the inherent noise and reflected waves, rendering it more difficult to determine a proper (reliable) time of flight through pulse peak measurements.

11.9 When reporting times of flight and calculated values of Young's modulus by this test method, specimen length, diameter, transducer frequency and emitted frequency should also be quoted so that these important measurement parameters are available for review by users of the data.

11.10 Reference (3) provides a review of experimental and theoretical investigations into sample size effects on ultrasonic measurements of elastic moduli. The principal conclusions from this review were:

11.10.1 The test method in C769 assumes that graphite is an isotropic and homogeneous material. The degree of isotropy and homogeneity depends on graphite grade. For anisotropic graphite, Young's modulus depends on all Poisson's ratios and the exact relationship for the dynamic Young's modulus of interest must be derived starting with the elasticity matrix of the appropriate order. For non-homogeneous material, that is, for this application, microfine, medium and coarse grain, or oxidized graphites, frequency dependent attenuation of the sound wave through a specimen can significantly distort the signal leading to erroneous or inconsistent measurements. Consequently, it is strongly recommended that the transducer frequency is carefully selected for the range of Young's moduli or longitudinal velocities and the range of specimen lengths under investigation. Furthermore, the selection of the signal analysis method should take into account other material intrinsic properties, such as scattering and attenuation.

11.10.2 The test method in C769 assumes that the specimen can be considered an infinite medium, that is, the specimen lateral dimensions are much larger than the wavelength through the specimen ($D >> \lambda$). An extreme case analyzed numerically has shown that the pulse distortion is severe enough that the apparent wave speed depends considerably on sample length and on whether the center or edge of the sample contributes most to the electrical signal produced by the receiving transducer.

11.10.3 Based upon a large number of interlaboratory studies involving two or more laboratories undertaken on graphite specimens of various geometries and from different grades, discrepancies in measured velocity values between laboratories on the same specimens are generally below 5 %. Hence, the experimental results show that the effects discussed in this paper contribute an uncertainty of ±5 % in measured speed, corresponding to a ±10 % discrepancy in modulus on as-manufactured graphites. However, graphite elastic and attenuation properties change significantly after exposure in a reactor environment. Specifically, attenuation increases due to oxidation and dispersion becomes more significant due to irradiation hardening, which practically corresponds to increased velocities and wavelengths in the sample, making the $D/\lambda$ ratio smaller. Hence, the uncertainty due to the effects on such material is likely to be increased.

11.10.4 Since a size effect study on irradiated graphite is usually not feasible and interlaboratory studies are difficult due to the issues with international transport of nuclear material, it is recommended that, for such material, extreme care is taken when comparing dynamic Young's modulus data from different facilities with different experimental setups.

## 12. Bend Strength by Four- and Three-Point Loading (Test Methods C651 and D7972)

12.1 The dependence of bend strength (and other measures of strength) on specimen size is a complex subject and this section of the guide draws the attention of the user to some of the key issues that need to be considered when undertaking tests.

12.2 Four mechanisms for scale effects in the strength of a semi-brittle material with an amorphous multi-scale structure such as graphite can be identified.

12.2.1 The first arises from the trade-off between sources of energy that initiate (or propagate) a crack in relation to inelastic sinks of energy that inhibit crack growth in a specimen that is sufficiently large that edge defects associated with the fracture mechanism do not apply.

12.2.2 The second is when edge effects associated with the fracture mechanism are important. This is typical of small specimens but may also occur when a crack initiates at an edge of a relatively large specimen.

12.2.3 For both these mechanisms, it may be possible to treat the specimen as a continuum except possibly locally to the crack initiation site.

12.2.4 The third is when the specimen as a whole cannot be treated as a continuum. This will be the case for specimens that are at most a small multiple of the length scale of the largest amorphous structures in the graphite.

12.2.5 The fourth is when the micro-scale structure affects the strength properties of macro-scale specimens.

12.3 The fourth mechanism has been investigated in Reference (4) using Weibull theory and comparisons with published data. The conclusions from this study are:

12.3.1 There is a sound theoretical basis for the Weibull strength theory but there are limitations to its application. It does not apply to very small specimens, to regions of very high stress gradient, to small sample sizes and to hybrid populations of material. Three kinds of extrapolation (for gauge length, gauge cross-section and load configuration) were investigated and it was concluded that the validity of such extrapolations could only be tested by empirical testing.

12.3.2 Empirical evidence is presented to illustrate how the Weibull strength theory is able to correlate strength at different scales. In the context of bend strength, it was shown that the theory enables test data for specimens of an appropriate size to estimate strength at a larger scale. The reduction in mean bend strength due to the scale effect was slight and the location parameter (that is, minimum bend strength) was quite large.

12.3.3 Small specimen test results suggest that the smallest lateral dimension required to avoid micro-scale effects (in PGA graphite) is at least 10× the grain size. However, without understanding which particular feature of the microstructure is relevant, this finding may not be applicable to other graphites.

12.4 The continuum damage mechanics model has been used to simulate fracture in three-point bending tests (Reference (5)).

12.4.1 The size of the specimen affects the predicted fracture properties in flexural testing, with the effect depending on the fracture property being considered as well as on the size range of the specimens. The predicted bend strength decreased with an increase in specimen size, in agreement with experiment. The numerical study showed that the size effect was more pronounced for specimens of smaller sizes, whereas the opposite was true in experimental findings. The Weibull modulus showed an increasing trend with an increase in specimen size, indicating a reduction in the scatter of the results as the specimens became larger.

12.5 Three-point bend strength data compiled from a broad literature review in Reference (6) have been trended against stressed volume and minimum beam dimension to grain size ratio. It is observed that while some trends can be discerned, dependences may be confounded by other factors. Dependences need to be drawn from carefully designed test programs rather than by a global compilation of historical test data. Reference (6) also provides a review of bend test data for gilsocarbon IM1-24 graphite, to support testing of small ($6\ mm^3 \times 6\ mm^3 \times 19\ mm^3$) beams. The findings are summarized below:

12.5.1 A factor of 0.77 was needed to convert three-point bend strength data for a small beam (using roller bearings) to equivalent manufacturer's four-point bend strength data measured on $19\ mm^3 \times 19\ mm^3 \times 178\ mm^3$ beams with a support span of 140 mm (using fixed bearings) and a load span of 47.5 mm.

12.5.2 The sensitivity of four-point bend strength to support and load span was found not to be significant for a manufacturer's test beam geometry of $19\ mm^3 \times 19\ mm^3 \times 178\ mm^3$ for support spans in the range 114 mm to 140 mm and load spans in the range 38 mm to 70 mm.

12.5.3 Studies of the influence of specimen support on bend strength for both large beams in four-point bend and small beams in three-point bend showed the frictional contribution of fixed supports was significant and that tests should be conducted using roller bearings.

12.5.4 Bend tests on small beams may be influenced by shear stresses. For the beam dimensions selected for the reported test program, shear stresses were shown by finite element analysis to be low compared to the maximum stress at failure in flexure.

12.5.5 The standard formula from elementary beam bending theory to evaluate stress in a three-point bend test requires a correction for small beams. For a small beam, the theoretical correction would reduce the maximum stress by approximately 7 % assuming a span of 15 mm.

12.5.6 Experimental tests on small beams confirm the influence of span and span to beam depth ratio on stress. Corrections based upon theory, experiment and finite element modelling are in good agreement.

12.5.7 Experimental tests to investigate stress in three-point bend beams with and without overhang on the supports show a small reduction in elementary beam theory correction for an extreme case.

12.5.8 The effects of friction, contact area and finite beam width were investigated using finite element calculations. Based upon a friction coefficient between graphite and steel of 0.1, the ratio of maximum stress under unit force per width without and with friction in a small beam in three-point bend was ~1.06. Changing the application of force from a point at the centerline of the beam to a finite contact area had no significant effect on the same beam. The effect of finite beam width on maximum longitudinal stress was judged to be negligible.

12.5.9 The stressed region in a bent beam is much smaller than the stressed region in an equivalent size tensile stress specimen, leading to considerably higher measured strengths measured in flexure. For a small beam, the ratio of flexural to tensile strength was found to be 1.51. This ratio may depend on the size of the stressed region compared to the grain size, so would differ for different graphite types and different specimen geometries. The effect of stress gradient on crack propagation may also be a contributing factor.

12.5.10 The effect of specimen volume on bend strength of a small beam in three-point bend with a fixed support span was investigated for a range of beam cross-sections, showing flexural strengths to be the same within one standard deviation with no evidence for cliff-edge effects.

12.5.11 Application of Weibull statistics to three-point and four-point bend test data indicated that the change with volume is smaller than would be expected from the scatter in the data.

12.5.12 While these studies focused on a single graphite type and test geometry, the results should inform users on strength behaviour for other graphites and test geometries.

## 13. Linear Thermal Expansion of Solid Materials with a Push-Rod Dilatometer (Test Method E228)

13.1 Test Method E228 applies generally to rigid solid materials of (preferable) right cylinder or slab geometries. There is no graphite or carbon-specific ASTM standard for this measurement. Ideally, test specimens should be 25 mm to 60 mm long and 5 mm to 10 mm in diameter or of equivalent proportions (although there is no fundamental limitation provided the instrument controls the maximum thermal gradient to better than ±2 °C/50 mm). The specimen length should be such that the accuracy of determining the expansion $\Delta L/L_0$ is at least ±20 µm/m.

13.2 The technique uses a standard, single-tube, pushrod dilatometer to measure the thermal expansion of the material per degree of temperature increase, for a specific temperature range.

13.3 The dilatometer system measures and records pushrod displacement and test specimen temperature data during slow-rate heating and cooling cycles. The mean Coefficient of Linear Thermal Expansion (CTE) of a solid, for a temperature range between $T_0$ and $T_1$ can be calculated from the following formula:

$$CTE\left(10^{-6}\,K^{-1}\right) = \alpha_m = \frac{1}{L_0} \times \frac{\Delta L}{\Delta T} \tag{5}$$

where:

$L_0\,(m)$ = linear dimension of the solid at temperature $T_0$ (K),

$\Delta T = T_1 - T_0$ (K) = temperature range, and,
$\Delta L = L_1 - L_0$ (μm) = observed length change.

13.4 The test specimen must be placed in the holder carefully, so that it does not change position during the measurement. Similarly, push rod alignment with the specimen, especially when measuring curved faces (that is, across the diameter), is crucial to the quality and repeatability of the measurements.

13.5 The measuring components of the dilatometer are normally manufactured from silica. A silica standard, which should be of similar length to the graphite test specimen, provides a baseline for the thermal behavior of the instrument and the results are used to establish "offsets" for the specimen measurements.

13.6 Tungsten has a similar CTE to graphite. A tungsten standard provides a means of checking the accuracy of measurement of specimen temperature and pushrod movement for realistic pushrod movements over the real temperature range. The standard should be of similar length to the graphite test specimen.

13.7 Pushrod movements of fractions of a micrometre can be resolved using modern displacement transducers, however, two factors can limit the accuracy of measurement of ΔL. First, the pushrod may move against the specimen, so that pushrod displacements do not reflect specimen length change accurately; second, pushrod displacements will not be measured accurately unless the linear variable displacement transducer temperature is very stable. For example, adding more water to the cooling water circuit can produce transducer temperature changes and hence, significant changes in apparent test specimen length.

13.8 The principle described in Section 6 should be applied to specimens with lengths or diameters below the specified limits in the standard. Silica and tungsten standards matching precisely the geometries of the test specimens should be used to correct the results to account for the characteristics of the dilatometer (thermal behavior, temperature measurement and temperature control). Consideration should be given to the use of melt standards to establish furnace temperatures in the vicinity of the specimen. As specimen sizes decrease, consideration should be given to the location of thermocouples within the dilatometer relative to the specimen and their repositioning, if necessary, to ensure that appropriate readings are recorded.

13.9 Once a test regime for the desired specimen geometry has been established, it is good practice to run some of the specimens used in the qualification exercise as secondary standards at prescribed intervals through a measurement campaign. As appropriate, it is also a good practice to archive these secondary standards for equipment/procedure checks at later date.

13.10 For particularly short test specimens, attention should be paid to the influence of coke grains present on the contact faces. This caution is also more significant if the test material is anisotropic.

## 14. Thermal Diffusivity by the Flash Method (Test Method E1461)

14.1 Test Method E1461 covers the determination of thermal diffusivity in isotropic solid materials by measuring the heat flow through thin circular disks. By pulsing radiant energy onto the surface of the specimen, the absorption of energy on the front surface and resulting back face temperature rise gives an indication of the flow of heat through the material. Assuming adiabatic one-dimensional heat transfer, the thermal diffusivity, α, is calculated by measuring the specimen thickness and the time for the rear face temperature to reach one-half of its maximum value:

$$\alpha = 0.3879L^2/t_{1/2} \quad (6)$$

where:

$L$ = specimen thickness, m, and
$t_{1/2}$ = half rise time, s.

14.2 The standard as published is limited in "special cases" to diameters as small as 6 mm and as large as 30 mm. Specimen diameter is generally limited by the apparatus used.

14.3 Thicknesses are listed as typically in the 1 mm to 6 mm range. The specimen thickness is of special consideration when nonstandard specimen sizes are used. The optimum thickness depends upon the magnitude of thermal diffusivity, and should be chosen such that the time to reach half of the maximum temperature falls within the 10 to 1000 ms range. Heat loss from the specimen is typically accounted for by models applied to the data, however these models are limited and specimens with a diameter to thickness ratio of less than 4 may have more heat loss at the specimen's circumference than can be accounted for in typical models. The accuracy of the rise time can fall below acceptable limits if heat loss through the sides of the specimen becomes a significant detriment to the heat values measured on the back face of the specimen. This effect is likely to be more of concern with large specimens rather than specimens constrained to small geometries.

14.4 An additional consideration for small specimen sizes is the number of grains being captured and the effect of grain boundaries on the heat flow characteristic of the material. The operator should be familiar with these characteristics by recording measurements for a number of test specimens with progressively larger thicknesses based on the largest known grain diameter and number of grains captured in each thickness. The competing effects of grain capture and material homogeneity will be counteracted by the heat loss through the sides of progressively thicker specimens, so all dimensions should be reported when performing scoping studies that evaluate the progressive effects of specimen size.

14.5 Reference (7) provides a detailed assessment of specimen size effects associated with the laser flash method. The principal conclusions from this study were:

14.5.1 Three radiation heat loss models (Cowan, Cape-Lehman, and Clark-Taylor) were tested and found to perform equally over the temperature range 25 °C to 1000 °C but their ability to correct for radiative heat loss was limited.

14.5.2 The Cowan model was selected for further investigation and it was determined that accurate heat loss corrections could be made for specimens with a diameter to length ratio greater than 2.

14.5.3 There are multiple parameters to consider when assessing limits on minimum specimen thickness. For larger grain graphites, measured diffusivity became unreliable as the specimen thickness approached the length scale of the microstructure. There may be relatively small grain graphites that do no exhibit any correlation between measured diffusivity and specimen thickness. In these cases, using a propagation of error analysis to establish uncertainty that is dominated by the finite laser pulse width to half rise time ratio can give guidance on minimum specimen thickness that is based on an acceptable measurement uncertainty.

14.5.4 As the specimen temperature rises, phenomenon scattering becomes the dominant resistance to heat transfer and the change in diffusivity related to microstructural resistances is insignificant. The temperature at which even larger grain graphites exhibit no correlation between specimen thickness and measured diffusivity was found to be ~400 °C.

## 15. Keywords

15.1 carbon; coefficient of thermal expansion; density; graphite; resistivity; resonance; sonic; tensile stress-strain; thermal diffusivity; velocity; Young's Modulus

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## SUMMARY OF CHANGES

Subcommittee D02.F0 has identified the location of selected changes to this standard since the last issue (D7775 – 11 (2015)) that may impact the use of this standard. (Approved Dec. 15, 2016.)

*(1)* Revised subsection 3.1, Section 5, and Section 11, including adding new subsection 11.10 and subsections.

*(2)* Added new Section 12.

*(3)* Added new subsection 14.5.

*(4)* Added new References section.