INTERNATIONAL

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Standard 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[1]

This standard is issued under the fixed designation D8356; 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 test method covers a procedure for measuring the longitudinal and transverse (shear) sonic velocities in manufactured carbon and graphite which can be used to obtain approximate values for the elastic constants: Young's modulus (E), the shear modulus (G), and Poisson's ratio (v).

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

C747 Test Method for Moduli of Elasticity and Fundamental Frequencies of Carbon and Graphite Materials by Sonic Resonance

D4175 Terminology Relating to Petroleum Products, Liquid Fuels, and Lubricants

D6300 Practice for Determination of Precision and Bias Data for Use in Test Methods for Petroleum Products, Liquid Fuels, and Lubricants

D7775 Guide for Measurements on Small Graphite Specimens

E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method

IEEE/ASTM SI 10 Standard for Use of the International System of Units (SI) (the Modern Metric System)

3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

3.1.1 *end correction time (T_e), n*—a fixed correction time associated with the potential interaction of the couplant medium and the test material.

3.1.2 *longitudinal sonic pulse, n*—a sonic pulse in which the displacements are in the direction of propagation of the pulse.

3.1.3 *pulse travel time, (T_t), n*—the total time, measured in seconds, required for the sonic pulse to traverse the specimen being tested, and for the associated electronic signals to traverse the transducer coupling medium and electronic circuits of the pulse-propagation system.

3.1.4 *shear or transverse sonic pulse, n*—a sonic pulse in which the displacements are perpendicular to the direction of propagation of the pulse.

3.1.5 *time of flight (ToF), n*—the total time, measured in seconds, required for the sonic pulse to traverse the specimen being tested ($T_t – T_0$).

[1] This test method 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.

3.1.6 *zero time, (T_0), n*—the travel time (correction factor), measured in seconds, associated with the transducer, coupling medium and electronic circuits in the pulse-propagation system.

4. Summary of Test Method

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

$$E = C_v \rho V_L^2 \quad (1)$$

where:

E = Young's modulus, Pa,
ρ = density, kg/m^3,
V_L = longitudinal signal velocity, m/s, and
C_v = Poisson's factor.

4.2 The Poisson's factor, C_v, is related to Poisson's ratio, v, by the equation:

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

4.3 If the Poisson's factor is unknown, it can be assumed as an approximation of the method. For nuclear graphites a typical Poisson's ratio of 0.2 corresponds to a Poisson's factor of 0.9.

4.4 If the wavelength is not a small fraction of the samples lateral dimensions, and instead is much larger than the specimens lateral dimensions, then Young's modulus, E, is given by Eq 1 with C_v set to unity rather the being determined by Eq 2.

4.5 The shear velocity can be obtained from Eq 3:

$$G = \rho V_S^2 \quad (3)$$

where:

G = shear modulus, Pa,
ρ = density, kg/m^3, and
V_S = shear signal velocity, m/s.

4.6 All of the elastic constants for an isotropic material can be calculated from two wave velocities, the longitudinal wave velocity and the shear wave velocity and the material density.[5] In particular,

$$\text{Poisson's ratio, } v = \frac{1 - \left[2\left(\frac{V_S}{V_L}\right)^2\right]}{2 - \left[2\left(\frac{V_S}{V_L}\right)^2\right]} \quad (4)$$

where V_S and V_L are the measured shear and longitudinal wave velocities (m/s) (see Eq 5 and Eq 6).

4.7 The shear modulus is given by Eq 3 and a Young's modulus, E, can be obtained from Eq 1 and Eq 2 with C_v calculated using the value of Poisson's ratio from Eq 4.

5. Significance and Use

5.1 Sonic velocity measurements are useful for comparing materials with similar elastic properties, dimensions, and microstructure.

5.2 Eq 1 provides an accurate value of Young's modulus only for isotropic, non-attenuative, non-dispersive materials of infinite dimensions. For non-isotropic graphite Eq 1 can be modified to take into account the Poisson's ratios in all directions. As graphite is a strongly attenuative material, the value of Young's modulus obtained with Eq 1 will be dependent on specimen length. If the specimen lateral dimensions are not large compared with the wavelength of the propagated pulse, then the value of Young's modulus obtained with Eq 1 will be dependent on the specimen lateral dimensions. The accuracy of the Young's modulus calculated from Eq 1 will also depend upon uncertainty in Poisson's ratio and its impact on the evaluation of the Poisson's factor in Eq 2. However, a value for Young's modulus Eq 1 or Eq 7) can be obtained for many applications, which is often in good agreement with the value obtained by other more accurate methods, such as in Test Method C747. The technical issues and typical values of corresponding uncertainties are discussed in detail in STP 1578.[6]

5.3 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 provide a value of the Young's modulus representative of the bulk material. Therefore it would be desirable to test a lower frequency (longer wavelength) to demonstrate that the range of obtained velocity values are within acceptable levels of accuracy. Significant signal attenuation should be expected when grain size of the material is greater than or about equal to the wavelength of the transmitted sonic pulse or the material is more porous than would be expected for as-manufactured graphite.

NOTE 1—Due to frequency dependent attenuation in graphite, the wavelength of the sonic pulse through the test specimen is not necessarily the same wavelength of the transmitting transducer.

5.4 If the sample is only a few grains thick, the acceptability of the method's application should be demonstrated by initially performing measurements on a series of dummy specimens covering a range of lengths between the proposed test specimen's length and a specimen length incorporating sufficient grains to adequately represent the bulk material.

6. Apparatus

6.1 *Driving Circuit,* consisting of an ultrasonic pulse generator.

6.1.1 The user should select a pulse frequency to suit the material microstructure and specimen elastic properties and

[3] Schreiber, Anderson, and Soga, *Elastic Constants and Their Measurement*, McGraw-Hill Book Co., 1221 Avenue of the Americas, New York, NY 10020, 1973.

[4] *American Institute of Physics Handbook*, 3rd ed., McGraw-Hill Book Co., 1221 Avenue of the Americas, New York 10020, 1972, pp. 3–98ff.

[5] Blessing, G. V., "The Pulsed Ultrasonic Velocity Method for Determining Material Dynamic Elastic Moduli," *Dynamic Elastic Modulus Measurement in Materials*, ASTM STP 1045, A Wolfenden, Ed., American Society for Testing and Materials, Philadelphia, PA 1990.

[6] ASTM Selected Technical Papers, STP 1578, *Graphite Testing for Nuclear Applications: The Significance of Test Specimen Volume and Geometry and the Statistical Significance of Test Specimen Population*, 2014, edited by Tzelepi and Carroll.

dimensions being tested. High frequencies are attenuated by carbon and graphite materials and, while typical practicable frequencies lie in the range 0.5 MHz to 2.6 MHz, the user may show that frequencies outside this range are acceptable.

6.2 *Transducer,* input, with suitable coupling medium (see 8.8.1 and Note 4).

6.3 *Transducer,* output, with suitable coupling medium (see 8.8.1 and Note 4).

6.4 *Computer,* with analogue to digital converter, or oscilloscope, and external trigger from driving circuit.

6.5 See Fig. 1 for a schematic of a typical test set-up.

NOTE 2—Some manufacturers combine items 6.1 and 6.4 into a single package with direct time readout. Such apparatus is acceptable and can operate satisfactorily provided the frequency of the propagated pulse is already known in order to check that wavelength requirements for the method are satisfied.

6.6 Constant transducer pressure is advisable for velocity determinations, especially shear-wave velocity measurements. This may be achieved through operator skill, but a constant force device such as a spring-loaded fixture is preferred. The transducers should be kept aligned to one another for consistency. Moreover, it is recommended that the recorded velocity value be the mean of at least three consistent measurements.

6.7 *Identification of the Pulse Onset*—The signal onset can be located by using the trace expansion facility available on most PC/oscilloscope interfaces or it can be taken as a fixed percentage of the amplitude of the fist peak of the received signal, for example, 5 %. Alternately, the back slope of the first peak of the received signal may be used. The selected method for determining signal onset should be applied consistently. See also the text in Section 8.

6.8 *Determination of Velocity*—See also the text in Section 8.

7. Test Specimens

7.1 *Selection and Preparation of Specimens*—Take special care to obtain representative specimens that are straight, uniform in cross section, and free from extraneous liquids. The specimen end faces shall be perpendicular to the specimen cylindrical surface to within 0.125 mm total indicator reading.

7.2 *Measurement of Weight and Dimensions*—Determine the weight and the mean specimen dimensions per Test Method C559.

7.2.1 For samples outside the specification of Test Method C559, follow the guidance of Guide D7775.

7.3 *Limitations on Dimensions*—These cannot be precisely specified as they will depend on the properties of the material being tested. In order to satisfy the theory that supports Eq 1, as a guide, the specimen should have a diameter (circular section specimens) or lateral dimensions (width, thickness) for rectangular section specimens that is at least a factor of five, greater than the wavelength of sound in the material under test. In practice the length of the specimen will be determined taking account of the comments in 5.3 and 5.4.

7.4 *Limitations on Ultrasonic Pulse Frequency*—Generally speaking, a better accuracy of Time of Flight (ToF) will be obtained at higher frequencies. However, attenuation increases at higher frequencies leading to weak and distorted signals.

NOTE 3—Transducer frequencies of 0.5 MHz to 5 MHz (depending upon the texture of the graphite under test) have been observed to yield satisfactory results.

8. Procedure

8.1 Condition specimens by washing in ethanol and drying the specimens at a minimum temperature of 110 °C for 2 h minimum followed by cooling in a desiccator.

8.2 Determine the specimen dimensions, mass, and bulk density according to Test Method C559 and considering the limitations in 7.1, 7.2, and 7.3.

8.3 For any given apparatus and choice of coupling medium, it is necessary to follow procedures to quantify the zero time T_0, and end correction time, T_e. Correction factor T_0 will be dependent upon the type of transducers and their performance over time should be regularly checked. T_0 must be

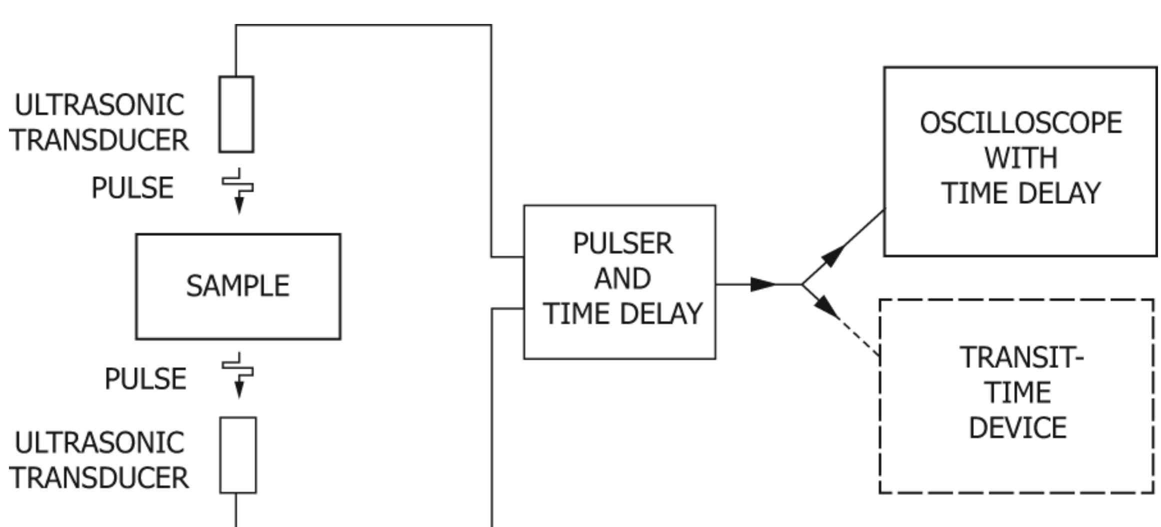

FIG. 1 Schematic of the Basic Experimental Arrangement for the Ultrasonic Pulsed Wave Transit Time Technique

re-quantified if the test set up is changed. T_e should be small (maybe too small to detect) and reflect the interaction between the coupling medium and the test material.

8.3.1 Determine whether the end correction time, T_e, is evident in the Time of Flight (ToF) measured on various length samples taken from a single bar. As modulus is likely to vary from sample to sample the recommended approach is to continually bisect a long rod, measuring each bi-section, until the required lower limit is reached. The end correction time, T_e, is obtained from a regression fit to the graph of ToF versus sample length.

8.4 Connect the apparatus as shown in Fig. 1, and refer to the manufacturer's instructions for setup precautions. Allow adequate time for equipment warm-up and stabilization.

8.5 Bring transducer faces into intimate contact but do not exceed manufacturer's recommended contact pressures.

8.6 Follow the vendor's instructions to adjust the instrumentation to match the transducer frequency to give good visual amplitude resolution.

8.7 Determine T_0, the travel time (zero correction) measured in seconds, associated with the electronic circuits in the pulse-propagation instrument and coupling (Fig. 2(a)). Ensure that the repeatability of the measurement is of sufficient precision to meet the required accuracy in Young's modulus.

8.8 Position the selected transducers on either ends of the specimen with a constant applied load. Obtain a stable signal trace for the sound wave in the material and capture the trace (if possible).

8.8.1 A coupling medium may be necessary to improve transmission of the sonic signal. In this case, apply a light coating of the coupling media to the faces of the test specimen that will contact the transducers. Alternatively, "soft rubber" tipped transducers can be effective if a fully noninvasive measurement is needed.

NOTE 4—The following coupling media may also be used: hydroxyethyl cellulose, petroleum jelly, high vacuum grease and water-based ultrasonic couplants. However these may be difficult to remove subsequently. Distilled water can provide a very satisfactory coupling medium without significant end effects, and surface water may be removed subsequently by drying. Manufacturers offer soft rubber-tipped transducers suitable for noninvasive measurements. With these transducers either good probe loading control or accurate determination of the soft rubber length is essential during measurement if good reproducibility is to be achieved.

8.9 Adjust the gain of electronic components to give good visual amplitude resolution and adjust the moveable cursers (if such a facility is available) to acquire the time of flight, T_t (see Fig. 2). The display expansion facility is useful in this regard.

8.10 For short samples it is 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 the peaks and troughs of the first two waves to form an average, (*2*) measurement of the zero positions in the signal to form an average and (*3*) determining the onset of a peak or trough by the moment when a fraction (for example, 5 %) of its amplitude is reached or (*4*) alternatively, the back

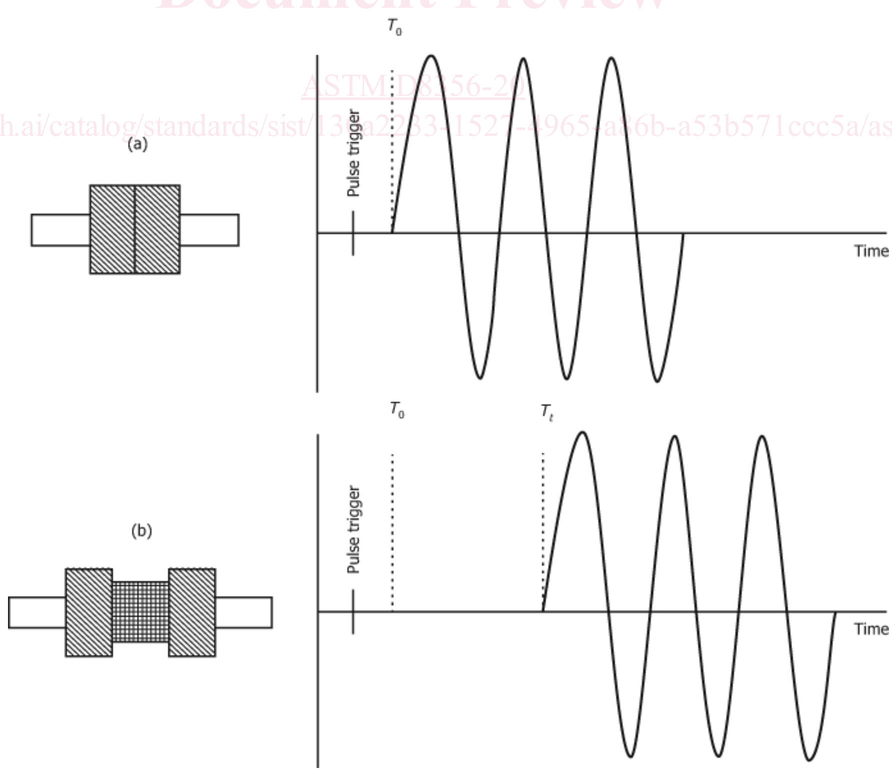

FIG. 2 Schematic Illustrating (a) Zero Time (T_0) Measurement for Face to Face Contact Between Transducers, and (b) Pulse Travel Time (T_t) Measurement ($T_t - T_0$) for the Sample Positioned Between the Transducers, based upon a Simplified Received Wave Signal and the Idealized Case where the Onset of the First Peak has been Detected

slope of the first peak of the received signal may be used. It is the responsibility of the user to choose a method for consistently and reproducibly estimating the time of time of flight. The selected signal onset should be applied consistently. Where the frequency of the transmitted signal has changed significantly due to attenuation of high frequency components in the specimen, the user should check that the chosen method provides adequate timing accuracy. The method used to determine the time of flight should be recorded as part of the measurement data.

8.11 The use of a shear couplant is recommended to obtain a stable, clear shear wave trace (see 6.6).

8.12 For shear velocity determinations, after determining $V_S(0°)$ rotate the specimen through 90° about its length axis and repeat 8.9 to determine $V_S(90°)$. Calculate V_S. Rotate another 90° about its length and repeat 8.9. Record the value of $V(180°)$ but do not use it in the calculation of the mean shear velocity (this value for comparison only). Thus two shear wave velocities will be determined, $V_s(0°)$ and $V_s(90°)$, each the mean of at least three measurements. The shear velocity, V_s, is the mean of $V_s(0°)$ and $V_s.(90°)$.

8.13 Clean specimen of couplant and replace transducers with longitudinal velocity transducers.

8.14 Repeat 8.3 – 8.7 and hence acquire longitudinal trance, time of flight, T_t, and longitudinal velocity, V_L.

8.15 For longitudinal wave measurements repeat the pulse-measurement until at least three consistent traces are obtained (recorded) and at least three consistent velocities are obtained. The orientation of the transducers relative to one another should be maintained between measurements.

8.16 Report data in accordance with Section 10.

NOTE 5—8.7 may vary according to the experimental apparatus used (8.8). Determine the corrected Time of Flight from the scope traces. Ensure that the repeatability of the measurement is of sufficient precision to meet the required accuracy in the Elastic constants.

8.17 It is good practice to monitor the performance and reproducibility of the sonic velocity equipment by periodically testing a reference sample of similar material and geometry to that typically used by the operator. This will monitor drift arising from deterioration in transducer performance. The accuracy of absolute velocity measurement can be checked by using certified standards calibrated using a method such as the resonant bar technique (Test Method C747). Standards need to be representative of the material being tested and have a similar geometry.

8.18 As the values of elastic constants obtained with this test method depend on the experimental setup and on specimen dimensions, microstructure, and elastic properties, agreement between two laboratories on a single geometry or one material does not ensure agreement on other geometries or other materials.

8.19 As the values of elastic constants obtained with this test method depend on specimen dimensions, microstructure, and elastic properties, validation of the technique for a certain geometry and material does not ensure the validity of the technique once the specimen elastic properties change due to environmental conditions (due to irradiation or oxidation, for example).

9. Calculation

9.1 *Velocity of Signal (Longitudinal or Transverse)*—For longitudinal wave:

$$V_L = \frac{L}{T_t - T_e - T_0} \tag{5}$$

where:

V_L = longitudinal signal velocity, m/s,
L = specimen length, mm,
T_t = Time of Flight, s,
T_e = end correction time, s, and
T_0 = zero time, s.

For shear wave:

$$V_S = \frac{L}{T_t - T_e - T_0} \tag{6}$$

where:

V_L = longitudinal signal velocity, m/s,
L = specimen length, mm,
T_t = Time of Flight, s,
T_e = end correction time, s, and
T_0 = zero time, s.

In the event that only longitudinal measurements can be performed, an approximate Young's Modulus may be calculated from Eq 7.

$$E_{approx} = 0.9\rho V_L^2 \tag{7}$$

where:

E_{approx} = Young's modulus, Pa (approximate),
ρ = density, kg/m^3, and
V_L = longitudinal signal velocity, m/s.

9.2 Since graphites are not necessarily isotropic, the value of Young's modulus cannot be determined solely from a velocity measurement in one direction. However, an approximate Young's modulus for each direction may be obtained using Eq 7 (based upon an assumed Poisson's ratio of 0.2). More accurate estimates of the Young's moduli require the determination of the full compliance matrix from a set of measurements of longitudinal and shear wave velocities perpendicular to one another (9.1).

9.3 *Conversion Factors*—See IEEE/ASTM SI 10.

9.4 *Elastic Constants*—Shear modulus, Poisson's ratio, and Young's modulus (Poisson's ratio corrected).

9.4.1 Calculate G, v, and $E_{(\text{Poisson's ratio corrected})}$ from Eq 1-4.

10. Report

10.1 The report shall include the following:

10.1.1 Specimen dimensions, weight, and test specimen orientation with respect to forming direction;

10.1.2 Sonic velocities for each specimen;

10.1.3 The method used to determine the time of flight including details of the signal onset used;

10.1.4 Density of each specimen, if calculated;

10.1.5 The elastic constants of each specimen, if calculated;