Designation: C 1548 – 02 (Reapproved 2007)

Standard Test Method for Dynamic Young's Modulus, Shear Modulus, and Poisson's Ratio of Refractory Materials by Impulse Excitation of Vibration[1]

This standard is issued under the fixed designation C 1548; 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 the measurement of the fundamental resonant frequencies for the purpose of calculating the dynamic Young's modulus, the dynamic shear modulus (also known as the modulus of rigidity), and the dynamic Poisson's ratio of refractory materials at ambient temperatures. Specimens of these materials possess specific mechanical resonant frequencies, which are determined by the elastic modulus, mass, and geometry of the test specimen. Therefore, the dynamic elastic properties can be computed if the geometry, mass, and mechanical resonant frequencies of a suitable specimen can be measured. The dynamic Young's modulus is determined using the resonant frequency in the flexural mode of vibration and the dynamic shear modulus is determined using the resonant frequency in the torsional mode of vibration. Poisson's ratio is computed from the dynamic Young's modulus and the dynamic shear modulus.

1.2 Although not specifically described herein, this method can also be performed at high temperatures with suitable equipment modifications and appropriate modifications to the calculations to compensate for thermal expansion.

1.3 The values are stated in SI units and are to be regarded as the standard.

1.4 *This standard may involve hazardous materials, operations, and equipment. 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]

C 71 Terminology Relating to Refractories

C 215 Test Method for Fundamental Transverse, Longitudinal, and Torsional Resonant Frequencies of Concrete Specimens

C 885 Test Method for Young's Modulus of Refractory Shapes by Sonic Resonance

C 1259 Test Method for Dynamic Young's Modulus, Shear Modulus, and Poisson's Ratio for Advanced Ceramics by Impulse Excitation of Vibration

3. Summary of Test Method

3.1 The fundamental resonant frequencies are determined by measuring the resonant frequency of specimens struck once mechanically with an impacting tool. Frequencies are measured with a transducer held lightly against the specimen using a signal analyzer circuit. Impulse and transducer locations are selected to induce and measure one of two different modes of vibration. The appropriate resonant frequencies, dimensions, and mass of each specimen may be used to calculate dynamic Young's modulus, dynamic shear modulus, and dynamic Poisson's ratio.

4. Significance and Use

4.1 This test method is non-destructive and is commonly used for material characterization and development, design data generation, and quality control purposes. The test assumes that the properties of the specimen are perfectly isotropic, which may not be true for some refractory materials. The test also assumes that the specimen is homogeneous and elastic. Specimens that are micro-cracked are difficult to test since they do not yield consistent results. Specimens with low densities have a damping effect and are easily damaged locally at the impact point. Insulating bricks can generally be tested with this technique, but fibrous insulating materials are generally too weak and soft to test.

4.2 For quality control use, the test method may be used for measuring only resonant frequencies of any standard size specimen. An elastic modulus calculation may not be needed or even feasible if the shape is non-standard, such as a slide gate plate containing a hole. Since specimens will vary in both size and mass, acceptable frequencies for each shape and material must be established from statistical data.

[1] This test method is under the jurisdiction of ASTM Committee C08 on Refractories and is the direct responsibility of Subcommittee C08.01 on Strength.

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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.

4.3 Dimensional variations can have a significant effect on modulus values calculated from the frequency measurements. Surface grinding may be required to bring some materials into the specified tolerance range.

4.4 Since cylindrical shapes are not commonly made from refractory materials they are not covered by this test method, but are covered in Test Method C 215.

5. Apparatus

5.1 *Electronic System*—The electronic system in Fig. 1 consists of a signal conditioner/amplifier, a signal analyzer, a frequency readout device, and a signal transducer for sensing the vibrations. The system should have sufficient precision to measure frequencies to an accuracy of 0.1 %. Commercial instrumentation is available which meets this requirement.[3]

5.1.1 *Frequency Analyzer*—This consists of a signal conditioner/amplifier to power the transducer and a digital waveform analyzer or frequency counter with storage capability to analyze the signal from the transducer. The waveform analyzer shall have a sampling rate of at least 20 000 Hz. The frequency counter should have an accuracy of 0.1 %.

5.1.2 *Sensor*—A piezoelectric accelerometer contact transducer is most commonly used, although non-contact transducers based on acoustic, magnetic, or capacitance measurements may also be used. The transducer shall have a frequency response in the range of 50 Hz to 10 000 Hz, and have a resonant frequency above 20 000 Hz. The sensor shall have a mark identifying the maximum sensitivity direction so that it can be properly oriented for each vibration mode.

5.2 *Impactor*—Because refractory materials are tested with specimens of various sizes, it is not feasible to specify an impactor with a specific size, weight, or construction method. However, hammer style impactors which have light weight handles with the impacting mass concentrated near the end are preferred to dropping vertical impactors. Steel hammer style impactors, with head weights between 0.3 and 3 % of the specimen weight, are recommended. To avoid damaging the surface of insulating bricks or other weak materials, plastic or rubber shapes should be substituted for the steel impactors.

[3] Equipment found suitable is available from J. W. Lemmons, Inc., 3466 Bridgeland Drive, Suite 230, St. Louis, MO 63044-260.

5.3 *Specimen Support*—The support shall permit the specimen to vibrate freely without restricting the desired mode of vibration. For room temperature measurements, soft rubber or plastic strips located at the nodal points are typically used. Alternately, the specimen can be placed on a thick soft rubber pad. For elevated temperature measurements, the specimen may be suspended from support wires wrapped around the specimen at nodal points and passing vertically out of the test chamber.

6. Test Specimen

6.1 *Preparation*—Test specimens shall be prepared to yield uniform rectangular shapes. Normally, brick sized specimens are used. Although smaller bars cut from bricks are easily tested for flexural resonant frequencies, it is more difficult to obtain torsional resonance in specimens of square cross-section. Some pressed brick shapes are dimensionally uniform enough to test without surface grinding, but specimens cut from larger shapes or prepared by casting or other means often require surface grinding of one or more surfaces to meet the dimensional criteria noted below.

6.2 *Heat Treatment*—All specimens shall be prefired to the desired temperature and oven dried before testing.

6.3 *Dimensional Ratios*—Specimens having either very small or very large ratios of length to maximum transverse dimensions are frequently difficult to excite in the fundamental modes of vibration. Best results are obtained when this ratio is between 3 and 5. For use of the equations in this method, the ratio must be at least 2.

6.4 *Dimensional Uniformity*—Rectangular specimens shall have surfaces that are flat and parallel to within ± 0.5 % of the nominal measured value.

6.5 *Weight (or Mass) and Dimensions*—Determine the weight (or mass) to the nearest ± 0.5 %. Measure each dimension to within ± 0.5 %.

7. Measurement of Impulse Resonant Frequencies

7.1 *Transverse Frequency*:

7.1.1 Support the specimen so that it may vibrate freely in the fundamental transverse mode. In this mode the nodal points (where the displacement is zero) are located at 0.224L from each end, where L is the specimen length. Vibrational displacements are a maximum at the ends of the specimen and about

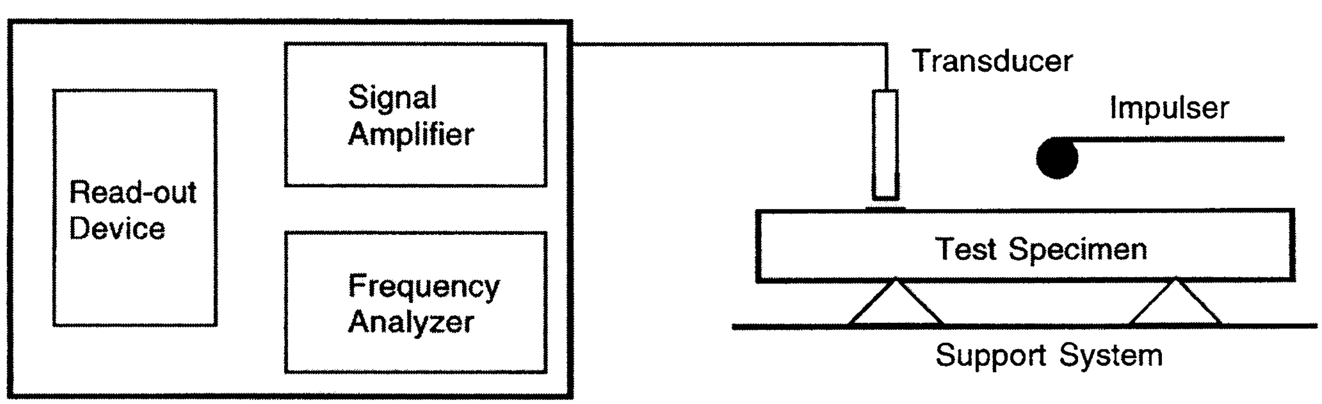

FIG. 1 Diagram of Test Apparatus

3/5 maximum at the center. The nodal points are shown in Fig. 2 along with recommended impact points and sensor locations. If the specimen does not have a square cross-section, support the specimen on its largest face such that it vibrates perpendicular to its thinnest dimension.

7.1.2 Turn on the electronic system and warm it up according the manufacturers instructions.

7.1.3 Position the sensor on the side face of the specimen at mid length, with the sensor oriented such that the most sensitive pick-up direction coincides with the vibration direction. In Fig. 2, the dot on the sensor indicates the most sensitive pickup direction of the sensor and it is pointed upward toward a top-center impact point. The sensor is typically held against the specimen with very light hand pressure, but some types could be temporarily attached to large specimens.

7.1.4 Select an impact hammer with a head weight 0.3 to 3 % of the specimen weight and lightly tap the top center of the specimen perpendicular to the surface. Note the reading displayed by the electronic system, allow a few seconds for existing vibrations to dampen in the specimen, and repeat the procedure at least 3 times until a consistent value is reproduced. Record that value and calculate the resonant frequency

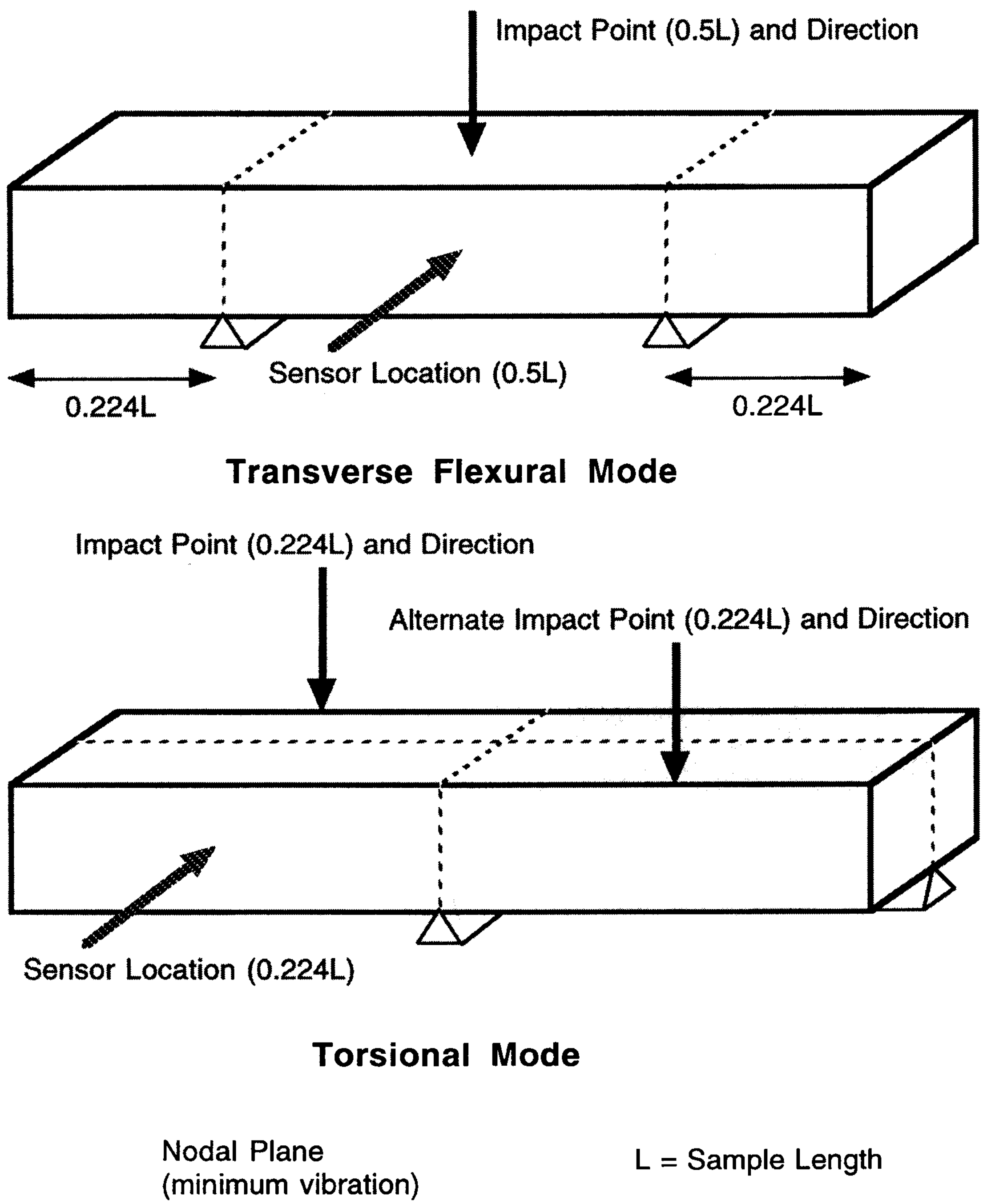

FIG. 2 Impact Points and Transducer Locations

from it per the manufacturer's instructions if frequency is not displayed directly. If a consistent value cannot be obtained, either the specimen is damaged or other modes of vibration are interfering with the measurement.

7.2 *Torsional Frequency*

7.2.1 Support the specimen so that it may vibrate freely in torsion. In this mode there is a single nodal point at the center and vibrations are a maximum at the ends. The impact and sensor pickup points are located at 0.224L from the ends. This location is a nodal point for flexural vibration and minimizes interference from flexural vibrations.

7.2.2 Turn on the electronic system and warm it up according to the manufacturers instructions.

7.2.3 Position the sensor on the side face of the specimen at 0.224L, with the sensor oriented such that the sensitive pick-up direction coincides with the vibration direction. In Fig. 2, the dot on the sensor indicates the most sensitive pickup direction of the sensor and it is pointed upward toward a top impact point.

7.2.4 Select an impact hammer with a head weight 0.3 to 3 % of the specimen weight and lightly tap the top of the specimen at a 0.224L location perpendicular to the surface. Note the reading displayed by the electronic system, allow a few seconds for existing vibrations to dissipate, and repeat the process at least 3 times until a consistent value is reproduced. Record that value and calculate the resonant frequency from it if frequency is not displayed directly.

8. Calculations

8.1 *Dynamic Young's Modulus*[4,5]*:*

8.1.1 From the fundamental flexural vibration of a rectangular bar:

$$E = 0.9465 \left(\frac{m f_f^2}{b}\right)\left(\frac{L^3}{t^3}\right) T_1 \quad (1)$$

where:

E = Young's modulus, Pa,
m = mass of the bar, g,
b = width of the bar, mm,
L = length of the bar, mm,
t = thickness of the bar, mm,
f_f = fundamental resonant frequency of the bar in flexure, Hz, and
T_1 = correction factor for fundamental flexural made to account for finite thickness of bar, Poisson's ratio, etc.

$$T_1 = 1 + 6.585\,(1 + 0.0752\mu + 0.8109\mu^2)\left(\frac{t}{L}\right)^2 - 0.868\left(\frac{t}{L}\right) - \frac{\left\{\left(8.340\,(1 + 0.2023\mu + 2.173\mu^2)\left(\frac{t}{L}\right)^4\right)\right\}}{\left\{\left(1.000 + 6.338\,(1 + 0.1408\mu + 1.536\mu^2)\left(\frac{t}{L}\right)^2\right)\right\}}$$

μ = Poisson's ratio.

8.1.1.1 If $L / t \geq 20$, T_1 can be simplified to:

$$T_1 = \left(1.000 + 6.585\left(\frac{t}{L}\right)^2\right)$$

and E can be calculated directly.

8.1.1.2 If $L / t < 20$, then an initial Poisson's ratio must be assumed to start the computations. An iterative process is then used to determine a value of Poisson's ratio, based on experimental Young's modulus and shear modulus. This iterative process is shown in Fig. 3 and described below.

(1) Determine the fundamental flexural and torsional resonant frequencies of the rectangular test specimen. Using Eq 2, the dynamic shear modulus of the test specimen is calculated from the fundamental torsional resonant frequency and the dimension and mass of the specimen.

(2) Using Eq 1, the dynamic Young's modulus of the rectangular test specimen is calculated from the fundamental flexural resonant frequency, the dimensions and mass of the specimen, and the initial/iterative Poisson's ratio.

(3) The dynamic shear modulus and Young's values modulus.calculated in steps (*1*) and (*2*) are substituted into Eq 3, for Poisson's ratio. A new value for Poisson's ratio is then calculated for another iteration starting at step (*2*).

(4) Steps (*2*) and (*3*) are repeated until no significant difference (2 % or less) is observed between the last iterative value and the final computed value of the Poisson's ratio. Self-consistent values for the moduli are thus obtained.

8.2 *Dynamic Shear Modulus*[6,7]*:*

8.2.1 From the fundamental torsional vibration of a rectangular bar:

$$G = \left\{\frac{(4\,L\,m\,f_t^2)}{(b\,t)}\right\}\left\{\frac{B}{(1 + A)}\right\} \quad (2)$$

where:

G = shear modulus, Pa, and
f_t = fundamental resonant frequency of the bar in torsion, Hz.

$$B = \frac{\left\{\left(\frac{b}{t}\right) + \left(\frac{t}{b}\right)\right\}}{\left\{4\left(\frac{t}{b}\right) - 2.52\left(\frac{t}{b}\right)^2 + 0.21\left(\frac{t}{b}\right)^6\right\}}$$

$$A = \frac{\left\{0.5062 - 0.8776\left(\frac{b}{t}\right) + 0.3504\left(\frac{b}{t}\right)^2 - 0.0078\left(\frac{b}{t}\right)^3\right\}}{\left\{12.03\left(\frac{b}{t}\right) + 9.892\left(\frac{b}{t}\right)^2\right\}}$$

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[4] Spinner, S., Reichard, T. W., and Tefft, W. E., "A Comparison of Experimental and Theoretical Relations Between Young's Modulus and the Flexural and Longitudinal Resonance Frequencies of Uniform Bars," *Journal of Research of the National Bureau of Standards—A. Physics and Chemistry*, Vol 64A, #2, March-April, 1960.

[5] Spinner, S., and Tefft, W. E., "A Method for Determining Mechanical Resonance Frequencies and for Calculating Elastic Moduli from these Frequencies," *Proceedings*, ASTM, 1961, pp. 1221-1238.

[6] Pickett, G., "Equations for Computing Elastic Constants from Flexural and Torsional Resonant Frequencies of Vibration of Prisims and Cylinders," *Proceedings*, ASTM, Vol 45, 1945, pp. 846-865.

[7] Shear Modulus Correction taken from Spinner, S., and Valore, R. C., "Comparisons Between the Shear Modulus and Torsional Resonance Frequencies for Bars and Rectangular Cross Sections," *Journal of Research*, National Bureau of Standards, JNBAA, Vol 60, 1058, RP2861, p. 459.

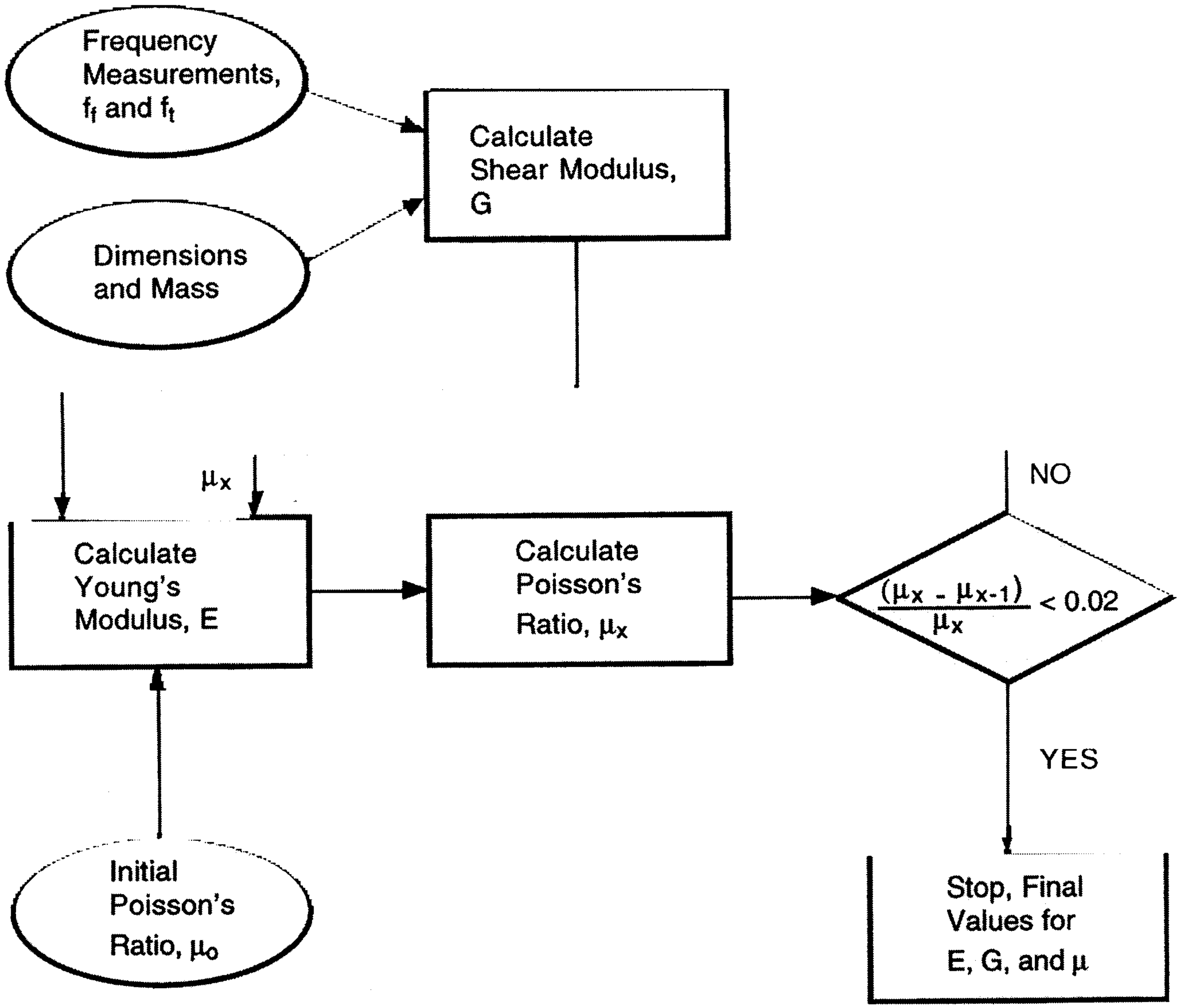

FIG. 3 Flow Chart for Iterative Determination of Poisson's Ratio

8.3 *Poisson's Ratio*:

8.3.1 From *E* and *G*:

$$\mu = \left(\frac{E}{2G}\right) - 1 \tag{3}$$

where:

μ = Poisson's ratio.

8.4 Use the following conversion factor:

$$1 \text{ Pa} = 1.450 \times 10^{-4} \text{ psi}$$

9. Report

9.1 The report shall include the following:

9.1.1 Identification of specific tests performed, a detailed description of the apparatus used, and an explanation of any deviations from the detailed practice.

9.1.2 Complete description of materials tested stating composition, number of specimens, specimen geometry and mass, specimen history, and any treatments to which the specimens have been subjected. Comments on dimensional variability, surface finish, edge conditions, observed changes after high temperature testing, etc. shall be included where pertinent.

9.1.3 Specimen temperature at measurement, number of measurements taken, numerical values obtained for measured fundamental resonant frequencies, and the calculated values for dynamic Young's modulus, dynamic shear modulus, dynamic Poisson's ratio for each specimen tested.

9.1.4 Date and name of the person performing the test.

10. Precision and Bias

10.1 *Interlaboratory Test Data*—An interlaboratory study was completed among six laboratories from 1999-2002. Six different types of refractories were tested for Young's modulus, shear modulus, and Poisson's ratio by each laboratory. The six types of refractories were a superduty fireclay, a high alumina brick (90 %), another high alumina brick (99 %), a zircon brick, an iso-pressed zircon brick, and an iso-pressed alumina brick. All samples were 3 by 4.5 by 9 in. in dimension. The same samples were then circulated among the participating laboratories, where two different operators at each laboratory performed a set of measurements.

10.2 *Precision*—Tables 1-3 contain the precision statistics for the Young's modulus, shear modulus, and Poisson's ratio results, respectively.

10.2.1 *Repeatability*—The maximum permissible difference due to test error between two test results obtained by one operator on the same material using the same test equipment is given by the repeatability interval (*r*) and the relative repeatability interval (%*r*). The 95 % repeatability intervals are given in Tables 1-3. Two test results that do not differ by more than the repeatability interval will be considered to be from the same population; conversely, two test results that do differ by more than the repeatability interval will be considered to be from different populations.

10.2.2 *Reproducibility*—The maximum permissible difference due to test error between two test results obtained by two operators in different laboratories on the same material using the same test equipment is given by the reproducibility interval (*R*) and the relative reproducibility interval (%*R*). The 95 % reproducibility intervals are given in Tables 1-3. Two test results that do not differ by more than the reproducibility interval will be considered to be from the same population; conversely, two test results that do differ by more than the reproducibility interval will be considered to be from different populations.

10.3 *Bias*—No justifiable statement can be made on the bias of the test method for measuring the elastic moduli of refractories because the values can be defined only in terms of a test method.

11. Keywords

11.1 dynamic shear modulus; dynamic Poisson's ratio; dynamic Young's modulus; elastic properties; impulse excitation; refractory shapes

TABLE 1 Precision Statistics for Young's Modulus

Material	Average Young's Modulus, psi ($\times 10^6$)	Standard Deviation Within Laboratories, *Sr* ($\times 10^5$)	Standard Deviation Betwen Laboratories, *SR* ($\times 10^5$)	Repeatability Interval, *r* ($\times 10^5$)	Reproducibility Interval, *R* ($\times 10^5$)
SuperDuty Fireclay	5.18	0.24	0.49	0.69	1.37
High Alumina (90 %)	5.49	0.60	1.61	1.68	4.51
High Alumina (99 %)	8.49	1.52	1.52	4.28	4.28
Zircon	12.01	1.15	1.15	3.23	3.23
Iso-Pressed Zircon	34.86	2.85	3.20	8.00	8.96
Iso-Pressed Alumina	52.85	7.93	7.93	22.20	22.20

Material	Coefficient of Variation Within Laboratories, *Vr*	Coefficient of Variation Between Laboratories, *VR*	Relative Repeatability, %*r*	Relative Reproducibility, %*R*
SuperDuty Fireclay	0.47	0.94	1.33	2.65
High Alumina (90 %)	1.09	2.93	3.07	8.22
High Alumina (99 %)	1.80	1.80	5.04	5.04
Zircon	0.96	0.96	2.69	2.69
Iso-Pressed Zircon	0.81	0.91	2.29	2.57
Iso-Pressed Alumina	1.50	1.50	4.20	4.20

TABLE 2 Precision Statistics for Shear Modulus

Material	Average Shear Modulus, psi (×10^6)	Standard Deviation Within Laboratories, *Sr* (×10^5)	Standard Deviation Betwen Laboratories, *SR* (×10^5)	Repeatability Interval, *r* (×10^5)	Reproducibility Interval, *R* (×10^5)
SuperDuty Fireclay	2.43	0.12	0.16	0.34	0.45
High Alumina (90 %)	2.45	0.16	0.44	0.45	1.25
High Alumina (99 %)	3.53	0.50	0.50	1.41	1,41
Zircon	4.94	0.21	0.21	0.58	0.58
Iso-Pressed Zircon	13.38	0.91	0.91	2.55	2.55
Iso-Pressed Alumina	20.97	0.76	1.16	2.13	3.27

Material	Coefficient of Variation Within Laboratories, *Vr*	Coefficient of Variation Between Laboratories, *VR*	Relative Repeatability, %*r*	Relative Reproducibility, %*R*
SuperDuty Fireclay	0.50	0.66	1.4	1.86
High Alumina (90 %)	0.65	1.83	1.83	5.12
High Alumina (99 %)	1.43	1.43	4.01	4.01
Zircon	0.42	0.42	1.19	1.19
Iso-Pressed Zircon	0.68	0.68	1.91	1.91
Iso-Pressed Alumina	0.36	0.55	1.02	1.56

TABLE 3 Precision Statistics for Poisson's Ratio

Material	Average Poisson's Ratio	Standard Deviation Within Laboratories, *Sr* (×10^5)	Standard Deviation Betwen Laboratories, *SR* (×10^5)	Repeatability Interval, *r* (×10^5)	Reproducibility Interval, *R* (×10^5)
SuperDuty Fireclay	0.07	0.00	0.01	0.01	0.02
High Alumina (90 %)	0.12	0.02	0.02	0.04	0.06
High Alumina (99 %)	0.20	0.01	0.01	0.02	0.03
Zircon	0.21	0.01	0.01	0.03	0.03
Iso-Pressed Zircon	0.30	0.01	0.01	0.02	0.03
Iso-Pressed Alumina	0.26	0.02	0.02	0.05	0.05

Material	Coefficient of Variation Within Laboratories, *Vr*	Coefficient of Variation Between Laboratories, *VR*	Relative Repeatability, %*r*	Relative Reproducibility, %*R*
SuperDuty Fireclay	0.00	14.20	14.20	28.57
High Alumina (90 %)	16.66	16.66	33.33	50.00
High Alumina (99 %)	5.00	5.00	10.00	15.00
Zircon	4.76	4.76	14.28	14.28
Iso-Pressed Zircon	3.33	3.33	6.66	10.00
Iso-Pressed Alumina	7.69	7.69	19.23	19.23