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Fine ceramics (advanced ceramics, advanced technical ceramics) — Test method for elastic moduli of monolithic ceramics at room temperature by sonic resonance

Céramiques techniques — Méthode d'essai des modules d'élasticité des céramiques monolithiques, à température ambiante, par résonance acoustique

Reference number
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Foreword

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The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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For an explanation on the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT) see the following URL: www.iso.org/iso/foreword.html.

The committee responsible for this document is ISO/TC 206, *Fine ceramics*.

This second edition cancels and replaces the first edition (ISO 17561:2002), which has been technically revised. It also incorporates the Technical Corrigendum ISO 17561:2002/Cor.1:2007.

Fine ceramics (advanced ceramics, advanced technical ceramics) — Test method for elastic moduli of monolithic ceramics at room temperature by sonic resonance

1 Scope

This International Standard describes the method of test for determining the dynamic elastic moduli of fine ceramics at room temperature by sonic resonance. This International Standard is for fine ceramics that are elastic, homogeneous and isotropic.[2]

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 3611, *Geometrical product specifications (GPS) — Dimensional measuring equipment: Micrometers for external measurements — Design and metrological characteristics*

ISO 13385 (all parts), *Geometrical product specifications (GPS) — Dimensional measuring equipment*

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

3.1
dynamic elastic moduli
adiabatic elastic moduli, which are dynamic Young's modulus, shear modulus and Poisson's ratio

Note 1 to entry: Adiabatic elastic moduli are obtained by the sonic resonance method.

3.1.1
Young's modulus
E
elastic modulus in tension or compression

$$E = \sigma / \varepsilon$$

where

E is Young's modulus in pascals;

σ is the tension or compression stress in pascals;

ε is the tension or compression strain.

3.1.2
shear modulus
G
elastic modulus in shear or torsion

$$G = \tau / \gamma$$

where

G is the shear modulus in pascals;

τ is the shear or torsional stress in pascals;

γ is the shear or torsional strain.

3.1.3
Poisson's ratio
ν
ratio of transverse strain to the corresponding axial strain resulting from uniformly distributed axial stress below the proportional limit of the material

Note 1 to entry: In isotropic materials, Young's modulus (E), shear modulus (G) and Poisson's ratio (ν) are related by the following formula:

$$\nu = E/(2G) - 1$$

3.2 vibration

3.2.1
flexural vibration
vibration apparent when the oscillation in a slender bar is in plane normal to the length dimension

Note 1 to entry: Also defined as vibration in a flexural mode.

3.2.2
torsional vibration
vibration apparent when the oscillation in each cross-section plane of a slender bar is such that the plane twists around the length dimension axis

Note 1 to entry: Also defined as vibration in a torsional mode.

3.3
resonance
state if, when a slender bar driven into one of the above modes of vibration, the imposed frequency is such that the resultant displacements for a given amount of driving force are at a maximum

Note 1 to entry: The resonant frequencies are natural vibration frequencies which are determined by the elastic modulus, mass and dimensions of the test piece.

3.4
fundamental frequency
lowest frequency of a periodic waveform

3.5
nodes
location(s) in slender rod or bar in *resonance* (3.3) having a constant zero displacement

Note 1 to entry: For the fundamental flexural resonance, the nodes are located at 0,224 L from each end, where L is the length of the rod or bar.

4 Summary of test method

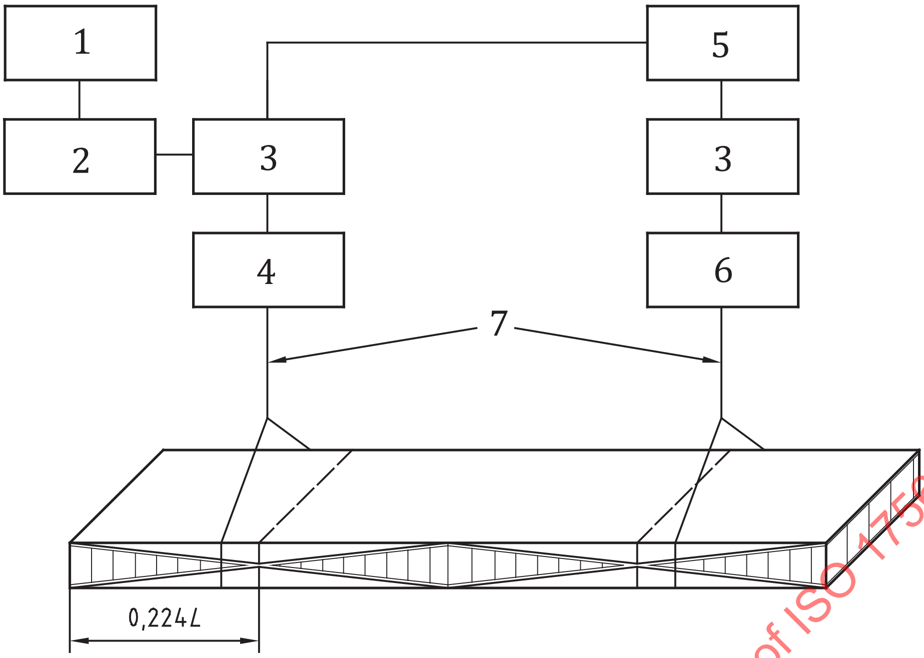
This test method measures the flexural or torsional frequencies of test specimens of rectangular prism or cylindrical geometry by exciting them at continuously variable frequencies. Mechanical excitation of the specimens is provided through the use of a transducer that transforms a cyclic electrical signal into a cyclic mechanical force on the test piece. A second transducer senses the resulting mechanical vibrations of the test piece and transforms them into an electrical signal. The amplitude and the frequency of the signal are measured by an oscilloscope or other means to detect resonance. The peak response is obtained at the resonant frequency. The fundamental resonant frequencies, dimensions and mass of the specimen are used to calculate the dynamic elastic moduli. The Young's modulus is determined from the flexural resonance frequency, and the shear modulus is determined from the torsional resonance frequency, together with the test piece dimensions and mass. Poisson's ratio is determined from the Young's modulus and the shear modulus.

5 Apparatus

5.1 General

There are various techniques that may be used to determine the resonant frequency of the test piece. The test piece may be excited by direct mechanical contact of a vibrator, or it may be suspended by a wire from a vibrator. It may be driven electromagnetically by attaching thin foils of magnetic material to one surface, or electrostatically by attaching an electrode to one surface.

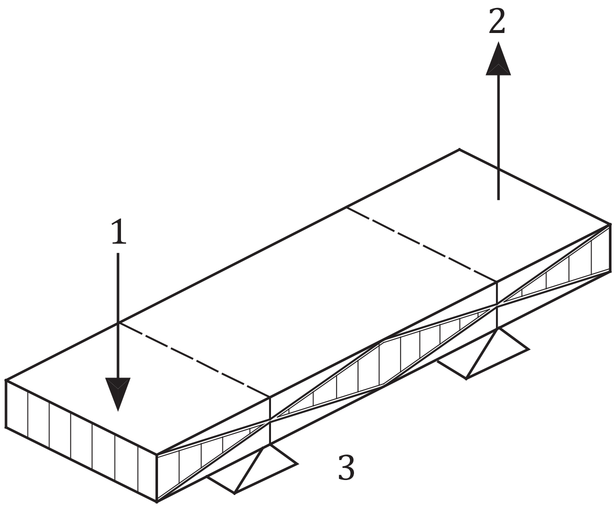
One example of the test apparatus is shown in Figure 1. The driving circuit consists of an oscillator, an amplifier, a driver and a frequency counter. The detecting circuit consists of a detector, an amplifier and an oscilloscope. Figure 1 shows the suspension style of the apparatus. The direct contact support style of the test apparatus, shown in Figure 2, is also possible. It consists of a variable-frequency audio oscillator, used to generate a sinusoidal voltage, and a power amplifier and suitable transducer to convert the electrical signal to a mechanical driving vibration. A frequency meter (preferably digital) monitors the audio oscillator output to provide accurate frequency determination. A suitable suspension coupling system supports the test piece. A transducer detector acts to detect mechanical vibration in the specimen and to convert it into an electrical signal which is passed through an amplifier and displayed on an indicating meter. The meter may be a voltmeter, a microammeter or an oscilloscope. An oscilloscope is recommended because it enables the operator to positively identify resonances, including higher order harmonics, by Lissajous figure analysis, which is a superposition of two perpendicular harmonics. If a Lissajous figure is desired, the output of the oscillator is also coupled to the horizontal plates of the oscilloscope.



Key

1 frequency counter
2 oscillator
3 amplifier
4 driver
5 oscilloscope
6 detector
7 suspending string

Figure 1 — Example of the test apparatus and the suspension for fundamental flexural resonance



Key
1 driving
2 detecting
3 flexural

Figure 2 — Example of the direct contact support of the test piece for fundamental flexural resonance

5.2 Oscillator

The oscillator shall be able to vary the frequency from 100 Hz to at least 30 kHz, with a frequency resolution of 1 Hz and a maximum frequency drift of 1 Hz/min.

5.3 Amplifier

The audio amplifier shall have a power output sufficient to ensure that the type of transducer used can excite any specimen, the mass of which falls within a specified range. A power amplifier in the detector circuit shall be impedance-matched with the type of detector transducer selected and shall serve as a prescope amplifier.

5.4 Driver

The driver shall be able to convert electrical vibration to mechanical vibration. The frequency response of the driver transducer across the frequency range of interest shall have at least a 6,5 kHz bandwidth before −3 dB power loss occurs.

NOTE For flexibility in testing, the bandwidth can, with advantage, be at least as large as the frequency range given in Table 1.

5.5 Detector

The detector shall generate a voltage proportional to the amplitude, velocity or acceleration of the mechanical vibration of the specimen. The frequency response of the detector across the frequency range of interest shall have at least a 6,5 kHz bandwidth before a −3 dB power loss occurs.

NOTE For flexibility in testing, the bandwidth can, with advantage, be at least as large as the frequency range given in Table 1.

Table 1 — Examples of the test piece size and the calculated resonant frequencies

Where the density = 3 g/cm³			
$L \times b\ (d) \times t$	E = 200 GPa ν = 0,25	E = 300 GPa ν = 0,25	E = 400 GPa ν = 0,25
75 × 15 × 3	f_f = 4 453 Hz f_t = 12 706 Hz	5 453 Hz 15 561 Hz	6 297 Hz 17 969 Hz
100 × 20 × 2	f_f = 1 676 Hz f_t = 5 016 Hz	2 053 Hz 6 143 Hz	2 371 Hz 7 094 Hz
75 × 20 × 2	f_f = 2 977 Hz f_t = 6 688 Hz	3 646 Hz 8 191 Hz	4 210 Hz 9 458 Hz
Where the density = 6 g/cm³			
$L \times b\ (d) \times t$	E = 200 GPa ν = 0,25	E = 300 GPa ν = 0,25	E = 400 GPa ν = 0,25
75 × 15 × 3	f_f = 3 148 Hz f_t = 8 984 Hz	3 856 Hz 11 004 Hz	4 453 Hz 12 706 Hz
100 × 20 × 2	f_f = 1 185 Hz f_t = 3 547 Hz	1 452 Hz 4 344 Hz	1 676 Hz 5 016 Hz
75 × 20 × 2	f_f = 2 105 Hz f_t = 4 729 Hz	2 578 Hz 5 792 Hz	2 977 Hz 6 688 Hz

where

L is the length in millimetres;

b is the width in millimetres;

d is the diameter in millimetres;

t is the thickness in millimetres;

f_f is the fundamental flexural resonant frequency in Hertz;

f_t is the fundamental torsional resonant frequency in Hertz.

5.6 Frequency counter

The frequency counter, preferably digital, shall be able to measure frequencies to within ±1 Hz.

5.7 Specimen suspension means

Any method of specimen support that permits the free vibration of the test piece with no significant effect on the vibration frequencies shall be used. Test pieces are commonly supported either by suspension from threads or wires or on direct contact supports. If the test piece is to be supported from beneath, the support shall be made of rubber, cork or similar material, and shall have a minimum contact area with the test piece. If the test piece is suspended from the driving and detecting transducers, fine thread or metal wires shall be used. The vibrating mass of the suspension system shall be less than 0,1 % of the mass of the test piece. For the electromagnetic or electrostatic method, the mass of any magnetic foil or electrode attached to the test piece shall be negligible compared with the mass of the test piece.

5.8 Micrometer

A micrometer with a resolution of 0,002 mm or 0,1 % of the specimen, in accordance with ISO 3611, shall be used to measure the thickness, width and diameter of the test piece. Alternative dimension measuring instruments that have a resolution of 0,002 mm or finer may be used.

5.9 Vernier calliper

A vernier calliper with a resolution of 0,05 mm or 0,1 % of the specimen, in accordance with ISO 13385 (all parts), shall be used to measure the length of the test piece. Alternative dimension measuring instruments that have a resolution of 0,05 mm or finer may be used.

5.10 Balance

A balance with a resolution of 1 mg or 0,1 % of the specimen or finer, shall be used to measure the weight of the test piece.

6 Test pieces

The test piece shall be a rectangular prism or a bar with a circular cross section. This test method is not satisfactory for test pieces that have major discontinuities, such as large cracks (surface or internal) or internal voids.

a) For flexural resonance, the length of the specimen (L) shall be greater than 40 mm, and the ratio of length to thickness (t), L/t, or length to diameter (d), L/d, shall be greater than 20.

b) For torsional resonance, L shall be greater than 40 mm, L/t shall be greater than 20, and the ratio of the width (b) to the thickness, b/t, shall be in the range of 3 to 10 (5 is recommended).

c) The parallelism of the specimen shall be within 0,1 % across the length and the width, and shall be within 0,5 % across the thickness.

d) The surface of the test piece shall be smooth and flat. The surface shall be finished using a fine grind (400 grit or finer). The machining procedure shall not affect the test results.

e) The edges of the specimen shall not be chamfered. However, if the chipping of the specimen from the edges affects the results, the edges may be chamfered, but the amount of the chamfering shall be as small as possible.

   NOTE ASTM C1259, Annex A2 describes correction for edge chamfers or radii in rectangular beams in the calculation of Young's Modulus.

f) For the suspension method, the mass of the specimen is, with advantage, at least 5 g in order to assist keeping a suspension system straight.

7 Test procedure

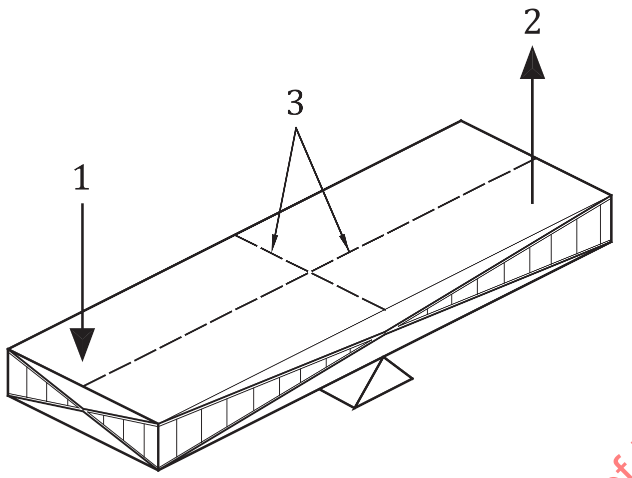
7.1 Measurement of the size and the mass

Dry the specimen until the mass is constant. Weigh the specimen to the nearest 1 mg or 0,1 % (whichever is greater). Measure the thickness and the width, or the diameter, to the nearest 0,002 mm or 0,1 % (whichever is greater) at three points on the test piece, and determine the average of the three measurements. Measure the length to the nearest 0,05 mm or 0,1 % (whichever is greater).

7.2 Positioning of the specimen

Position the specimen properly. If the fundamental mode of the flexural resonance is measured, the vibration nodes appear at a distance of 0,224 of the total length from each end. If the specimen is suspended by threads or wires that will be used to drive and detect vibration, position the threads outside the nodal points, as shown in Figure 1 (distance of 0,2 of the length from each end is recommended). If the specimen is to be supported from below, position two supports at the nodal points, as shown in Figure 2. If the fundamental mode of the torsional resonance is measured, the vibration nodes appear as shown in Figure 3. If the specimen is supported from below, position one support at the centre of the specimen, as shown in Figure 3. If the specimen is suspended by threads, position the supports at diagonally opposed corners of the specimen, as shown in Figure 4. The suspending devices shall permit the free vibration of the test piece.

Cotton thread is recommended for suspending the specimen. Cork or rubber is recommended for supporting the specimen.



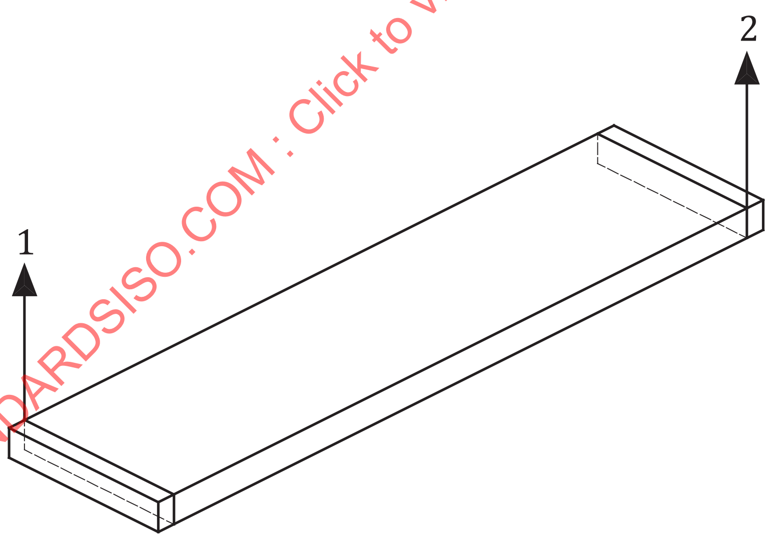
Key

1 driving

2 detecting

3 nodal

Figure 3 — Nodes and direct contact support on the fundamental torsional resonance



Key

1 driving

2 detecting

Figure 4 — Suspension for the fundamental torsional resonance

7.3 Measurement of resonant frequency

Activate the equipment so that the power adequate to excite the specimen is delivered to the driving transducer. Set the gain of the detector circuit high enough to detect vibration in the specimen and to display it on the oscilloscope screen with sufficient amplitude to accurately measure the frequency at which the signal amplitude is maximized. Adjust the oscilloscope so that a sharply defined horizontal baseline exists when the test piece is not excited. Scan frequencies with the audio oscillator until test piece resonance (flexural or torsional) is indicated by a sinusoidal pattern of maximum amplitude on the oscilloscope or by a single closed loop Lissajous pattern. It is recommended that the frequency scan start at a low frequency and then increase. To verify that the frequency is fundamental and not an overtone, either the node/antinode locations or one or more overtones should be identified. If a determination of the shear modulus is made, offset the coupling to the transducers so that the torsional mode of vibration may be induced and detected (see Figure 3 and Figure 4).

Currently, no certified reference artefacts for checking this measurement are available. Measurement system calibration for frequency should be undertaken using a suitable traceably certified frequency source. Laboratories should retain an identified test-piece for intermittent system repeatability checks, and for staff training.

NOTE The proper identification of the fundamental flexural mode is important as spurious frequencies inherent in the system may interfere, especially when greater excitation power and detection sensitivity are required for work with a specimen that has a poor response. One method of locating the nodes on the test piece is to move the detector along the length of the test piece; a node is indicated when the output amplitude goes to zero. An anti-node is indicated when the output amplitude reaches a local maximum. Another node location method (used often with string suspensions) is to lay a thin rod across the test piece at a presumed node or anti-node location. If the output amplitude is not affected, then the rod is on a node; if the output amplitude goes to zero, then the location is an anti-node. It is also possible to locate the nodes by spreading fine, free-flowing powder on the top surface of the test piece and observe its alignment at the nodal points. When several resonant flexural frequencies have been identified, the lowest frequency can be verified as the fundamental if the numerical ratios of the first three overtone frequencies to the lowest frequency are: 2,7, 5,4 and 8,9. Note that these ratios are for a Bernoulli-Euler (simple) beam under ideal conditions. Typically, the ratios will be slightly lower. A further possibility is localizing the nodes on the test piece by means of laser vibrometer.

8 Calculations

8.1 Calculate the dynamic Young's modulus for a rectangular prism from Formula (1):[3]

$$E = 0{,}946\,5\,\frac{m f_\mathrm{f}^2}{b}\left(\frac{L}{t}\right)^3\left[1 + 6{,}585\left(\frac{t}{L}\right)^2\right] \quad (1)$$

where

- E is the dynamic Young's modulus in pascals;
- m is the mass of the specimen in kilograms;
- f_f is the fundamental flexural resonant frequency in hertz;
- b is the width of the specimen in metres;
- L is the length of the specimen in metres;
- t is the thickness of the specimen in metres.

8.2 Calculate the dynamic Young's modulus for a bar with a circular cross section from Formula (2):[3]

$$E = 1{,}606\,7\left(m f_\mathrm{f}^2\right)\left(\frac{L^3}{d^4}\right)\left[1 + 4{,}939\left(\frac{d}{L}\right)^2\right] \quad (2)$$

where d is the diameter of the specimen in metres.

8.3 Calculate the dynamic shear modulus for a rectangular prism from Formula (3):[4]

$$G = \frac{4Lmf_{t}^{2}}{bt}\left(\frac{B}{1+A}\right) \tag{3}$$

where

G is the dynamic shear modulus in pascals;

f_t is the fundamental torsional resonant frequency in hertz;

B is a shape factor given by

$$B = \left[(b/t) + (t/b)\right] \Big/ \left[4(t/b) - 2{,}52(t/b)^2 + 0{,}21(t/b)^6\right];$$

A is an empirical correction factor, given by Figure 5 or by

$$A = \left[0{,}506\,2 - 0{,}877\,6(b/t) + 0{,}350\,4(b/t)^2 - 0{,}007\,8(b/t)^3\right] \Big/ \left[12{,}03(b/t) + 9{,}892(b/t)^2\right].$$

8.4 Calculate the dynamic shear modulus for a bar with a circular cross section from Formula (4):[4]

$$G = 16mf_{t}^{2}\left(\frac{L}{\pi d^{2}}\right) \tag{4}$$

8.5 Calculate the dynamic Poisson's ratio from Formula (5):

$$\nu = \frac{E}{2G} - 1 \tag{5}$$

where ν is the dynamic Poisson's ratio.