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Fine ceramics (advanced ceramics, advanced technical ceramics) — Mechanical properties of ceramic composites at ambient temperature in air atmospheric pressure — Determination of elastic properties by ultrasonic technique

Céramiques techniques (céramiques avancées, céramiques techniques avancées) — Propriétés mécaniques des céramiques composites à température ambiante sous air à pression atmosphérique — Détermination des propriétés élastiques par méthode ultrasonore

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

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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*.

Fine ceramics (advanced ceramics, advanced technical ceramics) — Mechanical properties of ceramic composites at ambient temperature in air atmospheric pressure — Determination of elastic properties by ultrasonic technique

1 Scope

This document specifies an ultrasonic method to determine the components of the elasticity tensor of ceramic matrix composite materials at room temperature. Young's moduli shear moduli and Poisson coefficients, can be determined from the components of the elasticity tensor.

This document applies to ceramic matrix composites with a continuous fibre reinforcement: unidirectional (1D), bidirectional (2D), and tridirectional (×D, with 2 < × ≤ 3) which have at least orthotropic symmetry, and whose material symmetry axes are known.

This method is applicable only when the ultrasonic wavelength used is larger than the thickness of the representative elementary volume, thus imposing an upper limit to the frequency range of the transducers used.

NOTE Properties obtained by this method might not be comparable with moduli obtained by ISO 15733, ISO 20504 and EN 12289.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. 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/IEC 17025, *General requirements for the competence of testing and calibration laboratories*

EN 1389, *Advanced technical ceramics — Ceramic composites — Physical properties — Determination of density and apparent porosity*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in CEN/TR 13233 and the following apply.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— IEC Electropedia: available at http://www.electropedia.org/

— ISO Online browsing platform: available at http://www.iso.org/obp

3.1

stress-strain relations for orthotropic material

elastic anisotropic behaviour of a solid homogeneous body described by the elasticity tensor of fourth order C_{ijkl}, represented in the contracted notation by a symmetrical square matrix (6 × 6)

Note 1 to entry: If the material has at least orthotropic symmetry, its elastic behaviour is fully characterized by nine independent stiffness components C_{ij}, of the stiffness matrix (C_{ij}), which relates stresses to strains, or equivalently by nine independent compliance components S_{ij} of the compliance matrix (S_{ij}), which relates strains to stresses. The stiffness and compliance matrices are the inverse of each other.

If the reference coordinate system is chosen along the axes of symmetry, the stiffness matrix C_{ij} and the compliance matrix S_{ij} can be written as follows:

$$\begin{bmatrix}\sigma_1\\ \sigma_2\\ \sigma_3\\ \sigma_4\\ \sigma_5\\ \sigma_6\end{bmatrix}=\begin{bmatrix}C_{11} & C_{12} & C_{13} & 0 & 0 & 0\\ C_{12} & C_{22} & C_{23} & 0 & 0 & 0\\ C_{13} & C_{23} & C_{33} & 0 & 0 & 0\\ 0 & 0 & 0 & C_{44} & 0 & 0\\ 0 & 0 & 0 & 0 & C_{55} & 0\\ 0 & 0 & 0 & 0 & 0 & C_{66}\end{bmatrix}\begin{bmatrix}\varepsilon_1\\ \varepsilon_2\\ \varepsilon_3\\ \varepsilon_4\\ \varepsilon_5\\ \varepsilon_6\end{bmatrix}$$

$$\begin{bmatrix}\varepsilon_1\\ \varepsilon_2\\ \varepsilon_3\\ \varepsilon_4\\ \varepsilon_5\\ \varepsilon_6\end{bmatrix}=\begin{bmatrix}S_{11} & S_{12} & S_{13} & 0 & 0 & 0\\ S_{12} & S_{22} & S_{23} & 0 & 0 & 0\\ S_{13} & S_{23} & S_{33} & 0 & 0 & 0\\ 0 & 0 & 0 & S_{44} & 0 & 0\\ 0 & 0 & 0 & 0 & S_{55} & 0\\ 0 & 0 & 0 & 0 & 0 & S_{66}\end{bmatrix}\begin{bmatrix}\sigma_1\\ \sigma_2\\ \sigma_3\\ \sigma_4\\ \sigma_5\\ \sigma_6\end{bmatrix}$$

Note 2 to entry: For symmetries of higher level than the orthotropic symmetry, the C_{ij} and S_{ij} matrices have the same form as here above. Only the number of independent components reduces.

3.2

engineering constants

compliance matrix components of an orthotropic material which are in terms of engineering constants:

$$\left[S_{ij}\right]=\begin{bmatrix}1/E_{11} & -\nu_{21}/E_{22} & -\nu_{31}/E_{33} & 0 & 0 & 0\\ -\nu_{12}/E_{11} & 1/E_{22} & -\nu_{32}/E_{33} & 0 & 0 & 0\\ -\nu_{13}/E_{11} & -\nu_{23}/E_{22} & 1/E_{33} & 0 & 0 & 0\\ 0 & 0 & 0 & 1/G_{23} & 0 & 0\\ 0 & 0 & 0 & 0 & 1/G_{13} & 0\\ 0 & 0 & 0 & 0 & 0 & 1/G_{12}\end{bmatrix}$$

where

E_{11}, E_{22} and E_{33}	are the elastic moduli in directions 1, 2 and 3, respectively;
G_{12}, G_{13} and G_{23}	are the shear moduli in the corresponding planes;
ν_{12}, ν_{13}, ν_{23}	are the respective Poisson coefficients.

3.3
angle of incidence
θ_i
angle between the direction 3 normal to the test specimen front face and the direction n_i of the incident wave

Note 1 to entry: See Figures 1 and 2.

3.4
refracted angle
θ_r
angle between the direction 3 normal to the test specimen front face and the direction n of propagation of the wave inside the test specimen

Note 1 to entry: See Figures 1 and 2.

3.5
azimuthal angle
ψ
angle between the plane of incidence (3, n_i) and plane (2, 3) where n_i corresponds to the vector oriented along the incident plane wave and direction 2 corresponds to one of the axes of symmetry of the material

Note 1 to entry: See Figure 1.

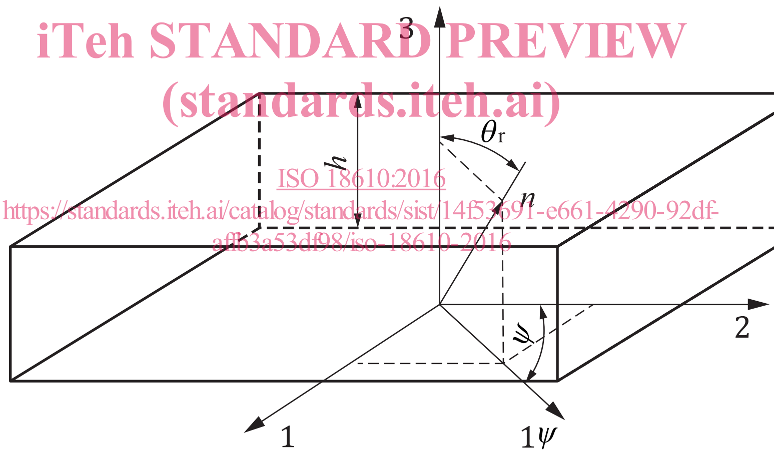


Figure 1 — Definition of angles

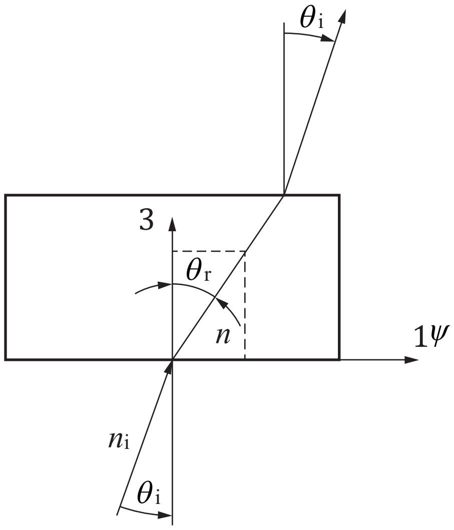


Figure 2 — Propagation in the plane of incidence

3.6
first critical angle
θ_c
angle of incidence θ_i that provides an angle of refraction of 90 degrees of the quasi longitudinal wave angle

3.7
unit vector
n
vector of length 1 oriented along the propagation direction of the incident plane wave inside the specimen, with its components n_k (k = 1, 2, 3):

$$n_1 = \sin\theta_r \sin\psi$$

$$n_2 = \sin\theta_r \cos\psi$$

$$n_3 = \cos\theta_r$$

Note 1 to entry: See Figures 1 and 2.

3.8
propagation velocity
$V(n)$
phase velocity of a plane wave inside the specimen in dependence on unit vector *n* (i.e. in dependence on ψ and θ_r)

Note 1 to entry: V_0 is the propagation velocity in the coupling fluid.

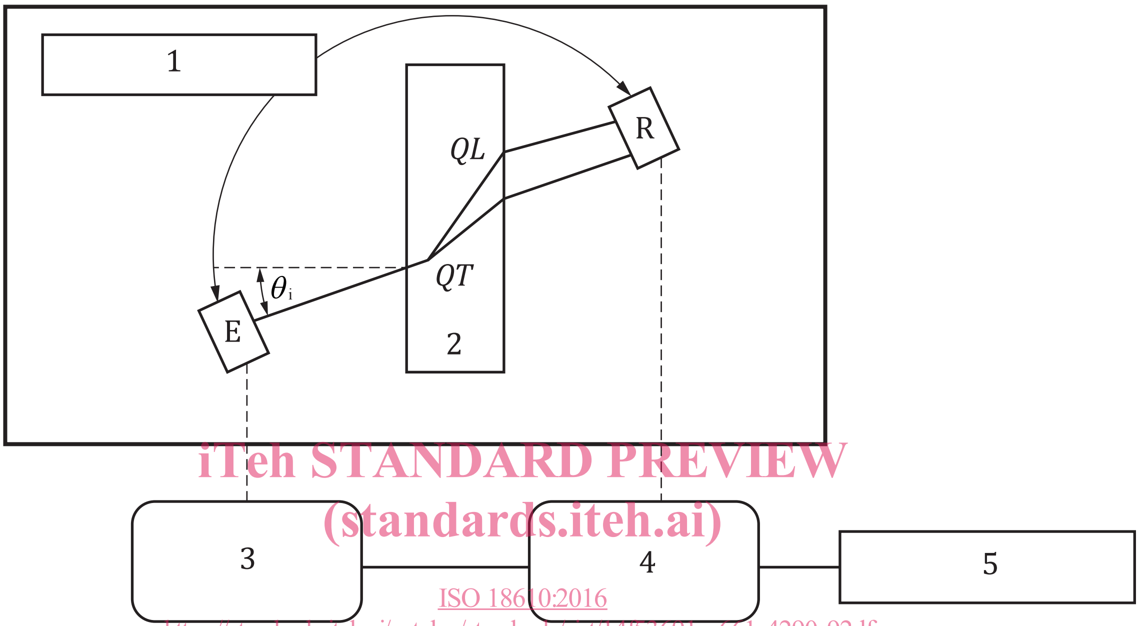
3.9
delay
$\delta t(n)$
difference between the time-of-flight of the wave when the test specimen is in place and the time-of-flight of the wave in the coupling fluid with the test specimen removed under the same configuration of the probes in dependence on unit vector *n*

3.10
bulk density
ρ
ratio of the mass of the material without porosity to its total volume including porosity

4 Principle

The determination of the elastic properties consists of calculating the coefficients of the propagation equation of an elastic plane wave, from a set of properly chosen velocity measurements along known directions.

A thin specimen with plane parallel faces is immersed in an acoustically coupling fluid (e.g. water), see Figure 3. The specimen is placed between a transmitter (T) and a receiver (R), which are rigidly connected to each other and have two rotational degrees of freedom. Using appropriate signal processing, the propagation velocities of each wave in the specimen are calculated.



Key

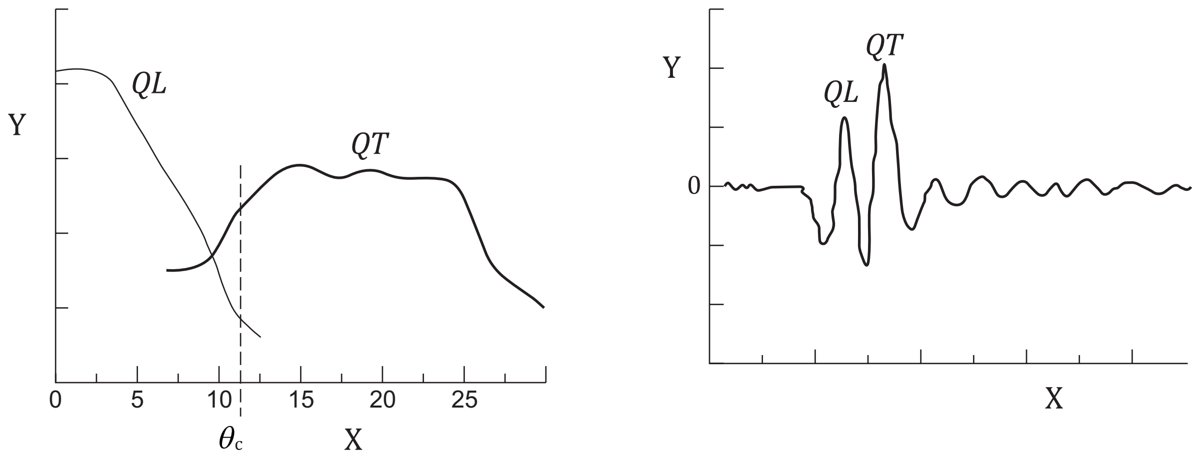
1 rotation drive
2 test object
3 pulse generator
4 digital oscilloscope
5 micro-computer

Figure 3 — Ultrasonic test assembly

Depending on the angle of incidence, the wave created by the pulse sent by the transmitter T is refracted within the material in one (a quasi longitudinal wave *QL*, or a quasi transverse wave *QT)*, two (*QL*+ *QT or* two quasi transverse waves QT_1, QT_2) or three bulk waves (QL+ QT_1+QT_2) that propagate in the solid at different velocities and in different directions.

The receiver R collects one, two or three pulses, corresponding to each of these waves.

The difference between the time-of-flight of each of the waves and the time-of-flight of the transmitted pulse in the coupling fluid without the test object is measured. The evaluation procedure is based on the measurement of the time-of-flight of the quasi-longitudinal and one or both quasi-transverse waves, and is only valid when the *QL* and the *QT* waves are appropriately separated (see Figure 4).



Key

Y amplitude

X angle of incidence

a) Amplitude of the *QL* and *QT* waves as a function of the angle of incidence with overlapping in the region of θ_c

Key

Y amplitude

X time

NOTE Both *QL* and *QT* waves are present and can be distinguished in the positive domain but are slightly overlapping in the negative domain.

b) Temporal waveform of the *QL* and *QT* waves at an angle of incidence, θ_i, close to the critical angle, θ_c

Figure 4 — Example of partial overlapping of *QL* and *QT* waves at an angle of incidence θ_i

From the propagation velocities, the components of the elasticity tensor are obtained through a least square regression analysis which minimizes the residuals of the wave propagation equations.

Young's moduli, shear moduli and Poisson coefficients are determined from these components.

5 Significance and use

Only two constants (Lamés coefficients, Young's modulus and Poisson coefficient, Young's and shear moduli, longitudinal and transverse wave velocities) are sufficient in order to fully describe the elastic behaviour of an isotropic solid body. When anisotropy, which is a specific feature of composite materials, shall be taken into account, the use of an elasticity tensor with a larger number of independent coefficients is needed. While conventional mechanical methods allow only a partial identification of the elasticity of anisotropic bodies, ultrasonic techniques allow a more exhaustive evaluation of the elastic properties of these materials, particularly transverse elastic moduli and shear moduli for thin specimens.

Successful application of the method depends critically on an appropriate selection of the central frequency of the transducers. Frequency shall be sufficiently low for the measurement to be representative of the elementary volume response, but at the same time high enough to achieve a separation between the *QL* and the *QT* waves.

The determination of elastic properties by the ultrasonic technique described here is based on a non-destructive dynamic measurement of wave propagation velocities. The determination of the values of Young's moduli, shear moduli and Poisson ratios need a single specimen.

6 Test equipment

6.1 Immersion tank with temperature measurement device

The temperature of the coupling fluid in the immersion tank should stay constant within ±0,5 °C for the full duration of the test.

The temperature measurement device shall be capable of measuring the temperature to within 0,5 °C.

This requirement is imposed because the wave propagation velocity in the coupling fluid is temperature sensitive.

6.2 Holder of the probes and test object

The holder of the ultrasonic probes or the holder of the test object shall allow a rotation to cover the range of angles of incidence θ_i between 0° and 90°. Additionally, it shall allow for discrete settings of the azimuthal angle ± of 0°, 45° and 90°. The accuracy in the measurement of the angles θ_i and ψ shall be better than 0,1° and 1°, respectively.

The probes shall be mounted in such a way that their relative position remains fixed during the test.

6.3 Probes

Piezoelectric broad-band probes adapted to the coupling fluid and able to generate longitudinal ultrasonic waves shall be used. Two probes with similar specifications (e.g. central frequency, bandwidth) shall be used as transmitter and receiver.

6.4 Pulse generator

The pulse generator shall be selected in accordance with the characteristics of the probes.

It shall be able to generate short-duration (<1 µs) sinusoidal pulses of voltage sufficient to provide a mechanical pulse by the transducer. The frequency of the exciting pulse shall be chosen, such as described in 9.1.

The interval between consecutive pulses shall be long compared with the travel time being recorded, typically greater than 1 ms, so that all signals from the preceding pulse have dissipated before initiating the next.

6.5 Signal display and recording system

Use any system, for instance, e.g. digital oscilloscope, with a minimum sampling frequency of 100 MHz that allows the recording of transmitted and received signals. The signal recording system is designed in order to allow one to see on the display the generated and the detected pulses on the same time-base and to determine the time-gap separating these two events.

7 Test object

The choice of the geometry of the test object depends on the nature of the material and the reinforcement structure. The thickness shall be large enough to allow separation of the echoes of the quasi longitudinal *QL* and quasi transverse *QT* waves and shall be representative of the material. The largest possible thickness is recommended, at least five times the size of the representative volume element (RVE) in the direction of propagation of the wave. The other dimensions of the test object shall be at least twice the diameter of the transducer. A test object with parallel faces is mandatory. The two faces shall be parallel of better than 0,1 mm.