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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

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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 $2 < \times \leq 3$) 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.

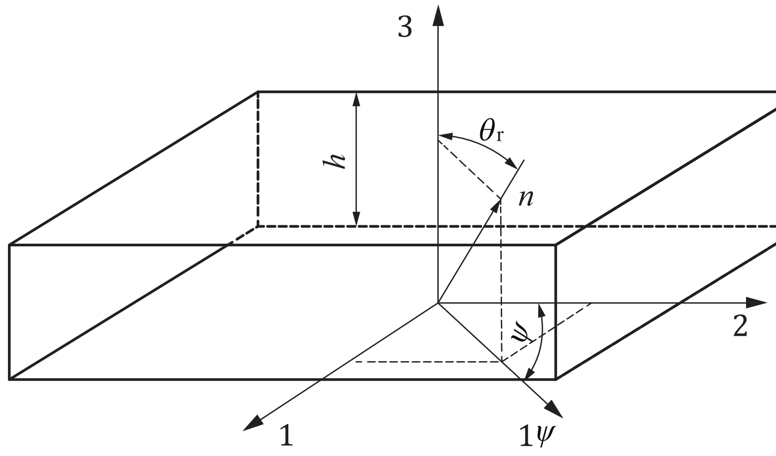


Figure 1 — Definition of angles

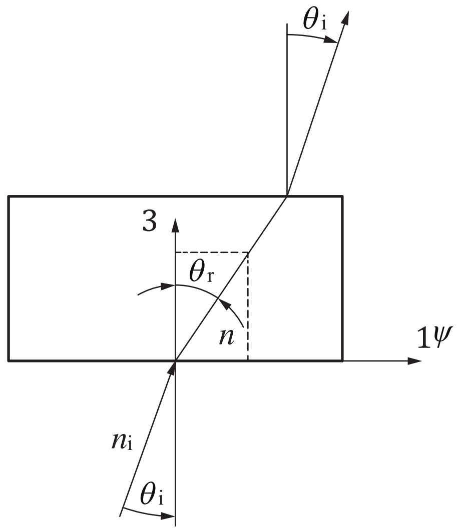


Figure 2 — Propagation in the plane of incidence

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_o is the propagation velocity in the coupling fluid.

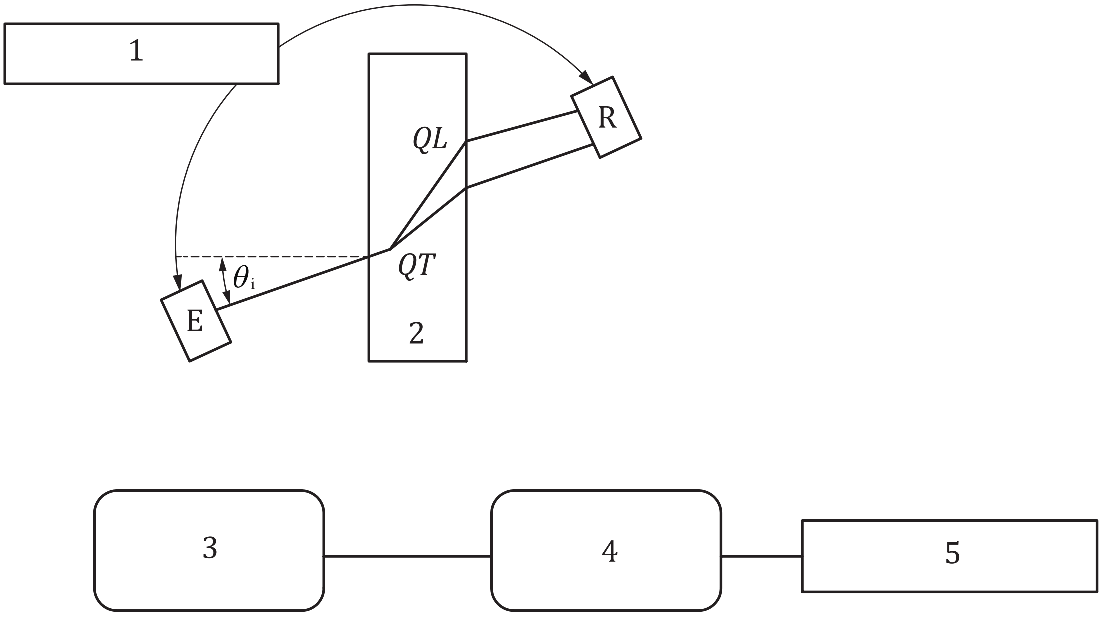
3.9
delay
$\delta t(n)$
difference between the time-of-flight of the wave when the test specimen is in place and the time-of-flight of the wave in the coupling fluid with the test specimen removed under the same configuration of the probes in dependence on unit vector *n*

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

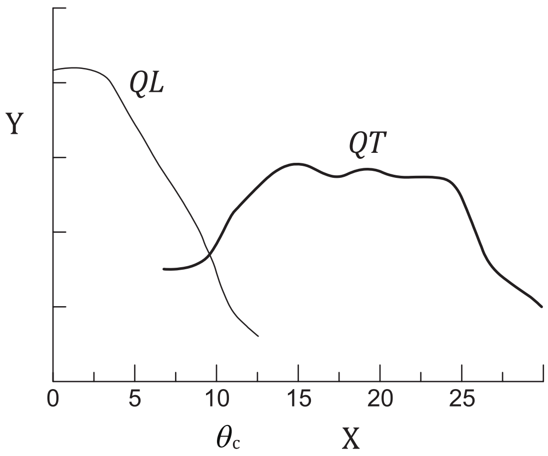
5 micro-computer

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).

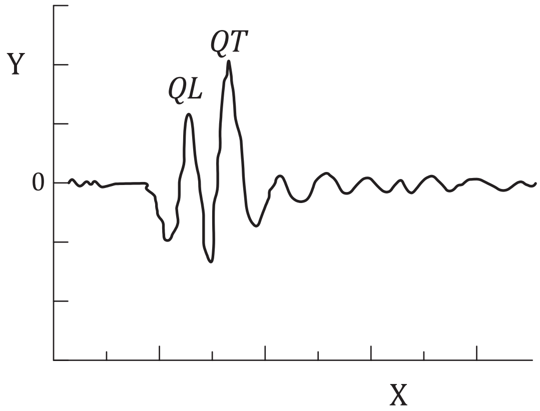


Key

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.

8 Test object preparation

The material symmetry axes shall be identified. If machining is required, it shall be performed in such a way that the material symmetry axes remain known at all times.

Machining procedures that do not cause damage to the test objects shall be clearly defined and recorded. These procedures shall be followed during machining of the test objects.

NOTE Usually, test objects from plates are cut with their longitudinal axis coinciding with one of the principal directions of the reinforcement.

One test object is sufficient to perform the test. Multiple measurements can be done on a single test object.

Care shall be taken to avoid chemical interaction between the coupling fluid and the test object.

9 Test procedure

9.1 Choice of frequency

The selection of the appropriate frequency is critical for the application of the method. The frequency shall be sufficiently low to ensure that the measurement is representative.

NOTE 1 An initial selection of $f < 0{,}2\,\frac{V}{d}$, where d is the characteristic length of the RVE in the direction of normal incidence, is proposed ($\theta_i = 0$).

Where d is the characteristic length of the RVE in the direction of normal incidence ($\theta_i = 0$) and V the propagation velocity inside the specimen and in that direction, is proposed.

NOTE 2 Because of the inverse relationship between wavelength λ and frequency f ($f = \frac{V}{\lambda}$), this corresponds to a wavelength λ of at least 5d.

For the selected frequency, the following additional criteria should be met:

a) measurable amplitude of the QL wave under normal incidence $\theta_i = 0$. If the amplitude is too small, the frequency shall be decreased;

b) time separation of the waves QL and QT when varying the angle of incidence θ_i [see Figure 4 b)]. This is promoted by increasing the frequency.

A minimum frequency of $\frac{3V}{2h}$ is recommended.

Because the frequency requirements for meeting the three mentioned criteria may be conflicting, there are cases where the method is not applicable. In these cases, the only remaining solution is to increase the thickness of the test object beyond the minimum thickness stipulated in Clause 7.

NOTE 3 For example, for a 2D SiC/SiC with a RVE of 0,5 mm (requiring a minimum thickness of the test object of 2,5 mm in accordance with Clause 7), the test frequency, in order for the measurement to be representative, is lower than 2,25 MHz (corresponding to wave velocities of around 5 000 m/s). On the other hand, for obtaining mode separation, the frequency is higher than $\frac{3V}{2h} = 3\text{ MHz}$. The method can therefore not be applied for the given thickness of 2,5 mm. An increase in thickness to 3,3 mm allows mode separation at a frequency of 2,25 MHz.

9.2 Establishment of the test temperature

Measure the temperature of the coupling fluid at a location between the transducers in the vicinity of the future position of the test specimen. Perform the reference measurement in accordance with 9.3. Perform the test in accordance with 9.4.

9.3 Reference test without test object

Record the signals from the transmitter and from the receiver versus time without a test object mounted.

9.4 Measurement with the test object

9.4.1 Determination of the bulk density and thickness

9.4.1.1 Determination of the bulk density

The bulk density shall be determined in accordance with EN 1389.

9.4.1.2 Measurement of the thickness

The thickness shall be measured in three positions on the test area with a calliper with an inaccuracy less than 0,02 mm in accordance with ISO 3611.

9.4.2 Mounting of the test object

Mount the test object in the holder.

Measure the temperature in the vicinity of the test object.

Make sure that the measured temperature falls within ±0,5 °C from that of the reference measurement.

The test object shall be oriented perpendicularly to the incoming beam. The inaccuracy of the perpendicularity between the beam and the specimen shall be below 0,1 mm.

The test specimen shall be mounted in such a way that one of the symmetry axes coincides with $\psi = 0°$ to within 1°.

9.4.3 Acquisition of different angles of incidence

Set acquisition plane by selecting azimuthal angle $\psi = 0°$, 45° and 90°. For each acquisition, plane measurements shall be made of the *QL* and *QT* signals at given values of the angle of incidence θ_i. The angle of incidence θ_i varies from 0° up to a maximum defined by a decrease of the amplitude of the *QT* wave to approximately one third of its maximum. The number of incidence angles shall be selected to optimise coverage over the range in which both the *QL* and *QT* waves appear.

Over the total range of θ_i usually a minimum of 20 measurements shall be performed.

The maximum angle θ_i is also configuration limited.

Only signals recorded at values of θ_i and ψ meeting the following conditions can be used for subsequent calculation and evaluation of C_{ij}:

a) the bulk waves are clearly identified (i.e. they can unambiguously be separated from other propagating waves);

b) the longitudinal *QL* and the transverse *QT* waves are clearly separated in time, making it possible to clearly separate *QL* from *QT*.

NOTE This is usually verified by representing the experimental results by velocity curves as shown in Annex A.

10 Calculation

10.1 Delay

For each value of ψ and θ_i, the delay $\delta t(n)$ on the *QL* and the *QT* waves is determined by comparing the signal received in the coupling fluid alone (reference signal), and the signal received when the test object is in the coupling fluid between transmitter and receiver.

NOTE The delay $\delta t(n)$ is usually obtained by computer assisted signal processing techniques.

10.2 Calculation of the propagation velocities

For each measurement of $\delta t(n)$, the associated propagation velocity $V(n)$ is determined by Formula (1):

$$V(n) = \frac{V_o}{\sqrt{1 + \frac{V_o \delta t(n)}{h}\left(\frac{V_o \delta t(n)}{h} - 2\cos\theta_i\right)}} \qquad (1)$$

where

$V(n)$ is the propagation velocity in the material, in metres per second ($m \cdot s^{-1}$);

V_o is the propagation velocity in the coupling fluid, in metres per second ($m \cdot s^{-1}$);

h is the thickness defined as the mean value of three measurements in the test area, in metres (m) (see 9.4.1.2);

$\delta t(n)$ is the delay, in seconds (s);

θ_i is the angle of incidence, in degrees (°).

10.3 Calculation of the refracted angle, θ_r

$$\theta_r = \arcsin\left[\frac{V(n) \times \sin\theta_i}{V_o}\right] \qquad (2)$$

where

θ_r is the refracted angle, in degrees (°).

10.4 Identification of the elastic constants, C_{ij}

10.4.1 Basic considerations

The phase velocities of the three propagating waves *QL*, QT_1, QT_2 are given by the eigenvalues of the propagation tensor Γ_{ij} according to Formula (3):

$$\mathrm{Det}\left(\Gamma_{ij} - \rho V^2(n)\delta_{ij}\right) = 0 \qquad (3)$$

and the polarization directions are the corresponding eigenvectors, with δ_{ij} Kronecker's symbol.

The wave propagation tensor in the case of an anisotropic material has the following general form:

$$\Gamma_{ij} = C_{ijkl} n_k n_l \tag{4}$$

where

C_{ijkl}	are the components of the stiffness tensor (in the contracted notation: C_{ij});
n_k and n_l (k, l = 1, 2, 3)	are the components of the propagation direction vector $n = (n_1, n_2, n_3)$;
n_1, n_2, n_3	are the direction cosines ($n_1 = \sin\theta_r \sin\psi$, $n_2 = \sin\theta_r \cos\psi$, $n_3 = \cos\theta_r$).

In the case of an orthotropic material, the components of the propagation tensor Γ_{ij} have the following form:

$$\Gamma_{11} = C_{11} n_1^2 + C_{66} n_2^2 + C_{55} n_3^2$$

$$\Gamma_{22} = C_{66} n_1^2 + C_{22} n_2^2 + C_{44} n_3^2$$

$$\Gamma_{33} = C_{55} n_1^2 + C_{44} n_2^2 + C_{33} n_3^2$$

$$\Gamma_{12} = \left(C_{12} + C_{66}\right) n_1 n_2$$

$$\Gamma_{13} = \left(C_{13} + C_{55}\right) n_1 n_3$$

$$\Gamma_{23} = \left(C_{23} + C_{44}\right) n_2 n_3$$

and inserting the eigenvalues: $\lambda(n) = \rho V^2(n)$, the following formula results:

$$f\left[C_{ij}, n, \lambda(n)\right] = (\Gamma_{11} - \lambda)(\Gamma_{22} - \lambda)(\Gamma_{33} - \lambda) + 2\Gamma_{12}\Gamma_{13}\Gamma_{23} - (\Gamma_{22} - \lambda)\Gamma_{13}{}^2 - (\Gamma_{11} - \lambda)\Gamma_{23}{}^2 - (\Gamma_{33} - \lambda)\Gamma_{12}{}^2 =$$
$$-\lambda^3 + \lambda^2(\Gamma_{11} + \Gamma_{22} + \Gamma_{33}) + \lambda\left(\Gamma_{12}{}^2 + \Gamma_{13}{}^2\Gamma_{23}{}^2 - \Gamma_{11}\Gamma_{22} - \Gamma_{11}\Gamma_{33} - \Gamma_{22}\Gamma_{33}\right) + \Gamma_{11}\Gamma_{22}\Gamma_{33} + 2\Gamma_{12}\Gamma_{13}\Gamma_{23} -$$
$$\Gamma_{11}\Gamma_{23}{}^2 - \Gamma_{22}\Gamma_{13}{}^2 - \Gamma_{33}\Gamma_{12}{}^2 = 0$$

In general, all the stiffness components, C_{ij}, can be evaluated by solving this formula inserting the velocity measurements recorded for different angles of incidence, which correspond to different combinations of ψ and θ_i angles.

At each value of ψ, a set of N measurements, for different angles of incidence θ_i corresponding to different propagation directions n_p in the test specimen, gives a set of M velocities $V(n_p) = V_p$ and thus M values of $\lambda(n_p) = \lambda_p$ because of $\lambda(n_p) = \rho V^2(n_p)$.

Then the minimization of the following expression is necessary:

$$F\left[C_{ij}\right] = \sum_{p=1}^{M}\left[\left(f\left[C_{ij}, n_p, \lambda\left(n_p\right)\right]\right)^2\right] \tag{5}$$

NOTE 1 Because of the possible occurrence of *QL*, *QT1*, *QT2*, the number of measured velocities *M* is larger than *N*.

NOTE 2 To minimize this expression, the use of a computer assisted methodology is necessary. Different algorithms are available for this purpose (such as Newton-Raphson, Simplex, conjugate gradient method, etc.).

However, this approach implies a lot of difficulties and accuracy problems. To simplify the calculations the following methodology described in 10.4.2 to 10.7 is proposed.

A confidence interval $I(C_{ij})$, associated with each identified stiffness component, shall be determined by a statistical analysis of the experimental velocities in each propagation plane.

10.4.2 Calculation of C_{33}

The C_{33} stiffness component is directly computed using the velocity V measured in normal incidence (e.g. angle $\theta_i = 0°$), with $\lambda = \rho V^2$ and with $\theta_i = \theta_r = 0$, $n_p = (0, 0, 1)$, $\Gamma_{33} - \lambda = 0$, and $\Gamma_{33} = C_{33}$ follows:

$$C_{33} = \rho V^2 \tag{6}$$

where

ρ is the bulk density, in kilogrammes per cubic metre (kg/m^3).

10.4.3 Calculation of C_{22}, C_{23} and C_{44}

The stiffness components, C_{22}, C_{23} and C_{44}, are determined from the velocity measurements recorded for the acquisition plane (2, 3) with $\psi = 0°$ as follows:

For this plane, $n_p = (0, \sin\theta_r, \cos\theta_r)$ and $\Gamma_{12} = \Gamma_{13} = 0$.

Thus, Formula (7) is minimized:

$$F\left[C_{22}, C_{23}, C_{44}\right] = \sum_{p=1}^{M}\left[\lambda^2 - \lambda\left(\Gamma_{22} + \Gamma_{33}\right) + \Gamma_{22}\Gamma_{33} - \Gamma_{23}^2\right]^2 \tag{7}$$

where M is the total number of measured velocities of a range of angles of incidence θ_i, each corresponding to a different propagation direction n_p.

With $\lambda_p = \rho V_p^2$ using the velocity measurements and $\Gamma_{33} = C_{33}$ from 10.4.2 follow C_{22}, C_{23} and C_{44}.

10.4.4 Calculation of C_{11}, C_{13} and C_{55}

The stiffness components, C_{11}, C_{13} and C_{55}, are determined from the velocity measurements recorded for the acquisition plane (1,2) with $\psi = 90°$ as follows:

For this plane, $n_p = (\sin\theta_r, 0, \cos\theta_r)$ and $\Gamma_{12} = \Gamma_{23} = 0$.

Thus, Formula (8) is minimized:

$$F\left[C_{11}, C_{13}, C_{55}\right] = \sum_{p=1}^{M}\left[\lambda^2 - \lambda\left(\Gamma_{11} + \Gamma_{33}\right) + \Gamma_{11}\Gamma_{33} - \Gamma_{13}^2\right]^2 \tag{8}$$

where M is the total number of measurements of a range of angles of incidence θ_i, each corresponding to a different propagation direction n_p.

With $\lambda_p = \rho V_p^2$ using the velocity measurements and $\Gamma_{33} = C_{33}$ from 10.4.2, then C_{11}, C_{13} and C_{55} can be calculated.

10.4.5 Calculation of C_{12} and C_{66}

The two remaining stiffness components, C_{12} and C_{66}, are determined using the velocities measured in the non-principal plane $\psi = 45°$ and the seven stiffness components determined here above.

For this plane $n_p = (\sin\theta_r / \sqrt{2}, \sin\theta_r / \sqrt{2}, \cos\theta_r)$.

Thus, Formula (9) is minimized:

$$F\left[C_{12},C_{66}\right]=\sum_{p=1}^{M}\begin{bmatrix}-\lambda^3+\lambda^2(\Gamma_{11}+\Gamma_{22}+\Gamma_{33})\\+\lambda(\Gamma_{12}^2+\Gamma_{13}^2+\Gamma_{23}^2-\Gamma_{11}\Gamma_{22}-\Gamma_{11}\Gamma_{33}-\Gamma_{22}\Gamma_{33})\\+\Gamma_{11}\Gamma_{22}\Gamma_{33}+2\Gamma_{12}\Gamma_{13}\Gamma_{23}-\Gamma_{11}\Gamma_{23}^2-\Gamma_{22}\Gamma_{13}^2-\Gamma_{33}\Gamma_{12}^2\end{bmatrix}^2 \tag{9}$$

where M is the total number of measurements of a range of angles of incidence θ_i, each corresponding to a different propagation direction n_p.

The number of modes that can experimentally be obtained in this plane differs according to the level of material symmetry and influences the quality of the identification process.

— Orthotropic symmetry (e.g. 2 < × ≤ 3D composites with nine independent elastic constants): the two quasi transverse waves are generated and can be obtained experimentally.

— Quadratic symmetry (e.g. 2D composites with six independent elastic constants):

  Materials having this level of symmetry, such as some composites with a balanced weave as reinforcement, have two equivalent directions. Thus, the plane ψ = 45° is a plane of symmetry. In this plane, only one transverse mode is generated and the stiffness components C_{12} and C_{66} cannot be determined independently. Only the linear combination $C^* = C_{12} + 2\ C_{66}$ can be calculated from the experimental data gathered in the plane ψ = 45 °.

  An additional piece of information is then needed. Using a method requiring physical contact (i.e. a method different from the one described in this document), the measurement of the velocity of a bulk wave, which propagates along direction 1 and polarized along direction 3, then allows one to identify directly the C_{66} stiffness component. The remaining C_{12} component is then identified using the eight stiffness components already determined and from the experimental data obtained in the ψ = 45° plane.

— Hexagonal symmetry (also called transversely isotropic symmetry, e.g. UD composites with five independent elastic constants): materials with this level of symmetry have a symmetry axis parallel to the longitudinal fibre direction and an isotropy plane normal to this direction. The two transverse modes are generated and can be experimentally obtained in the ψ = 45° plane.

— Isotropic symmetry: for materials having this level of symmetry, all directions are equivalent. All the planes of this kind of body are planes of symmetry. In each acquisition plane, only one transverse mode is stimulated. The linear combination C^* does not allow one to determine the nine stiffness components C_{ij} independently, because of the symmetry of the three acquisition planes. This difficulty, raising from the sensitivity of the stiffness components to the experimental data, does not exist when the two moduli representing the isotropic symmetry are required.

10.5 Polar plots of the velocity curves

An example currently used for the presentation of the results is shown in Annex A. In all of the cases, polar phase velocity plots are presented for different ψ angles. The symbols are stated for the phase velocity measurements at the different propagation directions n_p, on which the calculation of the stiffness components have been based. The solid lines indicate the back calculated phase velocities (eigenvalues of the wave propagation tensor) for different propagation directions using the calculated stiffness components. The velocity curves, together with the experimental data at different ψ, allow one to determine whether appropriate mode separation has been achieved.

After the calculation of the stiffness components, C_{ij}, is performed, these shall be introduced into Formula (10):

$$\mathrm{Det}\left(\Gamma_{ij} - \rho V^2 \delta_{ij}\right) = 0 \tag{10}$$

and the phase velocities, $V_p{}^c$, for the different propagation directions, n_p, are calculated as the eigenvalues of the propagation tensor, Γ_{ij}, using Formula (11):

$$V_p^c = \left(\frac{\lambda_p}{\rho}\right)^{1/2} \tag{11}$$

where λ_p are the calculated eigenvalues.

This back calculation of the phase velocities covers the complete range of refracted angles for all the used planes of incidence (ψ = 0°, 45° and 90°).

10.6 Calculation of the quadratic deviation and the confidence interval

During a characterization, it is necessary to quantify the level of uncertainty on the measured phase velocities through the use of the quadratic deviation of the experimental data around the identified solution.

The quadratic deviation σ shall be calculated with the following expression obtained from the experimental velocity set and velocities that are computed from the calculated stiffness tensor:

$$\sigma = \sqrt{\frac{1}{M}\sum_{p=1}^{M}\left(\frac{V_p^c - V_p}{V_p^c}\right)^2} \tag{12}$$

To quantify the sensitivity of the inversion algorithm for identifying elasticity constants from velocity data, a confidence interval, i.e. the variances associated with each identified stiffness component, should be determined by a statistical analysis of the experimental velocities measured in each propagation plane.

Since these confidence intervals are sensitive to the level of experimental data scatter and to the angle range of the velocity data, they enable one to establish the reliability of ultrasonic characterisation. This quantifies the sensitivity of the inversion algorithm for identifying elasticity constants from velocity data.

The different values of the confidence interval $I(C_{ij})$ are computed from the linearization of expression $F\left[C_{ij}\right] = \sum_{p=1}^{M}\left[f\left[C_{ij}, n_p\left(n_p\right)\right]\right]^2$, around the exact solution for each of the measured values using an appropriate computer programme.

10.7 Calculation of the engineering constants

Having the components, C_{ij}, of the stiffness matrix from the above described procedure, the following steps are needed for the determination of the engineering constants of an orthotropic material in general.

a) The components of the compliance tensor shall be calculated using the following relations:

$S_{11} = (C_{22}\, C_{33} - C_{23}{}^2)/C$ $\quad S_{12} = (C_{13}\, C_{23} - C_{12}\, C_{33})/C$ $\quad S_{44} = 1/C_{44}$

$S_{22} = (C_{33}\, C_{11} - C_{13}{}^2)/C$ $\quad S_{13} = (C_{12}\, C_{23} - C_{13}\, C_{22})/C$ $\quad S_{55} = 1/C_{55}$

$S_{33} = (C_{11}\, C_{22} - C_{12}{}^2)/C$ $\quad S_{23} = (C_{12}\, C_{13} - C_{23}\, C_{11})/C$ $\quad S_{66} = 1/C_{66}$

with

$C = C_{11}\, C_{22}\, C_{33} - C_{11}\, C_{23}^2 - C_{22}\, C_{13}^2 - C_{33}\, C_{12}^2 - 2\, C_{12}\, C_{23}\, C_{13}$

b) The engineering constants shall be calculated using the following relations:

$E_{11} = 1/S_{11}$	$G_{12} = 1/S_{66}$	$\nu_{12} = -S_{12}/S_{11}$
$E_{22} = 1/S_{22}$	$G_{13} = 1/S_{55}$	$\nu_{13} = -S_{13}/S_{33}$
$E_{33} = 1/S_{33}$	$G_{23} = 1/S_{44}$	$\nu_{23} = -S_{23}/S_{22}$

11 Test validity

11.1 Measurements

— Bulk waves shall be clearly identified and separated from other modes of propagation.

— *QL* and *QT* shall be separated.

11.2 Criterion of validity for the reliability of the C_{ij} components

— The quadratic deviation shall be less than 0,75 %.

NOTE When this condition is met, then the stiffness matrix determined by the procedure described in 10.4 is assumed to correspond to the effective elastic properties of the material under study.

12 Test report

The test report shall be in accordance with the reporting provisions of ISO/IEC 17025 and shall contain at least the following information:

a) name and address of the testing establishment;

b) date of test;

c) on each page, a unique report identification and page number;

d) customer name and address;

e) reference to this document, i.e. ISO 18610;

f) an authorizing signature;

g) any deviation from the method described, with appropriate validation, i.e. demonstrated to be acceptable to the parties involved;

h) drawing of the test object or its reference;

i) complete identification of the material tested including type, source, manufacturer’s code number, batch number;

j) description of the testing equipment: immersion tank, coupling fluid, probes, electronic instruments;

k) test frequency used;

l) temperature at which the test was performed;

m) identification of the coordinate system (see Annex A);

n) velocity curves including experimental values and back-calculated values from identified stiffness components C_{ij} (see Annex A);

o) the stiffness components (see Annex A);

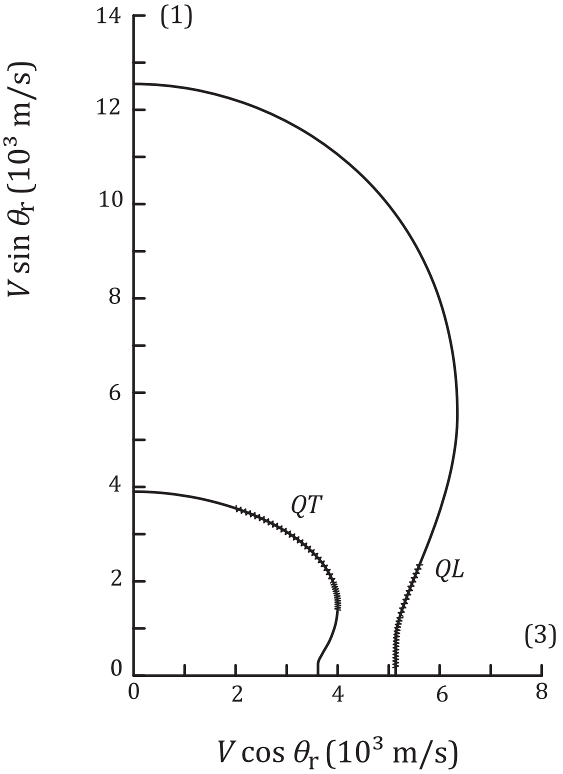
p) quadratic deviation;

q) engineering constants, if required.

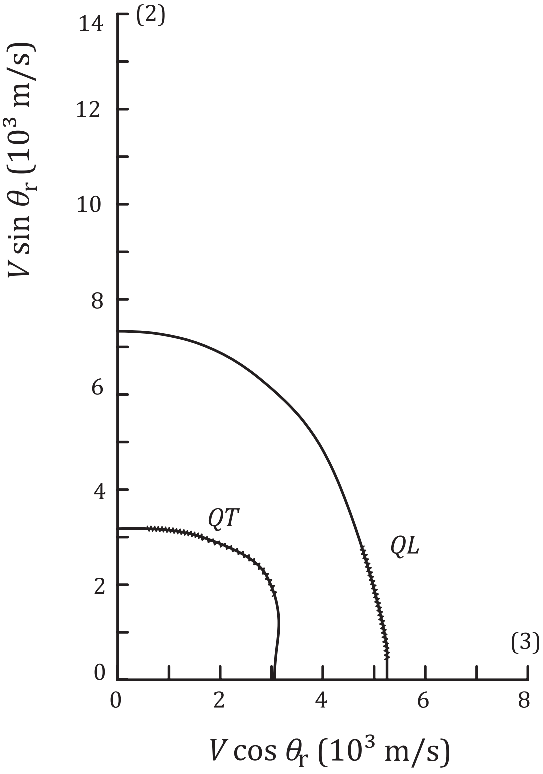
Annex A
(informative)

Example of a presentation of the results for a material with orthotropic symmetry

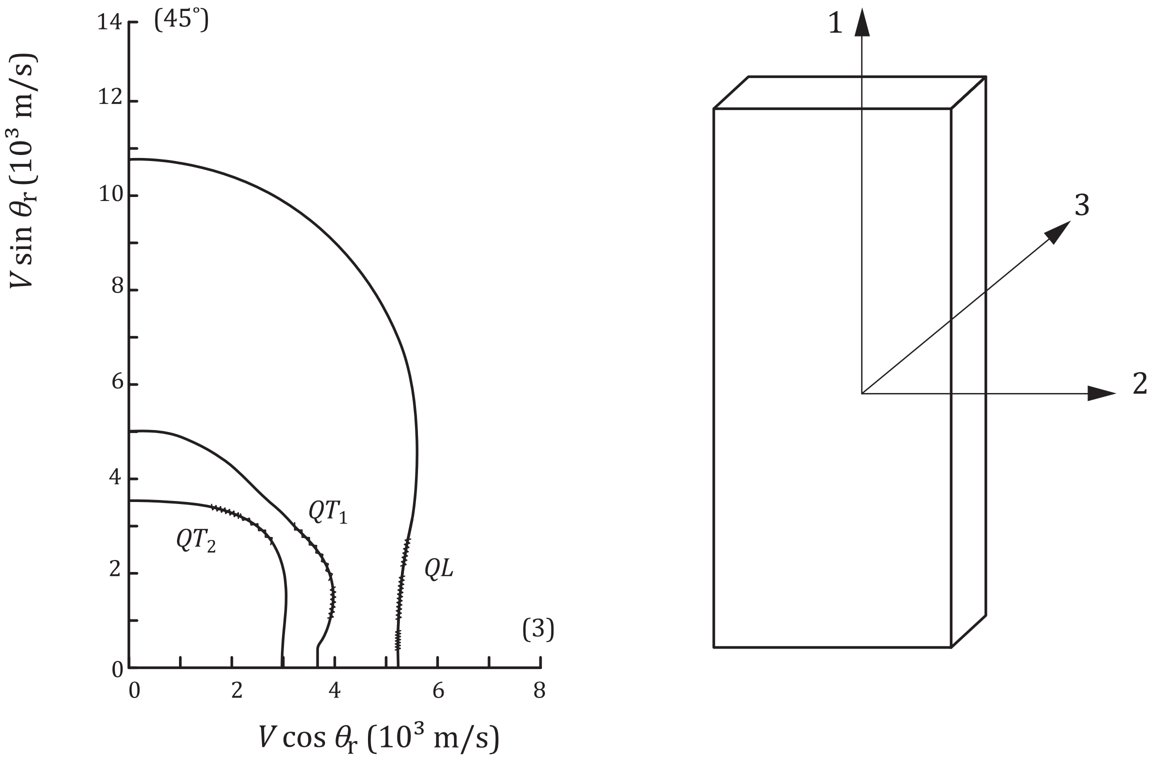
A.1 Velocity curves



a) Principal plane (1, 3) with ψ = 90°



b) Principal plane (2, 3) with ψ = 0°



c) Plane for ψ = 45°

d) Three directions

NOTE The crosses represent measured values, the solid lines are the velocities back calculated from the identified stiffness components.

Figure A.1 — Velocities in the a) (1,3), b) (2,3) and c) (3,45°) planes

A.2 Stiffness matrix with stiffness components

The values between brackets represent the 90 % confidence interval for each component.

The components are given in gigapascals (GPa).

$$\left[C_{ij}\right] = \begin{bmatrix} C_{11} = 396(23) & C_{12} = 98(10) & C_{13} = 35(3) & 0 & 0 & 0 \\ & C_{22} = 13(45) & C_{23} = 29(2) & 0 & 0 & 0 \\ & & C_{33} = 76(1) & 0 & 0 & 0 \\ & & & C_{44} = 24,6(0,3) & 0 & 0 \\ & \text{Sym} & & & C_{55} = 37,4(0,6) & 0 \\ & & & & & C_{66} = 81(5) \end{bmatrix}$$

A.3 Engineering constants

E_{11} = 323 GPa E_{22} = 105 GPa E_{33} = 70 GPa

G_{12} = 81 GPa G_{13} = 37,4 GPa G_{23} = 24,6 GPa

ν_{12} = 0,22 ν_{13} = 0,04 ν_{23} = 0,18

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