

English Version

Advanced technical ceramics - Mechanical properties of ceramic composites at room temperature - Determination of elastic properties by an ultrasonic technique

Céramiques techniques avancées - Propriétés mécaniques des céramiques composites à température ambiante - Détermination des propriétés élastiques par une méthode ultrasonore

Hochleistungskeramik - Mechanische Eigenschaften keramischer Verbundwerkstoffe bei Raumtemperatur - Bestimmung von elastischen Eigenschaften mittels Ultraschallwellen

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## Foreword

This document (EN 14186:2007) has been prepared by Technical Committee CEN/TC 184 "Advanced technical ceramics", the secretariat of which is held by BSI.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by May 2008, and conflicting national standards shall be withdrawn at the latest by May 2008.

This document supersedes ENV 14186:2002.

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## 1 Scope

This European Standard 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 European Standard applies to ceramic matrix composites with a continuous fibre reinforcement: unidirectional (1D), bidirectional (2D), and tridirectional ( $\times D$ , 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 wave length 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 EN 658-1, EN 658-2 and EN 12289.

## 2 Normative references

The following referenced documents are indispensable for the application 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.

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

CEN/TR 13233:2007, *Advanced technical ceramics — Notations and symbols*

EN ISO/IEC 17025, *General requirements for the competence of testing and calibration laboratories (ISO/IEC 17025:2005)*

ISO 3611, *Micrometer callipers for external measurements*

## 3 Terms and definitions

For the purposes of this document, the terms and definitions given in CEN/TR 13233:2007 and the following apply.

### 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 ( $6 \times 6$ )

NOTE 1 If the material has at least orthotropic symmetry, its elastic behaviour is fully characterised 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 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:

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

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;

$\nu_{12}$ ,  $\nu_{13}$ ,  $\nu_{23}$  are the respective Poisson coefficients

### 3.3 angle of incidence

$\theta_i$

angle between the direction 3 normal to the test specimen front face and the direction  $n_i$  of the incident wave (see Figure 1 and Figure 2)

**3.4**  
**refracted angle**

$\theta_r$

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 (see Figure 1 and Figure 2)

**3.5**  
**azimuthal angle**

$\psi$

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 (see Figure 1)

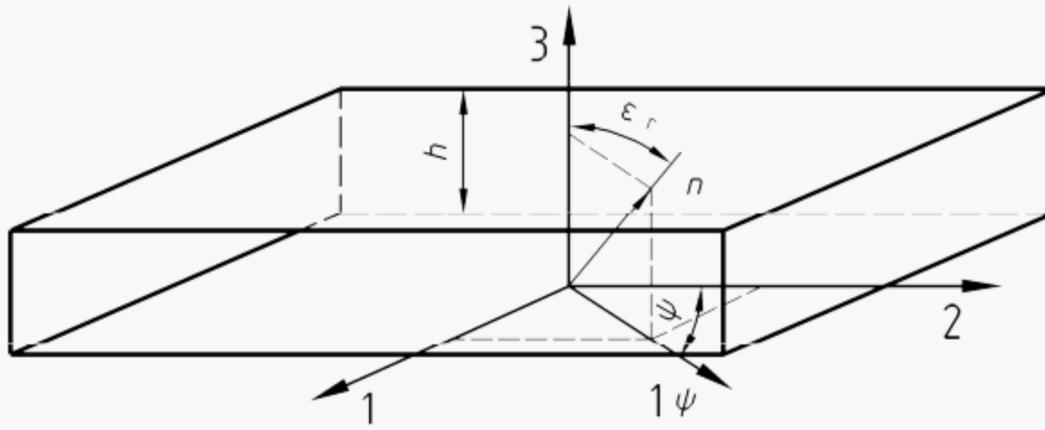


Figure 1 — Definition of the angles

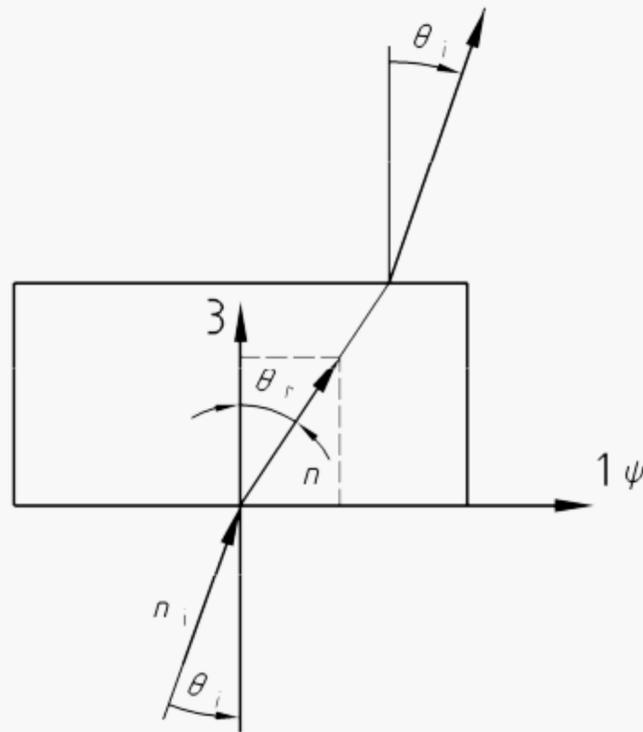


Figure 2 — Propagation in the plane of incidence

**3.6****unit vector***n*

unit vector oriented along the propagation direction of the incident plane wave inside the specimen, with its components  $n_k$  ( $k = 1, 2, 3$ ) (see Figure 1 and Figure 2):

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

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

$$n_3 = \cos \theta_r$$

**3.7****propagation velocity** $V(n)$ 

phase velocity of a plane wave inside the specimen in dependence on unit vector  $n$  (i.e. in dependence on  $\psi$  and  $\theta_r$ )

*NOTE*  $V_0$  is the propagation velocity in the coupling fluid.

**3.8****delay** $\delta t(n)$ 

difference between the flight time of the wave when the test specimen is in place and the flight time of the wave in the coupling fluid with the test specimen removed under the same configuration of the transducers in dependence on unit vector  $n$

**3.9****thickness of the test specimen***h*

thickness of the test specimen

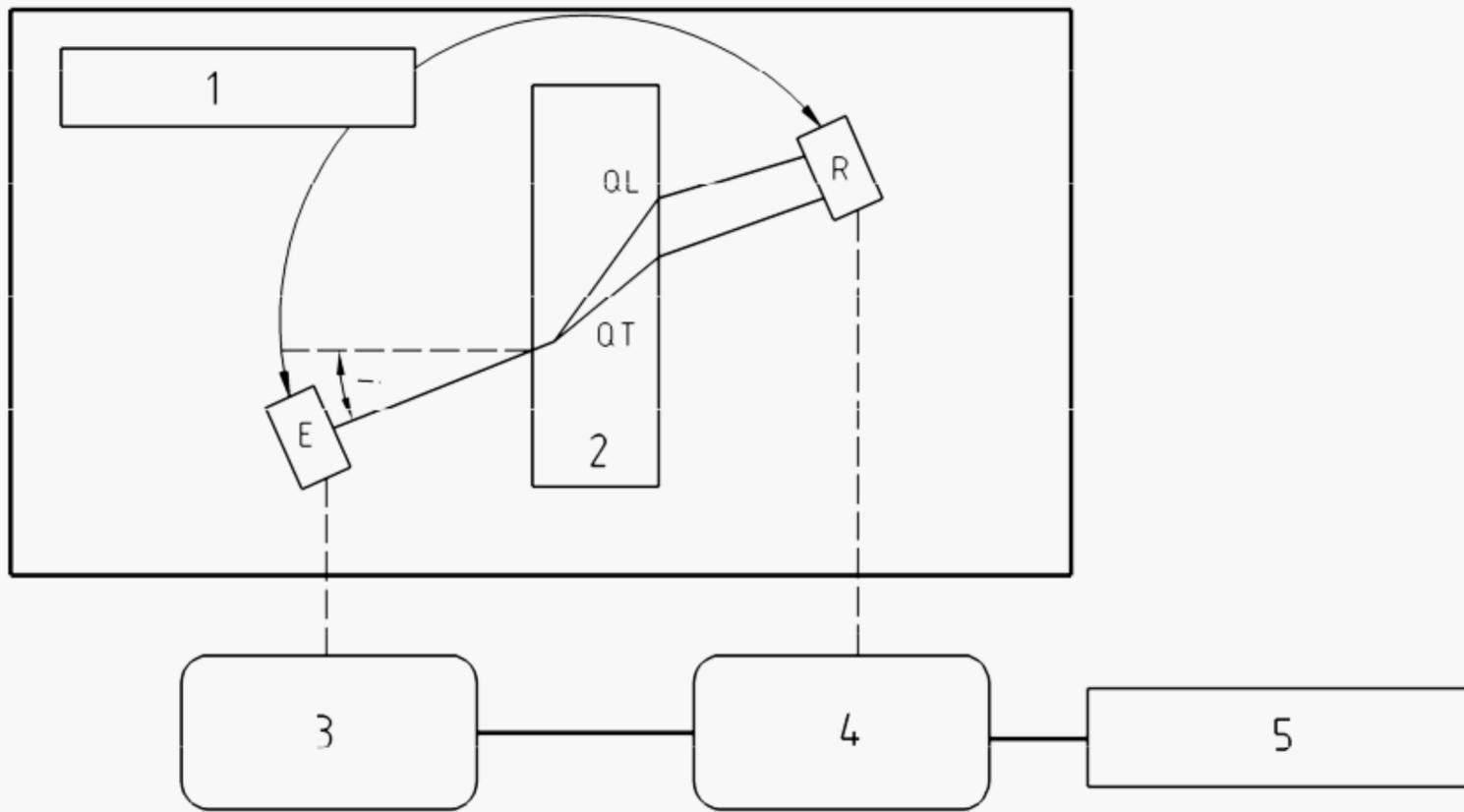
**3.10****bulk density** $\rho_b$ 

bulk density of the specimen

**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 an emitter (E) 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**

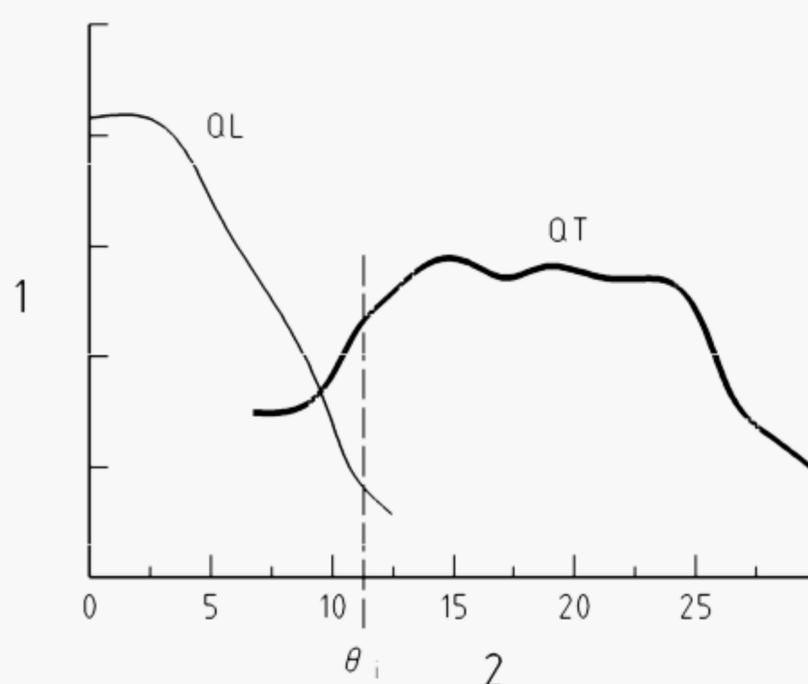
- 1 rotation drive
- 2 test specimen
- 3 pulse generator
- 4 digital oscilloscope
- 5 micro-computer

**Figure 3 — Ultrasonic test assembly**

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

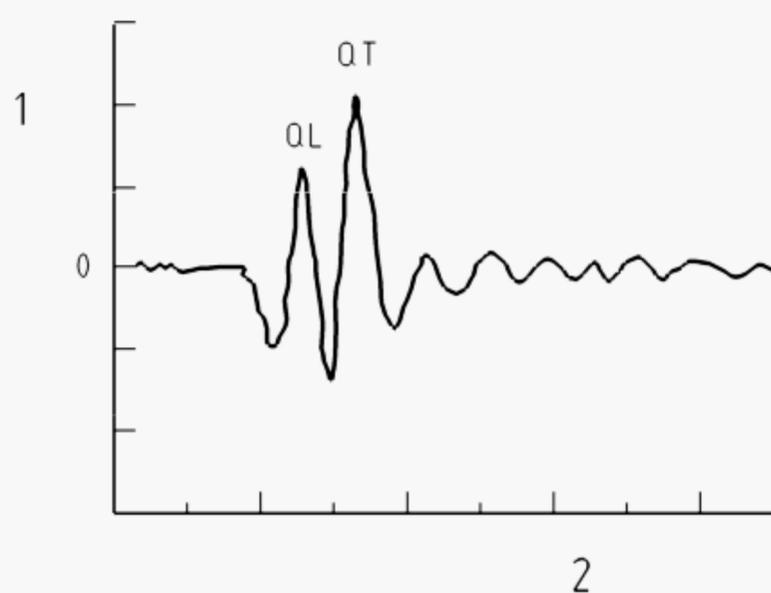
The receiver R collects one, two or three pulses, corresponding to each of these waves.

The difference in propagation time of each of the waves and the propagation time of the emitted pulse in the coupling fluid without the specimen is measured. The evaluation procedure is based on the measurement of the time difference 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**

- 1 amplitude
- 2 incidence angle

**Figure 4a) — Amplitude of the  $QL$  and  $QT$  waves as a function of the incidence angle**

**Key**

- 1 amplitude
- 2 time

**Figure 4b) — Temporal waveform of the overlapping  $QL$  and  $QT$  waves at an incidence angle  $\theta_i$**

**Figure 4 — Overlapping of  $QL$  and  $QT$  waves at an incidence angle  $\theta_i$**

From the propagation velocities the components of the elasticity tensor are obtained through a least square regression analysis which minimises 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 (Lamé's coefficients or Young's modulus and Poisson coefficient) are sufficient in order to fully describe the elastic behaviour of an isotropic 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.

Contrary to mechanical test methods, the determination of elastic properties by the ultrasonic method described here is not based on the evaluation of the stress-strain response over a given deformation range obtained under quasi-static loading conditions, but is based on a non-destructive dynamic measurement of wave propagation velocities. Therefore the values of Young's moduli, shear moduli and Poisson ratios determined by the two methods might not be comparable, particularly for ceramic matrix composites that exhibit non linear stress-strain behaviour.

NOTE Mechanical test methods are based on a measurement performed under isothermal conditions, whereas the ultrasonic method assumes adiabatic conditions.

In addition to the ultrasonic method described here, there also exist other non destructive methods to determine the elastic properties, for instance the resonant beam technique and the impulse excitation method. Each of these has its relative merits and disadvantages. The selection of a particular non destructive method shall be considered on a case-by-case basis.

## 6 Apparatus

### 6.1 Ultrasonic tank with thermostatic control

The ultrasonic tank shall be capable of maintaining the temperature of the coupling fluid constant to within  $\pm 0,1$  °C for the full duration of the test.

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

### 6.2 Temperature measurement device

The temperature measurement device shall be capable of measuring the temperature to within 0,1 °C, e.g. as set out in ISO 653.

### 6.3 Test specimen holder

The test specimen holder shall allow rotation of the test specimen around one axis to cover the range of angles of incidence  $\theta_1$  between 0 ° and 90 °. Additionally it shall allow for discrete settings of the azimuthal angle  $\Psi$  of 0 °, 45 ° and 90 °. The accuracy in the measurement of the angles  $\theta_1$  and  $\Psi$  shall be better than 0,01 ° and 1 ° respectively.

NOTE The accuracy required for the measurement of the angle of incidence  $\theta_1$  depends on the nature of the coupling fluid and is higher when using air as the coupling fluid. Commercially available goniometers with automatic positioning are commonly used for this purpose.

## 6.4 Transducers

Use piezoelectric broad-band transducers adapted to the coupling fluid and able to generate longitudinal ultrasonic waves. Two identical transducers are used as emitter and receiver.

## 6.5 Transducer holders

The transducer holders shall allow the transducers to be oriented towards each other. The transducers are mounted in such a way that their relative position remains fixed during the test.

## 6.6 Pulse generator

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

It shall be able to generate short-duration ( $< 1 \mu\text{s}$ ) pulses of voltage sufficient to provide a mechanical pulse through 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 been dissipated before initiating the next.

## 6.7 Signal recording system

Use any system, for instance: digital oscilloscope or dynamic analogue/digital board, with a minimum sampling frequency of 100 MHz that allows the recording of emitted and received signals. The signal recording system is designed in order to allow 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 specimens

The choice of test specimen geometry 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 specimen shall be at least twice the diameter of the transducer. A plate with parallel faces is recommended. The plane parallelism of the two faces shall be better than 0,05 mm.

## 8 Test specimen 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 specimens shall be clearly defined and recorded. These procedures shall be followed during machining of the test specimens.

NOTE 1 Usually, plate test specimens are cut with their longitudinal axis coinciding with one of the principal directions of the reinforcement.

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

Care shall be taken to avoid interaction between the coupling fluid and the test specimen (ingress into open porosity, chemical instability, absorption phenomena etc.).

NOTE 2 This can for instance be achieved by sealing the test specimen in an evacuated plastic bag, or by applying an appropriate coating.

## 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 size of the RVE in the direction of normal incidence, is proposed ( $\theta_1 = 0$ ).

NOTE 2 Because of the inverse relationship between wavelength  $\lambda$  and frequency  $f$  ( $f = \frac{V}{\lambda}$ ), this corresponds to a wavelength  $\lambda$  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  $\theta_1 = 0$ . 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  $\theta_1$  [see Figure 4b)]. This is promoted by increasing the frequency.

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

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 specimen thickness beyond the minimum thickness stipulated in clause 7.

NOTE 4 For example for a 2D SiC/SiC with a RVE of 0,5 mm (requiring a minimum test specimen thickness of 2,5 mm in accordance with clause 7), the transducer 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  $\frac{3V}{2h} = 3$  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

Switch on the thermostatic control to establish the required temperature of the coupling fluid. 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. Mount the test specimen in the test specimen holder in accordance with 9.4.2. Measure the temperature in the vicinity of the test specimen. Make sure that the temperature falls within  $\pm 0,1$  °C from that of the reference measurement. Perform the test in accordance with 9.4.

### 9.3 Reference test without test specimen

Record the signals from the emitter and from the receiver versus time without a test specimen mounted.

## 9.4 Measurement with the specimen

### 9.4.1 Measurement of the bulk density and of the thickness

#### 9.4.1.1 Measurement of the bulk density

Measure the bulk density in accordance with EN 1389.

#### 9.4.1.2 Measurement of the thickness

Measure the thickness in three positions on the test specimen with a micrometer with an accuracy of 0,01 mm in accordance with ISO 3611.

### 9.4.2 Mounting of the specimen

The specimen shall be oriented perpendicularly to the incoming beam. The accuracy of the perpendicularity between the beam and the specimen shall be 0,1 °.

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

### 9.4.3 Acquisition of different angles of incidence

Set acquisition plane by selecting azimuthal angle  $\Psi = 0^\circ$ ,  $45^\circ$  and  $90^\circ$ . For each acquisition plane measurements are made of the  $QL$  and  $QT$  signals at given values of the angle of incidence  $\theta_1$ . The incidence angle  $\theta_1$  varies from  $0^\circ$  up to a maximum defined by a decrease of the amplitude of the  $QL$  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.

NOTE 1 Over the total range of  $\theta_1$  usually a minimum of 20 measurements is performed.

In the angular range where  $QL$  and  $QT$  overlap the step in angle  $\theta_1$  shall be reduced.

NOTE 2 This range of  $\theta_1$  can be defined as  $\pm 5^\circ$  from the incidence angle where  $QL$  and  $QT$  have the same amplitude. In this range the step is set at  $0,5^\circ$ .

NOTE 3 Maximum  $\theta_1$  is also configuration limited.

Only signals recorded at values of  $\theta_1$  and  $\Psi$  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 4 This is usually verified by representing the experimental results by the velocity curves as shown in Annex A.

NOTE 5 Signal stability should be secured by repeating the experiment under given conditions ( $\Psi$ ,  $\theta_1$ ) at different time periods.

## 10 Calculation

### 10.1 Delay

For each value of  $\Psi$  and  $\theta_i$ , 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 specimen is in the coupling fluid.

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 the following equation:

$$V(n) = \frac{V_0}{\sqrt{1 + \frac{V_0 \delta t(n)}{h} \left( \frac{V_0 \delta t(n)}{h} - 2 \cos \theta_i \right)}} \quad (1)$$

where

- $V(n)$  is the propagation velocity in the material, in metres per second ( $\text{m}\cdot\text{s}^{-1}$ );
- $V_0$  is the propagation velocity in the coupling fluid, in metres per second ( $\text{m}\cdot\text{s}^{-1}$ );
- $h$  is the specimen thickness, in metres (m);
- $\delta t(n)$  is the delay, in seconds (s);
- $\theta_i$  is the angle of incidence, in degrees ( $^\circ$ ).

### 10.3 Calculation of the refracted angle $\theta_r$

$$\theta_r = \arcsin \left[ \frac{(V_n \times \sin \theta_i)}{V_0} \right] \quad (2)$$

where

- $\theta_r$  is the refracted angle, in degrees ( $^\circ$ ).

### 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  $\Gamma_{ij}$  according to the following equation:

$$\text{Det}(\Gamma_{ij} - p_b V_2(n) \delta_{ij}) = 0 \quad (3)$$

and the polarization directions are the corresponding eigenvectors, with  $\delta_{ij}$  Kronecker's symbol and  $p_b$  the bulk density.

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

$$\Gamma_{ij} = C_{ijkl} n_k n_l \quad (4)$$

where

$C_{ijkl}$  are the components of the stiffness matrix (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  $\Gamma_{ij}$  have the following form:

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

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

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

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

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

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

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

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

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

At each value of  $\psi$  a set of  $N$  measurements, for different angles of incidence  $\theta_i$  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  $\lambda(n_p) = \rho_b(V(n_p))^2$ .

Then the minimisation of the following expression is necessary:

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

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

NOTE 2 To minimise 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 to 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, with  $\lambda = \rho_b V^2$  and with  $\theta_i = \theta_r = 0$ ,  $n_p = (0, 0, 1)$ ,  $\Gamma_{33} - \lambda = 0$ , and  $\Gamma_{33} = C_{33}$  follows:

$$C_{33} = \rho_b V^2 \quad (6)$$

where

$\rho_b$  is the bulk density, in kilogrammes per cubic metre ( $\text{kg/m}^3$ ).

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

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

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

Thus the following equation is minimised:

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

where

$p = 1, 2, \dots, M$ ;

$M$  is the total number of measured velocities of a range of incident angles  $\theta_i$ , each corresponding to a different propagation direction  $n_p$ .

With  $\lambda_p = \rho_b V_p^2$  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 identified from the velocity measurements recorded for the acquisition plane (1,2) with  $\psi = 90^\circ$  as follows.

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

Thus the following equation is minimised:

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

where

$p = 1, 2, \dots, M$ ;

$M$  is the total number of measurements of a range of incident angles  $\theta_i$ , each corresponding to a different propagation direction  $n_p$ .

With  $\lambda_p = \rho_b V_p^2$  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 identified using the velocities measured in the non-principal plane  $\psi = 45^\circ$  and the seven stiffness components determined here above.

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

Thus the following equation is minimised:

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

where

$$p = 1, 2, \dots, M;$$

$M$  is the total number of measurements of a range of incident angles  $\theta_i$ , 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 < x ≤ 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  $\psi = 45^\circ$  is a plane of symmetry. In this plane only one transverse mode is stimulated 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  $\psi = 45^\circ$ .

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 European Standard), the measurement of the velocity of a bulk wave, which propagates along direction 1 and polarised along direction 3, then allows 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  $\psi = 45^\circ$  plane.

- Hexagonal symmetry (also called transversely isotropic symmetry, e.g. UD composites with five independent elastic constants): materials with this level of symmetry, such as the unidirectional composites, have a symmetry axis parallel to the longitudinal fibre direction and an isotropy plane normal to this direction. The two transverse modes are stimulated and can be experimentally obtained in the  $\psi = 45^\circ$  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 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.

**NOTE** In order to determine the accuracy of these three inverse problems and 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.

### 10.5 Back calculation of the phase velocities

After the calculation of the stiffness components  $C_{ij}$  is performed, these shall be introduced into the equation:

$$\text{Det}(\Gamma_{ij} - \rho_b V^2 \delta_{ij}) = 0 \quad (10)$$

and the phase velocities for the different propagation directions  $n_p$ ,  $V_p^c$  is calculated as the eigenvalues of the propagation tensor  $\Gamma_{ij}$  using the equation:

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

where  $\lambda_p$  are the calculated eigenvalues.

This back calculation of the phase velocities covers the complete range of refraction angles for all the used planes of incidence ( $\psi = 0^\circ, 45^\circ$  and  $90^\circ$ ).

### 10.6 Polar plots of the velocity curves

An example currently used for the presentation of the results is shown in Annex A. In all the cases polar phase velocity plots are presented for different  $\psi$  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  $\psi$  allow to determine whether appropriate mode separation has been achieved.

### 10.7 Calculation of the quadratic deviation

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

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

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

Calculation of the confidence interval  $I(C_{ij})$ .

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

### 10.8 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) Calculate the components of the compliance matrix using the following relations:

$$\begin{aligned}
 S_{11} &= (C_{22} C_{33} - C_{23}^2)/C & S_{12} &= (C_{13} C_{23} - C_{12} C_{33})/C & S_{44} &= 1/C_{44} \\
 S_{22} &= (C_{33} C_{11} - C_{13}^2)/C & S_{13} &= (C_{12} C_{23} - C_{13} C_{22})/C & S_{55} &= 1/C_{55} \\
 S_{33} &= (C_{11} C_{22} - C_{12}^2)/C & S_{23} &= (C_{12} C_{13} - C_{23} C_{11})/C & S_{66} &= 1/C_{66}
 \end{aligned}$$

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) Calculate the engineering constants using the following relations:

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

## 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 EN 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 European Standard, i.e. determined in accordance with EN 14186;
- f) an authorising signature;
- g) any deviation from the method described, with appropriate validation, i.e. demonstrated to be acceptable to the parties involved;
- h) specimen drawing or its reference;

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- i) complete identification of the material tested including type, source, manufacturer's code number, batch number;
- j) description of the testing assembly: ultrasonic tank, transducers, coupling fluid;
- k) 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

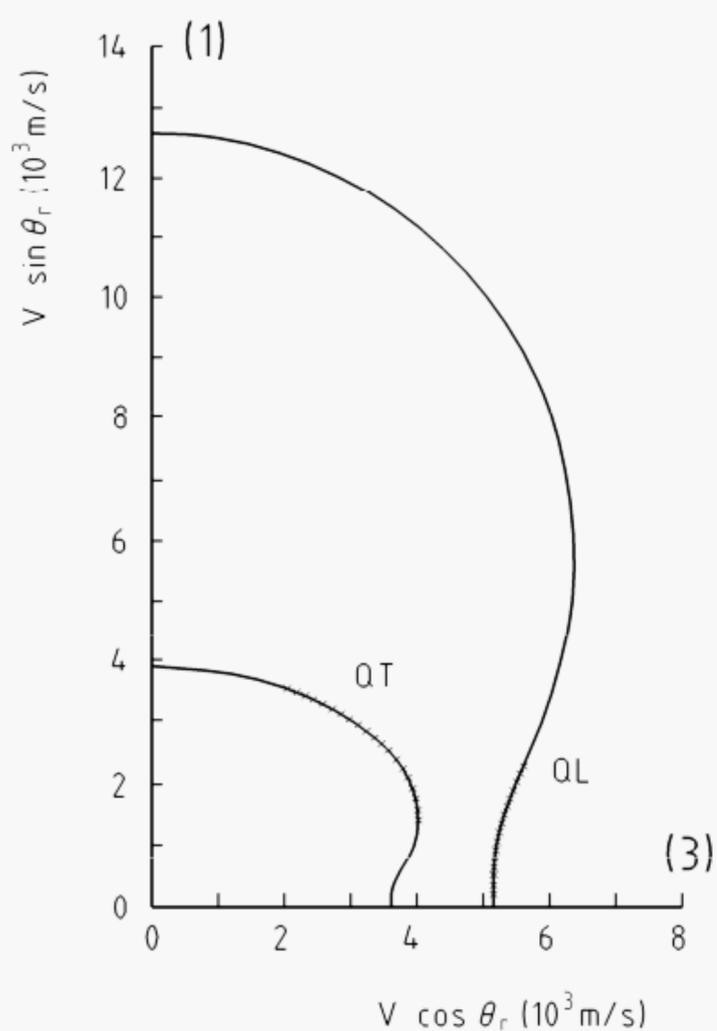


Figure A.1a) — Principal plane (1, 3) with  $\psi = 90^\circ$

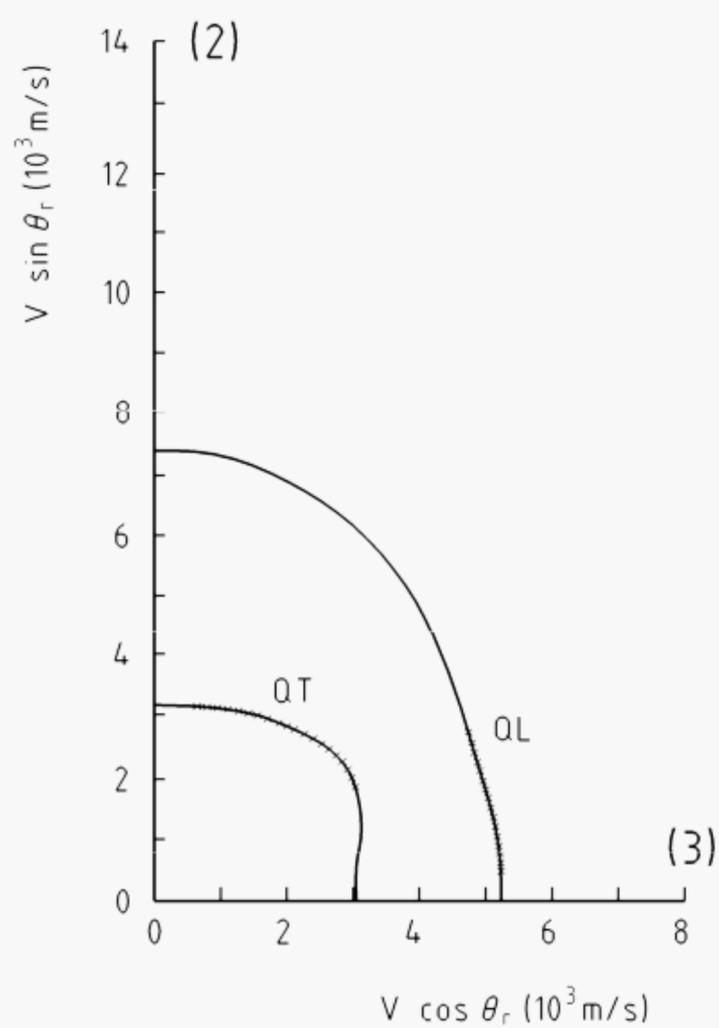


Figure A.1b) — Principal plane (2, 3) with  $\psi = 0^\circ$

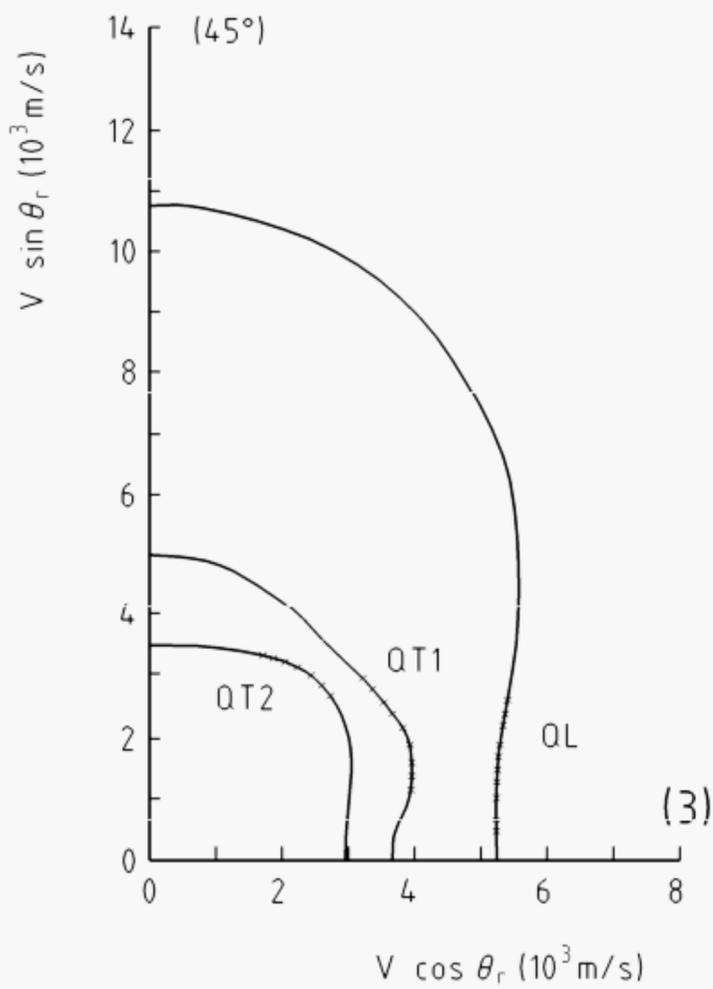


Figure A.1c) — Plane for  $\Psi = 45^\circ$

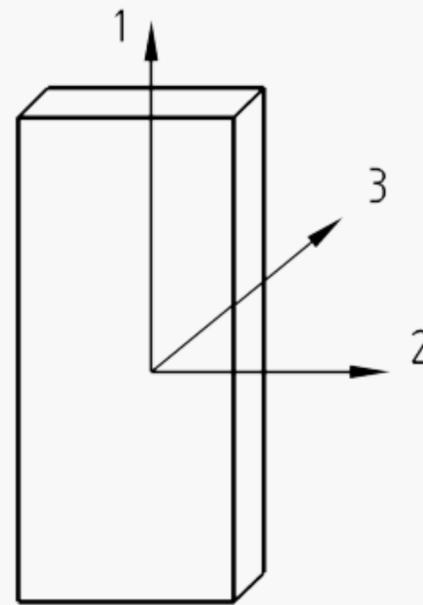


Figure A.1d) — Three directions

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 in gigapascals (GPa).

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

**A.3 Engineering constants**

$$E_{11} = 323 \text{ GPa}$$

$$G_{12} = 81 \text{ GPa}$$

$$\nu_{12} = 0,22$$

$$E_{22} = 105 \text{ GPa}$$

$$G_{13} = 37,4 \text{ GPa}$$

$$\nu_{13} = 0,04$$

$$E_{33} = 70 \text{ GPa}$$

$$G_{23} = 24,6 \text{ GPa}$$

$$\nu_{23} = 0,18$$

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