# SLOVENSKI STANDARD
# SIST EN 15335:2009

**01-februar-2009**

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**Sodobna tehniļ na keramika - Keramiļ ni kompoziti - Doloļ anje upogibnih lastnosti z resonanļ no metodo ģarka do 2000 ĆC**

Advanced technical ceramics - Ceramic composites - Determination of elastic properties by resonant beam method up to 2 000 °C

Hochleistungskeramik - Keramische Verbundwerkstoffe - Bestimmung der elastischen Eigenschaften bei Verwendung des Resonanz-Verfahrens bis 2 000 °C

Céramiques techniques avancées - Céramiques composites - Détermination des propriétés élastiques par une méthode de résonance sur poutres, jusqu'a 2 000 °C

**Ta slovenski standard je istoveten z:** **EN 15335:2007**

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**ICS:**

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| 81.060.30 | Sodobna keramika | Advanced ceramics |

**SIST EN 15335:2009** **en**

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# Advanced technical ceramics - Ceramic composites - Determination of elastic properties by resonant beam method up to 2 000 °C

Céramiques techniques avancées - Céramiques composites - Détermination des propriétés élastiques par une méthode de résonance sur poutres, jusqu'à 2 000 °C

Hochleistungskeramik - Keramische Verbundwerkstoffe - Bestimmung der elastischen Eigenschaften bei Verwendung des Resonanz-Verfahrens bis 2 000 °C

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EUROPEAN COMMITTEE FOR STANDARDIZATION
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# Foreword

This document (EN 15335:2007) 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of an identical text or by endorsement, at the latest by November 2007, and conflicting national standards shall be withdrawn at the latest by November 2007.

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard: Austria, Belgium, Bulgaria, Cyprus, Czech Republic, Denmark, Estonia, Finland, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Romania, Slovakia, Slovenia, Spain, Sweden, Switzerland and United Kingdom.

## 1 Scope

This European Standard specifies the resonant beam method for the determination of the dynamic elastic moduli of fibre reinforced ceramic matrix composites from 20 °C up to 2 000 °C in vacuum or inert atmosphere. The Young´s moduli and the shear moduli for different orientations with respect to the main axes of symmetry of the composite can be obtained.

This document applies to ceramic matrix composites with fibre reinforcement: short fibres, unidirectional (1D), bidirectional (2D), and tridirectional (xD, with $2 < x \leq 3$) which have at least orthothropic symmetry.

NOTE 1 Dynamic means that the elastic moduli are determined non-quasistatically, i.e. under adiabatic conditions, as with the ultrasonic method set out in ENV 14186. The elastic moduli determined by this method may not be compared with moduli obtained in an isothermal condition by stressing statically or quasistatically as with EN 658-1, EN 658-2, EN 1892, EN 1893, EN 12290 and EN 12291.

NOTE 2 The ceramic matrix composites with fibre reinforcement, listed above, are denoted as “composites” in the course of the document.

## 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 60584-1, *Thermocouples — Part 1: Reference tables (IEC 60584-1:1995)*

EN 60584-2, *Thermocouples — Part 2: Tolerances (IEC 60584- 2:1982 + A1:1989)*

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

ISO 3599, *Vernier callipers reading to 0,1 and 0,05 mm*

ISO 3611, *Micrometer callipers for external measurement*

## 3 Terms and definitions

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

### 3.1

**Young's modulus, $E$**

stress required in a material to produce unit strain in uniaxial extension or compression

### 3.2

**shear modulus, $G$**

shear stress required in a material to produce unit angular distortion

### 3.3

**Poisson's ratio, $\nu$**

ratio of transverse strain to the corresponding axial strain

NOTE $E_{11}$, $E_{22}$ 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 and $\nu_{12}$, $\nu_{13}$, $\nu_{23}$ are the respective Poisson ratios.

## 4 Principle

The test specimen, a long thin prismatic beam (ratio of length to width or length to height bigger than 10), cut from the composite along a specific orientation of interest, is excited to bending vibrations. The mechanical excitation at continuously variable frequencies is provided by means of a transducer that transforms cyclic electrical signals to cyclic mechanical forces on the test specimen. A second transducer senses the resulting mechanical vibrations of the test specimen and transforms them into electrical signals. The resulting spectrum (amplitude as function of frequency) up to frequencies enclosing the sixth mode of vibrations of the test specimen, is registered and stored in a spectrum analyser. From the resonant frequencies, i.e. the peaks of the spectrum (fundamental vibration and harmonics up to the sixth mode), the dimensions and the density of the test specimen, the elastic moduli can be calculated by numerically solving Timoshenko´s equation. For one test specimen the Young´s modulus and two shear moduli in directions perpendicular to each other can be achieved.

NOTE When anisotropy, a specific feature of composites, is taken into account, the elastic behaviour can be fully characterised only by the elasticity tensor with a certain number of independent coefficients, this number depending on the crystallographic symmetry of the composite (a method to determine the elasticity tensor for composites is given in ENV 14186). From these coefficients the elastic moduli (Young´s moduli, shear moduli and Poisson ratios) can be calculated.

## 5 Significance and use

The resonant beam method is frequently applied for the determination of the dynamic elastic moduli in standardisation [for example: EN 843-2, CEN/TS 820-5, ISO 17561, ASTM C848-88, ASTM C623-92(2005)]. For all these applications, it is common that the influence of shear, which is the bigger, the higher and the mode of vibration, is ruled out. For the calculation of results a formula basing on the Euler-Bernoulli relation, valid for the fundamental mode only, is applied and is combined with correction factors for higher modes (in general only the first mode, i.e. the first overtone is considered additionally to the fundamental mode). The shear moduli are determined separately from torsional vibrations.

The method specified in this document turns the problem around. The higher modes are taken into account (up to the sixth mode) and thus shear deformation is provoked for the determination of the shear moduli with bending vibrations. In this document an equation, derived from Timoshenko´s equation, taking into account for shear, is applied for the calculation of the moduli.

With the resonant beam method specified in this document, the elastic moduli are determined in principle for one test specimen. One Young´s modulus and two shear moduli are determined in directions perpendicular to each other. The Young´s modulus is the longitudinal modulus in the length direction of the prismatic test specimen, while the shear moduli are the moduli along the width and thickness of the test specimen (Poisson ratios can be calculated from these moduli). Thus it is important to observe in which direction, with respect to the crystallographic symmetry, the prismatic test specimen is cut from the composite.

For example, from a 2,5 D- or a 3 D-composite (orthothropic symmetry) test specimens can be cut out perpendicular to each other in reinforcing directions 1, 2 and 3, so that the Young´s moduli $E_{11}$, $E_{22}$, $E_{33}$ and the shear moduli $G_{12}$, $G_{23}$, $G_{13}$ can be determined.

For a plate of a 2D-composite (quadratic or tetragonal symmetry), with a test specimen cut out in the direction of the fibre reinforcement, direction 1, the technique specified in this document generates the longitudinal modulus $E_{11}$, the interlaminar shear modulus $G_{13}$ and the intralaminar shear modulus $G_{12}$. Additionally for such a plate the modulus $E_{22}$ could be determined from a specimen cut out in direction 2, but neither $E_{33}$ in the direction of the thickness of the plate, nor the shear modulus $G_{23}$.

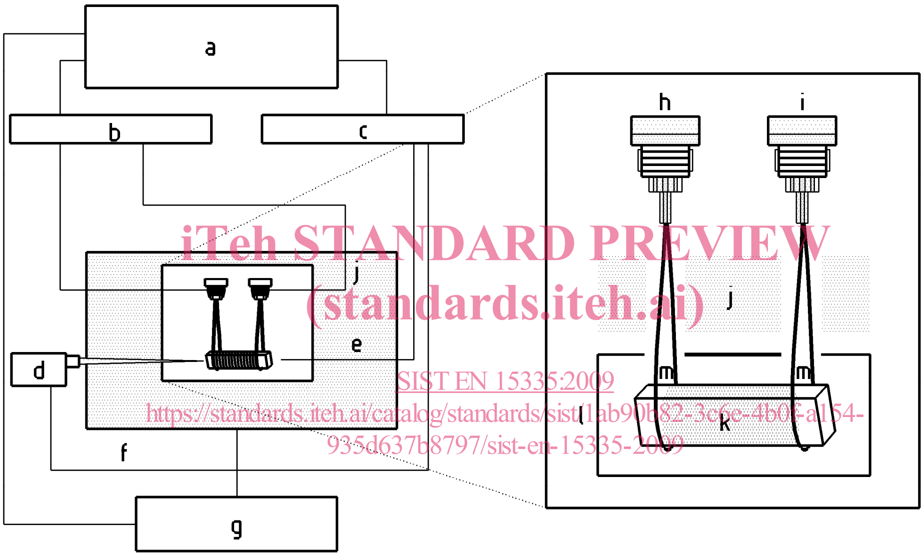
Contrary to mechanical test methods, the determination of elastic properties by the resonant beam method specified here is not based on the evaluation of the stress-strain response over a given deformation range obtained under quasi static loading conditions, but is based on a non-destructive dynamic measurement from vibrations at very small amplitudes. Therefore the values of Young´s modulus, shear modulus and Poisson ratio determined by the two types of methods just mentioned may not be comparable, particularly for ceramic matrix composites, which may exhibit non linear stress-strain behaviour.

# 6 Apparatus

## 6.1 Vacuum vessel

Inside a vacuum chamber the prismatic test specimen is suspended by loops of carbon fibre bundles (a few hundred fibres each) and excited to bending vibrations via one of these loops. The test specimen hangs into a heating element (optionally this should be partly removable when adjusting the test specimen at room temperature). The characteristic spectrum of bending vibrations is registered via the other loop at the temperatures of interest.

See an example of a vacuum vessel illustrated in Figure 1.



**Key**

| | |
|---|---|
| a | control and evaluation unit |
| b | network analyser |
| c | temperature control |
| d | pyrometer |
| e | thermocouple |
| f | vacuum vessel |
| g | pumping unit |
| h | transducer in a water-cooled housing: the Transmitter |
| i | transducer in a water-cooled housing: the Receiver |
| j | insulating felt |
| k | prismatic test specimen |
| l | heating element |
| m | carbon fibre loops |

NOTE The prismatic test specimen is excited to bending vibrations and the frequency spectrum is transmitted by carbon fibre-bundle loops attached to transducers in water-cooled housings (right), [1], [2].

**Figure 1 — Schematic of the resonant beam method apparatus**

## 6.2 Transducers

The transducers shall introduce sinusoidal vibrations of a certain range of frequencies (generated by a frequency synthesiser) to one end of the test specimen and collect the resonant spectrum of the test specimen on the other end. The transducers shall have a flat response curve (i.e. no resonances of its own). Both, sinusoidal vibrations and flat response are necessary to perform the method.

Piezo-ceramics (piezoelectric effect), with a range of operation 1 kHz to 200 kHz and a signal to noise ratio 20 dB, should be used.

## 6.3 Carbon fibre-bundle loops

Loops of carbon fibre-bundles with a few hundred fibres in the bundle to transmit and receive introduce and accept the spectrum of vibrations. The ends of each of the bundles are fixed by a polymeric glue into a short tube (the tube should be made of aluminium, outer diameter 3, inner diameter 1,5 mm, ending in a metric thread, M 2,5 should be used). The tubes with the glued in bundles are screwed into a cylindrical foot, which is directly glued to the transducers (piezo-ceramics). The transducers are enclosed in water cooled housing, the cylindrical foot with the screwed in tubes and the glued in bundles are protruding into the vacuum vessel. The carbon fibre-bundles shall be as long as to centre the prismatic test specimen into the hot zone of the heating element.

## 6.4 Pumping unit

To prevent perturbation and because of the presence of carbon or carbon/carbon heating elements, carbon fibre insulating felts and the carbon fibre-bundle loops for the suspension of the test specimen, a vacuum of 3 x $10^{-2}$ Pa or inert atmosphere (Argon) is required. A suitable pumping unit and a unit for refilling with inert gas should be installed.

## 6.5 Control and evaluation units

### 6.5.1 Frequency sweeping unit

A frequency sweeping unit to introduce the sinusoidal vibrations, combined to a linearly amplified recording unit to accept and store the resonant frequency spectrum (e.g. a network analyser), should be used.

### 6.5.2 Temperature control unit

For temperature measurement, either thermocouples conforming to EN 60584-1 or EN 60584-2 shall be used, or where thermocouples not conforming to EN 60584-1 or EN 60584-2 pyrometers are used, they shall be appropriately calibrated and the calibration data added to the test report.

NOTE 1 Type B thermocouples should be used.

NOTE 2 Vacuum combined with graphite heating elements and carbon fibre insulating felts may degrade the thermocouples, thus the calibration of the thermocouples should be controlled after every temperature cycle beyond 1 200 °C.

### 6.5.3 Personal computer

A personal computer for data acquisition, controlling and evaluation of results. The personal computer does not need any specific requirement.

## 6.6 Balance

A laboratory balance capable of weighing the test specimen to the nearest 1 mg.

### 6.7 Micrometer

A micrometer in accordance with ISO 3611 for measuring the dimensions of the test specimen to the nearest 0,01 mm.

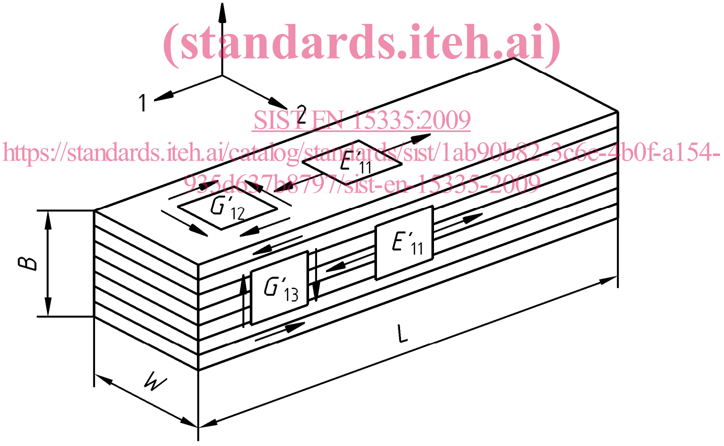
### 6.8 Callipers

Vernier callipers in accordance with ISO 3599 for measuring the length of the test specimen to the nearest 0,05 mm.

## 7 Test specimens

### 7.1 General

This test method measures Young's modulus parallel to the length $L$ of the prismatic test specimen and two shear moduli perpendicular to each other, by vibration along the width $W$ (edgewise vibration) and the thickness $B$ (flatwise vibration) of the test specimen, respectively. The test composite is likely to be elastically anisotropic. The prismatic test specimen should be cut out of the composite with respect to its crystallographic symmetry. That means the direction of the main reinforcement of the composite should be parallel to the length $L$ of the prismatic test specimen. This orientation of the individual beams along the symmetry axes is an important factor, which helps to decrease the number of fitting variables in the two-step evaluation.



**Key**

| | |
|---|---|
| 1, 2, 3 | axes of the Cartesian coordinate system for the specimen's notation |
| $L$ | length |
| $W$ | width |
| $B$ | thickness |
| $E'_{11}$ | longitudinal modulus |
| $G'_{13}$ | interlaminar shear modulus from flatwise vibration |
| $G'_{12}$ | intralaminar shear modulus from edgewise vibration, [1] |

NOTE 1 The horizontal lines indicate the plies of the composite.

NOTE 2 Flatwise vibration means transverse vibration of the specimen in the plane 1,3 (along the thickness $B$), edgewise vibration means transverse vibration in the plane 1,2 (along the width $W$).

**Figure 2 — Schematic representation of a test specimen, cut out from a plate of a 2D fibre reinforced ceramic matrix composite**

### 7.2 Shape and dimensions

The test specimen should be prismatic, long and thin, i. e. a rectangular prism with the ratio of length $L$ to width $W$ and length to thickness $B$ between 10 and 15 (see Figure 2). If possible, the test specimens should be cut out of the composite in the directions of the main axes of elastic symmetry of the composite. The dimensions should be chosen so that the volume of the test specimen is larger than the RVE (Representative Volume Element) of the composite. The dimensions of the test specimen should be such as to have a fundamental flexural resonant frequency in the range 1 kHz to 20 kHz. The dimensions for 2D carbon/carbon composites should be $80 \times 7 \times 5\ \text{mm}^3$.

### 7.3 Plane parallelism

The parallelism of the upper and lower surfaces perpendicular to the direction of flexural vibration should be equal or smaller than $W/100$ and $B/100$, respectively along the length of the test specimen. The parallelism of the ends of the test specimen should be equal or smaller than $L/200$.

### 7.4 Number of test specimens

The number of test specimens necessary to be cut out of the anisotropic composite to gain the elastic moduli depends on the crystallographic symmetry of the composite. Generally it is to mention that the test specimens shall be cut out of the composite in the directions of the main axes of elastic symmetry of the composite:

For unidirectionally (UD) -reinforced composites (hexagonal or transversely isotropic symmetry) two specimens are necessary. One specimen parallel to the direction of fibre reinforcement and a second specimen perpendicular to the reinforcement have to be cut out. With these two specimens the complete set of five elastic moduli can be achieved (an example for such a measurement is given in Annex A).

For bidirectionally (2D) -reinforced composites (quadratic or tetragonal symmetry), e.g. plates of composites reinforced by woven fabrics, also two specimens are necessary, but the complete set of six elastic moduli cannot be achieved. For a plate of a 2D-composite, with a test specimen cut out in the direction of the fibre reinforcement, direction 1, the technique specified in this document generates the longitudinal modulus $E_{11}$, the interlaminar shear modulus $G_{13}$ and the intralaminar shear modulus $G_{12}$. Additionally for such a plate the modulus $E_{22}$ could be determined from a specimen cut out in direction 2, perpendicular to direction 1, but neither $E_{33}$ in the direction of the thickness of the plate, nor the shear modulus $G_{23}$ (see Figure 2).

For tridirectionally (3D) –reinforced composites (orthothropic symmetry), e.g. composites reinforced by cubic fabrics or by stitched woven fabrics (2,5D –reinforced), three specimens are necessary. The complete set of nine elastic moduli is not achieved. Cutting out three specimens perpendicular to each other along the Cartesian coordinates 1,2,3, chosen along the main directions of reinforcement, provides 6 elastic moduli ($E_{11}, E_{22}, E_{33}, G_{12}, G_{13}, G_{23}$).

## 8 Test procedure

### 8.1 Test specimen preparation

Weigh a test specimen to the nearest 1 mg. Measure its cross-sectional dimensions at least at three positions along its length to the nearest 0,01 mm with the micrometer (6.7), and take the average. Measure the length to the nearest 0,05 mm with the Vernier callipers (6.8).

### 8.2 Adjustment of the apparatus

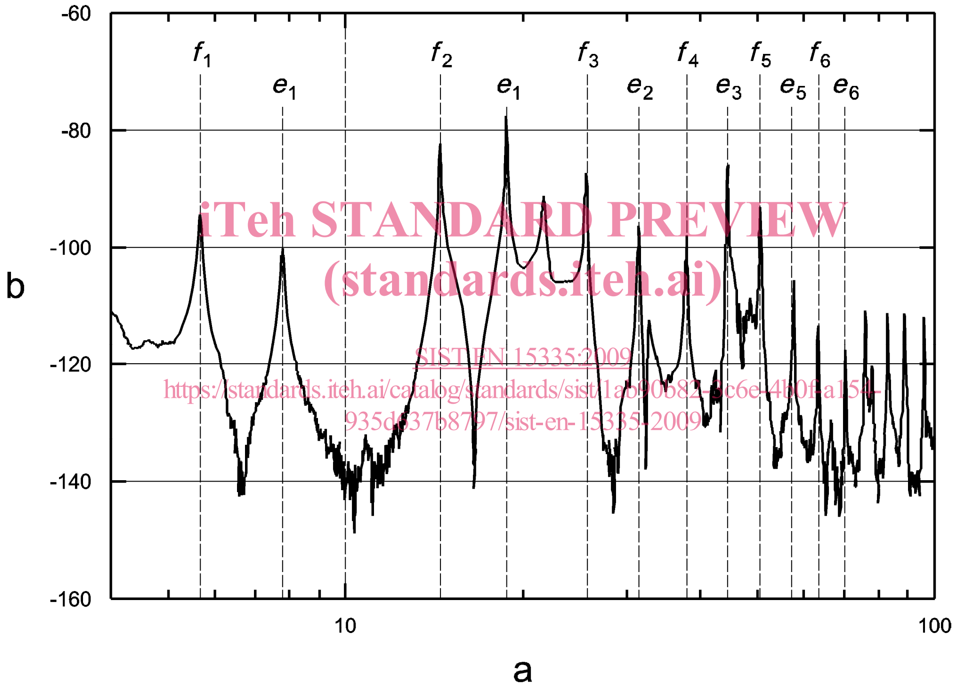
Put the test specimen by hand or using tweezers into the carbon fibre-bundle loops inside the vacuum vessel. The test specimen should hang free in the loops with the loops positioned 1 mm off the ends of the test specimen. One of the edges of the test specimen should face downwards.

Close the vacuum vessel. Remove the air by using the pumping unit to a vacuum of $3 \times 10^{-2}$ Pa.

For the case it is intended to run the test in inert atmosphere: flush with inert gas to a pressure of $5 \times 10^4$ Pa. Repeat this procedure one time, that means, remove the inert gas to a vacuum of $3 \times 10^{-2}$ Pa and flush with inert gas to a pressure of $5 \times 10^4$ Pa again.

## 8.3 Measurement at room temperature

Use the frequency sweeping unit (network analyser) from 6.5.1 and sweep the frequency from 1 kHz to 200 kHz (the range may be smaller depending on the material and the dimensions of the individual samples) and store the frequency spectrum on the recording unit (network analyser). An example for such a spectrum is given in Figure 3 (observe that the abscissa of Figure 3 is given in logarithmic scale). The local maxima denote the resonant sites. The notations $f_i$ and $e_i$ mean flatwise and edgewise transverse vibrations, respectively. The subscript i designs the harmonics (i = 1 for the fundamental mode i.e. the basic vibration, i = 2 for the first harmonic and so on). For the identification of the resonant sites and the calculation of the room temperature results continue with Clause 9. If it is not possible to identify the resonant sites, it is necessary to readjust the test specimen: remove the test specimen from the carbon fibre-bundle loops and put it in again as mentioned in 8.2 and then start again the procedure as described in 8.2 and 8.3.



**Key**

a frequency (kHz)

b signal (dBm)

**Figure 3 — Example for a frequency spectrum (carbon-carbon composite) from 4 to 100 kHz**

## 8.4 Measurement at high temperatures

Heat up the test specimen (a rate of 25 °C per minute should be maintained) and hold the temperature at steps of 200 °C. After the temperature at a specific temperature level stays constant, store the frequency spectrum (as performed in 8.3). The resonant sites already identified at room temperature (see 8.3) should appear again and should be identified at the specific temperature level again.

NOTE The resonant sites may shift with temperature and the shape of the sites may change, it is recommended to try to identify the sites again and to follow the shift at each level of temperature.