SLOVENSKI STANDARD

SIST-TS CEN/TS 820-5:2005

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Sodobna tehniļ na keramika ï Metode za preskuġanje monolitske keramike ï Termomehanske lastnosti ï 5. del: Ugotavljanje elastiļ nih modulov pri poviġanih temperaturah

Advanced technical ceramics - Methods of testing monolithic ceramics. Thermomechanical properties - Part 5: Determination of elastic moduli at elevated temperatures

Hochleistungskeramik - Monolithische Keramik - Thermomechanische Eigenschaften - Teil 5: Bestimmung der elastischen Moduln bei erhöhten Temperaturen

Céramiques techniques avancées - Méthodes d'essai des céramiques monolithiques - Propriétés thermomécaniques - Partie 5: Détermination des modules élastiques a températures élevées

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Advanced technical ceramics - Methods of testing monolithic ceramics. Thermomechanical properties - Part 5: Determination of elastic moduli at elevated temperatures

Céramiques techniques avancées - Céramiques monolithiques - Propriétés thermomécaniques - Partie 5: Détermination du module élastique à température élevée

Hochleistungskeramik - Monolithische Keramik - Thermomechanische Eigenschaften - Teil 5: Bestimmung der elastischen Moduln bei erhöhten Temperaturen

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Foreword

This document (CEN/TS 820-5:2004) has been prepared by Technical Committee CEN/TC 184 “Advanced technical ceramics”, the secretariat of which is held by BSI.

This document has been prepared under a mandate given to CEN by the European Commission and the European Free Trade Association.

EN 820 *Advanced technical ceramics — Methods of testing monolithic ceramics — Thermomechanical properties* comprises five parts:

Part 1: *Determination of flexural strength at elevated temperatures*

Part 2: *Determination of self-loaded deformation*

Part 3: *Determination of resistance to thermal shock by water quenching*

Part 4: *Determination of flexural creep deformation at elevated temperatures*

Part 5: *Determination of elastic moduli at elevated temperatures*

Part 4 is a European Prestandard (ENV) and Part 5 is a Technical Specification (CEN TS).

This document includes a bibliography.

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

1 Scope

This part of EN 820 describes methods for determining the elastic moduli, specifically Young's modulus, shear modulus and Poisson's ratio, of advanced monolithic technical ceramics at temperatures above room temperature. The Technical Specification prescribes three alternative methods for determining some or all of these three parameters:

A - the determination of Young's modulus by static flexure of a thin beam in three- or four-point bending.

B - the determination of Young's modulus by forced longitudinal resonance, or Young's modulus, shear modulus and Poisson's ratio by forced flexural and torsional resonance, of a thin beam.

C - the determination of Young's modulus from the fundamental natural frequency of a struck bar (impulse excitation method).

This Technical Specification extends the above-defined room-temperature methods described in ENV 843-2 to elevated temperatures. All the test methods assume the use of homogeneous test pieces of linear elastic materials. The test assumes that the test piece has isotropic elastic properties. At high porosity levels all of the methods can become inappropriate. The maximum grain size (see EN 623-3), excluding deliberately added whiskers, should be less than 10 % of the minimum dimension of the test piece.

NOTE 1 Method C in ENV 843-2 based on ultrasonic time of flight measurement has not been incorporated into this Technical Specification. Although the method is feasible to apply, it is specialised, and outside the capabilities of most laboratories. There are also severe restrictions on test piece geometries and methods of achieving pulse transmission. For these reasons this method has not been included in CEN/TS 820-5.

NOTE 2 The upper temperature limit for this test depends on the properties of the test pieces, and can be limited by softening within the timescale of the test. In addition, for method A there can be limits defined by the choice of test jig construction materials.

NOTE 3 Methods B and C may not be appropriate for materials with significant levels of porosity (i.e. >15%) which cause damping and an inability to detect resonances or natural frequencies, respectively.

NOTE 4 This method does not provide for the effects of thermal expansion, i.e. the measurements are based on room temperature dimensions. Depending upon the use to which the data are put, it can be necessary to make a further correction by multiplying each dimensional factor in the relevant equations by a factor $(1 + \overline{\alpha}\,\Delta T)$ where $\overline{\alpha}$ is the mean linear expansion coefficient over the temperature interval ΔT from room temperature.

2 Normative references

This Technical Specification incorporates by dated or undated reference, provisions from other publications. These normative references are cited at the appropriate places in the text, and the publications are listed hereafter. For dated references, subsequent amendments to or revisions of any of these publications apply to this Technical Specification only when incorporated in it by amendment or revision. For undated references the latest edition of the publication referred to applies (including amendments).

EN 820-1, *Advanced technical ceramics — Method of testing monolithic ceramics — Thermo-mechanical properties — Part 1: Determination of flexural strength at elevated temperatures*

EN 843-1:1995, *Advanced technical ceramics — Monolithic ceramics — Mechanical properties at room temperature — Part 1: Determination of flexural strength*

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

EN ISO 7500-1, *Metallic materials — Verification of static uniaxial testing machines — Part 1: Tension/compression testing machines — Verification and calibration of th eforce-measuring system (ISO 7500-1:1999)*

EN ISO/IEC 17025, *General requirements for the competence of testing and calibration laboratories (ISO/IEC 17025:1999)*

ISO/R 463, *Dial gauges reading in 0,01 mm, 0,001 in and 0,0001 in*

ISO 3611, *Micrometer callipers for external measurement*

ISO 6906, *Vernier callipers reading to 0,02 mm*

3 Terms and definitions

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

3.1
Young's modulus
stress required in a material to produce unit strain in uniaxial extension or compression

3.2
shear modulus
shear stress required in a material to produce unit angular distortion

3.3
Poisson's ratio
negative value of the ratio of lateral strain to longitudinal strain in an elastic body stressed longitudinally

3.4
static elastic moduli
elastic moduli determined in an isothermal condition by stressing statically or quasistatically

3.5
dynamic elastic moduli
elastic moduli determined non-quasistatically, i.e. under adiabatic conditions, such as in the resonant, ultrasonic pulse or impulse excitation methods

4 Method A: Static bending method

4.1 Principle

Using three- or four-point bending of a thin beam test piece, the elastic distortion is measured, from which Young's modulus may be calculated according to thin beam equations.

4.2 Apparatus

4.2.1 *Test jig*, in accordance with that described in EN 820-1 for flexural strength testing at elevated temperatures in terms of its function, i.e. the support and loading rollers shall be free to roll, and to articulate to ensure axial and even loading as described in EN 843-1. The test jig shall be made of materials which do not interact with the test piece and which remain essentially elastic at the maximum test temperature. A typical arrangement is shown in Figure 1.

NOTE 1 Articulation is not essential for carefully machined flat and parallel-faced test pieces.

The outer span of the test jig shall be 40 mm or greater.

NOTE 2 If the displacement is to be measured by method 1 (see 4.2.5), a span of up to 100 mm, or a span to thickness ratio in excess of 20, is recommended to obtain large displacements and to ensure that the compliance of the machine is a small correction if displacement is recorded as a machine cross-head movement.

The test jig may be either for three-point or four-point flexure. The latter method is required if displacement is determined by differential transducer.

4.2.2 *Mechanical testing machine*, capable of applying a force to the test jig at a constant displacement rate. The test machine shall be equipped for recording the load applied to the test jig at any point in time. The accuracy of the test machine shall be in accordance with EN ISO 7500-1, Grade 1 (1% of indicated load), and shall be capable of recording to a sensitivity of better than 0,1% of the maximum load employed.

4.2.3 *Thermal enclosure and control system*, surrounding the test piece, capable of achieving the maximum desired temperature and maintaining it to ± 2 ^{o}C for test temperatures up to 1000 ^{o}C, and ± 4 ^{o}C at higher temperatures.

NOTE The system can operate with an air or inert atmosphere, or with a vacuum inside the thermal enclosure. Especially with regard to use in vacuum, efforts should be made to ensure that the force applied at the test piece is correctly recorded by the load cell outside the enclosure, taking account of friction or elastic resistances in seals or bellows systems.

4.2.4 *Thermocouple*, conforming to EN 60584-2 for measuring the test piece temperature. The thermocouple shall be in close proximity to but shall not touch the test piece.

4.2.5 *Displacement measuring device*, for recording the displacement of the loaded test piece by one of two methods:

Method 1. Recording the apparent displacements of the test machine as the test piece is loaded in the test jig, and again with the test piece replaced by a ceramic bar at least 15 mm thick with flat and parallel faces to within 0,05 mm. The difference between these displacements is equivalent to the displacement of the test piece in the test jig. The displacement recording device (chart recorder, digital indicator, etc.) shall be calibrated by comparing machine cross-head displacement with the movement indicated on a dial gauge contacting the cross-head. The dial gauge shall be in accordance with ISO/R 463, or other certified device accurate to 0,01 mm.

The parts of the load train subjected to elevated temperatures shall be made of materials which remain elastic at the maximum test temperature.

Method 2. Recording the displacement of the test piece directly using a transducer extensometer contacting at least two defined points on the surface of the test piece between the support loading rollers in three-point or four-point bending. The defined points shall preferably be:

for four-point bending: the centre of the span and one or both loading rollers (see, for example, Figure 1, right);

for three-point bending: the centre of the span and one or both support rollers (see, for example, Figure 1, left).

NOTE The equations given in 4.5 assume these preferred positions. Other displacement detection positions require alternative formulations.

The transducer shall be capable of detecting movements with an accuracy of 0,001 mm, shall have output linear to 1 % over the expected displacement range in making this test and its sensitivity shall be calibrated to an accuracy of 0,1 %.

The extensometer parts subjected to elevated temperatures shall remain elastic to the maximum test temperature, and their tips shall not interact with the test piece (see also EN 820-1).

4.2.6 *Micrometer*, in accordance with ISO 3611, capable of recording to 0,01 mm.

4.2.7 *Dial gauge*, in accordance with ISO/R 463 or other suitable calibrated displacement measuring device, capable of recording to 0,01 mm.

4.3 Test pieces

Test pieces shall be rectangular section bars selected and prepared by agreement between parties. They may be directly prepared close to final dimensions or machined from larger blocks. This test measures Young's modulus parallel to the length of the test piece. If the test material is likely to be elastically anisotropic, care shall be taken in selection of the test piece orientation and in the interpretation of the test results.

The length of the test pieces shall be at least 10 mm longer than the test-jig span. The width of the test piece shall be in the range 4 mm to 10 mm. For method 1, the thickness of the test piece shall be in the range 0,8 mm to 1,5 mm. For method 2, the test piece may be up to 3 mm thick, but preferably should be in the range 1 mm to 2 mm thick. The test pieces shall be machined to final dimensions. They shall be flat and parallel-faced to better than ± 0,5 % of thickness on the faces to be placed on the loading rollers of the test jig. They shall similarly be machined flat and parallel-faced to better than ± 0,5 % of width on the side faces. For method 1 they shall not be chamfered. For method 2 they may be chamfered as specified in EN 843-1.

At least three test pieces shall be prepared.

4.4 Procedure

Measure the width and thickness of the test pieces at several places and record the average values.

Insert a test piece in the test-jig and centralise it in accordance with the requirements of EN 843-1. Select a maximum force to be applied to the test piece which will avoid fracture.

NOTE 1 The upper level of force can be estimated by employing the strength calculation in EN 843-1:1995, clause 8, and inserting a stress level of no more than 0,5 σ_f, where σ_f is the mean fracture stress expected at the test temperature.

Heat the thermal enclosure to the required test temperature and allow the temperature to stabilize such that the thermocouple recording test piece temperature varies by no more than 2 °C in a 15 minute period up to 1000 °C, and by no more than 4 °C at higher temperatures.

Apply a steadily increasing force to the test jig at a constant test machine cross-head displacement rate in the range 0,001 mm/min to 0,5 mm/min. Record the load and displacement (either cross-head displacement, or transducer displacement output) continuously. When the maximum selected force is achieved, reverse the direction of the machine and reduce the load to zero. Repeat the cycle at least twice more to the same peak load, or until repeatable results are obtained. Repeat the test on each test piece. If the machine displacement is to be employed (method 1) or if the transducer method is employed using a support roller as one of the defined points (method 2), replace the test piece with the thick parallel-sided steel (for use to 300 °C) or ceramic bar and repeat the loading cycles to the same peak load, recording load and displacement.

NOTE 2 The use of both loading and unloading cycles is required in order to take into account machine hysteresis in method 1, transducer hysteresis in method 2.

4.5 Calculation of results

4.5.1 From cross-head displacement (Method 1)

Inspect the recordings of load and displacement for the test piece and the thick steel or ceramic bar for uniformity and linearity. Select a region of the recordings from a minimum load of not less than 10 % of peak load or 0,2 N, whichever is the greater, to a maximum load of not more than 90 % of the peak load applied. The same load range shall be selected for each loading cycle on the test piece and the thick bar.

NOTE 1 The region of the recordings selected should avoid strong non-linearities at low load which may include irreproducible effects of machine movement and test piece alignment, and also the effects of cross-head reversal near peak load.

NOTE 2 If the force - displacement traces show evidence of a reduction of stiffness at upper load levels, this can be taken as evidence of plastic softening. At this temperature and any higher test temperature, the results of the test should be deemed invalid.

Calculate or measure the displacement recorded over the selected load range for each loading and unloading cycle for the test piece and for the thick bar. Calculate the average displacement in each direction. If the displacement of the first cycle is more than 2 % different from that of the second or subsequent cycle, ignore the first cycle when computing the average.

NOTE 3 The first cycle can show a different response to subsequent cycles as the test piece beds down into the test jig and the machine movement stabilizes.

Calculate Young's modulus according to the following formulae:

For displacement of loading points in three-point bending:

$$E = \frac{(P_2 - P_1) l^3}{4bh^3 (d_c - d_s)} \tag{1}$$

For displacement of loading points in four-point bending:

$$E = \frac{2(P_2 - P_1) d_1^2 (d_1 + 3d_2)}{bh^3 (d_c - d_s)} \tag{2}$$

where:

E = Young's modulus expressed as Newtons per square metre;

P_1 = Lower load level selected from recordings, expressed in Newtons;

P_2 = upper load level selected from recordings, expressed in Newtons;

l = test jig outer span, expressed in metres;

d_1 = test jig inner roller to outer roller spacing in four-point bending, expressed in metres;

d_2 = one half of the test jig inner span in four-point bending, expressed in metres;

b = test piece width, expressed in metres;

h = test piece thickness, expressed in metres;

d_c = displacement recorded for the test piece in the jig over load interval P_1 to P_2, expressed in metres;

d_s = displacement recorded for the thick bar in the jig over load interval P_1 to P_2, expressed in metres.

NOTE 4 For the case of quarter-point bending, $d_1 = d_2$, and equation 2 reduces to:

$$E = \frac{(P_2 - P_1) l^3}{8bh^3 (d_c - d_s)} \quad (2A)$$

Calculate the average Young's modulus figures for the loading and unloading curves. If these values differ by more than 2 %, repeat the tests. If they differ by less than 2 %, take the overall average as the determined value from the test.

4.5.2 From transducer displacement measurements (Method 2)

Use the procedure defined in 4.5.1 to obtain displacements for a defined load range. If one of the defined points for the transducer contact in three-point bending is the support roller, calculate the displacement recorded for the thick bar. Subtract the mean value of the thick bar displacement from the mean specimen displacement over the same load range for both loading and unloading.

NOTE 1 If the force - displacement traces show evidence of a reduction of stiffness at upper load levels, this can be taken as evidence of plastic softening. At this temperature and any higher test temperature, the results of the test should be deemed invalid.

For three-point bending using defined points at the span centre and one or both support rollers, calculate Young's modulus using equation 1.

For four-point bending using defined points at the span centre and one or both loading rollers, calculate Young's modulus from the formula:

$$E = \frac{3(P_2 - P_1) d_1 d_2^2}{bh^3 d_t} \quad (3)$$

where:

d_t = transducer displacement recorded between the test piece centre and the inner loading point in four-point bending over the selected load range, expressed in metres.

NOTE 2 For the case of quarter-point bending, $d_1 = d_2$, and equation 3 reduces to:

$$E = \frac{3(P_2 - P_1) l^3}{64bh^3 d_t} \quad (3A)$$

Calculate the average Young's modulus figures for the loading and unloading parts of the cycles. If these values differ by more than 2 %, repeat the tests. If they differ by less than 2 %, take the overall average as the determined value from the test.

4.6 Accuracy and interferences

A simple analysis based on the flexure equation can be used to show that the principal sources of error are in measuring the force range ($P_2 - P_1$), the thickness of the test piece b, and the deflection d_t or (d_c - d_s). In contrast, measurement of temperature, test span and test piece width play only a minor role. The following analysis is based on the conditions specified for this method, but the overall error is dependent on the choice of equipment, the accuracy of calibration, and the repeatability of mechanical contact between the system and the test piece. Assuming that the accuracy of recording the force is limited to ± 1 % by the calibration accuracy of the load cell in the test machine, the noise in the output, and any