



SLOVENSKI STANDARD
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Advanced technical ceramics - Thermomechanical properties of monolithic ceramics - Part 4: Determination of flexural creep deformation at elevated temperatures

Hochleistungskeramik - Thermomechanische Eigenschaften monolithischer Keramik - Teil 4: Bestimmung der Kriechverformung unter Biegebeanspruchung bei erhöhten Temperaturen

Céramiques techniques avancées - Propriétés thermomécaniques des céramiques monolithiques - Partie 4: Détermination de la déformation par fluage en flexion à températures élevées

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ICS:

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EUROPEAN STANDARD
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**Advanced technical ceramics - Thermomechanical properties of
monolithic ceramics - Part 4: Determination of flexural creep
deformation at elevated temperatures**

Céramiques techniques avancées - Propriétés
thermomécaniques des céramiques monolithiques - Partie
4 : Détermination de la déformation par fluage en flexion à
températures élevées

Hochleistungskeramik - Thermomechanische
Eigenschaften monolithischer Keramik - Teil 4:
Bestimmung der Kriechverformung unter
Biegebeanspruchung bei erhöhten Temperaturen

This European Standard was approved by CEN on 12 June 2009.

CEN members are bound to comply with the CEN/CENELEC Internal Regulations which stipulate the conditions for giving this European Standard the status of a national standard without any alteration. Up-to-date lists and bibliographical references concerning such national standards may be obtained on application to the CEN Management Centre or to any CEN member.

This European Standard exists in three official versions (English, French, German). A version in any other language made by translation under the responsibility of a CEN member into its own language and notified to the CEN Management Centre has the same status as the official versions.

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Foreword

This document (EN 820-4:2009) 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of an identical text or by endorsement, at the latest by January 2010, and conflicting national standards shall be withdrawn at the latest by January 2010.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. CEN [and/or CENELEC] shall not be held responsible for identifying any or all such patent rights.

This document supersedes ENV 840-4:2001.

EN 820 consists of five parts, under the general title "Advanced technical ceramics - Methods of testing monolithic ceramics – Thermomechanical properties":

- 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

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 the United Kingdom.

EN 820-4:2009 (E)**1 Scope**

This Part of EN 820 describes a procedure for undertaking flexural creep tests at elevated temperatures on advanced technical ceramics, mainly for the purposes of comparison of deformation behaviour of materials under stressed conditions and under any appropriate atmospheric condition.

NOTE This European Standard does not provide a method of acquiring engineering performance data since the stress distribution under flexural loading is indeterminate.

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 820-1, *Advanced technical ceramics – Methods of testing monolithic ceramics - Thermomechanical properties - Part 1: Determination of flexural strength at elevated temperatures*

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

EN 1006, *Advanced technical ceramics - Monolithic ceramics - Guidance on the selection of test pieces for the evaluation of properties*

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

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

EN ISO 7500-1, *Metallic materials - Verification of static uniaxial testing machines - Part 1: Tension/compression testing machines - Verification and calibration of the force-measuring system (ISO 7500-1:2004)*

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

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**
creep
time-dependent non-elastic deformation of a material under an applied stress
- 3.2**
creep rupture
failure of a test piece under nominally constant loading conditions resulting from an accumulation of microstructural damage
- 3.3**
stress rupture
catastrophic extension of a flaw having previously grown subcritically under constant nominal stress leading to failure of the test piece

3.4

subcritical crack growth

extension of existing cracks or flaws under stress which does not produce instant failure

4 Significance and use

The test is intended to evaluate the deformation of a test piece under nominally constant force as a function of time at elevated temperatures. In particular it can be used for materials comparison, or for determining the temperature at which creep deformation becomes significant for a prospective engineering use.

During the course of such a test, the test piece can fracture. This can be due either to a subcritical crack growth process unrelated to the mechanism of creep (stress rupture), or to the accumulation of creep damage leading to acceleration of creep rate and the linking of damage to form cracks (creep rupture). In some circumstances it is not possible to distinguish the mechanism of failure. In either case, the test piece lifetime under the imposed temperature and stress conditions can be an important aspect of a material's performance.

The analysis given in this European Standard (see 8.6) produces purely nominal data, assuming that the actual maximum nominal stress in the test piece is linearly proportional to the test force applied and is constant during the test. Moreover, an additional assumption of linear dependence of strain on stress is made for some deflection measurement methods. Furthermore, it does not give engineering creep data equivalent to separate pure tensile or compressive conditions. In many cases, the creep rate dependence is to the maximum stress, and can differ in tension and compression. Typically, the true maximum stress in the test piece is less than that calculated using Equation 1 because of faster relaxation at higher stress levels, and the true surface strain rate can be greater than a linear prediction in certain geometrical arrangements for determining the deformation, particularly if this is done using the relative displacement of the loading system. The Bibliography contains references to more detailed theoretical analyses of flexural creep accounting for such non-linearities.

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5 Principle

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The method involves supporting a bar test piece on two supports near its ends, heating it to the required elevated temperature which is maintained constant, applying a force to two loading points spaced symmetrically between the support points, and recording the deflection of the test bar with time.

The deflection of the test piece is measured indirectly and continuously or at appropriate time intervals during the test using the displacement of the loading system (see e.g. Figure 1a), or by using contacting extensometer rods at given positions on the test piece (see e.g. Figures 1b to 1e). The indirect measurement of deflection (Figure 1a) is converted into a nominal maximum surface strain in the test piece assuming a linear relationship between stress and accumulated strain. Similar assumptions are involved in analysing deflections between support points and the span centre (Figure 1b and 1c). When employing displacement measurement between the loading points and the span centre (Figures 1d and 1e), the analysis assumes uniform curvature of the test piece, a linear relationship between strain and distance from the neutral axis, and equal behaviour in tension and compression. The slope of the strain/time curve can be converted to a creep strain rate.

6 Apparatus

6.1 Creep test loading jig

The test jig is essentially a four-point bend flexural test jig similar to that described in EN 820-1 for flexural strength testing at elevated temperatures. It comprises a pair of parallel 5 mm diameter support rods positioned 40 mm apart on a refractory supporting structure. These rods shall be free to roll to eliminate friction effects. In contrast to the articulating requirement in EN 820-1, articulation is not required provided that the rods are accurately parallel in the horizontal plane to within 0,001 mm per mm length of rod.

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The loading assembly comprises a similar pair of freely rolling rods positioned on a loading block. The spacing between these rods shall be between 30 % and 50 % of the spacing of the support rods. The loading block shall be free to articulate relative to the loading column in order to permit alignment of the loading rods on the test piece upper surface.

NOTE 1 Subject to agreement between parties, other test piece support and loading spans can be employed. This can be particularly advantageous for creep-resistant materials. In addition, in some conditions it is recognized that freely rolling rollers, although preferred, may not be feasible. Such deviations from this method should be reported. The effect of restricted roller rotation may or may not be significant depending on the test material and the testing conditions. There is some evidence to suggest that the surface of glass-phase containing materials, or materials which oxidize to give a viscous glassy surface layer, can have a low coefficient of friction against the roller material at the test temperature, such that over the period of the test any friction becomes negligible. However, this situation cannot always be guaranteed.

The loading block shall be guided appropriately such that the loading rods are positioned mid-way between the support rods, thus centrally loading a test piece when placed on the support rods.

The parts of the loading jig shall be constructed from a ceramic material which is anticipated to be more resistant to deformation than the materials under test. In addition the support and loading rods shall be of a material which does not chemically react with the test piece.

NOTE 2 Suitable materials include high-purity alumina for use with most oxide-based test pieces, or sintered silicon carbide for most non-oxide ceramics.

NOTE 3 Test jig parts manufactured from sintered silicon carbide or other silicon-based non-oxide ceramics develop oxidation films in a short period of time when exposed to temperatures typically above 1 300 °C in an oxygen-containing atmosphere. This can cause prevention of rolling of rollers and impairment of jig function.

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6.2 Heating device

A heating device surrounds the loading jig in such a manner as permit access to the jig for the purposes of mounting and demounting test pieces. The heating device shall be capable of maintaining a constant test piece temperature to ± 3 °C over the duration of the test.

The temperature of the test piece shall be recorded using a thermocouple manufactured in accordance with EN 60584-2 allowing the use of reference tables in EN 60584-1 or, alternatively calibrated in a manner traceable to the International temperature scale ITS-90. The tip of the thermocouple shall be close to but not touching the test piece. It shall previously have been determined that the temperature of the test piece does not vary by more than ± 3 °C over its length when temperature has stabilized for more than 30 min.

The heating device can incorporate or be incorporated within a vacuum or other appropriate chamber for control of gas atmosphere if appropriate to the determination.

6.3 Loading device

The load shall be applied through a loading column to which the upper part of the jig is connected. The load can be generated by any appropriate means provided that a force constant to within ± 1 % can be generated at the test piece.

The force applied to the test piece shall be calibrated to an accuracy of 1 % in accordance with EN ISO 7500-1.

NOTE The mass of the loading system between the load cell and the test piece can be significant and should be taken into account, including any spring force due to the use of bellows systems for protective atmospheres, and any pressure differential between the external atmosphere and inside the test chamber.

6.4 Deflection measuring device

The flexural deflection of the test piece is monitored most effectively by determining the relative vertical displacement of two defined positions on the free under-surface of the deforming test piece (see Figures 1d and 1e).

The displacement is detected by use of extensometer rods passing into the loading jig and freely contacting the test piece surface, using a linear displacement transducer or similar device. The output of the displacement transducer shall have a sensitivity of better than 0,5 μm , shall have linear output with displacement to better than 1 % of the range over which it is used, and shall be calibrated to an accuracy of better than 1 % of range. The contacting force between the test piece and the extensometer rods shall be less than 1 % of the force applied to the test piece.

NOTE 1 The above method is preferred. Alternatively, the test piece displacement can be determined by recording the relative displacement of the support and loading parts of the load train provided that the deflection of the load train at the temperature of operation is less than 5 % of that of the test piece. Typical methods are shown in Figure 1a to 1c. The load train deflection can be determined by using a thick non-deforming test piece of a material such as sintered silicon carbide. The disadvantages of this method are that it does not account for time-dependent flattening of rollers and that linearity of stress and strain is assumed.

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