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Advanced technical ceramics - Methods of test for monolithic ceramics - Thermo-mechanical properties - Part 2: Determination of self-loaded deformation

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Hochleistungskeramik - Prüfverfahren für monolithische Keramik - Thermomechanische Eigenschaften - Teil 2: Bestimmung der Verformung unter Eigengewicht

Céramiques techniques avancées - Méthodes d'essai pour céramiques monolithiques - Propriétés thermo-mécaniques - Partie 2: Détermination de la déformation sous son propre poids

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Advanced technical ceramics - Methods of testing monolithic ceramics - Thermo-mechanical properties - Part 2: Determination of self-loaded deformation

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This European Standard was approved by CEN on 2 January 2003.

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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 Management Centre has the same status as the official versions.

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EUROPEAN COMMITTEE FOR STANDARDIZATION
COMITÉ EUROPÉEN DE NORMALISATION
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Contents

Foreword

This document (EN 820-2:2003) has been prepared by Technical Committee CEN/TC 184 "Advanced technical ceramics", the secretariat of which is held by BSI.

This document shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by October 2003, and conflicting national standards shall be withdrawn at the latest by October 2003.

This document supersedes ENV 820-2:1992.

EN 820 *Advanced technical ceramics – Methods of testing monolithic ceramics – Thermo-mechanical properties* consists of four parts:

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

— Part 2: *Determination of self-loaded deformation*

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

— Part 4: *Determination of flexural creep deformation 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, Czech Republic, Denmark, Finland, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Luxembourg, Malta, Netherlands, Norway, Portugal, Slovakia, Spain, Sweden, Switzerland and the United Kingdom.

1 Scope

This European Standard specifies a method for the determination of the temperature at which the self-loaded deformation of a ceramic test piece commences and the extent of this deformation.

2 Normative references

This European Standard 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 European Standard only when incorporated in it by amendment or revision. For undated references the latest edition of the publication referred to applies (including amendments).

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

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

ENV 1006, *Advanced technical ceramics - Methods of testing monolithic ceramics - Guidance on the sampling and selection of test pieces.*

3 Term and definition

For the purposes of this European Standard the following term and definition applies.

3.1
self-loaded deformation
the property induced by heat, which results in a ceramic body being readily deformed under its own weight

4 Principle

The method involves determining whether or not test bars supported only at their ends deform at a series of temperatures, recording any self-loaded deformation observed, and recording the results graphically. The temperature of commencement of self-loaded deformation, taken from the graph, is reported.

This test is intended as a simple method of determining the upper use temperature of ceramic components in a self-loaded condition. The criterion is an upper limit of flexural distortion. The test is suitable for use on furnace construction materials and on any ceramic materials, from which components are subjected to further processing at high temperatures.

5 Apparatus

5.1 Measurement apparatus, of the type shown in Figure 1. The distance between the knife edge supports shall be 160 mm ± 0,5 mm. The gauge for measuring deformation (sag or bow) shall be accurate to 0,01 mm.

NOTE The dial gauge shown in Figure 1 is an example only.

5.2 Steel bar, of dimensions as shown in Figure 2, with the top surface flat to ± 0,01 mm and parallel, to ± 0,01 mm, to the under surfaces in the regions of reduced thicknesses.

5.3 Laboratory furnace, capable of achieving 1600 °C, and of holding the temperature uniformly over the area occupied by the test pieces (see Figure 3) to within ± 5 °C. A controlled atmosphere, other than air, may be used if required. This shall be reported (see clause 10).

5.4 Two refractory supports, with square cross-section of approximately 12 mm (see Figure 3), made of material which is compatible with the test material at the test temperature. The supports shall be inspected after each firing and where there is evidence of reaction with the test pieces, the supports shall be replaced.

5.5 Thermocouples (two), one to control the furnace and one to measure the temperature of the test pieces. The thermocouples shall be either type R, type S or type B in accordance with EN 60584-1.

6 Test pieces

Materials for testing shall be selected in accordance with the guidance given in ENV 1006. Test pieces shall be rectangular bars, within the following range of dimensions:

a) length: 173 mm to 177 mm;

b) width: 11,5 mm to 12,5 mm;

c) thickness: 3,9 mm to 4,1 mm.

The test pieces shall be ground flat and parallel so that the thickness tolerance of a set of test pieces shall be ± 0,02 mm.

NOTE 1 A set is the number of test pieces which are required for the determination of the temperature of commencement of self-loaded deformation.

Two pieces are required for each determination at a single temperature. Test pieces may be either cut from fired ceramic products or may be prepared especially for this method of test.

NOTE 2 In the latter case they should be fired before testing in a manner comparable to production firing.

7 Procedure

1.1 Setting up of measurement gauge

Check that the measurement gauge (see 5.1) probe is positioned at the mid-point between the two knife edges, as shown in Figure 1. Place the steel bar (see 5.2) on the knife edges and adjust the gauge to read zero.

7.2 Marking of the test pieces

Mark the upper face of one end of the test piece with refractory paint, as the reference end. Draw a pencil line 8 mm from this reference end, across the top surface of the test piece, to act as a reference marker to be aligned with the knife edge of the reference gauge (see 7.3). Repeat the above procedure for the second test piece.

7.3 Measurement of test pieces before heat treatment

Place the test bar in the gauge, reference face uppermost so that the line across the width of the top surface (see 7.2) lies over a knife edge. Record the initial gauge reading (M_1) to the nearest 0,01 mm and whether it is positive or negative. Repeat for the second test piece to be included in the heat treatment (see 7.4).

7.4 Heat treatment of test pieces

Support the two test pieces, approximately 6 mm apart, on the two square cross-section refractory supports (see 5.4), which are positioned 160 mm ± 0,5 mm apart on a refractory batt (see Figure 3). Place the test pieces so that the pencil lines marked across the width of the test pieces are facing upwards and lie over the inside edge of one of the square cross-section supports (see Figure 3). Place the refractory batt with the test pieces in the furnace and position the measuring thermocouple (see 5.5) adjacent to the test pieces. Raise the temperature of the test pieces in the furnace by 300 °C/h to the test temperature without exceeding it, and then maintain that temperature for 1 h. Cool the furnace to room temperature and remove the test pieces.

NOTE The final test temperature should be agreed between the interested parties before the test (see clause 8).

7.5 Measurement of the test pieces after heat treatment

Remeasure the test pieces in accordance with the procedure given in 7.2 and 7.3 to obtain the final gauge reading (M_2).

7.6 Calculation of deformation

The self-loaded deformation, sag or bow, for each temperature tested is calculated from the following expression:

Deformation = M_1 - M_2

where

M_1 is the initial dial gauge reading (see 7.3);

M_2 is the final dial gauge reading (see 7.5).

The deformation shall be expressed in millimetres and the numerical mean of the deformation of both test bars subjected to the same heat treatment temperature shall be determined.

Report the deformation to the nearest 0,01 mm and the test temperatures to the nearest 5 °C.

8 Determination of the temperature of commencement of self-loaded deformation

8.1 Determination

8.1.1 Where deformation does not occur at the first test temperature, repeat the test described in clause 7 at a temperature approximately 100 K higher, using two new test pieces taken from the same set, and where necessary carry out further tests at intervals of 100 °C until deformation occurs. When a range of 100 °C over which deformation occurs has been established, carry out further tests at intervals of approximately 50 °C, 25 °C and 12,5 °C until the interval between the observation of ‘no deformation’ and ‘deformation’ (see 8.2) is between 10 °C and 15 °C.

8.1.2 Where deformation occurs at the first test temperature, repeat the test described in clause 7 at a temperature approximately 100 K lower, using two new test pieces taken from the same set, and where necessary carry out further tests at intervals of 100 °C until no deformation occurs. Establish the interval between ‘no deformation’ and ‘deformation’ as described in 8.1.1.

8.2 Limits

Draw a graph (see Figure 4) to show the deformation of the test pieces against temperature. Use this graph to determine any permanent deformation, as shown in Figure 4.

NOTE Such permanent deformation can be due to irreversible expansion, oxidation, or relief of residual stress and is not due to the load of the test piece (see 9).

For the purpose of this test, the state of ‘no deformation’ described in 8.1.1 and 8.1.2 occurs when the deformation is 0,03 mm or less, excluding the permanent deformation.

Report the temperature of commencement of self-loaded deformation, taken from the graph as shown in Figure 4.

9 Interferences

Other changes can occur during this test which lead to apparent permanent deformation, including permanent density changes, interval stress relief and oxidation. If oxidation or permanent density changes are suspected, checks shall be made on test-piece dimensions and reported in the test report (see 10 i). Residual stress relief may show variable positive or negative apparent deformation. If this is suspected, ensure sufficient testing is undertaken to distinguish the correct onset of permanent plastic deformation.

10 Test report

The results shall be reported in accordance with EN ISO/IEC 17025, and the test report shall include the following information:

a) name and address of the testing laboratory;

b) date of the tests, report identification, signatory;

c) reference to this method of test, i.e. ‘determined in accordance with EN 820-2;

d) description of the test material (manufacturer, type, batch number);

e) method of preparation of the test pieces (see clause 6);

f) atmosphere of the furnace used;

g) for each test temperature, the individual values of the deformation and the mean value (see 7.6);

h) test temperatures for the series of tests, the temperature at which self-loaded deformation commenced (see clause 8 and Figure 4), and the graph showing the results;

i) comments about the test or the test results.