SLOVENSKI STANDARD
SIST EN 14425-3:2010

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Sodobna tehnična keramika - Preskusne metode za ugotavljanje odpornosti monolitske keramike proti lomljenju - 3. del: Upogibni preskus z zarezo (metoda CNB)

Advanced technical ceramics - Test methods for determination of fracture toughness of monolithic ceramics - Part 3: Chevron notched beam (CNB) method

Hochleistungskeramik - Prüfverfahren zur Bestimmung der Bruchzähigkeit von monolithischer Keramik - Teil 3: Verfahren für Biegeproben mit Chevron-Kerb (CNB-Verfahren)

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Céramiques techniques avancées - Méthode d'essai de détermination de la ténacité à la rupture des céramiques monolithiques - Partie 3: Méthode de l'éprouvette à entaille en chevron

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Advanced technical ceramics - Test methods for determination of fracture toughness of monolithic ceramics - Part 3: Chevron notched beam (CNB) method

Céramiques techniques avancées - Méthode d'essai de détermination de la ténacité à la rupture des céramiques monolithiques - Partie 3: Méthode de l'éprouvette à entaille en chevron

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SIST EN 14425-3:2010

Contents

Page

iTeh STANDARD PREVIEW
(standards.iteh.ai)
SIST EN 14425-3:2010
https://standards.iteh.ai/catalog/standards/sist/6648526f-518f-4174-b18f-24a8778d250f/sist-en-14425-3-2010

SIST EN 14425-3:2010

Foreword

This document (EN 14425-3:2010) 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of an identical text or by endorsement, at the latest by December 2010, and conflicting national standards shall be withdrawn at the latest by December 2010.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.

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SIST EN 14425-3:2010
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SIST EN 14425-3:2010

1 Scope

This European Standard provides a test method for fracture toughness determination based on the chevron-notch method. For the purposes of this European Standard, the term monolithic includes particle and whisker reinforced advanced technical ceramics which can be regarded as macroscopically homogeneous. It does not include long-fibre reinforced ceramics.

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 843-1:2006, *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 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)*

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ISO 3611, *Micrometer callipers for external measurement*

SIST EN 14425-3:2010
https://standards.iteh.ai/catalog/standards/sist/6648526f-518f-4174-b18f-24a8778d250f/sist-en-14425-3-2010

3 Terms and definitions

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

3.1
chevron-notch test piece
test piece in which a two coplanar saw cuts are made at an angle to each other part way through a test piece to leave a remaining cross-section with a sharp tip from which a crack may be initiated in a controlled or semi-controlled manner

4 Principle, significance and use

In the chevron-notched beam method, a crack is generated during the test from a sharp tip resulting when two coplanar notches are cut in a test piece (see Figure 1). This overcomes the need to generate sharp planar cracks before commencing the test.

During the test, the crack front widens as the crack propagates from the tip, resulting in an increase in the force required to maintain growth. Countering this, the test piece becomes more compliant as the crack lengthens, and so the two effects result in a peak load being attained after some distance of propagation. The toughness is determined from the peak force applied.

This test method employs the chevron notch in a flexural strength test piece, loaded in four-point bending geometry. The advantage is that it can be performed on standard flexural strength test pieces.

SIST EN 14425-3:2010

The test is most appropriate for homogeneous isotropic materials, but can be used with care on anisotropic materials subject to the geometry of fracture remaining valid. In materials which are inhomogeneous on the scale of the crack dimensions, a wide scatter of results and the development of invalid non-planar cracks may occur. In addition, the test is valid primarily for materials which do not show rising crack resistance with increasing crack length. Since the crack width progressively increases as propagation occurs, the concurrent presence of rising crack resistance means that the crack front would no longer stay straight but would become convex, producing an uncertainty in the calculated results.

It should be noted that this test employs a slowly moving crack. The numerical value for the fracture toughness calculated from this method may not be the same as those from fast crack propagation tests, especially if the susceptibility to subcritical crack growth is significant.

5 Test apparatus

5.1 Flexural strength test apparatus of four-point bending type, in accordance with the requirements in EN 843-1.

5.2 Mechanical testing machine, capable of accurate recording of load/displacement data for loads in the range 0 N to 500 N. The load shall be calibrated in accordance with EN ISO 7500-1.

NOTE A stiff loading system (i.e. frame, load cell and fixtures, etc.) is advantageous for this test. The compliance is ideally less than 5×10^{-5} m/N (see [1]).

5.3 Micrometer in accordance with ISO 3611 but measuring to an accuracy of 0,002 mm.

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5.4 Calibrated device for measuring dimensions of the cut chevron after fracture, reading to an accuracy of 0,002 mm or better.

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NOTE This may be achieved by use of an appropriate travelling microscope, or a conventional microscope with a calibrated stage micrometer, or a microscope with a micrometer eyepiece.

SIST EN 14425-3:2010
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5.5 Humidity measuring device for measuring relative humidity to an accuracy of ± 2 %, e.g. those according to ISO 4677.

6 Test pieces

6.1 Material selection

Select the material or components from which test pieces are to be machined with reference to the considerations given in EN 1006.

6.2 Test piece dimensions

6.2.1 The test pieces shall preferably be of dimensions as for determination of flexural strength and described in EN 843-1. Alternatively, if availability of material permits, the dimensions may be scaled larger for convenience of machining of the notch.

NOTE It is usually advantageous to test coarse-grained or heterogeneous materials in larger test piece sizes because it improves the chances of obtaining a valid test (see 7.9).

6.2.2 The standard size test pieces shall be (3,00 ± 0,15) mm × (4,00 ± 0,15) mm in cross-section, and have a minimum length of 45 mm. The surfaces shall be machined to a flatness of better than 0,01 mm over the test piece length, and opposite pairs of faces shall be parallel to better than 0,01 mm over the test piece length. The section should be visibly rectangular.

NOTE Chamfering in accordance with EN 843-1 is unnecessary for this test.

SIST EN 14425-3:2010

6.2.3 The test piece surfaces shall be ground to a good quality finish using final grinding with a peripheral wheel of grit size between 320 and 500 mesh grit, using a depth of cut of no more than 0,002 mm for the last 0,04 mm of material removed from each surface. No edge treatment is necessary.

NOTE Although a large notch is placed in the test piece, the quality of grinding remains important for dimensional accuracy for registration in cutting the notch and for minimizing residual stresses.

6.3 Sawn notch

6.3.1 The notch is produced by two saw cuts at an appropriate angle to each other as shown in Figure 1. The notch width shall be less than 0,20 mm at the surface, and the notch root radius should be less than 0,1 mm. The grit size of the saw or grinding wheel employed shall be 320 mesh or smaller. For standard test pieces, the test piece shall be orientated such that the notch tip is opposite the 3 mm width face. The notch geometry may be selected to suit requirements. However, it is recommended that:

a) the angles of the two sides of the notch to the test piece faces are equal to within 1°, and the notch tip is central within the test piece to within 0,05 B where B is the test piece width;

b) the position of the notch tip, a_0, below the test piece surface is about 20 % of the depth of the test piece, W;

c) the positions of run-out of the notch through the test piece sides, a_{11}, a_{12} are closely similar, and are in the range 90 % to 100 % of the test piece depth, W.

NOTE 1 The use of a surface grinder or precision slicing saw permits straight-tipped cuts to be made. The use of a non-traversing saw, or a wire saw, is not recommended because the line of the notch tip produced is not straight. This can introduce a considerable error in the use of the calibration equations.

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NOTE 2 The reproducibility and quality of machining of the notch can influence the reproducibility of the test result. In general, narrower and more precise cutting encourages more reproducible initial stable crack growth and reduces the scatter in results.

SIST EN 14425-3:2010
https://standards.iteh.ai/catalog/standards/sist/6648526f-518f-4174-b18f-24a8778d250f/sist-en-14425-3-2010

6.3.2 A simple method of obtaining the required precision of cutting is to prepare a V-block with an end stop against which the test piece can be registered. The test piece is clamped on one side to permit the first cut, and is then rotated and clamped on the other side to allow the second cut of equal depth to be coplanar with the first. The angle of the V-block is the same as that required for the notch angle.

6.3.3 A minimum of five test pieces shall be prepared.

7 Procedure

7.1 Measure the external dimensions of the test piece at the notch to the nearest 0,002 mm using the micrometer (5.3).

7.2 Measure the notch lengths a_{11} and a_{12} (to the points where the roots of the notch meet the external surfaces) to the nearest 0,002 mm using the calibrated device (5.4). The difference between the average value a_1 and the individual values shall not be greater than 0,05 W, where W is the test piece thickness.

7.3 Adjust the sensitivity of the load recording device to permit an accuracy of subsequent measurement to better than 1 % of recorded values. Adjust the recording and plotting parameters of the recording device to permit clear identification of the load/time or load/displacement behaviour (see Figure 2).

7.4 Insert the test piece in the strength test apparatus with the tip of the chevron notch facing the outer support rollers, and align it carefully to ensure that the support and loading spans are centralised to within ± 0,2 mm in accordance with EN 843-1:2006.

7.5 Select for the test machine displacement rate a value which achieves fracture in typically 2 min to 5 min.

SIST EN 14425-3:2010

NOTE Typically this requires a machine displacement rate of about 0,05 mm/min or slower. If the fracture force range is known, it is possible to use a faster rate up to about 75 % of the fracture force, before switching to the slower rate.

7.6 Measure and record the relative humidity and temperature of the atmosphere in the proximity of the test.

7.7 Load the test piece in the flexural test apparatus and record the load/displacement curve produced as the test piece fractures.

7.8 Inspect the trace (see Figure 2). If the fracture exhibited stable characteristics with the load rising to a smooth maximum and then falling (Figure 2a)), or if the crack popped-in prior to the load passing through a smooth maximum (Figure 2b)), the test can be deemed to be valid. Determine the maximum test force, P_{max}. If the load reached a sharp maximum from which it fell suddenly towards zero without a second smooth maximum (Figure 2c)), fracture was initiated unstably and the test is invalid.

NOTE 1 If the test piece shows a loss of stiffness before fracture this is an indication of subcritical crack growth from the notch, even if a clear smooth maximum is not obtained. Such examples are currently considered to be marginally valid, and should be reported in the report.

NOTE 2 In order to induce stable propagation in such circumstances, one technique that has been found helpful is to fatigue the notch tip in compression. Invert the test piece in the flexural test apparatus, and load it several times to two or three times the estimated failure load for the normal position. Revert the test piece to its normal testing position and perform the test in the usual way. If invalid behaviour persists, an alternative method should be sought.

7.9 Inspect the chevron exposed by fracture on the test piece. Using the calibrated device (5.4) measure the position of the notch tip relative to the centre-line of the test piece. The deviation of the notch tip from the centre-line should not exceed 0,05 B, where B is the test piece width. The centre-lines of the two grooves forming the notch tip should coincide within 0,2 t where t is the notch width. Measure the distance of the notch tip from the free surface, a_0.

iTeh STANDARD PREVIEW
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7.10 Examine the fracture surface to determine how well the fracture followed the plane of the notch (see Figure 3). If the deviation of the fracture surface from the plane defined by the centres of the two parts of the notch exceeds 0,04 B at the point where the crack front had a width of 0,33 B, then the test is deemed invalid.

SIST EN 14425-3:2010
https://standards.iteh.ai/catalog/standards/sist/66485261-5181-4174-b181-24a8778d250f/sist-en-14425-3-2010

NOTE Such a deviation is commonly caused by strong anisotropy (toughness in the indented plane greater than in another orientation), inhomogeneity, or coarse grain size.

7.11 Repeat the procedure on further test pieces until at least five valid test results are obtained.

8 Calculation of results

For each test, calculate the fracture toughness $K_{I,cnb}$ from the following equation:

$$K_{\mathrm{I,cnb}} = Y^{*}_{\min}\left[\frac{P_{\max}(S_o - S_i)}{BW^{3/2}}\right] \tag{1}$$

where

$$Y^{*}_{\min} = (3{,}08 + 5{,}00\alpha_0 + 8{,}33\alpha_0^2)\left(1 + 0{,}007\left(\frac{S_i S_o}{W^2}\right)^{1/2}\right)\left(\frac{\alpha_1 - \alpha_0}{1 - \alpha_0}\right) \tag{2}$$

and where

$K_{I,cnb}$ is the fracture toughness determined by the CNB test, in MPa·m$^{1/2}$;

α_0 = a_0/W;