SLOVENSKI STANDARD
SIST EN 843-3:2005

01-november-2005

Nadomešča:
SIST ENV 843-3:2000

Advanced technical ceramics - Mechanical properties of monolithic ceramics at room temperature - Part 3: Determination of subcritical crack growth parameters from constant stressing rate flexural strength tests

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Hochleistungskeramik - Mechanische Eigenschaften monolithischer Keramik bei Raumtemperatur - Teil 3: Bestimmung der Parameter des unterkritischen Risswachstums aus Biegefestigkeitsprüfungen mit konstanter Spannungsrate

Céramiques techniques avancées - Propriétés mécaniques des céramiques monolithiques a température ambiante - Partie 3: Détermination des parametres de propagation sous-critique des fissures a partir des essais de résistance a la flexion réalisés a vitesse de contrainte constante

Ta slovenski standard je istoveten z: **EN 843-3:2005**

ICS:

81.060.30 Sodobna keramika Advanced ceramics

SIST EN 843-3:2005 **en**

EUROPEAN STANDARD

NORME EUROPÉENNE

EUROPÄISCHE NORM

EN 843-3

June 2005

ICS 81.060.30

Supersedes ENV 843-3:1996

English version

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EUROPEAN COMMITTEE FOR STANDARDIZATION
COMITÉ EUROPÉEN DE NORMALISATION
EUROPÄISCHES KOMITEE FÜR NORMUNG

Management Centre: rue de Stassart, 36 B-1050 Brussels

Ref. No. EN 843-3:2005: E

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Foreword

This document (EN 843-3:2005) 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of an identical text or by endorsement, at the latest by December 2005, and conflicting national standards shall be withdrawn at the latest by December 2005.

EN 843 *‘Advanced technical ceramics – Mechanical properties of monolithic ceramics at room temperature’* consists of six parts:

Part 1: *Determination of flexural strength*;

Part 2: *Determination of Young’s modulus, shear modulus and Poisson’s ratio*;

Part 3: *Determination of subcritical crack growth parameters from constant stressing rate flexural strength tests*;

Part 4: *Vickers, Knoop and Rockwell superficial hardness*;

Part 5: *Statistical analysis*;

Part 6: *Guide for fractographic examination*.

This document supersedes ENV843-3:1996.

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard: 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 European Standard specifies a method for the determination of subcritical crack growth parameters of advanced monolithic technical ceramics in the temperature range 15 °C to 30 °C by measuring the dependence of mean fracture strength on the rate of loading. The method is based on strength test procedures described in EN 843-1. This European Standard is not applicable to test pieces with artificially introduced flaws or cracks.

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, *Advanced technical ceramics — Mechanical properties of monolithic ceramics at room temperature — Part 1: Determination of flexural strength*

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 17025:1999)*

ISO 3611, *Micrometer callipers for external measurement*

ISO 4677-1, *Atmospheres for conditioning and testing — Determination of relative humidity — Part 1: Aspirated psychrometer method*

ISO 4677-2, *Atmospheres for conditioning and testing — Determination of relative humidity — Part 2: Whirling psychrometer method*

3 Terms and definitions

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

3.1
nominal flexural strength
maximum nominal stress at the instant of failure supported by the material when loaded in linear elastic bending

3.2
three-point flexure
means of bending a beam test piece whereby the testpiece is supported on bearings near its ends and a central load is applied

3.3
four-point flexure
means of bending a beam test piece whereby the test piece is supported on bearings near its ends and is equally loaded at two positions symmetrically disposed about the centre of the supported span

3.4
subcritical crack growth
extension of existing cracks or flaws under a stress which does not produce instant failure

3.5
subcritical crack growth parameters
parameters describing the relationship between crack velocity and stress intensity factor

4 Significance and use

Subcritical crack growth can occur in brittle solids at stress levels below that required to cause instantaneous failure. This effect may be caused by the testing environment, or by the intrinsic crack propagation behaviour of the material. The phenomenon leads to a decay of remaining strength in a manner determined by the loading history of the component or test piece.

NOTE 1 A review of subcritical crack growth may be found in [1].

The determination of subcritical crack growth parameters in accordance with this document allows the characterisation of the susceptibility of the material to subcritical crack growth, and thus its ability to support continued mechanical loading. Using these parameters it is possible to compare materials for susceptibility to loss of strength under load in particular environments, and to estimate the lifetime of a component used under similar loading and environmental conditions.

NOTE 2 The use of these parameters in design and lifetime estimation is not within the scope of this document.

The relationship between the stress intensity factor at the tip of a crack or flaw and the velocity of the subcritically growing crack may be given by:

$$v = A_0 \left(\frac{K_I}{K_{Ic}} \right)^n \qquad (1)$$

where

v is the velocity of the growing crack in metres per second;

A_0 is a constant in metres per second;

K_I is the critical stress intensity factor developed at the crack tip by the applied stress in Megapascals metres$^{1/2}$;

K_{Ic} = critical stress intensity factor at the crack tip required to cause instantaneous crack propagatio.

NOTE 3 There are other algebraic representations of this relationship which are less convenient mathematically, but may be physically more realistic in practice. See, e.g. [2] and [3]. [2] considers that practical data cannot reliably distinguish between various relationships. The mathematical analysis in this document therefore does not cover such alternative relationships.

In Equation (1), the value of n at room temperature is normally high, typically in the range fifteen to several hundred. At the lower end of this range, materials are very susceptible to subcritical crack growth, while at the upper end the phenomenon becomes insignificant. It should be recognised that Equation (1) implies a single simple relationship, but in practice there may be non-linearities. There are thought to be two principal causes of non-linearity:

a) At low stress intensity factors there may be no subcritical crack growth. This is termed the subcritical crack growth threshold, or "fatigue limit".

b) At intermediate stress intensity factors, the crack growth rate may be limited by the rate at which the environment can penetrate along the crack to control fracture at the tip. This results in a plateau effect, which is maintained to K_I levels at which crack growth can occur in the absence of an environmental effect.

It should be noted that n and A_0 are often functions of the environmental conditions employed. In particular, many ceramics show marked subcritical crack growth in humid air or in water, and much less marked effects in dry or inert conditions. The test environment shall be defined and controlled for reproducible results.

In this document, the parameter n and a parameter B_0, which is related to A_0, are determined from the effect of stressing rate on flexural strength.

NOTE 4 The term "dynamic fatigue" is frequently used to describe such tests, but tends to be misunderstood. Its use is discouraged.

Annex A shows how the mathematical formulation of the relationship between the subcritical crack growth parameters based on Equation (1) and the effect of stressing rate on strength is derived, yielding the basic formula:

$$\log \sigma_f = \log B_0 + \frac{1}{n+1} \log \dot{\sigma} \qquad (2)$$

where

σ_f is the nominal fracture strength of a test-piece in MPa;

B_0 is the constant;

$\dot{\sigma}$ is the stressing rate employed in MPa per second.

NOTE 5 This equation is strictly correct only if a consistent failure probability at each loading rate is employed, e.g. P_f = 0,5, which is calculable from a test piece population of ≥ 30 via, for example, fitting a Weibull distribution. If a smaller number of tests is used, as in this test method, the potential uncertainty is likely to be greater.

NOTE 6 This test method may be used only to measure B_0. Calculation of A_0 requires knowledge of other material parameters not determined by this test method.

The subcritical crack growth parameters are determined by employing several different stressing rates, plotting a graph of log σ_f versus log $\dot{\sigma}$, and calculating the slope (1/(n+1)) and the intercept (log B_0).

NOTE 7 If the test material is to be exposed to an environment in which severely corrosive processes are likely to occur, loss of strength with increasing exposure time may override true subcritical crack growth behaviour. If such an eventuality is suspected, it is recommended that a test is performed in which additional test pieces are exposed to the corrosive environment without stressing for a duration similar to that required for the slowest testing anticipated in the test series, and are subsequently strength tested to compare residual short-term strength with short-term strength before corrosion.

5 Apparatus

5.1 Test jig

The test jig shall be in accordance with the provisions described in EN 843-1, with the force being applied to the test piece through parallel self-aligning freely rotating loading rollers of adequate hardness. The test jig span may be either 20 mm ± 0,5 mm (span A) or 40 mm ± 0,5 mm (span B), and the loading may be in either three-point flexure or four-point flexure. In four-point flexure, the central loading span may be either 10 mm ± 0,2 mm for span A or 20 mm ± 0,2 mm for span B, and shall be symmetrically positioned to within 0,1 mm with respect to the outer support span. The spans and distances between load and support rollers shall be measured with a travelling microscope to the nearest 0,1 mm along the length of the test-piece using a travelling microscope or similar device (5.4.2).

The material from which the jig is constructed shall be compatible with the environment to be used for the tests, and shall not corrode in such a way as to impair its self-aligning and friction-free capability.

NOTE For tests in water, it is recommended that the test jig be constructed from stainless steel that does not suffer badly from crevice corrosion, e.g. 316 grade. The rollers may conveniently be machined from hard ceramics such as alumina, dense silicon nitride or silicon carbide.

5.2 Environmental control

If the tests are to be performed in any environment other than ambient air, an appropriate containment facility shall be constructed to allow the test conditions to be controlled. For tests in water, a simple water tank shall suffice. For controlled humidity or gaseous environments, an environmental chamber and control system is required. For corrosive chemical solutions, appropriate safety precautions shall be adopted for handling and containment as required by local regulations.

5.3 Test machine

The test jig and environmental control facility shall be assembled in a suitable mechanical testing machine which is capable of applying a force to the loading roller (three-point flexure) or equally to the two loading rollers (four-point flexure) in order to stress the test-piece. The machine shall be capable of applying the force at a range of constant rates covering at least four orders of magnitude within the range 100 N s^{-1} and 0,005 N s^{-1}.

NOTE 1 If the available machine does not offer pre-selected loading rates (load control), it is permissible to employ cross-head displacement rate control. The actual test piece stressing rate is determined from the tangent of the force/time record at the time of failure.

The test machine shall be equipped for recording the peak force applied to the test-piece. The accuracy of the test machine shall be in accordance with EN ISO 7500-1, Grade 1 (accuracy better than 1 % of indicated load).

The test machine shall be subject to regular certified calibration in accordance with ISO 7500-1. Before undertaking tests, the validity of the calibration shall be checked. It is essential that the accuracy of the force measuring device is maintained during loading to better than 1 % of the indicated load at all loading rates.

NOTE 2 Many older mechanical testing machines do not have an adequate response rate in load recording at fast loading rates. The minimum time to failure in such cases should be greater than 5 s.

5.4 Linear measuring devices

5.4.1 Micrometer, in accordance with ISO 3611, capable of recording to 0,01 mm and accurate to this level. Flat anvils shall be used to avoid damaging the test piece during measurement.

5.4.2 Travelling microscope, or other suitable device accurate to 0,05 mm, used for measuring span and distances between loading rollers of the test jig.

5.5 Drying oven

A drying oven capable of maintaining a temperature of 120 °C ± 5 °C.