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Hochleistungskeramik - Monolithische Keramik. Mechanische Eigenschaften bei Raumtemperatur - Teil 1: Bestimmung der Biegefestigkeit

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Foreword

This document (prEN 843-1:2004) has been prepared by Technical Committee CEN/TC 184 “Advanced technical ceramics”, the secretariat of which is held by BSI.

This document is currently submitted to the CEN Enquiry.

This document will supersede EN 843-1:1995.

1 Scope

This Part of EN 843 describes methods for determining the nominal flexural strength of advanced monolithic technical ceramic materials at ambient temperature. The available loading geometries are three- and four-point flexure, using rectangular section test pieces of two prescribed geometries: 20 mm support span (A) and 40 mm support span (B).

NOTE This standard differs from ISO 14704 in respect of span A (not included in the ISO version), the absence of the 30 mm span option, and the mandatory use of a fully articulating test jig.

The test is appropriate for materials with grain size less than 200 µm.

The test prescribes four categories of surface finish applied to the test pieces:

I: as-fired or annealed after machining;

II: machined using agreed grinding procedures and material removal rates;

III: standard finishing procedures:

III.1: finishing by grinding;

III.2: finishing by lapping/polishing.

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 623-4, *Advanced technical ceramics — General and textural properties of monolithic ceramics — Part 4: Surface roughness*

ENV 843-5, *Advanced technical ceramics — Mechanical properties of monolithic ceramics at room temperature — Part 5: Statistical analysis of fracture data*

EN ISO 7500-1, *Metallic materials. Verification of static uniaxial testing machines — Tension/compression testing machines — Verification and calibration of the force-measuring system*

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 Definitions

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 test piece is supported on bearings near its ends, and a central force 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 loaded equally at two positions symmetrically disposed about the centre of the supported span

NOTE The term **quarter-point flexure** is sometimes used for the four-point flexure geometry wherein the load positions are each one-quarter of the support span from the support bearings, as is the case in this standard.

4 Significance and use

This test is intended to be used for material development, quality control, characterisation and design data acquisition purposes. The strength level determined by the test is calculated on the basis of linear elastic bending of a thin beam on the assumption that the material being tested is elastically homogeneous and isotropic, and shows linear (Hookean) stress-strain behaviour.

The result obtained from a strength test is determined by a large number of factors associated with the microstructure of the material, the surface finishing procedure applied in preparation of the test pieces, the size and shape of the test piece, the mechanical function of the testing apparatus, the rate of load application and the relative humidity of the ambient atmosphere. As a consequence of the brittle nature of ceramics, there is usually a considerable range of results obtained from a number of nominally identical test pieces. These factors combined mean that caution in the interpretation of test results is required. For many purposes, and as described in this standard, the results of strength tests may be described in terms of a mean value and a standard deviation. Further statistical evaluation of results is required for design data acquisition, and may be desirable for other purposes (see ENV 843-5).

This method places closely defined restrictions on the size and shape of the test-piece and on the function of the test apparatus in order to minimize the errors that can arise as a consequence of the test method.

NOTE The basis for the choice of dimensions and tolerances of test-pieces and of the requirements of the test-jig may be found in reference [1].

All other test factors are required to be stated in the test report (see clause 9) in order to allow intercomparison of material behaviours. It is not possible rigorously to standardise particular surface finishes, since these are not absolutely controllable in mechanical terms. The inclusion of a standard preparation procedure (see 6.3) as one of the surface finish options in this method is intended to provide a means of obtaining a minimum amount of residual grinding damage in the test material.

The extrapolation of flexural strength data to other geometries of stressing, to multiaxial stressing, to other rates of stressing or to other environments should be viewed with caution.

The origin of fracture in a flexural test can be a valuable guide to the nature and position of strength-limiting defects. Fractography of test pieces is highly recommended. In particular, the test may identify fracture origins as being edge defects (caused by edge preparation), surface defects (caused by surface preparation), or internal defects (caused by manufacturing inhomogeneities such as pores, large grains, impurity concentrations, etc.). Not all advanced monolithic technical ceramics are amenable to clear fractography.

5 Apparatus

5.1 Test jig

The test jig shall function as specified below in order to minimize misalignments, twist and frictional forces applied to the test piece.

NOTE 1 The precise test jig design is not specified, only the function.

Schematic arrangements of the test jig function are shown in Figure 1(a) for three-point flexure, and Figure 1(b) for four-point flexure.

The test piece is supported on two bearing edges perpendicular to its length. The outer support bearing edges shall be parallel rollers of diameter approximately 1,5 times the test piece thickness. Diameters of between 2.2 mm and 2,5 mm (for span A; see 6.2) or between 4,5 mm and 5,0 mm (for span B; see 5.2) are recommended. The rollers shall be capable of rolling outward on flat support surfaces (Figure 2). One of the rollers shall additionally be capable of rotating about an axis parallel to the length of the test-piece such that torsional loading is minimized. The two rollers shall be positioned initially with their centres 20 mm ± 0,5 mm apart (span A) or 40 mm ± 0,5 mm apart (span B) with their axes parallel to 0,2 mm over their lengths (≥ 12 mm).

The separation of the centres of the rollers in their starting positions shall be measured to the nearest 0,1 mm with the travelling microscope (see 5.3.2) or other suitable device. The rollers shall be made from hardened steel or other hard material with a hardness greater than 40 HRC (Rockwell C-scale) for strengths less than 1,4 GPa, and not less than 46 HRC for strengths greater than 1,4 GPa. The rollers shall have a smooth burr-free surface finish with roughness less than 0,5 µm R_a, and shall have diameter uniform to ± 0,015 mm.

For three-point flexure, a third roller is located at the mid-point between and parallel to the two support rollers. This roller has the same diameter as the support rollers, and is similarly free to rotate about an axis parallel to the length of the test piece. Its position relative to the midpoint between the support rollers shall be to better than 0,2 mm, measured to the nearest 0,1 mm in a direction parallel to the length of the test piece using the travelling microscope or other suitable device (see 5.3.2).

For four-point flexure, two loading rollers are located at the quarter points (see clause 3.3 note), i.e. with inner spans 10 ± 0,2 mm (outer span A) or 20 ± 0,2 mm (outer span B), and are free to roll inwards. As with the three-point apparatus, the two rollers are also free to rotate separately about an axis parallel to the length of the test piece to allow alignment. The loading rollers shall be symmetrically positioned to within ± 0,1 mm. The distances between the centres of the support rollers and adjacent loading rollers shall be measured to the nearest 0,1 mm along the length of the test-piece perpendicular to the direction of loading, using the travelling microscope or other suitable device (see 5.3.2). The arrangement for loading shall ensure that equal forces are applied to the two loading rollers.

NOTE 2 The accurate and repeatable lateral positioning of loading rollers can best be achieved by ensuring that in the unloaded position, the support rollers are in lateral contact with stops which allow the rollers to roll outwards on their support planes towards the ends of the test-piece, and the inner loading rollers (four-point flexure) are in contact with stops which allow rolling inwards towards the middle of the test-piece. The rotation of the rollers is thus unhindered when load is applied.

5.2 Test machine

The test apparatus shall be arranged in a suitable mechanical testing machine which shall be 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 constant loading or displacement rate. The test machine shall be equipped for recording the peak load applied to the test piece. The accuracy of the test machine shall be in accordance with EN ISO 7500-1, Grade 1 (accuracy 1 % of indicated load).

5.3 Linear measuring devices

5.3.1 Micrometer

A micrometer in accordance with ISO 3611, but of resolution 0,002 mm, or alternative device measuring to this resolution.

5.3.2 Travelling microscope

A travelling microscope or other suitable device accurate to 0,05 mm (used for measurement of distance between loading rollers).

5.4 Drying oven

A drying oven capable of maintaining 120 °C ± 10 °C, or other device which has an equivalent heating effect.

5.5 Humidity measuring device

A device for measuring relative humidity to an accuracy of ± 2 %, e.g. those according to ISO 4677.

6 Test pieces

6.1 General

The test-pieces shall be selected and prepared according to agreement between the parties. They may either be specially processed to, or close to, the final required dimensions specified below, or may be machined from larger blocks or components.

NOTE 1 On occasion it may be desirable to test specimen geometries that fall outside the scope of this method. In such a case it is still advisable to follow the guidelines given in this standard concerning jig function to minimise errors of measurement.

NOTE 2 The strength of many types of advanced monolithic technical ceramics is strongly influenced by the machining procedure adopted in the preparation of the test pieces. Low strengths may be caused by grinding with coarse diamond grit sizes, and conversely, very high strengths may be obtained if care in polishing is taken. Some materials, especially those containing transformable zirconia, may be markedly strengthened by appropriate grinding schedules. Reporting of surface preparation conditions is therefore an important aspect of this test method.

6.2 Dimensions and tolerances

For span A (see 5.1), the test pieces shall be ≥ 25 mm in length, 2,5 mm ± 0,2 mm in width and 2,0 mm ± 0,2 mm in thickness, and for span B (see 5.1), the test pieces shall be ≥ 45 mm in length, 4,0 mm ± 0,2 mm in width and 3,0 mm ± 0,2 mm in thickness. Test piece dimensions may be outside these ranges, but deviations from the specification above shall be reported in the test report (8). The maximum tolerable variation in either cross-sectional dimension of each test piece shall be either:

a) for test pieces which have been machined (see 6.3.2 and 6.3.3), ± 0,02 mm, or

b) for as-fired test pieces (see 6.3.1), ± 0,1 mm along the test piece length, and ± 0,05 mm across the test piece width or thickness.

Accurate finishing or the test piece ends is not required.

NOTE 1 This allows some flexibility on actual dimensions, but ensures that the test-pieces are adequately uniform in cross-section for accuracy of stress calculation.

The cross-section of the test piece shall be rectangular to within conventional engineering practice. Out-of-squareness of sides to faces of the test pieces shall be less than 5° as determined by vernier protractor or engineering shadowgraph. For as-fired test pieces, the maximum tolerable twist along the length of the test piece shall be less than 2°, as determined by use of a shadowgraph or other suitable arrangement.

The two long edges bounding the face of the test piece to be subjected to tensile stress in the flexural test shall be chamfered at approximately 45° to a distance of 0,12 mm ± 0,03 mm as measured along the face or side of the test piece. Alternatively, if appropriate, the long edges may be rounded to 0,15 mm ± 0,05 mm.

NOTE 2 All four long edges may be chamfered or rounded.

The machining direction during chamfering shall be performed parallel to the length of the test piece.

NOTE 3 If the chamfer size exceeds that given above, the formulae employed for the calculation of fracture stress require modification according to reference [1].

Reject any test pieces which do not fulfil the above dimensional criteria.

6.3 Surface finish

NOTE Four surface conditions are categorised in this standard: I, II, III.1 and III.2.

6.3.1 I: As-fired:

The test-pieces may be tested in the as-fired (ex-kiln) condition without further surface preparation provided that they have dimensions within the tolerances given in 6.2 above. The long edge chamfer or radius (see 6.2) shall be applied before firing. Test pieces which are outside these limits shall be rejected.

6.3.2 II: Machined:

The test pieces may be prepared by machining in any relevant manner. If they are to be prepared from blocks of material and tested as-cut, they shall be carefully cut using a diamond saw (of grit size not greater than D151: approximately 125-150 μm[1)] in order to obtain a surface roughness R_t not greater than 5 μm (ignoring obvious pores; see ENV 623-4). The test pieces may be tested with a sawn, ground and/or lapped surface finish using an agreed sequence of abrasives, machining direction and material removal rates. They may also be annealed or refired after grinding. Full details of preparation procedures must be appended to the test results.

NOTE The use of a fine abrasive for a finishing process may not completely remove the effects of previous coarser grinding stages. Caution is advised in the interpretation of strength data related to finishing processes.

6.3.3 Standard preparation procedures:

6.3.3.1 Introduction

This procedure is recommended when there is no other specified method for the test. The aim is minimise the damage created in the test pieces due to the preparation method in order to examine strength more representative of the material than the machining. The recommended procedures cannot

1) Federation of European Abrasives Producers (FEPA) designation