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Advanced technical ceramics - Mechanical properties of ceramic composites at high temperature under air at atmospheric pressure - Determination of flexural strength

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Hochleistungskeramik - Mechanische Eigenschaften von keramischen Verbundwerkstoffen bei hoher Temperatur au Luft bei Atmosphärendruck - Bestimmunng der Beigefestigkeit

Céramiques techniques avancées - Propriétés mécaniques des céramiques composites a haute température sous air a pression atmosphérique - Détermination de la résistance en flexion

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This European Standard was approved by CEN on 7 September 2002.

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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
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Contents

Foreword

This document (EN 12789:2002) 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of an identical text or by endorsement, at the latest by April 2003, and conflicting national standards shall be withdrawn at the latest by April 2003.

This document supersedes ENV 12789:1998.

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, Iceland, Ireland, Italy, Luxembourg, Malta, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland and the United Kingdom.

1 Scope

This European Standard specifies the conditions for determination of the flexural strength of ceramic matrix composite materials with continuous fibre reinforcement under three-point or four-point bending for temperatures up to 1 700 °C in air at atmospheric pressure.

This standard applies to all ceramic matrix composites with a continuous fibre reinforcement, unidirectional (1D), bidirectional (2D), and tridirectional (xD, with $2 < x \leq 3$), loaded along one principal axis of reinforcement.

NOTE 1 In most cases, ceramic matrix composites to be used at high temperature in air are coated with an antioxidation coating.

NOTE 2 The purpose of this standard is to determine the flexural strength of a material under an oxidizing environment but not to measure material oxidation.

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 ISO 7500-1, *Metallic materials - Verification of static uniaxial testing machines - Part 1: Tension/compression testing machines (ISO 7500-1:1999)*.

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

EN 60584-2, *Thermocouples - Part 2: Tolerances. (IEC 60584-2:1982 + A1:1989)*.

ISO 3611, *Micrometer callipers for external measurement.*

3 Principle

A test piece of specified dimensions is heated to the testing temperature. It is subsequently loaded in flexure to fracture in such a way that failure occurs in tension or in compression along one principal axis of reinforcement.

The test is performed at constant crosshead displacement rate.

The test duration is limited to reduce any time dependent effects (creep, etc.).

4 Terms and definitions

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

4.1
test temperature, T
temperature at the centre of the test piece

4.2
maximum flexural force, F_m
highest recorded force in a flexural test on the test piece when tested to failure

4.3
flexural stress, σ
the nominal stress on the outer surface of the test piece, calculated at mid-span

NOTE This stress is conventionally calculated according to the simple beam theory, whose basic assumptions cannot be met by ceramic matrix composite materials.

4.4
flexural strength, $\sigma_{f,m}$
maximum flexural stress applied to a test piece that fractures during a flexural test

two flexural strengths can be distinguished:

— apparent flexural strength, $\sigma_{f,m,a}$, when the apparent dimensions are used

— effective flexural strength, $\sigma_{f,m,e}$, when the dimensions are corrected by a factor to account for the presence of the antioxidative protection

5 Apparatus

5.1 Test machine

The machine shall be equipped with a system for measuring the force applied to the test piece. The system shall conform to grade 1 according to EN ISO 7500-1.

5.2 Test jig

The test jig is composed of two parts, linked to the fixed and mobile parts of the machine. It has two outer support rollers and one (three-point bending) or two (four-point bending) inner support rollers.

The material of the jig and that of the rollers shall not react with that of the specimen, nor with the environment.

The cylindrical rollers shall have a diameter of 4 mm to 10 mm. Their length shall be at least equal to the width of the specimen. They shall be made of a material with a hardness at least equal to that of the specimen. The axes of the rollers shall be parallel to within 0,01 mm/mm.

The outer rollers (three and four-point bending) and inner rollers (four-point bending) shall be free to rotate (see Figure 1).

Either two or three rollers, for three-point or four-point bending respectively, shall be free to pivot around an axis parallel to the longitudinal direction of the test piece, in order to adapt to the non-parallelism of the upper and lower faces of unmachined test pieces(see Figure 1).

The rolling and pivoting ability of the rollers shall not be affected by heating. The distance between rollers shall be in accordance to clause 6.

The inner roller(s) shall be centred with respect to the outer rollers to within 0,2 mm. In the case of four-point bending a levelling system shall be used to ensure symmetrical loading of the test piece.

The performance shall not change because of heating.

5.3 Set-up for heating

The set-up for heating shall be constructed in such a way that the variation of temperature of part of the test piece which is between the outer support rollers is less than 50 °C at test temperature.

5.4 Temperature measurement

Thermocouples shall comply with EN 60584-1 and EN 60584-2. Alternatively, pyrometers or thermocouples which are not covered by EN 60584-1 and EN 60584-2, but which are appropriately calibrated can be used.

5.5 Data recording system

A calibrated recorder shall be used to record force-time curve. The use of a digital data recording system combined with an analogue recorder is recommended.

5.6 Micrometers

Micrometers used for the measurement of the dimensions of the test piece shall be in accordance with ISO 3611.

6 Test pieces

Recommended specimen dimensions are given in Table 1. These dimensions have been successfully used and take due account of the following factors:

The amount of material under load shall be representative of the nature of the material and of the reinforcement structure. This sets minimum limits to the span and to the width and thickness of the specimen. In the case of flexural, as opposed to tensile testing, also the distribution of the longitudinal reinforcement through the thickness has to be considered. When this distribution is not symmetrical with respect to the neutral plane, care shall be taken to insure that it is similar between specimens.

The thickness and the distance between the inner and outer roller(s) shall be chosen so as to avoid shear failure. This is achieved by setting a minimum limit to the ratio between the moment arm and the specimen thickness. A value of 10 is commonly used and translates into a minimum L/h ratio of 20 in three-point bending, and a minimum (L-Li)/h ratio of 20 in four-point bending with Li=L/3.

Other factors affecting the size and dimensions of the specimens are the type of the heating and of the loading system.

Table 1 — Recommended test specimen and span dimensions

Dimensions in millimetres

	1D, 2D & xD	Tolerance
L_t, total length	L + 10	± 1
b, width	10	± 0,2
h, thickness	2,5	± 0,2
Inner span four-point bending, L_i	25	± 0,1
Outer span four-point bending, L three-point bending, L	 75 50	 ± 0,1 ± 0,1

NOTE If it is necessary to define test pieces of different dimensions, the conditions of clause 6 should be taken into account.

7 Test piece preparation

7.1 Machining and preparation

During cutting out, care shall be taken to align the longitudinal test piece axis with one of the principal axes of reinforcement.

Specimens may be tested with either machined or non-machined top and bottom surfaces. In some cases machining is not recommended because it may cause breakage of the fibres near the top and bottom surfaces as well as damage to the antioxidation coating if present. When machining these surfaces, care shall be taken to maintain a reinforcement geometry across the thickness of the specimen, which is representative of the material in the as-processed condition. In particular, it shall be assured that the symmetry of an originally symmetric reinforcement geometry is preserved in the specimen cross section after machining.

Machining parameters which avoid damage to the material shall be established and documented. These parameters shall be adhered to during test piece preparation.

NOTE When specimens are machined from a plate which has been protected against oxidation, the cut surfaces of the specimen are unprotected. These surfaces should be protected to prevent oxidation.

7.2 Number of test pieces

At least five valid test results, as specified in paragraph 8.4 are required per loading condition. For specimens with a non-symmetric reinforcement geometry with respect to the neutral plane, tests in two orientations for which the tensile and compressive face are interchanged may be necessary.

If a statistical evaluation of the flexural strength is required, the number of test pieces shall be in accordance with ENV 843-5.

8 Test procedures

8.1 Test set-up: temperature considerations

The following determination shall be carried out under conditions representative of the tests, and shall be repeated every time there is a change in material, in specimen geometry, in loading attachments, etc. In establishing them, time shall be allowed for temperature stabilisation.

Prior to testing, the temperature gradient within the outer span shall be established over the temperature range of interest. This shall be done by measuring the specimen temperature at a minimum of three locations, which shall be the points over the supports and at the centre. The temperature variation within the outer span shall meet the requirements of paragraph 5.3.

During a series of tests, the test temperature can be determined either directly by measurement on the specimen itself, or indirectly from the temperature indicated by the temperature control device. In the latter case, the relationship between the control temperature and test piece temperature at the centre of the length shall be established beforehand on a dummy test piece over the range of temperature of interest. The dummy specimen shall be made of the same material as the specimen to be tested.

NOTE The relationship between the temperature indicated by the temperature control system and the test temperature is usually established simultaneously with the temperature gradient

Temperature may be measured by any means complying with paragraph 5.4. If thermocouples are used to measure the temperature at different locations of the specimen, they shall be embedded (and sealed if necessary) into a dummy specimen to a depth approximately equal to half the specimen dimension in the direction of insertion.