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Fine ceramics (advanced ceramics, advanced technical ceramics) — Mechanical properties of ceramic composites at room temperature — Determination of flexural strength

Céramiques techniques — Propriétés mécaniques des composites céramiques à température ambiante — Détermination de la resistance en flexion



Reference number ISO 17138:2014(E)

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Foreword

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For an explanation on the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the WTO principles in the Technical Barriers to Trade (TBT) see the following URL: Foreword - Supplementary information

The committee responsible for this document is ISO/TC 206, *Fine ceramics*.

Fine ceramics (advanced ceramics, advanced technical ceramics) — Mechanical properties of ceramic composites at room temperature — Determination of flexural strength

1 Scope

This International Standard describes a method for the determination of the flexural strength of ceramic matrix composite materials with continuous fibre reinforcement, under three-point or four-point bend at room temperature. This method applies to all ceramic matrix composites with a continuous fibre reinforcement, unidirectional (1D), bidirectional (2D), and tridirectional xD with $(2 < x \le 3)$ as defined in CEN/TR 13233, loaded along one principal axis of reinforcement.

NOTE The method is not intended to be used to obtain absolute values of strength for design purposes.

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO7500-1, Metallic materials — Verification of static uniaxial testing machines — Part 1: Tension/compression testing machines — Verification and calibration of the force-measuring system

ISO 3611, Geometrical product specifications (GPS) — Dimensional measuring equipment: Micrometers for external measurements — Design and metrological characteristics

CEN/TR 13233: 2007, Advanced technical ceramics — Ceramic composites — Notations and symbols

3 Terms, definitions, and symbols

For the purposes of this document, the terms and definitions given in CEN/TR 13233¹⁾ and the following apply.

3.1

maximum flexural force

 $F_{\rm m}$

highest recorded force in a flexural test on the test specimen when tested to failure

3.2

flexural stress

σ

nominal stress on the outer surface of the test specimen, calculated at mid-span

Note 1 to entry: This stress is conventionally calculated according to the beam theory, whose basic assumptions cannot be met by ceramic matrix composite materials.

3.3

flexural strength

 $\sigma_{\rm f.m}$

maximum flexural stress applied to a test specimen that fractures during a flexural test

¹⁾ An International Standard, ISO 19634, Fine ceramics (advanced ceramics, advanced technical ceramics) — Notations and symbols of ceramic composites is under preparation.

Principle 4

A beam is supported on two outer points, and subjected to an external load applied perpendicularly to a longitudinal axis. The test is performed at constant crosshead displacement rate.

Apparatus 5

5.1 Test machine

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

5.2 **Test fixture**

The test fixture 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 bend) or two (four-point bend) inner rollers for application of load.

The cylindrical rollers shall have a diameter between 4 mm and 10 mm. Their length shall be at least equal to the width of the test specimen. They shall be made of a material with hardness at least equal to that of the test specimen. The axes of the rollers shall be parallel to within 0,01 mm/mm. (See Figure 1.)

Pivoting rollers may be used to adapt the non-parallelism of the upper and lower faces of un-machined test specimens. Either two or three rollers, for three-point or four-point bend, respectively, shall be free to pivot around an axis parallel to the longitudinal direction of the test specimen.

The distance between rollers shall be in accordance with Clause 6.

The inner rollers shall be centred with respect to the outer rollers to within 0,2 mm. In case of four-point bend, the test fixture shall ensure symmetrical loading of the test specimen.

Data recording system 5.3

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

Dimension measuring devices 5.4

Devices used for measuring linear dimensions of the test specimen shall be accurate to ±0,1 mm. Micrometers shall be in accordance with ISO 3611.

Test specimens 6

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

The volume 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 test specimen. In case of flexural, as opposed to tensile, testing, 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 ensure that it is similar for all test specimens.

Two types of test specimens can be distinguished as follows:

as fabricated test specimens, where only the length and the width are machined to give to the test specimens the specified size. In this case, the tensile and compressive surfaces can be unmachined and thus irregular;

b) machined test specimens, where the length, the width, and the thickness of the test specimen have been machined.

Tolerance on thickness is only for machined test specimens. For as fabricated test specimens, the maximum difference in thickness of three measurements (at the centre and at each end of the outer span length) shall not exceed 5 % of the average of the three measurements.

The thickness of the test specimen 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 test specimen thickness (see <u>Table 1</u>). A value of 10 is commonly used and translates into a minimum L/h ratio of 20 in three-point bend, and a minimum $(L-L_i)/h$ ratio of 20 in four-point bend with $L_i = L/3$.

Table 1 — Recommended test specimen and span dimensions

Dimensions in millimetres

| | 1D, 2D, xD | Tolerance |
|------------------------------------|------------|-----------|
| $l_{ m t}$, total length | L + 20 | ±1 |
| b, width | 10 | ±0,2 |
| h, mean thickness of test specimen | ≥2 | ±0,2 |
| Inner span | | |
| $L_{ m i}$, four-point bend | 25 | ±0,1 |
| Outer span | | |
| L, four-point bend | 75 | ±0,1 |
| L, three-point bend | 50 | ±0,1 |

If it is necessary to define test specimens of different dimensions, the conditions of $\underline{\text{Clause 6}}$ should be taken into account.

7 Test specimen preparation

7.1 Machining and preparation

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

Test specimens can be tested with either machined or non-machined tensile and compressive surfaces. In some cases machining is not recommended because it might cause damage to the material near the tensile and compressive surfaces. When machining these surfaces, care shall be taken to maintain reinforcement geometry across the thickness of the test specimen, which is representative of the material in the as-processed condition. In particular, it shall be ensured that the symmetry of originally symmetric reinforcement geometry is preserved in the test 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 specimen preparation.

7.2 Number of test specimens

At least five valid test results (see 8.4) are required.

For test 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 might be necessary.

NOTE If statistical evaluation is required, the number of test specimens should be in accordance with EN 843–5.

Test procedures

Displacement rate

Use a constant crosshead displacement rate which allows quasi-static loading of test specimen and the absence of subcritical crack growth. (0.5 ± 0.1) mm/min or a rate which allows test specimen failure in about 1 min is recommended. The displacement rate shall be reported.

8.2 Measurement of dimensions

Test specimen dimensions 8.2.1

The width and thickness shall be measured to the nearest 0,01 mm in three points near the centre for three-point bend and between the inner rollers for four-point bend, and at each end of the test specimen.

The arithmetic means of the measurements shall be used for calculations.

Distances between supporting rollers 8.2.2

The distance between axes of the outer rollers and for four-point bend also the inner rollers shall be measured to the nearest 0,1 mm.

NOTE This can be achieved by the use of a travelling microscope or other suitable device accurate to 0,05 mm.

Testing technique 8.3

8.3.1 **Test specimen mounting**

The test specimen shall be installed in the test jig centred with respect to the rollers and with its longitudinal axis perpendicular to the axes of the rollers.

It is recommended to use a setting tool for this purpose. NOTE

Zero the load cell output. When required, a small preload can be applied in order to maintain correct positioning of the test specimen. This preload shall not increase beyond 5 % of the expected failure force at any time.

8.3.2 Measurements

Perform the measurements as follows:

- set the displacement rate on the test machine;
- zero the load cell;
- record the force versus time;
- load the test specimen up to failure;
- read the load signal after test specimen failure;
- note the position of fracture location relative to the mid-point of the test specimen to the nearest 1 mm and the failure mode (tensile or compressive).

8.4 Test validity

The following circumstances invalidate a test:

failure to specify and record test conditions;

- failure to meet specified test conditions;
- rupture of the test specimen not occurring in the tensile or compressive mode;
- rupture outside the zone where the bending moment is maximum (i.e. outside the central part, 1/3 L, for three-point bend, and outside the inner span for four-point bend);
- rupture is initiated under a roller.

8.5 Calculation of results

8.5.1 General

8.5.1.1 Test specimen origin

A diagram illustrating the reinforcement directions of the material with respect to the longitudinal axis of the test specimen shall always accompany the test results.

8.5.1.2 Flexural strength

When the onset of nonlinearity in the force displacement curve occurs close to maximum force, calculate the strength using the following expressions. Otherwise, the flexural strength is characterized by the maximum force that can be used to compare specimens with identical dimensions. Add the force displacement curve to the test report.

8.5.2 Three-point bend

$$\sigma_{\rm f,m} = \frac{3F_{\rm m}L}{2hh^2} \tag{1}$$

where

 $\sigma_{f,m}$ is the flexural strength, in megapascals (MPa);

 $F_{\rm m}$ is the maximum flexural force, in newtons (N;

- *L* is the outer span, in millimetres (mm);
- b is the mean width of the test specimen, in millimetres (mm);
- *h* is the mean thickness of the test specimen, in millimetres (mm).

8.5.3 Four-point bend

$$\sigma_{f,m} = \frac{3F_{\rm m}(L - L_{\rm i})}{2bh^2} \tag{2}$$

where

 $\sigma_{f,m}$ is the flexural strength, in megapascals (MPa);

 $F_{\rm m}$ is the maximum flexural force, in newtons (N);

- L is the outer span, in millimetres (mm);
- L_i is the inner span, in millimetres (mm);
- *b* is the mean width of the test specimen, in millimetres (mm);

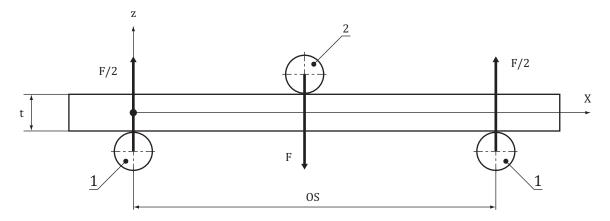
is the mean thickness of the test specimen, in millimetres (mm).

In cases where the test specimen is protected by a coating intended for specific environmental conditions, the calculation procedure of ENV 12789 should be applied.

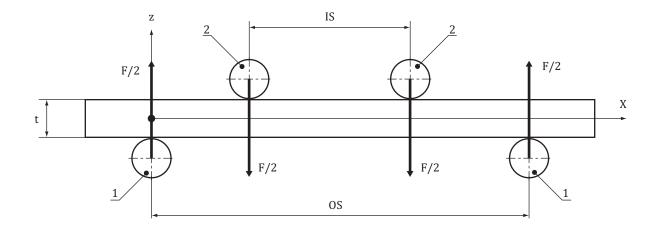
9 **Test report**

The test report shall contain at least the following information:

- name and address of the testing establishment; a)
- b) date of the test, unique identification of report and of each page, customer name and address and signatory;
- a reference to this International Standard, i.e. "determined in accordance with ISO 17138"; c)
- test specimen drawing or reference or geometry and information on machined or unmachined; d)
- span-to-depth ratio; e)
- f) description of the test material (material type, manufacturing code, batch number);
- description of the test set up, load cell, load train, test jig geometry; g)
- h) displacement rate;
- number of tests carried out and the number of valid results obtained; i)
- force versus time records; force versus displacement curves; j)
- valid results, mean values, and standard deviations (for Gaussian distribution); k)
- mode of failure and failure location of all the test specimens used for obtaining the above results; 1)
- test conditions, including test temperature, atmosphere, relative humidity, etc.



a) Three-point bending



b) Four-point bending

Key

- 1 support
- 2 inner roller
- IS inner span
- OS outer span
- t thickness

Figure 1 — Bend tests

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| [1] | ENV 12789: 2002, Advanced technical ceramics — Mechanical properties of ceramic composites at |
|-----|---|
| | high temperature under air at atmospheric pressure — Determination of flexural strength |

[2] EN 843–5, Advanced technical ceramics. Mechanical properties of monolithic ceramics at room temperature Statistical analysis

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