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Fine ceramics (advanced ceramics, advanced technical ceramics) — Mechanical properties of ceramic composites at ambient temperature in air atmospheric pressure — Determination of tensile properties

National foreword

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INTERNATIONAL STANDARD

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Fine ceramics (advanced ceramics, advanced technical ceramics) — Mechanical properties of ceramic composites at ambient temperature in air atmospheric pressure — Determination of tensile properties

Céramiques techniques — Propriétés mécaniques des céramiques composites à température ambiante sous air à pression atmosphérique — Détermination des propriétés en traction

Reference number
ISO 15733:2015(E)

Contents

Page

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*.

This second edition cancels and replaces the first edition (ISO 15733:2001), which has been technically revised.

Fine ceramics (advanced ceramics, advanced technical ceramics) — Mechanical properties of ceramic composites at ambient temperature in air atmospheric pressure — Determination of tensile properties

1 Scope

This International Standard specifies the conditions for determination of tensile properties of ceramic matrix composite materials with continuous fibre reinforcement at room temperatures. This International Standard applies to all ceramic matrix composites with a continuous fibre reinforcement, unidirectional (1D), bi-directional (2D), and tri-directional (*x*D, with $2 < x \leq 3$), loaded along one principal axis of reinforcement.

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

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.

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

ISO 7500-1:2004, *Metallic materials — Verification of static uniaxial testing machines — Part 1: Tension/compression testing machines — Verification and calibration of the force-measuring system*

3 Terms, definitions and symbols

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

3.1
calibrated length
l
part of the test specimen that has uniform and minimum cross-section area

3.2
gauge length
L_0
initial distance between reference points on the test specimen in the calibrated length

3.3
initial cross-section area
S_0
initial cross-section area of the test specimen within the calibrated length

3.4
effective cross-section area
$S_{0\ \mathrm{eff}}$
total area corrected by a factor, to account for the presence of a coating

3.5
longitudinal deformation
A
increase in the gauge length between reference points under a tensile force

3.6
longitudinal deformation under maximum tensile force
A_m
increase in the gauge length between reference points under maximum tensile force

3.7
tensile strain
ε
relative change in the gauge length defined as the ratio A/L_o

3.8
tensile strain under maximum force
ε_m
relative change in the gauge length defined as the ratio A_m/L_o

3.9
tensile stress
σ
tensile force supported by the test specimen at any time in the test divided by the initial cross-section area (S_o)

3.10
effective tensile stress
σ_{eff}
tensile force supported by the test specimen at any time in the test divided by the effective cross-section area ($S_{o\ eff}$)

3.11
maximum tensile force
F_m
highest recorded tensile force in a tensile test on the test specimen when tested to failure

3.12
tensile strength
σ_m
ratio of the maximum tensile force to the initial cross-section area (So)

3.13
effective tensile strength
$\sigma_{m\ eff}$
ratio of the maximum tensile force to the effective cross-section area

3.14
proportionality ratio or pseudo-elastic modulus
EP
slope of the initial linear section of the stress-strain curve, if any

Note 1 to entry: Examination of the stress-strain curves for ceramic matrix composites allows definition of the following cases:

a) Material with an initial linear domain in the stress-strain curve.

For ceramic matrix composites that have a mechanical behaviour characterized by an initial linear section, the proportionality ratio is defined as:

$$EP(\sigma_1,\sigma_2) = \frac{(\sigma_2 - \sigma_1)}{(\varepsilon_2 - \varepsilon_1)}$$

where (ε_1,σ_1) and (ε_2,σ_2) lie near the lower and the upper limits of the linear section of the stress-strain curve.

The proportionality ratio or pseudo-elastic modulus is termed the elastic modulus, E, in the single case where the linearity starts near the origin.

b) Material with no-linear section in the stress-strain curve.

In this case only stress-strain couples can be fixed.

3.15
effective proportionality ratio
EP_{eff}
slope of the linear section of the stress-strain curve, if any, when the effective tensile stress is used

4 Principle

A test specimen of specified dimensions is loaded in tension. The test is performed at constant crosshead displacement rate, or constant deformation rate (or constant loading rate). Force and longitudinal deformations are measured and recorded simultaneously.

NOTE The use of constant loading rate only gives a valid tensile curve when the material behaves linearly up to failure.

5 Apparatus

5.1 Test machine

The test machine shall be equipped with a system for measuring the force applied to the test specimen conforming to grade 1 or better according to ISO 7500-1:2004.

5.2 Load train

The load train configuration shall ensure that the load indicated by the load cell and the load experienced by the test specimen are the same.

The load train shall align the specimen axis with the direction of load application without introducing bending or torsion in the specimen. The alignment should be verified and documented in accordance with, for example, the procedure described in ISO 17161.

The maximum percent bending shall not exceed 5 at an average strain of 500×10^{-6}.

The grip design shall prevent the test specimen from slipping.

NOTE The choice of gripping system depends on material, on test specimen design and on alignment requirements.

5.3 Extensometer

The extensometer shall be capable of continuously recording the longitudinal deformation at test temperature and adapted for small deformation

For mechanical extensometer, the extensometer class should be ≤ 2 (see ISO 9513).

The use of an extensometer with the greatest gauge length is recommended.

The gauge length shall be the longitudinal distance between the two locations where the extensometer rods contact the test specimen.

Care should be taken to correct for changes in calibration of the extensometer that may occur as a result of operating under conditions different from calibration.

If an electro-optical extensometer is used, electro-optical measurements in transmission require reference marks on the test specimen. For this purpose, rods or flags shall be attached to the surface perpendicularly to its axis. The gauge length shall be the distance between the two reference marks. The material used for marks (and adhesive if used) shall be compatible with the test specimen material and shall not modify the stress field in the specimen.

The use of integral flags as parts of the test specimen geometry is not recommended because of stress concentration induced by such features.

Electro-optical extensometer is not recommended in the case where it is not possible to distinguish the colour of the reference marks and the test specimen.

5.4 Data recording system

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

5.5 Micrometers

Micrometers used for the measurement of the dimensions of the test specimen shall conform to ISO 3611.

6 Test specimens

6.1 The choice of the specimen geometry depends on several parameters:

— the nature of the material and of the reinforcement structure;

— the type of the gripping system.

Total length l_t, depends on the type of machine, on the type of grips and on the type of extensometer. It is recommended to use a total length of at least 100 mm.

The volume in the gauge length shall be representative for the material.

Two types of specimen can be distinguished:

— as fabricated specimens, where only the length and the width are machined to give to the specimens the proper size, in the case the two faces of the specimen may present an irregular surface,

— machined specimens where the length and the width and also the two sides of the specimen have been machined.

Tolerance on thickness is only for machined specimens. For as-fabricated specimens the difference in thickness taken out of three measurements (as the centre and at each end of the calibrated length) shall not exceed 5 % of the average of the three measurements.

6.2 Type 1 specimen is represented in Figure 1 and dimensions are given in Table 1.

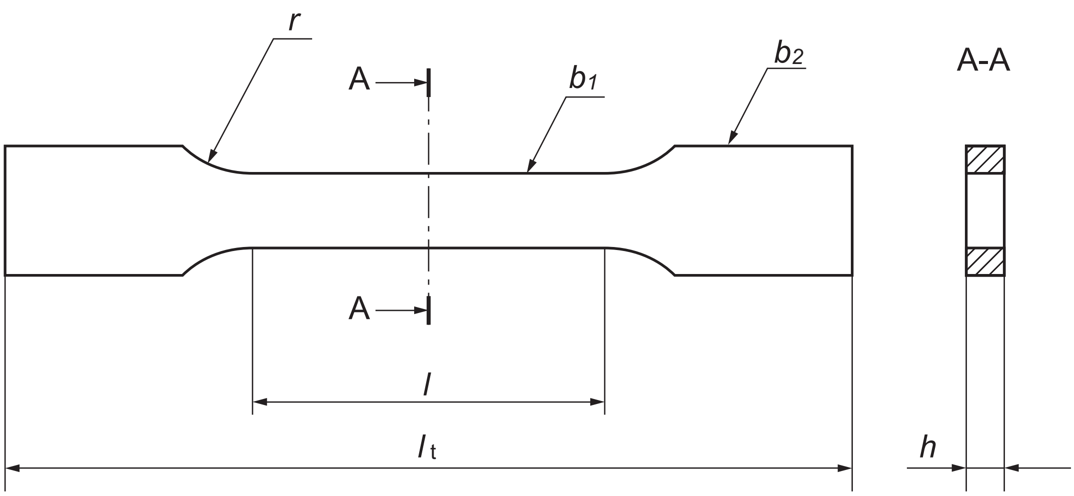

Figure 1 — Type 1 specimen geometry

Table 1 — Type 1 specimen dimensions

Parameter	**Dimensions** mm	
l_t, total length	≥ 100	±0,5
l, calibrated length	≥ 40	±0,2
h, thickness	≥ 3	±0,2
b_1, width in the calibrated length	≥ 8	±0,2
b_2, width	≥ 10	±0,2
r, shoulder radius	≥ 30	±2
Plane parallelism of machined parts	0,05	—

6.3 Type 2, is a straight-sided specimen. It is represented in Figure 2 and dimensions are given in Table 2.

NOTE This test specimen is easy to machine and its use allows mainly the determination of modulus.

Type 2 specimen should not be used for strength measurement.

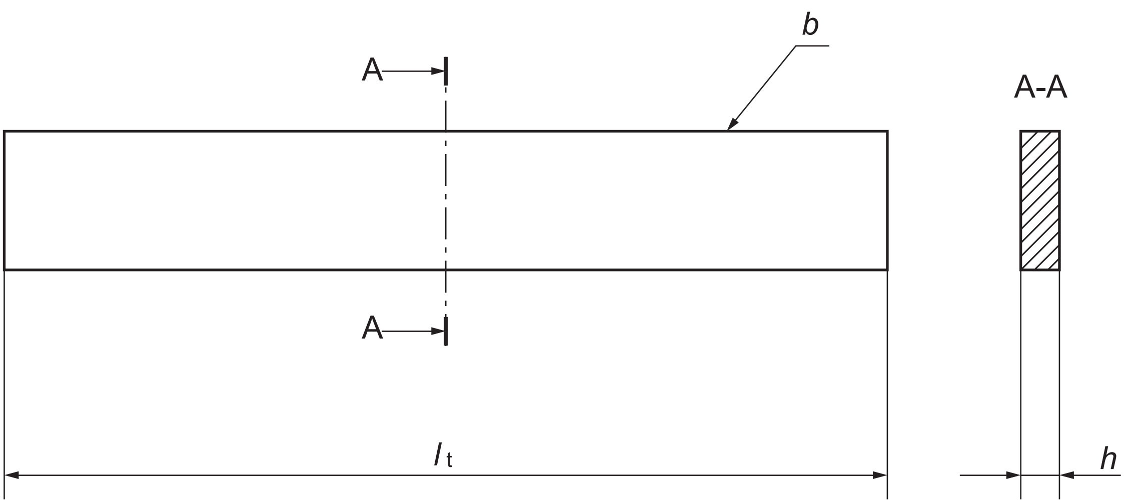

Figure 2 — Type 2 specimen geometry

Table 2 — Type 2 specimen dimensions

Parameter	Dimensions mm	
h, thickness	≥ 3	±0,2
b, width	≥ 10	±0,2
Plane parallelism of machined parts	0,05	—

6.4 Type 3 is a straight-sided specimen equipped with tabs.

Two types of tabs which cover the total gripped length can be used.

a) Metallic or composite tabs bonded or cured on the specimen. The dimensions are given in Table 3 and the specimen is represented in Figure 3. This type of specimen is mainly used for 1D, 2D and *x*D (with $2 < x \leq 3$) materials;

b) Polymeric tabs moulded on the specimen. The dimensions are given in Table 4 and the specimen is represented in Figure 4. This type of specimen is mainly used for 3D materials.

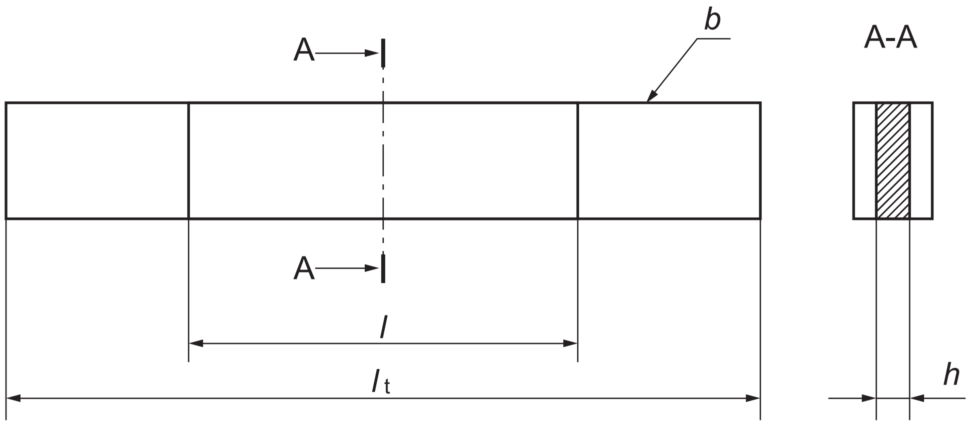

Figure 3 — Type 3 specimen geometry

Table 3 — Type 3 specimen dimensions

Parameter	Dimensions mm	
l_t, total length	≥ 100	±0,5
l, calibrated length	≥ 40	±0,2
Tab length	≥ 30	±0,2
h, thickness	≥ 3	±0,2
b, width	≥ 10	±0,2
Plane parallelism of machined parts and of tab faces	0,05	—
NOTE The thickness of the tabs is generally between 1 mm and 3 mm.		

Dimensions in mm

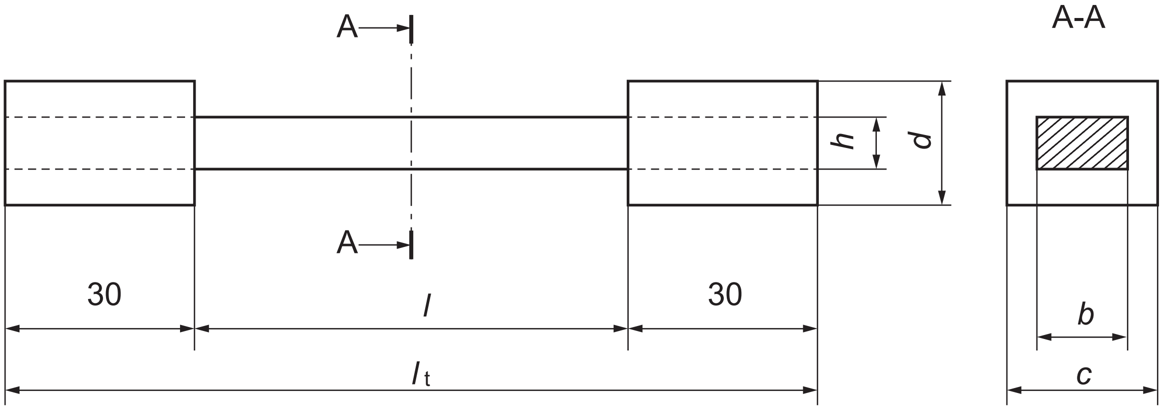

Figure 4 — Type 3 specimen with polymeric tabs

Table 4 — Dimensions of type 3 specimen with tabs

Parameter	**Dimensions** mm	
l_t, total length	≥ 100	±0,5
l, calibrated length	≥ 40	±0,2
Tabs	30 × c × d	±0,2
h, thickness	≥ 3	±0,2
b, width	≥ 10	±0,2
Plane parallelism of machined parts and of tab faces	0,05	–
NOTE c and d are determined so that the thickness of polymer on each side of the test specimen is at least 3mm.		

6.5 Type 4 test specimens: All test specimens designed for high temperature testing (see ISO 14574) can be used for ambient temperature testing.

NOTE It is customary to obtain results at room temperature when testing a material at high temperature and to do so the same specimens are used. However the costs of the test specimens for high temperature tests are generally much higher, and these types are not used when only ambient temperature properties are required.

7 Test specimen preparation

7.1 Machining and preparation

During cutting out, care shall be taken to align the test specimen axis with the desired fibre-related loading axis.

Machining parameters that 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 of specimens

At least five valid test results, as specified in 8.3, are required for any condition.

8 Test procedures

8.1 Test set-up: other considerations

8.1.1 Displacement rate

A displacement rate that allows specimen rupture within 1 min shall be used. The displacement rate and the loading mode shall be reported.

8.1.2 Measurement of test specimens dimensions

The cross-section area is determined at the centre of the specimen and at each end of the gauge length.

Dimensions shall be measured to an accuracy of ± 0,01 mm. The arithmetic means of the measurements shall be used for calculations.

If the test specimen is equipped with marks, the gauge length measured at room temperature, shall be known with an accuracy of ± 1 %.

8.2 Testing technique

8.2.1 Specimen mounting

The test specimen shall be installed in the gripping system with its longitudinal axis coinciding with that of the test machine.

Care shall be taken not to induce flexural or torsional loads.

8.2.2 Setting of extensometer

Install the extensometer longitudinally centred with axis of the test specimen and adjust to zero.

8.2.3 Measurements

— Zero the load cell.

— Zero the extensometer.

— Record the force versus longitudinal deformation.

— Load the test specimen.

8.3 Test validity

The following circumstances invalidate a test:

— failure to specify and record test conditions;

— specimen slippage;

— extensometer slippage.

In case of extensometer slippage the test is invalidated except for the maximum tensile force (F_m).

9 Calculation of results

9.1 Test specimen origin

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

9.2 Tensile strength

Calculate the tensile strength using one of the following equations:

$$\sigma_m = \frac{F_m}{S_o} \quad (1)$$

$$\sigma_{m\,eff} = \frac{F_m}{S_{o\,eff}} \quad (2)$$

where

σ_m is the tensile strength at room temperature, using the initial cross sectional area S_o, in MPa;

$\sigma_{m\,eff}$ is the tensile strength at room temperature using the effective cross sectional area $S_{o\,eff}$, in MPa;

F_m is the maximum tensile force in N;

S_o is the initial cross-sectional area of the specimen in square millimetres (mm^2);

$S_{o\,eff}$ is the effective cross-section area of the specimen corrected to take account of the oxidative protection in mm^2.

When using the effective cross-section area, the applied correction factor shall be given and justified in the test report.

9.3 Strain at maximum tensile force

$$\varepsilon_m = \frac{A_m}{L_o} \quad (3)$$

where

ε_m is the strain at the maximum tensile force;

A_m is the longitudinal deformation at the maximum tensile force in mm measured by the extensometer;

L_o is the gauge length, in mm.

9.4 Proportionality ratio or pseudo-elastic modulus, elastic modulus

9.4.1 Calculate the proportionality ratio or pseudo-elastic modulus *EP* defined between two points (A_1, F_1) and (A_2, F_2) measured near the lower and upper limits of the linear part of the force-deformation record, according to the following expression:

$$EP(\sigma_1,\sigma_2) = \frac{L_0}{S_0}\left(\frac{F_2 - F_1}{A_2 - A_1}\right) \times 10^{-3} \quad (4)$$

$$EP_{\text{eff}}(\sigma_1,\sigma_2) = \frac{L_\text{o}}{S_{\text{o eff}}}\left(\frac{F_2 - F_1}{A_2 - A_1}\right) \times 10^{-3} \quad (5)$$

where

EP	is the pseudo-elastic modulus, in GPa;
EP_{eff}	is the effective pseudo-elastic modulus, in GPa;
F	is the tensile force acting on the specimen, in N;
S_o	is the initial cross-sectional area of the specimen, in mm^2;
$S_{\text{o eff}}$	is the effective cross-section area of the specimen corrected to take account of the oxidation protection, in mm^2;
L_o	is the gauge length, at room temperature, in mm;
A	is the longitudinal deformation, in mm, measured on the curve corresponding to F.

9.4.2 Where the material has a linear behaviour at the origin, calculate the elastic modulus according to the following expression:

$$E = \frac{FL_\text{o}}{S_\text{o}A} \times 10^{-3} \quad (6)$$

$$E_{\text{eff}} = \frac{FL_\text{o}}{S_{\text{o eff}}A} \times 10^{-3} \quad (7)$$

where

E	is the tensile elastic modulus, in GPa;
E_{eff}	is the effective tensile elastic modulus, in GPa;
F	is the tensile force acting on the specimen, in N;
S_o	is the initial cross-sectional area of the specimen, in mm^2;
$S_{\text{o eff}}$	is the effective cross-section area of the specimen corrected to take account of the oxidation protection, in mm^2;
L_o	is the gauge length, in mm;
A	is the longitudinal deformation, in mm, measured on the curve corresponding to F.

Any point (A, F) on the linear section of the force-deformation record may be used for its determination.

9.4.3 For materials with no linear section in the stress-strain curve, it is recommended to use the couples of stress strain value corresponding to stresses of 0,1 σ_m and 0,5 σ_m unless other couples are fixed by agreement between parties.

10 Test report

The test report shall contain the following information:

a) name and address of the testing establishment;

b) date of the test, unique identification of report and of each page, customer name and address and signatory;

c) reference to this International Standard, i.e. ISO 15733;

d) test piece drawing or reference;

e) description of the test material (material type, manufacturing code, batch number);

f) load rate, displacement rate;

g) number of tests carried out and the number of valid results obtained;

h) force - longitudinal deformation records;

i) valid results, mean value and standard deviations of the tensile strength, the tensile strain at maximum tensile force, the (pseudo)-elastic modulus;

j) value of correction factor applied when effective cross-section area is used, and method to obtain it;

k) failure location of all the specimens used for obtaining the above results.

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