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Fine ceramics (advanced ceramics, advanced technical ceramics) — Mechanical properties of ceramic composites at high temperature — Determination of compression properties

Céramiques techniques — Propriétés mécaniques des céramiques composites à haute température — Détermination des caractéristiques en compression

Reference number
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Contents

Page

Foreword

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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 high temperature — Determination of compression properties

1 Scope

This International Standard specifies the conditions for determination of compression properties of ceramic matrix composite materials with continuous fibre reinforcement for temperatures up to 2 000 °C.

This International 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.

Two types of compression are distinguished:

a) compression between platens;

b) compression using grips.

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

EN 10002-4, *Metallic materials — Tensile test — Part 4: Verification of extensometers used in uniaxial testing*

CEN/TS 15867:2009, *Advanced technical ceramics — Ceramic composites — Guide to the determination of the degree of misalignment in unixial mechanical tests*

IEC 60584-1:1995, *Thermocouples — Part 1: Reference tables*

IEC 60584-2:1982, *Thermocouples — Part 2: Tolerances*

IEC 60584-2:1982, *Amendment 1*:1989

3 Terms and definitions

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

3.1
test temperature
T
temperature of the test piece at the centre of the gauge length

3.2
calibrated length
l
part of the test specimen that has uniform and minimum cross-section area

3.3
gauge length
L_0
initial distance between reference points on the test specimen in the calibrated length

3.4
controlled-temperature zone
part of the calibrated length, including the gauge length, where the temperature is within a range of 50 °C of the test temperature

3.5
initial cross-section area
A_0
initial cross-section area of the test specimen within the calibrated length, at test temperature

Note 1 to entry: Two initial cross-section areas of the test specimen can be defined as follows.

3.5.1
apparent cross-section area
total area of the cross section, $A_{0,a}$

3.5.2
effective cross-section area
total area corrected by a factor, to account for the presence of an antioxidant protection, $A_{0,e}$

3.6
longitudinal deformation
ΔL
decrease in the gauge length L between reference points under a compression force

3.7
compression strain
ε
relative change in the gauge length defined as the ratio $\Delta L/L_0$

Note 1 to entry: Its value corresponding to the maximum force shall be denoted as $\varepsilon_{c,m}$.

3.8
compression stress
σ
compression force supported by the test specimen at any time in the test divided by the initial cross-section area (A_0)

Note 1 to entry: Two compression stresses can be distinguished:

— apparent compression stress, σ_a, when the apparent cross-section area (or total cross-section area) is used;

— effective compression stress, σ_e, when the effective cross-section area is used

3.9
maximum compression force
F_m
highest recorded compression force in a compression test on the test specimen when tested to failure

3.10
compression strength
$\sigma_{c,m}$
ratio of the maximum compression force ($F_{\mathbf{m}}$) to the initial cross-section area (A_o)

Note 1 to entry: Two compression strengths can be distinguished:

— apparent compression strength, $\sigma_{c,m,a}$, when the apparent cross-section area (or total cross-section area) is used;

— effective compression strength, $\sigma_{c,m,e}$, when the effective cross-section area is used.

3.11
proportionality ratio or pseudo-elastic modulus, E_p
slope of the 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 a linear section in the stress-strain curve.

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

$$Ep(\sigma_1, \sigma_2) = \frac{\sigma_2 - \sigma_1}{\varepsilon_2 - \varepsilon_2} \qquad (1)$$

where (ε_1, σ_1) and (ε_2, σ_2) lie near the lower and 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 material has a linear behaviour from the origin.

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

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

Two proportionality ratios or pseudo-elastic moduli can be distinguished:

— apparent proportionality ratio, Ep_a, when the apparent compression stress is used;

— effective proportionality ratio, Ep_e, when the effective compression stress is used.

4 Principle

A test specimen of specified dimensions is heated to the test temperature, and loaded in compression. The test is performed at constant crosshead displacement rate, or constant deformation rate. Force and longitudinal deformation are measured and recorded simultaneously.

NOTE 1 The test duration is limited to reduce creep effects.

NOTE 2 Constant loading rate is only allowed in the case of linear stress-strain behaviour up to failure.

NOTE 3 In order to protect fixtures, it is recommended to use constant crosshead displacement rate when the test is carried out until rupture.

5 Apparatus

5.1 Test machine

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

NOTE This should prevail during actual test conditions of, e.g. gas pressure and temperature.

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 performance including the alignment system and the force transmitting system shall not change because of heating.

The load train shall align the specimen axis with the direction of load application without introducing bending or torsion in the specimen. The misalignment of the specimen shall be verified and documented according to the procedure described in CEN/TS 15867:2009 The maximum percent bending shall not exceed 5 at an average strain of 500×10^{-6}.

NOTE 1 The alignment should be verified and documented in accordance with, for example, the procedure described in CEN/TS 15867:2009.

There are two alternative means of load application.

a) Compression platens are connected to the load cell and on the moving crosshead. The parallelism of these platens shall be better than 0,01 mm, in the loading area, at room temperature and they shall be perpendicular to the load direction.

NOTE 1 The use of platens is not recommended for compression testing of 1D and 2D materials with low thickness due to buckling.

NOTE 2 A compliant interlayer material between the test specimen and platens may be used for testing macroscopically inhomogeneous materials to ensure even contact pressure. This material should be chemically compatible with both test specimen and platen materials.

b) Grips are used to clamp and load the test specimen.

The grip design shall prevent the test specimen from slipping. The grips shall align the test specimen axis with that of the applied force.

NOTE 3 Conformity with this requirement should be verified and documented according to, for example, the procedure described in Reference.[1]

NOTE 4 The grips or the platens may either be in the hot zone of the furnace or outside the furnace.

NOTE 5 When grips or platens are outside the furnace, a temperature gradient exists between the centre of the specimen, which is at the prescribed temperature, and the ends that are at the same temperature as the grips or platens.

5.3 Gastight test chamber

A gastight chamber could be used in this case.

The gastight chamber shall allow proper control of the test specimen environment in the vicinity of the test specimen during the test. The installation shall be such that the variation of load due to the variation of pressure is less than 1 % of the scale of the load cell being used.

Where a gas atmosphere is used, the gas atmosphere shall be chosen depending on the material to be tested and on test temperature. The level of pressure shall be chosen depending: on the material to be tested, on temperature, on the type of gas, and on the type of extensometry.

Where a vacuum chamber is used, the level of vacuum shall not induce chemical and/or physical instabilities of the test specimen material, and of extensometer rods, when applicable.

5.4 Set-up for heating

The set-up for heating shall be constructed in such a way that the temperature gradient within the gauge length is less than 20 °C at test temperature.

5.5 Extensometer

The extensometer shall be capable of continuously recording the longitudinal deformation at test temperature.

NOTE 1 The use of an extensometer with the greatest possible gauge length is recommended.

The linearity tolerance shall be less than or equal to 0,15 % of the extensometer range used.

The extensometer shall conform to class 1 or better of EN 10002-4. Two commonly used types of extensometer are the mechanical extensometer and the electro-optical extensometer.

If a mechanical extensometer is used, the gauge length shall be the initial longitudinal distance between the two locations where the extensometer rods contact the test specimen.

The rods may be exposed to temperatures higher than the test specimen temperature. Temperature and/or environment induced structural changes in the rod material shall not affect the accuracy of deformation measurement. The material used for the rods shall be compatible with the test specimen material.

NOTE 2 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.

NOTE 3 Rod pressure onto the test specimen should be the minimum necessary to prevent slipping of the extensometer rods.

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 perpendicular 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 the test temperature and shall not modify the stress field in the specimen.

NOTE 4 The use of integral flags as parts of the test specimen geometry is not recommended because of stress concentration induced by such features.

NOTE 5 An electro-optical extensometer is not recommended in the case where it's impossible to distinguish the colour of the reference marks and the test specimen.

5.6 Temperature measurement

For temperature measurement, either thermocouples conforming to IEC 60584-1 and IEC 60584-2 shall be used or, where thermocouples not conforming to IEC 60584-1 and IEC 60584-2 or pyrometers are used, calibration data shall be annexed to the test report.

5.7 Data recording system

A calibrated recorder may be used to record the force-deformation curve. However, the use of a digital data recording system combined with an analogue recorder is recommended.

5.8 Micrometers

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

6 Test specimens

6.1 General

The choice of specimen geometry depends on several factors, such as:

— nature of the material and of the reinforcement structure;

— type of heating system;

— type of loading system.

The volume in the gauge length shall be representative of the material and calibrated length shall be chosen such as to avoid buckling failure.

NOTE A test piece volume of a minimum of 5 representative volume elements is recommended

6.2 Compression between platens

A Type 1 specimen is commonly used and is represented on Figure 1.

Recommended dimensions are given in Table 1.

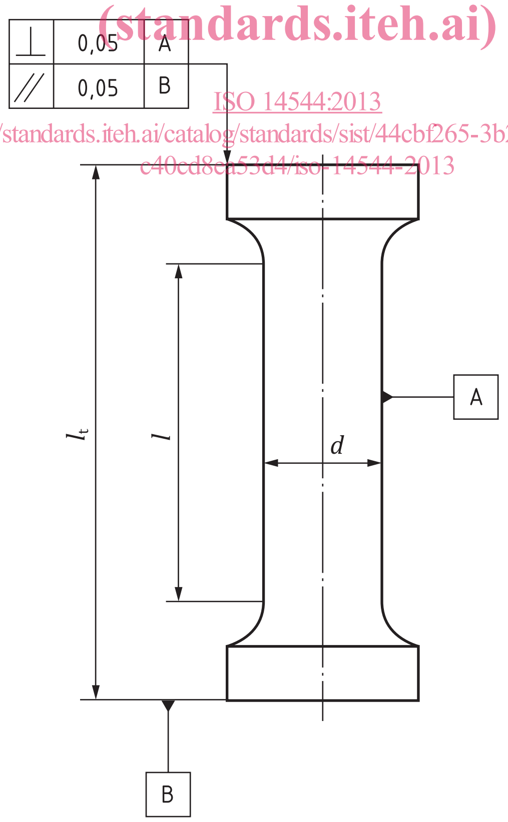

Figure 1 — Type 1 specimen geometry

Table 1 — Recommended dimensions for a Type 1 specimen

Dimensions in millimetres

	2D and *x*D	Tolerance
l, calibrated length	≥ 15	±0,5
l_t, total length	≥ 1,5 *l*	±0,5
d, circular or square section diameter or side length	≥ 8	±0,2
r, radius of shoulder	≥ 10	≥ 2
Parallelism of machined parts	0,05	
Perpendicularity of machined parts	0,05	
Concentricity of machined parts	0,05	

A Type 2 specimen is sometimes used and is represented in Figure 2.

Recommended dimensions are given in Table 2.

Figure 2 — Type 2 specimen geometry

Table 2 — Recommended dimensions for a Type 2 specimen

Dimensions in millimetres

	1D, 2D and *x*D	Tolerance
l, calibrated length	≥ 10	±0,5
d, circular or square section diameter or side length	≥ 10	±0,2
Parallelism of machined parts	0,05	
Perpendicularity of machined parts	0,05	

NOTE This specimen is mainly used when the thickness of the part is not sufficient to machine a specimen of type 1.

6.3 Test specimen used with grips

For these types of specimens, the total length l_t depends on furnace and gripping system.

A Type 3 specimen is represented in Figure 3.

Recommended dimensions are given in Tables 3 and 4.