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

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

Reference number
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Contents

Page

Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 14574 was prepared by Technical Committee ISO/TC 206, *Fine ceramics*.

Fine ceramics (advanced ceramics, advanced technical ceramics) - Mechanical properties of ceramic composites at high temperature - 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 for temperatures up to 2 000 °C.

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 International Standard is to determine the tensile properties of a material when it is placed under an oxidizing environment but not to measure material oxidation.

This International Standard applies to all ceramic matrix composites with a continuous fibre reinforcement, unidirectional (1D), bi-directional (2D), and tri-directional (xD, with $2 < x \leq 3$), loaded along one principal axis of reinforcement.

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.

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*

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

IEC 60584-2:1982+ Amendment 1:1989, *Thermocouples — Part 2: Tolerances*

3 Terms, definitions and symbols

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 controlled to within 50 °C of the test temperature

3.5
initial cross-section area
S_0
initial cross-section areas of the test specimen within the calibrated length, at test temperature

3.6
apparent cross-section area
$S_{0\ app}$
total area of the cross-section

3.7
effective cross-section area
$S_{0\ eff}$
total area corrected by a factor, to account for the presence of an anti-oxidative protection

3.8
longitudinal deformation
A
increase in the gauge length between reference points under a tensile force

3.9
longitudinal deformation under maximum tensile force
A_m
increase in the gauge length between reference points under maximum tensile force

3.10
tensile strain
ε
relative change in the gauge length defined as the ratio A/L_0

3.11
tensile strain under maximum force
ε_m
relative change in the gauge length defined as the ratio A/L_0 under the maximum force

3.12
tensile stress
σ
tensile force supported by the test specimen at any time in the test divided by the initial cross-section area (S_0)

3.13
apparent tensile stress
σ_{app}
tensile force supported by the test specimen at any time in the test divided by the apparent cross-section area (or total cross-section area)

3.14
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 ($S_{0\ eff}$)

3.15
maximum tensile force
F_m
highest recorded tensile force in a tensile test on the test specimen when tested to failure

3.16
tensile strength
σ_m
ratio of the maximum tensile force to the initial cross-section area (S_o)

3.17
apparent tensile strength
$\sigma_{m\ app}$
ratio of the maximum tensile force to the apparent cross-section area (or total cross-section area)

3.18
effective tensile strength
$\sigma_{m\ eff}$
ratio of the maximum tensile force to the effective cross-section area

3.19
proportionality ratio or pseudo-elastic modulus
EP
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:

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

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.

3.20
apparent proportionality ratio
EP_{app}
slope of the linear section of the stress-strain curve, if any, when the apparent tensile stress is used

3.21
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 heated to the test temperature, and loaded in tension. The test is performed at constant crosshead displacement rate, or constant deformation rate (or constant loading rate). Force and longitudinal deformation are measured and recorded simultaneously.

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

NOTE 2 When constant loading rate is used in the nonlinear region of the tensile curve, only the tensile strength can be obtained from the test. In this region constant crosshead displacement rate or constant deformation rate is recommended to obtain the complete curve.

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.

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

The attachment fixtures shall align the test specimen axis with the applied force direction.

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

The grip design shall prevent the test specimen from slipping.

There are two types of gripping systems:

— hot grips where the grips are in the hot zone of the furnace;

— cooled grips where the grips are outside the hot zone.

NOTE 2 The choice of gripping system will depend on material, on test specimen design and on alignment requirements.

NOTE 3 The hot grip technique is limited in temperature because of the nature and strength of the materials that can be used for grips.

NOTE 4 In the cooled grip technique, a temperature gradient exists between the centre which is at the prescribed temperature and the ends which are at the same temperature as the grips.

5.3 Test chamber

The test chamber shall be gastight and 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 the 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 gauge length is recommended.

The linearity tolerances shall be lower than 0,05 % of the extensometer range used.

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 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 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 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 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 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 devices

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 force-deformation curve. 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:

— nature of the material and of the reinforcement structure;

— type of heating system;

— type of gripping system.

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

NOTE 1 The total length *L* depends on furnace and gripping system. Generally, *L* is greater than 150 mm.

NOTE 2 A test piece volume of a minimum of 5 representative volume elements is recommended

6.2 Test specimens commonly used

Several types of test specimens may be used, as indicated in Figures 1 to 6 and Tables 1 to 6.

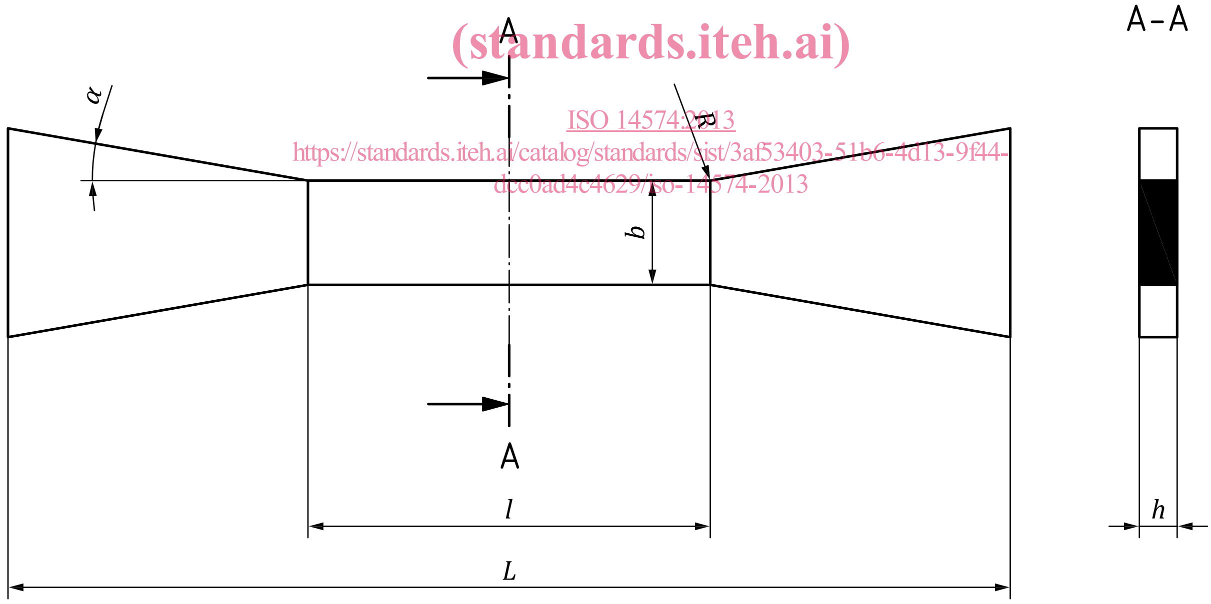

Figure 1 — Type 1 test specimen

Table 1 — Recommended dimensions for a Type 1 test specimen

Parameter	2D and *x*D	Tolerance
l, calibrated length	30 mm to 80 mm	± 0,5 mm
h, thickness	> 2 mm	± 0,2 mm
α, angle	10°to 30°	-
b, width of the calibrated length	8 mm to 20 mm	± 0,2 mm
R, radius	> 30 mm	± 2 mm
Parallelism of machined parts	0,05	-

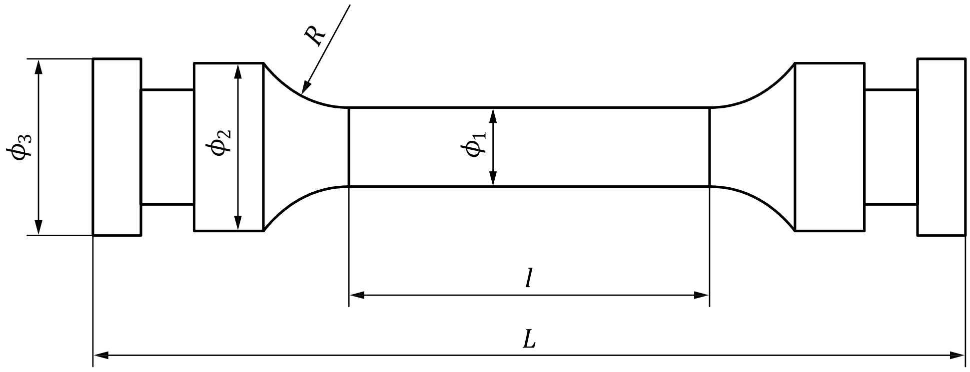

Figure 2 — Type 2 test specimen

Table 2 — Recommended dimensions for a Type 2 test specimen

Dimensions in millimetres

Parameter	*x*D	Tolerance
l, calibrated length	30 to 80	± 0,5
φ_1, diameter in calibrated length	8 to 20	± 0,2
φ_2, diameter	$\varphi_2 = \alpha\varphi_1$ (α = 1,2 to 2)	± 0,2
φ_3, diameter	$\varphi_3 = \beta\varphi_2$ (β = 1,2 to 2)	± 0,2
R, radius	> 30	± 2
Parallelism of machined parts	0,05	-