
**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*



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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*.

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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

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

3.7

effective cross-section area

$S_{0\text{ eff}}$
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 ($S_{0\text{ eff}}$)

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_0)**3.17****apparent tensile strength** $\sigma_{m \text{ app}}$

ratio of the maximum tensile force to the apparent cross-section area (or total cross-section area)

3.18**effective tensile strength** $\sigma_{m \text{ eff}}$

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 $(\varepsilon_1, \sigma_1)$ and $(\varepsilon_2, \sigma_2)$ 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 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 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](#).

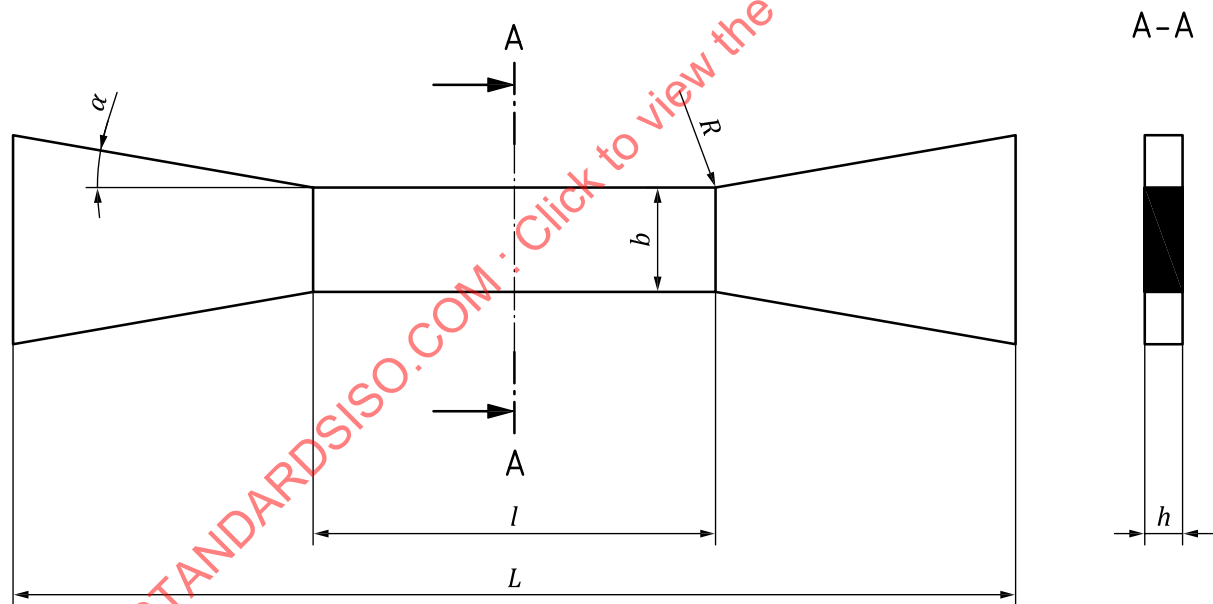
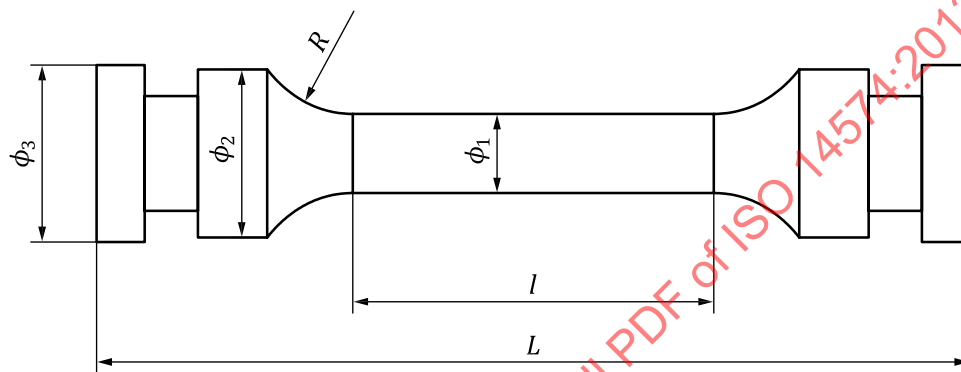


Figure 1 — Type 1 test specimen

Table 1 — Recommended dimensions for a Type 1 test specimen

Parameter	2D and xD	Tolerance
l , calibrated length	30 mm to 80 mm	$\pm 0,5$ mm
h , thickness	> 2 mm	$\pm 0,2$ mm
α , angle	10° to 30°	-
b , width of the calibrated length	8 mm to 20 mm	$\pm 0,2$ 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	xD	Tolerance
l , calibrated length	30 to 80	$\pm 0,5$
ϕ_1 , diameter in calibrated length	8 to 20	$\pm 0,2$
ϕ_2 , diameter	$\phi_2 = \alpha \phi_1$ ($\alpha = 1,2$ to 2)	$\pm 0,2$
ϕ_3 , diameter	$\phi_3 = \beta \phi_2$ ($\beta = 1,2$ to 2)	$\pm 0,2$
R , radius	> 30	± 2
Parallelism of machined parts	0,05	-

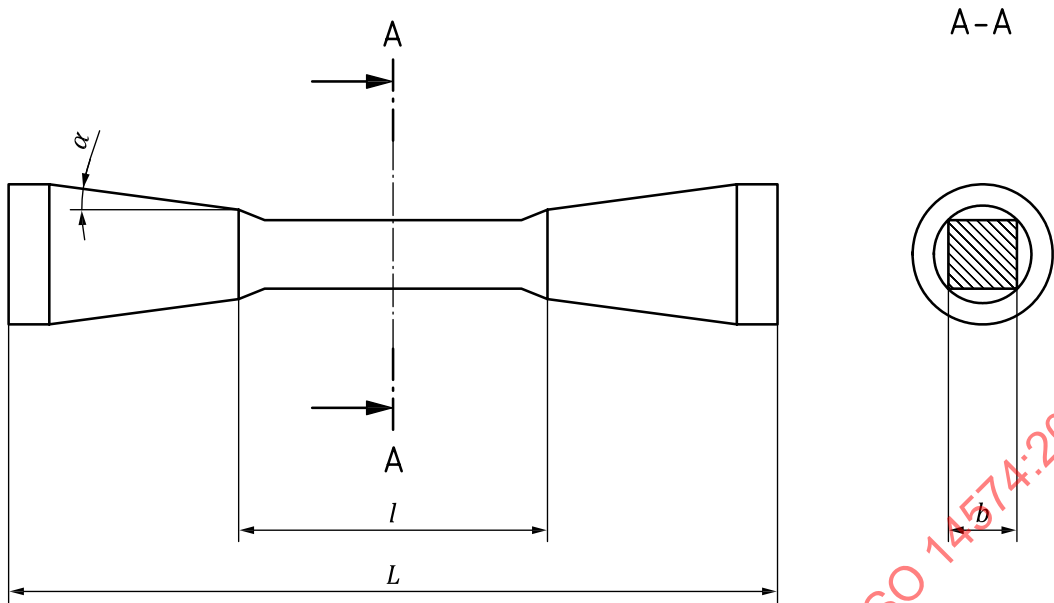


Figure 3 — Type 3 test specimen

Table 3 — Recommended dimensions for a Type 3 test specimen

Parameter	xD	Tolerance
<i>l</i> , calibrated length	30 mm to 80 mm	± 0,5 mm
<i>b</i> , width and thickness in the calibrated length	3 mm to 8 mm	± 0,2 mm
α , angle	7° to 30°	± 2°
Parallelism of machined parts	0,05 mm	-

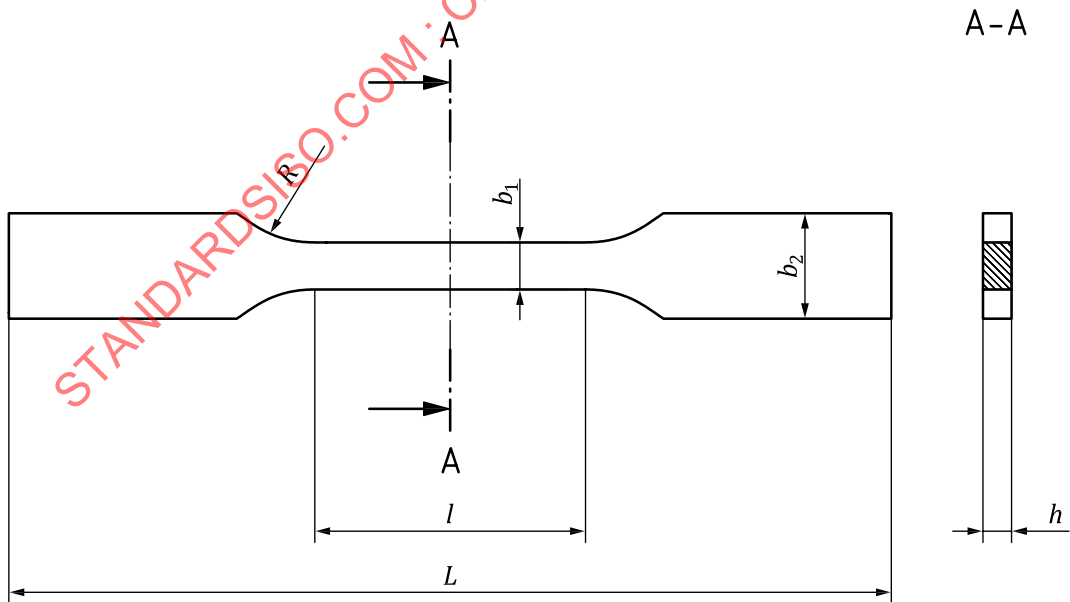


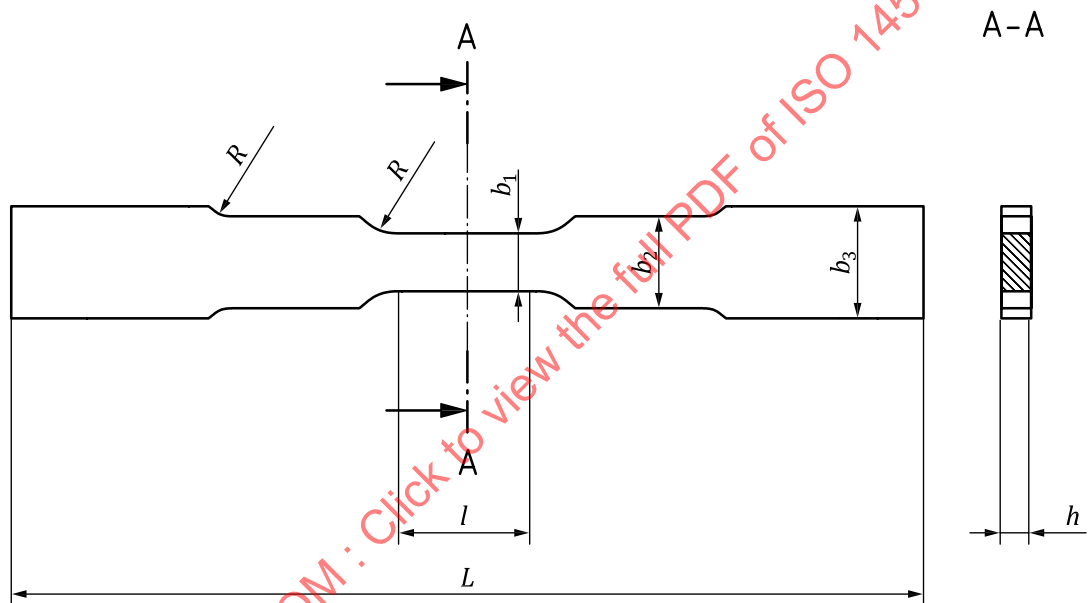
Figure 4 — Type 4 test specimen

Table 4 — Recommended dimensions for a Type 4 test specimen

Dimensions in millimetres

Parameter	2D and xD	Tolerance
l , calibrated length	30 to 80	$\pm 0,5$
h , thickness	> 2	$\pm 0,2$
b_1 , width in the calibrated length	8 to 20	$\pm 0,2$
b_2 , width	$b_2 = \alpha b_1$ with $\alpha = 1,2$ to 2	$\pm 0,2$
R , radius	> 30	± 2
Parallelism of machined parts	0,05	-

Variation of material properties with temperature may lead to a multi-section test specimen (Type 5) as represented in [Figure 5](#). Recommended dimensions are given in [Table 5](#).

**Figure 5 — Type 5 test specimen****Table 5 — Recommended dimensions for a Type 5 test specimen**

Dimensions in millimetres

Parameter	2D and xD	Tolerance
l , calibrated length	30 to 80	$\pm 0,5$
h , thickness	> 2	$\pm 0,2$
b_1 , width in the calibrated length	8 to 20	$\pm 0,2$
b_2 , width	$b_2 = \alpha b_1$ with $\alpha = 1,2$ to 2	$\pm 0,2$
b_3 , width	$b_3 = \beta b_2$ with $\beta = 1,2$ to 2	$\pm 0,2$
R , radius	> 30	± 2
Parallelism of machined parts	0,05	-

NOTE 1 When used with cooled grips, this multi-section specimen allows rupture in the controlled temperature zone.

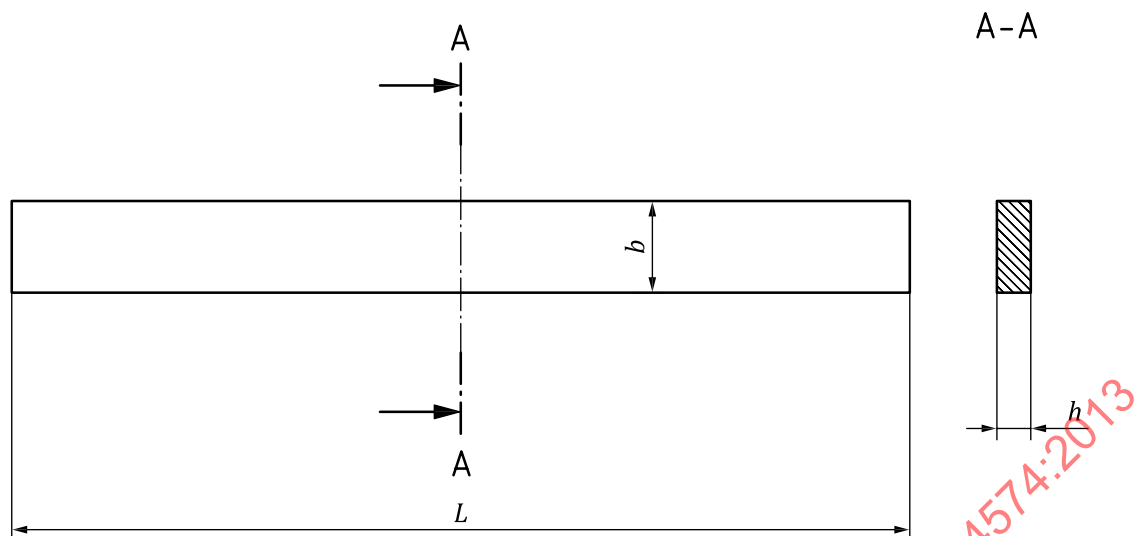


Figure 6 — Type 6 test specimen

NOTE 2 This test specimen is easy to machine and its use allows mainly the determination of modulus, as rupture may not happen all the time in the controlled temperature zone; it should not be used for strength measurement.

NOTE 3 This kind of specimen cannot be used for cold gripping systems.

Table 6 — Recommended dimensions for a Type 6 test specimen

Dimensions in millimetres

Parameter	1D, 2D and xD	Tolerance
h , thickness	> 2	$\pm 0,2$
b , width	8 to 20	$\pm 0,2$
Parallelism of machined parts	0,05	-

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 methods that do not cause damage to material are recommended. Machining parameters should be traceable.

These parameters shall be adhered to during test specimen preparation.

NOTE 1 When specimens are machined from a plate that has been protected against oxidation, the cut surfaces of the specimen are unprotected. These surfaces should be protected to prevent possible oxidation.

NOTE 2 When a cooled gripping system is used, the surface of the part of specimen that is at a temperature between the test temperature and the grips temperature may need appropriate anti-oxidant protection.

7.2 Number of tests of specimens

At least five valid test results, as specified in 8.4, are required for any condition.

8 Test procedures

8.1 Test set-up: Temperature considerations

8.1.1 General

The following determinations shall be carried out under conditions representative of the tests, and shall be repeated every time there is a change, e.g. in material, in specimen geometry, in gripping configuration. In establishing them, time shall be allowed for temperature stabilization.

8.1.2 Controlled temperature zone

Prior to testing, the temperature gradient within the calibrated length inside the furnace 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 extensometer reference points and midway between the two.

To establish the length of the controlled temperature zone, it is necessary to measure temperature also outside the gauge length. The temperature variation in the gauge length shall be within 20 °C at test temperature. The temperature variation in the part of the calibrated length, including the gauge length, shall be within 50 °C of the test temperature.

Temperatures shall be measured in accordance with 5.5. 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.

8.1.3 Temperature calibration

During a series of tests, the test temperature may 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, calibration will be necessary. The relationship between the control temperature and test specimen temperature at the centre of the gauge length shall be established beforehand on a dummy test specimen over the range of temperature of interest.

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

8.2 Test set-up: Other considerations

8.2.1 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. If the material to be tested is sensitive to creep at the temperature of test, the speed shall be significantly increased but impact loading shall be avoided.

8.2.2 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.

The cross-section area varies with temperature and the variation is very difficult to measure, for this reason, cross-section area is measured at room temperature.

Dimensions shall be measured to an accuracy of $\pm 0,01$ 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 %. If thermal expansion between room temperature and the test

temperature is less than the tolerance on the gauge length measurement, then the gauge length may be measured at room temperature. If this is not the case, the gauge length shall be corrected to take the thermal expansion into account, or shall be measured at the test temperature.

8.3 Testing technique

8.3.1 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.

In some cases, it is necessary to apply a preload during the whole heating period to prevent the alignment from being lost. The preload shall not increase beyond 5 % of the expected failure load at any moment.

8.3.2 Setting of extensometer

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

Where a contacting extensometer is positioned at ambient temperature, the extensometer output shall be adjusted to read zero after the stabilization period at test temperature.

NOTE Where the material has a high thermal expansion coefficient, the extensometer taking expansion into account in order to be close to zero when at test temperature should be mechanically pre-set.

8.3.3 Setting of inert atmosphere

When testing in an inert gas, any air or water vapour shall be removed before setting the inert atmosphere.

This can be achieved by establishing a vacuum (<10 Pa) in the enclosure, or by circulating inert gas.

When testing under vacuum, the vacuum level shall be in accordance with [5.3](#).

8.3.4 Heating of test specimen

The test specimen temperature shall be raised to the required test temperature, and this temperature shall be maintained for a period to allow for temperature stabilization and when applicable, for stabilization of the extensometer readout.

Two ways are possible.

- If the test specimen temperature is measured during the test on the specimen itself, this temperature shall be used to control the furnace.
- If it is not possible to directly measure the test specimen temperature during the test, then it is necessary to use the relationship between test specimen temperature and furnace temperature that has been established in [8.1](#).

Ensure that the test specimen stays in the initial state of stress during heating.

8.3.5 Measurements

Zero the load cell.

Zero the extensometer.

Record the force versus longitudinal deformation.

Register the temperature.

Load the test specimen.

If any, cool down under inert atmosphere down to a temperature where there is no more risk of material degradation of specimen and equipment before opening the test chamber.

8.4 Test validity

The following circumstances invalidate a test:

- failure to specify and record test conditions;
- specimen slippage;
- extensometer slippage;
- rupture in an area outside of the controlled temperature zone;
- change in atmosphere.

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:

$$\sigma_{mapp} = \frac{F_m}{S_{oapp}} \quad (1)$$

$$\sigma_{meff} = \frac{F_m}{S_{oeff}} \quad (2)$$

where

σ_{mapp} is the tensile strength at test temperature, T using the apparent area S_{oapp} , in megapascals (MPa);

σ_{meff} is the tensile strength at test temperature T using the effective area S_{oeff} , in megapascal (MPa);

F_m is the maximum tensile force in newtons (N);

S_{oapp} is the apparent cross-section area of the specimen in square millimetres (mm²);

S_{oeff} is the effective cross-section area of the specimen corrected to take account of the oxidative protection in square millimetres (mm²).

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