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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

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Foreword

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The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2. www.iso.org/directives

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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_o
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_o
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, $A_{o,a}$

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

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 $\Delta L/L_o$

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_o)

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:

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

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.

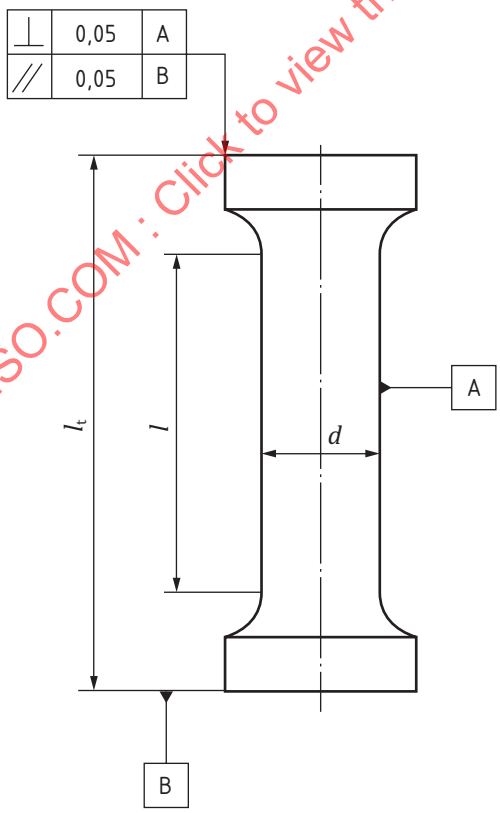

Figure 1 — Type 1 specimen geometry

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.

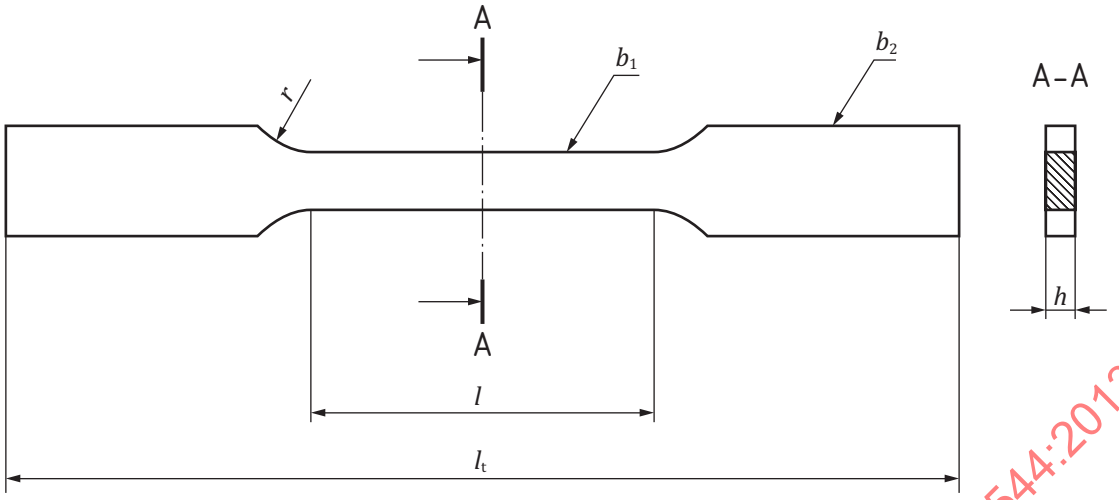

Figure 3 — Type 3 specimen geometry

Table 3 — Recommended dimensions for a Type 3 specimen

Dimensions in millimetres

	2D and xD	Tolerance
l, calibrated length	≥ 15	±0,5
h, thickness	≥ 2	±0,2
b_1, width in the calibrated length	≥ 8	±0,2
b_2, width	$b_2 = \alpha b_1$ with α = 1,2 to 2	±0,2
r, radius of shoulder	≥ 30	±2
Parallelism of machined parts	0,05	

Table 4 — Alternative recommended dimensions for a Type 3 specimen

Dimensions in millimetres

	2D and xD	Tolerance
l, calibrated length	≤ 15	±0,5
h, thickness	≥ 1	±0,2
b_1, width in the calibrated length	≥ 8	±0,2
b_2, width	$b_2 = \alpha b_1$ with α = 1,2 to 2	±0,2
r, radius of shoulder	≥ 30	±2
Parallelism of machined parts	0,05	

NOTE This type of specimen is recommended in the case of buckling with the specimen of Table 3. With this type of specimen, it is very difficult to obtain strain measurements.

A Type 4 is sometimes used and is represented in Figure 4.

Recommended dimensions are given in Table 5.

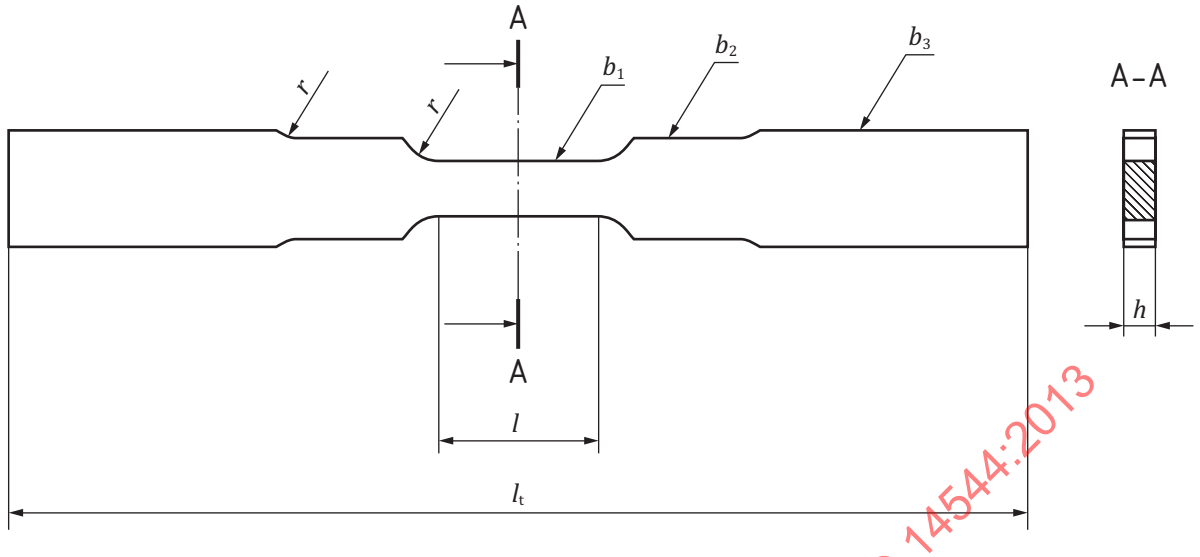

Figure 4 — Type 4 specimen geometry

Table 5 — Recommended dimensions for a Type 4 specimen

Dimensions in millimetres

	2D and *x*D	Tolerance
l, calibrated length	≥ 15	±0,2
h, thickness	3	±0,2
b_1, width in the calibrated length	8 to 20	±0,2
b_2, width	$b_2 = \alpha b_1$ with α = 1,2 to 2	±0,2
b_3, width	$b_3 = \beta b_2$ with β = 1,2 to 2	±0,2
r, radius of shoulder	≥ 30	±2
Parallelism of machined parts	0,05	

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

A Type 5 is sometimes used and is represented in Figure 5.

Recommended dimensions are given in Table 6.

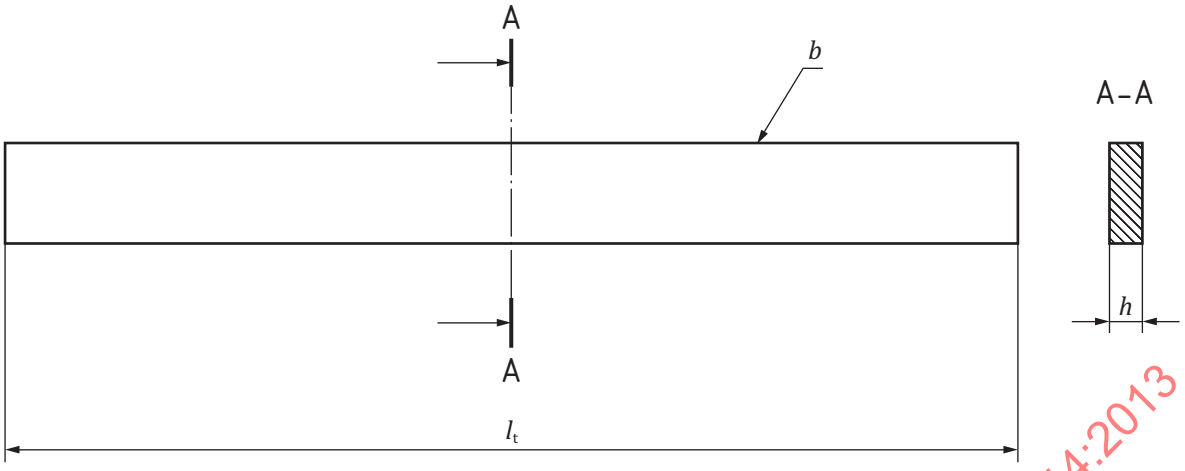

Figure 5 — Type 5 specimen geometry

Table 6 — Recommended dimensions for a Type 5 specimen

Dimensions in millimetres

	1D, 2D and xD	Tolerance
h, thickness	≤ 2	±0,2
b, width	8 to 20	±0,2
Parallelism of machined parts	0,05	

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

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.

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

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

7.2 Number of test 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 in material, specimen geometry, gripping configuration, etc. 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 in the controlled-temperature zone shall be within 50 °C of the test temperature.

Temperatures shall be measured in accordance with 5.6. 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-specimen dimensions

The cross-section area is determined at the centre of the specimen and at each end of the calibrated length. The arithmetic means of the measurements shall be used for calculations.

The necessary dimensions to calculate cross-section area are measured with an accuracy of ± 0,01 mm.

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.

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 room temperature.

8.2.3 Buckling

During a compression test, the test specimen may be subjected to buckling. To be sure of the validity of the test, it is necessary to verify that no buckling occurs in the conditions of the test.

One possibility is to place two extensometers on opposite sides of the specimen during the test.

Difference in extensometer responses will indicate buckling. If it is not possible to place two extensometers on the specimen under test conditions, then one of the procedures described in Annex A shall be used. Absence of buckling shall be verified every time there is a change in material or specimen geometry.

8.3 Testing technique

8.3.1 Specimen mounting

Install the test specimen in the gripping system or loading 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 the test temperature.

NOTE Where the material has a high thermal expansion coefficient, it is recommended to mechanically preset the extensometer taking expansion into account in order to be close to zero when at test temperature.

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 gas.

When testing under vacuum, the vacuum level shall be in accordance with 5.3.

8.3.4 Heating of test specimen

Raise the test specimen temperature to the required test temperature, and maintain this temperature 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 measure the test specimen temperature directly during the test, then it is necessary to use the relationship between the test specimen temperature and furnace temperature which 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 temperature, environment conditions (gas and pressure).

Load the test specimen.

If any, before opening the test chamber, cool down under inert atmosphere down to a temperature at which there is no further risk of material degradation.

Note the position of fracture location relative to the mid-point of the specimen to the nearest of 1 mm.

8.4 Test validity

The following circumstances invalidate a test:

— failure to specify and record test conditions;

— failure to meet specified test conditions;

— specimen slippage;

— extensometer slippage;

— rupture in an area outside of the controlled-temperature zone;

— buckling of the specimen.

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 Compression strength

Calculate the compression strength using one of the following equations:

$$\sigma_{c,m,a} = \frac{F_m}{A_{o,a}} \tag{2}$$

$$\sigma_{c,m,e} = \frac{F_m}{A_{o,e}} \quad (3)$$

where

$\sigma_{c,m,a}$ is the compression strength at temperature T, using the apparent area $A_{o,a}$, in megapascals (MPa);

$\sigma_{c,m,e}$ is the compression strength at temperature T, using the effective area $A_{o,e}$, in megapascals (MPa);

F_m is the maximum compression force, in newtons (N);

$A_{o,a}$ is the apparent cross-section area of the specimen, in square millimetres (mm^2);

$A_{o,e}$ is the effective cross-section area of the specimen corrected to take account of the oxidative protection, in square millimetres (mm^2).

9.3 Strain at maximum compression force

Calculate the strain using the following equation:

$$\varepsilon_{c,m} = \frac{\Delta L_{c,m}}{L_O} \quad (4)$$

where

$\varepsilon_{c,m}$ is the strain at the maximum compression force;

$\Delta L_{c,m}$ is the longitudinal deformation at the maximum compression force in millimetres (mm), measured by the extensometer;

L_o is the gauge length, in millimetres (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 $(\Delta L_1, F_1)$ and $(\Delta L_2, F_2)$ measured near the lower and upper limits of the linear part of the force-deformation record, according to the following equations:

$$Ep_a(\sigma_1, \sigma_2) = \frac{L_o(F_2 - F_1)}{A_{o,a}(\Delta L_2 - \Delta L_1)} \times 10^{-3} \quad (5)$$

$$Ep_e(\sigma_1,\sigma_2)=\frac{L_o(F_2-F_1)}{A_{o,a}(\Delta L_2-\Delta L_1)}\times 10^{-3} \quad (6)$$

where

Ep_a is the apparent pseudo-elastic modulus, in gigapascals (GPa);

Ep_e is the effective pseudo-elastic modulus, in gigapascals (GPa);

F is the compression force acting on the specimen, in newtons (N);

$A_{o,a}$ is the apparent cross-section area of the specimen in square millimetres (mm2);

$A_{o,e}$ is the effective cross-section area of the specimen corrected to take account of the oxidation protection, in square millimetres (mm^2);

L_o is the gauge length at temperature T, in millimetres (mm);

ΔL is the longitudinal deformation, in millimetres (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 equation:

$$E_a=\frac{FL_o}{A_{o,a}\Delta L}\times 10^{-3} \quad (7)$$

$$E_e=\frac{FL_o}{A_{o,e}\Delta L}\times 10^{-3} \quad (8)$$

where

E_a is the apparent compression elastic modulus, in gigapascals (GPa);

E_e is the effective compression elastic modulus, in gigapascals (GPa);

F is the compression force acting on the specimen, in newtons (N);

$A_{o,a}$ is the apparent cross-section area of the specimen, in square millimetres (mm^2);

$A_{o,e}$ is the effective cross-section area of the specimen corrected to take account of the oxidation protection, in square millimetres (mm^2);

L_o is the gauge length at temperature T, in millimetres (mm);

ΔL is the longitudinal deformation, in millimetres (mm), measured on the curve corresponding to F.

Any point (ΔL,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 values corresponding to stresses of $0{,}1\sigma_{c,m}$ and $0{,}5\sigma_{c,m}$ unless other couples are fixed by agreement between parties.