International Standard

ISO 14544

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

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

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Foreword

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This document was prepared by Technical Committee ISO/TC 206, *Fine ceramics,* in collaboration with the European Committee for Standardization (CEN) Technical Committee CEN/TC 184, *Advanced technical ceramics,* in accordance with the Agreement on technical cooperation between ISO and CEN (Vienna Agreement).

This second edition cancels and replaces the first edition (ISO 14544:2013), which has been technically revised.

The main changes are as follows:

— alignment of the terms and definition with the vocabulary standard ISO 20507;

— addition of illustration of compressive modulus in Annex A;

— addition of a calibration method of the test temperature by using a cartographic specimen equipped with thermocouples in Annex B.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

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

1 Scope

This document specifies procedures for determination of the compressive behaviour of ceramic matrix composite materials with continuous fibre reinforcement at elevated temperature in air, vacuum and inert gas atmospheres. This document applies to all ceramic matrix composites with a continuous fibre reinforcement, uni-directional (1D), bidirectional (2D) and multi-directional (xD, with $x > 2$), tested along one principal axis of reinforcement or off axis conditions for 2D and xD materials. This document also applies to carbon-fibre-reinforced carbon matrix composites (also known as carbon/carbon or C/C). Two cases of testing are distinguished: compression between platens and compression using grips.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements 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 — Design and metrological characteristics of micrometers for external measurements*

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

ISO 9513, *Metallic materials — Calibration of extensometer systems used in uniaxial testing*

ISO 19634, *Fine ceramics (advanced ceramics, advanced technical ceramics) — Ceramic composites — Notations and symbols*

ISO 20507, *Fine ceramics (advanced ceramics, advanced technical ceramics) — Vocabulary*

IEC 60584-1, *Thermocouples — Part 1: EMF specifications and tolerances*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 20507 and ISO 19634 and the following apply.

ISO and IEC maintain terminology databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at https://www.electropedia.org/

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

[SOURCE: ISO 20504:2022, 3.1]

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

[SOURCE: ISO 20504:2022, 3.2, modified — term and definition, words before initiation of the test deleted.]

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
S_o
cross-section area of the test specimen within the calibrated length, at room temperature before testing

3.5.1
apparent cross-section area
$S_{o\ app}$
area of the cross section

3.5.2
effective cross-section area
$S_{o\ eff}$
area corrected by a factor, to account for the presence of a coating

3.6
longitudinal deformation
A
decrease in the gauge length under a compressive force in the load direction

Note 1 to entry: The longitudinal deformation corresponding to the maximum compressive force is denoted as $A_{c,m}$.

3.7
compressive strain
ε
ratio of deformation to initial gauge length defined as the ratio A/L_0

Note 1 to entry: The compressive strain corresponding to the maximum compressive force is denoted as $\varepsilon_{c,m}$.

3.8
compressive force
F_c
uniaxial force carried by the test specimen at any time during the compression test

[SOURCE: ISO 20504:2022, 3.6, modified — word "compression" added.]

3.9
compressive stress
σ
compressive force (3.8) supported by the test specimen at any time in the test divided by the *initial cross-sectional area* (3.5) such that $\sigma = F_c/S_o$

[SOURCE: ISO 20504:2022, 3.8]

3.9.1
apparent compressive stress
σ_{app}
ratio of the *compressive force* (3.8) supported by the test piece to the *apparent cross-section area* (3.5.1)

3.9.2
effective compressive stress
σ_{eff}
ratio of the *compressive force* (3.8) carried by the test piece to the *effective cross-section area* (3.5.2)

3.10
maximum compressive force
$F_{c,m}$
highest force recorded or force at failure during a compressive test

3.11
compressive strength
$\sigma_{c,m}$
greatest *compressive stress* (3.9) applied to a test specimen when tested to failure

[SOURCE: ISO 20504:2022, 3.9]

3.11.1
apparent compressive strength
$\sigma_{c,m\ app}$
ratio of the *maximum compressive force* (3.10) to the *apparent cross-section area* (3.5.1)

3.11.2
effective compressive strength
$\sigma_{c,m\ eff}$
ratio of the *maximum compressive force* (3.10) to the *effective cross-section area* (3.5.2)

3.12
compressive modulus
E
slope of the linear section of the stress-strain curve at or near the origin

Note 1 to entry: It is possible that a linear part does not exist or does not start at the origin. The different situations are then described in the Annex A.

3.12.1
apparent compressive modulus
E_{app}
slope of the linear part of the stress-strain curve at or near the origin when the *apparent compressive stress* (3.9.1) is used

3.12.2
effective compressive modulus
E_{eff}
slope of the linear part of the stress-strain curve at or near the origin, when the *effective compressive stress* (3.9.2) 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 (or constant loading rate). Force and longitudinal deformation are measured and recorded simultaneously.

NOTE The test duration is limited to reduce creep effects.

When constant loading rate is used in the nonlinear region of the compressive curve, only the compressive 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 machine shall be equipped with a system for measuring the force applied to the test specimen that shall conform to grade 1 or better in accordance with ISO 7500-1. 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 at room temperature and documented. Several standards address this topic but it is recommended to comply with the procedure described in ISO 17161. The percent bending strain shall not exceed 5 % at an average strain of 500 x 10^{-6}.

There are two alternative means of load application:

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

   The use of platens is not recommended for compression testing of 1D and 2D materials with low thickness due to buckling. For high temperature tests set-up, the platens parallelism value specified in ISO 20504 is sometimes difficult to be determined by dimensional controls but remains a suitable recommendation.

   A compliant interlayer material between the test specimen and platens can 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.

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

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

NOTE 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

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 extensometer.

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. Primary vacuum (typically 1 Pa pressure) is recommended.

5.4 Set-up for heating

The set-up for heating shall be constructed in such a way that:

— the test coupon maximal temperature will never exceed the desired test temperature by more than 5 °C;

— the gauge length is actually included in the controlled temperature zone.

NOTE 1 When tests are performed in vacuum or inert gas atmospheres, this maximal temperature gradient of 50 °C in the controlled temperature zone is considered to be low enough to avoid large discrepancy of material behaviour in the gauge length and then to bias the material properties determination.

NOTE 2 This value of 50 °C is a maximum value of the temperature gradient of the controlled temperature zone especially for very high temperature test in cold grip configuration. If tests are performed at lower temperature, temperature gradient lower than 50 °C can be easily achieved.

If the tests are performed under oxidative environment, for CMC materials which are sensitive to oxidative degradation, the test duration and the controlled temperature zone thermal gradient parameters are to be set at the lowest values possible in order to limit the impact on the material properties of the oxidative degradation. For instance, for material such as CMC including a carbon interphase which are sensitive to chemical degradation, it is recommended to not exceed ±5 °C below 500 °C for the temperature gradient within the controlled temperature zone.

NOTE 3 An example of calibration method of test temperature and temperature gradient determination is described in the Annex B.

5.5 Strain measurement

5.5.1 General

For continuous measurement of the longitudinal deformation as a function of the applied force at high temperature, either suitable contacting or non-contacting extensometer may be used. Measurement of longitudinal deformation over a length as long as possible within the controlled-temperature zone of the test specimen is recommended.

5.5.2 Strain gauges

Strain gauges are used for the verification of the alignment on the test specimen at room temperature. They are not recommended to determine longitudinal deformation during testing at high temperature.

5.5.3 Extensometer

5.5.3.1 General

The extensometer shall be capable of continuously recording the longitudinal deformation at test temperature. The use of an extensometer with the greatest possible gauge length is preferable.

Extensometers shall meet the requirements of class 1 or less (class 0,5) in accordance with ISO 9513. Types of commonly used extensometers are described in 5.5.3.2 and 5.5.3.3.