
**Fine ceramics (advanced ceramics,
advanced technical ceramics) —
Mechanical properties of ceramic
composites at ambient temperature
in air atmospheric pressure —
Determination of tensile properties
of tubes**

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*Céramiques techniques — Propriétés mécaniques des composites
céramiques à température ambiante et à pression atmosphérique —
Détermination des propriétés en traction de tubes*

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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.

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 (see www.iso.org/directives).

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Fine ceramics (advanced ceramics, advanced technical ceramics) — Mechanical properties of ceramic composites at ambient temperature in air atmospheric pressure — Determination of tensile properties of tubes

1 Scope

This document specifies the conditions for the determination of tensile properties of ceramic matrix composite tubes with continuous fibre-reinforcement at ambient temperature in air atmospheric pressure. This document is specific to the tubular geometries since fibre architecture and specimen geometry factors are distinctly different in composite tubes than in flat specimens.

This document provides information on the uniaxial tensile properties and tensile stress-strain response such as tensile strength and strain, tensile elastic modulus and Poisson's ratio. The information may be used for material development, control of manufacturing (quality insurance), material comparison, characterization, reliability and design data generation for tubular components.

This document addresses, but is not restricted to, various suggested test piece fabrication methods. It applies primarily to ceramic and/or glass matrix composite tubes with a continuous fibrous-reinforcement: unidirectional (1D filament winding and tape lay-up), bi-directional (2D braid and weave) and tri-directional (xD, with $2 < x < 3$), loaded along the tube axis.

Values expressed in this document are in accordance with the International System of Units (SI).

NOTE In most cases, ceramic matrix composites to be used at high temperature in air are coated with an antioxidation coating.

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

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 17161, *Fine ceramics (advanced ceramics, advanced technical ceramics) — Ceramic composites — Determination of the degree of misalignment in uniaxial mechanical tests*

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

ISO 3611, *Geometrical product specifications (GPS) — Dimensional measuring equipment: Micrometers for external measurements — Design and metrological characteristics*

ASTM E2208-02, *Standard Guide for Evaluating Non-Contacting Optical Strain Measurement Systems*

3 Terms and definitions

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

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

- ISO Online browsing platform: available at <https://www.iso.org/obp>
- IEC Electropedia: available at <http://www.electropedia.org/>

**3.1
calibrated length**

l
part of the test specimen that has uniform and minimum external diameter

**3.2
gauge length**

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

**3.3
initial cross-section area**

S_0
area of the test specimen within the calibrated length

**3.4
effective cross-section area**

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

**3.5
external diameter**

d_o
outer distance through the centre of a tube from one side to the other

**3.6
internal diameter**

d_i
inner distance through the centre of a tube from one side to the other

**3.7
longitudinal deformation**

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

**3.8
longitudinal deformation under maximum tensile force**

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

**3.9
tensile strain**

ε_{zz}
relative change in the gauge length defined as the ratio A/L_0

**3.10
tensile strain under maximum force**

$\varepsilon_{zz,m}$
relative change in the gauge length defined as the ratio A_m/L_0

**3.11
circumferential strain**

$\varepsilon_{\theta\theta}$
relative change in circumferential direction in the gauge length

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

3.14 maximum tensile force

 F_m

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

3.15 tensile strength

 σ_m

ratio of the maximum tensile force to the initial cross-section area (S_0)

3.16 effective tensile strength

 $\sigma_{m,\text{eff}}$

ratio of the maximum tensile force to the effective cross-section area ($S_{0,\text{eff}}$)

3.17 proportionality ratio pseudo-elastic modulus

 E_p

slope of the initial linear section of the stress-strain curve

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Note 1 to entry: Examination of the stress-strain curves for ceramic matrix composites allows definition of the following cases:

- a) Material with an initial linear domain in 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.

3.18 effective proportionality ratio effective pseudo-elastic modulus

 $E_{p,\text{eff}}$

slope of the linear section of the stress-strain curve, if any, when the effective tensile stress is used

3.19 Poisson's ratio

 $\nu_{\theta z}$

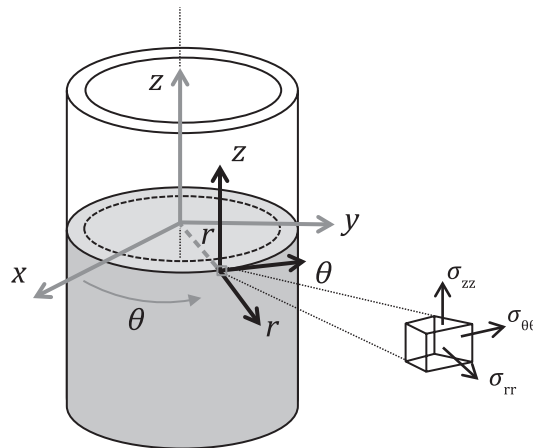
negative ratio of circumferential to axial strain

3.20 coordinate system

system used to determine location in space

Note 1 to entry: Cylindrical coordinates are adopted in the present document.

Note 2 to entry: The notations shown in [Figure 1](#) apply for space representation.



Key

- z axial
- r radial
- theta circumferential

Figure 1 — Cylindrical coordinate system used for the CMC tubes

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4 Principle

A prepared tubular test specimen of specified dimensions is loaded in monotonic uniaxial tension up to fracture. The test is performed at constant cross-head displacement, or constant strain (or constant loading rate). Both the applied force and resulting longitudinal strain are measured and recorded simultaneously. The uniaxial tensile strength and strain are determined from the maximum applied force, while the various other tensile properties are determined from the stress-strain response data.

Generally, the test is carried out under conditions of ambient temperature and environment.

NOTE 1 In uniaxial loading, the force is applied parallel to the tube axis. Monotonic refers to a continuous nonstop test rate with no reversals from test initiation to final fracture.

NOTE 2 The use of constant loading rate only gives a valid tensile curve when the behaviour is linear up to failure.

5 Apparatus

5.1 Test machine.

The test machine shall be equipped with a system for measuring the force applied to the tubular test specimen conforming to grade 1 or better in accordance with ISO 7500-1.

5.2 Test specimen gripping.

Various types of gripping device may be used to transmit the measured force applied by the testing machine to the tubular test specimen. It shall prevent the tubular test specimen from slipping.

The brittle nature of the matrices of continuous fibre ceramic composites (CFCCs) requires a uniform and continuous contact between the grip components and the gripped section of the tubular test specimen in order to minimize crack initiation and fracture in this area.

Gripping devices can be generally classified as those employing active grip interfaces and those employing passive grip interfaces that include gripping system with adhesive bonding or through a pin-loaded fixture. Examples, descriptions and designs for both the gripping types are discussed in [Annex A](#).

If an active grip interface system is used, the length of the grip section shall be long enough to develop sufficient friction force to transmit the tensile forces to the tubular test specimen. As a general rule, grip lengths are defined at least 1,5 times higher than the external diameter of the specimen. If the tubular test specimen is pulling out of the grips, thus failing to increase the clamping pressure, longer grip lengths might be needed.

To prevent crushing of the tubular test specimen by the lateral pressure and subsequent collapse of the tube wall, it is advisable to insert an end plug into the interior of the grip section of the tube specimen or to provide a suitable geometry for end collars (see [6.3](#)).

5.3 Load train couplers.

Various types of devices may be used to fix the active or passive grip interface assemblies to the testing machine. The load train couplers in conjunction with the type of gripping device play major roles in the alignment of the load train and extraneous bending imposed in the tubular test specimen; they can be generally classified as fixed and non-fixed and are discussed in [Annex A](#).

If each system type can be used, the load train configuration shall ensure that the load indicated by the load cell and the load experienced by the tubular test specimen are the same. The alignment shall be checked and documented in accordance with, for example, the procedure described in ISO 17161 adapted to the tubular geometry of specimen.

The maximum relative bending shall not exceed 5 % at an average strain of $5,10^{-4}$.

5.4 Strain measurement.

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5.4.1 General

Strain should be locally measured in order to avoid having to take into account the compliance of the machine. This may be by means of suitable extensometers, bonded resistance strain gauges or digital image correlation (DIC). If Poisson's ratio is to be determined, the tubular test specimen must be instrumented to measure strain in both longitudinal and circumferential directions.

5.4.2 Extensometers

Extensometers used for tensile testing of CFCC tubular test specimens shall be capable of continuously recording the longitudinal strain at test temperature. They shall be of class 1 in accordance with ISO 9513.

Extensometers with the highest gauge length are recommended (minimum of 25 mm required) and shall be centrally located in the mid region of the axial direction of the gauge section of the tubular test specimen.

If mechanical extensometers are used, the selected attachment should cause no damage to the specimen surface. In addition, the weight of the extensometers should be supported, so as not to introduce bending stresses in the tubular test specimen greater than that allowed in [5.3](#).

Extensometers should preferably be of a type that is capable of measuring elongation on two places of the tubular test specimen for averaging of strain and/or determination of in-situ relative bending. 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.

5.4.3 Strain gauges

5.4.3.1 General

Although extensometers are commonly used to measure strain in tensile test of CFCC tubes, strain can also be determined with bonded resistance strain gauges and suitable strain recording equipment. The strain gauges, the surface preparation and the bonding agents should be chosen to provide adequate performance on the tested materials.

Some guidelines on the use of strain gauges on CFCC tubes are as follows.

5.4.3.2 Strain gauge selection

Unless it can be shown that strain gauge readings are not unduly influenced by localized strain events such as fibre crossovers, strain gauges should be not less than 9 mm to 12 mm in length for the longitudinal direction and not less than 6 mm in length for the circumferential direction.

When testing braided or woven fabric composites, the strain gauges should have an active gauge length that is at least as great as the characteristic unit cell (repeating unit) of the reinforcement; this averages the localized strain effects of the fibre crossovers.

In uniaxial loading, a single-grid gauge pattern would normally be used with the gauge axis aligned to coincide with the longitudinal direction of the tubular test specimen.

NOTE Poisson's ratio can be determined with biaxial two-element (0–90) strain gauge rosettes which measure the strain in both the longitudinal and circumferential directions.

5.4.3.3 Surface preparation

The relatively rough surface of composites usually requires some preparation prior to strain gauge bonding. The basic steps have to include solvent degreasing, abrading or filing and cleaning.

Matrix-rich surfaces can usually be abraded with 320-grit silicon carbide paper (SCP-2) to produce a satisfactory matte finish. However, unless their surfaces have been machined or have received a smoothing treatment, tubular test specimens of poor matrix content composites or those with textured surface require alternative techniques.

NOTE A typical method is to apply a thin epoxy precoat to smooth the surface irregularities and finish by polishing.

Reinforcing fibres should not be exposed or damaged during the surface preparation process. In particular, abrasion should be kept to a minimum to avoid possible damage to fibres in the external surface of the composite.

5.4.4 Digital image correlation

5.4.4.1 General

The DIC method can also be used to determine local strain of CFCC tubular test specimens loaded in tensile from the displacement field measurement. The general procedure to be followed for estimating the strain shall be in accordance with ASTM E2208-02.

Some guidelines on the use of the DIC method on CFCC tubes are as follows.

5.4.4.2 Experimental setup

The experimental setup for DIC measurements requires a digital CCD camera coupled with an optical macro lens to acquire high spatial resolution micrographs (a minimum of 20 µm per pixel is recommended). In the present case, the use of a telecentric lens is required to overcome the curvature effect of the tubular test specimens.

The imaging conditions for DIC measurements have to be selected to ensure that the entire coupon surface is in the best focal plane of the camera and that the highest possible magnification could be attained. Annular illumination with white or monochromatic light is recommended to provide a correct signal-to-noise ratio.

The camera needs to be able to acquire micrographs at a suitable frame rate in order to achieve a sufficient temporal resolution of the test. Depending on the device, special timing and triggering control is required to synchronize the acquisition of the camera with the applied load.

For mechanical tensile test, the maximum frame rate of the camera limits the maximum speed of the displacement that can be imposed on the specimen. In general, the frame rate of the camera should be at least twice the displacement rate.

5.4.4.3 Surface preparation

The requirement for surface preparation depends on both the magnification of the imaging system and the surface characteristics of the composite. In general, the technique requires sharp grayscale information of the order of 1 pixel in size at the CCD recording device.

The most common way to prepare a suitable surface is the use of high-contrast speckle patterns. These can be obtained by applying a matt randomized coating such as speckled black dots on a homogenous white background. The composite materials with a pronounced roughness average are expected to naturally produce highly micro-textured images.

5.4.4.4 Calculation

Correlation may be then carried out using DIC commercial software. The selection of the correlation area in terms of dimensions should be such as calculations led to determine the longitudinal strain.

Reference to the software provider's instructions must be followed for the execution and interpretation of the measure. <https://standards.iteh.ai/catalog/standards/sist/d1cb4576-5cf8-47e6-8bd9-0c3f7fa39583/iso-20323-2018>

NOTE Poisson's ratio can be determined by applying a similar calculation to measure the circumferential strain on a close area.

5.5 Data recording system.

A calibrated recorder should be used to record the applied tensile force and the gauge section elongation (or strain) versus time. The use of a digital data recording system is recommended for this purpose.

Recording devices shall be accurate to within $\pm 0,1$ % for the entire testing system including readout unit, and shall have a minimum data acquisition rate of 10 Hz with a response of 50 Hz deemed more than sufficient.

Crosshead displacement of the test machine may also be recorded but shall not define displacement or strain in the gauge section, especially when self-aligning couplers are used in the load train.

5.6 Dimensions measurement.

Micrometers used for the measurement of the dimensions of the test specimen shall conform to ISO 3611. The internal and external diameters of the tubular test specimen should be measured with an accuracy of 0,02 mm or 1 % of the measured dimension, whichever is higher. Flat anvil type micrometer or calipers of similar resolution may be used for measuring the overall test specimen length and the defined gauge length.

Ball-tipped or sharp anvil micrometers are not recommended for tubular CFCCs because the resulting measurements may be affected by the peaks and troughs of the weave.

In some cases it is desirable, but not required, to determine dimensions of the tubular test specimen subtracted from surface roughness (internal and external diameters). Methods such as

contacting/optical profilometry or image analysis on a polished transverse cross section may be used for this purpose.

6 Tubular test specimens

6.1 Specimen specifications

6.1.1 General

CFCC tubes are fabricated in a wide range of sizes and geometries and across a wide spectrum of different reinforcement fibres, distinctive ceramic matrix materials and markedly different fabrication methods. The fibre architecture for CFCC tubes also has a broad range of configurations with different fibre loadings and directional variations. Therefore, it is currently not practical to define a single test specimen geometry that is applicable to all CFCC tubes.

The selection and definition of a tubular test specimen geometry depends on the purpose of the tensile test. In addition, grip devices and load train couplers (as discussed in [5.2](#) and [Annex A](#)) may influence the design of the test specimen geometry.

6.1.2 Dimension

The test method described applies to CFCC tubes with external diameters greater than 7 mm and a minimum of 0,5 mm in wall thickness, roughly corresponding to minimum one single layer. The ratio of external diameter to wall thickness (\varnothing/h) is commonly extended in the range of 5 to 30.

The total length of specimen depends on the selected experimental configuration for testing, but the volume in the gauge length shall be representative of the composite material. As a general rule, the calibrated length (l) should be commonly selected to keep the ratio (l/\varnothing) between 2 and 3 with a minimum value of 30 mm recommended.

Deviations outside the recommended ranges may be necessary depending upon the particular CFCC being evaluated.

6.1.3 Geometry

A straight-sided tube geometry that often does not require machining to obtain proper dimensions is recommended to carry out the test. It can be used with active and passive grips. For this geometry, both the internal and external surfaces of the specimens may be rough and irregular.

[Figure 2](#) represents a tubular test specimen with straight-sided geometry. Dimensional requirements for acceptable specimen are contained in [Table 1](#).

In some cases, another geometry of CFCC tube can be accepted. [Annex B](#) provides an example of contoured gauge test specimen tube geometry with dimensional requirements.