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Fine ceramics (advanced ceramics, advanced technical ceramics) — Test method for tensile stress-strain behaviour of continuous, fibre-reinforced composites at room temperature

Céramiques techniques — Méthode d'essai de comportement à la contrainte en traction des composites renforcés de fibres continues, à température ambiante

Reference number
ISO 15733:2001(E)

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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 3.

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 International Standard may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

International Standard ISO 15733 was prepared by Technical Committee ISO/TC 206, *Fine ceramics*.

Annex A forms a normative part of this International Standard, annex B is for information only.

Fine ceramics (advanced ceramics, advanced technical ceramics) — Test method for tensile stress-strain behaviour of continuous, fibre-reinforced composites at room temperature

1 Scope

This International Standard specifies the determination of in-plane tensile behaviour including stress-strain response under monotonic uniaxial testing of continuous fiber-reinforced ceramic matrix composites (CFRCMCs) at ambient temperature.

This International Standard addresses, but is not restricted to, various suggested test piece geometries, test piece fabrication methods, testing modes, testing rates, allowable bending, data collection and reporting procedures. This International Standard applies primarily to ceramic and/or glass matrix composites with continuous fiber reinforcement: uni-directional (1-D), bi-directional (2-D) and tri-directional (3-D) or other multi-directional reinforcements. Carbon fiber-reinforced carbon matrix (C/C) composites may also be tested using this International Standard, although caution is advised since this International Standard was developed primarily for CFRCMCs and any accommodations unique to C/C composites have not been included.

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

2 Normative references

The following normative documents contain provisions which, through reference in this text, constitute provisions of this International Standard. For dated references, subsequent amendments to, or revisions of, any of these publications do not apply. However, parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the normative documents indicated below. For undated references, the latest edition of the normative document referred to applies. Members of ISO and IEC maintain registers of currently valid International Standards.

ISO 286-1:1988, *ISO system of limits and fits — Part 1: Bases of tolerances, deviations and fits.*

ISO 3611:1978, *Micrometer callipers for external measurement.*

ISO 6892:1998, *Metallic materials — Tensile testing at ambient temperature.*

ISO 7500-1:1999, *Metallic materials — Verification of static uniaxial testing machines — Part 1: Tension/compression testing machines — Verification and calibration of the force-measuring system.*

ISO 9513:1999, *Metallic materials — Calibration of extensometers used in uniaxial testing.*

3 Terms and definitions

For the purposes of this International Standard, the following terms and definitions apply.

3.1
fine ceramic (advanced ceramic, advanced technical ceramic)
highly-engineered, high-performance predominately non-metallic, inorganic, ceramic material having specific functional attributes

3.2
axial strain
average longitudinal strain measured at the surface on opposite sides of the longitudinal axis of symmetry of the test piece by strain-sensing devices located at the mid length of the reduced section

3.3
bending strain
difference between the strain at the surface and the axial strain

NOTE In general, the bending strain varies from point to point around and along the reduced section of the test piece.

3.4
breaking force
force at which fracture occurs

3.5
ceramic matrix composite
material consisting of two or more materials (insoluble in one another), in which the major, continuous component (matrix component) is a ceramic, while the secondary component(s) (reinforcing component) may be ceramic, glass-ceramic, glass, metal or organic in nature; these components are combined on a macroscale to form a useful engineering material possessing certain properties or behaviour not possessed by the individual constituents

3.6
continuous fiber-reinforced ceramic matrix composite (CFRCMC)
ceramic matrix composite in which the reinforcing phase consists of a continuous fiber, continuous yarn or a woven fabric

3.7
fracture strength
tensile stress which the material sustains at the instant of fracture

NOTE Fracture strength is calculated from the force at fracture during a tensile test carried to rupture and the original cross-sectional area of the test piece.

3.8
gauge length
original length of that portion of the test piece over which strain or change of length is determined

3.9
irrecoverable cumulative damage energy (also known as, modulus of toughness)
strain energy per unit volume required to stress the material from zero to final fracture indicating the ability of the material to absorb energy beyond the elastic range (i.e., inherent damage tolerance of the material)

3.10
matrix-cracking stress
the applied tensile stress at which the matrix cracks into a series of roughly parallel blocks perpendicular to the tensile stress

3.11
modulus of elasticity
the ratio of stress to corresponding strain less than the proportional limit

3.12
proportional limit stress
the greatest stress which a material is capable of sustaining without any deviation from proportionality of stress to strain (Hooke's law)

3.13
percent bending
the bending strain times 100 divided by the axial strain

3.14
recoverable elastic energy (also known as, modulus of resilience)
strain energy per unit volume required to elastically stress the material from zero to the proportional limit indicating the ability of the material to absorb energy when deformed elastically and return it when the force is removed

3.15
slow crack growth
sub-critical crack growth (extension) which may result from, but is not restricted to, such mechanisms as environmentally-assisted stress corrosion or diffusive crack growth

3.16
tensile strength
the maximum tensile stress which a material is capable of sustaining

NOTE Tensile strength is calculated from the maximum force during a tensile test carried to rupture and the original cross-sectional area of the test piece.

3.17
test series
a discrete group of tests on individual test pieces conducted within a discrete period of time on a particular material configuration, test piece geometry, test condition or other uniquely definable qualifier (e.g., a test series composed of material A comprising ten test pieces of geometry B tested at a fixed rate in strain control to final fracture in ambient air)

4 Symbols and designations

Symbols used throughout this International Standard and their designations are given in Table 1.

Table 1 — Symbols and designations

Symbol	Designation	Unit	References
A	Surface area	mm^2	9.5.1 equations 1, 2a, 2b
d	Thickness	mm	Tables 2, 3 Figures 3, 4
E	Elastic modulus (Young's modulus)	MPa	9.5.7 equation 6, Figure 6
E_R	Recoverable elastic energy (modulus of resilience)	J/m^3	9.5.10 equation 7
E_T	Irrecoverable cumulative damage energy (modulus of toughness)	J/m^3	9.5.11 equations 8, 9
F	Force	N	9.5.1 equation 1
F_f	Force at fracture	N	9.5.5 equation 5
F_m	Maximum force	N	9.5.3 equation 4
l	Length, extensometer or test piece at any time	mm	9.5.2 equation 3
l_0	Length, original extensometer or test piece	mm	9.5.2 equation 3

Table 1 *(continued)*

Symbol	Designation	Unit	References
L	Length, total for straight-sided test piece geometry	mm	Table 3 Figure 4
L_1	Length, gauge for contoured test piece geometry	mm	Table 2 Figure 3
L_2	Length, total for contoured test piece geometry	mm	Table 2 Figure 3
n	Number of valid tests	1	10.1, g)
n_T	Number of total tests	1	10.1, g)
R	Radius, blend for contoured test piece geometry	mm	Table 2 Figure 3
SD	Standard deviation	var.	equation 11
R_f	Tensile strength at fracture	MPa	9.5.5 equation 5
R_m	Ultimate tensile strength	MPa	9.5.3 equation 4
V	Coefficient of variation	var.	equation 12
W	Width, total for straight-sided test piece geometry	mm	Table 2 Figure 4
W_1	Width, gauge for contoured test piece geometry	mm	Table 2 Figure 3
W_2	Width, grip for contoured test piece geometry	mm	Table 2 Figure 3
$\overline{X}$	Mean	1	equation 10
ε	Strain, normal	mm/mm	9.5.2 equation 3
ε_f	Strain, corresponding to R_f	mm/mm	9.5.6 Figure 6
ε_m	Strain, corresponding to R_m	mm/mm	9.5.4
ε_0	Strain, corresponding to σ_0	mm/mm	9.5.9 Figure 6
σ	Stress, normal	MPa	9.5.1 equation 1
σ_0	Stress, proportional limit	MPa	9.5.8 Figures 6, 7

5 Principle

This International Standard is for material development, material comparison, quality assurance, characterization, reliability and design data generation. Dissimilar material response of CFRCMCs in tension and compression prevents unambiguous characterization of material behaviour from flexural tests. Therefore, uniaxially-tested and uniformly-stressed tensile tests can provide information on fundamental material behaviour including stress-strain response, proportional limit and ultimate strengths, elastic constants, and strain-energy absorption.

This test consists of testing a test piece to fracture using a uniaxial tensile force for the purpose of determining tensile stress-strain response, various tensile strengths and corresponding strains, elastic constants and various deformation energies. Generally, this test is carried out under conditions of ambient temperature and environment.

6 Apparatus

6.1 Testing machine

The testing machine shall be verified in accordance with ISO 7500-1 and shall be of at least grade 1,0 unless otherwise specified.

6.2 Test piece gripping

Various types of gripping device may be used to transmit the measured force applied by the testing machine to the test piece. The brittle nature of the matrices of CFRCMCs requires a uniform interface between the grip components and the gripped section of the test piece in order to minimize crack initiation and fracture of the test piece in the gripped section. Gripping devices can be classified generally as those employing active and those employing passive grip interfaces.

6.2.1 Active grip interfaces

Active grip interfaces require continuous application of a mechanically-, hydraulically- or pneumatically-derived force (pressure) to transmit the force applied by the test machine to the test piece. Sufficient lateral pressure shall be applied to prevent slippage between the grip face and the test piece. Grip surfaces that are scored or serrated with a pattern similar to that of a single-cut file have been found to be satisfactory. See Figure 1.

NOTE Generally, these types of grip interface cause a force to be applied perpendicular to the surface of the gripped section of the test piece. Transmission of the uniaxial force applied by the test machine is then accomplished by friction between the test piece and the grip faces.

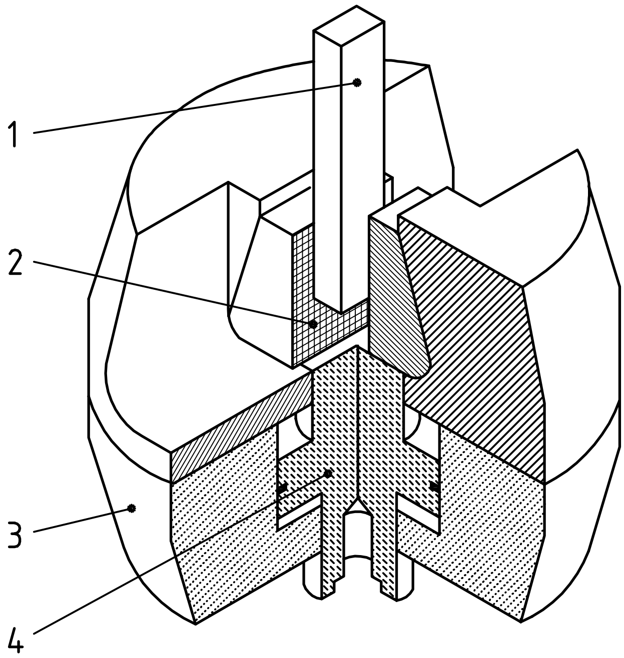
6.2.2 Passive grip interfaces

Passive grip interfaces transmit the force applied by the test machine to the test piece through a direct mechanical link. These mechanical links transmit the test forces to the test piece via geometrical features of the test pieces such as shank shoulders or holes in the gripped head. See Figure 2.

NOTE Generally, the uniaxial force is transmitted to the test piece through uniform contact along the entire test piece/grip interface thus minimizing eccentric forces.

6.2.3 Test train couplers

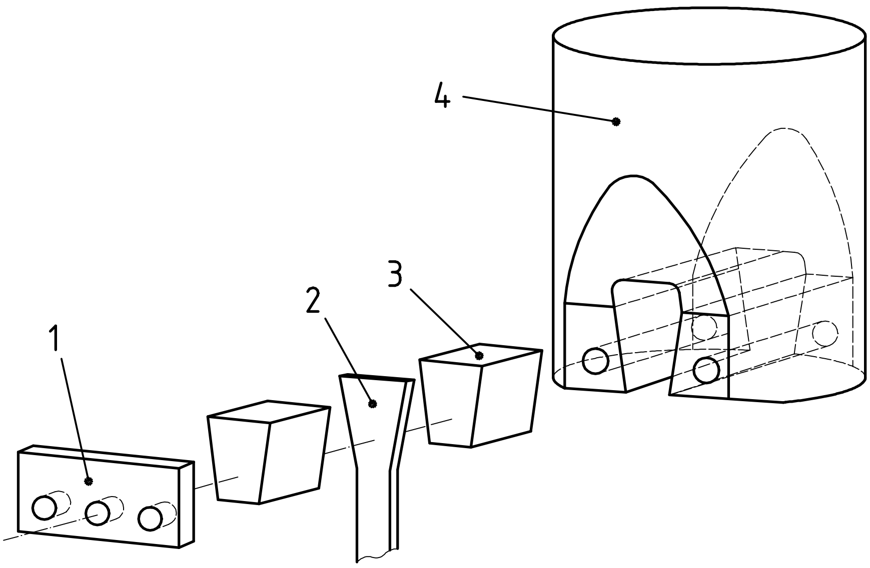
Various types of device (test-train couplers) may be used to attach the active or passive grip interface assemblies to the testing machine. The test-train couplers in conjunction with the type of gripping device play major roles in the alignment of the test train and subsequent bending imposed in the test piece. The efficacy of the test train couplers and grip interfaces is verified through the procedure discussed in 8.1 and Annex A.



Key

1 Test piece
2 Wedge grip
3 Grip body
4 Grip mechanism

Figure 1 — Example of an active grip interface



Key

1 Retaining plate
2 Test piece
3 Inserts for lateral centring of test piece
4 Grip attachment

Figure 2 — Example of a passive grip interface

6.3 Strain measurement

Strain measurement is required for tensile testing of CFRCMC test pieces in accordance with this International Standard.

Extensometers shall be of class 1 in accordance with ISO 9513. The extensometer gauge length shall be not less than 10 mm (25 mm preferred) and shall be centrally located in the mid region of the parallel length of the gauge section of the test piece.

Extensometers which are in mechanical contact with the test piece shall not cause damage to the test piece surface such that a detrimental effect on tensile behaviour is produced. Ensure that the extensometer does not introduce bending greater than that allowed in 8.1. Extensometers shall preferably be of a type that is capable of measuring elongation on both sides of a test piece (for averaging of strain and/or determination of in-situ percent bending).

Strain gauges may also be used to measure strain in tensile tests of CFRCMCs. Unless it can be shown that strain gauge readings are not unduly influenced by localized strain events such as fiber 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 transverse direction. The strain gauges, surface preparation and bonding agents should be chosen to provide adequate performance on the subject materials and suitable strain-recording equipment should be used.

6.4 Data acquisition

Obtain at least an autographic record of applied force and gauge section elongation or strain versus time using either analogue chart recorders or digital data acquisition systems. Recording devices shall be accurate to within 1 % of the selected range for the testing system including readout unit and should have a minimum data acquisition rate of 10 Hz with a response of 50 Hz deemed more than sufficient.

6.5 Dimension measurement

Micrometers and other devices used for measuring linear dimensions shall be accurate and precise to at least one half the smallest unit to which the individual dimension is required to be measured and shall be in accordance with ISO 3611. To obtain consistent measurements of cross sectional dimensions, use a flat, anvil-type micrometer. Ball-tipped or sharp anvil micrometers are not recommended for woven CFRCMCs because the resulting measurements may be affected by the peaks and troughs of the weave. Measure cross-sectional dimensions to within 0,02 mm using dimension-measuring devices with accuracies of 0,01 mm.

7 Test piece

7.1 Test piece geometry

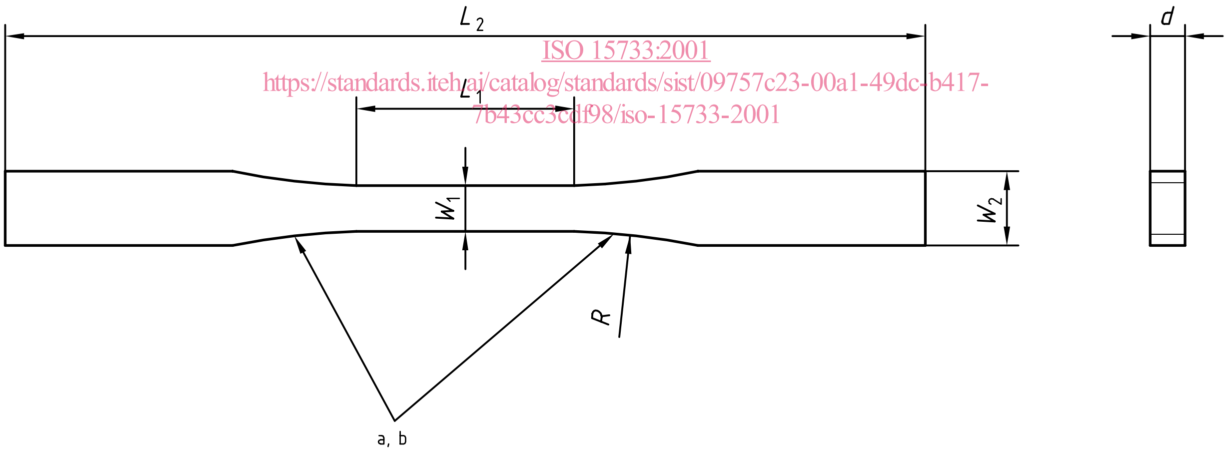
The choice of geometry of a tensile test piece is dependent on the ultimate use of the tensile behaviour data. For example, if the tensile strength of an as-fabricated component is required, the dimensions of the resulting test piece may reflect the thickness, width and length restrictions of the component. If it is desired to evaluate the effects of interactions of various constituent materials for a particular CFRCMC manufactured via a particular processing route, then the size of the test piece and resulting gauge section will reflect the desired volume or surface area to be sampled.

Therefore, no single test piece geometry can be recommended or prescribed to meet all the requirements of a particular testing programme or apparatus. Annex B contains further information on test piece geometries including a figure showing examples of successful test piece geometries used for CFRCMCs.

Certain dimensional requirements are contained in Tables 2 and 3 depending on whether contoured (Figure 3) or straight-sided geometries (Figure 4) are used, respectively.

Table 2 — Minimum dimensions of contoured test piece geometries (see Figure 3)

Dimension	Minimum value mm	Tolerance mm
Total length, L_2	⩾ 100	± 0,5
Gauge Length, L_1	⩾ 30	± 0,2
Thickness, d	⩾ 2 and at least a) three plies for simply woven materials or b) one unit cell width for complex woven materials	± 0,2
Gauge width, W_1	⩾ 6 and at least a) three fibre bundles for simply woven materials or b) one unit cell width for complex woven materials	± 0,2
Grip width, W_2	⩾ 10 and at least 1,4 × W_1	± 0,2
Blend radius, R	⩾ 35	± 2
Parallelism of machined part	0,05	



a Smooth and blend at intersection with width, W_1, of gauge section.

b Simple intersection (no steps of jogs) with width, W_2, of grip section.

Figure 3 — "Generic" countered test piece geometry (see Table 2)

