International Standard

ISO 22459

Fine ceramics (advanced ceramics, advanced technical ceramics) — Reinforcement of ceramic composites — Determination of distribution of tensile strength and tensile strain to failure of filaments within a multifilament tow at ambient temperature

Second edition
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Céramiques techniques — Renfort de céramiques composites — Détermination de la distribution de la résistance en traction et de la déformation à la rupture en traction de filaments dans un fil multifilamentaire à température ambiante

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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 22459:2020), of which it constitutes a minor revision.

The changes are as follows:

— Figure 2 has been updated;

— Figure 3 has been updated.

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) — Reinforcement of ceramic composites — Determination of distribution of tensile strength and tensile strain to failure of filaments within a multifilament tow at ambient temperature

1 Scope

This document specifies the conditions for the determination of the distribution of strength and rupture strain of ceramic filaments within a multifilament tow at room temperature by performing a tensile test on a multifilament tow.

This document applies to dry tows of continuous ceramic filaments that are assumed to act freely and independently under loading and exhibit linear elastic behaviour up to failure. The outputs of this method are not to be mixed up with the strengths of embedded tows determined by using ISO 24046.

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 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 10119, *Carbon fibre — Determination of density*

EN 1007-2, *Advanced technical ceramics — Ceramic composites — Methods of test for reinforcements — Part 2: Determination of linear density*

3 Terms and definitions

For the purposes of this document, the following terms and definitions 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
gauge length
L_0
initial distance between two reference points on the tow

Note 1 to entry: Usually the gauge length is taken as the distance between the gripped ends of the tow.

3.2
initial cross-section area
S_0
cross-section area of the tow

3.3
tow elongation
A
increase of the gauge length during the tensile test

3.4.1
total compliance
C_t
ratio of the measured displacement to the corresponding force during the tensile test

3.4.2
load train compliance
C_l
ratio of the load train elongation, excluding the specimen contribution, to the corresponding force during the tensile test

3.5
strain
ε
ratio of the tow elongation A to the gauge length L_0

3.6
filament rupture strain
$\varepsilon_{r,j}$
strain at step j in the non-linear parts of the force-displacement curve

3.7
filament strength
$\sigma_{r,j}$
ratio of the tensile force to the cross-section area of all unbroken filaments at step j in the non-linear parts of the force-displacement curve

3.8
average filament strength
$\bar{\sigma}_r$
statistical average strength of the filaments in the tow for each test determined from the Weibull strength distribution parameters of the filaments

3.9
mean filament strength
$\bar{\bar{\sigma}}_r$
arithmetic mean of the average strengths

4 Principle

A multifilament tow is loaded in tension at a constant displacement rate up to rupture of all the filaments in the tow. The force and displacement are measured and recorded. From the force-displacement curve the two-parameter Weibull distribution of the rupture strain and of the strength of the filaments is obtained by sampling the nonlinear parts of the curve at discrete intervals, j, which correspond to an increasing number of failed filaments in the tow.

5 Significance and use

Because measurement of the displacement directly on the tow is difficult, it is usually obtained indirectly via a compliance measurement which includes contributions of the loading train, the grips and the tabbing materials. These contributions have to be corrected for in the analysis. When it is possible to measure the tow elongation directly (by using a suitable extensometer system) this correction is not needed. The calculation of the results in Clause 10 also applies in this case by setting the load train compliance equal to zero.

The evaluation method is based on an analysis of the nonlinear domain of the force-displacement curve, which is caused by progressive filament failure during the test. The size of this domain is promoted by higher stiffness of the loading and gripping system. When the force-displacement curve does not show this nonlinear domain, the evaluation method of this document cannot be applied.

The distribution of filament rupture strains does not depend on the initial number of filaments for those tows that contain a large number of filaments; hence, it is not affected by the number of filaments which are broken before the test, provided this number remains limited. The determination of the filament strength distribution requires knowledge of the initial cross-sectional area of the tow. The variation in filament diameters, which affects the strength values, is not accounted for.

The Weibull parameters determined by this test method and extrapolated to the respective gauge length cannot be compared directly with those obtained from tensile tests on monofilaments according to ISO 19630 because of variability in test conditions[1].

6 Apparatus

6.1 Tensile testing equipment

The test machine shall be equipped with a system for measuring the force applied to the specimen and the displacement, or directly the tow elongation. The machine shall conform to grade 1 or better in ISO 7500-1. The grips shall align the specimen with the direction of the force. Slipping of the specimen in the grips shall be prevented.

NOTE   The use of a displacement transducer placed at the ends of the grips[5][6] (see Figure 1) or on the tow itself[4][5][6] will probably limit the contribution of different parts of the load train to the measured displacement, and hence increase the accuracy.
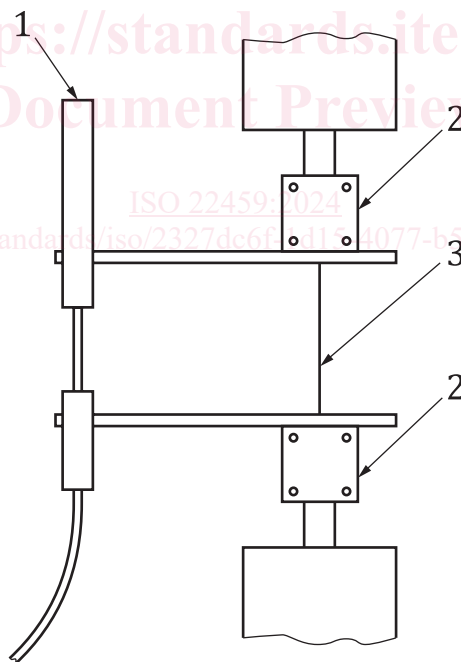
Key

1   displacement transducer

2   grip

3   test specimen

Figure 1 — Test setup (principle sketch)

6.2   Data recording

A calibrated recorder shall be used to record force-displacement curves. The use of a digital data recording system is recommended.

7   Test specimen

7.1   General

Specimens with a gauge length of 200 mm shall be used to establish the filament strength and filament rupture strain distributions. Specimens with gauge lengths of 100 mm and 300 mm shall be used to determine the load train compliance. Examples of two types of test specimen are given below.

7.2   Window type specimen

A window type specimen is shown in Figure 2. A stretched tow is fixed between two identical plates of material, each containing a central window. When the displacement is not measured directly on the tow, the height of the window defines the gauge length.

NOTE   This type of specimen has the advantage of easy handling.
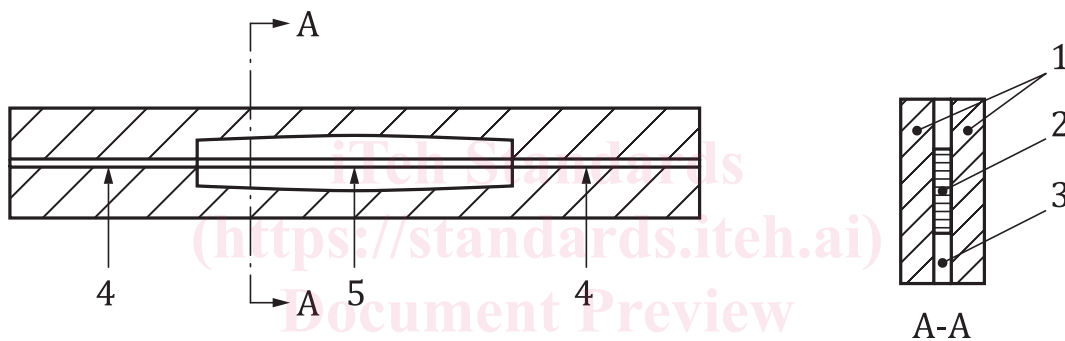
Key

1   plates
2   tow
3   glue
4   gripped end
5   gauge length

Figure 2 — Window type specimen (principle sketch, side view)

7.3   Cylindrical end type specimen

A cylindrical end type specimen is shown in Figure 3. Both ends of a stretched tow are fixed in small diameter cylindrical tubes generally made of metal. When the displacement is not measured directly on the tow, the distance between the inner ends of the tubes with the tow in a stretched condition defines the gauge length. Tube length shall be such that adhesion of tow specimen to tube is optimized. Length larger than 30 mm is recommended.
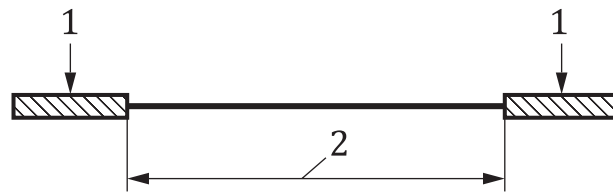
Key

1   tube

2   gauge length

Figure 3 — Cylindrical end type specimen (principle sketch)

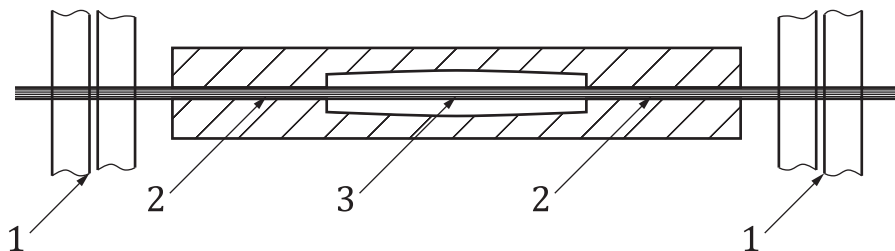## 8   Test specimen preparation

8.1   General

Extreme care shall be taken during specimen preparation to ensure that the procedure is repeatable from specimen to specimen. When glue is used, the same type and the same bonding length shall be used for the preparation of all test specimens of a given series. Specimens shall be handled with care to avoid breaking filaments.

High repeatability in specimen preparation is required in order to allow a correct determination of the load train compliance.

A sizing agent is present on certain fibres. It protects the filaments against damage during handling or prevents inter-filament friction during the tests. It should not be removed. Owing to its low Young's modulus, it does not contribute to load sharing. Care should be taken that the glue will not run into the tow outside the frame. Epoxy or resin that exhibit excellent wetting properties with SiC and Alumina-based ceramics are appropriate.

8.2   Window type specimen

An untwisted multifilament tow is glued between two identical plates made of cardboard or another suitable material. The filaments shall be stretched. To achieve this, both ends of the two plates are well soaked by the glue, then the tow is placed on the centreline of one of the plates under a small axial prestress. The ends of the tow extending beyond the plate are fixed by adhesive tapes onto a support (see Figure 4) and the parts of the tow in the gripping area are soaked with glue. The second plate is then pressed face to face with the first one.


Key

1   adhesive tapes

2   gripped end

3   gauge length, L_0

Figure 4 — Window type specimen, preparation (principle sketch)

8.3 Cylindrical end type specimen

The specimens are prepared on a support provided with alignment grooves in which the cylindrical tubes are placed. The untwisted multifilament tow is introduced into the tubes, stretched and glued (see Figure 5). The diameter of the cylindrical tubes shall be as small as possible, compatible with the size of the tow.
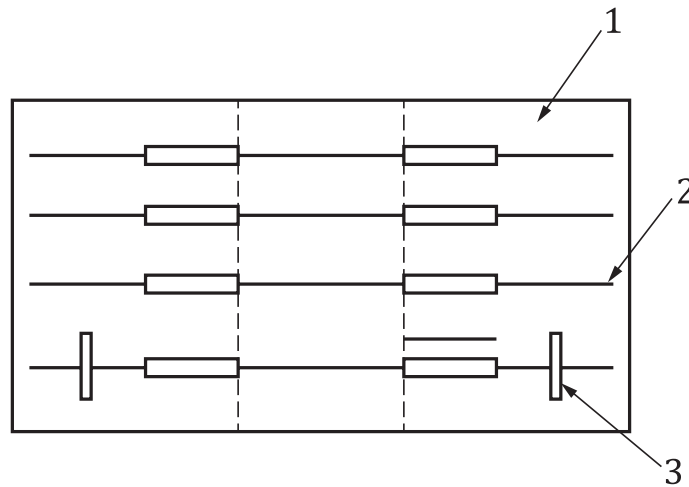
Key

1 support
2 groove for alignment
3 adhesive tapes

Figure 5 — Cylindrical end type specimen, preparation (principle sketch)

8.4 Number of test specimens

For the establishment of the distribution of filament strength and filament rupture strain, three valid tests, as specified in 9.7, of specimens with a 200 mm gauge length are needed. When the elongation of the tow is not measured directly, an additional three valid tests at the other two gauge lengths of 100 mm and 300 mm, as specified in 9.7, are required for the establishment of the load train compliance.

9 Test procedure

9.1 Determination of the initial cross-section area

For the purpose of determining the filament strength distribution, as well as the elastic modulus on the specimens with a 200 mm gauge length, the initial cross-section area of the multifilament tow shall be calculated from the linear density determined according to EN 1007-2, and from the density determined in ISO 10119. Alternatively, the initial cross-section area can be determined by measuring the number and the average diameter of the filaments in the tow, for instance through image analysis.

9.2 Determination of the gauge length

The gauge length shall be measured with an accuracy of ±0,5 mm.

9.3 Gripping

The specimen shall be placed in the test equipment in such a way that axial alignment is as accurate as possible. During gripping, care shall be taken not to load the specimen in tension. When the displacement is not measured directly on the tow, the specimen shall be inserted in the grips in such a way that the distance between the grips is equal to or less than the gauge length (see Figure 6).