
**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**

*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

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).

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

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For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT), see www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 206, *Fine ceramics*.

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 24046¹⁾.

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 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

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

1) Under preparation.

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

$\bar{\sigma}_r$

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.

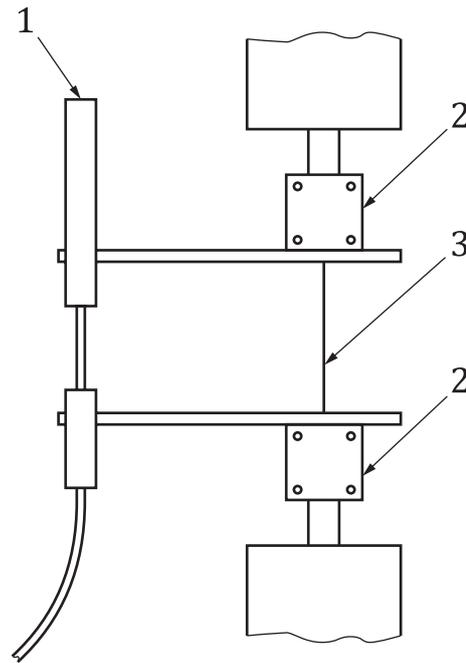
The Weibull parameters determined by this test method and extrapolated to the respective gauge length cannot be compared directly with those obtained from tensile tests on monofilaments according to ISO 19630 because of variability in test conditions^[1].

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

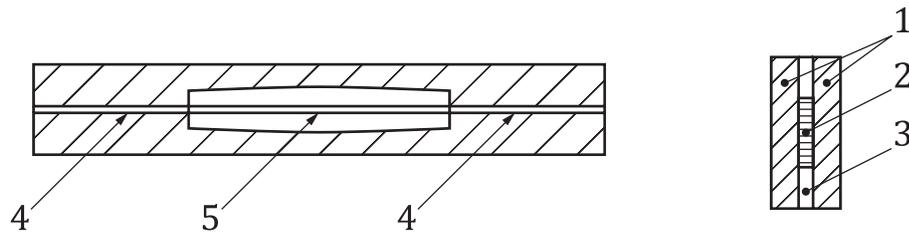
7.1 General

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

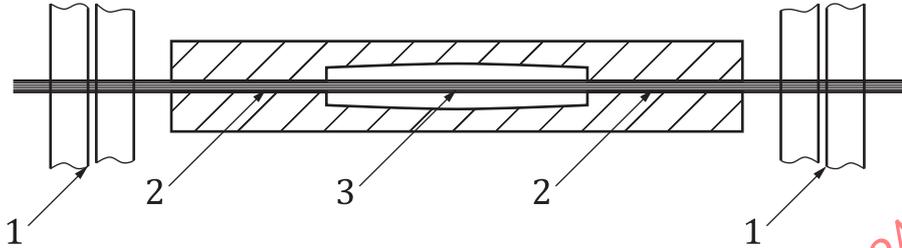
Extreme care shall be taken during specimen preparation to ensure that the procedure is repeatable from specimen to specimen. When glue is used, the same type and the same bonding length shall be used for the preparation of all test specimens of a given series. Specimens shall be handled with care to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

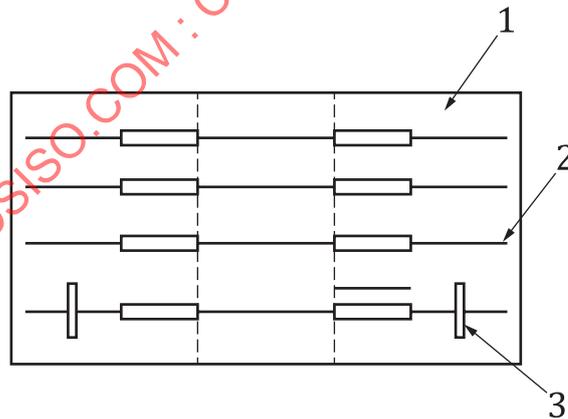


- Key**
- 1 adhesive tapes
 - 2 gripped end
 - 3 gauge length, L_0

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

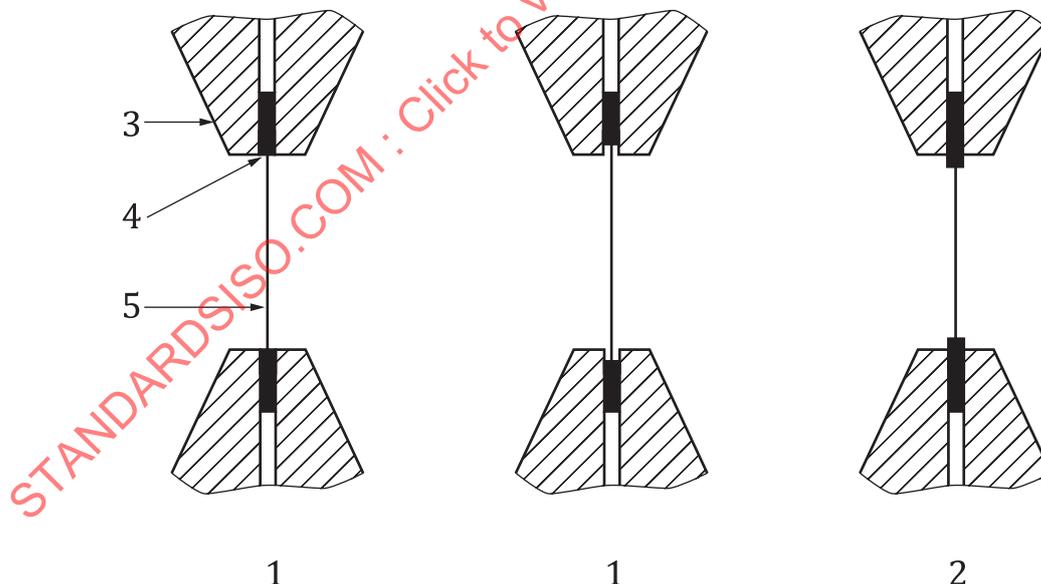
For the purpose of determining the filament strength distribution, as well as the elastic modulus on the specimens with a 200 mm gauge length, the initial cross-section area of the multifilament tow shall be calculated from the linear density determined according to EN 1007-2, and from the density determined 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 $\pm 0,5$ 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).



Key

- 1 acceptable
- 2 not acceptable
- 3 grip
- 4 tube
- 5 tow

Figure 6 — Test specimen mounting (principle sketch)

9.4 Selection of strain rate

A strain rate around 10^{-4}s^{-1} shall be used for all the tests. The corresponding crosshead displacement rate shall be determined from a test on a specimen with the largest gauge length of 300 mm performed according to 9.5. The force-displacement curve obtained from this test shall have the appearance shown in Figure 7. In particular, the curve shall have a linear followed by a nonlinear rising part, as well as a nonlinear decreasing part. It shall furthermore meet the validity requirements of 9.7. When the force-displacement curve does not meet these criteria, tests at lower crosshead displacement rates shall be performed until this is the case. Calculate the strain rate from the displacement rate and check whether it falls in the required range. If not, decrease the crosshead displacement rate until this is the case. Use this crosshead rate in all subsequent tests, irrespective of the gauge length of the specimen.

9.5 Test procedure

Mount the specimen in the load train. Set the displacement rate on the machine. When a window type specimen is used, carefully cut both sides of the supporting plates. Start the load versus displacement recording. Load the specimen up to failure under constant displacement rate. Remove the failed specimen from the grips. Check the validity requirements of 9.7.

It is recommended that a lubricant is introduced in the tow in order to reduce inter-filament friction. Lubricant oil and petrol have been used on SiC fibres.

9.6 Determination of load train compliance

Repeat steps 9.2, 9.3 and 9.5 three times for each of the gauge lengths of 100 mm, 200 mm and 300 mm.

9.7 Test validity

The test is invalid in the following circumstances:

- failure to specify and record test conditions;
- the linear region in the rising part of the force-displacement curve is lacking;
- one or more load drops with an amplitude larger than 5 % of the maximum force occur in the rising part of the force-displacement curve;
- filament rupture occurs preferentially in the grips or near the gripped ends (the test is valid when both parts of the test specimen after failure have a significant number of filaments, but not all, extending beyond half of the gauge length);
- one or more load drops at constant strain with an amplitude larger than 5 % of the maximum force occur beyond the point of maximum force of the force-displacement curve (this restriction does not apply for the determination of the load train compliance);
- the nonlinear domain at the origin of the curve exceeds 10 % of the displacement corresponding to the maximum force.

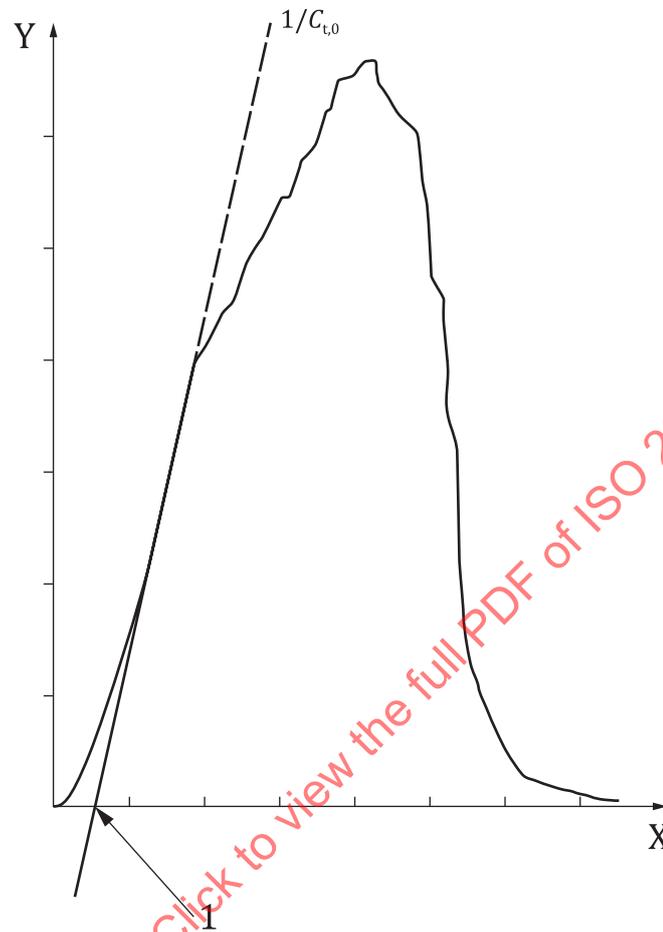
10 Calculation of results

10.1 Calculation of the load train compliance C_l

Calculate the initial total compliance $C_{t,0}$ (mm/N) for the tests at each of the gauge lengths from the slope $1/C_{t,0}$ of the linear rising part of the force (N)-displacement (mm) curve (see Figure 7). Calculate the average value $\bar{C}_{t,0}$ at each of the three gauge lengths.

Plot $\bar{C}_{t,0}$ against L_0 (see Figure 8).

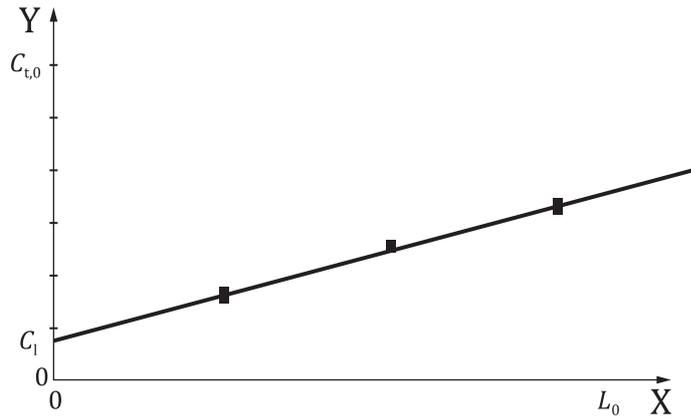
Perform a linear regression analysis of $\bar{C}_{t,0}$ versus L_0 and determine the load train compliance C_1 from the intercept at $L_0 = 0$.



Key

- X force F (N)
- Y displacement (mm)
- 1 true origin

Figure 7 — Force displacement curve and determination of true origin



Key

X gauge length L_0 (mm)

Y $\bar{C}_{t,0}$

Figure 8 — Determination of load train compliance

When the displacement is measured directly on the tow, $C_1 = 0$.

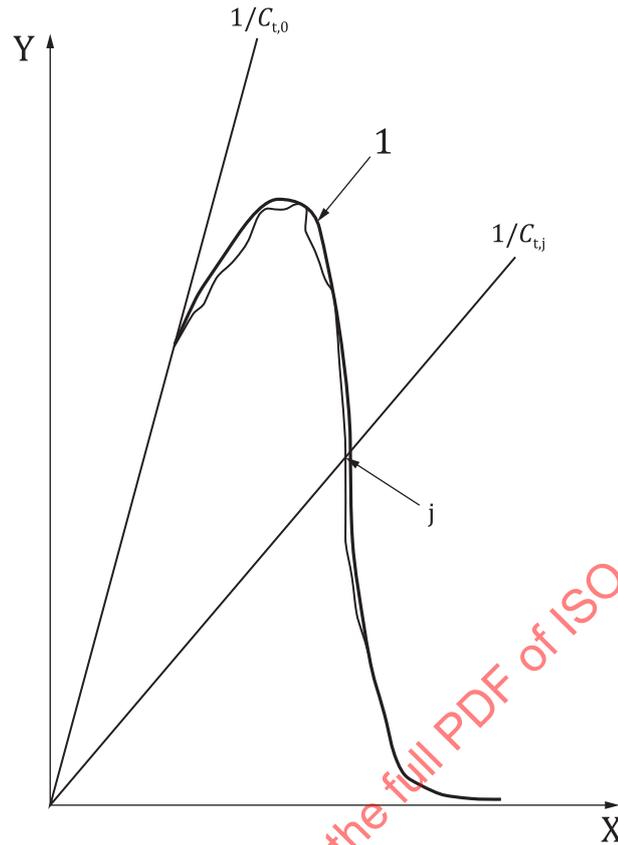
10.2 Calculation of probability of filament rupture P_j from the tests on specimens with a gauge length of 200 mm

10.2.1 Determination of the true origin

The true origin of the force-displacement curve is defined by the intersection of the linear rising part of the curve with the abscissa (see [Figure 7](#)).

10.2.2 Construction of envelope curve and determination of instantaneous compliance $C_{t,j}$

For each test draw the envelope to the nonlinear part of the force-displacement curve containing all the local maxima. Select at least 30 points on the envelope curve uniformly distributed along the abscissa, excluding the linear part (see [Figure 9](#)). For each point calculate $C_{t,j}$ from the slope $1/C_{t,j}$ of the line through the point j and the true origin of the force-displacement curve.

**Key**

- X force F (N)
 Y displacement (mm)
 1 envelope

Figure 9 — Construction of the envelope curve and instantaneous compliance

10.2.3 Probability of filament rupture

Calculate the probability of filament rupture P_j using [Formula \(1\)](#).

$$P_j = \frac{C_{t,j} - C_{t,0}}{C_{t,j} - C_1} \quad (1)$$

where

- P_j is the probability of filament rupture at step j ;
 $C_{t,j}$ is the total compliance at step j , in millimetres per newton (mm/N);
 $C_{t,0}$ is the initial total compliance determined from [10.1](#), in millimetres per newton (mm/N);
 C_1 is the load train compliance calculated from [10.1](#), in millimetres per newton (mm/N).

10.3 Distribution of filament rupture strain

10.3.1 Calculation of filament rupture strain

Calculate the filament rupture strain $\varepsilon_{r,j}$ for each point j using [Formula \(2\)](#).

$$\varepsilon_{r,j} = \frac{(C_{t,j} - C_1)F_j}{L_0} \quad (2)$$

where

- $\varepsilon_{r,j}$ is the filament rupture strain at step j ;
- $C_{t,j}$ is the total compliance at step j determined from [10.2.2](#), in millimetres per newton (mm/N);
- F_j is the force applied on the specimen at step j , in newtons (N);
- C_1 is the load train compliance calculated from [10.1](#), in millimetres per newton (mm/N);
- L_0 is the gauge length, in millimetres (mm).

10.3.2 Filament rupture strain distribution

To plot the consistent experimental distribution of filament failure strain data, the following procedure is recommended. Assign to each failure strain data obtained from laboratory testing a probability of failure P_j given by [Formula \(1\)](#). To create a graph representing the data P_j as a function of $\varepsilon_{r,j}$, plot P_j as the ordinate and $\varepsilon_{r,j}$ as the abscissa. The ordinate axis shall be labelled as probability of failure P_f . Similarly, the abscissa shall be labelled as failure strain.

For each test i ($i = 1, 2, 3$) with a gauge length of 200 mm, determine the shape parameter (modulus) and the scale parameter of the two-parameter Weibull distribution of the filament rupture strain distribution according to [Formula \(3\)](#):

$$P = 1 - \exp \left[- \frac{SL_0}{V_0} \left(\frac{\varepsilon}{\varepsilon_0} \right)^m \right] \quad (3)$$

where

- P is the probability of filament rupture;
- ε is the filament strain;
- ε_0 is the scale parameter;
- S is the initial cross-section area of the filament, in square millimetres (mm²);
- L_0 is the gauge length, in millimetres (mm);
- V_0 is the reference volume, in cubic millimetres (mm³).

The estimated statistical parameters are such that the values of probability of fracture calculated from theoretical [Formula \(3\)](#) fit the cumulative distribution of failure strains constructed using experimental strain data [[Formula \(2\)](#)] and associated values of probability given by [Formula \(1\)](#). Various fitting techniques can be used, like the method of least squares. The best fit in the least-squares sense minimizes the sum of squared residuals, a residual being the difference between an observed value and the fitted value provided by a model.

For the purposes of this document the determination of the confidence intervals on the Weibull parameters may be omitted. Instead, the average values \bar{m}_ε and $\bar{\varepsilon}_0$ are determined from the three tests.

10.4 Distribution of filament strength

10.4.1 Initial cross-section area

Calculate the initial cross-section area of the tow according to [Formula \(4\)](#).

$$S_0 = 10^{-3} \frac{t}{\rho} \quad (4)$$

where

S_0 is the initial cross-section area of the tow, in square millimetres (mm²);

t is the linear density of the fibre in tex (grams per thousand metres) determined from EN 1007-2;

ρ is the density in grams per cubic centimetre (g/cm³) according to ISO 10119.

[Formula \(3\)](#) does not apply when image analysis is used for the determination of S_0 .

10.4.2 Calculation of filament strength

Calculate the filament strength corresponding to each point j using [Formula \(5\)](#).

$$\sigma_{r,j} = \frac{F_j}{S_0 (1 - P_j)} \quad (5)$$

where

$\sigma_{r,j}$ is the filament strength at step j , in megapascals (MPa);

F_j is the force applied to the specimen at step j , in newtons (N);

S_0 is the initial cross-section area of the tow from [10.4.1](#), in square millimetres (mm²);

P_j is the probability of filament rupture at step j determined from [10.2.3](#).

10.4.3 Filament strength distribution

To plot the consistent experimental distribution of strength data, the following procedure is recommended. Assign to each strength data obtained from laboratory testing a probability of failure P_j given by [Formula \(1\)](#). To create a graph representing the data P_j as a function of $\sigma_{r,j}$, plot P_j as the ordinate and $\sigma_{r,j}$ as the abscissa. The ordinate axis shall be labelled as probability of failure P_f . Similarly, the abscissa shall be labelled as failure stress, preferably using units of MPa.

For each test i ($i = 1, 2, 3$) with a gauge length of 200 mm, determine the shape parameter (modulus) and the scale parameter of the two-parameter Weibull distribution of the filament strength according to [Formula \(6\)](#):

$$P = 1 - \exp \left[- \frac{SL_0}{V_0} \left(\frac{\sigma}{\sigma_0} \right)^m \right] \quad (6)$$

where

P is the probability of filament rupture;

σ is the filament strength, in megapascals (MPa);

σ_0 is the scale parameter in megapascals (MPa);