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Advanced technical ceramic - Ceramic composites - Methods of test for reinforcements - Part 6: Determination of tensile properties of filaments at high temperature

Hochleistungskeramik - Keramische Verbundwerkstoffe - Verfahren zur Prüfung der Faserverstärkungen - Teil 6: Bestimmung der Zugeigenschaften von Fasern bei hoher Temperatur

Céramiques techniques avancées - Céramiques composites - Méthodes d'essai pour renforts - Partie 6 : Détermination des propriétés en traction du filament à haute température

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Foreword

This document (EN 1007-6:2007) has been prepared by Technical Committee CEN/TC 184 “Advanced technical ceramics”, the secretariat of which is held by BSI.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by May 2008, and conflicting national standards shall be withdrawn at the latest by May 2008.

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1 Scope

This European Standard specifies the conditions for measurement of tensile properties a of single filament of ceramic fibres at high temperatures in air or inert atmosphere (vacuum or controlled atmosphere). The method applies to continuous ceramic filaments taken from tows, yarns, staple fibre, braids and knitting, that have strain to fracture less than or equal to 5 % and show linear elastic behaviour to fracture.

The method does not apply to testing for homogeneity of strength properties of fibres, nor does it assess the effects of volume under stress. Statistical aspects of fibre failure are not included.

Two methods are proposed depending on the temperature of the filament end:

— Hot end method: this method allows determination of tensile strength, of Young's modulus and of the stress strain curve.

NOTE 1 Current experience with this technique is limited to 1 300 °C, because of the application temperature of ceramic glue.

— Cold end method.

NOTE 2 This method is limited to 1 700 °C in air and 2 000 °C in inert atmosphere because of the limits of furnaces.

2 Normative references

The following referenced documents are indispensable for the application 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.

EN 1007-3, *Advanced technical ceramics — Ceramic composites — Methods of test for reinforcement — Part 3: Determination of filament diameter and cross-section area*

EN 1007-4, *Advanced technical ceramics — Ceramic composites — Methods of test for reinforcement — Part 4: Determination of tensile properties of filaments at ambient temperature*

EN ISO 7500-1, *Metallic materials - Verification of static uniaxial testing machines - Part 1: Tension/compression testing machines - Verification and calibration of the force-measuring system (ISO 7500-1:2004)*

EN ISO/IEC 17025, *General requirements for the competence of testing and calibration laboratories (ISO/IEC 17025:2005)*

EN 60584-1, *Thermocouples — Part 1: Reference tables (IEC 60584-1:1995)*

EN 60584-2, *Thermocouples — Part 2: Tolerances (IEC 60584-2:1982 + A1:1989)*

3 Terms and definitions

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

3.1
test temperature
T
temperature of the filament at the centre of the gauge length

3.2 Lengths

3.2.1
gauge length
L_o
initial distance between two reference points on the filament, where the temperature variation is within 20 °C at test temperature

3.2.2
test specimen length
L_f
initial distance between the gripped ends of the filament

3.2.3
uniformly heated length
L_h
length of the heated zone at the test temperature, where the temperature variation is within 20 °C (see Figure A.2)

3.2.4
gradient zone length
L_d
length of each part of the test specimen where the temperature decreases from the temperature at the end of the uniformly heated length to room temperature (see Figure A.2)

3.2.5
room temperature zone length
Lc
length of each part of the test specimen where the temperature is equal to room temperature

3.3
initial cross-section area
A_o
initial cross-section area of the filament within the gauge length determined at room temperature

3.4
maximum tensile force
Fm
highest recorded tensile force on the test specimen when tested to failure

3.5
tensile stress
σ
tensile force supported by the test specimen divided by the initial cross-section area

3.6
tensile strength
σ_m
ratio of the maximum tensile force to the initial cross-section area

3.7
longitudinal deformation
ΔL
increase of the gauge length during the tensile test

3.8 Compliance

3.8.1
total compliance
C_t
reciprocal of the slope in the linear part of the force/displacement curve

3.8.2
load train compliance
C_l
ratio of the force displacement excluding any test specimen contribution to the corresponding force during the tensile test

3.8.3
gradient zone compliance
C_d
ratio of the test specimen elongation in the temperature gradient zone length L_d to the corresponding force during the tensile test

3.8.4
cold zone compliance
C_c
ratio of the test specimen elongation at room temperature Lc to the corresponding force during the tensile test

3.8.5
hot zone compliance
C_h
ratio of the test specimen elongation in the uniformly heated length L_h to the corresponding force during the tensile test

3.9
strain
ε
ratio of the longitudinal deformation to the gauge length

3.10
fracture strain
ε_m
strain at failure of the test specimen

3.11
elastic modulus
E
slope of the linear part of the tensile stress-strain curve

4 Principle

A ceramic filament is heated to the test temperature and loaded in tension. The test is performed at constant force/displacement rate up to failure. Force and cross-head displacement are measured and recorded simultaneously. When required, the elongation is derived from the cross-head displacement using a compliance correction. The test duration is limited to reduce time dependent effects.

Subjecting the whole length of a fibre to temperatures well above 1 000 °C makes it difficult to fix the ends of the specimen into appropriate temperature proof extensions. In high temperature cold end tests this problem is avoided by keeping the junction at the ends of the test specimen at room temperature, allowing organic resins to be used as in the room temperature tests (EN 1007-4).

Two methods can thus be used:

— one consists of heating the filament over its total length (hot end method);

— one consists of heating only the central part of the filament (cold end method).

5 Apparatus

5.1 Test machine

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

The machine shall be equipped with a system for measuring the force displacement. The accuracy of the measurement shall be better than 1 µm.

5.2 Load train

The grips shall align the specimen with the direction of the force. Slippage of the filament in the grips shall be prevented. The load train performance including the alignment system and the force transmitting system shall not change because of heating.

5.3 Adhesive

Use a suitable adhesive for affixing the filament to the ends of the grip, such as epoxy resin, cement or sealing wax.

5.4 Test chamber

5.4.1 General

When testing under inert conditions, a gastight chamber allows proper control of the test environment during the test. The installation shall be such that the variation of the load due to the variation of pressure is less than 1 % of the scale of the load cell being used.

5.4.2 Gas atmosphere

The gas atmosphere shall be chosen depending on the material to be tested and on the test temperature. The level of pressure shall be chosen depending on the material to be tested, on the test temperature and on the type of gas.

5.4.3 Vacuum chamber

The level of vacuum shall not induce chemical and/or physical instabilities of the filament material.

5.5 Set-up for heating

The set-up for heating shall be constructed in such a way that the variation of temperature within the gauge length is less than 20 °C at test temperature.

5.6 Temperature measurement

Thermocouples shall comply with EN 60584-1 and EN 60584-2.

Alternatively, pyrometers or thermocouples which are not covered by EN 60584-1 and EN 60584-2, but which are appropriately calibrated, can be used.

5.7 Data recording system

Calibrated recorders may be used to record force-displacement curves.

The use of a digital data recording system combined with an analogue recorder is recommended.

6 Hot end method

6.1 General

In high temperature the test specimen strain can be determined in simple analogy to the room temperature method assuming that the test specimen sees isothermal conditions along its whole length. According to this hypothesis, the gauge length L_0 is equal to the test specimen length L_f.

6.2 Test specimens

Specimens with a gauge length of 25 mm shall be used to establish the force-displacement curves.

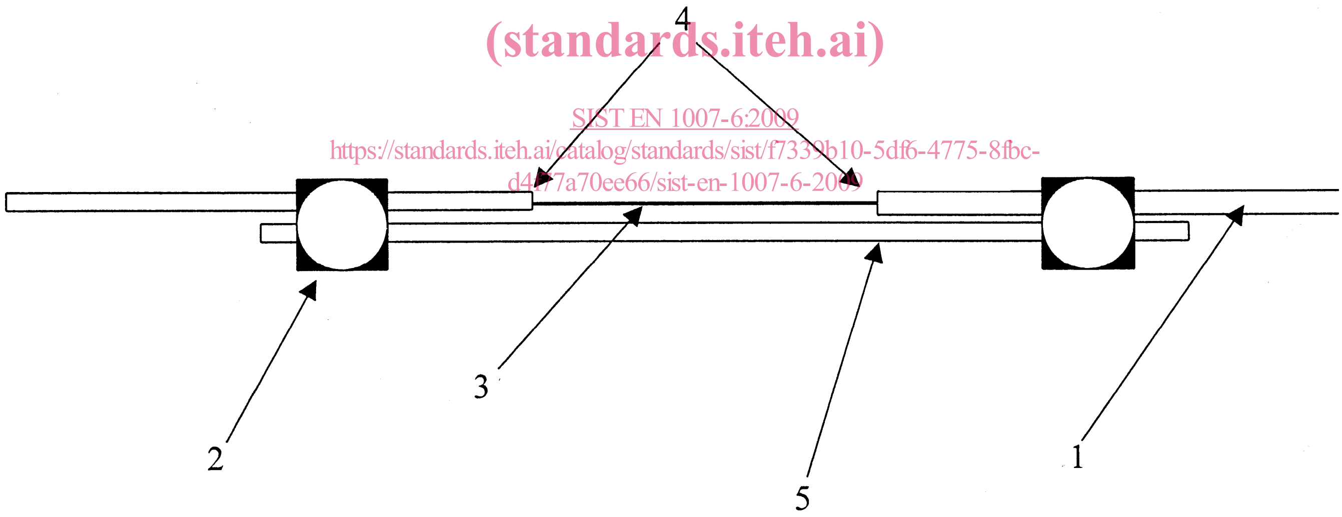
Specimens with a gauge length of 10 mm and 40 mm shall be used to determine the load train compliance C_l.

The tolerance on the gauge length is ± 1 mm.

6.3 Test specimen preparation

Extreme care shall be taken during test specimen preparation to ensure that the procedure is repeatable from test specimen to test specimen and to avoid handling damage.

NOTE As an example to prevent damage during *test* specimen manipulation and mounting, the assembly of test specimen and alumina tubes is maintained straight by an extra alumina rod, as shown in Figure 1.



key

1 alumina tubes

2 temporary screw attachment

3 test specimen

4 high temperature joints between the test specimen and the alumina tubes

5 alumina rod

Figure 1 — Test specimen assembly

6.4 Number of test specimens

For each test condition, five valid test results at a gauge length of 25 mm, are required.

For the determination of strain related properties, three additional tests at each gauge length of 10 mm and 40 mm are required in order to establish load-train compliance, C_L.

NOTE 1 If a statistical evaluation is required, the number of test specimens at a gauge length of 25 mm should be in accordance with EN 843-5.

NOTE 2 A compliance determination is not required if only strength needs to be determined.

6.5 Test procedure

6.5.1 Test set-up: determination of the temperature profile

The following determinations shall be carried out under actual test conditions.

Prior to testing, the temperature profile inside the furnace shall be established over the temperature range of interest. This shall be done by measuring the temperature at a minimum of three locations that correspond to the ends and the centre of the maximum gauge length.

NOTE 1 When the type of specimen assembly described in Figure 1 is used, the temperature profile may be determined inside the furnace at the end and at mid-way between the tubes positioned at the distance corresponding to the maximum gauge length and without the filament mounted.

During a series of tests, the test temperature is determined indirectly from the temperature indicated by the temperature control device.

The relation between the control temperature and the test temperature is established over the range of temperature of interest.

NOTE 2 Usually the determination of the temperature profile and the relation between control temperature and test temperature are established simultaneously.

6.5.2 Test set-up: other considerations

6.5.2.1 General

The dimension of the filament varies with the temperature and the variation is very difficult to measure.

6.5.2.2 Determination of the gauge length, L_0

The gauge length is measured to an accuracy of ± 0,1 mm at room temperature.

6.5.2.3 Determination of the initial cross-section area, A_0

The filament diameter and thus the initial cross-section area at test temperature, is measured at room temperature in accordance with EN 1007-3.

NOTE 1 In principle, the initial cross-section area is determined on the filament to be tested. In practice, this can be achieved by sampling the lengths to be tested from a single filament at intermittent locations and using the parts in between for diameter measurement. This assumes that for the lengths of fibres to be tested, the diameter does not vary significantly with the length.

NOTE 2 An alternative method consists of measuring the filament cross-section after fracture from a transverse cross-section taken from the part of the grips still containing embedded fibre. In this case, care is taken not to damage the fibre during preparation.

6.5.3 Testing technique

6.5.3.1 General

Follow the chronological steps.

6.5.3.2 Zero the load cell

6.5.3.3 Specimen mounting

Mount the specimen in the load train with its longitudinal axis coinciding with that of the test machine. Care shall be taken not to induce torsional loads or surface damage to the filament. The position of the gauge length relative to the furnace shall be identical to that previously used in 6.5.1.

6.5.3.4 Setting of the controlled atmosphere

When testing in inert gas, air and water vapour shall be removed before setting the inert atmosphere. This can be done by establishing vacuum (below 10 Pa) in the enclosure, or by circulating inert gas.

When testing under vacuum, the vacuum level shall be according to 5.4.3.

NOTE In view of the extreme oxidation sensitivity of some of the filament material, conventional flushing of the test chamber might not be sufficient to reduce the oxygen level below acceptable limits.

6.5.3.5 Heating of test specimen

Raise the test specimen temperature to the required test temperature and maintain this test temperature for a short period to allow for temperature stabilisation. The test specimen temperature is the furnace temperature. Ensure that the test specimen stays in the initial state of stress during heating.

6.5.3.6 Measurements

- Record temperature.
- Record vacuum or gas pressure if applicable.
- Set the cross-head speed.
- Record the force versus force/cross-head displacement curve up to failure.
- Cool down until the risk of degradation is removed before opening the test chamber.

6.5.3.7 Test validity

The following circumstances invalidate the test:

- failure to specify and record test conditions;
- any slippage in the load train as evidenced by a drop in the force/displacement curve, before reaching the maximum tensile force;
- any deviation from linearity in the load/cross-head displacement curve after the initial slack has been taken up.

The following circumstance invalidates only the strength and stress to failure:

- failure at the grip(s): see Figure 2.