SIST

SLOVENSKI STANDARD
SIST EN 1007-7:2010

01-oktober-2010

Nadomešča:

SIST-TS CEN/TS 1007-7:2007

Sodobna tehnična keramika - Keramični kompoziti - Preskusne metode za ojačitve - 7. del: Ugotavljanje porazdelitve natezne trdnosti in deformacij/obremenitev vlaken v svežnjih pri visoki temperaturi

Advanced technical ceramics - Ceramic composites - Methods of test for reinforcements - Part 7: Determination of the distribution of tensile strength and of tensile strain to failure of filaments within a multifilament tow at high temperature

Hochleistungskeramik - Keramische Verbundwerkstoffe - Verfahren zur Prüfung der Faserverstärkungen - Teil 7: Bestimmung der Verteilung von Zugfestigkeit und Zugdehnung von Fasern im Faserbündel bei hoher Temperatur

Céramiques techniques avancées - Céramiques composites - Méthodes d'essai pour renforts - Partie 7: Détermination de la distribution de la résistance en traction et de la déformation de traction à la rupture des filaments dans un fil à haute température

Ta slovenski standard je istoveten z: **EN 1007-7:2010**

ICS:

81.060.30	Sodobna keramika	Advanced ceramics

SIST EN 1007-7:2010 **en**

EUROPEAN STANDARD

NORME EUROPÉENNE

EUROPÄISCHE NORM

EN 1007-7

July 2010

ICS 81.060.30

Supersedes CEN/TS 1007-7:2006

English Version

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COMITÉ EUROPÉEN DE NORMALISATION
EUROPÄISCHES KOMITEE FÜR NORMUNG

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Ref. No. EN 1007-7:2010: E

Contents

Page

Foreword

This document (EN 1007-7:2010) 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of an identical text or by endorsement, at the latest by January 2011, and conflicting national standards shall be withdrawn at the latest by January 2011.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. CEN [and/or CENELEC] shall not be held responsible for identifying any or all such patent rights.

This document supersedes CEN/TS 1007-7:2006.

EN 1007, *Advanced technical ceramics — Ceramic composites. Methods of test for reinforcements*, has been prepared in 7 parts:

— *Part 1: Determination of size content*;

— *Part 2: Determination of linear density*;

— *Part 3: Determination of filament diameter and cross-section area*;

— *Part 4: Determination of tensile properties of filaments at ambient temperature*;

— *Part 5: Determination of distribution of tensile strength and of tensile strain to failure of filaments within a multifilament tow at ambient temperature*;

— *Part 6: Determination of tensile properties of filaments at high temperature*;

— *Part 7: Determination of the distribution of tensile strength and tensile strain to failure of filaments within a multifilament tow at high temperature*.

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard: Austria, Belgium, Bulgaria, Croatia, Cyprus, Czech Republic, Denmark, Estonia, Finland, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Romania, Slovakia, Slovenia, Spain, Sweden, Switzerland and the United Kingdom.

1 Scope

This European standard specifies the conditions, apparatus and procedure for determining the distribution of tensile strength and tensile strain to failure of ceramic filaments in multifilament tows at high temperature in air, vacuum or a controlled inert atmosphere.

This part of EN 1007 applies to tows of continuous ceramic filaments, which are assumed to act freely and independently under loading and behave linearly elastic up to failure.

Two methods are proposed depending on the temperature of the ends of the tow:

a) hot end method;

NOTE 1 The application of the hot end method is restricted by ceramic glues with sufficient shear strengths at the test temperature. Current experience with this technique is limited to 1 300 °C, because of the maximum application temperature of ceramic glues.

b) cold end method.

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

Both methods allow for a failure rate in the determination of distribution of tensile strain and tensile strength.

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

CEN/TR 13233:2007, *Advanced technical ceramics — Notations and symbols*

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)*

EN ISO 7500-1:2004, *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)*

ISO 10119, *Carbon fibre — Determination of density*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in CEN/TR 13233:2007 and the following apply.

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

3.2
lengths
initial distances between two reference points on the tow, disregarding thermal and mechanical strains

3.2.1
gauge length
L_0
part of the tow between the gripped ends, where the temperature variation is within 20 K of the test temperature

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

3.2.3
uniformly heated length
L_h
length of the heated zone within which the temperature variation is within 20 K of the test temperature

3.2.4
cold zone length
L_c
length of the tow, which is not uniformly heated

3.3
initial cross sectional area
A_0
sum of the cross sectional areas of all the filaments in the tow

3.4
tow elongation
ΔL
increase of the gauge length between the two reference points on the tow

3.5
tow strain
ε
ratio of the tow elongation ΔL to the gauge length L_o

3.6
tow maximum tensile force
F_{tow}
highest recorded tensile force on the test specimen when tested to failure

3.7
tow strength
σ_{tow}
ratio of the tow maximum tensile force to the cross sectional area of all unbroken filaments at maximum tensile force, F_{tow}

3.8
force at step *j*
F_j
force applied on the test specimen at step j

3.9
filament strain
ε_j
strain at step *j* in the non-linear parts of the force-displacement curve

3.10
filament strength
σ_j
ratio of the tensile force to the cross sectional area of all unbroken filaments at step *j* in the non-linear parts of the force-displacement curve

3.11
average filament rupture strain
$\overline{\varepsilon_r}$

statistical average rupture strain of the filaments in the tow for each test determined from the Weibull strain distribution parameters of the filaments

3.12

overall average filament rupture strain
$\overline{\overline{\varepsilon_r}}$
arithmetic mean of the average filament rupture strains

3.13
average filament strength
$\overline{\sigma_r}$
statistical average strength of the filaments in the tow for each test determined from the Weibull strength distribution parameters of the filaments

3.14
overall average filament strength
$\overline{\overline{\sigma_r}}$
arithmetic mean of the average filament strengths

3.15 Compliances

3.15.1
initial total compliance
C_t
inverse slope of the linear part of the force-displacement curve

3.15.2
instantaneous total compliance
$C_{t,j}$
inverse slope of the secant at any point *j* in the non-linear part of the force-displacement curve

NOTE The slope is taken from a line through any point of the force-displacement curve and the intersection point of the line of the initial total compliance with the abscissa (true origin).

3.15.3
load train compliance
C_l
ratio of the cross head displacement to the force, excluding any contribution of the test specimen to the displacement during the tensile test

3.15.4
cold zone compliance
C_c
ratio of the increase in test specimen length in the cold zone length L_c to the corresponding force during the tensile test

3.15.5
hot zone compliance
C_h
ratio of the increase in test specimen length in the uniformly heated length L_h to the corresponding force during the tensile test

4 Symbols and abbreviations

C_{tow} is the instantaneous total compliance of the tow at maximum tensile force.

E_h is the elastic modulus (Young's modulus) of the uniformly heated part of the tow.

E_c is the elastic modulus (Young's modulus) of the cold part of the tow.

5 Principle

A multifilament tow is heated to the test temperature and loaded in tension. The test is performed at a constant displacement rate up to failure. Force and cross-head displacement are measured and recorded simultaneously. When required, the longitudinal deformation is derived from the cross-head displacement using a compliance correction. From the force-displacement curve, the two-parameter Weibull distribution of the rupture strain and distribution of the rupture strength of the filaments is obtained by sampling the non-linear parts of the curve at discrete intervals *j*, which correspond to an increasing number of failed filaments in the tow. The test duration is limited to reduce time dependent effects.

Two methods can be used. The first method (hot-end method) consists of heating the tow over its total length. The second method (cold end method) consists of heating only a part of the test specimen length, the temperature profile of which is used to define the gauge length. The application of this method requires the realisation of three different heated zone lengths.

6 Significance and use

The measurement of strain directly on the tow is difficult, so it is usually achieved indirectly via a compliance measurement which includes contributions of the loading train, grips, tab materials, etc. These contributions shall be taken into account to achieve a correct 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 7 also applies in this case by setting the load train compliance equal to zero.

The evaluation method is based on an analysis of the non-linear increasing and decreasing parts of the force displacement curve. These parts are caused by progressive filament failures during the test, which is encouraged by a high stiffness of the loading and gripping system. When the force-displacement curve does not show these non-linear parts, the evaluation method of this part of EN 1007 cannot be applied.

The distribution of filament failure strains does not depend on the number of filaments in the tow and is hence not affected by the number of filaments which are broken before the test. The determination of the filament strength distribution requires knowledge of the initial cross sectional area of the tow and because the number of unbroken filaments within the tow prior to the test is usually unknown, the values for the filament strength represent a lower bound to these quantities. Also, the variation in the filament diameter, which affects the strength values, is not accounted for.

a) Hot end method: for the hot end method, the gauge length, defined as the uniformly heated length, equals the test specimen length.

NOTE 1 Subjecting the whole length of a tow to temperatures well above 1 000 °C, however, makes it difficult to fix the ends of the test specimen into appropriate temperature proof extensions.

b) Cold end method: for the cold end method, the test specimen length is the sum of the cold and the hot parts of the tow, with the gauge length is defined as the uniformly heated length. The temperature gradient zones along the tow axis are neglected.

NOTE 2 In this method, the problems associated with heating the clamps are avoided by heating only a central part of the tow and by keeping the junction at the ends of the test specimen at room temperature. This allows similar test specimen designs and organic resins to be used as in the room temperature test method (EN 1007-5). The interpretation of the results can be complicated by the superposition of the contributions from the cold and the hot tow zones.

The ratio of the hot part of the tow and the test specimen length is to be adjusted so as to keep the grips at room temperature, whilst the uniformly heated zone shall not be too short, in order to ensure a significant influence of the hot part on the overall failure behaviour of the tow.

7 Apparatus

7.1 Test machine

The test machine shall be equipped with a system for measuring the force applied to the test specimen, which shall conform to grade 1 according to EN ISO 7500-1:2004. Additionally, the machine shall be equipped with a system for measuring the cross head displacement with an accuracy better than 1 µm.

7.2 Load train

The grips shall align the test specimen with the direction of the force. Slippage of the test specimen in the grips shall be prevented as well as avoiding pre-damage due to gripping. The load train performance including the alignment system and the force transmitting system shall not change because of heating.

7.3 Adhesive

A suitable adhesive for fixing the tow ends to the grip, such as ceramic cement (hot-end method) or epoxy resin (cold-end method).

7.4 Test chamber

7.4.1 General

When testing under inert conditions, a gas-tight chamber allows for proper control of the test environment. 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.

7.4.2 Gaseous environment

The gaseous environment shall be chosen depending on the material to be tested and on the test temperature. The level of pressure shall be chosen based on the material to be tested, the test temperature, and on the type of gas. The gaseous environment shall not induce chemical and/or physical instability to the filament material.

7.4.3 Vacuum chamber

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

7.5 Set-up for heating

The set-up for heating shall be constructed in such a way that in a sufficiently extended gauge length the temperature variation is minimised and less than 20 K at the test temperature.

7.6 Temperature measurement

For temperature measurement, either thermocouples conforming to EN 60584-1 and -2 shall be used or, where thermocouples not conforming to EN 60584 or pyrometers are used, they shall be appropriately calibrated.

7.7 Data recording system

A calibrated recorder may be used to record force-displacement curves. The use of a digital data recording system combined with an analogue recorder is recommended.

8 Test procedure

8.1 Test specimens

— Hot end method

In high temperature hot end tests, it is assumed that the test specimen is exposed to isothermal conditions along its whole length and that the test temperature is equal to the furnace temperature. According to these hypotheses, the gauge length L_0 is equal to the test specimen length L_f.

Test specimens with a gauge length of 100 mm shall be used to establish force-displacement curves. To determine the load train compliance C_l additional test specimens with gauge lengths of 50 mm and 150 mm shall be used.

The gauge length shall be measured with an accuracy of ± 1 mm.