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Advanced technical ceramics — Mechanical properties of ceramic fibres at high temperature in a non-reactive environment — Determination of creep behaviour by the cold end method

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The UK participation in its preparation was entrusted to Technical Committee RPI/13, Advanced technical ceramics.

A list of organizations represented on this committee can be obtained on request to its secretary.

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Advanced technical ceramics - Mechanical properties of ceramic fibres at high temperature in a non-reactive environment - Determination of creep behaviour by the cold end method

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Hochleistungskeramik - Mechanische Eigenschaften von Keramikfasern bei hohen Temperaturen in einer reaktionsfreien Umgebung - Bestimmung des Kriechverhaltens im Kaltverbindungsverfahren

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Contents

Page

Foreword 3

1 Scope 4

2 Normative references 4

3 Terms and definitions 4

4 Principle 7

5 Significance and use 8

6 Apparatus 8
6.1 Test installations 8
6.2 Load train 8
6.3 Test chamber 9
6.4 Set-up for heating 9
6.5 Temperature measurement 9
6.6 Control of deformation 9
6.7 Data recording system 9
6.8 Determination of fibre cross sectional area 9

7 Test specimens 9
7.1 Test specimen preparation 9
7.2 Number of test specimens 11

8 Test procedures 11
8.1 Determination of the temperature profile in the furnace 11
8.2 Test set-up: Determination of the temperature profile and of the different lengths of each temperature zone in the furnace 11
8.3 Test set-up: Loading considerations 12
8.4 Test technique 12
8.5 Test validity 14

9 Calculation of results 14
9.1 Creep stress 14
9.2 Creep strain at time t 14

10 Test report 15

Bibliography 17

Foreword

This document (EN 15365:2010) 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of an identical text or by endorsement, at the latest by January 2011, and conflicting national standards shall be withdrawn at the latest by January 2011.

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1 Scope

This European Standard specifies the conditions for the determination of the tensile creep deformation and failure behaviour of single filaments of ceramic fibres at high temperature and under test conditions that prevent changes to the material as a result of chemical reaction with the test environment.

This European Standard applies to continuous ceramic filaments taken from tows, yarns, braids and knittings, which have strains to fracture less than or equal to 5 %.

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 60584 (all parts), *Thermocouples*

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

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
creep
time-dependent increase of gauge length starting from the time when the constant specified level of force is reached

3.2
creep threshold temperature
T_t
minimum temperature at which creep is detected

3.3
specimen temperature
T
temperature which varies along the fibre length in the cold grips case

NOTE See 8.2.

3.4
specimen temperature in the zone
T_i
temperature defined as: $T_t \leq T_i \leq T_t + i\,\Delta T$

3.5
total length
L
total length of the ceramic filament between the grips

3.6
length
L_i
length of the ceramic filament at temperature T_i

3.7
initial effective cross sectional area
A_0
initial cross sectional area of the ceramic filament within the gauge length

3.8
applied tensile force
F
constant force applied to the ceramic filament during the test

3.9
applied tensile stress
σ
applied tensile force divided by the initial cross sectional area

3.10
longitudinal deformation
ΔL
change in the total length of the ceramic filament caused by creep

3.11
longitudinal deformation
ΔL_i
change of the filament caused by creep at temperature T_i

3.12
tensile creep strain
$\varepsilon_{cr(T)}$
relative change in length in the controlled zone at time *t*, caused by creep at the temperature T

NOTE The value corresponding to rupture is denoted $\varepsilon_{cr,m}$.

3.13
creep rupture time
$t_{cr,m}$
time elapsed from the moment when loading is completed until the moment of rupture

3.14
creep strain rate
$\dot{\varepsilon}_{cr(T)}$
change in creep strain per unit time at time t at the temperature T_i

3.15
creep type
primary, secondary or tertiary creep

3.16
primary creep
part of the creep strain versus time curve which presents a decreasing creep strain rate

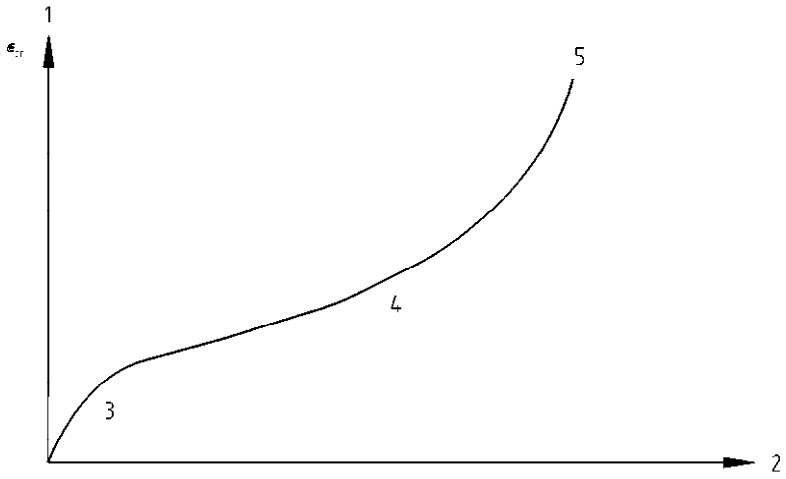
NOTE See Figure 1.

3.17
secondary creep
part of the creep strain versus time curve which presents a constant creep strain rate

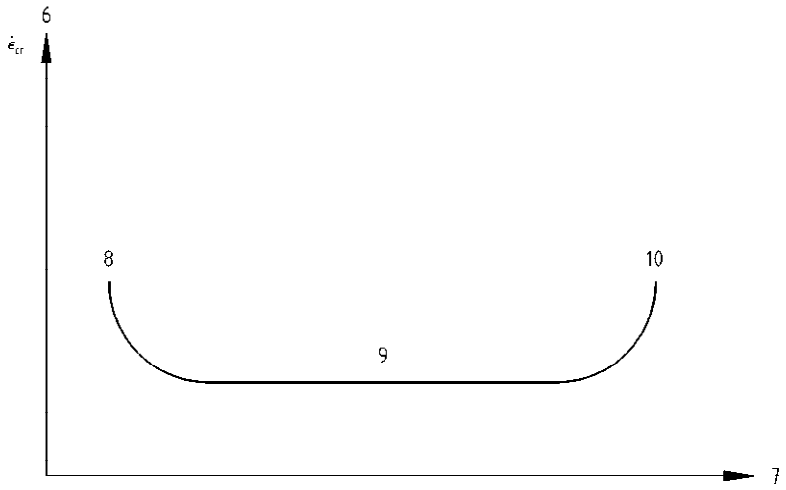
NOTE See Figure 1.

3.18
tertiary creep
part of the creep strain versus time curve which presents an increasing creep strain rate

NOTE See Figure 1.



a) Creep strain versus time



b) Creep strain rate versus time

Key

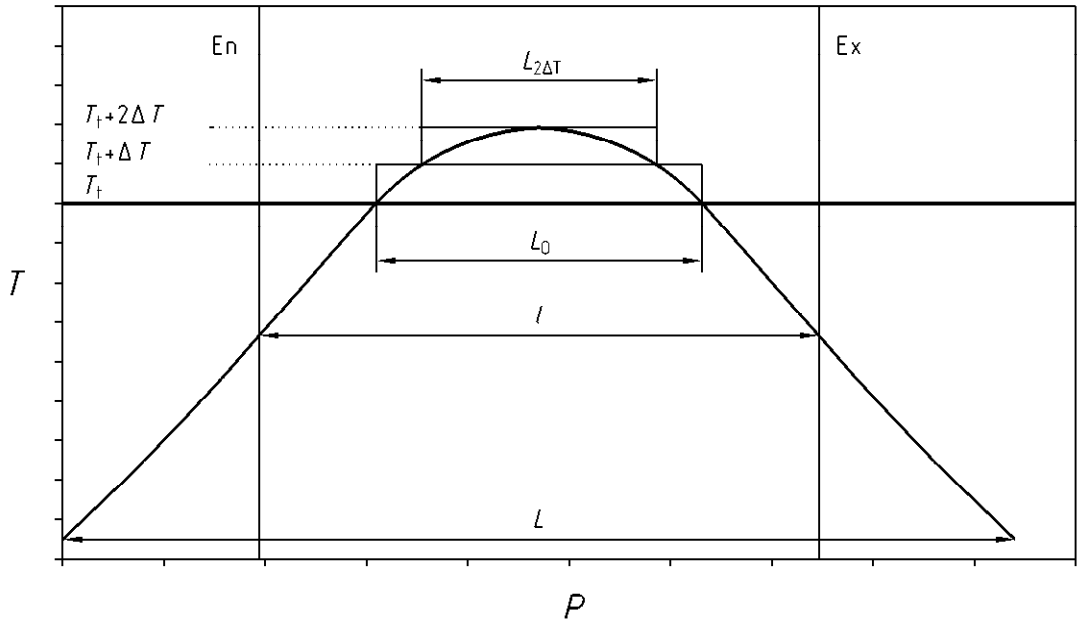
1 Creep strain ε_{cr}
2 Time t
3 Primary creep
4 Secondary creep
5 Tertiary creep
6 Creep strain rate $\dot{\varepsilon}_{cr}$ (creep strain with time)
7 Time
8 Primary creep
9 Secondary creep
10 Tertiary creep

Figure 1 — Creep strain and creep strain rate versus time curves

4 Principle

A ceramic filament is heated to the test temperature and loaded in tension until a specified level of force. This force is maintained at a constant level for a specified time or until rupture. The variation in the ceramic filament length is recorded in relation to time.

The specimen is held in cold grips and heated by a furnace. This experimental configuration provokes temperature variations along the filament, which have to be taken into account in order to determine the creep properties as function of temperature. Prior to testing, the temperature profile inside the furnace is established over the temperature range. The temperature range is then divided into several temperature zones defined by the operator, according to the following graph.



Key

T Temperature (°C)

l Length of the furnace

P Position (mm)

En Entrance

Ex Exit

L total length of the ceramic filament between the groups

$L_0 = L_{2\Delta T} + 2L_{\Delta T}$

where

$L_{2\Delta T}$ is the furnace length where the temperature T is in the range $T_t + \Delta T \leq T \leq T_t + 2\ \Delta T$;

$L_{\Delta T}$ is the furnace length where the temperature T is in the range $T_t \leq T \leq T_t + \Delta T$.

Figure 2 — Temperature profile in furnace

If T_t is considered to be the lowest temperature at which creep is observed, the temperature profile can be divided in several intervals as a function of T_t and ΔT, where ΔT is the difference in temperature between the different zones, fixed by the operator.

If we consider *i*, the entire number of zones, and L, the total fibre length, then we can define the following lengths:

— L_{20} is the furnace length where the temperature T is in the range 20 °C $\leq T \leq T_t$;

— $L_{\Delta T}$ is the furnace length where the temperature T is in the range $T_t \leq T \leq T_t + \Delta T$;

— $L_{2\Delta T}$ is the furnace length where the temperature T is in the range $T_t + \Delta T \leq T \leq T_t + 2\,\Delta T$;

— $L_{i\Delta T}$ is the furnace length where the temperature T is in the range $T_t + (i - 1)\,\Delta T \leq T \leq T_t + i\,\Delta T$.

Then L can be written:

$$L = L_{20} + L_{\Delta T} + L_{2\Delta T} + L_{3\Delta T} + \ldots + L_{i\Delta T} \quad (1)$$

Thus it is possible to determine the deformation in all of these different temperature zones. The inconvenience of this method is that determining the true deformation in the $L_{2\Delta T}$ zone requires the determination of the deformation in the lower temperature zones.

Below the temperature T_t and for a constant load applied to the fibre, the deformation is constant so that the strain rate is equal to zero.

5 Significance and use

Creep tests allow the comparison and the determination of parameters or behaviour laws and their extrapolation to long-term behaviour for different materials under constant load at high temperatures. These allow the conception and design of industrial parts with close control of tolerances for high temperature applications.

6 Apparatus

6.1 Test installations

NOTE Two different types of installation can be used, as specified in 6.1.1 and 6.1.2.

6.1.1 Test machine

The machine shall be equipped with a system for measuring the force applied to the test specimen. The machine shall have a load cell with a resolution of 10^{-3} N for the applied force. The displacement transducer shall have a resolution of at least 2 µ. This shall prevail during actual test conditions (pressure, temperature).

6.1.2 Creep testing rig

When a creep testing rig is used, the force application system shall be calibrated. The testing rig shall be equipped with a system to allow smooth loading of the ceramic filament(s). When this system is used, care shall be taken to ensure that the force applied to the ceramic filament remains constant to within 10^{-3} N, even when the material properties change and the environmental conditions (temperature, pressure) fluctuate.

6.2 Load train

The gripping system shall align the test specimen axis with that of the applied force.

The load train configuration shall ensure that the load indicated by the load cell and the load experienced by the test specimen are the same. The load train performance including the alignment and the force transmission shall not change because of heating.

6.3 Test chamber

6.3.1 General

The chamber shall allow proper control of the test specimen environment during the test and ensure that any variation of load during the test is less than 1 % of the scale of the load cell being used.

6.3.2 Gaseous environment

The gaseous environment shall be chosen depending on the material to be tested and on the test temperature. If the test is conducted in flowing gas, the rate of flow should be sufficiently high to exclude oxygen, but not so as to induce turbulence in the furnace. If a closed system is used the level of pressure shall be chosen depending on the material to be tested, on temperature, on the type of gas and on the type of extensometry.

6.3.3 Vacuum chamber

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

6.4 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 known to within 20 K.

NOTE Horizontal furnaces give a more symmetrical temperature profile than vertical devices.

6.5 Temperature measurement

For temperature measurement, either thermocouples conforming to EN 60584 (all parts) shall be used, or, where thermocouples not conforming to EN 60584 (all parts) or pyrometers are used, they shall be appropriately calibrated and the calibration data added to the test report.

6.6 Control of deformation

The deformation of the filament can be measured by the movement of the cross-head, with compensation being made for the compliance of the machine according to EN 1007-4. A direct technique for measuring the deformation of the filament is by speckle interferometry.

6.7 Data recording system

Calibrated recorders may be used to record force, longitudinal deformation and temperature versus time.

6.8 Determination of fibre cross sectional area

The fibre cross sectional area can be determined from a measurement of the fibre diameter, if the fibre is circular in section, or an average diameter can be determined if it is not. Measurements should conform to EN 1007-3.

7 Test specimens

7.1 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.

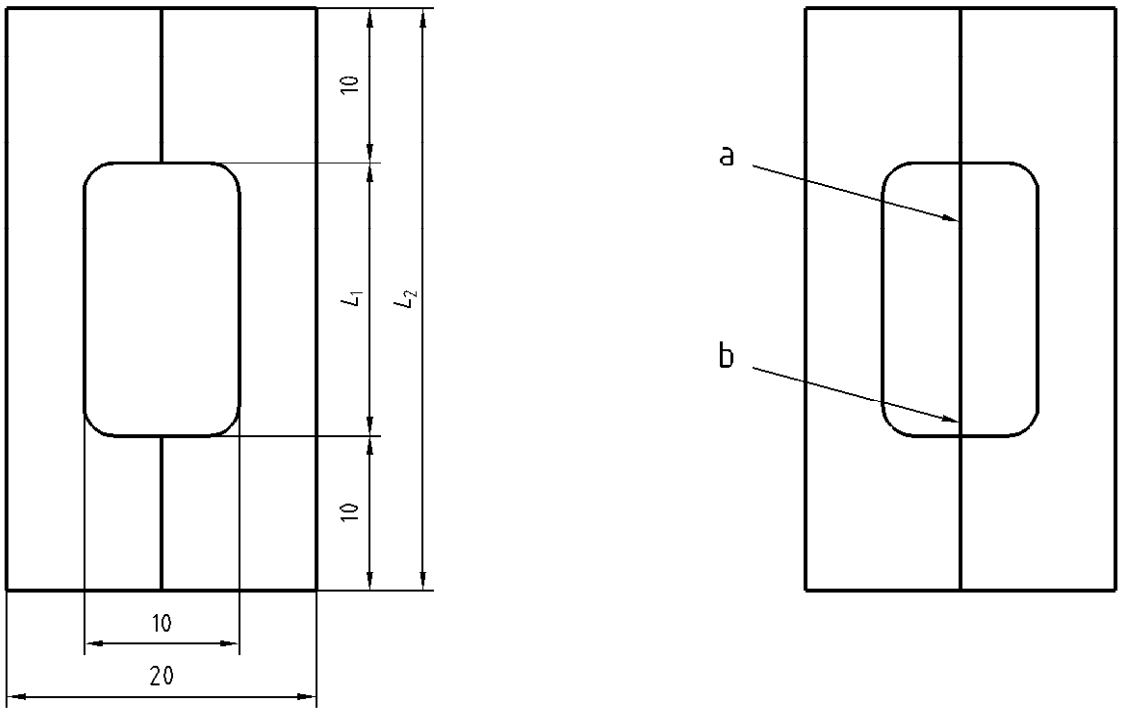
NOTE 1 The introduction of damage during test specimen preparation may result in a truncation of the strength distribution and is more critical the longer the length of the filament.

NOTE 2 During test specimen preparation and in particular when extracting a filament from the tow, the ratio of damaged filaments to the total number of extracted filaments should be minimised.

NOTE 3 To prevent damage during test specimen manipulation and mounting, an example of the assembly of a test specimen is shown in Figure 3. This test specimen preparation uses a mounting tab of thin paper, metal or plastic cut as shown in Figure 3, with a window. The length of the window is equal to the gauge length of the filament test specimen. An adhesive, suitable for affixing the filament to the ends of mounting tab, such as an epoxy resin, a cement or sealing wax, is used for this purpose.

NOTE 4 Another example of assembly, shown in Figure 4, can be used to prevent damage during test specimen manipulation and mounting.

Dimensions in millimetres



Key

a Filament

b Glue

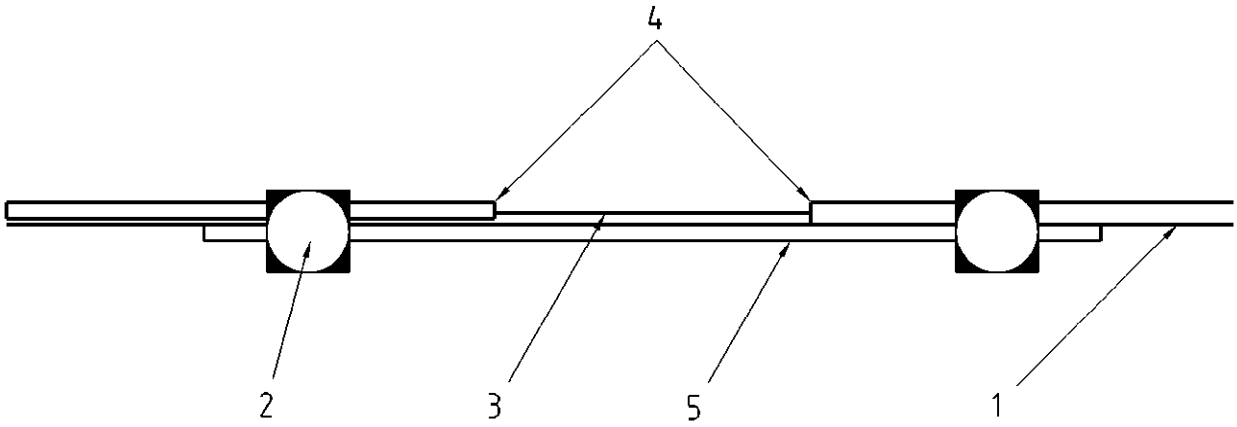
L_1 See Table 1 for dimensions

L_2 See Table 1 for dimensions

Figure 3 — Assembly of test specimen

Table 1 — Dimensions of L_1 and L_2

L_1 mm	L_2 mm
10 ± 0,5	30 ± 1
25 ± 0,5	45 ± 1
50 ± 0,5	70 ± 1



Key

1 Alumina tubes
2 Temporary screw attachment
3 Test specimen
4 Ceramic cement
5 Alumina rod

Figure 4 — Alternative test specimen assembly

7.2 Number of test specimens

At least three valid test results by temperature and by load are required.

8 Test procedures

8.1 Determination of the temperature profile in the furnace

The following procedures shall be carried out under conditions representative of the tests and shall be repeated every time there is a change in gripping configuration, etc.

8.2 Test set-up: Determination of the temperature profile and of the different lengths of each temperature zone in the furnace

Prior to testing, the temperature profile inside the furnace shall be established at each temperature considered. This shall be done by measuring the temperature at a minimum of ten locations distributed along the length of the empty furnace (Figure 2).

8.3 Test set-up: Loading considerations

For a given test temperature, it is recommended that the loading rate is set for the highest load level. Once these conditions are established they can be used for the lower load levels.

Loading shall be sufficiently fast to ensure that creep deformation does not add to the pure tensile deformation during the loading phase.

To determine the loading rate which minimises the creep phenomenon, it is necessary to carry out two tests with different loading rates, at the highest load level. If the longitudinal deformations differ by less than 10 % for the two tests, either of the two rates can be used for all tests. If the longitudinal deformations differ by more than 10 %, another test at a higher loading rate is carried out (and so on until deformations fall within 10 % of each other).

8.4 Test technique

8.4.1 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. Before applying the load, i.e. with the mounting unstrained, cut or burn through both sides of the mounting at mid-gauge. If burning is used, care shall be taken to avoid exposing the specimen to the flame.

NOTE 1 As the filament is very fragile, the test specimen breaks occasionally during this step.

NOTE 2 In some cases a preload should be applied during the whole heating period to prevent the alignment from being lost.

The preload shall not increase beyond 5 % of the expected failure load at any moment.

8.4.2 Setting of an inert environment

Before establishing an inert environment, any air or water vapour shall be removed. This can be done by creating a vacuum (below 10 Pa) in the chamber and subsequently introducing the inert gas, or by circulating inert gas (flushing).

Care shall be taken not to introduce too high tensile forces on the test specimen during setting of the inert environment.

NOTE 1 In view of the extreme oxidation sensitivity of some filament materials, conventional flushing of the test chamber may not be sufficient to reduce the oxygen level to an acceptable limit.

NOTE 2 Tensile forces are introduced on the test specimen when an overpressure of inert gas is applied and the machine is operated under zero load control with the load cell mounted outside the test chamber. This can be overcome by using displacement control during setting the controlled atmosphere and periodically reducing the load by adjusting the position of the cross head. Alternatively the load cell can be mounted inside the chamber, so that it is exposed to the same environment as the test specimen.

NOTE 3 Some ceramic fibres or carbon fibres need to be treated at a temperature higher than the test temperature in order to ensure fibre thermostability during the creep test.

When testing under vacuum, the vacuum level shall satisfy the requirements of 6.3.3. When the load cell is situated outside the vacuum chamber, zero load control shall not be used when establishing the vacuum, because of control signal instability caused by the decreasing pressure in the environmental chamber, except in the case where automatic load-pressure compensation is available for the equipment.

8.4.3 Heating of the test specimen

Heat the test specimen to the required temperature and maintain this temperature for a period to allow for the temperature to be stabilised.

8.4.4 Measurements

8.4.4.1 Test machine

When a test machine is used, measurements shall be taken according to the following procedure:

— record temperature, vacuum level or gas pressure before starting the test;

— zero the load cell;

— set the loading rate according to 8.3;

— load the test specimen to the required load level;

— operate under load control, so as to maintain the load at a constant level during the entire test;

— zero the deformation transducer;

— record longitudinal deformation versus time;

— check temperature stability during the test according to 8.4.5;

— record, at the end of the test, the temperature, vacuum level and gas pressure;

— if the test is carried out until failure, record the deformation to failure;

— cool under inert condition until the risk of degradation is removed before opening the test chamber.

8.4.4.2 Creep testing rig

When a creep testing rig is used, measurements shall be taken according to the following procedure:

— record temperature, vacuum level or gas pressure before starting the test;

— load the test specimen to the required load level according to 8.3;

— zero the deformation transducer;

— record longitudinal deformation versus time;

— check temperature stability during the test according to 8.4.5;

— record at the end of the test the temperature, vacuum level and gas pressure;

— if the test is carried out until failure, record the deformation to failure;

— cool under inert condition until the risk of degradation is removed before opening the test chamber.

8.4.5 Monitoring of temperature stability

During long-term testing, drifting of the temperature measurement signal over time may affect conformity with 6.4.

This shall be checked by comparing the temperature at or after the end of the test with the reading of a different calibrated temperature measurement device.

8.5 Test validity

The following circumstances invalidate a test:

— failure to specify and record test conditions;

— specimen slippage;

— change in atmosphere around the specimen;

— variation in temperature or in load outside tolerances during the test;

— failure outside the test temperature zone.

9 Calculation of results

9.1 Creep stress

Calculate the applied stress according to the following formula:

$$\sigma = \frac{F}{A_0} \tag{2}$$

where

σ is the applied stress, in megapascals;

F is the applied force, in newtons;

A_0 is the initial cross section, in square millimetres.

9.2 Creep strain at time *t*

9.2.1 Reading of the longitudinal deformation

On the longitudinal deformation versus time curve, read the longitudinal deformation at time t, ΔL, which corresponds to the whole deformation due to creep (where the load is constant), written by:

$$\Delta L = \Delta L_{T_t} + \Delta L_{T_t + \Delta T} + \Delta L_{T_t + 2\Delta T} + ... + \Delta L_{T_t + i\Delta T} \tag{3}$$

9.2.2 Calculation of the deformation due to the creep

Calculate successively the deformation due to the creep for each temperature zone for a given time and determine the creep rate.

$$\varepsilon_{tot} = \varepsilon_{cr}(T_t) + \varepsilon_{cr}(T_t + \Delta T) + ... + \varepsilon_{cr}(T_t + i\,\Delta T) \quad (4)$$

which can also be written as:

$$\varepsilon_{tot} = \frac{\Delta L_{\Delta T}}{L_{\Delta T}} + \frac{\Delta L_{2\Delta T}}{L_{2\Delta T}} + ... + \frac{\Delta L_{i\Delta T}}{L_{i\Delta T}} \quad (5)$$

where

ΔL is the total longitudinal deformation at time t, in millimetres;

$\Delta L_{\Delta T}$ is the longitudinal deformation in the furnace zone where the temperature T is between $T_t \leq T \leq T + \Delta T$ at time t, in millimetres;

$\Delta L_{2\Delta T}$ is the longitudinal deformation in the furnace zone where the temperature T is between $T + \Delta T \leq T \leq T + 2\,\Delta T$ at time t, in millimetres;

$\Delta L_{i\Delta T}$ is the longitudinal deformation in the furnace zone where the temperature T is between $T + (i - 1)\,\Delta T \leq T \leq T + i\,\Delta T$, at time t, in millimetres.

It is always essential to determine the creep deformation and the creep rate at the lower temperatures to obtain the creep deformation and the creep rate for any given higher temperature.

9.2.3 Drawing of the creep curve

Plot, successively for each temperature, the curve $\varepsilon_{cr(T)}$ versus time.

9.2.4 Creep strain at rupture

Read the creep strain at rupture from the curve.

9.2.5 Creep rupture time

Read the creep rupture time from the curve.

9.2.6 Creep strain rate curve

Plot, successively for each temperature, the curve $\dot{\varepsilon}_{cr(T)}$ creep strain rate versus time.

The creep rate for a given temperature is the sum of the rates of all the different temperature zones from the creep threshold temperature to the test temperature.

NOTE This curve may help discernment between primary, secondary and tertiary creep and shrinkage.

10 Test report

The test report shall contain the following information:

a) name and address of the testing establishment;

b) date of the test, unique identification of the report and of each page, customer name and address, and signatory;

c) reference to this European Standard, i.e. determined in accordance with EN 15365;

d) test piece drawing or reference;

e) description of the test material (material type, manufacturing code, batch number);

f) description of the test set-up: heating system, test frame type, temperature measurement device, extensometer, gripping system, load cell, loading rate, nature of inert environment and level of pressure or level of vacuum, load level, heating rate and test temperature at the beginning and at the end of the test;

g) temperature gradient over gauge length and controlled temperature zone;

h) number of tests carried out and the number of valid results obtained;

i) force longitudinal deformation records for the tests carried out to select the loading rate;

j) creep strain curves and creep strain rate versus time curves for each stress and temperature combination;

k) if applicable, creep strain at rupture and creep rupture time;

l) valid results, mean values and standard deviation (for Gaussian distribution).

Bibliography

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

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

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[5] ISO 3611, *Micrometer callipers for external measurement*

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