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Fine ceramics (advanced ceramics, advanced technical ceramics) — Test method for tensile creep of monolithic ceramics

Céramiques techniques — Méthodes d'essai pour le fluage en traction des céramiques monolithiques

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Foreword

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International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

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.

ISO 22215 was prepared by Technical Committee ISO/TC 206, *Fine ceramics*.

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Fine ceramics (advanced ceramics, advanced technical ceramics) — Test method for tensile creep of monolithic ceramics

1 Scope

This International Standard specifies the test method for determining the tensile-creep strain and creep rupture time under uniaxial constant force of monolithic fine ceramics and whisker or particulate-reinforced ceramic composites at high temperatures. This test method may be used for material development, material comparison, quality assurance, characterization and design data generation.

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.

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ISO 3611, *Micrometer callipers for external measurement*

ISO 4287, *Geometrical Product Specifications (GPS) — Surface texture: Profile method — Terms, definitions and surface texture parameters*

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ISO 7500-2, *Metallic materials — Verification of static uniaxial testing machines — Part 2: Tension creep testing machines — Verification of the force applied*

ISO 9513, *Metallic materials — Calibration of extensometers used in uniaxial testing*

ISO 15490:2000, *Fine ceramics (advanced ceramics, advanced technical ceramics) — Test method for tensile strength of monolithic ceramics at room temperature*

3 Terms and definitions

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

3.1
applied stress
value of tensile force applied to a test specimen divided by the original cross-sectional area of the gauge section

3.2
bending strain component
difference between strain at surface and average of strain measured on diametrically opposed sides and equally distant from the axis

3.3
creep rupture time
time required for a test specimen to fracture under constant force as a result of tensile creep

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3.4
creep strain
time-dependent fractional increase in the gauge length after applying a uniaxial tensile force

NOTE Value of the elongation divided by the gauge length.

3.5
elongation
increase of the gauge length at any moment during the test

3.6
gauge section
parallel portion having the same cross-section at the middle of a test specimen whose elongation is used for determining creep strain

3.7
gauge length
original length of the gauge section determined at room temperature before the test

3.8
gripped section
end section of a test specimen that is held by the gripping device of a tensile-creep testing apparatus

3.9
gripping device
device to hold a test specimen and to transfer a uniaxial tensile force to a test specimen during the test

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3.10
tensile creep
time-dependent deformation of a test specimen subjected to a uniaxial tensile force

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4 Principle

The test consists of applying a constant uniaxial tensile force to a test specimen for the purpose of determining the tensile-creep strain and creep rupture time.

5 Apparatus

5.1 Tensile-creep testing machine

The testing machine shall be such that the constant force can be applied along the axis of the test specimen without fluctuation during a tensile-creep test, while keeping to a minimum the inadvertent bending or torsion of the test specimen. The force shall be applied to the test specimen without shock (see 7.2).

It is recommended that the machine be isolated from external vibration and shock.

The machine shall be verified and shall meet the requirements of class 1 in ISO 7500-2.

5.2 Heating apparatus

A heating furnace provided with a temperature-control device shall be used for heating the specimen. The specimen temperature shall be maintained constant with time at 2 K during a tensile-creep test. The maximum deviation of the temperature over the gauge section shall be less than or equal to 5 K during a tensile-creep test.

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5.3 Temperature measuring apparatus

5.3.1 The temperature readout device shall have a resolution of 1 K or less.

5.3.2 When temperature is measured by a thermocouple, the measurement shall be accurate to 5 K, including the error inherent to a thermocouple, and any error in the measuring instruments. After assuring no chemical reactivity between the test material and thermocouple material, the thermocouple junctions shall be placed no more than 2 mm from the surface of the test specimen and shall be suitably screened from direct radiation from the furnace wall. The remaining portions of the wires within the furnace shall be thermally shielded and electrically insulated by suitable covering.

5.3.3 When a thermometer other than a thermocouple, such as a pyrometer, is used, it shall have an accuracy equivalent or superior to the above-specified one.

5.4 Gripping devices

Various types of gripping devices may be used to ensure that test specimens are held in such a way that the force is applied as axially as possible.

NOTE The gripping devices that have been used when measuring tensile creep of monolithic ceramics and their advantages/disadvantages are shown in ASTM C1291[3].

5.5 Elongation measuring device

The elongation shall be measured using an extensometer which meets the requirements of at least class 1 in ISO 9513, or by other means which ensure the same accuracy without interruption of the test.

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6 Test specimen

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6.1 Shape and dimensions

The shape and dimensions of the test specimens depend on several factors, including the purpose of the tensile test itself, the gripping devices and the shape and dimensions of the ceramic products whose tensile creep is to be determined. Therefore, various shapes and dimensions may be used.

The shapes and dimensions, however, shall be determined so that the tensile stress is applied uniformly in the gauge section (see 7.2). Also, care shall be taken so that stress concentrations that could lead to undesired fractures are as small as possible. In addition, the cross-section of the gauge section shall be uniform with a dimensional variation smaller than ± 0,5 %.

The test specimens that have been applied to measuring tensile strength of monolithic ceramics are shown in ISO 15490:2000, Annex A.

6.2 Test specimen preparation

Surface finishes in the gauge section of the order of average roughness 0,2 μm*Ra* to 0,4 μm*Ra*, as defined in ISO 4287 (measured in the longitudinal direction) are recommended in order to avoid surface roughness-related fracture.

It is highly recommended that the final grinding operation in the gauge section be performed along the longitudinal direction of the test specimen in order to ensure that grinding marks are parallel to the applied tensile stress.

Care shall be taken in the storage and handling of finished test specimens to avoid the introduction of random and severe flaws.

NOTE In some cases, the final surface finish may not be as important as the subsurface damage produced during the grinding process. This damage is not readily observed or measured.

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7 Procedures

7.1 Test specimen dimensions

The diameter or thickness and width of the gauge section of each test specimen shall be determined to within 0,02 mm beforehand, by using the micrometer in accordance with ISO 3611, or by using equipment that is equivalent or superior. Measurements on at least three different cross-sections in the gauge section shall be made. The average of the multiple measurements shall be used in calculating the cross-sectional area. The length of the gauge section shall be determined by points of attachment of the extensometer.

7.2 Axial alignment

The testing system shall be verified using the following procedures at room temperature before the test.

On a "dummy" test specimen that has exactly the same shape as that of a test specimen to be tested, three or four strain gauges shall be equally spaced around the circumferences on two cross-sectional planes. The strain-gauge planes shall be symmetrically located about the longitudinal midpoint of the gauge section, and shall be separated by at least 3/4 of the length of the gauge section. Care shall be taken to avoid placing the strain gauges too near geometric transitions in the gauge section, which can cause strain concentration and inaccurate measurements of the strain in the uniform gauge section. When the gauge section is not long enough to have two strain-gauge planes, one plane may be used. In this case, the location shall be the longitudinal midpoint of the gauge section. When axial strain gauges are mounted, the gauge axis shall be aligned with the stress axis so that the deviation is less than 0,035 rad (2°).

Mount the "dummy" specimen in the gripping device and apply a force so as to give one-half of the stress to be applied in the test. Evaluate the bending strain components using the following equation. The bending strain components verified hereby shall not exceed five.

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$$B = 2 \times \frac{\left[(\varepsilon_1 - \varepsilon_3)^2 + (\varepsilon_2 - \varepsilon_4)^2\right]^{1/2}}{\varepsilon_1 + \varepsilon_2 + \varepsilon_3 + \varepsilon_4} \times 100$$

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for four gauges and

$$B = 2 \times \frac{(\varepsilon_1^2 + \varepsilon_2^2 + \varepsilon_3^2 - \varepsilon_2\varepsilon_1 - \varepsilon_2\varepsilon_3 - \varepsilon_3\varepsilon_1)^{1/2}}{\varepsilon_1 + \varepsilon_2 + \varepsilon_3} \times 100$$

for three gauges, where B is the bending strain component, and ε_1, ε_2, ε_3 and ε_4 are strain readings for strain gauges.

NOTE It is preferable that the "dummy" specimen be of the same material as that to be tested.

7.3 Heating procedure

A specimen shall be heated to a test temperature at such a rate that the specimen and gripping device are free from thermal shock fractures, and shall be soaked to obtain a thermal equilibrium before the test starts. Regardless of whether a furnace/test specimen are heated from room temperature to the test temperature, or a test specimen is inserted into a furnace already at the test temperature, the soak time for the entire system to reach equilibrium shall be determined experimentally. The final temperature adjustment shall be conducted during this time.

7.4 Loading procedure

An initial force, which is the minimum one required for maintaining the axial alignment, may be applied to a specimen while heating the specimen. The initial force shall be less than 10 % of the test force.

After soaking the specimen at the test temperature, the force shall be applied along the test axis in such a manner as to minimize bending and torsion of the test specimen, without shock.

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7.5 Recording of temperature and of elongation

Throughout the test, it is important that sufficient readings be taken of the temperature of the test specimen, in order to demonstrate that the temperature conditions comply with the requirements of 5.3.

Either a continuous record or a sufficient number of readings of the elongation shall be made throughout the test so that the creep-time curve can be traced.

8 Determination of the results

The test results are determined from the preceding readings using the definitions given in Clause 3.

9 Test report

9.1 The following items shall be reported:

a) material of a specimen;

b) geometry and dimensions of a specimen;

c) surface roughness of a gauge section;

d) test condition:

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1) test temperature, and its reading intervals,

2) heating rate and soak time,

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3) applied stress,

4) atmosphere;

e) test results:

1) time-strain curve,

2) time-strain rate relation,

3) rupture time, fracture strain, and fracture location, when the test was conducted until fracture.

9.2 The following items shall preferably be appended:

a) specimen:

1) manufacturer,

2) product type or code,

3) chemical composition,

4) machining condition,

5) heat-treatment condition, if any,

6) available mechanical properties at room and test temperatures;

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