SLOVENSKI STANDARD
SIST EN 1389:2004

01-oktober-2004

Sodobna tehniļna keramika - Keramiļni kompoziti ï Fizikalne lastnosti - Doloļanje gostote in navidezne poroznosti

Advanced technical ceramics - Ceramic composites - Physical properties - Determination of density and apparent porosity

Hochleistungskeramik - Keramische Verbundwerkstoffe - Physikalische Eigenschaften - Bestimmung der Dichte und scheinbaren Porosität

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Céramiques techniques avancées - Céramiques composites - Propriétés physiques - Détermination de la masse volumique et de la porosité apparente

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Ta slovenski standard je istoveten z: **EN 1389:2003**

ICS:

81.060.30	Sodobna keramika	Advanced ceramics

SIST EN 1389:2004 **en**

2003-01.Slovenski inštitut za standardizacijo. Razmnoževanje celote ali delov tega standarda ni dovoljeno.

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EUROPEAN STANDARD

NORME EUROPÉENNE

EUROPÄISCHE NORM

EN 1389

December 2003

ICS 81.060.30

Supersedes ENV 1389:1994

English version

Advanced technical ceramics - Ceramic composites - Physical properties - Determination of density and apparent porosity

Céramiques techniques avancées - Céramiques composites - Propriétés physiques - Détermination de la masse volumique et de la porosité apparente

Hochleistungskeramik - Keramische Verbundwerkstoffe - Physikalische Eigenschaften - Bestimmung der Dichte und scheinbaren Porosität

This European Standard was approved by CEN on 3 November 2003.

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This European Standard exists in three official versions (English, French, German). A version in any other language made by translation under the responsibility of a CEN member into its own language and notified to the Management Centre has the same status as the official versions.

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EUROPEAN COMMITTEE FOR STANDARDIZATION
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Ref. No. EN 1389:2003 E

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Foreword

This document (EN 1389:2003) has been prepared by Technical Committee CEN/TC 184 “Advanced technial 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 June 2004, and conflicting national standards shall be withdrawn at the latest by June 2004.

This document supersedes ENV 1389:1994.

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

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1 Scope

This European Standard describes two methods for determination of the bulk density and open porosity of ceramic matrix composites with fibrous reinforcement.

Two methods are described and are designated as Methods A and B, as follows :

— method A : determination of bulk density only by measurement of dimensions and mass ;

— method B : determination of bulk density and open porosity by liquid displacement.

NOTE Method B is not suitable for the determination of the open porosity for materials which are known to have an average pore size of greater than 200 µm.

2 Normative references

This European Standard incorporates, by dated or undated reference, provisions from other publications. These normative references are cited at the appropriate places in the text and the publications are listed hereafter. For dated references, subsequent amendments to or revisions of any of these publications apply to this European Standard only when incorporated in it by amendment or revision. For undated references the latest edition of the publication referred to applies (including amendments).

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ENV 13233:1998, *Advanced technical ceramics – Ceramic composites – Notations and symbols.*

EN ISO 291, *Plastics – Standard atmospheres for conditioning and testing (ISO 291:1997).*

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EN ISO 1675, *Plastics – Liquid resins – Determination of density by the pyknometer method (ISO 1675:1985).*

ISO 758, *Liquid chemical products for industrial use – Determination of density at 20° C.*

ISO 3611, *Micrometer callipers for external measurement.*

3 Terms and definitions

For the purposes of this European Standard, the terms and definitions given in ENV 13233:1998 and the following apply.

3.1
open pores
pores that are penetrated by an immersion liquid in vacuum, or that are connected with the atmosphere, either directly or via one to another

3.2
closed pores
pores that are not penetrated by the immersion liquid, or that are not connected with the atmosphere

3.3
open porosity π_a
ratio of the total volume of the open pores in a porous body to its bulk volume

3.4
bulk volume, V_b
sum of the volumes of the solid material, the open pores and the closed pores in a porous body

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3.5
bulk density ρ_b
ratio of the mass of the dry material of a porous body to its bulk volume

4 Test specimens

4.1 General

The choice of specimen geometry depends on the nature of material and reinforcement structure. The mass of the test specimen shall be greater than 2 g and each dimension shall be greater than 3 mm, with the volume greater than 1 cm^3 (see 4.3).

4.2 Sampling

The sampling method shall be agreed between purchaser and manufacturer.

4.3 Number

At least five test specimens shall be taken from each sample.

NOTE Particular requirements for Method A are given in 5.3.

4.4 Conditioning and testing atmospheres

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The test specimens shall be conditioned in a standard testing atmosphere in accordance with EN ISO 291. During testing, the apparatus and test specimens shall be maintained at constant temperature, preferably at (23 ± 2) °C and (50 ± 5) % relative humidity.

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5 Method A

5.1 Principle

A test specimen of uniform geometry is dried and weighed. Its volume is determined by measurement of the appropriate dimensions. The density is calculated as mass per unit volume.

5.2 Apparatus

5.2.1 Balance, with accuracy of 0,1 mg.

5.2.2 Dimension measuring devices, used for measuring linear dimensions of the test specimen. The devices shall be accurate to ± 0,05 mm. Micrometers shall be in accordance with ISO 3611.

5.2.3 Drying oven, capable of maintaining a temperature of (110 ± 5) °C.

5.2.4 Desiccator, for storage of test specimens.

5.3 Test specimen

The shape of test specimens shall be such that the volume can be calculated from the external dimensions, such as rectangular parallelepipeds and right cylinders, discs or rods. Test specimens which do not have uniform dimensions and principal axes orthogonal to within 1° shall be ground to achieve such conditions.

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The plan parallelism of the faces used to determine the external dimensions shall be better than or equal to 0,05.

5.4 Procedure

Dry the test specimens in the oven at (110 ± 5) °C to constant mass, i.e. until two successive weighings made before and after at least 2 h in the drying oven do not differ by more than 0,03 %, transfer them to a desiccator and allow to cool to room temperature. Record the mass of each test specimen in ambient air, as soon as possible after removal from the desiccator.

Using the selected measuring device, measure the dimensions of each test specimen in at least three positions for each direction. e.g. length, width and height for parallelipipedal test specimen or lengths and diameter for a cylindrical test specimen. The value of each dimension is the arithmetic mean of the 3 measurements.

5.5 Results

Calculate the bulk volume of each test specimen from its mean dimensions. The bulk density ρ_b is given by the mass divided by the volume. Express the result in $kg.m^{-3}$ or in $g.cm^{-3}$.

$$\rho = \frac{m}{V_b}$$

where

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m is the test specimen mass, expressed in kilogram or in gram ;

V_b is the bulk volume of test specimen, expressed in cubic metre or cubic centimetre.

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The value of bulk density is expressed with a maximum of two significant digits.

6 Method B

6.1 Principle

The mass of the test specimen is determined by weighing in the following conditions :

(1) dry test specimen in air;

(2) test specimen immersed in a liquid which it has been impregnated under vacuum;

(3) test specimen in air while still impregnated with the liquid.

From these values its bulk density and open porosity are determined by calculation.

6.2 Apparatus and reagents

6.2.1 Drying oven, capable of maintaining a temperature of (110 ± 5) °C.

6.2.2 Balance, with an accuracy of 0,1 mg.

6.2.3 Degreased metal wire, of diameter not more than 0,15 mm or cradle (glass or stainless steel with holes), to contain test specimen and able to be easily immersed in the liquid.

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6.2.4 Evacuating equipment, capable of reducing the pressure to a value less than 2 500 Pa (25 mbar) and a means of measuring the pressure used.

6.2.5 Thermometer, capable of measuring to ± 0,1 °C.

6.2.6 Glass beaker, of a size allowing adequate clearance of its walls by the test specimen.

6.2.7 Desiccator, for storage of test specimens.

6.2.8 Manometer.

6.2.9 Absorbent cloth or tissue paper.

6.2.10 Immersion liquid, which may be either :

a) cold distilled water, containing a dilute solution of a surface active agent (concentration not more than 0,01 %) ;

b) an organic liquid, which shall be used for materials that are sensitive to contact with water.

6.3 Procedure

6.3.1 Determination of the mass of the dry test specimen

Dry the test specimen in the oven at (110 ± 5) °C to constant mass, after at least 2 h in the drying oven.

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Before each weighing, place the test specimen in the desiccator until it is cooled to room temperature, and weigh it as quickly as possible after removal from the desiccator. The mass thus determined is the mass of the dry test specimen (m_1).

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6.3.2 Determination of the apparent mass of the immersed test specimen

Place the cooled and dried test specimen in an airtight vessel. Seal the vessel and evacuate it until a pressure of less than 2 500 Pa is attained; maintain this vacuum for at least 15 min.

In order to ensure that all the air has been removed from the test specimen, isolate or disconnect the vessel from the vacuum pump and use the manometer to check that the pressure does not rise because of any de-gasing of the test specimen.

Progressively introduce the immersion liquid in such a way that, after 3 min, the test specimen is covered by about 20 mm of liquid.

Reconnect the vessel to the vacuum pump.

Maintain the vacuum until pressure stability is obtained to ensure complete penetration of the test specimen by the liquid.

Suspend the test specimen by the thin wire from the load-pan suspension point of the balance, perpendicular to the liquid, and weigh it while it is completely immersed in a quantity of the immersion liquid, contained in the beaker standing on the bridge (if used).

Remove bubbles from the surface of the test specimen and weigh. Remove the test specimen from the suspension wire, replace the wire in the liquid to the same suspension level and weigh. The difference between the two weighings is the apparent mass of the immersed test specimen (m_2). Determine the temperature of the immersion liquid, using the thermometer.