SLOVENSKI STANDARD
SIST ENV 1389:2000

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

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

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

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Descriptors: composite materials, reinforcing materials, ceramics, physical properties, determination, density (mass/volume), bulk density, porosity, immersion tests

English version

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CEN

European Committee for Standardization
Comité Européen de Normalisation
Europäisches Komitee für Normung

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Foreword

This European Prestandard has been prepared by Technical Committee CEN/TC 184 "Advanced technical ceramics", of which the secretariat is held by BSI.

CEN/TC 184 approved this European Prestandard by resolution 4/1992 during its fifth meeting, held in Brussels on 1992-03-31.

In accordance with the CEN/CENELEC Internal Regulations, following countries are bound to announce this European Prestandard: Austria, Belgium, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxembourg, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland and United Kingdom.

1 Scope

This European prestandard describes two methods for determination of the bulk density and apparent porosity of ceramic matrix composites with fibrous continuous reinforcement (1D, 2D, 3D).

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 apparent porosity by liquid displacement

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

The two methods do not measure the same properties and do not give equivalent test results. Corresponding methods for monolithic ceramics are given in EN 623-2.

2 Normative references

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

EN 623-2	Advanced technical ceramics - Monolithic ceramics - General and textural properties Part 2 : Determination of bulk density and apparent porosity
ISO 291	Plastics - Standard atmospheres for conditioning and testing
ISO 758	Liquid chemical products for industrial use - Determination of density at 20 degrees C.
ISO 1675	Plastics - Liquid resins - Determination of density by the pyknometer method
ISO 6906	Vernier callipers reading to 0,02 mm

3 Definitions and symbols

For the purposes of this European prestandard, the following definitions apply:

3.1 Open pores: Those pores that are penetrated by an immersion liquid in vacuum, or that are connected with the atmosphere, either directly or via one another.

3.2 Closed pores: Pores that are not penetrated by the immersion liquid, or that are not connected with the atmosphere.

3.3 Apparent porosity Π_a : – The ratio of the total volume of the open pores in a porous body to its bulk volume.

3.4 Bulk volume : The sum of the volumes of the solid material, the open pores and the closed pores in a porous body.

3.5 True volume: The volume of a body occupied by solid material, excluding all forms of porosity.

3.6 Geometric bulk density: The mass per unit total volume of a material including all porosity accessible and inaccessible from the surface, the volume being calculated from linear dimension.

3.7 Bulk density ρ_b : The ratio of the mass of the dry material of a porous body to its bulk volume.

4 Test pieces

4.1 Sampling

The sampling method shall be agreed between purchaser and manufacturer.

4.2 Number

At least five test pieces shall be taken from each sample. The test piece volume shall be taken into account in the elementary design of material and the minimal volume shall be about 1 cm^3.

NOTE : Particular requirements for Method A are given in 5.3.

4.3 Conditioning and testing atmospheres

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

5 Determination of geometric bulk density (Method A)

5.1 Principle

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

5.2 Apparatus

5.2.1 Balance, with accuracy of 0,1 mg

5.2.2 Calibrated measuring device, capable of repeatable and accurate measurement with accuracy of 0,01 mm or 0,05 %, e.g. vernier callipers, in accordance with ISO 6906, or a micrometer.

5.2.3 Drying oven, capable of monitoring a temperature of 110 °C ± 5 °C.

5.2.4 Desiccator, for storage of test pieces.

5.3 Test piece shape

Ceramic composites are unsuited to this method of measurement of bulk density unless the skin is flattened or removed by machining or other suitable method.

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

The mass of the test piece shall be greater than 2 g and each dimension shall be greater than 3 mm, with the volume greater than 1 cm^3 (see 4.2).

5.4 Procedure

Dry the test pieces in the oven at 110 °C ± 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 piece in ambient air, as soon as possible after removal from the desiccator.

Using the selected measuring device, measure the dimensions of each test piece in at least three positions for each direction, to an accuracy better than 0,01 mm, or 0,05 % of the smallest dimension. Measure the directions parallel to the principal geometric axes, e.g. the length and diameter for a disc or a rod.

Calculate the differences between the lowest and highest figures measured for each direction and reject the test piece if any difference exceed 1 % of the mean dimensions measured.

5.5 Results

Calculate the geometric volume of each test piece from its mean dimensions. The geometric bulk density ρ is given by the mass divided by the geometrical volume. Express the results in kg/m^3 or g/cm^3.

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

When:
m = test piece mass in grams
V = geometric volume of test piece in cubic centimetres
The accuracy of the measurement is no better than 1 %

6 Determination of bulk density and porosity by liquid displacement (Method B)

6.1 Principle

The mass of a dry test piece is determined by weighing, then its apparent mass when immersed in a liquid with which it has been impregnated under vacuum, and then its mass in air while still soaked with the liquid. From these values its bulk density and apparent porosity are determined by calculation.

6.2 Apparatus and reagents

6.2.1 Drying oven, capable of maintaining a temperature of 110 °C ± 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 piece and able to be easily immersed in the liquid.

6.2.4 Evacuating equipment, capable of reducing the pressure to a value less than 2500 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 piece.

6.2.7 Desiccator, for storage of test pieces.

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.