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Fine ceramics (advanced ceramics, advanced technical ceramics) — Determination of density and apparent porosity

Céramiques techniques — Détermination de la masse volumique et de la porosité apparente

Reference number
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Contents

Page

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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 18754 was prepared by Technical Committee ISO/TC 206, *Fine ceramics*.

This second edition cancels and replaces the first edition (ISO 18754:2003), which has been technically revised.

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Fine ceramics (advanced ceramics, advanced technical ceramics) — Determination of density and apparent porosity

1 Scope

This International Standard specifies methods for the determination of the apparent solid density, bulk density, apparent density and geometric bulk density of fine ceramics.

NOTE These methods are not appropriate for the determination of an apparent porosity greater than 10 %. For materials with higher porosity, the accuracy of the measurement may not be satisfactory. The method may also not give a satisfactory open porosity result if it is less than 0,5 %

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.

ISO 386:1977, *Liquid-in-glass laboratory thermometers — Principles of design, construction and use*

ISO 758:1976, *Liquid chemical products for industrial use — Determination of density at 20 degrees C*

ISO 13385-1:2011, *Geometrical product specifications (GPS) — Dimensional measuring equipment — Part 1: Callipers; Design and metrological characteristics*

EN 1006:2009, *Advanced technical ceramics — Monolithic ceramics — Guidance on the selection of test pieces for the evaluation of properties*

ISO/IEC 17025:2005, *General requirements for the competence of testing and calibration laboratories*

3 Terms and definitions

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

3.1
open pores
pores that are penetrated by an immersion liquid, or that are connected to 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 to the atmosphere

3.3
bulk volume
the sum of the respective volumes of the solid material, the open pores and the closed pores

3.4
apparent solid volume
the sum of the respective volumes of the solid material and the closed pores

3.5
apparent solid density
the ratio of the mass of the dry material to its apparent solid volume

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3.6
bulk density
the ratio of the mass of the dry material to its bulk volume

3.7
apparent porosity
the ratio of the volume of the open pores in a body to its bulk volume

3.8
theoretical density
TD
density of pore-free material

3.9
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 dimensions

4 Methods by impregnation with liquid

4.1 Apparatus

4.1.1 Drying oven, capable of being controlled at 110 °C ± 5 °C.

4.1.2 Balance, accurate to 0,1 mg for a test specimen under 10 g and 0,001 % of the mass of a test specimen for a specimen over 10 g.

4.1.3 Heating apparatus, in which the specimen may be boiled.

4.1.4 Thermometer, in accordance with ISO 386 with an accuracy of ± 1 °C.

4.1.5 Immersion liquid, distilled water or de-ionized water may be used for materials that do not react with water. For materials that are sensitive to contact with water, a suitable organic liquid shall be used.

4.1.6 Halter or basket, capable of supporting the test pieces in liquid in order to take suspended mass measurements.

4.1.7 Suspending wire, of diameter not more than 0,25 mm. The wire should be cleaned and de-greased. Where specimens of small mass are used, the suspending wire having a smaller diameter or the addition of a dilute solution of a suitable surfactant is recommended, because the error caused by the surface tension of the liquid on the wire cannot be neglected.

4.1.8 Evacuating equipment, (in the vacuum method) capable of reducing the pressure to a value not greater than 2,5 kPa, and with a means of measuring the pressure used.

4.2 Test specimen

The volume of each specimen shall be not less than 0,1 cm^3.

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When the volume of each individual test specimen is less than this value, a sufficient number of test specimens shall be taken so that the total volume of the specimens reaches the minimum of volume. In this case, the volume of each individual test specimen shall be not less than 0,04 cm^3.

NOTE In the case where the volume of test specimen is less than 0,04 cm^3, a geometric measurement for the machined specimen may be used only for the determination of bulk density. For the determination of the bulk density and apparent porosity, mercury porosimetry may be applied. However, a combination of a stereological measurement on a polished surface of the test specimen by microscopy is recommended for the reliability.

Any dust and chips liable to become detached during further handling shall be removed from the surface of each test piece.

Test specimens shall have smooth surfaces to sponge out droplets of the immersion liquid from the surface, since roughness limits the accuracy of the mass of the soaked test specimen.

4.3 Procedure

4.3.1 General

Drying in a desiccator (see 4.3.2) and the procedure according to 4.3.3, 4.3.4 and 4.3.6 shall only be carried out if the test specimen has open porosity, i.e. the density is less than 95 % of the theoretical density. If the test specimen has no open porosity, the mass of the dry test specimen m_1 is equal to the mass of the soaked test specimen m_3 and the apparent solid density is equal to the bulk density and the apparent porosity is 0.

NOTE Open porosity can be recognized if the measured weight value changes during weighing (see 4.3.5).

4.3.2 Determination of mass of dry test specimen

Weigh the test specimen in a dry state. If the test specimen has open porosity, dry the test specimen in a drying oven (4.1.1) controlled at 110 °C ± 5 °C, allow it to cool to room temperature in a desiccator and weigh. If the test specimen has no open porosity, the specimen can be dried, e.g. with a towel, and weighed. The mass thus determined is the mass of the dry test specimen, m_1. For the test specimen which may possibly break during boiling, determine the mass of the dry test specimen after the apparent mass of the immersed test specimen and the mass of the soaked test specimen have been determined.

4.3.3 Boiling method (method A)

Immerse the test specimen in the heating apparatus (4.1.3) taking care that the test specimen is covered with water at all time, boil for 3 h or more and allow to cool to room temperature. Water at ambient temperature may be used to cool the test specimen to room temperature. In this way the soaked test specimen is obtained.

The boiling method shall not apply to materials that react with water.

In the boiling method, an organic liquid shall not be used as the immersion liquid if the liquid vapours are explosive and toxic.

4.3.4 Vacuum method (method B)

Place the test specimen in an airtight container (see 4.1.8), evacuate to a pressure of less than 2,5 kPa and maintain it for 15 min in order to remove all the air from the open pores of the test specimen. Introduce the immersion liquid (4.1.5) so that the test specimen is covered completely. Gradually release the vacuum to atmospheric pressure and allow the test specimen to remain in the immersion liquid for an additional 30 min.

During the introduction of the immersion liquid, the vacuum pump shall be in continuous operation and be stopped upon completion of the introduction.

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For materials that react with water, a suitable organic liquid shall be used as the immersion liquid. In this case, the organic immersion liquid should be low volatile and nontoxic. The vapour pressure of the organic immersion liquid shall be less than 2,5 kPa at the temperature of the test.

NOTE Distilled paraffin and dibutyl phthalate may be used.

4.3.5 Determination of apparent mass of (if necessary immersed) test specimen

Place the test specimen in the halter or basket (4.1.6) and suspend the basket in the immersion liquid using the thin wire (4.1.7). Using the balance (4.1.2), measure the suspended mass while completely immersed in the immersion liquid. Remove the specimen from the halter or basket and reweigh the halter or basket when immersed in the immersion liquid to the same depth as when the test specimen was in place. Subtract the apparent mass of the immersed halter or basket from that when it contained the test specimen. The mass thus obtained is the apparent mass of the immersed test specimen, m_2.

Determine the temperature of the immersion liquid using the thermometer (4.1.4).

4.3.6 Determination of mass of soaked test specimen

If the test specimen has no open porosity, the mass of the dry test specimen m_1 is equal to the mass of the soaked test specimen m_3. The following procedure shall only be carried out if the test specimen has open porosity, i.e. the density is less than 95 % of the theoretical density.

Remove the test specimen from the liquid, sponge it rapidly and carefully with a wet absorbent cloth such as a gauze or chamois leather, to remove droplets of the immersion liquid from the surface of the test specimen and weigh it. The mass thus obtained is the mass of the soaked test specimen, m_3.

The absorbent cloth or the chamois leather shall previously have been completely saturated with the immersion liquid and lightly wrung out in order to avoid drawing out the liquid from the pores of the test specimen.

4.3.7 Determination of the density of the immersion liquid

Determine, to the nearest 1 kg/m^3, the density ρ_1 of the liquid used as the immersion liquid at the temperature of the test.

For water, the density is given in Table 1 as a function of temperature between 10 °C and 30 °C.

For an organic liquid, use the method given in ISO 758.

Table 1 — Density of water as a function of temperature between 10 °C and 30 °C

Temperature °C	ρ_1 kg/m^3	Temperature °C	ρ_1 kg/m^3	Temperature °C	ρ_1 kg/m^3
10	999,7	17	998,8	24	997,3
11	999,6	18	998,6	25	997,0
12	999,5	19	998,4	26	996,8
13	999,4	20	998,2	27	996,5
14	999,2	21	998,0	28	996,2
15	999,1	22	997,8	29	995,9
16	998,9	23	997,5	30	995,6

4.4 Accuracy of mass measurement

The mass measurement shall be made to the nearest 0,1 mg for a test specimen under 10 g and 0,001 % of the mass of a test specimen for a specimen over 10 g.

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4.5 Expression of results

4.5.1 Apparent solid density

The apparent solid density is the ratio of the mass of the dry material to its apparent solid volume and is given by Formula (1). The density shall be calculated to the second decimal place.

$$\rho_a = \frac{m_1}{m_1 - m_2} \times \rho_1 \quad (1)$$

where

ρ_a is the apparent solid density, expressed in kilograms per cubic metre;

m_1 is the mass of the dry test specimen, expressed in kilograms;

m_2 is the apparent mass of the immersed test specimen, expressed in kilograms;

ρ_1 is the density of the immersion liquid at the temperature of the test, expressed in kilograms per cubic metre.

4.5.2 Bulk density

The bulk density is given by Formula (2). The density shall be calculated to the second decimal place.

$$\rho_b = \frac{m_1}{m_3 - m_2} \times \rho_1 \quad (2)$$

where

ρ_b is the bulk density, expressed in kilograms per cubic metre;

m_3 is the mass of the soaked test specimen, expressed in kilograms.

4.5.3 Apparent porosity

The apparent porosity is the ratio of the volume of the open pores in a body to its bulk volume and is given by Formula (3). The porosity shall be calculated to the first decimal place.

$$\pi_a = \frac{m_3 - m_1}{m_3 - m_2} \times 100 \quad (3)$$

where

π_a is the apparent porosity, expressed as a percentage by volume.

5 Determination of geometric bulk density by measurement of dimensions and mass (method C)

5.1 Principle

A test piece of uniform geometry within specified tolerances is dried and weighed. Its volume is determined by measurement of the appropriate dimensions. The geometric bulk density (see 3.9) is calculated as mass per unit volume.

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5.2 Apparatus

5.2.1 Balance, with an accuracy in accordance with Table 2.

5.2.2 Calibrated measuring device, capable of repeatable and accurate measurement in accordance with Table 2, e.g. vernier callipers, or a micrometer in accordance with ISO 13385-1:2011.

NOTE For flat test piece surfaces, spherical measuring anvils with radii of curvature between 2 mm and 10 mm should be used. For cylindrical test piece surfaces, flat measuring anvils should be used. These anvils should be constructed of material with hardness of at least 500 HV30.

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

5.2.4 Desiccator, for storage of test pieces.

Table 2 — Accuracy and errors of density and porosity measurement

Parameter	Method C: Geometric bulk density (Clause 5)
Minimum test piece dimension in mm	3,0
Accuracy of measurement of dimension[a]	0,01 mm or 0,05 % of smallest dimension
Minimum test piece mass in g	2,0
Accuracy of weighing in g	0,001
Accuracy of density measurement (%)	1,0
[a] The maximum non-uniformity of any dimension should not exceed 1 % of its average value.	

5.3 Test pieces

Materials for testing shall be sampled in accordance with the guidance given in EN 1006:2009. The shape of test pieces shall be such that the volume can be calculated from the external dimensions.

NOTE 1 Ideal shapes are rectangular parallelepipeds and right cylinders, discs or rods.

NOTE 2 Test pieces which do not have uniform dimensions and principal axes orthogonal to within 1° should 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 (see Table 2). Where “as-fired” test pieces are used, press flashing shall be removed.

NOTE 3 The total volume of edge chips and surface pits or protrusions should not exceed approximately 0,1 % of the nominal total volume.

NOTE 4 Some types of material possess surface skins which are rough or soft in the “as-fired” state. These materials are unsuited to this method of measurement of bulk density unless the skin is flattened or removed by machining or another suitable method. Reaction-bonded silicon nitride is an example where such a surface deposit may be present.

5.4 Procedure

Dry the test pieces in the oven (see 5.2.3) 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 coolingdown to room temperature.

If the specimen has no open porosity no special drying and cooling down process and no desiccator cooling is needed. In this case dry the specimen e.g. with a towel.

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Record the mass of each test piece in ambient air, as soon as possible after removal from the desiccator or drying.

Using the selected measuring device (see 5.2.2), measure the dimensions of each test piece at least at three positions for each direction, to an accuracy equal to or 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, breadth and depth of a parallelepiped, the length and diameter for a disc or a rod.

NOTE If the dimensions of the test piece are too small to make three separate measurements in any direction, e.g. of the length of a rod of small diameter, a single measurement may be used, such a simplified procedure being reported [see Clause 6 f)].

Calculate the differences between the lowest and highest figures measured for each direction. Reject the test pieces if any difference exceeds 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 is given by the mass divided by the geometrical volume. Express the values of density in kilograms per cubic metre.

6 Test report

The test report shall be in accordance with the reporting provisions of ISO/IEC 17025 and shall contain the following information:

a) a reference to this International Standard, i.e. ISO 18754;

b) the method used (A: the boiling method, B: the vacuum method or C: the geometric bulk density method);

c) the immersion liquid, its density (measurement, literature citation) and the temperature of the test;

d) the individual values of the apparent solid density, bulk density, apparent porosity and geometric bulk density;

e) the mean values of the apparent solid density, bulk density, apparent porosity and geometric bulk density;

f) for method C, any use of the simplified measurement procedure (see 5.4).

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