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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

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Foreword

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The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT), see www.iso.org/iso/foreword.html.

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

This third edition cancels and replaces the second edition (ISO 18754:2013), which has been technically revised. The main changes compared to the previous edition are as follows:

— clarification of the different methods covered by the document;

— extension of the applicability field of the method to ceramic matrix composites (including the fibre reinforced matrix);

— incorporation of specific requirements in terms of geometry and sizes of the test specimens in this case.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

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

1 Scope

This document specifies methods for the determination of the apparent solid density, bulk density, apparent porosity and geometric bulk density of fine ceramics, including all ceramic matrix composites.

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

— Method A: Determination of bulk density, apparent solid density and apparent porosity by liquid displacement (Archimedes' method).

NOTE 1 This method is not appropriate for the determination of an apparent porosity greater than 10 %. For materials with higher porosity, the accuracy of the measurement might not be satisfactory. This method might also not give a satisfactory open porosity result if it is less than 0,5 %.

NOTE 2 This method is also not suitable for materials which are known to have an average pore size of greater than 600 µm.

— Method B: Determination of bulk density only, by measurement of geometric dimensions and mass.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements 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, *Liquid-in-glass laboratory thermometers — Principles of design, construction and use*

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

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

ISO/IEC 17025, *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.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at http://www.electropedia.org/

3.1
open pore
pore that is penetrated by an immersion liquid, or that is connected to the atmosphere, either directly or via one another

3.2
closed pore
pore that is not penetrated by the immersion liquid, or that is not connected to the atmosphere

3.3
bulk volume
V_b
sum of the respective volumes of the solid material, the *open pores* (3.1) and the *closed pores* (3.2) in a porous body

3.4
apparent solid volume
V_a
sum of the respective volumes of the solid material and the *closed pores* (3.2) in a porous body

3.5
bulk density
ρ_b
ratio of the mass of the dry material of a porous body to its *bulk volume* (3.3)

3.6
apparent solid density
ρ_a
ratio of the mass of the dry material of a porous body to its *apparent solid volume* (3.4)

3.7
apparent porosity
π_a
ratio of the total volume of the *open pores* (3.1) in a porous body to its *bulk volume* (3.3)

3.8
theoretical density
density of pore-free material

3.9
geometric bulk density
bulk density (3.5) of a porous body, the *bulk volume* (3.3) of which being calculated from linear dimensions

4 Method by liquid displacement (Method A)

4.1 Principle

The mass of the dry test piece, then its apparent mass when immersed in a liquid with which it has been impregnated, and finally its mass in air while still soaked with the impregnation liquid are determined by weighing. From the masses above, the bulk density, the apparent solid density and the apparent porosity are determined by calculation.

Impregnation of the test piece can be achieved either by maintaining it in a liquid under boiling conditions (Method A1) or under vacuum (Method A2).

4.2 Apparatus

4.2.1 Drying oven, capable of being controlled at (110 ± 5) °C.

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

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

4.2.4 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 or that cannot be fully wetted by water, a suitable organic liquid shall be used.

4.2.5 Basket, capable of supporting the test piece(s) in liquid in order to take suspended mass measurements (optional).

4.2.6 Suspending wire, of diameter not more than 0,25 mm. The wire should be cleaned and de-greased.

Whenever test pieces of small mass are used, a suspending wire of smaller diameter (e.g. 0,15 mm) or the addition to the immersion liquid of a dilute solution (e.g. not more than 0,01 %) of a suitable surfactant is recommended, because the error caused by the surface tension of the liquid on the wire cannot be neglected.

4.2.7 Evacuating equipment (Method A2), capable of reducing the pressure to a value not greater than 2,5 kPa, and with a means of measuring the pressure used.

4.2.8 Heating apparatus (Method A1), enabling the heating of the immersion liquid to its boiling point.

4.2.9 Glass beaker, of a size which allows adequate clearance of its wall by the test piece.

4.2.10 Absorbent cloth, such as a gauze, industrial wiping cloth or chamois leather.

4.3 Test pieces

4.3.1 General requirements

Materials for testing should be sampled in accordance with the guidance given in EN 1006. The volume of each specimen shall be not less than 0,1 cm^3.

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.

In cases 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 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.

4.3.2 Specific requirements for ceramic matrix composites

In addition to the requirements of 4.3.1, the length (l) and width (d) of a test piece should be at least equivalent to those containing more than two unit cells. The thickness (h) of a test piece should be at least equivalent to that containing more than three plies for 2D woven and more than two unit cells for 3D woven.

NOTE The specific terms used above and related to ceramic matrix composites are defined in ISO 20507.

4.4 Procedure

4.4.1 Determination of the mass of the dried test piece

Dry the test piece in the oven (see 4.2.1) at (110 ± 5) °C to constant mass, i.e. until two successive weight measurements, taken before and after at least 2 h in the drying oven, do not differ by more than 0,03 %. Transfer to a desiccator and allow to cool to room temperature.

NOTE For test pieces assumed to have no open porosity (i.e. assumed to have a density higher than 95 % of the theoretical density), the abovementioned drying procedure could be skipped and the specimens could be dried e.g. with a towel.

Record the mass of the test piece in ambient air, as soon as possible after removal from the desiccator or drying. The mass thus determined is the mass of the dry test specimen, m_1.

For a test piece which could possibly break during boiling (see 4.4.2), determine the mass of the dry test piece after the apparent mass of the immersed test piece and the mass of the soaked test piece have been determined.

4.4.2 Impregnation of the test piece with the immersion liquid

Impregnation of the test piece can be carried out in two ways.

a) Boiling method (Method A1)

Immerse the test piece in the glass beaker (4.2.9), taking care that the test piece is covered with water at all times. Using the heating apparatus (4.2.8), bring the water to ebullition and let it boil for 3 h or more. Allow to cool to room temperature. Water at ambient temperature may be used to cool the test piece to room temperature.

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

b) Vacuum method (Method A2)

Place the test specimen in an airtight container (see 4.2.7), evacuate to a pressure of less than 2,5 kPa and maintain for 15 min in order to remove all the air from the open pores of the test specimen. Introduce the immersion liquid (4.2.4) 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 shall be stopped upon completion of the introduction.

For materials that react with water, a suitable organic liquid (e.g. distilled paraffin or dibutyl phthalate) shall be used as the immersion liquid. In this case, the organic immersion liquid should be low volatile and non-toxic. The vapour pressure of the organic immersion liquid shall be less than 2,5 kPa at the temperature of the test.

NOTE For test pieces assumed to have no open porosity, this impregnation step could be skipped.

4.4.3 Determination of the apparent mass of the test piece

Place the impregnated test piece in the basket (4.2.5) and suspend the basket in the immersion liquid using the thin wire (4.2.6). Using the balance, measure the suspended mass while completely immersed in the immersion liquid. Remove the test piece from the basket and reweigh the 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 basket from the mass when it contained the test piece. The mass thus obtained is the apparent mass of the immersed test piece, m_2.

Determine the temperature of the immersion liquid using the thermometer (4.2.3).

4.4.4 Determination of the mass of the soaked test piece

Remove the test piece from the liquid, sponge it rapidly and carefully with a wet absorbent cloth – such as a gauze, chamois leather or industrial wiping cloth that would not leave lint or fibres on the test piece – to remove droplets of the immersion liquid from the surface of the test piece and weigh it. The mass thus obtained is the mass of the soaked test piece, 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 piece.

NOTE For test pieces assumed to have no open porosity (i.e. assumed to have a density higher than 95% of the theoretical density), this step could be skipped and the mass, m_3, taken equal to the mass of the dried test piece.

4.4.5 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

4.5 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.

4.6 Expression of results

4.6.1 Apparent solid density

Calculate the apparent solid density to the second decimal place according to Formula (1).

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

where

ρ_a is the apparent solid density, expressed in kilograms per cubic metre (kg/m^3);

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

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

ρ_1 is the density of the immersion liquid at the temperature of the test, expressed in kilograms per cubic metre (kg/m^3).

4.6.2 Bulk density

Calculate the bulk density to the second decimal place according to Formula (2).

$$\rho_b = \frac{m_1}{m_3 - m_2} \times \rho_1 \tag{2}$$

where

ρ_b is the bulk density, expressed in kilograms per cubic metre (kg/m^3);

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

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

m_3 is the mass of the soaked test specimen, expressed in kilograms (kg);

ρ_1 is the density of the immersion liquid at the temperature of the test, expressed in kilograms per cubic metre (kg/m^3).

4.6.3 Apparent porosity

Calculate the apparent porosity to the first decimal place according to Formula (3).

$$\pi_a = \frac{m_3 - m_1}{m_3 - m_2} \times 100 \tag{3}$$

where

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

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

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

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

5 Method by measurement of dimensions and mass (Method B)

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.

5.2 Apparatus

5.2.1 Balance, with an accuracy in accordance with Table 2 (with accuracy of 0,1 mg for a test specimen under 10 g and 0,001 % of the mass for a test specimen over 10 g).

5.2.2 Calibrated measuring device, capable of repeatable and accurate measurement in accordance with Table 2 (with accuracy of 0,01 mm or 0,05 % of smallest dimension), for example callipers or a micrometer in accordance with ISO 13385-1.

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 ± 5) °C.

5.2.4 Desiccator, for storage of test pieces.

Table 2 — Accuracy and errors of density and porosity measurement

Parameter	Method B: Geometric bulk density
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

5.3.1 General requirements

Materials for testing should be sampled in accordance with the guidance given in EN 1006. 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; specifically for ceramic matrix composites, the preferred shape is a machined rectangular parallelepiped.

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.

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

NOTE 2 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 might be present.

5.3.2 Specific requirements for ceramic matrix composites

In addition to the requirements of 5.3.1, the length (l) and width (d) of a test piece should be at least equivalent to those containing more than two unit cells. The thickness (h) of a test piece should be at least equivalent to that containing more than three plies for 2D woven and more than two unit cells for 3D woven.

NOTE The specific terms used above and related to ceramic matrix composites are defined in ISO 20507.

5.4 Procedure

5.4.1 Determination of the mass of the dried test pieces

Dry the test pieces in the oven (see 5.2.3) at (110 ± 5) °C to constant mass, i.e. until two successive weight measurements, taken 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.

NOTE For test pieces assumed to have no open porosity (i.e. assumed to have a density higher than 95 % of the theoretical density), the abovementioned drying procedure could be skipped and the specimens could be dried e.g. with a towel.

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

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 over 10 g.

5.4.2 Determination of the dimensions of the dried test pieces

Using the selected measuring device (see 5.2.2), measure the dimensions of each test piece in at least 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, for example the length, width and depth of a parallelepiped, the length and diameter for a disc or a rod.

If the dimensions of the test piece are too small to make three separate measurements in any direction, for example of the length of a rod of small diameter, a single measurement may be used, and then this simplified procedure reported [see Clause 6 j)].

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 Expression of results

Calculate the geometric volume of each test piece from its mean dimensions.

The bulk density, ρ_b, expressed in kilograms per cubic metre, kg/m^3, shall be calculated to the second decimal place according to Formula (4).

$$\rho_b = \frac{m_1}{V_b} \tag{4}$$

where

m_1 is the dry mass of the test piece;

V_b is the bulk volume of the test piece taken as its geometric volume.

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 document, i.e. ISO 18754;

b) the test laboratory and operator;

c) the date of measurement;

d) the type and configuration of the test specimen (material name);

