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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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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*.

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

1 Scope

This International Standard specifies a method for the determination of the apparent solid density, bulk density and apparent porosity of fine ceramics.

NOTE 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 may not be satisfactory. The method may also not give 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 °C*

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

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

4 Apparatus

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

4.2 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.

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

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

4.5 Immersion liquid, distilled water or ion exchanged water for materials that do not react with water. For materials that are sensitive to contact with water a suitable organic liquid shall be used.

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

4.7 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 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.

4.8 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.

5 Test specimen

The volume of each specimen shall be not less than 0,4 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.

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.

6 Procedure

6.1 Determination of mass of dry test specimen

Dry the test specimen in the drying oven (4.1) controlled at 100 °C ± 5 °C, allow it to cool to room temperature in a desiccator then weigh. Repeat this procedure until constant mass is reached. The mass thus determined is the mass of the dry test specimen, m_1. For any test specimen which may possibly break in 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.

6.2 Impregnation with liquid

6.2.1 Boiling method

Immerse the test specimen in the heating apparatus (4.3) 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.

6.2.2 Vacuum method

Place the test specimen in an air-tight container (4.8), 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 (4.5) 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.

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 vapor 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.

6.3 Determination of apparent mass of immersed test specimen

Place the test specimen in the halter or basket (4.6) and suspend the basket in the immersion liquid by use of the thin wire (4.7). Using the balance (4.2), measure the suspended mass when 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 (4.4).

6.4 Determination of mass of soaked test specimen

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.

6.5 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 described 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

6.6 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.

6.7 Repeat of measurement

The test shall be made more than twice on each test specimen to confirm the reproducibility of measurement.

7 Expression of results

7.1 Apparent solid density

The apparent solid density is given by Equation (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 meter;

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 meter.

7.2 Bulk density

The bulk density is given by Equation (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 meter;

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