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Fine ceramics (advanced ceramics, advanced technical ceramics) — Determination of absolute density of ceramic powders by pycnometer

Céramiques techniques — Détermination de la masse volumique absolue des poudres céramiques à l'aide d'un pycnomètre

Reference number
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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).

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. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

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For an explanation on 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 the following URL: www.iso.org/iso/foreword.html.

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

This second edition cancels and replaces the first edition (ISO 18753:2004), which has been technically revised.

The main changes compared to the previous edition are as follows:

— Clause 6 has been modified to include changes in list items d) and g) and to add a paragraph discussing factors affecting accuracy of test results;

— Table A.1 has been modified with new reference data for the absolute density of distilled water[1].

Fine ceramics (advanced ceramics, advanced technical ceramics) — Determination of absolute density of ceramic powders by pycnometer

1 Scope

This document specifies a method for determining the absolute particle density of fine ceramic powders or sintered parts using liquid pycnometry.

NOTE Other pycnometer methods like gas pycnometers (e.g. helium pycnometer), where a gas is used as media, also exist.

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

ISO 3507, *Laboratory glassware — Pyknometers*

ISO 6353-2, *Reagents for chemical analysis — Part 2: Specifications — First series*

ISO 6353-3, *Reagents for chemical analysis — Part 3: Specifications — Second series*

ISO 8213, *Chemical products for industrial use — Sampling techniques — Solid chemical products in the form of particles varying from powders to coarse lumps*

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 http://www.iso.org/obp

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

3.1
absolute particle density
absolute density of ceramic powders
density of an individual ceramic particle, a ceramic powder or sintered parts

Note 1 to entry: When an enclosed space occurs (pore) inside the particle or part, the space is considered to be part of the individual particle or part. The result of the test is then the absolute density of the ceramic particles or ceramic parts with closed porosity.

Note 2 to entry: If the intention of the test is to determine the absolute density of a ceramic material, the test is limited to particles or parts without closed porosity.

3.2
particle
ceramic powder or an amount of small sintered ceramic parts

EXAMPLE Grinding balls or other small parts.

Note 1 to entry: When the examined material has closed porosity inside the particles, the measured density is not the absolute density of the material but of the examined part.

3.3
pycnometry
method of measuring absolute particle density using a pycnometer

4 Preparation of measurement

4.1 Sampling

A representative sample (of powder or small parts) for analysis shall be taken in accordance with ISO 8213.

The maximum size of particles or parts which will be analysed shall be smaller than the neck of the used pycnometer.

4.2 Drying of sample

The selected sample is thoroughly dried in an air bath at approximately 110 °C, and then cooled in a desiccator to room temperature. In the case of powders or parts requiring a long drying time, the sample is thoroughly spread and stirred lightly once or twice midway through the drying process.

When the material is not stable at the heating temperature, the sample should be dried under reduced pressure instead of heating.

If necessary, it is recommended to record the mass loss as a function of time to ensure that the sample has reached equilibrium.

4.3 Preparation of immersion liquid

The immersion liquid used for pycnometry shall not be reactive and shall not dissolve the sample. A liquid with good wetting properties and a low evaporation rate under vacuum conditions shall be selected.

The following liquids for immersion may be used: distilled water; xylene as specified in ISO 6353-3; ethanol (e.g. 95 % purity by volume fraction) as specified in ISO 6353-2; or 1-butanol as specified in ISO 6353-3.

The absolute density of the immersion liquid at the measured temperature shall be determined using pycnometry, in accordance with ISO 758.

5 Apparatus

5.1 Pycnometer, Gay-Lussac type made of glass, of capacity 25 ml or 50 ml (see Figure 1), as specified in ISO 3507, or other suitable type of pycnometer.

5.2 Vacuum container, having an integrated pycnometer with a structure wherein internal conditions of the pycnometer can be observed. The pycnometer shall be connected to a vacuum pump, which is used to create the vacuum conditions.

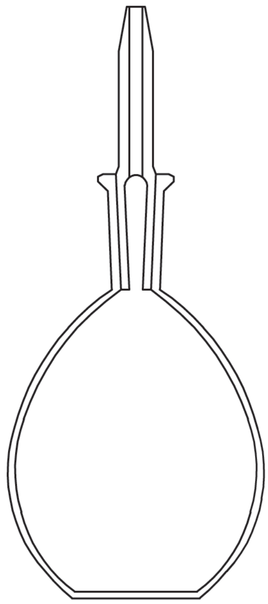

Figure 1 — Example of Gay-Lussac type pycnometer

5.3 Vacuum pump, capable of obtaining a vacuum of 666,5 Pa (5 mmHg) or better.

5.4 Vacuum gauge, capable of measuring from 0 kPa to 26,66 kPa (0 mmHg to 200 mmHg).

5.5 Balance, with a reciprocal sensitivity of 0,1 mg.

5.6 Thermometer, capable of reading to ±0,1 °C.

5.7 Ultrasonic bath, as an alternative to the vacuum pump and gauge.

6 Procedure

Measurements shall be carried out according to the following procedure. All masses shall be measured to the nearest 0,1 mg.

a) Clean the pycnometer (5.1), thoroughly dry it, then measure the mass, m_{p1}.

b) Transfer the powder or part sample (subsequently named as sample) to the pycnometer, filling the pycnometer to approximately one-third of its capacity, and measure the mass, m_{p2}.

c) Gently fill the pycnometer with immersion liquid until the sample is covered. It is permitted to slightly exceed this level. When the immersion liquid is dispensed, care should be taken not to scatter the sample.

d) Install the pycnometer containing the sample, immersed in liquid, in a vacuum container (5.2), reduce pressure to 13,33 kPa (100 mmHg) or less, and implement the degassing procedure. Allow

a reduction of pressure to a level at which the immersion liquid cannot boil, and carry out the degassing until bubbles cannot be generated from the sample.

An alternative degassing procedure to the evacuation method is to use an ultrasonic cleaning equipment (e.g. bath) until bubbles are no longer visible and a small amount of a suitable surface tension reducing surfactant in the liquid.

e) Remove the pycnometer from the vacuum container or from the ultrasonic bath and allow it to stand until the temperature of the liquid falls to room temperature. After thermal equilibrium is reached, record the temperature. The temperature of the sample/container shall be recorded and thermal equilibrium shall be reached before the mass is recorded.

f) Add the specific amount of immersion liquid to the pycnometer, and measure the mass, m_{p3}.

g) Remove the sample and the immersion liquid from the pycnometer. After washing and drying the pycnometer, fill with fresh immersion liquid to the specified level, and measure the mass, m_{p4}. Empty and dry the pycnometer and reweigh it (m_{p5}). If the apparent dry mass of the pycnometer varies by more than 0,5 mg, repeat the measurements.

h) Determine the absolute density of the immersion liquid at the measured temperature using pycnometry, in accordance with ISO 758, and determine the absolute density of the immersion liquid to the nearest 0,000 1 g/cm^3. Where distilled water is used as the immersion liquid, the absolute density for a given temperature, listed in Annex A, shall be used. The permissible temperature difference for measurements taken in Step f) shall be within ±1 °C.

The accuracy of the test results depends strongly on whether or not the whole surface of the analysed sample is completely wetted by the immersion liquid. Therefore, air bubbles should be avoided, for example, by using a small amount of a suitable surface tension reducing surfactant.

7 Calculation

Calculate the absolute particle density ρ at the temperature of measurement by means of Formula (1) by using the values obtained from Clause 6. Four significant digits shall be obtained.

$$\rho = \frac{(m_{p2} - m_{p1})}{(m_{p4} - m_{p1}) - (m_{p3} - m_{p2})} \rho_L \tag{1}$$

where

ρ is the absolute particle density in grams per cubic centimetre;

m_{p1} is the mass of the dry measuring container (pycnometer) in grams;

m_{p2} is the mass of the particle sample and the measuring container (pycnometer) in grams;

m_{p3} is the mass of the specific amount of particle sample, the immersion liquid and the measuring container (pycnometer) in grams;

m_{p4} is the mass of the specific amount of immersion liquid and the measuring container (pycnometer) in grams;

m_{p5} is the reweigh mass of the dry measuring container (pycnometer) in grams;

ρ_L is the density of the immersion liquid at measuring temperature in grams per cubic centimetre.

8 Tests in duplicate

Carry out tests in duplicate on representative analysis samples. If the difference between the original and duplicate results is greater than 0,03 g/cm^3, repeat the procedure.

9 Test report

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

a) the date of the test;

b) the name of the testing establishment;

c) a reference to this document, i.e. ISO 18753;

d) a description of the test material (manufacturer, type, batch or code number);

e) a description of the sample pre-treatments (evacuation time and evacuator, heating temperature and period, or alternatively the ultrasonic procedure);

f) the pycnometer (type, capacity);

g) a description of the immersion liquid used and temperature of measurements;

h) the absolute density of the sample;

i) comments concerning the test or test results.