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Fine ceramics (advanced ceramics, advanced technical ceramics) — Determination of densification properties of ceramic powders on natural sintering

Céramiques techniques (céramiques avancées, céramiques techniques avancées) — Détermination des propriétés de densification des poudres céramiques lors d'un frittage naturel

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Foreword

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

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 densification properties of ceramic powders on natural sintering

1 Scope

This document specifies the test method to determine the extent to which ceramic powder compacts made of granulated or ungranulated ceramic powders are densified, when they are sintered at a high temperature without the application of any external pressure or external densification force. The test method is applicable to pure oxides, mixtures of oxides and solid solutions, and is also applicable to non-oxides (e.g. carbides, nitrides) that can be sintered under vacuum or constant gas pressure (1 bar or less) to prevent oxidation or decomposition. The test method is not applicable to ceramics that can only be sintered using pressure-assisted sintering techniques such as hot pressing (HP), hot isostatic pressing (HIP), gas pressure sintering (GPS) or spark plasma sintering (SPS). Inorganic sintering additives can be used where their presence is reported.

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 3611, *Geometrical product specifications (GPS) — Dimensional measuring equipment: Micrometers for external measurements — Design and metrological characteristics*

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

ISO 17172, *Fine ceramics (advanced ceramics, advanced technical ceramics) — Determination of compaction properties of ceramic powders*

3 Terms and definitions

No terms and definitions are listed in this document.

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/

4 Principle

When ceramic powder compacts are heat-treated at high temperatures, they shrink and are densified due to sintering. The mass, dimensions (diameter and height), volume and apparent density of a ceramic powder compact are measured before and after sintering through thermal treatment. The variations in mass, dimensions, volume and apparent density depend on maximum temperature, dwell time, heating rate and apparent density after compaction, and can be expressed as a function of these parameters. For example, the variation in relative density can be plotted as a function of sintering temperature for each compacting pressure.

5 Symbols and designation

Symbols used throughout this document and their designations are given in Table 1.

Table 1 — Symbols, designations and units of mass, volume, density, dimension and sintering temperature

Symbol	Designation	Unit	Formula
D_a	Diameter of sample before sintering	mm	—
D	Diameter of sample after sintering	mm	—
H_a	Height of sample before sintering	mm	—
H	Height of sample after sintering	mm	—
m_a	Mass before sintering	g	—
m	Mass after sintering	g	—
V_a	Volume before sintering	cm^3	—
V	Volume after sintering	cm^3	—
T	Sintering temperature	°C	—
$\frac{\Delta D}{D_a}$	Relative diameter variation (shrinkage) at the end of sintering	—	(3)
$\frac{\Delta H}{H_a}$	Relative height variation (shrinkage) at the end of sintering	—	(4)
$\frac{\Delta m}{m_a}$	Relative mass variation at the end of sintering	—	(5)
$\frac{\Delta V}{V_a}$	Relative volume variation at the end of sintering	—	(6)
$\frac{\Delta \rho}{\rho_a}$	Relative density variation at the end of sintering	—	(7)
ρ_a	Apparent density before sintering	g/cm^3	(1)
ρ	Apparent density after sintering	g/cm^3	(2)
ρ_{th}	Theoretical density	g/cm^3	—

These characteristics are linked by relations in Formulae (1) to (7):

$$\rho_a = \frac{m_a}{V_a} \tag{1}$$

$$\rho = \frac{m}{V} \tag{2}$$

$$\frac{\Delta D}{D_a} = \frac{(D - D_a)}{D_a} \tag{3}$$

$$\frac{\Delta H}{H_a} = \frac{(H - H_a)}{H_a} \tag{4}$$

$$\frac{\Delta m}{m_a} = \frac{(m - m_a)}{m_a} \tag{5}$$

$$\frac{\Delta V}{V_a} = \frac{(V - V_a)}{V_a} \tag{6}$$

$$\frac{\Delta \rho}{\rho_a} = \frac{(\rho - \rho_a)}{\rho_a} \tag{7}$$

6 Apparatus

6.1 Cylindrical die, either double acting (floating type – see Figure 1) or single acting (see Figure 2), shall be made from hard material, preferably hardened steel or tungsten carbide. Upper and lower punches of adequate dimensions as indicated in Figure 1 and Figure 2 shall be used for producing cylindrical powder compacts. The upper part of the die shall be preferably designed to avoid damage to the powder compact during ejection due to spring-back. An ejection cone of height 5 mm, allowing an increase of the diameter at the top and the bottom of the die of approximately 1 %, as shown in Figure 1 and Figure 2, should be used.

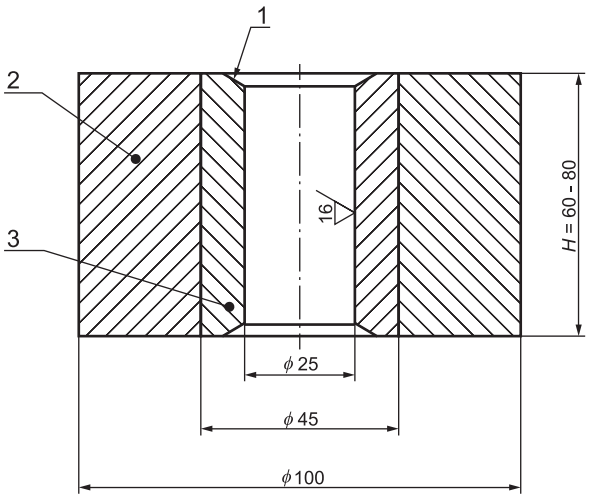
The die shall be of the floating type or of the type suspended from a spring (mode 1, see Figure 1), or of stationary type with only one moveable upper punch (mode 2, see Figure 2). The die shall be capable of making cylindrical powder compacts with a diameter of 10 mm to 26 mm and a height-to-diameter ratio of between 0,3 and 0,5 (mode 1), or with a diameter of 10 mm to 32 mm and a height-to-diameter ratio of between 0,15 and 0,25 (mode 2).

6.2 Furnace, should have a hot zone large enough to accommodate the required size and number of test pieces, and be capable of maintaining the test temperature (T) so that the maximum temperature variation in the hot zone is 10 °C. The furnace shall allow a constant heating rate, which can be controlled to within 2 °C/h. The furnace heating elements, thermal insulation and kiln furniture shall be selected to be chemically compatible with the test pieces, avoiding both surface reaction and generation of vapour pressure. The kiln furniture used to support the test pieces shall be a sintered piece of the test material with at least 80 % of theoretical density. If required, as is for non-oxides, the furnace shall be additionally capable of supplying constant vacuum or constant gas pressure (1 bar or less) of, for example, argon or nitrogen.

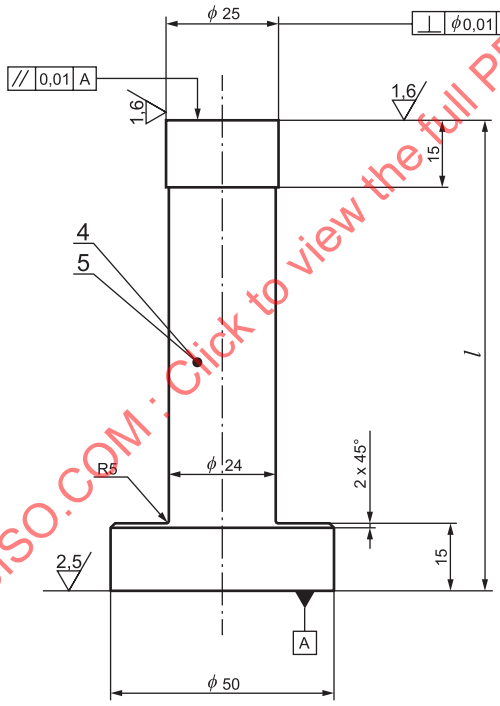
6.3 Press, capable of applying sufficient force with a precision of ±2 %.

6.4 Balance, capable of weighing at least 10 g with a resolution of ±0,001 g.

6.5 Micrometer, according to ISO 3611, or other suitable measuring device for measuring the dimensions of ceramic powder compacts with a resolution of ±0,01 mm.



a) Die

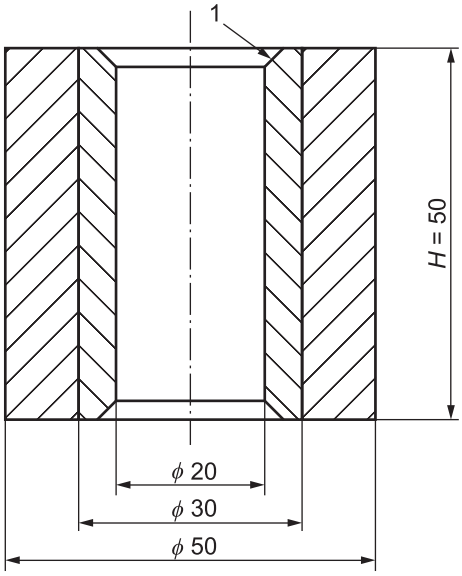


b) Upper and lower punch

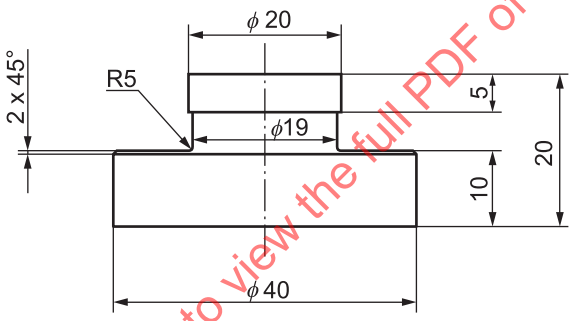
Key

1 ejection cone (height: 5 mm; increase of diameter: c. 1 %)
2 shrink ring
3 hard material
4 upper punch, $l = H - 10$
5 lower punch, $l = H + 35$

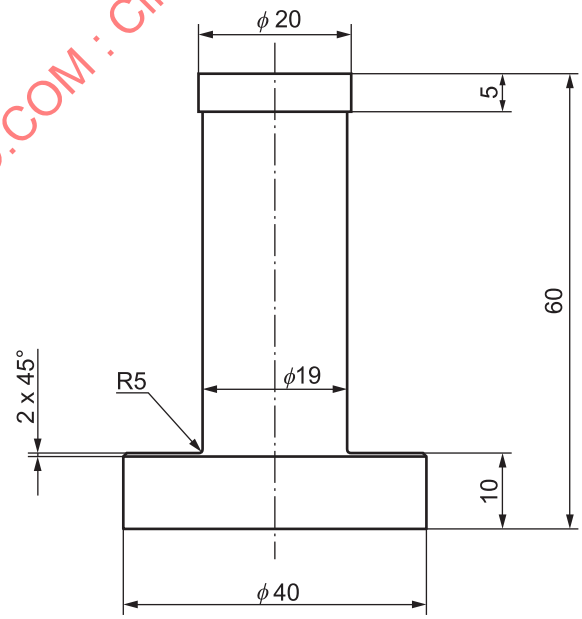
Figure 1 — Example of cylindrical die and punches for mode 1 compaction



a) Die



b) Lower punch



c) Upper punch

Key

1 ejection cone

Figure 2 — Example of cylindrical die and punches for mode 2 compaction

7 Sampling

7.1 In general, the granulated or ungranulated ceramic powder shall be tested in the as-received condition. In certain instances, the granulated or ungranulated ceramic powder can be dried. If the granulated or ungranulated ceramic powder is required to be dried, it shall be dried at (110 ± 5) °C for at least 24 h and cooled to room temperature in a desiccator until the test is performed. If the granulated or ungranulated ceramic powder contains organic additives or volatile substances, it shall not be dried.

7.2 Should there be any treatment (e.g. drying) of the granulated or ungranulated ceramic powder before the test, it shall be recorded in the test report.

8 Procedure

8.1 Compaction

Prepare the ceramic powder compacts in accordance with ISO 17172, making at least three pieces at each of the compacting pressures selected from those given in ISO 17172.

8.2 Heat treatment

8.2.1 Selection of test temperatures

Measurements shall be made over a range of test temperatures. The lower limit of the range is defined as the temperature at which the relative density (ρ/ρ_{th}) is approximately 0,9. The higher limit is defined as either:

a) the temperature at which the onset of de-densification resulting from grain growth is observed; or

b) the temperature at which a substantial loss of mass is recorded.

The temperature range shall be at least 100 °C.

Preliminary tests may be used to define the temperature range, in which case the thermal cycle specified in 8.2.2 shall be used.

8.2.2 Thermal cycle

Ceramics powder compacts shall be sintered under optimized heat treatment conditions that shall be recorded in the test report. The optimized heat treatment conditions consist of:

a) heating rates;

b) a dwell time at the test temperatures;

c) cooling rates;

d) atmosphere (e.g. air, vacuum, argon, nitrogen) and its pressure.

Cooling is normally achieved by switching off the heating in the furnace but can be controlled when required.

If the powder compacts contain organic additives, it may be necessary to remove them in a separate heat treatment at a lower temperature (<650 °C). Such an additional heat treatment for organic additive burn-out shall be recorded in the test report.

NOTE For oxide ceramics, the heat treatment conditions are often as follows:

1) heating from room temperature to 600 °C at 60 °C/h;

2) heating from 600 °C to the test temperature at 180 °C/h;

3) dwelling at the test temperature for at least 1 h;

4) natural cooling by switching off the heating in the furnace;

5) air atmosphere at ambient pressure.

8.3 Measurement

8.3.1 Measure the mass, m_a, the diameter, D_a, and the height, H_a of each ceramic powder compact before sintering. Calculate the apparent density, ρ_a, of each ceramic powder compact from the mass and dimensions in accordance with ISO 17172.

NOTE The mass, m_a, is the mass of the powder compact containing the ceramic powder, organic additives (if present) and humidity (if the powder is not dried). Organic additives and humidity will be responsible for most of the mass variation at the end of sintering. Knowing the amounts of organic additives and humidity within the powder compacts helps to distinguish this from other sources of mass variation.

The diameter of the powder compact shall be measured at least three times at different positions. For example, the diameter shall be measured at the top, middle and bottom along the height of powder compact. The height of the powder compact shall also be measured at least three times at different positions. The volume of the powder compact shall be calculated from its average diameter and its average height.

8.3.2 Carry out the heat treatment on the ceramic powder compacts in accordance with 8.2, making at least three test pieces for each combination of compaction pressure and sintering temperatures.

8.3.3 Measure the mass, m, the diameter, D, and the height, H, of each test piece after sintering. Calculate the apparent density, ρ, of each test piece from the mass and dimensions.

The diameter of the sintered test piece shall be measured at least three times at different positions. For example, the diameter shall be measured at the top, middle and bottom along the height of powder compact. The height of the sintered test piece shall also be measured at least three times at different positions. The volume of the sintered test piece shall be calculated from its average diameter and its average height.

9 Expression of results

9.1 Calculation

Report the densification properties of ceramic powders on natural sintering in three expressions of sintering shrinkage [as given in Clause 5, Formulae (3) and (4)], mass variation [as given in Clause 5, Formula (5)] and relative density as shown below in Formula (8):

$$\frac{\rho}{\rho_{th}} \tag{8}$$

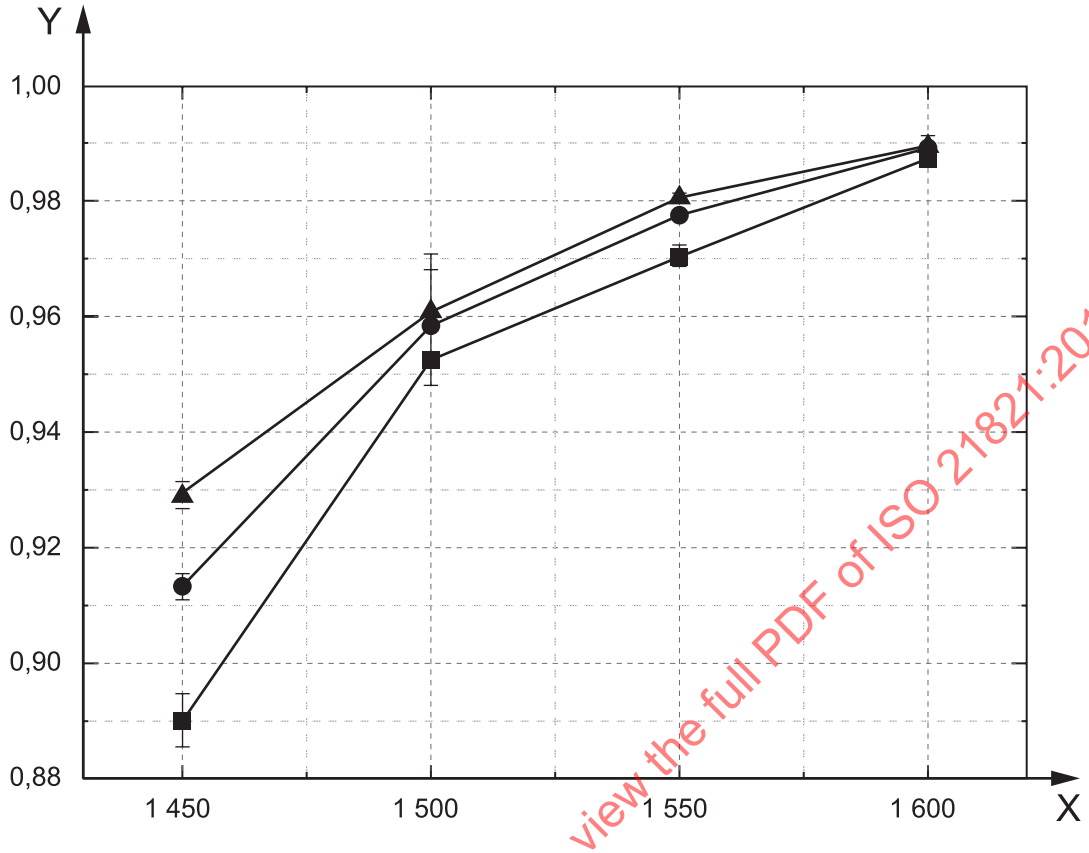
Express the densification properties as the average of the three results from three test pieces for each combination of compacting pressure and sintering temperature.

NOTE Mass variation, sintering shrinkage and relative density can be calculated and expressed in percentage.

9.2 Densification curve

Draw a set of the densification curves of ceramic powder representing the variation in the relative density as a function of the sintering temperature for each compaction pressure, as shown in Figure 3.

The images of microstructures of test pieces for each combination of compacting pressure and sintering temperature should be shown with the densification curve, as shown in Figure 4.



Key

X sintering temperature (°C)

Y relative density (ρ/ρ_{th})

50 MPa

100 MPa

200 MPa

Figure 3 — Example of densification curve of alumina powder