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ISO TC 206/WG 2

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

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Contents

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

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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	cm³	—
V	Volume after sintering	cm³	—
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/cm³	(1)
ρ	Apparent density after sintering	g/cm³	(2)
ρ_{th}	Theoretical density	g/cm³	—

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

$$\rho_a = \frac{m_a}{V_a} \quad (1)$$

$$\rho = \frac{m}{V} \quad (2)$$

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

Moved down [1]: Diameter of sample after sintering
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Moved down [2]: Height of sample after sintering
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Moved down [3]: Mass after sintering
Deleted: g¶
Moved (insertion) [3]
Deleted: ¶ V
Moved down [4]: Volume after sintering
Deleted: cm³¶
Moved (insertion) [4]
Deleted: ¶ ρ¶ ρth
Moved down [5]: Apparent density after sinteringTheoretical
Moved down [6]: Theoretical density
Moved down [7]: g/cm³
Moved down [8]: g/cm³
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$$\frac{\Delta H}{H_a} = \frac{(H - H_a)}{H_a} \tag{4}$$

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

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

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

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.

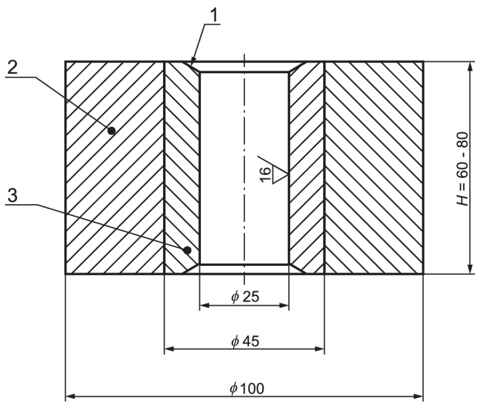
The die shall be of the floating type or of the type suspended from a spring (mode 1, see Figure 1), or of stationary type with only one moveable upper punch (mode 2, see Figure 2). The die shall be capable of making cylindrical powder compacts with a diameter of 10 mm to 26 mm and a height-to-diameter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

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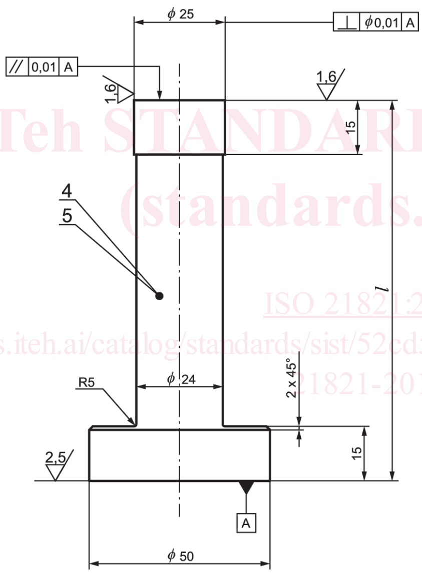

b) Upper and lower punch

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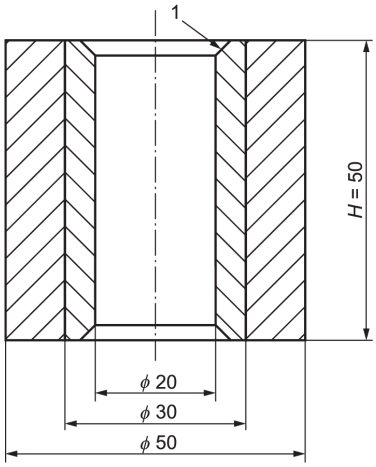
Key

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

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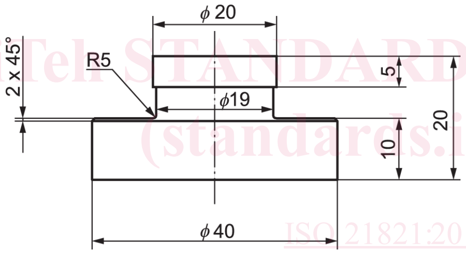

b) Lower punch

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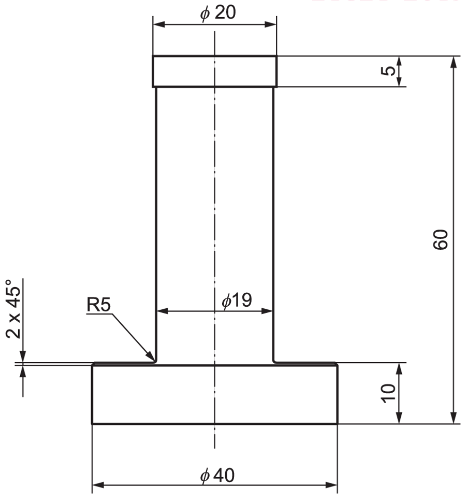

c) Upper punch

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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.

