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

Céramiques techniques — Détermination des propriétés de compactage des poudres céramiques

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

1 Scope

This International Standard specifies the testing method to determine the extent to which granulated or ungranulated ceramic powders are compacted, when subjected to uniaxial compressive loading in a confining die, under specified conditions.

2 Principle

Granulated or ungranulated ceramic powders are compacted uniaxially in a confining die by double-action pressing. Samples of the ceramic powders may be pressed either at a single specified pressure or at a series of specified pressures. After ejection from die, the apparent density of the ceramic powder compact is determined.

The apparent density obtained in the former case represents the compaction properties of the ceramic powder at the specified pressure. The apparent densities obtained in the latter case are utilized for drawing the compaction curve of the ceramic powder, which is a plot of apparent density as a function of compaction pressure.

3 Symbols and designation

Symbol	Designation	Unit
ρ_a	Apparent density	g/cm^3
m	Mass of ceramic powder compact	g
V	Volume of ceramic powder compact	cm^3

If the apparent density is measured at only one specified pressure, for example 100 MPa, the symbol becomes $\rho_a(100)$.

4 Apparatus

4.1 Cylindrical die

A cylindrical die should be made from hard material, preferably hardened steel or tungsten carbide. The die shall contain two upper and lower punches for producing cylindrical powder compacts and shall be of the floating type or of the type suspended from a spring. The die shall be capable of making cylindrical powder compacts with a diameter from 10 mm to 26 mm and a height to diameter ratio between 0,3 and 0,5, as indicated in figure 1. The upper part of the die shall be preferably designed to avoid damage to the powder

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compact during ejection due to spring-back. An ejection cone of height 5 mm, allowing an increase of the diameter at the top of the die of approximately 1%, as shown in figure 1, should be used.

4.2 Press

A press capable of applying sufficient force with a precision of ± 2%.

4.3 Balance

A balance capable of weighing at least 10 g with a precision of ± 0,0005 g.

4.4 Micrometer

A micrometer or other suitable measuring device for measuring the dimensions of ceramic powder compacts with a precision of ± 0,01 mm.

5 Sampling

5.1 In general, the powder should be tested in the as-received condition. In certain instances, the powder may be dried. If the powder is required to be dried, it should be dried at (110 ± 5)℃ for at least 24 h and cooled to room temperature in a desiccator until the test is performed. If the powder contains organic additives or volatile substances, it should not be dried.

5.2 Should there be any treatment (e.g. drying) of the powder before the test, it shall be recorded in the test report.

6 Procedure

6.1 Quantity

The quantity of the test sample shall be chosen to give the required number of powder compacts with dimensions as specified in 4.1. Per compaction pressure, three powder compacts shall be pressed (see 6.6). If necessary, preliminary tests should be made in order to establish the quantity of powder which is needed for fulfilling this requirement.

6.2 Cleaning of die and punches

Wipe the die cavity and the punches with a soft clean paper towel soaked with an appropriate solvent such as acetone. Allow the solvent to evaporate.

6.3 Powder testing conditions

6.3.1 Powders which do not contain a lubricant shall be compacted either :

a) in a dry die

Note Seizure and excessive die wear may occur, particularly at high compaction pressures.

b) in a die with lubricated walls (see 6.4.2)

c) in a dry die, after mixing a lubricant with the powder (see 6.4.3).

6.3.2 Powders which contain a lubricant shall be compacted in a dry die.

6.4 Lubrication

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6.4.1 Lubrication of the die walls or of the powder is likely to modify the compaction results. Similarly, depending on the type and the quantity of lubricant added to the powder, the results may vary within wide ranges. The test report (see clause 8) shall therefore mention whether or not lubrication had taken place, and if the lubrication was carried out on the walls of the die or on the powder.

Use one of the following two methods (see 6.4.2 and 6.4.3) if lubrication is applied.

6.4.2 In order to lubricate the die wall, apply a mixture or a solution of a lubricant in the volatile organic liquid, for example 20 g of stearic acid in 980 g of acetone. After any excess liquid has been drained away, allow the solution adhering to the walls to evaporate leaving a thin layer of lubricant.

Alternatively, press inert spheres (for example, polyurethane spheres) coated with lubricant in the die before testing. After removal of the pressed disc of inert material, a thin layer of lubricant remains on the walls of the die.

6.4.3 Lubricate the powder to be tested by thoroughly mixing it with a quantity (0,5 wt% to 2,0 wt%) of a suitable solid lubricant (for example, zinc stearate or stearic acid).

6.5 Compaction and ejection

6.5.1 Insert the lower punch into the die in order to leave a free volume in the die large enough to contain the ceramic powder (see figure 2(a)).

6.5.2 Pour the powder into the die and level off to a uniform filling height (see figure 2(a)).

6.5.3 Wipe the wall of the die, to remove any powder that may adhere to it during filling of the die.

6.5.4 Insert the upper punch into the die (see figure 2(b)).

6.5.5 Position the die with its upper and lower punches between the plate ends of the press. Apply and release a preliminary pressure. The pressure shall be lower than the lowest pressure used in the test.

Apply the selected test pressure and maintain for 1 min (see figure 2(c)).

The apparent density of the powder compact depends on the displacement speed of the punch and the rate of increase of the force, respectively. Therefore, the displacement speed of the punch or the rate of increase of the force shall be recorded in the test report.

Note Taking into account the different press technology, it is difficult to specify a rate of increase of the force as well as the displacement speed of the punch. At least one of these parameters is to be mentioned on the test report.

6.5.6 Perform decompression slowly (for example, 10 mm/min) to avoid breaking the powder compact.

6.5.7 Pull out the upper punch from the die.

6.5.8 Eject the powder compact by using a relative movement between the die and the lower punch (see figure 2(d)). Transfer the powder compact to a desiccators and keep it for 1 h to allow a possible expansion due to spring-back. Remove the powder compact from the desiccator, weigh its mass to the nearest 0,0005 g and measure its dimensions to the nearest 0,01 mm.

6.6 Compaction pressures

For plotting the compaction curve of the powder at different pressures, the pressures applied shall be 25 MPa, 50 MPa, 100 MPa and 200 MPa. If compaction properties are to be determined at a single pressure only, this pressure shall be one of the pressures given above.

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7 Expression of results

7.1 Calculation

The apparent density of the powder compact is given by the formula :

$$\rho_a = m / V \quad (1)$$

Calculate the apparent density in g/cm^3 to the nearest 0,02 g/cm^3.

Express the compaction properties for a given compacting pressure as the average of the three apparent densities, obtained at the specified pressure.

7.2 Compaction curve

Draw the compaction curve of the powder through points representing the average of determinants of ρ_a at each of the specified pressures (the variation in apparent density of powder compacts as a function of compressive pressure), as shown in figure 3.

8 Test report

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

a) the name of the testing establishment

b) date of the test, report identification and number, operator, signatory

c) temperature and relative humidity in laboratory

d) a reference to this International Standard

e) all details for identification of the ceramic powder (material type, manufacturer, batch or code number)

f) pre-treatment condition of granulated or ungranulated ceramic powder

g) the type, nature and amount of lubricant and any other organic additives used in the test, including

   1) lubrication of the die walls, or no lubrication

   2) lubrication of the powder, or no lubrication (state how the lubrication has been added)

   3) other organic additives

h) the compacting pressures

i) the displacement speed of the punch (or the rate of increase of the force)

j) the dimensions, mass and apparent density of the powder compacts

k) the result obtained including the compaction curve

l) details of any occurrence which may have affected the result

m) comments about the test or test results

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