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Sodobna tehniļna keramika - Preskusne metode za keramiļne praġke - 10. del: Ugotavljanje lastnosti za stiskanje

Advanced technical ceramics - Methods of test for ceramic powders - Part 10: Determination of compaction properties

Hochleistungskeramik - Prüfverfahren für keramische Pulver - Teil 10: Bestimmung der Verdichtungseigenschaften

Céramiques techniques avancées - Méthodes d'essai pour poudres céramiques - Partie 10: Détermination des propriétés de compaction

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Advanced technical ceramics - Methods of test for ceramic powders - Part 10: Determination of compaction properties

Céramiques techniques avancées - Méthodes d'essai des poudres céramiques - Partie 10: Détermination des propriétés de compaction

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EUROPEAN COMMITTEE FOR STANDARDIZATION
COMITÉ EUROPÉEN DE NORMALISATION
EUROPÄISCHES KOMITEE FÜR NORMUNG

Management Centre: rue de Stassart, 36 B-1050 Brussels

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Foreword

This document (EN 725-10:2007) has been prepared by Technical Committee CEN/TC 184 “Advanced technical ceramics”, the secretariat of which is held by BSI.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by March 2008, and conflicting national standards shall be withdrawn at the latest by March 2008.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. CEN [and/or CENELEC] shall not be held responsible for identifying any or all such patent rights.

This document supersedes EN 725-10:1996.

EN 725 *Advanced technical ceramics — Methods of test for ceramic powders* was prepared in parts as follows:

Part 1: *Determination of impurities in alumina*

Part 2: *Determination of impurities in barium titanate*

Part 3: *Determination of the oxygen content of non-oxides by thermal extraction with a carrier gas*

Part 4: *Determination of oxygen content in aluminium nitride by XRF analysis*

Part 5: *Determination of particle size distribution*

Part 6: *Determination of the specific surface area* [withdrawn]

Part 7: *Determination of the absolute density* [withdrawn]

Part 8: *Determination of tapped bulk density*

Part 9: *Determination of un-tapped bulk density*

Part 10: *Determination of compaction properties*

Part 11: *Determination of densification on natural sintering*

Part 12: *Chemical analysis of zirconia*

Parts 6 and 7 of the series were superseded in 2005 by EN ISO 18757 and EN ISO 18753 respectively.

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard: Austria, Belgium, Bulgaria, Cyprus, Czech Republic, Denmark, Estonia, Finland, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Romania, Slovakia, Slovenia, Spain, Sweden, Switzerland and the United Kingdom.

1 Scope

This Part of EN 725 describes methods for the determination of the compaction behaviour of ungranulated or granulated ceramic powders, when subjected to uniaxial compressive loading in a rigid die, under specified conditions.

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.

EN ISO/IEC 17025, *General requirements for the competence of testing and calibration laboratories (ISO/IEC 17025:2005)*

3 Principle

The powder is compacted uniaxially in a rigid die by double-action pressing. Samples of the powder may be pressed either at a single pressure or at a series of pressures. After ejection from the die, the apparent geometrical density of the compact is determined.

Where one pressure is used, the density obtained represents the compaction properties of the powder at that pressure. Where more than one pressure is used, the densities obtained are utilized for drawing the compaction curve of the powder, which is a plot of its density as a function of the compacting pressure.

4 Symbols

The symbols and associated units used in this part of EN 725 are set out in Table 1.

Table 1 — Symbols and units

Symbol	Designation	Unit
ρ_{geom}	Apparent geometrical density	kg/m^3
m	Mass of the compact	g
V	Volume of the compact	cm^3

If the apparent density is measured at one pressure only, for example 200 MPa, the symbol becomes, e.g. ρ_{geom} (200).

5 Apparatus

5.1 Die, which shall be made from hard material (e.g. tungsten carbide). The cylindrical die shall contain two punches for producing cylindrical compacts and shall be of the floating type or of the type suspended from a spring. The die shall be capable of making compacts of diameter 20 mm to 26 mm with a height to diameter ratio between 0,3 and 0,5, with tolerances as indicated in Figure 1. The upper part of the die shall be (preferably) designed to avoid damage to the compact during the ejection phase due to springback. 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.

The die may be fitted with a venting valve.

5.2 Press, capable of applying sufficient force with an accuracy of ± 1 %.

5.3 Balance, capable of weighing at least 10 g, to the nearest 0,005 g.

5.4 Micrometer, conforming to ISO 3611, or other suitable measuring device for measuring the dimensions of the compacts with a resolution of ± 0,01 mm.

6 Procedure

6.1 Drying

A sample containing organic additives shall be used as received.

A sample without organic additives shall be dried at 110 °C ± 5 °C until constant mass is attained. Store in a desiccator until the test is performed. During drying the sample layer shall have a maximum thickness of 5 mm.

6.2 Quantity

Three compacts with dimensions as specified in 5.1 shall be prepared for each pressure used in the determination. The quantity of powder shall be sufficient for the preparation of the total number of compacts (3 x the number of pressures used).

NOTE If necessary, preliminary tests should be made to establish the quantity of powder necessary to conform to this requirement.

6.3 Cleaning of the 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.

6.4 Powder testing conditions

Powders shall be compacted:

either

1) in a dry die

NOTE For powders not containing a lubricant, seizure and excessive die wear may occur, particularly at high compacting pressures

or

2) in a die with lubricated walls (see 6.5),

or

3) in a dry die, after mixing a lubricant with the powder (see 6.5.3).

6.5 Lubrication

6.5.1 Lubrication of the die walls or of the powder is likely to modify the compaction results. Similarly, depending on the type and 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 6.5.2 and 6.5.3) if lubrication is applied.

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

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

6.5.3 Lubricate the powder to be tested by thoroughly mixing it with an appropriate amount of a suitable solid lubricant (for example zinc stearate or stearic acid).

6.6 Compacting pressures

For plotting the compaction curve of a powder, a minimum of four pressures in a range agreed between the parties concerned shall be used. If compaction properties are to be determined at a single pressure only, this pressure shall also be agreed between parties prior to the test.

6.7 Compacting and ejection

6.7.1 Position the die using spacers between the die and the foot of the lower punch, to leave a free volume in the die large enough to contain the bulk powder (see Figure 2).

6.7.2 Pour a known weight of powder into the die, sufficient to almost fill the die cavity. The weight shall be the same for each compact in a series.

6.7.3 Remove any remaining powder adhering to the wall of the die cavity.

6.7.4 Insert the upper punch into the die.

6.7.5 Position the die with its punches between the plate ends of the press. Apply and release a preliminary pressure. This pressure shall be lower than the lower pressure used in the test. Remove the spacers supporting the die.

NOTE 1 If the die is supported by springs or by a similar system, it is unnecessary to apply this preliminary pressure.

Apply the selected test pressure and maintain for 1 min.

NOTE 2 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.

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

6.7.7 Eject the compact by using a relative movement between the die and the lower punch. Transfer the compact to a desiccator for 1 h to allow a possible expansion due to springback. Remove the compact from the desiccator, weigh to the nearest 0,005 g and measure to the nearest 0,01 mm, the dimensions (both height and diameter) in 3 different places, and take the average.

7 Expression of results

7.1 Calculation

The apparent geometrical density of the compact is given by the equation:

$$\rho_{geom} = \frac{m}{V} \quad (1)$$

where:

m is the mass of the compact, in grams;

V is the volume of the compact, in cubic centimetres.

Calculate the apparent geometrical density in grams per cubic centimetre and then convert the answer to kilograms per cubic metre, expressed to the nearest 20 kg/m^3.

Express the compaction properties for a given compacting pressure as the average of the three density figures, calculated to the nearest 20 kg/m^3, obtained at the compacting pressure.

7.2 Compaction curve

Draw the compaction curve of a powder through points representing the average of determinations of ρ_{geom} at each of the pressures tested.

8 Test report

The test report shall be in accordance with the reporting provisions of EN ISO/IEC 17025, and shall include at least the following information:

a) name and address of the testing establishment;

b) date of the test;

c) on each page, a unique report identification and page number;

d) customer name and address;

e) reference to this standard, i.e. determined in accordance with EN 725-10;

f) authorizing signature;

g) any deviation from the method described, with appropriate validation, i.e. demonstrated to be acceptable to the parties involved;

h) description of the powder (e.g. material type, manufacturer code, batch or code number);

i) 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 lubricant has been added);

   3) other organic additives;