SLOVENSKI STANDARD
SIST EN 725-10:2000

01-december-2000

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

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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

Ta slovenski standard je istoveten z: EN 725-10:1997

ICS:

81.060.30 Sodobna keramika Advanced ceramics

SIST EN 725-10:2000 en

EUROPEAN STANDARD

NORME EUROPÉENNE

EUROPÄISCHE NORM

EN 725-10

March 1997

ICS 81.060.99

Descriptors: ceramics, powdery materials, compression tests, compacting, determination, compressibility, testing conditions

English version

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CEN

European Committee for Standardization
Comité Européen de Normalisation
Europäisches Komitee für Normung

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Foreword

This European Standard has been prepared by Technical Committee CEN/TC184 "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of an identical text or by endorsement, at the latest by September 1997, and conflicting national standards shall be withdrawn at the latest by September 1997.

EN 725 consists of 11 Parts:

Part 1 : Determination of impurities in alumina
Part 2 : Determination of impurities in barium titanate (ENV)
Part 3 : Determination of oxygen content of non-oxides by thermal extraction
Part 4 : Determination of oxygen content of non-oxides by XRF analysis (ENV)
Part 5 : Determination of particle size distribution
Part 6 : Determination of specific area
Part 7 : Determination of absolute density
Part 8 : Determination of tapped density
Part 9 : Determination of untamped bulk density
Part 10 : Determination of compaction properties
Part 11 : Determination of reactivity on sintering (ENV)

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard: Austria, Belgium, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxembourg, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland and the United Kingdom.

1 Scope

This Part of EN 725 describes methods for the determination of the extent to which a ceramic powder is compacted, when subjected to uniaxial compressive loading in a confining die, under specified conditions.

NOTE : An example of the reporting of results is shown in annex A.

2 Normative references

This European Standard incorporates by dated or undated reference, provisions from other publications. These normative references are cited at the appropriate places in the text and in the publications listed hereafter. For dated references, subsequent amendments to or revisions of any of these publications apply to this European Standard only when incorporated in it by amendment or revision. For undated references the latest edition of the publication referred to applies.

ISO 3611 Micrometer callipers for external measurement.

3 Principle

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

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

4 Symbols and designation

Table 1

Symbol	Designation	Unit
ρ_a	Apparent 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 specified pressure only, for example 200 MPa, the symbol becomes ρ_a (200).

5 Apparatus

5.1 Die, which shall be made from hard material, preferably 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 phenomenon. 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 ± 2 %.

5.3 Balance, capable of weighing at least 10 g with an accuracy of ± 0,005 g.

5.4 Micrometer (see ISO 3611), or other suitable measuring device for measuring the dimensions of the compacts with an accuracy of ± 0,01 mm.

6 Procedure

6.1 Drying

NOTE : Powders containing organic additives should not be dried.

Dry the powder at 110 °C ± 5 °C for at least 24 h and until constant mass is attained. Store in a desiccator until the test is performed. During drying the powder layer shall have a maximum thickness of 5 mm.

6.2 Quantity

The quantity of the test sample shall be chosen to give the required number of compacts (see clause 8), with dimensions as specified in 5.1. Three compacts shall be used for each pressure used in the determination. If necessary, preliminary tests should be made in order to establish the quantity of powder which is needed for fulfilling 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

6.4.1 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 compacting pressures,

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

c) in a dry die, after mixing a lubricant with the powder (see 6.4.2).

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

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 the 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 a quantity (0,5 % to 1,5 %) of a suitable solid lubricant (for example zinc stearate or stearic acid).

6.6 Compacting and ejection

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

6.6.2 A known weight of powder, which shall be the same for each compact in a series, sufficient to almost fill the die cavity is poured into the die.

6.6.3 Wipe the wall of the die remaining free, to remove any powder that may have adhered to it during the filling of the mould.

6.6.4 Insert the upper punch into the die.

6.6.5 Position the die with its punches between the plate ends of the press. Apply and release a preliminary pressure. The pressure shall be lower than the lower pressure used in the test. Remove the spacers supporting the die. If the die is supported by springs, or by some similar system, it is unnecessary to apply this preliminary pressure.

Apply the selected test pressure and maintain for 1 min.

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.

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

6.6.7 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 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.

6.7 Compacting pressures

For plotting the compaction curve of a 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.

7 Expression of results

7.1 Calculation

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

$$\rho_a = \frac{m}{V}$$

where:

m is the mass of the compact, in grams

V is the volume of the compact, in cubic centimetres

Calculate the 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 specified pressure.