



BSI Standards Publication

# Fine ceramics (advanced ceramics, advanced technical ceramics) — Determination of compressive strength of ceramic granules

**National foreword**

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**Fine ceramics (advanced ceramics,  
advanced technical ceramics) —  
Determination of compressive  
strength of ceramic granules**

*Céramiques fines (céramiques avancées, céramiques techniques  
avancées) — Détermination de la force de compression des  
granules de céramique*





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## Foreword

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The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see [www.iso.org/directives](http://www.iso.org/directives)).

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For an explanation on the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the WTO principles in the Technical Barriers to Trade (TBT) see the following URL: [Foreword - Supplementary information](#).

The committee responsible for this document is ISO/TC 206, *Fine ceramics*.

# Fine ceramics (advanced ceramics, advanced technical ceramics) — Determination of compressive strength of ceramic granules

## 1 Scope

This International Standard specifies the testing method to determine the compressive strength of ceramic granules, when subjected to uniaxial compressive loading in a confining die, under specified conditions.

## 2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 7500-1, *Metallic materials — Calibration and verification of static uniaxial testing machines — Part 1: Tension/compression testing machines — Calibration and verification of the force-measuring system*

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

## 3 Principle

In general, a ceramic powder compact is made of ceramic granules. When ceramic granules are compacted uniaxially in a confining die by pressing, first they are rearranged during compaction. However, when a pressure higher than the critical pressure is applied to ceramic granules, the deformation and the fracture of granulated powder also occur. The final density of powder compact depends on the compressive strength (critical pressure) of ceramic granules. When ceramic granules are pressed in a die by using a universal testing machine (UTM), a load-displacement (or load-strain) curve can be obtained. Load-displacement curve can be converted into pressure-density curve by using the dimensions and density of a powder compact ejected from the die. The compressive strength can be determined from the slope change in the pressure-density curve.

## 4 Symbols and designation

Symbol	Designation	Unit
$\rho_p$	Green density of powder compact	g/cm <sup>3</sup>
$m$	Mass of powder compact	g
$V$	Volume of powder compact	cm <sup>3</sup>
$A$	Area of powder compact	cm <sup>2</sup>
$h$	Height of powder compact	cm
$l$	Displacement of powder compact	cm
$l_f$	Final displacement of powder compact	cm

## 5 Apparatus

**5.1 Cylindrical die**, which shall be made from a hard material, preferably hardened steel or tungsten carbide. The die for producing cylindrical compacts shall contain two punches and shall be of a floating type

or of type suspended from a spring. The die shall be capable of making powder compacts with a diameter in the range of 10 mm to 25 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 compact during ejection due to the spring-back phenomenon. An ejection cone of height about 5 mm, allowing a gentle increase of the diameter at the top of the die of approximately 1 %, as shown in [Figure 1](#), should be used.

**5.2 Universal Testing Machine (UTM)**, which can apply pressure at constant velocity to the ceramic granules. The accuracy of the test machine shall be in accordance with ISO 7500-1, Grade 1 (accuracy 1 % indicated load). The UTM shall be capable of simultaneously measuring compressive displacement and applied load and recording the load-displacement curve during the measurement.

**5.3 Balance**, capable of weighing a mass of at least 10 g with a precision of  $\pm 0,001$  g.

**5.4 Micrometer**, or other suitable measuring device for measuring the dimensions of the powder compacts with a precision of  $\pm 0,01$  mm or better.

## 6 Sampling

**6.1** In general, the ceramic granules should be tested in the as-received condition. In certain instances, the granules may be dried. If the granules are required to be dried, they should be dried at  $110\text{ °C} \pm 5\text{ °C}$  for at least 24 h and cooled to room temperature in a desiccator before the test is performed. If the granules contain organic additives or volatile substances, they should not be dried. During drying, the granule layer shall have a maximum thickness of 5 mm.

**6.2** Should there be any treatment (e.g. drying) of the ceramic granule before the test, this shall be recorded in the test report.

**6.3** The test shall be carried out on three different test portions.

## 7 Procedure

### 7.1 Quantity

The quantity of the test sample shall be chosen to give the required number of powder compacts (see [8.1](#)), with dimensions as specified in [5.1](#). If necessary, preliminary tests should be made in order to establish the quantity of powder which is needed for fulfilling this requirement.

### 7.2 Cleaning of the die and the 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.

### 7.3 Powder testing conditions

**7.3.1** Ceramic granules which do not contain a lubricant shall be compacted in either of the following:

a) dry die;

NOTE Seizure and excessive die wear can occur, particularly at high compaction pressures.

b) die with lubricated walls (see [7.4.1](#));

c) dry die, after mixing a lubricant with the powder (see [7.4.2](#)).



**7.3.2** Ceramic granules which contain a lubricant shall be compacted in a dry die.

## **7.4 Lubrication**

**7.4.1** Lubrication of the die walls or of the ceramic granules is likely to modify the compressive strength results. Similarly, depending on the type and the quantity of lubricant added to the ceramic granules, the results of packing and rearrangement can vary within wide ranges. The test report (see [Clause 9](#)) shall therefore mention whether or not lubrication had taken place, if the lubrication was carried out on the walls of the die or on the ceramic granules.

Use one of the following two methods (see [7.4.2](#) and [7.4.3](#)) if lubrication is applied.

**7.4.2** 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 (polyurethane spheres for instance) 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.

**7.4.3** If the granules are to be tested in a lubricated condition, lubricate the granules to be tested by thoroughly mixing it with a quantity (0,1 wt% to 3,0 wt%) of a suitable solid lubricant (for example, zinc stearate or stearic acid).

## **7.5 Compaction and ejection**

**7.5.1** Insert the lower punch into the die in order to leave a free volume in the die large enough to contain the bulk powder [see [Figure 2 a](#)].

**7.5.2** Pour the granules into the die and level off to a uniform filling height [see [Figure 2 a](#)].

**7.5.3** Wipe the wall of the die, to remove any granule that might have adhered to it during the filling of the die.

**7.5.4** Insert the upper punch into the die [see [Figure 2 b](#)].

**7.5.5** Position the die with its punches between the platens of UTM. Move the position of platens until the upper and lower parts of die meet the platens.

**7.5.6** Increase the uniaxial compressive loading to the ceramic granules using a constant crosshead speed of 1 mm/min and observe the development of the recorded load-displacement curve that shall be recorded.

**7.5.7** The slope of load-displacement curve decreases (see [Figure 3](#)) and becomes approximately constant as the normal compressive load to the ceramic granules increases. Stop the test and remove the compressive load when the slope of load-displacement curve is constant.

**7.5.8** Remove the lower punch from the die. Invert the die and, using the UTM, push up the upper punch to eject the powder compaction from the die. Compaction and compact removal steps are indicated in [Figure 2](#).

**7.5.9** Record the mass ( $m$ ) and the size [diameter and height ( $h$ )] of the ejected ceramic powder compact.

## 8 Expression of results

### 8.1 Load-displacement curve

Compaction of ceramic granules using a UTM allows a load-displacement curve to be recorded as shown in [Figure 3](#). From the load-displacement curve shown in [Figure 3](#), the density of the ceramic powder compact shall be calculated according to Formula (1) (for symbols, see [Clause 4](#)):

$$\rho_p = \frac{m}{V} = \frac{m}{[A \times \{h + (l_f - l)\}]} \quad (1)$$

Express the compaction properties for a given compressive pressures as the average of the density values determined from three independent measurements.

### 8.2 Pressure-density curve

The load-displacement curve recorded during the measurement by the UTM shall be converted into the pressure (= load/area of powder compact) density curve using Formula (1) given in [8.1](#) (see [Figure 4](#)). The x-axis indicating the compressive pressure shall be expressed on a logarithmic scale. The pressure-density curve should show two distinctive regions with different slopes. At low pressure region, the slope should be small while at pressure region above a certain level, the slope should be higher (see [Figure 4](#)).

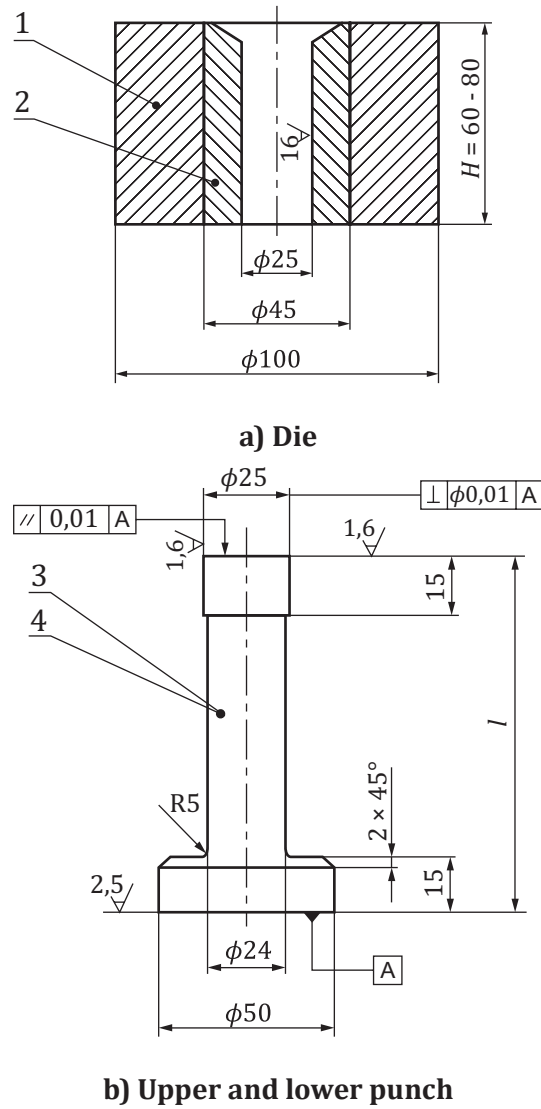
### 8.3 Compressive strength of ceramic granules

Extend each straight line from the two regions having approximately constant slopes and determine the intercept point. Take this intersection as the compressive strength of the ceramic granules as shown in [Figure 4](#).

## 9 Test report

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

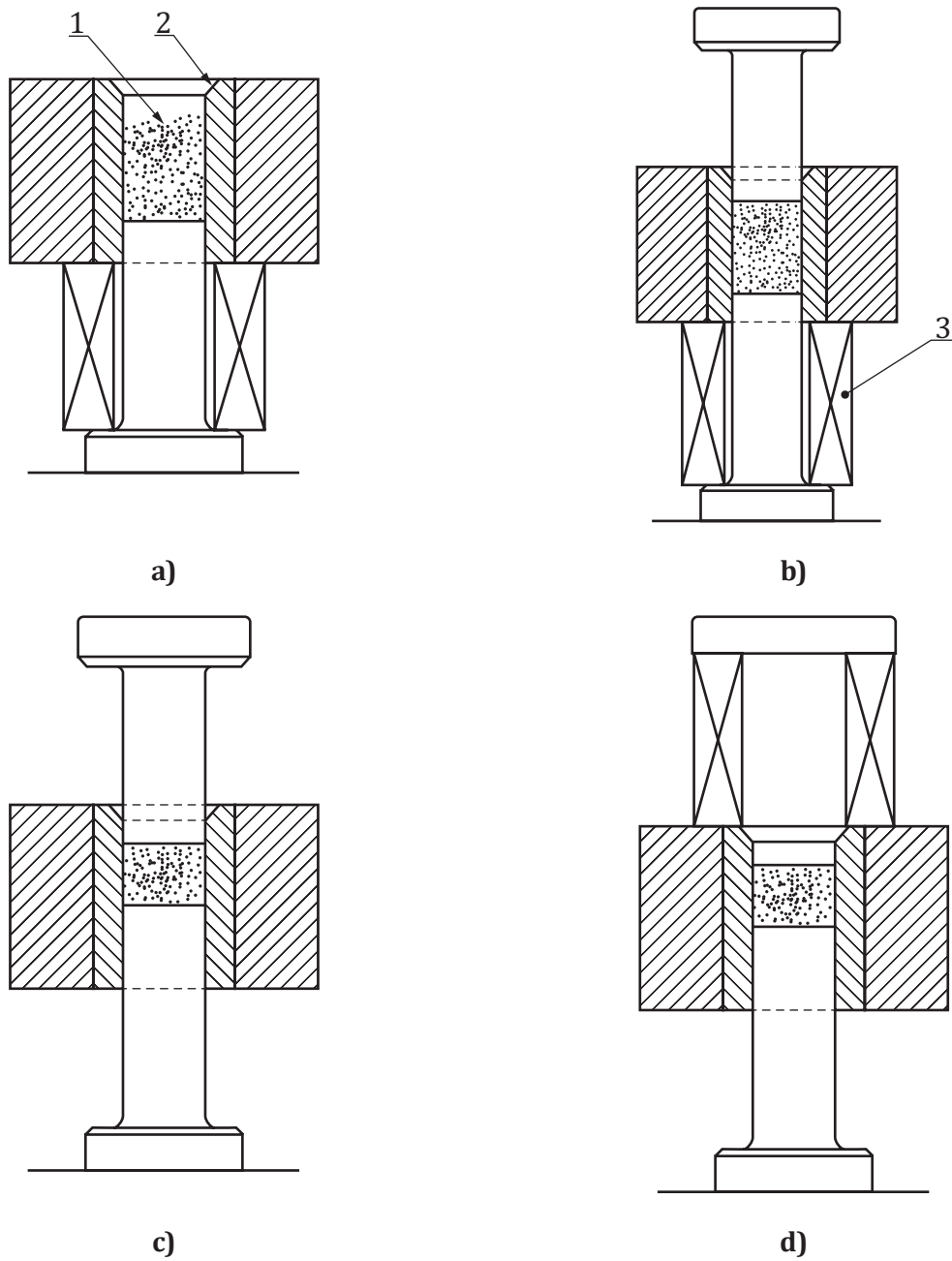
- a) reference to this International Standard, i.e. ISO 18591;
- b) name of the testing establishments;
- c) date of the test, report identification and number, operator, signatory;
- d) temperature and relative humidity in the laboratory;
- e) description of the powder (material manufacturer, batch or code number);
- f) dimensions of the powder compacts;
- g) type, nature and amount of lubricant and any other organic additives used in the test, including the following:
  - 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) compacting pressures;
- i) result obtained including the pressure-density curve;
- j) comments about the test or test results.



**Key**

- 1 shrink ring
- 2 hard material
- 3 upper punch,  $l = H - 10$
- 4 lower punch,  $l = H + 35$

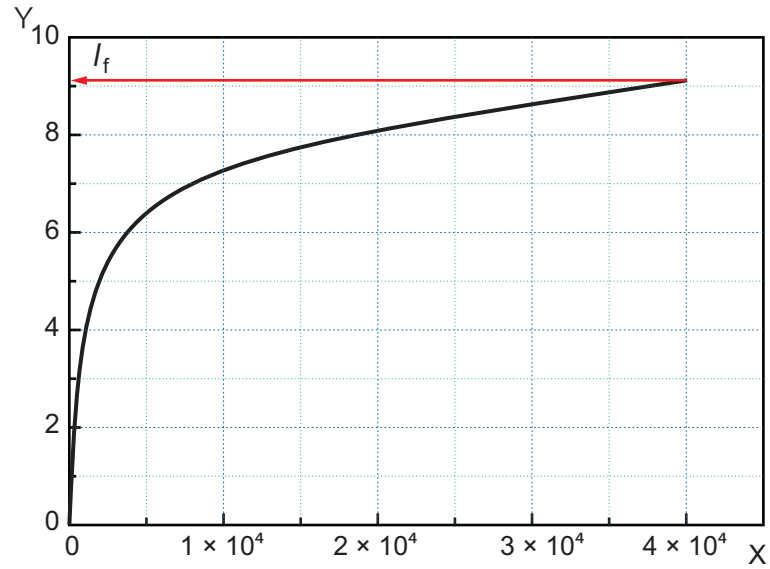
**Figure 1 — Example of cylindrical die and punches**



**Key**

- 1 powder
- 2 ejection cone
- 3 spacer

**Figure 2 — Diagram of compaction and ejection**

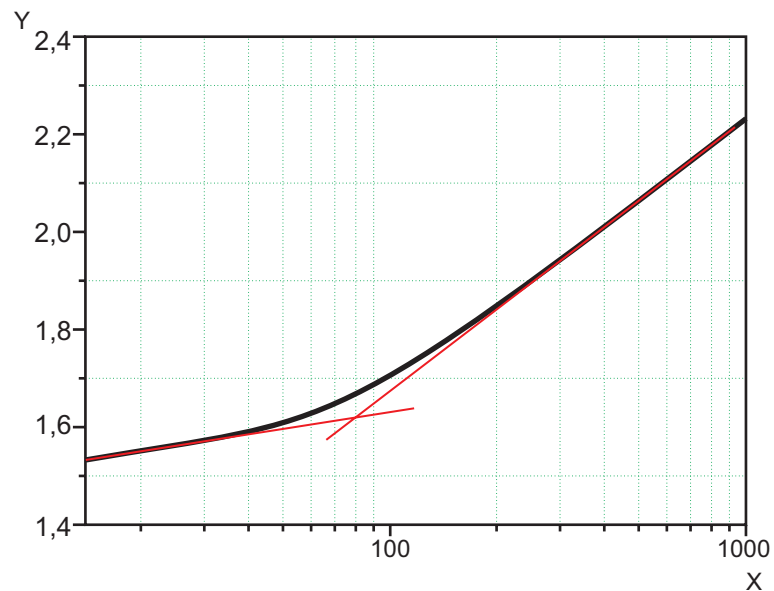


**Key**

- X load (N)
- Y displacement (mm)
- $l_f$  final displacement

NOTE Diameter of powder compact: 20  $\Phi$ ; mass of powder compact: 3 g; compressive rate: 1 mm/min.

**Figure 3 — Load-displacement curve of BaTiO3 granules**



**Key**

- X compressive pressure (MPa)
- Y density ( $\text{g}/\text{cm}^3$ )

NOTE Diameter of powder compact: 20 mm; mass of powder compact: 3 g; compressive rate: 1 mm/min.

**Figure 4 — Pressure-density curve of BaTiO3 granules**

## Bibliography

- [1] EN 725-10, *Advanced technical ceramics — Method of test for ceramic powders — Part 10: Determination of compaction properties*
- [2] JIS R1639-5, *Test methods of properties of fine ceramic granules — Part 5: Compressive strength of a single granule*
- [3] JIS R1639-6, *Test methods of properties of fine ceramic granules — Part 6: Compressive behavior of the granule beds*
- [4] KS L 1623, *Testing method for compressive strength of fine ceramic granules*









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