# INTERNATIONAL STANDARD

## ISO 14705

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# Fine ceramics (advanced ceramics, advanced technical ceramics) — Test method for hardness of monolithic ceramics at room temperature

*Céramiques techniques — Méthode d'essai de dureté des céramiques monolithiques à température ambiante*

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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).

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

This third edition cancels and replaces the second edition (ISO 14705:2008), which has been technically revised.

# Fine ceramics (advanced ceramics, advanced technical ceramics) — Test method for hardness of monolithic ceramics at room temperature

## 1   Scope

This document specifies a test method for determining the Vickers and Knoop hardness of monolithic fine ceramics at room temperature.

## 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 4545-1, *Metallic materials — Knoop hardness test — Part 1: Test method*

ISO 4545-2, *Metallic materials — Knoop hardness test — Part 2: Verification and calibration of testing machines*

ISO 4545-4, *Metallic materials — Knoop hardness test — Part 4: Table of hardness values*

ISO 6507-1, *Metallic materials — Vickers hardness test — Part 1: Test method*

ISO 6507-2, *Metallic materials — Vickers hardness test — Part 2: Verification and calibration of testing machines*

## 3   Terms and definitions

For the purposes of this document, the following terms and definitions apply.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

—   IEC Electropedia: available at http://www.electropedia.org/

—   ISO Online browsing platform: available at http://www.iso.org/obp

**3.1**
**Vickers hardness**
value obtained by dividing the applied force by the surface area of the indentation computed from the mean of the measured diagonals of the indentations, assuming that the indentation is an imprint of the undeformed indenter

Note 1 to entry: Vickers hardness may be expressed in two different units:

a)   with unit GPa, obtained by dividing the applied force in N by the surface area of the indentation in $mm^2$;

b)   Vickers hardness number, obtained by dividing the applied force in kgf by the surface area of the indentation in $mm^2$.

**3.2**
**Vickers indenter**
indenter in the shape of a right-angle pyramid with a square base and an angle between opposite faces of 136°

Note 1 to entry: See Table 1 and Figure 1.

**3.3**
**Knoop hardness**
value obtained by dividing the applied force by the projected area of the indentation computed from the measurement of the long diagonal of the indentation, assuming that the indentation is an imprint of the undeformed indenter

Note 1 to entry: The Knoop hardness may be expressed in two different units:

a)   with units of GPa, obtained by dividing the applied force in N by the projected area of the indentation in mm$^2$;

b)   Knoop hardness number, obtained by dividing the applied force in kgf by the projected area of the indentation in mm$^2$, without units specified.

**3.4**
**Knoop indenter**
indenter in the shape of a rhombic-based pyramid with the two angles between the opposite edges at 172,5° and 130°

Note 1 to entry: See Table 3 and Figure 6.

## 4   Vickers hardness

### 4.1   Principle

Forcing a diamond indenter in the form of a right-angle pyramid with a square base, and with a specified angle between opposite faces at the vertex into the surface of a test piece and measuring the length of the diagonals of the indentation left in the surface after removal of the test force, *F*. See Figure 1 and Figure 2.

### 4.2   Symbols, abbreviated terms and designations

**4.2.1**   See Table 1, Figure 1 and Figure 2.

**4.2.2**   The Vickers hardness is denoted by the symbol HV, preceded by the hardness value and followed by a number representing the test force (see Table 2).

Examples:

a)   Use of SI unit (GPa):

15,0 GPa HV 9,807 N represents a Vickers hardness of 15,0 GPa, determined with a test force of 9,807 N (1 kgf)

b)   Use of the Vickers hardness number (no units specified):

1 500 HV 1 represents a Vickers hardness number of 1 500, determined with a test force of 9,807 N (1 kgf).

**Table 1 — Symbols, abbreviated terms and designations for Vickers hardness testing**

| Symbol or abbreviated term | Designation |
|---|---|
| $\alpha$ | Angle between the opposite faces at the vertex of the pyramidal indenter (136° ± 0,5°) |
| $F$ | Test force, in newtons |
| $d$ | Arithmetic mean, in millimetres, of the two diagonals, $d_1$ and $d_2$ |
| HV | Vickers hardness<br><br>$$= \text{Constant} \times \frac{\text{Test force}}{\text{Surface area of indentation}}$$<br>a)  Units of GPa<br>$$= 0,001 \frac{2F \sin \frac{136°}{2}}{d^2} = 0,001\ 854 \frac{F}{d^2}$$<br>b)  Hardness number (no units specified)<br>$$= 0,102 \frac{2F \sin \frac{136°}{2}}{d^2} = 0,189\ 1 \frac{F}{d^2}$$ |
| $c$ | Arithmetic mean of the half of the two median crack lengths, $2c_1$ and $2c_2$ |
| SD | Standard deviation<br><br>$$= \sqrt{\frac{\sum \left(\overline{\text{HV}} - \text{HV}_n\right)^2}{n-1}}$$<br><br>where<br><br>$\overline{\text{HV}}$  is the arithmetic mean of the Vickers hardness $= \dfrac{\sum \text{HV}_n}{n}$;<br><br>$\text{HV}_n$  is the HV obtained from $n$th indentation;<br><br>$n$  is the number of indentations. |
| NOTE   Constant $= \dfrac{1}{g} = \dfrac{1}{9,807} = 0,102$, where $g$ is the acceleration due to gravity. ||

**Table 2 — Hardness symbols and the nominal values of test forces, $F$, for Vickers hardness testing**

| Hardness symbol | Test force, $F$ (nominal value) |
|---|---|
| HV 0,5 | 4,903 N |
| HV 1 | 9,807 N |
| HV 2 | 19,61 N |
| HV 3 | 29,42 N |
| HV 5 | 49,03 N |
| HV 10 | 98,07 N |
| HV 20 | 196,1 N |
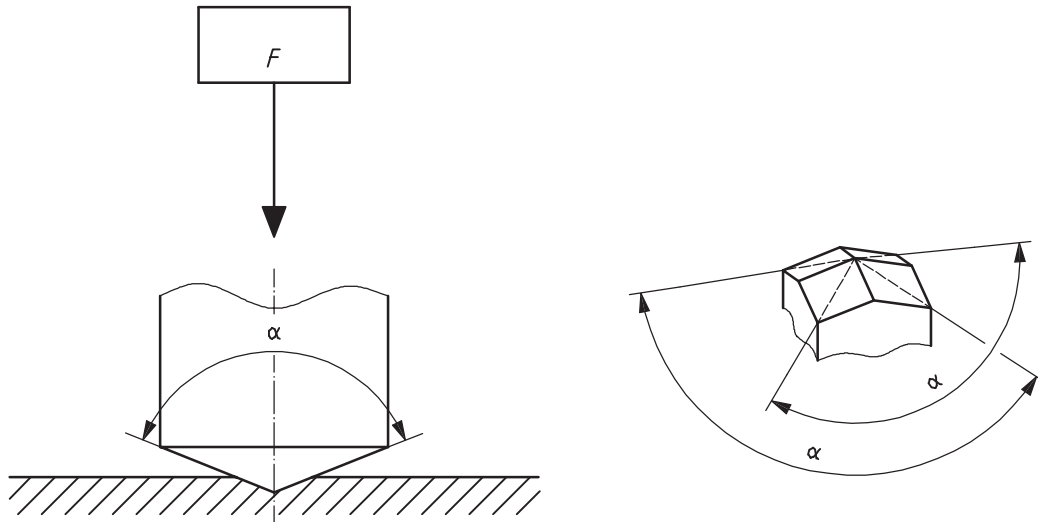
**Figure 1 — Vickers indenter (diamond pyramid)**
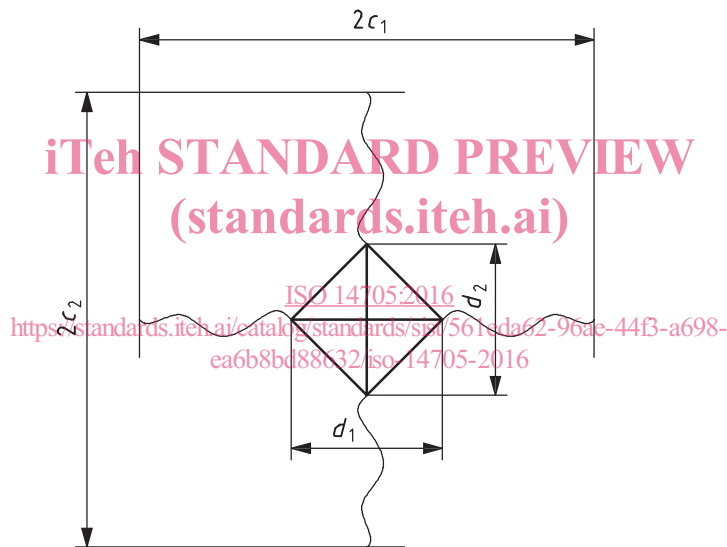


**Figure 2 — Vickers indentation**

## 4.3 Significance and use

Vickers indentation diagonal lengths are approximately 2,8 times shorter than the long diagonal of Knoop indentations, and the indentation depth is approximately 1,5 times deeper than Knoop indentations made at the same force. Vickers indentations are influenced less by the specimen surface flatness, parallelism of the diamond axis to the test piece surface normal, and surface finish than Knoop indentations, but these parameters should be considered nonetheless. Vickers indentations are much more likely to cause cracks in fine ceramics than Knoop indentations. Conversion between hardness scales shall not be made.

Vickers indentations on metallic materials are mainly formed by the plastic deformation. However, Vickers indentations on fine ceramics are formed by micro-cracking and micro-fracture, besides plastic deformation. This difference shall be noted for comparing the hardness of metals and ceramics.

## 4.4 Apparatus

**4.4.1 Testing machine**, capable of applying a predetermined test force in the range of 4,903 N (0,5 kgf) to 98,07 N (10 kgf), preferably 9,807 N (1 kgf), in accordance with ISO 6507-2. Verification of the test force shall be carried out in accordance with ISO 6507-2.

**4.4.2 Diamond indenter**, in the shape of a right-angle pyramid with a square base, as specified in ISO 6507-1 and ISO 6507-2. Verification of the indenter shall be carried out in accordance with ISO 6507-2.

**4.4.3 Measuring device**, capable of measuring the indentation diagonals with a readout resolution of ±0,2 μm or finer. A numerical aperture (NA) between 0,60 and 0,95 for the objective lens for the microscope is recommended. Verification of the measuring device shall be carried out in accordance with ISO 6507-2.

NOTE      Indirect verification can be carried out by means of standardized blocks calibrated in accordance with ISO 6507-3, following ISO 6507-2, or other approved and traceable ceramic standard reference blocks.

## 4.5 Test pieces

**4.5.1** The test shall be carried out on a surface which is smooth, flat and free from foreign matter. The test piece shall be polished to permit accurate measurement of the diagonal lengths of the indentation. Preparation shall be carried out in such a way that any alteration of the surface hardness is minimized. Surfaces shall not be thermally or chemically etched. If applicable, residual surface stresses shall be removed by suitable polishing or annealing procedures.

**4.5.2** The thickness of the test piece shall be at least 0,5 mm. It shall be at least 1,5 times the diagonal of the indentation, $d$, and at least 2 times the crack length, $c$, whichever is greater. No indentation damage shall be visible at the back of the test piece upon completion of the test.

## 4.6 Procedure

**4.6.1** In general, the test shall be carried out at room temperature within the limits of 10 °C to 35 °C. Tests carried out under controlled conditions shall be made at a temperature of 23 °C ± 5 °C.

**4.6.2** The test force shall be 9,807 N (1 kgf). In cases where significant chipping or lateral crack-spalling occurs or where the impression is too faint, the test forces within the range 4,903 N (0,5 kgf) to 196,1 N (20 kgf), listed in Table 2, may be used. Other instances where a heavier load may be required are where the grain structure is very coarse and the indentation area at lower loads may contact only a few grains of the material (e.g. a multiphase material).

**4.6.3** The following items shall be confirmed before the test.

a)   Check the zero of the measuring system.

b)   Check the measuring system using a calibrated scale or certified indentation in a test block.

c)   Check the operation of the loading system by performing a test on a certified test block.

d)   Check the condition of the indenter by examining the indentation made in the test block. Replace the indenter, if necessary, by taking into account the conditions given in 4.6.10.

e)   A test block with high hardness has to be used in order to obtain impressions in the same size range as expected during tests on ceramics.

**4.6.4** The indenter shall be cleaned prior to and during the test series, as ceramic powders or fragments from the ceramic test piece can adhere to the diamond indenter.

**4.6.5**  The test piece shall be placed on a rigid support. The support surface shall be clean and free from foreign matter. It is important that the test piece lies firmly on the support, so that displacement cannot occur during the test.

**4.6.6**  Carefully adjust the illumination and focusing conditions, in order to obtain the optimum view and clarity of the indentation. Both indentation tips shall be in focus at the same time. Do not change the focus when measuring the distance from tip to tip.

**4.6.7**  Bring the indenter into contact with the test surface and apply the test force in a direction perpendicular to the surface, without shock or vibration, until the applied force attains the specified value. The time from the initial application of the force until the full test force is reached shall not be less than 1 s nor greater than 5 s. The duration of application of the constant maximum test force shall be 15 s.

**4.6.8**  Throughout the test, the apparatus shall be protected from shock or vibration.

**4.6.9**  The distance between the centre of any indentations and the edge of the test piece shall be at least 2,5 times the mean diagonal of the indentation, and at least 5 times the mean length of the crack, as shown in Figure 3. The distance between the centres of two adjacent indentations shall be at least 4 times the mean diagonal of the indentation, and at least 5 times the mean length of the crack, as shown in Figure 3. If two adjacent indentations differ in size and crack length, the spacing shall be based on the mean diagonal of the larger indentation and the longer crack length.
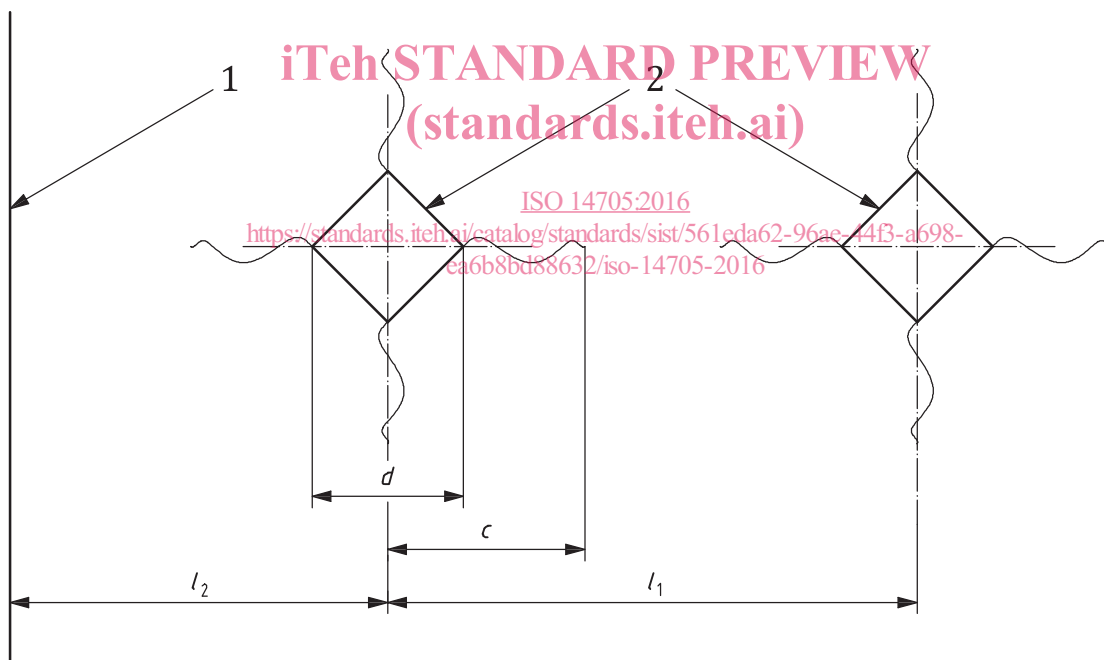


**Key**

1   edge of test piece

2   indentations

$c$   length from the centre of indentation to the end of crack

$d$   length of indent diagonal

$l_1$   distance between centres of indentations

  $l_1 \geq 4d$ and $5c$

$l_2$   distance from centre of indentation to the edge of sample

  $l_2 \geq 2,5d$ and $5c$

**Figure 3 — Closest permitted spacing between indentations and from indentation to the test piece edge for Vickers indentations**

**4.6.10**  The satisfactory condition of the indenter shall be verified frequently. Any irregularities in the shape of the indentation may indicate chipping, cracking or other deterioration of the indenter. If the examination of the indenter confirms this, then the test shall be rejected and the indenter replaced.

**4.6.11**  If there is excessive cracking from the indentation tips and sides, then the indentation shall be rejected and go unmeasured. If one of the tips of an indentation falls into a pore, the indentation shall be rejected. If the indentation lies in or on a large pore, the indentation shall be rejected. Figure 4 provides guidance on this assessment.

**4.6.12**  Measure the length of the two diagonals to within 0,2 μm for diagonals less than 50 μm, or to within 0,5 μm for diagonals equal to or more than 50 μm. The arithmetical mean of two readings shall be taken for the calculation of the Vickers hardness. If the difference of the two diagonals is more than 5 % of the mean value (see Figure 4), the result shall be rejected, and a check made of the parallelism and flatness of the test piece, and of the alignment of the indenter. Follow the manufacturer's instructions very carefully, with regards to the proper usage of the measuring crosshairs. Figure 5 is provided for guidance. The use of optical methods to enhance contrast (like Nomarski interferences) is not permitted.

**4.6.13**  At least five valid indentations shall be made for obtaining a mean result in accordance with this document.

**4.6.14**  Calculate the Vickers hardness, HV, for each valid indentation, using the formula in Table 1. Calculate the mean hardness for all valid indentations and the standard deviation. The calculated Vickers hardness shall be expressed with three significant numbers (e.g. 15,4 GPa HV 9,807 N or 1540 HV 1)

**4.6.15**  Alternatively, see ISO 6507-4 for conversion tables for use in tests made on flat surfaces.

## 4.7  Accuracy and uncertainties

The principal errors arising in a Vickers hardness test on advanced monolithic technical ceramics vary in magnitude according to the size of the indentation, and thus the indentation force used. The Vickers diamond geometry was originally chosen because natural cleavage planes of the diamond were employed. Variations in geometry between indenters are therefore small, and can usually be ignored except when indentations are of less than 20 μm diagonal length where the tip and edges near the tip may be variable between indenters. In particular, the edges may have flats up to 1 μm across on them, and this has the effect of cutting the corners off the indentation. The error that this introduces is insignificant if the indentation is larger than about 30 μm, but increases rapidly in importance as the size is reduced.

Determination of the diagonal lengths using cross-wires or other device attached to the instrument relies on the operator positioning them at the "true" opposing corners of the indentation. There is a subjective element in performing this task which increases with poor optical contrast and reducing size of the indentation. The possible errors can be reduced by experience, and by consistent use of high-hardness, preferably ceramic or hardmetal, test blocks to familiarize the eye at the start of measurement sessions. In this way, any systematic measurement bias can be reduced. In a round-robin exercise on high-alumina ceramics[4], it was found that when two individuals measure the same set of indentations on different measurement equipment, a poor correlation was obtained unless the true sizes of the indentations varied by more than ±1 μm. It follows that, discounting differences between machines, it cannot be guaranteed that any two observers will agree that one material is significantly harder than another unless the average indentation sizes are systematically smaller by more than 1 μm. Thus, even if it is possible to measure the indentation diagonal length to an apparent precision of 0,1 μm, or the optical resolution limit if larger, the ability to discriminate between materials is limited to an order of magnitude greater in size. Errors of this size assume significance when the indentation size is less than about 20 μm. In addition, there is the actual scatter in indentation sizes as a result of local microstructure variations such as grain size, grain orientation, secondary phase content, micro-cracking, porosity, etc. In a very uniform and homogeneous, hard, fine-grained material, the scatter in actual indentation sizes may be less than the potential measurement errors, and thus not be discernible. In a less-homogenous material, the true indentation size may vary significantly. In such a case, the