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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

Reference number
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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

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

ISO 14705 was prepared by Technical Committee ISO/TC 206, *Fine ceramics*.

This second edition cancels and replaces the first edition (ISO 14705:2000), 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 International Standard specifies a test method for determining the Vickers and Knoop hardness of monolithic fine ceramics at room temperature.

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.

ISO 4545-1:2005, *Metallic materials — Knoop hardness test — Part 1: Test method*

ISO 4545-2:2005, *Metallic materials — Knoop hardness test — Part 2: Verification and calibration of testing machines*

ISO 4545-3:2005, *Metallic materials — Knoop hardness test — Part 3: Calibration of reference blocks*

ISO 4545-4:2005, *Metallic materials — Knoop hardness test — Part 4: Table of hardness values*

ISO 6507-1:2005, *Metallic materials — Vickers hardness test — Part 1: Test method*

ISO 6507-2:2005, *Metallic materials — Vickers hardness test — Part 2: Verification and calibration of testing machines*

ISO 6507-3:2005, *Metallic materials — Vickers hardness test — Part 3: Calibration of reference blocks*

ISO 6507-4:2005, *Metallic materials — Vickers hardness test — Part 4: Tables of hardness values*

3 Terms and definitions

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

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 Vickers hardness may be expressed in two different units:

a) with units of GPa, obtained by dividing the applied force, in kN, 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, without the units specified.

NOTE 2 Use of Vickers hardness with units of GPa is preferred.

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°

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 Knoop hardness may be expressed in two different units:

a) with units of GPa, obtained by dividing the applied force, in kN, 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.

NOTE 2 The use of Knoop hardness with units of GPa is preferred.

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°

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 Figures 1 and 2.

4.2 Symbols, abbreviations and designations

4.2.1 See Table 1 and Figures 1 and 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 the 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, abbreviations and designations for Vickers hardness testing

Symbol or abbreviation	Designation
α	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 $= \text{Constant} \times \dfrac{\text{Test force}}{\text{Surface area of indentation}}$ (1) units of GPa (preferred) $= 0{,}001 \dfrac{2F \sin \dfrac{136°}{2}}{d^2} = 0{,}001\,854 \dfrac{F}{d^2}$ (2) hardness number (no units specified) $= 0{,}102 \dfrac{2F \sin \dfrac{136°}{2}}{d^2} = 0{,}189\,1 \dfrac{F}{d^2}$
c	Arithmetic mean of the half of the two median crack lengths $2c_1$ and $2c_2$
S.D.	Standard deviation $= \sqrt{\dfrac{\sum \left(\overline{\text{HV}} - \text{HV}_n\right)^2}{n-1}}$ where $\overline{\text{HV}}$ is the arithmetic mean of the Vickers hardness $= \dfrac{\sum \text{HV}_n}{n}$ HV_n is the HV obtained from *n*th indentation 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 4,903 N or HV 0,5	4,903 N
HV 9,807 N or HV 1	9,807 N
HV 19,61 N or HV 2	19,61 N
HV 29,42 N or HV 3	29,42 N
HV 49,03 N or HV 5	49,03 N
HV 98,07 N or HV 10	98,07 N
HV 196,1 N or HV 20	196,1 N

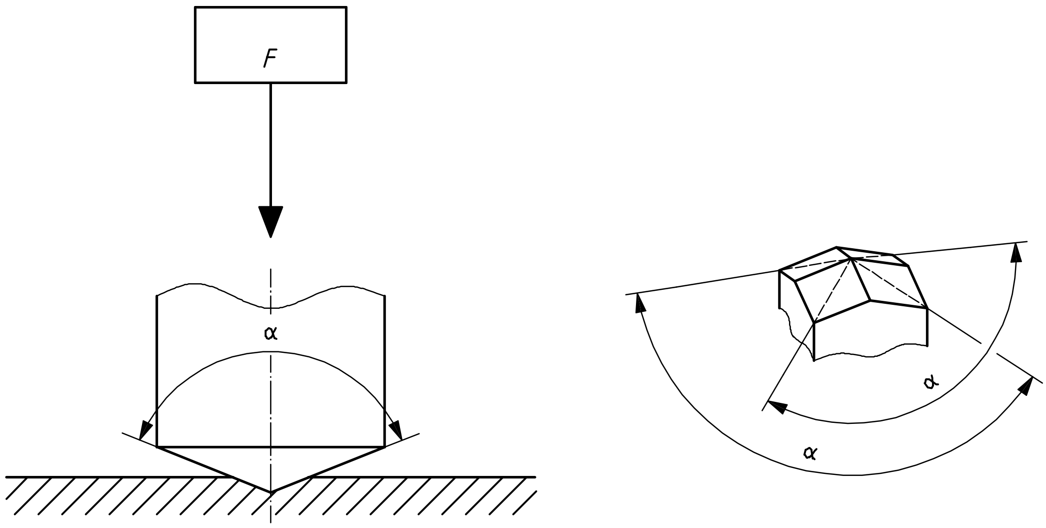


Figure 1 — Vickers indenter (diamond pyramid)

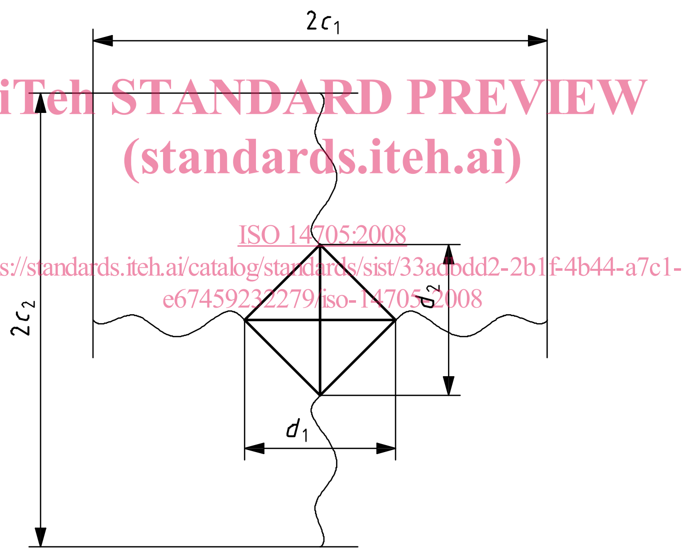


Figure 2 — Vickers indentation

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 are 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 hardnesses 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) of 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.

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 on 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 recommended test force is 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 examination of the indentation made in the test block. Replace the indenter, if necessary, by taking into account the conditions given in 4.6.10.

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.

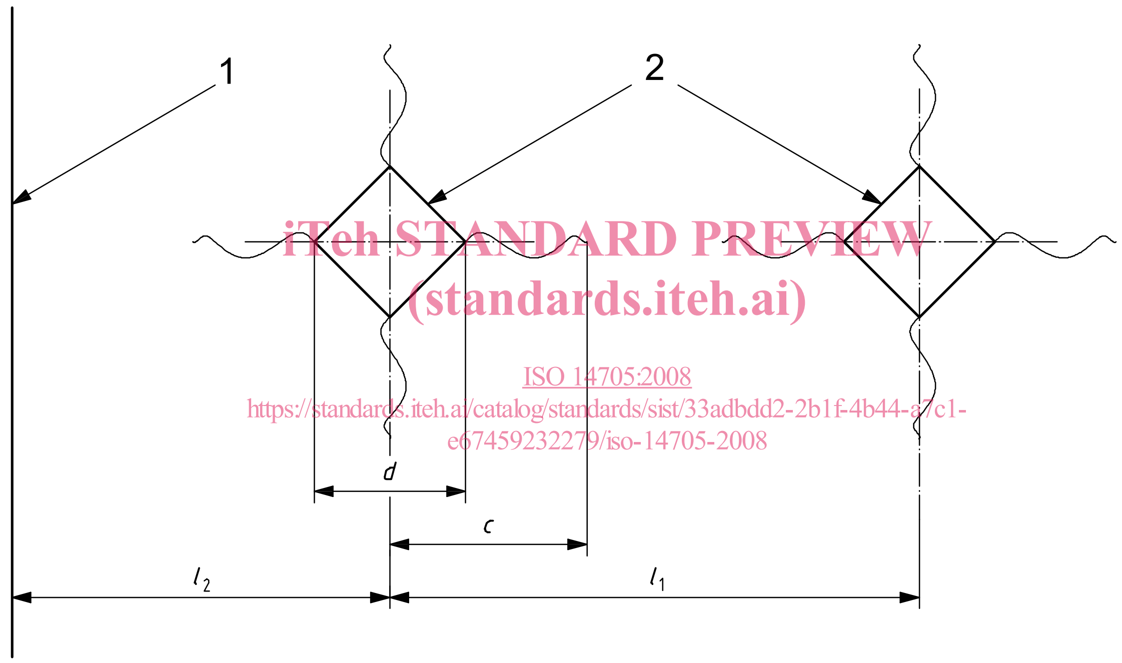
4.6.5 The test piece shall be placed on a rigid support. The support surface shall be clean and free from foreign matter. It is important that the test piece lies firmly on the support, so that displacement cannot occur during the test.

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

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

l_2 distance from centre of indentation to the edge of sample

$l_2 \geqslant 2{,}5d$ 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 (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, as regards the proper usage of the measuring crosshairs. Figure 5 is provided for guidance.

4.6.13 At least 5 valid indentations shall be made for obtaining a mean result in accordance with this International Standard.

4.6.14 Calculate the Vickers hardness, HV, for each valid indentation, using the equation in Table 1. Calculate the mean hardness for all valid indentations, and the standard deviation.

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

4.7 Test report

The test report shall include the following information:

a) a reference to this International Standard, i.e. ISO 14705:2008;

b) information on the test piece;

c) test conditions, i.e.:

1) thickness of test piece;

2) test force;

3) surface condition (polishing method);

4) test temperature, or certification that the test was done at “room temperature”;

5) the number of valid indentations and the total number of indentations made to obtain these valid indentations;

6) magnification of microscope;

d) the result obtained:

1) individual valid Vickers hardness values;

2) arithmetic mean of the Vickers hardness;

3) standard deviation (S.D.) of the Vickers hardness;

e) all operations not specified by this International Standard or regarded as optional;

f) details of any circumstances (such as extensive cracking or chipping, porosity, multiphase nature of the material, coarse grain size, etc.) which may have affected the result.