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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

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International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 3.

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 International Standard may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

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

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

1 Scope

This International Standard describes the method of test for determining the Vickers and Knoop hardness of monolithic fine ceramics at room temperature.

2 Normative references

The following normative documents contain provisions which, through reference in this text, constitute provisions of this International Standard. For dated references, subsequent amendments to, or revisions of, any of these publications do not apply. However, parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the normative documents indicated below. For undated references, the latest edition of the normative document referred to applies. Members of ISO and IEC maintain registers of currently valid International Standards.

ISO 4546:1993, *Metallic materials — Hardness test — Verification of Knoop hardness testing machines.*

ISO 6507-1:1997, *Metallic materials — Vickers hardness test — Part 1: Test method.*

ISO 6507-2:1997, *Metallic materials — Vickers hardness test — Part 2: Verification of testing machines.*

ISO 10250:1994, *Metallic materials — Hardness test — Tables of Knoop hardness values for use in tests made on flat surfaces.*

3 Terms and definitions

For the purposes of this International Standard, 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

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 units specified.

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

3.2
Vickers indenter
indenter in the shape of 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

The 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 Use of the 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 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)

1500 HV 1 represents a Vickers hardness number of 1 500, determined with a test force of 9,807 N (1 kgf).

Table 1 — Symbols and designations for Vickers hardness testing

Symbol	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 millimeters, 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 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 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	2 9,42 N
HV 49,03 N or HV 5	4 9,03 N
HV 98,07 N or HV 10	98,07 N

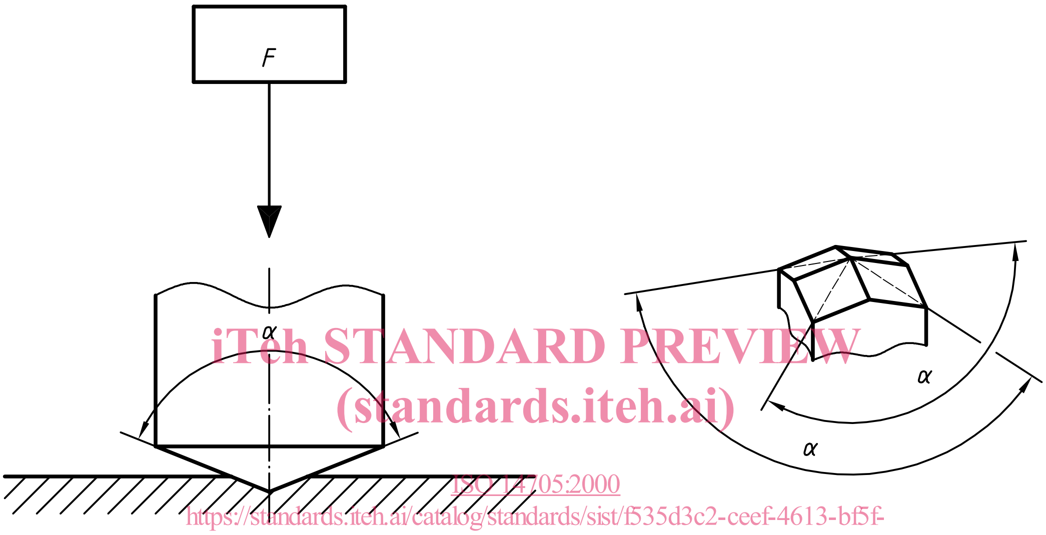


Figure 1 — Vickers indenter (diamond pyramid)

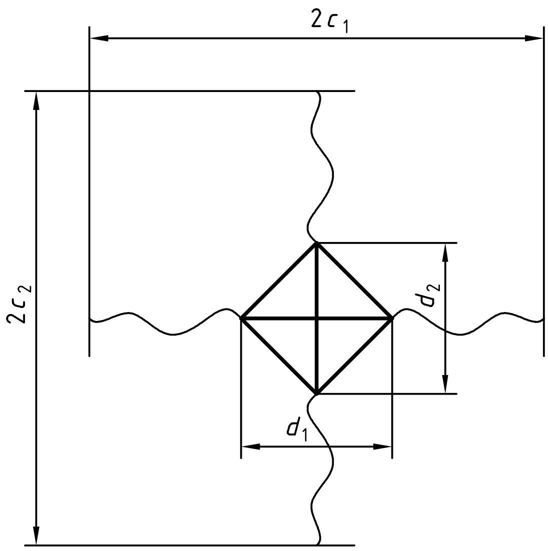


Figure 2 — Vickers indentation

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 load. Vickers indentations are influenced less by 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 6507-2. 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 0,65 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 may 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. 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 98,07 N (10 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.

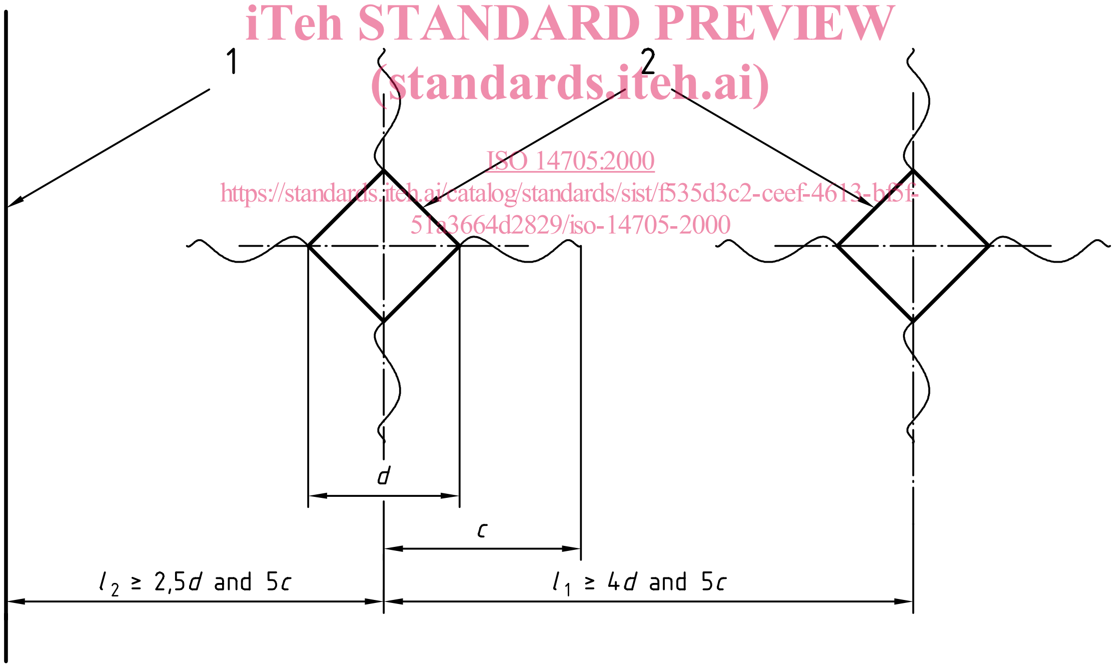
4.6.5 The test piece shall be placed on a rigid support. The support surface shall be clean and free from foreign matter. It is important that the test piece lie firmly on the support so that displacement cannot occur during the test.

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 approach velocity of the indenter shall not affect the hardness value. The time from the initial application of the force until the full test force is reached shall not be less than 2 s nor greater than 8 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.



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

d is the length of indent diagonal

l_1 is the distance between centres of indentations

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

Key

1 Edge of test piece

2 Indentations

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 in 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 6507-1, for conversion tables for use in tests made on flat surfaces.

4.7 Test report

The test report shall include the following information:

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

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) arithmetic mean of the Vickers hardness

2) 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.