INTERNATIONAL STANDARD

ISO 14705

Third edition
2016-12-15

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
ISO 14705:2016(E)

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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).

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. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

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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 World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT) see the following URL: www.iso.org/iso/foreword.html.

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
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 $= \text{Constant} \times \frac{\text{Test force}}{\text{Surface area of indentation}}$ a) Units of GPa $= 0{,}001 \frac{2F \sin \frac{136^\circ}{2}}{d^2} = 0{,}001\ 854 \frac{F}{d^2}$ b) Hardness number (no units specified) $= 0{,}102 \frac{2F \sin \frac{136^\circ}{2}}{d^2} = 0{,}189\,1 \frac{F}{d^2}$
c	Arithmetic mean of the half of the two median crack lengths, $2c_1$ and $2c_2$
SD	Standard deviation $= \sqrt{\frac{\sum \left(\overline{\text{HV}} - \text{HV}_n \right)^2}{n-1}}$ where $\overline{\text{HV}}$ is the arithmetic mean of the Vickers hardness $= \frac{\sum \text{HV}_n}{n}$; HV_n is the HV obtained from nth indentation; n is the number of indentations.
NOTE Constant $= \frac{1}{g} = \frac{1}{9{,}807} = 0{,}102$, 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

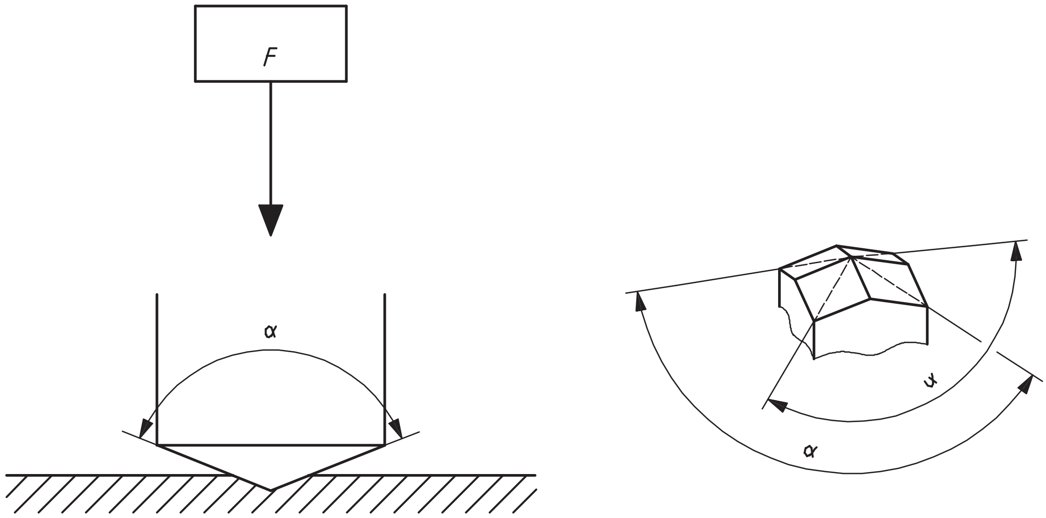


Figure 1 — Vickers indenter (diamond pyramid)

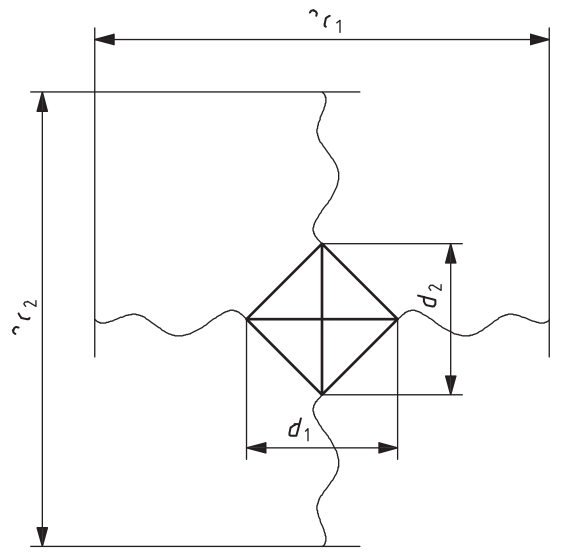


Figure 2 — Vickers indentation

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.

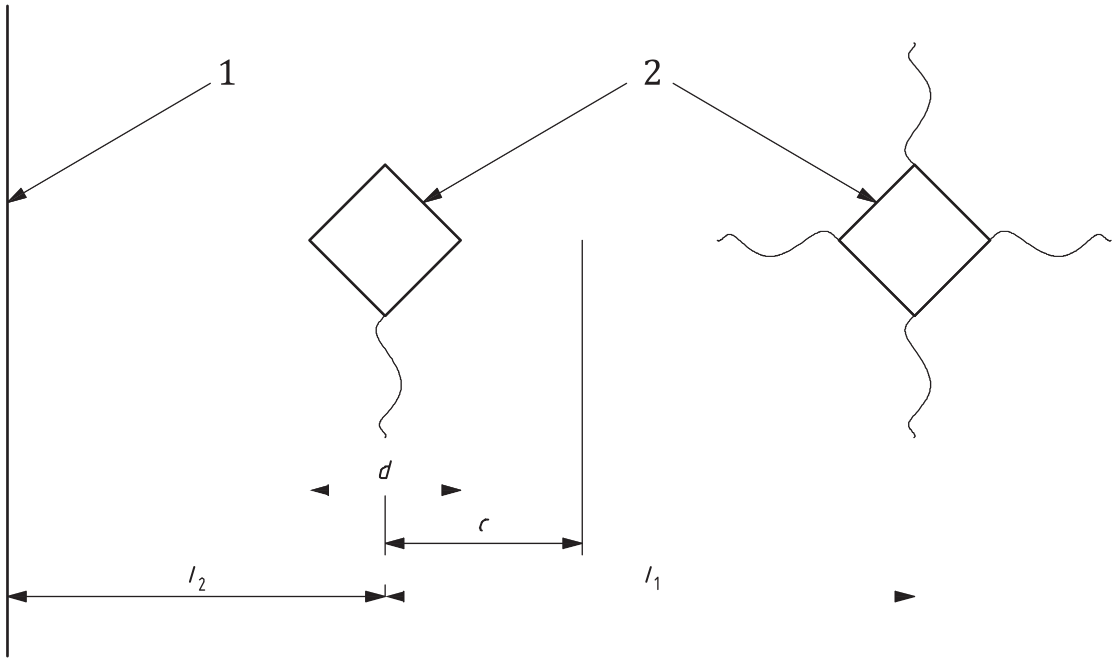
4.6.5 The test piece shall be placed on a rigid support. The support surface shall be clean and free from foreign matter. It is important that the test piece lies firmly on the support, so that displacement cannot occur during the test.

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 mean result may be determined by the choice of measurement position, deliberate or inadvertent. The certainty of mean result can be improved only by increasing the number of indentations, but the possibility of a human bias remains. The discrimination between inhomogeneous materials is poorer than for homogeneous ones. The use of the scanning electron microscope is not recommended for a number of reasons. The principal ones are that the topographic contrast produced by an indentation is not great, that the edges and corners are not always clearly defined, and that the actual magnification of the image requires careful calibration and checking for distortion in both directions.

In summary, the systematic and material inhomogeneity errors may be minimized by employing the highest possible measurement force consistent with no chipping or displacement of corners of the indentation. Under such conditions, the discrimination between materials is greatest. Tests at HV 1,0 represent an optimum force in terms of the range of materials which can give acceptable indentations. At greater forces, problems with quality of indentations can make measurement impossible for many materials, even though errors may be proportionately smaller. Even so, the possible errors contribute typically ±0,7 GPa in hardness (±70 in hardness number) as a confidence level. Microhardness tests are subject to much larger overall errors, typically ±2,0 GPa in hardness (±200 in hardness number) (10 % to 15 %) can be expected at HV 0,2, and greater at lower forces, and should not be used for any test required for a specification.

4.8 Test report

The test report shall include the following information:

a) a reference to this document, i.e. ISO 14705:2016;

b) an information on the test piece;

c) the test conditions, i.e.:

   1) thickness of test piece;

   2) test force, if different from 9,807 N (1 kgf), substatiate;

   3) surface condition (polishing method; describe other treatments);

   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:

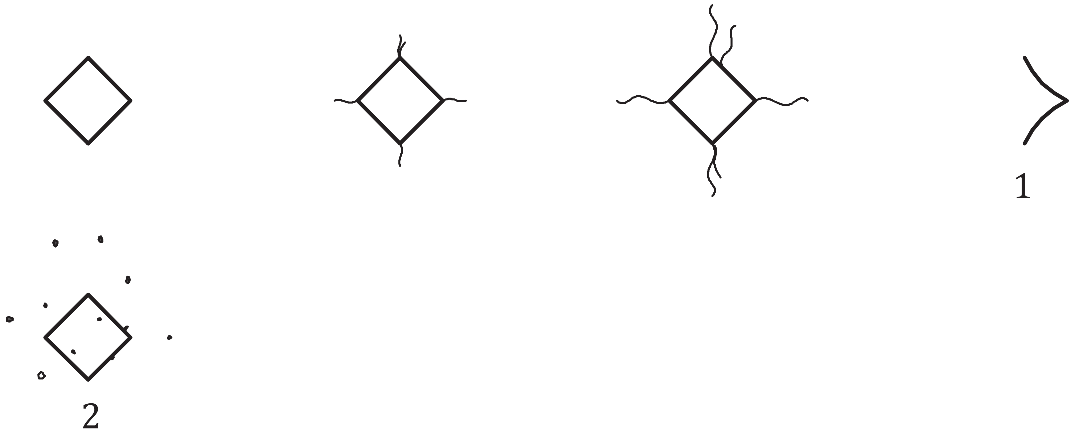
   1) individual valid Vickers hardness values;

   2) arithmetic mean of the Vickers hardness;

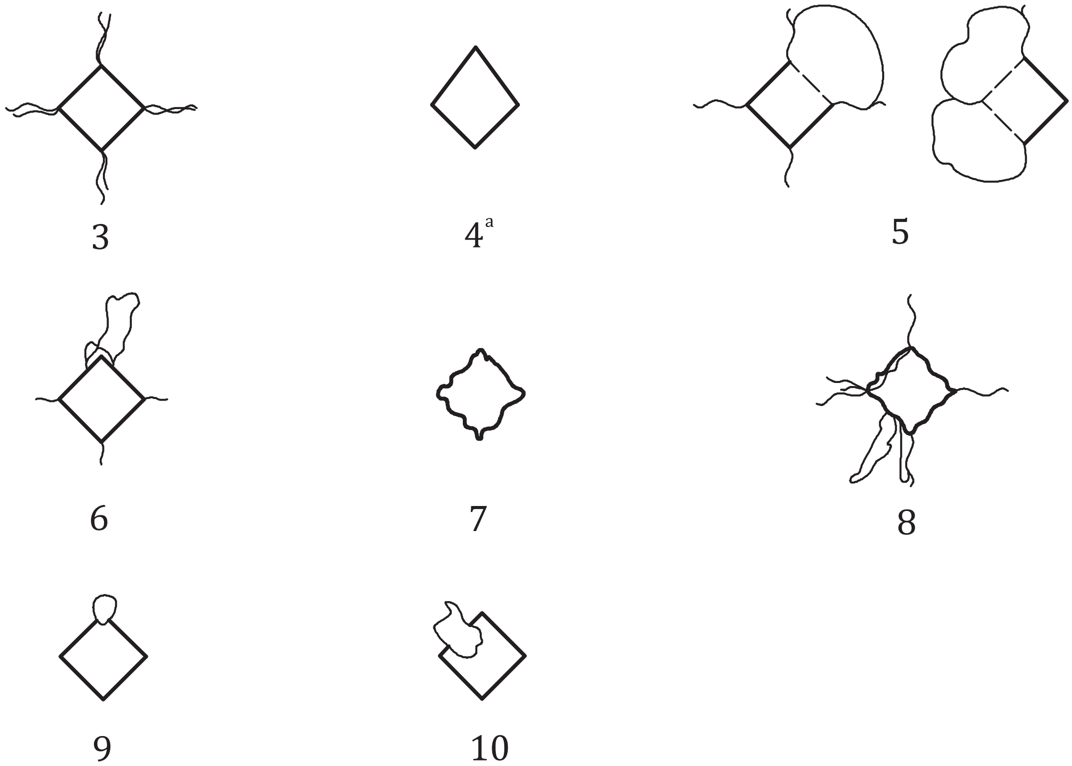
   3) standard deviation (SD) of the Vickers hardness;

e) all operations not specified by this document 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.



a) Acceptable indentations



b) Unacceptable indentations

Key

1 bowed edges

2 porosity

3 large tip cracks

4 asymmetrical

5 spalled edges

6 tip region displaced

7 chipping and ragged edges

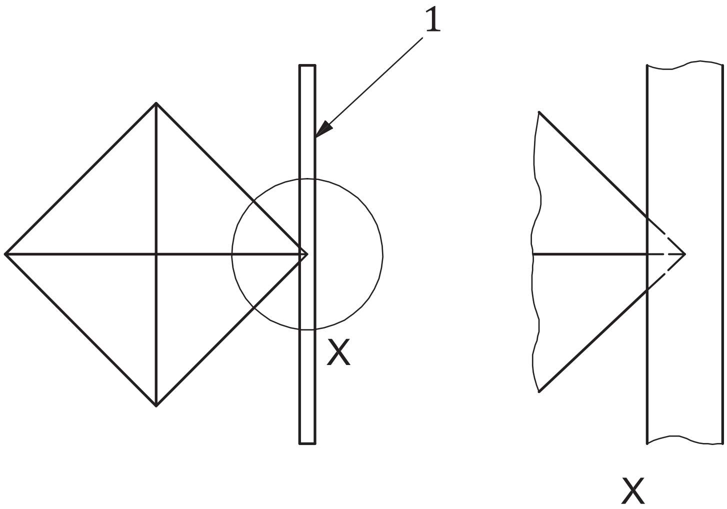
8 ragged edges (grain displacement, pullouts)

9 pore at tip

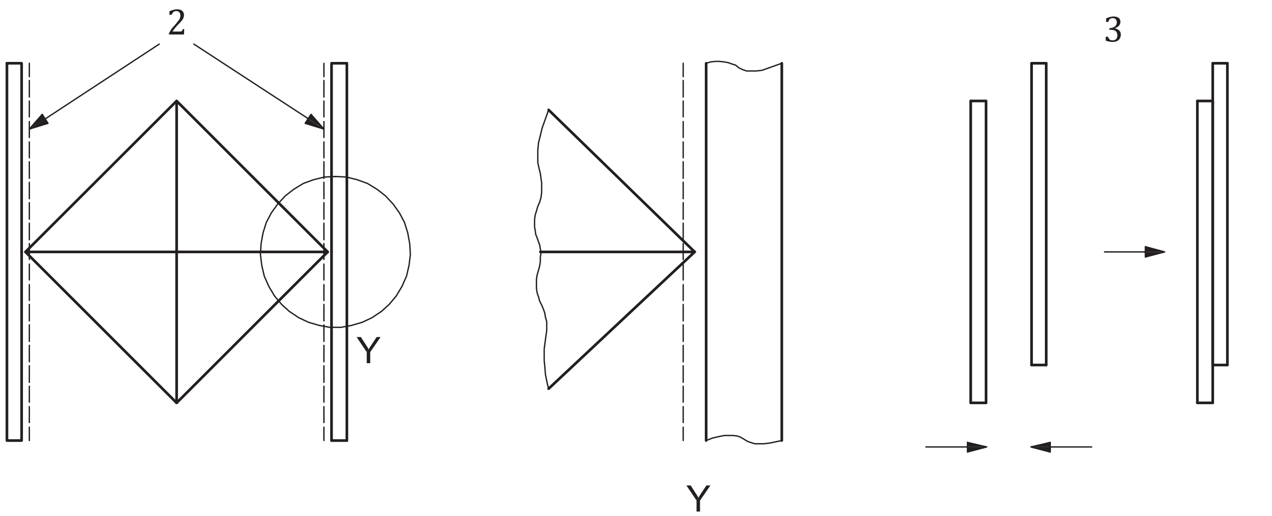
10 indentation on a large pore

a Permissible degree of asymmetry, as defined in 4.6.12.

Figure 4 — Guidelines for the acceptability of Vickers indentations

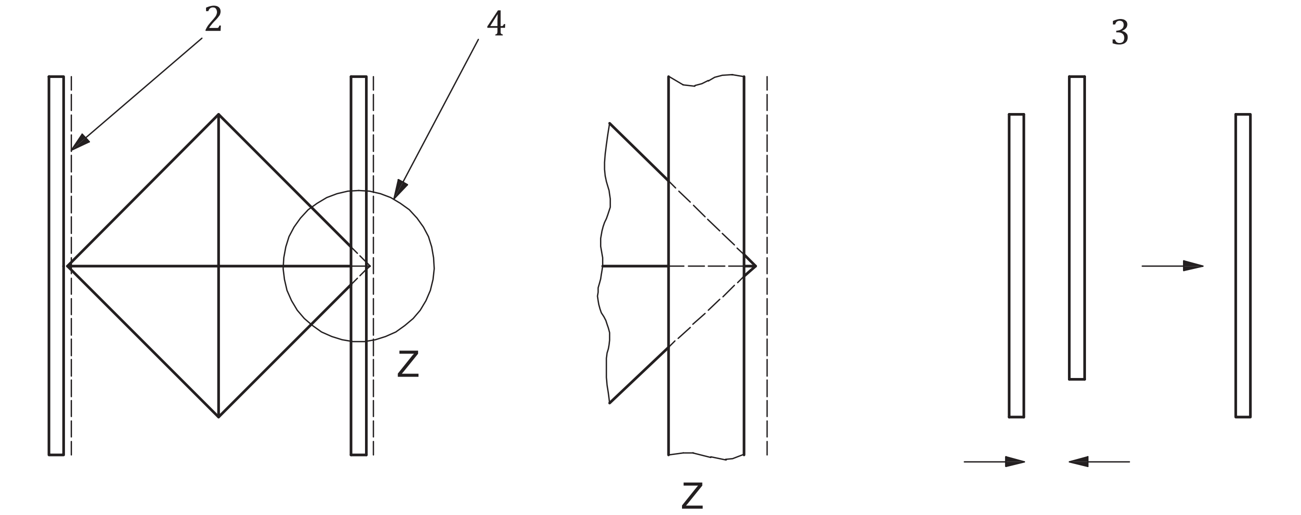


a) Incorrect, crosshair completely covers the tip.



b) Correct, **double crosshair measurement system.** The indentation is intended to be measured between two crosshairs or measuring lines. Indentation tips should be on the inside edge (in the fringe) of each crosshair. The measuring system is zeroed by bringing together the adjacent edges of the crosshairs, as shown on the right.

Figure 5 *(continued)*



c) Correct, single crosshair and some double crosshair measurement systems. The indentation tip is on the same side of the crosshair line(s). For a single crosshair system, the measuring system is zeroed when the crosshair is correctly positioned on one indentation tip, or with both lines superimposed in a double crosshair system, as shown on the right.

Key

1 crosshair

2 light fringe (size is exaggerated)

3 setting to zero

4 tip in the fringe

Figure 5 — Crosshair measurement systems for Vickers indentation

5 Knoop hardness

5.1 Principle

Forcing a diamond indenter, in the form of a rhombic-based pyramid with specified angles between opposite ridges at the vertex, into the surface of a test piece and measuring the length of the long diagonal of the indentation left in the surface after removal of the test force, *F*. See Figure 6 and Figure 7.

5.2 Symbols and designations

5.2.1 See Table 3, Figure 6 and Figure 7.

5.2.2 Knoop hardness is denoted by the symbol HK, preceded by the hardness value and supplemented by a number representing the test force (see Table 4).

Examples:

a) Use of SI unit (GPa):

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

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

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

Table 3 — Symbols, abbreviated terms and designations for Knoop hardness testing

Symbol or abbreviated term	Designation
F	Test force, in newtons
d	Length, in millimetres, of the long diagonal
k	Indenter constant, relating projected area of the indentation to the square of the length of the long diagonal
HK	Knoop hardness $= \text{Constant} \times \frac{\text{Test force}}{\text{Surface area of indentation}}$ a) units of GPa $= 0{,}001 \frac{F}{d^2 \cdot k} = 0{,}001 \frac{F}{0{,}070\,28 \cdot d^2} = 0{,}014\,23 \frac{F}{d^2}$ b) hardness number (no units specified) $= 0{,}102 \frac{F}{d^2 \cdot k} = 0{,}102 \frac{F}{0{,}070\,28 \cdot d^2} = 1{,}451 \frac{F}{d^2}$
SD	Standard deviation $= \sqrt{\frac{\sum \left(\overline{\text{HK}} - \text{HK}_n \right)^2}{n-1}}$ where $\overline{\text{HK}}$ is the arithmetic mean of the Knoop hardness $= \frac{\sum \text{HK}_n}{n}$; HK_n is the HK obtained from nth indentation; n is the number of indentations.

NOTE Constant $= \frac{1}{g} = \frac{1}{9{,}807} = 0{,}102$, where g is the acceleration due to gravity.

Indenter constant $k = \frac{\frac{\tan \frac{\beta}{2}}{2}}{2 \tan \frac{\alpha}{2}}$

where α and β are the angles between the opposite edges (see Figure 6).

The values of α and β specified in ISO 4545-2 are α = 172,5° ± 0,1° and β = 130° ± 0,1°.

Table 4 — Hardness symbols and nominal values of test force, *F*, for Knoop hardness testing

Hardness symbol	Test force, *F* (nominal value)
HK 0,1	0,980 7 N
HK 0,2	1,961 4 N
HK 0,3	2,942 1 N
HK 0,5	4,903 N
HK 1	9,807 N
HK 2	19,61 N
HK 5	49,03 N

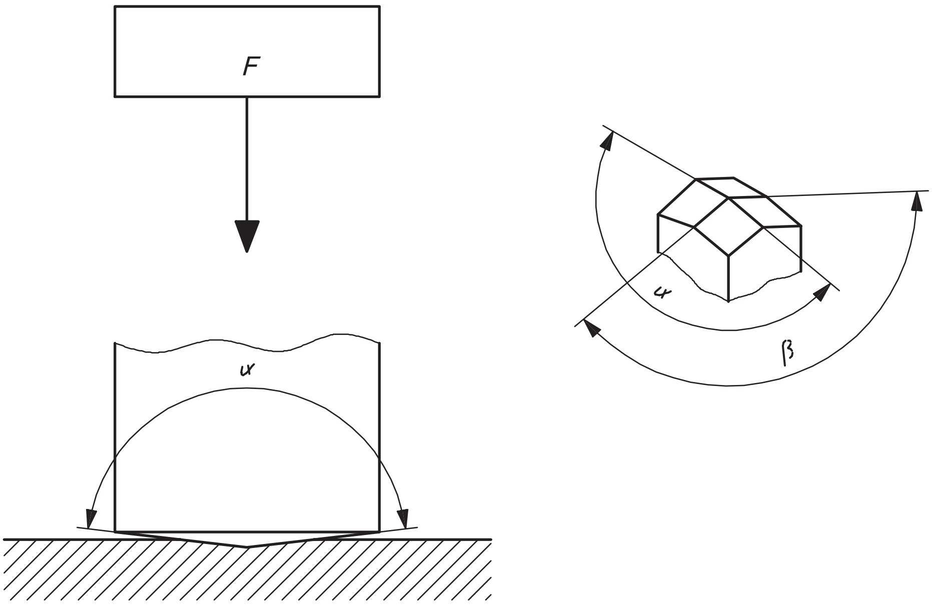


Figure 6 — Knoop indenter

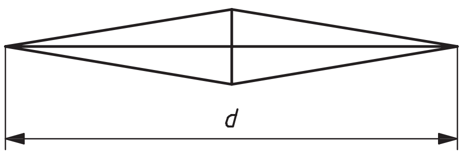


Figure 7 — Knoop indentation

5.3 Significance and use

For fine ceramics, the Knoop long diagonal length is approximately 2,8 times longer than the Vickers diagonal for the same load, and cracking is much less of a problem. On the other hand, the tip of a Knoop indentation is more difficult to discern precisely. A Knoop indentation is shallower than a Vickers indentation made at the same load. Conversion between hardness scales shall not be made.

Knoop indentations on metallic materials are mainly formed by plastic deformation. However, Knoop 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.

5.4 Apparatus

5.4.1 Testing machine, capable of applying a predetermined test force in the range of 0,980 7 N (0,1 kgf) to 49,03 N (5 kgf), preferably 9,807 N (1 kgf) or 19,61 N (2 kgf), in accordance with ISO 4545-2. Verification of the test force shall be carried out in accordance with ISO 4545-2.

5.4.2 Diamond indenter, in the shape of a rhombic-based pyramid, as specified in ISO 4545-1 and ISO 4545-2. Verification of the indenter shall be carried out in accordance with ISO 4545-2.

5.4.3 Measuring device, capable of measuring the indentation diagonal with a read-out resolution of 0,2 μm or finer. A numerical aperture (NA) of between 0,60 and 0,95 for the objective lens of the microscope is recommended. Verification of the measuring device shall be carried out in accordance with ISO 4545-2.

NOTE Indirect verification can be carried out by means of standardized blocks calibrated in accordance with ISO 4545-3, following ISO 4545-2, or other approved ceramic standard reference blocks.

5.5 Test pieces

5.5.1 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.

5.5.2 The thickness of the test piece shall be at least 0,5 mm. No indenting damage shall be visible at the back of the test piece upon completion of the test.

5.6 Procedure

5.6.1 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.

5.6.2 The test forces shall be 9,807 N (1 kgf) and 19,61 N (2 kgf). Test force 19,61 N (2 kgf) will have greater precision; 9,807 N (1 kgf) may be used if the higher test force is not available on the apparatus, if cracking is a problem, or if it is preferred for a specific requirement. If cracking is a problem, or in the case of a specific requirement, the test forces within the range of 0,980 7 N (0,1 kgf) to 49,03 N (5 kgf), listed in Table 4, may be used.

5.6.3 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 5.6.10.

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.

5.6.4 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.

5.6.5 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. The surface of the test piece being tested shall be on a plane normal to the axis of the indenter.

5.6.6 Carefully adjust the illumination and focusing conditions 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.

5.6.7 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 test force shall be 15 s.

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

5.6.9 Allow a distance of at least 1,5 times the long diagonal length between the indentations as shown in Figure 8. If there is evidence of interaction between lateral cracking (from the sides of the indentation), then the spacing shall be increased. The minimum distance between the limit of any indentation and the edge of the test piece shall be at least 1,5 times the long diagonal of the indentation.

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

5.6.11 If there is excessive cracking from the indentation sides, 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 9 provides guidance in this assessment.

5.6.12 Measure the length of the long diagonal to within 0,2 µm for less than 50 µm, or to within 0,5 µm for equal to or more than 50 µm. The length is used for the calculation of the Knoop hardness number. If one leg (one-half) of the long diagonal is more than 10 % longer than the other (see Figure 9), or if the ends of the diagonals are not both in the field of focus, the surface of the test piece may not be normal to the axis of the indenter. Align the test piece surface properly and make another indentation. Follow the manufacturer's instructions very carefully with regards to the proper usage of the measuring crosshairs. Figure 10 is provided for guidance.

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

5.6.14 Calculate the Knoop hardness, HK, for each valid indentation, using the formula in Table 3. Calculate the mean hardness for all valid indentations and the standard deviation. The calculated Knoop hardness shall be expressed with three significant numbers (e.g. 15,4 GPa HK 9,807 N or 1520 HK 1)

5.6.15 See ISO 4545-4 for a table of values for use in tests made on flat surfaces.

5.7 Accuracy and uncertainty

The Knoop test is conducted in the same manner as the Vickers test, except that only the long diagonal is measured. Since the indentation is much shallower than the Vickers indentation, the angle of intersection of the indentation surfaces with the original surface is small, and the optical contrast this produces in conventional measurement systems tends to be poorer than in the Vickers case. Possible measurement errors and biases are thus much larger, and as the round-robin exercise[4] demonstrated, the fractional error in the test results is similar to or greater than that for Vickers tests at the same force. Thus, although the HK test is sometimes recommended as being more benign to the material and covering more microstructure because of the greater length of the diagonal than in the Vickers case, the human element cancels out any advantages. The tendency for generating cracks at the tips of the long diagonal provides additional visual uncertainty for the observer.

The comments made in 4.7 concerning use of the scanning electron microscope also apply to Knoop tests.

Since Knoop tests are generally conducted at forces derived from masses of or less than 2,0 kg, the errors in measurement at HK 1,0 could be ±1,0 GPa (±100 in hardness number) (typically 5 % to 8 %) rising to ±2,5 GPa (±250 in hardness number) (typically 10 % to 15 %) at HK 0,2.

5.8 Test report

The test report shall include the following information:

a) a reference to this document, i.e. ISO 14705:2016;

b) an information on the test piece;

c) the test conditions:

   1) thickness of test piece;

   2) test force, if different from 0,980 7 N (1 kgf) or 19,61 N (2 kgf), substatiate;

   3) surface condition (polishing method; describe other treatments);

   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:

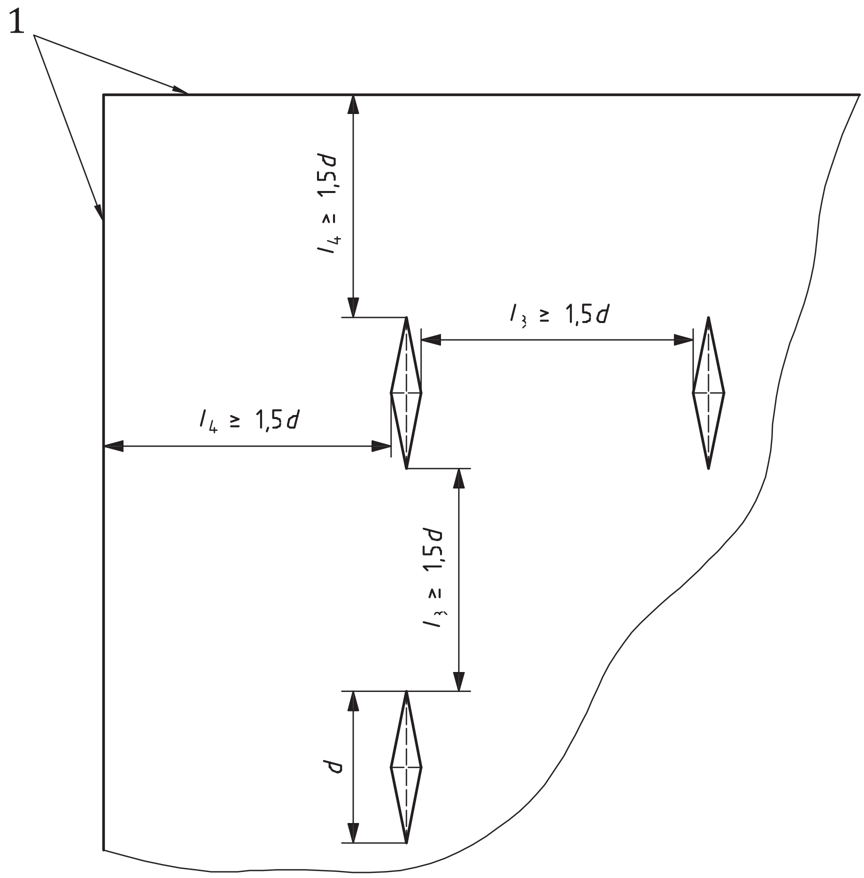
   1) individual valid Knoop hardness values;

   2) arithmetic mean of the Knoop hardness;

   3) standard deviation (SD) of the Knoop hardness;

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

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



Key

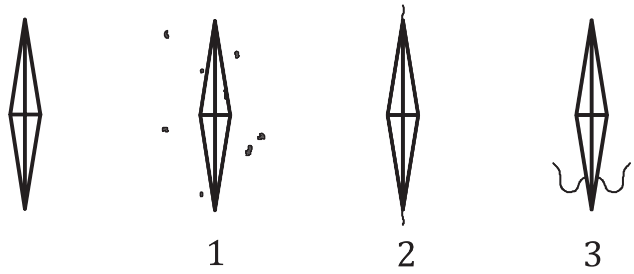
1 edge of test piece

d length of the long diagonal of indentation

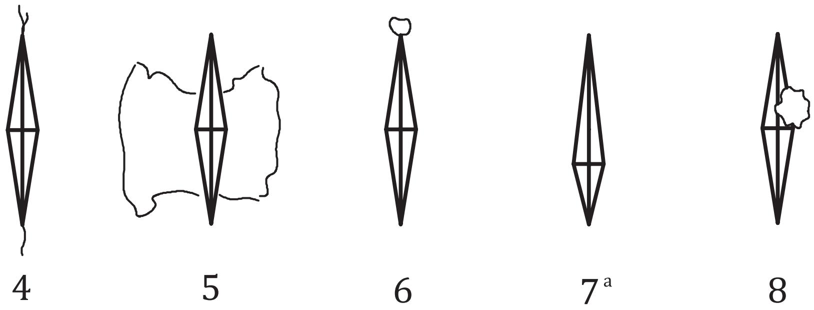
l_3 distance between nearest corners of indentations

l_4 distance from the corner of indentation to the edge of test piece

Figure 8 — Closest permitted spacing between indentations and from indentation to the edge of test piece for Knoop indentations



a) Acceptable indentations



b) Unacceptable indentations

Key

1 porosity

2 small tip cracks

3 small lateral cracks

4 large tip cracks

5 large lateral cracks

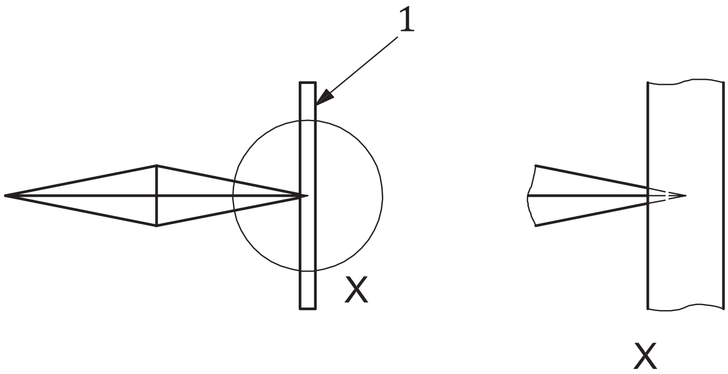
6 pore at tip

7 asymmetrical

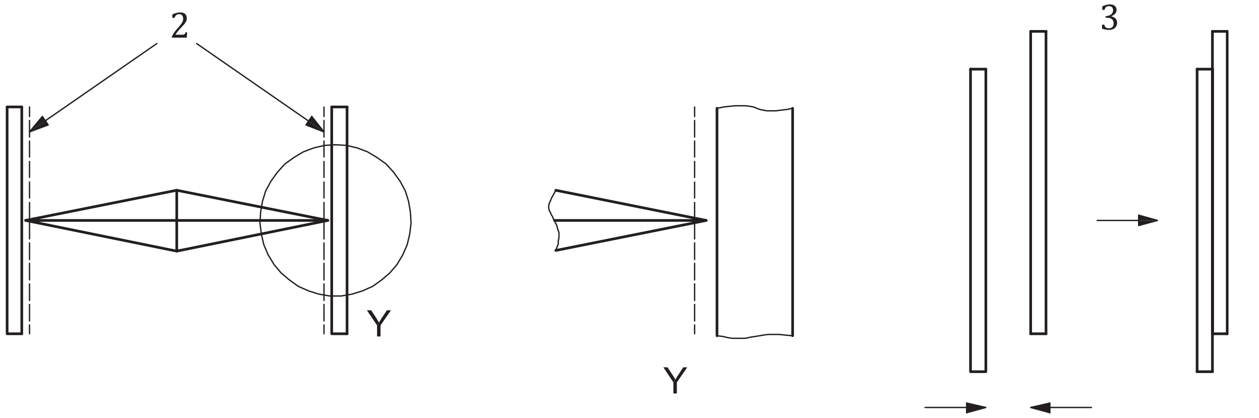
8 indentation on a large pore

a Permissible degree of asymmetry as defined in 5.6.12.

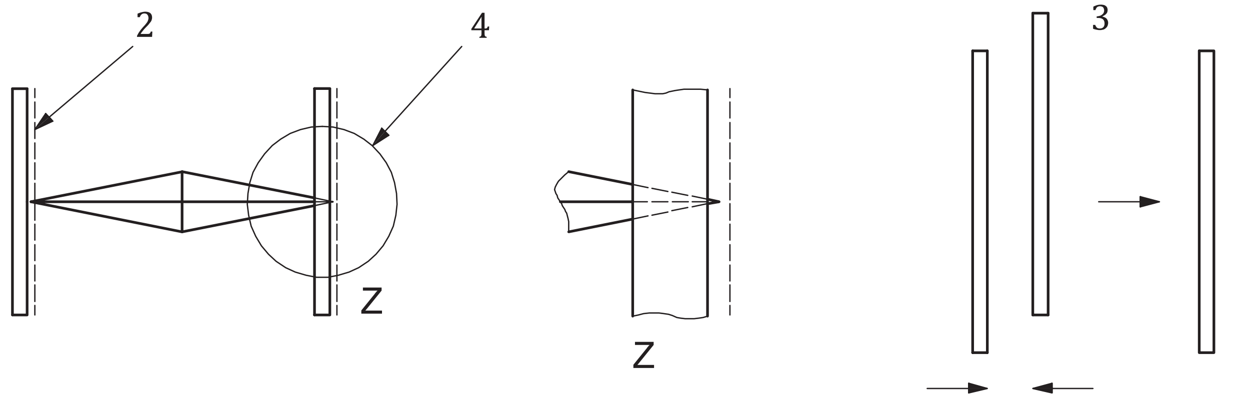
Figure 9 — Guidelines for the acceptability of Knoop indentations



a) Incorrect, crosshair completely covers the tip.



b) Correct, **double crosshair measurement system.** The indentation is intended to be measured between two crosshairs or measuring lines. Indentation tips should be on the inside edge (in the fringe) of each crosshair. The measuring system is zeroed by bringing together the adjacent edges of the crosshairs, as shown on the right.



c) Correct, single crosshair and some double crosshair measurement systems. The indentation tip is on the same side of the crosshair line(s). For a single crosshair system, the measuring system is zeroed when the crosshair is correctly positioned on one indentation, or with both lines superimposed in a double crosshair system, as shown on the right.

Key

1 crosshair
2 light fringe (size is exaggerated)
3 setting to zero
4 tip in the fringe

Figure 10 — Crosshair measurement system for Knoop indentation

Bibliography

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

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

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

[4] Butterfield D.M., Clinton D.J., Morell R. "The VAMAS Hardness Test Round-Robin on Ceramic Materials", VAMAS Report No. 3, National Physical Laboratory, Teddington, Middlesex, TW11 0LW, UK, April 1989, summarized in Euroceramics, Vol. 3, 3.339-3.345 (1989), published by Elsevier Science Publishers Ltd., London.

ICS 81.060.30

Price based on 20 pages