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ISO 4516

Second edition
2002-06-15

Metallic and other inorganic coatings — Vickers and Knoop microhardness tests

Revêtements métalliques et autres revêtements inorganiques — Essais de microdureté Vickers et Knoop

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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 3.

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

ISO 4516 was prepared by Technical Committee ISO/TC 107, *Metallic and other inorganic coatings*, Subcommittee SC 2, *Test methods*.

This second edition cancels and replaces the first edition (ISO 4516:1980), which has been technically revised.

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Metallic and other inorganic coatings — Vickers and Knoop microhardness tests

1 Scope

This International Standard describes the application of the Vickers and Knoop micro-indentation tests for determining the microhardness of metallic and other inorganic coatings. This method is applicable where indenter forces generally need to be below 10 N such as for electrodeposited coatings, autocatalytic coatings, sprayed coatings and anodic coatings on aluminium. It is applicable to measurements normal to the coated surface as described in 7.4 and to measurements on cross-sections as described in 7.3.

NOTE 1 Attention is drawn to ISO 4545, ISO 6507-1, ISO 6507-2 and ISO 6507-3, which describe Knoop and Vickers hardness testing of metallic materials. Other International Standards for instrumental indentation testing, the verification of microindentation testing instruments and for the verification of reference blocks to be used with such instruments are currently being developed (e.g. ISO 14577 Parts 1 to 4).

NOTE 2 Usually for hardness measurements of coating test forces in the microhardness range in accordance with ISO 6507-1 are used. However, since the largest possible test force should be selected, test forces of the low force and hardness ranges may also be used.

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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 1463, *Metallic and oxide coatings — Measurement of coating thickness — Microscopical method*

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

ISO 9002, *Quality systems — Model for quality assurance in production, installation and servicing*

3 Principle

A testing instrument slowly lowers an indenter vertically on to the test surface and holds it there for a specified time under a specified load (see 6.2). The tolerance of the applied test force is within 1 % of that specified.

An indenter is forced into the coating and the diagonal(s) of the indentation left in the surface after removal of the indenter is measured using a microscope. The indenter is applied such that the resultant indentation does not contain artefacts of the loading apparatus or procedure but rather is characteristic of the coating.

A number, known as the Vickers or Knoop hardness number, is derived from this measurement using the symbols and designations given in clause 4.

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4 Symbols and designations

Vickers and Knoop hardnesses are denoted respectively by the symbols HV and HK preceded by the hardness value and followed by:

a) a number representing the test force (force in newtons multiplied by proportionality factor 0,102) (see Table 1);

b) the application time of test force, in seconds, if different from the time specified in 6.3.

EXAMPLE 1 640 HV 0,1: Vickers hardness of 640 determined using a test force of 0,980 7 N applied for between 10 s and 15 s.

EXAMPLE 2 640 HK 0,1/20: Knoop hardness of 640 determined using a test force of 0,980 7 N applied for 20 s.

Table 1 — Symbols and designations

Symbol	Measuring unit	Designation	
		Vickers	Knoop
F	Force: N	Test force: N	Test force: N
d	Diagonal measurement: µm	Arithmetic mean of the two separately measured diagonals $d = \frac{d_1 + d_2}{2}$	Length of longer diagonal
HV and HK	—	Vickers hardness number = $(0{,}102\ F)/A_V$ = $189{,}1 \times 10^6\ F/d^2$	Knoop hardness number = $(0{,}102\ F)/A_K$ = $1\ 451{,}4 \times 10^6\ F/d^2$
A_V	mm^2	Sloping surface area of indentation (contact area)	
A_K	mm^2		Projected area of the indentation
t	µm	Coating thickness	Coating thickness
s	—	Standard deviation $s = \sqrt{\frac{(\overline{HV} - HV)^2}{(n-1)}}$	Standard deviation $s = \sqrt{\frac{(\overline{HK} - HK)^2}{(n-1)}}$
N	—	Number of measurements	Number of measurements
$\overline{HV}$ and $\overline{HK}$	—	$\overline{HV} = \Sigma\ HV/n$	$\overline{HK} = \Sigma\ HK/n$
V	%	Coefficient of variation $V = 100s/\overline{HV}$	Coefficient of variation $V = 100s/\overline{HK}$

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5 Apparatus

5.1 Testing instrument, to perform the task described in clause 3.

5.2 Indenters

5.2.1 Form and dimensions

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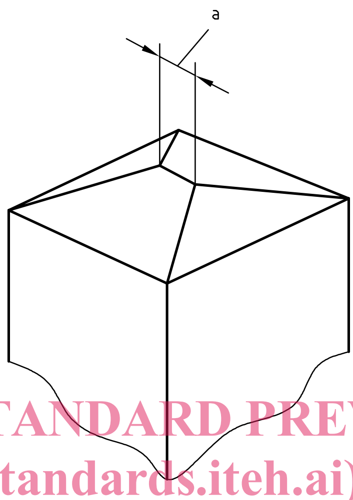
5.2.1.1 Vickers indenter

The indenter consists of a diamond in the form of a right pyramid with a square base (see Figure 1). The angle at the vertex between opposite faces shall be 136° ± 0,5°. This angle shall have been verified with a 2-circle goniometer of appropriate accuracy. The relation between the diagonals d_1 and d_2 and the depth of the indentation, h, is approximately 7:1.

The four faces shall be equally inclined to the axis of the indenter (within 0,5°) and shall meet at a point; any line of junction (offset) between two opposite faces shall not exceed 0,5 µm. The usual shape of the point is shown in Figure 2 as it would appear under high magnification.



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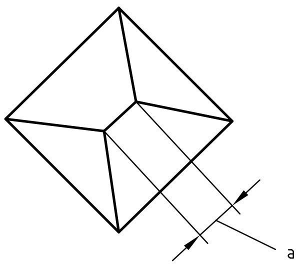
[a] Offset

Figure 1 — Vickers indenter



[a] Maximum offset 0,5 µm

Figure 2 — Vickers indenter offset

5.2.1.2 Knoop indenter

The indenter consists of a diamond tipped right pyramid with a rhomboid base (see Figure 3). The angles at the vertex shall be 172,5° ± 0,3° and 130 ± 0,3°. These angles shall have been verified with a 2-circle goniometer of appropriate accuracy. The four faces shall be equally inclined to the axis of the indenter (within 0,5°) and shall meet at a point; any line of junction (offset) between two opposite faces shall not exceed 1,0 µm. The usual shape of the point as it would appear under high magnification is shown in Figure 4. The relation between the long diagonal and the depth of the indentation is approximately 30:1.

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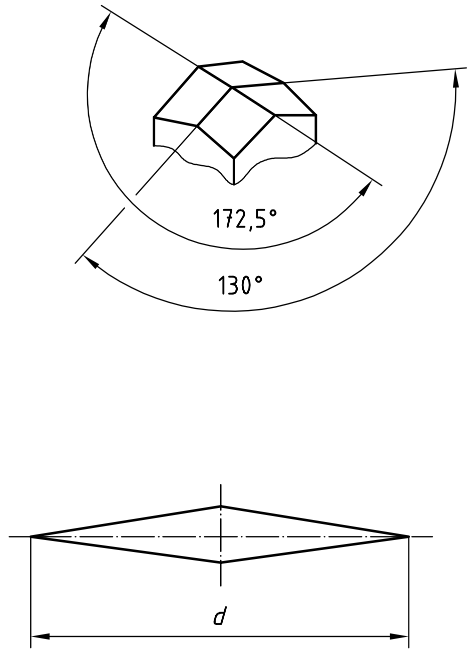

Figure 3 — Knoop indenter

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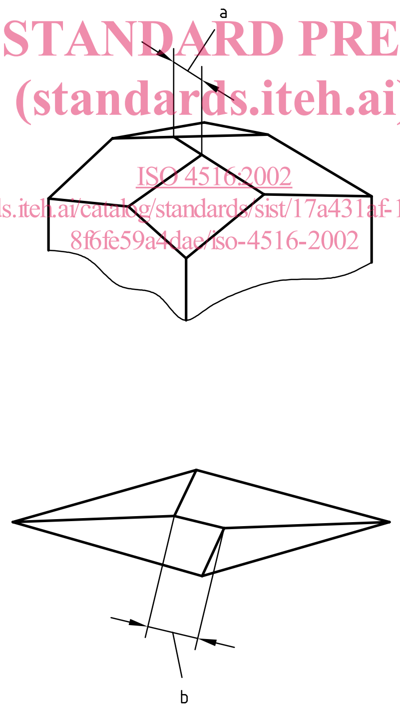

[a] Offset

[b] Maximum offset 1,0 µm

Figure 4 — Knoop indenter offset

5.2.2 Surface characteristics

The indenter faces shall be smooth and free from cracks or other imperfections or defects. The diamond shall be examined periodically. Any foreign materials shall be removed. The indenter shall be replaced if it is cracked, chipped or loose in its mounting.

NOTE The diamond can be cleaned by pressing it into copper or steel of low hardness, or by means of a suitable solvent not harmful to the equipment. The diamond can be examined using a scanning electron microscope or an optical microscope

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with a numerical aperture greater than 0,85. Cracks and other imperfections or defects can sometimes be detected by examining the shape and symmetry of the indentation. Additional cleaning techniques may be provided by the manufacturer.

5.3 Hardness reference blocks

The test blocks shall be cleaned immediately prior to use because frequently they are coated to prevent corrosion during storage.

To verify the hardness testing instrument and the measurement, the measurement shall be compared with blocks, the hardness of which is close to the range of interest. Each block shall be of a material the grain size of which is small compared with the indentation size and shall have a known uniform hardness measured at a particular test force specified by the calibrating authorities or the testing instrument manufacturer. The test force shall be within 25 % of that used in the actual tests. In addition:

a) the block shall be calibrated to be within 5 % of the true hardness point to point;

b) the test and support surfaces of the block shall be parallel to ± 0,000 5 mm/mm;

c) the maximum deviation in flatness shall not exceed 5 µm;

d) the test surface roughness *Ra* shall not exceed 0,1 µm;

e) the block shall be demagnetized by the manufacturer and maintained in that state by the user.

The frequency of indirect verification with reference blocks will depend on the frequency of use. It is normal to verify the instrument before each series of measurements; the frequency of indirect verification shall not exceed 12 months.

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6 Factors affecting measurement accuracy

6.1 Test force

The micro-indentation value obtained depends on the force applied to a greater extent than with macrohardness measurements (forces greater than 10 N). Because of a number of factors, including anisotropy, the test specimens shall be marked to indicate where the measurement has been carried out and the reference test area shall be recorded in the test report (see clause 8). To ensure that comparable hardness values are obtained the tests shall be performed using the same force (within 1 %) and application time of test force as those used in the actual tests.

To obtain the most accurate micro-indentation values for the coating, the maximum forces compatible with the thickness of the coating shall be used (see Figure 5, 7.3.1, 7.3.2 and 7.4). It is possible to obtain comparable results only if the same test force is used.

Table 2 lists a selection of test forces.

Table 2 — General guide to the selection of test forces (see Figure 5, 7.3.1, 7.3.2 and 7.4)

Material	Test force (*F*)	
	N	Test conditions
Coatings with hardness values greater than 300 (HV or HK)	0,981	HV 0,1 or HK 0,1
Hard anodic oxide coatings on aluminium	0,490	HV 0,05 or HK 0,05
Materials with hardness less than 300 (HV or HK), such as precious metals and their alloys, and thin coatings in general	0,245	HV 0,025 or HK 0,025

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