INTERNATIONAL STANDARD

ISO 4516

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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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Contents

Page

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.

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.

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.

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 $s = \sqrt{\frac{\left(\overline{\text{HV}} - \text{HV}\right)^2}{(n-1)}}$	Standard deviation $s = \sqrt{\frac{\left(\overline{\text{HK}} - \text{HK}\right)^2}{(n-1)}}$
N	—	Number of measurements	Number of measurements
$\overline{\text{HV}}$ and $\overline{\text{HK}}$	—	$\overline{\text{HV}} = \Sigma\ \text{HV}/n$	$\overline{\text{HK}} = \Sigma\ \text{HK}/n$
V	%	Coefficient of variation $V = 100s/\overline{\text{HV}}$	Coefficient of variation $V = 100s/\overline{\text{HK}}$

5 Apparatus

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

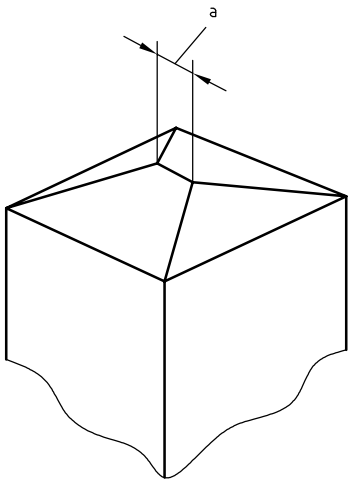
5.2 Indenters

5.2.1 Form and dimensions

5.2.1.1 Vickers indenter

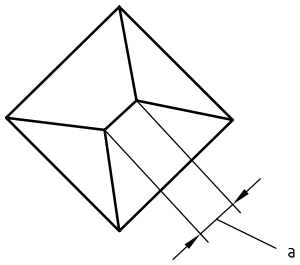
The indenter consists of a diamond in the form of a right pyramid with a square base (see Figure 1). The angle at the vertex between opposite faces shall be 136° ± 0,5°. This angle shall have been verified with a 2-circle goniometer of appropriate accuracy. The relation between the diagonals d_1 and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



a Offset

Figure 1 — Vickers indenter

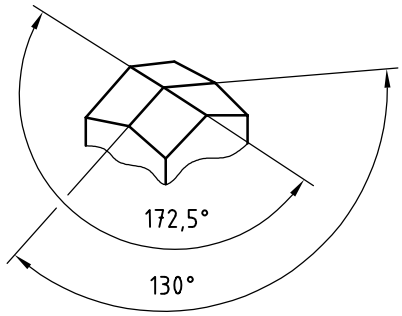


a Maximum offset 0,5 µm

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.



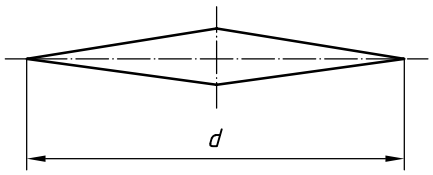
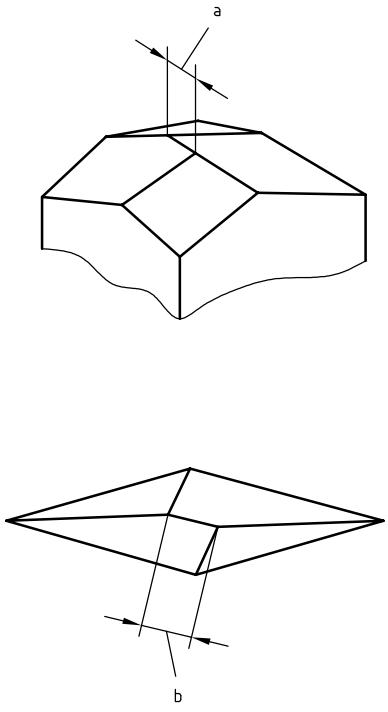


Figure 3 — Knoop indenter



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

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 $\pm$ 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.

6 Factors affecting measurement accuracy

6.1 Test force

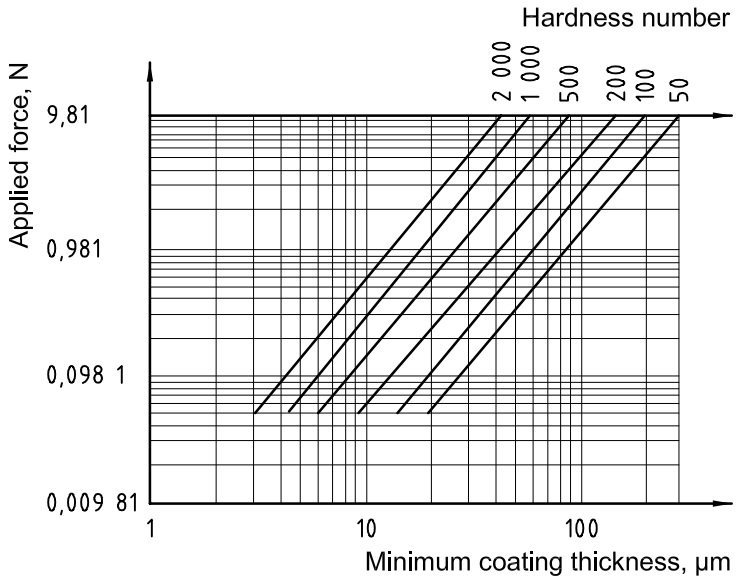
The micro-indentation value obtained depends on the force applied to a greater extent than with macrohardness measurements (forces greater than 10 N). Because of a number of factors, including anisotropy, the test specimens shall be marked to indicate where the measurement has been carried out and the 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.

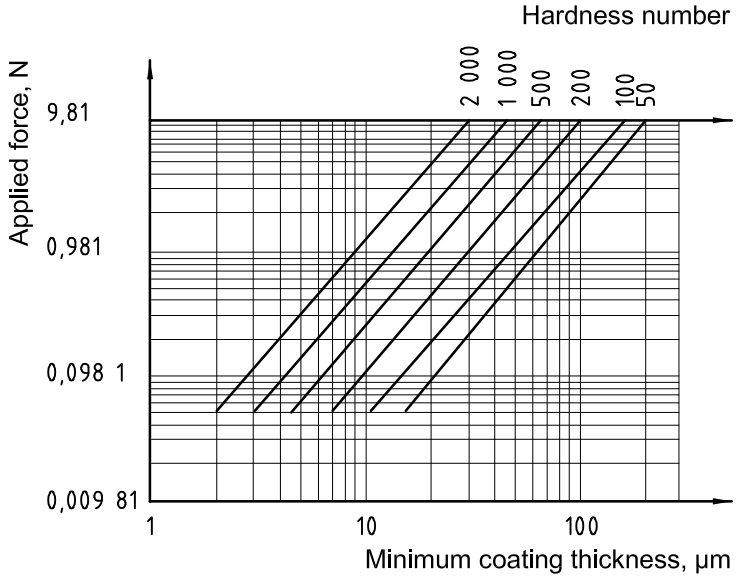
Table 2 lists a selection of test forces.

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



a) Vickers hardness



b) Knoop hardness

Figure 5 — Relationship between minimum coating thickness, applied force and hardness

6.2 Velocity of indenter

If the indenter is brought into contact with the test surface at too great a velocity, the hardness value obtained will be too low. The velocity of the indenter shall be such that any reduction in velocity will not result in a change in hardness value. The load as a function of time shall be measured on an appropriate instrument. At no time during the loading process shall the load exceed the specified load.

The velocity for most instruments shall not exceed 15 µm/s.

To determine whether the velocity is correct, repetitive tests at gradually decreasing velocities shall be made. The velocity below which there is no variation in the result is the velocity to use with the chosen force. These tests shall be performed with the same materials and test forces that will be used for the hardness measurements on the coating.

6.3 Application time of test force

The force should normally be applied for 10 s to 15 s. When the force is applied for an application time of test force other than 10 s to 15 s, this shall be stated in the test report (see clause 8).

If the test force is applied for less than 10 s, the size of the indentation can be time-dependent and the hardness values will be high. For some materials which exhibit distinct creep at room temperature, the application time of test force will be more critical. Longer application times can be more sensitive to vibration.

6.4 Vibration

Vibration represents a serious source of error irrespective of the force applied, but the effects are far more evident with small forces. In general, lower hardness values are obtained if vibrations are present. This source of error can be detected by comparative measurements on a specimen of known hardness nearly equal to the hardness of the test surface (see 5.3). The effects of vibration can be reduced by appropriately isolating the instrument from the environment, for example by mounting it on a suitable isolation table.

Acoustic noise which is not controlled by the isolation table can also contribute to errors in the hardness measurement and result in low values. Examples of sources of vibration might be:

a) cooling fans;

b) air conditioners;

c) highway noise.

6.5 Surface condition of the test piece

6.5.1 Roughness

If the test surface is rough, it may be impossible to measure accurately the length of the indentation diagonal. This is one reason why micro-indentation measurements are most often made on the coating cross-section. The test piece may be chemically, electrochemically or mechanically polished.

Polishing shall be carried out in such a way as to minimize local heating or work hardening that would change the measured hardness.

Because of the surface roughness of sprayed metal coatings, micro-indentation measurements of such coatings shall normally be made on the cross-section. Measurements on surfaces supplied in a fine ground condition should be made on the ground surface. The surface finish shall be specified in the test report (see clause 8). Since coatings that are prone to work hardening will always be affected to some extent by metallographic specimen preparation techniques, care shall be taken to minimize this effect.

6.5.2 Curvature of the surface

Surface curvature introduces a certain error in determining hardness which increases as the radius diminishes. On convex surfaces, higher hardness readings than the actual values can be obtained.

If the Vickers hardness test has to be carried out on a sample with an excessively curved surface, the influence of the curvature shall be compensated for by the use of correction factors in accordance with ISO 6507-1.

Knoop hardness values can be corrected approximately by using a factor that is obtained by testing samples with equal radius and known hardness nearly equal to the hardness of the test object. If the parts are cylindrical, the long diagonal is aligned in the direction of the cylinder axis. See 8.1 e).

6.6 Orientation

6.6.1 Alignment of the test surface

If the test surface is not perpendicular to the axis of the indenter, the measurement will not be valid. Accurate results will be obtained when the error in perpendicularity is less than 0,5°. If the material is isotropic, non-perpendicularity can exist when one leg of a diagonal is noticeably longer than the other leg of the same diagonal.

6.6.2 Rigidity

The test piece shall be held firmly so that displacement or movement does not occur during the test. To avoid distorting the indentation the specimen to be examined shall be positioned on the supporting table or in the mounting attachment so that the test surface is perpendicular to the effective axis of the test force. This position shall be maintained during the entire test. The position of the test surface shall not change by more than 0,5 µm during the test.

6.7 Brittle materials

If cracks occur during indentation, valid hardness numbers are not obtained. This difficulty may be overcome by using a reduced test force, with a subsequent reduction in accuracy.

6.8 Microscopic resolution

The measuring accuracy as specified in 7.6 shall be achieved by using a dry objective the numerical aperture of which is consistent with causing the indentation to fill the field of view. Using the illuminating system, the specimen shall be positioned at right angles to the optical axis. The size of the illumination aperture shall be adjusted by means of the illuminator diaphragm until the reflected light fills between two thirds and the whole of the objective aperture but does not extend beyond it. For very hard coatings (hardness > 750 HV) objective apertures $\geqslant 0{,}85$ shall be used.

NOTE 1 By using a green filter, the measurement can be carried out within the range of the eye's maximum sensitivity.

The magnification of the optical system shall be calibrated with a suitable stage micrometer.

NOTE 2 A significant reduction in accuracy occurs when the diagonal is less than 10 µm.

6.9 Location of indentation

Hardness measurements can be affected by the proximity of materials other than the coating; e.g., if the indentation is close to the substrate, and the substrate is softer than the coating, the hardness measurements obtained can be too low. If the material contains precipitates or inclusions then the indentation can be distorted near those inhomogeneities in the microstructure. This type of error can be indicated by the indentation not having its normal shape (see 7.3.1, 7.3.2 and 7.4).

7 Measuring procedure

7.1 General

Use each instrument in accordance with the manufacturer's instructions, observing the factors referred to in clause 6.

The hardness test can be carried out on a cross-section of the coating, or on the surface itself, provided that the characteristics of the coating (smoothness, thickness, etc.) permit accurate readings of the diagonal(s) of the indentation.

7.2 Selection of test force

Unless otherwise specified or decided upon for technical reasons, use the appropriate test force suggested in 6.1.

If for some reason these suggested test forces are not used, the hardness values obtained can differ markedly from those that would have been obtained if the suggested test forces had been used. The results, however, may be of value for comparative or control purposes.

7.3 Measurements on cross-sections

7.3.1 Coating thickness

When the Vickers indenter is used, the thickness of the coating at the point of measurement shall be sufficiently great to give indentations that conform to the following conditions when the surface is correctly aligned (see 6.6.1), and when one diagonal is at right angles to the edge of the coating. When the Vickers indenter is used, the thickness of the coating shall be at least 100 µm (HV 0,025) for soft coatings and 80 µm (HV 0,1) for hard coatings. In addition:

a) the two diagonals shall be of equal length to within 5 %;

b) the four edges of the indentation shall be of equal length to within 5 %.

When the Knoop indenter is used the thickness of the coating at the point of measurement shall be at least 40 µm for soft coatings ($<$ 300 HK) and at least 25 µm for hard coatings ($>$ 300 HK). This is approximately equivalent to 0,6 times the length of the long diagonal. The symmetry of the indentation shall be such that half the length of the long diagonal shall be within 20 % of the other half.

7.3.2 Test piece preparation

The test piece shall be mounted, polished and etched in accordance with ISO 1463. It is also possible to remove a part of the sample and coat it to a thickness of at least 12 µm with a material having approximately the same hardness as the original coating. Ensure that work hardening is minimal (see 6.5.1). If possible avoid testing etched surfaces.

7.3.3 Indentation

In the case of the Knoop indentation, the long diagonal shall be parallel to the coating substrate. The spacing of the Knoop indentations on hard coatings shall be 2,5 times the short diagonal from a substrate or interface, or from another indentation, and three times the short diagonal for soft coatings. In the case of the Vickers indentation, one of the diagonals shall be approximately 90° to the coating substrate interface. The distance of the indentation centre from another indentation centre shall be at least five times the length of the measured diagonal. For determining indentation spacing when testing laminated materials, a bond surface shall be considered as an edge. See 7.3.1.

7.4 Measurements normal to the coating surface

Before carrying out a hardness test normal to the surface, the thickness of the coating shall be measured in accordance with the method described in ISO 1463.

The applied force shall be such that the depth of indentation is less than one tenth of the thickness of the coating (see Figure 5).

The thickness of the coatings shall be at least 1,4 times the average length of the diagonals for the Vickers test, and at least 0,35 times the length of the longer diagonal for the Knoop test. The minimum thickness shall be 15 µm for Vickers and Knoop (see Figure 5).

7.5 Temperature

The test shall be carried out at 23 °C ± 5 °C. Any temperature outside this range shall be stated in the test report (see clause 8).

7.6 Optics

The hardness indentation shall be measured in the central area of the eyepiece field and the indentation area shall not exceed two thirds of the total field area.

The indentation shall be measured either with a micrometer eyepiece or, preferably, with a screw micrometer filar eyepiece. For differential measurements, the measuring cursor shall be moved into the measuring position from the same side and a reading shall be taken at the same edge.

The magnification shall be calibrated before each set of measurements or if changes to the optical system have been made that will alter the magnification.

The magnification of the optical system shall be calibrated with a stage micrometer having a tolerance of $< 0{,}2$ µm. The magnification shall be calibrated by the person making the hardness measurements. The measurement may be made using the appropriate filar eyepiece or by translating the stage with appropriate precision.

The use of a televisual optical system or image analysis can introduce errors due to the sensitivity to contrast of the measurement. Therefore, when using television systems, calibration shall be undertaken with appropriate standards.

7.7 Calculation

For calculating Vickers hardness, the average length of the diagonal, d, shall be the arithmetic mean of the length of two independently measured diagonals of the indentation. If the coating material is amorphous or has a grain size that is small compared with the indentation size, the hardness measurement shall be considered valid when the difference in the length of the two diagonals is less than 5 % of the longer diagonal.

The hardness value shall be obtained by taking at least five indentations for each specimen within a defined reference area on the significant surface and then calculating the mean hardness value for this group of measurements.

The coefficient of variation is normally expected to be less than 5 %, but if a larger value is obtained, it shall be reported in the test report. The range is defined as the difference between the maximum and minimum readings and may be specified in the test report (see clause 8).

7.8 Test coupons

Test coupons may be used when specified by an appropriate document. Test coupons may be used if the production parts are of such geometry that they are not suitable for hardness testing. This substitution is valid provided that the specimens are prepared using the production process or its closest equivalent. In the case of plated parts, the test coupons may serve as a useful tool for electrolyte control, particularly in the case of coatings

such as gold for which the hardness is sensitive to the composition of the plating solution and to all the electroplating variables.

The electroplating conditions for test coupons, such as current density, temperature, agitation and solution composition, shall be kept as close as possible to those used on production parts for the electroplating process under test.

8 Test report

8.1 Requirements

The microhardness test results shall be reported in accordance with ISO 9002 and shall include:

a) reference to this International Standard, i.e. ISO 4516;

b) the microhardness values obtained using appropriate symbols and designation (see clause 4);

c) location of the measurement (e.g., on a cross-section or normal to the surface) and reference area;

d) surface roughness;

e) the curvature of the surface;

f) the temperature of the measurement, if different from that specified in 7.5;

g) coefficient of variation;

h) coating thickness;

i) date of the measurement;

j) the name of the operator;

k) application time of test force (if it is other than 10 s to 15 s).

8.2 Sample test report

a)	Standard used in this measurement:	ISO 4516
b)	Microhardness:	800 HV 0,1/25
c)	Measurement carried out on:	cross-section
d)	Surface roughness:	0,1 µm Ra
e)	Surface curvature:	flat
f)	Measurement temperature:	25 °C
g)	Coefficient of variation: in %	0,1 %
h)	Coating thickness:	25 µm
i)	Date of this report:	2001-02-19
j)	Operator:	John Doe
k)	Application time:	25 s

Bibliography

[1] ISO 4545, *Metallic materials — Hardness test — Knoop test*

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

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

[4] ISO 14577-1, *Metallic materials — Instrumented indentation test for hardness and materials parameters — Part 1: Test method*

[5] ISO 14577-2, *Metallic materials — Instrumented indentation test for hardness and materials parameters — Part 2: Verification and calibration of testing machines*

[6] ISO 14577-3, *Metallic materials — Instrumented indentation test for hardness and materials parameters — Part 3: Calibration of reference blocks*

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