International Standard 4516

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION•МЕЖДУНАРОДНАЯ ОРГАНИЗАЦИЯ ПО СТАНДАРТИЗАЦИИ•ORGANISATION INTERNATIONALE DE NORMALISATION

Metallic and related coatings — Vickers and Knoop microhardness tests

Revêtements métalliques — Essais de microdureté Vickers et Knoop

First edition — 1980-05-15

UDC 669.058 : 620.178.152.341.4 **Ref. No. ISO 4516-1980 (E)**

Descriptors : metal coatings, tests, hardness tests, microhardness tests, Vickers hardness.

Price based on 7 pages

Foreword

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Draft International Standards adopted by the technical committees are circulated to the member bodies for approval before their acceptance as International Standards by the ISO Council.

International Standard ISO 4516 was developed by Technical Committee ISO/TC 107, *Metallic and other non-organic coatings*, and was circulated to the member bodies in August 1978.

It has been approved by the member bodies of the following countries :

Australia	Israel	Spain
Czechoslovakia	Italy	Sweden
Egypt, Arab Rep. of	Japan	Switzerland
France	Mexico	Turkey
Germany, F. R.	Netherlands	United Kingdom
Hungary	Poland	USA
India	Romania	USSR
Ireland	South Africa, Rep. of	

No member body expressed disapproval of the document.

Metallic and related coatings — Vickers and Knoop microhardness tests

0 Introduction

There are a number of factors involved in microhardness testing on which international agreement has not yet been reached. These include, in particular :

- the verification of the testing machines and of standard test blocks;
- the velocity of the indenter and its importance compared to that of its kinetic energy.

However, despite the many areas of ambiguity, the parameters specified in this International Standard are important enough to enable a considerable measure of standardization to be achieved, as the procedures described are used extensively in practice.

1 Scope and field of application

This International Standard describes the application of the Vickers and Knoop microhardness tests to metallic coatings.

This method is applicable where macrohardness testing is not suitable because of the large test forces required, that is, where indenter forces generally need to be below 10 N as for electrodeposited coatings, autocatalytic coatings, sprayed coatings and anodic coatings on aluminium.

To obtain satisfactory results, it is necessary to have a sufficient coating thickness, and it is usually advantageous to carry out the test on cross-sections (see 7.3 and 7.4).

NOTE — Attention is drawn to the fact that a general standard on Vickers and Knoop microhardness tests is being developed by ISO/TC 164, *Mechanical testing of metals*. The same technical committee is also developing standards for the verification of microhardness testing machines and for the verification of standardized test blocks to be used with such machines.

2 References

ISO/R 409, *Table of Vickers hardness values (HV) for metallic materials.*

ISO 1463, *Metallic and oxide coatings — Measurement of thickness by microscopical examination of cross-sections.*

3 Principle

The test consists of forcing an indenter into the metal coating and measuring with a microscope the diagonal(s) of the indentation left in the surface after removal of the indenter.

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

4 Symbols and designations

Symbol	Measuring unit	Designation	
		Vickers	Knoop
F	N (1 N ≈ 0,102 kgf)	Test force in newtons	Test force in newtons
d	µm	Arithmetic mean of the two separately measured diagonals d' and d'' $d = \frac{d' + d''}{2}$	Length of the longer diagonal
HV	—	Vickers hardness number $\frac{0{,}102 \times F}{A_v} = 1{,}854 \times 10^6 \frac{0{,}102 \times F}{d^2}$ where A_v is the sloping surface area of indentation, in square millimetres*	
HK	—		Knoop hardness number $\frac{0{,}102 \times F}{A_k} = 14{,}229 \times 10^6 \frac{0{,}102 \times F}{d^2}$ where A_k is the projected area of indentation, in square millimetres*

* The areas corresponding to the measured diagonal(s) of the indentation are given in tables provided by the testing instrument manufacturers.

The symbols, HV and HK, are each supplemented by a number indicating the test force used, expressed in newtons multiplied by 0,102 and therefore equal to the test force expressed in kilograms-force.[1)]

Example :

If a force of 0,245 N is applied during the Vickers or Knoop microhardness tests, the symbol would be respectively HV 0,025 or HK 0,025 (where 0,025 is derived, in both cases, from 0,102 × 0,245). Conversely, the symbol HV 0,025 or HK 0,025, signifies a Vickers or Knoop hardness, respectively, resulting from an applied test force of
0,025 × 9,807 = 0,245 N.

5 Apparatus

5.1 Testing instrument

The testing instrument slowly lowers an indenter vertically on to the test surface and holds it there for a specified time under a specified load.

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' and d'', and the depth of the indentation, t, is approximately

$$7\,t = \frac{d' + d''}{2}$$

The four faces shall be equally inclined to the axis of the indenter (within 0,3°) 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.

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 between two opposite edges shall be 172,5 ± 0,08° in the lengthwise direction and 130 ± 0,08° in the breadthwise direction. These angles shall have been verified with a 2-circle goniometer of appropriate accuracy.

1) Technical committee ISO/TC 17, *Steel*, agreed in June 1970 that the formula of calculation and the system of designation for the hardness shall be such that the numerical value of the hardness number remains unaffected by the introduction of the SI unit of force, the newton, instead of the obsolete unit, kilogram-force. Thus the multiplier 0,102 must be applied both in the formula of calculation and in the symbol used in the designation of hardness.

The four faces shall be equally inclined to the axis of the indenter, (within ± 0,2°) and shall meet at a point; any line of junction (offset) between two opposite faces shall not exceed 1 µm. The usual shape of the point as it would appear under high magnification, is shown in figure 4.

5.2.2 Surface characteristics

The indenter faces shall be smooth and free from cracks or other faults. The diamond shall be examined periodically. Any foreign material shall be removed. If the indenter is cracked, chipped or loose in its mounting, it shall be replaced. 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 examination of the diamond can be carried out using a stereo-microscope with 100 X magnification. Cracks and other faults can sometimes be detected by examining the shape and symmetry of the indentation.

5.3 Standardized test blocks

To verify the hardness testing instrument and the measurements, it is recommended that the measurement be compared with standardized test blocks, the range of hardness of which should correspond to the whole range of hardness of interest. Each test block should be of a compact-grained metal 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 should correspond to that used in the actual tests.

6 Factors relating to accuracy

6.1 Test force

The microhardness value obtained depends on the applied force to a greater extent than with macrohardness measurements (forces higher than 10 N). Because of a number of factors, including anisotropy, it is very important to indicate where on the test specimens the measurement has been carried out. Comparable hardness numbers will only be obtained if tests are performed using the same force and duration.

To obtain the most accurate microhardness value for the coating it is advisable to use the maximum forces compatible with the thickness of the coating (see figure 5 and 7.3.1, 7.4 and 7.3.2). It is possible to obtain comparable results only if the same test force is used. The following test forces shall be used for the coatings indicated.

0,245 N (0,025 kgf)	materials with hardness less than 300, precious metals and their alloys and thin coatings in general
0,490 N (0,050 kgf)	hard anodic oxide coatings on aluminium;
0,981 N (0,100 kgf)	materials other than precious metals with hardness numbers greater than 300.

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 its reduction will not result in a higher hardness number. The correct velocity for most instruments is between 15 and 70 µm/s. To determine whether the velocity is correct, repetitive tests at gradually decreasing velocities should be made. The velocity below which there are no more variations 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.

6.3 Duration of application of test force

The force should normally be applied for 10 to 15 s. When the force is applied for a duration other than 10 to 15 s, the actual time of application should be stated in the test report, as specified in 8.2. If the test force is applied for less than 10 s, the size of the indentation may be time-dependent and the hardness numbers will be high. For some materials which exhibit distinct creep at room temperature, the duration of application of the force will be more critical.

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 mounting the test specimen on a rigid support.

6.5 Surface condition of the specimen

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 microhardness measurements are most often made on the cross-section. The specimen may be chemically, electrochemically, or mechanically polished. Mechanical polishing should be carried out so as to minimize local heating or working that would change the hardness.

Because of the surface roughness of sprayed metal coatings, microhardness measurements of such coatings shall normally be made on the cross-section. Measurements on surfaces supplied in a fine ground condition can be made on the ground surface.

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 and, on a concave surfaces, lower hardness readings than the actual values are obtained.

If the Vickers hardness test has to be carried out on a sample with excessively curved surface, the influence of the curvature can be compensated for by correction factors (see ISO/R 409). Knoop hardness values can be corrected approximately by using a factor which 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 longer diagonal is aligned in the direction of the cylinder axis.

6.6 Orientation

6.6.1 Alignment of test surface

If the test surface is not perpendicular to the axis of the indenter, the measurement will not be valid. If the material is isotropic, non-perpendicularity exists when one leg of a diagonal is noticeably longer than the other leg of the same diagonal.

6.6.2 Surface inclination

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 action of the test force otherwise the indentation will be distorted. This position shall be maintained during the entire test.

6.7 Brittle materials

If cracks occur during the indentation, valid hardness numbers are not obtained. This difficulty is often overcome by using a reduced test force.

6.8 Microscopic resolution

The measuring accuracy as specified in 7.6 is achieved by using a dry objective of ⩾ 400 X. Using the illuminating system, the specimen shall be positioned at right angles to the optical axis. The size of the illumination aperture should 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.

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

6.9 Location of indentation

The size and shape of the indentation can be affected by the proximity of materials other than the coating. For example, if the indentation is close to the substrate, and the substrate is softer than the coating, the hardness measurement obtained may be too low. This source of error may be indicated by the indentation not having its normal shape (see 7.3.1, 7.3.3 and 7.4).

7 Measuring procedure

7.1 General

Use each instrument in accordance with the manufacturer's instructions, bearing in mind the factors mentioned 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 specified in 6.1. If for some reason other test forces are used, the hardness values obtained may differ markedly from those which would have been obtained if the specified 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 shall be sufficiently great to give indentations which will conform with the following conditions when the test surface is correctly aligned (see 6.5.3), and when one diagonal is at right angles to the edge of the coating.

a) each corner of the indentation shall be at least half the length of a diagonal from any edge of the coating;

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

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

When the Knoop indenter is used, the thickness of the coating shall be at least 40 µm for soft coatings (gold, copper and silver) and at least 25 µm for hard coatings (nickel, cobalt, iron, and hard precious metals and their alloys).

7.3.2 Specimen preparation

Remove a part of the sample and overplate it to a thickness of at least 12 µm with a contrasting metal having approximately the same hardness as the coating.

Mount, polish and etch the specimen in accordance with ISO 1463. Work hardening must be minimal (see 6.5.1).

7.3.3 Indentation

In the case of the Knoop indentation, the longer diagonal shall be in the middle of the coating and parallel to the coating edge. In the case of the Vickers indentation, one of the diagonals shall be at approximately 90° to the coating substrate interface. The distance of the indentation centre from another indentation centre shall be at least 2,5 times the length of the measured diagonal. When laminated material is tested, a bond surface shall be considered as an edge for determining indentation spacing.

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 using the suitable method described in the relevant International Standard.

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). This means that the thickness of the coating should 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. Satisfactory measurements can be obtained for thinner coatings if the hardness of the substrate and coating are very similar.

7.5 Temperature

The test shall be carried out at 23 ± 5 °C. The temperature shall be stated in the test report (see clause 9) if it is not 23 ± 5 °C.

7.6 Optics

The hardness indentation should 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 should always be moved into the measuring position from the same side and a reading always taken at the same edge.

The screw micrometer eyepiece is calibrated by comparing it with a stage micrometer having a tolerance of < 0,2 µm.

To achieve a measuring error of 5 % or lower, the indentation diagonals should be 16 µm or longer.

7.7 Calculation

For calculating Vickers hardness, *d* shall be the arithmetic mean of the length of the two independently measured diagonals of the indentation. If the material is amorphous or compact-grained, the hardness measurement is considered valid when the difference in the length of the two diagonals is less than 10 % of the longer diagonal.

Take at least five readings for each specimen and calculate the mean hardness value each time. The microhardness testing instrument manufacturers provide tables for calculating the hardness number.

7.8 Test coupons

Test coupons may be used if the production parts have insufficient plating thickness for measurement. The values obtained may not reflect the "true" hardness of the thin coatings on production parts. However, the values may be useful when they correlate with other coating properties, such as wear resistance. The test may serve as a useful tool for electroplating bath control, particularly in the case of coatings such as gold for which the hardness is sensitive to the composition of the bath 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 employed on production parts in the electroplating process under test.

8 Expression of results

8.1 Test results shall be recorded as the hardness number range (the highest and lowest numbers) obtained for at least five indentations. The average hardness number may also be recorded.

8.2 In expressing the results, the Vickers or Knoop hardness symbol, HV or HK, shall be preceded by the range determined and shall be followed by a number indicating the force (expressed in newtons multiplied by 0,102) and a second number indicating the duration of application of the force if other than 10 to 15 s.

Examples :

If a Vickers microhardness of 310 to 320 has been determined by using a test force of 0,981 N (0,1 kgf) applied during 10 to 15 s, the result shall be written as 310-320 HV 01.

The symbol 67-70 HK 0,025/20 signifies a Knoop hardness measured with a test force of 0,025 × 9,81 N = 0,245 N (0,025 kgf) applied for 20 s.

9 Test report

The report of the microhardness test results shall include :

a) the number and title of this International Standard;

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

c) whether the measurement was carried out on a cross-section or normal to the surface;

d) any unusual features noted during the measurement, such as temperatures other than normal temperatures (see 7.5), longer duration of force application than normal (see 6.3), etc.;

e) any operation regarded as optional or not included in this International Standard or in the International Standards to which references are made.

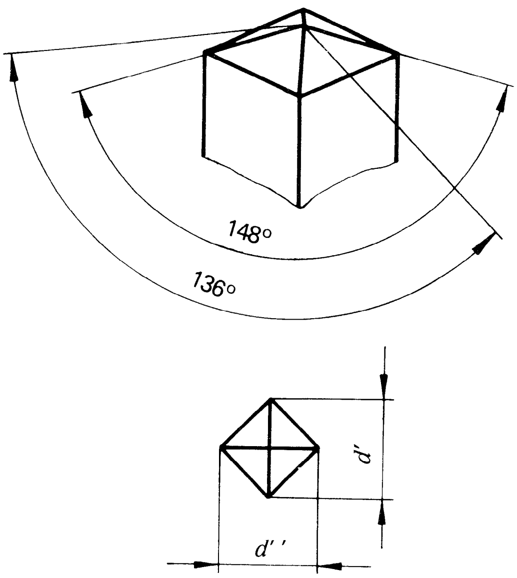

Figure 1 — Vickers indenter

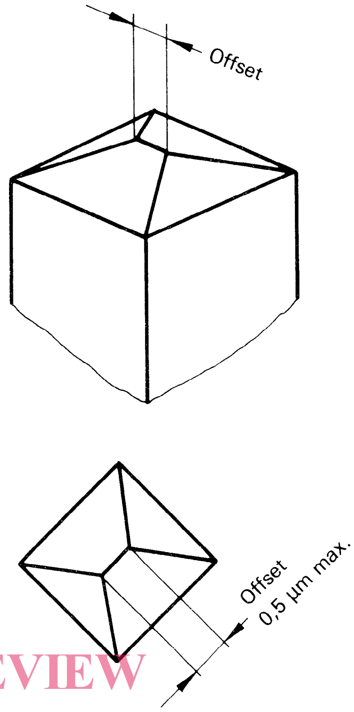

Figure 2 — Vickers indenter offset

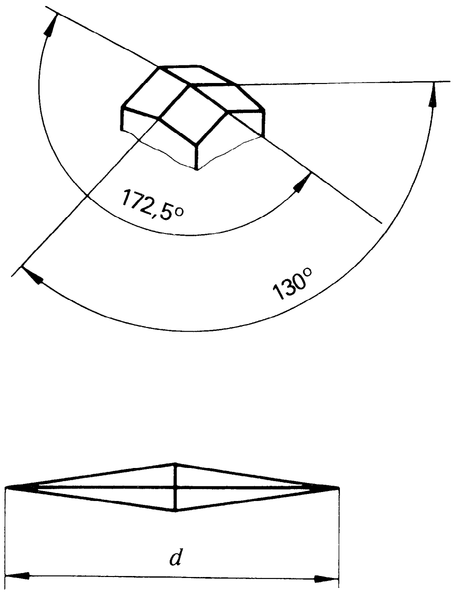

Figure 3 — Knoop indenter

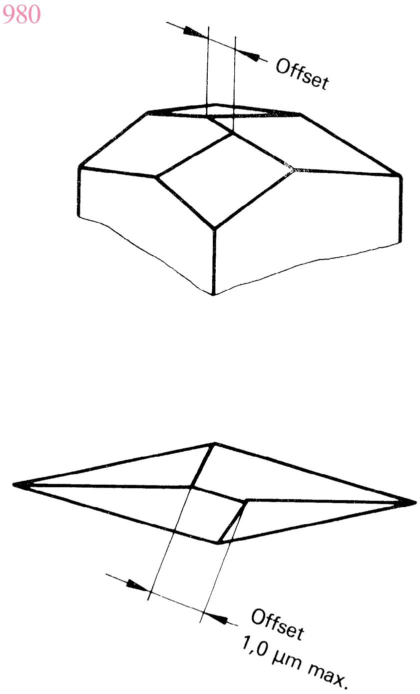

Figure 4 — Knoop indenter offset

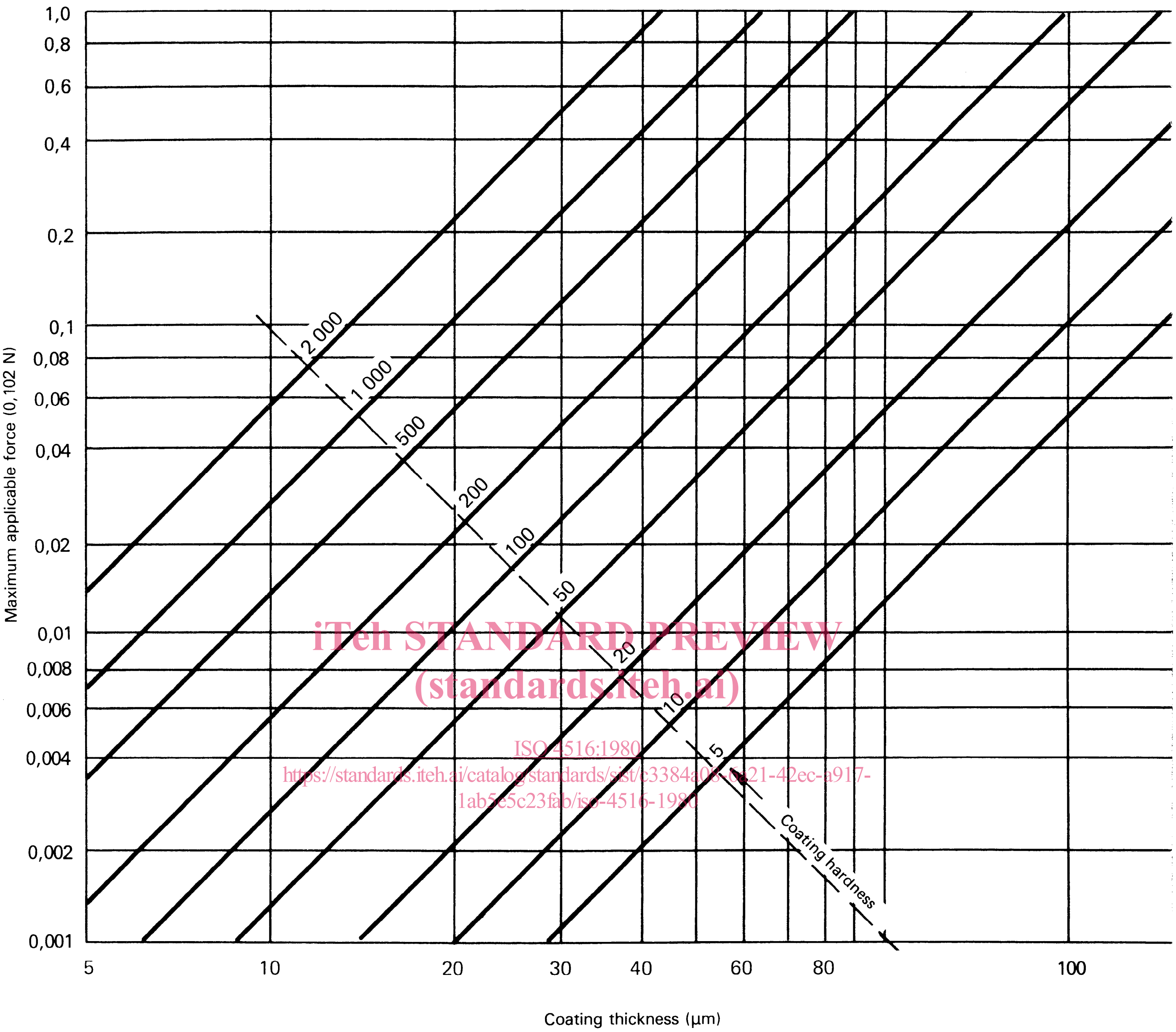

Figure 5 — Relation of maximum applicable force to coating thickness for the Vickers indenter.