
Hardmetals — Knoop hardness test

Métaux-durs — Essai de dureté 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 2.

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

ISO 22394 was prepared by Technical Committee ISO/TC 119, *Powder metallurgy*, Subcommittee SC 4, *Sampling and testing methods for hardmetals*.

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Introduction

Many metallurgical problems require the determination of hardness over very small areas. The special shape of the Knoop indenter makes it possible to place indentations much closer together than with a square Vickers indentation, e.g. to measure a steep hardness gradient. For a given long diagonal length, the depth and area of the Knoop indentation are known to be only 15 % of what they would be for a Vickers indentation with the same diagonal length.

Both Vickers and Knoop hardness tests were performed for a range of hardmetals, in order to investigate whether a specific International Standard is really required and if it compensates the limitations of the Vickers hardness test currently used. Knoop hardness tests were carried out independently in three institutes (KATS, Jinil Co., Seoul University) over a period of four months. The results of this test (see Annex A) show that this new International Standard regarding the Knoop hardness test is necessary.

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Hardmetals — Knoop hardness test

1 Scope

This International Standard specifies the method of the Knoop hardness test for hardmetals.

2 Normative references

The following referenced documents are indispensable for the application 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*

3 Principle

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Forcing a diamond indenter, in the form of a rhombic-based pyramid with specified angles between opposite faces at the vertex, into the surface of a test piece and measuring the long diagonal of the indentation left in the surface after removal of the test force, F , in accordance with ISO 4545-1.

4 Symbols and designations

4.1 The Knoop hardness, HK, is given by the quotient obtained by dividing the test force F by the projected area A_p of the indentation as represented by numerical value Equation (1):

$$HK = \frac{1}{g} \cdot \frac{F}{A_p} = 0,102 \cdot \frac{F}{c \cdot d^2} = 0,102 \cdot \frac{F}{\left(\frac{\tan\left(\frac{\beta}{2}\right)}{2 \cdot \tan\left(\frac{\alpha}{2}\right)} \right) \cdot d^2} = 0,102 \cdot \frac{F}{0,07028 \cdot d^2} = 1,451 \cdot \frac{F}{d^2} \quad (1)$$

where

g is the acceleration due to gravity, in metres per second squared (m/s^2), with a constant of 9,806 65;

F is the test force, in newtons(N);

A_p is the projected area of the permanent indentation, in square millimetres (mm^2);

5 Apparatus

5.1 The testing machine, in accordance with ISO 4545-2, should meet the following requirements:

- a) the indentation force should be calibrated to be within 1 % of the nominal value;
- b) the indenter should be vertically lowered on the surface of the test specimen at a rate lower than 0,1 mm/s.

5.2 Indenter, a diamond in the shape of a rhombic-based pyramid, as specified in ISO 4545-2.

5.3 Measuring device

5.3.1 The measuring device shall permit an estimation of the diagonal of the indentation to within $\pm 0,2 \mu\text{m}$.

5.3.2 The device for measuring the diagonal of the indentation shall be calibrated against an accurately ruled line scale (stage micrometer) or device of equivalent accuracy. The errors of the line scale shall be known within an uncertainty of $0,2 \mu\text{m}$.

5.3.3 The verification of the measuring device shall be carried out in accordance with ISO 4545-2.

6 Test pieces

6.1 The thickness of the layer removed from the surface of the test piece shall be not less than 0,2 mm.

The test shall be carried out on a surface which is free from foreign matter and, in particular, completely free from lubricants. The test surface shall be ground and polished with fine diamond cloths in order to avoid experimental difficulties and errors owing to rough surface.

Preparation shall be carried out in such a way that any alteration of the surface hardness, for example, due to heat or cold working, is minimized.

In determining the hardness of a test piece with a curved surface, a flat surface shall be provided on the test piece on which the hardness test is carried out.

The test-piece surface and support surface shall be parallel to obtain symmetrical indentations.

6.2 The prepared test piece shall be at least 10 times as thick as the indentation depth expected under the chosen force.

7 Procedure

7.1 The test is normally carried out at a temperature of $23 \text{ }^\circ\text{C} \pm 5 \text{ }^\circ\text{C}$. If the test is carried out at a temperature outside this range, it shall be noted in the test report.

7.2 The test force shall be lower than 490,3 N (50 kgf).

7.3 The test piece shall be placed on a rigid support. The support surfaces shall be clean and free from foreign matter (scale, oil, dirt, etc.). It is important that the test piece be firmly supported so that displacement cannot occur during the test. Focus the measuring microscope so that the specimen surface can be observed.

7.4 The indenter shall approach the surface within the velocity range of $15 \mu\text{m/s}$ to $70 \mu\text{m/s}$.

The time from the initial application of force until the full test force is reached shall not be less than 5 s nor greater than 10 s. The duration of the test force shall be from 10 s to 15 s.

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

7.6 If possible, at least three hardness determinations shall be made on a test piece.

7.7 The distance between the limit of any indentation and the edge of the test piece shall be at least 2 times the short diagonal of the indentation.

The distance between the limits of two adjacent indentations shall be at least 2,5 times the short diagonal of the indentation. If two adjacent indentations differ in size, the spacing shall be based on the short diagonal of the larger indentation.

7.8 The satisfactory condition of the indenter shall be verified frequently. Any irregularities in the shape of the indentation may show the poor condition of the indenter. If the examination of the indenter confirms this, then the test shall be rejected and the indenter renewed.

7.9 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, 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.

7.10 Attention is drawn to ISO 4545-4, which contains conversion tables for use in tests made on flat surfaces.

8 Expression of results

Report the arithmetical mean of the hardness values obtained, rounded to the nearest 10 HK.

9 Test report

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The test report shall include the following information:

- a) a reference to this International Standard; [ISO 22394:2010](https://standards.iteh.ai/catalog/standards/sist/642fb4e7-bc7d-4d22-bcc5-5e831e7e17d/iso-22394-2010)
- b) all details necessary for identification of the test sample;
- c) the result obtained;
- d) all options not specified by this International Standard, or regarded as optional;
- e) details of any occurrence which may have affected the result.

10 Significance

Knoop hardness measurements can be useful in studies of hardness gradients over small regions. However, the values should not be directly compared with Vickers hardness values. This is an ongoing subject of research (Reference [3] in the Bibliography) and recommendations cannot yet be given as to good practice for comparison values.

Annex A (informative)

Investigation regarding the demand of an International Standard for a Knoop hardness test for hardmetals

A.1 Test procedure

Hardmetal samples employed in this test are commercially available insert materials, the compositions of which are listed in Table A.1. The test loads applied are 1 kg to 50 kg. Tests to examine the distance effect (see Figures A.1 to A.4) were performed exclusively under 1 kg and 30 kg.

Table A.1 — Composition of the test pieces used

Test-piece number	Composition	Hardness (HRA)
1	WC-6%Co ^a	92 to 92,6
2	WC-12%Co ^a	90 to 90,8
3	TiCN(WC/MoC/TaC)-17%(Ni/Co) ^a	92,4 to 93
^a Percentages are mass fractions.		

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Both Vickers and Knoop hardness tests were performed in order to compare the two tests employed in hardmetals. Both tests were made at various loads of 5 kg to 50 kg, and 7 readings were taken under a given condition. Indentations were separated at a sufficient distance so as not to be influenced by each other.

Knoop hardness values were measured while increasing the distance from the adjacent indentation as well as while increasing the distance from the edge of the sample. The distance is expressed in terms of number of times the short diagonal of the impression (see Figures A.1 to A.4).

A.2 Test results

A.2.1 Comparison of Vickers and Knoop values

Hardness values in Table A.2 marked with the footnote "a" represent when a crack was formed around the impression made by the indenter. Whereas no crack was found in the Knoop sample, most of the Vickers sample reveals crack formation around the indentation, regardless of the loads applied. The micrographs in Figures A.5 to A.7 are taken under various situations, showing the formation of a hair crack around the Vickers indentation.