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Hardmetals — Knoop hardness test

Métaux-durs — Essai de dureté Knoop

Reference number
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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*.

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.

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

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):

$$\text{HK} = \frac{1}{g} \cdot \frac{F}{A_\text{p}} = 0{,}102 \cdot \frac{F}{c \cdot d^2} = 0{,}102 \cdot \frac{F}{\left(\dfrac{\tan\left(\dfrac{\beta}{2}\right)}{2 \cdot \tan\left(\dfrac{\alpha}{2}\right)}\right) \cdot d^2} = 0{,}102 \cdot \frac{F}{0{,}07028 \cdot d^2} = 1{,}451 \cdot \frac{F}{d^2} \qquad (1)$$

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);

c is an indenter constant which equals $\dfrac{\tan\left(\dfrac{\beta}{2}\right)}{2\cdot\tan\left(\dfrac{\alpha}{2}\right)}$, ideally with a constant of c = 0,0702 8;

d is the length of the long indentation diagonal, in millimetres (mm) (see Figure 1);

α is the angle with a value of 172,5° (see Figure 2);

β is the angle with a value of 130° (see Figure 2).

The indentation of d is assumed to be a rhombic-based pyramid with a base area as shown in Figure 1 and having, at the vertex, the same angles as the indenter (see Figure 2).

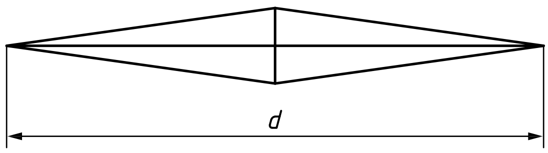


Figure 1 — Projected area of the indentation produced by the Knoop indenter

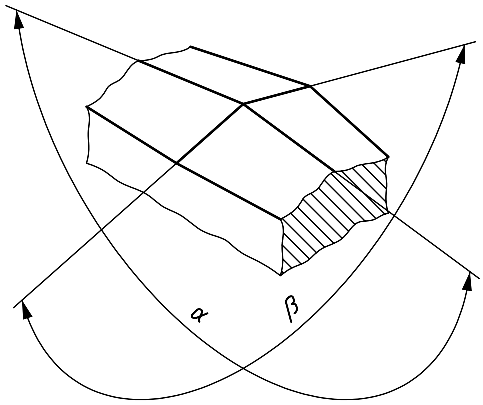


Figure 2 — Knoop indenter

4.2 The Knoop hardness is denoted by the symbol HK preceded by the hardness value and supplemented by a number representing the test force.

EXAMPLE 1 Use of the SI unit (GPa):

— 10 GPa HK 9,807 = Knoop hardness of 10 GPa, determined with a test force of 9,807 N (1 kgf).

EXAMPLE 2 Use of the Knoop hardness number (no units specified):

— 1 000 HK 1 = Knoop hardness number of 1 000, determined with a test force of 9,807 N (1 kgf).

EXAMPLE 3 The duration of loading, in seconds (s), if different from the time specified in 7.4:

— 1 000 HK 1 = Knoop hardness number of 1 000, determined with a test force of 9,807 N (1 kgf) applied for 10 s to 15 s;

— 1 000 HK 1/20 = Knoop hardness number of 1 000, determined with a test force of 9,807 N (1 kgf) applied for 20 s.

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 ±0,2 μm.

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 μm.

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 °C ± 5 °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 μm/s to 70 μm/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

The test report shall include the following information:

a) a reference to this International Standard;

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.		

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

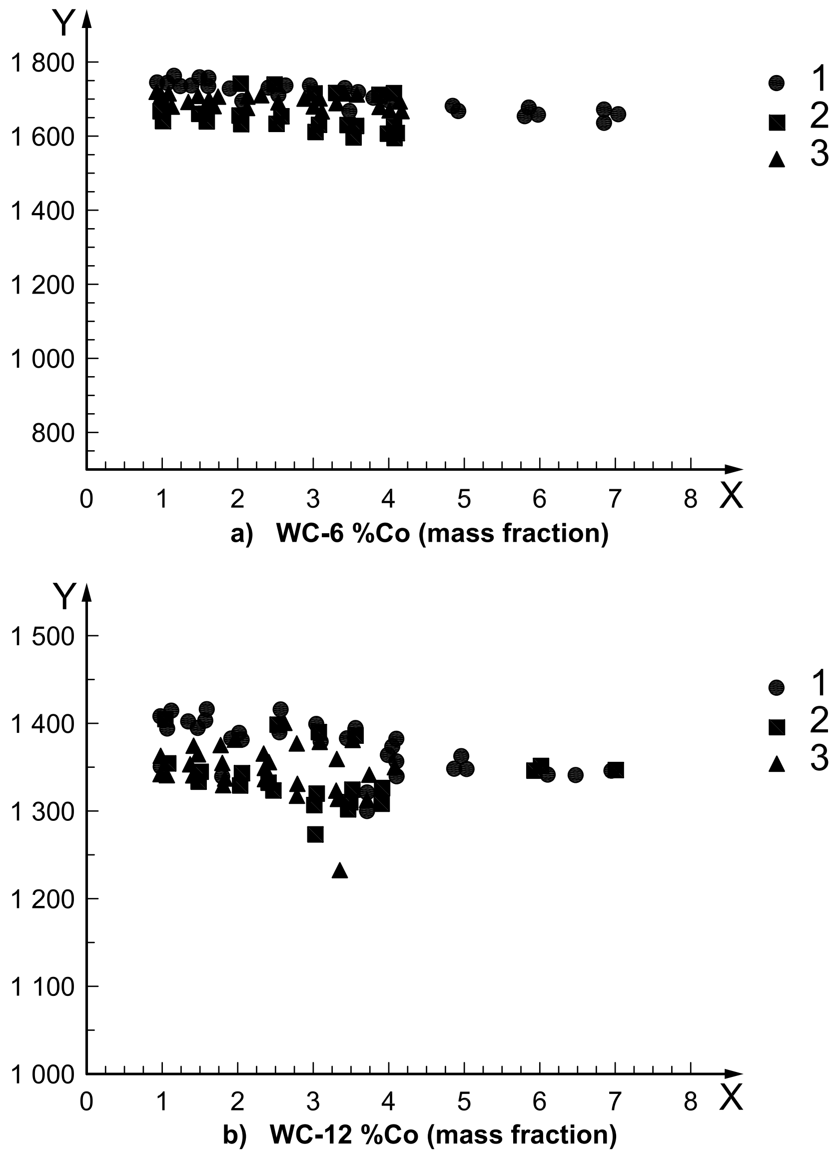
Hardness values in Table A.2 marked with the footnote "a" represent when a crack was formed around the impression made by the indenter. Whereas no crack was found in the Knoop sample, most of the Vickers sample reveals crack formation around the indentation, regardless of the loads applied. The micrographs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

Table A.2 — List of Vickers and Knoop hardness values

Institute	Composition	Load kg	Vickers hardness value	Knoop hardness value
A	WC-6 %Co[b]	5	1740 1740 1740 1693 1740 1693 1740	1693 1677 1726 1744 1677 1677 1677
		10	1748[a] 1714[a] 1748[a] 1748[a] 1748[a] 1748[a] 1714[a]	1692 1669 1669 1680 1680 1680 1680
		30	1736[a] 1736[a] 1755[a] 1755[a] 1736[a] 1736[a] 1736[a]	1661 1667 1661 1654 1661 1667 1674
		50	1722[a] 1737[a] 1722[a] 1722[a] 1722[a] 1722[a] 1737[a]	1658 1590[a] 1684 1643 1668 1658 1663
	WC-12 %Co[b]	5	1379 1346 1346 1379 1379 1379 1379	1381 1369 1357 1369 1393 1357 1369
		10	1378 1354 1378 1378 1354 1378 1354	1355 1355 1364 1347 1355 1347 1355
		30	1377[a] 1391[a] 1377[a] 1377[a] 1363 1377 1363	1323 1318 1323 1332 1342 1323 1342
		50	1371[a] 1350[a] 1371[a] 1371[a] 1361 1371 1382	1317 1317 1331 1331 1317 1331 1331
B	WC-6 %Co[b]	5	1605 1564 1605 1605 1648 1524 1605	1629 1598 1613 1613 1598 1613 1644
		10	1650 1619 1619 1650 1590 1650 1619	1602 1602 1613 1602 1613 1592 1602
		30	1625 1796 1755 1775 1775 1755 1736	1591 1609 1609 1597 1603 1609 1609
		50	1632 1692 1614 1662 1661 1691 1682	1571 1590 1594 1604 1604 1614 1614
	WC-12 %Co[b]	5	1314 1314 1346 1314 1346 1346 1283	1306 1298 1307 1314 1314 1306 1299
		10	1309 1288 1309 1309 1266 1309 1309	1310 1305 1304 1311 1316 1300 1304
		30	1363 1463 1463 1463 1405 1363 1391	1273 1287 1300 1278 1278 1296 1287
		50	1340 1426 1393 1414 1414 1404 1414	1272 1310 1303 1306 1299 1296 1292
C	WC-6 %Co[b]	5	1788[a] 1788[a] 1764[a] 1764[a] 1788[a] 1839[a] 1813[a]	1852 1861 1824 1752 1824 1752 1806
		10	1731[a] 1748[a] 1817[a] 1800[a] 1854[a] 1800[a] 1800[a]	1710 1746 1692 1721 1721 1710 1764
		30	1755[a] 1717[a] 1775[a] 1765[a] 1755[a] 1765[a] 1736[a]	1664 1680 1654 1641 1667 1674 1661
		50	1752[a] 1722[a] 1730[a] 1737[a] 1745[a] 1730[a] 1708[a]	1585 1648 1653 1653 1631 1648 1604
	WC-12 %Co[b]	5	1504 1524 1448 1485 1504 1504 1524	1418 1405 1470 1418 1418 1450 1424
		10	1427 1439 1414 1452 1452 1465 1427	1398 1390 1398 1398 1403 1377 1416
		30	1412[a] 1412[a] 1412[a] 1419[a] 1405[a] 1426[a] 1405[a]	1325 1347 1332 1321 1342 1337 1349
		50	1398[a] 1414[a] 1409[a] 1409[a] 1404[a] 1409[a] 1393[a]	1299 1335 1317 1324 1310 1324 1308

[a] Crack formation around indentation.

[b] Percentages are mass fractions.



Key

X distance between the limits of two adjacent indentations (times the short diagonal)

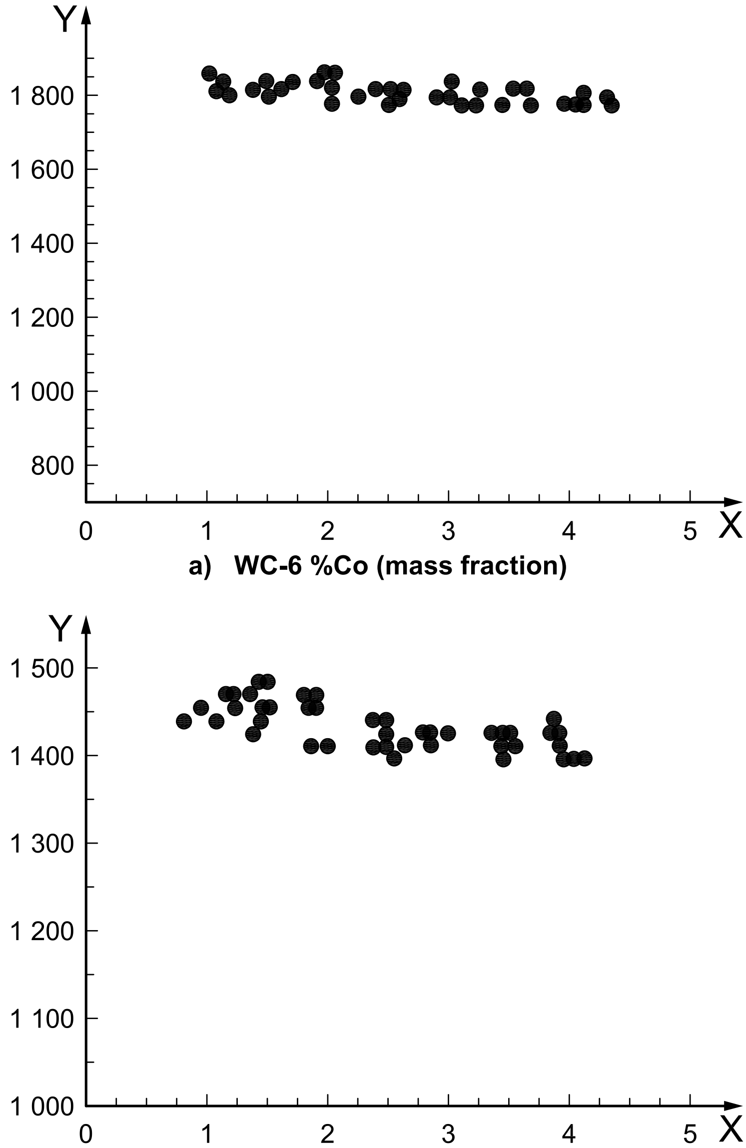
Y Knoop hardness (HK 30)

1 institute A

2 institute B

3 institute C

Figure A.1 — Knoop hardness values (HK 30) at the various distances between the limits of two adjacent indentations



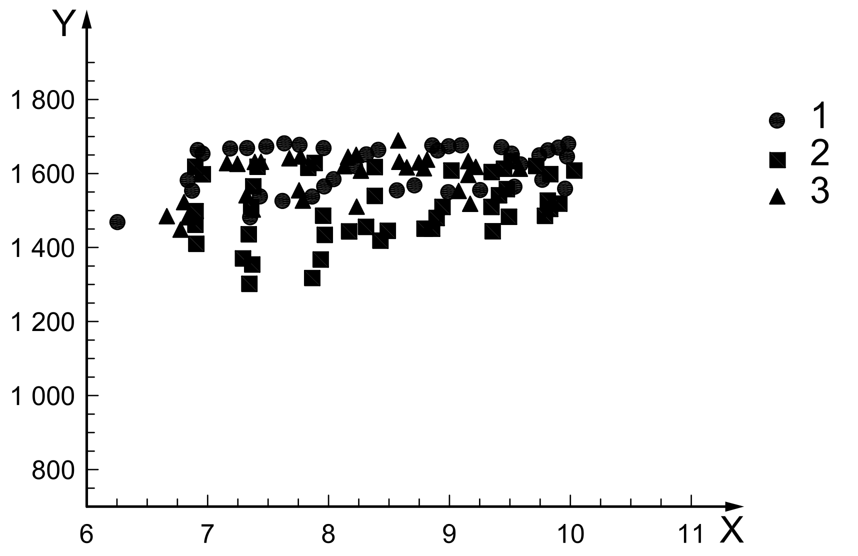
b) WC-12 %Co (mass fraction)

Key

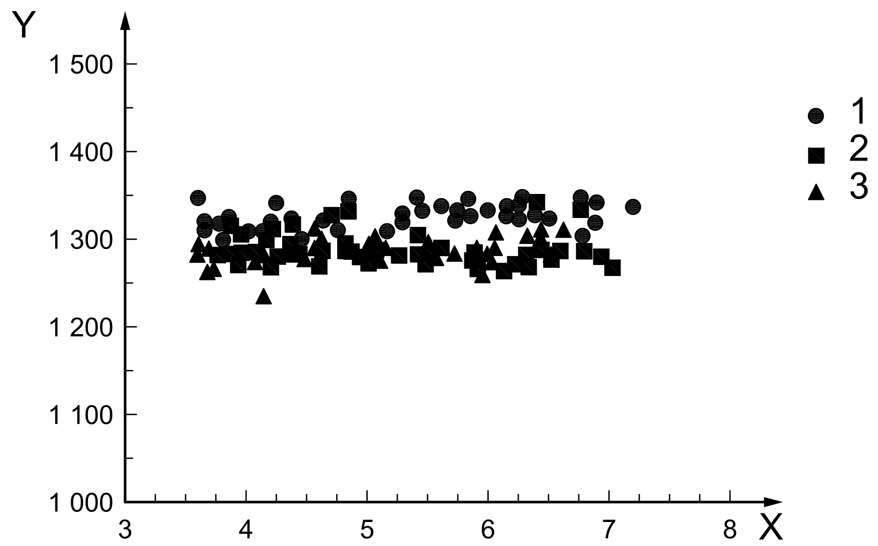
X distance between the limits of two adjacent indentations (times the short diagonal)

Y Knoop hardness (HK 1)

Figure A.2 — Knoop hardness values (HK 1) at the various distances between the limits of two adjacent indentations



a) WC-6 %Co (mass fraction)



b) WC-12 %Co (mass fraction)

Key

X distance between the limit of indentation and the edge of the test piece (times the short diagonal)

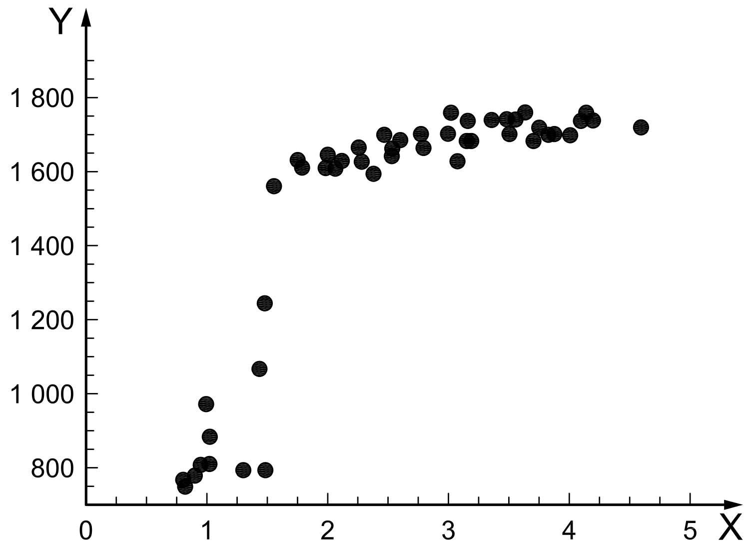
Y Knoop hardness (HK 30)

1 institute A

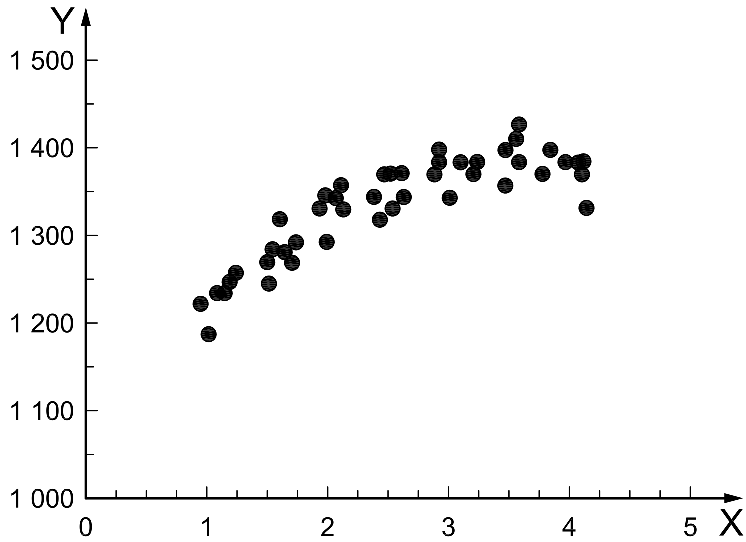
2 institute B

3 institute C

Figure A.3 — Knoop hardness values (HK 30) at the various distances between the limit of indentation and the edge of the test piece



a) WC-6 %Co (mass fraction)



b) WC-12 %Co (mass fraction)

Key

X distance between the limit of indentation and the edge of the test piece (times the short diagonal)

Y Knoop hardness (HK 1)

Figure A.4 — Knoop hardness values (HK 1) at the various distances between the limit of indentation and the edge of the test piece

A.2.2 Proximity effects – Adjacent indents

Knoop hardness values under the applied load of 30 kg (HK 30) are plotted against the distance between two adjacent indentations in Figure A.1.

In the case of WC-6 %Co [see Figure A.1 a)], the Knoop hardness value has a tendency to gradually decrease when increasing the distance between the indentations. However, the result of institute A shows that the decreasing tendency may be substantially mitigated (or disappears) when further increasing the distance over 4 times the short diagonal. WC-12 %Co [see Figure A.1 b)] shows the same trend as Figure A.1 a), except for the fact that Figure A.1 a) reveals wider scatter of data points.

Data shown in Figure A.2 were obtained under the applied load of 1 kg (HK 1). No crack was found around the indentation, even near the edge of the specimen at this load, while at 30 kg the region near the edge was fractured by the impression made by the indenter as explained in A.2.3. The longitudinal diagonal of the Knoop indentation at this load has such a sufficient length that it could be easily measured through the microscope without impairing accuracy, being 90 µm and 101 µm for WC-6 %Co and WC-12 %Co (mass fractions), respectively. The length corresponds to the diagonal of the Vickers indentation under the applied load of 7 kg to 8 kg. Knoop hardness testing has an advantage over Vickers hardness testing in that a much smaller indentation is allowed for a given accuracy of diagonal measurement. In order to obtain a constant Knoop hardness value, Figures A.1 and A.2 suggest that the distance between the adjacent indentations should be at least 4 times the short diagonal.

A.2.3 Proximity effects – Closeness to edges

Figure A.3 gives Knoop hardness values (HK 30) when increasing the distance from the edge of the test piece. The hardness values are nearly constant, and not dependent on the distance covered in Figure A.3.

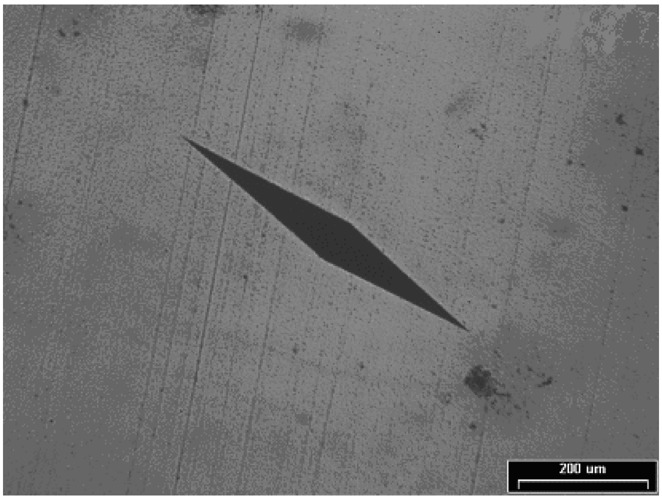
The hardness values could not be measured within a certain region from the edge of the specimen because of local fracture in this brittle hardmetal, brought about by the impression made by the indenter. The region is extended to 7 times the short diagonal in the case of WC-6 %Co [see Figure A.3 a)], whereas it is 3 times the short diagonal in the case of WC-12 %Co [see Figure A.3 b)]. Considering the brittleness of the specimen, the load applied seems to be too high for proper evaluation. Lighter loads should be applied for a more critical assessment.

Figure A.4 shows the results obtained under the applied load of 1 kg (HK 1). The Knoop hardness value steeply increases when increasing the distance from the edge of the specimen until a constant value is attained. In the region at a distance away from the edge of the specimen of more than 3 times the short diagonal of the indentation, the hardness value seems to be nearly constant.

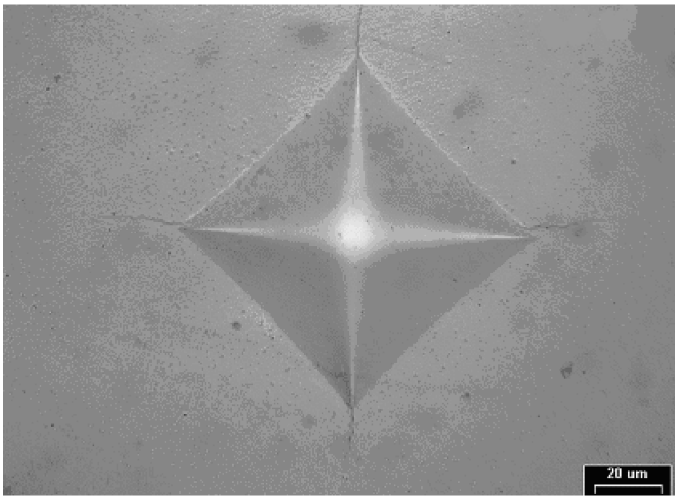
A.3 Summary

The establishment of this International Standard was necessary to compensate for the limitation of the Vickers hardness test (crack formation around the Vickers indentation).

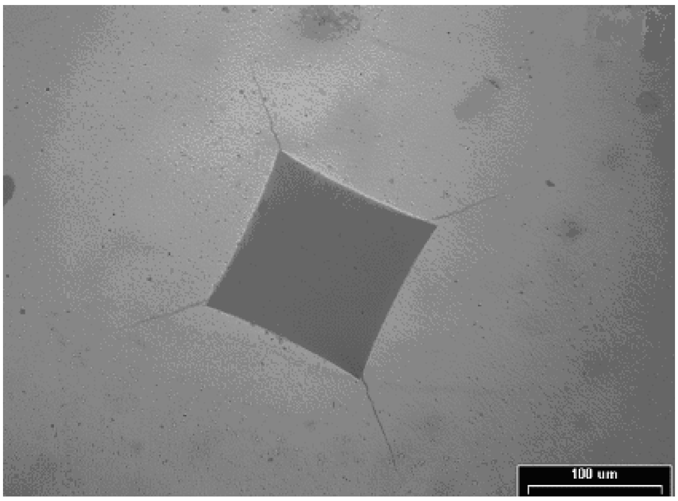
The minimum distance between the limits of two adjacent indentations shall be at least 4 times the short diagonal of the indentation. The minimum distance between the limit of any indentation and the edge of the test piece shall be at least 3 times the short diagonal of the indentation.



a) HK 30, 30 kg

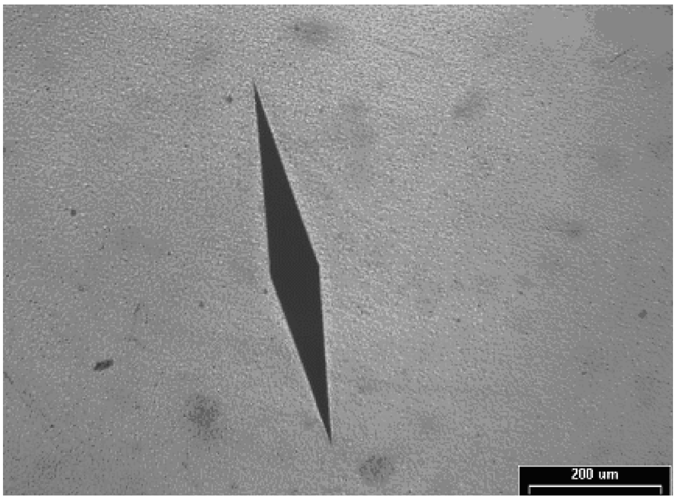


b) HV 10, 10 kg

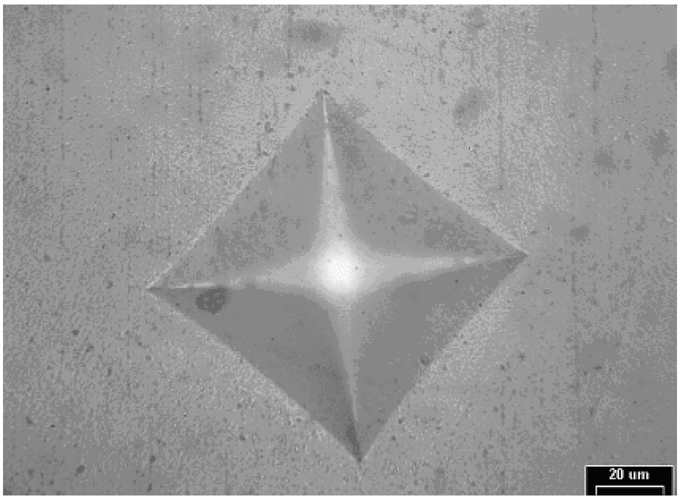


c) HV 30, 30 kg

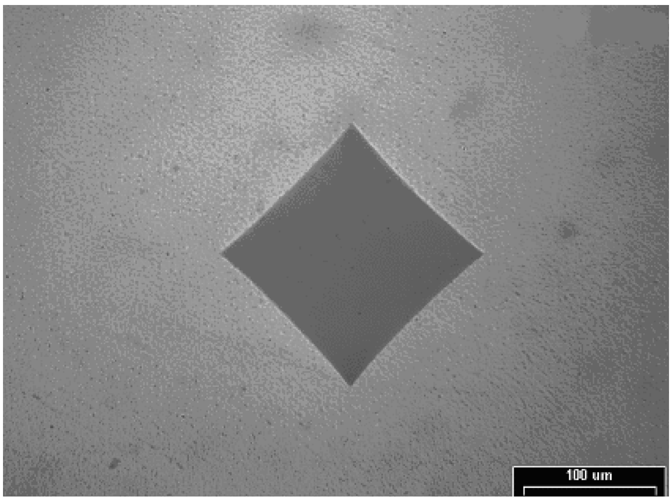
Figure A.5 — Test piece 1 – WC-6 %Co (mass fraction)



a) HK 30, 30 kg

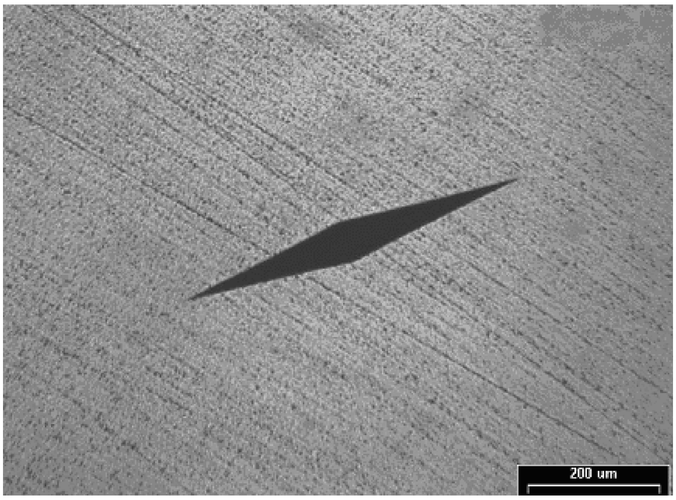


b) HV 10, 10 kg

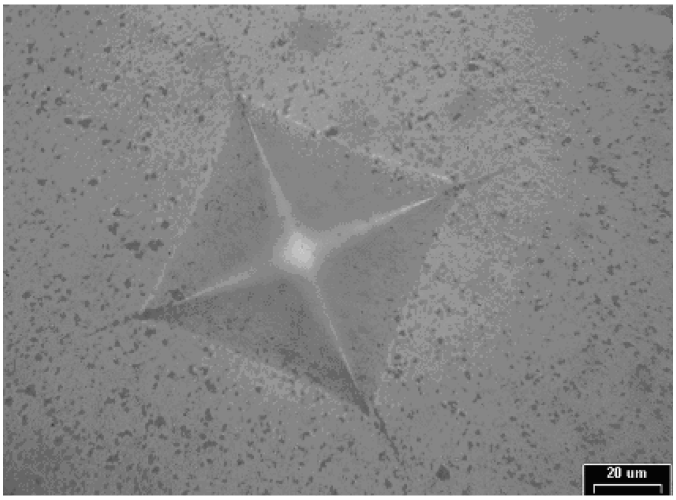


c) HV 30, 30 kg

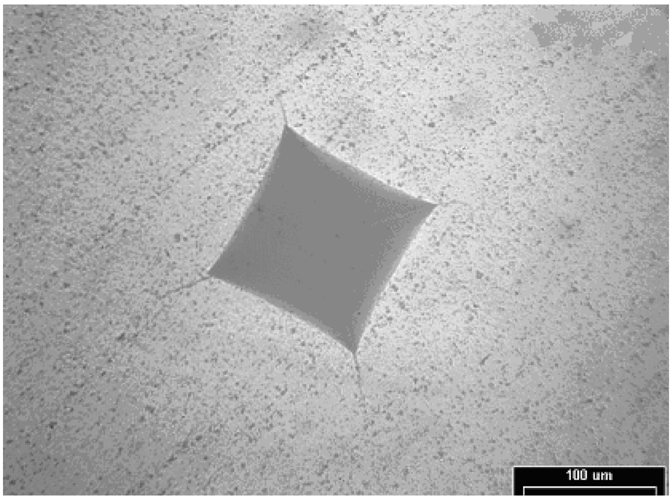
Figure A.6 — Test piece 2 – WC-12 %Co (mass fraction)



a) HK 30, 30 kg



b) HV 10, 10 kg



c) HV 30, 30 kg

Figure A.7 — Test piece 3 – TiCN(WC/MoC/TaC)-17 %(Ni/Co) (mass fraction)

Annex B
(informative)

Comparison of important test procedures in Vickers and Knoop hardness tests

Table B.1 — Comparison of important test procedures in Vickers and Knoop hardness tests

Test parameters		**Hardness test method** **Metallic material – Vickers** (see ISO 6507-1)	**Hardmetal – Vickers** (see ISO 3878)	**Metallic material – Knoop** (see ISO 4545-1)	**Hardmetal – Knoop** (see ISO 22394)
Surface layer		—	The layer removed is not less than 0,2 mm	—	The layer removed is not less than 0,2 mm
Test force		0,098 07 N to 980,7 N	9,807 N to 490,3 N (294,2 N preferred)	0,098 07 N to 9,807 N	> 490,3 N
Application of the test force	Time to the full test force	2 s to 8 s, for low-force hardness and microhardness tests > 10 s	2 s to 8 s	> 10 s	5 s to 10 s
	Approach velocity	> 0,2 mm/s	—	15 µm/s to 70 µm/s	> 70 µm/s
	Duration of test force	10 s to 15 s	10 s to 15 s	10 s to 15 s	10 s
Indentation	Edge of the test piece	The distance between the centres of any indentation and the edge of the test piece: — at least 2,5 times the mean diagonal (steel, Cu-alloys); — at least 3 times the mean diagonal (light metals, Pb-alloys, Sn-alloys).	— at least 2,5 times the mean diagonal.	The distance between the limit of any indentation and the edge of the test piece: — at least 2,5 times the short diagonal (steel, Cu-alloys); — at least 3 times the short diagonal (light metals, Pb-alloys, Sn-alloys).	— at least the same as the long diagonal.
Indentation	Adjacent indentation	The distance between the centres of two adjacent indentations: — at least 3 times the mean diagonal (steel, Cu-alloys); — at least 6 times the mean diagonal (light metals, Pb-alloys, Sn-alloys); based on larger indentation.	— at least 3 times the mean diagonal; based on larger indentation.	The distance between the limits of two adjacent indentations: — at least 3 times the short diagonal (steel, Cu-alloys); — at least 6 times the short diagonal (light metals, Pb-alloys, Sn-alloys); based on larger indentation.	— at least 2 times the long diagonal; based on larger indentation.

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

[2] ISO 3878, *Hardmetals — Vickers hardness test*

[3] CHICOT, D., MERCIER, D., ROUDET, F., SILVA, K., STAIA, M.H., LESAGE, J., Comparison of instrumented Knoop and Vickers hardness measurements on various soft materials and hard ceramics. *Journal of the European Ceramic Society* **27** (2007), pp. 1905-1911

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