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Metallic materials — Knoop hardness test —

Part 3: Calibration of reference blocks

Matériaux métalliques — Essai de dureté Knoop —

Partie 3: Étalonnage des blocs de référence

Reference number
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Foreword

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The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

For an explanation on the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT) see the following URL: www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 164, *Mechanical testing of metals*, Subcommittee SC 3, *Hardness testing*.

This second edition cancels and replaces the third edition (ISO 4545-3:2005), which has been technically revised.

Significant technical changes from the previous edition of this document include:

— the requirements for the maximum test surface area of the reference block have been added;

— the requirements for the maximum uncertainty of the line intervals on the stage micrometer have been revised;

— the requirements for the calibration and verification of the measuring system have been revised, as per ISO 4545-2;

— the requirements for the uniformity of the reference block hardness have been revised to account for different numbers of calibration indentations;

— the timing requirements for the approach velocity and the time duration at maximum test force have been revised to indicate a target time value;

— Annex B has been revised

A list of all parts in the ISO 4545 series can be found on the ISO website.

Metallic materials — Knoop hardness test —

Part 3: Calibration of reference blocks

1 Scope

This document specifies the method for the calibration of reference blocks to be used for the indirect verification of Knoop hardness testing machines as specified in ISO 4545-2.

The method is applicable only for indentations with long diagonals ≥0,020 mm.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements 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 376:2011, *Metallic materials — Calibration of force-proving instruments used for the verification of uniaxial testing machines*

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*

3 Terms and definitions

No terms and definitions are listed in this document.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— IEC Electropedia: available at http://www.electropedia.org/

— ISO Online browsing platform: available at http://www.iso.org/obp

4 Manufacture of the reference block

4.1 General

The block shall be specially manufactured for use as a hardness-reference block using a manufacturing process that will give the necessary homogeneity, stability of structure and uniformity of surface hardness.

4.2 Thickness

Each metal block to be calibrated shall be of a thickness not less than 5 mm.

4.3 Test surface area

The test surface area of the reference block shall not exceed 40 cm^2.

4.4 Magnetism

The reference blocks shall be free of magnetism. It is recommended that the manufacturer ensure that the blocks, if made of steel, have been demagnetized at the end of the manufacturing process (before calibration).

4.5 Flatness and parallelism

The maximum deviation in flatness of the test and support surfaces shall not exceed 0,005 mm. The maximum error in parallelism shall not exceed 0,010 mm in 50 mm.

4.6 Surface roughness

The test surface shall be free from scratches that interfere with the measurement of the indentations. The test surface roughness, *Ra*, shall not exceed 0,05 µm[1]. The bottom support surface shall be a finely ground finish or better.

4.7 Prevention of the regrind of the test surface

To verify that no material is subsequently removed from the reference block, the thickness at the time of calibration shall be marked on the reference block to the nearest 0,01 mm, or an identifying mark shall be made on the test surface [see 8.2 e)].

5 Calibration machine

5.1 General

In addition to fulfilling the general requirements specified in ISO 4545-2, the calibration machine shall also meet the requirements given in 5.2 to 5.6.

NOTE The criteria specified in this document for the performance of the calibration machine have been developed and refined over a significant period of time. When determining a specific tolerance that the machine needs to meet, the uncertainty associated with the use of measuring equipment and/or reference standards has been incorporated within this tolerance, and it would therefore be inappropriate to make any further allowance for this uncertainty by, for example, reducing the tolerance by the measurement uncertainty. This applies to all measurements made when performing a direct verification of the calibration machine.

5.2 Direct verification

The calibration machine shall be directly verified in intervals not exceeding 12 months.

Direct verification involves

— calibration of the test force,

— verification of the indenter,

— calibration and verification of the diagonal measuring system, and

— verification of the testing cycle, and if not possible, at least the force versus time behaviour.

5.3 Traceability of verification instruments

The instruments used for verification and calibration shall be traceable to national standards.

5.4 Test force

Each test force shall be verified at three different positions of the plunger, spaced at approximately equal increments covering the limits of travel used during testing. At each position, the force shall be measured three times using an elastic proving device, according to ISO 376:2011, Class 0,5 or better, or by another method having the same or better accuracy. Each measurement shall agree with the nominal value to within ±0,5 %.

5.5 Indenter

The indenter shall comply with ISO 4545-2 and meet the following requirements:

a) the four faces of the rhombic-based diamond pyramid shall be highly polished, free from surface defects, and flat within 0,000 3 mm;

b) the angle β (see ISO 4545-1), between opposite edges at the vertex of the diamond pyramid, shall be (130 ± 0,1)°;

c) the angle between the axis of the diamond pyramid and the axis of the indenter holder (normal to the seating surface) shall not exceed 0,3°;

d) the point of the diamond indenter shall be examined with a high-power measuring microscope or preferably with an interference microscope. If the four faces do not meet at a point, the line of junction, as described in ISO 4545-2, between opposite faces shall be less than 0,000 3 mm.

A valid calibration certificate shall exist which confirms the geometrical deviations of the indenter.

5.6 Diagonal measurement system

The scale of the diagonal measuring system shall be graduated to permit estimation of the diagonals of the indentation to the greater of 0,000 15 mm or 0,25 % of the length measured.

The system for measuring the long diagonal of the indentation shall be verified at each magnification and for each incorporated line scale to be used in two perpendicular measurement axes (if applicable), by performing measurements on an accurately ruled stage micrometer. Measurements shall be made at a minimum of five evenly spaced intervals, arranged centrally in the field of view, covering each working range.

The maximum expanded uncertainty of the distance between the line intervals on the stage micrometer shall be 0,000 2 mm or 0,04 %, whichever is greater.

Three measurements shall be made at each of the evenly spaced intervals. The maximum permissible error of each of the three diagonal measurements at each interval shall be the greater of 0,000 3 mm or 0,5 % of the length measured.

NOTE A helpful technique for adjusting optical systems that have Köhler illumination is given in Annex A.

6 Calibration procedure

The reference blocks shall be calibrated by a calibration machine as specified in Clause 5, at a temperature of (23 ± 5) °C, using the general procedure specified in ISO 4545-1.

During calibration, the thermal drift should not exceed 1 °C.

Bring the indenter into contact with the test surface and apply the test force in a direction perpendicular to the surface, without shock or vibration, until the applied force attains the specified value. The approach velocity of the indenter shall be within 0,015 mm/s to 0,07 mm/s. The time from the initial application of the force until the full test force is reached shall be 7^{+1}_{-1} s. The duration of the test force shall be 14^{+1}_{-1} s.

The maximum allowable vibrational acceleration reaching the machine shall be less than 0,005 g_n [g_n being the standard acceleration due to gravity (g_n = 9,806 65 m/s²)].

7 Number of indentations

On each reference block, a minimum of five reference indentations shall be made, uniformly distributed over the test surface. At least one of the indentations shall be identified as a reference indentation.

To reduce the measurement uncertainty, more than 5 indentations should be made. It is recommended to make 10, 15 or 25 indentations distributed over five locations on the reference block.

8 Uniformity of hardness

8.1 Relative non-uniformity

For each reference block, let H_1, H_2, ..., H_n be the n measured hardness values arranged in increasing order of magnitude corresponding to the measured diagonals d_1, d_2, ..., d_n in decreasing order of magnitude. The average hardness, $\overline{H}$, is calculated according to Formula (1):

$$\overline{H} = \frac{H_1 + H_2 + ... + H_n}{n} \tag{1}$$

The relative non-uniformity r_{rel}, expressed as a percentage of $\overline{H}$, is calculated according to Formula (2):

$$r_{rel} = 100 \times \frac{H_n - H_1}{\overline{H}} \tag{2}$$

The uniformity of the reference block is satisfactory if $d_1 - d_n \leq 0{,}001$ mm. If $d_1 - d_n > 0{,}001$ mm, the uniformity of the reference block is satisfactory, when r_{rel} is less than or equal to the percentages indicated in Table 1.

Table 1 — Maximum permissible non-uniformity

Hardness of block	Maximum permissible value of non-uniformity, r_{rel} %HK		
	HK 0,001 to HK 0,1	> HK 0,1 to HK 0,5	> HK 0,5 to HK 2
100 ≤ HK ≤ 200	16	14	8
200 < HK ≤ 250	10	14	8
250 < HK ≤ 650	8	8	6
HK > 650	6	6	4

8.2 Uncertainty of measurement

The determination of the uncertainty of measurement of hardness-reference blocks is given in Annex B.

9 Marking

Each reference block shall be marked with the following information:

a) arithmetic mean of the hardness values found in the calibration test, for example, 249 HK 1;

b) name or mark of the supplier or manufacturer;

c) serial number;

d) name or mark of the calibrating agency;

e) thickness of the block, or an identifying mark on the test surface (see 4.7);

f) year of calibration, if not indicated in the serial number.

All markings shall be placed on the test surface or on the side of the block. Any mark put on the side of the block shall be the right way up when the test surface is facing up.

10 Calibration certificate

Each delivered reference block shall be accompanied with a document giving at least the following information:

a) a reference to this document, i.e. ISO 4545-2;

b) the serial number of the block;

c) the date of calibration;

d) the arithmetic mean of the hardness values in the format defined in ISO 4545-1, and the value characterizing the non-uniformity of the block;

e) the information about the location of each reference indentation together with the length of the long diagonal.

11 Validity

The reference block is only valid for the scale for which it was calibrated.

The calibration validity should be limited to a duration of five years. Attention is drawn to the fact that, for Al- and Cu-alloys, the calibration validity should be reduced to two to three years.