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Metallic materials — Instrumented indentation test for hardness and materials parameters —

Part 3: Calibration of reference blocks

Matériaux métalliques — Essai de pénétration instrumenté pour la détermination de la dureté et de paramètres des matériaux —

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).

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The committee responsible for this document is ISO/TC 164, *Mechanical testing of metals*, Subcommittee SC 3, *Hardness testing*.

This second edition cancels and replaces the first edition (ISO 14577-3:2002), which has been technically revised.

ISO 14577 consists of the following parts, under the general title *Metallic materials — Instrumented indentation test for hardness and materials parameters*:

— *Part 1: Test method*

— *Part 2: Verification and calibration of testing machines*

— *Part 3: Calibration of reference blocks*

— *Part 4: Test method for metallic and non-metallic coatings*

Introduction

Hardness has typically been defined as the resistance of a material to permanent penetration by another harder material. The results obtained when performing Rockwell, Vickers, and Brinell tests are determined after the test force has been removed. Therefore, the effect of elastic deformation under the indenter has been ignored.

ISO 14577 (all parts) has been prepared to enable the user to evaluate the indentation of materials by considering both the force and displacement during plastic and elastic deformation. By monitoring the complete cycle of increasing and removal of the test force, hardness values equivalent to traditional hardness values can be determined. More significantly, additional properties of the material such as its indentation modulus and elasto-plastic hardness can also be determined. All these values can be calculated without the requirement to measure the indent optically. Furthermore, by a variety of techniques, the instrumented indentation test allows to record hardness and modulus depth profiles within a, probably complex, indentation cycle.

ISO 14577 (all parts) has been written to allow a wide variety of post test data analysis.

Metallic materials — Instrumented indentation test for hardness and materials parameters —

Part 3:
Calibration of reference blocks

1 Scope

This part of ISO 14577 specifies a method for the calibration of reference blocks to use for the indirect verification of testing machines for the instrumented indentation test as specified in ISO 14577-2:2015.

NOTE The reference blocks can be calibrated in accordance with the field of application of the testing machine or with the materials parameters which are being determined.

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 376, *Metallic materials — Calibration of force-proving instruments used for the verification of uniaxial testing machines*

ISO 4287, *Geometrical Product Specifications (GPS) — Surface texture: Profile method — Terms, definitions and surface texture parameters*

ISO 14577-1:2015, *Metallic materials — Instrumented indentation test for hardness and materials parameters — Part 1: Test method*

ISO 14577-2:2015, *Metallic materials — Instrumented indentation test for hardness and materials parameters — Part 2: Verification and calibration of testing machines*

3 Manufacture of reference blocks

3.1 The block shall be specially prepared and the attention of the manufacturer drawn to the requirement to use a manufacturing process that gives the necessary homogeneity, uniformity, and stability of structure.

3.2 Each block being calibrated shall be of a thickness not less than 2 mm for the nano range, not less than 5 mm for the micro, and not less than 16 mm for the macro range.

If it is required by the manufacturing process, the thickness of the reference blocks can be smaller.

3.3 The reference blocks shall be free from magnetic forces. It is recommended that the manufacturers ensure that the blocks, if of steel, are demagnetized at the end of the manufacturing process.

3.4 The reference block shall be constructed such that it can be mounted in the testing machine within the tilt limits specified in ISO 14577-1:2015.

NOTE If the reference block is mounted on its bottom, this condition is valid if the maximum deviation in flatness of the test and support faces does not exceed 5 µm in 50 mm and the maximum error in parallelism does not exceed 10 µm in 50 mm.

3.5 The test surface shall be free from scratches that interfere with the measurement of the indentations. Indentations between scratches are permitted.

For the macro and micro range, the surface roughness, *Ra*, shall not exceed 50 nm and 10 nm respectively for the test surface, and 0,8 µm for the support face, the sampling length, *l*, shall be 0,80 mm (see ISO 4287).

For the nano range, the surface roughness, *Ra*, shall not exceed 10 nm. It is recommended that the *Ra* be less than 1 nm to be of practical use for calibration purpose. If measured with an atomic-force-microscope (AMF), the sampling length, *l*, shall be 10 µm.

NOTE At the nano range, it is important to consider the spatial wavelength of the roughness as well as the amplitude.

3.6 In order to check that no material has been subsequently removed from the reference block, its thickness at the time of calibration shall be marked on it to the nearest 10 µm or an identifying mark shall be made on the test surface (see Clause 8).

For some nano range reference materials, it can be necessary to prepare a surface before the test in a manner that removes surface layers. In this case, a method such as a mark of defined depth should be used to reveal when a significant amount of material has been removed. Certification for nano range indentations can cover reference block depths much less than 10 µm.

4 Calibrating machine

4.1 General

In addition to fulfilling the general conditions specified in ISO 14577-2:2015, the calibrating machine shall also meet the requirements listed in 4.2 to 4.5. The calibrating machine shall be calibrated and verified directly at intervals not exceeding 24 months. Calibration and verification involves the following:

a) calibration of the test force;

b) verification of the indenter;

c) calibration of the displacement measuring system;

d) verification of the testing cycle.

The instruments used for verification and calibration shall be traceable to the National Standards, as far as available.

4.2 Calibration of the test force

The test force shall be calibrated according to ISO 14577-2:2015, 4.2, to a tolerance given in the following:

a) ±0,25 % for the macro range;

b) ±0,5 % for the micro range;

c) the larger of ±0,5 % or ±10 µN for the nano range.

The force shall be measured with elastic force-proving instruments of class 0,5 or better in accordance with ISO 376 or by another method having the same accuracy.

4.3 Verification of the indenter

4.3.1 General

The certified measured values (e.g. angle, radius, etc.) of the indenter shall be used in all calculations and, where indentation depth is ≤6 µm, the certified indenter area function with the relative uncertainty of less than 5 % shall be used.

In the nano and low micro ranges (h < 1 000 nm), the tolerances on the indenter angles are not normally achieved. The sharpness of the tip is likely to have the most significant impact on the measurement. It is difficult to determine the radius of curvature of an indenter to better than ±10 nm as this is likely the radius of an AFM probe. Indentation methods using certified indentation modulus reference blocks are easier for users, but give only a projected area value and so are ambiguous about shape. Due to the important requirement that the uncertainty of the measured area function be low, it is recommended to carefully consider the type of indenter and material parameter used for the calibration of reference blocks in the nano and low micro ranges.

4.3.2 Vickers indenter

4.3.2.1 The four faces of the square-based diamond pyramid shall be highly polished, free from surface defects, and flat to within 0,000 3 mm.

4.3.2.2 The angle between opposite faces of the vertex of the diamond pyramid shall be (136 ± 0,1)° (see ISO 14577-2:2015, Figure 2). The maximum uncertainty in the certified angle shall be ±0,15° at the 95 % confidence level.

The inclination of the axis of the diamond pyramid to the axis of the indenter holder (normal to the seating surface) shall be less than 0,3°.

The point of the diamond indenter should be examined using a high power measuring microscope or preferably using an interference microscope or an atomic-force-microscope.

4.3.2.3 If the four faces do not meet at a point, the line of conjunction between opposite faces shall be less than 0,001 mm. For indenters used in the micro and nano ranges, the length shall not exceed 0,000 25 mm.

4.3.2.4 It shall be verified that the quadrilateral that is being formed by the intersection of the faces with a plane perpendicular to the axis of the diamond pyramid has angles of (90 ± 0,4)° (see Figure 1).

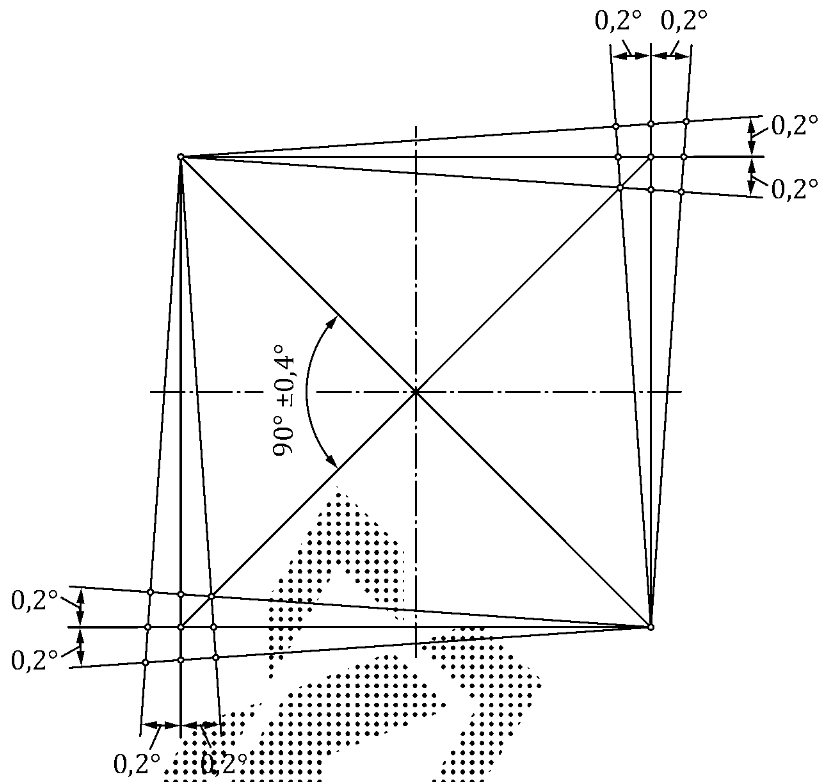

Figure 1 — Permissible difference of the sectional planes of the square form

4.3.3 Berkovich, modified Berkovich, corner cube indenters, hardmetal ball indenters, and spherical tipped conical indenters

For these indenters, it is recommended to use the tolerances given in ISO 14577-2:2015, 4.5.3, 4.5.4, and 4.5.5 as the minimum requirement. For triangular-based pyramidal indenters, the maximum uncertainty in the certified facet angle shall be ±0,15° at the 95 % confidence level.

4.4 Calibration of the displacement measuring device

4.4.1 The estimation capability required of the displacement measuring device depends on the size of the smallest indentation to be measured.

The scale of the displacement measuring system shall be graduated to permit estimation of the indentation depth in accordance with Table 1.

Table 1 — Estimation capability and maximum permissible error of the displacement measuring device

Range of application	Estimation capability of the measuring device	Maximum permissible error
Macro Micro and nano	10 nm 0,2 nm	0,5 % of h or 30 nm[a] 1 % of h or 5 nm[a]
[a] Whichever is greater. For the nano range, the tolerance of ±1 % of h is strongly recommended.		

The required estimation capability of the resulting indentation depth is fulfilled only if the uncertainties do not exceed the values given in Table 2.

Table 2 — Required uncertainty in zero-point determination and machine compliance

Range of application	Uncertainty in zero-point determination	Uncertainty in machine compliance
Macro Micro and nano	0,5 % of h 1 % of h	1 % of h 0,5 % of h

4.4.2 The displacement measuring device shall be verified according to ISO 14577-2:2015, 4.3.

The maximum permissible error shall not exceed the values given in Table 1.

4.5 Verification of the testing cycle

Recommended times for the different steps of calibration procedure and the velocities are given in Table 3.

For micro range testing, the maximum permissible vibration acceleration reaching the calibration machine shall be 0,005 g_n where g_n is the nominal gravitational acceleration (equal to 9,806 65 m/s^2). For control, a force-indentation depth curve is determined.

Table 3 — Recommended times for the testing cycle

Range of application	Maximum approach velocity of the indenter when contacting the reference block	Application time of test force	Duration time of test force	Removal time of test force
	µm/s	s	s	s
Macro Micro Nano	20 to 5 1 0, 1	30 30 30	30 30 30	10 10 10
NOTE For calibration of the indentation modulus (or indentation hardness) reference blocks, the maximum test force should be kept constant until the creep rate is reduced to less than 1 % of the initial displacement rate during the test force removal.				

5 Calibration procedure

The reference blocks shall be calibrated in a calibrating machine as described in Clause 4 at a temperature in the range specified in the calibration certificate [typically (23 ± 5) °C] using the general procedure specified in ISO 14577-1:2015 and taking into account the requirements of ISO 14577-2:2015, Clause 4.

6 Number of indentations

On each reference block, for the macro range, at least five indentations, and for the micro and nano ranges, 15 indentations shall be made. These shall be grouped in sets of five and the group locations distributed randomly on the surface, but their positions recorded for avoidance by future indents. For the nano range, it is recommended that the surface be marked or suitable coordinate system be established such that future indentations are guaranteed to avoid previously indented regions.

7 Uniformity of the reference blocks

7.1 For each reference block, the arithmetic mean value, $\overline{q}$, is calculated from the n values, q_1, ..., q_n, as given by Formula (1):

$$\bar{q} = \frac{q_1 + \ldots + q_n}{n} \quad (1)$$

where

q is the calibration value (materials parameter).

As a measure of the scattering, the experimental standard deviation is calculated as given by Formula (2):

$$s(q) = \sqrt{\frac{\sum_{i=1}^{n} (q_i - \bar{q})^2}{n-1}} \quad (2)$$

The relative scattering of the measured hardness values is the coefficient of variation or relative experimental standard deviation, V, expressed as a percentage as given by Formula (3):

$$V = \frac{s(q)}{\bar{q}} \times 100 \quad (3)$$

7.2 For the macro range, the maximum permissible coefficient of variation for the indirect verification purpose for HM, H_{IT}, and E_{IT} is 2 %. For the micro range, the maximum permissible coefficient of variation for the indirect verification purpose for E_{IT} is 2 %. For the nano range, the maximum permissible coefficient of variation for the indirect verification purpose for E_{IT} is 5 %. Higher coefficients of variation are allowed for reference blocks that are used in the nano and micro range for the determination of machine compliance (e.g. tungsten).

8 Marking

8.1 Each reference block shall be either marked directly or marked with a unique identifying code linking to a certificate containing the following particulars:

a) arithmetic mean of the values found in the calibration test, e.g. HM 0,5 / 20 / 20 = 8,70 GPa or E_{IT} 0,5 / 10 / 20 / 30 = 220,00 GPa;

b) name or mark of the supplier or manufacturer;

c) serial number;

d) name or mark of calibrating agency;

e) thickness of the block or an identifying mark on the test surface (see 3.6);

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

8.2 Any mark put on the side of the block shall be the right way up when the test surface is the upper face.

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

a) a reference to this part of ISO 14577 (i.e. ISO 14577-3:2015);

b) identity of the block;

c) date of calibration;

d) calibration temperature;

e) material and shape of the indenter and, where used, the detailed area function of the indenter;

f) testing cycle (control method and full description of the testing cycle); this should include

   1) set point values,

   2) rates and times of force,

   3) position and length of hold points, and

   4) data logging frequency or number of points logged for each section of the cycle;

g) method applied for the determination of the zero-point;

h) analysis methods;

i) individual values and their arithmetic mean of all calibrated values together with the experimental standard deviation coefficient of variation including

   1) the number of results used to derive these values, and

   2) the uncertainty in the calibrated value at the 95 % confidence level in accordance with ISO 14577-1:2015, Annex H;

j) diagram showing the superimposed force-displacement data (including zero-point) used to calculate the certified value of the reference block.

9 Validity

The reference block is valid only for the testing conditions for which it was calibrated and provided that the block fulfils the requirements of Clause 3.

NOTE Materials that plastically deform through dislocation mechanisms are known to return an indentation-size-dependent hardness. The hardness measured, even by self-similar geometry indenters is proportional to the inverse square root of the indent diameter. In the macro range, the small relative difference in indentation size means that this effect is not noticeable. However, in the micro and nano ranges, the effect is very significant. Consequently, hardness values are only directly comparable for indents of the same size. Therefore, only the indentation modulus can be used as a certified material parameter for the calibration of frame compliance and indenter area function in the nano and micro ranges (see Reference [1]).

It is recommended that the duration of the calibration validity is limited to five years.

Bibliography

[1] Hou D., Bushby A.J., Jennett N.M. Study of the interaction between the indentation size effect and Hall-Petch effect with spherical indenters on annealed polycrystalline copper. J. Phys. D Appl. Phys. 2008, **41** (7) p. 074006

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

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

[4] ISO/IEC Guide 98-3:2008, *Uncertainty of measurement — Part 3: Guide to the expression of uncertainty in measurement (GUM:1995)*

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