INTERNATIONAL STANDARD

ISO 14577-3

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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
ISO 14577-3:2002(E)

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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 3.

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 part of ISO 14577 may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 14577-3 was prepared by Technical Committee ISO/TC 164, *Mechanical testing of metals*, Subcommittee SC 3, *Hardness testing*.

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*

Annex A of this part of ISO 14577 is for information only.

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 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 need to measure the indent optically.

ISO 14577 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 be used for the indirect verification of testing machines for the instrumented indentation test, as specified in ISO 14577-2.

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

2 Normative references

The following normative documents contain provisions which, through reference in this text, constitute provisions of this part of ISO 14577. For dated references, subsequent amendments to, or revisions of, any of these publications do not apply. However, parties to agreements based on this part of ISO 14577 are encouraged to investigate the possibility of applying the most recent editions of the normative documents indicated below. For undated references, the latest edition of the normative document referred to applies. Members of ISO and IEC maintain registers of currently valid International Standards.

ISO 376:1999, *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 14577-1:2002, *Metallic materials — Instrumented indentation test for hardness and materials parameters — Part 1: Test method*

ISO 14577-2:2002, *Metallic materials — Instrumented indentation test for hardness and materials parameters — Part 2: Verification and calibration of testing machines*

ISO Guide to the Expression of Uncertainty in Measurement (GUM) [1)]

3 Manufacture of reference blocks

3.1 The block shall be specially prepared and the attention of the manufacturer drawn to the need to use a manufacturing process which will give the necessary homogeneity, uniformity and stability of structure.

1) Published in 1993; corrected and reprinted in 1995.

3.2 Each block to be calibrated shall be of a thickness not less than 2 mm for the nano range, not less than 6 mm for the micro and not less than 16 mm for the macro range.

NOTE If it is required by the manufacturing process, these values may be lower.

3.3 The reference blocks shall be free from magnetic forces. It is recommended that the manufacturer ensure that the blocks, if of steel, have been 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.

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 15 nm 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. 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 indentifying mark shall be made on the test surface (see clause 8).

NOTE 1 For some nano range reference materials, it may be a necessary step to prepare a surface before 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 may cover reference block depths much less than 10 µm.

NOTE 2 Examples of materials for reference blocks are given in annex A.

4 Calibrating machine

4.1 General

In addition to fulfilling the general conditions specified in ISO 14577-2, the calibrating machine shall also meet the requirements listed in 4.2 to 4.5. The calibrating machine shall be verified directly at intervals not exceeding 12 months. Direct verification involves:

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 National Standards as far as available.

4.2 Calibration of the test force

Each test force shall be accurate to the nominal value specified in ISO 14577-1 to within:

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,

and shall be calibrated as described in 4.3.1 of ISO 14577-2:2002.

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

4.3 Verification of the indenters

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 shall be used.

NOTE 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 the likely radius of an AFM probe. Indentation methods using certified modulus reference blocks are easier for users but only give a projected area value and so are ambiguous about shape. Because of the important requirement that the uncertainty of the measured area function is 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 ± 0,3)°, see Figure 2 of ISO 14577-2:2002. 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°.

NOTE 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 which is being formed by the intersection of the faces with a plane perpendicular to the axis of the diamond pyramid has angles of (90 ± 0,2)° (see Figure 1).

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

For these shapes of indenter it is recommended to use the tolerances given in 4.2.3, 4.2.4 and 4.2.5 of ISO 14577-2:2002 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.

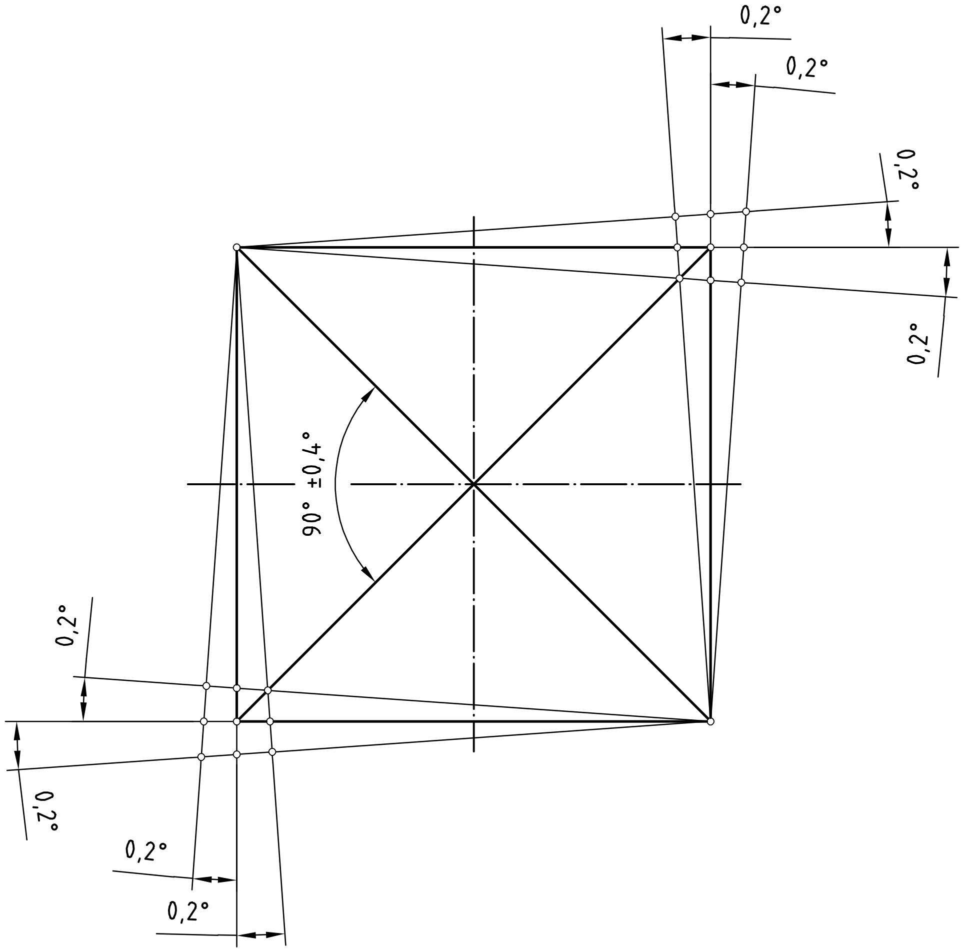

Figure 1 — Permissible difference of the sectional planes of the square form

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	10 nm	0,5 % of h or 30 nm[a]
Micro and nano	0,2 nm	1 % of h or 5 nm[a]

[a] Whichever is greater. For the nano range the tolerance of ± 1 % of h is strongly recommended.

4.4.2 The displacement measuring device shall be verified according to 4.4.2 of ISO 14577-2:2002.

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

4.5 Verification of the testing cycle

The times for the different steps of calibration procedure and the velocities are given in Table 2.

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 (9,806 65 m/s^2). For control a force-indentation depth curve is determined.

Table 2 — Times for the testing cycle

Range of application	Application time of test force s	Maximum approach velocity of the indenter when contacting the reference block µm/s	Duration time of test force s	Removal time of test force s
Macro	20	20 to 5	10	20
Micro	20	1	10	20
Nano	20	0, 1	10	20
NOTE For calibration of indentation modulus 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 and taking into account the requirements in 4.3.3 of ISO 14577-2:2002.

6 Number of indentations

On each reference block, for the macro range at least 5 indentations and for the micro and nano ranges 15 indentations shall be made. These shall be uniformly distributed over the test surface.

7 Uniformity of the reference blocks

NOTE In the following equations the symbol q for the calibration value (materials parameter) is generally used.

7.1 For each reference block from the n values q_1, ..., q_n the arithmetic mean value $\bar{q}$ is calculated:

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

As a measure of the scattering the experimental standard deviation is calculated as follows:

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

The relative scattering of the measured hardness values is the coefficient of variation or relative experimental standard deviation, expressed as a percentage:

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

7.2 The maximum permissible coefficient of variation for the indirect verification purpose for HM, H_{IT}, E_{IT} is 2 %.

NOTE Higher coefficients of variation are possible for reference blocks which are used for determination of machine compliance (e.g. tungsten).

8 Marking

8.1 Each reference block shall be marked with following particulars:

a) arithmetic mean of the values found in the calibration test, e.g. HM 0,1 = 249 N/mm^2 or E_{IT} = 220 000 N/mm^2;

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) reference to this part of ISO 14577, i.e. ISO 14577-3;

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;

   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:

   — the number of results used to derive these values;

   — the uncertainty in the calibrated value at the 95 % confidence level, in accordance to clause 8 of ISO 14577-1:2002;

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

NOTE The expanded uncertainty material parameter is $\pm t_p (\nu_{eff}) \times U_c (y)$ at 95 % confidence level, where t_p is t statistic for a “two-tailed test” with (ν_{eff}) degrees of freedom at 95 % confidence.

For $\nu_{eff} = 14$, $t_{95} = 2,14$; for $\nu_{eff} = 5$, $t_{95} = 2,78$

where

ν_{eff} is the effective number of degrees of freedom calculated according to the Welch-Satterthwaite equation (see annex G of ISO GUM:1995).

$U_c (y)$ is the combined standard uncertainty of parameter determined by root sum squared method (if uncorrelated), all calculated variances of parameters influencing the result

9 Validity

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

It is recommended that the duration of the calibration validity be limited to 5 years.

Annex A
(informative)

Examples of reference blocks

Table A.1 — Examples of reference blocks

Material	**Hardness range** HM 0,25 N/mm^2	E_{IT} **range** N/mm^2
steel	1 000 – 8 400	210 000
glass	3 000 – 5 000	65 000 to 85 000
glass BK 7	4 200	82 000
glass SF 6	2 800	56 000
ceramics	10 000 – 18 000	200 000 to 380 000
tungsten	4 000 – 5 000	411 000
fused silica	4 800 – 5 200	72 000

Bibliography

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

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