INTERNATIONAL STANDARD

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

Part 2: Verification and calibration of testing machines

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

Partie 2: Vérification et étalonnage des machines d'essai

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ISO copyright office
Ch. de Blandonnet 8 • CP 401
CH-1214 Vernier, Geneva, Switzerland
Tel. +41 22 749 01 11
Fax +41 22 749 09 47
copyright@iso.org
www.iso.org

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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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For an explanation on the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the WTO principles in the Technical Barriers to Trade (TBT), see the following URL: Foreword - Supplementary information

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 14577-2:2002), 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 need 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 2: Verification and calibration of testing machines

1 Scope

This part of ISO 14577 specifies the method of verification and calibration of testing machines for carrying out the instrumented indentation test in accordance with ISO 14577-1:2015.

It describes a direct verification method for checking the main functions of the testing machine and an indirect verification method suitable for the determination of the repeatability of the testing machine. There is a requirement that the indirect method be used in addition to the direct method and for the periodic routine checking of the testing machine in service.

It is a requirement that the indirect method of verification of the testing machine be carried out independently for each test method.

This part of ISO 14577 is also applicable for transportable testing machines.

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 3878, *Hardmetals — Vickers hardness test*

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

ISO 14577-3, *Metallic materials — Instrumented indentation test for hardness and materials parameters — Part 3: Calibration of reference blocks*

3 General conditions

3.1 Preparation

The machine shall be designed in such a way that it can be verified.

Before verification and calibration of the testing machine, it shall be checked to ensure that the conditions laid down in 3.2 to 3.4 are met.

3.2 Functional installation

The testing machine shall be configured to operate in compliance with and shall be installed in an environment that meets the requirements of this part of ISO 14577, ISO 14577-1:2015, and, where applicable, ISO 14577-3. The testing machine shall be protected from vibrations. For testing in the micro and nano ranges, the testing machine shall also be protected from air currents and temperature fluctuations.

The influence of environment on the data, i.e. the noise floor, shall be estimated by performing a low force (e.g. equivalent to the usual initial contact force) indentation on a CRM and analysing the displacement over time. The force variability is the indent stiffness (obtained from force removal curve) multiplied by the standard deviation of the displacement once any background drift in mean displacement has been subtracted. These uncertainties shall then be included in the total combined uncertainty tests as calculated in ISO 14577-1:2015, Clause 8 and Annex H.

3.3 Indenter

In order to get repeatable measurements of the force/indentation depth data set, the indenter holder shall be firmly mounted into the testing machine.

The indenter holder should be designed in such a way that the contribution to the overall compliance is minimized (see Annex A).

3.4 Application of the test force

The test force shall be applied and removed without shock or vibration that can significantly affect the test results. It shall be possible to verify the process of increasing, holding, and removal of the test force.

4 Direct verification and calibration

4.1 General

4.1.1 Direct verification and calibration shall be carried out at the constant temperature of use, typically 10 °C to 35 °C, but preferably in the range (23 ± 5) °C. If a range of operating temperatures is required, then direct calibration and verification should be carried out at suitable points over that temperature range to determine the calibration validity as a function of temperature. If necessary, a calibration correction function or a set of calibrations valid at specific operating temperatures can be determined.

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

4.1.3 Direct verification and calibration involves

a) calibration of the test force,

b) calibration of the displacement measuring device,

c) verification and calibration of the machine compliance,

d) verification of the indenter,

e) calibration and verification of the indenter area function, if the indentation depth is less than 6 µm, and

f) verification of the test cycle.

4.2 Calibration of the test force

4.2.1 Each range of force used shall be calibrated over the whole force range for both application and removal of the test force. A minimum of 16 evenly distributed points in the test force range shall be calibrated, i.e. 16 during application and 16 during removal of the test force. The procedure shall be repeated at least three times and the average calibration value shall be used. The maximum difference in calibration values shall not exceed half of the tolerances given in Table 1.

4.2.2 The test force shall be measured by a traceable method, for example, the following:

a) measuring by means of an elastic proving-device in accordance with class 1, or better of, ISO 376;

b) balancing against a force, accurate to within ±0,2 % applied by means of calibrated masses with mechanical advantage;

c) electronic balance with a suitable accuracy of 0,1 % of the minimum calibrated test force or 10 μg (0,1 μN) for the nano range.

For each measured point used for calibration, the difference between the measured and the nominal test force shall be within the tolerances given in Table 1.

Table 1 — Tolerances for test forces

Range of the test force F N	**Tolerances** %
$F \geq 2$	±1,0
$0{,}001 \leq F < 2$	±1,0
$F < 0{,}001$	±2,5[a]

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

4.3 Calibration of the displacement measuring device

4.3.1 The resolution required of the displacement measuring system depends on the size of the smallest indentation depth being measured. For the micro range, this value is by definition h = 0,2 μm; for the macro range it is typically ≥2 μm.

The scale of the displacement measuring device shall be graduated to permit a resolution of indentation depth measurement in accordance with Table 2.

4.3.2 The displacement measuring device shall be calibrated on the testing machine for every range used by means of a suitable method and a corresponding system. The device shall be calibrated at a minimum of 16 points in each direction evenly distributed throughout its travel. The procedure shall be repeated three times.

The following methods are recommended for the measurement of the relative displacement of the indenter: laser interference method, inductive method, capacitive method, and piezotranslator method.

For each measured point used for calibration, the difference between the measured and the nominal displacement shall be within the tolerances given in Table 2.

Table 2 — Resolution and tolerances of the displacement measuring device

Range of application	**Resolution of the displacement measuring device** nm	**Tolerances**
Macro	≤100	1 % of h
Micro	≤10	1 % of h
Nano	≤1	2 nm[a]

[a] For the nano range, a tolerance of <1 % of h (indentation depth) is strongly recommended.

4.3.3 Changes in temperature are commonly a dominant source of displacement drift. To minimize thermally induced displacement drift, the temperature of the instrument shall be maintained such that the displacement drift rate remains constant over the time period of one calibration cycle. The drift rate shall be measured during, immediately before, or immediately after each calibration cycle, e.g. by monitoring displacement during a suitable hold period. The displacement calibration data shall be corrected for thermal drift and the product of variation in drift rate and the duration of one calibration cycle shall be less than the tolerance given in Table 2. The drift rate uncertainty shall be included in the displacement calibration uncertainty calculation.

4.4 Verification and calibration of the machine compliance

4.4.1 General

See Annex D and ISO 14577-1:2015, Annex C.

This verification and calibration shall be carried out after the test force and the displacement measuring system have been calibrated in accordance with 4.2 and 4.3.

4.4.2 Procedure

The calibration and verification of machine compliance is carried out by the measurement of indentation modulus at a minimum of five different test forces. Method 3 as described in Annex D is recommended. In all cases, a suitable Certified Reference Material (CRM) shall be mounted into the instrumented indentation test system in the same way as future test samples will be mounted. This is to ensure that the CRM provides a faithful reproduction of each particular total machine compliance.

The compliance of the testing machine can be affected by the particular construction and mounting of an indenter and also the method used to mount a sample. For instance, mounting in plastics (e.g. PVC) can introduce an extra compliance into the measurement loop. The verification and calibration of machine compliance should be performed using the indenter that will be used for subsequent measurements.

For indentation depths, $h_c > 6$ µm, it is not necessary to take into account the real contact area function. For the verification and calibration of the machine compliance, a reference material with certified indentation modulus, independent from the indentation depth, shall be used. A material with a high ratio of $E_{IT} / \sqrt{H_{IT}}$ (such as tungsten) is recommended. The range for the test force is defined by the minimum test force that correlates to 6 µm indentation depth and the maximum possible test force of the testing machine. Large indentation depths have the advantage that errors in the area function are likely to be smaller; however, care shall be taken that the test is not biased by pile-up in the reference material. The measured compliance of the indentation shall then be compared with the calculated compliance for the indentation using the certified value of modulus. To recalibrate machine compliance, the product of the applied force and the detected difference in machine compliance is applied to the displacement data to refine the estimate of contact depth and, therefore, the machine compliance estimate at each force. This process is iterated until self-consistent values of machine compliance and contact depth are reached.

For indentation depths, <6 µm, the method above shall be applied, except that the actual area of contact, as calculated from the calibrated indenter area function, shall be used to calculate the contact compliance using the certified modulus of the CRM.

In many nano and micro range instruments, the machine compliance value is independent of force. However, if this is not the case, then a machine compliance function can be determined using the above procedure but a wider range of forces. The range for the test forces is defined by the indentation depths, >0,5 µm, and the maximum test force of the testing machine or the maximum test force for which no unusual test piece response (e.g. pile-up of metals or cracking of ceramics or glasses) occurs.

If the machine compliance is recalibrated, then an indirect validation shall be performed before use.

The calibration procedures detailed in Annex D require the use of reference materials (see ISO 14577-3) that shall be isotropic and homogeneous. It is assumed that the indentation modulus and Poisson’s ratio are independent of the indentation depth.

4.5 Calibration and verification of the indenter

4.5.1 General

The indenter used for the indentation test shall be calibrated. Evidence that the indenter complies with the requirements of this part of ISO 14577 shall be fulfilled by a calibration certificate from a qualified calibration laboratory and evidence from the most recent indirect verification that the indenter area function has not changed. The latter shall be provided using the verification methods described in Annex B and suitable certified reference materials. All specified indenter geometry parameters shall be measured and incorporated into the calibration certificate.

If the angle of the indenter deviates from the nominal value for an ideal geometry of the indenter, the average of certified angles for that indenter should be used in all applicable calculations at depths $h > 6$ µm.

NOTE A 0,2° error in the Vickers angle of 136° (2α) results in a 1 % systematic error in area.

Indenters for use in the nano range and in the micro range, indentation depth <6 µm, shall have their area function calibrated over the relevant indentation depth ranges of use. The indenter performance shall be verified periodically (see Clause 6).

Where non-diamond indenters are used, the values of elastic modulus and Poisson ratio shall be obtained and used instead of the diamond values in the appropriate analyses.

The angle for pyramidal and conical indenters shall be measured within the indentation depth ranges given in Table 3 and illustrated in Figure 1.

Table 3 — Values for the measuring ranges for the angle of pyramidal and conical indenters

Dimensions in micrometres

Indentation depth	Macro range	Micro range
h_1	6	0,2
h_2	200	Specified max. indentation depth

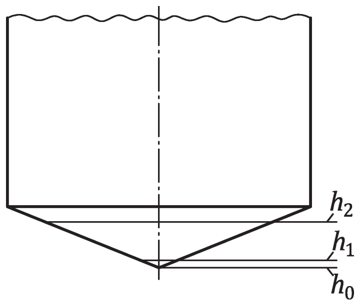

Figure 1 — Illustration of measuring ranges given in Table 3

4.5.2 Vickers indenter

4.5.2.1 The four faces of the right square-based diamond pyramid shall be smooth and free from surface defects and contaminants. For notes on cleaning of the indenter surface, see also ISO 14577-1:2015, Annex D.

The surface roughness of the indenter has a similar effect on measurement uncertainty as test piece roughness. When testing in the nano range, the indenter surface finish should be taken into consideration.

4.5.2.2 The angle between the opposite faces of the vertex of the diamond pyramid shall be 136° ± 0,3° (see Figure 2) (α = 68,0° ± 0,2°).

The angle shall be measured in the range between h_1 and h_2 (see Table 3 and Figure 1). The geometry and finish of the indenter shall be controlled over the whole calibrated indentation depth range, i.e. from the indenter tip, h_0, to the maximum calibrated indentation depth, h_2.

4.5.2.3 The angle between the axis of the diamond pyramid and the axis of the indenter holder (normal to the seating surface) shall not exceed 0,5°.

4.5.2.4 The four faces shall meet at a point. The maximum permissible length of the line of conjunction between opposite faces is given in Table 4 (see also Figure 3).

4.5.2.5 The radius of the tip of the indenter shall not exceed 0,5 µm for the micro range (see Figure 4).

4.5.2.6 The verification of the shape of the indenter shall be carried out using microscopes or other suitable devices.

If the indenter is used for testing in the micro or nano range, verification by a closed loop controlled atomic-force-microscope (AFM) should be carried out. For the nano range, this measurement is strongly recommended.

Table 4 — Maximum permissible length of the line of conjunction

Range of the indentation depth µm	Maximum permissible length of the line of conjunction µm
$h > 30$	1
$30 \geq h > 6$	0,5[a]
$h \leq 6$	≤ 0,5[b]

[a] This can be assumed to have been achieved when there is no detectable conjunction when the indenter is verified by an optical microscope at 400 × magnification.

[b] This shall be included when the correction of the shape of the indenter is taken into account; see ISO 14577-1:2015, C.2.

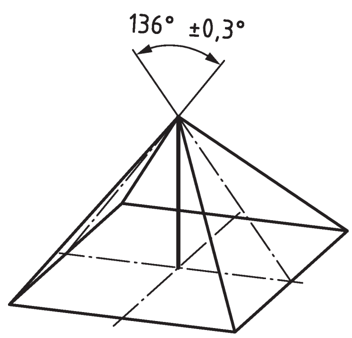

Figure 2 — Angle of the Vickers diamond pyramid