INTERNATIONAL STANDARD

ISO 14577-2

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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

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 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 14577-2 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 B forms a normative part of this part of ISO 14577. Annexes A, C and D are 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 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.

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. The indirect method shall be used in addition to the direct method and for the periodic routine checking of the testing machine in service.

The indirect method of verification of the testing machine shall 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 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 3878, *Hardmetals — Vickers hardness test*

ISO 6508-2, *Metallic materials — Rockwell hardness test — Part 2: Verification and calibration of testing machines (scales A, B, C, D, E, F, G, H, K, N, T)*

ISO 14577-1:2002, *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*

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

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

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 the conditions laid down in 3.2 to 3.4

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 document and of ISO 14577-1 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. This influence shall be checked by repeated measurements of the force/indentation depth curve.

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 would 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 The direct verification shall be carried out at a temperature of (23 ± 5) °C.

If a range of operating temperatures is required, then direct verification should be carried out at suitable points over that range to determine the validity of the calibration as a function of temperature. If necessary, a calibration correction function or a set of calibrations valid at specific operating temperatures may be determined.

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

4.1.3 Direct verification involves:

a) verification of the indenter;

b) calibration of the test force;

c) calibration of the displacement measuring device;

d) calibration of the machine compliance;

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

f) verification of the testing cycle.

4.2 Verification of the indenter

4.2.1 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[2)] 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 geometrical values shall be measured and incorporated into the calibration certificate. The indenter performance shall be verified periodically (see clause 6).

If the angle of the indenter deviates from the nominal value for an ideal geometry of the indenter, the average of the certified angles for that indenter should be used in all applicable calculations, e.g. 0,2° error in the Vickers angle of 136° results in a 1 % systematic error in area.

The angle for pyramidal and conical indenters shall be measured within the indentation depth ranges given in Table 1 (and illustrated in Figure 1). Indenters for use in the nano range and in the micro range (indentation depth $\leqslant$ 6 µm) shall have their area function calibrated over the relevant indentation depth ranges of use.

Table 1 — 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	120

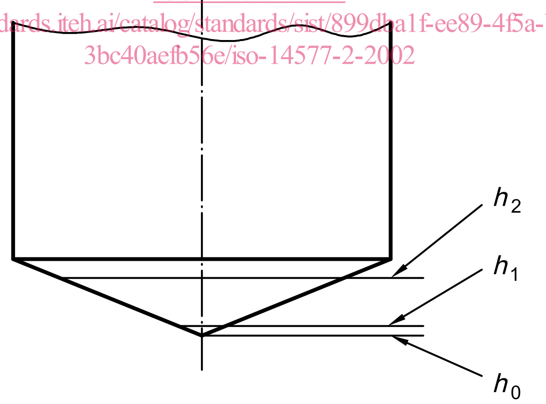


Figure 1 — Illustration of measuring ranges given in Table 1

4.2.2 Vickers indenter

4.2.2.1 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 annex D in ISO 14577-1:2002.

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.

2) See ISO/IEC 17025 [1].

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

The angle shall be measured in the range between h_1 and h_2 (see Table 1 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.

4.2.2.3 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°.

4.2.2.4 The four faces shall meet at a point. The maximum permissible length of the line of conjunction between opposite faces is given in Table 2 (see also Figure 3).

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

4.2.2.6 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 a verification by an atomic-force-microscope (AFM) is recommended.

Table 2 — 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
$30 \geqslant h > 6$	0,5[a]
$h \leqslant 6$	$\leqslant$ 0,5[b]

[a] This may 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] The correction of the shape of the indenter is taken into account, see C.2 in ISO 14577-1:2002.

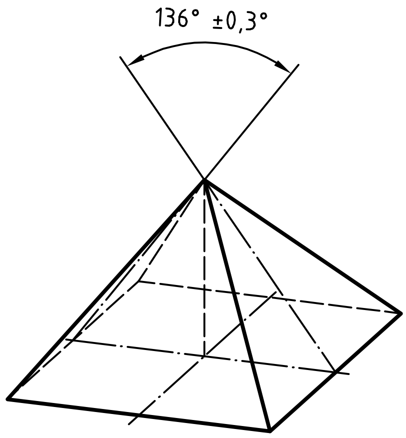
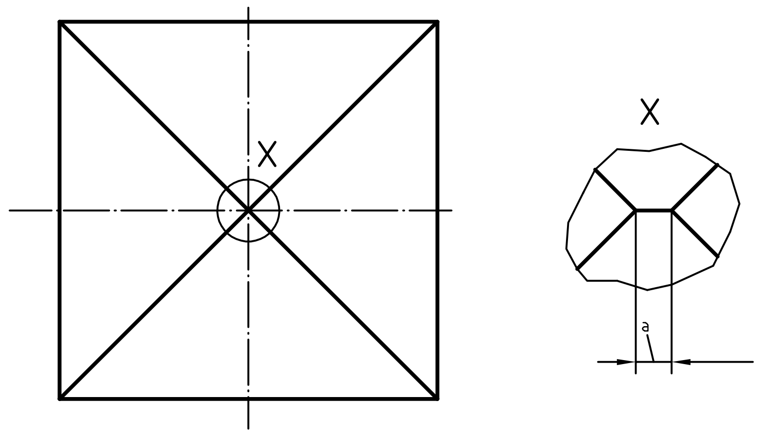


Figure 2 — Angle of the Vickers diamond pyramid



a Line of conjunction

Figure 3 — Line of conjunction on the tip of the indenter, schematically

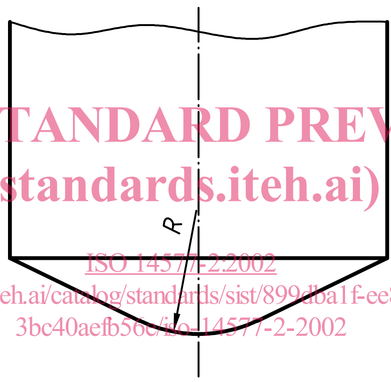


Figure 4 — Radius of the tip of the indenter

4.2.3 Berkovich, modified Berkovich and corner cube indenters

4.2.3.1 General

In practice there are two types of Berkovich pyramidal diamond indenters in common use. The Berkovich indenter [2] is designed to have the same surface area as a Vickers indenter at any given indentation depth. The modified Berkovich indenter [3] is designed to have the same projected area as the Vickers indenter at any given indentation depth.

4.2.3.2 The three faces of the triangular based diamond pyramid shall be smooth and free from surface defects and from contaminations. For notes on cleaning of the surface see also annex D in ISO 14577-1:2002.

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

4.2.3.3 The radius of the tip of the indenter shall not exceed 0,5 µm for the micro range and shall not exceed 0,2 µm for the nano range (see Figure 4).

4.2.3.4 The angle between the axis of the diamond pyramid and the three faces is designated α. The angle between the three faces of the diamond pyramid shall be 60° ± 0,3° (see Figure 5).

