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

**Part 1:
Test method**

iTeh STANDARD PREVIEW

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

Partie 1: Méthode d'essai

ISO 14577-1:2002

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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 14577-1 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*: (standards.iteh.ai)

- Part 1: Test method [ISO 14577-1:2002](https://standards.iteh.ai/catalog/standards/sist/993e9075-3478-4204-8b1a-329f633bb637/iso-14577-1-2002)
- Part 2: Verification and calibration of testing machines <https://standards.iteh.ai/catalog/standards/sist/993e9075-3478-4204-8b1a-329f633bb637/iso-14577-1-2002>
- Part 3: Calibration of reference blocks

Annexes A, C and E form a normative part of this part of ISO 14577. Annexes B, D and F 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.

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

Part 1: Test method

1 Scope

This part of ISO 14577 specifies the method of instrumented indentation test for determination of hardness and other materials parameters for the three ranges given in Table 1.

Table 1 — Ranges of application

| Macro range | Micro range | Nano range ^a |
|--|--|----------------------------------|
| $2 \text{ N} \leq F \leq 30 \text{ kN}$ | $2 \text{ N} > F; h > 0,2 \text{ }\mu\text{m}$ | $h \leq 0,2 \text{ }\mu\text{m}$ |
| ^a For the nano range the mechanical deformation strongly depends on the real shape of indenter tip and the calculated materials parameters are significantly influenced by the contact area function of the indenter used in the testing machine. Therefore careful calibration of both instrument and indenter shape is required in order to achieve an acceptable reproducibility of the materials parameters determined with different machines. | | |

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The macro and micro range are distinguished by the test forces in relation to the indentation depth.

Attention is drawn to the fact that the micro range has an upper limit given by the test force (2 N) and a lower limit given by the indentation depth of 0,2 μm .

The determination of hardness and other materials parameters is given in annex A.

At high contact pressures, damage to the indenter is possible. For this reason in the macro range, hardmetal indenters are often used. For test pieces with very high hardness and modulus of elasticity the influence of indenter deformation on the test result should be taken into account.

NOTE This test method can also be applied to thin metallic and non-metallic coatings and non-metallic materials. In this case the specifications in the relevant standards should be taken into account (see also 6.3).

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

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⁾

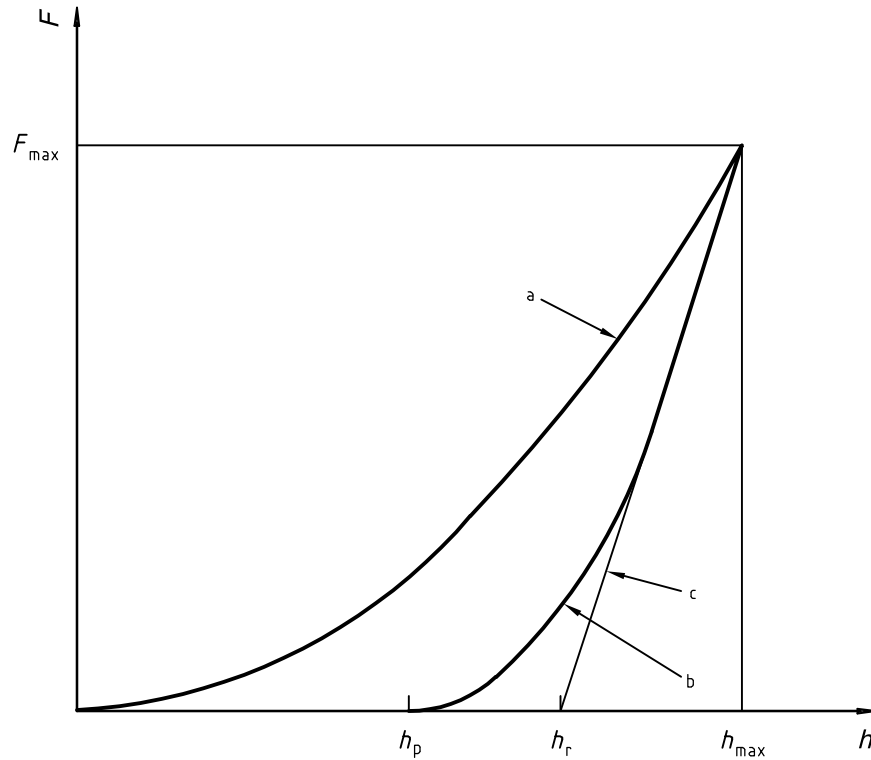
3 Symbols and designations

For the purposes of this International Standard, the symbols and designations in Table 2 shall be applied (see also Figure 1 and Figure 2).

Table 2 — Symbols and designations

| Symbol | Designation | Unit |
|-----------------|---|-------------------|
| $A_p(h_c)$ | Projected area of contact of the indenter at distance h_c from the tip | mm ² |
| $A_s(h)$ | Surface area of the indenter at distance h from the tip | mm ² |
| C_{IT} | Indentation creep | % |
| E_{IT} | Indentation modulus | N/mm ² |
| F | Test force | N |
| F_{max} | Maximum test force | N |
| h | Indentation depth under applied test force | mm |
| h_c | Depth of the contact of the indenter with the test piece at F_{max} | mm |
| h_{max} | Maximum indentation depth at F_{max} | mm |
| h_p | Permanent indentation depth after removal of the test force | mm |
| h_r | Point of intersection of the tangent c to curve b at F_{max} with the indentation depth-axis (see Figure 1) | mm |
| H_{IT} | Indentation hardness | N/mm ² |
| HM | Martens hardness | N/mm ² |
| HM _s | Martens hardness, determined from the slope of the increasing force/indentation depth curve | N/mm ² |
| r | Radius of spherical indenter | mm |
| R_{IT} | Indentation relaxation | % |
| W_{elast} | Elastic reverse deformation work of indentation | N·m |
| W_{total} | Total mechanical work of indentation | N·m |
| α | Angle, specific to the shape of the pyramidal indenter | ° |
| η_{IT} | Relation W_{elast}/W_{total} | % |
| NOTE 1 | To avoid very long numbers the use of multiples or sub-multiples of the units is permitted. | |
| NOTE 2 | 1 N/mm ² = 1 MPa. | |

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

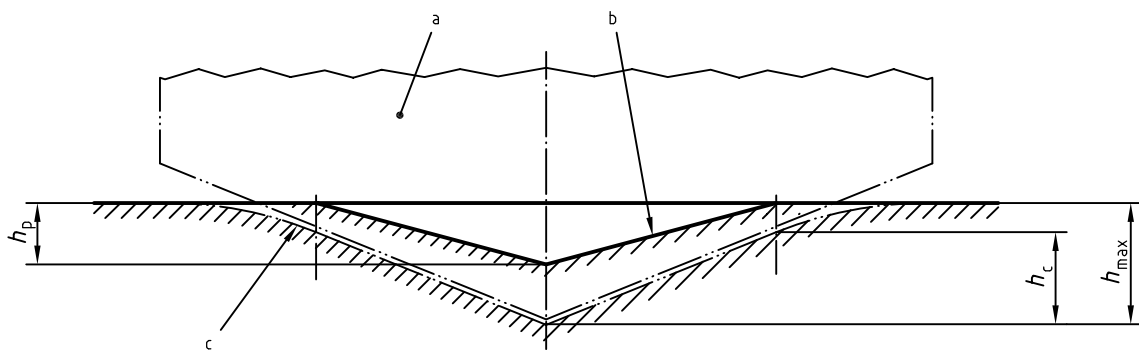


- a Application of the test force.
- b Removal of the test force.
- c Tangent to curve b at F_{max} .

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Figure 1 — Schematic representation of test procedure



- a Indenter.
- b Surface of residual plastic indentation in test piece.
- c Surface of test piece at maximum indentation depth and test force.

Figure 2 — Schematic representation of cross section of indentation

4 Principle

The continuous monitoring of the force and the depth of indentation can permit the determination of hardness and material properties (see Figure 1 and Figure 2). An indenter consisting of a material harder than the material under test with the following shapes and materials can be used:

- a) diamond indenter shaped as an orthogonal pyramid with a square base and with an angle $\alpha = 136^\circ$ between the opposite faces at the vertex (Vickers pyramid, see Figure A.1);
- b) diamond pyramid with triangular base (e. g. Berkovich pyramid, see Figure A.1);
- c) hardmetal ball (especially for the determination of the elastic behaviour of materials);
- d) diamond spherical indenter.

This part of ISO 14577 does not preclude the use of other indenter geometries, however, care should be taken in interpreting the results obtained with such indenters. Other materials like sapphire may also be used.

NOTE Due to the crystal structure of diamond, indenters that are intended to be spherical are often polyhedrons and have no ideal spherical shape.

The test procedure can either be force-controlled or displacement-controlled. The test force F , the corresponding indentation depth h and time are recorded during the whole test procedure. The result of the test is the data set of the test force and the relevant indentation depth as a function of time (see Figure 1 and annex B).

For a reproducible determination of the force and corresponding indentation depth, the zero point for the force/indentation depth measurement shall be assigned individually for each test (see 7.3).

Where time-dependent effects are being measured:

- a) using the force-controlled method, the test force is kept constant over a specified period and the change of the indentation depth is measured as a function of the holding time of the test force (see Figures A.3 and B.1);
- b) using the indentation depth controlled method the indentation depth is kept constant over a specified period and the change of the test force is measured as a function of the holding time of the indentation depth (see Figures A.4 and B.2).

The two kinds of control mentioned give essentially different results in the segments b of the curves in Figures B.1a) and B.2b).

5 Testing machine

5.1 The testing machine shall have the capability of applying predetermined test forces within the required scope and shall fulfill the requirements of ISO 14577-2.

5.2 The testing machine shall have the capability of measuring and reporting applied force, indentation displacement and time, throughout the testing cycle.

5.3 The testing machine shall have the capability of compensating for the machine compliance and of utilizing the appropriate indenter area function (see annex C in this document and 4.5 and 4.6 of ISO 14577-2:2002).

5.4 Indenters for use with testing machines may have various shapes, as specified in ISO 14577-2 (For further information on diamond indenters see annex D).

5.5 The testing machine shall maintain its calibration over the testing machine's usual operating temperature range.

The testing machine shall operate at a temperature within the permissible range specified in 7.1 and shall maintain its calibration within the limits prescribed in 4.4.3 of ISO 14577-2:2002.

6 Test piece

6.1 The test shall be carried out on a region of the test surface that allows the determination of the force/indentation depth curve for the respective indentation range within the required uncertainty. The contact area shall be free of fluids or lubricants except where this is essential for the performance of the test. This shall be described in detail in the test report. Care shall be taken that extraneous matter (e.g. dust particles) is not contained in the contact area.

For an explanation concerning the influence of the test piece roughness on the uncertainty of the results, see annex E. Surface finish has a significant influence on the test results.

The test surfaces shall be normal to the test force direction.

Tilt should be included in the uncertainty calculation. Typically test surface tilt is less than 1°.

6.2 Preparation of the test surface shall be carried out in such a way that any alteration of the surface hardness (e.g. due to heat or cold-working) is minimized.

Due to the small indentation depths in the micro and nano range, special precautions shall be taken during the test piece preparation. A polishing process that is suitable for particular materials shall be used (e.g. an electropolishing process).

6.3 The test piece thickness shall be large enough (or indentation depth small enough) such that the test result is not influenced by the test piece support. The test piece thickness should be at least $10 \times$ the indentation depth or $3 \times$ the indentation diameter (see 7.7), whichever is greater.

When testing coatings, the coating thickness should be considered to be the test piece thickness.

NOTE The above are empirically based limits. The exact limits of influence of support on test piece will depend on the geometry of the indenter used and the materials properties of the test piece and support.

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7 Procedure

7.1 The temperature of the test shall be recorded. Typically, tests are carried out in the range of ambient temperatures between 10 °C and 35 °C.

The temperature stability during a test is more important than the actual test temperature. Any calibration correction applied shall be reported along with the additional calibration uncertainty. It is recommended that tests, particularly in the nano and micro ranges, be performed in controlled conditions, in the range (23 ± 5) °C and less than 50 % relative humidity.

The individual test, however, shall be carried out at stable temperature conditions because of the requirement of high depth measuring accuracy. This means that:

- the test pieces shall have reached the ambient temperature before testing;
- the testing machine shall have reached a stable working temperature (operating manual should be consulted);
- other external influences causing temperature changes during individual test have been controlled.

To minimize thermally-induced displacement drift, the temperature of the testing machine shall be adequately maintained over the time period of one testing cycle, or a displacement drift correction shall be measured and applied (see 7.5 of this document and 4.4.3 of ISO 14577-2:2002). The uncertainty in the drift or in the drift correction shall be reported.

7.2 The test piece shall be firmly supported such that there is no significant increase in the testing machine compliance. The test piece shall either be placed on a support that is rigid in the direction of indentation, or fixed in a suitable test piece holder. The contact surfaces between test piece, support and test piece holder shall be free from extraneous matter which might increase the compliance (reduce the stiffness) of the test piece support.

7.3 The zero point for the measurement of the force/indentation depth curve shall be assigned individually to each test data set. It represents the first touch of the indenter with the test piece surface. The uncertainty in the zero-point shall be reported. The uncertainty in the assigned zero point shall not exceed 1 % of maximum indentation displacement for the macro and micro ranges. The zero point uncertainty for the nano range may exceed 1 % in which case the value shall be estimated and recorded in the test report.

Sufficient data points shall be recorded during the approach and first 10 % displacement of the increasing force/indentation depth curve, so that zero-point may be assigned with the permitted uncertainty. One of the two following example methods is recommended:

- a) Method 1: The zero-point is calculated by extrapolation of a fitted function (e.g. polynomial 2nd degree). The fit shall be applied to values within the range from zero to not more than 10 % of the maximum indentation depth. The uncertainty of the calculated zero point results from the fit parameters, the fitting function and the length of extrapolation.

The first part of the indentation curve (for instance up to 5 %) may be affected by vibration or other noise. The upper limit to the range of fit should be below the depth at which the contact response changes e.g. due to cracking or plastic yielding.

- b) Method 2: The zero-point is the touch point determined during the first increase of either the test force or the contact stiffness. At this touch point, the step size in force or displacement shall be small enough such that the zero point uncertainty is less than the limit required.

NOTE Typical small force steps values for the macro range are $10^{-4} F_{\max}$ and for the micro and nano range less than 5 μN .

7.4 The testing cycle shall be either force-controlled or indentation depth-controlled. The controlled parameters can vary either continuously or step by step. A full description of all parts of the testing cycle shall be stated in the report, including:

- a) the nature of the control (i.e. force or displacement control and whether a stepped or continuous change in the controlled parameters);
- b) the maximum force (or displacement);
- c) the force application (or displacement) rate;
- d) the length and position of each hold period;
- e) the data logging frequency (or number of data points).

NOTE Typical values are: e.g. force application and force removal time 30 s; hold time at maximum force 30 s; hold period to measure thermal drift 60 s at contact or after removal of 90 % of maximum force.

In order to obtain comparable test results the time taken for the test shall be taken into account.

7.5 The test force shall be applied without shock or vibration that can significantly affect the test results until either the applied test force or the indentation displacement attains the specified value. Force and displacement shall be recorded at the time intervals stated in the report.

During the determination of the touch point of the indenter with the test piece, the approach speed of the indenter should be low in order that the mechanical properties of the surface are not changed by the impact.

For micro range indentations it should not exceed 2 $\mu\text{m/s}$. Typical micro/nano range approach speeds are 10 nm/s to 20 nm/s or less during final approach.

NOTE At present the exact limit of the approach speed for the macro range is not known. It is recommended that users report the approach speed.