SLOVENSKI STANDARD
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Kovinski materiali - Instrumentirano vtiskanje pri preskušanju trdote in drugih lastnosti materialov - 1. del: Preskusna metoda (ISO/DIS 14577-1:2024)

Metallic materials - Instrumented indentation test for hardness and materials parameters - Part 1: Test method (ISO/DIS 14577-1:2024)

Metallische Werkstoffe - Instrumentierte Eindringprüfung zur Bestimmung der Härte und anderer Werkstoffparameter - Teil 1: Prüfverfahren (ISO/DIS 14577-1:2024)

Matériaux métalliques - Essai de pénétration instrumenté pour la détermination de la dureté et de paramètres des matériaux - Partie 1: Méthode d'essai (ISO/DIS 14577-1:2024)

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

Part 1: **Test method**

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

Partie 1: Méthode d'essai

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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.

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).

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

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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 third edition cancels and replaces the second edition (ISO 14577-1:2015), 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*

— *Part 5: Linear elastic dynamic instrumented indentation testing (DIIT)*

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.

Although the indentation modulus (E_{IT}) value obtained in this test method is not directly equivalent to Young's modulus or the orientation specific elastic modulus of the material indented, E_{IT} is, however, equivalent to the isotropic Hill average of the elastic plane strain modulus of the material when there is no pile up and no residual stress.

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 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 following three ranges:

— macro range: 2 $N \leq F \leq$ 30 kN;

— micro range: 2 $N > F$; $h >$ 0,2 µm;

— nano range: $h \leq$ 0,2 µm.

For the nano range, the mechanical deformation strongly depends on the real shape of indenter tip and the calculated material 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.

The macro and micro ranges 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 material parameters is given in Annex A.

At high contact pressures, damage to the indenter is possible. For test pieces with very high hardness and modulus of elasticity, permanent indenter deformation can occur and can be detected using suitable reference materials. Indentations that result in damage or permanent deformation of the indenter are excluded from the scope of this test method.

This test method can also be applied to thin metallic and non-metallic coatings and non-metallic materials. In this case, it is recommended that the specifications in the relevant standards be taken into account (see also 6.3 and ISO 14577-4).

The analysis methods used in this standard do not take into account the effects of residual stress and indentation pile up or sink in the test piece material, both of which cause an offset to the indentation response that changes the calculated measured value.

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

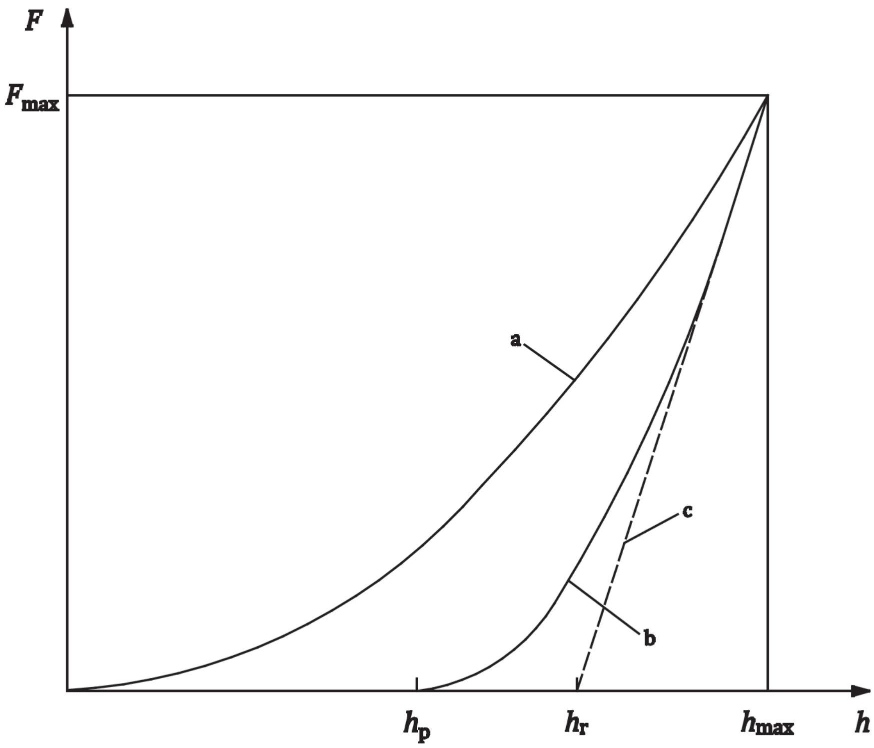
ISO/IEC Guide 98-3:2008, *Uncertainty of measurement — Part 3: Guide to the expression of uncertainty in measurement (GUM:1995)*

3 Symbols and designations

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

Table 1 — 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	%
C_T	Total measured compliance of the contact (dh/dF tangent to the force removal curve at maximum test force)	nm/mN
C_F	Machine compliance	nm/mN
C_S	Compliance of the contact after correction for machine compliance	nm/mN
E_{IT}	Indentation modulus of the test piece	GPa
E_r	Reduced plane strain modulus of the contact (combination of test piece and indenter plane strain moduli)	GPa
F	Test force	N
F_{max}	Maximum test force	N
H	Indentation depth under applied test force	mm
h_c	Contact depth 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 as identified on Figure 1	mm
H_{IT}	Indentation hardness	GPa
HM	Martens hardness	GPa
HM_s	Martens hardness, determined from the slope of the increasing force/indentation depth curve	GPa
HM_{diff}	Martens hardness, determined from the first derivative of h vs $\sqrt{F}$	GPa
ν_s	Poisson's ratio of the test piece	
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	Cone semi-angle or angle of facet to the indentation axis for pyramidal indenters	°
θ	Maximum angle between the contact surface and the indenter for calculation of radial displacement	°
η_{IT}	Ratio 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 The continued use of the unit N/mm^2 is allowed. 1 MPa = 1 N/mm^2.		



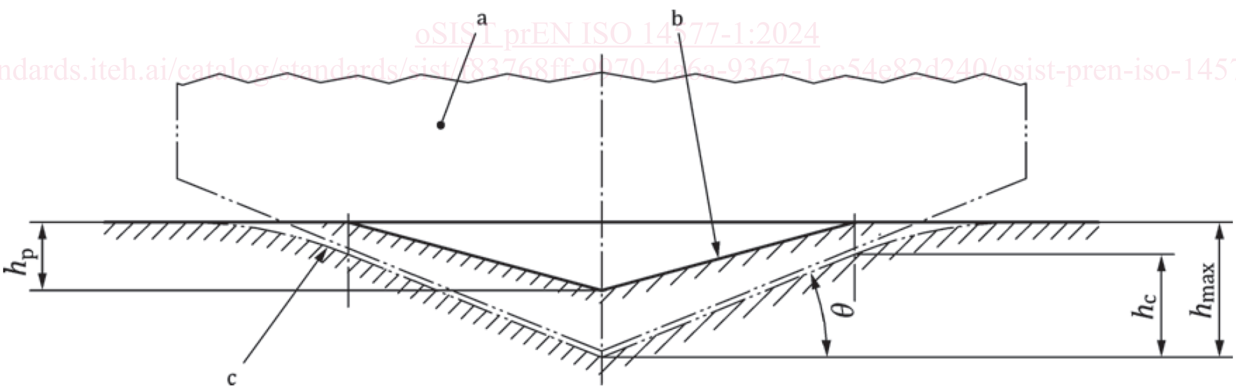
Key

a application of the test force

b removal of the test force

c tangent to curve b at F_{max}

Figure 1 — Schematic representation of the test procedure



Key

a indenter

b surface of residual plastic indentation in a test piece that has a "perfectly plastic" response

c surface of test piece at maximum indentation depth and test force

θ maximum angle between the test piece surface and the indenter

Figure 2 — Schematic representation of the cross section of indentation in the case of material "sink-in"

4 Principle

Continuous recording of the force and the depth of indentation permits 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 shall be used (i.e. the indenter shall not permanently deform and shall not be damaged as a result of indentation). The following shapes and materials can be used:

a) diamond indenter shaped as an orthogonal pyramid with a square base and with an angle α = 68° between the axis of the diamond pyramid and one of the faces (Vickers pyramid; see Figure A.1);

b) diamond pyramid with triangular base (e.g. modified Berkovich pyramid with an angle α = 65,27° between the axis of the diamond pyramid and one of the faces; see Figure A.1);

c) ball indenter (especially for the determination of the elastic behaviour of materials);

d) diamond spherical tipped conical indenter.

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

NOTE Due to the crystal structure of diamond, indenters that are intended to be spherical are often polyhedrons and do not have an 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 depths 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

— 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), and

— 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 segment b of the curves in Figure B.1 a) and Figure B.2 b) or in Figure B.1 b) and Figure B.2 a).

5 Testing machine

5.1 The testing machine shall have the capability of applying predetermined test forces or displacements within the required scope and shall fulfil 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 and ISO 14577-2, 4.5 and 4.6).

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

5.5 The testing machine shall operate at a temperature within the permissible range specified in 7.1 and shall maintain its calibration within the limits specified in ISO 14577-2, Clause 4.

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, in which case, this shall be described in detail in the test report. Care shall be taken that extraneous matter (e.g. dust particles) is not incorporated into the contact.

Generally, provided the surface is free from obvious surface contamination, cleaning procedures should be avoided. If cleaning is required, it shall be limited to the following methods to minimize damage:

— application of a dry, oil-free, filtered gas stream;

— application of a subliming particle stream of CO_2 (but keeping the surface temperature above the dew point);

— rinsing with a solvent (which is chemically inert to the test piece) and then setting it to dry.

If these methods fail and the surface is sufficiently robust, wipe the surface with a lint-free tissue soaked in solvent to remove trapped dust particles, after which, the surface shall be rinsed in a solvent as above.

Ultrasonic methods are known to create or increase damage to surfaces and coatings and should only be used with caution.

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 piece surfaces shall be normal to the test force direction. It is recommended that the angle difference between test surface normal and indenter axis is less than 1°. The effect of surface tilt should be included in the uncertainty calculation.

6.2 If necessary, preparation of the test surface shall be carried out in such a way that any alteration of the surface hardness and/or surface residual stress (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. When polishing is needed, a polishing process that is suitable for the particular materials shall be used.

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. For hardness and modulus measurements, the test piece thickness shall be at least 10 times the indentation depth or 3 times the indentation diameter (see 7.7), whichever is greater. For modulus measurements a sample thickness larger than 30 times the indentation depth is recommended. For measurements of thinner samples, it is recommended to follow the analysis described in ISO 14577-4 for coatings.

When testing coatings, the coating thickness should be considered as the test piece thickness. For testing thin samples or coatings, see ISO 14577-4 for better testing procedures.

NOTE The above are empirically based limits based on hardness testing. The effect of test piece thickness on the elastic modulus result is not the same as the effect on the hardness result. The exact limits of influence of support on test piece depend on the geometry of the indenter used and the materials properties of the test piece and support.

7 Procedure

7.1 The temperature of the test shall be recorded. Tests shall be performed within ±10 °C of the temperature of the instrument calibration.

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 (45 ± 10) % relative humidity.

The individual tests, 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),

— the ambient, instrument, and test temperature shall be within the range for which the machine calibration is valid, and

— 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 shall be measured and corrected. A decision tree to assist in estimating the drift during the experiment is shown in Figure 3. If the drift rate is significant, the displacement data shall be corrected by measuring the drift rate during a hold at an applied force as close to zero force as is practicable or during a hold at a suitable place in the force removal curve (see Annex G and ISO 14577-2 4.3.3). In all cases where the drift rate would influence the maximum indentation depth by more than 1 % within the total indentation cycle time, the drift rate shall be corrected (see tolerances in ISO 14577-2 Table 2 and 4.3.3). If a contact in the fully elastic regime can be obtained, a hold at initial contact is preferred. In this way, material influences (creep, visco-plasticity, cracking) can be minimized. When the measurement is significantly longer than the hold period for thermal drift measurement it is recommended to measure the thermal drift before and after the experiment and to correct the displacement using the average of the two drift rates. The difference between the two drift rates is an estimate of the drift rate uncertainty. The uncertainty due to the drift, or in the drift correction used, shall be reported.

To determine the drift of surface referenced instruments, contact between the reference indenter and the sample surface shall be elastic. This may be determined by calculation.

NOTE The significance of a particular drift rate upon the measurement depends upon the range of the indentation and the application.