INTERNATIONAL STANDARD

ISO 14577-1

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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

Reference number
ISO 14577-1:2002(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 14577-1 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*:

— *Part 1: Test method*

— *Part 2: Verification and calibration of testing machines*

— *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.

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 N $\leqslant F \leqslant$ 30 kN	2 N $> F$; $h >$ 0,2 µm	$h \leqslant$ 0,2 µm

[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.

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).

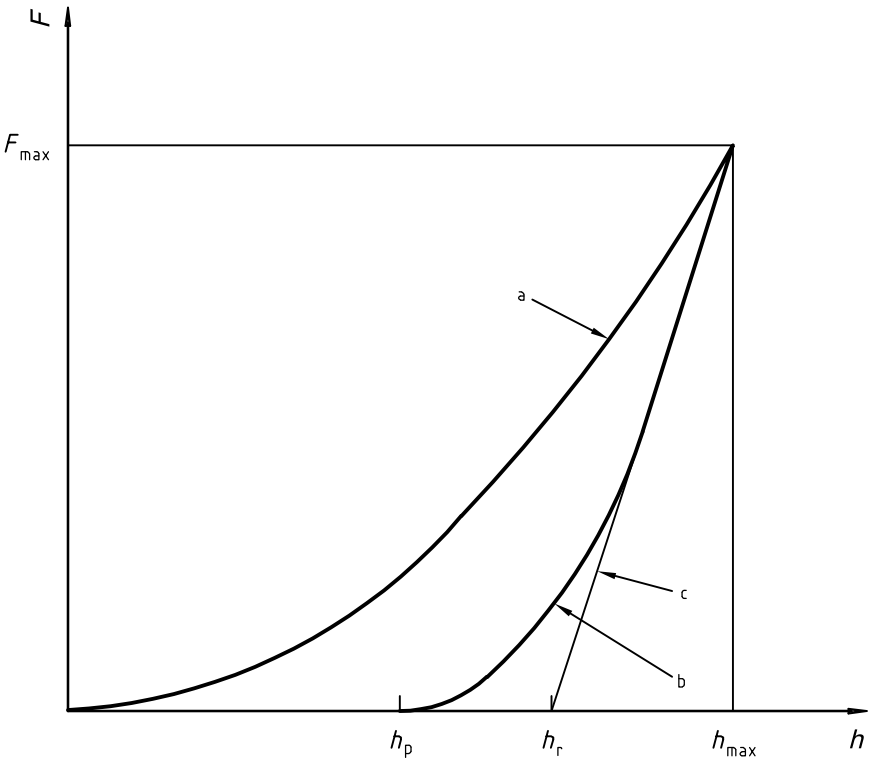
Table 2 — Symbols and designations

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.

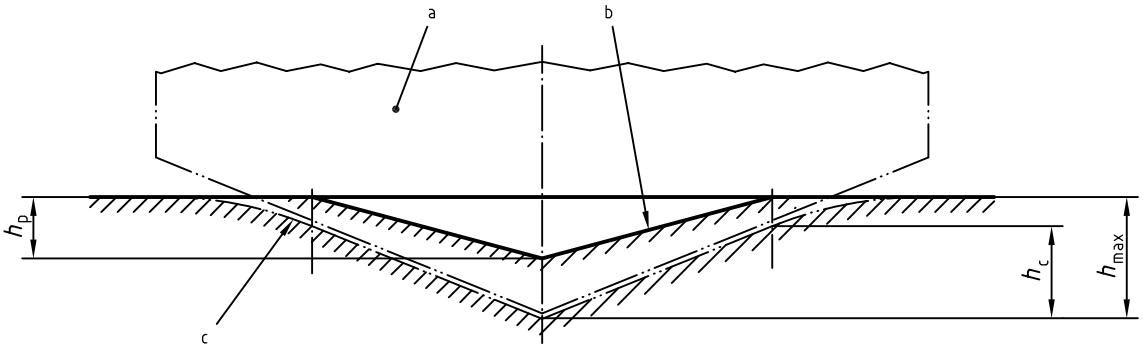


a Application of the test force.

b Removal of the test force.

c Tangent to curve b at F_{max}.

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 α = 136° 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 × the indentation depth or 3 × 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.

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 µm/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.

Force/indentation depth/time data sets are only comparable if the same test cycle (profile) is used. The test profile shall be specified in terms of either applied test force or indentation displacement as a function of time. The two most common cycles are:

a) constant applied test force rate;

b) constant indentation displacement rate.

The rate of applied test force removal is optional, subject to the requirement that sufficient data points be recorded during applied test force removal for any subsequent analysis.

The measurement drift rate shall be determined for each test cycle.This may be done for the micro and nano range by inserting hold periods after touching or at a convenient point in the applied test force removal procedure (typically at 10 % to 20 % of maximum test force).

In the macro range the measurement drift rate may be inferred from temperature data and knowledge of the instrument's drift response.

The force and depth data should be corrected by use of the measured drift rate.

A hold period at maximum applied test force may also be used to measure and/or ensure completion of time-dependent deformation before removal of applied test force commences.

7.6 Throughout the test the testing machine shall be protected from shock and vibration, air movements and variations in temperature that can significantly influence the test result.

7.7 It is important that the test results are not affected by the presence of an interface, free surface or by any plastic deformation introduced by a previous indentation in a series. The effect of any of these depends on the indenter geometry and the materials properties of the test piece. Indentations shall be at least three times their indentation diameter away from interfaces or free surfaces and the minimum distance between indentations shall be at least five times the largest indentation diameter.

The indentation diameter is the in-plane diameter at the surface of the test piece of the circular impression of an indent created by a spherical indenter. For non-circular impressions, the indentation diameter is the diameter of the smallest circle capable of enclosing the indentation. Occasional cracking may occur at the corners of the indentation. When this occurs, the indentation diameter should enclose the crack.

NOTE The minimum distances specified are best applicable to ceramic materials and metals such as iron and its alloys. For other materials it is recommended that separations of at least ten indentation diameters be used.

If in doubt, it is recommended that the values from the first indentation are compared with those from subsequent indentations in a series. If there is a significant difference, the indentations may be too close and the distance should be increased. A factor of a two-fold increase is suggested.

8 Uncertainty of the results

A complete evaluation of the uncertainty shall be carried out in accordance with ISO GUM.

Uncertainty of the results is a combination of uncertainties from a number of sources. These may be separated into two categories:

a) Type A uncertainties include:

— zero point assignation;

— measurement of force and displacement (including effects of ambient vibrations and magnetic field strength changes);

— fitting of the force-removal curve;

— thermal drift rate;

— contact area due to surface roughness.

b) Type B uncertainties include:

— force, displacement;

— testing machine compliance;

— indenter area function calibration values;

— calibration drift due to uncertainty in temperature of testing machine and time since last calibration;

— tilt of test surface.

It may not always be possible to quantify all the identified contributions to the random uncertainty. In this case, an estimate of Type A standard uncertainty may be obtained from the statistical analysis of repeated indentations into the test material. Care should be taken that Type B standard uncertainties which might contribute to the Type A standard uncertainty are not counted twice (see clause 4 of GUM:1995).

9 Test report

The test report shall include the following information:

a) reference to this part of ISO 14577, i.e. ISO 14577-1;

b) all details necessary for identifying the test piece;

c) material and shape of the indenter and, where used, the detailed area function of the indenter;

d) testing cycle (control method and full description of the cycle profile); this should include:

   1) set point values;

   2) rates and times of force or displacement;

   3) position and length of hold points;

   4) data logging frequency or number of points logged for each section of the cycle;

e) the result obtained, the total expanded uncertainty and the number of tests;

f) the method applied for the determination of the zero-point;

g) all operations not specified by this part of ISO 14577, or regarded as optional;

h) details of any occurrence which may have affected the results;

i) temperature of the test;

j) date and time of test;

k) analysis methods;

l) if required, all agreed additional information including determined values from the measured force/indentation depth curve and detailed information about the uncertainty budget.

NOTE It is frequently desirable to describe in the test report, the location of the indentation on the test piece.

Annex A
(normative)

Materials parameters determined from the force/indentation depth data set

A.1 General

Instrumented indentation force/indentation depth data sets may be used to derive a number of materials parameters.

A.2 Martens hardness HM [2)]

A.2.1 Determination of Martens hardness HM

Martens hardness is measured under applied test force. Martens hardness is determined from the values given by the force/indentation depth curve during the increasing of the test force, preferably after reaching the specified test force. Martens hardness includes the plastic and elastic deformation, thus this hardness value can be calculated for all materials.

Martens hardness is defined for both pyramidal indenters shown in Figure A.1. It is not defined for the Knoop indenter or for ball indenters.

Martens hardness is defined as the test force F divided by $A_s(h)$ the surface area of the indenter penetrating beyond the zero-point of the contact and is expressed in N/mm^2.

a) Vickers indenter

$$\mathrm{HM} = \frac{F}{A_s(h)} = \frac{F}{26{,}43 \times h^2} \qquad \text{(A.1)}$$

$$A_s(h) = \frac{4 \times \sin\left(\frac{\alpha}{2}\right)}{\cos^2\left(\frac{\alpha}{2}\right)} \times h^2 \qquad \text{(A.2)}$$

b) Berkovich indenter

$$\mathrm{HM} = \frac{F}{A_s(h)} = \frac{F}{26{,}43 \times h^2} \qquad \text{(A.1)}$$

$$A_s(h) = \frac{3 \times \sqrt{3} \times \tan\alpha}{\cos\alpha} \times h^2 \qquad \text{(A.2)}$$

For indentation depth < 6 µm the area function of the indenter cannot be assumed to be that of the theoretical shape, since all pointed indenters will have some degree of rounding at the tip and spherically-ended indenters (spherical and conical) are unlikely to have a uniform radius. The determination of the exact area function for a given indenter is particulary important for these indentation depths, but is beneficial for all indentation depths (see 4.2.1 and 4.6 of ISO 14577-2:2002).

For indentation depth < 6 µm the real surface area $A_s(h)$ shall be used for the calculation, see annex C and [3].

The test forces 1 N; 2,5 N; 5 N and 10 N and their decimal multiples should be chosen for easy comparison of hardness values.

2) Former designation Universal hardness HU, see [2].

For certain applications it may be useful to hold the specified test force over a specific time interval. The duration of the hold period of the test force should be documented to an accuracy of 0,5 s. The field of application for Martens hardness is given in Figure A.2.

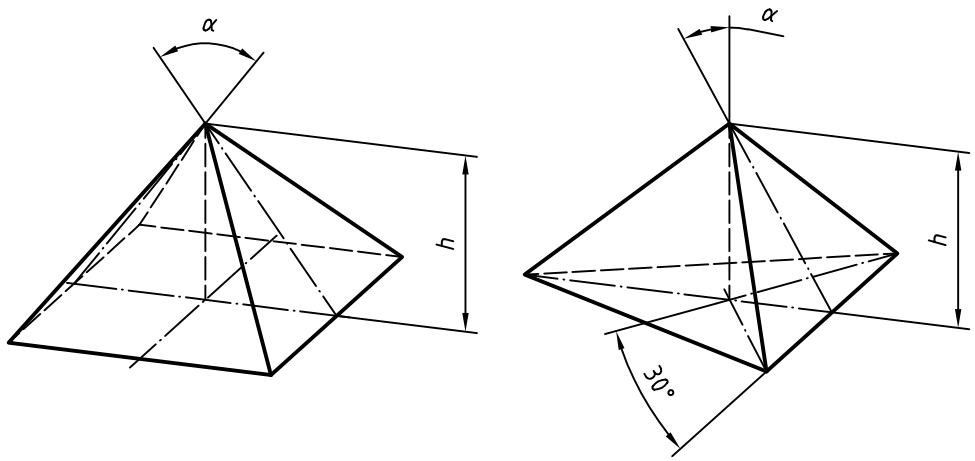
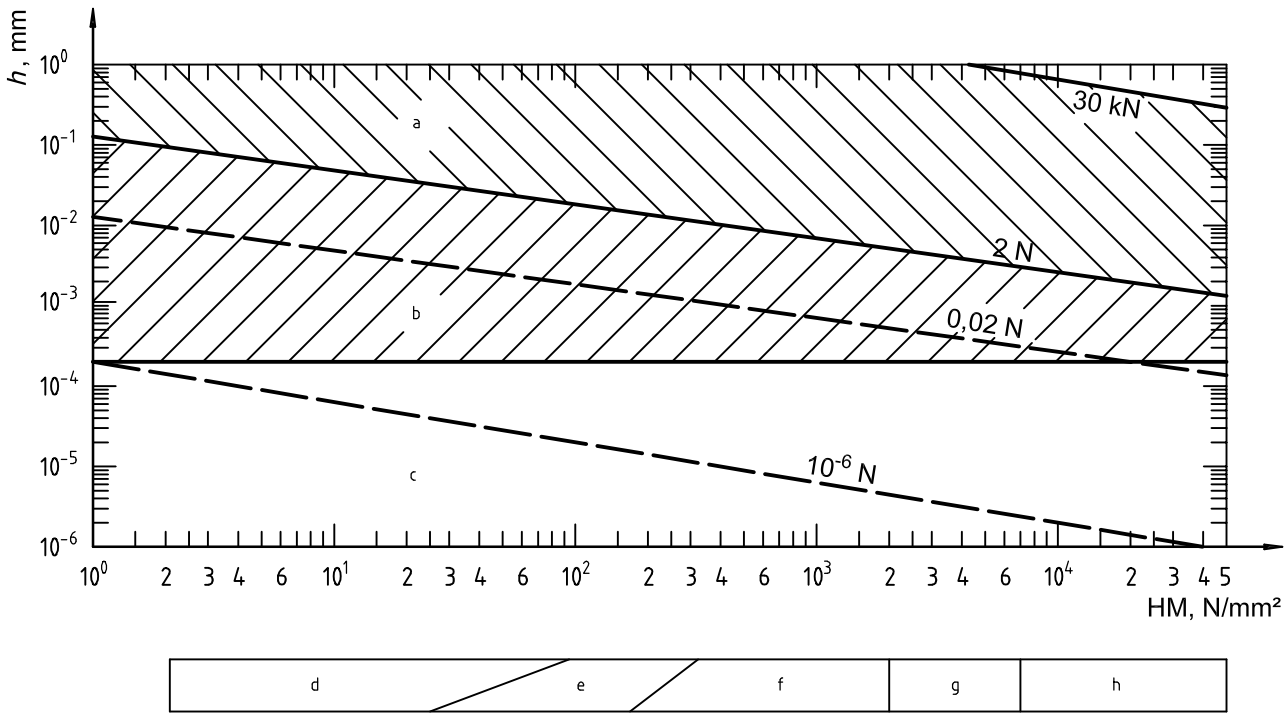


Figure A.1 — Shape of indenters for determination of HM

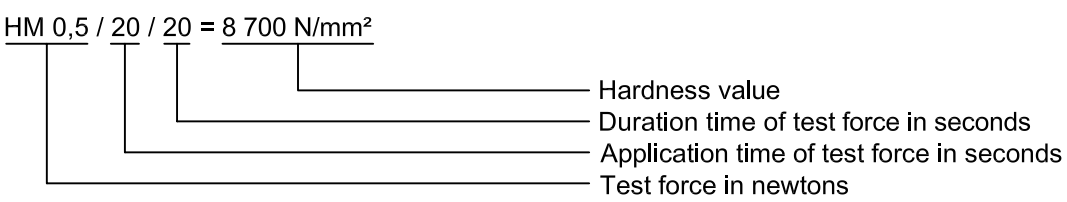


a Macro range
b Micro range
c Nano range
d Rubber
e Plastics
f Non-ferrous metals
g Steel
h Hardmetals, ceramics

Figure A.2 — Relationship between Martens hardness, indentation depth and test force

A.2.2 Designation of Martens hardness HM

EXAMPLE



A.3 Martens hardness, determined from the slope of the increasing force/indentation depth curve, HM_S

A.3.1 Determination of Martens hardness, HM_s

The method for the determination of the Martens hardness given by the slope of the increasing force/indentation depth curve, has the advantage of avoiding the zero-point determination for homogeneous materials.

For homogeneous materials (dimension of the inhomogenities in the region of surface is small in relation to the indentation depth) the following equation is at least partly valid (preferring pairs between 50 % F_{max} and 90 % F_{max}) for the force/indentation depth curve:

$$h = m \times \sqrt{F} \tag{A.3}$$

The slope m can be determined by a linear regression of the equation A.3. In this case it is possible to determine the hardness by the following modified method from the force/indentation depth curve:

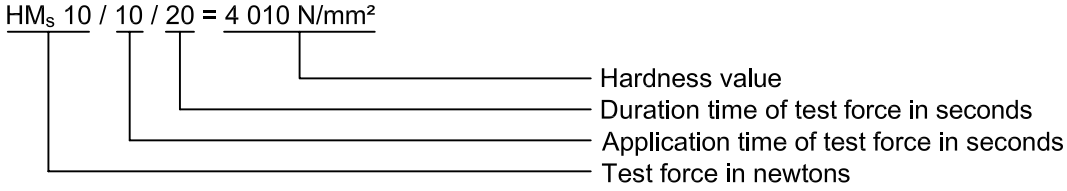
$$HM_s = \frac{1}{m^2 A_s(h)/h^2} \tag{A.4}$$

with

$$\frac{A_s(h)}{h^2} \quad \begin{cases} = 26{,}43 \text{ for Vickers indenter} \\ = 26{,}43 \text{ for Berkovich indenter} \end{cases}$$

A.3.2 Designation of Martens hardness, HM_s

EXAMPLE



NOTE The method for the determination of the Martens hardness determined from the slope of the increasing force/indentation depth curve has the advantage of independence of the uncertainty of the determination of the zero-point and the test piece roughness. There is also a lower influence of vibration at the location of the testing machine on the test results. For test pieces exhibiting a variation in hardness as a function of indentation depth, the determined hardness values HM_s will deviate from HM determined as in A.1.

A.4 Indentation hardness H_{IT}

A.4.1 Determination of indentation hardness H_{IT}

Indentation hardness H_{IT} is a measure of the resistance to permanent deformation or damage.

$$H_{IT} = \frac{F_{max}}{A_p} \tag{A.5}$$

where

F_{max} is the maximum applied force

A_p is the projected (cross-sectional) area of contact between the indenter and the test piece determined from the force-displacement curve and a knowledge of the area function of the indenter. See 4.6.2 of ISO 14577-2:2002.

Equation (A.5) defines hardness as the maximum applied force, divided by the projected (cross-sectional) contact area of the indenter with the testpiece. This definition is in accord with that generally agreed and first proposed by Meyer[4].

For indentation depth < 6 µm the area function of the indenter cannot be assumed to be that of the theoretical shape, since all pointed indenters will have some degree of rounding at the tip and spherically-ended indenters (spherical and conical) are unlikely to have a uniform radius. The determination of the exact area function for a given indenter is required for indentation depths < 6 µm, but is beneficial for larger indentation depths (see 4.2.1 and 4.6 of ISO 14577-2:2002).

NOTE 1 The area function is normally expressed as a mathematical function relating the projected area to the distance from the tip of the indenter. Where a relatively simple (cubic or polynomial) mathematical function is unable to describe the area function then an estimate may be made either graphically or by using a look-up table. Alternatively a different mathematical function can be used to describe different parts of the indenter or a spline function adopted.

For indentation depth > 6 µm a first approximation to the projected area, A_p, is given by the theoretical shape of the indenter.

For a Vickers indenter:

$A_p = 24{,}50 \times h_c^2$

For a perfect Berkovich indenter:

$$A_p = 23{,}96 \times h_c^2$$

For a modified Berkovich indenter (see 4.2.3 of ISO 14577-2:2002):

$$A_p = 24{,}50 \times h_c^2$$

where

h_c is the depth of contact of the indenter with the test piece calculated as follows:

$$h_c = h_{max} - \varepsilon\,(h_{max} - h_r)$$

Figure 2 schematically shows the different depths monitored during an indentation experiment. The theoretical basis of the method for the determination of contact depth is given in [5]. The contact depth is derived from the force removal curve using the tangent depth, h_r, and the maximum displacement, h_{max}, correcting for elastic displacement of the surface according to Sneddon's analysis[6], where ε depends on the indenter geometry (see Table A.1).

Table A.1 — Correction factor ε for different indenter geometries

Indenter Geometry	ε
Flat punch	1
Conical	$2(\pi - 2)/\pi = 0{,}73$
Paraboloid of revolution (includes spherical)	3/4
Berkovich, Vickers	3/4

h_r is derived from the force-displacement curve and is the intercept of the tangent to the unloading cycle at F_{max} with the displacement axis. Different methods have been used for the determination of h_r and can be essentially described by two approaches.

a) Linear Extrapolation Method (see [11]): this assumes that the first portion of the unloading curve is linear and simply extrapolates that linear portion to intercept the displacement axis.

NOTE 2 This method may be a good approximation for ductile materials e. g. aluminium, tungsten.

b) Power Law Method (see [5]): this recognizes the fact that the first portion of the removal curve of the test curve may not be linear, and can be described by a simple power law relationship:

$$F = K(h - h_p)^m$$

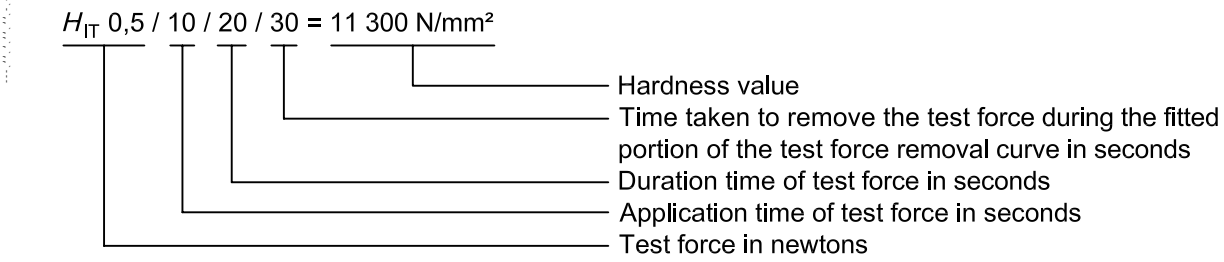
where K is a constant and m is an exponent which depends on indenter geometry.

Normally the upper 80 % of the removal curve of the test force is taken for the least-square fitting procedure, but this can be varied according to the "quality" of the unloading curve. If it is necessary to restrict the fit to 50 % or less, the indentation experiment shall be considered to be abnormal and care should be taken in interpretation. The tangent is found by differentiating the unloading curve and evaluating at F_{max}. The intercept of this tangent with the displacement axis yields h_r.

Information concerning the correlation of H_{IT} to other hardness scales is contained in annex F.

A.4.2 Designation of indentation hardness H_{IT}

EXAMPLE



A.5 Indentation modulus E_{IT}

A.5.1 Determination of indentation modulus E_{IT}

The indentation modulus E_{IT} can be calculated from the slope of the tangent for the calculation of indentation hardness H_{IT} (see A.4) and is comparable with the Young's modulus of the material. Significant differences between the indentation modulus E_{IT} and Young's modulus can occur, however, either pile-up or sink-in are present.

$$E_{IT} = \frac{1-(\nu_s)^2}{\frac{1}{E_r} - \frac{1-(\nu_i)^2}{E_i}} \tag{A.6}$$

$$E_r = \frac{\sqrt{\pi}}{2C\sqrt{A_p}} \tag{A.7}$$

where

ν_s is the Poisson's ratio of the test piece;

ν_i is the Poisson's ratio of the indenter (for diamond 0,07) (e.g., in [7]);

E_r is the reduced modulus of the indentation contact;

E_i is the modulus of the indenter (for diamond $1{,}14 \times 10^6$ N/mm^2) (e.g., in [7]);

C is the compliance of the contact, i.e. dh/dF of the test force removal curve evaluated at maximum test force (reciprocal of the contact stiffness);

A_p is the projected contact area, value of the indenter area function at the contact depth defined in accordance with 4.6 of ISO 14577-2:2002.

For $h > 6$ µm the following are valid:

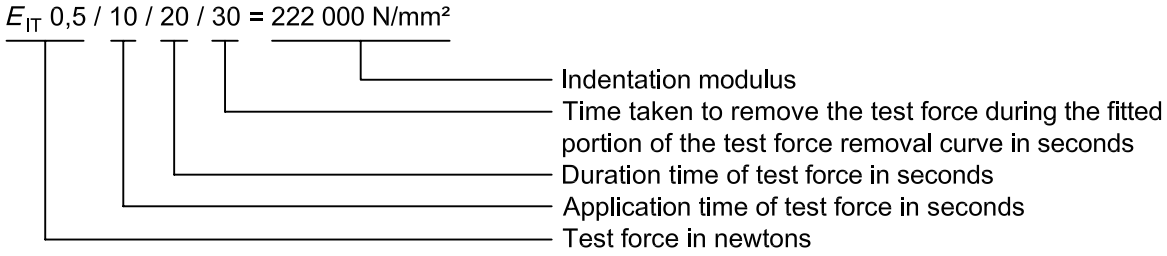
$\sqrt{A_p}$ = 4,950 × h_c for the Vickers indenter and the modified Berkovich indenter

= 4,895 × h_c for the Berkovich indenter

NOTE Equation (A.7) is based on an assumption of axis symmetric contact area. Correction for pyramidal indenters have been proposed in [15].

A.5.2 Designation of indentation modulus E_{IT}

EXAMPLE



NOTE For some materials a correlation between E_{IT} and tabular values for the Young's modulus of metals and metal alloys is demonstrated [8], [9].

A.6 Indentation creep C_{IT}

A.6.1 Determination of indentation creep C_{IT}

If the change of the indentation depth is measured with constant test force, a relative change of the indentation depth can be calculated. This is a value for the creep of the material [see Figures B.1 a), B.1 b) and A.3].

$$C_{IT} = \frac{h_2 - h_1}{h_1} \times 100 \qquad \text{(A.8)}$$

where

h_1 is the indentation depth at the time (t_1) of reaching the test force (which is kept constant) in millimetres;

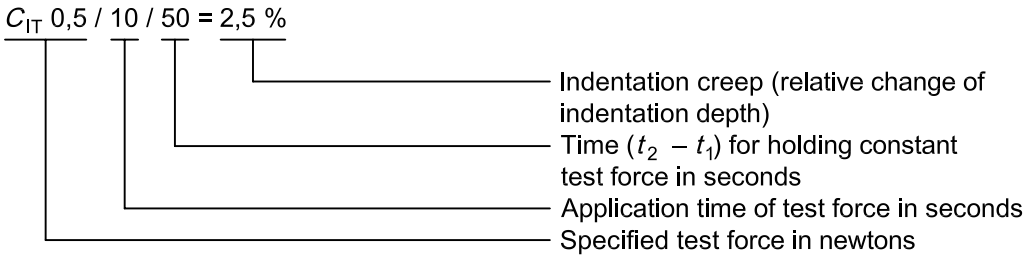
h_2 is the indentation depth at time (t_2) of holding the constant test force, in millimetres.

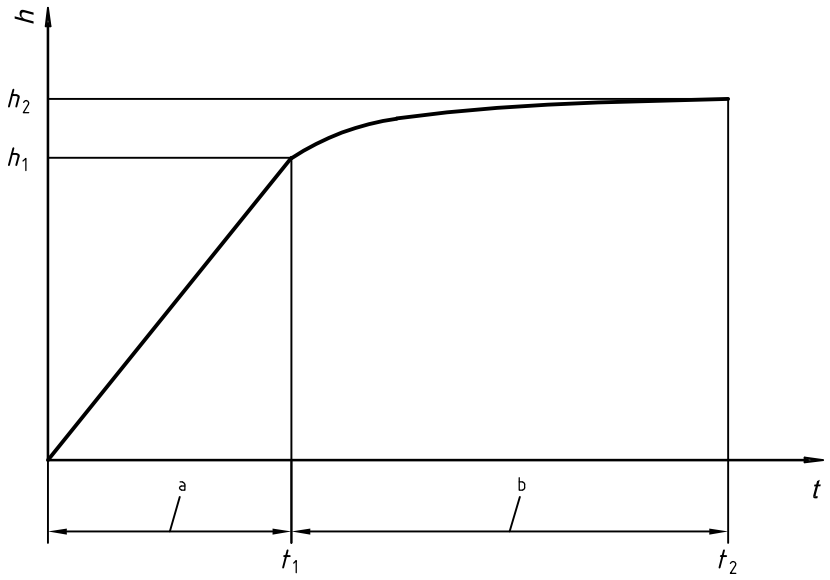
NOTE It should be noted that the creep data can be significantly influenced by thermal drift.

A.6.2 Designation of indentation creep C_{IT}

The relative change of the indentation depth (creep) is denoted by the symbol C_{IT}

EXAMPLE





a Application of the test force

b Test force kept constant from t_1 to t_2

Figure A.3 — Expression of indentation creep

A.7 Indentation relaxation R_{IT}

A.7.1 Determination of indentation relaxation R_{IT}

If the change of the test force is measured at a constant indentation depth a relative change of the test force can be calculated. This is a value for the relaxation of the material [see Figures B.2 a), B.2 b) and A.4].

$$R_{IT} = \frac{F_1 - F_2}{F_1} \times 100 \qquad \text{(A.9)}$$

where

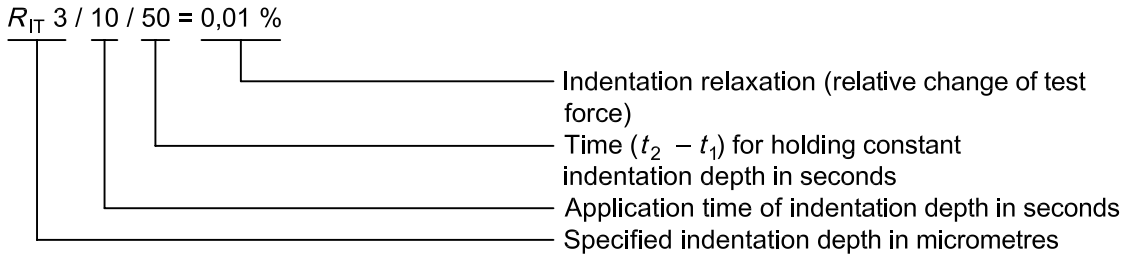
F_1 is the force at reaching the indentation depth which was kept constant, in N;

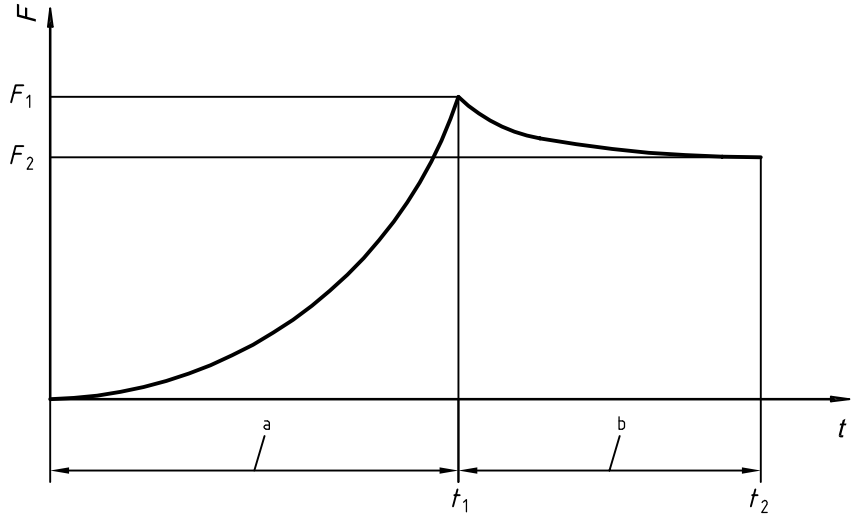
F_2 is the force after the time during which the indentation depth was kept constant, in N.

A.7.2 Designation of indentation relaxation R_{IT}

The relative change of test force (relaxation) is denoted by the symbol R_{IT}.

EXAMPLE





a Application of the indentation depth.

b Indentation depth kept constant from t_1 to t_2.

Figure A.4 — Expression of indentation relaxation

A.8 Plastic and elastic parts of the indentation work

A.8.1 Determination of plastic and elastic parts of the indentation work

The mechanical work W_{total} indicated during the indentation procedure is only partly consumed as plastic deformation work W_{plast}. During the removal of the test force the remaining part is set free as work of the elastic reverse deformation W_{elast}. According to the definition of the mechanical work as $W = \int F \mathrm{d}h$ both parts appear as different areas in Figure A.5. The relation

$$\eta_{IT} = \frac{W_{elast}}{W_{total}} \times 100 \tag{A.10}$$

contains information which is suitable for characterization of the test piece where

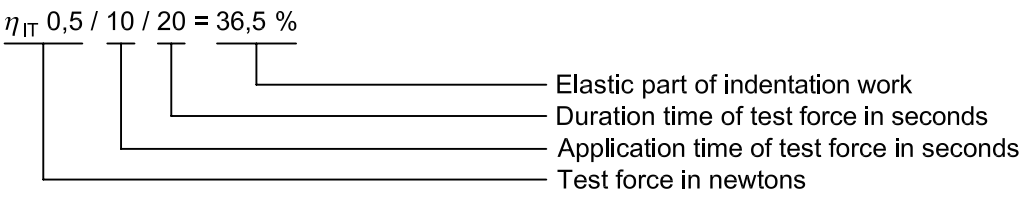
$$W_{total} = W_{elast} + W_{plast}$$

The plastic part W_{plast}/W_{total} follows as

$$100\ \% - \eta_{IT} \tag{A.11}$$

A.8.2 Designation of elastic part of indentation work η_{IT}

EXAMPLE



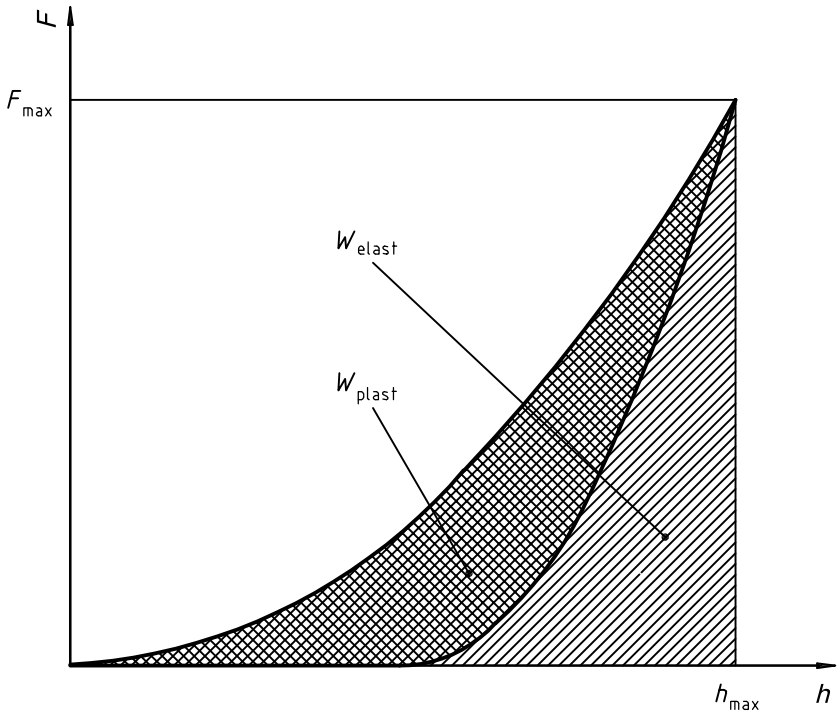
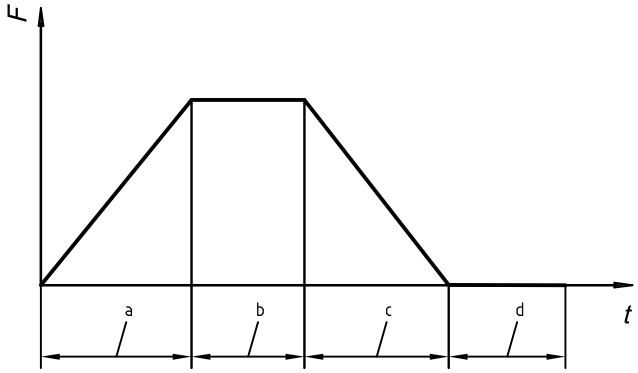


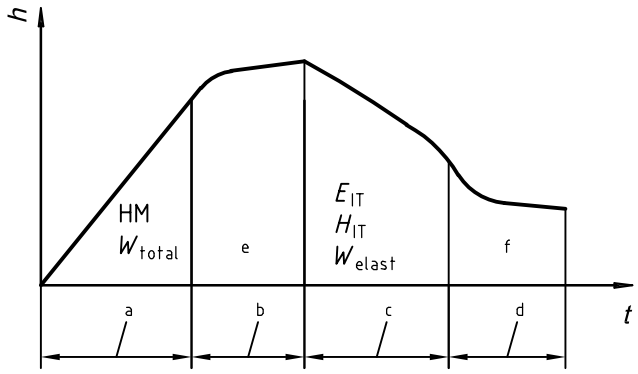
Figure A.5 — Plastic and elastic parts of the indentation work

Annex B
(informative)

Types of control use for the indentation process



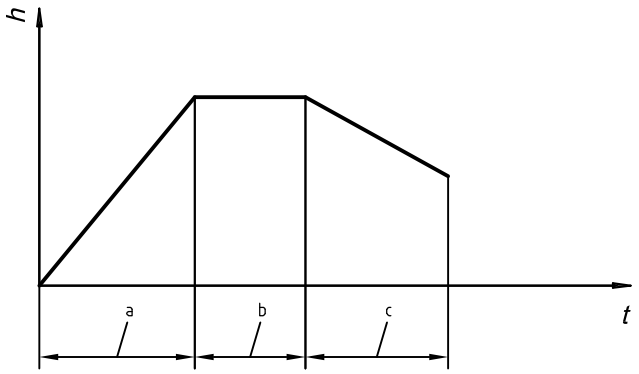
a)



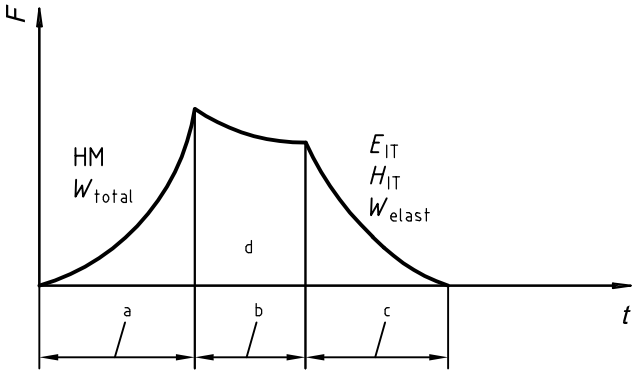
b)

- a Application of test force
- b Maximum test force
- c Removal of test force
- d Test force = 0 N
- e Indentation creep
- f Recovery at zero test force

Figure B.1 — Schematic representation of the test force controlled test procedure in dependence on time



a)



b)

- a Application of indentation depth
- b Maximum indentation depth
- c Decreasing of indentation depth
- d Relaxation under maximum indentation depth

Figure B.2 — Schematic representation of the indentation depth controlled test procedure in dependence on time

Annex C
(normative)

Machine compliance and indenter area function

C.1 Machine compliance

The applied test force not only acts on the test piece surface, it also acts on the parts of the testing machine and these are elastically deformed.

This elastic deformation causes an increase in the measured indentation depth which is not experienced at the indentation contact, but occurs between the reference planes in the testing machine.

Usually the additional indentation depth due to the test machine deformation is proportional to applied force. This additional compliance must be taken into account at all forces when indentation modulus is being measured as it acts directly to increase h_{max} and decrease the gradient of the tangent to the removal curve of the test force. The increase in the measured value of h_{max} is especially significant at high applied forces.

Procedures for the determination of the machine compliance in accordance with accepted methods (see 4.5 of ISO 14577-2:2002 and [11], [14]) shall be supplied by the manufacturer of the testing machine. Especially if the displacement measurement is bottom referenced, the machine compliance may influence the test results noticeably. The manufacturer of the machine shall determine the machine compliance before delivery.

C.2 Indenter area function

The calculation of the parameters described in A.2, A.4 and A.5 is based on the contact area (or projected area) of the indenter. However only the indentation depth is measured. Crucial differences can be found when comparing the actual contact area with the area calculated assuming an ideal indenter geometry, particularly at small measured indentation depths.

These differences are due to the rounding of the tip of the indenter — in the case of the Vickers pyramid, the line of conjunction (offset) and the deviation from the specified angle of the indenter, which are attributable to the usual production tolerances. Furthermore the actual contact area will be changed due to wear of the indenter tip.

To maintain the reproducibility of the results it is necessary to determine the actual contact area (or projected area) and use it in the calculation of the materials parameters.

Three methods of determining the area function are possible:

— a direct measurement method using a traceable atomic force microscope (AFM) (see [12]);

— indirectly by utilizing indentations into a material of known Young's modulus (see [11]);

— indirectly by observing the departure from the hardness calculated with the measured test force and the corresponding indentation depth (with the indentation depth independent hardness). This method needs special reference materials (e.g.: fused silica, BK7 glass). The method can be applied for indentation hardness as well as for the Martens hardness (indirectly by utilizing indentations into a material of known Young's modulus, see [13]). If this method is used for Martens hardness, the surface area function can be calculated from the increasing test force/indentation depth curve.

To determine the area function for the Martens hardness it is recommended to use reference materials with high plasticity.

For all indirect methods machine compliance should first be determined and the indentation depth data should be corrected accordingly. Alternatively, an iterative approach can be used.

The area function is normally expressed as a mathematical function relating the surface area or the projected area to the distance from the tip of the indenter. Where a relatively simple (cubic or polynomial) mathematical function is unable to describe the area function, an estimate may be made either graphically or by using a look-up table. Alternatively different mathematical functions can be used to describe different parts of the indenter, or a spline function used.

A procedure for the verification of the indenter area function is given in annex C of ISO 14577-2:2002.

NOTE Indentation area function and machine compliance correction can be determined simultaneously using an iterative procedure and multiple reference materials[14].

Annex D
(informative)

Notes on diamond indenters

Experience has shown that a number of initially satisfactory indenters can become defective, after a comparatively short time in use. This is due to small cracks, pits or other flaws in the surface. If such faults are detected in time, many indenters may be reclaimed by regrinding. If not, any small defects on the surface rapidly worsen and make the indenter useless.

Therefore:

— The condition of indenters should be monitored regularly for contamination or defects. For macro range indenters this can be achieved by inspection of the shape of an indentation into a reference block or routine test material as used in 6.3 of ISO 14577-2:2002.

— For micro and nano range indenters periodic optical inspection using a × 400 microscope is recommended for detection of contamination and gross defects.

— Detection of submicroscopic damage or contamination is possible by maintaining a good history or indirect verification and routine checking as in 6.2 and 6.3 of ISO 14577-2:2002 or by scanned probe microscopy of indentations or the indenter itself.

— The calibration certificate of the indenter is no longer valid when the indenter shows defects.

— Reground or otherwise repaired indenters should be recalibrated and recertified.

Contamination of the surface of the indenter may distort the test result. The source of contamination is most often testing on contaminated test pieces.

For micro and nano range indenters the cleaning procedure could be:

— Hold indenter firmly and indent into a freshly cleaved surface of expanded polystyrene several times. The plasticizers make a good solvent and the foam-like nature is unlikely to damage the tip of the diamond. Inspect using an optical microscope at × 400 or greater and indent gently into a small cotton wool ball soaked in acetone or water-free alcohol (e.g. high purity methanol or ethanol or iso-propanol) until there is no visible contamination.

— If contamination is not removed by repeating this process, indentation into either aluminium, glass or a clean wooden spatula may dislodge the contamination sufficiently for removal using the above cleaning procedure.

— Care should be taken not to subject the indenter to excessive normal and in particular lateral forces during the indentations as this could damage the indenter. One method is to use a sample that weighs less than the usual forces experienced by the indenter and gently and slowly lower the sample on to the upturned indenter, thereby limiting the maximum force to that of the sample weight.

Annex E
(normative)

Influence of the test piece surface roughness on the accuracy of the results

This annex is based on round-robin tests with Vickers indenters only.

Surface roughness causes an uncertainty in contact area due to asperity contact at very shallow indentation depths. At larger indentation depths the uncertainty in contact area is reduced and is most conveniently expressed as an uncertainty in indentation depth proportional to the arithmetic (mean deviation) surface roughness.

To maintain the contribution of surface roughness to the uncertainty in indentation depth below 5 %, h shall be at least 20 $\times$ the arithmetic mean deviation roughness Ra (see ISO 4287).

$$h \geqslant 20\ Ra \tag{E.1}$$

Table E.1 gives examples for surface roughness for different materials at different test forces.

Table E.1 — Examples for maximum permissible arithmetic surface roughness Ra for different test forces F

Examples of materials	Arithmetic mean deviation Ra for different test forces, µm			Martens hardness HM, N/mm^2
	0,1 N	2 N	100 N	
aluminium	0,13	0,55	4,00	600
steel	0,08	0,30	2,20	2 000
hardmetal	0,03	0,10	0,80	15 000

NOTE Round-robin-tests (see [10]) show that the standard deviation s_h of the indentation depth is approximately equal to the arithmetic roughness Ra. The requirement for uncertainty of $h < 5$ % leads to the minimum indentation depth.

For tests at the nano and lower limit of the micro range it may not be possible to meet the condition of formula (E.1) for higher hardness test pieces. To reduce the uncertainty in the mean value of the test result the number of tests may be increased. This should be stated in the test report.

It is recommended for tests in the nano and micro ranges that surface roughness of the indented contact area be measured or that this area be observed by suitable means. In many cases surface roughness may be inferred from comparison with test pieces of known roughness or from batch sampling of surface roughness. A visual inspection verifying a smooth, polished or 'mirror' finish is adequate for tests in the macro range.

Annex F
(informative)

Correlation of indentation hardness H_{IT} to Vickers hardness

Indentation hardness H_{IT} may be correlated to Vickers hardness HV for a wide range of materials by using a suitable scaling function.

CAUTION: Although H_{IT} may be correlated to HV in this way, any equivalent HV value so calculated should not be used as a substitute for HV.

A simple function may be derived for a perfect Vickers geometry indenter, or for a Vickers indenter where the projected area function is known. In this case measurements of hardness, H_{IT} are related to the Vickers Hardness numbers, HV, by a scaling factor. The ratio of projected area to surface area, at any particular distance from the tip of a perfect Vickers indenter, is a constant.

$$\frac{A_p}{A_s} = \frac{24{,}50}{26{,}43} = 0{,}927\,0 \tag{F.1}$$

The diagonal length d measured in conventional Vickers tests is related to A_p by:

$$d^2 = 2A_p;\ \mathrm{HV} = \frac{F \times A_p}{A_p \times A_s \times g_n} \tag{F.2}$$

thus

$$\mathrm{HV} = \frac{H_{IT} \times A_p}{g_n \times A_s} = 0{,}094\,5\ H_{IT} \tag{F.3}$$

where g_n acceleration due to gravity, typically 9,806 65 m/s^2.

For a Berkovich indenter, the following relationship exists:

$$\frac{A_p}{A_s} = \frac{23{,}96}{26{,}43} = 0{,}906\,5 \tag{F.4}$$

$$\mathrm{HV} = \frac{H_{IT} \times A_p}{g_n \times A_s} = 0{,}092\,4 \times H_{IT} \tag{F.5}$$

For a modified Berkovich indenter, the following relationship exists:

$$\frac{A_p}{A_s} = \frac{24{,}50}{26{,}97} = 0{,}908\,4 \tag{F.6}$$

$$\mathrm{HV} = \frac{H_{IT} \times A_p}{g_n \times A_s} = 0{,}092\,6 \times H_{IT} \tag{F.7}$$

Note that perfect indenter geometry is generally not maintained for small indentation depths (< 6 µm) and therefore this simple correlation may break down. The error induced by such an assumption will, in general, be most severe at small indentation depths.

For some materials a correlation between H_{IT} 1/10/20/30 and HV 0,1 is demonstrated in [5] and [8].

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