INTERNATIONAL STANDARD

ISO 14577-1

Second edition
2015-07-15

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 14577-1:2015(E)

Contents

Page

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).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

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

The committee responsible for this document is ISO/TC 164, *Mechanical testing of metals*, Subcommittee SC 3, *Hardness testing*.

This second edition cancels and replaces the first edition (ISO 14577-1:2002), which has been technically revised.

ISO 14577 consists of the following parts, under the general title *Metallic materials — Instrumented indentation test for hardness and materials parameters*:

— *Part 1: Test method*

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

— *Part 3: Calibration of reference blocks*

— *Part 4: Test method for metallic and non-metallic coatings*

Introduction

Hardness has typically been defined as the resistance of a material to permanent penetration by another harder material. The results obtained when performing Rockwell, Vickers, and Brinell tests are determined after the test force has been removed. Therefore, the effect of elastic deformation under the indenter has been ignored.

ISO 14577 (all parts) has been prepared to enable the user to evaluate the indentation of materials by considering both the force and displacement during plastic and elastic deformation. By monitoring the complete cycle of increasing and removal of the test force, hardness values equivalent to traditional hardness values can be determined. More significantly, additional properties of the material, such as its indentation modulus and elasto-plastic hardness, can also be determined. All these values can be calculated without the need to measure the indent optically. Furthermore, by a variety of techniques, the instrumented indentation test allows to record hardness and modulus depth profiles within a, probably complex, indentation cycle.

ISO 14577 (all parts) has been written to allow a wide variety of post-test data analysis.

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

Part 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 this reason in the macro range, hardmetal indenters are often used. For test pieces with very high hardness and modulus of elasticity, permanent indenter deformation can occur and can be detected using suitable reference materials. It is necessary that its influence on the test result be taken into account.

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).

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 14577-2:2015, *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

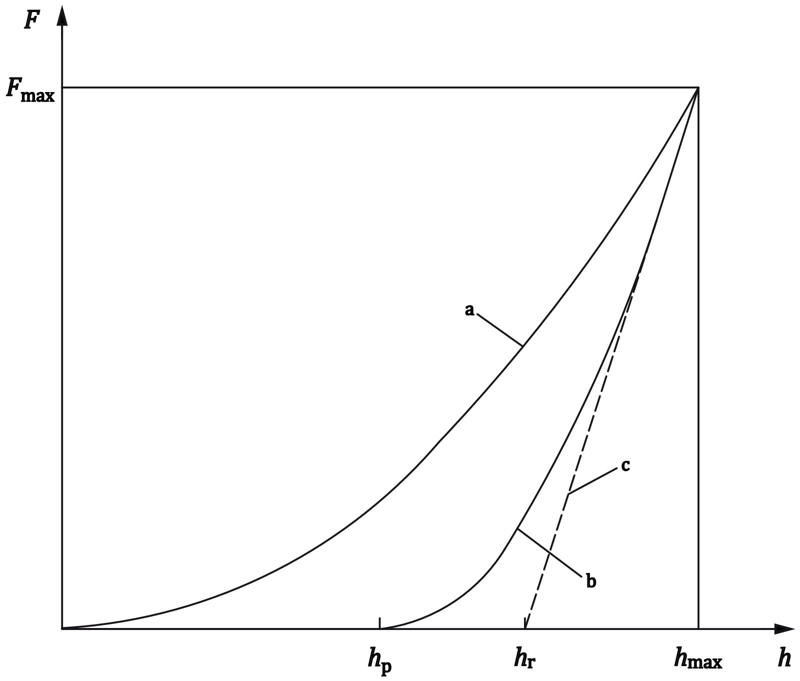
For the purposes of this document, the symbols and designations in Table 1 shall be applied (see also Figure 1 and Figure 2).

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	Instrument 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	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 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.



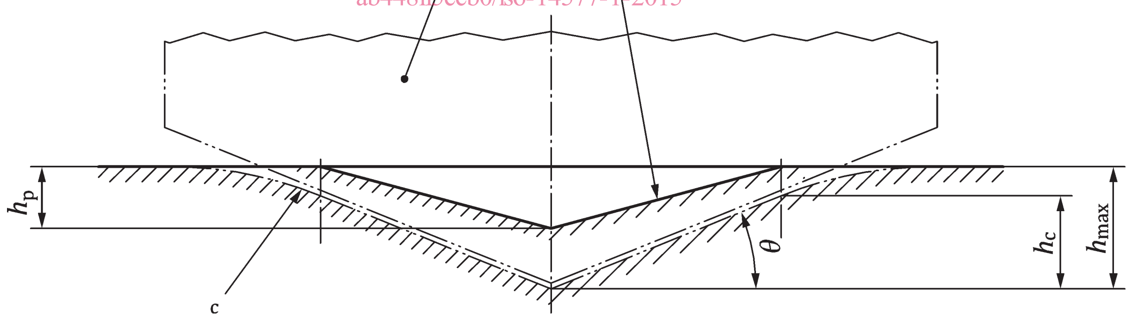
Key

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. 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) hardmetal ball (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 in 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 14577-2:2015, 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 14577-2:2015, 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 surfaces shall be normal to the test force direction. It is recommended that the test surface tilt is less than 1°. Tilt should be included in the uncertainty calculation.

6.2 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. 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. 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 as the test piece thickness. For testing coatings, see ISO 14577-4.

NOTE The above are empirically based limits. 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. 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 (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 ISO 14577-2:2015, Annex G 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. The uncertainty due to the drift, or in the drift correction used, shall be reported.

NOTE To determine the drift of surface referenced instruments, elastic contact between the reference and the surface is sufficient; contact of the indenter with the surface is not required and is not recommended.

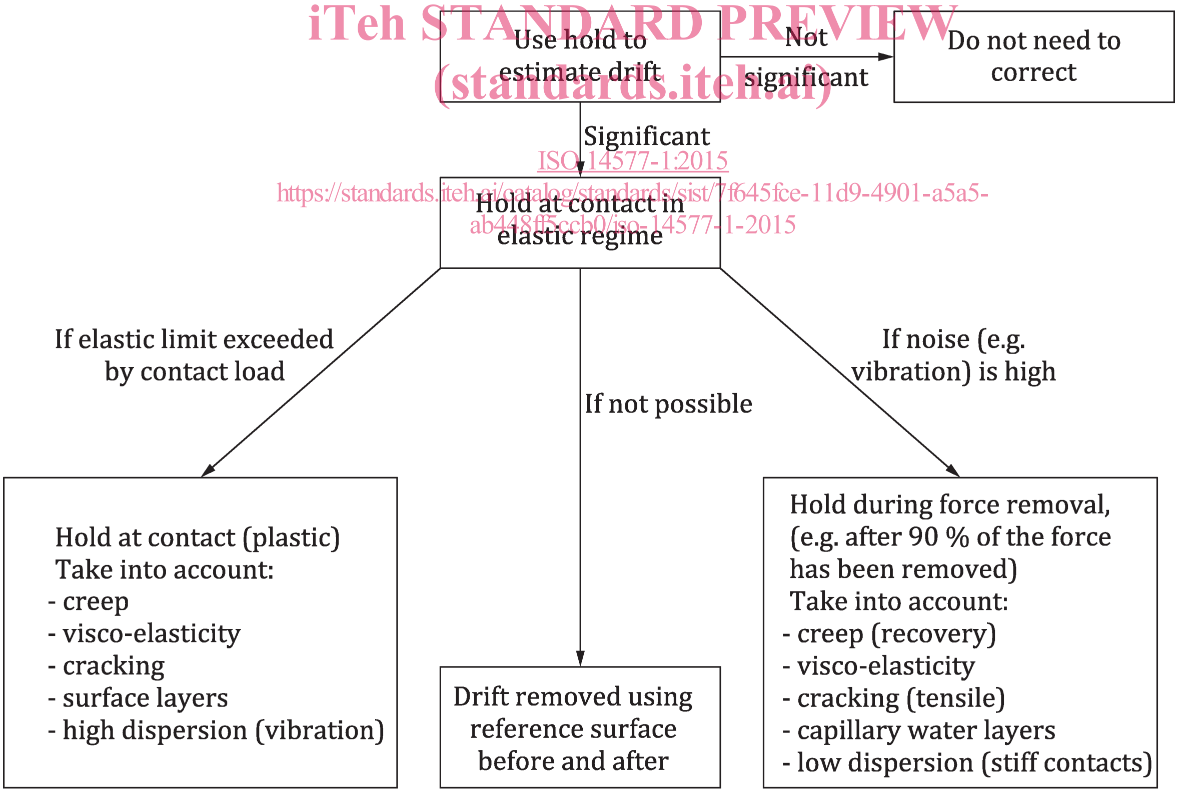


Figure 3 — Decision tree to assist in estimating thermal drift using a constant force hold period

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 be 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 can increase the compliance (reduce the stiffness) of the test piece support.

NOTE If the sample is supported by materials or mounting methods other than those used when determining the machine compliance, then the different elastic response of these materials and mounting methods can cause additional compliance.

7.3 The zero point for the measurement of the force/indentation depth curve shall be assigned individually to each test data set by one of the methods following. 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 should not exceed 1 % of maximum indentation displacement for the macro and micro ranges. The zero point uncertainty for the nano range can exceed 1 %, in which case the value shall be estimated.

a) Method 1: The zero-point is calculated by extrapolation of a fitted function to the force-application curve (see curve a in Figure 1); a power law fit with the exponent as a fitting parameter constrained to be $1 \leq m \leq 2$ is recommended. The fit shall be applied to values within the range from the first recorded data point to not more than 10 % of the maximum indentation depth. The first recorded data point shall be less than 2 % of F_{max} or less than 5 % of the maximum indentation depth and the fitted data shall not contain a change in indentation response such as the onset of plastic yielding. It is recommended that the first recorded data point be as close to the zero point as possible. The uncertainty of the calculated zero point results from the fit parameters, the fitting function and the length of extrapolation. The uncertainty is calculated as the standard error of the intercept of the fit with the zero force axes.

NOTE 1 The first part of the indentation curve (for instance up to 5 % of h_{max}) can be affected by vibration or other noise.

b) Method 2: The zero-point is the touch point determined from 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 2 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 can be either force-controlled or displacement-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 the following:

a) nature of the control (i.e. force or displacement control and whether a stepped or continuous change in the controlled parameters);

b) maximum force (or displacement);

c) force application (or displacement) function;

d) length and position of each hold period;

e) data logging frequency (or number of data points).

NOTE An example cycle for nano and micro ranges is the following: force application time, 30 s; hold at F_{max}, 30 s; force removal 10 s. A 60 s hold period to measure thermal drift can also be required (see Annex G).

The time taken for a test can influence the results obtained. 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 directly comparable only if the same indenter and 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, and

b) constant indentation displacement rate.

The rate of applied test force removal is subject to the requirements that: a sufficient number of data points for any subsequent analysis are recorded during applied test force removal, and that the total creep and any residual creep rate is within acceptable limits (see Annex G).

If the drift rate is significant (see 7.1 and Annex G), the force and depth data shall be corrected by use of the measured drift rate.

7.6 Throughout the test, the testing machine shall be protected from shock and vibration, air movements and variations in temperature, which 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 can occur at the corners of the indentation. When this occurs, the indentation diameter should enclose the crack.

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 10 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 might be too close and the distance should be increased. A factor of two increases in separation is suggested.

It can be desirable to measure thin coatings in cross-section (e.g. to avoid problems due to surface roughness). In this case, there might not be enough coating thickness to meet the minimum spacing requirements as specified above. Smaller spacing can be used if there is experimental evidence that this does not significantly influence the force/indentation depth/time data sets with respect to correctly spaced indentations on similar test pieces with thicker coatings.

8 Uncertainty of the results

A complete evaluation of the uncertainty shall be carried out in accordance with ISO/IEC Guide 98-3. A detailed description of two methods of evaluation of uncertainty is given in Annex H.

Method 1: This approach for determining uncertainty considers only those uncertainties associated with the overall measurement performance of the testing machine with respect to reference blocks (abbreviated as CRM below). These performance uncertainties reflect the combined effect of all of the separate uncertainties (indirect verification). When using this approach, it is important that, during the test, the individual machine components are operating in the exactly same way and within the tolerances of the indirect validation being used to estimate the uncertainties.

Method 2: This approach calculates a combined uncertainty from individual contributions. These can be grouped into random and systematic uncertainties. Individual parameters can contribute one or both types of uncertainty to the total measurement uncertainty. For example, the uncertainty in measured displacement can have a random component due to the resolution of the scale used and vibrational noise, etc., plus a systematic component due to the displacement sensor calibration uncertainty. The following sources of uncertainty shall be considered:

— zero point assignation;

— measurement of force and displacement (i.e. noise floor, including effects of ambient vibrations and magnetic field strength changes etc.);

— fitting of the force-removal curve;

— thermal drift rate;

— contact area due to surface roughness;

— 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 might not always be possible to individually quantify all of the identified contributions to the random uncertainty. In this case, an estimate of standard uncertainty can be obtained from the statistical analysis of repeated indentations into the test material. Care should be taken that systematic standard uncertainties that can contribute to the random standard uncertainty are not counted twice (see ISO/IEC Guide 98-3:2008, Clause 4).

A guideline for the evaluation of the uncertainty in determination of hardness and materials parameters (Annex A) is given in Annex H.

9 Test report

The test report shall include the following information:

a) reference to this part of ISO 14577, i.e. ISO 14577-1:2015;

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, and

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

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

f) method and functional form of any fit used for the determination of the zero-point;

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