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

Part 2:
Verification and calibration of testing machines

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

Partie 2: Vérification et étalonnage des machines d'essai

Reference number
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Foreword

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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).

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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 second edition cancels and replaces the first edition (ISO 14577-2:2002), 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.

INTERNATIONAL STANDARD **ISO 14577-2:2015(E)**

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

Part 2:

Verification and calibration of testing machines

1 Scope

This part of ISO 14577 specifies the method of verification and calibration of testing machines for carrying out the instrumented indentation test in accordance with ISO 14577-1:2015.

It describes a direct verification method for checking the main functions of the testing machine and an indirect verification method suitable for the determination of the repeatability of the testing machine. There is a requirement that the indirect method be used in addition to the direct method and for the periodic routine checking of the testing machine in service.

It is a requirement that the indirect method of verification of the testing machine be carried out independently for each test method.

This part of ISO 14577 is also applicable for transportable testing machines.

2 Normative references

The following 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 376, *Metallic materials — Calibration of force-proving instruments used for the verification of uniaxial testing machines*

ISO 3878, *Hardmetals — Vickers hardness test*

ISO 14577-1:2015, *Metallic materials — Instrumented indentation test for hardness and materials parameters — Part 1: Test method*

ISO 14577-3, *Metallic materials — Instrumented indentation test for hardness and materials parameters — Part 3: Calibration of reference blocks*

3 General conditions

3.1 Preparation

The machine shall be designed in such a way that it can be verified.

Before verification and calibration of the testing machine, it shall be checked to ensure that the conditions laid down in 3.2 to 3.4 are met.

3.2 Functional installation

The testing machine shall be configured to operate in compliance with and shall be installed in an environment that meets the requirements of this part of ISO 14577, ISO 14577-1:2015, and, where applicable, ISO 14577-3. The testing machine shall be protected from vibrations. For testing in the micro and nano ranges, the testing machine shall also be protected from air currents and temperature fluctuations.

The influence of environment on the data, i.e. the noise floor, shall be estimated by performing a low force (e.g. equivalent to the usual initial contact force) indentation on a CRM and analysing the displacement over time. The force variability is the indent stiffness (obtained from force removal curve) multiplied by the standard deviation of the displacement once any background drift in mean displacement has been subtracted. These uncertainties shall then be included in the total combined uncertainty tests as calculated in ISO 14577-1:2015, Clause 8 and Annex H.

3.3 Indenter

In order to get repeatable measurements of the force/indentation depth data set, the indenter holder shall be firmly mounted into the testing machine.

The indenter holder should be designed in such a way that the contribution to the overall compliance is minimized (see Annex A).

3.4 Application of the test force

The test force shall be applied and removed without shock or vibration that can significantly affect the test results. It shall be possible to verify the process of increasing, holding, and removal of the test force.

4 Direct verification and calibration

4.1 General

4.1.1 Direct verification and calibration shall be carried out at the constant temperature of use, typically 10 °C to 35 °C, but preferably in the range (23 ± 5) °C. If a range of operating temperatures is required, then direct calibration and verification should be carried out at suitable points over that temperature range to determine the calibration validity as a function of temperature. If necessary, a calibration correction function or a set of calibrations valid at specific operating temperatures can be determined.

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

4.1.3 Direct verification and calibration involves

a) calibration of the test force,

b) calibration of the displacement measuring device,

c) verification and calibration of the machine compliance,

d) verification of the indenter,

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

f) verification of the test cycle.

4.2 Calibration of the test force

4.2.1 Each range of force used shall be calibrated over the whole force range for both application and removal of the test force. A minimum of 16 evenly distributed points in the test force range shall be calibrated, i.e. 16 during application and 16 during removal of the test force. The procedure shall be repeated at least three times and the average calibration value shall be used. The maximum difference in calibration values shall not exceed half of the tolerances given in Table 1.

4.2.2 The test force shall be measured by a traceable method, for example, the following:

a) measuring by means of an elastic proving-device in accordance with class 1, or better of, ISO 376;

b) balancing against a force, accurate to within ±0,2 % applied by means of calibrated masses with mechanical advantage;

c) electronic balance with a suitable accuracy of 0,1 % of the minimum calibrated test force or 10 µg (0,1 µN) for the nano range.

For each measured point used for calibration, the difference between the measured and the nominal test force shall be within the tolerances given in Table 1.

Table 1 — Tolerances for test forces

Range of the test force F N	**Tolerances** %
$F \geq 2$	±1,0
$0{,}001 \leq F < 2$	±1,0
$F < 0{,}001$	±2,5[a]

[a] For the nano range, a tolerance of ±1 % is strongly recommended.

4.3 Calibration of the displacement measuring device

4.3.1 The resolution required of the displacement measuring system depends on the size of the smallest indentation depth being measured. For the micro range, this value is by definition h = 0,2 µm; for the macro range it is typically ≥2 µm.

The scale of the displacement measuring device shall be graduated to permit a resolution of indentation depth measurement in accordance with Table 2.

4.3.2 The displacement measuring device shall be calibrated on the testing machine for every range used by means of a suitable method and a corresponding system. The device shall be calibrated at a minimum of 16 points in each direction evenly distributed throughout its travel. The procedure shall be repeated three times.

The following methods are recommended for the measurement of the relative displacement of the indenter: laser interference method, inductive method, capacitive method, and piezotranslator method.

For each measured point used for calibration, the difference between the measured and the nominal displacement shall be within the tolerances given in Table 2.

Table 2 — Resolution and tolerances of the displacement measuring device

Range of application	**Resolution of the displacement measuring device** nm	**Tolerances**
Macro	≤100	1 % of h
Micro	≤10	1 % of h
Nano	≤1	2 nm[a]

[a] For the nano range, a tolerance of <1 % of h (indentation depth) is strongly recommended.

4.3.3 Changes in temperature are commonly a dominant source of displacement drift. To minimize thermally induced displacement drift, the temperature of the instrument shall be maintained such that the displacement drift rate remains constant over the time period of one calibration cycle. The drift rate shall be measured during, immediately before, or immediately after each calibration cycle, e.g. by monitoring displacement during a suitable hold period. The displacement calibration data shall be corrected for thermal drift and the product of variation in drift rate and the duration of one calibration cycle shall be less than the tolerance given in Table 2. The drift rate uncertainty shall be included in the displacement calibration uncertainty calculation.

4.4 Verification and calibration of the machine compliance

4.4.1 General

See Annex D and ISO 14577-1:2015, Annex C.

This verification and calibration shall be carried out after the test force and the displacement measuring system have been calibrated in accordance with 4.2 and 4.3.

4.4.2 Procedure

The calibration and verification of machine compliance is carried out by the measurement of indentation modulus at a minimum of five different test forces. Method 3 as described in Annex D is recommended. In all cases, a suitable Certified Reference Material (CRM) shall be mounted into the instrumented indentation test system in the same way as future test samples will be mounted. This is to ensure that the CRM provides a faithful reproduction of each particular total machine compliance.

The compliance of the testing machine can be affected by the particular construction and mounting of an indenter and also the method used to mount a sample. For instance, mounting in plastics (e.g. PVC) can introduce an extra compliance into the measurement loop. The verification and calibration of machine compliance should be performed using the indenter that will be used for subsequent measurements.

For indentation depths, $h_c > 6$ μm, it is not necessary to take into account the real contact area function. For the verification and calibration of the machine compliance, a reference material with certified indentation modulus, independent from the indentation depth, shall be used. A material with a high ratio of $E_{IT} / \sqrt{H_{IT}}$ (such as tungsten) is recommended. The range for the test force is defined by the minimum test force that correlates to 6 μm indentation depth and the maximum possible test force of the testing machine. Large indentation depths have the advantage that errors in the area function are likely to be smaller; however, care shall be taken that the test is not biased by pile-up in the reference material. The measured compliance of the indentation shall then be compared with the calculated compliance for the indentation using the certified value of modulus. To recalibrate machine compliance, the product of the applied force and the detected difference in machine compliance is applied to the displacement data to refine the estimate of contact depth and, therefore, the machine compliance estimate at each force. This process is iterated until self-consistent values of machine compliance and contact depth are reached.

For indentation depths, <6 μm, the method above shall be applied, except that the actual area of contact, as calculated from the calibrated indenter area function, shall be used to calculate the contact compliance using the certified modulus of the CRM.

In many nano and micro range instruments, the machine compliance value is independent of force. However, if this is not the case, then a machine compliance function can be determined using the above procedure but a wider range of forces. The range for the test forces is defined by the indentation depths, >0,5 μm, and the maximum test force of the testing machine or the maximum test force for which no unusual test piece response (e.g. pile-up of metals or cracking of ceramics or glasses) occurs.

If the machine compliance is recalibrated, then an indirect validation shall be performed before use.

The calibration procedures detailed in Annex D require the use of reference materials (see ISO 14577-3) that shall be isotropic and homogeneous. It is assumed that the indentation modulus and Poisson’s ratio are independent of the indentation depth.

4.5 Calibration and verification of the indenter

4.5.1 General

The indenter used for the indentation test shall be calibrated. Evidence that the indenter complies with the requirements of this part of ISO 14577 shall be fulfilled by a calibration certificate from a qualified calibration laboratory and evidence from the most recent indirect verification that the indenter area function has not changed. The latter shall be provided using the verification methods described in Annex B and suitable certified reference materials. All specified indenter geometry parameters shall be measured and incorporated into the calibration certificate.

If the angle of the indenter deviates from the nominal value for an ideal geometry of the indenter, the average of certified angles for that indenter should be used in all applicable calculations at depths $h > 6$ μm.

NOTE A 0,2° error in the Vickers angle of 136° (2α) results in a 1 % systematic error in area.

Indenters for use in the nano range and in the micro range, indentation depth <6 μm, shall have their area function calibrated over the relevant indentation depth ranges of use. The indenter performance shall be verified periodically (see Clause 6).

Where non-diamond indenters are used, the values of elastic modulus and Poisson ratio shall be obtained and used instead of the diamond values in the appropriate analyses.

The angle for pyramidal and conical indenters shall be measured within the indentation depth ranges given in Table 3 and illustrated in Figure 1.

Table 3 — Values for the measuring ranges for the angle of pyramidal and conical indenters

Dimensions in micrometres

Indentation depth	Macro range	Micro range
h_1	6	0,2
h_2	200	Specified max. indentation depth

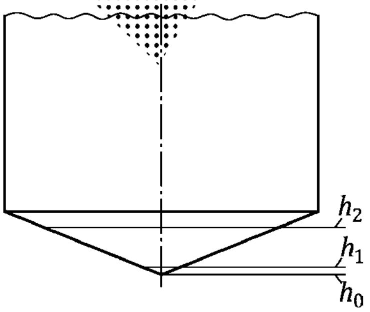


Figure 1 — Illustration of measuring ranges given in Table 3

4.5.2 Vickers indenter

4.5.2.1 The four faces of the right square-based diamond pyramid shall be smooth and free from surface defects and contaminants. For notes on cleaning of the indenter surface, see also ISO 14577-1:2015, Annex D.

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

4.5.2.2 The angle between the opposite faces of the vertex of the diamond pyramid shall be 136° ± 0,3° (see Figure 2) ($\alpha = 68{,}0° \pm 0{,}2°$).

The angle shall be measured in the range between h_1 and h_2 (see Table 3 and Figure 1). The geometry and finish of the indenter shall be controlled over the whole calibrated indentation depth range, i.e. from the indenter tip, h_0, to the maximum calibrated indentation depth, h_2.

4.5.2.3 The angle between the axis of the diamond pyramid and the axis of the indenter holder (normal to the seating surface) shall not exceed 0,5°.

4.5.2.4 The four faces shall meet at a point. The maximum permissible length of the line of conjunction between opposite faces is given in Table 4 (see also Figure 3).

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

4.5.2.6 The verification of the shape of the indenter shall be carried out using microscopes or other suitable devices.

If the indenter is used for testing in the micro or nano range, verification by a closed loop controlled atomic-force-microscope (AFM) should be carried out. For the nano range, this measurement is strongly recommended.

Table 4 — Maximum permissible length of the line of conjunction

Range of the indentation depth µm	**Maximum permissible length of the line of conjunction** µm
$h > 30$	1
$30 \geq h > 6$	0,5[a]
$h \leq 6$	≤ 0,5[b]

[a] This can be assumed to have been achieved when there is no detectable conjunction when the indenter is verified by an optical microscope at 400 × magnification.

[b] This shall be included when the correction of the shape of the indenter is taken into account; see ISO 14577-1:2015, C.2.

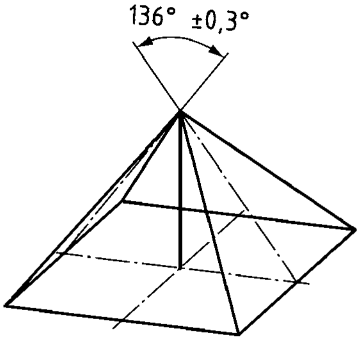
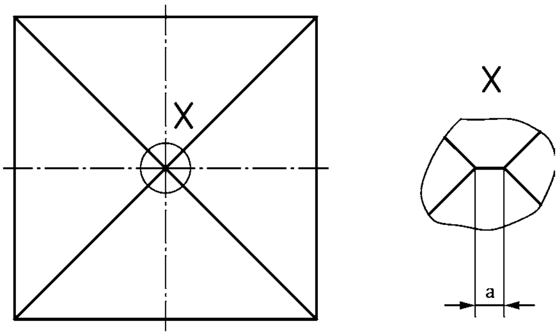


Figure 2 — Angle of the Vickers diamond pyramid



Key

a line of conjunction

Figure 3 — Line of conjunction on the tip of the indenter — Schematic

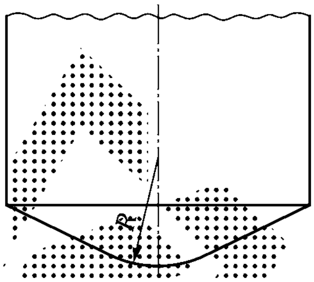


Figure 4 — Radius of the tip of the indenter

4.5.3 Berkovich, modified Berkovich, and corner cube indenters

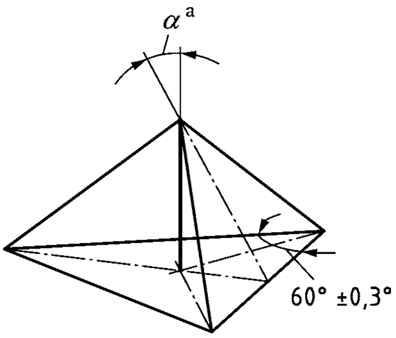
4.5.3.1 In practice, there are two types of Berkovich pyramidal diamond indenters in common use. The Berkovich indenter (see Reference [5]) is designed to have the same surface area as a Vickers indenter at any given indentation depth. The modified Berkovich indenter (see Reference [11]) is designed to have the same projected area as the Vickers indenter at any given indentation depth.

4.5.3.2 The three faces of the triangular based diamond pyramid shall be smooth and free from surface defects and from contaminations. For notes on cleaning of the surface, see also ISO 14577-1:2015, Annex D.

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

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

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



Key

a $\alpha = 65{,}03° \pm 0{,}30°$ for Berkovich indenter
$\alpha = 65{,}27° \pm 0{,}30°$ for modified Berkovich indenter
$\alpha = 35{,}26° \pm 0{,}30°$ for corner cube indenters

Figure 5 — Angle of the Berkovich and corner cube indenters

4.5.3.5 The verification of the shape of the indenter shall be carried out using microscopes or suitable devices.

If the indenter is used for testing in the micro and nano range, a measurement by a closed-loop controlled atomic-force-microscope (AFM) should be carried out. For the nano range, this measurement is strongly recommended.

4.5.4 Hard metal ball indenters

4.5.4.1 The characteristics of the hard metal balls shall be the following:

- — hardness: not less than 1 500 HV 10, when determined in accordance with ISO 3878;
- — density: $\rho = 14{,}8$ g/cm^3 ± 0,2 g/cm^3;

The following chemical composition is recommended:

a) cobalt (Co): 5,0 % to 7,0 %;

b) total carbides other than tungsten carbide: 2,0 %;

c) tungsten carbide (WC): balance.

4.5.4.2 The balls shall have a certified geometry. Batch certification methods are sufficient. The certificate shall show the diameter of the average value of at least three measured points of different positions. If any value differs from the permissible values of the nominal diameter (see Table 5), the ball (and/or the batch) shall not be used as an indenter.

Table 5 — Tolerances for ball indenters

Dimensions in millimetres

Ball diameter	Tolerance
10	±0,005
5	±0,004
2,5	±0,003
1	±0,003
0,5	±0,003

4.5.5 Spherical tipped conical indenters

The characteristics of spherical tipped conical indenters shall be as given in Table 6 (see also Figure 6).

Table 6 — Tolerances for sphero-conical indenters

Feature	Tolerance
$R_{av} \leq 50\ \mu m$	$\pm 0{,}25\ R_{av}$
$500\ \mu m > R_{av} > 50\ \mu m$[a]	$\pm 0{,}1\ R_{av}$
Cone included angle, 2α	
120°	±5°[a]
90°	±5°
60°	±5°
Cone flank angle, α	
60°	±5°
45°	±2,5°
30°	±2,5°

NOTE Centreline of cone to centreline of mount is within 0,01 mm.

[a] Rockwell diamond indenters (see ISO 6508-2) fulfil this requirement.

The instantaneous radius of curvature, $R(h)$, of the spherical cap at any indentation depth, h, measured from the point of first contact shall not vary by more than a factor of two from the average radius, R_{av}, as given by the condition in Formula (1):

$$0{,}5 \leq |R(h)/R_{av}| \leq 2 \tag{1}$$

Indenters with a spherical tipped cone shape are useful for many applications. These indenters are normally made from diamond but can also be made from other materials, e.g. ruby, sapphire, or hardmetal (WC-Co cemented carbide). They are intended to indent only with the spherical tip. If Hertzian contact mechanics are being used to interpret the indentation response, the value used for the indenter radius is critical. It is, therefore, recommended that the shape of each indenter be determined directly by a suitable measurement system, or indirectly by indentation into a certified reference material.

Surface roughness, *Ra*, should be minimized. Roughness causes an uncertainty in the actual area of contact and in the definition of the first contact point of the indenter with the test piece. Asperities have radii of contact vastly different from the average radius of the spherical cap and, therefore, behave very differently. If possible, the *Ra* of the diamond surface should be less than 1/20 of the usual indentation depth for an indenter.

NOTE Geometry suggests that the depth of the spherical cap, h_s, on a cone of included angle, 2α, and radius, R_{av}, is given by Formula (2):

$$h_s = R_{av}\,[1 - \sin(\alpha)] \tag{2}$$

In practice, there is a gradual transition from spherical cap to cone geometry that is hard to specify. Given this and the uncertainties in R_{av} and α allowed (see Table 4), caution should be exercised whenever the depth exceeds 0,5 h_s.

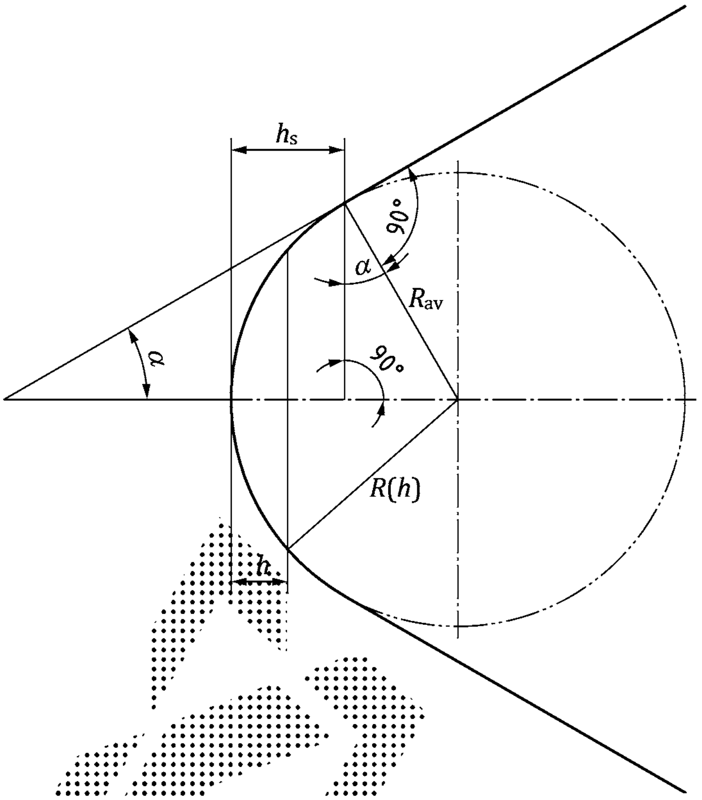


Figure 6 — Representation of the features of spherical indenters

4.6 Verification of the indenter area function

4.6.1 General

See ISO 14577-1:2015, Annex C.

4.6.2 Procedure

Procedures for the determination of indenter area function are given in Annex B.

The direct verification of the indenter area function consists of a comparison of the measured indenter area function with a documented indenter area function determined for the newly certified and calibrated indenter.

NOTE The indenter area function and machine compliance correction can be determined simultaneously using an iterative procedure and multiple reference materials (see Reference [8]).

If the difference in area between the measured and certified area functions (obtained as described in Annex B and expressed at each measured indentation depth as a percentage of the original certified area value) exceeds 30 % at any indentation depth in the range of calibration of an indenter, that indenter shall be discarded.

4.7 Verification of the testing cycle

The testing cycle (application of the test force, holding of the maximum test force, and removal of the test force) shall be measured with a tolerance of 0,1 s. The duration of each part of the testing cycle shall meet the requirements of ISO 14577-1:2015.

5 Indirect verification

5.1 General

Indirect verification should be carried out at the temperature of use by means of reference blocks calibrated in accordance with ISO 14577-3, or within the temperature at for which the calibration of the reference blocks is valid, typically (23 ± 5) °C. Indirect verification using a reference material shall be made to ensure the direct verification is valid and that no damage or contamination has occurred to the indenter tip.

Before measuring on the reference block, it is recommended to inspect and clean the indenter first using the procedure recommended in ISO 14577-1:2015, Annex D. If the results of these initial indentations indicate the presence of contamination or damage, then the indenter should be cleaned again before further trial indents are made. If after further cleaning, indentation into the reference material still indicates the presence of contamination or damage, then inspection with an optical microscope at a magnification of 400x is recommended. Detection of sub-microscopic damage or contamination is possible using appropriate microscopy of indents or the indenter. Where damage is detected, the indenter shall be replaced.

For an indirect validation decision tree, see Figure 7. The procedures for the determination of the machine compliance, C_F, and the area function, $A_p(h_c)$, calibration/verification shall be implemented before a new indenter is used. If, after applying the currently valid correction for machine compliance and indentation area function (obtained using a variable epsilon and a radial displacement correction, ISO 14577-1:2015, Annex I), a measured value from a reference block deviates from the certified value of the test piece by more than the maximum permissible amount of the limits specified in Table 7 (see Note 2) and repetition of the procedure using a newly verified and certified indenter and valid machine compliance correction corresponding to that indenter) also fails to reproduce the certified value, the testing machine shall be serviced and a full direct calibration be performed.

NOTE 1 The use of control charts is a sensitive way to determine changes in performances before a control limit is breached (see Annex C).

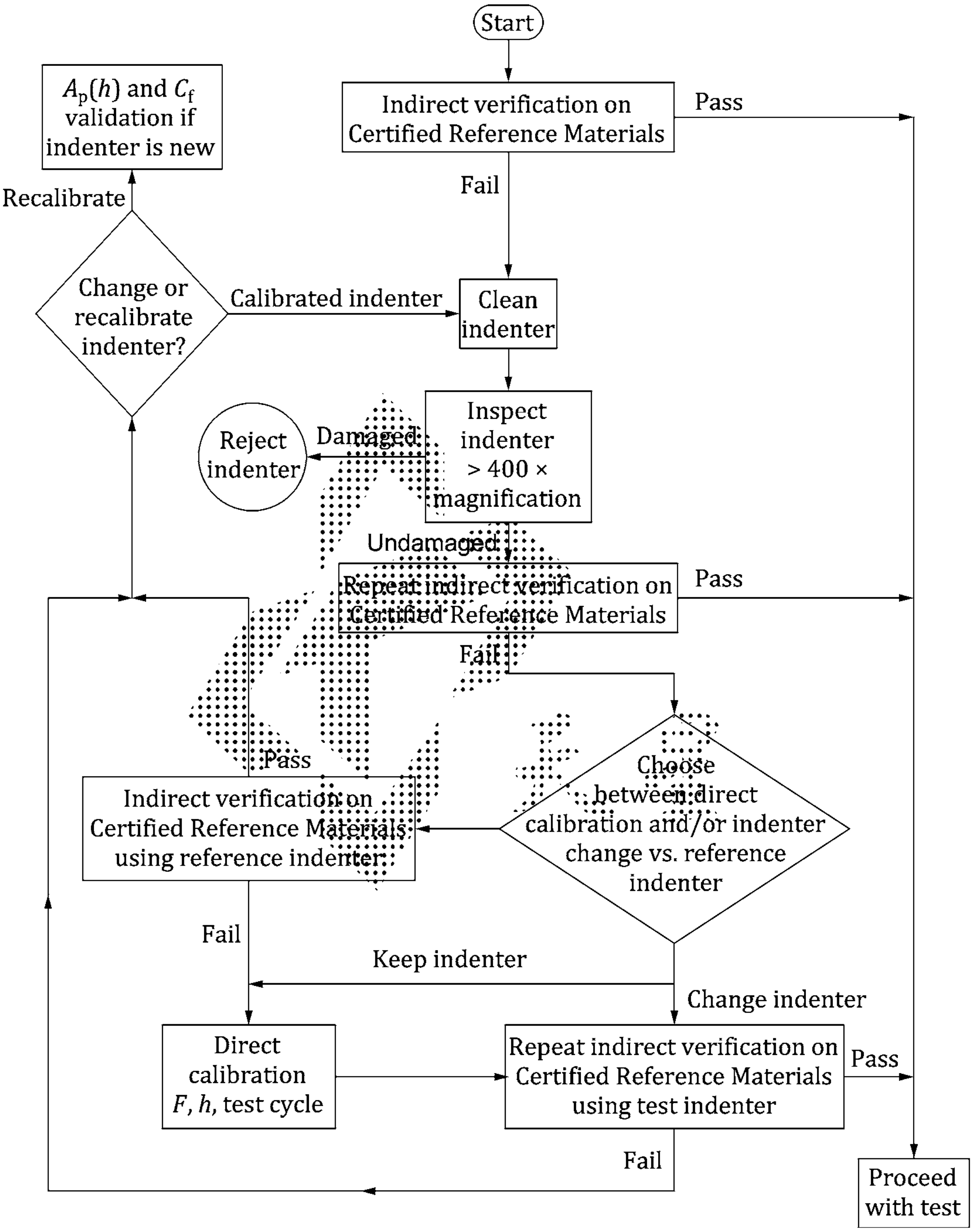


Figure 7 — Flow chart of decisions and actions to taken for indirect verification

NOTE 2 A reference indenter is a calibrated indenter used infrequently and only for checking the instrument and test indenter performance through indirect validation comparison.

5.2 Procedure

5.2.1 The indirect verification shall be carried out at least at the two test forces most frequently used. For tests with indentation depths <6 µm, this provides some verification of the contact area function. Indirect verification should be carried out on at least two reference blocks (CRMs) whose certified values differ significantly, e.g. by a factor of two. Indentations shall be performed at two or more forces that are an order of magnitude different or at least span the range of indentation forces or depths being measured.

For the indirect verification in the nano and micro range, it is recommended to use CRMs certified for indentation modulus.

5.2.2 If a testing machine is used only at one test force, it shall be verified only at this test force on at least two reference blocks with certified values that span the range of values of the test pieces being tested.

5.2.3 On each reference block, five measurements are recommended in accordance with ISO 14577-1:2015. For indentation depths <6 µm, at least 10 measurements at each test force on each block are recommended to reduce the uncertainty in repeatability of the measurement mean.

NOTE When a CRM is used for daily check of testing machine before routine measurements, three to five indentations are considered sufficient.

5.2.4 For each reference block, an arithmetic mean value, $\bar{q}$, from n values $q_1, ..., q_n$ (where q represents the materials parameters) is calculated as given in Formula (3):

$$\bar{q} = \frac{q_1 + \ldots + q_n}{n} \tag{3}$$

The experimental standard deviation shall be calculated as a parameter to describe the scatter of the measurement values as given by Formula (4):

$$s(q) = \sqrt{\frac{\sum_{i=1}^{n}(q_i - \bar{q})^2}{n-1}} \tag{4}$$

The relative scattering of the measured values is the coefficient of variation, expressed as a percentage, given by Formula (5):

$$V = \frac{s(q)}{\bar{q}} \times 100 \tag{5}$$

5.2.5 The repeatability of the testing machine under the particular verification conditions is determined by the coefficient of variation of the measured value.

The repeatability of the testing machine is considered satisfactory if it satisfies the conditions given in Table 7.

Table 7 — Repeatability of the testing machine

Material parameter	Nano range	Micro range		Macro range
		0,2 µm ≤ h ≤ 1 µm	h > 1 µm	
HM	5 %	5 %	2 %	2 %
$E_{IT}/H_{IT} < 50$	5 %	5 %	5 %	5 %
$E_{IT}/H_{IT} > 50$	5 %	10 %	5 %	5 %

5.2.6 The error of the testing machine is characterized by the difference given in Formula (6):

$$|\bar{q}-q| \tag{6}$$

where

$\bar{q}$ is the arithmetic mean value calculated from single measurements;

q is the specified value of the reference block used.

It is standardized using the *t*-statistic as given in Formula (7).

$$t=\frac{\bar{q}-q}{s(q)}\times\sqrt{n} \tag{7}$$

The value of t returned from the testing machine shall not exceed the critical value, t_c, for a two-tailed test, with $(n-1)$ degrees of freedom, at the 95 % confidence level (see Table 8 for example values).

Table 8 — Example values for *n* and t_c

n	5	10	15	20
t_c	2,78	2,26	2,14	2,09

This is given more accurately in Formula (8):

$$\frac{s(q)}{\sqrt{n}}=\left[\frac{(n-1)s^2+(n_{\text{cal}}-1)s_{\text{cal}}{}^2}{n+n_{\text{cal}}-2}\right]^{1/2}\times\left(\frac{1}{n}+\frac{1}{n_{\text{cal}}}\right)^{1/2} \tag{8}$$

where

s_{cal} is the experimental standard deviation (determined during calibration);

n_{cal} is the number of calibrations averaged for the certified reference block value.

In general, $s(q)$ is equivalent to s to a first approximation and this is more valid if n and n_{cal} are greater. See also ISO/IEC Guide 98-3:2008, Annex G for the use of Welch-Satterthwaite equation to define better the effective number of degrees of freedom to use when determining the critical *t*-statistic.

6 Intervals between calibrations and verifications

6.1 Direct verification and calibration

6.1.1 A complete direct verification and calibration shall be carried out when the machine is new.

6.1.2 A limited direct verification and calibration including points 4.2, 4.3, and 4.7 shall be carried out

a) when the result of the indirect verification is not satisfactory, and

b) at least at intervals not exceeding three years.

6.1.3 A direct verification with respect to 4.2 or 4.3 shall be carried out when the machine is installed or after its dismantling and reassembly or relocation. If it was shown before, that the calibration will not be changed during machine installation or after its dismantling and reassembling or relocation only an indirect verification will be required.

In all cases (6.1.1, 6.1.2, and 6.1.3), each calibration and direct verification shall be followed by an indirect verification. The machine compliance shall be verified after every change of the indenter.

6.2 Indirect verification

The indirect verification shall be carried out periodically or before tests requiring high accuracy. The period between two indirect verifications shall not exceed one year. It is recommended that more frequent indirect verifications be performed depending on how frequently the machine is used.

6.3 Routine checking

Before any series of tests, and periodically (e.g. daily) within each series, a test at two different test forces shall be performed on a test piece of known materials parameter. The result of this test shall be recorded on a suitable chart; for example, see Annex C. If the results are outside the normal range of reproducibility, an indirect verification shall be performed.

It is good practice that test indentations be performed both before and after each test series.

7 Verification report/Calibration certificate

The verification report/calibration certificate shall include at least the following information:

a) reference to this part of ISO 14577, i.e. ISO 14577-2:2015;

b) method of calibration/verification;

c) identification data of the testing machine;

d) means of calibration or verification [reference blocks (CRM), elastic proving devices, etc.];

e) test forces;

f) calibration/verification temperature;

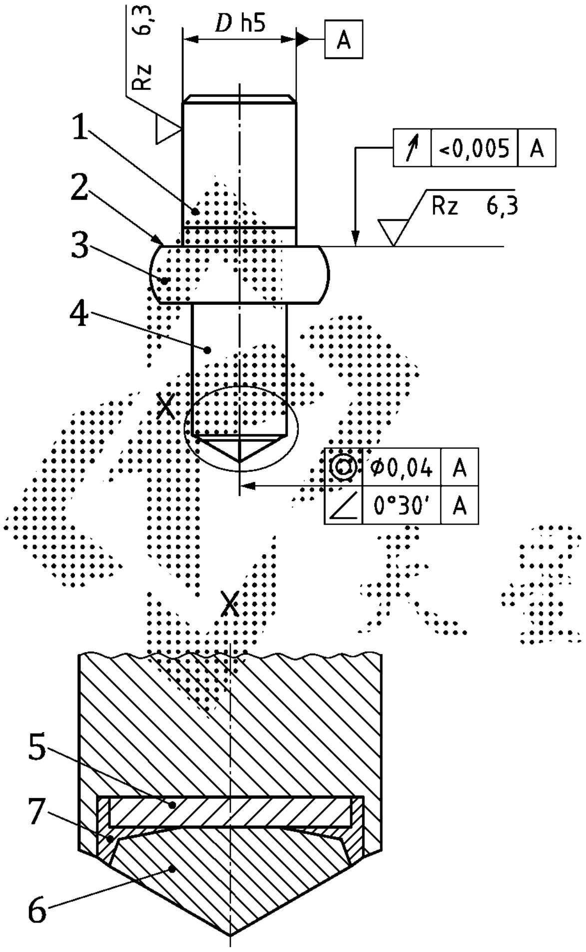
g) the results obtained, reported in the format required in ISO 14577-1:2015;

h) date of calibration/verification and reference to the calibration or verification institution.

Annex A
(informative)

Example of an indenter holder

Dimensions in millimetres; Surface roughness values in micrometres



Key

1 neck

2 seating face

3 seating

4 shank

5 support for the indenter (hardmetal)

6 indenter

7 embedding material

Figure A.1 — Example of a suitable design for the indenter holder

Annex B
(normative)

Procedures for determination of indenter area function

B.1 General

The following methods for determining the indenter area function are valid. Each of these methods results in the same indenter area function within the permissible tolerances.

B.2 Direct measurement method

The most appropriate method of direct measurement depends on the intended use of the indenter. A traceably calibrated atomic force microscope (AFM) is ideal for high resolution shape characterization of the last 1 µm or so of the indenter. Care shall be taken to ensure that the AFM measurements take account of the various sources of error and uncertainty; see References [9] and [13]. Traceably calibrated electron or optical microscopy methods can be more convenient if only larger indentation depths are intended.

B.3 Indirect measurement methods

B.3.1 These methods rely on using the test machine to perform indentation cycles on a material of certified properties. It, therefore, requires that all instrument calibrations required by this part of ISO 14577 be completed satisfactorily and that the machine compliance correction has been determined as in 4.5 or that an iterative indentation modulus based procedure is to be used; see Reference [8]. Once the force/displacement data have been corrected for machine compliance (and for thermal or other systematic drift), the following two methods may be applied.

B.3.2 If, for a material of known and depth independent Martens hardness, there is some experimental evidence that under the given experimental conditions the indenter size effect can be neglected, this material can be used as test piece and $A_S(h)$ can be derived for each specific indentation depth, h, measured at test force, F. This method is normally not useful for small indentation depths, e.g. less than 0,2 µm.

B.3.3 If a material of certified Young's modulus and Poisson's ratio or certified plane strain modulus is used as a test piece, a fit to the force removal curve may be used to determine the contact size and stiffness from which the contact compliance can be related to the indentation modulus of the test piece as given by Formula (B.1):

$$C_s = \frac{\sqrt{\pi}}{2} \frac{1}{E_r \sqrt{A_p}} \tag{B.1}$$

and

$$E_r = \frac{1-{v_s}^2}{E_s} + \frac{1-{v_i}^2}{E_i} \tag{B.2}$$

where

C_s is the compliance of the contact, dh/dF, at maximum applied force (reciprocal of the contact stiffness) after correction for machine compliance, C_F;

E_r is the reduced modulus;

A_p is the projected contact area, value of the indenter area function at the contact depth defined in the same way as for the calculation of hardness according to ISO 14577-1:2015, A.4;

v_s is the Poisson's ratio for the test piece;

v_i is the Poisson's ratio for the indenter (for diamond, it is equal to 0,07);

E_s is the Young's modulus of the material;

E_i is the Young's modulus of the indenter (for diamond, it is equal to $1{,}14 \times 10^6$ N/mm^2).

A variable epsilon (ε = 0,72 to 0,8) and radial displacement correction shall be used, see ISO 14577-1:2015. The method for estimation of radial displacement correction is given in ISO 14577-1:2015, Annex I. The radial correction is very small for most metals (<0,5 %) but reaches up to 5 % for highly elastic materials such as fused silica.

Thus, if a material of certified indentation (or Young's) modulus is used as a test piece, A_p can be derived for each specific contact depth and h_c by rearranging the above relationships (see ISO 14577-1:2015, A.4, for the determination of contact depth). Use of an iterative method and multiple reference materials permits the simultaneous measurement of indenter area function and machine compliance correction, see Reference [8].

The force range shall be selected to encompass the full range of likely indentation depths. For force controlled indentations, some preliminary experiments are required to establish the force range required to produce appropriate displacements in the reference material. A series of at least 10 different forces shall be chosen to span the range of interest and a total of at least 100 indentation cycles shall be made in the reference material. It is recommended to use many depths with three or more replicate indentations per depth with the mean value used to determine A_p. Thus, a plot of A_p versus indentation contact depth, h_c, is obtained. The use of load-partial unload method to obtain results from many indentation depths in one spot is also acceptable.

One advantage of using a modulus as the reference property is that the elastic response of the test piece is not sensitive to work hardening or thermal treatment or to the exact amount of creep that has occurred. All that is required is that the creep rate during force removal be negligible with respect to the force removal rate of the indentation experiment. Another advantage is that Young's modulus can be determined independently by non-indentation techniques. This eliminates circularity in the calibration traceability.

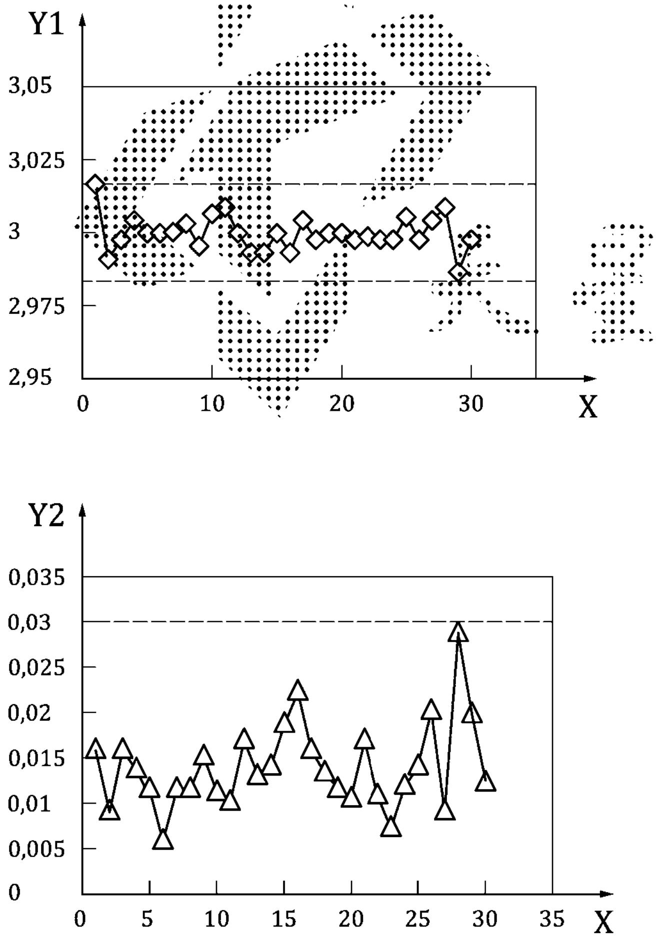
The area function is normally expressed as a mathematical function relating the projected or surface area to the distance from the tip of the indenter. At indentation depths where the area function cannot be described by a relatively simple (cubic or polynomial) mathematical function, an estimate can 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.

Annex C
(informative)

Examples for the documentation of the results of indirect verification

It is a benefit to chart the results of the routine checks and indirect verifications in order to monitor the performance of the testing machine over time. Each indirect verification should include a minimum of three or five force/indentation depth curves. The mean value and the standard deviation of the determined material parameter are recorded and documented in the form of a $\overline{q}/s$ chart (see Figures C.1 and C.2). Examples of suitable parameters to chart are indentation modulus or indentation depth at two predetermined test forces, e.g. $F_{\max}$ and 0,1 $F_{\max}$.

Any instability over time of the test force and displacement unit or contamination of the indenter is seen in both $\overline{q}/s$ charts. Wear of the indenter tip is most sensitively detected by the $\overline{q}/s$ chart of the results measured with 0,1 × $F_{\max}$.



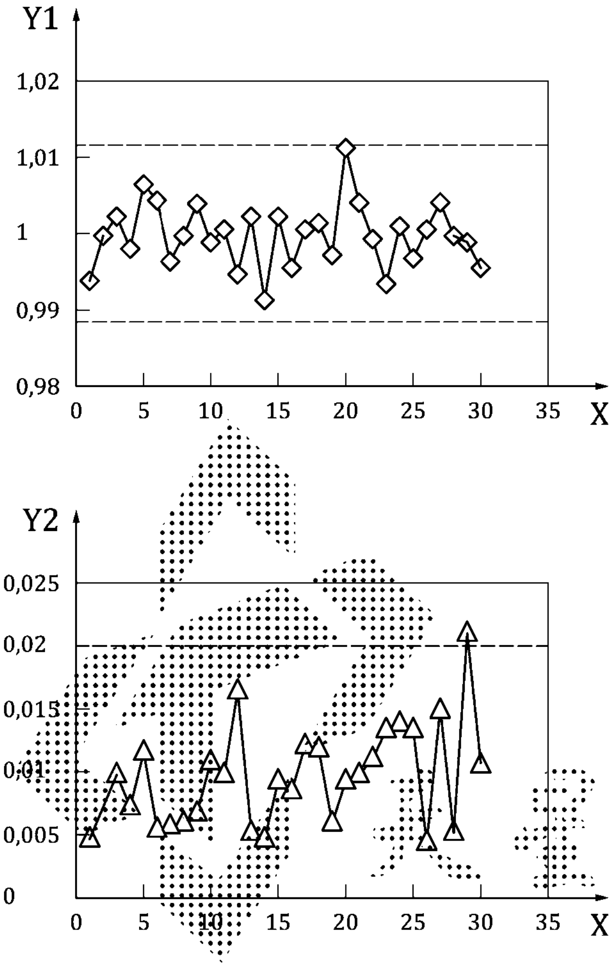
Key

Y1 mean value, in µm at $F_{\max}$

Y2 standard deviation, in µm

X number of indirect verifications

Figure C.1 — Example for $\overline{q}/s$ chart, indentation depth at $F_{\max}$



Key

Y1 mean value, in µm at 0,1 F_{max}

Y2 standard deviation, in µm

X number of indirect verifications

Figure C.2 — Example for $\overline{q}/s$ chart, indentation depth at 0,1 F_{max}

Annex D
(normative)

Machine compliance calibration procedure

D.1 General

The calibration procedures detailed below require the use of reference materials (see ISO 14577-3) which shall be isotropic and homogeneous. Young's modulus and Poisson's ratio are assumed to be independent of the indentation depth. The selected procedure including the ranges of test force, *F*, and indentation depth, *h*, for the performed calibration shall be reported.

D.2 Principle

The total measured compliance, C_T, is the sum of contact compliance, C_s, and the machine compliance, C_F, as given in Formula (D.1):

$$C_T = C_s + C_F \qquad \text{(D.1)}$$

where

C_T is derived from the derivative of the (uncorrected) test force removal curve at maximum force, as given in Formula (D.2);

C_s is the contact compliance of the specimen material, as given in Formula (D.3).

$$C_T = \left[\frac{\mathrm{d}F}{\mathrm{d}h}\right]^{-1} \qquad \text{(D.2)}$$

NOTE 1 Some instruments use tip-calibration routines that automatically assign machine compliance values. If this is the case, it is necessary that the machine compliance be determined, e.g. by the two reference material iterative methods in this Annex, be summed with any machine compliance assumed by the software in determining the area function of the tip.

$$C_s = \frac{\sqrt{\pi}}{2E_r} \cdot \frac{1}{\sqrt{A_p(h_c)}} \qquad \text{(D.3)}$$

$$\frac{1}{E_r} = \frac{1-\nu_s}{E_{IT}} + \frac{1-\nu_i}{E_i} \qquad \text{(D.4)}$$

$$h_c = h_{max} - \varepsilon F_{max} C_T \qquad \text{(D.5)}$$

NOTE 2 See ISO 14577-1:2015, A.4.

where

E_i is the Young's modulus of the indenter;

ν_i is the Poisson ratio of the indenter.

A variable epsilon (ε = 0,72 to 0,8) and radial displacement correction shall be used, see ISO 14577-1:2015. The method for estimation of radial displacement correction is given in ISO 14577-1:2015, Annex I.

Thus, the total compliance is as given in Formula (D.6):

$$C_{\mathrm{T}} = \frac{\sqrt{\pi}}{2E_{\mathrm{r}}} \cdot \frac{1}{\sqrt{A_{\mathrm{p}}(h_{\mathrm{c}})}} + C_{\mathrm{F}} \tag{D.6}$$

The following methods for the determination of the machine compliance are based on a series of indentation experiments that shall be performed using an isotropic reference material. The methods are ordered regarding rising effort and higher accuracy required to obtain the data on decreasing indentation depth. The assumptions of the methods are gathered in Table D.1.

Table D.1 — Required assumptions of the selected method

Method	E_{r} = const.	C_{F} = const.	Parameter input required	Ref.
1	yes	yes	$A_{\mathrm{p}}(h_{\mathrm{c}})$	[7]
2	yes	yes	none	[12]
3	yes	no	$A_{\mathrm{p}}(h_{\mathrm{c}})$, E_{r}	[10]
4	yes	no	E_{r1}, E_{r2}	[8]
5	yes	no	E_{r1}, E_{r2}, elastic deformation	[6]

D.3 Methods

D.3.1 General

Users should check that they know exactly what an automatic calibration function does. Some software can also calculate a machine compliance value. This shall be taken into account when calculating the final machine compliance obtained from the methods described in D.3.2 to D.3.6.

D.3.2 Method 1

This method is used, if

— the area function, $A_{\mathrm{p}}(h_{\mathrm{c}})$, is determined independently, for example, by replica (see Reference [7]) or atomic force microscopy, and

— a plot of C_{T} (uncorrected for machine compliance) versus $1/\sqrt{A_{\mathrm{p}}(h_{\mathrm{c}})}$ [see Formula (D.6)] is linear and intersects the compliance axis at the machine compliance, C_{F}.

NOTE Maximum test forces are typically in the 10 mN to 100 mN range. A minimum of 10 replicate indentations at any single force is recommended to obtain statistically valid calibration values. Tungsten is considered to be a suitable reference material because large stiff indentations are obtained for the force range of interest.

D.3.3 Method 2

If the area function is not known, a combined iterative procedure is used. Using the area function of the perfect indenter (ISO 14577-1:2015, A.4), an initial estimation of C_{F} and E_{r} is obtained by plotting C_{T} (uncorrected for machine compliance) versus $1/\sqrt{A_{\mathrm{p}}(h_{\mathrm{c}})}$ [see Formula (D.6)] for the two largest indentations; see Reference [12] Then, the new area function using all other indentations is calculated by rearranging Formula (D.6), as given in Formula (D.7):

$$A_{\mathrm{p}}(h_{\mathrm{c}}) = \frac{\pi}{4} \frac{1}{E_{\mathrm{r}}^{2}} \cdot \frac{1}{(C_{\mathrm{T}} - C_{\mathrm{F}})^{2}} \tag{D.7}$$

Using the new area function, the estimation of C_F and E_r is repeated with the Formula (D.6). The new values of C_F and E_r influence the area function after Formula (D.7). This procedure is iterated several times until convergence is achieved.

NOTE In Reference [12], aluminium and fused silica are used as reference materials. Test forces in the range 0,1 mN to 120 mN are used (3 mN to 120 mN in Al; 0,1 mN to 120 mN in fused silica) with each indentation test replicated 10 times. Fused silica is used to extend the area function to small distances from the tip.

D.3.4 Method 3

A reference material of certified modulus and a valid indenter area function are required. The machine compliance, C_F, is calculated by direct use of Formula (D.6) and substituting values for E_r, $A_p(h_c)$, and C_T. The first estimation of the machine compliance is used to correct the raw data to produce better values of the real contact depth at F_{max}, h_c. A new estimate for the machine compliance is then calculated using Formula (D.6). The whole procedure is iterated until convergence is achieved; see Reference [10].

Tungsten is a preferred reference material because it is an elastically isotropic, homogeneous material, and it is less prone to handling damage as compared to aluminium. Moreover, it has a high modulus and allows sufficient plastic deformation to give high contact stiffness, yielding more robust values of the machine compliance. Test forces up to 80 mN and a minimum of 10 replicate indentations is recommended to obtain statistically valid calibration values.

It is important for the accuracy of this method that the area function (at distances relatively far from the indenter tip) be well known, e.g. by independent AFM measurement, because the result is sensitive to that input.

D.3.5 Method 4

If the area function is not known, the combined iterative procedure of method 2 is performed on two materials with different hardness and elastic properties. Large indentations (100 mN to 200 mN range) shall be made into a stiff material [e.g. single crystal (100) tungsten] to obtain a value of the machine compliance and shallow indentations in fused silica (1 mN to 100 mN) to obtain the indenter area function. Using this approach, it was shown that after only a few iterations, the machine compliance and indenter area function can be obtained and the latter agreed with the area function obtained by an independent AFM measurement; see Reference [8].

Sapphire and fused silica may also be used as reference materials. Test force in the range 0,1 mN to 500 mN are used with each indentation test replicated 10 times.

D.3.6 Method 5

If the indenter tip is approximately a sphere, an elastic indentation occurs in the initial range of force application. For the spherical indenter, the machine compliance and the real indenter geometry (tip radius, R, instead of area function) is determined using two reference materials with different elastic properties, E_{r1}, E_{r2}, (see Reference [6]), as given in Formulae (D.8) and (D.9).

$$R = \left(\frac{3}{4}\right)^{2} \frac{F^{2}}{(h_1 - h_2)^{3}} \left(\frac{1}{E_{\mathrm{r1}}^{2/3}} - \frac{1}{E_{\mathrm{r2}}^{2/3}}\right)^{3} \tag{D.8}$$

$$C_{\mathrm{F}} = \frac{h_1 - \left(\frac{3}{4}\frac{F}{E_{\mathrm{r1}}}\right)^{2/3} \left(\frac{1}{R}\right)^{1/3}}{F} \tag{D.9}$$

NOTE The result is valid for one value of the test force, F, but for a range of the indentation depth, h_1, h_2.

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