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

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

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

Partie 4: Méthode d'essai pour les revêtements métalliques et non métalliques

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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 2.

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 document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 14577-4 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*

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

Introduction

The elastic and plastic properties of a coating are critical factors determining the performance of the coated product. Indeed many coatings are specifically developed to provide wear resistance that is usually conferred by their high hardness. Measurement of coating hardness is often used as a quality control check. Young's modulus becomes important when calculation of the stress in a coating is required in the design of coated components. For example, the extent to which coated components can withstand external applied forces is an important property in the capability of any coated system.

It is relatively straightforward to determine the hardness and indentation modulus of bulk materials using instrumented indentation. However, when measurements are made normal to a coated surface, depending on the force applied and the thickness of the coating, the substrate properties influence the result.

The purpose of this part of ISO 14577 is to provide guidelines for conditions where there is no significant influence of the substrate, and, where such influence is detected, to provide possible analytical methods to enable the coating properties to be extracted from the composite measurement. In some cases, the coating property can be determined directly from measurements on a cross-section.

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

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

1 Scope

This part of ISO 14577 specifies a method for testing coatings which is particularly suitable for testing in the nano/micro range applicable to thin coatings.

This test method is limited to the examination of single layers when the indentation is carried out normal to the test piece surface, but graded and multilayer coatings can also be measured in cross-section if the thickness of the individual layers or gradations is greater than the spatial resolution of the indentation process.

The test method is not limited to any particular type of material. Metallic, non-metallic and organic coatings are included in the scope of this part of ISO 14577.

The application of this part of ISO 14577 regarding measurement of hardness is only possible if the indenter is a pyramid or a cone with a radius of tip curvature small enough for plastic deformation to occur within the coating. The hardness of visco-elastic materials, or materials exhibiting significant creep will be strongly affected by the time taken to perform the test.

NOTE 1 ISO 14577-1, ISO 14577-2 and ISO 14577-3 define usage of instrumented indentation testing of bulk materials over all force and displacement ranges.

NOTE 2 The application of the method of this part of ISO 14577 is not needed if the indentation depth is so small that in any possible case a substrate influence can be neglected and the coating can be considered as a bulk material. Limits for such cases are given.

NOTE 3 The analysis used here does not make any allowances for pile-up or sink-in of indents. Use of Atomic Force Microscopy (AFM) to assess the indent shape allows the determination of possible pile-up or sink-in of the surface around the indent. These surface effects result in an under-estimate (pile-up) or over-estimate (sink-in) of the contact area in the analysis and hence may influence the measured results. Pile-up generally occurs for fully work-hardened materials. Pile-up of soft, ductile materials is more likely for thinner coatings due to the constraint of the stresses in the zone of plastic deformation in the coating. It has been reported that the piled up material results in an effective increase of the contact area for the determination of hardness, while the effect is less pronounced for the determination of indentation modulus, since the piled up material behaves less rigidly [1], [2].

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 1514, *Paints and varnishes — Standard panels for testing*

ISO 2808, *Paints and varnishes — Determination of film thickness*

ISO 3270, *Paints and varnishes and their raw materials — Temperatures and humidities for conditioning and testing*

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

ISO 14577-1:2002, *Metallic materials — Instrumented indentation test for hardness and materials parameters — Part 1: Test method*

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

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

3 Symbols and designations

The symbols and designations in ISO 14577-1, ISO 14577-2 and ISO 14577-3 and in Table 1 apply.

Table 1 — Symbols and designations

Symbol	Designation	Unit	Required in the test report
F	Test force	mN	✓
$A_p(h_c)$	Projected area of contact of the indenter at distance h_c from the tip	μm^2	—
H_c	Indentation hardness of the coating	$mN/\mu m^2$ [b]	✓
ν_i	Poisson's ratio of the indenter [a]	—	—
ν_s	Poisson's ratio of the test piece	—	—
a	Radius of contact area	µm	—
t_c	Film thickness	µm	✓
C_f	Frame compliance	µm/mN	✓
C_s	Contact compliance (test piece)	µm/mN	—
C_t	Total measured compliance	µm/mN	—
E	Young's modulus	$mN/\mu m^2$ [b]	—
E_c^*	Plane strain indentation modulus of the coating [c]	$mN/\mu m^2$	—
E_{IT}^*	Plane strain indentation modulus	$mN/\mu m^2$ [b]	✓
E_r	Reduced modulus of the indentation contact	$mN/\mu m^2$ [b]	—
Ra	Arithmetic mean deviation from the average height of the assessed profile (see ISO 4287).	µm	

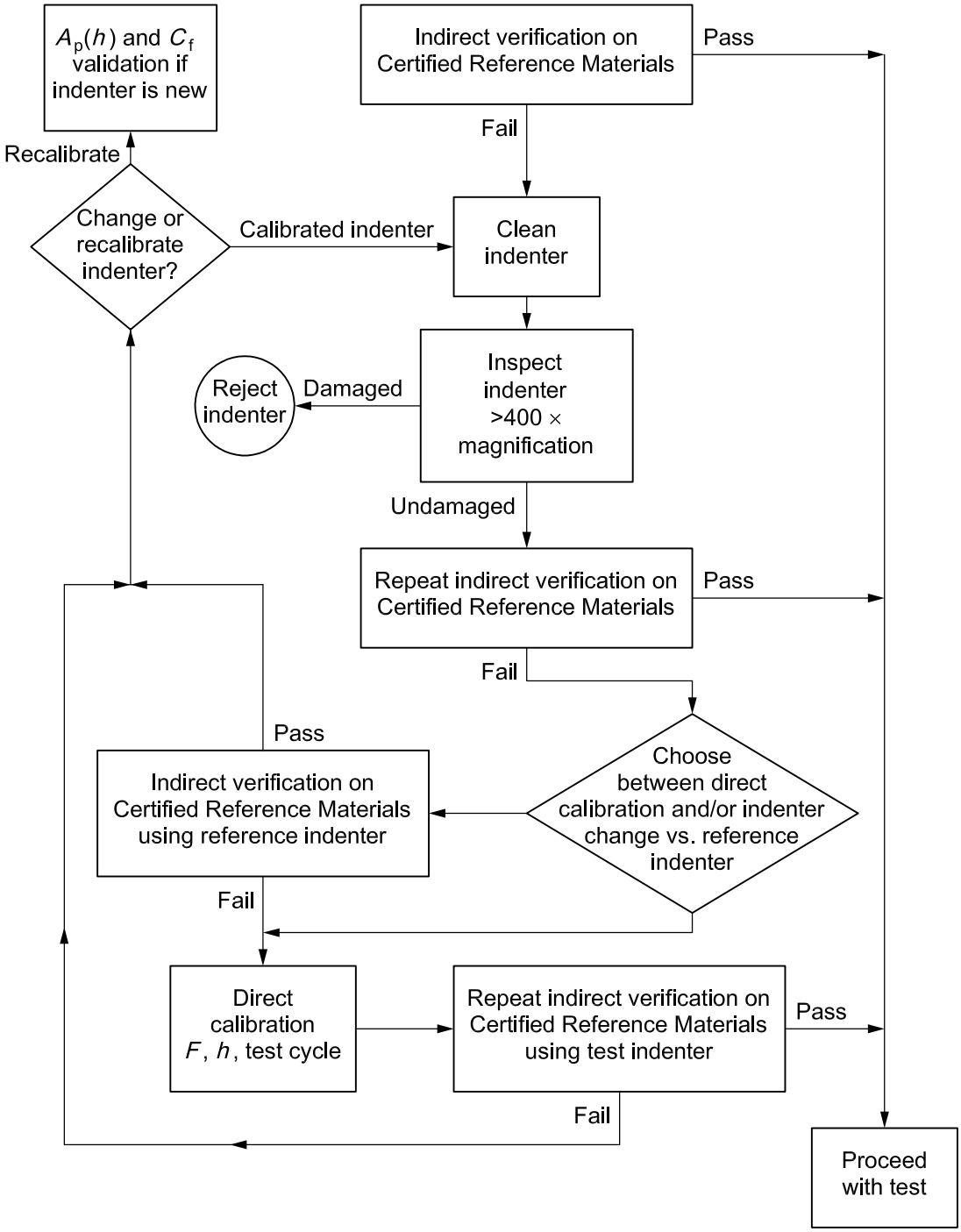
[a] For diamond $\nu_i = 0{,}07$.
[b] 1 $mN/\mu m^2$ = 1 GPa.
[c] $E_c^* = E_{IT}^*$ (at $a/t_c = 0$).

4 Verification and calibration of testing machines

The instrument shall be calibrated according to the procedures set out in ISO 14577-2 and Annex A.

Indirect verification using a reference material shall be made to ensure that a new direct verification is not needed and that no damage or contamination has occurred to the indenter tip. If the results of these initial indentations indicate the presence of contamination or damage, then the indenter should be cleaned using the procedure recommended in ISO 14577-1 before further trial indents are made. After cleaning, inspection with

an optical microscope at a magnification of greater than 400× 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 according to ISO 14577-2. The procedures for the determination of the frame compliance C_f and the area function $A_p(h_c)$ calibration/verification shall be implemented before a new indenter is used, see Figure 1.



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

Figure 1 — Flow chart of the decisions and actions to be taken in the case of indirect verification failure

The instrumented indentation instrument shall achieve the required mechanical and thermal stability before starting an indentation cycle, see 6.2.

Indentation experiments may be performed with a variety of differently shaped indenters which should be chosen to optimize the plastic and elastic deformation required for a given coating substrate system. Typical indenter shapes are Vickers, Berkovich, conical, spherical and corner cube.

For the determination of coating plastic properties, pointed indenters are recommended. The thinner the coating, the sharper the indenter should be. For the determination of coating elastic properties, any geometry indenter may be used provided that its area function is known. If only the elastic properties of the coating are required, indentations in the fully elastic regime are recommended (if possible) as this avoids problems due to fracture, pile up and high creep rates. A larger radius indenter tip or sphere will allow fully elastic indentations over a larger force range than a smaller radius indenter. However, too large a radius and surface effects will dominate the measurement uncertainties (roughness, surface layers, etc.). Too small a radius and the maximum force or displacement before plastic deformation begins, will be very low. The optimum can be identified by preliminary experiments or modelling (see Clause 7).

5 Test pieces

5.1 General

Generally, surface preparation of the test piece should be kept to a minimum and, if possible, the test piece should be used in the as-received state if the surface condition conforms to the criteria given in 5.2, 5.3 and 5.4.

The test piece shall be mounted using the same methods as employed for determination/verification of the instrument frame compliance, and shall be such that the test surface is normal to the axis of the indenter and such that the local surface at the proposed indentation site is less than $\pm 5°$ from the perpendicular to the indentation axis.

NOTE Possible methods for determining local slope include viewing with a high magnification microscope and measuring the distance before the surface is out of focus. Knowledge of the depth of focus of the lens gives an estimate of the local slope; also the perpendicularity and local slope can be checked in practice by imaging the indent if it is made by a non-spherical indenter.

5.2 Surface roughness

Indentation into rough surfaces will lead to increased scatter in the results with decreasing indentation depth. Clearly when the roughness value, Ra, approaches the same value as the indentation depth the contact area will vary greatly from indent to indent depending on its position relative to peaks and valleys at the surface. The final surface finish should be as smooth as available experience and facilities permit. The Ra value should be less than 5 % of the maximum penetration depth whenever possible.

NOTE 1 It has been shown that for a Berkovich indenter, the angle that the surface normal presents to the axis of indentation has to be greater than 7° for significant errors to result [3]. The important angle is that between the indentation axis and the local surface normal at the point of contact. This angle may be significantly different from the average surface plane for rough surfaces, see Note 2.

NOTE 2 While Ra has been recommended as a practical and easily understood roughness parameter, it should be borne in mind that this is an average and thus single peaks and valleys may be greater than this as defined by the Rz value, although the likelihood of encountering the maximum peak, for example, on the surface is small. Modelling to investigate the roughness of the coating surface has concluded that there are two limiting situations for any Ra value. When the 'wavelength' of the roughness (in the plane of the coating surface) is much greater than the indenter tip radius, the force-penetration response is determined by the local coating surface curvature, but when the wavelength is much less than the tip radius, asperity contact occurs and the effect is similar to having an additional lower modulus coating on the surface.

NOTE 3 In cases where coatings are used in the as-received condition, random defects (such as nodular growths or scratches) might be present. Where an indentation site imaging system is included in the testing machine, it is recommended that "flat" areas away from these defects be selected for measurement.

The roughness profilometer probe radius should be comparable to the indenter radius. If the roughness parameter Ra is determined with an AFM on a scan area, the size of this area should be agreed upon between the customer and the measurement laboratory. A scan area of 10 µm × 10 µm is recommended.

Some instruments are capable of scanning the indentation site before indentation. In this case areas with the required local slope and roughness may be selected for indentation in surfaces that might otherwise, on average, be too rough.

5.3 Polishing

It should be appreciated that mechanical polishing of surfaces can result in a change in the work hardening and/or the residual stress state of the surface and consequently the measured hardness. For ceramics, this is less of a concern than for metals, although surface damage can occur. Grinding and polishing shall be carried out such that any stress induced by the previous stage is removed by the subsequent stage, and the final stage shall be with a grade of polishing medium appropriate to the displacement scale being used in the test. If possible, electrochemical polishing should be used.

NOTE 1 Many coatings replicate the surface finish of the substrate. If it is acceptable to do so, surface preparation problems can be reduced by ensuring that the substrate has an appropriate surface finish, thus eliminating the need to prepare the surface of the coating. In some cases, however, changing the substrate surface roughness may affect other coating properties therefore care should be taken when using this approach.

NOTE 2 In coatings, it is common to get relatively large residual stresses (e.g. arising from thermal expansion coefficient mismatch between the coating and the substrate and/or stress induced by the coating deposition process). Thus, a stress free surface would not normally be expected. Furthermore, stress gradients in coatings are not uncommon, so that removal of excessive material during a remedial surface preparation stage may result in a significant departure from the original surface state.

NOTE 3 Polishing reduces the coating thickness and so the effects of the substrate will be enhanced when indenting normal to the surface. Where the data analysis requires an accurate knowledge of the coating thickness indented, polishing will require re-measurement of coating thickness. This again emphasises the need to carry out minimum preparation.

5.4 Surface cleanliness

Generally, provided the surface is free from obvious surface contamination, cleaning procedures should be avoided. If cleaning is required, it shall be limited to methods that minimise damage, for example

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

— application of subliming particle stream of CO_2 (taking care not to depress the surface temperature below the dew point), and

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

If these methods fail and the surface is sufficiently robust, the surface may be wiped with a lintless tissue soaked in solvent to remove trapped dust particles, then the surface shall be rinsed in a solvent as above. Ultrasonic methods are known to create or increase damage to coatings and should be used with caution.

5.5 Special requirements for paints and varnishes

5.5.1 Substrate

Permitted substrates are steel, glass, aluminium, plastic and wood. Prepare the test panels as described in 5.5.2 and 5.5.3. Their surface should be free of visible damages. If samples are drawn from coated articles, care should be taken that they are plane and will not be bent when being cut. The test panels should, when under load, not yield or start to vibrate.

Small samples should be adequately supported to prevent deformation of the test sample during measurement.

5.5.2 Preparation and coating of the substrate

The substrate for the test shall be prepared as described in ISO 1514 and shall be coated with the product or system to be tested according to the procedure laid down. Coating thickness should be more than ten times the indentation depth when values specific for the material should be determined.

5.5.3 Drying and conditioning of the test coating

The coated test panel should be dried, hardened and aged for the established time and under the established conditions, with at least 24 h storage under standard conditions as described in ISO 3270. Coating thickness should be determined according to one of the methods specified in ISO 2808.

6 Procedure

6.1 Test conditions

6.1.1 The indenter geometry, maximum force and/or displacement and force displacement cycle (with suitable hold periods) shall be selected by the operator to be appropriate to the coating to be measured and the operating parameters of the instrument used, see Figure 2.

Hardness values are only valid if plastic deformation has occurred and there is a residual indentation after force removal.

NOTE 1 A typical 'small' radius for hardness measurement is that of a Berkovich indenter ($<$ 250 nm). A typical 'large' radius for modulus measurement is $<$ 25 µm. In certain cases, a change of indenter can be avoided by force selection. The range of elastic deformation can be estimated by the formulas of Annex B.

NOTE 2 An example of a simplified stress analysis is given in 7.3, Note 4.

6.1.2 Where multiple indentations normal to the surface or indentations in cross-section are planned, each indent shall be positioned and separated according to ISO 14577-1:2002, 7.7.

NOTE Coatings can display a high degree of anisotropy, and thus the orientation of the indenter within the plane and the direction of indentation (normal or cross-section) can significantly alter the measured value of the hardness and sometimes the modulus.

6.1.3 The parameters of the instrumented indentation test are defined according to ISO 14577-1:2002, 7.4.

The following parameters of coating/substrate influencing the measurement result should be considered:

a) substrate hardness, Young's modulus and Poisson's ratio;

b) coating thickness;

c) surface roughness;

d) adhesion of the coating to the substrate (delamination of the coating should be avoided).

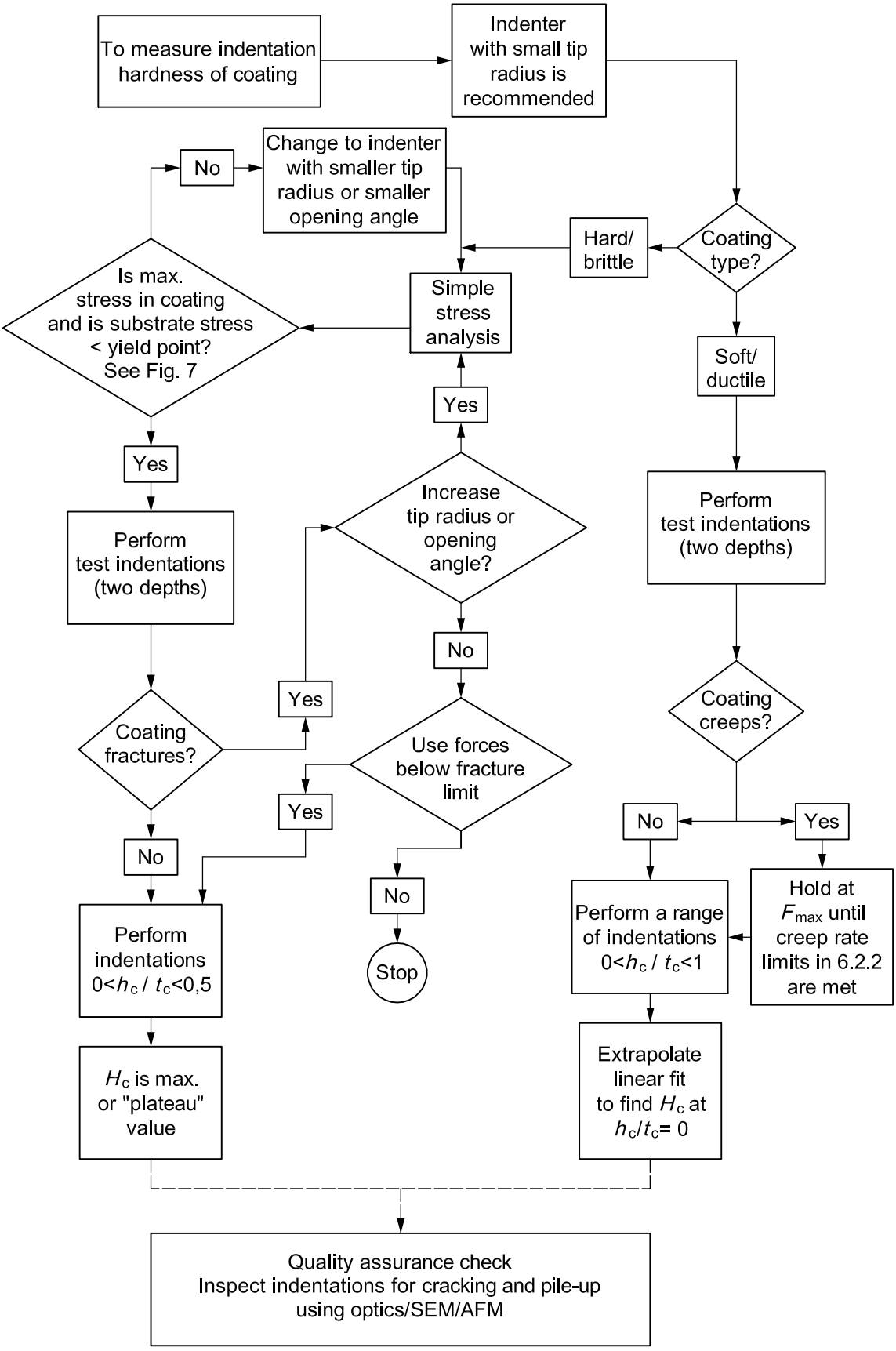
All these parameters should be kept constant if a direct comparison is to be made between two or more test pieces.

The time dependence of the material parameter being measured should be taken into account.

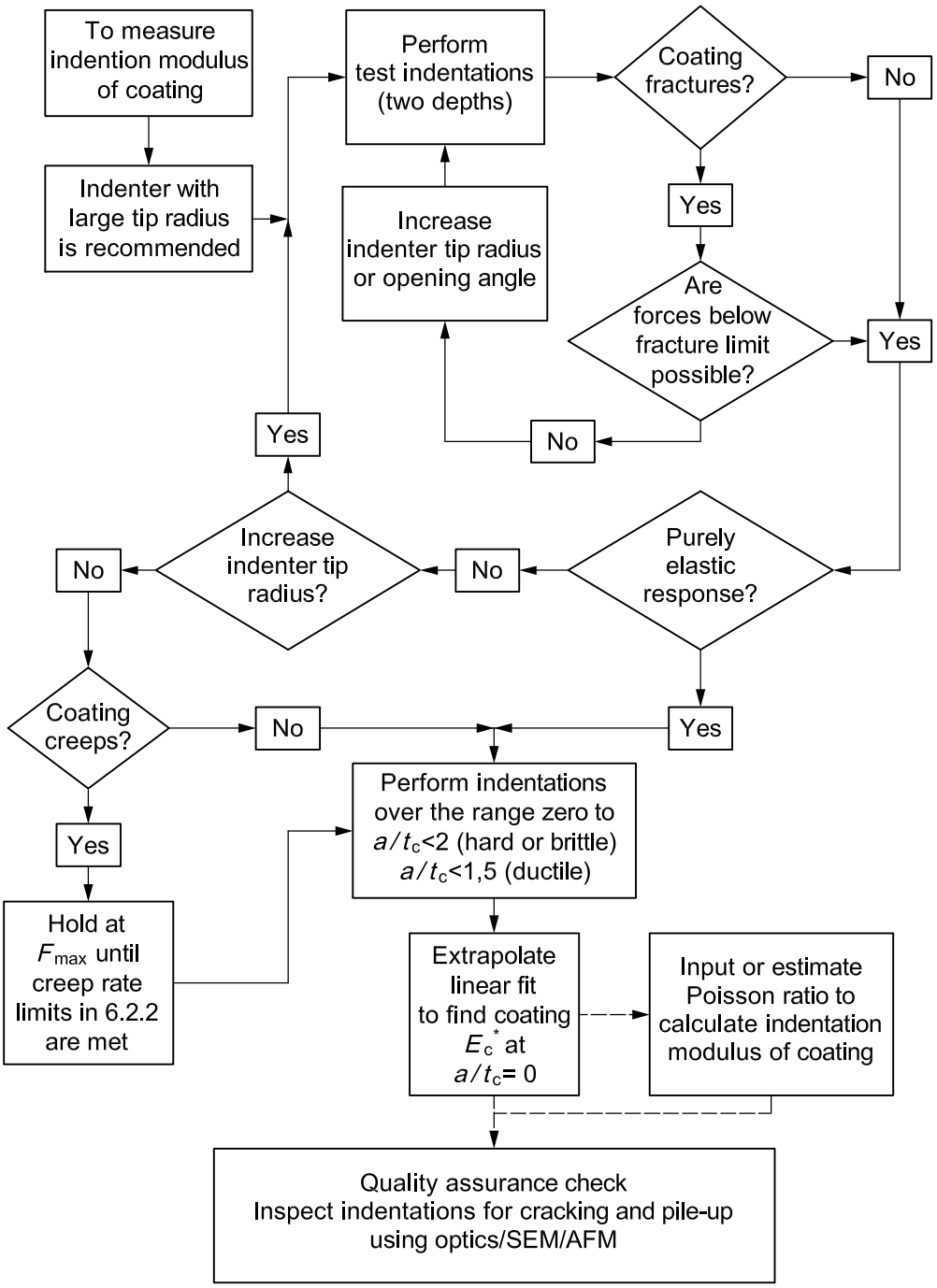
NOTE 1 Hardness and Young's modulus values can be affected by adhesion [4] to [8].

NOTE 2 Variations in test piece parameters other than hardness or modulus can affect measurement of these quantities. If the indentation depth is a sufficiently small fraction of the coating thickness, or the coating thickness may be reasonably well estimated and is constant for all indentation sites on a particular sample, it is possible to measure E_C* and H_C, without an accurate thickness measurement. If, however, the properties as a function of relative indentation depth are to be compared, an accurate thickness determination may be necessary. The exact limits depend on the ratio of properties of coating and substrate.

Normalizing procedures shall always be used when determining coating properties from coatings of different thickness.



a) For indentation hardness of coating



b) For indentation modulus of coating

Figure 2 — Flow chart for selection of indenter geometry and indentation parameters to measure coating properties

6.2 Measurement procedure

6.2.1 General

Introduce the prepared test piece and position it so that testing can be undertaken at the desired location.

Carry out the predetermined number of indentation cycles using the selected test conditions.

6.2.2 Force control experiments

A single force application and removal cycle shall be used. 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 as close to zero force as is practicable or during a hold at a suitable place in the force removal curve (e.g. after 90 % of the force has been removed). If a contact in the fully elastic regime (see Annex B) can be obtained, a hold at initial contact is preferred. In this way, material influences (creep, visco-plasticity, cracking) can be avoided. The hold period shall be sufficient to allow determination of the average displacement drift rate due to the instrument (e.g. due to temperature changes) and a linear correction shall be applied. The drift rate correction obtained in this way is only valid if the drift (as opposed to noise) in the displacement values determined during the hold period (wherever it is in the indentation cycle) is believed to result from purely instrumental effects (such as temperature) and not from indentation induced responses from the material (e.g. visco-elastic or anelastic creep, fracture, pressure induced phase transformations, etc.).

If material influences cannot be avoided, drift rates shall be measured before and after each indentation (e.g. using an elastic contact with a hard reference surface). Either the average linear drift rate may be calculated or the drift rate linearly interpolated over the time between the two measurements. The hold period shall be sufficient to allow determination of the drift in displacement due to temperature fluctuations. Drift (as opposed to noise) in displacement values determined during the hold period at 90 % force removal or at close to zero is believed to result from temperature changes and a linear correction should be applied.

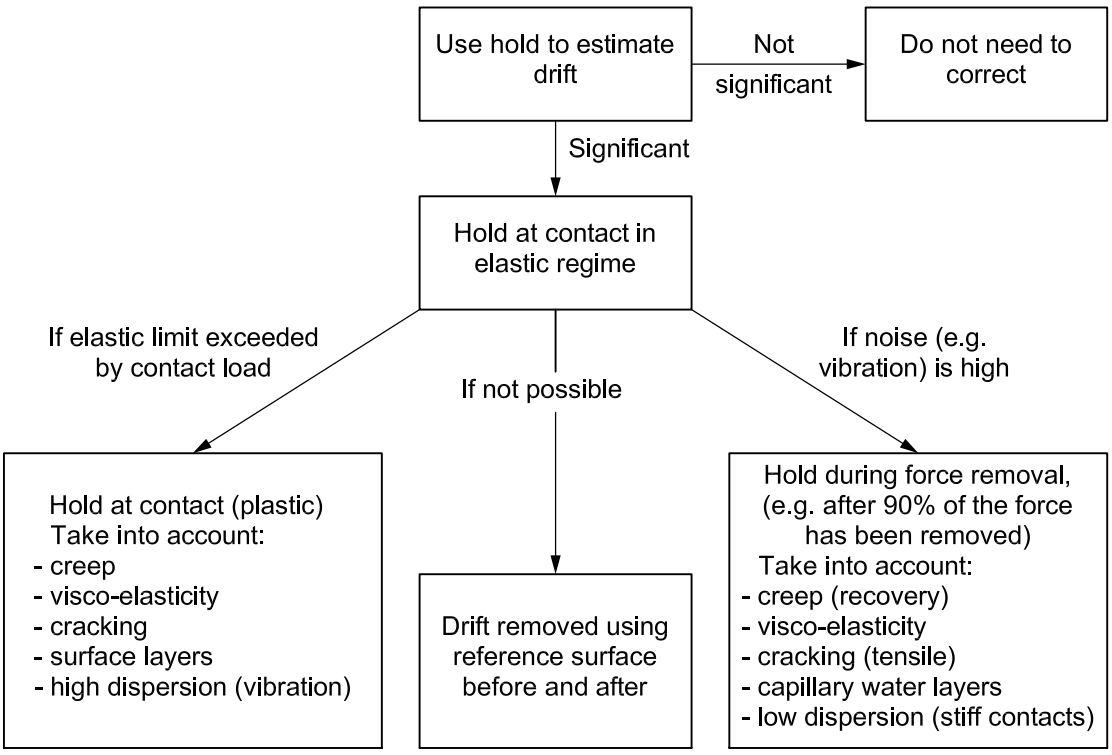


Figure 3 — Decision tree to assist in estimating the drift during a force controlled experiment

If no elastic contact can be obtained, there is no generally recommendable method. Depending on the material under investigation, a hold at initial force (e.g. visco-elastic material) or at 90 % removed force (e.g. soft material) may be preferred. Because of the stiffer contact (higher contact area) at 90 % removed force, dispersion of the data when using this method is generally lower. For difficult materials, a hold period at both ends of the indentation cycle may be included. It is recommended that the hold period be for at least the force application time, and the first 10 s to 20 s of the hold data should be discarded for the analysis since these initial data may be significantly influenced by time-dependent effects (material time-dependent deformation, formation of capillary surface layers) [9], [10].

A further hold period shall be performed at maximum force to allow for completion of any time dependent deformation. The minimum hold period length is therefore dependent on the instrument capability and the material being tested. The hold period shall be long enough and/or the time to remove force shall be short enough such that:

$$q_F > 10 \times q_C / C_t$$

where

q_F is the force removal rate;

q_C is the creep rate.

The creep rate is defined as the linear fit to the displacement vs. time for the last $>$ 30 data points before force removal begins.

NOTE 1 The error in measured compliance (due to the creep rate at the point of force removal) depends on the range of force removal data fitted, the fit algorithm used, the absolute contact compliance and the rate of force removal. An estimate of the worst case error in the measured contact compliance can be calculated using the following formula:

$$\text{error} = \frac{q_C}{\left[\left(\frac{\sqrt{\pi}}{2} \cdot \frac{\sqrt{H}}{E_r} \cdot \frac{1}{\sqrt{F_{max}}}\right) \cdot \left(\frac{dF}{dt}\right)\right]} \times 100\ \% \qquad (1)$$

The hardness (and modulus) may be depth dependent particularly if a non-self-similar indenter is used.

NOTE 2 When visco-elastic materials are tested, the drift rate will not necessarily reduce by increasing the hold at maximum force. Even if it does, the drift rate will reverse when the force is removed as visco-elastic recovery begins. The practice of using a measurement of creep rate just before force removal to apply a creep rate correction to the force removal data is not recommended. The elastic modulus of visco-elastic materials is better tested using an indentation cycle faster than the visco-elastic time constant or by using dynamic (ac) indentation methods.

Force application and removal rates may be the same but it is recommended that the removal rate should be higher than the application rate (if possible) to minimize the influence of creep. Slower force application reduces the hold period length required at F_{max} to achieve the necessary reduction in creep rate.

NOTE 3 The influence of the material creep behaviour on hardness and modulus results has been reported [9]. The results show that, especially for materials with low hardness-to-modulus ratio (which includes most metals) the modulus results are not reliable if the hold period is too short. A modulus error, due to creep, of more than 50 % can arise. The variation of the hold period produced a hardness change of up to 18 %. Reference [9] proposes hold periods dependent on the material type that range from 8 s for fused quartz to 187 s for aluminium. The criterion used was that the creep rate should have decayed to a value where the depth increase in one minute is less than 1 % of the indentation depth. It should be noted that creep of 1 % of the total indentation depth may cause a large change in the apparent contact stiffness in nearly perfectly plastic materials such as metals.

It is recommended that the creep rate be assessed in preliminary experiments. The force removal rate should be the highest possible that still ensures sufficient force removal data for the subsequent analysis.

6.2.3 Displacement control experiments

Hold periods shall be imposed as for the force control experiments by holding at constant force to determine drift. Target displacement rates should be corrected for the displacement drift rate. These rates should also be corrected for frame compliance.

7 Data analysis and evaluation of results for indentation normal to the surface

7.1 General

Before the data obtained during the indentation experiments can be analysed, it is necessary to have corrected the displacement data for significant thermal drift, determined the values of $A_p(h_c)$ and obtained C_s (the contact compliance) by correcting the data for the instrument frame compliance, C_f. The hardness and indentation modulus of the test piece can then be calculated using equations in ISO 14577-1:2002, Annex A. Annex A of this part of ISO 14577 describes the determination of C_s and C_f. The properties thus calculated according to ISO 14577-1 are composite properties for the coating/substrate combination. 7.2 and 7.3 provide methods for extracting the hardness and indentation modulus of the coating from the composite properties measured assuming that the coating properties are constant with depth.

NOTE 1 For indentation into a cross-section, the values obtained using ISO 14577-1 can be considered to be those of the coating, provided that the recommendations in 6.1.2 have been followed.

NOTE 2 Empirical guidelines are given in Reference [11] for hardness measurement of electroplated coatings on steels, where it is recommended that the indentation depth does not exceed one tenth the thickness of the coating, while for paint films [12] penetration of up to one third the coating thickness may be allowed. These approximations can be unsatisfactory in many cases and only apply to hardness measurements.

Test parameters for ductile and brittle coatings shall be considered separately.

For indentation normal to the surface, elastic deformation of the substrate will always occur for all coatings, even though this could be negligibly small for a thick compliant coating on a stiff substrate. Thus the measured modulus will always be the composite modulus of the coating and substrate and the value obtained will be a function of indentation depth.

For hardness measurement, it is recommended to use as small a radius indenter as possible (i.e. as sharp as possible) to limit the plastic deformation to be within the coating. A measurement of the uncoated substrate hardness is a useful guide to the appropriate choice of analysis (soft vs. hard). In some circumstances, it is possible to identify a range of indentation depth over which the measured hardness is constant (i.e. before the onset of substrate plastic deformation) and then carry out indentation experiments within this range.

Estimates of coating hardness and modulus may be extracted from the composite values E_{IT}^*, H_{IT} obtained from indentation normal to the surface by expressing those composite values as a function of contact radius a or contact depth h_c normalized to coating thickness. Measurement of coating thickness, t_c, is not required to obtain an accurate intercept value. However, if data from different thickness coatings are to be plotted together, or the maximum range of indentation depth for valid data is to be used, it is recommended to make a measurement of actual coating thickness to ensure the best reproducibility of results. For indenters of different geometries (e.g. Berkovich, Vickers, spherical, cone, etc.), a is approximated by the radius of a circle having the same area as the projected area of contact with the indenter:

$$a = \sqrt{\frac{A_p}{\pi}} \tag{2}$$

This value has exact equivalence for a spherical or conical indenter but becomes increasingly less physically meaningful as the axial symmetry of the indenter reduces, i.e. cone = sphere > Vickers > Berkovich.

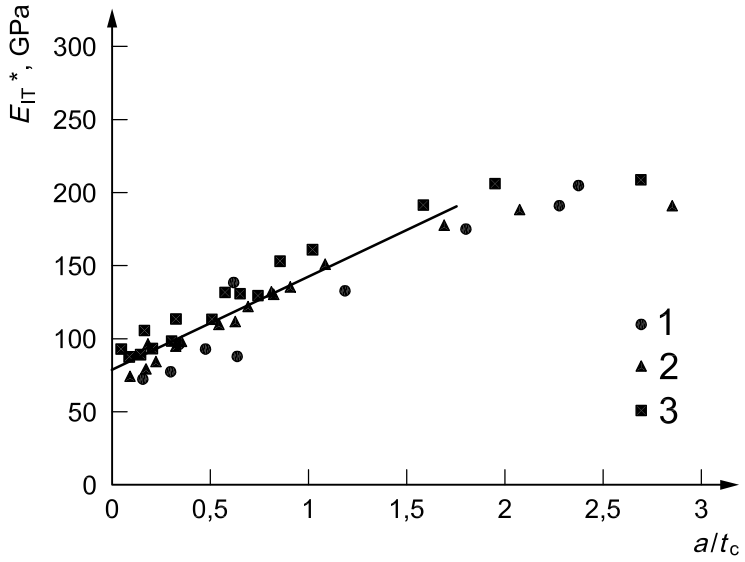
NOTE 3 It is relatively easy to measure the hardness of ductile coatings or the elastic modulus of brittle coatings. It is more difficult to determine the hardness of brittle or hard coatings or the elastic modulus of ductile coatings.

NOTE 4 Where t_c is not measured, nominal values of t_c may be used but comparison of data between coatings of different thicknesses will be less accurate.

7.2 Coating indentation modulus

In the case of force-controlled cycles and test pieces of unknown indentation response, a set of trial indentations shall be performed (e.g. at two widely spaced forces) and analysed to obtain estimates of the test force required for the range of a/t_c specified below. See Figure 2 b) for the selection of suitable indenter geometry and indentation parameters.

In the case of soft/ductile coatings, indentation force or displacement and indenter geometry shall be chosen such that data shall be obtained in the region where $a/t_c < 1{,}5$. The plane strain indentation modulus of the coating E_c^* is obtained by taking a series of measurements at different indentation depths and extrapolating a linear fit to plane strain indentation modulus vs. a/t_c to zero, see Figure 4.



Key

1 spherical indenter
2 Berkovich indenter
3 Vickers indenter

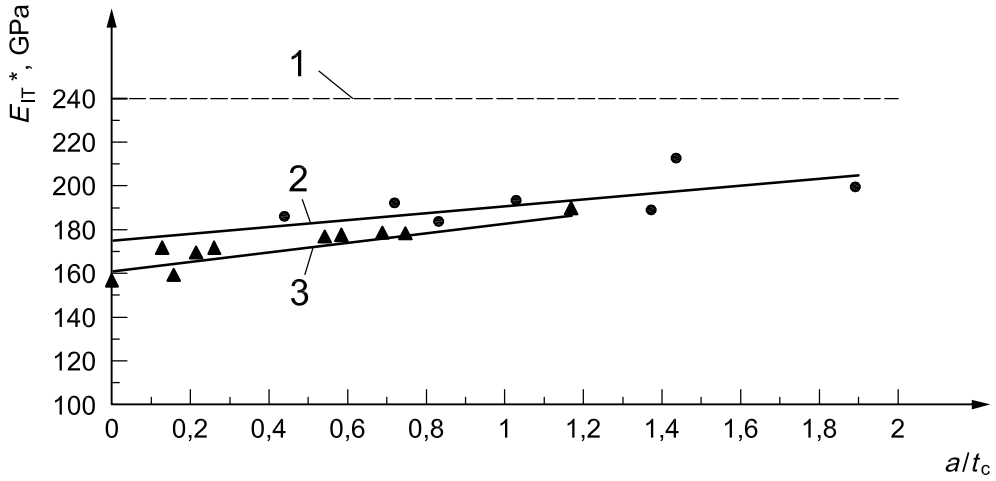
Figure 4 — Plane strain indentation modulus vs. normalized contact radius of Au on Ni, selected data for spherical, Berkovich and Vickers indenter

In the case of hard/brittle coatings, the indentation force or displacement and the indenter geometry shall be chosen such that data is obtained in the region $a/t_c < 2$. The plane strain indentation modulus of the coating, E_c^*, is obtained by taking a series of measurements at different indentation depths and extrapolating a linear fit to the measured test piece plane strain indentation modulus, E_{IT}^*, vs. a/t_c to zero, see Figure 5.

NOTE 1 A linear fit to plane strain indentation modulus vs. a/t_c to zero is a first approximation. However, in general, a non-linear relationship appears to apply and can be reproduced by finite element analysis (FEA). The exact nature of this non-linear relation is not known and so a linear fit over the restricted range indicated is a robust first approximation but is not applicable over a range wider than this.

At least 15 measurements at 3 or more different values of a/t_c or h_c/t_c shall be obtained before a valid extrapolation is possible. It is recommended that at least 50 measurements at 5 or more values of a/t_c or h_c/t_c be obtained to reduce the uncertainties of the extrapolation. In general, it is recommended to increase the number of measurements made at different a/t_c or h_c/t_c values, in preference to increasing the replications at fewer a/t_c or h_c/t_c points.

NOTE 2 The quickest and most reliable method for determining the range of applied forces required to obtain indentation results in the required range of a/t_c or h_c/t_c is to perform a couple of trial indentations at different forces. Quick estimates of the likely h_c values for lower maximum applied forces can be obtained by drawing parallel lines to the tangent to the initial force removal curve.



Key

1 plane strain indentation modulus of M2 tool steel substrate

2 linear fit for values determined with spherical indenter

3 linear fit for values determined with Berkovich indenter

Figure 5 — Plane strain indentation modulus vs. normalized contact radius of diamond-like carbon (DLC) on hardened tool steel (M2)

7.3 Coating indentation hardness

The same normalized parameter, a/t_c, can be used for the evaluation of hardness results. However, since this method requires self-similar geometry indenters (pointed indenters), the non-dimensional parameter h_c/t_c (ratio of contact depth to coating thickness) can also be used. See Figure 2 a) for the selection of suitable indenter geometry and indentation parameters.

In the case of soft, ductile coatings on a harder substrate, the coating indentation hardness is obtained from a linear extrapolation to zero of an H_{IT} vs. h_c/t_c plot over the range $0 < h_c/t_c < 1$, provided the substrate is deformed only elastically.

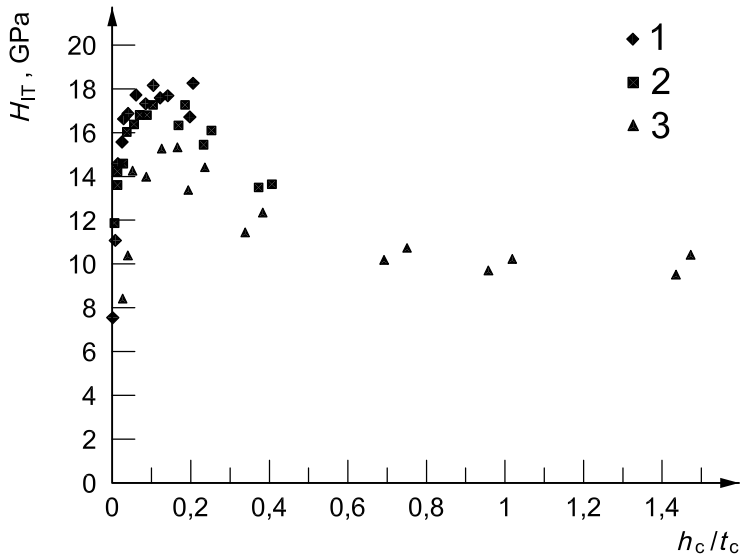
NOTE 1 The maximum limit of h_c/t_c for a linear plot depends on the hardness ratio of the coating and substrate. For example, for an Au coating on Ni, the hardness ratio is ~ 2,5 and the h_c/t_c limit is < 1; for an Al coating on optical glass (BK7) with a hardness ratio of ~ 8, the h_c/t_c limit is ~ 5. There is a lower limit of the contact depth to measure the hardness due to the tip rounding. A reproducible hardness value can be obtained for $h_c > 20$ % of tip radius (for a 250 nm tip radius, this is 50 nm).

It is recommended that at least 50 measurements at 5 or more values of a/t_c or h_c/t_c be obtained to reduce the uncertainties of the extrapolation. In general, it is recommended to increase the number of measurements made at different a/t_c or h_c/t_c values, in preference to increasing the replications at fewer a/t_c or h_c/t_c points. This is particularly the case for hard/brittle coatings on a softer substrate, where the plateau of hardness values is to be determined.

In the case of hard coatings on a softer substrate, the coating indentation hardness can only be determined with a sharp (small tip radius) indenter that causes yielding within the coating. It is recommended that an elastic stress analysis of the coating/substrate system be undertaken using the approximation of a spherical indenter of a radius equivalent to the tip radius of the self-similar geometry indenter. This will determine whether the coating or the substrate will yield first during indentation and, therefore, whether it is possible to determine the coating hardness at all. It is recommended that hardness values for the substrate be obtained for comparison, by testing if necessary. Delamination or fracture of the coating can be recognized by the hardness values obtained clustering at the substrate value, even at low h_c/t_c. Note, sharper indenters may cause fracture at lower forces than more blunt indenters.

The indentation force or displacement and indenter geometry shall be chosen such that h_c/t_c (or ~ a/t_c) is in a range where H_{IT} is a maximum. Commonly, the range is $0 < h_c/t_c < 0,5$. If a constant maximum value of H_{IT} (a

plateau) is observed over this range, this is the coating indentation hardness H_c. If only a maximum in H_{IT} occurs and indentation of a thicker coating yields the same value, then this is a strong indicator that this is the value for the coating. Otherwise this is only the minimum estimate of the coating indentation hardness, see Figure 6.



Key

1 t_c = 2 510 nm

2 t_c = 1 470 nm

3 t_c = 460 nm

NOTE 1 Selected data show the effect of substrate yield for the thinnest coating thickness. Data are from Berkovich indenter geometry (the indentation hardness of the coating is H_c = 18 GPa).

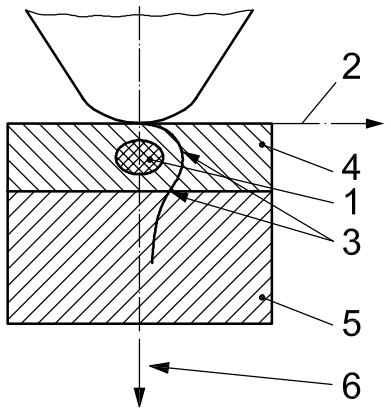
Figure 6 — Indentation hardness vs. normalized contact depth of diamond-like carbon (DLC) on tool steel M2

NOTE 2 The extent of substrate plastic deformation will depend upon a number of factors, including the relative difference in hardness and modulus between the coating and the substrate, adhesion, the coating thickness, the indenter radius of curvature ('sharpness') and the maximum force. Premature yielding of the substrate can be a particular problem in the case of hard and stiff coatings on softer substrates. However, if the film modulus is much less than the substrate modulus, premature yielding of the substrate can also be caused (e.g. SiO_2 on tungsten). There is a compromise between

a) using a sufficiently high force (e.g. close to but not exceeding the limit corresponding to the onset of plastic deformation of the substrate) in order to obtain the maximum of force-depth data thereby improving the precision of the measurement, and

b) indenting at a low enough displacement such that the plastic zone of the indentation does not interact with the substrate/coating interface, thus minimizing the influence of the substrate on the measurement; see Figure 7.

NOTE 3 Sharper indenters (lower radius tip and corners and/or smaller tip included angles) generally cause fracture at lower forces than blunter or lower aspect ratio indenters. Cracking can often be detected as sudden discontinuities or "pop-in" events in the load displacement curves of force-controlled indentations.



Key

1 plastic deformation
2 overlay of principal shear stress vs. depth under indenter
3 maximum shear stress in each material
4 coating
5 substrate
6 depth

NOTE Coating hardness is only measurable if coating yields first.

Figure 7 — Diagram of the principal shear stress as a function of depth under the indenter overlaid onto a diagram of an indentation into a coated substrate

NOTE 4 To measure the coating hardness, there should be sufficient yielding of the coating before the substrate yields. The best conditions for this are when the maximum of the principal shear stress occurs inside the coating and causes plastic deformation whilst the stress in the substrate below does not exceed the substrate yield stress. In a spherical contact, the maximum of the principal shear stress is approximately 0,47 (mean pressure) for $\nu = 0{,}3$ at $0{,}5a$ below the surface.

NOTE 5 The quickest and most reliable method for determining the range of applied forces required to obtain indentation results in the required range of a/t_c or h_c/t_c is to perform a couple of trial indentations at different forces. Quick estimates of the likely h_c values for lower maximum applied forces can be obtained by drawing parallel lines to the tangent to the initial force removal curve.

NOTE 6 Different procedures have been published which suggest methods that may be used to determine the onset of substrate plastic deformation by the evaluation of the force increasing branch of the indentation hysteresis curve, but none has yet been validated by the international community. These involve a method [13] in which the differential of the force with respect to displacement is plotted versus displacement, and the point of inflection is taken to be the depth at which plastic deformation of the substrate occurs. It has been proposed [14] that departure from a linear relationship of force versus the square of displacement is also an indication of the onset of plastic deformation of the substrate. However, there is no guarantee that the yield or deviation detected is that of the substrate. Also, there is always a slight deviation from the linear relationship especially for depths where the tip rounding has an influence.

NOTE 7 Both analytical and numerical models have been proposed for indentation of coating systems. Analytical models are usually only applicable to elastic deformation. As with the experimental approach, none of these have yet been validated by the international community. References [15] to [26] list some of the approaches being developed. For multilayer coatings or coatings with a graduation in properties, modelling is clearly much more difficult, and no generally accepted models are available. If wholly elastic measurements are possible, for instance using spherical indenters with a large enough radius, an exact calculation of the coating modulus from the measured composite value is possible [10], [27] to [29].

8 Test report

The test report shall be in accordance with ISO 14577-1 and shall contain the following additional information:

a) manufacturer, model, unique identifier of testing machine;

b) description of the test piece (e.g. dimensions, nominal coating thickness, coating material and number of layers); if known, it is recommended to report

— test piece preparation,

— surface roughness Ra,

— substrate properties (composition, hardness and Young's modulus), and

— actual coating thickness at the position of indentation;

c) results of the test (plane strain indentation modulus of the coating and/or indentation hardness of the coating and maximum limit of error in C_t due to creep) together with the uncertainty;

d) procedures adopted for calibration of force, displacement, frame compliance and indenter area function (including indenter tip radius), including the choice of reference material used for calibration and verification of instrument repeatability;

e) statement whether a correction for thermal drift was applied and if so the method used;

f) distance between indentations.

Uncertainties should include the following: force, displacement, zero point, area function, frame compliance, creep rate, extrapolation intercept (e.g. using methods similar to MS Excel LINEST regression analysis).

NOTE Procedures for the estimation of uncertainty will be developed during the revision of parts 1, 2 and 3 of ISO 14577.

Annex A
(normative)

Frame compliance calibration procedure

A.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. Some procedures need reference materials of known Young's modulus and Poisson's ratio. The selected procedure, including the ranges of test force F and indentation depth h for the performed calibration, shall be reported.

NOTE Ideally, reference materials are required with certified properties (Certified Reference Materials, CRMs). CRMs are not yet available, but work has started on their development. The need for CRMs arises from the fact that surface layers are present on most materials. These will have an increasing influence on the measured elastic and plastic properties at small indentation depths decreasing the accuracy of the indenter area function and frame compliance values at these depths. Currently recommended materials are freshly polished tungsten and fused silica. The forces used should not exceed the threshold at which cracking occurs (~ 80 mN to 100 mN for an average radius Berkovich tip indenting fused silica).

A.2 Principle

The total measured compliance, C_t, is the sum of contact compliance, C_s, and the frame compliance, C_f, thus:

$$C_t = C_s + C_f \tag{A.1}$$

where C_t is derived from the derivative of the (uncorrected) test force removal curve at maximum force

$$C_t = \left[\frac{dF}{dh}\right]^{-1} \tag{A.2}$$

Some instruments use tip-calibration routines that automatically assign frame compliance values. If this is the case, the frame compliance determined (e.g. by the two reference material iterative methods in this annex), should be summed with any frame compliance assumed by the software in determining the area function of the tip.

C_s is the contact compliance of the specimen material

$$C_s = \frac{\sqrt{\pi}}{2\,E_r} \cdot \frac{1}{\sqrt{A_P(h_c)}} \tag{A.3}$$

with

$$\frac{1}{E_r} = \frac{1}{E_{IT}^*} + \frac{1-\nu_i^2}{E_i} \tag{A.4}$$

and (see A.4 in ISO 14577-1:2002)

$$h_c = h_{max} - \varepsilon F_{max} C_t \tag{A.5}$$

where E_i and ν_i are, respectively, the Young's modulus and the Poisson ratio of the indenter.

Thus the total compliance is

$$C_t = \frac{\sqrt{\pi}}{2\,E_r} \cdot \frac{1}{\sqrt{A_p(h_c)}} + C_f \tag{A.6}$$

or

$$C_t = \frac{\sqrt{\pi}}{2\,E_r} \cdot \frac{\sqrt{H_{IT}}}{\sqrt{F_{max}}} + C_f \tag{A.7}$$

using the definition of the indentation hardness H_{IT} (see A.4 in ISO 14577-1:2002).

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

Table A.1 — Required assumptions of the selected method

Method	E_r = const.	H_{IT} = const.	C_f = const.	Parameter input needed	Ref.
1	yes	yes	yes	none	
2	yes	no	yes	$A_p(h_c)$	[32]
3	yes	no	yes	E_r	[33]
4	yes	no	no	$A_p(h_c)$, E_r	[30]
5	yes	no	no	E_{r1}, E_{r2}	[31]
6	yes	no	no	E_{r1}, E_{r2}, elastic deformation	[28]

A.3 Methods

Before using an automatic calibration function, the user should check that they know exactly what it does. Some software will also calculate a frame compliance value. This shall be taken into account when calculating the final frame compliance obtained from the methods described here.

A.3.1 Method 1

If the reduced modulus, E_r, and the indentation hardness, H_{IT}, are constant, a plot of C_t (uncorrected for frame compliance) versus $1/\sqrt{F_{max}}$ [Equation (A.7)] is linear and intersects the compliance axis at the frame compliance, C_f. Method 1 cannot be used for spherical indenters or for large tip radius indenters at small depths.

NOTE 1 The suggested plot will not be linear if H_{IT} is not constant. This will occur if there is a genuine variation in the plastic properties with depth or if the indentation does not reach indentation depths or mean indentation pressures sufficient to form a fully developed plastic zone.

NOTE 2 Hardness as defined in ISO 14577-1 is the material response to indentation by a particular shape of indenter. At low indentation depths, an indenter can be changing from a more self-similar shape to an effectively spherical shape. Hardness is therefore not constant and the measured E/H ratio of the material will vary with indentation depth.

A.3.2 Method 2

If the indentation hardness cannot be assumed to be independent of the indentation depth, the area function, $A_p(h_c)$, is determined independently, for example by replica [32] or atomic force microscopy. A plot of C_t (uncorrected for frame compliance) versus $1/\sqrt{A_p(h_c)}$ [Equation (A.6)] is linear and intersects the compliance axis at the frame compliance, C_f.

NOTE Maximum test forces are typically in the 10 mN to 100 mN range. A minimum of ten 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.

A.3.3 Method 3

If the area function is not known, a combined iterative procedure is used. Using the area function of the perfect indenter (ISO 14577-1:2002, A.4) and E_r to be calculated from the certified value of the reference material, an initial estimation of C_f is obtained by plotting C_t (uncorrected for frame compliance) versus $1/\sqrt{A_p(h_c)}$ [Equation (A.6)] for the two largest indentations [33]. Then the area function of the other indentation sizes are calculated from indentations by rearranging Equation (A.8)

$$A_p(h_c) = \frac{\pi}{4}\frac{1}{E_r^2}\cdot\frac{1}{(C_t - C_f)^2} \tag{A.8}$$

Using the new area function, the estimation of C_f is repeated with Equation (A.6). The new values of C_f influence the area function after Equation (A.8). The procedure is iterated several times until convergence is achieved.

NOTE 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 ten times. Fused silica is used to extend the area function to small distances from the tip.

A.3.4 Method 4

If a reference material of known modulus and the area function are available, then by direct use of Equation (A.6) and substituting values for E_r, $A_p(h_c)$ and C_t, the frame compliance C_f can be calculated. The first estimation of the frame 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 frame compliance is then calculated using Equation (A.6). The whole procedure is iterated until convergence is achieved [30].

NOTE Tungsten is the preferred reference material because it is an elastically isotropic, homogeneous material, and it is less prone to handling damage compared to aluminium. Moreover, it has a high modulus and allows sufficient plastic deformation to give a high contact stiffness, yielding more robust values of the frame compliance. Test forces up to 80 mN and a minimum of ten replicate indentations are 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) is well known (e.g. by independent AFM measurement), because the result is sensitive to that input.

A.3.5 Method 5

If the area function is not known, the combined iterative procedure of method 4 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 frame 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 frame compliance and indenter area function could be obtained, and the latter one agreed to the area function by an independent AFM measurement [31].

NOTE 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 ten times.

A.3.6 Method 6

If the indenter tip is approximately a sphere, an elastic indentation occurs in the initial range of force application (for criteria, see Annex B). For a spherical indenter, the frame 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} [28]:

$$R = \left(\frac{3}{4}\right)^2 \cdot \frac{F^2}{\left(h_1 - h_2\right)^3} \left(\frac{1}{{E_{r1}}^{2/3}} - \frac{1}{{E_{r2}}^{2/3}}\right)^3 \tag{A.9}$$

$$C_f = \frac{h_1 - \left(\frac{3F}{4E_{r1}}\right)^{2/3} \left(\frac{1}{R}\right)^{1/3}}{F} \tag{A.10}$$

The result is valid for only one value of the test force, F, and only for a range of the indentation depths between h_1 and h_2.

A.3.7 Method 7

The principle of the direct frame compliance method [34] is to apply the force while reducing contact compliance so that the total measured compliance can be used as the machine frame compliance. The method removes the requirement to know the diamond area function by replacing the indenter with a large flat punch indenter ($>$ 2 mm diameter). The punch is ground to be conformal to a polished Al sample by holding the two together and pulling a strip of fine grinding paper between them. The punch is then fixed to the sample using either a thin layer of cyanoacrylate or solder. The force-displacement curve is measured as the force is increased to the peak load of the instrument. The compliance C_f is determined by a linear fit to the force-displacement curve (Hooke's law, equation). It is recommended to take an average of 5 force-displacement curves.

This value is a valid measurement of the instrument frame compliance if the flat punch indenter makes an equivalent contribution to the total machine compliance as the test indenter. This assumption shall be validated by performing an indirect validation with the indenter to be used in subsequent testing.

If the slopes of the force application and removal curves are coincident, then thermal drift is negligible. Otherwise the effect of thermal drift should be accounted for.

Annex B
(normative)

Contact point and fully elastic regime

For an accurate determination of the indentation depth, it is essential to be able to determine the point at which the indenter first touches the surface of the test piece. The contact point is determined by the first detected change of the test force, the contact stiffness or the rates of force or displacement during the initial loading cycle, or the phase shift between tip movement and stimulation if a vibrating tip is used. Various instruments use different methods to determine the contact point, but the step size in force, displacement, force rate, displacement rate or phase during the approach and subsequent force application shall be small enough to allow determination of the zero point to the required uncertainty (see 7.3 of ISO 14577-1:2002).

If initial data in the fully elastic regime are obtained, the determination of the contact point can be done by back-extrapolation using the Hertzian analytical relationships for spherical indentation [1], [28], [31].

NOTE 1 The uncertainty in the zero point is affected by inhomogeneity of the sample (including the cracking of native oxide layers), surface roughness, the indenter geometry, the noise on the data (e.g. due to vibration), how well the mathematical function fits the trend in the data and to the length of extrapolation (i.e. the size of the initial force). When indenting surfaces of homogeneous materials with an average roughness (at the indentation site) of less than 1 nm, where the data have a displacement noise of less than 1 nm and are extrapolated back from an initial force of between 0,002 mN and 0,05 mN using a mathematical function that closely describes the form of the data, it is possible to achieve zero point uncertainties in the range 0,1 nm to 2 nm. However, this estimate should not be relied upon in any specific instance as the actual uncertainty can be strongly affected by any or all of the factors identified above.

The indentation depth (fully elastic displacement), h, and the radius of contact, a, are given by

$$h = \left(\frac{3F}{4E_{\mathrm{r1}}} \right)^{2/3} \cdot R^{-1/3} \tag{B.1}$$

$$a = \sqrt{2 \cdot h \cdot R \cdot \left(1 - \frac{h}{2R} \right)} \tag{B.2}$$

An elastic contact can be assumed if an indentation test with a maximum test force of at least two times the contact force shows sufficiently small differences between the curves of force application and force removal. After the thermal drift correction the differences shall not exceed 1 nm or 3 % of the maximum depth (the smaller value shall be used).

NOTE 2 First estimates for the displacement error introduced by an uncorrected depth offset for a given initial force can be derived from elastic theory [Equation (B.1)]. In Table B.1, elastic displacements for a range of material Young's moduli and indenter radii are given for initial contact forces of 1 µN, 5 µN and 10 µN. This is a minimum estimate assuming no plasticity. A fingerprint for the above limit of the elastic regime is given in Table B.2 but an experimental proof is needed.

NOTE 3 In all cases, consideration should be given to the likely influence of (capillary) surface films on the surface detection or definition. These films can act to place additional unmeasured forces on the indentation and hence obscure the real material response.

Table B.1 — Elastic indentation depth for different indenter radii

	Test force μN	Elastic indentation depth (nm) for different indenter radii (nm)					
		50	100	200	500	1 000	10 000
E = 5 GPa, ν = 0,3	1	7,22	5,72	4,55	3,35	2,66	1,23
	5	21,11	16,75	13,30	9,80	7,78	3,61
	10	33,51	26,60	21,11	15,55	12,34	5,73
E = 20 GPa, ν = 0,3	1	2,89	2,30	1,82	1,34	1,07	0,49
	5	8,46	6,71	5,33	3,93	3,12	1,45
	10	13,42	10,66	8,46	6,23	4,95	2,30
E = 70 GPa, ν = 0,3	1	1,29	1,03	0,81	0,60	0,48	0,22
	5	3,78	3,00	2,38	1,76	1,39	0,65
	10	6,01	4,77	3,78	2,79	2,21	1,03
E = 100 GPa, ν = 0,3	1	1,04	0,82	0,65	0,48	0,38	0,18
	5	3,04	2,41	1,91	1,41	1,12	0,52
	10	4,82	3,82	3,04	2,24	1,78	0,82
E = 200 GPa, ν = 0,3	1	0,69	0,55	0,44	0,32	0,25	0,12
	5	2,02	1,61	1,27	0,94	0,75	0,35
	10	3,21	2,55	2,02	1,49	1,18	0,55
E = 400 GPa, ν = 0,3	1	0,48	0,38	0,30	0,22	0,18	0,08
	5	1,41	1,12	0,89	0,65	0,52	0,24
	10	2,23	1,77	1,41	1,04	0,82	0,38
Test force displacement cycles shall be performed and results calculated using the analysis method described in A.3.6. The values of hardness and modulus derived shall be checked for their reasonableness. Elastic parameters of the diamond indenter are E = 1 140 GPa, ν = 0,07.							

Table B.2 — Calculated indentation depth for different Young's modulus materials, assuming a perfectly elastic response, according to Equation (B.1)

	Indenter radius = 50 nm		**Indenter radius** = 500 nm		**Indenter radius** = 10 000 nm	
	F (μN)	h (nm)	F (μN)	h (nm)	F (μN)	h (nm)
E = 5 GPa, ν = 0,3	1	7,2	1	3,4	1	1,2
	1	7,2	100	72	40 000	1 444
E = 70 GPa, ν = 0,3	1	1,3	1	0,6	1	0,2
	14	7,5	1 400	75	600 000	1 574
E = 200 GPa, ν = 0,3	1	0,7	1	0,3	1	0,1
	50	9,4	5 000	94	1 800 000	1 750
NOTE The depths calculated are for a 1 μN force and for the force required to exert a mean indentation pressure of 10 % of the Young's modulus (E/10 is the estimated maximum pressure any known material can withstand before plastic deformation will occur).						

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