# INTERNATIONAL STANDARD

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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/μm$^2$ [b] | ✓ |
| $\nu_i$ | Poisson's ratio of the indenter [a] | — | — |
| $\nu_s$ | 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/μm$^2$ [b] | — |
| $E_c$* | Plane strain indentation modulus of the coating [c] | mN/μm$^2$ | — |
| $E_{IT}$* | Plane strain indentation modulus | mN/μm$^2$ [b] | ✓ |
| $E_r$ | Reduced modulus of the indentation contact | mN/μm$^2$ [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/μm$^2$ = 1 GPa.

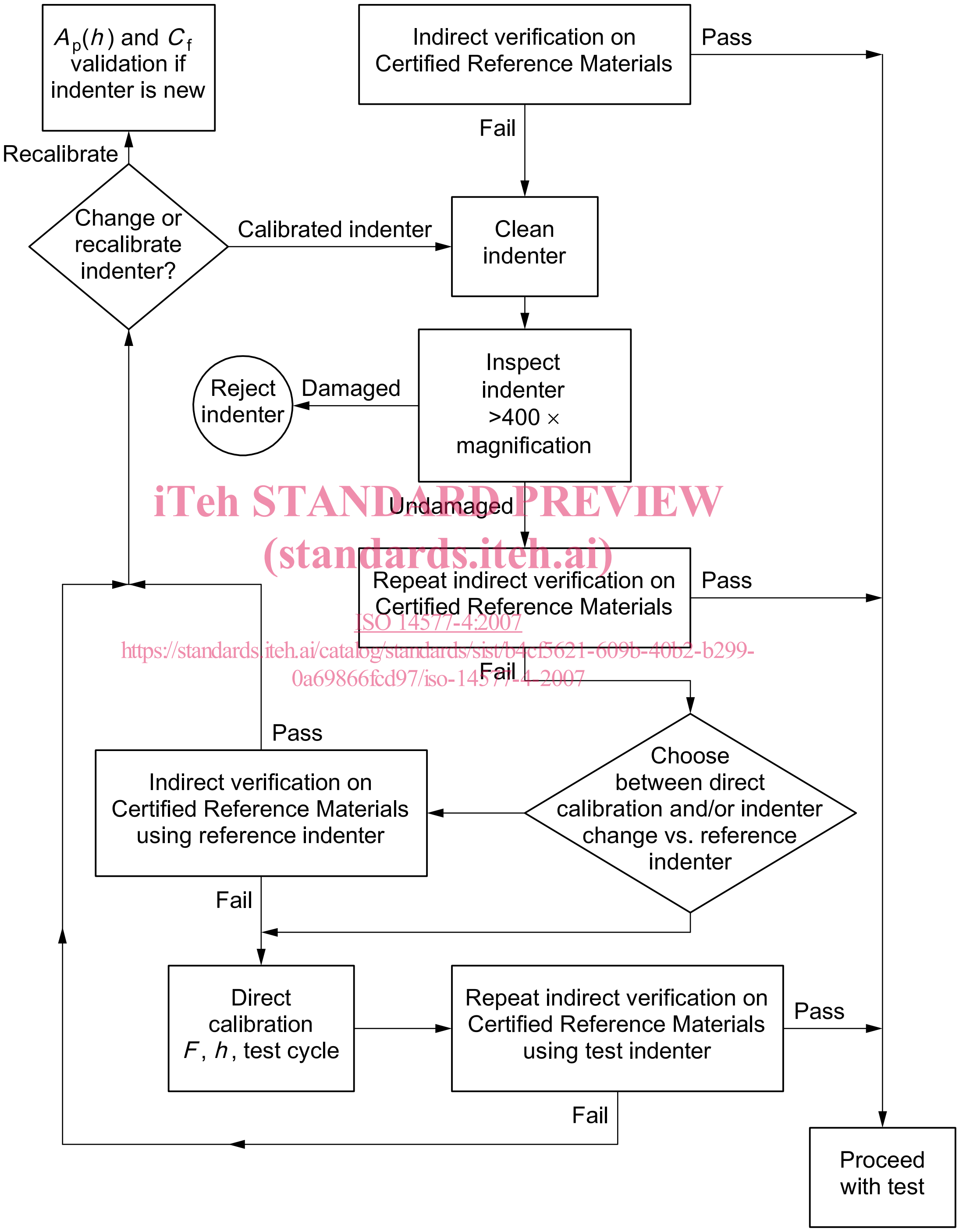
c $E_c^* = E_{IT}^*$ (at $a/t_c$ = 0).



## 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 ± 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.