
**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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ISO copyright office
Ch. de Blandonnet 8 • CP 401
CH-1214 Vernier, Geneva, Switzerland
Tel. +41 22 749 01 11
Fax +41 22 749 09 47
copyright@iso.org
www.iso.org

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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.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

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

For an explanation on the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT) see the following URL: www.iso.org/iso/foreword.html.

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 14577-4:2007), 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

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 a significant influence of the substrate is detected and 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. However, the application of this method of this part of ISO 14577 is not needed if the indentation depth is such a small fraction of the coating thickness 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.

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 and non-metallic coatings are included in the scope of this part of ISO 14577. In this part of ISO 14577, the term coating is used to refer to any solid layer with homogeneous properties different to that of a substrate it is connected to. The method assumes that coating properties are constant with indentation depth. Composite coatings are considered to be homogenous if the structure size is less than the indentation size.

The application of this part of ISO 14577 regarding measurement of indentation 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 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 documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

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

ISO 14577-2:2015, *Metallic materials — Instrumented indentation test for hardness and materials parameters — Part 2: Verification and calibration of testing machines*

ISO/IEC Guide 98-3, *Uncertainty of measurement — Part 3: Guide to the expression of uncertainty in measurement (GUM)*

3 Symbols and designations

ISO 14577-1:2015, Table 1 provides a listing of symbols and their related designations. Additional symbols and designations used in this international standard are included in [Table 1](#).

Table 1 — Symbols and designations

Symbol	Designation	Unit
a	Radius of contact	mm
t_c	Thickness of the coating	mm
E_{ITc}	Indentation modulus of the coating	GPa
E_{ITc}^*	Indentation plane-strain modulus of the coating	GPa
H_{ITc}	Indentation hardness of the coating	GPa

4 Calibration and direct verification of testing machines

The instrument shall be calibrated and directly verified according to the procedures set out in ISO 14577-2:2015, Clause 4.

Indirect verification according to the procedure specified in ISO 14577-2:2015, Clause 5, 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.

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](#).

5.2 Surface roughness

Indentation into rough surfaces will lead to increased scatter in the results with decreasing indentation depth (see ISO 14577-1:2015, Annex E). Clearly, when the roughness value, R_a , 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