INTERNATIONAL STANDARD

ISO 14577-4

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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

Reference number
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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