INTERNATIONAL STANDARD

ISO 16253

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Watch-cases and accessories — Vapour phase deposited coatings

Boîtes de montres et leurs accessoires — Revêtements déposés en phase vapeur

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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 16253 was prepared by Technical Committee ISO/TC 114, *Horology*, Subcommittee SC 6, *Precious metal coverings*.

Introduction

This International Standard has been developed in order to meet the global need for a definition of the requirements and tests that enable characterization of coatings applied by vapour phase deposition to watch-cases and their accessories.

It defines the terms used in connection with the main vapour phase deposition processes, and presents the requirements relating to the appearance and strength of the deposited coatings, as well as their resistance to physical and chemical attack. On the conclusion of an agreement between the customer and the supplier, some latitude is left as to the choice of standard test methods and determination of acceptable limit values.

This International Standard defines a simple structure, which facilitates identification of the base material, the deposited coatings and their thickness, in order to give information to consumers. It advises manufacturers to provide these indications in the documents supplied with the watch.

To protect the consumer, this International Standard stipulates the conditions for marking watch-cases and their accessories, as well as the required compliance with ISO 3160-1, which specifies the thickness and minimum gold content for gold alloy coatings.

Watch-cases and accessories — Vapour phase deposited coatings

1 Scope

This International Standard specifies the general requirements and test methods for hard coatings obtained by vapour phase deposition processes intended to improve mainly the surface aspect and wear and corrosion resistance, and/or to offer protection against contact-allergy. It is applicable to watch-cases and accessories.

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 3160-1, *Watch-cases and accessories — Gold alloy coverings — Part 1: General requirements*

ISO 3160-2, *Watch-cases and accessories — Gold alloy coverings — Part 2: Determination of fineness, thickness, corrosion resistance and adhesion*

ISO 3160-3[1)], *Watch cases and accessories — Gold alloy coverings — Part 3: Abrasion resistance tests of a type of coating on standard gauges*

ISO 4524-5, *Metallic coatings — Test methods for electrodeposited gold and gold alloy coatings — Part 5: Adhesion tests*

ISO 8654, *Colours of gold alloys — Definition, range of colours and designation*

ISO 9227, *Corrosion test in artificial atmospheres — Salt spray tests*

EN 12472, *Method for the simulation of wear and corrosion for the detection of nickel release from coated items*

CR 12471, *Screening test for nickel release from alloys and coatings in items that come into direct and prolonged contact with the skin*

1) Standard cancelled and replaced by ISO 23160 (in preparation).

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

3.1
vapour phase deposited coating
coating that is deposited by the transfer of atoms from a source to the substrate through vapour phase

NOTE The transfer can be purely physical (PVD: physical vapour deposition) or can be done through chemical reaction (CVD: chemical vapour deposition). The gas phase transfer can be used to form chemical compounds like ceramics, by injecting a reactive gas.

3.2
ion plating
coating process in which the substrate and the growing layer are continuously receiving impacts of energetic particles, usually ions from the plasma of a glow discharge, accelerated toward the biased substrate

3.3
sputtering
coating process in which atoms ejected from a target (cathode) surface by the impacts of gaseous ions from the plasma of a glow discharge, are deposited on to the substrate

3.4
hard coating
coating with a hard material such as titanium nitride (TiN) or titanium carbide (TiC) having a hardness value of more than 1 000 HV (Vickers hardness)

4 Requirements

4.1 Coating

4.1.1 Appearance of coating

The appearance of the coating shall be tested according to the method and conditions specified in Annex A. The coating on the significant surface of the coated item shall be free from harmful defects or from any sign of a defect which is detrimental to usage, such as coating peel-off, irregular colour tone, roughness, cracks, pits, exposure of base material and any permanent stain.

4.1.2 Thickness of coating

The thickness of the coating is specified according to agreement between the customer and the supplier. The thickness of the coating shall be tested according to one of the methods given in 5.3 and the coating thickness at the significant surface shall comply with the specified thickness.

4.1.3 Adhesion of coating

The adhesion of the coating shall be tested according to one of the methods given in 5.4 and the coating shall be free from such signs of defective adhesion as peeling or blister.

4.1.4 Corrosion resistance of coating

The corrosion resistance of the coating shall be tested according to one of the methods given in 5.5 and shall comply with the specified criteria, when the corrosion resistance is specified.

4.1.5 Hardness of coating

The hardness of the coating shall be tested according to one of the methods given in 5.6 and shall comply with the specified value, when the hardness of coating is specified.

4.1.6 Wear resistance of coating

The wear resistance of the coating shall be tested according to one of the methods given in 5.7.1 and shall comply with the specified criteria, when the wear resistance is specified.

Considering the very small thickness of the coating, a certain degree of wear of the edges can be considered as acceptable; whereas wear, change of colour or of appearance of the flat or rounded sides (with a large radius), is not acceptable.

Where parts are coated with a thin layer of gold, the wear resistance test can reveal a change in colour, whose criteria for acceptance should be defined between customer and supplier.

4.1.7 Nickel release

Where the base material of the coated parts is likely to release nickel, it is recommended to apply to them the method of simulation of wear and corrosion, according to 5.7.2. The measured values shall comply with the legislation in force in the country where the product is to be sold.

4.1.8 Colour of coating

The colour of the coating shall be measured before and after the wear resistance test, according to the method given in 5.8, and shall comply with the specified value, when the colour is specified.

4.2 Base material, processes and coatings

It is recommended to customers and suppliers to indicate the base material, the processes and the coatings, using Annex B.

4.3 Underlayer

If the substrate requires the deposit of an underlayer or barrier, its characteristics shall be verified before applying a vapour phase deposited coating, according to the existing standards.

5 Test methods

5.1 Test samples

Samples for testing shall be issued from the regular production batch. If it is not possible, a representative sample shall be prepared from the same base material as the product and treated in the same production batch.

5.2 Visual inspection

The visual inspection shall comply with the test method and test conditions specified in Annex A.

5.3 Thickness measurement

The measurement of the coating thickness can be made using one of the methods specified in ISO 3160-2.

In case of dispute, the measurement of a metallographic section examined by means of a scanning electron microscope shall be the reference.

5.4 Adhesion test

The adhesion of the coating shall be tested using either one of the test methods specified in ISO 3160-2 (or ISO 4524-5), or the scratch test specified in Annex C. The method and the test conditions shall be agreed upon between the customer and the supplier.

NOTE As the scratch test can be influenced by the nature of the base material, the thickness and the stresses in the coating, this test is more appropriate for carrying out comparisons of the adhesion between layers made under the same conditions.

5.5 Corrosion resistance test

The corrosion resistance shall be tested using one or several of the corrosion resistance test methods described in ISO 9227 for the neutral salt spray test and in ISO 3160-2 for the synthetic perspiration test and the porosity test. The choice of test method and test conditions shall be agreed upon between the customer and the supplier.

5.6 Hardness test

The hardness test shall be carried out on the surface coating using the Vickers or Knoop method. The load applied to the indenter shall be set so as to produce a depth of indentation in the coating of approximately 10 % of the coating thickness, to avoid any influence of the substrate. As an alternative, the nanoindentation method (i.e. Berkovitch indenter) can be used. The choice of test method and of test conditions shall be made by agreement between the customer and the supplier.

5.7 Wear resistance tests

5.7.1 Wear resistance test in relation with the decorative aspect of the coating

The wear resistance shall be tested, applying the method specified in ISO 3160-3, or a method that both parties, the customer and the supplier, can recognize as significant. The choice of test conditions shall be agreed upon between the customer and the supplier.

5.7.2 Tests for the simulation of wear and corrosion

If the measurement of nickel release is necessary or required, the coated items shall be submitted to the tests specified in EN 12472 for the simulation of two years' wear and corrosion, followed by the measurement of nickel release.

A laboratory approved by the customer and the supplier shall carry out the tests according to EN 12472.

For the supervision of current production of coated items, the test described in the report CR 12471 can be applied after the tests specified in EN 12472.

5.8 Measurement of colour

If a measurement is required, a spectrocolorimeter shall be used to measure the colour of the hard coating and of the decorative gold alloy coating on top of it.

It is recommended that the colour of the gold alloy coating be selected from the standard colours defined in ISO 8654. If not, the colour shall be specified using chromatic coordinates.

6 Identification and marking on the product

6.1 Identification

It is recommended that the base material and the coating(s) be identified in the documents supplied with the product, in order to inform the consumer. The identification system shall be clearly explained to the users. For the marking on watch-cases and accessories, see 6.2.

The identification shall be based on the structure a – b/c d/e f and on the following symbols and abbreviations:

a indicates the type of deposit process;

b indicates the type of base material;

c indicates the type of coating;

d indicates the minimum thickness of the coating, in micrometres;

e indicates the type of additional decorative coating;

f indicates the minimum thickness of the additional decorative coating, in micrometres.

Examples of deposit processes:

— IP: Ion Plating

— SP: Sputtering

— CVD: Chemical Vapour Deposition

Examples of base materials:

— SS: Stainless steel (SUS can be used)

— Bs: Brass

— Ti: Titanium and its alloys

Elemental symbols shall be used to indicate the type of base material when it is a metallic element, and accepted symbols for alloys are used when basic material is an alloy. In the other cases, the chemical symbol of the major element shall be used.

EXAMPLE Information shown on the documents supplied with a watch having a stainless steel (SS) case coated with a minimum thickness of 2 µm of titanium nitride (TiN) deposited by ion plating (IP) and having received in addition a layer of minimum 0,1 µm of gold (Au) by the same process.

— a – b/c d/e f

— IP – SS/TiN 2µm/Au 0,1 µm

6.2 Marking on the product

The marking of the type of coating process, the base material and the hard coating may be implemented on watch-cases and accessories. The letters shall have a minimum height of 0,8 mm for legibility to the naked eye.

The marking shall be based on the same symbols and abbreviations as in 6.1 and shall be restricted to the indications a, b and c.

EXAMPLE Stainless steel (SS) watch-case coated with titanium nitride (TiN) deposited by ion plating (IP) and followed by a gold deposit (Au) of 0,1 µm of gold, using the same process.

— a – b/c

— IP – SS/TiN or: IP – SUS/TiN

The indication of a gold alloy coating implemented on watch-cases and their accessories is only permitted when the product bears a layer of gold alloy meeting the following requirements of ISO 3160-1:

— minimum fineness of the gold alloy: 585/1 000;

— minimum thickness of the gold alloy layer: 5 µm.

Annex A
(normative)

Visual inspection of vapour phase deposited coatings

A.1 General

This annex specifies the test method and the test conditions for the visual inspection of vapour phase deposited coatings on metal and non-metal base material.

The test method and test conditions have been chosen in order to facilitate the detection and the inspection of such coating defects as lustre, brightness, irregular colour tone, roughness, burns, cracks, pits, development of arborization crystals and exposure of base material or primary plating, such signs of defective adhesion as blister and peeling, and additional presence of dirt and scratches.

A.2 Background and light source

The sample shall be placed against a background, whose colour makes a good contrast with the colour of the coated item, and illuminated over the test surface with an intensity of more than 500 lx using white light close to natural light, through frosted glass or similar.

A standard light source D65 with intensity of more than 500 lx shall be used, when arbitration is needed.

A.3 Procedure

For visual inspection, the sample shall be placed and held under normal service conditions, at the distance of distinct vision, usually 30 cm, under the conditions mentioned in A.2.

A.4 Evaluation method

Judgment of the specified quality shall be made by comparison, with the help of limit samples, showing the acceptable and non-acceptable defects by agreement between the customer and the supplier. A magnifying glass with a magnifying capability of more than $\times 4$ or less shall be used when visual judgment proves too difficult to the naked eye.

Annex B
(informative)

Base material, process and coatings

Table B.1 — Identification of the base material, of the coating process and of the layers

Parameter		Specification	Remarks
Part name		Watch-case	
Part number		****-**	
Base material			
	Material	Brass	
	Surface finish	Mirror finishing	
Process for under-layer		Galvanic plating	
	Coating material	PdNi	
	Layer thickness	3 µm	
Process for layers		Ion plating	
1st layer	Coating material	TiN	Hard coating Hardness $\geqslant$ 1 000 HV
	Layer thickness	$\geqslant$ 2 µm	
2nd layer	Coating material	Au	For decoration
	Layer thickness	$\geqslant$ 0,1 µm	

Annex C
(normative)

Evaluation of adhesion strength using the scratch test

C.1 Principle of measurement

The adhesion strength of the coating is evaluated by finding the critical load, L_c, which is capable of detaching the coating from the substrate, using a diamond tipped indenter.

C.2 Tester and test procedure

The scratch adhesion tester shall have a mechanism to scratch the surface of the coated sample by applying a load over a diamond tipped indenter. The diamond tip of the indenter shall have an angle of 120° and a tip radius of 0,2 mm, or a shape as agreed between the customer and the supplier.

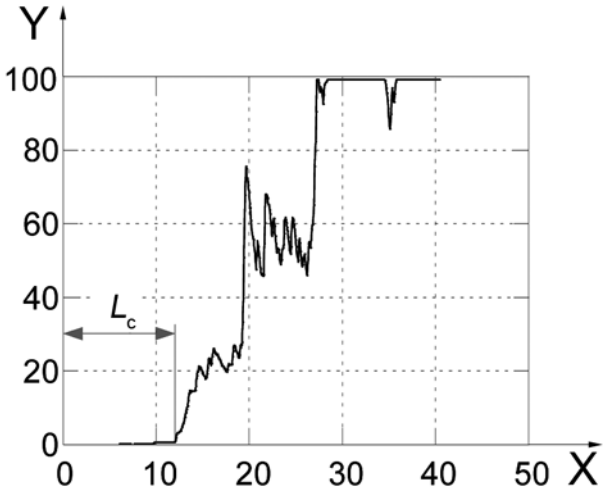
During the test, the diamond tipped indenter is drawn over the coated surface under a continuously increasing load (progressive loading mode). When the applied normal load is strong enough to create induced stresses between coating and substrate, reaching the value of the adhesion forces, the rupture of adhesion of the coating takes place. The load that corresponds to the failure is known as the critical load, L_c.

The test sample shall be mounted and fastened in order to present a horizontal test surface. The scratch force applied shall not cause the test sample to shift.

The indenter shall be placed vertically with respect to the test surface and the load shall be applied progressively on the indenter in order to obtain the desired scratch effect.

C.3 Evaluation method

The scratch adhesion tester is equipped with an acoustic emission signal detector and/or a piezo-electrical transducer, to monitor the frictional force. The output from the acoustic emission detector and the frictional force-measuring device are recorded to identify the critical load, L_c (see Figure C.1). The adhesion of the coating is evaluated by measuring the load applied, when peeling or chipping occurs, or by observing the test sample with an optical microscope or a scanning electron microscope.



Key

X vertical load in newtons

Y acoustic emission signal

Figure C.1 — Acoustic emission signal function of vertical load

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