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Metallic and other inorganic coatings — Measurement of mass per unit area — Review of gravimetric and chemical analysis methods

Revêtements métalliques et autres revêtements inorganiques — Mesurage de la masse surfacique — Présentation des méthodes d'analyse gravimétrique et chimique

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Foreword

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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).

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For an explanation of the voluntary nature of standards, 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 www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 107, *Metallic and other inorganic coatings*.

This second edition cancels and replaces the first edition (ISO 10111:2000), which has been technically revised. The following changes have been made:

a) a gravimetric method has been added for weighing the uncoated substrate and the finished sample;

b) the surface area increase caused by surface roughness has been considered to obtain a more realistic estimation of local geometric coating thickness (optional);

c) Annex A, which gives reagents for etching or stripping solutions, has been changed to informative as other solutions can be applied;

d) reagents in Annex A that referred to no longer existing standards or which contain hazardous chemicals have been removed;

e) outdated and uncited references in the Bibliography have been removed.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Metallic and other inorganic coatings — Measurement of mass per unit area — Review of gravimetric and chemical analysis methods

WARNING — The use of this document can involve hazardous materials, operations and equipment. It does not purport to address all of the safety or environmental problems associated with its use. It is the responsibility of users of this document to take appropriate measures to ensure the safety and health of personnel and the environment prior to application of this document.

1 Scope

This document gives guidelines for determining the average surface density over a measured area of anodic oxide or of a coating deposited autocatalytically, mechanically, by chemical conversion, by electrodeposition, by hot dip galvanizing and by chemical or physical vapour deposition using gravimetric and other chemical analysis procedures that have attained some degree of national or international standardization.

A variety of procedures are described and include:

— gravimetric procedures for chemical or electrochemical dissolution of the coating or the substrate to determine the coating surface density;

— gravimetric procedures for weighing the uncoated substrate and the coated (finished) specimen to determine the coating surface density;

— analytical procedures that utilize dissolution of the coating for determination of the coating surface density by instrumental chemical analysis methods.

With the exception of the gravimetric method as described in ISO 3892, this document does not give the measurement uncertainties of the methods cited.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements 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 2080, *Metallic and other inorganic coatings — Surface treatment, metallic and other inorganic coatings — Vocabulary*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 2080 apply.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at http://www.electropedia.org/

4 Principle

The mass of a coating over a measured area is determined by

a) weighing the test specimen before and after dissolving the coating in a reagent or electrolyte that does not attack the substrate, or

b) weighing the coating after dissolving the substrate in a reagent that does not attack the coating, or

c) dissolving both the coating and the substrate, or the coating alone, and quantitatively analysing the resulting solution, or

d) weighing the test specimen before and after the coating process, provided that the mass of the substrate material removed during those pre-treatment steps, after which a weighing would negatively affect the coating deposition steps, is negligible compared to the mass of the coating.

The surface density of the coating is calculated from the mass and area measurements. Its thickness is based on the mass, area and density of the coating material.

5 Special equipment

Certain specialized chemical, electrochemical and chemical analysis equipment is required for some of the specific methods referred to in Table A.1 (see Clauses 8 and 9).

6 Preparation of test specimen

6.1 Size

The specimen should be large enough to permit area and mass measurement of adequate accuracy (see Clauses 8 and 9).

6.2 Shape

The shape of the test specimen should be such that the surface area can be readily measured without difficulty. A rectangular or circular test specimen is usually suitable.

6.3 Edge condition

If the area to be measured is small and has to be known accurately, the edges may need to be dressed to remove smeared coating, to remove loose burrs and to provide well-defined and (for rectangles) straight edges. This should be considered for areas less than 100 mm^2.

One method of dressing the edges of a rectangular specimen involves clamping the specimen between two plastic or metal blocks with the edge of the specimen flush with the edges of the blocks and then grinding and polishing the edges metallographically.

6.4 Heat treatment

If the substrate has to be dissolved in such a way as to leave the coating intact, it may be desirable to first treat the test specimen so that the coating will not curl up tightly or fall apart. Some gold deposits of 1,5 mg/cm^2 (< 0,9 µm) will fall apart when their substrates are dissolved, but will support themselves after heat treatment at 120 °C for 3 h. If the thickness of a coating (instead of its surface density) is being determined, a heat treatment that could change the density of the coating material should not be used.

7 Measurement of coated area

7.1 Measurement method

Since the accuracy of the area measurement should be greater than the desired accuracy of the surface density measurement, the method of measuring the area depends on the desired accuracy and the specimen size.

7.2 Surface measuring equipment

7.2.1 Geometrical (projected) surface area

The area can be measured with a planimeter, but it is usually determined by linear measurements. Often a micrometre or vernier calliper is used. For large areas, however, a ruler may do.

For maximum accuracy, a measuring microscope should be used.

It may be difficult to measure directly the area of threaded articles with sufficient accuracy, in which case the area should be determined from drawings or published tables.

7.2.2 Surface area increase due to roughness (optional)

Surface roughness leads to an increase of the true surface area compared to the geometrical (projected) surface area as determined by the methods described in 7.2.1.

Using atomic force microscopy (AFM), confocal microscopy or interference microscopy and a suitable computer software, the relative surface area increase (RSAI) can be determined (at least for a small fraction of the total surface).

Enhance the geometrical (projected) surface by the RSAI to estimate the true surface area. Calculate the true surface density of the coating or the true coating thickness using this true surface area in order to obtain a better agreement with geometrical coating thickness measurements in cross-section.

The RSAI should be determined on the uncoated substrate before any coating is applied as some coatings tend to level surface roughness, but preferably after pre-treatment steps, which increase surface roughness, e.g. to improve adhesion.

7.3 Number of measurements

Because circular or rectangular specimens will not be perfectly circular or rectangular, each dimension should be measured in three places. For a rectangle, the length of each edge and the length and width through the centre should be measured and an average obtained for each dimension.

NOTE In the case of a cylinder, one would normally measure the diameter and length. In specifications for metallic coated wire (fencing) that has been electroplated or coated by other processes, the length of the wire specimen is not measured, but is, in effect, calculated from the mass (which is measured anyway), the radius and the density of the substrate material as follows:

$$l = \frac{m}{\pi r^2 \rho_s}$$

where

l is the length;

m is the mass;

r is the radius;

ρ_s is the density of the substrate.

8 Determination of mass of coating by chemical analysis

8.1 General

The chemical analysis method is very general. Both coating and substrate or the coating alone are dissolved in a suitable reagent (see Annex A for examples) and then the resulting solution is analysed for the coating material. For each coating-substrate-reagent combination, there may be several analytical methods, e.g. photometric or volumetric methods, atom absorption spectroscopy or inductively coupled plasma with optical emission or mass spectrometry.

8.2 Restrictions

The chemical analysis method cannot be used when the coating material cannot be completely removed from the substrate material by chemical means or when there is a constituent common to both that is not readily separated (e.g. nickel phosphorus alloy on nickel).

9 Gravimetric determination of mass of coating

9.1 Specimen size

Since the measurement uncertainty of the mass measurement should be less than the desired measurement uncertainty of the surface density measurement, the test specimen should be large enough for the coating to be weighed with the desired accuracy (see 9.2).

9.2 Limitations

In principle, the gravimetric procedures can be used to measure very thin coatings or to measure coatings over small areas, but not thin coatings over small areas. The limits depend on the required accuracy, e.g. 2,5 mg/cm^2 of coating might require 1 cm^2, but 0,1 mg/cm^2 of coating would require 25 cm^2 to obtain 2,5 mg of coating.

The gravimetric method does not indicate the presence of bare spots or sites with thicknesses lower than the specified minimum in the measuring areas. In addition, the single value obtained from each measuring area is the mean thickness of that area.

The measurement uncertainty of the gravimetric method is normally less than 5 % over a wide range of thicknesses (see ISO 3892).

9.3 Restrictions

The gravimetric procedures can be used for many coating–substrate combinations. Except for the procedure described in 9.5.4, they cannot be used where neither the coating nor substrate material can be completely removed, one from the other by chemical or physical means.

9.4 Gravimetric analysis equipment

A balance is required for gravimetric analysis, but the required sensitivity of the balance depends on the size of the test specimen, the coating thickness (coating mass) and the required accuracy of the measurement. The analytical balance should be capable of weighing to an accuracy of 0,1 mg for weighing the test pieces under examination before and after dissolution of the coatings or before and after the coating process, respectively.

For anodic and cathodic dissolution, a (constant) direct current source is necessary.

9.5 Procedure

9.5.1 General

The mass of coating may be determined by

a) weighing the test specimen before and after dissolving the coating (see Annex A for exemplary reagents) and taking the difference (see 9.5.2), or

b) dissolving the substrate (see Annex A for exemplary reagents) and weighing the coating directly (see 9.5.3), or

c) weighing the test specimen before and after deposition of the coating and taking the difference (see 9.5.4).

The first time a gravimetric method is used, it should be evaluated in accordance with 9.5.2.2 and 9.5.3.2.

9.5.2 Difference method with dissolution of the coating

9.5.2.1 First, clean the test specimen of any foreign material, then rinse it with alcohol (methanol, ethanol, isopropanol) or another suitable solvent, blow it dry with clean air and weigh it. Immerse the specimen in the appropriate reagent (see Annex A for examples) to dissolve the coating either chemically or electrochemically, rinse it with water, then with alcohol, blow it dry with clean air and weigh it again. The loss of mass is the mass of the coating.

9.5.2.2 To determine if any dissolution of the substrate has occurred, repeat the process with the stripped substrate, making sure that the substrate is immersed in the reagent for the same length of time as before. Any loss of mass enables one to make a judgement of a possible error due to any dissolution of the substrate with the coating during the stripping process.

9.5.3 Direct weighing method with dissolution of the substrate

9.5.3.1 Dissolve the substrate in the appropriate reagent (see Annex A for examples). Rinse the coating with water and then alcohol (methanol, ethanol, isopropanol) or another suitable solvent, blow it dry with clean air and weigh it.

9.5.3.2 To determine if any dissolution of the coating occurred, submit the isolated coating to the same stripping process, making sure that the coating is immersed in the stripping reagent for the same length of time as it was during the stripping process. Any loss of mass enables one to make a judgement of a possible error due to any dissolution of the coating with the substrate during the stripping process.

9.5.4 Difference method without dissolution

First, clean the uncoated test specimen of any foreign material, rinse it with alcohol or another suitable solvent, which does not attack the substrate material nor disturb the subsequent coating process, blow it dry with clean air and weigh it.

Then subject the uncoated test specimen to the coating process.

Afterwards, clean the coated test specimen of any foreign material, rinse it with alcohol or another suitable solvent, which attacks neither the coating nor uncovered zones of the substrate, blow it dry with clean air and weigh it.

The gain of mass is the mass of the coating.

As many of the required pre-treatment steps for the substrate material as possible should be done before the first weighing, especially when they remove substrate material. However, pre-treatment