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Anodizing of aluminium and its alloys — Determination of mass per unit area (surface density) of anodic oxidation coatings — Gravimetric method

Anodisation de l'aluminium et de ses alliages — Détermination de la masse par unité de surface (masse surfacique) des couches d'oxydation anodique — Méthode gravimétrique

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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).

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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 79, *Light metals and their alloys*, Subcommittee SC 2, *Organic and anodic oxidation coatings on aluminium*.

This fourth edition cancels and replaces the third edition (ISO 2106:2011), which has been technically revised. The main changes compared with the previous edition are as follows:

— phosphoric acid/sodium molybdate solution has been added as a test solution;

— the information of the test specimen has been added;

— Formula (2) has been corrected.

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.

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Anodizing of aluminium and its alloys — Determination of mass per unit area (surface density) of anodic oxidation coatings — Gravimetric method

1 Scope

This document specifies a gravimetric method for determining the mass per unit area (surface density) of anodic oxidation coatings on aluminium and its alloys.

The method is applicable to all oxidation coatings formed by anodizing aluminium and its alloys, either cast or wrought, and is suitable for most aluminium alloys, except those in which the mass fraction of copper is greater than 6 %.

NOTE 1 A high content of copper in the alloy can lead to increased dissolution of the substrate aluminium.

NOTE 2 If the thickness is known with sufficient precision (for example, using the method specified in ISO 2128), the determination of the mass per unit area (surface density) of the coatings will enable its apparent density to be calculated. Conversely, if the conditions of application of the coating and its density are known, the determination of its mass per unit area (surface density) can permit the calculation of the average mass and an approximate evaluation of the thickness (see Clause 9).

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 7583, *Anodizing of aluminium and its alloys — Terms and definitions*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 7583 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 anodic oxidation coating on a weighed test specimen of known surface area is dissolved without significantly attacking the substrate aluminium, using a solution of phosphoric acid and chromium(VI) oxide or phosphoric acid and sodium molybdate of specified concentration.

After dissolution of the coating, the test specimen is reweighed in order to calculate the loss in mass. The mass loss is related to the unit area covered by the coating, and is expressed in milligrams of coating per square decimetre of surface.

NOTE This is a destructive test.

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5 Reagents

Use only reagents of recognized analytical grade and only distilled water or deionized water.

Test solution A does not attack the substrate aluminium and it is not necessary to take uncoated surfaces into account.

Test solution B attacks the substrate aluminium to a limited extent. Therefore, it is necessary to prevent to dissolve.

5.1 Test solution A.

Phosphoric acid/chromic solution, prepared as follows:

— phosphoric acid, (ρ_{20} = 1,7 g/ml): 35 ml;

— chromium(VI) oxide: 20 g;

— water: make up to 1 000 ml.

WARNING — Chromium(VI) is toxic and shall be handled properly. Chromium (VI) solutions are hazardous to the environment and severely hazardous to waters.

5.2 Test solution B.

Phosphoric acid/sodium molybdate solution, prepared as follows:

— phosphoric acid, (ρ_{20} = 1,7 g/ml): 35 ml;

— disodium molybdate(VI) dihydrate: 10 g;

— water: make up to 1 000 ml.

WARNING — Molybdate(VI) is toxic and shall be handled properly. In the enforcement of this test, it is necessary to have sufficient indoor ventilation.

6 Apparatus

Usual laboratory apparatus and glassware and, in particular, the following.

6.1 Laboratory balance, with a resolution of 0,1 mg.

7 Preparation of test specimen

7.1 Sampling

The test specimen shall be taken from a significant surface of the product, where the anodic oxidation coating formed thereon satisfies the quality requirements for the application of the product. The test specimen shall not be taken from an edge of the part because of possible distortion and/or non-uniformity.

Where it is impossible to test the product itself, a test specimen that is representative of the product may be used. In this case, the test specimen used shall be made from the same material and prepared under the same conditions of finishing as those used for the preparation of the product. The aluminium alloy, the manufacturing conditions (kind and temper of the material), and the surface condition before treatment should be the same as those of the product. Pretreatment and anodizing should be performed in the same bath and under the same conditions as the treatment of the product.

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7.2 Size

The standard size of the test specimen should be 50 mm in length and 50 mm in width. Where it is impossible to take a test specimen of the standard size, the surface of the test specimen to be tested shall have an area of between 0,08 dm^2 and about 1 dm^2, and the mass of the test specimen shall not exceed 100 g.

7.3 Method of degreasing

If the surface is dirty or impregnated with oil, grease or similar material, this shall be removed with the aid of a suitable organic solvent in accordance with A.1. Dry the test specimen thoroughly in accordance with A.2.

8 Procedure

8.1 Method using test solution A

8.1.1 Treatment before test

The test shall be applied to one or more significant surfaces of the test specimen. The anodic oxidation coatings on the other surfaces shall be removed by a mechanical or chemical process, leaving intact the surface(s) to be tested. Alternatively, a protective agent, resistant to attack by the acidic test solution, shall be applied to the surfaces of the test specimen that are not to be tested.

Measure the area of the surface covered by an anodic oxidation coating. Weigh the test specimen to the nearest 0,1 mg.

8.1.2 Performance of the test

Immerse the test specimen in test solution A (5.1) for 10 min at 95 °C to 100 °C with efficient agitation.

Rinse the test specimen in water, dry in accordance with A.2 and reweigh it. Repeat the immersion, drying and weighing until no further loss in mass is observed.

NOTE The freshly made reagent will normally allow complete dissolution of the coating within 10 min. Its dissolving power diminishes with use; in general, 1 litre of solution is capable of dissolving 12 g of coating before the diminution becomes noticeable.

On coloured samples, smut residue can occur after the dissolution. This should be removed before weighing. Immersion in 28 % to 30 % nitric acid or wiping with gauze when rinsing can remove the smut.

8.2 Method using test solution B

8.2.1 Treatment before test

The test shall be applied to one or more significant surfaces of the test specimen. The anodic oxidation coatings on the other surfaces shall be removed by a mechanical or chemical process, leaving intact the surface(s) to be tested. Alternatively, a protective agent, resistant to attack by the acidic test solution, shall be applied to the surfaces of the test specimen that are not to be tested.

Measure the area of the surface covered by an anodic oxidation coating. Weigh the test specimen to the nearest 0,1 mg.

8.2.2 Performance of the test

Immerse the test specimen in test solution B (5.2) for 10 min at 95 °C to 100 °C with efficient agitation.

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After immersion for 10 min, the test specimen shall be pulled out from the test solution and observed. If the coating remains clearly, it should continue to be immersed without rinsing, drying and weighing until no coating is observed.

Rinse the test specimen in water, dry in accordance with A.2 and reweigh it. Repeat the immersion, drying and weighing at appropriate intervals. Table 1 provides details of the suggested ending of the coating's dissolution, at which point no further immersion is necessary.

Table 1 — Suggested ending of the coating's dissolution

Estimated total mass loss (mg/dm^2)	Mass loss by the last 10 min immersion
< 400	< 20 mg/dm^2
≥ 400	< 5 % of the estimated total mass loss (mg/dm^2)

The freshly prepared test solution can be used repeatedly. However, its dissolving power diminishes with use. In general, 1 litre of solution is capable of dissolving 3 g of coating. When exceeding this value, renew the test solution.

The dissolution of the coating can cause sedimentation in the test solution. Bumping and splashing can occur because of the sedimentation, in which case a renewal of the test solution is necessary.

On coloured samples, smut residue can occur after the dissolution. This should be removed before weighing. Immersion in 28 % to 30 % nitric acid or wiping with gauze when rinsing can remove the smut.

9 Expression of results

Calculate the mass per unit area of surface (surface density) of the coating, ρ_A, in milligrams per square decimetre, using Formula (1):

$$\rho_A = \frac{m_1 - m_2}{A} \tag{1}$$

where

ρ_A is the mass per unit area of surface (surface density) of the coating, in milligrams per square decimetre;

m_1 is the mass of the test specimen before dissolution of the coating, in milligrams;

m_2 is the mass of the test specimen after dissolution of the coating, in milligrams;

A is the area covered by the coating of which the mass is measured (without taking into account edges or other uncoated parts), in square decimetres.

Where required, the average thickness of the coating, δ, in micrometres, can be estimated using Formula (2):

$$\delta = \frac{\rho_A}{\rho \times 10} \tag{2}$$

where

δ is the average thickness of the coating, in micrometres;

ρ_A is the mass per unit area (surface density) of the coating, in milligrams per square decimetre;

ρ is the density of the coating, in grams per cubic centimetre.

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The density of the coating depends on the specific alloy, and the anodizing and the sealing process. It can be significantly reduced by long anodizing times. This density can vary considerably from about 1,5 g/cm^3 to over 3 g/cm^3.

For thin oxidation coatings on aluminium and its alloys with no more than a copper mass fraction of 0,3 %, produced under direct current in sulfuric acid solution, at a temperature of approximately 20 °C, the density may be assumed, by convention, to be equal to 2,6 g/cm^3 for sealed coatings and 2,4 g/cm^3 for unsealed coatings. However, the method gives only an approximate value of the thickness because there is considerable uncertainty about the density value.

The estimation of thickness is more accurate for thin coatings (10 µm and less).

10 Test report

The test report shall include at least the following information:

a) a reference to this document, i.e. ISO 2106:2019;

b) the type and identification of the product tested;

c) whether test solution A or B was used;

d) the result of the test (see Clause 9);

e) anything unusual noticed during the determination;

f) any operations not included in the procedure described in this document, or considered to be optional;

g) any unusual features observed;

h) the date of the test.

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