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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 anodiques — Méthode gravimétrique

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Foreword

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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 2106 was prepared by Technical Committee ISO/TC 79, *Light metals and their alloys*, Subcommittee SC 2, *Organic and anodic oxidation coatings on aluminium*.

This third edition cancels and replaces the second edition (ISO 2106:1982), which has been technically revised. It also incorporates the Amendment ISO 2106:1982/Amd.1:1983.

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

1 Scope

This International Standard 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 copper content is greater than 6 %.

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

NOTE 2 If the thickness is known with sufficient precision (for example, using the method specified in ISO 2128), 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 calculation of the average mass and an approximate evaluation of the thickness (see the Note in Clause 6).

2 Principle

The anodic oxidation coating on a weighed test piece of known surface area is dissolved without significantly attacking the basis metal, using a mixture of phosphoric acid and chromium(VI) of specified concentration.

After dissolution of the coating, the test piece is reweighed, and the loss in mass is calculated. 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 .

3 Reagent

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

3.1 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.

4 Apparatus

Usual laboratory apparatus and glassware, together with the following.

4.1 Laboratory balance, capable of weighing to an accuracy of 0,1 mg.

5 Procedure

5.1 Preparation of test piece

The surface of the test piece to be tested shall have an area of between 0,08 dm^2 and 1 dm^2, and the mass of the test piece shall not exceed 100 g. 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 (see Annex A).

To measure the mass of the coating on one surface only of the test piece, the coating on the other surface shall be removed by a mechanical or chemical process, leaving the significant surface intact. Alternatively, a protective agent, resistant to attack by the acidic test solution, shall be applied on the reverse surface of the test piece.

5.2 Performance of the test

Calculate the area of the surface covered by an anodic oxidation coating, weigh the test piece to the nearest 0,1 mg and immerse it in the phosphoric acid/chromic solution (see 3.1) for 10 min, at 95 °C to 100 °C, with efficient stirring. Rinse the test piece in water, dry and reweigh it (see Annex A). 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 l of solution is capable of dissolving 12 g of coating before the diminution becomes noticeable.

6 Expression of results

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

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

where

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

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

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

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

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

where

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

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

The density of the coating depends on the specific alloy, the anodizing and the sealing process. This density may vary, in normal processing conditions, between 2,3 g/cm^3 and 3 g/cm^3.

For thin oxidation coatings on aluminium and its alloys without copper, produced under direct current in sulfuric acid solution, at a temperature of approximately 20 °C, the density can be assumed to be equal to 2,6 g/cm^3 for sealed coatings and 2,4 g/cm^3 for unsealed coatings.

The method gives only an approximate value of the thickness because of uncertainty about the density value.

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

7 Test report

The test report shall include at least the following information:

a) a reference to this International Standard;

b) the type and identification of the product tested;

c) the result of the test (see Clause 6);

d) anything unusual noticed during the determination;

e) any operations not included in the procedure described in this International Standard, or considered to be optional;

f) the date of the test.

Annex A
(informative)

Recommended method for the drying of samples

A.1 Degrease the test piece by gentle agitation for 30 s in a suitable organic solvent at room temperature, remove, leave for 5 min in the ambient atmosphere (pre-drying), place in a drying oven preheated to 60 °C and leave for 15 min with the coated surface standing upright.

WARNING — Where organic solvents are used, carry out the degreasing operation and the pre-drying in a well-ventilated area to minimize exposure to solvent vapour.

A.2 Allow the test piece to cool for 30 min over silica gel in a closed desiccator.

A.3 After the acid treatment (see 5.2), repeat operations A.1 and A.2, omitting the treatment in the organic solvent.

Bibliography

[1] ISO 2128, *Anodizing of aluminium and its alloys — Determination of thickness of anodic oxidation coatings — Non-destructive measurement by split-beam microscope*