SLOVENSKI STANDARD
oSIST prEN ISO 2106:2019

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Anodizacija aluminija in aluminijevih zlitin - Ugotavljanje mase na enoto površine (površinska gostota) anodno oksidiranih prevlek - Gravimetrijska metoda (ISO/DIS 2106:2019)

Anodizing of aluminium and its alloys - Determination of mass per unit area (surface density) of anodic oxidation coatings - Gravimetric method (ISO/DIS 2106:2019)

Anodisieren von Aluminium und Aluminiumlegierungen - Bestimmung der Masse je Flächeneinheit (flächenbezogene Masse) von anodisch erzeugten Oxidschichten - Gravimetrisches Verfahren (ISO/DIS 2106:2019)

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 (ISO/DIS 2106:2019)

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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 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 on 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 the following URL: 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 to 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.

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 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, (1)), 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 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 http://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 basis metal, 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, 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.

5 Reagents

5.1 General

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

Test solution A does not attack bare metal of aluminium and it is not necessary to take uncoated surfaces into account.

Test solution B attacks bare metal of aluminium to a limited extent. Therefore it is necessary to prevent to dissolve.

5.2 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.

5.3 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.

6 Apparatus

Usual laboratory apparatus and glassware, together with laboratory balance with a readability 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 which 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.

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 Annex 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

Both surface of test specimen should have significant surface. If one surface have non-significant surface, the coating on its 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 non-significant surface of the test specimen.

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 test specimen in the test solution A (see 5.2) for 10 min, at 95 °C to 100 °C, with efficient stirring.

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, 1L of solution is capable of dissolving 12 g of coating before the diminution becomes noticeable.

8.2 Method using test solution B

8.2.1 Treatment before test

Both surface of test specimen should have significant surface. If one surface have non-significant surface, a protective agent, resistant to attack by the acidic test solution, shall be applied on its surface of the test specimen.

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

8.2.2 Performance of the test

Immerse the test specimen in the test solution B (see 5.3) for 10 min, at 95 °C to 100 °C, with efficient stirring.

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

Rinse the test specimen in water, dry in accordance with Annex A.2 and reweigh it. Repeat the immersion, drying and weighing at appropriate intervals until the mass loss becomes stable.

NOTE 1 The freshly test solution should be used every examination. The test solution can be used repeatedly. Its dissolving power diminishes with use; in general, 1L of solution is capable of dissolving 3 g of coating before the diminution becomes noticeable.

NOTE 2 When the base metal of aluminium dissolves in the test solution, its sedimentation may occur. In such case, its test solution must not be used to prevent the bumping of the solution by heating at approximately 100 °C. Reducing the number of rinsing and drying of the test specimen will shorten the dissolution time of the anodic oxidation coating and will prevent the sedimentation which cause bumping of the test solution.

NOTE 3 In the test solution B, base metal dissolution can be observed depending on a kind of alloy. Therefore, if the mass loss after immersion is 20 mg/dm^2 or less (5%), it is regarded as there is no mass change.

NOTE 4 Smut residue can occur on coloured samples after the dissolution. This should be removed by wiping before weighing.

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):

$$\rho_A = \frac{m_1 - m_2}{A} \quad (1)$$

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

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

$$\delta = \frac{\rho_A}{\rho \times 10} \quad (2)$$

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.

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 0,3 % copper, 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.

