INTERNATIONAL STANDARD

ISO 3210

Third edition
2010-07-01

Anodizing of aluminium and its alloys — Assessment of quality of sealed anodic oxidation coatings by measurement of the loss of mass after immersion in phosphoric acid/chromic acid solution

Anodisation de l'aluminium et de ses alliages — Évaluation de la qualité des couches anodiques colmatées par mesurage de la perte de masse après immersion en solution phosphochromique

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ISO 3210:2010
https://standards.iteh.ai/catalog/standards/sist/adef2cb5-6be4-4841-b077-5b754718efde/iso-3210-2010

Reference number
ISO 3210:2010(E)

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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 3210 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 3210:1983), which has been technically revised.

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Anodizing of aluminium and its alloys — Assessment of quality of sealed anodic oxidation coatings by measurement of the loss of mass after immersion in phosphoric acid/chromic acid solution

1 Scope

This International Standard specifies methods of assessing the quality of sealed anodic oxidation coatings on aluminium and its alloys by measurement of the loss of mass after immersion in phosphoric acid/chromic acid solution.

This International Standard consists of the following two methods.

— Method 1: assessment of quality of sealed anodic oxidation coatings by measurement of the loss of mass after immersion in phosphoric acid/chromic acid solution, without prior acid treatment.

— Method 2: assessment of quality of sealed anodic oxidation coatings by measurement of the loss of mass after immersion in phosphoric acid/chromic acid solution with prior acid treatment.

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Method 1 is applicable to anodic oxidation coatings intended for decorative or protective purposes or where resistance to staining is important.

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Method 2 is applicable to anodic oxidation coatings intended for architectural purposes. For less severe applications, Method 1 may be more suitable.

The methods are not applicable to the following:

— hard-type anodic oxidation coatings which normally are not sealed;

— anodic oxidation coatings that have been sealed only in dichromate solutions;

— anodic oxidation coatings produced in chromic acid solutions;

— anodic oxidation coatings that have undergone a treatment to render them hydrophobic.

NOTE The methods are destructive and can serve as reference methods in case of doubt or dispute regarding the results of the test for loss of absorptive power (see ISO 2143[1]), or the measurement of admittance (ISO 2931[2]).

2 Principle

An unsealed anodic oxidation coating on aluminium is dissolved rapidly by acid media, whereas a well-sealed coating will withstand long immersion without appreciable attack.

3 Reagents

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

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3.1 Predip solution, used only for Method 2.

Aqueous solution containing (470 ± 15) g/l nitric acid.

NOTE This solution can be obtained, for example, by diluting a 65 % nitric acid solution (ρ_{20} = 1,40 g/ml) with an equal volume of water.

3.2 Test solution.

Aqueous solution containing per litre, 35 ml of phosphoric acid (ρ_{20} = 1,7 g/ml) and 20 g of chromium(VI) oxide.

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 Preparation of test piece

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Cut a piece from the material to be tested, avoiding contact areas, such that there is an area of approximately 1 dm^2, but not less than 0,5 dm^2, of significant surface area. Normally, the mass of the test piece should not exceed 200 g.

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For hollow extrusions, take the test piece from the end of the sections where the total surface area has an anodic oxidation coating (due to the throwing power of the anodizing electrolyte).

In special cases, such as certain types of jigging, small hollow sections, etc., it is necessary to remove the anodic oxidation coating from the inside surface and to carry out the test on the coating on the outer surface of the extrusion.

6 Procedure

6.1 Method 1

6.1.1 Measure the total coated area of the test piece (excluding cut edges and other uncoated surfaces).

NOTE The test solution does not attack bare metal and it is not necessary to take uncoated surfaces into account.

Remove any surface bloom from the test piece by rubbing with a dry cloth.

6.1.2 Degrease the test piece in an organic solvent, e.g. acetone or ethanol (96 %), at room temperature according to the method specified in A.1.

6.1.3 Dry the test piece thoroughly in accordance with A.1 and A.2 and weigh immediately to the nearest 0,1 mg (mass m_1).

6.1.4 Immerse the test piece completely, standing upright, in the test solution (see 3.2) and leave it for exactly 15 min at a constant temperature of 38 °C ± 1 °C.

Uniformity of temperature within the test solution is very important; this can be achieved by using a water-bath and stirring continuously.

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Do not use the test solution after more than 4,5 g of anodic oxidation coating have been dissolved per litre of solution.

Do not use the test solution which has been in contact with materials other than anodized aluminium or its alloys.

6.1.5 Take the test piece from the test solution and rinse thoroughly, first under running water and then in distilled water or deionized water. Dry the test piece as specified in Annex A and weigh immediately to the nearest 0,1 mg (mass m_2).

6.1.6 During the operations described in 6.1.2 to 6.1.5, avoid touching the test piece with bare hands.

Take extreme care that the two drying operations required in 6.1.3 and 6.1.5 are carried out in the same reproducible way and avoid heating to temperatures above 60 °C.

6.2 Method 2

6.2.1 Measure the total coated area of the test piece (excluding cut edges and other uncoated surfaces).

NOTE The predip and test solution do not attack bare metal and it is not necessary to take uncoated surfaces into account.

6.2.2 Degrease the test piece in an organic solvent, e.g. acetone or ethanol (96 %), at room temperature according to the method specified in A.1.

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6.2.3 Dry the test piece thoroughly in accordance with A.1 and A.2, and weigh it immediately to the nearest 0,1 mg (mass m_1).

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6.2.4 Immerse the test piece completely, standing upright, in the predip solution (see 3.1) and leave for 10 min at a temperature of 19 °C ± 1 °C.

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6.2.5 Take the test piece from the predip and rinse it thoroughly, first under running water and then in distilled water or deionized water.

6.2.6 Immerse the test piece completely, standing it upright, in the test solution (see 3.2) and leave for exactly 15 min at a constant temperature of 38 °C ± 1 °C.

NOTE Uniformity of temperature within the test solution is very important; this can be achieved by using a water-bath and stirring continuously.

Do not use the test solution after more than 4,5 g of anodic oxidation coating have been dissolved per litre of solution.

Do not use the test solution which has been in contact with materials other than anodized aluminium or its alloys.

6.2.7 Take the test piece from the test solution and rinse it thoroughly, first under running water and then in distilled water or deionized water. Dry the test piece as specified in Annex A and weigh immediately to the nearest 0,1 mg (mass m_2).

6.2.8 During the operations described in 6.2.2 to 6.2.7, avoid touching the test piece with bare hands.

Take extreme care that the two drying operations required in 6.2.3 and 6.2.7 are carried out in the same reproducible way and avoid heating to temperatures above 60 °C.

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7 Expression of results

Calculate the loss in mass of surface δ_A, in milligrams per square decimetre, using Equation (1):

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

where

m_1 is the mass, in milligrams, of the test piece before immersion in the (predip and) test solution;

m_2 is the mass, in milligrams, of the test piece after immersion in the (predip and) test solution;

A is the coated surface area of the test piece, in square decimetres, in contact with the (predip and) test solution.

8 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;

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c) whether Method 1 or Method 2 was used;

d) how the significant surface area has been determined;

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e) whether the test solution has been stirred;

f) the result of the test (see Clause 7);

NOTE Acceptance levels will normally be specified in the relevant product specification.

g) any deviation, by agreement or otherwise, from the procedure specified;

h) the date of the test.

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Annex A
(normative)

Method for the drying of samples

A.1 Degrease the test piece by gentle agitation for 30 s in a organic solvent, e.g. acetone or ethanol (96 %), 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 exactly 15 min with the coated surfaces 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 and rinsing (see 6.1.5 and 6.2.7), repeat operations A.1 and A.2, omitting the use of organic solvent.

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