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Anodizing of aluminium and its alloys — Specification for hard anodic oxidation coatings on aluminium and its alloys

Anodisation de l'aluminium et de ses alliages — Spécification pour l'anodisation dure de l'aluminium et des alliages d'aluminium

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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).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

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 10074:2017), which has been technically revised. The main changes compared with the previous edition are as follows:

— pretest abrasion test numbers have been added as requirements to the abrasive wheel wear test.

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.

Introduction

Hard anodizing is an electrolytic treatment which results in the formation of a hard and usually thick coating of alumina used primarily for engineering purposes.

Hard anodizing can be applied to cast or wrought aluminium and aluminium alloys; however, alloys containing more than 5 % copper and/or 8 % silicon and die casting alloys require special anodizing procedures. To obtain optimum microhardness, wear resistance or low surface roughness characteristics, low contents of alloy are selected.

Unless otherwise specified, articles are anodized after all heat-treatment, machining, welding, forming and perforating operations. The best results are achieved on machined surfaces. Sharp edges are machined to a radius of at least 10 times the intended thickness to avoid “burning” and/or spalling.

Hard anodizing will usually result in a dimensional increase on each surface equal to about 50 % of the coating thickness. The dimensions of the component prior to anodizing will allow for this, if necessary.

The thickness is generally within the range of 25 µm to 150 µm. Low thickness (up to 25 µm) is sometimes used in a variety of applications, such as splines and threads. Normal thickness (50 µm to 80 µm) is used for wear or insulation requirements. High thickness (150 µm) is used for repairing purposes, but thick coatings tend to be softer in outer regions. Very hard coatings reduce the fatigue strength. This phenomenon can be minimized by applying shot peening before hard anodizing (see H.6), by reducing thickness and/or by sealing. Hard anodizing tends to increase surface roughness. This can be limited with low alloy contents and/or mechanical finishing.

Hard anodic oxidation coatings are mainly used to obtain the following:

— resistance to wear through abrasion or erosion;

— electrical insulation;

— thermal insulation;

— build-up (to repair parts out of tolerance on machining or worn parts);

— resistance to corrosion (when sealed).

Anodizing of aluminium and its alloys — Specification for hard anodic oxidation coatings on aluminium and its alloys

1 Scope

This document specifies requirements for hard anodic oxidation coatings on aluminium and its alloys, including test methods.

It also specifies the information to be supplied by the customer to the anodizer (see Annex A).

It is not applicable to coatings produced by processes such as those referred to as plasma electrolytic oxidation, micro-arc oxidation, plasma-chemical anodic oxidation, anodic spark deposition or spark anodizing.

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 1463, *Metallic and oxide coatings — Measurement of coating thickness — Microscopical method*

ISO 2106, *Anodizing of aluminium and its alloys — Determination of mass per unit area (surface density) of anodic oxidation coatings — Gravimetric method*

ISO 2360, *Non-conductive coatings on non-magnetic electrically conductive base metals — Measurement of coating thickness — Amplitude-sensitive eddy-current method*

ISO 2376, *Anodizing of aluminium and its alloys — Determination of breakdown voltage and withstand voltage*

ISO 4516, *Metallic and other inorganic coatings — Vickers and Knoop microhardness tests*

ISO 6344-1, *Coated abrasives — Grain size analysis — Part 1: Grain size distribution test*

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

ISO 8251, *Anodizing of aluminium and its alloys — Measurement of abrasion resistance of anodic oxidation coatings*

ISO 9227, *Corrosion tests in artificial atmospheres — Salt spray tests*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 7583 and the following 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/

3.1
lot
articles of the same nominal composition and temper which are processed together

3.2
lot acceptance test
test on a production *lot* (3.1) to determine its conformity to specified requirements

4 Material classification

The properties and characteristics of hard anodic oxidation coatings are significantly affected by both the alloy and the method of production.

Consequently, for the purposes of this document, materials are classified into five alloy groupings as follows.

— Class 1: all wrought alloys except those in Class 2.

— Class 2 (a): alloys of the 2 000 series and any other alloys that contain more than 5 % copper.

— Class 2 (b): alloys of the 5 000 series containing 2 % or more magnesium and alloys of the 7 000 series.

— Class 3 (a): casting alloys with less than 2 % copper and/or 8 % silicon.

— Class 3 (b): other casting alloys.

Information to be supplied by the customer to the anodizer shall be in accordance with Annex A.

5 Appearance

The significant surface shall be completely anodized. The visual appearance shall be substantially uniform. There shall be no spalling, blistering or powdery (burnt) areas. Visual examination shall be a lot acceptance test.

Crazing or microcracks shall not normally be a reason for rejection.

6 Thickness

Thickness measurements shall be made on the significant surfaces, but not within 5 mm of contact (jigging) marks, nor in the immediate neighbourhood of a sharp edge.

Measurement shall be made using either the non-destructive eddy current method described in ISO 2360 or the destructive microscopical method described in ISO 1463. In the case of a dispute, the microscopical method (ISO 1463) shall be used.

Measurement of thickness or, where relevant, final dimensions shall be dealt with in a lot acceptance test.

7 Surface density

The surface density (coating mass per unit area), when measured in accordance with ISO 2106 on unsealed anodic oxidation coatings with a nominal thickness of 50 µm ± 5 µm, shall have the minimum values given in Table 1.

If the coating thickness is not 50 µm, the surface density shall be corrected proportionately.

WARNING — A method specified in ISO 2106 requires the use of a reagent containing chromium(VI). Chromium(VI) is toxic and its solutions are hazardous to the environment and severely hazardous to waters.

Table 1 — Minimum surface density

Material class	Minimum acceptable value
Class 1	1 100 mg/dm^2
Class 2 (a) (b)	950 mg/dm^2
Class 3 (a)	950 mg/dm^2
Class 3 (b)	By agreement

8 Resistance to wear/abrasion

8.1 General

The resistance to wear/abrasion shall be measured on unsealed anodic oxidation coatings (see NOTE). Due to good correlation achieved with other properties, resistance to wear/abrasion shall be tested in accordance with B.1, using the abrasive wheel wear test method described in ISO 8251.

NOTE Resistance to abrasion can be measured on sealed anodic oxidation coatings but hydrothermal sealing and/or dyeing can reduce the resistance to abrasion/wear by over 50 %.

When the abrasive wheel wear test method is not appropriate (especially on some curved surfaces), resistance to wear/abrasion shall be tested in accordance with B.2, using the abrasive jet test method described in ISO 8251. This test gives an average for the total coating thickness.

The abrasive wheel wear test method assesses the resistance to abrasive wear. The abrasive jet test method assesses the resistance to erosive wear (erosion). Thus, the results are not necessarily comparable.

The Taber abrasion test method in accordance with B.3 may only be used when specified.

8.2 Abrasive wheel wear test method

The resistance to wear/abrasion shall be determined by the measurement of loss in coating thickness or loss in coating mass. When determined in accordance with B.1, using the abrasive wheel test method described in ISO 8251, the final value shall be an average of at least three tests using a load of 19,6 N ± 0,5 N and grade P240 silicon carbide paper (as described in ISO 6344-1). The loss in coating thickness or in coating mass shall exclude any loss due to the pretest abrasion.

The acceptance values shall be in accordance with Table 2. The standard specimen, in accordance with Annex C, shall be tested each day, under the same conditions as those used for the test specimen. When the loss in coating thickness is used, each thickness value shall be the average of 10 readings in the test area.

The time between hard anodizing and abrasion testing shall be at least 24 h. During this period, the test specimens shall be stored in the test environment.

Table 2 — Acceptance values for abrasive wheel wear test

Alloy	**Pretest abrasion number of double strokes** ds	**Abrasion test number of double strokes** ds	**Relative mean specific abrasion resistance acceptance value** % compared to standard specimen (in accordance with Annex C)
Class 1	100	800	≥80 %
Class 2 (a)	100	400	≥30 %
Class 2 (b)	100	800	≥55 %
Class 3 (a)[a]	100	400	≥55 % } or by agreement (see NOTE)
Class 3 (b)[a]	100	400	≥20 % } or by agreement (see NOTE)

NOTE The relative mean specific abrasion resistance (RMSAR) is given by the formula.

$$\text{RMSAR} = \frac{\text{Mean wear resistance of test specimen}}{\text{Mean wear resistance of standard specimen}} \times 100$$

where the wear resistance is the number of double strokes, which is necessary to remove 1 µm (or 1 mg) of coatings.

The wear resistance, R_w, is calculated using the following formula for thickness measurement or similarly for mass loss measurement:

$$R_w = \frac{N}{d_1 - d_2}$$

where

N is the number of double strokes excluding the pretest abrasion double strokes;

d_1 is the average thickness in micrometres or the average mass in milligrams after the pretest abrasion;

d_2 is the average thickness in micrometres or the average mass in milligrams after the abrasion test.

[a] Castings are not always suitable for abrasion/wear testing because of the surface condition and/or the structure of the anodic oxidation coating. In the unusual event of Class 3 alloys requiring to be tested, the abrasion/wear resistance acceptance value shall be agreed upon between the anodizer and the customer and can require special reference specimens.

8.3 Abrasive jet test method

The resistance to wear/abrasion shall be determined by either the mass of silicon carbide or the time required to penetrate the coating. When determined in accordance with B.2, using the abrasive jet test method described in ISO 8251, the final value shall be an average of at least three tests.

The acceptance values shall be in accordance with Table 3.

The time between hard anodizing and abrasion testing shall be at least 24 h. During this period, the test specimens shall be stored in the test environment.

Table 3 — Acceptance values for the abrasive jet test

Alloy	Relative mean specific abrasion resistance acceptance value % compared to standard specimen (see Annex C)
Class 1	≥80 %
Class 2 (a)	≥30 %
Class 2 (b)	≥55 %
Class 3 (a)[a]	≥55 % } or by agreement (see NOTE)
Class 3 (b)[a]	≥20 % } or by agreement (see NOTE)

NOTE The relative mean specific abrasion resistance (RMSAR) is given by the formula.

$$\text{RMSAR} = \frac{\text{Mean wear resistance of test specimen}}{\text{Mean wear resistance of standard specimen}} \times 100$$

where the wear resistance is the duration, in seconds, or mass of abrasive, in grams, necessary to remove 1 μm of coating thickness.

[a] Castings are not always suitable for abrasion/wear testing because of the surface condition and/or the structure of the anodic oxidation coating. In the unusual event of Class 3 alloys requiring to be tested, the abrasion/wear resistance acceptance value shall be agreed upon between the anodizer and the customer and can require special reference specimens.

8.4 Taber abrasion test method

When determined in accordance with B.3, the final value shall be an average of at least three tests. The acceptance values shall be in accordance with Table 4.

Table 4 — Acceptance values for the Taber abrasion test

Alloy	Acceptance value (maximum loss in mass) mg
Class 1	15
Class 2 (a)	35
Class 2 (b)	25
Class 3	[a]

[a] Castings are not always suitable for abrasion/wear testing because of the surface condition and/or the structure of the anodic oxidation coating. In the unusual event of Class 3 alloys requiring to be tested, the abrasion/wear resistance acceptance value shall be agreed upon between the anodizer and the customer and can require special reference specimens.

9 Vickers microhardness

The Vickers microhardness of the hard anodic oxidation coating, when measured in accordance with ISO 4516 on a coating with a thickness of 25 μm to 50 μm, shall have the minimum values given in Table 5.

The test load should be 0,49 N and, for thin anodic oxidation coatings or anodic oxidation coatings of some alloys, the test load should be agreed upon between the anodizer and the customer.

Table 5 — Acceptance values for the Vickers microhardness test

Alloy	**Microhardness,** HV 0,05
Class 1	400
Class 2 (a)	250
Class 2 (b)	300
Class 3 (a)	250
Class 3 (b)	By agreement

NOTE Coatings thicker than 50 μm can have lower microhardness values, especially in the outer regions.

10 Resistance to corrosion

This test is only applicable to sealed oxidation coatings.

If a corrosion test is required (see Annex A), the anodic oxidation coating shall be tested for 336 h in accordance with ISO 9227 [neutral salt spray (NSS) test].

A test specimen with a normal anodic oxidation coating thickness of 50 µm shall not show, after 336 h of exposure to neutral salt spray, any corrosion pits except those within 1,5 mm of jigging marks or corners.

NOTE Failure of this test can indicate flaws or discontinuities in the anodic oxidation coating and not a sealing deficiency.

Annex A
(normative)

Information to be supplied by the customer to the anodizer

The following information shall be supplied, when appropriate, by the customer to the anodizer:

a) a reference to this document, i.e. ISO 10074;

b) material designated (alloy and temper);

c) the extent of significant surface(s);

d) the thickness of the anodic oxidation coating required;

e) the preferred position and dimensions of the contact (jigging) marks;

f) final dimensional tolerances;

g) any special characteristic required, such as corrosion resistance, electrical insulation, freedom from surface scratches, lot hardness requirements or roughness before and after treatment;

h) sampling procedure, if required (see Annex D);

i) any requirements for measurement of breakdown voltage (in accordance with Annex E);

j) any requirements for process qualification and approval (in accordance with Annex F);

k) any special packaging and/or delivery requirement (see Annex G);

l) any special pre-treatment or post-treatment (especially sealing) required (see Annex H).

Annex B
(normative)

Abrasion test

B.1 Abrasive wheel wear test

Abrasion testing using the abrasive wheel wear test described in ISO 8251 has shown good correlation with surface density, and as such, it is the preferred test method. However, higher loads are required because of the higher abrasion resistance. The increased surface roughness of these coatings can cause measurement difficulties, so the top 2 µm or 3 µm shall be abraded to produce a more reproducible starting point by a pretest abrasion of 100 double strokes.

The increased abrasion resistance of hard anodic oxidation coatings requires the use of a higher load (19,6 N ± 0,5 N) and coarser silicon carbide paper (P240 in accordance with ISO 6344-1).

For some applications, comparative abrasive/wear testing using an agreed reference specimen may be preferred.

NOTE If loss in coating mass is used, it is important that there be no delay between weighing and testing, and/or between testing and reweighing.

B.2 Abrasive jet test

The abrasive jet test method is particularly suitable for components of complex or asymmetrical shape. The jet nozzle recommended by ISO 8251 uses high air velocities and low air flowrates. This results in very rapid abrasion and the possibility of normally measuring the time to penetrate the coating. Test conditions are shown in Table B.1.

Table B.1 — Test conditions of abrasive jet test

Parameter	Test conditions
Air pressure (kPa)	15
Mesh size of abrading medium (µm)	125
Flowrate of abrading medium (g/min)	25 ± 1

B.3 Taber abrasion test

B.3.1 Preparation of the grinding wheels

Clean the grinding wheels after each test, for 50 cycles using S11 paper.

Every four cycles, reface the grinding wheels with a diamond grinding machine, taking care to remove as little material as possible.

The limiting duration for use is one year after delivery.

B.3.2 Preparation of the test specimens

Allow at least 24 h to elapse between hard anodizing and abrasion testing. During this period, store the test specimens in the test environment.

B.3.3 Procedure

Place the test specimen on the wheel stand, which has been set to rotate at 60 r/min ± 2 r/min or 72 r/min ± 2 r/min.

NOTE The speed of rotation is the same irrespective of power supply frequency. If the speed of rotation is different, the test result can be different.

Set both CS 17 grinding wheels and load each of them with 1 000 g.

Place the dust extractor nozzle within 0,8 mm to 1,5 mm of the test specimen.

Start the extractor.

Set the cycle selector on 1 000 cycles.

Start the test.

When the apparatus stops, remove the test specimen from the wheel stand.

Weigh the test specimen to the nearest 0,1 mg (mass m_0).

Place the test specimen on the wheel stand again.

Set the CS 17 grinding wheels.

Adjust the dust extractor nozzle.

Start the extractor.

Set the cycle selector on 10 000 cycles.

Record the temperature and humidity.

Start the test.

When the apparatus stops, remove the test specimen from the wheel stand.

Record the temperature and humidity at the end of the test.

Reweigh the test specimen to the nearest 0,1 mg (mass m_1).

B.3.4 Expression of results

The loss in mass, Δm, in milligrams, is given by Formula (B.1):

$$\Delta m = (m_0 - m_1) \quad \text{(B.1)}$$

where

m_0 is the mass of the test specimen after 1 000 cycles, in milligrams;

m_1 is the mass of the test specimen after 10 000 cycles, in milligrams.

B.4 Test report

The test report shall include at least the following information:

a) a reference to this document, i.e. ISO 10074;

b) identification of the test specimen and, if appropriate, the standard specimen and the reference specimen;

c) the test method used;

d) the result of the test;

e) temperature and humidity recording before and after each test;

f) any deviations from the procedure;

g) any unusual features observed including the conduct of the test or the nature of the test specimen or test area;

h) the date of the test;

and for the abrasive wheel wear test:

i) the force between the abrasive wheel and the surface of the specimen;

j) the abrading medium used;

and for the abrasive jet test:

k) the angle between the plane of the test area and the jet axis;

l) the abrading medium and its particle size;

m) the air or gas pressure.

Annex C
(normative)

Preparation of standard specimen

The standard anodized specimen for abrasion test purposes shall be prepared from polished or bright-rolled aluminium sheet as follows:

Aluminium specification:	Al 99,5
Thickness:	at least 2 mm

NOTE 1 The recommended size of test specimen is 140 mm × 70 mm or 100 mm × 100 mm.

The following processing conditions shall be carefully observed:

Pretreatment:	degreasing only (light caustic etching or acid pickling is permissible)
Anodizing:	bath composition
Free sulfuric acid concentration:	180 g/l ± 2 g/l
Aluminium concentration:	1 g/l to 5 g/l
Rest:	deionized water

Conditions of anodizing:

Temperature:	0 °C ± 0,5 °C
Current density:	3,5 A/dm^2 ± 0,35 A/dm^2
Strong agitation:	with a large volume of low pressure air or solution circulation
Anodizing time:	40 min
Anodic coating thickness:	50 µm ± 5 µm

The coating shall be unsealed and dried with cold air. The standard specimen shall be anodized vertically with the longitudinal axis positioned horizontally in the bath while maintaining vigorous agitation over the anode surface and smooth direct current with no more than 5 % ripple. Not more than 20 standard specimens shall be anodized at one time, and the volume of the electrolyte shall be not less than 10 l per test specimen.

NOTE 2 A standard specimen is tested at least once each day of testing.

NOTE 3 The standard specimens are most accurate and reproducible if anodized singly with careful control of all the conditions.

NOTE 4 The thicknesses of the anodic oxidation coatings of standard specimens have inherent variations of ±10 %.

Annex D
(informative)

Sampling procedures

When a customer wishes to be ensured that the quality of a lot or lots of anodized articles conforms to the quality specified, it is recommended that sampling be carried out in accordance with one of the sampling plans given in ISO 2859-1, using the guidance given in ISO 28590.

Thus, when a complete order, anodized in the same plant, is delivered in a series of three or more lots, a sampling plan shall be chosen on the basis of an acceptable quality limit (AQL) which represents the average percentage of nonconforming parts which the customer is prepared to tolerate.

Annex E
(normative)

Breakdown voltage

If required by the customer, the breakdown voltage of the anodic oxidation coating shall be determined using the method described in ISO 2376. The minimum acceptable value of breakdown voltage shall be agreed upon between the customer and the anodizer.

Annex F
(normative)

Process qualification and approval

When a process qualification is required, sample parts and/or panels shall be prepared for approval by the customer before production commences. Qualification items shall be processed according to an identified process schedule and the items shall be tested for compliance with the qualification requirements. No deviation from the process schedule shall be permitted without prior approval of the customer.

