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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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Contents

Page

Foreword iv
Introduction v
1 Scope 1
2 Normative references 1
3 Terms and definitions 2
4 Material classification 2
5 Appearance 2
6 Thickness 2
7 Surface density 3
8 Resistance to wear/abrasion 3
8.1 General 3
8.2 Abrasive wheel test method 3
8.3 Abrasive jet test method 4
8.4 TABER test method 5
9 Vickers microhardness 5
10 Resistance to corrosion 5
Annex A (normative) **Information to be supplied by the customer to the anodizer** 6
Annex B (normative) **Preparation of standard specimen** 7
Annex C (normative) **Abrasion testing** 8
Annex D (informative) **Sampling procedures** 11
Annex E (normative) **Breakdown voltage** 12
Annex F (normative) **Process qualification and approval** 13
Annex G (informative) **Packaging and delivery of anodized articles** 14
Annex H (informative) **Processing guidance** 15
Bibliography 17

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 10074 was prepared by Technical Committee ISO/TC 79, *Light metals and their alloys*, Subcommittee SC 2, *Organic and anodic oxidation coatings on aluminium*.

This second edition cancels and replaces the first edition (ISO 10074:1994), which has been technically revised.

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 phenomena can be minimized 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

— 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 International Standard specifies requirements for hard anodic oxidation coatings on aluminium and its alloys, including test methods.

Information to be supplied by the customer to the anodizer is given in Annex A.

NOTE This International Standard 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 referenced documents are indispensable for the application 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 1463:2003, *Metallic and oxide coatings — Measurement of coating thickness — Microscopical method*

ISO 2106:—[1)], *Anodizing of aluminium and its alloys — Determination of mass per unit area (surface density) of anodic oxidation coatings — Gravimetric method*

ISO 2360:2003, *Non-conductive coatings on non-magnetic electrically conductive basis materials — Measurement of coating thickness — Amplitude-sensitive eddy-current method*

ISO 2376:—[2)], *Anodizing of aluminium and its alloys — Determination of electric breakdown potential*

ISO 4516:2002, *Metallic and other inorganic coatings — Vickers and Knoop microhardness tests*

ISO 7583:1986, *Anodizing of aluminium and its alloys — Vocabulary*

ISO 8251:—[3)], *Anodizing of aluminium and its alloys — Measurement of abrasion resistance of anodic oxidation coatings*

ISO 9227:2007, *Corrosion tests in artificial atmospheres — Salt spray tests*

1) To be published. (Revision of ISO 2106:1982)

2) To be published. (Revision of ISO 2376:1972)

3) To be published. (Revision of ISO 8251:1987 and ISO 8252:1987)

3 Terms and definitions

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

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

3.2
lot acceptance tests
tests on a production lot to determine its conformance to the requirements of this specification

3.3
significant surface
part of the article covered or to be covered by the coating and for which the coating is essential for serviceability and/or appearance

NOTE Adapted from ISO 2064:1996, Definition 3.1.

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 International Standard, 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;

— 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.

5 Appearance

The significant surface shall be completely anodized and 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.

NOTE The usual coating thickness is between 40 µm and 60 µm (see introduction and Annex A). The test specimen is produced according to the conditions given in Annex B.

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.

Table 1 — Minimum surface density

Material class	Minimum acceptable value
Class 1	1 100 mg/dm^2
Class 2	950 mg/dm^2
Class 3 (a)	950 mg/dm^2
Class 3 (b)	By agreement
If the coating thickness is not 50 µm, the surface density shall be corrected proportionately.	

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 Annex C, C.1, using the abrasive wheel 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 method is not appropriate (especially on some curved surfaces), resistance to wear/abrasion shall be tested in accordance with C.2, using the abrasive jet test method described in ISO 8251:—, Clause 6. This test gives an average for the total coating thickness.

The TABER method (see C.3) may only be used when specified.

8.2 Abrasive wheel 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 C.1, using the abrasive wheel wear test method described in ISO 8251:—, Clause 5, the final value shall be an average of at least three tests, using a load of 19,6 N ± 0,5 N and silicon carbide paper of 240 mesh size.

The acceptance values shall be in accordance with Table 2. The standard specimen 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 ten readings in the test area.

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

Table 2 — Acceptance values for abrasive wheel test

Alloy	Number of double strokes (ds)	Relative mean specific abrasion resistance acceptance value % compared to standard specimen (see Annex B)
Class 1	800 to 100	⩾ 80 %
Class 2 (a)	400 to 100	⩾ 30 %
Class 2 (b)	800 to 100	⩾ 55 %
Class 3 (a)[a]	400 to 100	⩾ 55 % } or by agreement (see Note)
Class 3 (b)[a]	400 to 100	⩾ 20 % }

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

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

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

[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 may require special reference panels.

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 C.2, using the abrasive jet test method described in ISO 8251:—, Clause 6, the final value shall be an average of at least three tests.

The acceptance values shall be in accordance with Table 3.

Table 3 — Acceptance values for the abrasive jet test

Alloy	Relative mean specific abrasion resistance acceptance value % compared to standard specimen (see Annex B)
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 % }

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

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

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 may require special reference panels.

8.4 TABER test method

When determined in accordance with C.3, the TABER abrasion acceptance values shall be in accordance with Table 4.

Table 4 — Acceptance values for the TABER abrasive test

Alloy	**Acceptance value** (maximum loss in mass) mg
Class 1	15
Class 2 (a)	35
Class 2 (b)	25
Class 3	See Note
NOTE 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 may require special reference panels.	

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.

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.	

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 between the anodizer and the customer.

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 piece with a normal anodic oxidation coating thickness of 50 µm shall not show, after 336 h 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 necessarily a sealing failure.

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) the number of this International Standard;

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 (see Annex E);

j) any requirements for process qualification and approval (see Annex F);

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

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

Annex B
(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
Radius:	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 compressed air or solution circulation
Anodizing time:	40 min
Anodic coating thickness:	50 µm ± 5 µm

The coating shall be unsealed and air dried. 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 litres 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 Standard specimens at the present time have inherent variations of ± 10 %.

Annex C
(normative)

Abrasion testing

C.1 Abrasive wheel testing

Abrasion testing using the test described in ISO 8251:—, Clause 5 has shown good correlation between the hardness and 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 should be abraded to produce a more reproducible starting point by a pretest abrasion of 100 double strokes.

Class 2(a) alloys tend to be less abrasion resistant than Class 1 alloys, so a lower number of test double strokes has been specified in order to remove about 5 µm per 400 double strokes (see ISO 8251:—, Clause 5).

The increased abrasion resistance of hard anodic oxidation coatings requires the use of a higher load (19,6 N) and coarser silicon carbide paper (240 mesh size). For some applications, comparative abrasive/wear testing using an agreed reference specimen may be preferred (see ISO 8251:—, 5.3.6).

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

C.2 Abrasive jet testing

The abrasive jet test method is particularly suitable for components of complex or asymmetrical shape. The method described in ISO 8251:—, Clause 6 permits the use of two different nozzle assemblies. The nozzle described in Figure D.2 in 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.

The nozzle described in Figure D.3 in ISO 8251:— uses low air velocities and high air flowrates. This results in much longer test times (approximately 10 times) but the weight of abrasive medium used to penetrate the coating can be normally measured.

Table C.1 — Comparison of nozzles used in abrasive jet testing

Nozzle	ISO 8251:—, Figure D. 2	ISO 8251:—, Figure D.3
Air pressure (kPa)	15	15
Air flowrate (l/min)	15 ± 1	67 ± 2
Mesh size of abrading medium (µm)	125	125
Flowrate of abrading medium (g/min)	25 ± 1	25 ± 1

C.3 TABER abrasion test method

C.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 1 year after delivery.

C.3.2 Preparation of the test pieces

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

C.3.3 Procedure

Place the test piece on the wheel stand which has been set to rotate at 60 r/min ± 2 r/min or 70 r/min ± 2 r/min.

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 piece.

Start the extractor.

Set the cycle selector on 1 000 cycles.

Start the test.

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

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

Place the test piece 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 piece from the wheel stand.

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

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

C.3.4 Expression of results

The loss in mass Δm, in milligrams, is given by the equation

$$\Delta m = (m_0 - m_1)$$

where

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

m_1 is the mass, in milligrams, of the test piece after 10 000 cycles;

C.3.5 Test report

The test report shall include at least the following information:

a) a reference to this International Standard;

b) identification of the test specimen (including the alloy) and, if appropriate, the agreed reference specimen;

c) the calculated value of loss in mass;

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

e) any other observation concerning the conduct of the test or the nature of the test piece or test area;

f) the date of the test.

Annex D
(informative)

Sampling procedures

When a customer wishes to be assured 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[1], using the guidance given in ISO 2859-10[2].

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 level (AQL) which represents the average percentage of non-conforming parts which the purchaser is prepared to tolerate.

Annex E
(normative)

Breakdown voltage

If required by the customer (see Annex A), the breakdown voltage of the anodic oxidation 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.

Annex G
(informative)

Packaging and delivery of anodized articles

G.1 Packaging

Anodized articles should be packaged in a way that ensures that they will be protected during shipment and storage against damage due to mishandling, exposure to the weather or any normal hazard.

G.2 Delivery

Anodized articles should be prepared for shipment and delivery in accordance with good practice prevailing in the industry, to satisfy the carrier and assure safe transportation to the point of delivery. Packaging should conform to carrier rules and regulations applicable to this mode of transportation.

Annex H
(informative)

Processing guidance

H.1 Introduction

The equipment and processes employed should provide anodic oxidation coatings which meet the requirements of this International Standard.

Unless otherwise agreed, the processing conditions should be decided upon by the anodizer.

H.2 Masking (optional)

The purpose of masking is to prevent the treatment of certain areas of the article, especially for articles which contain parts which are not made of aluminium but of steel, brass or organic substances.

The different techniques available include

— wax,

— varnish or paint,

— mechanical masking, or

— use of traditional anodizing (such as chromic acid anodizing).

H.3 Jigging

The jigs should be made of aluminium alloy or titanium to ensure easy handling of articles and good electrical and mechanical contact.

The junction between article and jig should generally be achieved by clamping or bolting.

H.4 Degreasing

Surfaces should be clean and free from grease, oil, oxide, scale or other foreign matter. Different methods of degreasing available include

— degreasing by immersion in a solvent,

— degreasing using a vapour phase solvent, or

— alkaline or acid degreasing.

H.5 Pickling or etching (optional)

Pickling or etching may be used to prepare a deoxidized surface. However, it is seldom used before hard anodizing because of its effect on surface roughness. If it is necessary, an appropriate acid solution should be used.

H.6 Shot peening (optional)

Hard anodizing results in a reduction of the fatigue resistance of aluminium alloys. Shot peening before anodizing reduces the loss in fatigue properties.

H.7 Hard anodizing

Hard anodizing is usually carried out under the following conditions:

— electrolyte: the baths are generally composed of sulfuric acid and deionized water with or without additive(s);

— agitation: a strong and uniform agitation is important to remove heat from the work pieces;

— temperature: normally in the range −10 ˚C to +5 ˚C; for some special processes, the upper limit may attain +20 ˚C;

— electric current: the current used can be direct, alternative or direct with superimposed alternating or pulsating current.

H.8 Sealing (optional)

Sealing is normally carried out in boiling water with or without additive(s) when it is necessary to com- promise between hardness abrasion resistance and corrosion resistance. Sealing tends to lower the abrasion resistance and the microhardness of the anodic oxidation coating, and can lead to microcracks.

H.9 Mechanical finishing (optional)

Lapping or grinding can be used on articles to reach a precise dimension or to improve the surface roughness.

H.10 Impregnation (optional)

Coatings of molybdenum disulfide, polytetrafluoroethene (PTFE) or other approved materials can be applied over the hard anodic oxidation coating to improve the friction characteristics.

H.11 Solution control

Anodizing bath: the composition of the anodizing bath should be controlled by chemical analysis, performed at least weekly when the bath is in use, and the solution should be maintained within predetermined limits.

Sealing bath: the sealing bath should be checked for pH, and conductivity or additive concentration, as appropriate. These tests should be performed at least weekly, and the solution maintained within predetermined limits.

Bibliography

[1] ISO 2064:1996, *Metallic and other inorganic coatings — Definitions and conventions concerning the measurement of thickness*

[2] ISO 2859-1:1999, *Sampling procedures for inspection by attributes — Part 1: Sampling schemes indexed by acceptance quality limit (AQL) for lot-by-lot inspection*

[3] ISO 2859-10:2006, *Sampling procedures for inspection by attributes — Part 10: Introduction to the ISO 2859 series of standards for sampling for inspection by attributes*

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