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Aerospace series — Tartaric-Sulphuric-Acid anodizing of aluminium and aluminium wrought alloys for corrosion protection and paint pretreatment (TSA)

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National foreword

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Aerospace series - Tartaric-Sulphuric-Acid anodizing of aluminium and aluminium wrought alloys for corrosion protection and paint pre-treatment (TSA)

Série aérospatiale - Anodisation tartrique de l'aluminium et des alliages d'aluminium corroyés pour protection contre la corrosion et peinture (TSA)

Luft- und Raumfahrt - Weinsäure-Schwefelsäure-Anodisieren (TSA) von Aluminium und Aluminium-Knetlegierungen für den Korrosionsschutz und zur Vorbehandlung

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Foreword

This document (EN 4704:2012) has been prepared by the Aerospace and Defence Industries Association of Europe - Standardization (ASD-STAN).

After enquiries and votes carried out in accordance with the rules of this Association, this Standard has received the approval of the National Associations and the Official Services of the member countries of ASD, prior to its presentation to CEN.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by August 2012, and conflicting national standards shall be withdrawn at the latest by August 2012.

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1 Scope

This European Standard defines the requirement for Tartaric-Sulphuric-Acid (TSA) anodizing of aluminium and wrought alloys for corrosion protection and paint pre-treatment.

The purpose of this European Standard is to give design and quality requirements to manufactures.

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

EN 2101, *Aerospace series — Chromic acid anodizing of aluminium and wrought aluminium alloys*

EN 3665, *Aerospace series — Test methods for paints and varnishes — Filiform corrosion resistance test on aluminium alloys*

EN 6072, *Aerospace series — Metallic materials — Test methods — Constant amplitude fatique testing*

EN ISO 2106, *Anodizing of aluminium and its alloys — Determination of mass per unit area (surface density) of anodic oxidation coatings — Gravimetric method (ISO 2106:2011)*

EN ISO 2409, *Paints and varnishes — Cross-cut test (ISO 2409)*

EN ISO 9227 *Corrosion tests in artificial atmospheres — Salt spray tests (ISO 9227)*

EN ISO 15528, *Paints, varnishes and raw materials for paints and varnishes — Sampling (ISO 15528)*

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

3.1
de-anodizing
process, which removes the anodic oxide

3.2
smut
precipitations of alloying elements (e. g. Cu, Fe, Zn, Si) on the surface of parts after a process step normally after alkaline etching step

3.3
Mechanically Disturbed Layer
MDL
layer that is present at the surface resulting from the rolling process of the material

3.4
pit
surface corrosion defect at which the anodic coating is penetrated

3.5
process instruction
document that describes the detailed process, parameters, detailed steps, etc.

3.6
alloys
all aluminium alloys, that are treated with the TSA process in the specific shop

3.7
re-anodizing
repetition of the anodizing process step after complete de-anodizing

3.8
sealing
sealing (of the anodized layers) is applied to close the pores produced by the acid anodizing process. It is usually applied in demineralized water bath. Sealing can improve the corrosion resistance performance of the anodic film.

3.9
dichromate sealing
the dichromate sealing process has to be applied for unpainted parts

It content one of the following products:

— Sodium dichromate;

— Potassium dichromate.

It includes chromate and has to be replaced by a chromium III chemical conversion coating when available.

4 Principle

4.1 General description of process

TSA process is an electrochemical process used for generating an aluminium oxide layer for corrosion protection and surface treatment prior to application of a corrosion-inhibiting primer.

The TSA process is characterized by using an electrolyte composed of tartaric and sulphuric acid. Suitable surface preparation steps, e. g. alkaline cleaning, alkaline etching, acidic pickling/desmutting and subsequent steps such as rinsing (and sealing, if applicable), have to be defined and are integral to the TSA process.

4.2 Limitations

All processes that can compromise the anodic film such as forming, or heat-treatment shall be performed prior to surface preparation of the parts to be anodized.

TSA anodizing shall not be applied:

— for metal bonding applications;

— inside of oxygen pipes;

— for tubes, pipes and open holes with a length to diameter ratio higher than 10:1;

— for trapped holes with a length to a diameter ratio greater than 5:1;

— for parts or assemblies (e.g. spot-welded and riveted), which can permanently entrap;

— for components which can permanently entrap treatment solutions, except components that can be adequately masked.

NOTE The formation of oxide layer influences the dimensions of the part and should be considered for close tolerance parts.

5 Requirements

5.1 Technical requirements

5.1.1 Prior to anodizing step:

— the parts shall be free of oil, grease, marking inks and other surface contaminations;

— the surface shall be free from precipitations or smut from alloying elements or pre-processes indicated by the bright and uniform appearance of the surface;

— parts/areas that do not require anodizing shall be masked accordingly;

— mechanically disturbed layer shall be removed either by mechanical or chemical processes;

— in case of chemical pre-treatment, the final step prior to anodizing shall be acidic pickling, preferably chromate-free;

— in case of re-anodizing all residuals from the previous anodizing shall be completely removed.

5.1.2 During the anodizing process:

— the parts shall not be subjected to any tensile, flexure, torsion or other stress;

— the process shall performed such, that parts do not dry between single process steps (pre-treatment, anodizing, etc.).

5.1.3 After the anodizing procedure:

— parts have to be adequately rinsed with water according to 5.1.4;

— afterwards, the parts shall be either:

  — dried and painted within 16 h. The parts can be kept in a dry and dust-free atmosphere for 168 h before painting;

    or

  — sealed to achieve the desired corrosion resistance of the anodic film;

  — appropriate measure should be taken to prevent and to eliminate possible microbiological contaminations of the rinsing bath, e.g. UV radiation.

5.1.4 Water quality:

— All water used for preparation of the TSA bath and for the final rinsing step as well as for the sealing process shall be demineralized.

The water shall comply with the following requirements:

— pH value at 25 °C : 5,0 to 7,0;

— total residue [mg/l] : ≤ 20;

— conductivity [µS/cm] : ≤ 20.

5.2 Engineering requirements

5.2.1 Requirements to the anodic film

5.2.1.1 Visual aspects

The film shall be free of powdery areas, burnings, blisters and discontinuities such as scratches, ruptures or other damages.

5.2.1.2 Film thickness

The thickness of the anodic film shall be in a range of 2 µm to 7 µm. The thickness of the film can be checked by eddy current method (EN ISO 2360), optical microscope (EN ISO 1463), scanning electronic microscope (SEM), or equivalent examinations.

5.2.2 Corrosion prevention performance of unpainted parts

When tested in accordance to EN ISO 9227 the anodised and sealed post-treated specimens shall show:

— less than 2 pits/dm^2;

— no pit shall exceed 0,8 mm in diameter;

— no patchy dark grey areas (spots, streaks or marks) after an exposure time of minimum 14 days according to EN ISO 9227 for 2024 T351 unclad and 7175 T7351 unclad. There can also other materials be used if they are defined between the supplier and the customer.

The area of 2 mm from the specimen edges and marks shall not be evaluated.

5.2.3 Paint adhesion on anodic film

The coating system shall be agreed between the interested parties.

Test the adhesion in accordance with EN ISO 2409.

The damage of painted specimens shall not exceed:

— class 0 (according to EN ISO 2409) for specimen at initial stage;

— class 1 (according to EN ISO 2409) for specimen after 14 days of water immersion at (23 ± 2) °C.

5.2.4 Corrosion prevention performance of painted parts

5.2.4.1 Filiform corrosion

The maximum length of the longest filament shall not exceed 2 mm on either side of the scratches at 40 days of exposure when tested in accordance with EN 3665. Alloy in accordance with 5.2.2.

5.2.4.2 Corrosion resistance

There shall not be any corrosion, blister, extending further than max. 1,25 mm on either side of the scratch at 125 days of exposure when tested in accordance with EN ISO 9227. Alloy in accordance with 5.2.2.

5.2.5 Fatigue Requirements

The fatigue specimen shall be manufactured and tested in accordance with EN 6072.

All the fatigue test values shall be plotted on the Wöhler curves presented in Annex A (mean and minimum curves).

— at least 50 % of the sample shall be on or above the mean curve;

— 90 % of the samples shall be above the minimum curve.

5.3 Quality requirements

5.3.1 General requirements

All process steps, parameters and tests shall be defined in a process instruction.

5.3.2 Test frequency

The details and frequency for permanent and regular quality control checks shall be defined by the quality assurance of the shop considering:

— the ageing, contamination and the efficiency of the bath;

— the production rate.

After specific events as re-adjustment of refilling of the bathes appropriate performance of the process shall be ensured by quality tests.

5.3.3 Visual aspect of the parts

All relevant areas of the parts have to be covered by the anodic film. The anodic film shall be free of powdery areas, burnings, blisters and discontinuities such as scratches, ruptures or other damages. There shall be no water stains on surfaces to which a coating is to be applied.

The parts have to be inspected by naked eyes under constant light conditions. Checking of powdery areas or dusting is made with the help of a white and dry rag, which is slightly rubbed on the part to check. A slight dusting may be accepted so far as the paint appearance is not altered.

5.3.4 Thickness of anodic film

The thickness of the film can be checked by eddy current method (EN ISO 2360), optical microscope (EN ISO 1463), scanning electronic microscope (SEM), or equivalent examinations.

Alternatively 5.3.7 can be applied.

5.3.5 Film weight

The film weight shall be > 22 mg/dm^2 when tested. The film weight shall be determined in accordance with EN ISO 2106.

Alternatively 5.3.6 can be applied.

5.3.6 Corrosion resistance of anodic film

When tested in accordance with EN ISO 9227 the anodized and sealed specimens shall show:

— less than 2 pits/dm^2;

— no pit shall exceed 0,8 mm in diameter;

— no patchy dark grey areas (spots, streaks or marks).

after an exposure time of minimum 14 days for 2024 T351 unclad and 7175 T7351 unclad.

5.3.7 Sealing quality

After rinsing and scrubbing under water with a cotton cloth, the blue discoloration of the sealed surface shall be very light (a very faint colour is allowable) or have completely disappeared when tested in accordance with EN 2101, Annex B.

5.3.8 Paint adhesion

Refer to 5.2.3 however:

— specimen shall be painted before 16 h after anodizing;

— adhesion testing is performed according to EN ISO 2409 at initial stage only.

5.3.9 Quality check of the process–summary

All series production checks are summarized in Table 1 below.

Table 1 — Serial checks and minimum checking frequencies (see also 5.3.2)

Property	Requirement	Frequency	Material [a]	No of specimens per alloy
Visual aspect of parts	5.3.3	in accordance with EN ISO 15528, Table 1	— all parts	Not applicable
Thickness of anodic film	5.3.4	— regular, to be agreed between the interested parties; — after bath filling; — after production interruption.	— 2024 T3 clad — 7175 T7351 unclad	2
Film weight (optional)	5.3.5	— regular, to be agreed between the interested parties; — after bath filling; — after production interruption.	— 2024 T3 clad — 7175 T7351 unclad	2
Corrosion resistance	5.3.6	— regular, to be agreed between the interested parties; — after bath filling; — after production interruption.	— 2024 T3 unclad — 7175 T7351 unclad	3
Sealing quality	5.3.7	— regular	— 2024 T3 clad or unclad — relevant production alloys	1
Paint adhesion	5.3.8	— regular	— 2024 T3 clad or unclad — relevant production alloys	1

[a] There can also other materials be used if they are defined between the supplier and the customer.

6 Re-anodizing

One re-anodizing is allowed; any further re-anodizing shall be agreed between the interested parties.

7 Qualification Test Report (QTR)

The qualification test report shall document the qualification tests performed and its results. It shall include at least all the following information:

a) name and address of the production shop;

b) process instruction;

c) all the details regarding surface preparation (alkaline degreasing, acidic pickling etc.);

d) detailed process parameters (bath compositions, temperatures, times, voltage, current density, etc.);

e) date of the anodizing film realisation and date of the tests;

f) equipment, test method, test parameters and test conditions used, etc.;

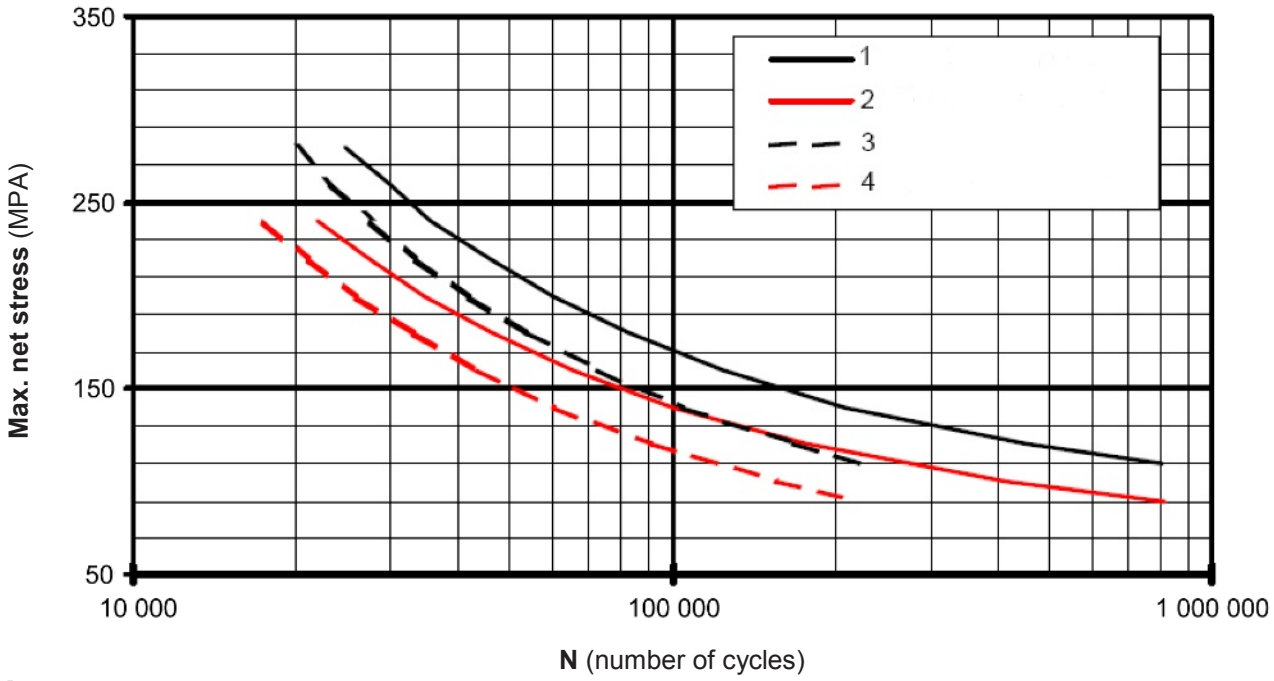
g) detailed report of the test results. This report shall present all the test values. For the visual inspection, the workshop to be qualified shall supply pictures of treated samples (specially after salt spray exposure);

h) any incident that may have affected the results and any deviation from this European Standard.

Annex A
(informative)

Wöhler curves and stress levels – Reference fatigue curves

See Figure A.1 and Table A.1.



Key

1 2024 T351 mean curve
2 7175 T7351 mean curve
3 2024 T351 min. curve
4 7175 T7351 min. curve

Figure A.1

Table A.1

2024 T351			7175 T351		
Stress max.	**Log N mean**	**Log N** min.	**Stress** max.	**Log N mean**	**Log N** min.
280	4,39	4,30	240	4,34	4,24
260	4,47	4,37	220	4,44	4,33
240	4,55	4,44	200	4,54	4,42
220	4,66	4,53	180	4,67	4,52
200	4,78	4,62	160	4,81	4,64
180	4,92	4,73	150	4,90	4,71
160	5,09	4,86	140	5,00	4,78
140	5,32	5,02	120	5,25	4,96
120	5,66	5,22	100	5,62	5,19
110	5,91	5,35	90	5,91	5,34

Bibliography

EN ISO 1463, *Metallic and oxide coatings — Measurement of coating thickness — Microscopical method (ISO 1463)*

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

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