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FOR SPACE STANDARDIZATION

Space product assurance

Black-anodizing of metals with inorganic dyes

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Foreword

This Standard is one of the series of ECSS Standards intended to be applied together with the management, engineering and product assurance standards in space projects and applications. ECSS is a cooperative effort of the European Space Agency, national space agencies and European industry associations for the purpose of developing and maintaining common standards.

Requirements in this Standard are defined in terms of what shall be accomplished, rather than in terms of how to organize and perform the necessary work. This allows existing organizational structures and methods to be applied where they are effective, and for the structures and methods to evolve as necessary without rewriting the standards.

The formulation of this Standard takes into account the existing ISO 9000 family of documents.

This Standard has been prepared by editing ESA PSS-01-703, reviewed by the ECSS Product Assurance Panel and approved by the ECSS Steering Board.

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Introduction

The thermal control onboard spacecraft is mainly passive. It is based on the thermo-optical properties of the surfaces, namely emissivity and absorbance. The ratio of these two properties defines the equilibrium temperature of the surface. This Standard aims at providing the thermal-optical properties given by a surface treatment applied on a metallic surface to achieve an emissivity versus absorbance ratio close to unity, as requested for many applications.

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Scope

This Standard defines the measurements and verifications to be performed to guarantee that an anodized coating is adequate for the intended application. The rigorous requirements set by this Standard ensure the high reliability of surface treatments intended to withstand normal terrestrial conditions and the vibrational G-loads and environment imposed on spacecraft and associated equipment where surfaces require high solar absorptance, high emittance, high optical blackness, or a combination of these properties.

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Normative references

The following normative documents contain provisions which, through reference in this text, constitute provisions of this ECSS Standard. For dated references, subsequent amendments to, or revisions of any of these publications do not apply. However, parties to agreements based on this ECSS Standard are encouraged to investigate the possibility of applying the most recent editions of the normative documents indicated below. For undated references, the latest edition of the publication referred to applies.

ECSS-P-001	ECSS - Glossary of terms
ECSS-Q-70-04	Space product assurance - Thermal cycling test for the screening of space materials and processes
ECSS-Q-70-09	Space product assurance - Measurement of thermo-optical properties of thermal control materials
ECSS-Q-70-13	Space product assurance - Measurements of the peel and pull-off strength of coatings and finishes using pressure-sensitive tapes

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Terms, definitions and abbreviated terms

3.1 Terms and definitions

For the purposes of this document the terms and definitions given in ECSS-P-001 and the following apply.

3.1.1
anodizing
placing a protective film coating on a metal surface by an electrolytic or chemical action

3.1.2
batch (material)
material that originates from the same casting lot, and followed the same transformation processes and the same heat treatment

3.1.3
emittance (ε)
ratio of the radiant intensity of the specimen to that emitted by a black body radiator at the same temperature and under the same geometric and wavelength conditions

Exampe 1 Hemispherical emittance (ε_h) - conditions for incidence or viewing of flux over a hemispherical region.

Exampe 2 Normal emittance (ε_n) - conditions for incidence or viewing through a solid angle normal to the specimen. Ratio refers to the emissivity normal to the surface of the emitting body

3.1.4
solar absorbance (α_s)
ratio of the solar radiant flux absorbed by a material (or body) to that incident upon it

NOTE Differentiation is made between two methods:

1. Spectroscopic method using a photospectrometer covering the range from 0,25 μm to 2,5 μm for the determination of α_s.
2. Portable equipment using a xenon flash for relative measurements (α_p).

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Principles and prerequisites of black-anodizing

4.1 Principles

The black-anodizing of a metal consists of submitting the part to be coated to a controlled oxidation treatment during which the metallic surface is transformed into an oxide layer, partly growing in the substrate. Compounds or dyes are sealed in the oxidized layer, thus providing the required optical or thermo-optical properties to the surface.

NOTE For space applications, only inorganic dyes are suitable, as radiation do not strongly impact on their thermo-optical properties.

4.2 Prerequisites

4.2.1 General

The adequacy of these treatments shall be demonstrated through a qualification programme. Depending on the specific project requirements, the coating applied shall fulfil part or all the acceptance criteria given in Clause 6 of this Standard.

4.2.2 Metal to be black-anodized

a. Not all metals and alloys are suitable for black-anodizing treatment. The most commonly treated are titanium, aluminium and their alloys. In particular, the composition of the alloy, the manufacturing process and the alloy temper can have an impact on the response to the black-anodizing.

b. Aluminium alloys containing a high amount of copper (5%), zinc (6%) or silicon (5%) are known to respond poorly to some treatments.

c. Wrought alloys are generally easier to treat than cast alloys.

d. The suitability of the alloy with respect to the anodizing process shall be demonstrated on test pieces having the same specific characteristics as the workpiece (see 6.2.1).

NOTE Failure occurred with 7xxx and 2xxx alloys with processes based on sulphuric acid anodizing. Failures were evidenced by an adhesive tape-test after thermo-cycling under vacuum.

4.2.3 Potential limitations on parts geometry and structure

a. The manufacturing process of the part to be coated can have an impact on its response to the black-anodizing process. Also the geometry of the hardware can limit the possibility to coat the part.

b. To ensure the best result for anodizing parts, the following shall be applied:

1. Unless otherwise stated, the parts to be treated shall be made of the same alloy and shall be from the same batch.
2. Unless otherwise stated, the parts shall only be made of the metal to be black-anodized, and any insertion of another metal shall be avoided.
3. The parts shall be designed in such a way that the chemical products used in the process can be thoroughly rinsed. The presence of drain holes is a possible means to avoid the retention of chemical within the hardware.
4. The parts shall contain no screws, no rivets nor other fastener elements.
5. If a part contains welded elements, a qualification programme shall be set up to qualify the black-anodizing on the specific weld. This qualification programme shall include a validation of the cleaning method used to rinse off the chemicals used for anodizing.
6. If a brazing alloy is used, the compatibility of the braze with the black-anodizing process shall be demonstrated.
7. Only welding and brazing processes for which the seam is continuous shall be used.

4.2.4 Aspect and dimensions of parts prior to anodizing

a. Care shall be taken while handling the parts to be black-anodized as the surface quality of an anodization layer mimics that of the underlying substrate.

b. In case of scratching, shock or any other event disturbing the surface aspect, the customer shall be informed and a nonconformance report issued.

c. The layer formed during anodizing consumes the bulk metal to grow both within and outside the initial dimensions of the bulk. The growth mechanisms of the layer and its impact on final hardware dimensions, including the potential grinding off of a porous part, shall be communicated to the customer.

d. The customer shall provide a written agreement on geometry impact prior to starting the black-anodizing process.

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Black-anodizing conditions

5.1 Specific equipment and environmental conditions

5.1.1 Facilities

a. The work area shall be clean and free of dust. Air used for ventilation shall be filtered to prevent contamination of the workpieces by moisture, oil or dust.

b. The environment shall fulfil the requirements of national health regulations. Individual protective equipment shall be used according to national health regulations.

5.1.2 Surface preparation and cleaning

a. A surface preparation treatment shall be performed on the part to be coated to remove any oil dust or other contaminants from the surface.

b. Lint-free gloves or surgical latex talc-free gloves shall be worn when handling the parts.

c. Other surface treatments can be applied to the part to, for example, improve the quality of the surface or increase its roughness.

d. Masking can be applied to the parts not to be anodized.

5.2 Anodizing

5.2.1 General

In the electrochemical process, each part that is anodized needs electrical contact.

a. The electrical contact shall be of a size compatible with the size of the hardware to be processed and with the process specifications.

b. The location of the electrical contact shall be chosen not to interfere with the functional usage of the part to be treated.

c. Depending on the exact electrochemical process used, specific parameters shall be controlled. Among those parameters are:

   1. composition and pH of the electrolyte,
   2. location of the electrodes and their distance with respect to the part,
   3. temperature of the bath,
   4. stirring of the bath,
   5. volume of the electrolyte with respect to that of the treated part, and
   6. process time.

d. A record of the in-process data shall be part of the process procedure.

5.2.2 Blackening

A difference shall be made between the "two-step" colouring and the "one-step" or "integral" colouring methods.

5.2.2.1 Two-step colouring

a. Step 1: The part shall be immersed in a bath containing an inorganic dye (metal compound); commonly used metals include tin, cobalt, nickel, and copper. The dye particles are driven into the pores of the oxide, with or without a current applied.

b. Step 2: The pores shall then be sealed, e.g. by immersion of the part in a boiling water based solution.

   NOTE After anodizing, the obtained oxide layer presents a columnar porous structure.

c. In such colouring techniques, it shall be verified that:

   1. The required thermo-optical properties are obtained, hence the minimum amount of dye in the coating is reached (see ECSS-Q-70-09).
   2. The coating does not expel dye during adhesion; this ensures that the pores of the coating are sealed (see ECSS-Q-70-13).

      NOTE A typical minimum thickness value for two-step colouring is 20 μm.

5.2.2.2 Integral colouring

a. This so-called one-step process combines anodizing and colouring to simultaneously form and colour the oxide cell wall. The layer obtained is generally very dense close to the bulk and porous in its outer part.

b. In such colouring techniques, it shall be verified that:

   1. The porous part of the coating is treated according to the process specifications (ground-off in most cases).
   2. The required thermo-optical properties are obtained (see ECSS-Q-70-09).

      NOTE A typical minimum thickness value is 8 μm.

5.2.3 Treatment termination

After completion of the black-anodizing process, the part treated shall be thoroughly rinsed in water and distilled water and then dried.

5.3 Handling and storage of materials and parts

5.3.1 Material storage

a. The materials shall be stored in a cleanliness-controlled area, with an ambient temperature of (20 ± 3) °C and a relative humidity of (55 ± 10) %.

b. The parts shall be stored in such a way as to prevent any damage or contamination by dust, moisture or grease.

c. Limited-life materials shall be labelled with their shelf lives and date of manufacture, or date of delivery if the date of manufacture is not known.

5.3.2 Material handling and storage

a. Coated parts shall only be handled with clean nylon or lint-free gloves.

b. The coated parts shall not be bent, twisted or subjected to any stress that can damage the coating.

c. Coated surfaces shall be shielded from contact by using polyethylene or polypropylene bags or sheets.

d. Mechanical damage shall be avoided by packing the polyethylene- or polypropylene-wrapped workpieces in clean, dust- and lint-free material.

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Acceptance criteria

6.1 Visual inspection

a. The parts shall be visually inspected with the naked eye, in particular at the locations of joints and small gaps.

b. Changes in surface colour or aspect shall be reported as input for the acceptance testing.

6.2 Verification testing

6.2.1 General

Unless otherwise dictated by project requirements, the following applies:

a. Every batch shall be subjected to acceptance tests as listed below, and any other tests that are dictated by mission or project requirements.

b. The test pieces shall be from the same material batch as the workpieces.

c. They shall have followed the same manufacturing process (including surface finishing).

d. They shall be cut in the same direction and have the same geometries and welding features as the workpieces (see 4.2.3)..

e. The specific characteristics of the workpieces (e.g. brazed joint, and small gap) shall be duplicated on the test pieces.

f. The test pieces shall be prepared at the same time as the workpieces to enable destructive and other tests to be performed.

g. Thermo-optical properties of the parts to be measured according to ECSS-Q-70-09. The requirements are:

1. Solar absorptance (α_s) shall be at least 0,93.
2. Normal emittance (ε_n) shall be at least 0,90.

NOTE The colour uniformity assessed by visual inspection is not directly linked to the above measurements, as the wavelengths of interest are not the same. The absorbtance and emittance uniformity is therefore often verified using the visual colour uniformity as input.

h. Thermal cycling. 100 cycles in a vacuum of $1,5 \times 10^{-3}$ Pa from –100 °C to +100 °C according to ECSS-Q-70-04 without any loss of adhesion.

i. Adhesion properties shall be measured according to ECSS-Q-70-13.

j. There shall be no signs of anodizing lifting from the substrate.

k. Thickness to be measured by any suitable means to verify that it is in accordance with process requirements.

l. Additional acceptance tests. Depending on the particular project or mission requirements, any of the following tests, or other specified tests, may be invoked in order to evaluate the finish of the workpiece or test piece:

1. vibration;
2. radiation;
3. cleaning;
4. outgassing;
5. humidity;
6. thermal shock;
7. salt spray;
8. electrical resistivity.

m. The acceptance shall be performed at all locations representative of the workpiece.

n. Tests detailed in ECSS-Q-70 series specifications should be used whenever possible, in preference to those of other documents.

6.2.2 Welding

a. Electrical conductivity shall be applied during most welding processes. The anodic coating shall be ground away where the weld is applied. This normally results in a very disturbed zone around the welding area.

b. Welding of black-anodized parts should not be performed.

c. If a black-anodized surface is welded, the impact of the welding shall be validated with dedicated engineering samples and dedicated qualification.

6.2.3 Repairing

a. Black-anodizing of a piece of hardware should be performed in one single run.

b. In the case of non-properly anodized parts requiring a repair, the repair procedure shall be validated with dedicated engineering samples and dedicated qualification.

NOTE The growth mechanism of the anodized layer is a complex process and coatings can have different morphologies.

7 Quality assurance

7.1 General

The quality assurance requirements are defined in ECSS-Q-20.

7.2 Data

The quality records (e.g. logbooks) shall be retained for at least ten years unless otherwise stated, and contain as a minimum the following:

a. copy of final inspection documentation;

b. index of limited-life articles and their use times;

c. nonconformance reports and corrective actions;

d. copy of the inspection and test results with reference to the relevant procedure, personnel, tools, equipment and baths;

e. an event log which is a chronological history of process operations and parameters, inspections and tests;

f. details of failure mode (if applicable).

7.3 Nonconformance

Any nonconformance that is observed in respect of the process shall be dispositioned in accordance with the quality assurance requirements, see ECSS-Q-20-09.

7.4 Calibration

Each reference standard and piece of measuring equipment shall be calibrated. Any suspected or actual equipment failure shall be recorded as a project nonconformance report so that previous results can be examined to ascertain whether re-inspection and re-testing is required. The customer shall be notified of the nonconformance details.

7.5 Traceability

Traceability shall be maintained throughout the process from incoming inspection to final test, including details of test equipment and personnel employed in performing the task.

7.6 Operator and Inspector training and qualification

a. Trained and competent personnel shall be employed for all operations and inspections related to the black-anodizing process.

b. Records shall be maintained of the training and qualification status of the operators and inspection personnel.

Annex A (informative) Recipes for aluminium treatment

A.1 General

In this Annex two baseline techniques for black-anodizing aluminium alloys are provided. When applied the processes described below are strictly applied, the coating does not always fulfil all the requirements of this Standard. An adaptation of those processes based on the know-how of the surface treatment companies increases their reliability.

A.2 Special utilities

a. Chemical bath(s) capable of :
   1. Containing the corrosive solution processes,
   2. Accommodating the workpiece,
   3. Maintaining the solution temperatures:
      (24 ± 2) °C
      (25 ± 2) °C
      (45 ± 2) °C
      (93 ± 2) °C
      (99 ± 1) °C
   4. Air agitation of the solution.

b. Ultrasonic bath capable of accommodating the workpiece (if used).

A.3 Hazards, health and safety precautions

a. For materials and parts with hazardous characteristics, see ECSS-Q-40. Attention shall be paid to health and safety precautions. Hazards to personnel, equipment and materials shall be controlled and minimized.

b. Location of hazards. Items and controls shall be so located that personnel are not exposed to hazards such as chemical burns, electric shocks, cutting edges, sharp points or toxic atmospheres.

c. Warning notes: Suitable warning and caution notes shall be provided in operations, storage, transport, testing, assembly, maintenance and repair instructions, and distinctive markings on hazardous items, equipment or facilities for personnel protection.

d. Process procedures are described in detail in the following subclauses, while simplified processes are provided in Tables A-3 to Table A-6.

e. The details of the type of hazard for each material used in the process are given in Table A-1.

Table A-1: Chemical species used and associated hazard

Item no.	Description	Type of hazard
1	Trichloro-ethylene	Harmful
2	Trisodium phosphate, $Na_3PO_412H_20$	Harmful
3	Sodium carbonate, Na_2CO_3	Harmful
4	Nitric acid, HNO3	Corrosive, oxidizing
5	Sulphuric acid, H_2SO_4	Corrosive
6	Deionized water	-
7	Cobalt acetate (reagent grade)	-
8	Ammonium hydrosulphide (reagent grade)	Corrosive
9	Nickel acetate (reagent grade)	Harmful
10	Boric acid	-
Harmful: Substances can have limited effects on health and should not be inhaled, swallowed or absorbed through the skin.		
Corrosive: Substances can destroy living tissue and contact with the skin shall be avoided.		
Oxidizing: Substances produce highly exothermic reactions in contact with other substances, especially flammable or combustible materials. Separation from other hazardous substances and use in no-smoking zones should be observed.		

NOTE Other chemical products can be used instead of those indicated, e.g. sodium hydrosulphite instead of ammonium hydrosulphide.

A.4 Surface preparation

a. Before anodizing, all parts shall be cleaned in the sequence given. These treatments shall immediately precede anodizing:

1. Vapour degreasing in trichloro-ethylene for 30 minutes.
2. Etching shall be carried out in a solution of:
   Trisodium phosphate $Na_3PO_412H_20$ 12,5 g/l
   Sodium carbonate Na_2CO_3 6,2 g/l
   The temperature of the solution should be held at (93 ± 2) °C with a part-immersion time of 5 minutes.
3. Rinsing with water.
4. Desoxidizing for 3 minutes at room temperature in a 50 volume percent solution of nitric acid in water.
5. Rinsing with water.
6. Removal of all residues before anodizing by scrubbing or ultrasonic cleaning.
7. Visual inspection of the workpiece shall be performed to check the absence of residues or particles. If residues or particles are found, a

complementary cleaning procedure shall be applied, for instance, ultrasonic cleaning or scrubbing.

8. Thorough rinsing in deionized or distilled water.

b. The above cleaning sequence may be performed using different solutions from the ones indicated, provided that the prepared surface is fully degreased, etched, and free of particles and residues.

A.5 Anodizing process set-up

A.5.1 Hardware

Cathode materials should be either lead or aluminium.

Any metallic part of a suspension device that makes contact with the electrolyte shall be of aluminium or titanium.

Workpieces shall be suspended in such a way that good electrical contact is maintained throughout the treatment.

A.5.2 Anodizing bath composition

The anodizing electrolyte shall consist of a solution of sulphuric acid 150 g/l in deionized or distilled water. During the lifetime of the electrolyte, it shall be kept within the limits given in Table A-2.

Table A-2: Anodizing electrolyte composition limits.

Chemical species	**Requirement**
Sulphuric acid H_2SO_4	120 g/l to 180 g/l
Chloride	Lower than the equivalent of 0,2 g NaCl/l
Aluminium content	Lower than 3 g/l
Fluorine content	Lower than 0,001 g/l

A.5.3 Experimental parameters

A.5.3.1. Temperature

a. The bath temperature during anodizing shall be kept within the manufacturer's specification.

b. Air agitation of the electrolyte shall be sufficient to keep the specimen to electrolyte temperature difference below 0,5 °C.

A.5.3.2. Current

Current density shall be between 1 A/dm^2 and 2 A/dm^2.

A.5.3.3. Duration

Anodizing time shall be set to give a thickness of the anodic film that fulfils the project requirements in terms of surface optical properties, corrosion and wear resistance. Typical thicknesses can range from 10 to 35 microns. The thickness can be estimated from the following formula:

$$\frac{(current\ density)(time)}{3{,}22} = thickness \qquad (1)$$

with

- current density expressed in A/dm^2
- time expressed in minutes
- thickness expressed in μm.

A.5.3.4. Cleaning

Immediately after removal from the bath, the parts shall be washed to remove the electrolyte, and finally rinsed with deionized water.

A.6 Dyeing and sealing process

A.6.1 General

There should be minimum delay between the rinsing and dyeing operations to avoid lowering the absorptive capacity of the film. However, dyeing can be delayed up to a maximum of 1 hour, if the anodized parts are kept in cold deionized water.

A.6.2 Cobalt sulphide black dying process

A.6.2.1. Solutions

The three bath solutions shall consist of:

a. A solution in deionized water of 200 g/l reagent-grade cobalt acetate to be heated to (45 ± 2) °C.

b. A solution in deionized water of 30 g/l reagent-grade ammonium hydrosulphide, maintained at (24 ± 2) °C.

c. A solution of 5 g/l nickel acetate and 5 g/l boric acid to be maintained at a temperature of (99 ± 1) °C. The pH of the solution shall be kept at 5,5 to 5,8.

A.6.2.2. Procedure

a. The wet parts shall be immersed in the cobalt acetate solution, maintained at (45 ± 2) °C, for 15 minutes.

b. The workpieces shall then be rinsed with deionized water to remove excess cobalt acetate solution.

c. Immediately thereafter, the workpieces shall be immersed in the ammonium hydrosulphide solution at (24 ± 2) °C until a deep black coloration is attained. This takes between 5 and 15 minutes.

d. Immediately afterwards, the parts shall be sealed by immersion in the solution of nickel acetate and boric acid at (99 ± 1) °C and pH 5,5 to 5,8, for 25 minutes.

A.6.3 Nickel sulphide black dyeing process

A.6.3.1. Solutions

The two bath solutions shall consist of:

a. A solution in deionized water of 50 g/l reagent-grade nickel acetate; maintained at (25 ± 2) °C.

b. Ammonium hydrosulphide 25 %.

A.6.3.2. Procedure

a. The wet parts shall be immersed in the nickel acetate solution at (25 ± 2) °C for 3 minutes.

b. The workpieces shall then be rinsed with deionized water to remove excess nickel acetate solution.

c. Immediately thereafter, the workpieces shall be immersed in ammonium hydrosulphide for 3 minutes.

d. The workpieces shall be rinsed with deionized water.

e. Steps b. to d. of the above procedure shall be repeated until the parts are deep black. This takes normally 3 to 5 dips.

f. The parts shall be sealed by immersion in boiling deionized water for 25 minutes.

A.7 Summary of the two processes described in Annex A

The tables below summarize the two processes described in this Annex.

Table A-3: Summary of pre-treatment (see A.4)

Process step	Solution composition	Required conditions
Vapour degreasing	Trichloro-ethylene	30 min
Etching	Trisodium phosphate 12,5 g/l Sodium carbonate 6,2 g/l	(93 ± 2) °C 5 min
Rinsing	Deionized water	
Desoxidizing	50% nitric acid in water	3 min
Rinsing	Deionized water	
Cleaning		Scrubbing / ultrasonic
Rinsing	Deionized / distilled water	

Table A-4: Summary of anodizing (see A.5)

Process step	Solution composition	Required conditions
Immersing	Sulphuric acid 150 g/l in deionized / distilled water $[Cl^-]$ < equivalent 0,2 g NaCl/l $[Al^{3+}]$ < 3 g/l $[F^-]$ < 0,001 g/l	Temperature within manufacturer's specification $1 < J < 2$ A dm^{-2} Time set to reach 25 to 35 µm thickness (see A.5.3.3)
Rinsing	Deionized water	

Table A-5: Dyeing with cobalt sulphide (see A.6.2)

Process step	Solution composition	Required conditions
Immersing	200 g/l cobalt acetate in deionized water	(45 ± 2) °C 15 min
Rinsing	Deionized water	Remove excess cobalt acetate
Dyeing	30 g/l ammonium hydrosulphide in deionized water	(24 ± 2) °C until deep-black colour (5 min to 15 min)
Pore sealing	5 g/l nickel acetate, 5 g/l boric acid	(99 ± 1) °C 5,5< pH < 5,8 25 min
Dyeing shall commence within 1 hour of anodizing.		

Table A-6: Dyeing with nickel sulphide (see A.6.3)

Process step	Solution composition	Required conditions
Immersing	50 g/l nickel acetate in deionized water	(25 ± 2) °C 3 min
Rinsing	Deionized water	Remove excess nickel acetate
Immersing	Ammonium hydrosulphide 25%	3 min
Rinsing	Deionized water	
Repeat above steps until part is deep black (3 to 5 times)		
Pore sealing	Deionized water	Boiling 25 min
Dyeing shall commence within 1 hour of anodizing.		

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Bibliography

ECSS-Q-20	Space product assurance - Quality assurance
ECSS-Q-20-09	Space product assurance – Nonconformance control system
ECSS-Q-40	Space product assurance – Safety

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ECSS Change Request / Document Improvement Proposal

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1. Originator's name: **Organization:** **e-mail:**	**2. ECSS Document number:** **3. Date:**

4. Number.	**5. Location of deficiency** clause page (e.g. 3.1 14)		**6. Changes**	**7. Justification**	**8. Disposition**

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