# BS EN 4707:2014



# **BSI Standards Publication**

Aerospace series — Acid pickling of aluminum and aluminum alloy without hexavalent chromium



BS EN 4707:2014 BRITISH STANDARD

#### National foreword

This British Standard is the UK implementation of EN 4707:2014.

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A list of organizations represented on this committee can be obtained on request to its secretary.

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#### **English Version**

# Aerospace series - Acid pickling of aluminum and aluminum alloy without hexavalent chromium

Série aérospatiale - Décapage acid de láluminium et des alliages dáluminium sans chrome hexavalent

Luft- und Raumfahrt - Beizen von Aluminium und Aluminiumlegierungen ohne sechswertiges Chrom

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# **Foreword**

This document (EN 4707:2014) 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of an identical text or by endorsement, at the latest by October 2014, and conflicting national standards shall be withdrawn at the latest by October 2014.

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# 1 Scope

This standard specifies the acid pickling of aluminium and aluminium alloys.

It is applicable whenever referenced.

# 2 Purpose of process

- **2.1** Pre-treatment before penetrant testing/penetrant inspection.
- **2.2** Pre-treatment before anodizing.
- **2.3** Final treatment for parts prior to adhesive bonding application.
- **2.4** Final treatment before welding or brazing.
- **2.5** Pre-treatment before chemical milling.
- **2.6** Final treatment for parts prior to painting application.
- **2.7** Pre-treatment for parts prior to surface conversion coatings.
- **2.8** Desmutting (Blanchiment), after alkaline etching or acid pickling.

#### 3 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 9100, Quality Management Systems - Requirements for Aviation, Space and Defense Organizations

#### 4 Information for the processor

- Designation;
- Number of the material standard and metallurgical condition of the latter;
- Sequence of operations;
- Areas to be processed;
- Treatment.

## 5 Condition of parts prior to processing

Fabrication of the parts shall have been completed before pickling.

#### **Toolings:**

Toolings, fixturings, and metal tooling masking must be protected from the corrosion and/or free from corrosion or another deterioration prejudicial to the treatment during their use.

Design of the toolings and racking must be achieved in order to:

avoid any retention of air or solution on the parts,

- allow the neutralisation and the suppression of the solutions during rinsing operations,
- avoid any accidental contact during treatment between parts and tank equipments, and between various parts.

#### Tanks and rackings:

Materials used must be resistant to temperature and chemically compatible with the products involved in the sequence of operations.

#### 6 Process schedule

The pickling process shall be performed according to Table 1.

Table 1

		Post Pickling processes								
Process schedule		Penetrant testing/ penetrant inspection	Anodizing	Adhesive bonding application	Welding Or brazing	Preparation before masking for chemical milling	Painting application	Surface conversion coatings	Desmutting , after etching or pickling	
Pre-treatments – Clause 7		Applicable	Applicable	Applicable	Applicable	Not applicable	Applicable	Applicable	Applicable	
Pickling – Clause 8		Applicable	Applicable	Applicable	Applicable	Applicable	Applicable	Applicable	Applicable	
Doot	Rinsing	Applicable	Applicable	Applicable	Applicable	Applicable	Applicable	Applicable	Applicable	
Post- treatments	Drying	Applicable	Not applicable	Applicable	Applicable	Applicable	Applicable	Not applicable	Not applicable	
Post pickling maximum lead-time		< 16 h <sup>a</sup>	Immediately in process	< 8 h	< 8 h <sup>a</sup>	< 8 h <sup>a</sup> or Immediately in process	< 16 h <sup>a</sup>	Immediately in process	Immediately in process	

<sup>&</sup>lt;sup>a</sup> Recommended (in case of over lead time an appropriate storage and visual inspection (according 11.1) should be carried out before and after the post pickling process).

#### 7 Pre-treatments

Heavily soiled/Polluted parts shall be emulsion cleaned, vapour degreased or solvent cleaned.

If there is a significant natural oxide film (ex.: laminated parts or oxidized parts), carry out abrasive blasting or chemical deoxidizing.

# Sequence of operations

- 1) Pre-degreasing:
- alkaline degreasing (with rinsings);
- or solvent;
- or vapour solvent.

NOTE If necessary (laminated parts / oxidized parts), do a chemical deoxidizing (with rinsings) or abrasive blasting after pre-degreasing.

A second alkaline degreasing shall be performed after pre-degreasing, if necessary.

The alkaline degreasing shall be:

- corrosion inhibited;
- preferably of a non-silicated type (especially for dye penetrant inspection).

Generally alkaline solutions with pH between 9 and 11 are considered as a degreasing-etching solutions, whereas those with pH between 7 and 9, are considered only as a degreasing solution.

For the parts to be bonded, a non-silicated alkaline solution is mandatory.

3) After alkaline degreasing/cleaning, the parts shall be thoroughly water rinsed and inspected for water break. Cleaning shall be repeated if break occurs.

#### 8 Treatment

Immediately after pre-treatments, the parts shall be pickled in one of the solutions specified below.

To ensure a well pickled surface, excessive bath loading and shielding of parts by each other shall be avoided:

Type of substances that can be used in pickling:

- Nitric or sulfonitric;
- Phosphosulfonitric;
- Sulfonitro-ferric fluoride free;
- Sulfonitro-ferric with fluoride.

#### 9 Rinsing

After each treatment bath, water rinsing is mandatory. This operation has to be composed by at least one recycled rinsing. Recycled rinsing is generally completed by non-recycled or low-flow rate rinsing. Several quality of rinsing water can be used but the water used before surface treatment (see Table 1) rinsing shall have a conductivity not more than 70  $\mu$ S/cm.

Rinsing shall be carried out consecutively to the pickling step in running water with a temperature not exceeding 40 °C.

# 10 Drying

The parts shall be dried except for those submitted for subsequent chemical treatment, anodic treatment or desmutting/chemical conversion ...

The temperature shall not exceed 80 °C.

After drying, parts shall be protected against contamination.

In case of bonding and paint applications, parts shall be stored under clean, dry conditions.

# 11 Required characteristics and inspection

#### 11.1 Parts

#### 11.1.1 Inspection before treatment

By visual inspection, make sure that the parts to be protected/treated are free from defects such as: scratches, corrosion pits, cracks ... prejudicial to the aspect or the effectiveness of protection. Adherent chips and metal particles must be removed beforehand using a clean cloth.

#### 11.1.2 Cleanliness

During the degreasing phases, each rinsing operation must be followed by a water break test in order to evaluate the wettability (and the cleanliness) of the surfaces.

This test consists of covering the parts with a uniform water film. This water film must cover the parts for at least 30 s without any discontinuity in the film. Otherwise, degreasing phases have to be repeated.

#### 11.1.3 Inspection after treatment

When subjected to visual inspection, the pickled surfaces shall be of a homogeneous appearance and free from pitting, stains and contamination.

#### 11.2 Process

- 11.2.1 Air used for drying or other operations shall be substantially dry and free from oil.
- 11.2.2 Chemical analysis of the pickling solutions (and different baths involved in the sequence of operations) shall be carried out at regular intervals to determine the content of heavy metal, chloride, iron copper etc., when requested.
- 11.2.3 The bath shall be discarded when pitting of parts occurs or when solid material starts to precipitate on cooling the solution to ambient temperature. The process must be managed by monitoring the significant parameters as aluminium and polluant contents.
- 11.2.4 The purity of the rinsing water shall be determined by conductivity/resistivity measurements.
- 11.2.5 When required for adhesive bonding, the test to the relevant bonding standard shall be carried out to ensure that the process has been correctly applied.
- 11.2.6 Each pickling process must be characterized by its specific etching rate range. All pickling process must not lead to localized attack.
- 11.2.7 The etching rate of the complete sequence of operations must be periodically measured to ensure the control of the process.

NOTE The etching rate and maximum dipping time cannot be generally specified but has to be specified according to final customer requirements.

#### 12 Quality assurance

#### 12.1 Approval of the processor

See EN 9100.

## 12.2 Process approval

The processor shall carry out:

- pickling on a series of test pieces or pre-production parts agreed between the purchaser and processor;
- tests specified in this standard, unless otherwise agreed between the purchaser and processor (e.g.: dissolution rate, morphology, roughness, ...).

When the test results have been accepted/considered as satisfactory by the purchaser, he shall give his written approval to start production.

The procedure shall not be changed without previous agreement from the purchaser.

# 12.3 Acceptance

After pickling the parts shall have the required characteristics according to Clause 12.

The frequency and nature of the inspection shall be specified in the design documents or agreed between processor and purchaser.

Parts that do not meet the requirements shall be rejected.

Parts which develop water break shall be reprocessed.

# 13 Health, safety and environmental aspects

The locally applicable regulations and laws shall be observed.

# Annex A (normative) Pickling bath

# Table A.1

	Post pickling application								
Pickling bath	Penetrant testing/ Penetrant inspection	Anodizing	Adhesive bonding application	Chemical milling	Welding or brazing	Painting/ Organic film application	Surface conversion coatings	Desmutting, after etching or pickling	
Nitric Or sulfonitric	Applicable	Applicable	Applicable	Applicable	Applicable	Applicable	Applicable	Applicable	
Phospho- sulfonic	Not applicable	Applicable	Not applicable	Not applicable	Not applicable	Applicable	Applicable	Not applicable	
Sulfonitro- ferric Without fluoride	Applicable	Applicable	Applicable	Applicable	Applicable	Applicable	Not applicable	Applicable	
Sulfonitro- ferric With fluoride	Applicable	Applicable	Not applicable	Applicable	Applicable	Applicable	Not applicable	Applicable	





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