

AIPS
Airbus Process Specification
Pickling of Titanium and its alloys

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1 Scope

This Airbus Process Specification defines the requirement for "Pickling of Titanium and its alloys".

The purpose of this specification is to give design and quality requirements to manufacturers. Although the essential requirements of a process are defined, the specification does not give complete in house process instructions, these shall be given in the manufacturers detailed process instruction and supporting work instructions.

This specification shall not be used as an inspection document unless parts or assemblies have been manufactured according to this specification.

It shall be applied when mentioned in the relevant standard, material specification or drawing.

2 Normative references

This Airbus specification incorporates by dated or undated reference provisions from other publications. All normative references cited at the appropriate places in the text are listed hereafter. For dated references, subsequent amendments to or revisions of any of these publications apply to this Airbus specification only when incorporated in it by amendment of revision. For undated references, the latest issue of the publication referred to shall be applied.

AIMS 03-18-002	Titanium Ti-P99002 annealed sheet and strip 0,1 mm < a < 6,0 mm
AIMS 03-18-004	Titanium Alloy (Ti6Al4V) annealed sheet 0,3 mm < a < 6,0 mm
AIMS 04-04-001	Corrosion inhibiting primer
ISO 4287	Geometrical Product Specification – Surface texture: Profile method
ISO 2409	Paints and varnishes – Cross cut test
ASTM-E-1447	Determination of Hydrogen in Titanium and Titanium Alloys by the Inert Gas Fusion Thermal Conductivity/Infrared Detection Method
EN 9103	Quality management systems – Variation management of key characteristics

3 Definition, applicability and limitations

3.1 General description of process

The pickling process of Titanium and its alloys is a chemical surface treatment used for removing all traces of contamination, oxides, scales and any other trace of dissimilar materials without causing damage of base metal.

According to the specific pickling process, indicated in the relevant Process Instruction, the process is applicable to following applications:

- General cleaning for removing marks, traces, metallic contamination after or before other treatments as a heat treatment, hot forming, superplastic hot forming, welding, chemical-electrolytic treatment, etc.
- Cleaning and material removal pretreatment before penetrant-liquid inspection.
- Cleaning and pretreatment before paint application.
- Cleaning and pretreatment before non-structural adhesive bonding (except acidic pickling process).

According to the specific pickling process, indicated in the relevant Process Instruction, the process may be applicable from parts with no oxidized surfaces (e.g. machined parts) to parts with heavily oxidized surfaces (e.g. after heat treatments).

If needed the pickling process may be combined with mechanical methods (abrasive blasting) and indicated in the Process Instruction and according to the relevant AIPS for the mechanical cleaning process.

3.2 Definitions

Pickling:	Chemical treatment to remove all surface contaminations, scale, oxide rests, by immersing the part in an acid or alkaline solution.
Scale:	Heavy oxidised surface layer formed during heat treatments, forging operation, etc.
Blasting:	Cleaning of parts to remove rests of contamination on surface by mechanical abrasion by means of an abrasive material.
Etching factor:	Amount of material removed by etching as a function of weight loss.
Batch:	A batch consists of the set of parts treated with the same parameters (time, temperature,...).
Process Instruction:	Document that describes the detailed process, parameter, detailed steps, etc., which have to be successfully qualified against this AIPS.

3.3 Applicability

This Airbus specification is applicable when invoked by the drawing directly or through another document for the purpose given in the scope. When processing to AIPS 09-02-005 is required it shall be invoked on the drawing by the words "Pickling of titanium and its alloys according to AIPS 09-02-005". Process instructions shall not be called on drawing.

3.4 Limitations

Pickling process shall not be applied to parts with a very complex geometry or shape that could avoid free flow of degreasing and pickling solutions across its surface, unless it has been demonstrated that no retention and homogeneous treatment is obtained.

This process shall also not be used on parts containing joints and cavities where it is not possible to mask prior to pickling or to ensure complete removal of the pickling solution.

Joined parts with dissimilar materials shall not be pickled unless other materials have been previously masked properly to avoid localised attacks.

Pickling process is not allowed as pre-treatment for structural bonding.

Acidic pickling shall not be applied to parts that have to be subsequent bonded.

The use of chlorinated products in the treatment baths of titanium or titanium alloys is prohibited.

4 Requirements

4.1 Technical requirements

Before the pickling step, parts shall be completely cleaned. Cleaning can be aqueous degreasing, vapour-phase organic solvents degreasing for strongly contaminated parts, blasting for parts with a high content of scale, or salt bath immersion to remove external layers.

The surface of the parts must be free of grease, paint, diffusion zones or other surface contaminations.

Parts must be completely immersed in the processing solution to prevent localized effects, except when otherwise qualified.

Baths shall be properly agitated after a long period of non use, and after adding chemical products or water.

For cleaning operations, note the limitations for the use of chlorinated cleaning agents.

All pretreatments shall be indicated in the Process Instruction and the overall process shall fulfill the requirement of this AIPS.

4.2 Engineering requirements

4.2.1 Visual Aspect (appearance)

Pickled parts shall have a uniform appearance and be free of grease, oil, oxides, scales, smut, corrosion or other contamination on the surface. No damage from the pickling operation, for example pitting or cracking shall be observed. Surfaces shall be of a uniform grey colour.

4.2.2 Dimensional control and surface roughness measurement

The treated parts must fulfil the required dimensional limits and tolerances in the Design Drawing. Surface roughness measured according to ISO 4287 must be maintained within required limits for each case and shall not be higher than 4 μm (Ra) in any case.

4.2.3 Etching rate

Checking according to § 5.2.4, pickling solution shall have a controlled etching factor such that it shall be and recorded and maintain its value within established range in the Process Instruction.

4.2.4 Hydrogen content

In case where the Process Instruction allows immersion times in pickling baths longer than 10 minutes or material removal higher than 0.025 mm, the hydrogen content increase of the material, measured according to ASTM-E-1447, shall be lower than the relevant material specification limit and no more than 30 ppm for Ti6Al4V and 20 ppm for Ti T40.

4.2.5 Paint adhesion (only for qualification as pre-treatment for painting)

When the pickling process is a pretreatment for subsequent paint application and when tested according to ISO 2409, the measured adherence shall be Gt0 between the base material and the paint at the initial state. After 14 days water immersion a Gt1 damage can be allowed.

4.3 Quality requirements

4.3.1 General requirements

When processing according to this AIPS all installations shall be continuously operated within the parameters defined in the Process Instruction.

All installations shall comply with the general and cleanliness requirements as defined in the Process Instruction.

This shall be ensured by permanent control of:

- The facilities (installation and equipment) and the products used.
- Conformance of the application process conditions to the requirements of this document and the Process Instruction.

All records of test / inspection results shall be stored adequately under the control of the quality assurance of the shop.

All operators of the process shall be adequately trained.

If doubts exist as to whether any one of the requirements is not satisfied, the process must be stopped and appropriate corrective action taken to ensure that the quality assurance requirements will subsequently be fulfilled.

4.3.2 Test frequency

Minimum test frequency is indicated in following chapters. 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 additional quality tests.

4.3.3 Bath composition

All the process baths shall be always kept in absolute cleanliness conditions and composition and other established process parameters values shall always be within the indicated limits. If necessary, composition can be adjusted with suitable additions.

Chemical analysis of baths (concentration of chemical compounds and dissolved titanium alloy) shall be performed by suitable chemical methods with the frequency established by Quality Assurance department and as a function of bath use. Obtained composition values and quantity of added components shall be recorded. Measurements also shall be performed before running of a new bath, after bath replenishment with chemical compounds or water and when the bath is warmed up after stopping.

4.3.4 Requirements to the pickling process

4.3.4.1 Visual Aspect (appearance)

100% of parts shall be visually inspected according to § 5.2.1.1. and shall show a uniform appearance and be free of grease, oil, oxides, scales, smut, corrosion or other contamination on the surface. No damage from the pickling operation, for example pitting or cracking shall be observed. Surfaces shall be of a uniform grey colour.

4.3.4.2 Etching rate

Etching rate measured according to § 5.2.1.4. shall be obtained before running a new bath and after each replenishment with chemical compounds or water. Quality Assurance Department may establish another frequency of testing. The etching factor shall be recorded and maintain its value within established range in the Process Instruction.

4.3.4.3 Hydrogen content

Measurement of hydrogen content shall be performed according to § 5.2.1.5. with a frequency of testing established by Quality Assurance Department when the pickling process is operated with long immersion times (> 10 minutes), material removal higher than 0.025 mm or when there is an elevated risk of Titanium embrittlement. Hydrogen content increase shall be lower than the relevant material specification limit and no more than 30 ppm for Ti6Al4V and 20 ppm for T40.

4.3.4.4 Paint adhesion (only as pre-treatment for painting)

Frequency and number of samples as defined by the relevant Quality Assurance Department, but must be carried out at least once per month for baths used for pickling process as a pre-treatment for subsequent painting. When tested according to § 5.2.1.6., the measured adherence shall be Gt0 between the base material and the paint at the initial state. After water immersion a Gt1 damage can be allowed.

Table 1 : Quality requirements with minimum test frequency

Property	Requirement	Test description	Minimum frequency	Material	Number of samples
Composition of bath	4.3.3.	4.3.3.	Regular according to Quality Assurance department / before starting new bath / after bath replenishment / after bath refilling/ once a week	N/A	N/A
Visual aspect of parts	4.2.1.	5.2.1.1.	100 % of parts	N/A	N/A
Etching factor	4.2.4.	5.2.1.4.	Regular according to Quality Assurance department/ before starting new bath / after bath replenishment / after bath refilling	Ti6Al4V-annealed	- 1
Hydrogen content	4.2.5.	5.2.1.5.	Regular according to Quality Assurance department	Ti6Al4V – Annealed Ti-T40 - annealed	-3 -3
Paint adhesion (only as pre-treatment for painting)	4.2.6.	5.2.1.6.	Regular according to Quality Assurance department / once per month	Ti6Al4V-annealed	- 3 (dry) - 3 (after water immersion)

4.4 Key Characteristic

Key Characteristics acc. to EN9103 are defined by responsible engineering based on a risk analysis for parts manufactured by this process. Key characteristics shall be defined on product level and if necessary also on process level.

They shall be subject to variation control by production organization according to EN9103.

Key Characteristics do not relieve the production organization from meeting all engineering requirements defined in this document.

Table 2: Key Characteristic

No.	Product Key Characteristic		Sub.- No.	Process Key Characteristic	
	Designation	Requirement/ Limit		Designation	Requirement/ Limit
1	Visual Aspect	Surface free of oils, greases paint, diffusion zones or other contaminants §4.1	1.1	Correct pre-treatment	Acc. to AIPI 09-01-002 AIPI 09-01-003 AIPI 02-02-002 AIPI 02-02-003
		Uniform surface free of oxides, scales smut, corrosion or contamination §4.2.1	1.2	Correct bath composition and etching time	AIPI 09-02-005
2	Surface roughness (& Mechanical Properties)	$Ra \leq 4.0 \mu m$ §4.2.2	2.1	Correct bath temperature, bath composition and etching time	AIPI 09-02-005
3	Hydrogen embrittlement	Lower than the relevant material specification §4.2.4	3.1	Correct bath composition and etching time	AIPI 09-02-005
4	Paint adhesion (only when used as pre-treatment for paint)	Gt0 dry test Gt1 wet test as per §4.2.5	4.1	Ensure that Product Key Characteristics 1 and 3 are met.	Acc. to AIPI 09-02-005

5 Process qualification

5.1 Basic qualification requirements

The general process qualification, the qualification of industrial facilities and Airbus quality assurance are described in prevailing procedure documents.

The pickling process shall be stable and reproducible before launching the technical qualification, so that the selected set of process steps and parameters is taken for the qualification.

The production shop shall use its facilities under serial production conditions for the performance of the qualification tests.

Process parameters, environmental conditions etc. applied for qualification shall ensure process performance within the entire process window applied at the relevant shop.

Qualification of the anodizing process shall consider all paint or paint schemes that are subsequently applied at the specific shop.

Qualification test program (QTP)

The means for showing compliance with the requirements for achieving technical qualification of the process shall be defined by the qualification test program.

This includes the standard qualification test defined by this AIPS hereinafter (chapter 5.2) as well as any amendment or deviation.

5.2 Standard Qualification Test Program

This chapter provides the details of the standard test program, which shall be taken into account for the definition of the specific qualification test program (QTP) for the technical qualification of a shop.

In addition to the acceptance criteria, specific test requirements are defined as amendment and/or deviation from the specified test method.

5.2.1 Demonstration of the required performance and quality level

5.2.1.1 Visual aspect (appearance)

Three specimens of parts shall be visually inspected by naked eyes under constant light conditions or with a magnifying glass of any magnification. Then check for colour, uniformity, burns, scratches or other discontinuities. Inspection must detect the possible presence of foreign particles or substances, oxide or scale rests, pits and assure uniform surface aspect after the treatment. Surfaces shall have a uniform appearance, absence of grease, oil, oxides, scales or other contamination. No damage coming from handling operation shall be observed. Surfaces shall be of a uniform grey colour.

5.2.1.2 Dimensional control and roughness measurement

Three samples shall be sized before and after treatment. They shall be subjected to roughness measurement according to ISO 4287 and surface roughness measured must be maintained within required limits for each case and shall not be higher than 4 µm (Ra) in any case.

5.2.1.3 Etching factor

The etching factor of pickling solutions shall be determined by measuring the weight loss. Weight shall be obtained with a precision of 0,1 mg. Initial sample thickness shall be measured with a suitable micrometer (measurement until 0,01 mm). Measurements are performed on samples of Ti6Al4V, with representative dimensions of 1 dm². Samples shall be treated with the same parameters (Time and Temperature) as the parts of the batch. Etching factor shall be calculated according following formula:

$$F(mm/h/face) = \left(\frac{Initial_weight(g) - Final_weight(g)}{Initial_weight(g)} \right) \times \left(\frac{Initial_thickness(mm) \times 60}{2 \times immersion_time(min)} \right)$$

Etching rate shall fulfil the requirements indicated the Process Instruction.

5.2.1.4 Hydrogen content

Determination of increase on hydrogen content on surface shall be done on three samples of Ti6Al4V and three of T40 with a thickness of 0,7-1,2 mm after an immersion time enough to remove 0,025 mm of material per surface (immersion time of more than 10 min.). Analysis shall be done with a vacuum hot extraction method, according to ASTM-E-1447. Hydrogen content increase shall be lower than the relevant material specification limit and no more than 30 ppm for Ti6Al4V and 20 ppm for T40.

5.2.1.5 Paint adhesion (only as pre-treatment for painting)

Paint adhesion test specimens shall be paint primed manufactured in accordance with AIMS 04-04-001 and tested in accordance with ISO 2409. Three samples shall be tested at initial state and three samples after 14 days of deionized water immersion at (23 ± 2)°C. The measured adherence shall be Gt0 between the base material and the paint at the initial state. After water immersion a Gt1 damage can be allowed.

Table 3: Specimens selection for Qualification Test Program

Property	Test description	Material	Number of samples
Visual aspect of parts	5.2.1.1.	Ti6Al4V-annealed	- 3
Dimensional and roughness control	5.2.1.3.	Ti6Al4V-annealed	- 3 - 3
Etching factor	5.2.1.4.	Ti6Al4V-annealed	- 3
Hydrogen content	5.2.1.5	Ti6Al4V – annealed Ti-T40 - annealed	-3 -3
Paint adhesion (only as pre-treatment for painting)	5.2.1.6.	Ti6Al4V-annealed	- 3 (dry) - 3 (after water immersion)

5.3 Qualification test report (QTR)

The qualification test report shall document the qualification tests performed and its results. It has to be written in English language.

The test report shall make reference to this AIPS and include at least all the following information:

- Name and address of the production shop
- Process instruction
- All the details regarding surface preparation (cleaning, descaling, alkaline degreasing, etc.)
- Detailed process parameters (bath compositions, temperatures, times, etching factor for applicable case, etc.).
- Date of the performed pickling process for the pieces and date of the tests.
- Equipments, test methods, test parameters and test conditions used, etc.,
- Detailed report of the test results. This report shall present all the test values.
 - o For the visual inspection, the workshop to be qualified shall supply pictures of treated samples (to check for pickling efficiency and absence of contamination traces, or rests of dissimilar materials, etc.).
 - o For dimensional control, record change of dimensions and roughness values.
 - o Obtained etching factor values shall also be recorded, including precision and apparatus for weight and thickness measurement.
 - o For hydrogen content, record the results of analysis (increase of hydrogen content on parts), including a description of used apparatus and extraction method.
- Any incident that may have affected the results and any deviation from this AIPS.

RECORD OF REVISIONS

Issue	Clause modified	Description of modification
1 12/06		New Standard
2 04/08	§4.2.4, §4.3.4.3, §5.2.1.3, §5.2.1.4	New issue, text changed in the hydrogen embrittlement paragraph, modification of the method to determine the etching rate
3 04/10	4.4	Implementation of key characteristics