

# Process Specification for Passivation and Pickling of Metallic Materials

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**Engineering Directorate**

**Structural Engineering Division**

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# Process Specification for Passivation and Pickling of Metallic Materials

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REVISIONS		
VERSION	CHANGES	DATE
Baseline	Original version	5/96
A	Formatting, modified callout, replacement of canceled QQ-P-35C with AMS-QQ-P-35, addition of pickling of Monel	11/16/98
B	Moved requirement from section 5 to section 6, allowed Type VI passivation for precipitation-hardened stainless steels, added alternate embedded iron verification test, modified other periodic testing.	5/17/01
C	Allowed a shop option for choosing the passivation type in section 3.0; wording changes in section 6.2; wording changes in section 9.0, and added requirement that personnel certification be written; and modified the definition of "Passivation" in section 10.	1/25/02
D	Moved paragraph 3.1 to paragraph 6.1 and updated, renumbered, and renamed remaining paragraphs in section 6.	10/19/05
E	Removed alternate embedded iron verification test. Reworded first paragraph of section 8. Added Custom 465 and 21-6-9 to list of alloys.	09/08/06
F	Updated references and signature page. Corrected Table for 420, 416, 416se alloys and added Nitronic 60 alloy. Corrected the reference paragraph to 3.2.1.1 from 4.4.1.2 for the humidity test. Removed type VII and VIII passivation because these chemistries are not used on JSC hardware.	7/15/2019
G	Moved caution notes to beginning of section 3.0. Moved Table 1 from section 3.0 to section 6.0. Deleted SOP 009.66 from section 3.0. Clarified that JSC hardware is required to be Method 1 and Class 4. Specified pre and post treatments. Prohibited descaling prior to passivation. Added Type VII and Type VIII to Table 1 for historical continuity. Combined general passivation and special passivation requirements and section 6.2 and 6.3. Combined general pickling requirements into pickling requirements and renumbered section. Alphabetized and formatted definitions section 10. Updated formatting and drawing note examples.	1/22/2021

## 1.0 SCOPE

This process specification establishes technical requirements for the passivation of corrosion-resistant steel in the manufacture of JSC flight hardware. In addition, it covers specialized pickling treatments for the removal of foreign particles from nickel-copper alloys.

Note: All references in this document to passivation refer to corrosion-resistant steel and all references in this document to pickling refer to specialized pickling for the removal of foreign metal from the surface of nickel-copper alloys.

## 2.0 APPLICABILITY

This process specification covers passivation treatments for use on austenitic, ferritic, martensitic, and precipitation-hardening corrosion-resistant steels. In addition, it covers specialized pickling treatments for the removal of foreign particles from nickel-copper alloys.

## 3.0 USAGE

**NOTE:** Do not passivate stainless steel parts that have hardened surfaces from nitriding, carbonitriding, or from other similar diffusion treatments. Severe corrosion and/or severe pitting of the hardened surfaces could occur.

**NOTE:** Do not passivate carburized stainless steels. The chromium combines with the carbon to form chromium carbides along the surface, which do not benefit from passivation.

Call out this process specification on the engineering drawing by using a drawing note that identifies the process specification and the type of passivation or pickling treatment. For example:

### **PASSIVATE PER NASA/JSC PRC-5002, TYPE VI**

If the passivation process is not critical to a particular design use:

### **PASSIVATE PER NASA/JSC PRC-5002**

When no passivation type is specified, then the process type will be selected by the shop from Table 1 (Section 6.2). The decision to allow this shop option should be coordinated in advance with the cognizant Materials and Processes engineer.

The types of passivation processes performed on JSC hardware are:

Type II	Medium-temperature nitric acid solution with sodium dichromate additive
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Type VI	Low-temperature nitric acid solution
Type VII	Medium Temperature Nitric Acid (not recommended for new design)
Type VIII	Medium Temperature, High Nitric Acid Concentration (not recommended for new design)

Type VI is the preferred process when permitted by Table 1. Type VI is the only in-house process at JSC. It utilizes no hazardous hexavalent chromium and is available in more metal finishing facilities. Older drawings that have Type VII and Type VIII listed should be updated to Type VI whenever possible. Type VII and Type VIII are not recommended for new design.

Alloys not listed in Table 1 may be passivated to this specification when approved by the cognizant Materials and Processes engineer via engineering drawing.

For pickling treatments of nickel-copper alloys, the standard engineering drawing callout shall be similar to the following example:

**PICKLE PER NASA/JSC PRC-5002, TYPE L**

Type L refers to a solution of hydrochloric acid and ferric chloride (Formula L from page 41 in Huntington Alloy IAI-21 (Second Edition, 1987).

**4.0 REFERENCES**

Document Number	Document Title
SOP-007.1	Preparation and Revision of Process Specifications
JPR 8500.4	Engineering Drawing System Requirements
AMS 2700	Passivation of Corrosion Resistant Steels
ASTM B117	Standard Practice for Operating Salt Spray (Fog) Apparatus
Huntington Alloy Booklet IAI-21 (Second Edition, 1987)	"Fabricating," Special Metals, Inc.
AMS-STD-753	Corrosion-Resistant Steel Parts: Sampling, Inspection and Testing for Surface Passivation
MPCV 70156	Cross Program Fluid Procurement and Use Control Specification

## 5.0 MATERIALS REQUIREMENTS

None

## 6.0 PROCESS REQUIREMENTS

### 6.1 WORK INSTRUCTIONS

Perform all work to written procedures. The work instructions shall contain sufficient detail to ensure that the manufacturing process produces consistent, repeatable products that comply with this specification.

For work performed at JSC facilities, these work procedures are documented via Detailed Process Instruction (DPI).

For contracted work, the contractor shall be responsible for preparing, maintaining, and certifying written work procedures that meet the requirements of this specification.

### 6.2 REQUIREMENTS FOR PASSIVATION

**NOTE:** Descaling or pickling before passivation is prohibited without additional direction being provided by the engineering drawing.

All passivation of corrosion-resistant steel shall be conducted according to the requirements of AMS 2700 *Passivation of Corrosion Resistant Steels*, with the modifications included in this process specification. Only Method 1 Nitric Acid shall be used. All corrosion resistance testing shall meet Class 4. When no passivation type is specified, the metal finishing facility has the option of using any type process, as allowed by Table 1.

Pre-treatment shall consist of a detergent cleaning and deionized water rinse per MPCV 70156 *Cross Program Fluid Procurement and Use Control Specification* Table 3.5-31.

Post treatment, parts should be rinsed with deionized water and dried.

The metal finishing facility shall maintain a record of the control procedures used for a given passivation treatment. The passivation solution shall be analyzed weekly to verify that the concentration of nitric acid (and sodium dichromate if applicable) meet the specified ranges in AMS 2700. The bath temperature of the passivation tank shall be verified before each use.

If a metal finishing facility uses the Type VI process for alloys in Table 1 marked with an asterisk (\*), the process bath shall be tested monthly to verify that the chloride concentration is less than 1000 ppm. Testing of bath for chloride before parts are processed is also acceptable.

**NOTE:** This specification and some drawings use the Roman numeral system for type from AMS-QQ-P-35.

Table 1: Passivation Types for Specific Alloys

Type II	Type VI	Type VII (not recommended for new design)	Type VIII (not recommended for new design)
	MP159		
	MP35N		
	IN718		
A286	A286		A286
13-8Mo	13-8Mo*		13-8Mo
15-5PH	15-5PH*		15-5PH
15-7Mo	15-7Mo*		15-7Mo
17-4PH	17-4PH*		17-4PH
17-7PH	17-7PH*		17-7PH
Custom 455	Custom 455*		Custom 455
Custom 465	Custom 465*		
21-6-9 (Nitronic 40)	21-6-9 (Nitronic 40)		
AISI 201	AISI 201	AISI 201	
AISI 202	AISI 202	AISI 202	
AISI 218 (Nitronic 60)	AISI 218 (Nitronic 60)		
	AISI 301	AISI 301	
	AISI 302	AISI 302	
AISI 303			AISI 303
AISI 303Se			AISI 303Se
	AISI 304	AISI 304	
	AISI 304L	AISI 304L	
	AISI 304H		AISI 304H
	XM-7	XM-7	
	AISI 304N	AISI 304N	
	AISI 305	AISI 305	
	AISI 308	AISI 308	
	AISI 309	AISI 309	
	AISI 309S	AISI 309S	
	AISI 309Cb	AISI 309Cb	
	AISI 310	AISI 310	
	AISI 310S	AISI 310S	
	AISI 311	AISI 311	
	AISI 314	AISI 314	
	AISI 315	AISI 315	
	AISI 316	AISI 316	

Type II	Type VI	Type VII (not recommended for new design)	Type VIII (not recommended for new design)
	AISI 316L	AISI 316L	
	AISI 316H		
	AISI 321	AISI 321	
	AISI 321H		
	AISI 329	AISI 329	
	AISI 347		
	AISI 347H		
AISI 347S			AISI 347S
AISI 347Se			AISI 347Se
AM350			AM350
AM355			AM355
Almar 362			Almar 362
AISI 403			AISI 403
AISI 405			AISI 405
AISI 409			AISI 409
AISI 410			AISI 410
AISI 414			AISI 414
AISI 416			AISI 416
AISI 416Se			AISI 416Se
AISI 420			AISI 420
AISI 429			AISI 429
		AISI 430	
AISI 430F			
AISI 430FSe			
AISI 431			AISI 431
AISI 434		AISI 434	
AISI 436		AISI 436	
AISI 440A			AISI 440A
AISI 440B			AISI 440B
AISI 440C			AISI 440C
AISI 440F			AISI 440F
AISI 440FSe			AISI 440FSe
AISI 442		AISI 442	
		AISI 446	
XM-27			

### 6.3 REQUIREMENTS FOR PICKLING

All pickling treatments shall be conducted within the guidelines of Huntington Alloy Booklet IAI-21 (Second Edition, 1987).

For pickling operations, the acidic solution shall be controlled such that copper flash will



not occur. General guidelines on the prevention and removal of copper flash are provided on page 36 in Huntington Alloy IAI-21 (Second Edition, 1987). The acidity levels shall be periodically monitored to ensure that the bath is at the proper initial strength. Make fresh solution when salts begin to crystallize on the sides of the tank. Analysis of the pickling solution shall be done periodically to verify that the metallic content is below 150 grams per liter (g/L). The solution must be discarded if the 150 g/L level is exceeded.

## **7.0 PROCESS QUALIFICATION**

None required.

## **8.0 PROCESS VERIFICATION**

The surface(s) being passivated or pickled shall be inspected for cleanliness prior to chemical treatment. The post-treatment verification details are in Section 8.1 or 8.2. The method used and result(s) shall be included in the facility or vendor certificate of conformance of this process.

### **8.1 VERIFICATION OF PASSIVATION PROCESS**

After the passivation process has been completed, the passivated components must exhibit chemically clean surfaces that show no etching, pitting, or frosting. A slight discoloration will be allowed if the subject surface passes the water break free test.

Each lot of parts shall be tested to verify corrosion resistance using one of the following four (4) test methods that are detailed in AMS 2700:

- (1) Water immersion test per AMS-STD-753 (Method 100)
- (2) High humidity test per section 3.2.1.1 of AMS 2700.
- (3) Salt spray testing for a minimum of 2 hours according to the procedures of ASTM B117.
- (4) Copper Sulfate test per AMS-STD-753 (Method 102)

### **8.2 VERIFICATION OF PICKLING PROCESS**

Verification that the pickled surface is free of embedded iron or other metal shall be accomplished using one or more of the following test methods:

- (1) Scanning electron microscopy with backscatter mode detection and windowless energy dispersive spectroscopy can be utilized. This is a very expensive technique and should be limited to small items of a limited quantity.

- (2) Potassium Ferricyanide-Nitric Acid Solution of AMS-STD-753 (Method 103) is an ultra-sensitive technique for small batches that can be used if inspectors have proper training in its use.
- (3) A salt spray test utilizing a solution of 1% sodium chloride for 12-24 hours can be used for large scale testing. Any free iron on the surface will appear as rust spots.

**9.0 TRAINING AND CERTIFICATION OF PERSONNEL**

The supervisor shall certify personnel performing passivation and pickling treatments of metallic materials. This training certification shall be written and records kept on file. For processes performed by an outside vendor, the development of an appropriate training program shall be the responsibility of the vendor.

**10.0 DEFINITIONS**

<b>Word</b>	<b>Meaning</b>
Corrosion-resistant steel	An alloyed steel that contains chromium in excess of 10.5 wt-%.
Passivation	The chemical treatment of a stainless steel alloy or iron based superalloy with a mild oxidizer, such as nitric acid, to remove free iron or other contaminants embedded in the surface, for example tool steel from machining. Passivation treatment also produces an oxide film with superior corrosion resistance to the oxide film that occurs naturally. Passivation is not generally effective in removing oxide scale or heat tint.
Pickling	The chemical treatment of metallic materials with an aqueous acid solution that results in the removal of surface oxides or scale formed during heating. This method also removes other foreign metals and other substances.
Water Break Free Test	A test to determine if a surface is chemically clean by the use of a distilled water rinse. If the surface is clean, the water will break and spread across the surface in a sheet-like manner. A contaminated surface will cause the water to form droplets and “bead.”