

Process Specification for Pickling, Etching, and Descaling of Metals

Engineering Directorate

Structural Engineering Division

June 2020



National Aeronautics and
Space Administration

Lyndon B. Johnson Space Center
Houston, Texas

Process Specification for Pickling, Etching, and Descaling of Metals

Prepared by: Signature on File 06/25/2020
 Daila Gonzalez
 Materials and Processes
 Branch/ES4
 Date

Reviewed by: Signature on File 06/26/2020
 Jay Bennett
 Materials and Processes
 Branch/ES4
 Date

Reviewed by: Signature on File 06/26/2020
 John Figert
 Materials and Processes
 Branch/ES4
 Date

Approved by: Signature on File 06/29/2020
 Brian Mayeaux
 Materials and Processes
 Branch/ES4
 Date

Process Specification for Pickling, Etching, and Descaling of Metals

REVISIONS		
VERSION	CHANGES	DATE
--	Original version	2/03/00
A	Changed Division name. Corrected errors in metal removal thickness before penetrant inspection in Sections 3.0 and 6.3.2. Re-titled and revised Section 6.1.	10/2004
B	Extensive Changes in sections 6, 7, and 8, associated with the processing of titanium.	12/2004
C	Retitled Section 4.0 (Applicable Documents) to (References) and updated document titles and numbers. Reformatted tables in Section 6.3 (Special Requirements),	2/2007
D	Updated document title to include descaling of metals; added statement to Section 1.0 Scope and Section 6.3.2 Etching for Penetrant Inspection stating electropolishing (PRC 5009) is the first choice prior to fluorescent penetrant inspection; added listing of alloys recommended for electropolishing; updated pickling, etching, and descaling description in Section 3.0 Usage; updated engineering drawing callouts in Section 3.0; added statement to Section 6.3.2 imposing process control coupons to verify minimum metal removal when etching prior to fluorescent dye penetrant inspection; added Figure 1 to Section 6.3.3 Descaling to Remove EDM Recast Layer as an example of a cross sectional view of EDM layers; added Figures 2-4 to demonstrate descaling effectivity to remove EDM recast layer; updated etching (Section 6.3.2) and descaling (Section 6.3.3 and 6.3.4) process requirements tables; deleted tensile coupon requirements to verify freedom from hydrogen embrittlement from Section 8.1 Additional Requirements for Titanium; added Figure 5 to Section 8.1 to demonstrate alpha case discoloration and removal post descaling on Ti 6-4 surface; updated Section 9.0 Training and Certification of Personnel; updated/added definitions to Section 10 Definitions; minor grammatical corrections.	06/2020

Verify correct version before use.

1.0 **SCOPE**

This document provides the standard requirements for etching, pickling, and descaling of metals by acids or alkali aqueous solutions.

It does not address the light pickling of Monel to remove embedded foreign metals, which is covered by the passivation PRC-5002.

Electropolishing is the first choice for pre-fluorescent penetrant inspection for the following alloys:

- Austenitic (300 series) Stainless Steels, including 302, 304, 316, 321, 347
- Precipitation Hardened Stainless Steels, including 15-5PH, 17-4PH, 17-7PH
- Miscellaneous Stainless Steels, including A-286, Nitronic 60
- Inconel Alloys, including Inconel 625, Inconel 718
- Multi-Phase Alloys, including MP35N, MP159
- Monel Alloys, including Monel 400, Monel R-405, Monel K-500
- Hastelloy, including C-276

Electropolishing may also be used for removing electrical discharge machining (EDM) layers and scale for the above alloys, depending on the application.

The process specification for electropolishing corrosion-resistant steel is PRC-5009.

2.0 **APPLICABILITY**

This specification shall be applicable whenever a pickling, etching, or descaling is invoked per Section 3.0, "Usage". It also applies whenever shop procedures that meet this specification are invoked by another process specification or shop instruction.

3.0 **USAGE**

This specification covers a wide variety of processes and metals, so the engineering drawing callout shall be specific enough to ensure that the proper work instructions are used.

"Pickling" is typically used to describe a process that removes light surface oxides and removes contamination from surfaces that are oxidized or corroded.

"Etching" is typically used to describe a process that removes parent metal in a controlled manner, sometimes to achieve specific surface textures.

"Descaling" is typically used to describe the removal of oxide scale or heavy heat tint caused by heat treating or welding.

The following drawing callouts are for standard JSC processes.

Stainless Steels**PICKLE TO REMOVE EDM RECAST LAYERS PER NASA/JSC PRC-5010.**

(including CRES 300 series (except for 303), 15-5PH, and A286)

ETCH FOR PENETRANT INSPECTION PER NASA/JSC PRC-5010.

(15-5PH only)

PICKLE TO REMOVE HEAT TINT PER NASA/JSC PRC-5010.

(including CRES 300 series (except for 303), 15-5PH, and A286)

Nickel Alloys**ETCH FOR PENETRANT INSPECTION PER NASA/JSC PRC-5010.**

(IN718 only)

Titanium (6Al4V)**DESCALE TO REMOVE EDM RECAST LAYERS PER NASA/JSC PRC-5010. PICKLE TO REMOVE HEAT TINT PER NASA/JSC PRC-5010.**Aluminum (Most common alloys)**DESCALE TO REMOVE EDM RECAST LAYERS PER NASA/JSC PRC-5010 ETCH FOR PENETRANT INSPECTION PER NASA/JSC PRC-5010.**

All new pickling, etching and descaling processes shall be qualified for the alloy at each facility performing the processing. Once a process is qualified, the qualification testing does not have to be repeated for that alloy if no essential process variables have changed and no processing problems are discovered. The details of the qualification shall be recorded in a Process Qualification Record (PQR) with any limitations of the process so noted. All PQRs shall be stored on the ES Library on DDMS.

Pickling, etching and descaling processes for other metals and other purposes can be qualified when necessary, but the designer should consult with the cognizant materials engineer to ensure that a procedure can be developed and successfully qualified before manufacturing needs the process. The remelt zone and heat affected areas caused by EDM (Figure 1, not drawn to scale) can affect fatigue, surface hardness, and other properties. An example is 6Al-4V titanium which can form cracks in brittle, oxygen-enriched layers, zones and phases (alpha case). If these zones and layers are present in EDM processed materials, production coupons may be required and these coupons shall be metallographically examined to ensure complete removal of these detrimental zones and layers.

A significant amount of material may be removed during pickling, etching, or descaling processing. In most cases, this will be compensated for during machining, but there may be some cases for which dimensional control may be difficult to maintain. The typical maximum metal removal after etching aluminum and titanium for penetrant inspection is .0006 inches and for stainless and nickel-based alloys are .0004 inches. For parts with tight dimensional control, the design engineer may choose to add specific drawing notes and/or coordinate closely with M&P and manufacturing.

A frosted appearance is common on stainless steel and nickel based alloys as a result of etching.

4.0 REFERENCES

The following documents were used in developing this specification:

SOP-007.1 *Preparation and Revision of Process Specifications*

JPR 8500.4 *Engineering Drawing System Manual*

The following documents are called out as an extension of the requirements given in this specification. All documents listed are assumed to be the current revision unless a specific revision is listed.

ASTM A380 *Standard Practice for Cleaning, Descaling, and Passivation of Stainless Steel Parts, Equipment, and Systems*

ASTM B600 *Descaling and Cleaning Titanium and Titanium Alloy Surfaces.*

NASA/JSC TI-5000-01 *Metal Finishing Technicians, Training Instruction for*

5.0 MATERIAL REQUIREMENTS

None

6.0 PROCESS REQUIREMENTS

6.1 WORK INSTRUCTIONS

All work shall be performed 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 consist of Detailed Process Instructions (DPI's).

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

6.2 GENERAL REQUIREMENTS

Pickling, etching, and descaling processes shall be controlled to effectively remove oxidation or damage on metal surfaces while minimizing any deleterious effect on the surface. The surface finish shall be smooth, without pitting or attack of grain boundaries. For susceptible metals, the process should not have a negative effect on the mechanical properties of the metal.

General requirements for bath compositions are listed in the sections for each special process. The specific compositions and operating parameters of each process shall be detailed in the work instruction and summarized in the PQR. Work instructions for stainless steels and alloys shall follow the standard practices of ASTM A 380. Work instructions for titanium alloys shall be in compliance with the standard practices of ASTM B600.

Free machining stainless steels shall not be pickled, etched, or descaled.

While removing the EDM layers on titanium parts, the surface can become resistant to chemical reaction. If this occurs, the part shall be lightly blasted with abrasives to reactivate the surface and the part returned to the processing solution. Maximum abrasive sizes are #10 for glass bead and 220 grit for alumina and silicon carbide.

High temperature caustic solutions may be used to precondition titanium prior to acid treatments. This treatment may remove all visible scale or oxidation, but a flash pickle afterwards is required at a minimum.

Oxygen and nitrogen diffuse into the titanium at high temperatures and form a brittle phase called alpha case, which can readily fracture and subsequently initiate several types of service or test failures. The visual inspection of titanium parts after pickling or descaling shall include verification that alpha case has been removed. Visual inspector shall meet the training requirements in section 9.0.

6.3 SPECIAL REQUIREMENTS

6.3.1 ETCHING FOR SUBSEQUENT METAL FINISHING

Etching processes used as preparation of metals surfaces for subsequent metal finishing shall use process solutions controlled and qualified by the subsequent metal finishing process and its work instructions.

6.3.2 ETCHING PRIOR TO FLOURESCENT PENETRANT INSPECTION

When metal parts are etched for penetrant inspection, a minimum layer of metal must be removed to meet NDE reliability requirements. For aluminum and titanium, the minimum metal to be removed is 0.0004 inches. For stainless steel and nickel-based alloys, the minimum metal to be removed is 0.0002 inches. Process control coupons shall be used to verify minimum metal removed.

Table 1 provides etching process requirements prior to fluorescent penetrant inspection.

Alloy	Process Solution*	Temperature	Notes
15-5PH	50 ml HCl, 10g copper sulfate, 50 ml water (Marble's Reagent)	Ambient	immerse or swab until contrast is obtained
IN718	50 ml sat. aqueous copper sulfate, 50 ml HCl	Ambient	immerse or swab until contrast is obtained
Monel 400 and K500	Concentrated nitric acid (colorless)	Ambient	swab or immerse for 3-5 minutes
Titanium 6-4	30 ml nitric acid 3 ml HF 67 ml water	Ambient	swab or immerse for 3-5 minutes
Aluminum	Etch: 10 g NaOH, 90 ml water Desmut: 50% nitric acid (2000 & 7000 Series)	Ambient	swab or immerse for 5-15 minutes immerse (under a minute)

Table 1: Etching Process Requirements Prior to Fluorescent Penetrant Inspection

*Any quantity of etch solution may be prepared provided the ratios in the table are maintained.

6.3.3 DESCALING TO REMOVE EDM RECAST LAYER

Qualification coupons shall be processed that verify that the recast layer will be completely removed. The EDM cutting schedule used in qualification shall have a cutting current (usually denoted as IP) that is the same or higher than the production parts.

Figure 1 shows a cross-sectional view of the EDM layers. The HAZ plus the Remelt Zone is often only about 0.0001 inches. The oxidized zone is often about 0.0005 inches for stainless and about 0.0020 inches for titanium alloys. The oxidized zone contains gaps, voids and laps between highly oxidized particles that are partially fused to the Remelt Zone layer. This porous, oxidized zone can trap liquids during subsequent processing if it is not removed. In addition, this oxidized layer contains brass particles from the EDM wire.

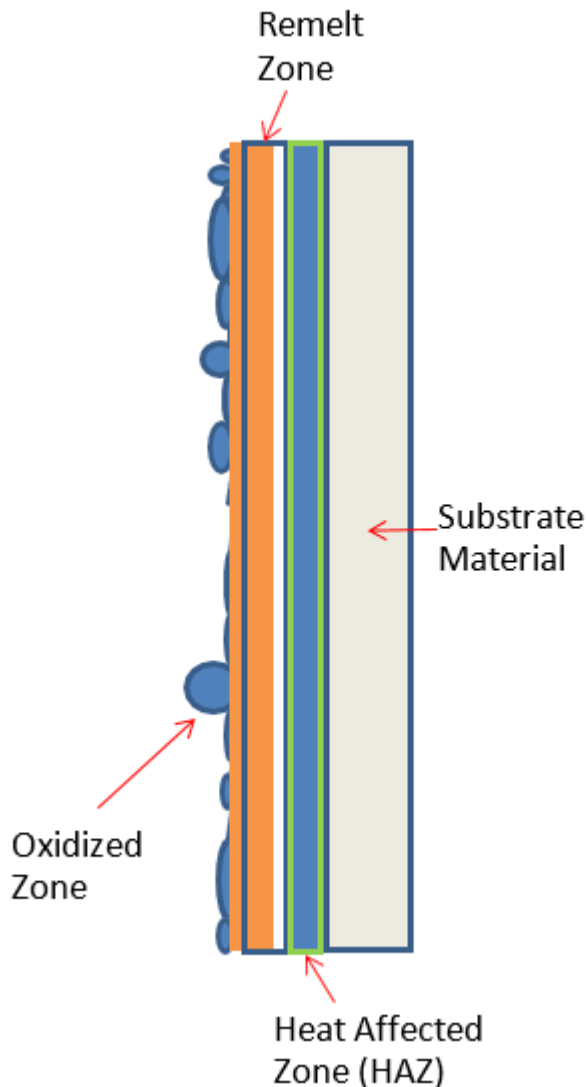


Figure 1: Electrical Discharge Machining (EDM) Layers

Table 2 provides descaling process requirements to remove EDM recast layer.

Alloy	Process Solution	Temperature	Notes
Stainless Steels CRES 300 series (except 303), 15- 5PH, A286	nitric HF acid	Ambient	Concentrations per ASTM A380
Titanium	Pickle: nitric acid 20-30ml HF acid 2.0-3.0ml balance water, to make up 100ml	Ambient	10:1 nitric/HF ratio or greater

Aluminum	phosphoric acid 80% acetic acid 15% nitric acid 5%	212°F	
----------	--	-------	--

Table 2: Descaling Process Requirements to Remove EDM Recast Layer

Figures 2-4 depict EDM surfaces prior to and post descaling on Al 6061 T6, CRES 304, and Ti-6-4.

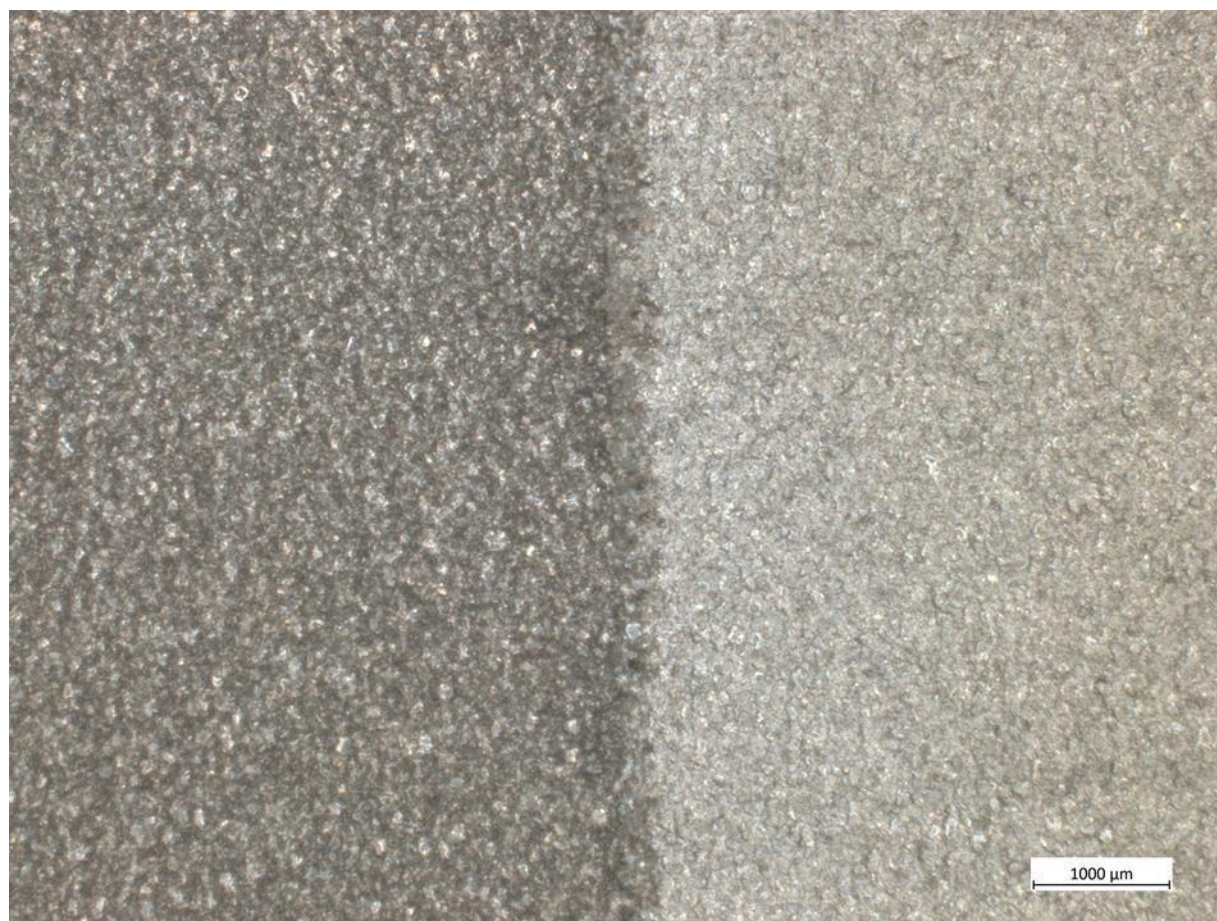


Figure 2: Al 6061 T6 wire EDM surface (left), descaled surface (right).



Figure 3: CRES 304 wire EDM surface (left), descaled surface (right).



Figure 3: Ti 6-4 wire EDM surface (left), descaled surface (right).

6.3.4 DESCALING TO REMOVE SCALE AND HEAT TINT

When parts are to be descaled to remove scale or heat tint, the metal parts shall be immersed in the process bath for as long as necessary to remove the oxides, up to the maximum time allowed by the work instructions. Usually the dimensional changes are uncontrolled. Qualification coupons shall be processed at the maximum time allowed by the work instructions.

Table 3 provides descaling process requirements to remove scale and heat tint.

Table 3: Descaling Process Requirements to Remove Scale and Heat Tint

Alloy	Process Solution	Temperature	Notes
Stainless Steels CRES 300 series (except 303), 15- 5PH, A286	Nitric Acid HF Acid	Ambient	Concentrations per ASTM A380
Monel	FeCl3 HCl	Ambient	
Titanium	Pretreat: NaOH 40-50g water 100ml Pickle: Nitric Acid 20-30ml HF Acid 2.0-3.0ml balance water, to make up 100ml	240-280F Ambient	Pretreat is optional for heat tint. 10:1 Nitric/HF ratio or greater
Aluminum	NaOH 5%	Ambient	

7.0 PROCESS QUALIFICATION

The work instructions for each process shall be qualified on non-flight hardware or coupons before the flight hardware is subjected to the process. The processes shall be individually qualified for each alloy and are valid only for a specific alloy or processing facility. Each process shall be evaluated with qualification coupons to determine adherence to the general requirements for pickling, etching, or descaling and for any special requirements. Once a process is qualified, the testing does not have to be repeated if no essential process variables have changed and no processing problems are discovered.

Process qualification results shall be recorded on a PQR and signed by the metal finishing supervisor and an engineer from ES4, Materials and Processes Branch. The PQR shall

Verify correct version before use.

include, as a minimum, the alloy processed, the process solution compositions, the essential processing variables, and the results of the examinations.

Approved PQR's shall be stored on the ES Home page.

8.0 PROCESS VERIFICATION

Each part shall be inspected after processing to verify that the surface finish is smooth, free from oxide, scale, pitting or detrimental grain boundary etching.

Process Control coupons shall be used to determine the etch rate when the surface will be etched before NDE inspection. These coupons shall be made of the same alloy as the part being processed. The verification coupons shall be thin and flat to maximize the differential thickness measurement. Verification coupons shall be etched and the thickness changed determined on the day of processing before the production work is processed. The bath's etch rate shall be determined and the etch time adjusted to guarantee the minimum metal removal is achieved on that day.

Records of self-inspection and MIPs shall be kept as quality assurance records.

8.1 ADDITIONAL REQUIREMENTS FOR TITANIUM

The processing solutions for titanium in this specification use a 10:1 or greater nitric acid/hydrofluoric acid ratio per ASTM B600. The use of this ratio will limit hydrogen pick-up in titanium alloys from nitric acid/hydrofluoric acid pickling (or chemical milling).

When the 10:1 ratio is not maintained, hydrogen analysis of titanium alloy process samples shall be performed.

- Representative samples must come from the same mill lot of titanium alloy. These samples shall be processed with the production parts and sent to a laboratory for total hydrogen analysis.
- The results shall be reviewed by M&P.

Hydrogen in titanium and titanium alloys can be measured accurately by several laboratory methods including ASTM E1447. It is very important that testing be done in a well-equipped laboratory by well-trained personal with known ppm hydrogen calibration standards.

Titanium parts shall be visually inspected to verify that alpha case has been removed (Figure 5).



Figure 4: Ti 6-4 discoloration (alpha case) post heat treating in air at 1100F for 2hrs (left). Ti 6-4 surface post alpha case removal using 30% nitric acid, 3% hydrofluoric acid and 67% H2O solution (right).

9.0 TRAINING AND CERTIFICATION OF PERSONNEL

Training requirements for metal finishing technicians shall be written to cover the requirements of this specification. Trainee shall be certified following the successful working under the supervision of the metal finishing facility manager. Training and certification records shall be kept.

Inspectors shall be trained and certified to inspect for EDM and alpha case removal by M&P.

For work performed at JSC facilities, these requirements shall be satisfied by the training and certification of personnel per TI-5000-01 Training Instruction.

For work performed outside JSC facilities, engineering shall ensure technicians and inspectors are trained. Training shall be documented.

10.0 DEFINITIONS

Descaling A chemical or mechanical process for removing the thick layer of oxides formed on some metals at elevated temperatures.

EDM	A metal fabrication process whereby a desired shape is obtained by using electrical discharges (sparks). Material is removed from the work piece by a series of rapidly recurring current discharges between two electrodes, separated by a dielectric liquid and subject to an electric voltage
Electropolishing	A method of polishing metal surfaces by applying an electric current through an electrolytic bath in a process that is the reverse of plating. The metal to be polished is made the anode in an electric circuit. Anodic dissolution of the protuberant burrs and sharp edges occurs at a faster rate than over the flat surfaces and crevices, possibly because of locally higher current densities. The result produces an exceedingly flat, smooth, brilliant surface.
Etching	The localized chemical or electrochemical attack of a metal surface. Controlled etching of metals can be used to reveal structural details or open tight cracks for fluorescent penetrant inspection. By contrast, uncontrolled or excessive etching of metals by an acid solution can cause damage to the surface integrity.
Mandatory Inspection Point (MIP)	A second-party inspection process designated during a manufacturing operation.
Passivation	A non-electrolytic finishing process that makes stainless steel more rust-resistant. The passivation process typically uses nitric or citric acid to remove free iron from the surface. This results an inert, thin protective oxide layer that is less likely to chemically react with air and cause corrosion.
Pickling	The chemical removal of surface oxides by immersion in an aqueous acid solution.