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400 COMMONWEALTH DRIVE, WARRENDALE, PA 15096

AEROSPACE RECOMMENDED PRACTICE

ARP 1843

Issued 7-1-84
Revised

SURFACE PREPARATION FOR STRUCTURAL ADHESIVE BONDING
Titanium Alloy Parts

1. SCOPE:

1.1 This recommended practice establishes the procedures and requirements for the cleaning of titanium alloy parts prior to adhesive bonding operations.

1.2 The surface treatments described herein have proven effective for producing a suitable surface for application of adhesive primers, films, or pastes prior to bonding operations.

2. APPLICABLE DOCUMENTS:

The following publications form a part of this recommended practice to the extent specified herein. The latest issue of Aerospace Material Specifications (AMS) shall apply. The applicable issue of other documents shall be as specified in AMS 2350.

2.1 SAE Publications: Available from SAE, 400 Commonwealth Drive, Warrendale, PA 15096.

AMS 2350 - Standards and Test Methods
AMS 3803 - Wipes, Cotton, Loosely Woven
AMS 4911 - Titanium Alloy Sheet, Strip, and Plate, 6Al - 4V, Annealed

2.2 ASTM Publications: Available from American Society for Testing and Materials, 1916 Race Street, Philadelphia, PA 19103.

ASTM D1002 - Strength Properties of Adhesives in Shear by Tension Loading (Metal-to-Metal)

2.3 U.S. Government Publications: Available from Commanding Officer, Naval Publications and Forms Center, 5801 Tabor Avenue, Philadelphia, PA 19120.

Printed in U.S.A.

2.3.1 Federal Specifications:

O-C-303 - Chromium Trioxide, Technical
O-N-350 - Nitric Acid, Technical
O-S-809 - Sulfuric Acid, Technical
O-T-620 - Trichloroethane 1,1,1, Technical, Inhibited, (Methyl Chloroform)
TT-M-261 - Methyl Ethyl Ketone, Technical
UU-P-268 - Paper, Kraft, Wrapping
CCC-C-640 - Cloth, Cheesecloth, Cotton, Bleached and Unbleached

2.3.2 Military Specifications:

MIL-P-17667 - Paper, Wrapping, Chemically Neutral (Non Corrosive)
MIL-T-81533 - Trichloroethane 1,1,1 (Methyl Chloroform), Inhibited, Vapor Degreasing
MIL-C-87962 - Cloths, Cleaning, for Aircraft Fuel Tanks

3. PROCESSING MATERIALS, SOLUTIONS, AND EQUIPMENT:

3.1 **Deionized Water:** The total ionizable solids should not exceed 100 parts per million (ppm) when measured on an in-line conductance bridge calibrated for sodium chloride.

3.2 Etch Solution:

3.2.1 **Solution Preparation:** Prepare the etch solution as follows, maintaining the solution below 38°C (100°F).

Chemical	Concentration
Concentrate Etch (See 6.2.1.1)	10% by volume
Nitric Acid (See 6.2.1.2)	21% by volume
Chromic Acid (See 6.2.1.3)	4 oz ± 0.1 per gal of solution (30 g ± 1 per L of solution)
Deionized Water	remainder

3.2.1.1 Fill tank half full of deionized water.

3.2.1.2 Add chromic acid slowly with agitation.

3.2.1.3 Add nitric acid slowly with agitation.

3.2.1.4 Add the etch concentrate (See 6.2.1.1) slowly with agitation.

3.2.1.5 Add the remainder of the deionized water. Slowly agitate.

3.2.2 Etch Solution Control: Control the etch solution as follows:

Parameter	Control Limits
Etch rate	
With Alkaline Cleaning	T.B.D.*
Without Alkaline Cleaning	T.B.D.*
CrO_3	3.5 to 4.2 oz/gal (26.2 to 31.5 g/L)
Free Nitric Acid	24 to 29 oz/gal (180 to 217 g/L)
Etch Concentrate	T.B.D.*

*To be determined after accumulation of data for 6 months or less, and a statistical evaluation thereof.

3.2.2.1 Etch Rate:

3.2.2.1.1 For each type of etch rate, use two deburred specimens of AMS 4911 titanium alloy sheet machined to a 125 microin. (3.2 μm) or smoother surface texture, and not less than 9 sq in. (0.006 m^2) area per side, and any constant thickness between 0.020 and 0.060 in. (0.50 and 1.50 mm).

3.2.2.1.2 Solvent clean, liquid hone, and alkaline clean as in 3.7.1 through 3.7.3.

3.2.2.1.2.1 Alkaline clean for 25 min. ± 1 only when determining etch rate with alkaline cleaning (See 3.2.2).

3.2.2.1.3 Air dry for not less than 1 hr or to constant weight.

3.2.2.1.4 Weigh specimen on an analytical balance to the nearest 0.0001 gram.

3.2.2.1.5 Determine average panel thickness with micrometer to the nearest 0.0001 in. (0.002 mm) based on not less than 5 readings.

3.2.2.1.6 Immerse specimen in the etch solution of 3.2.1 for 15 min. ± 5.

3.2.2.1.7 Rinse as in 3.7.4.2 and 3.7.4.3 and air dry for not less than 1 hour. Reweigh specimens and determine weight loss for each to the nearest 0.0001 gram.

3.2.2.1.8 Calculate etch rate as follows:

$$\frac{2 \text{ x weight loss x thickness in in. (mm)}}{\text{original weight}} = \text{in. (mm)/surface per hr}$$

3.2.2.2 Determination of CrO_3 Concentration:

3.2.2.2.1 Take a 10.0 mL sample of the etch solution of 3.2.1 and dilute to 100.0 mL in a volumetric flask.

3.2.2.2.2 Transfer 4.0 mL of the above solution to a flask and add 100 mL of distilled water. Then add 10 mL of 50% sulfuric acid, H_2SO_4 (See 6.2.3).

3.2.2.2.3 To the above solution add 10 mL of 20% potassium iodide (KI) (See 6.2.4) solution, followed by 10 mL of soluble starch solution (See 6.2.5).

3.2.2.2.4 Titrate the above solution with 0.1 N sodium thiosulfate (See 6.2.12) solution to the end point.

3.2.2.2.5 Calculate the CrO_3 as follows:

$$CrO_3 \text{ in oz per gal} = \frac{A}{7.489} \qquad (CrO_3 \text{ in g/L} = A)$$

where: A = mL of 0.1 N sodium thiosulfate to titrate 3.2.2.2.4.

3.2.2.3 Determination of Free Nitric Acid in Etch Solution:

3.2.2.3.1 Dilute 5.0 mL of solution with distilled water to 100.0 mL in a volumetric flask.

3.2.2.3.2 Transfer 20.0 mL of the above solution to a volumetric flask and dilute with distilled water to 100.0 millilitres.

3.2.2.3.3 Transfer 10.0 mL of the above solution to a 250 mL Erlenmeyer flask and add 25.0 mL of 0.2 N ferrous ammonium sulfate (See 6.2.6) solution.

3.2.2.3.3.1 To prepare a 0.2 N ferrous ammonium sulfate solution:

add 100 mL concentrated sulfuric acid to 600 mL distilled water;
add 78.4 g of $Fe(NH_4)_2(SO_4)_2 \cdot 6H_2O$;
mix to dissolve;
dilute to total of 1 L with distilled water.

3.2.2.3.4 To the solution of 3.2.2.3.3 add 20 mL of concentrated sulfuric acid (See 6.2.3), and boil the solution approximately 5 min. until the color is a clear green-yellow.

3.2.2.3.5 Cool flask under tap water, add 50 mL of distilled water, and cool again.

3.2.2.3.6 Add 3 drops of 0.025 M Ferroin (See 6.2.8) indicator and titrate with standardized 0.200 N potassium dichromate (See 6.2.7) to a bright green.

3.2.2.3.6.1 To prepare 0.200 N potassium dichromate ($K_2Cr_2O_7$), add 9.806 g potassium dichromate to distilled water to yield a volume of 1 litre.

3.2.2.3.7 Run a blank through the above procedure, substituting 10 mL of distilled water for the solution aliquot in 3.2.2.3.3. Run a blank daily.

3.2.2.3.8 Calculate free nitric acid as follows:

$$\frac{16.8(C - D)}{3} = \text{oz per gal}; \quad \frac{125.8(C - D)}{3} = \text{g/L}$$

where C = mL of 0.200 N potassium dichromate to titrate blank in 3.2.2.3.7.
D = mL of 0.200 N potassium dichromate to titrate solution in 3.2.2.3.6.

3.2.2.4 Determination of the Etch Concentrate Content:

3.2.2.4.1 Transfer 1 mL of the etch tank solution (See 3.2.1) to a volumetric flask, using a polyethylene pipette. Dilute to 200 mL with distilled water.

3.2.2.4.2 Transfer 25 mL of the above solution to a 150 mL beaker and add 25 mL of buffer solution (See 6.2.9).

3.2.2.4.3 Prepare 0.01, 0.004, and 0.001 molar fluoride solutions by diluting 10, 4, and 1 mL of 1 molar fluoride stock solution to 100.0 mL with distilled water.

3.2.2.4.4 Transfer 25 mL of the 0.01 molar fluoride solution to a 150 mL beaker and add 25 mL of the buffer solution. Repeat this procedure on the 0.004 and 0.001 molar fluoride solutions.

3.2.2.4.5 After adjusting the zero on the pH/mV meter (having an expanded range of ± 200 mV readable to 0.1 mV), attach an instrument fluoride electrode (See 6.3.1) and a reference electrode (See 6.3.2) and measure the millivolt potential of the three standard fluoride solutions.

3.2.2.4.6 Prepare a working curve by plotting millivolts versus molar fluoride concentration on semilogarithmic graph paper.

3.2.2.4.7 Measure the millivolt potential of the unknown prepared in 3.2.2.4.2. Use the working curve to obtain the fluoride ion concentration. Use the fluoride ion concentration to calculate the etch concentrate content as follows:

Etch Concentrate, % by volume = Fluoride ion concentration determined from the curve.

3.3 Alkaline Cleaning Solution:

3.3.1 Solution Preparation: Prepare the alkaline cleaning solution as follows, maintaining the solution at 93° to 100°C (200° to 212°F):

Chemical	Concentration
Alkaline Cleaner (See 6.2.2)	2.5 to 3.0 lb per gal (300 to 360 g/L)
Tap Water	Remainder

3.3.2 Solution Control by Free Alkalinity Analysis:

3.3.2.1 Transfer a cooled 25 mL sample to a 250 mL volumetric flask and dilute to the mark.

3.3.2.2 Pipette 20 mL of the diluted sample to a beaker.

3.3.2.3 Titrate sample with 1 N sulfuric acid to pH 11.4. If possible use a pH meter that compensates for temperature changes of the sample.

If a pH meter is unavailable, use a suitable indicator (See 6.2.13) dissolved in concentration of 0.01% in water and titrate until color changes from red to yellow.

3.3.2.4 Record titration and calculate free alkalinity concentration:

oz per gal = 4.44 E (g/L = 33.25 E)

where: E = mL of 1 N H_2SO_4 to titrate solution in 3.3.2.3.

3.3.2.5 Control concentration on the basis of the free alkalinity concentration of 2.5 to 3.0 lb per gal (300 to 360 g/L) of solution.

3.3.2.5.1 The useful life of the alkaline cleaner is shortened by extended operation at 93° to 100°C (200° to 212°F). When the tank is not in use, the temperature should be lowered to below 80°C (180°F) to extend solution life.

3.4 Liquid Honing Slurry:

3.4.1 Aluminum Oxide Slurry Preparation:

3.4.1.1 Fill the clean hopper of a liquid honing unit approximately one-half full with clean water.

3.4.1.2 Add sufficient 220 grit (70 μm) aluminum oxide abrasive to maintain not less than 35% by volume of settled solids.

3.4.1.3 Agitate thoroughly to keep the abrasive material in suspension. When thoroughly mixed, fill the hopper to the operation level with clean water and mix again.

3.4.2 Slurry Control: The percent solids in slurry may be determined as follows:

3.4.2.1 Sample the slurry directly from the nozzle.

3.4.2.2 Thoroughly mix the slurry prior to filling a 100 mL, graduated centrifuge tube to the 100 mL mark.

3.4.2.3 Allow the slurry to settle for not less than 30 minutes.

3.4.2.4 The volume of settled solids should be not less than 35 mL, read directly on the centrifuge tube.

3.4.2.5 When solids content drops below 35%, or when a slow blast rate indicates a low solids content, add abrasive material as required. Either method is acceptable for slurry control.

3.4.2.6 To maintain cleanliness of the parts, the slurry should be periodically discarded and remixed. For equipment that is in daily use the slurry should be remixed at least once every third working day. For equipment used only occasionally, the slurry should be remixed when the accumulated operating time exceeds 50 hours.

3.5 Equipment and Facilities:

3.5.1 Drying Equipment: Use a circulating-air oven or open-top dryer for drying parts, designed to prevent contact of parts with combustion gases or products (if present). Equipment shall be capable of maintaining temperature between 38° and 80°C (100° and 175°F) and be equipped with a temperature recorder accurate to ±3°C (±5°F) of actual temperature. Makeup air shall be from the cleaning room, or equivalent sources.

3.5.2 Liquid Honing Equipment: Cabinets and accessories should be fabricated from corrosion-resistant steel and be supplied with pressure controlling devices.

3.5.3 Compressed Air Supply: Compressed air shall be dry, filtered, and oil6free.

3.5.4 Tank Heating Equipment: Heating coils should be equipped with automatic temperature controls.

3.5.5 Etch Solution Tank: Tank, lines, and continuous circulating pump should be lined with suitable polyvinyl chloride (PVC) coating.

3.5.6 Press or Autoclave: Should be capable of maintaining pressure and temperature as required by applicable adhesive bonding specifications.

3.5.7 Rinse Tanks:

3.5.7.1 Separate rinse tanks should be used for alkaline cleaning and for etch solution processing.

3.5.7.2 The alkaline cleaner rinse tank should be air agitated. The etch solution rinse tank should be agitated by either pump circulation, or by arranging the fresh water inlet flow to provide mild agitation over the entire tank. The responsible engineering organization should approve any such inlet design.

3.5.8 Racks and Fixtures: Use unalloyed titanium for racks, fixtures, and wires to hold parts during cleaning. Hollow stock should be avoided in those portions of the rack which will be immersed in the cleaning solution. Racks should be designed to prevent solution entrapment.

3.5.8.1 An acceptable alternate material for rack construction is polypropylene (See 6.1.4).

3.5.9 pH/Millivolt Meter: The pH/mV meter used in 3.2.2.4.5 should have an expanded range of ±200 mV, readable to 0.1 mV (See 6.3.1).

3.5.10 Cleaning and Processing Facility:

3.5.10.1 Cleaning, priming and drying operations should be performed in areas free of fume producing machinery, combustion engines, or other chemical processing solutions not required for this cleaning operation.

3.5.10.2 Tanks for processing titanium alloy and aluminum alloy parts prior to adhesive bonding should be used exclusively for this purpose. They may be located in the same enclosure; however, the two processing lines shall be completely separate with no common tanks. A separate hoist should be used for each line. However, in event of an equipment failure of either hoist, special provisions for emergency transfer of a hoist from one process line to another are acceptable. No direct crossovers between adjacent cleaning line tanks are permissible at any time. The honeycomb core cutting operation should be isolated from the cleaning tanks to prevent contamination of the tanks with particulate aluminum. Fumes from sources outside the area where cleaning, drying, and priming operations are performed should be minimized by either of the following methods:

3.5.10.2.1 Prohibiting use of internal combustion engines, machining operations, and nonrelated chemical processing equipment in the same building where cleaning, priming, and drying operations are performed.

3.5.10.2.2 Performing these operations in areas which are completely enclosed or isolated from the surrounding shop area. Sufficient clean outside air should be introduced to maintain a slight positive pressure. Doors and windows should be kept closed.

3.5.10.3 Specific facilities and operating procedures for minimizing fumes from internal combustion engines, machining operations, and non-related chemical processing equipment in areas where cleaning, priming, and drying operations are performed should be approved in writing by the responsible engineering organization. Changes in facilities or operating procedures, or both, which could affect the amount of fumes entering these areas may be made only with the written approval of the responsible engineering organization.

3.6 Processing Requirements:

3.6.1 Cleaned parts should be primed within 8 hr of completion of drying. After 8 hr, parts shall be recleaned in accordance with 3.7.

3.6.2 The rinse tank for the alkaline cleaner should have a conductance of not more than 6,500 μmhos/mm (6,500 μsiemens/mm) when the parts are placed in the tank. A tap water spray may be used while extracting parts from the rinse tank in lieu of this requirement. The etch rinse tank should be controlled at not more than 800 μmhos/cm (800 μsiemens/cm).

3.6.3 The liquid honing machine should be restricted to titanium alloy parts only and should be used only to prepare parts for adhesive bonding.

3.6.3.1 Small corrosion-resistant steel parts, 1 sq ft (0.1 m^2) and under in surface area, to be adhesive bonded may be honed in this equipment.

3.6.4 All personnel should wear clean, white gloves (See 6.1.1) when handling cleaned parts or racking liquid honed parts.

3.6.5 Only titanium alloy parts and racks and polypropylene racks (See 6.1.4) should be immersed in the etch solution (See 3.2.1).

3.6.6 The cleaning control coupons should utilize the alloy, primer, and film adhesive currently in production use for the parts they represent.

3.6.7 Cleaned parts shall be uniform in appearance.

3.6.7.1 Variations between areas of different thicknesses are acceptable. Welded assemblies may exhibit variations on different component parts. Weldments may be darkened. Material may show variations due to microstructure (marbling). Discoloration due to build-up of black powder-like material (smut) is acceptable. Areas which will not be bonded may show variations other than the above and be acceptable.

3.6.8 Sanding and trimming of adhesive or any other particulate producing process should not be performed in areas where cleaning and drying operations take place.

3.6.9 Times of cleaning operations for parts and test panels should be recorded on the work order.

3.6.10 Persons who clean parts prior to adhesive bonding should be qualified.

3.6.11 Chemical additions to the processing tanks may be made only with the approval of the responsible quality assurance organization or other controlling agency.

3.6.12 Parts may be recleaned three times only.

3.7 Procedures:

3.7.1 Solvent Cleaning: Solvent clean parts to remove excess grease and oil contamination by applying methyl ethyl ketone (MEK) (See 6.2.10) to the parts with a brush or cloth (See 6.1.2) saturated with the solvent; (1,1,1 trichloroethane (See 6.2.11) may be used in place of MEK if the part is to be subsequently alkaline cleaned). Use clean solvent and wiping materials. Do not use a container contaminated with primer. Store solvent wiped parts in a dust-free area or wrap in clean wax-free kraft paper (See 6.1.3).

3.7.2 Liquid Honing: Liquid hone the bond area of the parts and all lap shear test panels at the same time as the parts they represent, as follows:

3.7.2.1 Position the nozzle at an angle not greater than 60 deg with the part surface. Keep the nozzle not less than 2 in. (50 mm) from the surface being liquid honed.

3.7.2.2 Execute smooth and continuous passes with the nozzle to prevent uneven erosion of the surfaces.

3.7.2.3 Rinse to remove abrasive residue.

3.7.3 Alkaline Cleaning:

3.7.3.1 If alkaline cleaning will not take place within 2 hr of liquid honing, wrap the parts in clean wax-free kraft paper.

3.7.3.2 Rack all lap shear control specimens with the parts they represent.

3.7.3.3 Clean parts and control specimens in the alkaline cleaning solution (3.3) for 20 to 30 min. and rinse in air agitated tap water. Rinse water conductance should be not more than 6,500 μmhos/mm (6,500 μsiemens/mm) prior to immersion of parts in tank or parts may be spray rinsed with tap water for 2 to 4 min. after removal from rinse tank. Continue processing immediately after allowing parts to drain sufficiently to prevent excessive drag-over to the etch solution tank.

3.7.4 Conducting the Etching:

3.7.4.1 Immerse parts and lap shear control specimens in the etch solution (3.2) for 15 to 20 minutes.

3.7.4.2 Rinse parts thoroughly in room temperature tap water (use etch rinse tank only) for 2 to 4 minutes. It is acceptable to allow parts to remain in this rinse tank until an inspector arrives to conduct the water-break test of 4.3.1.

3.7.4.3 Spray rinse parts with room temperature deionized water for 2 to 4 minutes. Inspect for water breaks in accordance with 4.3.1.

3.7.4.4 Dry parts at 38° to 80°C (100° to 175°F) for not less than 30 min. in oven or open-top dryer.

3.7.4.5 After drying, store parts in a dust-free area.

3.7.4.5.1 If priming will not occur within 4 hr after completion of the drying cycle, wrap parts in clean wax-free kraft paper.

3.7.4.5.2 If parts are not primed within 8 hr after completion of the drying cycle, including any time they are wrapped in kraft paper, reclean in accordance with 3.7. Parts may be recleaned three times only.

3.8 Safety:

3.8.1 Wear rubber gloves, rubber apron, and chemical face mask or goggles when handling chemicals or solutions.

3.8.2 When diluting or preparing acid or alkaline solutions, always add acid or alkaline cleaner to water; never the reverse. Pour acid or alkaline cleaner slowly, avoiding splashing. Stir at intervals during mixing. Do not add acid or alkaline cleaner while stirring.

3.8.3 Avoid inhaling acid, alkaline, or solvent vapors; if enough vapor is inhaled to cause discomfort, secure first aid.

3.8.4 Seal acid containers immediately after using to prevent accidental spilling or escape of fumes.

3.8.5 If acid or alkaline materials contact the body or eyes, flush affected area immediately with large amounts of water. If concentrated acid contacts the body, remove contaminated clothing and immediately take a shower. If eyes are involved, rinse continuously with warm water for not less than 15 minutes. Secure first aid in all cases.

3.8.6 Correctly label and maintain the identity of all containers as to their contents.

3.8.7 Isolate acids and alkalies from incompatible materials.

3.8.8 The precautions for storing, using, and handling of both flammable and nonflammable solvents should conform to local safety regulations.

3.8.9 Approved eye protection, protective clothing, and respirator should be used during sanding and trimming processes. Where possible, a vacuum dust-collecting system should be employed.

4. QUALITY ASSURANCE PROVISIONS:

4.1 Surveillance: The responsible quality assurance organization should maintain surveillance to ensure adequate compliance with these requirements.

4.2 Inspection Records: Inspection records should be kept complete and should contain all data necessary to determine compliance with the requirements of this recommended practice.

4.3 Inspection:

4.3.1 During fabrication, visually inspect parts for water-break-free surface following the deionized water rinse. Parts exhibiting water-breaks should be recleaned. Parts may be recleaned three times only. A water-break-free surface will maintain a continuous film of water for not less than 30 sec after rinsing.

4.3.2 Visually inspect parts after drying for uniformity of appearance (See 3.6.7).

4.3.3 Ensure that cleaning operations are performed only by qualified personnel. Qualified personnel are those who have demonstrated by written or practical proficiency tests, or both, that they possess the skills and job knowledge necessary to ensure acceptable workmanship.

4.4 Process Control: Solutions should be prepared and maintained within the limits specified herein.

4.4.1 Alkaline Cleaner Solution: Analyze once a week. This frequency may be decreased to once every two weeks if the test results of five consecutive tests are within the specification limits.

4.4.2 Etch Solution: Analyze at least once every three days of continuous operation, or before each use if not used within the last three days.

4.4.2.1 The chromate, nitrate, and etch concentrate analysis frequencies may be decreased to once every week of continuous operation if the test results of five consecutive tests of a given property are within the limits specified herein.

4.4.2.2 The etch rate analysis frequency may not be decreased.