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**Structural Examination of Titanium Alloys
Etch-Anodize Inspection Procedure**

1. SCOPE:

1.1 Purpose:

This specification covers a procedure for revealing the macrostructure and microstructure of selected titanium alloys.

1.2 Application:

This procedure has been used typically for detecting segregation, inclusions, and other defects in alpha-beta and certain alpha titanium alloys.

1.3 Safety-Hazardous Materials:

While the materials, methods, applications, and processes described or referenced in this specification may involve the use of hazardous materials, this specification does not address the hazards which may be involved in such use. It is the sole responsibility of the user to ensure familiarity with the safe and proper use of any hazardous materials and to take necessary precautionary measures to ensure the health and safety of all personnel involved.

2. APPLICABLE DOCUMENTS:

The issue of the following documents in effect on the date of the purchase order forms a part of this specification to the extent specified herein. The supplier may work to a subsequent revision of a document unless a specific document issue is specified. When the referenced document has been cancelled and no superseding document has been specified, the last published issue of that document shall apply.

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2.1 SAE Publications:

Available from SAE, 400 Commonwealth Drive, Warrendale, PA 15096-0001 or www.sae.org.

AMS 4901 Titanium Sheet, Strip, and Plate, Commercial Pure-Annealed, 70.0 ksi (485 MPa)

2.2 ASTM Publications:

Available from ASTM, 100 Barr Harbor Drive, West Conshohocken, PA 19428-2959 or www.astm.org.

ASTM E 1447 Determination of Hydrogen in Titanium and Titanium Alloys by the Inert Gas Fusion Thermal Conductivity Method

2.3 ASME Publications:

ASME B46.1 Surface Texture, Roughness, Waviness and Lay

3. TECHNICAL REQUIREMENTS:

3.1 Macrostructure:

3.1.1 Specimens:

3.1.1.1 Bars, Extrusions, Plates, and Stock for Forging or Extruding: Specimens shall be transverse cross-sections not less than 0.50 inch (12.7 mm) thick, cut from the product to be tested. Specimens from stock for alpha beta forging or alpha beta extruding shall be heated to a temperature $50\text{ F} \pm 25$ ($28\text{ C} \pm 14$) degrees below the beta transus as determined for the heat, held at heat for 60 minutes ± 5 , and cooled at a rate equivalent to air cool or faster. Specimens from stock for beta forging or beta extruding shall be heated to a temperature $50\text{ F} \pm 25$ ($28\text{ C} \pm 14$) degrees above the beta transus as determined for the heat. Specimens shall be machined to have surface texture of 70 micro-inches (1.8 μm) AA or finer, determined in accordance with ANSI B46.1.

3.1.1.2 Forgings: When dimensions permit, forgings shall be machined all over to remove approximately 0.040 inch (1.02 mm) of metal to ensure freedom from alpha case. If forgings are to be inspected by ultrasonic inspection procedures, the configuration used for such inspection is acceptable. Surface texture shall be 70 microinches (1.8 μm) AA or finer, determined in accordance with ANSI B46.1.

3.1.1.3 Finished Parts: Specimens shall be complete parts. No special preparation is required.

3.1.2 Procedure:

3.1.2.1 Inspect fixtures, prior to use, for distortion, loose fittings, worn areas and corrosion. Electrical contact on finished parts must be in a noncritical area.

- 3.1.2.2 Specimens shall be thoroughly cleaned to ensure that all surfaces are free from dirt, grease, oil, and other foreign materials by immersion for not less than two minutes in an alkaline cleaning solution maintained at 110 to 180 °F (43 to 82 °C). Specimens that do not contain recesses, in which solvent could be trapped, may be vapor degreased before cleaning in the alkaline cleaner.
- 3.1.2.2.1 Following alkaline cleaning, specimens shall be rinsed in clean tap water and inspected for water breaks. If water breaks are observed, parts shall be recleaned as in 3.1.2.2, swabbing if necessary, and then rinsed. Cleaning shall be repeated until no water breaks are observed.
- 3.1.2.3 Areas on finished parts that are not to be “etch-anodize” inspected shall be protected by a suitable masking material.
- 3.1.2.3.1 When inspecting finished parts and assemblies, surfaces which are to be masked, to prevent “etch-anodizing”, shall include inspection areas or surfaces, balance and identification markings applied by electrolytic etching, hardfaced areas, and non-titanium surfaces.
- 3.1.2.4 Immerse the specimen for 80 to 100 seconds after the onset of gassing in an agitated acid salt solution, maintained at room temperature. Immersed parts shall be agitated to remove entrapped air. Immediately after removal, immerse the specimen in clean tap water. Use a pressure spray to remove smut. The concentration of acid salt solution shall be maintained at a level which will remove 0.00003 to 0.00005 inch (0.76 to 1.27 μm) of metal in 80 to 100 seconds.
- 3.1.2.5 Preset rectifier voltage at 30 volts D.C. ± 1 and turn off power. Batteries may be used to provide this voltage. Set timer at 30 seconds ± 1 . Immerse the specimen in an agitated aqueous solution containing 13 to 17 ounces/gallon (97 to 127 g/L) of hydrated trisodium phosphate, maintained at a pH of 8.5 ± 0.5 (See 8.3) and a temperature of 70 °F ± 10 (21 °C ± 5). Apply current and anodize for 30 seconds ± 1 . Do not agitate specimen with the current on. Remove the specimen from the anodizing solution and rinse in clean water. DO NOT TOUCH THE SPECIMEN WITH BARE HANDS. If the amperage does not fall below to one ampere, the part may be anodized for an additional 30 second cycle.
- 3.1.2.6 Immerse the specimen in an aqueous solution containing 367 g/L by volume technical grade nitric acid (HNO_3) (sp gr 1.408) and 31.5 g/L by volume technical grade hydrofluoric acid (70% HF), maintained at room temperature, to obtain a light blue-to-gray background and develop maximum contrast between any segregation and the background. Immersion time shall be measured from time of complete immersion of the specimen. Immersion times for typical alloys are as shown in Table 1; times for other alloys must be developed:

TABLE 1 - Immersion Times

Alloy	Immersion Time, Seconds
Ti-6Al-4V	2 to 10
Ti-8Al-1Mo-1V	15 to 25
Ti-6Al-2Sn-4Zr-2Mo	10 to 20

- 3.1.2.7 Remove the specimen from the nitric-hydrofluoric acid solution, immerse in clean tap water as rapidly as possible, and rinse thoroughly, followed by immersion in hot water at 190 to 210 °F (88 to 99 °C) to assist drying. Dry immediately with a clean, oil-free, dry air blast.
- 3.1.2.8 Inspect the specimen immediately for macrostructure and defects such as segregation (See 8.4), laps, folds, cracks, inclusions, and pitted areas using 1 to 10X magnification and light intensity not lower than 200 foot-candles (2153 lx).
- 3.2 Microstructure:
- If examination of the etch-anodized specimens indicates the need for further evaluation, the microstructure shall be developed as follows:
- 3.2.1 Specimens: Shall be those which were etch-anodized. When necessary to permit the desired evaluation, the specimens shall be cut for examination of other planes.
- 3.2.2 Procedure:
- 3.2.2.1 Polish and etch the specimens to be examined, using metallographic techniques or replication techniques (See 8.5 and 8.6), which will clearly reveal the microstructure to be evaluated.
- 3.2.2.2 Examine the specimens to determine the microstructure and the nature of indications found in the macrostructure.
- 3.3 Parts shall be uniform in color and appearance after coating.
4. QUALITY ASSURANCE PROVISIONS:
- 4.1 The hydrogen pick-up permitted when this inspection procedure is used shall not exceed 20 ppm, tested, using a 0.040 inch (1.0 mm) maximum thickness titanium test strip in accordance with AMS 4901. Test frequency shall be as specified by purchaser. Hydrogen content shall be determined in accordance with ASTM E 1447.
5. PREPARATION FOR DELIVERY:
- Not applicable.
6. ACKNOWLEDGMENT:
- A vendor shall mention this specification number and its revision letter in all quotations and when acknowledging purchase orders.
7. REJECTIONS:
- Not applicable.

8. NOTES:

- 8.1 A change bar (|) located in the left margin is for the convenience of the user in locating areas where technical revisions, not editorial changes, have been made to the previous issue of a specification. An (R) symbol to the left of the document title indicates a complete revision of the specification, including technical revision. Change bars and (R) are not used in original publications, nor in specifications that contain editorial changes only.
- 8.2 Equipment:
- 8.2.1 The tanks for the solutions of 3.1.2.3 and 3.1.2.6 should be lined with polyvinyl chloride or comparable lining material; all others may be unlined.
- 8.2.2 Temperature indicators and controllers should be used with the alkaline cleaner, anodizing, and hot water tanks.
- 8.2.3 Mechanical stirrers and/or air agitation are required in the alkaline cleaner tank, acid tanks, and the anodizing tank.
- 8.2.4 Timers should be used on both acid tanks and the anodizing tanks.
- 8.2.5 Rectifier requirements are:
- 8.2.5.1 Maximum of 6% ripple at 30 volts, no load.
- 8.2.5.2 Capable of increasing voltage to 30 volts D.C. in 1 to 3 seconds and preferably automatically controlled.
- 8.2.5.3 A non ramp controlled rectifier should be equipped with a time actuated switch that, in less than three seconds after turning on the current, cut out a 1.2 ohm resistor installed in series with the load (resistor reduces the initial surge of current and thereby reduces the chance of arcing). A ramp controlled rectifier should have a voltage/current regulation with an adjustable current ramp regulator that has a zero to maximum rise time between one and three seconds.
- 8.2.6 Fixtures should be designed so that no non-titanium surfaces are exposed to any of the processing solutions.
- 8.2.7 Contact surfaces of fixtures should be contoured to match the contour of specimens in the contact areas and shall be kept clean and bright to prevent arcing and pitting.
- 8.3 The pH of the trisodium phosphate solution of 3.1.2.5 can be controlled by additions of phosphoric acid (H_3PO_4) to lower the pH or of sodium hydroxide (NaOH) to raise it.