

**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:** Primarily 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 following publications form a part of this specification to the extent specified herein. The latest issue of SAE publications 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.

2.1.1 **Aerospace Material Specifications:**

AMS 2350 - Standards and Test Methods

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2.2 ANSI Publications: Available from American National Standards Institute, Inc., 1430 Broadway, New York, New York 10018.

ANSI B46.1 - Surface Texture

### 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 (12.7mm) thick, cut from the product to be tested. Specimens from stock for forging or extruding shall be heated to a temperature  $50^{\circ}\text{F} \pm 25$  ( $28^{\circ}\text{C} \pm 14$ ) below the beta transus as determined for the heat, held at heat for 60 minutes  $\pm 5$ , and cooled at a rate equivalent to air cool or faster. Specimens shall be machined to have surface texture of 70 micro-inches (1.8  $\mu\text{m}$ ) 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  $\mu\text{m}$ ) 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 2 minutes in a alkaline cleaning solution maintained at  $110^{\circ} - 180^{\circ}\text{F}$  ( $43^{\circ} - 82^{\circ}\text{C}$ ). Specimens which 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 or vapor degreasing, 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 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 - 100 seconds 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 0.03 - 0.05 mil (0.76 - 1.27  $\mu\text{m}$ ) of metal in 80 - 100 seconds.

3.1.2.5 Preset rectifier voltage at 30 volts  $\pm$  1 and turn off power. Batteries maybe used to provide this voltage. Set timer at 30 seconds  $\pm$  1. Immerse the specimen in an agitated aqueous solution containing 13 - 17 ounces/gallon (97 - 127 g/L) of trisodium phosphate, maintained at a pH of 8.5  $\pm$  0.5 (See 8.3) and a temperature of 70°F  $\pm$  10 (21°C  $\pm$  5). Do not agitate specimen with the current on. Apply current and anodize for 30 seconds  $\pm$  1. Remove the specimen from the anodizing solution and rinse in clean water. DO NOT TOUCH THE SPECIMEN WITH BARE HANDS.

3.1.2.6 Immerse the specimen in an aqueous solution containing 35% by volume technical grade nitric acid ( $\text{HNO}_3$ ) (sp. gr 1.408) and 2.5% 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 follows; times for other alloys must be developed:

Alloy	Immersion Time, Seconds
Ti -6Al-4V	2 - 10
Ti -8Al-1Mo-1V	15 - 25
Ti -6Al-2Sn-4Zr-2Mo	10 - 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° - 210°F (88° - 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 - 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.

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4. QUALITY ASSURANCE PROVISIONS: Not applicable.

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 Marginal Indicia: The phi (0) symbol is used to indicate technical changes from the previous issue of this specification.

8.2 Equipment:

8.2.1 The tanks for the solutions of 3.1.2.3 and 3.1.2.6 must be lined with polyvinyl chloride or comparable lining material; all others may be unlined.

8.2.2 Temperature indicators and controllers shall 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 shall 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 in 1 - 3 seconds and preferably automatically controlled.