

AEROSPACE MATERIAL SPECIFICATION

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Superseding AMS 2642B

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.

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)

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2.2 ASTM Publications:

Available from ASTM, 100 Barr Harbor Drive, P.O. Box C700, 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:

Available from ASME International, 22 Law Drive, Box 2900, Fairfield, NJ 07007-2900 or www.asme.org.

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

3. TECHNICAL REQUIREMENTS:

3.1 Materials and Solutions:

3.1.1 Etch Solution: Prepare an acid salt solution and maintain at room temperature. The concentration of acid salt solution shall be maintained at a level that will remove 0.00003 to 0.00005 inch (0.76 to 1.27 μm) of metal in 80 to 100 seconds.

3.1.2 Anodize Solution: An aqueous solution of 13 to 17 ounces/gallon (97 to 127 grams/L) of hydrated trisodium phosphate shall be prepared. Maintain at pH of 8.5 ± 0.5 (See 8.3) and $70^\circ\text{F} \pm 10$ ($21^\circ\text{C} \pm 5$).

3.1.3 Back Strip Solution: An agitated aqueous solution of 42.5 to 51.5 ounces/gallon (320 to 385 grams/liter) of nitric acid (technical grade) and 1.3 to 4.7 ounces/gallon (10 to 35 grams per liter) hydrofluoric acid (technical grade) . Maintain at room temperature.

3.1.4 Specimens for Macrostructure:

3.1.4.1 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 extrusion shall be heated to a temperature $50^\circ\text{F} \pm 25$ ($28^\circ\text{C} \pm 14$) 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 extrusion shall be heated to a temperature $50^\circ\text{F} \pm 25$ ($28^\circ\text{C} \pm 14$) degrees above the beta transus as determined for the heat. Specimens shall be machined to have surface texture of 70 microinches (1.8 μm) AA or finer, determined in accordance with ASME B46.1.

- 3.1.4.2 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 ASME B46.1.
- 3.1.4.3 Finished Parts: Specimens shall be complete parts. No special preparation is required.
- 3.2 Equipment:
- 3.2.1 A DC voltage source that shall be capable of providing current with a maximum of 6% ripple at 30 volts, no load and shall be capable of increasing voltage to 30 volts D.C. in 1 to 3 seconds. Automatic control is preferred.
- 3.2.2 Fixtures shall be free of distortion, loose fittings, worn areas and corrosion.
- 3.3 Preparation:
- 3.3.1 Specimens shall be free from water breaks. If water breaks are observed, parts shall be recleaned and reinspected for water break until no water breaks are observed.
- 3.3.2 Location of electrical contact points on finished parts shall be acceptable to purchaser.
- 3.3.3 Areas on finished parts that are not to be etch-anodize-inspected shall be protected by a suitable masking material. Such areas shall include inspection areas or surfaces, balance and identification markings applied by electrolytic etching, hardfaced areas, and nontitanium surfaces.
- 3.3.4 Macrostructure Procedure:
- 3.3.4.1 Specimens shall be immersed for 80 to 100 seconds after the onset of gassing in an agitated acid salt solution in accordance with 3.1.1 Immersed parts shall be agitated to remove entrapped air. Immediately after removal, specimens shall be immersed in clean tap water. Specimens shall be pressure sprayed as required to remove smut.
- 3.3.4.2 Specimens shall be immersed in an agitated aqueous solution in accordance with 3.1.2 and an anodic direct current voltage of 30 volts D.C. \pm 1 applied for 30 seconds \pm 1. Specimens shall not be agitated when the voltage is applied. Specimens shall be removed from the anodizing solution and immediately rinsed 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 up to 4 more additional 30-second cycles to achieve less than one ampere.

- 3.3.4.3 Specimens shall be immersed in solution in accordance with 3.1.3 until a light blue-to-gray background color is obtained and maximum contrast between any segregation and the background develops. Immersion time shall be measured from time of complete immersion of the specimen. Immersion times for typical alloys are as shown in Table 1 for a hydrofluoric acid concentration of 20 grams/liter; times and concentrations 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

- 3.3.4.4 Immediately rinse and dry.

- 3.3.4.5 The specimens shall be immediately inspected for macrostructure and defects such as segregation (See 8.6), laps, folds, cracks, inclusions, and pitted areas using 1 to 10X magnification and light intensity not lower than 200 foot-candles (2153 lx).

- 3.3.4.6 The hydrogen pick-up permitted when this inspection procedure is used shall not exceed 20 ppm when tested in accordance with ASTM E 1447.

3.4 Microstructure:

If examination of the etch-anodized specimens indicates the need for further evaluation, the microstructure shall be developed as follows:

- 3.4.1 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.

3.4.2 Procedure:

- 3.4.2.1 Polish and etch the specimens to be examined, using metallographic techniques or replication techniques (See 8.7 and 8.8) that will clearly reveal the microstructure to be evaluated.

- 3.4.2.2 Examine the specimens to determine the microstructure and the nature of indications found in the macrostructure.

- 3.5 Parts shall be uniform in color and appearance after coating.

4. QUALITY ASSURANCE PROVISIONS:

- 4.1 Test specimen for hydrogen pick-up (3.3.4.6) shall be 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.

4.2 Acceptance Tests: Macrostructure (3.3.4), hydrogen pickup (3.3.4.6), visual examination (3.5), and, when applicable, microstructure (3.4) shall be performed to determine product acceptance.

4.3 Periodic Tests: Tests to maintain all of the solutions used herein are periodic tests and shall be performed at a frequency selected by the processor unless frequency of testing is specified by purchaser. See 8.4.

5. PREPARATION FOR DELIVERY:

Not applicable.

6. ACKNOWLEDGMENT:

A processor 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 3.1.1 and 3.1.3 should be lined with polyvinyl chloride or comparable lining material; all others may be unlined. Compatible plastic tanks could also be used.

8.2.2 Temperature indicators and controllers should be used with the alkaline cleaner, anodizing, and hot water tanks.

8.2.3 For best results mechanical stirrers and/or air agitation are recommended 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 A nonramp-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 provide 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 nontitanium 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 can be controlled by additions of phosphoric acid (H_3PO_4) to lower the pH or of sodium hydroxide (NaOH) to raise it.

8.4 ARP 4992, Periodic Test Plan for Process Solutions, is recommended to satisfy the requirements for the control of processing solutions.

8.5 Specimens that do not contain recesses, in which solvent could be trapped, may be vapor degreased before cleaning in the alkaline cleaner.

8.6 Appearance:

Various types of segregation appear as follows:

8.6.1 High Aluminum Defect (HAD) (Aluminum-Stabilized Alpha): Appears as a deeper blue irregular line, with or without branches, on a lighter blue-to-gray background.

8.6.2 Alpha Case (Interstitial Element-Stabilized Alpha (Oxygen and Nitrogen Contamination)): Also appears blue. Differentiation between this and aluminum-stabilized alpha can generally be achieved by examination of microstructure using surface replication techniques.

8.6.3 Beta Fleck (Vanadium-Stabilized Beta): Appears as a gray or white line, with or without branches. This type of segregation is frequently associated with aluminum-stabilized alpha, appearing as an adjacent parallel line.

8.6.4 Unalloyed Titanium: Also appears light gray or white. Differentiation between this and vanadium-stabilized beta can be achieved by examination of microstructure using surface replication techniques.

8.7 The preferred procedure for preparing specimens for direct examination of microstructure is as follows: