

Structural Examination of Titanium Alloys
Chemical Etch Inspection Procedure

RATIONALE

This document revision was issued as part of the SAE Five Year Review process.

1. SCOPE

1.1 Purpose

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

1.2 Application

This inspection procedure has been used typically for detecting defects and segregation in titanium alloys. This procedure should not be used on finished parts.

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 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 document 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 canceled and no superseding document has been specified, the last published issue of that document shall apply.

2.1 ASME Publications

Available from American Society of Mechanical Engineers, 22 Law Drive, P.O. Box 2900, Fairfield, NJ 07007-2900, Tel: 973-882-1170, www.asme.org.

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

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3. TECHNICAL REQUIREMENTS

3.1 Materials and Solutions

3.1.1 Specimens for Revealing Macrostructure

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 in the transverse direction. Specimens from stock for forging or extruding shall be heated to $50\text{ }^{\circ}\text{F} \pm 25$ ($28\text{ }^{\circ}\text{C} \pm 14$) degrees below the beta transus as determined for the heat, held at heat for 60 minutes ± 5 , and cooled at a rate equivalent to an air cool or faster. Surface roughness shall be of 70 microinches (1.8 μm) AA or less, determined in accordance with ASME B46.1.

3.1.1.2 Forgings

When dimensions permit, forgings shall be rough-machined to clean up and to ensure freedom from alpha case. A minimum of 0.030 inch (0.76 mm) of material shall be left on the surfaces of the forgings between final etching and finish machined configuration to ensure freedom from intergranular attack and hydrogen pickup on finished parts. If forgings are to be inspected by ultrasonic inspection procedures, the configuration used for such inspection is acceptable. Surface roughness shall be 130 microinches (3.3 μm) AA or less, determined in accordance with ASME B46.1.

3.1.2 Specimens for Revealing Microstructure

The specimens on which macrostructure was developed shall be sectioned through the areas of concern and, also, where practical, shall be sectioned for examination of other planes.

3.2 Equipment

3.2.1 The etchant tank, fixtures, racks, and baskets shall be lined/coated with or fabricated from a material that will not react with the etchant (See Table 1).

3.2.2 The tank shall be equipped with exhaust ventilation to vent gases from the vigorous reaction.

3.3 Preparation

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.

3.4 Procedure

3.4.1 Macrostructure Inspection

Specimens shall be etched in an aqueous acid solution, maintained at room temperature, for sufficient time to develop a well-defined macrostructure (see 8.2). The solution in Table 1, or an alternative solution acceptable to purchaser, shall be used.

TABLE 1 - ETCHANT

Ingredient	Grams per liter*	Ounces per gal*
Nitric Acid, Technical Grade	142 ± 20	19.0 ± 2.7
Hydrofluoric Acid, Technical Grade	60 ± 9	8.0 ± 1.2
Water	Balance	Balance

* Expressed as HNO_3 or HF respectively by weight.

3.4.1.1 The etching rate of the solution shall be maintained to remove 0.002 to 0.004 inch (0.05 to 0.10 mm) of metal in a five minute period.

3.4.1.2 Rinse and Dry

Specimens shall be rinsed immediately in clean water for several minutes followed, as required, by pressurized spraying to remove smut.

Remove water from specimens by blowing dry with clean air.

3.4.1.3 Specimens shall be visually inspected for macrostructure and defects, such as segregation, (See 8.2), laps, folds, cracks, and inclusions using 1X magnification and a light intensity not lower than 200 foot-candles (2153 lx).

3.4.2 Microstructure Inspection

If examination of the macrostructure indicates the need for further evaluation, the microstructure shall be developed as follows:

3.4.2.1 The specimens to be examined shall be polished and etched using metallographic techniques that will clearly reveal the microstructure to be evaluated (See 8.3).

3.4.2.2 The specimens shall be examined to determine the microstructure and the nature of indications found in the macrostructure.

4. QUALITY ASSURANCE PROVISIONS

4.1 Acceptance Tests

Tests for macrostructure (3.4.1) and, when applicable, microstructure (3.4.2) shall be performed to determine product acceptance.

4.2 Periodic Test

The test to maintain the etchant solution (3.4.1.1) is a periodic test and shall be performed at a frequency selected by the processor unless frequency of testing is specified by the cognizant engineering organization. See 8.4.

5. PREPARATION FOR DELIVERY

Not applicable.

6. ACKNOWLEDGMENT

The 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 (I) 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 this specification. An (R) symbol to the left of the document title indicates a complete revision of the specification, including technical revisions. Change bars and (R) are not used in original publications, nor in specifications that contain editorial changes only.

8.2 Segregation

Segregation is similar to other material defects in that it can occur throughout the product in any frequency, size, shape, form, or severity. However, the extreme ends of the ingot are most likely to have segregation. Segregation will appear, after macroetching, as an area of high silvery luster on a matte grey background. Typical segregation in titanium 6Al-4V alloy billet is shown in Figure 1. Segregation can be distinguished from staining or other discoloration by rubbing the surface with a rubber eraser. Segregation cannot be rubbed out whereas staining or discoloration will disappear. After segregation has been obliterated by rubbing with abrasive paper, it will reappear after localized re-etching.

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

8.3.1 Polish electrolytically in the following solution at 40 volts \pm 1 direct current for approximately five seconds:

- 78 mL perchloric acid
- 120 mL reagent water (See ASTM D 1193 Type II)
- 700 mL ethanol
- 100 mL 2-butoxy ethanol

8.3.2 Etch with Kroll's reagent (5% hydrofluoric acid, (sp gr 1.15), 12% nitric acid, (sp gr 1.42), 83% water, by volume), for sufficient time to develop the microstructure, wash in warm running water, and dry. Specimens may be rinsed in a solution of sodium bicarbonate and again rinsed in warm running water, if desired, between the rinsing and drying operations.

8.3.3 Mechanical polishing or other suitable means may be used provided equivalent microstructural presentations are developed.

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

8.5 Terms used in this AMS are clarified in ARP1917.

8.6 Dimensions and properties in inch/pound units and the Fahrenheit temperatures are primary; dimensions and properties in SI units and the Celsius temperatures are shown as the approximate equivalents of the primary units and are presented only for information.