

Heat Treatment of Titanium and Titanium Alloys

NOTICE

This document is intended to replace MIL-H-81200. The original issue of AMS-H-81200 was taken directly from Military Specification MIL-H-81200A and contained only minor editorial and format changes required to bring it into conformance with the publishing requirements of SAE technical standards. This revision B changes the heat treatment requirements for parts from "may be heat treated in accordance with the requirements of AMS 2801" to "shall be heat treated in accordance with AMS 2801". As an exception, it allows continuation of heat treatment procedures for specific parts which have been previously acceptable to the purchaser. It also adds the requirement that the heat treat parameters used for heat-treat-response-tests of raw material conform to the parameters used for heat treatment of parts (See 3.1.1).

The original Military Specification was adopted as an SAE standard under the provisions of the SAE Technical Standards Board (TSB) Rules and Regulations (TSB 001) pertaining to accelerated adoption of government specifications and standards. TSB rules provide for (a) the publication of portions of unrevised government specifications and standards without consensus voting at the SAE Committee level, and (b) the use of the existing government specification or standard format.

Under Department of Defense policies and procedures, any qualification requirements and associated qualified products lists are mandatory for DOD contracts. Any requirement relating to qualified products lists (QPL's) has not been adopted by SAE and is not part of this SAE technical document.

SAE Technical Standards Board Rules provide that: "This report is published by SAE to advance the state of technical and engineering sciences. The use of this report is entirely voluntary, and its applicability and suitability for any particular use, including any patent infringement arising therefrom, is the sole responsibility of the user."

SAE reviews each technical report at least every five years at which time it may be reaffirmed, revised, or cancelled. SAE invites your written comments and suggestions.

Copyright 2002 Society of Automotive Engineers, Inc.

All rights reserved. No part of this publication may be reproduced, stored in a retrieval system or transmitted, in any form or by any means, electronic, mechanical, photocopying, recording, or otherwise, without the prior written permission of SAE.

**TO PLACE A DOCUMENT ORDER:**

Tel: 877-606-7323 (inside USA and Canada)  
Tel: 724-776-4970 (outside USA)  
Fax: 724-776-0790  
Email: [custsvc@sae.org](mailto:custsvc@sae.org)  
<http://www.sae.org>

**SAE WEB ADDRESS:**

## 1. SCOPE:

## 1.1 Purpose:

This specification covers the heat treatment of titanium and titanium alloy mill products (raw material), including wrought and cast products, by material producers. This specification also covers furnace equipment requirements, test procedures, and general information for heat treating procedures, heat treating temperatures, and material test procedures for the heat treatment of titanium and titanium alloys. It also describes procedures which, when followed, have produced the desired properties within the limitations of the respective alloys.

## 1.2 Heat treatments:

The heat treatments covered by this specification are:

Anneal	Solution heat treatment
Beta anneal	Beta solution heat treatment
Recrystallization anneal	Age
Duplex anneal	Stress relief

## 1.3 Alloys:

In addition to Commercially Pure Titanium (Ti40, Ti55, and Ti70), the following titanium alloys are covered by this specification:

<u>Alpha alloys</u>	<u>Alpha-Beta alloys</u>	<u>Beta alloys</u>
6Al-2Sn-4Zr-2Mo	6Al-4V	13V-11Cr-3Al
5Al-2.5Sn	6Al-4V ELI	3Al-8V-6Cr-4Mo-4Zr
5Al-2.5Sn ELI	6Al-6V-2Sn	15V-3Al-3Cr-3Sn
6Al-2Cb-1Ta-0.8Mo	3Al-2.5V	10V-2Fe-3Al
8Al-1Mo-1V	6Al-2Sn-4Zr-6Mo	
11Sn-5Zr-2Al-1Mo	6Al-2Sn-2Zr-2Mo-2Cr-0.25Si	
	5Al-2Sn-2Zr-4Mo-4Cr	

## 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 U.S. Government Publications:

Available from DODSSP, Subscription Services Desk, Building 4D, 700 Robbins Avenue, Philadelphia, PA 19111-5094.

MIL-STD-45662 Calibration System Requirement

## 2.2 ASTM Publications:

Available from ASTM, 100 Barr Harbor Drive, West Conshohocken, PA 19428-2959 or [www.astm.org](http://www.astm.org).

ASTM E 3 Metallographic Specimens, Preparation of  
ASTM E 8 Tension Testing of Metallic Materials  
ASTM E 146 Chemical Analysis of Zirconium and Zirconium Alloys  
ASTM E 290 Semi-Guided Bend Test for Ductility of Metallic Materials

## 2.3 SAE Publications:

Available from SAE, 400 Commonwealth Drive, Warrendale, PA 15096-0001 or [www.sae.org](http://www.sae.org).

AMS 2750 Pyrometry  
AMS 2801 Heat Treatment of Titanium Alloy Parts

## 2.4 Order of precedence:

In the event of a conflict between the text of this document and the references cited herein, the text of this document shall take precedence. Nothing in this document, however, shall supersede applicable laws and regulations, unless a specific exemption has been obtained.

## 3. REQUIREMENTS:

### 3.1 General:

All heating and quenching equipment and procedures applied shall yield products complying with the requirements of appropriate acquisition documents. Equipment and procedures shall be designed to minimize the introduction of hydrogen, oxygen, nitrogen or other contaminants and in any case shall not allow introduction beyond levels established by the acquisition documents. Deviation from process requirements specified herein or the application of processes different from those contained herein, may be used provided that compliant products result, these exceptions have been proven satisfactory, and that they are made known to the purchaser with accompanying data or other justification to support the deviation prior to application of the deviant process.

- 3.1.1 Heat treatment of mill products and titanium alloy parts: The requirements specified herein are applicable to the heat treatment of mill products (raw material) (see 6.4.1). Producer heat treatment of heat-treat-response-test samples shall conform to AMS 2801 (see 4.8.6.1). Parts (see 6.4.2) shall be heat treated in accordance with AMS 2801 except as specified in 3.1.1.1.
- 3.1.1.1 It is permissible, for specific parts, to use equipment, practices and test methods which conformed to AMS-H-81200 or MIL-H-81200 and were previously acceptable to the purchaser.
- 3.2 Heating systems:
- 3.2.1 Batch furnaces:
- 3.2.1.1 General requirements: Such furnaces may employ electrical heating elements or fuel combustion as heat sources. Muffle furnaces and retorts are also allowed. Allowable environments surrounding the furnace charge during heating are: inert gas (argon or helium), vacuum, slightly oxidizing mixtures resulting from the combustion in air of hydrocarbons (gas or oil), and air itself. When removal of surface contamination is not feasible, inert gas or vacuum environment shall be employed. The selection of an atmosphere shall be such as to establish conformance with 3.1.
- 3.2.1.2 Inert gases: Inert gases within the furnace shall be circulated as necessary to protect all surfaces of the workpieces comprising the furnace charge. The dew point of the inert gases shall be minus 65°F (-54°C) or lower. This requirement shall be met during all stages of a heating, soaking or cooling cycle. Ducts and zones which are to contain furnace charges shall be so sealed as to prevent contamination of any charge to the degree that it is rendered nonconforming to specified material requirements.
- 3.2.1.3 Vacuum: Vacuum furnaces used for outgassing hydrogen shall be capable of reducing hydrogen concentrations within the charge to levels complying with 3.1. Vacuum furnaces and retorts used for prevention of surface contamination shall be capable of yielding product conforming to 3.1.
- 3.2.1.4 Combusted hydrocarbons: Furnaces heated by the combustion in air of gas or oil shall contain a slightly oxidizing gas mixture. There shall be no impingement of flame upon the furnace charge.
- 3.2.1.5 Prohibited atmospheres: Endothermic, exothermic, hydrogen, and cracked ammonia atmospheres shall not be used during any heat treatment operation.
- 3.2.1.6 Furnace purging: Prior to thermal treatment of workpieces, each furnace which has contained an atmosphere unacceptable for heat treating (see 3.2.1.5) shall be purged with air or inert gas, as applicable.

- 3.2.1.6.1 Purging prior to introducing air or combusted hydrocarbons: The volume of purging air introduced shall be at least twice the volume of the furnace chamber. During purging, the minimum temperature within the chamber shall be the intended soaking temperature of the charge. When air-flow purging is impractical, the furnace temperature shall be set at 200°F above the intended soaking temperature, and be held at that temperature for a minimum of four hours. Following purging, the furnace shall be stabilized at the required temperature, charged, and the charge heated and soaked in accordance with 3.2.1.7, as applicable. Following the thermal treatment and any subsequent cleaning, pickling, or other process which may introduce hydrogen contamination, specimens shall be taken from the charge and subjected to the test specified in 4.7.3. Results shall show compliance with 3.6.5, as applicable.
- 3.2.1.6.2 Purging prior to introducing inert gas: Procedures for purging shall comply with 3.2.1.2. The volume of gas introduced shall be at least twice the volume of the furnace chamber. Furnaces shall be charged while cold, and then purged and filled with inert gas. The charge shall then be heated and soaked in accordance with 3.2.1.7, as applicable. Following the thermal treatment, samples shall be taken from the charge and subjected to the test specified in 4.7.3. Results shall show conformance to 3.6.5, as applicable. Additionally, samples shall be taken from the charge and examined in accordance with 4.7.4.2. Results shall show conformance to 3.6.6.
- 3.2.1.7 Temperature uniformity: Batch furnaces shall be so controlled that, during heating and soaking periods, temperatures at all points within the working zones are less than the maxima of the ranges specified in Tables I, III, IV, and V, as applicable to the product. After a charge has reached a pre-selected soaking temperature throughout its thickness within a specified range, the temperature at any point in the working zone shall lie within the limits specified below, as applicable to the thermal treatment intended. Regardless of temperature tolerances, no soaking temperature during any thermal treatment shall be higher than the applicable maximum, nor lower than the applicable minimum of the specified range.

<u>Heat treatment</u>	<u>Temperature tolerance</u>	
	°F	°C
Annealing	±25	±14
Beta annealing or beta solution heat treating	±25	±14
Recrystallization annealing	±25	±14
Duplex annealing	±25	±14
Solution heat treating	±25	±14
Stress relieving	±25	±14
Aging	±15	±8

### 3.2.2 Continuous furnaces:

- 3.2.2.1 General requirements: Such furnaces may be heated by radiation from electrically-energized heating elements or by combusted hydrocarbons.

- 3.2.2.2 Temperature control: A temperature profile from furnace entry to exit shall be so developed and maintained that the charge within the working zone experiences the appropriate thermal cycle to the degree necessary for eventual product acceptability in terms of specified requirements.
- 3.2.2.3 Continuous vacuum furnaces: Continuous vacuum furnaces shall be so sealed as to minimize hydrogen, oxygen and nitrogen absorption of the product and in any case shall not allow absorption beyond levels established by the acquisition documents.
- 3.2.3 Continuous induction heating: Such a heating method shall be applied only to the annealing of thin-walled tubing and extrusions of thin sections. The technique shall be such that the workpiece being heated is of uniform temperature around the perimeter of its cross-section. Prior to production, values of the process parameters which produce acceptable product shall be determined and documented.
- 3.2.4 Pyrometry and furnace temperatures control: The requirements and procedures for control and testing of furnaces, ovens, vacuum furnaces and allied pyrometric equipment used for heat treatment shall be in accordance with AMS 2750.
- 3.2.4.1 System accuracy: Each system shall be set to control working temperatures and be corrected to within the applicable tolerances specified herein.
- 3.3 Quenching facilities and media:
- 3.3.1 Quenching baths: Quenching baths holding water or oil shall be of such dimensions, volume, and construction that products quenched therein will, upon aging, develop the properties specified within applicable product documents. Mechanical stirring of the bath may be applied when necessary.
- 3.3.2 Spray or flow quenching: Continuous furnaces discharging solution heat treated alloy sheet, plate, and strip may be equipped with a quenchant system which directs a spray or streams of quenchant onto the product as it emerges from the furnace. The spray or flow of quenchant shall be applied evenly over the workpiece width, top and bottom surfaces, over a period of time and at a volume rate such that the resulting product will upon aging develop properties meeting specified requirements.
- 3.3.3 Location of quenching facility: Quenching and handling facilities shall be located such that contact between quenchant and workpieces occurs within the time required for compliance with 3.1 and for the Ti-6Al-4V and Ti-6Al-4V ELI alloys, within the limits specified in Table II.
- 3.3.4 Quenching media: Use of molten salt baths for quenching is prohibited.
- 3.4 Ancillary equipment:
- Jigs, fixtures, trays, hangers, racks, ventilators, etc. shall be so designed and constructed that each workpiece can be processed in accordance with this specification.

### 3.5 Thermal treatment parameter values:

The parameters (ie. temperatures, times, etc.) for the various thermal treatment processes shall be as specified herein, except where deviation has been accepted by the purchaser in accordance with 3.1 (see 6.2).

- 3.5.1 Solution heat treating: Solution heat treating of parts, mill product, castings, and forgings shall be as specified in Table I, as applicable.
- 3.5.2 Quenching: All heat treatable titanium alloys, except alloys which can be cooled in air or inert gas, shall be quenched by complete immersion in water or oil, as applicable, or by water spray or flow when applicable to quenching sheet, strip or plate. Maximum delay times for Ti-6Al-4V and Ti-6Al-4V ELI alloys shall conform to Table II, and for other alloys shall be as necessary to develop required properties.
- 3.5.3 Aging: Solution heat treated alloy workpieces shall be aged in accordance with Table III, as applicable. Workpieces shall be cooled from the aging temperature in air, an inert gas, or in the aging furnace.
- 3.5.4 Stress-relieving treatment: Time-temperature cycles for stress relieving shall be as specified in Table IV, as applicable. Workpieces may be cooled from the stress relieving temperature in air, an inert gas, or in the stress-relieving furnace.
- 3.5.5 Annealing: Time-temperature cycles for annealing shall be as specified in Table V, as applicable. For duplex annealing of Ti-6Al-4V and Ti-6Al-4V ELI alloys, see Table V, note 6.
- 3.5.6 Beta annealing: When such annealing or beta solution heat treatment is specified (see 6.2), a lot of workpieces of Ti-6Al-4V, Ti-6Al-4V ELI, Ti-6Al-6V-2Sn, or other alpha-beta alloy shall be soaked at a temperature which is  $50 \pm 25^{\circ}\text{F}$  ( $30 \pm 15^{\circ}\text{C}$ ) above the determined beta transus of the lot (see 4.7.4.1). The soaking time shall be such that all portions of the furnace charge and of each workpiece including midsection are soaked for at least 30 minutes. Following soaking, the lot shall be cooled in air or inert gas to ambient temperature. Furnace cooling is not permitted. Water quenching shall not be performed, unless specified in the contract or on the drawing. When water quenching is specified, the products of Ti-6Al-4V, Ti-6Al-4V ELI, and Ti-6Al-6V-2Sn shall be given a second anneal between  $1350^{\circ}\text{F}$  ( $732^{\circ}\text{C}$ ) and  $1400^{\circ}\text{F}$  ( $760^{\circ}\text{C}$ ) for 1 to 3 hours.
- ### 3.6 Process requirements other than those specified in 3.5:
- 3.6.1 General requirements: All heating, quenching and other processing equipment used for thermal treating shall be capable of producing end product conforming to 3.1. All units of a lot shall be heated uniformly and on the whole piece, never on a portion only. For coiled product heated within a continuous furnace or straight product heated within an induction coil, the product shall be heated uniformly in its cross-section.

- 3.6.2 Surface cleanliness: Prior to thermal treatment, surfaces of workpieces shall be free from heavy lubricants, halogen compounds, and other foreign matter, which will cause product to become noncompliant. Product coated with light oils need not be cleaned prior to thermal treatment, provided that the oil either vaporizes or burns off during preheating. Halogenated solvents (see sample list 6.3.5) and methanol shall not be used to degrease workpieces, unless the workpieces are subsequently cleaned using an alkaline solution or an acid pickle. Such cleaning shall be performed prior to thermal treatment.
- 3.6.3 Holding fixtures: All racks, support or fixtures contacting workpieces shall be made of heat resistant metal such as 300 series stainless steel or nickel base alloys. The use of ceramic or other suitable non-reacting material is also permitted. Fixtures shall be designed to permit free flow of heating and quenching media around each workpiece and to minimize distortion of workpieces being treated.
- 3.6.4 Protective coatings: The use of coatings to protect against scaling and to ease scale removal is permissible, on condition that resultant product meets the requirements of 3.1. Such a condition shall be demonstrable by test data.
- 3.6.5 Hydrogen contamination: Hydrogen concentrations in workpieces which are submitted for inspection, after all thermal treatments and manufacturer's processing, shall show no concentrations in excess of those specified in the acquisition documents when tested for in accordance with 4.7.3. Control of hydrogen absorption shall stem from control of furnace atmospheres, cleanliness of workpiece surfaces, and acid pickling. Where maximum hydrogen concentration is not specified in acquisition documents, the maximum allowable hydrogen concentration shall be as agreed upon between the contracting activity and the contractor.
- 3.6.5.1 Rework of product contaminated by hydrogen: An excessive hydrogen concentration found in a lot may be reduced to an acceptable concentration by heating the lot in a vacuum furnace conforming to 3.2.1.3. Such action shall be reported to the purchaser. Heating under vacuum which results in overaging of a lot shall be cause for rejection of that lot. Salvage by re-solution heat treating and aging shall be performed only with the consent of the purchaser. Records of all re-heat treatments shall be prepared and maintained in accordance with 4.8.4.
- 3.6.6 Surface contamination: Surface contamination after heat treatment shall be removed by chemical or mechanical means. The surfaces of machined, ground, blasted or acid-pickled workpieces shall not exhibit the effects of absorbed oxygen or nitrogen to the degree that the surface contamination of the product exceeds the levels specified in the acquisition documents when tested in accordance with 4.7.4.2.
- 3.7 Product monitoring:
- Periodic monitoring of heat treated workpieces to determine compliance to 3.1 shall include the evaluation of tensile and bend properties.

3.7.1 Tensile properties: Specimens taken from thermally treated workpieces in accordance with 4.6.3 and tested in accordance with 4.7.1 shall exhibit tensile strengths, yield strengths, elongations, and reductions in area in compliance with applicable requirements of acquisition documents. Tension testing shall be performed in accordance with Table VII of this specification unless otherwise specified in the acquisition documents.

3.7.2 Bend properties: Flat-rolled product of 0.187 inch (4.75 mm) nominal thickness or less when sampled in accordance with 4.6.3.1 shall exhibit no cracks or separation in any direction when examined at 20X magnification after having been tested in accordance with 4.7.2.

#### 4. QUALITY ASSURANCE PROVISIONS:

##### 4.1 Responsibility for inspection:

Unless otherwise specified in the contract or purchase order, the contractor is responsible for the performance of all inspection requirements as specified herein. Except as otherwise specified in the contract or purchase order, the contractor may use his own or any other facilities suitable for the performance of the inspection requirements specified herein, unless disapproved by the purchaser. The purchaser reserves the right to perform any of the inspections set forth in the specification where such inspections are deemed necessary to assure that supplies and services conform to the prescribed requirements.

4.1.1 Responsibility for compliance: All items shall meet all requirements of Sections 3. The inspection set forth in this specification shall become a part of the contractor's overall inspection system or quality program. The absence of any inspection requirements in the specification shall not relieve the contractor of the responsibility of ensuring that all products or supplies submitted to the Government for acceptance comply with all requirements of the contract. Sampling inspection, as part of manufacturing operations, is an acceptable practice to ascertain conformance to requirements, however, this does not authorize submission of known defective material, either indicated or actual, nor does it commit the Government to accept defective material.

##### 4.2 Quality conformance tests:

4.2.1 Periodic Tests: Tests to determine conformance to the following requirements are classified as periodic tests and, unless otherwise specified by the contracting activity (see 6.2), shall be performed at the frequency specified herein, as applicable to furnace type.

- a. Daily check of the dew point of the inert gases.
- b. Monthly test of furnace pyrometer systems accuracy (see 4.5.1)
- c. Weekly checks for hydrogen pickup or contamination, except for processes wherein every thermally treated lot is analyzed, or for treatments in a vacuum furnace or in inert gas.
- d. At least one surface contamination examination weekly (see 4.7.4.2) of product thermally treated in a vacuum furnace or in inert gas, in order to detect possible leakage.
- e. Quarterly calibration of furnace instruments as in 4.5.
- f. Quarterly system accuracy tests and instrument calibration of stress relieving as in 4.5.
- g. Temperature surveys of furnace (see 4.3. for frequency).

- 4.2.2 Preproduction tests: Tests to determine conformance to the following requirements are classified as preproduction tests and shall be performed prior to any production heat treating:
- a. Furnace temperature uniformity or distribution (see 4.4.)
  - b. Pyrometer system accuracy as in 4.5.
  - c. Furnace instrument calibration as in 4.5.
  - d. Dew point of the inert gas when such gas is used.
  - e. Hydrogen contamination.
  - f. Leak rate.
- 4.3 Equipment Calibration and Tests:
- 4.3.1 Pyrometric calibration: Pyrometric equipment shall be calibrated in accordance with AMS 2750.
- 4.3.2 Test procedures for equipment: Heat treating equipment shall be tested in accordance with AMS 2750.
- 4.4 Survey requirements:
- 4.4.1 General: Procedures for surveying furnaces shall be in accordance with AMS 2750.
- 4.4.2 Furnace temperatures when making initial surveys: Furnaces used for thermal treatment shall be surveyed at the highest and lowest service temperatures anticipated as governed by the furnace application and also at one or more intermediate temperatures so that span between tested temperatures does not exceed 600°F.
- 4.4.3 Survey requirements, batch furnaces:
- 4.4.3.1 Number and location of thermocouples during initial surveys: In the furnace, thermocouples shall be placed in accordance with AMS 2750, as applicable. Thermocouples may also be attached to the furnace charges at exposed surfaces and within the charges. The number and distribution of these thermocouples shall be subject to purchaser approval (see 6.2).
- 4.4.4 Survey requirements, continuous furnaces, all gaseous atmospheres:
- 4.4.4.1 Furnace temperatures when making initial surveys: The maximum and minimum temperatures within the working zones shall not exceed those specified in 4.4.2, as applicable to the intended thermal treatment. Furnaces used for more than one kind of thermal treatment shall be surveyed at the highest and lowest anticipated service temperatures.
- 4.4.4.2 Number and locations of the thermocouples during initial surveys: Thermocouples shall be placed in the furnace in the number and locations which will enable the determination of entry-to-exit temperatures profiles at each working temperature. A minimum of two thermocouples shall be attached to each furnace charge and accompany the charge through the furnace.
- 4.4.5 Survey requirements, continuous furnaces, vacuum:

- 4.4.5.1 Number and locations of thermocouples during initial surveys: Thermocouples within the furnace shall be placed in accordance with 4.4.4.2.
- 4.4.5.2 Survey procedure: Except as otherwise specified herein, the survey procedure shall conform to 4.4.2. When the furnace charge cannot be wired with thermocouples without destroying the vacuum, the survey shall entail inspections of product after thermal treatment. Such inspections shall include, but not be limited to: tension tests (see 4.7.1), bend tests (see 4.7.2), determination of hydrogen concentration (see 4.7.3), and metallographic examinations (see 4.7.4). Such inspections shall be performed on the first lot of each product passed through a new or refurbished furnace (see 4.6.2 for lot definition).
- 4.4.6 Survey requirements, induction heating systems:
- 4.4.6.1 Thermal treatments and workpiece temperatures: Only solution heat treating, stress relieving, and annealing treatments shall be carried out by induction heating. The temperatures in 4.4.2 shall be considered as nonmandatory. Other temperatures appropriate to products to be heat treated may be selected, provided that the requirements of 3.1 are met.
- 4.4.6.2 Temperature measurement: To determine uniformity of temperature around the cross-sectional perimeter of a workpiece, a minimum of four thermocouples shall be attached around such perimeter approximately 90 degrees apart.
- 4.4.6.3 Survey procedure: Workpiece with thermocouples attached shall be passed through the induction coil at a rate and power density which will result in sufficient heating to accomplish the desired result. Temperature readings need not be taken while the thermocouple hot junctions are within the induction coil. Several workpiece passages at various rates and power densities may be needed before proper conditions can be determined.
- 4.5 Calibration:
- Calibration of equipment as specified in 4.3 shall be carried out in accordance with MIL-STD-45662.
- 4.5.1 Accuracy of furnace pyrometric systems:
- 4.5.1.1 General requirements: The accuracy of such systems shall be checked by procedures in accordance with AMS 2750, except as otherwise specified herein. The test thermocouple, test potentiometer, and cold junction compensation system shall within the previous 3 months have been calibrated against National Institute of Standards and Technology (NIST) primary or secondary certified thermocouples and potentiometers to an accuracy of  $\pm 2^{\circ}\text{F}$  ( $\pm 1^{\circ}\text{C}$ ) of NIST true temperature.
- 4.5.1.2 Special requirements: When the furnace construction is such that test thermocouples can not be placed adjacent to working thermocouples, a plan for the calibration of pyrometric systems shall be adopted by the party responsible under contract for heat treatments administered. This plan shall be subject to disapproval by the contracting activity (see 6.2).

**4.6 Sampling for product monitoring:**

Such sampling shall be for inspection for conformance to 3.1. The sampling requirements of the subparagraphs herein shall not apply to a product covered by a sampling plan within a product specification. Subject to the contracting activity's approval (see 6.2), product specification test results may be used to satisfy any one of the inspections specified in section 4.7 to demonstrate conformance to 3.1.

- 4.6.1 Unit of inspection: The unit of inspection shall be one piece of rod, bar, sheet, plate, or shape, one coil of strip, or one forged or cast semi-finished part.
- 4.6.2 Lot: A lot shall consist of a group of product units of the same heat, mechanically and thermally treated to substantially the same properties using the same pieces of equipment, such treatment being applied to the units as a batch, or to the group unit-by-unit over essentially a continuous time interval not to exceed 8 hours, and inspected at the same time.
- 4.6.3 Specimen selection: Specimens for each inspection (see 4.7) shall be selected in accordance with 4.6.3.1, 4.6.3.2, 4.6.3.3, 4.6.3.4, 4.6.3.5, 4.6.3.6, and 4.6.3.7, as applicable. Specimens of suitable dimensions shall be removed from product where configuration and dimensions permit. Where such removal is impossible, specimens shall be taken from a sample piece of appropriate dimensions and of the same heat as a product unit with which the sample is heat treated.
- 4.6.3.1 Wrought product except forged parts: Two specimens for tension testing in accordance with 4.7.1 shall be excised from each lot of such product after the final thermal treatment. In addition, from each lot of product within the thickness range specified in 3.7.2, two specimens shall be excised for the bend test specified in 4.7.2. Bars, billets, and blooms to be manufactured into forged parts shall, when specified on the drawing, have test blanks removed, forged into specimens simulating the forged parts to be made, heat treated, and tested in accordance with 4.7.1.
- 4.6.3.2 Forgings: Unless otherwise specified in product specifications or other acquisition documents, each furnace charge of raw or rough-machined forgings shall be accompanied by two blanks with comparable mechanical working from the same heat as the lot being heated. These blanks shall be suitable for machining into test specimens conforming to 4.7.1, as applicable, and be from locations complying with product specifications or other acquisition documents, as applicable.
- 4.6.3.3 Standard components: When heat treating standard components, such as nuts and bolts, for which the frequency of testing is specified, the applicable requirements of the component product specification shall take precedence.
- 4.6.3.4 Cast parts: Unless otherwise specified, each furnace load of cast parts undergoing heat treatment shall be accompanied by three tension specimens as specified in 4.7.1, poured within the same casting lot as the parts represented unless specimens from actual castings are used.
- 4.6.3.5 Induction heated product: Unless otherwise specified in acquisition documents, two blanks to be made into specimens conforming to 4.7.1, as applicable, shall be removed from the first and the last workpiece of the lot treated.

- 4.6.3.6 Sampling for analysis of hydrogen concentration: Product heated in air at temperatures equal to or less than 1000°F (540°C), or under vacuum or inert gas need not be sampled and analyzed after such treatments. From product heated above 1000°F, two specimens shall be taken from each thermally treated lot and tested in accordance with 4.7.3 for conformance to 3.6.5. When the product is strip, one specimen shall be taken from each end of a coil. The other product shall be sampled randomly.
- 4.6.3.7 Sample for metallographic examinations for surface contaminations: Product heated at temperatures equal to or less than 1000°F (540°C), need not be sampled and examined, unless otherwise specified. From product heated above 1000°F, two specimens from each thermally treated lot shall be subjected to the examination specified in 4.7.4.2 for compliance with 3.6.6. When the product is strip, one specimen shall be taken from each end of a coil. The other product shall be sampled randomly. For examinations for adequate purging (see 3.2.1.6), only one sample need be taken from the furnace charge.
- 4.7 Test methods:
- Unless other test methods are specified in other product acquisition documents, the test methods specified herein shall apply.
- 4.7.1 Tension test: Specimens selected in accordance with 4.6, as applicable, shall be prepared and tested in accordance with ASTM E 8 for conformance to 3.7.1. The rate of strain during testing shall be 0.003 to 0.007 in./in./minute until the yield strength at the designated offset has been exceeded, after which the rate of strain shall be increased so as to result in fracture within one additional minute. In case of dispute over strain rates, a strain rate of 0.005 in./in./minute shall be applied until the designated offset yield point has been exceeded, after which the crosshead speed shall be a minimum of 0.1 in./minute until fracture.
- 4.7.2 Bend test: Specimens selected in accordance with 4.6.3.1 shall be tested for conformance to 3.7.2 by applying test procedures within ASTM E 290 as modified herein. Specimen edges in the width direction shall be free of burrs. Deburring by any conventional technique is allowed. Specimen widths shall not be less than one inch (25.4 mm), nor shall specimen lengths be less than 2 inches (50.8 mm). During testing, each specimen shall be supported as a simple beam across a female die conforming to Figure 1; such die selected in accordance with the specified bend to be imparted. Each specimen shall be bent by a 3-point load resulting from the pressure of a male die centered over the female die, the male die being manufactured in accordance with Figure 1, and selected for the test in accordance with the specified bend to be imparted. Bend tests may be performed on a power brake.
- 4.7.3 Hydrogen analysis: Specimens selected in accordance with 4.6.3.6 shall be analyzed by the hot extraction method specified in ASTM E 146 to determine compliance with 3.6.5. Other methods may be used subject to the approval of the contracting activity.

#### 4.7.4 Metallographic examinations:

4.7.4.1 Determination of beta transus: When beta annealing of an alpha-beta alloy is specified (see 3.5.6), representative samples from the lot to be so annealed shall be taken for solution heat treating and quenching. Each test specimen shall be of such dimensions that its center will cool faster than the critical rate during the quench. A range of solution heat treating temperatures spanning the nominal beta transus shall be applied using a different temperature for each specimen. Following quenching, specimens for metallographic examination shall be prepared in accordance with ASTM E 3, as applicable, etched in a suitable solution, and examined at magnifications to 500X to determine the amount of primary alpha phase present. The temperature at which this phase is no longer present shall be deemed the beta transus of the lot. Such temperature may be determined by interpolation. In lieu of metallography, a beta transus may be determined by means of a differential thermal analyzer.

4.7.4.2 Metallographic examination for surface contamination by oxygen and nitrogen: Specimens selected in compliance with 4.6.3.7 shall be prepared according to ASTM E 3, as applicable, etched in a suitable solution, and examined at 200X or higher magnification to determine conformance to 3.6.6.

#### 4.8 Records:

4.8.1 Retention of inspection records: Unless otherwise specified in the acquisition documents, inspection records shall be on file for five years and shall be available for examination by the contracting activity.

4.8.2 Records of calibration: Records of calibration shall be kept for five years and shall be available for examination by the contracting activity.

4.8.3 Test results: Results of all tests required by this specification shall be retained for five years after date of performance, and shall be available for examination by the contracting activity.

4.8.4 Furnace records: Records relative to the identification and history of usage of each furnace shall be maintained as evidence of compliance with this specification. Information recorded shall include as a minimum the furnace number or description, size, temperature range of usage, type(s) of thermal treatment applied (solution heat treatment, annealing, etc.), temperature(s) at which uniformity was surveyed, dates of each survey, number and locations of thermocouples during each survey, and dates and other specifics of substantial repairs or alterations. These records shall be kept for 5 years after the date of performance or as otherwise specified in the acquisition documents.

4.8.5 Records of hydrogen outgassing treatments: During a hydrogen outgassing treatment, the working temperature, the soaking time, and absolute pressure within the furnace shall be recorded. Records shall be retained and be available for review in accordance with 4.8.1.

4.8.6 Noncompliance: If any test result fails to meet the requirements specified herein, the cause of failure shall be determined and the equipment repaired if applicable. If tests indicate improper heat treatment, the equipment and process shall not be used for heat treatment of titanium alloys until the deviation(s) is corrected and satisfactory performance is re-established. Questionable material shall be investigated, categorized as conforming or non-conforming and disposed of accordingly. Evaluation of the equipment and/or material shall be documented and the appropriate corrective action shall be taken and documented. The quality assurance organization shall notify the contracting activity of nonconformance when previously heat treated lots are suspect.

4.8.6.1 Response-to-Heat-Treatment Tests: During the 18 month period following publication of AMS-H-81200A, if samples heat treated in accordance with AMS 2801 do not meet tensile properties requirements, material may be accepted based on successful tests of additional samples heat treated in accordance with AMS-H-81200. The actual heat treatment times and temperatures and the properties obtained for both treatments shall be included in the report.

## 5. PACKAGING:

This section is not applicable to this document.

## 6. NOTES:

(This section contains information of a general or explanatory nature that may be helpful but is not mandatory.)

### 6.1 Intended use:

This specification is intended for development and control of heat treating procedures applied to titanium and titanium alloy wrought and cast product.

### 6.2 Acquisition requirements:

Acquisition documents must specify the following:

- a. Title, number, and date of this specification.
- b. Issue of DODISS to be cited in the solicitation, and if required, the specific issue of individual documents referenced (see 2.1).
- c. Use of thermal treatment parameter values other than those specified herein (see 3.5).
- d. Use of beta annealing (see 3.5.6).
- e. Frequency of periodic tests if other than 4.2.1.
- f. Approval of number and distribution of thermocouples as in 4.4.3.1.
- g. Furnace construction approval as in 4.5.1.2.
- h. Whether product specification test results may be used, as in 4.6.

### 6.3 General information:

- 6.3.1 Avoidance of contamination during thermal treatments: The following paragraphs list those means which may be adopted to avoid contamination of the heat treated product with hydrogen, oxygen, and nitrogen. Information on the reactions between these elements and titanium and its alloys may be found in MIL-HDBK-697, "Titanium and Titanium Alloys".
- 6.3.1.1 Hydrogen contamination: Such contamination of bare workpieces may be avoided by heating within either a vacuum, or an inert gas of low dew point. Avoidance may also be achieved by coating workpieces with an impermeable material. Bare workpieces should not be in contact with dissociated hydrocarbons or cracked ammonia. Contaminated workpieces may be salvaged by heating under vacuum.
- 6.3.1.2 Oxygen and nitrogen contamination: Since contamination resulting from contact with these gases during thermal treatment is confined to a thin surface layer, the contaminate may be removed by mechanical or chemical means, as applicable. If the chemical means is acid pickling, then care should be exerted to avoid hydrogen contamination. Where surface removal can be applied, considerable latitude in selecting atmospheric environments is afforded. Where removal of contaminated layers is not feasible, contamination may be minimized by either very brief contact with a contaminating atmosphere, the application of a protective coating, or heating within an inert atmosphere or vacuum.
- 6.3.2 Heat treating temperatures: The hardening heat treatments selected for the alpha-beta and beta alloys are designed to provide the best combination of strength, ductility, notch and fracture toughness. Whenever specified practice appears questionable, or if preproduction tests produce erratic results, the heat treater is advised to consult the applicable product specification to establish corrective action. Scaling of titanium and titanium alloys starts at about 1000°F (540°C). Heating above 1000°F under oxidizing conditions results in increasingly severe surface scaling as well as diffusion of oxygen. Oxygen diffusion results in a hard, brittle surface layer. If the solution heat treating temperature is below the minimum specified, complete solution is not effected and the optimum mechanical properties are not developed. The selection of solution heat treating temperatures should be guided by beta transus temperatures, nominal values of which are within Table VI.
- 6.3.3 Quenching: For the alpha-beta alloys, a rapid quenching after solution heat treating is necessary in order to meet the minimum mechanical properties.
- 6.3.4 Annealing stability: Thermal stability of the alpha-beta titanium alloys depends upon the transformation characteristics of beta phase. The slow cooling stabilizing anneal is designed to produce a stable beta capable of resisting further transformation when exposed to elevated temperature in service. In regard to other alloys, such as Ti-6Al-2Sn-4Zr-2Mo, stabilization treatment applied subsequent to the solution heat treatment achieves the required stability.

- 6.3.5 Stress corrosion: Titanium alloys are susceptible to stress corrosion by halides at temperatures above 550°F (290°C). For this reason, particular care must be taken to insure proper cleanliness, that is, absence of halogen compounds on parts heat treated or used above 550°F. Halogenated solvents should be avoided for cleaning titanium alloys. A sample list of halogenated hydrocarbons is shown below.

Halogenated hydrocarbons

Carbon tetrachloride	Methyl chloride	Trichlorofluoromethane
Trichlorotrifluoroethane	Methyl iodide	Octafluorocyclobutane
Trichloroethylene	Trichloroethane	

- 6.3.6 Rate of heating: When material, size, or design of parts, or the operating conditions are such that no cracking or excessive warpage results, the parts may be charged into the heat treating furnace at any desired temperature which does not exceed the maximum working temperature specified for the process and the material involved. Parts of complicated design having abrupt changes of section or sharp corners, and parts which have been previously hardened above 120 ksi (827 MPa), should be subcritically annealed, or preheated prior to charging into furnaces which are at or above any transformation temperature. This does not apply to aging of beta alloys. Alternatively, the furnace temperature can be lowered several hundred degrees prior to charging to avoid cracking and minimize distortion.
- 6.3.7 Soaking times: The soaking times listed within the tables are approximately correct for heating in air, in a gaseous atmosphere, or in a vacuum. The soaking times appropriate to a particular lot will vary with the composition of material, capacity of heating elements, and size of charge, as well as the thickness of the individual part. Excessive soaking times should be avoided, so as to minimize scaling, avoid surface contamination, and avoid beta grain growth when solution heat treating alpha-beta alloys.
- 6.3.8 Shape influence: Most of the published literature and the data in this specification are based on tests of round specimens of various diameters. In order to apply these data successfully to actual parts, it is convenient to visualize the parts as simple geometric shapes such as rounds, hexagons, squares, plates or tubes. These shapes can then be considered as the size round which will have approximately the same cooling rates as that of the simple shape. The relationship between the various simple shapes and the corresponding rounds is shown in Figure 2.
- 6.3.9 Metrication: Dimensions in inch/pound units and Fahrenheit temperatures are primary; dimensions in SI units and Celsius temperatures are shown as the approximate equivalents of the primary units and are presented only for information purposes.

#### 6.4 Definitions:

6.4.1 Raw material (e.g., sheet, plate, bar, forgings, castings and extruded shapes): Usually identified by a heat or lot number, is usually destructively tested for acceptance. It is heat treated, usually by or for a material producer, in accordance with a material specification which may require, by reference, conformance to a heat treating specification. It may include rough or partially machined or similarly modified parts heat treated by a material producer in accordance with the procedures and destructive testing requirements of a material specification.

6.4.2 Parts: Usually identified by a part number, are produced from raw material in accordance with the requirements of a drawing and are usually tested by nondestructive means only. They are heat treated by or for a fabricator, in accordance with a drawing, purchase order, fabrication order, or heat treatment specification. At the time of heat treatment, they may resemble raw material.

#### 6.5 Subject term (key word) listing:

Aging  
Alloy  
Anneal  
Beta transus  
Furnace  
Heat treatment  
Hydrocarbons  
Quench  
Stress-corrosion  
Stress relief  
Titanium

#### 6.6 Marginal indicia:

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 revisions only.

TABLE I. Solution heat treating schedule for raw materials and semi-finished parts 1/

Material	Solution heat treating temperature		Times at temperature or Soaking times, minutes		Cooling method g/ 10/ 10/ 2/	
	Sheet, Strip and Plate °F	Bars, Forgings and Castings °C	Sheet, Strip and Plate 10/	Bars, Forgings and Castings 10/ 2/		
Alpha Alloys						
8A1-1Mo-1V <u>6/</u>	---	1650-1850	900-1010	---	20-90 <u>3/</u>	
Alpha-Beta Alloys						
6A1-4V	1650-1775	1650-1775	900-970	2-90	20-120	Water quench
6A1-6V-2Sn <u>5/</u>	1550-1700	1550-1700	870-925	2-60	20-90	Water quench
6A1-2Sn-4Zr-2Mo	1500-1675	1650-1800	900-980	2-90	20-120	Water quench
6A1-2Sn-4Zr-6Mo	1500-1675	1500-1675	815-915	2-90	20-120	Air cooled
11Sn-5Zr-2Al-1Mo <u>6/</u>	---	1625-1675	885-915	---	20-120	Air cooled
6A1-2Sn-2Zr-2Mo-2Cr-0.25Si	1600-1700	1600-1700	870-925	2-60	20-120	Water quench
5A1-2Sn-2Zr-4Mo-4Cr <u>6/</u>	---	1450-1500	790-815	---	20-120	Water quench
Beta Alloys						
13V-11Cr-3Al <u>4/</u>	1400-1500	1400-1500	760-815	2-60	20-60	g/
3A1-8V-6Cr-4Mo-4Zr	1450-1700	1450-1700	790-925	2-60	20-90	g/
15V-3A1-3Cr-3Sn <u>7/</u>	1400-1500	---	---	2-30	20-90	3/
10V-2Fe-3Al <u>6/</u>	---	1300-1425	705-775	---	60-120	Water quench

## Notes:

- 1/ Soaking time shall be considered to begin as soon as the lowest reading control thermocouple is at the lower limit of the specified solution treating temperature range. (see 6.3.7).
- 2/ Longer soaking times may be necessary for specific forgings. Shorter soaking times are satisfactory when soak time is accurately determined by thermocouples attached to the load. Soaking time should be measured from the time the furnace charge reaches the soaking temperature (see 3.2.1.7)
- 3/ Air cool or faster.
- 4/ For material less than 0.100 inch (2.54 mm). For thickness greater than 0.100 inch, duplex solution treatment is applicable as follows, 1300°F to 1375°F (705 to 745°C) for 50 to 80 minutes, air-cooled, then 1400°F to 1450°F (760 to 790°C), 20 to 60 minutes.
- 5/ Air cooling may be applied in relatively thin sections. Water quench is required for thick sections.
- 6/ No solution heat treating cycle is specified for sheet, strip and plate.
- 7/ No solution heat treating cycle is specified for bars, forgings and castings.
- 8/ When vacuum furnace equipment is used, inert gas cooling may be applied in lieu of air cooling.
- 9/ An air cool may be applied to sections up to 0.50 inch thick (12.7 mm). A water quench shall be applied to sections greater than 0.50 inch thick.
- 10/ See paragraph 6.3.8.

TABLE II. Maximum quench delay.

Nominal thickness, inches (mm)	Maximum delay time (seconds) <u>1/</u> <u>2/</u>
Up to 0.25 (6.4)	6
0.26 (6.5) to 0.99 (25.3)	8
1.00 (25.4) and over	10

## Notes:

- 1/ Quench delay time begins when the furnace door starts to open, and ends when the last corner of the load is immersed in the quenchant.
- 2/ Times shown apply to Ti-6Al-4V and Ti-6Al-4V ELI. Alloys more beta-stabilized are more tolerant of quench delay.

SAENORM.COM : Click to view the full PDF of AMS-H-81200a

TABLE III. Aging schedule

Material	Aging temperature		Soaking times, hours 1/
	°F	°C	
<b>Alpha Alloys</b>			
8Al-1Mo-1V	1000-1150	540-620	8 - 24 <u>2/</u>
11Sn-5Zr-2Al-1Mo	900-1000	480-540	20 - 30 <u>2/ 3/</u>
<b>Alpha-Beta Alloys</b>			
6Al-4V	900-1275	480-690	2 - 8 <u>2/ 3/</u>
6Al-6V-2Sn	875-1150	470-620	2 - 10
6Al-2Sn-4Zr-2Mo	1050-1150	565-620	2 - 8 <u>2/</u>
6Al-2Sn-4Zr-6Mo	1050-1250	480-675	4 - 8
6Al-2Sn-2Zr-2Mo-2Cr-0.25Si	900-1250	480-675	2 - 10
5Al-2Sn-2Zr-4Mo-4Cr	1100-1250	480-675	4 - 8 <u>3/</u>
<b>Beta Alloys</b>			
13V-11Cr-3Al <u>4/</u>	825-975	440-525	2 - 60 <u>3/</u>
3Al-8V-6Cr-4Mo-4Zr	875-1150	470-620	2 - 24 <u>3/</u>
15V-3Al-3Cr-3Sn	900-1250	480-675	2 - 24 <u>3/</u>
10V-2Fe-3Al	900-1150	480-620	8 - 10 <u>3/</u>

## Notes:

- 1/ See note 1/, Table I, for definition of soaking time and paragraph 6.3.7.
- 2/ See Table V for duplex annealing. An 8-hour stabilizing treatment at 1050-1100°F (565-595°C) can be considered an aging treatment.
- 3/ Aging time and temperature depends on strength level desired.
- 4/ Springs may be aged at 800°F (425°C).

TABLE IV. Stress relief schedule

Material	Stress relief temperature		Soaking time, minutes 1/
	°F	°C	
Commercially pure (All grades)	900-1100	480-595	15 - 240
Alpha Alloys			
5Al-2.5Sn	1000-1200	540-650	15 - 360
5Al-2.5Sn ELI	1000-1200	540-650	15 - 360
6Al-2Cb-1Ta-0.8Mo	1000-1200	540-650	15 - 60
8Al-1Mo-1V	1100-1400	595-760	10 - 75
11Sn-5Zr-2Al-1Mo	900-1000	480-540	120 - 480
Alpha-Beta Alloys			
3Al-2.5V	700-1100	370-595	15 - 240
6Al-4V 2/	900-1200	480-650	60 - 240
6Al-4V ELI 2/	900-1200	480-650	60 - 240
6Al-6V-2Sn	900-1200	480-650	60 - 240
6Al-2Sn-4Zr-2Mo	900-1200	480-650	60 - 240
5Al-2Sn-2Zr-4Mo-4Cr	900-1200	480-650	60 - 240
6Al-2Sn-2Zr-2Mo-2Cr-0.25Si	900-1200	480-650	60 - 240
Beta Alloys			
13V-11Cr-3Al	1300-1350	480-730	30 - 60
3Al-8V-6Cr-4Mo-4Zr	1300-1400	705-760	10 - 60
15V-3Al-3Cr-3Sn	1450-1500	790-815	30 - 60
10V-2Fe-3Al	1250-1300	675-705	30 - 60

## Notes:

- 1/ See paragraph 6.3.7.
- 2/ Stress relief may be accomplished simultaneously with hot forming at temperatures of 1400°F (760°C) to 1450°F (790°C). Caution should be exercised in stress relieving titanium alloys that have been strengthened by solution treating and aging. The stress relieving temperature should not exceed the aging temperature used in heat treatment.