

Submitted for recognition as an American National Standard

**HEAT TREATMENT OF TITANIUM ALLOY PARTS**

1. SCOPE:

1.1 Purpose: This specification covers the engineering requirements for heat treatment by part fabricators (users) or their vendors or subcontractors of parts (See 1.1.2) made from the following titanium alloys.

Commercially Pure	6A1-4V(ELI)	3A1-8V-6Cr-4Mo-4Zr
3A1-2.5V	6A1-6V-2Sn	13V-11Cr-3A1
5A1-2.5Sn	6A1-2Sn-4Zr-2Mo	10V-2Fe-3A1
8A1-1Mo-1V	6A1-2Sn-4Zr-6Mo	15V-3Cr-3A1-3Sn
6A1-4V		

1.1.1 Other Alloys: This specification may be used for heat treatment of parts made from other than the above alloys provided temperatures, times, and quenchants are specified by the cognizant engineering organization.

1.1.2 Parts: Finished and semi-finished parts including raw material heat treated by the parts vendor or fabricator, or their vendors or subcontractors, during the fabrication process. (See 8.1.1).

1.2 Qualification of Raw Material: The temperature, soaking time, and cooling rate requirements specified herein are applicable for testing (by material producers, warehouses/distributors, and forge shops) for capability to respond to heat treatment (See 8.1.2) when some or all of these requirements are not included in the procurement specification.

1.3 Raw Material: Heat treatment by material producers, forge shops, and warehouses/distributors, or their vendors, of products listed below should be performed in accordance with the procurement specification. (See 8.1.2).

Sheet, Plate, Foil, Bar, Rod, Wire, Extruded Shapes, Forgings, and Castings.

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1.4 Heat Treatments: Heat treatments and their abbreviations covered by this specification are as follows:

Anneal (ANN)	Solution Treat (ST)
Stress Relief (SR)	Beta Solution Treat (BST)
Age (AGE)	Solution Treat and Age (STA)
Overage (OA)	Solution Treat and Over Age (STOA)
Cold Worked and Aged (CWA)	
Beta Solution Treated and Overaged (BSTOA)	

1.5 Safety - Hazardous Material: 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 publications shall be the issue in effect on the date of the purchase order.

2.1 SAE Publications: Available from SAE, 400 Commonwealth Drive, Warrendale, PA 15096-0001.

2.1.1 Aerospace Material Specifications:

AMS-2750 - Pyrometry  
AMS-4901 - Titanium Sheet, Strip, and Plate, Annealed,  
70,000 psi (482 MPa) Yield Strength

2.1.2 Aerospace Recommended Practices:

ARP1962 - Certification of Heat Treating Personnel

2.2 ASTM Publications: Available from ASTM, 1916 Race Street, Philadelphia, PA 19103-1187.

ASTM B 600 - Descaling and Cleaning Titanium and Titanium Alloy Surfaces  
ASTM E 146 - Chemical Analysis of Zirconium and Zirconium Alloys

2.3 U.S. Government Publications: Available from Standardization Documents Order Desk, Building 4D, 700 Robbins Avenue, Philadelphia, PA 19111-5094.

2.3.1 Federal Specifications:

BB-H-1168 - Helium, Technical

2.3.2 Military Specifications:

MIL-A-18455 - Argon, Technical  
 MIL-P-27407 - Propellant Pressurizing Agent, Helium

2.3.3 Military Standards:

MIL-STD-794 - Parts and Equipment, Procedures for Packaging and Packing of

3.1 Equipment:

3.1.1 Pyrometry: Shall conform to AMS-2750.

3.1.2 Furnaces:

3.1.2.1 Temperature Uniformity: Shall conform to the following, tested in accordance with AMS-2750:

Heat Treatment	Temperature Tolerance	
	°F	°C
Aging	±15(1)	±8(1)
Solution	±25	±14
Stress Relief	±25	±14
Anneal	±25	±14

(1) ±10°F (±6°C) for aging, at 1025°F (552°C) and below, parts made from 6Al-6V-2Sn, 13V-11Cr-3Al, 15V-3Cr-3Al-3Sn, 10V-2Fe-3Al, and 3Al-8V-6Cr-4Mo-4Zr alloys.

3.1.2.2 Heating Media:

3.1.2.2.1 Air and Non-Inert Atmospheres: Shall be free of reducing gases and other contaminants which may produce surface contamination in excess of that to be removed (See 3.2.10) or which may result in excess hydrogen pick-up (See 3.1.2.2.5). Direct fired furnaces shall be controlled so that the flame is slightly oxidizing and there is no flame impingement on the parts. A coupon (See 3.1.2.2.5) shall accompany one load each week heated above 1200°F (649°C) and be subsequently tested for excess hydrogen pick-up and for surface contamination in excess of that to be removed. Parts with net dimensions shall not be heated above 1000°F (538°C) in air or non-inert atmosphere furnaces unless coated with a protective coating (See 3.1.2.2.1.2).

3.1.2.2.1.1 Furnaces to be used above 1200°F (649°C) which have contained a contaminating atmosphere (e.g. endothermic, dissociated ammonia) shall be equipped so as to prevent leakage of the contaminating atmosphere into the working zone. Such furnaces shall be purged and tested for hydrogen pick-up before heat treating the first load of titanium parts.

- 3.1.2.2.1.2 Protective coatings may be used to avoid or minimize the detrimental effects of heating media when approved by the cognizant engineering organization.
- 3.1.2.2.2 Inert Atmospheres (Helium and Argon): Shall meet the composition requirements of MIL-P-27407, Type I, MIL-A-18455, or BB-H-1168 as applicable. The dew point of the gas shall be  $-65^{\circ}\text{F}$  ( $-54^{\circ}\text{C}$ ) or lower as it enters the furnace. For loads to be heated above  $1000^{\circ}\text{F}$  ( $538^{\circ}\text{C}$ ), containing parts having surfaces from which no material will be removed, two coupons (See 3.1.2.2.5) shall accompany each load and be subsequently tested for alpha case (See 8.4). Test coupons are not needed for heat treatments under  $1000^{\circ}\text{F}$  ( $538^{\circ}\text{C}$ ).
- 3.1.2.2.3 Vacuum: Vacuum pressure and leak rate shall be determined at room temperature before heating each load. Vacuum pressure shall be lower than  $0.1\ \mu\text{m}$  of mercury and leak rate shall be lower than  $3\ \mu\text{m}$  of mercury per one-quarter hour with the vacuum pump isolated from the furnace chamber. Cooling may be accelerated by back-filling with inert gas conforming to 3.1.2.2.2. For loads to be heated above  $1000^{\circ}\text{F}$  ( $538^{\circ}\text{C}$ ) containing parts having surfaces from which no material will be removed, two coupons (See 3.1.2.2.5) shall accompany each load and be subsequently tested for alpha case (See 8.4). Test coupons are not needed for heat treatments under  $1000^{\circ}\text{F}$  ( $538^{\circ}\text{C}$ ).
- 3.1.2.2.4 Molten Salt and Fluidized Bed: Prohibited.
- 3.1.2.2.5 Tests: Coupons of AMS-4901 composition, nominally 0.020 inch (0.51 mm) thick by 1 inch (25 mm) wide shall be used to confirm conformance with heating media requirements. Hydrogen pick-up by coupons heated in air and other non-inert atmosphere furnaces shall not exceed 25 ppm when analyzed in accordance with ASTM E 146. Coupons heat treated in vacuum or inert gas atmosphere furnaces shall be free from surface contamination determined in accordance with either the microhardness or bend test of AMS-4901.
- 3.1.3 Auxiliary Equipment: Fixtures, jigs, hangers, trays, racks, etc shall be employed as necessary. The equipment shall not reduce the heating, cooling, or quenching rates below those required for proper heat treatment.
- 3.1.4 Quench Tanks: Shall be of sufficient size to permit complete immersion of parts and free movement of the quench medium adjacent to all surfaces of parts. Equipment shall be provided for agitation or circulation of the quench medium and/or the parts. The volume of quenchant, and any auxiliary cooling equipment, shall be sufficient to maintain (1) a water quench below  $100^{\circ}\text{F}$  ( $38^{\circ}\text{C}$ ) during the quench and (2) an oil quench between  $60^{\circ}$  and  $160^{\circ}\text{F}$  ( $16^{\circ}$  and  $71^{\circ}\text{C}$ ) at the start of the quench and below  $200^{\circ}\text{F}$  ( $93^{\circ}\text{C}$ ) during a quench. In addition, quench oils shall be used within the temperature range recommended by the oil manufacturer.

### 3.2 Procedures:

- 3.2.1 Cleaning: Parts shall be cleaned, prior to heat treatment, in accordance with ASTM B 600 or other method approved by the cognizant engineering organization. Part surfaces shall be free of halogen compounds, such as residue from halogenated solvents and coolants, and salt from sweaty hands. Surfaces of parts, fixtures, racks, etc shall be clean and free of dirt, water, oil, grease, paint, ink, crayon markings, die pick-up, fingerprints, and other foreign material. After cleaning and prior to heat treatment in inert gas or vacuum furnaces, personnel handling parts shall wear clean, white cotton gloves or equivalent.
- 3.2.2 Racking: Parts shall be racked to ensure uniform heating and cooling throughout the load. Parts shall not be nested unless tests with load thermocouples have demonstrated that the arrangement will not affect uniformity of heating and cooling.
- 3.2.3 Heat Treatment: Shall be performed as specified herein unless an alternate treatment has been specified by the cognizant engineering organization. Treatments for alloys not covered herein shall be as specified by the cognizant engineering organization. In both cases, the treatment specified should include the name (e.g. anneal, age), the set temperature, the soaking time, and quench or cooling medium.
- 3.2.4 Control Instruments: Shall be set either at the set temperature specified or at an offset temperature based on the last temperature uniformity determination. The offset temperature shall be within 5°F (3°C) for aging and 10°F (6°C) for other treatments of the specified set temperature and shall be posted on the instrument. The offset temperature shall be selected to optimize the temperature distribution within the furnace so that the highest and lowest temperatures are equidistant from the set temperature. For solution heat treatment of loads without load thermocouples in air and atmosphere (inert and non-inert), furnaces shall be stabilized at the set or offset temperature before loading parts.
- 3.2.5 Start of Soaking Time: Soaking time starts when all temperature control sensors and load thermocouples (if used) reach the temperature described by the set or offset temperature minus the appropriate tolerance.
- 3.2.6 Solution Heat Treating: Shall be performed in accordance with Table I. Re-solution treatment is permitted only with the approval of the cognizant engineering organization.

TABLE I  
SOLUTION HEAT TREATMENTS

ALLOY	SET °F	TEMPERATURE °C	MINIMUM SOAKING TIME minutes (3,5)	QUENCH (2,7)
6Al-4V sheet	1725 (6)	941 (6)	15	water
6Al-4V ELI	1725 (6)	941 (6)	15	water
6Al-4V other	1750 (6)	954 (6)	15	water
6Al-6V-2Sn	1625	885	20	water
13V-11Cr-3Al	1400	760	60	(1)
3Al-8V-6Cr-4Mo-4Zr	1500	816	30	(1)
10V-2Fe-3Al (4)	Beta-50	Beta-28	60	(1)
15V-3Cr-3Al-3Sn	1450	788	30	(1)
6Al-2Sn-4Zr-2Mo	1770	966	120	(1)
8Al-1Mo-1V	1825	996	60	(1)
6Al-2Sn-4Zr-6Mo	1600	871	60	air or oil

- (1) Thickness: under 1/2 inch (12.7 mm) - Air  
1/2 to 2 inches (12.7 to 51 mm) - All forms of 8Al-1Mo-1V and 6Al-2Sn-4Zr-2Mo plus forgings of 13V-11Cr-3Al, 3Al-8V-6Cr-4Mo-4Zr, and 15V-3Cr-3Al-3Sn - Oil or water; other alloys and forms - Air, oil or water.  
Over 2 inches (51 mm) - Water
- (2) Inert gas back-fill may be substituted for air cool in vacuum and inert atmosphere furnaces
- (3) Soaking times shown are for material up to 0.10 inch (2.5 mm) thick. For thicknesses up to 1 inch (25 mm), add 1 minute to the value shown for each additional 0.1 inch (2.5 mm) of thickness. For thicknesses over 1 inch (25 mm), add 20 minutes for each 1/2 inch (12.7 mm) of thickness or fraction thereof over 1 inch (25 mm). Thickness is the minimum dimension of the heaviest section of the part. The tolerance for soaking time is plus 10%, minus 0.
- (4) The beta transus temperature will be furnished to the heat treater (See 8.1.8).
- (5) For loads without load thermocouples, at least 75% of the soaking time shall occur after recovery to within 10°F (6°C) of the original set or offset temperature. Conformance with this requirement may dictate using a longer soaking time than specified in the table.
- (6) 1900°F (1038°C) for beta solution treatment.

(7) Quench delay time (elapsed time between start of furnace door opening and completion of immersion) for oil or water quenching shall not exceed the following:

Nominal Thickness		Delay Time Seconds
Inches	Millimetres	
Up to 0.025, excl	Up to 0.64, excl	6
0.025 to 0.10, excl	0.64 to 2.5, excl	10
0.10 to 1.0, excl	2.5 to 25, excl	15
1.0 and over	25 and over	30

3.2.7 Aging: Shall be performed in accordance with Table II. Environment during cooling after aging shall be compatible with the heating environment, i.e., it shall not increase alpha case thickness.

TABLE II

AGING TREATMENTS (5)

ALLOY	TEMPER	SET TEMPERATURE		SOAKING TIME Hours (1)
		°F	°C	
6Al-4V	STA900	900	482	8
6Al-4V	STOA1300	1300	704	4
6Al-6V-2Sn	STA1000	1000	538	2.5
13V-11Cr-3Al	STA800	800	427	10
13V-11Cr-3Al	(3)CWA800	800	427	10
3Al-8V-6Cr-4Mo-4Zr	STA925	925	496	20
3Al-8V-6Cr-4Mo-4Zr	(3)CWA1000	1000	538	6
10V-2Fe-3Al (4)	STA925	925	496	8
10V-2Fe-3Al (4)	STA975	975	524	8
15V-3Cr-3Al-3Sn	STA950	950	510	8
6Al-2Sn-4Zr-2Mo	STA1100	1100	593	8
8Al-1Mo-1V	STA1100	1100	593	8
6Al-2Sn-4Zr-6Mo	STA1100	1100	593	6

(1) Soaking times shown are for material up to 0.10 inch (2.5 mm) thick. Add 20 minutes for each additional 1/2 inch (12.7 mm) or fraction thereof. The thickness is the minimum dimension of the heaviest section of the part. Tolerance for soaking time is plus 1/4 hour, minus 0.

(2) For loads without load thermocouples, at least 75% of the soaking time shall occur after recovery to within 10°F (6°C) of the original set or offset temperature. Conformance with this requirement may dictate using longer soaking time than specified in the table.

(3) Cold worked instead of solution heat treated.

(4) When no temper designation is specified, the STA 925 temper (See 8.3) shall be used.

- (5) When mechanical property requirements are specified, other aging treatments (including those used by the material producer to confirm response to heat treatment) may be used to meet requirements. If sampling and testing are not specified along with the mechanical properties, any adjustment of the time or temperature shall be validated by testing not less than two specimens from the same heat and lot of material heat treated with each lot of parts.
- 3.2.8 Stress Relieving: Shall consist of soaking for 2 hours  $\pm$  0.25 at 1100°F (593°C) and air or furnace cooling except as in 3.2.8.1 and 3.2.8.2.
- 3.2.8.1 Parts made from beta alloys (See 8.1.6) and any parts which have been solution heat treated (and not aged) shall not be stress relieved.
- 3.2.8.2 For aged parts, the stress relieving temperature shall be 50°F (28°C) below the aging temperature.
- 3.2.9 Annealing: Shall consist of soaking for 2 hours  $\pm$  0.25 at 1300°F (704°C) and air or furnace cooling except as in 3.2.9.1, 3.2.9.2 or 3.2.9.3.
- 3.2.9.1 Parts made from beta alloys and parts which have been solution treated (and not aged) shall not be annealed.
- 3.2.9.2 Parts made from 6Al-6V-2Sn alloy shall be cooled to 1000°F (538°C) at a rate of not over 300°F (167°C) per hour.
- 3.2.9.3 Parts made from 5Al-2.5Sn alloy shall be annealed for 2 hours  $\pm$  0.25 at 1500°F (816°C) and air or furnace cooled.
- 3.2.10 Descaling: Required for parts heated above 1000°F (538°C) in an environment other than an inert atmosphere or vacuum. Sufficient material shall be removed to ensure uncontaminated material on all surfaces.
- 3.2.10.1 Metal removal may be accomplished mechanically, by immersion in molten salt, by a chemical method in accordance with ASTM B 600, or by other method acceptable to purchaser. It need not be done immediately after heat treatment. It may be postponed until later in the manufacturing schedule. Table III provides an approximate guide for metal removal after heating in air.

TABLE III

RECOMMENDED METAL REMOVAL  
mils/mm

Exposure Temperature °F (°C)	Hours of Exposure						
	0.2	0.5	1	2	6	10	20
1100 (593)	-	0.3/0.01	0.5/0.01	0.5/0.01	0.5/0.01	1/0.03	2/0.05
1300 (704)	0.3/0.01	0.5/0.01	1/0.03	1/0.03	2/0.05	3/0.08	3/0.08
1400 (760)	0.5/0.01	1/0.03	1/0.03	2/0.08	3/0.08	3/0.08	6/0.15
1500 (816)	1/0.03	1/0.03	2/0.05	3/0.12	4/0.10	6/0.15	-
1600 (871)	2/0.05	2/0.05	3/0.08	5/0.13	8/0.20	10/0.25	-
1700 (927)	2/0.05	3/0.08	4/0.10	6/0.15	10/0.25	-	-
1800 (982)	3/0.08	4/0.10	6/0.15	10/0.25	-	-	-
1900 (1038)	5/0.13	8/0.20	10/0.39	-	-	-	-
2000 (1093)	6/0.15	10/0.25	-	-	-	-	-

3.3 Qualification of Vendors (Subcontractors):

- 3.3.1 Facilities performing heat treatment in accordance with this specification shall be approved by the cognizant quality assurance organization (See 4.4).
- 3.3.2 Personnel performing or directing the performance of heat treatment in accordance with this specification shall be certified in accordance with ARP1962.

4. QUALITY ASSURANCE PROVISIONS:

- 4.1 Responsibility for Inspection: Except as specified in 4.4.1, the heat treatment processor shall be responsible for the performance of all tests and inspections specified herein. The heat treatment processor may use his own facilities or any commercial laboratory acceptable to the cognizant quality assurance organization.
- 4.1.1 The procuring activity reserves the right to perform any surveillance, tests, or inspection of parts, and to review heat treatment records and results of tests and inspections to verify that the heat treatment conformed to the requirements of this specification.
- 4.1.2 Responsibility for Inspection for Direct U.S. Government Orders: The contractor shall be responsible for all inspection requirements specified herein. Except as otherwise specified herein, the contractor may use his own or any other facilities suitable for the performance of the inspection specified herein, unless disapproved by the government. The government reserves the right to perform any of the inspections set forth in the specification where such inspections are deemed necessary to ensure conformance to prescribed requirements.

4.2 Records: Records shall be available to purchaser for not less than five years after heat treatment. The records shall contain all data necessary to verify conformance to the requirements of this specification.

4.3 Sampling and Testing: Not applicable.

4.4 Approval of Vendors (Subcontractors): By quality assurance organizations; will normally be based on the following:

4.4.1 Approval of the vendor's shop procedure document which must include a full description of all equipment and procedures that will be used to meet the requirements of this specification and AMS-2750.

4.4.2 Competence of vendor's personnel.

4.5 Furnace Log and Recorder Chart Entries:

4.5.1 Each furnace log entry or equivalent documentation such as a shop traveler, shall be signed by approved personnel (See 4.4.2) and shall include not less than the following:

Load number  
Part number  
Shop order number(s)  
Number of parts  
Type of material and alloy designation  
Equipment identification and furnace number  
Thickness of parts (See 8.1.5)  
Solution heat treating, aging, etc, temperatures  
Part time at temperature  
Dewpoint or other atmosphere control parameter, as applicable  
such as vacuum furnace leak rate  
Quench media or cooling method  
Surface contamination test results performed in accordance with AMS-4901  
Date.

4.5.2 Furnace temperature recorder charts shall include the following information for each load:

Load number  
Shop order number(s)  
Time loaded (with AM, PM noted)  
Time soaking commences  
Verification of alignment of recorder chart with the scale  
Approved personnel's identification  
Furnace number  
Date.