

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

1.1.1 Other Alloys: This specification may be used for heat treatment of parts made from alloys other than the above, 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 fabricators, or their vendors or subcontractors, during the fabrication process. (See 8.2.2.)

1.2 Qualification of Raw Material:

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

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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 8.2.1.)

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

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)	

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.

AMS 2750	Pyrometry
AMS 4901	Titanium Sheet, Strip, and Plate, Annealed, 70.0 ksi (483 MPa) Yield Strength

ARP1962	Certification of Heat Treating Personnel
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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 1477	Determination of Hydrogen in Titanium and Titanium Alloys by the Inert Gas Fusion Thermal Conductivity Method

2.3 U.S. Government Publications:

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

BB-H-1168 Helium, Technical

MIL-A-18455 Argon, Technical

ML-P-27407 Propellant Pressurizing Agent, Helium

MIL-STD-2073-1 DoD Materiel, Procedures for Development and Application of, Packaging Requirements

3. TECHNICAL REQUIREMENTS:

3.1 Equipment:

3.1.1 Pyrometry: Shall conform to AMS 2750.

3.1.2 Furnaces:

3.1.2.1 Temperature Uniformity: Shall be as shown in Table 1.

TABLE 1 - Temperature Tolerance

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

¹ +10°F (t₆ °C) for parts made from the following five alloys if temperature is 1025 °F (552 °C) or lower: 6Al-6V-2Sn, 13V-11Cr-3Al, 15V-3Cr-3Al-3Sn, 10V-2Fe-3Al, and 3Al-8V-6Cr-4Mo-4Zr.

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 °F (-54 °C) or lower as it enters the furnace. For loads to be heated above 1000 °F (538 °C), containing parts having surfaces from which no material will be removed, one coupon (see 3.1.2.2.5) shall accompany each load and be subsequently tested for alpha case (see 8.5). Test coupons are not needed for heat treatments under 1000 °F (538 °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 μ m of mercury and leak rate shall be lower than 3 μ 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 °F (538 °C) containing parts having surfaces from which no material will be removed, one coupon (see 3.1.2.2.5) shall accompany each load and be subsequently tested for alpha case (see 8.5). Test coupons are not needed for heat treatments under 1000 °F (538 °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 1447. 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 or a metallographic technique approved by the cognizant quality assurance organization.
- 3.1.2.2.5.1 For heat treat loads containing small parts (e.g., fastener components) such parts may be substituted for the coupons specified in 3.1.2.2.5.
- 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 °F (38 °C) during the quench, (2) a polymer quench below 120 °F (49 °C), and (3) an oil quench between 60 and 160 °F (16 and 71 °C) at the start of the quench and below 200 °F (93 °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 perspiration. 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: (R)

3.2.2.1 Parts, other than rivets, bolts, nuts, and other small parts, shall be racked to ensure uniform heating and cooling throughout the load. These parts shall not be nested unless tests with load thermocouples (1) have established the necessary additional soaking time required and (2) have demonstrated that the arrangement will not affect uniformity of heating and cooling.

3.2.2.2 Rivets, bolts, nuts, and other small parts, with maximum thickness of 0.5 inch (13 mm), may be racked as in 3.2.2.1, or heated and soaked in baskets or continuous furnaces. When processed in baskets, maximum thickness of layers and minimum space between layers shall be 1 inch (25 mm). When processed in continuous furnaces, parts shall not be layered.

3.2.2 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.

3.2.4.1 The posting of offset temperatures shall preclude misinterpretation by (R) specifying both the "desired" temperature and the corresponding "set" temperature (e.g., "When 700 °F is desired, set at 704 °F").

3.2.5 Start of Soaking (See 8.2.10) Time: Shall be determined by one of the (R) following methods:

3.2.5.1 Batch Furnaces - Method 1: When the furnace temperature, as shown by the (R) controlling indicating or recording instrument(s), reaches the set or offset temperature.

3.2.5.2 Batch Furnaces - Method 2: When the furnace temperature, as shown by the (R) controlling indicator or recording instrument(s), reaches the minimum of the applicable range (see 3.2.5.1.1).

3.2.5.2.1 The minimum of the applicable range is the temperature described by the (R) set or offset temperature minus the tolerance specified for furnace temperature uniformity.

3.2.5.2.2 When Method 2 is used, at least 75% of soaking time shall be after the (R) furnace temperature has reached the half-tolerance temperature (See 3.2.5.1.2.1).

3.2.5.2.2.1 The half-tolerance temperature is the temperature described by the (R) set or offset temperature minus half of the tolerance specified for furnace temperature uniformity.

3.2.5.3 Batch Furnaces - Method 3: When the temperature of at least two load (R) sensors in contact with parts reaches the minimum of the applicable range (see 3.2.5.2.1).

3.2.5.4 Batch Furnaces - Method 4: When the temperature of at least two load (R) sensors in contact with parts, positioned so as to reflect the temperature at the center of the coldest parts (see 3.2.5.4.1), reaches the half-tolerance temperature (see 3.2.5.2.2.1). If this method is used, the soaking time may be reduced to that shown in Table 2 for 0.10 inch (2.5 mm) thickness.

3.2.5.4.1 The coldest parts are those in the coldest portion of the furnace as (R) shown by the last temperature uniformity test.

- 3.2.5.5 Continuous Furnaces: Soaking time starts when parts enter the zone of (R) the furnace shown by the last temperature uniformity test to be within the range described by the set temperature and the applicable tolerance.
- 3.2.6 Solution Heat Treating: Shall be performed in accordance with Table 2. Re-solution treatment is permitted only when approved by the cognizant engineering organization.
- 3.2.7 Aging: Shall be performed in accordance with Table 4. Environment during cooling after aging shall be compatible with the heating environment, i.e., it shall not increase alpha case thickness.
- 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 8.2.6) 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) degrees per hour.

(R) TABLE 2 - Solution Heat Treatments

Alloy	Set Temperature °F	Set Temperature °C	Minimum Soaking Time Minutes (3,5)	Quench (2,7)
6Al-4V sheet	1725 (6)	941 (6)	15	water or polymer
6Al-4V ELI	1725 (6)	941 (6)	15	water or polymer
6Al-4V other	1750 (6)(7)	954 (6)	15	water or polymer
6Al-6V-2Sn	1625	885	20	water or polymer
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	60	(1)
8Al-1Mo-1V	1825	996	60	(1)
6Al-2Sn-4Zr-6Mo	1600	871	60	(1)

- (1) a. For thicknesses under 0.5 inch (12.7 mm) - Air cool or faster.
 b. For thicknesses from 0.5 to 2 inches (12.7 to 51 mm) - Air, oil, polymer, or water except (1) air shall not be used for 8Al-1Mo-1V or 6Al-2Sn-4Zr-2Mo and (2) air shall not be used for forgings of 13V-11Cr-3Al, 3Al-8V-6Cr-4Mo-4Zr, or 15V-3Cr-3Al-3Sn.
 c. For thicknesses 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 (see 8.2.10) shown are for parts 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 minus 0, plus 100%.

(4) The beta transus temperature will be furnished to the heat treater (see 8.2.8).

(5) Start of soaking time shall conform to 3.2.5.

(6) 1900 °F (1038 °C) for beta solution treatment.

(7) When mechanical property requirements are specified (e.g., as is usual for fasteners), an alternate set temperature within the range of 1650 to 1775 °F (899 to 968 °C) and oil quenching may be used. If sampling and testing are not specified along with the mechanical properties, the use of the alternate temperature shall be validated by testing not less than two specimens, heat treated with each lot of parts, from the same heat and lot of material.

(8) Quench delay time (elapsed time between start of furnace door opening and completion of immersion) for oil or water quenching shall not exceed the time as shown in Table 3.

TABLE 3 Quench Delay Time

Nominal Thickness Inches	Nominal Thickness Millimeters	Delay Time Seconds
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

(R) TABLE 4 Aging Treatments (5)

Alloy	Temper (4)	Set Temperature °F	Set Temperature °C	Soaking Time Hours (1)
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	CWA800(3)	800	427	10
3Al-8V-6Cr-4Mo-4Zr	STA925	925	496	20
3Al-8V-6Cr-4Mo-4Zr	CWA1000(3)	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 (see 8.2.10) 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 shall be plus 0.5 hour or 5%, whichever is greater than zero.
- (2) Start of soaking time shall conform to 3.2.5.
- (3) Cold worked instead of solution heat treated.
- (4) When no temper designation is specified, the STA 925 temper (See 8.4) 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

3.2.9.3 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 5 provides an approximate guide for metal removal after heating in air.

TABLE 5 - Recommended Metal Removal
mils/mm

Exposure Temperature °F (°C)	Hours of Exposure 0.2	Hours of Exposure 0.5	Hours of Exposure 1	Hours of Exposure 2	Hours of Exposure 6	Hours of Exposure 10	Hours of Exposure 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	2.0/0.05	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 in accordance with ARP1962 or other established procedures acceptable to purchaser.

3.3.2 Personnel performing or directing the performance of heat treatment in (R) accordance with this specification shall be approved in accordance with ARP1962 or other established procedures acceptable to purchaser.

4. QUALITY ASSURANCE PROVISIONS:

4.1 Responsibility for Inspection:

Except as specified in 4.1.1, 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 Furnace Log and Recorder Chart Entries:

4.4.1 Each furnace log entry or equivalent documentation such as a shop traveler, shall be signed by approved personnel (See 8.2.11.2) 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 8.2.5)
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

4.4.2 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

4.5 Reports:

The heat treatment processor shall furnish with each shipment a report showing the results of tests to determine conformance to the technical requirements and stating that the parts were processed in accordance with specified requirements. This report shall include the purchase order number, AMS 2801A, shop order number, part number or product identification, alloy designation, identification of furnace(s) used, load numbers(s), thermal processing temperatures, times and cooling cycles used, date(s), and quantity of parts.

5. PREPARATION FOR DELIVERY:

5.1 Identification:

Identification of parts provided to the heat treatment processor shall be maintained on the parts at delivery.

5.2 Packaging:

Parts shall be packaged to ensure protection from damage during shipment and storage.

5.2.1 Packages of parts shall be prepared for shipment in accordance with commercial practice and in compliance with applicable rules and regulations pertaining to the handling, packaging, and transportation of the parts to ensure carrier acceptance and safe delivery.

5.2.2 For direct U.S. Military procurement, packaging shall be in accordance with (R) MIL-STD-2073-1, Level C, unless Level A is specified in the request for procurement.

6. ACKNOWLEDGMENT:

The heat treatment processor shall mention this specification number and its revision letter in all quotations and when acknowledging purchase orders.

7. REJECTIONS:

Parts not meeting the requirements of this specification, or to modifications authorized by the cognizant engineering organization, will be subject to rejection and shall be submitted for disposition in accordance with purchaser's procedures for nonconformance.

B. NOTES:

8.1 The (R) symbol 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. If the symbol is next to the specification title, it indicates a complete revision of the specification.