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MSFC-SPEC-469 February 23, 1968

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## GEORGE C. MARSHALL SPACE FLIGHT CENTER

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

#### SPECIFICATION

## TITANIUM AND TITANIUM ALLOYS,

#### HEAT TREATMENT OF

This specification has been approved by the George C. Marshall Space Flight Center (MSFC) and is available for use by MSFC and associated contractors.

1. SCOPE

1.1 This specification covers the requirements for the heat treatment of titanium and titanium alloy products including annealing, solution treatment, aging, stress-relief, and the thermal aspects of elevated temperature forming and sizing.

2. APPLICABLE DOCUMENTS

2.1 The following documents form a part of this specification to the extent specified herein. Unless otherwise indicated, the issue in effect on the date of invitation for bids or request for proposals shall apply.

STANDARDS

Federa1

FED-STD-151

Metals; Test Methods.

PUBLICATIONS

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NPC 200-3

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(Copies of specifications, standards, drawings, and publications required by contractors in connection with specific procurement functions should be obtained from the procuring activity or as directed by the contracting officer.)

2.2 <u>Other publications</u>. - The following documents form a part of this specification to the extent specified herein. Unless otherwise indicated, the issue in effect on the date of invitation for bids or request for proposals shall apply.

American Society for Testing and Materials

ASTM E 120-66

Methods for Chemical Analysis of Titanium and Titanium Base Alloys.

(Application for copies should be addressed to the American Society for Testing and Materials, 1916 Race Street, Philadelphia, Pennsylvania 19103.)

REQUIREMENTS

3.1 Equipment.

3.1.1 <u>Heating media</u>. - Unless otherwise specified, titanium and titanium alloy products shall be heat treated in air, inert atmosphere, or vacuum. The use of salt bath media is prohibited.

3.1.1.1 Oxidizing atmosphere. - Titanium and titanium alloy products that are heat treated in any oxidizing atmosphere at a temperature greater than 1100 degrees Fahrenheit (°F) shall be coated as specified in 3.2.3. Coatings shall not be present during straightening and forming operations (see 3.5).

3.1.1.2 <u>Inert atmosphere</u>. - The dewpoint of inert atmospheres such as argon or helium shall be minus 65 °F or lower.

# 3.1.2 Heating.

3.1.2.1 <u>Furnace types</u>. - Titanium and titanium alloy parts shall be heat treated in electric, fuel-fired muffle, or sealed retort furnaces. Equipment used for steel heat treatment may be used, provided that provisions are made to prevent contaminating the work load by hydrogen and all products of combustion.

3.1.2.2 <u>Temperature control</u>. - The maximum temperature variation of all points in the working zone of the furnace shall not exceed  $\pm 10$  °F at operating temperatures below 1400 °F and  $\pm 25$  °F at operating temperatures above 1400 °F.

3.1.2.3 Accuracy of furnace control instruments. - The accuracy of temperature measuring and controlling instruments shall be checked as specified in 4.3 at regular intervals or upon request of the procuring activity representative. The intervals at which the temperaturemeasuring instruments shall be checked are dependent upon the type of thermocouple, working temperature, frequency of operation, and furnace atmosphere, but shall not be more than 30 days. If instruments are replaced or not used during a 30-day interval, they shall be checked before use.

3.1.2.4 <u>Temperature-measuring and recording equipment</u>. - Automatic controlling and recording instruments shall be used. Temperature sensing elements should be located in or as close as possible to the working zone. They shall be in such a location as to give accurate measurement of the working or sealing, or both, zone temperatures. When tested as specified in 4.3.2, the temperature recordings shall be within  $\pm 5$  °F of the temperature indications of the calibrating equipment.

3.1.3 Quenching.

3.1.3.1 <u>Water circulation and cooling</u>. - All quenching shall be in water that is circulated and cooled, if necessary, to meet the temperature requirements of 3.1.3.2. Make-up water shall be added to prevent build-up of concentration of dissolved salts in the quench tanks.

3.1.3.2 <u>Temperature control</u>. - The water temperature at the beginning of the quench shall not exceed 100 °F and its temperature shall not exceed 140 °F at any time during the quenching operation. The water temperature indications shall have an accuracy of  $\pm 2$  °F.

3.1.3.3 Quench delay. - The maximum quench delay shall not exceed 4 seconds for parts having a thickness of 0.091 inch or less in its thinnest section; 5 seconds for parts having a thickness greater than 0.091 inch but less than 0.500 inch in its thinnest section. For parts in which the section thicknesses are greater than 0.500 inch, the maximum delay shall not exceed 7 seconds.

3.1.4 <u>Miscellaneous equipment</u>. - Fixtures, jigs, sizing tools, trays, racks, and quenching presses shall be used as required to prevent and correct warpage, distortion, unequal heating, and unequal quenching. This equipment shall be fabricated from material that will not contaminate the titanium or titanium alloys during heat treatment.

# 3.2 Preparation for heat treatment.

3.2.1 <u>Cleanliness</u>. - The surfaces of all titanium and titanium alloy products shall be visibly free from all contaminants, including fingerprints, dirt, flux, lubricants, scale, water, or any foreign material, before being placed in the furnace for heat treatment. Fixtures, jigs, sizing tools, trays, and racks directly in contact with the work shall also be free from these contaminants. Precautions shall be taken to prevent contamination of the furnace atmosphere. The use of chlorinated hydrocarbons such as trichloroethylene or carbon tetrachloride, and methyl alcohol for solvent cleaning prior to heat treatment is prohibited.

3.2.2 <u>Scale removal</u>. - Titanium or titanium alloy products with scale shall have the base metal removed to a minimum depth specified in table I below all microscopic evidence of contamination before heat treatment operations.

3.2.3 <u>Coatings</u>. - Coating material shall be used on all titanium and titanium alloy parts heat treated in air or in oxidizing atmospheres at temperatures greater than 1100 °F (see 3.1.1.1). These coatings are to be used for one heat treatment only, and shall be applied on surfaces conforming to 3.2.1. Coating thickness should not be thicker than is necessary to protect the surface to which it is applied. Use either inhibitor or barrier (ceramic) type coatings but not both. Coatings shall not be used on titanium or titanium alloy parts heat treated in vacuum or inert atmospheres. Parts should be handled with care to avoid abrading or damaging the coating.

3.2.3.1 <u>Coating removal</u>. - Coating shall be removed by any process that prevents embrittlement and selective corrosive attack of the titanium metal substrate.

3.2.4 <u>Process approval</u>. - All cleaning, descaling, pickling, and precoating processes and materials shall require the approval of the procuring activity.

	Minimum metal removal				
Nominal diameter or	Bar, billet,		Die	Sheet	
distance between opposite faces	rod, and hand forgings	Extrusions	forgings or plate		
(inches)	(inches)	(inches)	(inches)	(inches)	
0.000 to 0.500 incl.	0.016	0.015	0.010	0.008	
0.501 to 1.000 incl.	0.023	0.015	0.020		
1.001 to 1.500 incl.	0.032	0.015	0.020		
1.501 to 2.000 incl.	0.032	0.015	0.032		
2.001 to 2.500 incl.	0.063		0.032		
2.501 to 3.000 incl.	0.063		0.032		
3.001 to 4.000 incl.	0.094		0.045		
4.001 to 6.000 incl.	0.125		0.045		

Table I. Contaminated surface removal.

# 3.3 Charging.

3.3.1 <u>Sizes and configurations of parts</u>. - Parts of different size and configuration shall not be solution treated in the same load unless it has been established that the lightest section in the load will not receive excessive surface contamination as a result of the longer heat-up time required for the heavier sections.

3.3.2 <u>Racking</u>. - Parts shall be racked or suspended in such a manner to allow free circulation of heat transfer and quenching media.

3.3.3 <u>Coupons</u>. - When process control coupons are used, they shall be selectively located within the load at top, middle, and bottom to be representative of the heating characteristics of the load.

# 3.3.4 Furnace temperature.

3.3.4.1 <u>Air atmosphere</u>. - When the heat media is air, the furnace shall be at the temperature specified for the alloy being heat treated before placing the material in the furnace.

3.3.4.2 <u>Vacuum or inert gas atmosphere</u>. - When the heat media is vacuum or inert gas, the furnace shall be at room temperature when the material to be heat treated is placed in the furnace. The time required to reach operating temperature is not specified because of the variability of furnace loads and heating capabilities, but should be minimum.

3.3.4.3 <u>Recovery and heat-up temperatures</u>. - During recovery or heat-up, the heating transfer media and metal temperature shall not exceed the maximum specified for the alloy being heat treated.

3.3.5 Furnace time.

3.3.5.1 <u>Recovery time</u>. - Recovery time shall not exceed 35 minutes after charging.

3.3.5.2 <u>Heat-up time</u>. - Heat-up times are not specified because of variability of furnace loads, material thickness, and furnace heating capabilities.

3.3.5.3 <u>Hold time</u>. - Hold time shall begin when all of the parts within the load, including ones of varying thickness, have reached the temperature specified.

3.4 Heat treatments.

3.4.1 Solution heat treatment.

3.4.1.1 <u>Temperatures and times</u>. - Titanium alloy products, which can be solution heat treated, should be solution treated within the temperature ranges specified in table II. When ususual circumstances make compliance with table II impractical, thus, subject to the procuring activity approval, temperatures may be adjusted to obtain desired properties.

3.4.1.2 <u>Quenching</u>. - Titanium alloy products, except for alloy 13V-11Cr-3AL less than 2 inches thick which may be air cooled, shall be quenched by total immersion in water. Water circulation and cooling temperature control and quench delay shall be as specified in 3.1.3.

7

	Heat treating temperatu °F	emperature	Soaking time minutes (See note 2.)			
、 Alloy	Sheet, strip, and plate	Bars and forgings	Sheet, strip, and plate	Bars and forgings		
13V-11Cr-3AL	1400 - 1500 See note 3.	1400 - 1500 See note 3.	15 - 30	30 - 60		
7AL-4Mo	1675 <b>-</b> 1775	1675 <b>-</b> 1775	30 - 90	30 - 120		
6AL-4V	1600 <b>-</b> 1725	1675 - 1750	5 - 30	15 - 60		
6AL-6V-2Sn	1600 - 1700	1600 - 1700	15 - 30	30 - 60		
4AL-3Mo-1V	1620 - 1660	1700 - 1750	10 - 30	60 - 120		

Table II. Solution heat treating schedule. (See note 1.)

## NOTES:

- 1. See 3.4.6.
- 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 all furnace control instruments indicate recovery to the minimum process range.
- 3. Synonymous with annealing.

3.4.2 <u>Aging heat treatment</u>. - Aging schedules for titanium alloy products are specified in table III. The charge should be held at the temperature range for a sufficient period of time, depending upon section size, for the necessary aging to take place and to ensure that the specified properties will be developed. When unusual circumstances make compliance with table III impractical, thus, subject to the procuring activity approval, temperatures may be adjusted to obtain the desired properties. Titanium alloy products shall be air cooled from the aging temperature.

MSFC-SPEC	2-469	9
February	23,	1968

Alloy	Aging temperature °F	Soaking time hours
13V-11Cr-3AL	850 - 950	2 - 60 See note 2.
7AL-4Mo	975 - 1200	4 - 8
6AL-4V	920 - 1050	4 - 6
6AL-6V-2Sn	875 - 1125	4 - 8
4AL-3Mo-1V	900 - 950	6 - 12 See note 3.

Table III. Aging schedule. (See note 1.)

NOTES:

1. See 3.4.6.

2. Time depends on strength level desired.

3. For intermediate strength age 1050 °F to 1150 °F for 1/6 to 6 hours, using shorter time at the higher temperatures.

3.4.3 <u>Complex annealing treatment</u>. - Recommended annealing timetemperature cycles for treatment of titanium alloys, where solution treatment and aging is not recommended, are given in table IV. Complex annealing follows normal mill annealing (furnace cooling) and involves air cooling from the annealing cycle temperatures. The time at temperature depends upon section size.

3.4.4 <u>Stress-relieving treatment</u>. - Recommended time-temperature cycles for stress-relieving, fully annealed structural parts to remove cold work and restore compressive yield strength are specified in table V. The time at temperature depends upon section size and amount of cold work in the material. The products shall be air cooled from the stress-relieving temperature.

Table IV.	Complex annealing schedule.	
	(See note 1.)	

Alloy	Annealing temperature °F	Treating time hours
8AL-1Mo-1V Sheet-single annealed condition	1435 - 1465 See note 2.	1 - 8
Sheet - duplex annealed condition See note 3.	1435 - 1465 See note 2. Followed by 1435 - 1465 See note 4.	1 - 8 1/4 - 1
Bar and forging - Duplex annealed	1650 - 1850 Followed by 1000 - 1125 See note 5.	3/4 - 1 1/4 8 - 24

NOTES:

- 1. See 3.4.6.
- 2. Furnace cooled to 700 °F, then air cooled.
- 3. For good creep resistance combined with high toughness.
- 4. Air cool.
- 5. For minimum distortion 1650 °F to 1675 °F, 50 to 65 minutes followed by 1100 °F to 1125 °F, 7 to 9 hours. For high creep resistance 1825 °F to 1850 °F, 50 to 65 minutes, followed by 1100 °F to 1125 °F, 7 to 9 hours.

3.4.4.1 Weldments. - The selected time-temperature cycle for stressrelief of weldments depends on the initial heat treat condition of the welded titanium metals. Titanium welded in the solution treated condition shall be stress-relieved by aging as recommended in table III. For titanium welded in the aged condition, selection of the stress-relief cycle shall be based on maximum reduction of residual stress commensurate with achieving mechanical property requirements specified in the related procurement documents or detailed in applicable drawing and purchase orders.

3.4.4.2 <u>Titanium alloys in the aged condition</u>. - The selected timetemperature cycle for stress-relief shall be based on maximum reduction of residual stress commensurate with achieving mechanical property requirements specified in the related procurement documents or detailed in applicable drawings and purchase orders.

Alloy	Stress-relieving temperature °F	Stress relief time hours
Commercially pure	1000 - 1100 975 - 1000 880 - 925 780 - 825	1/3 - 2/3 1/2 - 1 2 - 4 7 - 8
5AL-2.5Sn and 5AL-2.5Sn ELI	1175 - 1200 1090 - 1125 990 - 1025	$\frac{1}{4} - \frac{1}{2}$ $\frac{3}{4} - 2$ $\frac{4}{2} - 6$
8AL-1Mo-1V	1075 - 1125 See note 2. 1300 - 1450 See note 3.	3/4 - 1 1/4 1/6 - 1/2
13V-11Cr-3AL	900 - 1000 See note 4.	1/4 - 60
7AL-4Mo	900 - 1300 See note 5.	1/2 - 8
6AL-4V and 6AL-4V ELI	900 - 1200 See note 6.	1/2 - 50
6AL-6V-2Sn	1000 - 1100	1/2 - 3
4AL-3Mo-1V	900 - 1100	1/2 - 8

Table V. Stress relief schedule. (See note 1.)

NOTES:

- 1. See 3.4.6.
- 2. Stress relief depends upon annealing cycle employed. For single annealed sheet (see table IV).
- 3. Stress relief for duplex annealed sheet (see table IV).
- 4. During aging. If aging is not employed, heat treat at 1050 °F to 1150 °F for 1/2 to 2 hours. Solution treatment (table II) may be synonymous with stress relief annealing for this alloy.
- 5. Use highest practical temperature.
- For 100-percent relief, 1000 °F for 50 hours or 1200 °F for 5 hours. For 50-percent relief, 1000 °F for 5 hours or 1100 °F for 1/2 hour.

3.4.5 <u>Annealing treatment</u>. - Recommended time-temperature cycles for annealing titanium and titanium alloy products are specified in table VI. The soaking period to obtain full annealing for maximum ductility will vary depending on the section thickness being annealed.

Alloy	Annealing temperature °F	Annealing time hours
Commercially pure	1000 - 1300	1/2 - 2 See note 2.
5AL-2.5Sn and 5AL-2.5Sn ELI	1325 - 1550	1/4 - 4 See note 2.
8AL-1Mo-1V	1430 - 1470 See note 3.	8
13V-11Cr-3AL	1430 - 1470 See note 4.	1/4 - 1
7AL-4Mo	1425 - 1450 See note 5. See note 6.	1 - 8
6AL-4V	1275 - 1550 See note 5. See note 7.	1/2 - 8
6AL-4V ELI	1275 - 1550 See note 8.	1/2 - 8
6AL-6V-2Sn	1300 - 1500	2 - 3 See note 2.
4AL-3Mo-1V	1225 - 1250 See note 5.	2 - 4

Table VI. Annealing schedule. (See note 1.)

NOTES:

1. See 3.4.6.

2. Air cool.

3. For sheet. For bar and forgings, anneal 1650 °F to 1850 °F for 1 hour, with a water quench (see 3.1.3) or air cooling, followed

NOTES: (continued)

by heating at 1050 °F to 1100 °F for 8 hours with air cooling. See table IV (complex annealing) for annealing condition to increase toughness and creep resistance.

- 4. Solution treatment (see table II) is suggested for annealing.
- 5. Slow cooling to 1000 °F to 1050 °F in furnace at a rate not greater than 300 °F per hour.
- This is for maximum formability. For maximum creep properties, heat treat after the annealing treatment for 24 hours at 1000 °F to 1050 °F.
- 7. For sheet, heat treat 1300 °F to 1350 °F, 1 hour, cooled at a rate of not greater than 50 °F per hour to 800 °F. Air cooling may be used for lower ductility requirements. For bar and forgings, heat treat at 1275 °F to 1325 °F, for 2 hours and air cool. For hydrogen removal by vacuum annealing, heat treat at 1300 °F to 1500 °F, for 1/2 to 2 hours, then furnace cool to 1100 °F maximum.
- 8. Air cool from temperature. For bar and forgings, heat treat at 1275 °F to 1325 °F for 2 hours and air cool. For hydrogen removal by vacuum annealing, heat treat at 1300 °F to 1500 °F, for 1/2 to 2 hours, then furnace cool to 1100 °F maximum.

3.4.6 <u>Time and temperature adjustments</u>. - When circumstances make compliance with the requirements of these tables questionable for heat treatment, the time and temperatures may be adjusted to develop the desired properties of titanium and titanium alloys specified in the related procurement documents for titanium materials or detailed in applicable drawings and purchase orders.

3.5 Straightening and forming operations.

3.5.1 Straightening.

3.5.1.1 <u>Annealed parts</u>. - Annealed parts shall not be straightened after stress relieving. After cold forming and prior to stress relieving, annealed parts may be straightened by cold forming or hot sizing. Hot formed parts shall be straightened by cold working or additional cold forming and stress relieving or hot sizing.

3.5.1.2 <u>Solution treated parts</u>. - Solution treated parts shall be straightened by cold working prior to aging, or simultaneous hot sizing and aging.

3.5.1.3 <u>Solution treated and aged parts</u>. - Straightening of solution treated and aged parts shall be achieved by hot sizing during the applicable stress relief cycle.

3.5.2 <u>Elevated temperature forming and sizing</u>. - All of the requirements stated for straightening shall apply to fabrication by sizing, forming and stress relieving, or aging during sizing and forming. However, lubricants shown to be free of chlorides and surface contaminants may be used.

3.5.2.1 <u>Equipment for hot forming and sizing</u>. - Resistance heating or heated dies may be used for hot forming, provided time and temperature controls meet the requirements of this specification. Heating by the application of a flame directly to the part is prohibited. Equipment shall be provided so that the temperature of the part may be controlled and accurately determined during sizing and forming. Temperature indicating crayons or other potential contaminants shall not be used.

3.5.2.2 <u>Hot forming and sizing</u>. - Parts may be hot formed at the temperature given for stress relieving in table V. Use shortest time practical to limit surface contamination.

3.5.2.3 <u>Simultaneous aging and sizing</u>. - Solution treated parts may be sized during the aging cycle. Aging in the hot sizing fixture for the entire aging cycle is not required; parts may be cooled and reheated to complete aging.

3.5.3 <u>Cold forming</u>. - Parts may be cold formed at any temperature below the specified hot forming temperatures. When cold forming at temperatures above 800 °F, the time shall be limited to 1 hour.

3.5.4 <u>Surface condition</u>. - The surface condition of metal prior to performance straightening and forming operations shall be of sufficient quality that substrate cracking does not occur under any conditions.

3.6 Contamination.

3.6.1 Carbide and oxygen layer.

3.6.1.1 <u>Removal of scale</u>. - Scale due to heat treatment shall be completely removed from all surfaces of finished parts. Contamination shall also be removed from all surfaces before secondary fabrication such as forming or welding. Hydride descaling followed by nitric

hydrofluoric pickling is the preferred method. If scale is heavy in some areas and light in others, it is advisable to descale by mechanical abrasion (sand/shot blast) prior to hydride descaling. The descalingpickling process as well as metal removal by pickling must be controlled to minimize hydrogen content. Hydrogen pick-up shall be monitored in combination with the heat treatment, by specimens tested in accordance with 4.5.2. Abrasive cleaning by chloride free vapor honing is an alternate method of contamination and metal removal. This method may be used provided acceptable control of uniformity in material removal is achieved and the surface finish does not exceed 35 roughness height rating (RHR).

3.6.1.2 Minimum depth of base metal removal. - Base metal below the contaminated layer (oxygen rich alpha layer) shall be removed from all surfaces of the parts after solution treatment or annealing. Pickling is the preferred method for the minimum metal removal provided it is controlled and monitored for hydrogen pick-up in combination with descaling and heat treatment. A minimum of 0.002-inch base metal shall be removed from all surfaces which were stress relieved or annealed in air above 1300 °F for 1/2 hour. For parts heat treated for more than 1/2 hour in air above 1550 °F, the minimum removal of base metal shall be 0.003 inch. Metallographic examination of 100X to determine the depth of scale and oxygen rich alpha layer shall be performed for each thermal treatment process, atmosphere, and material. Where the depth of the scale and oxygen rich alpha layer exceeds 0.001 inch, base metal removal shall be three times the layer depth.

3.6.2 <u>Hydrogen.</u> - When analyzed as specified in 4.5.2, the total hydrogen content of the metal after all heat treating, forming, and associated cleaning and pickling operations shall not exceed the amount specified in table VII, and the hydrogen pick-up shall not exceed 40 parts per million.

3.6.2.1 <u>Rejected loads</u>. - Furnace loads that have been rejected due to failing to meet the requirements of 3.6.2 may be salvaged by vacuum annealing in accordance with the time and temperature requirements of table VI.

3.6.2.2 Hydrogen content determination. - After vacuum annealing the hydrogen content of the load shall be determined by selecting test specimens as specified in 4.4 and analyzing them as specified in 4.5.2. If the test specimens are taken from process control coupons, the coupons shall have a hydrogen content representative of the hydrogen content of the rejected load. The coupons shall be included with the load as specified in 3.3.3.

		Hydrogen (parts per million)								
Alloy	Sheet	Strip	Plate	Bar	Forging	Billet	Extrusion	Wire	Tubing	
Commercially pure	150	150	150	125	125	100	125	150	150	
5AL-2.5Sn	200	200	200	200	200	175	150	150	-	
5AL-2.5SN ELI	200	200	200	200	200	175	150	150	-	
8AL-1Mo-1V	150	150	150	125	125	100	125	125	-	
13V-11Cr-3AL	200	200	200	175	175	·· 175	-	-	-	
7AL-4Mo	-	-	125	125	-	100	125	125	-	
6AL-4V	150	150	150	125	125	100	125	150	-	
6AL-4V ELI	150	150	150	125	125	100	125	150	125	
6AL-6V-2Sn	150	-	150	150	150	125	150	125	-	
4AL-3Mo-1V	150	150	150	-	-	-	-	-	-	

Table VII. Maximum allowable hydrogen content.

3.6.2.3 <u>Reheat treatment</u>. - When the hydrogen content of a rejected load has been reduced to a level less than the limit specified in 3.6.2, the material shall be reheat treated to the desired condition.

# 3.7 Mechanical characteristics.

3.7.1 <u>Tensile properties</u>. - When tested as specified in 4.6.1, the tensile strength, yield strength, and elongation based on a gage length of 2 inches or 4 diameters, as applicable, shall be as specified in the applicable procurement document or in the contract or order (see 6.3).

3.7.2 <u>Bend test properties</u>. - When tested as specified in 4.6.2, the heat-treated specimens shall show no evidence of cracking or metal separation.

## 4. QUALITY ASSURANCE PROVISIONS

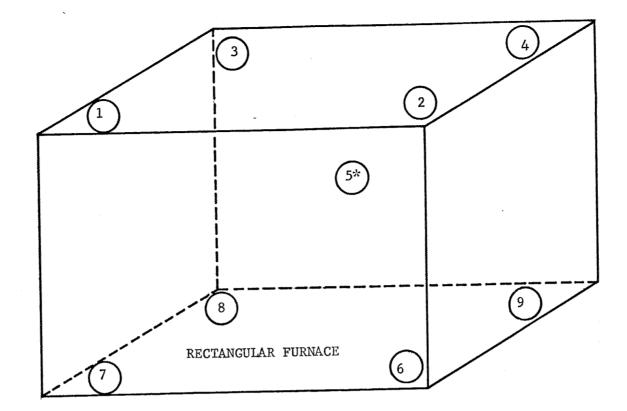
4.1 <u>Responsibility for inspection</u>. - Unless otherwise specified in the contract or the purchase order (see 6.3), the supplier is responsible for the performance of all tests and inspection requirements as specified herein. Except as otherwise specified, the supplier may utilize his own facility or any commercial laboratory acceptable to MSFC. The procuring activity or its designated representative reserves the right to perform any or all of the inspections set forth in the specification where such inspections are deemed necessary to ensure supplies and services conform to the prescribed requirements.

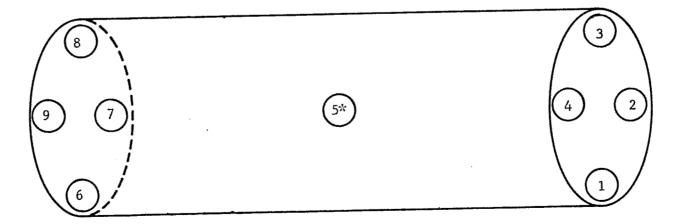
4.2 <u>Quality control</u>. The supplier shall establish and maintain an inspection system which conforms to the applicable requirements of NASA Quality Publication NPC 200-3.

4.3 <u>Temperature survey</u>. - A temperature survey shall be made at the highest and lowest temperatures of the operating range of the furnace to determine conformance to 3.1.2.4. Temperature surveys shall be performed before the first use of the furnace in heat treating titanium or titanium alloys and as specified in 3.1.2.4 thereafter.

4.3.1 <u>Survey procedure</u>. - Test temperature sensing elements shall be placed at the extremities and the center of the operating zone of the furnace. Nine sensing elements shall be used for heating zones that are 150 cubic feet or smaller. The distribution of the sensing elements is shown in figure 1. For heating zones larger than 150 cubic feet, an additional test sensing element is required for each 15-cubic foot increase in size. The additional sensing elements shall be located to provide the best coverage of the larger heating zone. Before thermal equilibrium is reached, none of the temperature readings shall exceed maximum temperature of the range being surveyed. After thermal equilibrium has been reached, temperature readings shall be recorded for at least 1/2 hour after the furnace reaches the operating temperature (high and low range) and shall be continued until the recurrent temperature pattern has been established. The maximum temperature variation shall not exceed that stipulated in 3.1.2.2.

4.3.2 <u>Temperature recording equipment calibration</u>. - The accuracy of temperature recording equipment shall be checked under operating conditions. The check shall be made by inserting a calibrated test temperature-sensing element adjacent to furnace temperature-sensing element and comparing the temperature indications of the two elements. (Check for conformance to 3.1.2.4.)





\* Located at approximate furnace center.

Figure 1. Location of temperature-sensing elements.

4.3.3 <u>Calibration</u>. - Before use the temperature indicating test equipment shall be calibrated against Bureau of Standards certified temperature-sensing elements. Temperature readings shall be within  $\pm 1$  °F of the indications of the calibrating equipment.

4.4 <u>Test specimens</u>. - Specimens used to determine the degree of contamination or mechanical characteristics shall be taken from each load of heat-treated products or from coupons heat treated with the load.

4.4.1 <u>Specimens taken from heat-treated products</u>. - For each test using specimens taken from the heat-treated products, one specimen shall be taken from an item heat treated at the top of the load, one from the center, and one from the bottom of the load.

4.4.2 Process control coupons.

4.4.2.1 <u>Material</u> - <u>Process</u> control coupons shall be of the same material composition and average thickness as the products being heat treated and shall be representative of the same heat and lot as the product being heat treated.

4.4.2.2 <u>Dimensions</u>. - Process control coupons shall be of such dimensions and shape that tensile and bend test specimens can be machined from the coupons which will conform to the requirements of 4.6.1.1 and 4.6.2.1.

4.4.2.3 Loading. - Process control coupons shall be positioned with the load as specified in 3.3.3.

4.4.2.4 <u>Processing</u>. - Process control coupons shall accompany the heat-treat load throughout all annealing, solution treating, aging, stress relieving, and pickling operations.

4.5 Contamination.

4.5.1 <u>Carbide and oxygen layer</u>. - Three specimens from each load shall be examined metallographically to determine depth of the oxygen diffused layer or free carbide.

4.5.2 <u>Hydrogen</u>. - Hydrogen content after all processing shall be determined by vacuum fusion analysis, using the method covered in ASTM E 120-66. The hydrogen pick-up shall be determined by comparing the final content with the content before processing.

4.6 Mechanical testing.

4.6.1 <u>Tensile testing</u>.

4.6.1.1 <u>Specimens</u>. - Three test specimens, selected as specified in 4.4, shall be tested to determine the tensile properties of the heattreated titanium or titanium alloy products. Specimens from bars, wire, or extrusions shall be taken so that the longitudinal axis is parallel to the major axis of the product form. Tensile specimens for flat products (plate, sheet, and strip) shall be taken so that the longitudinal axis is perpendicular to the direction of rolling (transverse specimen). For forgings, specimen location and direction shall be as stated in the applicable procurement documents (see 6.3).

4.6.1.2 <u>Test procedure</u>. - Tensile testing shall be conducted in accordance with Method 211.1 of Standard FED-STD-151, except that the strain rate shall be approximately 0.005 inch per inch per minute through the yield strength, and then increased to produce failure in approximately 1 minute. (Check for conformance to 3.7.1.)

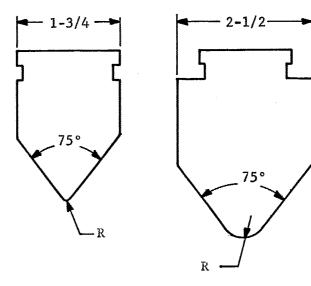
4.6.2 Bend test.

4.6.2.1 <u>Specimens</u>. - Three test specimens, selected as specified in 4.4, shall be tested to determine the bend test properties of the heat-treated titanium or titanium alloy products. Specimens shall be machined to the form and thickness dimensions in accordance with Method 231 of FED-STD-151, except the width shall not be less than 1 inch and the length shall not be less than 2 inches.

4.6.2.2 <u>Test apparatus</u>. - The bend test apparatus shall consist of one or more dies with a 75-degree V-slot, similar to that shown in figure 2, and matching punches of various radii at the tips of the punches. This equipment may be used on sheet, plate, or strip forms of titanium and titanium alloy products of various thickness. The use of power break equipment is optional. The male die shall have a radius equivalent to that specified in the applicable procurement specifications, detail drawings, or in the contract or order (see 6.3). The female die shall be of the channel type.

4.6.2.3 <u>Test procedure</u>. - The specimen shall be supported as a simple beam and bent by pressure of a rounded edge of the required radius applied, using a testing speed of 2 to 3 inches per minute. After the specimen has been bent through the full 105 degrees, the bend area shall be examined at 10X magnification. (Check for conformance to 3.7.2.)

MALE 105° CLOSED "V" DIE



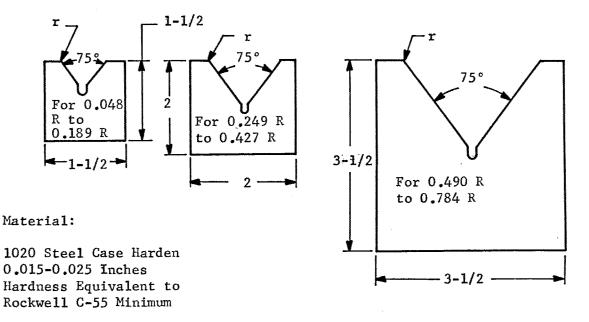
All dies to be at least 2-1/2"long

Material:

1020 Steel, Case Harden 0.015-0.025 Inches Hardness Equivalent to Rockwell C-55 Minimum

R = Bend Radius

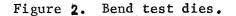
FEMALE 105° CLOSED "V" DIE



r - .010" Min Radius to Prevent Galling and Binding

R = Bend Radius

Dimensions in inches



4.6.3 <u>Rejected loads</u>. - If any specimen fails to pass the tensile tests (see 4.6.1) or the bend test (see 4.6.2), the heat-treat load from which the specimen was selected shall be rejected. Rejected loads may be reheat treated at the discretion of the procuring activity.

5. PREPARATION FOR DELIVERY

The requirements of Section 5 are not applicable to this specification.

6. NOTES

6.1 <u>Intended use</u>. - The heat-treating processes covered by this specification are intended for use in the heat treatment of titanium and titanium alloys.

- 6.2 Definitions.
  - (a) Annealing is a heat treatment performed to provide ductility for subsequent operations.
  - (b) Solution heat treatment is performed before aging and to obtain a condition suitable for aging.
  - (c) Aging is a heat treatment performed subsequent to solution treatment to attain high strength resulting from the precipitation of a second phase.
  - (d) Stress relieving is performed to relieve stresses resulting from sizing, forming, machining, welding, quenching, or mechanical working.
  - (e) Quench delay time is the time lapse between initial opening of the furnace door, and complete immersion of the part in the quenching media.
  - (f) Recovery time is the time lapse between insertion of the load in the furnace and return of the furnace temperature to the operating control temperature.
  - (g) Heat-up time is the time required for the work load to reach operating control temperature.

- (h) Holding time is the time at a given temperature necessary to accomplish the required heat treatment. Hold time varies with the alloy and the required heat treatment.
- (i) Hot sizing is the heating of parts in presses or fixtures to make minor changes in shape so that the part will conform to blue print tolerances.
- (j) Simultaneous hot sizing and aging is the heating of a part held in sizing fixtures for the required aging time-temperature cycle in order to simultaneously age the alloy and size the part to the finish contour.
- (k) Hydrogen pick-up is the increase in hydrogen content of heat-treated products due to heat treatment or pickling processes.

6.3 Ordering data. - Procurement documents should specify the following:

- (a) Title, number, and date of this specification.
- (b) Where the required tests and examinations will be performed if other than the supplier's facilities (see 4.1).
- (c) Radius of bend test die (see 4.6.2.2).
- (d) Tensile properties required (see 3.7.1).
- (e) Direction and location of test specimens for forgings (see 4.6.1.1).

<u>Notice</u>. - When Government drawings, specifications, or other data are used for any purpose other than in connection with a definitely related Government procurement operation, the United States Government thereby incurs no responsibility nor any obligation whatsoever; and the fact that the Government may have formulated, furnished, or in any way supplied the said drawings, specifications, or other data, is not to be regarded by implication or otherwise as in any manner licensing the holder or any other person or corporation, or conveying any rights or permission to manufacture, use, or sell any patented invention that may in any way be related thereto.

(Suggested changes should be directed to the Chairman, MSFC Specification Coordination Board, R-P&VE-VNR, Marshall Space Flight Center, Alabama 35812.)

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