

N 66-10673

FACILITY FORM 602

(ACCESSION NUMBER)

295
(PAGES)

(THRU)

(CODE)

19
(CATEGORY)

(NASA CR OR TMX OR AD NUMBER)

NASA-CR-54761

NASA

(Potassium Corrosion Test Loop Development
Topical Report No. 3)

**MATERIAL SPECIFICATIONS FOR ADVANCED
REFRACTORY ALLOYS**

GPO PRICE \$ _____

CFSTI PRICE(S) \$ _____

Hard copy (HC) 6.00

Microfiche (MF) 1.50

53 July 65

by

D.N. Miketta

and

R.G. Frank

prepared for

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

Contract NAS 3-2547

**SPACE POWER AND PROPULSION SECTION
MISSILE AND SPACE DIVISION**

GENERAL  ELECTRIC

CINCINNATI, OHIO 45215

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POTASSIUM CORROSION TEST LOOP DEVELOPMENT

TOPICAL REPORT NO. 3

MATERIAL SPECIFICATIONS FOR ADVANCED REFRACTORY ALLOYS

by
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Prepared for
NATIONAL AERONAUTICS AND SPACE ADMINISTRATION
Contract NAS 3-2547

October 1, 1965

Technical Management
NASA-Lewis Research Center
Space Power System Division
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SPACE POWER AND PROPULSION SECTION
MISSILE AND SPACE DIVISION
GENERAL ELECTRIC COMPANY
CINCINNATI, OHIO 45215

FOREWORD

The work described herein was performed by the General Electric Company under the sponsorship of the National Aeronautics and Space Administration under Contract NAS 3-2547. The purpose was the preparation of comprehensive material specifications for the procurement of advanced refractory alloys for evaluation in boiling and condensing potassium environments which simulate projected space electric power systems. Although the specifications found herein contain the General Electric Company's designation, anyone who so desires are encouraged to use the contents of the prepared specifications and assign their own specification numbers.

The material specifications were prepared by Mr. D. N. Miketta (deceased) under the direction of Mr. R. G. Frank, Manager, Physical Metallurgy, Materials and Processes. This work was administered for the General Electric Company by Dr. J. W. Semmel, Jr., Manager, Materials and Processes. Mr. E. E. Hoffman, Manager, Corrosion Technology, acts as Program Manager of the Advanced Refractory Alloy Corrosion Loop Program which will evaluate the material performance. Messieurs T. A. Moss and R. L. Davies are the Technical Managers for the National Aeronautics and Space Administration.

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I. INTRODUCTION

A forced circulation, two phase boiling and condensing potassium loop testing program is being conducted under NASA Contract NAS 3-6474 to evaluate an advanced refractory alloy containment material and candidate turbine materials for application in space electric power systems. A prototype of the test loop has been designed, constructed with the Cb-1Zr containment alloy and Mo-TZM turbine alloy, and operated under NASA Contract NAS 3-2547. The advanced alloy test facility includes two loops; lithium will be heated by direct resistance in the primary loop and will be used in a heat exchanger to boil potassium in the secondary corrosion test loop. Heat rejection for condensation in the secondary loop will be accomplished by radiation in a high-vacuum environment. The compatibility of the selected materials will be evaluated at conditions representative of space electric power system operating conditions, e.g.:

1. Boiling temperature - 2050°F
2. Superheat temperature - 2150°F
3. Condensing temperature - 1400°F
4. Subcooling temperature - 1000°F
5. Mass flow rate - 40 lb/hr
6. Boiler exit vapor velocity - 50 ft/sec
7. Average heat flux in boiler
 - Plug (0 to 18 inches) - 250,000 BTU/hr ft²
 - Helix (18 to 250 inches) - 5,000 BTU/hr ft²

One of the primary requirements in the selection and qualification of candidate materials is the ability to procure the necessary mill products to specifications that will assure the utilization of only high quality products with uniform and reproducible properties. This report covers the preparation of material specifications for four candidate alkali metal containment alloys and two candidate turbine alloys. The candidate containment alloys selected by NASA are FS-85 and D-43 columbium alloys and T-111 and T-222 tantalum alloys. The candidate turbine alloys are Cb-132M columbium alloy and Mo-TZC molybdenum alloy.

II. PREPARATION OF MATERIAL SPECIFICATIONS

Tentative material specifications were prepared for the procurement of (1) bar and rod, (2) sheet, plate and strip and (3) seamless tubing and pipe for the four candidate containment alloys, D-43, FS-85, T-111 and T-222, and bar and rod for the two candidate turbine alloys, Cb-132M and Mo-TZC. Preparation of the tentative specifications for the four candidate containment alloys was based primarily on information gained from the past experience of the materials producers; preparation of the tentative specifications for the two turbine alloys was based primarily on experience at General Electric. The following material producers and laboratories were visited by GE personnel prior to the preparation of the tentative specifications for the containment alloys; those alloys on which information was obtained are indicated:

<u>Company</u>	<u>Alloy</u>			
	<u>D-43</u>	<u>FS-85</u>	<u>T-111</u>	<u>T-222</u>
E.I. duPont de Nemours & Company, Incorporated	X	-	-	-
Fansteel Metallurgical Corporation	X	X	X	X
Metals Division, National Research Corporation	-	-	X	X
Stellite Division, Union Carbide Corporation	X	X	X	X
Westinghouse Electric Corporation, Astronuclear Laboratory	-	-	X	X

Upon completion of the tentative specifications, inquiries were forwarded to the material producers requesting preliminary price and delivery information to fulfill the procurement requirements for the Corrosion Loop Program as well as soliciting comments and recommendations with respect to the tentative specifications. Generally, the response from the material producers was most gratifying, and it was apparent that the majority of the producers invested considerable time and effort in attempting to establish mutually acceptable specifications. Suggested changes that were made by the material producers can be categorized in the following sections of the specifications:

- Chemical Composition
- Mechanical Properties

Tensile
Stress-Rupture

- Grain Size
- Dimensional Tolerances
- Ultrasonic Inspection Procedures and Requirements

A comparison of the chemical composition established in the tentative specification, recommended revisions in the chemical composition made by the material producers and the final revision in the chemical composition incorporated in the specification can be made in Tables I through VI. Similarly, comparisons of the specified room temperature tensile properties can be made in Tables VII through X, stress-rupture properties in Tables XI through XIV and grain size in Table XV. In making the final revisions to the specifications, considerable emphasis was placed on recommendations received from the originators of the alloy. Limits for stress-rupture properties were based on data supplied by NASA. Also, it was desirable to arrive at specifications that are amenable to at least two suppliers for each alloy.

No changes were made from the tentative specifications in the dimensional tolerances or ultrasonic inspection procedures and requirements. The majority of the producers requested changes in dimensional tolerances that were convenient for their own equipment and the tolerances established for the tentative specifications appeared to be as suitable as any that were proposed. The suggested changes in ultrasonic procedures were unacceptable for technical reasons.

After the final revisions were made to the specifications, inquiries were sent to the material producers again for final price and delivery quotations. A summary of the response received from the material producers for the containment alloys is given in Table XVI. In the case of FS-85 and T-111 alloys, a minimum of two major producers quoted to the specifications with no exceptions; only one major producer quoted T-222 and D-43 alloys to the specifications without taking exceptions. No quotations were received without exception to the specifications for the turbine alloys Cb-132M and Mo-TZC; a summary of the response received from the material producers for the turbine alloys is given in Table XVII.

With the selection of T-111 alloy for the primary material of construction for the corrosion loop and both Cb-132M and Mo-TZC alloys for the turbine simulator, purchase orders were placed with the following producers:

<u>Alloy</u>	<u>Producer</u>	<u>Exceptions to Specifications</u>
1. T-111	Fansteel Metallurgical Corporation	1. No exceptions
2. Cb-132M	Universal Cyclops Steel Corporation	2. H to be 10 ppm maximum instead of 5 ppm maximum

<u>Alloy</u>	<u>Producer</u>	<u>Exceptions to Specifications</u>
3. Mo-TZC	Climax Molybdenum Company of Michigan	3. No guarantee of mechanical properties
4. Mo-TZC	GE-Lamp Metals & Com- ponents Department	4. Single vacuum arc melt, No guarantee of properties, C range 0.10 - 0.15%, Ti range 1.15 - 1.55%, Zr range 0.13 - 0.23%

The quantity of material ordered and delivery schedule are given in Table XVIII.

TABLE I. RECOMMENDED REVISIONS TO THE SPECIFIED CHEMICAL COMPOSITION OF D-43 (Cb-10W-1Zr-0.1C) COLUMBIUM ALLOY

	Ingot Chemistry										Final Product Analyses									
	Element										Element, ppm									
	ppm																			
	W	Zr	C	O	N	H	Ta	Mo	Ni	Co	Fe	Cb	C	O	N	H	C	O	N	H
	Min.	Max.	Min.	Max.	Min.	Max.	Min.	Max.	Min.	Max.	Min.	Max.	Min.	Max.	Min.	Max.	Min.	Max.	Min.	Max.
Tentative Specification	9	11	0.75	1.25	800	1200	100	50	100	300	200	50	50	50	50	50	50	50	50	50
Recommended Revisions by Material Producer																				
DuPont	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
Fansteel	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
Wah Chang	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
Stellite	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
Universal Cyclops	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
Superior (Tubing Only)	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
Final Revisions Incorporated in Specifications	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-

(1) For Foil Less than 0.010 Inch Thick and for Wire Less than 0.030 Inch Diameter, the Oxygen Content Shall be Less than 300 ppm.

(2) For Tube with Less than 0.020 Inch Wall Thickness, the Oxygen Content Shall be Less than 400 ppm.

TABLE II. RECOMMENDED REVISIONS TO THE SPECIFIED CHEMICAL COMPOSITION OF FS-85 (Cb-28Ta-10.5W-0.9Zr) COLUMBIUM ALLOY

	Ingot Chemistry												Final Product Analyzes																							
	Element												Element, ppm																							
	W/O						ppm						C						O						N						H					
	Min.	Max.	Min.	Max.	Min.	Max.	Min.	Max.	Min.	Max.	Min.	Max.	Min.	Max.	Min.	Max.	Min.	Max.	Min.	Max.	Min.	Max.	Min.	Max.	Min.	Max.	Min.	Max.	Min.	Max.	Min.	Max.				
Ta																																				

(1) For Foil Less than 0.010 Inch Thick, for Wire Less than 0.030 Inch Diameter and for Tube with Less than 0.020 Inch Wall Thickness the Carbon Content Shall be Less than 150 ppm.

(2) For Foil Less than 0.010 Inch Thick, for Wire Less than 0.030 Inch Diameter and for Tube With Less Than 0.020 Inch Wall Thickness the Oxygen Contents Shall be Less than 300 ppm.

TABLE III. RECOMMENDED REVISIONS TO THE SPECIFIED CHEMICAL COMPOSITION OF T-111 (Ta-8W-2Hf) TANTALUM ALLOY

INDEX III. RECOMMENDED REVISIONS

		Ingot Chemistry										Final Product Analyses										
		Element										Element, ppm										
W/O		Hf		C		O	N	H	Cb	Mo	Ni	Co	Fe	V	Ta	C		O	N	H		
Min.	Max.	Min.	Max.	Max.	Max.	Max.	Max.	Max.	Max.	Max.	Max.	Max.	Max.	Max.	Max.	Max.	Var.	Max.	Max.	Var.	Max.	
7	9	2.0	2.8	50	100	50	10	300	200	50	50	50	50	20	Bal.	50	+10	150	+20	75	+10	+2
Tentative Specification																						
Recommended Revisions by Material Producer																						
Westinghouse																						
-	-	1.8	2.4	30	30	30	-	-	-	-	-	-	-	-	-	-	-	100	+20	50	+10	-
Wah Chang																						
-	-	-	3.0	-	-	-	-	750	-	-	-	-	-	-	-	-	-	-	-	-	-	-
Stellite																						
-	-	-	-	-	-	-	100	1000	-	-	-	-	-	-	-	-	-	-	-	-	-	-
Fansteel																						
-	-	-	-	-	-	-	-	1000	500	-	-	-	-	-	-	-	-	-	-	-	-	-
Universal Cyclops																						
-	-	-	-	-	-	-	-	1000	-	-	-	-	-	-	-	-	-	-	-	-	-	-
Superior (Tube Only)																						
-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	100	-	200	-	100	-	15
Final Revisions Incorporated in Specifications																						
-	-	1.8	2.4	-	-	-	-	1000	-	-	-	-	-	-	-	(1)	-	(2)	-	(3)	-	-

(1) For Foil Less than 0.010 Inch Thick, for Wire Less than 0.030 Inch Diameter and for Tube with Less than 0.020 Inch Wall Thickness, the Carbon Content Shall be Less than 75 ppm.

(2) For Foil Less than 0.010 Inch Thick, for Wire Less than 0.030 Inch Diameter and for Tube with Less than 0.020 Inch Wall Thickness, the Oxygen Content Shall be Less than 300 ppm.

(3) For Foil Less than 0.010 Inch Thick, for Wire Less than 0.030 Inch Diameter and for Tube with Less than 0.020 Inch Wall Thickness, the Nitrogen Content Shall be Less than 100 ppm.

TABLE IV. RECOMMENDED REVISIONS TO THE SPECIFIED CHEMICAL COMPOSITION OF T-222 (Ta-10.4W-2.4Hf-0.01C) TANTALUM ALLOY

Ingot Chemistry												Final Product Analyses																						
Element												Element, ppm																						
W/O		Hf		C		O		N		H		Cb		Mo		Ni		Co		Fe		V		Ta		C		O		N		H		
Min.	Max.	Min.	Max.	Min.	Max.	Min.	Max.	Min.	Max.	Min.	Max.	Min.	Max.	Min.	Max.	Min.	Max.	Min.	Max.	Min.	Max.	Min.	Max.	Min.	Max.	Min.	Max.	Min.	Max.	Min.	Max.	Min.	Max.	
Tentative Specification																																		
9	10	2.2	2.8	100	150	100	100	50	10	10	300	200	50	50	50	50	50	50	20	20	Bal.	100	150	+10	150	+20	75	+10	10	+2				
Recommended Revisions by Material Producer																																		
Westinghouse																																		
9.6	11.2	-	-	80	200	30	30	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	80	200	-	100	-	50	-	10	-	
Wah Chang																																		
-	11	2	3	75	-	-	-	-	-	-	750	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	
Stellite																																		
-	-	-	-	50	300	-	100	-	1000	-	1000	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	
Fansteel																																		
-	-	-	-	80	200	-	-	-	1000	500	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	80	200	-	-	-	-	-	-	
Universal Cyclops																																		
-	-	-	-	-	-	-	-	-	-	-	1000	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	
Final Revisions Incorporated in Specifications																																		
9.6	11.2	-	-	80	175	-	-	-	-	-	1000	-	-	-	-	-	-	-	-	-	-	-	-	-	-	80	175	-	(1)	-	(2)	-	-	

- (1) For Foil Less than 0.010 Inch Thick, for Wire Less than 0.030 Inch Diameter and for Tube with Less than 0.020 Inch Wall Thickness, the Oxygen Content Shall be Less than 300 ppm.
- (2) For Foil Less than 0.010 Inch Thick, for Wire Less than 0.030 Inch Diameter and for Tube with Less than 0.020 Inch Wall Thickness, the Nitrogen Content Shall be Less than 100 ppm.

TABLE V. RECOMMENDED REVISIONS TO THE SPECIFIED CHEMICAL COMPOSITION OF Mo-TZC (Mo-1.25Ti-0.15Zr-0.12C) ALLOY

	Ingot Chemistry										Final Product Analyses									
	Element					ppm					Element, ppm									
	W/o					C					C									
	Mo Min. Max.	Ti Min. Max.	Zr Min. Max.	H Min. Max.	N Min. Max.	O Min. Max.	Si Min. Max.	W Min. Max.	Fe Min. Max.	Cr Min. Max.	Sn(1) Min. Max.	Pb Min. Max.	Ta Min. Max.	Cb Min. Max.	V Min. Max.	Min. Max.	Var. Max.	Min. Max.	Var. Max.	H Min. Max.
Mo-TZC Alloy																				
Tentative Specification	98.5	1.20	1.30	0.13	0.18	1400	1700	20	10	5	120	80	80	25	40	10	100	100	100	100
Recommended Revisions by Material Producer																				
Climax	Bal.	1.1	1.4	0.25	0.40	800	1200	-	-	-	-	-	-	-	-	-	-	-	-	-
GE-IMCD	Bal.	(2)	-	(3)	-	1000	1500	-	-	-	-	-	-	-	-	-	-	-	-	-
Final Revisions In- corporated in Speci- fications	-	-	-	-	-	1100	1400	-	-	-	-	-	-	-	-	-	-	-	-	-

(1) Ni, Cu, Al, Ca, Mn, Mg and Co - 20 ppm Maximum - No Revision.

(2) Require 0.4 W/o Spread.

(3) Require 0.1 W/o Spread.

TABLE VI. RECOMMENDED REVISIONS TO THE SPECIFIED CHEMICAL COMPOSITION OF Cb-132M (Cb-20Ta-15W-5Mo-2Zr-0.13C) ALLOY

	Ingot Chemistry												Final Product Analysis																	
	Element												Element, ppm																	
	W/Q						C						O						N						H					
	Cb	Min.	Max.	Ta	Min.	Max.	W	Min.	Max.	Mo	Min.	Max.	Zr	Min.	Max.	C	Min.	Max.	O	Min.	Max.	N	Min.	Max.	H	Min.	Max.	Var.		
Cb-132M Alloy																														
Tentative Specification	Bal.	-	18.5	21.5	13.5	16.5	4.5	5.5	1.75	2.25	1100	1500	100	50	5	50	50	50	1100	1500	±50	200	+20	100	+10	5	5	+2		
Recommended Revisions by Material Producer																														
Universal Cyclops	-	-	-	-	-	-	-	-	-	-	-	-	-	-	10	-	-	100	-	-	-	-	-	-	-	-	-	-		
DuPont	-	-	-	-	-	-	-	-	-	-	-	-	-	-	20	200	100	200	-	-	-	-	-	-	-	-	-	-		
Final Revisions Incorporated in Specifications	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-		

TABLE VII. RECOMMENDED REVISIONS TO THE SPECIFIED ROOM TEMPERATURE TENSILE
PROPERTIES OF D-43 (Cb-10W-1Zr-0.1C) ALLOY

Tentative Specification	Ultimate Strength, ksi		0.2% Yield Strength, ksi		Elongation, %	
	Min.	Max.	Min.	Max.	Min.	Max.
Recommended Revisions by Vendor	70	90	50	70	20	
DuPont (1,2)	-	Delete	-	Delete	15	
Wah Chang	-	Delete	-	Delete	10	
Stellite (3)	-	-	-	-	-	
Universal Cyclops	-	-	-	-	-	
Fansteel (4)	-	-	-	-	-	
Final Revisions Incorporated in Specification for Bar	-	Delete	-	Delete	15	
Final Revisions Incorporated in Specifications for Sheet and Tubing	-	-	-	-	15	

- (1) For Sheet 0.010 to 0.050 Inch Thick Only.
(2) For Rod 1.0 Inch in Diameter and Under Only.
(3) No Guarantee, Information Only.
(4) Requested Deletion of Maximum Values for Bar and Rod.

TABLE VIII. RECOMMENDED REVISIONS TO THE SPECIFIED ROOM TEMPERATURE TENSILE PROPERTIES OF THE FS-85 (Cb-28Ta-10.5W-0.9Zr) ALLOY

	Ultimate Strength, ksi		0.2% Yield Strength, ksi		Elongation, %	
	Min.	Max.	Min.	Max.	Min.	Max.
Tentative Specification	70	90	50	70	20	
Recommended Revisions by Vendor						
DuPont(1)	-	Delete	-	Delete	-	
Wah Chang	-	Delete	-	Delete	10	
Stellite(1)	-	-	-	-	-	
Universal Cyclops	75	95	60	80	-	
Fansteel(2)	-	100	-	80	-	
Final Revisions Incorporated in Specifications	-	100	-	80	-	

(1) No Guarantee, Information Only.

(2) Requested Deletion of Maximum Values for Bar and Rod.

TABLE IX. RECOMMENDED REVISIONS TO THE SPECIFIED ROOM TEMPERATURE TENSILE PROPERTIES OF THE T-111 (Ta-8W-2Hf) ALLOY

	Ultimate Strength, ksi		0.2% Yield Strength, ksi		Elongation, %	
	Min.	Max.	Min.	Max.	Min.	Max.
Tentative Specification	95	110	85	100	20	
Recommended Revisions by Vendor						
Westinghouse	80	-	65	-	-	
Wah Chang	90	Delete	80	Delete	-	
NRC	80	-	70	-	-	
Stellite (1)	-	-	-	-	-	
Universal Cyclops	-	-	-	-	-	
Fansteel (2)	85	-	75	-	-	
Final Revisions Incorporated in Specifications	80	-	65	-	-	

(1) No Guarantee, Information Only.

(2) Requested Deletion of Maximum Values for Bar and Rod.

TABLE X. RECOMMENDED REVISIONS TO THE SPECIFIED ROOM TEMPERATURE TENSILE PROPERTIES OF THE T-222 (Ta-10.4W-2.4Hf-0.01C) ALLOY

	Ultimate Strength, ksi		0.2% Yield Strength, ksi		Elongation, %	
	Min.	Max.	Min.	Max.	Min.	Max.
Tentative Specification	100	120	90	100	20	
Recommended Revisions by Vendor						
Westinghouse	105	125	100	120	-	
Wah Chang	105	Delete	95	Delete	-	
NRC	-	-	-	110	-	
Stellite(1)	-	-	-	-	-	
Universal Cyclops	95	115	85	105	-	
Fansteel (2)	-	125	-	110	-	
Final Revisions Incorporated in Specifications	105	125	100	120	-	

(1) No Guarantee, Information Only.

(2) Requested Deletion of Maximum Values for Bar, Rod and Tube.

TABLE XI. RECOMMENDED REVISIONS TO THE SPECIFIED HIGH TEMPERATURE STRESS-
RUPTURE PROPERTIES OF D-43 (Cb-10W-1Zr-0.1C) ALLOY

	Test Temp., °F	Strength, ksi	Minimum Life, Hours
Tentative Specification	2200	20	20
Recommended Revisions by Vendor			
DuPont (1)	--	15	--
Wah Chang (2)	--	13	--
Stellite (1)	--	--	--
Universal Cyclops (1)	--	--	--
Fansteel (1)			
Final Revisions Incorporated in Specifications	--	12	--

(1) No Guarantee, Information Only.

(2) Will Take Exception, Cannot Perform.

TABLE XII. RECOMMENDED REVISIONS TO THE SPECIFIED HIGH TEMPERATURE STRESS-
RUPTURE PROPERTIES OF FS-85 (Cb-28Ta-10.5W-0.9Zr) ALLOY

	<u>Test Temp., °F</u>	<u>Strength, ksi</u>	<u>Minimum Life, Hours</u>
Tentative Specification	2200	20	20
Recommended Revisions by Vendor			
DuPont (1)	--	--	--
Wah Chang (2)	--	14	--
Stellite (1)	--	--	--
Universal Cyclops (1)	--	--	--
Fansteel (1)	2000	--	--
Final Revisions Incorporated in Specifications	--	16	--

(1) No Guarantee, Information Only.

(2) Will Take Exception, Cannot Perform.

TABLE XIII. RECOMMENDED REVISIONS TO THE SPECIFIED HIGH TEMPERATURE STRESS-
RUPTURE PROPERTIES OF T-111 (Ta-8W-2Hf) ALLOY

	<u>Test Temp., °F</u>	<u>Strength, ksi</u>	<u>Minimum Life, Hours</u>
Tentative Specification	2400	20	20
Recommended Revisions by Vendor			
Westinghouse(1)	--	--	--
Wah Chang(2)	--	--	--
NRC(3)	--	--	--
Universal Cyclops(3)	--	--	--
Stellite(3)	--	--	--
Fansteel(3)	--	--	--
Final Revisions Incorporated in Specifications	--	19	--

(1) No Comments.

(2) Will Take Exception, Cannot Perform.

(3) No Guarantee, Information Only.

TABLE XIV. RECOMMENDED REVISIONS TO THE SPECIFIED HIGH TEMPERATURE STRESS-
RUPTURE PROPERTIES OF T-222 (Ta-10.4W-2.4Hf-0.01C) ALLOY

	Test Temp., °F	Strength, ksi	Minimum Life, Hours
Tentative Specifications	2400	30	20
Recommended Revisions by Vendor			
Westinghouse (1)	--	--	--
Wah Chang (2)	--	--	--
NRC (3)	--	--	--
Universal Cyclops (3)	--	24	--
Stellite (3)	--	--	--
Fansteel (3)	--	--	--
Final Revisions Incorporated in Specifications	--	--	--

(1) No Comments.

(2) Will Take Exception, Cannot Perform.

(3) No Guarantee, Information Only.

TABLE XVI. SUMMARY OF MATERIALS PRODUCERS RESPONSE TO INQUIRIES FOR
PROCUREMENT OF ADVANCED REFRACTORY ALLOYS FOR ALKALI METAL CONTAINMENT

Material Producer	T-111			T-222			FS-85			D-43		
	Price/ Delivery	Exceptions to Specs.		Price/ Delivery	Exceptions to Specs.		Price/ Delivery	Exceptions to Specs.		Price/ Delivery	Exceptions to Specs.	
E.I. duPont de Nemours & Company, Incorporated	A	---		A	---		B	D		B	D	
Fansteel Metallurgical Corporation	B	C		B	C		B	C		B	C	
GE-Lamp Metals & Components Department	A	---		A	---		A	---		A	---	
Kawecki Chemical Company	A	---		A	---		A	---		A	---	
Metals Division, National Research Corporation	B	C		A	---		A	---		A	---	
Nuclear Metals, Tube Division of Tectron, Inc.	A	---		A	---		A	---		A	---	
Superior Tube Company	A	---		A	---		A	---		A	---	
Stellite Division, Union Carbide Corporation	B	C		A ⁽¹⁾	---		B	C		A ⁽¹⁾	---	
Universal Cyclops Steel Corporation	A	---		A	---		A	---		A	---	
Wah Chang Corporation	B	D		B	D		B	D		A ⁽¹⁾	---	
Wolverine Tube Division of the Calumet Hecla Corp.	B	D		B	D		B	D		B	D	

A Indicates No Quotation Received.

B Indicates Quotation Received

C Indicates No Exception To Specification.

D Indicates Exceptions to Specification.

(1) No Quotation Received; However, Producer May Have Bid if Alloy was Selected.

TABLE XVII. SUMMARY OF MATERIALS PRODUCERS RESPONSE TO INQUIRIES FOR PROCUREMENT OF
ADVANCED REFRACTORY ALLOYS FOR USE IN SPACE ELECTRIC POWER TURBOMACHINERY

Material Producers	Mo-TZC		Cb-132M	
	Price/ Delivery	Exceptions to Specs.	Price/ Delivery	Exceptions to Specs.
Climax Molybdenum Company of Michigan	B	C	A	---
E.I. duPont de Nemours & Company, Incorporated	A	---	B(1)	C
Fansteel Metallurgical Corporation	A	---	B(2)	C
GE-Lamp Metals & Components Depart- ment	B	C	A	---
Kawecki Chemical Company	////(3)	---	A	---
Stellite Division, Union Carbide Corporation	A	---	A	---
Universal Cyclops Steel Corpora- tion	A	---	B	C
Wah Chang Corporation	A	---	B(4)	C

A Indicates No Quotation Received.

(2) CPFF

B Indicates Quotation Received.

(3) Not on Bidders List.

C Indicates Exceptions to Specifications.

(4) Product of Effort.

(1) 3-Inch Diameter Bar Only.

TABLE XVIII. REFRACTORY ALLOY REQUIREMENTS FOR CORROSION TEST LOOP

	Weight, lbs	Delivery Schedule Weeks
<u>A. Containment Test Alloy (T-111)</u>		
<u>1. Rod</u>		
0.125 inch dia.	15	14
0.250 inch dia.	1	14
0.500 inch dia.	6	14
0.625 inch dia.	3	14
1.000 inch dia.	21	14
1.125 inch dia.	5	14
1.500 inch dia.	7	14
2.000 inch dia.	82	14
2.500 inch dia.	94	14
3.125 inch dia.	75	14
	309	
<u>2. Bar</u>		
1.0 inch x 1.0 inch	8	14
	8	
<u>3. Wire</u>		
0.062 inch dia.	6	16
0.094 inch dia.	8	16
	14	
<u>4. Sheet/Foil/Plate</u>		
0.005 inch x 3.5 inch	0.6	16
0.006 inch x 3.5 inch	0.2	16
0.040 inch x 12.0 inch	21	14
0.500 inch x 6.125 inch	22	14
	43.8	
<u>5. Tube/Pipe</u>		
2.25 inch OD x 0.375 inch wall	40	16
2.50 inch OD x 0.500 inch wall	50	16
3.00 inch OD x 0.375 inch wall	50	16
3.25 inch OD x 0.250 inch wall	40	16
3.25 inch OD x 0.500 inch wall	73	16
1.00 inch OD x 0.100 inch wall	95	24

TABLE XVIII. (Cont'd)

	<u>Weight, lbs</u>	<u>Delivery Schedule Weeks</u>
0.375 inch OD x 0.065 inch wall	70	24
0.375 inch OD x 0.008 inch wall	3	24
3/8 inch Sch. 80 pipe	26	24
3/4 inch Sch. 80 pipe	9	24
	456	
B. <u>Turbine Alloy</u>		
1. <u>Rod (Mo-TZC)</u>		
2.0 inch dia.	55	20
1.0 inch dia.	11	20
	66	
2. <u>Rod (Cb-132M)</u>		
2.0 inch dia.	27	18
1.0 inch dia.	6	18
	33	

III. MATERIAL SPECIFICATIONS

Subsequent to the formal issuance of the specifications, the numbering system was revised. A listing of the specifications showing the original and revised specification numbers is given below. It should be noted that separate specifications were prepared to less stringent requirements for foil and wire.

<u>Title</u>	<u>Specification Number</u>	
	<u>Revised</u>	<u>Original</u>
A. FS-85 (Cb-28Ta-10.5W-0.9Zr) Columbium Alloy		
Bar and Rod	01-0021-00-B	SPPS-30-R1
Sheet, Plate and Strip	01-0020-00-B	SPPS-28-R1
Seamless Tubing and Pipe	01-0022-00-B	SPPS-32-R1
Foil	01-0042-00-A	SPPS-62
Wire	01-0046-00-A	SPPS-66
B. D-43 (Cb-10W-1Zr-0.1C) Columbium Alloy		
Bar and Rod	01-0013-00-B	SPPS-20-R1
Sheet, Plate and Strip	01-0038-00-B	SPPS-57-R1
Seamless Tubing and Pipe	01-0037-00-B	SPPS-56-R1
Foil	01-0041-00-A	SPPS-61
Wire	01-0045-00-A	SPPS-65
C. T-111 (Ta-8W-2Hf) Tantalum Alloy		
Bar and Rod	01-0015-00-B	SPPS-22-R1
Sheet, Plate and Strip	01-0040-00-B	SPPS-59-R1
Seamless Tubing and Pipe	01-0035-00-B	SPPS-54-R1
Foil	01-0043-00-A	SPPS-63
Wire	01-0048-00-A	SPPS-68
D. T-222 (Ta-10.4W-2.4Hf-0.01C) Tantalum Alloy		
Bar and Rod	01-0014-00-C	SPPS-21-R2
Sheet, Plate and Strip	01-0039-00-C	SPPS-58-R2
Seamless Tubing and Pipe	01-0036-00-C	SPPS-55-R2
Foil	01-0044-00-C	SPPS-64-R1
Wire	01-0047-00-B	SPPS-67R1
E. Cb-132M (Cb-20Ta-15W-5Mo-2Zr-0.13C) Columbium Alloy		
Bar and Rod	01-0010-00-A	SPPS-16-R1
Bar and Rod	01-0010-01-A	---
F. Mo-TZC (Mo-1.25Ti-0.15Zr-0.12C) Molybdenum Alloy		
Bar and Rod	01-0011-00-C	---
Bar and Rod	01-0011-01-C	---

SPECIFICATION

BAR AND ROD: FS-85 (Cb-28Ta-10.5W-0.9Zr) ALLOY

prepared for
NATIONAL AERONAUTICS AND SPACE ADMINISTRATION
Contract NAS 3-2547

SPACE POWER AND PROPULSION SECTION
MISSILE AND SPACE DIVISION
GENERAL ELECTRIC COMPANY
CINCINNATI, OHIO 45215

SPECIFICATION

BAR AND ROD: FS-85 (Cb-28Ta-10.5W-0.9Zr) ALLOY

1. SCOPE

1.1. Scope. This specification covers FS-85 (Cb-28Ta-10.5W-0.9Zr) alloy in bar and rod form intended for high temperature structural applications and alkali metal containment.

2. APPLICABLE DOCUMENTS

2.1. Government Documents. None

2.2. Non-Government Documents

ASTM Designation E8-57T (26 December 1957)	Method of Tension Testing of Metallic Materials
ASTM Designation E29-58T (1958)	Recommended Practices for Des- ignating Significant Places in Specified Limiting Values
ASTM Designation (Pending)	Methods for Chemical Analysis of Reactor and Commercial Columbium
ASTM E112-61 (1961)	Estimating Average Grain Size of Metals
AMS 2635 (15 August 1958)	Radiographic Inspection
AMS 2645 (1 March 1955)	Fluorescent Penetrant Inspec- tion
AMS 2646 (1 March 1955)	Contrast Dye Penetrant Inspec- tion

3. REQUIREMENTS

3.1. Acknowledgments. The vendor shall mention this specification in all quotations and all purchase order acknowledgments.

3.2. Manufacture. Material covered by this specification shall be made from ingots which have been double vacuum melted by the electron beam and/or consumable electrode arc melting processes. Breakdown operations shall be performed with conventional extrusion, forging and rolling equipment normally found in primary ferrous and nonferrous plants.

3.3. Processing. The starting stock size, processing temperatures, percentages of reduction, in-process annealing temperatures and times shall be selected by the vendor to achieve the grain size range specified in paragraph 3.6 and mechanical properties specified in paragraph 3.7. The amount of total reduction from the turned ingot to the finished product shall exceed 75%. The amount of final reduction for each mill product, imparted just prior to the final vacuum heat treatment and the total reduction since the previous recrystallization anneal shall be reported in the certificate of compliance.

3.4. Condition

3.4.1. General. The finished product shall be supplied in the recrystallized condition throughout the cross-sectional area to the grain size range specified in paragraph 3.6. All annealing shall be carried out in a vacuum less than 1×10^{-5} torr.

3.4.2. Heat Treatment. All mill products to be annealed shall be thoroughly degreased, chemically cleaned, and protected from furnace parts by a layer of fresh tantalum, columbium, or Cb-12r alloy foil 0.002-inch thick or greater. When annealing is carried out in a vacuum greater than 1×10^{-5} torr, with the prior approval of the purchaser, all mill products shall be enclosed in a chemically cleaned tantalum, columbium or columbium-1% zirconium alloy retort or wrapped in a minimum of two layers of fresh tantalum, columbium, or columbium-1% zirconium alloy foil 0.002-inch thick or greater. The conditions of final annealing shall be reported in the certificate of compliance.

3.4.3. Contamination. All items are to be free of contamination or internal oxidation. After final heat treatment, the material shall be examined metallographically for evidence of possible contamination caused by unsatisfactory heat treating atmospheres or processing con-

ditions. A microhardness traverse shall show a hardness increase not greater than 50 VHN from the center to the surface of a cross-sectional sample of the final product. At the discretion of the purchaser, samples taken to include at least one surface of the final product, and not exceeding 0.050-inch thick, may be chemically analyzed by the purchaser for oxygen, nitrogen, hydrogen and carbon. The analyses shall not exceed the limits set forth in paragraph 3.5.3. Any indication of contamination shall be cause for rejection of all material represented by that sample. The material shall be acceptable if the contaminated layer is completely eliminated before shipment by a machining operation within the specified dimensions and tolerances.

3.5. Chemical Composition

3.5.1. Ingot/Billet Composition. The chemical composition of ingots and billets for conversion to finished products shall conform to Table I (page 5). A minimum of four analyses shall be obtained as follows: ingot top-center, mid-radius and edge, and ingot bottom-center; all analyses must conform to ranges stated in Table I.

3.5.2. Final Product Composition. The manufacturer's ingot analyses shall be considered the chemical analyses of products supplied under this specification (Table I) except carbon, oxygen, nitrogen and hydrogen content which shall be determined on the finished product.

3.5.3. Check Analysis. Finished product analysis shall not exceed the following limits or variations:

<u>Element</u>	<u>Check Analysis Limits, Max., ppm</u>	<u>Permissible Variations in Check Analysis, ppm</u>
Carbon	100	+ 10
Oxygen	200	+ 20
Nitrogen	100	+ 10
Hydrogen	10	+ 2

3.6. Grain Size. The grain size of the final products shall conform to the following limits:

TABLE I
CHEMICAL COMPOSITION
FS-85 (Cb-28Ta-10.5W-0.9Zr) ALLOY

<u>Element</u>	<u>Minimum Content</u> <u>ppm</u>	<u>Maximum Content</u> <u>ppm</u>
Carbon	-	100
Nitrogen	-	75
Oxygen	-	100
Hydrogen	-	10
Molybdenum	-	200
Nickel	-	50
Cobalt	-	50
Iron	-	50
Tantalum	26 w/o	29 w/o
Tungsten	10 w/o	12 w/o
Zirconium	0.6 w/o	1.1 w/o
Columbium	Remainder	-

<u>Product Diameter or Thickness, Inches</u>	<u>Minimum Allowable ASTM Grain Size No.</u>	<u>Allowable Spread in ASTM Grain Size Nos. in Any One Item</u>	<u>% R_x Minimum</u>
0.125 to 0.250	4	2	100
0.250 to 0.500	4	2	100
0.500 to 1.0	4	2	100
1.0 to 2.0	4	2	95
Greater than 2.0	3	3	90

3.7. Mechanical Properties. The final product shall satisfy the following mechanical property requirements:

3.7.1. Room Temperature Tensile Properties. Representative samples of the material in final form shall be capable of the following property limits at room temperature (65°-85°F).

<u>Ultimate Tensile Strength, ksi</u>		<u>0.2% Yield Strength, ksi</u>		<u>Elong., % in 4D</u>
<u>Minimum</u>	<u>Maximum</u>	<u>Minimum</u>	<u>Maximum</u>	<u>Minimum</u>
70	100	50	80	20

3.7.2. Stress-to-Rupture Tests. The material shall be capable of achieving the following stress-rupture life under suitable environmental conditions (see paragraph 5.4.3).

<u>Test Temp., °F</u>	<u>Stress, ksi</u>	<u>Minimum Life Hours</u>
2200	16	20

Chemical analysis of stress-rupture specimens after test shall demonstrate that the degree of environmental contamination did not exceed the following limits: total increase in oxygen plus nitrogen content--less than 100 ppm; increase in hydrogen content--less than 5 ppm; increase in carbon content--less than 10 ppm. The following limits shall apply to check analyses of the analytical results:

Carbon	$\pm$	10 ppm
Oxygen	$\pm$	50 ppm
Nitrogen	$\pm$	50 ppm
Hydrogen	$\pm$	2 ppm

3.8. Tolerances

3.8.1. Rolled, Swaged, or Drawn Rounds

3.8.1.1. Definition. Rod - 3.5 inches in diameter or less.

3.8.1.2. Diameter. The permissible variation in diameter and the limits of out-of-roundness of descaled rounds shall not exceed those in Table II (refer to page 8).

3.8.1.3. Cut Lengths. Maximum length variation shall be 0.25-inch.

3.8.1.4. Straightness. Maximum deviation shall be 0.050 inch per foot in any length.

3.8.2. Square or Rectangular Bar

3.8.2.1. Definition. Bar - any straight product with a rectangular cross-section 0.187 inch or more thick and less than 5 inches wide.

3.8.2.2. Dimensions. Unless otherwise specified, forged or rolled square and rectangular shapes shall have the following tolerances:

<u>Thickness</u>	<u>Length</u>	<u>Width</u>
± 0.025 inch or $\pm 5\%$, whichever is less	± 0.125 inch	± 0.125 inch

3.8.2.3. Straightness of Bar. Maximum deviation shall be 0.050 inch per foot in any length.

3.9. Reports. The manufacturer shall supply at least three copies of a report showing non-proprietary manufacturing methods, processing conditions, and test procedures and results for each lot of material in the shipment. The report shall also include the number of the specification and the purchase order or contract number.

TABLE II
PERMISSIBLE DIMENSIONAL VARIATIONS FOR ROUND BAR

<u>Diameter</u> <u>Inches</u>	<u>Diameter</u> <u>Variation</u> <u>Inch</u>	<u>Out-of-Roundness</u> <u>Inch</u>
0.125 to 0.281	+ 0.002, -0.002	0.004
Over 0.281 to 0.406	+ 0.010, -0.005	0.008
Over 0.406 to 0.625	+ 0.010, -0.005	0.012
Over 0.625 to 0.875	+ 0.015, -0.005	0.015
Over 0.875 to 1.000	+ 0.020, -0.005	0.015
Over 1.000 to 1.375	+ 0.020, -0.010	0.018
Over 1.375 to 1.500	+ 0.020, -0.015	0.020
Over 1.500 to 1.625	+ 0.025, -0.015	0.020
Over 1.625 to 2.000	+ 0.030, -0.030	0.025
Over 2.000 to 2.500	+ 0.032, -0.032	0.025
Over 2.500 to 3.250	+ 0.032, -0.032	0.027
Over 3.250 to 3.500	+ 0.045, -0.045	0.040
Centerless Ground Rounds		
0.0625 to 2.0	+ 0.002, -0.002	
Over 2.0	+ 0.003, -0.002	

4. MAXIMUM ALLOWABLE DISCONTINUITIES

4.1. General. The finished product shall be visibly free from oxide or scale of any nature, grease, oil, residual lubricants, and other extraneous materials. Cracks, laps, seams, gouges, and fins shall be unacceptable.

4.2. Porosity and Inclusions. Indications of internal porosity and non-metallic inclusions greater than 0.020 inch or 3% of the thickness, whichever is smaller, shall be unacceptable. Those indications in the range 0.010 inch to 0.020 inch or 2% of the thickness or diameter, whichever is smaller, shall be a minimum of 0.500 inch apart; those indications less than 0.010 inch shall be a minimum of 0.12 inch apart.

4.3. Surface Rework. All surface pores, gouges, and other defects deeper than 0.005 inch or 3% of the thickness, whichever is smaller, shall be unacceptable. Surface imperfections may be faired smooth to remove any notch effect provided dimensional tolerances are still maintained.

5. QUALITY ASSURANCE PROVISIONS

5.1. Vendor Responsibility. The manufacturer shall make all tests and inspections of the material covered by this specification before shipment, unless otherwise specified. All test and inspection results shall be furnished to the purchaser.

5.2. Customer Review. The purchaser or his representative may witness the testing and inspection of the material. The manufacturer shall give the purchaser ample notice of the time and place of designated tests. If the purchaser's representative is not present at this time and a new date is not set, the requirement for purchaser's inspection at the place of testing is waived. When the purchaser's representative is present at the appointed time and place, the manufacturer shall afford him, without charge, all reasonable facilities to assure that the material is being furnished in accordance with this specification. This inspection shall not interfere unnecessarily with production operations.

5.3. Sample Selection. Care shall be exercised to insure that the samples selected for testing and chemical analyses are representative of the material and uncontaminated by the sampling procedure. Samples for the determination of mechanical properties shall be selected so as to consume a minimum amount of material, i.e., samples may be taken transverse to the final working direction from bar of sufficient width

or from bar greater than 2 inches in diameter. If there is any question about the sampling technique or the analysis, the methods for sampling and analysis shall be those agreed to by the buyer and seller. The location of test samples shall be reported in the certificate of compliance.

5.4. Test Methods

5.4.1. Chemical Analysis. Chemical analyses shall be conducted by mutually acceptable procedures, such as the vacuum fusion methods for gases, the combustion method for carbon, and the spectrochemical methods for metallic elements. Disputes shall be settled by accepted referee methods, such as the ASTM "Methods for Chemical Analysis of Reactor and Commercial Columbium."

5.4.2. Tensile Test. The tension test shall be conducted in accordance with ASTM Designation E8-57T, "Methods of Tension Testing of Metallic Materials." Yield strength shall be determined by the offset (0.2%) method. The tensile properties shall be determined using a strain rate of 0.005 inch per inch per minute up to 0.6% offset and then 0.05 inch, plus or minus 0.02 inch, per inch per minute to fracture.

5.4.3. Stress-Rupture Test. Stress-rupture properties of specimens shall be determined by mutually acceptable testing techniques. Suggested testing techniques for determining stress-rupture properties are:

Specimens shall be tested in a vacuum of 1×10^{-6} torr or better. The vacuum system shall incorporate an optically tight liquid nitrogen cold trap or a getter-ion pump.

Specimens shall be held for a half hour at the test temperature before application of load.

Test temperature shall be maintained at plus or minus 10°F during the test.

5.4.4. Grain Size. Grain size determinations shall be made according to ASTM Specification E112-61, "Estimating the Average Grain Size of Metals."

5.5. Number of Tests Required. Representative test specimens from the finished product representing each ingot and each lot of material shall be taken to determine conformity to this specification. The minimum frequency of these tests shall be:

Finished Product Chemistry - one per lot per ingot

Tensile Test - two per lot per ingot

Stress-Rupture Test - two per lot per ingot

Grain Size - two per lot per ingot

Microhardness Traverse - one per lot per ingot

5.6. Retest and Rework

5.6.1. Surface Contamination. Any sample or specimen exhibiting obvious surface contamination or improper preparation which disqualifies it as a truly representative sample shall be replaced with a new sample.

5.6.2. Rework. If inspection and test results of a lot do not conform to the requirements of this specification, the lot may be reworked at the option of the manufacturer. The lot shall be acceptable if all test results, after reworking, conform to this specification.

5.7. Inspection

5.7.1. Methods of Inspection

5.7.1.1. Radiographic. Whenever specified, the product shall be radiographed and found free of porosity and inclusions as specified in paragraph 4.2 using the technique described in AMS 2635, "Radiographic Inspection." The radiographs and product shall be identified so the exact position of each radiograph can be correlated with the specific area on the particular product.

5.7.1.2. Ultrasonic. All material 0.125-inch diameter and larger shall be inspected ultrasonically.

5.7.1.2.1. Method and Equipment. The finished products shall be ultrasonically inspected by the immersed technique at 5 mc or above. Transducers shall be no larger than 0.75-inch diameter. Surface finishes shall be no rougher than 125 rms. Inspection shall be by longitudinal wave and by shear wave in two perpendicular directions, i.e., longitudinal and transverse and shall be with focused transducers appropriate to the diameter being inspected (360 degree transducers are allowable

where appropriate). Automatic equipment which traverses a spiral path is satisfactory; but three traverses shall be made, one with the transducer in the circumferential shear position, one with the transducer in the axial shear position, and one with the transducer in the longitudinal wave position, unless otherwise specified.

5.7.1.2.2. Calibration of Bar and Rod. Calibration shall be on notches and holes in a segment of the material reserved solely for calibration purposes or in a calibration specimen of similar nature and shape. The depth of the notches shall be 3% of the bar thickness, 1.5% of the rod diameter, or 0.005 inch, whichever is smaller; the width, no greater than depth; the length, greater than beam width. The notches shall be placed perpendicular to the direction of the shear wave beam and perpendicular to the surface, e.g., axial and circumferential notches on bar. In addition to the notches, a 0.020-inch diameter hole shall be made at least 0.5-inch deep in the calibration piece parallel to the surface at a distance from the surface of $1/2$ the thickness or diameter or, if the thickness exceeds 0.750 inch, $1/4$, $1/2$ and $3/4$ the thickness. Calibration settings to achieve 80% amplitude of these notches or holes along with the magnitude of the other applicable calibration defects shall be recorded. For example, on bar with shear wave, the notch on the near surface should be set at 80% and the amplitudes recorded for the indications from the hole and the notch on the far surface. Gain settings should be recorded to achieve 80% as above and 80% on each of the other applicable calibration defects. For longitudinal wave, only the 0.020-inch diameter holes, with additional holes at $1/4$ and $1/2$ the thickness if the thickness exceeds 0.750-inch shall be used for calibration.

5.7.1.2.3. Evaluation. Evaluation during inspection shall be made against the appropriate calibration defect. For example, on bar with shear wave, the defects on or near the far surface shall be compared to the calibration from the far surface notch; defects near the center shall be compared to the calibration from the hole at the appropriate depth; defects on the near surface shall be compared to the calibration from the near surface notches.

5.7.1.2.4. Reports. The ultrasonic inspection report shall contain the equipment serial numbers, calibration amplitudes and gain settings, and the amplitude and location of each defect whose amplitude is 60% or greater.

5.7.1.2.5. Rejection. The above procedure shall be followed, and indications of defects which exceed the magnitude obtained from the appropriate calibrated notch in the sample shall be cause for rejection, unless otherwise agreed by the purchaser and vendor.

5.7.1.3. Penetrant Inspection. The exterior surface of the product shall be penetrant inspected and found free of flaws as specified in paragraph 4.3 using AMS 2645, "Fluorescent Penetrant Inspection," or AMS 2646, "Contrast Dye Penetrant Inspection." All parts thus inspected shall be marked with ink stamps as described in the specification; impression stampings or etching shall be unacceptable.

5.7.1.4. Reports. The manufacturer shall supply at least three copies of a report showing inspection results for each lot of material in the shipment. The report shall also include the number of the specification and the purchase order or contract number.

5.8. Rejection. Material not conforming to this specification or to any authorized modification shall be subject to rejection. Unless otherwise specified, rejected material may be returned to the manufacturer at the manufacturer's expense if the purchaser does not receive other instructions for disposition within three weeks after notice of rejection.

5.9. Referee. If the manufacturer and the purchaser disagree about the conformance of the material to the requirements of this specification or any special test specified by the purchaser, a mutually acceptable referee's test shall be used to determine conformance.

6. PREPARATION FOR DELIVERY

6.1. Identification. Each bundle, box, or carton shall be legibly and conspicuously marked or tagged with the number of this specification, purchase order or contract number, type, ingot number, lot number, nominal size, and the gross, net, and tare weights. When each bundle, box or carton consists of components from more than one ingot number or lot number, each component shall be identified individually.

6.2. Packing. Each individual item shall be wrapped in heavy gauge polyethylene film or similar material and packed in a manner assuring safe delivery when properly transported by any common carrier.

7. DEFINITIONS

7.1. Lot. A lot shall include all material of the same size, shape, condition and finish from one heat of material and which has received the same processing, has been annealed in the same vacuum annealing charge and has been processed simultaneously in all operations in which temperatures may reach 500°F or above. When process temperatures and environments are closely controlled or when closely adjacent sizes receive similar processing, lots may be combined for chemical, tensile and stress-rupture tests only, provided prior written approval has been obtained from the General Electric Company.

7.2. Check Analysis. An analysis may be requested by the purchaser of the metal after it has been processed into finished mill forms, to verify the composition within a heat or lot. Check analysis tolerances do not broaden the specified heat analysis requirements but rather, cover variations between laboratories in the measurement of the chemical content.

7.3. Significance of Numerical Limits. For determining compliance with the specified limits for requirements of the properties listed below, an observed value or a calculated value shall be rounded off using the rounding-off method in ASTM Designation E29-58T, "Recommended Practices for Designating Significant Places in Specified Limiting Values."

<u>Test</u>	<u>Rounded-Off Unit for Observed or Calculated Value</u>
Chemical composition and dimensional tolerances (when expressed decimally)	Nearest unit in the last right-hand place of figures of the specified limit
Tensile strength	Nearest 100 psi
Elongation	Nearest 1%
Rupture life	Nearest 0.1 hour

SPECIFICATION

SHEET, PLATE, AND STRIP: FS-85 (Cb-28Ta-10.5W-0.9Zr) ALLOY

prepared for
NATIONAL AERONAUTICS AND SPACE ADMINISTRATION
Contract NAS 3-2547

SPACE POWER AND PROPULSION SECTION
MISSILE AND SPACE DIVISION
GENERAL ELECTRIC COMPANY
CINCINNATI, OHIO 45215

SPECIFICATION

SHEET, PLATE, AND STRIP: FS-85 (Cb-28Ta-10.5W-0.9Zr) ALLOY

1. SCOPE

1.1. Scope. This specification covers FS-85 (Cb-28Ta-10.5W-0.9Zr) alloy in sheet, plate, and strip form intended for high temperature structural applications and alkali metal containment.

2. APPLICABLE DOCUMENTS

2.1. Government Documents. None

2.2. Non-Government Documents

ASTM Designation E8-57T
(26 December 1957)

Method of Tension Testing of
Metallic Materials

ASTM Designation E29-58T
(1958)

Recommended Practices for Des-
ignating Significant Places in
Specified Limiting Values

ASTM Designation (Pending)

Methods for Chemical Analysis
of Reactor and Commercial
Columbium

ASTM E112-61
(1961)

Estimating Average Grain Size
of Metals

AMS 2242A
(1 December 1950)

Tolerances, Corrosion and Heat
Resistant Sheet, Strip and Plate

AMS 2635
(15 August 1958)

Radiographic Inspection

AMS 2645
(1 March 1955)

Fluorescent Penetrant Inspec-
tion

AMS 2646
(1 March 1955)

Contrast Dye Penetrant Inspec-
tion

MAB-176-M
(6 September 1961)

Evaluation Test Methods for Re-
fractory Metal Sheet Materials

3. REQUIREMENTS

3.1. Acknowledgments. The vendor shall mention this specification in all quotations and all purchase order acknowledgments.

3.2. Manufacture. Material covered by this specification shall be made from ingots which have been double vacuum melted by the electron beam and/or consumable electrode arc melting processes. Breakdown operations shall be performed with conventional extrusion, forging and rolling equipment normally found in primary ferrous and nonferrous plants.

3.3. Processing. The starting stock size, processing temperatures, percentages of reduction, in-process annealing temperatures and times shall be selected by the vendor to achieve the grain size range specified in paragraph 3.6 and mechanical properties specified in paragraph 3.7. The amount of total reduction from the turned ingot to the final product shall exceed 75%. The amount of final reduction for each mill product, imparted just prior to the final vacuum heat treatment and the total reduction since the previous recrystallization anneal, shall be reported in the certificate of compliance.

3.4. Condition

3.4.1. General. The finished product shall be supplied in the recrystallized condition throughout the cross-sectional area to the grain size range specified in paragraph 3.6.

3.4.2. Heat Treatment. All annealing shall be carried out in a vacuum of less than 1×10^{-5} torr. All mill products to be annealed shall be thoroughly degreased, chemically cleaned and protected from furnace parts by a layer of fresh tantalum, columbium or Cb-1Zr alloy foil 0.002-inch thick or greater. When annealing is carried out in a vacuum greater than 1×10^{-5} torr, with the prior approval of the purchaser, all mill products shall be enclosed in a chemically cleaned tantalum, columbium or Cb-1Zr alloy retort or wrapped in a minimum of two layers of fresh tantalum, columbium or Cb-1Zr alloy foil 0.002-inch thick or greater. The conditions of final annealing shall be reported in the certificate of compliance.

3.4.3. Contamination. All items are to be free of contamination or internal oxidation. After final heat treatment, the material shall be examined metallographically for evidence of possible contamination caused by unsatisfactory heat treating atmospheres or processing conditions. A microhardness traverse shall show a hardness increase not greater than 50 VHN from the center to the surface of a cross sectional sample of the final product. At the discretion of the purchaser, samples taken to include at least one surface of the final product, and not exceeding 0.050-inch thick, may be chemically analyzed by the purchaser for oxygen, nitrogen, hydrogen and carbon. The analyses shall not exceed the limits set forth in paragraph 3.5.3. Any indication of contamination shall be cause for rejection of all material represented by that sample. The material shall be acceptable if the contaminated layer is completely eliminated before shipment by a machining operation within the specified dimensions and tolerances.

3.5. Chemical Composition

3.5.1. Ingot/Billet Composition. The chemical composition of ingots and billets for conversion to finished products shall conform to Table I (page 5). A minimum of four analyses shall be obtained as follows: ingot top-center, mid-radius and edge, and ingot bottom-center; all analyses must conform to ranges stated in Table I.

3.5.2. Final Product Composition. The manufacturer's ingot analyses shall be considered the chemical analyses for products supplied under this specification (Table I), except carbon, oxygen, nitrogen and hydrogen content which shall be determined on the finished product.

3.5.3. Check Analysis. Finished product analysis shall not exceed the following limits or variations:

<u>Element</u>	<u>Check Analysis Limits, Max., ppm</u>	<u>Permissible Variations in Check Analysis, ppm</u>
Carbon	100	+ 10
Oxygen	200	+ 20
Nitrogen	100	+ 10
Hydrogen	10	+ 2

TABLE I
CHEMICAL COMPOSITION

FS-85 (Cb-28Ta-10.5W-0.9Zr) ALLOY

<u>Element</u>	<u>Minimum Content</u> <u>ppm</u>	<u>Maximum Content</u> <u>ppm</u>
Carbon	-	100
Nitrogen	-	75
Oxygen	-	100
Hydrogen	-	10
Molybdenum	-	200
Nickel	-	50
Cobalt	-	50
Iron	-	50
Tantalum	26 w/o	29 w/o
Tungsten	10 w/o	12 w/o
Zirconium	0.6 w/o	1.1 w/o
Columbium	Remainder	-

3.6. Grain Size. The grain size of the final products shall conform to the following limits:

<u>Product Thickness, Inches</u>	<u>Minimum Allowable ASTM Grain Size No.</u>	<u>Allowable Spread in ASTM Grain Size Nos. in Any One Item</u>	<u>% R_x Minimum</u>
0.010 to 0.060	6	2	100
0.060 to 0.125	4	2	100
0.125 to 0.187	4	2	100
0.187 to 0.500	3	3	95
0.500 to 1.0	3	3	95
Greater than 1.0	3	3	90

3.7. Mechanical Properties. The final product shall satisfy the following mechanical property requirements:

3.7.1. Room Temperature Tensile Properties. Representative samples of the material in final form shall be capable of the following property limits at room temperature (65°-85°F):

<u>Ultimate Tensile Strength, ksi</u>		<u>0.2% Yield Strength, ksi</u>		<u>Elong., % in 2 Inches</u>
<u>Minimum</u>	<u>Maximum</u>	<u>Minimum</u>	<u>Maximum</u>	<u>Minimum</u>
70	100	50	80	20

3.7.2. Stress-to-Rupture Tests. The material shall be capable of achieving the following stress-rupture life under suitable environmental conditions (see paragraph 5.4.3).

<u>Test Temp., °F</u>	<u>Stress, ksi</u>	<u>Minimum Life Hours</u>
2200	16	20

Chemical analysis of stress-rupture specimens after test shall demonstrate that the degree of environmental contamination did not exceed the following limits: total increase in oxygen plus nitrogen content--

less than 100 ppm; increase in hydrogen content--less than 5 ppm; increase in carbon content--less than 10 ppm. The following limits shall apply to check analyses of the analytical results:

Carbon	±	10 ppm
Oxygen	±	50 ppm
Nitrogen	±	50 ppm
Hydrogen	±	2 ppm

3.7.3. Bend Ductility. Representative samples of the materials in final form shall withstand the following bend test at room temperature without failure when tested according to procedures described in the most recent revision of the Materials Advisory Board report MAB-176M, "Evaluation Test Methods for Refractory Metal Sheet Materials." The samples shall be sectioned with the long axis of the bend specimens perpendicular to the final rolling direction.

3.7.3.1. Sheet 0.060 inch in thickness and under shall be bent over a 1T radius through 105° at a ram speed of 1 inch per minute and subsequently flattened for a total bend of 180°.

3.7.3.2. Sheet over 0.060 inch to 0.187 inch in thickness shall be bent over a 1T radius through 105° at a ram speed of 1 inch per minute.

3.8. Tolerances

3.8.1. Plate

3.8.1.1. Definition. Plate includes material 6 inches wide or over and 0.187 inch or more in thickness.

3.8.1.2. Dimensions. Plate dimensions shall conform to the following tolerances:

<u>Thickness</u>	<u>Width</u>	<u>Length</u>
± 0.025 inch or ± 5% whichever is less	± 0.125 inch	± 0.125 inch

3.8.1.3. Flatness. Flatness tolerance on plate shall conform to AMS 2242A, "Tolerances, Corrosion and Heat Resistant Sheet, Strip and Plate."

3.8.2. Sheet

3.8.2.1. Definition. Sheet includes material 6 inches wide or over and up to 0.187 inch in thickness.

3.8.2.2. Dimensions. Sheet dimensions shall conform to those presented in Table II.

3.8.2.3. Flatness. See paragraph 3.8.3.3.

3.8.3. Strip

3.8.3.1. Definition. Strip includes material less than 6 inches wide and up to 0.187 inch in thickness.

3.8.3.2. Dimensions. Strip dimensions shall conform to those presented in Table II.

3.8.3.3. Flatness. Total deviation from flatness of sheet and strip shall not exceed 6% as determined by the formula:

$$\frac{H}{L} \times 100 = \% \text{ Flatness Deviation}$$

where

H = maximum distance from a flat reference surface

and

L = minimum distance from this point to the point of contact with the reference surface.

The actual values shall be reported. In determining flatness, the sheet shall not be subject to external pressure at any point but shall lie freely on a flat surface during measurement. Oilcanning will be reported. An estimate of the extent (area, height, etc.,) of these defects shall be made.

3.9. Reports. The manufacturer shall supply at least three copies of a report showing non-proprietary manufacturing methods, processing conditions, and test procedures and results for each lot of material in the shipment. The report shall also include the number of the specification and the purchase order or contract number.

TABLE II
DIMENSIONAL TOLERANCES FOR SHEET AND STRIP

<u>Material Thickness, Inch</u>	<u>Width, Inches</u>	<u>Thickness Tolerances, Inch</u>
0.010-0.019	to 24	± 0.001
0.020-0.039	to 24	± 0.0015
0.040-0.059	to 24	± 0.002
0.060-0.089	to 24	± 0.003
0.090-0.129	to 24	± 0.004
0.130-0.159	to 24	± 0.005
0.160-0.187	to 24	± 0.010

<u>Material Thickness, Inch</u>	<u>Width Tolerances, Inch</u>
0.010-0.059	+ 0.031, -0
0.060-0.125	+ 0.046, -0
0.126-0.187	+ 0.125, -0

<u>Material Thickness, Inch</u>	<u>Length Tolerances, Inch</u>
0.010-0.059	+ 0.046, -0
0.060-0.125	+ 0.062, -0
0.126-0.187	+ 0.125, -0

4. MAXIMUM ALLOWABLE DISCONTINUITIES

4.1. General. The finished product shall be visibly free from oxide or scale of any nature, grease, oil residual lubricants, and other extraneous materials. Cracks, laps, seams, gouges, and fins shall be unacceptable.

4.2. Porosity and Inclusions. Indications of internal porosity and non-metallic inclusions greater than 0.020 inch or 3% of the thickness, whichever is smaller, shall be unacceptable. Those indications in the range 0.010 inch to 0.020 inch or 2% of the thickness, whichever is smaller, shall be a minimum of 0.50 inch apart; those indications less than 0.010 inch shall be a minimum of 0.12 inch apart.

4.3. Surface Rework. All surface pores, gouges, and other defects deeper than 0.005 inch or 3% of the thickness, whichever is smaller, shall be unacceptable. Surface imperfections may be faired smooth to remove any notch effect provided dimensional tolerances are still maintained.

5. QUALITY ASSURANCE PROVISIONS

5.1. Vendor Responsibility. The manufacturer shall make all tests and inspections of the material covered by this specification before shipment, unless otherwise specified. All test and inspection results shall be furnished to the purchaser.

5.2. Customer Review. The purchaser or his representative may witness the testing and inspection of the material. The manufacturer shall give the purchaser ample notice of the time and place of designated tests. If the purchaser's representative is not present at this time and a new date is not set, the requirement for purchaser's inspection at the place of testing is waived. When the purchaser's representative is present at the appointed time and place, the manufacturer shall afford him, without charge, all reasonable facilities to assure that the material is being furnished in accordance with this specification. This inspection shall not interfere unnecessarily with production operations.

5.3. Sample Selection. Care shall be exercised to insure that the samples selected for testing and chemical analyses are representative of the material and uncontaminated by the sampling procedure. Samples for the determination of mechanical properties shall be selected so as to consume a minimum amount of material, i.e., specimens may be taken

transverse to the final working direction from plate and sheet and from strip if of sufficient width. If there is any question about the sampling technique or the analysis, the methods for sampling and analysis shall be those agreed to by the buyer and seller. The location of test samples shall be reported in the certificate of compliance.

5.4. Test Methods

5.4.1. Chemical Analysis. Chemical analyses shall be conducted by mutually acceptable procedures, such as the vacuum fusion methods for gases, the combustion method for carbon, and the spectrochemical methods for metallic elements. Disputes shall be settled by accepted referee methods, such as the ASTM "Methods for Chemical Analyses of Reactor and Commercial Columbium."

5.4.2. Tensile Test. The tension test shall be conducted in accordance with ASTM Designation E8-57T, "Methods of Tension Testing of Metallic Materials." Yield strength shall be determined by the offset (0.2%) method. The tensile properties shall be determined using a strain rate of 0.005 inch per inch per minute up to 0.6% offset and then 0.050 inch, plus or minus 0.02 inch, per inch per minute to fracture.

5.4.3. Stress-Rupture Test. Stress-rupture properties of specimens shall be determined by mutually acceptable testing techniques. Suggested testing techniques for determining stress-rupture properties are:

Specimens shall be tested in a vacuum of 1×10^{-6} torr or better. The vacuum system shall incorporate an optically tight liquid nitrogen cold trap or a getter-ion pump.

Specimens shall be held for a half hour at the test temperature before application of load.

Test temperature shall be maintained at plus or minus 10°F during the test.

5.4.4. Grain Size. Grain size determinations shall be made according to ASTM Specification E112-61, "Estimating the Average Grain Size of Metals."

5.5. Number of Tests Required. Representative test specimens from the finished product representing each ingot and each lot of material shall be taken to determine conformity to this specification. The minimum frequency of these tests shall be:

Finished Product Chemistry - one per lot per ingot

Tensile Test - two per lot per ingot

Stress-Rupture Test - two per lot per ingot

Bend Test - two per lot per ingot

Grain Size - two per lot per ingot

Microhardness Traverse - one per lot per ingot

5.6. Retest and Rework

5.6.1. Surface Contamination. Any sample or specimen exhibiting obvious surface contamination or improper preparation which disqualifies it as a truly representative sample shall be replaced with a new sample.

5.6.2. Rework. If inspection and test results of a lot do not conform to the requirements of this specification, the lot may be reworked at the option of the manufacturer. The lot shall be acceptable if all test results, after reworking, conform to this specification.

5.7. Inspection

5.7.1. Methods of Inspection

5.7.1.1. Radiographic. Whenever specified, the product shall be radiographed and found free of porosity and inclusions as specified in paragraph 4.2 using the technique described in AMS 2635, "Radiographic Inspection." The radiographs and product shall be identified so the exact position of each radiograph can be correlated with the specific area on the particular product.

5.7.1.2. Ultrasonic. Unless otherwise agreed to by the purchaser and the vendor, the material shall be inspected ultrasonically.

5.7.1.2.1. Method and Equipment. The finished products shall be ultrasonically inspected by the immersed technique at 5 mc or above. Transducers shall be no larger than 0.75-inch diameter. Surface finishes shall be no rougher than 125 rms. Inspection shall be by longitudinal wave and by shear wave in two perpendicular directions, i.e., longitudinal and transverse shear.

Transducers for the shear wave inspection shall be focused, preferably cylindrically, to a beam no more than 0.125-inch wide in its smaller dimension (where it enters the material being inspected). Cylindrically-focused transducers shall not exceed 2 inches in length. The focal distance shall be adjusted when the transducer is beamed perpendicular to the surface of the calibration piece; then this focal distance shall be maintained throughout the actual inspection. After the focal distance is established, an appropriate shear wave angle shall be set and the calibration notch indication shall be set at 80% on the indication where the sound beam traverses one or two thicknesses of the sheet (depending on whether the notch is on the far side or incident side of the sheet). Calibration gain settings shall be recorded when the calibration defect is on both the incident and the far side of the sheet. If there is any difference in the indication, that gain setting giving an 80% indication from the side which produces the smaller indication shall be used for inspection. Calibration shall be done before and after the ultrasonic inspection, or at the beginning and end of each work shift. If the magnitude of indication from the calibration notch differs 10% or more from the previous calibration, all material inspected since then shall be reinspected.

5.7.1.2.2. Calibration of Plate. Calibration shall be on notches and holes in a segment of the material reserved solely for calibration purposes. The depth of the notches shall be 0.005 inch, the width shall be 0.005 inch and the length greater than the ultrasonic beam width. The notches shall be placed on the surface of the calibration piece perpendicular to the direction of the intended shear wave inspection, i.e., transverse and longitudinal and at least 1 inch from the edge of the plate. In addition, a 0.020-inch diameter hole shall be made in the calibration piece parallel to the surface to a depth of at least 0.750 inch at a point one-half the thickness of the plate. If the thickness of the plate exceeds 0.750 inch, similar holes shall also be made at points one-quarter and three-quarters of the plate thickness. Calibration settings to achieve 80% amplitude of the notches and holes, along with the magnitude of the other applicable calibration defects, shall be recorded. For example, on plate using a shear wave, the notch on the near surface should be set at 80% and the amplitude recorded for the indications from the hole and notch on the far surface. Gain settings should be recorded to achieve 80% as above and 80% on each of the other applicable calibration defects. For longitudinal wave inspection, only the 0.020-inch diameter holes shall be used for calibration.

5.7.1.2.3. Calibration of Sheet and Strip. The sheet shall be inspected by a shear wave beam pointed in both longitudinal and transverse directions. Calibration shall be done on notches cut perpendicular to the direction of the beam in pieces of sheet of the same material and thickness as that to be inspected. If that portion is later trimmed and scrapped, the calibration notches may be made on a section of the actual sheet. The depth of the calibration notches shall be 3% of the sheet thickness; width, no greater than the depth; length, no more than 1 inch. All notches shall be at least 1 inch from the edge of the sheet. Duplicate notches may be made on the opposite face of the sheet in locations where the sound beam will not intersect both notches in a single traverse, or the sheet may be turned over during calibration to determine the relative response from the calibration notch on both the incident and far side of the sheet.

5.7.1.2.4. Evaluation. Evaluation during inspection shall be made against the appropriate calibration defect. For example, with shear wave, the defects on or near the far surface shall be compared to the calibration from the far surface notch; defects near the center shall be compared to the calibration from the hole at the appropriate depth; defects on the near surface shall be compared to the calibration from the near surface notch.

5.7.1.2.5. Reports. The ultrasonic inspection report shall contain the equipment serial numbers, calibration amplitudes and gain settings and the amplitude and location of each defect whose amplitude is 60% or greater.

5.7.1.2.6. Rejection. The above procedure shall be followed, and indications of defects which exceed the magnitude obtained from the appropriate calibrated notch in the sample shall be cause for rejection; unless otherwise agreed by the purchaser and vendor.

5.7.1.3. Penetrant Inspection. The exterior surface of the product shall be penetrant inspected and found free of flaws as specified in paragraph 4.3 using AMS 2645, "Fluorescent Penetrant Inspection," or AMS 2646, "Contrast Dye Penetrant Inspection." All parts thus inspected shall be marked with ink stamps as described in the specification; impression stampings or etching shall be unacceptable.

5.7.1.4. Reports. The manufacturer shall supply at least three copies of a report showing inspection results for each lot of material in the shipment. The report shall also include the number of the specification and the purchase order or contract number.

5.8. Rejection. Material not conforming to this specification or to any authorized modification shall be subject to rejection. Unless otherwise specified, rejected material may be returned to the manufacturer at the manufacturer's expense if the purchaser does not receive other instructions for disposition within three weeks after notice of rejection.

5.9. Referee. If the manufacturer and the purchaser disagree about the conformance of the material to the requirements of this specification or any special test specified by the purchaser, a mutually acceptable referee's test shall be used to determine conformance.

6. PREPARATION FOR DELIVERY

6.1. Identification. Each bundle, box, or carton shall be legibly and conspicuously marked or tagged with the number of this specification, purchase order or contract number, type, ingot number, lot number, nominal size, and the gross, net, and tare weights. When each bundle, box or carton consists of components from more than one ingot number or lot number, each component shall be identified individually.

6.2. Packing. Each individual item shall be wrapped in heavy gauge polyethylene film or other similar material and packed in a manner assuring safe delivery when properly transported by any common carrier.

7. DEFINITION

7.1. Lot. A lot shall include all material of the same size, shape, condition and finish from one heat of material and which has received the same processing, has been annealed in the same vacuum annealing charge and has been processed simultaneously in all operations in which temperatures may reach 500°F or above. When process temperatures and environments are closely controlled or when closely adjacent sizes receive similar processing, lots may be combined for chemical, tensile and stress-rupture tests only, provided prior written approval has been obtained from the General Electric Company.

7.2. Check Analysis. An analysis may be requested by the purchaser of the metal after it has been processed into finished mill forms, to verify the composition within a heat or lot. Check analysis tolerances do not broaden the specified heat analysis requirements but rather cover variations between laboratories in the measurement of the chemical content.

7.3. Significance of Numerical Limits. For determining compliance with the specified limits for requirements of the properties listed below, an observed value or a calculated value shall be rounded off using the rounding-off method in ASTM Designation E29-58T, "Recommended Practices for Designating Significant Places in Specified Limiting Values."

<u>Test</u>	<u>Rounded-Off Unit for Observed or Calculated Value</u>
Chemical composition and dimensional tolerances (when expressed decimally)	Nearest unit in the last right-hand place of figures of the specified limit
Tensile strength	Nearest 100 psi
Elongation	Nearest 1%
Rupture life	Nearest 0.1 hour

01-0022-00-B
SPPS-32-R1
23 December 1964
Page 1 of 15

SPECIFICATION

SEAMLESS TUBING AND PIPE: FS-85 (Cb-28Ta-10.5W-0.9Zr) ALLOY

prepared for
NATIONAL AERONAUTICS AND SPACE ADMINISTRATION
Contract NAS 3-2547

SPACE POWER AND PROPULSION SECTION
MISSILE AND SPACE DIVISION
GENERAL ELECTRIC COMPANY
CINCINNATI, OHIO 45215

SPECIFICATION

SEAMLESS TUBING AND PIPE: FS-85 (Cb-28Ta-10.5W-0.9 Zr) ALLOY

1. SCOPE

1.1. Scope. This specification covers FS-85 (Cb-28Ta-10.5W-0.9Zr) alloy in tube and pipe form intended for high temperature structural applications and alkali metal containment.

2. APPLICABLE DOCUMENTS

2.1. Government Documents. None

2.2. Non-Government Documents

ASTM Designation E8-57T (26 December 1957)	Method of Tension Testing of Metallic Materials
ASTM Designation E29-58T (1958)	Recommended Practices for Designating Significant Places in Specified Limiting Values
ASTM Designation (Pending)	Methods for Chemical Analysis of Reactor and Commercial Columbium
ASTM Designation E112-61 (1961)	Estimating Average Grain Size of Metals
AMS 2635 (15 August 1958)	Radiographic Inspection
AMS 2645 (1 March 1955)	Fluorescent Penetrant Inspection
AMS 2646 (1 March 1955)	Contrast Dye Penetrant Inspection

3. REQUIREMENTS

3.1. Acknowledgments. The vendor shall mention this specification in all quotations and all purchase order acknowledgments.

3.2. Manufacture. Material covered by this specification shall be made from ingots which have been double vacuum melted by the electron beam and/or consumable electrode arc melting processes. Breakdown operations shall be performed with conventional extrusion, forging, tube reducing and drawing equipment normally found in ferrous and non-ferrous plants.

3.3. Processing. The starting stock size, processing temperatures, percentages of reduction, in-process annealing temperatures and times shall be selected by the vendor to achieve the grain size range specified in paragraph 3.6 and mechanical properties specified in paragraph 3.7. The total amount of reduction from the turned ingot to the final product shall exceed 75%. The amount of final reduction for each mill product, imparted just prior to the final vacuum heat treatment and the total reduction since the last recrystallization anneal shall be reported in the certificate of compliance.

3.4. Condition

3.4.1. General. The finished product will be supplied in the recrystallized condition throughout the cross-sectional area to the grain size range specified in paragraph 3.6.

3.4.2. Heat Treatment. All annealing shall be carried out in a vacuum of less than 1×10^{-5} torr. All mill products to be annealed shall be thoroughly degreased, chemically cleaned and protected from furnace parts by a layer of fresh tantalum, columbium or Cb-1Zr alloy foil 0.002-inch thick or greater. When annealing is carried out in a vacuum greater than 1×10^{-5} torr, with the prior approval of the purchaser, all mill products shall be enclosed in a chemically cleaned tantalum, columbium or Cb-1Zr alloy retort or wrapped in a minimum of two layers of fresh tantalum, columbium or Cb-1Zr alloy foil 0.002-inch thick or greater. The conditions of final annealing shall be reported in the certificate of compliance.

3.4.3. Contamination. All items are to be free of contamination or internal oxidation. After final heat treatment, the material shall be examined metallographically for evidence of possible contamination caused by unsatisfactory heat treating atmospheres or processing conditions. A microhardness traverse shall show a hardness increase not

greater than 50 VHN from the center to the surface of a cross sectional sample of the final product. At the discretion of the purchaser, samples taken to include at least one surface of the final product, and not exceeding 0.050-inch thick, may be chemically analyzed by the purchaser for oxygen, nitrogen, hydrogen and carbon. The analyses shall not exceed the limits set forth in paragraph 3.5.3. Any indication of contamination shall be cause for rejection of all material represented by that sample. The material shall be acceptable if the contaminated layer is completely eliminated before shipment by a machining operation within the specified dimensions and tolerances.

3.5. Chemical Composition

3.5.1. Ingot/Billet Composition. The chemical composition of ingots and billets for conversion to finished products shall conform to Table I (page 5). A minimum of four analyses shall be obtained as follows: ingot top-center, mid-radius and edge, and ingot bottom-center; all analyses must conform to ranges stated in Table I.

3.5.2. Final Product Composition. The manufacturer's ingot analyses shall be considered the chemical analysis for products supplied under this specification (Table I) except carbon, oxygen, nitrogen and hydrogen content which shall be determined on the finished product.

3.5.3. Check Analysis. The finished product analysis shall not exceed the following limits or variations:

For Wall Thicknesses 0.020 Inch or Greater

<u>Element</u>	<u>Check Analysis Limits, Max., ppm</u>	<u>Permissible Variations in Check Analysis, ppm</u>
Carbon	100	+ 10
Oxygen	200	+ 20
Nitrogen	100	+ 10
Hydrogen	10	+ 2

TABLE I
CHEMICAL COMPOSITION
FS-85 (Cb-28Ta-10.5W-0.9Zr) ALLOY

<u>Element</u>	<u>Minimum Content</u> <u>ppm</u>	<u>Maximum Content</u> <u>ppm</u>
Carbon	-	100
Nitrogen	-	75
Oxygen	-	100
Hydrogen	-	10
Molybdenum	-	200
Nickel	-	50
Cobalt	-	50
Iron	-	50
Tantalum	26.0 w/o	29.0 w/o
Tungsten	10.0 w/o	12.0 w/o
Zirconium	0.60 w/o	1.10 w/o
Columbium	Remainder	-

For Wall Thicknesses Less Than 0.020 Inch

<u>Element</u>	<u>Check Analysis Limits, Max., ppm</u>	<u>Permissible Variations in Check Analysis, ppm</u>
Carbon	150	+ 10
Oxygen	300	+ 20
Nitrogen	100	+ 10
Hydrogen	10	+ 2

3.6. Grain Size. The grain size of the final products shall conform to the following limits:

<u>Product Wall Thickness, Inches</u>	<u>Minimum Allowable ASTM Grain Size No.</u>	<u>Allowable Spread in ASTM Grain Size Nos. in Any One Item</u>	<u>% R_x Minimum</u>
Less than 0.010	6	2	100
0.010 to 0.065	6	2	100
0.065 to 0.125	5	2	100
0.125 to 0.250	4	2	95
0.250 to 0.500	3	3	90

3.7. Mechanical Properties. The final product shall satisfy the following mechanical property requirements:

3.7.1. Room Temperature Tensile Properties. Representative samples of the material in final form shall be capable of the following property limits at room temperature (65°-85°F).

<u>Ultimate Tensile Strength, ksi</u>		<u>0.2% Yield Strength, ksi</u>		<u>Elong., %⁽¹⁾</u>
<u>Minimum</u>	<u>Maximum</u>	<u>Minimum</u>	<u>Maximum</u>	<u>Minimum</u>
70	100	50	80	20

(1) % Elongation in 4D for Threaded or Button-Head Test Specimens; in 2 Inches for Flat Test Specimens.

3.7.2. Stress-to-Rupture Tests. The material shall be capable of achieving the following stress-rupture life under suitable environmental conditions (see paragraph 5.4.3):

<u>Test Temp., °F</u>	<u>Stress, ksi</u>	<u>Minimum Life Hours</u>
2200	16	20

Chemical analysis of stress-rupture specimens after test shall demonstrate that the degree of environmental contamination did not exceed the following limits: total increase in oxygen plus nitrogen content--less than 100 ppm; increase in hydrogen content--less than 5 ppm; increase in carbon content--less than 10 ppm. The following limits shall apply to check analyses of the analytical results:

Carbon	±	10 ppm
Oxygen	±	50 ppm
Nitrogen	±	50 ppm
Hydrogen	±	2 ppm

3.7.3. Hydrostatic Test. Each tube, 1/8 inch or larger in outside diameter with a wall thickness of 0.015 inch or over, shall be tested to a hydrostatic pressure sufficient to produce a fiber stress of 12,000 psi. The test pressure, not to exceed 10,000 psi, shall be determined by the equation ($P = 2St/D$), where:

P = hydrostatic test pressure in pounds per square inch;

S = 12,000 psi

t = average wall thickness of the tube in inches;

D = outside diameter of the tube in inches.

3.7.4. Flare Test. A section of the heat treated tube shall be capable of being flared without cracking. The flare shall be made with a tool having a 60-degree included angle until the specified outside diameter has been increased by 15%.

3.8. Tolerances

3.8.1. Diameter and Wall Thickness. The permissible variations in diameter and wall thickness of tube shall not exceed those prescribed in Table II (refer to page 9).

3.8.2. Length. When tube is ordered cut-to-length, the usable length shall not be less than that specified, but a variation of plus 1/8 inch will be permitted in lengths up to 6 feet. In lengths over 6 feet, a variation of plus 1/4 inch will be permitted, unless otherwise specified.

3.8.3. Straightness. The tube shall be free of bends or kinks. For lengths up to 10 feet, the maximum bow shall not exceed one part in 1200; for lengths greater than 10 feet, the maximum bow shall not exceed one part in 600, unless otherwise agreed upon.

3.9. Reports. The manufacturer shall supply at least three copies of a report showing non-proprietary manufacturing methods, processing conditions, and test procedures and results for each lot of material in the shipment. The report shall also include the number of the specification and the purchase order or contract number.

4. MAXIMUM ALLOWABLE DISCONTINUITIES

4.1. General. Cracks, laps, seams, fins, and tears shall be unacceptable. The surface shall also be free from oxide or scale of any nature, grease, oil, residual lubricants, or other extraneous material.

4.2. Porosity and Inclusions. Indications with dimensions greater than 3% of the wall thickness shall be unacceptable. Indications with dimensions in the range of 1% to 3% of wall thickness must be a minimum of 0.50 inch apart. Indications with dimensions less than 1% of the wall thickness must be a minimum of 0.12 inch apart.

4.3. Surface Rework. Defects less than 3% of the nominal wall thickness detected by penetrant or ultrasonic inspection may be removed by grinding provided the wall thickness is not decreased below that permitted in Table II (refer to page 9).

5. QUALITY ASSURANCE PROVISIONS

5.1. Vendor Responsibility. The manufacturer shall make all tests and inspections of the material covered by this specification before shipment unless otherwise specified. All test and inspection results shall be furnished to the purchaser.

TABLE II
PERMISSIBLE VARIATIONS IN TUBE DIMENSIONS

<u>Nominal OD</u> <u>Inches</u>	<u>OD</u> <u>Inch</u>	<u>ID</u> <u>Inch</u>	<u>Wall</u> <u>Thickness</u> <u>%</u>
0.187 to but not incl. 0.625	± 0.004	± 0.004	± 10
0.625 to but not incl. 1.000	± 0.005	± 0.005	± 10
1.000 to but not incl. 2.000	± 0.0075	± 0.0075	± 10
2.000 to but not incl. 3.000	± 0.010	± 0.010	± 10
3.000 to but not incl. 4.000	± 0.0125	± 0.0125	± 10

NOTES: -----

- (1) Tolerances are applicable to only the two dimensions specified on the purchase order, e.g., outside diameter and wall; inside diameter and wall; outside diameter and inside diameter.
- (2) For tolerances applicable for very small tubes (less than 0.187-inch diameter) or very thin-wall tubes (less than 0.010-inch thick), the producer shall be consulted.
- (3) For tubes having an inside diameter less than 60% of the outside diameter or a wall 3/4 inch or over thick, which cannot be successfully drawn over a mandrel, the inside diameter may vary by an amount equal to plus or minus 10% of the wall thickness. The wall thickness of these tubes may vary plus or minus 12.5% from that specified.
- (4) Ovality measured at any cross section: For tubes with nominal wall thickness less than 3% of the nominal outside diameter, the ovality tolerances are double the tolerances in column 2 or 3. For ovality tolerances for tubes with wall thickness less than 2% nominal outside diameter, the producer shall be consulted.

5.2. Customer Review. The purchaser or his representative may witness the testing and inspection of the material. The manufacturer shall give the purchaser ample notice of the time and place of designated tests. If the purchaser's representative is not present at this time and a new date is not set, the requirement for purchaser's inspection at the place of testing is waived. When the purchaser's representative is present at the appointed time and place, the manufacturer shall afford him, without charge, all reasonable facilities to assure that the material is being furnished in accordance with this specification. This inspection shall not interfere unnecessarily with production operations.

5.3. Sample Selection. Care shall be exercised to insure that the sample selected for testing is representative of the material and uncontaminated by the sampling procedure. If there is any question about the sampling technique or the analysis, the methods for sampling and analysis shall be those agreed to by the buyer and seller. The specimen configuration selected for the performance of the testing required in paragraphs 5.4.2 and 5.4.3 shall be mutually agreed upon by the vendor and purchaser prior to the placement of a purchase order. The location of all test samples shall be reported in the certificate of compliance.

5.4. Test Methods

5.4.1. Chemical Analysis. Chemical analyses shall be conducted by mutually acceptable procedures, such as the vacuum fusion methods for gases, the combustion method for carbon, and the spectrochemical methods for metallic elements. Disputes shall be settled by accepted referee methods, such as the ASTM "Methods for Chemical Analysis of Reactor and Commercial Columbium."

5.4.2. Tensile Test. The tension test shall be performed in accordance with ASTM Designation E8-57T, "Methods of Tension Testing of Metallic Materials." Yield strength shall be determined by the offset (0.2%) method. The tensile properties shall be determined using a strain rate of 0.005 inch per inch per minute up to 0.6% offset and then 0.050 inch, plus or minus 0.020 inch, per inch per minute to fracture.

5.4.3. Stress-Rupture Tests. Stress-rupture properties of specimens shall be determined by mutually acceptable testing techniques. Suggested testing techniques for determining stress-rupture properties are:

Specimens shall be tested in a vacuum of 1×10^{-6} torr or better. The vacuum system shall incorporate an optically tight liquid nitrogen cold trap or a getter-ion pump.

Specimens shall be held for a half hour at the test temperature before application of load.

Test temperature shall be maintained at plus or minus 10°F during the test.

5.4.4. Grain Size. Grain size determinations shall be made according to ASTM Specification E112-61, "Estimating the Average Grain Size of Metals."

5.5. Number of Tests Required. Representative test specimens from the finished product representing each ingot and each lot of material shall be taken to determine conformity to this specification. The minimum frequency of these tests shall be:

Finished Product Chemistry - one per lot per ingot

Tensile Test - two per lot per ingot

Stress-Rupture Test - two per lot per ingot

Flare Test - two per lot per ingot

Grain Size - two per lot per ingot

Microhardness Traverse - one per lot per ingot

Hydrostatic Proof Test - 100%

5.6. Retest and Rework

5.6.1. Surface Contamination. Any sample or specimen exhibiting obvious surface contamination or improper preparation which disqualifies it as a truly representative sample shall be replaced with a new sample.

5.6.2. Rework. If inspection and test results of a lot do not conform to the requirements of this specification, the lot may be reworked at the option of the manufacturer. The lot shall be acceptable if all test results, after reworking, conform to this specification.

5.7. Inspection

5.7.1. Methods of Inspection

5.7.1.1. Radiographic. When specified, the product shall be radiographed and found free of porosity and inclusions as specified in paragraph 4.2 using the techniques described in AMS 2635, "Radiographic Inspection." The radiographs and product shall be identified so that the exact position of each radiograph can be correlated with the specific area on a particular product.

5.7.1.2. Ultrasonic Inspection. Unless otherwise agreed to by the purchaser and the vendor, the material shall be inspected ultrasonically.

5.7.1.2.1. Method and Equipment. Ultrasonic inspection shall be by the immersed technique at 5 mc or higher frequency using focused transducers. Inspection shall be by both circumferential and axial shear techniques with longitudinal wave being added when the wall thickness is greater than 0.150 inch. For longitudinal wave technique and for circumferential shear, transducers up to 2 inches long may be used with or without automatic equipment to rotate the tube past the transducer. If spiral pattern inspection traverse is not used, steps must be taken to assure that the ultrasonic beam remains in the same position relative to the tubing so the beam-to-tubing angle remains constant. For axial (longitudinal) shear, transducers must have no greater than 0.5 inch axial length. Transducers must be cylindrically focused for a diameter range which includes the tubing on which it is to be used.

5.7.1.2.2. Calibration. Calibration shall be on notches (a total of four, two axial and two circumferential), cut in the tube on both the outside and inside surface unless otherwise specified. The depth of the notches shall be 3% of the wall thickness to a minimum depth of 0.001 inch; the width, no greater than depth; the length, at least that of the ultrasonic beam with a maximum length of 1 inch. Material having a wall thickness greater than 0.150 inch shall also have a 0.020-inch diameter hole machined into the wall in the longitudinal direction at mid-point of the wall thickness. Focusing shall be done to maximize the indication from the inside diameter notch placed properly for the type of inspection contemplated. After focusing is completed, the inside diameter indication shall be set at 80% and gain setting recorded. Gain setting for 80% on the outside diameter notch shall also be recorded. Inspection shall be at the gain setting for the inside diameter indication. A distance corresponding to the wall thickness shall be marked on the oscilloscope. Focal distance to the part to be inspected shall

be set to that used for the calibration piece before beginning inspection. Calibration shall be done both before and after the inspection or at the beginning and end of each work shift. If calibration has changed (gain change greater than 5%), all inspections since the previous calibration shall be repeated.

5.7.1.2.3. Rejection. Rejection shall be by any indication which exceeds the amplitude of the respective calibration indication; i.e., inside diameter defects shall be compared to the indication from the notch on the inside diameter, and outside diameter defects shall be compared to the indication from the notch on the outside diameter. Defects less than half the thickness from the surface or less than 0.150 inch from the surface, whichever is smaller, shall be compared to the outside diameter calibration indication. Defects more than half the thickness from the incident surface or more than 0.150 inch from the surface shall be compared to the indications from the inside diameter calibration notch.

5.7.1.2.4. Reports. The ultrasonic inspection report shall contain the equipment serial numbers, calibration amplitudes and gain settings and the amplitude and location of each defect whose amplitude is 60% or greater.

5.7.1.3. Penetrant Inspection. The exterior surface of the product shall be penetrant inspected and found free of flaws as specified in paragraph 4.3 using AMS 2645, "Fluorescent Penetrant Inspection," or AMS 2646, "Contrast Dye Penetrant Inspection." All parts thus inspected shall be marked with ink stamps as described in the specification; impression stampings or etching shall be unacceptable.

5.7.1.4. Reports. The manufacturer shall supply at least three copies of a report showing inspection results for each lot of material in the shipment. The report shall also include the number of the specification and the purchase order or contract number.

5.8. Rejection. Material not conforming to this specification or to any authorized modification shall be subject to rejection. Unless otherwise specified, rejected material may be returned to the manufacturer at the manufacturer's expense if the purchaser does not receive other instructions for disposition within three weeks after notice of rejection.

5.9. Referee. If the manufacturer and the purchaser disagree concerning the conformance of the material to the requirements of this specification or any special test specified by the purchaser, a mutually acceptable referee's test shall be used to determine conformance.

6. PREPARATION FOR DELIVERY

6.1. Identification. Each bundle, box, or carton shall be legibly and conspicuously marked or tagged with the number of this specification, purchase order or contract number, type, ingot number, lot number, nominal size, and the gross, net and tare weights. When each bundle, box or carton consists of components from more than one ingot number or lot number, each component shall be identified individually.

6.2. Packing. The ends of each pipe or tube shall be sealed with suitable plastic caps and each individual item shall be wrapped in heavy gauge polyethylene or similar material and packed in a manner assuring safe delivery when properly transported by a common carrier.

7. DEFINITIONS

7.1. Lot. A lot shall include all material of the same size, shape, condition and finish from one heat of material and which has received the same processing, has been annealed in the same vacuum annealing charge and has been processed simultaneously in all operations in which temperatures may reach 500°F or above. When process temperatures and environments are closely controlled or when closely adjacent sizes receive similar processing, lots may be combined for chemical, tensile and stress-rupture tests only, provided prior written approval has been obtained from the General Electric Company.

7.2. Check Analysis. An analysis may be requested by the purchaser of the metal, after it has been processed into finished mill forms, for the purpose of verifying the composition within a heat or lot. Check analysis tolerances do not broaden the specified heat analysis requirements but rather cover variations between laboratories in the measurement of the chemical content.

7.3. Significance of Numerical Limits. For determining compliance with the specified limits for requirements of the properties listed below, an observed value or a calculated value shall be rounded off using the rounding-off method in ASTM Designation E29-58T, "Recommended Practices for Designating Significant Places in Specified Limiting Values."

<u>Test</u>	<u>Rounded-Off Unit for Observed or Calculated Value</u>
Chemical composition and dimensional tolerances (when expressed decimally)	Nearest unit in the last right-hand place of figures of the specified limit
Tensile strength	Nearest 100 psi
Elongation	Nearest 1%
Rupture life	Nearest 0.1 hour

SPECIFICATION

FOIL: FS-85 (Cb-28Ta-10.5W-0.9Zr) ALLOY

prepared for
NATIONAL AERONAUTICS AND SPACE ADMINISTRATION
Contract NAS 3-2547

SPACE POWER AND PROPULSION SECTION
MISSILE AND SPACE DIVISION
GENERAL ELECTRIC COMPANY
CINCINNATI, OHIO 45215

SPECIFICATION

FOIL: FS-85 (Cb-28Ta-10.5W-0.9Zr) ALLOY

1. SCOPE

1.1. Scope. This specification covers FS-85 (Cb-28Ta-10.5W-0.9Zr) alloy in foil form intended for high temperature non-structural applications.

2. APPLICABLE DOCUMENTS

2.1. Government Documents. None

2.2. Non-Government Documents

ASTM Designation (Pending)

Methods for Chemical Analysis
of Reactor and Commercial
Columbium

3. REQUIREMENTS

3.1. Acknowledgments. The vendor shall mention this specification in all quotations and all purchase order acknowledgments.

3.2. Manufacture. Material covered by this specification shall be made from ingots which have been double vacuum melted by the electron beam and/or consumable electrode arc melting processes. Breakdown operations shall be performed with conventional extrusion, forging and rolling equipment found in primary ferrous and nonferrous plants.

3.3. Processing. The starting stock size, processing temperatures, percentages of reduction, in-process annealing temperatures and times shall be selected by the vendor.

3.4. Condition. The finished product shall be supplied in the fully recrystallized condition through the cross-sectional area. All annealing shall be carried out in a vacuum of less than 1×10^{-5} torr. All mill

products to be annealed shall be thoroughly degreased, chemically cleaned and protected from furnace parts by a layer of fresh tantalum, columbium or Cb-1Zr alloy foil 0.002-inch thick or greater. When annealing is carried out in a vacuum greater than 1×10^{-5} torr, with the prior approval of the purchaser, all mill products shall be enclosed in a chemically cleaned tantalum, columbium or Cb-1Zr alloy retort or wrapped of a minimum of two layers of fresh tantalum, columbium or Cb-1Zr alloy foil 0.002-inch thick or greater. The conditions of final annealing shall be reported in the certificate of compliance.

3.5. Chemical Composition

3.5.1. Ingot/Billet Composition. The chemical composition of ingots and billets for conversion to finished products shall conform to Table I (page 4). A minimum of four analyses shall be obtained as follows: ingot top-center, mid-radius and edge, and ingot bottom-center; all analyses must conform to ranges stated in Table I.

3.5.2. Final Product Composition. The manufacturer's ingot analyses shall be considered the chemical analyses for products supplied under this specification (Table I), except carbon, oxygen, nitrogen and hydrogen content which shall be determined on the finished product.

3.5.3. Check Analysis. Finished product analysis shall not exceed the following limits or variations:

<u>Element</u>	<u>Check Analysis Limits, Max., ppm</u>	<u>Permissible Variations in Check Analysis, ppm</u>
Carbon	150	+ 10
Oxygen	300	+ 20
Nitrogen	100	+ 10
Hydrogen	10	+ 2

3.6. Bend Ductility. Representative samples of the materials in final form shall withstand a 180° bend without failure.

3.7. Tolerances

3.7.1. Definition. Foil includes material less than 12 inches wide and up to and including 0.010-inch thick.

TABLE I
CHEMICAL COMPOSITION
FS-85 (Cb-28Ta-10.5W-0.9Zr) ALLOY

<u>Element</u>	<u>Minimum Content</u> <u>ppm</u>	<u>Maximum Content</u> <u>ppm</u>
Carbon	-	100
Nitrogen	-	75
Oxygen	-	100
Hydrogen	-	10
Molybdenum	-	200
Nickel	-	50
Cobalt	-	50
Iron	-	50
Tantalum	26 w/o	29 w/o
Tungsten	10 w/o	12 w/o
Zirconium	0.6 w/o	1.1 w/o
Columbium	Remainder	-

3.7.2. Dimensions. Foil dimensions shall conform to the following limits:

<u>Material Thickness</u> <u>Inches</u>	<u>Thickness Tolerances</u> <u>Inch</u>	<u>Width Tolerance</u> <u>Inch</u>
Less than 0.003	+0.0008, -0.0000	+0.031, -0.000
0.003 to 0.005	+0.001	+0.031, -0.000
0.005 to 0.010	+0.0015	+0.031, -0.000

3.8. General. The finished product shall be visibly free from oxide or scale of any nature, grease, oil, residual lubricants, and other extraneous materials. Cracks, laps, seams, gouges, and fins shall be unacceptable.

4. QUALITY ASSURANCE PROVISIONS

4.1. Vendor Responsibility. The manufacturer shall make all tests and inspections of the material covered by this specification before shipment, unless otherwise specified. All test and inspection results shall be furnished to the purchaser.

4.2. Sample Selection. Care shall be exercised to insure that the samples selected for testing and chemical analyses are representative of the material and uncontaminated by the sampling procedure. If there is any question about the sampling technique or the analysis, the methods for sampling and analysis shall be those agreed to by the buyer and seller.

4.3. Chemical Analysis. Chemical analyses shall be conducted by mutually acceptable procedures, such as the vacuum fusion methods for gases, the combustion method for carbon, and the spectrochemical methods for metallic elements. Disputes shall be settled by accepted referee methods, such as the ASTM "Methods for Chemical Analyses of Reactor and Commercial Columbium."

4.4. Number of Tests Required. Representative test specimens from the finished product representing each ingot and each lot of material shall be taken to determine conformity to this specification. The minimum frequency of these tests shall be:

Finished Product Chemistry - one per lot per ingot

Bend Test - two per lot per ingot

4.5. Reports. The manufacturer shall supply at least three copies of a report showing non-proprietary manufacturing methods, processing conditions, and test procedures and results for each lot of material in the shipment. The report shall also include the number of the specification and the purchase order or contract number.

4.6. Rejection. Material not conforming to this specification or to any authorized modification shall be subject to rejection. Unless otherwise specified, rejected material may be returned to the manufacturer at the manufacturer's expense if the purchaser does not receive other instructions for disposition within three weeks after notice of rejection.

4.7. Referee. If the manufacturer and the purchaser disagree about the conformance of the material to the requirements of this specification or any special test specified by the purchaser, a mutually acceptable referee's test shall be used to determine conformance.

5. PREPARATION FOR DELIVERY

5.1. Identification. Each bundle, box, or carton shall be legibly and conspicuously marked or tagged with the number of this specification, purchase order or contract number, type, ingot number, lot number, nominal size, and the gross, net, and tare weights. When each bundle, box or carton consists of components from more than one ingot number or lot number, each component shall be identified individually.

5.2. Packing. Each individual item shall be wrapped in heavy gauge polyethylene film or other similar material and packed in a manner assuring safe delivery when properly transported by any common carrier.

6. DEFINITION

6.1. Lot. A lot shall include all material of the same size, shape, condition and finish from one heat of material and which has received the same processing, has been annealed in the same vacuum annealing charge and has been processed simultaneously in all operations in which temperatures may reach 500°F or above. When process temperatures and environments are closely controlled or when closely adjacent sizes receive similar processing, lots may be combined for chemical, tensile and stress-rupture tests only, provided prior written approval has been obtained from the General Electric Company.

6.2. Check Analysis. An analysis, made or requested by the purchaser of the metal after it has been processed into finished mill forms, to verify the composition within a heat or lot. Check analysis tolerances do not broaden the specified heat analysis requirements but rather cover variations between laboratories in the measurement of the chemical content.

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SPPS-66
23 December 1964
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SPECIFICATION

WIRE: FS-85 (Cb-28Ta-10.5W-0.9Zr) ALLOY

prepared for
NATIONAL AERONAUTICS AND SPACE ADMINISTRATION
Contract NAS 3-2547

SPACE POWER AND PROPULSION SECTION
MISSILE AND SPACE DIVISION
GENERAL ELECTRIC COMPANY
CINCINNATI, OHIO 45215

SPECIFICATION

WIRE: FS-85 (Cb-28Ta-10.5W-0.9Zr) ALLOY

1. SCOPE

1.1. Scope. This specification covers FS-85 (Cb-28Ta-10.5W-0.9Zr) alloy in wire form for use as weld filler material in fabricating components intended for high temperature structural applications and alkali metal containment.

2. APPLICABLE DOCUMENTS

2.1. Government Documents. None

2.2. Non-Government Documents

ASTM Designation (Pending)

Methods for Chemical Analysis
or Reactor and Commercial
Columbium

3. REQUIREMENTS

3.1. Acknowledgments. The vendor shall mention this specification in all quotations and all purchase order acknowledgments.

3.2. Manufacture. Material covered by this specification shall be made from ingots which have been double vacuum melted by the electron beam and/or consumable electrode arc melting processes. Breakdown and rolling equipment normally found in primary ferrous and nonferrous plants.

3.3. Processing. The starting stock size, processing temperatures, percentages of reduction, in-process annealing temperatures and times shall be selected by the vendor.

3.4. Condition. The finished product shall be supplied in the fully recrystallized condition throughout the cross-sectional area. All annealing shall be carried out in a vacuum less than 1×10^{-5} torr.

All mill products to be annealed shall be thoroughly degreased, chemically cleaned and protected from furnace parts by a layer of fresh tantalum, columbium, or Cb-1Zr alloy foil 0.002-inch thick or greater. When annealing is carried out in a vacuum greater than 1×10^{-5} torr, with the prior approval of the purchaser, all mill products shall be enclosed in a chemically cleaned tantalum, columbium or Cb-1Zr alloy retort or wrapped in a minimum of two layers of fresh tantalum, columbium or Cb-1Zr alloy foil 0.002-inch thick or greater. The conditions of final annealing shall be reported in the certificate of compliance.

3.5. Chemical Composition

3.5.1. Ingot/Billet Composition. The chemical composition of ingots and billets for conversion to finished products shall conform to Table I (page 4). A minimum of four analyses shall be obtained as follows: ingot top-center, mid-radius and edge, and ingot bottom-center; all analyses must conform to ranges stated in Table I.

3.5.2. Final Product Composition. The manufacturer's ingot analyses shall be considered the chemical analyses of products supplied under this specification (Table I) except carbon, oxygen, nitrogen and hydrogen content which shall be determined on the finished product.

3.5.3. Check Analysis. The finished product analysis shall not exceed the following limits or variations:

For Material Greater than 0.030 Inch in Diameter

<u>Element</u>	<u>Check Analysis Limits, Max., ppm</u>	<u>Permissible Variations in Check Analysis, ppm</u>
Carbon	100	+ 10
Oxygen	200	+ 20
Nitrogen	100	+ 10
Hydrogen	10	+ 2

TABLE I
CHEMICAL COMPOSITION
FS-85 (Cb-28Ta-10.5W-0.9Zr) ALLOY

<u>Element</u>	<u>Minimum Content</u> <u>ppm</u>	<u>Maximum Content</u> <u>ppm</u>
Carbon	-	100
Nitrogen	-	75
Oxygen	-	100
Hydrogen	-	10
Molybdenum	-	200
Nickel	-	50
Cobalt	-	50
Iron	-	50
Tantalum	26 w/o	29 w/o
Tungsten	10 w/o	12 w/o
Zirconium	0.6 w/o	1.1 w/o
Columbium	Remainder	-

For Material 0.030 Inch and Less in Diameter

<u>Element</u>	<u>Check Analysis Limits, Max., ppm</u>	<u>Permissible Variations in Check Analysis, ppm</u>
Carbon	150	+ 10
Oxygen	300	+ 20
Nitrogen	100	+ 10
Hydrogen	10	+ 2

3.6. Tolerances

3.6.1. Definition. Wire - material less than 0.125 inch in diameter.

3.6.2. Diameter. The permissible variation in diameter shall not exceed the following limits:

<u>Diameter, Inch</u>	<u>Diameter Variation, Inch</u>
0.005 to 0.009	± 0.0002
0.010 to 0.019	± 0.0003
0.020 to 0.029	± 0.0005
0.030 to 0.061	± 0.001
0.062 to 0.125	± 0.002

3.7. General. The finished product shall be visibly free from oxide or scale of any nature, grease, oil, residual lubricants, and other extraneous materials. Cracks, laps, seams, gouges, and fins shall be unacceptable.

4. QUALITY ASSURANCE PROVISIONS

4.1. Vendor Responsibility. The manufacturer shall make all tests and inspections of the material covered by this specification before shipment, unless otherwise specified. All test and inspection results shall be furnished to the purchaser.

4.2. Sample Selection. Care shall be exercised to insure that the samples selected for testing and chemical analyses are representative of the material and uncontaminated by the sampling procedure. If there is any question about the sampling technique or the analysis, the methods for sampling and analysis shall be those agreed to by the buyer and seller.

4.3. Chemical Analysis. Chemical analyses shall be conducted by mutually acceptable procedures, such as the vacuum fusion methods for gases, the combustion method for carbon, and the spectrochemical methods for metallic elements. Disputes shall be settled by accepted referee methods, such as the ASTM "Methods for Chemical Analysis of Reactor and Commercial Columbium."

4.4. Number of Tests Required. Representative test specimens from the finished product representing each ingot and each lot of material shall be taken to determine conformity to this specification. The minimum frequency of these tests shall be:

Finished Product Chemistry - one per lot per ingot

4.5. Reports. The manufacturer shall supply at least three copies of a report showing non-proprietary manufacturing methods, processing conditions, and test procedures and results for each lot of material in the shipment. The report shall also include the number of the specification and the purchase order or contract number.

4.6. Rejection. Material not conforming to this specification or to any authorized modification shall be subject to rejection. Unless otherwise specified, rejected material may be returned to the manufacturer at the manufacturer's expense if the purchaser does not receive other instructions for disposition within three weeks after notice of rejection.

4.7. Referee. If the manufacturer and the purchaser disagree about the conformance of the material to the requirements of this specification or any special test specified by the purchaser, a mutually acceptable referee's test shall be used to determine conformance.

5. PREPARATION FOR DELIVERY

5.1. Identification. Each bundle, box, or carton shall be legibly and conspicuously marked or tagged with the number of this specification, purchase order or contract number, type, ingot number, lot number,

nominal size, and the gross, net, and tare weights. When each bundle, box or carton consists of components from more than one ingot number or lot number, each component shall be identified individually.

5.2. Packing. Each individual item shall be wrapped in heavy gauge polyethylene film or similar material and packed in a manner assuring safe delivery when properly transported by any common carrier.

6. DEFINITIONS

6.1. Lot. A lot shall include all material of the same size, shape, condition and finish from one heat of material and which has received the same processing, has been annealed in the same vacuum annealing charge and has been processed simultaneously in all operations in which temperatures may reach 500°F or above. When process temperatures and environments are closely controlled or when closely adjacent sizes receive similar processing, lots may be combined for chemical, tensile and stress-rupture tests only, provided prior written approval has been obtained from the General Electric Company.

6.2. Check Analysis. An analysis, made or requested by the purchaser of the metal after it has been processed into finished mill forms, to verify the composition within a heat or lot. Check analysis tolerances do not broaden the specified heat analysis requirements but rather cover variations between laboratories in the measurement of the chemical content.

SPECIFICATION

BAR AND ROD: D-43 (Cb-10W-1Zr-0.1C) ALLOY

prepared for
NATIONAL AERONAUTICS AND SPACE ADMINISTRATION
Contract NAS 3-2547

SPACE POWER AND PROPULSION SECTION
MISSILE AND SPACE DIVISION
GENERAL ELECTRIC COMPANY
CINCINNATI, OHIO 45215

SPECIFICATION

BAR AND ROD: D-43 (Cb-10W-1Zr-0.1C) ALLOY

1. SCOPE

1.1. Scope. This specification covers D-43 (Cb-10W-1Zr-0.1C) alloy in bar and rod form intended for high temperature structural applications and alkali metal containment.

2. APPLICABLE DOCUMENTS

2.1. Government Documents. None

2.2. Non-Government Documents

ASTM Designation E8-57T
(26 December 1957)

Method of Tension Testing of
Metallic Materials

ASTM Designation E29-58T
(1958)

Recommended Practices for Des-
ignating Significant Places in
Specified Limiting Values

ASTM Designation (Pending)

Methods for Chemical Analysis
of Reactor and Commercial
Columbium

ASTM E112-61
(1961)

Estimating Average Grain Size
of Metals

AMS 2635
(15 August 1958)

Radiographic Inspection

AMS 2645
(1 March 1955)

Fluorescent Penetrant Inspec-
tion

AMS 2646
(1 March 1955)

Contrast Dye Penetrant Inspec-
tion

3. REQUIREMENTS

3.1. Acknowledgments. The vendor shall mention this specification in all quotations and all purchase order acknowledgments.

3.2. Manufacture. Material covered by this specification shall be made from ingots which have been double vacuum melted by the electron beam and/or consumable electrode arc melting processes. Breakdown operations shall be performed with conventional extrusion, forging and rolling equipment normally found in primary ferrous and nonferrous plants.

3.3. Processing. The starting stock size, processing temperatures, percentages of reduction, in-process annealing temperatures and times shall be selected by the vendor to achieve the grain size range specified in paragraph 3.6 and mechanical properties specified in paragraph 3.7. The amount of total reduction from the turned ingot to the finished product shall exceed 75%. The amount of final reduction for each mill product, imparted just prior to the final vacuum heat treatment and the total reduction since the previous recrystallization anneal shall be reported in the certificate of compliance.

3.4. Condition

3.4.1. General. The finished product shall be supplied in the recrystallized condition throughout the cross-sectional area to the grain size range specified in paragraph 3.6. All annealing shall be carried out in a vacuum less than 1×10^{-5} torr.

3.4.2. Heat Treatment. All mill products to be annealed shall be thoroughly degreased, chemically cleaned, and protected from furnace parts by a layer of fresh tantalum, columbium or Cb-1Zr alloy foil 0.002-inch thick or greater. When annealing is carried out in a vacuum greater than 1×10^{-5} torr, with the prior approval of the purchaser, all mill products shall be enclosed in a chemically cleaned tantalum, columbium or columbium-1% zirconium alloy retort or wrapped in a minimum of two layers of fresh tantalum, columbium or columbium-1% zirconium alloy foil 0.002-inch thick or greater. The conditions of final annealing shall be reported in the certificate of compliance.

3.4.3. Contamination. All items are to be free of contamination or internal oxidation. After final heat treatment, the material shall be examined metallographically for evidence of possible contamination caused by unsatisfactory heat treating atmospheres or processing conditions. A microhardness traverse shall show a hardness increase not

greater than 50 VHN from the center to the surface of a cross sectional sample of the final product. At the discretion of the purchaser, samples taken to include at least one surface of the final product, and not exceeding 0.050-inch thick, may be chemically analyzed by the purchaser for oxygen, nitrogen, hydrogen and carbon. The analyses shall not exceed the limits set forth in paragraph 3.5.3. Any indication of contamination shall be cause for rejection of all material represented by that sample. The material shall be acceptable if the contaminated layer is completely eliminated before shipment by a machining operation within the specified dimensions and tolerances.

3.5. Chemical Composition

3.5.1. Ingot/Billet Composition. The chemical composition of ingots and billets for conversion to finished products shall conform to Table I (page 5). A minimum of four analyses shall be obtained as follows: ingot top-center, mid-radius and edge, and ingot bottom-center; all analyses must conform to ranges stated in Table I.

3.5.2. Final Product Composition. The manufacturer's ingot analyses shall be considered the chemical analyses of products supplied under this specification (Table I) except carbon, oxygen, nitrogen and hydrogen content which shall be determined on the finished product.

3.5.3. Check Analysis. Finished product analysis shall not exceed the following limits or variations:

<u>Element</u>	<u>Check Analysis Limits, Max., ppm</u>	<u>Permissible Variations in Check Analysis, ppm</u>
Carbon	800 min; 1200 max	± 50
Oxygen	200	+ 20
Nitrogen	100	+ 10
Hydrogen	20	+ 5

3.6. Grain Size. The grain size of the final products shall conform to the following limits:

TABLE I
CHEMICAL COMPOSITION
D-43 (Cb-10W-1Zr-0.1C) ALLOY

<u>Element</u>	<u>Minimum Content</u> <u>ppm</u>	<u>Maximum Content</u> <u>ppm</u>
Carbon	800	1200
Nitrogen	-	75
Oxygen	-	100
Hydrogen	-	100
Tantalum	-	1000
Molybdenum	-	200
Nickel	-	200
Cobalt	-	50
Iron	-	200
Tungsten	9.0 w/o	11.0 w/o
Zirconium	0.75 w/o	1.25 w/o
Columbium	Remainder	-

<u>Product Diameter or Thickness, Inches</u>	<u>Minimum Allowable ASTM Grain Size No.</u>	<u>Allowable Spread in ASTM Grain Size Nos. in Any One Item</u>	<u>% R_x Minimum</u>
0.125 to 0.250	4	2	90
0.250 to 0.500	4	2	90
0.500 to 1.0	4	2	90
1.0 to 2.0	4	2	90
Greater than 2.0	3	3	90

3.7. Mechanical Properties. The final product shall satisfy the following mechanical property requirements:

3.7.1. Room Temperature Tensile Properties. Representative samples of the material in final form shall be capable of the following property limits at room temperature (65°-85°F).

<u>Ultimate Tensile Strength, ksi</u>	<u>0.2% Yield Strength, ksi</u>	<u>Elong., % in 4D</u>
<u>Minimum</u>	<u>Minimum</u>	<u>Minimum</u>
70	50	15

3.7.2. Stress-to-Rupture Tests. The material shall be capable of achieving the following stress-rupture life under suitable environmental conditions (see paragraph 5.4.3).

<u>Test Temp., °F</u>	<u>Stress, ksi</u>	<u>Minimum Life Hours</u>
2200	12	20

Chemical analysis of stress-rupture specimens after test shall demonstrate that the degree of environmental contamination did not exceed the following limits: total increase in oxygen plus nitrogen content--less than 100 ppm; increase in hydrogen content--less than 5 ppm; increase in carbon content--less than 10 ppm. The following limits shall apply to check analyses of analytical results:

Carbon	$\pm$	10 ppm
Oxygen	$\pm$	50 ppm
Nitrogen	$\pm$	50 ppm
Hydrogen	$\pm$	2 ppm

3.8. Tolerances

3.8.1. Rolled, Swaged, or Drawn Rounds

3.8.1.1. Definition. Rod - 3.5 inches in diameter or less.

3.8.1.2. Diameter. The permissible variation in diameter and the limits of out-of-roundness of descaled rounds shall not exceed those in Table II (refer to page 8).

3.8.1.3. Cut Lengths. Maximum length variation shall be 0.25 inch.

3.8.1.4. Straightness. Maximum deviation shall be 0.050 inch per foot in any length.

3.8.2. Square or Rectangular Bar

3.8.2.1. Definition. Bar - any straight product with a rectangular cross section 0.187 inch or more thick and less than 5 inches wide.

3.8.2.2. Dimensions. Unless otherwise specified, forged or rolled square and rectangular shapes shall have the following tolerances:

<u>Thickness</u>	<u>Length</u>	<u>Width</u>
± 0.025 inch or $\pm 5\%$ whichever is less	± 0.125 inch	± 0.125 inch

3.8.2.3. Straightness of Bar. Maximum deviation shall be 0.050 inch per foot in any length.

3.9. Reports. The manufacturer shall supply at least three copies of a report showing non-proprietary manufacturing methods, processing conditions, and test procedures and results for each lot of material in the shipment. The report shall also include the number of the specification and the purchaser order or contract number.

TABLE II
PERMISSIBLE DIMENSIONAL VARIATIONS FOR ROUND BAR

<u>Diameter</u> <u>Inches</u>	<u>Diameter</u> <u>Variation</u> <u>Inch</u>	<u>Out-of-Roundness</u> <u>Inch</u>
0.125 to 0.281	+ 0.002, -0.002	0.004
Over 0.281 to 0.406	+ 0.010, -0.005	0.008
Over 0.406 to 0.625	+ 0.010, -0.005	0.012
Over 0.625 to 0.875	+ 0.015, -0.005	0.015
Over 0.875 to 1.000	+ 0.020, -0.005	0.015
Over 1.000 to 1.375	+ 0.020, -0.010	0.018
Over 1.375 to 1.500	+ 0.020, -0.015	0.020
Over 1.500 to 1.625	+ 0.025, -0.015	0.020
Over 1.625 to 2.000	+ 0.030, -0.030	0.025
Over 2.000 to 2.500	+ 0.032, -0.032	0.025
Over 2.500 to 3.250	+ 0.032, -0.032	0.027
Over 3.250 to 3.500	+ 0.045, -0.045	0.040
 Centerless Ground Rounds		
0.0625 to 2.0	+ 0.002, -0.002	
Over 2.0	+ 0.003, -0.002	

4. MAXIMUM ALLOWABLE DISCONTINUITIES

4.1. General. The finished product shall be visibly free from oxide or scale of any nature, grease, oil, residual lubricants, and other extraneous materials. Cracks, laps, seams, gouges, and fins shall be unacceptable.

4.2. Porosity and Inclusions. Indications of internal porosity and non-metallic inclusions greater than 0.020 inch or 3% of the thickness, whichever is smaller, shall be unacceptable. Those indications in the range 0.010 inch to 0.020 inch or 2% of the thickness or diameter, whichever is smaller, shall be a minimum of 0.500 inch apart; those indications less than 0.010 inch shall be a minimum of 0.12 inch apart.

4.3. Surface Rework. All surface pores, gouges, and other defects deeper than 0.005 inch or 3% of the thickness, whichever is smaller, shall be unacceptable. Surface imperfections may be faired smooth to remove any notch effect provided dimensional tolerances are still maintained.

5. QUALITY ASSURANCE PROVISIONS

5.1. Vendor Responsibility. The manufacturer shall make all tests and inspections of the material covered by this specification before shipment, unless otherwise specified. All test and inspection results shall be furnished to the purchaser.

5.2. Customer Review. The purchaser or his representative may witness the testing and inspection of the material. The manufacturer shall give the purchaser ample notice of the time and place of designated tests. If the purchaser's representative is not present at this time and a new date is not set, the requirement for purchaser's inspection at the place of testing is waived. When the purchaser's representative is present at the appointed time and place, the manufacturer shall afford him, without charge, all reasonable facilities to assure that the material is being furnished in accordance with this specification. This inspection shall not interfere unnecessarily with production operations.

5.3. Sample Selection. Care shall be exercised to insure that the samples selected for testing and chemical analyses are representative of the material and uncontaminated by the sampling procedure. Samples for the determination of mechanical properties shall be selected so as to consume a minimum amount of material, i.e., specimens may be taken transverse to the final working direction from bar of sufficient width or from rod greater than 2 inches in diameter. If there is any question about the sampling technique or the analysis, the methods for sampling

and analysis shall be those agreed to by the buyer and seller. The location of test samples shall be reported in the certificate of compliance.

5.4. Test Methods

5.4.1. Chemical Analysis. Chemical analyses shall be conducted by mutually acceptable procedures, such as the vacuum fusion methods for gases, the combustion method for carbon, and the spectrochemical methods for metallic elements. Disputes shall be settled by accepted referee methods, such as the ASTM "Methods for Chemical Analysis of Reactor and Commercial Columbium."

5.4.2. Tensile Test. The tension test shall be conducted in accordance with ASTM Designation E8-57T, "Methods of Tension Testing of Metallic Materials." Yield strength shall be determined by the offset (0.2%) method. The tensile properties shall be determined using a strain rate of 0.005 inch per inch per minute up to 0.6% offset and then 0.050 inch, plus or minus 0.02 inch, per inch per minute to fracture.

5.4.3. Stress-Rupture Test. Stress-rupture properties of specimens shall be determined by mutually acceptable testing techniques. Suggested testing techniques for determining stress-rupture properties are:

Specimens shall be tested in a vacuum of 1×10^{-6} torr or better. The vacuum system shall incorporate an optically tight liquid nitrogen cold trap or a getter-ion pump.

Specimens shall be held for a half hour at the test temperature before application of load.

Test temperature shall be maintained at plus or minus 10°F during the test.

5.4.4. Grain Size. Grain size determinations shall be made according to ASTM Specification E112-61, "Estimating the Average Grain Size of Metals."

5.5. Number of Tests Required. Representative test specimens from the finished product representing each ingot and each lot of material shall be taken to determine conformity to this specification. The minimum frequency of these tests shall be:

Finished Product Chemistry - one per lot per ingot

Tensile Test - two per lot per ingot

Stress-Rupture Test - two per lot per ingot

Grain Size - two per lot per ingot

Microhardness Traverse - one per lot per ingot

5.6. Retest and Rework

5.6.1. Surface Contamination. Any sample or specimen exhibiting obvious surface contamination or improper preparation which disqualifies it as a truly representative sample shall be replaced with a new sample.

5.6.2. Rework. If inspection and test results of a lot do not conform to the requirements of this specification, the lot may be reworked at the option of the manufacturer. The lot shall be acceptable if all test results, after reworking, conform to this specification.

5.7. Inspection

5.7.1. Methods of Inspection

5.7.1.1. Radiographic. Whenever specified, the product shall be radiographed and found free of porosity and inclusions as specified in paragraph 4.2 using the technique described in AMS 2635, "Radiographic Inspection." The radiographs and product shall be identified so the exact position of each radiograph can be correlated with the specific area on the particular product.

5.7.1.2. Ultrasonic. All material 0.125-inch diameter and larger shall be inspected ultrasonically.

5.7.1.2.1. Method and Equipment. The finished products shall be ultrasonically inspected by the immersed technique at 5 mc or above. Transducers shall be no larger than 0.75-inch diameter. Surface finishes shall be no rougher than 125 rms. Inspection shall be by longitudinal wave and by shear wave in two perpendicular directions, i.e., longitudinal and transverse and shall be with focused transducers appropriate to the diameter being inspected (360 degree transducers are allowable where appropriate). Automatic equipment which traverses a spiral path is satisfactory; but three traverses shall be made, one with the transducer in the circumferential shear position, one with the transducer

in the axial shear position, and one with the transducer in the longitudinal wave position, unless otherwise specified.

5.7.1.2.2. Calibration of Bar and Rod. Calibration shall be on notches and holes in a segment of the material reserved solely for calibration purposes or in a calibration specimen of similar nature and shape. The depth of the notches shall be 3% of the bar thickness, 1.5% of the rod diameter, or 0.005 inch, whichever is smaller; the width, no greater than depth; the length, greater than beam width. The notches shall be placed perpendicular to the direction of the shear wave beam and perpendicular to the surface, e.g., axial and circumferential notches on bar. In addition to the notches, a 0.020-inch diameter hole shall be made at least 0.5-inch deep in the calibration piece parallel to the surface at a distance from the surface of $1/2$ the thickness or diameter or, if the thickness exceeds 0.750 inch, $1/4$, $1/2$ and $3/4$ the thickness. Calibration settings to achieve 80% amplitude of these notches or holes along with the magnitude of the other applicable calibration defects shall be recorded. For example, on bar with shear wave, the notch on the near surface should be set at 80% and the amplitudes recorded for the indications from the hole and the notch on the far surface. Gain settings should be recorded to achieve 80% as above and 80% on each of the other applicable calibration defects. For longitudinal wave, only the 0.020-inch diameter holes, with additional holes at $1/4$ and $1/2$ the thickness if the thickness exceeds 0.750 inch, shall be used for calibration.

5.7.1.2.3. Evaluation. Evaluation during inspection shall be made against the appropriate calibration defect. For example, on bar with shear wave, the defects on or near the far surface shall be compared to the calibration from the far surface notch; defects near the center shall be compared to the calibration from the hole at the appropriate depth; defects on the near surface shall be compared to the calibration from the near surface notch.

5.7.1.2.4. Reports. The ultrasonic inspection report shall contain the equipment serial numbers, calibration amplitudes and gain settings and the amplitude and location of each defect whose amplitude is 60% or greater.

5.7.1.2.5. Rejection. The above procedures shall be followed and indications of defects which exceed the magnitude obtained from the appropriate calibrated notch in the sample shall be cause for rejection, unless otherwise agreed by the purchaser and vendor.

5.7.1.3. Penetrant Inspection. The exterior surface of the product shall be penetrant inspected and found free of flaws as specified in paragraph 4.3 using AMS 2645, "Fluorescent Penetrant Inspection," or AMS 2646, "Contrast Dye Penetrant Inspection." All parts thus inspected shall be marked with ink stamps as described in the specification; impression stampings or etching shall be unacceptable.

5.7.1.4. Reports. The manufacturer shall supply at least three copies of a report showing inspection results for each lot of material in the shipment. The report shall also include the number of the specification and the purchase order or contract number.

5.8. Rejection. Material not conforming to this specification or to any authorized modification shall be subject to rejection. Unless otherwise specified, rejected material may be returned to the manufacturer at the manufacturer's expense if the purchaser does not receive other instructions for disposition within three weeks after notice of rejection.

5.9. Referee. If the manufacturer and the purchaser disagree about the conformance of the material to the requirements of this specification or any special test specified by the purchaser, a mutually acceptable referee's test shall be used to determine conformance.

6. PREPARATION FOR DELIVERY

6.1. Identification. Each bundle, box, or carton shall be legibly and conspicuously marked or tagged with the number of this specification, purchase order or contract number, type, ingot number, lot number, nominal size, and the gross, net, and tare weights. When each bundle, box or carton consists of components from more than one ingot number or lot number, each component shall be identified individually.

6.2. Packing. Each individual item shall be wrapped in heavy gauge polyethylene film or similar material and packed in a manner assuring safe delivery when properly transported by any common carrier.

7. DEFINITIONS

7.1. Lot. A lot shall include all material of the same size, shape, condition and finish from one heat of material and which has received the same processing, has been annealed in the same vacuum annealing charge and has been processed simultaneously in all operations in which temperatures may reach 500°F or above. When process temperatures and

environments are closely controlled or when closely adjacent sizes receive similar processing, lots may be combined for chemical, tensile and stress-rupture tests only, provided prior written approval has been obtained from the General Electric Company.

7.2. Check Analysis. An analysis may be requested by the purchaser of the metal after it has been processed into finished mill forms, to verify the composition within a heat or lot. Check analysis tolerances do not broaden the specified heat analysis requirements but rather cover variations between laboratories in the measurement of the chemical content.

7.3. Significance of Numerical Limits. For determining compliance with the specified limits for requirements of the properties listed below, an observed value or a calculated value shall be rounded off using the rounding-off method in ASTM Designation E29-58T, "Recommended Practices for Designating Significant Places in Specified Limiting Values."

<u>Test</u>	<u>Rounded-Off Unit for Observed or Calculated Value</u>
Chemical composition and dimensional tolerances (when expressed decimally)	Nearest unit in the last right-hand place of figures of the specified limit
Tensile strength	Nearest 100 psi
Elongation	Nearest 1%
Rupture life	Nearest 0.1 hour

01-0038-00-B
SPPS-57-R1
23 December 1964
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SPECIFICATION

SHEET, PLATE, AND STRIP: D-43 (Cb-10W-1Zr-0.1C) ALLOY

prepared for
NATIONAL AERONAUTICS AND SPACE ADMINISTRATION
Contract NAS 3-2547

SPACE POWER AND PROPULSION SECTION
MISSILE AND SPACE DIVISION
GENERAL ELECTRIC COMPANY
CINCINNATI, OHIO 45215

SPECIFICATION

SHEET, PLATE, AND STRIP: D-43 (Cb-10W-1Zr-0.1C) ALLOY

1. SCOPE

1.1. Scope. This specification covers D-43 (Cb-10W-1Zr-0.1C) alloy in sheet, plate, and strip form intended for high temperature structural applications and alkali metal containment.

2. APPLICABLE DOCUMENTS

2.1. Government Documents. None

2.2. Non-Government Documents

ASTM Designation E8-57T
(26 December 1957)

Method of Tension Testing
of Metallic Materials

ASTM Designation E29-58T
(1958)

Recommended Practices for
Designating Significant
Places in Specified Limiting
Values

ASTM Designation (Pending)

Methods for Chemical Analysis
of Reactor and Commercial
Columbium

ASTM E112-61
(1961)

Estimating Average Grain Size
of Metals

AMS 2242A
(1 December 1950)

Tolerances, Corrosion and
Heat Resistant Sheet, Strip
and Plate

AMS 2635
(15 August 1958)

Radiographic Inspection

AMS 2645
(1 March 1955)

Fluorescent Penetrant Inspec-
tion

AMS 2646
(1 March 1955)

Contrast Dye Penetrant Inspec-
tion

MAB-176-M
(6 September 1961)

Evaluation Test Methods for Re-
fractory Metal Sheet Materials

3. REQUIREMENTS

3.1. Acknowledgments. The vendor shall mention this specification in all quotations and all purchase order acknowledgments.

3.2. Manufacture. Material covered by this specification shall be made from ingots which have been double vacuum melted by the electron beam and/or consumable electrode arc melting processes. Breakdown operations shall be performed with conventional extrusion, forging and rolling equipment normally found in primary ferrous and nonferrous plants.

3.3. Processing. The starting stock size, processing temperatures, percentages of reduction, in-process annealing temperatures and times shall be selected by the vendor to achieve the grain size range specified in paragraph 3.6 and mechanical properties specified in paragraph 3.7. The amount of total reduction from the turned ingot to the final product shall exceed 75%. The amount of final reduction for each mill product, imparted just prior to the final vacuum heat treatment and the total reduction since the previous recrystallization anneal, shall be reported in the certificate of compliance.

3.4. Condition

3.4.1. General. The finished product shall be supplied in the recrystallized condition throughout the cross-sectional area to the grain size range specified in paragraph 3.6.

3.4.2. Heat Treatment. All annealing shall be carried out in a vacuum of less than 1×10^{-5} torr. All mill products to be annealed shall be thoroughly degreased, chemically cleaned and protected from furnace parts by a layer of fresh tantalum, columbium or Cb-1Zr alloy foil 0.002-inch thick or greater. When annealing is carried out in a vacuum greater than 1×10^{-5} torr, with the prior approval of the purchaser, all mill products shall be enclosed in a chemically cleaned tantalum, columbium or Cb-1Zr alloy retort or wrapped in a minimum of two layers of fresh tantalum, columbium or Cb-1Zr alloy foil 0.002-inch thick or greater. The conditions of final annealing shall be reported in the certificate of compliance.

3.4. Contamination. All items are to be free of contamination or internal oxidation. After final heat treatment, the material shall be examined metallographically for evidence of possible contamination caused by unsatisfactory heat treating atmospheres or processing conditions. A microhardness traverse shall show a hardness increase not greater than 50 VHN from the center to the surface of a cross sectional sample of the final product. At the discretion of the purchaser, samples taken to include at least one surface of the final product, and not exceeding 0.050-inch thick, may be chemically analyzed by the purchaser for oxygen, nitrogen, hydrogen and carbon. The analyses shall not exceed the limits set forth in paragraph 3.5.3. Any indication of contamination shall be cause for rejection of all material represented by that sample. The material shall be acceptable if the contaminated layer is completely eliminated before shipment by a machining operation within the specified dimensions and tolerances.

3.5. Chemical Composition

3.5.1. Ingot/Billet Composition. The chemical composition of ingots and billets for conversion to finished products shall conform to Table I (page 5). A minimum of four analyses shall be obtained as follows: ingot top-center, mid-radius and edge, and ingot bottom-center; all analyses must conform to ranges stated in Table I.

3.5.2. Final Product Composition. The manufacturer's ingot analyses shall be considered the chemical analyses for products supplied under this specification (Table I), except carbon, oxygen, nitrogen and hydrogen content which shall be determined on the finished product.

3.5.3. Check Analysis. Finished product analysis shall not exceed the following limits or variations:

<u>Element</u>	<u>Check Analysis Limits, Max., ppm</u>	<u>Permissible Variations in Check Analysis, ppm</u>
Carbon	800 min; 1200 max	± 50
Oxygen	200	+ 20
Nitrogen	100	+ 10
Hydrogen	20	+ 5

TABLE I
CHEMICAL COMPOSITION
D-43 (Cb-10W-1Zr-0.1C) ALLOY

<u>Element</u>	<u>Minimum Content</u> <u>ppm</u>	<u>Maximum Content</u> <u>ppm</u>
Carbon	800	1200
Nitrogen	-	75
Oxygen	-	100
Hydrogen	-	100
Tantalum	-	1000
Molybdenum	-	200
Nickel	-	200
Cobalt	-	50
Iron	-	200
Tungsten	9.0 w/o	11.0 w/o
Zirconium	0.75 w/o	1.25 w/o
Columbium	Remainder	-

3.6. Grain Size. The grain size of the final products shall conform to the following limits:

<u>Product Thickness, Inches</u>	<u>Minimum Allowable ASTM Grain Size No.</u>	<u>Allowable Spread in ASTM Grain Size Nos. in Any One Item</u>	<u>% Rx Minimum</u>
0.010 to 0.060	6	2	90
0.060 to 0.125	4	2	90
0.125 to 0.187	4	2	90
0.187 to 0.500	3	3	90
0.500 to 1.0	3	3	90
Greater than 1.0	3	3	90

3.7. Mechanical Properties. The final product shall satisfy the following mechanical property requirements:

3.7.1. Room Temperature Tensile Properties. Representative samples of the material in final form shall be capable of the following property limits at room temperature (65°-85°F):

<u>Ultimate Tensile Strength, ksi</u>		<u>0.2% Yield Strength, ksi</u>		<u>Elong., % in 2 Inches</u>
<u>Minimum</u>	<u>Maximum</u>	<u>Minimum</u>	<u>Maximum</u>	<u>Minimum</u>
70	90	50	70	15

3.7.2. Stress-to-Rupture Tests. The material shall be capable of achieving the following stress-rupture life under suitable environmental conditions (see paragraph 5.4.3)

<u>Test Temp., °F</u>	<u>Stress, ksi</u>	<u>Minimum Life Hours</u>
2200	12	20

Chemical analysis of stress-rupture specimens after test shall demonstrate that the degree of environmental contamination did not exceed the following limits: total increase in oxygen plus nitrogen content--

less than 100 ppm; increase in hydrogen content--less than 5 ppm; increase in carbon content--less than 10 ppm. The following limits shall apply to check analyses of the analytical results:

Carbon	±	10 ppm
Oxygen	±	50 ppm
Nitrogen	±	50 ppm
Hydrogen	±	2 ppm

3.7.3. Bend Ductility. Representative samples of the materials in final form shall withstand the following bend test at room temperature without failure when tested according to procedures described in the most recent revision of the Materials Advisory Board report MAB-176-M, "Evaluation Test Methods for Refractory Metal Sheet Materials." The samples shall be sectioned with the long axis of the bend specimens perpendicular to the final rolling direction.

3.7.3.1. Sheet 0.060 inch in thickness and under shall be bent over a 1T radius through 105° at a ram speed of 1 inch per minute and subsequently flattened for a total bend of 180°.

3.7.3.2. Sheet over 0.060 inch to 0.187 inch in thickness shall be bent over a 1T radius through 105° at a ram speed of 1 inch per minute.

3.8. Tolerances

3.8.1. Plate

3.8.1.1. Definition. Plate includes material 6 inches wide or over and 0.187 inch or more in thickness.

3.8.1.2. Dimensions. Plate dimensions shall conform to the following tolerances:

<u>Thickness</u>	<u>Width</u>	<u>Length</u>
± 0.025 inch or ± 5% whichever is less	± 0.125 inch	± 0.125 inch

3.8.1.3. Flatness. Flatness tolerance on plate shall conform to AMS 2242A, "Tolerances, Corrosion and Heat Resistant Sheet, Strip and Plate."

3.8.2. Sheet

3.8.2.1. Definition. Sheet includes material 6 inches wide or over and up to 0.187 inch in thickness.

3.8.2.2. Dimensions. Sheet dimensions shall conform to those presented in Table II.

3.8.2.3. Flatness. See paragraph 3.8.3.3.

3.8.3. Strip

3.8.3.1. Definition. Strip includes material less than 6 inches wide and up to 0.187 inch in thickness.

3.8.3.2. Dimensions. Strip dimensions shall conform to those presented in Table II.

3.8.3.3. Flatness. Total deviation from flatness of sheet and strip shall not exceed 6% as determined by the formula:

$$\frac{H}{L} \times 100 = \% \text{ Flatness Deviation}$$

where

H = maximum distance from a flat reference surface
and

L = minimum distance from this point to the point of contact with the reference surface.

The actual values shall be reported. In determining flatness, the sheet shall not be subject to external pressure at any point but shall lie freely on a flat surface during measurement. Oilcanning will be reported. An estimate of the extent (area, height, etc.,) of these defects shall be made.

3.9. Reports. The manufacturer shall supply at least three copies of a report showing non-proprietary manufacturing methods, processing conditions, and test procedures and results for each lot of material in the shipment. The report shall also include the number of the specification and the purchase order or contract number.

TABLE II
DIMENSIONAL TOLERANCES FOR SHEET AND STRIP

<u>Material Thickness, Inch</u>	<u>Width, Inches</u>	<u>Thickness Tolerances, Inch</u>
0.010-0.019	to 24	± 0.001
0.020-0.039	to 24	± 0.0015
0.040-0.059	to 24	± 0.002
0.060-0.089	to 24	± 0.003
0.090-0.129	to 24	± 0.004
0.130-0.159	to 24	± 0.005
0.160-0.187	to 24	± 0.010

<u>Material Thickness, Inch</u>	<u>Width Tolerances, Inch</u>
0.010-0.059	+ 0.031, -0
0.060-0.125	+ 0.046, -0
0.126-0.187	+ 0.125, -0

<u>Material Thickness, Inch</u>	<u>Length Tolerances, Inch</u>
0.010-0.059	+ 0.046, -0
0.060-0.125	+ 0.062, -0
0.126-0.187	+ 0.125, -0

4. MAXIMUM ALLOWABLE DISCONTINUITIES

4.1. General. The finished product shall be visibly free from oxide or scale of any nature, grease, oil, residual lubricants, and other extraneous materials. Cracks, laps, seams, gouges and fins shall be unacceptable.

4.2. Porosity and Inclusions. Indications of internal porosity and non-metallic inclusions greater than 0.020 inch or 3% of the thickness, whichever is smaller, shall be unacceptable. Those indications in the range 0.010 inch to 0.020 inch or 2% of the thickness, whichever is smaller, shall be a minimum of 0.50 inch apart; those indications less than 0.010 inch shall be a minimum of 0.12 inch apart.

4.3. Surface Rework. All surface pores, gouges, and other defects deeper than 0.005 inch or 3% of the thickness, whichever is smaller, shall be unacceptable. Surface imperfections may be faired smooth to remove any notch effect provided dimensional tolerances are still maintained.

5. QUALITY ASSURANCE PROVISIONS

5.1. Vendor Responsibility. The manufacturer shall make all tests and inspections of the material covered by this specification before shipment, unless otherwise specified. All test and inspection results shall be furnished to the purchaser.

5.2. Customer Review. The purchaser or his representative may witness the testing and inspection of the material. The manufacturer shall give the purchaser ample notice of the time and place of designated tests. If the purchaser's representative is not present at this time and a new date is not set, the requirement for purchaser's inspection at the place of testing is waived. When the purchaser's representative is present at the appointed time and place, the manufacturer shall afford him, without charge, all reasonable facilities to assure that the material is being furnished in accordance with this specification. This inspection shall not interfere unnecessarily with production operations.

5.3. Sample Selection. Care shall be exercised to insure that the samples selected for testing and chemical analyses are representative of the material and uncontaminated by the sampling procedure. Samples for the determination of mechanical properties shall be selected so as

to consume a minimum amount of material, i.e., specimens may be taken transverse to the final working direction from plate and sheet and from strip if of sufficient width. If there is any question about the sampling technique or the analysis, the methods for sampling and analysis shall be those agreed to by the buyer and seller. The location of test samples shall be reported in the certificate of compliance.

5.4. Test Methods

5.4.1. Chemical Analysis. Chemical analyses shall be conducted by mutually acceptable procedures, such as the vacuum fusion methods for gases, the combustion method for carbon, and the spectrochemical methods for metallic elements. Disputes shall be settled by accepted referee methods, such as the ASTM "Methods for Chemical Analyses of Reactor and Commercial Columbium."

5.4.2. Tensile Test. The tension test shall be conducted in accordance with ASTM Designation E8-57T, "Methods of Tension Testing of Metallic Materials." Yield strength shall be determined by the offset (0.2%) method. The tensile properties shall be determined using a strain rate of 0.005 inch per inch per minute up to 0.6% offset, and then 0.05 inch, plus or minus 0.02 inch, per inch per minute to fracture.

5.4.3. Stress-Rupture Test. Stress-rupture properties of specimens shall be determined by mutually acceptable testing techniques. Suggested testing techniques for determining stress-rupture properties are:

Specimens shall be tested in a vacuum of 1×10^{-6} torr or better. The vacuum system shall incorporate an optically tight liquid nitrogen cold trap or a getter-ion pump.

Specimens shall be held for a half hour at the test temperature before application of load.

Test temperature shall be maintained at plus or minus 10°F during the test.

5.4.4. Grain Size. Grain size determinations shall be made according to ASTM Specification E112-61, "Estimating the Average Grain Size of Metals."

5.5. Number of Tests Required. Representative test specimens from the finished product representing each ingot and each lot of material shall be taken to determine conformity to this specification. The minimum frequency of these tests shall be:

Finished Product Chemistry - one per lot per ingot

Tensile Test - two per lot per ingot

Stress-Rupture Test - two per lot per ingot

Bend Test - two per lot per ingot

Grain Size - two per lot per ingot

Microhardness Traverse - one per lot per ingot

5.6. Retest and Rework

5.6.1. Surface Contamination. Any sample or specimen exhibiting obvious surface contamination or improper preparation which disqualifies it as a truly representative sample shall be replaced with a new sample.

5.6.2. Rework. If inspection and test results of a lot do not conform to the requirements of this specification, the lot may be reworked at the option of the manufacturer. The lot shall be acceptable if all test results, after reworking, conform to this specification.

5.7. Inspection

5.7.1. Methods of Inspection

5.7.1.1. Radiographic. Whenever specified, the product shall be radiographed and found free of porosity and inclusions as specified in paragraph 4.2 using the technique described in AMS 2635, "Radiographic Inspection." The radiographs and product shall be identified so the exact position of each radiograph can be correlated with the specific area on the particular product.

5.7.1.2. Ultrasonic. Unless otherwise agreed to by the purchaser and the vendor, the material shall be inspected ultrasonically.

5.7.1.2.1. Method and Equipment. The finished products shall be ultrasonically inspected by the immersed technique at 5 mc or above. Transducers shall be no larger than 0.75-inch diameter. Surface finishes shall be no rougher than 125 rms. Inspection shall be by longitudinal wave and by shear wave in two perpendicular directions, i.e., longitudinal and transverse shear.

Transducers for the shear wave inspection shall be focused, preferably cylindrically, to a beam no more than 0.125-inch wide in its smaller dimension (where it enters the material being inspected). Cylindrically-focused transducers shall not exceed 2 inches in length. The focal distance shall be adjusted when the transducer is beamed perpendicular to the surface of the calibration piece; then this focal distance shall be maintained throughout the actual inspection. After the focal distance is established, an appropriate shear wave angle shall be set and the calibration notch indication shall be set at 80% on the indication where the sound beam traverses one or two thicknesses of the sheet (depending on whether the notch is on the far side or incident side of the sheet). Calibration gain settings shall be recorded when the calibration defect is on both the incident and the far side of the sheet. If there is any difference in the indication, that gain setting giving an 80% indication from the side which produces the smaller indication shall be used for inspection. Calibration shall be done before and after the ultrasonic inspection or at the beginning and end of each work shift. If the magnitude of indication from the calibration notch differs 10% or more from the previous calibration, all material inspected since then shall be reinspected.

5.7.1.2.2. Calibration of Plate. Calibration shall be on notches and holes in a segment of the material reserved solely for calibration purposes. The depth of the notches shall be 0.005 inch, the width shall be 0.005 inch and the length greater than the ultrasonic beam width. The notches shall be placed on the surface of the calibration piece perpendicular to the direction of the intended shear wave inspection, i.e., transverse and longitudinal and at least 1 inch from the edge of the plate. In addition, a 0.020-inch diameter hole shall be made in the calibration piece parallel to the surface to a depth of at least 0.750 inch at a point one-half the thickness of the plate. If the thickness of the plate exceeds 0.750 inch, similar holes shall also be made at points one-quarter and three-quarters of the plate thickness. Calibration settings to achieve 80% amplitude of the notches and holes, along with the magnitude of the other applicable calibration defects, shall be recorded. For example, on plate using a shear wave, the notch on the near surface should be set at 80% and the amplitude recorded for

the indications from the hole and notch on the far surface. Gain settings should be recorded to achieve 80% as above and 80% on each of the other applicable calibration defects. For longitudinal wave inspection, only the 0.020-inch diameter holes shall be used for calibration.

5.7.1.2.3. Calibration of Sheet and Strip. The sheet shall be inspected by a shear wave beam point in both longitudinal and transverse directions. Calibration shall be done on notches cut perpendicular to the direction of the beam in pieces of sheet of the same material and thickness as that to be inspected. If that portion is later trimmed and scrapped, the calibration notches may be made on a section of the actual sheet. The depth of the calibration notches shall be 3% of the sheet thickness; width, no greater than the depth; length, no more than 1 inch. All notches shall be at least 1 inch from the edge of the sheet. Duplicate notches may be made on the opposite face of the sheet in locations where the sound beam will not intersect both notches in a single traverse, or the sheet may be turned over during calibration to determine the relative response from the calibration notch on both the incident and far side of the sheet.

5.7.1.2.4. Evaluation. Evaluation during inspection shall be made against the appropriate calibration defect. For example, with shear wave, the defects on or near the far surface shall be compared to the calibration from the far surface notch; defects near the center shall be compared to the calibration from the hole at the appropriate depth; defects on the near surface shall be compared to the calibration from the near surface notch.

5.7.1.2.5. Reports. The ultrasonic inspection report shall contain the equipment serial numbers, calibration amplitudes and gain settings and the amplitude and location of each defect whose amplitude is 60% or greater.

5.7.1.2.6. Rejection. The above procedure shall be followed, and indications of defects which exceed the magnitude obtained from the appropriate calibrated notch in the sample shall be cause for rejection, unless otherwise agreed by the purchaser and vendor.

5.7.1.3. Penetrant Inspection. The exterior surface of the product shall be penetrant inspected and found free of flaws as specified in paragraph 4.3 using AMS 2645, "Fluorescent Penetrant Inspection," or AMS 2646, "Contrast Dye Penetrant Inspection." All parts thus inspected shall be marked with ink stamps as described in the specification; impression stampings or etching shall be unacceptable.

5.7.1.4. Reports. The manufacturer shall supply at least three copies of a report showing inspection results for each lot of material in the shipment. The report shall also include the number of the specification and the purchase order or contract number.

5.8. Rejection. Material not conforming to this specification or to any authorized modification shall be subject to rejection. Unless otherwise specified, rejected material may be returned to the manufacturer at the manufacturer's expense if the purchaser does not receive other instructions for disposition within three weeks after notice of rejection.

5.9. Referee. If the manufacturer and the purchaser disagree about the conformance of the material to the requirements of this specification or any special test specified by the purchaser, a mutually acceptable referee's test shall be used to determine conformance.

6. PREPARATION FOR DELIVERY

6.1. Identification. Each bundle, box or carton shall be legibly and conspicuously marked or tagged with the number of this specification, purchase order or contract number, type, ingot number, lot number, nominal size, and the gross, net, and tare weights. When each bundle, box or carton consists of components from more than one ingot number or lot number, each component shall be identified individually.

6.2. Packing. Each individual item shall be wrapped in heavy gauge polyethylene film or other similar material and packed in a manner assuring safe delivery when properly transported by any common carrier.

7. DEFINITION

7.1. Lot. A lot shall include all material of the same size, shape, condition and finish from one heat of material and which has received the same processing, has been annealed in the same vacuum annealing charge and has been processed simultaneously in all operations in which temperatures may reach 500°F or above. When process temperatures and environments are closely controlled or when closely adjacent sizes receive similar processing, lots may be combined for chemical, tensile and stress-rupture tests only, provided prior written approval has been obtained from the General Electric Company.

7.2. Check Analysis. An analysis may be requested by the purchaser of the metal after it has been processed into finished mill forms, to verify the composition within a heat or lot. Check analysis tolerances do not broaden the specified heat analysis requirements but rather cover variations between laboratories in the measurement of the chemical content.

7.3. Significance of Numerical Limits. For determining compliance with the specified limits for requirements of the properties listed below, an observed value or a calculated value shall be rounded off using the rounding-off method in ASTM Designation E29-58T, "Recommended Practices for Designating Significant Places in Specified Limiting Values."

<u>Test</u>	<u>Rounded-Off Unit for Observed or Calculated Value</u>
Chemical composition and dimensional tolerances (when expressed decimally)	Nearest unit in the last right-hand place of figures of the specified limit
Tensile strength	Nearest 100 psi
Elongation	Nearest 1%
Rupture life	Nearest 0.1 hour

SPECIFICATION

SEAMLESS TUBING AND PIPE: D-43
(Cb-10W-1Zr-0.1C) ALLOY

prepared for
NATIONAL AERONAUTICS AND SPACE ADMINISTRATION
Contract NAS 3-2547

SPACE POWER AND PROPULSION SECTION
MISSILE AND SPACE DIVISION
GENERAL ELECTRIC COMPANY
CINCINNATI, OHIO 45215

SPECIFICATION

SEAMLESS TUBING AND PIPE: D-43
(Cb-10W-1Zr-0.1C) ALLOY

1. SCOPE

1.1. Scope. This specification covers D-43 (Cb-10W-1Zr-0.1C) alloy in tube and pipe form intended for high temperature structural applications and alkali metal containment.

2. APPLICABLE DOCUMENTS

2.1. Government Documents. None

2.2. Non-Government Documents

ASTM Designation E8-57T
(26 December 1957)

Method of Tension Testing of
Metallic Materials

ASTM Designation E29-58T
(1958)

Recommended Practices for
Designating Significant Places
in Specified Limiting Values

ASTM Designation (Pending)

Methods for Chemical Analysis
of Reactor and Commercial
Columbium

ASTM Designation E112-61
(1961)

Estimating Average Grain Size
of Metals

AMS 2635
(15 August 1958)

Radiographic Inspection

AMS 2645
(1 March 1955)

Fluorescent Penetrant Inspec-
tion

AMS 2646
(1 March 1955)

Contrast Dye Penetrant Inspec-
tion

3. REQUIREMENTS

3.1. Acknowledgments. The vendor shall mention this specification in all quotations and all purchase order acknowledgments.

3.2. Manufacture. Material covered by this specification shall be made from ingots which have been double vacuum melted by the electron beam and/or consumable electrode arc melting processes. Breakdown operations shall be performed with conventional extrusion, forging, tube reducing and drawing equipment normally found in primary ferrous and nonferrous plants.

3.3. Processing. The starting stock size, processing temperatures, percentages of reduction, in-process annealing temperatures and times shall be selected by the vendor to achieve the grain size range specified in paragraph 3.6 and mechanical properties specified in paragraph 3.7. The total amount of reduction from the turned ingot to the final product shall exceed 75%. The amount of final reduction for each mill product, imparted just prior to the final vacuum heat treatment and the total reduction since the last recrystallization anneal shall be reported in the certificate of compliance.

3.4. Condition

3.4.1. General. The finished product will be supplied in the recrystallized condition throughout the cross-sectional area to the grain size range specified in paragraph 3.6.

3.4.2. Heat Treatment. All annealing shall be carried out in a vacuum of less than 1×10^{-5} torr. All mill products to be annealed shall be thoroughly degreased, chemically cleaned and protected from furnace parts by a layer of fresh tantalum, columbium or Cb-1Zr alloy foil 0.002-inch thick or greater. When annealing is carried out in a vacuum greater than 1×10^{-5} torr, with the prior approval of the purchaser, all mill products shall be enclosed in a chemically cleaned tantalum, columbium or Cb-1Zr alloy retort or wrapped in a minimum of two layers of fresh tantalum, columbium or Cb-1Zr alloy foil 0.002-inch thick or greater. The conditions of final annealing shall be reported in the certificate of compliance.

3.4.3. Contamination. All items are to be free of contamination or internal oxidation. After final heat treatment, the material shall be examined metallographically for evidence of possible contamination caused by unsatisfactory heat treating atmospheres or processing conditions. A microhardness traverse shall show a hardness increase not

greater than 50 VHN from the center to the surface of a cross-sectional sample of the final product. At the discretion of the purchaser, samples taken to include at least one surface of the final product, and not exceeding 0.050-inch thick, may be chemically analyzed by the purchaser for oxygen, nitrogen, hydrogen and carbon. The analyses shall not exceed the limits set forth in paragraph 3.5.3. Any indication of contamination shall be cause for rejection of all material represented by that sample. The material shall be acceptable if the contaminated layer is completely eliminated before shipment by a machining operation within the specified dimensions and tolerances.

3.5. Chemical Composition

3.5.1. Ingot/Billet Composition. The chemical composition of ingots and billets for conversion to finished products shall conform to Table I (page 5). A minimum of four analyses shall be obtained as follows: ingot top-center, mid-radius and edge, and ingot bottom-center; all analyses must conform to ranges stated in Table I.

3.5.2. Final Product Composition. The manufacturer's ingot analyses shall be considered the chemical analysis for products supplied under this specification (Table I) except carbon, oxygen, nitrogen and hydrogen content which shall be determined on the finished product.

3.5.3. Check Analysis. Finished product analysis shall not exceed the following limits or variations:

For Wall Thicknesses 0.020 Inch or Greater

<u>Element</u>	<u>Check Analysis Limits, ppm</u>		<u>Permissible Variations in Check Analysis, ppm</u>
	<u>Max.</u>	<u>Min.</u>	
Carbon	1200	800	± 50
Oxygen	200	-	+ 20
Nitrogen	100	-	+ 10
Hydrogen	20	-	+ 5

For Wall Thicknesses Less Than 0.020 Inch

<u>Element</u>	<u>Check Analysis Limits, ppm</u>		<u>Permissible Variations in Check Analysis, ppm</u>
	<u>Max.</u>	<u>Min.</u>	
Carbon	1200	800	± 50

TABLE I
CHEMICAL COMPOSITION
D-43 (Cb-10W-1Zr-0.1C) ALLOY

<u>Element</u>	<u>Minimum Content</u> <u>ppm</u>	<u>Maximum Content</u> <u>ppm</u>
Carbon	800	1200
Nitrogen	-	75
Oxygen	-	100
Hydrogen	-	100
Tantalum	-	1000
Molybdenum	-	200
Nickel	-	200
Cobalt	-	50
Iron	-	200
Tungsten	9.0 w/o	11.0 w/o
Zirconium	0.75 w/o	1.25 w/o
Columbium	Remainder	-

<u>Element</u>	<u>Check Analysis Limits, ppm</u>		<u>Permissible Variations in Check Analysis, ppm</u>
	<u>Max.</u>	<u>Min.</u>	
Oxygen	400	-	+ 20
Nitrogen	100	-	+ 10
Hydrogen	20	-	+ 5

3.6. Grain Size. The grain size of the final products shall conform to the following limits:

<u>Product Wall Thickness, Inches</u>	<u>Minimum Allowable ASTM Grain Size No.</u>	<u>Allowable Spread in ASTM Grain Size Nos. in Any One Item</u>	<u>% R_x Minimum</u>
Less than 0.010	6	2	90
0.010 to 0.065	6	2	90
0.065 to 0.125	5	2	90
0.125 to 0.250	4	2	90
0.250 to 0.500	3	3	90

3.7. Mechanical Properties. The final product shall satisfy the following mechanical property requirements:

3.7.1. Room Temperature Tensile Properties. Representative samples of the material in final form shall be capable of the following property limits at room temperature (65°+85°F).

<u>Ultimate Tensile Strength, ksi</u>		<u>0.2% Yield Strength, ksi</u>		<u>Elong., %(1)</u>
<u>Minimum</u>	<u>Maximum</u>	<u>Minimum</u>	<u>Maximum</u>	<u>Minimum</u>
70	90	50	70	15

(1) % Elongation in 4D for Threaded or Button-Head Test Specimens; in 2 Inches for Flat Specimens.

3.7.2. Stress-to-Rupture Tests. The material shall be capable of achieving the following stress-rupture life under suitable environmental conditions (see paragraph 5.4.3).

<u>Test Temp., °F</u>	<u>Stress, ksi</u>	<u>Minimum Life Hours</u>
2200	12	20

Chemical analysis of stress-rupture specimens after test shall demonstrate that the degree of environmental contamination did not exceed the following limits: total increase in oxygen plus nitrogen content--less than 100 ppm; increase in hydrogen content--less than 5 ppm; increase in carbon content--less than 10 ppm. The following limits shall apply to the check analyses of analytical results:

Carbon	±	10 ppm
Oxygen	±	50 ppm
Nitrogen	±	50 ppm
Hydrogen	±	2 ppm

3.7.3. Hydrostatic Test. Each tube, 1/8 inch or larger in outside diameter with a wall thickness of 0.015 inch or over, shall be tested to a hydrostatic pressure sufficient to produce a fiber stress of 12,000 psi. The test pressure, not to exceed 10,000 psi, shall be determined by the equation ($P = 2St/D$), where:

- P = hydrostatic test pressure in pounds per square inch;
- S = 12,000 psi;
- t = average wall thickness of the tube in inches;
- D = outside diameter of the tube in inches.

3.7.4. Flare Test. A section of the heat treated tube shall be capable of being flared without cracking. The flare shall be made with a tool having a 60-degree included angle until the specified outside diameter has been increased by 15%.

3.8. Tolerances

3.8.1. Diameter and Wall Thickness. The permissible variations in diameter and wall thickness of tube shall not exceed those prescribed in Table II (refer to page 9).

3.8.2. Length. When tube is ordered cut-to-length, the usable length shall not be less than that specified, but a variation of plus 1/8 inch will be permitted in lengths up to 6 feet. In lengths over 6 feet, a variation of plus 1/4 inch will be permitted, unless otherwise specified.

3.8.3. Straightness. The tube shall be free of bends or kinks. For lengths up to 10 feet, the maximum bow shall not exceed one part in 1200; for lengths greater than 10 feet, the maximum bow shall not exceed one part in 600, unless otherwise agreed upon.

3.9. Reports. The manufacturer shall supply at least three copies of a report showing non-proprietary manufacturing methods, processing conditions, and test procedures and results for each lot of material in the shipment. The report shall also include the number of the specification and the purchase order or contract number.

4. MAXIMUM ALLOWABLE DISCONTINUITIES

4.1. General. Cracks, laps, seams, fins, and tears shall be unacceptable. The surface shall also be free from oxide or scale of any nature, grease, oil, residual lubricants, or other extraneous material.

4.2. Porosity and Inclusions. Indications with dimensions greater than 3% of the wall thickness shall be unacceptable. Indications with dimensions in the range of 1% to 3% of wall thickness must be a minimum of 0.50 inch apart. Indications with dimensions less than 1% of the wall thickness must be a minimum of 0.12 inch apart.

4.3. Surface Rework. Defects less than 3% of the nominal wall thickness detected by penetrant or ultrasonic inspection may be removed by grinding provided the wall thickness is not decreased below that permitted in Table II (refer to page 9).

5. QUALITY ASSURANCE PROVISIONS

5.1. Vendor Responsibility. The manufacturer shall make all tests and inspections of the material covered by this specification before shipment unless otherwise specified. All test and inspection results shall be furnished to the purchaser.

TABLE II
PERMISSIBLE VARIATIONS IN TUBE DIMENSIONS

Nominal OD <u>Inches</u>	OD <u>Inch</u>	ID <u>Inch</u>	Wall Thickness <u>%</u>
0.187 to but not incl. 0.625	± 0.004	± 0.004	± 10
0.625 to but not incl. 1.000	± 0.005	± 0.005	± 10
1.000 to but not incl. 2.000	± 0.0075	± 0.0075	± 10
2.000 to but not incl. 3.000	± 0.010	± 0.010	± 10
3.000 to but not incl. 4.000	± 0.0125	± 0.0125	± 10

NOTES: -----

- (1) Tolerances are applicable to only the two dimensions specified on the purchase order, e.g., outside diameter and wall; inside diameter and wall; outside diameter and inside diameter.
- (2) For tolerances applicable for very small tubes (less than 0.187-inch diameter) or very thin-wall tubes (less than 0.010-inch thick), the producer shall be consulted.
- (3) For tubes having an inside diameter less than 60% of the outside diameter or a wall 3/4 inch or over thick, which cannot be successfully drawn over a mandrel, the inside diameter may vary by an amount equal to plus or minus 10% of the wall thickness. The wall thickness of these tubes may vary plus or minus 12.5% from that specified.
- (4) Ovality measured at any cross section: For tubes with nominal wall thickness less than 3% of the nominal outside diameter, the ovality tolerances are double the tolerances in column 2 or 3. For ovality tolerances for tubes with wall thickness less than 2% nominal outside diameter, the producer shall be consulted.

5.2. Customer Review. The purchaser or his representative may witness the testing and inspection of the material. The manufacturer shall give the purchaser ample notice of the time and place of designated tests. If the purchaser's representative is not present at this time and a new date is not set, the requirement for purchaser's inspection at the place of testing is waived. When the purchaser's representative is present at the appointed time and place, the manufacturer shall afford him, without charge, all reasonable facilities to assure that the material is being furnished in accordance with this specification. This inspection shall not interfere unnecessarily with production operations.

5.3. Sample Selection. Care shall be exercised to insure that the sample selected for testing is representative of the material and uncontaminated by the sampling procedure. If there is any question about the sampling technique or the analysis, the methods for sampling and analysis shall be those agreed to by the buyer and seller. The specimen configuration selected for the performance of the testing required in paragraphs 5.4.2 and 5.4.3 shall be mutually agreed upon by the vendor and the purchaser prior to the placement of a purchase order. The location of all test samples shall be reported in the certificate of compliance.

5.4. Test Methods

5.4.1. Chemical Analysis. Chemical analyses shall be conducted by mutually acceptable procedures, such as the vacuum fusion methods for gases, the combustion method for carbon, and the spectrochemical methods for metallic elements. Disputes shall be settled by accepted referee methods, such as the ASTM "Methods for Chemical Analysis of Reactor and Commercial Columbium."

5.4.2. Tensile Test. The tension test shall be performed in accordance with ASTM Designation E8-57T, "Methods of Tension Testing of Metallic Materials." Yield strength shall be determined by the offset (0.2%) method. The tensile properties shall be determined using a strain rate of 0.005 inch per inch per minute up to 0.6% offset and then 0.050 inch, plus or minus 0.020 inch, per inch per minute to fracture.

5.4.3. Stress-Rupture Tests. Stress-rupture properties of specimens shall be determined by mutually acceptable testing techniques. Suggested testing techniques for determining stress-rupture properties are:

Specimens shall be tested in a vacuum of 1×10^{-6} torr or better. The vacuum system shall incorporate an optically tight liquid nitrogen cold trap or a getter-ion pump.

Specimens shall be held for a half hour at the test temperature before application of load.

Test temperature shall be maintained at plus or minus 10°F during the test.

5.4.4. Grain Size. Grain size determinations shall be made according to ASTM Specification E112-61, "Estimating the Average Grain Size of Metals."

5.5. Number of Tests Required. Representative test specimens from the finished product representing each ingot and each lot of material shall be taken to determine conformity to this specification. The minimum frequency of these tests shall be:

Finished Product Chemistry - one per lot per ingot

Tensile Test - two per lot per ingot

Stress-Rupture Test - two per lot per ingot

Flare Test - two per lot per ingot

Grain Size - two per lot per ingot

Microhardness Traverse - one per lot per ingot

Hydrostatic Proof Test - 100%

5.6. Retest and Rework

5.6.1. Surface Contamination. Any sample or specimen exhibiting obvious surface contamination or improper preparation which disqualifies it as a truly representative sample shall be replaced with a new sample.

5.6.2. Rework. If inspection and test results of a lot do not conform to the requirements of this specification, the lot may be reworked at the option of the manufacturer. The lot shall be acceptable if all test results, after reworking, conform to this specification.

5.7. Inspection

5.7.1. Methods of Inspection

5.7.1.1. Radiographic. When specified, the product shall be radiographed and found free of porosity and inclusions as specified in paragraph 4.2 using the techniques described in AMS 2635, "Radiographic Inspection." The radiographs and product shall be identified so that the exact position of each radiograph can be correlated with the specific area on a particular product.

5.7.1.2. Ultrasonic Inspection. Unless otherwise agreed to by the purchaser and the vendor, the material shall be inspected ultrasonically.

5.7.1.2.1. Method and Equipment. Ultrasonic inspection shall be by the immersed technique at 5 mc or higher frequency using focused transducers. Inspection shall be by both circumferential and axial shear techniques with longitudinal wave being added when the wall thickness is greater than 0.150 inch. For longitudinal wave technique and for circumferential shear, transducers up to 2 inches long may be used with or without automatic equipment to rotate the tube past the transducer. If spiral pattern inspection traverse is not used, steps must be taken to assure that the ultrasonic beam remains in the same position relative to the tubing so the beam-to-tubing angle remains constant. For axial (longitudinal) shear, transducers must have no greater than 0.5 inch axial length. Transducers must be cylindrically focused for a diameter range which includes the tubing on which it is to be used.

5.7.1.2.2. Calibration. Calibration shall be on notches (a total of four, two axial and two circumferential), cut in the tube on both the outside and inside surface unless otherwise specified. The depth of the notches shall be 3% of the wall thickness to a minimum depth of 0.001 inch; the width, no greater than depth; the length, at least that of the ultrasonic beam with a maximum length of 1 inch. Material having a wall thickness greater than 0.150 inch shall also have an 0.020 inch diameter hole machined into the wall in the longitudinal direction at mid-point of the wall thickness. Focusing shall be done to maximize the indication from the inside diameter notch placed properly for the type of inspection contemplated. After focusing is completed, the inside diameter indication shall be set at 80% and gain setting recorded. Gain setting for 80% on the outside diameter notch shall also be recorded. Inspection shall be at the gain setting for the inside diameter indication. A distance corresponding to the wall thickness shall be marked on the oscilloscope. Focal distance to the part to be inspected shall be set to that used for the calibration

piece before beginning inspection. Calibration shall be done both before and after the inspection or at the beginning and end of each work shift. If calibration has changed (gain change greater than 5%), all inspections since the previous calibration shall be repeated.

5.7.1.2.3. Rejection. Rejection shall be by any indication which exceeds the amplitude of the respective calibration indication; i.e., inside diameter defects shall be compared to the indication from the notch on the inside diameter, and outside diameter defects shall be compared to the indication from the notch on the outside diameter. Defects less than half the thickness from the surface or less than 0.150 inch from the surface, whichever is smaller, shall be compared to the outside diameter calibration indication. Defects more than half the thickness from the incident surface or more than 0.150 inch from the surface shall be compared to the indications from the inside diameter calibration notch.

5.7.1.2.4. Reports. The ultrasonic inspection report shall contain the equipment serial numbers, calibration amplitudes and gain settings and the amplitude and location of each defect whose amplitude is 60% or greater.

5.7.1.3. Penetrant Inspection. The exterior surface of the product shall be penetrant inspected and found free of flaws as specified in paragraph 4.3 using AMS 2645, "Fluorescent Penetrant Inspection," or AMS 2646, "Contrast Dye Penetrant Inspection." All parts thus inspected shall be marked with ink stamps as described in the specification; impression stampings or etching shall be unacceptable.

5.7.1.4. Reports. The manufacturer shall supply at least three copies of a report showing inspection results for each lot of material in the shipment. The report shall also include the number of the specification and the purchase order or contract number.

5.8. Rejection. Material not conforming to this specification or to any authorized modification shall be subject to rejection. Unless otherwise specified, rejected material may be returned to the manufacturer at the manufacturer's expense if the purchaser does not receive other instructions for disposition within three weeks after notice of rejection.

5.9. Referee. If the manufacturer and the purchaser disagree concerning the conformance of the material to the requirements of this

specification or any special test specified by the purchaser, a mutually acceptable referee's test shall be used to determine conformance.

6. PREPARATION FOR DELIVERY

6.1. Identification. Each bundle, box, or carton shall be legibly and conspicuously marked or tagged with the number of this specification, purchase order or contract number, type, ingot number, lot number, nominal size, and the gross, net and tare weights. When each bundle, box or carton consists of components from more than one ingot number or lot number, each component shall be identified individually.

6.2. Packing. The ends of each pipe or tube shall be sealed with suitable plastic caps and each individual item shall be wrapped in heavy gauge polyethylene or similar material and packed in a manner assuring safe delivery when properly transported by a common carrier.

7. DEFINITIONS

7.1. Lot. A lot shall include all material of the same size, shape, condition and finish from one heat of material and which has received the same processing, has been annealed in the same vacuum annealing charge and has been processed simultaneously in all operations in which temperatures may reach 500°F or above. When process temperatures and environments are closely controlled or when closely adjacent sizes receive similar processing, lots may be combined for chemical, tensile and stress-rupture tests only, provided prior written approval has been obtained from the General Electric Company.

7.2. Check Analysis. An analysis either made or requested by the purchaser of the metal, after it has been processed into finished mill forms, for the purpose of verifying the composition within a heat or lot. Check analysis tolerances do not broaden the specified heat analysis requirements but rather cover variations between laboratories in the measurement of the chemical content.

7.3. Significance of Numerical Limits. For determining compliance with the specified limits for requirements of the properties listed below, an observed value or a calculated value shall be rounded off using the rounding-off method in ASTM Designation E29-58T, "Recommended Practices for Designating Significant Places in Specified Limiting Values."

<u>Test</u>	<u>Rounded-Off Unit for Observed or Calculated Value</u>
Chemical composition and dimensional tolerances (when expressed decimally)	Nearest unit in the last right- hand place of figures of the specified limit
Tensile strength	Nearest 100 psi
Elongation	Nearest 1%
Rupture life	Nearest 0.1 hour

SPECIFICATION

FOIL: D-43 (Cb-10W-1Zr-0.1C) ALLOY

prepared for
NATIONAL AERONAUTICS AND SPACE ADMINISTRATION
Contract NAS 3-2547

SPACE POWER AND PROPULSION SECTION
MISSILE AND SPACE DIVISION
GENERAL ELECTRIC COMPANY
CINCINNATI, OHIO 45215

SPECIFICATION

FOIL: D-43 (Cb-10W-1Zr-0.1C) ALLOY

1. SCOPE

1.1. Scope. This specification covers D-43 (Cb-10W-1Zr-0.1C) alloy in foil form intended for high temperature non-structural applications.

2. APPLICABLE DOCUMENTS

2.1. Government Documents. None

2.2. Non-Government Documents

ASTM Designation (Pending)

Methods for Chemical Analysis
of Reactor and Commercial
Columbium

3. REQUIREMENTS

3.1. Acknowledgments. The vendor shall mention this specification in all quotations and all purchase order acknowledgments.

3.2. Manufacture. Material covered by this specification shall be made from ingots which have been double vacuum melted by the electron beam and/or consumable electrode arc melting processes. Breakdown operations shall be performed with conventional extrusion, forging and rolling equipment normally found in primary ferrous and non-ferrous plants.

3.3. Processing. The starting stock size, processing temperatures, percentages of reduction, in-process annealing temperatures and times shall be selected by the vendor.

3.4. Condition. The finished product shall be supplied in the fully recrystallized condition throughout the cross-sectional area. All annealing shall be carried out in a vacuum of less than 1×10^{-5} torr. All

mill products to be annealed shall be thoroughly degreased, chemically cleaned and protected from furnace parts by a layer of fresh tantalum, columbium or Cb-1Zr foil 0.002-inch thick or greater. When annealing is carried out in a vacuum greater than 1×10^{-5} torr, with the prior approval of the purchaser, all mill products shall be enclosed in a chemically cleaned tantalum, columbium or Cb-1Zr retort or wrapped in a minimum of two layers of fresh tantalum, columbium or Cb-1Zr alloy foil 0.002-inch thick or greater. The conditions of final annealing shall be reported in the certificate of compliance.

3.5. Chemical Composition

3.5.1. Ingot/Billet Composition. The chemical composition of ingots and billets for conversion to finished products shall conform to Table I (page 4). A minimum of four analyses shall be obtained as follows: ingot top-center, mid-radius and edge, and ingot bottom-center; all analyses must conform to ranges stated in Table I.

3.5.2. Final Product Composition. The manufacturer's ingot analyses shall be considered the chemical analyses for products supplied under this specification (Table I), except carbon, oxygen, nitrogen and hydrogen content which shall be determined on the finished product.

3.5.3. Check Analysis. Finished product analysis shall not exceed the following limits or variations:

<u>Element</u>	<u>Check Analysis Limits, Max., ppm</u>	<u>Permissible Variations in Check Analysis, ppm</u>
Carbon	800 min; 1200 max	± 50
Oxygen	300	+ 20
Nitrogen	100	+ 10
Hydrogen	20	+ 5

3.6. Bend Ductility. Representative samples of the materials in final form shall withstand a 180° bend without failure.

3.7. Tolerances

3.7.1. Definition. Foil includes material less than 12 inches wide and up to and including 0.010-inch thick.

TABLE I
CHEMICAL COMPOSITION
D-43 (Cb-10W-1Zr-0.1C) ALLOY

<u>Element</u>	<u>Minimum Content</u> <u>ppm</u>	<u>Maximum Content</u> <u>(ppm)</u>
Carbon	800	1200
Nitrogen	-	75
Oxygen	-	100
Hydrogen	-	100
Tantalum	-	1000
Molybdenum	-	200
Nickel	-	200
Cobalt	-	50
Iron	-	200
Tungsten	9.0 w/o	11.0 w/o
Zirconium	0.75 w/o	1.25 w/o
Columbium	Remainder	-

3.7.2. Dimensions. Foil dimensions shall conform to the following limits:

<u>Material Thickness</u> <u>Inches</u>	<u>Thickness Tolerances</u> <u>Inch</u>	<u>Width Tolerance</u> <u>Inch</u>
Less than 0.003	+0.0008, -0.0000	+0.031, -0.000
0.003 to 0.005	+0.001	+0.031, -0.000
0.005 to and incl. 0.010	<u>±</u> 0.0015	+0.031, -0.000

3.8. General. The finished product shall be visibly free from oxide or scale of any nature, grease, oil, residual lubricants, and other extraneous materials. Cracks, laps, seams, gouges, and fins shall be unacceptable.

4. QUALITY ASSURANCE PROVISIONS

4.1. Vendor Responsibility. The manufacturer shall make all tests and inspections of the material covered by this specification before shipment, unless otherwise specified. All test and inspection results shall be furnished to the purchaser.

4.2. Sample Selection. Care shall be exercised to insure that the samples selected for testing and chemical analyses are representative of the material and uncontaminated by the sampling procedure. If there is any question about the sampling technique or the analysis, the methods for sampling and analysis shall be those agreed to by the buyer and seller.

4.3. Chemical Analysis. Chemical analyses shall be conducted by mutually acceptable procedures, such as the vacuum fusion methods for gases, the combustion method for carbon, and the spectrochemical methods for metallic elements. Disputes shall be settled by accepted referee methods, such as the ASTM "Methods for Chemical Analyses of Reactor and Commercial Columbium."

4.4. Number of Tests Required. Representative test specimens from the finished product representing each ingot and each lot of material shall be taken to determine conformity to this specification. The minimum frequency of these tests shall be:

Finished Product Chemistry - one per lot per ingot

Bend Test - two per lot per ingot

4.5. Reports. The manufacturer shall supply at least three copies of a report showing non-proprietary manufacturing methods, processing conditions, and test procedures and results for each lot of material in the shipment. The report shall also include the number of the specification and the purchase order or contract number.

4.6. Rejection. Material not conforming to this specification or to any authorized modification shall be subject to rejection. Unless otherwise specified, rejected material may be returned to the manufacturer at the manufacturer's expense if the purchaser does not receive other instructions for disposition within three weeks after notice of rejection.

4.7. Referee. If the manufacturer and the purchaser disagree about the conformance of the material to the requirements of this specification or any special test specified by the purchaser, a mutually acceptable referee's test shall be used to determine conformance.

5. PREPARATION FOR DELIVERY

5.1. Identification. Each bundle, box, or carton shall be legibly and conspicuously marked or tagged with the number of this specification, purchase order or contract number, type, ingot number, lot number, nominal size, and the gross, net, and tare weights. When each bundle, box or carton consists of components from more than one ingot number or lot number, each component shall be identified individually.

5.2. Packing. Each individual item shall be wrapped in heavy gauge polyethylene film or other similar material and packed in a manner assuring safe delivery when properly transported by any common carrier.

6. DEFINITION

6.1. Lot. A lot shall include all material of the same size, shape, condition and finish from one heat of material and which has received the same processing, has been annealed in the same vacuum annealing charge and has been processed simultaneously in all operations in which temperatures may reach 500°F or above. When process temperatures and environments are closely controlled or when closely adjacent sizes receive similar processing, lots may be combined for chemical, tensile and stress-rupture tests only, provided prior written approval has been obtained from the General Electric Company.

6.2. Check Analysis. An analysis, made or requested by the purchaser of the metal after it has been processed into finished mill forms, to verify the composition within a heat or lot. Check analysis tolerances do not broaden the specified heat analysis requirements but rather cover variations between laboratories in the measurement of the chemical content.

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SPPS-65
23 December 1964
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SPECIFICATION

WIRE: D-43 (Cb-10W-1Zr-0.1C) ALLOY

prepared for
NATIONAL AERONAUTICS AND SPACE ADMINISTRATION
Contract NAS 3-2547

SPACE POWER AND PROPULSION SECTION
MISSILE AND SPACE DIVISION
GENERAL ELECTRIC COMPANY
CINCINNATI, OHIO 45215

SPECIFICATION

WIRE: D-43 (Cb-10W-1Zr-0.1C) ALLOY

1. SCOPE

1.1. Scope. This specification covers D-43 (Cb-10W-1Zr-0.1C) alloy in wire form for use as weld filler material in fabricating components intended for high temperature structural applications and alkali metal containment.

2. APPLICABLE DOCUMENTS

2.1. Government Documents. None

2.2. Non-Government Documents

ASTM Designation (Pending)

Methods for Chemical Analysis
of Reactor and Commercial
Columbium

3. REQUIREMENTS

3.1. Acknowledgments. The vendor shall mention this specification in all quotations and all purchase order acknowledgments.

3.2. Manufacture. Material covered by this specification shall be made from ingots which have been double vacuum melted by the electron beam and/or consumable electrode arc melting processes. Breakdown operations shall be performed with conventional extrusion, forging and rolling equipment normally in primary ferrous and nonferrous plants.

3.3. Processing. The starting stock size, processing temperatures, percentages of reduction, in-process annealing temperatures and times shall be selected by the vendor.

3.4. Condition. The finished product shall be supplied in the fully recrystallized condition throughout the cross-sectional area. All annealing shall be carried out in a vacuum less than 1×10^{-5} torr.

All mill products to be annealed shall be thoroughly degreased, chemically cleaned and protected from furnace parts by a layer of fresh tantalum, columbium or Cb-1Zr alloy foil 0.002-inch thick or greater. When annealing is carried out in a vacuum greater than 1×10^{-5} torr, with the prior approval of the purchaser, all mill products shall be enclosed in a chemically cleaned tantalum, columbium or Cb-1Zr alloy retort or wrapped in a minimum of two layers of fresh tantalum, columbium or Cb-1Zr alloy foil 0.002-inch thick or greater. The conditions of final annealing shall be reported in the certificate of compliance.

3.5. Chemical Composition

3.5.1. Ingot/Billet Composition. The chemical composition of ingots and billets for conversion to finished products shall conform to Table I (page 4). A minimum of four analyses shall be obtained as follows: ingot top-center, mid-radius and edge, and ingot bottom-center; all analyses must conform to ranges stated in Table I.

3.5.2. Final Product Composition. The manufacturer's ingot analyses shall be considered the chemical analyses of products supplied under this specification (Table I) except carbon, oxygen, nitrogen and hydrogen content which shall be determined on the finished product.

3.5.3. Check Analysis. The finished product analysis shall not exceed the following limits or variations:

For Material Greater than 0.030 Inch in Diameter

<u>Element</u>	<u>Check Analysis Limits, Max., ppm</u>	<u>Permissible Variations in Check Analysis, ppm</u>
Carbon	800 min; 1200 max	± 50
Oxygen	200	+ 20
Nitrogen	100	+ 10
Hydrogen	20	+ 5

TABLE I
CHEMICAL COMPOSITION
D-43 (Cb-10W-1Zr-0.1C) ALLOY

<u>Element</u>	<u>Minimum Content</u> <u>ppm</u>	<u>Maximum Content</u> <u>ppm</u>
Carbon	800	1200
Nitrogen	-	75
Oxygen	-	100
Hydrogen	-	100
Tantalum	-	1000
Molybdenum	-	200
Nickel	-	200
Cobalt	-	50
Iron	-	200
Tungsten	9.0 w/o	11.0 w/o
Zirconium	0.75 w/o	1.25 w/o
Columbium	Remainder	-

For Material 0.030 Inch and Less in Diameter

<u>Element</u>	<u>Check Analysis Limits, Max., ppm</u>	<u>Permissible Variations in Check Analysis, ppm</u>
Carbon	800 min; 1200 max	± 50
Oxygen	300	+ 20
Nitrogen	100	+ 10
Hydrogen	20	+ 5

3.6. Tolerances

3.6.1. Definition. Wire - material less than 0.125 inch in diameter.

3.6.2. Diameter. The permissible variation in diameter shall not exceed the following limits:

<u>Diameter, Inch</u>	<u>Diameter Variation, Inch</u>
0.005 to 0.009	± 0.0002
0.010 to 0.019	± 0.0003
0.020 to 0.029	± 0.0005
0.030 to 0.061	± 0.001
0.062 to 0.125	± 0.002

3.7. General. The finished product shall be visibly free from oxide or scale of any nature, grease, oil, residual lubricants, and other extraneous materials. Cracks, laps, seams, gouges, and fins shall be unacceptable.

4. QUALITY ASSURANCE PROVISIONS

4.1. Vendor Responsibility. The manufacturer shall make all tests and inspections of the material covered by this specification before shipment, unless otherwise specified. All test and inspection results shall be furnished to the purchaser.

4.2. Sample Selection. Care shall be exercised to insure that the samples selected for testing and chemical analyses are representative of the material and uncontaminated by the sampling procedure. If there is any question about the sampling technique or the analysis, the methods for sampling and analysis shall be those agreed to by the buyer and seller.

4.3. Chemical Analysis. Chemical analyses shall be conducted by mutually acceptable procedures, such as the vacuum fusion methods for gases, the combustion method for carbon, and the spectrochemical methods for metallic elements. Disputes shall be settled by accepted referee methods, such as the ASTM "Methods for Chemical Analysis of Reactor and Commercial Columbium."

4.4. Number of Tests Required. Representative test specimens from the finished product representing each ingot and each lot of material shall be taken to determine conformity to this specification. The minimum frequency of these tests shall be:

Finished Product Chemistry - one per lot per ingot

4.5. Reports. The manufacturer shall supply at least three copies of a report showing non-proprietary manufacturing methods, processing conditions, and test procedures and results for each lot of material in the shipment. The report shall also include the number of the specification and the purchase order or contract number.

4.6. Rejection. Material not conforming to this specification or to any authorized modification shall be subject to rejection. Unless otherwise specified, rejected material may be returned to the manufacturer at the manufacturer's expense if the purchaser does not receive other instructions for disposition within three weeks after notice of rejection.

4.7. Referee. If the manufacturer and the purchaser disagree about the conformance of the material to the requirements of this specification or any special test specified by the purchaser, a mutually acceptable referee's test shall be used to determine conformance.

5. PREPARATION FOR DELIVERY

5.1. Identification. Each bundle, box, or carton shall be legibly and conspicuously marked or tagged with the number of this specification, purchase order or contract number, type, ingot number, lot number,

nominal size and the gross, net, and tare weights. When each bundle, box or carton consists of components from more than one ingot number or lot number, each component shall be identified individually.

5.2. Packing. Each individual item shall be wrapped in heavy gauge polyethylene film or similar material and packed in a manner assuring safe delivery when properly transported by any common carrier.

6. DEFINITIONS

6.1. Lot. A lot shall include all material of the same size, shape, condition and finish from one heat of material and which has received the same processing, has been annealed in the same vacuum annealing charge and has been processed simultaneously in all operations in which temperatures may reach 500°F or above. When process temperatures and environments are closely controlled or when closely adjacent sizes receive similar processing, lots may be combined for chemical, tensile and stress-rupture tests only, provided prior written approval has been obtained from the General Electric Company.

6.2. Check Analysis. An analysis, made or requested by the purchaser of the metal after it has been processed into finished mill forms, to verify the composition within a heat or lot. Check analysis tolerances do not broaden the specified heat analysis requirements but rather cover variations between laboratories in the measurement of the chemical content.

SPECIFICATION

BAR AND ROD: T-111 (Ta-8W-2Hf) ALLOY

prepared for
NATIONAL AERONAUTICS AND SPACE ADMINISTRATION
Contract NAS 3-2547

SPACE POWER AND PROPULSION SECTION
MISSILE AND SPACE DIVISION
GENERAL ELECTRIC COMPANY
CINCINNATI, OHIO 45215

SPECIFICATION

BAR AND ROD: T-111 (Ta-8W-2Hf) ALLOY

1. SCOPE

1.1. Scope. This specification covers T-111 (Ta-8W-2Hf) alloy in bar and rod form intended for high temperature structural applications and alkali metal containment.

2. APPLICABLE DOCUMENTS

2.1. Government Documents. None

2.2. Non-Government Documents

ASTM Designation E8-57T
(26 December 1957)

Method of Tension Testing of
Metallic Materials

ASTM Designation E29-58T
(1958)

Recommended Practices for Des-
ignating Significant Places in
Specified Limiting Values

ASTM Designation E112-61
(1961)

Estimating the Average Grain
Size of Metals

AMS 2635
(15 August 1958)

Radiographic Inspection

AMS 2645
(1 March 1955)

Fluorescent Penetrant Inspec-
tion

AMS 2646
(1 March 1955)

Contrast Dye Penetrant Inspec-
tion

3. REQUIREMENTS

3.1. Acknowledgments. The vendor shall mention this specification in all quotations and all purchase order acknowledgments.

3.2. Manufacture. Material covered by this specification shall be made from ingots which have been double vacuum melted by the electron beam and/or consumable electrode arc melting processes. Breakdown operations shall be performed with conventional extrusion, forging and rolling equipment normally found in primary ferrous and nonferrous plants.

3.3. Processing. The starting stock size, processing temperatures, percentages of reduction, in-process annealing temperatures and times shall be selected by the vendor to achieve the grain size range specified in paragraph 3.6 and mechanical properties specified in paragraph 3.7. The amount of total reduction from the turned ingot to the finished product shall exceed 75%. The amount of final reduction for each mill product, imparted just prior to the final vacuum heat treatment and the total reduction since the previous recrystallization anneal, shall be reported in the certificate of compliance.

3.4. Condition

3.4.1. General. The finished product shall be supplied in the recrystallized condition throughout the cross-sectional area to the grain size range specified in paragraph 3.6.

3.4.2. Heat Treatment. All annealing shall be carried out in a vacuum of less than 1×10^{-5} torr. All mill products to be annealed shall be thoroughly degreased, chemically cleaned and protected from furnace parts by a layer of fresh tantalum, columbium or Cb-1Zr foil 0.002-inch thick or greater. When annealing is carried out in a vacuum greater than 1×10^{-5} torr, with the prior approval of the purchaser, all mill products shall be enclosed in a chemically cleaned tantalum, columbium or columbium-1% zirconium alloy retort or wrapped in a minimum of two layers of fresh tantalum, columbium or columbium-1% zirconium alloy foil 0.002-inch thick or greater. The conditions of final annealing shall be reported in the certificate of compliance.

3.4.3. Contamination. All items are to be free of contamination or internal oxidation. After final heat treatment, the material shall be examined metallographically for evidence of possible contamination caused by unsatisfactory heat treating atmospheres or processing conditions. A microhardness traverse shall show a hardness increase not greater than 50 VHN from the center to the surface of a cross sectional sample of the final product. At the discretion of the purchaser, samples taken to include at least one surface of the final product, and not exceeding 0.050-inch thick, may be chemically analyzed by the

purchaser for oxygen, nitrogen, hydrogen and carbon. The analyses shall not exceed the limits set forth in paragraph 3.5.3. Any indication of contamination shall be cause for rejection of all material represented by that sample. The material shall be acceptable if the contaminated layer is completely eliminated before shipment by a machining operation within the specified dimensions and tolerances.

3.5. Chemical Composition

3.5.1. Ingot/Billet Composition. The chemical composition of ingots and billets for conversion to finished products shall conform to Table I (page 5). A minimum of four analyses shall be obtained as follows: ingot top-center, mid-radius and edge, and ingot bottom-center; all analyses must conform to ranges stated in Table I.

3.5.2. Final Product Composition. The manufacturer's ingot analyses shall be considered the chemical analysis for products supplied under this specification (Table I) except carbon, oxygen, nitrogen and hydrogen content which shall be determined in the finished product.

3.5.3. Check Analysis. Finished product analysis shall not exceed the following limits or variations:

<u>Element</u>	<u>Check Analysis Limits, Max., ppm</u>	<u>Permissible Variations in Check Analysis, ppm</u>
Carbon	50	+ 10
Oxygen	150	+ 20
Nitrogen	75	+ 10
Hydrogen	10	+ 2

3.6. Grain Size. The grain size of the final products shall conform to the following limits:

<u>Product Diameter or Thickness, Inches</u>	<u>Minimum Allowable ASTM Grain Size No.</u>	<u>Allowable Spread in ASTM Grain Size Nos. in Any One Item</u>	<u>% R_x Minimum</u>
0.125 to 0.250	4	2	100
0.250 to 0.500	4	2	100
0.500 to 1.0	4	2	100
1.0 to 2.0	4	2	95
Greater than 2.0	3	3	90

TABLE I
CHEMICAL COMPOSITION
T-111 (Ta-8W-2Hf) ALLOY

<u>Element</u>	<u>Minimum Content</u> <u>ppm</u>	<u>Maximum Content</u> <u>ppm</u>
Carbon	-	50
Nitrogen	-	50
Oxygen	-	100
Hydrogen	-	10
Columbium	-	1000
Molybdenum	-	200
Nickel	-	50
Cobalt	-	50
Iron	-	50
Vanadium	-	20
Tungsten	7.0 w/o	9.0 w/o
Hafnium	1.8 w/o	2.4 w/o
Tantalum	Remainder	-

3.7. Mechanical Properties. The final product shall satisfy the following mechanical property requirements:

3.7.1. Room Temperature Tensile Properties. Representative samples of the material in final form shall be capable of the following property limits at room temperature (65°-85°F).

<u>Ultimate Tensile Strength, ksi</u>		<u>0.2% Yield Strength, ksi</u>		<u>Elong., % in 4D</u>
<u>Minimum</u>	<u>Maximum</u>	<u>Minimum</u>	<u>Maximum</u>	<u>Minimum</u>
80	110	65	100	20

3.7.2. Stress-to-Rupture Tests. The material shall be capable of achieving the following stress-rupture life under suitable environmental conditions (see paragraph 5.4.3).

<u>Test Temp., °F</u>	<u>Stress, ksi</u>	<u>Minimum Life Hours</u>
2400	19	20

Chemical analysis of stress-rupture specimens after test shall demonstrate that the degree of environmental contamination did not exceed the following limits: total increase in oxygen plus nitrogen content--less than 50 ppm; increase in hydrogen content--less than 5 ppm; increase in carbon content--less than 10 ppm. The following limits shall apply to the analytical results:

Carbon	±	10 ppm
Oxygen	±	25 ppm
Nitrogen	±	25 ppm
Hydrogen	±	2 ppm

3.8. Tolerances

3.8.1. Rolled, Swaged, or Drawn Rounds

3.8.1.1. Definition. Rod - 3.5 inches in diameter or less.

3.8.1.2. Diameter. The permissible variation in diameter and the limits of out-of-roundness of descaled rounds shall not exceed those in Table II (refer to page 8).

3.8.1.3. Cut Lengths. Maximum length variation shall be 0.25 inch.

3.8.1.4. Straightness. Maximum deviation shall be 0.050 inch per foot in any length.

3.8.2. Square or Rectangular Bar

3.8.2.1. Definition. Bar - any straight product with a rectangular cross-section 0.187 inch or more thick and less than 5 inches wide.

3.8.2.2. Dimensions. Unless otherwise specified, forged or rolled square and rectangular shapes shall have the following tolerances:

<u>Thickness</u>	<u>Length</u>	<u>Width</u>
± 0.025 inch or $\pm 5\%$, whichever is less	± 0.125 inch	± 0.125 inch

3.8.2.3. Straightness of Bar. Maximum deviation shall be 0.050 inch per foot in any length.

3.9. Reports. The manufacturer shall supply at least three copies of a report showing non-proprietary manufacturing methods, processing conditions, and test procedures and results for each lot of material in the shipment. The report shall also include the number of the specification and the purchase order or contract number.

4. MAXIMUM ALLOWABLE DISCONTINUITIES

4.1. General. The finished product shall be visibly free from oxide or scale of any nature, grease, oil, residual lubricants, and other extraneous materials. Cracks, laps, seams, gouges, and fins shall be unacceptable.

4.2. Porosity and Inclusions. Indications of internal porosity and non-metallic inclusions greater than 0.020 inch or 3% of the thickness, whichever is smaller, shall be unacceptable. Those indications in the range 0.010 inch to 0.020 inch or 2% of the thickness or diameter, whichever is smaller, shall be a minimum of 0.500 inch apart; those indications less than 0.010 inch shall be a minimum of 0.12 inch apart.

TABLE II

PERMISSIBLE DIMENSIONAL VARIATIONS FOR ROUND BAR

<u>Diameter Inches</u>	<u>Diameter Variation Inch</u>	<u>Out-of-Roundness Inch</u>
0.125 to 0.281	+ 0.002, -0.002	0.004
Over 0.281 to 0.406	+ 0.010, -0.005	0.008
Over 0.406 to 0.625	+ 0.010, -0.005	0.012
Over 0.625 to 0.875	+ 0.015, -0.005	0.015
Over 0.875 to 1.000	+ 0.020, -0.005	0.015
Over 1.000 to 1.375	+ 0.020, -0.010	0.018
Over 1.375 to 1.500	+ 0.020, -0.015	0.020
Over 1.500 to 1.625	+ 0.025, -0.015	0.020
Over 1.625 to 2.000	+ 0.030, -0.020	0.025
Over 2.000 to 2.500	+ 0.032, -0.032	0.025
Over 2.500 to 3.250	+ 0.032, -0.032	0.027
Over 3.250 to 3.500	+ 0.045, -0.045	0.040

Centerless Ground Rounds

0.0625 to 2.0	+ 0.002, -0.002
Over 2.0	+ 0.003, -0.002

4.3. Surface Rework. All surface pores, gouges, and other defects deeper than 0.005 inch or 3% of the thickness, whichever is smaller, shall be unacceptable. Surface imperfections may be faired smooth to remove any notch effect provided dimensional tolerances are still maintained.

5. QUALITY ASSURANCE PROVISIONS

5.1. Vendor Responsibility. The manufacturer shall make all tests and inspections of the material covered by this specification before shipment, unless otherwise specified. All test and inspection results shall be furnished to the purchaser.

5.2. Customer Review. The purchaser or his representative may witness the testing and inspection of the material. The manufacturer shall give the purchaser ample notice of the time and place of designated tests. If the purchaser's representative is not present at this time and a new date is not set, the requirement for purchaser's inspection at the place of testing is waived. When the purchaser's representative is present at the appointed time and place, the manufacturer shall afford him, without charge, all reasonable facilities to assure that the material is being furnished in accordance with this specification. This inspection shall not interfere unnecessarily with production operations.

5.3. Sample Selection. Care shall be exercised to insure that the samples selected for testing and chemical analyses are representative of the material and uncontaminated by the sampling procedure. Samples for the determination of mechanical properties shall be selected so as to consume a minimum amount of material, i.e., specimens may be taken transverse to the final working direction from bar of sufficient width or from rod greater than 2 inches in diameter. If there is any question about the sampling technique or the analysis, the methods for sampling and analysis shall be those agreed to by the buyer and seller. The location of test samples shall be reported in the certificate of compliance.

5.4. Test Methods

5.4.1. Chemical Analysis. Chemical analyses shall be conducted by mutually acceptable procedures, such as the vacuum fusion methods for gases, the combustion method for carbon, and the spectrochemical methods for metallic elements.

5.4.2. Tensile Test. The tension test shall be conducted in accordance with ASTM Designation E8-57T, "Methods of Tension Testing of Metallic Materials." Yield strength shall be determined by the offset (0.2%) method. The tensile properties shall be determined using a strain rate of 0.005 inch per inch per minute up to 0.6% offset, and then 0.05 inch, plus or minus 0.02 inch, per inch per minute to fracture.

5.4.3. Stress-Rupture Test. Stress-rupture properties of specimens shall be determined by mutually acceptable testing techniques. Suggested testing techniques for determining stress-rupture properties are:

Specimens shall be tested in a vacuum of 1×10^{-6} torr or better. The vacuum system shall incorporate an optically tight liquid nitrogen cold trap or a getter-ion pump.

Specimens shall be held for a half hour at the test temperature before application of load.

Test temperature shall be maintained at plus or minus 10°F during the test.

5.4.4. Grain Size. Grain size determinations shall be made according to ASTM Specification E112-61, "Estimating the Average Grain Size of Metals."

5.5. Number of Tests Required. Representative test specimens from the finished product representing each ingot and each lot of material shall be taken to determine conformity to this specification. The minimum frequency of these tests shall be:

Finished Product Chemistry - one per lot per ingot

Tensile Test - two per lot per ingot

Stress-Rupture Test - two per lot per ingot

Grain Size - two per lot per ingot

Microhardness Traverse - one per lot per ingot

5.6. Retest and Rework

5.6.1. Surface Contamination. Any sample or specimen exhibiting obvious surface contamination or improper preparation which disqualifies it as a truly representative sample shall be replaced with a new sample.

5.6.2. Rework. If inspection and test results of a lot do not conform to the requirements of this specification, the lot may be reworked at the option of the manufacturer. The lot shall be acceptable if all test results, after reworking, conform to this specification.

5.7. Inspection

5.7.1. Methods of Inspection

5.7.1.1. Radiographic. Whenever specified, the product shall be radiographed and found free of porosity and inclusions as specified in paragraph 4.2 using the technique described in AMS 2635, "Radiographic Inspection." The radiographs and product shall be identified so the exact position of each radiograph can be correlated with the specific area on the particular product.

5.7.1.2. Ultrasonic. All material 0.125-inch diameter and larger shall be inspected ultrasonically.

5.7.1.2.1. Method and Equipment. The finished products shall be ultrasonically inspected by the immersed technique at 5 mc or above. Transducers shall be no larger than 0.75-inch diameter. Surface finishes shall be no rougher than 125 rms. Inspection shall be by longitudinal wave and by shear wave in two perpendicular directions, i.e., longitudinal and transverse and shall be with focused transducers appropriate to the diameter being inspected (360 degree transducers are allowable where appropriate). Automatic equipment which traverses a spiral path is satisfactory; but three traverses shall be made, one with the transducer in the circumferential shear position, one with the transducer in the axial shear position, and one with the transducer in the longitudinal wave position, unless otherwise specified.

5.7.1.2.2. Calibration of Bar and Rod. Calibration shall be on notches and holes in a segment of the material reserved solely for calibration purposes or in a calibration specimen of similar nature and shape. The depth of the notches shall be 3% of the bar thickness, 1.5% of the rod diameter, or 0.005 inch, whichever is smaller; the width, no greater

than depth; the length, greater than beam width. The notches shall be placed perpendicular to the direction of the shear wave beam and perpendicular to the surface, e.g., axial and circumferential notches on bar. In addition to the notches, a 0.020-inch diameter hole shall be made at least 0.5-inch deep in the calibration piece parallel to the surface at a distance from the surface of $1/2$ the thickness or diameter or, if the thickness exceeds 0.750 inch, $1/4$, $1/2$ and $3/4$ of thickness. Calibration settings to achieve 80% amplitude of these notches or holes along with the magnitude of the other applicable calibration defects shall be recorded. For example, on bar with shear wave, the notch on the near surface should be set at 80% and the amplitudes recorded for the indications from the hole and the notch on the far surface. Gain settings should be recorded to achieve 80% as above and 80% on each of the other applicable calibration defects. For longitudinal wave, only the 0.020-inch diameter holes, with additional holes at $1/4$ and $1/2$ the thickness if the thickness exceeds 0.750 inch, shall be used for calibration.

5.7.1.2.3. Evaluation. Evaluation during inspection shall be made against the appropriate calibration defect. For example, on bar with shear wave, the defects on or near the far surface shall be compared to the calibration from the far surface notch; defects near the center shall be compared to the calibration from the hole at the appropriate depth; defects on the near surface shall be compared to the calibration from the near surface notch.

5.7.1.2.4. Reports. The ultrasonic inspection report shall contain the equipment serial numbers, calibration amplitude and gain settings, and the amplitude and locations of each defect whose amplitude is 60% or greater.

5.7.1.2.5. Rejection. The above procedure shall be followed, and indications of defects which exceed the magnitude obtained from the appropriate calibrated notch in the sample shall be cause for rejection, unless otherwise agreed by the purchaser and vendor.

5.7.1.3. Penetrant Inspection. The exterior surface of the product shall be penetrant inspected and found free of flaws as specified in paragraph 4.3 using AMS 2645, "Fluorescent Penetrant Inspection," or AMS 2646, "Contrast Dye Penetrant Inspection." All parts thus inspected shall be marked with ink stamps as described in the specification; impression stampings or etching shall be unacceptable.

5.7.1.4. Reports. The manufacturer shall supply at least three copies of a report showing inspection results for each lot of material in the shipment. The report shall also include the number of the specification and the purchase order or contract number.

5.8. Rejection. Material not conforming to this specification or to any authorized modification shall be subject to rejection. Unless otherwise specified, rejected material may be returned to the manufacturer at the manufacturer's expense if the purchaser does not receive other instructions for disposition within three weeks after notice of rejection.

5.9. Referee. If the manufacturer and the purchaser disagree about the conformance of the material to the requirements of this specification or any special test specified by the purchaser, a mutually acceptable referee's test shall be used to determine conformance.

6. PREPARATION FOR DELIVERY

6.1. Identification. Each bundle, box, or carton shall be legibly and conspicuously marked or tagged with the number of this specification, purchase order or contract number, type, ingot number, lot number, nominal size, and the gross, net, and tare weights. When each bundle, box or carton consists of components from more than one ingot number or lot number, each component shall be identified individually.

6.2. Packing. Each individual item shall be wrapped in a heavy gauge polyethylene film or similar material and packed in a manner assuring safe delivery when properly transported by any common carrier.

7. DEFINITIONS

7.1. Lot. A lot shall include all material of the same size, shape, condition and finish from one heat of material and which has received the same processing, has been annealed in the same vacuum annealing charge and has been processed simultaneously in all operations in which temperatures may reach 500°F or above. When process temperatures and environments are closely controlled or when closely adjacent sizes receive similar processing, lots may be combined for chemical, tensile and stress-rupture tests only, provided prior written approval has been obtained from the General Electric Company.

7.2. Check Analysis. An analysis may be requested by the purchaser of the metal after it has been processed into finished mill forms, to verify the composition within a heat or lot. Check analysis tolerances do not broaden the specified heat analysis requirements but rather cover variations between laboratories in the measurement of the chemical content.

7.3. Significance of Numerical Limits. For determining compliance with the specified limits for requirements of the properties listed below, an observed value or a calculated value shall be rounded off using the rounding-off method in ASTM Designation E29-58T, "Recommended Practices for Designating Significant Places in Specified Limiting Values."

<u>Test</u>	<u>Rounded-Off Unit for Observed or Calculated Value</u>
Chemical composition and dimensional tolerances (when expressed decimally)	Nearest unit in the last right-hand place of figures of the specified limit
Tensile strength	Nearest 100 psi
Elongation	Nearest 1%
Rupture life	Nearest 0.1 hour

01-0040-00-B
SPPS-59-R1
23 December 1964
Page 1 of 16

SPECIFICATION

SHEET, PLATE, AND STRIP: T-111 (Ta-8W-2Hf) ALLOY

prepared for
NATIONAL AERONAUTICS AND SPACE ADMINISTRATION
Contract NAS 3-2547

SPACE POWER AND PROPULSION SECTION
MISSILE AND SPACE DIVISION
GENERAL ELECTRIC COMPANY
CINCINNATI, OHIO 45215

SPECIFICATION

SHEET, PLATE, AND STRIP: T-111 (Ta-8W-2Hf) ALLOY

1. SCOPE

1.1. Scope. This specification covers T-111 (Ta-8W-2Hf) alloy in sheet, plate and strip form intended for high temperature structural applications and alkali metal containment.

2. APPLICABLE DOCUMENTS

2.1. Government Documents. None

2.2. Non-Government Documents

ASTM Designation E8-57T
(26 December 1957)

Method of Tension Testing of
Metallic Materials

ASTM Designation E29-58T
(1958)

Recommended Practices for
Designating Significant Places
in Specified Limiting Values

ASTM Designation E112-61
(1961)

Estimating the Average Grain
Size of Metals

AMS 2242A
(1 December 1950)

Tolerances, Corrosion and
Heat Resistant Sheet, Strip
and Plate

AMS 2635
(15 August 1958)

Radiographic Inspection

AMS 2645
(1 March 1955)

Fluorescent Penetrant Inspec-
tion

AMS 2646
(1 March 1955)

Contrast Dye Penetrant Inspec-
tion

MAB-176-M
(6 September 1961)

Evaluation Test Methods for
Refractory Metal Sheet Mate-
rials

3. REQUIREMENTS

3.1. Acknowledgments. The vendor shall mention this specification in all quotations and all purchase order acknowledgments.

3.2. Manufacture. Material covered by this specification shall be made from ingots which have been double vacuum melted by the electron beam and/or consumable electrode arc melting processes. Breakdown operations shall be performed with conventional extrusion, forging and rolling equipment normally found in primary ferrous and nonferrous plants.

3.3. Processing. The starting stock size, processing temperatures, percentages of reduction, in-process annealing temperatures and times shall be selected by the vendor to achieve the grain size range specified in paragraph 3.6 and mechanical properties specified in paragraph 3.7. The amount of total reduction from the turned ingot to the final product shall exceed 75%. The amount of final reduction for each mill product, imparted just prior to the final vacuum heat treatment and the total reduction since the previous recrystallization anneal, shall be reported in the certificate of compliance.

3.4. Condition

3.4.1. General. The finished product shall be supplied in the recrystallized condition throughout the cross-sectional area to the grain size range specified in paragraph 3.6.

3.4.2. Heat Treatment. All annealing shall be carried out in a vacuum of less than 1×10^{-5} torr. All mill products to be annealed shall be thoroughly degreased, chemically cleaned and protected from furnace parts by a layer of fresh tantalum, columbium or Cb-1Zr foil 0.002-inch thick or greater. When annealing is carried out in a vacuum greater than 1×10^{-5} torr, with the prior approval of the purchaser, all mill products shall be enclosed in a chemically cleaned tantalum, columbium or Cb-1Zr alloy retort or wrapped in a minimum of two layers of fresh tantalum, columbium or Cb-1Zr alloy foil 0.002-inch thick or greater. The conditions of final annealing shall be reported in the certificate of compliance.

3.4.3. Contamination. All items are to be free of contamination or internal oxidation. After final heat treatment, the material shall be examined metallographically for evidence of possible contamination caused by unsatisfactory heat treating atmospheres or processing

conditions. A microhardness traverse shall show a hardness increase not greater than 50 VHN from the center to the surface of a cross sectional sample of the final product. At the discretion of the purchaser, samples taken to include at least one surface of the final product, and not exceeding 0.050-inch thick, may be chemically analyzed by the purchaser for oxygen, nitrogen, hydrogen and carbon. The analyses shall not exceed the limits set forth in paragraph 3.5.3. Any indication of contamination shall be cause for rejection of all material represented by that sample. The material shall be acceptable if the contaminated layer is completely eliminated before shipment by a machining operation within the specified dimensions and tolerances.

3.5. Chemical Composition

3.5.1. Ingot/Billet Composition. The chemical composition of ingots and billets for conversion to finished products shall conform to Table I (page 5). A minimum of four analyses shall be obtained as follows: ingot top-center, mid-radius and edge, and ingot bottom-center; all analyses must conform to ranges stated in Table I.

3.5.2. Final Product Composition. The manufacturer's ingot analyses shall be considered the chemical analysis for products supplied under this specification (Table I) except carbon, oxygen, nitrogen and hydrogen content which shall be determined in the finished product.

3.5.3. Check Analysis. Finished product analysis shall not exceed the following limits or variations:

<u>Element</u>	<u>Check Analysis Limits, Max., ppm</u>	<u>Permissible Variations in Check Analysis, ppm</u>
Carbon	50	+ 10
Oxygen	150	+ 20
Nitrogen	75	+ 10
Hydrogen	10	+ 2

3.6. Grain Size. The grain size of the final products shall conform to the following limits:

TABLE I
CHEMICAL COMPOSITION
T-111 (Ta-8W-2Hf) ALLOY

<u>Element</u>	<u>Minimum Content</u> <u>ppm</u>	<u>Maximum Content</u> <u>ppm</u>
Carbon	-	50
Nitrogen	-	50
Oxygen	-	100
Hydrogen	-	10
Columbium	-	1000
Molybdenum	-	200
Nickel	-	50
Cobalt	-	50
Iron	-	50
Vanadium	-	20
Tungsten	7.0 w/o	9.0 w/o
Hafnium	1.8 w/o	2.4 w/o
Tantalum	Remainder	-

<u>Product Thickness, Inches</u>	<u>Minimum Allowable ASTM Grain Size No.</u>	<u>Allowable Spread in ASTM Grain Size Nos. in Any One Item</u>	<u>% R_x Minimum</u>
0.010 to 0.060	6	2	100
0.060 to 0.125	4	2	100
0.125 to 0.187	4	2	100
0.187 to 0.500	3	3	95
0.500 to 1.0	3	3	95
Greater than 1.0	3	3	90

3.7. Mechanical Properties. The final product shall satisfy the following mechanical property requirements:

3.7.1. Room Temperature Tensile Properties. Representative samples of the material in final form shall be capable of the following property limits at room temperature (65°-85°F).

<u>Ultimate Tensile Strength, ksi</u>		<u>0.2% Yield Strength, ksi</u>		<u>Elong., % in 2 Inches</u>
<u>Minimum</u>	<u>Maximum</u>	<u>Minimum</u>	<u>Maximum</u>	<u>Minimum</u>
80	110	65	100	20

3.7.2. Stress-to-Rupture Tests. The material shall be capable of achieving the following stress-rupture life under suitable environmental conditions (see paragraph 5.4.3).

<u>Test Temp., °F</u>	<u>Stress, ksi</u>	<u>Minimum Life Hours</u>
2400	19	20

Chemical analysis of stress-rupture specimens after test shall demonstrate that the degree of environmental contamination did not exceed the following limits: total increase in oxygen plus nitrogen content--less than 50 ppm; increase in hydrogen content--less than 5 ppm; increase in carbon content--less than 10 ppm. The following limits shall apply to check analyses of the analytical results:

Carbon	$\pm$	10 ppm
Oxygen	$\pm$	25 ppm
Nitrogen	$\pm$	25 ppm
Hydrogen	$\pm$	2 ppm

3.7.3. Bend Ductility. Representative samples of the materials in final form shall withstand the following bend test at room temperature without failure when tested according to procedures described in the most recent revision of the Materials Advisory Board report MAB-176-M, "Evaluation Test Methods for Refractory Metal Sheet Materials." The samples shall be sectioned with the long axis of the bend specimens perpendicular to the final rolling direction.

3.7.3.1. Sheet 0.060 inch in thickness and under shall be bent over a 1T radius through 105° at a ram speed of 1 inch per minute and subsequently flattened for a total bend of 180°.

3.7.3.2. Sheet over 0.060 inch to 0.187 inch in thickness shall be bent over a 1T radius through 105° at a ram speed of 1 inch per minute.

3.8. Tolerances

3.8.1. Plate

3.8.1.1. Definition. Plate includes material 6 inches wide or over and 0.187 inch or more in thickness.

3.8.1.2. Dimensions. Plate dimensions shall conform to the following tolerances:

<u>Thickness</u>	<u>Width</u>	<u>Length</u>
$\pm$ 0.025 inch or $\pm$ 5% whichever is less	$\pm$ 0.125 inch	$\pm$ 0.125 inch

3.8.1.3. Flatness. Flatness tolerance on plate shall conform to AMS 2242A, "Tolerances, Corrosion and Heat Resistant Sheet, Strip and Plate."

3.8.2. Sheet

3.8.2.1. Definition. Sheet includes material 6 inches wide or over and up to 0.187 inch in thickness.

3.8.2.2. Dimensions. Sheet dimensions shall conform to those presented in Table II.

3.8.2.3. Flatness. See paragraph 3.8.3.3.

3.8.3. Strip

3.8.3.1. Definition. Strip includes material 6 inches wide or less and up to 0.187 inch in thickness.

3.8.3.2. Dimensions. Strip dimensions shall conform to those presented in Table II.

3.8.3.3. Flatness. Total deviation from flatness of sheet and strip shall not exceed 6% as determined by the formula:

$$\frac{H}{L} \times 100 = \% \text{ Flatness Deviation}$$

where

H = maximum distance from a flat reference surface

and

L = minimum distance from this point to the point of contact with the reference surface.

The actual values shall be reported. In determining flatness, the sheet shall not be subject to external pressure at any point but shall lie freely on a flat surface during measurement. Oilcanning will be reported. An estimate of the extent (area, height, etc.,) of these defects shall be made.

3.9. Reports. The manufacturer shall supply at least three copies of a report showing non-proprietary manufacturing methods, processing conditions and test procedures and results for each lot of material in the shipment. The report shall also include the number of the specification and the purchase order or contract number.

TABLE II

DIMENSIONAL TOLERANCES FOR SHEET AND STRIP

<u>Material Thickness, Inch</u>	<u>Width, Inches</u>	<u>Thickness Tolerances, Inch</u>
0.010-0.019	to 24	± 0.001
0.020-0.039	to 24	± 0.0015
0.040-0.059	to 24	± 0.002
0.060-0.089	to 24	± 0.003
0.090-0.0129	to 24	± 0.004
0.130-0.159	to 24	± 0.005
0.160-0.187	to 24	± 0.010

<u>Material Thickness, Inch</u>	<u>Width Tolerances, Inch</u>
0.010-0.059	+ 0.031, -0
0.060-0.125	+ 0.046, -0
0.126-0.187	+ 0.125, -0

<u>Material Thickness, Inch</u>	<u>Length Tolerances, Inch</u>
0.010-0.059	+ 0.046, -0
0.060-0.125	+ 0.062, -0
0.126-0.187	+ 0.125, -0

4. MAXIMUM ALLOWABLE DISCONTINUITIES

4.1. General. The finished product shall be visibly free from oxide or scale of any nature, grease, oil residual lubricants, and other extraneous materials. Cracks, laps, seams, gouges, and fins shall be unacceptable.

4.2. Porosity and Inclusions. Indications of internal porosity and non-metallic inclusions greater than 0.020 inch or 3% of the thickness, whichever is smaller, shall be unacceptable. Those indications in the range 0.010 inch to 0.020 inch or 2% of the thickness, whichever is smaller, shall be a minimum of 0.500 inch apart; those indications less than 0.010 inch shall be a minimum of 0.12 inch apart.

4.3. Surface Rework. All surface pores, gouges, and other defects deeper than 0.005 inch or 3% of the thickness, whichever is smaller, shall be unacceptable. Surface imperfections may be faired smooth to remove any notch effect provided dimensional tolerances are still maintained.

5. QUALITY ASSURANCE PROVISIONS

5.1. Vendor Responsibility. The manufacturer shall make all tests and inspections of the material covered by this specification before shipment, unless otherwise specified. All test and inspection results shall be furnished to the purchaser.

5.2. Customer Review. The purchaser or his representative may witness the testing and inspection of the material. The manufacturer shall give the purchaser ample notice of the time and place of designated tests. If the purchaser's representative is not present at this time and a new date is not set, the requirement for purchaser's inspection at the place of testing is waived. When the purchaser's representative is present at the appointed time and place, the manufacturer shall afford him, without charge, all reasonable facilities to assure that the material is being furnished in accordance with this specification. This inspection shall not interfere unnecessarily with production operations.

5.3. Sample Selection. Care shall be exercised to insure that the samples selected for testing and chemical analyses are representative of the material and uncontaminated by the sampling procedure. Samples for the determination of mechanical properties shall be selected so as to consume a minimum amount of material, i.e., specimens may be taken

transverse to the final working direction from plate and sheet and from strip if of sufficient width. If there is any question about the sampling technique or the analysis, the methods for sampling and analysis shall be those agreed to by the buyer and seller. The location of test samples shall be reported in the certificate of compliance.

5.4. Test Methods

5.4.1. Chemical Analysis. Chemical analyses shall be conducted by mutually acceptable procedures, such as the vacuum fusion methods for gases, the combustion method for carbon and the spectrochemical methods for metallic elements.

5.4.2. Tensile Test. The tension test shall be conducted in accordance with ASTM Designation E8-57T, "Methods of Tension Testing of Metallic Materials." Yield strength shall be determined by the offset (0.2%) method. The tensile properties shall be determined using a strain rate of 0.005 inch per inch per minute up to 0.6% offset and then 0.050 inch, plus or minus 0.020 inch, per inch per minute to fracture.

5.4.3. Stress-Rupture Test. Stress-rupture properties of specimens shall be determined by mutually acceptable testing techniques. Suggested testing techniques for determining stress-rupture properties are:

Specimens shall be tested in a vacuum of 1×10^{-6} torr or better. The vacuum system shall incorporate an optically tight liquid nitrogen cold trap or a getter-ion pump.

Specimens shall be held for a half hour at the test temperature before application of load.

Test temperature shall be maintained at plus or minus 10°F during the test.

5.4.4. Grain Size. Grain size determinations shall be made according to ASTM Specification E112-61, "Estimating the Average Grain Size of Metals."

5.5. Number of Tests Required. Representative test specimens from the finished product representing each ingot and each lot of material shall be taken to determine conformity to this specification. The minimum frequency of these tests shall be:

Finished Product Chemistry - one per lot per ingot

Tensile Test - two per lot per ingot

Stress-Rupture Test - two per lot per ingot

Bend Test - two per lot per ingot

Grain Size - two per lot per ingot

Microhardness Traverse - one per lot per ingot

5.6. Retest and Rework

5.6.1. Surface Contamination. Any sample or specimen exhibiting obvious surface contamination or improper preparation which disqualifies it as a truly representative sample shall be replaced with a new sample.

5.6.2. Rework. If inspection and test results of a lot do not conform to the requirements of this specification, the lot may be reworked at the option of the manufacturer. The lot shall be acceptable if all test results, after reworking, conform to this specification.

5.7. Inspection

5.7.1. Methods of Inspection

5.7.1.1. Radiographic. Whenever specified, the product shall be radiographed and found free of porosity and inclusions as specified in paragraph 4.2 using the technique described in AMS 2635, "Radiographic Inspection." The radiographs and product shall be identified so the exact position of each radiograph can be correlated with the specific area on the particular product.

5.7.1.2. Ultrasonic. Unless otherwise agreed to by the purchaser and the vendor, the material shall be inspected ultrasonically.

5.7.1.2.1. Method and Equipment. The finished products shall be ultrasonically inspected by the immersed technique at 5 mc or above. Transducers shall be no larger than 0.75-inch diameter. Surface finishes shall be no rougher than 125 rms. Inspection shall be by

longitudinal wave and by shear wave in two perpendicular directions, i.e., longitudinal and transverse shear.

Transducers for the shear wave inspection shall be focused, preferably cylindrically, to a beam no more than 0.125-inch wide in its smaller dimension (where it enters the material being inspected). Cylindrically-focused transducers shall not exceed 2 inches in length. The focal distance shall be adjusted when the transducer is beamed perpendicular to the surface of the calibration piece; then this focal distance shall be maintained throughout the actual inspection. After the focal distance is established, an appropriate shear wave angle shall be set and the calibration notch indication shall be set at 80% on the indication where the sound beam traverses one or two thicknesses of the sheet (depending on whether the notch is on the far side or incident side of the sheet). Calibration gain settings shall be recorded when the calibration defect is on both the incident and the far side of the sheet. If there is any difference in the indication, that gain setting giving an 80% indication from the side which produces the smaller indication shall be used for inspection. Calibration shall be done before and after the ultrasonic inspection or at the beginning and end of each work shift. If the magnitude of indication from the calibration notch differs 10% or more from the previous calibration, all material inspected since then shall be reinspected.

5.7.1.2.2. Calibration of Plate. Calibration shall be on notches and holes in a segment of the material reserved solely for calibration purposes. The depth of the notches shall be 0.005 inch, the width shall be 0.005 inch and the length greater than the ultrasonic beam width. The notches shall be placed on the surface of the calibration piece perpendicular to the direction of the intended shear wave inspection, i.e., transverse and longitudinal and at least 1 inch from the edge of the plate. In addition, a 0.020-inch diameter hole shall be made in the calibration piece parallel to the surface to a depth of at least 0.750 inch at a point one-half the thickness of the plate. If the thickness of the plate exceeds 0.750 inch, similar holes shall also be made at points one-quarter and three-quarters of the plate thickness. Calibration settings to achieve 80% amplitude of the notches and holes, along with the magnitude of the other applicable calibration defects, shall be recorded. For example, on plate using a shear wave, the notch on the near surface should be set at 80% and the amplitude recorded for the indications from the hole and notch on the far surface. Gain settings should be recorded to achieve 80% as above and 80% on each of the other applicable calibration defects. For longitudinal wave inspection only the 0.020-inch diameter holes shall be used for calibration

5.7.1.2.3. Calibration of Sheet and Strip. The sheet shall be inspected by a shear wave beam pointed in both longitudinal and transverse directions. Calibration shall be done on notches cut perpendicular to the direction of the beam in pieces of sheet of the same material and thickness as that to be inspected. If that portion is later trimmed and scrapped, the calibration notches may be made on a section of the actual sheet. The depth of the calibration notches shall be 3% of the sheet thickness; width, no greater than the depth; length, no more than 1 inch. All notches shall be at least 1 inch from the edge of the sheet. Duplicate notches may be made on the opposite face of the sheet in locations where the sound beam will not intersect both notches in a single traverse, or the sheet may be turned over during calibration to determine the relative response from the calibration notch on both the incident and far side of the sheet.

5.7.1.2.4. Evaluation. Evaluation during inspection shall be made against the appropriate calibration defect. For example, with shear wave, the defects on or near the far surface shall be compared to the calibration from the far surface notch; defects near the center shall be compared to the calibration from the hole at the appropriate depth; defects on the near surface shall be compared to the calibration from the near surface notch.

5.7.1.2.5. Reports. The ultrasonic inspection report shall contain the equipment serial numbers, calibration amplitudes and gain settings and the amplitude and location of each defect whose amplitude is 60% or greater.

5.7.1.2.6. Rejection. The above procedure shall be followed, and indications of defects which exceed the magnitude obtained from the appropriate calibrated notch in the sample shall be cause for rejection, unless otherwise agreed by the purchaser and vendor.

5.7.1.3. Penetrant Inspection. The exterior surface of the product shall be penetrant inspected and found free of flaws as specified in paragraph 4.3 using AMS 2645, "Fluorescent Penetrant Inspection," or AMS 2646, "Contrast Dye Penetrant Inspection." All parts thus inspected shall be marked with ink stamps as described in the specification; impression stampings or etching shall be unacceptable.

5.7.1.4. Reports. The manufacturer shall supply at least three copies of a report showing inspection results for each lot of material in the shipment. The report shall also include the number of the specification and the purchase order or contract number.

5.8. Rejection. Material not conforming to this specification or to any authorized modification shall be subject to rejection. Unless otherwise specified, rejected material may be returned to the manufacturer at the manufacturer's expense if the purchaser does not receive other instructions for disposition within three weeks after notice of rejection.

5.9. Referee. If the manufacturer and the purchaser disagree about the conformance of the material to the requirements of this specification or any special test specified by the purchaser, a mutually acceptable referee's test shall be used to determine conformance.

6. PREPARATION FOR DELIVERY

6.1. Identification. Each bundle, box, or carton shall be legibly and conspicuously marked or tagged with the number of this specification, purchase order or contract number, type, ingot number, lot number, nominal size, and the gross, net, and tare weights. When each bundle, box or carton consists of components from more than one ingot number or lot number, each component shall be identified individually.

6.2. Packing. Each individual item shall be wrapped in heavy gauge polyethylene film or other similar material and packed in a manner assuring safe delivery when properly transported by any common carrier.

7. DEFINITIONS

7.1. Lot. A lot shall include all material of the same size, shape, condition and finish from one heat of material and which has received the same processing, has been annealed in the same vacuum annealing charge and has been processed simultaneously in all operations in which temperatures may reach 500°F or above. When process temperatures and environments are closely controlled or when closely adjacent sizes receive similar processing, lots may be combined for chemical, tensile and stress-rupture tests only, provided prior written approval has been obtained from the General Electric Company.

7.2. Check Analysis. An analysis may be made or requested by the purchaser of the metal after it has been processed into finished mill forms, to verify the composition within a heat or lot. Check analysis tolerances do not broaden the specified heat analysis requirements but rather cover variations between laboratories in the measurement of the chemical content.

7.3. Significance of Numerical Limits. For determining compliance with the specified limits for requirements of the properties listed below, an observed value or a calculated value shall be rounded off using the rounding-off method in ASTM Designation E29-58T, "Recommended Practices for Designating Significant Places in Specified Limiting Values."

<u>Test</u>	<u>Rounded-Off Unit for Observed or Calculated Value</u>
Chemical composition and dimensional tolerances (when expressed decimally)	Nearest unit in the last right-hand place of figures of the specified limit
Tensile strength	Nearest 100 psi
Elongation	Nearest 1%
Rupture Life	Nearest 0.1 hour

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SPPS-54-R1
23 December 1964
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SPECIFICATION

SEAMLESS TUBING AND PIPE: T-111 (Ta-8W-2Hf) ALLOY

prepared for
NATIONAL AERONAUTICS AND SPACE ADMINISTRATION
Contract NAS 3-2547

SPACE POWER AND PROPULSION SECTION
MISSILE AND SPACE DIVISION
GENERAL ELECTRIC COMPANY
CINCINNATI, OHIO 45215

SPECIFICATION

SEAMLESS TUBING AND PIPE: T-111 (Ta-8W-2Hf) ALLOY

1. SCOPE

1.1. Scope. This specification covers T-111 (Ta-8W-2Hf) alloy in tube and pipe form intended for high temperature structural application and alkali metal containment.

2. APPLICABLE DOCUMENTS

2.1. Government Documents. None

2.2. Non-Government Documents

ASTM Designation E8-57T
(26 December 1957)

Method of Tension Testing of
Metallic Materials

ASTM Designation E29-58T
(1958)

Recommended Practices for
Designating Significant Places
in Specified Limiting Values

ASTM Designation E112-61
(1961)

Estimating Average Grain Size
of Metals

AMS 2635
(15 August 1958)

Radiographic Inspection

AMS 2645
(1 March 1955)

Fluorescent Penetrant Inspection

AMS 2646
(1 March 1955)

Contrast Dye Penetrant Inspection

3. REQUIREMENTS

3.1. Acknowledgments. The vendor shall mention this specification in all quotations and all purchase order acknowledgments.

3.2. Manufacture. Material covered by this specification shall be made from ingots which have been double vacuum melted by the electron beam and/or consumable electrode arc melting processes. Breakdown operations shall be performed with conventional extrusion, forging, tube reducing and drawing equipment normally found in primary ferrous and nonferrous plants.

3.3. Processing. The starting stock size, processing temperatures, percentages of reduction, in-process annealing temperatures and times shall be selected by the vendor to achieve the grain size range specified in paragraph 3.6 and mechanical properties specified in paragraph 3.7. The total amount of reduction from the turned ingot to the final product shall exceed 75%. The amount of final reduction for each mill product, imparted just prior to the final vacuum heat treatment, and the total reduction since the last recrystallization anneal shall be reported in the certificate of compliance.

3.4. Condition

3.4.1. General. The finished product will be supplied in the recrystallized condition throughout the cross-sectional area to the grain size range specified in paragraph 3.6.

3.4.2. Heat Treatment. All annealing shall be carried out in a vacuum of less than 1×10^{-5} torr. All mill products to be annealed shall be thoroughly degreased, chemically cleaned and protected from furnace parts by a layer of fresh tantalum, columbium or Cb-1Zr alloy foil 0.002-inch thick or greater. When annealing is carried out in a vacuum greater than 1×10^{-5} torr, with the prior approval of the purchaser, all mill products shall be enclosed in a chemically cleaned tantalum, columbium or Cb-1Zr alloy retort or wrapped in a minimum of two layers of fresh tantalum, columbium or Cb-1Zr alloy foil 0.002-inch thick or greater. The conditions of final annealing shall be reported in the certificate of compliance.

3.4.3. Contamination. All items are to be free of contamination or internal oxidation. After final heat treatment, the material shall be examined metallographically for evidence of possible contamination caused by unsatisfactory heat treating atmospheres or processing conditions. A microhardness traverse shall show a hardness increase not greater than 50 VHN from the center to the surface of a cross sectional sample of the final product. At the discretion of the purchaser, samples taken to include at least one surface of the final product, and not exceeding 0.050-inch thick, may be chemically analyzed by the

purchaser for oxygen, nitrogen, hydrogen and carbon. The analyses shall not exceed the limits set forth in paragraph 3.5.3. Any indication of contamination shall be cause for rejection of all material represented by that sample. The material shall be acceptable if the contaminated layer is completely eliminated before shipment by a machining operation within the specified dimensions and tolerances.

3.5. Chemical Composition

3.5.1. Ingot/Billet Composition. The chemical composition of ingots and billets for conversion to finished products shall conform to Table I (page 5). A minimum of four analyses shall be obtained as follows: ingot top-center, mid-radius and edge, and ingot bottom-center; all analyses must conform to ranges stated in Table I.

3.5.2. Final Product Composition. The manufacturer's ingot analyses shall be considered the chemical analyses for products supplied under this specification (Table I) except carbon, oxygen, nitrogen and hydrogen content which shall be determined on the finished product.

3.5.3. Check Analysis. The finished product analysis shall not exceed the following limits or variations:

For Wall Thicknesses 0.020 Inch or Greater

<u>Element</u>	<u>Check Analysis Limits, Max., ppm</u>	<u>Permissible Variations in Check Analysis, ppm</u>
Carbon	50	+ 10
Oxygen	150	+ 20
Nitrogen	75	+ 10
Hydrogen	10	+ 2

For Wall Thicknesses Less Than 0.020 Inch

<u>Element</u>	<u>Check Analysis Limits, Max., ppm</u>	<u>Permissible Variations in Check Analysis, ppm</u>
Carbon	75	+ 10
Oxygen	300	+ 20
Nitrogen	100	+ 10
Hydrogen	10	+ 2

TABLE I
CHEMICAL COMPOSITION
T-111 (Ta-8W-2Hf) ALLOY

<u>Element</u>	<u>Minimum Content</u> <u>ppm</u>	<u>Maximum Content</u> <u>ppm</u>
Carbon	-	50
Nitrogen	-	50
Oxygen	-	100
Hydrogen	-	10
Columbium	-	1000
Molybdenum	-	200
Nickel	-	50
Cobalt	-	50
Iron	-	50
Vanadium	-	20
Tungsten	7.0 w/o	9.0 w/o
Hafnium	1.8 w/o	2.4 w/o
Tantalum	Remainder	-

3.6. Grain Size. The grain size of the final products shall conform to the following limits:

<u>Product Wall Thickness, Inches</u>	<u>Minimum Allowable ASTM Grain Size No.</u>	<u>Allowable Spread in ASTM Grain Size Nos. in Any One Item</u>	<u>% Rx Minimum</u>
Less than 0.010	6	2	100
0.010 to 0.065	6	2	100
0.065 to 0.0125	5	2	100
0.125 to 0.250	4	2	95
0.250 to 0.500	3	3	90

3.7. Mechanical Properties. The final product shall satisfy the following mechanical property requirements:

3.7.1. Room Temperature Tensile Properties. Representative samples of the material in final form shall be capable of the following property limits at room temperature (65°-85°F).

<u>Ultimate Tensile Strength, ksi</u>		<u>0.2% Yield Strength, ksi</u>		<u>Elong., %⁽¹⁾</u>
<u>Minimum</u>	<u>Maximum</u>	<u>Minimum</u>	<u>Maximum</u>	<u>Minimum</u>
80	110	65	100	20

(1) % Elongation in 4D for Threaded or Button-Head Test Specimens; in 2 Inches for Flat Specimens.

3.7.2. Stress-to-Rupture Tests. The material shall be capable of achieving the following stress-rupture life under suitable environmental conditions (see paragraph 5.4.3):

<u>Test Temp., °F</u>	<u>Stress, ksi</u>	<u>Minimum Life Hours</u>
2400	19	20

Chemical analyses of stress-rupture specimens after test shall demonstrate that the degree of environmental contamination did not exceed the following limits: total increase in oxygen plus nitrogen content--less than 50 ppm; increase in hydrogen content--less than 5 ppm; increase in carbon content--less than 10 ppm. The following limits shall apply to check analyses of the analytical results:

Carbon	$\pm$	10 ppm
Oxygen	$\pm$	25 ppm
Nitrogen	$\pm$	25 ppm
Hydrogen	$\pm$	2 ppm

3.7.3. Hydrostatic Test. Each tube, 1/8 inch or larger in outside diameter with a wall thickness of 0.015 inch or over, shall be tested to a hydrostatic pressure sufficient to produce a fiber stress of 12,000 psi. The test pressure, not to exceed 10,000 psi, shall be determined by the equation ($P = 2St/D$), where:

P = hydrostatic test pressure in pounds per square inch;

S = 12,000 psi;

t = average wall thickness of the tube in inches;

D = outside diameter of the tube in inches.

3.7.4. Flare Test. A section of the heat treated tube shall be capable of being flared without cracking. The flare shall be made with a tool having a 60-degree included angle until the specified outside diameter has been increased by 15%.

3.8. Tolerances

3.8.1. Diameter and Wall Thickness. The permissible variations in diameter and wall thickness of tube shall not exceed those prescribed in Table II (refer to page 8).

3.8.2. Length. When tube is ordered cut-to-length, the usable length shall not be less than that specified, but a variation of plus 1/8 inch will be permitted in lengths up to 6 feet. In lengths over 6 feet, a variation of plus 1/4 inch will be permitted, unless otherwise specified.

TABLE II
PERMISSIBLE VARIATIONS IN TUBE DIMENSIONS

<u>Nominal OD</u> <u>Inches</u>	<u>OD</u> <u>Inch</u>	<u>ID</u> <u>Inch</u>	<u>Wall</u> <u>Thickness</u> <u>%</u>
0.187 to but not incl. 0.625	± 0.004	± 0.004	± 10
0.625 to but not incl. 1.000	± 0.005	± 0.005	± 10
1.000 to but not incl. 2.000	± 0.0075	± 0.0075	± 10
2.000 to but not incl. 3.000	± 0.010	± 0.010	± 10
3.000 to but not incl. 4.000	± 0.0125	± 0.0125	± 10

- NOTES: -----
- (1) Tolerances are applicable to only the two dimensions specified on the purchase order, e.g., outside diameter and wall; inside diameter and wall; outside diameter and inside diameter.
 - (2) For tolerances applicable for very small tubes (less than 0.187-inch diameter) or very thin-wall tubes (less than 0.010-inch thick), the producer shall be consulted.
 - (3) For tubes having an inside diameter less than 60% of the outside diameter or a wall 3/4 inch or over thick, which cannot be successfully drawn over a mandrel, the inside diameter may vary by an amount equal to plus or minus 10% of the wall thickness. The wall thickness of these tubes may vary plus or minus 12.5% from that specified.
 - (4) Ovality measured at any cross section: For tubes with nominal wall thickness less than 3% of the nominal outside diameter, the ovality tolerances are double the tolerances in column 2 or 3. For ovality tolerances for tubes with wall thickness less than 2% nominal outside diameter, the producer shall be consulted.

3.8.3. Straightness. The tube shall be free of bends or kinks. For lengths up to 10 feet, the maximum bow shall not exceed one part in 1200; for lengths greater than 10 feet, the maximum bow shall not exceed one part in 600; unless otherwise agreed upon.

3.9. Reports. The manufacturer shall supply at least three copies of a report showing non-proprietary manufacturing methods, processing conditions, and test procedures and results for each lot of material in the shipment. The report shall also include the number of the specification and the purchase order or contract number.

4. MAXIMUM ALLOWABLE DISCONTINUITIES

4.1. General. Cracks, laps, seams, fins, and tears shall be unacceptable. The surface shall also be free from oxide or scale of any nature, grease, oil, residual lubricants, or other extraneous material.

4.2. Porosity and Inclusions. Indications with dimensions greater than 3% of the wall thickness shall be unacceptable. Indications with dimensions in the ranges of 1% to 3% of wall thickness must be a minimum of 0.50-inch apart. Indications with dimensions less than 1% of the wall thickness must be a minimum of 0.12 inch apart.

4.3. Surface Rework. Defects less than 3% of the nominal wall thickness detected by penetrant or ultrasonic inspection may be removed by grinding provided the wall thickness is not decreased below that permitted in Table II (refer to page 9).

5. QUALITY ASSURANCE PROVISIONS

5.1. Vendor Responsibility. The manufacturer shall make all test and inspections of the material covered by this specification before shipment unless otherwise specified. All test and inspection results shall be furnished to the purchaser.

5.2. Customer Review. The purchaser or his representative may witness the testing and inspection of the material. The manufacturer shall give the purchaser ample notice of the time and place of designated tests. If the purchaser's representative is not present at this time and a new date is not set, the requirement of purchaser's inspection at the place of testing is waived. When the purchaser's representative is present at the appointed time and place, the manufacturer shall afford him, without charge, all reasonable facilities to assure that the material is being furnished in accordance with this specification. This inspection shall not interfere unnecessarily with production operation.

5.3. Sample Selection. Care shall be exercised to insure that the sample selected for testing is representative of the material and uncontaminated by the sampling procedure. If there is any question about the sampling technique or the analysis, the methods for sampling and analysis shall be those agreed to by the buyer and seller. The specimen configuration selected for the performance of the testing required in paragraph 5.4.2. and 5.4.3 shall be mutually agreed upon by the vendor and purchaser prior to placement of a purchase order. The location of all test samples shall be reported in the certificate of compliance.

5.4. Test Methods

5.4.1. Chemical Analysis. Chemical analyses shall be conducted by mutually acceptable procedures, such as the vacuum fusion methods for gases, the combustion method for carbon, and the spectrochemical methods for metallic elements.

5.4.2. Tensile Test. The tension test shall be performed in accordance with ASTM Designation E8-57T, "Methods of Tension Testing of Metallic Materials." Yield strength shall be determined by the offset (0.2%) method. The tensile properties shall be determined using a strain rate of 0.005 inch per inch per minute up to 0.6% offset and then 0.050 inch, plus or minus 0.020 inch, per inch per minute to fracture.

5.4.3. Stress-Rupture Test. Stress-rupture properties of specimens shall be determined by mutually acceptable testing techniques. Suggested testing technique for determining stress-rupture properties are:

Specimens shall be tested in a vacuum of 1×10^{-6} torr or better. The vacuum system shall incorporate an optically tight liquid nitrogen cold trap or a getter-ion pump.

Specimens shall be held for a half hour at the test temperature before application of load.

Test temperature shall be maintained at plus or minus 10°F during the test.

5.4.4. Grain Size. Grain size determinations shall be made according to ASTM Specification E112-61, "Estimating the Average Grain Size of Metals."

5.5. Number of Tests Required. Representative test specimens from the finished product representing each ingot and each lot of material shall be taken to determine conformity to this specification. The minimum frequency of these tests shall be:

Finished Product Chemistry - one per lot per ingot

Tensile Test - two per lot per ingot

Stress-Rupture Test - two per lot per ingot

Flare Test - two per lot per ingot

Grain Size - two per lot per ingot

Microhardness Traverse - one per lot per ingot

Hydrostatic Proof Test - 100%

5.6. Retest and Rework

5.6.1. Surface Contamination. Any sample or specimen exhibiting obvious surface contamination or improper preparation which disqualifies it as a truly representative sample shall be replaced with a new sample.

5.6.2. Rework. If inspection and test results of a lot do not conform to the requirements of this specification, the lot may be reworked at the option of the manufacturer. The lot shall be acceptable if all test results, after reworking, conform to this specification.

5.7. Inspection

5.7.1. Methods of Inspection

5.7.1.1. Radiographic. When specified, the product shall be radiographed and found free of porosity and inclusions as specified in paragraph 4.2 using the techniques described in AMS 2635, "Radiographic Inspection." The radiographs and product shall be identified so that the exact position of each radiograph can be correlated with the specific area on a particular product.

5.7.1.2. Ultrasonic Inspection. Unless otherwise agreed to by the purchaser and the vendor, the material shall be inspected ultrasonically.

5.7.1.2.1. Method and Equipment. Ultrasonic inspection shall be by the immersed technique at 5 mc or higher frequency using focused transducers. Inspection shall be by both circumferential and axial shear techniques with longitudinal wave being added when the wall thickness is greater than 0.150 inch. For longitudinal wave technique and for circumferential shear, transducers up to 2 inches long may be used with or without automatic equipment to rotate the tube past the transducer. If spiral pattern inspection traverse is not used, steps must be taken to assure that the ultrasonic beam remains in the same position relative to the tubing so the beam-to-tubing angle remains constant. For axial (longitudinal) shear, transducers must have no greater than 0.5 inch axial length. Transducers must be cylindrically focused for a diameter range which includes the tubing on which it is to be used.

5.7.1.2.2. Calibration. Calibration shall be on notches (a total of four, two axial and two circumferential), cut in the tube on both the outside and inside surface unless otherwise specified. The depth of the notches shall be 3% of the wall thickness to a minimum depth of 0.001 inch; the width, no greater than depth; the length, at least that of the ultrasonic beam with a maximum length of 1 inch. Material having a wall thickness greater than 0.150 inch shall also have an 0.020-inch diameter hole machined into the wall in the longitudinal direction at mid-point of the wall thickness. Focusing shall be done to maximize the indication from the inside diameter notch placed properly for the type of inspection contemplated. After focusing is completed, the inside diameter indication shall be set at 80% and gain setting recorded. Gain setting for 80% on the outside diameter notch shall also be recorded. Inspection shall be at the gain setting for the inside diameter indication. A distance corresponding to the wall thickness shall be marked on the oscilloscope. Focal distance to the part to be inspected shall be set to that used for the calibration piece before beginning inspection. Calibration shall be done both before and after the inspection or at the beginning and end of each work shift. If calibration has changed (gain change greater than 5%), all inspections since the previous calibration shall be repeated.

5.7.1.2.3. Rejection. Rejection shall be by any indication which exceeds the amplitude of the respective calibration indication; i.e., inside diameter defects shall be compared to the indication from the notch on the inside diameter, and outside diameter defects shall be compared to the indication from the notch on the outside diameter. Defects less than half the thickness from the surface or less than 0.150 inch from the surface, whichever is smaller, shall be compared

to the outside diameter calibration indication. Defects more than half the thickness from the incident surface or more than 0.150 inch from the surface shall be compared to the indications from the inside diameter calibration notch.

5.7.1.2.4. Reports. The ultrasonic inspection report shall contain the equipment serial numbers, calibration amplitudes and gain settings and the amplitude and location of each defect whose amplitude is 60% or greater.

5.7.1.3. Penetrant Inspection. The exterior surface of the product shall be penetrant inspected and found free of flaws as specified in paragraph 4.3 using AMS 2645, "Fluorescent Penetrant Inspection," or AMS 2646, "Contrast Dye Penetrant Inspection." All parts thus inspected shall be marked with ink stamps as described in the specification; impression stampings or etching shall be unacceptable.

5.7.1.4. Reports. The manufacture shall supply at least three copies of a report showing inspection results for each lot of material in the shipment. The report shall also include the number of the specification and the purchase order or contract number.

5.8. Rejection. Material not conforming to this specification or to any authorized modification shall be subject to rejection. Unless otherwise specified, rejected material may be returned to the manufacturer at the manufacturer's expense if the purchaser does not receive other instructions for disposition within three weeks after notice of rejection.

5.9. Referee. If the manufacturer and the purchaser disagree concerning the conformance of the material to the requirements of this specification or any special test specified by the purchaser, a mutually acceptable referee's test shall be used to determine conformance.

6. PREPARATION FOR DELIVERY

6.1. Identification. Each bundle, box, or carton shall be legibly and conspicuously marked or tagged with the number of this specification, purchase order or contract number, type, ingot number, lot number, nominal size, and the gross, net and tare weights. When each bundle, box or carton consists of components from more than one ingot number or lot number, each component shall be identified individually.

6.2. Packing. The ends of each pipe or tube shall be sealed with suitable plastic caps and each individual item shall be wrapped in heavy gauge polyethylene or similar material and packed in a manner assuring safe delivery when properly transported by a common carrier.

7. DEFINITIONS

7.1. Lot. A lot shall include all material of the same size, shape, condition and finish from one heat of material and which has received the same processing, has been annealed in the same vacuum annealing charge and has been processed simultaneously in all operations in which temperatures may reach 500°F or above. When process temperatures and environments are closely controlled or when closely adjacent sizes receive similar processing, lots may be combined for chemical, tensile and stress-rupture tests only, provided prior written approval has been obtained from the General Electric Company.

7.2. Check Analysis. An analysis may be requested by the purchaser of the metal, after it has been processed into finished mill forms, for the purpose of verifying the composition within in a heat analysis requirements but rather cover variations between laboratories in the measurement of the chemical content.

7.3. Significance of Numerical Limits. For determining compliance with the specified limits for requirements of the properties listed below, an observed value or a calculated value shall be rounded off using the rounding-off method in ASTM Designation E29-58T, "Recommended Practices for Designating Significant Places in Specified Limiting Values."

	<u>Rounded-Off Unit for Observed or Calculated Value</u>
Chemical composition and dimensional tolerances (when expressed decimally)	Nearest unit in the last right- hand place of figures of the specified limit
Tensile strength	Nearest 100 psi
Elongation	Nearest 1%
Rupture life	Nearest 0.1 hour

SPECIFICATION

FOIL: T-111 (Ta-8W-2Hf) ALLOY

prepared for
NATIONAL AERONAUTICS AND SPACE ADMINISTRATION
Contract NAS 3-2547

SPACE POWER AND PROPULSION SECTION
MISSILE AND SPACE DIVISION
GENERAL ELECTRIC COMPANY
CINCINNATI, OHIO 45215

SPECIFICATION

FOIL: T-111 (Ta-8W-2Hf) ALLOY

1. SCOPE

1.1. Scope. This specification covers T-111 (Ta-8W-2Hf) alloy in foil form intended for high temperature non-structural applications.

2. APPLICABLE DOCUMENTS

2.1. Government Documents. None

2.2. Non-Government Documents. None

3. REQUIREMENTS

3.1. Acknowledgments. The vendor shall mention this specification in all quotations and all purchase order acknowledgments.

3.2. Manufacture. Material covered by this specification shall be made from ingots which have been double vacuum melted by the electron beam and/or consumable electrode arc melting processes. Breakdown operations shall be performed with conventional extrusion, forging and rolling equipment normally found in primary ferrous and nonferrous plants.

3.3. Processing. The starting stock size, processing temperatures, percentages of reduction, in-process annealing temperatures and times shall be selected by the vendor.

3.4. Condition. The finished product shall be supplied in the fully recrystallized condition throughout the cross-sectional area. All annealing shall be carried out in a vacuum of less than 1×10^{-5} torr. All mill products to be annealed shall be thoroughly degreased, chemically cleaned and protected from furnace parts by a layer of fresh tantalum, columbium or Cb-1Zr alloy foil 0.002 inch thick or greater. When annealing is carried out in a vacuum greater than 1×10^{-5} torr,

with the prior approval of the purchaser, all mill products shall be enclosed in a chemically cleaned tantalum, columbium or Cb-1Zr alloy retort or wrapped in a minimum of two layers of fresh tantalum, columbium or Cb-1Zr alloy foil 0.002-inch thick or greater. The conditions of final annealing shall be reported in the certificate of compliance.

3.5. Chemical Composition

3.5.1. Ingot/Billet Composition. The chemical composition of ingots and billets for conversion to finished products shall conform to Table I (page 4). A minimum of four analyses shall be obtained as follows: ingot top-center, mid-radius and edge, and ingot bottom-center; all analyses must conform to ranges stated in Table I.

3.5.2. Final Product Composition. The manufacturer's ingot analyses shall be considered the chemical analysis for products supplied under this specification (Table I) except carbon, oxygen, nitrogen and hydrogen content which shall be determined in the finished product.

3.5.3. Check Analysis. Finished product analysis shall be not exceed the following limits or variations:

<u>Element</u>	<u>Check Analysis Limits, Max., ppm</u>	<u>Permissible Variations in Check Analysis, ppm</u>
Carbon	75	+ 10
Oxygen	300	+ 20
Nitrogen	100	+ 10
Hydrogen	10	+ 2

3.6. Bend Ductility. Representative samples of the materials in final form shall withstand a 180° bend without failure.

3.7. Tolerances

3.7.1. Definitions. Foil includes material less than 12 inches wide and up to and including 0.010-inch thick.

3.7.2. Dimensions. Foil dimensions shall conform to the following limits:

TABLE I
CHEMICAL COMPOSITION
T-111 (Ta-8W-2Hf) ALLOY

<u>Element</u>	<u>Minimum Content</u> <u>ppm</u>	<u>Maximum Content</u> <u>ppm</u>
Carbon	-	50
Nitrogen	-	50
Oxygen	-	100
Hydrogen	-	10
Columbium	-	1000
Molybdenum	-	200
Nickel	-	50
Cobalt	-	50
Iron	-	50
Vanadium	-	20
Tungsten	7.0 w/o	9.0 w/o
Hafnium	1.8 w/o	2.4 w/o
Tantalum	Remainder	-

<u>Material Thickness</u> <u>Inches</u>	<u>Thickness Tolerances</u> <u>Inch</u>	<u>Width Tolerances</u> <u>Inch</u>
Less than 0.003	+0.0008, -0.0000	+0.031, -0.000
0.003 to 0.005	± 0.001	+0.031, -0.000
0.005 to 0.010	± 0.0015	+0.031, -0.000

3.8. General. The finished product shall be visibly free from oxide or scale of any nature, grease, oil, residual lubricants, and other extraneous materials. Cracks, laps, seams, gouges, and fins shall be unacceptable.

4. QUALITY ASSURANCE PROVISIONS

4.1. Vendor Responsibility. The manufacturer shall make all tests and inspections of the material covered by this specification before shipment, unless otherwise specified. All test and inspection results shall be furnished to the purchaser.

4.2. Sample Selection. Care shall be exercised to insure that the sample selected for testing and chemical analyses are representative of the material and uncontaminated by the sampling procedure. If there is any question about the sampling technique or the analysis, the methods for sampling and analysis shall be those agreed to by the buyer and seller.

4.3. Chemical Analysis. Chemical analyses shall be conducted by mutually acceptable procedures, such as the vacuum fusion methods for gases, the combustion method for carbon, and the spectrochemical methods for metallic elements.

4.4. Number of Tests Required. Representative test specimens from the finished product representing each ingot and each lot of material shall be taken to determine conformity to this specification. The minimum frequency of these tests shall be:

Finished Product Chemistry - one per lot per ingot

Bend Test - two per lot per ingot

4.5. Reports. The manufacturer shall supply at least three copies of a report showing non-proprietary manufacturing methods, processing conditions, and test procedures and results for each lot of material in the shipment. The report shall also include the number of the specification and the purchase order or contract number.

4.6. Rejection. Material not conforming to this specification or to any authorized modification shall be subject to rejection. Unless otherwise specified, rejected material may be returned to the manufacturer at the manufacturer's expense if the purchaser does not receive other instructions for disposition within three weeks after notice of rejection.

4.7. Referee. If the manufacturer and the purchaser disagree about the conformance of the material to the requirements of this specification or any special test specified by the purchaser, a mutually acceptable referee's test shall be used to determine conformance.

5. PREPARATION FOR DELIVERY

5.1. Identification. Each bundle, box, or carton shall be legibly and conspicuously marked or tagged with the number of this specification, purchase order or contract number, type, ingot number, lot number, nominal size, and the gross, net, and tare weights. When each bundle, box or carton consists of components from more than one ingot number or lot number, each component shall be identified individually.

5.2. Packing. Each individual item shall be wrapped in heavy gauge polyethylene film or other similar material and packed in a manner assuring safe delivery when properly transported by any common carrier.

6. DEFINITIONS

6.1. Lot. A lot shall include all material of the same size, shape, condition and finish from one heat of material and which has received the same processing, has been annealed in the same vacuum annealing charge and has been processed simultaneously in all operations in which temperatures may reach 500°F or above. When process temperatures and environments are closely controlled or when closely adjacent sizes receive similar processing, lots may be combined for chemical, tensile and stress-rupture tests only, provided prior written approval has been obtained from the General Electric Company.

6.2. Check Analysis. An analysis, made or requested by the purchaser, of the metal after it has been processed into finished mill forms, to verify the composition within a heat or lot. Check analysis tolerances do not broaden the specified heat analysis requirements but rather cover variations between laboratories in the measurement of the chemical content.

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SPPS-68
23 December 1964
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SPECIFICATION

WIRE: T-111 (Ta-8W-2Hf) ALLOY

prepared for
NATIONAL AERONAUTICS AND SPACE ADMINISTRATION
Contract NAS 3-2547

SPACE POWER AND PROPULSION SECTION
MISSILE AND SPACE DIVISION
GENERAL ELECTRIC COMPANY
CINCINNATI, OHIO 45215

SPECIFICATION

WIRE: T-111 (Ta-8W-2Hf) ALLOY

1. SCOPE

1.1. Scope. This specification covers T-111 (Ta-8W-2Hf) alloy in wire form for use as weld filler material in fabricating components intended for high temperature structural applications and alkali metal containment.

2. APPLICABLE DOCUMENTS

- 2.1. Government Documents. None
- 2.2. Non-Government Documents. None

3. REQUIREMENTS

- 3.1. Acknowledgments. The vendor shall mention this specification in all quotations and all purchase order acknowledgments.
- 3.2. Manufacture. Material covered by this specification shall be made from ingots which have been double vacuum melted by the electron beam and/or consumable electrode arc melting processes. Breakdown operations shall be performed with conventional extrusion, forging, and rolling equipment normally found in primary ferrous and nonferrous plants.
- 3.3. Processing. The starting stock size, processing temperatures, percentages of reduction, in-process annealing temperatures and times shall be selected by the vendor.
- 3.4. Condition. The finished product shall be supplied in the fully recrystallized condition throughout the cross-sectional area. Annealing shall be carried out in a vacuum of less than 1×10^{-5} torr. All mill products to be annealed shall be thoroughly degreased, chemically cleaned and protected from furnace parts by a layer of fresh tantalum, columbium or Cb-1Zr alloy foil 0.002-inch thick or greater. When

annealing is carried out in a vacuum greater than 1×10^{-5} torr, with the prior approval of the purchaser, all mill products shall be enclosed in a chemically cleaned tantalum, columbium or Cb-1Zr alloy re-tort or wrapped in a minimum of two layers of fresh tantalum, columbium or Cb-1Zr alloy foil 0.002-inch thick or greater. The conditions of final annealing shall be reported in the certificate of compliance.

3.5. Chemical Composition

3.5.1. Ingot/Billet Composition. The chemical composition of ingots and billets for conversion to finished products shall conform to Table I (page 4). A minimum of four analyses shall be obtained as follows: ingot top-center, mid-radius and edge, and ingot bottom-center; all analyses must conform to ranges stated in Table I.

3.5.2. Final Product Composition. The manufacturer's ingot analyses shall be considered the chemical analysis for products supplied under this specification (Table I) except carbon, oxygen, nitrogen and hydrogen content which shall be determined in the finished product.

3.5.3. Check Analysis. Finished product analysis shall not exceed the following limits or variations:

For Material Greater Than 0.030 Inch in Diameter

<u>Element</u>	<u>Check Analysis Limits, Max., ppm</u>	<u>Permissible Variations in Check Analysis, ppm</u>
Carbon	50	+ 10
Oxygen	150	+ 20
Nitrogen	75	+ 10
Hydrogen	10	+ 2

For Material 0.030 Inch and Less in Diameter

<u>Element</u>	<u>Check Analysis Limits, Max., ppm</u>	<u>Permissible Variations in Check Analysis, ppm</u>
Carbon	75	+ 10
Oxygen	300	+ 20
Nitrogen	100	+ 10
Hydrogen	10	+ 2

TABLE I
CHEMICAL COMPOSITION
T-111 (Ta-8W-2Hf) ALLOY

<u>Element</u>	<u>Minimum Content</u> <u>ppm</u>	<u>Maximum Content</u> <u>ppm</u>
Carbon	-	50
Nitrogen	-	50
Oxygen	-	100
Hydrogen	-	10
Columbium	-	1000
Molybdenum	-	200
Nickel	-	50
Cobalt	-	50
Iron	-	50
Vanadium	-	20
Tungsten	7.0 w/o	9.0 w/o
Hafnium	1.8 w/o	2.4 w/o
Tantalum	Remainder	-

3.6. Tolerances

3.6.1. Definition. Wire - material less than 0.125 inch in diameter.

3.6.2. Diameter. The permissible variation in diameter shall not exceed the following limits:

<u>Diameter, Inch</u>	<u>Diameter Variation, Inch</u>
0.005 to 0.009	± 0.0002
0.010 to 0.019	± 0.0003
0.020 to 0.029	± 0.0005
0.030 to 0.061	± 0.001
0.062 to 0.125	± 0.002

3.7. General. The finished product shall be visibly free from oxide or scale of any nature, grease, oil, residual lubricants, and other extraneous materials. Cracks, laps, seams, gouges and fins shall be unacceptable.

4. QUALITY ASSURANCE PROVISIONS

4.1. Vendor Responsibility. The manufacturer shall make all tests and inspections of the material covered by this specification before shipment, unless otherwise specified. All test and inspection results shall be furnished to the purchaser.

4.2. Sample Selection. Care shall be exercised to insure that the samples selected for testing and chemical analyses are representative of the material and uncontaminated by the sampling procedure. If there is any question about the sampling technique or the analysis, the methods for sampling and analysis shall be those agreed to by the buyer and seller.

4.3. Chemical Analysis. Chemical analyses shall be conducted by mutually acceptable procedures, such as the vacuum fusion methods for gases, the combustion method for carbon, and the spectrochemical methods for metallic elements.

4.4. Number of Tests Required. Representative test specimens from the finished product representing each ingot and each lot of material shall be taken to determine conformity to this specification. The minimum frequency of these tests shall be:

Finished Product Chemistry - one per lot per ingot

4.5. Reports. The manufacturer shall supply at least three copies of a report showing non-proprietary manufacturing methods, processing conditions, and test procedures and results for each lot of material in the shipment. The report shall also include the number of the specification and the purchase order or contract number.

4.6. Rejection. Material not conforming to this specification or to any authorized modification shall be subject to rejection. Unless otherwise specified, rejected material may be returned to the manufacturer at the manufacturer's expense if the purchaser does not receive other instructions for disposition within three weeks after notice of rejection.

4.7. Referee. If the manufacturer and the purchaser disagree about the conformance of the material to the requirements of this specification or any special test specified by the purchaser, a mutually acceptable referee's test shall be used to determine conformance.

5. PREPARATION FOR DELIVERY

5.1. Identification. Each bundle, box, or carton shall be legibly and conspicuously marked or tagged with the number of this specification, purchase order, or contract number, type, ingot number, lot number, nominal size, and the gross, net, and tare weights. When each bundle, box or carton consists of components from more than one ingot number or lot number, each component shall be identified individually.

5.2. Packing. Each individual item shall be wrapped in heavy gauge polyethylene film or similar material and packed in a manner assuring safe delivery when properly transported by any common carrier.

6. DEFINITIONS

6.1. Lot. A lot shall include all material of the same size, shape, condition and finish from one heat of material and which has received the same processing, has been annealed in the same vacuum annealing charge and has been processed simultaneously in all operations in which

temperatures may reach 500°F or above. When process temperatures and environments are closely controlled or when closely adjacent sizes receive similar processing, lots may be combined for chemical, tensile and stress-rupture tests only, provided prior written approval has been obtained from the General Electric Company.

6.2. Check Analysis. An analysis, made or requested by the purchaser of the metal after it has been processed into finished mill forms, to verify the composition within a heat or lot. Check analysis tolerances do not broaden the specified heat analysis requirements but rather cover variations between laboratories in the measurement of the chemical content.

SPECIFICATION

BAR AND ROD: T-222 (Ta-10.4W-2.4Hf-0.01C) ALLOY

prepared for
NATIONAL AERONAUTICS AND SPACE ADMINISTRATION
Contract NAS 3-2547

SPACE POWER AND PROPULSION SECTION
MISSILE AND SPACE DIVISION
GENERAL ELECTRIC COMPANY
CINCINNATI, OHIO 45215

SPECIFICATION

BAR AND ROD: T-222 (Ta-10.4W-2.4Hf-0.01C) ALLOY

1. SCOPE

1.1. Scope. This specification covers T-222 (Ta-10.4W-2.4Hf-0.01C) alloy in bar and rod form for high temperature structural applications and alkali metal containment.

2. APPLICABLE DOCUMENTS

2.1. Government Documents. None

2.2. Non-Government Documents

ASTM Designation E8-57T
(26 December 1957)

Method of Tension Testing of
Metallic Materials

ASTM Designation E29-58T
(1958)

Recommended Practices for Des-
ignating Significant Places in
Specified Limiting Values

ASTM Designation E112-61
(1961)

Estimating the Average Grain
Size of Metals

AMS 2635
(15 August 1958)

Radiographic Inspection

AMS 2645
(1 March 1955)

Fluorescent Penetrant Inspec-
tion

AMS 2646
(1 March 1955)

Contrast Dye Penetrant Inspec-
tion

3. REQUIREMENTS

3.1. Acknowledgments. The vendor shall mention this specification in all quotations and all purchase order acknowledgments.

3.2. Manufacture. Material covered by this specification shall be made from ingots which have been double vacuum melted by the electron beam and/or consumable electrode arc melting processes. Breakdown operations shall be performed with conventional extrusion, forging and rolling equipment normally found in primary ferrous and nonferrous plants.

3.3. Processing. The starting stock size, processing temperatures, percentages of reduction, in-process annealing temperatures and times shall be selected by the vendor to achieve the grain size range specified in paragraph 3.6 and mechanical properties specified in paragraph 3.7. The amount of total reduction from the turned ingot to finished product shall exceed 75%. The amount of final reduction for each mill product, imparted just prior to the final vacuum heat treatment and the total reduction since the previous recrystallization anneal, shall be reported in the certificate of compliance.

3.4. Condition

3.4.1. General. The finished product shall be supplied in the recrystallized condition throughout the cross-sectional area to the grain size range specified in paragraph 3.6. All annealing shall be carried out in a vacuum of less than 1×10^{-5} torr.

3.4.2. Heat Treatment. All mill products to be annealed shall be thoroughly degreased, chemically cleaned and protected from furnace parts by a layer of fresh tantalum, columbium or Cb-1Zr foil 0.002-inch thick or greater. When annealing is carried out in a vacuum greater than 1×10^{-5} torr, with the prior approval of the purchaser, all mill products shall be enclosed in a chemically cleaned tantalum, columbium, or columbium-1% zirconium alloy retort or wrapped in a minimum of two layers of fresh tantalum, columbium or columbium-1% zirconium alloy foil 0.002-inch thick or greater. The conditions of final annealing shall be reported in the certificate of compliance.

3.4.3. Contamination. All items are to be free of contamination or internal oxidation. After final heat treatment, the material shall be examined metallographically for evidence of possible contamination caused by unsatisfactory heat treating atmospheres or processing conditions. A microhardness traverse shall show a hardness increase not greater than 50 VHN from the center to the surface of a cross-sectional sample of the final product. At the discretion of the purchaser, samples taken to include at least one surface of the final product, and not exceeding 0.050-inch thick, may be chemically analyzed by the

purchaser for oxygen, nitrogen, hydrogen and carbon. The analyses shall not exceed the limits set forth in paragraph 3.5.3. Any indication of contamination shall be cause for rejection of all material represented by that sample. The material shall be acceptable if the contaminated layer is completely eliminated before shipment by a machining operation within the specified dimensions and tolerances

3.5. Chemical Composition

3.5.1. Ingot/Billet Composition. The chemical composition of ingots and billets for conversion to finished products shall conform to Table I (page 5). A minimum of four analyses shall be obtained as follows: ingot top-center, mid-radius and edge, and ingot bottom-center; all analyses must conform to ranges stated in Table I.

3.5.2. Final Product Composition. The manufacturer's ingot analyses shall be considered the chemical analysis for products supplied under this specification (Table I) except carbon, oxygen, nitrogen and hydrogen content which shall be determined in the finished product.

3.5.3. Check Analysis. Finished product analysis shall not exceed the following limits or variations:

<u>Element</u>	<u>Check Analysis Limits, Max., ppm</u>	<u>Permissible Variations in Check Analysis, ppm</u>
Carbon	80 min; 175 max.	± 10
Oxygen	150	+ 20
Nitrogen	75	+ 10
Hydrogen	10	+ 2

3.6. Grain Size. The grain size of the final products shall conform to the following limits:

<u>Product Diameter or Thickness, Inches</u>	<u>Minimum Allowable ASTM Grain Size No.</u>	<u>Allowable Spread in ASTM Grain Size Nos. in Any One Item</u>	<u>% Rx Minimum</u>
0.125 to 0.250	4	2	100
0.250 to 0.500	4	2	100
0.500 to 1.0	4	2	100
1.0 to 2.0	4	2	95
Greater than 2.0	3	3	90

TABLE I
CHEMICAL COMPOSITION
T-222 (Ta-10.4W-2.4Hf-0.01C) ALLOY

<u>Element</u>	<u>Minimum Content</u> <u>ppm</u>	<u>Maximum Content</u> <u>ppm</u>
Carbon	80	175
Nitrogen	-	50
Oxygen	-	100
Hydrogen	-	10
Columbium	-	1000
Molybdenum	-	200
Nickel	-	50
Cobalt	-	50
Iron	-	50
Vanadium	-	20
Tungsten	9.6 w/o	11.2 w/o
Hafnium	2.2 w/o	2.8 w/o
Tantalum	Remainder	-

3.7. Mechanical Properties. The final product shall satisfy the following mechanical property requirements:

3.7.1. Room Temperature Tensile Properties. Representative samples of the material in final form shall be capable of the following property limits at room temperature (65°-85°F).

<u>Ultimate Tensile Strength, ksi</u>		<u>0.2% Yield Strength, ksi</u>		<u>Elong., % in 4D</u>
<u>Minimum</u>	<u>Maximum</u>	<u>Minimum</u>	<u>Maximum</u>	<u>Minimum</u>
105	125	100	120	20

3.7.2. Stress-to-Rupture Tests. The material shall be capable of achieving the following stress-rupture life under suitable environmental conditions (see paragraph 5.4.3).

<u>Test Temp., °F</u>	<u>Stress, ksi</u>	<u>Minimum Life Hours</u>
2400	30	20

Chemical analysis of stress-rupture specimens after test shall demonstrate that the degree of environmental contamination did not exceed the following limits: total increase in oxygen plus nitrogen content--less than 50 ppm; increase in hydrogen content--less than 5 ppm; increase in carbon content--less than 10 ppm. The following limits shall apply to check analyses of the analytical results:

Carbon	±	10 ppm
Oxygen	±	25 ppm
Nitrogen	±	25 ppm
Hydrogen	±	2 ppm

3.8. Tolerances

3.8.1. Rolled, Swaged, or Drawn Rounds

3.8.1.1. Definition. Rod - 3.5 inches in diameter or less.

3.8.1.2. Diameter. The permissible variation in diameter and the limits of out-of-roundness of descaled rounds shall not exceed those in Table II (refer to page 8).

3.8.1.3. Cut Lengths. Maximum length variation shall be 0.25 inch.

3.8.1.4. Straightness. Maximum deviation shall be 0.050 inch per foot in any length.

3.8.2. Square or Rectangular Bar

3.8.2.1. Definition. Bar - any straight product with a rectangular cross-section 0.187 inch or more thick and less than 5 inches wide.

3.8.2.2. Dimensions. Unless otherwise specified, forged or rolled square and rectangular shapes shall have the following tolerances:

<u>Thickness</u>	<u>Length</u>	<u>Width</u>
± 0.025 inch or $\pm 5\%$, whichever is less	± 0.125 inch	± 0.125 inch

3.8.2.3. Straightness of Bar. Maximum deviation shall be 0.050 inch per foot in any length.

3.9. Reports. The manufacturer shall supply at least three copies of a report showing non-proprietary manufacturing methods, processing conditions, and test procedures and results for each lot of material in the shipment. The report shall also include the number of the specification and the purchase order or contract number.

4. MAXIMUM ALLOWABLE DISCONTINUITIES

4.1. General. The finished product shall be visibly free from oxide or scale of any nature, grease, oil, residual lubricants, and other extraneous materials. Cracks, laps, seams, gouges, and fins shall be unacceptable.

4.2. Porosity and Inclusions. Indications of internal porosity and non-metallic inclusions greater than 0.020 inch or 3% of the thickness, whichever is smaller, shall be unacceptable. Those indications in the range 0.010 inch to 0.020 inch or 2% of the thickness or diameter, whichever is smaller, shall be a minimum of 0.500 inch apart; those indications less than 0.010 inch shall be a minimum of 0.12 inch apart.

TABLE IIPERMISSIBLE DIMENSIONAL VARIATIONS FOR ROUND BAR

<u>Diameter Inches</u>	<u>Diameter Variation Inch</u>	<u>Out-of-Roundness Inch</u>
0.125 to 0.281	+ 0.002, -0.002	0.004
Over 0.281 to 0.406	+ 0.010, -0.005	0.008
Over 0.406 to 0.625	+ 0.010, -0.005	0.012
Over 0.625 to 0.875	+ 0.015, -0.005	0.015
Over 0.875 to 1.000	+ 0.020, -0.005	0.015
Over 1.000 to 1.375	+ 0.020, -0.010	0.018
Over 1.375 to 1.500	+ 0.020, -0.015	0.020
Over 1.500 to 1.625	+ 0.025, -0.015	0.020
Over 1.625 to 2.000	+ 0.030, -0.020	0.025
Over 2.000 to 2.500	+ 0.032, -0.032	0.025
Over 2.500 to 3.250	+ 0.032, -0.032	0.027
Over 3.250 to 3.500	+ 0.045, -0.045	0.040

Centerless Ground Rounds

0.0625 to 2.00	+ 0.002, -0.002
Over 2.00	+ 0.003, -0.002

4.3. Surface Rework. All surface pores, gouges, and other defects deeper than 0.005 inch or 3% of the thickness, whichever is smaller, shall be unacceptable. Surface imperfections may be faired smooth to remove any notch effect provided dimensional tolerances are still maintained.

5. QUALITY ASSURANCE PROVISIONS

5.1. Vendor Responsibility. The manufacturer shall make all tests and inspections of the material covered by this specification before shipment, unless otherwise specified. All test and inspection results shall be furnished to the purchaser.

5.2. Customer Review. The purchaser or his representative may witness the testing and inspection of the material. The manufacturer shall give the purchaser ample notice of the time and place of designated tests. If the purchaser's representative is not present at this time and a new date is not set, the requirement for purchaser's inspection at the place of testing is waived. When the purchaser's representative is present at the appointed time and place, the manufacturer shall afford him, without charge, all reasonable facilities to assure that the material is being furnished in accordance with this specification. This inspection shall not interfere unnecessarily with production operations.

5.3. Sample Selection. Care shall be exercised to insure that the samples selected for testing and chemical analyses are representative of the material and uncontaminated by the sampling procedure. Samples for the determination of mechanical properties shall be selected so as to consume a minimum of material, i.e., specimens may be taken transverse to the final working direction from bar of sufficient width or from rod greater than 2 inches in diameter. If there is any question about the sampling technique or the analysis, the methods for sampling and analysis shall be those agreed to by the buyer and seller. The location of test samples shall be reported in the certificate of compliance.

5.4. Test Methods

5.4.1. Chemical Analysis. Chemical analyses shall be conducted by mutually acceptable procedures, such as the vacuum fusion methods for gases, the combustion method for carbon, and the spectrochemical methods for metallic elements.

5.4.2. Tensile Test. The tension test shall be conducted in accordance with ASTM Designation E8-57T, "Methods of Tension Testing of Metallic Materials." Yield strength shall be determined by the offset (0.2%) method. The tensile properties shall be determined using a strain rate of 0.005 inch per inch per minute up to 0.6% offset and then 0.05 inch, plus or minus 0.02 inch, per inch per minute to fracture.

5.4.3. Stress-Rupture Test. Stress-rupture properties of specimens shall be determined by mutually acceptable testing techniques. Suggested testing techniques for determining stress-rupture properties are:

Specimens shall be tested in a vacuum of 1×10^{-6} torr or better. The vacuum system shall incorporate an optically tight liquid nitrogen cold trap or a getter-ion pump.

Specimens shall be held for a half hour at the test temperature before application of load.

Test temperature shall be maintained at plus or minus 10°F during the test.

5.4.4. Grain Size. Grain size determinations shall be made according to ASTM Specification E112-61, "Estimating the Average Grain Size of Metals."

5.5. Number of Tests Required. Representative test specimens from the finished product representing each ingot and each lot of material shall be taken to determine conformity to this specification. The minimum frequency of these tests shall be:

Finished Product Chemistry - one per lot per ingot

Tensile Test - two per lot per ingot

Stress-Rupture Test - two per lot per ingot

Grain Size - two per lot per ingot

Microhardness Traverse - one per lot per ingot

5.6. Retest and Rework

5.6.1. Surface Contamination. Any sample or specimen exhibiting obvious surface contamination or improper preparation which disqualifies it as a truly representative sample shall be replaced with a new sample.

5.6.2. Rework. If inspection and test results of a lot do not conform to the requirements of this specification, the lot may be reworked at the option of the manufacturer. The lot shall be acceptable if all test results, after reworking, conform to this specification.

5.7. Inspection

5.7.1. Methods of Inspection

5.7.1.1. Radiographic. Whenever specified, the product shall be radiographed and found free of porosity and inclusions as specified in paragraph 4.2 using the technique described in AMS 2635, "Radiographic Inspection." The radiographs and product shall be identified so the exact position of each radiograph can be correlated with the specific area on the particular product.

5.7.1.2. Ultrasonic. All material 0.125-inch diameter and larger shall be inspected ultrasonically.

5.7.1.2.1. Method and Equipment. The finished products shall be ultrasonically inspected by the immersed technique at 5 mc or above. Transducers shall be no larger than 0.75-inch diameter. Surface finishes shall be no rougher than 125 rms. Inspection shall be by longitudinal wave and by shear wave in two perpendicular directions, i.e., longitudinal and transverse and shall be with focused transducers appropriate to the diameter being inspected (360 degree transducers are allowable where appropriate). Automatic equipment which traverses a spiral path is satisfactory; but three traverses shall be made, one with the transducer in the circumferential shear position, one with the transducer in the axial shear position, and one with the transducer in the longitudinal wave position, unless otherwise specified.

5.7.1.2.2. Calibration of Bar and Rod. Calibration shall be on notches and holes in a segment of the material reserved solely for calibration purposes or in a calibration specimen of similar nature and shape. The depth of the notches shall be 3% of the bar thickness, 1.5% of the rod diameter, or 0.005 inch, whichever is smaller; the width, no greater

than depth; the length, greater than beam width. The notches shall be placed perpendicular to the direction of the shear wave beam and perpendicular to the surface, e.g., axial and circumferential notches on bar. In addition to the notches, a 0.020-inch diameter hole shall be made at least 0.5-inch deep in the calibration piece parallel to the surface at a distance from the surface of $1/2$ the thickness or diameter or, if the thickness exceeds 0.750 inch, $1/4$, $1/2$ and $3/4$ of thickness. Calibration settings to achieve 80% amplitude of these notches or holes along with the magnitude of the other applicable calibration defects shall be recorded. For example, on bar with shear wave, the notch on the near surface should be set at 80% and the amplitudes recorded for the indications from the hole and the notch on the far surface. Gain settings should be recorded to achieve 80% as above and 80% on each of the other applicable calibration defects. For longitudinal wave, only the 0.020-inch diameter holes, with additional holes at $1/4$ and $1/2$ the thickness if the thickness exceeds 0.750 inch, shall be used for calibration.

5.7.1.2.3. Evaluation. Evaluation during inspection shall be made against the appropriate calibration defect. For example, on bar with shear wave, the defects on or near the far surface shall be compared to the calibration from the far surface notch; defects near the center shall be compared to the calibration from the hole at the appropriate depth; defects on the near surface shall be compared to the calibration from the near surface notch.

5.7.1.2.4. Reports. The ultrasonic inspection report shall contain the equipment serial numbers, calibration amplitudes and gain settings, and the amplitude and locations of each defect whose amplitude is 60% or greater.

5.7.1.2.5. Rejection. The above procedure shall be followed, and indications of defects which exceed the magnitude obtained from the appropriate calibrated notch in the sample shall be cause for rejection, unless otherwise agreed by the purchaser and vendor.

5.7.1.3. Penetrant Inspection. The exterior surface of the product shall be penetrant inspected and found free of flaws as specified in paragraph 4.3 using AMS 2645, "Fluorescent Penetrant Inspection," or AMS 2646, "Contrast Dye Penetrant Inspection." All parts thus inspected shall be marked with ink stamps as described in the specification; impression stampings or etching shall be unacceptable.

5.7.1.4. Reports. The manufacturer shall supply at least three copies of a report showing inspection results for each lot of material in the shipment. The report shall also include the number of the specification and the purchase order or contract number.

5.8. Rejection. Material not conforming to this specification or to any authorized modification shall be subject to rejection. Unless otherwise specified, rejected material may be returned to the manufacturer at the manufacturer's expense if the purchaser does not receive other instructions for disposition within three weeks after notice of rejection.

5.9. Referee. If the manufacturer and the purchaser disagree about the conformance of the material to the requirements of this specification or any special test specified by the purchaser, a mutually acceptable referee's test shall be used to determine conformance.

6. PREPARATION FOR DELIVERY

6.1. Identification. Each bundle, box, or carton shall be legibly and conspicuously marked or tagged with the number of this specification, purchase order or contract number, type, ingot number, lot number, nominal size, and the gross, net, and tare weights. When each bundle, box or carton consists of components from more than one ingot number or lot number, each component shall be identified individually.

6.2. Packing. Each individual item shall be wrapped in heavy gauge polyethylene film or similar material and packed in a manner assuring safe delivery when properly transported by any common carrier.

7. DEFINITIONS

7.1. Lot. A lot shall include all material of the same size, shape, condition and finish from one heat of material and which has received the same processing, has been annealed in the same vacuum annealing charge and has been processed simultaneously in all operations in which temperatures may reach 500°F or above. When process temperatures and environments are closely controlled or when closely adjacent sizes receive similar processing, lots may be combined for chemical, tensile and stress-rupture tests only, provided prior written approval has been obtained from the General Electric Company.

7.2. Check Analysis. An analysis may be requested by the purchaser of the metal after it has been processed into finished mill forms, to

verify the composition within a heat or lot. Check analysis tolerances do not broaden the specified heat analysis requirements but rather cover variations between laboratories in the measurement of the chemical content.

7.3. Significance of Numerical Limits. For determining compliance with the specified limits for requirements of the properties listed below, an observed value or a calculated value shall be rounded off using the rounding-off method in ASTM Designation E29-58T, "Recommended Practices for Designating Significant Places in Specified Limiting Values."

<u>Test</u>	<u>Rounded-Off Unit for Observed or Calculated Value</u>
Chemical composition and dimensional tolerances (when expressed decimally)	Nearest unit in the last right-hand place of figures of the specified limit
Tensile strength	Nearest 100 psi
Elongation	Nearest 1%
Rupture life	Nearest 0.1 hour

SPECIFICATION

SHEET, PLATE, AND STRIP: T-222 (Ta-10.4W-2.4Hf-0.01C) ALLOY

prepared for

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

Contract NAS 3-2547

SPACE POWER AND PROPULSION SECTION

MISSILE AND SPACE DIVISION

GENERAL ELECTRIC COMPANY

CINCINNATI, OHIO 45215

SPECIFICATION

SHEET, PLATE, AND STRIP: T-222 (Ta-10.4W-2.4Hf-0.01C) ALLOY

1. SCOPE

1.1. Scope. This specification covers T-222 (Ta-10.4W-2.4Hf-0.01C) alloy in sheet, plate and strip form intended for high temperature structural applications and alkali metal containment.

2. APPLICABLE DOCUMENTS

2.1. Government Documents. None

2.2. Non-Government Documents

ASTM Designation E8-57T
(26 December 1957)

Method of Tension Testing of
Metallic Materials

ASTM Designation E29-58T
(1958)

Recommended Practices for Des-
ignating Significant Places
in Specified Limiting Values

ASTM Designation E112-61
(1961)

Estimating the Average Grain
Size of Metals

AMS 2242A
(1 December 1950)

Tolerances, Corrosion and Heat
Resistant Sheet, Strip and Plate

AMS 2635
(15 August 1958)

Radiographic Inspection

AMS 2645
(1 March 1955)

Contrast Dye Penetrant Inspec-
tion

MAB-176-M
(6 September 1961)

Evaluation Test Methods for
Refractory Metal Sheet Mate-
rials

3. REQUIREMENTS

3.1. Acknowledgments. The vendor shall mention this specification in all quotations and all purchase order acknowledgments.

3.2. Manufacture. Material covered by this specification shall be made from ingots which have been double vacuum melted by the electron beam and/or consumable electrode arc melting processes. Breakdown operations shall be performed with conventional extrusion, forging and rolling equipment normally found in primary ferrous and nonferrous plants.

3.3. Processing. The starting stock size, processing temperatures, percentages of reduction, in-process annealing temperatures and times shall be selected by the vendor to achieve the grain size range specified in paragraph 3.6 and mechanical properties specified in paragraph 3.7. The amount of total reduction from the turned ingot to the final product shall exceed 75%. The amount of final reduction for each mill product, imparted just prior to the final vacuum heat treatment and the total reduction since the previous recrystallization anneal, shall be reported in the certificate of compliance.

3.4. Condition

3.4.1. General. The finished product shall be supplied in the recrystallized condition throughout the cross-sectional area to the grain size range specified in paragraph 3.6.

3.4.2. Heat Treatment. All annealing shall be carried out in a vacuum of less than 1×10^{-5} torr. All mill products to be annealed shall be thoroughly degreased, chemically cleaned and protected from furnace parts by a layer of fresh tantalum, columbium or Cb-12Zr foil 0.002-inch thick or greater. When annealing is carried out in a vacuum greater than 1×10^{-5} torr, with the prior approval of the purchaser, all mill products shall be enclosed in a chemically cleaned tantalum, columbium or Cb-12Zr alloy retort or wrapped in a minimum of two layers of fresh tantalum, columbium or Cb-12Zr alloy foil 0.002-inch thick or greater. The conditions of final annealing shall be reported in the certificate of compliance.

3.4.3. Contamination. All items are to be free of contamination or internal oxidation. After final heat treatment, the material shall be examined metallographically for evidence of possible contamination caused by unsatisfactory heat treating atmospheres or processing conditions. A microhardness traverse shall show a hardness increase not

greater than 50 VHN from the center to the surface of a cross sectional sample of the final product. At the discretion of the purchaser, samples taken to include at least one surface of the final product, and not exceeding 0.050-inch thick, may be chemically analyzed by the purchaser for oxygen, nitrogen, hydrogen and carbon. The analyses shall not exceed the limits set forth in paragraph 3.5.3. Any indication of contamination shall be cause for rejection of all material represented by that sample. The material shall be acceptable if the contaminated layer is completely eliminated before shipment by a machining operation within the specified dimensions and tolerances.

3.5. Chemical Composition

3.5.1. Ingot/Billet Composition. The chemical composition of ingots and billets for conversion to finished products shall conform to Table I (page 5). A minimum of four analyses shall be obtained as follows: ingot top-center, mid-radius and edge, and ingot bottom-center; all analyses must conform to ranges stated in Table I.

3.5.2. Final Product Composition. The manufacturer's ingot analyses shall be considered the chemical analysis for products supplied under this specification (Table I) except carbon, oxygen, nitrogen and hydrogen content which shall be determined in the finished product.

3.5.3. Check Analysis. Finished product analysis shall not exceed the following limits or variations:

<u>Element</u>	<u>Check Analysis Limits, Max., ppm</u>	<u>Permissible Variations in Check Analysis, ppm</u>
Carbon	80 min; 175 max.	± 10
Oxygen	150	+ 20
Nitrogen	75	+ 10
Hydrogen	10	+ 2

3.6. Grain Size. The grain size of the final products shall conform to the following limits:

TABLE ICHEMICAL COMPOSITIONT-222 (Ta-10.4W-2.4Hf-0.01C) ALLOY

<u>Element</u>	<u>Minimum Content</u> <u>ppm</u>	<u>Maximum Content</u> <u>ppm</u>
Carbon	80	175
Nitrogen	-	50
Oxygen	-	100
Hydrogen	-	10
Columbium	-	1000
Molybdenum	-	200
Nickel	-	50
Cobalt	-	50
Iron	-	50
Vanadium	-	20
Tungsten	9.6 w/o	11.2 w/o
Hafnium	2.2 w/o	2.8 w/o
Tantalum	Remainder	-

<u>Product Thickness, Inches</u>	<u>Minimum Allowable ASTM Grain Size No.</u>	<u>Allowable Spread in ASTM Grain Size Nos. in Any One Item</u>	<u>% R_x Minimum</u>
0.010 to 0.060	6	2	100
0.060 to 0.125	4	2	100
0.125 to 0.187	4	2	100
0.187 to 0.500	3	3	95
0.500 to 1.0	3	3	95
Greater than 1.0	3	3	90

3.7. Mechanical Properties. The final product shall satisfy the following mechanical property requirements:

3.7.1. Room Temperature Tensile Properties. Representative samples of the material in final form shall be capable of the following property limits at room temperature (65°-85°F).

<u>Ultimate Tensile Strength, ksi</u>		<u>0.2% Yield Strength, ksi</u>		<u>Elong., % in 4D</u>
<u>Minimum</u>	<u>Maximum</u>	<u>Minimum</u>	<u>Maximum</u>	<u>Minimum</u>
105	125	100	120	20

3.7.2. Stress-to-Rupture Tests. The material shall be capable of achieving the following stress-rupture life under suitable environmental conditions (see paragraph 5.4.3.).

<u>Test Temp., °F</u>	<u>Stress, ksi</u>	<u>Minimum Life Hours</u>
2400	30	20

Chemical analysis of stress-rupture specimens after test shall demonstrate that the degree of environmental contamination did not exceed the following limits: total increase in oxygen plus nitrogen content--less than 50 ppm; increase in hydrogen content--less than 5 ppm; increase in carbon content--less than 10 ppm. The following limits shall apply to check analyses of the analytical results:

Carbon	±	10 ppm
Oxygen	±	25 ppm
Nitrogen	±	25 ppm
Hydrogen	±	2 ppm

3.7.3. Bend Ductility. Representative samples of the materials in final form shall withstand the following bend test at room temperature without failure when tested according to procedures described in the most recent revision of the Materials Advisory Board report MAB-176M, "Evaluation Test Methods for Refractory Metal Sheet Materials." The samples shall be sectioned with the long axis of the bend specimens perpendicular to the final rolling direction.

3.7.3.1. Sheet 0.060 inch in thickness and under shall be bent over a 1T radius through 105° at a ram speed of 1 inch per minute and subsequently flattened for a total bend of 180°.

3.7.3.2. Sheet over 0.060 inch to 0.187 inch in thickness shall be bent over a 1T radius through 105° at a ram speed of 1 inch per minute.

3.8. Tolerances

3.8.1. Plate

3.8.1.1. Definition. Plate includes material 6 inches wide or over and 0.187 inch or more in thickness.

3.8.1.2. Dimensions. Plate dimensions shall conform to the following tolerances:

<u>Thickness</u>	<u>Width</u>	<u>Length</u>
± 0.025 inch or ± 5%, whichever is less	± 0.125 inch	± 0.125 inch

3.8.1.3. Flatness. Flatness tolerance on plate shall conform to AMS 2242A, "Tolerances, Corrosion and Heat Resistant Sheet, Strip and Plate."

3.8.2. Sheet

3.8.2.1. Definition. Sheet includes material 6 inches wide or over and up to 0.187 inch in thickness.

3.8.2.2. Dimensions. Sheet dimensions shall conform to those presented in Table II.

3.8.2.3. Flatness. See paragraph 3.8.3.3.

3.8.3. Strip

3.8.3.1. Definition. Strip includes material 6 inches wide or less and up to 0.187 inch in thickness.

3.8.3.2. Dimensions. Strip dimensions shall conform to those presented in Table II.

3.8.3.3. Flatness. Total deviation from flatness of sheet and strip shall not exceed 6% as determined by the formula:

$$\frac{H}{L} \times 100 = \% \text{ Flatness Deviation}$$

where

H = maximum distance from a flat reference surface

and

L = minimum distance from this point to the point of contact with the reference surface.

The actual values shall be reported. In determining flatness, the sheet shall not be subject to external pressure at any point but shall lie freely on a flat surface during measurement. Oilcanning will be reported. An estimate of the extent (area, height, etc.,) of these defects shall be made.

3.9. Reports. The manufacturer shall supply at least three copies of a report showing non-proprietary manufacturing methods, processing conditions, and test procedures and results for each lot of material in the shipment. The report shall also include the number of the specification and the purchase order or contract number.

4. MAXIMUM ALLOWABLE DISCONTINUITIES

4.1. General. The finished product shall be visibly free from oxide or scale of any nature, grease, oil, residual lubricants, and other extraneous materials. Cracks, laps, seams, gouges, and fins shall be unacceptable.

TABLE II

DIMENSIONAL TOLERANCES FOR SHEET AND STRIP

<u>Material Thickness, Inch</u>	<u>Width, Inches</u>	<u>Thickness Tolerances, Inch</u>
0.010-0.019	to 24	± 0.001
0.020-0.039	to 24	± 0.0015
0.040-0.059	to 24	± 0.002
0.060-0.089	to 24	± 0.003
0.090-0.0129	to 24	± 0.004
0.130-0.159	to 24	± 0.005
0.160-0.187	to 24	± 0.010

<u>Material Thickness, Inch</u>	<u>Width Tolerances, Inch</u>
0.010-0.059	+ 0.031, -0
0.060-0.125	+ 0.046, -0
0.126-0.187	+ 0.125, -0

<u>Material Thickness, Inch</u>	<u>Length Tolerances, Inch</u>
0.010-0.059	+ 0.046, -0
0.060-0.125	+ 0.062, -0
0.126-0.187	+ 0.125, -0

4.2. Porosity and Inclusions. Indications of internal porosity and non-metallic inclusions greater than 0.020 inch or 3% of the thickness, whichever is smaller, shall be unacceptable. Those indications in the range 0.010 inch to 0.020 inch or 2% of the thickness, whichever is smaller, shall be a minimum of 0.500 inch apart; those indications less than 0.010 inch shall be a minimum of 0.12 inch apart.

4.3. Surface Rework. All surface pores, gouges and other defects deeper than 0.005 inch or 3% of the thickness, whichever is smaller, shall be unacceptable. Surface imperfections may be faired smooth to remove any notch effect provided dimensional tolerances are still maintained.

5. QUALITY ASSURANCE PROVISIONS

5.1. Vendor Responsibility. The manufacturer shall make all tests and inspections of the material covered by this specification before shipment, unless otherwise specified. All test and inspection results shall be furnished to the purchaser.

5.2. Customer Review. The purchaser or his representative may witness the testing and inspection of the material. The manufacturer shall give the purchaser ample notice of the time and place of designated tests. If the purchaser's representative is not present at this time and a new date is not set, the requirement for purchaser's inspection at the place of testing is waived. When the purchaser's representative is present at the appointed time and place, the manufacturer shall afford him, without charge, all reasonable facilities to assure that the material is being furnished in accordance with this specification. This inspection shall not interfere unnecessarily with production operations.

5.3. Sample Selection. Care shall be exercised to insure that the samples selected for testing and chemical analyses are representative of the material and uncontaminated by the sampling procedure. Samples for the determination of mechanical properties shall be selected so as to consume a minimum amount of material, i.e., specimens may be taken transverse to the final working direction from plate and sheet and from strip if of sufficient width. If there is any question about the sampling technique or the analysis, the methods for sampling and analysis shall be those agreed to by the buyer and seller. The location of test samples shall be reported in the certificate of compliance.

5.4. Test Methods

5.4.1. Chemical Analysis. Chemical analyses shall be conducted by mutually acceptable procedures, such as the vacuum fusion methods for gases, the combustion method for carbon, and the spectrochemical methods for metallic elements.

5.4.2. Tensile Test. The tension test shall be conducted in accordance with ASTM Designation E8-57T, "Methods of Tension Testing of Metallic Materials." Yield strength shall be determined by the offset (0.2%) method. The tensile properties shall be determined using a strain rate of 0.005 inch per inch per minute up to 0.6% offset and then 0.050 inch, plus or minus 0.020 inch, per inch per minute to fracture.

5.4.3. Stress-Rupture Tests. Stress-rupture properties of specimens shall be determined by mutually acceptable testing techniques. Suggested testing techniques for determining stress-rupture properties are:

Specimens shall be tested in a vacuum of 1×10^{-6} torr or better. The vacuum system shall incorporate an optically tight liquid nitrogen cold trap or a getter-ion pump.

Specimens shall be held for a half hour at the test temperature before application of load.

Test temperature shall be maintained at plus or minus 10°F during the test.

5.4.4. Grain Size. Grain size determinations shall be made according to ASTM Specification E112-61, "Estimating the Average Grain Size of Metals."

5.5. Number of Tests Required. Representative test specimens from the finished product representing each ingot and each lot of material shall be taken to determine conformity to this specification. The minimum frequency of these tests shall be:

Finished Product Chemistry - one per lot per ingot

Tensile Test - two per lot per ingot

Stress-Rupture Test - two per lot per ingot

Bend Test - two per lot per ingot

Grain Size - two per lot per ingot

Microhardness Traverse - one per lot per ingot

5.6. Retest and Rework

5.6.1. Surface Contamination. Any sample or specimen exhibiting obvious surface contamination or improper preparation which disqualifies it as a truly representative sample shall be replaced with a new sample.

5.6.2. Rework. If inspection and test results of a lot do not conform to the requirements of this specification, the lot may be reworked at the option of the manufacturer. The lot shall be acceptable if all test results, after reworking, conform to this specification.

5.7. Inspection

5.7.1. Methods of Inspection

5.7.1.1. Radiographic. Whenever specified, the product shall be radiographed and found free of porosity and inclusions as specified in paragraph 4.2 using the technique described in AMS 2635, "Radiographic Inspection." The radiographs and product shall be identified so the exact position of each radiograph can be correlated with the specific area on the particular product.

5.7.1.2. Ultrasonic. Unless otherwise agreed to by the purchaser and the vendor, the material shall be inspected ultrasonically.

5.7.1.2.1. Method and Equipment. The finished products shall be ultrasonically inspected by the immersed technique at 5 mc or above. Transducers shall be no larger than 0.75-inch diameter. Surface finishes shall be no rougher than 125 rms. Inspection shall be by longitudinal wave and by shear wave in two perpendicular directions, i.e., longitudinal and transverse shear.

Transducers for the shear wave inspection shall be focused, preferably cylindrically, to a beam no more than 0.125-inch wide in its smaller dimension (where it enters the material being inspected). Cylindrically-focused transducers shall not exceed 2 inches in length. The focal distance shall be adjusted when the transducer is beamed perpendicular to the surface of the calibration piece; then this focal distance shall be maintained throughout the actual inspection. After the focal distance is established, an appropriate shear wave angle shall be set and

the calibration notch indication shall be set at 80% on the indication where the sound beam traverses one or two thicknesses of the sheet (depending on whether the notch is on the far side or incident side of the sheet). Calibration gain settings shall be recorded when the calibration defect is on both the incident and the far side of the sheet. If there is any difference in the indication, that gain setting giving an 80% indication from the side which produces the smaller indication shall be used for inspection. Calibration shall be done before and after the ultrasonic inspection or at the beginning and end of each work shift. If the magnitude of indication from the calibration notch differs 10% or more from the previous calibration, all material inspected since then shall be reinspected.

5.7.1.2.2. Calibration of Plate. Calibration shall be on notches and holes in a segment of the material reserved solely for the calibration purposes. The depth of the notches shall be 0.005 inch, the width shall be 0.005 inch and the length greater than the ultrasonic beam width. The notches shall be placed on the surface of the calibration piece perpendicular to the direction of the intended shear wave inspection, i.e., transverse and longitudinal and at least 1 inch from the edge of the plate. In addition, a 0.020-inch diameter hole shall be made in the calibration piece parallel to the surface to a depth of at least 0.750 inch at a point one-half the thickness of the plate. If the thickness of the plate exceeds 0.750 inch, similar holes shall also be made at points one-quarter and three-quarters of the plate thickness. Calibration settings to achieve 80% amplitude of the notches and holes, along with the magnitude of the other applicable calibration defects, shall be recorded. For example, on plate using a shear wave, the notch on the near surface should be set at 80% and the amplitude recorded for the indications from the hole and notch on the far surface. Gain settings should be recorded to achieve 80% as above and 80% on each of the other applicable calibration defects. For longitudinal wave inspection, only the 0.020-inch diameter holes shall be used for calibration.

5.7.1.2.3. Calibration of Sheet and Strip. The sheet shall be inspected by a shear wave beam pointed in both longitudinal and transverse directions. Calibration shall be done on notches cut perpendicular to the direction of the beam in pieces of sheet of the same material and thickness as that to be inspected. If that portion is later trimmed and scrapped, the calibration notches may be made on a section of the actual sheet. The depth of the calibration notches shall be 3% of the sheet thickness; width no greater than the depth; length, no more than 1 inch. All notches shall be at least 1 inch

from the edge of the sheet. Duplicate notches may be made on the opposite face of the sheet in locations where the sound beam will not intersect both notches in a single traverse, or the sheet may be turned over during calibration to determine the relative response from the calibration notch on both the incident and far side of the sheet.

5.7.1.2.4. Evaluation. Evaluation during inspection shall be made against the appropriate calibration defect. For example, with shear wave, the defects on or near the far surface shall be compared to the calibration from the far surface notch; defects near the center shall be compared to the calibration from the hole at the appropriate depth; defects on the near surface shall be compared to the calibration from the near surface notch.

5.7.1.2.5. Reports. The ultrasonic inspection report shall contain the equipment serial numbers, calibration amplitudes and gain settings and the amplitude and location of each defect whose amplitude is 60% or greater.

5.7.12.6. Rejection. The above procedure shall be followed and indications of defects which exceed the magnitude obtained from the appropriate calibrated notch in the sample shall be cause for rejection, unless otherwise agreed by the purchaser and vendor.

5.7.1.3. Penetrant Inspection. The exterior surface of the product shall be penetrant inspected and found free of flaws as specified in paragraph 4.3 using AMS 2645, "Fluorescent Penetrant Inspection," or AMS 2646, "Contrast Dye Penetrant Inspection." All parts thus inspected shall be marked with ink stamps as described in the specification; impression stampings or etching shall be unacceptable.

5.7.1.4. Reports. The manufacturer shall supply at least three copies of a report showing inspection results for each lot of material in the shipment. The report shall also include the number of the specification and the purchase order or contract number.

5.8. Rejection. Material not conforming to this specification or to any authorized modification shall be subject to rejection. Unless otherwise specified, rejected material may be returned to the manufacturer at the manufacturer's expense if the purchaser does not receive other instructions for disposition within three weeks after notice of rejection.

5.9. Referee. If the manufacturer and the purchaser disagree about the conformance of the material to the requirements of this specification or any special test specified by the purchaser, a mutually acceptable referee's test shall be used to determine conformance.

6. PREPARATION FOR DELIVERY

6.1. Identification. Each bundle, box, or carton shall be legibly and conspicuously marked or tagged with the number of this specification, purchase order or contract number, type, ingot number, lot number, nominal size, and the gross, net, and tare weights. When each bundle, box or carton consists of components from more than one ingot number or lot number, each component shall be identified individually.

6.2. Packing. Each individual item shall be wrapped in heavy gauge polyethylene film or other similar material and packed in a manner assuring safe delivery when properly transported by any common carrier.

7. DEFINITIONS

7.1. Lot. A lot shall include all material of the same size, shape, condition and finish from one heat of material and which has received the same processing, has been annealed in the same vacuum annealing charge and has been processed simultaneously in all operations in which temperatures may reach 500°F or above. When process temperatures and environments are closely controlled or when closely adjacent sizes receive similar processing, lots may be combined for chemical, tensile and stress-rupture tests only, provided prior written approval has been obtained from the General Electric Company.

7.2. Check Analysis. An analysis may be requested by the purchaser of the metal after it has been processed into finished mill forms, to verify the composition within a heat or lot. Check analysis tolerances do not broaden the specified heat analysis requirements but rather cover variations between laboratories in the measurements of the chemical content.

7.3. Significance of Numerical Limits. For determining compliance with the specified limits for requirements of the properties listed below, and observed value or a calculated value shall be rounded off using the rounding-off method in ASTM Designation E29-58T, "Recommended Practices for Designating Significant Places in Specified Limiting Values."

<u>Test</u>	<u>Rounded-Off Unit for Observed or Calculated Value</u>
Chemical composition and dimensional tolerances (when expressed decimally)	Nearest unit in the last right-hand place of figures of the specified limit
Tensile strength	Nearest 100 psi
Elongation	Nearest 1%
Rupture life	Nearest 0.1 hour

SPECIFICATION

SEAMLESS TUBING AND PIPE: T-222
(Ta-10.4W-2.4Hf-0.01C) ALLOY

prepared for
NATIONAL AERONAUTICS AND SPACE ADMINISTRATION
Contract NAS 3-2547

SPACE POWER AND PROPULSION SECTION
MISSILE AND SPACE DIVISION
GENERAL ELECTRIC COMPANY
CINCINNATI, OHIO 45215

SPECIFICATION

SEAMLESS TUBING AND PIPE: T-222
(Ta-10.4W-2.4Hf-0.01C) ALLOY

1. SCOPE

1.1. Scope. This specification covers T-222 (Ta-10.4W-2.4Hf-0.01C) alloy in tube and pipe form intended for high temperature structural applications and alkali metal containment.

2. APPLICABLE DOCUMENTS

2.1. Government Documents. None

2.2. Non-Government Documents

ASTM Designation E8-57T
(26 December 1957)

Method of Tension Testing of
Metallic Materials

ASTM Designation E29-58T
(1958)

Recommended Practices for
Designating Significant Places
in Specified Limiting Values

ASTM Designation E112-61
(1961)

Estimating Average Grain Size
of Metals

AMS 2635
(15 August 1958)

Radiographic Inspection

AMS 2645
(1 March 1955)

Fluorescent Penetrant Inspec-
tion

AMS 2646
(1 March 1955)

Contrast Dye Penetrant Inspec-
tion

3. REQUIREMENTS

3.1. Acknowledgments. The vendor shall mention this specification in all quotations and all purchase order acknowledgments.

3.2. Manufacture. Material covered by this specification shall be made from ingots which have been double vacuum melted by the electron beam and/or consumable electrode arc melting processes. Breakdown operations shall be performed with conventional extrusion, forging, tube reducing and drawing equipment normally found in primary ferrous and nonferrous plants.

3.3. Processing. The starting stock size, processing temperatures, percentages of reduction, in-process annealing temperatures and times shall be selected by the vendor to achieve the grain size range specified in paragraph 3.6 and mechanical properties specified in paragraph 3.7. The total amount of reduction from the turned ingot to the final product shall exceed 75%. The amount of final reduction for each mill product, imparted just prior to the final vacuum heat treatment and the total reduction since the last recrystallization anneal shall be reported in the certificate of compliance.

3.4. Condition

3.4.1. General. The finished product will be supplied in the recrystallized condition throughout the cross-sectional area to the grain size range specified in paragraph 3.6.

3.4.2. Heat Treatment. All annealing shall be carried out in a vacuum of less than 1×10^{-5} torr. All mill products to be annealed shall be thoroughly degreased, chemically cleaned and protected from furnace parts by a layer of fresh tantalum, columbium or Cb-1Zr alloy foil 0.002-inch thick or greater. When annealing is carried out in a vacuum greater than 1×10^{-5} torr, with the prior approval of the purchaser, all mill products shall be enclosed in a chemically cleaned tantalum, columbium or Cb-1Zr alloy retort or wrapped in a minimum of two layers of fresh tantalum, columbium or Cb-1Zr alloy foil 0.002-inch thick or greater. The conditions of final annealing shall be reported in the certificate of compliance.

3.4.3. Contamination. All items are to be free of contamination or internal oxidation. After final heat treatment, the material shall be examined metallographically for evidence of possible contamination caused by unsatisfactory heat treating atmospheres or processing conditions. A microhardness traverse shall show a hardness increase not greater than 50 VHN from the center to the surface of a cross sectional sample of the final product. At the discretion of the purchaser, samples taken to include at least one surface of the final product, and not exceeding 0.050-inch thick, may be chemically analyzed by the

purchaser for oxygen, nitrogen, hydrogen and carbon. The analyses shall not exceed the limits set forth in paragraph 3.5.3. Any indication of contamination shall be cause for rejection of all material represented by that sample. The material shall be acceptable if the contaminated layer is completely eliminated before shipment by a machining operation within the specified dimensions and tolerances.

3.5. Chemical Composition

3.5.1. Ingot/Billet Composition. The chemical composition of ingots and billets for conversion to finished products shall conform to Table I (page 5). A minimum of four analyses shall be obtained as follows: ingot top-center, mid-radius and edge, and ingot bottom-center; all analyses must conform to ranges stated in Table I.

3.5.2. Final Product Composition. The manufacturer's ingot analyses shall be considered the chemical analyses for products supplied under this specification (Table I) except carbon, oxygen, nitrogen and hydrogen content which shall be determined on the finished product.

3.5.3. Check Analysis. The finished product analysis shall not exceed the following limits or variations:

For Wall Thicknesses 0.020 Inch or Greater

<u>Element</u>	<u>Check Analysis Limits, ppm</u>		<u>Permissible Variations in Check Analysis, ppm</u>
	<u>Maximum</u>	<u>Minimum</u>	
Carbon	175	80	± 10
Oxygen	150	-	+ 20
Nitrogen	75	-	+ 10
Hydrogen	10	-	+ 2

For Wall Thicknesses Less Than 0.020 Inch

<u>Element</u>	<u>Check Analysis Limits, ppm</u>		<u>Permissible Variations in Check Analysis, ppm</u>
	<u>Maximum</u>	<u>Minimum</u>	
Carbon	175	80	± 10
Oxygen	300	-	+ 20
Nitrogen	100	-	+ 10
Hydrogen	10	-	+ 2

TABLE I
CHEMICAL COMPOSITION
T-222 (Ta-10.4W-2.4Hf-0.01C) ALLOY

<u>Element</u>	<u>Minimum Content</u> <u>ppm</u>	<u>Maximum Content</u> <u>ppm</u>
Carbon	80	175
Nitrogen	-	50
Oxygen	-	100
Hydrogen	-	10
Columbium	-	1000
Molybdenum	-	200
Nickel	-	50
Cobalt	-	50
Iron	-	50
Vanadium	-	20
Tungsten	9.6 w/o	11.2 w/o
Hafnium	2.2 w/o	2.8 w/o
Tantalum	Remainder	-

3.6. Grain Size. The grain size of the final products shall conform to the following limits:

<u>Product Wall Thickness, Inches</u>	<u>Minimum Allowable ASTM Grain Size No.</u>	<u>Allowable Spread in ASTM Grain Size Nos. in Any One Item</u>	<u>% R_x Minimum</u>
Less than 0.010	6	2	100
0.010 to 0.065	6	2	100
0.065 to 0.125	5	2	100
0.125 to 0.250	4	2	95
0.250 to 0.500	3	3	90

3.7. Mechanical Properties. The final product shall satisfy the following mechanical property requirements:

3.7.1. Room Temperature Tensile Properties. Representative samples of the material in final form shall be capable of the following property limits at room temperature (65°-85°F).

<u>Ultimate Tensile Strength, ksi</u>		<u>0.2% Yield Strength, ksi</u>		<u>Elong., %(1)</u>
<u>Minimum</u>	<u>Maximum</u>	<u>Minimum</u>	<u>Maximum</u>	<u>Minimum</u>
105	125	100	120	20

(1) % Elongation in 4D for Threaded or Button-Head Test Specimens; in 2 Inches for Flat Specimens.

3.7.2. Stress-to-Rupture Tests. The material shall be capable of achieving the following stress-rupture life under suitable environmental conditions (see paragraph 5.4.3):

<u>Test Temp., °F</u>	<u>Stress, ksi</u>	<u>Minimum Life Hours</u>
2400	30	20

Chemical analyses of stress-rupture specimens after test shall demonstrate that the degree of environmental contamination did not exceed the following limits: total increase in oxygen plus nitrogen content--less than 50 ppm; increase in hydrogen content--less than 5 ppm; increase in carbon content--less than 10 ppm. The following limits shall apply to check analyses of analytical results:

Carbon	±	10 ppm
Oxygen	±	25 ppm
Nitrogen	±	25 ppm
Hydrogen	±	2 ppm

3.7.3. Hydrostatic Test. Each tube, 1/8 inch or larger in outside diameter with a wall thickness of 0.015 inch or over, shall be tested to a hydrostatic pressure sufficient to produce a fiber stress of 12,000 psi. The test pressure not to exceed 10,000 psi, shall be determined by the equation ($P = 2St/D$), where:

P = hydrostatic test pressure in pounds per square inch;

S = 12,000 psi;

t = average wall thickness of the tube in inches;

D = outside diameter of the tube in inches.

3.7.4. Flare Test. A section of the heat treated tube shall be capable of being flared without cracking. The flare shall be made with a tool having a 60-degree included angle until the specified outside diameter has been increased by 15%.

3.8. Tolerances

3.8.1. Diameter and Wall Thickness. The permissible variations in diameter and wall thickness of tube shall not exceed those prescribed in Table II (refer to page 8).

3.8.2. Length. When tube is ordered cut-to-length, the usable length shall not be less than that specified, but a variation of plus 1/8 inch will be permitted in lengths up to 6 feet. In lengths over 6 feet, a variation of plus 1/4 inch will be permitted, unless otherwise specified.

TABLE II
PERMISSIBLE VARIATIONS IN TUBE DIMENSIONS

<u>Nominal OD</u> <u>Inches</u>	<u>OD</u> <u>Inch</u>	<u>ID</u> <u>Inch</u>	<u>Wall</u> <u>Thickness</u> <u>%</u>
0.187 to but not incl. 0.625	± 0.004	± 0.004	± 10
0.625 to but not incl. 1.000	± 0.005	± 0.005	± 10
1.000 to but not incl. 2.000	± 0.0075	± 0.0075	± 10
2.000 to but not incl. 3.000	± 0.010	± 0.010	± 10
3.000 to but not incl. 4.000	± 0.0125	± 0.0125	± 10

NOTES: -----

- (1) Tolerances are applicable to only the two dimensions specified on the purchase order, e.g., outside diameter and wall; inside diameter and wall; outside diameter and inside diameter.
- (2) For tolerances applicable for very small tubes (less than 0.187-inch diameter) or very thin-wall tubes (less than 0.010-inch thick), the producer shall be consulted.
- (3) For tubes having an inside diameter less than 60% of the outside diameter or a wall 3/4 inch or over thick, which cannot be successfully drawn over a mandrel, the inside diameter may vary by an amount equal to plus or minus 10% of the wall thickness. The wall thickness of these tubes may vary plus or minus 12.5% from that specified.
- (4) Ovality measured at any cross section: For tubes with nominal wall thickness less than 3% of the nominal outside diameter, the ovality tolerances are double the tolerances in column 2 or 3. For ovality tolerances for tubes with wall thickness less than 2% nominal outside diameter, the producer shall be consulted.

3.8.3. Straightness. The tube shall be free of bends or kinks. For lengths up to 10 feet, the maximum bow shall not exceed one part in 1200; for lengths greater than 10 feet, the maximum bow shall not exceed one part in 600, unless otherwise agree upon.

3.9. Reports. The manufacturer shall supply at least three copies of a report showing non-proprietary manufacturing methods, processing conditions and test procedures and results for each lot of material in the shipment. The report shall also include the number of the specification and the purchase order or contract number.

4. MAXIMUM ALLOWABLE DISCONTINUITIES

4.1. General. Cracks, laps, seams, fins and tears shall be unacceptable. The surface shall also be free from oxide or scale of any nature, grease, oil, residual lubricants, or other extraneous material.

4.2. Porosity and Inclusions. Indications with dimensions greater than 3% of the wall thickness shall be unacceptable. Indications with dimensions in the range of 1% to 3% of wall thickness must be a minimum of 0.50 inch apart. Indications with dimensions less than 1% of the wall thickness must be a minimum of 0.12 inch apart.

4.3. Surface Rework. Defects less than 3% of the nominal wall thickness detected by penetrant or ultrasonic inspection may be removed by grinding, provided the wall thickness is not decreased below that permitted in Table II (refer to page 8).

5. QUALITY ASSURANCE PROVISIONS

5.1. Vendor Responsibility. The manufacturer shall make all test and inspection of the material covered by this specification before shipment unless otherwise specified. All test and inspection results shall be furnished to the purchaser.

5.2. Customer Review. The purchaser or his representative may witness the testing and inspection of the material. The manufacturer shall give the purchaser ample notice of the time and place of designated tests. If the purchaser's representative is not present at this time and a new date is not set, the requirement for purchaser's inspection at the place of testing is waived. When the purchaser's representative is present at the appointed time and place, the manufacturer shall afford him, without charge, all reasonable facilities to assure that the material is

being furnished in accordance with this specification. The inspection shall not interfere unnecessarily with production operations.

5.3. Sample Selection. Care shall be exercised to insure that the sample selected for testing is representative of the material and uncontaminated by the sampling procedure. If there is any question about the sampling technique or the analysis, the methods for sampling and analysis shall be those agreed to by the buyer and seller. The specimen configuration selected for the performance of the testing required in paragraphs 5.4.2. and 5.4.3. shall be mutually agreed upon by the vendor and purchaser prior to placement of a purchase order. The location of all test samples shall be reported in the certificate of compliance.

5.4. Test Methods

5.4.1. Chemical Analysis. Chemical analyses shall be conducted by mutually acceptable procedures, such as the vacuum fusion methods for gases, the combustion method for carbon, and the spectrochemical methods for metallic elements.

5.4.2. Tensile Test. The tension test shall be performed in accordance with ASTM Designation E8-57T, "Methods of Tension Testing of Metallic Materials." Yield strength shall be determined by the offset (0.2%) method. The tensile properties shall be determined using a strain rate of 0.005 inch per inch per minute up to 0.6% offset and then 0.050 inch, plus or minus 0.020 inch, per inch per minute to fracture.

5.4.3. Stress-Rupture Test. Stress-rupture properties of specimens shall be determined by mutually acceptable testing techniques. Suggested testing techniques for determining stress-rupture properties are:

Specimens shall be tested in a vacuum of 1×10^{-6} torr or better. The vacuum system shall incorporate an optically tight liquid nitrogen cold trap or a getter-ion pump.

Specimens shall be held for a half hour at the test temperature before application of load.

Test temperature shall be maintained at plus or minus 10°F during the test.

5.4.4. Grain Size. Grain size determinations shall be made according to ASTM Specification E112-61, "Estimating the Average Grain Size of Metals."

5.5. Number of Tests Required. Representative test specimens from the finished product representing each ingot and each lot of material shall be taken to determine conformity to this specification. The minimum frequency of these tests shall be:

Finished Product Chemistry - one per lot per ingot

Tensile Test - two per lot per ingot

Stress-Rupture Test - two per lot per ingot

Flare Test - two per lot per ingot

Grain Size - two per lot per ingot

Microhardness Traverse - one per lot per ingot

Hydrostatic Proof Test - 100%

5.6. Retest and Rework

5.6.1. Surface Contamination. Any sample or specimen exhibiting obvious surface contamination or improper preparation which disqualifies it as a truly representative sample shall be replaced with a new sample.

5.6.2. Rework. If inspection and test results of a lot do not conform to the requirements of this specification, the lot may be reworked at the option of the manufacturer. The lot shall be acceptable if all test results, after reworking, conform to this specification.

5.7. Inspection

5.7.1. Methods of Inspection

5.7.1.1. Radiographic. When specified, the product shall be radiographed and found free of porosity and inclusions as specified in paragraph 4.2 using the techniques described in AMS 2635, "Radiographic Inspection." The radiographs and product shall be identified so that the exact position of each radiograph can be correlated with the specific area on a particular product.

5.7.1.2. Ultrasonic Inspection. Unless otherwise agreed to by the purchaser and the vendor, the material shall be inspected ultrasonically.

5.7.1.2.1. Method and Equipment. Ultrasonic inspection shall be by the immersed technique at 5 mc or higher frequency using focused transducers. Inspection shall be by both circumferential and axial shear techniques with longitudinal wave being added when the wall thickness is greater than 0.150 inch. For longitudinal wave technique and for circumferential shear, transducers up to 2 inches long may be used with or without automatic equipment to rotate the tube past the transducer. If spiral pattern inspection traverse is not used, steps must be taken to assure that the ultrasonic beam remains in the same position relative to the tubing so the beam-to-tubing angle remains constant. For axial (longitudinal) shear, transducers must have no greater than 0.5 inch axial length. Transducers must be cylindrically focused for a diameter range which includes the tubing on which it is to be used.

5.7.1.2.2. Calibration. Calibration shall be on notches (a total of four, two axial and two circumferential), cut in the tube on both the outside and inside surface unless otherwise specified. The depth of the notches shall be 3% of the wall thickness to a minimum depth of 0.001 inch; the width, no greater than depth; the length, at least that of the ultrasonic beam with a maximum length of 1 inch. Material having a wall thickness greater than 0.150 inch shall also have an 0.020-inch diameter hole machined into the wall in the longitudinal direction at mid-point of the wall thickness. Focusing shall be done to maximize the indication from the inside diameter notch placed properly for the type of inspection contemplated. After focusing is completed, the inside diameter indication shall be set at 80% and gain setting recorded. Gain setting for 80% on the outside diameter notch shall also be recorded. Inspection shall be at the gain setting for the inside diameter indication. A distance corresponding to the wall thickness shall be marked on the oscilloscope. Focal distance to the part to be inspected shall be set to that used for the calibration piece before beginning inspection. Calibration shall be done both before and after the inspection or at the beginning and end of each work shift. If calibration has changed (gain change greater than 5%), all inspections since the previous calibration shall be repeated.

5.7.1.2.3. Rejection. Rejection shall be by any indication which exceeds the amplitude of the respective calibration indication; i.e., inside diameter defects shall be compared to the indication from the notch on the inside diameter, and outside diameter defects shall be compared

to the indication from the notch on the outside diameter. Defects less than half the thickness from the surface or less than 0.150 inch from the surface, whichever is smaller, shall be compared to the outside diameter calibration indication. Defects more than half the thickness from the incident surface or more than 0.150 inch from the surface shall be compared to the indications from the inside diameter calibration notch.

5.7.1.2.4. Reports. The ultrasonic inspection report shall contain the equipment serial numbers, calibration amplitudes and gain settings and the amplitude and location of each defect whose amplitude is 60% or greater.

5.7.1.3. Penetrant Inspection. The exterior surface of the product shall be penetrant inspected and found free of flaws as specified in paragraph 4.3 using AMS 2645, "Fluorescent Penetrant Inspection," or AMS 2646, "Contrast Dye Penetrant Inspection." All parts thus inspected shall be marked with ink stamps as described in the specification; impression stampings or etching shall be unacceptable.

5.7.1.4. Reports. The manufacturer shall supply at least three copies of a report showing inspection results for each lot of material in the shipment. The report shall also include the number of the specification and the purchase order or contract number.

5.8. Rejection. Material not conforming to this specification or to any authorized modification shall be subject to rejection. Unless otherwise specified, rejected material may be returned to the manufacturer at the manufacturer's expense if the purchaser does not receive other instructions for disposition within three weeks after notice of rejection.

5.9. Referee. If the manufacturer and the purchaser disagree concerning the conformance of the material to the requirements of this specification or any special test specified by the purchaser, a mutually acceptable referee's test shall be used to determine conformance.

6. PREPARATION FOR DELIVERY

6.1. Identification. Each bundle, box, or carton shall be legibly and conspicuously marked or tagged with the number of this specification, purchase order or contract number, type, ingot number, lot number, nominal size, and the gross, net and tare weights. When each bundle

box or carton consists of components from more than one ingot number or lot number, each component shall be identified individually.

6.2. Packing. The ends of each pipe or tube shall be sealed with suitable plastic caps and each individual item shall be wrapped in heavy gauge polyethylene or similar material and packed in a manner assuring safe delivery when properly transported by a common carrier.

7. DEFINITIONS

7.1. Lot. A lot shall include all material of the same size, shape, condition and finish from one heat of material and which has received the same processing, has been annealed in the same vacuum annealing charge and has been processed simultaneously in all operations in which temperatures may reach 500°F or above. When process temperatures and environments are closely controlled or when closely adjacent sizes receive similar processing, lots may be combined for chemical, tensile and stress-rupture tests only, provided prior written approval has been obtained from the General Electric Company.

7.2. Check Analysis. An analysis may be requested by the purchaser of the metal, after it has been processed into finished mill forms, for the purpose of verifying the composition within a heat or lot. Check analysis tolerances do not broaden the specified heat analysis requirements but rather cover variations between laboratories in the measurement of the chemical content.

7.3. Significance of Numerical Limits. For determining compliance with the specified limits for requirements of the properties listed below, an observed value or a calculated value shall be rounded off using the rounding-off method in ASTM Designation E29-58T, "Recommended Practices for Designating Significant Places in Specified Limiting Values."

<u>Test</u>	<u>Rounded-Off Unit for Observed or Calculated Value</u>
Chemical composition and dimensional tolerances (when expressed decimally)	Nearest unit in the last right-hand place of figures of the specified limit
Tensile strength	Nearest 100 psi
Elongation	Nearest 1%
Rupture life	Nearest 0.1 hour

SPECIFICATION

FOIL: T-222 (Ta-10.4W-2.4Hf-0.01C) ALLOY

prepared for
NATIONAL AERONAUTICS AND SPACE ADMINISTRATION
Contract NAS 3-2547

SPACE POWER AND PROPULSION SECTION
MISSILE AND SPACE DIVISION
GENERAL ELECTRIC COMPANY
CINCINNATI, OHIO 45215

SPECIFICATION

FOIL: T-222 (Ta-10.4W-2.4Hf-0.01C) ALLOY

1. SCOPE

1.1. Scope. This specification covers T-222 (Ta-10.4W-2.4Hf-0.01C) alloy in foil form for high temperature non-structural applications.

2. APPLICABLE DOCUMENTS

2.1. Government Documents. None

2.2. Non-Government Documents. None

3. REQUIREMENTS

3.1. Acknowledgments. The vendor shall mention this specification in all quotations and all purchase order acknowledgments.

3.2. Manufacture. Material covered by this specification shall be made from ingot which have been double vacuum melted by the electron beam and/or consumable electrode arc melting processes. Breakdown operations shall be performed with conventional extrusion, forging and rolling equipment normally found in primary ferrous and nonferrous plants.

3.3. Processing. The starting stock size, processing temperatures, percentages of reduction, in-process annealing temperatures and times shall be selected by the vendor.

3.4. Condition. The finished product shall be supplied in the fully recrystallized condition throughout the cross-sectional area. All annealing shall be carried out in a vacuum of less than 1×10^{-5} torr. All mill products to be annealed shall be thoroughly degreased, chemically cleaned and protected from furnace parts by a layer of fresh tantalum, columbium or Cb-1Zr alloy foil 0.002 inch thick or greater. When annealing is carried out in a vacuum greater than 1×10^{-5} torr, with the prior approval of the purchaser, all mill products shall be enclosed

in a chemically cleaned tantalum, columbium or Cb-1Zr alloy retort or wrapped in a minimum of two layers of fresh tantalum, columbium or Cb-1Zr alloy foil 0.002-inch thick or greater. The conditions of final annealing shall be reported in the certificate of compliance.

3.5. Chemical Composition

3.5.1. Ingot/Billet Composition. The chemical composition of ingots and billets for conversion to finished products shall conform to Table I (page 4). A minimum of four analyses shall be obtained as follows: ingot top-center, mid-radius and edge, and ingot bottom-center; all analyses must conform to ranges stated in Table I.

3.5.2. Final Product Composition. The manufacturer's ingot analyses shall be considered the chemical analysis for products supplied under this specification (Table I) except carbon, oxygen, nitrogen and hydrogen content which shall be determined in the finished product.

3.5.3. Check Analysis. Finished product analysis shall not exceed the following limits or variations:

<u>Element</u>	<u>Check Analysis Limits, Max., ppm</u>	<u>Permissible Variations in Check Analysis, ppm</u>
Carbon	80 min; 175 max	± 10
Oxygen	300	+ 20
Nitrogen	100	+ 10
Hydrogen	10	+ 2

3.6. Bend Ductility. Representative samples of the materials in final form shall withstand a 180° bend without failure.

3.7. Tolerances

3.7.1. Definition. Foil includes material less than 12 inches wide and up to and including 0.010-inch thick.

3.7.2. Dimensions. Foil dimensions shall conform to the following limits:

TABLE I
CHEMICAL COMPOSITION

T-222 (Ta-10.4W-2.4Hf-0.01C) ALLOY

<u>Element</u>	<u>Minimum Content</u> <u>ppm</u>	<u>Maximum Content</u> <u>ppm</u>
Carbon	80	175
Nitrogen	-	50
Oxygen	-	100
Hydrogen	-	10
Columbium	-	1000
Molybdenum	-	200
Nickel	-	50
Cobalt	-	50
Iron	-	50
Vanadium	-	20
Tungsten	9.6 w/o	11.2 w/o
Hafnium	2.2 w/o	2.8 w/o
Tantalum	Remainder	-

<u>Material Thickness</u> <u>Inch</u>	<u>Thickness Tolerances</u> <u>Inch</u>	<u>Width Tolerances</u> <u>Inch</u>
Less than 0.003	+0.0008, -0.0000	+0.031, -0.000
0.003 to 0.005	± 0.001	+0.031, -0.000
0.005 to 0.010	± 0.0015	+0.031, -0.000

3.8. General. The finished product shall be visilby free from oxide or scale of any nature, grease, oil, residual lubricants, and other extraneous materials. Cracks, laps, seams, gouges, and fins shall be unacceptable.

4. QUALITY ASSURANCE PROVISIONS

4.1. Vendor Responsibility. The manufacturer shall make all tests and inspections of the material covered by this specification before shipment, unless otherwise specified. All test and inspection results shall be furnished to the purchaser.

4.2. Sample Selection. Care shall be exercised to insure that the samples selected for testing and chemical analyses are representative of the material and uncontaminated by the sampling procedure. If there is any question about the sampling technique or the analysis, the methods for sampling and analysis shall be those agreed to by the buyer and seller.

4.3. Chemical Analysis. Chemical analyses shall be conducted by mutually acceptable procedures, such as the vacuum fusion methods for gases, the combustion method for carbon, and the spectrochemical methods for metallic elements.

4.4. Number of Tests Required. Representative test specimens from the finished product representing each ingot and each lot of material shall be taken to determine conformity to this specification. The minimum frequency of these tests shall be:

Finished Product Chemistry - one per lot per ingot

Bend Test - two per lot per ingot

4.5. Reports. The manufacturer shall supply at least three copies of a report showing non-proprietary manufacturing methods, processing conditions, and test procedures and results for each lot of material in the shipment. The report shall also include the number of the specification and the purchase order or contract number.

4.6. Rejection. Material not conforming to this specification or to any authorized modification shall be subject to rejection. Unless otherwise specified, rejected material may be returned to the manufacturer at the manufacturer's expense if the purchaser does not receive other instructions for disposition within three weeks after notice of rejection.

4.7. Referee. If the manufacturer and the purchaser disagree about the conformance of the material to the requirements of this specification or any special test specified by the purchaser, a mutually acceptable referee's test shall be used to determine conformance.

5. PREPARATION FOR DELIVERY

5.1. Identification. Each bundle, box or carton shall be legibly and conspicuously marked or tagged with the number of this specification, purchase order or contract number, type, ingot number, lot number, nominal size, and the gross, net, and tare weights. When each bundle, box or carton consists of components from more than one ingot number or lot number, each component shall be identified individually.

5.2. Packing. Each individual item shall be wrapped in heavy gauge polyethylene film or other similar material and packed in a manner assuring safe delivery when properly transported by any common carrier.

6. DEFINITIONS

6.1. Lot. A lot shall include all material of the same size, shape, condition and finish from one heat of material and which has received the same processing, has been annealed in the same vacuum annealing charge and has been processed simultaneously in all operations in which temperatures may reach 500°F or above. When process temperatures and environments are closely controlled or when closely adjacent sizes receive similar processing, lots may be combined for chemical, tensile and stress-rupture tests only, provided prior written approval has been obtained from the General Electric Company.

6.2. Check Analysis. An analysis, made or requested by the purchaser, of the metal after it has been processed into finished mill forms to verify the composition within a heat or lot. Check analysis tolerances do not broaden the specified heat analysis requirements but rather cover variations between laboratories in the measurement of the chemical content.

01-0047-00-B
SPPS-67-R1
12 April 1965
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SPECIFICATION

WIRE: T-222 (Ta-10.4W-2.4Hf-0.001C) ALLOY

prepared for
NATIONAL AERONAUTICS AND SPACE ADMINISTRATION
Contract NAS 3-2547

SPACE POWER AND PROPULSION SECTION
MISSILE AND SPACE DIVISION
GENERAL ELECTRIC COMPANY
CINCINNATI, OHIO 45215

SPECIFICATION

WIRE: T-222 (Ta-10.4W-2.4Hf-0.001C) ALLOY

1. SCOPE

1.1. Scope. This specification covers T-222 (Ta-10.4W-2.4Hf-0.01C) alloy in wire form for use as weld filler material in fabricating components intended for high temperature structural applications and alkali metal containment.

2. APPLICABLE DOCUMENTS

2.1. Government Documents. None

2.2. Non-Government Documents. None

3. REQUIREMENTS

3.1. Acknowledgments. The vendor shall mention this specification in all quotations and all purchase order acknowledgments.

3.2. Manufacture. Material covered by this specification shall be made from ingots which have been double vacuum melted by the electron beam and/or consumable electrode arc melting processes. Breakdown operations shall be performed with conventional extrusion, forging and rolling equipment found in primary ferrous and nonferrous plants.

3.3 Processing. The starting stock size, processing temperatures, percentages of reduction, in-process annealing temperatures and times shall be selected by the vendor.

3.4. Condition. The finished product shall be supplied in the fully recrystallized condition throughout the cross-sectional area. Annealing shall be carried out in a vacuum of less than 1×10^{-5} torr. All mill products to be annealed shall be thoroughly degreased, chemically cleaned and protected from furnace parts by a layer of fresh tantalum, columbium or Cb-1Zr alloy foil 0.002-inch thick or greater. When

annealing is carried out in a vacuum greater than 1×10^{-5} torr, with the prior approval of the purchaser, all mill products shall be enclosed in a chemically cleaned tantalum, columbium or Cb-1Zr alloy re-tort or wrapped in a minimum of two layers of fresh tantalum, columbium or Cb-1Zr alloy foil 0.002-inch thick or greater. The conditions of final annealing shall be reported in the certificate of compliance.

3.5. Chemical Composition

3.5.1. Ingot/Billet Composition. The chemical composition of ingots and billets for conversion to finished products shall conform to Table I (page 4). A minimum of four analyses shall be obtained as follows: ingot top-center, mid-radius and edge, and ingot bottom-center; all analyses must conform to ranges stated in Table I.

3.5.2. Final Product Composition. The manufacturer's ingot analyses shall be considered the chemical analysis for products supplied under this specification (Table I) except carbon, oxygen, nitrogen and hydrogen content which shall be determined in the finished product.

3.5.3. Check Analysis. Finished product analysis shall not exceed the following limits or variations:

For Material Greater than 0.030 Inch in Diameter

<u>Element</u>	<u>Check Analysis Limits, Max., ppm</u>	<u>Permissible Variations in Check Analysis, ppm</u>
Carbon	80 min; 175 max	± 10
Oxygen	150	+ 20
Nitrogen	75	+ 10
Hydrogen	10	+ 2

For Material 0.030 Inch and Less in Diameter

<u>Element</u>	<u>Check Analysis Limits, Max., ppm</u>	<u>Permissible Variations in Check Analysis, ppm</u>
Carbon	80 min; 175 max	± 10
Oxygen	300	+ 20
Nitrogen	100	+ 10
Hydrogen	10	+ 2

TABLE I
CHEMICAL COMPOSITION
T-222 (Ta-10.4W-2.4Hf-0.01C) ALLOY

<u>Element</u>	<u>Minimum Content</u> <u>ppm</u>	<u>Maximum Content</u> <u>ppm</u>
Carbon	80	175
Nitrogen	-	50
Oxygen	-	100
Hydrogen	-	10
Columbium	-	1000
Molybdenum	-	200
Nickel	-	50
Cobalt	-	50
Iron	-	50
Vanadium	-	20
Tungsten	9.6 w/o	11.2 w/o
Hafnium	2.2 w/o	2.8 w/o
Tantalum	Remainder	-

3.6. Tolerances

3.6.1. Definition. Wire - material less than 0.125 inch in diameter.

3.6.2. Diameter. The permissible variation in diameter shall not exceed the following limits:

<u>Diameter, Inch</u>	<u>Diameter Variation, Inch</u>
0.005 to 0.009	± 0.0002
0.010 to 0.019	± 0.0003
0.020 to 0.029	± 0.0005
0.030 to 0.061	± 0.001
0.062 to 0.125	± 0.002

3.7. General. The finished product shall be visibly free from oxide or scale of any nature, grease, oil, residual lubricants, and other extraneous materials. Cracks, laps, seams, gouges, and fins shall be unacceptable.

4. QUALITY ASSURANCE PROVISIONS

4.1. Vendor Responsibility. The manufacturer shall make all tests and inspections of the material covered by this specification before shipment, unless otherwise specified. All test and inspection results shall be furnished to the purchaser.

4.2. Sample Selection. Care shall be exercised to insure that the samples selected for testing and chemical analyses are representative of the material and uncontaminated by the sampling procedure. If there is any question about the sampling technique or the analysis, the methods for sampling and analysis shall be those agreed to by the buyer and seller.

4.3. Chemical Analysis. Chemical analyses shall be conducted by mutually acceptable procedures, such as the vacuum fusion methods for gases, the combustion method for carbon, and the spectrochemical methods for metallic elements.

4.4. Number of Tests Required. Representative test specimens from the finished product representing each ingot and each lot of material shall be taken to determine conformity to this specification. The minimum frequency of these tests shall be:

Finished Product Chemistry - one per lot per ingot

4.5. Reports. The manufacturer shall supply at least three copies of a report showing non-proprietary manufacturing methods, processing conditions, and test procedures and results for each lot of material in the shipment. The report shall also include the number of the specification and the purchase order or contract number.

4.6. Rejection. Material not conforming to this specification or to any authorized modification shall be subject to rejection. Unless otherwise specified, rejected material may be returned to the manufacturer at the manufacturer's expense if the purchaser does not receive other instructions for disposition within three weeks after notice of rejection.

4.7. Referee. If the manufacturer and the purchaser disagree about the conformance of the material to the requirements of this specification or any special test specified by the purchaser, a mutually acceptable referee's test shall be used to determine conformance.

5. PREPARATION FOR DELIVERY

5.1. Identification. Each bundle, box, or carton shall be legibly and conspicuously marked or tagged with the number of this specification, purchase order or contract number, type, ingot number, lot number, nominal size, and the gross, net, and tare weights. When each bundle, box or carton consists of components from more than one ingot number or lot number, each component shall be identified individually.

5.2. Packing. Each individual item shall be wrapped in heavy gauge polyethylene film or similar material and packed in a manner assuring safe delivery when properly transported by any common carrier.

6. DEFINITIONS

6.1. Lot. A lot shall include all material of the same size, shape, condition and finish from one heat of material and which has received the same processing, has been annealed in the same vacuum annealing charge and has been processed simultaneously in all operations in which

temperatures may reach 500°F or above. When process temperatures and environments are closely controlled or when closely adjacent sizes receive similar processing, lots may be combined for chemical, tensile and stress-rupture tests only, provided prior written approval has been obtained from the General Electric Company.

6.2. Check Analysis. An analysis, made or requested by the purchaser of the metal after it has been processed into finished mill forms, to verify the composition with a heat or lot. Check analysis tolerances do not broaden the specified heat analysis requirements but rather cover variations between laboratories in the measurement of the chemical content.

01-0010-00-A
SPPS-16-R1
23 December 1965
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SPECIFICATION

BAR AND ROD: Cb-132M (Cb-20Ta-15W-5Mo-2Zr-.13C) ALLOY

prepared for
NATIONAL AERONAUTICS AND SPACE ADMINISTRATION
Contract NAS 3-2547

SPACE POWER AND PROPULSION SECTION
MISSILE AND SPACE DIVISION
GENERAL ELECTRIC COMPANY
CINCINNATI, OHIO 45215

SPECIFICATION

BAR AND ROD: Cb-132M (Cb-20Ta-15W-5Mo-2Zr-.13C) ALLOY

1. SCOPE

1.1. Scope. This specification covers Cb-132M (Cb-20Ta-15W-5Mo-2Zr-.13C) alloy in bar and rod form intended for high temperature structural applications in alkali metal systems.

1.2. <u>Classes.</u>	SPPS-16A	Recrystallized
	SPPS-16B	Stress-Relieved

2. APPLICABLE DOCUMENTS

2.1. Government Documents. None

2.2. Non-Government Documents

ASTM Designation E8-57T (26 December 1957)	Method of Tension Testing of Metallic Materials
ASTM Designation E29-58T (1958)	Recommended Practices for Des- ignating Significant Places in Specified Limiting Values
ASTM Designation (Pending)	Methods for Chemical Analysis of Reactor and Commercial Columbium
ASTM E112-61 (1961)	Estimating Average Grain Size of Metals
AMS 2635 (15 August 1958)	Radiographic Inspection
AMS 2645 (1 March 1955)	Fluorescent Penetrant Inpsec- tion
AMS 2646 (1 March 1955)	Contrast Dye Penetrant Inspec- tion

3. REQUIREMENTS

3.1. Acknowledgments. The vendor shall mention this specification in all quotations and all purchase order acknowledgments.

3.2. Manufacture. Material covered by this specification shall be made from ingots which have been double vacuum melted by the electron beam and/or consumable electrode arc melting processes. Breakdown operations shall be performed with conventional extrusion, forging and rolling equipment normally found in primary ferrous and nonferrous plants.

3.3. Processing. The starting stock size, processing temperatures, percentages of reduction and in-process and final annealing, aging or stress-relieving temperatures and times shall be selected by the vendor to achieve the structure or grain size range specified in paragraph 3.6 and mechanical properties specified in paragraph 3.7. The amount of total reduction from the turned ingot to the final products of Classes A and B shall exceed 75%; the amount of final reduction imparted to mill products of Class B since the last Rx anneal and prior to final stress-relief heat treatment shall not exceed 80%. The amount of final reduction for each mill product, imparted just prior to the final vacuum heat treatment and the total reduction since the previous recrystallization anneal shall be reported in the certificate of compliance.

3.4. Condition

3.4.1. Class A. The finished product shall be supplied in the recrystallized condition throughout the cross sectional area to the grain size range specified in paragraph 3.6.

3.4.2. Class B. The finished product shall be supplied in the stress-relieved condition throughout the cross sectional area as specified in paragraph 3.6.

3.4.3. Heat Treating. All heat treating shall be carried out in a vacuum less than 1×10^{-5} torr. All mill products to be heat treated shall be thoroughly degreased, chemically cleaned and protected from furnace parts by a layer of fresh tantalum, columbium or Cb-1Zr alloy foil, 0.002-inch thick or greater. When annealing is carried out in a vacuum greater than 1×10^{-5} torr, with the prior approval of the purchaser, all mill products shall be enclosed in a chemically cleaned columbium, tantalum or Cb-1Zr alloy retort or wrapped in a minimum of

two layers of fresh tantalum, columbium or Cb-1Zr alloy foil 0.002-inch thick or greater. The conditions of final heat treating shall be reported in the certificate of compliance.

3.4.4. Contamination. All items are to be free of contamination or internal oxidation. After final heat treatment, the material shall be examined metallographically for evidence of possible contamination caused by unsatisfactory heat treating atmospheres or processing conditions. A microhardness traverse shall show a hardness increase not greater than 50 VHN from the center to the surface of a cross sectional sample of the final product. At the discretion of the purchaser, samples taken to include at least one surface of the final product, and not exceeding 0.050-inch thick, may be chemically analyzed by the purchaser for oxygen, nitrogen, hydrogen and carbon. The analyses shall not exceed the limits set forth in paragraph 3.5.3. Any indication of contamination shall be cause for rejection of all material represented by that sample. The material shall be acceptable if the contaminated layer is completely eliminated before shipment by a machining operation within the specified dimensions and tolerances.

3.5. Chemical Composition

3.5.1. Ingot/Billet Composition. The chemical composition of ingots and billets for conversion to finished products shall conform to Table I.(page 5). A minimum of four analyses shall be obtained as follows: ingot top-center, mid-radius and edge, and ingot bottom-center; all analyses must conform to ranges stated in Table I.

3.5.2. Final Product Composition. The manufacturer's ingot analyses shall be considered the chemical analyses of products supplied under this specification (Table I) except carbon, oxygen, nitrogen and hydrogen content which shall be determined on the finished product.

3.5.3. Check Analysis. Finished product analysis shall not exceed the following limits or variations:

<u>Element</u>	<u>Check Analysis Limits, Max., ppm</u>	<u>Permissible Variations in Check Analysis, ppm</u>
Carbon	1100 min.; 1500 max.	± 50
Oxygen	200	+ 20
Nitrogen	100	+ 10
Hydrogen	5	+ 2

TABLE I
CHEMICAL COMPOSITION
Cb-132M (Cb-20Ta-15W-5Mo-2Zr-.13C) ALLOY

<u>Element</u>	<u>Minimum Content</u> <u>ppm</u>	<u>Maximum Content</u> <u>ppm</u>
Carbon	1100	1500
Nitrogen	-	50
Oxygen	-	100
Hydrogen	-	5
Nickel	-	50
Cobalt	-	50
Iron	-	50
Tantalum	18.5 w/o	21.5 w/o
Tungsten	13.5 w/o	16.5 w/o
Molybdenum	4.5 w/o	5.5 w/o
Zirconium	1.75 w/o	2.25 w/o
Columbium	Remainder	-

3.6. Structure

3.6.1. Class A. The grain size of the final product shall conform to the following limits:

<u>Product Diameter or Thickness, Inches</u>	<u>Minimum Allowable ASTM Grain Size No.</u>	<u>Allowable Spread in ASTM Grain Size Nos. in Any One Item</u>	<u>% R_x Minimum</u>
0.125 to 0.250	4	2	90
Over 0.250 to 0.500	4	2	90
Over 0.500 to 1.0	4	2	90
Over 1.0 to 2.0	4	2	90
Greater than 2.0	3	3	90

3.6.2. Class B. The grain size of the final product shall conform to the following limits:

<u>Product Diameter or Thickness, Inches</u>	<u>Minimum Allowable Equivalent ASTM Grain Size No. Perpendicular to Direction of Work</u>	<u>% R_x Maximum</u>
0.125 to 0.250	4	5
Over 0.250 to 0.500	4	5
Over 0.500 to 1.0	4	5
Over 1.0 to 2.0	4	5
Greater than 2.0	3	5

3.7. Mechanical Properties. The final product shall satisfy the following mechanical property requirements:

3.7.1. Room Temperature Tensile Properties (Tentative). Representative samples of the material in final form shall be capable of the following property limits at room temperature (65°-86°F).

<u>Class</u>	<u>Ultimate Tensile Strength, ksi</u>		<u>0.2% Yield Strength, ksi</u>		<u>Elong., % in 4D</u>
	<u>Minimum</u>	<u>Maximum</u>	<u>Minimum</u>	<u>Maximum</u>	<u>Minimum</u>
A	90	125	80	110	7
B*	140	190	115	160	20

* Longitudinal to the Direction of Work.

3.7.2. Stress-to-Rupture Tests (Tentative). The material shall be capable of achieving the following stress-rupture life under suitable environmental conditions (see paragraph 5.4.3).

<u>Class</u>	<u>Test Temp. °F</u>	<u>Stress, ksi</u>	<u>Minimum Life Hours</u>
A	2200	40	20
B*	2200	30	20

* Longitudinal to the Direction of Work.

Chemical analysis of stress-rupture specimens after test shall demonstrate that the degree of environmental contamination did not exceed the following limits: total increase in oxygen plus nitrogen content--less than 100 ppm; increase in hydrogen content--less than 5 ppm; increase in carbon content--less than 10 ppm. The following limits shall apply to the analytical results:

Carbon	±	10 ppm
Oxygen	±	50 ppm
Nitrogen	±	50 ppm
Hydrogen	±	2 ppm

3.8. Tolerances

3.8.1. Rolled, Swaged or Drawn Rounds

3.8.1.1. Definition. Rod - 3.5 inches in diameter or less.

3.8.1.2. Diameter. The permissible variation in diameter and the limits of out-of-roundness of descaled rounds shall not exceed those in Table II (refer to page 9).

3.8.1.3. Cut Lengths. Maximum length variation shall be 0.25 inch.

3.8.1.4. Straightness. Maximum deviation shall be 0.050 inch per foot in any length.

3.8.2. Square or Rectangular Bar

3.8.2.1. Definition. Bar - any straight product with a rectangular cross-section 0.187 inch or more thick and less than 5 inches wide.

3.8.2.2. Dimensions. Unless otherwise specified, forged or rolled square and rectangular shapes shall have the following tolerances:

<u>Thickness</u>	<u>Length</u>	<u>Width</u>
± 0.025 Inch or $\pm 5\%$, whichever is less	± 0.125 Inch	± 0.125 Inch

3.8.2.3. Straightness of Bar. Maximum deviation shall be 0.050 inch per foot in any length.

3.9. Reports. The manufacturer shall supply at least three copies of a report showing non-proprietary manufacturing methods, processing conditions, and test procedures and results for each lot of material in the shipment. The report shall also include the number of the specification and the purchase order or contract number.

4. MAXIMUM ALLOWABLE DISCONTINUITIES

4.1. General. The finished product shall be visibly free from oxide or scale of any nature, grease, oil, residual lubricants, and other extraneous materials. Cracks, laps, seams, gouges, and fins shall be unacceptable.

TABLE II
PERMISSIBLE DIMENSIONAL VARIATIONS FOR ROUND BAR

<u>Diameter</u> <u>Inches</u>	<u>Diameter</u> <u>Variation</u> <u>Inch</u>	<u>Out-of-Roundness</u> <u>Inch</u>
0.125 to 0.281	+ 0.002, -0.002	0.004
Over 0.281 to 0.406	+ 0.010, -0.005	0.008
Over 0.406 to 0.625	+ 0.010, -0.005	0.012
Over 0.625 to 0.875	+ 0.015, -0.005	0.015
Over 0.875 to 1.000	+ 0.020, -0.005	0.015
Over 1.000 to 1.375	+ 0.020, -0.010	0.018
Over 1.375 to 1.500	+ 0.020, -0.015	0.020
Over 1.500 to 1.625	+ 0.025, -0.015	0.020
Over 1.625 to 2.000	+ 0.030, -0.030	0.025
Over 2.000 to 2.500	+ 0.032, -0.032	0.025
Over 2.500 to 3.250	+ 0.032, -0.032	0.027
Over 3.250 to 3.500	+ 0.045, -0.045	0.040

Centerless Ground Rounds

0.062 to 2.00	+ 0.002, -0.002
Over 2.00	+ 0.003, -0.002

4.2. Porosity and Inclusions. Indications of internal porosity and non-metallic inclusions greater than 0.020 inch or 3% of the thickness, whichever is smaller, shall be unacceptable. Those indications in the range 0.010 inch to 0.020 inch or 2% of the thickness or diameter, whichever is smaller, shall be a minimum of 0.500 inch apart; those indications less than 0.010 inch shall be a minimum of 0.12 inch apart.

4.3. Surface Rework. All surface pores, gouges, and other defects deeper than 0.005 inch or 3% of the thickness, whichever is smaller, shall be unacceptable. Surface imperfections may be faired smooth to remove any notch effect provided dimensional tolerances are still maintained.

5. QUALITY ASSURANCE PROVISIONS

5.1. Vendor Responsibility. The manufacturer shall make all tests and inspections of the material covered by this specification before shipment, unless otherwise specified. All test and inspection results shall be furnished to the purchaser.

5.2. Customer Review. The purchaser or his representative may witness the testing and inspection of the material. The manufacturer shall give the purchaser ample notice of the time and place of designated tests. If the purchaser's representative is not present at this time and a new date is not set, the requirement for purchaser's inspection at the place of testing is waived. When the purchaser's representative is present at the appointed time and place, the manufacturer shall afford him, without charge, all reasonable facilities to assure that the material is being furnished in accordance with this specification. This inspection shall not interfere unnecessarily with production operations.

5.3. Sample Selection. Care shall be exercised to insure that the samples selected for testing and chemical analyses are representative of the material and uncontaminated by the sampling procedure. Samples for the determination of mechanical properties shall be selected so as to consume a minimum amount of material, i.e., specimens from material of Class A may be taken transverse to the final working direction from bar of sufficient width or from rod greater than 2.0 inches in diameter. If there is any question about the sampling technique or the analysis, the methods for sampling and analysis shall be those agreed to by the buyer and seller. The location of all test samples shall be reported in the certificate of compliance.

5.4. Test Methods

5.4.1. Chemical Analysis. Chemical analyses shall be conducted by mutually acceptable procedures, such as the vacuum fusion methods for gases, the combustion method for carbon, and the spectrochemical methods for metallic elements. Disputes shall be settled by accepted referee methods, such as the ASTM "Methods for Chemical Analysis of Reactor and Commercial Columbium."

5.4.2. Tensile Test. The tension test shall be conducted in accordance with ASTM Designation E8-57T, "Methods of Tension Testing of Metallic Materials." Yield strength shall be determined by the offset (0.2%) method. The tensile properties shall be determined using a strain rate of 0.005 inch per inch per minute up to 0.6% offset and then 0.050 inch, plus or minus 0.020 inch, per inch per minute to fracture.

5.4.3. Stress-Rupture Test. Stress-rupture properties of specimens shall be determined by mutually acceptable testing techniques. Suggested testing techniques for determining stress-rupture properties are:

Specimens shall be tested in a vacuum of 1×10^{-6} torr or better. The vacuum system shall incorporate an optically tight liquid nitrogen cold trap or a getter-ion pump.

Specimens shall be held for a half hour at the test temperature before application of load.

Test temperature shall be maintained at plus or minus 10°F during the test.

5.4.4. Grain Size. Grain size determinations shall be made according to ASTM Specification E112-61, "Estimating the Average Grain Size of Metals."

5.5. Number of Tests Required. Representative test specimens from the finished product representing each ingot and each lot of material shall be taken to determine conformity to this specification. The minimum frequency of these tests shall be:

Finished Product Chemistry - one per lot per ingot

Tensile Test - two per lot per ingot

Stress-Rupture Test - two per lot per ingot

Grain Size - two per lot per ingot

Microhardness Traverse - one per lot per ingot

5.6. Retest and Rework

5.6.1. Surface Contamination. Any sample or specimen exhibiting obvious surface contamination or improper preparation which disqualifies it as a truly representative sample shall be replaced with a new sample.

5.6.2. Rework. If inspection and test results of a lot do not conform to the requirements of this specification, the lot may be reworked at the option of the manufacturer. The lot shall be acceptable if all test results, after reworking, conform to this specification.

5.7. Inspection

5.7.1. Methods of Inspection

5.7.1.1. Radiographic. Whenever specified, the product shall be radiographed and found free of porosity and inclusions as specified in paragraph 4.2 using the technique described in AMS 2635, "Radiographic Inspection." The radiographs and product shall be identified so the exact position of each radiograph can be correlated with the specific area on the particular product.

5.7.1.2. Ultrasonic. All material 0.125-inch diameter and larger shall be inspected ultrasonically.

5.7.1.2.1. Method and Equipment. The finished products shall be ultrasonically inspected by the immersed technique at 5 mc or above. Transducers shall be no larger than 0.75-inch diameter. Surface finishes shall be no rougher than 125 rms. Inspection shall be by longitudinal wave and by shear wave in two perpendicular directions, i.e., longitudinal and transverse and shall be with focused transducers appropriate to the diameter being inspected (360 degree transducers are allowable where appropriate). Automatic equipment which traverses a

spiral path is satisfactory; but three traverses shall be made, one with the transducer in the circumferential shear position, one with the transducer in the axial shear position, and one with the transducer in the longitudinal wave position, unless otherwise specified.

5.7.1.2.2. Calibration of Bar and Rod. Calibration shall be on notches and holes in a segment of the material reserved solely for calibration purposes or in a calibration specimen of similar nature and shape. The depth of the notches shall be 3% of the bar thickness, 1.5% of the rod diameter, or 0.005 inch, whichever is smaller; the width, no greater than depth; the length, greater than beam width. The notches shall be placed perpendicular to the direction of the shear wave beam and perpendicular to the surface, e.g., axial and circumferential notches on bar. In addition to the notches, a 0.020-inch diameter hole shall be made at least 0.5-inch deep in the calibration piece parallel to the surface at a distance from the surface of $1/2$ the thickness or diameter or, if the thickness exceeds 0.750 inch, $1/4$, $1/2$ and $3/4$ the thickness. Calibration settings to achieve 80% amplitude of these notches or holes along with the magnitude of the other applicable calibration defects shall be recorded. For example, on bar with shear wave, the notch on the near surface should be set at 80% and the amplitudes recorded for the indications from the hole and the notch on the far surface. Gain settings should be recorded to achieve 80% as above and 80% on each of the other applicable calibration defects. For longitudinal wave, only the 0.020-inch diameter holes, with additional holes at $1/4$ and $1/2$ the thickness if the thickness exceeds 0.750 inch, shall be used for calibration.

5.7.1.2.3. Evaluation. Evaluation during inspection shall be made against the appropriate calibration defect. For example, on bar with shear wave, the defects on or near the far surface shall be compared to the calibration from the far surface notch; defects near the center shall be compared to the calibration from the hole at the appropriate depth; defects on the near surface shall be compared to the calibration from the near surface notch.

5.7.1.2.4. Reports. The ultrasonic inspection report shall contain the equipment serial numbers, calibration amplitudes and gain settings, and the amplitude and location of each defect whose amplitude is 60% or greater.

5.7.1.2.5. Rejection. The above procedure shall be followed, and indications of defects which exceed the magnitude obtained from the appropriate calibrated notch in the sample shall be cause for rejection, unless otherwise agreed by the purchaser and vendor.

5.7.1.3. Penetrant Inspection. The exterior surface of the product shall be penetrant inspected and found free of flaws as specified in paragraph 4.3 using AMS 2645, "Fluorescent Penetrant Inspection," or AMS 2646, "Contrast Dye Penetrant Inspection." All parts thus inspected shall be marked with ink stamps as described in the specification; impression stampings or etching shall be unacceptable.

5.7.1.4. Reports. The manufacturer shall supply at least three copies of a report showing inspection results for each lot of material in the shipment. The report shall also include the number of the specification and the purchase order or contract number.

5.8. Rejection. Material not conforming to this specification or to any authorized modification shall be subject to rejection. Unless otherwise specified, rejected material may be returned to the manufacturer at the manufacturer's expense if the purchaser does not receive other instructions for disposition within three weeks after notice of rejection.

5.9. Referee. If the manufacturer and the purchaser disagree about the conformance of the material to the requirements of this specification or any special test specified by the purchaser, a mutually acceptable referee's test shall be used to determine conformance.

6. PREPARATION FOR DELIVERY

6.1. Identification. Each bundle, box or carton shall be legibly and conspicuously marked or tagged with the number of this specification, purchase order or contract number, type, ingot number, lot number, nominal size, and the gross, net, and tare weights. When each bundle, box or carton consists of components from more than one ingot number or lot number, each component shall be identified individually.

6.2. Packing. Each individual item shall be wrapped in heavy gauge polyethylene film or similar material and packed in a manner assuring safe delivery when properly transported by any common carrier.

7. DEFINITIONS

7.1. Lot. A lot shall include all material of the same size, shape, condition and finish from one heat of material and which has received the same processing, has been annealed in the same vacuum annealing charge and has been processed simultaneously in all operations in which temperatures may reach 500°F or above. When process temperatures and

environments are closely controlled or when closely adjacent sizes receive similar processing, lots may be combined for chemical, tensile and stress-rupture tests only, provided prior written approval has been obtained from the General Electric Company.

7.2. Check Analysis. An analysis may be requested by the purchaser of the metal after it has been processed into finished mill forms, to verify the composition within a heat or lot. Check analysis tolerances do not broaden the specified heat analysis requirements but rather cover variations between laboratories in the measurement of the chemical content.

7.3. Significance of Numerical Limits. For determining compliance with the specified limits for requirements of the properties listed below, an observed value or a calculated value shall be rounded off using the rounding-off method in ASTM Designation E29-58T, "Recommended Practices for Designating Significant Places in Specified Limiting Values."

<u>Test</u>	<u>Rounded-Off Unit for Observed or Calculated Value</u>
Chemical composition and dimensional tolerances (when expressed decimally)	Nearest unit in the last right-hand place of figures of the specified limit
Tensile strength	Nearest 100 psi
Elongation	Nearest 1%
Rupture life	Nearest 0.1 hour

SPECIFICATION

BAR AND ROD: Cb-132M (Cb-20Ta-15W-5Mo-2Zr-0.13C) ALLOY

prepared for

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

Contract NAS 3-2547

SPACE POWER AND PROPULSION SECTION

MISSILE AND SPACE DIVISION

GENERAL ELECTRIC COMPANY

CINCINNATI, OHIO 45215

BAR AND ROD: Cb-132M
(Cb-20Ta-15W-5Mo-2Zr-0.13C) ALLOY

- CONTINUED

DATE

6 May 1965

NO.

01-0010-01-A

The following exception to Specification 01-0010-00-A is:

Section 3.5.3. Check Analysis. Finished product analysis shall not exceed the following limits or variations:

<u>Element</u>	<u>Check Analysis Limits, Max., ppm</u>	<u>Permissible Variation in Check Analysis, ppm</u>
Hydrogen	10	+2

Table I - Chemical Composition: The maximum hydrogen content shall not exceed 10 ppm.

SPECIFICATION

BAR AND ROD: Mo-TZC (Mo-1.25Ti-0.15Zr-0.12C) ALLOY

prepared for
NATIONAL AERONAUTICS AND SPACE ADMINISTRATION
Contract NAS 3-2547

SPACE POWER AND PROPULSION SECTION
MISSILE AND SPACE DIVISION
GENERAL ELECTRIC COMPANY
CINCINNATI, OHIO 45215

BAR AND ROD: Mo-TZC (Mo-1.25Ti-0.15Zr-0.12C)
ALLOY

- CONTINUED

DATE

June 3, 1965

NO.

01-0011-00-C

BAR AND ROD: Mo-TZC (Mo-1.25Ti-0.15Zr-0.12C) ALLOY

1. SCOPE

1.1. Scope. This specification covers Mo-TZC (Mo-1.25Ti-0.15Zr-0.12C) alloy in bar and rod form intended for high temperature structural applications in alkali metal systems.

1.2. Classes. A Recrystallized
B Stress-Relieved

2. APPLICABLE DOCUMENTS

2.1. Government Documents. None

2.2. Non-Government Documents

ASTM Designation E8-57T
(26 December 1957)

Method of Tension Testing of Metallic
Materials

ASTM Designation E29-58T
(1958)

Recommended Practices for Designating
Significant Places in Specified Limiting
Values

ASTM E112-61
(1961)

Estimating Average Grain Size of Metals

AMS 2635
(15 August 1958)

Radiographic Inspection

AMS 2645
(1 March 1955)

Fluorescent Penetrant Inspection

AMS 2646
(1 March 1955)

Contrast Dye Penetrant Inspection

3. REQUIREMENTS

3.1. Acknowledgments. The vendor shall mention this specification in all quotations and all purchase order acknowledgments.

3.2. Manufacture. Material covered by this specification shall be made from ingots which have been double vacuum melted by the electron beam and/or consumable electrode arc melting processes. Breakdown operations shall be performed with conventional extrusion, forging and rolling equipment found in primary ferrous and nonferrous plants.

3.3. Processing. The starting stock size, processing temperatures, percentages of reduction and in-process and final annealing, aging or stress-relieving temperatures and times shall be selected by the vendor to achieve the structure or grain size range specified in paragraph 3.6 and mechanical properties specified in paragraph 3.7. The

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amount of total reduction from the turned ingot to the final products of Classes A and B shall exceed 75%; the amount of final reduction imparted to mill products of Class B since the last recrystallization anneal and prior to the final stress-relief heat treatment shall not exceed 80%. The amount of final reduction for each mill product, imparted just prior to the final vacuum heat treatment and the total reduction since the previous recrystallization anneal shall be reported in the certificate of compliance.

3.4. Condition

3.4.1. Class A. The finished product shall be supplied in the recrystallized condition throughout the cross-sectional area to the grain size range specified in paragraph 3.6.

3.4.2. Class B. The finished product shall be supplied in the stress-relieved condition throughout the cross-sectional area as specified in paragraph 3.6.

3.4.3. Heat Treating. All heating shall be carried out in a vacuum less than 1×10^{-4} torr or in a hydrogen atmosphere with a dew point of less than -100°F . All mill products to be heat treated shall be thoroughly degreased, chemically cleaned and protected from the furnace parts by an appropriate refractory metal layer. The conditions of final heat treating shall be reported in the certificate of compliance.

3.4.4. Surface Contamination. All items are to be free of contamination or internal oxidation determined by the following technique: representative samples of each bar diameter shall be subject to thermal treatment sufficient to induce approximately 80% recrystallization throughout the core of the cross-sectional area. Indications of inhibited recrystallization at or near the surface by metallographic examination of transverse sections of heat treated samples shall be construed as contamination and be cause for rejection of all material represented by that sample. The material shall be acceptable if the contaminated layer is completely eliminated before shipment by a machining operation with the specified dimension and tolerances.

3.5. Chemical Composition

3.5.1. Ingot/Billet Composition. The chemical composition of ingots and billets for conversion to finished products shall conform to Table I (page 4). A minimum of four analyses shall be obtained as follows: ingot top-center, mid-radius and edge, and ingot bottom-center; all analyses must conform to ranges stated in Table I.

3.5.2. Final Product Composition. The manufacturer's ingot analyses shall be considered the chemical analyses of products supplied under this specification (Table I) except carbon, oxygen, nitrogen and hydrogen content which shall be determined on the finished product.

3.5.3. Check Analysis. Finished product analysis shall not exceed the following limits or variations:

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TABLE I

CHEMICAL COMPOSITIONMo-TZC (Mo-1.25Ti-0.15Zr-0.12C) ALLOY

Element	Minimum Content w/o	Maximum Content w/o	Analysis Required
Molybdenum	98.50	---	↑
Titanium	1.20	1.30	
Zirconium	0.13	0.18	
Carbon	0.11	0.14	
		ppm	
Oxygen	---	20	↑
Nitrogen	---	10	
Hydrogen	---	5	↓
Tungsten	---	120	
Silicon	---	50	↓
Iron	---	50	
Chromium	---	25	↓
Tin	---	40	
Nickel	---	20	↓
Copper	---	20	
Aluminum	---	20	↓
Calcium	---	20	
Manganese	---	20	↓
Magnesium	---	20	
Cobalt	---	20	↓
Lead	---	10	
Tantalum	---	100	↓
Columbium	---	100	
Vanadium	---	50	↓

Each
IngotWhen so
Specified

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<u>Element</u>	<u>Check Analysis Limits, Max., ppm</u>	<u>Permissible Variations in Check Analysis, ppm</u>
Carbon	1100 min.; 1400 max.	± 50
Oxygen	25	+ 5
Nitrogen	15	+ 2
Hydrogen	5	+ 2

3.6. Structure

3.6.1. Class A. The grain size of the final product shall conform to the following limits:

<u>Product Diameter or Thickness, Inches</u>	<u>Minimum Allowable ASTM Grain Size No.</u>	<u>Allowable Spread in ASTM Grain Size Nos. in Any One Item</u>	<u>% R_x Minimum</u>
0.125 to 0.250	4	2	90
Over 0.250 to 0.500	4	2	90
Over 0.500 to 1.0	4	2	90
Over 1.0 to 2.0	4	2	90
Greater than 2.0	3	3	90

3.6.2. Class B. The grain size of the final product shall conform to the following limits:

<u>Product Diameter or Thickness, Inches</u>	<u>% R_x Maximum</u>
0.125 to 0.250	5
Over 0.250 to 0.500	5
Over 0.500 to 1.0	5
Over 1.0 to 2.0	5
Greater than 2.0	5

3.7. Mechanical Properties. The final product shall satisfy the following mechanical property requirements:

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3.7.1. Room Temperature Tensile Properties (Tentative). Representative samples of the material in final form shall be capable of the following property limits at room temperature (65°-85°F).

Class	Ultimate Tensile Strength, ksi		0.2% Yield Strength, ksi		Elong., % in 4D
	Minimum	Maximum	Minimum	Maximum	Minimum
A	80	110	60	90	5
B*	125	175	100	140	15

* Parallel to the Direction of Work.

3.7.2. Stress-To-Rupture Tests (Tentative). The material shall be capable of achieving the following stress-rupture life under suitable environmental conditions (see paragraph 5.4.3).

Class	Test Temp. °F	Stress, ksi	Minimum Life Hours
A	2400	35	20
B*	2400	30	20

* Parallel to the Direction of Work.

3.8. Tolerances

3.8.1. Rolled, Swaged or Drawn Rounds

3.8.1.1. Definition. Rod - 3.5 inches in diameter or less.

3.8.1.2. Diameter. The permissible variation in diameter and the limits of out-of-roundness of descaled rounds shall not exceed those in Table II (refer to page 7).

3.8.1.3. Cut Lengths. Maximum length variation shall be 0.25 inch.

3.8.1.4. Straightness. Maximum deviation shall be 0.050 inch per foot in any length.

3.8.2. Square or Rectangular Bar

3.8.2.1. Definition. Bar - any straight product with a rectangular cross section 0.187 inch or more thick and less than 5 inches wide.

3.8.2.2. Dimensions. Unless otherwise specified, forged or rolled, square or rectangular shapes shall have the following tolerances:

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TABLE II

PERMISSIBLE DIMENSIONAL VARIATIONS FOR ROUND BAR

<u>Diameter Inches</u>	<u>Diameter Variation Inch</u>	<u>Out-of-Roundness Inch</u>
0.125 to 0.281	+0.002, -0.002	0.004
Over 0.281 to 0.406	+0.010, -0.005	0.008
Over 0.406 to 0.625	+0.010, -0.005	0.012
Over 0.625 to 0.875	+0.015, -0.005	0.015
Over 0.875 to 1.000	+0.020, -0.005	0.015
Over 1.000 to 1.375	+0.020, -0.010	0.018
Over 1.375 to 1.500	+0.020, -0.015	0.020
Over 1.500 to 1.625	+0.025, -0.015	0.020
Over 1.625 to 2.000	+0.030, -0.020	0.025
Over 2.000 to 2.500	+0.032, -0.032	0.025
Over 2.500 to 3.250	+0.032, -0.032	0.027
Over 3.250 to 3.500	+0.045, -0.045	0.040
Centerless Ground Rounds		
0.0625 to 2.00	+0.002, -0.002	
Over 2.00	+0.003, -0.002	

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ThicknessLengthWidth

± 0.025 inch or $\pm 5\%$,
whichever is less

 ± 0.125 inch ± 0.125 inch

3.8.2.3. Straightness of Bar. Maximum deviation shall be 0.050 inch per foot in any length.

3.9. Reports. The manufacturer shall supply at least three copies of a report showing non-proprietary manufacturing methods, processing conditions, and test procedures and results for each lot of material in the shipment. The report shall also include the number of the specification and the purchase order or contract number.

4. MAXIMUM ALLOWABLE DISCONTINUITIES

4.1. General. The finished product shall be visibly free from oxide or scale of any nature, grease, oil, residual lubricants, and other extraneous materials. Cracks, laps, seams, gouges, and fins shall be unacceptable.

4.2. Porosity and Inclusions. Indications of internal porosity and non-metallic inclusions greater than 0.020 inch or 3% of the thickness, whichever is smaller, shall be unacceptable. Those indications in the range 0.010 inch to 0.020 inch or 2% of the thickness or diameter, whichever is smaller, shall be a minimum of 0.500 inch apart; those indications less than 0.010 inch shall be a minimum of 0.12 inch apart.

4.3. Surface Rework. All surface pores, gouges, and other defects deeper than 0.005 inch or 3% of the thickness, whichever is smaller shall be unacceptable. Surface imperfections may be faired smooth to remove any notch effect provided dimensional tolerances are still maintained.

5. QUALITY ASSURANCE PROVISIONS

5.1. Vendor Responsibility. The manufacturer shall make all tests and inspections of the material covered by this specification before shipment, unless otherwise specified. All test and inspection results shall be furnished to the purchaser.

5.2. Customer Review. The purchaser or his representative may witness the testing and inspection of the material. The manufacturer shall give the purchaser ample notice of the time and place of designated tests. If the purchaser's representative is not present at this time and a new date is not set, the requirement for purchaser's inspection at the place of testing is waived. When the purchaser's representative is present at the appointed time and place, the manufacturer shall afford him, without charge, all reasonable facilities to assure that the material is being furnished in accordance with this specification. This inspection shall not interfere unnecessarily with production operations.

5.3. Sample Selection. Care shall be exercised to insure that the samples selected for testing and chemical analyses are representative of the material and uncontaminated by the sampling procedure. Samples for the determination of mechanical properties shall be selected so as to consume a minimum amount of material, i.e., specimens from

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material of Class A may be taken transverse to the final working direction from bar of sufficient width or from rod greater than 2 inches in diameter. If there is any question about the sampling technique or the analysis, the methods for sampling and analysis shall be those agreed to by the buyer and seller. The location of all test samples shall be reported in the certificate of compliance.

5.4. Test Methods

5.4.1. Chemical Analysis. Chemical analyses shall be conducted by mutually acceptable procedures, such as the vacuum fusion methods for gases, the combustion method for carbon, and the spectrochemical methods for metallic elements. Disputes shall be settled by accepted referee methods.

5.4.2. Tensile Test. The tensile test shall be conducted in accordance with ASTM Designation E8-57T, "Methods of Tension Testing of Metallic Materials." Yield strength shall be determined by the offset (0.2%) method. The tensile properties shall be determined using a strain rate of 0.005 inch per inch per minute up to 0.6% offset and then 0.050 inch, plus or minus 0.02 inch, per inch per minute to fracture.

5.4.3. Stress-Rupture Test. Stress-rupture properties of specimens shall be determined by mutually acceptable testing techniques. Suggested testing techniques for determining stress-rupture properties are:

Specimens shall be tested in a vacuum of 1×10^{-5} torr or better. The vacuum system shall incorporate an optically tight liquid nitrogen cold trap or a getter-ion pump.

Specimens shall be held for a half hour at the test temperature before application of load.

Test temperature shall be maintained at plus or minus 10°F during the test.

5.4.4. Grain Size. Grain size determinations shall be made according to ASTM Specification E112-61, "Estimating the Average Grain Size of Metals."

5.5. Number of Tests Required. Representative test specimens from the finished product representing each ingot and each lot of material shall be taken to determine conformity to this specification. The minimum frequency of these tests shall be:

Finished Product Chemistry - one per lot per ingot

Tensile Test - two per lot per ingot

Stress-Rupture Test - two per lot per ingot

Grain Size - two per lot per ingot

5.6. Retest and Rework

5.6.1. Surface Contamination. Any sample or specimen exhibiting obvious surface contamination or improper preparation which disqualifies it as a truly representative sample shall be replaced with a new sample.

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5.6.2. Rework. If inspection and test results of a lot do not conform to the requirements of this specification, the lot may be reworked at the option of the manufacturer. The lot shall be acceptable if all test results, after reworking, conform to this specification.

5.7. Inspection

5.7.1. Methods of Inspection

5.7.1.1. Radiographic. Whenever specified, the product shall be radiographed and found free of porosity and inclusions as specified in paragraph 4.2 using the technique described in AMS 2635, "Radiographic Inspection." The radiographs and product shall be identified so the exact position of each radiograph can be correlated with the specific area on the particular product.

5.7.1.2. Ultrasonic. All material 0.125-inch diameter and larger shall be inspected ultrasonically.

5.7.1.2.1. Method and Equipment. The finished product shall be ultrasonically inspected by the immersed technique at 5 mc or above. Transducers shall be no larger than 0.75-inch diameter. Surface finishes shall be no rougher than 125 rms. Inspection shall be by longitudinal wave and by shear wave in two perpendicular directions, i.e., longitudinal and transverse and shall be with focused transducers appropriate to the diameter being inspected (360 degree transducers are allowable where appropriate). Automatic equipment which traverses a spiral path is satisfactory; but three traverses shall be made, one with the transducers in the circumferential shear position, one with the transducer in the axial shear position, and one with the transducer in the longitudinal wave position, unless otherwise specified.

5.7.1.2.2. Calibration of Bar and Rod. Calibration shall be on notches and holes in a segment of the material reserved solely for calibration purposes or in a calibration specimen of similar nature and shape. The depth of the notches shall be 3% of the bar thickness, 1.5% of the rod diameter, or 0.005 inch, whichever is smaller; the width, no greater than depth; the length, greater than beam width. The notches shall be placed perpendicular to the direction of the shear wave beam and perpendicular to the surface, e.g., axial and circumferential notches on bar. In addition to the notches, a 0.020-inch diameter hole shall be made at least 0.5-inch deep in the calibration piece parallel to the surface at a distance from the surface of 1/2 the thickness or diameter or, if the thickness exceeds 0.750 inch, 1/4, 1/2 and 3/4 the thickness. Calibration settings to achieve 80% amplitude of these notches or holes along with the magnitude of the other applicable calibration defects shall be recorded. For example, on bar with shear wave, the notch on the near surface should be set at 80% and the amplitude recorded for the indications from the hole and the notch on the far surface. Gain settings should be recorded to achieve 80% as above and 80% on each of the other applicable calibration defects. For longitudinal wave, only the 0.020-inch diameter holes, with additional holes at 1/4 and 1/2 the thickness if the thickness exceeds 0.750 inch, shall be used for calibration.

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5.7.1.2.3. Evaluation. Evaluation during inspection shall be made against the appropriate calibration defect. For example, on bar with shear wave, the defects on or near the far surface shall be compared to the calibration from the far surface notch; defects near the center shall be compared to the calibration from the hole at the appropriate depth; defects on the near surface shall be compared to the calibration from the near surface notch.

5.7.1.2.4. Reports. The ultrasonic inspection report shall contain the equipment serial numbers, calibration amplitudes and gain settings, and the amplitude and location of each defect whose amplitude is 60% or greater.

5.7.1.2.5. Rejection. The above procedures shall be followed and indications of defects which exceed the magnitude obtained from the appropriate calibrated notch in the sample shall be cause for rejection, unless otherwise agreed by the purchaser and vendor.

5.7.1.3. Penetrant Inspection. The exterior surface of the product shall be penetrant inspected and found free of flaws as specified in paragraph 4.3 using AMS 2645, "Fluorescent Penetrant Inspection," or AMS 2646, "Contrast Dye Penetrant Inspection." All parts thus inspected shall be marked with ink stamps as described in the specification; impression stampings or etching shall be unacceptable.

5.7.1.4. Reports. The manufacturer shall supply at least three copies of a report showing inspection results for each lot of material in the shipment. The report shall also include the number of the specification and the purchase order or contract number.

5.8. Rejection. Material not conforming to this specification or to any authorized modification shall be subject to rejection. Unless otherwise specified, rejected material may be returned to the manufacturer at the manufacturer's expense if the purchaser does not receive other instructions for disposition within three weeks after notice of rejection.

5.9. Referee. If the manufacturer and the purchaser disagree about the conformance of the material to the requirements of this specification or any special test specified by the purchaser, a mutually acceptable referee's test shall be used to determine conformance.

6. PREPARATION FOR DELIVERY

6.1. Identification. Each bundle, box or carton shall be legibly and conspicuously marked or tagged with the number of this specification, purchase order or contract number, type, ingot number, lot number, nominal size, and the gross, net, and tare weights. When each bundle, box or carton consists of components for more than one ingot number or lot number, each component shall be identified individually.

6.2. Packing. Each individual item shall be wrapped in heavy gauge polyethylene film or similar material and packed in a manner assuring safe delivery when properly transported by any common carrier.

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7. DEFINITIONS

7.1. Lot. A lot shall include all material of the same size, shape, condition and finish from one heat of material and which has received the same processing, has been annealed in the same vacuum annealing charge and has been processed simultaneously in all operations in which temperatures may reach 500°F or above. When process temperatures and environments are closely controlled or when closely adjacent sizes receive similar processing, lots may be combined for chemical, tensile, and stress-rupture tests only, provided prior written approval has been obtained from the General Electric Company.

7.2. Check Analysis. An analysis, made or requested by the purchaser of the metal after it has been processed into finished mill forms, to verify the composition with a heat or lot. Check analysis tolerances do not broaden the specified heat analysis requirements but rather cover variations between laboratories in the measurement of the chemical content.

7.3. Significance of Numerical Limits. For determining compliance with the specified limits for requirements of the properties listed below, an observed value or a calculated value shall be rounded off using the rounding-off method in ASTM Designation E29-58T, "Recommended Practices for Designating Significant Places in Specified Limiting Values."

<u>Test</u>	<u>Rounded-Off Unit for Observed or Calculated Value</u>
Chemical composition and dimensional tolerances (when expressed decimally)	Nearest unit in the last right-hand place of figures of the specified limit
Tensile strength	Nearest 100 psi
Elongation	Nearest 1%
Rupture Life	Nearest 0.1 hour

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SPECIFICATION

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prepared for
NATIONAL AERONAUTICS AND SPACE ADMINISTRATION
Contract NAS 3-2547

SPACE POWER AND PROPULSION SECTION
MISSILE AND SPACE DIVISION
GENERAL ELECTRIC COMPANY
CINCINNATI, OHIO 45215

SP 1073 A

BAR AND ROD: Mo-TZC (Mo-1.25Ti-0.15Zr-0.12C) ALLOY	- CONTINUED	DATE June 3, 1965	NO. 01-0011-01-C

Exceptions to specification No. 01-0011-00-C:

Paragraph 3.2. Manufacture is changed to read: "Material covered by this specification shall be made from ingots which have been single vacuum melted by the electron beam and/or consumable electrode arc melting processes, etc."

Paragraph 3.3. Processing is changed to read: "..... the amount of final reduction imparted to mill products of Class B since the last recrystallization anneal and prior to the final stress-relief heat treatment shall not exceed 85%, etc."

Paragraph 3.4.4. Surface Contamination requirement is deleted.

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