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ADVANCED REFRACTORY ALLOY CORROSION LOOP PROGRAM

Quarterly Progress Report No. 4
For Quarterly Ending April 15, 1966

By

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and

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prepared for

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

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SPACE POWER AND PROPULSION SECTION

MISSILE AND SPACE DIVISION

GENERAL ELECTRIC

CINCINNATI, OHIO 45215

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ADVANCED REFRACTORY ALLOY CORROSION LOOP PROGRAM

QUARTERLY PROGRESS REPORT 4

Covering the Period
January 15, 1966 to April 15, 1966

Edited by

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NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

Lewis Research Center

Under Contract NAS 3-6474

April 29, 1966

Technical Management
NASA - Lewis Research Center
Space Power Systems Division
R. L. Davies

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

FOREWORD

The work described herein is sponsored by the National Aeronautics and Space Administration under Contract NAS 3-6474. For this program, Mr. R. L. Davies is the NASA Project Manager.

The program is being administered for the General Electric Company by Dr. J. W. Semmel, Jr., and E. E. Hoffman, is acting as the Program Manager. J. Holowach, the Project Engineer, is responsible for the loop design, facilities, procurement, and test operations. R. W. Harrison, the Project Metallurgist, is responsible for the materials procurement, utilization, and evaluation aspects of the program. Personnel making major contributions to the program during the current reporting period include:

Alkali Metal Purification and Handling - Dr. R. B. Hand, L. E. Dotson and J. R. Reeves.

Welding and Joining - W. R. Young and S. R. Thompson.

Refractory Alloy Procurement - R. G. Frank and L. B. Engel.

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ADVANCED REFRACTORY ALLOY CORROSION LOOP PROGRAM

I. INTRODUCTION

This report covers the period from January 15, 1966 to April 15, 1966 of a program to fabricate, operate for 10,000 hours, and evaluate a potassium corrosion test loop constructed of T-111 (Ta-8W-2Hf) alloy. Materials for evaluation in the turbine simulator include Mo-TZC and Cb-132M. The loop design will be similar to the Prototype Loop; a two-phase, forced convection, potassium corrosion test loop which is being developed under Contract NAS 3-2547. Lithium will be heated by direct resistance in a primary loop. Heat rejection for condensation in the secondary loop will be accomplished by radiation in a high vacuum environment to the water cooled chamber. The compatibility of the selected materials will be evaluated at conditions representative of space electric power system operating conditions, namely:

a. Boiling temperature, 2050°F
b. Superheat temperature, 2150°F
c. Condensing temperature, 1400°F
d. Subcooling temperature, 1000°F
e. Mass flow rate, 40 lb/hr
f. Boiler exit vapor velocity, 50 ft/sec
g. Average heat flux in plug (0-18 inches), 240,000 BTU/hr ft^2
h. Average heat flux in boiler (0-250 inches), 23,000 BUT/hr ft^2

II. SUMMARY

During the fourth quarter of the program, work proceeded on the topics abstracted below:

Significant effort was made in the past quarter in monitoring the fabrication of the advanced refractory alloys. Poor yields in the fabrication of the T-111 alloy have necessitated the melting of two additional ingots. The processing of these ingots is being expedited to minimize the delay to the program.

Twenty-nine pounds of lithium were transferred to the hot trap. Analyses of this lithium after 126 hours of hot trapping at 1500°F indicated an average nitrogen concentration of less than 8 ppm.

The completed Alkali Metal Purification and Handling System drawings have been submitted to the NASA Program Manager for approval.

The results of a study to determine suitable welding conditions and post-weld annealing treatment for welds between Cb-1Zr alloy and T-111 alloy sheet have indicated that the proposed joints between the Cb-1Zr surge tanks and T-111 fill lines of Corrosion Loop I (T-111) may be produced with manual TIG welding practices, if Cb-1Zr filler material and 2300-2400°F post-weld annealing treatments are used.

III. PROGRAM STATUS

A. MATERIALS PROCUREMENT

1. T-111 Alloy - Fansteel Metallurgical Corporation. T-111 alloy rods measuring 2-1/4-inch diameter and 3-3/4-inch diameter were salvaged from the first two T-111 alloy ingots (Fansteel ingots No. 111-D-1632 and No. 111-D-1633) which cracked during extrusion. The 2-1/4-inch diameter material has been processed into 1/8-inch to 1-1/2-inch diameter rods and 0.062- and 0.094-inch diameter wire; the 3-3/4-inch diameter material has been processed into 0.005- and 0.009-inch thick foil, 0.040-inch thick sheet, and 0.500-inch thick plate. Processing temperatures were less than 800°F. All of these products except the 1-1/2-inch diameter rod were given a final anneal of (1475°C) 2687°F for one hour; the 1-1/2-inch diameter rod was annealed at (1475°C) 2687°F for two hours.

Quality assurance testing of 0.005- and 0.009-inch thick foil and the 0.062-, 0.094-, and 1/8-inch diameter wire was completed at Fansteel Metallurgical Corporation, and the material was received the week of March 6, 1966. Quality assurance testing of the 0.040-inch thick sheet, 0.500-inch thick plate and the 1/4-inch to 1-1/2-inch diameter rods also was completed at Fansteel Metallurgical Corporation, and the material was received the week of April 10, 1966.

The chemical analyses of the arc-cast ingots, No. 111-D-1632 and No. 111-D-1633, and their extruded bars are shown in Table I; chemical analyses of finished products are shown in Table II. The room temperature tensile properties, stress-rupture properties, grain size, and hardness data for the 0.040-inch thick sheet, 0.500-inch thick plate, and 1/4-inch to 1-1/2-inch diameter rods are presented in Table III. Fansteel's metallographic examination of all the products after final annealing revealed them to be 100% recrystallized.

An investigation to delineate the possible cause of the cracking in the first two T-111 alloy extrusions continued. A Rockwell hardness survey across a transverse slice from one of the first 5-1/8-inch diameter T-111 alloy extrusions (No. 111-D-1632) which had cracked, revealed no significant hardness

TABLE I. CHEMICAL ANALYSES[1] OF ARC CAST AND EXTRUDED T-111 ALLOY INGOTS

NO. 111-D-1632 AND NO. 111-D-1633

Sample	Sample Location	Chemical Analysis % W	Hf	ppm O	N	H	C	Cb	Mo	Ni	Co	Fe	V
Arc cast ingot No. 111-D-1632	Top, sidewall	7.70	2.33	--	--	--	--	110	100	<10	<10	20	<10
	Bottom, mid-radius	7.87	2.43	25	20	<5	<10	50	100	<10	<10	20	<10
Extruded ingot No. 111-D-1632	Nose[2], center	7.70	2.08	20	23	5	13	160	100	<10	<10	<10	<10
	Nose[2], mid-radius	7.74	2.02	11	17	4	9	200	70	<10	<10	<10	<10
	Nose[2], edge	7.64	2.32	15	21	6	11	200	200	<10	<10	<10	<10
	Tail[3], center	7.85	2.42	12	22	2	<10	160	100	<10	<10	<10	<10
Arc cast ingot No. 111-D-1633	Top, sidewall	7.80	2.43	--	--	--	--	60	100	<10	<10	20	<10
	Bottom, mid-radius	7.87	2.03	25	10	10	10	110	100	<10	<10	20	<10
Extruded ingot No. 111-D-1633	Nose[2], center	7.62	2.25	16	15	5	10	90	200	<10	<10	<10	<10
	Nose[2], mid-radius	7.93	2.34	18	16	2	18	80	110	<10	<10	<10	<10
	Nose[2], edge	7.72	2.31	27	16	5	15	80	60	<10	<10	<10	<10
	Tail[3], center	7.93	2.05	13	18	12	23	90	200	<10	<10	<10	<10

(1) Analyses performed by Fansteel.

(2) Equivalent to top of arc cast ingot.

(3) Equivalent to bottom of arc cast ingot.

TABLE II. CHEMICAL ANALYSES[1] OF T-111 ALLOY MILL PRODUCTS

FROM INGOTS NO. 111-D-1632 AND NO. 111-D-1633

Ingot No.	Mill Product Form	Mill Product Size	Chemical Analysis, ppm O	N	H	C
111-D-1632	Plate	0.500-inch thick	<10	20	9	10
	Sheet	0.040-inch thick	34	17	5	14
	Foil	0.005-inch thick	41	20	5	12
111-D-1633	Rod	1.5 -inch dia.	to be determined			
	Rod	1.0 -inch dia.	11	24	1	9
	Rod	0.500-inch dia.	30	20	<1	17
	Rod	0.250-inch dia.	15	23	<1	13
	Wire	0.125-inch dia.	69	21	<1	33
	Wire	0.094-inch dia.	111	20	<1	43
	Wire	0.62 -inch dia.	110	22	1	42

(1) Analyses performed by Fansteel.

TABLE III. MECHANICAL PROPERTIES AND GRAIN SIZE OF T-111 ALLOY MILL PRODUCTS FROM INGOTS NO. 111-D-1632 AND NO. 111-D-1633[1]

	Mill Product		Room Temperature Tensile Properties			Stress-Rupture Life in Vacuum (10^{-6} torr) at 2400°F/19,000 psi	Hardness, DPH		ASTM Grain
Ingot No.	Form	Size	Ultimate Strength, 1000 psi	0.2% Yield Strength, 1000 psi	Elong. %	Life Hours	Core	Edge	Size
111-D-1632	Plate	0.500-inch thick	91.9/91.0	79.9/77.4	40/43	11.5/13.8	213	245	6.5
	Sheet	0.040-inch thick	91.8/92.2	82.6/82.5	30/31	16.2/15.7	226	223	7.5
111-D-1633	Rod	1.5-inch dia.							
	Rod	1.0-inch dia.	83.0/83.5	70.0/69.3	47/44	13.2/15.5	207	226	4 to 5.5
	Rod	0.500-inch dia.	89.6/88.5	73.0/72.3	42/43	16.9/16.5	216	226	6.5
	Rod	0.250-inch dia.	89.9/89.6	76.4/76.6	33/35	12.7/14.4	234	237	6.5

(1) As reported by Fansteel.

variation across the billet and the average hardness was found to be 61 R_a. Subsequently, the transverse slice from the extrusion was sectioned, as shown in Figure 1, to prepare specimens for metallographic examination and chemical analysis. Samples were obtained from the center and from the edge of the extrusion. The metallographic examination has been completed and representative photomicrographs of the extrusion are shown in Figures 2 and 3. No major amount of precipitate or second phases were observed at any of the locations examined.

Chemical analyses results of the slice are shown in Table IV. Based on the chemical analyses of the one cross section of the extrusion, the ingot appeared to be reasonably homogeneous. The results of chemical analysis do not indicate any obvious concentrations that might contribute to observed cracking.

Trial forgings of portions of the third T-111 alloy extrusion (No. 111-D-1670), which were conducted at Anderson-Schumaker, Chicago, Illinois, and Westinghouse Electric Corporation, Materials Manufacturing Division, Blairsville, Pennsylvania, were unsuccessful due to center cracking of the billets. The trial forging that was attempted at Anderson-Schumaker utilized a 6,000-pound hammer, flat dies and an initial billet temperature of 2300°F. The billet was heated in air without a protective coating. In an attempt to work the center of the billet more extensively, the trial forging conducted at Westinghouse utilized a 1,000-ton press. The dies consisted of a flat top die and a closed bottom die. The billet was heated to the 2300°F forging temperature in an Inconel retort containing flowing argon gas. In the latter case, the center cracking was confined to the ends. After conditioning to remove any contamination resulting from the previous forging operation, this billet was heated to 1700°F in an air furnace and rod rolled to 2.1-inch diameter without reheating. Subsequently, two 4.5-inch diameter billets were rolled in a similar manner to 3-1/8-inch diameter to be utilized for the production of 3/8-inch OD and one-inch OD tubing.

The two 3-1/8-inch diameter rods were machined into 2.93-inch diameter x 9-inch long billets and shipped to Wolverine Tube Division of Calumet-Hecla.

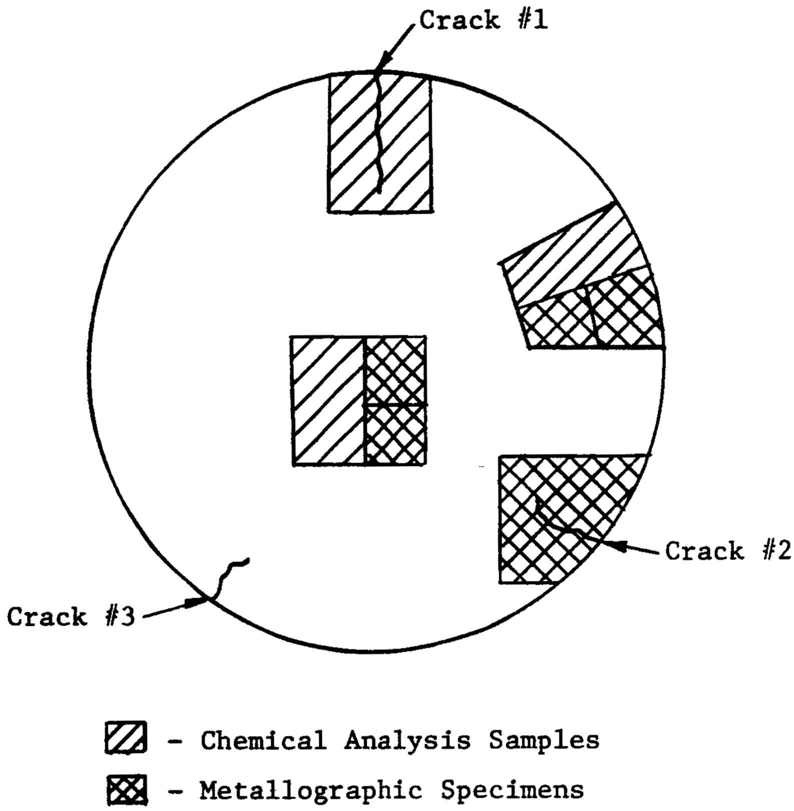
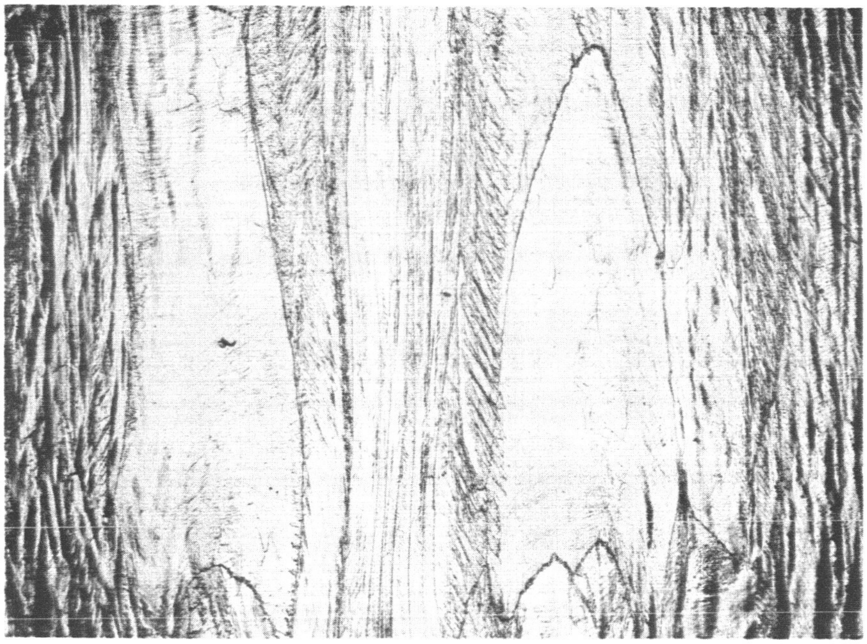
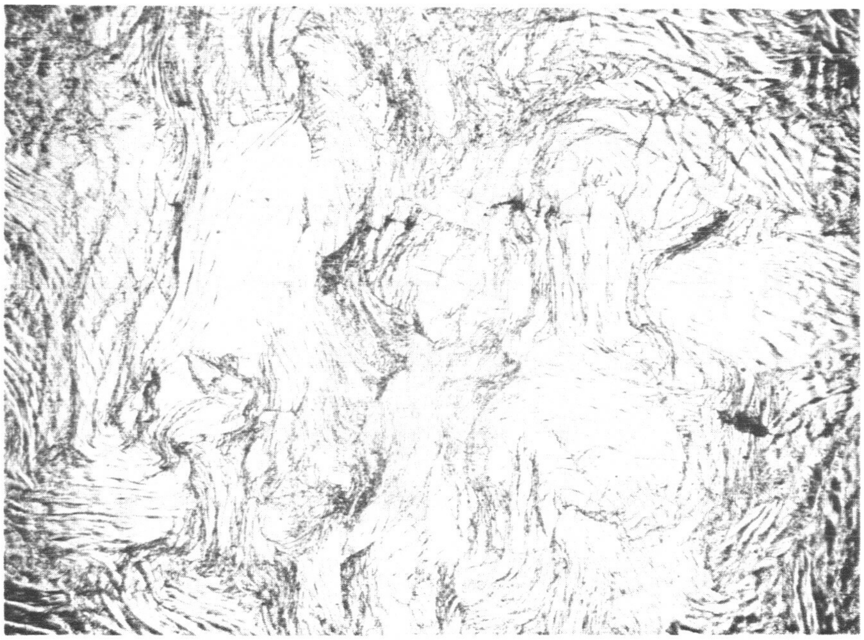


Figure 1. Sectioning of Transverse Slice From Extrusion of Fansteel Ingot No. 111-D-1632.



(a) Longitudinal Section

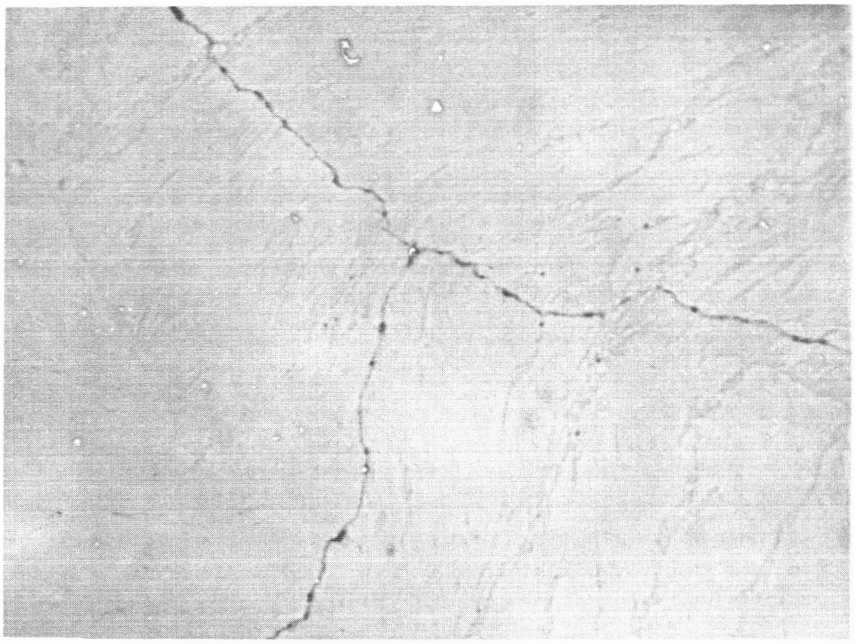


(b) Transverse Section

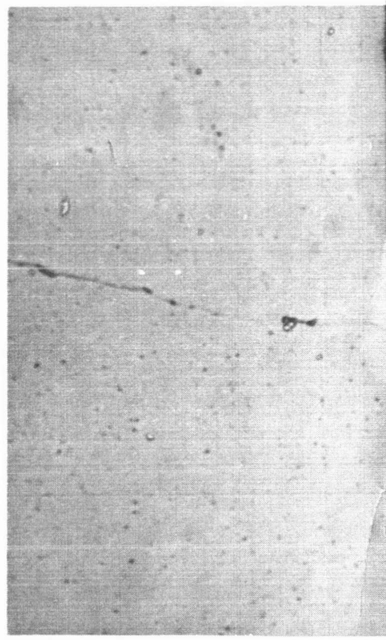
C1068-2

Figure 2. The Microstructure of the Center of the Transverse Slice from the T-111 Alloy Extrusion-Fansteel Heat No. 111-D-1632.

Etchant: 30HCl, 30HF, $15HNO_3$ (a) B170511
Magnification: 30X (b) B170411



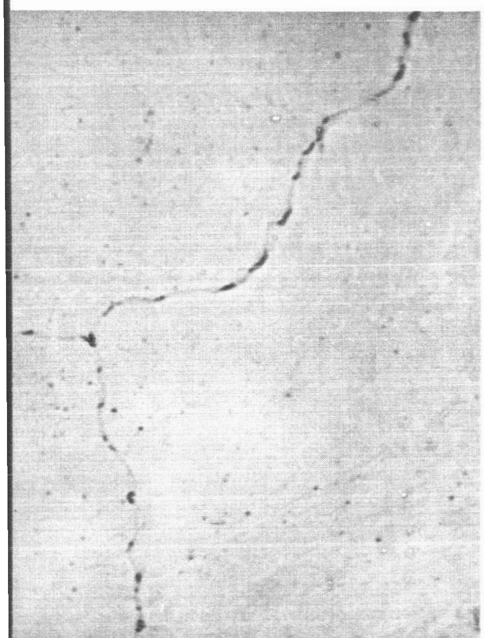
(a) Center of Extrusion



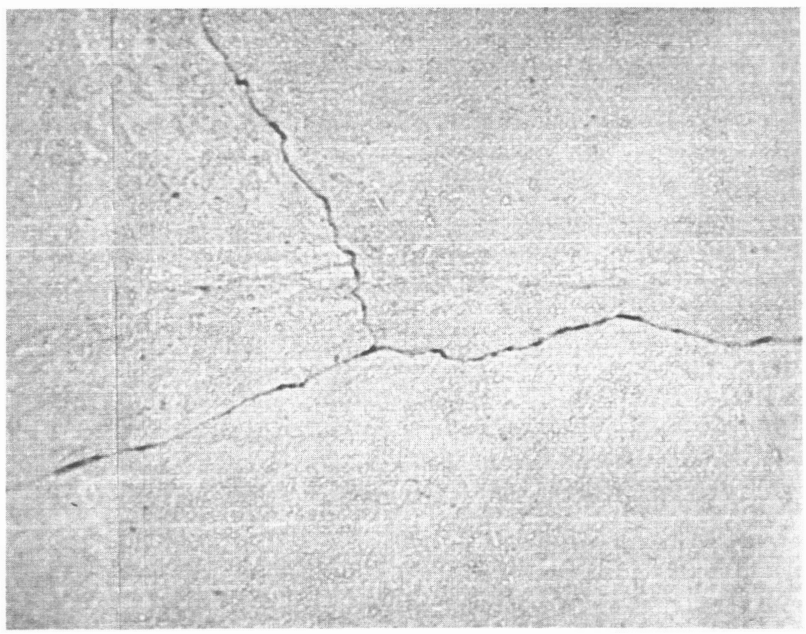
(b) Periphery of Ext

Figure 3. Microstructures of Transverse Sections fr

Etchant: Electrolytically Anodized
Magnification: 100X



rusion Away from a Crack



(c) Periphery of Extrusion Next to a Crack

om the T-111 Extrusion-Fansteel Heat No. 111-D-1632. C1068-3

(a) B170413
(b) B170213
(c) B1706111

TABLE IV. CHEMICAL ANALYSES OF CRACKED T-111 ALLOY

EXTRUSION-FANSTEEL INGOT NO. 111-D-1632

Element	Method of Analyses	Chemical Analyses: Periphery Near Crack	Chemical Analyses: Periphery Away From Crack	Chemical Analyses: Center
W	(1)	7.25%	7.31%	6.97%
Hf	(1)	2.45%	2.32%	2.28%
C	(2)	30 ppm	20 ppm	30 ppm
O	(3)	70 ppm	60 ppm	109 ppm
N	(3)	12 ppm	10 ppm	14 ppm
Ag	(4)	--	--	--
Al	(4)	--	(6)	(6)
B	(4)	--	--	--
Be	(4)	--	--	--
Bi	(4)	--	--	--
Ca	(4)	--	--	--
Cb	(4)	--	--	--
Cd	(4)	--	--	--
Co	(4)	--	--	--
Cr	(4)	--	(7)	(7)
	(5)	10 ppm	--	10 ppm
Cu	(4)	--	--	--
Fe	(4)	--	(6)	(6)
	(5)	64 ppm	--	34 ppm
K	(4)	--	--	--
Mg	(4)	--	(6)	(6)
Mn	(4)	--	(6)	(6)
Mo	(4)	--	--	--
Na	(4)	--	--	--
Ni	(4)	--	(7)	(7)
	(5)	<10 ppm	--	<10 ppm
Pb	(4)	--	--	--
Si	(4)	--	(6)	(6)
Ti	(4)	--	(6)	(6)
V	(4)	--	--	--
Y	(4)	--	--	--
Zn	(4)	--	--	--
Zr	(4)	--	--	--

(1) Wet analysis (Ledoux & Co. Inc., Teaneck, N.J.)
(2) Conductometric
(3) Vacuum fusion
(4) Spectrographic (semi-quantitative)
(5) Colormetric (Ledoux & Co. Inc., Teaneck, N.J.)
(6) Trace amount (< 500 ppm)
(7) <50 ppm

NOTE: Semi-quantitative spectrographic analyses revealed no difference in concentration of those elements that were detected between the center and edge of the extrusion.

Subsequently, the billets were drilled to make tube hollows. During the report interim, five hollows were extruded into tube blanks using procedures developed at Wolverine. The processing procedures are considered proprietary by Wolverine Tube.

Extrusion of the remaining T-111 alloy tube hollows is now scheduled for the week of May 2, 1966 and delivery of the finished 3/8-inch OD and 1-inch OD tubes is scheduled for the week of June 6, 1966.

The EB melting of a fourth T-111 alloy ingot (No. 111-D-1104) was completed at Fansteel Metallurgical Corporation the week of February 20, 1966 and vacuum arc melting of this ingot was completed at Universal Cyclops Steel Corporation on March 7, 1966. The ingot was machined to 8.44 inches in diameter, cut into two equal lengths and shipped to Canton Drop Forging Company for extrusion. The two billets were canned in Type 304 SS seamless pipe by heliarc welding and extruded on March 23, 1966. The extrusion parameters are presented in Table V. After extrusion, the cans were removed and the two billets were conditioned for rolling at Braeburn Alloy Steel Division of Continental-Copper and Steel Industries Incorporated the week of April 24, 1966.

The extrusion of the fourth ingot resulted in a yield lower than anticipated; therefore, the 2-inch and 3.125-inch diameter rod, 1-inch x 2-inch bar and 2.25 OD hollows will be made from a fifth ingot which is scheduled for EB melting the week of April 25, 1966. Delivery of this material is expected the week of July 11, 1966.

2. Mo-TZC Alloy - Climax Molybdenum Company. The 6-inch diameter Mo-TZC alloy ingot[1] was turned to 5.85-inch diameter for extrusion at DuPont. The ingot was induction heated to 3180°F in argon atmosphere and extruded through a 3-inch ID, ZrO_2 coated conical die. The extrusion parameters are shown in Table VI. As the result of severe die wash, the extrusion diameter was close

(1) Advanced Refractory Alloy Corrosion Loop Program, Quarterly Progress Report No. 3 for Period Ending January 15, 1966, NASA Contract NAS 3-6474, NASA-CR-54911, February 3, 1966, p. 8.

TABLE V. EXTRUSION PARAMETERS FOR THE 8.44-INCH DIAMETER MACHINED T-111 ALLOY INGOTS*

Machined ingot size	-	8.44-inch diameter	
Can size	-	9-1/16-inch OD x 1/4-inch thick wall Type 304 SS seamless pipe with a 2-inch thick Type 304 SS nose block and a 1/2-inch thick Type 304 SS back-up block contained within the can	
Leader block/ Follow-up block	-	9-1/16-inch diameter x 6-inch long mild steel	
Container size	-	9-1/4-inch ID	
Die size/Design	-	5-1/8-inch ID/conical	
Die coating	-	None	
Extrusion ratio	-	3.2/1	
Lubricant	-	Hot die grease similar to Fiske 604	
Furnace temperature/ Soak time in salt bath	-	2300°F/75 minute	
Extrusion pressure (Hydraulic)	-	Billet A	~2300 psi peak 2200 psi run out
		Billet B	~2100 psi peak 2000 psi run out
Maximum allowable pressure	-	3850 psi	
Cooling procedure	-	Air cooled	

* Extruded at Canton Drop Forging Company, Canton, Ohio, on March 23, 1966.

TABLE VI. EXTRUSION PARAMETERS FOR THE 5.85-INCH DIAMETER MACHINED Mo-TZC ALLOY INGOT*

Billet size	-	5.85-inch diameter
Container size	-	6.0-inch ID
Die size/Design	-	3.0-inch ID/90° conical
Die coating	-	ZrO_2
Extrusion ratio	-	3.8/1
Lubricant	-	Billet coated with 7052 glass; glass lubricated with Fiske grease. Die lubricated with 7052 glass powder and a glass-wool pad.
Billet temperature	-	3180°F (induction heated in argon)
Extrusion pressure	-	126,000 psi peak 115,500 psi run out
Cooling procedure	-	Air cooled to 1700°F; cooled in Sil-O-Cel from 1700°F to room temperature.

* Extruded at E.I. du Pont de Nemours & Company, Inc., Baltimore, Maryland, on February 2, 1966.

to 3-1/8-inch. Also, there appeared to be a loss of lubrication on one side of the extrusion which resulted in a series of transverse surface cracks along the unlubricated surface. These cracks were successfully removed during subsequent conditioning of the billet for rolling at Hoskins Corporation, Detroit, Michigan.

The results of recrystallization studies of longitudinal samples from the mid-radius of the extruded billet are summarized in Table VII. Although an annealing temperature of 3400°F resulted in an essentially recrystallized structure, metallographic examination revealed continuous films of Mo_2C at the grain boundaries. Similarly, the sample which was heated at 3200°F for one hour also exhibited some Mo_2C in the grain boundaries. Since the presence of Mo_2C in the grain boundaries is not a desirable condition for subsequent working operations, the post-extrusion heat treatment was limited to a stress-relief anneal at 3000°F for one hour in vacuum. A solution anneal at temperatures on the order of 3750°F was not employed because of possible grain growth and the fact that only a 52% reduction is required to reach the final size of 2-inch diameter.

On March 16, 1966, the conditioned and stress relieved Mo-TZC alloy billet was successfully rolled by Hoskins Corporation, Detroit, Michigan, to 2-1/8-inch diameter rod. A rolling temperature of 2400°F was utilized. The 2-inch diameter rod was machined from a portion of the rolled rod and testing of this material was completed. Shipment of the 2-inch diameter rod is scheduled for April 18, 1966. The remaining 2-1/8-inch diameter rod is scheduled for rolling to 1-inch diameter by Climax Molybdenum Company, Coldwater, Michigan on April 21, 1966.

3. Mo-TZC Alloy - General Electric, LMCD. The 1-3/8-inch thick x 2.0-inch wide x 7.0-inch long Mo-TZC alloy plate produced from heats 4451 (M92) and 4453 (M97) was received on January 27, 1966. The chemical analyses of the rolled plate, as supplied by GE-LMCD, are shown in Table VIII and the mechanical properties are presented in Table IX.

The five bars of Mo-TZC alloy, measuring 3/4-inch thick x 3/4-inch wide x 7.0-inch long, produced from heat No. 4454 were received on February 21, 1966.

TABLE VII. RECRYSTALLIZATION[1] OF 3-1/8-INCH DIAMETER Mo-TZC ALLOY EXTRUSION[2]

Temperature[3] °F	Vickers Hardness Number	% Recrystallization
As-extruded	232	10
2800	240	15
3000	236	15
3200	229	25
3400	201	98

(1) Conducted by the Climax Molybdenum Company of Michigan, Ann Arbor, Michigan.

(2) Extruded at E.I. du Pont de Nemours & Company, Inc., Baltimore, Maryland, February 2, 1966, in 6.0-inch OD container--ingot diameter 5.85-inches.

(3) Temperatures maintained for one hour in vacuum.

TABLE VIII. CHEMICAL ANALYSES[1] OF Mo-TZC ALLOY PRODUCED BY GENERAL ELECTRIC-LMCD

Chemical Analysis	Heat No. 4451 (M92)		Heat No. 4453 (M97)	
	Nose[2]	Tail	Nose	Tail
Ti	1.4 %	1.3 %	1.4 %	1.3 %
Zr	0.20%	0.19%	0.20%	0.17%
O	2 ppm	4 ppm	5 ppm	5 ppm
N	3	3	2	2
H	2	1	2	2
C	1172	1169	1205	1120
Al	<8	<8	<8	<8
Ca	<5	<5	<5	<5
Si	48	22	27	27
Cr	9	<8	11	10
Fe	47	47	35	19
Ni	<5	<5	<5	6
Cu	4	4	7	8
W	<150	<150	<150	<150
Mn	10	10	12	12
Mg	<10	<10	<10	<10
Sn	<7	<7	12	13
Co	<20	<20	<20	<20
Pb	<10	<10	<10	<10
Ta	<100	<100	<100	<100
Cb	<100	<100	<100	<100
V	<50	<50	<50	<50
Ag	<1	<1	<1	<1

(1) Analyses performed by GE-LMCD

Samples obtained from rolled 1-3/8-inch thick plate. Ti and Zr determined by wet chemistry.

C determined by conductometric techniques. O, N, H determined by vacuum fusion.

Other metallics analyzed by spectrographic techniques.

(2) Indicates location of chemistry sample with respect to extrusion.

TABLE IX. MECHANICAL PROPERTIES OF Mo-TZC ALLOY[1] PRODUCED BY GENERAL ELECTRIC-LMCD

		Room Temperature Tensile Properties[3]			Stress-Rupture Life[4] Vacuum (10^{-6} Torr) at 2400°F/30,000 psi	
Heat No.	Specimen Location[2]	Ultimate Strength, 1000 psi	0.2% Yield Strength, 1000 psi	Elongation %	Life Hours	Elongation %
4451 (M92)	Nose[5]	134.6	105.9	1.3	37.81	12.4
	Tail[6]	126.5	105.0	1.3	20.94	13.3
4453 (M97)	Nose[7]	96.6	95.2	0.4	28.57	2.2
	Tail[7]	94.7	92.8	0.3	21.88	3.5

(1) Specimens obtained from rolled 1-3/8-inch thick plate and tested by GE-LMCD.

(2) Indicates location of mechanical property sample with respect to the extrusion.

(3) Specimens were 0.250-inch diameter with 1.0-inch gauge length. Strain rate 0.005-inch/inch/minute.

(4) Specimens were 0.159-inch diameter with 1.0-inch gauge length.

(5) Tensile specimen axis perpendicular to rolling direction and specimen annealed for 1 hour at 2192°F before testing; stress-rupture specimen axis parallel to rolling direction and specimen annealed 1 hour at 2372°F before testing.

(6) Tensile specimen axis perpendicular to rolling direction and stress-rupture specimen axis parallel to rolling direction. Both specimens were annealed 1 hour at 2372°F before testing.

(7) Tensile and stress-rupture specimen axis parallel to rolling direction and all specimens annealed 1 hour at 2372°F before testing.

The chemical analyses of the rolled bar as supplied by GE-LMCD, are shown in Table X, and the mechanical properties are presented in Table XI.

4. Cb-132M Alloy - Universal Cyclops Steel Corporation. A second Cb-132M alloy ingot was double EB melted and triple vacuum arc melted by Wah Chang Corporation to replace the original extrusion that had cracked during the straightening operation at Universal Cyclops Steel Corporation. The ingot has been canned in molybdenum and is scheduled for extrusion at DuPont on April 20, 1966.

B. ALKALI METAL PURIFICATION AND HANDLING

The 35-pound capacity lithium hot trap and the vacuum-argon system were installed on the purification dolly. The hot trap was outgassed prior to filling, at temperatures up to 1500°F over a two-week period. At 500°F, following the outgassing operation, pressure was 4 x 10^{-7} torr, and the gas evolution rate was well below one micron-liter per hour.

The hot trap was filled with 29 pounds of lithium filtered at 400°F from the shipping container. The Mine Safety Appliance Research Corporation level probe was used in locating the lithium level and proved to be very sensitive and reproducible. Subsequently, the lithium was hot trapped for 126 hours at 1500°F and then sampled. Nitrogen analyses indicated concentrations of: 11, 10, 5 and 3 ppm. Analyses of the hot trapped lithium for carbon, oxygen and metallic elements is in progress.

The Cb-1Zr tubing received for the thermocouple and level probe wells for the lithium still receiver was rejected due to cracks indicated by ultrasonic and visual inspection. The tubing has been reordered. Fabrication of other components of the purification system will be initiated while the tubing is being replaced.

The drawings for the alkali metal purification and handling systems have been submitted to the NASA Program Manager for final approval.

TABLE X. CHEMICAL ANALYSES[(1)] OF Mo-TZC ALLOY HEAT NO. 4454

PRODUCED BY GENERAL ELECTRIC-LMCD

Chemical Analysis	Location[(2)] Nose	Location[(2)] Tail
Ti	1.4 %	1.4 %
Zr	0.21%	0.21%
O	5 ppm	7 ppm
N	2	3
H	2	2
C	1314	1303
Al	<8	16
Ca	<5	11
Si	15	23
Cr	15	10
Fe	35	19
Ni	9	7
Cu	5	<4
W	100	136
Mn	18	13
Mg	16	<10
Sn	10	<7
Co	<20	<20
Pb	<10	<10
Ta	<100	<100
Cb	<100	<100
V	<50	<50
Ag	<1	<1

(1) Analyses performed by GE-LMCD.

Samples obtained from rolled 3/4-inch thick plate. Ti and Zr analyzed by wet chemistry.

C determined by conductometric techniques. O, N, H analyzed by vacuum fusion.

Other metallics analyzed by spectrographic techniques.

(2) Indicates location of chemistry sample with respect to extrusion.

TABLE XI. MECHANICAL PROPERTIES(1) OF Mo-TZC ALLOY PRODUCED BY GENERAL ELECTRIC-LMCD

Heat No.	Specimen Location(2)	Room Temperature Tensile Properties(3): Ultimate Strength, 1000 psi	0.2% Yield Strength, 1000 psi	Elongation %	Stress-Rupture Life(4) Vacuum (10^{-6} Torr) at 2400°F/30,000 psi: Life Hours	Elongation %
4454	Nose	111.3	101.9	2.1	19.33	13.4
	Tail	95.9	94.5	0.4	---	--

(1) Properties determined by GE-LMCD. Tensile and stress-rupture specimen axis parallel to rolling direction of 3/4-inch thick plate and all specimens annealed 1 hour at 2372°F before testing.

(2) Indicates location of mechanical property sample with respect to extrusion.

(3) Specimens were 0.250-inch diameter with 1.0-inch gauge length.

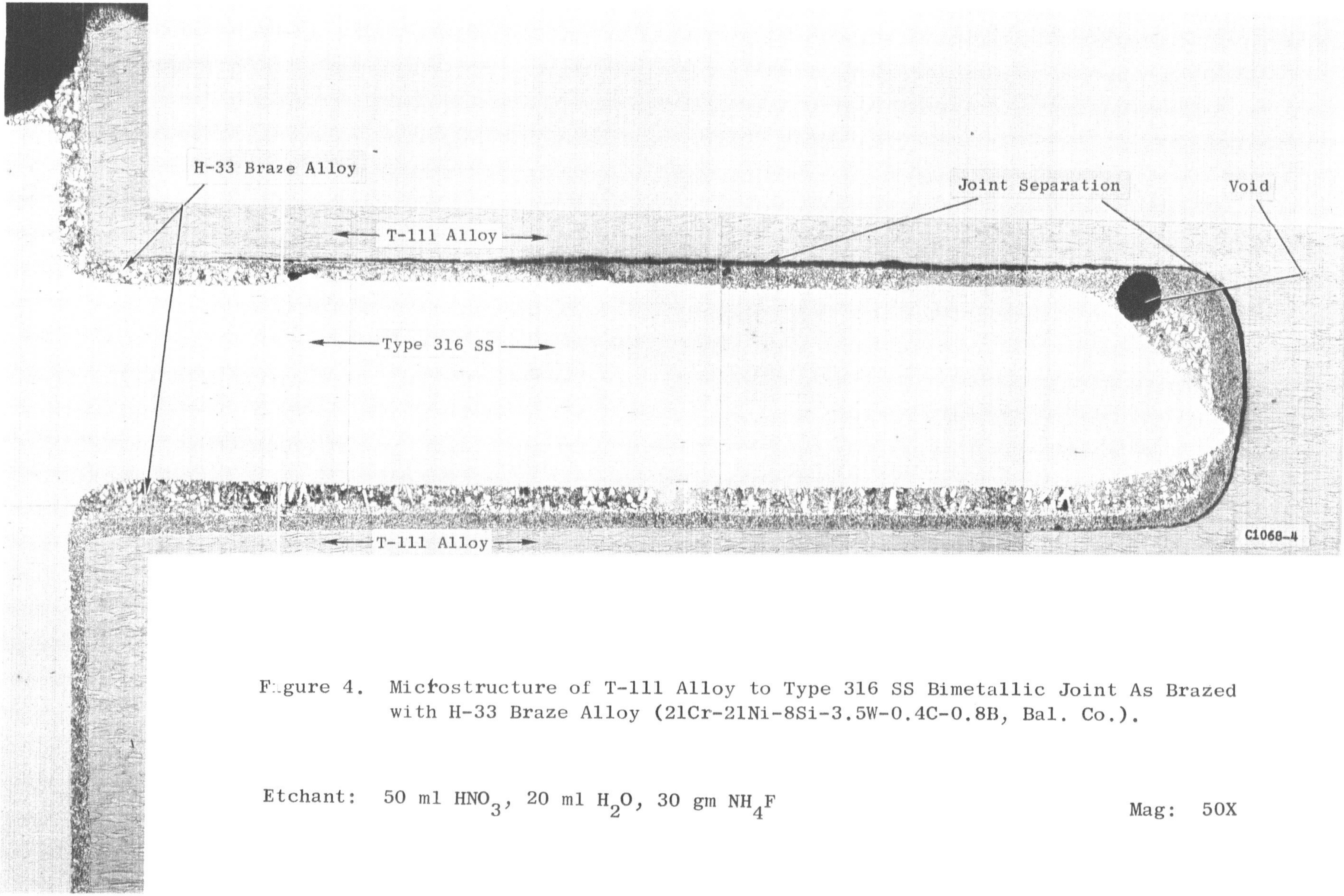
(4) Specimens were 0.159-inch diameter with 1.0-inch gauge length.

C. QUALITY ASSURANCE

1. Specifications. Specifications for welding and post-weld vacuum annealing of T-111 alloy, SPPS No. 03-0025-00-A and SPPS No. 03-0037-00-A, respectively, were issued after approval by the NASA Program Manager. A new specification, SPPS No. 03-0021-00-A, outlining the cleaning and handling procedures to be used for the Corrosion Loop I (T-111) components and assemblies was completed and submitted to the NASA Program Manager for approval. Specifications for the installation and the calibration of tungsten-rhenium thermocouples, SPPS No. 03-0019-00-A and SPPS No. 03-0020-00-A, respectively, have been issued.

2. Bimetallic Joints. T-111 alloy to Type 316 SS joints are required on the NaK lines of the slack diaphragm pressure transducers. A tongue-in-groove joint equivalent to those used for Cb-1Zr to Type 316 SS joints on the Prototype Corrosion Loop was machined and brazed at 2160°F in accordance with specification SPPS No. 03-0008-00-B. Joint evaluation consisted of metallographic examination and hardness determinations.

The microstructure of the brazed joint is shown in Figure 4. Although excellent braze alloy flow was evident, it was observed that a separation occurred along the outside diameter of the stainless steel tongue, an area which is under tensile stress as a result of differential contraction during cooling. It was not possible to determine the exact cause of this separation. It could have occurred during cool-down from the brazing cycle or during the sectioning for metallographic examination. In the latter case, joint "hoop" stresses are released by cutting, possibly allowing the T-111 alloy member to revert to its original unstressed state. The more likely cause of this separation, which was not observed in similar Cb-1Zr alloy to stainless steel joints, involves the higher strength of T-111 alloy as compared to Cb-1Zr. In this case, failure at the braze interface might occur instead of deformation of the T-111 alloy member. It is planned to conduct additional brazing trials to resolve these uncertainties when additional T-111 tubing is obtained. It is anticipated that minor adjustments in brazing procedure and joint geometry will correct the defect found in this first trial joint.



F:.gure 4. Microstructure of T-111 Alloy to Type 316 SS Bimetallic Joint As Brazed with H-33 Braze Alloy (21Cr-21Ni-8Si-3.5W-0.4C-0.8B, Bal. Co.).

Etchant: 50 ml HNO_3, 20 ml H_2O, 30 gm NH_4F Mag: 50X

The hardness traverse across the braze joint, shown in Figure 5, indicated that no unusual interactions had occurred between the braze alloy and the joint components.

3. T-111 to Cb-1Zr Alloy Welding. Corrosion Loop I (T-111) will utilize the Cb-1Zr surge tanks which were used in the Prototype Corrosion Loop system. The transition to T-111 alloy will occur between the T-111 alloy loop fill tubes and the Cb-1Zr alloy surge tanks. Although the welding requirements for the T-111 and Cb-1Zr alloys are quite similar, the selection of a weld filler wire and post-weld annealing treatment required a brief welding study. Tungsten inert gas welds were made in both 0.040-inch and 0.080-inch thick sheets of T-111 and Cb-1Zr materials to provide samples required for the study of weldment bend characteristics after exposure to various post-weld thermal cycles. Those welds between the 0.040-inch thick sheet were made by the automatic weld process without filler additions, while those between the 0.080-inch stock were made by the manual process using either Cb-1Zr or T-111 filler material. One- and two-hour post-weld annealing treatments at 2200°F, 2300°F and 2400°F were performed on several automatic weld samples, while additional samples were aged at 1500°F for 50 hours after welding. Other samples of these automatic welds were then exposed to both the various annealing treatments and the 1500°F age treatment. Subsequent bend testing (1T bend radius) at room temperature and -100°F indicated that optimum weld stability was associated with the 2300°F and 2400°F treatments, as shown in Table XII. Hence, these overaging temperatures were selected for post-weld treatments on the manual weld samples. Samples of the manual welds were given one-hour heat treatments at 2300°F and 2400°F, aging treatments of 1500°F for 50 hours, while others were both overaged and reaged at these times and temperatures. The bend data (Table XII) for these welds pointed out that the selected post-weld annealing treatments successfully stabilized those welds prepared with Cb-1Zr filler; while samples made with T-111 filler were still subject to additional aging during the 1500°F/50-hour reage cycle. Hardness testing was also performed on selected samples to provide additional clarification of the bend test results. The hardness data obtained are also summarized in Table XII.

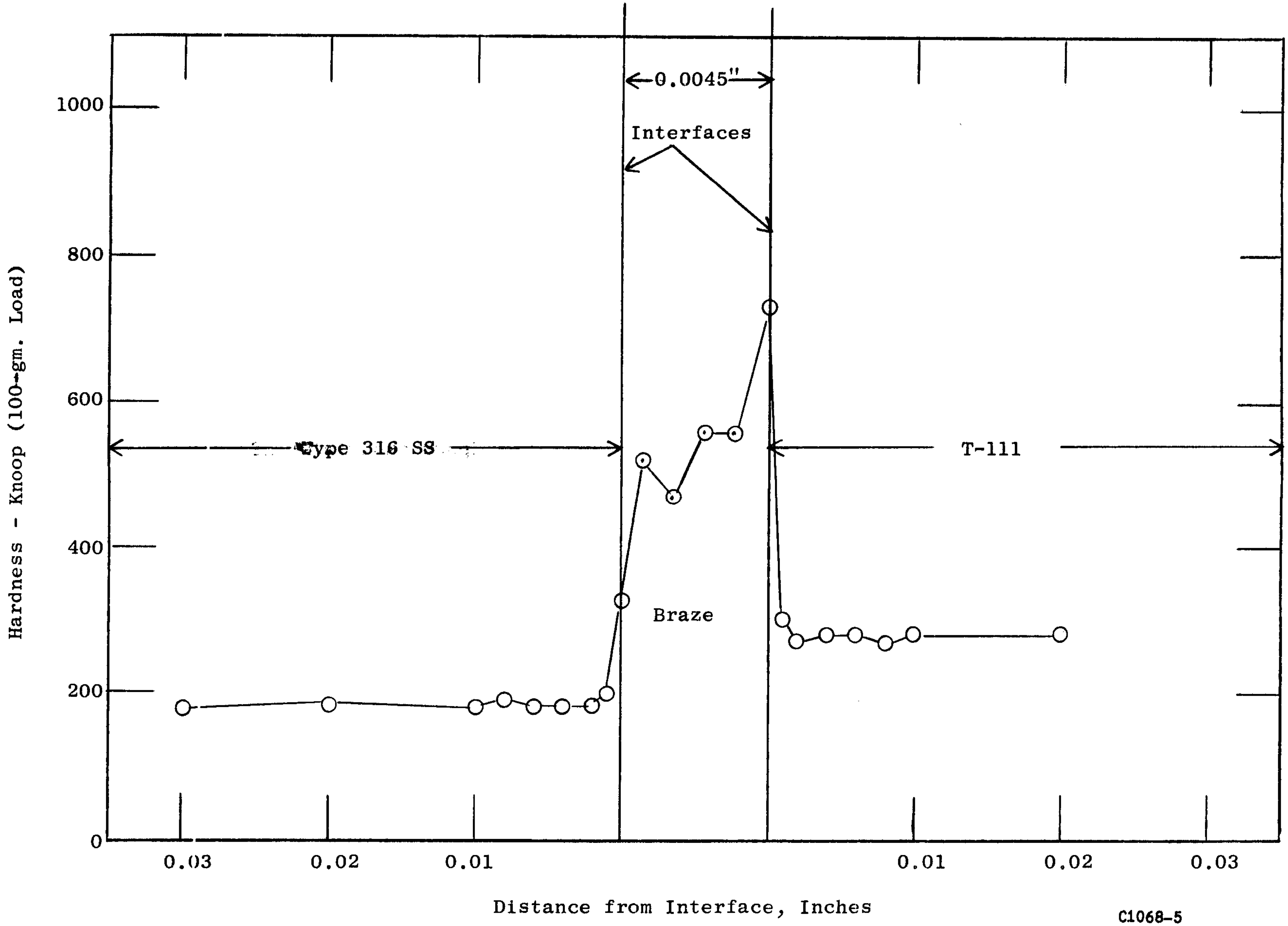


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Figure 5. Hardness Traverse Across T-111 to 316 SS Brazed Joint (As-Brazed) H-33 Braze Alloy: 21Cr-21Ni-3.5W-8.0Si-0.8B-0.4C-Bal. Co.

TABLE XII. SUMMARY OF BEND AND HARDNESS DATA OF AGED T-111 TO Cb-1Zr WELD SPECIMENS

	Filler Material	Heat Treat Condition	Bend Test Results(1) Room Temp.	Bend Test Results(1) -100°F	Hardness, Knoop (100-gm. Test Load) T-111 HAZ(2)	Hardness, Knoop (100-gm. Test Load) Weld	Hardness, Knoop (100-gm. Test Load) Cb-1Zr HAZ(2)
I.							
Automatic Welds (0.040-inch thick sheet)	None	1500°F/50 hr	B(70°-90°) (Cb HAZ)		273	256	241
		2200°F/1 hr	D				
		2200°F/2 hr	D				
		2300°F/1 hr	D		250	142	123
		2300°F/2 hr	D				
		2400°F/1 hr	D		227	136	115
		2200°F/1 hr + 1500°F/50 hr	D	B(70°-90°) (Cb HAZ)			
		2200°F/2 hr + 1500°F/50 hr		D			
		2300°F/1 hr + 1500°F/50 hr	D	D	281	150	115
		2300°F/2 hr + 1500°F/50 hr	D	D			
		2400°F/1 hr + 1500°F/50 hr	D	D	277	153	129
II.							
Manual Welds (0.080-inch thick sheet)	T-111	1500°F/50 hr	B(5°-10°weld)		265	320	230
	T-111	2300°F/1 hr	D				
	T-111	2400°F/1 hr	D				
	T-111	2300°F/1 hr + 1500°F/50 hr	B(10°-20°weld)		263	196	115
	T-111	2400°F/1 hr + 1500°F/50 hr	B(10°-20°weld)		235	250	121
	Cb-1Zr	1500°F/50 hr	B(30°-40°) (Cb HAZ)		240	218	224
	Cb-1Zr	2300°F/1 hr	D				
	Cb-1Zr	2400°F/1 hr	D				
	Cb-1Zr	2300°F/1 hr + 1500°F/50 hr	D		277	144	138
	Cb-1Zr	2400°F/1 hr + 1500°F/50 hr	D		219	139	136

(1) All specimens bent over a 1T radius
D indicates ductile - full bend through 90° to 105° without fracture
B indicates brittle - numbers in parenthesis indicate approximate bend angle at failure

(2) HAZ = Heat Affected Zone.

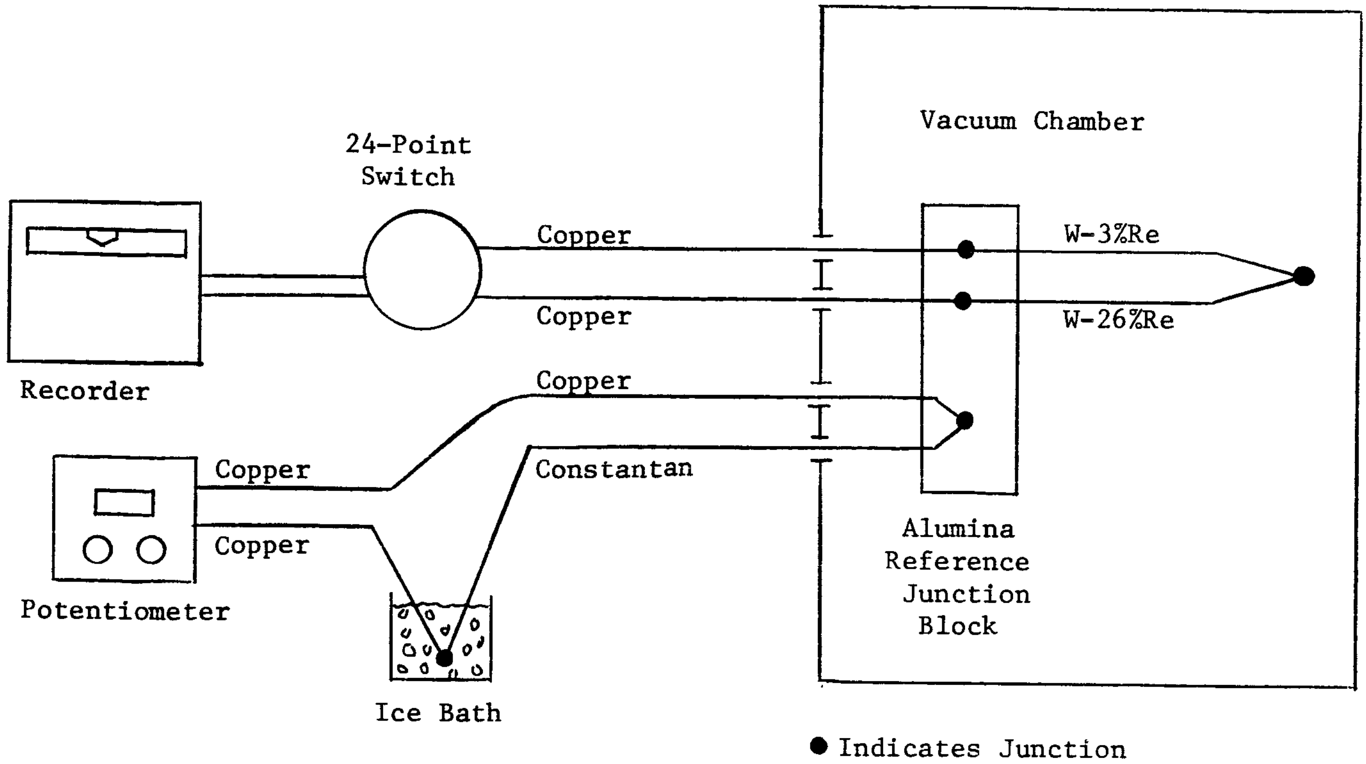
In summary, the necessary joint between the Cb-1Zr surge tank and the T-111 fill lines may be made by manual TIG welding techniques, using Cb-1Zr alloy as filler material. Post-weld annealing at 2300-2400°F for 1 hour has been shown to produce good thermal stability and excellent room temperature ductility of the weldments.

4. Thermocouple Circuitry. The Prototype Corrosion Loop thermocouple circuit, shown in Figure 6, included a thermocouple reference block located inside the vacuum chamber; thereby reducing the required length of the high cost tungsten-rhenium wire. Copper wire was substituted to complete the thermocouple circuit from the reference junction block to the multipoint recording potentiometer. Each loop temperature measurement, using this circuitry, required the manual addition of a correction factor associated with the temperature of the reference junction block inside the vacuum chamber.

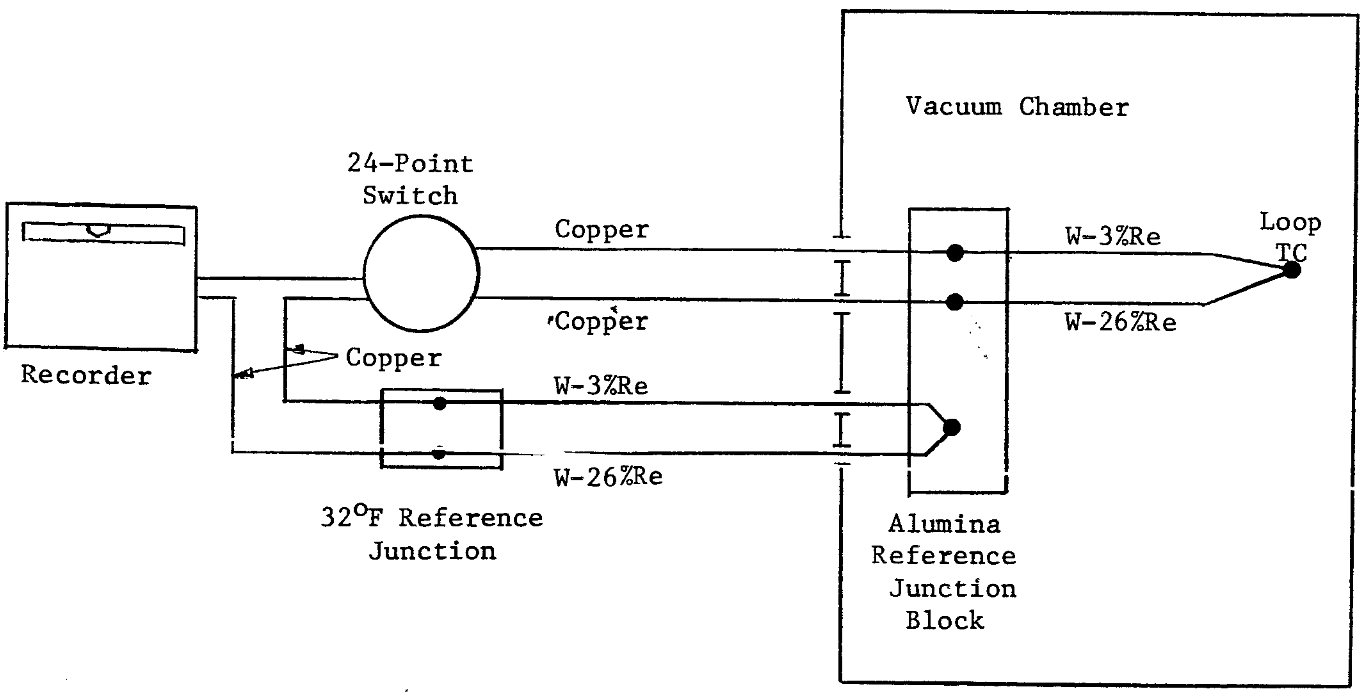
The proposed circuit for Corrosion Loop I (T-111) incorporates a thermoelectrically cooled, ice point calibration standard and automatically adds the reference junction block temperature correction factor, thereby directly recording the loop temperatures (Figure 6).

D. LOOP DESIGN

The major portion of the Corrosion Loop I (T-111) design drawings have been approved by the NASA Program Manager.



Prototype Corrosion Loop Thermocouple Circuit



Proposed Corrosion Loop I Thermocouple Circuit

Figure 6. Corrosion Loop Thermocouple Circuitry.

IV. FUTURE PLANS

A. Monitoring the fabrication of refractory alloy materials for Corrosion Loop I (T-111) will continue with special emphasis placed on meeting the new schedules.

B. Fabrication of the Lithium Metal Purification and Handling System for Corrosion Loop I (T-111) will continue.

C. Additional T-111 to Type 316 SS bimetallic joint specimens will be brazed and evaluated.

PUBLISHED REPORTS

Quarterly Progress Reports	For Quarter Ending
Report No. 1 (NASA-CR-54477)	July 15, 1965
Report No. 2 (NASA-CR-54845)	October 15, 1965
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Attention: Librarian

TRW Inc.
TRW Systems Group
One Space Park
Redondo Beach, California 90278
Attn: Dr. H. P. Silverman

Sandia Corporation
Aerospace Nuclear Safety Division
Sandia Base
Albuquerque, New Mexico 87115
Attn: Mr. A. J. Clark (3)