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ADVANCED REFRACTORY ALLOY CORROSION LOOP PROGRAM

Quarterly Progress Report No. 2
For Quarter Ending October 15, 1965

By
R. W. HARRISON
and
E. E. HOFFMAN

prepared for
NATIONAL AERONAUTICS AND SPACE ADMINISTRATION
CONTRACT NAS 3-6474

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 2

Covering the Period
July 15, 1965 to October 15, 1965

Edited by

R. W. Harrison

Project Metallurgist

Approved by

E. E. Hoffman

Manager, Corrosion Technology

Prepared for

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

Lewis Research Center

Under Contract NAS 3-6474

October 29, 1965

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

SPACE POWER AND PROPULSION 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 responsile 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.

Refractory Alloy Procurement - R. G. Frank

Quality Assurance and Reliability - G. L. Hilbrich

CONTENTS

LIST OF ILLUSTRATIONS

LIST OF TABLES

ADVANCED REFRACTORY ALLOY CORROSION LOOP PROGRAM

I INTRODUCTION

This reports covers the period, from July 15, 1965 to October 15, 1965, 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 BTU/hr ft^2

II SUMMARY

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

On August 6, 1965, notification was received from the NASA Program Manager that T-111 was selected as the containment alloy and that the Mo-TZC and Nb Cb-132M alloys were selected for inclusion in the turbine simulator for this program. The alloy T-111 is of primary interest to NASA for application in large facilities for developing components for space power systems.

All refractory alloy materials for loop fabrication have been ordered and should be received about March 15, 1966.

The lithium from Lithium Corporation of America, Bessemer City, North Carolina, has been received.

Revisions in the Prototype Corrosion Loop assembly drawings were made based on experience gained in fabrication, installation and operation of this loop. The changes were reviewed with the NASA Program Manager and will be employed in fabrication of Corrosion Loop I following approval of the incorporated revisions by NASA.

Specifications for cleaning, welding and heat treating loop components are being prepared.

III PROGRAM STATUS

A. Material Procurement

Notification was received on August 6, 1965, from the NASA Program Manager that the T-111 alloy had been selected as the containment alloy for this program. The nozzles and blade specimens of the turbine simulator for the T-111 alloy loop will be made of Mo-TZC alloy and Cb-132M alloy. Subsequently, all refractory alloy material required for Corrosion Loop I fabrication was ordered. A list of the shapes, sizes, and weights are given in Table I.

In the production of T-111 alloy ingot by Fansteel Metallurgical Corporation, Muskogee, Oklahoma, tantalum and tungsten powders are blended in one large charge. The powders are subsequently hydrostatically compacted and sintered into electrode bars. Consolidation of the T-111 alloy is accomplished first by double EB melting the Ta-8W sintered bars into 5-inch ingots. The hafnium addition to the alloy is accomplished by welding hafnium strips to the full length of the electrode produced from the 5-inch EB ingots and this electrode is vacuum arc melted, producing a 7-3/4-inch diameter T-111 alloy ingot. To-date all melting of the T-111 alloy has been completed and the ingots have been shipped to Canton Drop Forge, Canton, Ohio, for extrusion in stainless steel cans.

The Mo-TZC alloy will be obtained from two sources. Climax Molybdenum Corporation, Lansing, Michigan, and General Electric Company, Lamp Metals and Components Department, Cleveland, Ohio. Two vendors were selected to avoid program delay in the event of processing difficulties. On October 4, 1965, melting of a 9-inch diameter Mo-TZC ingot was completed at Climax. The cropped and trimmed ingot, measuring approximately 7 inches in diameter was vacuum annealed at 2800°F for 1 hours, and will be canned in molybdenum and shipped to Allegheny

TABLE I. REFRACTORY ALLOY REQUIREMENTS FOR CORROSION LOOP I

	Weight, lbs.	
A. Test Alloy (T-111)		
1. Rod		
0.125 inch dia.	15	
0.250 inch dia.	1	
0.500 inch dia.	6	
0.625 inch dia.	3	
1.000 inch dia.	21	
1.125 inch dia.	5	
1.500 inch dia.	7	
2.000 inch dia.	82	
2.500 inch dia.	94	
3.125 inch dia.	75	
		309
2. Bar		
1.0 inch x 1.0 inch	8	
1.0 inch x 2.0 inch	116	
		124
3. Wire		
0.062 inch dia.	6	
0.094 inch dia.	8	
		14
4. Sheet/Foil/Plate		
0.005 inch x 3.5 inch	0.6	
0.006 inch x 3.5 inch	0.2	
0.040 inch x 12.0 inch	21	
0.500 inch x 6.125 inch	22	
		43.8
5. Tube/Pipe		
2.35 inch OD x 0.375 inch wall	40	
2.50 inch OD x 0.500 inch wall	50	
3.00 inch OD x 0.375 inch wall	50	
3.25 inch OD x 0.250 inch wall	40	
		180

TABLE I (Cont'd)

	Weight, lbs.	
5. Tube/Pipe		
3.25 inch OD x 0.500 inch wall	73	
1.00 inch OD x 0.100 inch wall	95	
0.375 inch OD x 0.065 inch wall	70	
0.375 inch OD x 0.008 inch wall	3	
3/4 inch Schedule 80 pipe	9	
		250
B. Tantalum		
1. Rod		
0.250 inch dia.	1	
0.625 inch dia.	2	
1.125 inch dia.	5	
		8
2. Bar		
1.00 inch x 1.00 inch	9	
0.500 inch x 1.00 inch	8	
		17
3. Wire		
0.020 inch dia.	0.3	
		0.3
4. Sheet/Plate		
0.032 inch x 0.75 inch	0.2	
0.062 inch x 2.125 inch	3	
0.250 inch x 4.00 inch	86	
		89.2
C. Cb-1Zr Alloy		
1. Rod		
0.5 inch dia.	7	
0.625 inch dia.	1	
1.25 inch dia.	9	
		17

TABLE I. (Cont'd)

		Weight, lbs.
2. Wire		
0.062 inch dia.	2	
0.094 inch dia.	3	
		5
3. Sheet/Foil/Plate		
0.002 inch x 0.5 inch	30	
0.002 inch x 3.50 inch	10	
0.005 inch x 8.00 inch	15	
0.0175 inch x 12.0 inch	9	
0.030 inch x 24.0 inch	14	
0.125 inch x 4.0 inch	2	
0.250 inch x 6.0 inch	17	
		97
4. Tube		
2.75 inch OD x 0.125 inch wall	15	
		15
D. Turbine Alloy		
TZC		
Rod		
2.0 inch dia.	55	
1.0 inch dia.	11	
	66	
Plate		
1.375 inch thick	35.0	
0.75 inch thick	7.5	
	42.5	
Cb-132M		
Rod		
2.0 inch dia.	28.3	
1.0 inch dia.	6.5	
	34.8	
		143.3

Ludlum, Watervliet, New York. Three 6-inch diameter Mo-TZC ingots are to be melted at General Electric by November 8, 1965. Universal Cyclops Corporation, Bridgeville, Pennsylvania, has scheduled receipt of an EB and arc melted Cb-132M alloy ingot from Wah Chang Corporation, Albany, Oregon, by November 1, 1965, for further processing.

All materials necessary for fabrication of Corrosion Loop I are expected to be on hand by March 15, 1966.

T-111 alloy and Cb-1Zr alloy bellows were received from Mini-Flex Corporation, Lawndale, California. Subsequently, the bellows were leak checked, inspected, and classified as to quality.

Stereographic visual examination of the valve bellows at a magnification of 30X indicated a less coarse surface texture on T-111 alloy bellows than that of the Cb-1Zr alloy bellows. Photomicrographs of these bellows are shown in Figures 1 and 2. This type of surface texture is typical of ductile metals following forming operations which require extensive deformation. Metallographic examination of the cross section of the convolutions indicated good uniformity of wall thickness in these bellows. As an example, the wall thickness variation between the apex and the sides of convolutions in a T-111 alloy bellows was measured as less than 0.001 inch, Figure 3. The grain size in the convolutions corresponds to ASTM 7-8. A series of T-111 alloy bellows blanks representing the various stages in bellows fabrication are depicted in Figure 4. The axial deflections of two T-111 bellows and a Cb-1Zr bellows as a function of load determined in similar manner previously employed with the trail bellows (1), are

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(1) Harrison, R. W. and Hoffman, E. E., "Advanced Refractory Alloy Corrosion Loop Program," Quarterly Progress Report No. 1 for Period Ending July 15, 1965, NASA Contract NAS 3-6474, NASA-CR-544477.

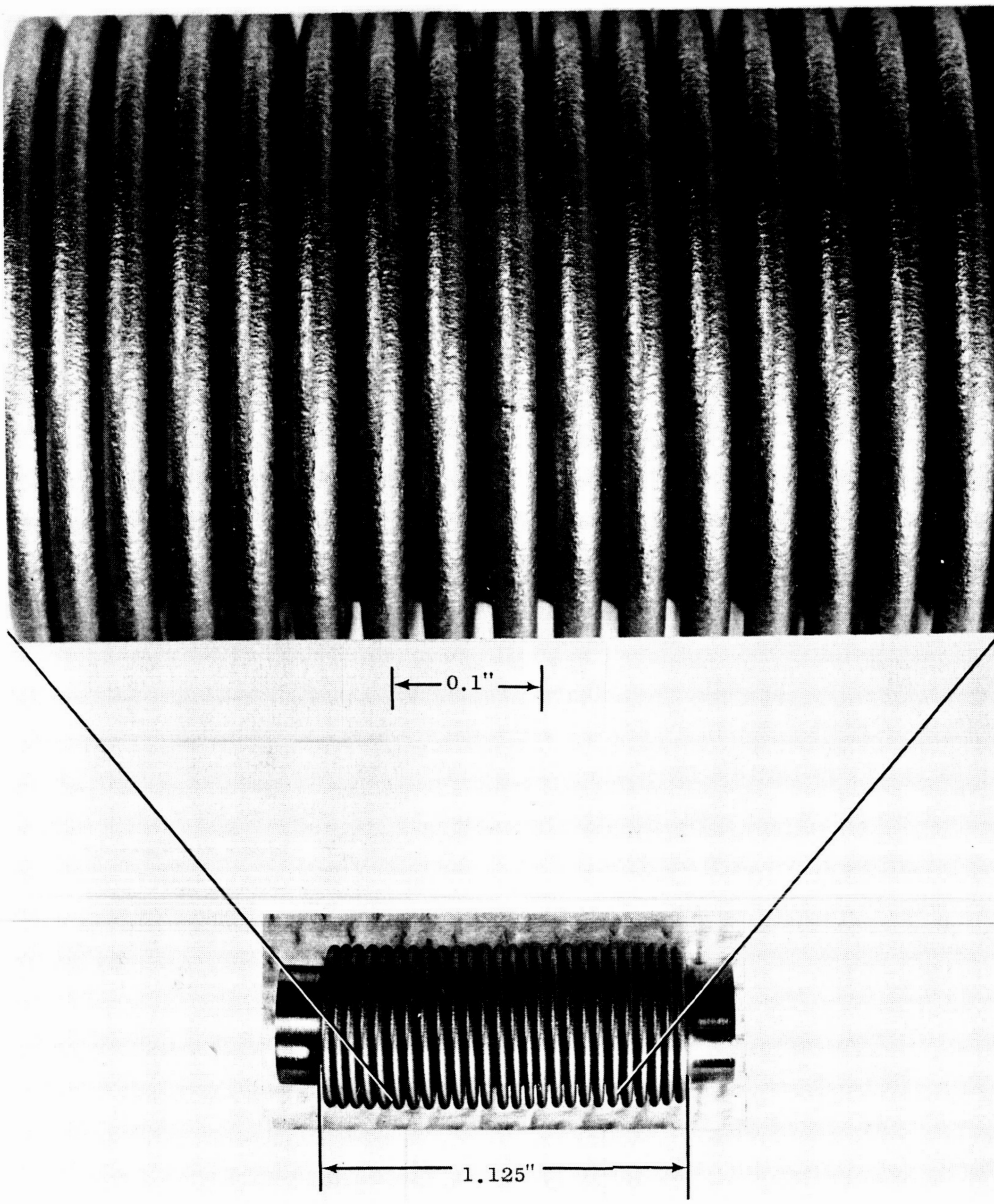


Figure 1. T-111 Alloy Valve Bellows. (Top - C65082015)
(Bottom - C65082006)

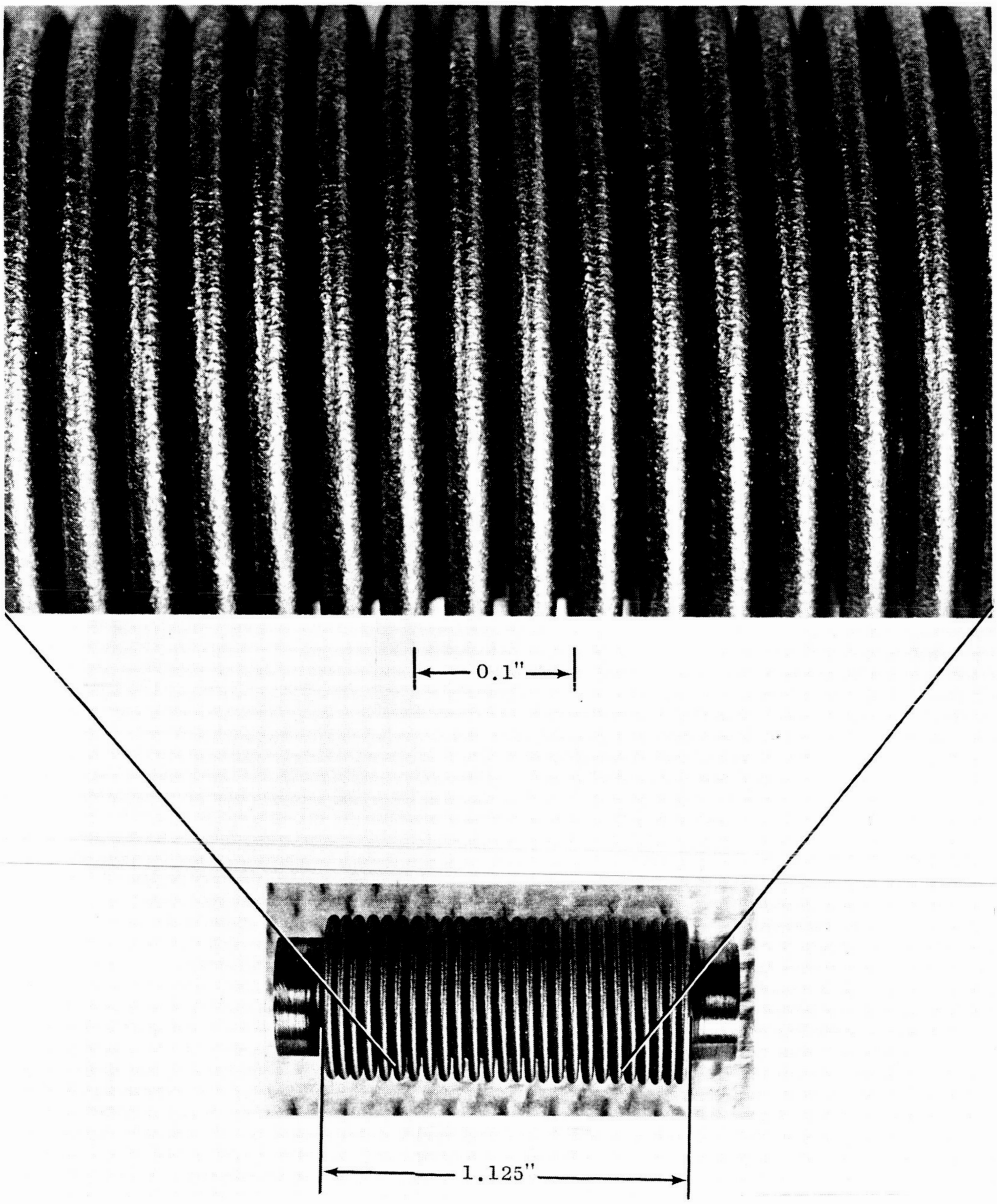


Figure 2. Cb-1Zr Alloy Valve Bellows. (Top - C65082014)
(Bottom - C65082007)

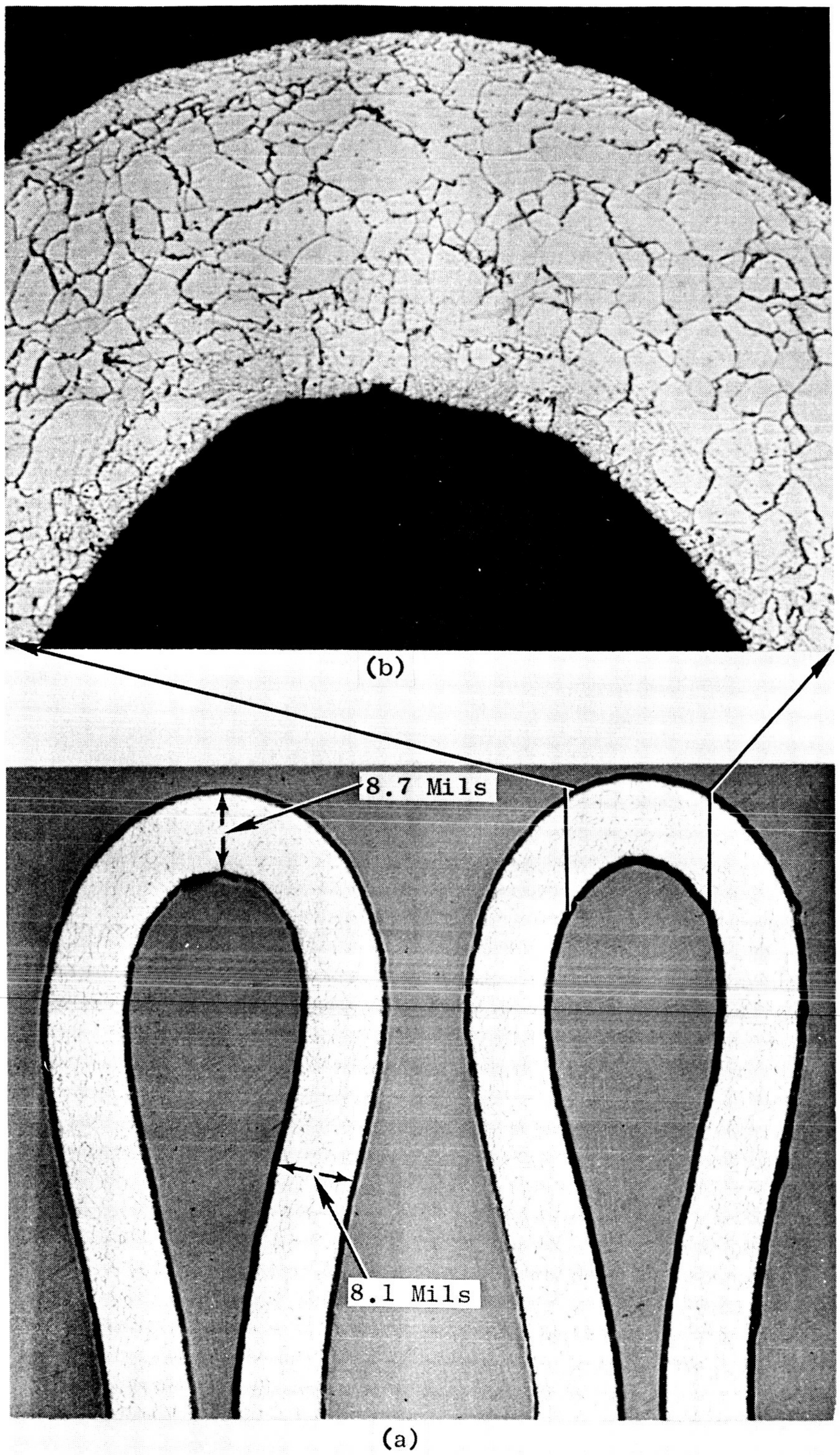


Figure 3. (a) A Transverse Section of T-111 Alloy Bellows Convolutions Showing the Uniformity of the Wall Thickness (Orig. A830213). (b) A Transverse Section at the Apex of a Convolution Indicating an ASTM Grain Size 7-8 (Orig. A830212).

Etchant: 40%HNO_3-40%HF-20%HCL Orig. Mag: (a) 50X (b) 250X

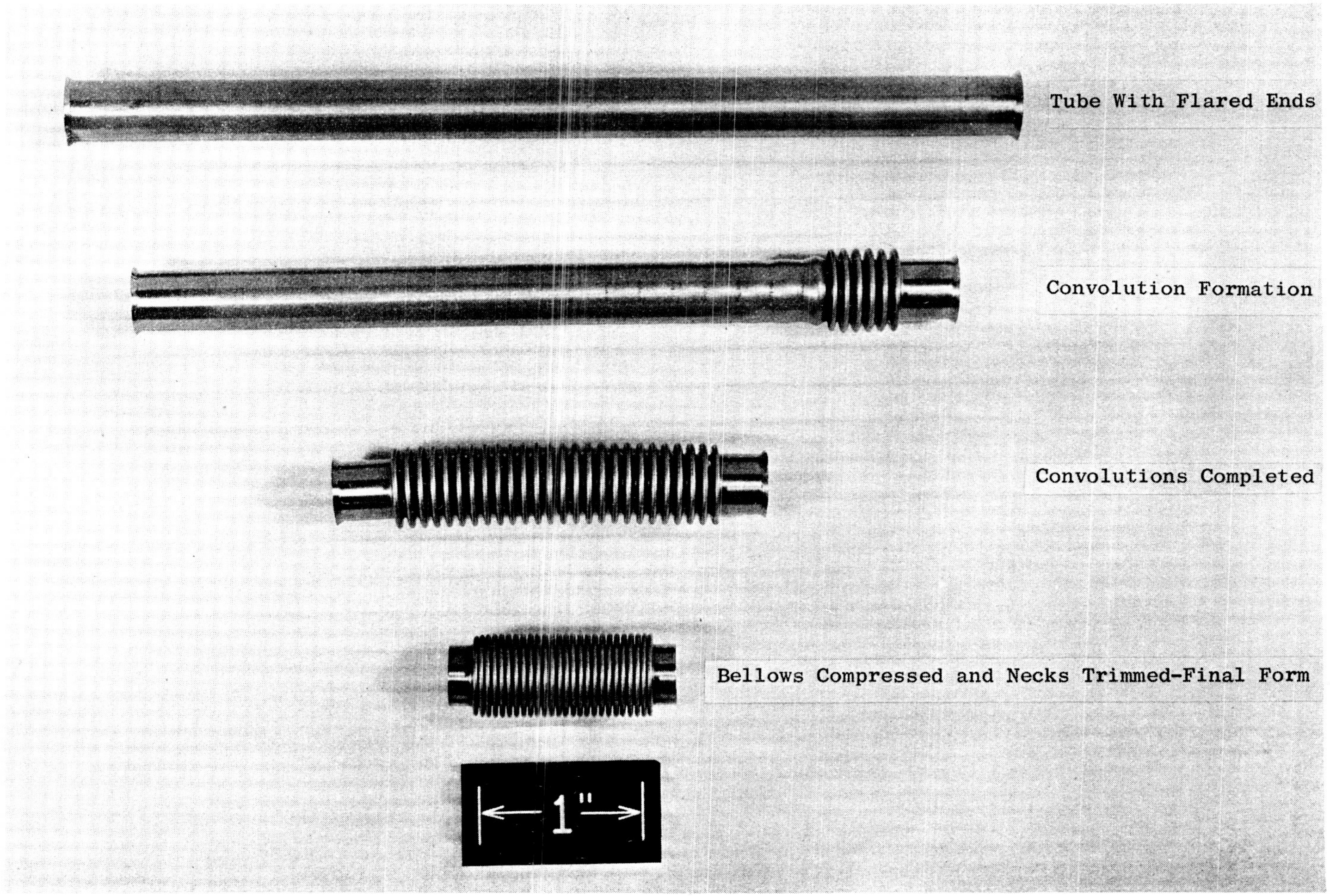


Figure 4. Stages in the Formation of T-111 Alloy Bellows. (C65082016)

presented in Table II. No permanent set was observed in these room temperature tests of the bellows in the 0.1-inch travel necessary for valve operation. Sufficient T-111 alloy bellows of this type are on hand and will be employed in the loop isolation and metering valves.

All parts for the vacuum system, argon manifold, and hot trap for the lithium purification system have been ordered. All of the Cb-1Zr for the lithium still is on hand. The majority of the parts have been ordered and received including the liquid metal level probe from Mine Safety Appliance Research Corporation, Callery, Pennsylvania, and the parts for the hot trap.

B. Alkali Metal Purification

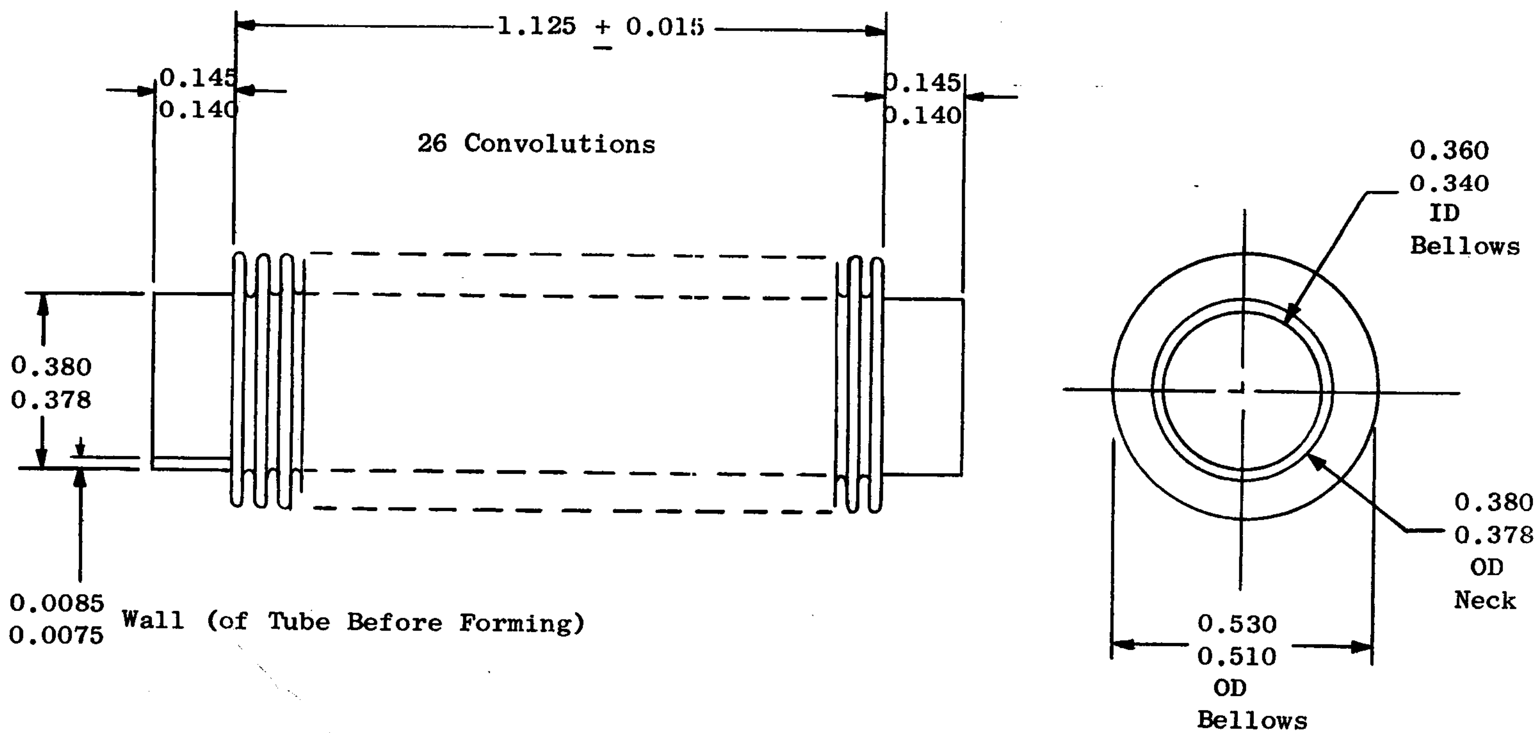
A special stainless steel shipping container for lithium, Figure 5, was fabricated, outgassed, leak checked (no leak detected at a detection limit of 5×10^{-11} std. cc air/sec) and back filled with high purity argon. This container was sent to the Lithium Corporation of America, Bessemer City, North Carolina, where it was filled with 50 pounds of high purity lithium and returned to General Electric.

The lithium to be used in Corrosion Loop I will be purified in a titanium lined, zirconium gettered hot trap of the type which was used to purify potassium for the Prototype Loop. It will be further purified by distillation at reduced pressure in a distillation apparatus which is essentially a duplicate of the one used to distill potassium for the Prototype Loop. Certain minor revisions will be incorporated into the lithium still. One is that two "I" tubes will be added to the still pot to determine liquid levels in the still pot during distillation. These tubes will indicate the desired upper and lower lithium levels by abrupt changes in electrical resistance. Provision will also be made to measure

TABLE II. ROOM TEMPERATURE COMPRESSIVE PROPERTIES OF T-111 ALLOY AND AND Cb-1Zr ALLOY VALVE BELLOWS

Alloy Specimen	Elastic Spring Rate Lbs/Inch	Total Deflection at Yield Inch	Load at Yield Lbs	Permanent Set In/Full 0.1 Inch Deflection*	Load Required for Full 0.1 Inch Deflection* Lbs
T-111-1	428	0.140	60	0	42.8
T-111-2	443	0.131	58	0	44.3
Cb-1Zr-1	185	0.112	20.8	0	18.5

* 0.1-Inch Deflection Normal Travel Range of Valve.



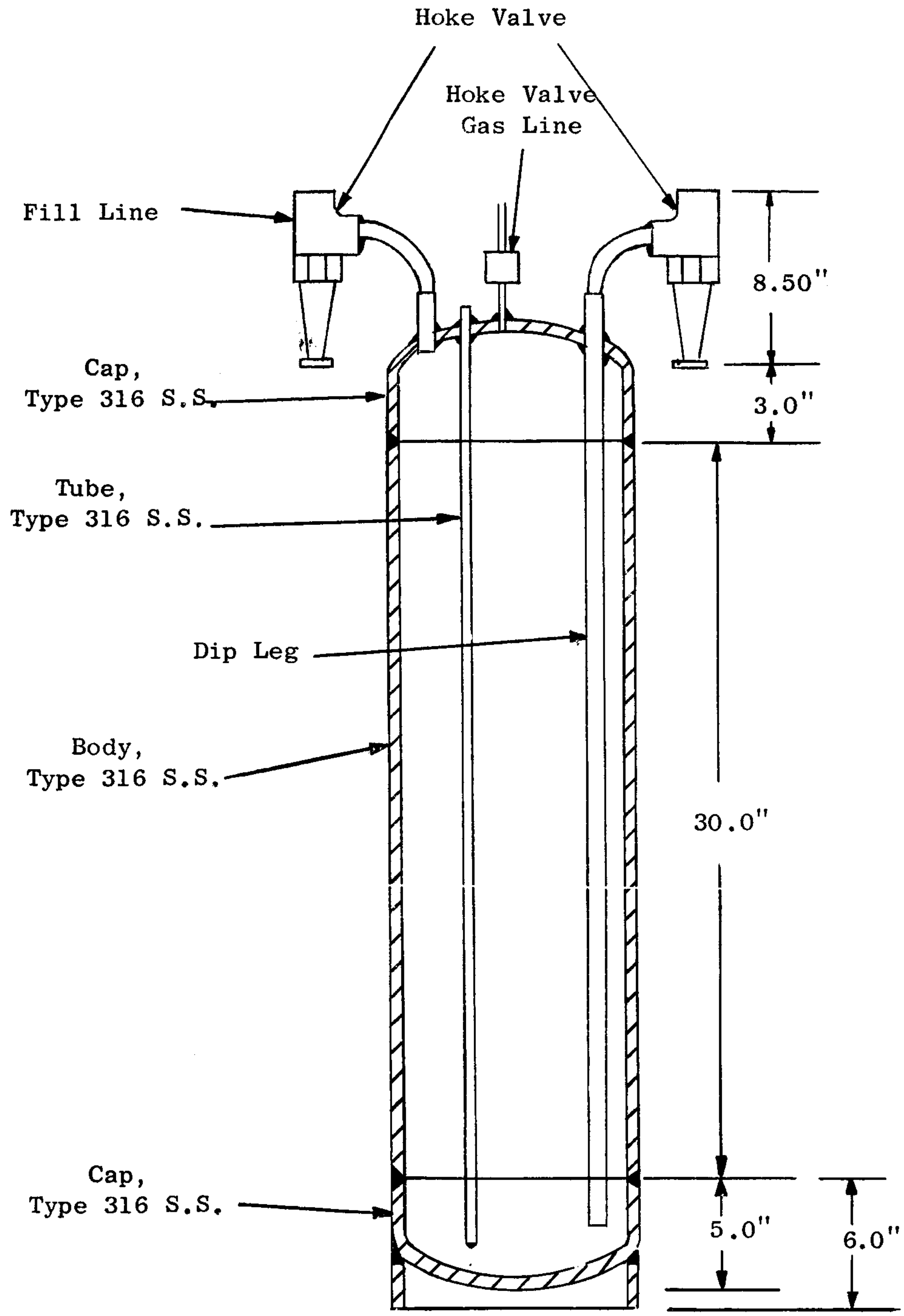


Figure 5. Fifty-Pound Capacity Lithium Shipping Container.

the lithium level in the still received with an inductive type liquid level probe which has been purchased from Mine Safety Appliance Research Corporation, Callery, Pennsylvania. This probe will also be used to measure lithium levels in the hot trap.

The Prototype Loop potassium purification and transfer system drawings are being revised in preparation for submittal to NASA for approval prior to initiation of fabrication of the lithium purification system.

Many of the parts for the purification system have been received and fabrication has started on the hot trap. The titanium liner for this hot trap has been completed.

C. Loop Design

The assembly drawing of the Corrosion Loop I was completed during the past quarter. A schematic drawing of the loop reflecting the location of various components and the design conditions is shown in Figure 6. The Prototype Loop configuration was modified to reflect the experience gained in the fabrication and operation of the Prototype Loop. In addition to the design changes in selected components, regrouping of several subassemblies is in progress to facilitate the welding and heat treating of the completed assembly. Particular care will be given to assure sufficient bends are incorporated in the loop design to allow for thermal expansion. Completion of the design phase of this task is scheduled during the next quarter.

The changes which are being made in the Prototype Corrosion Loop design are as follows:

1. Relocation of Primary EM Pump - During the installation of the Prototype Loop, some difficulty was experienced in inserting the primary and secondary EM pump ducts in their respective vacuum tank

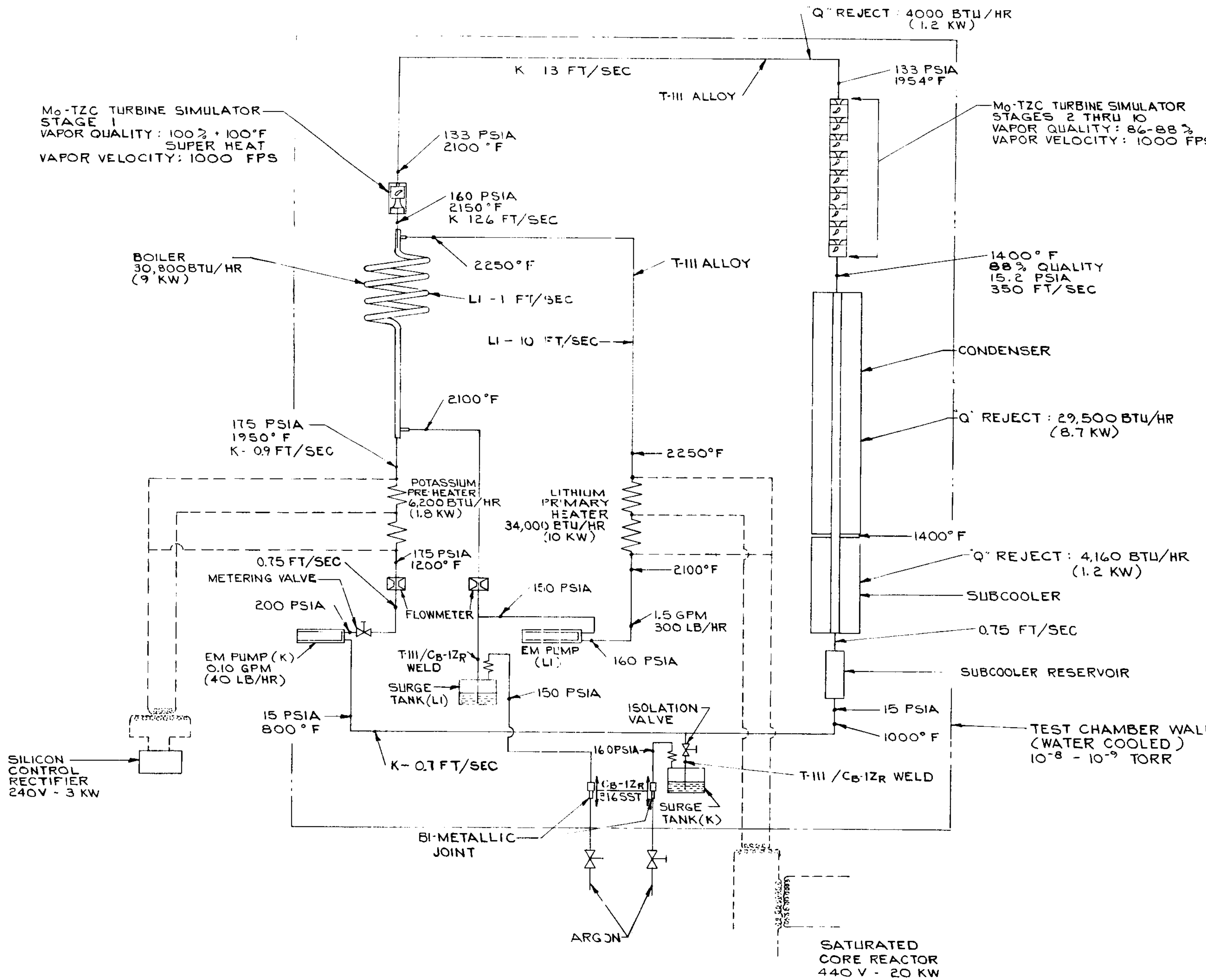


Figure 6. Schematic Diagram of Corrosion Loop I.

ports because of the 90° angle between them. Moving the primary EM pump from 90° to 45° from the secondary EM pump will eliminate this installation problem for Corrosion Loop I.

2. <u>Boiler Plug Length</u> - During the start-up and early operation of the Prototype Corrosion Loop, it was observed that two features of the test system which were most responsible for the boiling stability were the pressure drop across the metering valve and the 12-inch long plug section located in the boiler entrance region. The boiler plug is a flow swirling device formed by wrapping and tack welding a 1/16-inch diameter wire on a 1/8-inch diameter rod using a 1-inch pitch. This plug is located in the first 12 inches of the 1/4-inch ID boiler tube.

   Although the Prototype Loop has been observed to operate in a stable manner with the bulk of the boiling occurring in various locations along the boiler length, operation is most stable when the boiling is occurring principally in the plug section. Movement of the boiling region was observed during test start-up due to changes in the test conditions and during routine operation as a result of momentary power fluctuations. Periods of substantial operational instability are associated with the movement of the boiling location to a new region of the boiler. More substantial perturbations of the system conditions; sodium flow, sodium temperature, potassium flow, etc.; are required to dislodge the boiling region from the plug section than to cause movement of this region from other portions of the boiler.

The observations cited above indicate that lengthening of the plug portion of the boiler would result in more stable loop operation during start-up and during periods when loop test conditions may change as a result of changes in loop performance or power fluctuations. For this reason the plug will be increased from 12 to 18 inches for Corrosion Loop 1.

A review of the heat transfer characteristics of the plug section of the prototype boiler indicates that lengthening the plug from 12 to 18 inches would not significantly change the overall performance of the boiler. The vapor quality at the exit of the plug would only increase from 72 to 77% as shown in Figure 7.

Although the addition of the 6 inches to the plug length will increase the heat flux in this section compared to the comparable length with no plug, the average heat flux in the boiler plug will decrease from 295,000 BTU/hr ft^2 for the 12-inch plug to 240,000 BTU/hr ft^2 for the 18-inch plug.

3. Sodium Heat Length - There are indications of possible induction heating of the insulating foil on the Prototype Loop sodium heater. To minimize this minor problem, the number of turns in each coil of the lithium heater was increased from 2-1/2 to 3-1/2 to increase the overall electrical resistance and reduce the current required for the design power input. The increased length and the higher electrical resistivity of the T-111 alloy with respect to Cb-1Zr will increase the overall resistance of the heater by 25%.

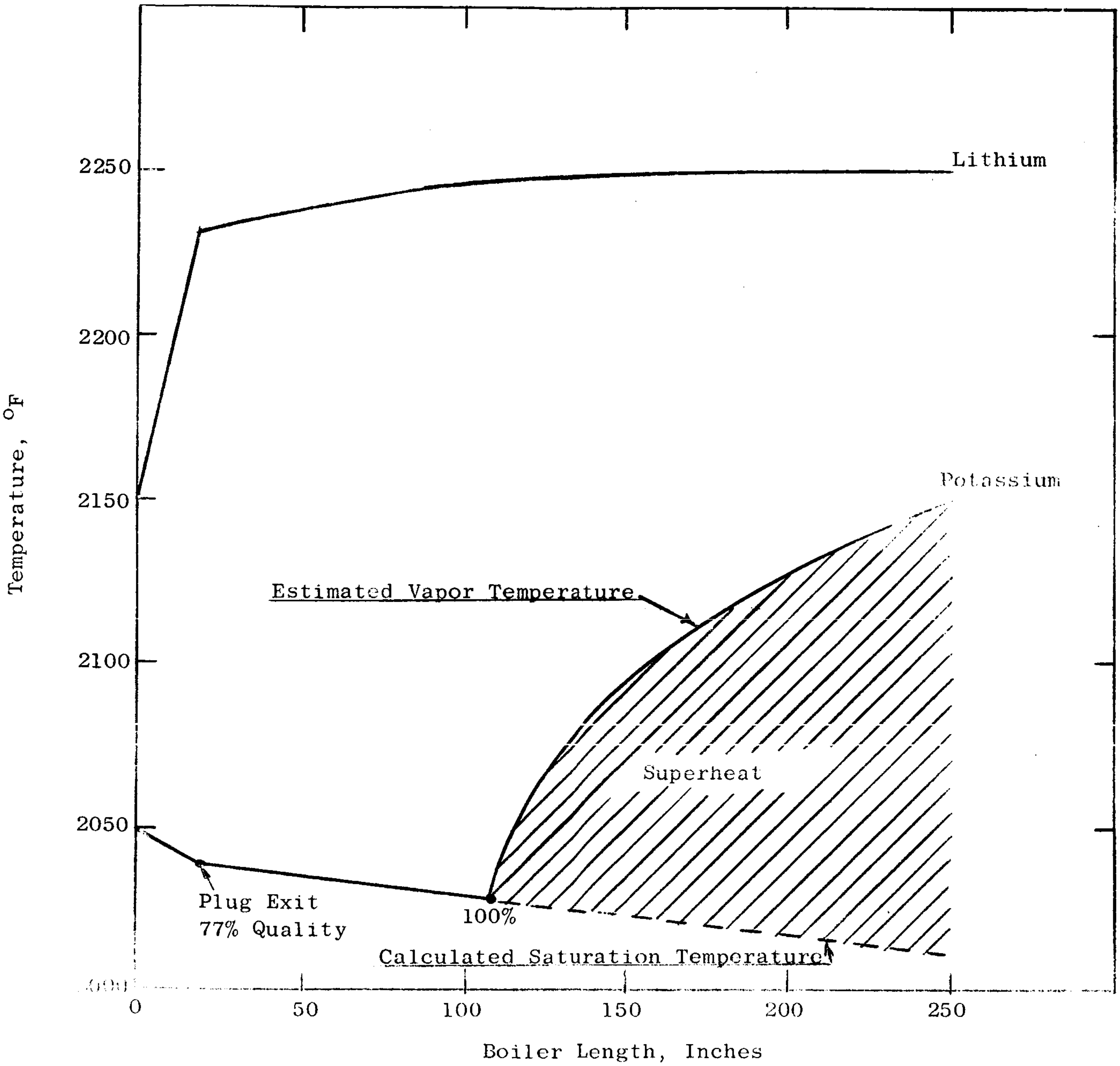


Figure 7. Estimated Temperature Distribution and Quality as a Function of Boiler Length for Corrosion Loop I.

4. <u>Pressure Transducer</u> - An additional slack diaphragm pressure transducer was added downstream of the metering valve and adjacent to the original stress diaphragm pressure transducer. The slack diaphragm pressure transducer is less sensitive to zero shift and should provide a better indication of changes in the pressure drop across the boiler and the metering valve. The fast response pressure transducer has proven to be an extremely fine instrument to detect stability and will be retained.

5. <u>Subcooler Reservoir</u> - The reservoir was redesigned in an effort to remove particles that might be present in the flow circuit by diverting the potassium through a double reversed, low velocity flow path. An increase in the pressure drop as a function of time during one period of Prototype Loop operation indicated that ~~small solid~~ particles may have been partially restricting the 0.004-inch diameter annulus of the metering valve.

6. <u>Metering Valve</u> - A redesign of the plug to provide better flow control at a flow rate of 40 lbs per hour of potassium was made by reducing the include angle of the plug control surface from 75° to 10°. Water flow tests to check the flow characteristics of the new design will be made when the new plug becomes available. The pinion gear of the valve drive was changed from a 6 to 10 tooth gear for smoother operation at the higher torque level. A change in valve gear material from stainless steel to hardened steel on cemented carbides is also being considered to reduce the galling tendency.

7. Condenser - Distortion and out-of-roundness were observed in the Prototype Corrosion Loop condenser tube after TIG welding the 1/4-inch thick fins to the 0.126-inch wall, 3/8-inch Schedule 80 pipe. For Corrosion Loop I, the distortion will be eliminated by replacing the 3/8-inch pipe with a 1 x 2-inch diamond shaped bar which has been center gun-drilled to form a 0.42-inch diameter hole in the 60-inch long condenser. This design change will also move the weld zone away from the flow passage and thereby simplify the corrosion evaluation of this component.

D. Quality Assurance

A specification for the cleaning and handling of components and assemblies for alkali metal service has been prepared and will be submitted to the NASA Project Manager for approval. Specifications for welding and heat treating T-111 alloy components are also being prepared. Following receipt of T-111 alloy tubes, pickling tests will be conducted to determine pickling rates and the effect of pickling on surface quality. As-received and pickled specimens will be evaluated by metallographic examination.

IV FUTURE PLANS

A. The progress in the fabrication of the loop materials will be monitored.

B. The drawing revisions for the lithium distillation and transfer systems will be completed and submitted for NASA.

C. Operating procedures for the new liquid metal level probe will be developed.

D. The design modifications for Corrosion Loop I will be essentially completed.

E. Specifications for cleaning, welding, and heat treating loop components will be completed and submitted to the NASA Project Manager for approval.

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