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ADVANCED REFRACTORY ALLOY CORROSION LOOP PROGRAM

Quarterly Progress Report No. 9 For Quarter Ending July 15, 1967

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

prepared for
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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 9

Covering the Period
April 15, 1967 to July 15, 1967

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Prepared for
NATIONAL AERONAUTICS AND SPACE ADMINISTRATION
Lewis Research Center

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NASA - Lewis Research Center
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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 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 H. Bradley.

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

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

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ADVANCED REFRACTORY ALLOY CORROSION LOOP PROGRAM

I. INTRODUCTION

This report covers the period from April 15, 1967 to July 15, 1967. The primary task of this program is to fabricate, operate for 10,000 hours and evaluate a T-111 Rankine System Corrosion Test Loop. Materials for evaluation include the containment alloy, T-111 (Ta-8W-2Hf) and the turbine candidate materials Mo-TZC and Cb-132M which are located in the turbine simulator of the two-phase potassium circuit of the system. The loop design will be similar to the Cb-1Zr Rankine System Corrosion Loop; a two-phase, forced convection, potassium corrosion test loop which has been developed under Contract NAS 3-2547. Lithium will be heated by direct resistance in a primary loop. Heat rejection for condensation in the secondary potassium 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 ft²
- h. Average heat flux in boiler (0-250 inches), 23,000 BTU/hr ft²

In addition to the primary program task cited above the program also includes capsule testing to evaluate advanced tantalum alloys of the ASTAR 811 type (Ta-8W-1Hf-1Re) in both potassium and lithium.

Also included in the program is the fabrication, 5000-hour operation and evaluation of a 2600°F, high flow velocity, pumped lithium loop designed to evaluate the compatibility of the ASTAR 811 type alloys, T-111, T-222, and the tungsten alloy, W-25Re-30Mo.

II. SUMMARY

The fabrication of all loop components with the exception of the valves and nine-stage turbine simulator was completed.

The lithium has been distilled and analyzed.

The post test evaluation of the preliminary capsule tests of weld specimens of ASTAR 811 and ASTAR 811CN alloys was completed. The capsule tests have been modified to include the evaluation of ASTAR 811C alloy.

A failure mode effects analysis report for Corrosion Loop I (T-111) is in preparation.

III. PROGRAM STATUS

A. CORROSION LOOP I (T-111) FABRICATION

The fabrication status of T-111 Corrosion Loop components is as follows:

1. Slack Diaphragm Transducer

Taylor Instrument Company returned one of six T-111 transducers when a leak was detected across the diaphragm. This transducer was cut open and the defective diaphragm was removed. Fluorescent penetrant inspection revealed two radial cracks in the diaphragm near the heat affected zone of the weld. Subsequent metallographic examination revealed an intergranular crack in otherwise normal diaphragm material.

The transducer was repaired by welding a new diaphragm in place, and it was subsequently proof tested by applying ten vacuum evacuation cycles on the NaK side of the diaphragm. Mass spectrometer leak testing indicated no failure, and welding and postweld annealing of the assembly was completed. A final mass spectrometer leak test indicated no diaphragm leakage.

The five acceptable transducers were each pulsated five times between vacuum and 0.5 psig by the Taylor Instrument Company. There was no evidence of leakage after testing.

The six transducers will be filled with NaK by the Taylor Instrument Company by the end of July.

2. Lithium and Potassium EM Pump Ducts

The interference fits between the wrapper and EM pump helix were made successfully for both the lithium and potassium EM pumps. The pumps were then processed through the welding and final outside diameter machining phases of their fabrication. The completed EM pump ducts are shown in Figure 1. X

The pump ducts were postweld annealed at 2400°F for 1 hour in the General Electric-IMCD vacuum furnace. All conditions were in accordance with Specification SPPS 03-0037-00-A except that pressure was 5×10^{-5} torr at the start of each furnace run and decreased to 1.2×10^{-5} torr during the 1 hour

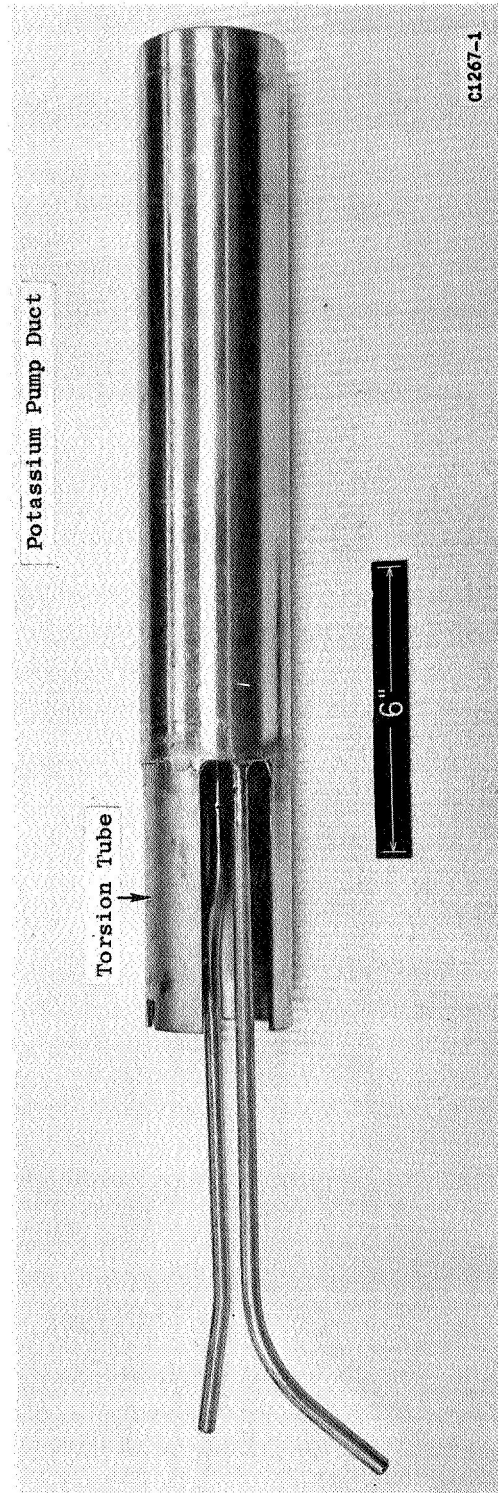
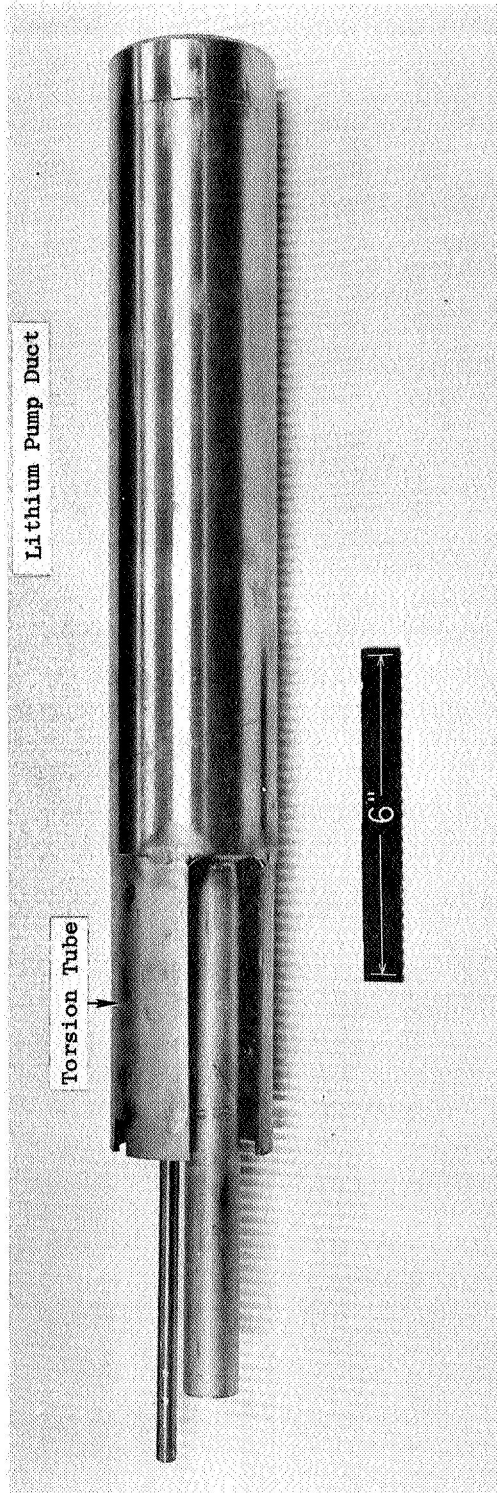


Figure 1. T-111 Alloy EM Pump Ducts for Corrosion Loop I (T-111).
(Orig. C67081421)
(Orig. C67081425)

at temperature. These conditions had been shown to be acceptable by a furnace qualification run performed prior to the postweld anneal of the EM pump duct. Chemical analysis results, shown in Table I, indicate negligible contamination of T-111 alloy sheet specimens placed in the furnace during the qualification run.

3. Throttle and Isolation Valves

The valve body blanks were received from the vendor, Hoke, Inc., Cresskill, New Jersey. The T-111 alloy nipple tubes, 0.375-inch OD x 0.065-inch wall, were then welded to these blanks, and the assemblies were returned to Hoke for final machining. The valve bellows assemblies were also received. Subsequently, the Cb-1Zr bellows were electron beam welded into the T-111 alloy assemblies. Completion of the valves is pending receipt of the final machined valve bodies and valve plugs from Hoke.

4. Turbine Simulator

The ten nozzles were final machined. Results of the inspection of the nozzle diameters are shown in Table II. These dimensions will be used for reference during post test evaluation along with the spare nozzles, part 5 S/N1 (Mo-TZC) and part 6 S/N1 (Cb-132M alloy).

The nozzle assemblies consist of a nozzle, blade, and two pads which support the blade. The single stage turbine simulator nozzle assembly is shown in Figure 2. The nine remaining nozzle assemblies are currently having the blade assemblies fitted into the nozzles. The nozzle, blade, and blade pads of each of the nozzle stages shall be weighed following final cleaning prior to assembly of the turbine simulator.

5. Condenser

The honing of the inside diameter of the two 30-inch long condenser bars was completed. The welding of the condenser was then completed with the joining of the two condenser bars and tantalum fins as shown in Figure 3. This component is now ready for assembly with the nine stage turbine simulator and sub-cooler reservoir to form the condenser major assembly.

TABLE I. RESULTS OF CHEMICAL ANALYSES OF T-111 SHEET
FOR HEAT TREATMENT QUALIFICATION OF GE-LMCD
VACUUM FURNACE

	PRESSURE, TORR		ELEMENT, ppm ^(a)			
	START OF	END OF	O	N	H	C
	RUN	RUN				
Pre-Test Analysis (MCN 02B-010) ^(b)			65	5	1	10
Analysis after one hour at 2460°F in the vacuum furnace	2.8 x 10 ⁻⁵	2.2 x 10 ⁻⁵				
Wrapped Specimen ^(c)			63,61	7,7	1,1	3,4
Unwrapped Specimen			68,85	8,8	2,2	7,9

(a) Analytical Procedures: O, N, and H; Vacuum Fusion
C; Combustion Conductometric

(b) 0.040-inch thick sheet

(c) One specimen wrapped with one overlapped layer of 0.002-inch thick
Cb-1Zr foil

TABLE II. RESULTS OF INSPECTION OF NOZZLE HOLE DIAMETERS^(a)

PART NO.	NOZZLE DIAMETERS PER DRAWING INCHES		ACTUAL DIAMETERS ^(b) INCHES	
			0°	90°
1	.0885	.0895	.089200	.089150
2	.0875	.0885	.088075	.087775
3	.0965	.0975	.096380	.096380
4	.1065	.1075	.108350	.108200
5 S/N1	.1175	.1185	.116940	.116475
5 S/N2	.1175	.1185	.118050	.118070
6 S/N1	.1295	.1305	.129975	.129925
6 S/N2	.1295	.1305	.129225	.129225
7	.1445	.1455	.145725	.145750
8	.1595	.1605	.159850	.159825
9	.1775	.1785	.178325	.178525
10	.1985	.1995	.198625	.198660

(a) Measurements made by the Automation and Measurement Division of Bendix, Dayton, Ohio, by means of an electronic comparator using a frictionless electronic transducer compared to master gauge blocks.

(b) The accuracy of the measurement(s) is ± 0.0001

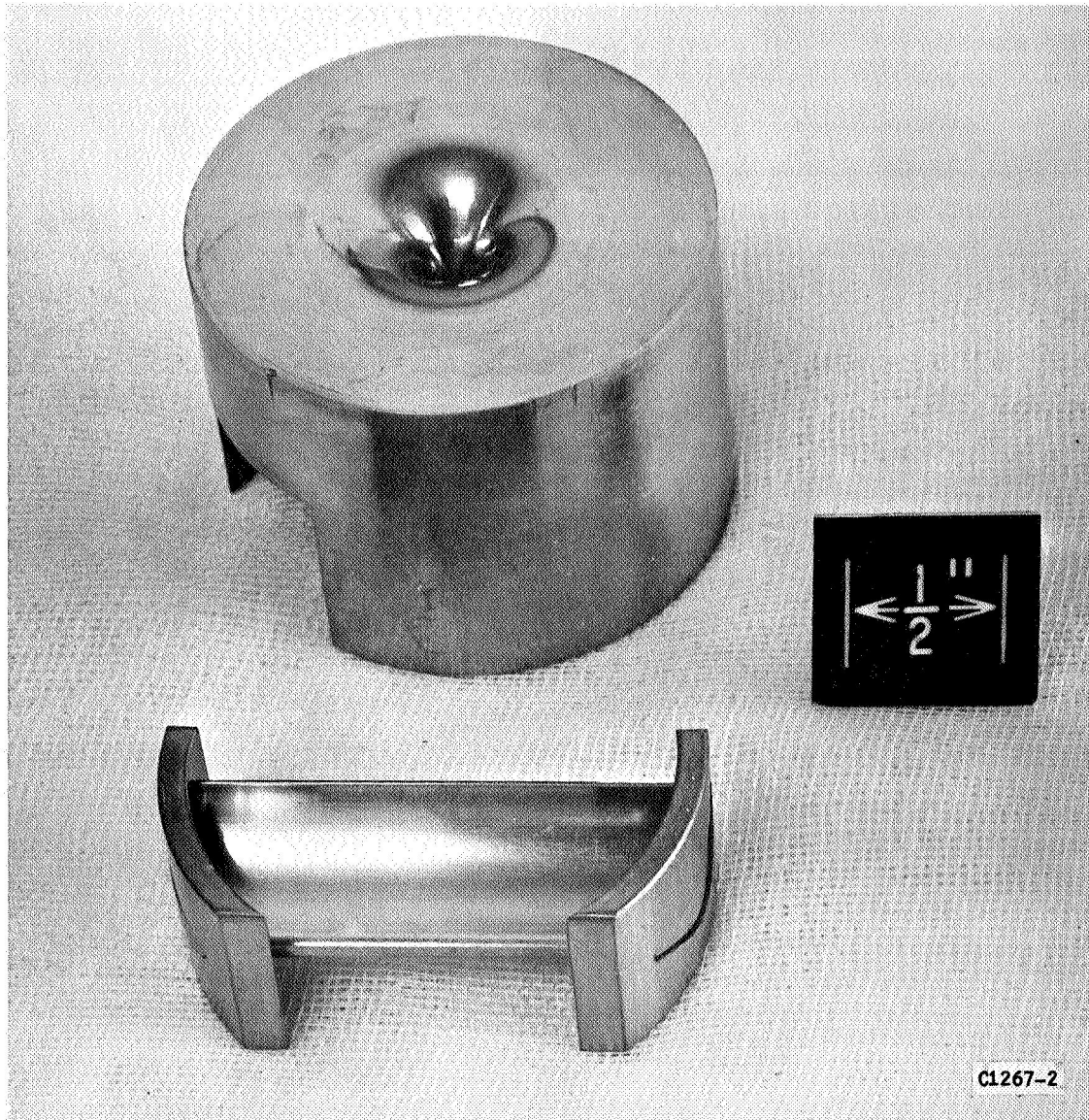


Figure 2. First Stage Nozzle and Blade Assembly (Mo-TZC) for the Turbine Simulator of Corrosion Loop I (T-111). (Orig. C67072546)

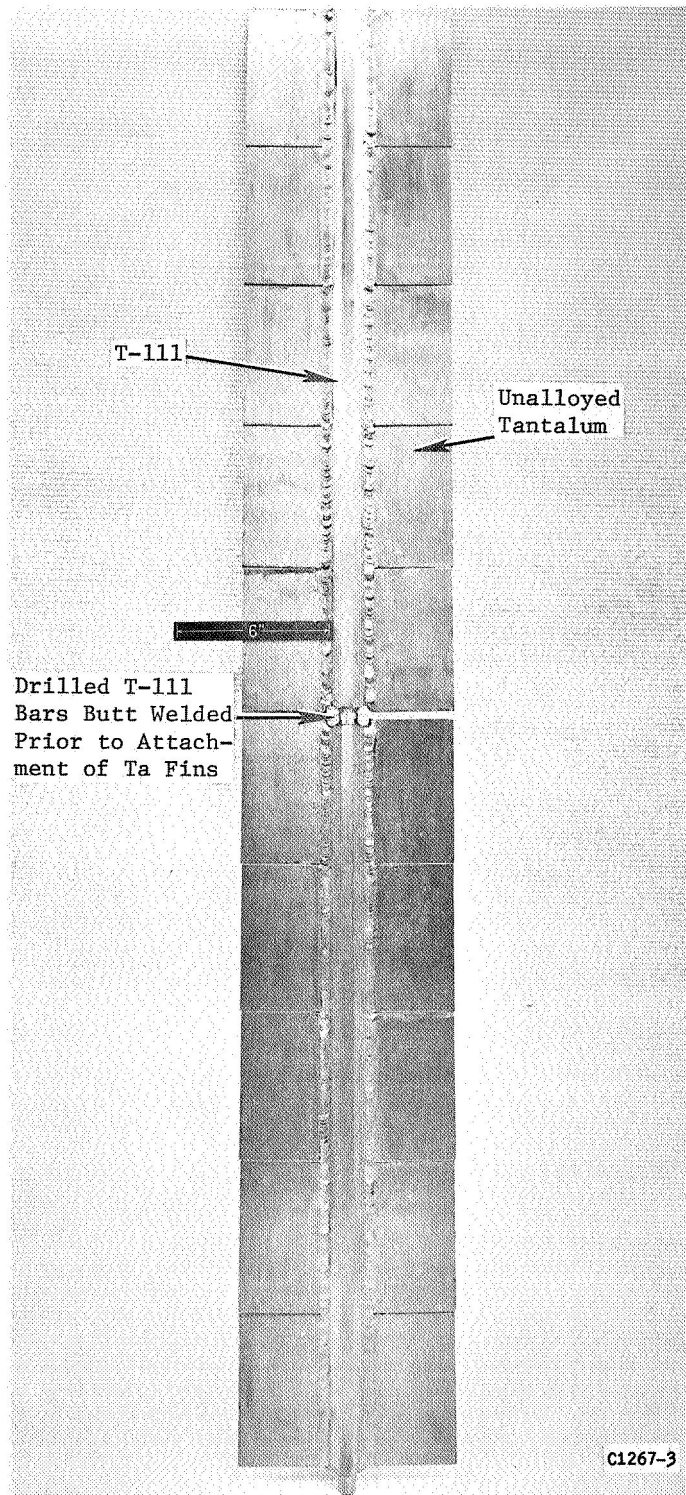


Figure 3. Corrosion Loop I (T-111) Condenser Prior to Coating of Tantalum Fins With Iron Titanate. (Orig. C67081423)

6. Boiler

The tube-in-tube boiler was formed successfully. The sugar which was used to pack the internal tube and annulus during forming was removed by mechanical vibration. When no additional sugar could be removed, a deionized water flush was initiated. The water was heated to 150°F and was allowed to flush through the boiler for 15 hours. An additional cold water flush for 30 minutes was used for the final rinse. At this time water samples were equilibrated for two hours in the boiler and tested for sugar using the Molisch⁽¹⁾ test. Duplicate test determinations indicated less than 50 ppm sugar, which is the limit of detection of the method used.

The final welding of the boiler inlet and outlet connectors was then completed. The assembled boiler and helical plug insert are shown in Figure 4. This component will be incorporated into the boiler assembly, which includes the potassium preheater and single stage turbine simulator, during the next report period.

7. Potassium Preheater and Lithium Heater

The potassium preheater and lithium heater were completed during the last quarter. The lithium heater assembly, Figure 5, will be postweld annealed along with the boiler and condenser assemblies. The potassium preheater, which is shown in Figure 6, will be welded to the boiler during the next report period.

B. ALKALI METAL PURIFICATION

Fifteen pounds of lithium (in three 5-pound lots) were distilled at an average temperature of 1235°F, at the rate of 115 gms/hr. The first two 5-pound batches of distilled lithium were returned to the hot trap to dilute

(1) Cheronis, N. D., "Techniques of Organic Chemistry", Interscience Publishers, Inc., New York, New York, 1954, p 466-467.

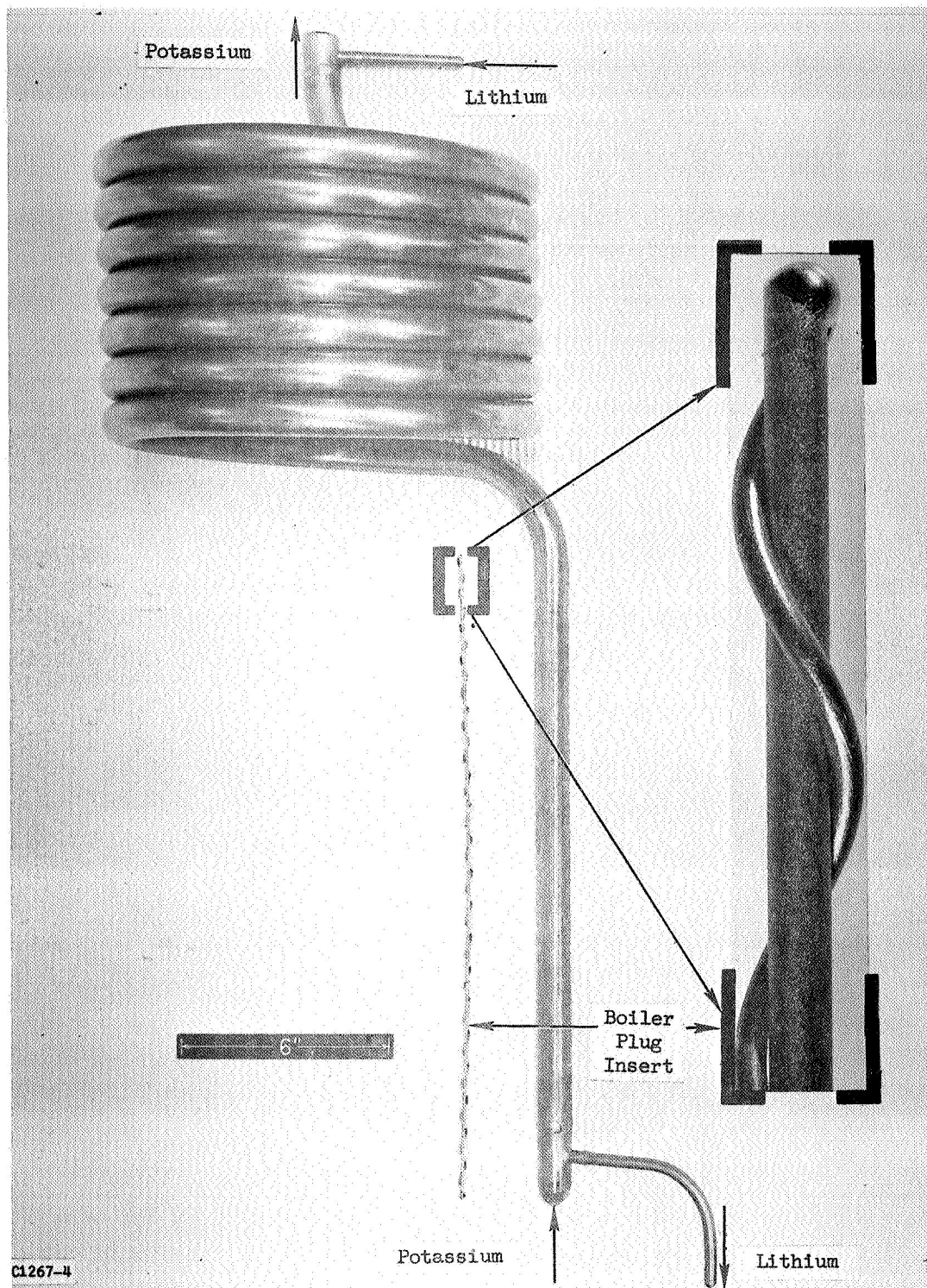


Figure 4. Tube-in-Tube Boiler of Corrosion Loop I (T-111) Prior to Insertion of the Boiler Plug. (Orig. C67071832) (Orig. C67072436)

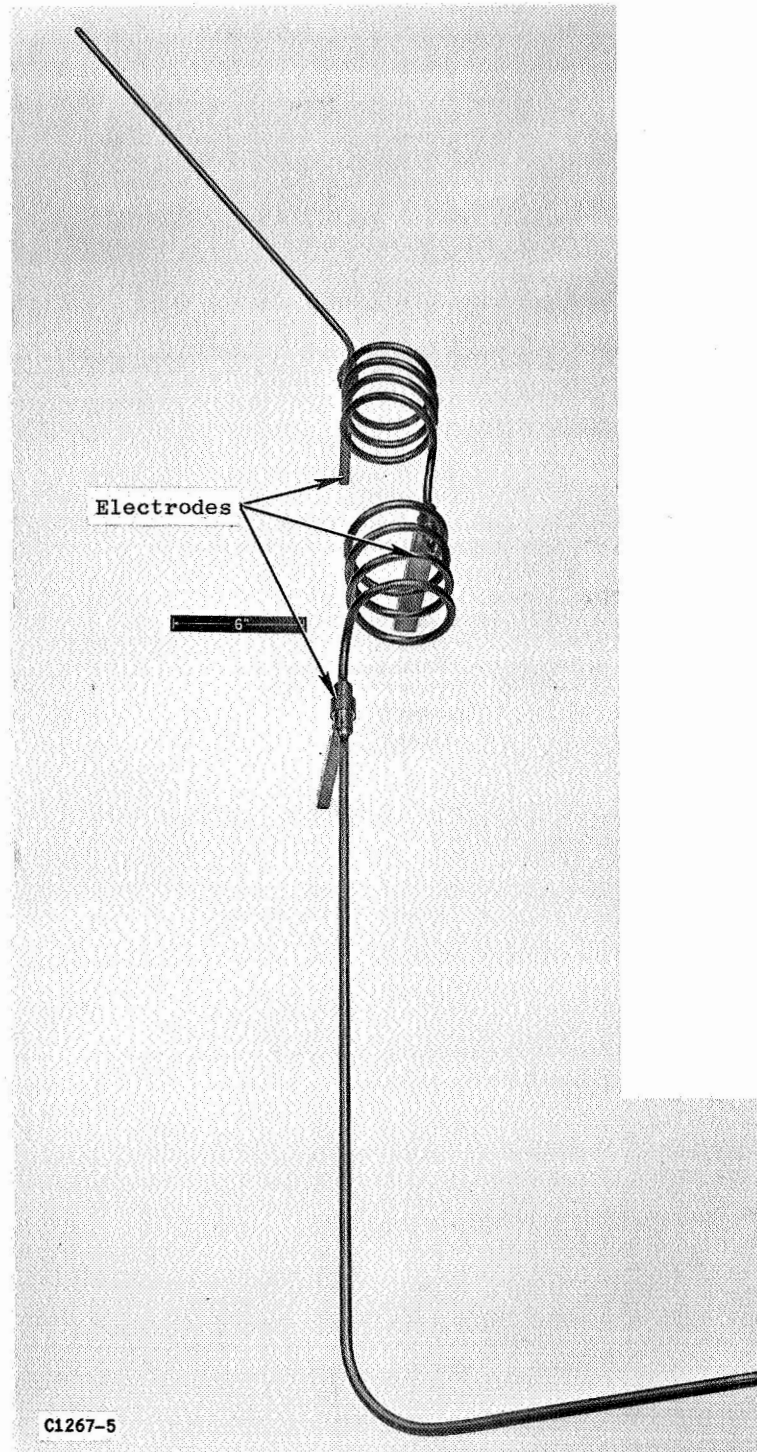


Figure 5. Lithium Heater Assembly of Corrosion Loop I (T-111).
(Orig. C67071830)

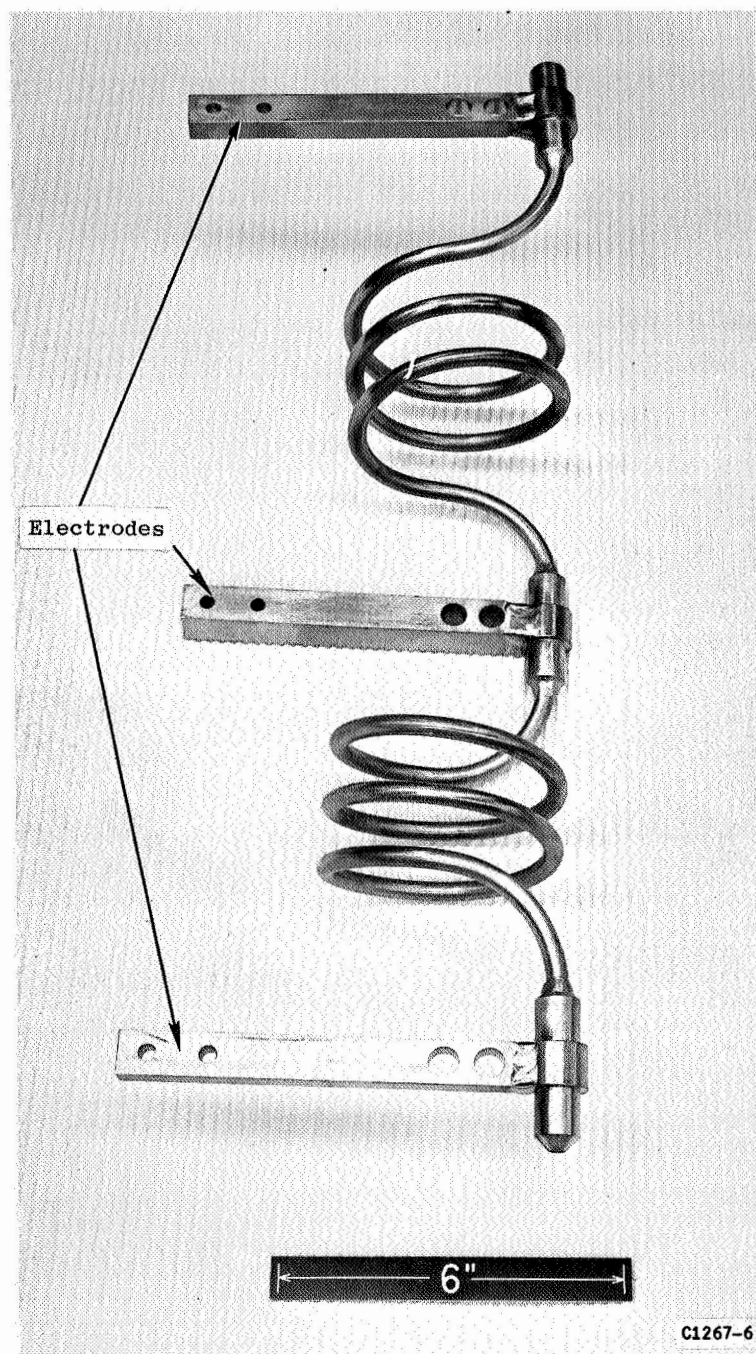


Figure 6. Potassium Preheater of Corrosion Loop I (T-111).
(Orig. C67052559)

the lithium which was inadvertently added to the still receiver during the initial fill of the still pot⁽²⁾. Two samples of distilled lithium have been obtained from the still and analyzed for oxygen, nitrogen, carbon, and metallic impurities. The analytical history of this material is presented in Tables III and IV.

The I-tubes used to determine the liquid level in the still pot functioned very well during the distillation as did the entire distillation system. The Cb-1Zr, below the bimetallic joints, at the bottom of the condenser was controlled in the temperature range of 390°F to 450°F with an air flow rate of about 200 SCFH through the condenser cooling coils.

The data in Table III shows that the carbon and calcium concentration in the lithium were reduced by filtering, and that nitrogen, carbon, and calcium concentrations were reduced by hot trapping and filtering. Neither filtering nor hot trapping had any significant effect on the concentration of oxygen. Distillation reduced the concentrations of metallic impurities but had no significant effect on nitrogen and carbon concentrations.

Oxygen analyses were performed by General Atomic, San Diego, California using fast neutron activation techniques. It was anticipated that the oxygen level would be reduced by distillation. The lithium may have been contaminated during sampling or analysis. This postulate will be checked by analyzing another sample. If similar oxygen concentration are obtained, an additional quantity of lithium will be distilled and analyzed.

C. ADVANCED TANTALUM ALLOY CAPSULE TESTS

The results of the preliminary capsule tests indicated welded ASTAR 811 and ASTAR 811CN alloys containing oxygen in concentrations in excess of 400 ppm lacked sufficient compatibility with potassium to be considered for

(2) Advanced Refractory Alloy Corrosion Loop Program, Quarterly Progress Report Number 8 for Period Ending April 15, 1967, NASA Contract NAS 3-6474, to be published.

TABLE III. ANALYSIS OF LITHIUM FOR OXYGEN, NITROGEN, AND CARBON

<u>SAMPLE DESIGNATION</u>	<u>CHEMICAL ANALYSIS - ppm IN LITHIUM</u>			
	<u>O^(a)</u>	<u>N^(b)</u>	<u>C^(c)</u>	<u>Ca^(d)</u>
<u>As-Received</u>				
Sample Number 292	130 ± 25	801	134	133
	130 ± 25	834	158	
		871		
<u>After Filtering at 400°F through a 5-micron Filter</u>				
Sample Number 293	150 ± 28	755	97	53
	160 ± 28	778	101	
		841		
<u>After Hot Trapping at 1500°F for 126 Hours</u>				
Sample Number 309	106 ± 16	11		5
	123 ± 13	10		
		5		
Sample Number 627		12	38	
		14	48	
<u>Distilled</u>				
Sample Number 1031	245 ± 36	19	33	
		18	42	<5
Sample Number 1064	138 ± 41	4	32	
		22	56	
			59	

- (a) Fast neutron activation
 (b) Micro Kjeldahl
 (c) Combustion conductometric
 (d) Spectrographic

TABLE IV. ANALYSIS OF LITHIUM FOR METALLIC IMPURITIES

ELEMENT	Metallic Impurities - ppm in Lithium ^(a)			
	Sample Designation			
	AS RECEIVED (SAMPLE NO. 292)	FILTERED (SAMPLE NO. 293)	HOT TRAPPED (SAMPLE NO. 309)	DISTILLED (SAMPLE NO. 1031)
Ag	< 5	< 5	< 5	< 5
Al	< 5	< 5	5	< 5
B	<50	<50	<50	<50
Ba	75	50	<50	<50
Be	< 5	< 5	< 5	< 5
Ca	133	53	5	< 5
Cb	<25	<25	<25	<25
Co	< 5	< 5	< 5	< 5
Cr	< 5	< 5	< 5	< 5
Cu	< 5	< 5	28	< 5
Fe	< 5	< 5	5	< 5
Mg	5	5	< 5	< 5
Mn	< 5	< 5	< 5	< 5
Mo	< 5	< 5	< 5	< 5
Na	53	133	80	<25
Ni	< 5	< 5	28	< 5
Pb	<25	<25	<25	<25
Si	5	5	28	5
Sn	<25	<25	<25	<25
Sr	5	5	< 5	< 5
Ti	< 5	< 5	< 5	<25
V	<25	<25	< 5	<25
Zr	< 5	< 5	< 5	<25

(a) Spectrographic analysis

capsule construction. The capsule tests have been modified to include the testing of ASTAR 811C (Ta-8W-1Hf-1Re-0.012C) alloy in place of the ASTAR 811 alloy. Two potassium reflux capsules will be constructed from ASTAR 811C alloy sheet and will contain two contaminated (300 ppm oxygen) bend specimens.

One specimen will be in the as-welded condition whereas the other specimen will be welded and postweld annealed. The ASTAR 811C alloy's resistance to corrosion by potassium can, therefore, be evaluated in both the as-received condition (represented by the capsule) and oxygen contaminated condition (represented by the specimens).

A similar plan will be employed to evaluate the lithium corrosion resistance of the ASTAR 811C alloy. Two thermal convection capsules will be constructed of ASTAR 811C alloy sheet. Bend specimens of contaminated ASTAR 811C in the aforesaid conditions will be suspended in the lithium by means of hooks on the T-111 alloy downcomer tube. The design of the ASTAR 811CN mass transfer capsule and the test plans of all the capsules have not changed⁽³⁾.

D. QUALITY ASSURANCE

A failure mode and effects analysis report for Corrosion Loop I (T-111) is in preparation.

(3) Advanced Refractory Alloy Corrosion Loop Program, Quarterly Progress Report Number 6 for Period Ending October 15, 1966, NASA Contract NAS 3-6474, NASA-CR-72177, p. 14-20.

IV. FUTURE PLANS

- A. Fabrication of the Corrosion Loop I (T-111) sub-assemblies will be completed, and the sub-assemblies will be post-weld annealed at 2400°F.
- B. The distilled lithium will be resampled and analyzed.
- C. Specimens of oxygen contaminated ASTAR 811C alloy will be prepared and capsule fabrication will be initiated.
- D. The failure mode and effects analysis report for Corrosion Loop I (T-111) will be issued.

PUBLISHED REPORTS

<u>Quarterly Progress</u>	<u>For Quarter Ending</u>
Report No. 1 (NASA-CR-54477)	July 15, 1965
Report No. 2 (NASA-CR-54845)	October 15, 1965
Report No. 3 (NASA-CR-54911)	January 15, 1966
Report No. 4 (NASA-CR-72029)	April 15, 1966
Report No. 5 (NASA-CR-72057)	July 15, 1966
Report No. 6 (NASA-CR-72177)	October 15, 1966
Report No. 7 (NASA-CR-72230)	January 15, 1967
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