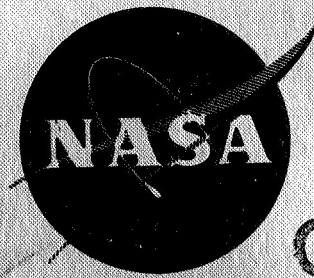


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ADVANCED REFRACTORY ALLOY CORROSION LOOP PROGRAM

QUARTERLY PROGRESS REPORT NO. 13

For Quarter Ending July 15, 1968

prepared by
R. W. Harrison

prepared for
NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

NASA Lewis Research Center
Contract NAS 3-6474
Robert L. Davies, Project Manager
Materials Section

NUCLEAR SYSTEMS PROGRAMS
SPACE SYSTEMS
GENERAL  ELECTRIC
CINCINNATI, OHIO 45215

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MISSILE AND SPACE DIVISION
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prepared for
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Period April 16 to July 15, 1968
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FOREWORD

The work described herein is sponsored by the National Aeronautics and Space Administration under Contract NAS 3-6474. R. L. Davies of NASA - Lewis Research Center is the NASA Technical Manager.

The program is being administered for the General Electric Company by E. E. Hoffman, and R. W. Harrison is acting as the Program Manager. J. Holowach, the Project Engineer, is responsible for the loop design, facilities procurement, and test operations. 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.

ADVANCED REFRACTORY ALLOY CORROSION LOOP PROGRAM

I. INTRODUCTION

This report covers the period from April 15, 1968 to July 15, 1968. 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 Test Loop; a two-phase, forced convection, potassium corrosion test loop which has been tested under Contract NAS 3-2547.⁽¹⁾ 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-1Re-1Hf) in both potassium and lithium.

⁽¹⁾ Hoffman, E. E. and Holowach, J., Cb-1Zr Rankine System Corrosion Test Loop, Potassium Corrosion Test Loop Development Topical Report No. 67, R66SD3016, General Electric Company, Cincinnati, Ohio, May 1, 1968.

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 at conditions simulating an out-of-pile thermionic reactor system.

II. SUMMARY

The alkali metals were removed from the loop by draining and subsequent vacuum distillation.

Leak checking confirmed the leak to be in the boiler coils and the boiler was removed from the loop.

The boiler was cleaned of residual alkali metal by reaction with liquid ammonia. Subsequent helium mass spectrometer leak checking pinpointed the leak in a butt weld in the 0.375-inch boiler tube.

Examination at 30X magnification indicated the leak to be a minute intergranular crack in the weld nugget.

Analysis of distilled lithium has indicated high concentrations of metallics not found in previous samples of lithium.

III. PROGRAM STATUS

A. T-111 RANKINE SYSTEM CORROSION TEST LOOP

It had been previously established that a leak existed between the primary and secondary circuits of the loop within the boiler.⁽²⁾ Radio-graphs of the welds in the boiler did not indicate the source of the leak. Consequently, it was decided to drain both circuits, to distill the residual metal out of these circuits and attempt to locate the leak by other means.

1. Draining the Alkali Metals from the Loop

The loop was prepared for draining of the lithium circuit and distillation of the residual alkali metal using procedures established previously with the Cb-1Zr Rankine System Corrosion Test Loop.⁽¹⁾ Six quartz infrared lamps^(a) with tungsten filaments were installed stainless steel brackets welded to the loop support structure. The lamps were connected in parallel to the main heater power supply used in the loop operation. A stainless steel thermal shield was wrapped around the loop support structure to thermally insulate the loop and quartz heaters from the test chamber bell jar. Special precautions were taken in positioning the quartz lamps so that the ends would protrude through the thermal insulation shield out of the heater zone to reduce

(1) Hoffman, E. E. and Holowach, J., Cb-1Zr Rankine System Corrosion Test Loop, Potassium Corrosion Test Loop Development Topical Report No. 67, R66SD3016, General Electric Company, Cincinnati, Ohio, May 1, 1968.

(2) Advanced Refractory Alloy Corrosion Loop Program, Quarterly Progress Report No. 12 for Period Ending April 15, 1968, NASA Contract NAS 3-6474.

(a) General Electric Type 3800 Te/VB, 3800 watts, 570 volts, 1/2-inch OD x 43 1/2-inch long.

the possibility of failure of the lamp ends from overheating. The lithium and potassium surge tanks were partially shielded from the quartz heaters to maintain a lower temperature than the loop so that the alkali metal distilled from the loop would condense in the cooler surge tank located in the lower portion of the vacuum chamber.

The potassium still used in the initial drain of the secondary circuit, was reassembled and reattached to valve KK of the potassium circuit, omitting the valve between valve KK and the still pot.⁽³⁾

The lithium drain container, shown in Figure 1, was fabricated and attached to valve KK of the primary circuit. This container was fabricated from a 24-inch section of 4-inch, schedule 10, Type 304 stainless steel pipe and 4-inch, Type 304 stainless steel pipe caps, and had a volume of 6600 cc. It was weighed prior to attachment to the drain line and the vacuum-argon line.

Both the lithium and the potassium drain systems were baked out and helium leak checked per SPPS Specification 03-0013-00-B and no leaks were found in excess of 5×10^{-10} std cc of air per second at temperatures from 400° to 700°F. The potassium circuit had previously been drained of liquid metal.⁽²⁾

After a low power electrical checkout of the heating lamps, the bell jar was lowered to the spool section and the vacuum chamber was sealed. The turbomolecular pump was used to rough pump the vacuum chamber for 24

⁽³⁾op. cit., Quarterly Progress Report No. 12, Figure 19.

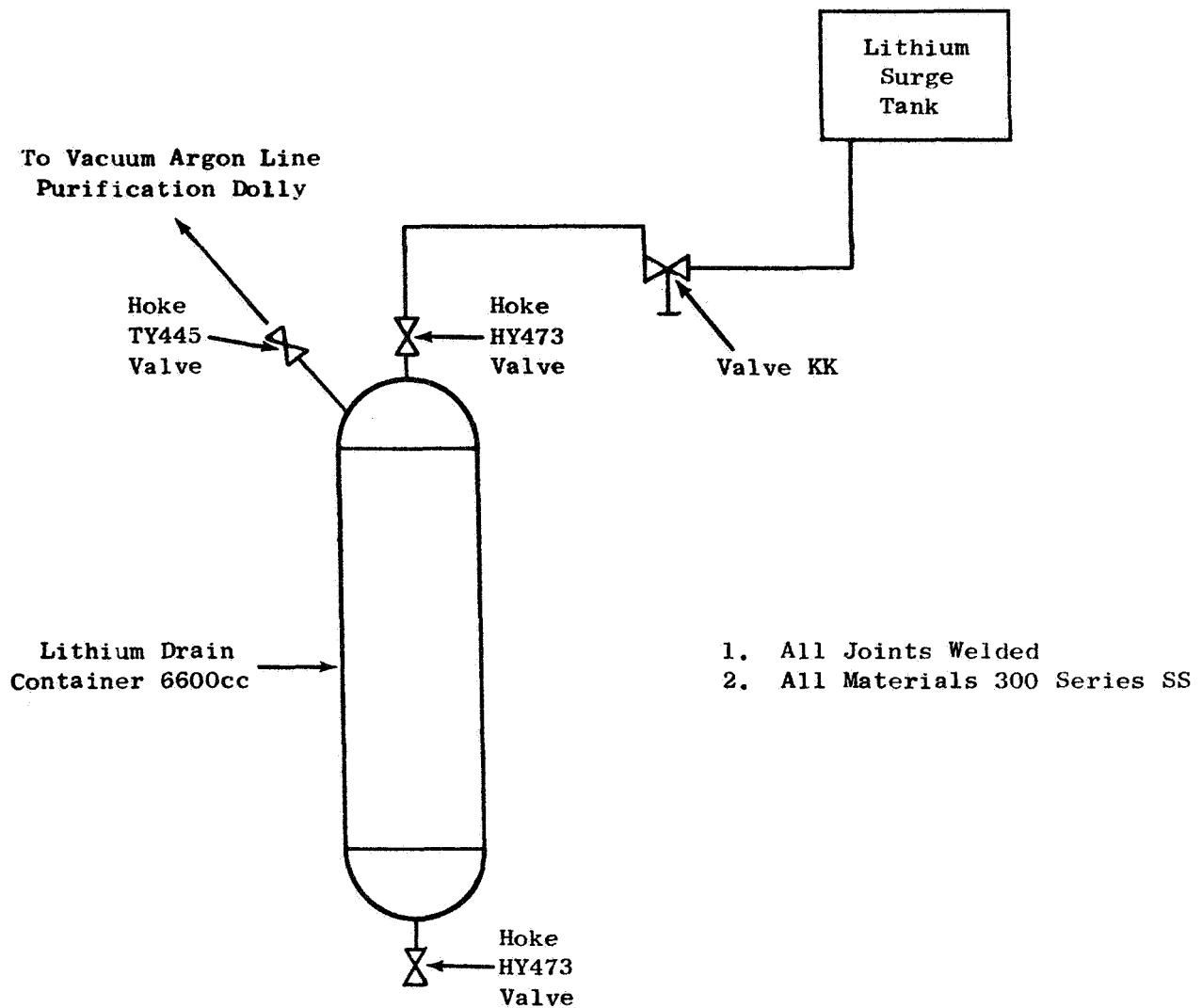


Figure 1. Schematic of System Used for Draining Lithium from the Primary Circuit of T-111 Rankine System Corrosion Test Loop.

hours before the ion pump was turned on. The chamber bakeout was turned on when the chamber pressure was in the 10^{-7} torr range. The system was outgassed for approximately 100 hours with only the vacuum system bakeout on. The loop temperature during this period was approximately 500°F. The EM pump power was turned on at approximately 10% of the rated power to heat-up the pump duct.

The lithium EM pump power was increased to 40% of rated power, and the lithium loop temperature increased to 1000°F. After circulating lithium for 4 hours; the lithium circuit was evacuated and the lithium drained from the loop. After draining the lithium circuit, the EM pump power was reduced to 10% of the rated power to maintain the pump duct temperature at 1000°F.

Approximately 3600 cc of metal was removed from this circuit as indicated by level probe measurements of the liquid level in the container. The level probe also indicated an additional interface about 1-1/2 inch above the bottom of the container which corresponded to about 200 cc of potassium. After cooling the container to room temperature, a radiograph showed a void separating the two layers and confirmed the level probe indication.

After the initial draining operation, both circuits were evacuated with the vacuum system on the potassium purification dolly. The quartz lamp power was then slowly increased in small increments, to hold the vacuum chamber pressure in the 10^{-7} torr range. The potassium EM pump power was increased and the power to the potassium preheater was turned on.

Typical operating conditions during the distillation period from 4-25-68 to 5-15-68, are as follows:

Chamber Pressure	3×10^{-8} torr
Average Lithium Circuit Temperature	750°F
Average Potassium Circuit Temperature	750°F
Boiler Temperature	850°F
Potassium Preheater Temperature	1300°F
Lithium Surge Tank	540°F
Potassium Surge Tank	580°F
EM Pump Duct Temperature (Li and K)	1200°F

During the distillation period, the vacuum chamber bakeout heaters on the spool section and the sump were not turned on so that the lower portion of the loop (surge tanks) would be at a lower temperature to condense the alkali metal, distilled from the loop, in the surge tanks. Typical vacuum chamber wall temperatures during the distillation were as follows:

Bell Jar	550°F	(Power On)
Spool	280°F	(Power Off)
Sump	185°F	(Power Off)

Periodically the surge tank drain valves were opened to gravity drain the distillate from the surge tank. The surge tank would then be pressurized to drive any remaining alkali metal into the drain tank.

A material balance on each metal was obtained with the following results:

Lithium:

Placed in loop	3750 cc
Removed during sampling	<u>350 cc</u>
Balance in loop and surge tank	3400 cc

According to both level probe and radiograph measurements, 3330 cc of lithium were removed by draining and distillation. The container was subsequently reweighed and the weight of the combined lithium and the potassium in the container was found to be 1725 grams. Taking the densities for lithium and potassium as 0.499 g/cc and 0.770 g/cc respectively at 600°F, the container temperature, the respective weights were calculated to be 1660 grams for 3330 cc of lithium and 154 grams for 200 cc of potassium; or a total of 1814 grams which is in reasonable agreement with the weight actually found.

Potassium:

Placed in loop	2350 cc
Removed during sampling	<u>350 cc</u>
Balance in loop and surge tank	2000 cc

A total of 1150 cc of potassium were drained from the loop including the 950 cc removed during the initial draining operation and the 200 cc in the lithium drain container. Approximately 850 cc of potassium have not been accounted for; and where it is has not been determined.

It will be noted that only 2350 cc of potassium was placed in the loop. Actually, the charge pot volume is 3750 cc, the same as for the lithium, and according to the technique used to transfer the potassium this quantity should have been transferred to the surge tank.⁽¹⁾ However, subsequent to the initial draining operation, it has been determined that about 1400 cc of potassium remains in the charge pot. At present this is not understood, although it is conceivable that, if the valve at the bottom of the charge pot did not seat properly when closed, potassium from the surge tank could have been returned to the charge pot during sampling operations after flushing the loop.

During this period, the metering and isolation valves were opened and closed to dislodge any particulate matter which could block the draining of the loop. The valve operation became more difficult, requiring higher torque to open and close the valve, after each operation. The valves finally seized after less than 25 cycles and could not be operated using the maximum torque (5 ft-lbs) of the rotary vacuum feedthrough. Both valves seized in the open position which allowed the distillation to be completed without repairing the valves.

As reported earlier,⁽¹⁾ the metering valve had seized during the test start up. The tungsten carbide sleeve bearing on the pinion gear shaft had been replaced with an unlubricated commercially available 52100 steel ball bearing when the vacuum chamber had been opened to install the tungsten quartz lamps for distillation of the residual alkali metal. The isolation valve, which had only limited service in

the loop operation, had the original tungsten carbide sleeve bearing. Although both valve bearings had seized, the ball bearing had operated for a far greater number of cycles than the sleeve bearing. An examination of the ball bearing showed that the failure was due to the deterioration of the low carbon steel ball separator which was completely deformed and partially wedged between the balls and the race. A ball bearing replacement with high temperature material used for the inner and outer race and tungsten carbide balls without a ball separator is planned for future operation.

2. Preliminary Leak Checking of the Boiler Before Cutting

The methods to be employed in repairing the boiler, if possible, are contingent upon the location of the leak between the potassium and lithium circuits. There are four welds in the potassium circuit of the boiler, as shown in Figure 2, which could be the source of the leak. If the leak is found to be either at the top or bottom weld areas of the boiler, appropriate repair plans can be devised. If the leak is within the boiler coils, repair may not be possible and a new boiler may be required.

An attempt was made to locate the leak in the potassium boiler by comparing the pressure rise rate at room temperature and during heating of localized areas of the boiler. The pressure rise in the potassium loop as measured with a thermocouple gauge is shown in Figure 3 for the boiler at room temperature and as the boiler inlet, outlet, and the center coils were successively heated to approximately 400°F. During

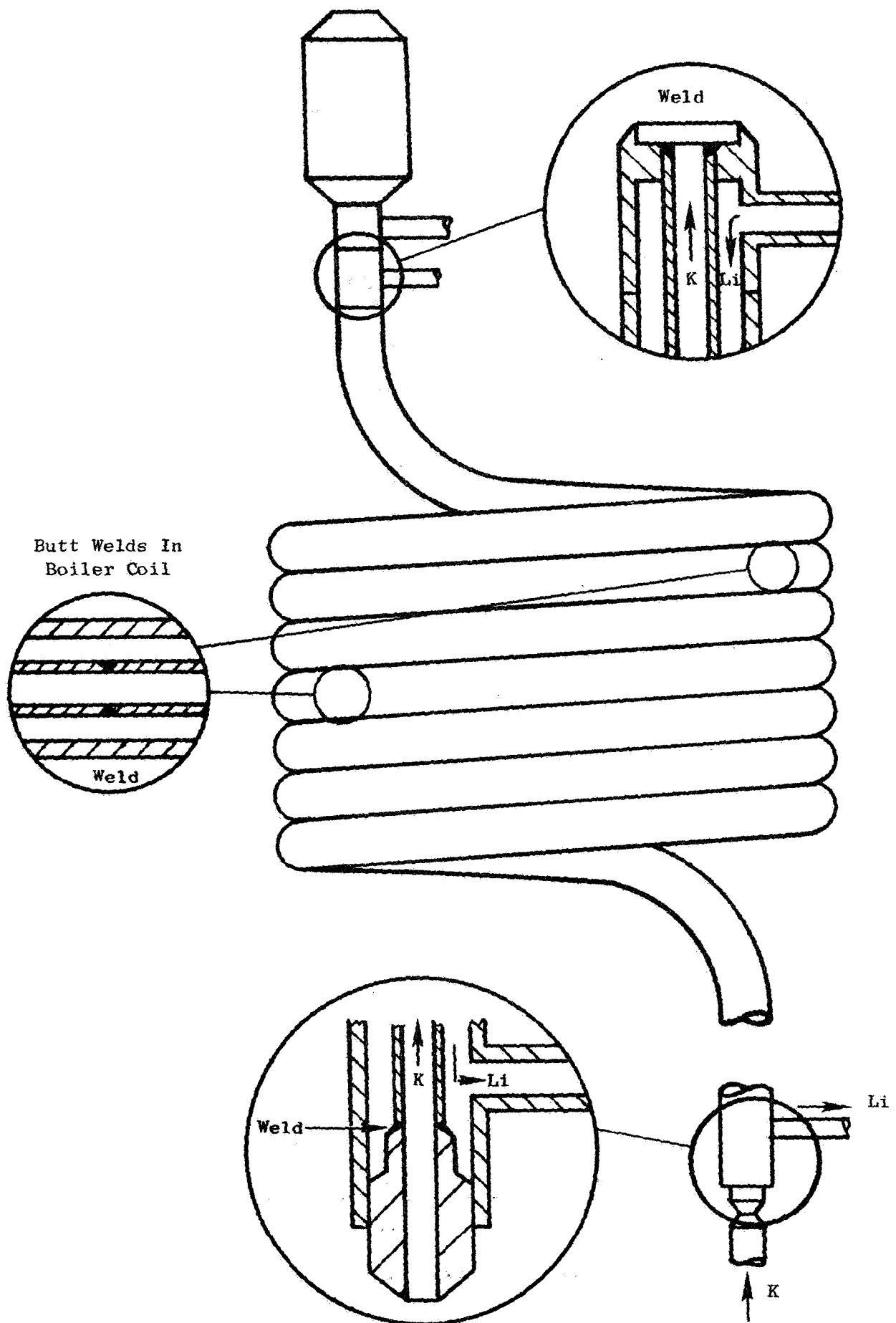


Figure 2. Welds in 0.375-Inch OD T-111 Alloy Tubing in T-111 Rankine System Corrosion Test Loop Which Separate the Potassium and Lithium Circuits.

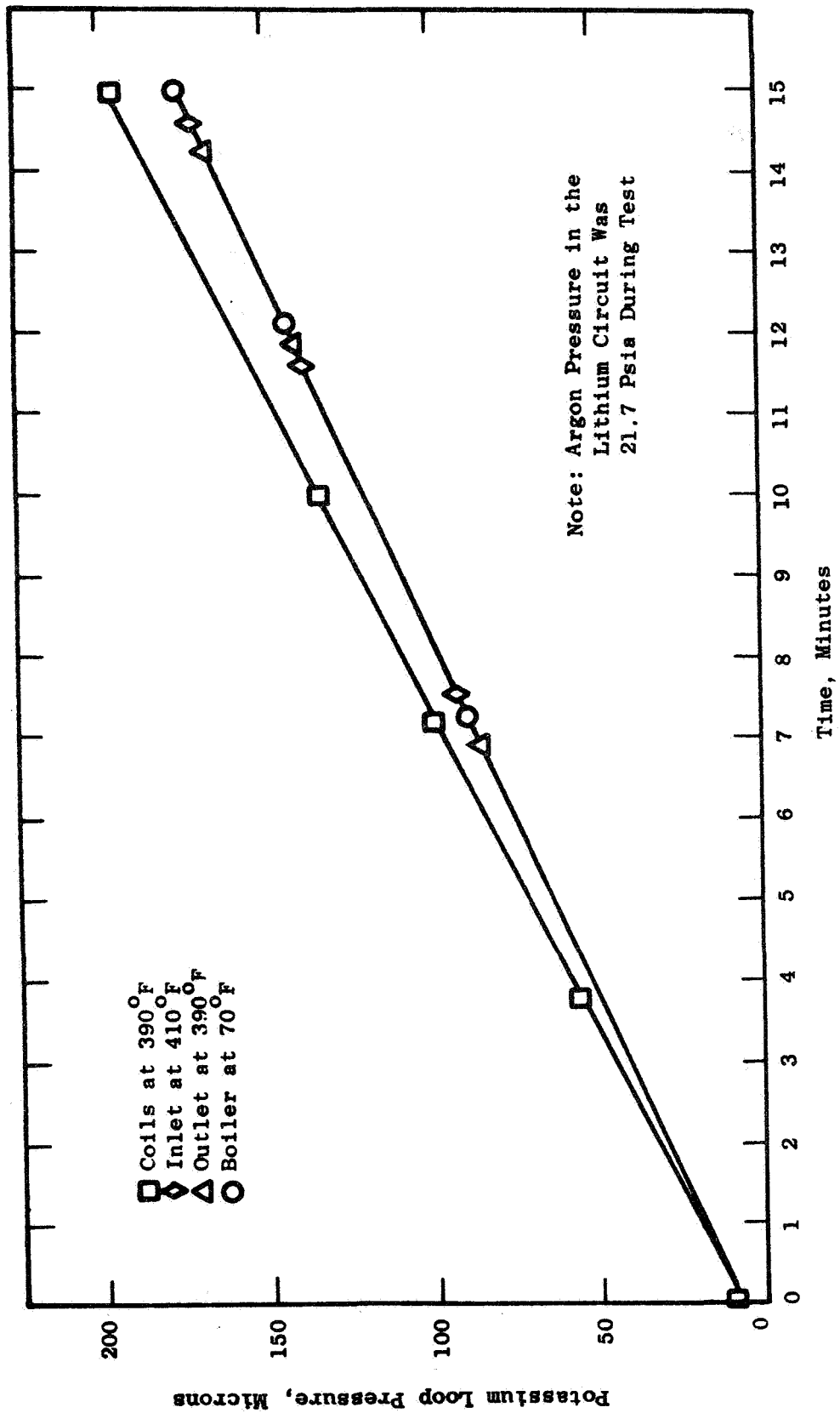


Figure 3. Pressure Rise in Potassium Circuit of T-111 Corrosion Loop
as a Function of Boiler Temperature.

these tests the argon pressure in the lithium loop was 21.7 psia. No significant change in the pressure rise rate was observed when the boiler inlet and outlet were heated; however, a significant change in the pressure rise rate was observed when the center section of the boiler was heated to 400°F.

Following these measurements, the entire boiler was heated, and the pressure rise rate was measured and compared to the room temperature pressure rise rate as shown in Figure 4. Again, the hot boiler had a significantly higher pressure rise rate than the room temperature measurement. A pressure rise rate was also measured with the lithium loop pressure at 0.2 psia and the entire boiler at 400°F. The slight increase in the pressure after 15 minutes of testing indicated that the change in the pressure rise rate was primarily due to the leak and not to outgassing.

A second method of locating the leak was made by measuring the steady state leak rate as measured by a helium mass spectrometer leak detector. The procedure was similar to that described above for the pressure rise rate measurements. The lithium loop was pressurized with helium to 0.4 psia. The potassium circuit was evacuated to the 10^{-4} torr range with the helium leak detector. At these conditions a steady state reading on the helium level meter of the leak detector could be stabilized at 7.5×10^{-8} std cc/sec of helium. Any change in the helium leak rate would be indicated by a corresponding change on the helium level meter assuming the pumping speed of the leak detector remained constant. The

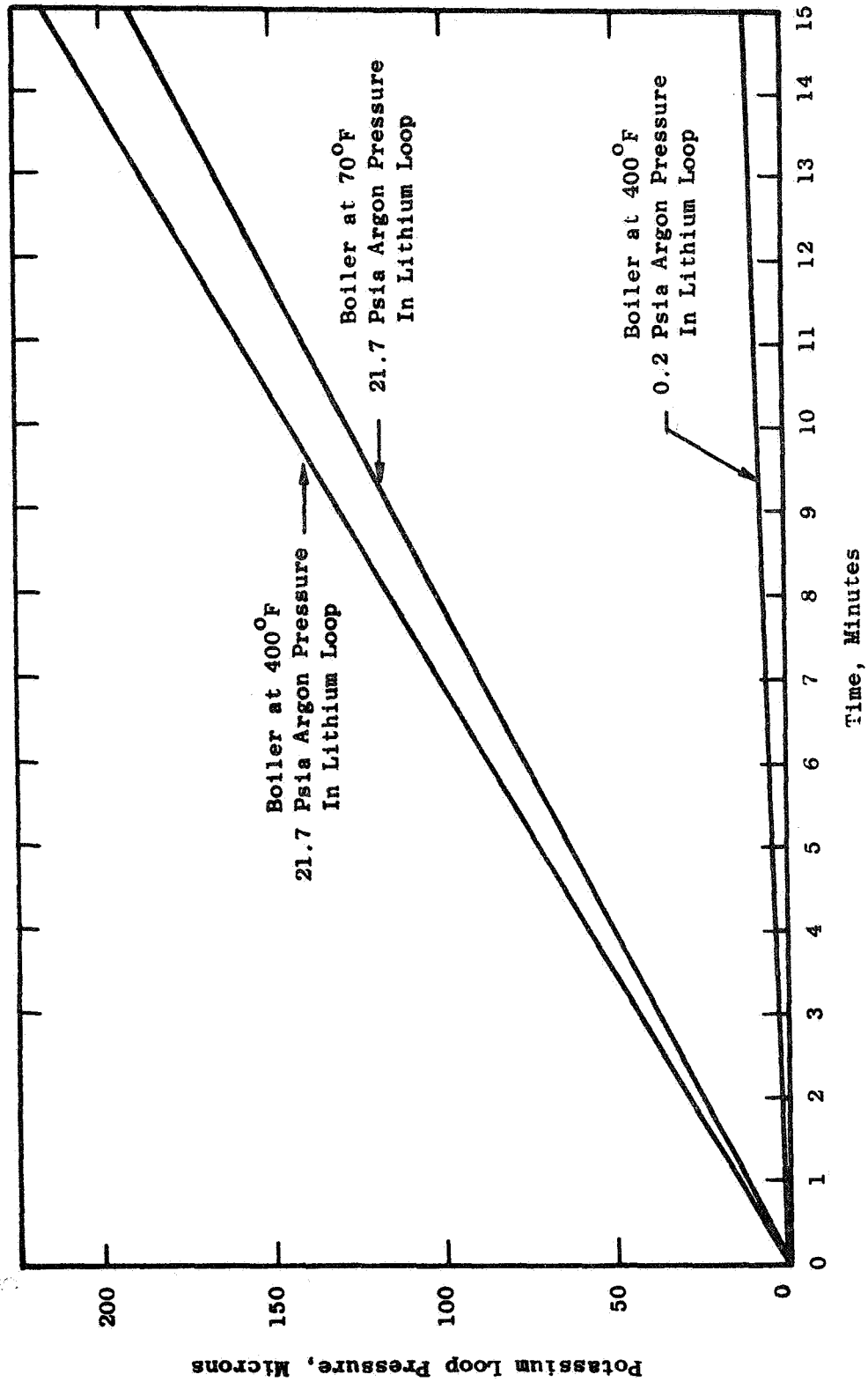


Figure 4. Pressure Rise in Potassium Circuit of T-111 Corrosion Loop as a Function of Boiler Temperature and Lithium Circuit Helium Pressure.

inlet to the boiler was then heated to 380°F for approximately 20 minutes with a slight decrease in the helium level as shown in Figure 5. The heater power to the boiler inlet was turned off, and the boiler outlet was heated to 400°F. Again, the helium level decreased at the same rate as observed when the inlet was heated. When the center section of the boiler which consists of seven 10-inch diameter coils was heated, the helium level increased indicating a higher leak rate. The leak rate continued to increase even after the power was turned off. Subsequently, with the boiler at room temperature, the lithium circuit was pressurized with argon to 35 psig while the potassium circuit was evacuated. The boiler remained at these conditions for three days without indicating a leak as evidenced by the lack of a drop in pressure in the lithium circuit.

The results of these tests indicated that the leak might be in the center section of the boiler, and that the leak was only apparent when the boiler was heated above the melting point of lithium (367°F).

3. Boiler Cutting Procedures

The procedures employed in cutting the boiler were such to minimize the possibility of contaminating the loop by either air reacting with the residual alkali metal in the loop once a cut was made or chips from the cutting operation falling into the loop. To prevent contamination of the alkali metal all cutting operations were performed in inert gas (argon) atmosphere. To accomplish this a vinyl (0.020-inch thick) chamber was

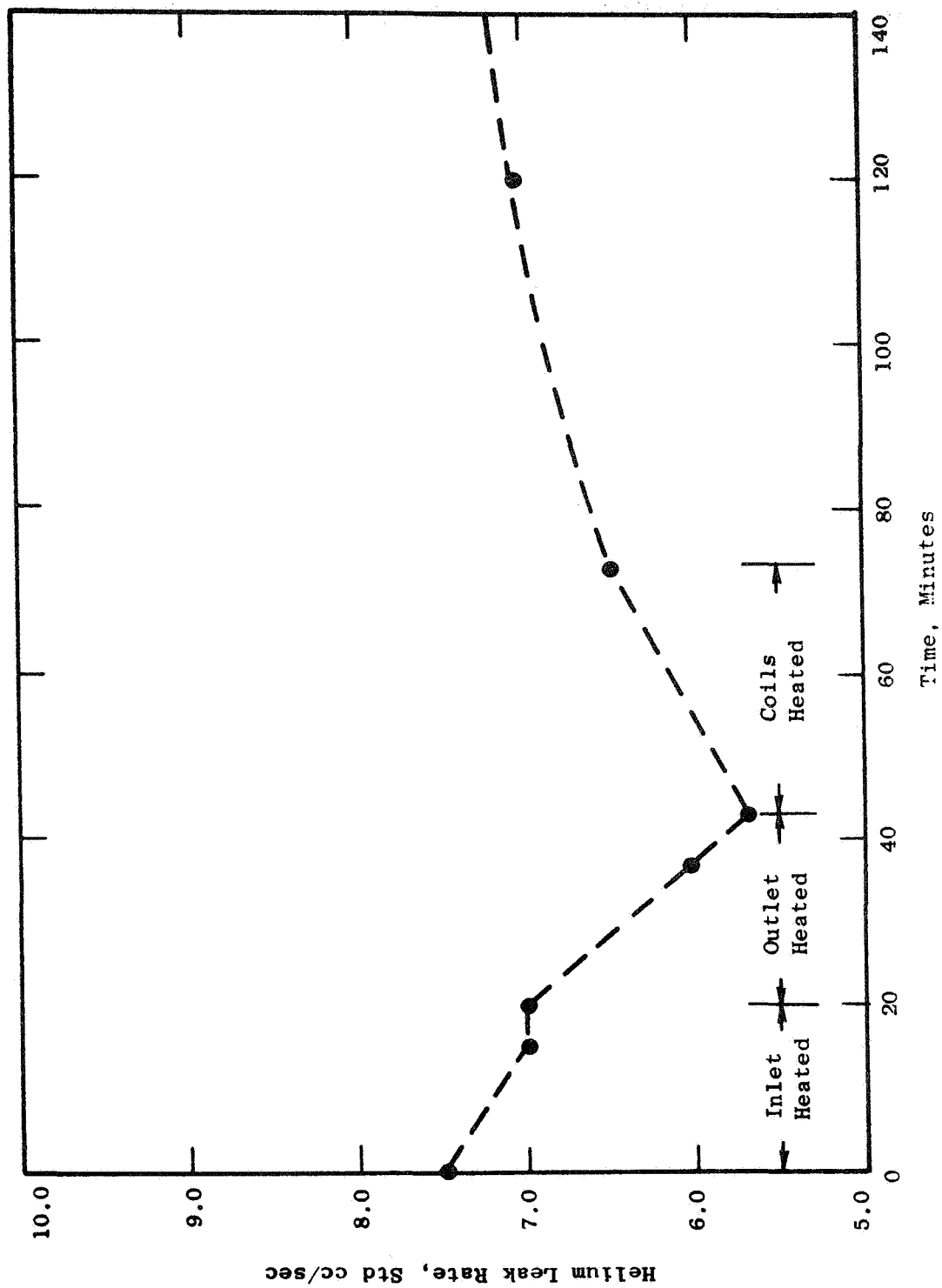


Figure 5. Effect of Heating Localized Sections of T-111 Corrosion Loop Boiler on Helium Level in Evacuated Potassium Circuit Measured with a General Electric M-60 Helium Leak Detector.

designed, fabricated, and assembled around the loop. The chamber shown in Figure 6 is fitted with neoprene gloves appropriately located for access to the areas to be cut at the top and bottom of the boiler. The chamber was sealed from the atmosphere at its base by a rubber cushioned stainless steel expansion ring pressing the vinyl against the inside of the vacuum chamber spool piece. The vacuum bell jar could therefore be lowered over the vinyl chamber and mate with the spool piece flange without interference at the sealed end of the vinyl chamber. This arrangement provided the capability of evacuating the vinyl chamber inside the bell jar. The annulus between the vinyl chamber and the bell was also evacuated to prevent collapse of the chamber. Following an overnight pump-down the chamber was backfilled with argon. Analysis of the exiting argon was performed with a Beckman Model 80 Trace Oxygen Analyzer and a Model 27901 electrolytic hygrometer shown in Figure 7. Typical analyses of less than 10 ppm oxygen and less than 70 ppm water vapor were obtained. Subsequently the bell jar would be removed and cutting operations would be initiated. Gas analysis was continued during operations in the vinyl chamber. The water vapor concentration in the chamber increased with time but typical analyses indicated maximum concentrations of less than 200 ppm when the gas filled chamber was maintained at ambient conditions. Only small increases in oxygen concentration were noted.

All boiler cuts were made with a tubing cutter to eliminate chip formation which could fall into the loop. The cutters were fitted with three cutting wheels such that cuts could be made without 360° rotation,



Figure 6. Vinyl Chamber Used for Cutting Operations on Boiler of T-111 Corrosion Test Loop. (C68062842)

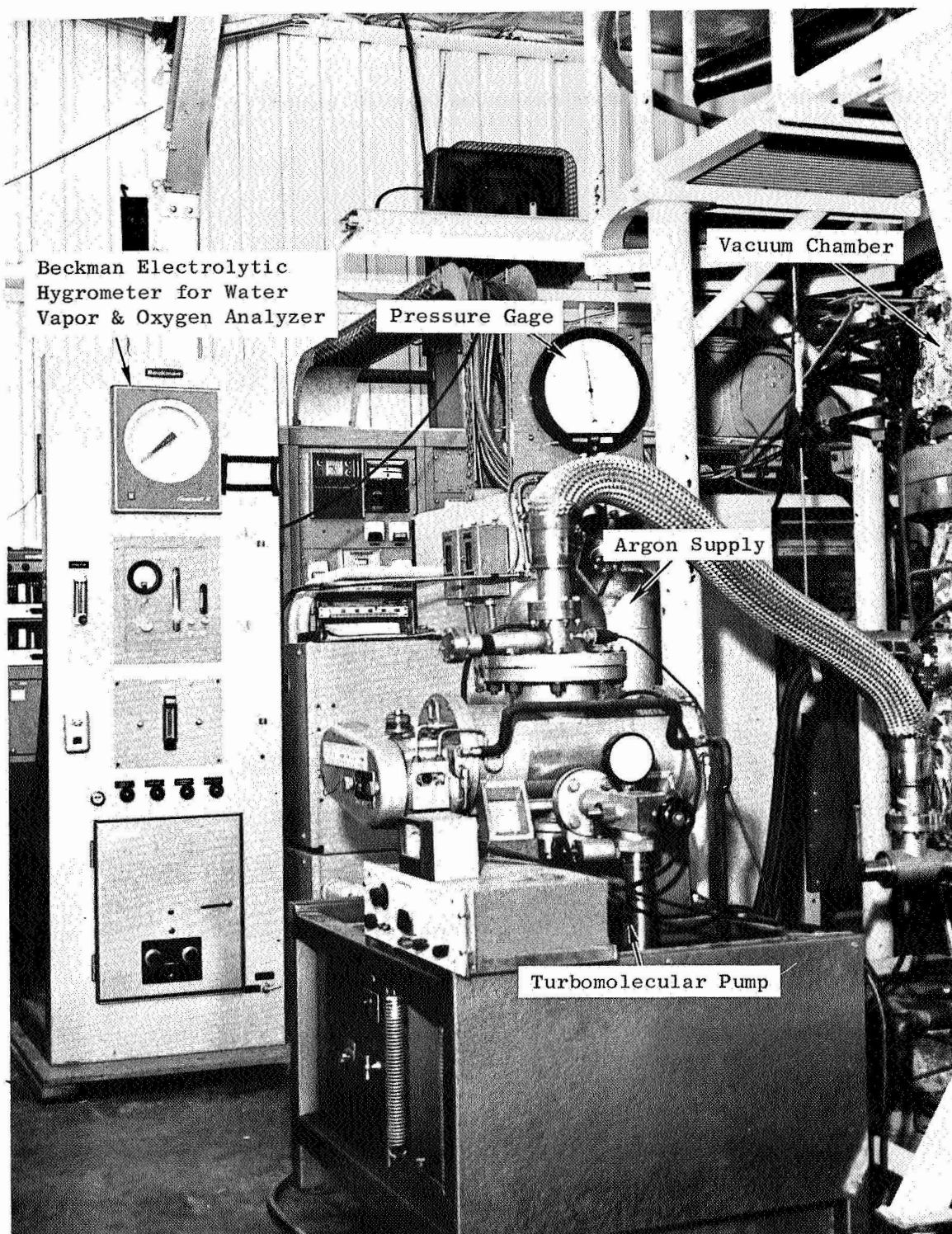


Figure 7. Vacuum Equipment and Instrumentation Used for Evacuation and Backfilling of Vinyl Chamber and Analysis of Exiting Inert Gas. (C68062846)

which was impossible in some cases because of interference with parts of the loop.

4. Removal of the Boiler From the Loop

As described previously in Section A.2 of this report the welds separating the potassium and lithium circuits of the boiler are possible sources of the leak. If the leak was at the top of the boiler in the weld shown previously in Figure 2, the boiler could be repaired without removal. The initial cut was therefore made to expose this weld for inspection and leak checking and subsequent repair if required.

On June 20, 1968 the boiler was cut exposing the top weld. The potassium circuit, which had been under a positive argon pressure was immediately sealed with expandable plugs (Imperial Model 140F Tubing Test Plugs) as shown in Figure 8. The seal is accomplished by a rubber grommet expanded against the inside diameter of the tube by the action of a brass sleeve actuated with the wing nut as shown in Figure 9.

Inspection and helium mass spectrometer leak checking of the weld did not reveal a leak at this location. Subsequent room temperature leak checking of the boiler coils and the bottom of boiler also did not reveal a leak. The vinyl chamber was removed.

The boiler was heated to 400°F at various locations as previously described with helium in the potassium circuit and the mass spectrometer leak detection pumping on the lithium circuit. Again no leaks were found at the top of the boiler; however, definite leak indications were

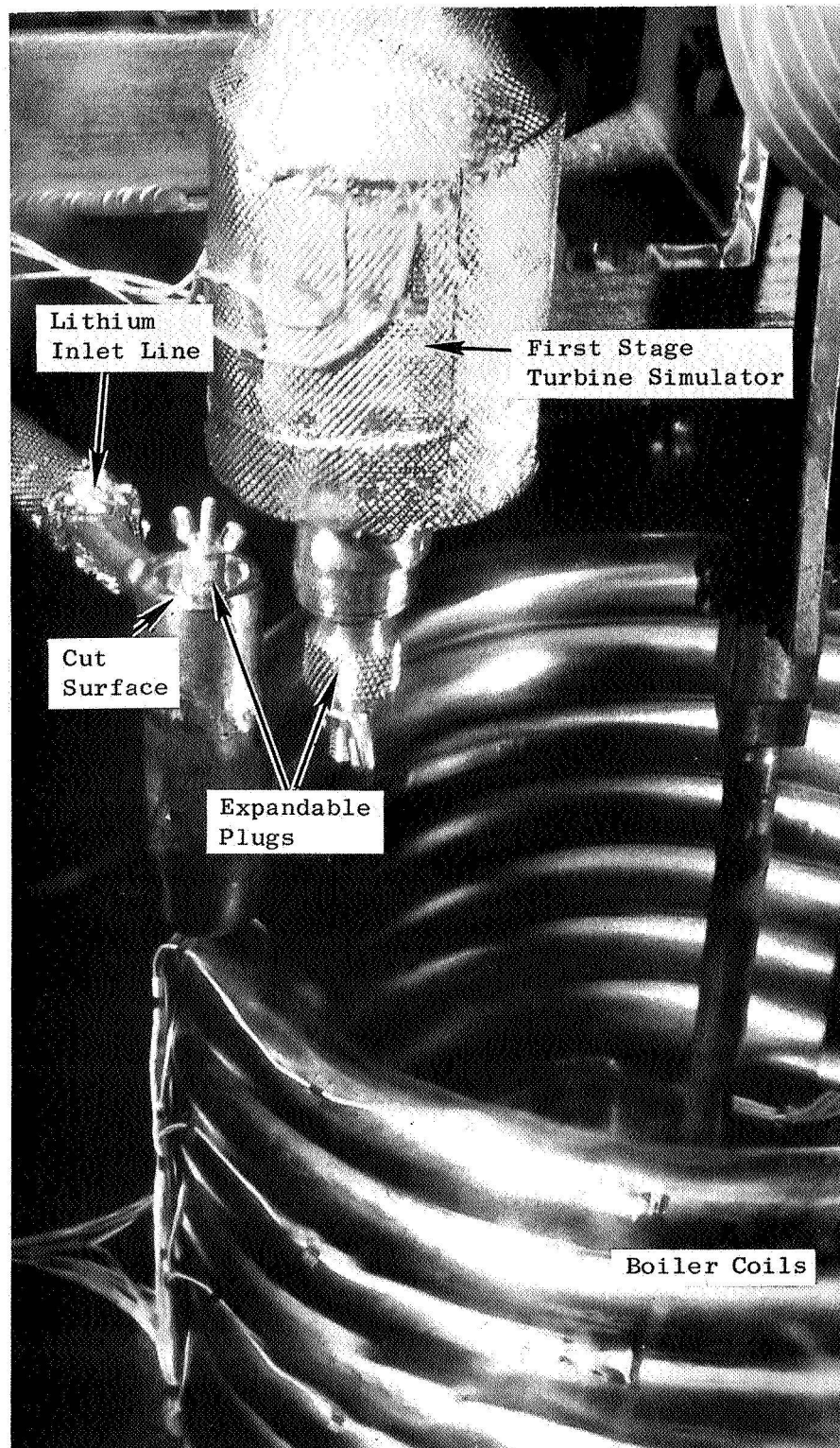


Figure 8. T-111 Corrosion Test Loop Boiler Following Initial Cut.. The Potassium Circuit was Sealed from Atmosphere with Expandable Plugs. (C68062845)

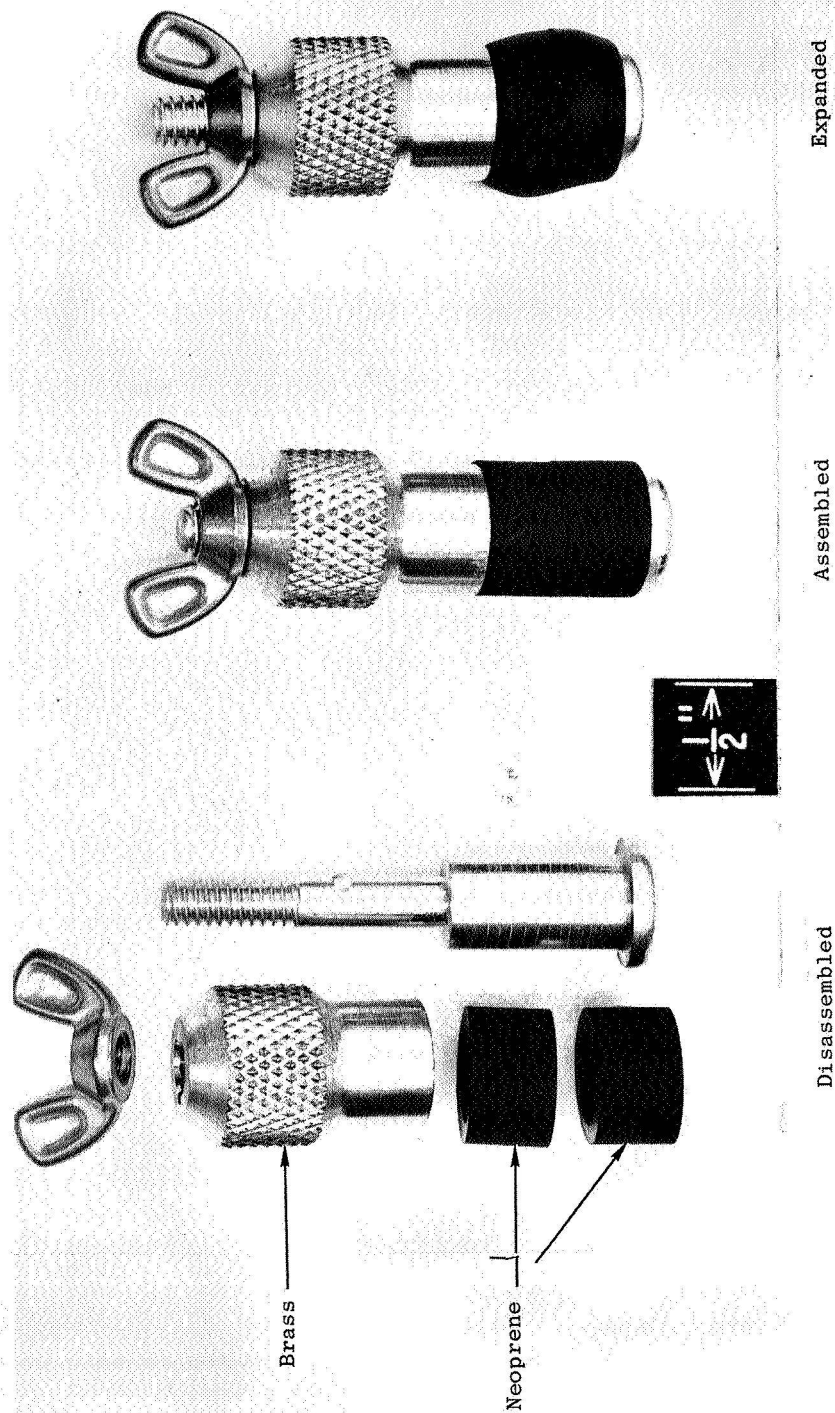


Figure 9. Expandable Plugs Used to Seal T-111 Alloy Corrosion Test Loop Tubing After Cutting. (C680719136)

noted when the center coils were heated to 400°F. The leak could not be detected when the boiler was returned to room temperature. Similar behavior was obtained in previous leak tests described in Section B and suggests the leak could be a fine crack which could plug with residual lithium (M.P. 357°F).

Since it could definitely be determined that the leak was not at the top weld and this was the only weld that could be repaired in place preparations were made to cut the boiler from the loop. Three additional cuts were required to remove the boiler: the lithium inlet line, the lithium exit line and the potassium inlet just above the top electrode as shown in Figure 10. The vinyl chamber was re-installed and the lithium inlet and outlet lines were cut and sealed. Similar techniques were employed as previously described with the exception of the seals on the boiler which were made with Swagelok fittings as shown in Figure 12. The Swagelok caps were substituted for the expandable plugs to facilitate subsequent attachment of the boiler to the cleaning apparatus for the removal of residual alkali metal. The final cut to remove the boiler was made at the potassium inlet. Tube wall thickness in this area, 0.250-inches, was too large for cutting with the tubing cutter. The wall thickness was therefore reduced by hand filing to 0.125 inches over a 0.750-inch length and V-grooved in the area to be cut to 0.050-inches prior to final cutting with the tubing cutter within the vinyl chamber. The boiler was removed from the loop on July 10, 1968.

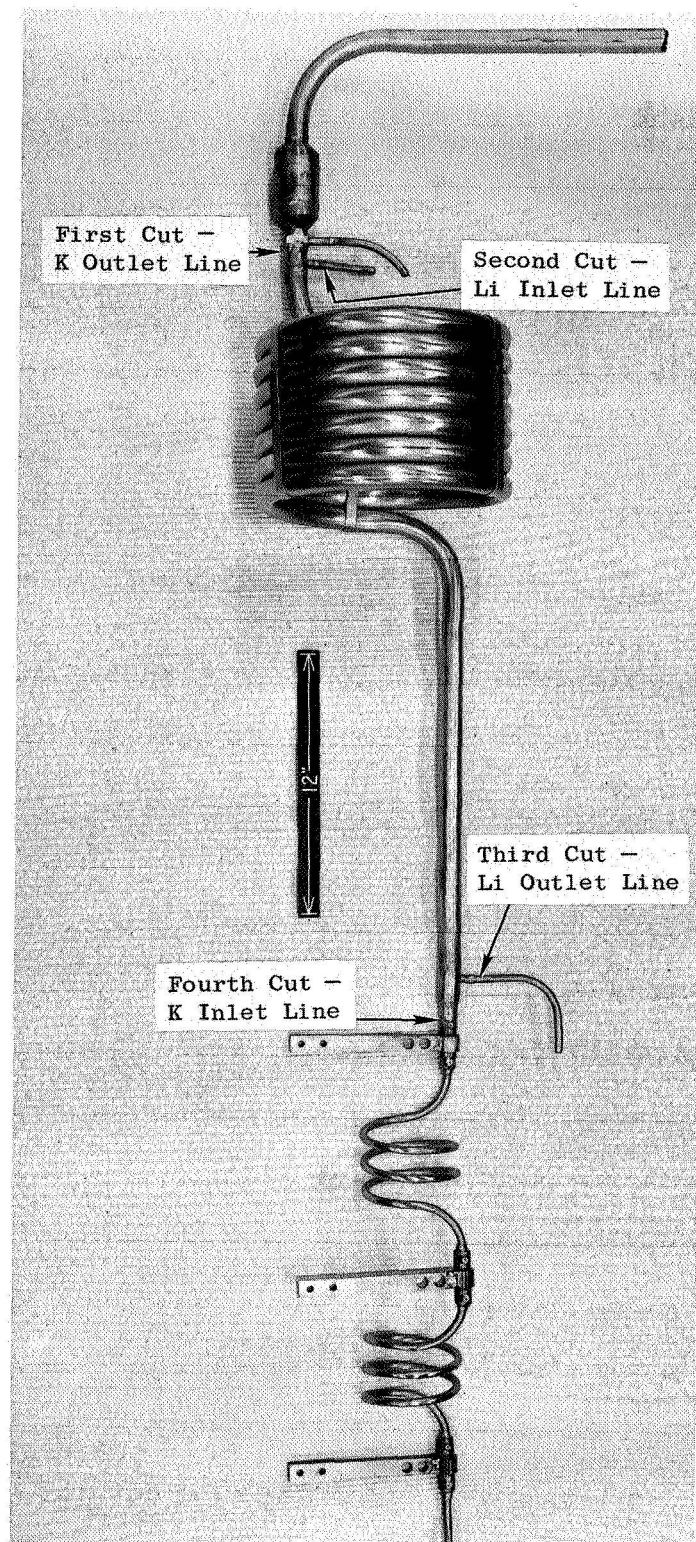


Figure 10. Location of Tubing Cuts Made to Remove Boiler from Loop as Indicated on Boiler Assembly of T-111 Rankine System Corrosion Test Loop Before Final Assembly. (C67100412)

5. Cleaning the Boiler

The proposed method of removing the residual potassium and lithium from the T-111 boiler by flushing with anhydrous liquid ammonia (99.999%, $< 10 \text{ ppm H}_2\text{O}$) was tested by cleaning a simulated boiler constructed of 0.5 inch OD, type 316 stainless steel tubing. This apparatus consisted of two coils and contained a section of 1/8-inch stainless steel rod around which wrapped specimens of 0.060-inch diameter T-111 and Cb-1Zr wire. This special section was included to simulate the plug region of the T-111 loop. The lithium drain container containing the lithium and a small amount of potassium removed from the loop was emptied into another container through the simulated boiler. Argon was then blown through the apparatus so that most of the alkali metal was removed. The residual alkali metal was then flushed out with liquid ammonia using the procedure described below for the actual boiler. The T-111 and Cb-1Zr specimens showed no significant change in carbon, hydrogen, oxygen or nitrogen concentrations as a result of the cleaning operation, thus qualifying the method for cleaning the T-111 boiler.

The T-111 boiler previously sealed in an argon atmosphere was connected to the liquid ammonia system as shown in Figure 11 and the system was evacuated up to the liquid ammonia valve. The system was back-filled with argon and the lithium and potassium circuits were tested individually to determine if a gas passage existed in each circuit. The lithium circuit was subsequently capped, and the argon flow was adjusted to 50 ml/min through the potassium circuit. With this argon

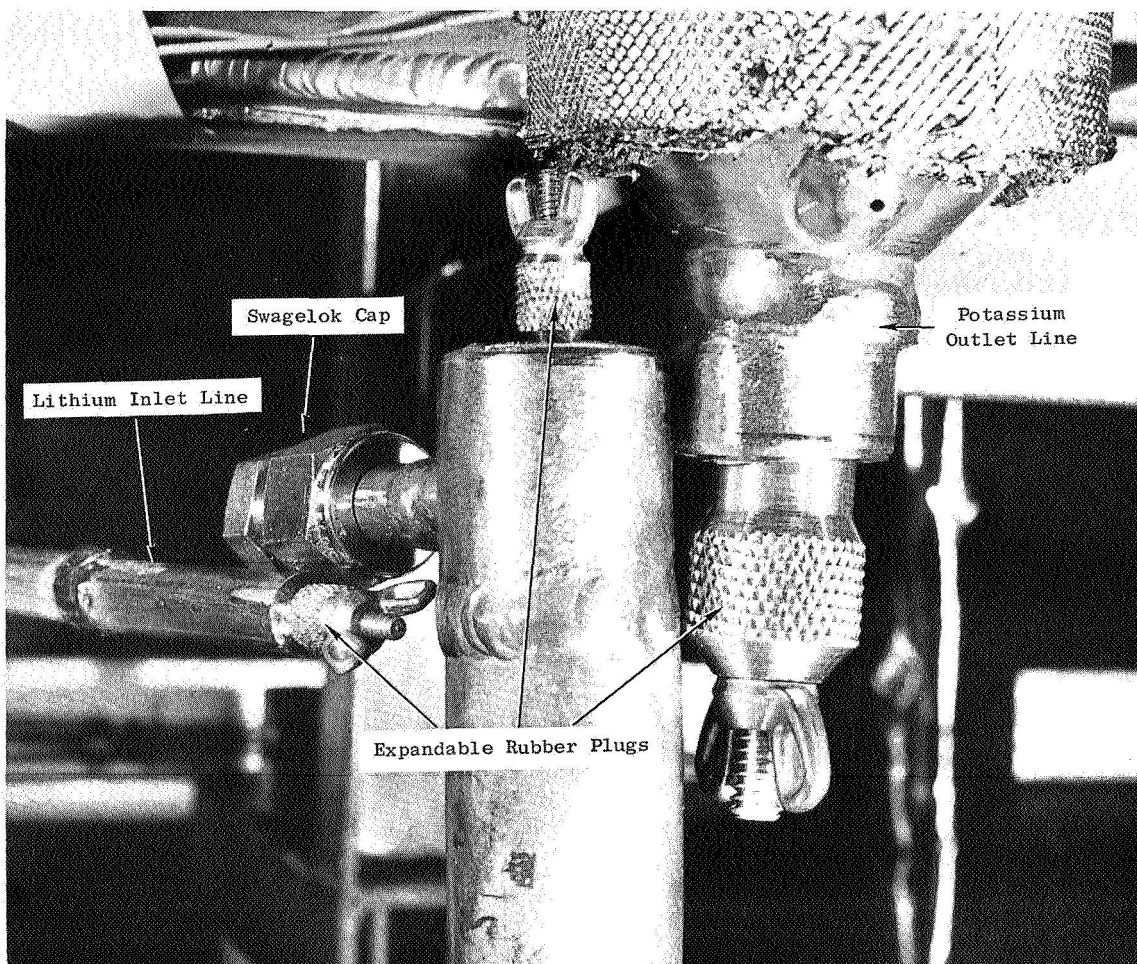


Figure 11. Top of Boiler After Cutting and Sealing Lithium Inlet and Potassium Exit Lines. (C680719137)

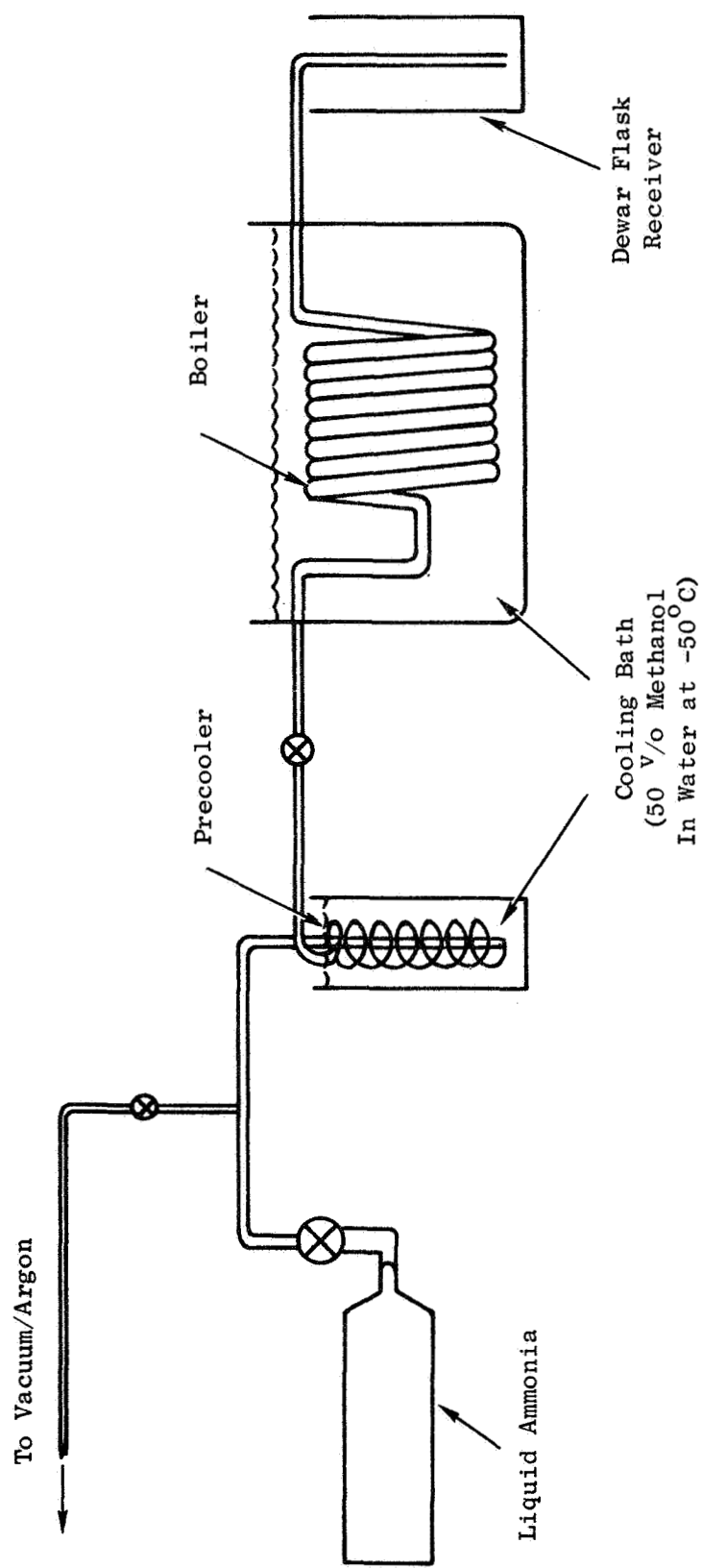


Figure 12. Schematic of Apparatus Used to Remove Residual Alkali Metal from Boiler by Dissolution in Liquid Ammonia.

flow maintained, the boiler and liquid ammonia cooling coil were cooled to -50°C (measured with a thermometer) by pouring liquid nitrogen (-196°C) into a 50 percent by volume mixture of methanol and water. When the boiler reached -50°C , the temperature necessary to maintain the ammonia in a liquid state, the argon flow valve was closed as the valve to the ammonia bottle was opened initiating ammonia flow. The first portion of liquid ammonia emerging from the boiler was dark blue in color, but after 500 ml of liquid ammonia had passed through the boiler the effluent was clear, indicating that the bulk of the potassium had been flushed out in about 10 minutes. The flushing was continued for an additional 45 minutes, at which time about 2000 ml of liquid ammonia had passed through the boiler.

The procedure for cleaning the potassium circuit was applied to cleaning the lithium circuit with similar results. The lithium circuit contained a greater quantity of alkali metal than the potassium circuit and required a greater quantity of liquid ammonia to remove the bulk of the lithium. The liquid ammonia flushing of the lithium circuit was also continued for 45 minutes after the disappearance of the blue color.

Both circuits were purged with argon at room temperature to remove residual ammonia. They were then filled* with absolute ethanol ($<500 \text{ ppm H}_2\text{O}$) which was drained after a 48 hour dwell time in the boiler. The circuits were again filled with ethanol which was subsequently analyzed by flame photometric techniques. No lithium or potassium was indicated in the analysis ($<10 \text{ ppm Li}$, $<10 \text{ ppm K}$). This procedure was repeated using

* Approximately 200 cc in the potassium circuit and 1450 cc in the lithium circuit.

distilled water with similar analytical results. The boiler was finally dried with an ethanol rinse and argon purge in preparation for locating the leak.

6. Locating the Leak in the Boiler

The cleaned and dried boiler was subsequently attached to a General Electric M-60 helium mass spectrometer leak detector. With the leak detector pumping in the lithium circuit, a helium flow was established through the potassium circuit. A leak was observed with a quantitative leak rate of 8×10^{-5} std cc/sec equivalent air leakage.

The area of the leak was determined by use of ethanol and a leveling bulb attached to the potassium circuit. The boiler was placed in an upright position with the potassium exit line at the top, and a 250 cc leveling bulb half filled with ethanol was attached to the potassium inlet line with rubber tubing. The leveling bulb was adjusted so that the level of the ethanol within the potassium tube was near the bottom of the boiler. A flow of helium was maintained into the potassium exit line to keep helium in the vapor spaces above the ethanol level, and the leak detector was adjusted to give a high output reading. By slowly raising the leveling bulb, the liquid level within the boiler was moved upward until the ethanol covered the leak as indicated by a sudden decrease in the reading of the output meter. Using this procedure, the leak was located within the coiled section of the boiler, in the second turn from the top.

The ethanol was then drained from the boiler and the helium flow continued until the boiler was dry and the leak reappeared. The boiler

was then inverted (potassium inlet line at the top) and the same procedure was repeated. The indicated leak location was at approximately the same place in the boiler as in the previous test. However, since the exact shape of the liquid interface within the boiler was not known, the precise position of the leak could not be determined. At this point, it was estimated that the leak was known to be within a section of the boiler tube approximately 20 inches long.

The ethanol was again drained from the boiler and the boiler dried until the leakage was re-established. The boiler was then placed in a horizontal position and the leveling bulb was positioned level with the axis of the coil. The boiler was then slowly rotated about its axis so that the liquid interface within the boiler moved along the length of the tube. Using this technique, the most probable leak location was narrowed to a section of tubing approximately 6 inches long.

Inspection of radiographs of the boiler which had been previously obtained, indicated that one of the butt welds in the potassium tube was near the center of the section of tubing in which the leak had been localized. It was concluded at this time that in all probability the leak was in this weld.

The boiler was then cut apart and the suspected section of potassium tube containing the butt weld was removed. The section was leak checked and no leakage was found at first. However, after soaking the tubing in boiling water for 15 minutes, the leak reappeared and was localized using

a fine helium jet. Using this helium probe technique, the leak was found to be near the center of the weld and within an area about 0.1 inch diameter. Very careful inspection of this area with a microscope revealed some surface irregularities but not obvious cracks or pinholes.

The tube was pressurized with air and the suspected leak area was coated with a soap film and observed at 30x magnification. Bubbles were observed at the leak as shown in Figure 13, and appeared to come from a hole; however, closer examination indicated a grain boundary crack as the source of the leak which is shown in Figure 14. Subsequently the weld nugget section was removed from the tube and ground longitudinally up to the crack area in preparation for metallographic examination. At this time a similar but much shorter crack was observed at 30x magnification on the ID of the tube.

7. Alkali Metal Purity

Subsequent to the loop draining operation, a number of lithium samples have been obtained and analyzed in an effort to determine the source of the aluminum and silicon which appear as contaminants. Also some additional values for oxygen in lithium (Sample 1492) as determined by the distillation method have been received from R. Gahn at NASA-Lewis. Additional values for oxygen on Sample 1492 as determined by fast neutron activation have also been received from E. Strain of ORNL. All the analytical data is presented in Table I. The good agreement on the oxygen concentration as determined by both distillation and fast neutron activation indicates the oxygen present is associated with the lithium and not with other elements.

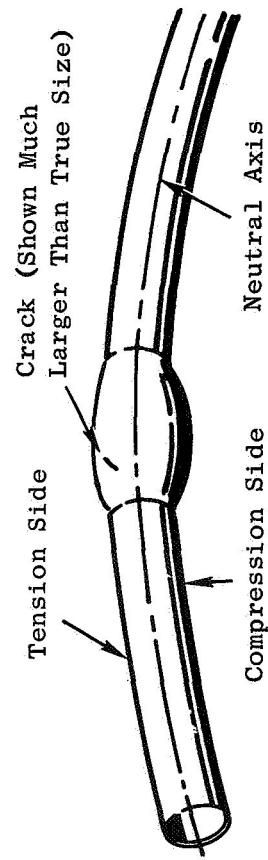
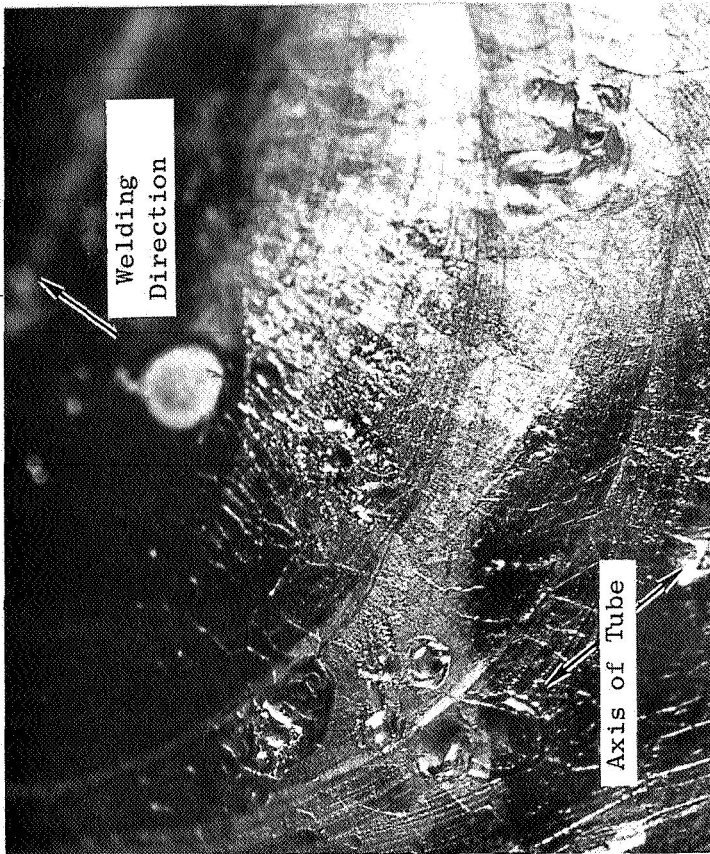
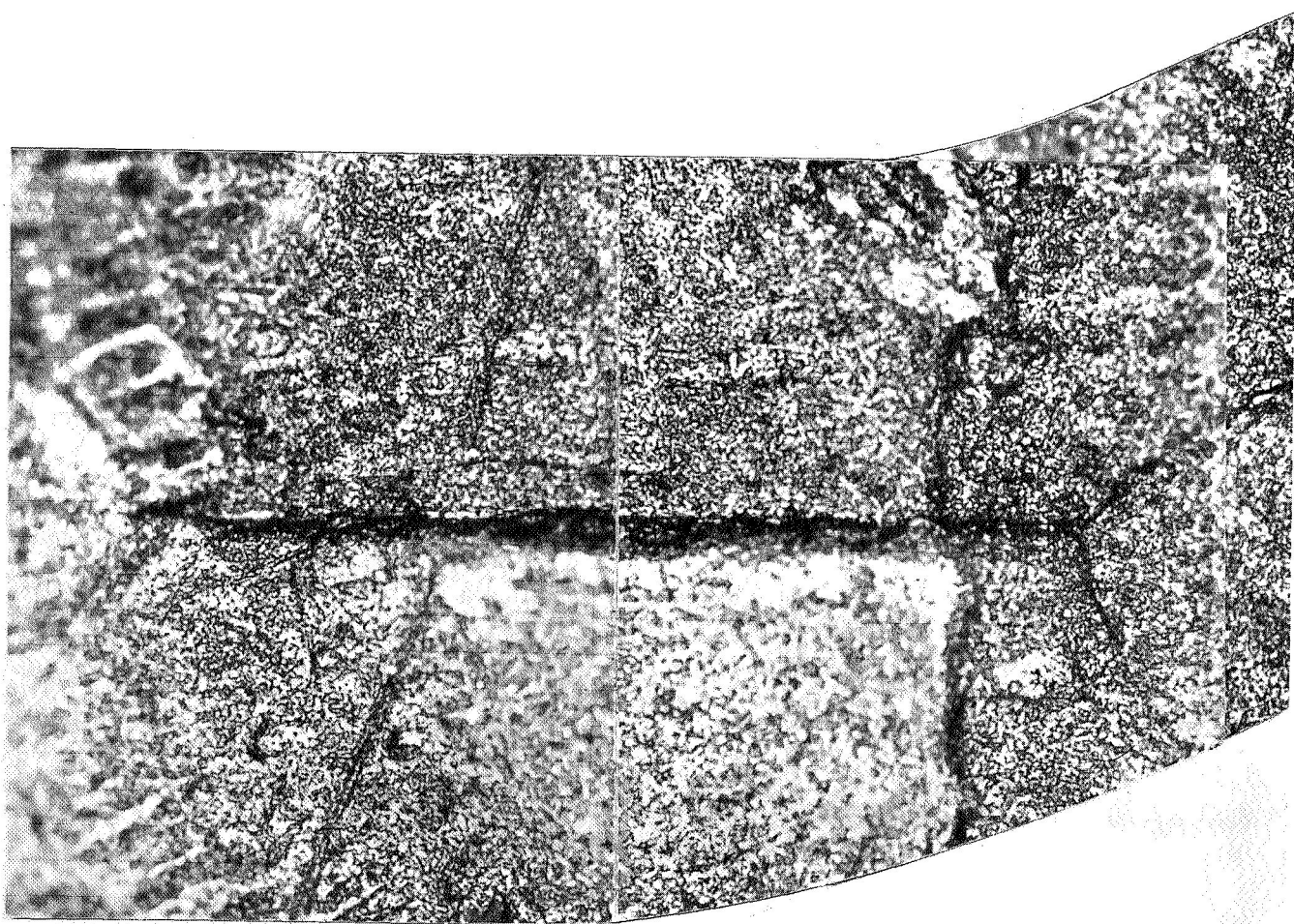


Figure 13. Location of Leak in Weld Nugget of 0.375-Inch Diameter T-111 Tube Removed from Boiler. The Bubbles were Produced by Air Pressurization of Tube with a Soap Film Coating on OD. (C68081422, C68081423, C680814250 Mag.: 10X





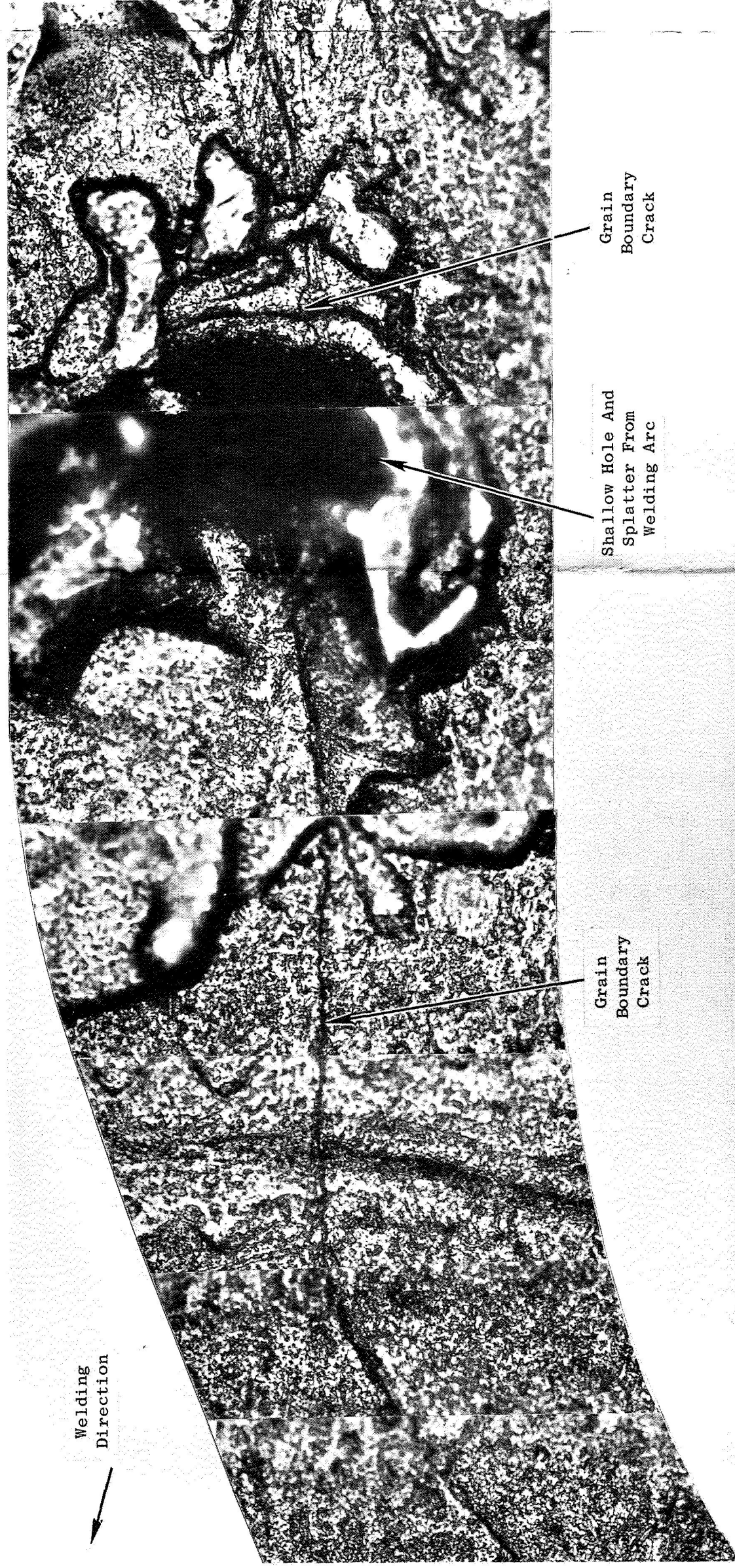


Figure 14. Grain Boundary Crack in Weld Nugget of 0.375-Inch Diameter Tube Responsible for Leak in the Boiler of T-111 Rankine System Corrosion Test Loop. (F92011-92018) Mag.: 500X

TABLE I

ANALYSIS OF THE LITHIUM FOR THE T-111 RANKINE SYSTEM CORROSION TEST LOOP

Element	Concentration, ppm in the Metal ^(a)								
	Loop Sample ^(b)				Hot Trapped ^(c)		Distilled ^(d)		Distilled ^(e)
	GE	NSL	NASA	ORNL	GE	NSL	GE	NSL	NSL
Ag	< 5				< 5	ND	< 5	ND	ND
Al	75	< 25			< 5	ND	125	185	65
B	< 50				< 50	ND	< 50	ND	ND
Ba	< 50				< 75	ND	< 75	ND	ND
Be	< 5				< 5		< 5		
C	39, 50				34		53, 33, 88		
Ca	25	< 25			25	55	25	95	50
Cb	< 25				< 25	ND	< 25	ND	ND
Co	< 5				< 5	ND	< 5	ND	ND
Cr	5	< 25			< 5	800	25	280	120
Cu	5	< 5			50	50	5	60	< 5
Fe	< 5	15			5	150	< 5	120	25
Hf									
K		< 25				< 50		< 50	Trace
Li									
Mg	5	< 10			< 5	< 50	< 5	< 50	Trace
Mn	< 5				< 5	ND	< 5	ND	ND
Mo	< 5				< 5	ND	< 5	ND	ND
N	< 5				7, 15				
Na	125	150			< 75	< 50	< 75	< 50	Trace
Ni	< 5				25	1550	< 5	700	280
O	39, 39, 18		30, 22 35, 29 32, 38 29	31, 40					
Pb	< 50				< 75	< 100	< 75	> 100	ND
Si	75	< 25			25	100	> 125	700	600
Sn	< 50				< 25	ND	< 25	ND	ND
Sr	5				5	< 50	5	< 50	ND
Ta		< 250							
Ti	< 5				< 25	ND	< 25	ND	ND
V	< 5					ND		ND	ND
W						ND		ND	ND
Zn						ND		ND	ND
Zr	< 25				< 25	ND	< 25	ND	ND

(a) The lithium sample is extruded, reacted with water in platinum and converted to Li_2CO_3 by bubbling CO_2 through the solution. The dried Li_2CO_3 is blended and split between analytical laboratories. Concentrations are calculated for the metal.

(b) After loop flush. Samples at 400°F. Sample No. 1492.

(c) From hot trap on purification system. Sample No. 1793.

(d) From still receiver sampled @ 375°F. Sample No. 1806-A

(e) From still receiver sampled @ 375°F. (Separate Li Aliquot converted to Li_2CO_3) Sample No. 1806-B.

Lithium sample No. 1793 was removed from the hot trap after hot trapping an additional batch of lithium and sample No. 1806 was removed from the still receiver after distilling this batch of hot trapped lithium. The spectrographic results for metallic impurities again show high values for aluminum and silicon, but for the first time are indicating high and variable values for iron, chromium, and nickel in distilled lithium. This has led to a decision to remove the large stainless steel, sintered powder, filter from the still receiver dip leg and sample directly from the receiver. This will be done during the next report period. Regardless of the results, it is planned to return all distilled lithium from the receiver back to the hot trap and distill it again.

It was conjectured that the particulate matter found in the potassium could have originated during welding of the stainless steel purification system since some vaporization and redeposition on inner tube walls does occur. Consequently, an experiment has been performed to determine whether or not the redeposited metal (soot) obtained during the welding of Type 321SS shows higher concentrations of the more volatile alloying elements than the parent metal since it was these elements which were found in highest concentration in the potassium.⁽¹⁾ The spectrographic results obtained on the soot were as follows:

<u>Element</u>	<u>Concentration</u>
Fe ₂ O ₃	10%
MnO, NiO	5 - 25%
Cr ₂ O ₃	1 - 10%
Al ₂ O ₃ , SiO ₂	0.5 - 5%
CrO, PbO	0.1 - 1%

<u>Element</u>	<u>Concentration</u>
CaO	0.05 - 0.5%
MgO	0.01 - 0.1%
Ag, Co, Ti, Zr	0.005 - 0.05%
As, B, Bi, Cg	Not Detected
Cd, Mo, Sb, Sn	
Sr, V, W, Zn	

The concentrations are reported as the oxides because it was assumed that the soot was present as oxide after exposure to air and oxide spectrographic standards were used.

These results indicate that, with the exception of manganese, the soot contains the alloying elements in about the same ratios as the parent metal. A considerable amount of this soot is produced at points where the greatest weld penetration occurs due to the higher quantity of molten material present and the higher temperature. In this experiment, a total of 2.9 mg of soot was obtained for a weld (in argon) which was 7 inches long; however, 1.3 mg of this total was deposited in an area of about one square inch. Such deposits, if picked up by the alkali metal during transfers to loops, could be a source of contamination in the potassium. A complete evaluation of what portions of the transfer system will have to be replaced, to prevent further addition of any entrapped particulate matter to the loop, is being made and will be finalized during the next reporting period.

B. 2600°F LITHIUM LOOP

Final assembly of the 2600°F Lithium Loop will be accomplished upon completion of the lithium heater subassembly. This subassembly can not be made until the tensile and corrosion test specimens of the ASTAR alloys

are heat treated at conditions to be specified by the NASA Program Manager.

C. ADVANCED TANTALUM ALLOY CAPSULE TESTS

Assembly of the lithium thermal convection capsule test facility has been initiated. The capsules were previously filled with lithium⁽³⁾ and are ready for testing.

⁽³⁾ Advanced Refractory Alloy Corrosion Loop Program, Quarterly Progress Report No. 11 for Period Ending January 15, 1968 NASA Contract 3-6474 CR-72382.

IV. FUTURE PLANS

- A. Complete the examination of the crack in the boiler tube to ascertain the failure mechanism.
- B. The location of the leak in the boiler was such that the boiler could be repaired by removal of one coil. The details of this approach will be recommended to the NASA Program Manager for approval.
- C. Metallographic examination and chemical analysis of T-111 samples removed from the boiler will be performed to qualify the boiler for possible reuse.
- D. Testing of the lithium thermal convection capsules will be initiated.

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