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PUMPED LITHIUM LOOP TEST TO EVALUATE ADVANCED REFRACTORY METAL ALLOYS AND SIMULATED NUCLEAR TEST ELEMENTS

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16. Abstract

This report covers the results of a program whose objective was to determine the performance of refractory metal alloys and Uranium Nitride Fuel Element specimens in flowing 1900°F (1083°C) lithium. The results demonstrate the suitability of the selected materials to perform satisfactorily from a chemical compatibility standpoint.

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FOREWORD

The work described herein was performed by the General Electric Company under the sponsorship of the National Aeronautics and Space and Administration under contract NAS 3-6474. The program was conducted during the period of _____, 196__ to January, 1972.

Mr. J. Holowach was responsible for the design, instrumentation, and initial operation of the experiment. Preparation of the specifications for the purchase of the refractory alloy materials and performance of the quality assurance testing required prior to the release of the material for fabrication were the responsibility of Mr. R. G. Frank.

Messrs. W. R. Young, P. A. Blanz, H. Mann, and C. North contributed to the fabrication aspects of the program. Messrs. R. B. Hand, L. E. Dotson, and S. A. Roof were responsible for the purification, handling, and sampling of the alkali metals used in the loop test. Messrs. H. Bradley and L. A. Paian performed the chemical analyses. Dr. T. F. Lyon was responsible for the calibration of the partial pressure analyzer and the interpretation of the spectra obtained during test operation. Messrs. T. P. Irwin and A. C. Losekamp instrumented the loop and operated the loop during the 5000-hour test. Mr. Irwin was also responsible for the disassembly of the loop following test and the preparation of specimens for chemical, metallographic, and mechanical evaluation. Mr. G. R. Anderson performed the X-ray diffraction analyses. Messrs. J. P. Smith and G. P. Brandenburg performed the metallurgical interpretation and evaluation results.

The program was administered for the General Electric Company by Mr. E. E. Hoffman. J. Holowach acted as Program Manager during the design instrumentation and test initiation phases of this program. Mr. R. W. Harrison acted as Program Manager for the test operation phase of the program. Messrs. P. L. Stone and R. L. Davies were the Technical Managers for the National Aeronautics and Space Administration.

In keeping with NASA policy, The International System of Units (S.I. units) are used as the prime units in this report. However, most of the measurements were actually made using more customary domestic engineering units which are included as the secondary units in this report.

SUMMARY

The effect of 1040°C (1900°F) flowing lithium on ASTAR 811C (Ta-8W-1Hf-0.025C) ASTAR 811CN (Ta-8W-1Re-.012 C-.012N), W-Mo-Re alloy 256 (W-25 a/o Re-30 a/o Mo) and on T-111 (Ta-8W-2Hf) clad uranium nitride fuel element specimens was studied in a pumped loop. The test loop was run for a total of 7500 hours with a scheduled shutdown at 2500 hours to insert new fuel element specimens. The purpose of the test was to determine the suitability of the selected materials to perform satisfactorily from a chemical compatibility standpoint.

The results of the 7500 hour test indicated that no chemical attack occurred in the refractory alloys under test and the operating temperature of the test did not cause any significant transfer of interstitials from the carbon and carbon-nitrogen strengthened tantalum alloys. The simulated nuclear fuel elements were evaluated at the Lewis Research and there was no chemical attach noted after the exposure to lithium.

The evaluation conducted on the test loop material and the advanced refractory alloys included metallographic and chemical analysis test. The evaluations revealed no adverse chemical attack.

INTRODUCTION

This report describes the design, fabrication, operation, and evaluation of a T-111 alloy, 1040°C (1900°F) pumped-lithium loop. The primary purpose of this test program was to evaluate, in high-velocity molten lithium, the long-term corrosion resistance of T-111-clad UN fuel element specimens such as must be used in a space nuclear power system (such as that described in ref. 1). The test was operated in two parts. In the initial 2500 hours of testing, three sound, simulated fuel element specimens were tested. At the end of this period, the loop was shut down, and the fuel element test section removed. One of the original fuel specimens was replaced with another sound specimen. Another of the original specimens was replaced with a specimen which had a machined-in longitudinal defect 6mm long x 0.0007mm wide (¼ inch long x 0.003 inch wide). The loop was then reassembled and operated an additional 5000 hours.

In addition to the fuel element specimens, the loop also contained specimen coupons for posttest tensile evaluation of the refractory metal alloys ASTAR 811CN, ASTAR 811C, and W-Re-Mo alloy 256. Also, the corrosion test section contained cylinders of ASTAR 811C, ASTAR 811CN, and T-111 alloys for evaluation of corrosion resistance. All of these specimens were contained in the loop for the total 7500 hours of operation.

At the completion of the 7500 hours' testing, the loop was disassembled, all specimens were evaluated, and some of the loop components also were evaluated. Most of the evaluation results are described in this report. However, the simulated fuel pins were evaluated at the NASA-Lewis Research Center, and the results of the evaluation are described in references 2 and 3.

LOOP DESIGN

The primary test objective was to determine the corrosion resistance of T-111-clad fuel specimens and several advanced alloys to pumped high-velocity lithium in a forced-circulation test loop. The principal design criteria are summarized in Table 1.

The loop was designed to operate for 7500 hours at a maximum temperature of 1040°C (1900°F) with a temperature gradient of 40°C (70°F) in the heat rejection section of the loop. The fuel specimen test section was designed so that it could be removed from the loop after the initial 2500 hours of operation and one of the three fuel specimens could be replaced with a fuel specimen containing a defect in the cladding. The test section then could be welded back into the loop and the test continued for an additional 5000 hours.

The loop is illustrated in the isometric drawing, Figure 1, which shows the relative position and orientation of the principal loop components. A schematic diagram of the loop at design steady-state operating conditions is shown in Figure 2. With the exception of the Cb-1Zr surge tank, the loop was fabricated from T-111 alloy, and it contained the following test specimens:

1. Tensile test specimens* (ASTAR 811C, ASTAR 811CN, W-Re-Mo Alloy 256 (W-25 a/o Re-30 a/o Mo).
2. Corrosion specimens (T-111, ASTAR 811C, ASTAR 811CN).
3. Fuel specimens (T-111-clad). T-111 (Ta-8W-2Hf), ASTAR 811C (Ta-8W-1Hf-0.025C), ASTAR 811CN (Ta-8W-1Hf-0.012C-0.012N)

The principal components of the loop and their functions are described below:

A. ELECTROMAGNETIC PUMP

A helical induction electromagnetic pump with a T-111 alloy duct circulated liquid lithium at a flow rate of 105 Kg/hr (236 pounds per hour) at an inlet temperature of 1000°C (1820°F). The duct was wrapped with Cb-1Zr foil insulation and enclosed in a stainless steel can which was welded to a 10 cm (4-inch) diameter port on the vacuum chamber completing the vacuum-tight enclosure around the T-111 alloy duct. The pump stator was mounted on an overhead trolley that allowed it to slide over the stainless steel can. The electrical windings of the stator were air-cooled by a blower mounted on the stator casing.

B. LITHIUM HEATER

The electrical resistance lithium heater consisted of two sections of .95 cm (0.375-inch) OD x .016 cm (0.065-inch) wall T-111 tubing wound in a 10 cm (4-inch) diameter coil. At each end of the heater and

at the center, a 2.54 cm (1-inch) diameter section was butt-welded to the tubes as a support and for the attachment of the electrodes. The electrodes, which are not in contact with lithium, were made from unalloyed tantalum. A 20-kw saturable-core reactor with a high-current, stepdown transformer was used to supply electrical power to the lithium heater. The heater was designed to operate at a lithium exit temperature of 1040°C (1900°F) in steady-state operation. A .032 cm (0.125-inch) diameter, T-111 alloy rod insert was welded in each heater coil to increase the local lithium velocity to 3m/sec (10 feet per sec).

C. FUEL SPECIMEN TEST SECTION

A section of the loop at the exit of the lithium heater was designed to contain three T-111-clad fuel test specimens in series as shown in Figure 3. The fuel specimens are 1.9 cm (0.75 inch) in diameter and 4.5 cm (1.79 inches) long including the end caps. Two Mo-TZM corrosion specimens were used to center the fuel specimens in the test section. The lithium flowed in the annulus formed by the fuel specimens and the test section housing at a design velocity of 1.5 m/sec (5 feet per second). The fuel specimen section was wrapped with multiple layers of Cb-1Zr foil to achieve a near-isothermal condition of 1040°C (1900°F).

D. TENSILE SPECIMEN TEST SECTION

A section in the top horizontal leg of the loop was designed to contain tensile specimens of three advanced refractory alloys: ASTAR 811C, ASTAR 811CN, and W-Re-Mo Alloy 256. The tensile specimens were inserted in pairs with a small gap separating each pair. In this manner the lithium velocity through the gap was 3 m/sec (10 feet per second) with stagnant lithium in contact with the other surface of the test specimen. Identical control specimens to those in contact with lithium were attached to the outer surface of the test section. The entire section, including the control specimens, was wrapped with multiple layers of Cb-1Zr foil to achieve a near-isothermal condition at 1040°C (1900°F). The control specimens were evaluated in conjunction with the lithium-exposed specimens to separate the thermal effects from the effects of the alkali metal exposure.

E. CORROSION SPECIMEN TEST SECTION

A section of the loop was designed to contain tubular specimens of three refractory alloys: T-111, ASTAR 811C, and ASTAR 811CN for posttest corrosion evaluation in a 2.54 cm (1-inch) diameter container tube. A .95 cm (0.375-inch) tube was located in the center hole of each corrosion specimen to increase the lithium velocity to 3 m/sec (10 feet per second). The corrosion specimen test section also functioned as a heat rejector for the loop, and its effective radiating area was increased by enclosing the 2.54 cm (1-inch) diameter container tube in a 7.5 cm (3-inch) diameter lithium-filled

jacket that was open at the bottom to the main loop. Radiation from the lithium-filled jacket to the vacuum test chamber walls provided for the 40°C (70°F) temperature gradient in the loop.

F. ELECTROMAGNETIC FLOWMETER

A permanent magnet flowmeter with a flux density of .3 tesla (3000 gauss) was used to measure the lithium flow rate. The flow tube was .95 cm (0.375-inch) OD by .016 cm (0.065-inch) wall T-111 alloy tube. T-111 alloy wire electrodes were welded to the tubes at right angles to the magnetic flux field. The EMF output of the electrodes was monitored continuously during the test to form a permanent record of any variations during the entire test period.

The tube and the magnet were partially shielded by multiple layers of Cb-1Zr foil to limit the temperature of the magnet to less than 480°C (900°F) during operation.

MATERIALS

A. QUALITY ASSURANCE FOR THE PROCUREMENT OF REFRACTORY METALS

The quality assurance program was established to provide adequate identification and documentation of the quality of the refractory metal alloys and cermets used in the construction of the high-temperature alkali metal valves and the associated test facility. The majority of the quality assurance measures were performed and certified to be within specification by the materials producer. Check tests performed by the General Electric Company, NSP, generally were limited to chemical analyses of the interstitial elements, metallographic examination, grain size and hardness measurements, and visual inspection of the incoming products.

Upon receipt of material from the materials producers, a Material Control Number (MCN) was assigned to each homogeneous lot of material. A homogeneous lot includes all material of the same size, shape, condition, and finish from one heat of material and which has received the same processing, has been annealed in the same vacuum annealing charge, and has been processed in the same manner in all operations in which the processing temperatures exceed 260°C (500°F).

B. LITHIUM PROCUREMENT AND PURIFICATION

High-purity lithium was commercially procured for the test. The material was analyzed on receipt. It was then subjected to an initial purification procedure, wherein it was filtered through a stainless steel filter having a nominal pore size of 5 microns into a titanium-lined, stainless steel hot trap containing a zirconium getter bundle. The lithium was subsequently hot-trapped at 820°C (1500°F) for 100 hours. The principal result of the hot trapping operation was the reduction in nitrogen concentration of the lithium. The final purification step was vacuum distillation at 680°C (1250°F). The distillation step reduced the oxygen and metallic impurity concentrations. The details of the lithium distillation and the effect of each step on the purity have been described elsewhere (ref 4).

C. PRELIMINARY SCREENING TESTS

The 1040°C (1900°F) pumped lithium loop was originally envisioned as a 1425°C (2600°F) Loop; however, changes in scope and interest areas revised this initial plan. During the planning of the 1420°C (2600°F) Loop it was generally thought that the T-111 and W-Re-Mo Alloy 256 would withstand the exposure with no problems; however, there was some concern as to what might happen should some slight contamination of the materials occur before test exposure. Therefore, preliminary capsule tests were performed to evaluate the effect of contamination level on subsequent lithum exposure. The capsule tests, described in detail in Appendix C, showed that these materials could be exposed in lithium up to 1420°C (2600°F) without fear of catastrophic lithium penetration.

TEST FACILITY

A. VACUUM SYSTEM

The entire loop was contained in a 60 cm (24-inch) diameter vacuum chamber capable of a cold-wall vacuum of 1.3×10^{-7} newtons/m^2 (1×10^{-9} torr). A 1000-liter-per-second getter-ion pump was used to maintain the vacuum chamber pressure in the 10^{-6} newtons/m^2 (10^{-8}-torr) range during the test. The loop was supported by a polished stainless steel structure using slotted bolts and washers to facilitate outgassing of the assembly and eliminate virtual leaks. The support structure was welded to a 56 cm (22-inch) high center spool section used to facilitate both the manufacturing and installation of the loop. The vacuum chamber was rough-pumped with a turbomolecular pump backed by a 15-cfm mechanical forepump.

The vacuum chamber consisted of three sections: the bell jar, the spool piece in which the loop was supported, and the sump. The three sections were joined together with flanges utilizing a copper wire gasket for sealing. Electrical resistance strip heaters were attached to the chamber wall to permit continuous bakeout of the chamber to 260°C (500°F). The chamber was covered with an aluminum shroud which thermally insulates the chamber, results in more uniform heating, and provides maxiumum safety to operating personnel. The chamber had water cooling channels welded to the wall.

B. CHAMBER PRESSURE AND PARTIAL PRESSURE MEASUREMENT

The vacuum chamber contained a hot filament ionization gauge and a mass spectrometer. Reduction of data from these two instruments involves a procedure in which the total pressure is obtained from the ionization gauge reading, corrected for relative concentrations of the various gas species. The relative concentrations are obtained from the mass spectrometer.

The hot filament ionization gauges may be used alone to obtain the approximate total pressure in the chamber, or what is sometimes referred to as the "nitrogen equivalent pressures." The output of the ionization gauge was fed to a strip chart recorder continuously during the test for a permanent record of the chamber pressure.

The mass spectrometer used was a 90-degree magnetic sector type with 5-cm radius of curvature. The ion detector was a 10-stage electron multiplier. A .3 tesla (3-kilogauss) permanent magnet focusses the ion beam and electrostatic scanning results in good peak separation in the mass-to-charge range between 2 and 50.

C. HEATER POWER SUPPLY AND CONTROL SYSTEM

The lithium heater power supply consisted of a combined temperature controller and stepless power regulator system with a stepdown current

transformer. The system had continuous power rating of 20 kva with 440-volt, single-phase input. The combined temperature controller and power regulator included:

1. A manual power adjustment,
2. Automatic set point control,
3. Current limiter control.

Power was supplied to the heater from a silicon-controlled rectifier with a stepdown current transformer. Output of the secondary was rated at 5 kva and 1000 amperes. The preheater temperature is controlled by a proportional temperature controller.

D. SAFETY CIRCUITS

The entire system was protected by several safety switches, interlocks, etc. to provide protection against most conceivable malfunctions. The interlocks served to minimize danger to equipment. Protection was provided by monitoring the following functions:

1. Cooling water to vacuum chamber and feedthroughs,
2. Loop temperatures,
3. EM pump winding temperature,
4. Chamber pressure.

Most type malfunctions can be observed by monitoring the preceding. Each is connected to its own safety system.

LOOP FABRICATION

All handling, assembly, welding, and cleaning were performed to established GE-NSP procedures as described in the following GE-NSP specifications:

P4AYA20-S1	Chemical Cleaning of Columbium, Tantalum, and Their Alloys:
P8AYA13-S1	Welding of Columbium, Tantalum, and Their Alloys by the Inert Gas Tungsten Arc Process;
P10DYA12	Stress-Relief Vacuum-Annealing of T-111 (Ta-8W-2Hf) Alloy;
P3CYA16	Leak-Testing Using a Helium Mass Spectrometer Leak Detector;
P3AYA14	Radiographic Inspection;
P1YA17	Thermocouple Installation - Refractory Loop Systems in High-Vacuum Environment.

A. ELECTROMAGNETIC PUMP DUCT

The EM pump duct was assembled from machined T-111 parts. Prior to inserting the helical duct in the outer wrapper, the .95 cm (3/8-inch) OD exit line was welded to the helical duct. The interference fit between the helical duct and wrapper was next performed without incident. The helical duct was immersed in liquid nitrogen to provide a diametral clearance between it and the outer wrapper which remained at room temperature. The closure welds on the end cap and connector were then made. Final machining of the outside diameter, completed the EM pump duct.

B. LITHIUM HEATER

The heater was fabricated by first installing the .3 cm (1/8-inch) diameter rod, to increase the flow rate, into the .95 cm (3/8-inch) OD x .16 cm (0.065 inch) wall straight tubing. The tubing, with the rod insert, was then formed into coils and welded to the T-111 end fittings. The tantalum electrodes had previously been welded to the end fittings. The electrodes were made from tantalum rather than T-111 since they are not in contact with lithium during the test operation.

C. FUEL ELEMENT SPECIMEN TEST SECTION

1. Fuel Element Specimens

The simulated fuel element specimens were fabricated at the NASA-

Lewis Research Center using the procedures described in reference 2. Each of the five specimens used in this test consisted of high density UN pellets clad with T-111. A thin barrier layer of unalloyed tungsten foil was interposed between the UN and T-111 to prevent any fuel-clad reactions. The T-111 end caps and clad for the UN fuel pellets were machined at GE-NSP and then shipped to NASA. Final assembly of the fuel specimens, welding, and postweld annealing were performed by the NASA. The end caps and containment tube were designed so that the caps fit inside the tube about one-quarter inch and butt against a recess on the ID of the tube. This was done to prevent the end caps from applying a compressive load on the fuel specimens during welding shrinkage; instead the loads were supported by the seat in containment tube. The assembled clad specimens were then shipped to GE-NSP for testing. One of the specimens (LT-6), used only during the final 5000 hours of testing, contained an EDM-machined longitudinal slot, .0007mm (0.003 inch) wide x 6mm (0.25 inch) long, to simulate a clad defect.

Radiographs at GE-NSP of the fuel specimens for the initial 2500-hour test (LT-1, LT-2, LT-3) showed that some of the tungsten dome-shaped end spacers (ref. 2) had become broken sometime between the assembly at NASA and insertion into the test loop at GE-NSP, presumably due to the brittle nature of the tungsten. It was decided that the 2500 hours of testing could be performed without adversely affecting the result; however, the dome-shaped spacers for the final 5000-hour specimens (LT-5 and LT-6) were formed from T-111 rather than tungsten. Radiographs of these specimens at GE-NSP prior to testing showed the spacers to be intact; however, one of them was dimpled somewhat, presumably due to a slightly tight fit during assembly.

2. Specimen Housing

Components of the fuel test subassembly are shown in Figure 4. All hardware, including the fuel element cladding, was T-111 except for the Mo-TZM spacers. These spacers served a dual purpose in that they also were evaluated for their corrosion resistance. The fuel specimens were interlocking in that the male end of one specimen fit into the female end of the other specimen. The interlocking feature of the fuel specimens and Mo-TZM spacers provided the necessary uniform gap between the fuel specimen and the ID of the containment tube to produce the required 1.5 m/sec. (5 ft/sec) lithium flow in this test section. It also facilitated easy removal of selected specimens at the end of the first 2500 hours' operation for replacement with a defective-clad specimen. Since the fuel specimens were solid, all lithium flow was along the narrow annulus between the specimen and the containment tube.

This subassembly was situated vertically in the loop, the bottom cap being welded directly to the heater assembly and the top cap welded to the tensile section.

D. TENSILE TEST SPECIMEN SUBASSEMBLY

Components of the tensile specimen subassembly are shown in Figure 5. All material, except the specimens, was T-111 alloy. This subassembly consisted of three individual specimen holders, each of which contained two (except for the W-Re-Mo Alloy 256 .1 cm (0.040-inch) thick coupons held within close-fitting slots on the inside and two specimens strapped to the outside flats shown in Figure 3. The W-25Re-30Mo* coupons were only .05 cm (0.020 inch) thick; therefore, each slot contained two coupons instead of one for a total of four specimens in that particular specimen holder. The gap between the "inside" specimens was controlled to obtain the desired lithium velocity of 3 m/sec (10 ft/sec) over one surface of each coupon. The coupons strapped to the outside of each specimen holder were used as control specimens since they saw the same thermal history as the inside coupons but were not exposed to lithium. The holes in the "inside" coupons, as shown in Figure 6, were for pins to prevent movement during the high-velocity lithium flow.

Final heat treatments of the specimens prior to insertion into the specimens holders were as follows:

ASTAR 811C - Cold-rolled; heat-treated 1/2 hour at 1980°C (3600°F) in vacuum;

ASTAR 811CN - Recrystallized 1 hour at 1650°C (3000°F) in vacuum;

W-Re-Mo Alloy 256 - 20 minutes at 1400°C (2550°F) in hydrogen.

The sequence of fabrication for the tensile specimen subassembly was as follows: (1) insert interior specimen coupons and pins into each of the three specimen holders, (2) weld the three holders together, and (3) weld the end fitting and blowdown tube to the specimen holders. The completed subassembly is shown in Figure 7. The "outside" specimen coupons were attached after assembly welding of the various subcomponents and were included in the postweld anneal.

Fixtures at the top of the end fitting were used to hang the loop in the support structure during test operation. The blowdown tube was used to aid removal of lithium at the completion of the test. All specimen coupons were cut into standard tensile test specimens at the completion of the 7500 hours of testing for posttest evaluation.

E. CORROSION TEST SPECIMEN SUBASSEMBLY

All components and specimens for the corrosion test specimen subassembly are shown in Figure 8. The parts shown were fabricated from

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* Atomic percent

T-111 with the exception of the ASTAR 811C and ASTAR 811CN specimens. Heat treatments of the specimens before loading into the loop were as follows:

T-111 - Recrystallized 1 hour at 1315°C (2400°F) in vacuum;

ASTAR 811C - Recrystallized 1 hour at 1650°C (3000°F) in vacuum, cooled to room temperature, reheated for 1/2 hour at 1980°C (3600°F) in vacuum;

ASTAR 811CN - Recrystallized 1 hour at 1650°C (3000°F) in vacuum.

Because of raw material restrictions for the ASTAR 811CN, it was necessary to make eight disc-shaped pieces and stack these to be equivalent in length to one of the ASTAR 811C and T-111 specimens. A .05 cm (0.020-inch) T-111 wire was wound around the center rod (two-inch pitch) to improve flow characteristics and to aid in maintaining concentricity. Because of the number of parts involved and the close fits involved, this was the most difficult subassembly to put together. The sequence of operations was as follows:

a) weld top alignment fixture to center rod,
b) weld bottom alignment fixture to specimen containment tube,
c) spot weld .05 cm (0.020-inch) wire to center rod,
d) slip connector over center rod,
e) slip specimens over center rod,
f) insert specimens and center rod assembly into the specimen containment tube,
g) weld specimen containment assembly to connector,
h) weld connector, end caps, and lithium jacket to complete assembly as shown in the right half of Figure 8.
i) finally, the extension tube, fitting, and tubes to the EM pump and surge tank were welded.

This subassembly was designed so that the lithium in the annulus between the ID of the specimens and the center rod flows at 3 m/sec (10 ft/sec). Because of the close fit between the OD of the specimen and the ID of the containment tube, essentially no flow occurs at this interface. Also, there was a .95 cm (3/8-inch) gap between the exit of the corrosion section (bottom alignment fixture) and the bottom end cap; this allows the cavity between the OD of the specimen containment tube and the ID of the jacket to fill with essentially static lithium. The purpose of the static lithium reservoir was to aid in the heat rejection in this subassembly to provide the necessary 40°C (70°F) temperature gradient between the lithium entering the corrosion specimen test section and the lithium exiting.

F. SURGE TANK

Because of its low operating temperature, the surge tank was the only portion of the loop fabricated from Cb-1Zr. The tank shell was

fabricated initially by seam-welding a piece of rolled plate. The Cb-1Zr tubing for gas pressurization, draining, and filling were then welded in place. Finally, the end caps were welded in place to complete this component. The surge tank and related tubing were postweld vacuum-annealed for 1 hour at 1200°C (2200°F).

G. FINAL ASSEMBLY OF THE LOOP

The heater, fuel test section, tensile specimen section, and corrosion specimen section subassemblies were located in their proper orientation and welded together to form the U-shaped test section portion of the entire loop. This assembly was then postweld annealed in the vacuum chamber at Stellite Division of the Cabot Corporation, Kokomo, Indiana. Results of chemical analysis of T-111 test coupons heat-treated with the loop are shown in table V-I. The assembly was then bolted into the stainless steel support structure. The EM pump and surge tank were fitted into their proper locations, held in place with temporary supports, and transferred to the welding chamber. All final refractory metal welds and postweld anneals were then performed. These welds were subsequently radiographed and helium mass spectrometer leak-checked and found to be free of defects. The loop and support structure (shown in Figure 9) were then mounted on the 60 cm (24-inch) diameter spool piece and transferred to the portable laminar flow clean room facility.

LOOP INSTALLATION AND INSTRUMENTATION

A. THERMAL INSULATION

Thermal insulation consisting of multiple layers of Cb-1Zr foil was simultaneously applied to the loop as the thermocouples were installed. The foil used on all circular pipe sections was .005 cm (0.002 inch) thick x 1.2 cm (0.5 inch) wide, which had been dimpled by passing the foil between a hardened steel, coarse-knurled roller working against a hard plastic sheet. The effective thickness of the foil after dimpling was between .023 to .030 cm (0.009 to 0.012 inch). The insulation was attached by spot welding the foil to the loop and to itself as succeeding layers were applied. A minimum number of spot welds were used to minimize conduction heat losses through the foil. A molybdenum spot welder electrode was used to avoid contamination of the foil surfaces with copper, and an argon cover gas was used to protect all welded areas from oxidation. The entire loop was insulated as described above except for the corrosion specimen section which is to act as the heat rejection section of the loop. The outer surface of the 7.5 cm (3-inch) diameter lithium containment tube, which surrounds the tube containing the corrosion specimens, was grit-blasted with Al_2O_3 to produce an effective emmitance of approximately 0.4.

B. INSTRUMENTATION

All thermocouples installed on the loop were split-junction, W-25Re/W-3Re thermocouples which were insulated with Al_2O_3. The completely assembled, instrumented, and insulated loop is shown in Figure 10. The thermocouple feedthroughs and cold-junction, reference-temperature compensation have been previously described in detail (ref 5).

C. INSTALLATION OF THE SPOOL PIECE IN THE TEST FACILITY

At the completion of the preceding steps, the loop was covered with a protective polyethylene bag and moved from the fabrication and assembly area to the test area. The spool piece and loop were clamped to the 60 cm (24-inch) diameter vacuum chamber sump, and all final electrical, water, and mechanical connections were made. All thermocouples were then checked for continuity to the control panel. The complete loop and vacuum system ready for enclosure within the bell jar is shown in Figure 11.

PRETEST OPERATION

A. PUMPDOWN AND BAKEOUT OF THE VACUUM SYSTEM

The bell jar was located in place on the spool piece, clamped down, and pumpdown of the chamber was initiated. Vacuum chamber bakeouts and the surge tank heater were turned on when the chamber pressure reached 1.3×10^{-5} newton/m^2 (1×10^{-7} torr), and bakeout was allowed to continue until after the loop was filled with lithium. Prior to filling, the loop was pressurized with argon to 35×10^4 newtons/m^2 (50 psig) and an argon mass scan taken in the vacuum chamber to verify that no leaks were present prior to exposure to lithium.

B. FILLING THE LOOP WITH LITHIUM

With the spool piece located in place on the vacuum chamber sump, the necessary stainless steel gas and liquid metal lines were welded between the loop and the liquid metal transfer system. Bakeout and pumpdown of all loop, fill, and transfer lines was begun immediately. The lithium used to fill the loop was high-purity, vacuum-distilled, and hot-trapped of an analysis shown in Table 3.

The filling procedure was as follows:

a. Heat loop with vacuum chamber bakeout and surge tank heater to at least 200°C (400°F);
b. Sample lithium from charge pot for oxygen and nitrogen;
c. Fill surge tank with approximately 3500 cc of lithium;
d. Pressurize surge tank to 35 newtons/m^2 (50 psig), thus filling the loop with lithium and allow to circulate 30 minutes;
e. Dump lithium into surge tank;
f. Repeat d and e for a total of three flushes;
g. Sample lithium from surge tank for oxygen and nitrogen analyses.

The analysis of the lithium sample, Table 3, taken from the loop after flushing indicated 28 ppm oxygen and 2-3 ppm nitrogen, both of which were well within the allowable maximum limits.

C. CHECKOUT AND CALIBRATION OF LOOP INSTRUMENTATION

Before bringing the loop to full design operating conditions, the lithium heater and EM pump power supplies and controllers were checked out for response and reliability. Also, all safety circuits were checked out to ascertain that they operated as required. Finally, the thermocouples were checked out at a low-lithium flow rate (low ΔT in loop) to verify their calibration. All these operations were performed at a maximum lithium temperature of 540°C (1000°F).

LOOP OPERATION

A. LOOP STARTUP

The loop was operated on manual heater temperature control for the first 100 hours. Since only minor power adjustments were required during this initial 100 hours of operation, the heater power was switched to automatic control and subsequently operated in this mode for the entire test with less than 5°C (10°F) temperature variation. During the first 500 hours, the loop experienced several shutdowns, totaling 55 hours, resulting from safety circuits being activated. The major cause of the shutdowns was the result of voltage instabilities caused by inclement weather. None of the shutdowns were caused by loop instabilities. Two steps were taken to minimize shutdowns in the future. First, an alarm system was connected with the GE-NSP Security Headquarters and was activated when the loop temperature exceeded preset boundaries during nonregular working hours. Secondly, a 440-volt, 100-kva constant-voltage transformer was connected to the main power supply. After the installation of the voltage stabilizer, only two hours' total down time was encountered and this was caused by temporary electrical outages in facility power.

B. LOOP PERFORMANCE DURING INITIAL 2500-HOUR TEST

1. Loop Temperatures

The loop temperatures remained very stable during the entire 2500-hour operating period. Figure 12 shows temperatures typical of the 2500-hour operation. Unfortunately, a number of the thermocouples on the heat rejection jacket became inoperative during this test period; however, more than sufficient data was obtained from the remaining operative thermocouples. Subsequent examination at the planned 2500-hour shutdown showed that failure had occurred because of inadequate allowance for differential thermal expansion between the W/Re thermocouples and the T-111 jacket. The defective thermocouples were repaired at that time and operated satisfactorily during the final 5000 hours of testing as discussed later in this section.

2. Test Chamber Environment - Partial Pressure Gas Analysis

The chamber pressure and partial pressure of the various gaseous species in the test chamber during the 2500-hour test period are shown in Figure 13. During the initial 100 hours, all pressures dropped rather sharply; this was followed by a somewhat decreased rate of pressure drop during the next 650 hours, and during the remainder of the test there is a general trend for the partial pressure of most gaseous species to be leveling off. Data points are shown only at 250-hour intervals for clarity; however, residual gas analyses are obtained at least every 24 hours; and the ion gage pressure is monitored continuously. This data for the 1040°C (1900°F) Lithium Loop is in good

agreement with the T-111 loop data presented elsewhere (ref 5) in that H_2, N_2-CO and Ar are the major gas species present in the chamber.

C. TERMINATION OF THE 2500-HOUR LOOP TEST

1. Loop Shutdown

Upon completion of the 2500 hours of testing, the lithium was hot-dumped (1000°F) into the surge tank and then immediately drained into the alkali metal transfer system charge pot and a sample taken. The loop was then pressurized through the blowdown line to further remove any lithium. Analysis of the lithium compared to the pretest analysis is shown in Table 4. The increased nitrogen is somewhat unusual, especially in view of the fact that after the 7500-hour test period, the nitrogen analysis is essentially the same as the pretest value.

2. Lithium Distillation

After cooling all components to room temperature, the vacuum chamber bell jar was removed. Visual inspection of the loop showed all components to be in excellent condition. The Cb-1Zr dimpled foil thermal insulation was removed from the fuel specimen test section. Two new layers of foil were applied and a tungsten resistance furnace placed around the fuel specimen test section; finally, the bell jar was replaced on the spool piece. After evacuating the loop and vacuum chamber, the bakeout ovens and the surge tank heater were turned on to bring all loop temperatures to a minimum of 200°C (400°F). Residual lithium was then distilled from the fuel test section for eight hours at 1040°C (1900°F) utilizing the tungsten resistance heater. During distillation, the loop was evacuated using the getter-ion pump in the lithium transfer system. After cooling all components to room temperature, the bell jar and distillation furnace were removed, and all electrical, gas, and liquid metal lines were disconnected from the spool piece.

D. REMOVAL AND REPLACEMENT OF FUEL ELEMENT TEST SECTION

After completion of the above operations, the loop was moved from the test area to the weld laboratory. The loop and spool pieces were then mounted in the motor-driven, rotary weld fixture which was installed in an 3 meter (eight-foot) diameter weld chamber. The weld chamber was then evacuated and backfilled with helium according to GE-NSP Specification P8AYA13-S1. The fuel test section was removed by cutting the loop with a tubing cutter at locations (1) and (2), shown in Figure 14, and the open ends of the loop plugged with expandable stoppers prior to opening the weld chamber to air. The fuel specimen subassembly was radiographed to verify the integrity of the specimens, and finally a cut was made at location (3), shown in Figure 14 to remove the fuel specimens. Visual examination showed all components to be in excellent condition, and no traces of lithium were present, indicating the effectiveness of the 1040°C (1900°F) distillation. The appearance of the T-111-

clad fuel specimens and Mo-TZM spacers before and after the 2500-hour lithium exposure was nearly the same, with some discoloration of the Mo-TZM spacers being observed. After removal of the fuel specimens from the housing, they were radiographed to verify their integrity; no difference could be seen between the pretest and posttest radiographs.

After pickling, the fuel test section was reassembled reusing the same T-111 end caps and housing. Specimens were inserted in the following sequence:

Lithium Flow ↓

1. T-111-clad fuel element, LT-5: untested;
2. Mo-TZM spacer: untested;
3. T-111-clad fuel element, LT-2: tested during the initial 2500 hours of the 1040°C (1900°F) Lithium Loop;
4. Mo-TZM spacer: tested during the initial 2500 hours of the 1040°C (1900°F) Lithium Loop;
5. T-111-clad fuel element, LT-6: untested containing a longitudinal clad defect (planned).

This subassembly was then rewelded at location (3) (Figure 14) and then welded back into the loop at locations (1) and (2) . The loop was then removed from the weld chamber and fixture and subsequently moved back to the test area. All thermocouples were repaired; two layers of Cb-1Zr dimpled foil were applied to the fuel test section; and the tungsten resistance heater was reassembled around the fuel test section. The spool piece and bell jar were placed on the vacuum chamber sump, and pumpdown of the chamber was initiated in preparation for the postweld anneal. The anneal was performed for one hour at 1315°C (2400°F) with a maximum chamber pressure of 8×10^{-4} newtons/m^2 (6×10^{-6} torr). During the anneal, all vacuum chamber bakeout heaters and the surge tank heater were turned on to maintain loop temperatures at a minimum of 200°C (400°F). Also, during the anneal the loop was evacuated through the blowdown line to prevent pressure buildup by the high temperature. The loop was leak-checked before and after the anneal by pressurizing the loop with 35×10^4 newtons/m^2 (50 psia) argon through the blowdown line while monitoring the vacuum chamber for argon with the partial pressure gas analyzer. No leaks were detected during either check.

After removal of the annealing furnace, the fuel specimen test section was wrapped with Cb-1Zr dimpled foil.

At the completion of the postweld anneal on the new fuel specimen test section, the loop was filled with lithium and sampled as described elsewhere. Analysis of the lithium indicated 183 and 200 ppm nitrogen and 132 ppm oxygen. The high nitrogen content cannot be explained at this time; however, the ASTAR 811CN specimens and possibly the T-111-clad UN fuel specimens could be sources. It should also be noted that the nitrogen was high (120 ppm) at the completion of the initial 2500 hours of testing. Based on these facts and with the

concurrence of the NASA Program Manager, it was decided to proceed with the test.

E. LOOP PERFORMANCE DURING THE FINAL 5000 HOURS OF TESTING

The lithium heater power and EM pump power were gradually increased to bring the loop back to the design operating condition. Again, power rate increases were kept low enough so that the outgassing rate was low enough to maintain the chamber pressure below 1.3×10^{-5} newtons/m^2 (1×10^{-7} torr). In bringing the loop back to operating conditions, it was discovered that an argon pocket existed at the top of the heat rejection lithium jacket. A review of the situation indicated that the presence of the gas pocket would not jeopardize the test. The lithium jacket was included in the design since the loop originally was to be operated at 1425°C (2600°F). For 1040°C (1900°F) operation, the temperature drop is 40°C (65°F), even with the gas pocket, as shown in Figure 15. This is sufficiently close to the calculated design temperature drop of 40°C (70°F); therefore, it was decided, with the concurrence of the NASA Project Manager, to continue the test for the remaining scheduled 5000 hours without attempting to remove the argon pocket. The test operated very stably with no indications of gas in the loop itself. Typical operating conditions after the restart of the test following the planned shutdown to replace the fuel element specimens are shown in Figure 15. These temperatures agree quite well with the temperatures during the initial 2500 hours of testing as shown earlier in Figure VIII-1. The chamber pressure and partial pressure of the various gaseous species in the vacuum chamber since the restart are shown in Figure 16 along with comparable data for the initial 2500 hours of testing. No significant differences can be seen for the two test periods. In summary, it can be said that the test operated very smoothly, trouble-free, and essentially on design conditions for the entire 7500-hour test period.

F. TERMINATION OF THE FINAL 5000 HOURS OF TESTING

At the completion of the planned 7500-hour test period, the maximum loop temperature was decreased to 815°C (1500°F). The vacuum chamber bakeout heaters, surge tank heater, and alkali metal transfer system heaters were turned on to bring all temperatures to 200°C (400°F) minimum. The surge tank was then evacuated through the gas pressurization line to allow the lithium to drain from the loop into the surge tank. After the lithium cooled to 540°C (1000°F), it was transferred to the alkali metal handling and sampling system by argon pressurization through the loop blowdown line and opening the valve to the handling system. The valve was closed and the loop again evacuated. The loop was repressurized through the blowdown line and the drain valve opened again; this procedure was repeated until no change was detected on the level probe in the charge pot, indicating that no further drainage was occurring. The loop was finally pressurized with argon to 17×10^4 n/m^2 (25 psig), all valves closed, and electrical power shut off. The analysis of the lithium at the completion of the 7500 hours compares quite favorably to

the pretest values as shown in Table 5. The reason for the abnormally high nitrogen analysis obtained on the lithium during the scheduled 2500-hour shutdown is not apparent.

Following completion of the lithium analysis, the bell jar was removed and the loop carefully examined. No anomalous or unusual features were observed, and all components appeared in good condition.

POSTTEST OPERATIONS

A. LITHIUM DISTILLATION

Vacuum distillation was selected as the method of removing residual lithium from the fuel element test section and the corrosion test section. This procedure was also utilized to remove lithium from the fuel element test section following completion of the 2500-hour test as described elsewhere. The dimpled foil, thermal insulation was first removed from the 2.54 cm (1-inch) fuel specimen containment tube; two new layers were then applied and a tungsten resistance furnace placed around the test section.

Because of the many small interstices and the close-fitting parts in the corrosion specimen test section, it was also decided that distillation of this section would facilitate removal of residual lithium during subsequent ammonia cleaning. A furnace consisting of four 45 cm (18-inch) long quartz lamps was fabricated and placed around the corrosion test section.

After installing the furnaces, the bell jar was replaced and the chamber evacuated. The two distillation furnaces, the chamber bakeout heaters, the surge tank heater, and EM pump were turned on to bring all temperatures to a minimum of 200°C (400°F). The loop was evacuated and communication to the loop via both the blowdown line and the surge tank gas pressurization line was verified. The fuel specimen test section was then heated to 1025°C (1875°F), and the corrosion section was heated to 675°C (1250°F) for 125 hours. The distillation period was increased from 8 hours, used for the 2500-hour shutdown, to 125 hours to insure as complete a lithium removal as practical from the defect fuel specimen. Once every 24 hours during the distillation period, the loop was pressurized through the blowdown line and condensed lithium drained through the surge tank drain line.

At the completion of the 125-hour distillation, the loop was pressurized with argon to 35×10^4 newtons/m^2 (50 psig) and the vacuum chamber monitored with the mass spectrometer to assure that no small leaks had developed in the loop during the 7500-hour test. No indications were detected while the loop was hot or after cooling all components to room temperature.

The bell jar and distillation furnaces were then removed, and all electrical, gas, and liquid metal lines were disconnected from the spool piece.

B. DISASSEMBLY OF THE LOOP

After completion of the above operations, the loop was moved from the test area to the weld laboratory. The loop and spool piece were then mounted in the motor-driven, rotary weld fixture, installed in the

eight-foot-diameter, weld chamber and the weld chamber was evacuated and backfilled with helium according to GE-NSP Specification P8AYA13-S1. All test sections were removed by cutting with a tubing cutter as described below. All connections were made with stainless steel Swagelok fittings.

The first cut was made at location (1) in Figure 17. After making a cut, an argon line was attached to one side of the cut, and a gas path through the entire loop circuit was verified. The argon line was removed and a valve and cap attached to both sides of the cut to facilitate subsequent handling and ammonia cleaning.

The heater was then removed by cutting at location (2) in Figure 17. Expandable rubber stoppers were inserted into the fitting on both sides of the cut and sealed. The fuel element specimen section was then removed by cutting at location (3) indicated in Figure 17. Again, expandable rubber stoppers were inserted into both sides of the cut and sealed.

Finally, the tensile and corrosion test sections were removed as a unit by cutting the 2.54 cm (1-inch) line at location (4) in Figure 17. Valves and caps were attached to both sides of the cut to provide a seal and to be used during subsequent ammonia cleaning.

The EM pump and surge tank were not removed from the spool piece since no posttest evaluation was planned for these components.

C. REMOVAL AND CLEANING OF FUEL ELEMENT TEST SPECIMENS

The fuel element test section, sealed in helium atmosphere, was transferred to the four-foot-diameter, vertical weld chamber for ease of handling during final disassembly. The weld chamber was evacuated and backfilled with helium during the disassembly. The specimens were removed by cutting the 2.54 cm (1-inch) OD housing just below the top end fitting as was done for the 2500-hour shutdown. The three T-111-clad fuel specimens and two Mo-TZM spacers were then easily slipped from the housing.

At the time of the fuel section disassembly, there was some concern that the removal of lithium by reacting with liquid ammonia may introduce hydrogen in the T-111 and cause embrittlement; therefore, special handling and cleaning procedures were devised. This concern has since been proven to be unwarranted. The three specimens were placed in weighed, sealed, glass, screw-cap bottles and removed from the weld chamber for weighing. Immediately after weighing, the specimens were returned to the weld chamber. Specimen LT-2 was then removed from the sealed bottle and cleaned in two consecutive ethanol rinses and dried. The ethanol rinses and a control were analyzed for lithium by photometry; no lithium was detected in either the rinses or the control sample. The limit of detection is 0.1 μg/ml; approximately 50 ml of ethanol was used for each rinse. Specimen LT-5 was then rinsed in liquid ammonia at -50°C;

no evidence of reaction with lithium was observed. This specimen was finally rinsed in ethanol and allowed to dry. These two samples (LT-2 and LT-5) were then removed from the weld chamber and, for the first time since the shutdown, exposed to air. Because of the built-in clad defect, specimen LT-6 was never exposed to air prior to shipment to the NASA for further evaluation.

D. FINAL CLEANING AND DISASSEMBLY OF THE TENSILE AND CORROSION TEST SECTIONS

The L-shaped tensile-corrosion subassembly was cleaned by flushing with liquid ammonia at -50°C as shown schematically in Figure 18. The test section was flushed for one hour; then it was allowed to soak in nonflowing liquid ammonia for two hours to permit penetration of the ammonia between all of the tight-fitting specimens and housing. The subassembly was again flushed with liquid ammonia for fifteen minutes, blown clear with argon, flushed with alcohol, and then finally rinsed with deionized water.

The heater section of the loop was cleaned in a similar manner except that no soaking was used. Since no evaluation was scheduled for the heater, no further work was performed on it.

The tensile specimen coupons were removed from their respective holders by cutting at the welds joining the three specimen holders and simply sliding the coupons from the holders. No further cleaning of the specimens was necessary.

The corrosion specimen test section was disassembled by first removing the 7.5 cm (3-inch) OD jacket by cutting with a tubing cutter near the weld at the top of the jacket. Next, the 2.54 cm (1-inch) specimen containment tube was cut with a tubing cutter, also at the top. The specimens were then slipped from the tube by gently tapping on the OD with a wood mallet. Some of the specimens were bonded to each other; however, they were separated also by tapping with a mallet. The center rod and swirler wire were easily slipped from the ID of the specimens. No further cleaning of the specimens was necessary.

POSTTEST EVALUATION

A. FUEL ELEMENT TEST SPECIMENS

1. Visual Examination

Following removal of the T-111-clad fuel element test specimens from the specimen housing and final cleaning, a visual examination was performed. The three fuel specimens appeared bright and shiny with no evidence of any discoloration or surface reactions.

2. Weight Measurement

As discussed previously, the three fuel element test specimens exposed during the final 5000 hours of testing were initially placed in weighed, sealed, glass, screw-cap bottles and removed from the weld chamber for weighing prior to cleaning. Following this initial weighing, the specimens were returned to the weld chamber and specimens LT-2 and LT-5 subsequently cleaned as described in an earlier section. Following cleaning, specimens LT-2 and LT-5 were then removed from the weld chamber and, for the first time since the shutdown, exposed to air. Because of the built-in clad defect, specimen LT-6 was never exposed to air. Specimens LT-2 and LT-5 were then reweighed. The difference between these final weights and the weights in the sealed helium-filled bottles was on the order of 0.001 gram and is probably due to taring and related problems rather than any real weight change.

A summary of the pretest and final weights of the fuel specimens obtained after both the 2500-hour and 7500-hour operation periods is given in Table 6. In all cases, the weights represent an average of three consecutive measurements with a maximum spread of $\pm$ 0.0003 gram. The balance calibration and zero point are checked at the beginning and end of each weighing period. Also, an untested specimen is weighed at the same time as the pre- and posttest measurements; these values indicate a reproducibility of $\pm$ 0.0005 gram in comparing pre- and posttest data.

The weight change data for the sound fuel element specimens, LT-1, LT-2, LT-3, and LT-4, indicate most of the rather small weight losses noted occurred during the first 2500 hours of testing. Specimen LT-2, which was in the test section during the entire 7500 hours of testing, exhibited approximately 80 percent of its weight loss in the first 2500 hours of testing. As indicated in Table 6, the defect clad specimen, LT-6, had an indicated weight loss of 0.0043 gram during the 5000 hours of exposure based on the difference between the before-test weight of the specimen and the posttest weight obtained using a tared containment bottle. Subsequent weight determinations at NASA-Lewis on the specimen alone following removal from the bottle indicated a weight loss of 0.0133 gram. The latter weight loss is considered to be more accurate because of errors introduced in the weighing of the specimen and bottle together.

3. Radiographic Examination

Following completion of the initial 2500 hours of testing and completion of the final 5000 hours of testing, the specimens were also radiographed (LT-6 was contained in a helium-filled, sealed glass bottle during radiography) to verify the integrity of the tungsten liner and UN fuel pellets. No signs of degradation or defects were observed due to the test exposures.

This completed GE-NSP posttest evaluation of the fuel element specimens. They were then packed for shipment to NASA-Lewis for a more thorough examination. The defect element (LT-6) was packed in doubly contained, padded, helium-filled, sealed glass bottles for shipment to prevent exposure to air.

Further posttest evaluation was performed by Gordon K. Watson of NASA-Lewis. Results of this evaluation are reported in references 2 and 3.

B. Mo-TZM FUEL ELEMENT SPACERS

1. Visual Examination

Metallographic evaluation of the three Mo-TZM spacers, located between the fuel specimens, has been completed. All three exposed specimens and one unexposed control specimen were examined. Specimen LT-1A was exposed for a total of 7500 hours which consisted of 2500- and 5000-hour consecutive test periods with a change of fuel element test specimens at the end of the initial 2500-hour test period. Specimen LT-2A was tested only during the initial 2500 hours. Specimen LT-3A was tested only during the final 5000 hours.

Posttest visual examination of the spacers following the initial 2500 hours of testing indicated that discoloration of the Mo-TZM spacers had occurred. The spacers were grey in color. Visual examination of the spacers following the final 5000 hours of testing indicated the presence of the same grey discoloration.

2. Weight Measurements

The pre- and posttest weights of the Mo-TZM spacers following both the 2500-hour and 7500-hour operation periods are given in Table 7. Note that all three spacers exhibited small weight gains and that the rate of weight gain was highest during the first 2500 hours of testing.

The weight gain for the 5000-hour specimen is essentially the same as the 7500-hour specimen indicating that a protective surface film may be forming during the lithium exposure.

3. Chemical Analysis

Chemical analyses were performed on the Mo-TZM spacers located between the fuel element test specimens during the 1040°C (1900°F) Pumped Lithium Loop Test. The results are presented in Table 8. Of significance was a slight increase in nitrogen content of all three specimens indicating the discoloration of the surface may be the result of the formation of a thin nitride surface film. This film will also be discussed in the metallographic examination which is to follow.

4. Metallographic Examination

Typical microstructures of the exposed and unexposed specimens are shown in Figures 19 through 22. As seen in Figure 19, the pretest specimen has a cold-worked structure. The 5000- and 7500-hour specimens Figures 21 and 22 both exhibit completely recrystallized microstructures. Except for some surface recrystallization, the 2500-hour specimen, Figure 20, still shows a cold-worked microstructure. The surface recrystallization of the 2500-hour specimen is probably strain induced from residual machining stresses. Some directionality of the grains can still be seen in the 5000- and 7500-hour specimens even though they are completely recrystallized.

The other significant observation in the metallographic examination is the presence of a thin, adherent, semicontinuous, nonmetallic-appearing coating on all three exposed specimens as shown in Figures 20, 21 and 22. This coating appeared on all surfaces except the ID. During testing all surfaces except the ID were in contact with 1.6 m/sec (5 ft/sec) lithium; the ID was in direct contact with the T-111 clad of the fuel specimen. The thickness of the coating was essentially the same for all three specimens 0.0013 - 0.0020 cm (0.0005 - 0.008 inch) regardless of the exposure times.

C. TENSILE SPECIMENS

The top horizontal leg of the loop was designed to contain tensile coupon specimens of three advanced refractory alloys: ASTAR 811C, ASTAR 811CN, and W-Re-Mo Alloy 256 for exposure to flowing lithium with a velocity of approximately 3.3 m/sec (10 ft/sec). Identical control specimens to those in contact with lithium were also attached to the outer surface (vacuum) of the test section to separate the thermal effects of the alkali metal exposure since they saw the same thermal history as the inside coupons but were not exposed to lithium. Following completion of the 7500-hour test the tensile test section was removed from the loop, cleaned, and the specimens removed from the test section as described previously. Evaluation of the test specimens included visual examination, weight change measurements, tensile testing, chemical analysis, Metallographic examination, and X-ray diffraction analysis.

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* All specimens were nickel plated prior to metallographic preparation to minimize rounding of specimen edges during polishing.

1. Visual Examination

A slight uniform discoloration of the ASTAR 811C and W-Re-Mo Alloy 256 tensile coupons exposed to lithium was noted. There was no discoloration of ASTAR 811CN coupons exposed to lithium or of the ASTAR 811C, ASTAR 811CN, or W-Re-Mo Alloy 256 coupons exposed to vacuum. Examinations of all specimen coupons at 30X revealed no cracking or surface reactions, other than the discoloration described.

2. Weight Change Measurement

The tensile specimen coupons were weighed to the nearest 0.0001 gram immediately after cleaning and their weights compared with pretest weights. The results of these weighings are shown in Table 9. The weights shown are an average of three individual determinations made on each specimen. It can be seen in Table 9 that both ASTAR 811C specimen coupons exposed to lithium and all four W-Re-Mo Alloy 256 specimen coupons exposed to lithium showed a weight increase following 7500 hours of testing. The ASTAR 811CN specimens exposed to lithium showed a small weight decrease. All specimens exposed to vacuum at the same temperature as the specimens exposed to lithium for the 7500-hour test period showed very small weight decreases.

The weight increases for the ASTAR 811C and the W-Re-Mo Alloy 256 specimens exposed to lithium are consistent with visual examination since these specimens showed a slight uniform discoloration. Chemical and metallographic were used to characterize these surface films.

3. Tensile Testing

Tensile specimens were machined from the coupons exposed to lithium and vacuum within the loop and from pretest unexposed coupons. The tensile specimens were machined at Metcut Research Associates, Inc., Cincinnati, Ohio, to the dimensions given in Figure 23.

All tensile coupon specimens were in the fully recrystallized condition prior to exposure to the loop test conditions. Those specimens in the pretest recrystallized condition were given a simulated postweld anneal of 1 hour at 1315°C (2400°F). The posttest specimens had been annealed for 1 hour at 1315°C (2400°F) during the postweld annealing of loop components during fabrication, in addition to being exposed to lithium at 1040°C (1900°F) for 7500 hours. This anneal gave the pretest control specimens the same thermal history as the posttest specimens prior to their exposure to test conditions. This approach facilitated a determination of the effect of the lithium and vacuum exposure during the loop test on the mechanical properties of the alloys. The results of the room temperature tensile tests performed to determine the effects of the lithium exposure and the vacuum exposure on the ultimate tensile strength, yield strength, and ductility of the tested alloy specimens are shown in Table 10.

The ultimate tensile and yield strengths of the ASTAR 811C alloy in the posttest condition showed a definite decrease from their pretest values. For example, the decreases noted in the 0.2-percent yield strength were 15 percent for the lithium exposed specimen and 10 percent for the vacuum exposed specimen. The ultimate and yield strengths of the vacuum exposed ASTAR 811C alloy are slightly higher than the values obtained on the specimens exposed to lithium indicating possible effects of lithium exposure on the alloy. It should also be noted that the ASTAR 811C alloy in the lithium exposed posttest condition showed a slight discoloration and weight gain during the test. There was little change noted in the ductility of the ASTAR 811C alloy specimens as a result of the lithium and vacuum exposures.

The results of the room temperature tensile tests on the ASTAR 811CN specimens were similar to those obtained on the ASTAR 811C specimens described above, however, the decreases in ultimate and yield strengths as a result of lithium and vacuum exposures were greater. For example, the decreases noted in the 0.2-percent yield strength were approximately 25 percent for both the lithium and vacuum exposure specimens. These results for the 811CN specimen are in reasonable agreement with the 33-percent decrease in 0.2-percent yield strength (96.0 ksi to 64.4 ksi) noted for 811CN tensile specimens exposed to lithium for 3500 hours at approximately 1135°C (2075°F) in the Valve Test Loop (ref 6). It is of interest to note that the 811CN alloy exhibited approximately 10 percent more tensile elongation that the 811C alloy in all conditions. It may be concluded from the results on both the 811C and 811CN specimens that the changes noted in the room temperature tensile properties are primarily the result of the thermal treatment and not the lithium exposure.

The ultimate tensile and yield strengths of the W-Re-Mo Alloy 256 specimens in the posttest condition showed a definite decrease from their pretest values. Unlike the ASTAR alloys, the W-Re-Mo Alloy 256 showed no change in ultimate tensile strength between specimens in the lithium exposed and vacuum exposed posttest conditions. The yield strengths of the vacuum exposed W-Re-Mo Alloy 256 specimens are slightly higher than those of the lithium exposed specimens. There was a slight reduction in ductility of the W-Re-Mo Alloy 256 specimens in the posttest conditions as compared with pretest values although there was no difference in ductility values between the two posttest conditions. Coupling the changes in ultimate tensile strength, ductility, and slight changes in yield strength of W-Re-Mo Alloy 256 specimens in the two separate posttest conditions with the results of pretest values, it is felt at this time that changes are associated with thermal conditions rather than lithium exposure.

4. Chemical Analysis

Samples for chemical analysis were removed from the ends of the tensile specimen coupons and analyzed for oxygen, nitrogen, and hydrogen by the vacuum fusion technique and for carbon by the combustion conducto-

metric technique. Samples were obtained in the pretest annealed,* post-test lithium-exposed,** and posttest vacuum-exposed** conditions. The results of these analyses are presented in Table 11 along with chemical analyses of pretest recrystallized material. In general, little change in oxygen, nitrogen, hydrogen, or carbon concentration was observed as a result of the one hour at 1316°C (2400°F). A slight increase in the nitrogen concentration of ASTAR 811CN (107 to 130 ppm) and decrease in the oxygen concentration of the W-Re-Mo alloy (89 to 40 ppm) was noted. As may be seen in Table 11, no differences in hydrogen concentration were noted in any of the test specimens.

The changes in the carbon, nitrogen, and oxygen concentrations of the specimens as a result of the 7500-hour exposure at 1040°C (1900°F) to flowing lithium and the vacuum environment are given in Table 12. The marked difference in behavior between the ASTAR 811C and 811CN is somewhat surprising considering the similar base compositions of the two alloys.***

The long-term exposure to lithium resulted in significant increases in the carbon concentration in 811C and decreases in the carbon, nitrogen, and oxygen concentrations of the 811CN specimen. The loss in carbon and nitrogen noted in the 811CN specimen in this test is in agreement with the losses noted on 811CN specimens exposed to flowing lithium at 1120°C (2050°F) in the Valve Test Loop, (ref 6). The W-Re-Mo Alloy 256 specimen showed increases in carbon and nitrogen concentrations as a result of exposure to lithium, which is also in agreement with increases in these elements noted in specimens in the referenced test, (ref 6).

The changes noted in the chemistry of the test specimens as a result of the long-term exposure to the 10^{-7} N/m^2 (10^{-9} torr) vacuum environment at 1040°C (1900°F) are also given in Table 12. The changes noted for the 811C and 811CN specimens were similar to those noted as a result of exposure to lithium, i.e., gains in carbon and nitrogen concentration of the 811C specimen and losses in these elements in the 811CN specimen.

5. Metallographic Examination

Metallographic examination of the tensile specimens after exposure to the lithium revealed no evidence of corrosion. Examination of those specimens exposed to the high-temperature vacuum environment revealed no

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* Pretest material given a simulated postweld anneal of 1315°C (2400°F)

** Posttest material given one-hour anneal of 1315°C (2400°F) during postweld anneal of test loop.

*** ASTAR 811C Specimens: Ta-7.3W-0.98Re-0.86Hf.
ASTAR 811CN Specimens: Ta-6.5W-1.03Re-1Hf.

evidence of surface reactions. Representative photomicrographs of each specimen are shown in Figures 24 through 26.

Examination of the ASTAR 811C specimens exposed to lithium and vacuum revealed slight grain growth when compared to the unexposed specimens. No grain growth was observed in the exposed W-Re-Mo Alloy 256 specimens.

The ASTAR 811CN alloy specimens contained in the loop were inadvertently exposed in the as-rolled condition rather than the recrystallized condition. Following exposure to the lithium and vacuum at 1040°C (1900°F), the ASTAR 811CN alloy specimens were found to be partially recrystallized. Figure 25 shows the pretest and posttest ASTAR 811CN microstructures.

During room temperature tensile testing, the exposed ASTAR 811CN alloy specimens exhibited longitudinal cracking extending from the fracture surface. Examples of this type of cracking are shown in Figure 27. It is believed that the cracking is associated with induced stresses introduced during the rolling operation. Since the sheet specimens were in the as-rolled condition prior to exposure, it is probable that the residual stress remained in the partially recrystallized specimens after the 5000-hour exposure at 1040°C (1900°F) to contribute to this type of fracture. As necking occurs the stress is built in the region of maximum strain near the surface of the specimen and is relieved during fracture by propagation of the longitudinal crack.

Additional metallographic examination of the lithium-exposed ASTAR 811C alloy test specimen revealed the presence of the surface film. This film is believed to be Ta_2C, detected by X-ray diffraction of the specimen surface. No surface film or reaction areas were observed on the lithium-exposed W-Re-Mo Alloy 256 specimens. As indicated earlier, X-ray diffraction of the surface of this alloy showed the presence of the complex compound $(Mo-W)_2C$.

6. X-ray Diffraction Analysis

X-ray diffraction analysis on tensile specimens exposed to lithium within the loop indicated the presence of the complex compound $(Mo-W)_2C$ on the surface of the W-Re-Mo Alloy 256 and single-phase Ta_2C on the surface of the ASTAR 811C alloy. The presence of the carbides is in good agreement with the previously reported weight gains and discolorations noted on these alloys. Chemical analyses of these two alloys also showed an increase in carbon interstitial concentration. There was no second phase detected by X-ray diffraction analysis of the ASTAR 811CN alloy.

D. CORROSION SPECIMENS

As described previously a vertical section of the loop was designed to contain tubular corrosion specimens of the three advanced tantalum refractory alloys: T-111, ASTAR 811C and ASTAR 811CN for posttest

corrosion evaluation in a 1-inch diameter container tube. The corrosion specimens were exposed to flowing lithium with a velocity of approximately 3.3 m/sec (10 ft/sec). The flow was directed through the annulus formed by the corrosion specimens and the center rod. The OD surfaces were in contact with essentially stagnant lithium during the test. Evaluation of the corrosion test specimens included visual examination, weight change measurements, chemical analysis, metallographic examination and X-ray diffraction analysis.

1. Visual Examination

The appearance of the corrosion specimen test section specimens before test and following test revealed that the ASTAR 811C and 811CN specimens had a definite gold-colored surface possibly due to the formation of a thin nitride film. No discoloration of the T-111 specimens was noted.

2. Weight Change Measurement

Table 13 lists the weight changes for the corrosion test specimens arranged in the order of the specimens position in the test section. All specimens showed significant weight gains with the exception of the T-111 specimens. Not all the specimens were of the same length and, therefore, the weight change per unit of ID surface area which is given in Table 13 is the best measure of weight change. The weight gains per unit of ID area were slightly larger in the lower temperature regions of the test section near the lithium exit. It is surprising that the 811C specimens showed significantly larger weight gains than the 811CN specimens when one considers the similarity in the base composition of the two alloys. The T-111 specimen from the top of the test section showed a very slight weight loss while the specimen from near the lithium exit exhibited a very small weight gain.

3. Chemical Analysis

Samples for chemical analysis were removed from the corrosion test specimens and analyzed for oxygen, nitrogen, and hydrogen by the vacuum fusion technique and for carbon by the combustion conductometric technique. Samples were obtained in the pretest annealed and posttest lithium exposed conditions. The results of these analyses are presented in Table 14.

In general the ASTAR 811CN specimens showed an increase in carbon concentration in the cooler portions of the specimen housing and increases in nitrogen and oxygen concentrations. ASTAR 811C alloy specimens showed a somewhat different behavior with regard to carbon concentration, when compared with the ASTAR 811CN alloy specimens

The carbon concentration of the ASTAR 811C alloys decreased while there was an increase in nitrogen and oxygen concentrations. The T-111

alloy specimens showed an increase in carbon and oxygen concentrations but little change in nitrogen concentration.

If the chemical analyses of the ASTAR 811C and ASTAR 811CN alloy tensile specimens are compared with the chemical analyses of the ASTAR 811C and ASTAR 811CN alloy corrosion specimens indication of interatitial movement is noted. The carbon concentration of the ASTAR 811C migrates from the cooler area of the loop to the hotter area while the carbon concentration of the ASTAR 811CN migrates from the hotter area of the loop to the cooler area.

4. Metallographic Examination

Samples were removed from the corrosion specimens for metallographic examination. Metallographic examination of the corrosion specimens revealed no evidence of corrosion. Representative photomicrographs of each specimen are shown in Figures 28 through 30. There was no evidence of a built-up surface reaction on either the ID or OD of the specimens exposed to flowing or stagnant lithium respectively.

5. X-ray Diffraction Analysis

Corrosion specimen rings of lithium exposed T-111, ASTAR 811C, and ASTAR 811CN alloys were sectioned and flattened so that on X-ray diffraction analysis could be performed on the inside diameter surface of the specimens. There was no second phase detected by the use of X-ray diffraction analysis on the surface of the T-111 alloy specimen. Single-phase HFC was detected on the surface of the ASTAR 811C and ASTAR 811CN alloy specimens. Chemical analysis of the ASTAR 811CN alloy specimens showed an increase in carbon interstitial concentration. It should be noted, however, that chemical analysis of the ASTAR 811C alloy specimens showed a decrease in the carbon interstitial concentration, indicating a loss of the carbide strengthening element.

E. LOOP COMPONENTS

1. Visual Examination

The major loop components (fuel element test section, tensile specimen coupon holder and corrosion specimen test section) were sectioned, cleaned, and visually examined. Visual examination of these sections, revealed nothing of an unusual nature. Following removal of the heat rejection jacket from the corrosion specimen test section a reaction zone was observed in the area of the lithium-argon interface region discussed previously. This reaction zone was evident by a difference in the surface appearance of the tube.

2. Chemical Analysis

Chemical analyses (C, O, N, H) were performed on specimens cut from

the fuel specimen housing and corrosion specimen housing portions of the loop. Samples were selected near the inlet portions of the fuel specimen and corrosion specimen housings and from the liquid-gas interface region of the corrosion specimen housing. Both of these components were fabricated from T-111 alloy tubing. The analyses of these components after the 7500 hour exposure to flowing lithium is compared with the pretest unexposed analyses in Table 15. Generally changes in interstitial concentrations were slight and their significance is difficult to determine. The only large increase was the interstitial oxygen concentration of the corrosion housing tubing at the location of the lithium-argon interface. This increase is not surprising due to the presence of the argon gas pocket for 5000 hours. Generally speaking, however, chemical concentration changes of the T-111 alloy tubing were slight and no detrimental effects due to the lithium exposure were observed.

3. Metallographic Examination

Sections were removed from the fuel element and corrosion specimen housings for metallographic examination. Sections were removed from the 1.0" OD T-111 Alloy fuel element housing tubing in the lithium inlet and lithium exit regions. No evidence of corrosion was observed, however, the amount of second phase precipitate observed was greater in the lithium inlet section. Representative photomicrographs are shown in Figure 31.

Sections were removed from the lithium inlet and lithium-argon interface regions of the 1.0" OD T-111 Alloy corrosion specimen housing. Again no corrosion was observed but massive second phase precipitation was present. Representative photomicrographs from the two regions examined are presented in Figures 32 and 33. The second phase precipitate was present near the surface of both the ID and OD of the tubing, as shown in Figure 33, which were both essentially exposed to stagnant lithium. As shown in Figure 34 there was a surface reaction zone present which was observable in the as polished condition but was lost following etching and identification was not possible.

4. Bend Testing

In addition to the planned posttest evaluation of the 1040°C (1900°F) pumped lithium loop, a number of T-111 Alloy tubing segments were removed from the fuel element and corrosion specimen housings and flattened to qualitatively measure their ductility.

Initially, the specimens were prepared by cutting approximately 1.2 cm (0.5-inch) rings from the containment tubing with a tubing cutter. The specimens were then ground flat on metallographic polishing paper through 600-grit silicon carbide using water as the lubricant. Generally about .1 cm (0.05 inch) was removed during this operation. The fine-polished flat surface was desirable for observation of crack initiation and propagation. The I.D. edges were lightly filed with a jeweler's file to smooth the burr from the cutting operation.

Virtually all samples prepared in this manner cracked in an extremely brittle manner after negligible deformation (less than .05 cm, 0.020-inch). To eliminate this brittle behavior two operations proved successful. Specimens prepared by wet polishing were given a one hour anneal at 1040°C (1900°F) in vacuum and subsequently flattened. The 1040°C (1900°F) anneal restored ductility to the specimens. Secondly, it was found that specimens would deform in a ductile manner if the cut surfaces were hand filed with a "bastard", half round, cross cut file rather than polished. Representative photographs of bent fuel element housing rings are shown in Figure 35. A bent corrosion specimen housing ring is shown in Figure 36. To fully understand this brittle-ductile behavior pattern of T-111 Alloy tubing material additional evaluation needs to be performed.

The results of a study of this behavior conducted at Lewis Research Center (ref 7) has attributed this behavior to hydrogen embrittlement susceptability resulting from a grain boundary sensitivity existing in T-111 after aging.

SUMMARY OF RESULTS

The purpose of this study was to evaluate the possible corrosion effects occurring in a flowing lithium loop containing the refractory metals and nuclear fuel materials that might be present in a lithium-cooled, space power reactor loop operating at 1040°C (1900°F).

Based upon the results of 7500 hours of loop operation, the following summarizes the principle results and the conclusions drawn.

(1) There was no significant corrosion effects on or caused by the T-111-clad UN fuel specimens in the lithium loop. This is true for both the sound fuel element specimens and the defected fuel element specimens where UN was exposed to the flowing lithium stream.

(2) There was a loss in the tensile strength of the tantalum and tungsten alloy specimens which were exposed to the high-temperature loop conditions. Based on the comparison of room temperature tensile properties of materials exposed to vacuum and lithium, it must be concluded that the loss in strength is from thermal effects rather than the lithium exposure. Future work in this area would require that high-temperature creep testing be done on exposed materials to determine the full extent of the effect of high temperature lithium and vacuum exposure on the long-term performance of the materials.

(3) The evaluation of the 7500-hour test loop components revealed no significant corrosion in the system. Based upon these test results, it is concluded that the materials combination tested as representative of a lithium-cooled reactor system should be compatible with the lithium environment.

APPENDIX A

Lithium Capsule Tests of Tantalum and Tungsten Alloy Sheet Specimens

As was discussed elsewhere in the text, the 1040°C (1900°F) Lithium Loop was originally scheduled for 1425°C (2600°F) operation; however, change in NASA goals, etc. revised the operating conditions and objectives of the test. For 1425°C (2600°F) operation, there was some apprehension as to the effects of contamination fabrication on the subsequent lithium exposure of the alloys being considered. The processing history of typical specimens indicated that some contamination of the specimens might occur prior to exposure in the loop, and for this reason, it was deemed advisable to evaluate typical specimens in 1425°C (2600°F) isothermal lithium capsule tests of 100 hours' duration.

The conditions of the various sheet specimens prior to exposure to lithium are listed below:

T-111, as-received.

T-111, as-received plus autoclaving for 3 hours at 1750°C (3180°F) in 6.8×10^7 N/m^2 (10,000 psig) helium plus 3 hours at 1425°C (1600°F) in 1.4×10^6 N/m^2 (200 psig) helium.

W-30Re-30Mo, [a] powder process product sheet, as-fabricated.

W-30Re-30Mo, Powder process product sheet, as-fabricated plus autoclaving for 3 hours at 1750°C (3180°F) in 6.8×10^7 N/m^2 (10,000 psig) helium.

W-30Re-30Mo, arc cast product sheet, as-fabricated.

W-30Re-30Mo, arc cast product sheet, as-fabricated plus autoclaving for 3 hours at 1750°C (3180°F) in 6.8×10^7 N/m^2 (10,000 psig) helium.

Two T-111 alloy capsules, measuring 2.54 cm (1.0-inch) diameter x .25 cm (0.10-inch) wall thickness x 14 cm (5.5-inch) length, were used to test the specimens listed above. The two T-111 alloy sheet specimens were placed in one of these capsules and the four W-30Re-30Mo alloy sheet specimens were placed in the other capsule. Lithium used to partially fill these capsules was purified by hot-gettering with zirconium sheet for 240 hours at 815°C (1500°F). Samples of lithium taken during the filling operation were analyzed and found to contain 12 ppm nitrogen. Both the capsule filling and electron beam welding sealing techniques were performed in a 10^{-3} N/m^2 (10^{-5} torr) vacuum environment.

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[a] Atomic percent.

The filled capsules were wrapped with Cb-1Zr foil and heated for 100 hours at 1425°C (2600°F) in a Brew vacuum furnace at a pressure of less than 1.3×10^{-3} N/m^2 (1×10^{-5} torr). Following completion of the test, the capsules were opened in an inert atmosphere chamber, and the lithium was melted and drained from the capsules. Residual lithium was removed from the tungsten alloy specimens by distillation while the T-111 specimens were cleaned by dissolution of the residual lithium in liquid ammonia. Weight change, chemical analysis, metallographic examination, and microhardness surveys were used in the evaluation of the six test specimens.

The pretest and posttest appearance of the T-111 sheet specimens is illustrated in Figure 37. Chemical analysis and weight change information on the T-111 specimens in given in Table 16. A total of three analyses at two different laboratories were performed for most of the elements listed. The principal changes noted as a result of the 100-hour exposure to the lithium were the significant weight loss and oxygen concentration decrease in the sheet specimen which was autoclaved and heat treated prior to exposure. Approximately 80 percent of the total weight loss noted is attributed to the phenominal decrease in oxygen concentration. Previous investigators have noted substantial leaching of oxygen from unalloyed columbium and tantalum by lithium (ref 8), but never with gettered alloys, such as T-111 containing 2% hafnium, to the extent noted in this experiment. The relative high temperature of this experiment compared to most of the other test results which have been reported is thought to be the major factor responsible for the nearly total deoxidation observed in the current experiment.

The metallographic appearance of cross sections of the T-111 sheet specimen which was heat treated before exposure to lithium in a T-111 capsule for 100 hours at 2600°F is shown in Figure 38. The gross oxygen contamination of the T-111 sheet is readily apparent as evidenced by the abundance of both grain boundary and bulk precipitate, which is assumed to be hafnium oxide particles. Lithium exposure caused a substantial reduction in the amount of oxide precipitate in the structure. The surface regions are particularly void of precipitate suggesting that a slight oxygen gradient still exists in the sheet specimen despite the full-section oxygen concentration of only 24 ppm. Diamond Pyramid Hardness (DPH) surveys were performed on specimen cross sections before and after exposure and indicated no detectable hardness gradient in the specimens. It is interesting to note that the sheet specimen was slightly harder following lithium exposure (and deoxidation) than before test. No corrosion of the specimen surface could be detected.

The chemical analyses of the powder product and arc cast product W-30Re-30Mo (a/o) sheet before and following exposure to lithium for 100 hours at 2600°F are given in Table 17. No significant changes in concentrations of oxygen, nitrogen, hydrogen, or carbon were detected. The GE-NMPO carbon analyses, which are considerably lower than the GE-SPPS carbon numbers, are considered to be the more accurate of the two sets

of values. Unfortunately, there was insufficient sample to obtain post-test GE-NMPO carbon values. The positive weight changes listed are attributed in large measure to surface contamination during the vacuum distillation operation used to remove residual lithium from the specimens. (Dissolution in liquid ammonia has been adopted as the standard cleaning procedure for alkali metal test specimens.)

The metallographic appearance of the autoclaved W-30Re-30Mo sheet specimens before and following exposure to lithium is shown in Figures 39 and 40. Powder product sheet specimens of the alloy are presented in Figure 39 and show the fine-grained structure typical of the alloy prepared by this method. Examination of the surface of the tested specimen revealed no evidence of corrosion. The grain boundary particles evident in the microstructure of the tested specimens is sigma phase which developed as a result of the thermal exposure of the corrosion test. Microstructures of autoclaved and tested sheet specimens prepared from arc cast alloy are illustrated in Figure 40 and again no evidence of corrosion was observed. The grain size of the arc cast product sheet is quite a bit larger than the powder product materials shown in Figure 39 and the structure is considerably cleaner. Again, a considerable amount of sigma phase developed as a result of the thermal treatment during the corrosion test.

The capsule test results described above indicate that T-111 and W-30Re-30Mo (a/o) specimens of the type described above can be placed in the 1425°C (2600°F) Lithium Loop without fear of catastrophic lithium penetration.

APPENDIX B

Flowmeter Calibration

The flowmeter was calibrated by making an energy balance across the lithium heater and plotting this calculated rate as a function of millivolts output at different EM pump power levels.

The flow rate is calculated from the following relationship.

$$W = \frac{(Q_W - Q_{HL})}{(C_p)\ (T_7 - T_4)} \times 3414$$

where W = flow rate (pounds per hour);

Q_W = heater power input (kilowatts);

Q_{HL} = heat losses (estimated)(Kilowatts);

C_P = specific heat of lithium at operating temperature (Btu per pound per °F);

T_7 = heater exit temperature (°F);

T_4 = heater inlet temperature (°F);

3414 = conversion from Btu per hour to kw.

This calculation is repeated for a number of heater power levels, EM pump power levels, and ΔT's to yield a sufficient number of points to obtain the plot shown in Figure 41.

PREVIOUSLY PUBLISHED PROGRESS REPORTS FOR THIS CONTRACT

Quarterly Progress	For Quarter Ending
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
Report No. 8 (NASA-CR-72335)	April 15, 1967
Report No. 9 (NASA-CR-72336)	July 15, 1967
Report No. 10 (NASA-CR-72352)	October 15, 1967
Report No. 11 (NASA-CR-72383)	January 15, 1968
Report No. 12 (NASA-CR-72452)	April 15, 1968
Report No. 13 (NASA-CR-72483)	July 15, 1968
Report No. 14 (NASA-CR-72505)	October 15, 1968
Report No. 15 (NASA-CR-72527)	January 15, 1969
Report No. 16 (NASA-CR-72560)	April 15, 1969
Report No. 17 (NASA-CR-72592)	July 15, 1969
Report No. 18 (NASA-CR-72620)	October 15, 1969
Report No. 19 (NASA-CR-72662)	January 15, 1970
Report No. 20 (NASA-CR-72739)	April 15, 1970
Report No. 21 (NASA-CR-72782)	July 15, 1970
Report No. 22 (NASA-CR-72818)	October 15, 1970
Report No. 23 (NASA-CR-72853)	January 15, 1971
Report No. 24 (NASA-CR-72853	May 13, 1971

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TABLE 1

LITHIUM LOOP DESIGN OPERATING CONDITIONS

Primary Loop Construction Material		T-111
Coolant		Lithium
Flow Rate		105 Kg/hr (236 lbs/hr)
Velocity		
Heater		3 m/sec (10 ft/sec)
Fuel Section		1.5 m/sec (5 ft/sec)
Tensile Test Section		3 m/sec (10 ft/sec)
Corrosion Specimen Section		3 m/sec (10 ft/sec)
Internal Pressure		$3.5/10^5 N/m^2$ (50 psia)
Maximum Temperature		1040°C (1900°F)
Temperature Drop in Heat Rejector		40°C (70°F)
Power Input		5 kw
Test Duration	Phase I	2500 hours
	Phase II	5000 hours
Test Environment (Vacuum)		1.3×10^{-6} N/m^2 (1×10^{-8} torr)

TABLE 2

RESULTS OF CHEMICAL ANALYSIS OF T-111 SPECIMENS FROM 1-HOUR - 2400° F VACUUM ANNEAL OF THE 1040° C (1900° F) LITHIUM LOOP AT STELLITE

Specimen	Chemical Analysis, ppm			
	C	O	N	H
Unexposed[a]	52	14	26	1
Annealed (wrapped in Cb-1Zr foil)	47	15	6	< 1
Annealed (not wrapped)	57	59	9	< 1

(a) MCN 04B-121-01, 0.040-inch-thick sheet.

TABLE 3

LITHIUM ANALYSIS - 1040° C (1900° F) LITHIUM LOOP

	Concentration, ppm		
	From Still Receiver[a]	From Transfer System[b]	From Loop[c]
Oxygen	32,49	25	28
Nitrogen	3,4	2,2	2,3
Carbon	88	86	76
Silver	< 5	< 5	< 5
Aluminum	25	5	5
Boron	< 75	< 75	< 75
Barium	< 75	< 75	< 50
Beryllium	< 5	< 5	< 5
Calcium	50	5	25
Columbium	< 25	< 25	< 25
Cobalt	< 5	< 5	< 5
Chromium	< 5	< 5	< 5
Copper	5	5	5
Iron	5	< 5	5
Magnesium	5	5	5
Manganese	< 5	< 5	< 5
Molybdenum	< 5	< 5	< 5
Sodium	< 125	< 125	--
Nickel	< 5	< 5	< 5
Lead	< 50	< 50	< 50
Silicon	5	5	< 5
Tin	< 25	< 25	< 25
Strontium	25	< 5	50
Titanium	< 25	< 25	< 25
Vanadium	< 25	< 25	< 25
Zirconium	< 25	< 25	< 25

(a) Sample No. 2767 - Lithium from still receiver prior to 1040° C (1900° F) loop fill.

(b) Sample No. 2776 - Lithium from 1900°F loop fill system flush at 260° C (500° F).

(c) Sample No. 2777 - Lithium from 1900°F loop flush at 230° C (450° F).

TABLE 4

LITHIUM ANALYSIS - 1040° C (1900° F) LITHIUM LOOP

	Pretest	Posttest 2500 Hrs
Oxygen	28	149
Nitrogen	2, 3	118
Carbon	76	99
Silver	< 5	< 5
Aluminum	5	25
Boron	< 75	< 75
Barium	< 50	< 75
Beryllium	< 5	< 5
Calcium	25	5
Columbium	< 25	< 25
Cobalt	< 5	< 5
Chromium	< 5	< 5
Copper	5	< 5
Iron	5	< 5
Magnesium	5	< 5
Manganese	< 5	< 5
Molybdenum	< 5	< 5
Sodium	--	< 75
Nickel	< 5	< 5
Lead	< 50	< 50
Silicon	< 5	5
Tin	< 25	< 25
Strontium	50	50
Titanium	< 25	< 25
Vanadium	< 25	< 25
Zirconium	< 25	< 25

TABLE 5

LITHIUM ANALYSIS - 1040° C (1900° F) LITHIUM LOOP

	Pretest	Position 2500 Hrs	Position 7500 Hrs
Oxygen	28	149	55
Nitrogen	2, 3	118	< 1
Carbon	76	99	57
Silver	< 5	< 5	< 5
Aluminum	5	25	< 5
Boron	< 75	< 75	< 75
Barium	< 50	< 75	< 75
Beryllium	< 5	< 5	< 5
Calcium	25	5	5
Columbium	< 25	< 25	< 25
Cobalt	< 5	< 5	< 5
Chromium	< 5	< 5	< 5
Copper	5	< 5	< 5
Iron	5	< 5	5
Magnesium	5	< 5	< 5
Manganese	< 5	< 5	< 5
Molybdenum	< 5	< 5	< 5
Sodium	--	< 75	< 25
Nickel	< 5	< 5	< 5
Lead	< 50	< 50	< 50
Silicon	< 5	5	< 5
Tin	< 25	< 25	< 25
Strontium	50	50	5
Titanium	< 25	< 25	< 25
Vanadium	< 25	< 25	< 25
Zirconium	< 25	< 25	< 25

TABLE 6

WEIGHTS OF T-111 CLAD UN FUEL ELEMENT SPECIMENS BEFORE AND AFTER EXPOSURE TO FLOWING LITHIUM AT 1040° C (1900° F)

Specimen No.	Test Hours	Specimen Weight, grams		
		Before Test	After Test	Change
LT-1	2500	135.9875	135.9853	-0.0022
LT-2	2500	135.2553	135.2524	-0.0029
LT-2[a]	7500	135.2553	135.2516	-0.0037
LT-3	2500	135.5796	135.5769	-0.0027
LT-5	5000	133.5430	133.5427	-0.0003
LT-6	5000	134.3446	134.3403[b] 134.3313[c]	-0.0043 -0.0133

a) Specimen No. LT-2 was removed and weighed after 2500 hours of testing and replaced in test section for the final 5000 hours of loop operation.

b) Tare weight; defect clad specimen posttest weight obtained with sample in sealed helium-filled transfer bottle.

c) Posttest weight of defect clad specimen obtained at NASA-Lewis Research Center following removal from transfer bottle.

TABLE 7

WEIGHTS OF Mo-TZM SPACERS LOCATED BETWEEN FUEL ELEMENT TEST SPECIMENS DURING 1040° C (1900° F) LITHIUM LOOP TEST

Specimen No.	Test Hours	Specimen Weight, grams		
		Before Test	After Test	Change
LT-1A	2500	8.7253	8.7289	+ 0.0036
LT-1A[a)]	7500	8.7253	8.7323	+ 0.0070
LT-2A	2500	8.6406	8.6446	+ 0.0040
LT-3A	5000	8.6177	8.6245	+ 0.0068

a) Spacer LT-1A was removed and weighed after 2500 hours of testing and replaced in test section for the final 5000 hours of loop operation.

TABLE 8

CHEMICAL ANALYSES OF Mo-TZM SPACERS LOCATED BETWEEN FUEL ELEMENT TEST SPECIMENS DURING 1040° C (1900° F) PUMPED LITHIUM LOOP TEST

Specimen No.	Test Hours	Chemical Analysis, ppm*			
		C	O	H	N
LT-1A	7500	213	17	< 1	14
LT-2A	2500	216	18	< 1	8
LT-3A	5000	212	6	< 1	15
LT-4A	Untested	217	22	< 1	2

*Average of Duplicate Determinations.

TABLE 9

SPECIMEN COUPON WEIGHT MEASUREMENTS BEFORE AND AFTER 7500-HOUR EXPOSURE TO LITHIUM IN THE 1040° C (1900° F) PUMPED LITHIUM LOOP

	Specimen No.	Material	Weight Before Test (g)	Weight After Test (g)	Weight Change mg	Weight Change mg/cm^2
Test Section Entrance 1040° C (1900° F)			Specimens Exposed to Lithium			
	4A	ASTAR 811C	15.0123	15.0182	+5.9	+0.3252
	4B	ASTAR 811C	14.7590	14.7650	+6.0	+0.3307
	5A	ASTAR 811CN	14.9751	14.9742	-0.9	-0.0496
	5B	ASTAR 811CN	15.0736	15.0720	-1.6	-0.0882
Lithium Flow	6A	W-Re-Mo Alloy 256	7.0170	7.0200	+3.0	+0.1653
	6B	W-Re-Mo Alloy 256	6.9246	6.9277	+3.1	+0.1708
	6C	W-Re-Mo Alloy 256	7.1674	7.1699	+2.5	+0.1378
	6D	W-Re-Mo Alloy 256	7.1639	7.1666	+2.7	+0.1488
Test Section Exit 1030° C (1890° F)			Specimens Exposed to Vacuum			
Vacuum	7A	ASTAR 811C	15.0204	15.0202	-0.2	-0.0124
	7B	ASTAR 811C	15.1010	15.1008	-0.2	-0.0124
	8A	ASTAR 811CN	13.5103	13.5100	-0.3	-0.0186
	8B	ASTAR 811CN	13.9453	13.9448	-0.5	-0.0310
	9A	W-Re-Mo Alloy 256	6.3122	6.3119	-0.3	-0.0186
	9B	W-Re-Mo Alloy 256	6.1645	6.1640	-0.5	-0.0310

TABLE 10

RESULTS OF ROOM TEMPERATURE TENSILE TESTS FOR 1040° C (1900° F) PUMPED LITHIUM LOOP

Specimen No.	Material	U.T.S.(ksi)	0.02% Y.S.(ksi)	0.2% Y.S.(ksi)	Elongation (%)
	Pretest[a]				
1A	ASTAR 811C	95.9	72.3	76.5	20.0
2A	ASTAR 811CN	107.9	97.3	99.4	28.0
2B	ASTAR 811CN	107.3	94.8	100.3	30.0
3A	W-Re-Mo Alloy 256	154.8	137.5	140.2	13.0
3B	W-Re-Mo Alloy 256	154.0	138.8	140.3	11.8
	Posttest[b] **- Exposed to Lithium at 1040° C (1900° F)**				
4A	ASTAR 811C	83.1	59.4	64.6	18.8
4B	ASTAR 811C	85.3	61.4	65.2	20.0
5A	ASTAR 811CN	90.3	73.8	74.4	28.0
5B	ASTAR 811CN	90.3	73.6	73.8	30.4
6A	W-Re-Mo Alloy 256	142.9	126.1	129.5	10.4
6B	W-Re-Mo Alloy 256	143.7	127.9	132.4	7.0
6C	W-Re-Mo Alloy 256	145.4	127.1	133.2	10.0
6D	W-Re-Mo Alloy 256	145.2	121.7	133.9	8.0
	Posttest[b] **- Exposed to Vacuum at 1040° C (1900° F)**				
7A	ASTAR 811C	88.4	64.1	68.6	16.0
7B	ASTAR 811C	87.3	60.7	68.6	15.6
8A	ASTAR 811CN	94.9	74.6	77.6	25.2
8B	ASTAR 811CN	93.8	69.5	74.3	27.8
9A	W-Re-Mo Alloy 256	146.2	132.8	136.0	7.0
9B	W-Re-Mo Alloy 256	144.4	132.1	135.0	10.2

Test Section Entrance
1040° C (1906° F)
Lithium Flow
Test Section Exit
1040° C (1898° F)

(a) Simulated postweld anneal of 1 hour at 1315° C (2400° F).

(b) Annealed 1 hour at 2400°F during postweld anneal of loop.

TABLE 11

RESULTS OF TENSILE SPECIMEN COUPON CHEMICAL ANALYSES FOR 1040° C (1900° F) PUMPED LITHIUM LOOP

Specimen No.	Material		Chemical Analyses, ppm C	N	O	H
	Pretest - Recrystallized					
	ASTAR 811C		232, 270	2, 3	2, 10	1, 1
		Avg.	251	3	6	1
	ASTAR 811CN		152, 174	101, 113	87, 91	< 1, 1
		Avg.	163	107	89	1
	W-Re-Mo Alloy 256		14, 16	< 1, 3	6, 11	1,< 1
		Avg.	15	3	9	1
	Pretest Plus Anneal[a]					
1A	ASTAR 811C		257, 262	7, 9	13, 16	< 1,< 1
		Avg.	260	8	15	< 1
2A	ASTAR 811CN		170, 176	127, 132	34, 46	< 1,< 1
		Avg.	173	130	40	< 1
3A	W-Re-Mo Alloy 256		19, 22	2, 3	14, 24	1,< 1
		Avg.	21	3	19	1
	Posttest[b] - Exposed to Lithium at 1040° C (1900° F)					
4A	ASTAR 811C		287, 291	11, 12	10, 14	< 1,< 1
		Avg.	289	12	12	< 1
5A	ASTAR 811CN		144, 153	81, 83	19, 20	< 1,< 1
		Avg.	149	82	20	< 1
6A	W-Re-Mo Alloy 256		29, 43	3, 4	31, 35	< 1,< 1
		Avg.	36	4	33	< 1
	Posttest[b] - Exposed to 10^{-9} Torr Vacuum at 1040° C (1900° F)					
7A	ASTAR 811C		277, 281	10, 11	23, 24	< 1,< 1
		Avg.	279	11	24	< 1
8A	ASTAR 811CN		149, 158	119, 124	31, 33	< 1,< 1
		Avg.	154	122	32	< 1
9A	W-Re-Mo Alloy 256		19, 22	1, 1	19, 30	< 1,< 1
		Avg.	21	1	25	< 1

(a) Simulated postweld anneal at 1 hour at 1315° C (2400° F).

(b) Annealed 1 hour at 1315° C (2400° F) during postweld anneal of loop.

TABLE 12

SUMMARY OF CHEMISTRY CHANGES IN TENSILE SPECIMENS

Material	Changes* in Concentrations of C, N, and O as a Result of: Exposure to Flowing Lithium for 7500 Hours at 1040° C (1900° F) ppm C	N	O	Exposure to 10^{-9} Torr Vacuum Environment for 7500 Hours at 1040° C (1900° F) ppm C	N	O
ASTAR 811C	+ 29	+ 4	- 3	+ 19	+ 3	+ 9
ASTAR 811CN	- 24	- 48	- 20	- 19	- 8	- 8
W-Re-Mo Alloy 256	+ 15	+ 1	+ 14	0	- 2	+ 6

* Changes obtained by comparing specimens exposed for 7500 hours with the control specimens (pretest plus anneal).

TABLE 13

WEIGHTS OF CORROSION TEST SPECIMENS FROM 7500-HOUR
1040° C (1900° F) PUMPED LITHIUM LOOP

	Specimen Identification	Material	Weight, grams		Weight Change	
			Preiest	Postiest	grams	mg/cm²*
Lithium Inlet ~1040° C (1900° F)	11A8	ASTAR 811CN	24.0758	24.0785	+0.0027	+1.2
	11A7	ASTAR 811CN	24.0865	24.0892	+0.0027	+1.2
	11A6	ASTAR 811CN	23.9222	23.9243	+0.0021	+0.96
	11A5	ASTAR 811CN	24.0440	24.0458	+0.0018	+0.83
	11A4	ASTAR 811CN	23.8564	23.8588	+0.0024	+1.1
	11A3	ASTAR 811CN	24.0591	24.0612	+0.0021	+0.96
	11A2	ASTAR 811CN	24.0628	24.0643	+0.0015	+0.69
	11A1	ASTAR 811CN	24.0602	24.0618	+0.0016	+0.73
	10A	ASTAR 811C	193.0870	193.1257	+0.0387	+2.2
	9A	T-111	192.6500	192.6476	-0.0024	-0.13
	11B8	ASTAR 811CN	24.0176	24.0215	+0.0039	+1.8
	11B7	ASTAR 811CN	23.9931	23.9967	+0.0036	+1.6
	11B6	ASTAR 811CN	24.0181	24.0218	+0.0037	+1.7
	11B5	ASTAR 811CN	24.0846	24.0882	+0.0036	+1.6
	11B4	ASTAR 811CN	24.0878	24.0912	+0.0034	+1.6
Lithium Flow	11B3	ASTAR 811CN	24.1080	24.1115	+0.0035	+1.6
	11B2	ASTAR 811CN	23.9448	23.9487	+0.0039	+1.8
	11B1	ASTAR 811CN	24.1180	24.1213	+0.0033	+1.5
	10B	ASTAR 811C	192.8546	192.8987	+0.0441	+2.5
	9B	T-111	191.5730	191.5682	-0.0048	-0.28
	11C8	ASTAR 811CN	23.9946	23.9987	+0.0041	+1.9
	11C7	ASTAR 811CN	24.0642	24.0684	+0.0042	+1.9
	11C6	ASTAR 811CN	23.9796	23.9827	+0.0031	+1.4
	11C5	ASTAR 811CN	24.0153	24.0186	+0.0033	+1.5
	11C4	ASTAR 811CN	24.1136	24.1171	+0.0035	+1.6
	11C3	ASTAR 811CN	24.0268	24.0299	+0.0031	+1.4
	11C2	ASTAR 811CN	24.0431	24.0462	+0.0031	+1.4
	11C1	ASTAR 811CN	23.9224	23.9258	+0.0034	+1.6
Lithium Exit ~1000° C (1830° F)	10C	ASTAR 811C	194.0899	194.1337	+0.0438	+2.5
	9C	T-111	193.2919	193.2924	+0.0005	+0.03

* Only the ID surface which was in contact with the high velocity lithium was used in this calculation.

TABLE 14

RESULTS OF CORROSION SPECIMEN CHEMICAL ANALYSES FOR 1040°C (1900°F) PUMPED LITHIUM LOOP

	Specimen No.	Material		Chemical Analyses, ppm C	N	O	H
	Post Test Material						
Lithium Inlet 1900°F, 1038°C	11A8	ASTAR 811CN		110,110	21,37	75,97	<1
			Avg.	110	29	86	
	10A	ASTAR 811C		160,170	13,17	28,33	<1
			Avg.	165	15	31	
	9A	T-111		80,90	8,9	7,18	<1
			Avg	85	9	13	
	11B8	ASTAR 811CN		140,150	21,28	55,58	<1
			Avg.	145	25	57	
Lithium Flow	10B	ASTAR 811C		160,170	15,22	27,37	<1
			Avg.	165	19	32	
	9B	T-111		67,75	1,6	6,20	<1
			Avg.	71	4	13	
	11C	ASTAR 811CN		150,160	40,41	10,12	<1
			Avg.	155	41	11	
	10C	ASTAR 811C		140,150	9,12	36,53	<1
			Avg.	145	11	45	

TABLE 14

RESULTS OF CORROSION SPECIMEN CHEMICAL ANALYSES FOR 1040°C (1900°F) PUMPED LITHIUM LOOP

	Specimen No.	Material		Chemical Analyses, ppm C	N	O	H
	Post Test Material						
Lithium Exit 1830°F, 999°C	9C	T-111		69,80	14,28	29,44	<1
			Avg.	75	21	37	
	Pre-Test Material						
		ASTAR 811 CN		125,129	15,26	12,21	2,5
			Avg.	127	21	17	4
		ASTAR 811C		232,270	<1	10,1	2,3
				251	<1	6	3
		T-111		13,18	13,13	5,5	10,11
			Avg.	16	13	5	11

TABLE 15

RESULTS OF CHEMICAL ANALYSES OF LOOP
COMPONENTS FROM THE 1900°F PUMPED LITHIUM TEST LOOP

Loop Component	Tube Size OD, Inches	Material	Chemical Analysis, ppm			
			C	N	O	H
Pretest Material	1	T-111	22(1)	<1(1)	26(1)	6(1)
Fuel Housing Li Inlet	1	T-111	29(2)	7,8	31,40	1,<1
		Avg	29	8	36	1
Corrosion Housing Li Inlet	1	T-111	29,32	14,17	39,44	1,2
		Avg	31	16	42	2
Corrosion Housing Li-Ar Interface	1	T-111	22,29	14,22	93,196	1,1
		Avg	26	18	145	1

(1) Average of Two Analysis

(2) Single Analysis

TABLE 16

CHEMICAL ANALYSIS OF T-111 SPECIMENS BEFORE AND FOLLOWING EXPOSURE TO LITHIUM[a] FOR 100 HOURS AT 1425° C (2600° F)

Specimen Description[b]	Concentration, ppm O	N	H	C	Weight Change mg cm^{-2}
T-111 Sheet, As received Before Lithium Test:	35,35[c] (29)	16,15 (5)	1,1 (<1)	110,122 (93)	-0.036
After Lithium Test:	18,11	37,44	1,1	158 (87)	
T-111 Sheet, 3 Hours at 1750° C (3180° F) in 10,000 psig Helium Plus 3 Hours at 1425° C (2600° F) in 200 psig Helium Before Lithium Test:	2877,2164 (2292)	18,15 (19)	20,21		-0.24
After Lithium Test:	24,24 (24)	75,69	2,2	183 (20)	

(a) Container capsule: T-111, nitrogen concentration of lithium before test: 12 ppm

(b) Sheet specimen thickness: 0.35 cm (0.015 in.)

(c) Upper numbers: GE-SPPS analysis; lower number ( ): GE-NMPO analysis

TABLE 17

CHEMICAL ANALYSIS OF W-30Re-30Mo (a/a) SHEET SPECIMENS BEFORE AND FOLLOWING EXPOSURE TO LITHIUM[a] FOR 100 HOURS AT 1425° C (2600° F)

Specimen Description[b]	Concentration, ppm O	N	H	C	Weight Change mg cm^{-2}
Powder Product Sheet, Before Autoclaving:[c]					
Before Lithium Test:	18,12[d]	1,4	1,1	77,4	
After Lithium Test:	26,18	3,1	2,1	25	+ 0.033
Powder Product Sheet, After Autoclaving:					
Before Lithium Test:	18,9	2,1	1,2	46,13	
After Lithium Test:	46,17	5,1	3,1	23	+ 0.07
Arc Cast Sheet, Before Autoclaving:					
Before Lithium Test:	59,11	5,2	4,1	96,19	
After Lithium Test:	52,27	4,4	3,1	82	+ 0.088
Arc Cast Sheet, After Autoclaving:					
Before Lithium Test:	30,26	6,3	2,2	63,11	
After Lithium Test:	43,26	2	2,1	52	+ 0.10

(a) Container capsule: T-111; nitrogen concentration of lithium before test = 12 ppm

(b) Sheet specimen thickness: 0.010 inches

(c) Autoclaving treatment: 3 hours at 1750° C (3180° F) in 10,000 psig helium

(d) First number: GE-SPPS analysis; second number: GE-NMPO analysis

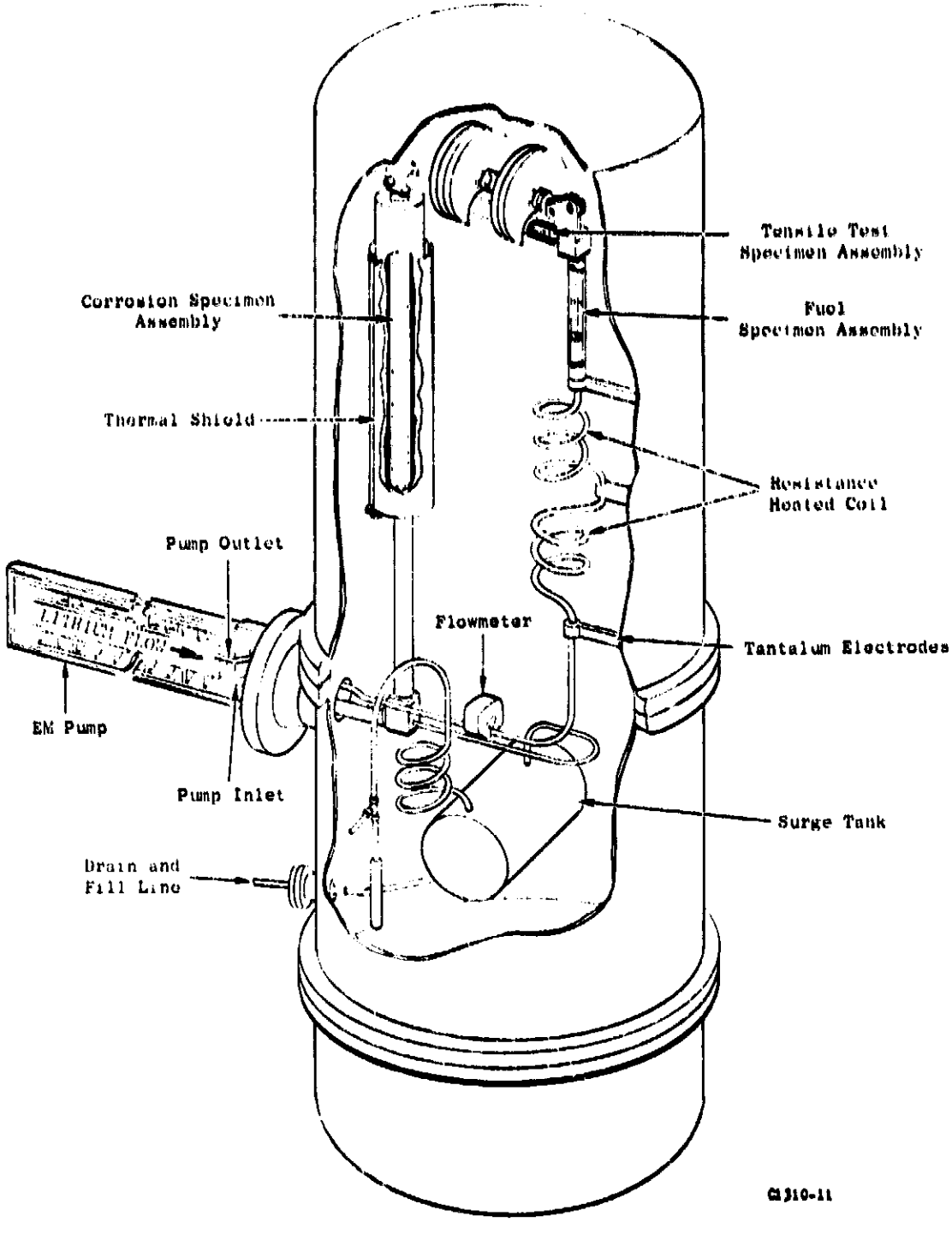


Figure 1. - Isometric drawing of 1040° C (1900° F) lithium loop.

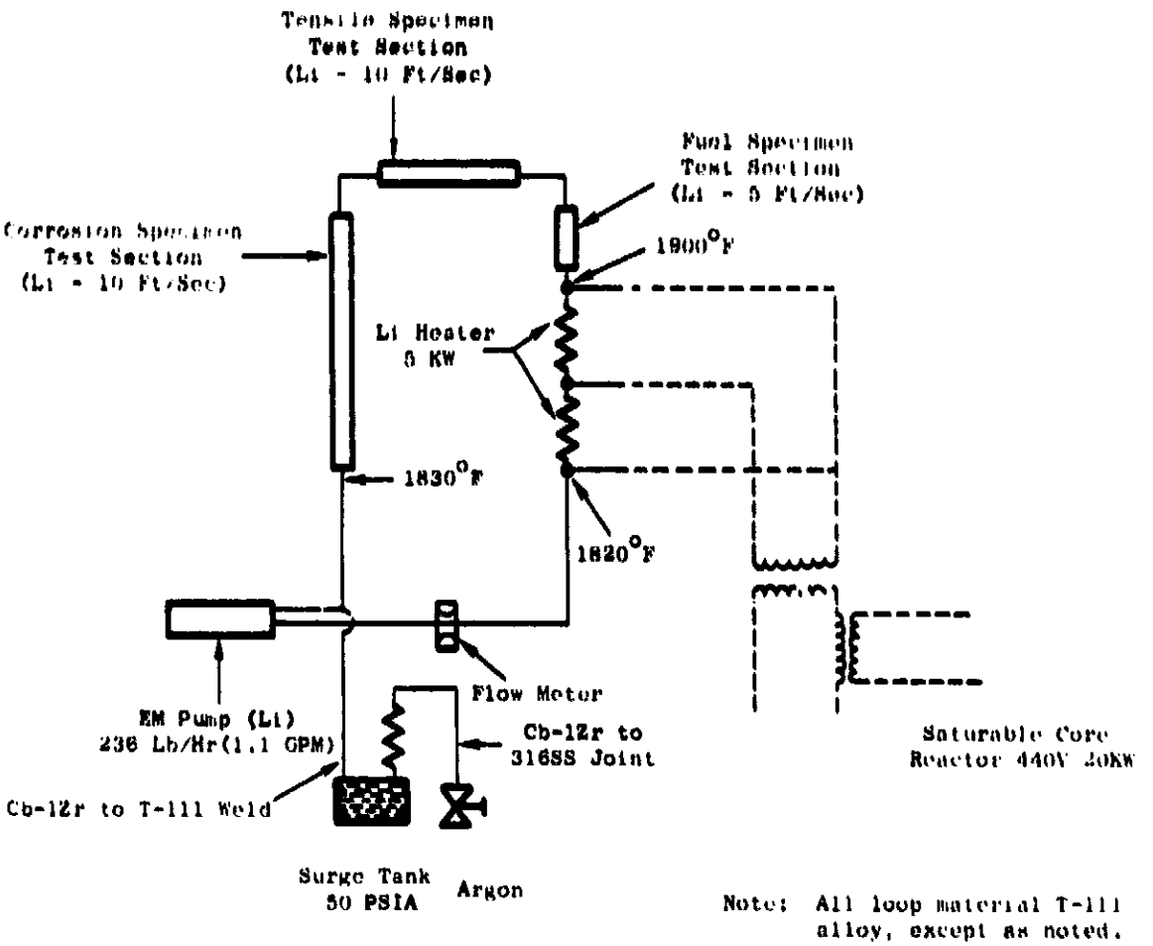


Figure 2. - Schematic diagram of 1040° C (1900° F) lithium loop, showing design operating conditions.

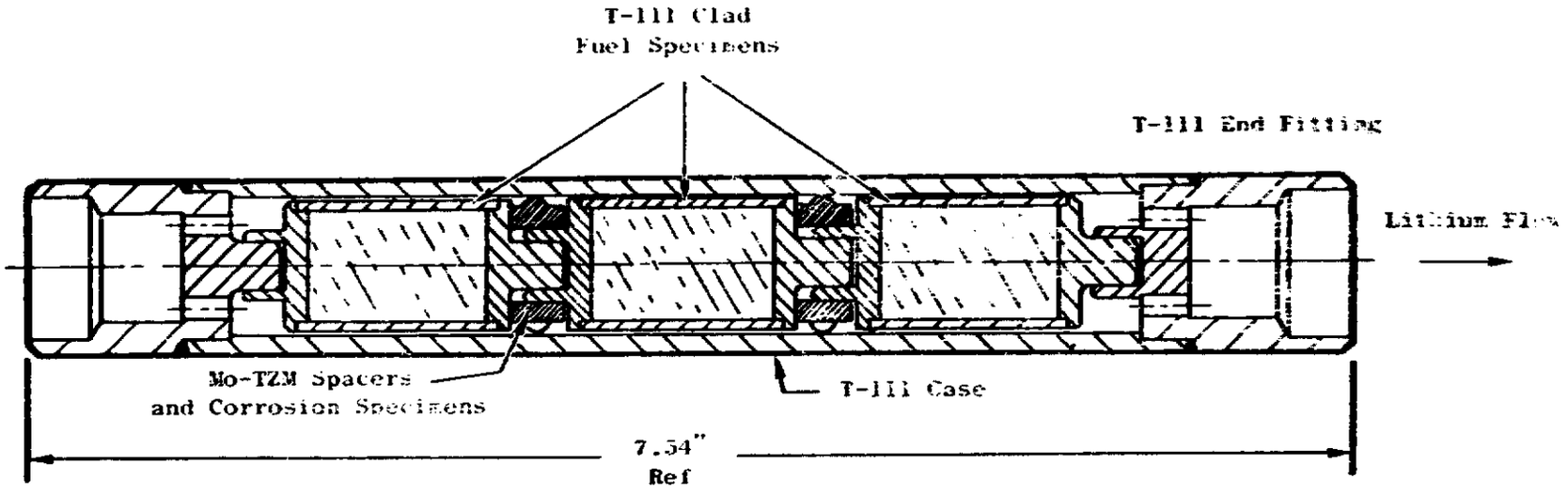


Figure 3. - Fuel specimen test section - 1040° C (1900° F) lithium loop.

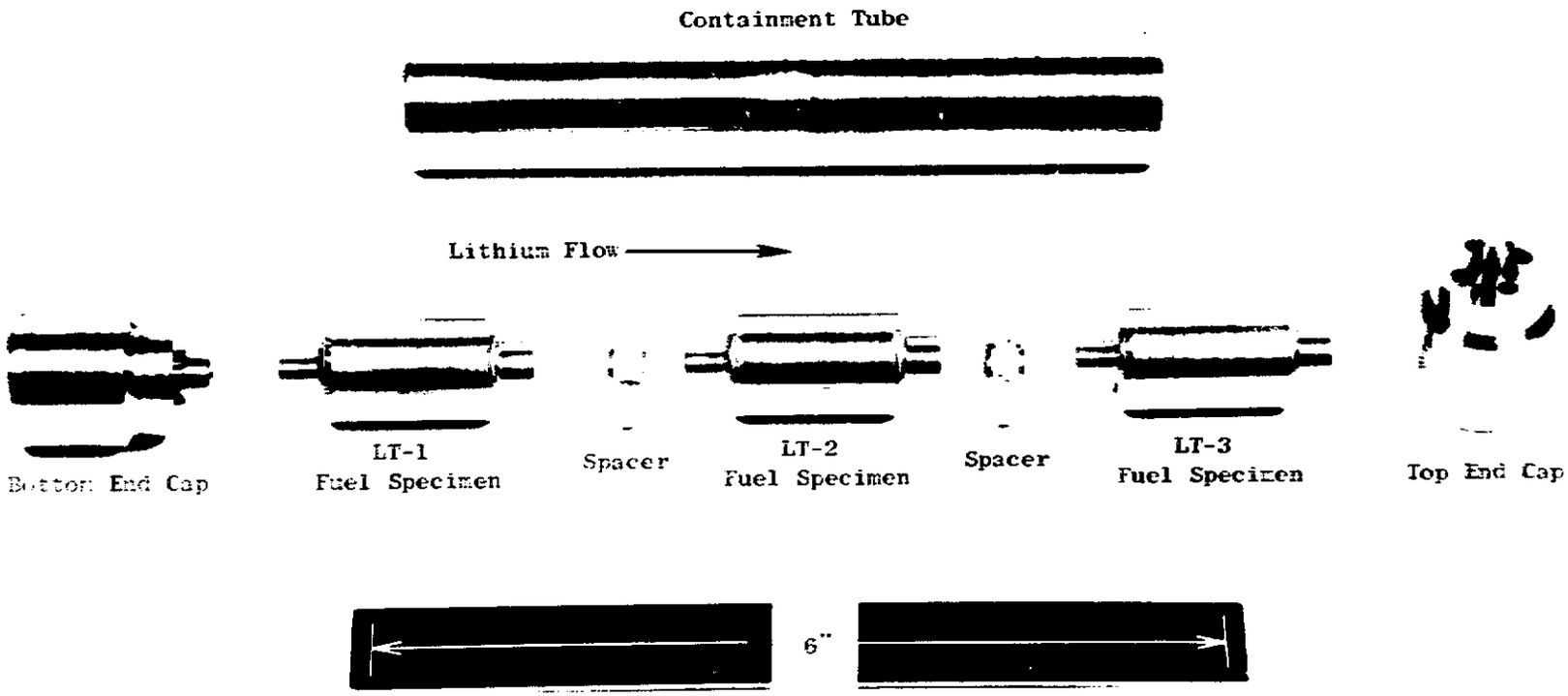


Figure 4. - Components of clad fuel element specimen capsule subassembly - 1040° C (1900° F) lithium loop. Containment tube, caps and specimen clad: T-111. Spacers, Mo-TZM.

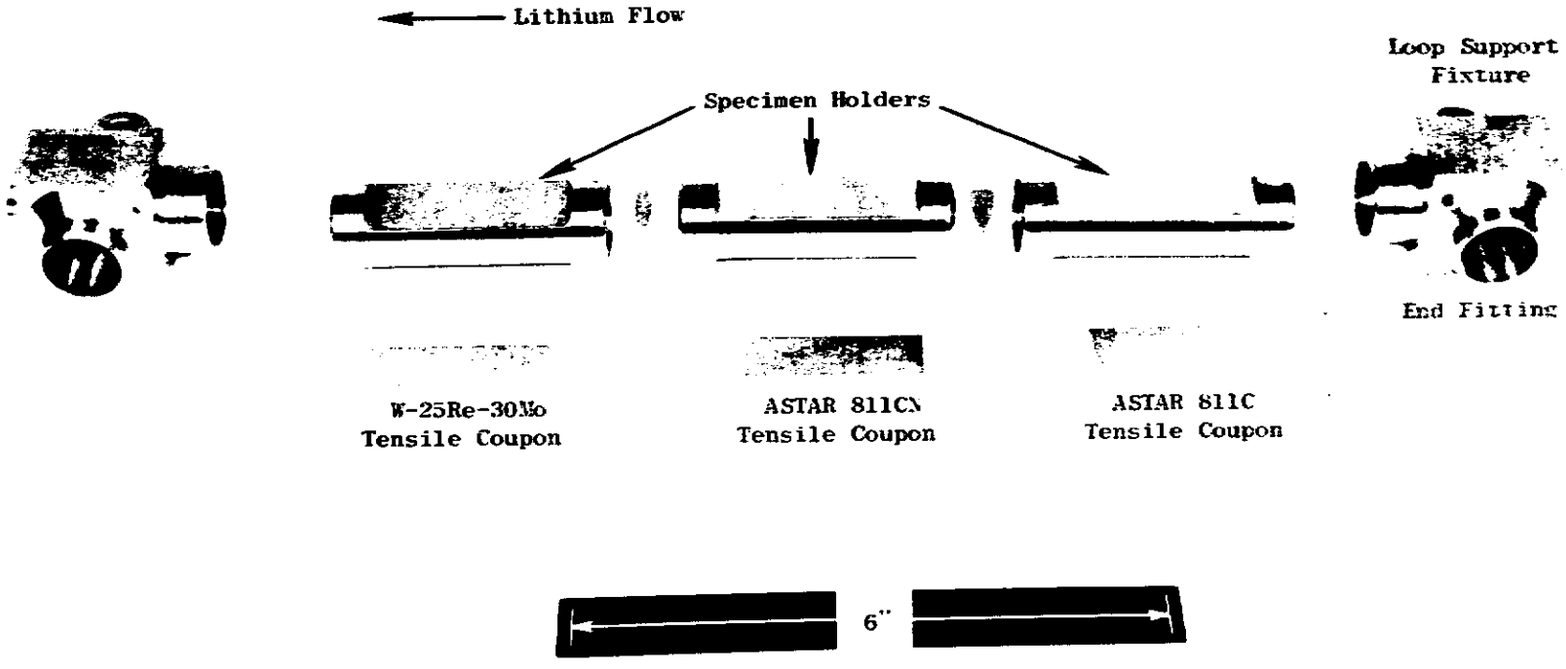


Figure 5. - Components of tensile test specimen subassembly - 1040° C (1900° F) lithium loop. Specimen holder and fittings: T-111.

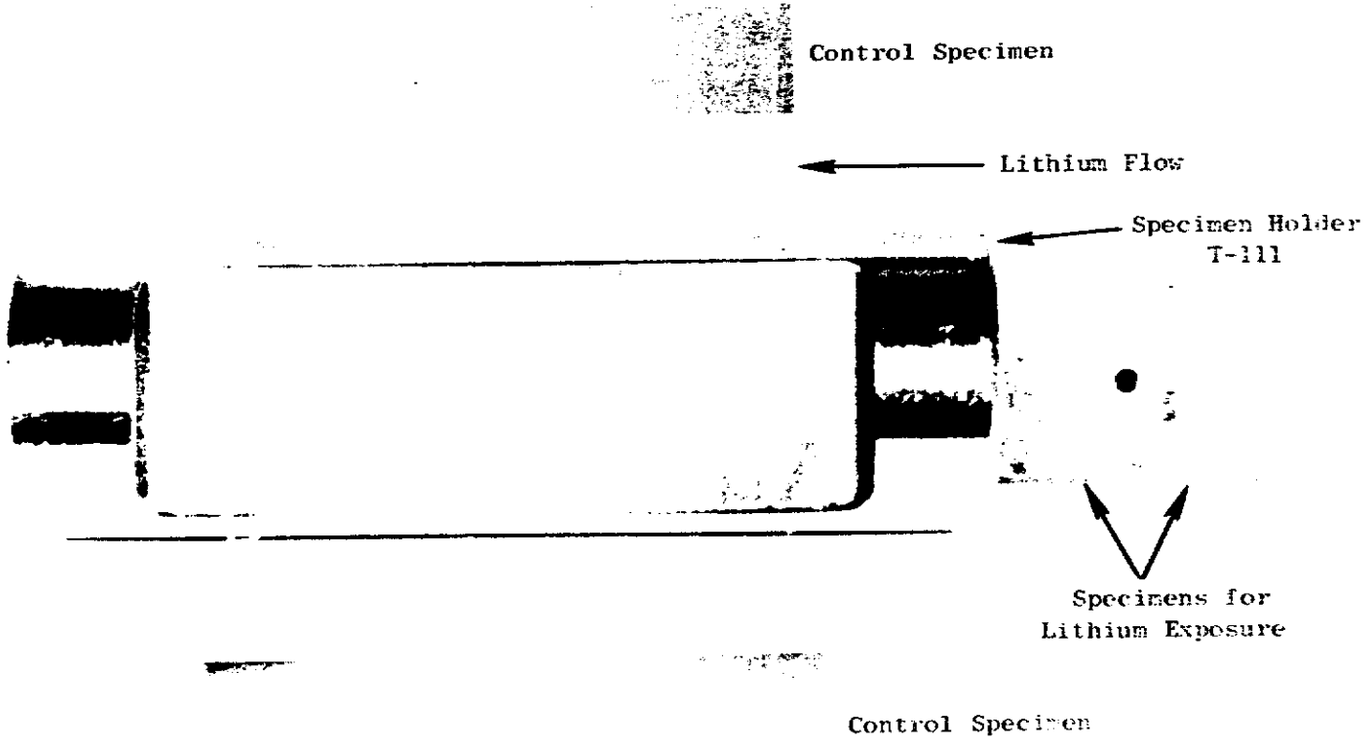


Figure 6. - Assembly of tensile test specimen holder - 1040° C (1900° F) lithium loop.

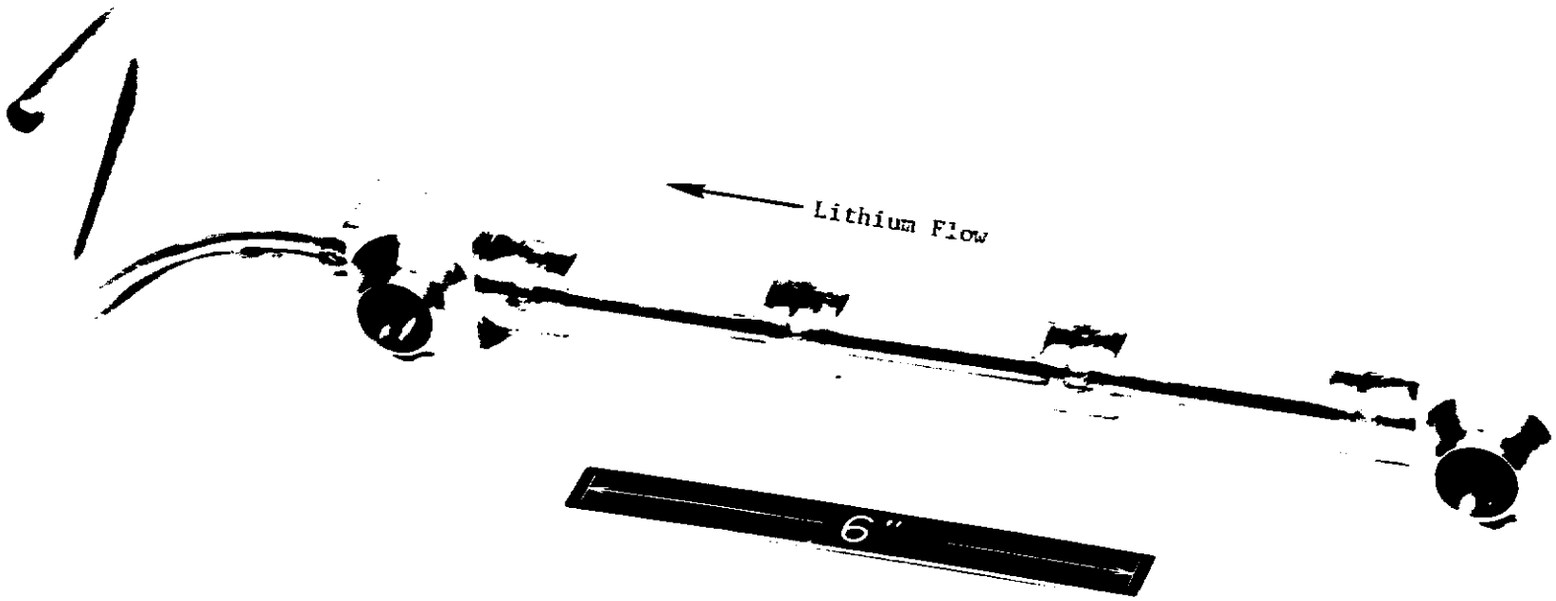


Figure 7. - Tensile test specimen subassembly - 1040° C (1900° F) lithium loop.

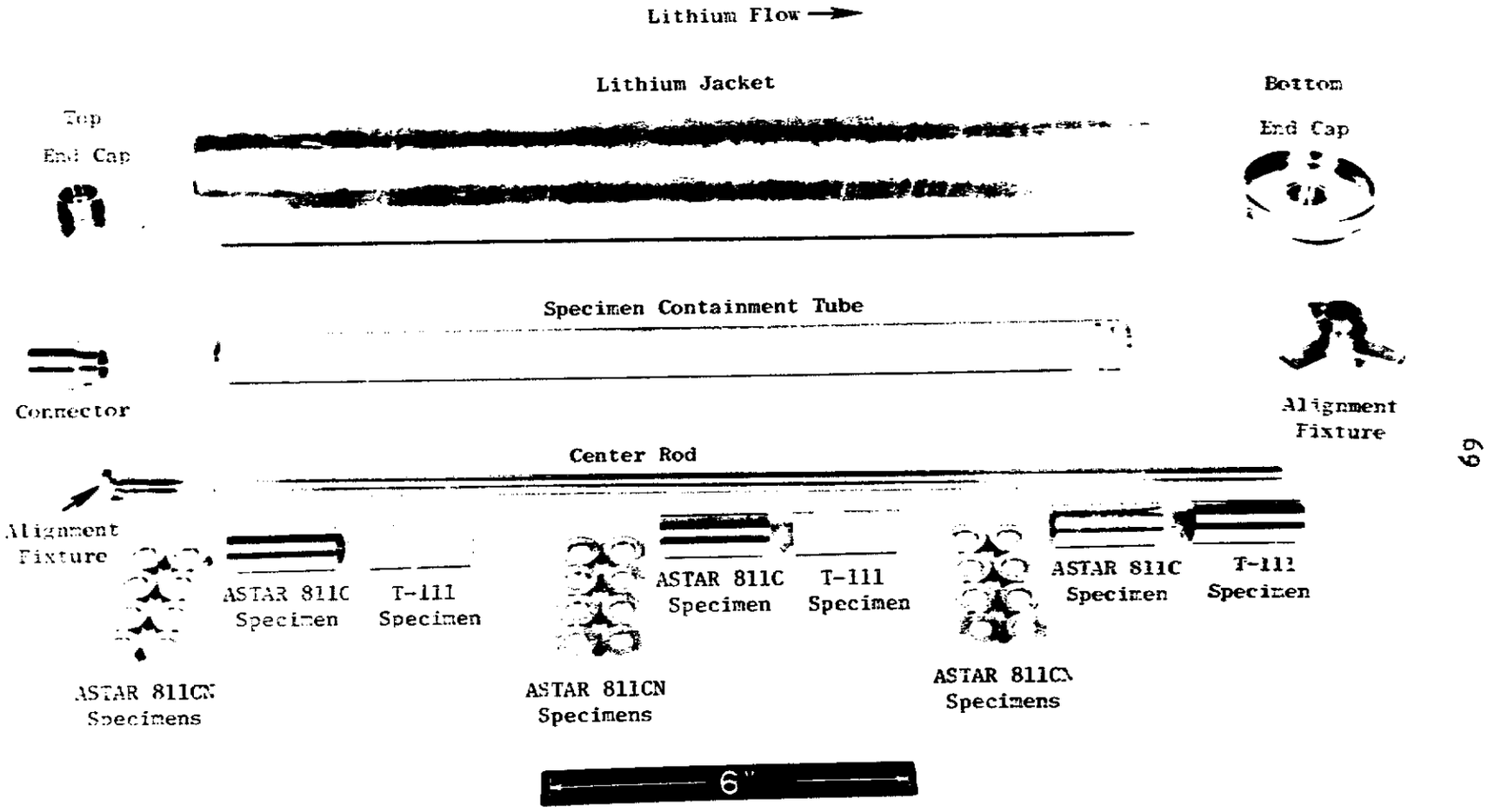


Figure 8. – Components of corrosion specimen subassembly – 1040° C (1900° F) lithium loop. All components not designated are T-111.

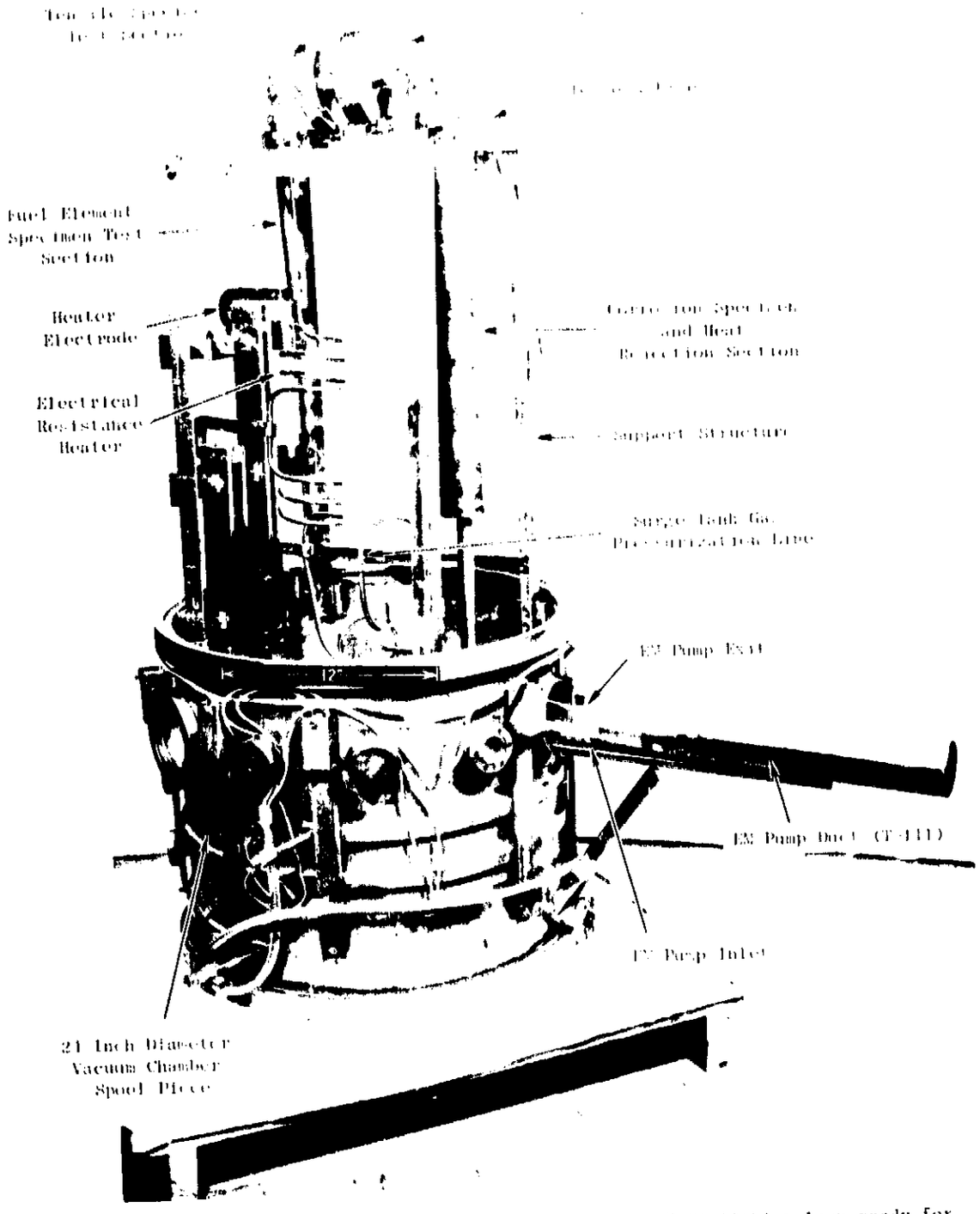


Figure 9. - Assembled T-111 alloy 1040° C (1900° F) lithium loop ready for instrumentation and insulation. (69-11-9B)

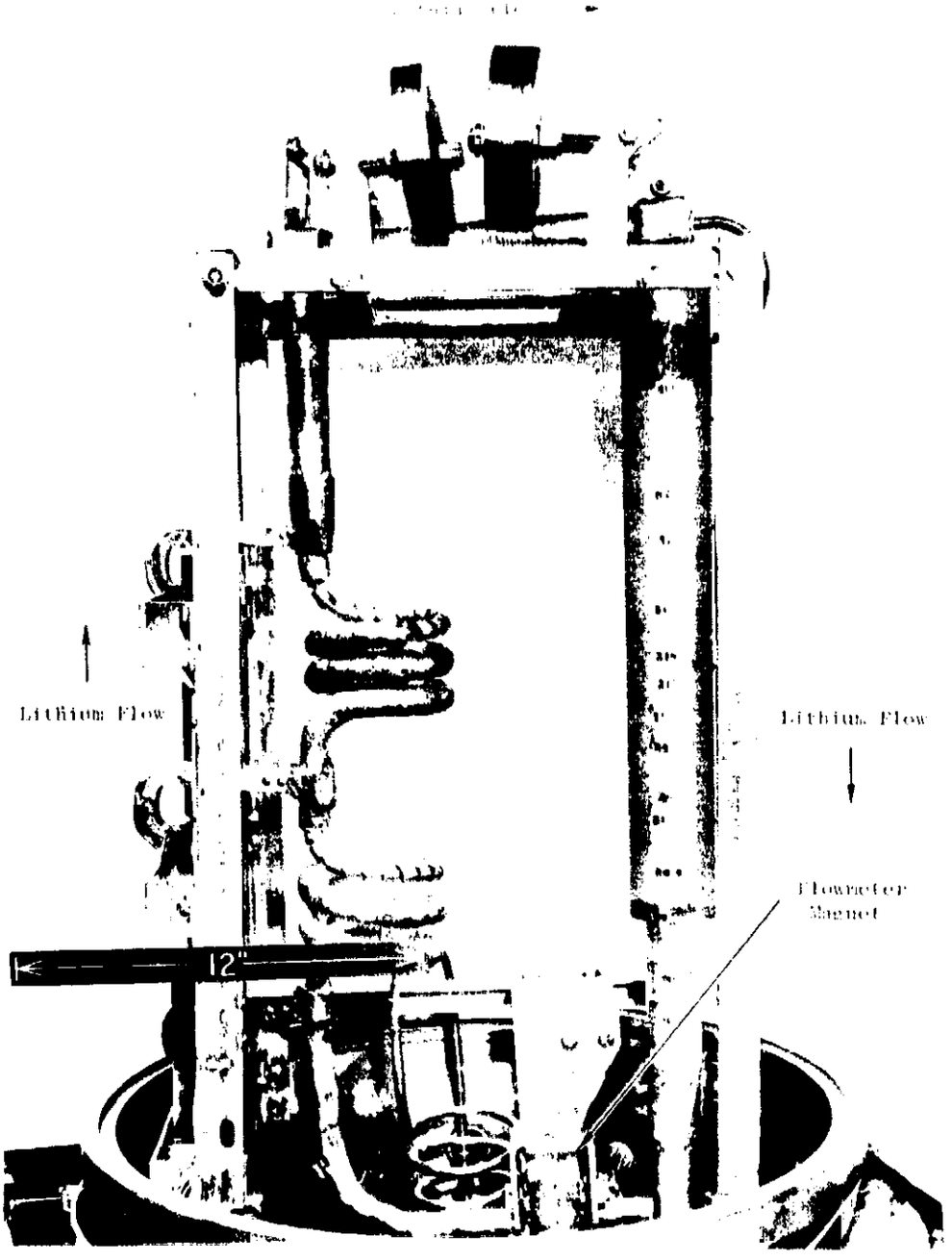


Figure 10. - Completely instrumented and insulated 1040° C (1900° F) lithium loop. (70-1-2A)

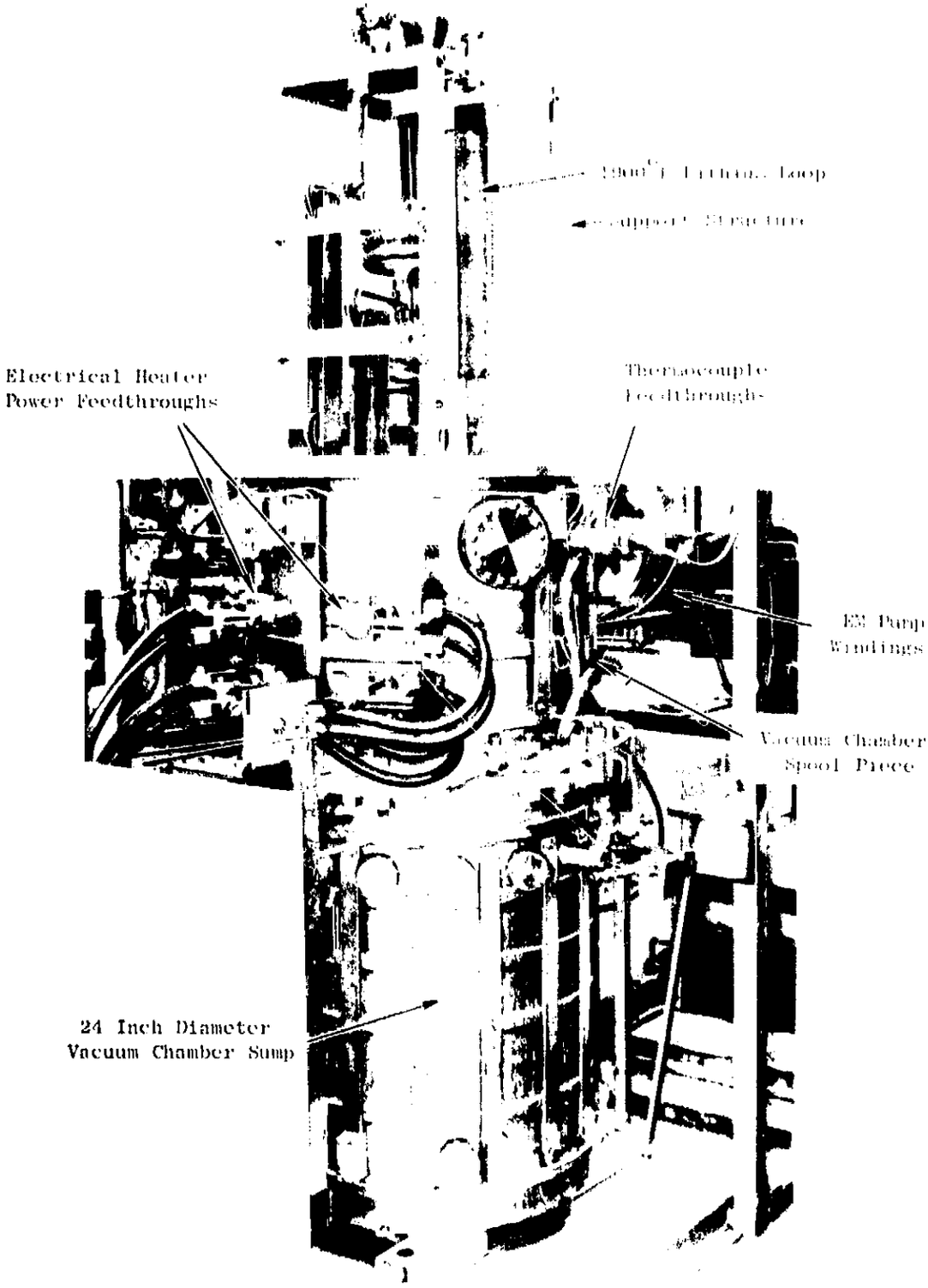


Figure 11. - 1040° C (1900° F) lithium loop located on vacuum chamber just prior to positioning of the bell jar. (C-61-2B)

Date 5-18-70 Test Time 2500 Hrs.
Chamber Pressure 2.3 x 10^{-9} Torr
Heater 39 A 305 V
Flow 2.2 mv 1.0 GPM

TEMPERATURES IN ^{o}F

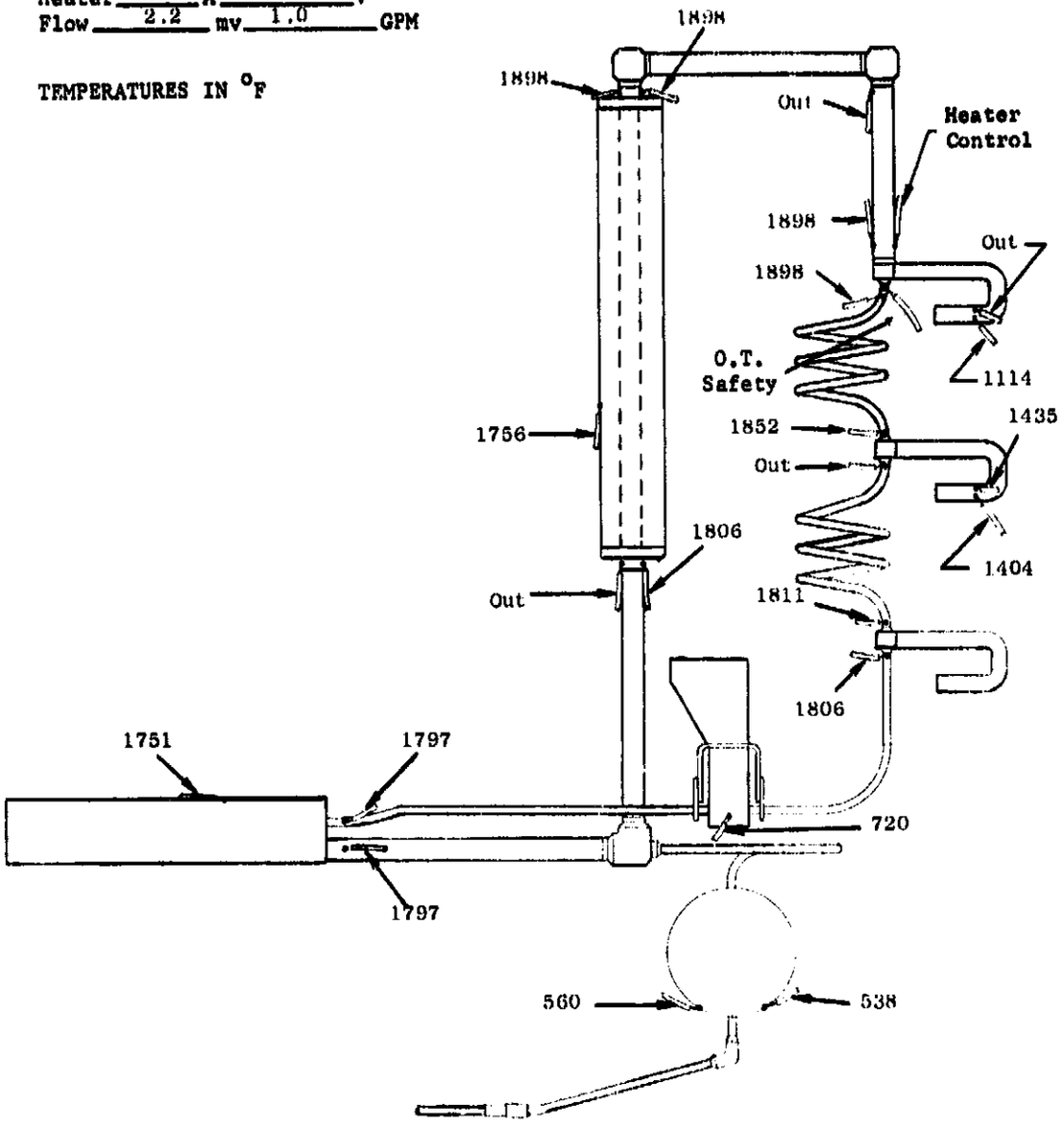


Figure 12. - 1040° C (1900° F) lithium loop operating temperatures - 2500 hours.

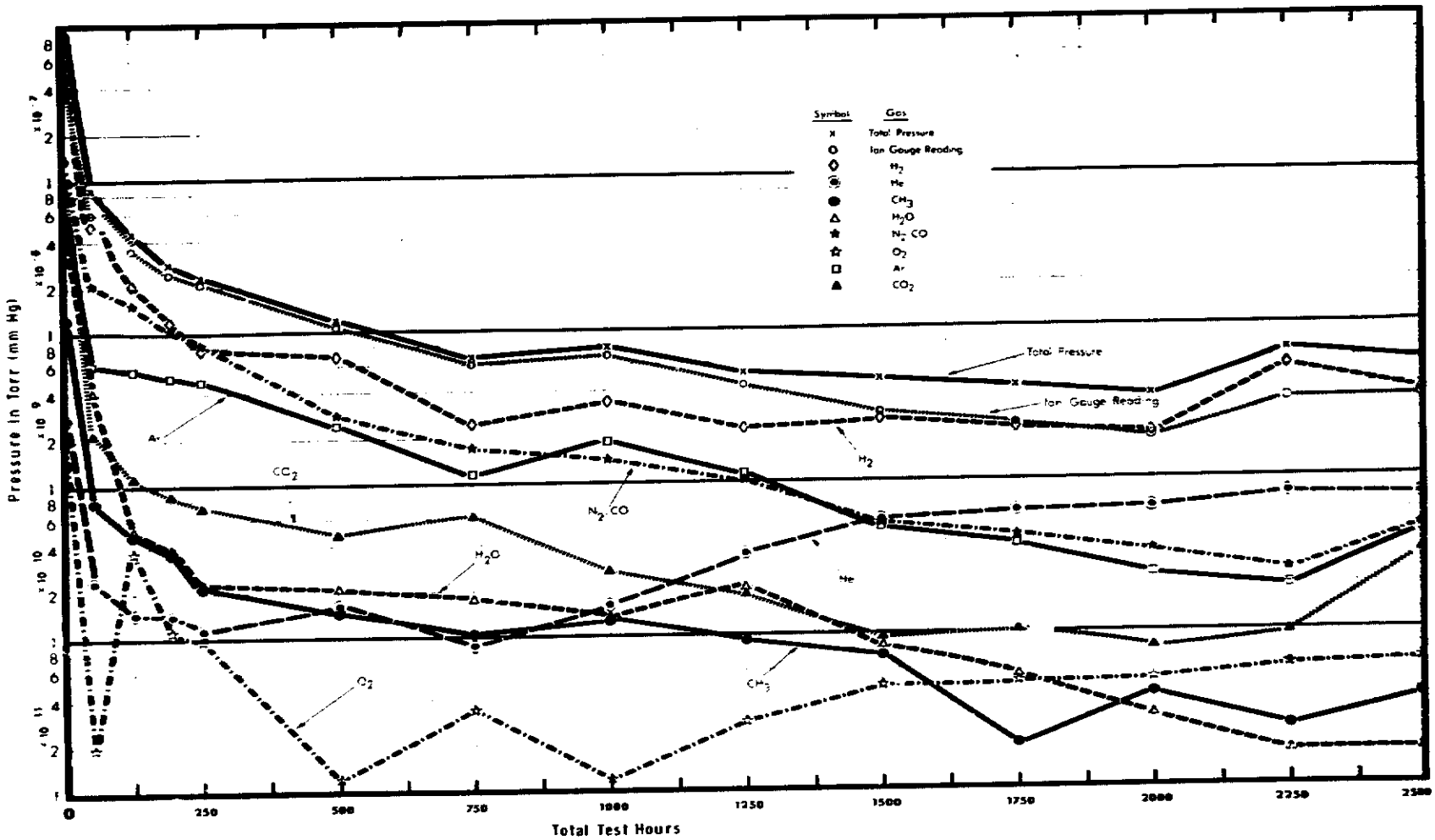


Figure 13. - Test chamber environment during testing 1040° C (1900° F) lithium loop.

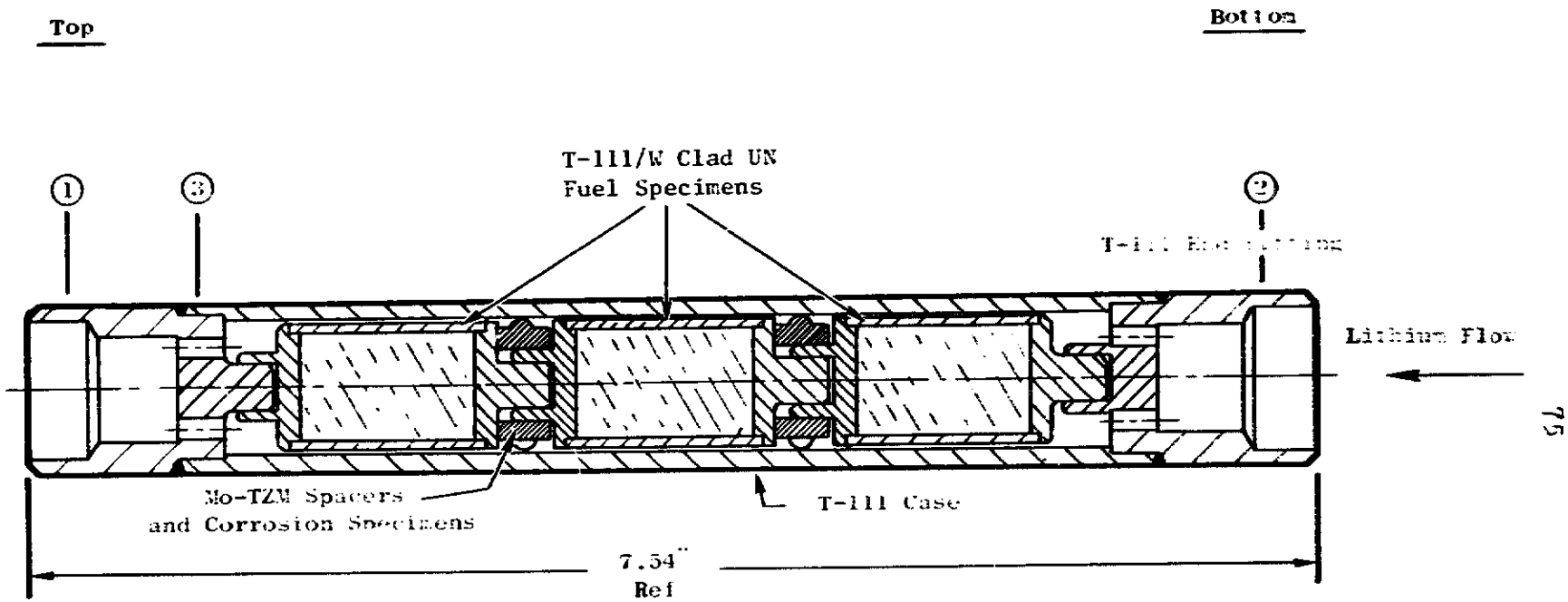


Figure 14. - Fuel specimen test section - 1040° C (1900° F) lithium loop.

(470-1)

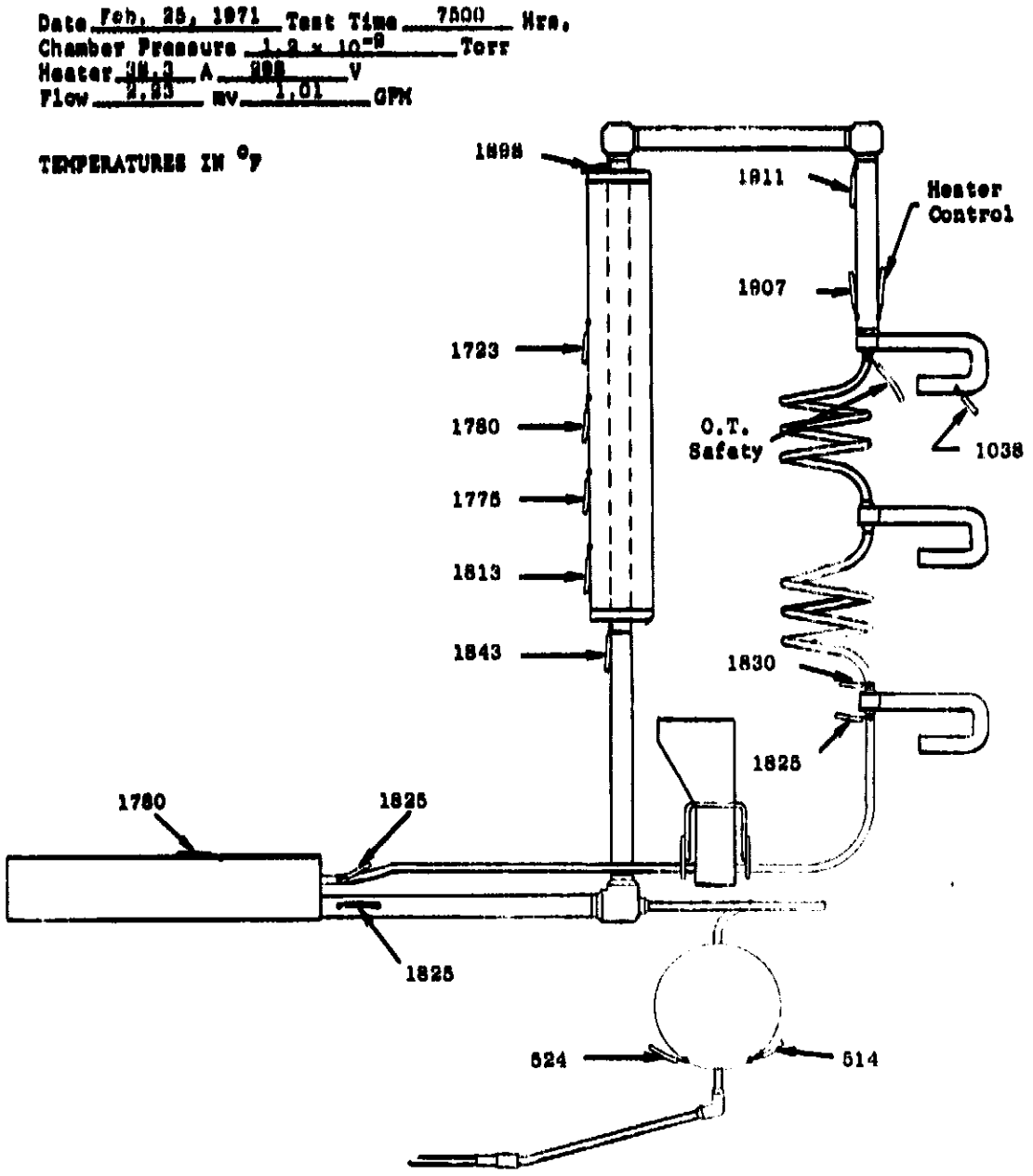


Figure 15. - 1040° C (1900° F) lithium loop operating temperatures - 7500 hours.

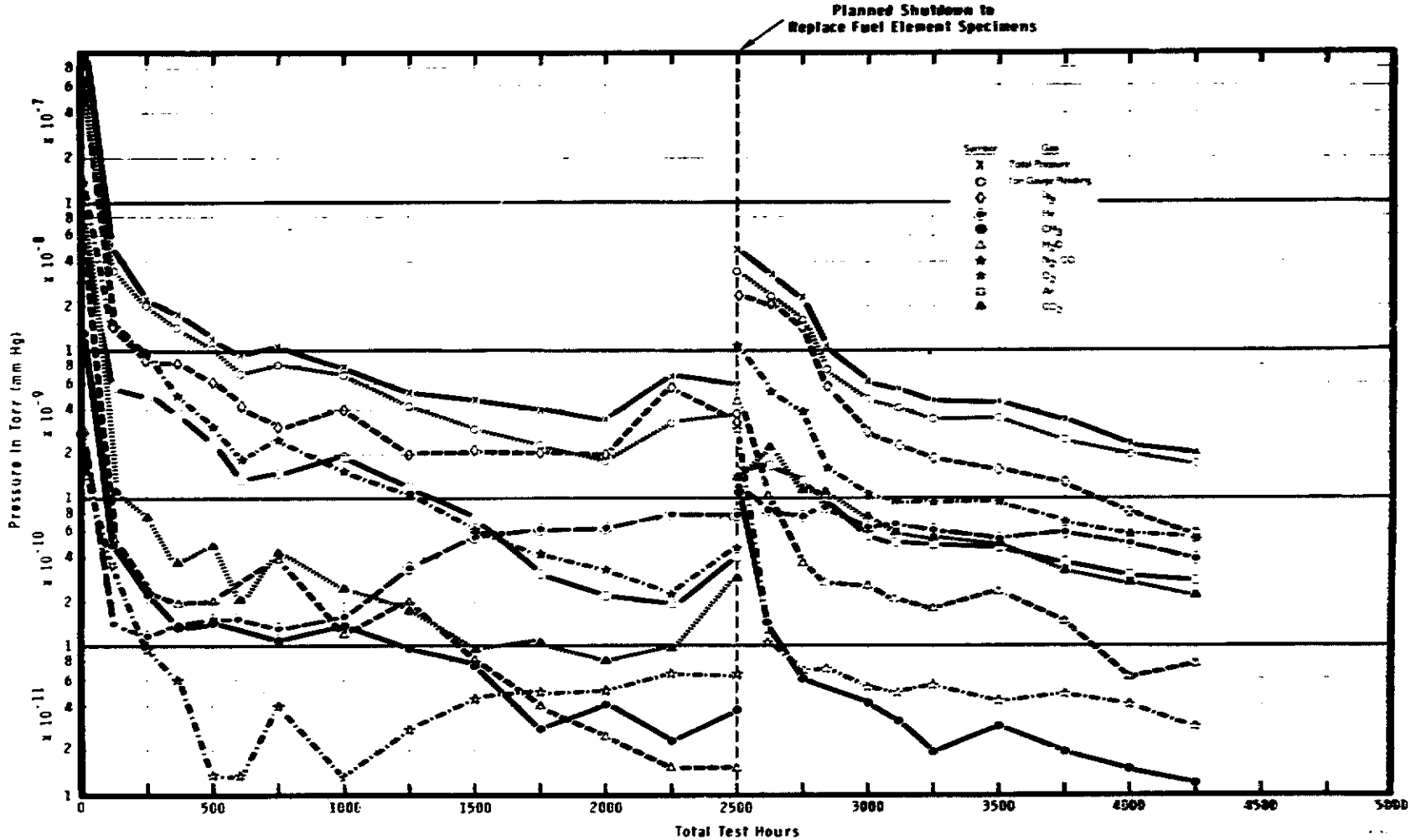


Figure 16. - Test chamber environment during testing of 1040° C (1900° F) lithium loop.

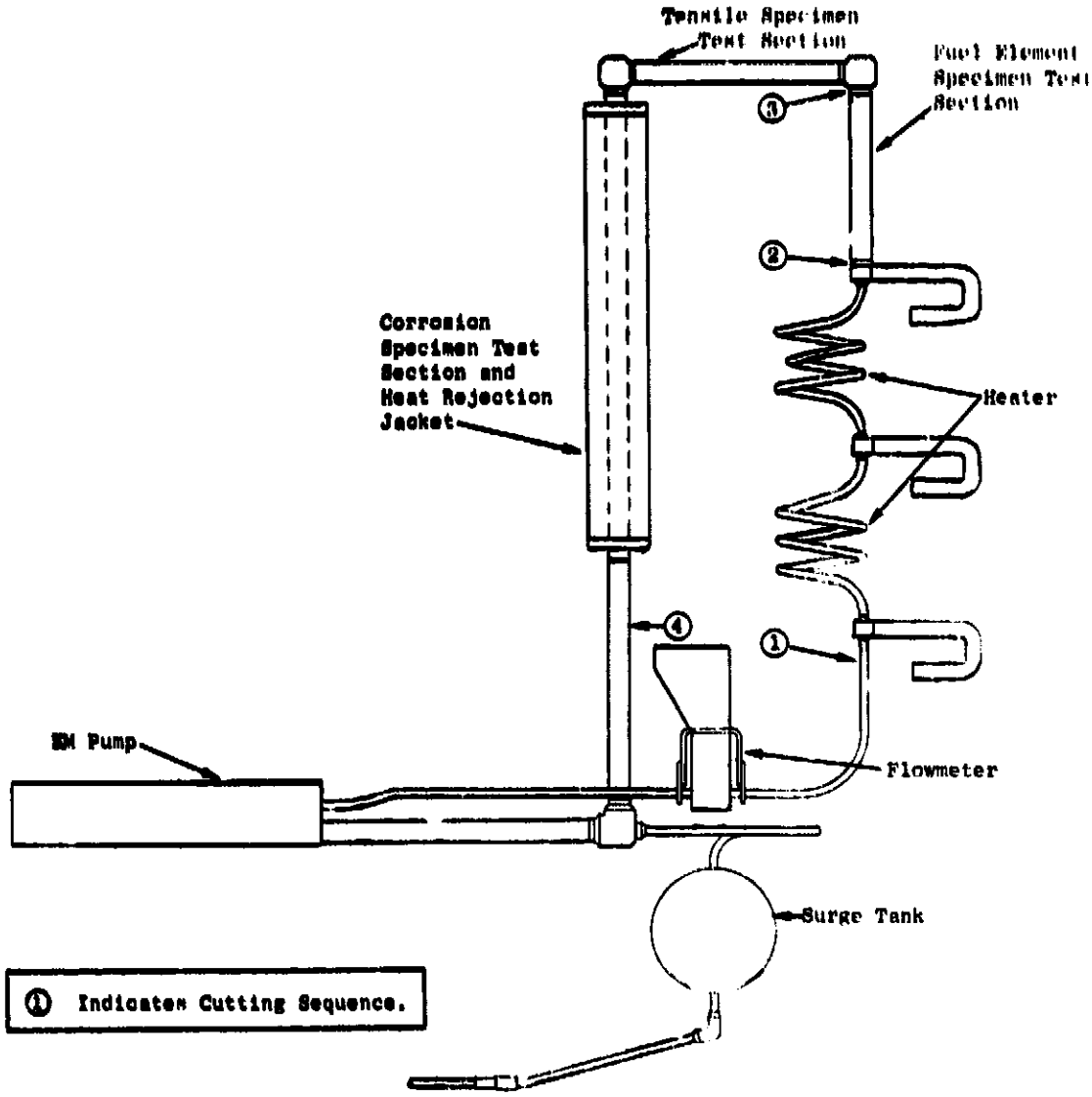


Figure 17. - 1040° C (1900° F) lithium loop showing location of cuts to remove the test sections of heater for posttest evaluation.

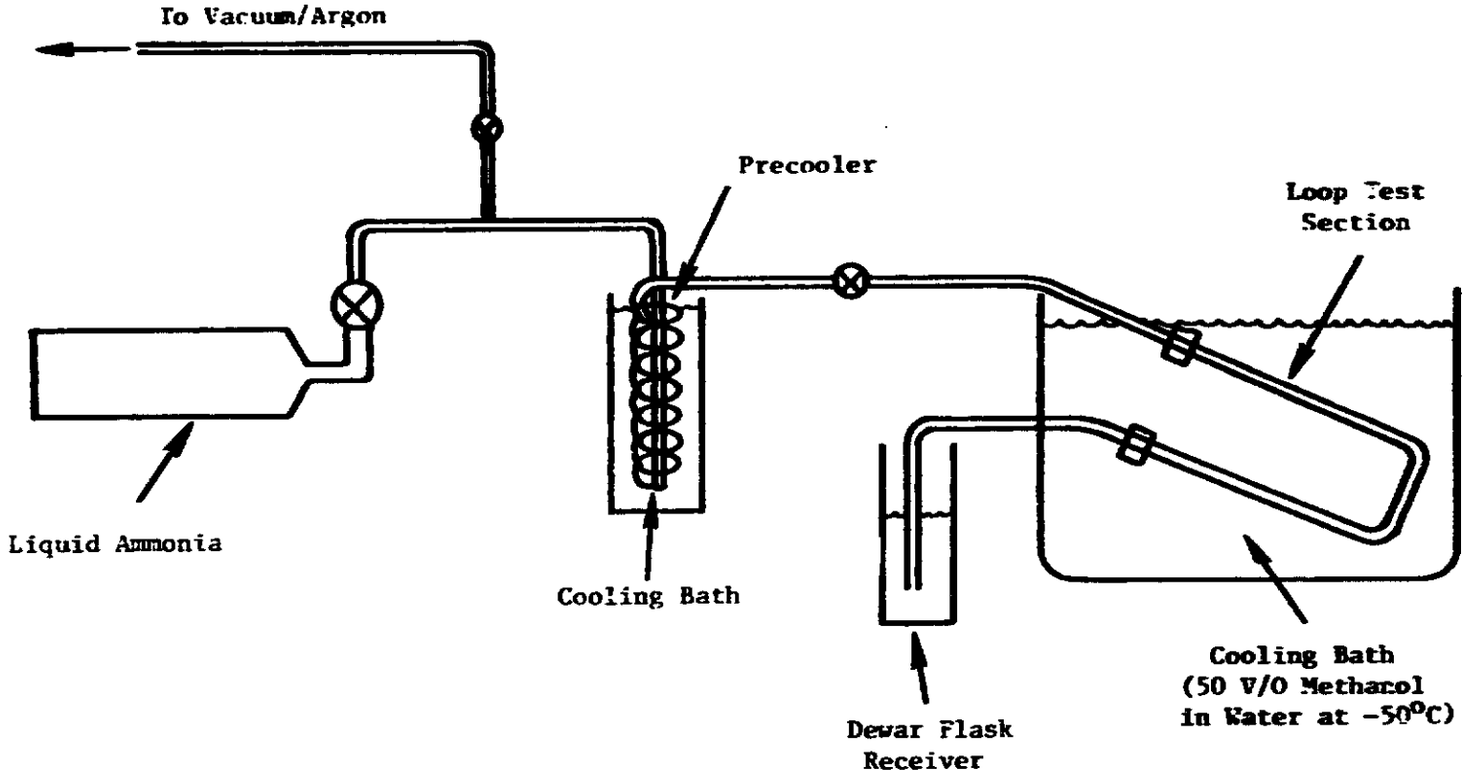


Figure 18. - Schematic of apparatus used to remove residual lithium from the loop test section by dissolution in liquid ammonia.

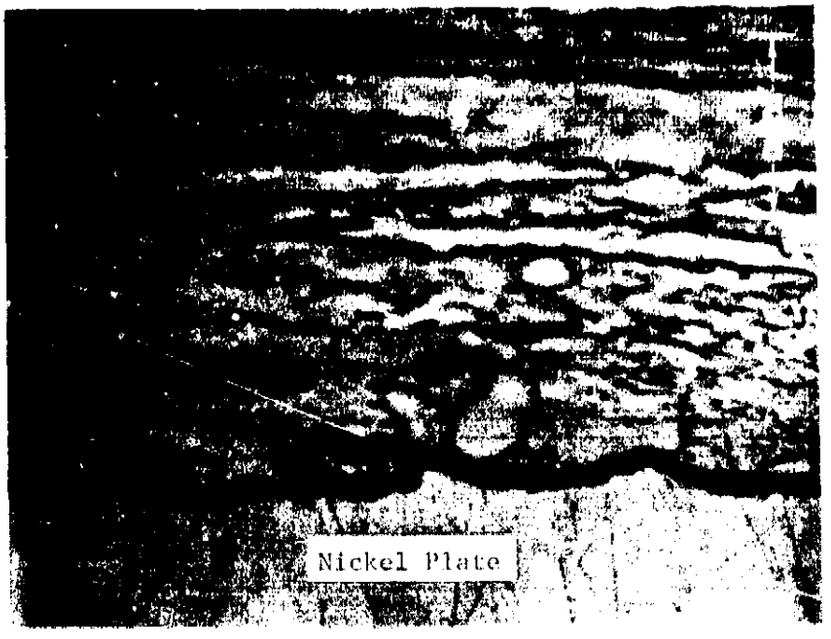
Unetched 1000X J86041C

ID Surface

Etched: 50% Murakami's 100X J86041D

OD Surface

Figure 19. - Pretest microstructure of Mo-TZM fuel element spacer for 1040° C (1900° F) pumped lithium loop.



Etched: 50% Murakami's 1000X J86021E

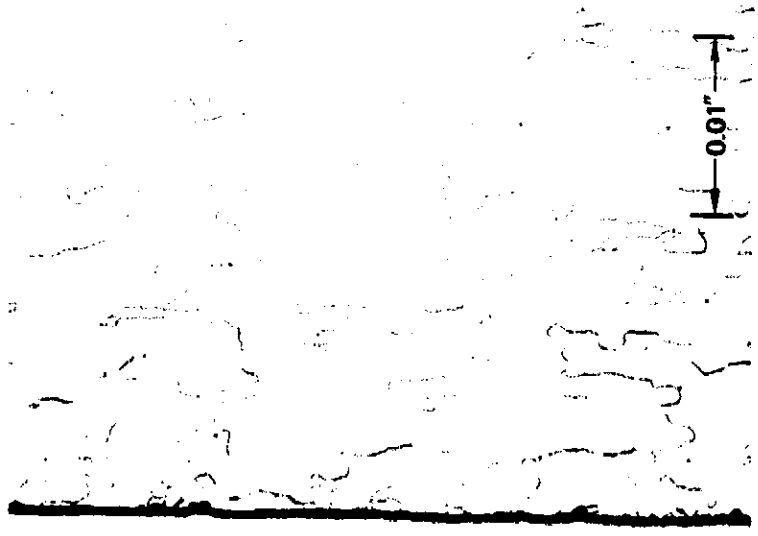
Nickel Plate

Etched: Murakami's 100X J86021D

Figure 20. - Mo-TZM fuel element spacer after 2500 hours exposure in the 1040° C (1900° F) pumped lithium loop.

Nickel Plate

Unetched 1000X J86031E



Nickel Plate

Etched: 50% Murakami's 100X J86031H

Figure 21. - Mo-TZM fuel element spacer after 5000 hours exposure in the 1040° C (1900° F) pumped lithium loop.

Nickel Plate

Etched: 50% Murakami's 1000X J86011E

Nickel Plate

Etched: 50% Murakami's 100X J86011D

Figure 22. - Mo-TZM fuel element spacer after 7500 hours exposure in the 1040° C (1900° F) pumped lithium loop.

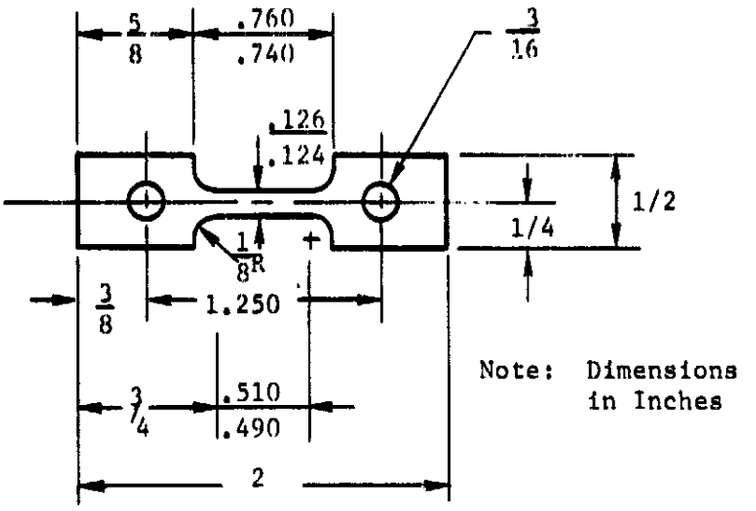


Figure 23. - Design of tensile test specimens machined from sheet coupons exposed in 1040° C (1900° F) lithium loop.

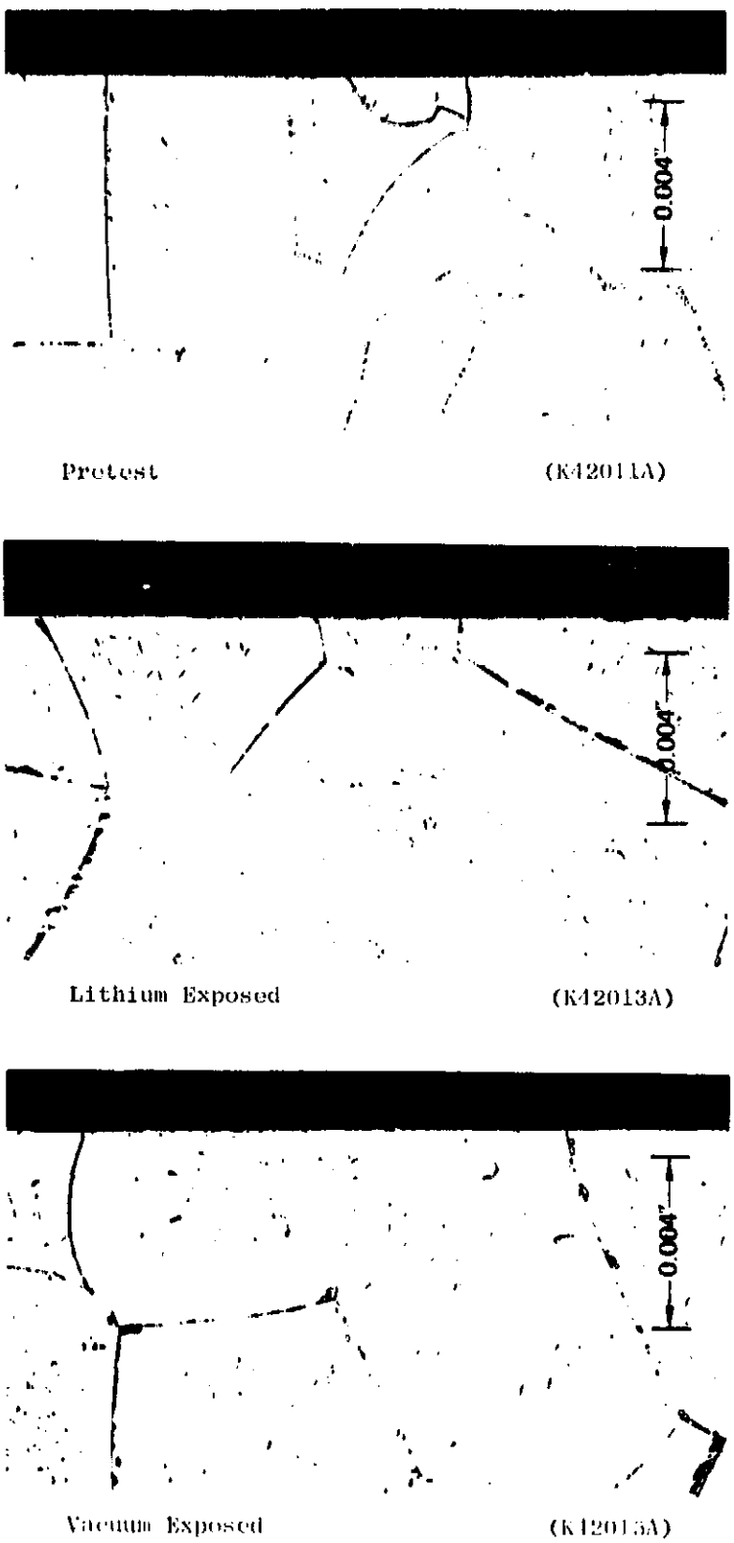
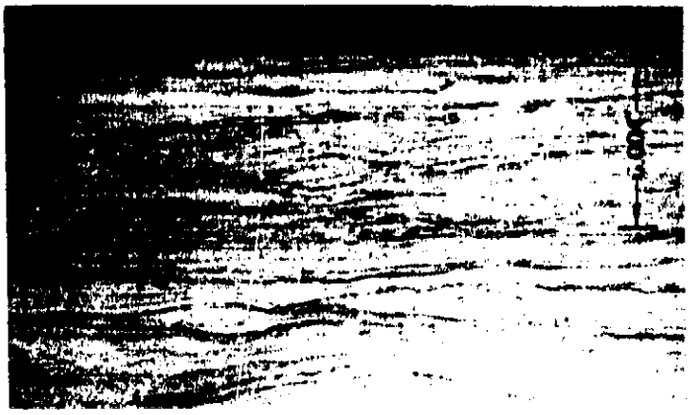
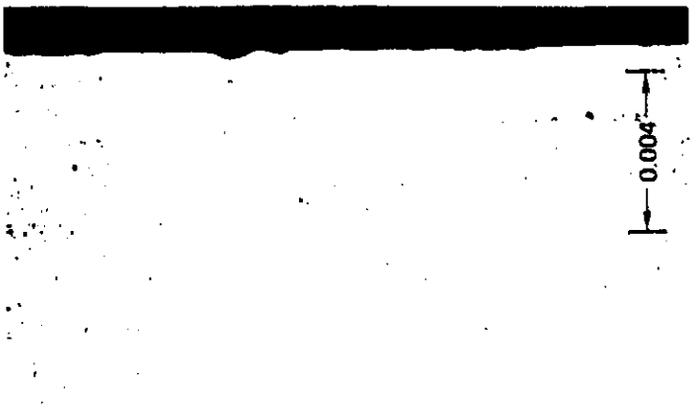


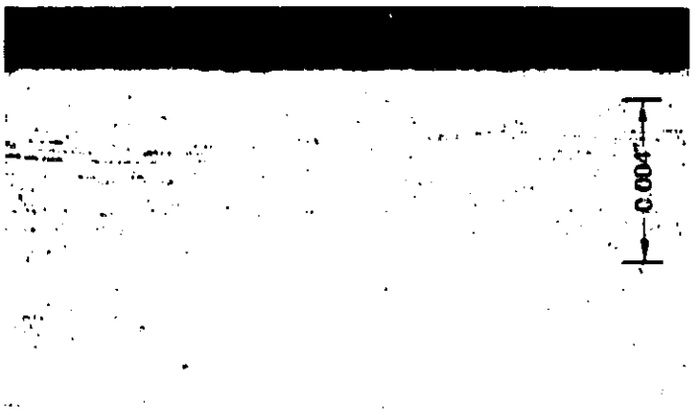
Figure 24. - Microstructures of ASTAR 811C alloy tensile specimens. No corrosion was observed on the lithium exposed specimens. Etchant: 30 g NH_4F - 50 ml HNO_3 - 20 ml H_2O.



Pretest (C950112)

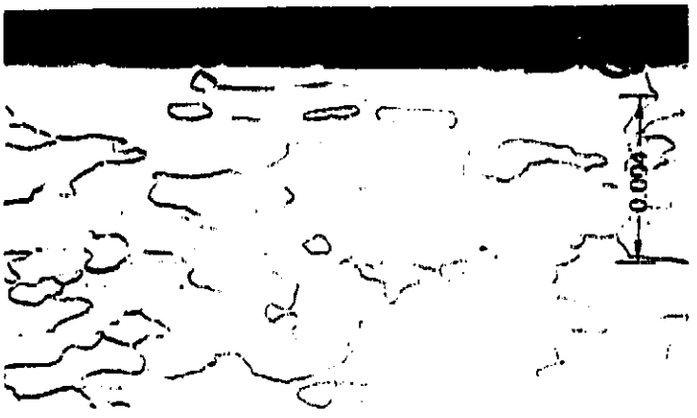


Lithium Exposed (K42023A)

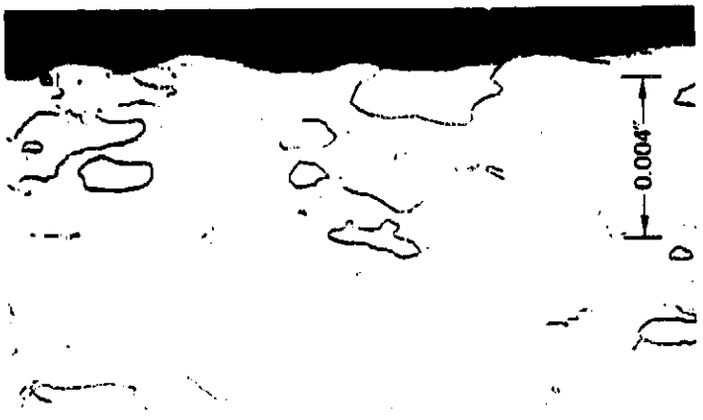


Vacuum Exposed (K42025A)

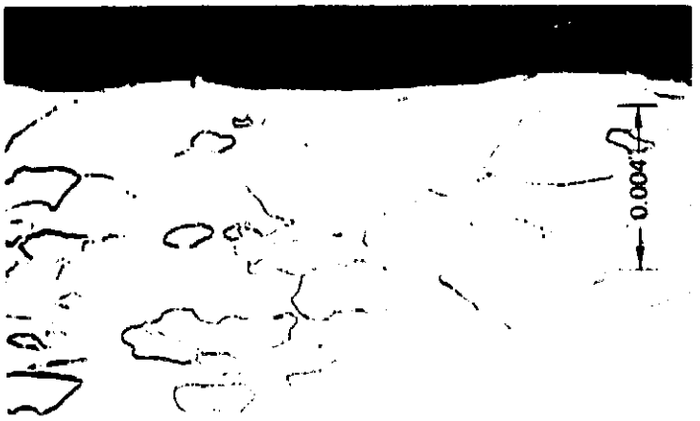
Figure 25. - Microstructures of ASTAR 811CN alloy tensile specimens. No corrosion was observed on the lithium exposed specimens. **Etchant:** 30 g NH_4F - 50 ml HNO_3 - 20 ml H_2O.



Pretest (K42031A)

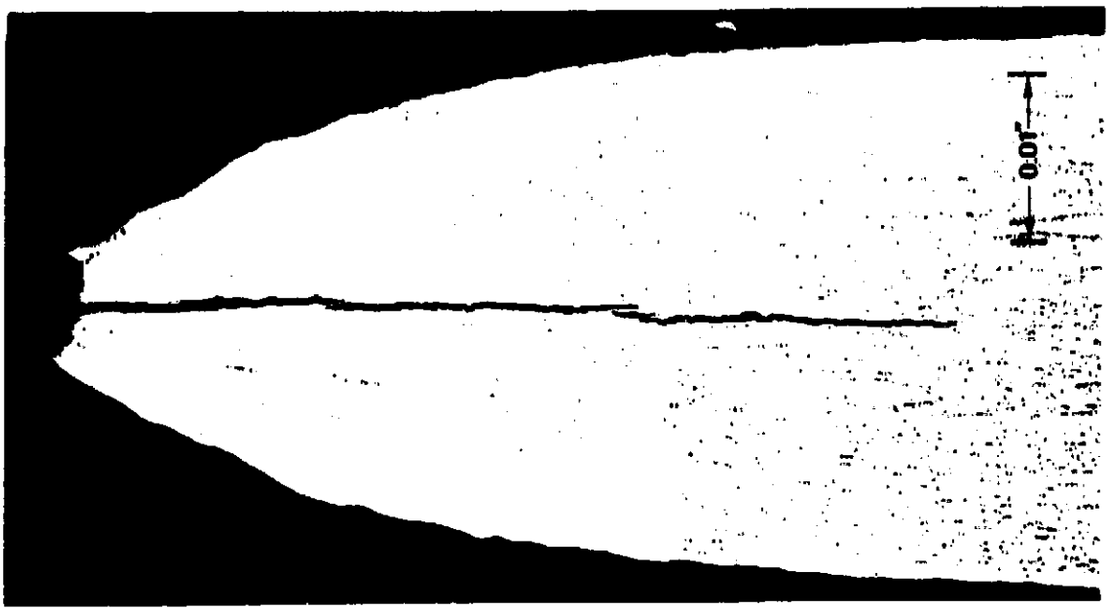


Lithium Exposed (K42033A)



Vacuum Exposed (K42035A)

Figure 26. - Microstructures of W-Re-Mo alloy 256 tensile specimens. No corrosion was observed on the lithium exposed specimens. Etchant: 20% Murakamis.

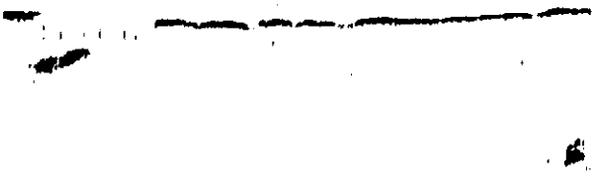


Lithium Exposed (K42024A, K42024B)

Vacuum Exposed (K42026A)

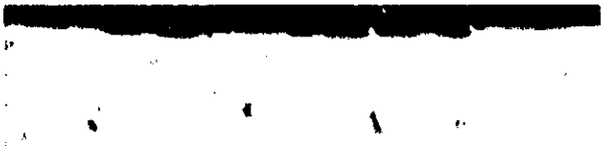
Figure 27. - Microstructures of ASTAR 811CN alloy tensile specimens exhibiting longitudinal cracking from fracture surface. Specimens exposed to lithium and vacuum for 5000 hours at 1040° C (1900° F) prior to tensile testing.

Etchant: 30 g NH_4F, 50 ml HNO_3, 20 ml H_2O.



Specimen 9A K23011A

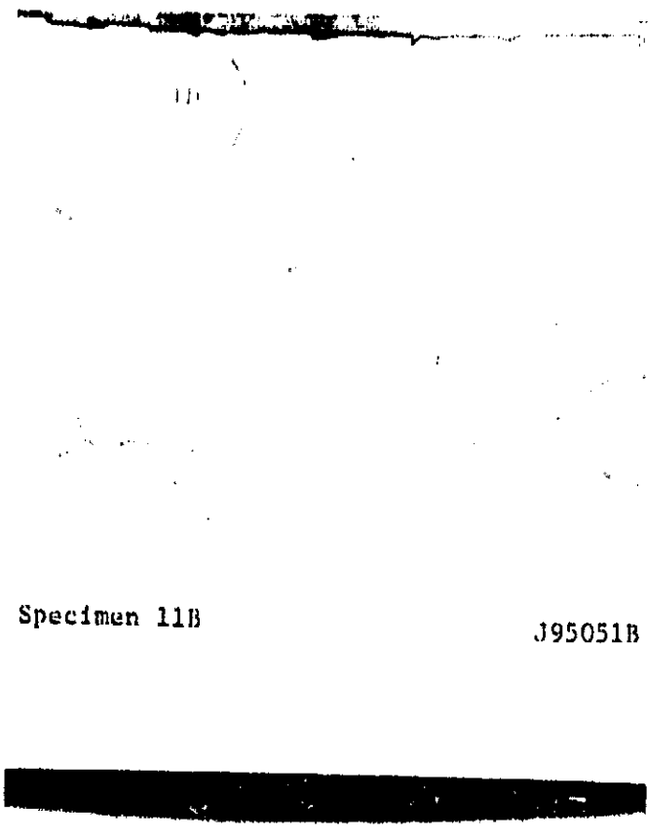
Specimen 9B J95021A



Specimen 9C J95031A

Figure 28. - Typical microstructures of T-111 alloy corrosion specimens exposed to flowing lithium in the 1040° C (1900° F) pumped lithium loop for 7500 hours. 100X

Etchant: 30 g NH_4F, 50 ml HNO_3, 20 ml H_2O.

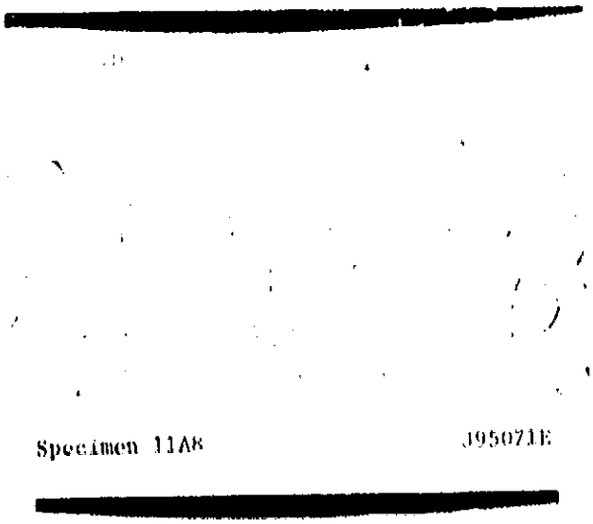


Specimen 11B J95051B

Specimen 11C J95061B

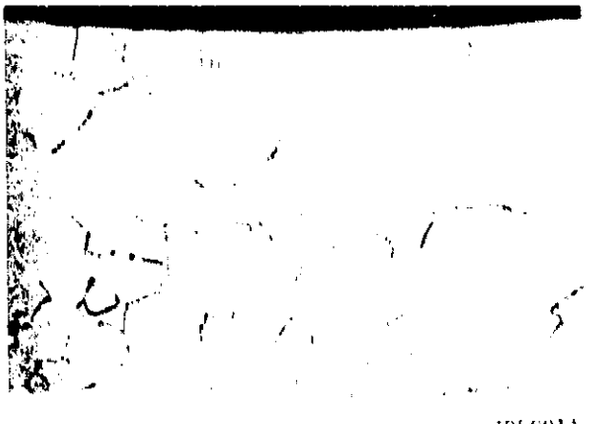
Figure 29. - Typical microstructures of ASTAR 811C alloy corrosion specimens exposed to flowing lithium in the 1040° C (1900° F) pumped lithium loop for 7500 hours. 100X

Etchant: 30 g NH_4F, 50 ml HNO_3, 20 ml H_2O.



Specimen 11A8 J95071E

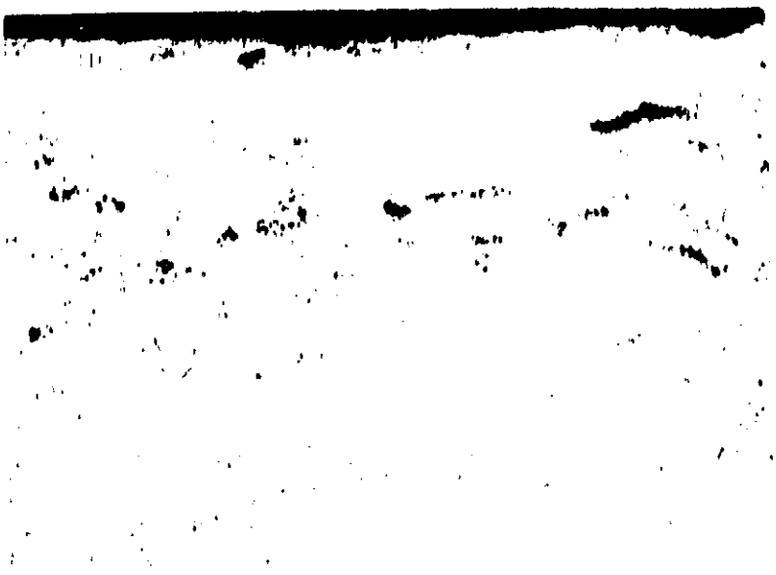
Specimen 11B8 J95081A



Specimen 11C1 J95091A

Figure 30. - Typical microstructures of ASTAR 811CN alloy corrosion specimens exposed to flowing lithium in the 1040° C (1900° F) pumped lithium loop for 7500 hours. 100X

Etchant: 30 g NH_4F, 50 ml HNO_3, 20 ml H_2O.



Lithium inlet 387011C

Lithium exit E230?1A

Figure 31. - Microstructure of Ti-11 alloy fuel element housing tubing in transverse section following 7500 hours exposure to flowing lithium in the 1040° C (1900° F) pumped lithium test loop. 250X

Etchant: 30 g NH_4F, 50 ml HNO_3, 20 ml H_2O.

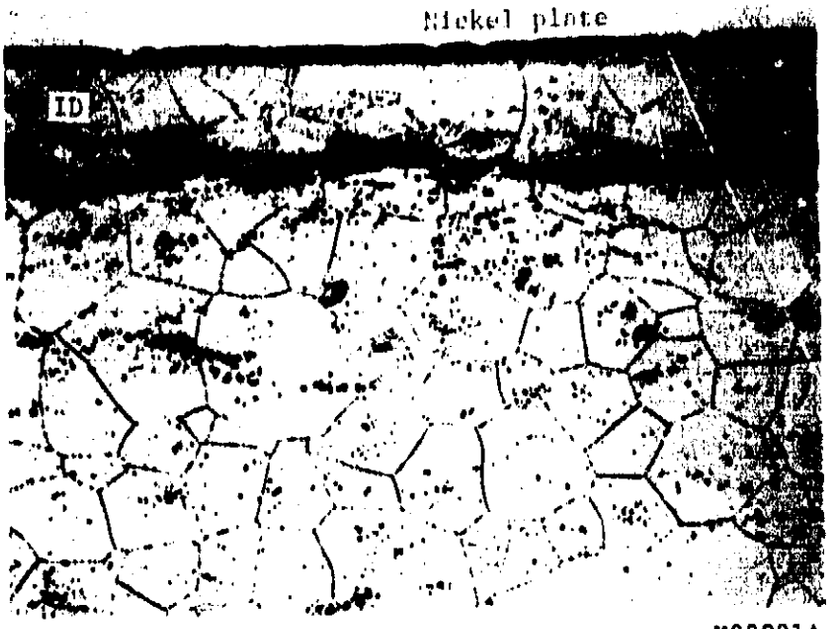
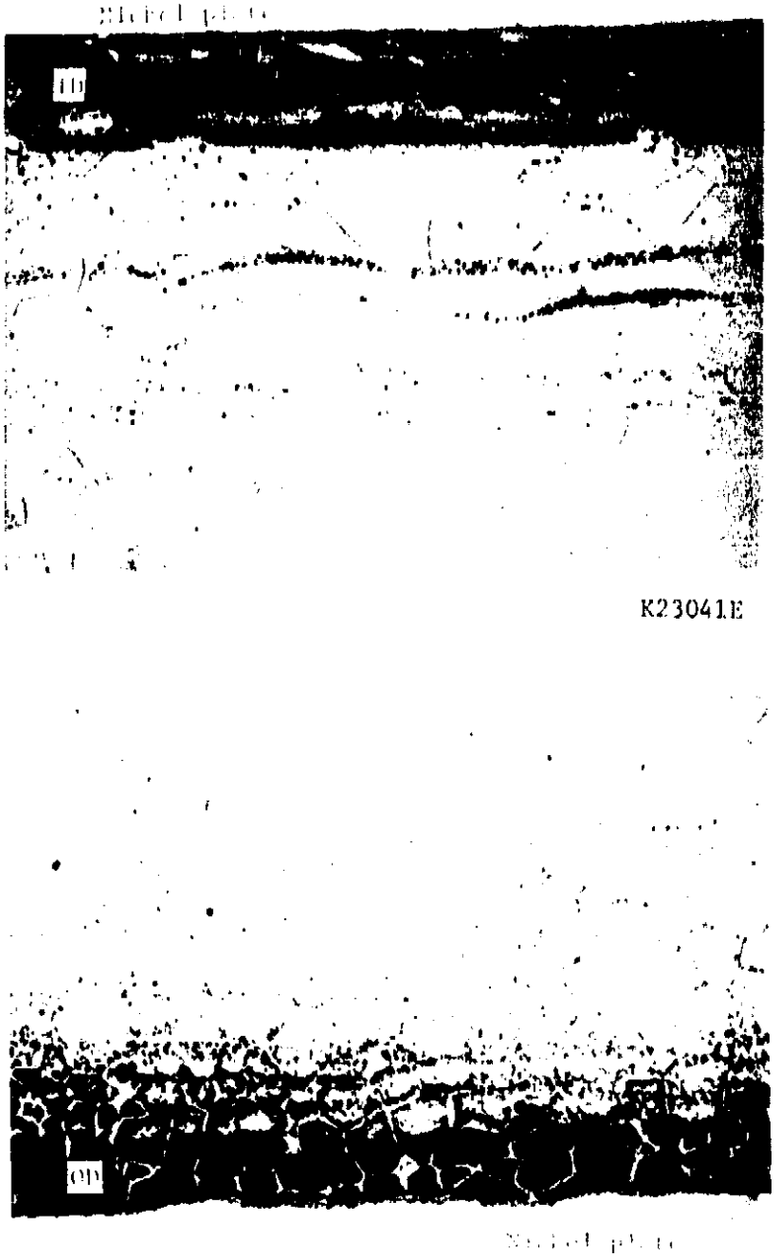


Figure 32. - Transverse microstructure of T-111 alloy corrosion specimen housing tubing following 7500 hours exposure to essentially stagnant lithium in the 1040° C (1900° F) pumped lithium test loop. 250X

Etchant: 30 g NH_4F, 50 ml HNO_3, 20 ml H_2O.

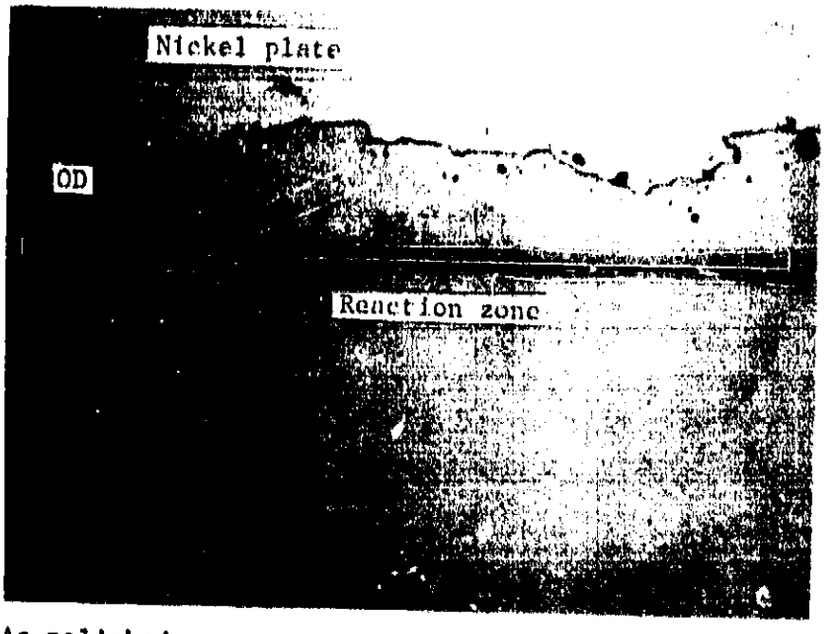


K23041E

K23041F

Figure 33. - Longitudinal microstructure of T-111 alloy corrosion specimen housing tubing in region of Li-Ar interface following 7500 hours exposure to essentially stagnant lithium in the 1040° C (1900° F) pumped lithium test loop. 250X

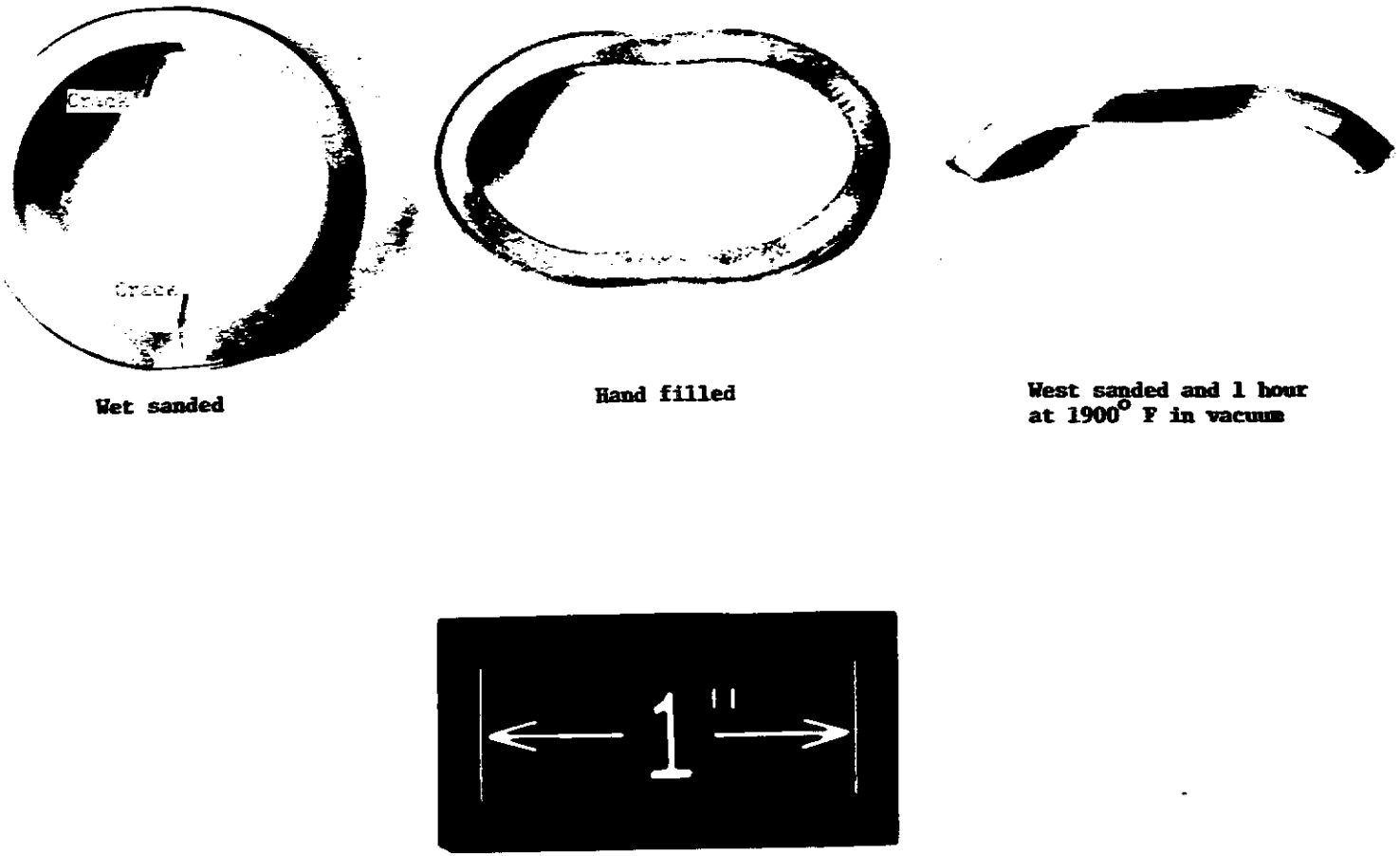
Etchant: 30 g NH_4F, 50 ml HNO_3, 20 ml H_2O.



As polished

K23041C

Figure 34. - Longitudinal section of T-111 alloy corrosion specimen housing tubing in region of Li-Ar interface following 7500 hours exposure to essentially stagnant lithium in the 1040° C (1900° F) pumped lithium loop. 1000X



Wet sanded

Hand filled

West sanded and 1 hour at 1900° F in vacuum

Figure 35. - T-111 fuel element housing ductility test specimens. All three specimens were removed from the fuel element specimen housing of the 1040° C (1900° F) pumped lithium test loop.

(P71-9-1A)

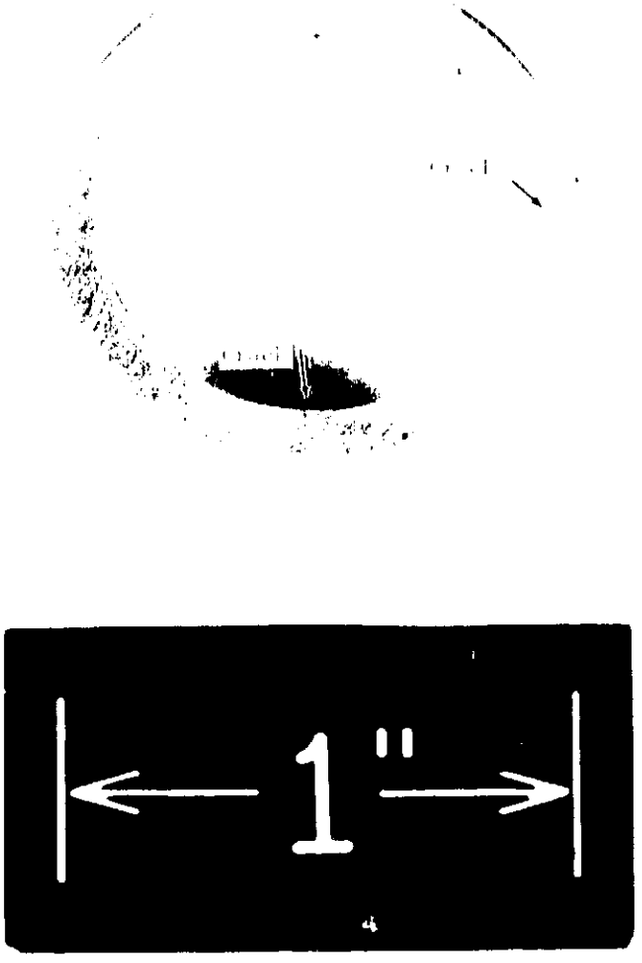


Figure 36. - Bend ductility specimen from corrosion specimen housing. Specimen prepared by wet polishing prior to bending. (P-71-9-1F)

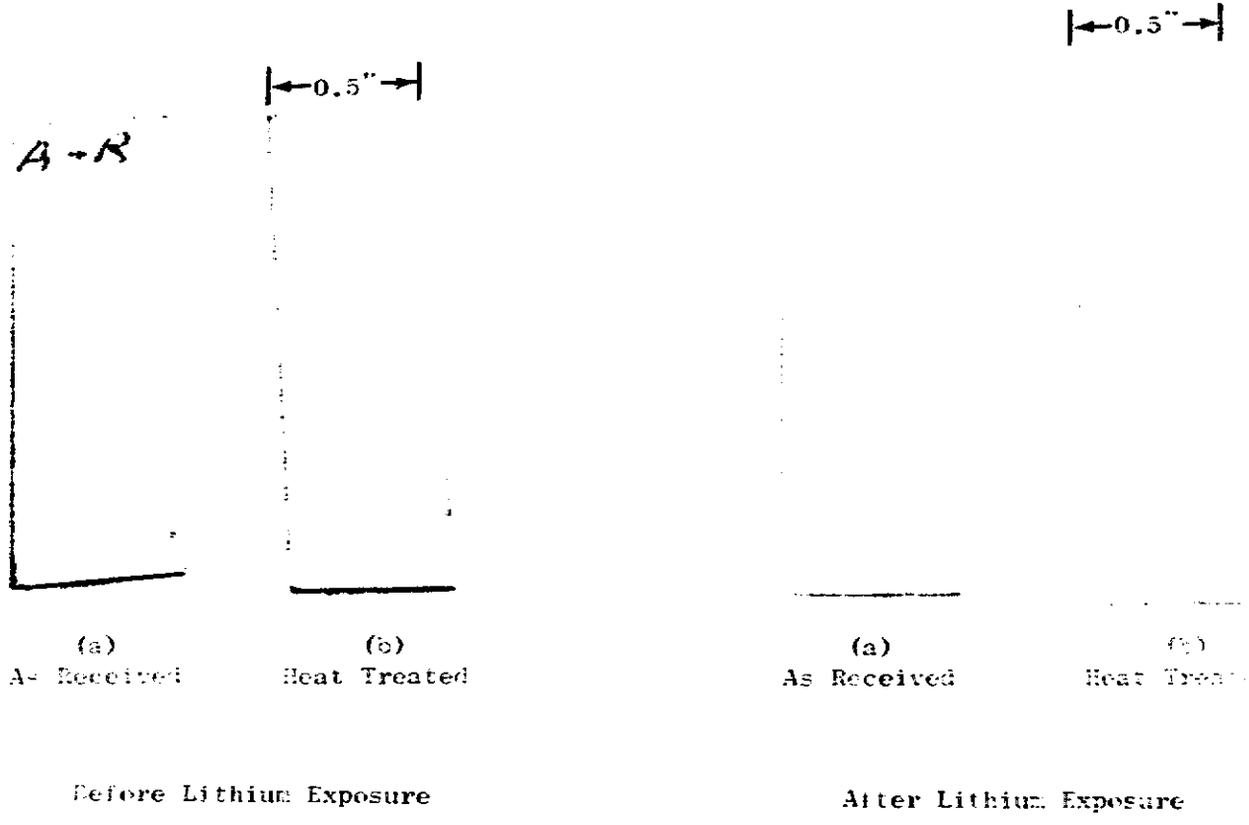
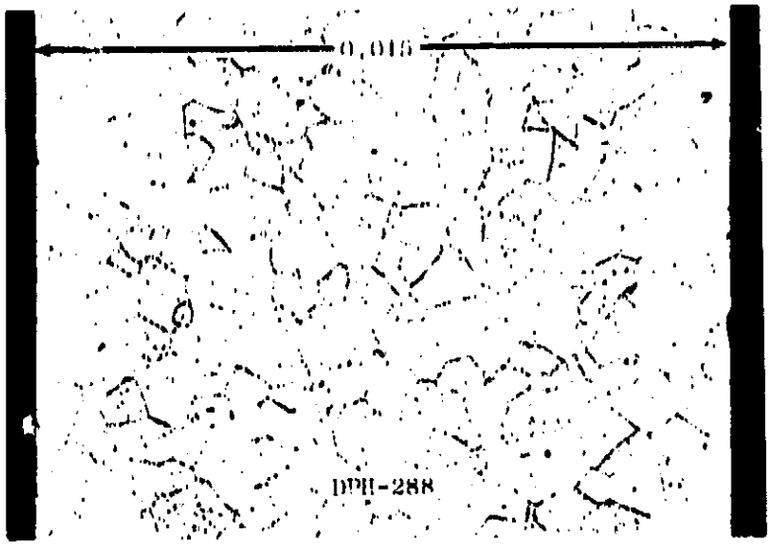
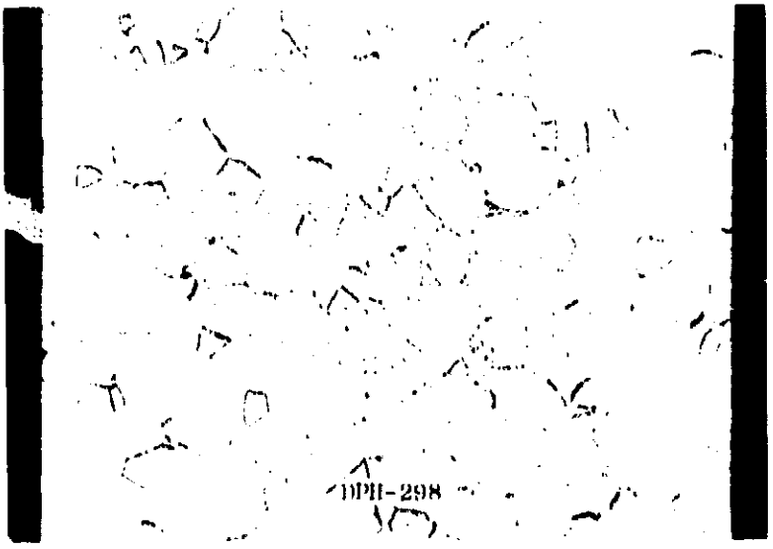


Figure 37. - T-111 alloy sheet specimens, as received and heat treated (3 hrs at 1750° C (3180° F) in 10,000 psig helium plus 3 hours at 1425° C (2600° F) in 200 psig helium) shown before and after exposure to lithium at 1425° C (2600° F) for 100 hours.



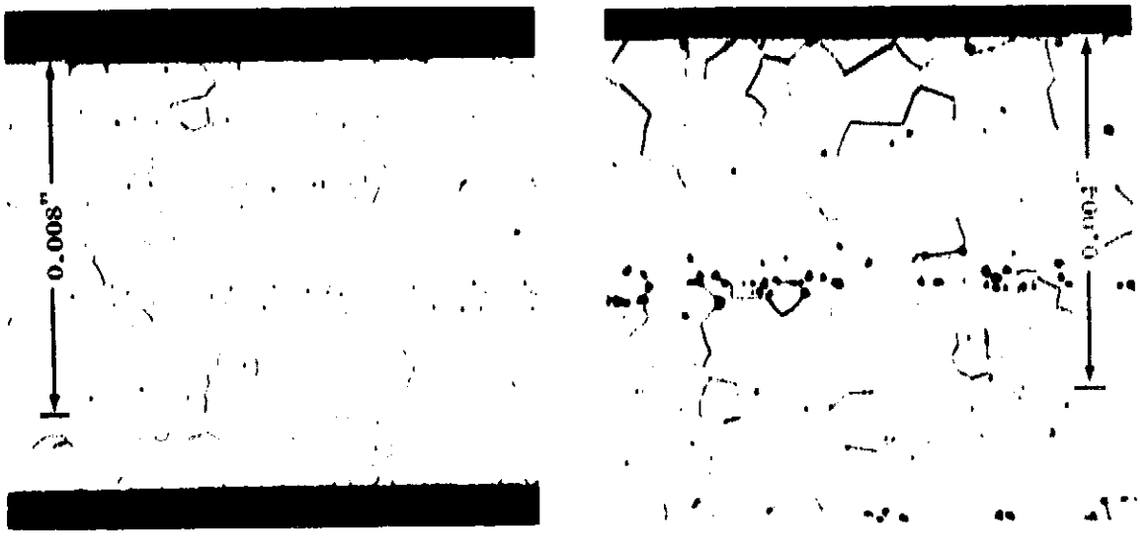
(a) Before Exposure to Lithium
Oxygen Conc. (avg.) = 2440ppm



(b) Following Exposure to Lithium
Oxygen Conc.: 24ppm

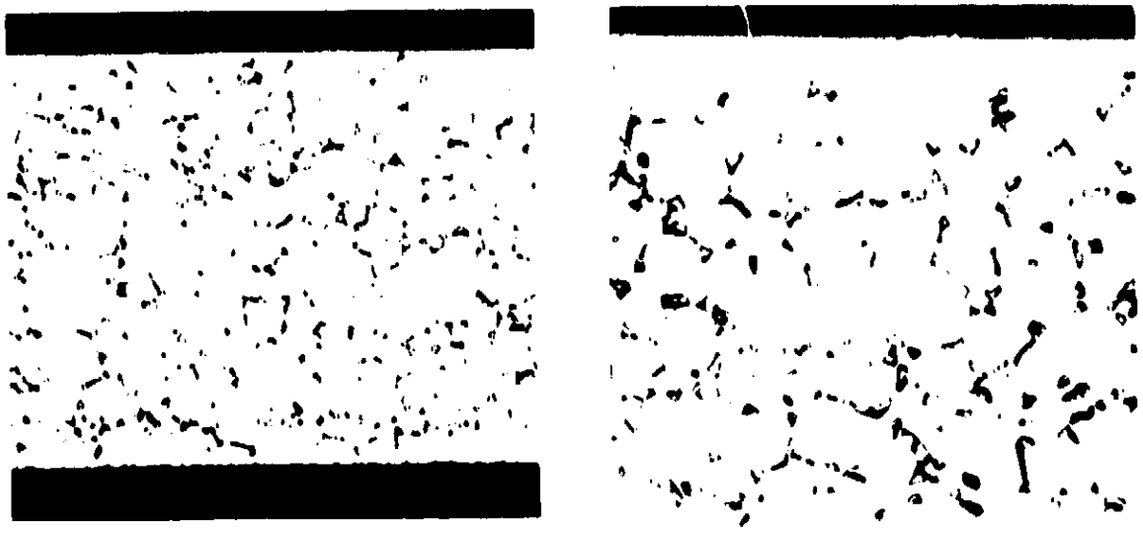
Figure 38. Metallographic Appearance of Heat Treated T-111 Sheet Specimens Before (a) and Following (b) Exposure to Lithium for 100 Hours at 1425° C (2600° F).

Etchant: 30gNH_4F-50mlHNO_3-20mlH_2O (a) F190313 (250X)
(b) F190413 (250X)



250X E970311 500X E970312

(a) Before Exposure to Lithium



250X E970411 500X E970413

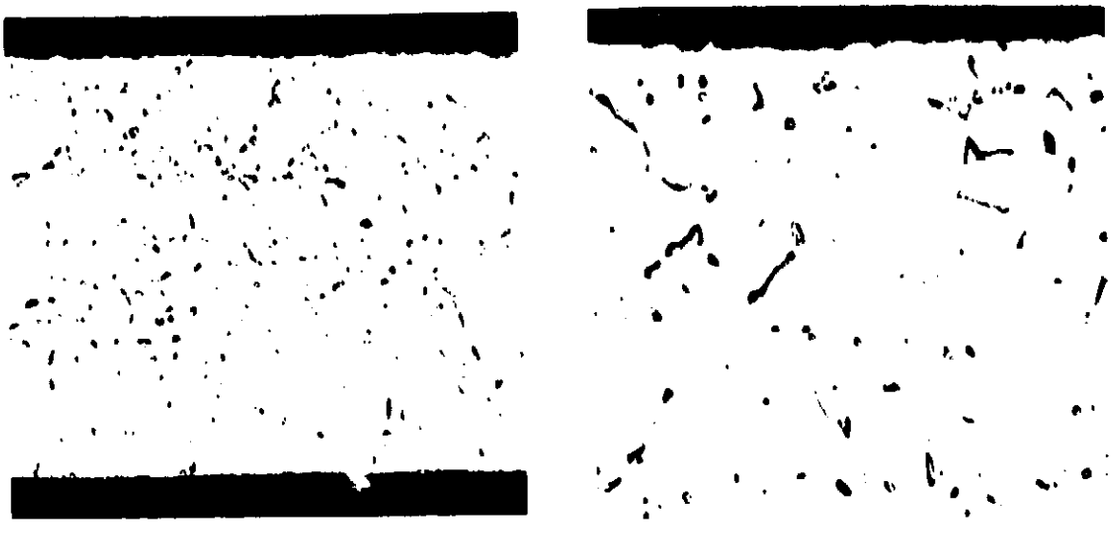
(b) Following Exposure to Lithium

Figure 39. Metallographic Appearance of Heat Treated W-30Re-30Mo(a/o) Powder Product Sheet Before (a) and Following Exposure to Lithium for 100 Hours at 1425° C (2600° F). The Second Phase in the Microstructures is sigma.

Etchant: 20% Murakamis

250X E970711 500X E970712

(a) Before Exposure to Lithium



250X E970811 500X E970812

(b) Following Exposure to Lithium

Figure 40. Metallographic Appearance of Heat Treated W-30Re-30Mo(a/o) Arc Cast Product Sheet Before (a) and Following Exposure to Lithium for 100 Hours at 1425° C (2600° F). The Second Phase in the Microstructures is sigma.

Etchant: 20% Murakamis's

NASA-Lewis-Com'l