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ADVANCED REFRACTORY ALLOY CORROSION LOOP PROGRAM

QUARTERLY PROGRESS REPORT NO. 22

For Quarter Ending October 15, 1970

Prepared by
R. W. Harrison
J. P. Smith

prepared for
NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

NASA Lewis Research Center
Contract NAS 3-6474
R. L. Davies and P. L. Stone, Project Managers
Materials and Structures Division

NUCLEAR SYSTEMS PROGRAMS
SPACE DIVISION
GENERAL ELECTRIC
CINCINNATI, OHIO 45215

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approved by
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NUCLEAR SYSTEMS PROGRAMS
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July 15, 1970 to October 15, 1970

November 12, 1970

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NASA Lewis Research Center
Cleveland, Ohio
R. L. Davies and P. L. Stone, Project Managers
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TABLE OF CONTENTS

LIST OF ILLUSTRATIONS

LIST OF ILLUSTRATIONS (Continued)

LIST OF ILLUSTRATIONS (Continued)

FOREWORD

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

The Program Manager for the General Electric Company is R. W. Harrison. Personnel making major contributions to the program during the current reporting period include:

T-111 Corrosion Loop Posttest Evaluation - J. Smith, A. Losekamp

Metallography - I. Miller

Microprobe - G. Anderson

1900°F Lithium Loop - J. Smith, T. Irwin

Lithium Thermal Convection Loop - G. Brandenburg, A. Losekamp, T. Irwin

ADVANCED REFRACTORY ALLOY CORROSION LOOP PROGRAM

I. INTRODUCTION

This report covers the period from July 15, 1970 to October 15, 1970 of a four-part program, as described below.

A. T-111 Rankine System Corrosion Test Loop

The initial task of this program was to fabricate, operate for 10,000 hours, and evaluate a T-111 Rankine System Corrosion Test Loop. Materials for evaluation include the containment alloy, T-111 (Ta-8W-2Hf) and the turbine candidate materials Mo-TZC and Cb-132M which were located in the turbine simulator of the two-phase potassium circuit of the system. The loop design is similar to the Cb-1Zr Rankine System Corrosion Test Loop; a two-phase, forced convection, potassium corrosion test loop which was tested under Contract NAS 3-2547.(1) Lithium was heated by direct resistance in a primary loop. Heat rejection for condensation in the secondary potassium loop was accomplished by radiation in a high vacuum environment to the water cooled chamber. The compatibility of the selected materials were evaluated at conditions representative of space electric power system operating conditions, namely:

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

This loop completed 10,000 hours of testing in March 1970 and is undergoing evaluation.

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(1) Hoffman, E. E. and Holowach, J., Cb-1Zr Rankine System Corrosion Test Loop, Potassium Corrosion Test Loop Development Topical Report No. 7, NASA-CR-1509, 1970.

B. 1900°F Lithium Loop

Also included in the program is the fabrication, 7500-hour operation, and evaluation of a 1900°F, high flow velocity (1 gpm), pumped lithium loop designed to evaluate the compatibility of T-111 clad uranium nitride fueled specimens, ASTAR 811 type alloys, T-111, Mo-TZM and W-Re-Mo Alloy 256,* at conditions simulating a space power reactor system. This loop completed 2500 hours and underwent a scheduled shutdown. The loop has been placed back on test with two new fuel pins to be tested for 5000 hours additional time at the same conditions.

C. Advanced Tantalum Alloy Capsule Test

The program also included capsule testing to evaluate advanced tantalum alloys of the ASTAR 811 type (Ta-8W-1Re-1Hf) in both potassium and lithium. Refluxing potassium capsule tests at 2200°F and lithium thermal convection capsule tests at 2400°F have completed 5000 hours of testing, and a final report is being written.

D. Lithium Thermal Convection Loop

A new modification has been added to the program to design, fabricate, and operate a natural circulation lithium loop at 2500-2700°F. The loop will be fabricated from T-111 alloy and will contain chemistry, metallography, and creep/tensile specimens of T-111 and advanced tantalum alloys of the ASTAR type.

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*W-25 a/o Re-30 a/o Mo (W-29 w/o Re-18 w/o Mo)

II. SUMMARY

Planned metallographic examination of loop components and turbine simulators from the T-111 Rankine System Corrosion Test Loop is complete. Although some indication of possible minor alkali metal attack was observed, no area of gross corrosion was observed. In general, microstructural changes were readily correlated with changes in interstitial content of the T-111 alloy.

Replacement of the fuel element specimens and other final preparations for the restart of the 1900°F Lithium Loop were completed. The loop was brought back to test conditions on July 31, 1970 and as of October 15, 1970 the loop had logged an additional 1825 hours since the replacement of the fuel specimens for a total accumulated test time of 4338 hours.

The Advanced Refractory Alloy Corrosion Loop program has been modified to include the design, fabrication, instrumentation and evaluation of a T-111 alloy Lithium Thermal Convection Loop Test to evaluate mass transfer of interstitial elements in T-111 and ASTAR alloys.

III. PROGRAM STATUS

A. T-111 Rankine System Corrosion Test Loop

Primary emphasis in this task during the current reporting period was on completing the metallographic examination of selected areas of the loop. Microprobe analysis and microhardness measurements were performed on areas which showed anomalies in microstructure or chemical analysis.

1. Boiler Plug Section

Metallographic specimens were examined from the twenty-one areas shown in Figure 1. The microstructures were compared with pretest microstructures, such as those shown in Figure 2, to delineate any changes that occurred as a result of the test exposure. All specimens were nickel plated prior to mounting to maintain edge retention. T-111 specimens were etched with 30 gr NH_4F-50 ml HNO_3-20 ml H_2O. In addition to the T-111 specimens from the loop, samples were also examined from the nozzles and blades of the first, second, sixth and tenth stage turbine simulators all of which were made from Mo-TZC except the sixth stage which was Cb-132M alloy. As can be seen in Figure 1, over a third of the specimens examined were from the plug section of the boiler since, based on previous experience,(1) this is one of the more critical areas of a loop of this type with respect to changes in microstructure. Particular attention was given to the 3/8-inch diameter potassium containment tube because of the high heat flux across the wall of this tube and the fact that visual examination of the lithium exposed side showed the lower six inches to be coated with HfN.(2) A longitudinal section was cut from the bottom of the 3/8-inch tube where it was welded to the fitting; also transverse sections were taken about six inches above the fitting and from two areas near the top of the swirler wire insert. The HfN coating was best examined in the as-polished condition, as shown in Figure 3, since the etchant used for T-111 attacked

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(2) Advanced Refractory Alloy Corrosion Loop Program Quarterly Progress Report No. 21 for period ending July 15, 1970. NASA-CR-72782 (GESP-546)

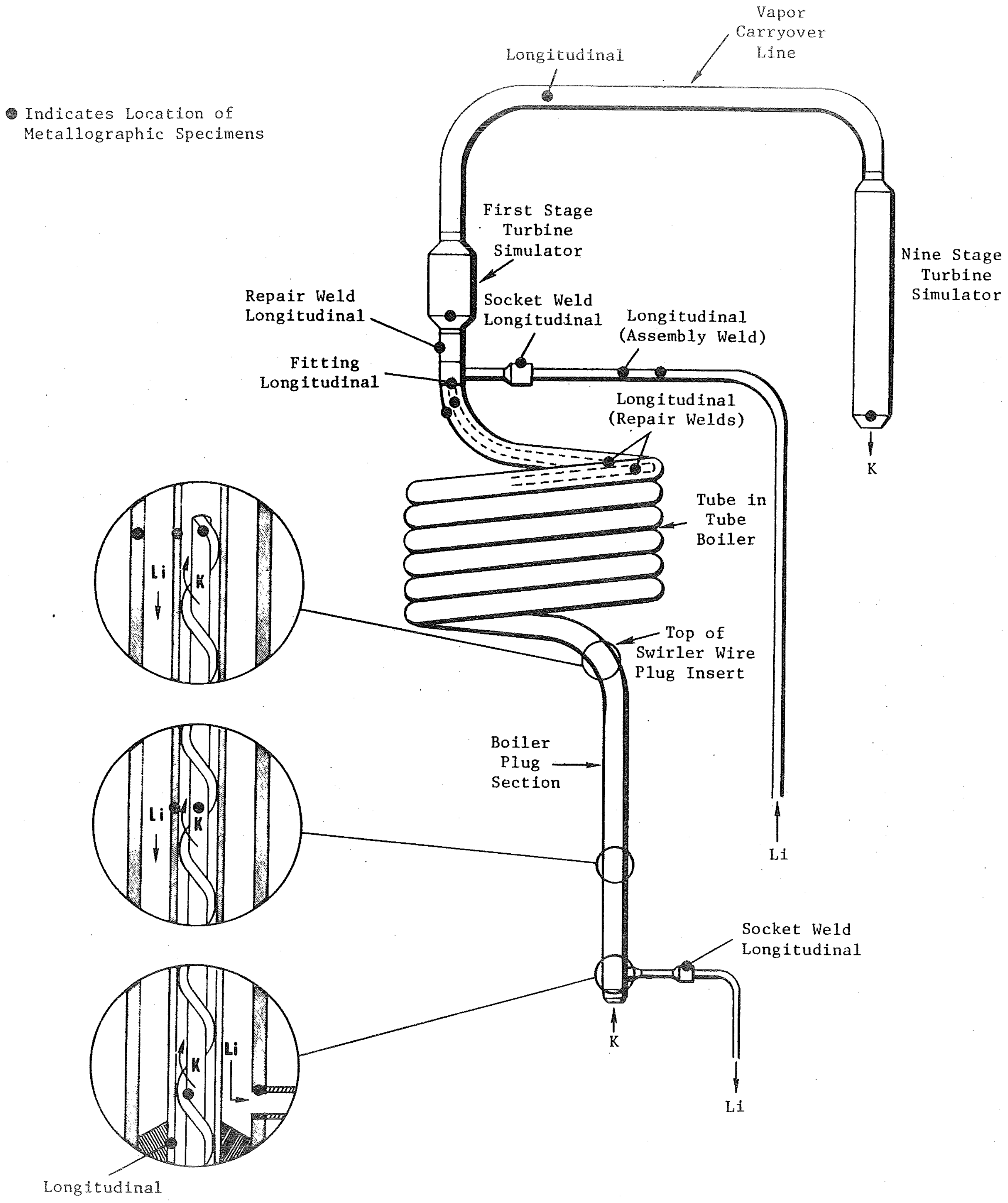
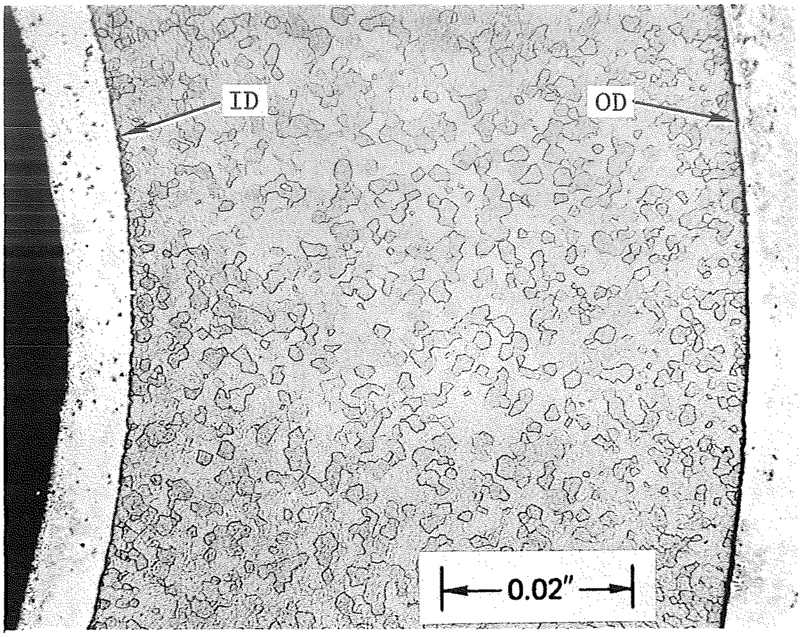
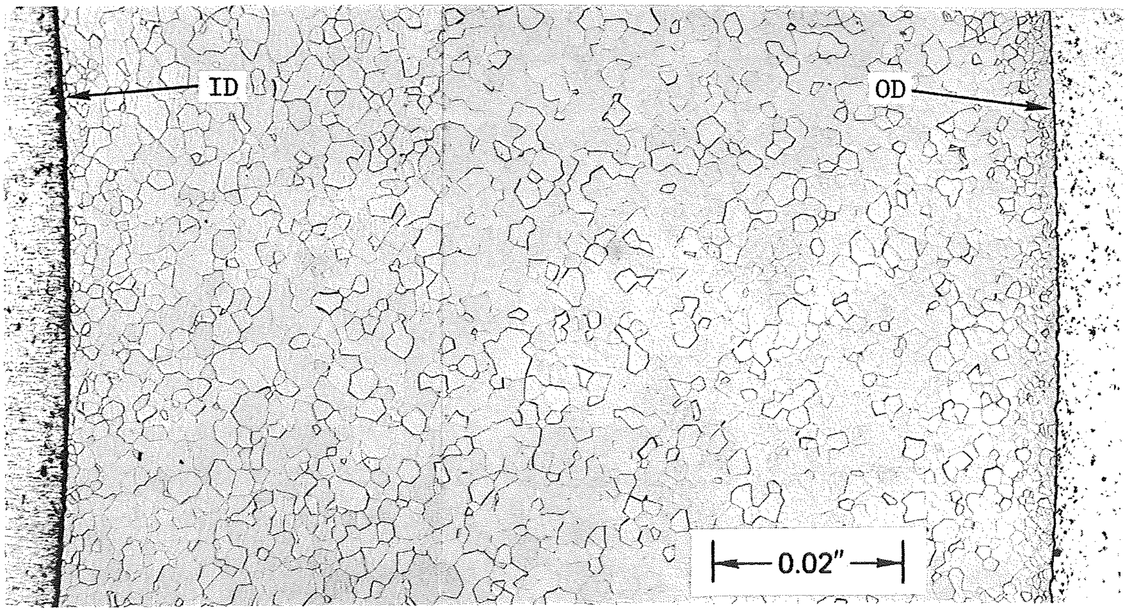


Figure 1. Location of Specimens for Metallographic Examination from the T-111 Rankine System Corrosion Test Loop Following 10,000 Hours of Continuous Operation. (All are Transverse Sections Unless Noted Otherwise.)



H67031B Etched

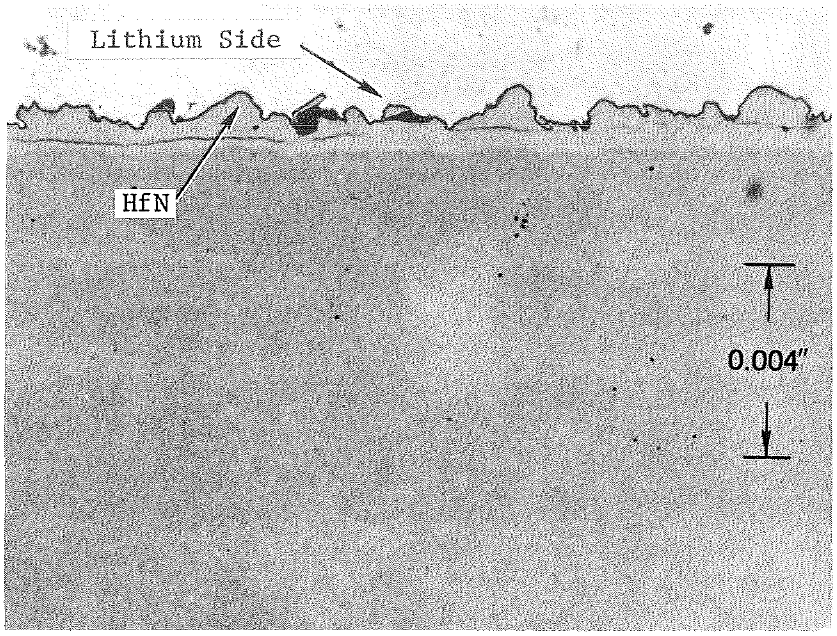
a. 3/8-inch OD tubing



H67011A,B Etched

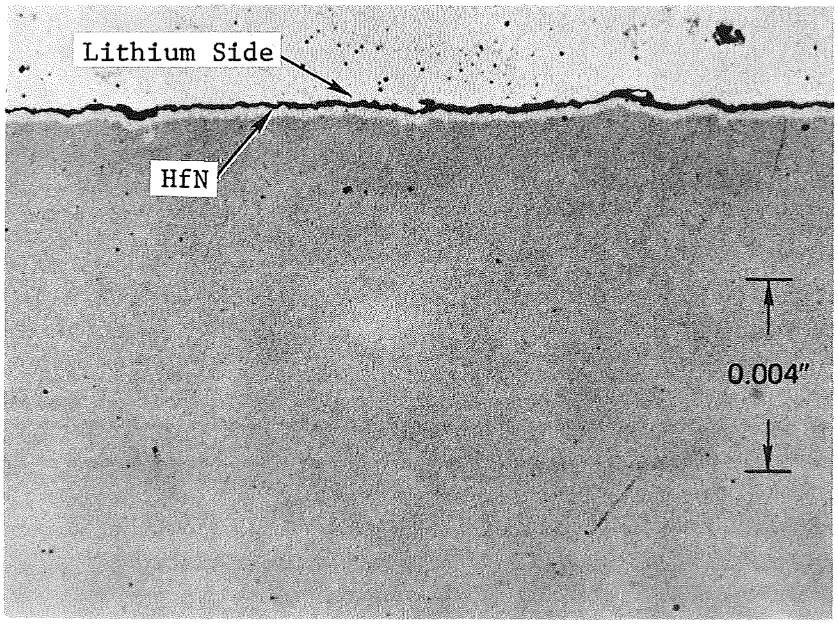
b. 1-inch OD tubing

Figure 2. Pretest Microstructure of T-111 Tubing Used for T-111 Rankine System Corrosion Test Loop.



H64011E As-Polished

a. On weld metal at tube-fitting interface



H64011H As-Polished

b. On tube approximately 1/2-inch above fitting

Figure 3. Hafnium Nitride Deposit Near Bottom of 3/8-inch OD T-111 Tube in Plug Section of Boiler.

the HfN rather rapidly. The major difference between the microstructure shown in Figure 3a, which is taken from the weld area, and that shown in Figure 3b, which represent the tube OD surface about 1/2 inch above the weld, is the thickness of the HfN coating. The HfN in the weld region, which operated at the lowest temperature in the plug, is significantly thicker than the coating on the tube and contains cracks which were not observed in the thinner coating on the tube. It is significant that the cracks were in the HfN coating and not at the HfN-T-111 interface. Examination of the ID (potassium) surface of this specimen indicated anomalous microstructural features at the fitting - weld metal and tube-weld metal interfaces. The microstructure shown in Figure 4, is from the fitting-weld metal interface, and is typical of these anomalous microstructural features. In the as-polished condition they appear as a light and dark island within the normally gray colored T-111 matrix; however after etching, the affected area is seen to be much larger than indicated in the as polished condition, and generally contains a relatively heavily etched (black) core. Note also the evidence of grain boundary attack on the periphery which does not show up in the as-polished condition. Whether this grain boundary structure results from potassium attack or reaction of material in the grain boundaries with the etchant is difficult to ascertain. Microprobe analysis of this area for Ta, Hf, W, K, Fe, and Cr showed the area to be rich in Fe and Cr as illustrated by the scans in Figure 5: no other elements were detected. Although the source of the Fe and Cr cannot be readily explained, their presence in the potassium circuit is undoubtedly related to the previous findings of Fe, Cr, and Ni in the residue from the distillation of the potassium drained from the loop.(3) Presumably the original source is inadvertent contamination by stainless steel; however, the exact source and the reason for the particularly high concentration in this one weld area are currently unknown. It should be pointed out that all other areas where microprobe evaluation was performed were also checked for Fe and Cr; however, no indications were found. Microhardness traverses in the vicinity of these areas showed them to have a hardness of 200-225 units higher than the average matrix

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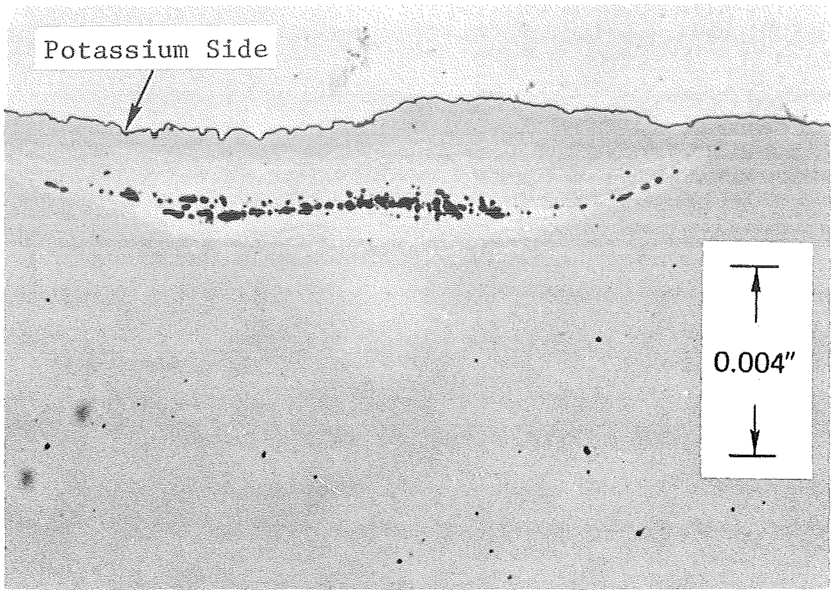
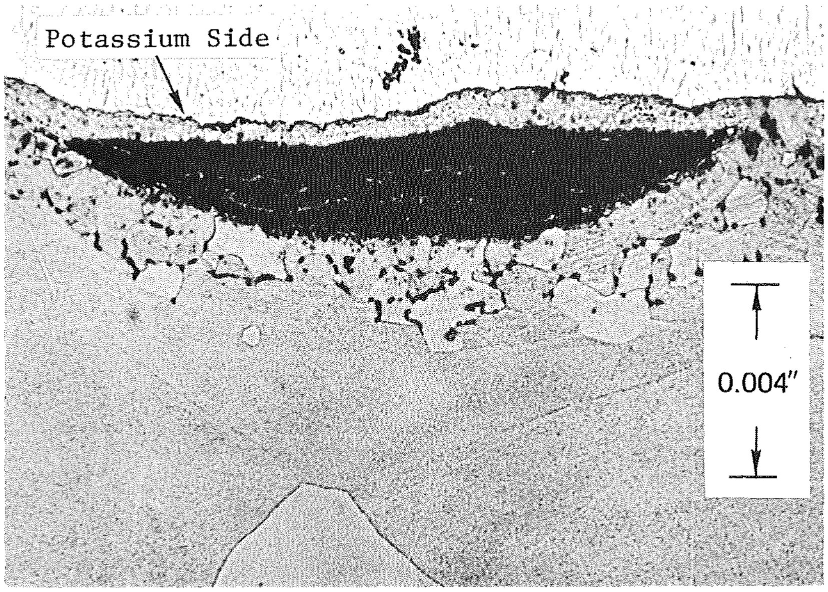
(3) Advanced Refractory Alloy Corrosion Loop Program Quarterly Progress Report No. 20 for period ending April 15, 1970. NASA-CR-72739 (GESP-491).

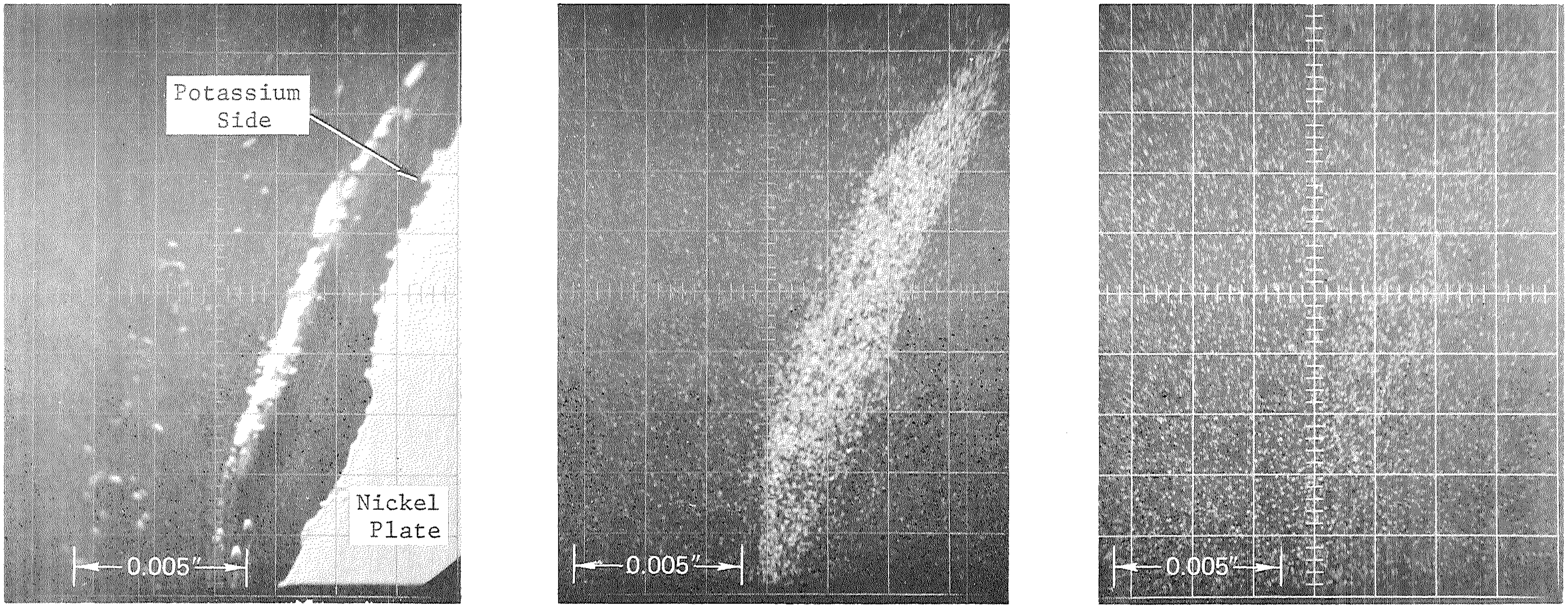


H64011A As-Polished



H64011K Etched

Figure 4. Base Metal-Weld Interface at Bottom of the 3/8-inch OD T-111 Potassium Containment Tube in the Plug Section of the Boiler Before and After Etching.



a. Sample current image

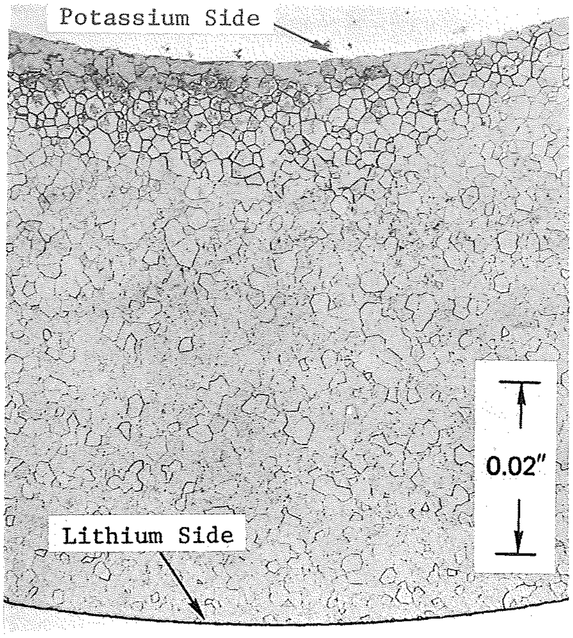
b. Iron X-ray image

c. Chromium X-ray image

Figure 5. Microprobe Scans of the Base Metal-Weld Interface at the Bottom of the 3/8-inch OD T-111 Potassium Containment Tube in the Plug Section of the Boiler. Same Area as Figure 4.

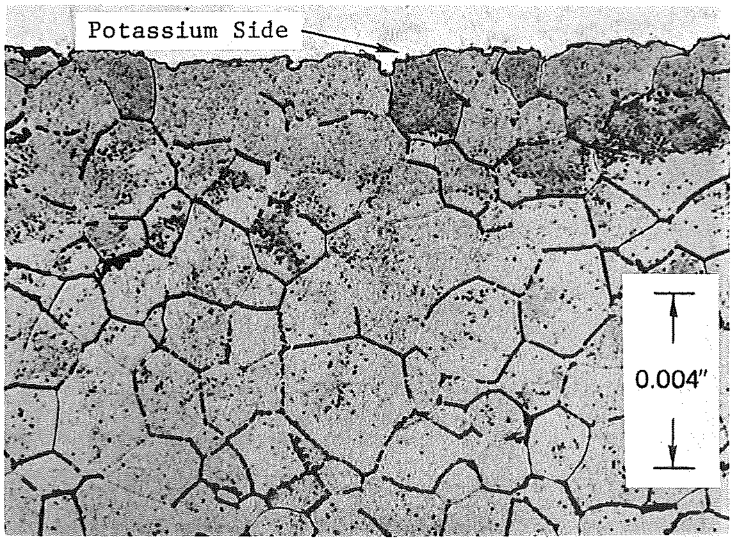
hardness of about 225 DPH (100 grams load). A transverse specimen from the 3/8-inch tube was cut from an area near the edge of the HfN coated region which occurred six inches above the lithium exit. This region of the tube is believed to correspond to the area where the ID of the tube begins to be exposed to vapor approaching 100% quality. This is somewhat supported by the fact that the oxygen concentration at the ID below the interface was 16 ppm, but just above the interface 948 ppm was found.(3) Some evidence of localized corrosion was evident in this area as seen in Figure 6. The corrosion area was localized, i.e., it did not extend around the total ID surface but was essentially confined to the area shown in Figure 6. This localized effect is believed to be associated with the swirling of the flow path and probably corresponds to transformation of high oxygen liquid to pure potassium vapor in this local area. Microhardness measurements showed no difference or gradients among various locations in the specimen.

Two additional specimens were obtained from the region near the top of the plug section. The first section to be examined was cut essentially even with the top of the swirler wire insert and consisted of a clean equiaxed microstructure as shown in Figure 7. Since the chemical analysis in this region of the tube had indicated both high oxygen concentration (1100 ppm) and very steep oxygen gradient it was decided that an additional specimen should be taken from an area closer to the higher oxygen specimen (~ 1/2-inch above the end of the swirler insert). The microstructure of this specimen, shown in Figure 8 varies from that of the lower specimen, Figure 7, and is probably more typical of the high oxygen area. The reason for the high oxygen was discussed in the previous report.(2) It can be seen in Figure 8 that the reaction zone at the tube ID is not uniform in thickness which could be a result of the swirling action of the potassium being ejected from the end of the swirler wire insert. The reaction zone consists of three different regions as shown in Figure 9. The region closest to the ID, shown in Figure 9a, consists primarily of a very fine evenly divided precipitate in the matrix and a slightly coarser grain boundary precipitate. Near the mid-wall, shown in Figure 9c, the microstructure is dominated by a relatively large grain boundary precipitate and somewhat fewer in number but equally large matrix precipitate. Between these two regions, shown



H64021A Etched

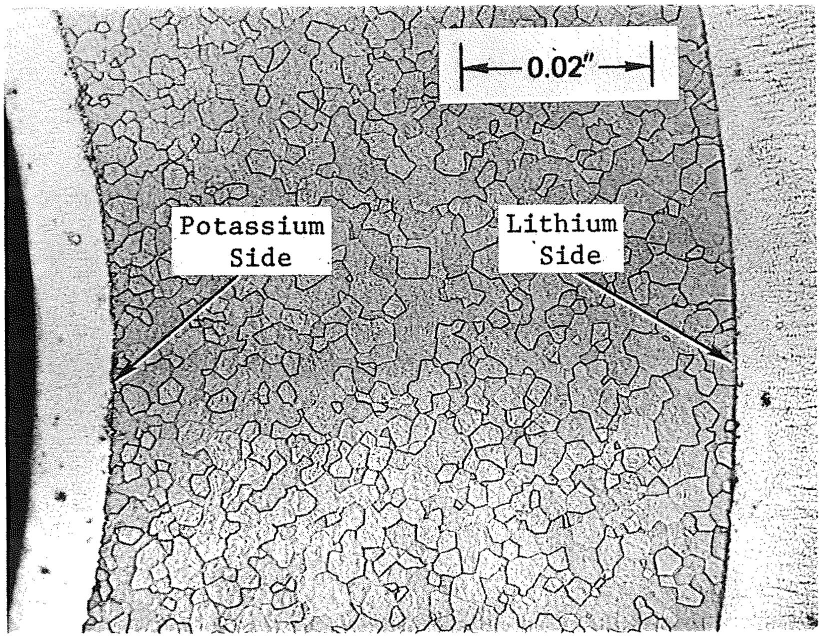
a. Total wall



H64021D Etched

b. Inner wall

Figure 6. T-111 Potassium Containment Tube to the Plug Section Approximately Six-Inches Above Lithium Exit from Boiler.



H64031A Etched

Figure 7. T-111 Potassium Containment Tube in the Plug Section of the Boiler Adjacent to the Top of the Swirler Wire Insert.

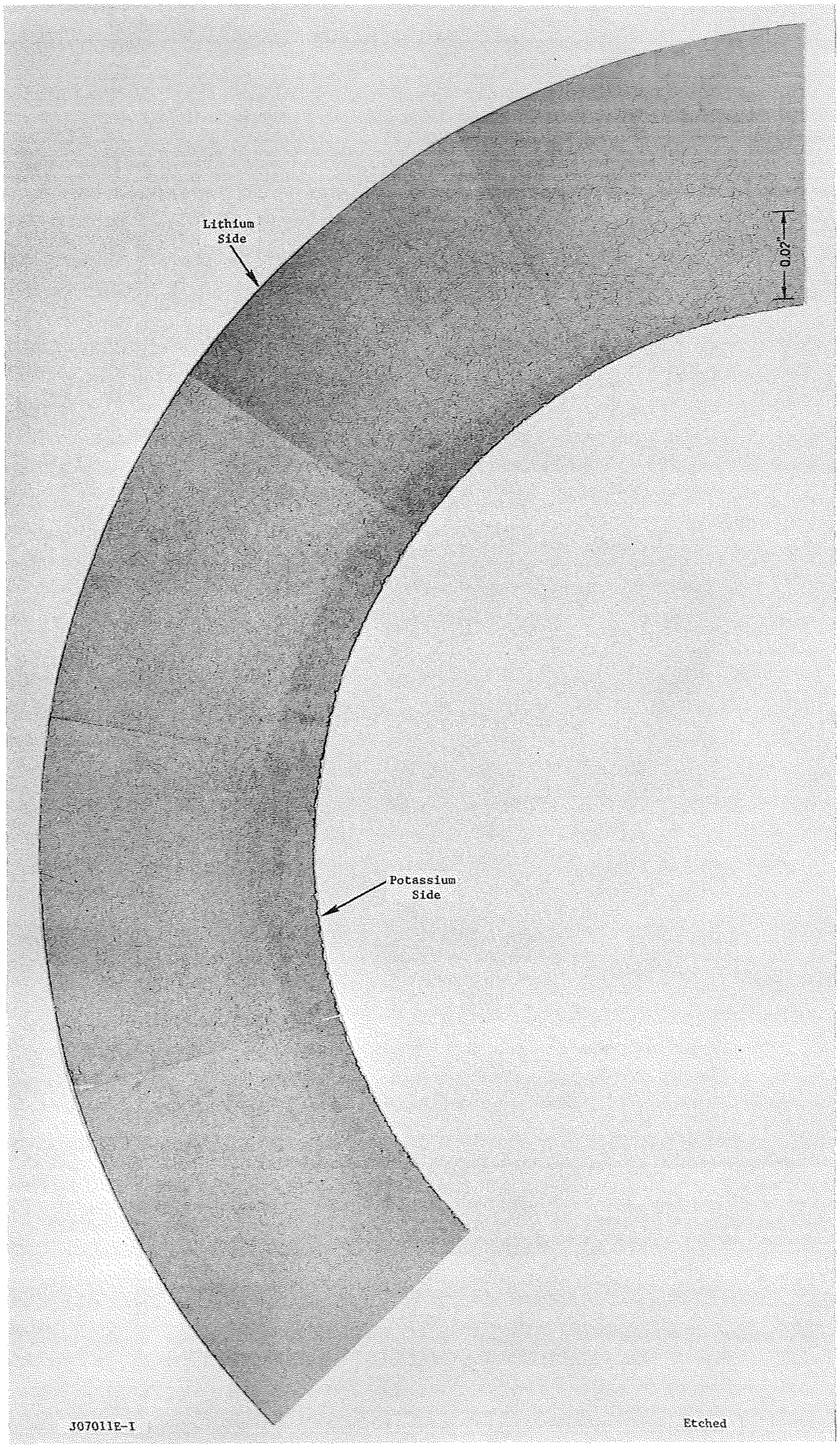
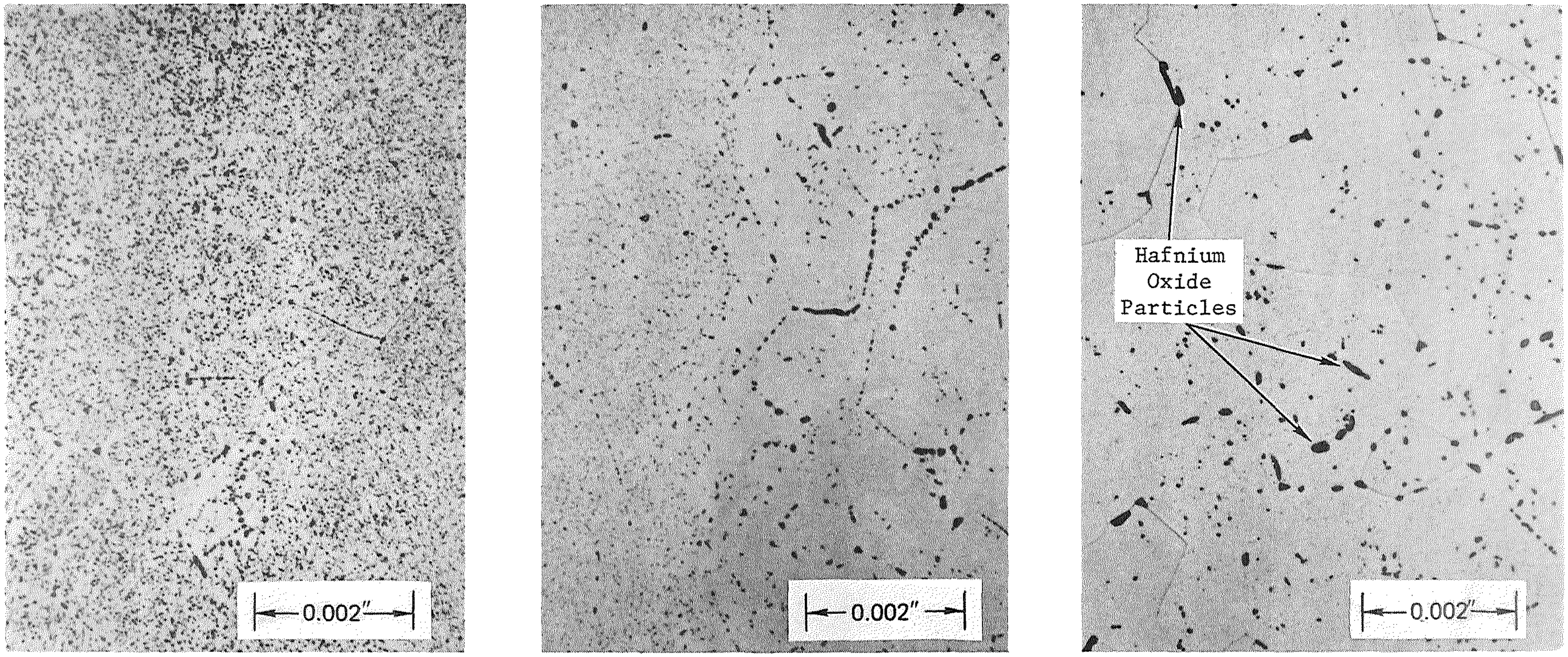


Figure 8. 3/8-Inch OD Potassium Containment Tube Approximately One-Half Inch Above Top of Swirler Wire Insert.



J07011N Etched

a. Near potassium side

J07011C Etched

b. Approximately 0.015 inch from potassium side

J07011M Etched

c. Approximately 0.030 inch from potassium side - near mid-wall

Figure 9. T-111 Potassium Containment Tube Approximately 1/2-inch Above the Top of the Swirler Wire Insert. Same Area as Figure 8.

in Figure 9b there is a transition region consisting primarily of a fine matrix precipitate and an area immediately adjacent to it of large grain boundary precipitates but clean matrix. Microprobe analyses were performed on this specimen to determine the identity of the precipitate particles. Although the identity of the fine particles could not be identified, because the size is below the resolution limit of the microprobe, the larger particles are believed to be hafnium oxide because (1) the hafnium content in the region of the larger particles was approximately four times that of the matrix and (2) all of the larger particles fluoresced in the electron beam.

Specimens of the swirler wire insert at locations near the bottom, six-inches from the lithium exit, and the top were also examined. Typical areas from the top and bottom of the swirler wire are shown in Figure 10. Microprobe analyses were performed in an attempt to identify the dark phase. Results from the top sample showed the hafnium content of some of the particles to be three times that of the matrix; however, results of analyses of the bottom specimen are not complete as yet. The specimen from six inches above the lithium exit represents a specimen adjacent to the specimen from the 3/8-inch tube shown in Figure 6. As seen in Figure 11, a similar effect was observed in the swirl wire as was seen in the tube section, i.e., a localized area of apparent corrosion. To determine if this effect was associated with the swirling action as proposed earlier, the specimen was reground to determine if the corrosion area appeared in a different location. On re-etching it was found that indeed the area had rotated significantly from the original location supporting the swirling effect. Microhardness traverses in several areas of these specimens showed no significant deviation from the nominal matrix hardness.

Specimens were also examined from the 1-inch tube at the top and bottom of the plug section. No differences could be seen from the pretest material except perhaps a very slight amount of grain growth as shown in Figure 12 (compare with Figure 2).

2. Boiler

Two areas were examined from the coiled portion of the tube in tube boiler: (a) the repair weld region(4) and (b) sections from the top of the

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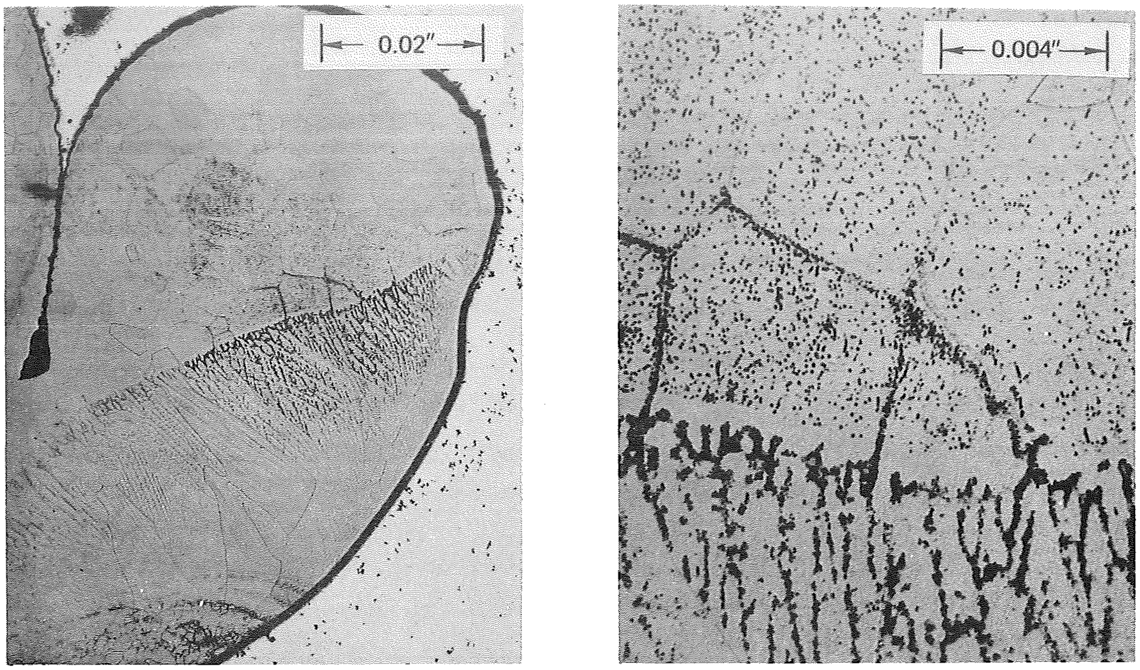
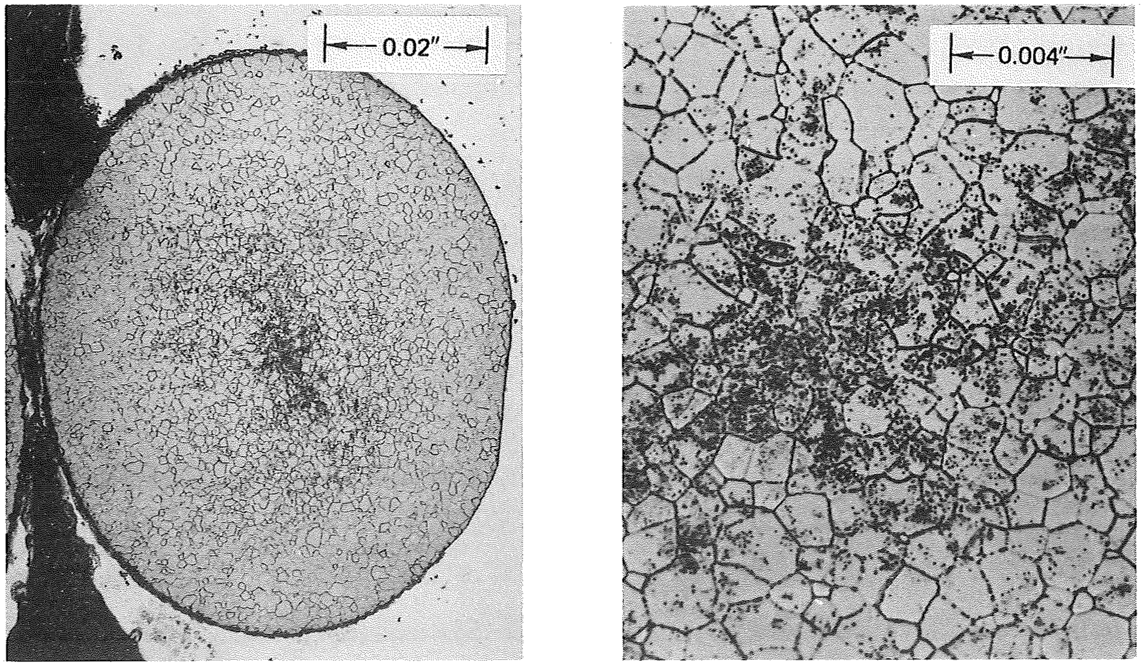
(4) Advanced Refractory Alloy Corrosion Loop Program Quarterly Progress Report No. 14 for period ending October 15, 1968, NASA-CR-72505 (GESP-189).



H64081A Etched H64081D Etched

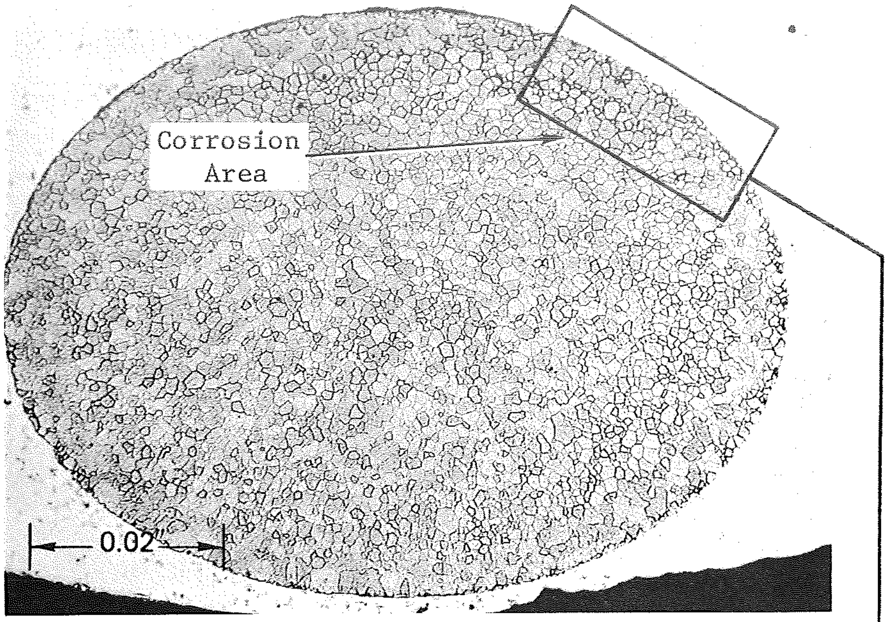
a. Near bottom - potassium inlet showing tack weld joining wire and centerbody



H64061A Etched H64061B Etched

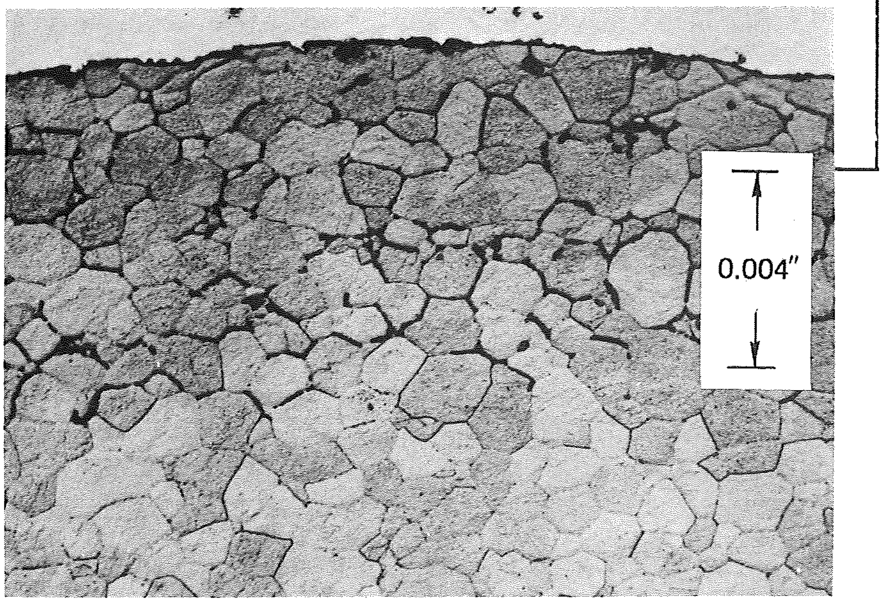
b. Near top of insert

Figure 10. T-111 Swirler Wire from Plug Section of Boiler.



H64071A Etched

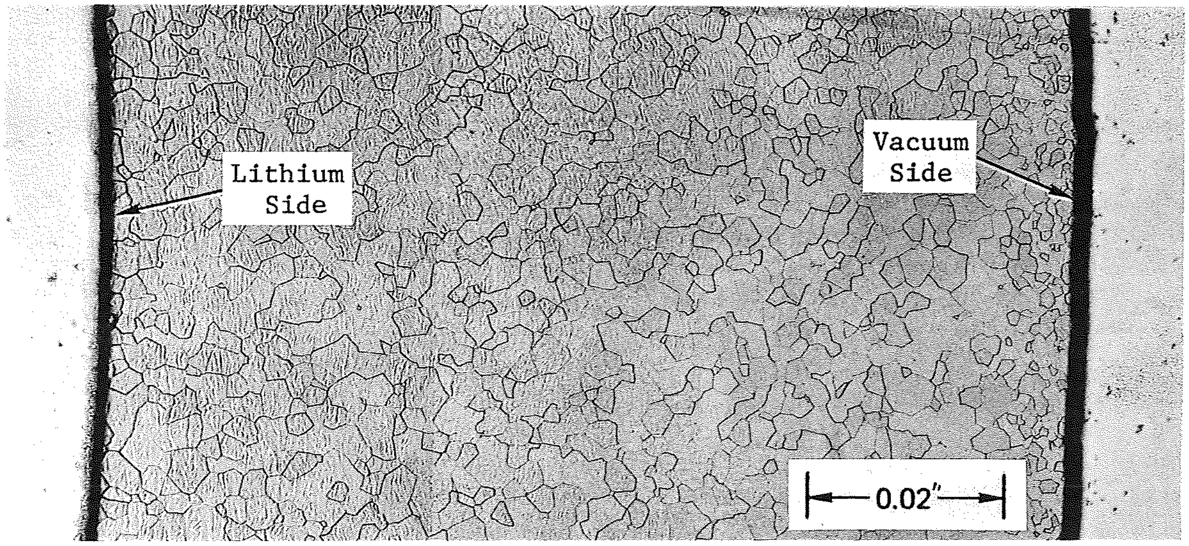
a. Overall



H64071B Etched

b. Corrosion area

Figure 11. T-111 Swirler Wire From Plug Section of the Boiler Approximately 6 Inches From the Lithium Exit.



H64101A,B Etched

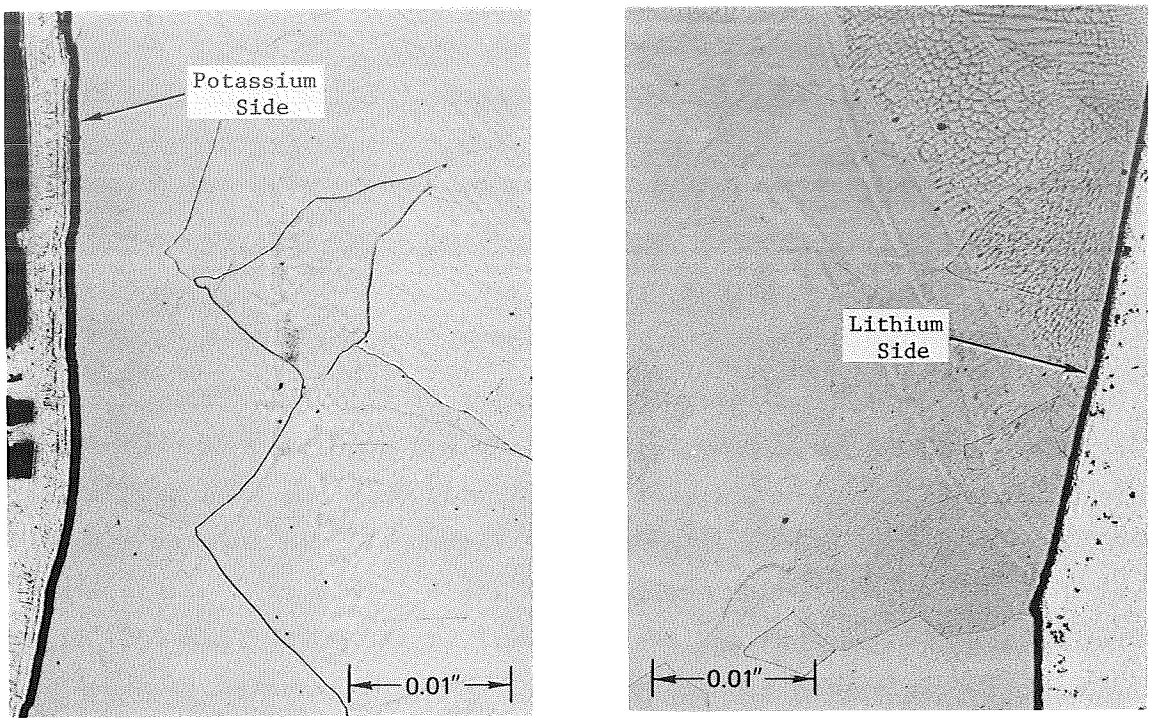
Figure 12. Lithium Containment Tube (1-inch OD) from Top of Plug Section.

boiler which were representative of the hottest (2240°F) part of the boiler. The repair welds on both the 3/8 and 1-inch tubing appeared to be in excellent condition as shown in Figure 13. The areas shown represent the weld metal-base metal interface, which past experience has shown to be the most critical from the standpoint of alkali metal compatibility, and, as clearly seen, no evidence of corrosion is evident.

The specimens from the hottest portion of the boiler showed evidence of strain induced grain growth in both the 3/8 and 1-inch tubes. These specimens were taken from a section of tubing that had been bent almost 90° during pretest forming. As seen in Figures 14 and 15, strain induced grain growth occurred completely across the 0.1-inch wall of the 1-inch lithium containment tube and over one-half of the 0.060-inch wall of the 3/8-inch potassium containment tube. As would be expected, maximum grain growth occurred 90° from the neutral axis in both cases. In between the maximum and minimum stress axis, the microstructure (not shown) consisted of mixed fine and coarse grains.

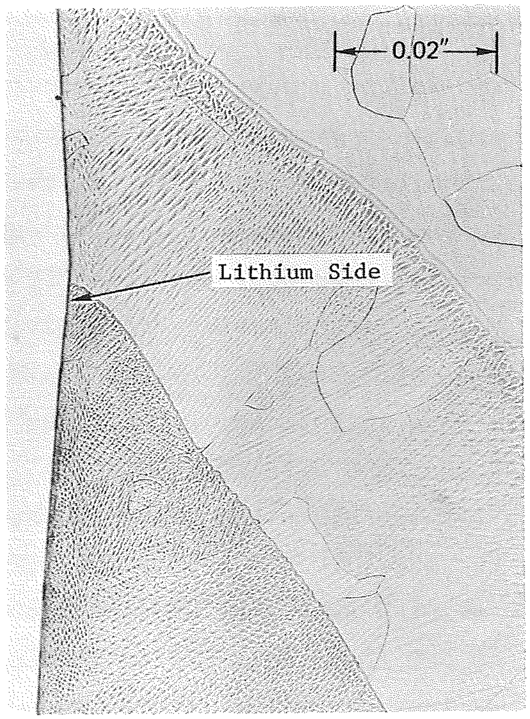
3. Field Repair Welds

In addition to the boiler repair welds discussed above, which were made essentially according to standard weld practice, four field welds were necessary to reinstall the boiler into the test loop.(4) These were made according to the same existing welding specifications for refractory alloys; however, they were performed in a portable weld chamber attached to the 48-inch diameter vacuum chamber spool piece of the test chamber. Since there was a possibility of certain unknown variables which could have influenced the weld quality, three of the four field welds were examined metallographically. Two of these were socket welds, in the lithium circuit. The weld exposed to the hottest lithium (2250°F) at the inlet to the boiler (heater exit) is shown in Figure 16, and it is readily seen that no detrimental effects were detected due to the 10,000 hours exposure to flowing lithium. The second socket weld was located on the lithium return line (2090°F) and, although no indications of typical alkali metal attack were observed, a very fine, somewhat banded, general precipitate was present as shown in Figure 17. This same type of structure was also seen to a somewhat lesser degree at the bottom of the plug section (photos not shown)



H64041E Etched H64041C Etched

a. 3/8-inch OD potassium containment tube



H64121A Etched

b. 1-inch OD lithium containment tube

Figure 13. Repair Welds in Top Coil of the 3/8- and 1-inch T-111 Tubing from the Boiler.

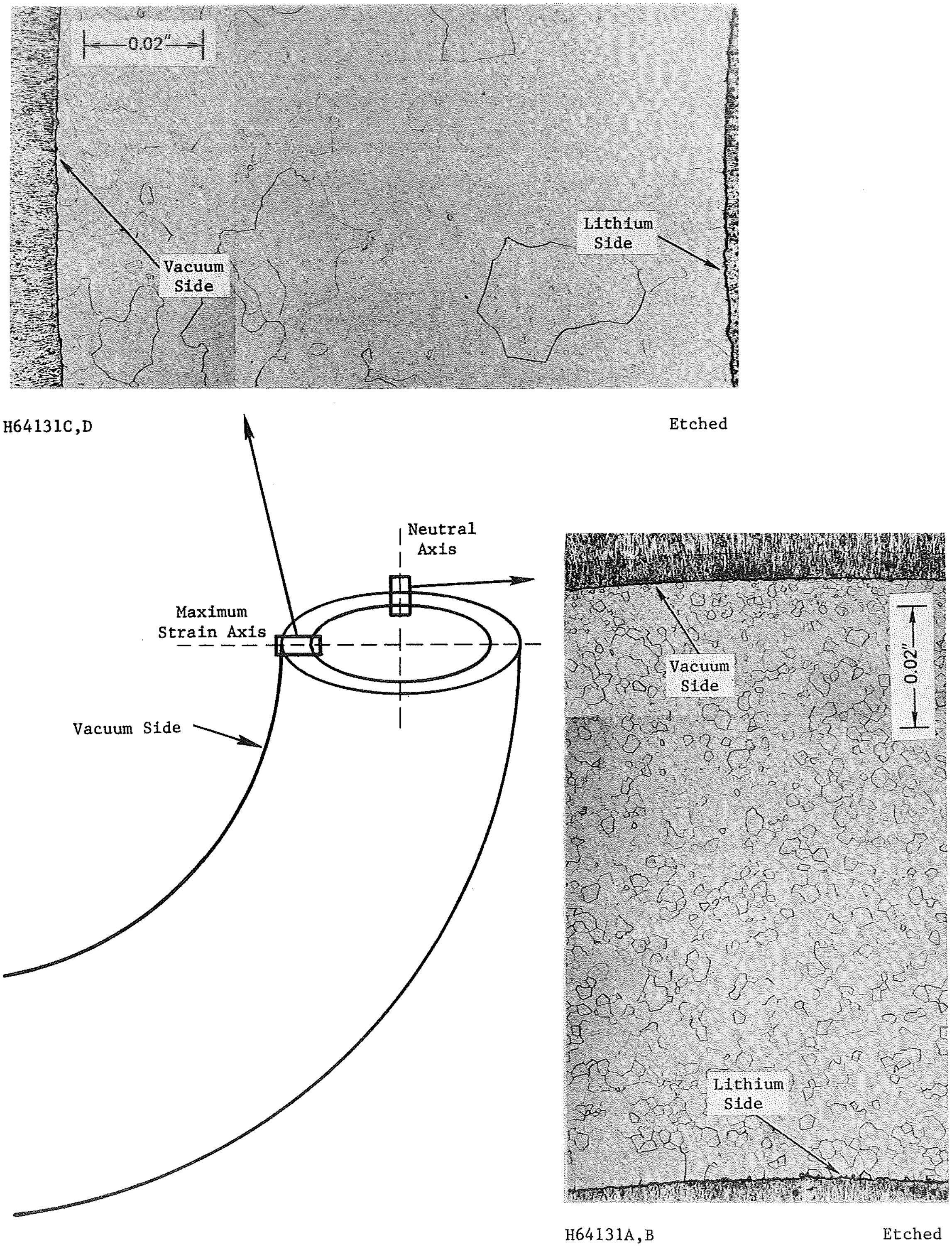


Figure 14. 1-inch OD Lithium Containment Tube from Top of Boiler Following 10,000 Hours of Continuous Operation at Approximately 2240°F Illustrating Effect of Strain from Pretest Forming Operation on Grain Growth.

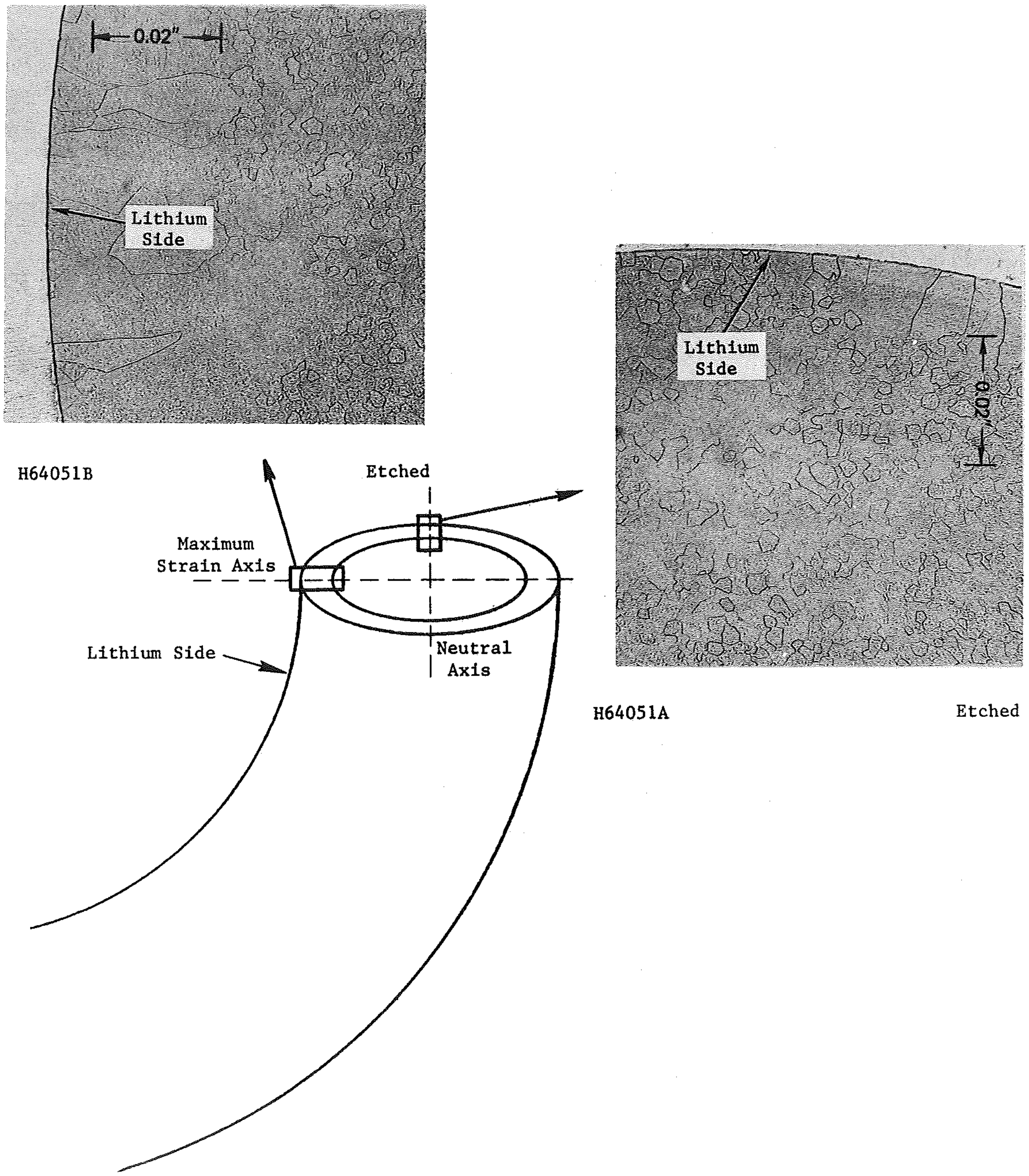
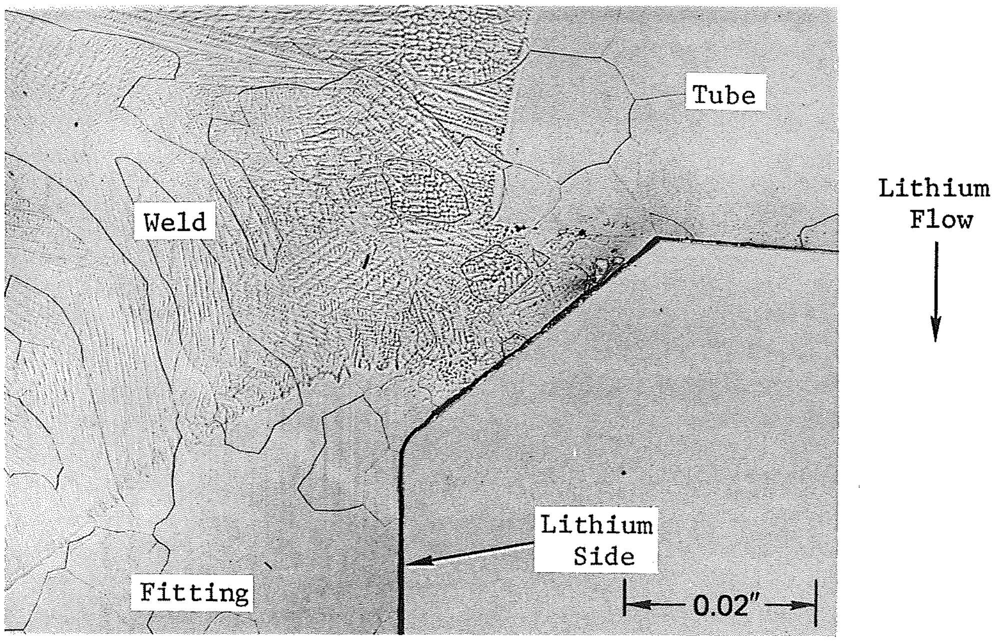


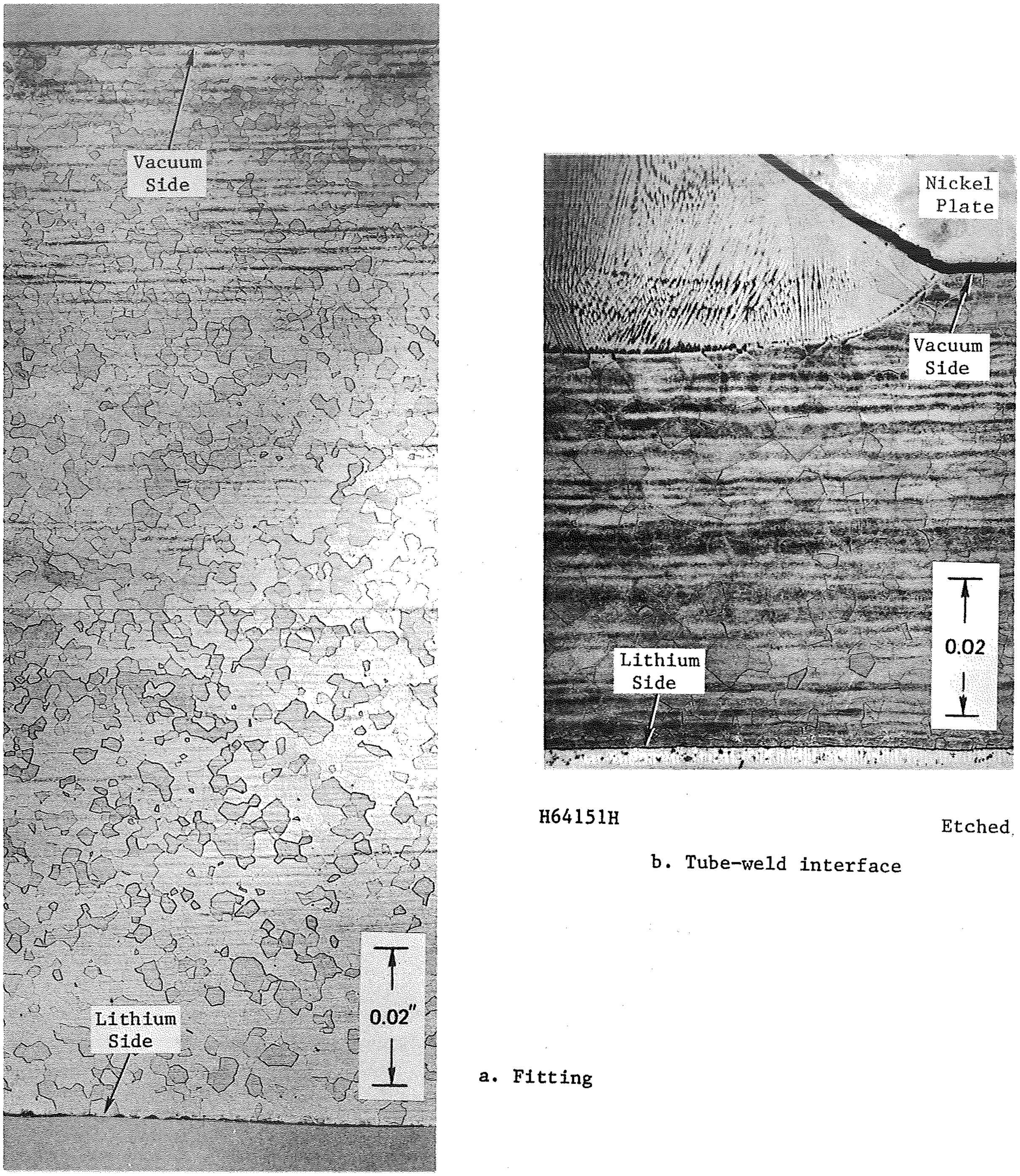
Figure 15. 3/8-inch Potassium Containment Tube from Top of Boiler Illustrating Effect of Strain from Pretest Forming on Grain Growth During the 10,000 Hour Test. Adjacent to Sample in Figure 14.



H64141D

Etched

Figure 16. Repair Socket Weld in 3/8-inch Lithium Inlet to the Boiler.



H64151H

Etched

b. Tube-weld interface

a. Fitting

H64151 I,J

Etched

Figure 17. Repair Socket Weld in 3/8-inch Lithium Exit Line from Boiler.

discussed earlier in this report and also in the T-111 portion of the High Temperature Alkali Metal Valve Loop.(5) Note in Figure 17 that although the precipitate extends completely through the 0.060-inch wall of the 3/8-inch OD tube, only about 1/3 of the outer portion of the fitting wall shows the precipitate. As shown in the higher magnification photomicrographs of Figure 18, the precipitate appears as small individual globular type precipitate in both the matrix and grain boundaries. Since this precipitate does not appear to adversely affect the reliability of the alloy in an alkali metal system in any obvious manner, no attempts have been made to identify the second phase since it is beyond the scope of the existing program. However, if necessary, electron microscope (extraction and transmission) techniques could undoubtedly be developed to identify the composition and origin of this phase. The random locations of this fine precipitate in tested T-111 components suggests that its presence is related to compositional inhomogeneities and thermal effects rather than liquid metal interactions.

Of all the specimens examined the only area which showed typical intra-as well as inter-granular alkali metal attack was in the heat affected zone of the repair weld joining the boiler exit to the first stage turbine simulator housing inlet. This area was relatively small and maximum grain boundary penetration was about 0.005 inch as shown in Figure 19. Note that there appears to be a very fine reaction layer ahead of the gross grain boundary attack as indicated by the shading of Figure 19. This same type of reaction layer can also be seen in the corrosion area of the 3/8-inch tube from the plug section previously shown in Figure 6.

4. Miscellaneous Loop Components

A section from the 3/8-inch line between the lithium heater and boiler inlet was examined since it represents material exposed to the highest loop temperature (2250°F). This material exhibited the

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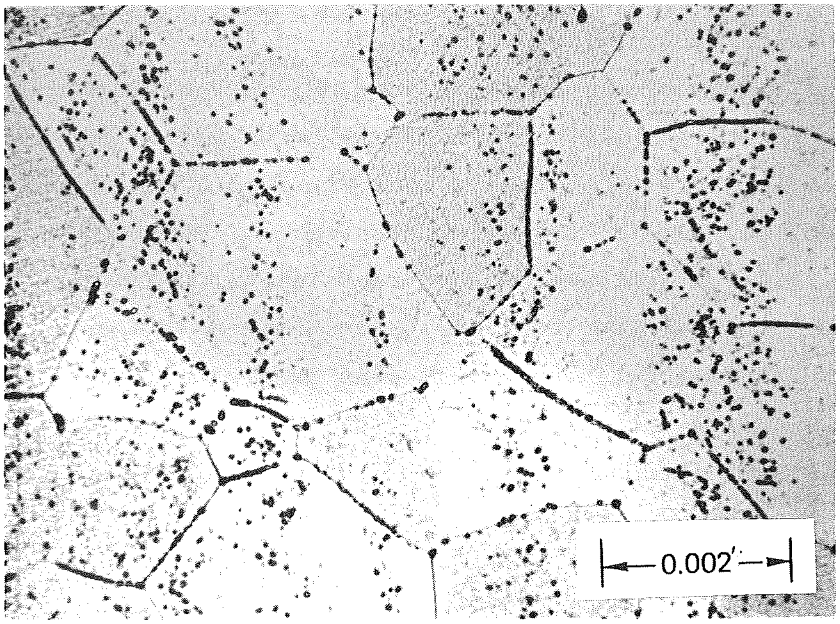
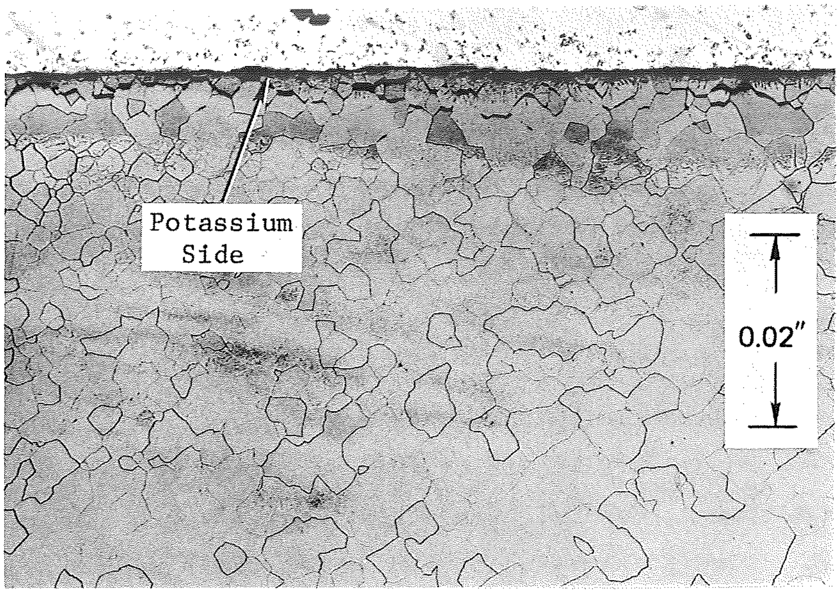
(5) Harrison, R. W., and Holowach, J., Final Report, High Temperature Alkali Metal Valve Test Program, April 15, 1970, NASA Contract NAS 3-8514, Report No. GESP-508.

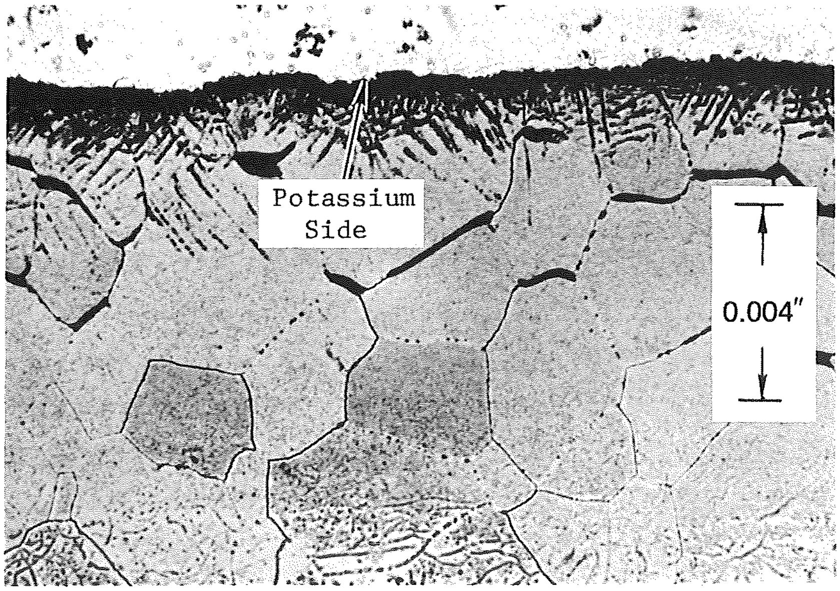


H64151K Etched

Figure 18. 3/8-inch Tube at Repair Socket Weld in Lithium Exit Line from Boiler. Same Area as Figure 17.



H64161D Etched



H64161E Etched

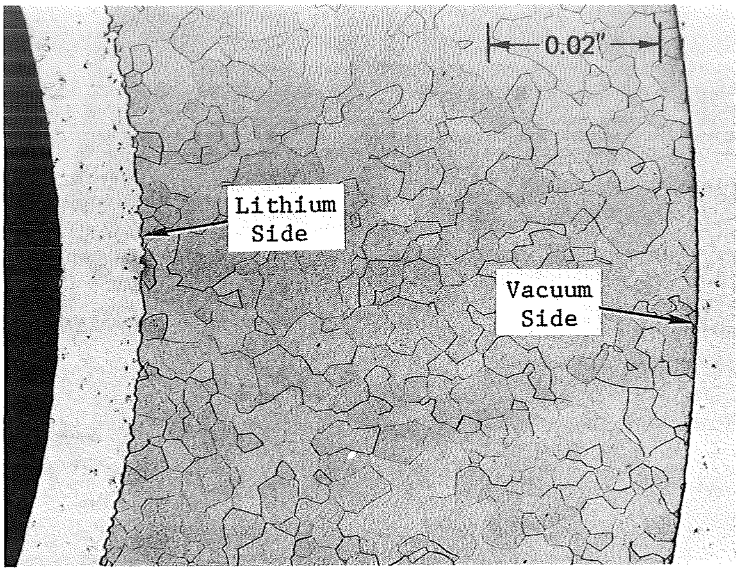
Figure 19. Heat Affected Zone of Field Weld Joining Top of Boiler to Bottom of First Stage Turbine Simulator.

cleanest microstructure of any the tubing examined as shown in Figure 20. Also as seen in Figure 20 the grain size is approximately three times that of the pretest material shown in Figure 2a. Also, note that this is still considerably less grain growth than the strain induced growth at the top of the boiler (Figures 14 and 15).

The vapor crossover line was examined because bend tests showed this material to be the most brittle in the loop. As shown in Figure 21, compressing of ring segments from the crossover line caused fracture with little or no deformation compared to similar tests on specimens of pretest material or from the two locations within the boiler. Although some cracks were observed in the boiler specimens shown in Figure 21 and a specimen from the 1800°F region of the nine-stage turbine simulator housing (not shown), the cracks occurred only after considerable deformation and at a slow propagation rate. On the other hand, the crossover line cracked with little or no deformation and the crack propagated across the wall instantaneously. Optical microscopy of a specimen from the vapor crossover line showed the presence of a fine, grain boundary precipitate throughout the cross section as shown in Figure 22. Because of the extreme brittle nature of this material, Scanning Electron Microscopy (SEM) was performed on specimens from the fractured ring specimens. For comparison, the SEM study also included material from the boiler and pretest material neither of which are brittle. The results showed, in agreement with the optical microscopy, that the grain boundaries of the vapor crossover tube contain a fine ($\sim$ 0.5 micron) precipitate and the samples break essentially 100% intergranularly as shown in Figure 23c. On the other hand, the boiler material and the pretest material broke in a much more ductile manner as shown in Figure 23a and 23b, and, where visible, the grain boundaries are void of precipitate particles. Further SEM studies have shown that the particles contain 1.2 - 2.1 times the hafnium content than the nominal matrix composition; whereas, they contain 0.3 - 0.6 times the tantalum content of the matrix. Tungsten, carbon or oxygen which are also possible constituents of the particles could not be detected because of equipment limitations.

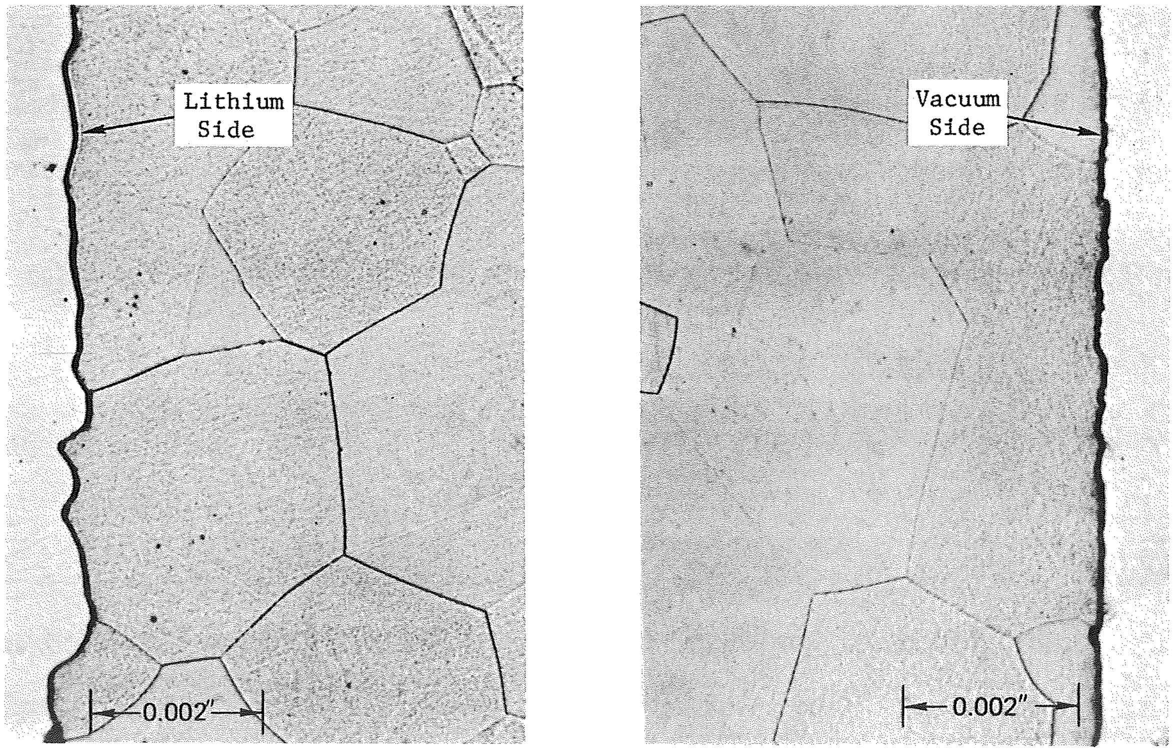
5. Turbine Simulator Nozzles and Blades

Sections from the Mo-TZC first, second and tenth stages and the Cb-132M sixth stage nozzle and blades were examined in the vapor impingement area. No change in general microstructure was observed in any of the specimens examined as shown in Figures 24-27. The only anomalous feature is a thin coating on the leading edge of the first and second stage blades and on the second stage nozzle as shown in Figures 24 and 25. Microprobe evaluation will be performed to determine the composition of the film. Typical pretest photomicrographs of each material are shown in Figure 28.



H64181A Etched

a. Total Wall



H64181B Etched

b. Inner Wall

H64181C Etched

c. Outer Wall

Figure 20. Lithium Line Connecting Heater Exit to the Boiler Inlet.

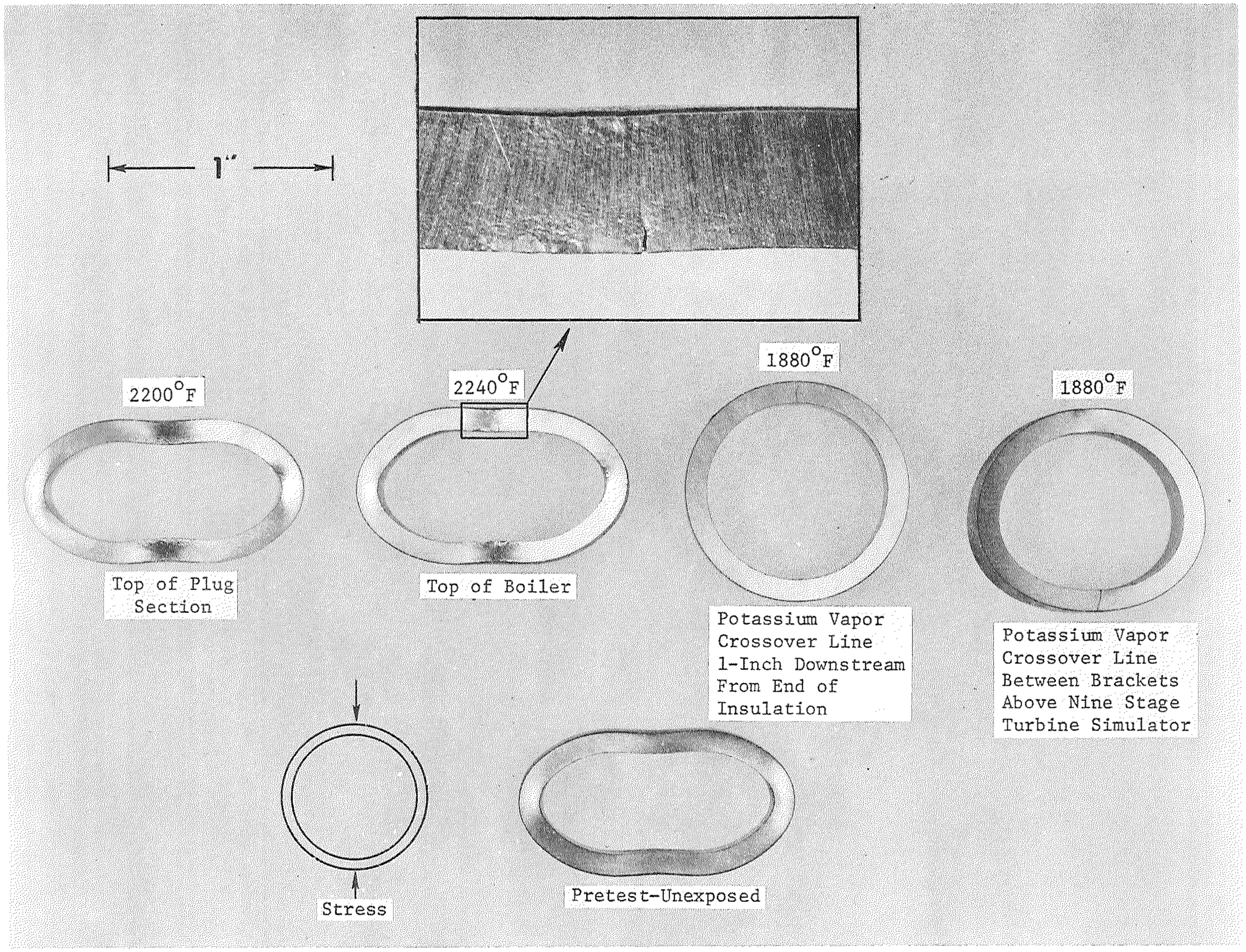
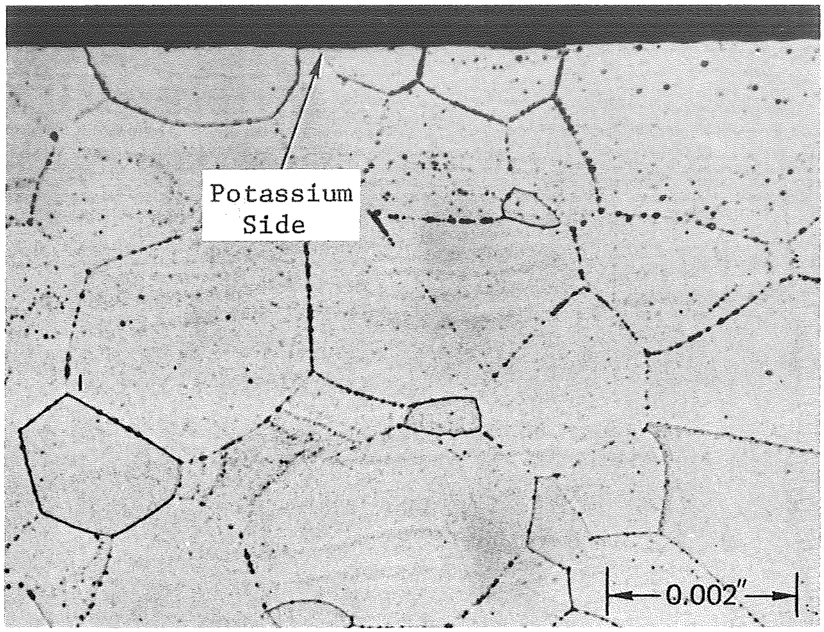
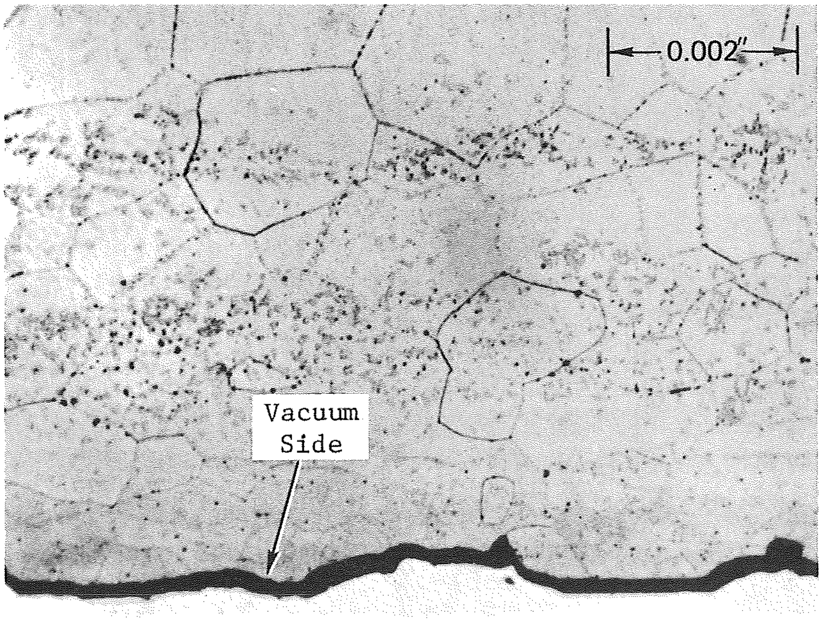


Figure 21. Specimens of 1-Inch OD x 0.1-Inch Wall T-111 Pipe Following 10,000 Hours Exposure in the T-111 Rankine System Corrosion Test Loop. Ring Specimens Deformed at Room Temperature. (P70-6-4D, P70-6-4F)



H64191B Etched

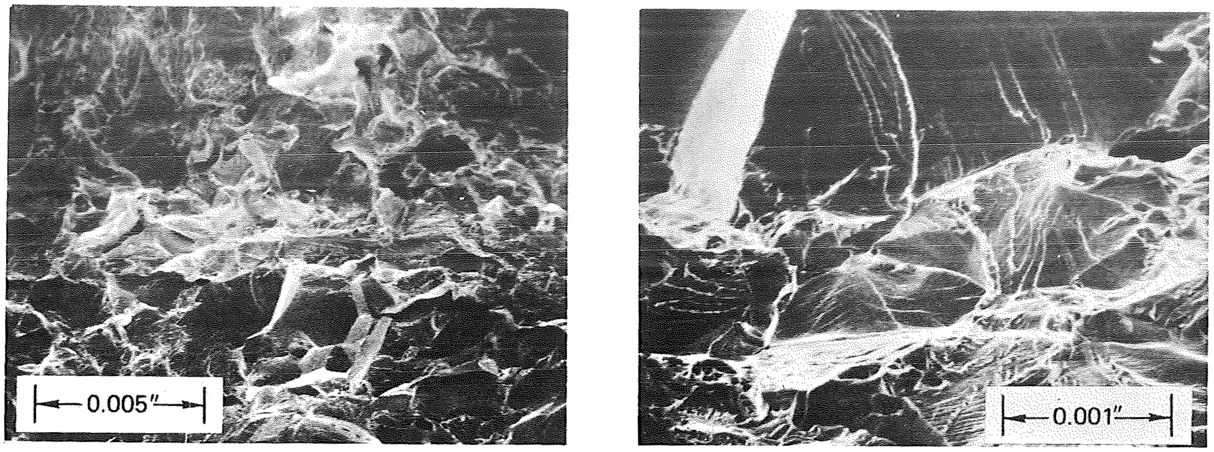
a. Inner Wall



H64191I Etched

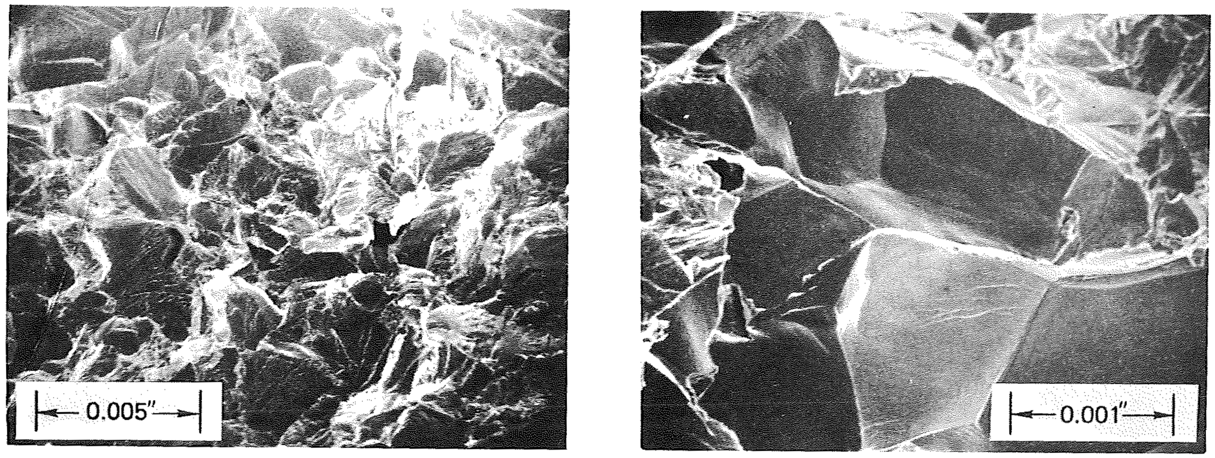
b. Outer Wall

Figure 22. 1-Inch OD Potassium Vapor Crossover Line Following 10,000 Hours of Continuous Operation at Approximately 1880°F.



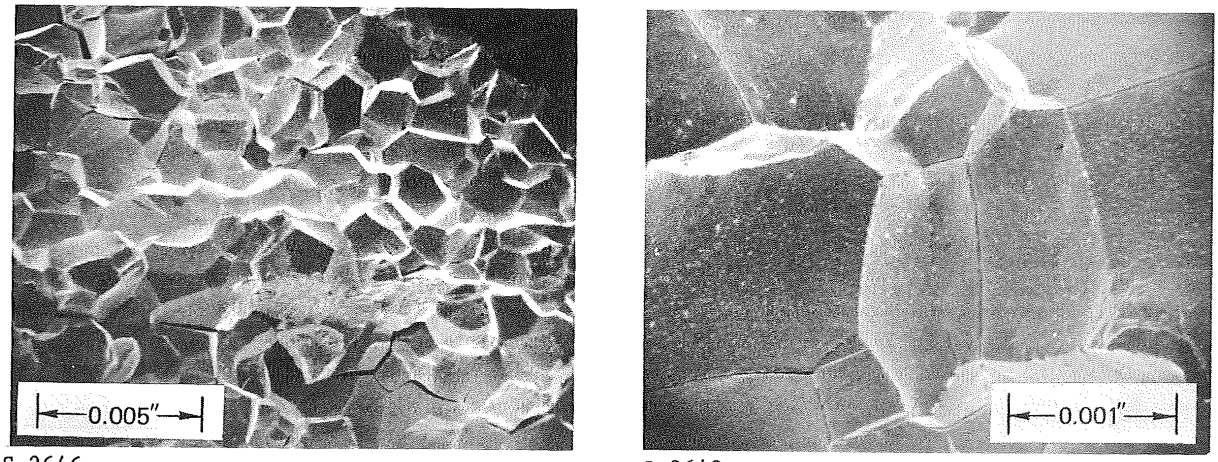
S-2663 As-Fractured S-2659 As-Fractured

a. Pretest



S-2657 As-Fractured S-2651 As-Fractured

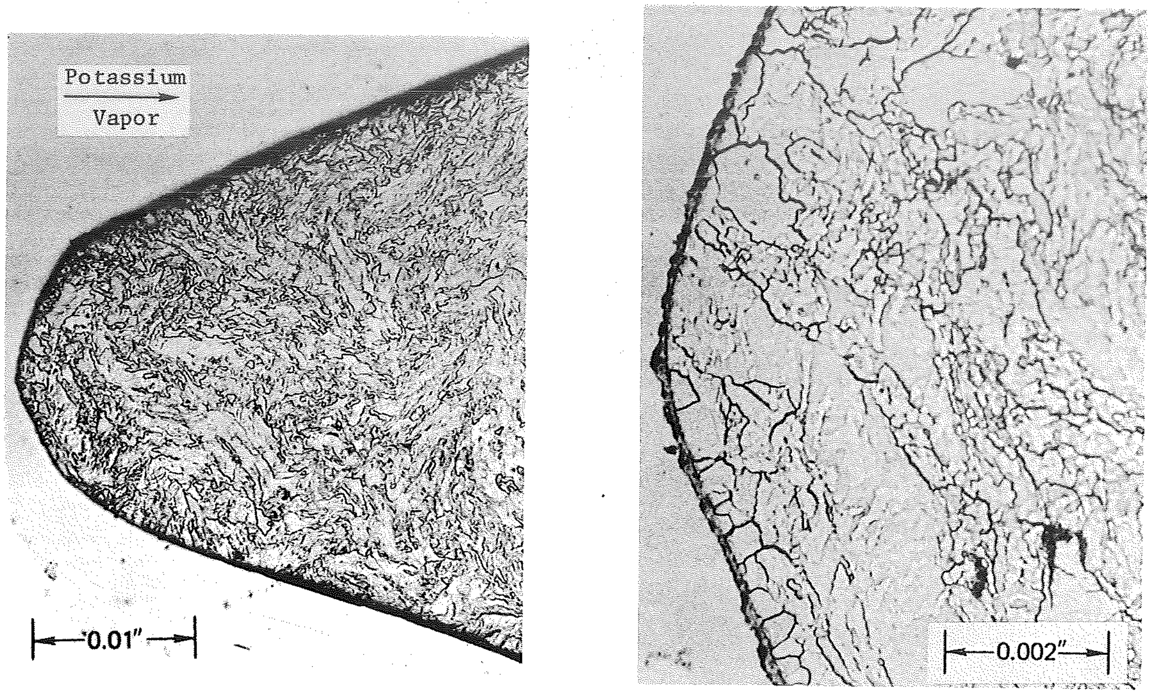
b. Boiler - 2240°F



S-2646 As-Fractured S-2648 As-Fractured

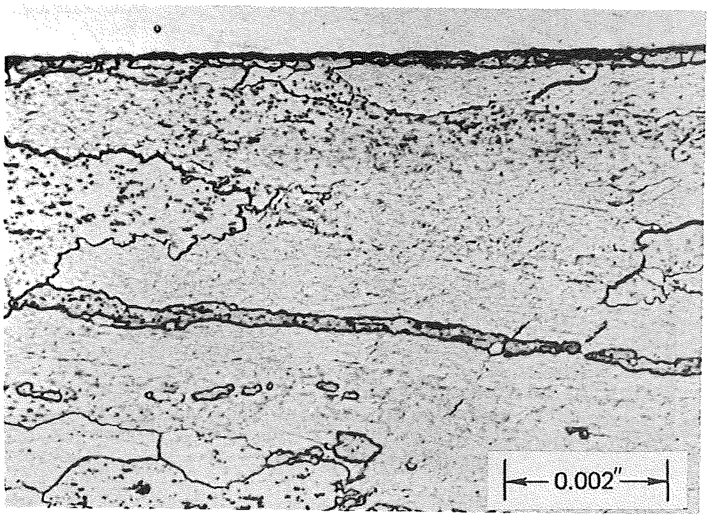
c. Vapor Crossover Line - 1880°F

Figure 23. Scanning Electron Micrographs of Various Components of T-111 Rankine System Corrosion Test Loop. All are Fractures from Compression Tests on Rings from 1-inch OD Tubing. (P71-2-3H)



H66011A Etched H66011B Etched

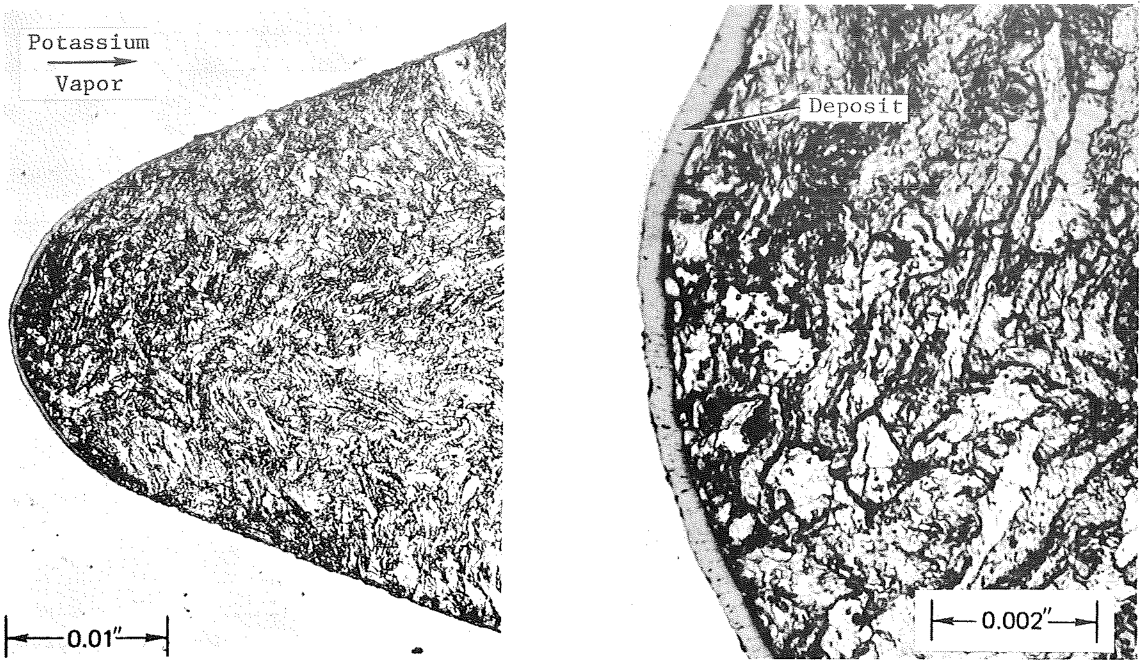
a. Blade



H66021B Etched

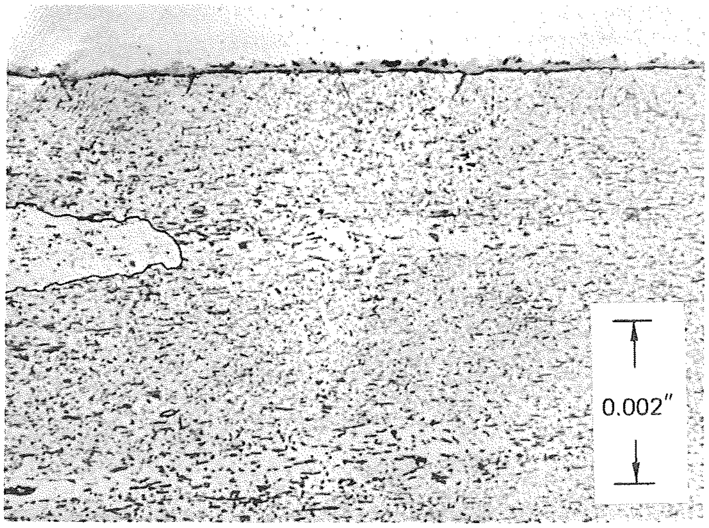
b. Nozzle

Figure 24. Mo-TZC Alloy First STage Nozzle and Blade Turbine Simulator Following 10,000 Hours of Continuous Exposure to Flowing Potassium Vapor in the T-111 Rankine System Corrosion Test Loop.



H66041A Etched H66041C Etched

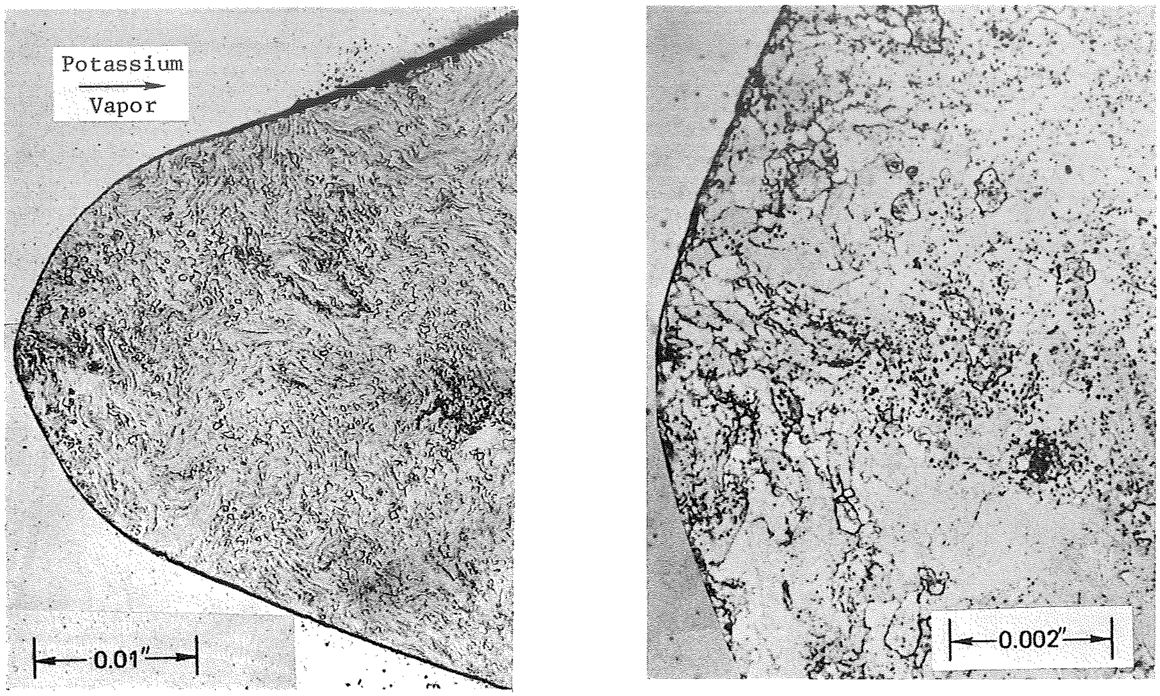
a. Blade



H66051C Etched

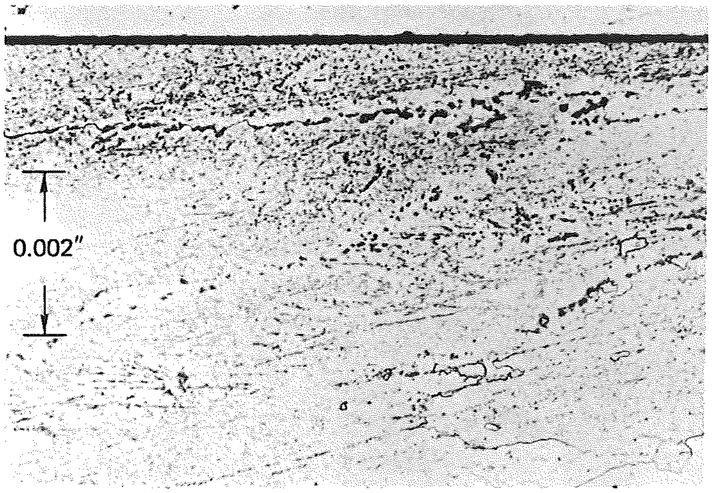
b. Nozzle

Figure 25. Mo-TZC Alloy Second Stage Nozzle and Blade Turbine Simulator Following 10,000 Hours of Continuous Exposure to Flowing Potassium Vapor in the T-111 Rankine System Corrosion Test Loop.



H65011B Etched H65011A Etched

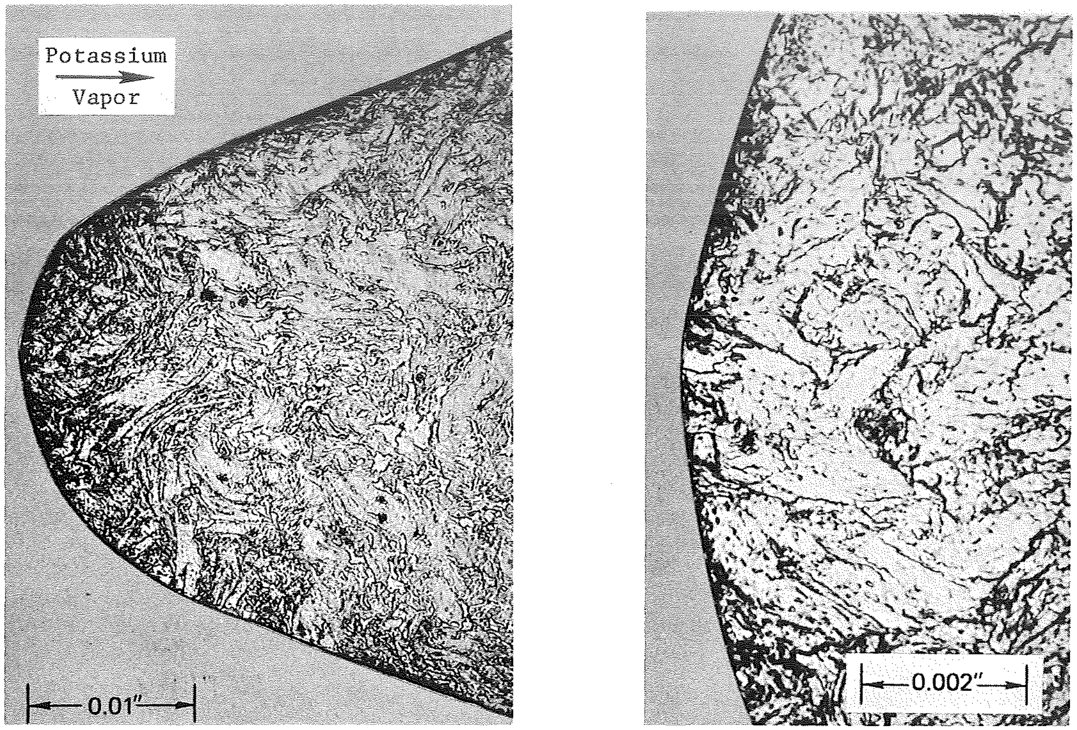
a. Blade



H65021A Etched

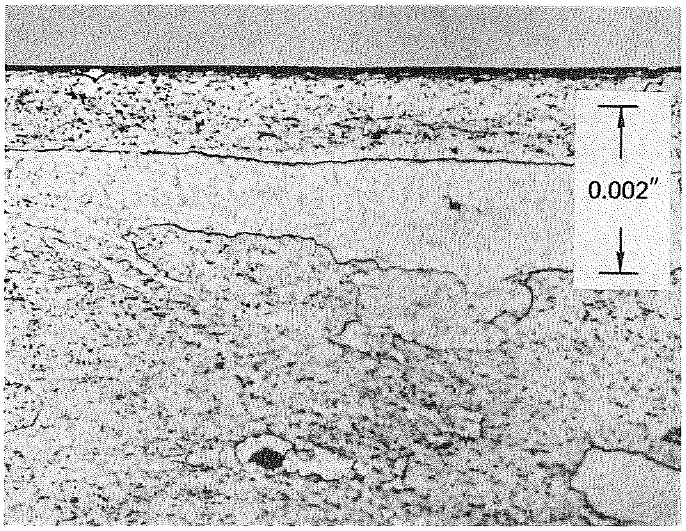
b. Nozzle

Figure 26. Cb-132M Alloy Sixth Stage Nozzle and Blade Turbine Simulator Following 10,000 Hours of Continuous Exposure to Potassium Vapor in the T-111 Rankine System Corrosion Test Loop.



H66061A Etched H66061B Etched

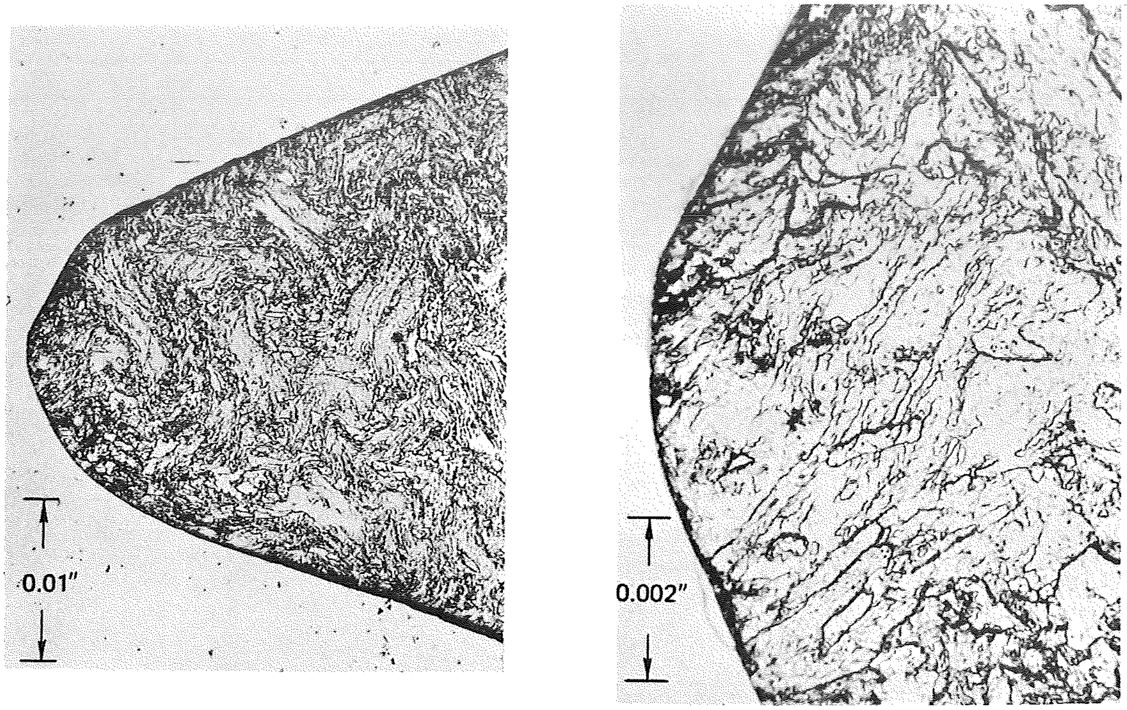
a. Blade



H66071A Etched

b. Nozzle

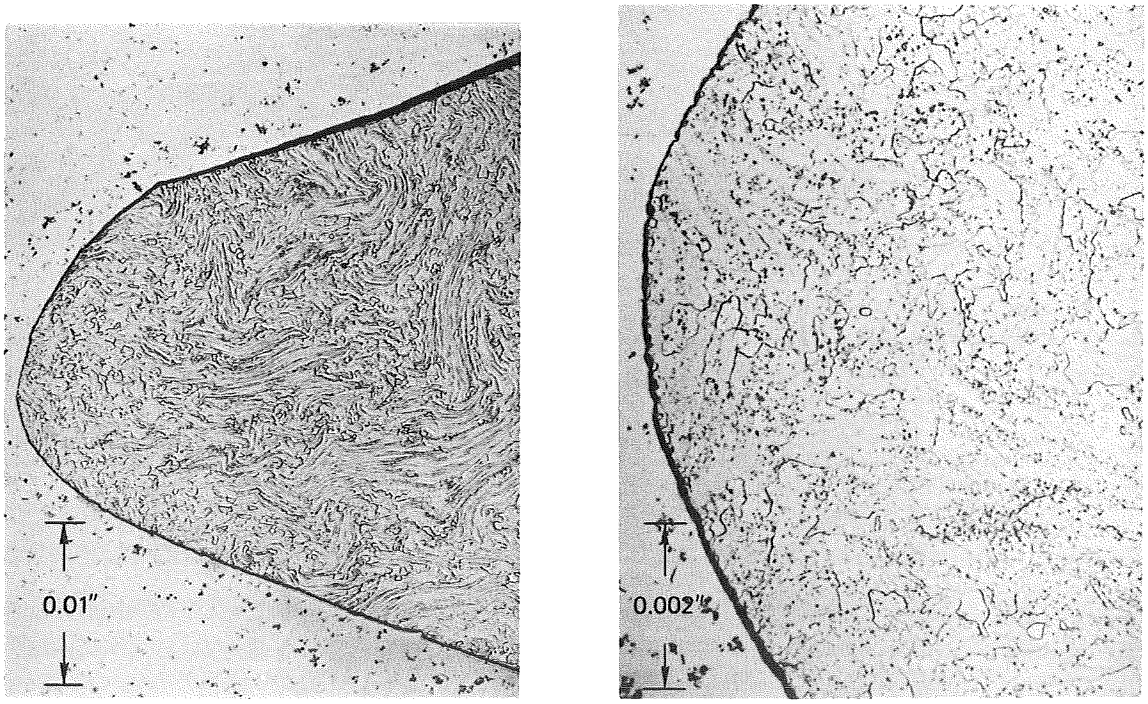
Figure 27. Mo-TZC Alloy Tenth Stage Nozzle and Blade Turbine Simulator Following 10,000 Hours of Continuous Exposure to Potassium Vapor in the T-111 Rankine System Corrosion Test Loop.



H66081A Etched

H66081B Etched

a. Mo-TZC Alloy



H65031B Etched

H65031A Etched

b. Cb-132M Alloy

Figure 28. Pretest Microstructure of Materials Used for Turbine Simulator of the T-111 Rankine System Corrosion Test Loop.

B. 1900°F Lithium Loop

At the completion of the postweld anneal on the new fuel specimen test section, the loop was filled with lithium and sampled. 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. On July 31, 1970 the loop was brought back to test conditions. 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 2600°F. For 1900°F operation the temperature drop is 65°F, even with the gas pocket, as shown in Figure 29. This is sufficiently close to the calculated design temperature drop of 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 has been operating very stably, with no indications of gas in the loop itself. Typical operating conditions since the restart of the test following the planned shutdown to replace the fuel element specimens are shown in Figure 29. These temperatures agree quite well with the temperatures during the initial 2500 hours of testing.(6) The chamber pressure and partial pressure of the various gaseous species in the vacuum chamber since the restart are shown in Figure 30 along with comparable data for the initial 2500 hours of testing. No significant differences can be seen for the two test periods. In general the test is operating very smoothly as of October 15, 1970 had logged an additional 1825 hours since the planned shutdown for a total accumulated test time of 4338 hours. Only two hours of test time were lost since the restart and these were caused by electrical instabilities in the building power supply on three separate occasions.

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(6) Advanced Refractory Alloy Corrosion Loop Program Report No. 20 for period ending April 15, 1970, NASA Contract NAS 3-6474, NASA-CR-72739 (GESP-491).

Date October 15, 1970 Test Time 4330 Hrs.
Chamber Pressure 1.7×10^{-9} Torr
Heater 37.9 A 290 V
Flow 2.22 mv 1.01 GPM

TEMPERATURES IN °F

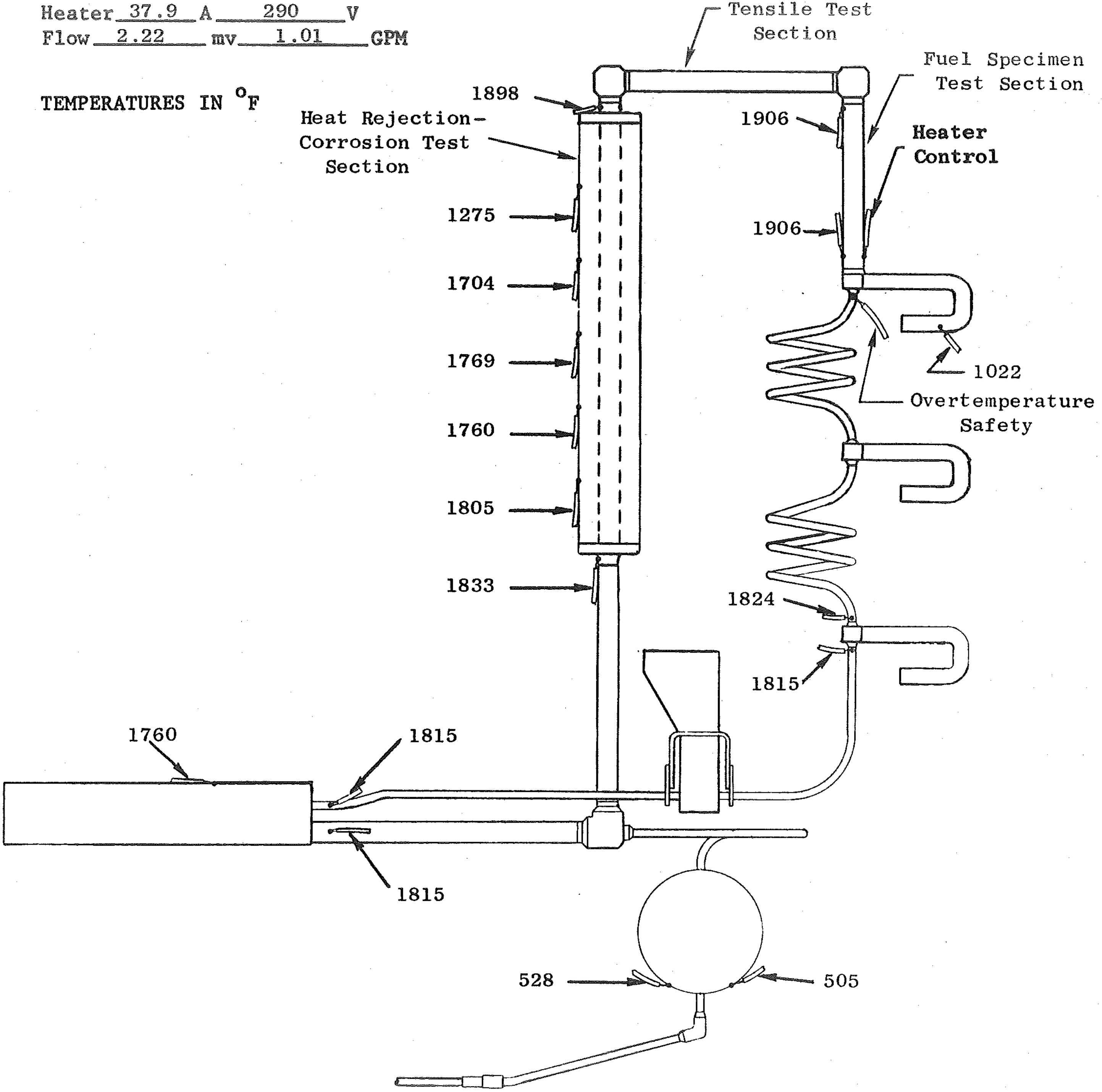


Figure 29. 1900°F Lithium Loop Operating Temperatures - 4330 Hours

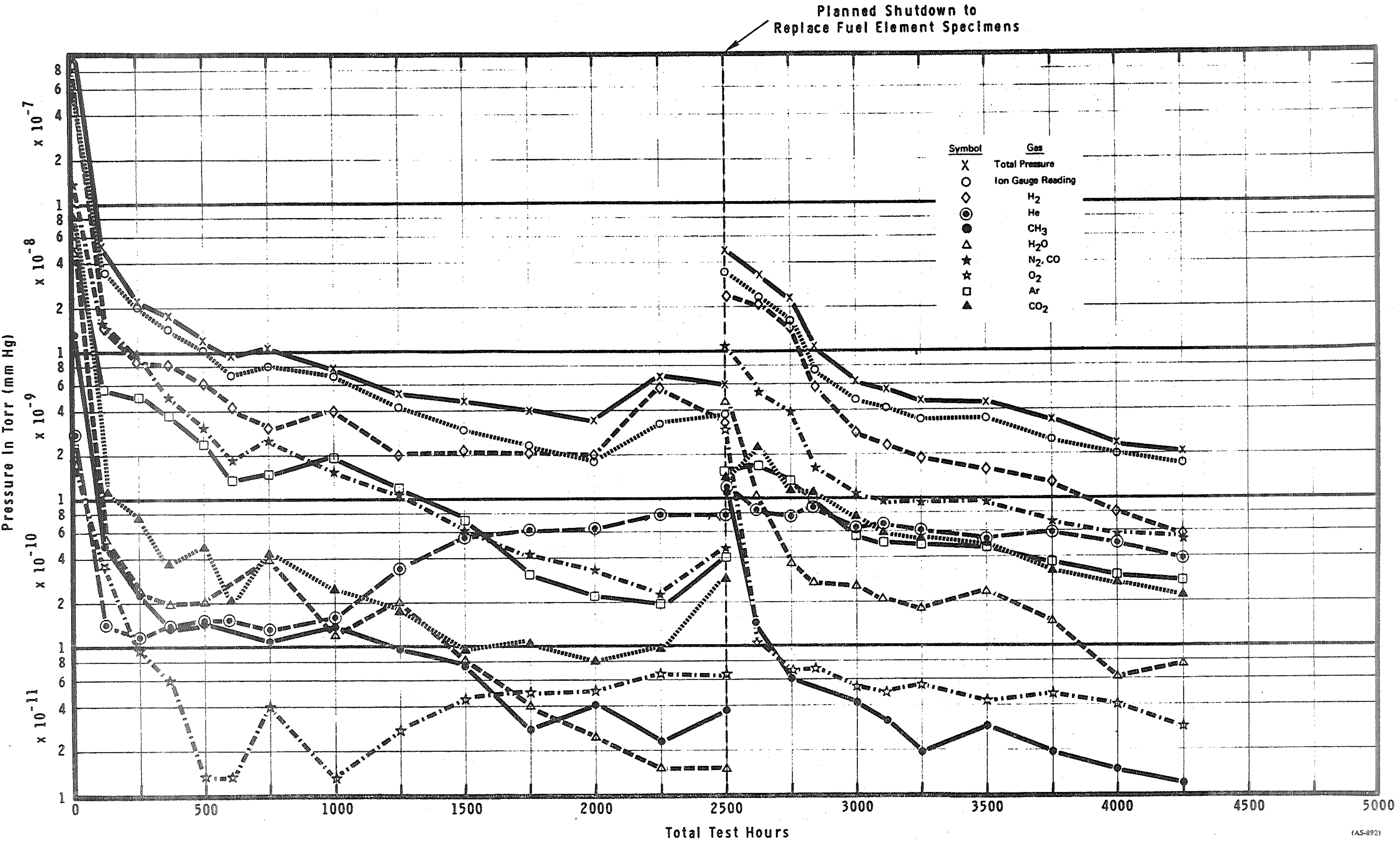


Figure 30. Test Chamber Environment During Testing of 1900°F Lithium Loop.

C. Lithium Thermal Convection Loop

The Advanced Refractory Alloy Corrosion Loop program has been modified to include the design, fabrication, instrumentation and evaluation of a T-111 alloy thermal convection loop test. The primary objective of the test program is to evaluate mass transfer of interstitial elements in T-111 and ASTAR alloys in a liquid lithium, thermally induced circulation, test loop. The test loop will be fabricated from T-111 alloy, and it will contain the following test specimens:

1. Tensile test specimens of T-111 and ASTAR 811C alloys.
2. Chemistry and metallography specimens of T-111 alloy and of the ASTAR type alloys.

The test loop will be operated for 5000 hours in the 2500-2700°F temperature range in an ultra-high vacuum chamber capable of a cold wall vacuum of 10^{-10} torr. Temperature profiles in the hot and cold leg vertical test sections will be used to calculate the lithium flow rate.

The loop is illustrated in the isometric drawing, Figure 31 , which shows the relative position and orientation of the principle loop components. The function of the principle components of the loop, shown in Figure 32 , and the test specimens are described below:

1. Test Specimens

T-111 alloy and ASTAR 811C alloy sheet tensile specimens for insertion in the hot and cold leg vertical test sections of the test loop were machined to the dimensions shown in Figure 33.

This special design test specimen allows for removal of material for chemical and metallographic evaluation following exposure to flowing lithium under test conditions, while retaining a standard tensile specimen, for determination of possible strength changes due to mass transfer of interstitial elements or structural changes. The specimen design thus permits a direct correlation between mass transfer of interstitial elements, structure, and possible changes in strength of the respective alloys.

T-111 alloy sheet specimens for insertion in the upper and lower curved sections of the test loop were machined to the dimensions shown

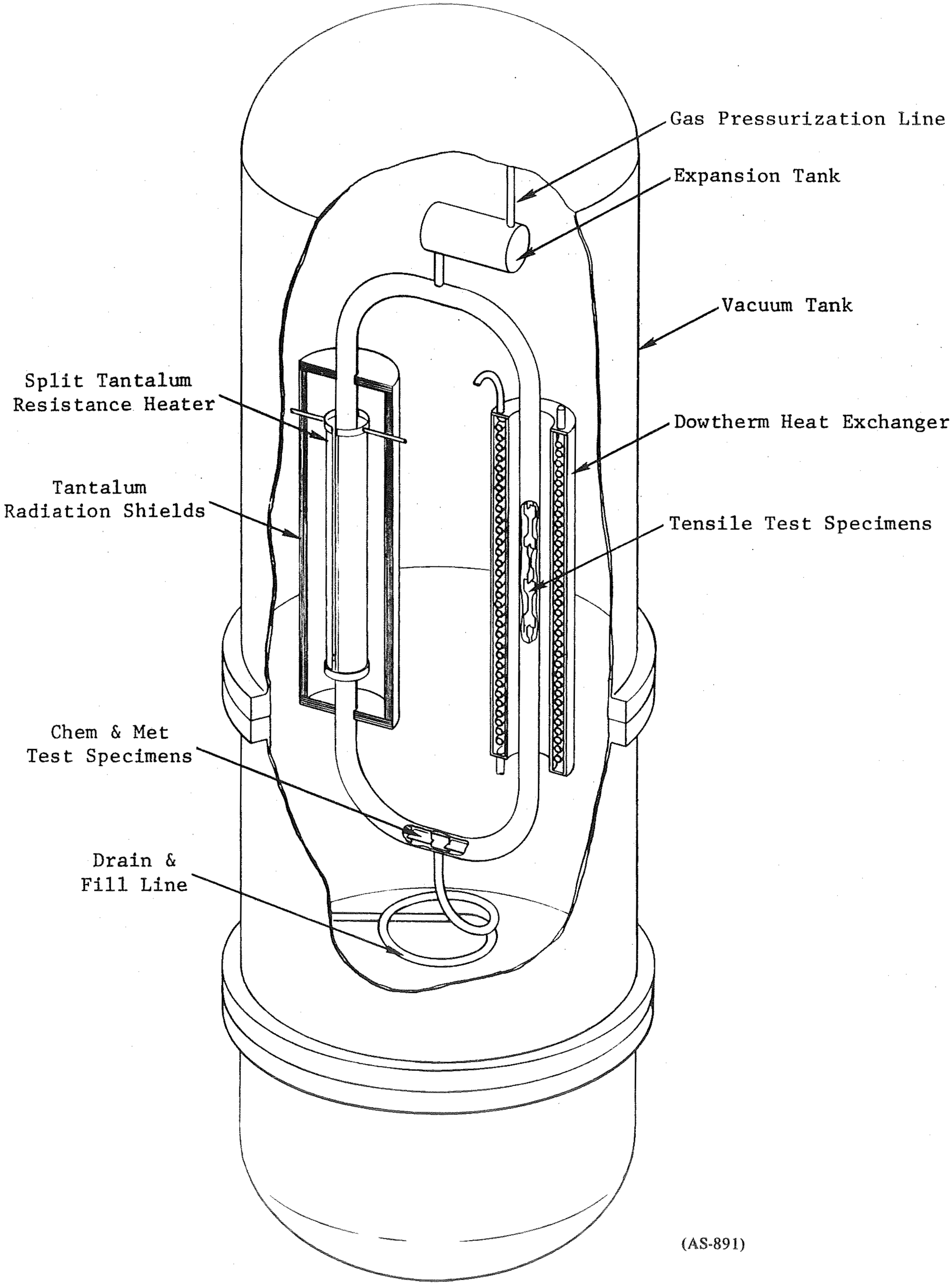


Figure 31. Isometric Drawing of Lithium Thermal Convection Test Loop.

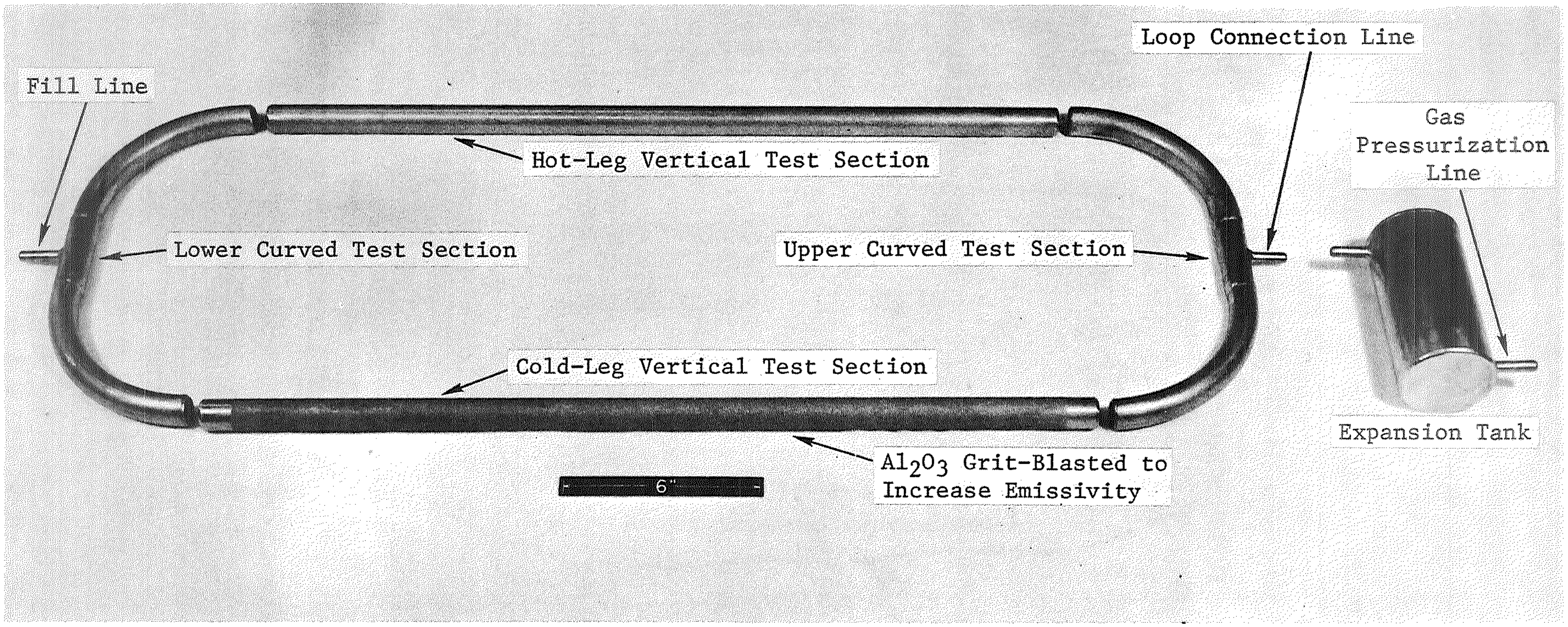
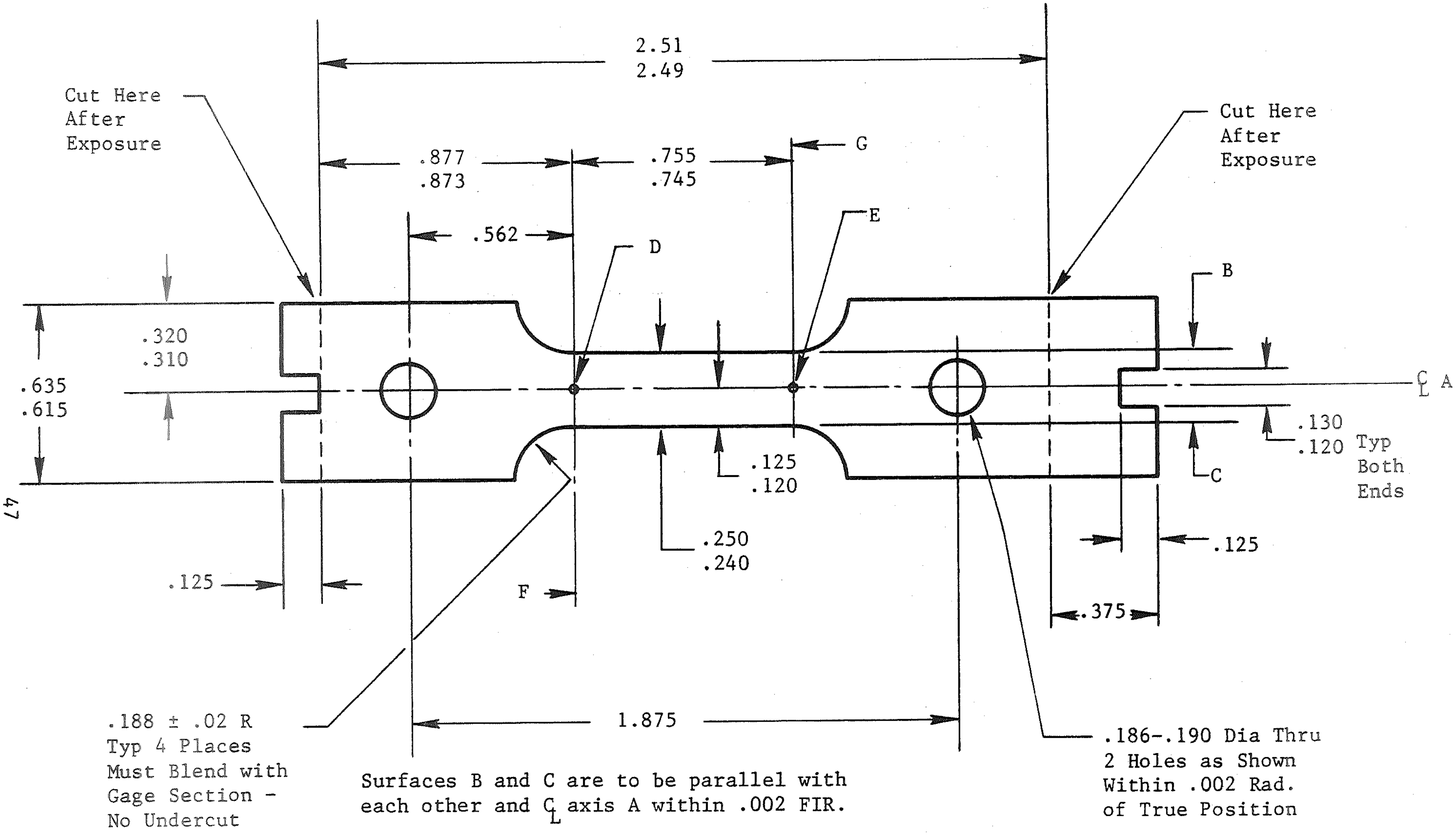


Figure 32. Relative Position of Major Loop Components for Lithium Thermal Convection Loop Prior to Fabrication. (P70-10-7G)



.188 ± .02 R
Typ 4 Places
Must Blend with
Gage Section -
No Undercut

.186-.190 Dia Thru
2 Holes as Shown
Within .002 Rad.
of True Position

Surfaces B and C are to be parallel with each other and ℄ axis A within .002 FIR.

Planes F and G must be established through points D and E and perpendicular to surfaces B and C within .002 FIR.

Figure 33. Design of Special Sheet Tensile Specimen for Insertion in the Vertical Test Sections of the Lithium Thermal Convection Test Loop.

Figure 34. This specimen will be used for chemical and metallographic evaluation, and will be used to monitor changes in the concentration of interstitials and metallurgical structure of specimens of the T-111 alloy and ASTAR-type alloys in the portions of the loop which connect the hot and cold legs.

2. Split Tantalum Resistance Heating Element

Heating of the hot-leg vertical test section of the loop will be accomplished with a split tantalum resistance heating element. The resistance heater will be fabricated entirely of unalloyed tantalum and will be 2.5-inches in diameter x 18.0-inches long. The upper and lower rings of the heater will be formed from two pieces of 0.062-inch thick x 0.5-inch wide tantalum strip which will enclose the tantalum foil of the heater body. The heater body will be fabricated from 0.005-inch thick tantalum foil which has been corrugated with approximately 0.125-inch corrugations extending along the length of the heater. Corrugating the tantalum foil will increase its stiffness over the relatively long 18.0-inch length as well as increasing its emissivity. The electrodes are machined from 0.5-inch diameter tantalum rod and are butt welded to the upper tantalum rings. A 20 kw saturable core reactor with a high current, step down transformer will be used to supply electrical power to the tantalum heater.

To reduce heater losses the heater will be enclosed in an insulation can fabricated from 15 layers of 0.003-inch tantalum foil.

3. Dowtherm Filled Water Cooled Heat Sink

Heat shall be rejected in the cold-leg vertical test section by radiation to a Dowtherm-filled water cooled heat sink surrounding the T-111 piping. The components required to fabricate the heat sink are shown in Figure 35. The main components consist of a 5.0-inch schedule 40 x 24.0-inch long Type 304 stainless steel outer shell, 0.375-inch OD x 0.049-inch wall Type 304 stainless steel tube water cooling coil, and a 3.0-inch schedule 40 x 24.0-inch long Type 304 stainless steel inner shell. Following assembly of the heat sink it will be filled with Dowtherm "A" through a 0.375-inch OD x 0.049-inch wall Type 304 stainless steel fill tube which will then be sealed by welding.

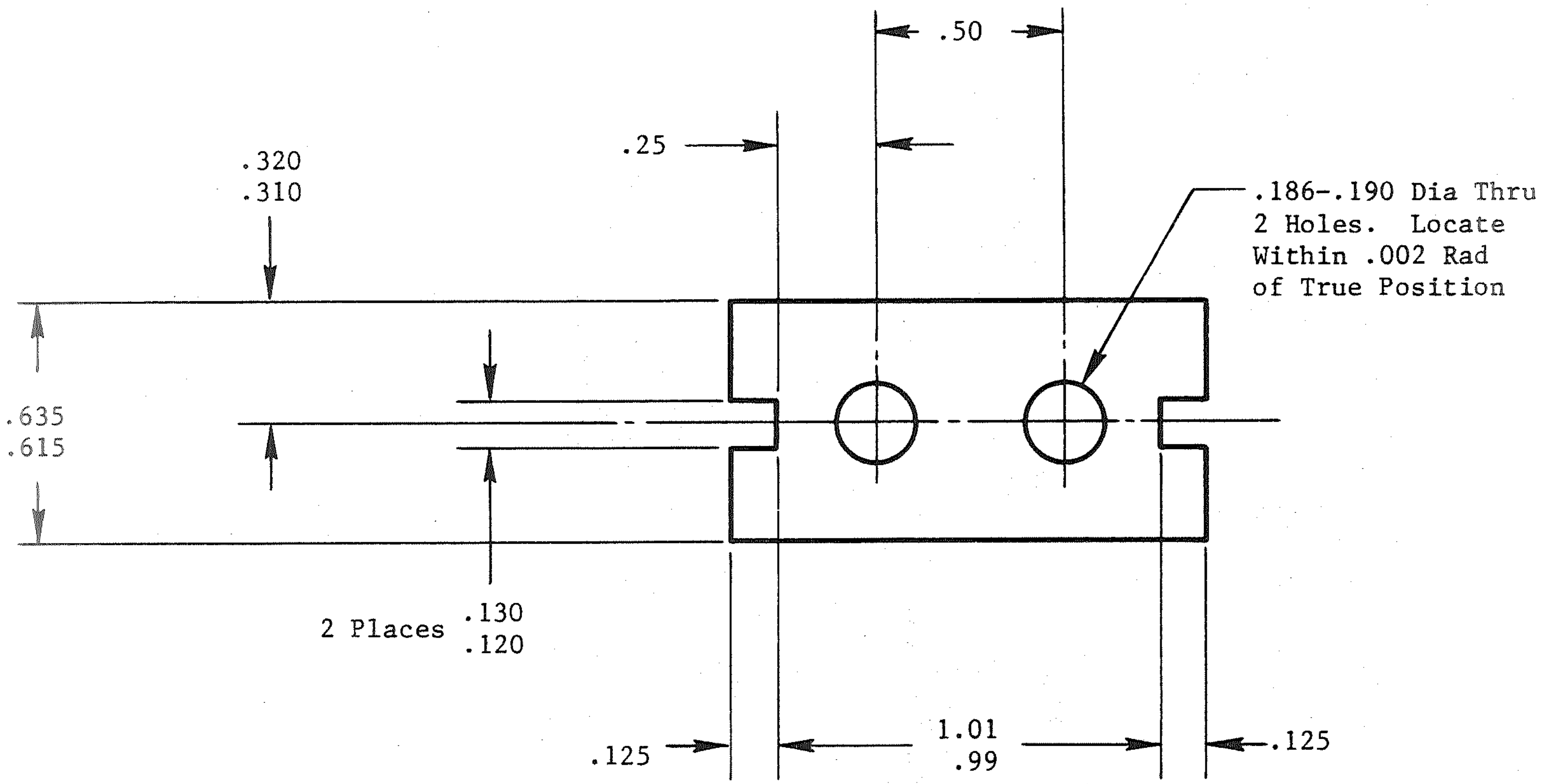


Figure 34. Design of Sheet Test Specimen for Insertion in the Curved Test Sections of the Lithium Thermal Convection Test Loop.

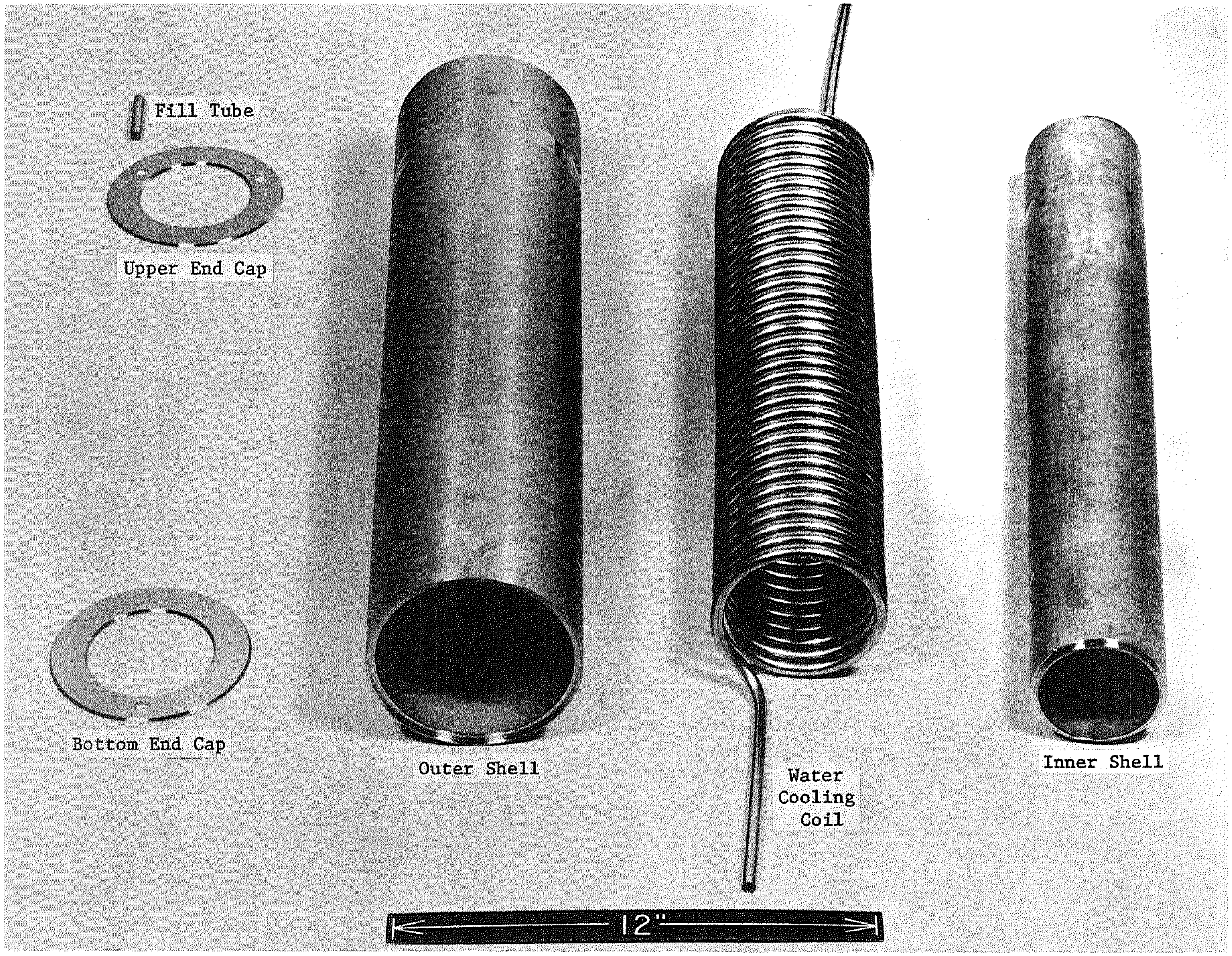


Figure 35. Components of Dowtherm-Filled Water Cooled Heat Sink Prior to Fabrication. (P70-10-14C)

Dowtherm "A" was chosen as the fluid to fill the heat exchanger cavity because of its low vapor pressure in the temperature range of interest, 300° to 400°F. Following welding of the water lines to the vacuum feedthrough tubes, thermistors will be positioned inside the water lines at the inlet and outlet positions of the heat exchanger in order to permit accurate measurement of the temperature rise of the water. The total heat radiated will be determined by measuring the flow rate of cooling water and the inlet and outlet water temperatures at the heat exchanger. Water flow-measurements will be determined by weighing volumes collected in a ten-minute period.

4. Hot and Cold Leg Vertical Test Sections

The hot and cold leg vertical test sections of the loop are fabricated from 1.0-inch OD x 0.100-inch wall x 28.0-inches long seamless T-111 alloy tube. Each leg will contain ten sheet tensile specimens machined to the dimensions of the special design tensile specimen shown previously in Figure 33. T-111 and ASTAR 811C alloy specimens will be alternated in each leg. The cold-leg vertical test section has been grit-blasted with alumina to increase its emissivity. The specimens and the hot-leg vertical test section are shown before assembly in Figure 36. The test specimens are lock wired together with unalloyed tantalum wire.

5. Upper and Lower Curved Test Section

The upper and lower curved test sections of the loop, shown in Figure 37, were fabricated from 1.0-inch OD x 0.100-inch wall seamless T-111 alloy tube, which were obtained from the outer shell of the tube-in-tube boiler used in the T-111 Rankine System Corrosion Test Loop. Although this tubing was exposed to lithium for 10,000 hours, posttest evaluation indicated no effects of the alkali metal exposure which would prohibit its reuse. The straight piece in the middle of each curved test section was fabricated from previously unexposed T-111 alloy. These test sections will contain the chemical and metallographic test specimens of T-111 alloy and ASTAR type alloys shown previously in

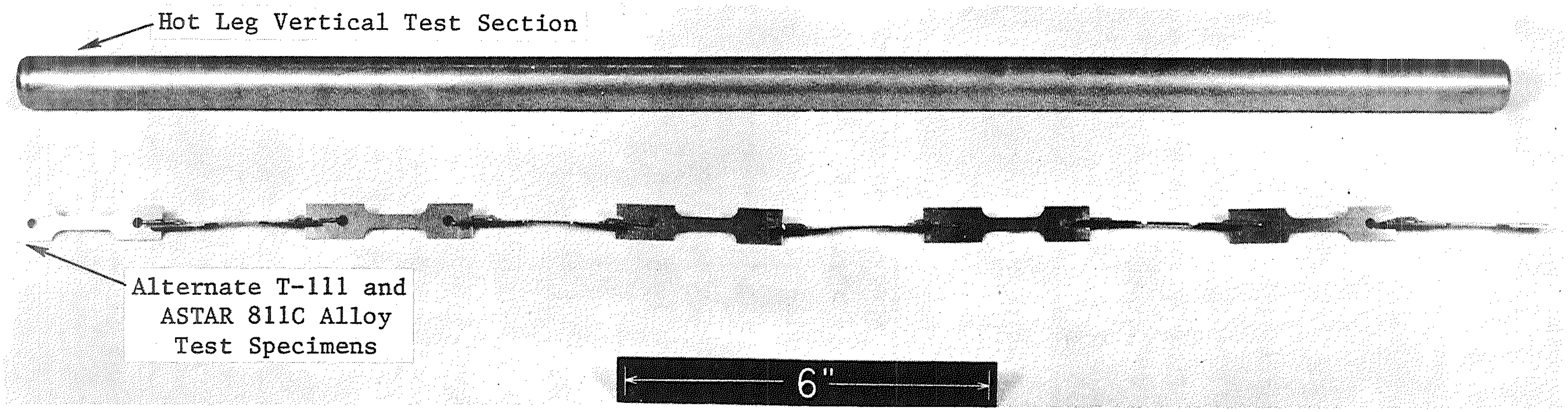


Figure 36. Hot Leg Vertical Test Section of Lithium Thermal Convection Loop with T-111 Alloy and ASTAR 811C Alloy Sheet Tensile Specimens to be Inserted Prior to Test. (P70-10-7D)

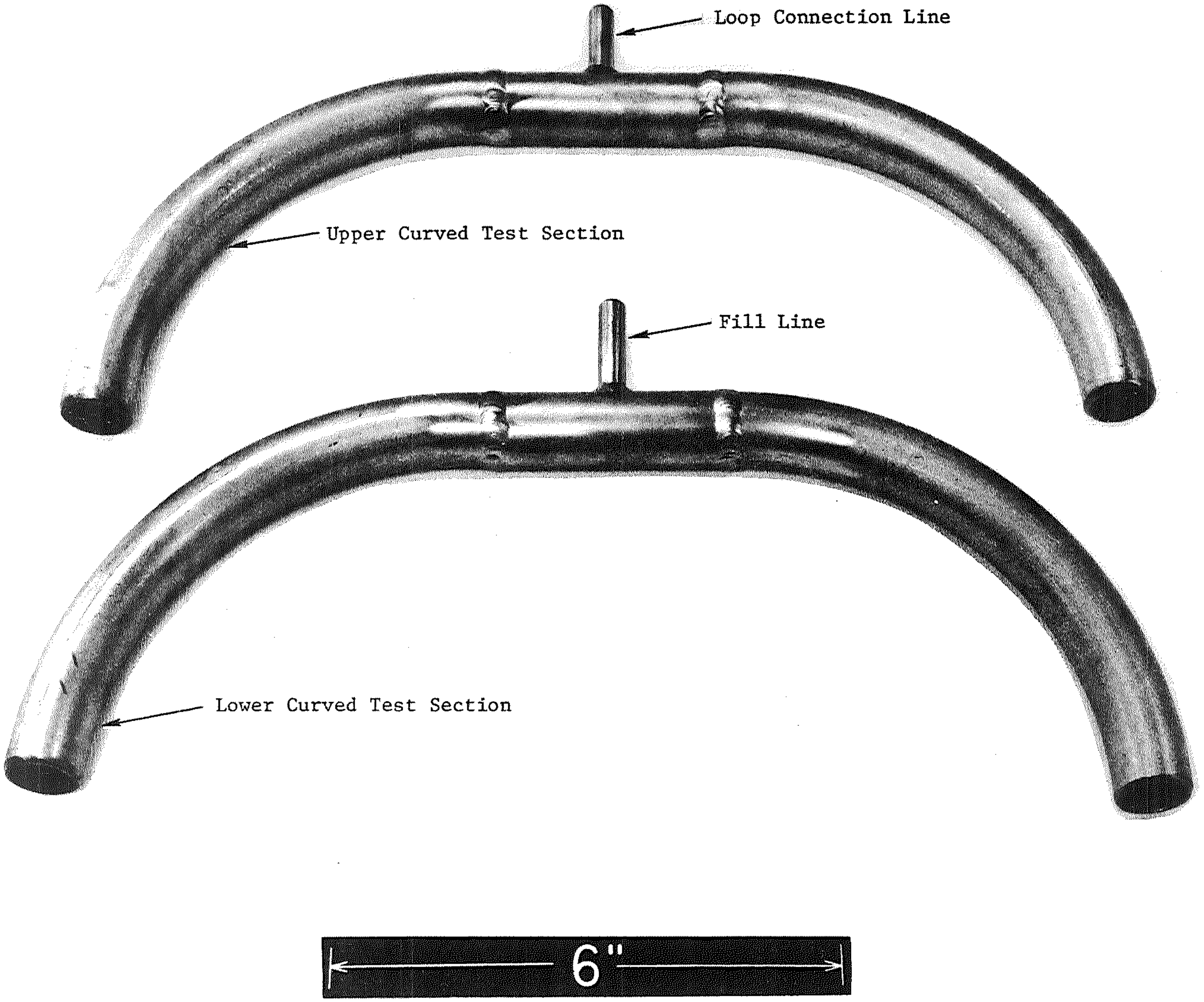


Figure 37. Upper and Lower Curved Test Sections for Lithium Thermal Convection Loop. (P70-10-7E)

Figure 34. The lower curved test section with typical specimens to be inserted are shown in Figure 38. The specimens are lock wired together with unalloyed tantalum wire.

6. Expansion Tank

The expansion tank for the test loop has been fabricated from 3.0-inch OD x 0.080-inch x 6.0-inch seamless T-111 alloy tube, and is shown in Figure 39. The gas pressurization line and the loop connection line are 0.375-inch OD x 0.065-inch wall seamless T-111 alloy tubes. The end caps for the tank were machined from 0.100-inch thick T-111 alloy plate.

7. Lithium

The loop will be filled with lithium which has been purified by hot trapping and vacuum distillation. A sample of the lithium will be obtained during the filling of the loop and will be analyzed for oxygen, nitrogen, carbon, hydrogen and metallic impurities. Testing will not be initiated unless the oxygen concentration of the lithium is less than 150 ppm and the nitrogen concentration is less than 20 ppm.

8. Test Chamber

The entire loop will be contained in a 24-inch diameter vacuum chamber capable of a cold wall vacuum of 1×10^{-10} torr. A 1000 liter per second getter ion pump will be used to maintain the vacuum chamber pressure in the 10^{-8} torr range during the test. The loop will be 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 will be welded to a 22-inch high center spool section that is used to facilitate both the manufacturing and installation of the loop.

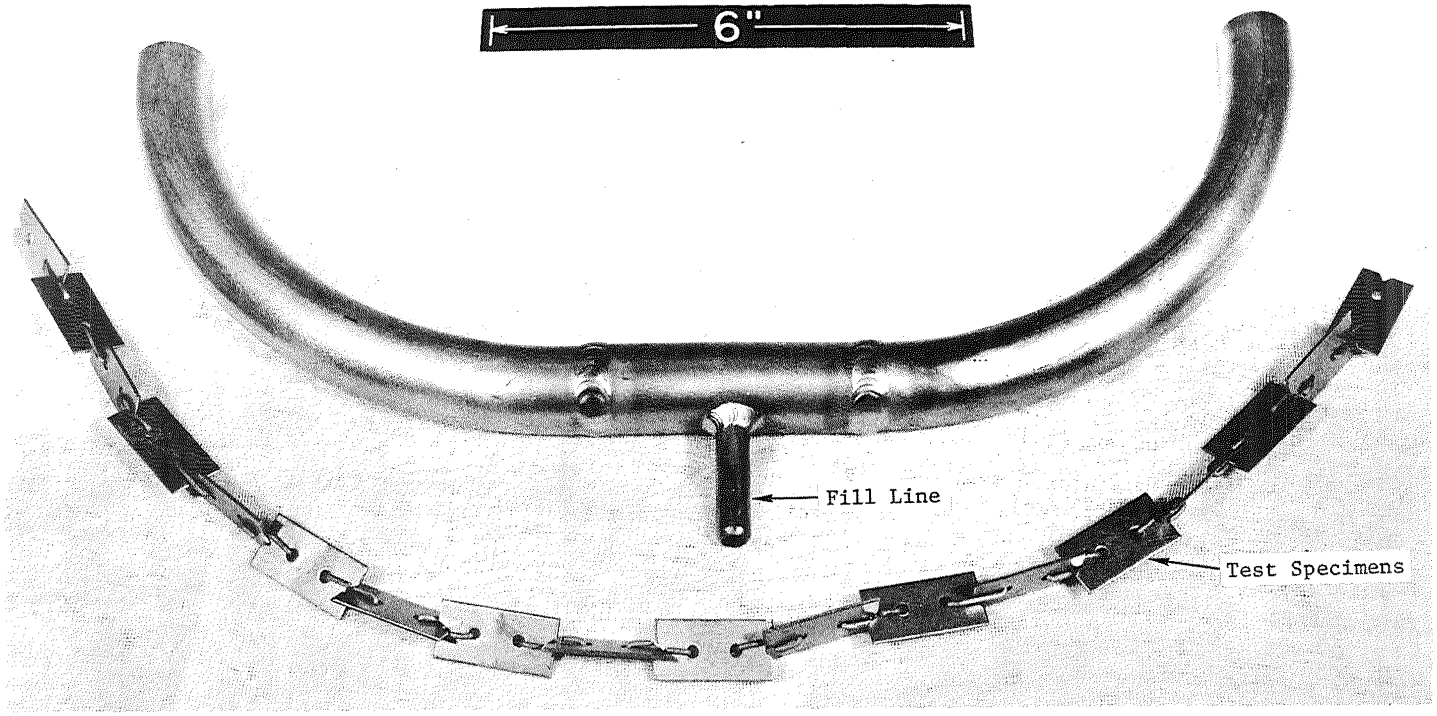


Figure 38. Lower Curved Test Section of Lithium Thermal Convection Loop with Typical Specimens to be Inserted Prior to Test. (P70-10-7F)

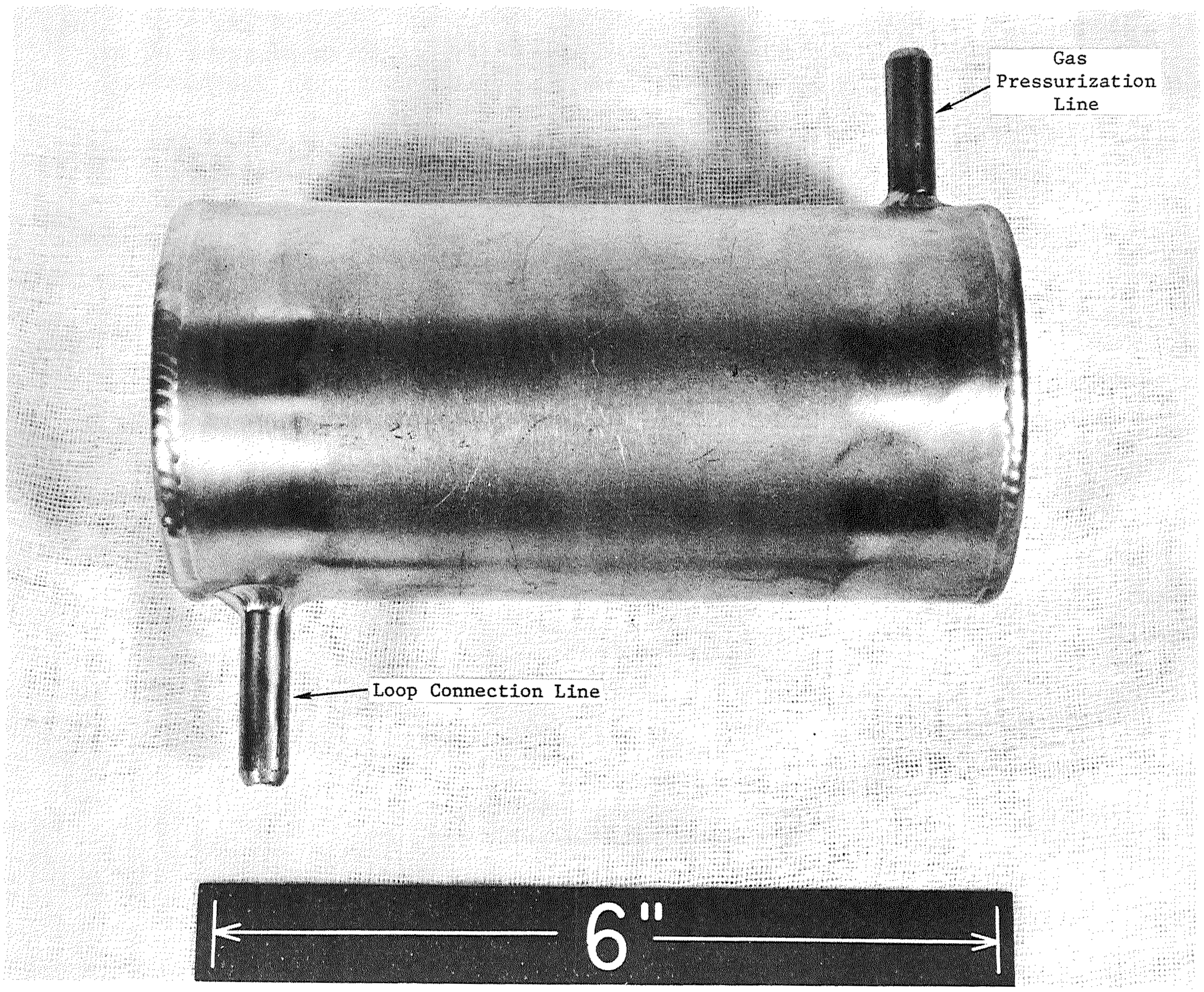


Figure 39. Expansion Tank for Lithium Thermal Convection Loop. (P70-10-7B)

IV. FUTURE PLANS

Complete posttest evaluation of the T-111 Rankine System Corrosion Test Loop and issue a final topical report.

Continue testing of 1900°F Lithium Loop.

Complete fabrication and instrumentation of the Lithium Thermal Convection Loop. Tentative plans indicate the possibility of test initiation during the next quarterly reporting period.

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
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