NASA CONTRACTOR REPORT

NASA CR-1810

REFRACTORY METAL VALVES FOR 1900° F SERVICE IN ALKALI METAL SYSTEMS

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Prepared by
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NATIONAL AERONAUTICS AND SPACE ADMINISTRATION • WASHINGTON, D. C. • MAY 1971

1. Report No. NASA CR-1810	2. Government Accession No.	3. Recipient's Catalog No.
4. Title and Subtitle REFRACTORY METAL VALVES FOR 1900° F SERVICE IN ALKALI METAL SYSTEMS		5. Report Date May 1971
		6. Performing Organization Code
7. Author(s) R. W. Harrison and J. Holowach		8. Performing Organization Report No. GESP-508
		10. Work Unit No.
9. Performing Organization Name and Address General Electric Company Cincinnati, Ohio 45215		11. Contract or Grant No. NAS 3-8514
		13. Type of Report and Period Covered Contractor Report
12. Sponsoring Agency Name and Address National Aeronautics and Space Administration Washington, D. C. 20546		14. Sponsoring Agency Code
15. Supplementary Notes		

16. Abstract

Refractory metal metering and isolation valves were designed and evaluated in a forced circulation lithium loop operated at a maximum temperature of 2100° F for 5000 hours in an ultra high vacuum (10^{-9} torr) environment. The valves, which were exposed to a maximum temperature of 1900° F, were cycled during testing to simulate actual operation. Rhenium seats and W-25Re plugs were employed to prevent diffusion bonding during testing. Posttest evaluation of the valve components indicated the materials were compatible with the alkali metal environment and no degradation had occurred as a result of valve operation. Sheet tensile specimens of T-111 (Ta-8W-2Hf), T-222 (Ta-9.6W-2.4Hf-.01C), ASTAR 811 (Ta-8W-1Hf-1Re), ASTAR 811C (Ta-8W-0.7Hf-1Re-.025C), ASTAR 811CN (Ta-8W-1Hf-Re-.012C-.012N), and W-Re-Mo Alloy 256 (W-29Re-18Mo) were placed in a portion of the loop circuit which operated at a maximum temperature of 2100° F with a 200° F temperature drop along the length of the specimen containment tube. Metallurgical evaluation of these specimens indicated no alkali metal attack. The results of room temperature and 2000° F tensile tests following the 5000 hour exposure showed no changes in strength or ductility that could be attributed to the lithium exposure.

17. Key Words (Suggested by Author(s)) Refractory metal High-temperature valves Alkali metal valves	18. Distribution Statement Unclassified - unlimited

19. Security Classif. (of this report)	20. Security Classif. (of this page)	21. No. of Pages	22. Price*
Unclassified	Unclassified	166	$3.00

*For sale by the National Technical Information Service, Springfield, Virginia 22151

FOREWORD

The research described in this report was conducted by the General Electric Company under NASA contract NAS 3-8514. Mr. Phillip L. Stone of the NASA Lewis Research Center Materials and Structures Division was the NASA Project Manager. The report was originally issued as General Electric report GESP-508.

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I. INTRODUCTION

This report describes the results of a program to design and evaluate 1-inch, refractory metal alloy, metering and isolation valves for alkali metal service in the 1200° to 1900°F temperature range for 5000 hours in an ultrahigh vacuum environment. In addition to determining the mechanical cycling and temperature capabilities of the valves, the solid phase bonding characteristics of the isolation valve plug and seat were also determined. The Cb-1Zr Pumped Sodium Loop[(1)] which was built and operated under Contract NAS 3-2547 was reconstructed for the valve test in the existing 2-foot-diameter by 5-foot-high vacuum chamber. Although valves of the type tested would more likely be used in the potassium circuit of a Rankine Cycle space power system, lithium was selected as the test fluid since the lithium corrosion resistance of advanced refractory alloy containment materials was also to be evaluated in this loop.

Sheet tensile specimens (two specimens each of T-111, T-222, ASTAR 811, ASTAR 811C, ASTAR 811CN, and W-Re-Mo Alloy 256)* were located in the heat rejection section of the loop circuit which operated at a maximum temperature of 2100°F and a minimum temperature of 1900°F.

Three Cb-1Zr slack diaphragm pressure transducers were used to obtain the operating characteristics of the EM pump and the pressure drop across the 1-inch metering valve during loop operation.

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(1) Hoffman, E. E. and Holowach, J., Cb-1Zr Pumped Sodium Loop, NASA CR-1135, November 1968.

*Composition of these alloys is given in Table I, Section II

The valves were actuated during the loop test under simulated operational conditions given below:

Lithium Temperature At Valves °F	Test Period Hours	Total Test Hours	Cumulative Cycles On Isolation Valves	Cumulative Cycles On Metering Valve	
			Full Travel	1/4 Travel	3/4 Travel
1200	100	100	2	10	2
1400	200	300	6	30	6
1600	200	500	10	50	10
1700	500	1000	15	100	20
1800	500	1500	25	150	30
1900	1000	2500	45	250	50
1900	500	3000	55	300	60
1900	500	3500	65	350	70
1900	500	4000	75	400	80
1900	500	4500	85	450	90
1900	500	5000	95	500	100

The isolation valve was closed and maintained in the closed position using 12 in-lb of torque on the magnetic rotary feedthrough during the steady state operation. The torque required to unseat the valve plug on the valve was measured at the end of each steady state run.

Upon completion of 5000 hours of operation, the loop was completely dismantled for metallurgical evaluation of the effects of lithium exposure upon the various components and test specimens in the system.

II. VALVE TEST DESIGN

A. DESIGN OF THE METERING AND ISOLATION VALVES

1. Initial Design

Refractory alloy, bellows sealed valves are not commercially available because of the limited demand. The procurement of refractory valves even on special order is a complex process since commercial valve manufacturers are not familiar with refractory metal properties and technology and do not have the special welding and heat treating facilities required in joining refractory alloy parts. However, arrangements can be made with several valve manufacturers to design and machine all necessary valve components with the welding, heat treating and material control being done by the customer.

The design of the metering and isolation valves was subcontracted to Hoke, Inc., Cresskill, New Jersey, with similar arrangements as previously negotiated on the procurement of Cb-1Zr alloy valves for the Cb-1Zr Rankine System Corrosion Test Loop.[(2)] The general design specifications submitted to Hoke, Inc. were as follows:

Type: Bellows seal, manually operated

Maximum Body Temperature: 1900°F

Maximum Pressure: 200 psia

Fluid: Lithium

Materials: All parts in contact with process fluid to be T-111 except a W-25Re alloy plug and a rhenium* seat

Valve Stem Thrust Device: Saginaw ball screw**

Connections: 1.00-inch x 0.100-inch-wall T-111 tube

Environment: 1×10^{-8} torr

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(2) 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.

* A TiC-10Cb seat was originally considered; however, the results of a lithium compatibility test, described in Appendix A, indicated this material lacked sufficient corrosion resistance to lithium. A lithium compatibility test of rhenium, also described in Appendix A, indicated this material to be compatible with lithium.

** Saginaw Steering Gear Division, General Motors Corp., Saginaw, Michigan.

The results of the design effort by Hoke, Inc. was the Model 443X3 valve shown in Figure 1. GE-NSP's main effort in the design of the valve was in the specification and procurement of materials, and the detail design of the welded joints especially the bellows assembly. Bellows fabrication and associated heat treatments as well as the quality assurance tests performed on this component are described in Section IV, Valve Test Loop Fabrication.

2. Design Modifications

Before testing of the valve shown in Figure 1 was initiated a number of design modifications were incorporated to eliminate deficiencies in the valve actuation portion of the design uncovered during pretest checkout tests. An additional design modification was made as a result of valve testing experience during the first 2500 hour test period. These modifications are described in detail below.

During the pretest checkout of the metering and isolation valves of the T-111 Rankine System Corrosion Test Loop the tungsten carbide sleeve bearings seized on the Mo-TZM shaft of the pinion gear after a limited number of operations (less than 25) at temperatures up to 600°F.[3] These bearings are similar to those in the High Temperature Alkali Metal Valve design shown in Figure 1. The bearings were replaced with self aligning ball bearings with two rows of balls rolling on the spherical surface of the outer race which could easily accommodate any misalignment between the bearing and the shaft. Each bearing consists of a VASCO Hypercut* inner and outer race with 0.125-inch diameter tungsten carbide balls.

Four ball bearings of this type were assembled, ultrasonically cleaned, and installed on the high temperature alkali metal valves. Subsequently the valve actuation systems were connected to the metering and isolation valves and operating torque tests were performed in air at room temperature. The valves were torqued to 72 in-lbs** in the fully closed positions to insure no slippage of the flexible shaft actuation

(3) Advanced Refractory Alloy Corrosion Loop Program, Quarterly Progress Report No. 12 for Period Ending July 15, 1968, NASA Contract NAS 3-6474, NASA-CR-72483.

*Nominal Composition: Fe-9.5Mo-8Co-3.75Cr-1.5W-1.15V-1.07C

**Maximum torque of the rotary magnetic feedthrough

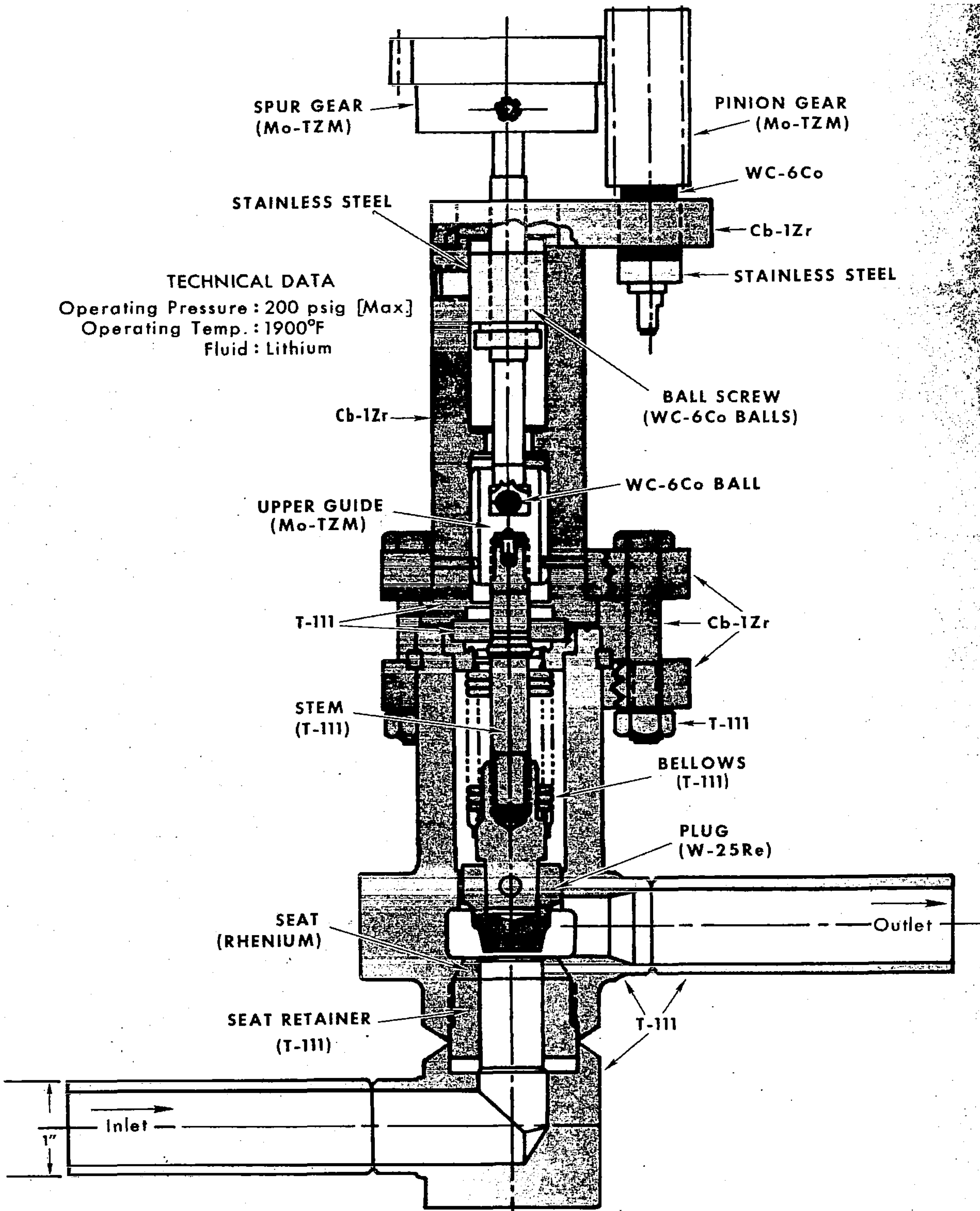

Figure 1. Preliminary Design of the High Temperature Alkali Metal Valve. Hoke Inc., Cresskill, New Jersey, Model 443X3. (C68011903)

systems before closing the chamber. With repeated cycling the operation of the valve became increasingly more difficult. The valve acutation systems were disassembled and the parts inspected. Severe wear was observed in the Mo-TZM ball screw and upper guide due to sliding friction between the two parts during the opening portion of the valve operation cycle.

Various approaches to solve this problem were considered, including surface hardening of the Mo-TZM parts and replacement of the Mo-TZM with hardened VASCO Hypercut. VASCO Hypercut in the hardened condition was selected as a replacement for Mo-TZM as a result of the trouble free performance of the VASCO Hypercut ball bearings on the pinion gear of the valve during the pretest checkout.

In this regard a thrust bearing design was adopted using VASCO Hypercut races and tungsten carbide balls as shown in Figure 2. Additional modifications to the valve were required as a result of the substitution of this iron base alloy for the refractory alloy previously used (Mo-TZM). The stem was increased in length to lower the temperature of the VASCO Hypercut parts by moving them farther away from the seat which would operate at a maximum temperature of 1900°F. The bonnet length was increased correspondingly and the extension was fabricated from VASCO Hypercut in the annealed condition.

A stem length of 5 inches was selected based on calculations which indicated a maximum temperature of 900°F in contact with the VASCO Hypercut lower thrust bearing. The results of these calculations are shown graphically in Figure 3. Weight loss tests of VASCO Hypercut in vacuum at temperatures as high as 1400°F indicated negligible weight losses ($< 0.05\%$ in 100 hours) and no vaporization problems were therefore anticipated in the proposed application.

As a result of the severe wear observed on the Mo-TZM pinion and spur gear teeth following 2500 hours of testing, described in Section VI of this report, an additional design modification was made. The Mo-TZM pinion and spur gears were replaced with VASCO Hypercut gears. These gears showed no signs of wear in the additional 2500 hours of valve testing. The final valve design is shown schematically in Figure 4 and the parts are shown before assembly in Figure 5.

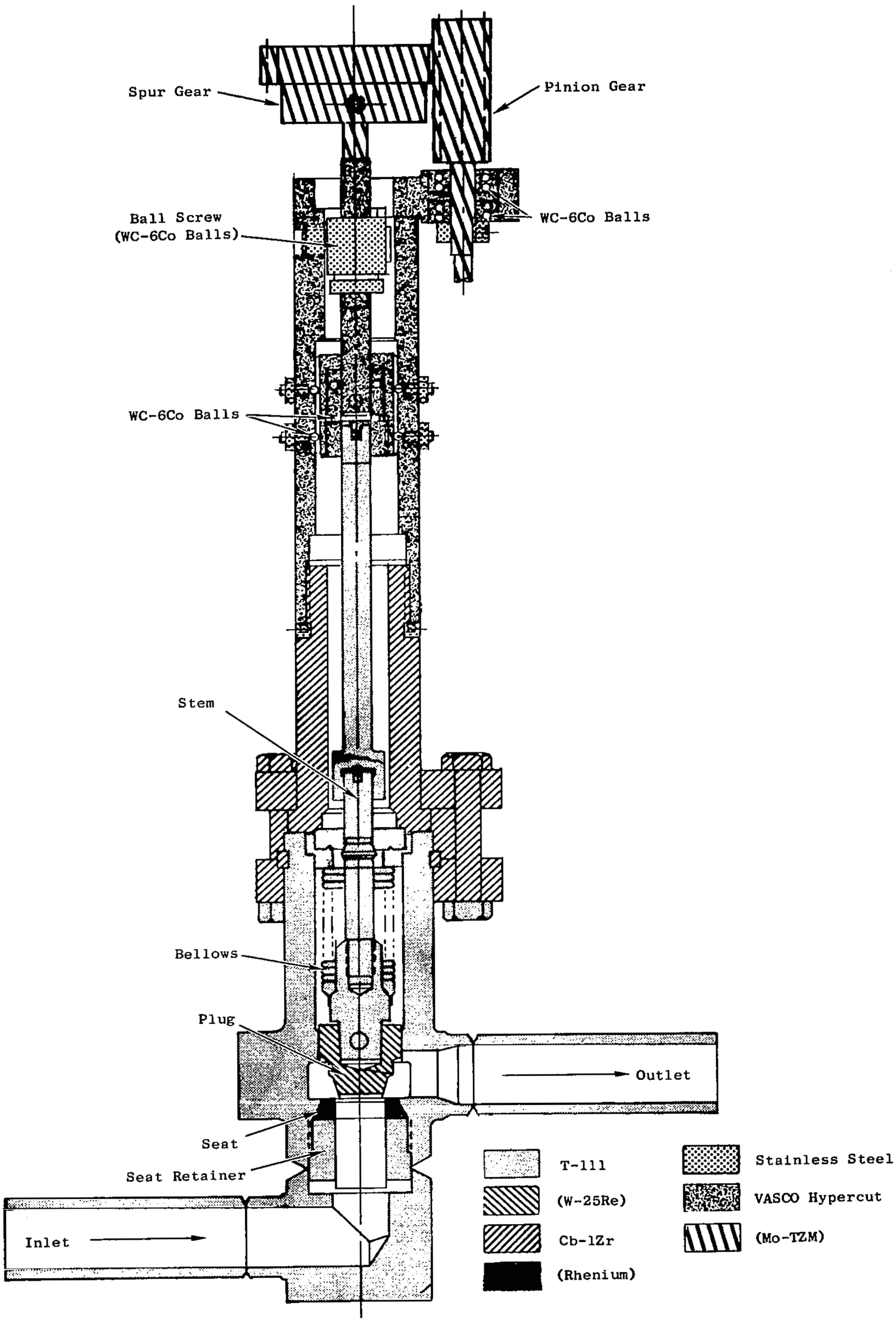

Figure 2. Intermediate Design of the High Temperature Alkali Metal Valve.

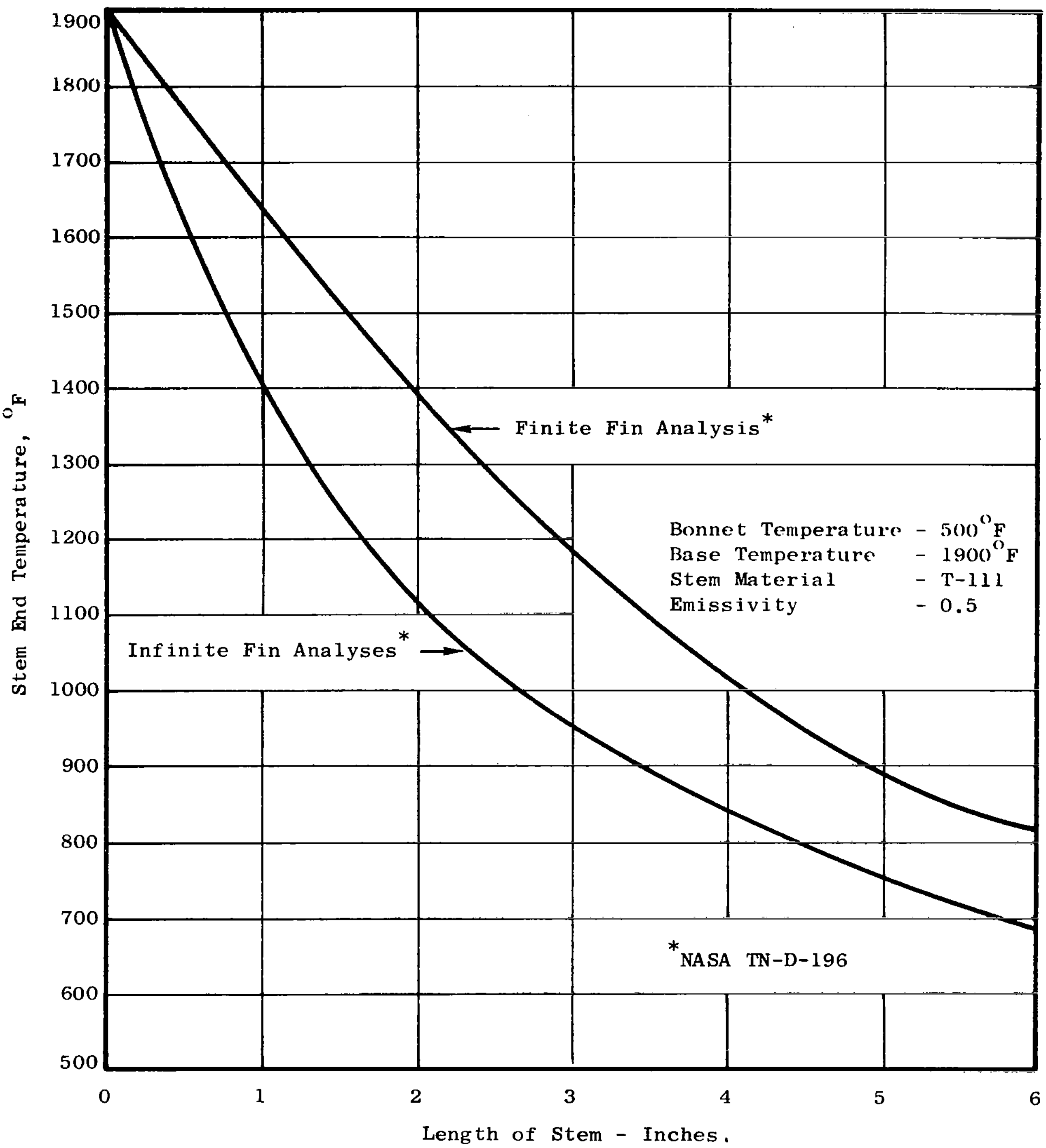

Figure 3. High Temperature Alkali Metal Valve Stem Temperature as a Function of Length. Stem Diameter 0.375-Inch.

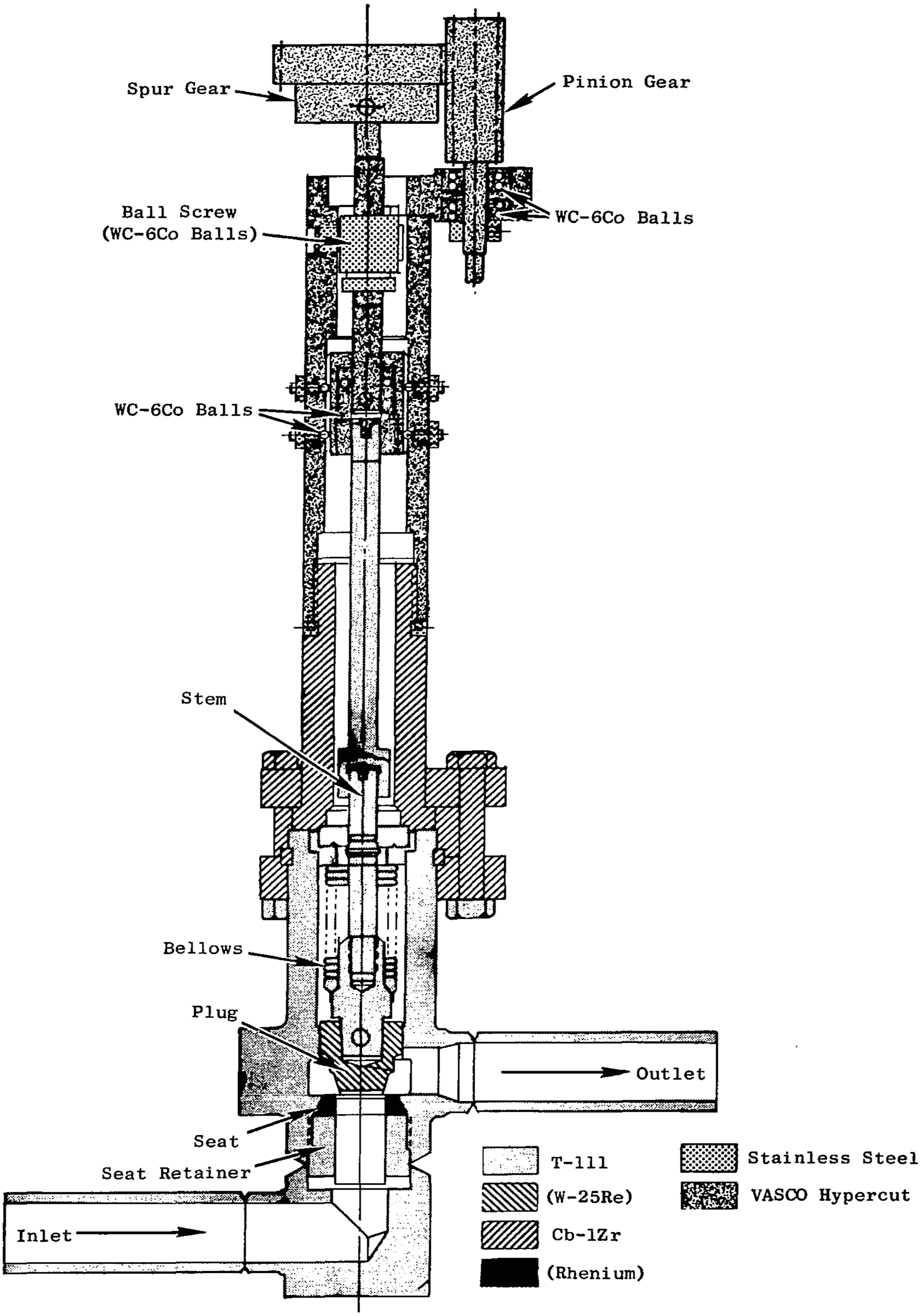

Figure 4. Final Design of the High Temperature Alkali Metal Valve.

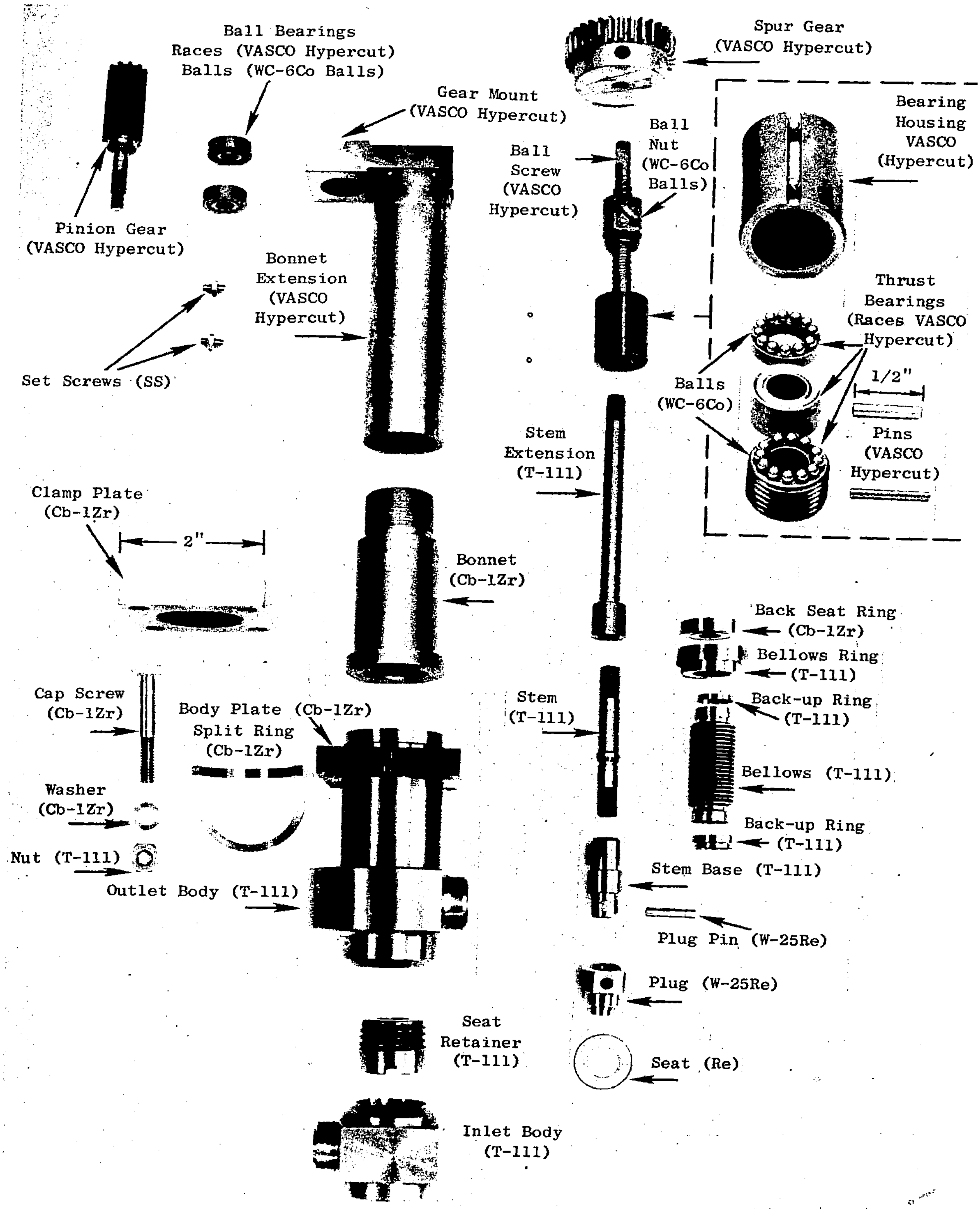

Figure 5. High Temperature Alkali Metal Valve Parts Before Assembly. (P69-8-15)

In addition to the schematic drawing of the valve shown in Figure 4 an assembly drawing and list of associated parts is included in Appendix B. The isolation valve is identical in design to the metering valve with the exception of a central hole drilled in the plug to allow flow to continue in the loop while the valve plug is seated.

B. LOOP DESIGN

The High Temperature Alkali Metal Valve Test Loop is shown schematically in Figure 6. The Cb-1Zr Pumped Sodium Loop[1] was reconstructed for this test with the inclusion of T-111 piping section containing the test valves and a Cb-1Zr tensile test specimen section containing sheet tensile specimens of T-111, T-222, ASTAR 811, ASTAR 811C, ASTAR 811CN and W-Re-Mo Alloy 256. These alloys, the compositions of which are presented in Table I, are under consideration for alkali metal containment in advanced space power systems. The specimen containment tube was grit blasted to increase its emissivity and thereby produce a 200°F temperature drop in the loop at the maximum operating temperature of 2100°F. Tensile specimens were exposed so that in addition to the determination of the corrosion resistance of these alloys, the effects of the lithium exposure on their mechanical properties could be ascertained. Three Cb-1Zr slack diaphragm pressure transducers were included in the loop circuit to obtain the operating characteristics of the EM pump and the pressure drop across the 1-inch metering valve during loop operation.

[1] Hoffman, E. E. and Holowach, J., Cb-1Zr Pumped Sodium Loop, NASA Contractor Report, NASA-CR-1135, November 1968.

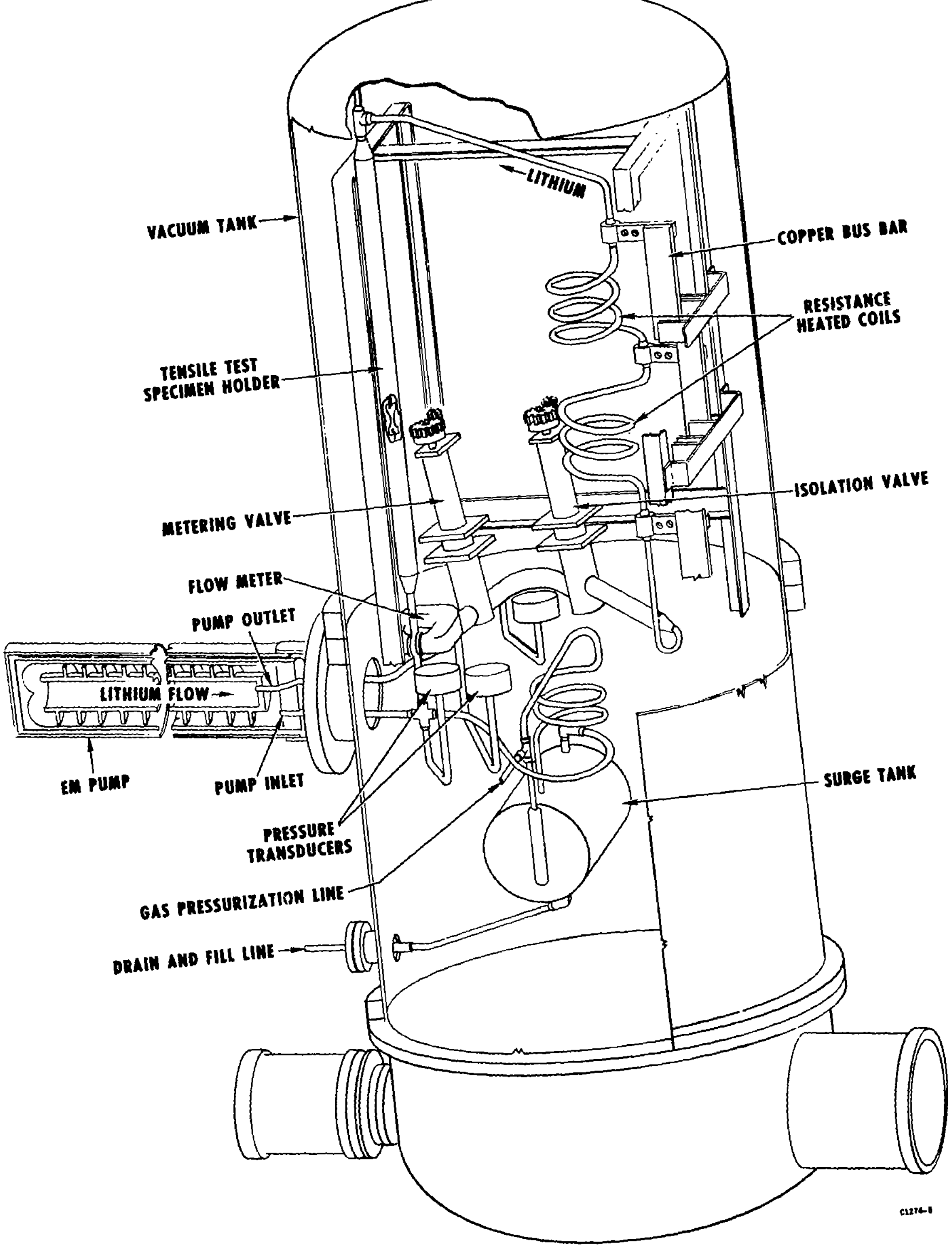

Figure 6. High Temperature Alkali Metal Valve Test Loop

TABLE I

THE COMPOSITION OF ADVANCED REFRACTORY ALLOYS
INCLUDED IN THE TENSILE TEST SPECIMEN SECTION
OF THE HIGH TEMPERATURE ALKALI METAL VALVE TEST LOOP

Alloy	Nominal Composition (Weight Percent)
T-111	Ta-8W-2Hf
T-222	Ta-9.6W-2.4Hf-.01C
ASTAR 811	Ta-8W-1Hf-1Re
ASTAR 811C	Ta-8W-0.7Hf-1Re-.025C
ASTAR 811CN	Ta-8W-1Hf-1Re-.012C-.012N
W-Re-Mo Alloy 256	W-29Re-18Mo

III. MATERIALS

A. Valve Materials Selection

The selection of materials for the valve seat and plug required special consideration because of the planned high temperature operation. The valves in the T-111 Rankine System Corrosion Test Loop, NASA Contract NAS 3-6474, operated at a maximum temperature of 1320°F, and employed a Mo-TZM plug and T-111 seat. While adequate from an alkali metal compatibility standpoint, these materials were deemed inadequate to prevent bonding of the seat and plug in the isolation valve under the applied torque (1-ft lb) during long term testing at a maximum temperature of 1900°F. Materials requirements for valves for alkali metal service are similar to those requirements for bearings, namely, alkali metal compatibility and resistance to diffusion bonding. Results from a NASA sponsored program at GE-NSP, "Materials for Potassium Lubricated Journal Bearings," NASA Contract NAS 3-2534 indicated excellent properties of TiC-10Cb.[4] This material was selected as the seat material with W-25Re as the plug. This tungsten alloy has all the advantages of tungsten in resistance to diffusion bonding but is more ductile and more easily machined.

Although TiC-10Cb had previously demonstrated corrosion resistance to potassium,[4] the TiC-10Cb valve seat material showed poor resistance to lithium in isothermal capsule tests,* and unalloyed rhenium was selected as a replacement. Rhenium was selected because of its resistance to diffusion bonding as indicated by its high melting point (5733°F), its room temperature ductility,[5] and its reported[6,7] corrosion resistance to lithium to 2500°F. Subsequent capsule tests at GE-NSP indicate rhenium to be corrosion resistant to lithium at 2100°F.*

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(4) Materials for Potassium Lubricated Journal Bearings, Final Report, NASA Contract NAS 3-2534, GESP-100, September 1966.

(5) Sims, Chester F.,"Properties of Rhenium," Rhenium, Edited by B. W. Gonser, Elsevier Publishing Company, New York, 1962, p. 33.

(6) Hoffman, E.E.,"Corrosion of Materials by Lithium at Elevated Temperatures, ORNL-2674, March 6, 1959, p. 111.

(7) DeMastry, J.A. and Griesenauer, N.M.,"Investigation of High-Temperature Refractory Metals and Alloys for Thermionic Converters, AFPL-TR-65-29, April 1965, p. 26.

*Described in Appendix A.

VASCO Hypercut is a cobalt-type high speed steel capable of being heat treated to Rockwell C-70. It is easily machined in the annealed condition and can be finished by grinding. Its high hardness and good impact strength make this alloy attractive for use as a bearing. The nominal composition of this alloy procured from Vanadium Alloys Steel Company, Latrobe, Pennsylvania, is presented in Table II.

B. 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 producers. 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 material 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 exceeded 500°F.

A listing of the refractory metal alloys and cermets procured for the program, the specifications to which they were procured, and the results of the quality assurance tests are presented in Appendix C.

C. Lithium Procurement and Purification

Fifty pounds of LCA High-Purity Lithium was obtained from the Lithium Corporation of America, Bessemer City, North Carolina. 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

TABLE II

NOMINAL COMPOSITION OF VASCO HYPERCUT[a]

Element	Weight Percent
Iron	74.50
Molybdenum	9.50
Cobalt	8.00
Chromium	3.75
Tungsten	1.50
Vanadium	1.15
Carbon	1.07
Manganese	0.22
Silicon	0.22

[a] Vanadium Alloys Steel Company, Latrobe, Pa.

lined, stainless steel hot trap containing a zirconium getter bundle. The hot trap is shown in position in the purification facility in Figure 7 and before assembly in Figure 8. The lithium was subsequently hot trapped at 1500°F for 100 hours. The principal result of the hot trapping operation is the reduction in nitrogen concentration of the lithium. The final purification step was vacuum distillation at 1250°F. The assembled lithium still before installation in the purification facility is shown in Figure 9. The distillation step reduces the oxygen and metallic impurity concentrations. Chemical analysis of the lithium after each purification step is compared with the analysis of the as-received material in Table III. The slightly higher nitrogen and carbon concentrations in the lithium following distillation are within the accuracy of the analytical technique employed in the analyses.

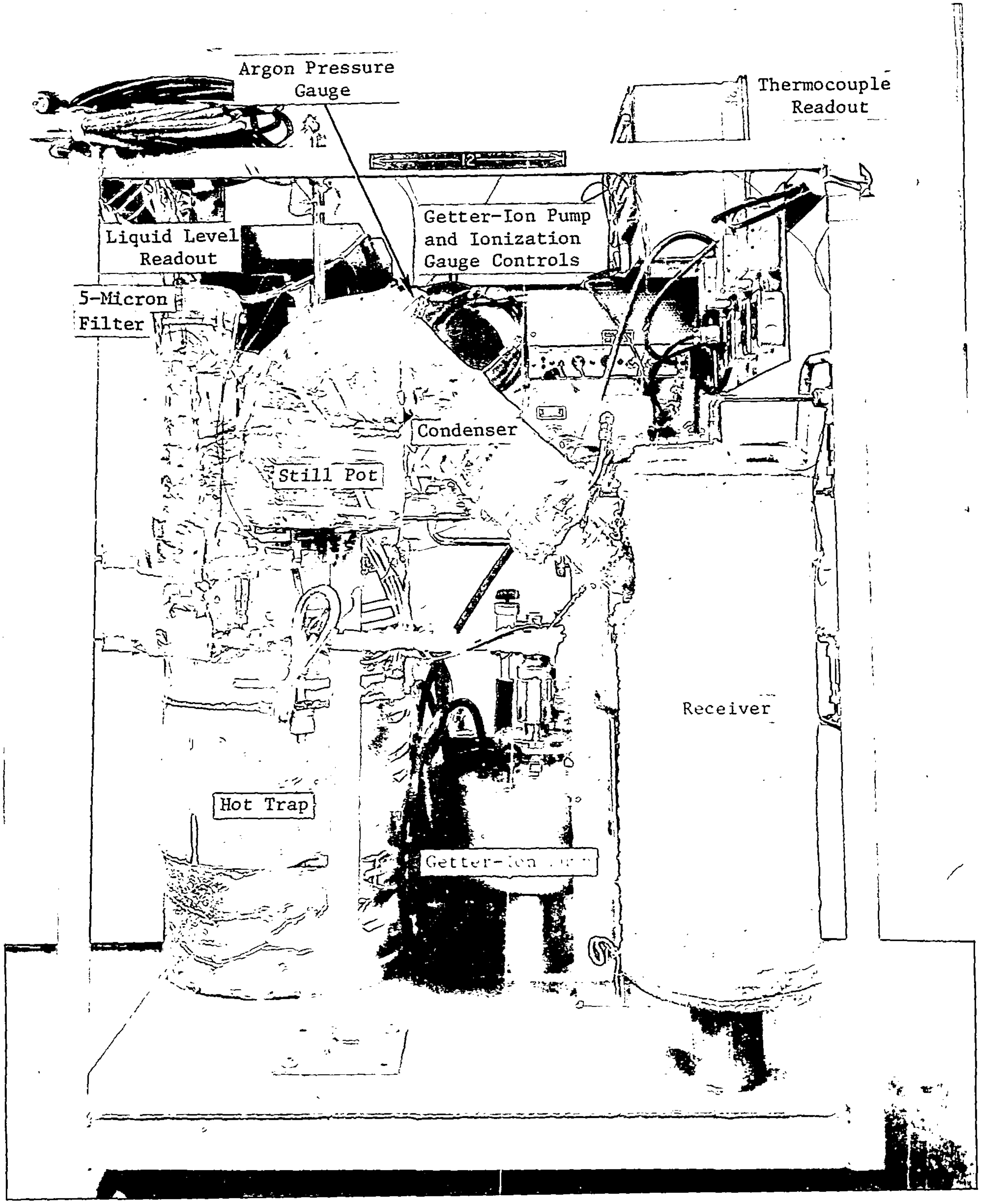


Figure 7. The Lithium Purification Facility. (Orig. C68022909)

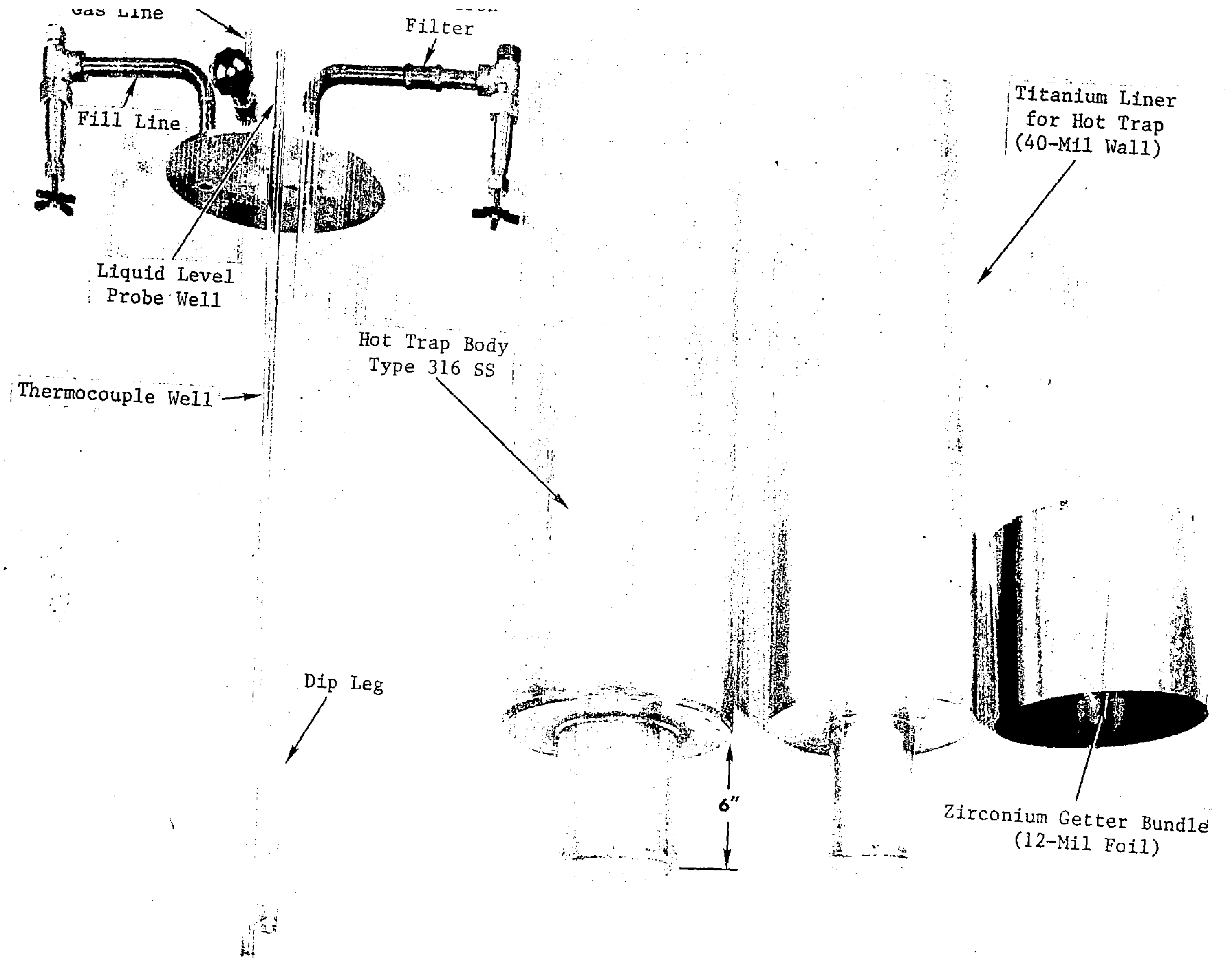


Figure 8. The Lithium Hot Trap Prior to Assembly. (C65021019)

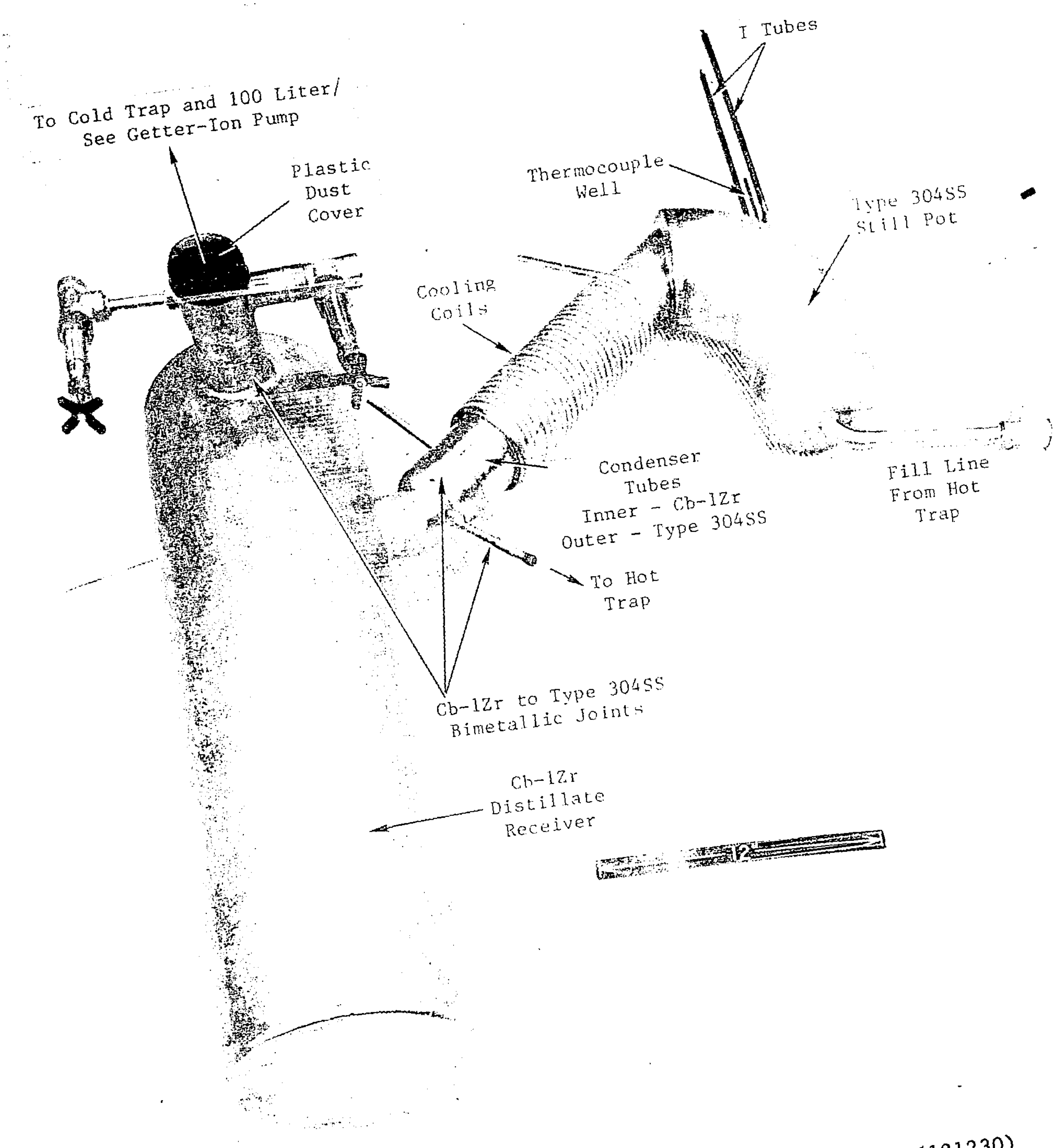


Figure 9. The Lithium Still After Assembly. (C66121230)

TABLE III

LITHIUM ANALYSIS

Element	Concentration, ppm in Lithium[a] As-Received	Filtered	Hot Trapped		Distilled	
O	130 ± 25[b]	150 ± 28	-		13[e]	
N	840[c]	775	11		20	
C	146[d]	98	34		67	
	Spectrographic Analysis					
			GE	NSL[f]	GE	NSL
Ag	< 5	< 5	< 5	ND	5	ND
Al	< 5	< 5	< 5	< 5	5	< 25
B	ND[g]	-	< 50	ND	< 50	ND
Ba	-	-	< 75	ND	< 50	ND
Be	< 5	< 5	< 5	-	< 5	ND
Ca	133	53	25	55	< 5	20
Cb	< 25	< 25	< 25	ND	< 25	ND
Co	< 5	< 5	< 5	ND	< 5	ND
A	< 5	< 5	< 5	800	< 5	< 25
Cu	< 5	< 5	50	50	5	< 5
Fe	< 5	< 5	5	150	25	20
K	-	-	-	< 50	-	< 50
Mg	5	5	< 5	< 50	< 5	< 500
Mn	< 5	< 5	< 5	ND	< 25	ND
Mo	< 5	< 5	< 5	ND	< 5	ND
Na	53	133	< 75	< 50	< 50	< 500
Ni	< 5	< 5	25	1550	< 5	< 25
Pb	< 25	< 25	< 75	< 100	< 75	ND
Si	5	5	25	100	< 25	< 25
Sn	< 25	< 25	< 25	ND	< 25	ND
Sr	-	-	5	< 50	< 5	ND
Ti	< 5	< 5	< 25	ND	< 5	ND
V	< 25	< 25	-	ND	< 25	ND
W	-	-	-	ND	-	ND
Zn	-	-	-	ND	-	ND
Zr	< 5	< 5	< 25	ND	< 25	ND

(a) The lithium sample is extruded, reacted with water in platinum and converted to Li_2CO_3 by bubbling CO_2 through the solution. The dried Li_2CO_3 is blended and split between analytical laboratories. Concentrations are calculated for the metal.
(b) Fast neutron activation analysis by General Atomic, San Diego, California.
(c) Micro-Kjeldahl analysis.
(d) Combustion - Gas Chromatograph analysis.
(e) Vacuum distillation method.
(f) National Spectrographic Laboratory.
(g) Not Detected.

IV. VALVE TEST LOOP FABRICATION

A. Bellows Fabrication

The T-111 valve bellows were fabricated from 0.625-inch OD T-111 tubes with a 0.008-inch wall produced at Superior Tube Company, Norristown, Pennsylvania. All tubes were ultrasonically tested at Automation Industries, Columbus, Ohio in both the longitudinal and circumferential shear modes with a 0.001-inch notch as the standard defect. Twenty-six of the 62 tubes purchased were free of any defect indications within the limit of detection of the ultrasonic test equipment and were selected for forming into bellows.

Twenty-four bellows, one of which is shown in Figure 10 were hydraulically formed with an internal pressure of 2400 psi by Miniflex Corporation, Van Nuys, California. Attempts by Miniflex Corporation to form the bellows 0.930-inch OD originally specified were not successful due to grain boundary separation as a result of severe overdrawing at the outer convolution surface. No bellows of this diameter could be produced without leaks. The forming apparatus was reset for a smaller diameter, 0.850-inch OD, bellows and the operation proceeded without difficulty. The "orange peel" surface typical of materials stressed beyond their elastic limit is still apparent on the bellows of the lesser diameter shown in Figure 11; however, it was not as coarse, and no grain boundary separation was observed in the 0.850 inch diameter bellows. Although the outer diameter specification was reduced from 0.930 inch to 0.850 inch, the total linear deformation of the formed bellows before permanent set occurred was 0.23 inch. This exceeded the operation requirement originally established by Hoke, Inc. for the 1-inch valve.

Crack propagation of T-111 alloy originating at surfaces cut by an abrasive wheel were experienced in forming the bellows neck. During the forming operation where the diameter of the bellows neck is increased from 0.625 to 0.650 inch, cracks originating at the tube ends of the bellows, which had been cut by an abrasive wheel, propagated into the end convolution. The problem was eliminated by grinding back and polishing the ends before forming. Similar results were observed in processing

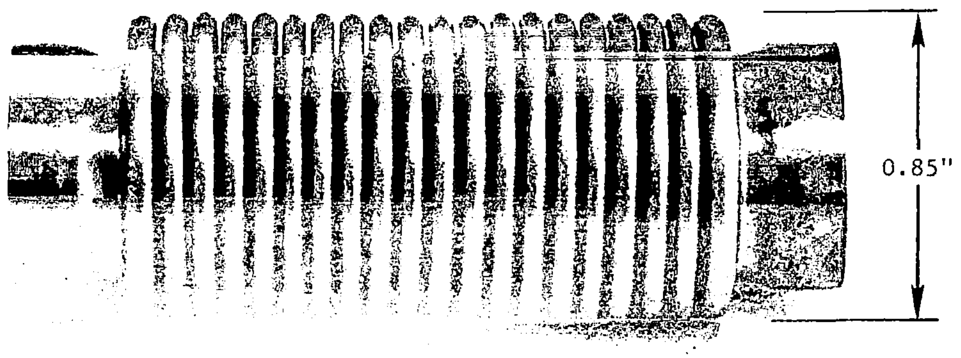


Figure 10. T-111 Bellows (0.85 Inch OD x 0.59 Inch ID) Formed by Mini-Flex Corporation. (C67112004)

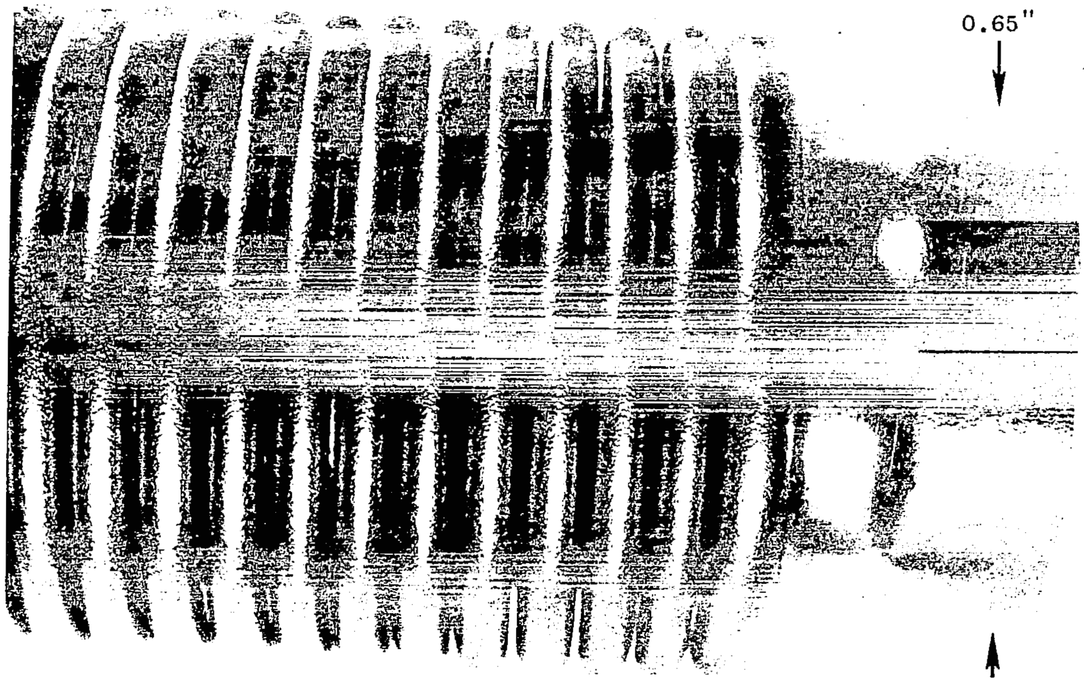


Figure 11. Surface of T-111 Bellows Convolutions Formed by Mini-Flex Corporation. The Outside Diameter of the Convolutions is 0.85 inches. Attempts to Form Larger Diameter Bellows were Unsuccessful as a Result of Grain Boundary Separation from Overdrawing. (C68011538)

T-111 tubes for various NSP programs.[8]

The bellows, shown previously in Figure 10, have 20 convolutions with a 0.850-inch OD and 0.590-inch ID with an average overall length of 2.2 inches.

Various additional inspections performed by GE-NSP at the time indicated that the bellows were suitable for use in the fabrication of valves for the loop. However, more detailed metallographic inspection of additional specimens cut from several of the bellows which were destructively evaluated revealed several shallow tears most often located on the outer surface of the OD convolution. The maximum depth of surface tearing observed was one mil. Since less than 25% of the surfaces of the formed bellows could be visually inspected at low magnification (30x), additional qualification testing was initiated.

A bellows cycling test to simulate the life service of the bellows was conducted, in a special test fixture shown in Figure 12, to determine if the tears in selected bellows would propagate during mechanical cycling. Normally, the bellows is installed in the valve so that it is at its normal free length when the valve is in the closed position; opening of the valve compresses the bellows. However, due to stack-up of normal shop tolerances in the valve parts, the bellows slightly extended from its normal free length during assembly. The maximum operating compression stroke of the valve is 0.220-inch although only 0.125-inch travel is required to lift the plug completely out of the seat.

A bellows with the largest defects that could be detected by a 30x visual inspection was selected and cycled 50 times with a stroke of ± 0.125-inch. The bellows was helium leak checked after 5, 10, 25 and 50 cycles with no indication of leaks. A microscopic examination of the bellows following cycling showed no propagation of the tears. The test was repeated for an additional 50 cycles with a maximum compression stroke of 0.220-inch. The bellows was again leak tested

[8] Ekvall, R. A., Frank, R. G., Young, W. R., "T-111 Alloy Cracking Problems During Processing and Fabrication," Ch. VIII in Recent Advances in Refractory Alloys for Space Power Systems, NASA SP-245, 1970.

with no indication of leaks and microscopic examination showed no propagation of the tears. The manually operated bellows cycling test fixture is shown in Figure 12.

A second bellows, also with detectable tears, was annealed for 1 hour at 2400°F before being mechanically cycled as described above. Again the bellows showed no propagation of tears and was helium leak tight after the 100 cycles.

Although the above cycling test indicated that the valve bellows would operate successfully in service, it was desirable for maximum reliability to produce bellows without any visible surface defects. It was hoped that careful polishing of the tubes would remove even minute surface defects that could develop into tears as the result of the strain induced in the tube walls during forming of the bellows convolutions.

Arrangements were made with Mini-Flex Corporation for the forming of 15 additional bellows with GE-NSP responsible for the intermediate inspection and the final sizing to length of the bellows. Nine, 15-inch long T-111 tubes which had passed ultrasonic inspection without any known defects were polished with diamond paste. The ID of the tubes, which appeared smoother than the OD of the tubes, was polished for 40 minutes with 6-micron diamond paste. The OD of the tube was polished for 30 minutes with 15-micron diamond paste followed by 15 minutes of polishing with 6-micron diamond paste. The improvement in the smoothness of the ID and OD surfaces of the tubes as a result of diamond polishing is evident in Figure 13.

The polished bellows tube blanks were formed into 15 bellows. By obtaining the bellows from Mini-Flex prior to the final sizing operation, which reduces the bellows length from 3-3/4-inches to 2-1/8-inches, it was possible to perform a more detailed visual inspection (30x) at the bellows surface between the convolutions. This inspection revealed no tears of the type observed in the first lot of bellows.

A test was also performed to determine if an intermediate anneal (1 hour at 2400°F) prior to the final sizing operation would improve

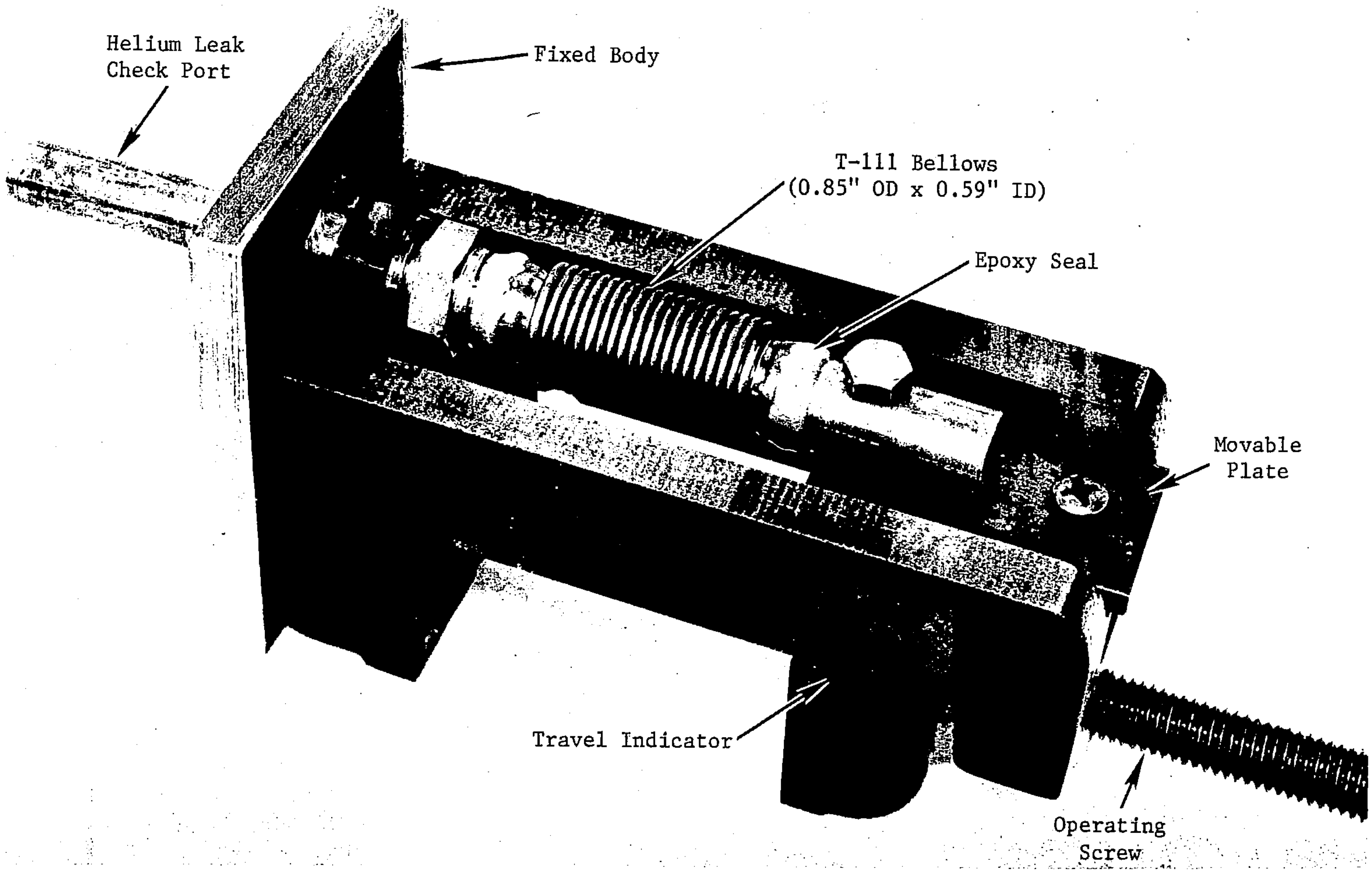
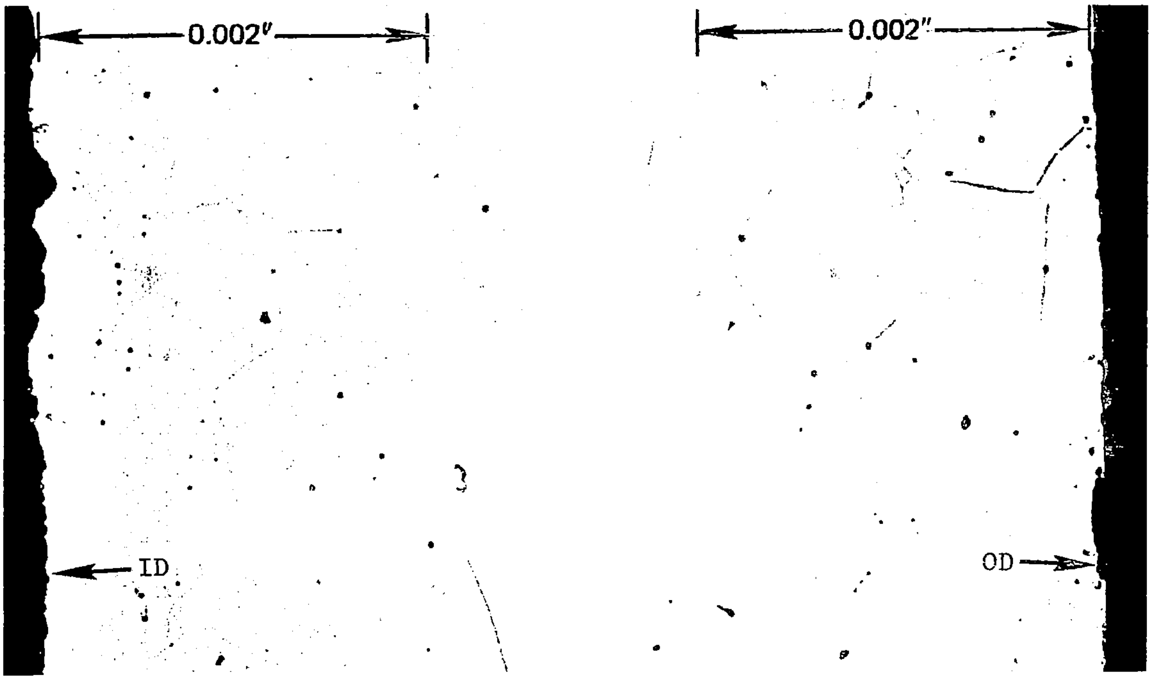
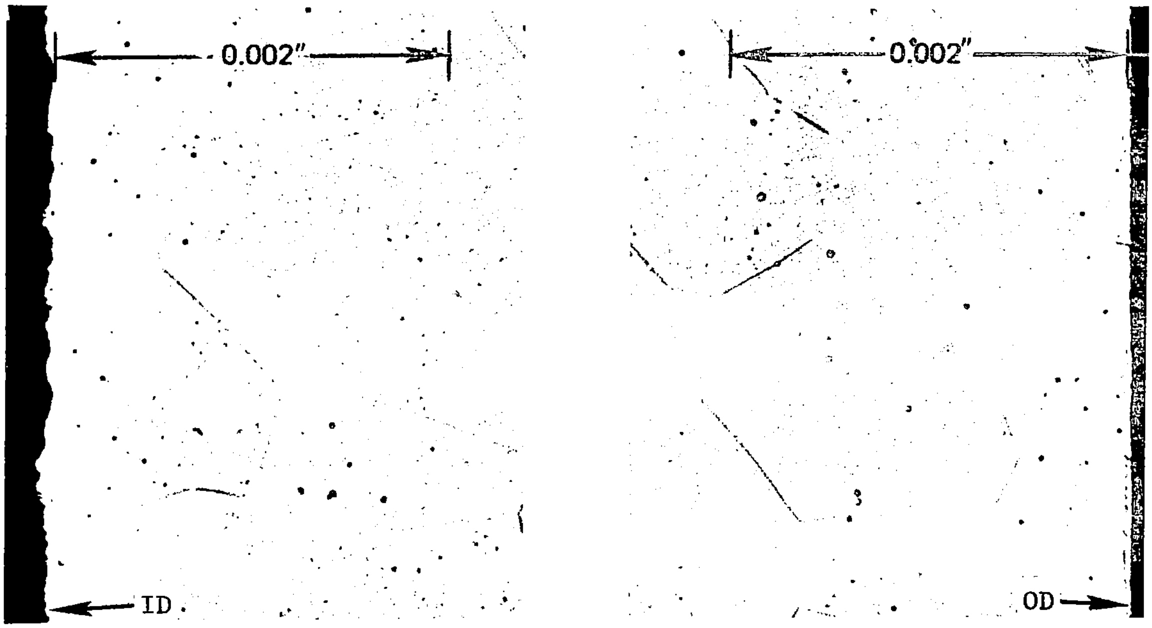


Figure 12. Mechanical Cycling Test Fixture for the T-111 Bellows. (C67120719)



(a) Before Diamond Polishing (b)



(c) Following Diamond Polishing (d)

Figure 13. Metallographic Appearance of ID and OD of Transverse Sections of the T-111 Bellows Tubing (0.625 Inch OD x 0.0085 Inch Wall) in the As-Received Condition (top) and Following Polishing with Diamond Paste (lower).
Etchant: $30gmNH_4F-50mlHNO_3-20mlH_2O$ a) F120113 c) F210111
b) F120114 d) F210112

the surface quality of the bellows resulting from the final sizing operation. The annealed bellows and a second bellows which was not heat treated were sized and compared. The appearance of the unannealed bellows before and after the sizing operation is illustrated in Figure 14. Annealing of the bellows caused a marked change in the collapsing mode of the bellows during sizing and resulted in an overall bellows length 1/8-inch shorter than the design length of 2.18 inches. Low power visual inspection (30x) and metallographic examination of the two types of bellows following the sizing operation indicated that the unannealed bellows had a higher quality surface than the annealed bellows.

Based on these results, bellows from the second (polished) lot were sized without an intermediate anneal. All of the sized bellows were free of leaks and three with no visual defects were selected for use in the construction of the high temperature valves. The metallographic appearance of the OD and ID regions of the T-111 bellows of the type selected for use in the valves is illustrated in Figure 15. The OD region is slightly harder and the wall is one mil thinner than the ID region of the bellows.

B. Other Valve Parts

An exploded view of the actual valve parts prior to the initiation of welding of the various components is given in Figure 16. An enlarged view of the W-25Re plug and the rhenium seat of the metering valve are shown in Figure 17. The plug of the by-pass valve to be used in determining the solid phase bonding characteristics of the plug and seat materials is identical to the metering plug except that the by-pass plug has 0.187-inch hole through the plug. Both plugs are shown in Figure 18 which describes the assembly of a valve plug on an EB welded bellows stem assembly.

C. Loop and Valve Fabrication

Tensile test specimens of T-111, T-222, ASTAR 811, ASTAR 811C, ASTAR 811CN and W-Re-Mo Alloy 256 for use as control and loop specimens

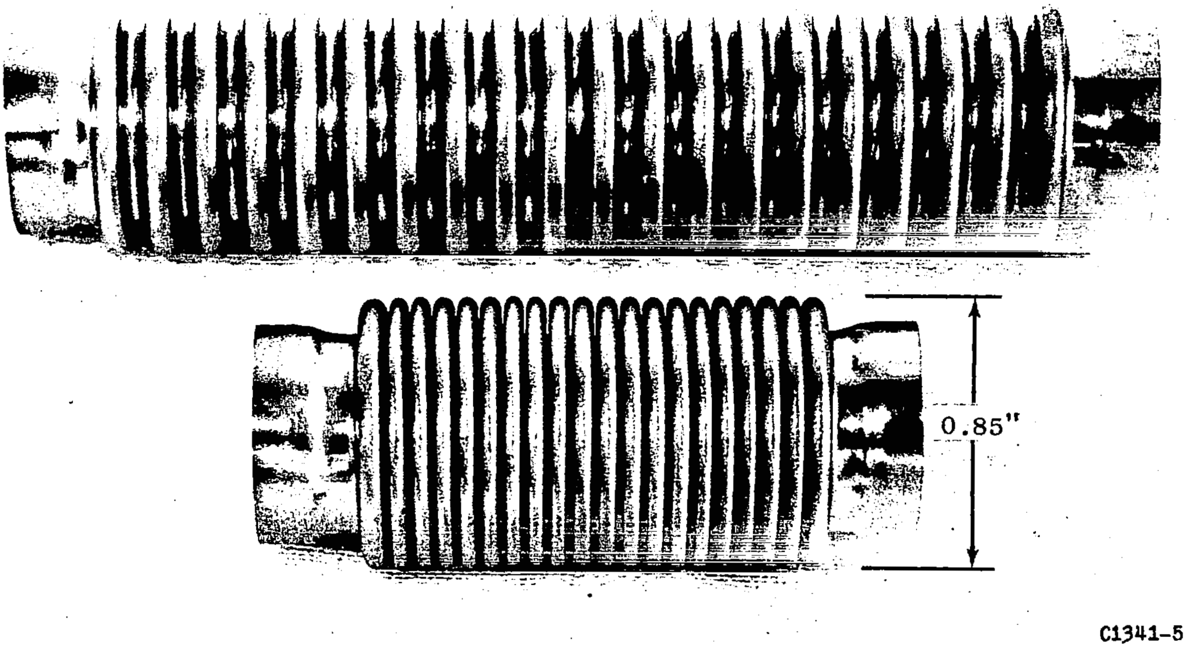


Figure 14. T-111 Bellows (0.85 Inch OD x 0.59 Inch ID x 0.008 Inch Wall), (top) Following the Initial Forming Operation by Mini-Flex Corporation and (lower) Following the Final Sizing Operation by GE-NSP. (C68010914)

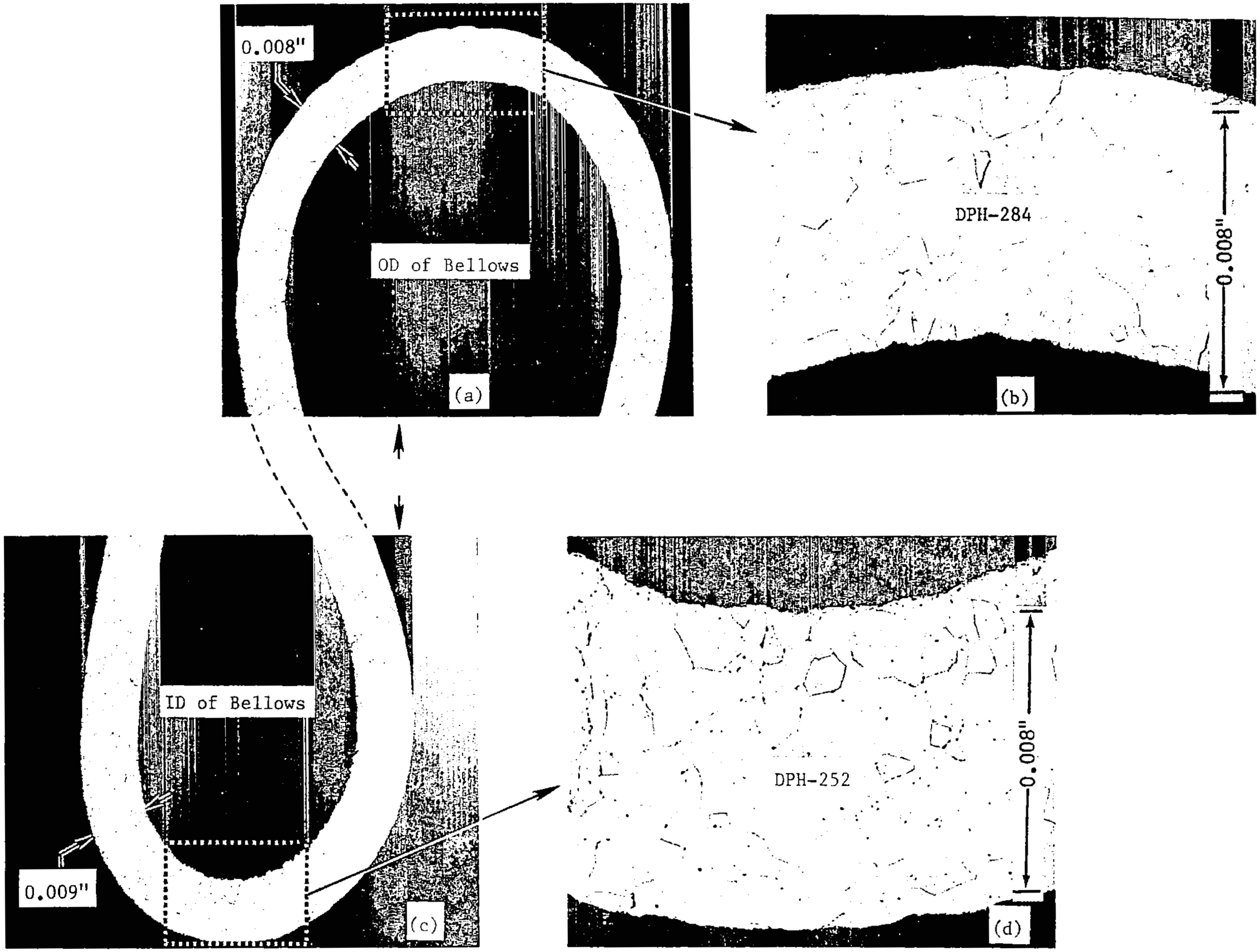


Figure 15. Microstructure of the OD and ID Regions of a T-111 Bellows of the Type to be Used in the Fabrication of the Valves for the High Temperature Alkali Metal Valve Loop. Etchant: 30gmNH_4F-50mlHNO_3-20mlH_2O a) 50X (F240213) c) 50X (F240214) b) 250X (F240216) d) 250X (F240215)

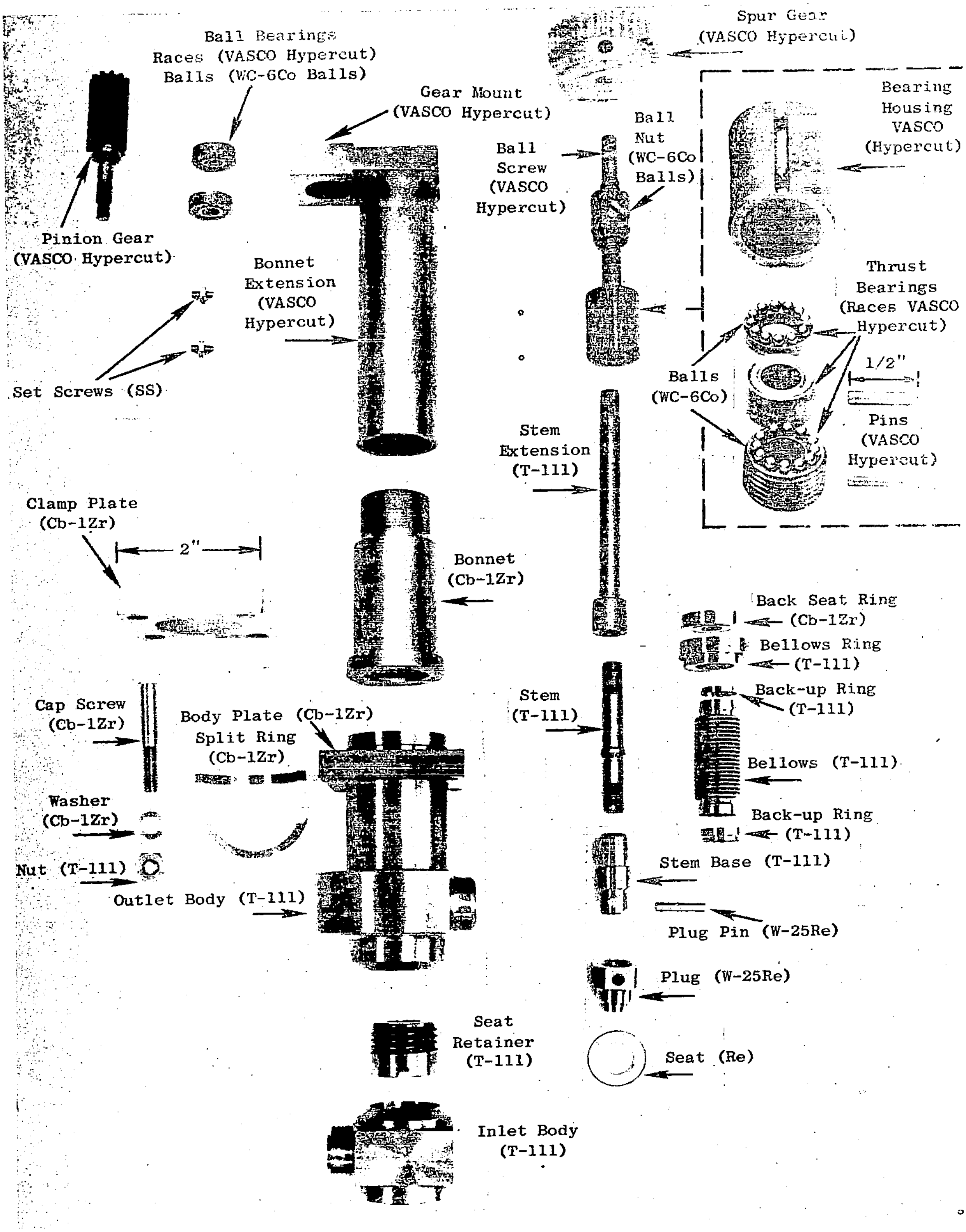


Figure 16. High Temperature Alkali Metal Valve Parts Before Assembly. (P69-8-15)

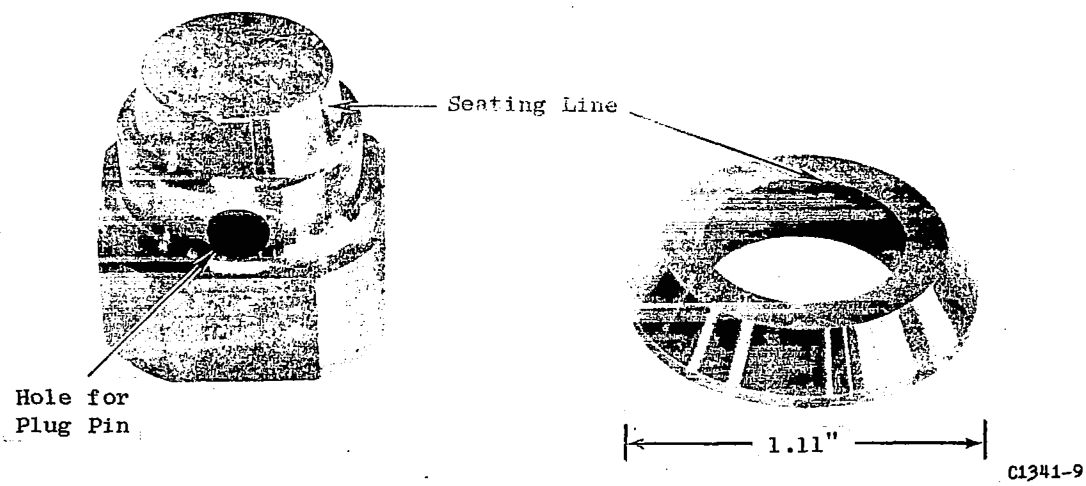


Figure 17. W-25Re Plug and Rhenium Seat of the 1-Inch Refractory Metal High Temperature Alkali Metal Valve. (C67112230)

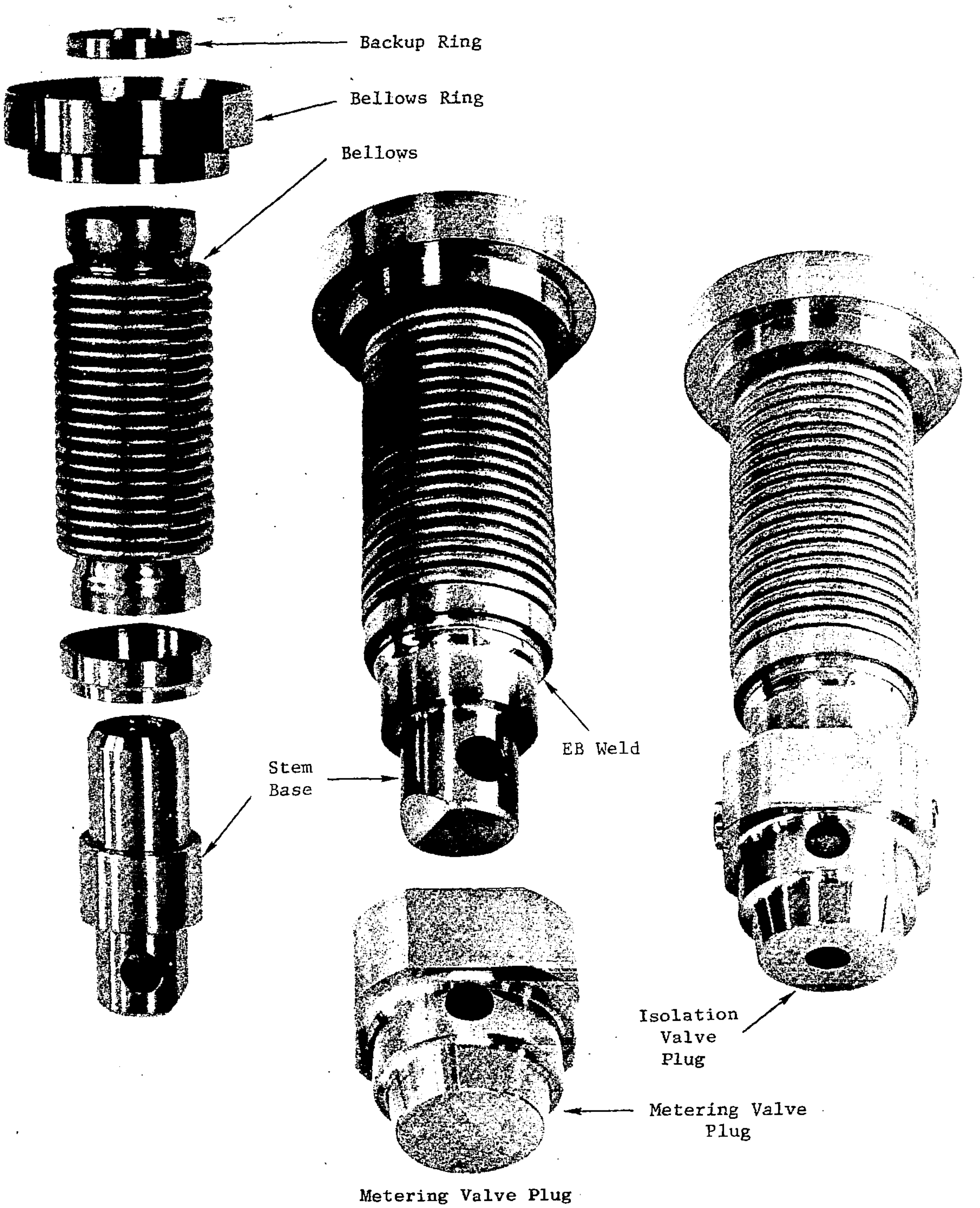


Figure 18. Valve Plug, Stem Base and Bellows Assembly. (C67090653, C680131153, C680131154 & C67112230)

were machined, inspected and weighed. The tensile test specimens were assembled and inserted into the specimen holder and welded onto the heater completing this section as shown in Figure 19. This subassembly and the surge tank assembly, shown in Figure 20, were annealed for one hour at 2200°F in the Abar-90 furnace. The heat treated components were wrapped with one layer of Cb-1Zr foil in accordance with NSP Specification 03-0037-00-A.

Three Pt/Pt-10 Rh thermocouples were positioned at various elevations inside the Cb-1Zr retort of the annealing furnace to determine the uniformity of the temperature of the loop parts. The temperature of the loop parts was maintained at 2200°F ± 10°F during the one-hour anneal. The results of chemical analysis of a T-111 specimen that was annealed with the loop components are given in Table IV and indicate that no significant increase in interstitial elements occurred during annealing.

The valve subassembly which consists of the two valves and two slack diaphragm pressure transducers is shown in Figure 21. Since both T-111 and Cb-1Zr alloy components were involved, two postweld annealing temperatures were required. The T-111 valves and associated T-111 tubing were furnace annealed at 2400°F for one hour. The results of the furnace qualification per NSP Specification 03-0037-00-A are shown in Table V. During the actual furnace run, the pressure varied from 2.2×10^{-5} torr at the start to 9.1×10^{-6} torr at the end of the run.

The Cb-1Zr slack diaphragm transducers and Cb-1Zr tubing for the entrance and exit of the valves were then welded into the assembly. These welds were annealed individually at 2200°F for one hour using heaters positioned over the weld area. The pressure within the welding chamber during these runs never exceeded 1×10^{-5} torr.

The final assembly of the High Temperature Alkali Metal Valve Test Loop started with the positioning of the subassemblies in the loop support structure as shown in Figure 22. The valve subassembly is shown in its final position in Figure 23. After the correct orientation of each subassembly and the overall alignment of the loop had been verified, the loop was removed from the vacuum chamber spool section in its support

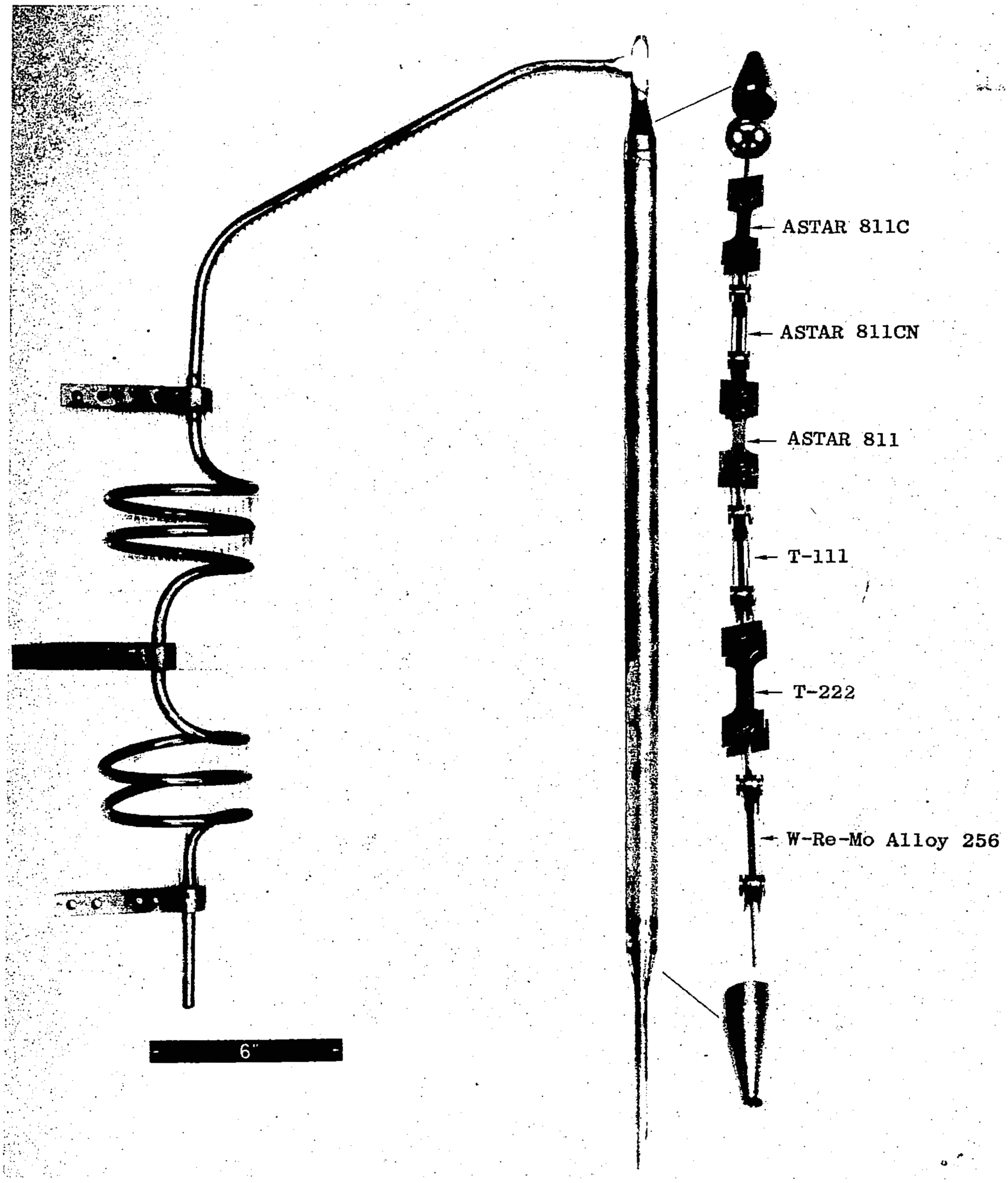


Figure 19. Cb-1Zr Heater and Tensile Specimen Holder Sections of the High Temperature Alkali Metal Valve Test Loop. Stringer of Refractory Alloy Specimens Shown on Right. (C67071835)

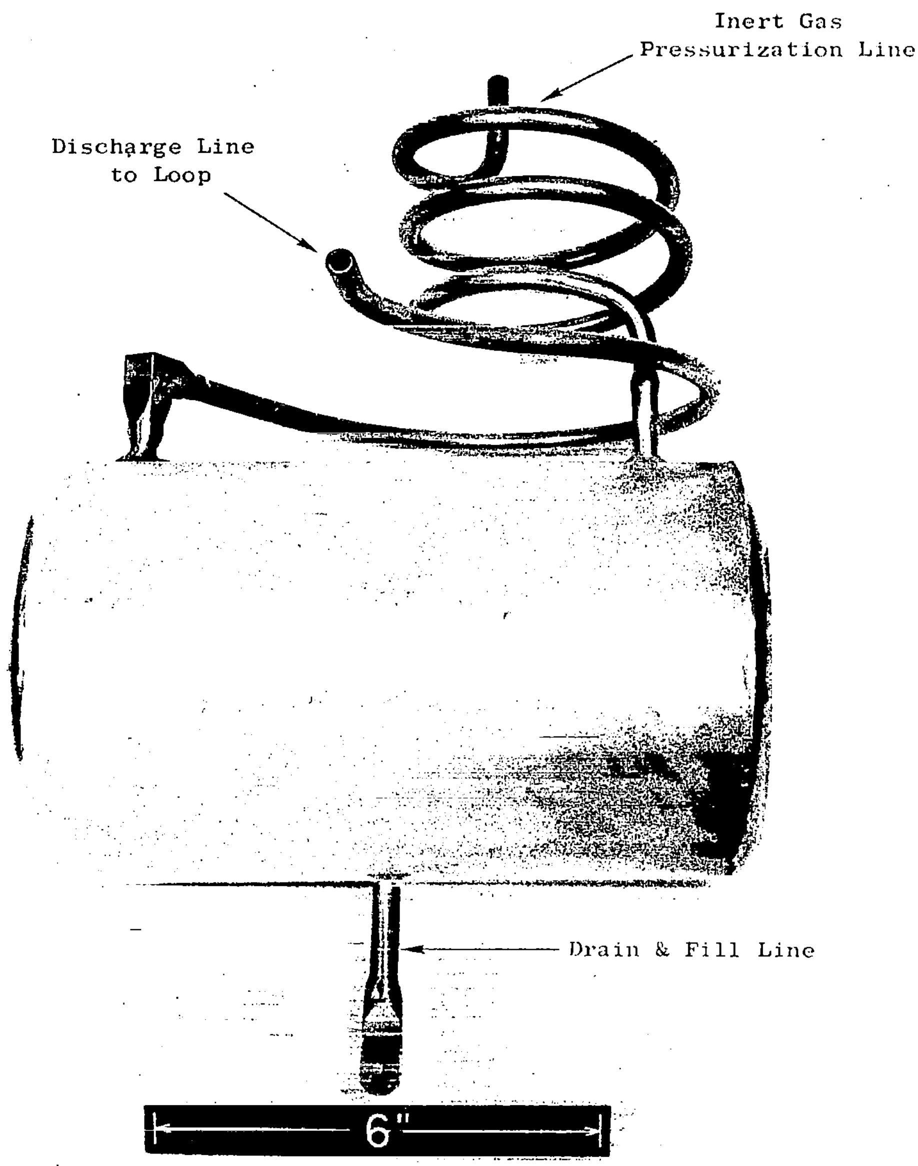


Figure 20. Cb-1Zr Surge Tank Assembly. (C67071831)

TABLE IV

CHEMICAL ANALYSIS OF T-111 SHEET SPECIMEN FOLLOWING HEAT TREATMENT[(a)] OF HIGH-TEMPERATURE ALKALI METAL VALVE LOOP COMPONENTS

Element	Concentration, ppm[(b)]	
	Before Anneal	Wrapped Specimen[(c)] After Anneal
O	65	73, 74
N	5	7, 9
H	1	1, 1
C	10	14, 20

(a) One hour at 2200°F vacuum heat treatment performed in Abar-90 furnace at Stellite - UCC (now Cabot Corporation) maximum pressure 1×10^{-5} torr.

(b) Analytical Methods: O, N, and H - Vacuum Fusion
C - *Combustion Conductometric*

(c) 0.040-inch-thick specimen wrapped with one layer of 0.002-inch-thick Cb-1Zr foil.

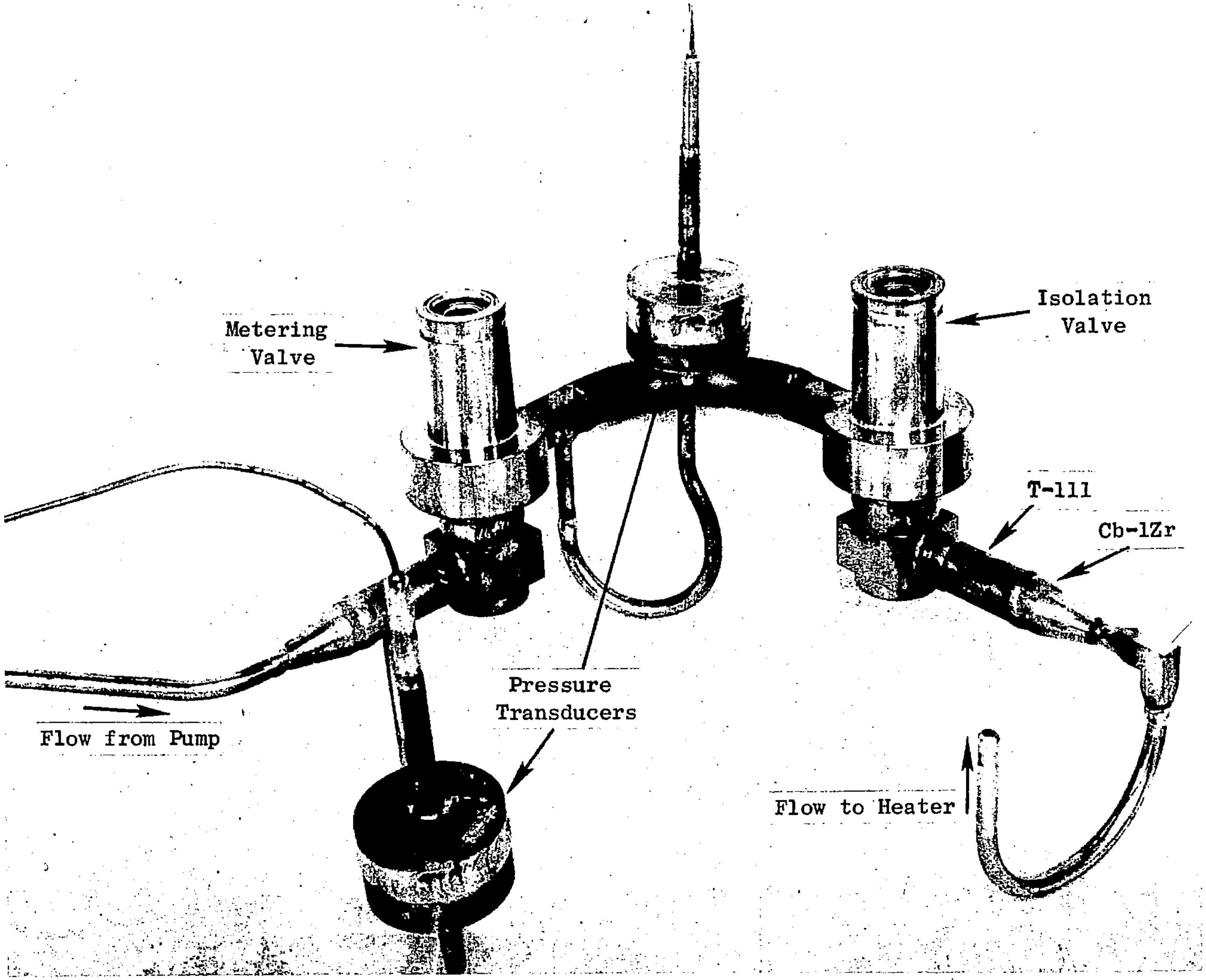


Figure 21. Valve and Pressure Transducer Subassembly - High Temperature Alkali Metal Valve Loop. (C68022314)

TABLE V

CHEMICAL ANALYSIS OF T-111 SHEET SPECIMEN FOLLOWING QUALIFICATION OF GENERAL ELECTRIC BREW FURNACE MODEL 92Y [a]

Element	Concentration, ppm[b]	
	Before Anneal	Wrapped Specimen[c] After Anneal
O	65	57, 65
N	5	11, 13
H	1	1, 1
C	10	17, 21

(a) One hour at 2400°F heat treatment at maximum pressure of 3.4 x 10^{-5} torr.

(b) Analytical Methods: O, N, and H - Vacuum Fusion
C - Combustion Conductometric

(c) 0.040-inch-thick specimen was wrapped with layer of 0.002-inch-thick Cb-1Zr foil during heat treatment.

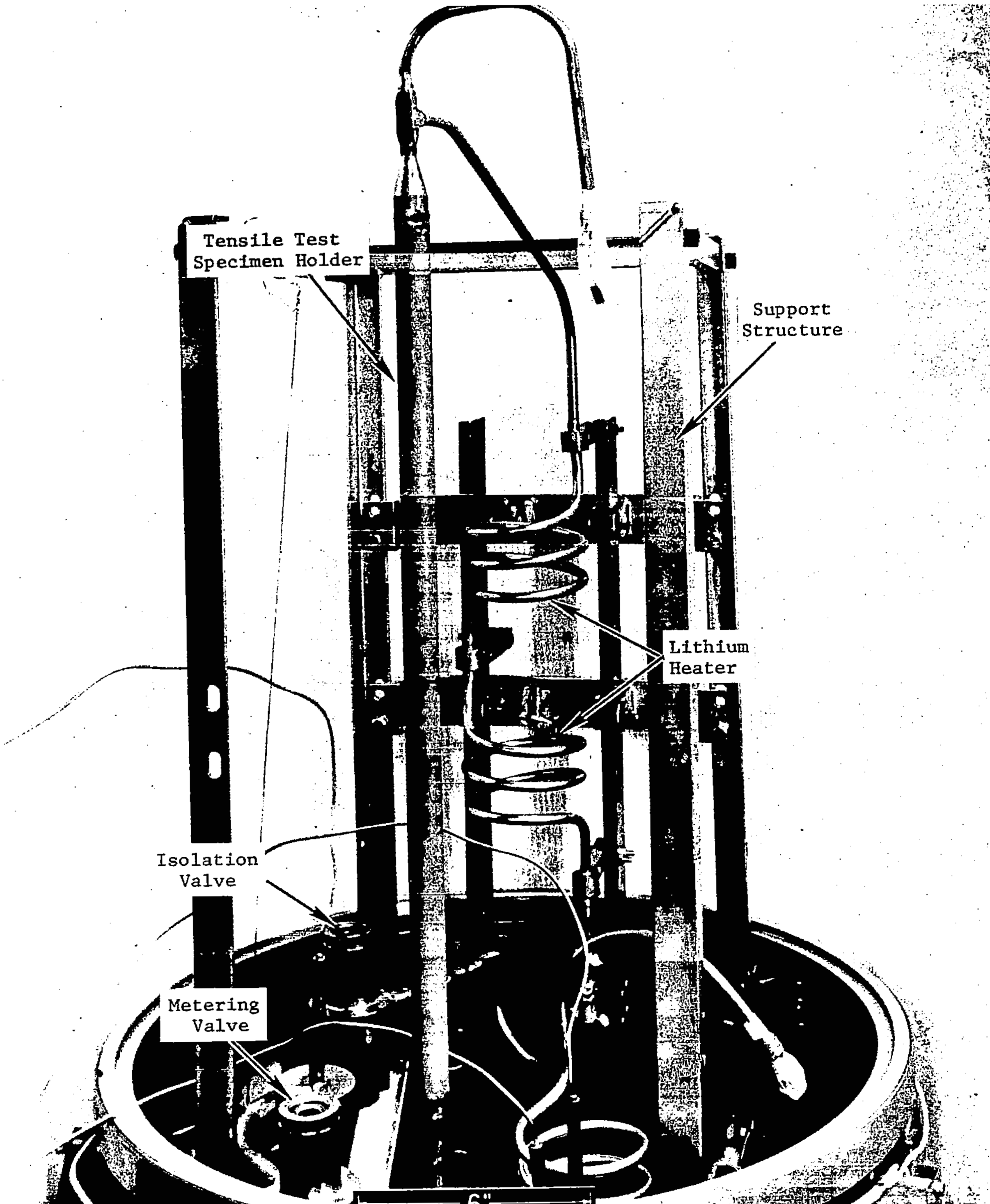


Figure 22. High Temperature Alkali Metal Valve Test Loop in the 24 Inch Diameter Vacuum Chamber Spool Section Prior to Final Welding. (C68031434)

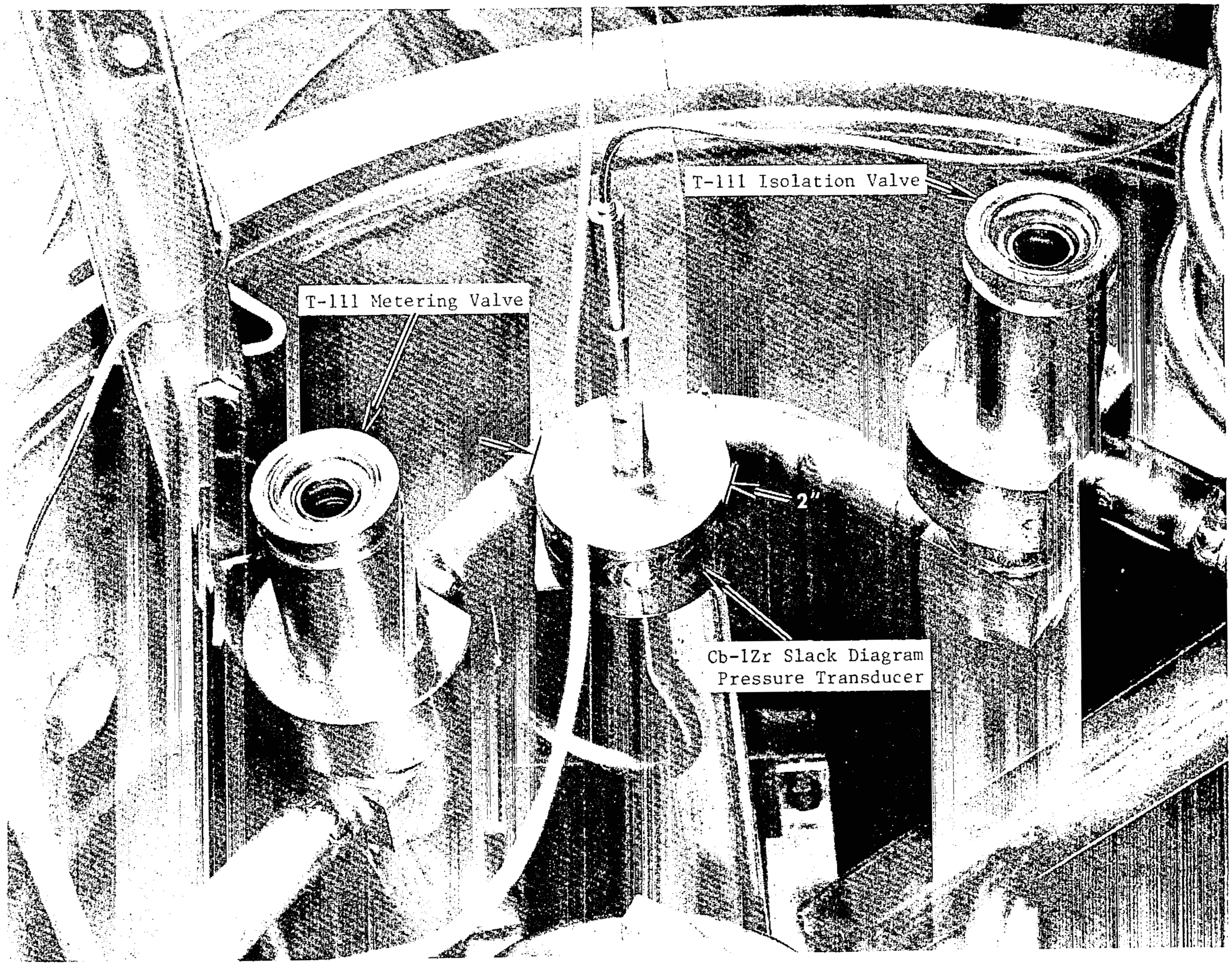


Figure 23. High Temperature Alkali Metal Valve Subassembly. (C68031433)

structure as shown in Figure 24 with temporary supports added to hold the EM pump duct for the final welding of the loop in the weld chamber.

The surge tank, a slack diaphragm pressure transducer, the EM pump inlet line and the exit of the tensile test section were first joined together at the common tee connection with four welds. The final assembly of all the loop components was made in the 8-foot diameter extension to the weld chamber with the loop held in the support structure as shown in Figure 24. Three welds were necessary to complete the assembly. The seven welds were then annealed at 2200°F for one hour using tungsten wire radiant heaters positioned over the weld areas. The pressure in the weld chamber during the annealing runs never exceeded 1×10^{-5} torr.

The loop was then transferred from the weld chamber to the vacuum chamber spool section and attached to the support structure. The stainless steel tubes for the gas pressurization, alkali metal fill and drain lines were then welded to the loop. The vacuum chamber closures of these lines and the slack diaphragm pressure transducers were made to their respective vacuum feedthrough in the spool piece. The welding of the stainless steel insulation can of the EM pump duct assembly to the vacuum chamber spool piece completed the assembly of the loop. The entire assembly was helium leak checked and no leaks were observed. The loop was then covered with a polyethylene bag for transfer to the test operation area for insulation and instrumentation.

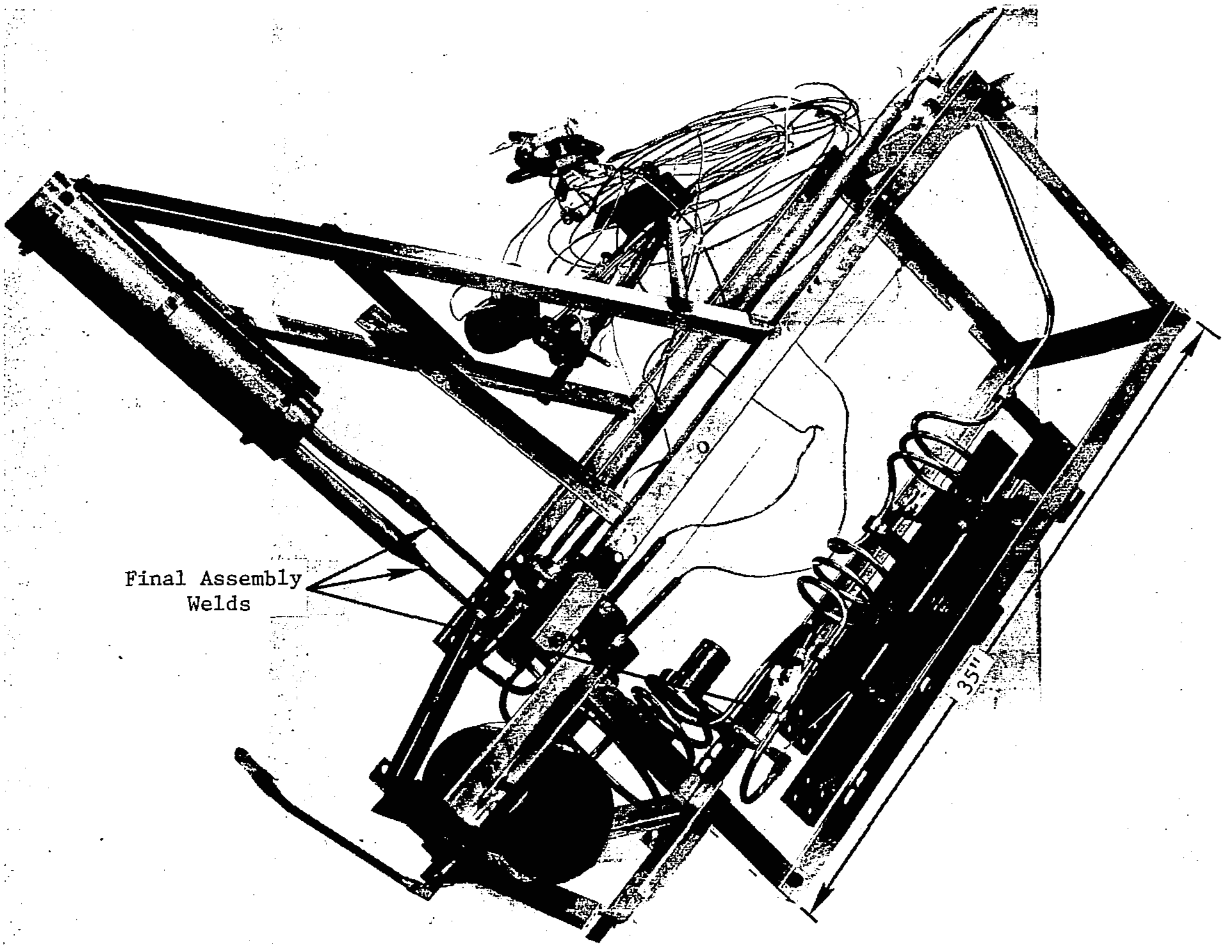


Figure 24. High Temperature Alkali Metal Valve Test Loop in Support Structure Used for Final Assembly in the 8 Foot Diameter Extension to the Weld Chamber. (C68031814)

V. INSTRUMENTATION AND INSULATION

The High-Temperature Alklai Metal Valve Test Loop was transferred from the fabrication area to a portable air shelter in the test area for the installation of components, attachment of loop thermocouples, and the application of Cb-1Zr foil thermal insulation. The reinforced polyethylene 15-foot diameter air shelter is shown in Figure 25. A constant supply of filtered air is forced into the shelter by a blower which maintains an internal pressure of 0.2 psig to support the entire weight of the polyethylene hemisphere. The walls of the air shelter are sealed to the concrete floor by sand bags resting on a reinforced apron to resist the upward force of the internal air pressure. A semirigid, steel-framed air lock is attached to the main room to permit personnel movement into and out of the air shelter without releasing the supporting air pressure. The interlock area is also used as a change room by the technicians entering the air shelter. All personnel engaged in instrumenting the loop were required to wear lint-free, white shoe covers, coveralls, and gloves. All loop components were vacuum cleaned, then washed with ethyl alcohol, and vacuum cleaned again to remove any dust or foreign material that could have accumulated during fabrication or final shipment of the loop to the test area.

Temporary supports used in the fabrication and transfer of the loop to the test area were removed and permanent supports and brackets were installed. The loop is primarily supported by the lithium heater electrodes which are bolted to 2-inch wide by 0.25-inch thick OFHC copper bus bars which are fastened to the support structure. The bus bars are electrically insulated from the support structure by alumina (99.7% Al_2O_3) insulators. Additional fixed supports were used for the slack diaphragm pressure transducers, the permanent magnet flowmeter, and the surge tank.

The location and designation of the 29 thermocouples which were installed on the loop are shown in Figure 26. A typical thermocouple circuit, as shown in Figure 26, originates at the hot junction of the thermocouple; the leads are routed along the support structure to a

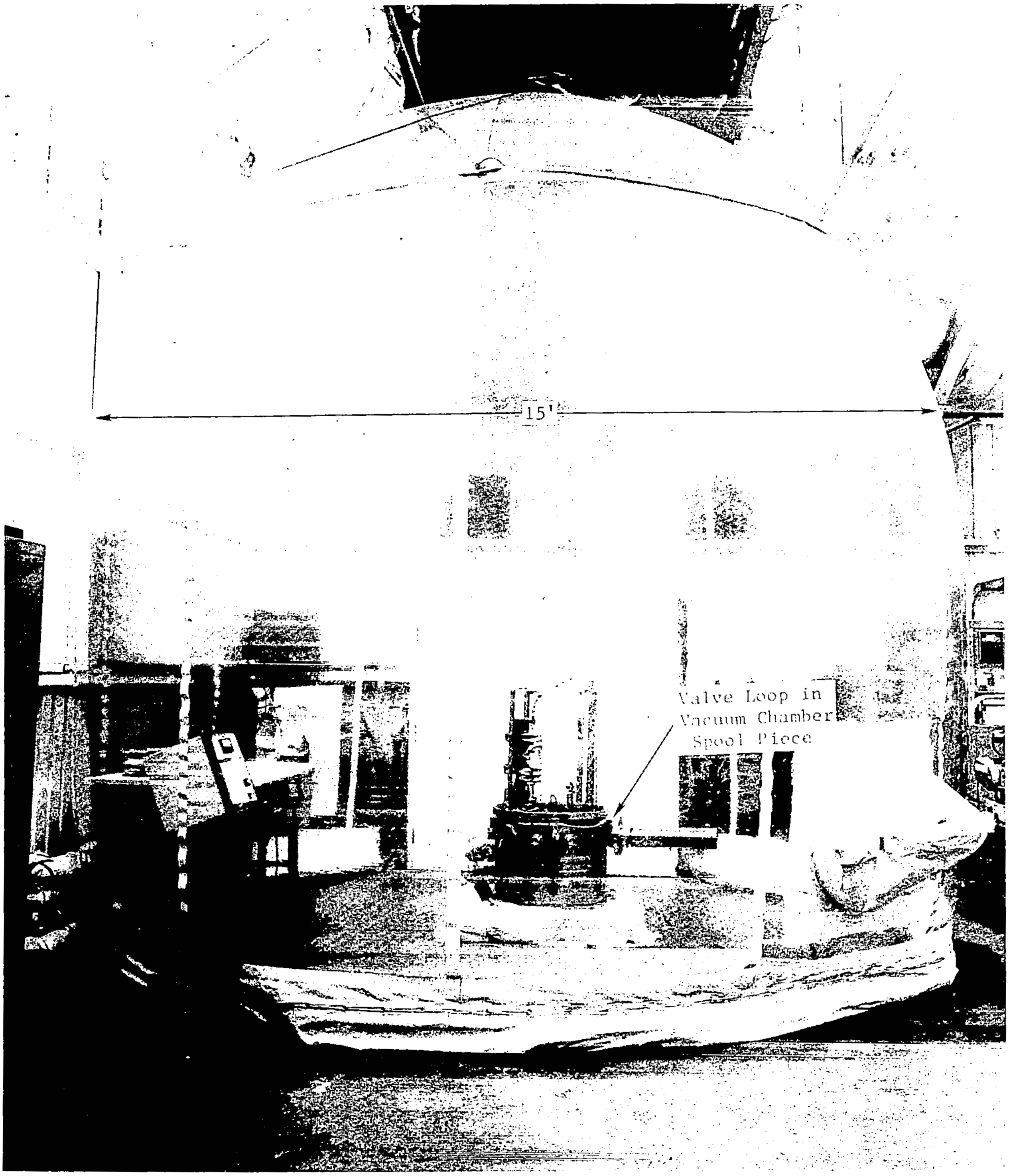


Figure 25. High Temperature Alkali Metal Valve Test Loop During Instrumentation and Insulation in the Air Shelter. (C68052227)

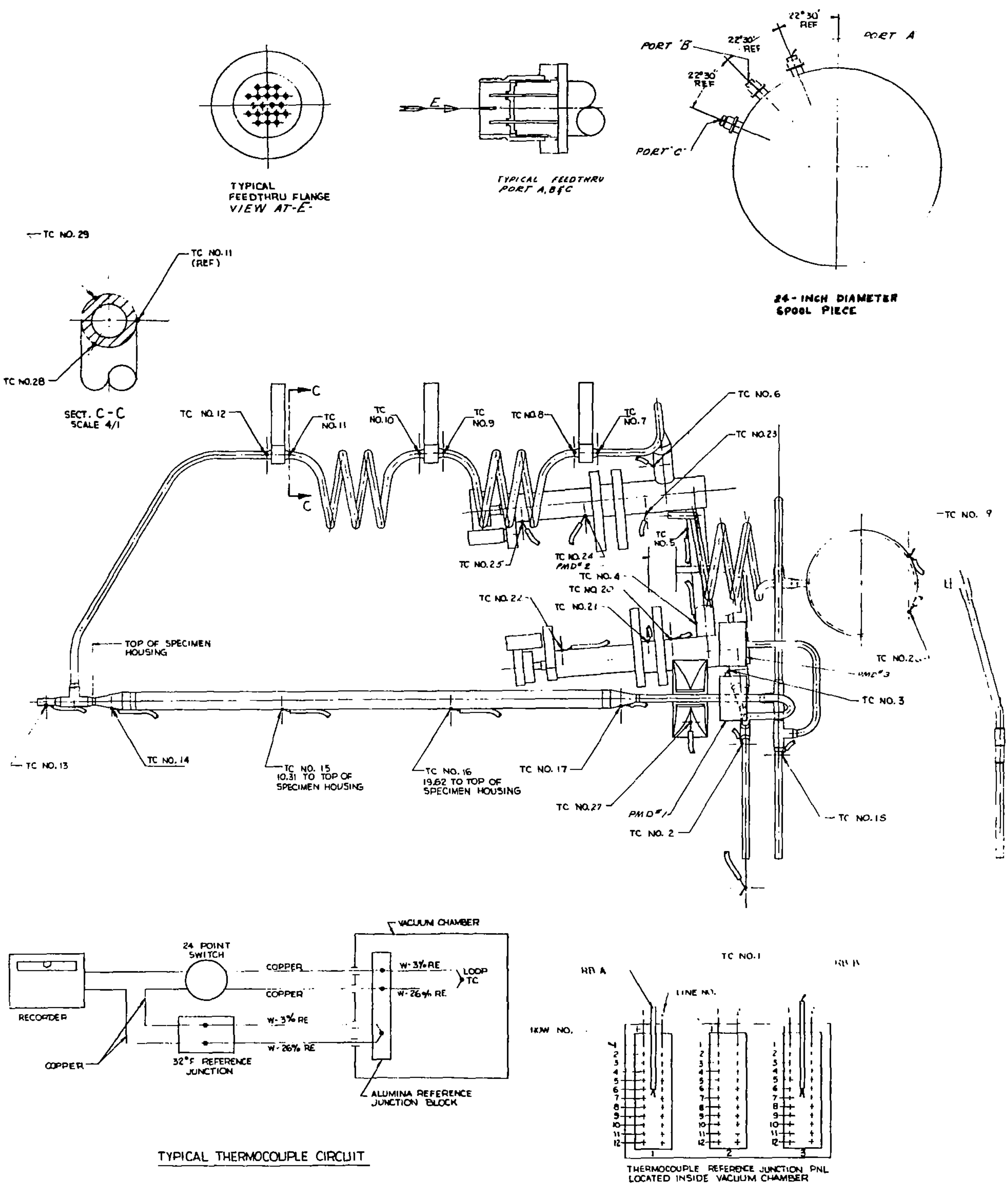


Figure 26. Instrumentation for the High Temperature Alkali Metal Valve Test Loop.

reference junction block, shown in Figure 27, attached to the inside wall of the spool section. The thermocouple reference junction block consists of an alumina (99.7 Al_2O_3) terminal strip mounted on a copper block and mechanically fastened to the walls of the chamber. The entire assembly is shielded from the loop to minimize temperature gradients in the junction block as well as maintain a lower absolute temperature approaching that of the water-cooled vacuum tank wall. At the reference junction block, a transition from the thermocouple wire to copper wire is made, and the copper wires are routed through the thermocouple vacuum feedthroughs, shown in Figure 28, to a 24-point recording potentiometer. The temperature of the thermocouple reference junction block was measured by W-3Re/W-25Re thermocouples which were electrically connected to the recording circuit as shown in Figure 26 to automatically compensate for variations in the reference block temperature during operation.

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 0.002 inch thick x 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 0.009 to 0.012 inch. The insulation was attached to the tube by spot welding the foil to the tube and to itself as succeeding layers were applied as shown for the lithium heater in Figure 29. A minimum number of spot welds were used to minimize conduction heat losses through the foil. A molybdenum electrode was substituted for the copper spot welder electrode to avoid contamination of the foil surfaces with copper and an argon cover gas was used to protect all welded areas from oxidation.

The bodies of the high temperature alkali metal valves were insulated with 4-inch wide strips of dimpled Cb-1Zr foil as shown in Figures 30 and 31. Eight layers of foil were form fitted to the contour of the body leaving the valve bonnet and gear drive exposed to allow radiation cooling during test operation.

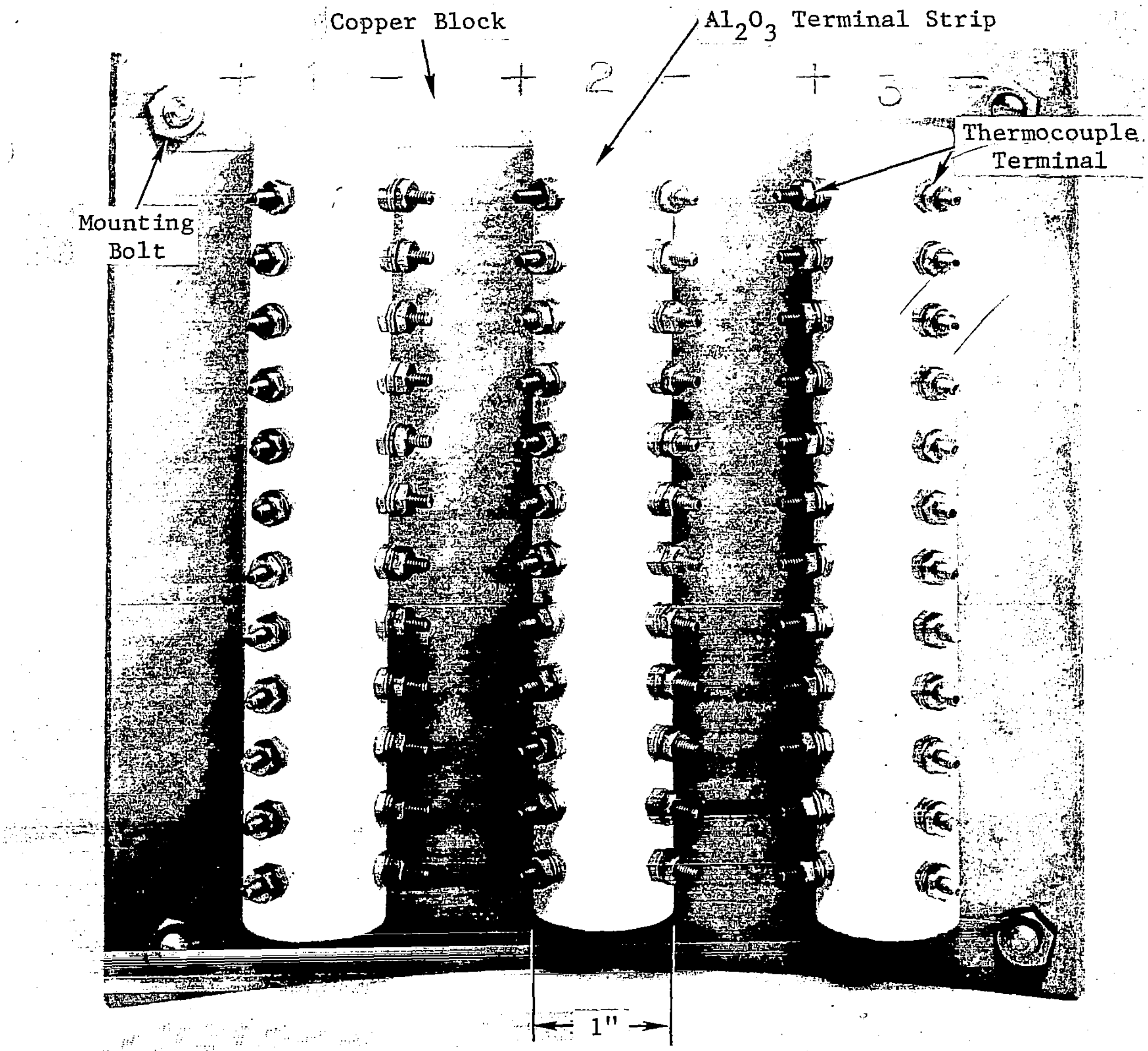


Figure 27. Thermocouple Reference Junction Block for the High Temperature Alkali Metal Valve Test Loop. (C68031435)

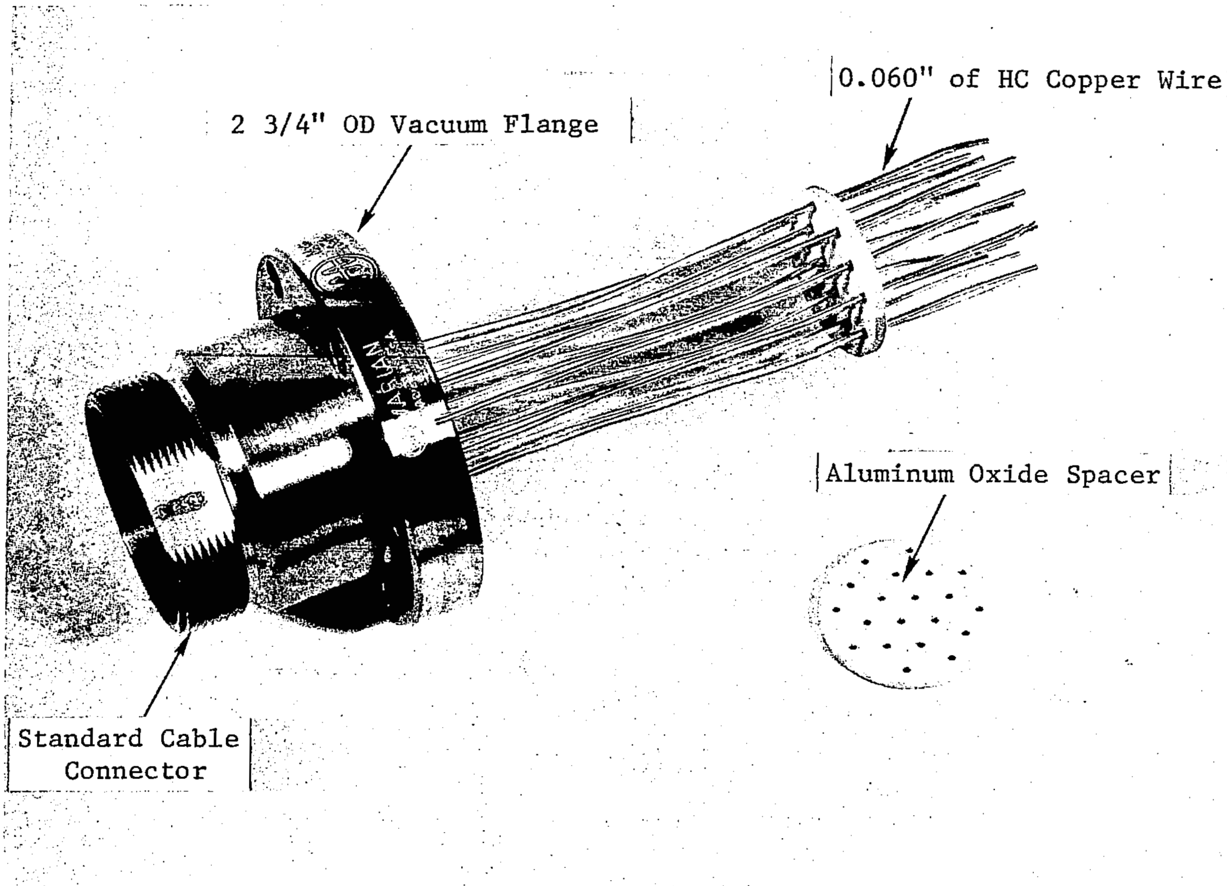


Figure 28. Twenty Pin (Copper Thermocouple Lead Vacuum Feedthrough) Varian Model 954-5013. (C67122141)

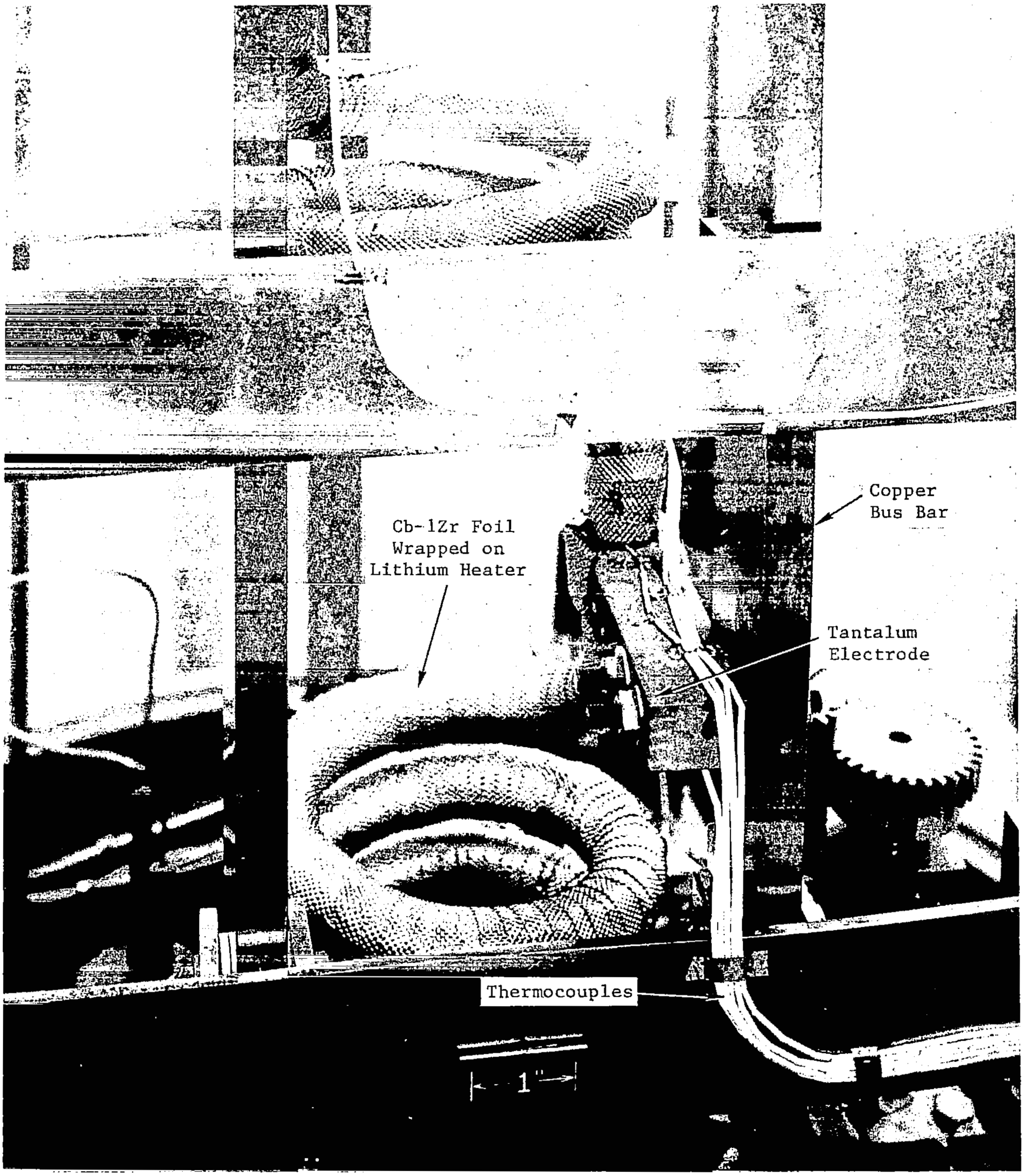


Figure 29. Lithium Heater - High Temperature Alkali Metal Valve Test Loop Insulated with Wraps of Dimpled Cb-1Zr Foil. (C68052223)

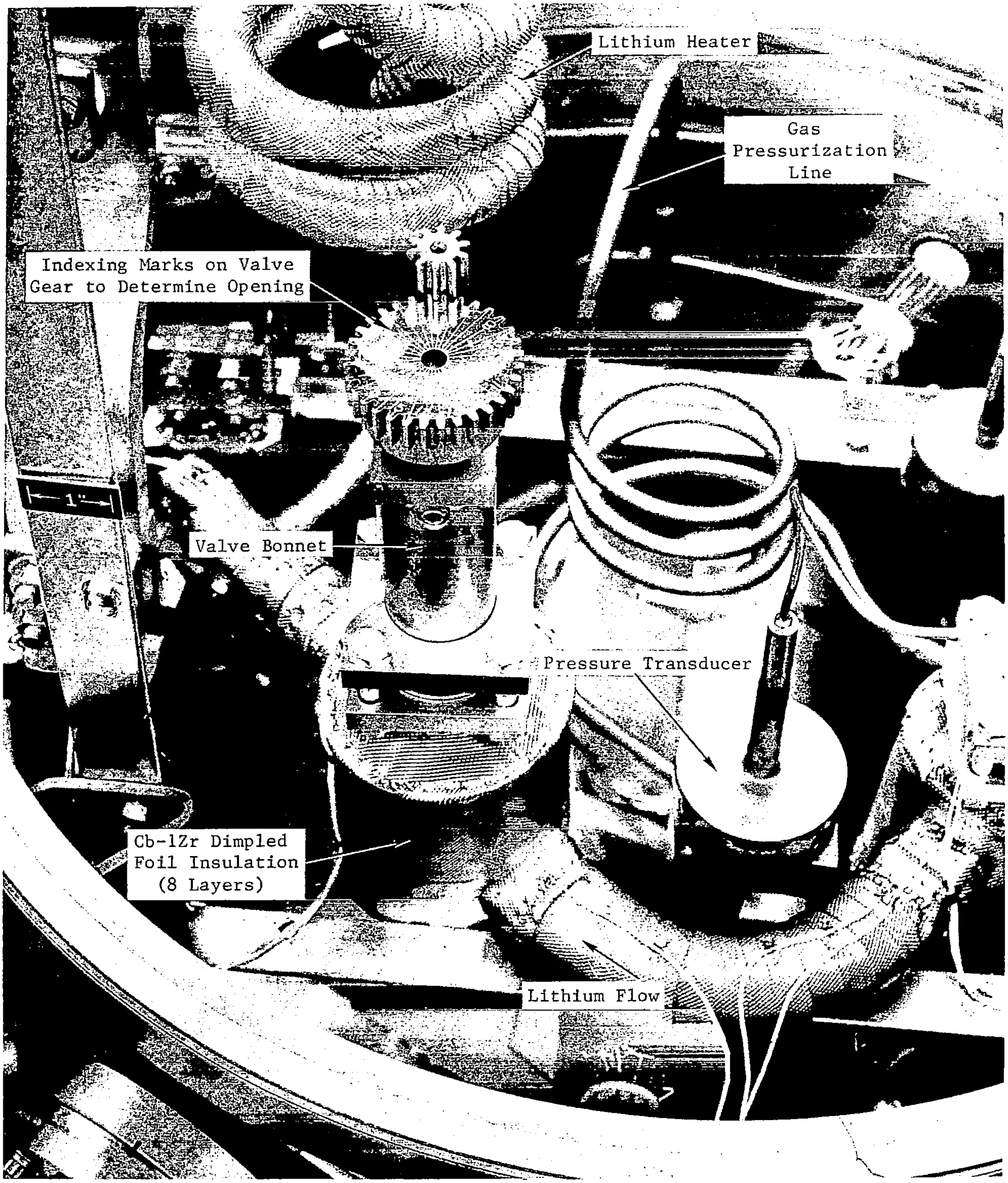


Figure 30. High Temperature Alkali Metal Valve Test Loop Following Instrumentation and Insulation. (C68052221)

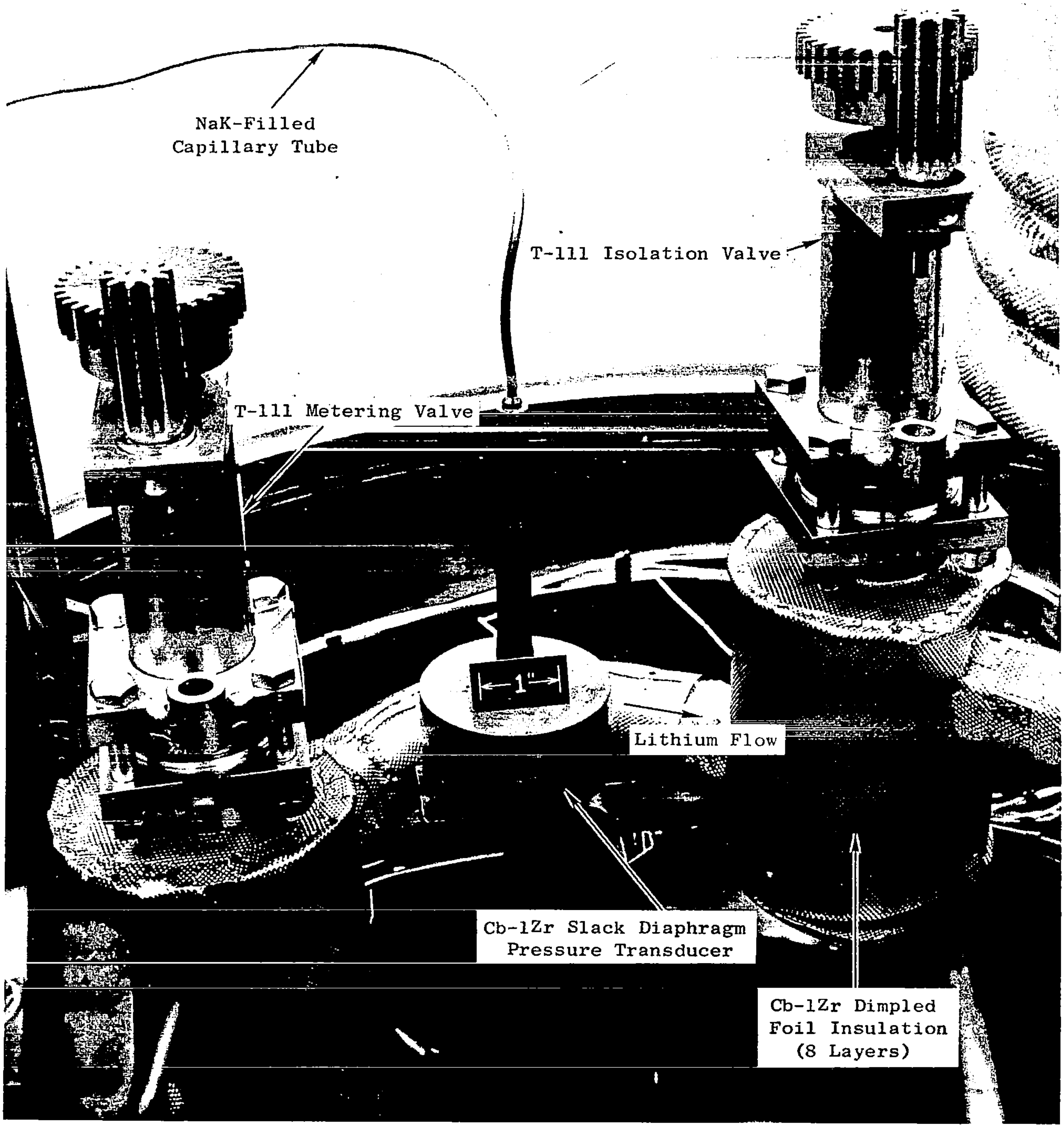


Figure 31. Alkali Metal Valves - High Temperature Alkali Metal Valve Test Loop. (C68052224)

The loop is shown in Figure 32 prior to moving the assembly to the test facility shown in Figure 33. The lower portion of the loop can be seen in Figure 34.

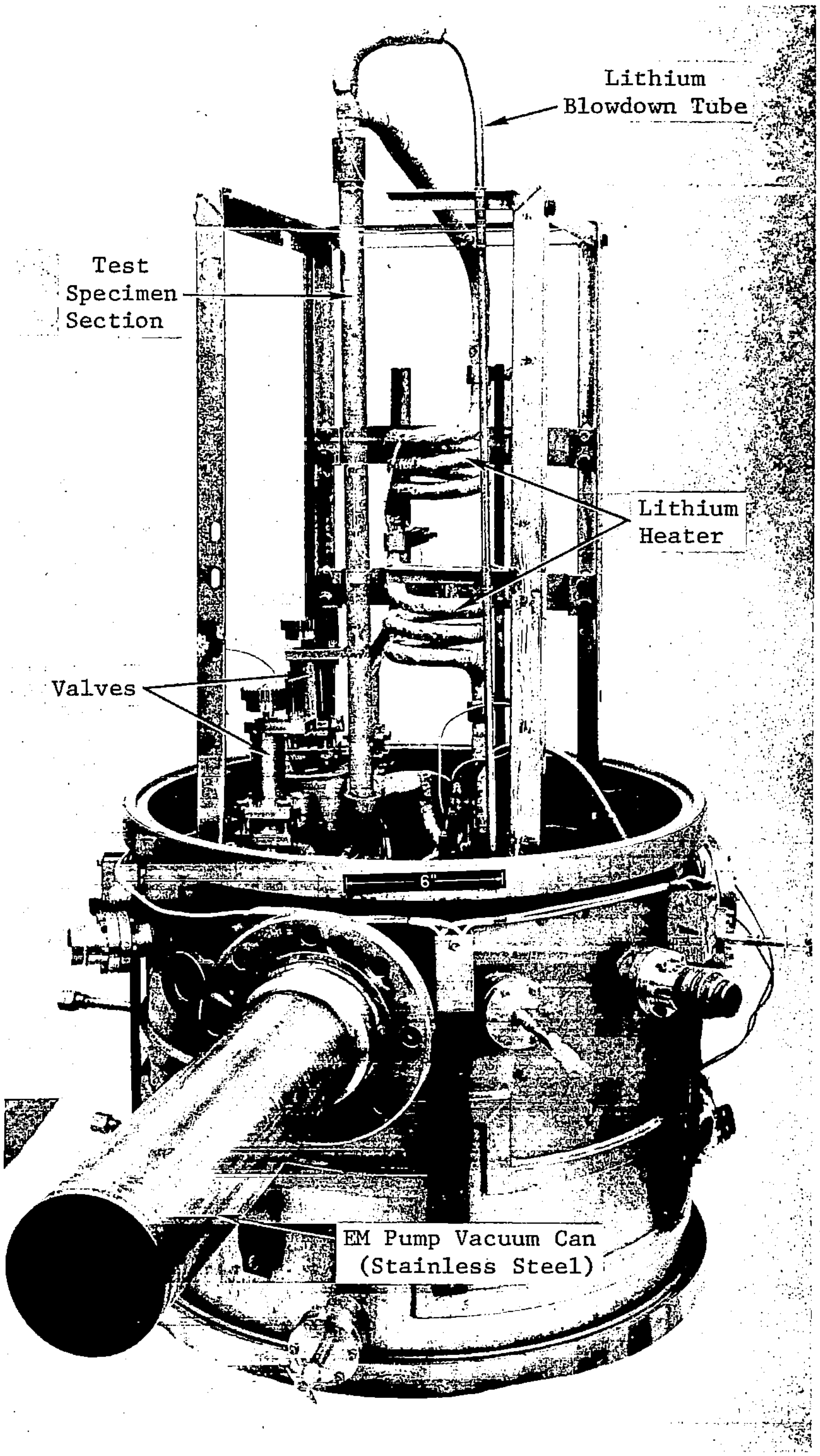


Figure 32. High Temperature Alkali Metal Valve Test Loop Following Instrumentation and Insulation. (C68052225)

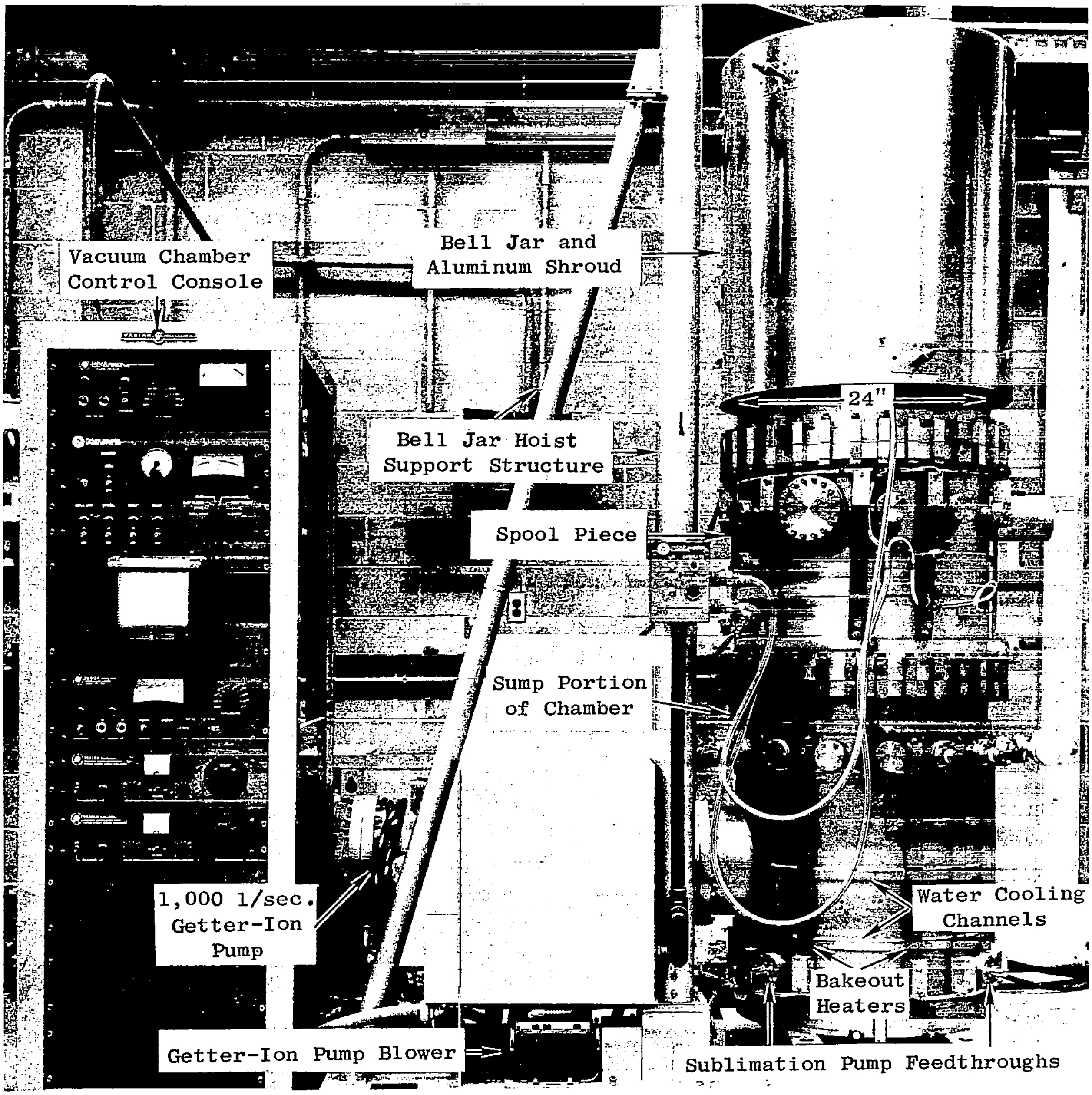


Figure 33. The 24-Inch Diameter by 90-Inch High Getter-Ion Pumped Vacuum Chamber and Associated Equipment Used for Testing the High Temperature Alkali Metal Valve Test Loop. (C64011410)

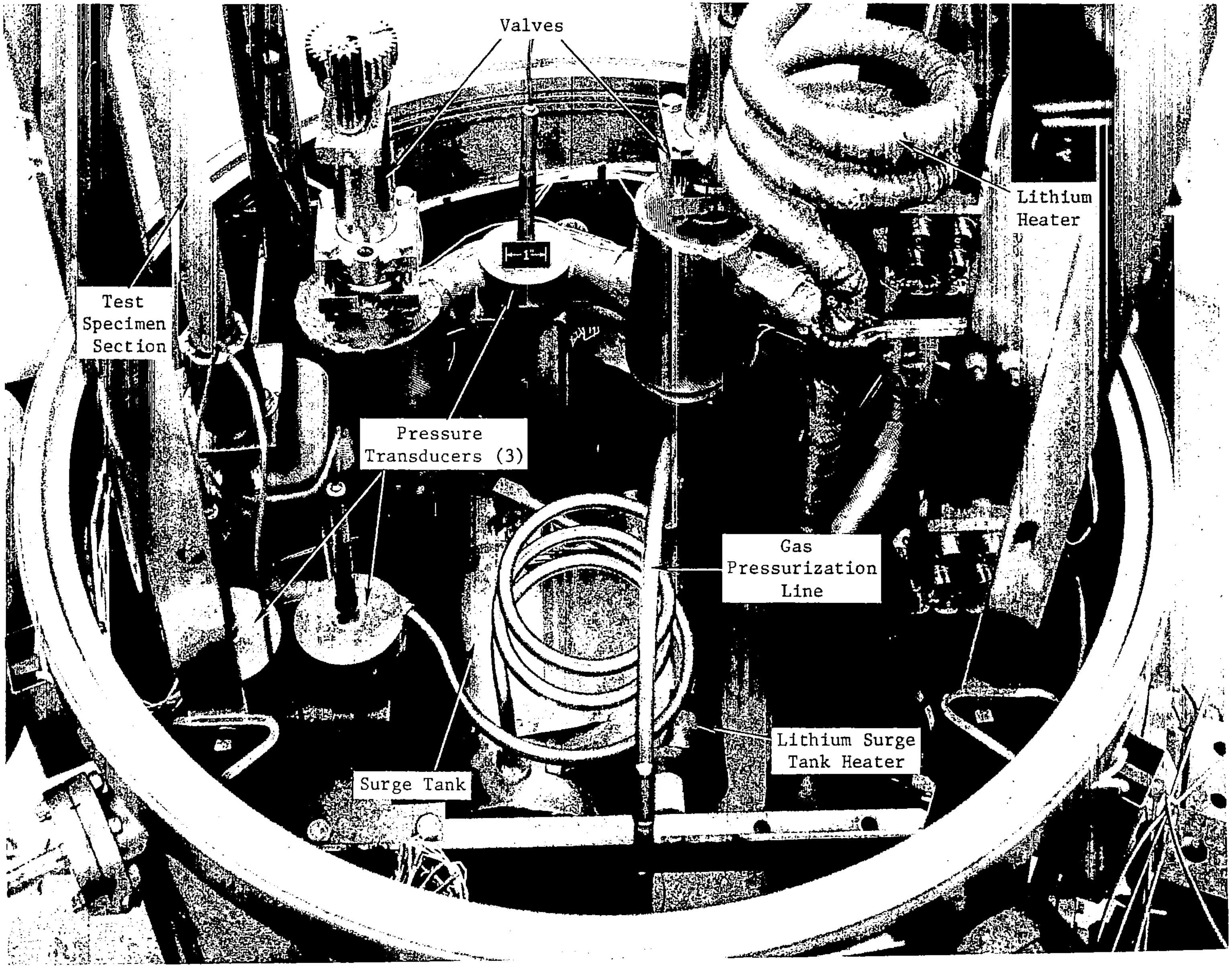


Figure 34. Lower Portion of the High Temperature Alkali Metal Valve Test Loop. (C68052226)

VI. VALVE TEST OPERATION

A. Installation of the Loop in the Vacuum Chamber

Following insulation and instrumentation of the loop mounted in the spool section, the unit was transferred from the air shelter to the sump section of the vacuum chamber and bolted in place. The final connections joining the loop proper to the auxiliary test equipment and the control console were then made and these are listed below:

1. The loop pressurization line was connected to an argon gas supply. The external argon gas supply system was of all welded stainless steel construction with bellows-sealed valves and a metal diaphragm pressure regulator.[(a)] The argon system was used to pressurize the lithium and thereby, prevent boiling in the heater coils during loop operation. The entire system was helium leak checked after assembly and all components were baked out at 300°F under vacuum.

   The argon gas supply used for pressurization of the loop analyzed[(b)] as follows:

Ar	- 99.997%	CO	- 1 ppm
O_2	- 5 ppm	CO_2	- 1 ppm
N_2	- 5 ppm	H_2	- 0.25 ppm
CH_4	- 0.2 ppm	H_2O	- 4.5 ppm

2. The EM pump stator was slipped over the pump duct insulation can and bolted to a fixed flange on the vacuum chamber.

3. The valve actuators were assembled and attached to the metering and isolation valves as shown in Figure 35. The systems were operated in air for two complete cycles to check out the entire actuation system including the cable and magnetic rotary vacuum feedthrough.

(a) Thermco Company, LaPorte, Indiana, 0-400 psig.

(b) Vendor Analysis - Matheson Company, Inc.

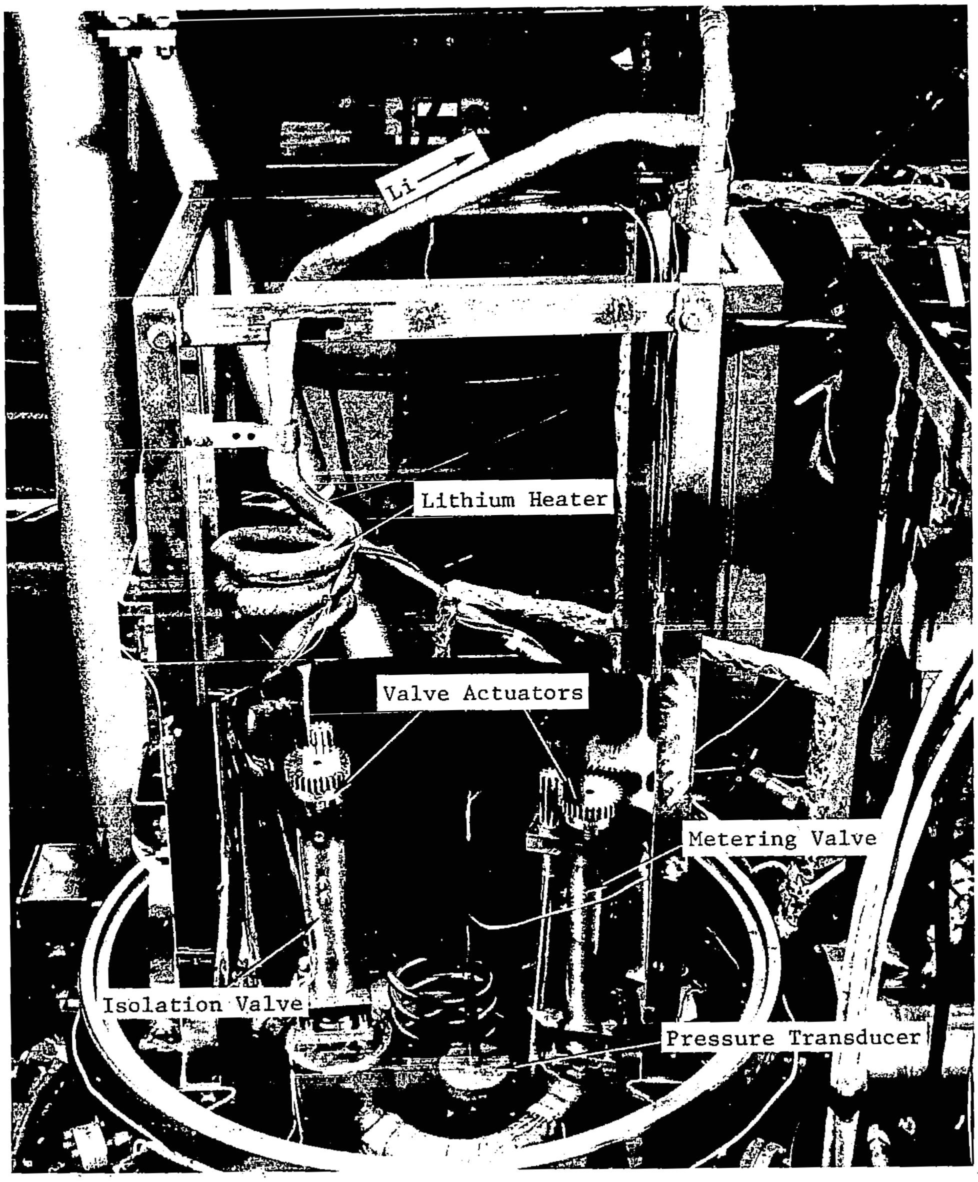


Figure 35. High Temperature Alkali Metal Metering and Isolation Valves Final Assembly into the Loop. (P68-10-6B)

4. The thermocouple leads were connected to the reference junction and resistance, and continuity checks were repeated on all thermocouples from the loop to the recorder. The flowmeter and the pressure transducers connections to the recording potentiometers were completed at this time.

B. Pumpdown and Bakeout of the Vacuum Chamber

The bell jar was bolted to the spool flange and the vacuum chamber was evacuated with the turbomolecular pump overnight before the 1000 liter per second getter-ion pump was turned on. When the chamber pressure was reduced below 1×10^{-6} torr the bakeout heaters were turned on. The steady state temperatures of the various loop and vacuum components varied from a minimum of 250°F to 525°F with all bakeout heaters on. During this period the loop itself was being evacuated through the getter-ion pumped vacuum system on the lithium filling apparatus.

Although the loop system proper was helium leak checked on numerous occasions during fabrication and following final assembly, an additional scheduled leak check was performed in the test chamber which assured that there were no leaks in the loop. This final leak test of the loop was performed by scanning for helium with the partial pressure analyzer while pressurizing the inside of the loop with helium. The loop was found to be leak tight. This result and the indicated leak tightness of the test chamber itself confirmed that outgassing was the principal source of the chamber pressure and the bakeout was continued.

C. Filling the Loop with Lithium

The purity of the distilled lithium immediately after distillation was excellent; however, because of prior indications of particulate matter in distilled lithium,(3) high purity argon was admitted to the receiver through the dip leg in order to agitate the lithium. This technique would produce a sample containing particulate matter if any was present. The sample was taken immediately after agitation. The analysis of these samples are shown in Table VI. Although slight increases

(3) Advanced Refractory Alloy Corrosion Loop Program, Quarterly Progress Report No. 12 for Period Ending April 15, 1968, NASA Contract NAS 3-6474, NASA-CR-72452.

TABLE VI

CHEMICAL ANALYSIS OF LITHIUM FOR THE HIGH-TEMPERATURE ALKALI METAL VALVE LOOP

Element	Concentration, ppm in Lithium			
	From the Still Receiver		From the Transfer System	From the Loop[a]
	As-Distilled	Agitated[b]		
O	9, 10		30, 72	13
N	8, 9	17, 20	5, 7	25, 33
C	30, 55	60	68	47
Ag	< 5	< 5	< 5	< 5
Al	< 5	25	5	5
B	< 10	< 50	< 50	< 50
Ba	< 10			
Be	< 5	< 5	< 5	< 5
Ca	< 5	25	5	5
Cb	< 25	< 25	< 25	< 25
Co	< 5	< 5	< 5	< 5
Cr	< 5	< 5	< 5	< 5
Cu	25-50	5-25	25	< 5
Fe	< 5	5	5	5
Mg	< 5	25	< 5	5
Mn	< 5	25	< 5	< 5
Mo	< 5	< 5	< 5	< 5
Na	< 50	< 50	< 75	< 50
Ni	< 5	< 5	< 5	< 5
Pb	< 75	< 25	< 50	< 50
Si	< 1	50	5	5
Sn	< 5	< 25	< 25	< 25
Sr	< 5			
Ti	< 5	< 5	< 25	< 25
V	< 50	< 25	< 25	< 25
Zr	< 25	< 25	< 25	< 25

(a) Taken from loop surge tank after circulating for 1 hour at 500°F.

(b) Lithium in still receiver agitated with argon gas before sampling.

in concentration of some impurities were noted in the sample (No. 1986) taken after agitating the lithium, these increases were not considered large enough to preclude the lithium from use in the loop.

The lithium transfer system was attached to the still receiver and the loop fill valve as shown in Figure 36. The transfer system contains a 7-micron sintered, Type 316, stainless steel filter, and a filter made of double dutch twill tantalum cloth in the transfer line between the 7-micron filter and the sampler. The tantalum screen filter is shown prior to assembly in Figure 37. The combination of these filters was employed to prevent particulate matter from entering the loop during filling. The disposal tank between the tantalum filter and charge valve permitted flushing and draining of the lithium transfer system before filling the loop. The transfer system and the loop were outgassed at 400-700°F with a getter-ion pump until the pressure rise rate was less than one micron liter per minute.

The following operations were then conducted to fill and prepare the loop for operation (with Reference to Figure 36):

1. The transfer system including the charge pot was filled with lithium (3500 cc capacity) between the receiver (valve L) and the loop fill valve (valve KK).

2. The lithium in the transfer system was then dumped into the disposal tank.

3. The transfer system was refilled and sampled. Analysis of the sample shown in Table X indicated the lithium purity was excellent.

4. The loop surge tank was then filled with the charge pot inventory (3500 cc). The lithium level in the charge pot was monitored with the level probe until it indicated that no lithium remained in the charge pot.

5. After circulating the lithium in the loop for one hour at about 500°F, the lithium was returned to the surge tank and a sample taken. The surge tank was pressurized and lithium circulation re-established in the loop. The loop temperature

ITEM	IDENTIFICATION NO.	DESCRIPTION OR NAME	
1	(SEE NOTE 19)	ARGON BOTTLE (SEE NOTE 1)	
2		PRESSURE REGULATOR (SEE NOTE 2)	
3		PRESSURE GAUGE - 0-30 PSIA (SEE NOTE 3)	
4	(SEE NOTE 8)	NIPPLE 1" WITH CONFLAT FLANGES	
5	(SEE NOTE 4)	THERMOCOUPLE GAGE - VEECO	
6	SEE NOTE 5)	SORPTION PUMP CAT# 22HP112	
7	CAT# 1402 B	ROUGHING PUMP (SEE NOTE 6)	
8	SST VITON	VALVE - BELLOWS SEAL ¼ IPS (SEE NOTE 4)	
9	CAT# TY442	VALVE WITH ¼ SWAGELOK OUTLET (SEE NOTE 7)	
10	#HY473A MOD6	VALVE (SEE NOTE 7)	
12		THERMOWELL	
13		INSULATION - FIBERFRAX TYPE 45WF	
		½" THICK - FOR BACKING (SEE NOTE 8)	
14		HOT TRAP - 35 LB Li @ 1500°F	
15	(SEE NOTE 9)	HEATER - HEAVY-DUTY CAL - SP 1000W @ 110V	
16	(SEE NOTE 10)	FILTER, 5 MICRON SINTERED, 316 SST	
17		STILL POT 304 SST	
18		CONDENSER Cb-1Zr & 304 SST	
19		BI-METALLIC JOINT, Cb-1Zr to 304 SST	
20	224 SERIES 2" TRAP	CRYOSORB COLD TRAP WITH CONFLAT FLANGES	
21	CAT# TY445	VALVE (SEE NOTE 7)	
22		CHARGE POT 304 SST	
23	CAT# HY477A	VALVE (SEE NOTE 7)	
24	(SEE NOTE 3)	PRESSURE GAGE 0-200 PSIA	
25		DISPOSAL TANK	
26	(SEE NOTE 21)	MECHANICAL PUMP CAT# 6902E	
27		VACUUM TANK LOOP	
28	(SEE NOTE 4)	NUDE GAGE BGT-75	
29		DISTILLATE RECEIVER	
30		VACUUM HOSE 1½" OD	
31	(SEE NOTE 22)	TA SCREEN FILTER	
32	(SEE NOTE 18)	1B FILTER NUPRO 7 MICRON SINTERED	

ITEM	IDENTIFICATION NO.	DESCRIPTION OR NAME	
31		PIPE ½" SCH 10 304 SST	
32	CAT# 810-1-8W-316	MALE CONNECTOR ½ PIPE TO ½ TUBE (SEE NOTE 13)	
33		PIPE ½" SCH 10 304 SST	
34	CAT# 600-6-316	UNION ½ TYPE	
35	CAT# 54897-8	CONOSEAL UNION FITTING ½ OD TUBE	
		(SEE NOTE 13)	
36		TUBING ½" OD x .035 WALL 304 SST	
37	SERIES 205	VALVE - 2" HIGH VACUUM WITH CONFLAT	
		FLANGES (SEE NOTE 11)	
38	MODEL 951-5027	VALVE 1½" HIGH VACUUM WITH CONFLAT	
		FLANGES (SEE NOTE 17)	
39	MODEL RG-75K	ION GAGE - VEECO WITH CONFLAT FLANGES.	
		(SEE NOTES 4)	
40	(SEE NOTE 11)	BELLOWS 2" WITH CONFLAT FLANGES	
41	(SEE NOTE 11)	BELLOWS 1" WITH CONFLAT FLANGES	
42	(SEE NOTE 8 & 11)	NIPPLE 2", WITH 1-6 GE FLANGE	
43	(SEE NOTE 17)	FLANGE - ROTATABLE CONFLAT # 954-5078	
44	(SEE NOTE 11)	CROSS 1" " " #214092	
45	(SEE NOTE 11)	TEE 1" " " "	
46	MODEL 22TP250	TRIODE ION PUMP 100 L/SEC	
		GE FLANGES (SEE NOTE 3)	
47		ZIRCONIUM GETTER	
48		OPTICAL BAFFLE	
49		VALVE 2" HIGH VAC, WITH CONFLAT	
		& GE FLANGES (SEE NOTES 8 & 11)	
50	MODEL 951-5007	HIGH VACUUM VALVES (SEE NOTE 17)	
51			
52	MODEL 19-580	PRESSURE REGULATOR (SEE NOTE 13)	
53	9410536	SAMPLING SYSTEM	
54		TUBING ½ OD x .065 WALL - 304 SST	
55	(SEE NOTE 11)	TEE 2" WITH CONFLAT FLANGES	
56	(SEE NOTE 20)	BELLOWS 1½" ½ 18	
57		HANDLING SYSTEM TERMINATES AT THIS POINT	
58	CAT 22HB021	FORE LINE TRAP (SEE NOTE 5)	
59	SEE NOTE 7	VALVE, CAT # HY473	
60		LIQUID LEVEL INDICATOR WELL	
61		TITANIUM LINER	
62		LIQUID LEVEL INDICATOR I TUBES	

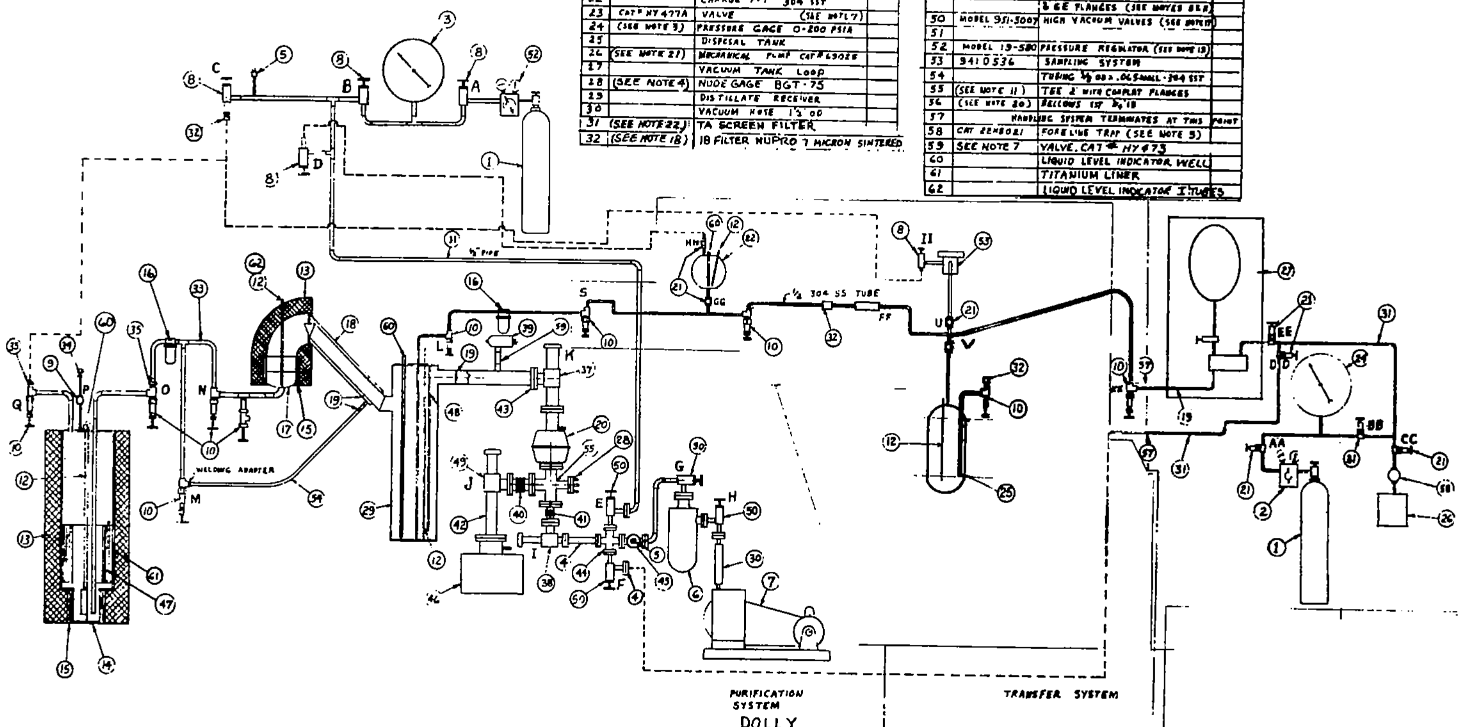


Figure 36. Lithium Purification and Transfer System Schematic Diagram.

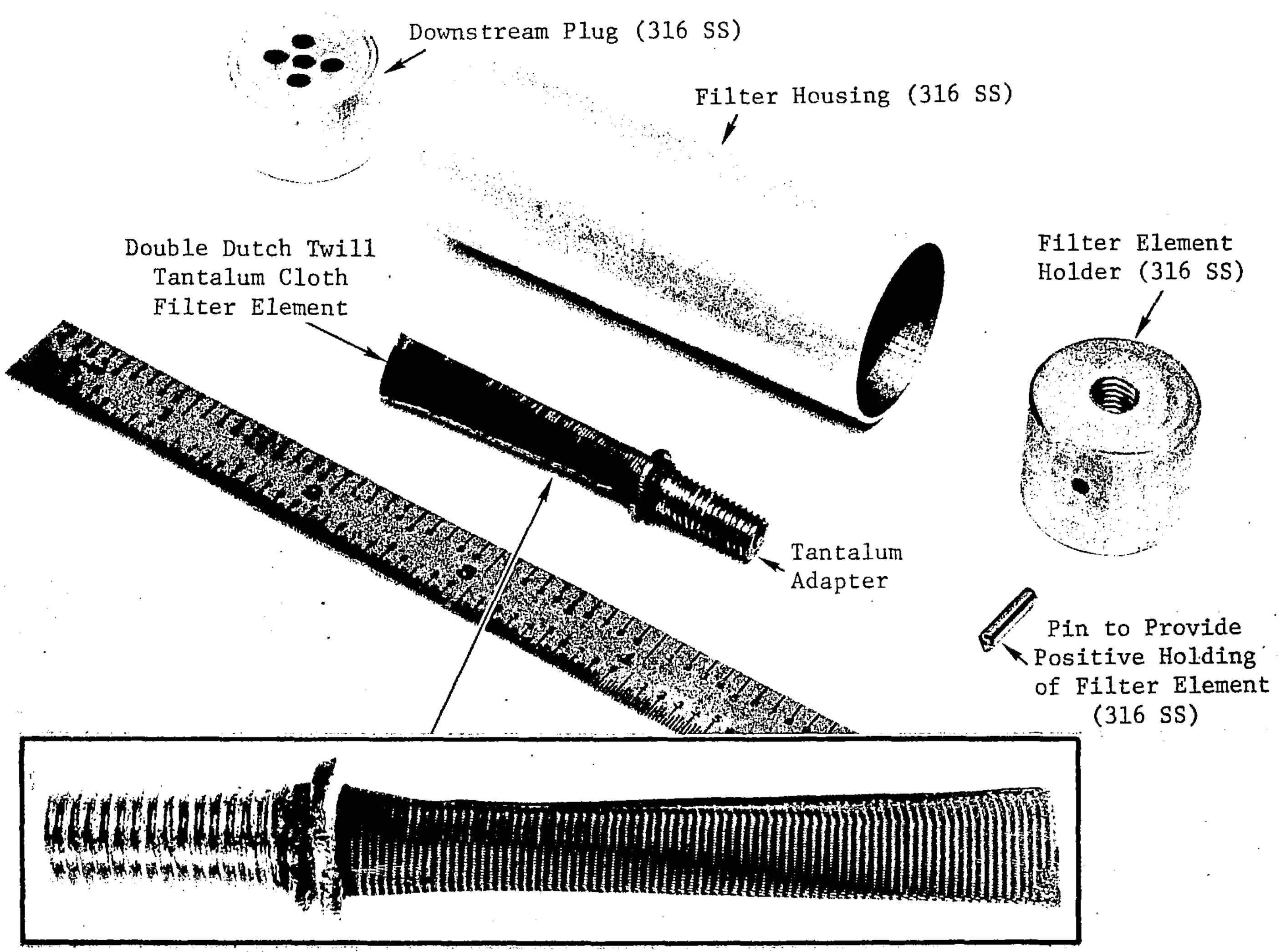


Figure 37. Tantalum Screen Filter Prior to Assemblv. (C68062804)

was maintained at 750°F until the results of the analysis were obtained. The analysis, shown in Table X, indicated excellent purity and high temperature loop operation could be initiated.

D. Loop Instrumentation Checkout

The checkout of loop instrumentation indicated all equipment and controls operated as required during the low temperature (< 1000°F) runs.

Calibration of the three slack diaphragm pressure gauges, which had been originally calibrated with inert gas before filling the loop with lithium, was repeated using a calibrated Wallace and Tiernan Model No. FA145 pressure gauge as the standard. All pressure gauges showed good linearity and excellent repeatability. The calibration results for the lithium pump outlet gauge are shown in Figure 38 and are typical of the results obtained for the other two pressure transducers.

The loop thermocouples were checked by operating the loop at near isothermal conditions and comparing the indicated temperature of each thermocouple with those adjacent to it. Although thermocouple calibration runs at temperatures in the range of the design operating conditions (1200°F to 2100°F) were desired, heat losses from the uninsulated test section prevented isothermal operation. A near isothermal condition in the loop was achieved by operating at a relatively high lithium flow rate (1.5 gpm) with the vacuum chamber on 500°F bakeout. The temperature distribution in the loop at the 630°F calibration run is shown in Figure 39.

After completion of the calibration tests, the loop safety circuits were tested and set according to the test plan. These included the following:

1. Loss of cooling water to the electric power feedthroughs.
2. Lithium heater overtemperature.
3. EM pump winding overtemperature.
4. Vacuum chamber leak.

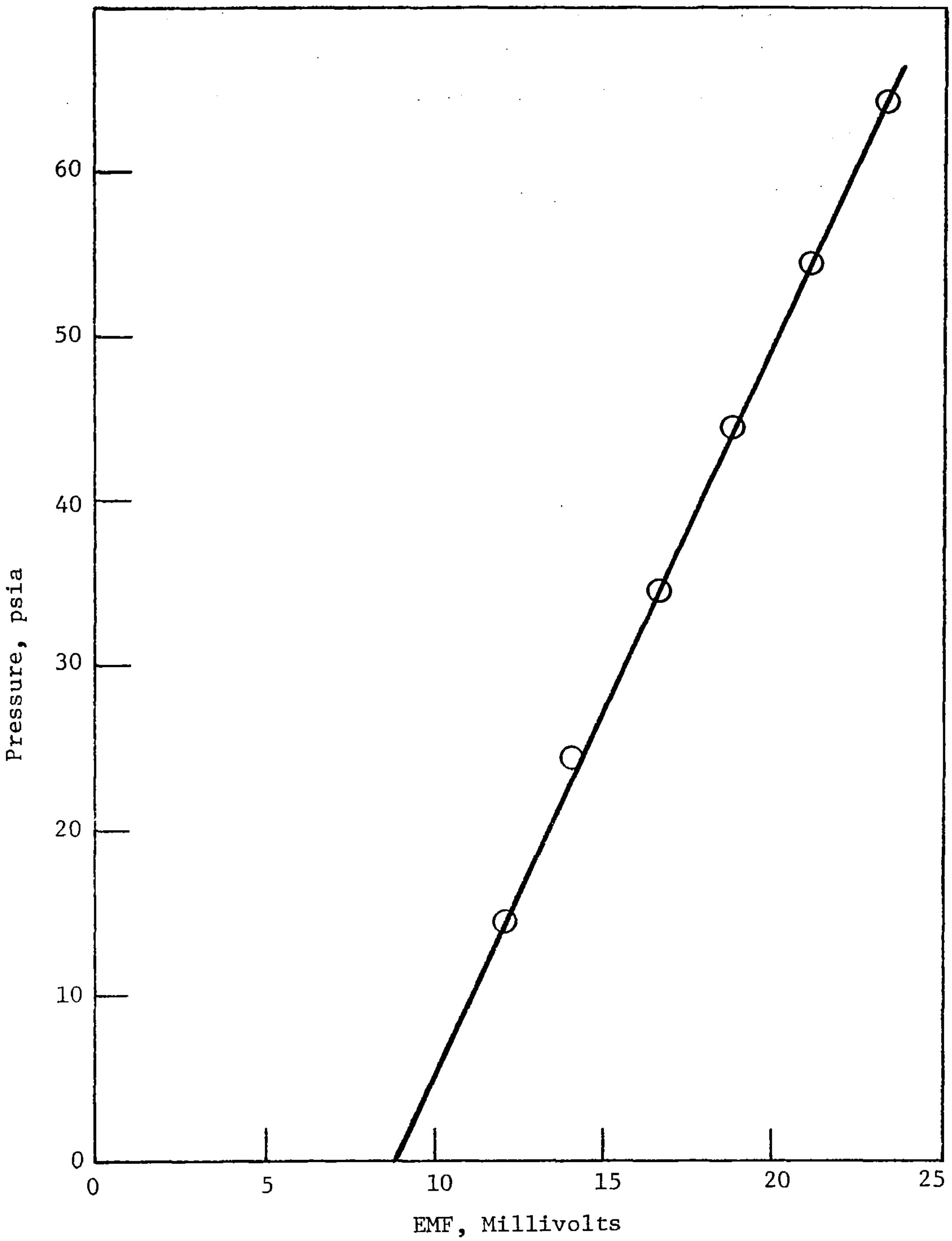


Figure 38. Calibration of Slack Diaphragm Pressure Transducer at EM Pump Outlet - High Temperature Alkali Metal Valve Test Loop.

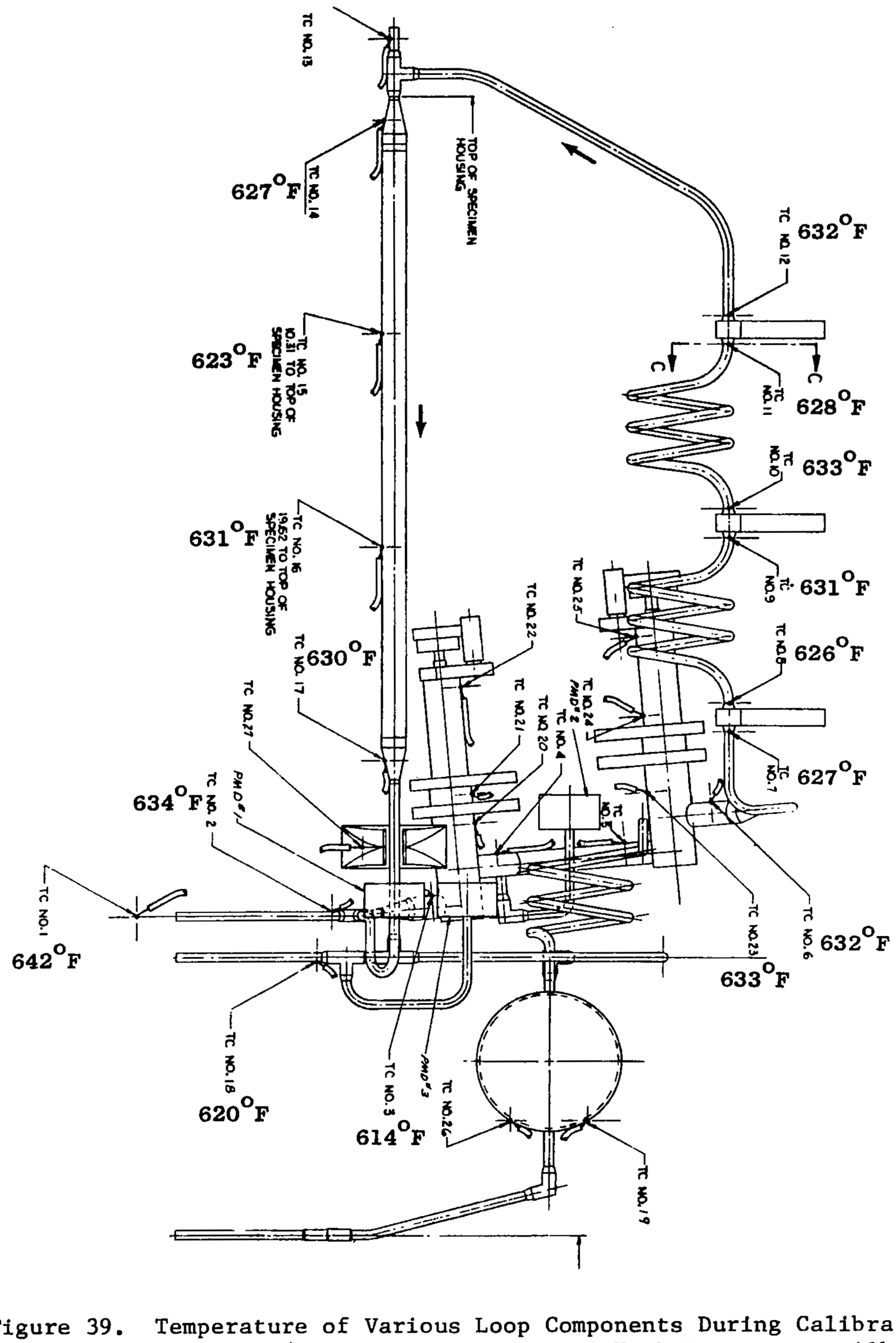


Figure 39. Temperature of Various Loop Components During Calibration of W-25Re/W-3Re Thermocouples on High Temperature Alkali Metal Valve Loop.

The electrical circuit for the vacuum chamber leak safety circuit was tested but not set. The safety circuit fires an explosive valve releasing high purity argon gas into the chamber if the vacuum chamber pressure increases above 3×10^{-6} torr. The explosive valve was armed after steady state conditions had been reached to reduce the probability of inadvertently flooding of the chamber due to an ion pump instability (argon gas release).

E. Valve Testing

The valves were actuated during the loop test under simulated operational conditions given in Table VII. The isolation valve was closed and maintained in the closed position using 12 in-lb of torque on the magnetic rotary feedthrough during the steady state operation. The torque was applied by suspending a measured weight from the magnetic rotary feedthrough actuator on the outside of the vacuum chamber. At the end of each steady state run the torque required to unseat the valve plug on the valve was measured by adding the necessary weight to turn the actuator.

The valve test was initiated when the lithium temperature at the valves was increased to 1200°F. During the first 100 hour period, the isolation valve was closed with an applied load of 12 in-lb of torque on the magnetic rotary drive feedthrough. After 100 hours, a break-away torque of 5.2 in-lb was required to open the isolation valve. The isolation valve was then operated for two cycles from fully open to fully closed. The metering valve was then operated for 10 cycles of 1/4 travel and 2 cycles of 3/4 travel. In each operation, the valve again opened more smoothly than in the closing operation.

The lithium temperature at the valves was increased to 1400°F and the loop operated an additional 200 hours. A break-away torque of 5.3 in-lb was required to open the isolation valve. The isolation valve was then operated for an additional 4 cycles for a total of 6 complete cycles. The metering valve was operated for 15 of the scheduled 20 cycles of 1/4 travel when the set screw in the mechanical connector attaching the flexible cable to the magnetic drive feedthrough became loose. The loop was shutdown and the set screw retorqued and tested under applied load. The flexible cable on the isolation valve was

TABLE VII

VALVE CYCLING SCHEDULE

Lithium Temperature At Valves °F	Test Period Hours	Total Test Hours	Cumulative Cycles On Isolation Valves Full Travel	Cumulative Cycles On Metering Valve 1/4 Travel	Cumulative Cycles On Metering Valve 3/4 Travel
1200	100	100	2	10	2
1400	200	300	6	30	6
1600	200	500	10	50	10
1700	500	1000	15	100	20
1800	500	1500	25	150	30
1900	1000	2500	45	250	50
1900	500	3000	55	300	60
1900	500	3500	65	350	70
1900	500	4000	75	400	80
1900	500	4500	85	450	90
1900	500	5000	95	500	100

also inspected at this time. Some separation of the woven cable wires was observed at the brazed joint of the cable connector, and appeared to be a result of insufficient braze penetration during manufacture. The cable was replaced with a new assembly which was baked out for 16 hours at 700°F before installation.

The chamber was closed and the loop returned to the 1400°F valve operating temperature. The metering valve was then operated to complete its scheduled cycling without any further difficulties. The lithium temperature at the valves was increased to 1600°F for a 200 hour continuous run before actuation of the valves would again be performed.

The test loop completed 500 hours of operation, the last 200 hours of operation at the 1600°F valve temperature and as planned, actuation of the valves was initiated. The measured break-away torque on the isolation valve was 11.7 in-lb as compared to an original break-away torque of 5.2 in-lb which was measured after 100 hours operation at 1200°F.* The isolation valve was then fully opened to start the cycling operation which requires the completion of 4 cycles from fully open to fully closed. During the initial closing of the valve, the over pressure safety circuit was actuated; the explosive valve fired and flooded the vacuum chamber with argon. Simultaneously, all electrical power to the lithium heater and EM pump was interrupted. The lithium in the loop was immediately dumped into the surge tank before it could solidify in the loop.

The explosive valve was immediately replaced but not armed and the vacuum chamber was evacuated. The vacuum chamber pressure was 1.5×10^{-8} torr after overnight pumping (14 hours) indicating that the firing of the over pressure safety circuit was not due to an air leak in the vacuum chamber. During the subsequent heating of the loop, the partial pressure gas analyzer was used to monitor the residual gas in the vacuum chamber to detect any change in the gas composition which would indicate the cause of the over pressure signal. Although the total pressure of the vacuum chamber increased during the bakeout period, no significant changes in the residual gas composition were observed. The loop was

*The increase in torque required to open the isolation valve was found to be a result of galling of the Mo-TZM pinion and spur gears and not as a result of bonding of the plug and seat.

then filled with lithium from the surge tank and low-temperature test operation was started.

It is believed that the over pressure safety circuit was actuated by a gas burst which may have come from the valve actuator system during its operation. The isolation valve actuation system was operated during the low-power run and the effects on the total pressure observed. Although a noticeable change in the total pressure was observed, (2×10^{-7} torr to 3×10^{-7} torr) the pressure level was an order of magnitude less than that required to fire the argon explosive valve circuit which was set at 3×10^{-6} torr.

The lithium temperature at the valves was brought back to 1600°F and the valves actuated as scheduled. The isolation valve was cycled from fully open to fully closed four times. The metering valve was then operated for twenty cycles of 1/4 of the total stem travel and four cycles of 3/4 travel. The valve actuation was extremely rough during the closing portion of each cycle; in fact, at times the torque required to turn the valve exceed the 72 in-lb capacity of the rotary magnetic feedthrough. The cycling could only be completed by jogging or reversing the direction of operation when the actuation system tripped. The vacuum chamber pressure showed large but decreasing pressure fluctuations during the valve cycling operation.

The lithium temperature at the valves was increased from 1600°F to 1700°F. After 22 hours of steady state operation, the explosive valve circuit of the argon flooding safety system was connected. In less than two hours after arming the explosive valve, the safety circuit was actuated; the explosive valve fired flooding the vacuum chamber with argon and interrupting the loop power.

The explosive valve was again replaced but not armed and the vacuum chamber evacuated. Steady state operation at 1700°F was achieved in five hours; however after only two additional hours of operation, the loop again shut down as a result of an ion pump instability argon gas release. Similar "argon instability" problems were experienced during testing of the Cb-1Zr Rankine System Corrosion Test Loop and are described in detail in the final report.[2] The pressure burst that actuated the loop power safety circuit was of sufficient magnitude to also actuate the ion-pump power safety circuit (6×10^{-5} torr). Since the explosive valve of the argon flooding safety circuit was not armed, the chamber was not flooded with argon and the ion-pump could be immediately started. The loop was operated at low power until the vacuum chamber pressure was less than 5×10^{-7} torr. The loop was again brought to 1700°F steady state operation, only two hours after the shutdown. At this time, a decision was made to continue loop operation without arming the explosive valve until the probability of inadvertently flooding the chamber with argon due to an ion pump instability was minimized.

The loop completed 1000 hours total operation and the valves were subsequently cycled as scheduled. The measured break-away torque on the isolation valve after 500 hours at 1700°F was 21.0- in-lb. After only five of the ten cycles planned for the isolation valve, the actuation system seized preventing further operation. The metering valve cycles, fifty cycles of 1/4 travel and ten cycles of 3/4 travel, were completed without difficulty.

Although the planned cycles on the isolation valve were not completed the valve inlet temperature was increased to the next test temperature, 1800°F, for the next 500 hour test period. VASCO Hypercut spur and pinion gears were ordered at this time as replacements for the Mo-TZM gears in the valve actuation systems. Galling of the Mo-TZM gears was believed responsible for the valve operational difficiulties and verified during inspection at the completion of 2500 hours of testing (Figure 45).

The loop completed the scheduled 500 hours of operation at a valve temperature of 1800°F without any interruption. The metering valve was then operated for fifty cycles of 1/4 travel and ten cycles of 3/4

(2) 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.

travel. The isolation valve, which had previously seized after completing only 5 cycles of the scheduled 10 cycles of operation at 1700°F, was found to be operable and completed the scheduled 10 cycles of full travel at 1800°F. Although the valve could be operated, the valve actuation movement was extremely rough, and at times the torque required to operate the valve exceeded the 72 in-lb capacity of the rotary magnetic feedthrough. The cycling schedule could only be completed by jogging or reversing the direction of the valve operation when the magnetic rotary feedthrough tripped. The valve temperature was increased to 1900°F.

After 1000 hours of test operation at a valve inlet temperature of 1900°F, the metering valve was operated for 100 cycles of 1/4 travel and 20 cycles of 3/4 travel. An initial break-away torque of 30 in-lb was required to operate the isolation valve. The isolation valve was then operated for 20 cycles of full travel. As described previously, the valve actuation movement was extremely rough, and at times the torque required to operate the valve exceeded the 72 in-lb capacity of the rotary magnetic feedthrough. The cycling schedule could only be completed by jogging or reversing the direction of the valve operation when the magnetic rotary feedthrough tripped.

The ball bearing housing temperature of the valve as a function of the valve inlet temperature is shown in Figure 40. The actual operating temperature is significantly less than the 1000°F estimated safe operating temperature based on hardness data for VASCO Hypercut bearings.

The average electrical power input to the lithium heater as a function of average loop operating temperature is shown in Figure 41. The single-phase AC electric power was measured by a Hall effect wattmeter at the output of the low-voltage power transformer.

The lithium flow rate vs. valve travel for the metering valve, obtained in the pretest checkout of the loop, is shown in Figure 42. The experimental data was adjusted for a constant 10 psi pressure drop for comparison with the water flow tests conducted by Hoke, Inc.

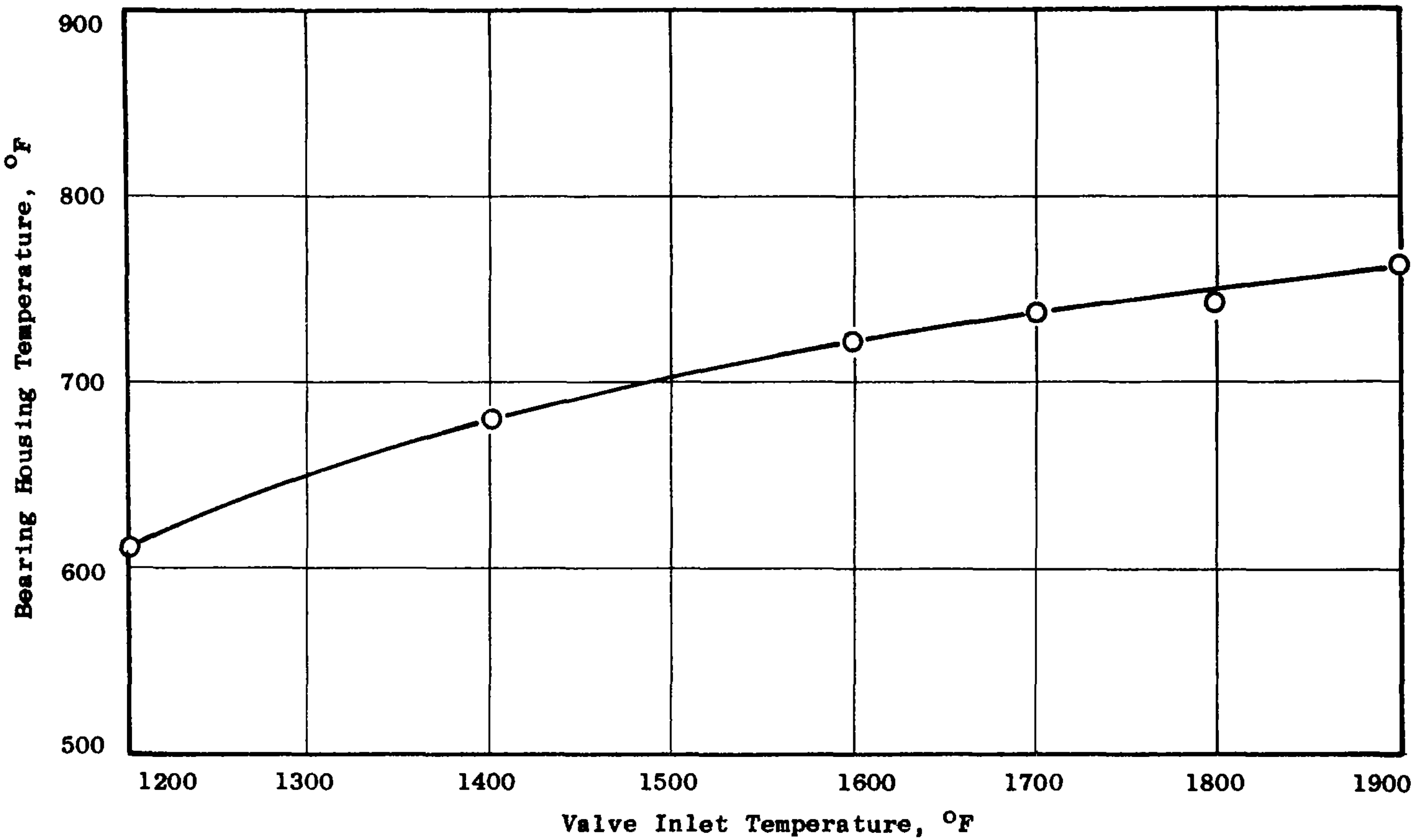


Figure 40. **Bearing Housing Temperature as a Function of Valve Inlet Temperature During High Temperature Alkali Metal Valve Test.**

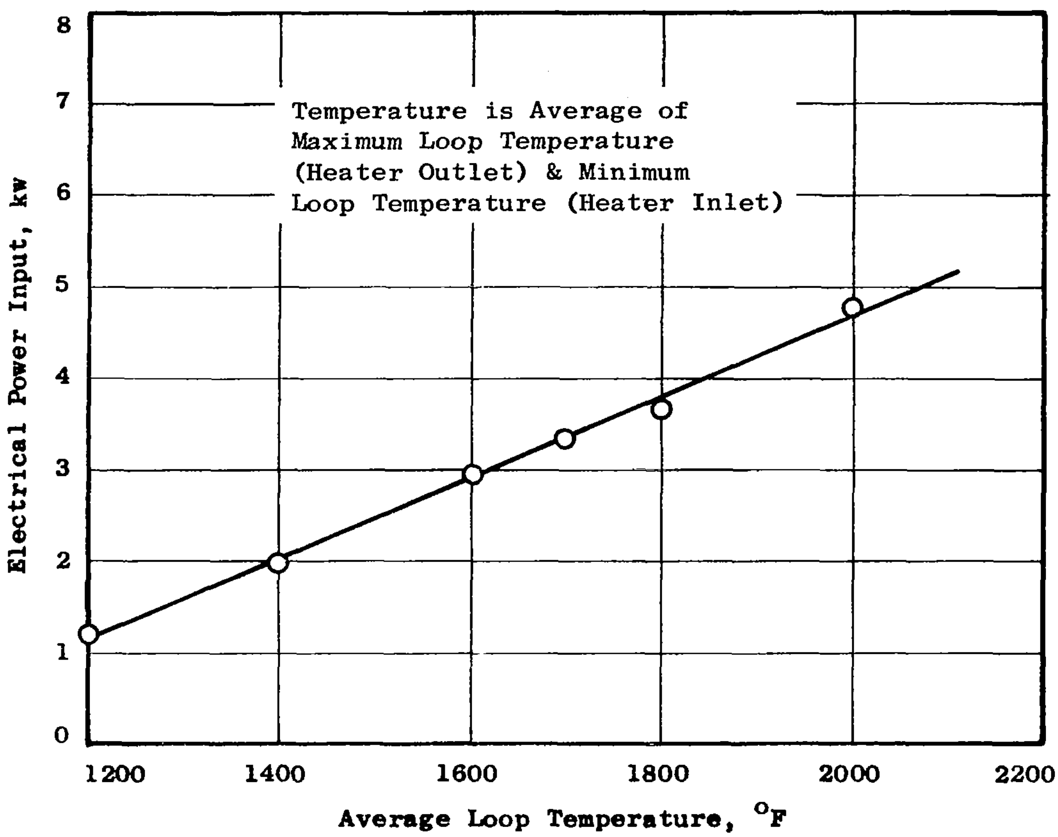


Figure 41. **Average Loop Temperature as a Function of Electrical Power Input for High Temperature Alkali Metal Valve Loop.**

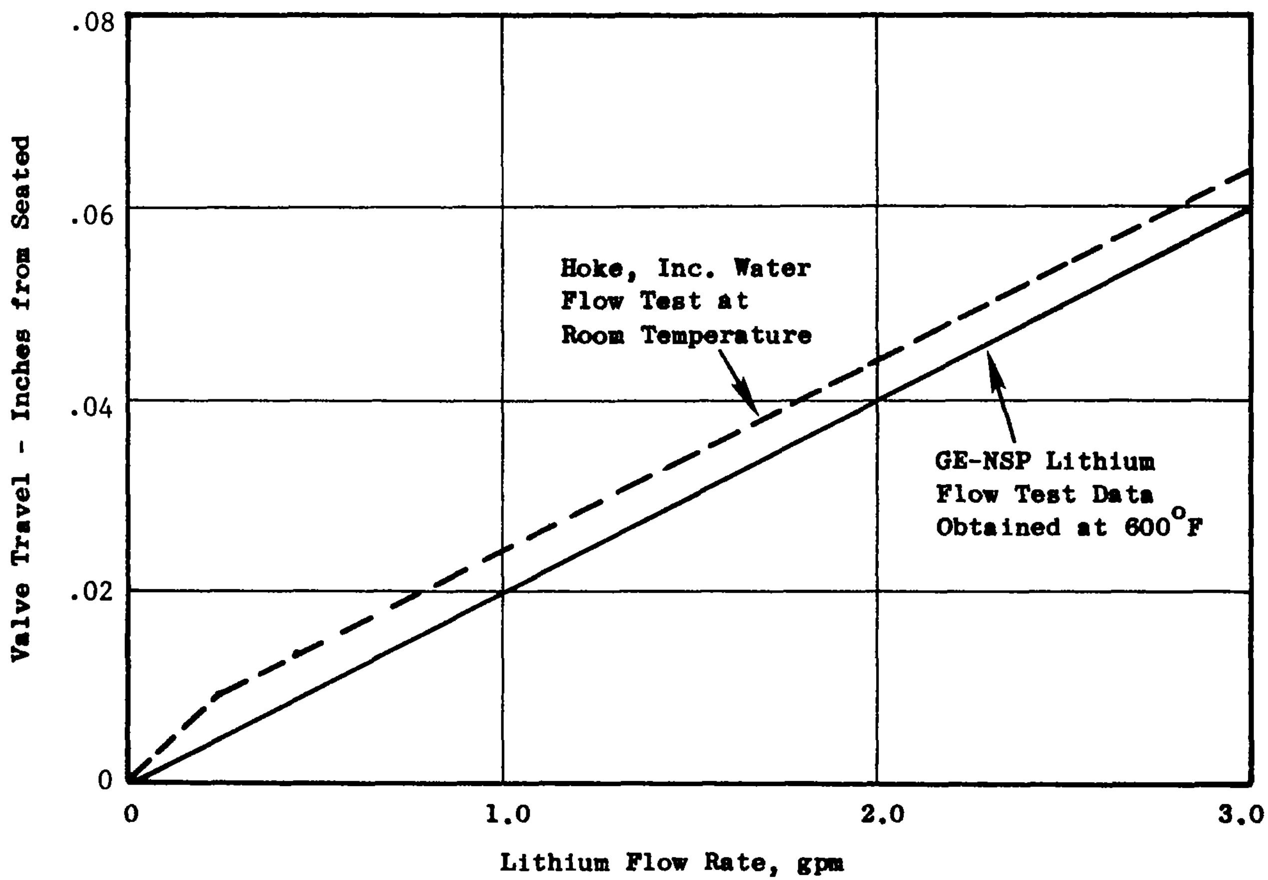


Figure 42. Comparison of Water and Lithium Flow Test Data for High Temperature Alkali Metal Valve Based on 1900°F Lithium and 10 psi Pressure Drop Across the Valve.

The output voltage of the permanent magnet flowmeter as a function of the lithium flow rate is shown in Figure 43. The flowmeter was calibrated by making an energy balance across the lithium heater during loop operation. The calibration curve is for loop operation at 1900°F which is the design operating temperature for the steady state conditions during the 1500-to 2500-hour period of loop operation.

The test loop was shutdown for inspection after having successfully completed 2500 hours of operation. The last 1000 hours of steady state operation was completed without interruption at a maximum loop temperature of 2100°F and a valve inlet temperature of 1900°F. Typical steady state conditions during this test period are shown in Figure 44.

The vacuum chamber was opened for visual inspection and extreme wear of the Mo-TZM pinion and spur gears was observed as shown in Figure 45. Particles pulled out of the gear surfaces were found on the top of the gear mount. These observations verified the foregone conclusion that Mo-TZM galling accounted for the resistance observed in actuation of the valves.

The Mo-TZM gears were replaced with VASCO Hypercut spur and pinion gears. The VASCO Hypercut gears were previously heat treated to a hardness of R_c65 and oil blackened to improve wear and corrosion resistance.

The Cb-1Zr foil on the upper portion of the lithium heater was removed to replace thermocouples which had become inoperative during the 2500 hours of testing.

Some of the inner layers of the foil in this area had formed into sponge-like masses. Chemical analyses of the degraded foil and new foil indicated no constituents or changes which would explain the foil appearance. The foil was replaced with new foil after replacement of the thermocouples.

Following a check of loop instrumentation the vacuum chamber was closed and pumpdown initiated.

The loop was brought to temperature and logging of test hours commenced. The operation conditions for the next 2500 hours were:

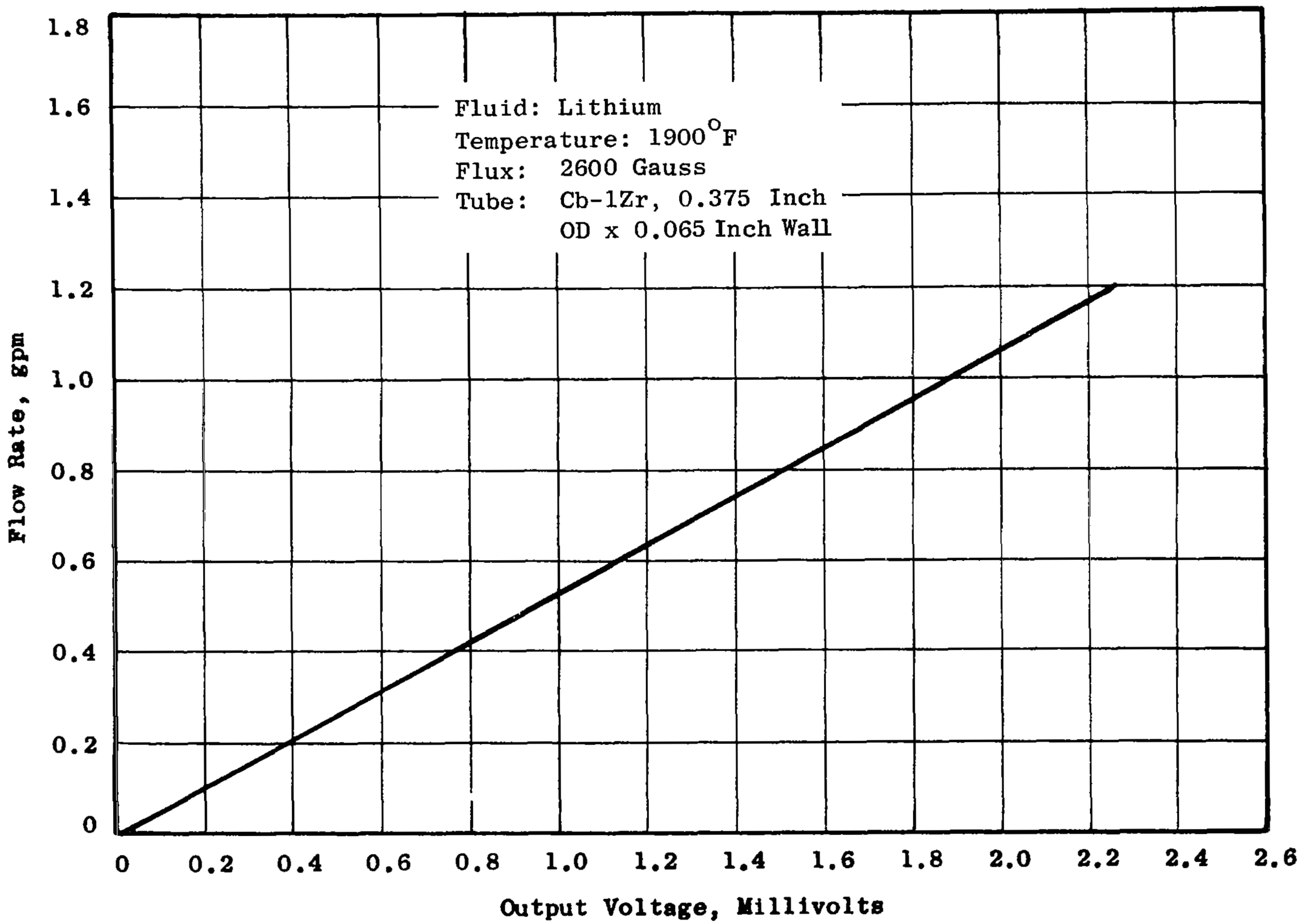


Figure 43. **Output Voltage Versus Flow Rate for the High Temperature Alkali Metal Valve Loop.**

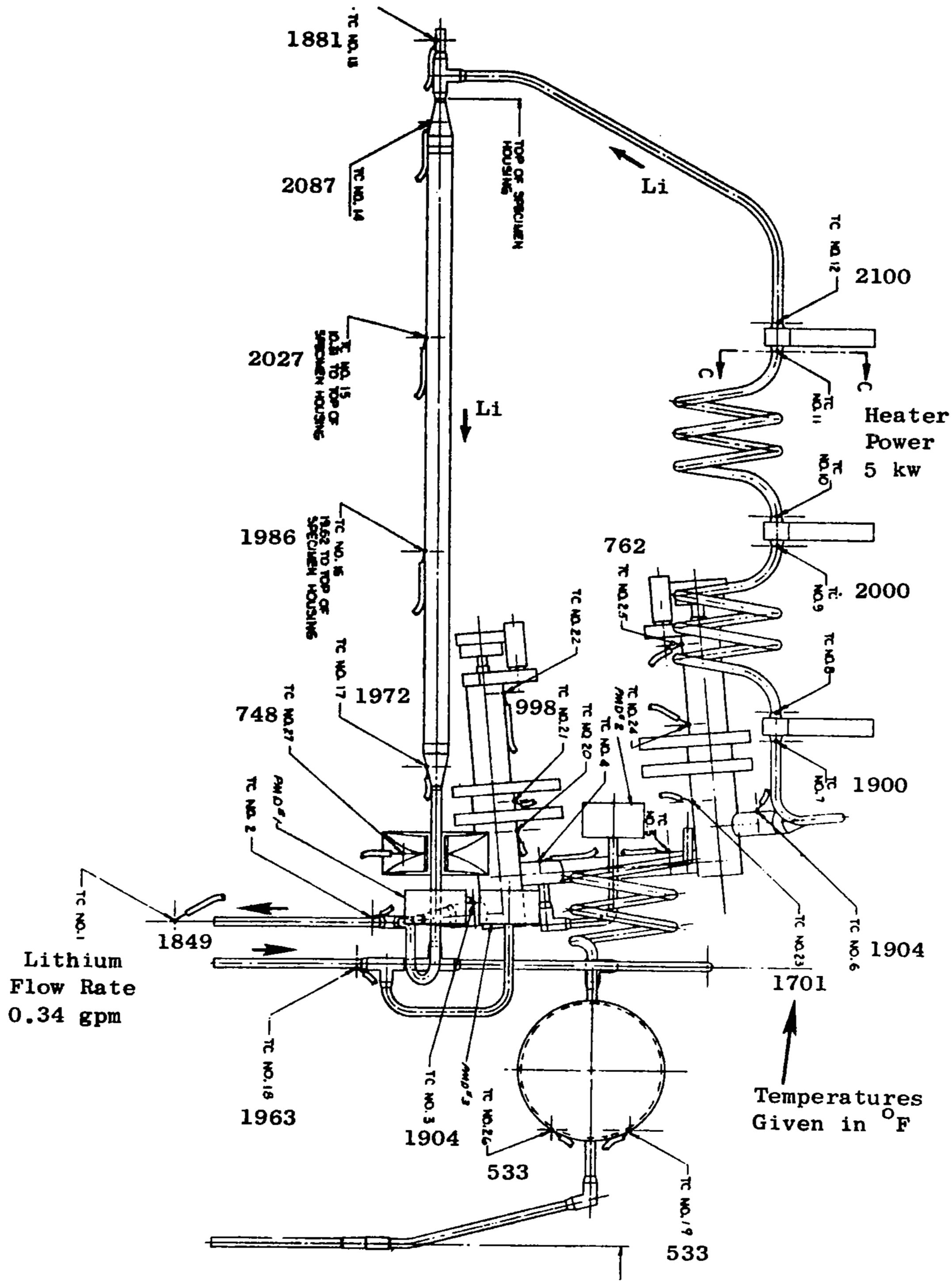


Figure 44. Temperature Distribution of the High Temperature Alkali Metal Valve Loop on 1/13/69 After 1667 Hours of Test Operations.

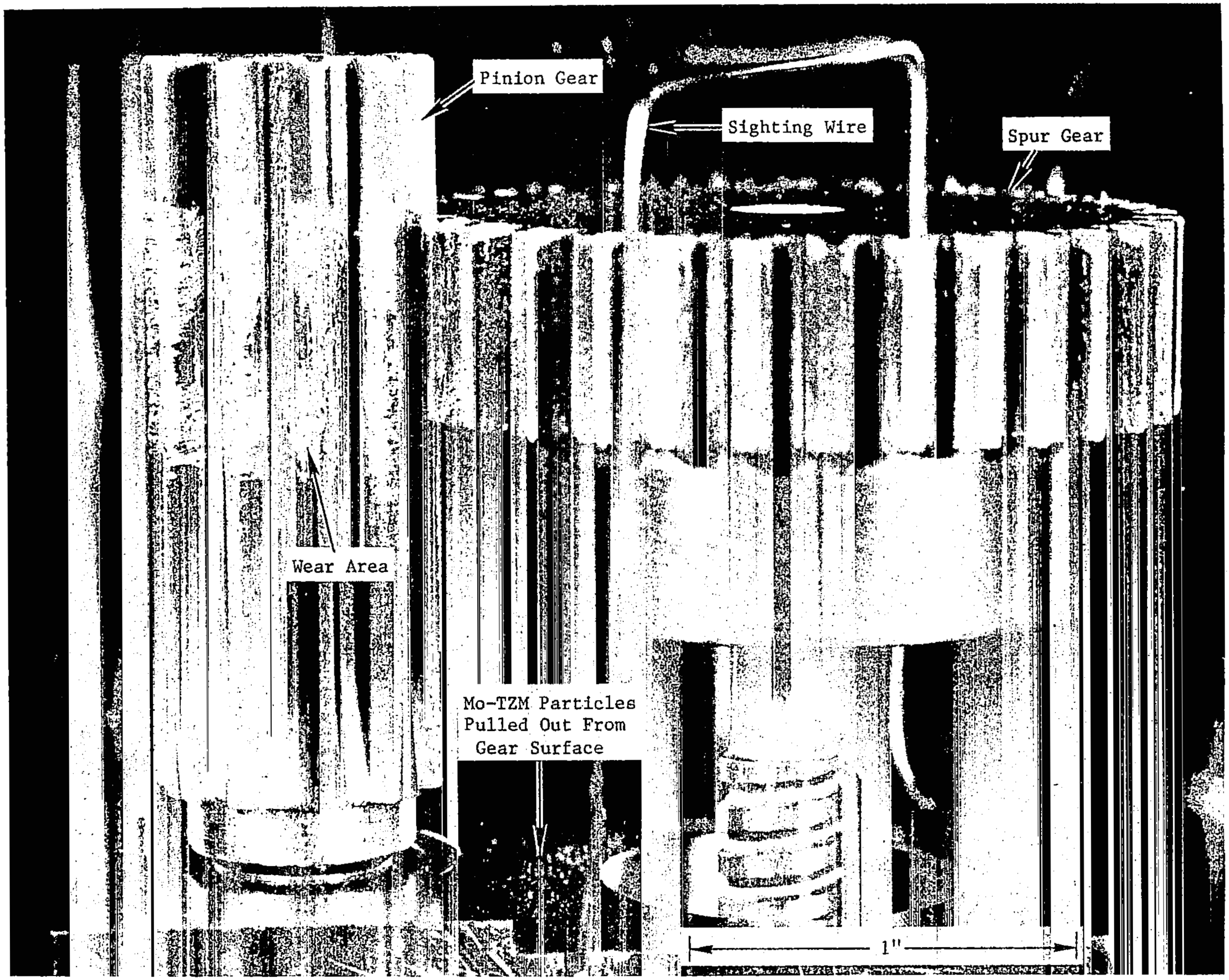


Figure 45. Mo-TZM Spur and Pinion Gears on the Metering Valve of the High Temperature Alkali Metal Valve Test After 2500 Hours of Testing. (P69-2-26B)

Lithium Heater Outlet	2100°F ± 50°F
Lithium Heater Inlet	1900°F ± 25°F
Lithium Flow	0.42 gpm ± 0.05 gpm

During this test period the valves were cycled at 500 hour intervals. The torque required to operate the isolation valve decreased from 16.8 in-lb after a total of 3000 hours of testing to 8 in lb at the completion of the 5000 hour test. These values can be compared to a torque of 40.8 in-lb required to operate the valve before replacing the Mo-TZM pinion and spur gears with VASCO Hypercut gears.

F. Posttest Sampling of the Lithium

The loop temperature was lowered to 1000°F in preparation for sampling the lithium. The draining and sampling lines were attached to the loop facility as shown in Figure 46. Following helium mass spectrometer leak checking the sampling system was outgassed at 375-450°F until the pressure rise rate was less than 10 micron-liters per minute at temperature. The lithium from the loop was drained into the surge tank. The loop was then pressurized from the control console through the loop blow down line. Valves (1) and (2) were opened intermittently to flush the dump line with lithium prior to sampling. Valve (3) was then opened and a sample tube was flushed by overflowing lithium into the sampling reservoir. The sample tube was cooled to room temperature and removed for analysis of the lithium. The remaining system was left intact should additional sampling be required.

The analysis of the lithium is shown in Table VIII. The oxygen concentration increase in the lithium is consistent with the fact that at the loop operating temperatures lithium will remove oxygen from the Cb-1Zr loop material,* and the volume of Cb-1Zr to volume of lithium is sufficiently high. The lithium in the loop was subsequently drained into the dump tank through valves (1) and (2) by pressurizing the loop through the blow down line.

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* Oxygen decreases in Cb-1Zr loop components indicated in Table XIII of this report.

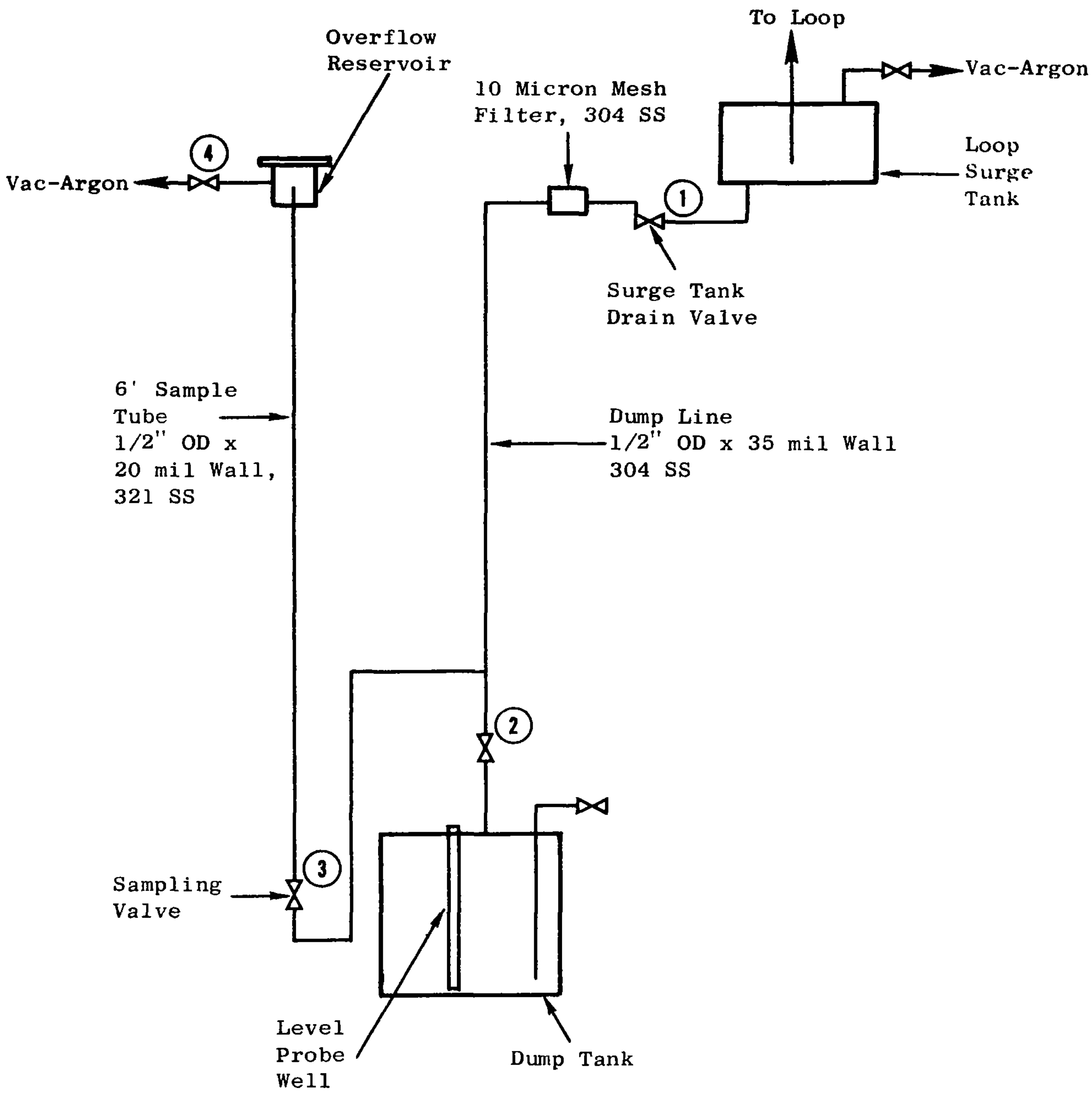


Figure 46. Schematic Diagram of the Draining and Sampling of the Lithium From the High Temperature Valve Loop.

TABLE VIII

CHEMICAL ANALYSIS OF LITHIUM

HIGH TEMPERATURE ALKALI METAL VALVE LOOP

Element	Concentration, ppm in Lithium	
	Before Test[a]	After Test[b]
O	13	193, 221
N	25, 33	1, 1
C	47	34, 51, 70
Ag	< 5	< 5
Al	5	5
B	< 50	< 75
Ba	-	-
Be	< 5	< 5
Ca	5	5
Cb	< 25	< 25
Co	< 5	< 5
Cr	< 5	< 5
Cu	< 5	< 5
Fe	5	5
Mg	5	5
Mn	< 5	< 5
Mo	< 5	< 5
Na	< 50	< 75
Ni	< 5	< 5
Pb	< 50	< 50
Si	5	5
Sn	< 25	< 25
Sr	-	-
Ti	< 25	< 25
V	< 25	< 25
Zr	< 25	< 25

(a) Removed from the loop before testing was initiated.

(b) Removed from the loop following 5000 hours of testing.

VII. POSTTEST EVALUATION

A. Tensile Test Specimens

1. Visual Examination

As described previously sheet tensile specimens of ASTAR 811C, ASTAR 811CN, ASTAR 811, T-111, T-222, and W-Re-Mo Alloy 256 were exposed to flowing lithium in a specimen housing which was part of the loop circuit.

Prior to sectioning the specimen housing, radiographs were obtained to delineate the position of the specimens within. The radiographs which were taken at three different locations revealed that the specimens were perfectly aligned within the housing.

The specimen housing was subsequently opened in a glove box equipped with a recirculating argon purification system capable of maintaining the atmosphere at less than 1 ppm oxygen and less than 1 ppm water vapor. The tensile test specimens and the specimen housing were then cleaned of all residual lithium by reaction with liquid ammonia, followed by a final deionized water rinse.

Upon completion of cleaning the tensile test specimens were carefully examined visually. There were no significant changes observed in any of the specimens.

2. Weight Change Measurements

The cleaned specimens 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 IX. It can be seen that the direction of the weight changes, i.e. plus or minus, are consistent for the two specimens of each alloy. The magnitude of the changes are also consistent for the ASTAR 811CN, T-111, and W-Re-Mo Alloy 256 specimens. Overall the changes are small and their significance is difficult to ascertain.

3. Chemical Analysis

Samples were removed from the corners of the tensile specimen tabs and analyzed for oxygen, nitrogen, and hydrogen by the vacuum fusion

TABLE IX

SPECIMEN WEIGHT MEASUREMENTS BEFORE AND AFTER 5000 HOURS OF EXPOSURE TO LITHIUM IN THE HIGH TEMPERATURE ALKALI METAL VALVE LOOP

	Specimen	Weight Before Test	Weight After Test	Change
Entrance to Specimen Test Section 2087°F	ASTAR 811C	16.9840 g.	16.9850 g.	+ 0.0010 g.
		16.7661 g.	16.7665 g.	+ 0.0004 g.
	ASTAR 811CN	15.7695 g.	15.7691 g.	- 0.0004 g.
		15.4443 g.	15.4437 g.	- 0.0006 g.
	ASTAR 811	15.4543 g.	15.4546 g.	+ 0.0003 g.
Lithium Flow ↓		15.2836 g.	15.2846 g.	+ 0.0010 g.
	T-111	15.8614 g.	15.8594 g.	- 0.0020 g.
		15.7623 g.	15.7604 g.	- 0.0019 g.
	T-222	16.5066 g.	16.5054 g.	- 0.0012 g.
		16.3279 g.	16.3277 g.	- 0.0002 g.
	W-Re-Mo Alloy 256	7.9034 g.	7.9053 g.	+ 0.0019 g.
		7.9024 g.	7.9041 g.	+ 0.0017 g.
Exit of Specimen Test Section 1972°F				

technique and for carbon by the combustion conductometric technique. Samples were obtained in the as-received, pretest annealed and posttest conditions. The results of these analyses are presented in Table X. In general little change in oxygen, nitrogen, hydrogen or carbon concentration was observed as a result of the one hour at 2200°F anneal. A slight decrease in oxygen concentration in the ASTAR 811CN alloy specimens and a slight increase in carbon concentration in the ASTAR 811 alloy specimens was observed after annealing.

Chemical analyses of the specimens after exposure to the flowing lithium for 5000 hours showed numerous changes in interstitial element concentrations. The ASTAR 811C alloy specimens showed only an increase in nitrogen content. The ASTAR 811CN alloy specimens lost carbon, nitrogen and oxygen. These losses may explain the observed weight losses reported earlier. It is interesting to note that the changes in nitrogen concentration in the ASTAR 811C and ASTAR 811CN alloy specimens (ASTAR 811C increased and ASTAR 811CN decreased) resulted in the final nitrogen concentrations being approximately equal in the two alloys. The ASTAR 811 alloy showed only a decrease in oxygen concentration. Again it is interesting that the ASTAR 811 alloy did not pick-up nitrogen as was observed in the ASTAR 811C alloy specimens.

The T-111 alloy specimens exhibited a nitrogen increase and an oxygen loss. The observed weight loss previously discussed can, therefore, not be accounted for by interstitial changes alone. The T-222 alloy specimens also picked up nitrogen and lost oxygen as did the T-111 alloy, but the nitrogen increase was significantly larger than that observed in the T-111 alloy.

The W-Re-Mo Alloy 256 alloy specimens showed increases in carbon, nitrogen and oxygen concentration after exposure to lithium. These specimens were the only specimens which showed an increase in oxygen concentration.

It is difficult to draw any specific conclusions regarding the observed changes in interstitial concentrations in view of the variety of materials involved as well as the thermal gradient present in the tensile test section but it appears that some nitrogen mass transfer has occurred as indicated by the analysis of the tantalum alloy specimens.

TABLE X

RESULTS OF TENSILE SPECIMEN CHEMICAL ANALYSIS

Specimen		Pretest Analyses, ppm As-Received				Pretest Analyses, ppm One Hour Anneal at 2200°F				Posttest* Analyses, ppm 5000 Hour Exposure to Lithium				Tensile Specimen Exposure Temperature Gradient
		C	N	O	H	C	N	O	H	C	N	O	H	
ASTAR 811C		237,249	4,6	10,13	2,3	270,284	4,4	10,9	< 1,< 1	251,254	30,32	5,8	< 1,< 1	Entrance to Specimen
	Avg	243	5	12	3	277	4	10	< 1	253	31	7	< 1	Test Section 2087°F
ASTAR 811CN		173,177	80,71	38,34	3,2	167,205	60,74	16,16	< 1,< 1	131,133	31,30	7,7	< 1,< 1	
	Avg	175	76	36	3	186	67	16	< 1	132	31	7	< 1	
ASTAR 811		11,16	11,7	9,10	3,2	31,48	11,6	12,11	< 1,< 1	29,29	8,6	4,3	< 1,< 1	Lithium Flow ↓
	Avg	14	9	10	3	40	9	12	< 1	29	7	4	< 1	
T-111		19,18	3,4	38,33	2,2	21,34	3,2	36,32	< 1,< 1	30,31	41,46	4,6	< 1,< 1	
	Avg	19	4	36	2	28	3	34	< 1	31	44	5	< 1	
T-222		119,101	5,5	66,66	4,4	83,107	4,6	45,49	< 1,< 1	99,107	111,115	9,4	< 1,< 1	
	Avg	110	5	66	4	95	5	47	< 1	103	113	7	< 1	Exit of Specimen
W-Re-Mo Alloy 256		8,4	< 1,< 1	11,8	< 1,< 1	14,6	1,1	8,8	< 1,< 1	20,14	12,4	17,16	< 1,< 1	Test Section 1972°F
	Avg	6	< 1	10	< 1	10	1	8	< 1	17	8	17	< 1	

*Annealed one hour at 2200°F during postweld anneal.

4. Tensile Testing

Room temperature and 2000°F vacuum tensile tests were performed on specimens in the as-received, pretest annealed, and posttest conditions. The results of the room temperature tensile tests are shown in Table XI and the results of the 2000°F vacuum tensile tests are shown in Table XII.

All materials were in the recrystallized condition when received with the exception of the ASTAR 811C and T-222 alloys. The ASTAR 811CN, ASTAR 811, and T-111 alloys had been given a recrystallization anneal of 1 hour at 3000°F while the W-Re-Mo Alloy 256 was given a recrystallization anneal of 20 minutes at 2550°F. For the purposes of total test data evaluation it would have been desirable for all specimens to be in the fully recrystallized condition prior to exposure to lithium. Those specimens in the pretest annealed condition had been given a simulated postweld anneal of 1 hour at 2200°F. The posttest specimens had also been annealed for 1 hour at 2200°F during the postweld annealing of loop components during fabrication, in addition to being exposed to lithium at 2100°F for 5000 hours.

As would be anticipated, the ultimate tensile and yield strengths of all specimens in each testing condition, i.e., pretest as-received, pretest annealed, and posttest, were less at 2000°F as compared to the room temperature results. Associated with the decrease in strength was an increase in elongation with the exception of the ASTAR 811CN samples whose ductility at 2000°F was slightly less than its room temperature ductility.

The pretest anneal and lithium exposure of the T-111 alloy specimens had little effect upon its ultimate strength. The T-111 specimens exhibited a room temperature ultimate tensile strength of 94,000 psi in the posttest and pretest annealed condition as compared to 97,000 psi in the as-received condition. The lithium exposed T-111 specimens exhibited a slightly higher ultimate strength at 2000°F (56,800 psi) compared to the strength of as-received specimens (54,400 psi). The spread cannot be considered significant in either case. The yield strength and ductility of the T-111 alloys exhibited little or no change due to exposure to the lithium in either the room temperature

TABLE XI

RESULTS OF ROOM TEMPERATURE TENSILE TESTS FOR HIGH TEMPERATURE ALKALI METAL VALVE LOOP

	Specimen	U.T.S. (ksi)	0.02% Y.S. (ksi)	0.2% Y.S. (ksi)	RA (%)	Elongation (%)
Pretest - As Received						
	ASTAR 811C	168.0	137.0	159.0	39.0	5.0
	ASTAR 811CN	116.0	104.0	104.0	42.0	24.0
	ASTAR 811[a]	88.7	71.0	74.5	59.5	36.0
	T-111[a]	96.9	89.0	93.0	83.0	35.0
	T-222	132.0	121.0	128.0	73.0	12.0
	W-Re-Mo Alloy 256[a]	163.0	142.0	144.0	5.5	11.0
Pretest - Annealed 1 Hour at 2200°F						
	ASTAR 811C	132.0	112.0	117.0	30.5	13.0
	ASTAR 811CN[a]	105.0	93.9	96.0	54.5	30.0
	ASTAR 811[a]	87.5	71.9	76.1	68.0	33.5
	T-111[a]	93.8	87.5	90.9	72.5	36.5
	T-222[a]	120.0	101.0	106.0	68.0	24.0
	W-Re-Mo Alloy 256[a]	162.0	136.0	143.0	10.5	11.5
Posttest[b] - Annealed 1 Hour at 2200°F During Postweld Anneal of Loop						
Entrance to Specimen Test Section 2087°F	ASTAR 811C[a]	94.2	72.5	78.0	68.0	28.0
↓	ASTAR 811CN	90.5	62.3	64.4	62.0	33.0
Lithium Flow	ASTAR 811[a]	87.5	73.5	73.5	62.5	30.0
↓	T-111[a]	93.9	82.0	87.9	65.0	34.0
↓	T-222[a]	112.0	91.5	95.0	64.0	25.5
Exit of Specimen Test Section 1972°F	W-Re-Mo Alloy 256[a]	160.0	135.0	142.0	8.0	9.0

(a) Exhibited double yielding on load deflective curve.

(b) 5000 hour exposure to flowing lithium through indicated temperature gradient.

TABLE XII

RESULTS OF 2000°F VACUUM TENSILE TESTS FOR HIGH TEMPERATURE ALKALI METAL VALVE LOOP

Specimen	Vacuum Torr (x 10^{-6})	U.T.S. (ksi)	0.02% Y.S. (ksi)	0.2% Y.S. (ksi)	RA (%)	Elongation (%)
Pretest - As-Received						
ASTAR 811C	3.0	82.8	51.1	62.5	58.5	18.0
ASTAR 811CN	3.8	58.5	29.8	34.2	53.0	22.0
ASTAR 811	2.8	43.7	19.3	23.7	53.5	35.0
T-111	4.2	54.4	28.6	30.0	62.5	40.0
T-222	2.4	78.8	49.9	61.6	49.0	14.5
W-Re-Mo Alloy 256	2.2	67.5	51.0	58.0	40.0	20.0
Pretest - Annealed 1 Hr @ 2200°F						
ASTAR 811C	1.0	76.9	47.7	59.5	40.0	11.5
ASTAR 811CN	2.2	56.5	30.0	33.4	61.5	28.0
ASTAR 811	3.8	44.0	22.2	24.7	68.0	33.0
T-111	1.8	55.5	28.0	30.6	65.0	39.0
T-222	4.5	73.0	46.7	56.7	48.0	20.0
W-Re-Mo Alloy 256	3.4	66.8	47.4	54.4	27.0	15.0
Posttest* - Annealed 1 Hr @ 2200°F During Postweld Anneal of Loop						
ASTAR 811C	1.1	50.2	23.7	27.6	69.5	31.5
ASTAR 811CN	1.3	47.9	26.4	30.8	75.5	29.0
ASTAR 811	2.4	43.9	21.2	24.0	67.0	36.0
T-111	3.2	56.8	25.0	30.0	77.0	36.5
T-222	3.2	68.6	41.6	47.8	43.5	26.5
W-Re-Mo Alloy 256	1.7	69.2	50.5	57.6	33.5	15.5

Entrance to Specimen Test Section 2087°F

↓ Lithium Flow ↓

Exit of Specimen Test Section 1972°F

*5000 hour exposure to flowing lithium through indicated temperature gradient.

or 2000°F tensile tests. It is concluded that the lithium exposure had no effect upon the strength and ductility of the T-111 alloy.

The ultimate and yield strength of the ASTAR 811CN alloy specimens although greater than the T-111 alloy samples in the as-received condition declined to a strength less than that of the T-111 alloy after exposure. This characteristic was true of both the room temperature and 2000°F test data.

The effects of the lithium exposures on the W-Re-Mo Alloy 256 were negligible. There was a slight reduction in ultimate and yield strengths of the lithium exposed specimens as compared to the as-received specimens in both the room temperature and 2000°F tests. The tungsten alloy specimens although exhibiting a greater strength than the T-111 alloy specimens in both pretest and posttest conditions showed a lesser ductility. The tungsten alloy specimens showed a greater percentage reduction of strength between room temperature and 2000°F than did the T-111.

The lithium exposure of the ASTAR 811 specimens had negligible effect upon their strength and ductility at both room temperature and 2000°F. Although exhibiting comparable ductility to the T-111 alloy specimens the ASTAR 811 alloy had less strength than the T-111 alloy.

The effects of the lithium exposure on the T-222 alloy showed an approximate 14% reduction in strength of the posttest sample compared to the pretest sample at both room temperature and 2000°F. Similarly, the ASTAR 811C alloy showed an approximate 40% reduction in strength of the posttest specimen compared to the pretest specimen at both room temperature and 2000°F. Considering weight change measurements, chemical analyses, and metallographic examination (to be discussed later) the reduction of strength and increase in ductility is most likely a result of recrystallization of the T-222 and ASTAR 811C specimens during the lithium exposure and not directly due to any effects of the lithium.

The strength of the lithium exposed ASTAR 811C alloy specimens at room temperature are comparable to the T-111 alloy specimens but exhibit less ductility. At 2000°F the ASTAR 811C specimens exhibit less strength and ductility than do the T-111 alloy specimens.

The strength of the lithium exposed T-222 alloy specimens is greater than that of the T-111 alloy at both room temperature and 2000°F while exhibiting a lesser ductility in both cases.

Load deflection curves of the room temperature tensile tests showed that many specimens exhibited a double yielding characteristic as noted in Table XI. There was no consistency to the type of specimen or testing condition for the presence of the double yielding. However, as will be explained in the metallographic examination section, some tensile specimens exhibited longitudinal cracking during fracture. All specimens exhibiting this type of fracture produced a double yielding characteristic on the load deflection curve.

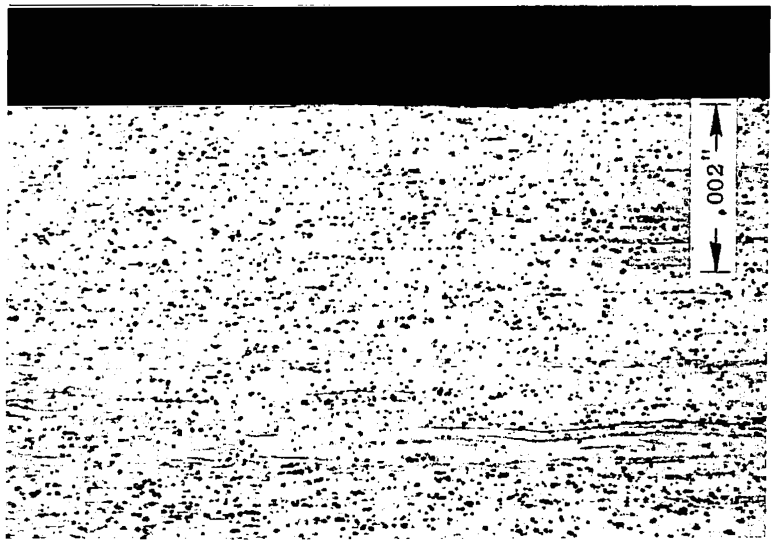
5. Metallographic Examination

Metallographic examination of the tensile specimens after exposure to the lithium revealed no evidence of corrosion. Representative photomicrographs of each specimen are shown in Figures 47 through 52.

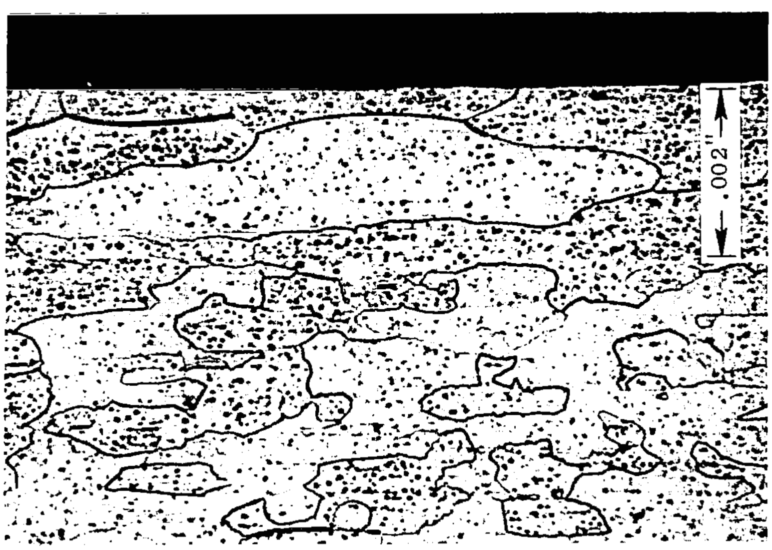
The ASTAR 811C and T-222 alloy specimens which had not been recrystallized prior to testing were found to be recrystallized after the 5000 hour exposure to lithium at 2100°F. Figures 53 and 54 show the pretest and posttest conditions of the ASTAR 811C and T-222 alloy specimen respectively.

During tensile testing a few specimens exhibited longitudinal cracking extending from the fracture surface. A typical example of this type of cracking is shown in the photomicrograph of an ASTAR 811C specimen tested at room temperature in Figure 55. The longitudinal cracking occurred in both room temperature and 2000°F tensile specimens although the double yielding characteristic was only observed on specimens tested at room temperature, as described above. A few specimens which had no obvious longitudinal cracks were found to have signs of incipient cracking in grain boundaries when viewed at higher magnifications. To explain the cause and effect of this cracking additional study would be required beyond the scope of this program.

Metallographic examination revealed that none of the specimens exhibited grain growth to any significant degree.



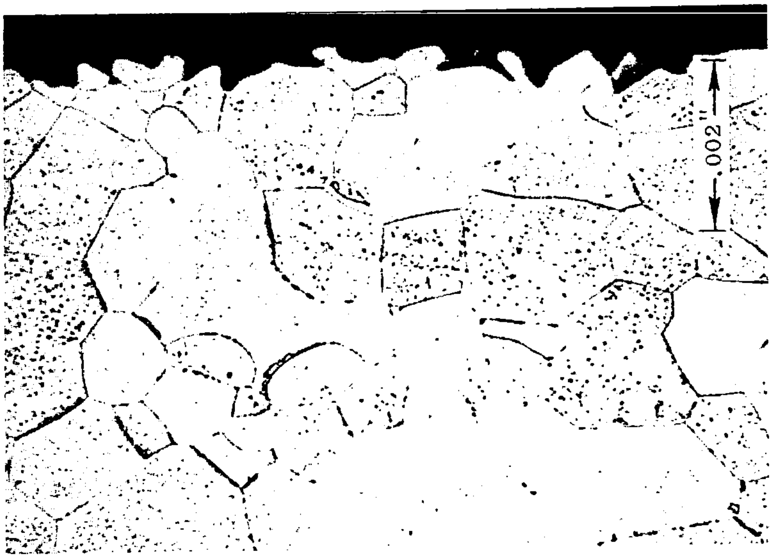
MB-1299J



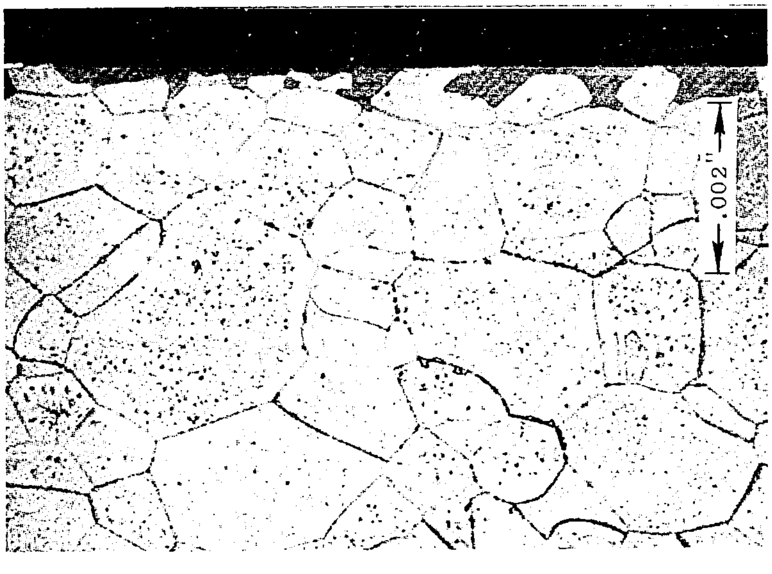
MB-1299M

Figure 47. Microstructures of ASTAR 811C Alloy Before and After Exposure to Flowing Lithium for 5000 Hours at 2100°F. No Corrosion was Observed. The Alloy Recrystallized During the Test Exposure.

Etchant: $30gNH_4F-20mlHNO_3-20mlH_2O$



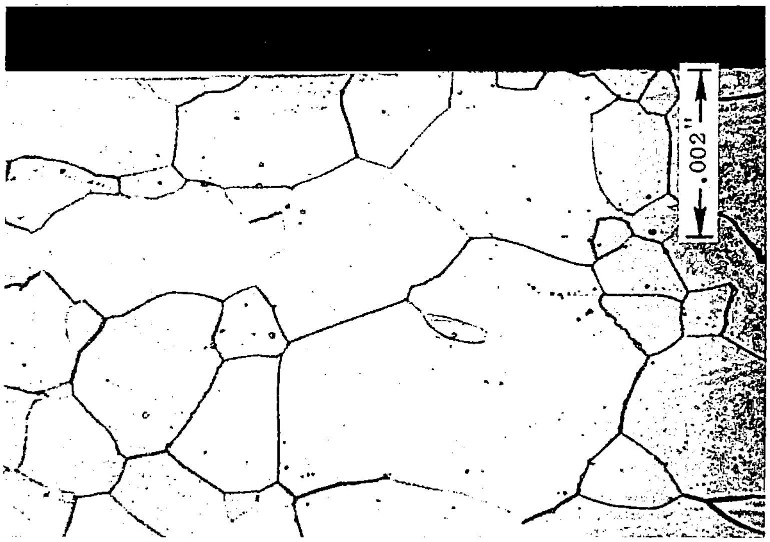
MB-1300J



MB-1300I

Figure 48. Microstructures of ASTAR 811CN Alloy Before and After Exposure to Flowing Lithium for 5000 Hours at 2100°F. No Corrosion was Observed. The Surface Roughening is Believed to be a Result of Acid Pickling Prior to Loop Assembly.

Etchant: $30gNH_4F-20mlHNO_3-20mlH_2O$



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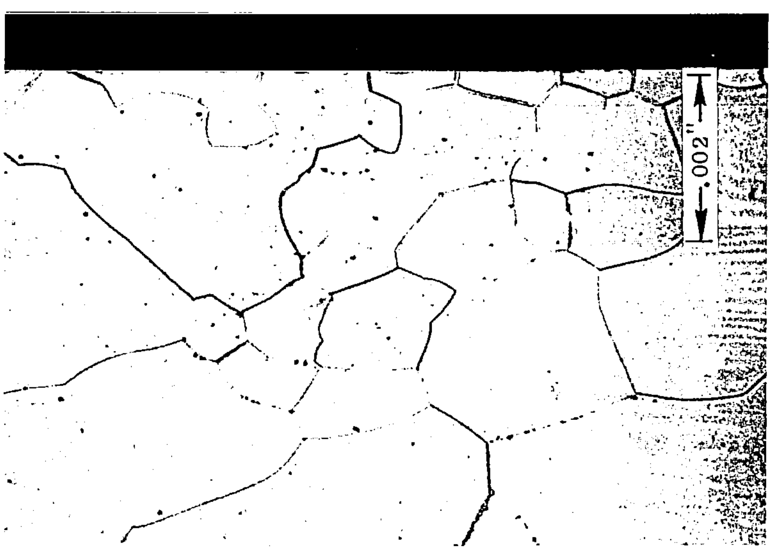


MB-1201E

Figure 49. Microstructures of ASTAR 811 Alloy Before and After Exposure to Flowing Lithium for 5000 Hours at 2100°F. No Corrosion was Observed.

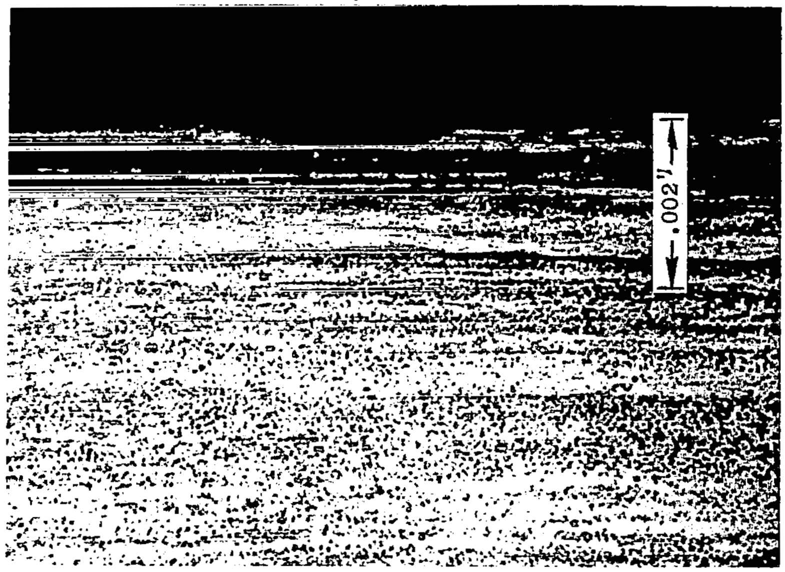
Etchant: $30gHN_4F-20mlHNO_3-20mlH_2O$

MB-1302E

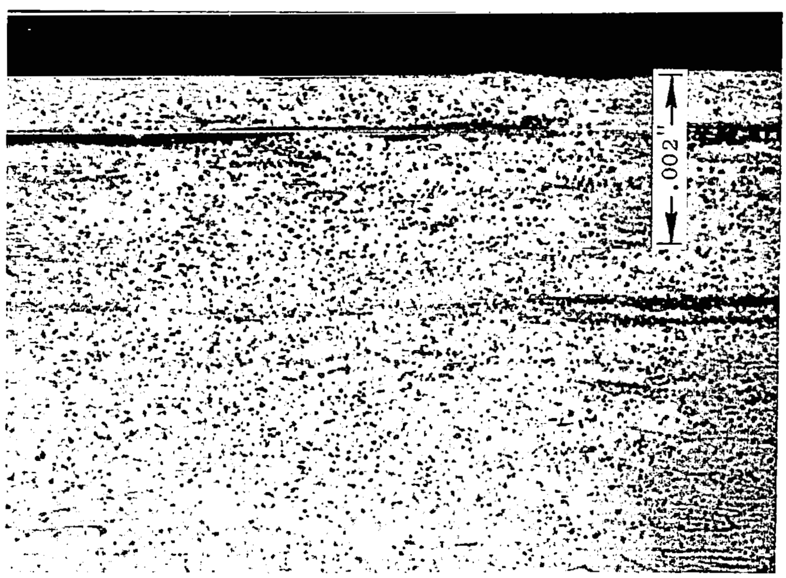
MB-1302G

Figure 50. Microstructures of T-111 Alloy Before and After Exposure to Flowing Lithium for 5000 Hours at 2100°F. No Corrosion was Observed. The Precipitate in the Posttest Microstructure Results from the Thermal Exposure.

Etchant: $30gNH_4F-20mlHNO_3-20mlH_2O$



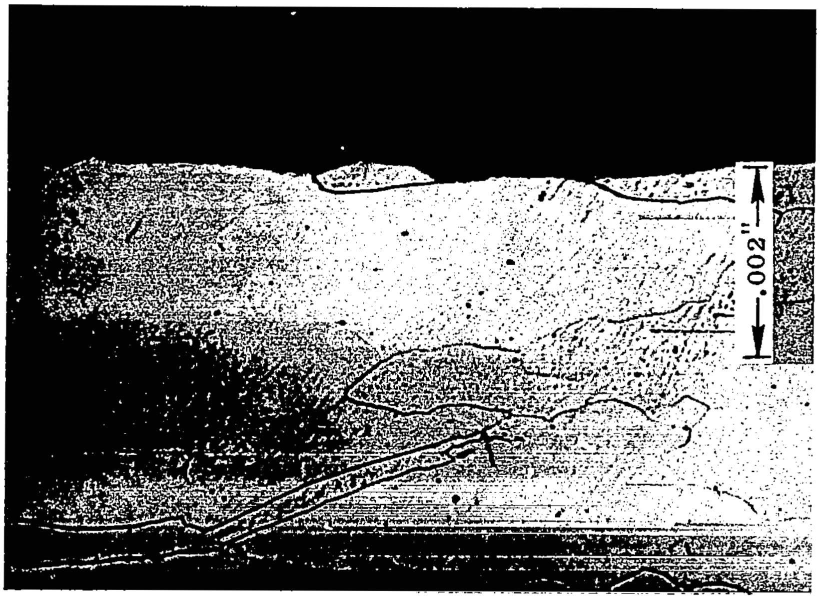
D580212



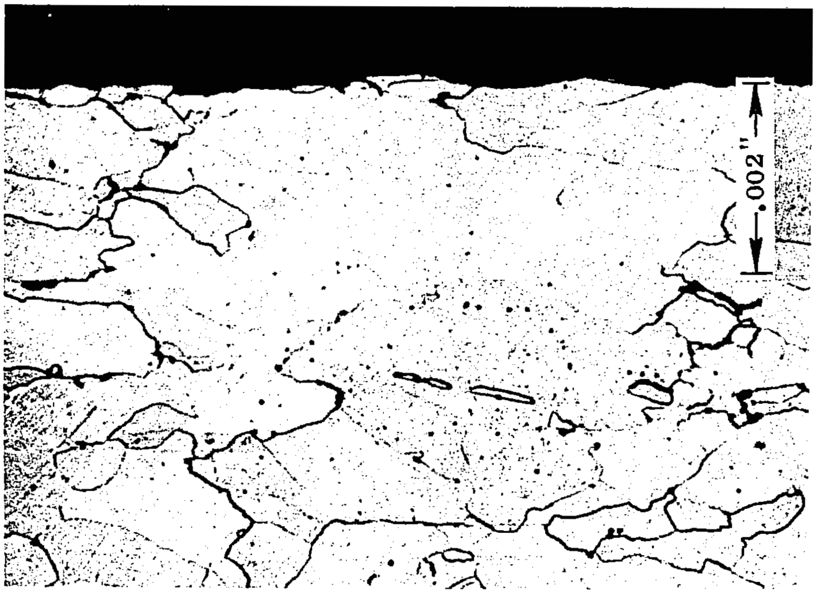
MB-1303H

Figure 51. Microstructures of T-222 Alloy Before and After Exposure to Flowing Lithium for 5000 Hours at 2100°F. No Corrosion was Observed. The Alloy Partially Recrystallized During Testing.

Etchant: $30gNH_4F-20mlHNO_3-20mlH_2O$



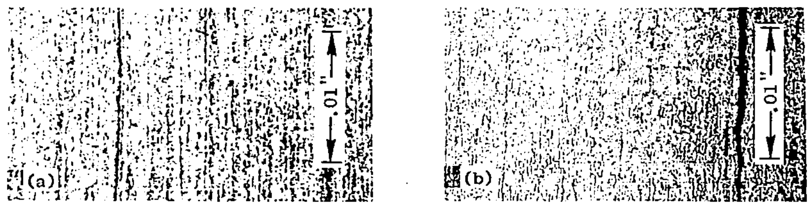
MB-1304E



MB-1304D

Figure 52. Microstructures of W-Re-Mo Alloy 256 Before and After Exposure to Flowing Lithium for 5000 Hours at 2100^{o}F. No Corrosion was Observed.

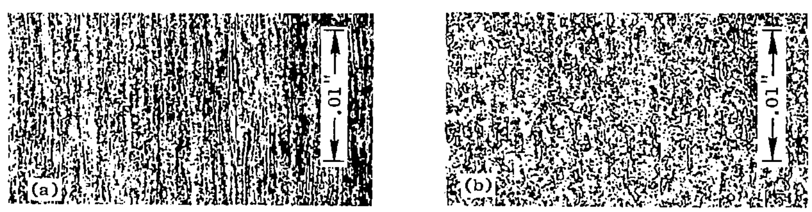
Etchant: 20% Murakamis



D580211 MB-1303G

Figure 53. Comparison of T-222 Alloy Microstructures in the (a) Pretest As-Worked Condition and (b) Posttest Partially Recrystallized Condition.

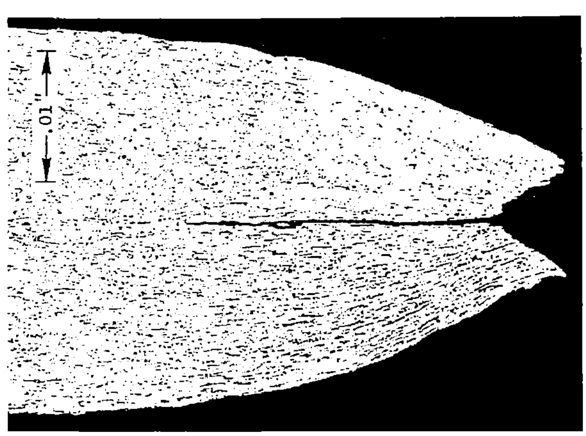
Etchant: $30gNH_4F-20mlHNO_3-20mlH_2O$



D570211 MB-1299A

Figure 54. Comparison of ASTAR 811C Microstructures in the (a) Pretest As-Worked Condition and (b) Posttest Recrystallized Condition.

Etchant: $30gNH_4F-20mlHNO_3-20mlH_2O$



MB-1299H

Figure 55. Microstructure of ASTAR 811C Alloy Tensile Specimen Exhibiting Longitudinal Cracking from Fracture Surface. Specimen Exposed to Flowing Lithium for 5000 Hours at $2100^{o}F$ Prior to Tensile Testing.

Etchant: $30gNH_4F-20mlHNO_3-20mlH_2O$

B. Valve and Loop Materials

1. Loop Materials

a) Visual Examination

Sections were removed from the Cb-1Zr portion of the loop at the heater inlet, heater outlet, test specimen housing inlet, and test specimen outlet for metallographic examination and chemical analysis. Visual examination of these sections revealed a very thin gold discoloration on the ID of the sections at the test specimen outlet and the heater inlet. Further examination revealed that the gold discoloration was present on the ID of all of the Cb-1Zr between these two points.

b) Chemical Analysis

Chemical analyses (C,O,H,N) were performed on specimens cut from selected critical areas of the various loop components. Areas selected were heater inlet, heater exit, tensile section inlet and tensile section outlet and the tubing connecting the two valves. All of these components are Cb-1Zr except the tubing between the valves which is T-111. The analyses of these components before and after the 5000 hour exposure to lithium are summarized in Table XIII. Large decreases in carbon concentration were noted at the heater exit and tensile section inlet. The heater inlet and tensile section outlet which operated at a lower temperature than the above mentioned components showed significant decreases in oxygen concentration but little or no change in nitrogen or carbon concentrations. The T-111 tubing between the valves also had a significant decreased oxygen concentration but, unlike the Cb-1Zr components, also had increased carbon and nitrogen concentrations even though the temperature was essentially the same as the Cb-1Zr heater inlet. The increased nitrogen concentrations in the T-111 tubing is in agreement with the observed nitrogen increases in the T-111 tensile specimens discussed previously under tensile test results.

In addition to the total wall analyses described above, gradient analyses were performed to determine the C,O,H,N concentrations across the tube wall of the Cb-1Zr tensile test section. The analyses were performed on the tensile test section tubing because it was sufficiently large to

TABLE XIII

RESULTS OF CHEMICAL ANALYSES OF LOOP COMPONENTS FROM
HIGH TEMPERATURE ALKALI METAL VALVE LOOP

Location in Loop	Material	Tube Size OD, inches	Temperature °F		Chemical Analysis, ppm C	N	O	H
Heater Inlet, total wall	Cb-1Zr	3/8	1905		46,54	77,61	105,78	< 1,< 1
				Avg	50	69	91	< 1
Heater Exit, total wall	Cb-1Zr	3/8	2100		26,47	2,2	10,10	1,1
				Avg	36	2	10	1
Tensile Section Inlet, total wall	Cb-1Zr	1	2090		46,59	7,4	10,13	2,2
				Avg	52	5	11	2
inner 0.040-inch of wall					54,59	3,3	16,7	5,4
				Avg	56	3	11	4
Tensile Section Exit, total wall	Cb-1Zr	1	1970		75,60	23,21	109,77	< 1,< 1
				Avg	67	22	93	< 1
inner 0.040-inch of wall					63,54	25,23	13,12	1,1
				Avg	58	24	12	1
outer 0.040-inch of wall					56,61	15,15	92,97	2,3
				Avg	58	15	96	2
Between Valves	T-111	1	1905		81,71	43,33	12,8	2,1
				Avg	76	38	10	1
Before Test	Cb-1Zr	3/8	-		60,70	29,65	159,110	5,3
				Avg	65	47	135	4
Before Test	Cb-1Zr	1	-		60,90	21,62	253,307	2,1
				Avg	75	41	280	1
Before Test	T-111	1	-		21,23	< 1,< 1	24,28	6,5
				Avg	22	< 1	26	5

also facilitate obtaining tensile and metallographic specimens from the same areas. The results of the gradient analyses are also presented in Table XIII. The carbon concentrations was found to be essentially constant across the tube wall and no significant difference was apparent between the inlet and exit end of the tube. The oxygen concentration on the lithium side (ID) of the inlet end was reduced considerably as a result of exposure to the lithium and, in fact, both the total wall and ID oxygen concentrations are the same indicating that the concentration is constant and that essentially all of the oxygen has been removed from the tube at this location by dissolution in the lithium. At the exit end of the tube, the ID oxygen concentration was found to be identical to the ID oxygen concentration at the inlet; however, the vacuum side (OD) and total wall analyses were significantly higher indicating a gradient of 12-95 ppm oxygen across the wall at the lithium exit temperature of 1972°F. The results of nitrogen gradient analyses show somewhat different trends than the oxgyen analyses. Table XIII shows that the ID and total wall nitrogen concentrations were similar at the inlet end, as was the case for oxygen. However, at the exit no removal of nitrogen appears to have occurred, even on the ID surface which was in direct contact with the flowing lithium.

The significance of these gradients in interstitial elements will be discussed in greater detail in connection with the metallographic and tensile properties later in this report.

c) Metallographic and Microprobe Evaluation

In addition to the metallographic evaluation of the valve components to be discussed in the next section of this report, evaluation was also performed on the various loop components such as the heater and tensile specimen housing. As was pointed out in the discussion on the visual examination, some of the Cb-1Zr components showed indications of discoloration or gold deposits on the lithium exposed surfaces. Verification of this deposit was obtained on the metallographic sections, in particular on the ID surface of the heater inlet as shown in Figure 56. To more fully evaluate the composition of the coating, microprobe and x-ray diffraction analyses were performed on specimens cut from the heater inlet

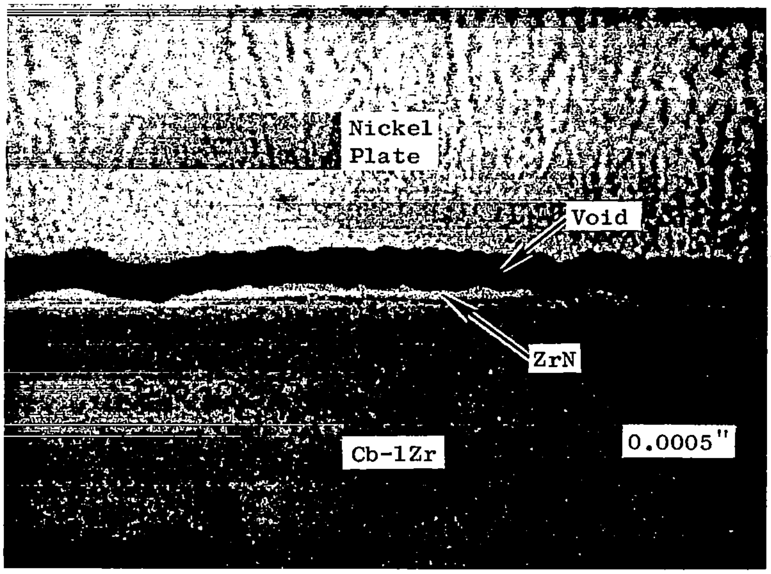


Figure 56. Transverse Section of Cb-1Zr Heater Inlet After Exposure to Flowing Lithium for 5000 Hours at 1905°F. As-Polished. (MB-1257C)

area. The x-ray diffraction results showed only the presence of ZrN. Microprobe traces across the coating indicated high zirconium content and depletion of columbium in the coating. These two techniques in addition to the gold color of the coating prove conclusively the composition to be ZrN and also substantiate the mass transfer of nitrogen in the loop as discussed previously under chemical analysis. Metallographic examination of the heater exit, tensile specimen section inlet and exit showed significant grain growth at all three locations as shown in Figures 57 and 58. Typical pretest microstructure of the tensile specimen housing material is shown in Figure 59. The heater exit, which operated at 2100°F, and tensile section inlet, which operated at 2090°F showed the greatest change with significant grain growth throughout the entire section of both pieces. The tensile section exit, which operated at the lowest temperature of the three areas, namely 1970°F, showed very large grains on the inner one-third of the wall, lesser grain growth on the middle-third and little or no grain growth on the outer (vacuum) wall. The grain size of the inlet area ID was much larger than the exit area ID as shown in Figure 58. At the inlet the maximum grain size observed was about ASTM 00 compared to a grain size of ASTM 4 at the exit. The pretest grain size (Figure 59) is about ASTM 8.

The results of the metallographic evaluation of the tensile specimen housing correlate extremely well with the chemical gradient analyses presented previously in Table XIII. These results show that the grain growth was a function of oxygen depletion and operating temperature; i.e., the maximum grain growth occurred at the highest temperature and at the lowest oxygen concentration (inlet). Although the oxygen on the lithium exposed surface of the inlet and outlet were identical, less grain growth was observed at the exit because of its 115°F lower operating temperature. In addition it appears that nitrogen does not effect grain growth under these conditions since the nitrogen level at the exit was essentially unchanged from its pretest value of 24 ppm, yet considerable grain growth was observed in this area (Figure 58).

Although the evaluation of welds or bimetallic joints was not a major objective of this program, there were four Cb-1Zr to T-111 welds in the loop. These welds were located where the Cb-1Zr pressure

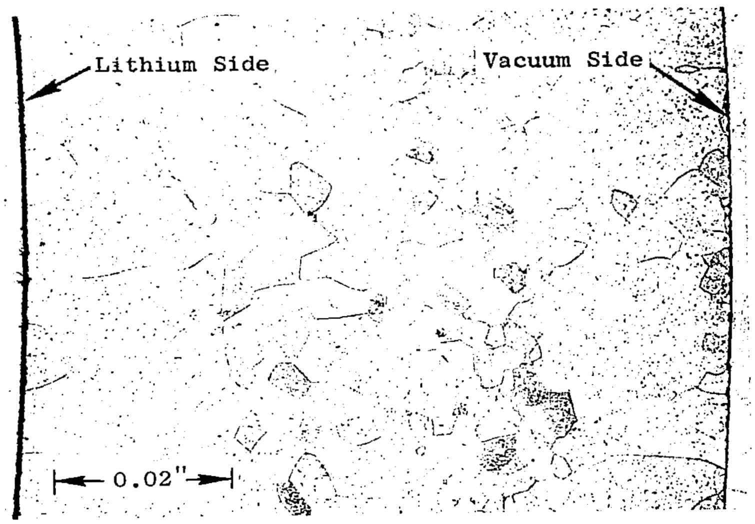
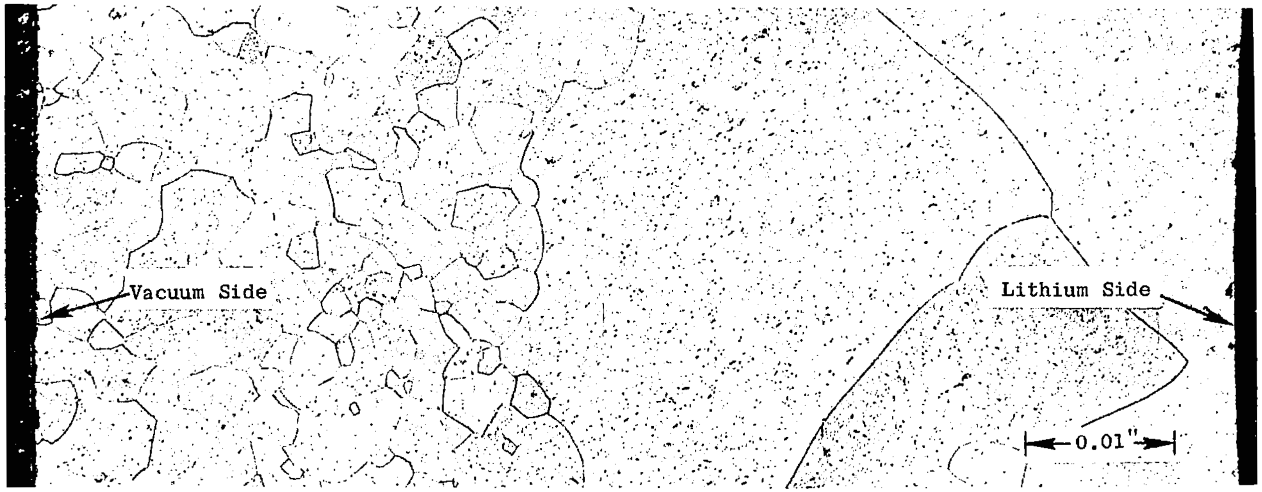


Figure 57. Transverse Section of Cb-1Zr Heater Exit After Exposure to Flowing Lithium for 5000 Hours at 2100°F. (MB-1249A)

Etchant: 60 Glycerine, $20HNO_3$, 20HF

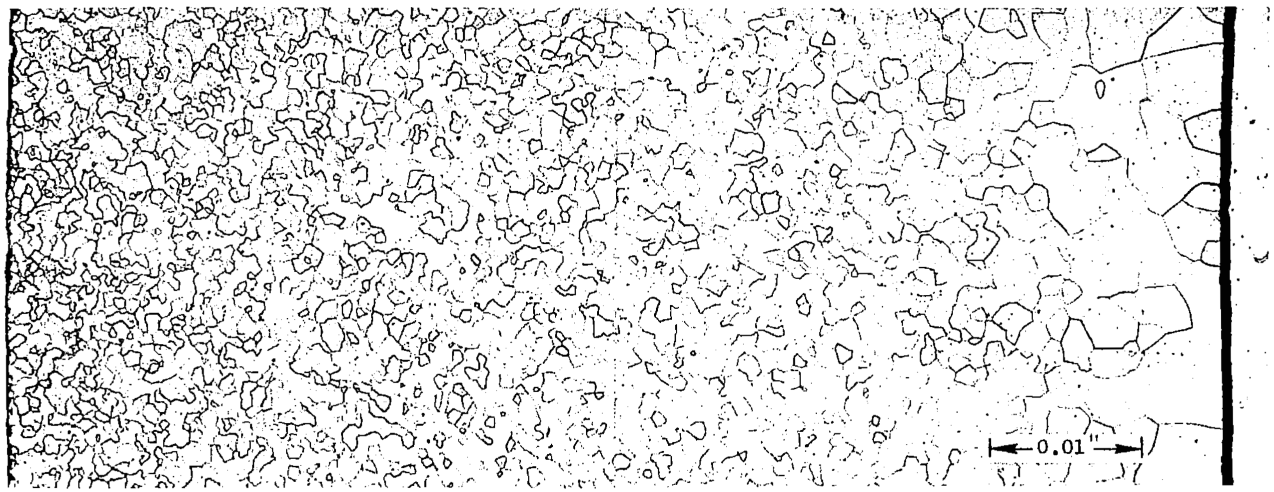


a) Lithium Inlet (2090°F)

MB-1249C&D

Vacuum (OD)

Lithium (ID)



b) Lithium Outlet (1970°F)

MB-1248C&D

Figure 58. Microstructure of Transverse Section from Cb-1Zr Tensile Specimen Housing After Exposure to Flowing Lithium for 5000 Hours.

Etchant: 60 Glycerine, $20HNO_3$, 20HF

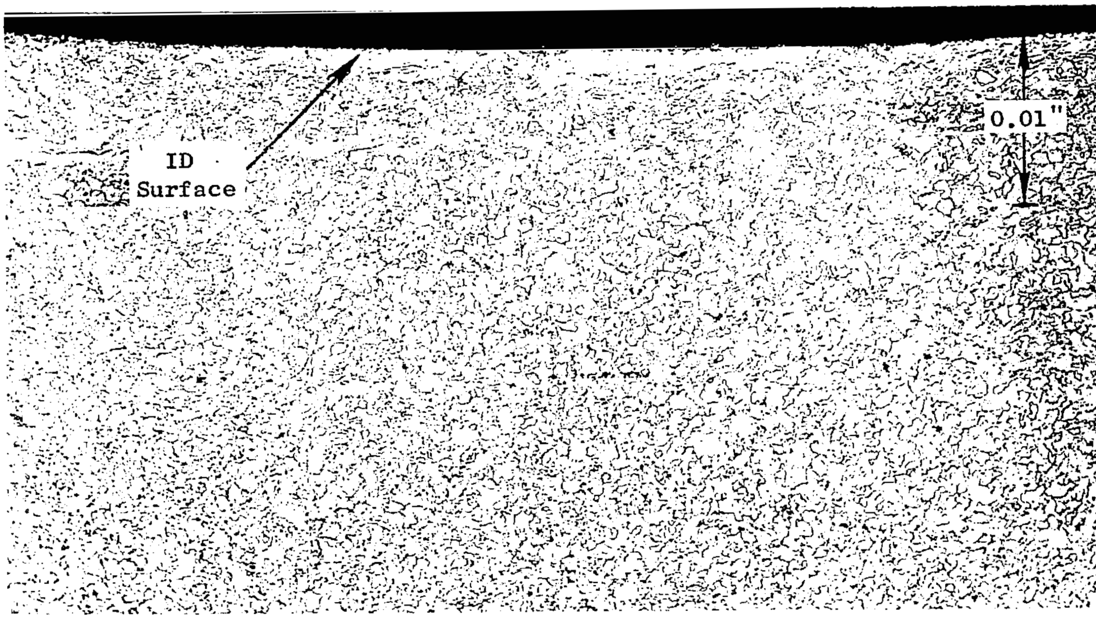


Figure 59. Pretest Microstructure of Cb-1Zr Tensile Specimen Housing Material. (K160)

transducers, EM pump, and heater were connected to the T-111 valve subassembly. The weld at the inlet to the metering valve was selected for evaluation as it operated at the highest temperature. The two tubes were jointed in a two pass butt type weld using Cb-1Zr filler wire. A composite photomicrograph of the weld after the 5000 hours of exposure to lithium is shown in Figure 60. The darkening of the T-111 near the surface is discussed later in this report. The composition of the base metal, heat affected zones, and fusion zone was determined by microprobe analysis at the points indicated on Figure 60 and are summarized in Table XIV. The most significant result was that the entire fusion zone appeared to be essentially homogeneous; and no gradient was observed in the weld. Traces from the ID surface to the OD surface for Ta, W, Hf, Cb, and Zr showed no gradient or inhomogeneity between the first and second pass. Some minor fluctuations were encountered occasionally during hafnium traces; however, these were extremely small and it was difficult to differentiate between normal equipment drift and concentration change. The results of the OD-ID trace are not included in Table XIV since they were essentially the same as the results of the longitudinal traces along the weld.

d) Tensile Tests

To further explore the extensive grain growth observed in the tensile specimen housing tube and the resultant effects on mechanical properties, this section was machined so that representative specimens were obtained of both the OD and ID surfaces. This was accomplished by removing either the OD or ID by turning on a lathe, slicing the tube longitudinally, and finally cutting standard tensile specimens. The results of room temperature tensile tests on ID and OD specimens are shown in Table XV which show increasing strength and ductility with decreasing grain size. Although the strength of the coarse grain material was only slightly lower than the fine grained material, the more significant factor is the observed difference in ductility. The ductility of the coarse grain material was about one-half that of the fine grained material.

2. Valve Materials

a) Visual Examination

Examination of the disassembled valves showed that all parts were

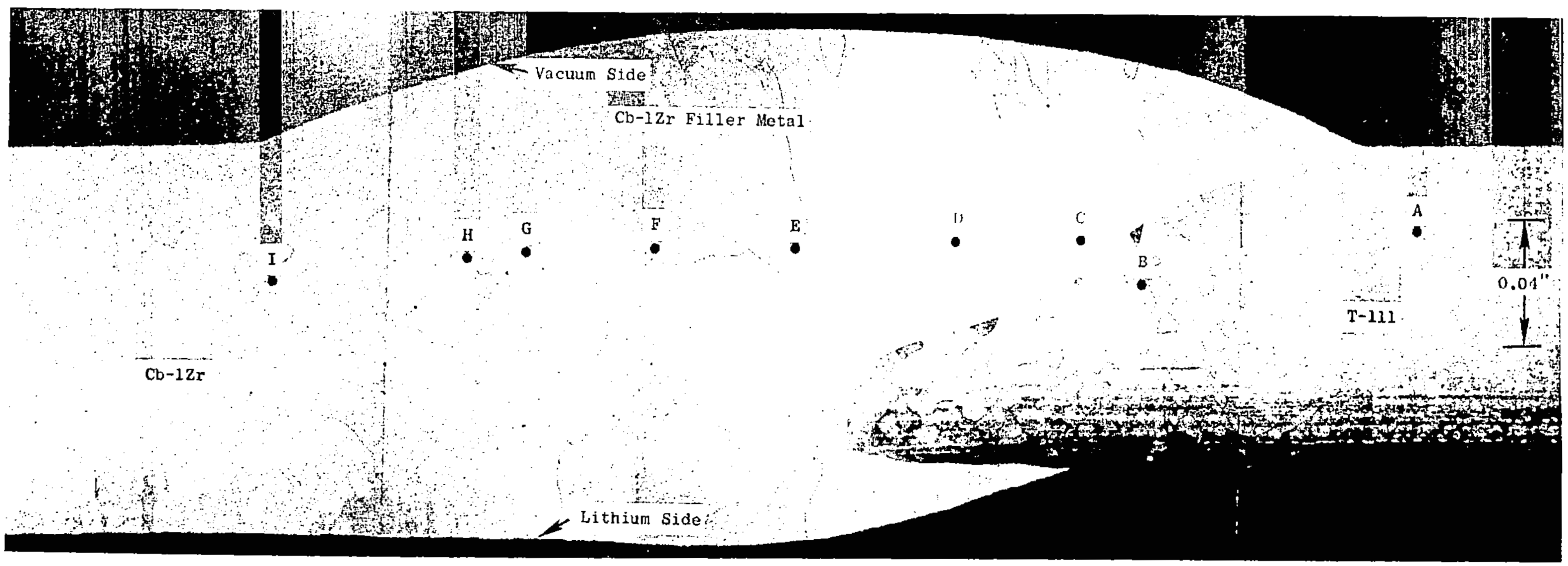


Figure 60. Composite Photomicrograph of Cb-1Zr to T-111 Weld After 5000 Hours Exposure to Flowing Lithium at 1905°F. (G39011)

Etchant: 60 Glycerine, 20HNO_3, 20HF

TABLE XIV

RESULTS OF MICROPROBE ANALYSES OF Cb-1Zr TO T-111 BIMETALLIC WELD

	Composition, weight percent				
Position*	Ta	Cb	Hf	W	Zr
A	89	< 1	2	9	< 1
B	89	< 1	2	9	< 1
C	22	71	< 1	2	2
D	19	72	< 1	2	2
E	19	71	< 1	3	2
F	20	71	< 1	2	2
G	20	79	< 1	3	2
H	22	72	< 1	2	2
I	< 1	100	< 1	< 1	1

*Refer to Figure 60

NOTE: Because of the limitations of the microprobe apparatus the results are not true quantitative values; hence they do not necessarily total 100%. The results are intended primarily to indicate relative concentration in each location.

TABLE XV

RESULTS OF ROOM TEMPERATURE TENSILE TESTS ON Cb-1Zr SPECIMEN HOUSING CONTAINING FLOWING LITHIUM

Specimen Location	Exposure Temp. °F	U.T.S. (ksi)	0.2% Yield (ksi)	Elong. %	Chemical Analysis,* ppm				Grain Size (ASTM No.)
					Posttest				
					C	O	N	H	
Inlet-ID	2090	35.6	23.7	22.5	57	12	3	4	00
		37.6	25.6	15.0					
Exit-ID	1970	39.8	26.1	27.0	59	12	24	1	4
		40.0	24.2	28.0					
Exit-OD	1970	42.0	27.9	34.0	59	95	15	3	8
		43.3	25.8	32.5					
					Pretest				
					75	280	41	2	8

*Average of duplicate analyses

in excellent condition. No evidence of corrosion, wear or abrasion was observed. The plugs and seats were in particularly good condition, as seen in the photograph of the metering valve plug in Figure 61. A gold deposit was observed as indicated in Figure 62. No identification of the deposit was possible because it was too thin but it was thought to be ZrN.

b) Weight Measurements

Weights were obtained on the W-25Re plugs before and after the test exposure and the results are given in Table XVI. Posttest weights of the unalloyed rhenium valve seats could not be obtained because the T-111 alloy seat retainer was securely bonded to the seat.

c) Chemical Analysis

Chemical analyses for C,O,H,N were performed on the plug, seat, and the bellows from both the metering and isolation valves. Analysis was also performed on the T-111 tubing connecting the two valves. The results, shown in Table XVII show that in most cases the concentration of interstitial elements increased. This is not unexpected since the valves were operating at the lowest temperatures in the loop; hence, a natural location for transfer of interstitials from the lithium to the other materials.

d) Metallographic and Microprobe Evaluation

No evidence of any corrosion was observed in the plug or seat from either valve. Typical photomicrographs are shown in Figures 63 and 64. The only notable change of any kind in the plug or seats was the presence of second phase particles in the rhenium seats of both valves; these are presumably oxide particles resulting from the increased oxygen concentration as shown in Table XVII.

The bellows of both valves showed no signs of deterioration or corrosion from the 5000 hour lithium exposure as shown in Figure 65. Comparison of the pretest microstructure and fabrication history of the bellows discussed earlier in this report indicates that the surface layer seen on the high magnification photomicrographs of Figure 65 are thin recrystallized layers resulting from cold work introduced during

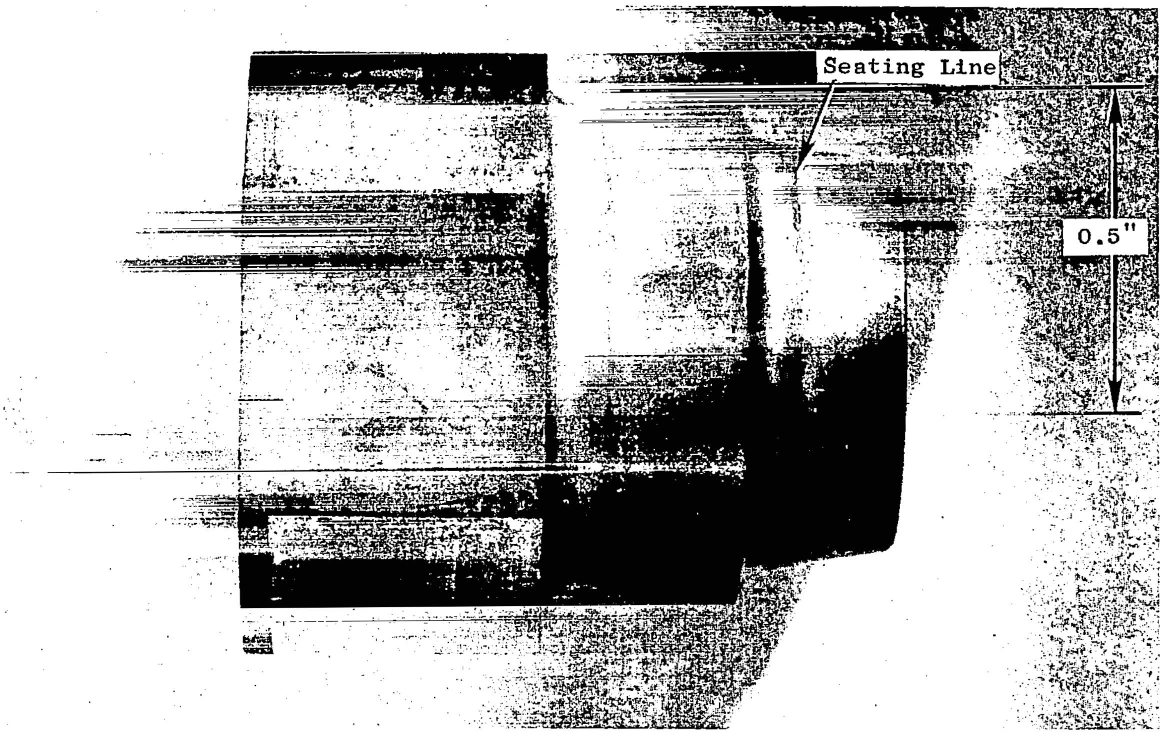


Figure 61. W-25Re Metering Valve Plug After 5000 Hours Exposure to Flowing Lithium at 1905°F. (P69-8-22D)

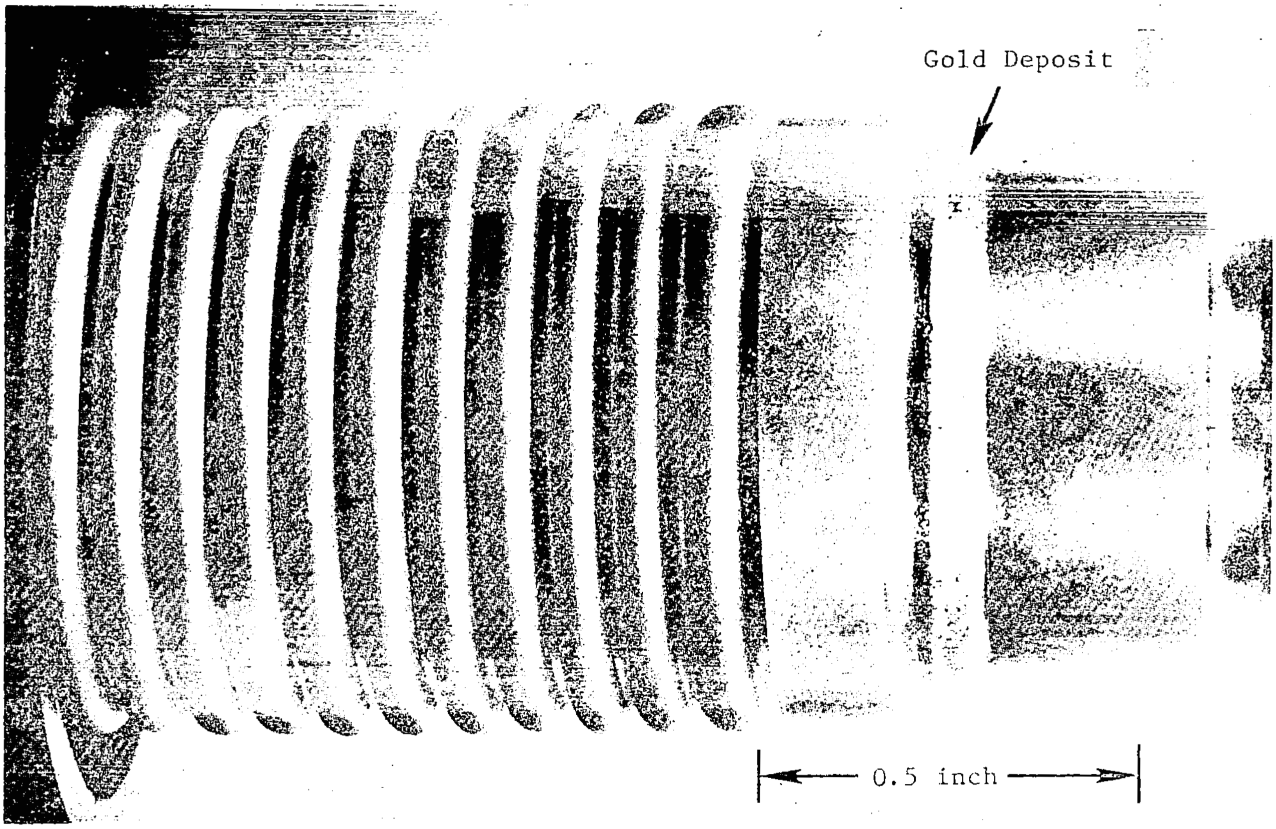


Figure 62. T-111 Bellows, Back-Up Ring and Stem Base After 5000 Hours Exposure to Lithium at 1905°F. (P69-8-22B)

TABLE XVI

WEIGHT MEASUREMENTS ON W-25Re VALVE PLUGS
BEFORE AND AFTER 5000 HOURS OF EXPOSURE TO LITHIUM

	Weight Before Test, gm	Weight After Test, gm	Change gm
Metering Valve Plug	111.8488	111.8494	+0.0006
Isolation Valve Plug	108.5907	108.5960	+0.0053

TABLE XVII

RESULTS OF CHEMICAL ANALYSES OF VALVE COMPONENTS
FROM HIGH TEMPERATURE ALKALI METAL VALVE LOOP

Component	Material	Temp.°F		Pretest Analyses, ppm				Posttest Analyses, ppm			
				C	N	O	H	C	N	O	H
Isolation Valve Seat	Re	1905		13,18	8,11	1,1	1,1	62,52	2,< 1	24,25	< 1,< 1
			Avg	15	9	1	1	57	1	24	< 1
Isolation Valve Plug	W-25Re	1905		8,5	< 1,< 1	7,7	< 1,< 1	41,34	< 1,< 1	14,12	< 1,< 1
			Avg	6	< 1	7	< 1	37	< 1	13	< 1
Isolation Valve Bellows	T-111	1700		42,43	17,7	109,79	12,9	15,18	27,14	87,100	1,1
			Avg	42	12	94	10	16	20	93	1
Metering Valve Seat	Re	1905		13,18	8,11	1,1	1,1	39,26	< 1,< 1	10,13	< 1,< 1
			Avg	15	9	1	1	32	< 1	11	< 1
Metering Valve Plug	W-25Re	1905		8,5	< 1,< 1	7,7	< 1,< 1	86,118	< 1,< 1	12,9	< 1,< 1
			Avg	6	< 1	7	< 1	102	< 1	10	< 1
Metering Valve Bellows	T-111	1700		42,43	17,7	109,79	12,9	83,85	39,64	106,80	3,4
			Avg	42	12	94	10	84	51	93	3
Tubing Between Valves	T-111	1905		21,23	< 1,< 1	24,28	6,5	81,71	43,33	12,8	2,1
			Avg	22	< 1	26	5	76	38	10	1

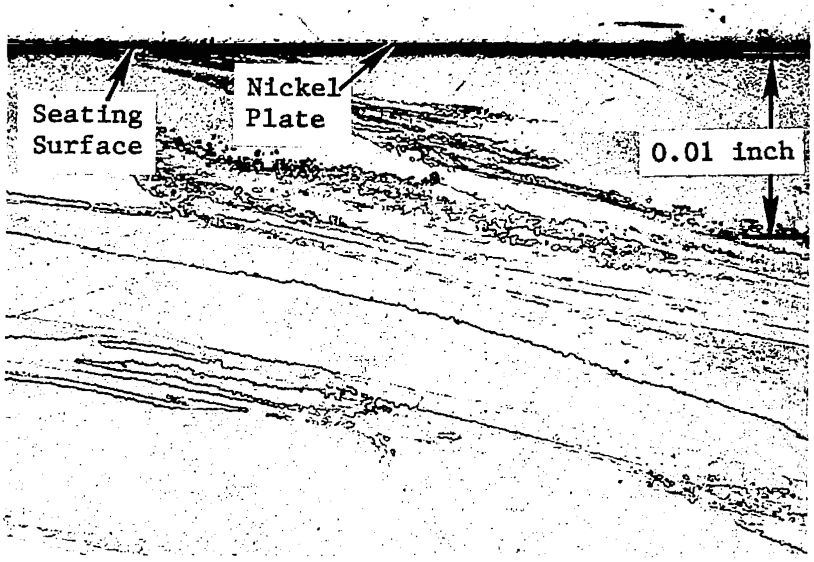


Figure 63. Microstructure of W-25Re Metering Valve Plug After Exposure to Flowing Lithium for 5000 Hours at 1905°F. (MB-1251A)

Etchant: 20% Murakamis

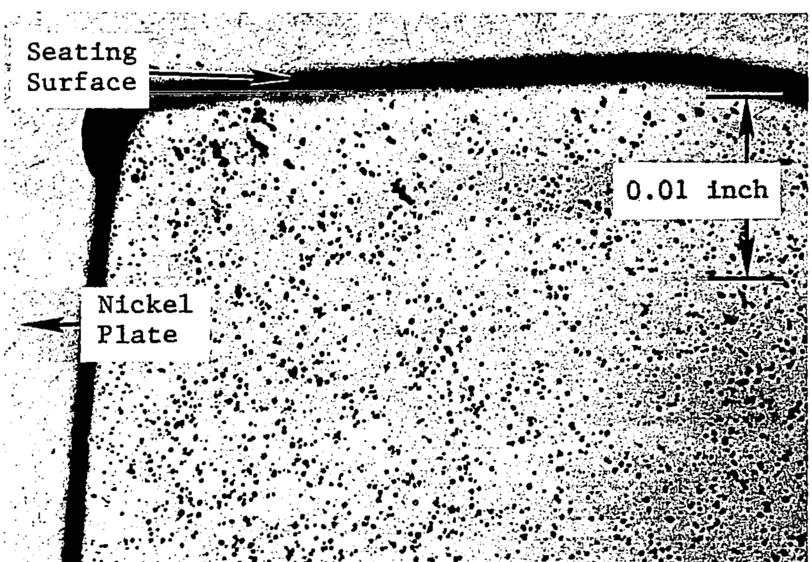
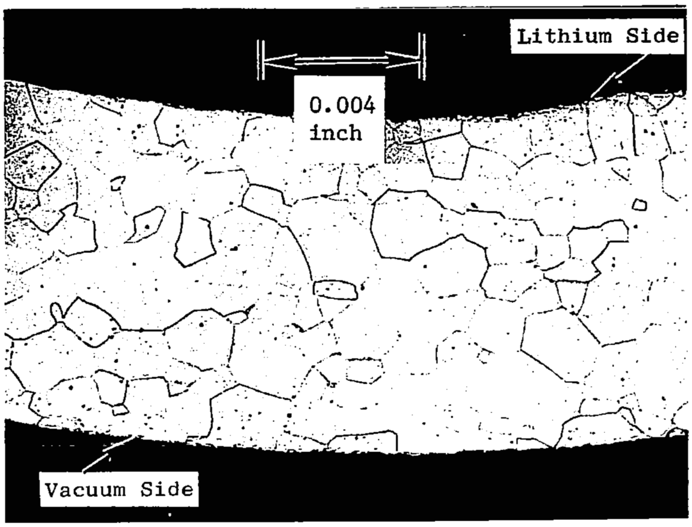
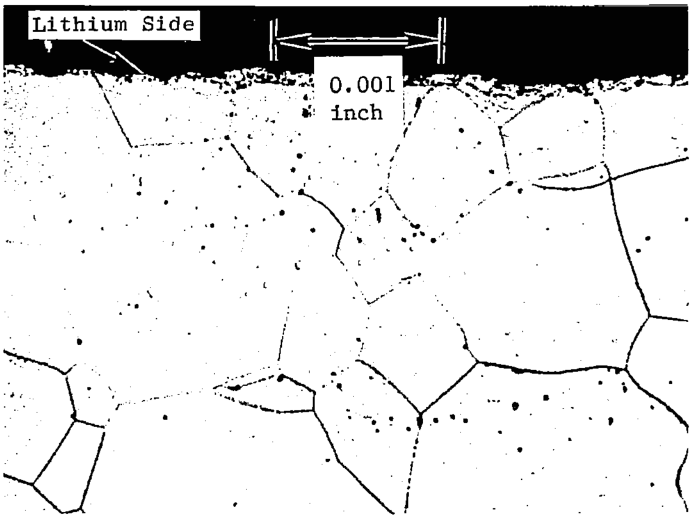


Figure 64. Microstructure of Rhenium Isolation Valve Seat After Exposure to Flowing Lithium for 5000 Hours at 1905°F. (MB-1246A)

Unetched



MB-1259A



MD-1259B

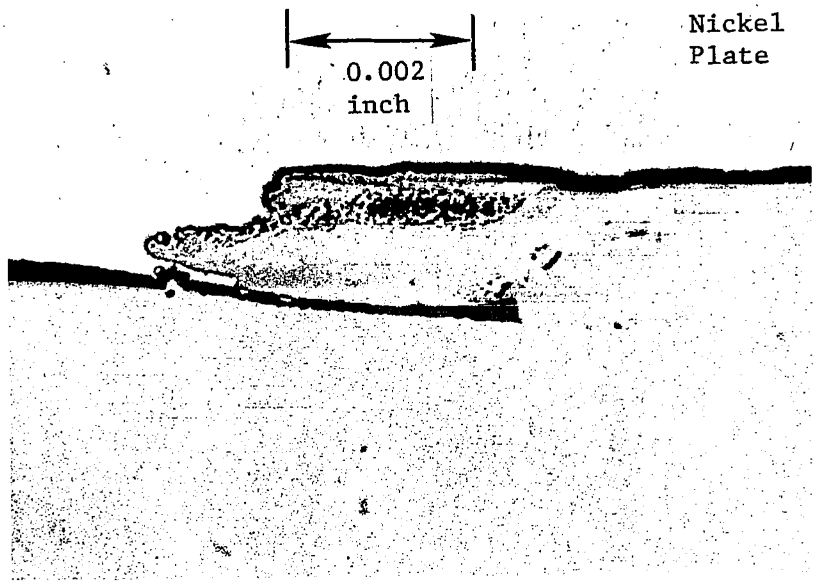
Figure 65. T-111 Metering Valve Bellows After Exposure to Lithium at $1700^{o}F$ for 5000 Hours.
Etchant: $NH_4F-20HNO_3-20H_2O$

the polishing operation prior to forming the bellows, and the subsequent thermal treatments. It should be pointed out that the bellows were exposed to relatively static lithium; hence, any effects compared to exposure in flowing lithium should be less.

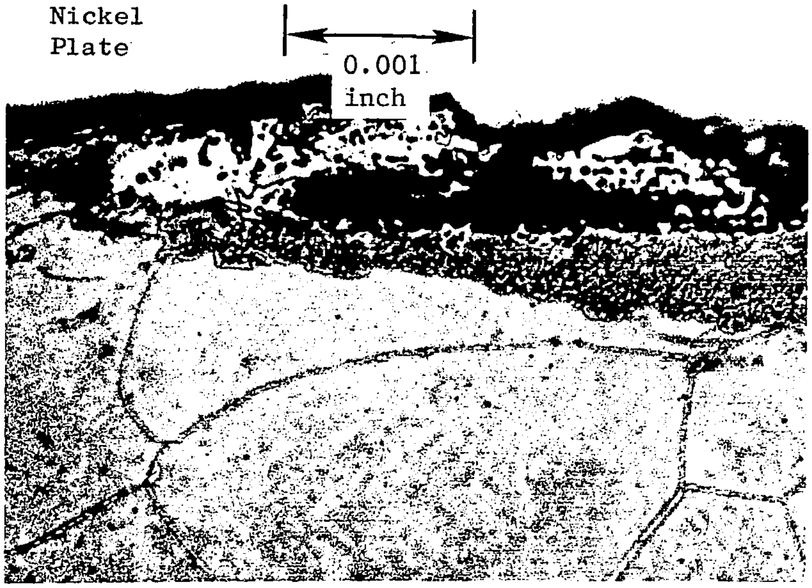
As was pointed out earlier, the major observable changes in microstructure were in the T-111 tubing exposed to flowing lithium in the region of the valve. Two types of noticeable changes in the T-111 microstructure were observed as shown in Figures 66 and 67.

A "spongy" area was observed on the surface, as shown in Figure 66, and was generally associated with what appeared to be a lap on the surface indicating that the change could also be associated with contamination from entrapped air. Microprobe analysis of these types of areas showed them to be enriched in tungsten (approximately twice that of the normal content) and depleted in tantalum and hafnium.

The type of microstructural change illustrated in Figure 67 is more difficult to explain. Microprobe analysis of the light and dark areas failed to produce any significant differences in metallic constituents. It is possible that the observed microstructural changes are the result of gradients in interstitial concentrations below the sensitivity of the analysis technique (1000 ppm). Electron beam microprobe analysis of the white grain boundary and surface phase shown in Figure 67 also failed to produce any conclusive results since the size of the second phase was less than the electron beam diameter and therefore the necessary resolution needed to identify the phase could not be achieved.



As-Polished MB1256E



Etchant: $30gNH_4F-20HNO_3-20H_2O$ MB1256F

Figure 66. Microstructures of T-111 Tubing at the Exit of Isolation Valve After Exposure to Flowing Lithium for 5000 Hours at 1905°F.

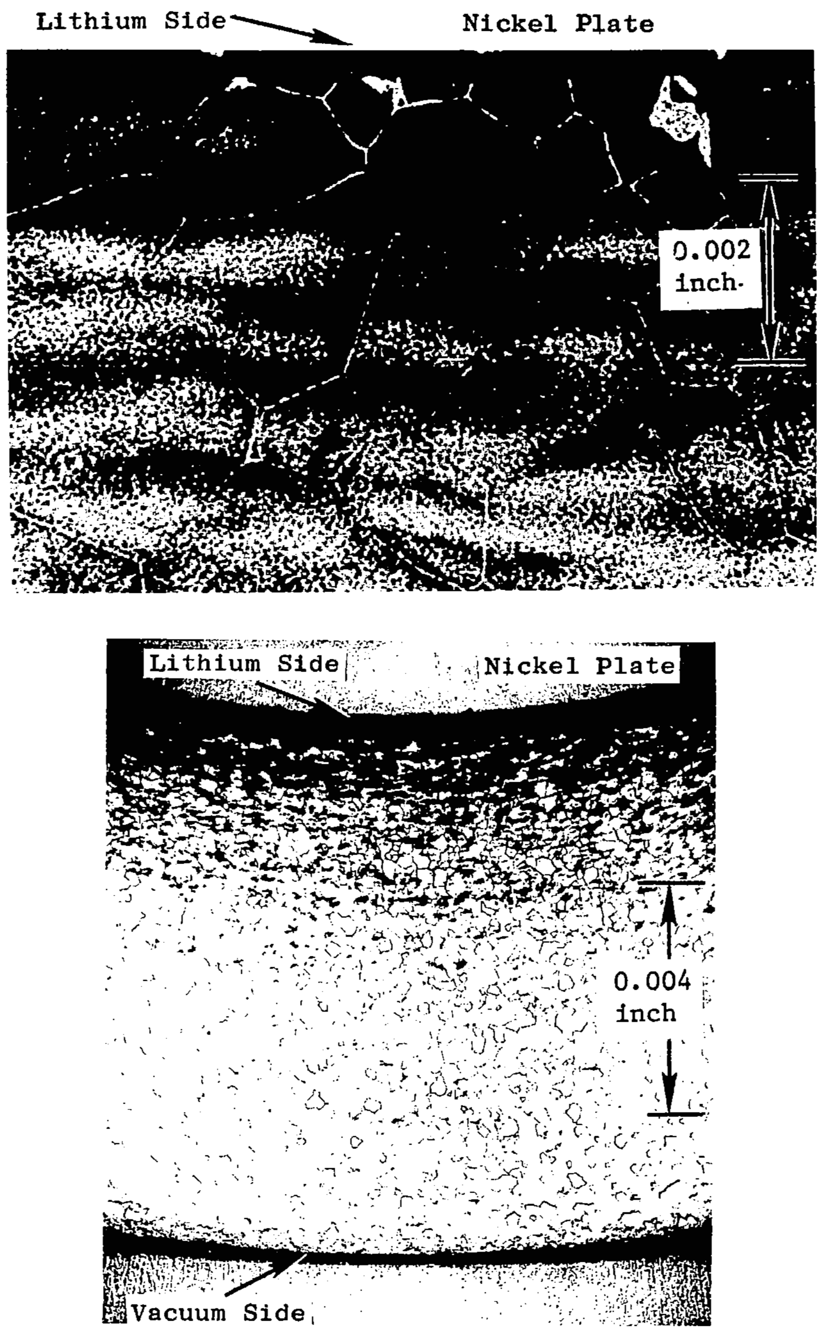


Figure 67. Microstructure of T-111 Tubing Between the Valve After Exposure to Flowing Lithium for 5000 Hours at 1905°F. (MB-1250B&D)

Etchant: $30gNH_4F-50mlHNO_3-50mlH_2O$

VIII. SUMMARY AND CONCLUSIONS

Two refractory metal alloy valves were successfully designed, fabricated, and operated in flowing lithium at temperatures up to 1900°F for 5000 hours. The isolation valve was cycled for 95 cycles between the full open and full closed position and the torque to open and close the valve on each cycle measured. The metering valve was cycled between 1/4 and 3/4 travel during this same period.

The W-25Re plugs and rhenium seats in each valve exhibited excellent performance with no sign of wear, abrasion, bonding or lithium attack observed in either valve. The T-111 bellows and other refractory and non-refractory metal components all performed without serious degradation. The only problem was galling of Mo-TZM gears; these were replaced at the planned 2500 hour shutdown with VASCO Hypercut gears which performed without problems during the second 2500 hours of operation.

APPENDIX A

LITHIUM COMPATIBILITY TESTS OF VALVE SEAT MATERIALS

1. TiC-10Cb

An isothermal capsule test was conducted to determine the compatibility of the valve seat material, TiC-10Cb, with lithium. The test was conducted at 2100°F for 60 hours using a Cb-1Zr container. Metallographic examination of the specimen following exposure to lithium revealed attack which varied in depth from a minimum of 20 mils to a maximum of 70 mils in the form of subsurface voids. The appearance of the valve seat and cross-sections of the seat before and after exposure to lithium is illustrated in Figure 68. The extent of lithium penetration is clearly delineated in the cross-section of the tested seat. Microhardness tests using a 500 g load indicated an average hardness of 1360 DPH in the unattacked regions and 850 DPH in the attacked areas. The appearance of as-polished cross-sections of the TiC-10Cb specimens before and after exposure is illustrated in Figure 69. The corroded area illustrated is representative of the regions which showed the most extensive lithium penetration.

Although the exact mechanism of the lithium attack of the TiC-10Cb has not been defined, it is very probable that the presence of oxygen in the material is responsible for the poor corrosion resistance. The effect of the oxygen concentration of refractory metals, such as columbium and tantalum, or their corrosion resistance to lithium, has been demonstrated by numerous studies.[9,10] The oxygen concentration of several of the ten purchased TiC-10Cb valve seats before and after exposure to lithium and potassium are listed in Table XVIII. It may be noted that despite the considerable scatter in oxygen concentrations of the various valve seats, all the analyses indicated quite high oxygen levels.

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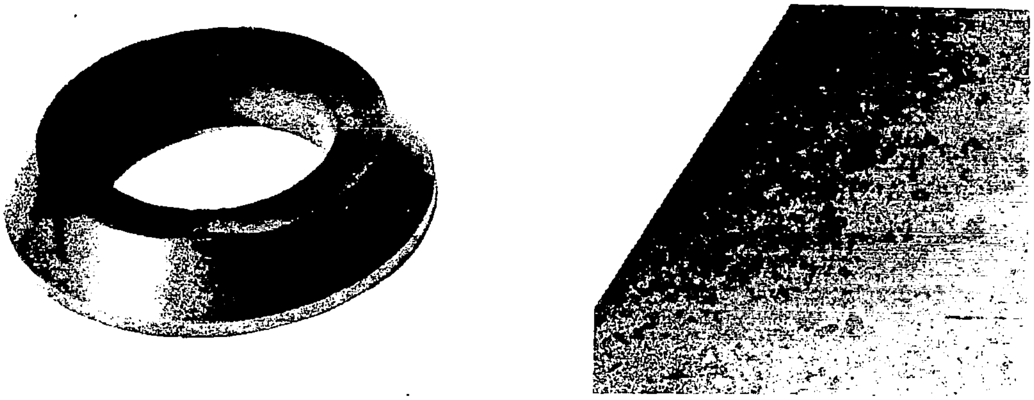
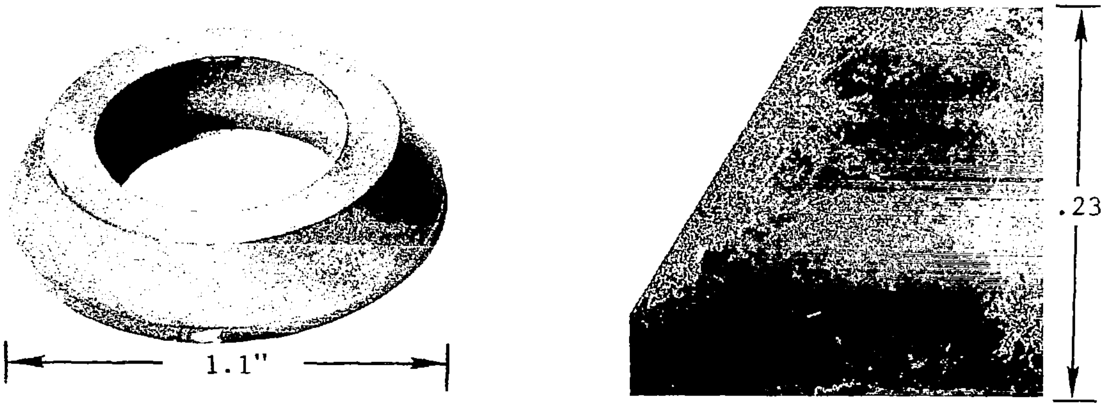
[9] DiStefano, J. R. and Hoffman, E. E., "Corrosion Mechanisms in Metal-Alkali Metal Systems," The Science and Technology of Tungsten, Tantalum, Molybdenum, Niobium and Their Alloys, ed. by N. E. Promisel (Pergamon Press, London, 1964), pp. 257-288.

[10] DiStefano, J. R.,"Corrosion of Refractory Metals by Lithium," ORNL-3551, March 1964.

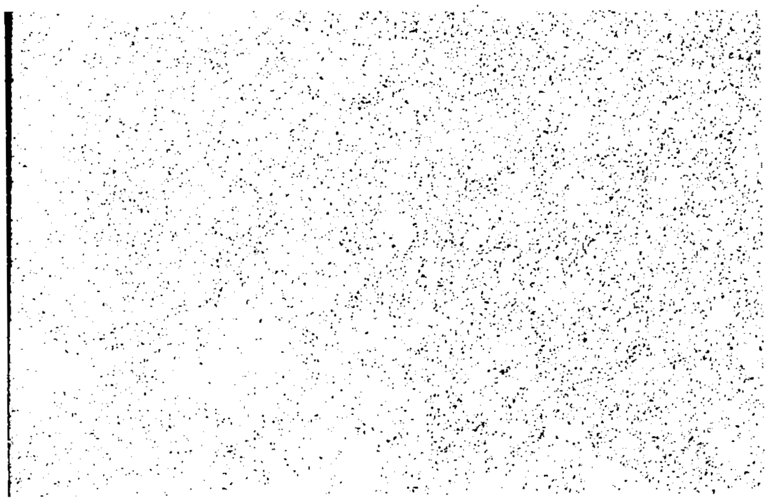


a) Before Exposure to Lithium

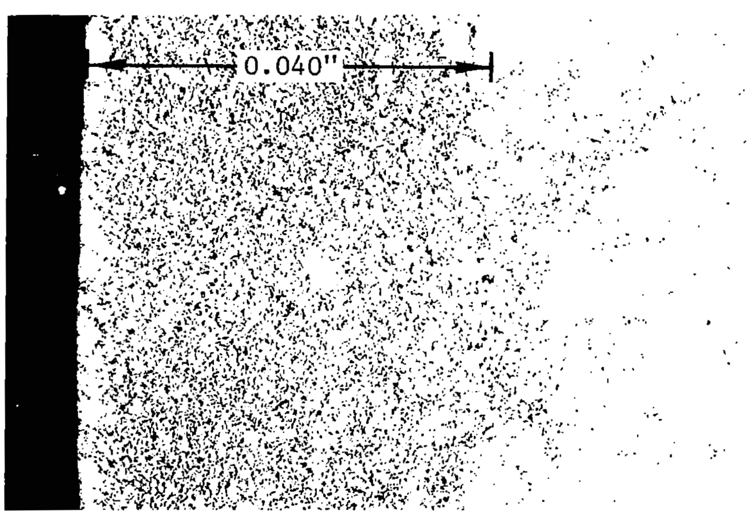


b) After Exposure to Lithium

Figure 68. TiC+10%Cb Valve Seats Before and Following Exposure to Lithium for 60 Hours at 2100°F. The Depth of Lithium Penetration is Apparent in the As-Polished Transverse Section of the Tested Valve Seat. (C67081422) (C67080434) (C67080435)



a) Before Exposure to Lithium



b) After Exposure to Lithium

Figure 69. TiC+10%Cb Valve Seat Specimens Before and Following Exposure to Lithium for 60 Hours at 2100°F.

As-Polished, 50X

a) (E390124)
b) (E270111)

TABLE XVIII

CHEMICAL ANALYSES OF TiC-10Cb VALVE SEATS

Sample Designation	Concentration, ppm O	N	H
Valve Seat No. 3 (as-received)	970[a]	68	32
	1200[a]		
	1400[a]		
	1330	68	32
Valve Seat No. 2 (as-received)	8330	697	164
	4337	352	35
Valve Seat No. 1 (exposed to Li at 2100°F for 60 hours)	7823	512	47
	6229	450	94
Valve Seat No. 2 (exposed to K at 2100°F for 60 hours)	3705	115	≤ 1

[a] Analyses performed by National Spectrographic Lab; all other analyses performed by GE-NSP.

As indicated in Table XVIII, an isothermal capsule test was also conducted on one of the valve seats in potassium. This test was performed in order to obtain a comparison of the corrosion resistance of this lot of TiC-10Cb with an earlier test of a different lot of this material. Both the earlier experiment, which was conducted at 1600°F for 1000 hours,(11) and the current experiment, which was conducted at 2100°F for 60 hours, showed that this material is unattacked by potassium.

Potential sources for TiC-10Cb were contacted to examine the possibility of obtaining material with a very low oxygen content that might be resistant to lithium attack. It appeared, however, that this approach would not assure success because of the technical difficulty of preparing material with an oxygen content below approximately 200 ppm and because lithium tests of TiC at JPL revealed substantial attack(12) despite the fact that the oxygen content of the material was only 350 ppm.* In other words, the value of the permissible oxygen concentration to avoid lithium attack has not been determined and it may be lower than the value that can be assured by current processing techniques.

As a result, rhenium was selected as an alternate valve seat material for the High Temperature Alkali Metal Valve Test Program.

2. Rhenium

Three finished machined valve seats and a $\frac{1}{2} \times 1\frac{1}{4} \times \frac{1}{4}$ inch thick specimen from the same powder metallurgy plate that was used to make the valve seats was obtained from the Cleveland Refractory Division of the Chase Brass and Copper Company for pretest chemistry, hardness

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(11) Materials for Potassium Lubricated Journal Bearings, Quarterly Progress Report No. 9, Quarter Ending July 22, 1965, NASA Contract NAS 3-2534, NASA-CR-54892, pp. 15.

(12) "Space Programs Summary 37-41, Volume IV, Supporting Research and Advanced Development" for the Period August 1 to September 30, 1966, Jet Propulsion Laboratory, October 31, 1966, p. 127.

*A sample of the TiC material was obtained from JPL, and analyses performed by GE-NSP indicated that the material contained approximately 350 ppm oxygen.

measurements, and compatibility tests. A chemical analysis of the as received material is given in Table XIX.

An isothermal capsule test was conducted to determine the compatibility of unalloyed rhenium with lithium. A $\frac{1}{2}$ x $\frac{1}{2}$ x $\frac{1}{4}$-inch thick specimen was run at 2100°F for 100 hours in a 1.00-inch OD x 0.100-inch wall x 6-inch long Cb-1Zr capsule with approximately 15 cc of lithium in a high temperature furnace at a vacuum of 6 x 10^{-6} torr. After test, the capsule was opened in an argon atmosphere and the excess lithium was drained from the capsule and test specimen. The residual lithium was removed from the specimen and test capsule with liquid ammonia. Metallographic examination of the specimen following its exposure to lithium showed no significant change from the pretest condition as shown in Figure 70. Some porosity is evident in this powder metallurgy product which has a theoretical density of 98+ percent. Microhardness measurements using a 500 g load showed no significant changes after exposure to lithium as shown in Table XX. The variations in hardness of the various regions are not considered to be significant and all values obtained (approximately DPH 240) are slightly lower than the value of approximately 300 reported by Sims[(13)] for recrystallized rhenium. The results of the chemical analysis before and following exposure to lithium are given in Table XXI. A slight apparent increase in the carbon concentration of the rhenium specimen was noted. Although there was a small residual concentration of carbon present (approximately 45 ppm) in the lithium, it is difficult to understand how rhenium, which does not form a stable carbide phase,[(14)] could pick up carbon in the presence of the Cb-1Zr container, since both columbium and zirconium form stable carbides.

[(13)] Sims, C. T., "Properties of Rhenium, Rhenium, Edited by B. W. Gonser, Elservier Publishing Company, 1962, p.33.

[(14)] Ibid, p. 27.

TABLE XIX

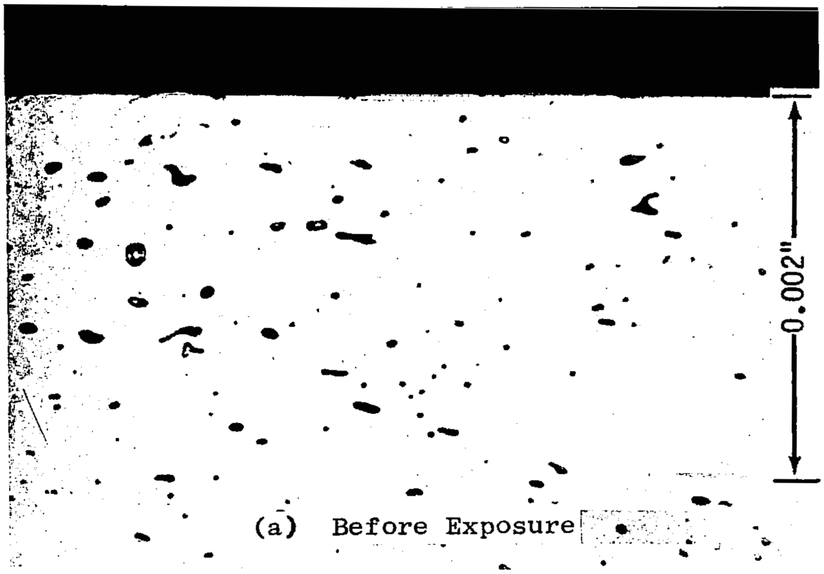
CHEMICAL ANALYSIS[a] OF AS RECEIVED RHENIUM PLATE[b] FROM WHICH THE VALVE SEATS FOR THE HIGH TEMPERATURE ALKALI METAL VALVE WERE MACHINED

Element	Chemical Analysis, ppm
O	8,11
N	1, 1
H	1, 1
C	13,18
Fe	180
Cr	10
Cu	9
B,Mo,Na,K	< 20
Si,Al,Ca,Mg,Mn,Ni,Sn,Ti,Zr,Co	< 10

(a) All analyses performed by National Spectrographic Laboratory, Cleveland, Ohio except O, N, H, and C analyses which were performed by GE-NSP.

Analytical methods: O, N, and H; Vacuum Fusion
C; Combustion Conductometric

(b) Vendor—Cleveland Refractory Metals Division, Chase Brass and Copper Company.



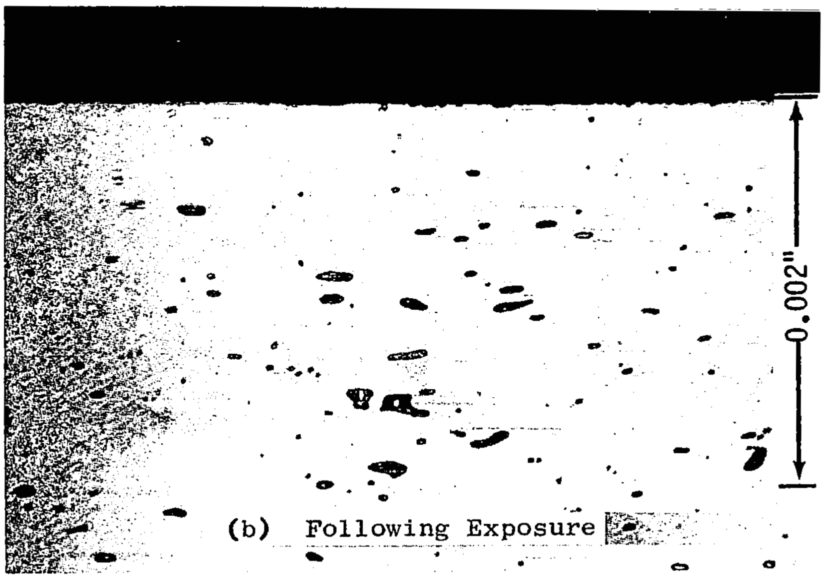


Figure 70. Microstructure of Rhenium Specimens Before and Following Exposure to Lithium for 100 Hours at 2100°F.

As-Polished

a) 1000X (F170111)
b) 1000X (F170121)

TABLE XX

MICROHARDNESS OF RHENIUM SPECIMEN BEFORE AND FOLLOWING EXPOSURE TO LITHIUM FOR 100 HOURS AT 2100°F[(a)]

Distance From Surface of Specimen	Diamond Pyramid Hardness (500 g Load)	
	As Received	After Lithium Exposure
0.005-inch	235	266
0.010-inch	245	245
0.015-inch	240	235
0.020-inch	225	242
0.125-inch (center)	250	213

(a) Isothermal test of the Cb-1Zr foil wrapped Cb-1Zr capsule conducted in the R.D. Brew Co. Model 424B Vacuum Furnace at a pressure of 6×10^{-6} torr.

TABLE XXI

RESULTS OF CHEMICAL ANALYSIS OF RHENIUM SPECIMEN
BEFORE AND FOLLOWING EXPOSURE TO LITHIUM FOR 100 HOURS AT 2100°F

	Chemical Analysis, ppm[a]			
	O	N	H	C
Before Exposure	8	1	1	13
	11	1	1	18
Following Exposure	12	1	1	33
	13	1	2	58

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

APPENDIX B

DRAWING AND PARTS LIST FOR HIGH TEMPERATURE ALKALI METAL VALVE

Item	Hoke* Part No.	Title	Material	NSP Drawing No.
1.	furn by NSP	Gear, pinion	VASCO Hypercut	119C2625P1
2.	80429-1	Ball Bearing		47B116049G2
3.	92241-030	Ring, Inner Brg	VASCO Hypercut	47B116044P2
4.	92240-030	Ring, Outer Brg	VASCO Hypercut	47C144012P2
5.	5130	Ball - 1/8 dia.	WC-6Co	47B116049P7
6.	474-149	Mount, Gear	VASCO Hypercut	47C144032
7.	53-323	SCR, Set (Hex Soc Cup ft) 6-32 x 3/16 lg	SST303	
8.	474-150	Collar	VASCO Hypercut	
9.	474-132	SCR,CAP,HEX HD 5/16-18 x 2 lg	Cb-1Zr	
10.	474-133	Plate, Clamp	Cb-1Zr	
11.	474-148	Ring, Back-seat	Cb-1Zr	
12.	474-122	Assembly Bellows		
13.	474-124	Ring, Bellows	T-111	
14.	474-125	Ring, Back-up	T-111	
15.	474-126	Bellows	T-111	
16.	474-127	Base, stem	T-111	
17.	474-128	Ring, Back-up	T-111	
18.	58-144	Caplug (No. 13-X)	Polyethylene	
19.	474-135	Nipple - 1" OD x 100 W.T.	T-111	
20.	474-131	Body, Outlet	T-111	
21.	474-121	Body, Inlet, Globe	T-111	
22.	474-137	Retainer, seat	T-111	
23.	474-138	Seat - inlet	Re	
24.	474-139	Plug, by-pass	W-25Re	
24.	474-140	Plug	W-25Re	
25.	474-141	Pin, Plug	W-25Re	
26.	474-142	Stem, Bellows	T-111	
27.	474-143	Nut, hex 5/16-18	T-111	
28.	474-144	Washer	Cb-1Zr	
29.	474-134	Plate, body	Cb-1Zr	
30.	474-145	Ring, split	Cb-1Zr	
31.	474-136	Bonnet, lower	Cb-1Zr	47C144033
32.	92239-030	Valve stem extension	T-111	47B116066
33.	53-379	SCR SET (Hex soc flat pt) No. 4-40 x 1/8 lg	SST303	AN565AC4H2
34.	92232-030	Pin	VASCO Hypercut	47B116055P2
35.	92237-030	Bushing, brg support	VASCO Hypercut	47B116054
36.	5130	Ball - 1/8 dia.	Wc-6Co	47D174816P2
37.	92236-030	Bearing race, lower	VASCO Hypercut	47B116053P2
38.	53-378	SCR Set (hex soc flat pt) No. 8-32 x 7/16 lg	SST303	AN565AC8H7
39.	5378	Nut, hex 8-32	SST18-8	MS35649-84
40.	92235-030	Bearing Housing	VASCO Hypercut	47C144025
41.	92234-030	Bearing race, upper	VASCO Hypercut	47B116052
42.	92238-030	Pin	VASCO Hypercut	47B116055P3
43.	474-152	SCR, Set (hex soc conept) 3/8-16 x 7/16 lg	SST303	
44.	92231-030	Bonnet, upper	VASCO Hypercut	47D174818
45.	furn by NSP	Assembly-ball nut	SST 17-4PH	119C2893P2
46.	92233-030	Ball Screw	VASCO Hypercut	47C144024P2
47.	53-322	SCR, Set (hex soc cup pt) 1/4-20 x 5/8 lg	SST303	
48.	furn by NSP	Spur gear	VASCO Hypercut	119C2624P1

*Hoke, Inc.; Cresskill, N.J.

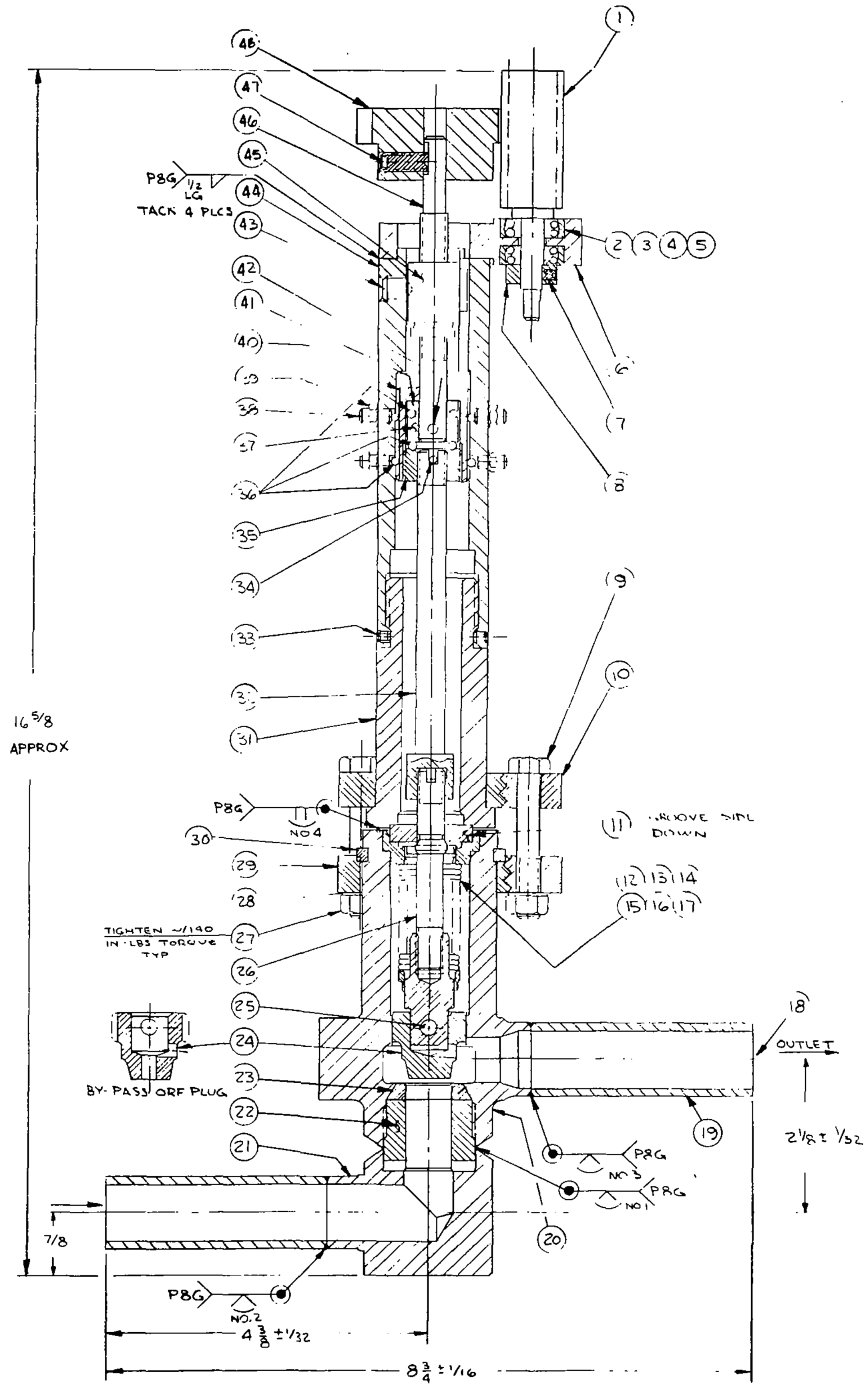


Figure 71. High Temperature Alkali Metal Valve Drawing.

APPENDIX C

QUALITY ASSURANCE TESTS OF REFRACTORY METAL MILL PRODUCTS AND CERMETS

The results of the quality assurance tests are presented in Tables XXII through XXV. A summary of the quality assurance test results with respect to meeting the requirements of the specification is shown in Table XXVI.

The failure of the 0.040-inch thick Cb-1Zr alloy sheet (MCN 07A-007-01) to meet the minimum grain size requirements was not considered serious since the deviation from the specification was small and the material is not a part of the loop assembly. The 0.040-inch thick Cb-1Zr sheet was utilized for welding quality control test coupons.

Although the oxygen content reported for the 0.094-inch diameter Cb-1Zr alloy wire (MCN 07A-034-01) was slightly above the specification limits,* the wire was considered acceptable for its intended use as weld filler material. Also, the 0.094-inch diameter T-111 alloy wire (MCN 07A-015-01) which was found to be in the cold worked condition was acceptable since its intended use is weld filler material.

The large variations in grain size and percent recrystallization found in the 3.0-inch diameter T-111 alloy rod (MCN 07A-017-01) were accepted because the thinnest sections in the final machined part, the valve body outlet, will still have a sufficient number of grains across the wall in the thinnest direction to be structurally sound.

The 0.250-inch diameter W-25Re alloy rod (MCN 07A-031-01) which was found to be 100% recrystallized rather than in the preferred stress relieved condition was considered acceptable because the strength and ductility requirements in its ultimate use as the valve plug pin are very modest for W-25Re alloy.

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*Specification requires 270 ppm oxygen maximum; wire analyzed 276 and 290 ppm.

TABLE XXII. RESULTS OF QUALITY ASSURANCE TEST PROGRAM

Alloy	MCN Number	Mill Product		Vendor	Heat Number	Specifications		Meets All Specification Requirements	Remarks
		Form	Size			Number	Major Exceptions		
Cb-1Zr	07A-003-(1-8)	Foil	0.002" x 0.5" x 4.39 lbs	Union Carbide	5179	01-0054-00-A	None	Yes	
	07A-035-01	Sheet	0.0175" x 12.0" x 19.375"	Fansteel	80B950	01-0003-00-B	None	Yes	
	07A-036-01	Sheet	0.0175" x 12.0" x 19.750"	Fansteel	80B950	01-0003-00-B	None	Yes	
	07A-007-01	Sheet	0.040" x 12.0" x 12.0"	Union Carbide	51245	01-0053-01-B	None	No	Grain size below minimum
	07A-037-01	Bar	0.5" x 2.0" x 6.0"	Wah Chang	912-1211	01-0003-00-B	No Stress-Rupture	Yes	
	07A-038-01	Bar	0.5" x 6.0" x 25.0"	Wah Chang	912-1211	01-[illegible]-00-B	No Stress-Rupture	Yes	
	07A-028-01	Bar	0.5" x 1.0" x 6.0"	Union Carbide	5155	01-0003-00-B	None	Yes	
	07A-029-01	Bar	0.5" x 1.0" x 6.0"	Union Carbide	5155	01-0008-00-B	None	Yes	
	07A-005-01	Wire	0.094" Diameter x 5.20 lbs	Union Carbide	5191	01-0055-00-A	None	No	High oxygen (718 ppm) rejected to vendor
	07A-034-01	Wire	0.094" Diameter x 5.40 lbs	Union Carbide	51200	01-0055-00-A	None	No	High oxygen (276/290 ppm)
	07A-004-01	Rod	0.250" Diameter x 9.0"	Union Carbide	5191	01-0052-01-B	None	Yes	
	07A-039-01	Rod	0.5" Diameter x 22.0"	Wah Chang	911-70589	01-0003-04-B	None	Yes	
	07A-006-01	Rod	0.625" Diameter x 36.0"	Union Carbide	51200	01-[illegible]-01-B	None	Yes	
	07A-040-01	Rod	1.125" Diameter x 6.0"	Union Carbide	5155	01-0003-00-B	None	Yes	
	07A-041-01	Rod	1.125" Diameter x 6.0"	Union Carbide	5155	01-0003-00-B	None	Yes	
	07A-042-01	Rod	2.0" Diameter x 6.0"	Fansteel	2701	[illegible]-00-B	2400°F Anneal	Yes	
	07A-043-01	Rod	2.0" Diameter x 12.0"	Union Carbide	5154	01-0003-00-B	None	No	Stress-rupture life below min.
	07A-044-01	Tube	0.375" OD x 0.065" Wall x 9.0"	Wah Chang	98-70546	01-0004-02-B	No Stress-Rupture	Yes	
	07A-025-01	Tube	0.375" OD x 0.065" Wall x 76.5"	Wah Chang	98-70546	01-0004-02-B	No Stress-Rupture	Yes	
	07A-026-01	Tube	0.375" OD x 0.065" Wall x 76.5"	Wah Chang	98-70546	01-0004-02-B	No Stress-Rupture	Yes	
	07A-027-01	Tube	0.375" OD x 0.065" Wall x 77.0"	Wah Chang	98-70546	01-0004-02-B	No Stress-Rupture	Yes	
	07A-045-01	Tube	1.0" OD x 0.100" Wall x 7.0"	Wah Chang	98-70546	01-0004-02-B	None	Yes	
	07A-046-01	Tube	1.0" OD x 0.080" Wall x 44.5"	Wah Chang	98-70117	01-0004-01-A	None	Yes	
T-111	07A-047-01	Foil	0.005" x 3.5" x 48.0"	Fansteel	111-D-1632	01-0043-00-A	None	Yes	
	07A-008-01	Foil	0.005" x 5.0" x 2.48 lbs	Wah Chang	65076	01-0043-00-A	None	Yes	
	07A-014-01	Sheet	0.040" x 4.0" x 12.0"	Wah Chang	65080	01-0040-00-B	None	Yes	Post rupture oxygen high (+80 ppm)
	07A-013-01	Sheet	0.040" x 12.0" x 12.0"	Wah Chang	65080	01-0040-00-B	None	Yes	Post rupture oxygen high (+80 ppm)
	07A-012-01	Sheet	0.062" x 2.0" x 9.0"	Wah Chang	65080	01-[illegible]-00-B	None	Yes	Post rupture oxygen high (+80 ppm)

TABLE XXII. RESULTS OF QUALITY ASSURANCE TEST PROGRAM (CONT.)

Alloy	MCN Number	Mill Product: Form	Mill Product: Size	Vendor	Heat Number	Specifications: Number	Specifications: Major Exceptions	Meets All Specifications Requirements	Remarks
T-111	07A-048-01	Wire	0.094" Diameter x 5.0"	Fansteel	111-D-1633	01-0048-00-A	None	Yes	
	07A-015-01	Wire	0.094" Diameter x 4.82 lbs	Wah Chang	65080	01-0048-00-A	None	No	Not recrystallized
	07A-009-01	Rod	0.500" Diameter x 18.0"	Wah Chang	65080	01-0015-00-B	None	Yes	Post rupture oxygen high (+80 ppm & +180 ppm)
	07A-010-01	Rod	0.875" Diameter x 9.0"	Wah Chang	65080	01-0015-00-B	None	Yes	Post rupture oxygen high (+75 ppm)
	07A-011-01	Rod	1.750" Diameter x 12.0"	Wah Chang	65080	01-0015-00-B	None	Yes	
	07A-049-01	Rod	2.0" Diameter x 4.125"	Fansteel	111-D-1102	01-0015-00-B	None	Yes	
	07A-002-01	Rod	2.0" Diameter x 24.375"	Fansteel	111-D-1670	01-0015-00-B	None	Yes	
	07A-017-01	Rod	3.0" Diameter x 24.0"	Wah Chang	65080	01-0015-00-B	None	No	Large variance in grain size and percent recrystallization
	07A-018-(1-63)	Tube[(1)]	0.625" OD x 0.0085" Wall x 76.5"	Superior	111-D-1670	01-0035-00-A	None	Yes	
	07A-060-01	Tube	1.0" OD x 0.100" Wall x 2.0"	Fansteel	111-D-1670	01-0035-00-B	None	Yes	
T-222	07A-001-01	Sheet	0.040" x 4.187" x 12.125"	Westinghouse	Ta-43-FS-2	Best Effort	----	---	
ASTAR 811	07A-052-01	Sheet	0.040" x 2.0" x 12.0"	Westinghouse	NASV-22	Best Effort	----	---	
ASTAR 811C	07A-053-01	Sheet	0.042" x 4.0" x 6.250"	Westinghouse	NASV-20	Best Effort	----	---	
ASTAR 811CN	07A-054-01	Sheet	0.040" x 0.78" x 12.0"	Westinghouse	NASV-23	Best Effort	----	---	
Mo-TZM	07A-021-01	Wire	0.093" Diameter x 6.0'	Climax	TZM 7480	CMX-WB-TZM-2	None	Yes	
	07A-020-01	Rod	0.375" Diameter x 20.0"	Climax	TZM 7484	CMX-WB-TZM-2	None	Yes	
	07A-023-01	Rod	0.50" Diameter x 36.0"	Climax	TZM 7495	CMX-WB-TZM-2	None	Yes	
	07A-081-01	Rod	0.875" Diameter x 5.875"	Climax	TZM 7874	CMX-WB-TZM-2	None	Yes	
W-25Re	07A-022-01	Rod	0.125" Diameter x 10.0"	Cleveland Refractory Metals	-----------------	01-0067-00-A (MOD)	Processing, Chemistry, and Hardness	Yes	Vendor analyses for C, O, and N high, but G.E. check analyses acceptable
	07A-031-01	Rod	0.250" Diameter x 6.0"	Rhenium Alloys, Inc.	WC-950	P.O. 037-284331	----	No	100% recrystallized
	07A-030-01	Rod	1.125" Diameter x 7.75"	Wah Chang	-----------------	P.O. 037-271914	----	Yes	Vendor analyses for O and N high, but G.E. check analyses acceptable
W-25Re-30Mo (atomic percent)	07A-019-(1-5)	Sheet	0.030" x 3.0" x 4.625"	GE-NMPO	-----------------	Best Effort	----	---	
Re	07A-032-01	Plate	0.250" x 0.5" x 1.250"	Cleveland Refractory Metals	(2)	P.O. 037-284596	----	Yes	
	07A-032-02	Plate	0.250" x 1.250" x 1.250"	Cleveland Refractory Metals	(2)	P.O. 037-284596	----	Yes	
	07A-033-(1-3)	Valve Seats	Hoke Drawing No. 474-138	Cleveland Refractory Metals	(2)	P.O. 037-284596	----	Yes	

TABLE XXII. RESULTS OF QUALITY ASSURANCE TEST PROGRAM (CONT.)

Alloy	MCN Number	Mill Product		Vendor	Heat Number	Specifications		Meets All Specification Requirements	Remarks
		Form	Size			Number	Major Exceptions		
TiC-10Cb	07A-024-(1-10)	Valve Seats	Hoke Drawing No. 474-138	Kennametal Inc.	Powder Batch 8038	01-0025-00-B	None	Yes	One seat failed penetrant inspection

(1) Tubing from MCN 07A-002-01.

(2) Heat number is not available, but 07A-032-01, 07A-032-02, and 07A-033-(1-3) were manufactured from one bar.

TABLE XXIII. CHEMICAL ANALYSIS OF REFRACTORY ALLOY MILL PRODUCTS AND CERMETS

Alloy	MCN Number	Mill Product Form	Mill Product Size	Heat Number	Sample Source	Analyzed By	Chemical Analyses, ppm C	O	N	H	Other %
Cb-1Zr	SPECIFICATION 01-0064-00-A						Max 150	Max 300	Max 100	Max 10	Zr 0.8-1.2
	07A-003-(1-8)	Foil	0.002" x 0.5" x 4.39 lbs	5179	Ingot	Vendor	10	35	26	2	0.81
					Final Product	Vendor	30	87	61	9	-------
	SPECIFICATION 01-0003-00-B						Max 100	Max 300	Max 300	Max 15	Zr 0.8-1.2
	07A-035-01	Sheet	0.0175" x 12.0" x 19.375"	808950	Ingot	Vendor	--------	--------	--------	-------	1.06
					Final Product	Vendor	5	20	46	5	-------
					Final Product	G.E.	50	281	91	1	-------
	07A-036-01	Sheet	0.0175" x 12.0" x 19.750"	808950	Ingot	Vendor	-------	--------	--------	-------	1.06
					Final Product	Vendor	5	20	46	5	-------
					Final Product	G.E	50	281	91	1	-------
	SPECIFICATION 01-0063-01-B						Max 200	Max 300	Max 100	Max 10	Zr 0.8-1.2
	07A-007-01	Sheet	0.040" x 12.0" x 12.0"	51254	Ingot	Vendor	20	118	68	4	1.15
					Final Product	Vendor	30 (1)	42 (1)	55 (1)	3 (1)	-------
					Final Product	G.E.	22	84	68	<1	-------
	SPECIFICATION 01-0003-00-B						Max 100	Max 300	Max 300	Max 15	Zr 0.8-1.2
	07A-037-01	Bar	0.5" x 2.0" x 6.0"	912-1211	Ingot	Vendor	<30 (1)	145 (1)	65 (1)	5 (1)	1.04 (1)
					Final Product	Vendor	<30 (1)	80	50	4	-------
					Final Product	G.E.	30 (1)	130 (1)	44 (1)	1 (1)	-------
	07A-038-01	Bar	0.5" x 6.0" x 25.0"	912-1211	Ingot	Vendor	<30 (1)	145	65	5	1.04 (1)
					Final Product	Vendor	<30 (1)	80	50	4	-------
					Final Product	G.E.	30 (1)	130	44	1	-------
	07A-028-01	Bar	0.5" x 1.0" x 6.0"	5155	Final Product	Vendor	10 (1)	<1	15	4	-------
					Final Product	G.E.	20	18	13	1	-------
	07A-029-01	Bar	0.5" x 1.0" x 6.0"	5155	Final Product	Vendor	10 (1)	<1	15	4	-------
					Final Product	G.E.	20	18	13	1	-------
	SPECIFICATION 01-0055-00-A						Max 100	Max 250	Max 100	Max 10	Zr 0.8-1.2
	07A-005-01	Wire	0.094" Diameter x 5.20 lbs	5191	Ingot	Vendor	70	55	54	4	0.95
					Final Product	Vendor	100 (1)	250 (1)	95 (1)	2 (1)	-------
					Final Product	G.E.	79	718	96	3	-------
	07A-034-01	Wire	0.094" Diameter x 5.40 lbs	51200	Ingot	Vendor	60	110	60	4	1.01
					Final Product	Vendor	38 (1)	290 (1)	82 (1)	1 (1)	-------
					Final Product	G.E.	89	286	87	2	-------
	SPECIFICATION 01-0062-01-B						Max 200	Max 300	Max 100	Max 10	Zr 0.8-1.2
	07A-004-01	Rod	0.250" Diameter x 9.0"	5191	Ingot	Vendor	30	55	54	4	0.96
					Final Product	Vendor	10	98	95	3	-------
	SPECIFICATION 01-0003-04-B						Max 100	Max 300	Max 300	Max 10	Zr 0.8-1.2
	07A-039-01	Rod	0.5" Diameter x 22.0"	911-70559	Ingot	Vendor	30 (1)	165 (1)	68 (1)	4.9 (1)	0.91 (1)
					Final Product	Vendor	30 (1)	148 (1)	30 (1)	1.9	-------
					Final Product	G.E.	32	156	35	<1	-------

TABLE XXIII. CHEMICAL ANALYSIS OF REFRACTORY ALLOY MILL PRODUCTS AND CERMETS (CONT.)

Alloy	MCN Number	Mill Product Form	Mill Product Size	Heat Number	Sample Source	Analyzed By	C	O	N	H	Other %	
							Chemical Analyses, ppm					
Cb-1Zr	SPECIFICATION 01-0052-01-B						Max 200	Max 300	Max 100	Max 10	Zr 0.8-1.2	
	07A-006-01	Rod	0.625" Diameter x 36.0"	51200	Ingot	Vendor	60	110	60	4	1.01	
					Final Product	Vendor	70	163	57	6	-------	
	SPECIFICATION 01-0003-00-B						Max 100	Max 300	Max 300	Max 15	Zr 0.8-1.2	
	07A-040-01	Rod	1.125" Diameter x 6.0"	5155	Final Product	Vendor	10	6	23	7	-------	
					Final Product	G.E.	30 (1)	18	41	2	-------	
	07A-041-01	Rod	1.125" Diameter x 6.0"	5155	Final Product	Vendor	10	6	23	7	-------	
					Final Product	G.E.	30 (1)	18	41	2	-------	
	07A-042-01	Rod	2.0" Diameter x 6.0"	2701	Ingot	Vendor	<10	81	68	<5	0.90	
					Final Product	Vendor	<10	90	50	7	-------	
					Final Product	Vendor	10 (1)	97.5 (1)	61.5 (1)	9 (1)	-------	
					Final Product	G.E.	110	141	48	8	-------	
	07A-043-01	Rod	2.0" Diameter x 12.0"	5154	Ingot	Vendor	46 (1)	38	20 (1)	6 (1)	0.90	
					Final Product	Vendor	10	18	24	7	-------	
					Final Product	G.E.	25 (1)	22 (1)	22 (1)	9 (1)	-------	
	SPECIFICATION 01-0004-02-B						Max 100	Max 300	Max 300	Max 10	Zr 0.8-1.2	
	07A-044-01	Tube	0.375" OD x 0.065" Wall x 9.0"	98-70546	Ingot	Vendor	65 (1)	200 (1)	55 (1)	3 (1)	0.87 (1)	
					Final Product	Vendor	80	110	65	3	-------	
					Final Product	G.E.	80 (1)	159	29	5	-------	
	07A-025-01	Tube	0.375" OD x 0.065" Wall x 76.5"	98-70546	Ingot	Vendor	65 (1)	200 (1)	55 (1)	3 (1)	0.87 (1)	
					Final Product	Vendor	80	110	65	3	-------	
					Final Product	G.E.	80 (1)	159	29	5	-------	
	07A-026-01	Tube	0.375" OD x 0.065" Wall x 76.5"	98-70546	Ingot	Vendor	65 (1)	200 (1)	55 (1)	3 (1)	0.87 (1)	
					Final Product	Vendor	80	110	65	3	-------	
					Final Product	G.E.	80 (1)	159	29	5	-------	
	07A-027-01	Tube	0.375" OD x 0.065" Wall x 77.0"	98-70546	Final Product	Vendor	80	110	65	3	-------	
					Final Product	G.E.	45 (1)	224	67	9	-------	
	07A-045-01	Tube	1.0" OD x 0.100" Wall x 7.0"	98-70546	Ingot	Vendor	65 (1)	200 (1)	55 (1)	3 (1)	0.87 (1)	
					Final Product; Stress-Relieved 1800°F/1 hr	Vendor	90	200	35	2	--------	
					Final Product; 2200°F/1 hr	G.E.	40	226	64	6	--------	
	SPECIFICATION 01-0004-01-A						Max 100	Max 300	Max 300	Max 10	Zr 0.8-1.2	
	07A-046-01	Tube	1.0" OD x 0.080" Wall x 44.5"	98-70117	Ingot	Vendor	50 (1)	265 (1)	55 (1)	3.2 (1)	0.89 (1)	
					Final Product	Vendor	80	260	85	3.5	-------	
					Final Product	G.E.	75 (1)	253	21	2	-------	
T-111	SPECIFICATION 01-0043-00-A						Max 75	Max 300	Max 100	Max 10	W 7.0-9.0	Hf 1.8-2.4
	07A-047-01	Foil	0.005" x 3.5" x 49.0"	111-D-1632	Ingot	Vendor	10	25	20	5	7.79 (1)	2.38 (1)
					Extruded Bar	Vendor	11 (2)	15 (2)	21 (2)	4 (2)	7.73 (2)	2.21 (2)
					Final Product	Vendor	12	41	20	5.2	-------	-------
	07A-0[illegible]-01	Foil	0.005" x 5.0" x 2.48 lbs	65076	Ingot	Vendor	<40 (3)	50 (1)	14 (3)	2.8 (1)	8.60 (3)	1.93 (3)
					Final Product	Vendor	<40	[illegible]	9	2.6	-------	-------
					Final Product	G.E.	56 (1)	[illegible]	4 (1)	10 (1)	-------	-------
	SPECIFICATION 01-0040-00-B						Max 50	Max 150	Max 75	Max 10	W 7.0-9.0	Hf 1.8-2.4
	07A-0[illegible]	Sheet	0.040" x 4.0" x 12.0"	65080	Ingot	Vendor	<40 (2)	100 (1)	24 (2)	3.2 (1)	8.88 (2)	1.91 (2)
					Final Product	Vendor	<40	50	15	1.8	-------	-------
					Final Product	G.E.	26 (1)	41 (1)	4 (1)	3 (1)	-------	-------

TABLE XXIII. CHEMICAL ANALYSIS OF REFRACTORY ALLOY MILL PRODUCTS AND CERMETS (CONT.)

Alloy	MCN Number	Mill Product: Form	Mill Product: Size	Heat Number	Sample Source	Analyzed By	Chemical Analysis, ppm: C	O	N	H		Others %
T-111	07A-013-01	Sheet	0.040" x 12.0" x 12.0"	65080	Ingot	Vendor	<40 (2)	100 (1)	24 (2)	3.2 (1)	8.88 (2)	1.91 (2)
					Final Product	Vendor	<40	50	15	1.8	--------	--------
					Final Product	G.E.	21 (1)	37 (1)	4 (1)	2 (1)	--------	--------
	07A-012-01	Sheet	0.062" x 2.0" x 9.0"	65080	Ingot	Vendor	<40 (2)	100 (1)	24 (2)	3.2 (1)	8.88 (2)	1.91 (2)
					Final Product	Vendor	<40	<50	20	1.7	--------	--------
					Final Product	G.E.	23 (1)	31 (1)	4 (1)	3 (1)	--------	--------
	SPECIFICATION 01-0048-00-A						Max 50	Max 150	Max 75	Max 10	W 7.0-9.0	Hf 1.8-2.4
	07A-048-01	Wire	0.094" Diameter x 5.0"	111-D-1633	Ingot	Vendor	10	25	10	10	7.74 (1)	2.23 (1)
					Extruded Bar	Vendor	17	19	16	6	7.80 (2)	2.24 (2)
					Finished Product	Vendor	33	69	21	<1	--------	--------
					Finished Product	G.E.	18 (1)	68	5	1	8.41	2.73
	07A-015-01	Wire	0.094" Diameter x 4.82 lbs	65080	Ingot	Vendor	<40 (2)	100 (1)	24 (2)	3.2 (1)	8.88 (2)	1.91 (2)
					Final Product	Vendor	<40	110	13	3.7	--------	--------
					Final Product	G.E.	22 (1)	41 (1)	4 (1)	2 (1)	--------	--------
	SPECIFICATION 01-0015-00-B						Max 50	Max 150	Max 75	Max 10	W 7.0-9.0	Hf 1.8-2.4
	07A-009-01	Rod	0.500" Diameter x 18.0"	65080	Ingot	Vendor	<40 (2)	100 (1)	24 (2)	3.2 (1)	8.88 (2)	1.91 (2)
					Final Product	Vendor	<40	<50	15	2.7	--------	--------
					Final Product	G.E.	29 (1)	15 (1)	4 (1)	3 (1)	--------	--------
	07A-010-01	Rod	0.875" Diameter x 9.0"	65080	Ingot	Vendor	<40 (2)	100 (1)	24 (2)	3.2 (1)	8.88 (2)	1.91 (2)
					Final Product	Vendor	<40	<50	8	2.6	--------	--------
					Final Product	G.E.	23 (1)	14 (1)	5 (1)	5 (1)	--------	--------
	07A-011-01	Rod	1.750" Diameter x 12.0"	65080	Ingot	Vendor	<40 (2)	100 (1)	24 (2)	3.2 (1)	8.88 (2)	1.91 (2)
					Final Product	Vendor	<40	90	20	2.1	--------	--------
					Final Product	G.E.	23 (1)	17 (3)	3 (3)	7 (1)	--------	--------
	07A-049-01	Rod	2.0" Diameter x 4.125"	111-D-1102	Ingot	Vendor	10	53	20	5	7.97 (1)	2.4 (1)
					Extruded Bar	Vendor	20 (2)	51 (2)	21 (2)	3 (2)	7.94 (2)	2.28 (2)
					Final Product	Vendor	20	73	26	3.7	--------	--------
					Final Product	G.E.	26 (1)	43 (1)	7 (1)	4 (1)	--------	--------
	07A-002-01	Rod	2.0" Diameter x 24.375"	111-D-1670	Ingot	Vendor	10	72	20	5	7.70 (3)	2.17 (3)
					Extruded Bar	Vendor	29 (2)	20 (2)	16 (2)	7 (2)	7.66 (2)	2.37 (2)
					Final Product	Vendor	26	30	31	1.7	--------	--------
					Final Product	G.E.	25 (1)	39	13	13	--------	--------
	07A-017-01	Rod	3.0" Diameter x 24.0"	65080	Ingot	Vendor	<40 (2)	100 (1)	24 (2)	3.2 (1)	8.88 (2)	1.91 (2)
					Final Product	Vendor	<40	70	12	1.5	--------	--------
					Final Product	G.E.	23 (1)	7 (1)	6 (1)	2 (1)	--------	--------
	SPECIFICATION 01-0035-00-A						Max 50	Max 200	Max 75	Max 10	W 7.0-9.0	Hf 1.8-2.4
	07A-018-(1-63)	Tube (1)	0.625" OD x 0.0065" Wall x 76.5"	111-D-1670	Ingot	Vendor	10	72	20	5	7.70 (3)	2.17 (3)
					Final Product	Vendor	39	84	14	8	--------	--------
					Final Product	G.E.	43 (1)	94 (1)	12 (1)	11 (1)	--------	--------
	07A-050-01	Tube	1.0" OD x 0.100" Wall x 2.0"	111-D-1670	Ingot	Vendor	10	72	20	8	7.70 (3)	2.17 (3)
					Extruded Bar	Vendor	29 (2)	20 (2)	16 (2)	7 (2)	7.65 (2)	2.37 (2)
					Final Product	Vendor	23	24	45	5	--------	--------
					Final Product	G.E.	27	26	5	6	--------	--------
T-222	SPECIFICATION: BEST EFFORT						C	O	N	H	W	Hf
	07A-001-01	Sheet	0.040" x 4.187" x 12.125"	Ta-43-FS-2	Final Product	Vendor	90	17	10	--------	10.6	2.58
					Final Product	G.E.	92 (1)	46 (1)	9 (1)	2 (1)	--------	--------

TABLE XXIII. CHEMICAL ANALYSIS OF REFRACTORY ALLOY MILL PRODUCTS AND CERMETS (CONT.)

Alloy	MCN Number	Mill Product Form	Mill Product Size	Heat Number	Sample Source	Analyzed By	Chemical Analyses, ppm C	O	N	H	Other %			
ASTAR 811	SPECIFICATION	BEST EFFORT									W	Hf	Re	
	07A-052-01	Sheet	0.040" x 2.0" x 12.0"	NASV-22	Ingot	Vendor	13(1)	16(1)	12(1)	------	8.0	1.0	1.0	
					Final Product (As-rolled)	G.E.	19(1)	14(1)	12(1)	1(1)	---	---	---	
					Final Product (3000°F/1 hr)	G.E.	12	16	2	<1	---	---	---	
ASTAR 811C	SPECIFICATION:	BEST EFFORT									W	Hf	Re	
	07A-053-01	Sheet	0.042" x 4.0" x 6.250"	NASV-20	Final Product	G.E.	204(1)	12(1)	8(1)	1(1)	---	---	---	
ASTAR 811CN	SPECIFICATION.	BEST EFFORT									W	Hf	Re	
	07A-054-01	Sheet	0.040" x 0.78" x 12.0"	NASV-23	Ingot	Vendor	120(1)	18(1)	120(1)	------	8.0	1.0	1.0	
					Final Product (As-rolled)	G.E.	127(1)	17(1)	21(1)	4(1)	---	---	---	
					Final Product (3000°F/1 hr)	G.E.	127	17	75(1)	2	---	---	---	
Mo-TZM	SPECIFICATION CMX-WB-TZM-2						100-300	Max 25	Max 20	Max 5	Ti 0.40-0.55	Zr 0.06-0.12		
	07A-021-01	Wire	0.093" Diameter x 6.0'	TZM 7469	Final Product	Vendor	170	<3	<1	<1	0.50	0.091		
	07A-020-01	Rod	0.375" Diameter x 20.0"	TZM 7464	Final Product	Vendor	180	4	2	<1	0.46	0.088		
	07A-023-01	Rod	0.50" Diameter x 36.0"	TZM 7495	Final Product	Vendor	160	<3	<1	<1	0.55	0.103		
	07A-051-01	Rod	0.875" Diameter x 5.875"	TZM 7876	Final Product	Vendor	160(1)	<4(1)	1(1)	<1(1)	0.49	0.105		
					Final Product	G.E.	176(1)	5(1)	7(1)	<1(1)	----	-----		
W-25Re	SPECIFICATION 01-0067-00-A (Mod)						Max 30	Max 40	Max 10	Max ---	Re 24.5-25.5			
	07A-022-01	Rod	0.125" Diameter x 10.0"	--------- -	Final Product	Vendor	53(1)	79(1)	18(1)	15(1)	25.05			
					Final Product	G.E	9(1)	12(1)	1(1)	2(1)	---------			
	SPECIFICATION - P.O. 037-284331						Max 80	Max 75	Max 25	Max 5	Re 24.8-25.1			
	07A-031-01	Rod	0.250" Diameter x 6.0"	WC-950	Final Product	Vendor	80(1)	20(1)	15	1	25.11			
					Final Product	G.E.	31(1)	43(1)	<1	<1	---- ----			
	SPECIFICATION - P.O. 037-271914						Max 80	Max 75	Max 25	Max 5	Re 24.8-25.1			
	07A-030-01	Rod	1.125" Diameter x 7.75"	----------	Final Product	Vendor	<40(1)	90(1)	31	2	25.40(1)			
					Final Product	G.E.	7(1)	7(1)	<1	<1	24.84			
W-25Re-30Mo (atomic percent)	SPECIFICATION:	BEST EFFORT					C	O	N	H	W	Hf		
	07A-019-(1-5)	Sheet	0.030" x 3.0" x 4.625"	----------	Final Product	G.E.	15(1)	9(1)	2(1)	1(1)	-------	-------		
Re	SPECIFICATION - P.O. 037-284596						Max 50	Max 40	Max 10	Max 5	W	Hf	Re	
	07A-037-01	Plate	0.250" x 0.5" x 1.250"	-----------	Final Product	G.E.	16(1)	10(1)	1(1)	1(1)	---	---	---	
	07A-037-02	Plate	0.250" x 1.250" x 1.250"	-----------	Final Product	G.E.	16(1)	10(1)	1(1)	1(1)	---	---	---	
	07A-033-(1-3)	Seat	Hoke Drawing 474-138	---- ------	Final Product	G.E.	16(1)	10(1)	1(1)	1(1)	---	---	---	
TiC-10Cb	SPECIFICATION 01-0025-00-B						O No Requirement	W Max 2.0%	Fe Max 0.5%	Co Max 0.5%	Ni Max 0.5%	Al Max 0.01%	Cb 9.5-10.5%	TiC Min 85.0
	24-(1-10)	Valve Seats		Batch No. 8038	Final Product	Vendor	-----	1.89	<0.10	0.43	0.40	-----	9.67	Remainder
					Final Product	G.E.	0.098	0.84	0.074	0.091	0.076	0.005	9.36	Remainder

1 Average of 2 analyses.

2 Average of 4 analyses or more

3 Average of 3 analyses.

TABLE XXIV. MECHANICAL PROPERTIES & GRAIN SIZE OF REFRACTORY ALLOY MILL PRODUCTS

Alloy	MCN Number	Mill Product Form	Mill Product Size	Heat Number	Final Heat Treatment	Room Temperature Tensile Properties Ult. Ksi	0.2%Y.S. Ksi	Elong. %	Stress-Rupture Life Hours	Stress-Rupture Life Elong, %	Bend or Flare	Hardness Bulk	Hardness Micro (DPH) Surface	Hardness Micro (DPH) Center	Grain Size Vendor ASTM No.	Grain Size OK ASTM No.	Recrystallization (%) Vendor	Recrystallization (%) OK
Cb-1Zr	SPECIFICATION 01-0054-00-A			-	-	-	-	-	-	-	180°w/No Fail.	-	-	-	-	-	100%	
	07A-003-(1-18)	Foil	0.002" x 0.5" x 4.39 lbs	5179	2400°F/1 hr	-	-	-	-	-	Passed Spec.	-	-	-	-	-	-	-
	SPECIFICATION 01-0003-00-B			-	-	Max 75	Max 60	Min 10	Min 25	-	105°w/No Fail.	Max $90R_b$	Max 50 VHN Gradient		Min 3		100	
	07A-035-01	Sheet	0.0175" x 12.0" x 19.375"	80B950	2200°F/1 hr	42.9(1)	29.9(1)	25.5(1)	-	-	-	$49.5R_{30-T}$	95	95	6.5	-	-	-
	07A-036-01	Sheet	0.0175" x 12.0" x 19.750"	80B950	2200°F/1 hr	42.9(1)	29.9(1)	25.5(1)	-	-	-	$49.5R_{30-T}$	95	95	6.5	-	-	-
	SPECIFICATION 01-0053-01-B			-	-	Max 75	Max 60	Min 20	-	-	105°w/No Fail.	Max $90R_b$	Max 50 VHN Gradient		Min 6		100	
	07A-007-01	Sheet	0.040" x 12.0" x 12.0"	51245	2400°F/1 hr	43.3(1)	28.5(1)	28.7(1)	-	-	Passed Spec.	-	-	-	-	5	-	100
	SPECIFICATION 01-0003-00-B			-	-	Max 75	Max 60	Min 10	Min 25	-	-	Max $90R_b$	Max 50 VHN Gradient		Min 3		100	
	07A-037-01	Bar	0.5" x 6.0" x 25.0"	912-1211	2200°F/1 hr	43.4(1)	26.7(1)	29.0(1)	>30(2)	-	-	$60R_b$	-	-	8	-	-	-
	07A-038-01	Bar	0.5" x 6.0" x 25.0"	912-1211	2200°F/1 hr	43.4(1)	26.7(1)	29.0(1)	21(2)	-	-	$60R_b$	-	-	8	-	-	-
	07A-028-01	Bar	0.5" x 1.0" x 6.0"	5155	2200°F/1 hr	38.9(1)	14.7(1)	43.0(1)	29(2)	45(2)	-	-	-	-	-	-	-	-
	07A-029-01	Bar	0.5" x 1.0" x 6.0"	5155	2200°F/1 hr	38.9(1)	14.7(1)	43.0(1)	24(2)	55(2)	-	-	86	95	5-6	-	-	-
	SPECIFICATION 01-0055-00-A			-	-	-	-	-	-	-	-	-	-	-	-	-	100	
	07A-005-01	Wire	0.094" Diameter x 5.20 lbs	5191	2400°F/1 hr	-	-	-	-	-	-	-	-	-	-	6-8	-	100
	07A-034-01	Wire	0.094" Diameter x 5.40 lbs	51200	2400°F/1 hr	-	-	-	-	-	-	-	-	-	-	-	-	-
	SPECIFICATION 01-0062-01-B			-	-	Max 75	Max 60	Min 20	-	-	-	Max $90R_b$	-	-	Min 4		100	
	07A-004-01	Rod	0.250" Diameter x 9.0"	5191	2200°F/1 hr	53.3(1)	31.6(1)	37.6(1)	-	-	-	$49R_b$	-	-	10	-	-	-
	SPECIFICATION 01-0003-04-B			-	-	Max 75	Max 60	Min 10	-	-	-	Max $90R_b$	-	-	Min 3		100	
	07A-039-01	Rod	0.5" Diameter x 22.0"	911-70559	2200°F/1 hr	41.8(1)	26.2(1)	41.5(1)	-	-	-	86DPH	86	85	7	-	-	-
	SPECIFICATION 01-0052-01-B			-	-	Max 75	Max 60	Min 20	-	-	-	Max $90R_b$	Max 50 VHN Gradient		Min 4		100	
	07A-006-01	Rod	0.625" Diameter x 36.0"	51200	2400°F/1 hr	46.9(1)	26.1(1)	40.7(1)	-	-	-	$46R_b$	-	-	6	-	-	-
	SPECIFICATION 01-0003-00-B			-	-	Max 75	Max 60	Min 10	Min 25	-	-	Max $90R_b$	Max 50 VHN Gradient		Min 3		100	
	07A-040-01	Rod	1.125" Diameter x 6.0"	5155	2200°F/1 hr	-	-	-	-	-	-	-	-	97	5-6	-	-	-
	07A-041-01	Rod	1.125" Diameter x 6.0"	5155	2200°F/1 hr	-	-	-	-	-	-	-	-	97	5-6	-	-	-

TABLE XXIV. MECHANICAL PROPERTIES & GRAIN SIZE OF REFRACTORY ALLOY MILL PRODUCTS (CONT.)

Alloy	MCN Number	Mill Product Form	Mill Product Size	Heat Number	Final Heat Treatment	Room Temperature Tensile Properties Ult. Ksi	0.2%Y.S. Ksi	Elong. %	Stress-Rupture Life Hours	Elong. %	Bend or Flare	Hardness Bulk	Micro (DPH) Surface	Micro (DPH) Center	Grain Size Vendor ASTM No.	GE ASTM No.	Recrystallization (%) Vendor	GE
Cb-1Zr	07A-042-01	Rod	2.0" Diameter x 6.0"	2701	2400°F 1 hr	36.9$^{(1)}$	22.5$^{(1)}$	29$^{(1)}$	>29$^{(2)}$	-	-	$45R_b$	129	117	-	-	Partial	-
	07A-043-01	Rod	2.0" Diameter x 12.0"	5154	2200°F/1 hr	39.4$^{(1)}$	34.8$^{(1)}$	37$^{(1)}$	12$^{(1)(2)}$	41$^{(1)(2)}$	-	-	-	112	5-6	-	Partial	-
	SPECIFICATION 01-0004-02-B				-	Max 75	Max 60	Min 10	Min 25	-	Flare (60°) 1.15 x Dia	Max $90R_b$	Max 50 VHN Gradient		Min 4		100	
	07A-044-01	Tube	0.375" OD x 0.065" Wall x 9.0"	98-70546	2200°F 1 hr	36.3$^{(1)}$	18.1$^{(1)}$	36$^{(1)}$	>28$^{(1)(2)}$	>1$^{(1)(2)}$	Two Passed	$51R_b$	-	-	7	-	-	-
	07A-025-01	Tube	0.375" OD x 0.065" Wall x 78.5"	98-70546	2200°F/1 hr	36.3$^{(1)}$	18.1$^{(1)}$	36$^{(1)}$	>28$^{(1)(2)}$	>1$^{(1)(2)}$	Two Passed	$51R_b$	-	-	7	-	-	-
	07A-026-01	Tube	0.375" OD x 0.065" Wall x 76.5"	98-70546	2200°F/1 hr	36.3$^{(1)}$	18.1$^{(1)}$	36$^{(1)}$	>28$^{(1)(2)}$	>1$^{(1)(2)}$	Two Passed	$51R_b$	-	-	7	-	-	-
	07A-027-01	Tube	0.375" OD x 0.065" Wall x 77.0"	98-70546	2200°F/1 hr	36.3$^{(1)}$	18.1$^{(1)}$	36$^{(1)}$	-	-	Two Passed	$51R_b$	-	-	6-8	-	Surface Cold Worked	-
	07A-045-01	Tube	1.0" OD x 0.100" Wall x 7.0"	98-70546	1800°F/1 hr	87.3$^{(1)}$	67.4$^{(1)}$	34$^{(1)}$	-	-	Two Passed	-	-	-	-	-	Stress-Relieved	-
					1800°F/1 hr + 2200°F/1 hr	45.2$^{(1)}$	34.8$^{(1)}$	30$^{(1)}$	37.4$^{(2)}$	>2$^{(2)}$	-	-	-	-	-	-	-	-
	SPECIFICATION 01-0004-01-A			-	-	Max 75	Max 60	Min 10	Min 25	-	Flare (60°) 1.15 x Dia	Max $90R_b$	Max 50 VHN Gradient		Min 5		100	
	07A-046-01	Tube	1.0" OD x 0.080" Wall x 44.5"	98-70117	2200°F/1 hr	46.7$^{(1)}$	25.2$^{(1)}$	26.5$^{(1)}$	>100$^{(1)(2)}$	-	Two Passed	$28R_b$	-	-	-	-	-	-
T-111	SPECIFICATION 01-0043-00-A			-	-	-	-	-	-	-	180°w/No Fail.	-	-	-	-	-	100	
	07A-047-01	Foil	0.005" x 3.5" x 48.0"	111-D-1632	2685°F/1 hr	-	-	-	-	-	180° Passed	-	-	-	-	-	100	-
	07A-008-01	Foil	0.005" x 5.0" x 2.48 lbs	65076	3000°F/1 hr	-	-	-	-	-	180° Passed	215BHN	-	-	-	7	100	100
	SPECIFICATION 01-0040-00-B			-	-	Max 110	Max 100	Min 20	Min 20	-	105°w/No Fail.	-	Max 50 VHN Gradient		Min 6		100	
	07A-014-01	Sheet	0.040" x 4.0" x 12.0"	65080	3000°F/1 hr	93.8$^{(1)}$	84.1$^{(1)}$	28.5$^{(1)}$	20.1$^{(1)(3)}$	-	180° Passed	217BHN	235	237	9	7-8	100	100
	07A-013-01	Sheet	0.040" x 12.0" x 12.0"	65080	3000°F/1 hr	93.8$^{(1)}$	84.1$^{(1)}$	28.5$^{(1)}$	20.1$^{(1)(3)}$	-	180° Passed	217BHN	235	237	9	7-8	100	100
	07A-012-01	Sheet	0.062" x 2.0" x 9.0"	65080	3000°F/1 hr	93.8$^{(1)}$	78.8$^{(1)}$	29$^{(1)}$	20.1$^{(1)(3)}$	-	105° Passed	217BHN	237	223	9	7-8	100	100
	SPECIFICATION 01-0048-00-A			-	-	-	-	-	-	-	-	-	-	-	-	-	100	
	07A-048-01	Wire	0.094" Diameter x 5.0"	111-D-1633	2685°F/1 hr	-	-	-	-	-	-	-	-	-	-	6	100	100
	07A-015-01	Wire	0.094" Diameter x 4.82 lbs	65080	None Cold Worked	-	-	-	-	-	-	217BHN	-	-	-	-	-	As-worked
	SPECIFICATION 01-0015-00-B			-	-	Max 110	Max 100	Min 20	Min 20	-	-	-	Max 50 VHN Gradient		Min 4		100	
	07A-009-01	Rod	0.500" Diameter x 18.0"	65080	3000°F/1 hr	91.0$^{(1)}$	75.5$^{(1)}$	45$^{(1)}$	20$^{(1)(3)}$	-	-	217BHN	224	226	9	7	100	100
	07A-010-01	Rod	0.875" Diameter x 9.0"	65080	3000°F/1 hr	92.5$^{(1)}$	76.4$^{(1)}$	44.5$^{(1)}$	20$^{(1)(3)}$	-	-	217BHN	238	225	8.5	7	100	100

TABLE XXIV. MECHANICAL PROPERTIES & GRAIN SIZE OF REFRACTORY ALLOY MILL PRODUCTS (CONT.)

Alloy	MCN Number	Mill Product Form	Mill Product Size	Heat Number	Final Heat Treatment	Room Temperature Tensile Properties Ult. Ksi	0.2%Y.S. Ksi	Elong. %	Stress-Rupture Life Hours	Stress-Rupture Life Elong. %	Bend or Flare	Hardness Bulk	Hardness Micro (DPH) Surface	Micro (DPH) Center	Grain Size Vendor ASTM No.	Grain Size GE ASTM No.	Recrystallization (%) Vendor	Recrystallization (%) GE
T-111													Max 50 VHN Variance		Min 4		95	
	07A-011-01	Rod	1.750" Diameter x 12.0"	65080	3000°F/1 hr	87.8[1]	71.3[1]	42.5[1]	20.1[1][3]	-	-	217BHN	254	233	5.5-6	3.5	100	100
	07A-049-01	Rod	2.0" Diameter x 4.125"	111-D-1102	2685°F/2 hrs[6] +3000°F/1 hr	86.4[1][6]	75.4[1]	42.5[1]	16.6[3] 21.5[3][4]	-	-	-	225	209	4-6	5	100	100
	07A-002-01	Rod	2.0" Diameter x 24.375"	111-D-1670	2685°F/2 hrs[6] +3000°F/1 hr	69.9[1][6]	84.2[1]	39.5[1]	9.4[3] 22.8[3][4]	-	-	-	219	213	5-7	7	100	100
															Min 3		90	
	07A-017-01	Rod	3.0" Diameter x 24.0"	65080	3000°F/1 hr	90.9[1]	74.9[1]	32.5[1]	20.1[1][3]	-	-	217BHN	254	233	5.5-6	0-5	40-75	25-100
	SPECIFICATION 01-0035-00-A			-	-	Max 110	Max 100	Min 20	Min 20	-	Flare-15% OD Increase	-	Max 50 VHN Variance		Min 5		100	
	07A-018-(1-63)	Tube[5]	0.625" OD x 0.0065" Wall x 76.5"	111-D-1670	3000°F/1 hr	88.5[1]	74.0[1]	27.5[1]		-	3 at 15% Passed	-	-	-	6.5	6.5	-	100
	07A-050-01	Tube	1.0" OD x 0.100" Wall x 2.0"	111-D-1670	3000°F/1 hr	91.4	74.5	33	38.9[1]	-	-	-	224	217	5	5	100	100
T-222	SPECIFICATION: BEST EFFORT																	
	07A-001-01	Sheet	0.040" x 4.187" x 12.125"	Ta-43-FS-2	None Cold Worked	123.0[3]	112.0[3]	23.5[3]	26.2[1]	28[11]	-	-	-	-	-	-	-	0
ASTAR 811	SPECIFICATION: BEST EFFORT																	
	07A-052-01	Sheet	0.040" x 2.0" x 2.0" x 12.0"	NASV-22	3000°F/1 hr	-	-	-	-	-	-	208DPH[1]	-	-	-	6	-	100
ASTAR 811C	SPECIFICATION: BEST EFFORT																	
	07A-053-01	Sheet	0.042" x 4.0" x 6.250"	NASV-20	None Cold Worked	-	-	-	-	-	-	343DPH	-	-	-	-	-	0
ASTAR 811CN	SPECIFICATION: BEST EFFORT																	
	07A-054-01	Sheet	0.040" x 0.78" x 12.0"	NASV-23	3000°F/1 hr	-	-	-	-	-	-	290DPH[1]	-	-	-	6	-	100
Mo-TZM	SPECIFICATION CMX-WB-TZM-2												Mid-Radius					
				-	-	Min 115	Min 100	Min 18	No Requirement		-	-	Min 260	Max 320	-	-	Stress Relieved	
	07A-021-01	Wire	0.093" Diameter x 6.0'	TZM 7469		146.0[1]	109.7[1]	22[1]	-	-	-	-	293	332	-	-	-	-
	07A-020-01	Rod	0.375" Diameter x 20.0"	TZM 7464	2200°F/1/2 hr	146.8[1]	139.6[1]	24	-	-	-	-	309	319	-	-	-	-
	07A-023-01	Rod	0.50" Diameter x 36.0"	TZM 7495	2250°F/1/2 hr	122.4[1]	115.1[1]	30[1]	-	-	-	-	279	292	-	-	-	-
	07A-051-01	Rod	0.875" Diameter x 5.875"	TZM 7876	2300°F/3/4 hr	122.9[1]	104.1[1]	30[1]	-	-	-	-	279	288	-	-	-	0
W-25Re	SPECIFICATION 01-0057-00-A (Mod)																	
	07A-022-01	Rod	0.125" Diameter x 10.0"	-	2552°F/5 min.	-	-	-	-	-	-	-	-	-	-	10[9]	-	50

TABLE XXIV. MECHANICAL PROPERTIES & GRAIN SIZE OF REFRACTORY ALLOY MILL PRODUCTS (CONT.)

Alloy	MCN Number	Mill Product Form	Mill Product Size	Heat Number	Final Heat Treatment	Room Temperature Tensile Properties Ult. Ksi	0.2%Y.S. Ksi	Elong. %	Stress-Rupture Life Hours	Elong. %	Bend or Flare	Hardness Bulk	Hardness Micro (DPH) Surface	Center	Grain Size Vendor ASTM No.	GE ASTM No.	Recrystallization (%) Vendor	GE
W-25Re	SPECIFICATION: PO 037-284331			-	-	-	-	-	-	-	-	-	-	-	-	-	Stress Relieved	
	07A-031-01	Rod	0.250" Diameter x 6.0"	WC-950	3090°F/10 min.	-	-	-	-	-	-	430	-	-	-	7-8	-	100
	SPECIFICATION: PO 037-271914			-	-	-	-	-	-	-	-	Max 575 DPH	Max 75 DPH Variance		-	-	Max ≤5	-
	07A-030-01	Rod	1.125" Diameter x 7.75"	-	2375°F/1 hr	-	-	-	-	-	-	433(7)	438	435	-	-	<5	5
W-25Re-30Mo (atomic percent)	SPECIFICATION: BEST EFFORT			Batch No.														
	07A-019-(1-5)	Sheet	0.030" x 3.0" x 4.625"	-	2685°F/20 min.	-	-	-	-	-	90° over 2TR	$42R_C$	-	-	-	6	-	100
Re	SPECIFICATION: PO 037-134596																	
	07A-032-01	Plate	0.250" x 0.500" x 1.250"	-	-	-	-	-	-	-	-	239DPH	235	230	-	7	-	100

Alloy	MCN Number	Form	Batch No.	Sintering Data Temp.	Time	Atmosphere	Density >6.10 g/cm³ Vendor	GE	Transverse Rupture	Porosity <ASTM A-4 or B-2 Vendor	GE	Hardness >91.0 R_A Vendor	GE	Grain Size Vendor ASTM No.	GE ASTM No.	Recrystallization (%) Vendor	GE
TiC-10Cb	SPECIFICATION 01-0025-00-B																
	07A-024-(1-10)	Valve Seat	6038	2950°F	30 min.	Vacuum	4.89 g/cm³(8)	4.90 g/cm³(7)	87 x 10^3psi	A-4, 2 spots, B	A-2	$92.4R_A$	1066DPH	-	11	-	-

(1) Average of 2 results.

(2) 2000°F @ 10,000 psi.

(3) 2400°F @ 19 ksi.

(4) Heat treated by GE at 3000°F/1 hr; thermal treatment superimposed over previous thermal condition.

(5) Tubing from MCN 07A-002-01.

(6) Material given an additional 3000°F/1 hr anneal before use.

(7) Average of 4 tests.

(8) Average of all parts.

(9) For recrystallized portions.

(10) Average of 3 tests.

(11) 2400°F @ 30,000 psi - More extensive data reported in "Pilot Production & Evaluation of Tantalum Alloy Sheet", R. L. Ammon, A. M. Fillippi, & D. L. Harrod, April 28, 1964 to October 30, 1965; WANL-PR-M-014 (October 30, 1965).

TABLE XXV. RESULTS OF NONDESTRUCTIVE QUALITY ASSURANCE TESTS OF REFRACTORY ALLOY MILL PRODUCTS AND CERMETS

Alloy	MCN Number	Mill Product		Heat Number	Nondestructive Tests		
		Form	Size		Penetrant	Ultrasonic	Hydrostatic
Cb-1Zr	07A-035-01	Sheet	0.0175" x 12.0" x 19.375"	809950	100% Passed	100% Passed	--
	07A-036-01	Sheet	0.0175" x 12.0" x 19.750"	809950	100% Passed	100% Passed	--
	07A-037-01	Bar	0.5" x 2.0" x 6.0"	912-1211	100% Passed	100% Passed	--
	07A-038-01	Bar	0.5" x 6.0" x 25.0"	912-1211	100% Passed	100% Passed	--
	07A-028-01	Bar	0.5" x 1.0" x 6.0"	5155	100% Passed	100% Passed	--
	07A-028-01	Bar	0.5" x 1.0" x 6.0"	5155	100% Passed	100% Passed	--
	07A-039-02	Rod	0.5" Diameter x 22.0"	911-70559	100% Passed	100% Passed	--
	07A-008-01	Rod	0.625" Diameter x 36.0"	51200	100% Passed	100% Passed	--
	07A-040-02	Rod	1.125" Diameter x 6.0"	5155	100% Passed	100% Passed	--
	07A-041-01	Rod	1.125" Diameter x 6.0"	5155	100% Passed	100% Passed	--
	07A-042-01	Rod	2.0" Diameter x 6.0"	2701	100% Passed	100% Passed	--
	07A-043-01	Rod	2.0" Diameter x 12.0"	5154	100% Passed	100% Passed	--
	07A-044-02	Tube	0.375" OD x 0.065" Wall x 9.0"	98-70546	100% Passed	100% Passed	100% Passed - 4180 psi, 5 sec.
	07A-025-01	Tube	0.375" OD x 0.065" Wall x 78.5"	98-70546	100% Passed	100% Passed	100% Passed - 4180 psi, 5 sec.
	07A-026-01	Tube	0.375" OD x 0.065" Wall x 76.5"	98-70546	100% Passed	100% Passed	100% Passed - 4190 psi, 5 sec.
	07A-027-01	Tube	0.375" OD x 0.065" Wall x 77.0"	98-70546	100% Passed	100% Passed	100% Passed - 4160 psi, 5 sec.
	07A-045-01	Tube	1.0" OD x 0.100" Wall x 7.0"	98-70546	100% Passed	100% Passed	100% Passed - 2600 psi, 5 sec.
	07A-046-01	Tube	1.0" OD x 0.080" Wall x 44.5"	98-70117	--	100% Passed	--
T-111	07A-014-01	Sheet	0.040" x 4.0" x 12.0"	65080	100% Passed	100% Passed	--
	07A-013-01	Sheet	0.040" x 12.0" x 12.0"	65080	100% Passed	100% Passed	--
	07A-012-01	Sheet	0.062" x 2.0" x 9.0"	65080	100% Passed	100% Passed	--
	07A-009-01	Rod	0.500" Diameter x 18.0"	65080	100% Passed	100% Passed	--
	07A-010-01	Rod	0.875" Diameter x 9.0"	65080	100% Passed	100% Passed	--
	07A-011-01	Rod	1.750" Diameter x 12.0"	65080	100% Passed	100% Passed	--
	07A-049-01	Rod	2.0" Diameter x 4.125"	111-D-1102	100% Passed	100% Passed	--
	07A-002-01	Rod	2.0" Diameter x 24.375"	111-D-1670	100% Passed	100% Passed	--
	07A-017-01	Rod	3.0" Diameter x 24.0"	65080	100% Passed	100% Passed	--

TABLE XXV. RESULTS OF NONDESTRUCTIVE QUALITY ASSURANCE TESTS OF REFRACTORY ALLOY MILL PRODUCTS AND CERMETS (CONT.)

Alloy	MCN Number	Mill Product: Form	Mill Product: Size	Heat Number	Nondestructive Tests: Penetrant	Nondestructive Tests: Ultrasonic	Nondestructive Tests: Hydrostatic
T-111	07A-018-(1-63)	Tube	0.625" OD x 0.0065" Wall x 76.5"	--	100% Passed	26 Tubes - 100% Passed 20 Tubes - Defects not rejectable 17 Tubes - Rejectable (5% Criteria)	--
	07A-050-01	Tube	1.0" OD x 0.100" Wall x 2.0"	111-D-1670	100% Passed	100% Passed	Passed
Mo-TZM	07A-021-01	Wire	0.093" Diameter x 6.0'	TZM 7460	100% Passed	--	--
	07A-020-01	Rod	0.375" Diameter x 20.0"	TZM 7464	100% Passed	--	--
	07A-023-01	Rod	0.50" Diameter x 36.0"	TZM 7496	100% Passed	100% Passed	--
	07A-061-01	Rod	0.875" Diameter x 5.875"	TZM 7876	100% Passed	100% Passed	--
W-25Re	07A-030-01	Rod	1.125" Diameter x 7.75"	--	100% Passed	100% Passed	--
Re	07A-033-(1-3)	Seat	Hoke Drawing 474-130	--	100% Passed	--	--
TiC-10Cb	07A-024-(1-10)	Seat	Hoke Drawing 474-130	--	100% Passed	--	--

TABLE XXVI. SUMMARY OF OVERALL QUALITY ASSURANCE TEST RESULTS

Material	Form	Number of Lots	Chemistry		Tensile Properties		Stress-Rupture		Hardness		Grain Size		Penetrant*		Ultrasonic*		Hydrostatic*		Flare		Bend	
			Passed	Failed	Passed	Failed	Passed	Failed	Passed	Failed	Passed	Failed	Passed	Failed	Passed	Failed	Passed	Failed	Passed	Failed	Passed	Failed
Cb-1Zr	Foil	1	1	0	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	1	0
	Sheet	3	3	0	3	0	-	-	2	0	2	0	2	0	2	0	-	-	-	-	1	0
	Bar	4	4	0	4	0	4	0	1	0	3	0	4	0	4	0	-	-	-	-	-	-
	Wire	2	0	2	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
	Rod	7	7	0	5	0	1	1	5	0	6	0	6	0	6	0	-	-	-	-	-	-
	Tube	6	6	0	6	0	5	0	6	0	4	0	5	0	6	0	5	0	6	0	-	-
T-111	Foil	2	2	0	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	2	0
	Sheet	3	3	0	3	0	3	0	3	0	3	0	3	0	3	0	-	-	-	-	3	0
	Wire	2	2	0	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
	Rod	6	6	0	6	0	6	0	6	0	5	1	6	0	6	0	-	-	-	-	-	-
	Tube	2	2	0	2	0	1	0	1	0	2	0	64	0	47	17	1	0	2	0	-	-
Mo-TZM	Wire	1	1	0	1	0	-	-	1	0	1	0	1	0	-	-	-	-	-	-	-	-
	Rod	3	3	0	3	0	-	-	3	0	3	0	3	0	2	0	-	-	-	-	-	-
W-25Re	Rod	3	3	0	-	-	-	-	1	0	2	1	1	0	1	0	-	-	-	-	-	-
Re	Plate	1	1	0	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
	Valve Seats	1	1	0	-	-	-	-	-	-	-	-	3	0	-	-	-	-	-	-	-	-
TiC-10Cb	Valve Seats	1	1	0	-	-	-	-	1	0	1	0	10	0	-	-	-	-	-	-	-	-

* Total Number of Pieces Tested

The failure of the densities of the TiC-10Cb cermet valve seats (MCN 07A-024-(1-10) to meet the specification requirements was found by further study to be the result of too stringent density requirements in the specification. The density requirement in the specification was determined from very limited data and the specimens from which this data was obtained were found to contain sufficient tungsten to effect the density measurements. The more recent TiC-10Cb cermet valve seats had a much lower tungsten content and therefore a lower density. A comparison of the measured bulk density of the valve seats with a theoretical density, determination by x-ray diffraction, showed the density of the valve seats to be 95.3% of the theoretical density. This would generally be considered an acceptable density for this type of material. However, the inherently high oxygen content of the TiC-10Cb cermet valve seats resulted in the necessity to replace them by unalloyed rhenium (MCN 07A-033-(1-3). The TiC-10Cb valve seats with high oxygen content were found to undergo significant corrosion when placed in contact with lithium at elevated temperatures.*

*Appendix A

APPENDIX D

BRITTLE BEHAVIOR OBSERVED IN T-111 PIPING REMOVED FROM THE VALVE LOOP FOLLOWING TESTING

In addition to the planned posttest evaluation of the Valve Loop, a number of T-111 tubing segments were removed from the valve assembly and flattened to qualitatively measure their ductility. It was surprising to find that all the T-111 specimens were brittle and cracked with very little deformation of the tube segments.

The three specimens tested were removed from the valve assembly as shown in Figure 72. Specimen A was a half ring segment containing the Cb-1Zr to T-111 weld transition joint. Specimen B was a full ring tubing segment at the metering valve inlet. Specimen C was also a full ring tubing segment removed from the piping between the metering valve and the isolation valve.

The T-111/Cb-1Zr weld joint specimen is shown before and after flattening in Figure 73. Cracking occurred almost immediately upon loading and, as can be seen in Figure 73, only occurred in the T-111. The crack stopped at the Cb-1Zr weld nugget.

The tube specimen from the metering valve inlet cracked in three pieces, as shown in Figure 74, again with very little deformation. It was difficult to ascertain as to whether the cracks initiated at the tubing ID or OD.

The tube specimen from the piping between the valves cracked as shown in Figure 75. This specimen deformed to a greater degree before cracking. Closer examination of the tube end indicated the cracks initiate at both the ID and OD where the tensile forces are greatest during flattening.

The cause of this brittle behavior is unknown although it is suspected to be associated with an aging reaction. All the T-111 specimens had been heated at 1900^{o}F for 3500 hours in the loop test. Additional evaluation would need to be performed to elucidate the cause of this brittle behavior.

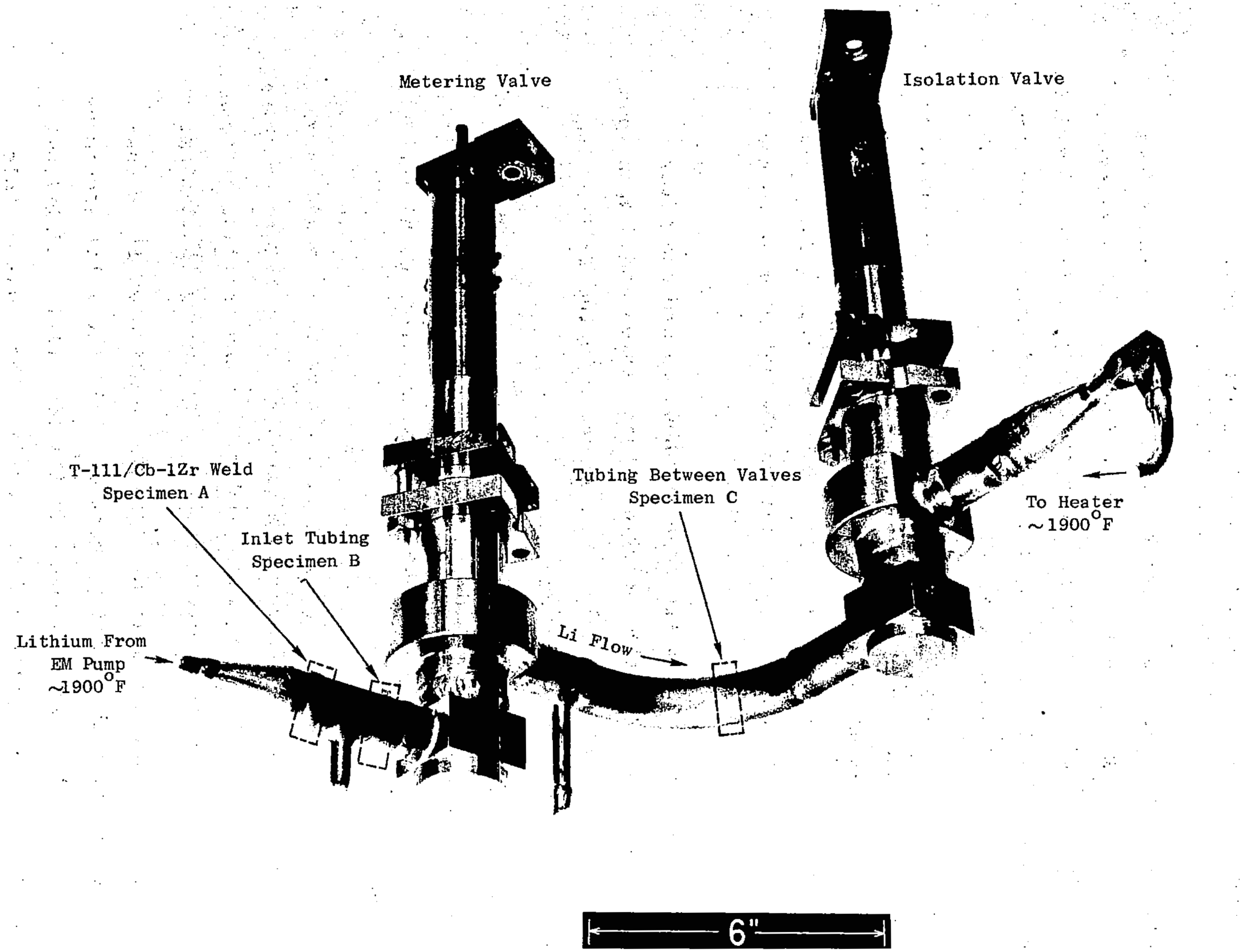

Figure 72. Metering and Isolation Valve Assembly As-Removed from the Loop Upon Completion of 5000 Hours of Testing Showing Location of Specimens Removed for Bend Testing. (Orig. P69-7-42C)

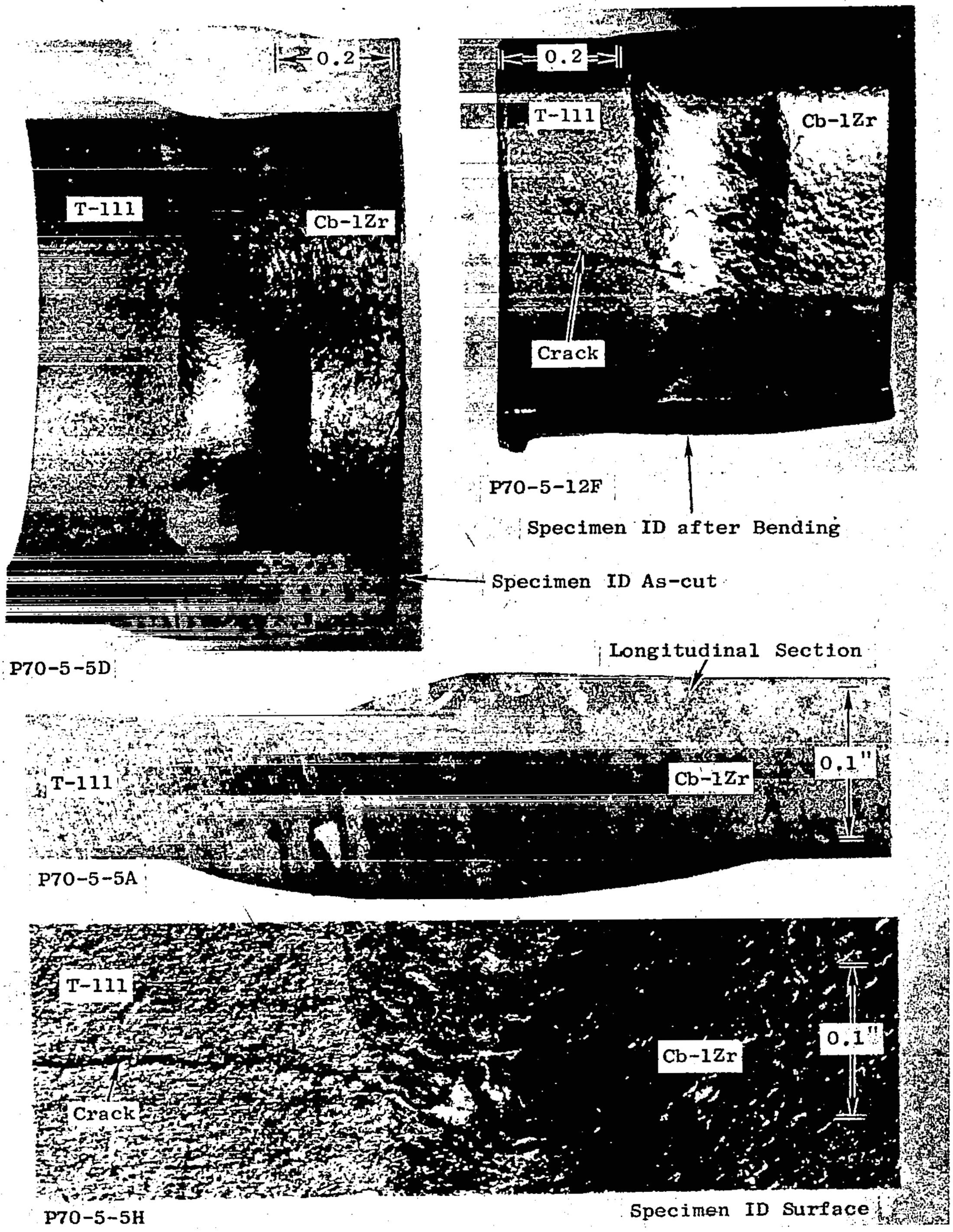

Figure 73. T-111/Cb-1Zr Transition Weld Showing a Crack in the T-111 as a Result of Flattening After 5000 Hours of Testing in the High Temperature Alkali Metal Valve Loop. (Specimen A - Figure 1)

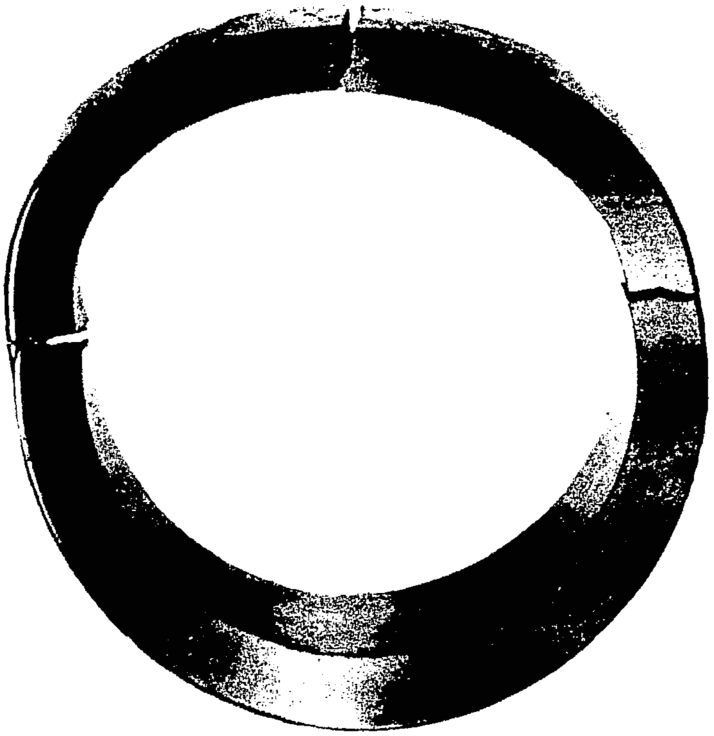

Figure 74. T-111 Tubing from the Metering Valve Inlet Which Cracked as a Result of Flattening After 5000 Hours of Testing in the High Temperature Alkali Metal Valve Loop. (Specimen B - Figure 1) (P70-6-4C)

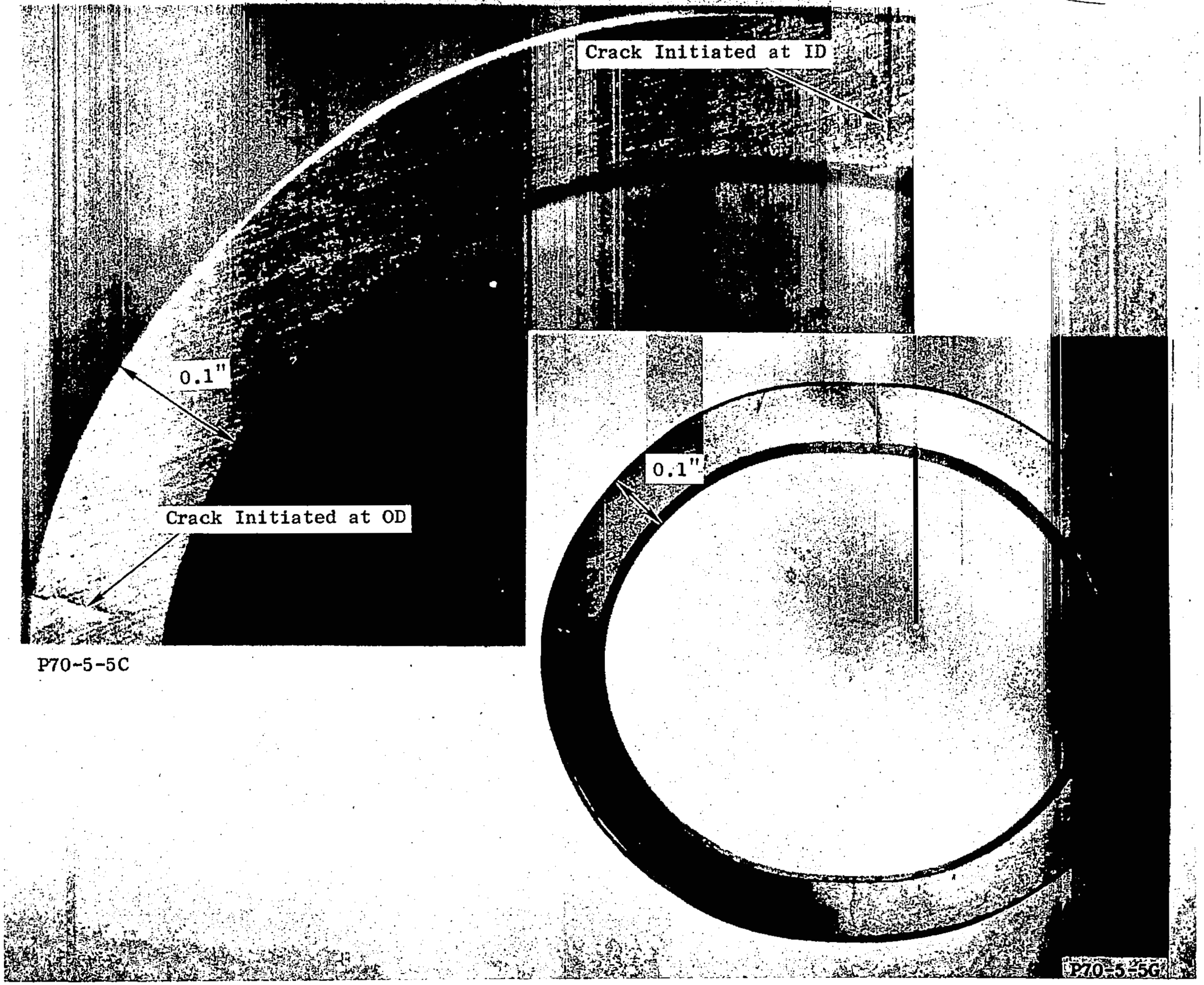

Figure 75. T-111 Tubing from the Line Between the Metering and Isolation Valves Which Cracked as a Result of Flattening After 5000 Hours of Testing in the High Temperature Alkali Metal Valve Loop.