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POTASSIUM CORROSION TEST LOOP DEVELOPMENT

Quarterly Progress Report No. 4
For Quarter Ending July 15, 1964

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MISSILE AND SPACE DIVISION
GENERAL ELECTRIC
CINCINNATI, OHIO 45215

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POTASSIUM CORROSION TEST LOOP DEVELOPMENT

QUARTERLY PROGRESS REPORT 4

Covering the Period

April 15, 1964 through July 15, 1964

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Lewis Research Center

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SPACE POWER AND PROPULSION SECTION
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POTASSIUM CORROSION TEST LOOP DEVELOPMENT

I INTRODUCTION

This report covers the period, from July 15, 1964 to August 15, 1964, of a program to develop a prototype corrosion test loop for the evluation of refractory alloys in boiling and condensing potassium environments which simulate projected space electric power systems. The envisioned prototype test consists of a two-loop Cb-1Zr facility; sodium will be heated by direct resistance in a primary loop and will be used in a heat exchanger to boil potassium in the secondary, corrosion test loop. Heat rejection for condensation in the secondary loop will be accomplished by radiation in a high vacuum environment. The immediate corrosion test design conditions are shown below; it is expected that the temperature could be increased by about 400°F when testing is extended to include refractory alloys stronger than Cb-1Zr.

1. Boiling temperature, 1900°F
2. Superheat temperature, 2000°F
3. Condensing temperature, 1350°F
4. Subcooling temperature, 800°F
5. Mass flow rate, 20 to 40 lb/hr
6. Vapor velocity, 100 to 150 ft/sec
7. Average heat flux in the potassium boiler - 50,000 to 100,000 BTU/hr ft^2

The development program is proceeding with the construction and operation of three Cb-1Zr test loops, each of which will be used in a sequence of component evaluation and endurance testing. Loop I, a natural convection loop, has been operated for 1,000 hours with liquid sodium at a maximum temperature of 2260° to 2380°F to evaluate the electrical power vacuum feedthroughs, thermocouples, the method of attaching the electrodes, the electrical resistivity characteristics of the heater segment, and the use of thermal and electrical insulation. Construction of Loop II, a single-phase, forced-circulation loop which will be operated for 2,500 hours with liquid sodium at about

2000°F to evaluate the primary loop EM pump, a flowmeter, flow control and isolation valves, and pressure transducers, has been completed. This loop had operated for 310 hours at the end of the current reporting period. The Pre-prototype Corrosion Test Loop, a two-loop system design of which has been completed will include a boiler, turbine simulator, and condenser in addition to the above components. This facility will be used to develop and endurance test (2,500 hours) the components required to achieve stable operation at the corrosion test design conditions.

The quarterly reports issued for this program will summarize the status of the work with respect to design considerations, construction procedures, and test results. The topical report on Loop I is expected to be released in February, 1965. Detailed topical reports will also be issued to describe each test loop. Additional topical reports will be prepared to cover such areas as materials specifications, purification of potassium and sodium, and inert gas purification and analysis.

II PROGRAM STATUS

1. Component Evaluation Test Loop I

The termination and general performance of the component Evaluation Test Loop I was reported in the previous progress report of this program[1]. A schematic of the loop and the operating conditions are shown in Figure 1.

During the disassembly of the loop from the test support structure, samples of the 2-mil Cb-1Zr foil taken from reflective insulation from all regions were checked for ductility and appearance change. All of the foil specimens were comparable in ductility and appearance to the before-test foil.

Following removal from the test chamber, the sodium was drained from the loop by inverting it and admitting the sodium to a previously evacuated stainless steel storage container. The loop was heated with heating tapes to a temperature of 350°F (maximum) during the draining operation. Aluminum foil was used to prevent direct contact between the heating tapes and the loop.

The loop was cut into sections 6 to 8 inches in length and stripped of residual sodium in absolute ethyl alcohol. The stripping solution for each section of tubing was carefully filtered in an effort to collect any mass transfer crystals which might be present. No deposits were evident on any of the loop sections and only several very small metallic particles were recovered from the solutions during filtering. These particles were examined at 30X and their appearance indicated that they might be mass transfer crystals.

Eighteen metallographic specimens were taken from the loop. Hardness surveys were performed on four of the metallographic specimens. Seven tube sections were also cut from various regions of the loop and flattened at room temperature to determine the post-test ductility. The bend specimens were also examined metallographically. Chemical analyses were performed on five specimens to determine changes in interstitial element concentrations. The results of these examinations are

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[1] Potassium Corrosion Test Loop Development, Quarterly Progress Report #3, Covering the Period January 15, 1964 to April 15, 1964, NASA Contract NAS 3-2547, NASA-CR-54081.

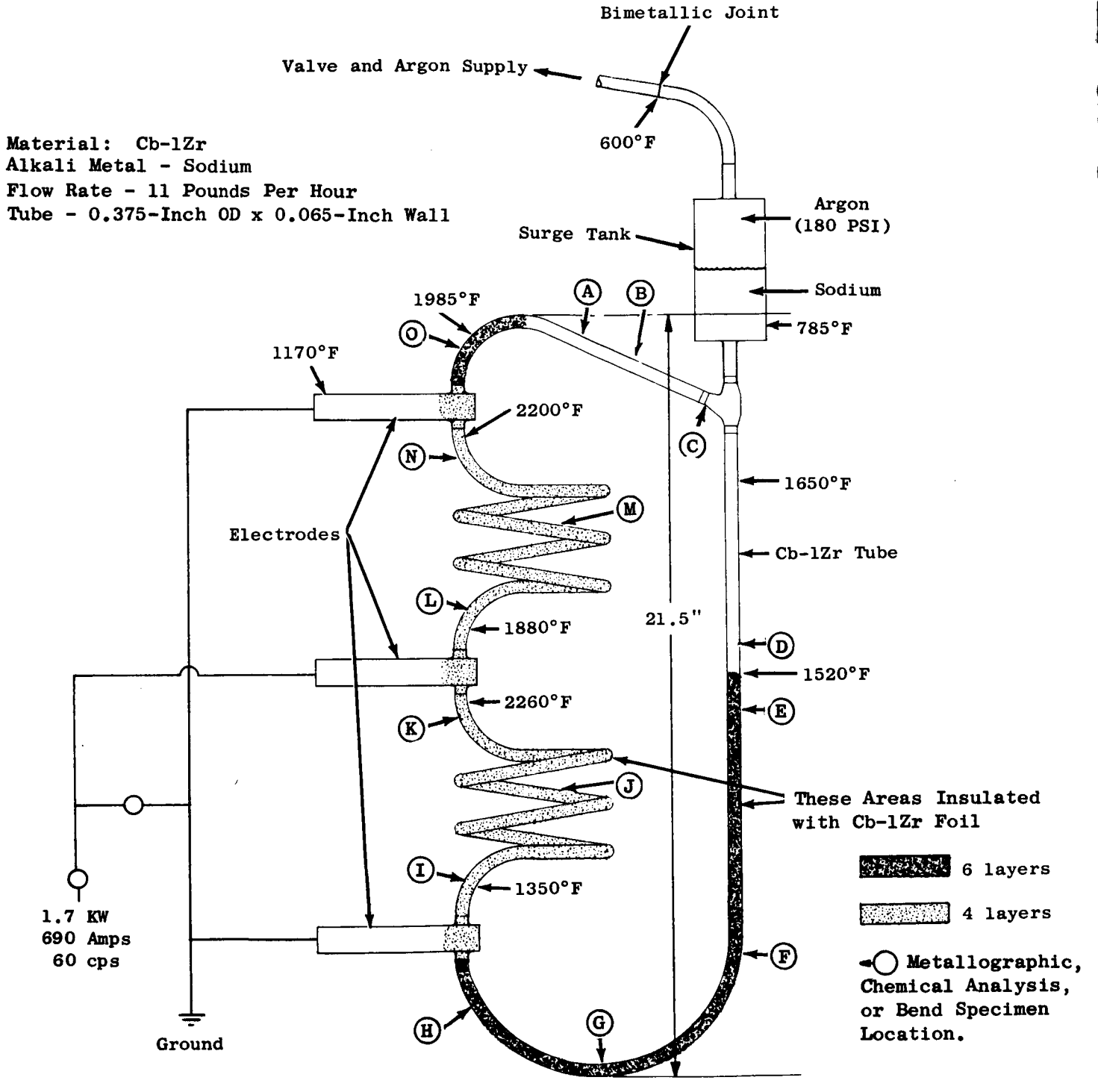


Figure 1. Component Evaluation Test Loop I. - (0800, 31 March 1964)

discussed briefly below and will be covered in more detail in the topical report on this test to be released in February, 1965.

The phenomenon of strain induced grain growth observed in the lot of Cb-1Zr tubing used to fabricate Loop I was discussed in an earlier progress report[2]. Metallographic examination of the 25 specimens from the loop revealed varying degrees of excessive grain growth in all sections of the loop which were bent during fabrication and operated in excess of 1900°F during the loop test. The typical appearance of the tubing in the hottest regions (2200°-2260°F) of the heater coils is illustrated in Figure 2. In these regions many of the grains had diameters of approximately 25 mils compared to before test diameters of approximately 1 mil. This micrograph reveals the slightly irregular inner surface of the tube wall which was comparable to the condition of the surface before test. Figure 3 shows the tube wall from the heater exit region and exhibits the duplex grain size found in many regions of the loop which operated in the 1950° to 2100°F temperature range.

The coldest portion of the loop was in the heater inlet region (1350°F) and the metallographic appearance of this region is shown in Figure 4. The slightly darker band near the ID surface was confirmed by repeated polishing and etching and is due to the presence of a very fine precipitate in this region. Chemical analysis (Table I) of the total cross section of the tube wall in this location (Region H) did not reveal any marked shift in the oxygen and nitrogen concentration; however, the carbon concentration was slightly higher than in the before test material and the hydrogen content was substantially greater. Results of a hardness survey in this darkened area in Figure 4 revealed a Knoop hardness of 115 (average) as compared to 95 (average) in the same zone of other tube sections.

The post-test ductility of 1/2-inch long tube sections taken from eight regions of the loop was checked by flattening the tubes using a strip of 0.060-inch thick sheet as an internal support. Five of the specimens were found to be quite ductile, but duplicate specimens taken from Region O (Figure 1) and one specimen from Region A cracked in an extremely brittle and transgranular fashion. All of the bend specimens were examined metallographically and cross sections of three of

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[2] Potassium Corrosion Test Loop Development, Quarterly Progress Report #3, Covering the Period January 15, 1964 to April 15, 1964, NASA Contract NAS 3-2547, NASA-CR-54081, pp 40-48.

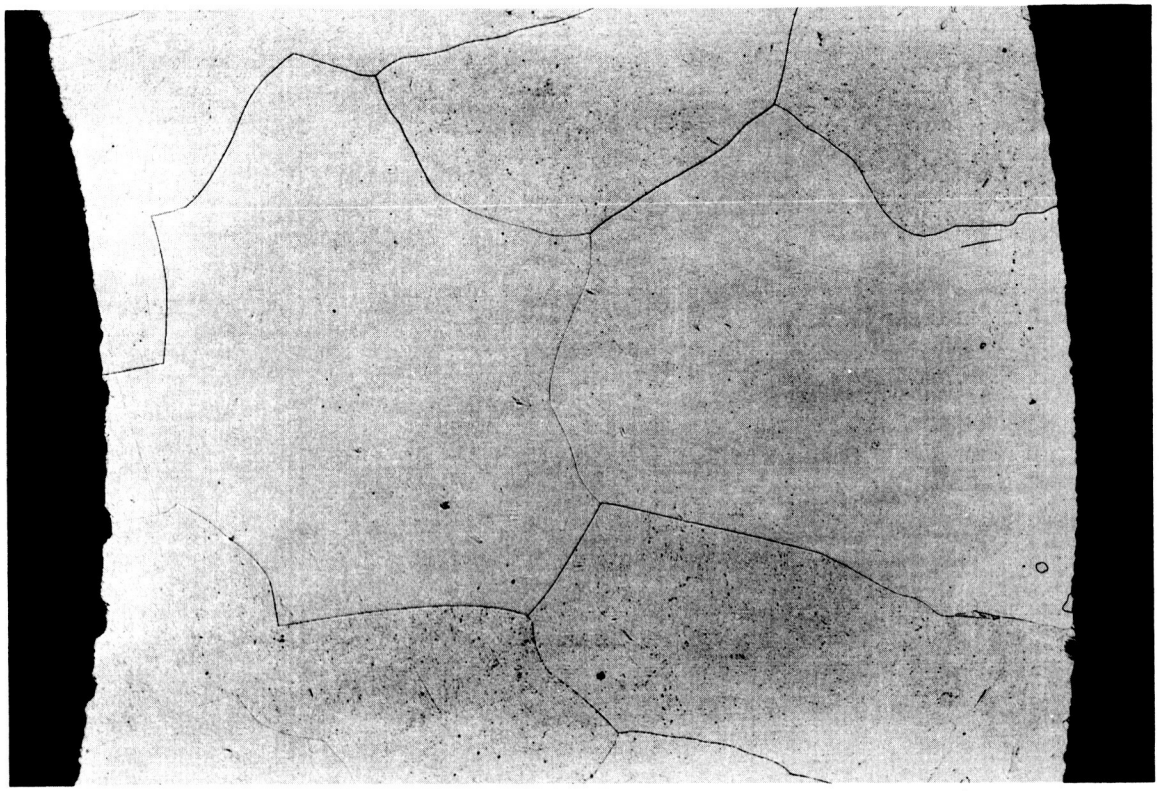


Figure 2. Micrograph of a Cross Section of Loop I Tubing from the Middle of the Top Heater Coil (Region M in Figure 1).

Etchant: 60%Glycerine-20%HF-20%HNO_3 Mag. 75X (K4192)

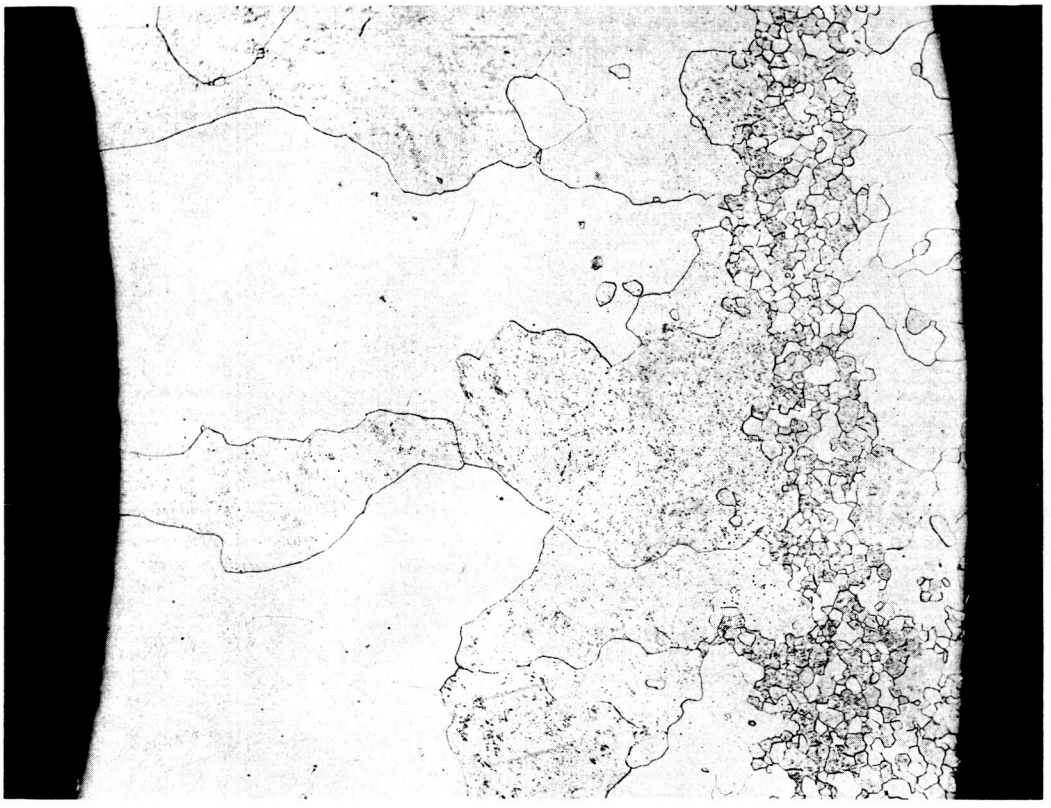


Figure 3. Micrograph of a Cross Section of Loop I Tubing from the Heater Exit (Region O in Figure 1).

Etchant: 60%Glycerine-20%HF-20%HNO_3 Mag. 75X (K3832)

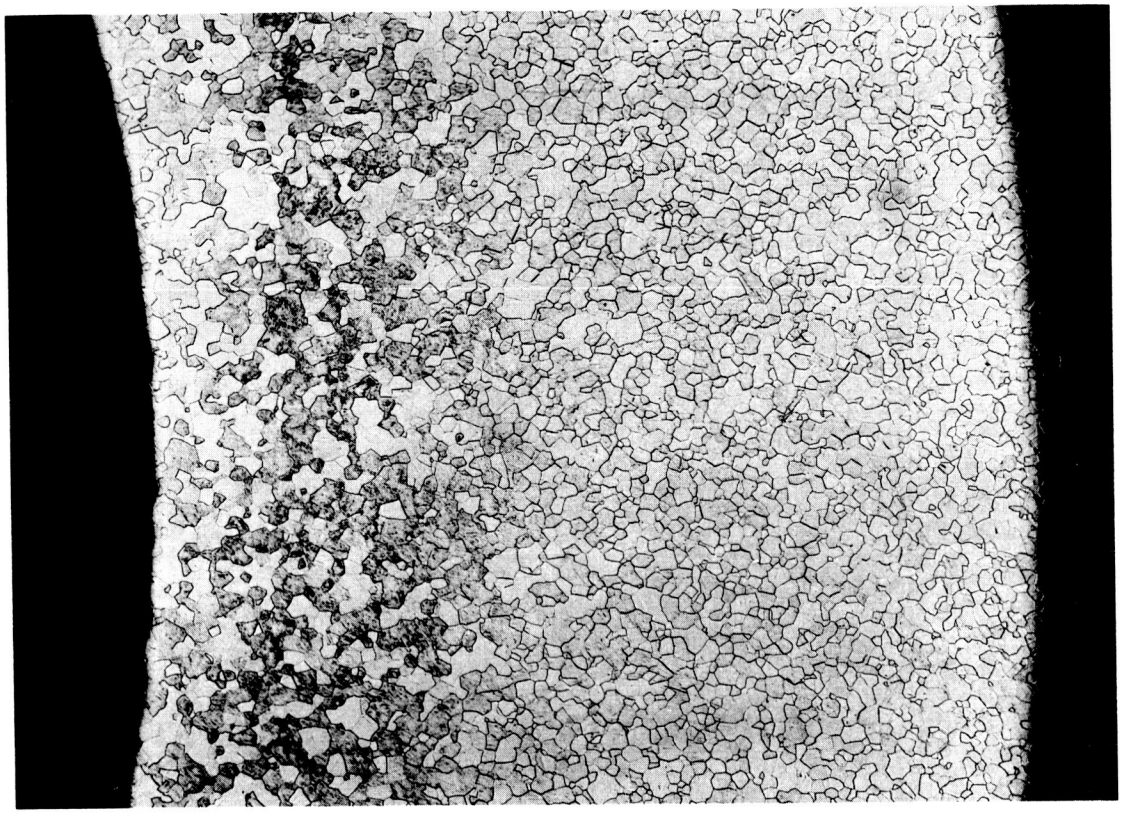


Figure 4. Micrograph of a Cross Section of Loop I Tubing from the Cold Return Line Below Bottom Heater Coil (Region H in Figure 1).

Etchant: 60%Glycerine-20%HF-20%HNO_3 Mag. 75X (K3829)

TABLE I

CHEMICAL ANALYSES OF VARIOUS Cb-1Zr TUBE

SPECIMENS FROM COMPONENT EVALUATION LOOP I

Specimen Description	Concentration, ppm O	N	H	C
Region A*				
Outer 21 Mils**	310	11	50	-
Middle 21 Mils	171	5	79	-
Inner 21 Mils	199	5	62	-
Region H				
Total Cross Section	190	3	37	40,50
Region M				
Total Cross Section	69	3	13	-
Region O				
Total Cross Section	194,216	14,16	173,174	-
Before Test (MCN 404)	114,136	1,9	1,4	20,30

* Location Given in Figure 1.

** Outer Surface Grit Blasted with Alumina Prior to Test.

these are shown in Figure 5. These specimens have been heavily etched to show the grain structure. Figure 5(a) is the fine grained specimen from the coolest part of the loop. The dark band near the inner surface is the same region as that shown at higher magnification in Figure 4. No cracks were detected in this specimen. Figure 5(b) shows the specimens from near the top of the heater coil. This region was bent in forming the heater coil and operated 2200°F during the test. All the grains in this specimen were of the order of 15-25 mils in diameter. No cracks were observed. The specimens taken from the heater exit area (Region O) were brittle as shown in Figure 5(c). This fracture is shown at higher magnification in Figure 6. The hydrogen concentration of this region was quite high and is discussed in more detail below. The brittle transgranular fracture may possibly be due to an intragranular segregation of hydrogen which might normally be concentrated in the grain boundaries of fine-grained material. No data were sought or obtained to support this hypothesis. It may be noted that although the fracture did propagate through the fine-grained region near the outer surface of the surface, there was evidence of some strain in this region. These results demonstrate the importance of testing different heats of Cb-1Zr and other refractory alloys to determine the susceptibility to and resultant effects of strain induced grain growth.

The chemical results obtained on specimens from several regions of the loop are given in Table I. The oxygen analyses listed show little if any pickup from the test chamber environment. The relatively high oxygen concentration of the outer layer of Region A is attributed to the alumina grit blasting of the surface of the tube to raise the emittance. Preliminary results of an alumina grit blasting experiment on Cb-1Zr sheet indicate that an oxygen increase of several hundred ppm can be expected in tubing with wall thickness of 0.065-inch. The oxygen is in the form of imbedded Al_2O_3 particles on the surface. There is the possibility of the Al_2O_3 reacting with Cb-1Zr at elevated temperatures but there was no evidence of such a reaction between the Al_2O_3 thermocouple insulation and the Cb-1Zr tubing in Loop I at temperatures up to 2250°F. The relatively low oxygen concentrations of the specimens from the upper heater coil (Region M) suggests a possible leaching of oxygen from this hot region. Additional wall gradient analyses would be required to confirm this.

No increase or migration of nitrogen in the various specimens was indicated by the analyses. As mentioned above when discussing the results of metallographic examination, the specimen from the coldest section of the Loop (Region H) showed evidence of a fine precipitate near the inner surface and this may possibly be associated with the slightly

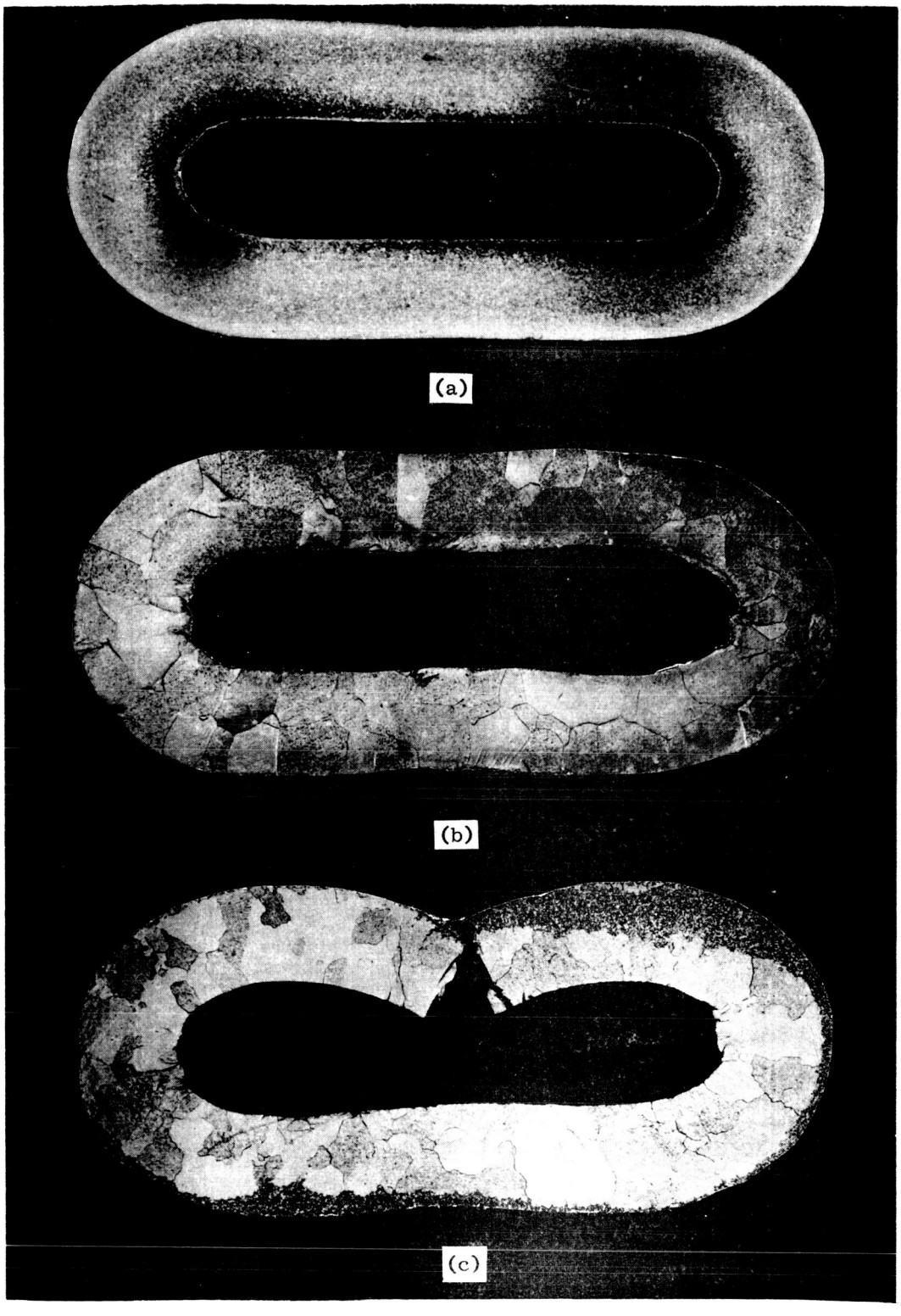


Figure 5. Micrographs of 0.375-Inch OD x 0.065-Inch Wall Cb-1Zr Tubing from Loop I. Specimens were Flattened at Room Temperature Following Test. Specimen Locations Given in Figure 1.

a) Heater Inlet, 1350°F (Region H) Mag. 10X (C64090209)
b) Heater Coil, 2200°F (Region N) Mag. 10X (C64090210)
c) Heater Exit, 1985°F (Region O) Mag. 10X (C64081402)

Etchant: 60%Glycerine-20%HF-20%HNO_3

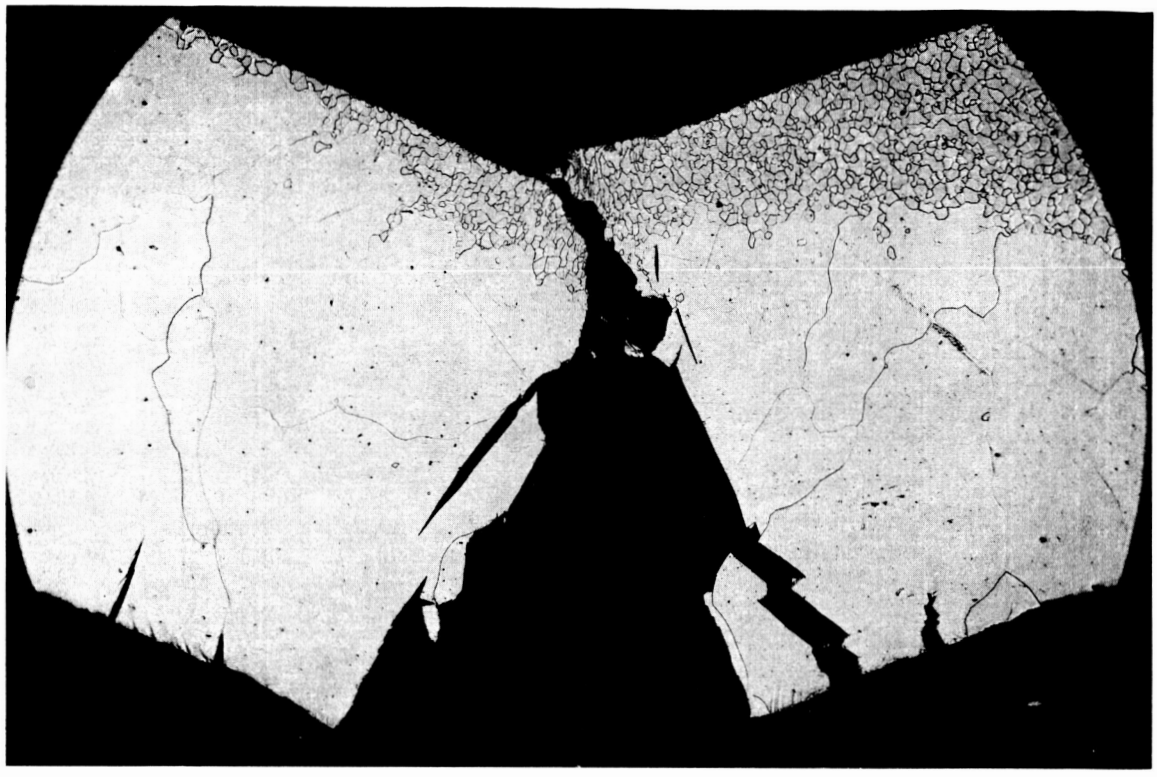


Figure 6. Micrograph of a Cross Section of the Loop I Tube Wall from Heater Exit (Region O in Figure 1).

Etchant: 60%Glycerine-20%HF-20%HNO_3 Mag. 50X (K3825)

higher carbon concentration of the specimen from this region compared to the before test analysis. Such a conclusion, however, is not strongly supported by the limited carbon analyses which were obtained.

The hydrogen concentration of the specimens from the loop are much higher than would be expected. It has been suggested by other investigators that hydrogen pickup by columbium alloys can occur during the removal of residual alkali metal from tubing when ethyl alcohol is used as the solvent. An analysis was performed on a tube specimen from the top heater (Region M). This section of the loop required several days of soaking in alcohol and alcohol plus water solutions to remove a considerable quantity of sodium which remained in the coil after draining the loop. The hydrogen analysis of this section was found to be 13 ppm as indicated in Table I. Specimens from the heater inlet area (Region H) and the heater exit area (Regions A and O) were practically free of sodium when the loop was sectioned and were completely reacted after several minutes in the alcohol solution. These samples had the considerable higher hydrogen concentrations than the specimen from the heater coil described above. In summary, the source of hydrogen and the variations found in various regions of the loop is not understood at the present time. Several additional analyses will be performed to determine if the hydrogen concentration is related to the brittle behavior of the specimens from Region O. McCoy and Douglas[3] have stated that "hydrogen concentrations in excess of 500 ppm seriously embrittle columbium at room temperature" but no data have been located regarding the effect of hydrogen concentration on the embrittlement of Cb-1Zr. A hardness survey on the tube wall in the brittle region of the loop showed that the entire wall had a Knoop hardness which varied between 88 and 100, which is comparable to the hardness (97) of the recrystallized tubing prior to test.

2. Loop II Fabrication

The fabrication of Loop II was completed during this report period. Several components, such as the EM pump duct, stressed diaphragm pressure transducer and slack diaphragm pressure transducer, were also completed and assembled on the loop.

---

[3] McCoy, H. E. and Douglas, D. A., "Effect of Various Gaseous Contaminants on the Strength and Formability of Columbium," Columbium Metallurgy, Interscience Publishers, New York, 1961, p 112.

The fabrication of the EM pump duct presented some difficulty when the first attempt to produce the interference fit between the finned duct and outer duct was unsuccessful. The procedure used involved heating of the outer duct to 1200°F in high-purity helium. After temperature stabilization, the cool finned duct was inserted, using a "vee" fixture for alignment. Although this procedure was successful in previous instances, in this case seizing of the finned duct occurred at approximately the mid-point of the outer duct.

Subsequently, the outer duct was cut off at the end of the interference fit, re-machined for weld preparation, and a second interference fit attempted from the other end of the finned duct. For this interference fit, the one-half length of the outer duct was heated to 500°F in air while the finned duct was immersed in liquid nitrogen. No difficulty was encountered in producing the interference fit using this procedure.

One additional circumferential weld at the mid-point of the EM pump duct was then required. Since this weld was in contact with the finned duct, special precautions were taken to avoid any blockage in the alkali metal flow channel. The root weld pass was radiographed extensively with no indication of excessive weld drop-through. An air flow pressure drop test was then made which indicated satisfactory performance. This test was repeated after the final weld passes and the results of this test showed an insignificant (less than 3%) increase in the overall pressure drop.

The stressed diaphragm transducer has an 0.015-inch thick T-111 alloy (Ta-8W-2Hf) diaphragm for pressure sensing. This diaphragm is welded to a T-111 alloy housing which is, in turn, welded to a Cb-1Zr alloy body and process tube. A T-111 alloy probe mount is welded to the center of the diaphragm. The magnetic probe assembly is then brazed to the probe mount to complete the assembly.

Two sets of hardware were processed to develop the electron beam welding technique required for this assembly. The electron beam welds for the diaphragm assembly were produced without difficulty. However, the joint between the T-111 alloy housing and the Cb-1Zr alloy resulted in cracking of the T-111 alloy. This cracking was attributed to the highly localized heat input of the electron beam in combination with a slight joint gap introduced by metal removal during the pickling operation.

To eliminate this problem, the tungsten-inert gas welding process was used to produce this joint on the second set of hardware. As an added precaution, an electron beam weld was made between these components on the outside diameter, thus effecting a double seal between the T-111 and the Cb-1Zr alloy components.

The electron beam welds between the T-111 alloy probe mount and diaphragm, and between the diaphragm to housing were repeated on the second set of hardware without difficulty. The chemical analyses of weld metal specimens obtained before and after each electron beam weld sequence are reported in Table II per specification SPPS-14.

The final fabrication step was the brazing of the magnetic probe assembly to the probe mount. The configuration was a socket joint in which an 0.015-inch diameter stainless steel wire from the magnetic probe was inserted into a suitable hole in the T-111 alloy probe mount. Brazing was accomplished using localized heating with a graphite-tipped, heater probe under high-purity helium in the welding chamber. The brazing alloy selected was Premabraz-615 (61.5Ag-24Cu-14.5In). This alloy was applied to the joint manually in wire form. The completed assembly was mass spectrometer leak tested successfully and submitted to instrumentation for room temperature calibration. In the future, the probe wire will be joined to the diaphragm mount by electron beam welding, rather than brazing, to increase the temperature capability of this joint.

The assembly of Loop II proceeded in the following sequence: 1) the heater coil sub-assembly, Figure 7, which included the heater coil portion of the loop plus the section containing the metering valve was fabricated, 2) the surge tank sub-assembly which included the drain/fill valve and the isolation valve was fabricated, 3) these sub-assemblies were post-weld heat treated at the Stellite Division of the Union Carbide Corporation, 4) the stressed-diaphragm transducer process tube was welded in place and post-weld heat treated, 5) the heater coil and surge tank sub-assemblies were joined, 6) the EM pump duct was welded in place, and 7) the slack-diaphragm transducer was welded to the loop. The joining of the latter two components was accomplished in the eight-foot diameter welding extension chamber after integration of the loop, vacuum chamber spool section, and support structure. All the post-weld anneals were conducted in the welding vacuum chamber, using a small refractory metal heater which provided localized heating of the welded joint. A typical heat treatment is illustrated in Figure 8. The refractory metal heater in this case was positioned around the welds

TABLE II

RESULTS OF CHEMICAL ANALYSES OF Cb-1Zr ELECTRON BEAM WELDMENTS[a] PER SPECIFICATION SPPS-14

	Element, ppm			
	O	N	H	C
Base Metal Analyses by Vendor MCN-417	130	85	1	50
Base Metal Analyses by GE MCN-417	142	22	2	30,40
T-111 Housing to Cb-1Zr Retainer Body (Process Sheet EB-4)				
Sample Weld[b] Before	107	53	3	40
Sample Weld After	102	50	3	30
T-111 Diaphragm to Probe Mount (Process Sheet EB-5)				
Sample Weld Before	142	47	7	30
Sample Weld After	108	51	2	40

a All Welds Made at a Pressure of 5×10^{-5} Torr.

b Welds Analyses are on Welds Made on the Base Material Listed Above, Before and After Welding of the Components Listed.

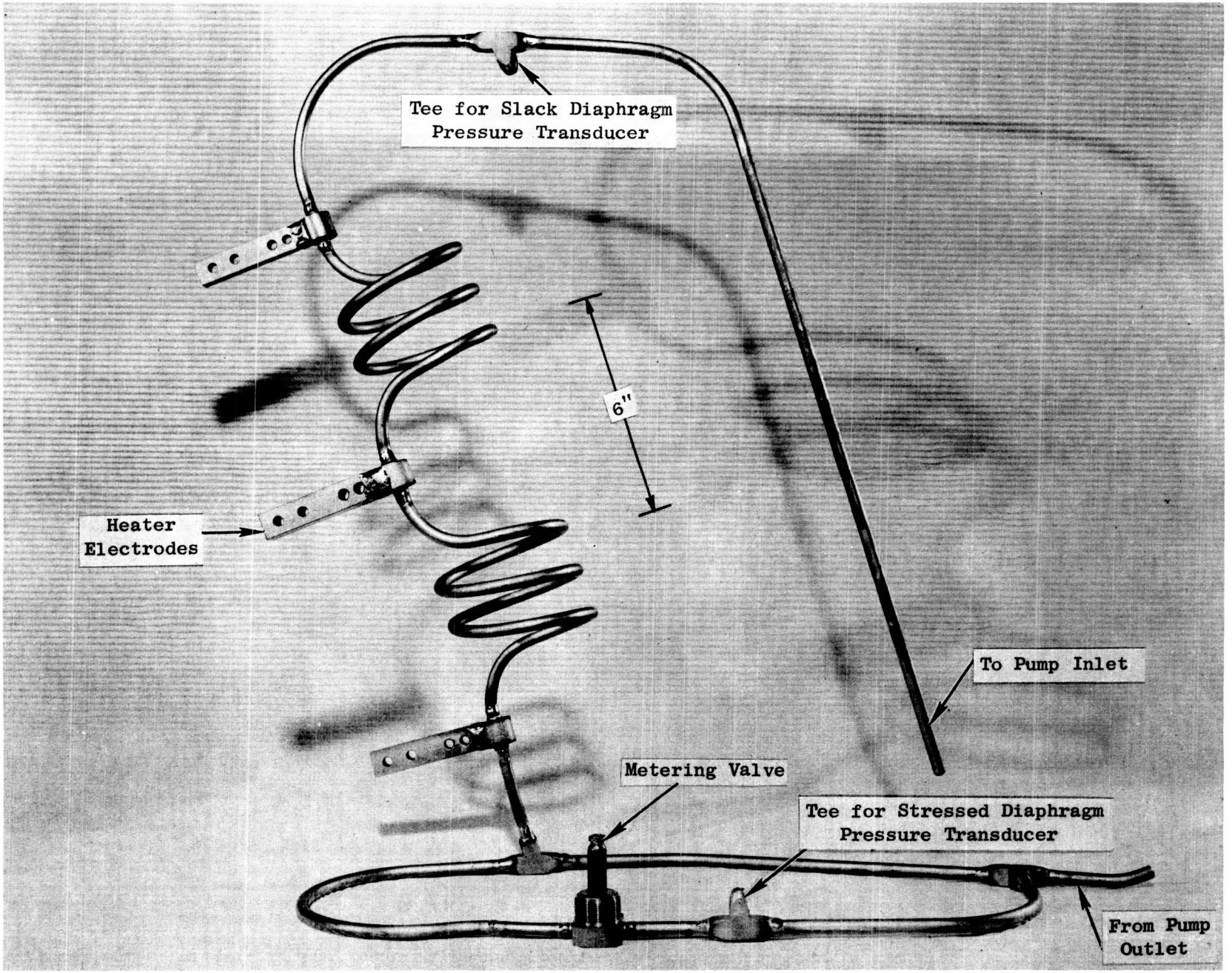


Figure 7. Loop II Heater Coil Sub-Assembly (Original - C64042329).

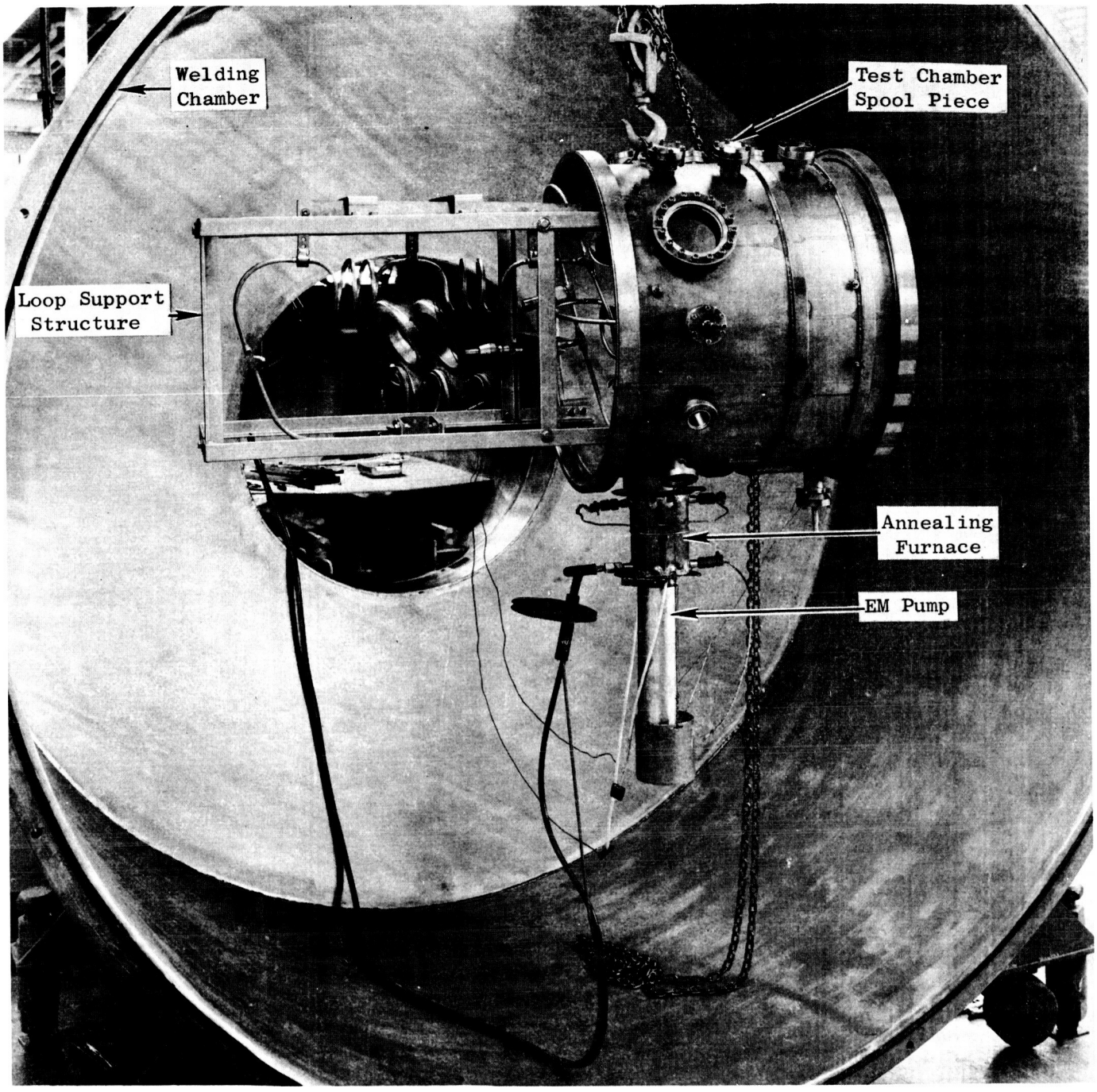


Figure 8. Loop II During Final Assembly in the Vacuum Purge Welding Chamber Extension. The Refractory Metal Furnace Used to Anneal Loop Weldments is Shown in Position for Heat Treatment of the EM Pump Duct Attachment Welds. (Original - C64060134)

attaching the EM pump duct to the loop. Power for the heater was supplied by the welding torch and the temperature was monitored by two Pt vs Pt+10%Rh thermocouples attached to the weld.

The stainless steel vacuum components were added to the spool section of the vacuum chamber in the final assembly operation. Two vacuum seal welds were required on the entry ports for the sodium fill line and inert gas line. The outer can for the EM pump duct and its support flange were also welded in position.

Welding of the Cb-1Zr alloy component required for final assembly resulted in several minor deviations from Specification SPPS-3C. Although the welding chamber with the extension attached has been previously evacuated to less than 1×10^{-5} torr, the addition of the spool section resulted in slower pumping speeds and slightly higher ultimate vacua. Back-filling with purified helium was, therefore, conducted after evacuation to 1.9×10^{-5} torr and 2.6×10^{-5} torr for the EM pump and slack diaphragm pressure transducer welds, respectively. Another deviation occurred during heat treatment of the EM pump duct attachment welds. In this case the heater shorted externally after operation for fifteen minutes at 2200°F. Review of the aging reactions of Cb-1Zr weldments indicated this time at temperature was sufficient to avoid subsequent aging reactions. Therefore, no further annealing was performed.

The qualification of the vacuum furnaces used for post-weld annealing treatments was conducted in accordance with Specification SPPS-3C. Results of chemical analyses are reported in Table III. Bend tests performed in accordance with Specification SPPS-3C indicated full bend ductility in all cases.

A deviation from Specification SPPS-3C occurred in the degree of vacuum attainable during heat treatment. It was found necessary, due to furnace limitations, to set the upper limit of pressure at 5×10^{-5} torr rather than the 1×10^{-5} torr specified. This upper limit on pressure was considered acceptable since qualification runs which preceeded the actual parts were acceptable under these conditions. As an added precaution, two layers of 0.002-inch thick Cb-1Zr foil were used to wrap the loop and components to further minimize the chances for contamination.

TABLE III

RESULTS OF CHEMICAL ANALYSES OF Cb-1Zr WELDS FOR HEAT TREATMENT QUALIFICATION

	Pressure, Torr		Element, ppm			
	Start of Run	End of Run	O	N	H	C
Base Metal Analysis by Vendor (MCN 417)[a]			130	85	1	50
Base Metal Analysis by G.E. (MCN 417)			142	22	2	30,40
Weld Metal After 2200°F/One Hour in R.D. Brew Vacuum Furnace (Model 424-B, 4-1/2-Inch Dia. x 7-Inch) at G.E. (Unwrapped)	3.6×10^{-5}	1.2×10^{-5}	129	56	5	60
Weld Metal After 2200°F/One Hour in VASCO Welding Chamber at G.E. (Unwrapped)	4.9×10^{-5}	1.7×10^{-5}	199	53	7	30
Weld Metal After 2200°F/One Hour in the C. I. Hayes, Inc. Furnace (Model PS-107, 20-Inch Dia. x 36-Inch) at Stellite Division, UCC (Wrapped)[b]	5.0×10^{-5}	2.5×10^{-5}	123	46	3	--
Weld Metal After 2200°F/One Hour in G.E. Vacuum Furnace (G.E. Catalog No. 221L804G1, 6-Inch Dia. x 10-Inch) at G.E. (Wrapped)	5.0×10^{-5}	1.0×10^{-5}	151	38	1	70

[a] 0.040-Inch Thick Sheet.

[b] Specimens Wrapped with One Overlapped Layer of 0.002-Inch x 0.5-Inch Cb-1Zr Foil. All Component were Wrapped with Two Layers of this Foil During Heat Treatment.

3. Sodium for Loop II

The schematic drawing of sodium purification and transfer system for Loop II was shown in a previous report[4]. Reactor grade sodium metal obtained from U.S. Industrial Chemicals Company was transferred from the shipping container to a titanium-lined, zirconium-gettered hot trap where it was outgassed at 600° to 800°F and subsequently, hot trapped for 102 hours at 1300° to 1370°F. After hot trapping, the hot-trap and charge-pot assembly were incorporated into the loop filling, sampling and dumping plumbing system adjacent to the Loop II test chamber. The assembled system was then helium leak checked and found to be free of leaks at room temperature. The transfer system was outgassed at 250° to 300°F until the pressure rise rate was reduced to 0.95 micron-liters per minute. A pressure of 1×10^{-5} torr was obtained in the system prior to flushing the stainless steel transfer system with three pounds of hot trapped sodium. Following oxygen analysis of the sodium sample taken during the flushing operation, the Loop II surge tank was filled with 4.5 pounds of sodium. The surge tank was pressurized to 100 psia with high-purity argon to fill the loop, and sodium was pumped through the loop at 500°F for one hour. The loop surge tank was then evacuated, causing the sodium to drain back into the surge tank. A sodium sample was subsequently taken from the surge tank and analyzed to determine if the oxygen level of the initial charge was sufficiently low to start the test.

The analytical results obtained on the sodium samples taken after hot trapping, after flushing the transfer system, and after filling and flushing the loop are given in Figure 9. Statistical analysis of these data indicate an oxygen concentration of 2 ppm, a 9.7 microgram oxygen blank for the analytical technique, and a 3σ value of + 5.9 micrograms. Metallic analyses on the hot trapped sodium and the loop flush sodium are shown in Table IV. Essentially no change in metallic constituents was indicated.

4. Loop II Instrumentation, Installation, and Operation

A. Instrumentation

Upon completion of the welding and heat treating of the Loop II assembly, a final helium mass spectrometer leak check of the assembly

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4 Potassium Corrosion Test Loop Development, Quarterly Progress Report #3, Covering the Period January 15, 1964 to April 15, 1964, NASA Contract NAS 3-2547, NASA-CR-54081, p 28.

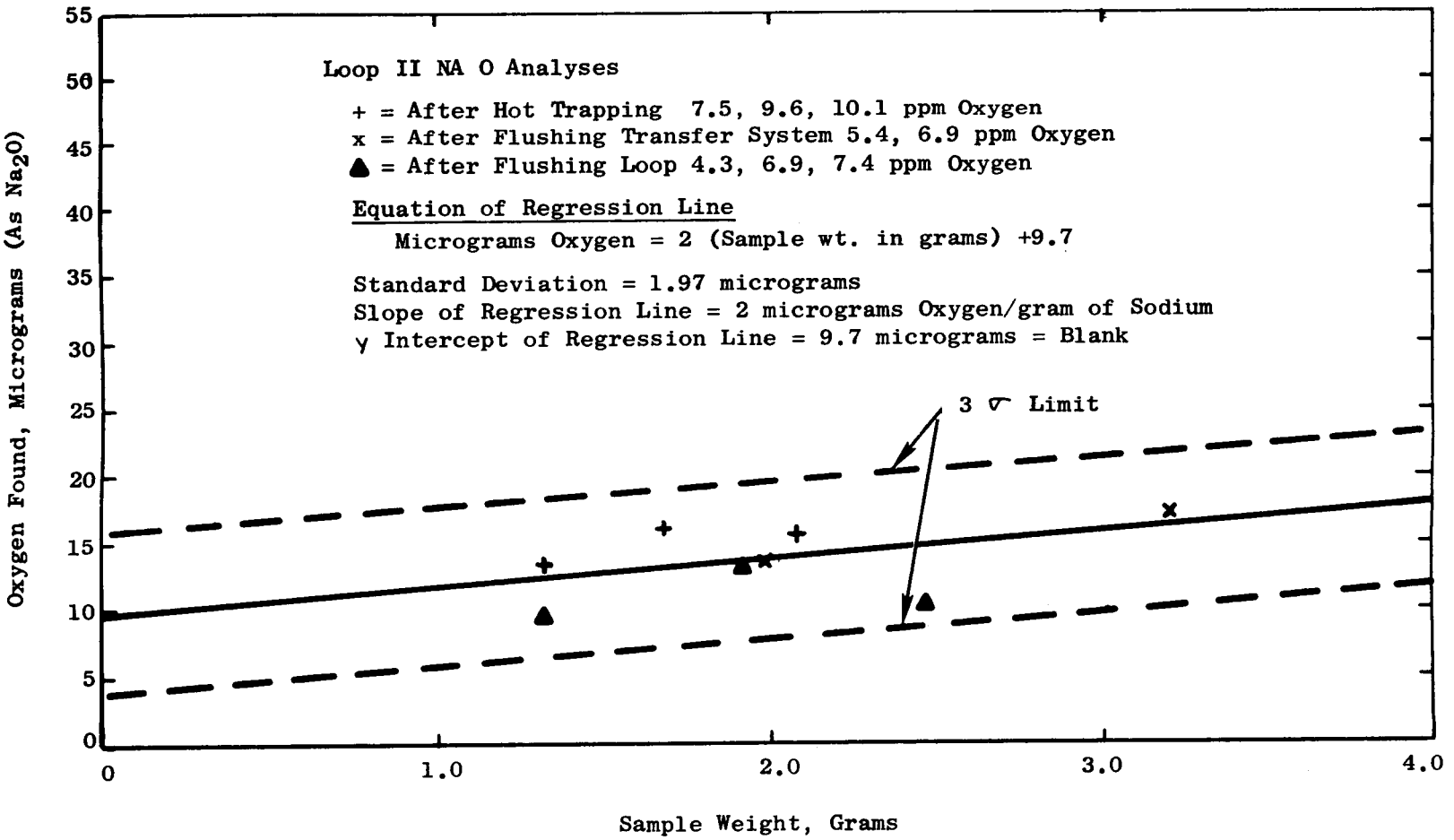


Figure 9. Oxygen Analyses on Loop II Sodium.

TABLE IV

METALLIC ANALYSES OF LOOP II Na

	Chemical Analysis, ppm										
	Fe	B	Co	Mn	Al	Hg	Zn	Sn	Cu	Pb	Cr
After Hot Trapping	1	<5	<5	<1	<1	<1	<50	<5	<1	<5	<1
After Loop II Flush	1	<5	<5	<1	3	1	<50	<5	<1	<5	<1

	Chemical Analysis, ppm											
	Si	Ti	Ni	Mo	V	Be	Ag	Zr	Sr	Ba	Ca	K
After Hot Trapping	<10	<5	<1	<5	<1	<1	<1	<10	<1	<3	<1	75
After Loop II Flush	<10	<5	<1	<5	<1	<1	<1	<10	<1	<3	3	40

was made. No leaks were detected using a helium mass spectrometer having a sensitivity of 5×10^{-10} standard c.c. of air per second. Loop II was then transferred to a portable, air supported clean room for instrumentation, insulation and installation of auxiliary loop equipment.

The portable clean room shown in Figure 10 measures 15 feet in diameter x 15 feet high. It is fabricated from re-inforced transparent plastic sheet and is supported by internal pressure of approximately 0.2 psi provided by an air blower. A double door, interlock chamber permits traffic to move into and out of the clean room without the loss of the internal pressure which would result in the collapse of the shelter. All personnel engaged in instrumenting the loop are required to wear lint free, white shoes covers, coveralls, and gloves. All components, including the loop and spool section, were vacuum cleaned, then washed with ethyl alcohol, and vacuum cleaned again to remove any dust or other foreign material that may have accumulated during the fabrication or in the final shipment of the loop to the test area.

1. Temperature Measurement

W-3%Re vs W-25%Re thermocouples were used exclusively in instrumenting Loop II. The 0.005-inch diameter wire used was from a single lot and calibrated in an argon atmosphere against a standard platinum-rhodium thermocouple over the 200°-2200°F temperature range. A similar calibration is scheduled during the next quarter in a 1×10^{-6} torr vacuum. The locations of the 28 thermocouples used to measure the loop operating temperature are shown in Figure 11. Two thermocouple installation procedures were used. Alumina-insulated, beaded-junction thermocouples were inserted into wells located in the EM pump inlet and outlet reducers, while the remainder of the thermocouples were of the split-junction type with each wire being welded to the tube wall. Two hole, 99.7% Al_2O_3 insulators were used to electrically insulate the wires inside the vacuum chamber. The thermocouple wires were routed from the junction to a 99.7% Al_2O_3 terminal block attached to the loop support structure. The terminal block was used for convenience in the installation and checkout of thermocouples since the transition from tungsten-rhenium wire to copper wire is made outside of the vacuum chamber. The tungsten-rhenium wires are run from the terminal block along the support structure through an octal vacuum feedthrough, shown in Figure 12, to a reference junction box which is maintained at 150°F. Copper wire leads connect the reference junction box to a 24-point recording potentiometer.

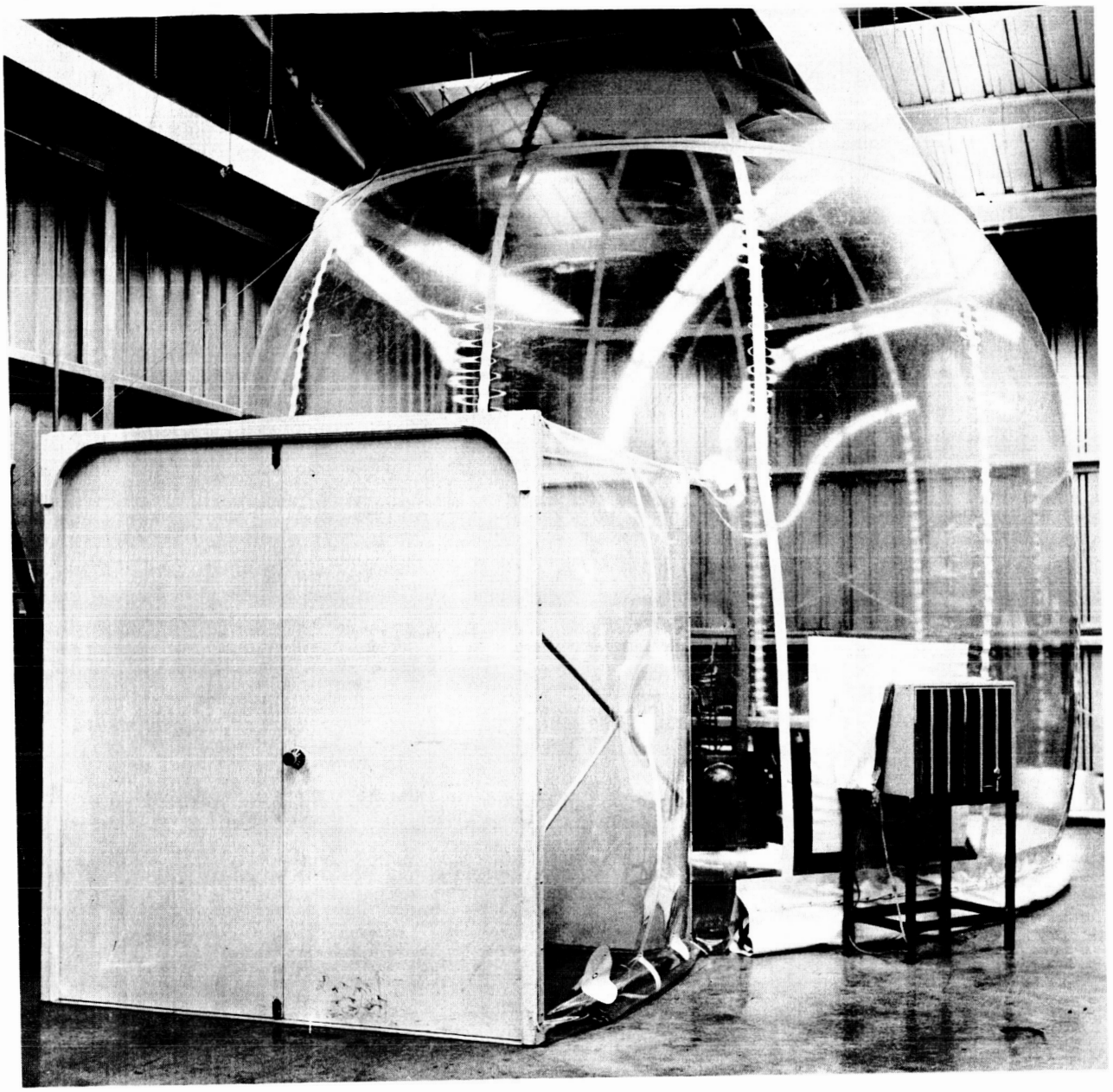


Figure 10. Portable Clean Room. (C64061101)

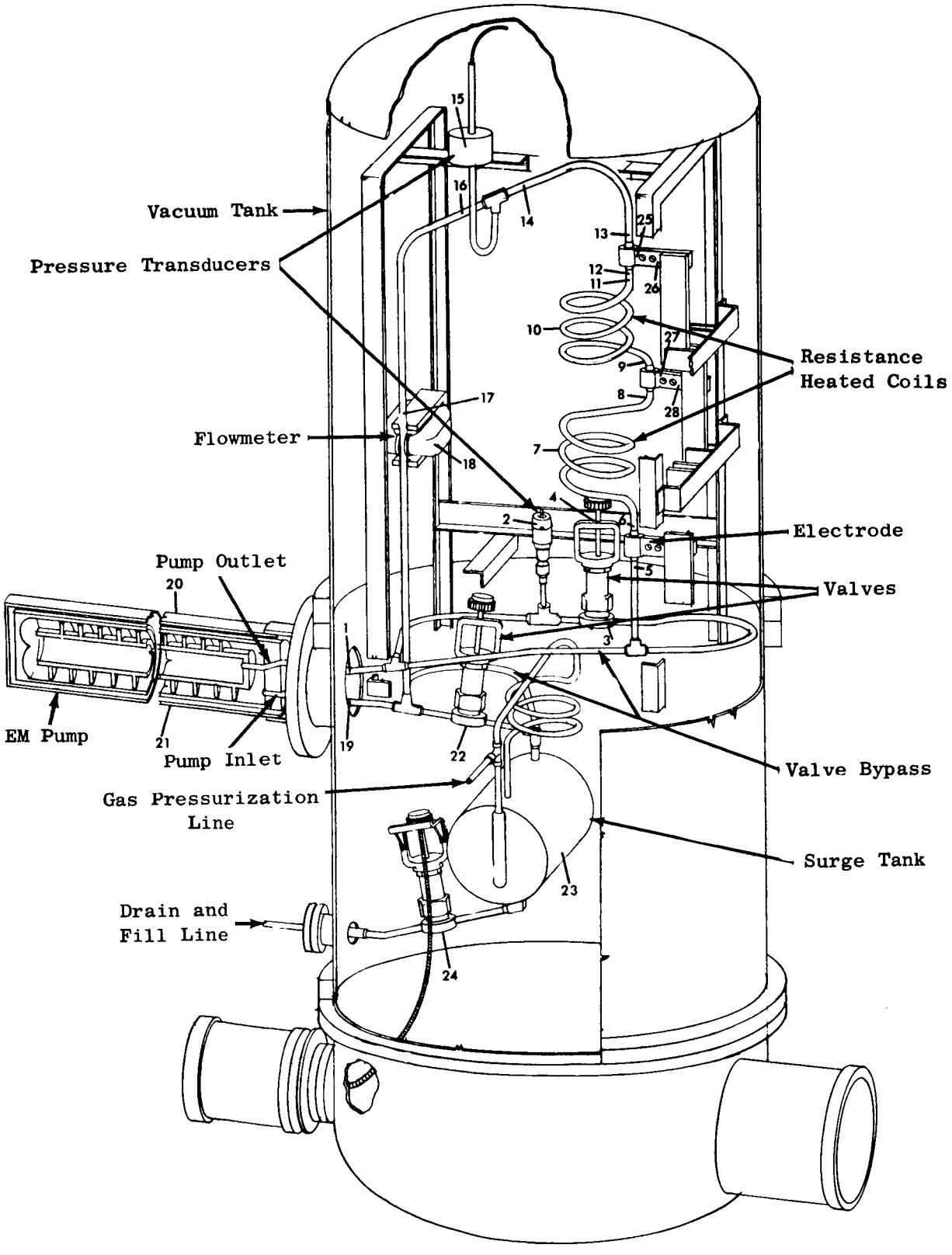
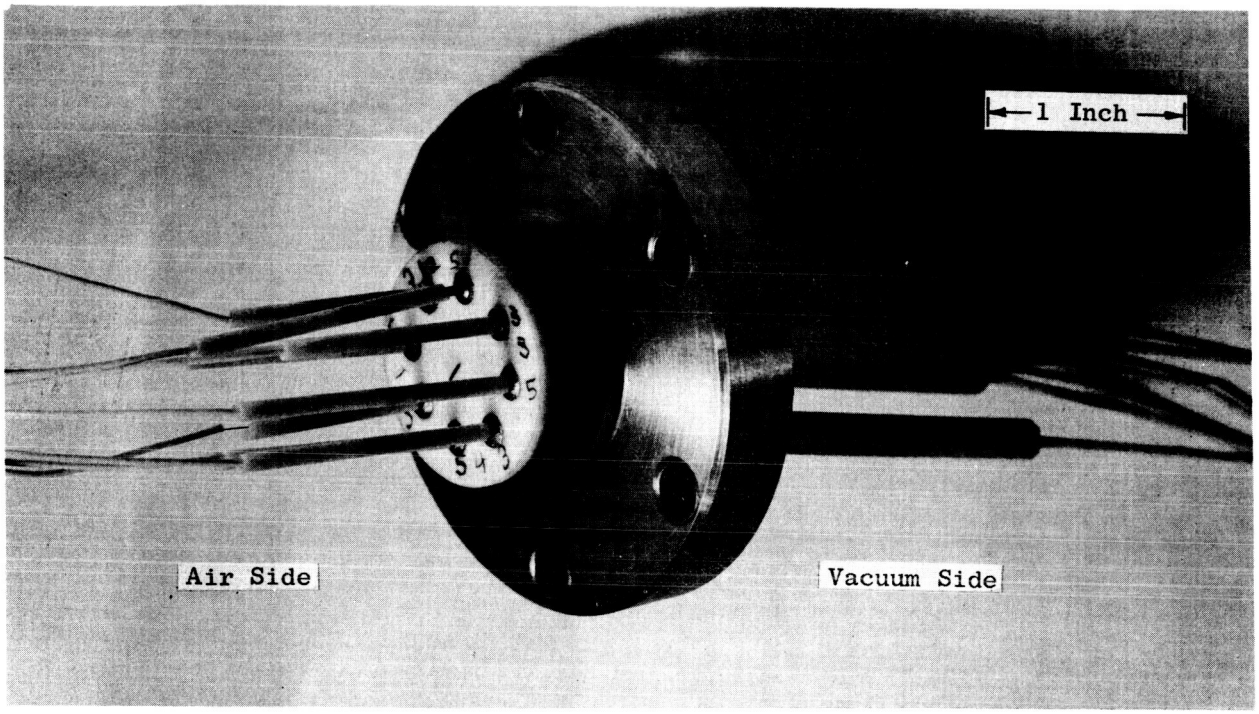


Figure 11. Component Evaluation Test Loop II Showing Location of W-3%Re vs W-25%Re Thermocouples. (C64061705)



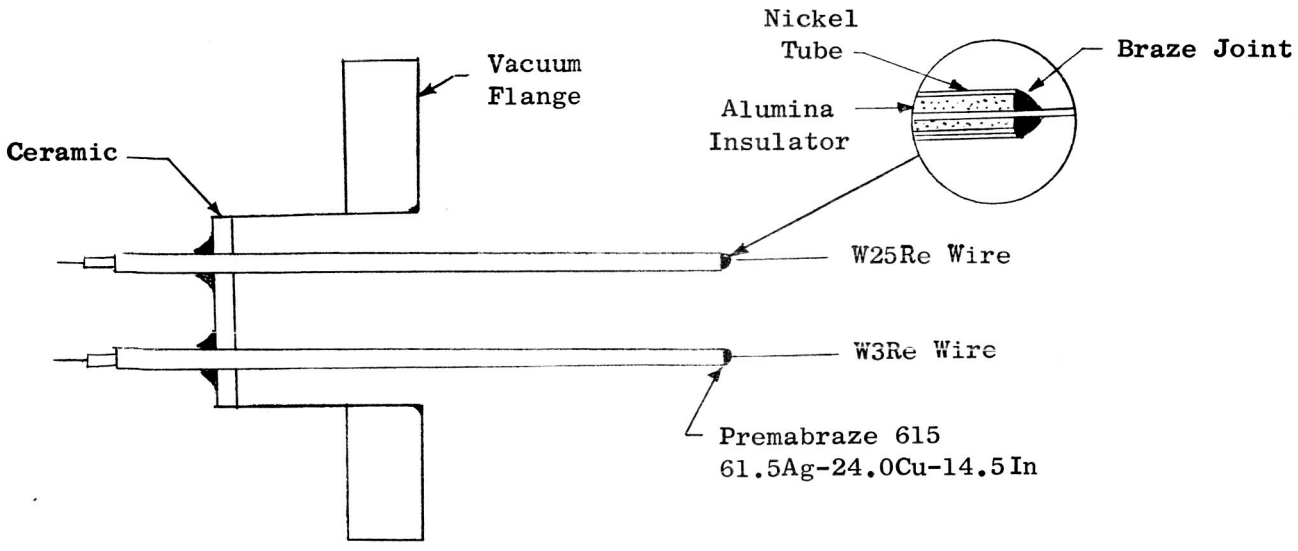


Figure 12. High-Vacuum W-Re Thermocouple Feedthrough Assembly. (C64061104)

The major source of error that can and has occurred in the above system is in the octal vacuum feedthrough where the wire is brazed with Premabraze 615 (61.5Ag-24Cu-14.5In) braze alloy in a small 0.050-inch diameter nickel tube. A significant error arises if the thermocouple wire makes a second electrical contact (in addition to the brazed vacuum seal) to the nickel tubulation since during loop operation the tubulation can have a several hundred degree temperature differential from the brazed end (vacuum end) to the opposite end. This problem was observed in Loop I operation and duplicated in bench tests which gave several hundred degrees error under similar test conditions. The problem has been eliminated by carefully inserting a small diameter, single-hole insulator inside the nickel tube from the air side which electrically insulates the thermocouple wire from the tube.

2. Pressure Measurements

Pressure measurements in Loop II are restricted to a single slack diaphragm electronic volumetric pressure transmitter in the high temperature circuit and an all refractory alloy stressed diaphragm transducer in the low-flow, low-temperature bypass line. In addition to the two liquid metal pressure gauges, an argon gas pressure gauge located on the control console and connected to the top of the surge tank is also used to monitor the loop pressure during operation and for calibration of the liquid metal pressure gauges.

The volumetric pressure transducer consists of a diaphragm housing, diaphragm, NaK-filled capillary, and bourdon tube. All parts which are wet by the process fluid (sodium in Loop II, potassium in the Pre-prototype Loop) are made from Cb-1Zr alloy. The diaphragm housing and the process tube which connects it to the loop are shown near the top in Figure 13, which is a photograph of the Loop II taken during installation. The slack diaphragm pressure transducer gauge has a pressure range of 0-150 psia at a maximum temperature of 2000°F and a response time of one second for 63% of the range. Similar units fabricated from stainless steel and L-605 alloy have been used in liquid metal heat transfer test loops at General Electric.

Because of the relatively slow frequency response of the slack diaphragm type of transmitter, an all refractory alloy, stressed diaphragm transducer was designed and fabricated for evaluation in Loop II. This transducer consists of a housing containing a 0.015-inch thick T-111 alloy diaphragm which serves as a barrier to keep the alkali metal in the loop system. The diaphragm serves as a stressed member and its deflection, which is proportional to the load upon it,

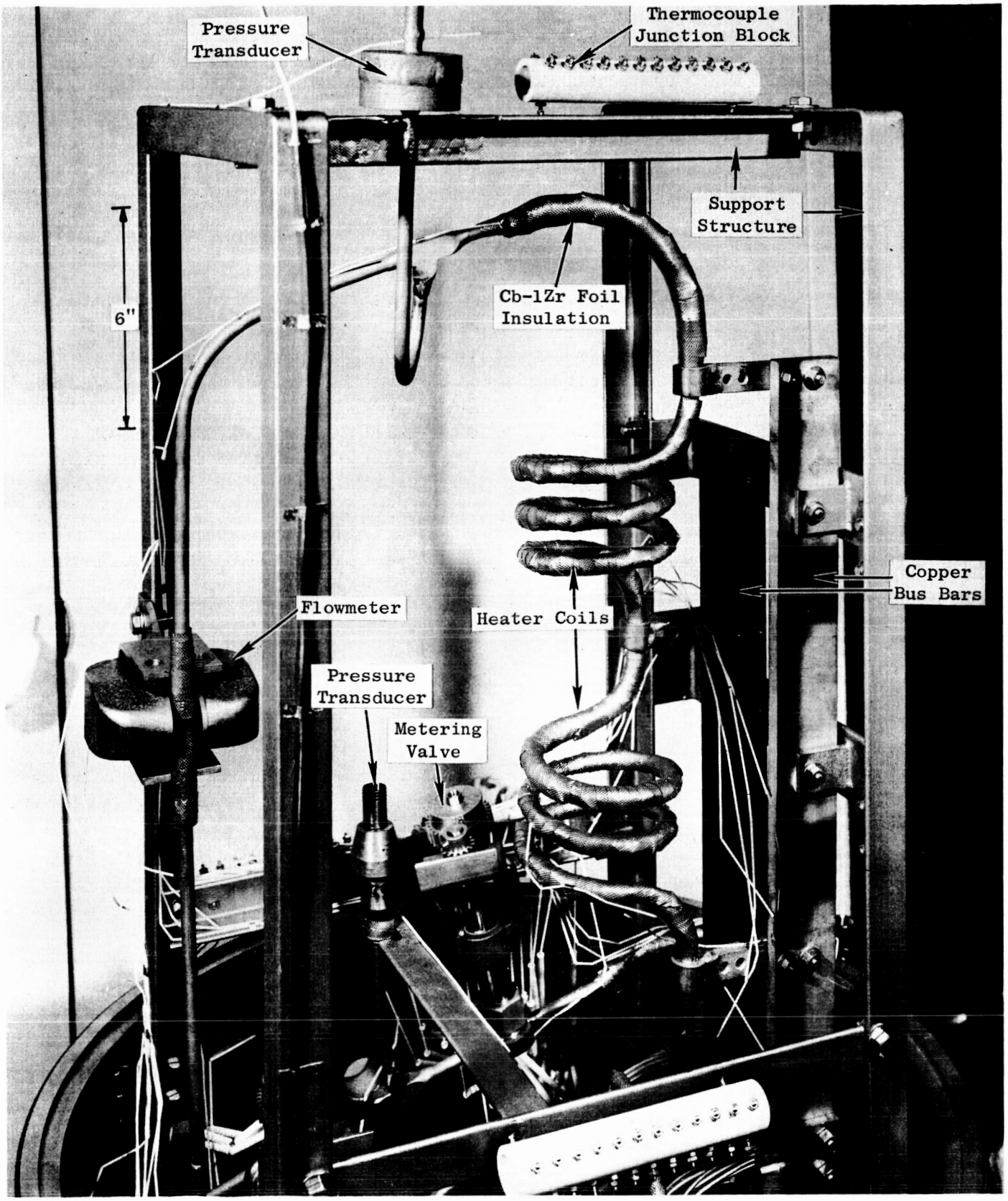


Figure 13. Loop II Partially Instrumented. (C64061106)

is the measured quantity. The stressed diaphragm acts as a stiff spring and it is this feature which provides the high-response characteristics of this type of transducer. The high stresses in the diaphragm also tend to limit the operating temperature and long term accuracy capabilities of the transducer. Diaphragm deflection is measured by means of a linear variable differential transformer which is mounted at the diaphragm housing and is activated by a magnetic core attached to the diaphragm by means of a stainless steel wire, as described previously under Loop II fabrication.

A preliminary calibration indicated that the transducer was functioning properly before assembly into the loop system. A description of the output signal in millivolts versus absolute pressure level is given in Table V. The severe non-linearity of the system output could be corrected by manipulation of the electronic circuitry but the primary purpose of the present application is rapid response, and this does not warrant the additional expense required to improve the linearity. Due to the difficulties involved in experimentally determining the actual frequency response characteristics, a calculated value will be used.

3. Sodium Flow Rate

The sodium flow rate of Loop II is measured by a permanent magnet flowmeter designed to operated at a liquid metal temperature of 2200°F. This flowmeter is shown in Figure 13. The 2400 Gauss permanent Alnico V magnet has been stabilized by repeated thermal cycling from room temperature to 900°F. The magnetic flux as a function of temperature after thermal stabilization is shown in Table VI in the room temperature to 900°F range. These values are used in calculating the sodium flow rate at the design temperature. The small effect of magnet temperature on the flux measured is significantly lower than published data for Alnico V which shows a 1.1% loss in flux per 100°F temperature rise below 600°F. A re-calibration of the magnet will be made upon completion of Loop II testing.

Cb-1Zr foil insulation is used to thermally shield the magnet. The flow tube is fabricated from 0.375-inch OD Cb-1Zr tube with 0.065-inch wall, which is the principal Loop II plumbing material. The Cb-1Zr electrodes, which are 0.062-inch diameter wire, were purchased to the same chemistry specifications as the tube to minimize the spurious thermal emf which could result from dissimilar materials at temperature. A calculated output voltage vs flow rate from the Loop II flowmeter is shown in Figure 14.

TABLE V

PRELIMINARY ROOM TEMPERATURE

CALIBRATION REFRACTORY PRESSURE TRANSDUCER

Pressure* psig	Output Signal MV
0	94.5
15	106.94
30.3	118.90
45.0	129.28
59.5	136.62
80.5	144.47
95.8	149.10
113.5	154.15
134.0	159.64
152.5	163.50

* Argon Gas Used. Pressure Measured with a Heise Gauge (Model No. H6812) 0-200 psig Range in 0.5 psig Increments.

TABLE VI

MAGNETIC FLUX VS TEMPERATURE FOR LOOP II ALNICO V FLOWMETER

MAGNET AFTER THERMAL STABILIZATION AND PRIOR TO THE LOOP TEST

Magnet Temp., °F	Gauss Magnetic Flux		
	Run #1	Run #2	Run #3
70	2400	2400	2400
200	2400	2400	2400
400	2400	2400	2400
500	2400	2400	2400
600	2400	2370	2400
700	2400	2370	2400
800	2370	2370	2400
900	2370	2370	2370

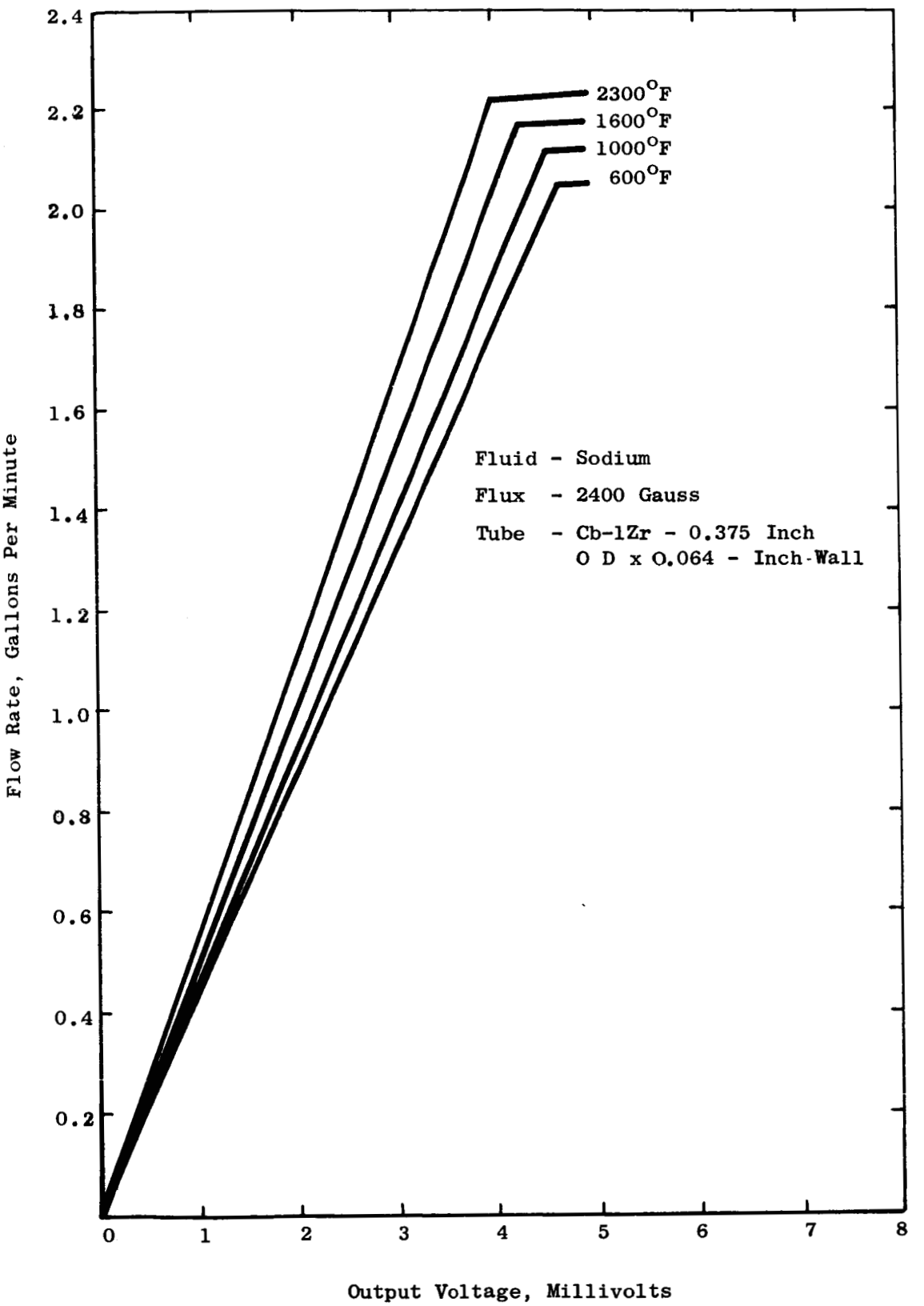


Figure 14. Calculated Output Voltage vs Flow Rate for the Loop II Permanent Magnet Flowmeter.

B. Installation

Thermal insulation was added to the heater assembly, the flowmeter tube, and the EM pump inlet and outlet during the instrumentation of the loop in the clean room. The insulation consisted of multiple layers of dimpled 0.002-inch thick by 0.5-inch wide Cb-1Zr foil wrapped around the tube assembly as shown in Figure 13.

The stainless steel support structure was adjusted to accommodate the loop and individual support members for the pressure transducers and surge tank were positioned and bolted in place. The electric power copper bus bars for the heater assembly were installed with high-purity Al_2O_3 insulators to isolate the heater from the support structure.

The final step in the installation of the loop in the spool section before transfer to the vacuum tank was the assembly of the valves. Figure 15 shows an exploded view of the valve assembly, and Figure 16 shows the assembled valve with the gear train which is required to permit the flexible cable to be connected to the valve from the rotary vacuum feedthrough which is located on the lower vacuum tank (sump). Bench tests of the valve assembly in air in the arrangement required for Loop II operation showed that less than 2 inch-lbs of torque applied to the rotary vacuum feedthrough is sufficient to operate the valve with 100 psi internal pressure. Rotary feedthroughs rated at 4-inch pounds are used to drive the valves.

Figure 17 shows Loop II mounted on the spool piece just prior to its transfer to the vacuum chamber. Following attachment to the sump of the vacuum chamber, the sodium and argon pressure lines were connected, the EM pump electrical windings were bolted to the flange shown in Figure 17, and the thermocouples and pressure gauges connected to the readout equipment. Safety circuits on the test system were then checked out.

The safety circuits include the following:

a) An overtemperature thermocouple which turns off heater power to the loop if the loop temperature exceeds the control temperature by 200°F.

b) An explosive valve argon flooding system which is actuated by the getter-ion pump current if the pump current exceeds 700 m.a. (equivalent to a pressure of 5×10^{-5} torr).

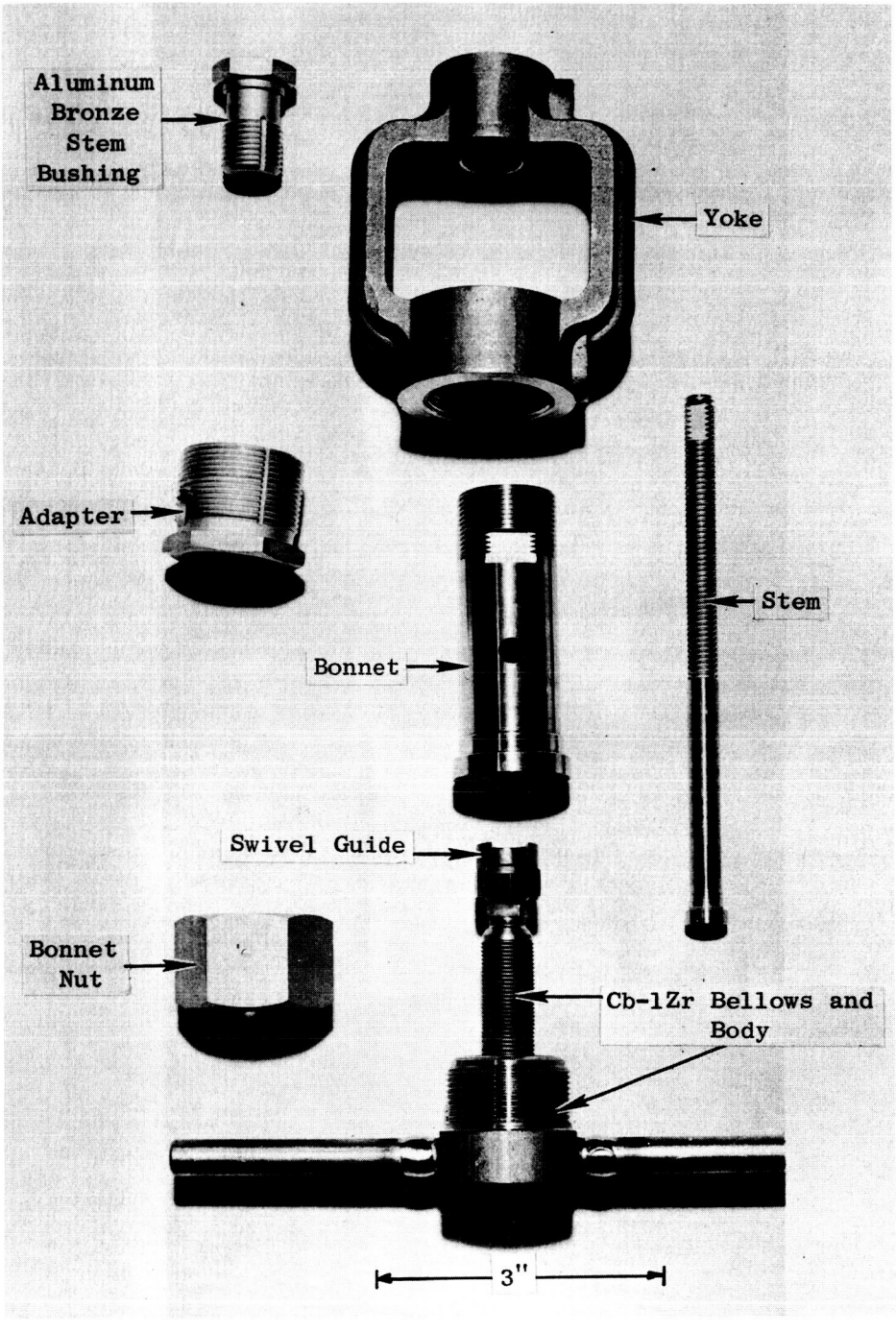


Figure 15. Exploded View of Hoke Valve Used on Loop II. All Parts Shown with the Exception of the Bellows, Body and Stem Bushing are Made of Stainless Steel. The Stem Bushing, Adapter, Bonnet Nut, and the Threaded Portion of the Stem were Gold Plated to Minimize Galling. (C64060420)

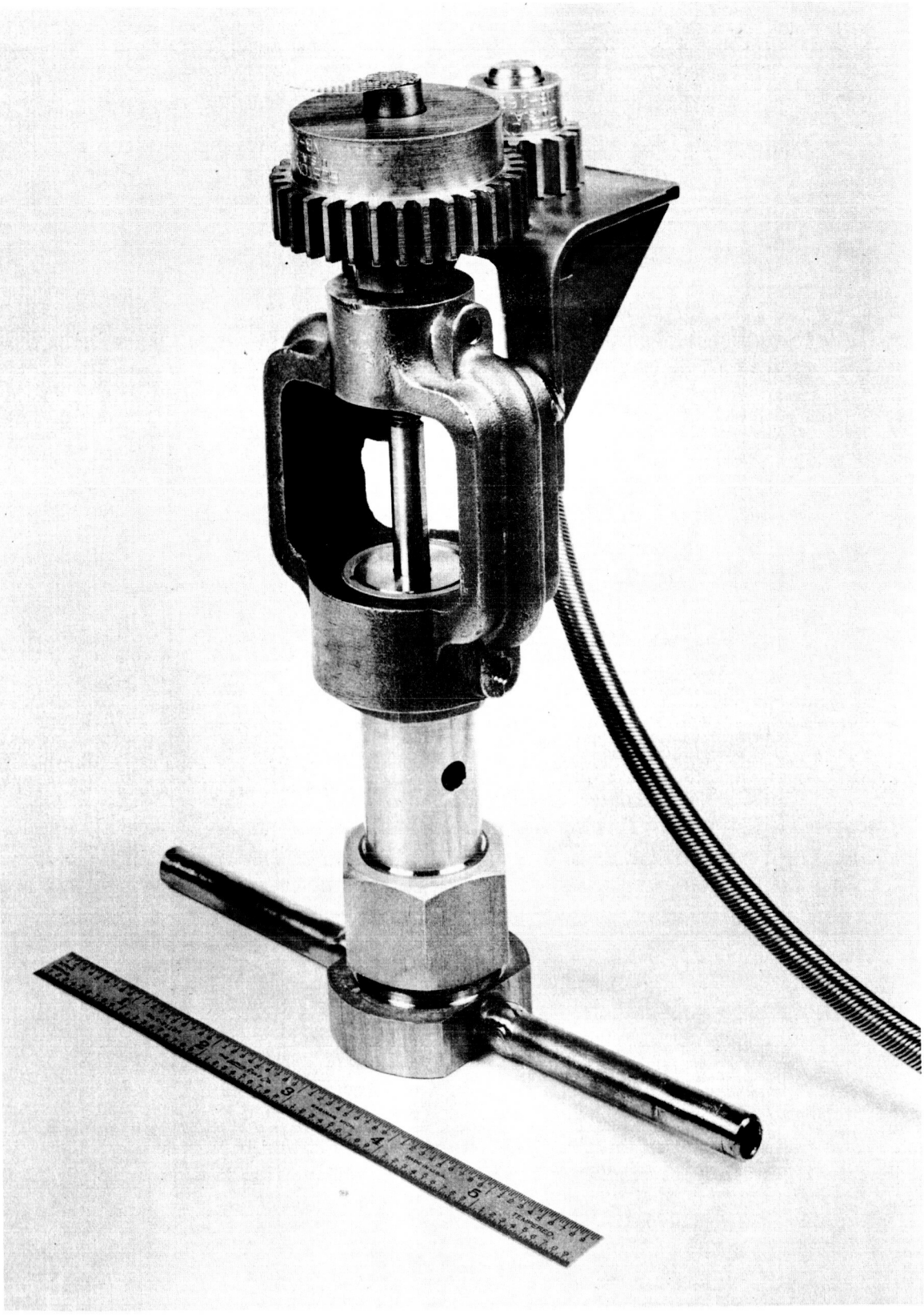


Figure 16. Assembled Valve and Gear Train. Flexible Stainless Steel Cable to Magnetically Coupled Rotary Vacuum Feedthrough is Shown on the Right. (C64090426)

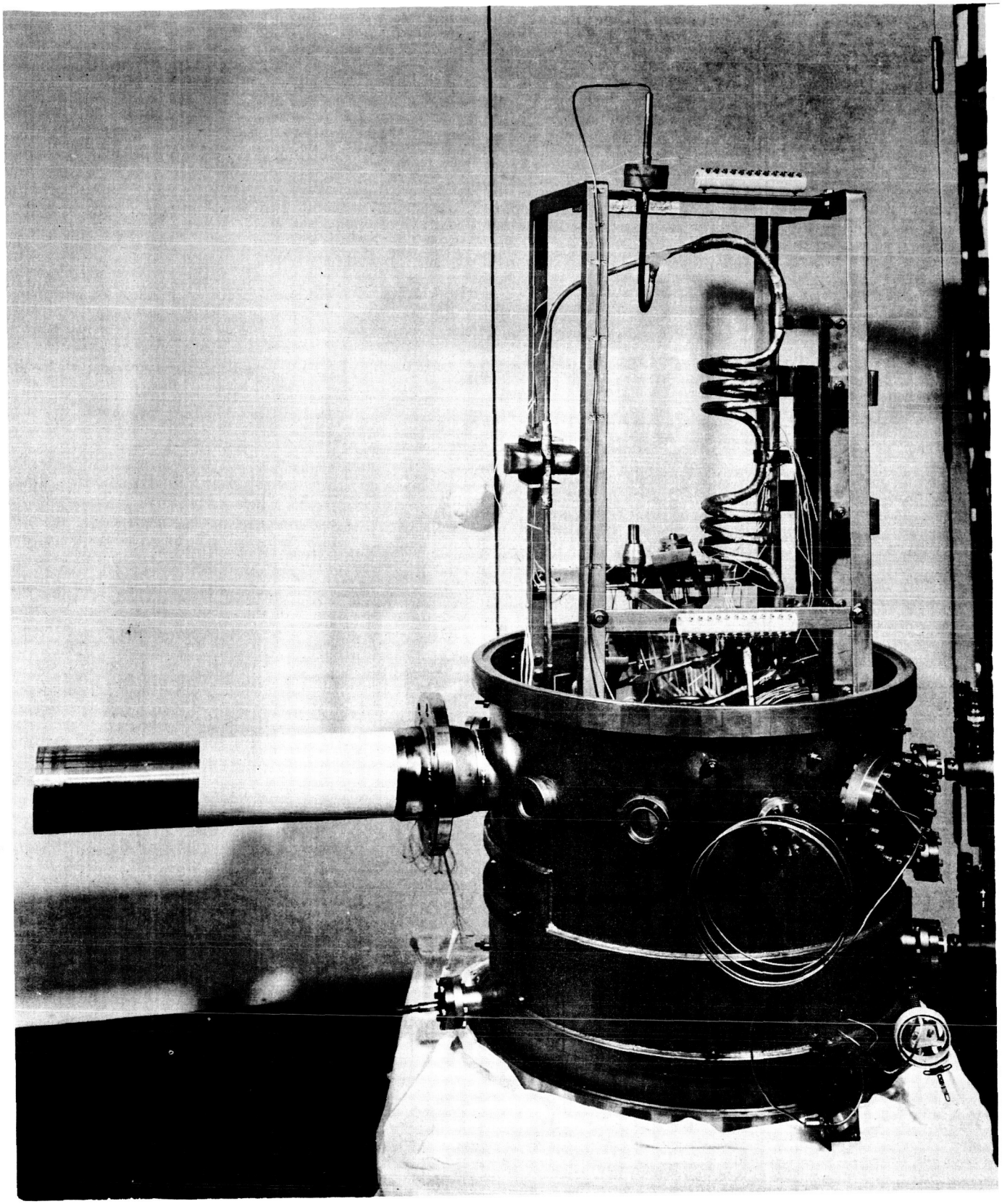


Figure 17. Loop II and Spool Piece Near Completion of Installation and Instrumentation. Stainless Steel Can Which Forms Vacuum Enclosure Around Cb-1Zr EM Pump Duct is Shown on the Left. (C64061105)

c) A relay in the ionization gauge control which shuts off the loop heater power and the EM pump if the pressure rises to above 1.5×10^{-7} torr.

The control console for Loop II is shown in Figure 18.

The sodium transfer, flushing, and sampling operations and results have been described in the preceding section.

C. Test Chamber Bakeout and Loop II Startup

A summary of test chamber pressure vs time during the bakeout and test startup period is given in Figure 19. The bakeout was interrupted at 237 hours to repair and modify the metering valve drive actuation system. As was anticipated, a considerable time was required to outgas the system components. Most of these components were on bakeout for approximately 170 hours. While on bakeout the temperatures of the various components in the vacuum environment varied from a minimum of 250°F to a maximum of 525°F. Significant pressure rises occurred each time the loop temperature was increased, but remained at these higher pressure levels for only a few hours. The maximum pressures observed during these periods are given in Figure 19. Mass/charge ratios of 2 (H_2) and 28 (N_2 or CO) made up 22% and 36%, respectively, of the total gas pressure (8.6×10^{-6} torr), when the loop was filled with sodium and increased in temperature to 500°F. Hydrogen became the principal gas present when the sodium in the loop reached a temperature of 950°F. At this time (192 hours), the total pressure was 8.0×10^{-7} torr and the hydrogen accounted for 42% of the total pressure.

During the period from 288 to 292 hours when the sodium temperature was being increased from 1000° to 1970°F, the pressure increased to 6.5×10^{-6} torr but dropped to less than 1×10^{-6} torr in three hours. The titanium sublimation pumps were very effective in reducing the pressure in the system during the peak gas load period associated with startup of the test.

D. Loop II Operation

Since the loop reached the desired test conditions on July 2, the ionization gauge pressure has exceeded 1×10^{-7} torr for approximately 50 hours and during this period hydrogen has been the principal gas in the system.

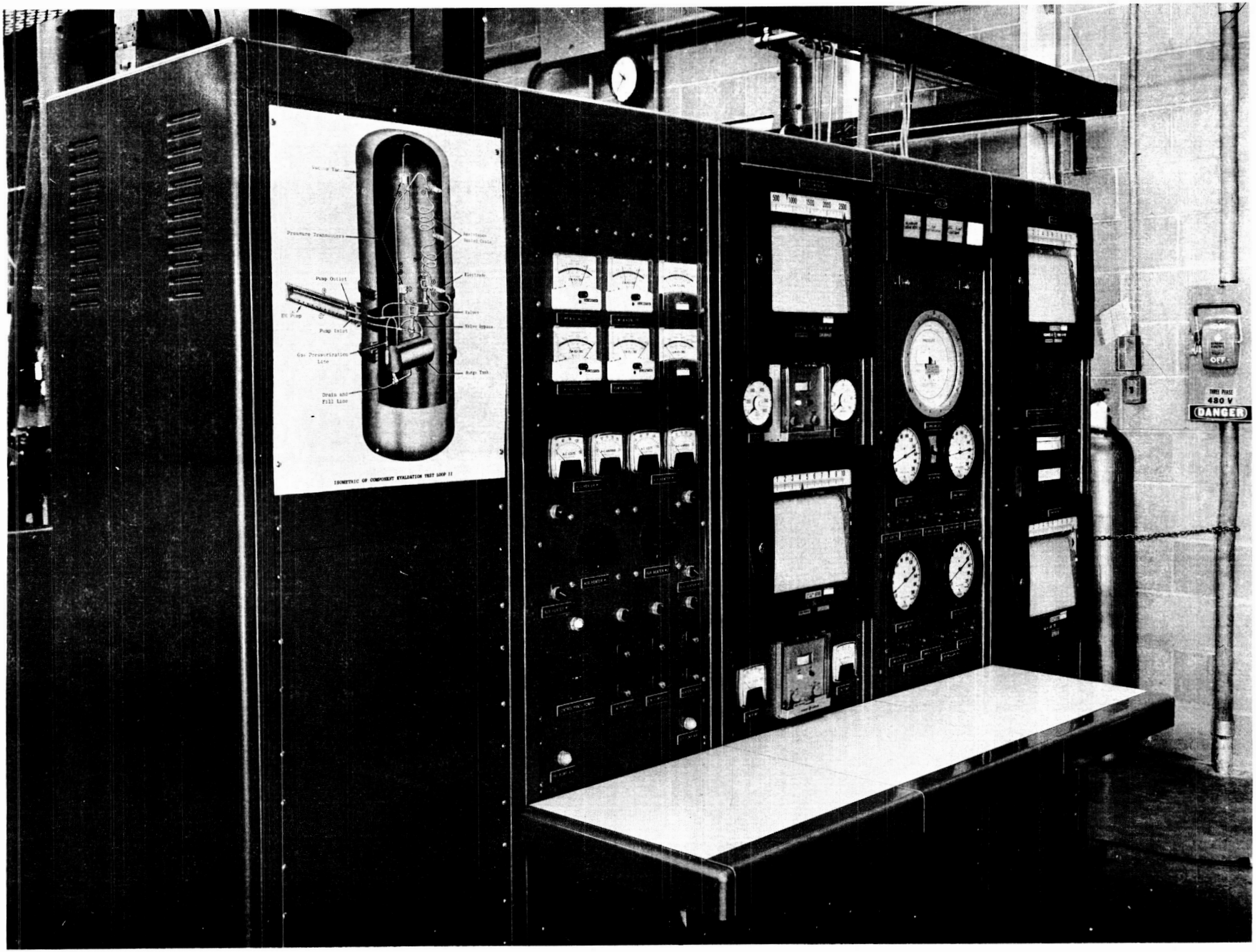


Figure 18. Control Console of Component Evaluation Test Loop II. (C64061831)

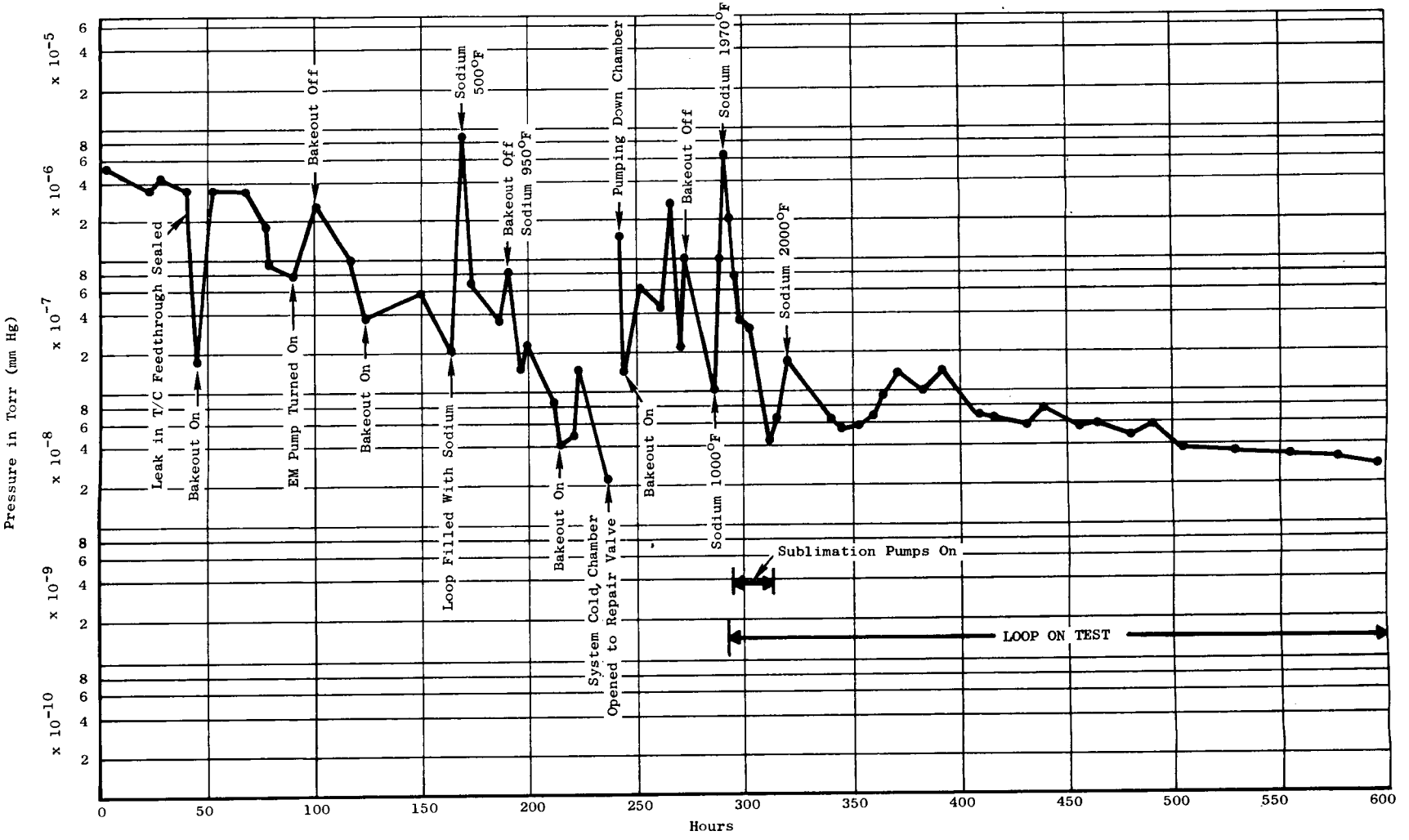


Figure 19. Vacuum Chamber Pressure as a Function of Time for Loop II Bakeout and Initial 310 Hours of Loop Operation.

As of July 15, Loop II has completed 310 hours of uninterrupted operation. The principal test conditions at this time are listed below:

Heater outlet temperature	-	2060°F
Pump inlet temperature	-	1985°F
Pump outlet temperature	-	1975°F
Metering valve temperature	-	660° to 900°F
Loop pressure	-	144 psia
Sodium flow rate	-	1 gpm
Electrical power input	-	6.75 KW
Chamber pressure	-	2.7×10^{-8} torr

The results of a partial pressure analyzer scan at this time are given in Table VII. The analyzer was calibrated using helium and nitrogen, and the results of this calibration were used in converting the indicated pressure to true pressure. As may be noted in Table VII, mass/charge-2 (H_2) and -28 (N_2, CO), are the principal species present and there is a reasonably good correlation between the ionization gauge pressure and the total of the partial pressures. More detailed information regarding the calibration of the residual gas analyzer will be presented in the next section of this report.

During the steady state testing, several adjustments to the metering valve were required to increase the flow in the bypass line to maintain a valve temperature of 800°F. The flow in the bypass line, which is a small fraction of the one gallon per minute flow in the main line, is not measured, but can easily be detected by temperature changes in the bypass. It is apparent that some type of flow restriction or blockage is developing in the bypass probably at the valve seat which has the smallest flow passage in the entire loop. The flow area in the valve with the metering valve adjusted to give an initial temperature of 800°F is approximately 0.002-inch^2 with a flow gap between the plug and valve seat of 0.0043-inch.

TABLE VII

RESULTS OF PARTIAL PRESSURE ANALYZER

SCAN OF LOOP II CHAMBER ENVIRONMENT

Time: 08:00 - July 15, 1964

Total Hours of Loop Operation: 310

Ionization Gauge Pressure: 2.7×10^{-8} Torr

Mass/Charge	Ion Current amps	Indicated Pressure Torr	True Pressure Torr
2	2.8×10^{-10}	0.30×10^{-8}	0.70×10^{-8}
4	7.8×10^{-11}	0.08×10^{-8}	0.46×10^{-8}
16	1.0×10^{-11}	0.01×10^{-8}	0.01×10^{-8}
18	2.5×10^{-10}	0.30×10^{-8}	0.34×10^{-8}
28	4.7×10^{-10}	0.62×10^{-8}	0.62×10^{-8}
32	Not Detected	Not Detected	Not Detected
40	1.8×10^{-10}	0.26×10^{-8}	0.17×10^{-8}
44	6.6×10^{-11}	0.10×10^{-8}	0.07×10^{-8}
		TOTAL TRUE PRESSURE	2.37×10^{-8}

5. Residual Gas Analysis in the Loop II System

During the operation of Loop II, the partial pressure gas analyzer* is being used to monitor the composition of the residual gases in the vacuum chamber. In order to obtain quantitative data, the analyzer was calibrated against the system ion gauge for several pure gases admitted into the system through a variable leak valve.

The procedure used for this calibration was essentially the same as that utilized in the calibration of the partial pressure analyzer used for analysis of the welding chamber helium. The calibration and calculation procedures have been described in the previous progress report[5]. It was found, however, upon the introduction of a pure gas into the system there was an appreciable increase in the concentration of practically every residual gas in the system. This effect was most pronounced when the inert gases, argon and helium, were admitted. In one particular case, upon introducing argon into the system, the helium ion current was actually greater than the argon ion current. More typically, the ion current due to the gas admitted was about 75% of the total ion current when inert gases were admitted, and about 90% of the total for the more active gases, with considerable variation from these typical values. This effect is most probably due to evolution of previously pumped gases from the ion pump. Because of this effect, it was necessary in the data reduction procedure to first estimate the response of the analyzer to each of the residual gases in the system, and then to correct the ionization gauge reading for the presence of all species which had concentrations sufficient to affect the ion gauge indication.

Two separate calibrations of the partial pressure analyzer have been made. The first and more complete calibration was performed before the loop was assembled in the vacuum chamber. Base pressure for these tests were about 2×10^{-10} torr and calibrations were made for the pure gases H_2, He, N_2, A, and CO_2 at pressure generally between 5×10^{-7} and 5×10^{-9} torr as indicated by the ion gauge.

The second calibration was performed after the loop had been installed in the chamber but before actual operation of the loop. This check was made to determine whether the response of the analyzer had

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* Vacuum Products Division, General Electric Company, Model 514.

5 Potassium Corrosion Test Loop Development, Quarterly Progress Report #3, Covering the Period January 15, 1964 to April 15, 1964, NASA Contract NAS 3-2547, NASA-CR-54081, p 53.

changed due to actual instability of the analyzer system with time or to some other factor such as accidental movement of the analyzer tube relative to the magnet. For the second calibration base pressures of about 3×10^{-8} torr were obtained and calibrations were made for He and N_2 at pressures as high as 8×10^{-7} torr.

The results of these two calibrations are shown in Figure 20 where the ratio of the partial pressure analyzer ion current to the indicated ion gauge pressure is plotted against mass of the calibrating gas. As may be seen from Figure 20, there is relatively little mass discrimination in the analyzer system; that is, the response of the analyzer to high mass species is the same (within a factor of 2) as the response to the low masses. Apparently some small change in the response of the analyzer occurred between the two calibrations. The data from the second calibration are being used to calculate the partial pressure during the Loop II tests.

For a particular residual gas in the system, the data of Figure 20 may be used directly to obtain the indicated ion gauge pressure. In order to obtain the true partial pressure, one must divide the indicated ion gauge pressure by the relative response of the ion gauge. In Table VIII are given the various factors necessary to convert the partial pressure analyzer ion current to either indicated pressure or to the true pressure. These factors were used in calculating the indicated and true pressure listed in Table VII.

Figure 21 is a mass spectrum of the residual gases in the vacuum system after 196 hours of loop operation. In examination of this spectrum, the ion current multiplier setting should be carefully noted. Thus, while the peak height at mass/charge ratio (m/e) = 4 is greater than that at m/e = 2, the actual ion current at m/e = 2 is nearly a factor of 10 greater than that at m/e = 4. Similarly, the peak m/e = 55 which at first glance appears to be a major peak, is actually comparable to the peak at m/e = 25.

The gas species associated with each of the major peaks in this spectrum, along with the true pressure calculated from the data of Table VIII, are shown in Table IX.

From the data of Table IX, it may be seen that H_2 is the most abundant residual gas at a pressure of 1.8×10^{-8} which is about half the total pressure. The sum of the partial pressure calculated from the mass spectrum is appreciably less than the ion gauge reading.

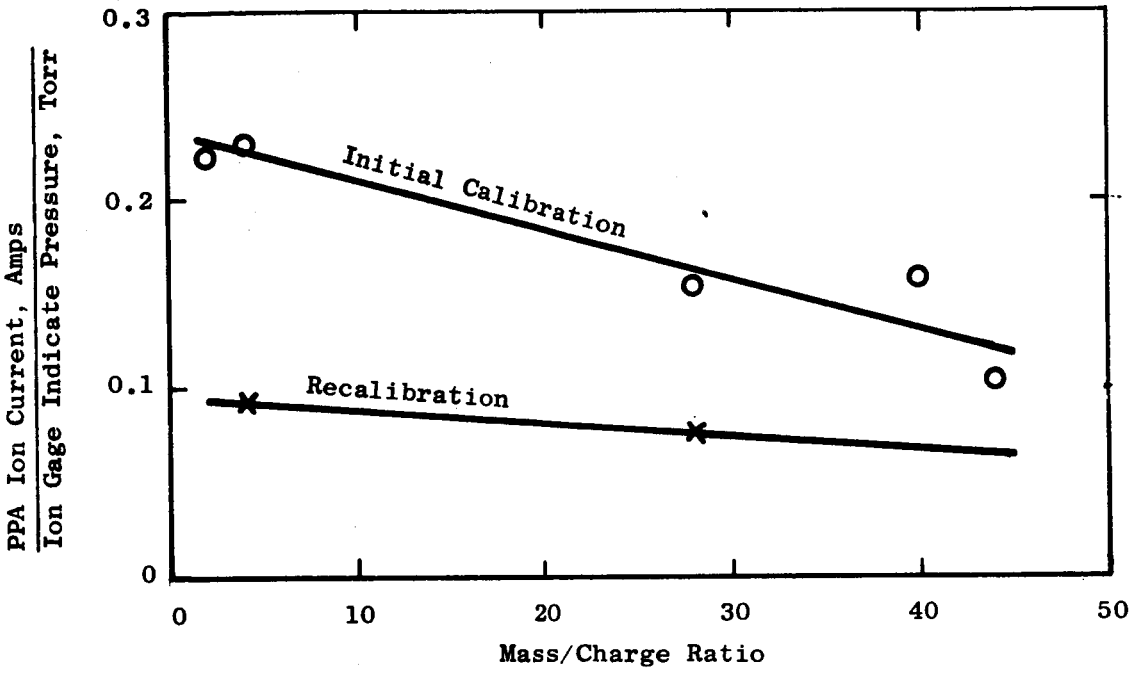


Figure 20. Calibration of Partial Pressure Analyzer.

TABLE VIII

CONVERSION FACTORS OBTAINED FROM RECALIBRATION OF THE PARTIAL PRESSURE ANALYZER

Gas	Mass	Ion Gauge Sens. Relative to N_2	To Obtain Indicated Pressure Multiply PPA Ion Current By	To Obtain True Pressure Multiply PPA Ion Current By
H_2	2	0.42	10.6	25.2
He	4	0.19	10.8	56.8
CH_4	16	1.07	11.8	11.0
H_2O	18	0.89	12.1	13.6
N_2 or CO	28	1.00	13.2	13.2
O_2	32	0.85	13.7	16.1
A	40	1.56	14.7	9.4
CO_2	44	1.37	15.4	11.2

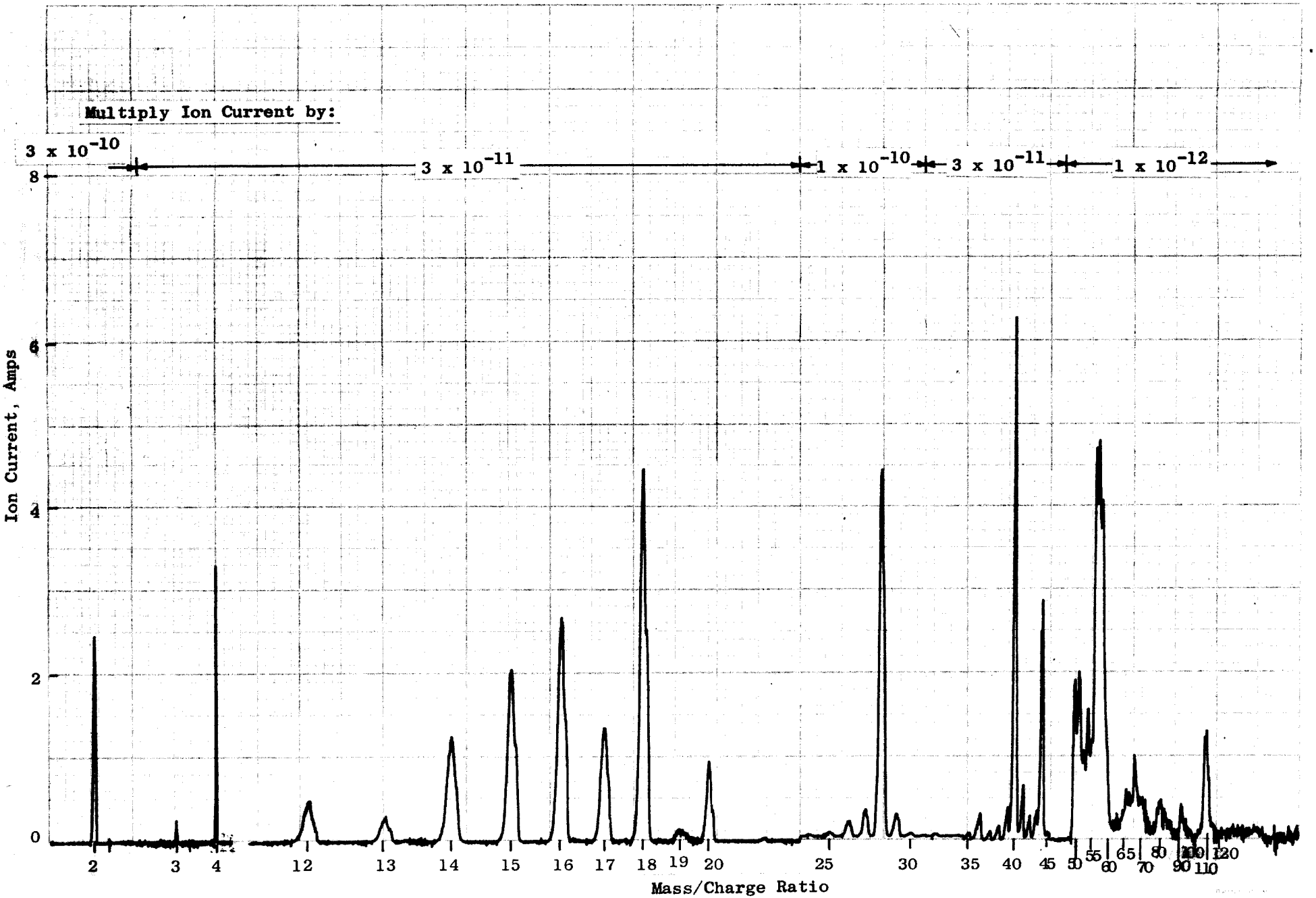


Figure 21. Mass Spectrum of Residual Gases in Loop II Vacuum System. Maximum Loop Temperature: 2020°F, Total Hours of Loop Operation: 169.

TABLE IX

PARTIAL PRESSURES OF MAJOR SPECIES IN SPECTRUM OF FIGURE 21

Gas	Mass Charge, m/e	True Pressure Torr
H_2	2	1.8×10^{-8}
He	4	5.7×10^{-9}
CH_4	16	8.6×10^{-10}
H_2O	18	1.8×10^{-9}
N_2 or CO	28	5.9×10^{-9}
A	40	1.8×10^{-9}
CO_2	44	1.0×10^{-9}
	TOTAL TRUE PRESSURE	3.5×10^{-8}
	IONIZATION GAUGE PRESSURE	5.5×10^{-8}

The presence of hydrocarbons in the system is evidenced by the characteristic grouping of peaks in certain mass ranges. The CH_4 group between masses 16 and 12 is due to ions CH_4^+, CH_3^+, CH_2^+, CH^+, and C^+. Similarly, the C_2H_6 group contributes peaks between masses 30 and 24; the C_3H_8 group between 44 and 36; the C_4H_{10} group between 58 and 48; the C_5H_{12} group between 72 and 60; the C_6H_{14} group between 86 and 72; the C_7H_{16} group between 100 and 84; and the C_8H_{18} group between 114 and 96. It may be seen in Figure 21 that the analyzer resolves adjacent peaks up to about m/e=50. The origin of these hydrocarbon peaks has not been determined, but they probably arise from heavier molecules which are cracked in high temperature regions of the loop, or perhaps in the analyzer itself.

6. Pre-prototype Loop Design

The major design effort on the Pre-prototype Loop during the past quarter has been the reorientation and repositioning of the loop components to accommodate a change in the boiler length and condenser configuration. Alteration of the condenser configuration also necessitated the re-design of the adjustable radiation shields.

The boiler length was increased from 155 inches to approximately 200 inches following an analysis of the boiler design using a computer program based on the latest 300 KW heat transfer data[6]. The new computer program is more conservative than the one previously used and is based on limiting the permissible heat flux for nucleate boiling.

The major design change during the past quarter was the replacement of the helical tube radiation type condenser with a finned straight tube radiation condenser. The re-design was made to increase the entrance vapor velocity to 400 feet per second which is in the range of interest for advanced space condensers. Longitudinal fins were added to the tube to increase the heat rejection and decrease the required condenser length to prevent the frictional pressure loss from being excessive at the higher vapor velocity. The finned tube condenser design features are given in Table X. Figure 22 is an isometric drawing of the Pre-prototype Loop with the re-designed condenser in its relative position and orientation in respect to the principal loop components.

Several combinations of condenser tube and fin thickness were considered during the design of the Pre-prototype Loop. Welding tests were conducted on three of the combinations, and the specimens are

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[6] Alkali Metals Boiling and Condensing Investigation, Quarterly Progress Report #6, Covering the Period October 1, 1964 through December 31, 1964, NASA Contract NAS 3-2528, May, 1964.

TABLE X

PRE-PROTOTYPE FINNED TUBE CONDENSER

Condensing Temperature	1350°F
Heat Rejection Capacity	30,200 BTU/hr
Length	5.5 Feet
Tube OD	0.625-Inch
Tube Wall Thickness	0.126-Inch
Number of Fins	2
Fin Geometry	0.25-Inch Thick by 4-Inch Wide
Inlet Vapor Velocity	400 Feet Per Second
Potassium Flow Rate	40 Lbs/hr
Calculated Pressure Drop	0.5 psi

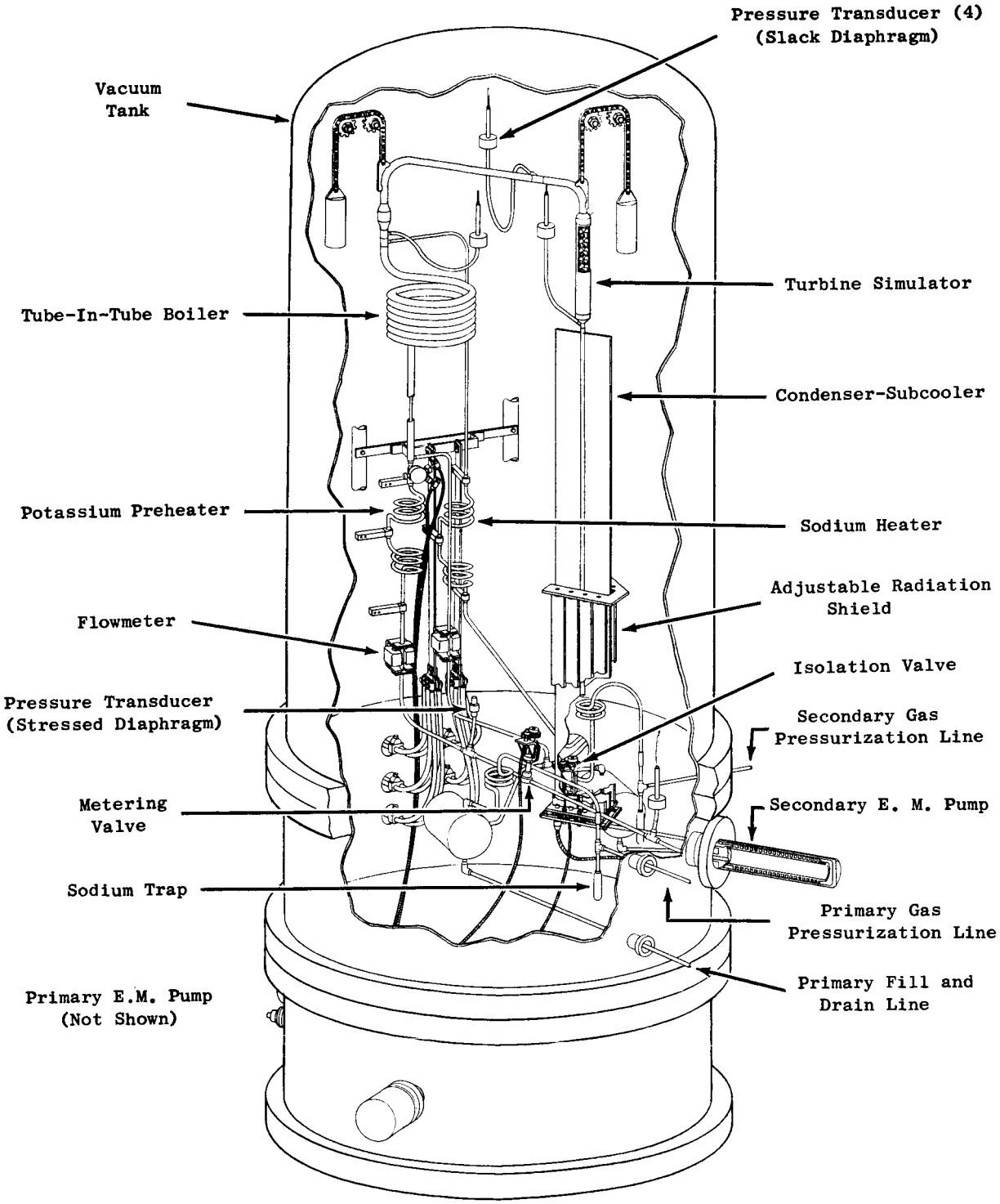


Figure 22. Isometric of the Pre-prototype Corrosion Test Loop.

shown in Figure 23. Configuration (c) in this photograph was the one chosen. A cross section through Specimen C is shown in Figure 24. The distortion resulting from the welding of the various combinations studied are given in Table XI. Minimum and maximum ID's of the 0.394-inch and 0.428-inch, respectively, were found along a one-inch length of Specimen C, the selected configuration for the Pre-prototype Loop.

In order to determine the required surface area for the condenser fin, emittance measurements were made on 0.017-inch sheet specimens of Cb-1Zr having three surface conditions. The specimens were prepared at General Electric and the emittance tests were conducted by T. G. Kollie and D. L. McElroy of the Oak Ridge National Laboratory in a 5×10^{-7} torr environment using techniques which have been described in the literature[7]. The emittance of each specimen was checked by cycling twice between 372° and 1472°F while making the measurements. The detailed results will be covered in the topical report on the Pre-prototype Loop. Photographs showing the surfaces of the specimens and average emittance values obtained at two temperatures are given in Figure 25. Micrographs of cross section views of the pickled and grit blasted surfaces are shown in Figure 26. These results indicated that the coarse mechanical deformation achieved by the dimpling treatment did increase the emittance of the specimen. A more effective and practical method of achieving this gross roughening of the surface is to grit blast with coarse alumina particles prior to grit blasting with the 50-micron alumina. It is planned to use 500-micron alumina for the initial roughening of the surface. This double treatment is expected to yield emittance values of approximately 0.50 at 1350°F. The effect of grit blasting with alumina on the oxygen concentration of Cb-1Zr specimens is discussed later in this report.

In addition to the re-design of the condenser, the adjustable radiation shield was re-designed for the new condenser geometry. The loop support structure and a number of detail drawings of loop components were also completed. The completed drawings of the entire loop assembly will be issued and submitted to NASA for approval during the next quarter.

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7 Kollie, T. G. and McElroy, D. L., "The Total Hemispherical Emittance of Platinum, Columbium-1% Zirconium, and Polished and Oxidized INOR-8 in the Range 100° to 1200°C," Symposium on the Measurement of Thermal Radiation Properties of Solids, NASA-SP-31, September, 1962.

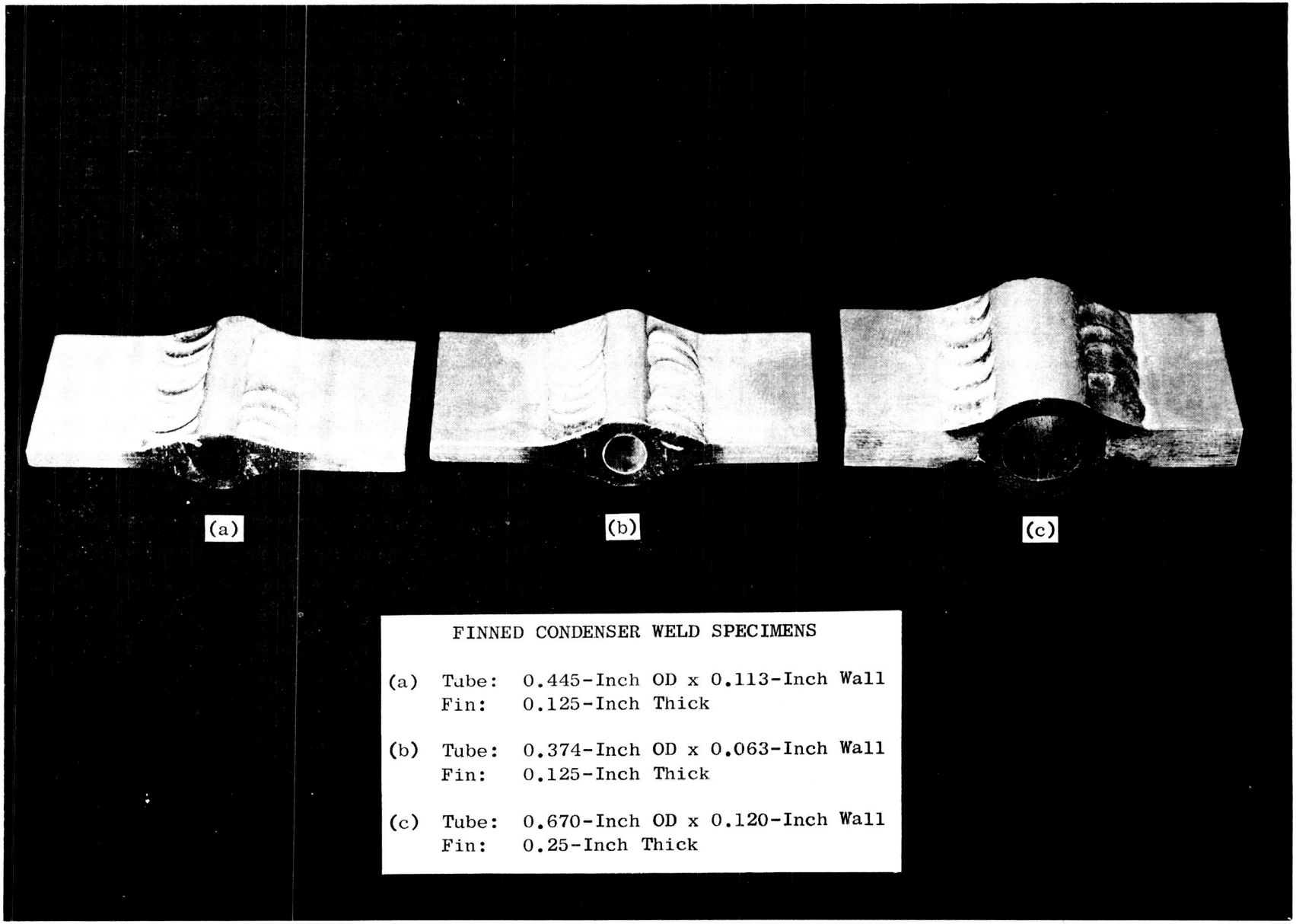


Figure 23. Cb-1Zr TIG Welded Condenser Tube-Fin Specimens. (C64060901)

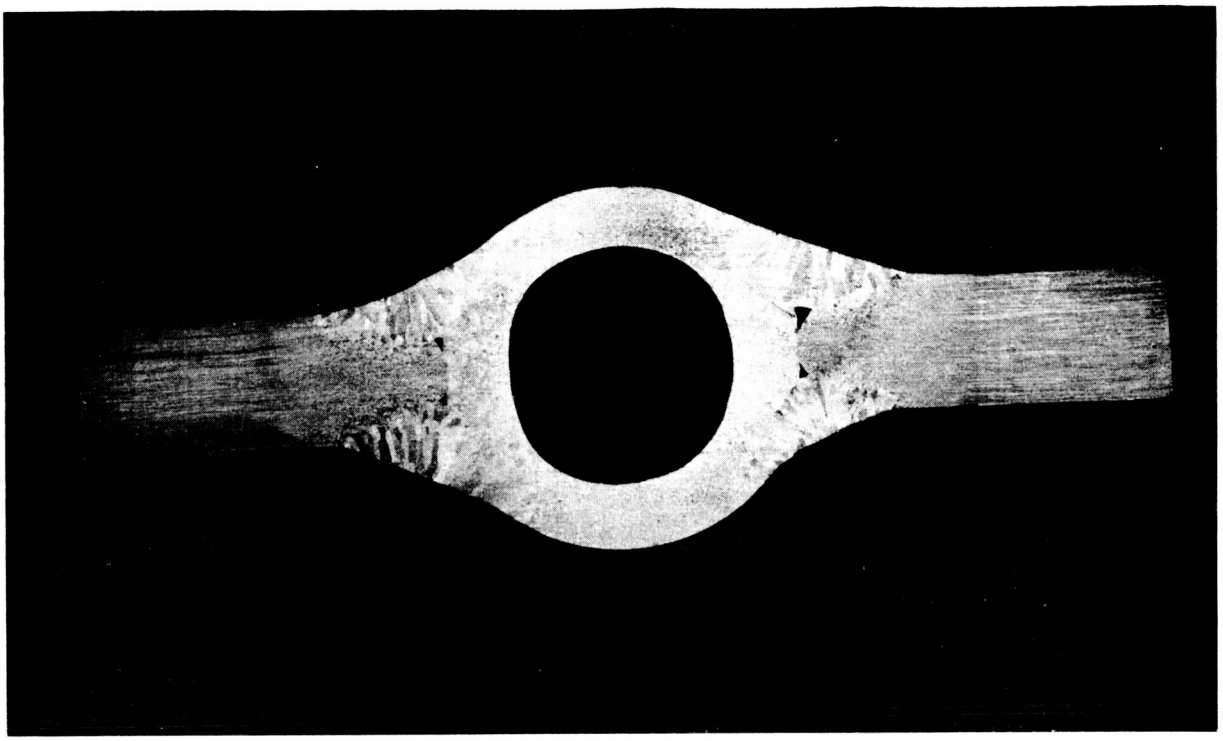
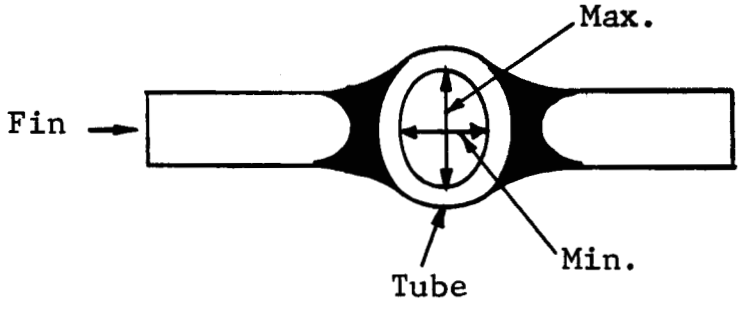


Figure 24. Micrograph of a 0.25-Inch Thick Cb-1Zr Fin TIG Welded to 0.670-Inch OD x 0.120-Inch Wall Cb-1Zr Tube. The ID Distortion Noted is Presented in Table XI.

Etchant: 60%Glycerine-20%HF-20%HNO_3 Mag. 3X (C64060903)

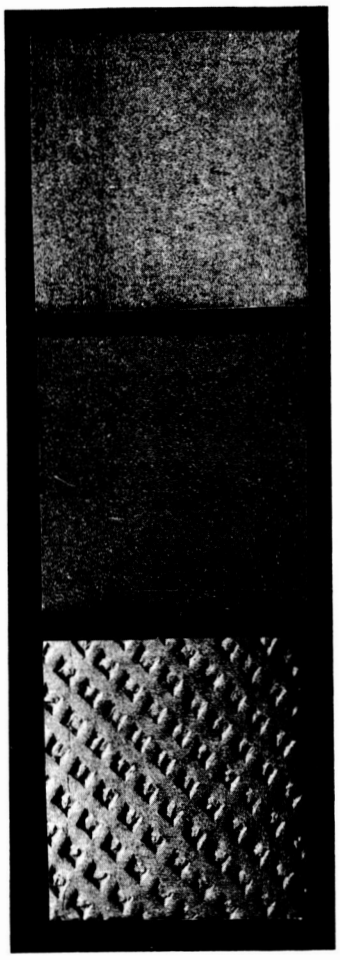
TABLE XI

DISTORTION RESULTS OBSERVED ON Cb-1Zr CONDENSER TUBE-FIN TIG WELDED SPECIMENS



	Tube Dimensions Before Welding, Inch	Tube Dimensions After Welding, Inch*
Specimen A	OD - 0.445	Max. 0.225-0.227
	ID - 0.219	ID
	Fin Thickness 0.125	Min. 0.200-0.201
Specimen B	OD - 0.374	Max. 0.268-0.273
	ID - 0.248	ID
	Fin Thickness 0.125	Min. 0.194-0.197
Specimen C	OD - 0.670	Max. 0.422-0.428
	ID - 0.431	ID
	Fin Thickness 0.25	Min. 0.394-0.400

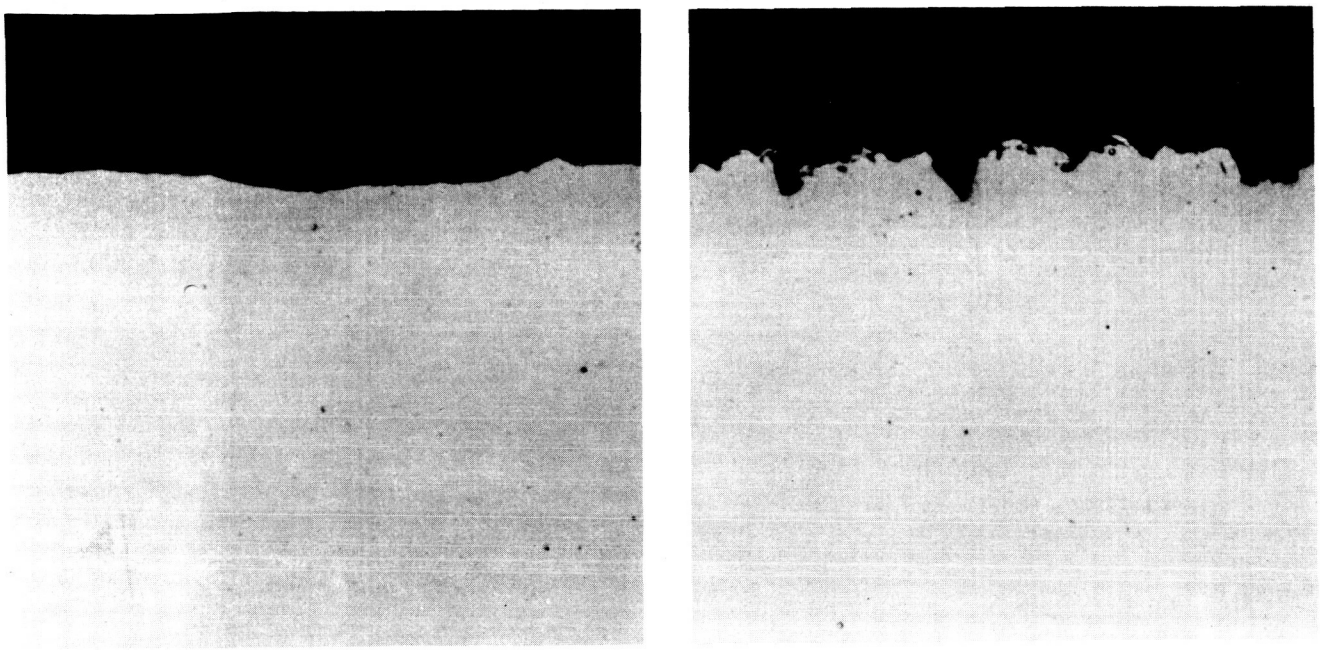
* Maximum Variation in 4 Measurements Taken Along the ID of One-Inch Long of Weld Specimens.



1/2-Inch

Surface Treatment	Total Hemispherical Emittance	
	600°F	1350°F
As-Pickled	0.16	0.21
Pickled and Grit Blasted with 50-Micron Al_2O_3	0.35	0.42
Pickled, Dimpled and Grit Blasted with 50-Micron Al_2O_3	0.38	0.46

Figure 25. Surfaces of Cb-1Zr Emittance Specimens Tested to Determine the Required Surface Area of the Pre-prototype Loop Condenser Fin. (C64060814)



(a) K3654 (b) K3652

Figure 26. Micrograph of Surfaces of Cb-1Zr Emittance Specimens.

a) Before Grit Blasting
b) After Grit Blasting with 50-Micron Al_2O_3 Particles

As Polished Magnification: 1000X

7. Pre-prototype Loop Fabrication

The machining of the primary and secondary EM pump ducts continued during this report period. In view of the difficulties encountered on the Loop II EM pump duct, each outer duct has been stress-relieved at 1800°F for one hour prior to the final honing operations. This treatment was an added precaution to minimize residual stress introduced by machining operations. The scheduled completion date is August 31, 1964.

The boiler prototype which was formed from half-hard copper was received from the forming vendor. A radiograph of one of the copper coils is shown in Figure 27. A dimensional inspection indicated a maximum outer tube eccentricity of plus 0.050-inch and minus 0.080-inch from the nominal 1.002-inch outside diameter. This condition occurred at the ends of the coil section. The coil itself was a uniform plus 0.030-inch, minus 0.037-inch from nominal. This condition is considered acceptable for the design of the Pre-prototype Loop boiler.

The degree of concentricity maintained between the inner and the outer tubes was determined radiographically. The annular spacing, nominally 0.208-inch varied from a minimum 0.15-inch to a maximum 0.22-inch in the coil proper, which is considered acceptable. This condition occurred at the midpoint between spacers. The bends at the end of the coil produced slightly greater eccentricity. No accurate measurements could be obtained, however, since no spacers were located on the free ends of the bends.

This boiler prototype was formed with sugar packed into the annular spacing and the inner tube. After the above inspection, the sugar was dissolved by rinsing with water. An organic micro-analytical technique was used to permit detection of sucrose in the wash water. The validity of this test will be determined by examination for residual sugar after cutting apart the boiler prototype. This destructive evaluation will be conducted after a second radiographic inspection is made to insure a negligible change in tube concentricity as a result of sugar removal.

The six slack diaphragm pressure transducers are now scheduled for NaK filling early in September. A NaK sampler of the type shown in a previous report[8] was constructed and shipped to Taylor Instrument Company for obtaining a NaK specimen at the time of filling the Pre-prototype Loop slack diaphragm transducers.

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8 Potassium Corrosion Test Loop Development, Quarterly Progress Report #3, for Period Ending April 15, 1964, NASA Contract NAS-3-2547, NASA-CR-54091, p 22.

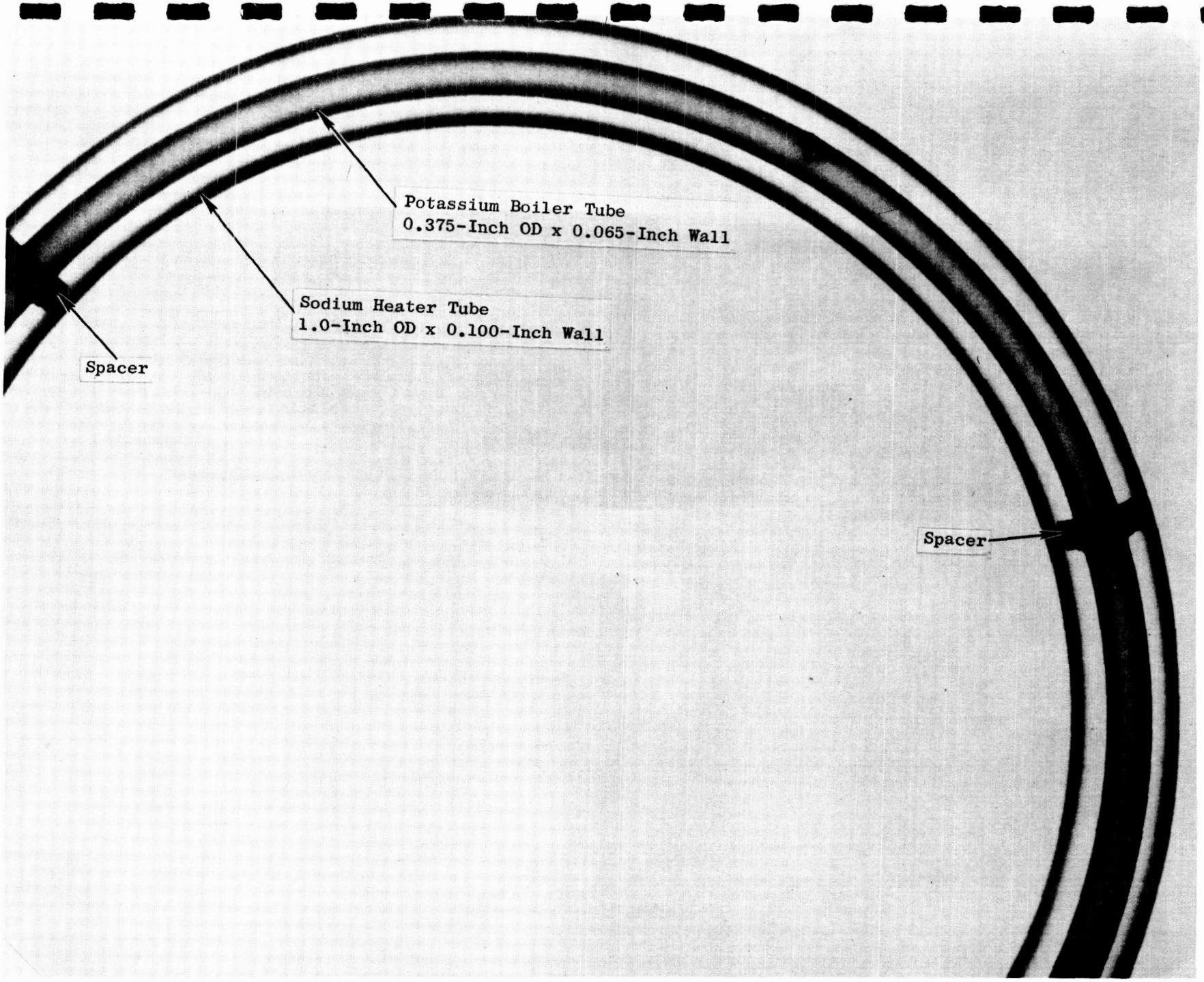


Figure 27. Radiograph of a Copper Mockup of the Pre-prototype Loop Boiler. Packed Sugar was Used to Maintain Concentricity of Tubes During Forming.

8. Alkali Metal Purification and Handling System for the Pre-prototype Loop

The schematic diagram of the potassium side of the Pre-prototype Loop was completed and the procedures were written. Detailed drawings of the 50-pound potassium hot trap, the high vacuum still, the vacuum-argon system, and the portable dolly for these components have been completed. The potassium transfer system drawings are also completed but are currently being revised. The sodium purification system used on Loop II will also be used to prepare sodium for the Pre-prototype Loop primary circuit, but the transfer system will be similar in design to that which will be used for the secondary potassium circuit.

Procurement procedures have been initiated to obtain the parts needed for the Pre-prototype Loop purification and transfer system.

9. Pre-prototype Loop Test Chamber

The 48-inch diameter by 128-inch vacuum chamber for the Pre-prototype Corrosion Loop successfully completed its acceptance test during the past quarter. It had been reported that the system failed to pass its acceptance test due to an air leak which would develop in the 2400 liter per second ion pump during the bakeout of the system. The ion pump was shipped back to the vendor for repairs and returned to General Electric. During transit from the vendor to General Electric the pump received some slight damage to the magnets and their support channels. The pump was leak checked and tested and the damage cited did not appear to have affected the pump performance. The specification test was completed under the supervision of the vendor's field engineer. The pump down of the system appeared normal and compared favorably to the vendor's performance curves obtained during the preliminary testing, prior to shipment of the system to General Electric, where the pressure of 7.5×10^{-11} torr was reached using the titanium sublimation pumps to assist the ion pump. The system reached 4×10^{-10} torr after 60 hours of operation including a 24-hour bakeout without the use of the titanium sublimation pumps and thereby exceed the specification requirement of 1×10^{-9} torr in 100 hours or less.

10. Refluxing Potassium Compatibility Tests

The machining of the test facility for the reflux capsule compatibility test of the Mo-TZM has been completed and construction has been initiated. Fabrication of the capsules has been completed and they are now ready for cleaning and filling with potassium purified by distilling and hot trapping.

11. Material Procurement

Ultrasonic inspection of the 12-foot lengths of Cb-1Zr alloy tubing procured for the tube-in-tube boiler of the Pre-prototype Loop was completed. Two of the three 0.375-inch OD x 0.065-inch thick wall tubes had single defects beyond the specified limit, i.e., 3% of the wall thickness, and the third tube was found to be defect free. The defects were located on the OD of the tubes approximately 17 and 28 inches from the ends. Also, all of the 1.0-inch OD x 0.100-inch thick wall tubes had one or more rejectable defects near the ends of the tubes. Although the re-design of the tube-in-tube boiler now requires approximately 200 inches of tubing, there will be sufficient sound stock after removal of the defects at the ends of the tubing to fabricate each component of the duplex boiler with only a single butt weld. This weld will be well removed from any critical area. Quality assurance testing (chemical analyses, room temperature tensile and 2000°F stress-rupture testing, and grain size determinations) is in progress to establish the final acceptability of the sound section of the tubing.

Continuing improvements in the design of the various components of the Pre-prototype Loop has required additional procurement of refractory alloy mill products. Forty-eight inches of 1.0-inch diameter Mo-TZM alloy were ordered for the fabrication of the turbine simulator blade configuration. This will provide sufficient raw stock to produce the required number of blades plus spares from a single heat of material. Delivery of the Mo-TZM bar is expected by mid-July.

The design change from the coiled radiation type condenser to the finned straight tube condenser necessitated procurement of Cb-1Zr alloy pipe and plate. Thirteen feet of 3/8-inch diameter Schedule 80 (0.675-inch OD x 0.126-inch thick wall) pipe was ordered with the delivery scheduled for the end of August. The fins will be constructed from 2 plates each 0.250 inch thick x 5.0 inches wide x 72 inches long. Delivery of these items is expected early in August.

Partial delivery has been made of the 0.032-inch thick Cb-1Zr alloy sheet ordered for the fabrication of the radiation shield assembly on the condenser/subcooler of the Pre-prototype Loop. The remaining quantity was shipped early in July.

12. Helium Analysis System

In order to properly evaluate the precision and accuracy of the helium analyses, a cylinder of helium has been obtained which contains

10 to 20 ppm of O_2, H_2, CO, and N_2. In addition, a cylinder of ultra-high purity helium has been obtained in which these contaminants are at levels well below 1 ppm.

By examination of the mass spectra obtained from these gases, several effects have been noted. In the first place, loss of oxygen has been observed and this effect seems to be due either to adsorption on the walls of the system or reaction with elements of the system or, possibly, reaction with other constituents of the gas. This effect may be considerably reduced by admission of oxygen into the system at a relatively high pressure before analysis of the sample.

The second observation is that when pure helium is introduced an appreciable increase in the background spectrum is observed, especially for hydrogen. This effect could be due to desorption of gases from the walls of the system.

Several changes in procedures will be necessary in order to obtain reliable analyses of the helium. These are:

1. Calibration of the system against gas of known impurity content rather than against the system ion gauge.

2. Obtain the background spectrum from the introduction of a sample of pure helium rather than from the residual gas mass spectrum.

3. Periodic introduction of oxygen into the system.

It appears very doubtful that reliable analyses for water can ever be obtained with the present system due to its very strong adsorption.

Analyses of a number of cylinders of commercial grade helium have indicated that the as-received gas is not of consistently high purity. Arrangements have now been made to obtain helium with a maximum analyzed impurity content of 2 ppm O_2 and 2 ppm H_2O. This gas will be shipped in cylinders marked and reserved for exclusive use on the corrosion loop program. These steps should assure that the as-received helium will be of high purity. The purification train will, of course, continue to be used to further purify the gas before admission to the welding chamber. This high-purity helium will be available for the Pre-prototype Loop fabrication.

13. Leak Tests on Conoseal Tube Unions

It is intended to use Conoseal* Tube Unions to make and break liquid metal and vacuum-argon lines in certain applications such as the Pre-prototype Loop alkali metal purification, transfer and sampling systems. The leakage of air through such seals must be extremely low to prevent contamination of the alkali metals. A number of tests have been performed in order to determine the air leakage through such seals during temperature cycling and when gaskets are replaced. The tests were performed using the General Electric M-60 helium leak detector. The detection limit of the leak detector at the time of the tests was 4.6×10^{-11} std. cc per second of air.

The procedure was as follows: TIG weld the Conoseal Union members to 0.5-inch OD x 0.035-inch wall stainless steel tubes. Place the gasket in the union and tighten to the recommended torque. Close one end with a Swagelok** plug fitting and connect the other end to the leak detector. Measure the leak rate at room temperature. Heat with heating tapes to the test temperature and remeasure the leak rate. Repeat the leak measurement during temperature cycling. The results of the tests are given in Table XII.

The maximum leak rate observed was 4.5×10^{-10} std. cc of air per second. This is equivalent to a contamination rate of 1.2×10^{-13} grams of oxygen per second or about 3.8 micrograms of oxygen per year. This is a negligible rate since it would take about one thousand years to contaminate ten pounds of alkali metal to a detectable level. Procedures for the proper use of these fittings are given in Specification SPPS-53, "Stainless Steel Conoseal Tube Unions".

Despite these encouraging results, the philosophy of this program is to minimize the use of mechanical seals and to use them only in the low temperature ($< 500^{\circ}F$) handling systems.

14. Effect of Grit Blasting with Alumina on the Oxygen Concentration of Cb-1Zr

Metallographic examination and chemical analyses of Loop I test components have indicated that Cb-1Zr surfaces which have been grit

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* Marmon Division, Aeroquip Corporation

** Crawford Fitting Company

TABLE XII

RESULTS OF THERMAL CYCLING LEAK TESTS ON CONOSEAL* TUBE UNIONS

Test No.	Temperature °F	Leak Rate x 10^{10} Std. cc of Air/Sec	Remarks
1	RT	2.3	
2	480	3.6	
3	RT	2.7	
4	480	4.5	
5	RT	2.7	
6	RT	2.7	New Gasket
7	477	2.7	
8	RT	1.8	
9	500	1.8	
10	RT	3.2	
11	RT	3.6	New Gasket
12	380	4.1	
13	RT	2.7	Bagged and He Added
14	400	3.2	
15	RT	1.4	

* Marmon Division, Aeroquip Corporation

blasted* with alumina (Al_2O_3) in accordance to specification SPPS-12, "Grit Blasting Columbium and Columbium Alloy Products", contain particles of embedded alumina. Since it will be of interest to determine the interstitial (particularily oxygen) concentration gradient in loop components, a test was conducted to determine the extent of oxygen increase that results from grit blasting with alumina powder.

A Cb-1Zr disc, 0.0175-inch thick by 0.3125-inch diameter was grit blasted on one side for about 20 seconds at which time the visual appearance of the specimen ceased to change. After grit blasting, the specimen was washed in water and rinsed in ethyl alcohol. The oxygen concentration of the specimen before grit blasting was 174 ppm and 1016 ppm afterwards, indicating a net increase of 842 ppm. This would result in an apparent oxygen increase of 234 ppm in a 0.065-inch thick tube wall.

Chemical analyses of various zones of the tube wall and microhardness traverses of the tube wall of Loop I indicate that no significant diffusion of oxygen into the tube wall occurred as a result of residual alumina particles from the grit blasting operation. Many of the alumina particles are embedded in the surface during grit blasting and the triangular particle evident near the surface in the micrograph shown in Figure 26B is typical. The sections examined operated at 2000°F for 1,000 hours.

In the future grit blasting with alumina will not be performed on areas where oxygen analyses will be required or on surfaces which will be operated in excess of 1800°F.

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* The grit blasting treatment is required to increase the emittance of surfaces where heat rejection by radiation must be high.

III FUTURE WORK

A. Operation of Loop II will be continued and approximately 2,000 hours of the 2,500 hour test will be completed.

B. The completed drawings of the entire Pre-prototype Loop assembly will be issued and submitted to NASA for approval during the next quarter.

C. Installation of the auxiliary operation and control equipment for the Pre-prototype Loop and test chamber will be completed.

D. Design of purification, transfer and sampling systems for the sodium and potassium to be used in the primary and secondary circuits, respectively, of the Pre-prototype Loop will be completed. Procurement of components and initiation of fabrication will also begin.

E. Six slack diaphragm pressure transducers will be filled with high-purity NaK by the vendor.

F. Machining procedures required for the manufacture of the Pre-prototype loop turbine simulator will be established and fabrication initiated.

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