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MATERIALS FOR POTASSIUM LUBRICATED JOURNAL BEARINGS

Quarterly Progress Report No. 5
For Quarter Ending July 22, 1964

EDITED BY R. G. FRANK

prepared for
NATIONAL AERONAUTICS AND SPACE ADMINISTRATION
CONTRACT NAS 3-2534

SPACE POWER AND PROPULSION SECTION
MISSILE AND SPACE DIVISION
GENERAL ELECTRIC
CINCINNATI 15, OHIO

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QUARTERLY PROGRESS REPORT NO. 5

Covering the Period
April 22, 1964 to July 22, 1964

edited by

R. G. Frank
Program Manager

approved by

J. W. Semmel, Jr.
Manager, Materials and Processes

prepared for

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

Contract NAS 3-2534

Technical Management
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SPACE POWER AND PROPULSION SECTION
MISSILE AND SPACE DIVISION
GENERAL ELECTRIC COMPANY
CINCINNATI, OHIO, 45215

Foreword

The work described herein is being performed by the General Electric Company under the sponsorship of the National Aeronautics and Space Administration under Contract NAS 3-2534. Its purpose, as outlined in the contract, is to evaluate materials suitable for potassium lubricated journal bearing and shaft combinations for use in space system turbogenerators and, ultimately, to recommend those materials most appropriate for such employment.

R. G. Frank, Manager, Physical Metallurgy, Materials and Processes, is administering the program for the General Electric Company. L. B. Engel, Jr., D. N. Miketta, T. F. Lyon, W. H. Hendrixson and B. L. Moor are directing the program investigations. The design for the friction and wear tester is being executed by H. H. Ernst and B. L. Moor.

R. L. Davies of the National Aeronautics and Space Administration is the technical manager for this study.

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I. INTRODUCTION

The program reviewed in this fifth quarterly report, covering activities from April 22, 1964 to July 22, 1964, is performed under the sponsorship of the National Aeronautics and Space Administration. Its purpose is to evaluate materials suitable for potassium lubricated journal bearing and shaft applications in space system turbogenerators operating over a 400°F to 1600°F temperature range. The critical role of bearings in such systems demands the maximum reliability attainable within today's state-of-the-art. Achieving this reliability requires an interdisciplinary approach employing the best mechanical designs of journal bearings combined with the selection of the optimum materials to serve as the structural members. Satisfying this latter requirement constitutes the aim of this program.

A number of investigators have conducted studies in this field and their contributions have advanced the state-of-the-art considerably (Section VIII, Ref. 1). Although their work is significant, there are no common criteria for a comparison of the existing data. Therefore, establishing a unified approach to the development and evaluation of materials for potassium lubricated bearing application is deemed essential. The program involves a comprehensive investigation of material properties adjudged requisite to reliable journal bearing operation in the proposed environment. This includes: 1) corrosion testing of individual materials and potential bearing couples in potassium liquid and vapor, 2) determination of hot hardness, hot compressive strength, modulus of elasticity, thermal expansion and dimensional stability characteristics, 3) wetting tests by potassium and 4) friction and wear measurements of selected bearing couples in high vacuum and in liquid potassium.

In cooperation with the cognizant NASA Technical Manager, 14 candidate materials were selected (Table I) from a compilation of existing data on available materials. The materials reviewed fall into four broad categories:

- Superalloys and refractory alloys with and without surface treatment.
- Commercial metal bonded carbides.
- Refractory compounds such as stable oxides, carbides, borides and nitrides.
- Cermets based on the refractory metals and stable carbides.

Each material is procured from appropriate suppliers to mutually acceptable specifications and subsequently is subjected to chemical, physical and metallurgical analyses to document its characteristics before utilization in the program. After the documentation of processes and properties, the candidate materials undergo corrosion, dimensional stability, thermal expansion, compression and hot hardness testing. Considering the bearing material requirements and the preliminary information obtained on materials subjected to both potassium and non-potassium testing, a number of materials combinations will be selected in cooperation with and subject to the approval of the NASA Technical Manager. Potassium corrosion and wetting tests and friction and wear measurements in high vacuum and liquid potassium will then proceed with these combinations.

The ultimate product of this program will be a recommendation, substantiated with complete documentation, of the material or materials which have the greatest potential for use in alkali metal journal bearings in high speed, high temperature, rotating machinery for space applications. Hopefully, the results will indicate the future course of alloy or material development specifically designed for alkali metal lubricated journal bearing and shaft combinations.

TABLE I: CANDIDATE BEARING MATERIALS

Material Class	Candidate Material
A. Nonrefractory Metals and Alloys	Star J (17%W-32%Cr-2.5%Ni-3%Fe-2.5%C-Bal. Co)
B. Refractory Metals and Alloys	Mo-TZM (0.5%Ti-0.08%Zr-Bal. Mo) W
C. Fe-Ni-Co Bonded Carbides	Carboloy 907 (74%WC-20%TaC-6%Co) Carboloy 999 (97%WC-3%Co) K601 (84.5%W-10%Ta-5.5%C)
D. Carbides	TiC
E. Oxides	Lucalox (Al_2O_3) Zircoa 1027 (ZrO_2)
F. Borides	TiB_2
G. Refractory Metal Bonded Carbides	TiC+5%W TiC+10%Mo TiC+10%Cb Grade 7178

II. SUMMARY

During the fifth quarter of this program, the topics abstracted below were covered and the results are interpretatively presented in this report.

Partial shipment of test specimens of each specimen type were received during the report interim. The procurement status of the test specimens is: corrosion, 93% complete; dimensional stability, 66% complete; thermal expansion, 54% complete; hot hardness, 71% complete; compression, 7% complete.

Thirteen pounds of potassium were purified for the isothermal capsule corrosion tests by vacuum distillation followed by hot trapping in a titanium lined, zirconium gettered hot trap. Chemical analyses after purification show the majority of the metallic impurities to be below the detectable limits and the oxygen content to be less than 10 ppm.

Three separate checkout tests were performed with the isothermal capsule corrosion test facility in the 10^{-9} torr range. The mean temperature deviations were found to be $\pm$ 0.74% at a mean test temperature of 1628°F; $\pm$ 1.40% at a mean test temperature of 1200°F; and $\pm$ 2.13% at a mean test temperature of 809°F. Three sets of 6 Cb-1Zr alloy capsules containing 2 specimens of Carboloy Grades 907 and 999, Lucalox, ZrO_2, Mo-TZM and tungsten, respectively, were filled with potassium and tested isothermally for 1,000 hours at 1600°, 1200° and 800°F. The chamber pressure at the end of the test was 9.9 x 10^{-10} torr. Thirty-four additional capsules have been prepared and are ready for filling with potassium.

Two checkout tests were conducted with the dimensional stability test facility in the 10^{-9} torr range. The mean temperature deviations were found to be $\pm$ 0.49% at a mean temperature 1603°F; $\pm$ 0.62% at a mean temperature of 1202°F and $\pm$ 0.83% at a mean temperature of 757°F. Duplicate test specimens for 10 materials were placed on test at 1600°F and 1200°F. At the end of this reporting period the 1600°F test had accumulated 540 hours and the 1200°F test had accumulated 440 hours. The pressure at that time was 8.5 x 10^{-9} torr.

Checkout tests on the thermal expansion and hot hardness facilities indicate that the test environments are suitable for the evaluation of all materials in the current program. The test programs will be initiated in the next report interim.

The design of the liquid potassium friction and wear tester was reviewed extensively during the reporting period and, with the

exception of the potassium heater, all drawings have been finalized and approved by the NASA Technical Manager. Purchase orders for the construction of the various components of the test rig were released and fabrication should be completed by the end of November, 1964.

III. MATERIALS PROCUREMENT

Delivery of the various candidate bearing material specimens continued throughtout the report period. With the exception of TiB_2, either complete or partial shipments of each specimen type were received for all of the materials. Shipment of specimens of the TiB_2 material has been delayed until early August. Table II summarizes the procurement status for the fourteen materials as of July 22, 1964. The majority of the vendors have had only minor difficulties in producing the configurations required for the corrosion, dimensional stability, thermal expansion and hot hardness specimens. Rejection of a greater number of samples in final inspection resulted in slippages of the promised delivery dates for these specimens. It was necessary to process additional specimen blanks in order to produce the required number of acceptable test specimens. The compression specimens (See Figure 1, Reference 4), with their relatively larger mass and more intricate shape, have presented considerably more problems in fabrication. As of July 22, 1964, a complete set of compression specimens had been received of only one material, i.e., the Mo-TZM alloy.

The corrosion and dimensionsal stability test specimens for six of the candidate bearing materials were received early in the quarter. The six materials involved were Carboloy Grade 999, Carboloy Grade 907, Lucalox, Zircoa 1027, Mo-TZM alloy and unalloyed tungsten. The specimens machined from the Mo-TZM alloy and unalloyed tungsten bar stock were penetrant inspected at General Electric using the post-emulsification process (AMS 2645) and minor edge chipping was found on several specimens of each material. Six corrosion test specimens from each of four of the candidate materials, i.e., Carboloy Grade 999, Carboloy Grade 907, Lucalox and Zircoa 1027, were released on May 1, 1964 for the necessary pretest cleaning, weighing and dimensional measurement operations. A similar number of corrosion test specimens of the Mo-TZM alloy and unalloyed tungsten were released on May 6, 1964.

Two visits were made to Kennametal, Inc., Latrobe, Pennsylvania, to expedite the processing of the corrosion and dimensional stability specimens of the six materials being supplied by them and to discuss their overall progress and future delivery dates. In reviewing the work done on the lot of TiC, required for the fabrication of the pure TiC, and the refractory metal bonded TiC test specimens, it was pointed out that the TiC had picked-up a surprisingly large amount of WC (3-5%) during the milling operation. The milling operation is carried out in a WC lined container utilizing WC balls. Efforts by the vendor to eliminate this condition have been unsuccessful.

Milling the TiC in steel containers resulted in the contamination

TABLE II

PROCUREMENT STATUS OF THE CANDIDATE BEARING MATERIALS TEST SPECIMENS - 7-22-64

			Corrosion			Dimensional Stability			Test Specimen Identity Thermal Expansion			Hot Hardness			Compression			
Material	Specification	Vendor	P.O. No.	Date Rec'd/ Promised	Quantity	P.O. No.	Date Rec'd/ Promised	Quantity	P.O. No.	Date Rec'd/ Promised	Quantity	P.O. No.	Date Rec'd/ Promised	Quantity	P.O. No.	Date Rec'd/ Promised	Quantity	MCN Series
Carboloy 999	SPPS-24T	GE-MPD	037-121815	4-29-64	17	037-121815	4-29-64	8	037-122247	6-10-64	3	037-122247	6-10-64	3	037-122247	8-17-64	10	1035
Carboloy 907	SPPS-23T	GE-MPD	037-121815	4-29-64	18	037-121815	4-29-64	11	037-122247	6-10-64	3	037-122247	6-10-64	3	037-122247	8-17-64	10	1036
Mo-TZM (Raw Stock)	SPPS-15	Amer. Metal Climax, Inc.	037-121885	3-6-64	0.437" ø x 48"	037-121885	3-6-64	1.25" ø x 36"	037-122249	5-5-64	0.187" ø x 12"	037-122249	5-5-64	0.750" ø x 12"	037-122249	5-5-64	1.56" ø 36"	--
Mo-TZM (Machining)	--	Dawson Carbide Industries, Inc.	037-124116	4-29-64	16	037-124116	4-29-64	10	037-122284	6-30-64	3	037-122284	6-30-64	3	037-122284	7-6-64	10	1037
Tungsten (Raw Stock)	SPPS-40T	Universal Cyclops Steel Corp.	037-122059	4-1-64	0.437" ø x 48"	037-122059	4-1-64	1.25" ø x 36"	037-122249	6-10-64	0.187" ø x 12"	037-122249	6-5-64	0.750" ø x 12"	037-122249	8-14-64	1.56" ø x 36"	--
Tungsten (Machining)	--	Dawson Carbide Industries, Inc.	037-124116	4-29-64	16	037-124116	4-29-64	10	037-122284	6-30-64	3	037-122284	6-30-64	3	037-122284	2 wks after receipt of mat'l	10	1038
Lucalox	SPPS-29T	GE-Lamp Glass	037-121835	5-4-64	16	037-121835	5-4-64	12	037-122243	7-7-64	2 1	037-122243	7-27-64	3	037-122243	8-21-64	10	1039
Zircoa 1027	SPPS-31T	Zirconium Corp. of America	037-121868	5-4-64 6-12-64	11 5	037-121868	5-4-64	10	037-122248	6-12-64	3	037-122248	6-12-64	3	037-122248	8-14-64	10	1040
K601	SPPS-25T	Kennametal, Inc.	037-122216	6-10-64	17	037-122216	6-10-64 7-22-64	8 2	037-122245	7-22-64 8-19-64	2 1	037-122245	6-10-64	3	037-122245	8-19-64	10	1041
TiC	SPPS-27T	Kennametal, Inc.	037-121842	6-10-64 8-19-64	14 2	037-121842	6-10-64 7-22-64	9 1	037-122244	8-19-64	3	037-122244	7-22-64	3	037-122244	8-19-64	10	1042
TiC + 5%W	SPPS-33T	Kennametal, Inc.	037-121842	7-22-64	16	037-121842	7-22-64	10	037-122244	8-19-64	3	037-122244	8-19-64	3	037-122244	8-19-64	10	1043
TiC + 10%Mo	SPPS-34T	Kennametal, Inc.	037-121842	7-22-64	10	037-121842	7-22-64 8-19-64	8 2	037-122444	8-19-64	3	037-122244	7-22-64	3	037-122244	8-19-64	10	1044
TiC + 10%Cb	SPPS-35T	Kennametal, Inc.	037-121842	6-10-64 7-22-64	6 10	037-121842	6-10-64 8-19-64	4 6	037-122444	8-19-64	3	037-122244	8-19-64	3	037-122244	8-19-64	10	1045
Grade 7178	SPPS-37T	Kennametal, Inc.	037-122257	6-10-64 7-22-64	12 4	037-122257	6-10-64 7-22-64	9 1	037-122257	6-10-64	3	037-122257	6-10-64	3	037-122257	9-1-64	10	1046
Star J	SPPS-18T	Stellite Division, UCC	037-121822	6-19-64	16	037-121822	9-1-64	10	037-122248	9-1-64	3	037-122248	7-16-64	3	037-122248	9-1-64	10	1047
TiB_2	SPPS-37T	Norton Co.	037-121867	8-11-64	16	037-121867	8-31-64	10	037-122251	8-31-64	3	037-122251	8-31-64	3	037-122252	9-11-64	10	1048

of the TiC by iron which could not be removed chemically without seriously affecting the quality of the TiC. Although the WC could be eliminated by using a TiC lined mill and TiC balls, the cost would be prohibitive for this program. Therefore, a decision was made to accept the TiC with the WC impurity with the understanding that the columbium, molybdenum and tungsten bonded TiC materials would be processed from this master batch of TiC powder and would, therefore, contain similar quantities of the WC as an impurity. In the case of the tungsten bonded TiC, it was agreed to add the tungsten metal in the full 5% amount as originally planned.

Radiographic inspection of the initial group of TiC+10%Mo specimens revealed a void near the centerline of the majority of the specimens. This condition was tentatively attributed to possible oxygen pick-up by the molybdenum powder during the milling operation and the resultant formation of MoO_3 during the vacuum sintering.

Sound specimens of the TiC+10%Mo composition have since been produced by the vendor by using a coarser grade of molybdenum powder and pre-sintering the green compacts at a temperature of 1300°F before the final sintering operation. Radiographic inspection of the specimens was performed in the pre-sintered state so that defective specimens could be discarded before proceeding with the final sintering and grinding operations.

By early June, dimensional stability test specimens were received for 10 of the candidate materials and, on June 12, 1964, four dimensional stability specimens of each of the 10 materials were released for test. The materials included Carboloy Grade 999, Carboloy Grade 907, Lucalox, Zircoa 1027, Mo-TZM alloy, unalloyed tungsten, K-601, TiC, TiC+10%Cb and Grade 7178. An additional specimen of each of the Carboloy Grade 907, Mo-TZM alloy and the Lucalox materials was given a heat treatment of 1 hour at 1800°F in a vacuum of 1×10^{-5} torr prior to their release for inclusion in the group of specimens to be tested at 1600°F. The purpose of the vacuum heat treatment is to establish any advantageous effect a pre-test thermal treatment might have on the dimensional stability of the materials by relieving possible machining stresses induced by the final grinding operation.

With the approach of the completion of the first 1,000-hour corrosion test in mid-July, additional specimens were released for pretest preparations for the second test run. Six specimens each of K-601, TiC, TiC+10%Cb, Grade 7178 and Star J were released on July 7, 1964 and six specimens of TiC+5%W and two specimens of TiC+10%Mo were released on July 22, 1964.

In general, the vendors have been co-operative in the documentation of the material histories. Samples of the various elemental constituents and of the blended powders were taken, identified and stored in sealed vials. Complete processing data were recorded in the vendor's files and pertinent data provided with the various specimens. As material data accumulates, a foremat will be developed for presentation of the information of the various materials. Each vendor has expressed a willingness to provide processing information within the bounds of proprietary interests.

In addition to the data supplied with the specimens by the vendors, further quality assurance testing of the as-received material is being performed at General Electric. The data being obtained as part of the quality assurance program includes: micro-structural analyses, hardness, chemical analyses, surface roughness, random density measurements and, in the case of the compression specimens, ultrasonic inspection. An extra Mo-TZM alloy compression specimen will be notched to serve as a ultrasonic standard. Three notches, each 0.0038-inch deep x 0.003-inch wide by 0.500-inch long, will be machined in the reduced section of the specimen. One notch will be parallel to the long axis of the specimen, the second notch will be perpendicular to the first notch and one quarter the distance around the gauge section and the third notch will be inscribed in the radius of the reduced section. Transverse and longitudinal shear wave techniques will be used in the inspection of each compression specimen.

Each vendor has provided a metallographic specimen of their material. The specimens were polished according to vendor recommended polishing and etching procedures. These specimens will be used for comparative purposes in the preparation of metallographic samples of the as-received materials and those tested specimens requiring metallographic examination as a basis of evaluation.

At the end of the report interim, the overall procurement status, based on the total number of specimens ordered, was approximately 62% complete. The percentage completion by specimen type is summarized in the following paragraphs:

1) Corrosion Test Specimens - 93% complete. The delivery of 2 make-up specimens of TiC and the full set of 16 TiB_2 specimens early in August will complete the procurement of this configuration.

2) Dimensional Stability Specimens - 66% complete. Two specimens of TiC+10%Mo, 6 of TiC+10%Cb and 10 of both the Star J and TiB_2 specimens are due early in August. Delivery of the Star J samples is still in question. Three attempts have been made to fabricate the specimens from the Star J material and each attempt has met with rejection of the entire lot of each run. Apparently the heavy section size of the specimen configuration causes large thermal stresses to be induced during the finish grinding operation and results

in surface checking or cracking. The vendor has tried variations in stress-relieving and grinding techniques with little success to date.

3) Thermal Expansion Specimens - 54% complete. One sample of K-601 and 3 each of TiC, TiC+5%W, TiC+10%Mo, TiC+10%Cb, Star J and TiB_2 are required to complete procurement of this configuration. The majority of the materials is scheduled for delivery early in August.

4) Hot Hardness Specimens - 71% complete. Delivery of 3 specimens of each of the following materials is required: Lucalox TiC+5%W, TiC+10%Cb and TiB_2. The Lucalox specimens have been promised for the last week of July. The remaining specimens should be delivered by mid-August.

5) Compression Specimens - 7% complete. The only finished specimens received by the end of the reporting interim were machined from wrought bar of Mo-TZM alloy. Fabrication of the compression specimens from the unalloyed tungsten material was delayed because of processing problems encountered by the raw stock vendor in attempting to produce the 1.56-inch diameter stock. The initial extrusion cracked during rolling. A second extrusion was processed and the vendor feels confident that the required bar stock can be produced by early August. The machining vendor has promised delivery two weeks after receipt of the 1.56-inch diameter bar. The majority of the specimens produced by powder processes are promised in the latter part of August.

Each material has been assigned a material control number (MCN) and each individual test specimen is identified within the material control number by a letter code to indicate the type of test, i.e., A - corrosion specimens, B - dimensional stability specimens, C - thermal expansion specimens, etc., and a chronological serial number. The original material control number of each material will be assigned to all future specimens of that material. Table III summarizes the MCN identification of those specimens received by the end of the report interim.

TABLE III

IDENTIFICATION OF CANDIDATE BEARING MATERIAL TEST SPECIMENS

Material	MCN Series	Corrosion Quantity	Corrosion Identities	Dimensional Stability Quantity	Dimensional Stability Identities	Thermal Expansion Quantity	Thermal Expansion Identities	Hot Hardness Quantity	Hot Hardness Identities	Compression Quantity	Compression Identities
Carboloy 999	1035	17	MCN 1035-A- 1 Thru 17	10 (1)	MCN 1035-B- 1 Thru 8	3	MCN 1035-C- 1 Thru 3	3	MCN 1035-D- 1 Thru 3	0	---
Carboloy 907	1036	18	MCN 1036-A- 1 Thru 18	11	MCN 1036-B- 1 Thru 11	3	MCN 1036-C- 1 Thru 3	3	MCN 1036-D- 1 Thru 3	0	---
Mo-TZM	1037	16	MCN 1037-A- 1 Thru 16	10 (2)	MCN 1037-B- 1 Thru 10	3	---	3	---	10	---
Tungsten	1038	16 (3)	MCN 1038-A- 1 Thru 16	10 (4)	MCN 1038-B- 1 Thru 10	3	---	3	---	0	---
Lucalox	1039	16	MCN 1039-A- 1 Thru 16	12	MCN 1039-B- 1 Thru 12	3	---	0	---	0	---
Zircoa 1027	1040	16	MCN 1040-A- 1 Thru 16	10	MCN 1040-B- 1 Thru 10	3	MCN 1040-C- 1 Thru 3	3	MCN 1040-D- 1 Thru 3	0	---
K-601	1041	17	MCN 1041-A- 1 Thru 17	10	MCN 1041-B- 1 Thru 8	2	---	3	MCN 1041-D- 1 Thru 3	0	---
TiC	1042	14	MCN 1042-A- 1 Thru 14	10	MCN 1042-A- 1 Thru 9	0	---	3	---	0	---
TiC + 5%W	1043	16	---	10	---	0	---	0	---	0	---
TiC + 10%Mo	1044	16	---	8	---	0	---	3	---	0	---
TiC + 10%Cb	1045	16	MCN 1045-A- 1 Thru 6	4	MCN 1045-B- 1 Thru 4	0	---	0	---	0	---
Grade 7178	1046	16	MCN 1046-A- 1 Thru 12	10	MCN 1046-B- 1 Thru 9	3	MCN 1046-C- 1 Thru 3	3	MCN 1046-D- 1 Thru 3	0	---
Star J	1047	16	---	0	---	0	---	3	---	0	---
TiB_2	1048	0	---	0	---	0	---	0	---	0	---
TOTALS		210	224 - On Order	115	140 - On Order	23	42 - On Order	30	42 - On Order	10	140 - On Order

(1) Zyglo defects - 2 specimens: MCN 1035-B-9 and 10.
(2) Zyglo defects - 1 specimen: MCN 1037-B-10.
(3) Zyglo defects - 2 specimens: MCN 1038-A-15 and 16.
(4) Zyglo defects - 1 specimen: MCN 1038-B-10.

IV. TEST FACILITIES

Potassium Purification

During this report period, 13 pounds of high purity potassium metal were processed through the purification train and chemical analyses of various samples of this material were performed. The apparatus used is described in detail in Quarterly Progress Report No. 2 (Ref. 2). The apparatus consists of a transfer container, a high vacuum still, condenser and receiver, a titanium-lined, zirconium-gettered hot trap and a high vacuum pumping station as shown in Figure 1. Except for the hot trap liner and getter, all parts which contact liquid potassium are made of 300 series stainless steels. The receiver is fitted with a glass viewport flanged on with a copper, shear-type seal. The glass and copper do not contact the liquid metal at any time.

Pressure in the still and manifold was monitored with ionization gauges and the pressure in the hot trap was monitored with a Bourdon type vacuum-pressure gauge. Pure argon was used in the system until all leak checking had been completed, after which purified helium was used. The helium was purified by passing it through a previously regenerated 13X molecular sieve at room temperature, and then through titanium turnings at 1450°F. Helium purified in this fashion consistently shows less than 1 ppm each of oxygen and water.

The liquid metal levels in the purification system were determined in the following manner:

1) Transfer Container - By an O-ring sealed thermocouple well which can be moved up and down to make or break electrical contact with the liquid metal surface.

2) Still - Calculated from quantities of potassium that were transferred from the transfer container and collected in the receiver; and by temperature profiles obtained from the thermocouple well in the still pot.

3) Receiver - By visually observing the liquid level through the sight port before and after transfer to the hot trap.

The procedures used for the preparation and checkout of the purification system and in purifying the potassium were as follows:

1) Fabricate, outgas and helium leak check all components. No leaks in excess of 5×10^{-10} std. cc. of air per second were permitted.

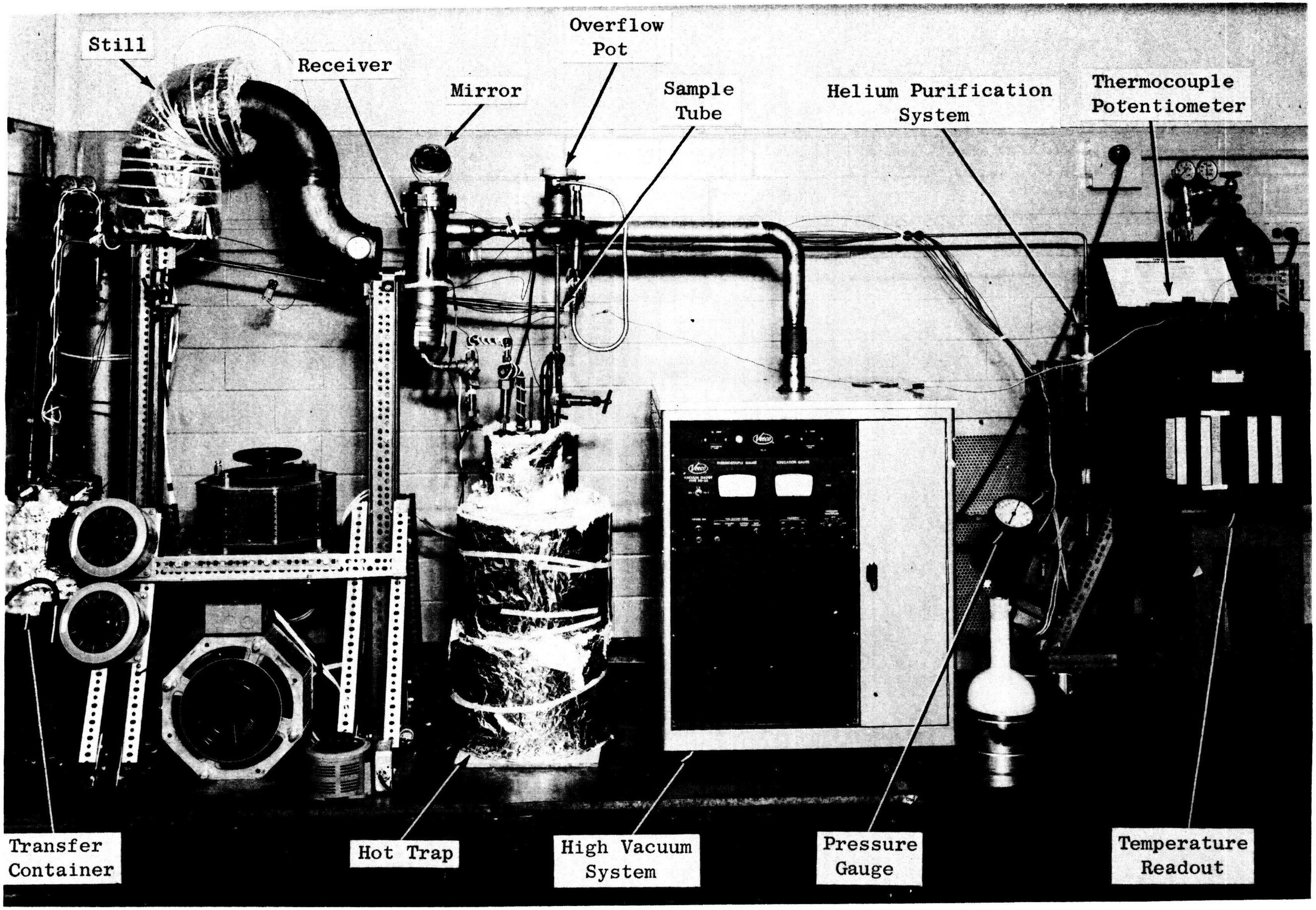


Figure 1. Potassium Purification System. (C64051946)

2) Assemble components and again outgas and leak check. The system was leak checked at room temperature and at temperatures in excess of 300°F.

3) The potassium was transferred from the shipping container (Drum D-80) to the transfer container and then outgassed for approximately 2 hours at temperatures up to 450°F. Most of the gas evolution took place between 315° and 385°F.

4) The still, condenser, receiver, hot trap and transfer lines were evacuated, brought to temperature and potassium transferred to the evacuated still by virtue of the cover gas pressure (about 18 psia) in the transfer container. Transfers to the pot were made in batches of 500 to 800 grams during distillation. The pressure obtained in the still just prior to admitting the potassium was 2.7×10^{-6} torr, the pressure rise rate was 0.6×10^{-3} torr/hour and the outgassing rate was 9×10^{-3} torr-liters/hour.

5) The still temperature was increased to 520° - 535°F and the potassium distilled at a rate of about two pounds per hour. The receiver pressure varied between 1.3 and 5.2×10^{-5} torr during distillation. The potassium was heated from the sides of the still. A temperature gradient was maintained from the top to the bottom with the potassium temperature at the surface about 10°F higher than at the bottom to assure surface evaporation.

6) The receiver was filled to the bottom of the condenser outlet tube and then drained to the weld between the pipe and pipe cap so that the same quantity of potassium was drained into the hot trap each time. A typical temperature profile through the system during distillation was:

Location	Temperature, °F
Transfer Container, Inside	255
Transfer Container, Valve	263
Transfer Container, Filter	252
Still, Valve	229
Still, Bottom	524
Potassium, Bottom	528
Potassium, Top	537
Condenser, Top	360
Condenser, Middle	317
Condenser, Bottom	398
Condenser, Outlet	198

Location	Temperature, ^{o}F
Receiver, Top	84
Receiver, Bottom	133
Receiver, Inside	135
Receiver, Valve	115
Hot Trap, Valve	165
Hot Trap, Inside	254

7) In order to melt the potassium for transfer to the hot trap, the receiver was heated to 160°F. The residual potassium, left in the receiver at room temperature for two months, (with the manifold valve to the receiver closed), has shown absolutely no change in appearance; the surface has remained silvery and mirror-like.

8) After distillation, the hot trap was pressurized to about 3 psig with helium and the potassium was hot trapped for 36 hours between 1350° and 1400°F.

Potassium samples were obtained before, during and after purification. All samples were analyzed fcr oxygen by the mercury amalgamation method and appropriate samples also were spectrographically analyzed for metallic impurities. The analytical results, presented in Table IV, indicate that, with the exception of calcium, the metallic impurity contents changed very little during purification, with the majority of the elements being below the detectable limits. The data for oxygen indicate a significant reduction in the oxygen content. It should be noted that no analytical blank, (believed to be approximately 8 micrograms), has been substracted from the data. If the blank is actually that high, the actual oxygen content after purification is about 3 ppm.

Corrosion

Three checkout tests were completed with the isothermal corrosion test facility described in Quarterly Progress Reports No. 3 (Ref. 3) and 4 (Ref. 4). In the first test the susceptor was heated to 1600°F, while maintaining a pressure less than 1×10^{-6} torr. After 43 hours, the pressure was 9×10^{-8} torr as measured by a tubular Bayard-Alpert ionization gauge located on the side of the test chamber. The power required to hold this temperature was approximately 440 watts, assuming a power factor of 0.9. The temperature values obtained with Pt vs Pt+10%Rh thermocouples at this power level are shown in Table V. The variation of temperature with time was evaluated and the mean deviation was found to be $\pm$ 0.06% at 1586°F over a 20-hour interval.

TABLE IV: CHEMICAL ANALYSES (1) OF POTASSIUM

Sample Identification	O (2)	Fe	B	Co	Mn	Al	Mg	Sn	Cu	Pb	Cr	Si	Ti	Ni	Mo	V	Be	Ag	Zr	Sr	Ba	Ca	Na	Cb	Remarks
		Chemical Analyses, ppm in KCl																							
Drum D-80																									
Vendor Analyses	11	12	<10	<5	1	<2	4	<5	4	<5	<5	<25	<5	<5	<3	<1	<1	<1	<10	<1	<3	11	25		
GE Analysis	14.8	<1		<1	<1	<1	<1	<5	<1	<1	<1	<1	<1	1	<1	<1	<1	<1	<5			1	<5	<1	
	21.6																								
	26.9																								
(3) (4)	53.8																								
Vacuum	54.5																								
After Outgassing	1																								
After Hot Trapping (5)																									
25 Hours, Sampled at 1300°F	8.9	5		1	<1	10	<1	<5	25	<1	15	25	1	1	<1			<1	<5			5	<5	>25	Contaminated by Pt Dish
	20.1																								
36 Hours, Sampled at 280°F	7.6	>25		<1	5	5	<1	<5	1	<1	>25	5	1	>25	5			<1	>5			15	10	5	Contaminated by Spatula
	14.0																								
Duplicate of 36-Hour Sample		1		<1	<1	<1	1	<5	<1	<1	<1	1	<1	<1	<1			<1	<5			10	<5	<1	
Capsule Filling																									
Capsules 7-12 (Cast Inside Tank)	24.2	1		<1	<1	1	<1	<1	<1	<1	<1	5	<1	<1	<1			<1	<5			1	10	<1	Average Calculated by Total μgms O/Total Sample Weight
	41.7																								
Average	28.7																								
Capsules 7-12 (Transfer Line)	13.2																								
	8.8																								
	11.1																								
Average	10.7																								
Capsules 13-18 (Cast Inside Tank)	13.2	5		<1	<1	<1	<1	<1	<1	<1	<1	1	<1	<1	<1			<1	<5			<1	5	<1	
	13.0																								
	16.9																								
Average	14.4																								
Capsules 13-18 (Transfer Line)	12.6																								
Capsules 19-24 (Cast Inside Tank)	14.9	1		<1	<1	<1	<1	<1	<1	<1	<1	1	<1	<1	<1			<1	<5			<1	>5	<1	
	15.4																								
	26.8																								
Average	17.5																								
Capsules 19-24 (Transfer Line)	14.9																								
	12.4																								
	8.3																								
Average	10.9																								
Capsules 25-28 (Cast Inside Tank)	25.1	5		<1	<1	1	<1	<1	<1	<1	<1	15	<1	<1	<1			<1	<5			>25	>10	<1	
	16.1																								
	10.4																								
Average	15.5																								
Capsules 25-28 (Transfer Line)	16.3	1		<1	<1	<1	<1	<1	<1	<1	<1	5	<1	<1	<1			<1	<5			1	>5	<1	
	15.6																								
	7.7																								
Average	11.8																								

(1) Analysis of Metallic Elements in KCl by Spectrographic Techniques
(2) Analysis of Oxygen as K_2O by Mercury Amalgamation Method
(3) Amalgamation Apparatus Environment: Purgon Purified by Passing through 13 x Molecular Sieve plus Titanium except where noted
(4) Vacuum 5 x 10^{-6} Torr Maximum
(5) Sampling Temp approx 200°F except where noted

TABLE V: CORROSION TEST FACILITY TEMPERATURE DATA FOR CHECKOUT TEST No. 1

(Power Level, 440 Watts)

Thermocouple Location	Temp., ^{o}F
Top of Capsule #1 (1)	1570
Top of Capsule #2	1616
Top of Capsule #3	1584
Top of Capsule #4	1585
Top of Capsule #5	1592
Top of Capsule #6	1619
Bottom of Capsule #1 (1)	1604
Bottom of Capsule #2	1601
Bottom of Capsule #3	1604
Bottom of Capsule #4	1588
Bottom of Capsule #5	1593
Bottom of Capsule #6	1593
T.C. #1 - Heater Support Table (1)	645
T.C. #2 - Top of Susceptor (1)	1578
T.C. #3 - Center of Susceptor (1)	1634
T.C. #4 - Between 5th and 6th Radial Heat Shield (1)	1076
T.C. #6 - Bottom of Susceptor	1594
Main Support Table	410

(1) Location of These Thermocouples is Shown in Figure 4.

The instrumentation techniques used for the thermocouples are shown in Figures 2 and 3. All junctions between the thermocouple leads, i.e., terminal blocks and feedthroughs, were tested at 200°F to evaluate any errors which could be introduced by these dissimilar metal junctions. The mean error for the entire facility was ± 0.04% with the maximum error being + 0.38% at a simulated test temperature of 1600°F. Because of the high AC current heaters used to heat the susceptors, the DC thermocouple output was checked at the test temperatures for induced AC current. The output showed AC in the microvolt range which would cause no discernible error. Temperature distributions were obtained only after the susceptor had been stabilized for a minimum time of 12 hours at any one power level.

After 43 hours at 1600°F, the temperature was lowered to 1200°F and held for 17 hours. The pressure at the end of this time was 6.2×10^{-8} torr and the power level was approximately 212 watts. No temperature distribution was obtained at this power level because of the wide variation in temperature values obtained at 1600°F. Checkout test No 1 was terminated subsequently for facility modification, designed to improve the temperature distribution in the susceptor and reduce the main support table temperature. The following modifications were made.

1) The thermocouples located on the tops of the capsules were shielded from direct exposure to the heating element above the susceptor to reduce the possibility of excessive heating of the thermocouple junctions.

2) The molybdenum support pins, which held the susceptor away from the main support table, were removed and the number of lower axial heat shields was increased from 5 to 10. This modification should reduce the heat loss into the main support table and thereby raise the temperature of the bottom section of the susceptor.

Capsule #6 was removed from the facility because of potassium leakage from a defective weld in the top end cap as the result of insufficient penetration. The potassium was cleaned from the vacuum chamber. No deposits were found in or on the susceptor.

After the modifications were completed, checkout test No. 2 was initiated. The susceptor temperature was brought up to 1600°F, and held for 47 hours, with the pressure again maintained below 1×10^{-6} torr. The pressure at the end of this time was 9.3×10^{-8} torr as measured by the Bayard-Alpert ionization gauge, and the power level throughout this test was approximately 435 watts. The temperature values obtained are shown in Table VI. Although the main support table temperature was reduced 160°F, the temperature readings obtained on the capsules still varied widely. The contact between the thermocouple junctions and the capsules was checked at the test temperature as a possible source of error. Although the

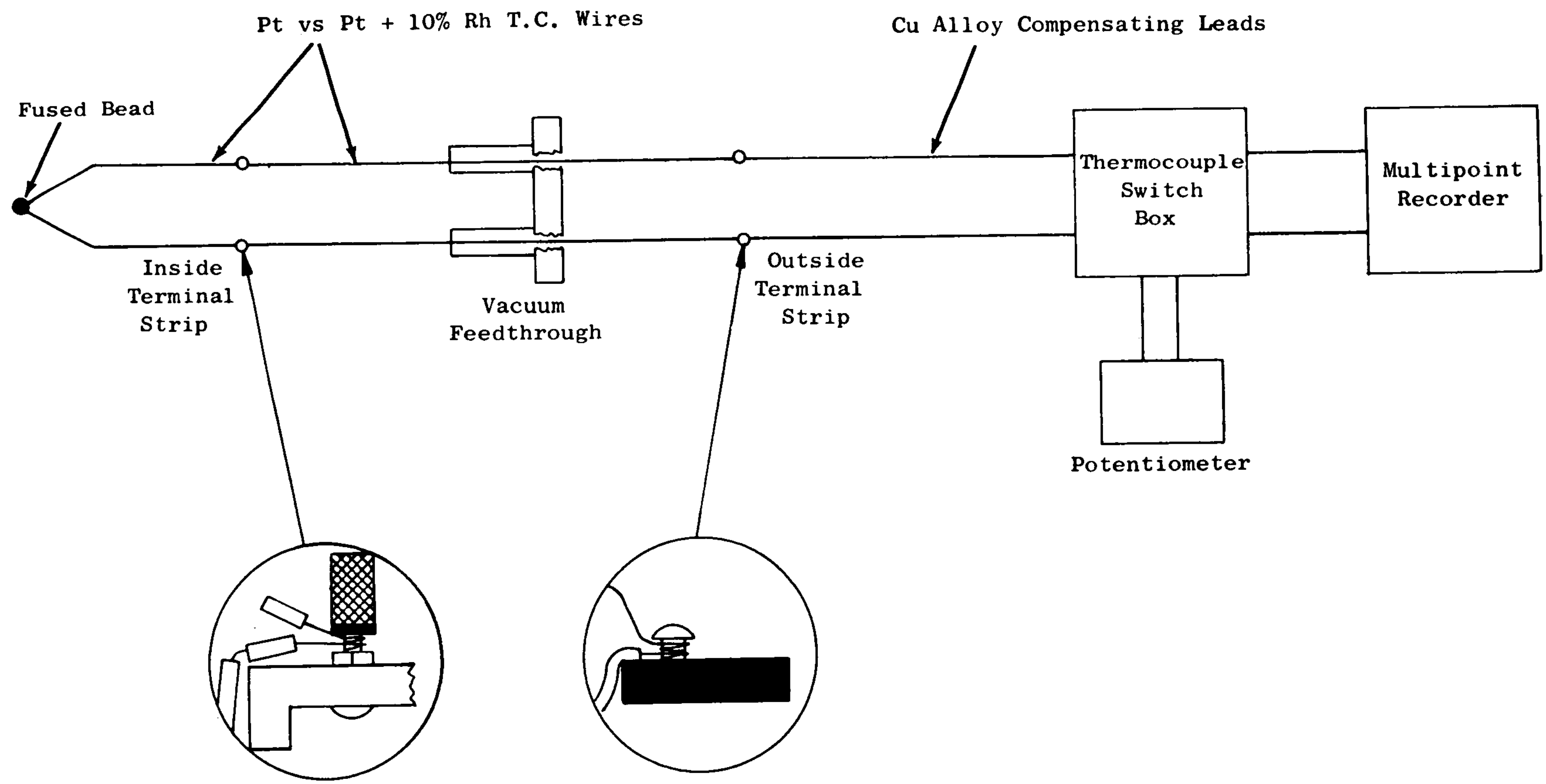
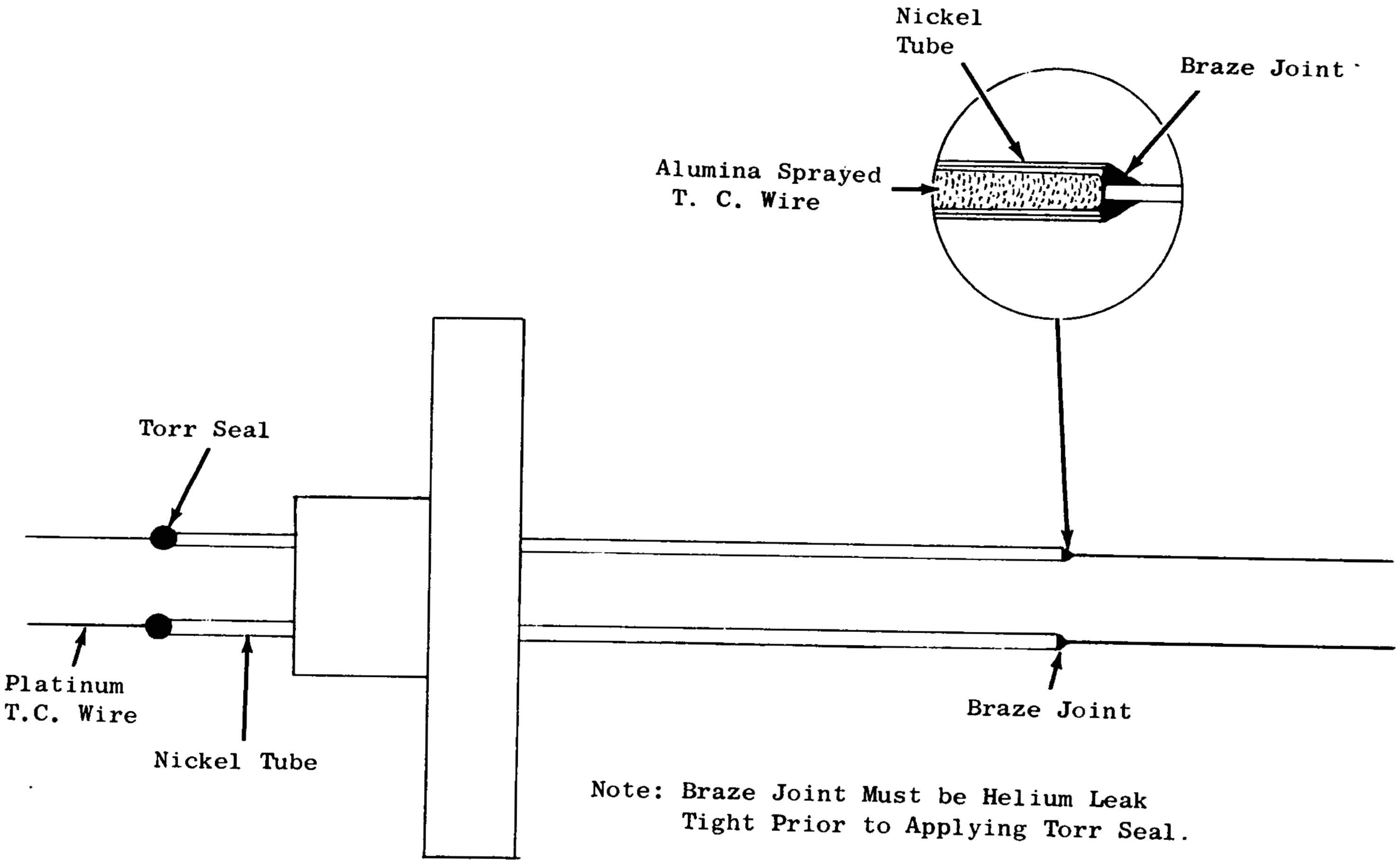


Figure 2. Diagram of Temperature Measurement Instrumentation Used on the Corrosion Test Facility.



Note: Braze Joint Must be Helium Leak Tight Prior to Applying Torr Seal.

Braze Alloy	Composition, %			Melting Point, °F	Flow Point, °F
	Ag	Cu	In		
Permabraz #615	61.5	24.0	14.5	1155	1300

Figure 3. Feedthrough Design Used for Bringing Thermocouple Wires into High Vacuum Chamber.

TABLE VI: CORROSION TEST FACILITY TEMPERATURE DATA FOR CHECKOUT TEST No. 2

(Power Level, 435 Watts)

Thermocouple Location	Temp., °F
Top of Capsule #1 (1)	1630
Top of Capsule #2	1608
Top of Capsule #3	(2)
Top of Capsule #4	1620
Top of Capsule #5	(2)
Top of Capsule #6	(3)
Bottom of Capsule #1 (1)	1549
Bottom of Capsule #2	(2)
Bottom of Capsule #3	1585
Bottom of Capsule #4	1590
Bottom of Capsule #5	(2)
Bottom of Capsule #6	(3)
T.C. #5 - Outer Heat Shield (1)	468
Main Support Table	248

(1) Location of These Thermocouples is Shown in Figure 4.

(2) No Reading.

(3) Dummy Capsule Removed After Checkout Test No. 1 Because of Defective Weld.

electrical continuity tests were positive, insufficient contact for proper heating of the junctions was suspected. Therefore, checkout test No2 was terminated so that the thermocouple junctions could be wired onto the capsule with Cb-1Zr alloy wire, as shown in Figure 4.

Checkout test No3 was also conducted at a susceptor temperature of 1600°F and, in this case, was held at the test temperature for 23 hours. The pressure at the end of this time was 2.55×10^{-8} torr as measured by the Bayard-Alpert ionization gauge and the power requirement was approximately 440 watts. The temperature values for checkout test No3 are recorded in Table VII. At a mean capsule temperature of 1628°F, the mean deviation was found to be $\pm$ 12°F or $\pm$ 0.74%. Subsequently, the temperature was lowered to 1200°F and held for 24 hours. The pressure at the end of this period was 3.0×10^{-9} torr and the power level throughout the test was approximately 201 watts. The temperature readings are shown in Table VII. At a mean capsule temperature of 1200°F, the mean deviation was found to be $\pm$ 16.8°F or $\pm$ 1.40%. The temperature was lowered again and held at 800°F for 24 hours with the pressure at the end of the 24-hour period at 1.8×10^{-9} torr. The power level at this temperature was approximately 48.7 watts. The temperature distribution is shown in Table VII. At a mean capsule temperature of 809°F, the mean deviation was found to be $\pm$ 17.2°F or $\pm$ 2.13%.

After verbal approval of the temperature distributions from the NASA Technical Manager, checkout test No3 was terminated. Two additional isothermal furnace assemblies were to be installed and the chamber readied for the actual testing of the candidate bearing materials.

Dimensional Stability

The component parts for the two isothermal dimensional stability test facilities, all of which were completed during the previous report period, were thoroughly cleaned, assembled and installed in the Varian high vacuum chamber (Figure 10, Ref. 3). The cleaning procedures employed for the various materials in the facility are summarized in Table VIII.

Prior to the final assembly and installation, one of the facilities was loaded with 30 unalloyed molybdenum dimensional stability test specimens as shown in Figure 5.

The loaded test facility was instrumented with 16 Pt vs Pt+10%Rh thermocouples. One thermocouple was attached to the surface of each of the top 5 specimen boxes; one was attached to the bottom surface of each of the bottom 5 specimen boxes; one was attached to the outer surface of each of the 6 specimen boxes in one stack. The thermocouples entered the facility through appropriate openings as shown in

TABLE VII: CORROSION TEST FACILITY TEMPERATURE DATA FOR CHECKOUT TEST No. 3

	Temp., °F		
	Power Level		
Thermocouple Location	440 Watts	201 Watts	48.7 Watts
Top of Capsule #1 (1)	1640	1215	824
Top of Capsule #2	1620 (3)	1208 (3)	824
Top of Capsule #3	1642	1219	827
Top of Capsule #4	1639	1219	828
Top of Capsule #5	1641	1219	827
Top of Capsule #6	(2)	(2)	(2)
Bottom of Capsule #1 (1)	1619	1189	798
Bottom of Capsule #2	1612	1179	788
Bottom of Capsule #3	1601 (3)	1169 (3)	784
Bottom of Capsule #4	1604 (3)	1175 (3)	790
Bottom of Capsule #5	1614	1186	798
Bottom of Capsule #6	(2)	(2)	(2)
T.C. #1 Heater Support Table (1)	740	536	364
T.C. #5 Outer Heat Shield (1)	507	351	235
Main Support Table	256	177	123

(1) Location of These Thermocouples is Shown in Figure 4.

(2) Dummy Capsule Removed After Checkout Test No. 1 Because of Defective Weld.

(3) Value Not Used in Computation of Mean Capsule Temperature or Mean Deviation.

TABLE VIII: CLEANING PROCEDURES USED FOR COMPONENT PARTS OF DIMENSIONAL STABILITY TEST FACILITY

Component	Cleaning Method
Stainless Steel	Pickled in a Solution of 1 Part 37% HCl + 1 Part 70% HNO_3, Rinsed in Water, Followed by Final Rinse in Ethyl Alcohol
Molybdenum	Vapor Blasted, Rinsed in Water Followed by Final Rinse in Ethyl Alcohol
Tantalum	Cleaned with Acetone and Rinsed in Ethyl Alcohol
Copper	Pickled in Dilute ($\sim$ 30%) HNO_3, Rinsed in Water Followed by Final Rinse in Ethyl Alcohol
Cb-1Zr Alloy	Pickled in Solution of 1 Part 70% HNO_3 + 1 Part 48% HF + 5 Parts H_2O. Rinsed in Water Followed by Final Rinse in Ethyl Alcohol

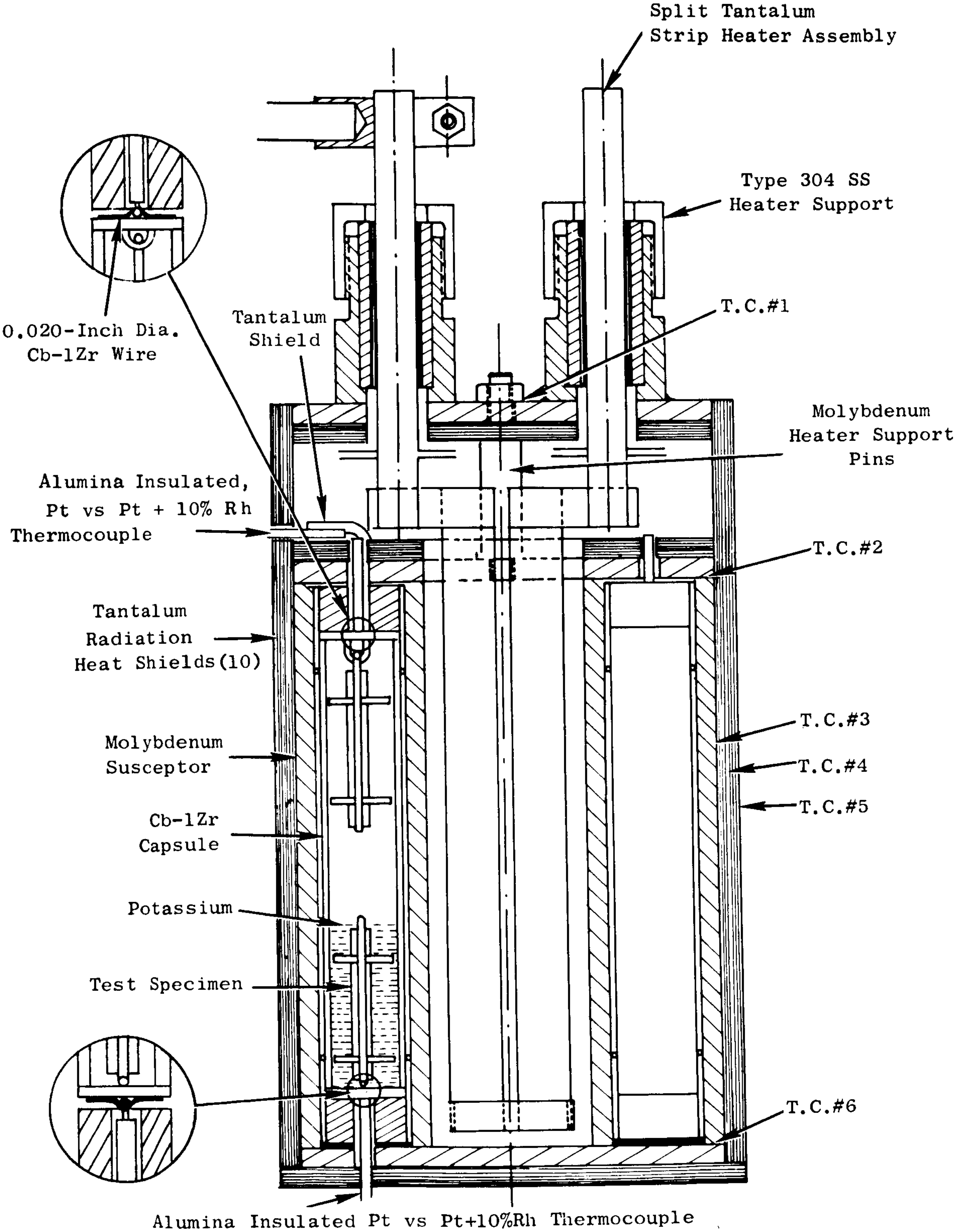


Figure 4. Isothermal Corrosion Capsule Furnace Facility Showing Instrumentation and Other Final Design Modifications.

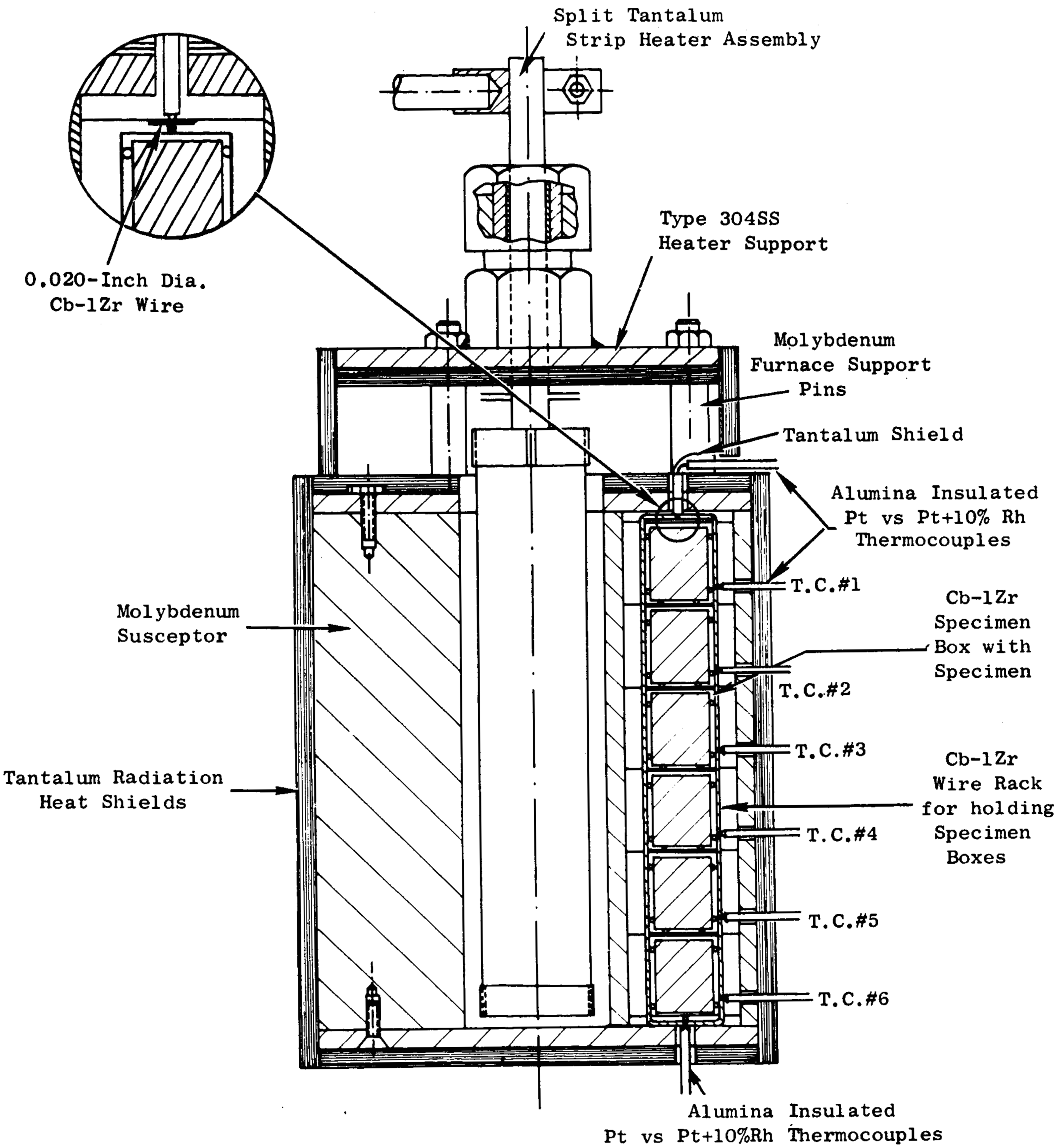


Figure 5. Isothermal Furnace Facility for Conducting Dimensional Stability Tests Showing Location of Thermocouples in Checkout Test No. 1.

Figure 5. Figure 6 is a photograph of the test facility installation with the thermocouples attached. The same type of thermocouple feed-throughs, terminal blocks, recorders and switch boxes are being used for the dimensional stability tests as are being used for the corrosion tests.

After the installation of the facilities was completed, the vacuum chamber was closed and evacuated to 1 x 10^{-7} torr as indicated by a Bayard-Alpert ionization gauge. Subsequently, the susceptor was slowly heated to approximately 1500°F during which time the vacuum reached a maximum of 4 x 10^{-6} torr. After the susceptor had reached the checkout temperature, the pressure decreased to 1 x 10^{-7} torr. The facility remained at the 1500°F checkout temperature for a period of 16 hours. Temperature measurements taken during this time were inconsistent. The temperature profile obtained after the susceptor had been at temperature for 16 hours is shown in Table IX.

Although the temperature variation within each group of thermocouples, i.e., top, bottom and side, is reasonably good, there was a significant difference in the average temperature from group to group. As a result of these data, checkout test No 1 was terminated. An analysis of the instrumentation showed that the thermocouples, which were indicating the higher temperature, were installed through the top of the susceptor. They were exposed to the heating elements and were probably heated to a higher temperature than the susceptor. Another possible cause for error is insufficient length of the thermocouple lead wire at the test temperature. In the latter situation the thermocouple could result in a low temperature reading. This condition could exist for the thermocouples installed through the side and bottom of the susceptor where only 1/2-inch length of thermocouple is at the test temperature.

The test facility was reinstrumented in an effort to correct the possible sources of instrumentation error, believed to be responsible for the inconsistent temperature readings. All of the thermocouples and the tantalum shielding around the susceptor were removed and 14 Pt vs Pt+10%Rh thermocouples were reinstalled through the holes in the side of the molybdenum susceptor, as shown in Figure 7. The bead of the thermocouple is held firmly in place against the surface of the specimen box by means of tantalum wire. The only change in instrumentation techniques was: (1) all of the thermocouple leads were attached to the sides of the susceptor rather than to the outer heat shield, thus permitting a greater length of lead wire to be at the test temperature and (2) the thermocouple leads were affixed to the sides of all of the Cb-1Zr alloy specimen boxes to eliminate any possible direct radiation. One thermocouple was attached to the outer surface of each of the top 5 specimen boxes; one was attached to the outer surface of each of the bottom 5 specimen boxes; and one was attached to each of the 4 remaining specimen boxes in one column of specimens. For checkout test purpose only, one thermocouple was inserted through the top of the susceptor and attached to the top surface of the top specimen box in one column.

TABLE IX: DIMENSIONAL STABILITY FACILITY CHECKOUT TEST No. 1

Specimen		Temperature, °F Column No. 1	2	3	4	5	Average Temperature, °F
1	(Top)	1511 (1)	1501 (1)	1504 (1)	1518 (1) 1464 (2)	-	1509
2					1476 (2)		
3					1469 (2)		
4					1485 (2)		
5					1479 (2)		
6	(Bottom)	1428 (3)	1380 (3)	1425 (3)	1462 (2) 1433 (3)	1414 (3)	1416

(1) Thermocouples Installed Through the Top of the Susceptor.

(2) Thermocouples Installed Through the Side of the Susceptor.

(3) Thermocouples Installed Through the Bottom of the Susceptor.

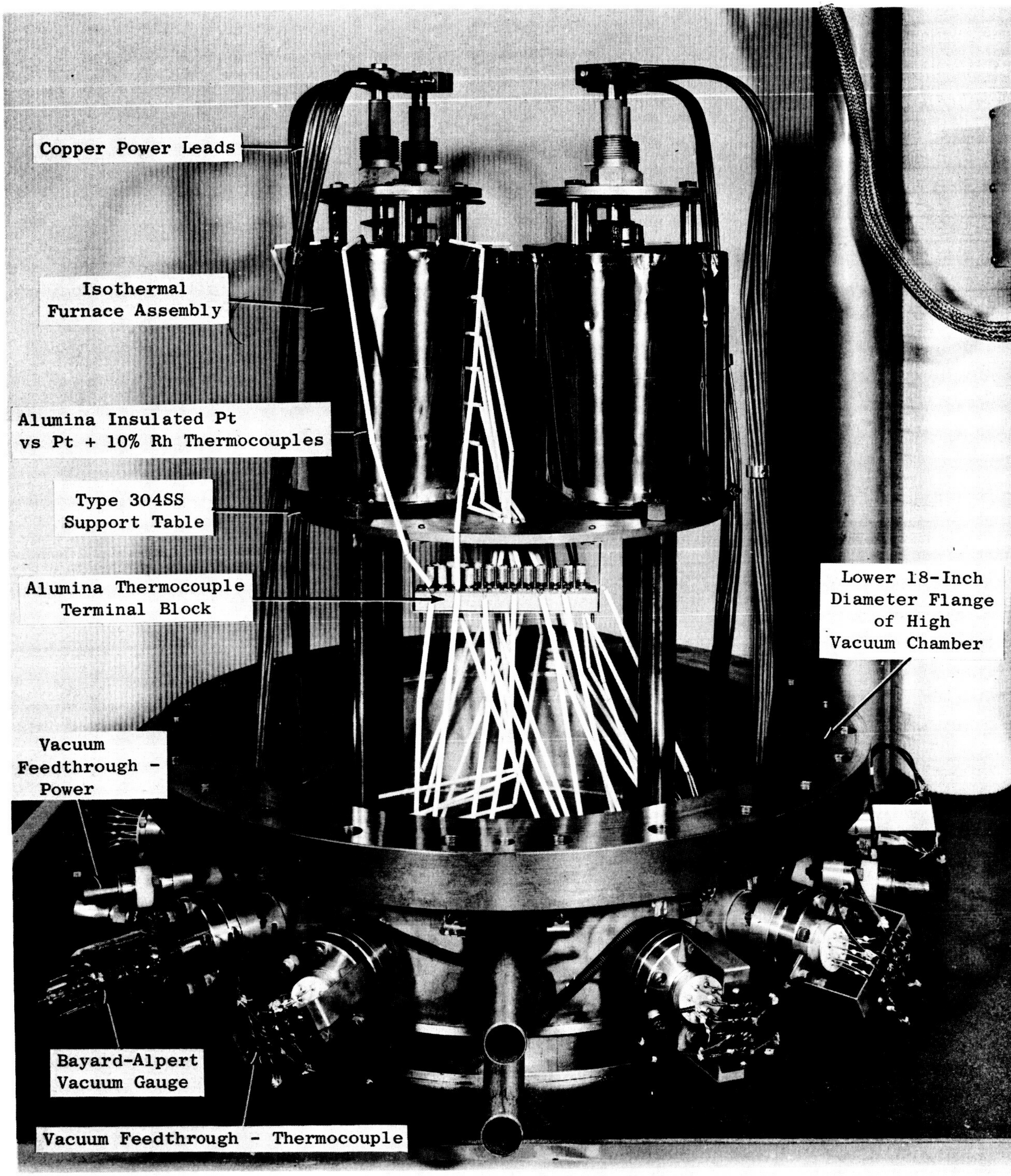


Figure 6. Dimensional Stability Test Facility Prior to First Checkout Test. (C64050816)

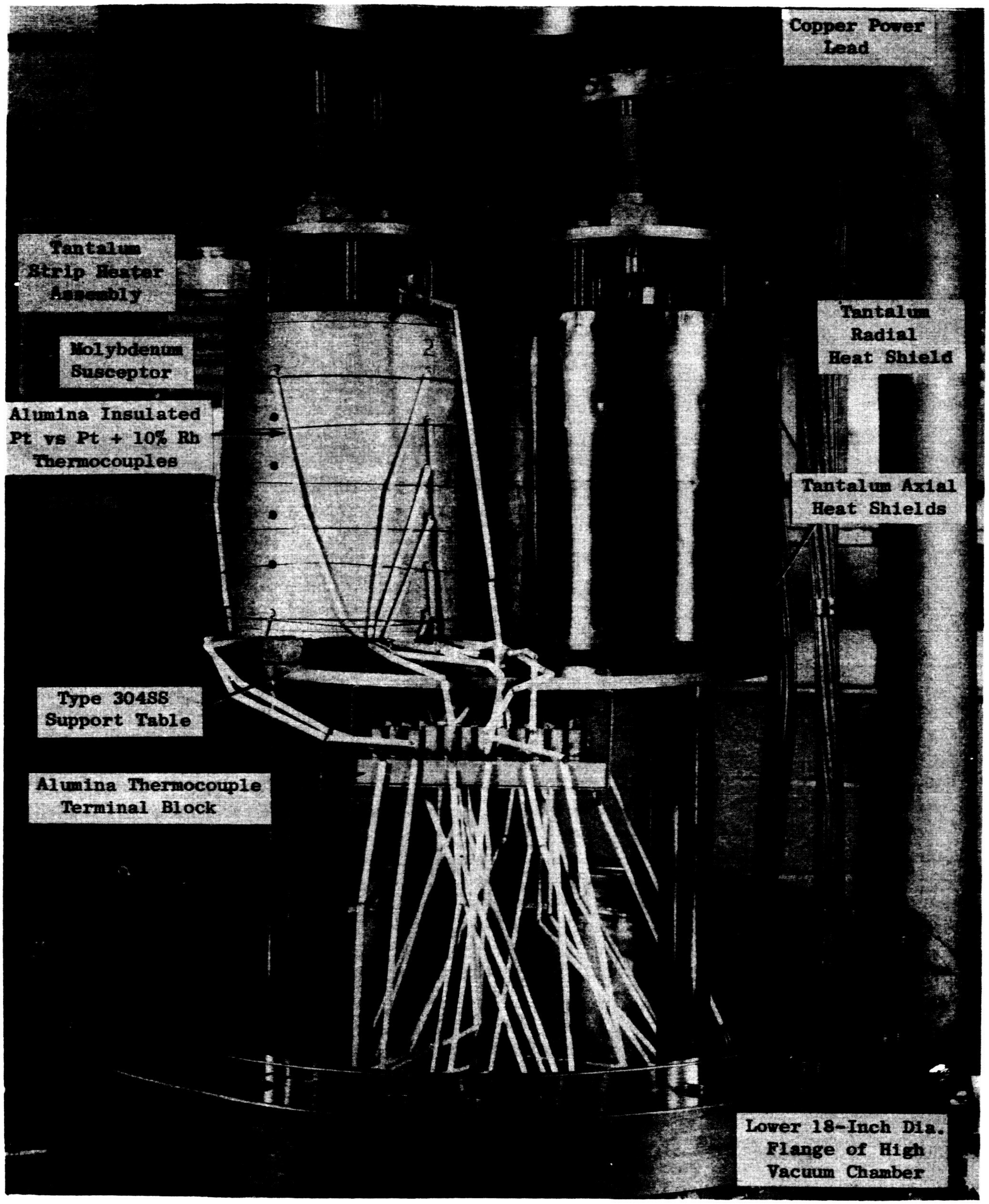


Figure 7. Dimensional Stability Test Facility Prior to Second Check-Out Test (C64061613)

After reinstrumentation, the vacuum chamber was closed and evacuated to 4.8×10^{-9} torr, as indicated by a Bayard-Alpert ionization gauge, after a 12-hour bakeout at 325°F. Subsequently, the susceptor was slowly heated to 1600°F during which time the pressure reached a maximum of 6×10^{-6} torr. Two hours after the susceptor had reached 1600°F, the pressure had decreased to the 10^{-7} torr range and after 24 hours the pressure had decreased to the 10^{-8} torr range. The facility remained at this temperature for a period of 400 hours after which time the pressure had decreased to 3×10^{-8} torr. The temperature was lowered to 750°F where it remained for 24 hours and then was raised to 1200°F and held for 30 hours. The checkout test was then terminated. The temperature profiles, obtained at the three test temperatures, are shown in Tables X, XI and XII. A plot of the change in pressure during the checkout test is shown in Figure 8. Note that the second susceptor was heated to 1600°F for 300 hours for the purpose of outgassing and that the maximum pressure during the heatup was 2.4×10^{-7} torr.

Cb-1Zr alloy sheet specimens, 0.060-inch thick, were placed on the top of the top specimen boxes of each column of specimens before the susceptor cover was installed and were exposed to both checkout tests. The samples were analyzed for oxygen, nitrogen, hydrogen and carbon to determine the amount of environmental contamination. The data are shown in Table XIII. The increase in oxygen content is attributed to the large amount of outgassing experienced during the initial heating up of the facility. Considerably less oxygen pickup is expected in subsequent test runs.

Compression

Detailed drawings of the load train components (Figures 13, Ref. 3) were prepared in final form and forwarded to NASA. The two 5.75-inch diameter x 2.0-inch thick pieces of Mo-TZM alloy to be utilized in the fabrication of the anvil supports (drawing MDLO-101-4) in the load train were ordered from the American Metal Climax Company to a modified version of General Electric specification SPPS-15. The material was received and machined into finished components by the end of the report interim. The Lucalox insulating cup and ring (drawing MDLO-101-3 and 3A) are being manufactured by the GE-Lamp Glass Department. Although some difficulty was encountered in producing crack-free insulating cups, two crack-free parts were successfully produced and forwarded to GE-Evendale. The insulating ring proved to be more difficult to fabricate. The diameter of the configuration, 5.75 inches, was beyond the available fabrication capabilities at GE-Lamp Glass Department and modifications to their normal processing procedures were necessary to produce the part. Delivery is expected late in September. In order to expedite the room temperature tests, a duplicate set of rings, will be machined from steel.

The remaining components of the load train were machined from the required materials that had been on hand.

TABLE X: DIMENSIONAL STABILITY FACILITY CHECKOUT TEST No. 2
TEMPERATURE PROFILE FOR 1600°F

Specimen		Temperature, °F Column No. 1	2	3	4	5	Average Temperature, °F
1	(Top)	1603	1605	1606	1604	1605	1605
2					1611		
3					1611		
4					1609		
5					1599		
6	(Bottom)	1585	1587	1590	1583	1585	1586

Mean Temperature	1603°F
Mean Deviation	± 7.8°F
Mid-Range Temperature	1597°F
Maximum Deviation from Mid-Range Temperature	± 10.5°F

TABLE XI: DIMENSIONAL STABILITY FACILITY CHECKOUT TEST No. 2
TEMPERATURE PROFILE FOR 1200°F

Specimen		Temperature, °F Column No. 1	2	3	4	5	Average Temperature, °F
1	(Top)	1206	1208	1208	1206	1208	1207
2					1211		
3					1210		
4					1207		
5					1198		
6	(Bottom)	1186	1190	1193	1183	1186	1188

Mean Temperature	1202°F
Mean Deviation	± 7.5°F
Mid-Range Temperature	1200°F
Maximum Deviation from Mid-Range Temperature	± 12°F

TABLE XII: DIMENSIONAL STABILITY FACILITY CHECKOUT TEST No. 2
TEMPERATURE PROFILE FOR 750°F

Specimen		Temperature, °F Column No. 1	2	3	4	5	Average Temperature, °F
1	(Top)	762	763	764	763	765	763
2					763		
3					763		
4					759		
5					752		
6	(Bottom)	746	749	754	744	746	748

Mean Temperature	757°F
Mean Deviation	± 6.3°F
Mid-Range Temperature	756°F
Maximum Deviation from Mid-Range Temperature	± 8°F

TABLE XIII: CHEMICAL ANALYSES OF Cb-1Zr ALLOY ENVIRONMENTAL CONTROL SPECIMENS(1) FOR DIMENSIONAL STABILITY CHECKOUT TESTS

Element	Analyses, ppm	
	Before Exposure	After Exposure to Checkout Tests No. 1 (1500°F) and No. 2 (1600°F)
O (2)	80 (4)	159 (5)
N (2)	42 (4)	26 (5)
H (2)	1 (4)	<1 (5)
C (3)	30 (4)	25 (4)

(1) MCN 418-2, 0.060-Inch Thick Sheet

(2) By Vacuum Fusion Analysis

(3) By Conductometric Analysis

(4) Average of Duplicate Analyses

(5) One Analysis

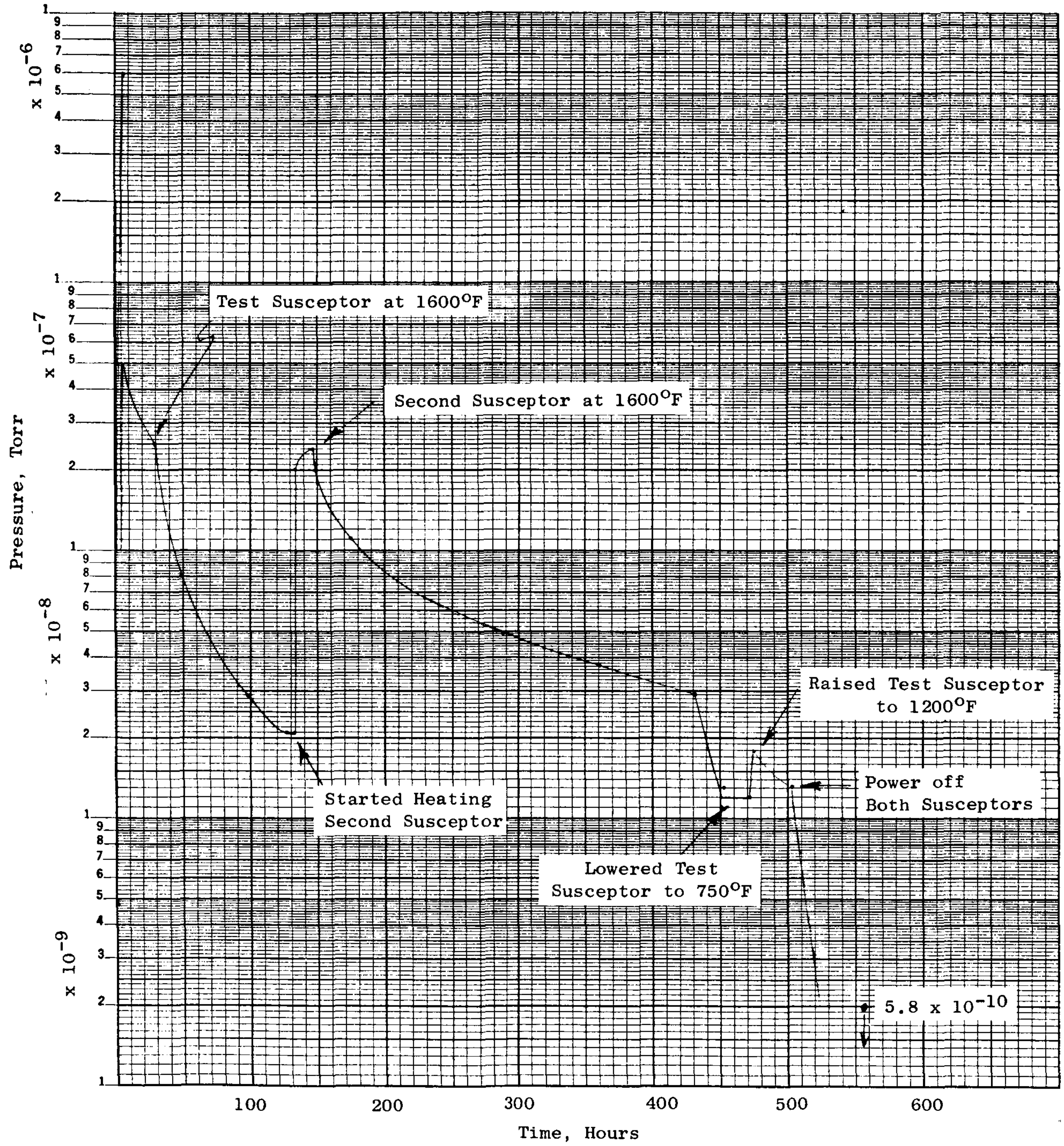


Figure 8. Pressure Curve for Dimensional Stability Check-Out Test No. 2

Hot Hardness

A trial run was made on a second sample of Cb-1Zr alloy to evaluate the improvement in vacuum environment obtained as a result of the thorough cleaning of all of the tester components, installation of new O-rings, fresh pump oils and shortening of the flexible metal bellows between the test chamber and pumping system. Table XIV presents the hardness data obtained in this checkout test. Again, an increase in hardness of the Cb-1Zr alloy specimen was observed upon cooling to room temperature. However, the magnitude of the increase was not as great as that observed after the first checkout test (Ref. 3), i.e., 29 Kg/mm^2 vs 45 Kg/mm^2. Although no hardness gradient could be detected on a microhardness traverse of the sectioned specimen, i.e., the data showed a hardness of 129 knoop 0.002 inch from the surface and an average of 135 knoop to a depth of 0.080 inch from the surface, gas analyses of the sample after test, Table XV, would indicate that a slight amount of surface contamination did occur.

In order to determine the degree of surface contamination that might occur in a material less sensitive to environmental conditions than Cb-1Zr alloy, a Mo-TZM alloy specimen was tested in the hot hardness facility under the existing vacuum capabilities. The data are shown in Table XVI and Figure 9. The lack of surface hardening, as shown by the hardness data obtained from the heating and cooling cycles, together with the favorable chemical analyses data taken from the test specimen and reported in Table XVII indicate that the present facility is suitable to be used for testing the existing candidate bearing materials. However, an additional effort will be made to improve the vacuum system before the test program is initiated. This will include the following modifications:

1) Enlarge the pumping port on the chamber by a factor of approximately 5.

2) Install a Chevron type liquid nitrogen baffle on the top of the diffusion pump.

3) Outgass the rather massive alumina furnace core and anvil at $2000^{o}F$ in a vacuum of 10^{-5} torr for several hours.

Upon completion of this work, the empty furnace will be cycled above the required test temperature several times and a final checkout run will be made with Cb-1Zr alloy test specimen.

TABLE XIV: HOT HARDNESS TEST DATA FOR Cb-1Zr ALLOY

Specimen: Cb-1Zr Alloy No. 3
0.5-Inch Cube
One Surface Polished to Approximately 2-5 RMS

Time, Min.	Vacuum, Torr	Temperature, $^{\circ}F$	Hardness Number, Kg/mm^2
-	-	R.T.	97 (1)
-	-	R.T.	85 (2)
0	8×10^{-5}	R.T.	87 (3)
4	1×10^{-4}	110	87
10	5×10^{-4}	197	70
22	2×10^{-4}	323	68
30	3×10^{-4}	433	62
35	4×10^{-4}	525	55
40	4×10^{-4}	610	57
47	3×10^{-4}	691	56
55	3×10^{-4}	791	60
64	2×10^{-4}	855	62
75	2×10^{-4}	925	62
80	2×10^{-4}	985	61
97	1×10^{-4}	1102	61
112	1×10^{-4}	1192	63
125	1×10^{-4}	1300	62
135	2×10^{-4}	1417	64
150	2×10^{-4}	1490	69
160	2×10^{-4}	1600	61
165	2×10^{-4}	1632	78
170	2×10^{-4}	1647	77
255	4×10^{-5}	608	80
260	4×10^{-5}	596	90
-	-	R.T.	127 (4)

NOTES: (1) Room temperature hardness of the sample on the Tukon tester using 100-gram load:

97 Kg/mm^2
97 Kg/mm^2
98 Kg/mm^2
Average 97 Kg/mm^2

(2) Room temperature hardness of the sample on the hot hardness tester using a Vickers Pyramid Diamond, 100-gram load and a 15-second hold:

80 Kg/mm^2
87 Kg/mm^2
87 Kg/mm^2
Average 85 Kg/mm^2

(3) Begin test cycle.

(4) Room temperature hardness of the tested sample on the Tukon tester using a 100-gram load:

121 Kg/mm^2
133 Kg/mm^2
126 Kg/mm^2
Average 127 Kg/mm^2

TABLE XV: GAS ANALYSES OF Cb-1Zr ALLOY HOT HARDNESS SPECIMEN NO. 3

Sample Identity	Chemical Analyses(1), ppm O	N	H
A - 0.125-inch cube sectioned to include one surface exposed to test atmosphere	170(2)	35(2)	6(2)
B - 0.125-inch cube from center of specimen	138(3)	24(3)	3(3)

(1) By vacuum fusion analysis

(2) Average of duplicate analyses

(3) One analysis

TABLE XVI: HOT HARDNESS DATA FOR Mo-TZM ALLOY

Specimen: Mo-TZM No. 1 (MCN 436)
0.5-Inch Cube
One Surface Polished to Approximately 2-5 RMS

Time, Min.	Vacuum, Torr	Temperature, °F	Hardness Number, Kg/mm^2
-	-	R.T.	275 (1)
-	-	R.T.	273 (2)
0	8×10^{-5}	R.T.	280 (3)
30	1.4×10^{-4}	200	245
35	2.4×10^{-4}	300	238
40	3.8×10^{-4}	550	208
60	3.5×10^{-4}	600	200
75	3.0×10^{-4}	700	216
85	2.8×10^{-4}	800	200
95	2.6×10^{-4}	900	204
110	2.6×10^{-4}	1000	198
-	-	1100	194
125	-	1142	187
135	1.6×10^{-4}	1300	191
140	3.0×10^{-4}	1400	185
150	3.0×10^{-4}	1500	191
175	-	1600	-
200	1×10^{-4}	1040	182
220	1×10^{-4}	855	200
235	1×10^{-4}	745	-
-	-	573	216
290	-	509	219
-	5×10^{-5}	435	226
360	5×10^{-5}	363	230
385	5×10^{-5}	324	226
410	5×10^{-5}	292	230
445	5×10^{-5}	249	235
-	-	R.T.	274 (4)

NOTES:

(1) Room temperature hardness of the sample on a Tukon tester using a 300-gram load:

	274 Kg/mm^2
	268 Kg/mm^2
	284 Kg/mm^2
Average	275 Kg/mm^2

TABLE XVI (cont'd)

(2) Room temperature hardness of the sample on the hot hardness tester using a Vickers Diamond Pyramid 300-gram load and a 15-second hold:

	279 Kg/mm^2
	268 Kg/mm^2
	271 Kg/mm^2
Average	273 Kg/mm^2

(3) Begin test cycle.

(4) Room temperature hardness of the tested specimen on the Tukon tester using a 300-gram load:

	262 Kg/mm^2
	277 Kg/mm^2
	291 Kg/mm^2
	268 Kg/mm^2
Average	274 Kg/mm^2

TABLE XVII: GAS ANALYSES OF THE Mo-TZM ALLOY HOT HARDNESS SPECIMEN

Sample Identity	Chemical Analyses[1], ppm O	N	H
A - 0.125-inch cube sectioned to include one surface exposed to test atmosphere	30	4	4
B - 0.125-inch cube sectioned from the center of specimen	17	1	2
C - As-received material	20	15	1

(1) By vacuum fusion analysis

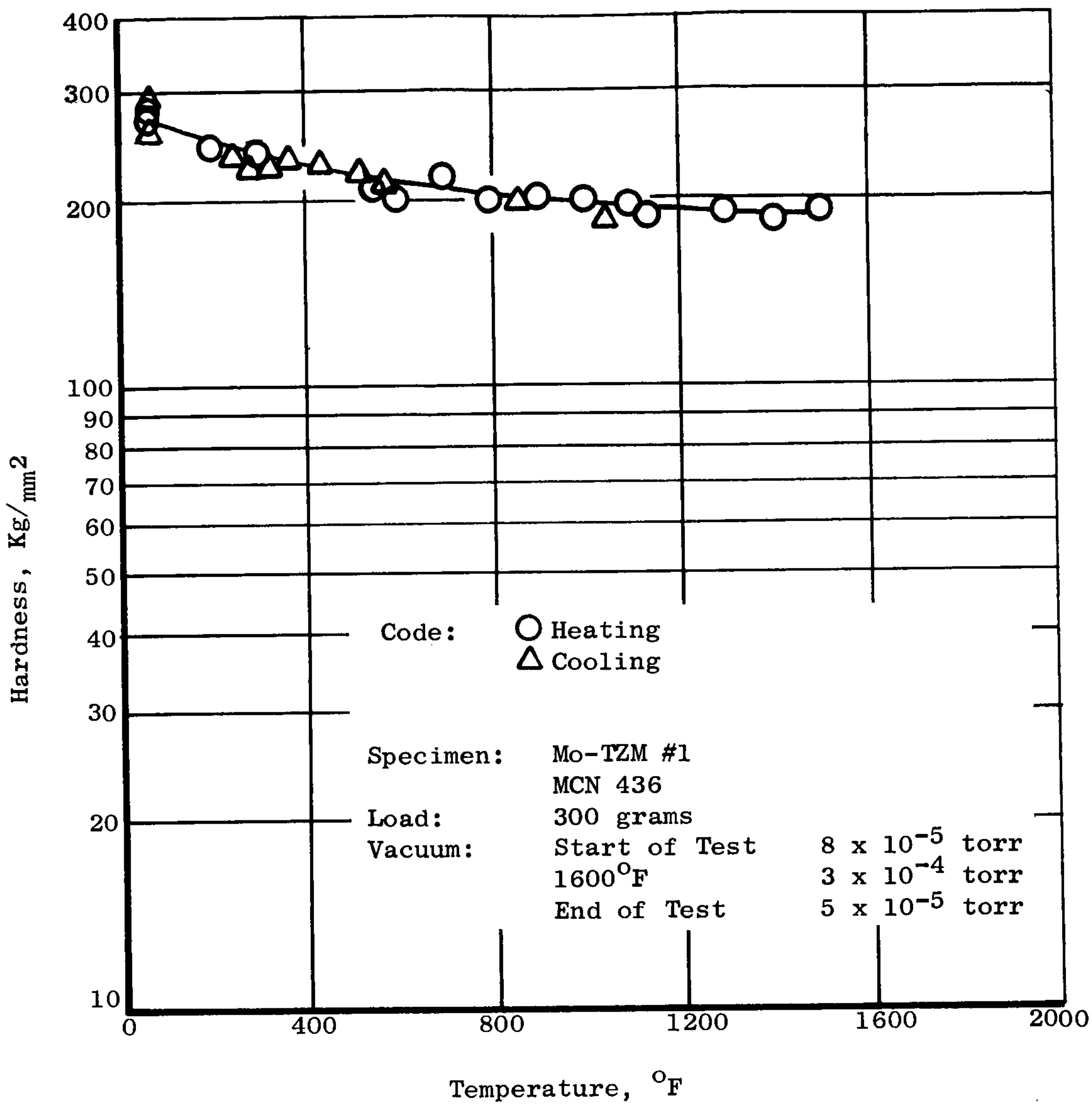


Figure 9. Hot Hardness of Mo-TZM Alloy

Thermal Expansion

The modifications required to introduce an inert atmosphere into the Chevenard Dilatometer were completed and a trial run was made using a Cb-1Zr alloy specimen. The modification consisted of the joining of a short length of glass tubing and a 2-way glass valve to an opening drilled through one wall of the quartz tube specimen holder. A small vacuum/pressure dynapump was attached to one exit of the glass valve and an inert gas bottle to the other exit by means of teflon tubing. The dynapump is capable of reducing the pressure within the quartz tube specimen holder to one-half an atmosphere within a few seconds.

After completing the necessary alignments of the Cb-1Zr alloy specimen, the Pyros standard and the recording mechanism, the quartz tube containing the specimen was evacuated and purged approximately 40 times by manipulation of the two-way valve. Then the valve was opened to the inert gas and a slight positive pressure of tank argon was maintained throughout the test. The specimen was tested through a typical test cycle which required approximately 8 hours to heat from room temperature to 1600°F and cool down to room temperature.

Examination of the exposed specimen revealed that approximately 90 percent of the surface of the specimen retained the pre-test metallic luster. The only area that showed evidence of discoloration was the tip of the specimen which was in contact with the quartz push rod. In sectioning the tested specimen for gas analysis, this discolored tip proved extremely hard, indicative of localized contamination. Three areas of the specimen, front tip (discolored), middle length and rear tip, were analyzed for gas content. The results, summarized in Table XVIII, showed that further modifications to the system were required before an acceptable test environment could be achieved.

The teflon gas lines were replaced with stainless steel tubing and welded construction or stainless steel swagelok fittings employed. Glass to glass connections were made with ground glass fittings and glass to metal connections were made by the use of brass swageloks.

The partial evacuation/purge method was abandoned and replaced with a positive flow purging system by joining a glass nipple to the quartz tube containing the Pyros standard, similar to the existing nipple on the test specimen tube. A glass cross-over tube joining the back ends of the quartz tubes completed the circuit for the inert gas. Glass stop-cocks were placed at the nipples of the quartz tubes. A trap and bubbler, which consists of Erlenmeyer flask joined

TABLE XVIII: GAS ANALYSES OF Cb-1Zr ALLOY SPECIMENS TESTED IN THE CHEVENARD DILATOMETER

Specimen	Instrument Modifications	Sample Location in Specimen	Element, ppm O	Element, ppm N	Element, ppm H
Untested Material	---	Random	94	22	8
#1	None	a) Front Tip Next to Push Rod	310	25	20
		b) 1/8-Inch From Sample (a)	287	12	19
#2	40 Pre-Test Vacuum/Argon Purges. Positive Pressure of Argon During Test	a) Front Tip Next to Push Rod	480	20	29
		b) 1-Inch From Sample (a)	161	17	25
		c) Back Tip	155	19	25

by glass tubing and small lengths of rubber tubing, were joined to the outlet side of the system. The first flask is empty and the second flask contains vacuum oil so that the rate of purging can be observed. A freshly activated gas purification train (Figure 5, Ref. 2) was installed at the gas source and the commercially pure argon was replaced with high purity helium (99.999%).

The inert gas circuit, during purging, now consists of passing the helium into the quartz tube containing the Pyros standard, to the quartz tube containing the specimen via the glass cross-over tube, and finally through the trap and bubbler. The revised system is shown in Figure 10.

Using the Cb-1Zr wire for a trial run, the system was purged for several hours with purified helium at room temperature. The outlet stop-cock was closed and a slight positive pressure was maintained on the system while the specimen was subjected to the test cycle. Examination of the specimen after cooling to room temperature showed that the Cb-1Zr alloy was completely free of discoloration.

A standard thermal expansion test specimen of the Cb-1Zr alloy was exposed to a similar cycle to verify the trial run. Again, the specimen emerged without discoloration and samples for gas analyses were sectioned from the front, middle and back end of the specimen. Although the analyses had not been completed by the end of the report interim, it appears that a satisfactory test environment has been achieved.

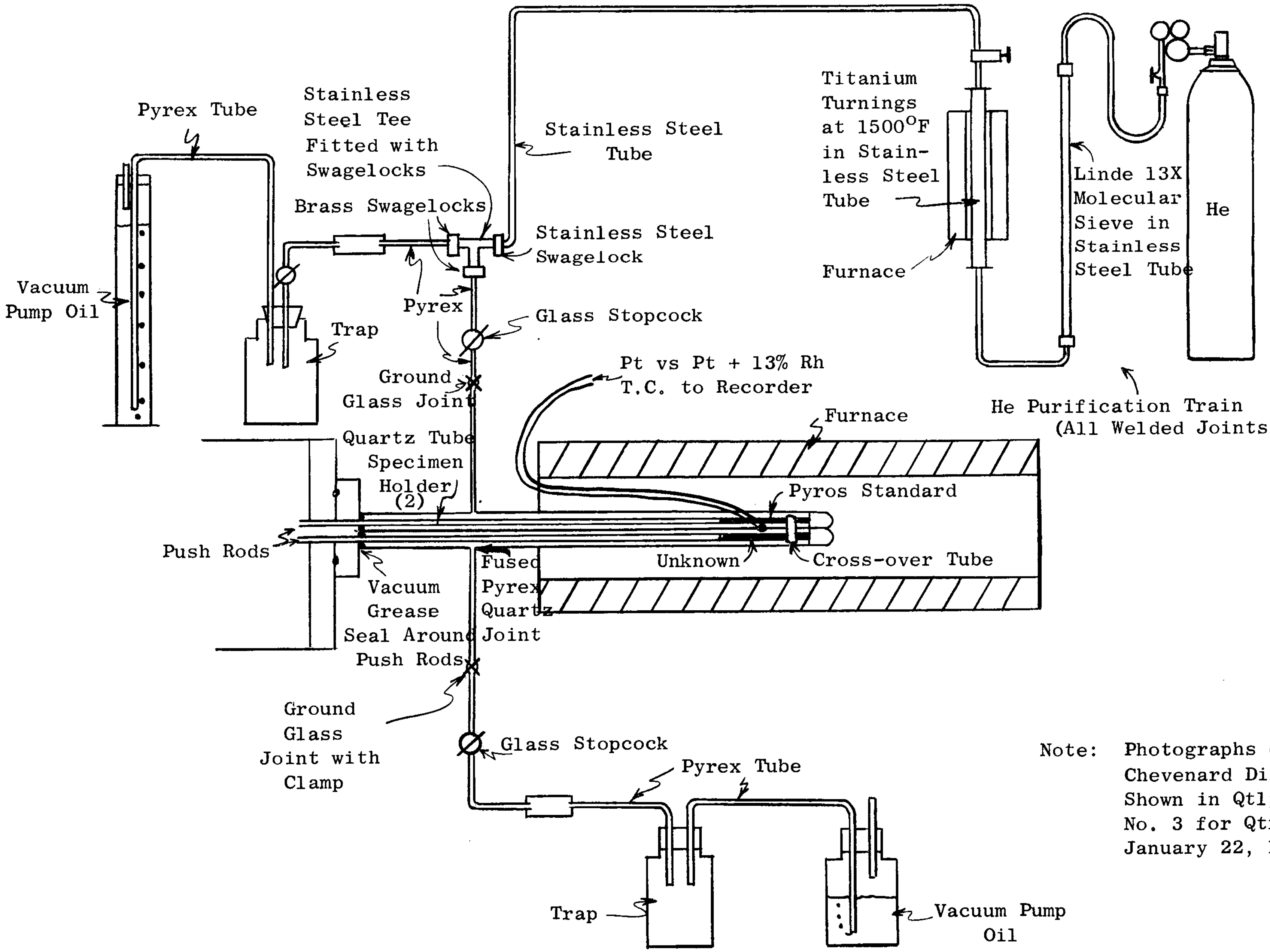
Friction and Wear in High Vacuum

A design review of the high vacuum tester was initiated in preparation for re-submission of inquiries to the vendors. The review will be completed and purchase inquiries sent to potential vendors in the next report interim.

Friction and Wear in Liquid Potassium

The design of the potassium friction and wear tester was extensively reviewed during the reporting period. All detail drawings were finalized and three sets of 60 drawings with appropriate process specifications were forwarded to the NASA Technical Manager for approval on June 23, 1964. With the exception of the potassium sump heater, verbal approval to proceed with the construction of the tester was received from NASA on July 8, 1964.

The drawings were released to vendors during the latter part of June for quotations. All quotations have been received and purchase orders were released for a large portion of the work, including the procurement of the refractory metals for the internal components of the tester.



Note: Photographs of Chevenard Dilatometer Shown in Qtly. Report No. 3 for Qtr. ending January 22, 1964.

Figure 10. Thermal Expansion Test Facility Showing Inert Gas Supply and Purification System.

Main Assembly. The major order for the fabrication of the tester was placed with the McGregor Manufacturing Company of Troy, Michigan. Except for the components listed below, McGregor will fabricate one complete assembly to G.E. drawing 246D907 plus spare parts.

Item	Part No.	Designation
1	P2	Motor
2	P39	Bearings
3	P50	Heater
4	P57	Baffle
5	P58	(Disc) Specimen holder
6	P59	(Disc) Specimen holder/ sleeve assembly
7	P101	Specimen holder assembly (loading arms)

McGregor is presently placing orders for material for fabrication.

The purchase of two spare P29 magnets, P30 magnets and P31 diaphragms is still being negotiated. For cost purposes, the Cb-1Zr alloy sump (P56) and the inner chamber of the vacuum chamber (P64) will be welded fabrications instead of the intended one piece construction. The Cb-1Zr alloy sump will be fabricated from three pieces (bottom piece, cylinder-cone and flange) and will be welded by General Electric to specification SPPS 3B,"Welding of Columbium - 1% Zirconium Alloy by the Inert-Gas Tungsten Arc Process." The addition of the welds to the Cb-1Zr alloy sump are of no cause for concern since they are not primary seals. However, the single girth weld, which joins the bottom piece of the stainless steel vacuum chamber to the seamless cylinder-cone, is a primary seal and will require radiographic inspection and leak checking with a mass spectrometer to specification AMS 2635, "Radiographic Inspection", and SPPS 26,"Mass Spectrometric Leak Detection Using Helium,"respectively.

Loading Arm. As presently designed, the bellows for the rider specimen holder assembly (P101) requires rotation and translation of one end of the bellows in a plane parallel to the other end.

This property of a bellows is not normally required, and therefore, is not available from the manufactures or engineering records. Samples of the bellows, intended to be used, were obtained and it was discovered that the forces required to achieve the lateral translation are significantly larger than the loads which are to be transmitted and measured. Since the lateral translation force is expected to vary with chamber pressure and temperature, the accuracy of the measurement of forces imposed on the specimens might be compromised. Therefore, the bellows attachment system was redesigned and quotations on the new design are being received. Redesign of the load arms is not expected to delay assembly.

A device for balancing the loading arms and obtaining tare weights under pressure has been conceived and detail drawings are now being prepared. Similarly, a design was completed and drawings are being prepared for a dummy thermocouple well which will be installed in a loading-arm hole and will extend into the stream of potassium expelled from the disc sample. The purpose of this thermocouple is to measure potassium temperature at the location of the test specimens and compare this temperature against the temperature recorded by the thermocouple in the well which touches the bottom of the sump.

Ball Bearings. The ball bearings (P39) for the main shaft were ordered from Industrial Tectonics, Inc. of Compton, California. The bearings are to be made from Type 440-C stainless steel as are the nickel-plated, one piece, machined retainers. The inner side will have extra stock on one face to provide a puller groove, believed to be necessary in the potassium environment. Delivery is expected in 14 weeks.

Present plans are to order several sets of commercially available bearings to permit the assembly of the tester if the shaft and bearing housings are completed before the special bearings are available.

Baffle and Disc Specimen Holders. Manufacture of the baffle (P57), specimen holder (P58) and specimen holder/sleeve assembly (P59) was awarded to the Universal Machine Company of Fenton, Michigan. Universal Machine will begin fabrication of the components as soon as the refractory alloy mill products are received.

Bakeout Heater. The Alco Research Instrument Department of American Standard was contacted and has supplied information indicating that one of their commercial products, Aerocoax miniature electric heating elements, can be supplied in the proper lengths and can supply sufficient heating power to bakeout the potassium sump during evacuation.

Details of the shaping of these heating elements to cover the desired area inside the tester vacuum chamber (item P64, GE drawing 246D907), and the brazing of the elements into place remain to be completed.

Potassium Sump Heater. Since the potassium friction and wear tester must function under severe operating conditions with respect to temperature, speed and pressure, the selection of the most suitable method of heating the potassium has been extremely difficult. Although the immersion heater was previously selected (Ref. 3 & 4), because of the critical nature of the component and the lack of experience with columbium clad heaters of this design, the NASA Technical Manager requested an extensive review of feasible methods of heating the potassium to 1600°F. Those methods reviewed were as follows:

1) Closed Loop Design

A. Indirect Heating Systems - Potassium is heated in the sump by means of a heat exchanger.

(1) Gas Fired Heat Exchanger - Externally heated air is forced through Cb-1Zr alloy clad Inconel tubes immersed in the potassium sump, Figure 11.

(2) NaK Heat Exchanger - Externally heated NaK is pumped through Cb-1Zr alloy tubes immersed in the potassium sump, Figure 12.

B. Direct Heating Systems - Power is supplied directly into the potassium pool.

(1) Tube Resistance Heater - Potassium is pumped through Cb-1Zr alloy tubing through which an electric current is passed. Heating is accomplished by the electrical resistance of the tube and the potassium. At 500 amps and 20 volts, a tube 1226 inches long is required to produce 10 KW of heating, Figure 13.

(2) Immersed Radiation Heater - A shielded electrical lead of molybdenum, which is brought into the tester and terminates in an evacuated Cb-1Zr alloy enclosure, immersed in the potassium pool, provides the electrical power for six Ta-10W alloy coil resistors. The circuit is grounded through the enclosure and the potassium pool. Heating is accomplished by radiation from the resistors to the Cb-1Zr alloy enclosure. At 500 amps and 20 volts, the 0.062-inch diameter heaters operate at 4200°F to provide 10 KW of heating, Figure 14.

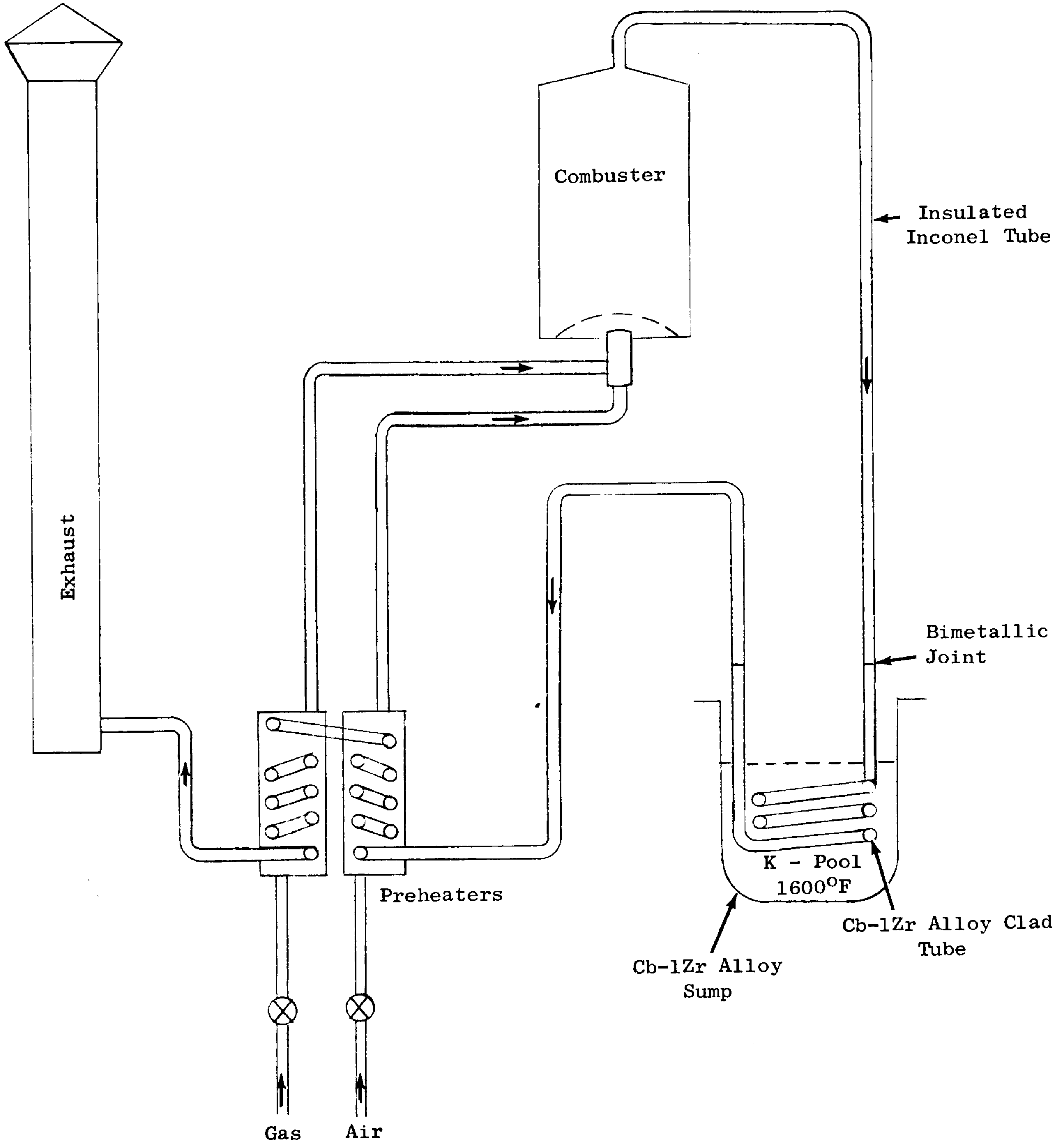


Figure 11. Gas Fired Heat Exchanger for Heating Potassium in Friction and Wear Tester.

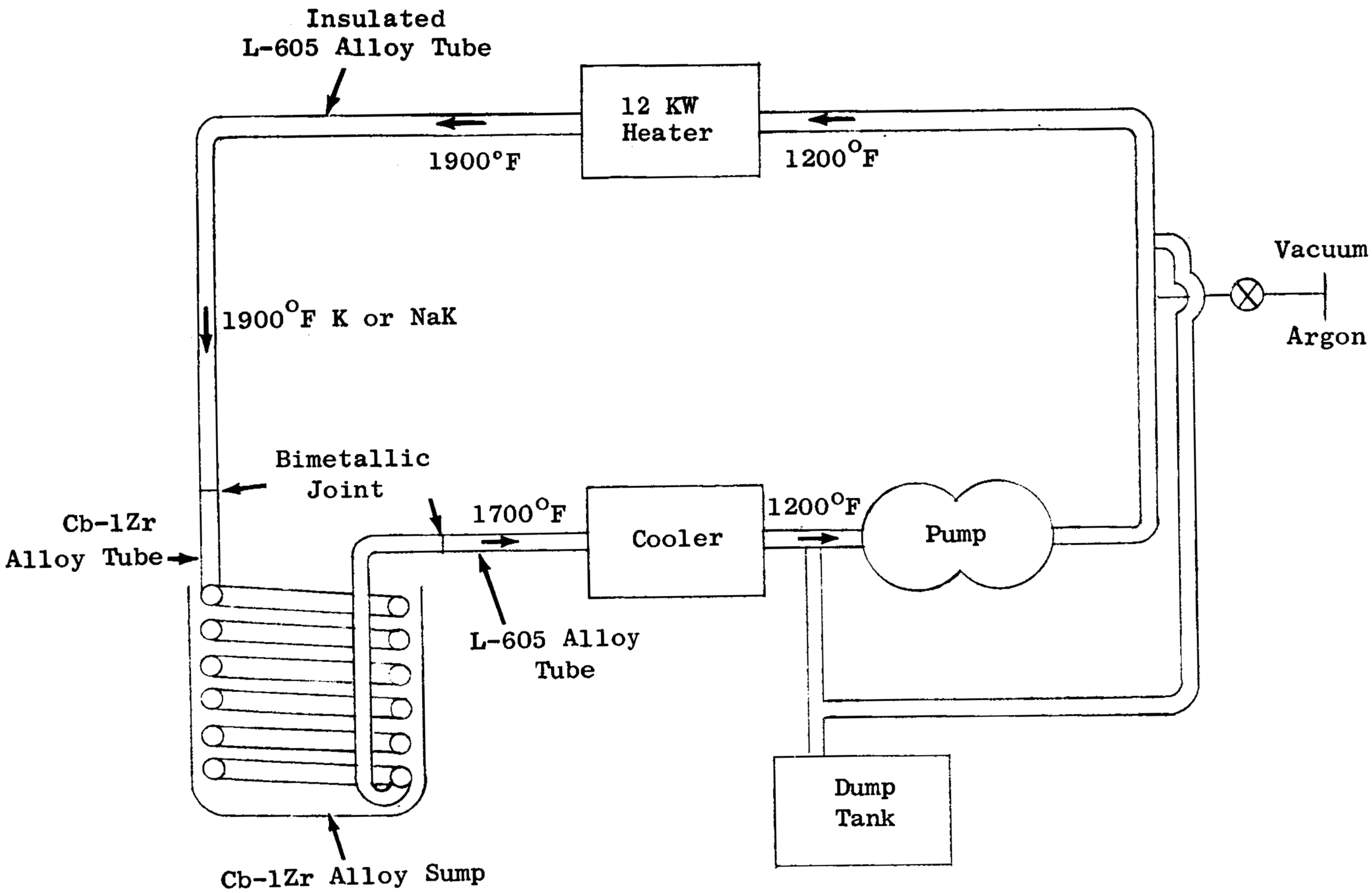


Figure 12. NaK Heat Exchanger for Heating Potassium in Friction and Wear Tester.

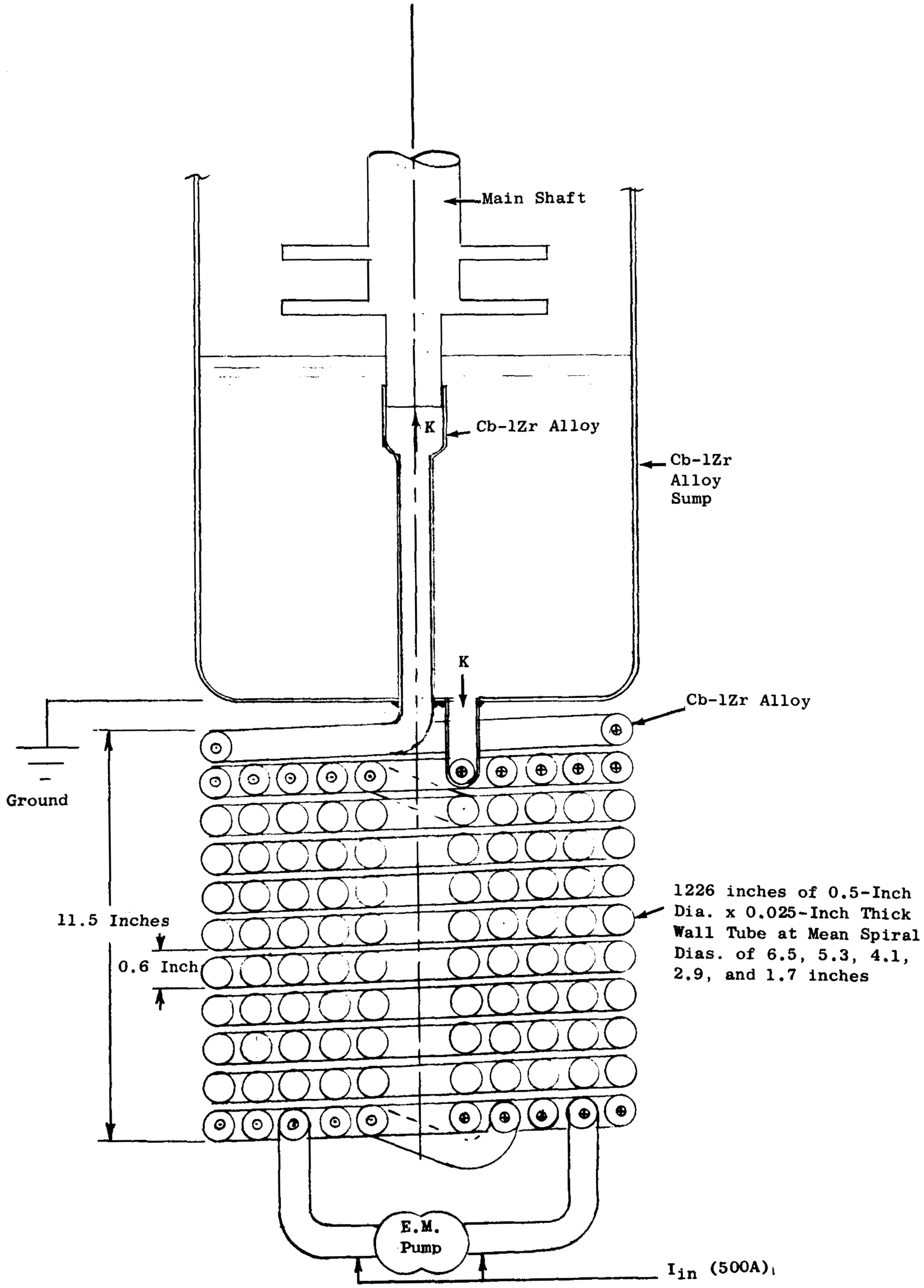


Figure 13. Tube Resistance Heater for Heating Potassium in the Friction and Wear Tester.

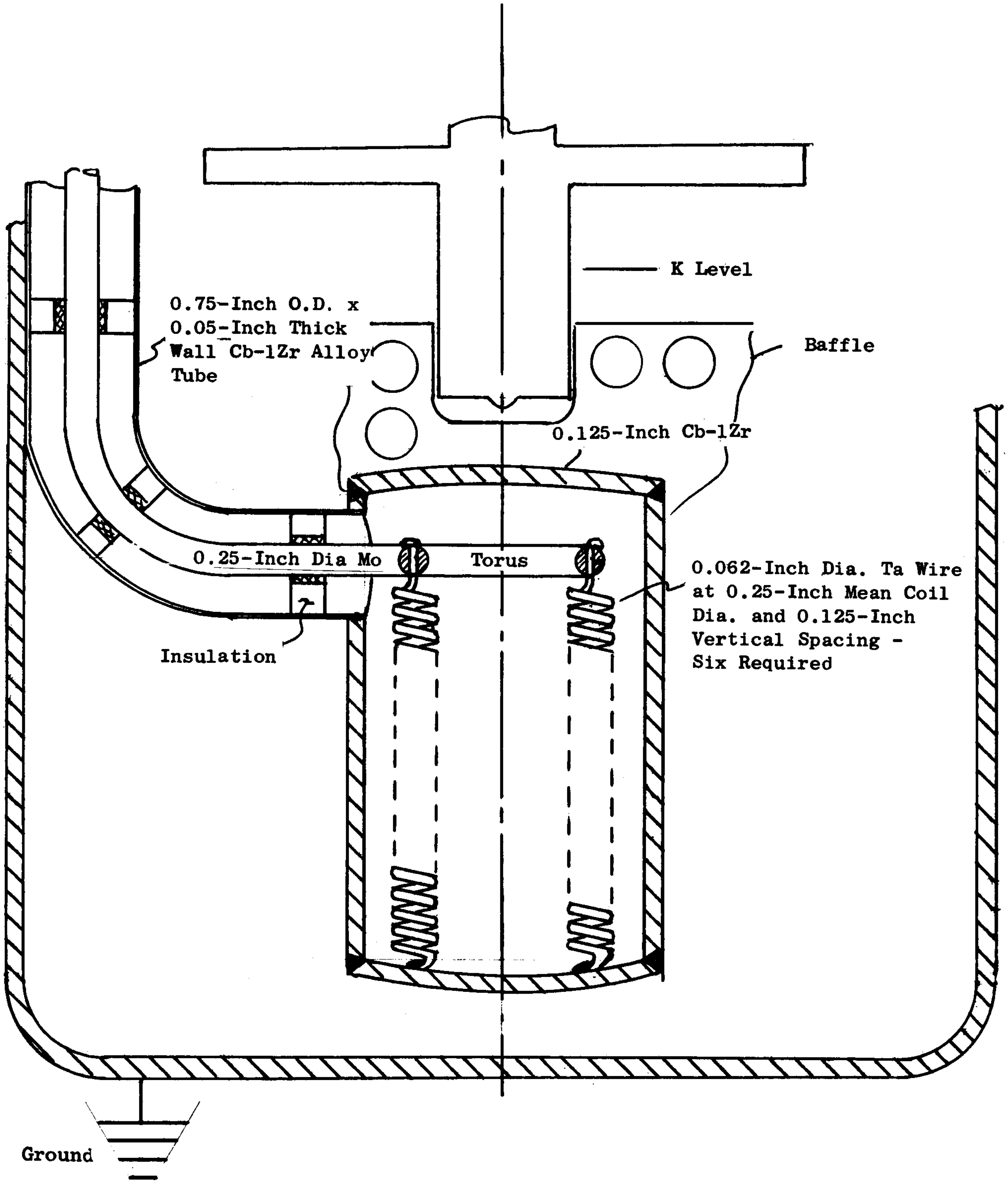


Figure 14. Immersed Radiation Heater for Heating Potassium in the Friction and Wear Tester.

(3) Immersed Conductive Heater - Standard 230-volt, 44-ampere current is supplied to seven Cb-1Zr alloy clad, BN insulated, Nichrome heating elements immersed in the potassium. The design is described in detail in Quarterly Progress Report No. 4, (page 18, Ref. 4).

2) Open Loop Design - A confined volume of potassium is heated externally and allowed to flow by gravity over the test specimens, Figure 15.

Other methods of heating which were reviewed, but were discarded for major technical deficiencies, are:

1) Induction Heating - This method requires a massive power supply, because of the high-frequency current needed. The electrical leads are massive and it would be extremely difficult to fit the coils into the tester or environmental chamber. If the coil is placed within the tester, shorting would be a problem and a special coil material would be required. If the coil is located externally, the chamber wall would be suscepted and the efficiency of the radiation heating would be extremely low.

2) Resistance Heating - Heat is provided through an anode and cathode dipped into the pool and requires currents of 25,000 amperes, or more, due to the low resistivity of the potassium. The massive leads could not be worked into the tester and the power supply system would be very expensive.

3) Radiation Heating - Heating of the outside of the stainless steel vacuum chamber and re-radiation to the Cb-1Zr alloy sump is a very inefficient method. Even with radiation shielding, approximately half of the heat would be radiated away from the sump. The current required for a tantalum, split-cylinder heater surrounding the stainless steel vacuum chamber would be approximately 1160 amperes. Also, the temperature to which the stainless steel could be heated would be limited. Cooling of the primary environmental seal would be a problem.

Of the heating methods considered, the immersion conductive heater, presently included as a detail drawing of the potassium tester, and the immersed radiation heater appear to be the most practical designs. Further evaluation of both of the methods of heating is in progress and a final decision will be made next month.

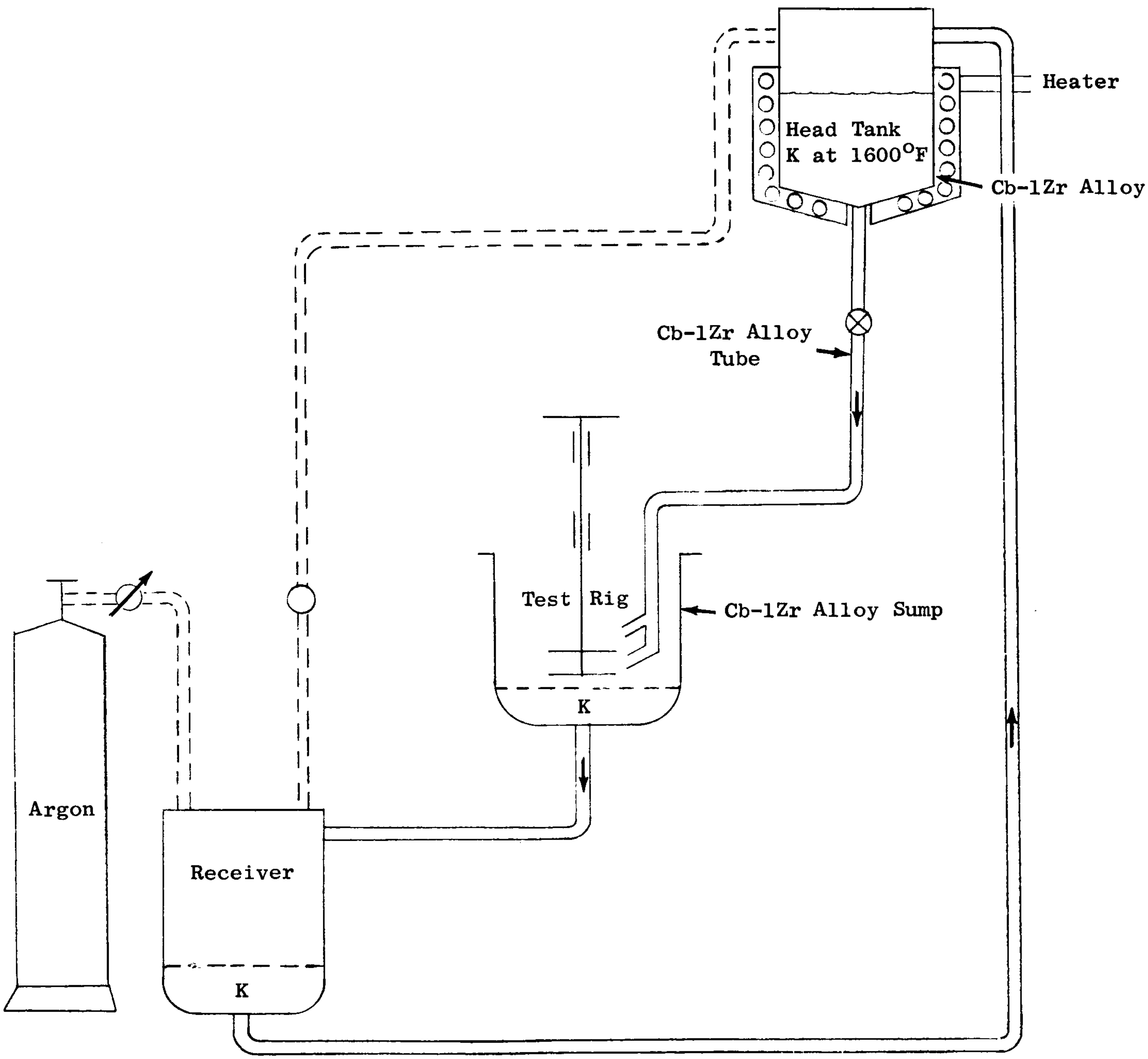


Figure 15. Open Loop Design for Gravity Feeding of Hot Potassium to Friction and Wear Specimens.

Test Facility. Minor changes were made to the design of the test facility for the potassium friction and wear test rig, described in detail in Quarterly Progress Report No. 4, (Ref. 4). For safety reasons the auxiliary heater and cooler incorporating polyphenyl ether as the heat exchanger fluid was redesigned to use air. The air heating system consists of two electric air heaters with a heating capacity of 6 KW each, connected in series. The new heating system is designed to supply air at a maximum temperature of 1000°F for evaporating potassium and bakeout during the vacuum cycle.

A Taylor gauge was incorporated in the tester to measure the pressure directly in the test rig during high pressure operation, i.e., 50 psi. Signals received from the Taylor gauge will control the diaphram valve regulating the air pressure on the external surface of the metal diaphragm of the magnetic clutch on the top of the test rig. In addition, two pressure switches were added, one to shut off the power to the test rig in the event of a loss of air pressure over the diaphragm and the other to shut off the power in the event of a sudden increase in pressure within the test rig.

Hand valves were replaced by remotely operated diaphragm valves to further improve the safety features of the facility during operation.

The detail design of the titanium-lined hot trap was completed. The hot trap will be constructed from Type 316 stainless steel and will contain 3.5 pounds of zirconium foil to achieve a ratio of 5 grams of potassium per square inch of gettering surface.

V. TEST PROGRAM

Corrosion

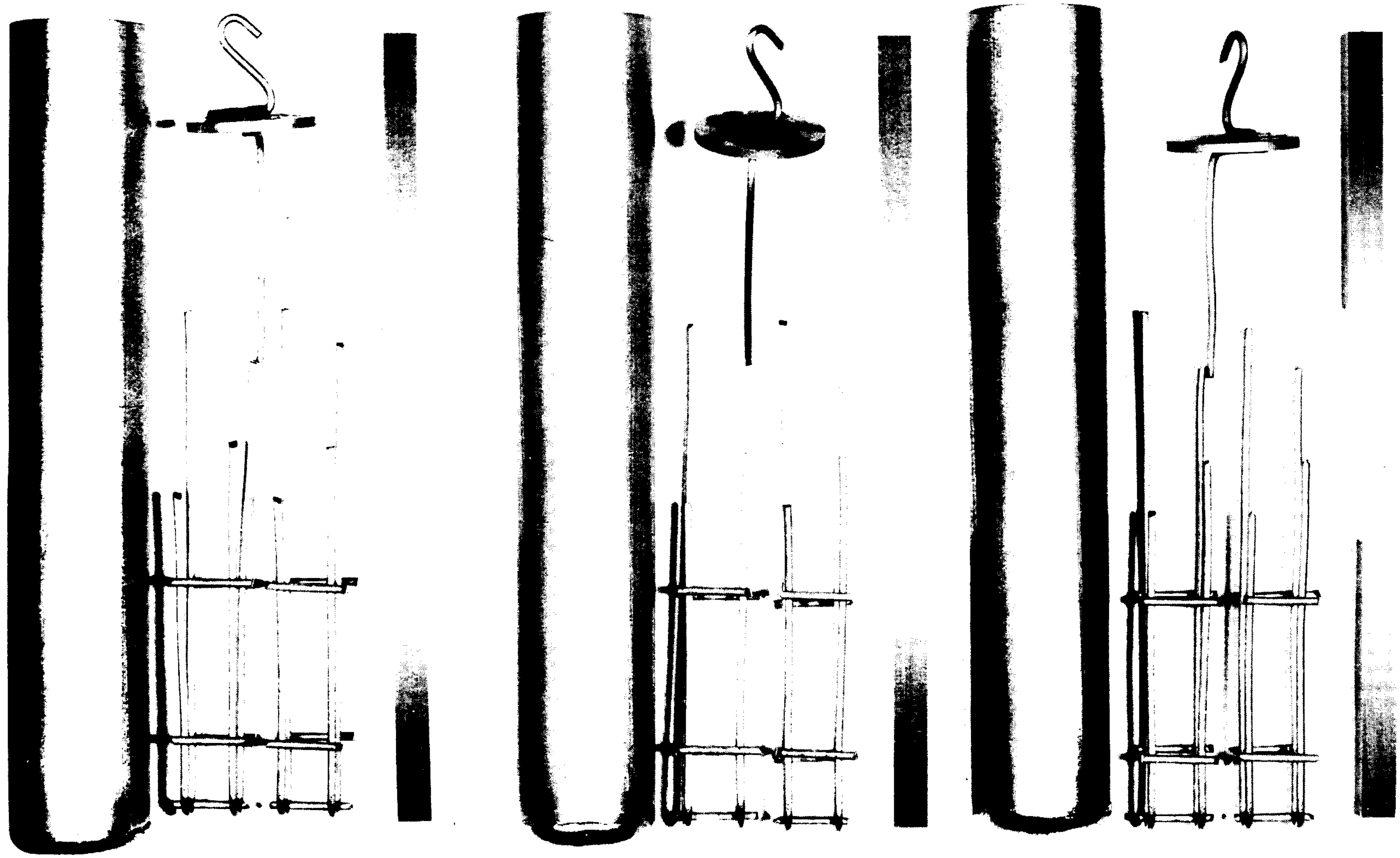
Eighteen Cb-1Zr alloy corrosion capsule assemblies containing Carboloy Grades 907 and 999, Mo-TZM alloy, tungsten, Lucalox and ZrO_2 specimens have been prepared, filled with purified potassium, vacuum sealed and tested for 1,000 hours at temperatures of 800°, 1200° and 1600°F in a vacuum of 10^{-8} to 10^{-9} torr. The fabricated capsules, described in Quarterly Progress Report No. 4 (Ref. 4), were cleaned according to SPPS specification 11A, "Chemical Cleaning of Columbium and Columbium Alloy Products." Because the inside surface finish of acid cleaned capsules was found to be the same as that of liquid honed material, i.e., 22-28 rms, the capsules were left in the as-pickled condition. After cleaning, the ID of each capsule was measured at both the top and bottom to $\pm$ 0.0001 inch. The welded specimen holders were cleaned in concentrated HNO_3 and then thoroughly rinsed in water followed by a final rinse in ethyl alcohol.

The specimens were cleaned individually in ethyl alcohol prior to dimensional measuring to $\pm$ 0.0005 inch. Typical capsules, specimen holders, and specimens are shown in Figures 16 and 17. Subsequently, the individual specimens were ultrasonically cleaned in ethyl alcohol and then rinsed in ethyl alcohol. After sufficient drying time, i.e., 30 minutes, the specimens were weighed to $\pm$ 0.0005 gram and immediately placed in the specimen holders. Then, the specimen holders were placed into the capsules.

The loaded capsules were filled with purified potassium using the vacuum filling facility described in Quarterly Progress Report No. 3 (Ref. 3). Chemical analyses of the potassium samples taken during the fillings are shown in Table IV.

The oxygen values obtained from samples in the transfer line and those cast within the weld chamber indicate a low oxygen content for the potassium. Although a comparison of the data obtained from the two different locations indicate that a small amount of contamination of the potassium may have occurred in the weld chamber, the consistency of the potassium samples differed in that the samples from the transfer line were more solid. Poorly cast specimens could lead to some contamination during transfer to the amalgamation apparatus. However, casting is an inherent characteristic of the capsule filling technique and it is not possible to heat the capsules during filling.

In plotting the original oxygen data as micrograms of K_2O vs sample weight, as shown in Figures 18 and 19, it can be seen that the



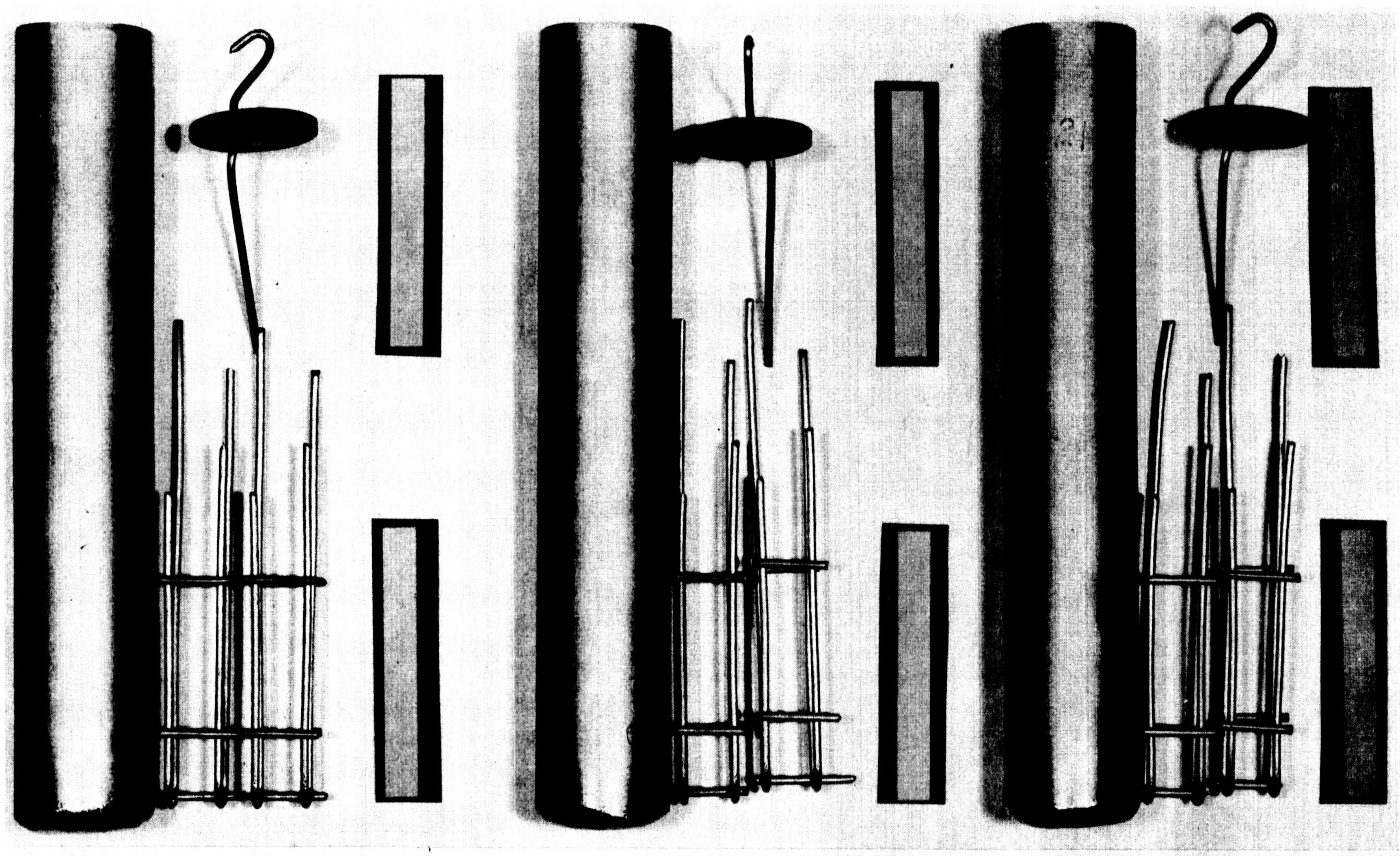
CAPSULE BIC-7
SPEC. 1036-A1-A2

CAPSULE BIC-8
SPEC. 1036-A3-A4

CAPSULE BIC-9
SPEC. 1036-A5-A6

|←1 INCH→|

Figure 16. Cb-1Zr Alloy Capsules, Specimen Holders and Carboloy Grade 907 Test Specimens Prior to Loading the Capsule, Filling with Potassium and Vacuum Sealing. (C64051211)



CAPSULE BIC-19
SPEC. 1039-A1-A2

CAPSULE BIC-20
SPEC. 1039-A3-A4

CAPSULE BIC-21
SPEC. 1039-A5-A6

|←1 INCH→|

Figure 17. Cb-1Zr Alloy Capsules, Specimen Holders and Lucalox Test Specimens Prior to Loading the Capsule, Filling with Potassium and Vacuum Sealing. (C64051933)

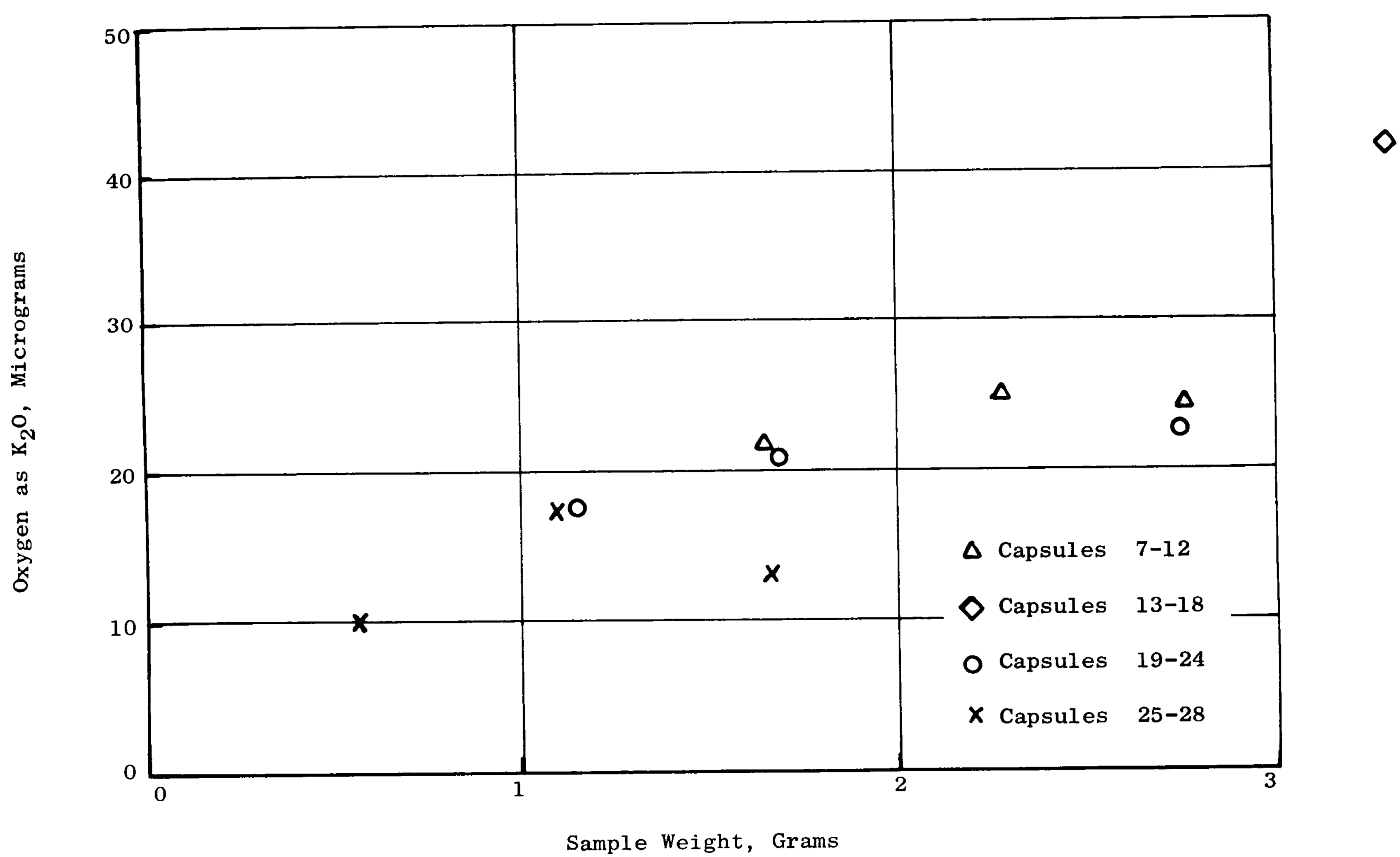


Figure 18. Oxygen Analyses of Potassium Samples Taken from Transfer Line.

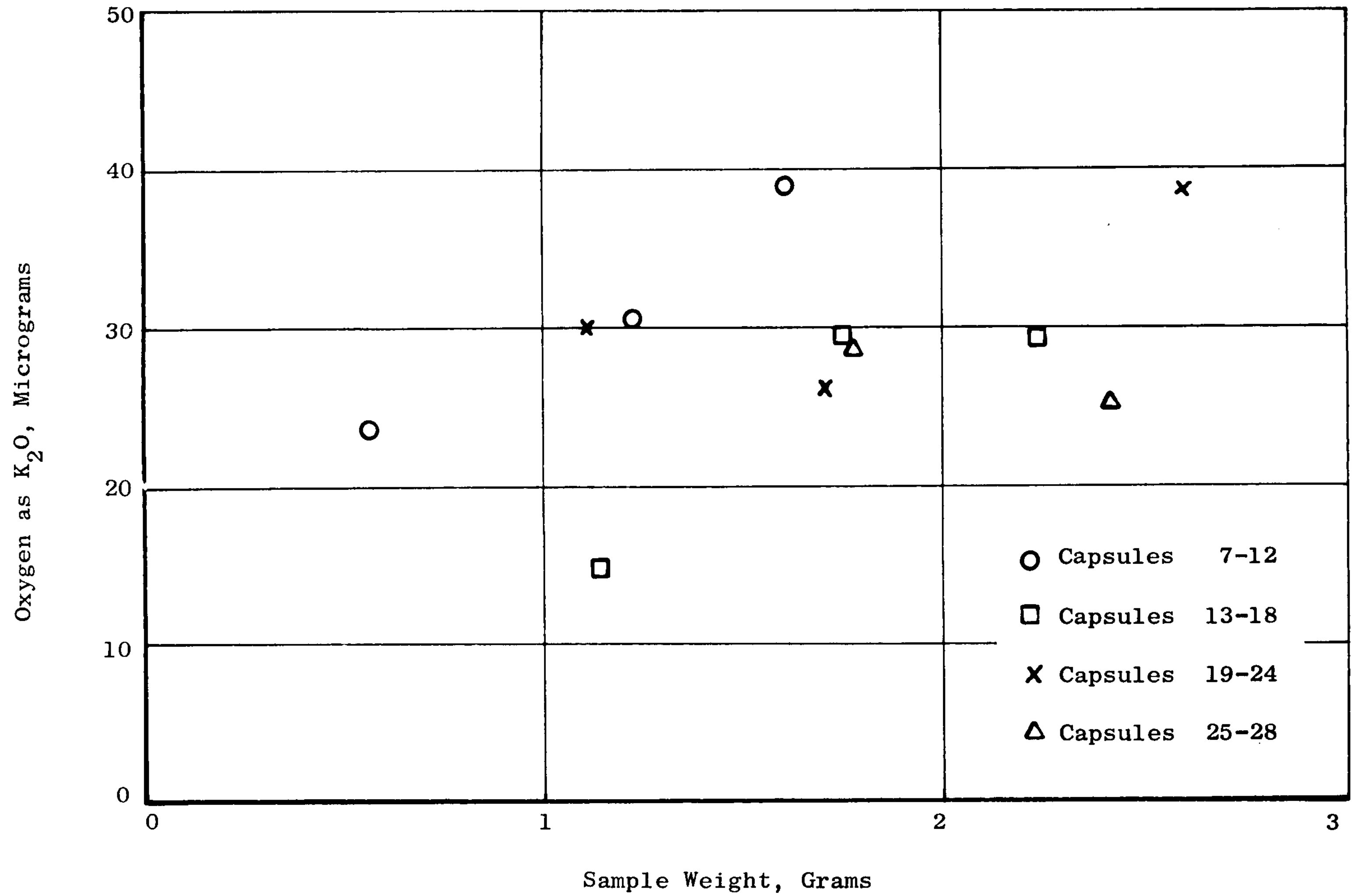


Figure 19. Oxygen Analyses of Potassium Samples Cast Inside Weld Chamber

data from the samples taken from the transfer lines outside the welding chamber more nearly conform to the expected rectilinear relationship than the data for samples that were cast inside the chamber. Also, the values obtained from the cast samples were generally slightly higher. However, a statistical treatment of the data in Figure 18 yields a correlation coefficient of 0.85 and a standard deviation of 5.4 micrograms, both of which are too high to be of real significance. An analytical blank exists which has not been eliminated. Although the above statistical treatment indicates that the blank was about 5 micrograms, it is believed that it is actually larger. The consensus is that the actual oxygen content of the potassium coming from the hot trap is less than 5 ppm and that the amount of contamination occurring during filling is probably not greater than another 5 ppm.

It should be pointed out that the oxygen values shown in Table IV are calculated assuming oxygen as K_2O in potassium. The metallic impurity values are calculated as weight of impurity per weight of potassium chloride.

After each capsule was filled with approximately 12 grams of purified potassium, it was immediately sealed by electron beam welding. The pressure during fillings was maintained in the 10^{-5} torr range and the normal time required to fill and seal one capsule was approximately 30 minutes. Of the first eighteen capsules filled, 4 capsules were rejected because of improper potassium levels as shown by radiographic examination of each capsule and also by the weight of potassium in the capsules. Modifications were made in the measuring cup and 4 capsules were filled to replace the ones not used. Radiographic inspection also revealed that three capsules lacked full penetration welds in the top end cap. These welds were repaired and reinspected prior to testing. The capsules were then grit blasted with 50-micron Al_2O_3 grit.

The completed capsules were placed in the corrosion test facility, described previously (Ref. 3 and page 16 of this report), which is located in a Varian high vacuum chamber, shown in Figure 20. The chamber is 24 inches in diameter x 54 inches high and is made of Type 304 stainless steel. The chamber is equipped with 4 cryogenic molecular sieve roughing pumps, a 1000 ℓ/sec getter-ion pump and it is bakeable to 750°F.

The capsules were instrumented on both the top and bottom end caps with Pt vs Pt+10%Rh thermocouples. Two thermocouples which had been calibrated at zinc, aluminum and copper melting points were installed on capsules in each susceptor. These thermocouples will be recalibrated at the end of each 1,000 hours of testing to evaluate the stability of the thermocouples in high vacuums.

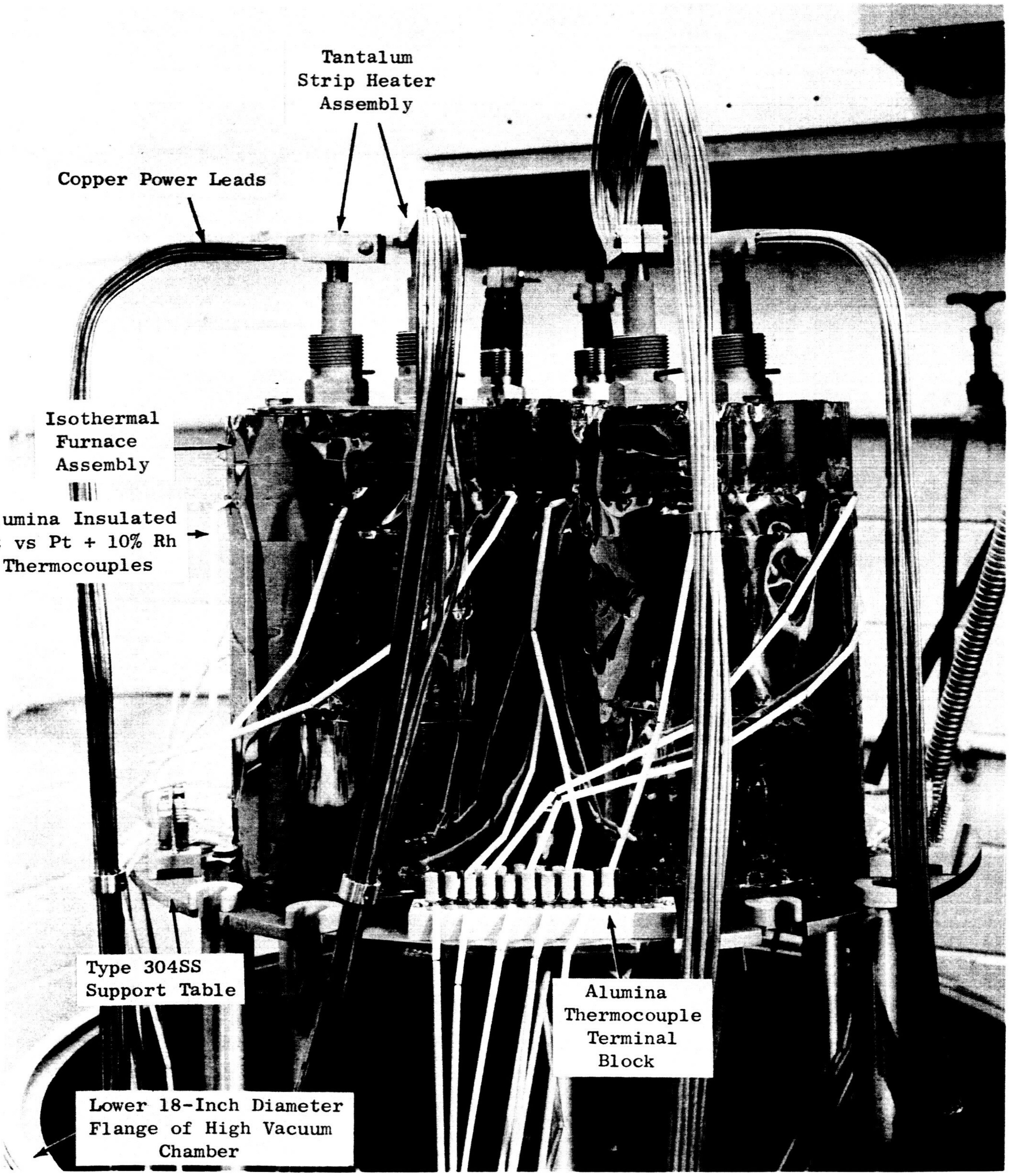


Figure 20. Isothermal Corrosion Capsule Test Facility Prior to the First 1,000-Hour Test at 800°, 1200° and 1600°F. (C64072401)

The chamber was sealed, evacuated to 3.8×10^{-6} torr and given a 10-hour bakeout at 400°F after which the pressure dropped to 4×10^{-8} torr. The testing sequence consisted of initiating the 1600°F test first, followed by the 800°F test and then the 1200°F test. Approximately 45 hours were required to bring each susceptor up to the test temperature and still maintain the chamber pressure at less than 1×10^{-6} torr. Twenty hours after the 1200°F test reached the test temperature the pressure in the chamber was 8×10^{-8} torr. The plot of the pressure change in the test is shown in Figure 21. All pressure values are obtained from a tubular Bayard-Alpert ionization gauge located on the side of the chamber. The mean test temperature and their deviations are shown in Table XIX.

Thirty-four additional Cb-1Zr alloy capsules were fabricated and cleaned in the same manner as discussed earlier. Of these, fifteen capsule assemblies containing K601, Star J, TiC, TiC+10%Cb and Grade 7178 specimens have been prepared. The capsule assemblies are now ready for filling with potassium and electron beam sealing.

A vacuum distillation apparatus was designed for the cleaning of the corrosion test specimens after exposure to potassium. A schematic of the facility is shown in Figure 22. Verbal approval has been received from the NASA Technical Manager to proceed with the construction of the facility and all the necessary materials are on order. The operating conditions will be determined by appropriate checkout tests prior to the actual cleaning of test specimens.

Dimensional Stability

After the completion of the second checkout run, the two test facilities, described previously (Ref. 3, 4) and on page 22 of this report, were loaded with duplicate specimens of each of 10 materials in preparation for testing at 1600° and 1200°F. Table XX lists the materials to be tested along with their identifying numbers, condition and test temperature. Note that one additional specimen of each of 3 materials was heat treated for 1 hour at 1800°F in vacuum and is to be tested at 1600°F to determine whether residual grinding stresses will influence the dimensional stability.

Before the specimens were installed in the test facility, they were cleaned individually in ethyl alcohol and accurately measured to ± 0.00005 inch. The specimens were then ultrasonically cleaned in ethyl alcohol, rinsed in ethyl alcohol, air dried for 30 minutes and then weighed to ± 0.0005 gram. Immediately after weighing, each specimen was placed in a separate Cb-1Zr alloy box. Then the specimens were placed in the susceptors in random order as shown in Tables XXI and XXII. The Pt vs Pt+10%Rh thermocouples were installed in the same manner and location as was used for the second checkout run (see page 27 of this report). The specific location of each thermocouple is indicated in Table XXI and XXII.

After the test setup was completed, the chamber was closed, evacuated to 1×10^{-6} torr and given a 12-hour bakeout at 572°F. At the end of the reporting period the 1200°F test had accumulated 440 hours and the 1600°F test had accumulated 540 hours of test with the pressure at 8.5×10^{-9} torr as indicated by a Bayard-Alpert ionization gauge.

TABLE XIX: TEST TEMPERATURES FOR FIRST 1,000-HOUR ISOTHERMAL CAPSULE CORROSION TEST

Test Temperature, °F	Mean Temperature, °F	Mean Temp Deviation °F	%
1600	1594	± 14.8	± 0.932
1200	1199	± 13.2	± 1.09
800	795	± 9.3	± 1.2

TABLE XX: IDENTITY OF TEST SPECIMENS FOR DIMENSIONAL STABILITY TEST No. 1

Material	Identity	Condition	Test Temperature, °F
Carboloy Grade 999	MCN 1035-B-1	As-Received	1600
Carboloy Grade 999	MCN 1035-B-2	As-Received	1600
Carboloy Grade 999	MCN 1035-B-3	As-Received	1200
Carboloy Grade 999	MCN 1035-B-4	As-Received	1200
Carboloy Grade 907	MCN 1036-B-1	As-Received	1600
Carboloy Grade 907	MCN 1036-B-2	As-Received	1600
Carboloy Grade 907	MCN 1036-B-3	As-Received	1200
Carboloy Grade 907	MCN 1036-B-4	As-Received	1200
Carboloy Grade 907	MCN 1036-B-11	As-Received + 1 Hour at 1800°F - Vacuum	1600
Mo-TZM	MCN 1037-B-1	As-Received	1600
Mo-TZM	MCN 1037-B-2	As-Received	1600
Mo-TZM	MCN 1037-B-3	As-Received	1200
Mo-TZM	MCN 1037-B-4	As-Received	1200
Mo-TZM	MCN 1037-B-9	As-Received + 1 Hour at 1800°F - Vacuum	1600
Tungsten	MCN 1038-B-1	As-Received	1600
Tungsten	MCN 1038-B-2	As-Received	1600
Tungsten	MCN 1038-B-3	As-Received	1200
Tungsten	MCN 1038-B-4	As-Received	1200
Lucalox (Al_2O_3)	MCN 1039-B-1	As-Received	1600
Lucalox (Al_2O_3)	MCN 1039-B-2	As-Received	1600
Lucalox (Al_2O_3)	MCN 1039-B-3	As-Received	1200
Lucalox (Al_2O_3)	MCN 1039-B-4	As-Received	1200
Lucalox (Al_2O_3)	MCN 1039-B-12	As-Received + 1 Hour at 1800°F - Vacuum	1600
ZrO_2	MCN 1040-B-1	As-Received	1600
ZrO_2	MCN 1040-B-2	As-Received	1600
ZrO_2	MCN 1040-B-3	As-Received	1200
ZrO_2	MCN 1040-B-4	As-Received	1200
K601	MCN 1041-B-1	As-Received	1600
K601	MCN 1041-B-2	As-Received	1600
K601	MCN 1041-B-3	As-Received	1200
K601	MCN 1041-B-4	As-Received	1200

TABLE XX (cont'd)

Material	Identity	Condition	Test Temperature, °F
TiC	MCN 1042-B-1	As-Received	1600
TiC	MCN 1042-B-2	As-Received	1600
TiC	MCN 1042-B-3	As-Received	1200
TiC	MCN 1042-B-4	As-Received	1200
TiC+10%Cb	MCN 1045-B-1	As-Received	1600
TiC+10%Cb	MCN 1045-B-2	As-Received	1600
TiC+10%Cb	MCN 1045-B-3	As-Received	1200
TiC+10%Cb	MCN 1045-B-4	As-Received	1200
Grade 7178	MCN 1045-B-1	As-Received	1600
Grade 7178	MCN 1045-B-2	As-Received	1600
Grade 7178	MCN 1045-B-3	As-Received	1200
Grade 7178	MCN 1045-B-4	As-Received	1200

TABLE XXI: LOCATION OF SPECIMENS FOR DIMENSIONAL STABILITY TEST RUN NO. 1
1600°F TEST

Specimen	Column 1	Column 2	Column 3	Column 4	Column 5
1 (Top)	Mo-TZM* MCN 1037-B-1	TiC* MCN 1042-B-1	Grade 999* MCN 1035-B-1	W* MCN 1038-B-2	TiC* MCN 1042-B-2
2	K601 MCN 1041-B-1	Grade 907* MCN 1036-B-1	TiC+10%Cb MCN 1045-B-1	Grade 7178* MCN 1046-B-1	K601 MCN 1041-B-2
3	Grade 907 MCN 1036-B-2	Lucalox MCN 1039-B-12	Mo-TZM MCN 1037-B-9	TiC+10%Cb MCN 1045-B-2	Lucalox MCN 1039-B-1
4	Grade 907* MCN 1036-B-2	Grade 7178 MCN 1046-B-2	Mo-TZM MCN 1037-B-2	ZrO_2 MCN 1040-B-2	Lucalox* MCN 1039-B-2
5	Mo	Grade 999* MCN 1045-B-2	ZrO_2* MCN 1040-B-2	W* MCN 1038-B-1	Mo
6 (Bottom)	Mo	Mo	Mo	Mo	Mo

* Indicates thermocouples are attached to these specimen boxes.

TABLE XXII: LOCATION OF SPECIMENS FOR DIMENSIONAL STABILITY TEST NO. 1
1200°F TEST

Specimen	Column 1	Column 2	Column 3	Column 4	Column 5
1 (Top)	Mo-TZM* MCN 1037-B-3	TiC* MCN 1042-B-3	Grade 999* MCN 1035-B-3	W* MCN 1038-B-4	TiC* MCN 1042-B-4
2	K601 MCN 1041-B-3	Grade 907* MCN 1036-B-3	TiC+10%Cb MCN 1045-B-3	Grade 7178 MCN 1046-B-3	K601 MCN 1041-B-4
3	Grade 907 MCN 1036-B-4	Grade 7178 MCN 1046-B-4	Mo-TZM MCN 1037-B-4	TiC+10%Cb MCN 1045-B-4	Lucalox MCN 1039-B-3
4	W* MCN 1038-B-3	Grade 999* MCN 1045-B-4	ZrO_2* MCN 1040-B-4	ZrO_2* MCN 1040-B-3	Lucalox* MCN 1039-B-2
5	Mo	Mo	Mo	Mo	Mo
6 (Bottom)	Mo	Mo	Mo	Mo	Mo

* Indicates thermocouples are attached to these specimen boxes.

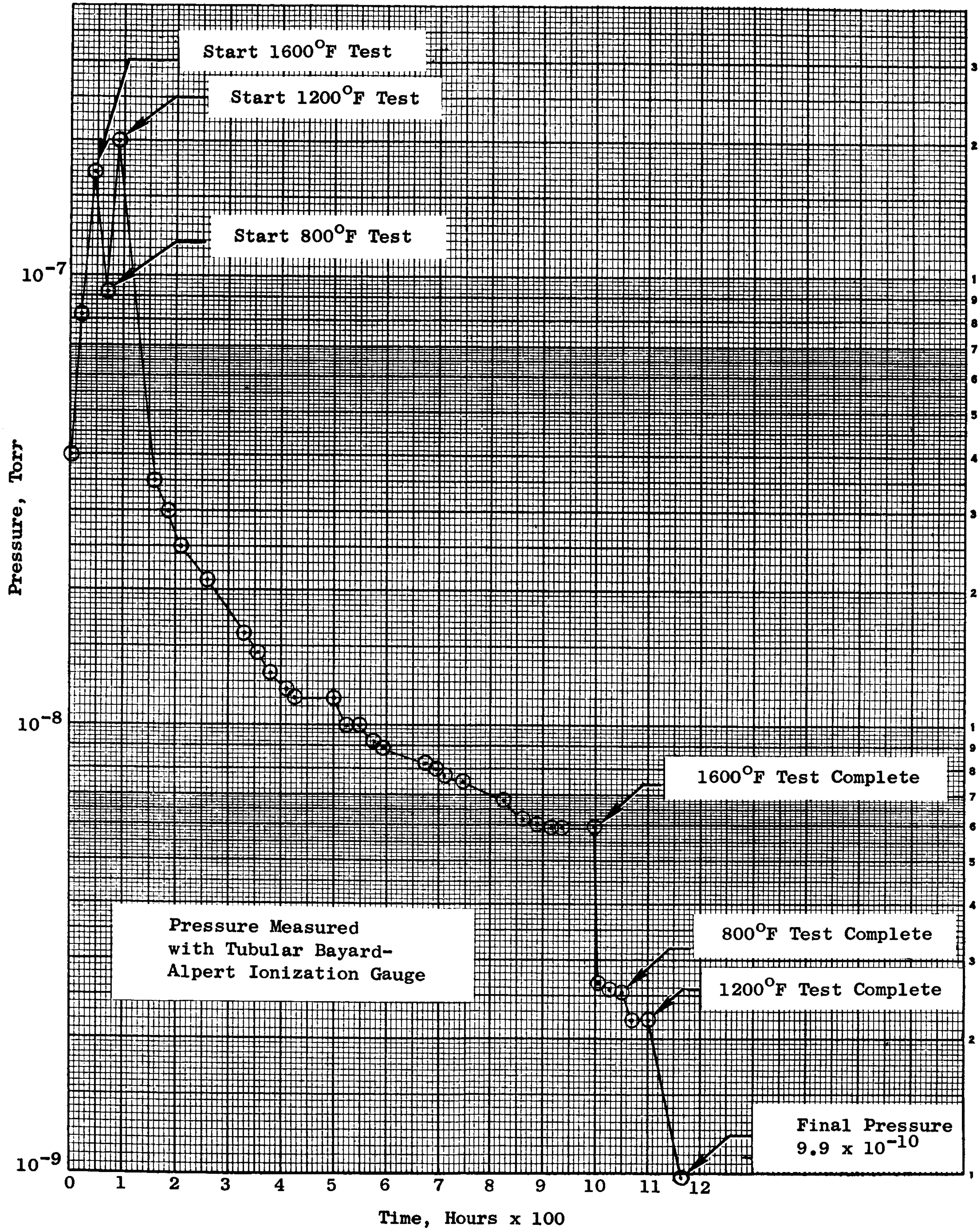


Figure 21. Pressure Curve for First 1,000-Hour Isothermal Corrosion Capsule Test.

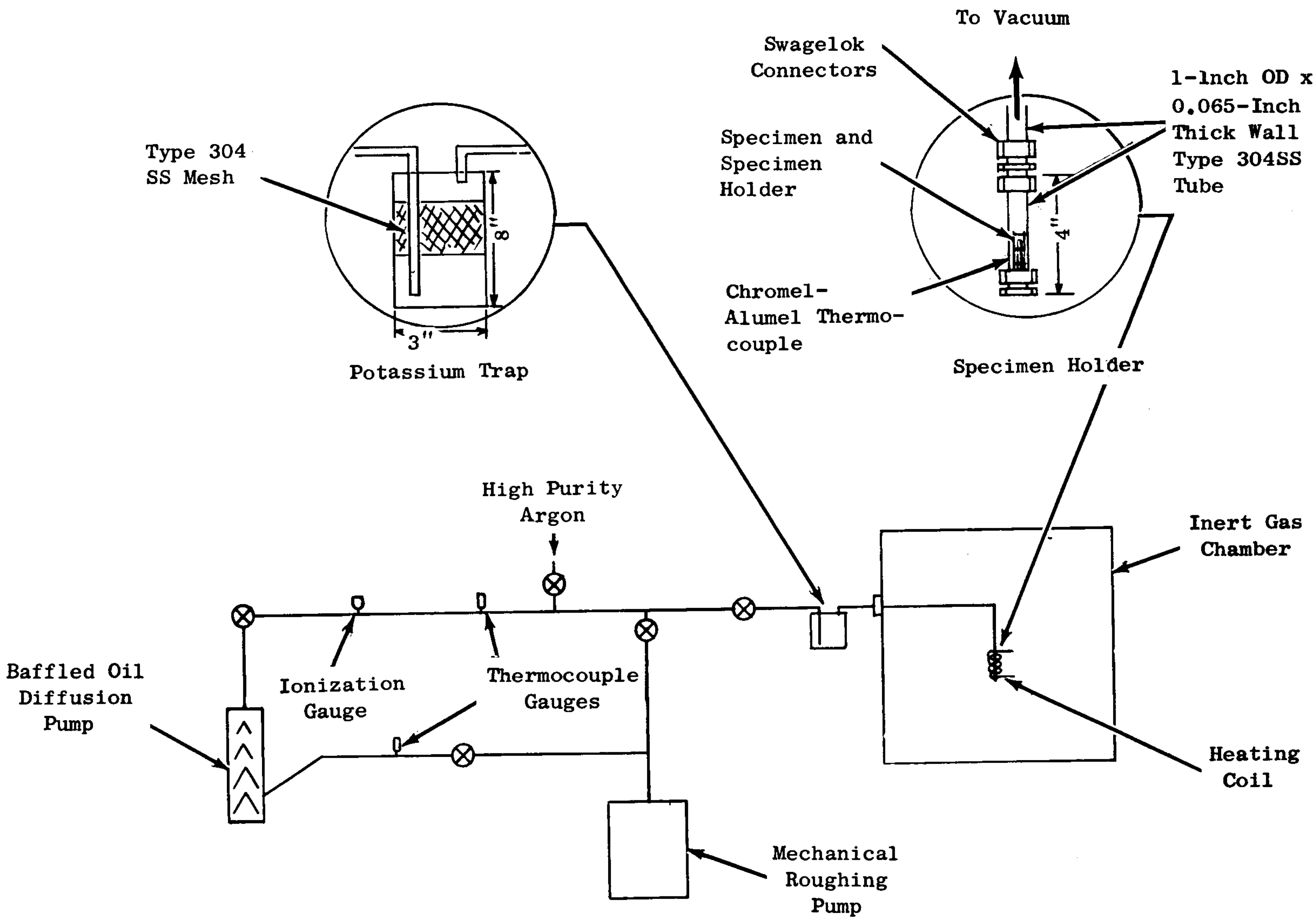


Figure 22. Vacuum Distillation Cleaning Facility for Corrosion Specimens After Exposure to Potassium.

VI. FUTURE PLANS

The summary which follows enumerates the steps to be pursued during the succeeding quarter to implement this study.

1) All of the remaining test specimens for the corrosion, dimensional stability, thermal expansion, hot hardness and compression test programs will be received.

2) The second 1,000-hour isothermal capsule corrosion test, incorporating 4 test facilities (2 at 1600°F, 1 at 1200°F and 1 at 800°F) and 20 capsules, will be completed.

3) The first 1,000-hour dimensional stability test covering 10 materials at 1600°F and 1200°F will be completed and a second 1,000-hour test will be initiated.

4) The hot hardness and thermal expansion test programs will be initiated.

5) Fabrication of the components for the load train for the compression tests will be completed and the facility checked out using Mo-TZM test specimens.

6) The design of the potassium heater for the friction and wear tester will be finalized and component parts ordered.

7) Purchase orders will be placed for the construction of the high vacuum friction and wear test rig.

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