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GENERATION OF LONG TIME CREEP DATA ON REFRACTORY ALLOYS AT ELEVATED TEMPERATURES

FIFTH QUARTERLY REPORT

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TRW EQUIPMENT LABORATORIES
THOMPSON RAMO WOOLDRIDGE INC.
CLEVELAND, OHIO

Fifth Quarterly Report

for

June 26, 1964 to September 26, 1964

GENERATION OF LONG TIME CREEP DATA OF REFRACTORY ALLOYS AT ELEVATED TEMPERATURES

CR 54228

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National Aeronautics and Space Administration

Contract No. NAS 3-2545

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November 9, 1964

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FOREWORD

The work described herein is being performed by Thompson Ramo Wooldridge Inc. under the sponsorship of the National Aeronautics and Space Administration under contract NAS-3-2545. The purpose of this study is to obtain design creep data on refractory metal alloys for use in space power systems.

The program is administered for Thompson Ramo Wooldridge Inc. by E. A. Steigerwald, Program Manager. J. Sawyer is the Principal Investigator. H. Philleo and R. Ebert contributed to the program.

ABSTRACT

Twelve of the fourteen ultra-high vacuum creep furnaces and the central control console are now being installed and two of these units have been placed in operation. Creep data are presented for FS-85 columbium alloy at 2000°F, 7000 psi and for tungsten-25% rhenium alloy at 3200°F, 5000 psi. Various problems affecting the 1,000 and 10,000 hour reliability of the system have been encountered. These problems and the corrective measures taken are described.

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I. INTRODUCTION

The object of this program is to obtain long-time creep data on selected refractory alloys for use in space power systems. Previous reports have described the approach to the problem, description of the equipment and methods, and some early creep test results.

During the fifth quarter, creep testing has been initiated and data have been obtained on FS-85 columbium and tungsten-25% rhenium alloys. Twelve additional ultra-high vacuum furnaces and the central control console were received and are being installed. Tests up to and during this time revealed the following equipment problems which affected the reliability of the systems:

1. Corrosion of the water-cooled insulators inside the vacuum chamber,

2. Distortion of gauge mark images due to the method of making the sight port, and

3. Control of temperature gradients.

Each of these problems has been examined and will be discussed in subsequent sections of this report.

II. CREEP TEST RESULTS

Two alloys—FS-85 columbium and tungsten-25% rhenium—were evaluated. The composition and processing history of these materials is summarized in Table I.

TABLE I

COMPOSITION AND PROCESSING HISTORY OF MATERIALS EVALUATED

	Composition	Weight %			
Alloy	W	Zr	Other	Cb	Processing History
FS-85* (sheet)	9.49	0.80	Ta-2.76	Bal	Received in worked condition recrystallized 1 hour at 2600°F
W-25 Re (sheet)	Bal		Re-25.5		Received in worked condition recrystallized 48 hours at 3200°F prior to load application

*Received from NASA.

Initial creep tests were performed on the FS-85 columbium alloy in an effort to allow a qualitative comparison to be made with existing NASA data and to evaluate the measurement techniques developed in this program. The test results obtained at 2000°F under a 4000 psi stress were presented in the previous quarterly report. The second test was also performed at 2000°F with an applied stress of 7000 psi. During the test, strain measurements were made every few minutes in the first hour and subsequently at the rate of approximately one a day. The creep data are presented in Table II and summarized in Figures 1 and 2. As shown in Figure 1, significant primary creep occurred during the first twenty minutes under load.

Two data points, marked with arrows in Figure 2, fell considerably below the average curve. This deviation resulted from loosening of a set screw holding the lower penta prism on the optical extensometer. The condition was corrected after these two data points were obtained. After 507.5 hours, the test was terminated due to a small water leak in the insulator. The pressure variation during the test is shown in Figure 3. The leak problem will be discussed further in a subsequent section of the report. A total creep of 0.075% (2" gauge length) was obtained in the FS-85 alloy after 507.5 hours at 2000°F and 7000 psi.

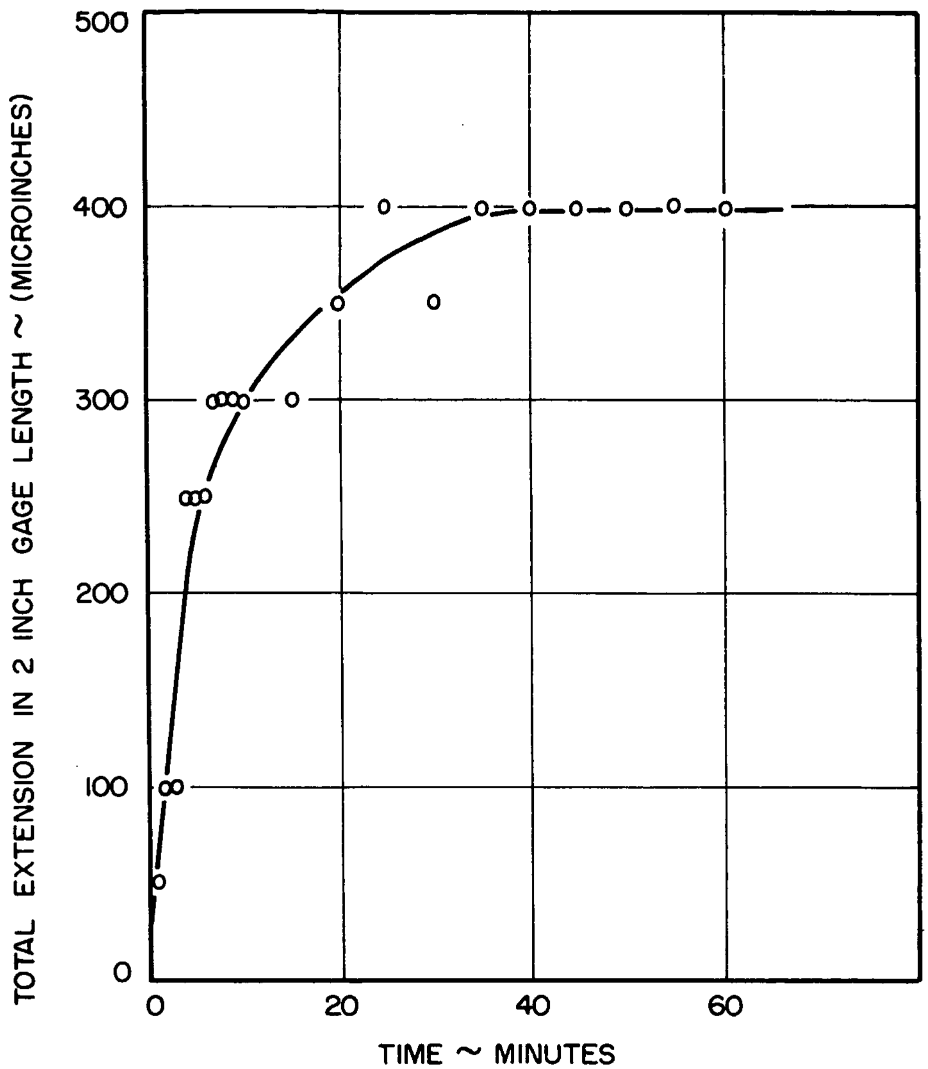
TABLE II

CREEP TEST DATA FOR FS-85 COLUMBIUM ALLOY SHEET* AT 2000°F, 7000 PSI

Time (Minutes)	Length Change ΔL (in)** (2" G. L.)	Strain (%)	Pressure (Torr)	Time (Hours)	Length Change ΔL (in)** (2" G. L.)	Strain (%)	Pressure (Torr)
1	5×10^{-5}	.0025	4.6×10^{-9}	2.1	4.7×10^{-4}	.024	4.3×10^{-9}
2	1.0×10^{-4}	.005		29.5	6.7×10^{-4}	.034	2.2×10^{-9}
3	1.0×10^{-4}	.005		48.0	7.0×10^{-4}	.035	2.0×10^{-9}
4	2.5×10^{-4}	.012		73.6	7.2×10^{-4}	.036	1.0×10^{-9}
5	2.5×10^{-4}	.012		97.1	6.5×10^{-4}	.032	9.6×10^{-10}
6	2.5×10^{-4}	.012		121.5	6.6×10^{-4}	.033	8.1×10^{-10}
7	3.0×10^{-4}	.015		140.4	8.4×10^{-4}	.042	7.2×10^{-10}
8	3.0×10^{-4}	.015		164.1	9.1×10^{-4}	.046	6.0×10^{-10}
9	3.0×10^{-4}	.015		219.6	1.04×10^{-3}	.052	8.3×10^{-10}
10	3.0×10^{-4}	.015		237.8	1.08×10^{-3}	.054	1.2×10^{-9}
15	3.0×10^{-4}	.015		262.4	1.13×10^{-3}	.056	1.0×10^{-9}
20	3.5×10^{-4}	.018		284.5	1.19×10^{-3}	.060	8.1×10^{-10}
25	4.0×10^{-4}	.020		313.3	1.27×10^{-3}	.064	6.5×10^{-10}
30	3.5×10^{-4}	.018		337.3	1.30×10^{-3}	.065	6.6×10^{-10}
35	4.0×10^{-4}	.020		402.9	1.45×10^{-3}	.072	8.1×10^{-10}
40	4.0×10^{-4}	.020		426.0	1.45×10^{-3}	.072	1.2×10^{-9}
45	4.0×10^{-4}	.020		449.1	1.48×10^{-3}	.074	1.0×10^{-9}
50	4.0×10^{-4}	.020		473.1	1.51×10^{-3}	.076	1.5×10^{-9}
55	4.0×10^{-4}	.020		497.0	1.51×10^{-3}	.076	5.0×10^{-7}
60	4.0×10^{-4}	.020		507.5	1.46×10^{-3}	.073	1.9×10^{-6}

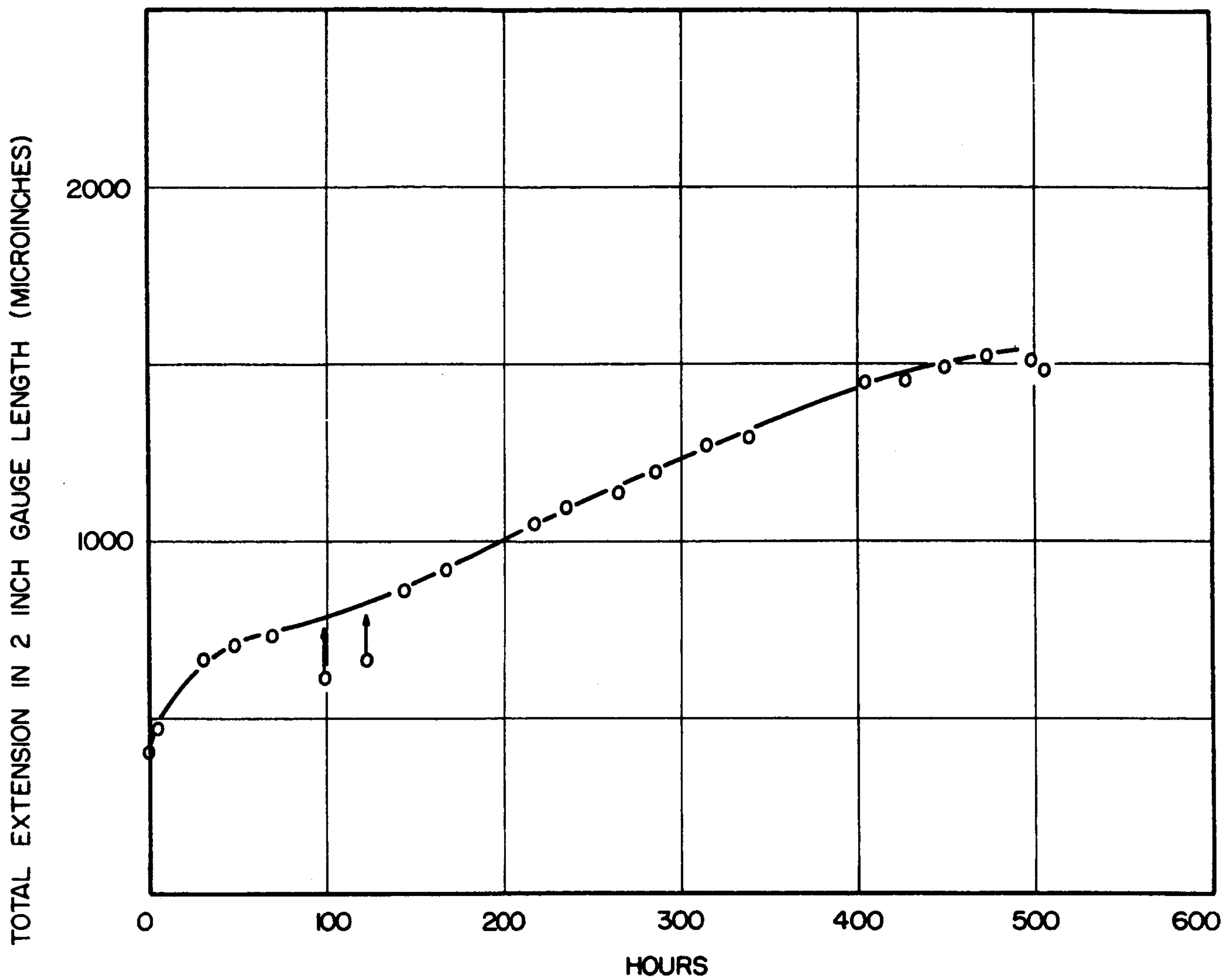
*Annealed 1 hour, 2600°F, pressure $< 1 \times 10^{-6}$ Torr.

**Strain values for 1-60 minute time intervals are single readings; all other values are averages of 10 readings.



CREEP DATA, TEST NO. 2 COLUMBIUM ALLOY FS-85, 2000°F, 7000 PSI, < 10^{-8} TORR, ONE HOUR PRIOR ANNEAL AT 2600° F.

FIG. 1



CREEP DATA, TEST NO. 2 COLUMBIUM ALLOY FS-85, 2000° F, 7000 PSI, < 10^{-8} TORR, ONE HOUR PRIOR ANNEAL AT 2600° F.

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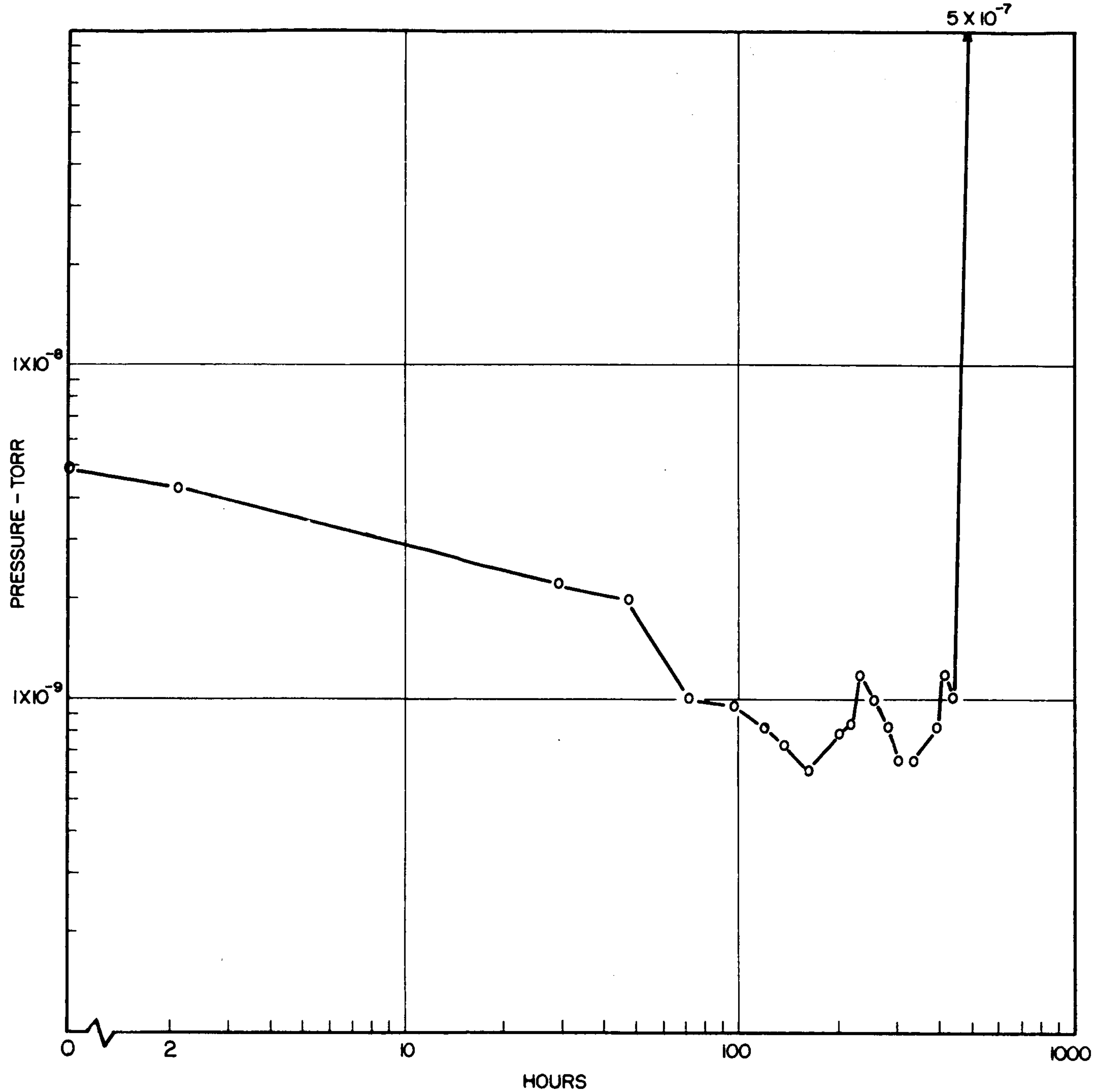
FIG. 2



VARIATION OF VACUUM WITH TIME FOR TEST NO. 2 COLUMBIUM ALLOY, FS-85 2000° F, 7000 PSI.

FIG. 3

A tungsten-25% rhenium sheet specimen (0.030" thick) was tested at 3200°F and 5000 psi stress. A total strain of 5% was obtained after 40.5 hours. The test was discontinued since 5% creep was selected as the end point criterion for sheet tests conducted for less than 1000 hours. The strain data are presented in Table III and Figure 4.

A comparison of the microstructure before and after testing is shown in Figures 5A and B. Substantial thermal etching occurred on the surface of the specimen during testing, and the photomicrograph in Figure 5B is representative of the surface without any subsequent preparation. Additional metallographic and chemical analysis of the tungsten-rhenium specimen is currently being conducted.

III. INSTALLATION OF CREEP FURNACES AND CONSOLE

During the period covered by this report, twelve new ultra-high vacuum creep units were received and are being installed. In contrast to the first unit, these chambers are connected to the central console which was also received during this period. Units (Numbers 3 through 7) are shown in Figure 6. Pipefitting and electrical work is nearly completed on all twelve systems and two of the systems have been operated at 3200°F.

The first unit received, in which all earlier tests were run, was returned to the vendor for modification to correct misalignment of the furnace opening and sight port.

The central control console as installed in the laboratory is shown in Figure 7. Starting at the left and going from top to bottom, the first section consists of a selector switch (A) and output terminals (B) for the thermocouples attached to the specimens. Initially, the true specimen temperature is determined from the readings of these thermocouples. The selector switch had not yet been installed when the photograph was taken. The next three panel sections below the thermocouple selector switch contain the temperature controllers, ammeters, voltmeters, and timers for furnaces 1, 2, and 3. Below these are two of the five power supplies (C) for the ion pumps. The five power supplies are connected in parallel and feed all fourteen vacuum units. The next cabinet contains the temperature controllers, etc., for furnaces 4, 5, 6, and 7. At the bottom of this cabinet is the constant temperature reference oven (D).

TABLE III

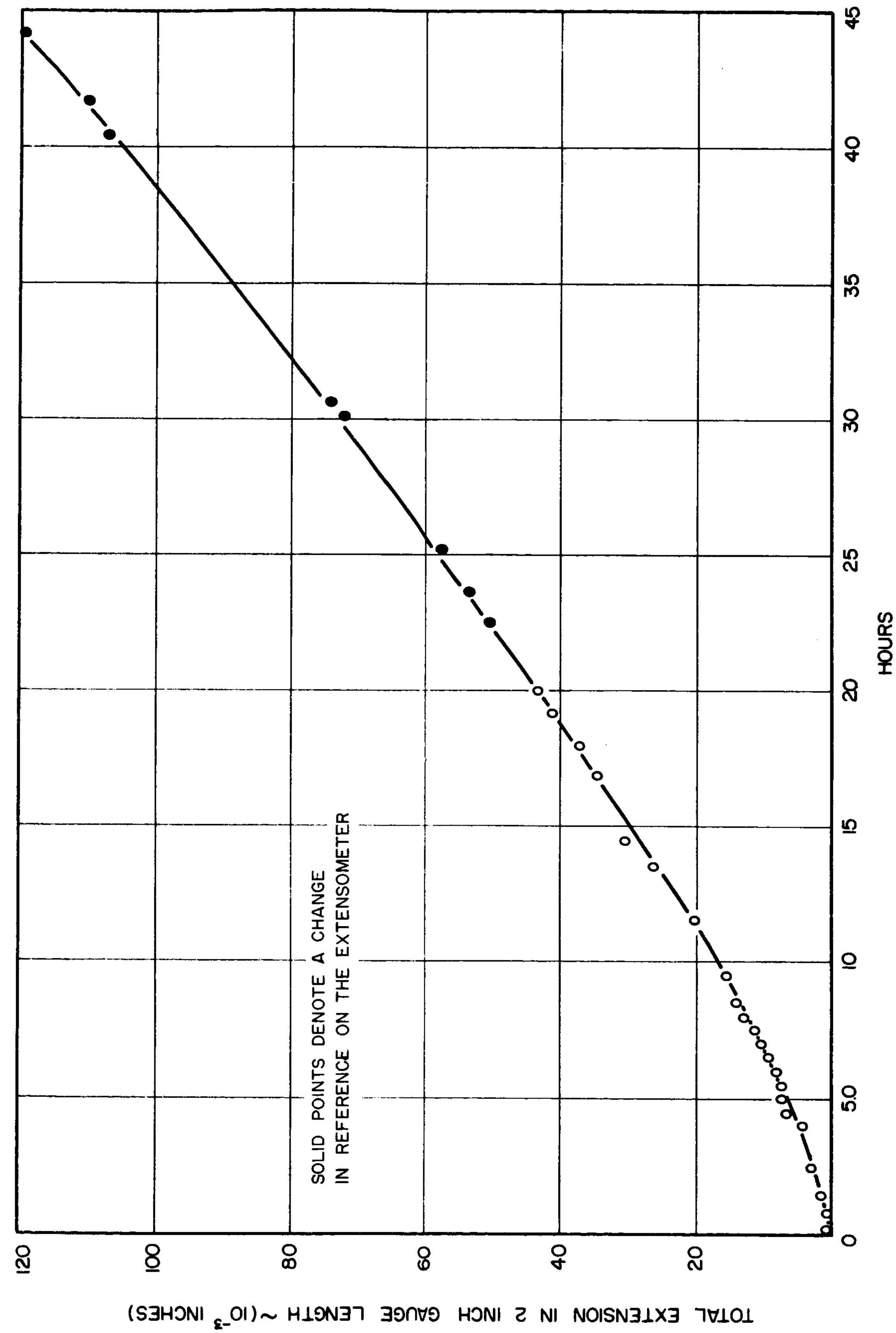
CREEP TEST OF W-25% RE ALLOY SHEET AT 3200°F AND 5000 PSI

Time (Minutes)	Length Change Δ L (in)* (2" G. L.)	Strain (%)	Pressure (Torr)
1			1.0×10^{-8}
2	2.5×10^{-4}	.013	
3	3.0×10^{-4}	.015	
4	3.0×10^{-4}	.015	
6	2.5×10^{-4}	.013	
8	2.5×10^{-4}	.013	
10	2.5×10^{-4}	.013	
15	2.0×10^{-4}	.010	
25	1.0×10^{-4}	.005	
35	2.5×10^{-4}	.013	
40	4.0×10^{-4}	.020	
45	6.0×10^{-4}	.030	

Time (Hours)	Length Change L (in)* (2" G. L.)	Strain (%)	Pressure (Torr)
1.5	1.30×10^{-3}	.065	
2.5	3.25×10^{-3}	.162	
4.0	4.90×10^{-3}	.245	
4.5	7.05×10^{-3}	.352	
5.0	7.25×10^{-3}	.362	
5.5	7.35×10^{-3}	.368	
6.0	8.45×10^{-3}	.422	
6.5	9.60×10^{-3}	.480	
7.0	1.085×10^{-2}	.542	
7.5	1.190×10^{-2}	.595	
8.0	1.315×10^{-2}	.658	
8.5	1.450×10^{-2}	.725	
9.5	1.575×10^{-2}	.788	
11.5	2.010×10^{-2}	1.00	
13.5	2.630×10^{-2}	1.32	
14.5	3.070×10^{-2}	1.54	
16.8	3.430×10^{-2}	1.72	7.8×10^{-9}
18.1	3.730×10^{-2}	1.86	
19.1	4.140×10^{-2}	2.07	
20.0	4.340×10^{-2}	2.50	
22.6**	5.095×10^{-2}	2.54	
25.2	5.725×10^{-2}	2.86	8.0×10^{-9}
30.0	7.215×10^{-2}	3.61	
30.6	7.41×10^{-2}	3.81	
40.5	1.067×10^{-1}	5.34	6.8×10^{-9}
41.4	1.099×10^{-1}	5.49	
44.5	1.203×10^{-1}	6.03	

*Strain values for first 60 minutes are individual readings. All other strain values are averages of ten readings.

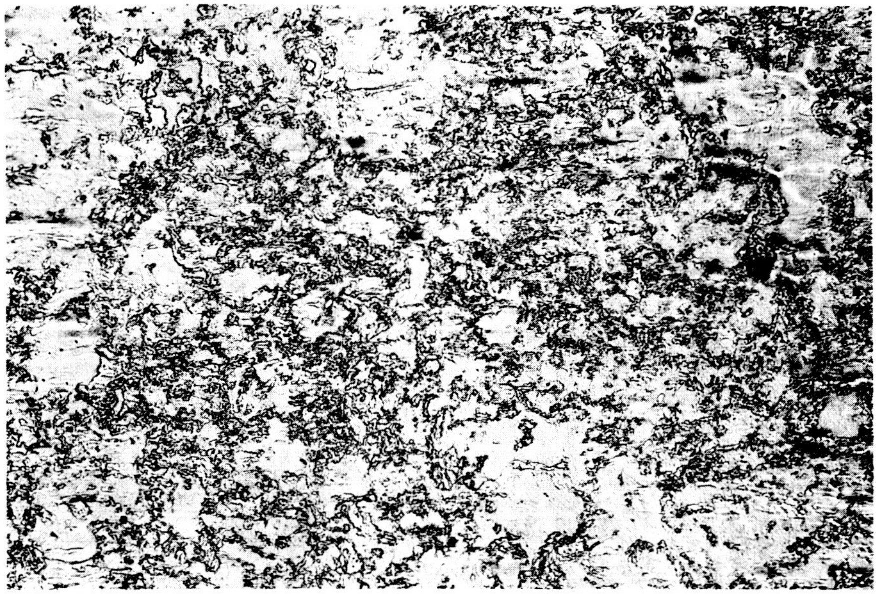
**Change was made in extensometer reference point. Readings taken after twenty hours were corrected for this change.



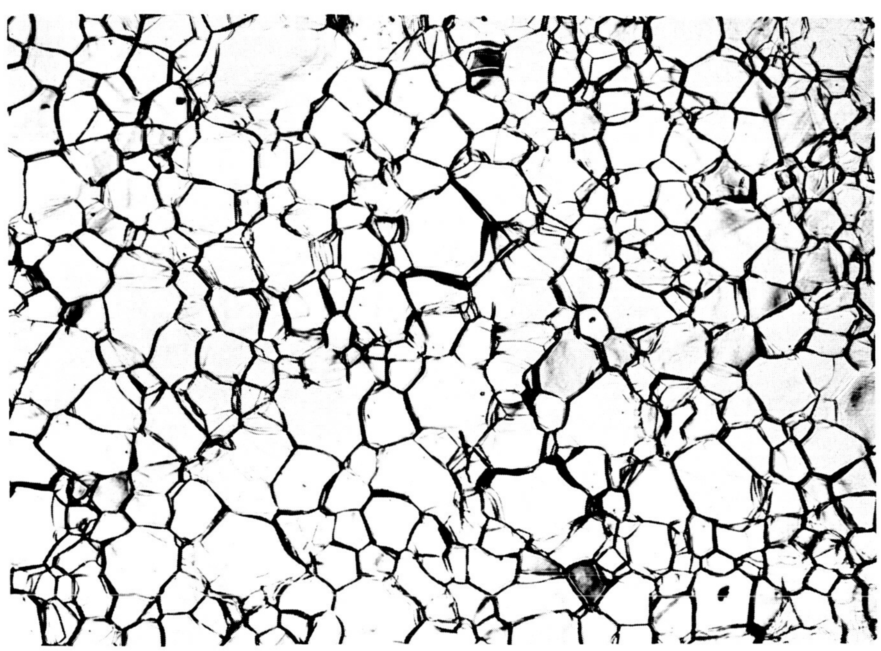
CREEP DATA, TUNGSTEN -25% RHENIUM, 3200°F, 5000 PSI. < 10^{-8} TORR,
48 HOUR ANNEAL AT 3200°F PRIOR TO LOAD APPLICATION.

6861

FIG. 4



a. 100X



b. 100X

(a) W-25% Re ALLOY, AS-RECEIVED SHEET SURFACE, ETCHANT; 15% HF, 15% H_2SO_4, 8% HNO_3, 62% H_2O.

(b) W-25% Re ALLOY, SURFACE OF SHEET AFTER 44.5 HOURS AT 3200°F IN VACUUM OF 1.0×10^{-8} TO 6.8×10^{-9} TORR. NO ETCHANT USED.

6863

FIG. 5

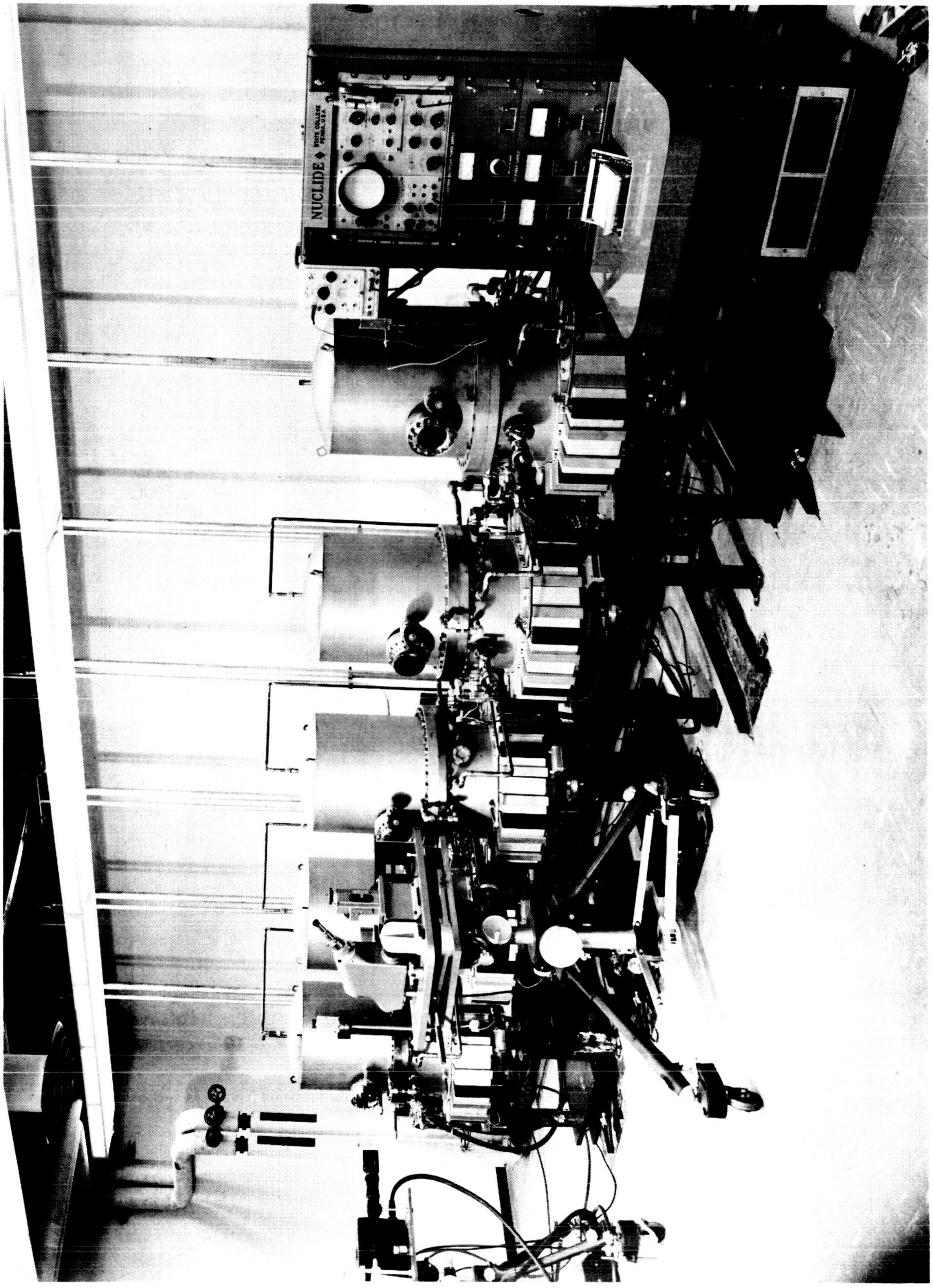


FIGURE 6 VACUUM CREEP FURNACES (NOS. 3 THROUGH 7) WITH RADIATION PYROMETER, EXTENSOMETER AND MASS ANALYZER

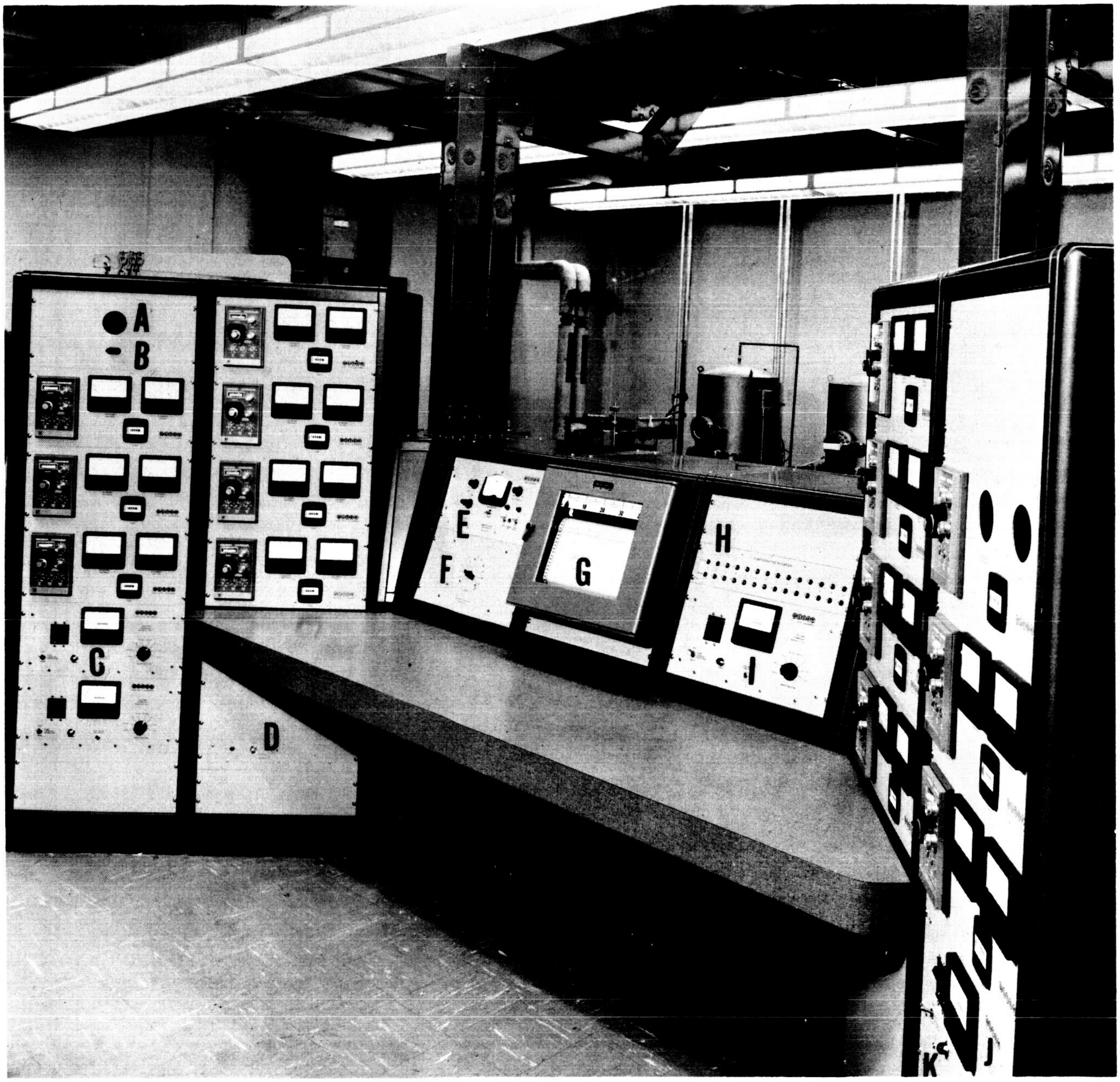


FIGURE 7 CONTROL CONSOLE FOR ULTRA-HIGH VACUUM CREEP FURNACES

All thermocouple extension lead wires come through the reference oven to the recording, measuring, and controlling instruments. At the top left of the center section is the vacuum gauge (E). The selector switch (F) below the vacuum gauge connects the read-out system to any of the fourteen Kreisman gauges mounted on each vacuum creep unit. The recorder (G) in the center of the panel records the temperatures for each of the fourteen furnaces in millivolts. To the right of the recorder are fourteen indicator lights (H) which show whether each of the two ion pumps on each vacuum unit is operating. Another power supply (I) for the ion pumps is below these lights. The two cabinets on the right contain the temperature controllers, ammeters, voltmeters, and timers for furnaces 8 through 14 and the remaining two ion pump power supplies (J and K).

IV. EQUIPMENT MODIFICATION

A. Corrosion

As previously mentioned, after 507.5 hours of operation at 2000°F, the second vacuum creep test with FS-85 had to be discontinued because of a leak in one of the water-cooled insulators (Figure 8) which support the heating element. Close inspection showed that the failure had occurred in the Kovar alloy due to pitting corrosion.

A discussion of the problem with the manufacturer of the rust inhibitor used in the cooling system resulted in the following recommendations:

1. Increase the chromate content of the cooling water to 1000-2000 ppm.

2. Place filters in the system to remove suspended solids from the coolant.

3. Control the chloride ion content of the coolant to 100 ppm or less.

In addition to controlling the cooling water, it was decided that the corrosion problem could be minimized by:

1. Redesign of the part to eliminate either the water or the Kovar, and

2. Coating the Kovar with a bakeable epoxy resin to protect it from the coolant.

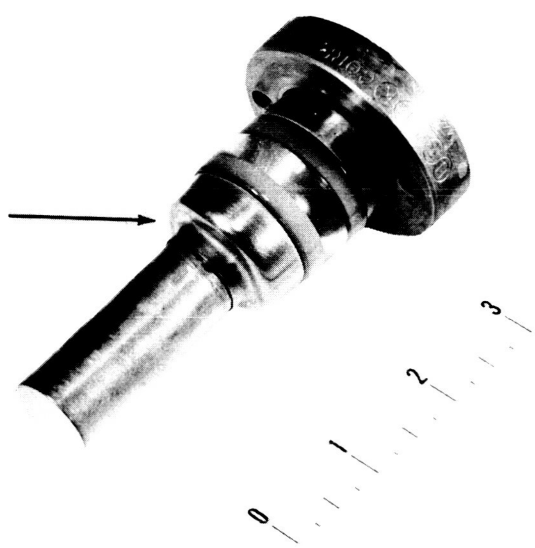


FIGURE 8 WATER-COOLED INSULATOR, ARROW INDICATES AREA WHERE PITTING CORROSION CAUSED LEAKS

A series of tests were run to determine the effectiveness of some of these measures. Coupons of Kovar were exposed to chromate-treated water (500 ppm) with a positive DC voltage of 18 volts applied to the specimens. The specimens showed pitting corrosion which appeared identical to that found in the insulators. Increasing the chromate content to about 1500 ppm reduced but did not eliminate the attack. Without the applied voltage, corrosion was much slower. Although the current supplied to the heating element is AC, not DC, there may be a partial rectification of the current so that some pulsating DC current could result.

Since the pitting corrosion observed on the test specimens closely approximated that found in the insulator, the laboratory test provided a simple technique to evaluate corrosion susceptibility. A photomicrograph taken at 30x (Figure 9) shows some of the pitting corrosion on the specimen exposed in plain tap water for 3 hours with a positive 18 volts DC applied voltage. The specimen immersed in water with 1500 ppm chromate showed very little pitting, and the specimen coated with epoxy novalac resin showed no pitting in the coated area.

As a result of these tests several insulators were coated with Dow DEN 438 epoxy novalac resin, and these components are now being tested. A second approach which involved complete elimination of the water in the critical insulator section is also being evaluated in the next series of creep tests. The effect of operating these leads uncooled on the ultimate vacuum will be determined and reported in the next quarterly report.

B. Filtration

The presence of small particles or suspensions in the coolant system can contribute to corrosion failures through the formation of concentration cells. Two large flow filters have been installed in the system (Figure 10) to remove contaminants without seriously restricting fluid flow. Each filter contains 22 replaceable cartridges, and the piping is arranged so that water flow can be switched through the other filter or through a bypass when the cartridges must be replaced. The filters are sized to remove all particles 10 microns or larger while giving a pressure drop of approximately two psi.

C. Sight Ports

Difficulty in making strain measurements was encountered due to slight distortions of the gauge-mark images caused by the glass in the sight port. The initial sight ports had a "Housekeeper Seal" which bonded optically flat Pyrex glass to Kovar. Although quite adequate for most purposes, this type of window was not suitable for use with the high precision optical extensometer. The glass near the edge of the port was slightly distorted by the

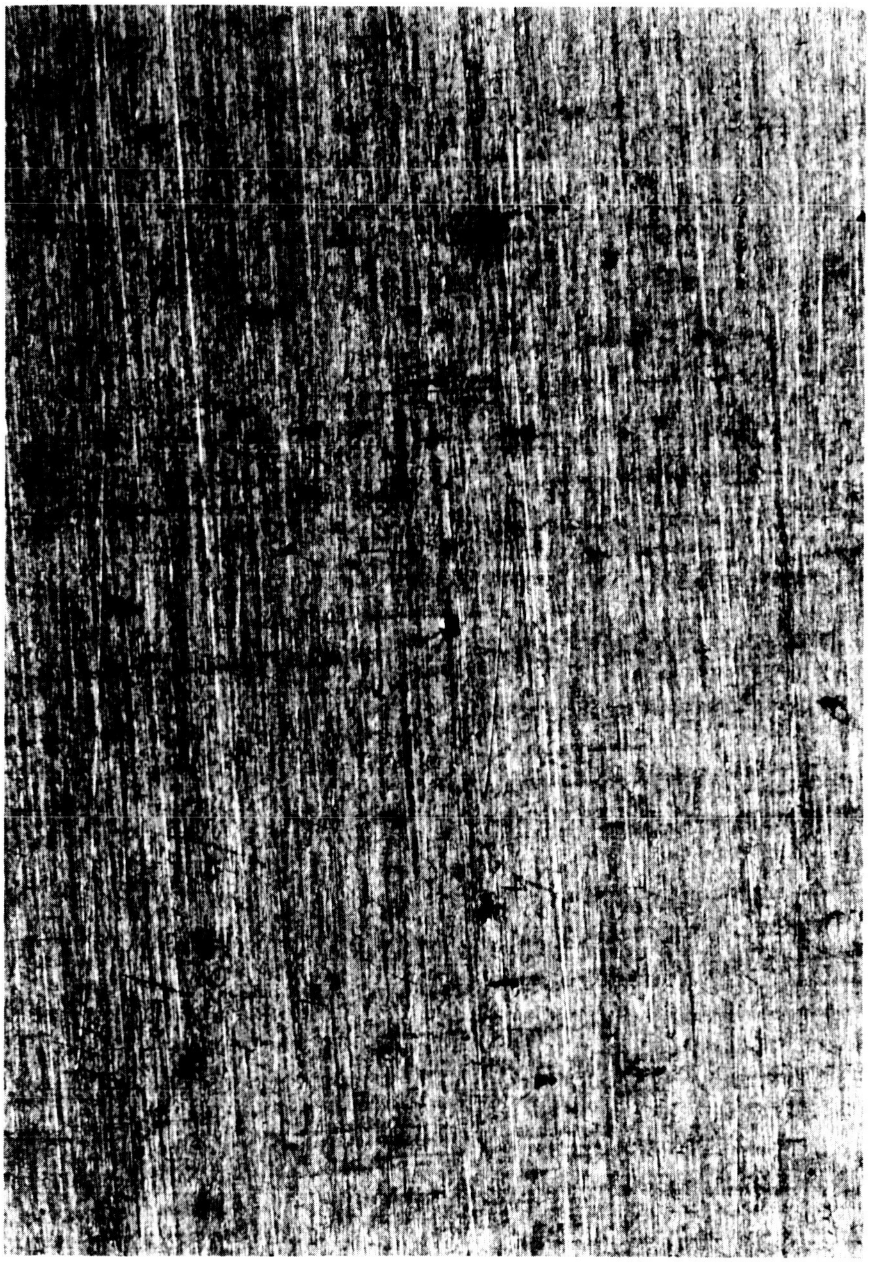


FIGURE 9 PHOTOMICROGRAPH OF KOVAR SPECIMEN SHOWING PITTING CORROSION. TWO HOURS EXPOSURE IN UNTREATED TAP WATER WITH 18 VOLTS DC APPLIED VOLTAGE

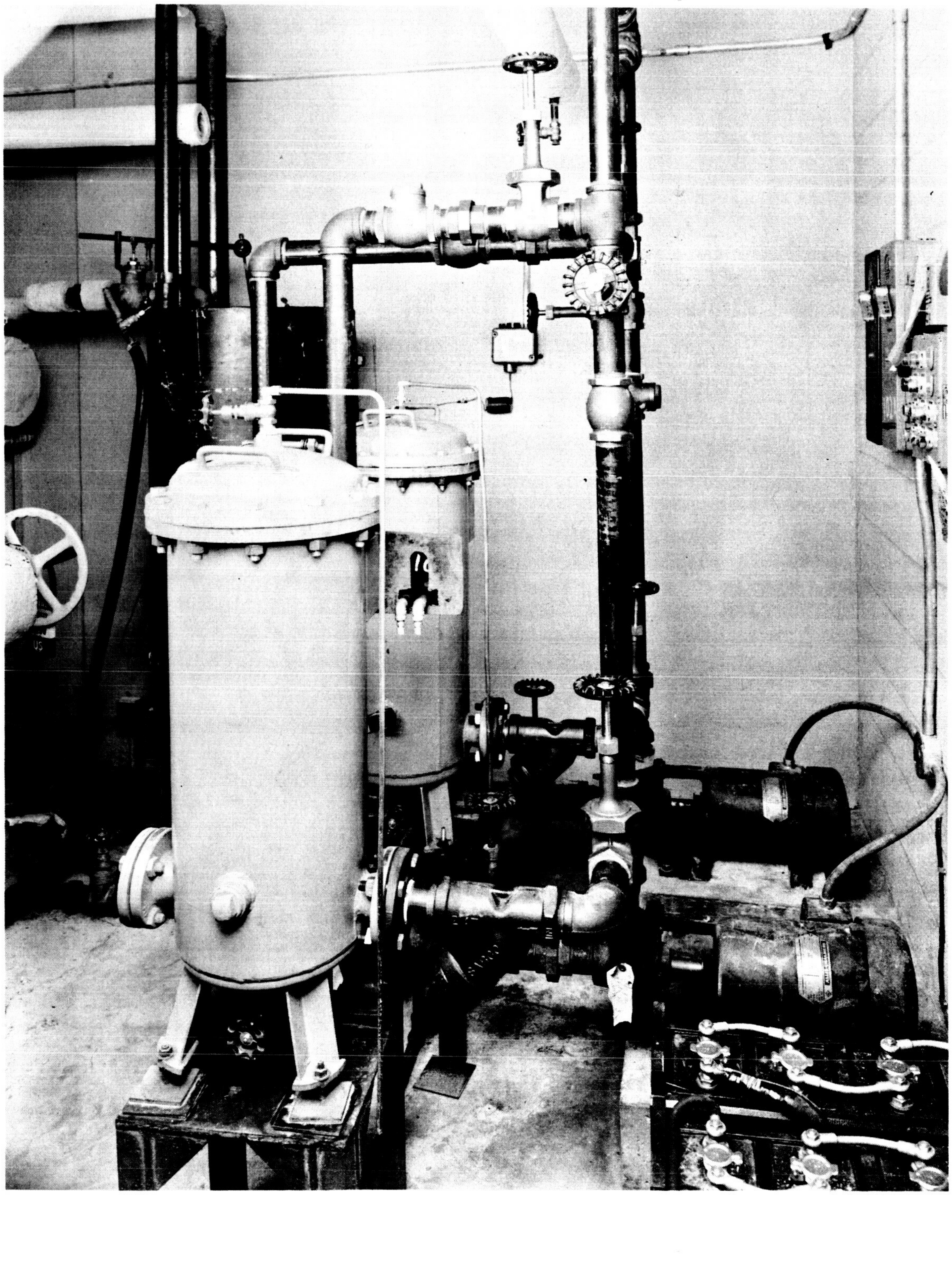


FIGURE 10 FILTERS IN WATER-COOLING SYSTEM

fusion sealing process, and this distortion altered the images received by the extensometer. Tests conducted with an optical flat showed that all sight ports of this type had appreciable curvature near the edge of the glass. Additional distortion due to atmospheric pressure, when the system is under vacuum, was found to be negligible. Because of this problem, a different sight port was designed to improve the surface flatness at the edge of the glass.

The two types of sight ports are shown in Figure 11. At the left is the unsatisfactory design which employs the "Housekeeper Seal." At the right is the revised port which has an optically flat Pyrex glass held between two gold O-ring seals. Although tests with an optical flat showed some distortion of the glass, it was not severe enough to influence the optical extensometer measurements. Redesigned ports are being made to replace the original type in all the vacuum chambers.

D. Temperature Gradient

In early tests with the first vacuum creep furnace, the temperature gradient was excessive; the temperature at the top of the 2-inch gauge section being nearly 30°F lower than at the bottom. To remedy this condition, a tantalum heat shield was installed which extended from the top of the heating element to slightly below the midpoint. This shield, which was attached as shown in Figure 12, reduced radiation losses in the upper section sufficiently to bring the temperature gradient within acceptable limits (± 5°F at 1800°F) under the existing conditions of temperature and specimen configuration.

V. STATUS OF ALLOYS TO BE TESTED

The following summary shows the status of the alloys to be tested in this program:

Alloy	Form	Source	Shipping Dates and Comments
FS-85	Sheet	NASA	Received and tested
TZM	Disc forging	Climax Molybdenum	Received
ST-222	Plate	Westinghouse	Raw material rejected by Westinghouse on 3 shipments. Expected shipment 11/20/64
Cb-132-M	Plate	Universal Cyclops	Original material cracked, reordered. Revised delivery 1/15/65.

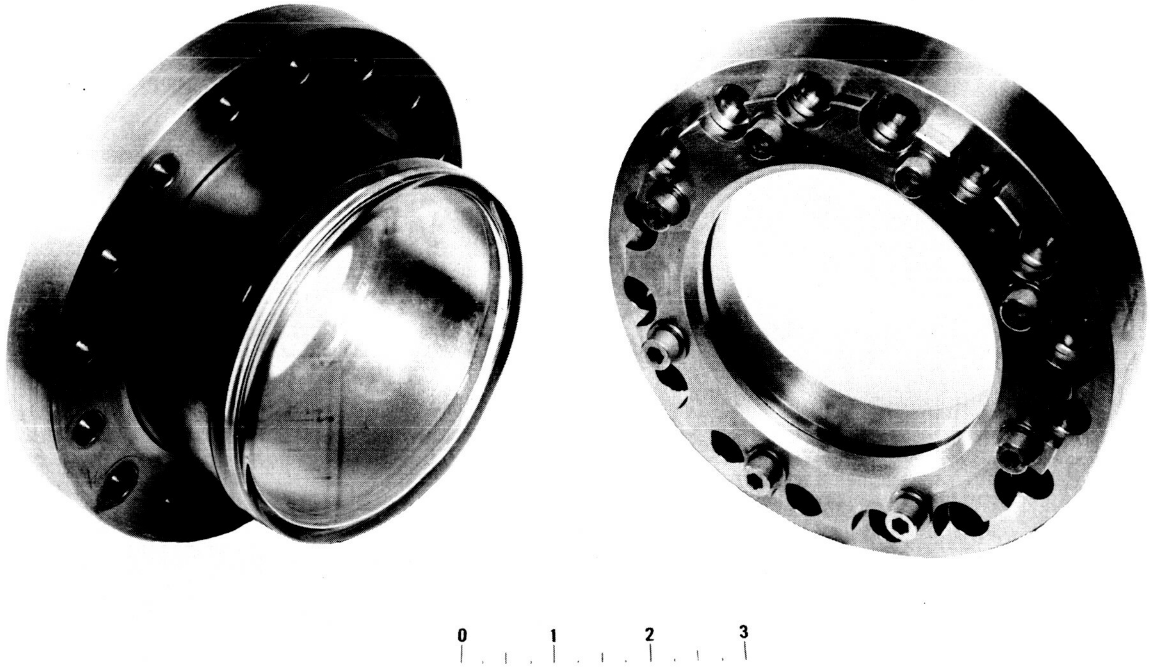


FIGURE 11 SIGHT PORTS FOR VACUUM CHAMBER. NEW TYPE IS AT RIGHT.

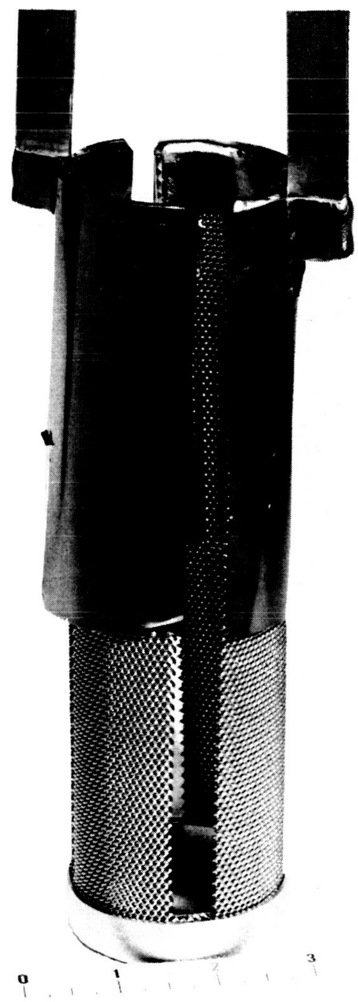


FIGURE 12 TUNGSTEN MESH HEATING ELEMENT WITH TANTALUM RADIATION SHIELDS ATTACHED TO IMPROVE GRADIENT

Alloy	Form	Source	Shipping Dates and Comments
TZC	Plate	General Electric	11/27/64
AS-30	Plate	General Electric	11/4/64
W	Sheet	Universal Cyclops	Received.
W-25% Re	Sheet	Wah Chang	Received and currently being tested.
Sylvania-A	Sheet	Sylvania	Original material cracked, reordered. Revised delivery 2/15/65.

VI. FUTURE WORK

Installation of the remaining creep units will be completed. Tests will be conducted at 3200°F on tungsten-25% rhenium and tungsten sheet alloys for times up to 1000 hours. Tests will be initiated on TZM and AS-30 turbine alloys at temperatures of 2000 and 2200°F. Primary emphasis will be placed on the latter type materials.

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