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

by J. C. Sawyer and E. A. Steigerwald

Prepared by
TRW EQUIPMENT LABORATORIES
Cleveland, Ohio
for Lewis Research Center

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

By J. C. Sawyer and E. A. Steigerwald

Prepared under Contract No. NAS 3-2545 by
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NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

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FOREWORD

The work described herein was performed by TRW Inc. under NASA Contract NAS 3-2545. The purpose of this study was to obtain design creep data on refractory metal alloys for use in advanced space power systems.

The program was administered for TRW Inc. by E. A. Steigerwald, Program Manager. J. C. Sawyer was the principal investigator, and R. R. Ebert contributed to the program. The Technical Manager was Paul E. Moorhead, Space Power Systems Division, NASA-Lewis Research Center. The report was originally issued as TRW Report ER-7203.

ABSTRACT

Creep tests were conducted on selected refractory alloys in a vacuum environment ($< 1 \times 10^{-8}$ torr) for times between 100 and 15,000 hours. Since the ultimate program goal was to provide design data for space electric power systems, particular emphasis was placed on measuring creep extension below 1%. The resulting data were evaluated in terms of the relative properties of columbium, molybdenum, tantalum, and tungsten-base alloys. Larson-Miller and Manson-Haferd techniques were used to present the data in parametric form. In the 1800°F (982°C) to 2200°F (1204°C) temperature range, the molybdenum-base alloys TZC and TZM possess the best creep properties for potential turbine applications. The variability of the creep properties as a function of heat of material was determined for the T-111 alloy.

In addition to comparison of alloy creep properties, chemical analysis, metallography, and tensile test data are presented to characterize each of the materials before and after creep testing.

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I INTRODUCTION

Material requirements for space electric-power systems necessitate the use of alloys which provide both high-temperature strength and resistance to liquid-metal corrosion. Alloys based on the refractory metals columbium, molybdenum, tantalum, and tungsten are currently the most suitable materials which meet these specifications.

The design of space electric-power systems which operate for a year or more requires a thorough knowledge of the elevated temperature creep properties of the alloys involved under conditions which simulate the service environment. While turbine and clad components of space power systems are not necessarily exposed to the vacuum of space, they do operate in environments such as metal vapor or liquid metals where the partial pressure of reactive gases is extremely low. Consequently, to obtain representative creep data it is necessary to perform tests in a vacuum environment of $< 1 \times 10^{-8}$ torr which causes little or no interstitial contamination over long periods of time.

The initial work of this program was to generate 1,000 hour creep data on selected refractory alloys which have potential use in advanced space electric-power systems. The materials examined were obtained in the form of rolled sheet, forged or rolled plate, and vapor-deposited tubing. The sheet and vapor-deposited tubing were representative of materials to be used for cladding and tubing applications while the plate provided material comparable to the form employed for turbine components. Following the initial tests, the alloys with the greatest creep strength were selected for additional evaluation aimed at developing 5,000 - 15,000 hour creep data for design purposes.

II EQUIPMENT

A. Creep Chambers

Creep testing of the refractory alloys was carried out in fourteen vacuum test chambers, seven of which are shown in Figure 1. The control console, shown in Figure 2, contains the necessary temperature controllers, ion pump power supplies, pressure readout, and other instrumentation for all fourteen systems (1)*.

Figure 3 and 4 are a photograph and schematic diagram of a single creep chamber with a double walled, water cooled removable bell. The only penetrations are a 4-inch diameter optically flat sight port slanted 5 degrees and a magnetically rotated shutter protecting the window. The spool piece, immediately below the main flange, is also double walled for water cooling. The spool piece penetrations consist of high and medium current feedthroughs, octal-tublar thermocouple feedthrough, cold cathode vacuum gage, roughing valve, and residual gas analyzer. The integral ion pump, beneath the spool piece, consists of sixteen cubicles which provide a pumping speed of at least 400 liters per second. An integral pump was used rather than an appendage pump because of the advantages of space conservation and possible higher pumping speeds. The external weight pan capable of applying 2000 pounds of dead-weight load to the specimen in the test chamber is located in the lower part of the unit. Lead weights are applied to the specimen by means of a hydraulic jack built into the weight support. The pull rod passing through the chamber utilizes a stainless steel bellows to isolate the penetration from the atmosphere. Because barometric pressure changes acting on the bellows cause the absolute magnitude of the load to vary, the minimum external weight for 1 percent accuracy of load is about 350 pounds. For lower loads, the internal weights of stainless steel are used, thus eliminating the bellows in the load train. In this case the pull rod attached to the external weight pan is replaced by an internal weight pan support which is actuated by the external hydraulic jack. With this arrangement, the internal weights can be applied to the specimen by the external jack acting through the bellows.

* Numbers in parentheses refer to references in the Bibliography.

FIGURE 1 SEVEN OF FOURTEEN ULTRA-HIGH VACUUM CREEP UNITS

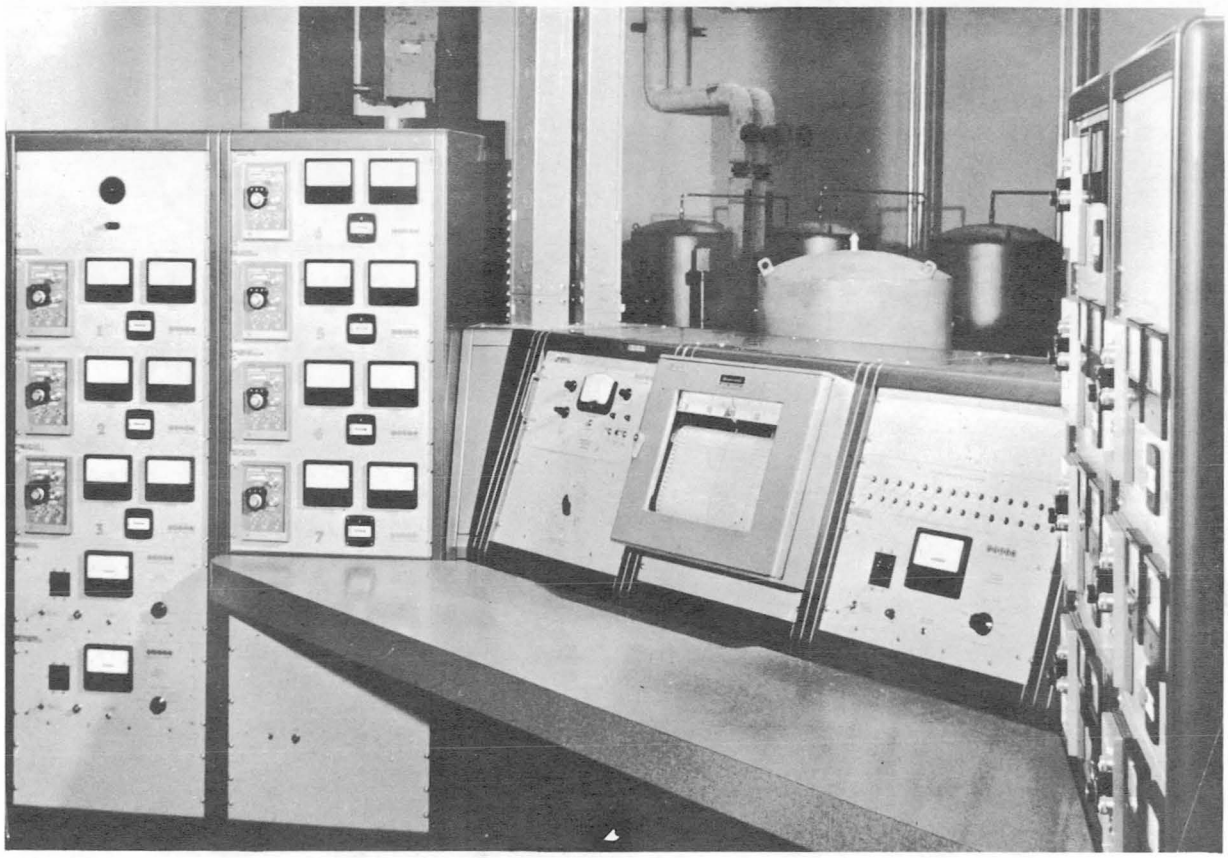


FIGURE 2 CONTROL CONSOLE FOR ULTRA-HIGH VACUUM CREEP LABORATORY

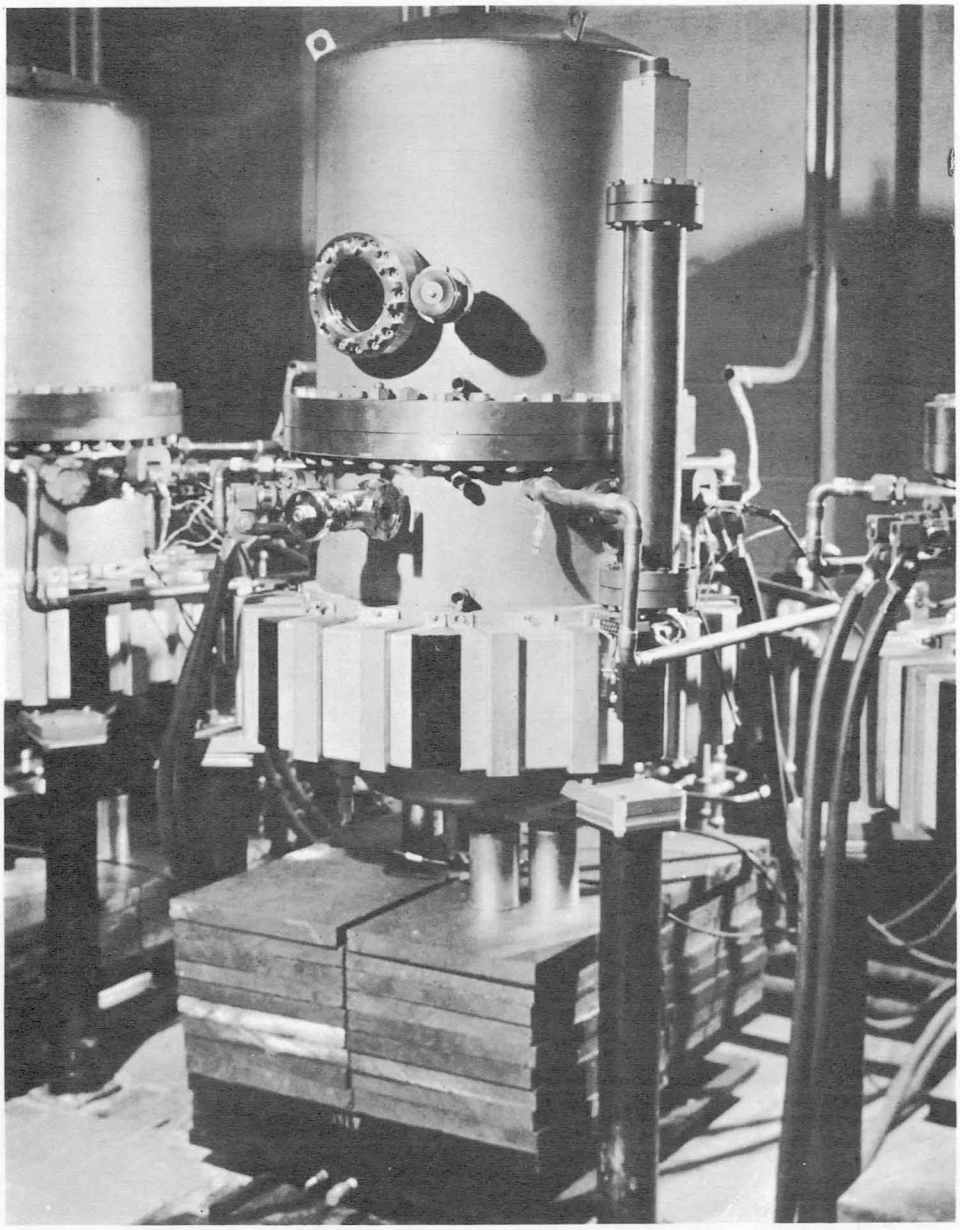


FIGURE 3 ULTRA-HIGH VACUUM CREEP CHAMBER

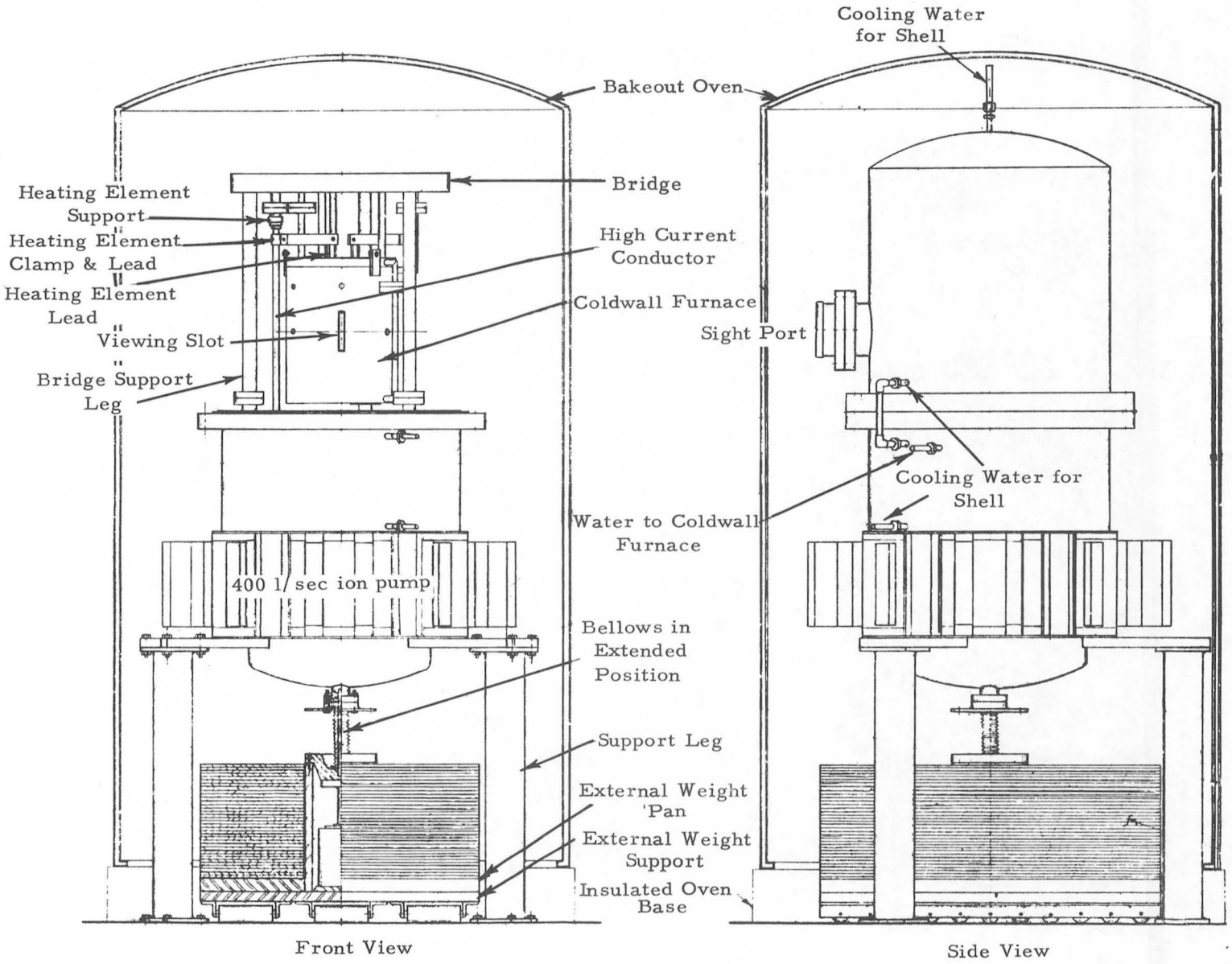


FIGURE 4 SCHEMATIC OF ULTRA HIGH VACUUM CREEP UNIT

The unit with the bell removed, to expose the bridge and furnace construction, is shown in Figure 5. The bridge is the uppermost support for the specimen with the load being carried by three tubular legs which also serve as a means of conveying cooling water to the furnace. By unbolting three flanges, the furnace coldwall and shield pack can be removed exposing the (8" x 2" dia.) heating element (Figure 6). With this element design and six tantalum radiation shields, the temperature gradients over the 2-inch gage length lie within the limits of:

1800°F (982°C)	0-5°F (0-3°C)
2000°F (1093°C)	0-7°F (0-4°C)
2200°F (1204°C)	0-9°F (0-5°C)

At 2600°F (1427°C), the observed gradient was approximately 10°F (5-6°C).

All fourteen systems are of ultra-high vacuum construction to maintain a pressure of $< 1 \times 10^{-8}$ torr during testing.

B. Temperature Control

The high-current, low-voltage power required to bring the furnace to operating temperature is obtained from a tapped 12 KVA step-down transformer and saturable-core reactor. The control signal for the saturable-core reactor is supplied by a magnetic amplifier and temperature controller at the main console.

The transistorized three-mode controller is capable of holding the furnace temperature cycle to ±1°F. Two tungsten-3% rhenium/tungsten-25% rhenium thermocouples located between the heating element and the first radiation shield serve to sense and control the furnace temperature. These thermocouples are wired in parallel at the controller to provide redundancy. A third thermocouple, also located between the heating element and the first radiation shield, is attached to a multipoint recorder for general monitoring purposes. A fourth tungsten-3% rhenium/tungsten-25% rhenium thermocouple is calibrated before use and is attached directly to the specimen. This special thermocouple is used during the initial setting of the furnace temperature. All four thermocouples are connected to a 150°F ± 1/4°F (52°C) reference junction oven.

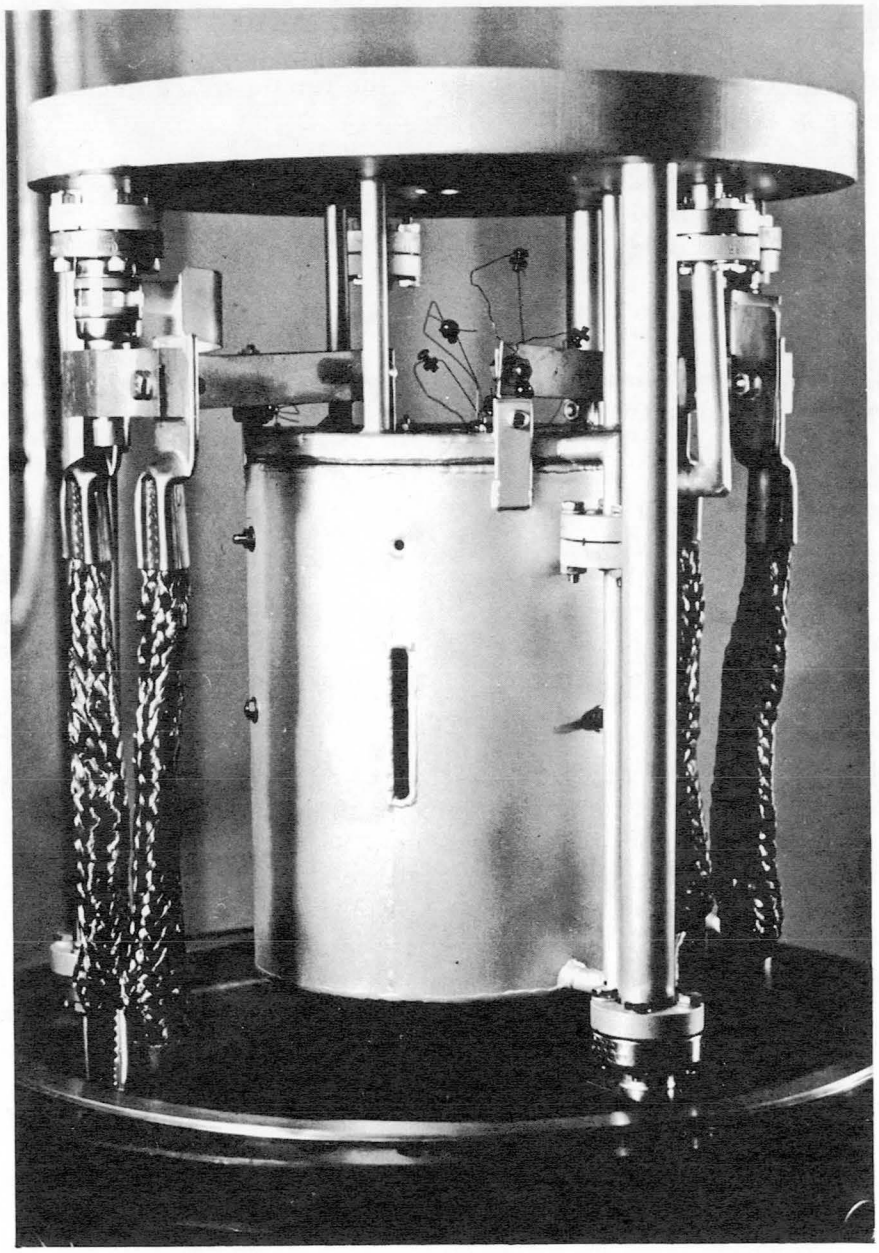


FIGURE 5 CREEP UNIT WITH BELL REMOVED SHOWING BRIDGE AND COLDWALL FURNACE

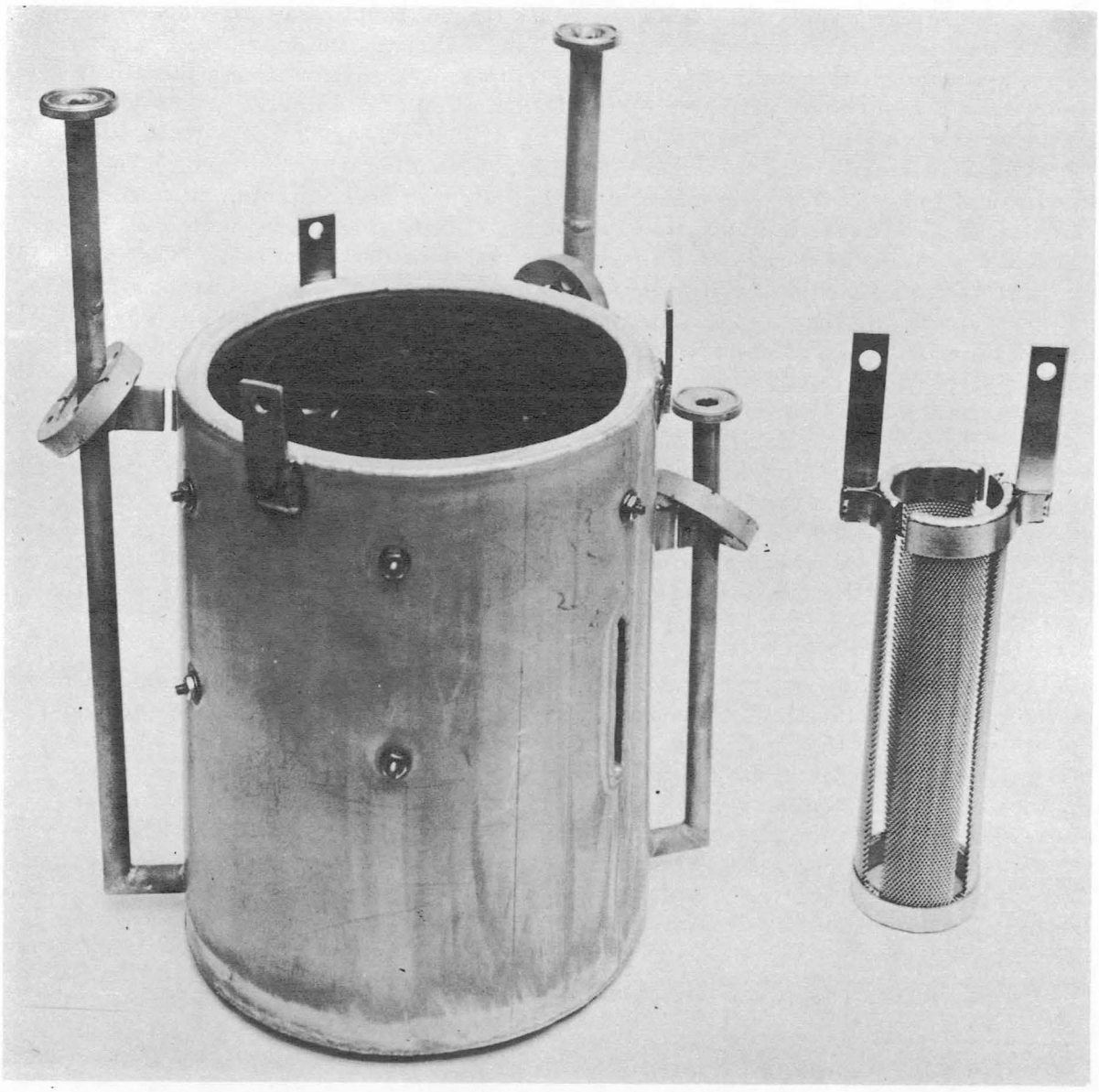


FIGURE 6 FURNACE COLDWALL AND TUNGSTEN MESH ELEMENT

Thermocouples of all types are subject to a time dependent change in EMF output under isothermal conditions. To compensate for these anticipated changes, the absolute temperature during test was maintained by an optical pyrometer. In practice the specimen was brought to the desired test temperature using the calibrated thermocouple attached to the specimen as a temperature standard. The use of this thermocouple was continued during the temperature stabilization period which lasted 50 to 100 hours. At this time a new reference was established using an optical pyrometer having the ability to detect a temperature difference of 1°F. In use the pyrometer was sighted on the specimen with the field of view being 0.050 inch diameter. Filters were introduced in front of the telescope lens until a reading was obtained on the instrument meter. The pyrometer was then sighted on a tungsten filament lamp contained inside the vacuum chamber. This filament was heated by DC current to obtain the same reading as observed with the specimen. The current passing through the filament was then measured to an accuracy of one part in 10,000 thus establishing an internal standard. Periodically the temperature of the specimen was measured at the same spot and based on the readings, corrective adjustments were made at the controller. The reference lamp in the vacuum chamber was also checked to determine whether clouding of the sight port was affecting the accuracy of the temperature readings. No evidence of sightport clouding was observed during this program. Maintenance of the temperature in this manner was based on the assumption that the emissivity of the standard lamp filament and the specimen do not change significantly with time (2). This assumption appears to be valid, for no significant change in input power to the furnace was observed during creep tests lasting more than 10,000 hours.

The use of an optical pyrometer to maintain isothermal conditions allows an assessment of the time dependent characteristics of the calibrated thermocouple attached to the specimen. Figure 7 shows the change in EMF output of tungsten-3% rhenium/tungsten-25% rhenium thermocouples as a function of time at various temperatures. These data reveal that a significant linear decrease occurs in the EMF output at all temperatures, and that the drift is not the same for all tungsten-rhenium thermocouples. Figure 7 also shows that stabilization of the EMF output does not occur even at times up to 12,000 hours. This was complemented by the observation that the EMF output of the control and recorder thermocouples continue to drift downward at about the same rate despite the fact that they have seen previous service.

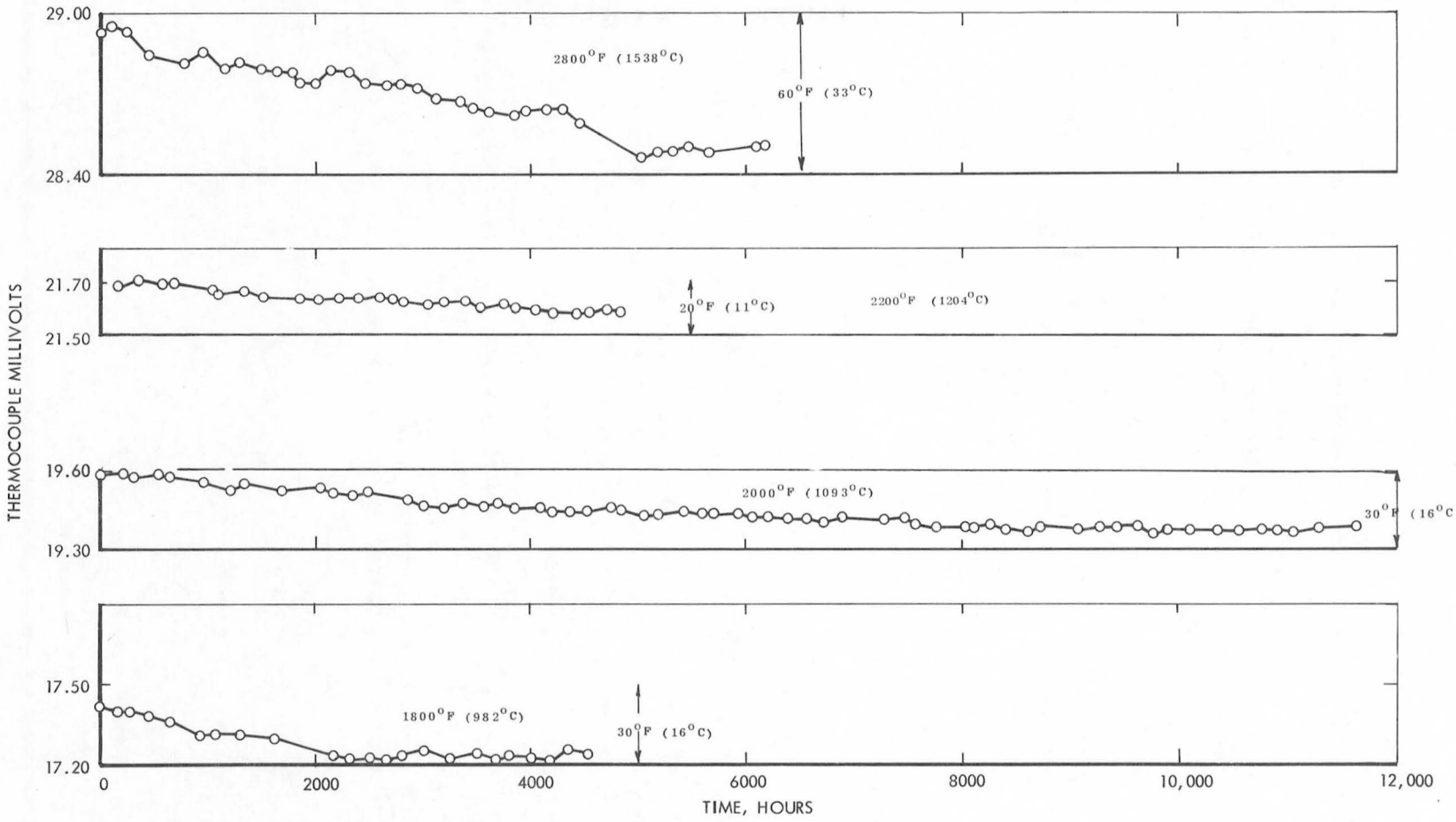


FIGURE 7 TIME DEPENDENT CHANGE IN EMF OUTPUT OF TUNGSTEN - 5% RHENIUM/TUNGSTEN - 25% RHENIUM THERMOCOUPLES AT VARIOUS TEMPERATURES.

C. Creep Measurement

The extensometer, Figure 8, used to measure creep consisted of two telescopes mounted one above the other with the lower telescope movable by means of a micrometer screw. The two images produced were optically superimposed and magnified 40X. A mercury lamp in combination with a condensing lens and mirror provided illumination of the field of view. At 2600°F (1427°C) and higher, where the specimen spectral emission overrides the external illumination, the mercury lamp was operated at twice its normal wattage to provide the additional intensity.

The fiducial marks used were horizontal lines 2.00 inches apart scribed on the specimen surface. By adjusting the micrometer screw, the images of the two lines could be superimposed and the distance between them read directly from the micrometer. The accuracy achieved was equal to the sensitivity of the equipment, i.e., 50 micro-inches.

D. Residual Gas Analysis

At least six of the vacuum creep units were equipped with a residual gas analyzer. These may be seen in Figures 1 and 3 as a large tublar member to the right of the removable bell. The instrument was a time-of-flight mass spectrometer schematically illustrated in Figure 9. The principle of operation is based on the property that the time for a given ion to traverse a spatial distance is proportional to the square root of the mass. Referring to Figure 9, the ions are generated by electron bombardment of the residual gas in the ionizing region. Batches of ions are accelerated into the flight tube 10,000 times each second. The termination of the flight is sensed by the electron multiplier at the right end of the tube and recorded as shown in Figure 10.

Monitoring of the residual gas composition during various tests, Table 1, shows that the bulk of the gas was composed of H_2O, CO-N_2, and A. The gas content as a function of holding time for a T-111 specimen (Table 1-B) tended to decrease in water vapor and increase in hydrocarbon. During heating to 3000°F (1649°C) an appreciable increase in hydrogen evolution occurred presumably as a result of specimen outgassing. In general the pattern of residual gas composition was not constant and depended on the specific unit and specimen being tested.

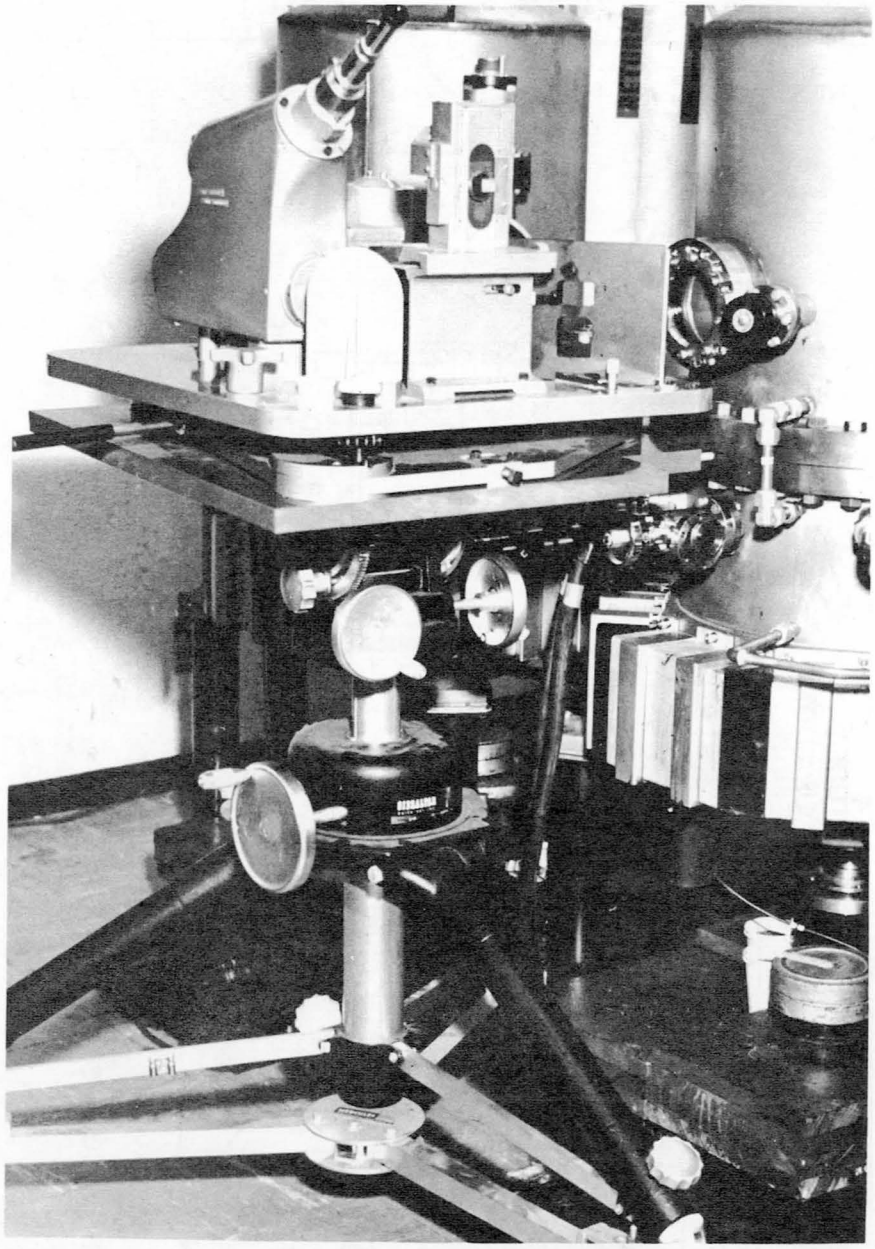


FIGURE 8 OPTICAL EXTENSOMETER

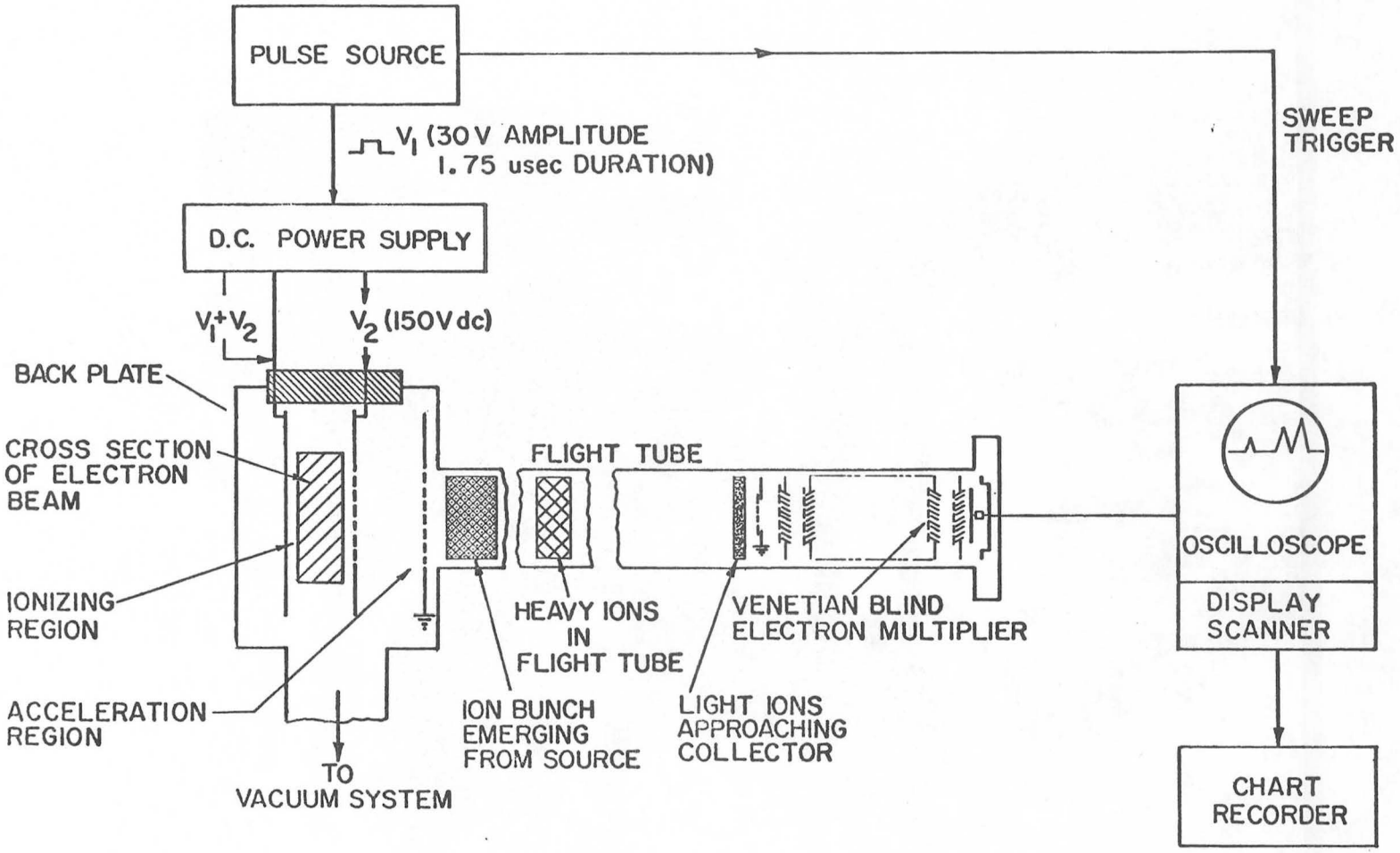


FIGURE 9 SCHEMATIC DIAGRAM OF RESIDUAL GAS ANALYZER

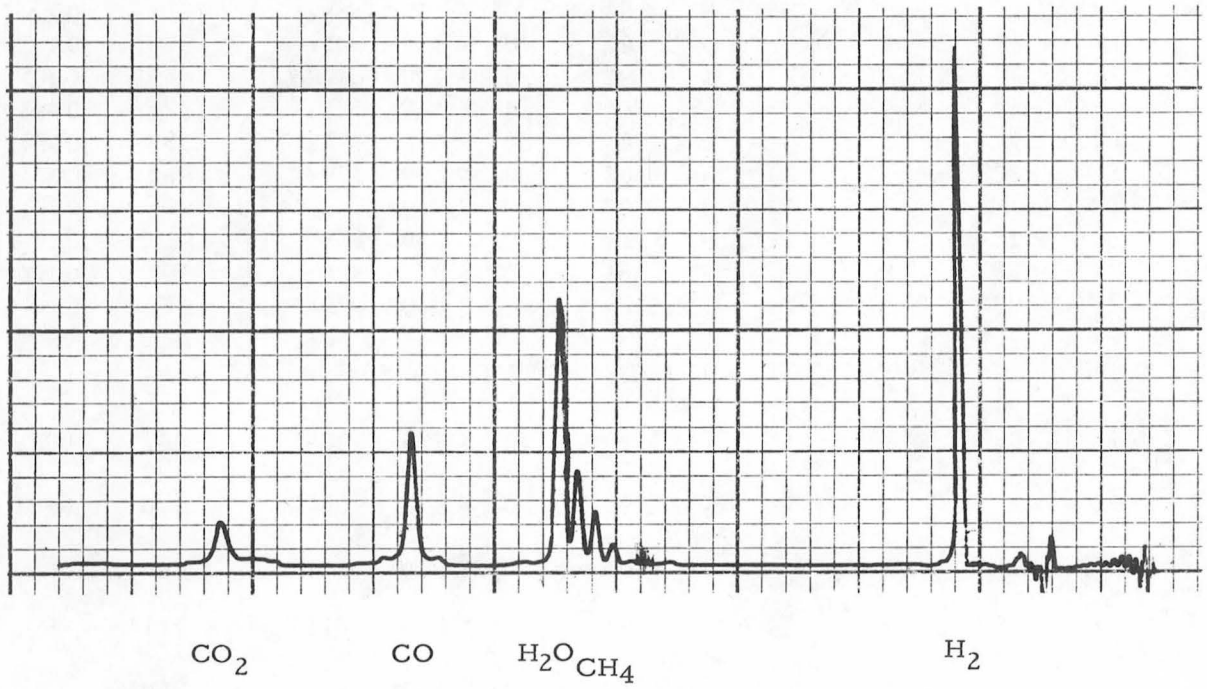


FIGURE 10 RECORD OF 10^{-8} TORR RESIDUAL GAS ANALYSIS

TABLE 1 - Residual Gas Analysis

A. Composition of Residual Gas in Vacuum Creep Chambers

Unit No.	Temperature °F	Temperature °C	Time Hours	Pressure Torr	Percent H_2	He	CH_4	CH_4-O	H_2O	CO-N_2	A	CO_2
2	2000	1093	1535	4.4×10^{-9}	24	2	-	6	28	22	11	6
3	2056	1124	1912	6.8×10^{-10}	13	-	-	2	31	27	-	27
6	2000	1093	1175	6.9×10^{-10}	40	-	-	3	17	31	-	9
8-1	1856	1013	3340	1.9×10^{-9}	16	1	1	7	18	34	14	9
8-2	3200	1760	520	6.6×10^{-9}	4	1	1	4	30	34	6	20

B. Analysis of Unbaked Unit T-111 Specimen

60	16	0.0	6.0×10^{-8}	2	<1	2	6	66	7	<1	17
400	204	0.8	7.0×10^{-7}	19	5	4	8	31	17	10	5
600	315	3.1	6.0×10^{-7}	17	6	3	7	27	19	14	6
1000	538	5.1	6.0×10^{-7}	20	6	3	5	17	29	14	5
1200	648	6.5	6.0×10^{-7}	20	6	2	6	22	23	13	6
1200	648	71.8	6.2×10^{-9}	9	3	0	7	34	30	8	9
2600	1427	73.8	3.7×10^{-9}	19	5	2	6	19	27	11	11
3000	1649	73.9	8.6×10^{-7}	16	5	2	6	16	31	14	10
3000	1649	74.9	4.8×10^{-7}	16	6	3	6	18	27	15	8
600	315	75.9	3.2×10^{-8}	15	8	2	6	24	26	14	4
2600	1427	76.5	2.0×10^{-8}	18	6	2	5	21	28	14	6
2600	1427	77.5	4.0×10^{-8}	17	6	4	6	22	26	12	6
2600	1427	96.5	2.4×10^{-8}	15	4	0	7	28	31	10	6
2600	1427	268.0	5.5×10^{-9}	4	0	0	0	14	65	7	9

E. System Reliability

Creep tests of 1,000 to 15,000 hours duration require that the equipment be capable of maintaining the desired vacuum and temperature throughout the test period. To accomplish this, each individual creep unit was protected such that a failure of any one was an independent event which did not affect the operation of the others.

To allow for failure of city power, a stand-by-diesel engine driving a 100 KVA, 480 volt, three-phase generator was used to supply auxiliary power within seven seconds after loss of city power. This system was checked weekly for proper operation.

Cooling water for the creep chambers was provided by a recirculating system with an external cooling tower and a circulating pump capable of providing 80 gallons of filtered water per minute. Redundant pumping and filtering were used with automatic change-over in the event of failure of either pump or filter. The recirculated water was chromate treated and make-up water was supplied from the city mains through a water softener and deionizer. A weekly check was maintained of the pH, chromate, chloride, and hardness content. Typical values were:

pH	- 8-9 ppm	Chloride	- 4-7 ppm
Chromate	- 2000 ppm	Hardness	- 4 ppm

The laboratory air conditioning was operated constantly to maintain a temperature of 70-73°F (21-23°C) and humidity was controlled to prevent rusting of the instruments. Despite periodic failures of city power, the system has been in operation for over 20,000 hours (2.3 years) without shutdown.

III TEST PROCEDURE

A. Specimens

Creep tests were conducted with two forms of material, sheet, and plate and the geometries of the test specimens are shown in Figures 11 and 12. As a general rule the orientations of the specimens with respect to the working direction of the material were as follows:

Material Form	Specimen Axis Parallel to:
Sheet	Rolling Direction
Plate	Primary Extruding Direction
Disc Forging	Radius of Disc

The first operation following machining was to locally polish the specimen surface in preparation for gage mark scribing. The specimen was then placed on a Kentron Hardness Tester and moved under the Knoop diamond indenter. The scribe marks produced by the diamond using 100-200 gram load were transverse to the specimen axis and 2.00 inches apart. The next operation was to attach the calibrated thermocouple to the specimen. For bar specimens, the thermocouple was wired to the gage section using molybdenum wire. With sheet specimens, two 0.020-inch holes were drilled in the specimen just outside the gage section. Thermocouple wires were passed through these holes and spot welded to the specimen surface.

B. Test Setup

Installation of the specimen and pumping the vacuum chamber followed a routine procedure. The specimen was first installed in the grips which were equipped with universal joints to provide for axial loading and specimen expansion during heat-up. Thermocouples were attached to lead wires and the load placed on the lower pullrod. In all cases ultra-high vacuum procedures were carefully followed to minimize contamination.

Initial pumping of the system was accomplished by four sorption pumps cooled with liquid nitrogen. After roughing, the ion pump was turned on and the complete system baked at 400°F for at least 8 hours.

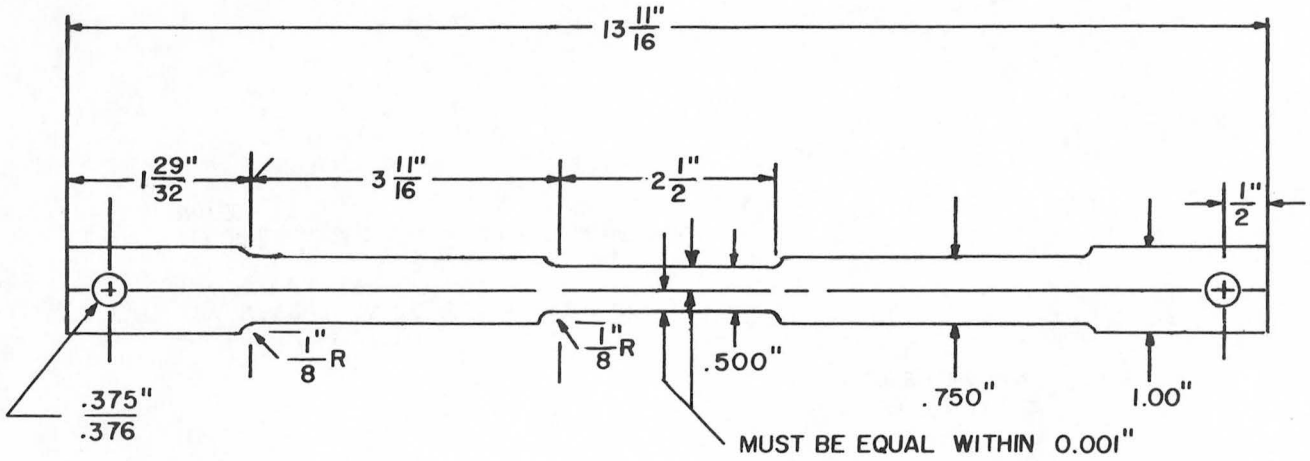
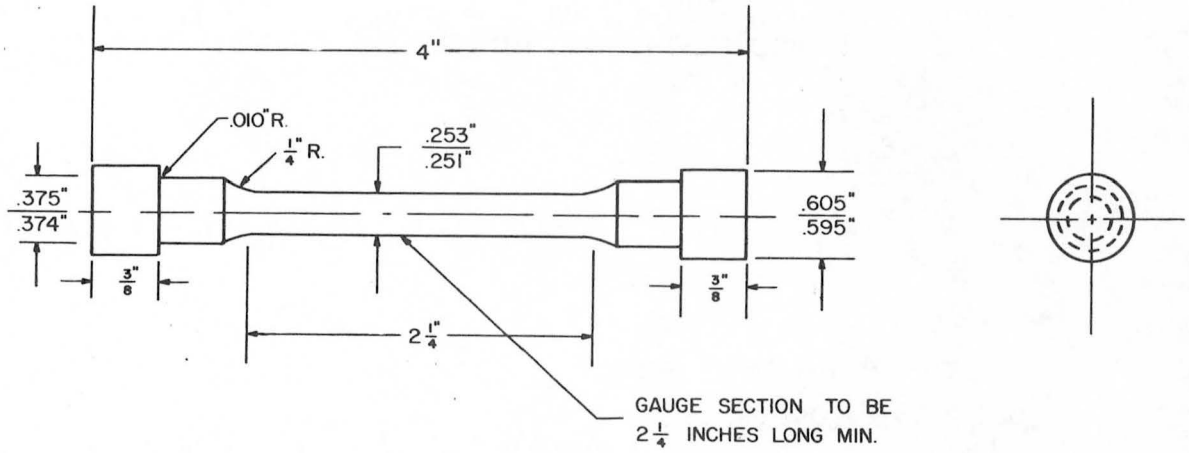


FIGURE 11 CREEP SPECIMEN USED FOR SHEET STOCK



NOTE: ANY TAPER IN GAUGE SECTION MUST BE TOWARDS CENTER

ALL TOLERANCES ± .010" UNLESS OTHERWISE NOTED

FIGURE 12 CREEP SPECIMEN USED FOR PLATE STOCK

C. Creep Testing

Following baking and cooling, temperature was manually raised to the desired value with the pressure, which increased during heating, being kept below 1×10^{-6} torr. Depending on the cleanliness of the system, the required time to reach temperature was about eight hours. When necessary, heat treatment was performed in situ.

Following heat treatment the temperature was allowed to decrease to at least 600°F (316°C) prior to reheating to the test temperature. Prior to application of the load, a 1-2 hour stabilization period at temperature was employed. For the first hour of test, creep readings were taken every few minutes. After this, extension measurements were made on a periodic basis depending on the particular creep rate involved. In addition to the creep measurements, data relative to the various test parameters were recorded (see Figure 13).

When a creep test was completed, the furnace was cooled to room temperature under load and the specimen removed for post-test examination. In selected tests, chemical analysis, hardness, and tensile strength determinations were made.

Date	10/13/66	10/14/66	10/17/66	10/18/66	10/19/66	10/20/66	10/21/66	10/24/66	10/25/66
Time	07:35	07:40	07:30	07:30	07:45	07:35	07:30	07:30	10:35
Press	3.5×10^{-9}	3.7×10^{-9}	4.2×10^{-9}	4.0×10^{-9}	3.2×10^{-9}	3.8×10^{-9}	3.1×10^{-9}	3.0×10^{-9}	4.0×10^{-9}
Volts	15.5+	15.6	15.6	15.6	15.6	15.6	15.6	15.6	15.6
Amps	378	378	378	378	378	378	378	380-	380-
Set Point	20+270	20+271	20+271	20+271	20+271	20+271	20+271	20+271	20+271
PB%	100	100	100	100	100	100	100	100	100
Reset	0	0	0	0	0	0	0	0	0
Rate	0.7	0.7	0.7	0.7	0.7	0.7	0.7	0.7	0.7
Recorder	25.4	25.4	25.4+	25.4	25.4	25.4	25.4	25.4	25.4-
Std. T.C.	25.787 25.760	—	—	—	25.776	—	—	—	—
R.T.	73.0	71.4	72.6	73.0	71.4	73.2	72.4	74.0	73.8
Water, Bell	0	0	0	0	0	0	0	0	0
Water, Cent.	2.5	2.5	2.5	2.5	2.5	2.4	2.4	2.4	2.5
Water, Right	2.1	2.1	2.0	2.1	2.1	2.0	2.0	2.0	2.0
35 ma 2½ Opt. Temp.	16+25	—	—	—	30	—	—	—	—
A Filters	—	—	—	—	—	—	—	—	—
Std. Cur.	—	—	—	—	—	—	—	—	—
Std. Filters	—	—	—	—	—	—	—	—	—
Length	2.01660	2.01660	2.01670	2.01670	2.01675	2.01680	2.01685	2.01705	2.01710
Exten.	.00385	.00385	.00395	.00395	.00400	.00405	.00410	.00430	.00435
Elapsed Time	568.4	592.5	664.3	688.3	712.6	736.4	760.3	832.3	859.4
	.192%	.192%	.198%	.198%	.200%	.202%	.205%	.215%	.218%

Unit # 3 **Temperature** 2600°F

Test # S-28 **Stress** 500 PSI

Material T-111 **Initial Length (no load)** 2.01275

T-111-D-1670
1Hr, 3000°F **Initial Length under Load** 2.01275

FIGURE 13 TYPICAL LOG OF RECORDED DATA

IV MATERIALS

A. Materials and Processing

The materials tested during this program consisted of the following alloys:

Columbium-Base	Molybdenum-Base	Tantalum-Base	Tungsten-Base
AS-30 (1)*	TZC (3)	T-111 (5)	Arc-Melted W (1)
Cb-132M (1)	TZM (3)	T-222 (1)	Vapor-Deposited W (1)
	Cb-Modified TZM(1)	ST-222 (1)	W-25% Re (1)
		Astar 811C (1)	Sylvania A (1)

Whenever possible detailed processing information was obtained for each material and these data are summarized in Appendix I. To facilitate locating the data for a particular material, an index is provided on the Appendix I title page. Tension test data for some of the materials tested are presented subsequently in subdivision E of this section.

TZC, a molybdenum-base alloy for turbine application, was fabricated by several methods. The first heat (M-80) was found to have low room temperature ductility presumably due to the method of processing which involved finishing a 2 inch x 4 inch sheet-bar by rolling at 2935°F (1585°C) using small reductions (approximately 4%) on each pass. Subsequently, Heat M-91 was prepared with the same chemistry, but a lower finishing temperature of 2372°F (1300°C) and larger reductions to provide room temperature ductility. A third lot of TZC plate (Heat 4345) was prepared by a second vendor. In this case a round extrusion was first upset then broad forged at 2400°F (1316°C) to the desired thickness. This method of processing also produced room temperature ductility in a conventional tension test.

* ( ) number of heats examined.

The TZM molybdenum-base alloy was obtained from two different sources. One lot of material designated Heat 7502 was purchased in the form of a disc forging approximately 11 inches diameter and 3/4 inch thick. A second lot of material (Heat KDTZM-1175) consisted of a section from a disc forging obtained from an AiResearch Corp. program. This sample was processed for improved creep resistance (3) through the development of a fine carbide dispersion. In order to produce this effect, it was necessary to work with a carbon level above 0.02%. Columbium-modified TZM was included as a relatively new alloy offering potential as a turbine material. Since disc forgings of this alloy were not available, it was tested in the form of a 5/8 inch diameter wrought bar. Commercial TZM bar 5/8 inch diameter (Heat 7463) was included in the studies to determine the influence of material form on the creep properties.

Tantalum-base alloys T-111 and T-222 were evaluated in sheet form. The T-111 which represents one of the most promising materials for tubing application was extensively tested using 5 different heats.

The tungsten and tungsten-base alloys were tested in sheet form. Pure tungsten and tungsten-25% rhenium were prepared by arc-melting while Sylvania A, a tungsten-hafnium-carbon alloy, was processed by powder methods. It was found during testing that cracking of the Sylvania A specimens frequently occurred outside of the gage section after 40-100 hours of creep, but no metallurgical causes could be found to account for this brittle behavior. The vapor-deposited tungsten was prepared by General Atomics Corporation (4) and machined into bar specimens from a longitudinal strip cut from a vapor-deposited tube. *

B. Chemistry of Materials

The detailed chemical analyses of the materials used in this program are given in Appendix II and a condensed summary is presented in Table 2.

Evaluating the chemical data requires recognition of the inherent variability in quantitative analysis of refractory materials. "Round Robin" analyses conducted by the Materials Advisory Board (5) serves as an illustration. Samples from the same lot of molybdenum-base TZM were analyzed by various cooperating laboratories and the following results were obtained:

* These were a 1/8" diameter specimen of special design for adaptation to available grips.

TABLE 2

Chemical Composition of Alloys Being Evaluated in Creep Program (Weight %) (1)

Material	W	Re	Cb	Mo	Ta	Hf	C	Ti	Zr	ppm N_2	ppm O_2	ppm H_2	
AS-30 (Heat C5)	21.0		Bal				.09		1.04	100	60	15	
Cb-132M(Heat KC1454)	15.6		Bal	4.72	19.7		.16		2.10	24	4	4	
TZC (Heat M-80)				Bal.			.127	1.02	.17	18	41	10	
(Heat M-91)				Bal.			.113	1.17	.27	34	37	10	
(Heat 4345)				Bal.			.075	1.19	.16	9	19	2	
TZM (Heat 7502)				Bal.			.010	.44	.10	100	20	7	
(Heat 7463)				Bal.			.016	.48	.08	1	2	1	(2)
(Heat KDTZM-1175)				Bal.			.035	.61	.12	31	34	9	
Cb Modified - (Heat 7503)			1.00	Bal.			.015	.48	.095	30	39	4	
T-111 (Heat 70616)	8.50				Bal.	2.30	.0044			20	55	6	
(Heat D-1670)	7.9				Bal.	2.17	.001			20	72	5	(2)
(Heat D-1102)	7.9				Bal.	2.28	.0034			34	20	3	(2)
(Heat 65079)	8.70				Bal.	2.30	.003			50	130	4	
(Heat MCN 02A065)	8.60				Bal.	1.95	.004			20	100	3	(2)
T-222 (Heat Al-Ta-43)	9.57				Bal.	2.93	.012			26	35	11	
(Heat Ta-43-SF2)	8.77				Bal.	2.53	.012			30	38	7	
Astar 811C	8.0	1.0			Bal.	0.7	.250						(3)
Tungsten (Arc-Melted) (Heat KC1357)	Bal.						.0058			16	9	3	

TABLE 2 (Continued)

Material	W	Re	Cb	Mo	Ta	Hf	C	Ti	Zr	ppm N_2	ppm O_2	ppm H_2
Tungsten (Vapor Deposited)	Bal.						.0012			15	12-14	1-3 (2)
Tungsten -25% Rhenium (Arc-Melted) (Heat 35-75002)	Bal.	24.88					.0070			7	61	13
Sylvania A	Bal.					.52	.0300			17	20	3

(1) - TRW Analysis
(2) - Vendor Analysis
(3) - Nominal Composition

1. Cooperating laboratories, using their own particular methods for analysis could duplicate their own results. However, comparison of results between laboratories showed considerable variation.

2. When all cooperating laboratories used the same method, the variation between laboratories was reduced.

A typical example of the scatter experienced in the MAB Program in analyzing molybdenum-base TZM is shown with 95% confidence limits:

Carbon	-	0.029 ± 0.017%
Zirconium	-	0.105 ± 0.075%
Nitrogen	-	20 ± 8 ppm

It follows that considerable judgement must be exercised in comparing chemical analyses of refractory alloys.

Post-test analyses of selected specimens were undertaken to determine the effect of both heat treatment and vacuum environment on the chemistry and these results are included in Appendix II. To facilitate evaluation of the results, the pertinent data have been summarized in Table 3. The distribution of the changes in the carbon content suggest variation of analytical results rather than a true change of composition. This is substantiated by the observation that the first five carbon values, representing the greatest change, are associated with a carbon content of approximately 0.1%. The remaining values, which show much less change, are associated with a carbon content of approximately 0.01%. The distribution of the changes in both the hydrogen and oxygen contents indicate a decrease due to heating in the vacuum environment. Nitrogen, like carbon, reflects analysis variability.

The results of these tests indicate that heating refractory metals in a vacuum environment of $< 1 \times 10^{-8}$ torr for 16,000 hours does not cause interstitial contamination.

TABLE 3

Chemical Composition Changes Due to Testing

Material	Heat No.	Unit No.	Test No.	Time Hours	Test Temp. °F	Test Temp. °C	Change in Composition, ppm C	H_2	O_2	H_2
AS-30	C5	11	B-6	1,193	2000	1093	-200	-6	-20	-83
Cb-132M	KC1454	10	B-13	568	2056	1125	-100	-2	- 1	- 4
TZC	M-80	10	B-5, A, B	1,985	2000	1093	+700	-5	-18	+ 6
TZC	M-80	2	B-9	16,002	2000	1093	-300	-9	-18	- 8
TZC	M-80	13	B-12	14,239	2056	1125	+300	-8	-14	-11
TZM	7502	6	B-3	10,048	2000	1093	+ 20	0	+11	+30
T-111	70616	7	S-16	1,675	2600	1426	+ 6	+4	-45	+20
T-111	70616	7	S-19	4,870	2200	1204	- 24	-3	-27	-14
W	KC1357	7	S-5	32	3200	1760	- 25	+2	+ 1	- 6
W	KC1357	5	S-9	3,886	3200	1760	- 18	0	+28	-10
W-25%Re	3.5-75002	3	S-4	97	3200	1760	- 10	-8	-51	+ 3
W-25%Re	3.5-75002	4	S-8	1,306	3200	1760	+ 10	-2	-22	-12

C. Metallography of Materials

The materials used in the refractory alloy creep program were metallographically examined in the as-received condition and after applying the heat treatment used prior to creep testing. These treatments are summarized in Table 4.

1. AS-30, Heat C5

The microstructure of the as-received AS-30 alloy, shown in Figure 14 is representative of the condition after rolling at 2100°F (1149°C) without further heat treatment. Since uniform cross-rolling was used in processing, the structures of edges parallel and perpendicular to rolling were identical.

2. Cb-132M, Heat KC1454

Figure 15 shows the as-received Cb-132M alloy. The microstructure is representative of the condition after rolling at 2400°F (1315°C) without further heat treatment. Figure 16 shows the microstructure after annealing at 3092°F (1700°C) for one hour. Only partial recrystallization occurred at this heat treatment.

3. TZC, Heat M-80

Figure 17 illustrates the structure of the TZC, Heat M-80, material in the as-received condition. Annealing at 3092°F (1700°C) for one hour resulted in only partial recrystallization, Figure 18.

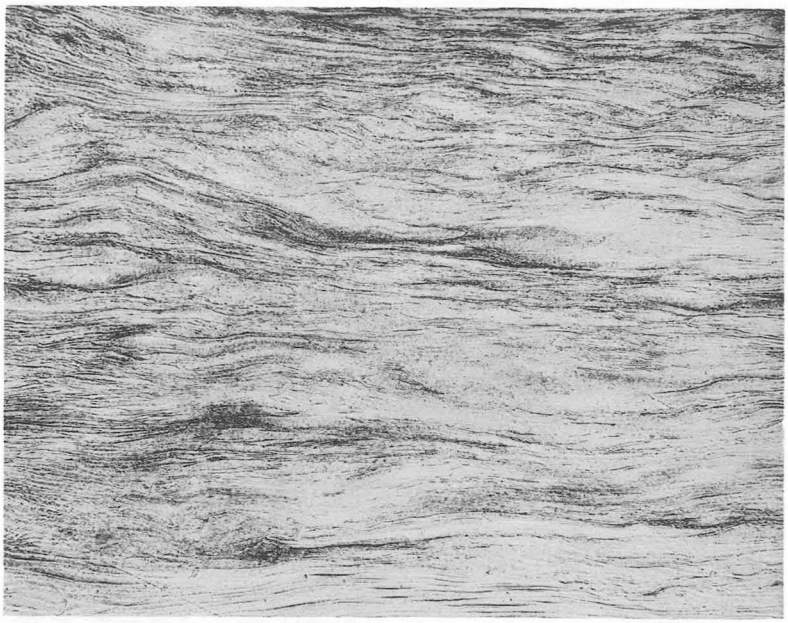
4. TZC, Heat M-91

The as-received microstructure of the TZC, Heat M-91, material finish rolled at 2372°F (1300°C) is shown in Figure 19. The two cross sections of the plate exhibited the same structure due to the uniform cross-rolling that was employed. Annealing at 3092°F (1700°C) for one hour, Figure 20, caused complete recrystallization. Subsequent aging for 5 hours at 2400°F (1315°C) produced no apparent change in the microstructure as observed at 100X. However, a comparison of the aged and unaged structures at 6000X shows that aging produced a slight increase in the size of the precipitate, Figure 21. A pretest stress relief treatment of 2400 or 2500°F (1315 or 1371°C) did not significantly change the appearance of the as-received material.

TABLE 4

Heat Treatments Applied Prior to Creep Testing

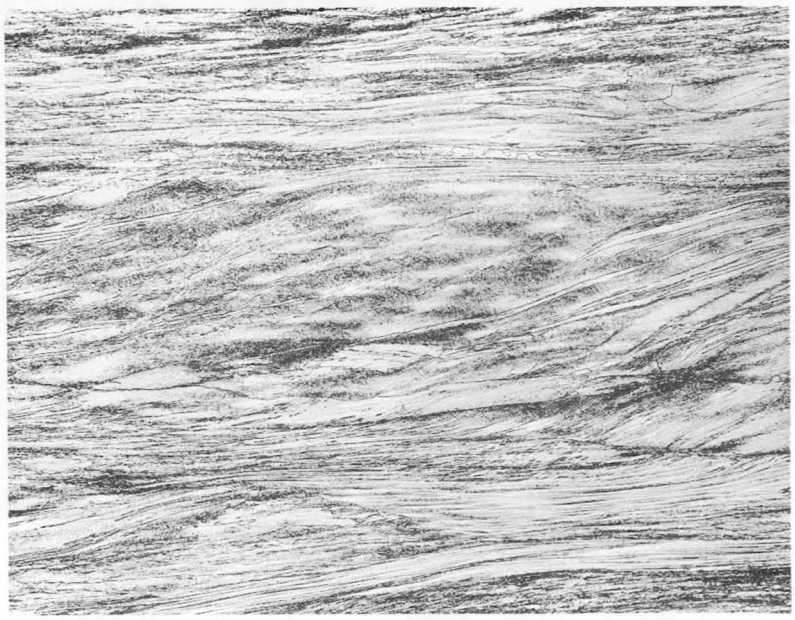
Material	Heat No.	Temperature °F	Temperature °C	Time Hours
AS-30	C5	None	-	-
Cb-132M	KC1454	3092	1700	1
TZC	M-80	None	-	-
	M-80	3092	1700	1
	M-91	3092	1700	1
	M-91	3092 + 2400	1700 + 1315	1 + 5
	M-91	2500	1371	1
	M-91	2400	1315	1
	4345	2500	1371	1
TZM	7502	None	-	-
	7502	2850	1566	1
	KDTZM-1175	None	-	-
	7463	None	-	-
	Cb-Modified	None	-	-
	4305-4	None	-	-
T-111	70616	2600	1427	1
	70616	3000	1649	1
	65079	3000	1649	1
	D-1670	3000	1649	1
	D-1102	3000	1649	1
	MCN02A065	3000	1649	1
T-222	Al-TA-43	2800	1538	1
	Al-TA-43	3000	1649	1
Astar 811C		3600	1982	1/2
Tungsten	KC1357	3200	1760	1
	KC1357	2800	1538	1
	Vapor Deposited	3200	1760	1
	Vapor Deposited	2800	1538	1
W-25%Re	3.5-75002	3200	1760	1
Sylvania A		3200	1760	1



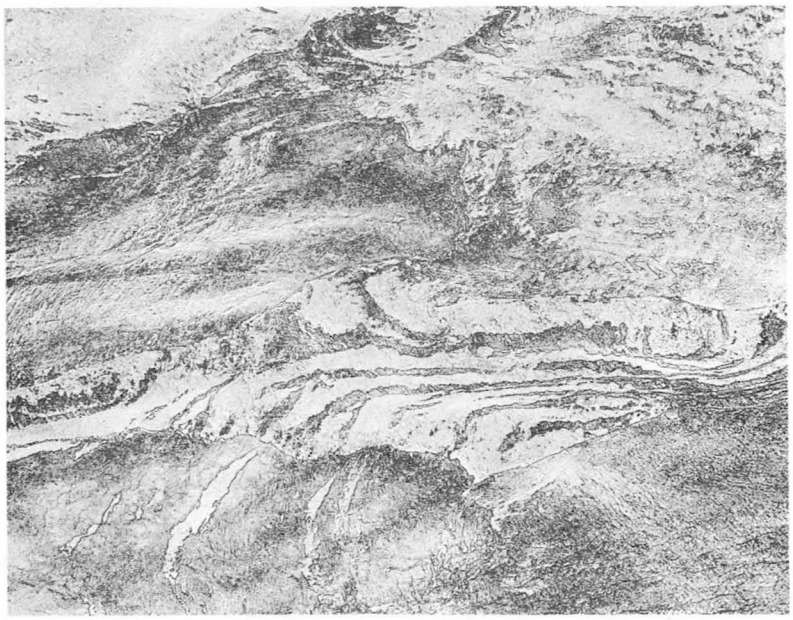
EDGE OF PLATE

SURFACE OF PLATE

FIGURE 14 MICROSTRUCTURE OF AS-30 PLATE (HEAT C-5) IN AS-RECEIVED CONDITION. ETCHANT: 15% HF, 15% H_2SO_4, 8% HNO_3, 62% H_2O. 100 X

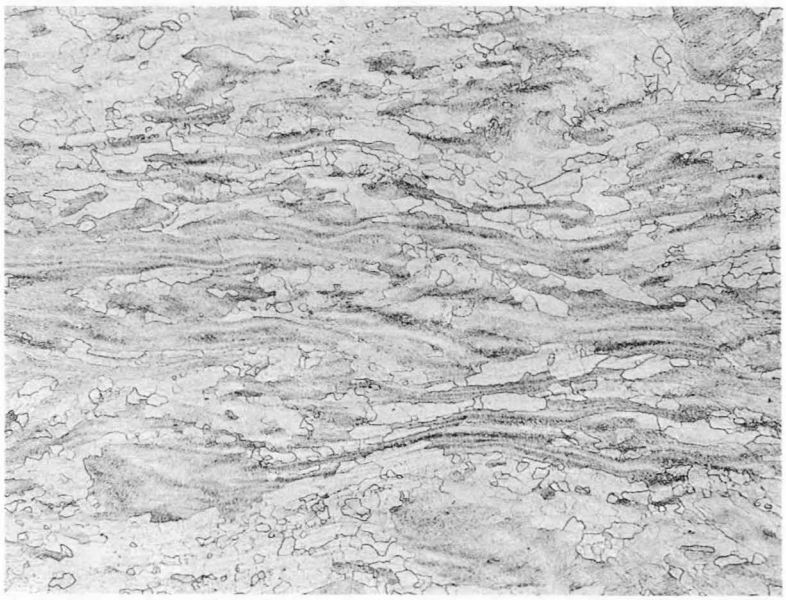


EDGE OF PLATE



SURFACE OF PLATE

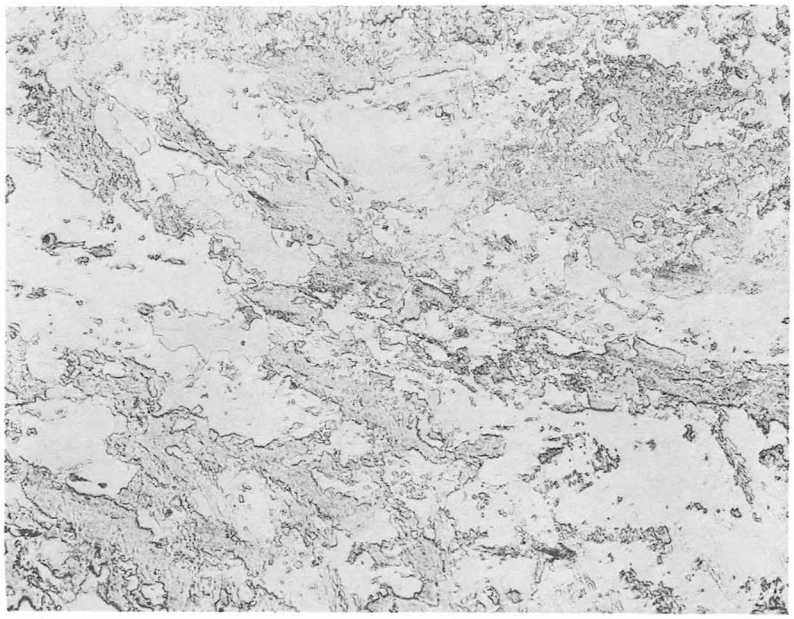
FIGURE 15 MICROSTRUCTURE OF Cb-132M PLATE (HEAT KC1454) IN AS-RECEIVED CONDITION.
ETCHANT: 15% HF, 15% H_2SO_4, 8% HNO_3, 62% H_2O. 100X



EDGE OF PLATE

FIGURE 16 MICROSTRUCTURE OF Cb-132M PLATE (HEAT KC 1454) AFTER ANNEALING AT 3092°F (1700°C), 1 HOUR ETCHANT: 15% HF, 15% H_2SO_4, 8% HNO_3, 62% H_2O. 100 X

EDGE OF PLATE

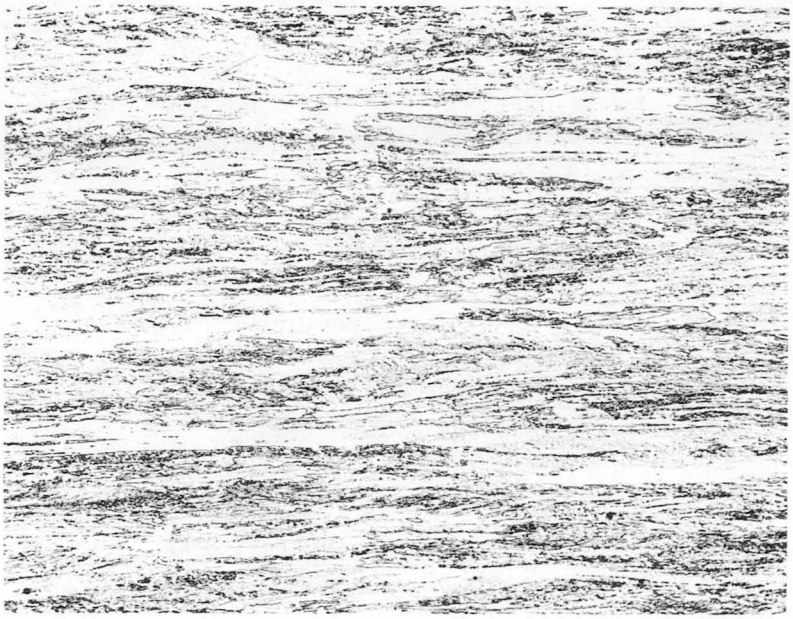


SURFACE OF PLATE

FIGURE 17 MICROSTRUCTURE OF TZC PLATE (HEAT M-80) IN AS-RECEIVED CONDITION. ETCHANT: 15% HF, 15% H_2SO_4, 8% HNO_3, 62% H_2O. 100 X

EDGE OF PLATE

FIGURE 18 MICROSTRUCTURE OF TZC PLATE (HEAT M-80) AFTER ANNEALING 3092 F (1700 C), ONE HOUR.
ETCHANT: 15% HF, 15% H_2SO_4, 8% HNO_3, 62% H_2O. 100X



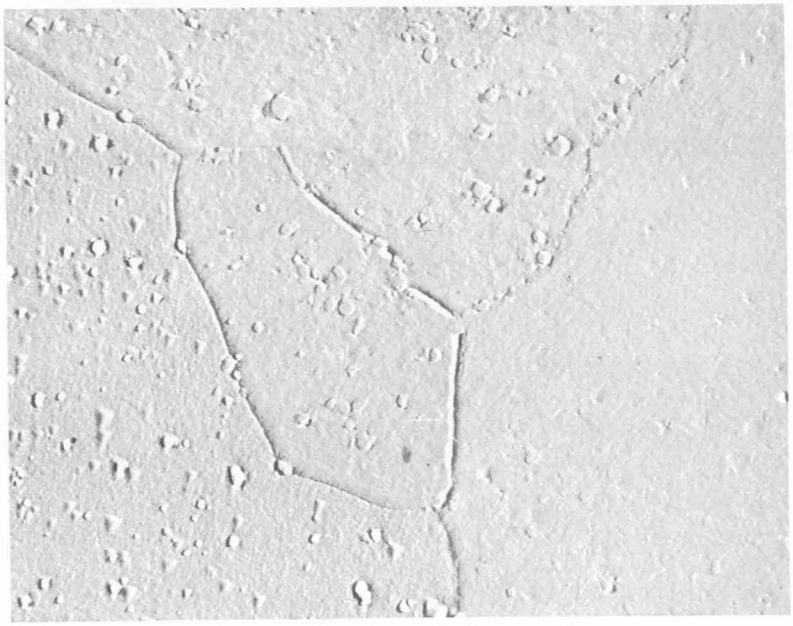
EDGE OF PLATE

SURFACE OF PLATE

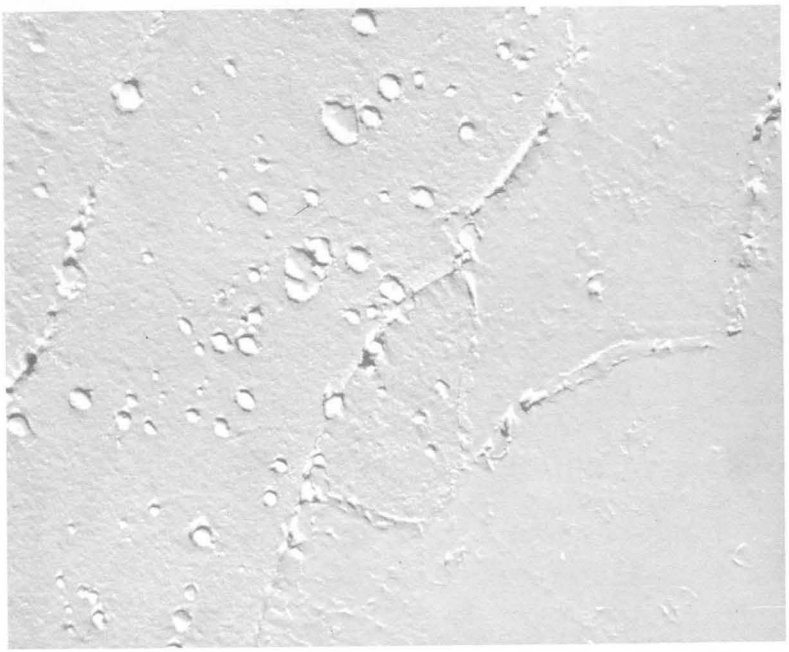
FIGURE 19 MICROSTRUCTURE OF TZC PLATE (HEAT M-91) IN AS-RECEIVED CONDITION. ETCHANT: 15% HF, 15% H_2SO_4, 8% HNO_3, 62% H_2O. 100X

EDGE OF PLATE

FIGURE 20 MICROSTRUCTURE OF TZC PLATE (HEAT M-91) AFTER ANNEALING 3092°F (1700°C), ONE HOUR.
ETCHANT: 15% HF, 15% H_2SO_4, 8% HNO_3, 62% H_2O. 100X



ANNEALED UNAGED



AGED 2400°F (1315°C) 5 HOURS

FIGURE 21 CROSS SECTION MICROSTRUCTURE TZC PLATE (HEAT M-91) ANNEALED 3092°F (1700°C), ONE HOUR. ETCHANT: 15% HF, 15% H_2SO_4, 8% HNO_3, 62% H_2O. 6000X

5. TZC Heat 4345

Figures 22 and 23 show the microstructure of the TZC, Heat 4345, material in the as-received condition. Both edges as well as the face of the plate are shown because of the variation of the microstructure. Stress relieving at 2500°F (1371°C) prior to testing, produced no change in the microstructure.

6. TZM Heat 7502

TZM Heat 7502 was supplied in the form of a pancake forged disc approximately 11 inches in diameter and 3/4 inch thick. By virtue of the processing, the macrostructure was not uniform throughout the cross section as shown by Figure 24. The 100X microstructures as a function of position in the disc are shown in Figure 25. Even greater differences could be observed if the areas examined were located near the top or bottom surfaces of the disc. Figure 26 shows the variation of microstructure between surfaces parallel and perpendicular to the radius while Figure 27 illustrates the microstructure of the disc face. This material was tested in both the stress relieved 2200°F (1204°C) and recrystallized 2850°F (1566°C) conditions. Figure 28 indicates that the flow lines still persist despite the one hour treatment at 2850°F (1566°C). Figure 29 illustrates the microstructure at two points along the radius and the attending grain size difference. These results indicate that the microstructure of this particular heat of TZM cannot be simply defined because of the method of producing the stock. Since the orientation of the creep specimen places the gage section at mid-radius of the disc, a microstructural variation along the two inch gage length existed.

7. TZM Heat KDTZM-1175

The TZM material representing Heat KDTZM-1175 was a section of a pancake forged disc stress relieved at 2300°F (1260°C). Creep tests were performed without further heat treatments. Figure 30 shows the microstructure in three directions. Because of the limited amount of material received, a detailed examination of the microstructure throughout the disc was not made, however, due to the nature of the processing, the upset forging would probably exhibit the same type variations observed with Heat 7502. A detailed description of the processing of this material can be found in Reference 3 covering a molybdenum forging program conducted in supplort of the SPUR-SNAP 50 program.

EDGE OF PLATE PERPENDICULAR TO EXTRUSION DIRECTION

FACE OF PLATE

FIGURE 22 MICROSTRUCTURE TZC PLATE (HEAT 4345) IN AS-RECEIVED CONDITION. ETCHANT: MURAKAMI'S. 100X

EDGE OF PLATE PARALLEL TO EXTRUSION DIRECTION

FIGURE 23 MICROSTRUCTURE TZC PLATE (HEAT 4345) IN AS-RECEIVED CONDITION
ETCHANT: MURAKAMI'S. 100X

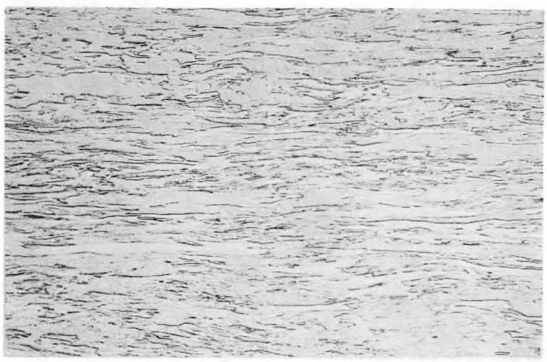
CENTER OF FORGING

EDGE OF FORGING

FIGURE 24 PHOTOGRAPH OF MACROETCHED SECTION OF AS-RECEIVED TZM PANCAKE-FORGED DISC (HEAT 7502), SHOWING FLOW LINES DUE TO FORGING. ETCHANT: MURAKAMI'S. 1-1/2X

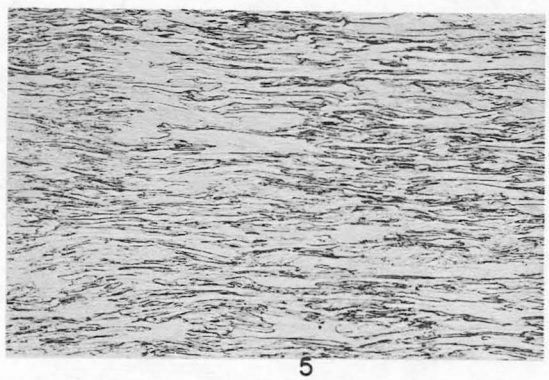
1

2



3

4



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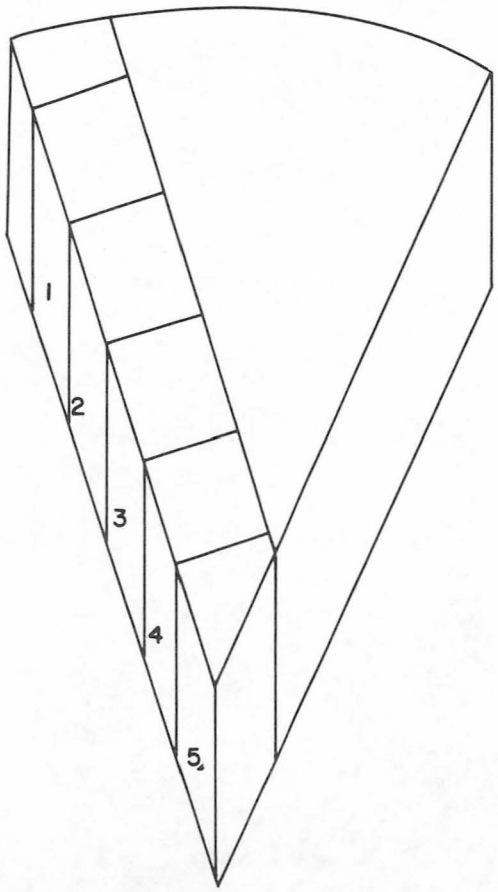
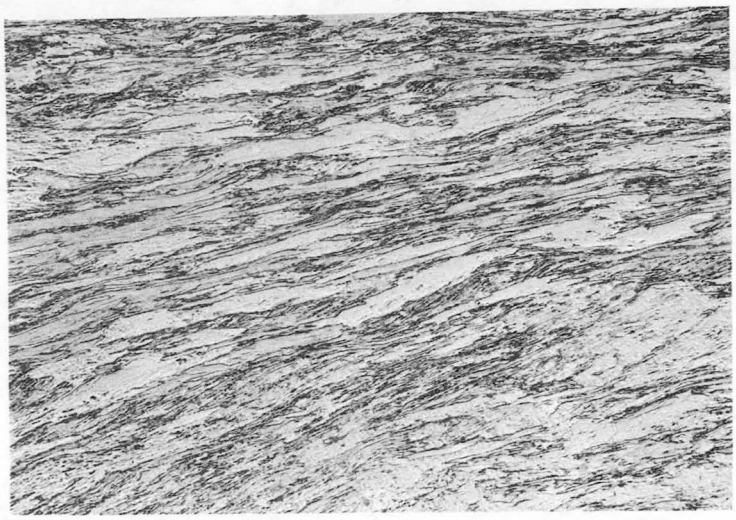
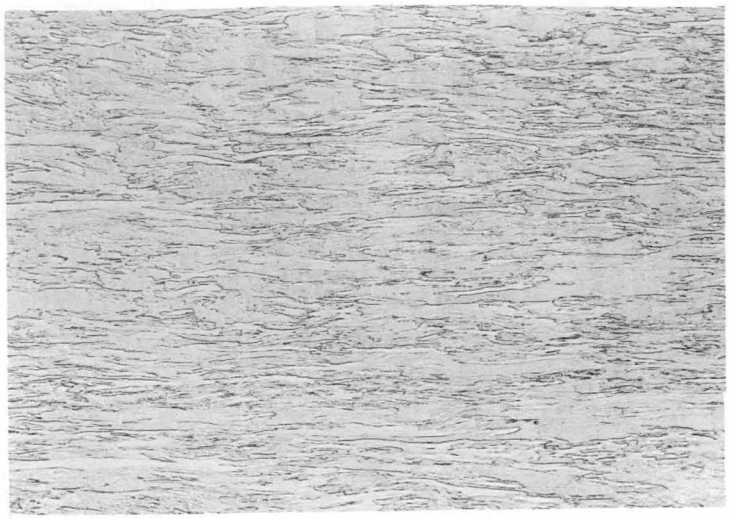


MICROSECTIONS
PERPENDICULAR TO RADIAL DIRECTION

FIGURE 25 PHOTOMICROGRAPHS SHOWING ETCHED SECTIONS OF TZM PANCAKE-FORGED DISC (HEAT 7502) IN AS-RECEIVED CONDITION. ETCHANT: MURAKAMI'S. 100X



EDGE COINCIDENT WITH RADIAL DIRECTION



EDGE PERPENDICULAR TO RADIAL DIRECTION

FIGURE 26 MICROSTRUCTURE OF AS-RECEIVED TZM PANCAKE FORGED DISC (HEAT 7502) AS A FUNCTION OF THE ORIENTATION OF THE FORGING. ETCHANT: MURAKAMI'S. 100X

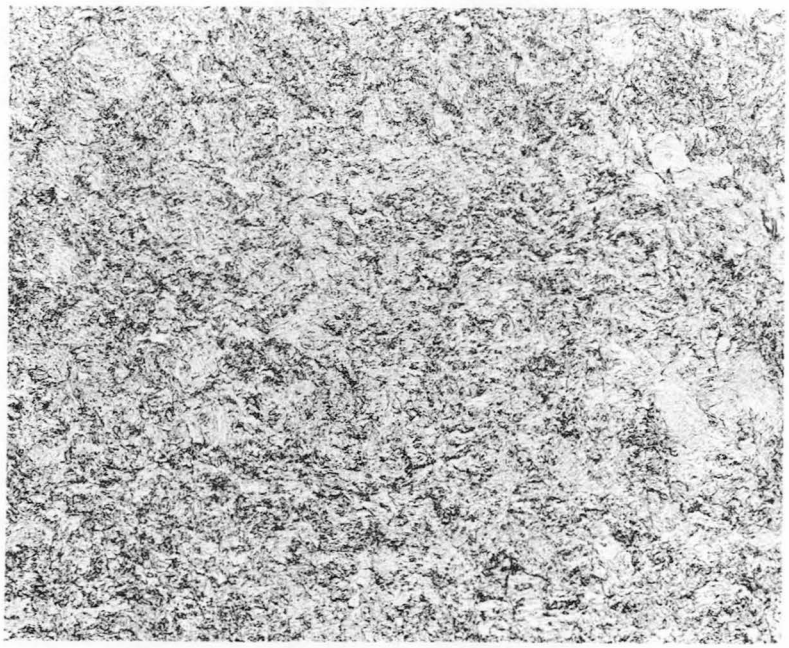
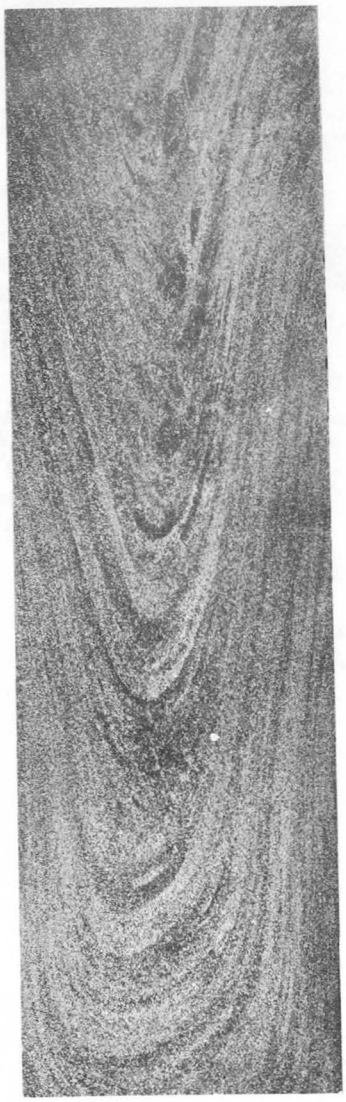


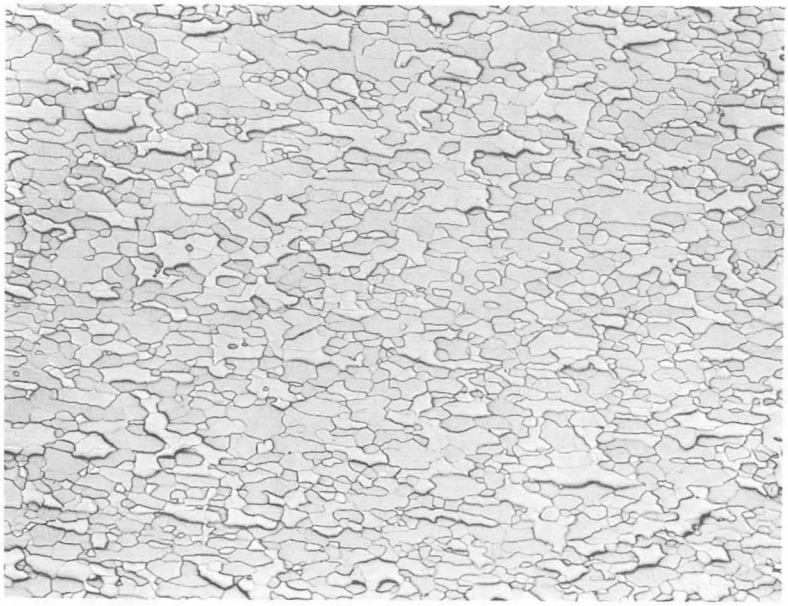
FIGURE 27 MICROSTRUCTURE OF FACE OF TZM PANCAKE-FORGED DISC (HEAT 7502) IN AS-RECEIVED CONDITION. ETCHANT: MURAKAMI'S. 100X

CENTER OF FORGING

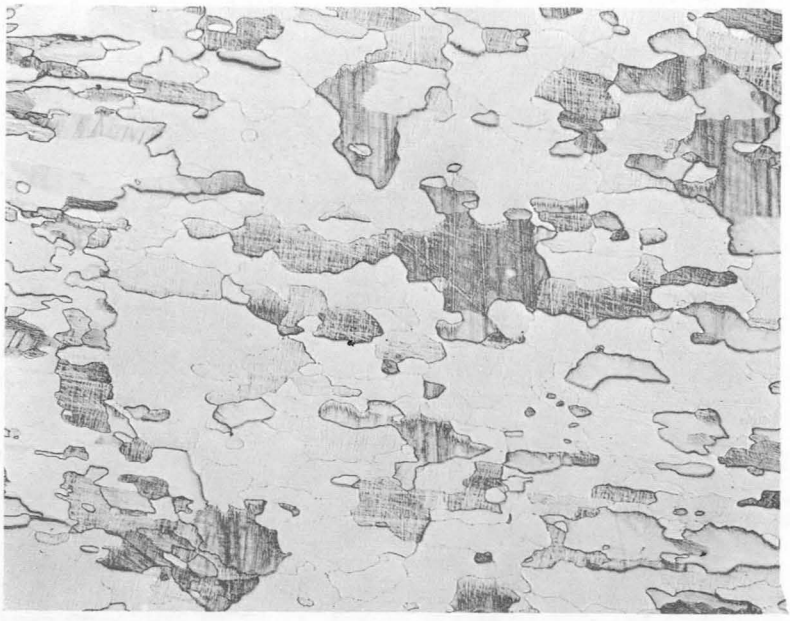


EDGE OF FORGING

FIGURE 28 PHOTOGRAPH OF MACROETCHED SECTION OF TZM PANCAKE-FORGED DISC (HEAT 7502) AFTER RECRYSTALLIZATION AT 2850°F (1566°C), ONE HOUR. ETCHANT: MURAKAMI'S. 1-1/2X



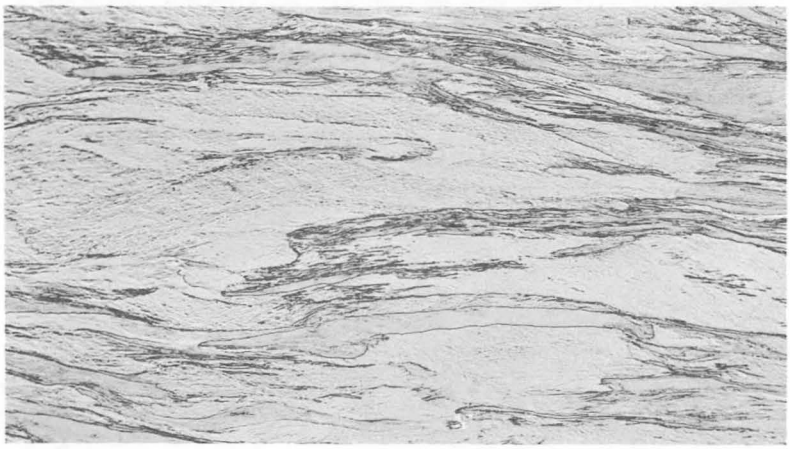
EDGE OF CROSS SECTION



CENTER OF CROSS SECTION

FIGURE 29 PHOTOMICROGRAPHS OF TZM PANCAKE-FORGED DISC (HEAT 7502) AFTER RECRYSTALLIZING ONE HOUR AT 2850°F (1588°C). ETCHANT: MURAKAMI'S. 100X

SURFACE



EDGE PARALLEL TO RADIAL DIRECTION

EDGE PERPENDICULAR TO RADIAL DIRECTION

FIGURE 30 MICROSTRUCTURES OF AS-RECEIVED TZM PANCAKE-FORGED DISC (HEAT KDT ZM 1175) AS A FUNCTION OF ORIENTATION IN THE FORGING. ETCHANT: 15% HF, 15% H_2SO_4, 8% HNO_3, 62% H_2O. 100X

8. TZM Heat 7463 (Commerical Bar)

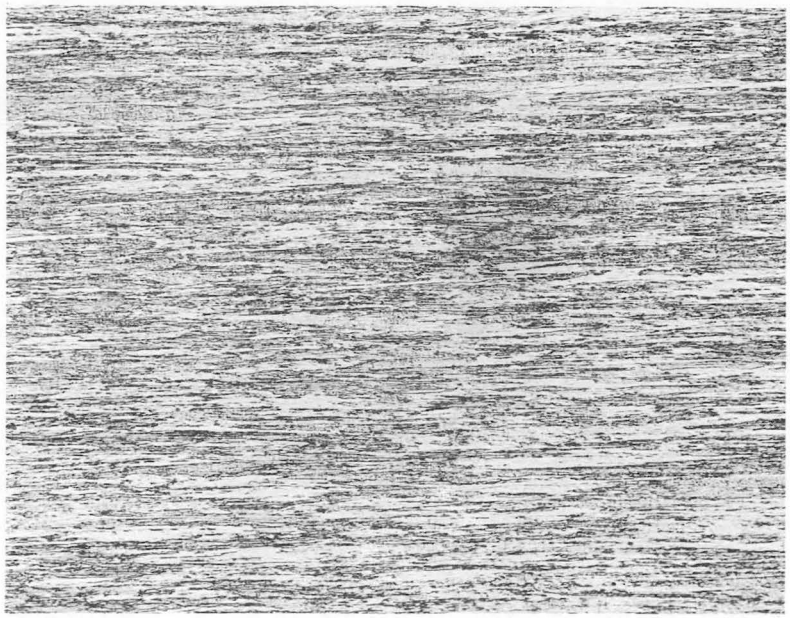
Figure 31 presents the longitudinal and transverse microstructures of commercial TZM Heat 7463. Because the processing method combines extrusion and swaging to the final diameter of 5/8 inch, the microstructure of the cross section is very uniform. At 500X, Figure 32, the improved resolution shows more clearly the nature of the heavily worked microstructure.

9. Columbium-Modified TZM Heat 4305-4

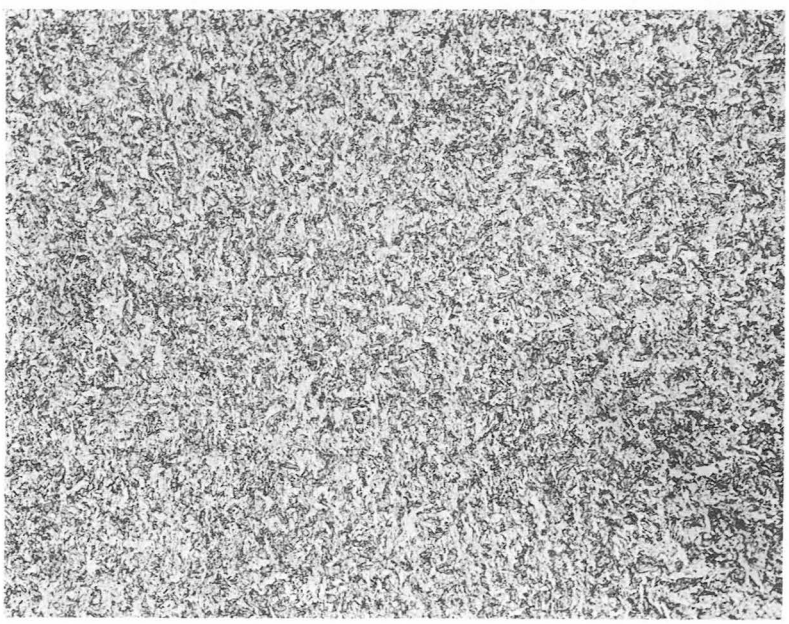
The longitudinal and transverse microstructures of the columbium-modified TZM 5/8 inch diameter bar are shown in Figure 33. The structure was similar to that of the commercial TZM bar although the degree of residual cold work was not as severe. To determine the effect of columbium addition on the recrystallization behavior, specimens were annealed for one hour at various temperatures and the results are presented in Figure 34. The left photomicrograph shows the as-received material, i.e., stress relieved one hour at 2500°F (1371°C). Annealing one hour at 2800°F (1538°C), Figure 34 center, caused recrystallization of a few isolated areas. A slightly greater number of recrystallized areas was apparent after one hour at 3000°F (1649°C), Figure 34 right. The results indicate that the columbium addition tended to raise the recrystallization temperature of the TZM alloy.

10. T-111 Heat 70616

Figure 35 shows the as-received microstructure of T-111 Heat 70616. Although recrystallized at 2400°F (1316°C) prior to shipping, a residual worked structure was still evident in the form of well-defined stringers. The recrystallized material was studied with the electron microscope and electron microprobe. Examination at 6000X, Figure 37, suggested that the stringers were caused by preferential etching of the specimen to produce parallel troughs which appeared as dark lines at 100X. Figure 37 also shows that grain boundaries cross these areas. From these observations and the electron microprobe analysis which failed to show a chemical difference associated with the trough, it was concluded that the areas do not represent an accumulation of precipitates or inclusions.

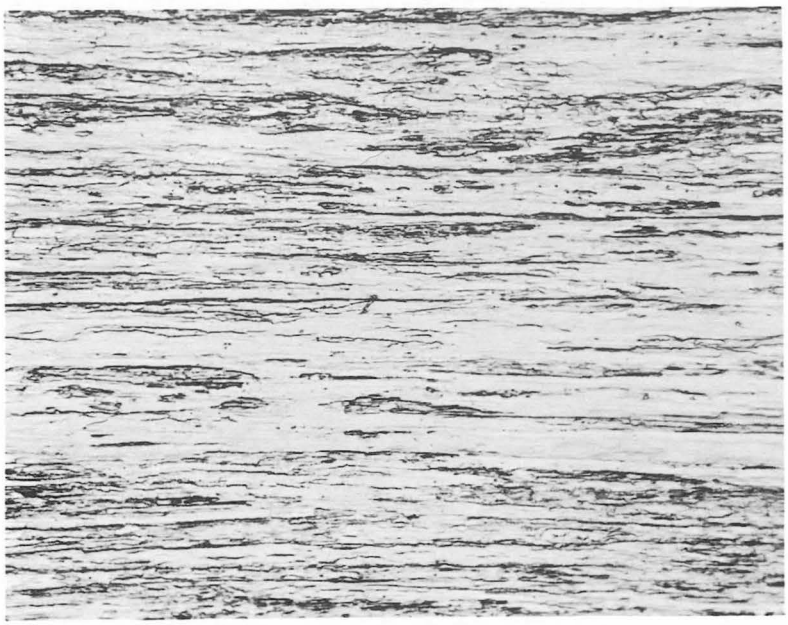


LONGITUDINAL SECTION

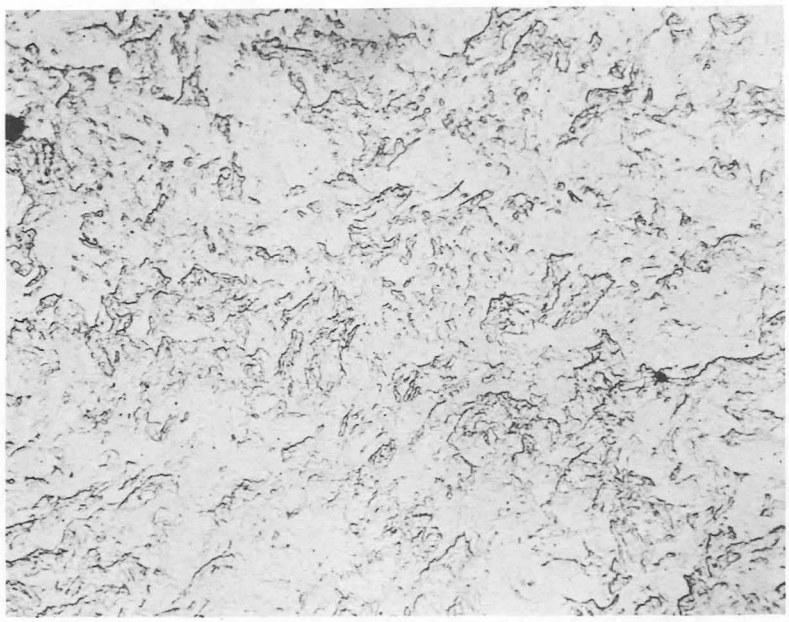


TRANSVERSE SECTION

FIGURE 31 MICROSTRUCTURE OF AS-RECEIVED COMMERCIAL TZM SWAGED BAR (HEAT 7463). ETCHANT: MURAKAMI'S. 100X



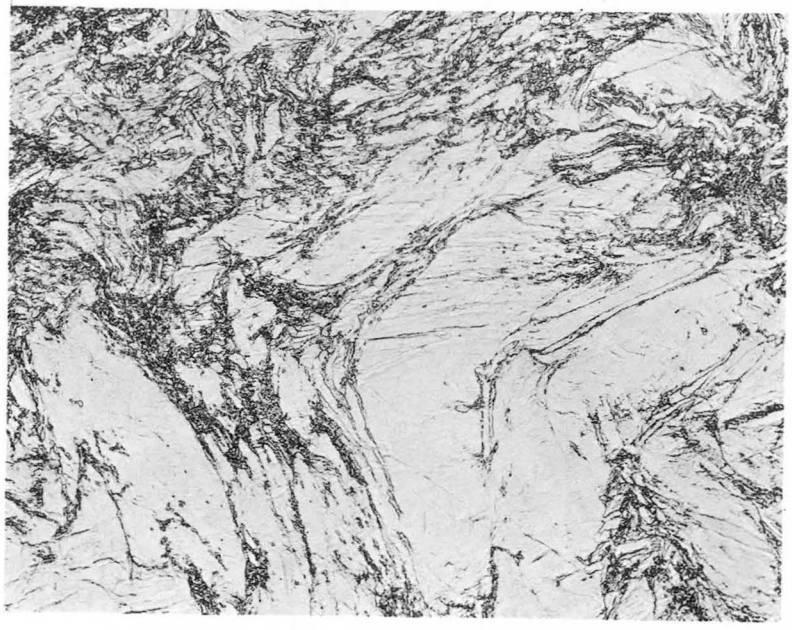
LONGITUDINAL SECTION



TRANSVERSE SECTION

FIGURE 32 MICROSTRUCTURE OF AS-RECEIVED COMMERCIAL TZM SWAGED BAR (HEAT 7463). ETCHANT: MURAKAMI'S. 500X

LONGITUDINAL SECTION



TRANSVERSE SECTION

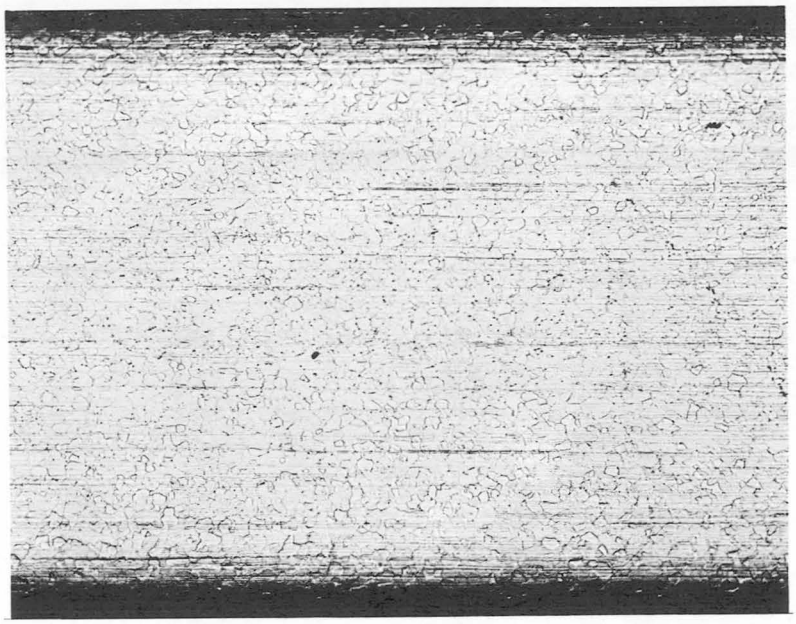
FIGURE 33 MICROSTRUCTURE OF AS-RECEIVED COLUMBIUM-MODIFIED BAR (HEAT 4305-4). ETCHANT: MURAKAMI'S. 100X

ANNEALED 1 HOUR AT 2500°F (1371°C)

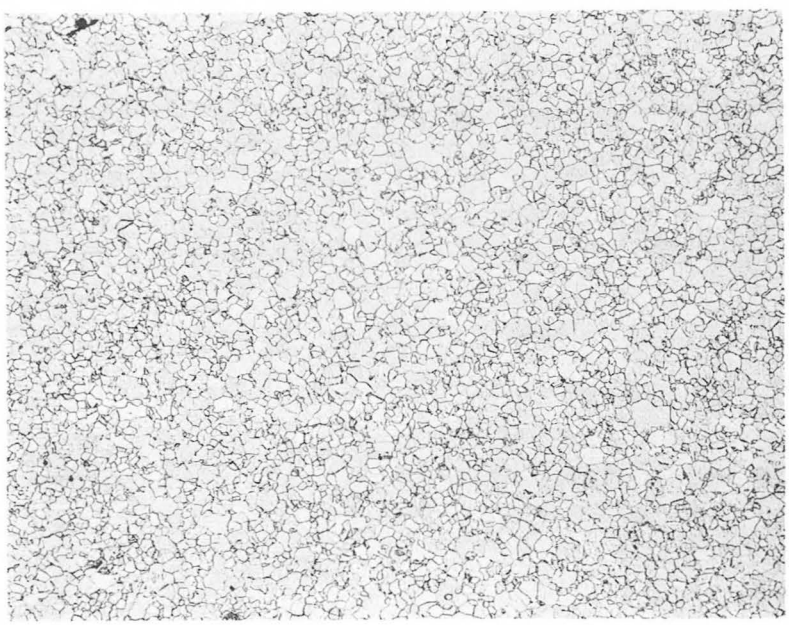
ANNEALED 1 HOUR AT 2800°F (1538°C)

ANNEALED 1 HOUR AT 3000°F (1649°C)

FIGURE 34 COLUMBIUM-MODIFIED TZM ANNEALED AT VARIOUS TEMPERATURES.
SURFACE PERPENDICULAR TO AXIS OF BAR.
ETCHANT: MURAKAMI'S. 100X

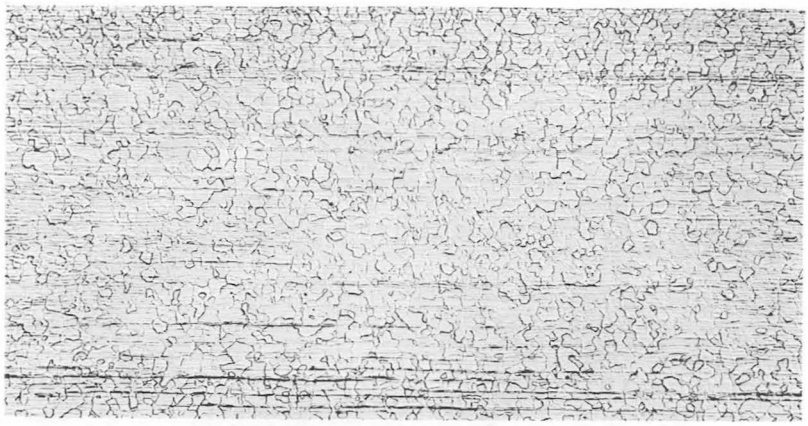


EDGE OF SHEET

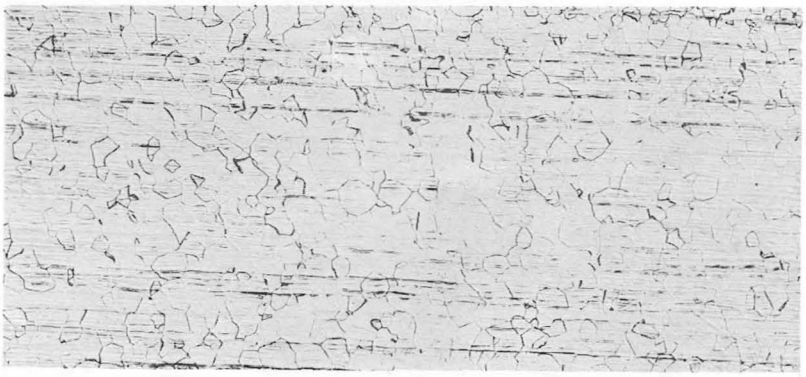


SURFACE OF SHEET

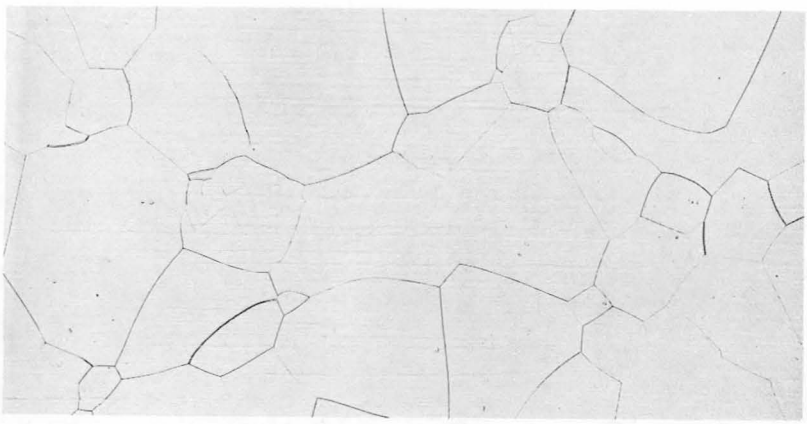
FIGURE 35 MICROSTRUCTURE OF T-111 SHEET (HEAT 70616) RECRYSTALLIZED 2400°F (1316°C), ONE HOUR. ETCHANT: 15% HF, 15% H_2SO_4, 8% HNO_3, 62% H_2O. 100 X



2600°F (1427°C)



3000°F (1649°C)



3500°F (1877°C)

FIGURE 36 MICROSTRUCTURE OF T-111 SHEET (HEAT 70616) RECRYSTALLIZED AT VARIOUS TEMPERATURES - ONE HOUR EDGE OF SHEET. ETCHANT: 15% HF, 15% H_2SO_4, 8% HNO_3, 62% H_2O. 100X

FIGURE 37 ELECTRONMICROGRAPH OF T-111 SHEET (HEAT 70616) ANNEALED AT 2600°F (1427°C) FOR ONE HOUR. CROSS-SECTION SHOWING DETAILS OF STRIATIONS. ETCHANT: 15% HF, 15% H_2SO_4, 8% HNO_3, 62% H_2O. 6000X

11. T-111 Heats 65079, D-1670, D-1102, MCN02A065

The microstructures of these heats are identical to Heat 70616 with the only difference being the grain size as noted below:

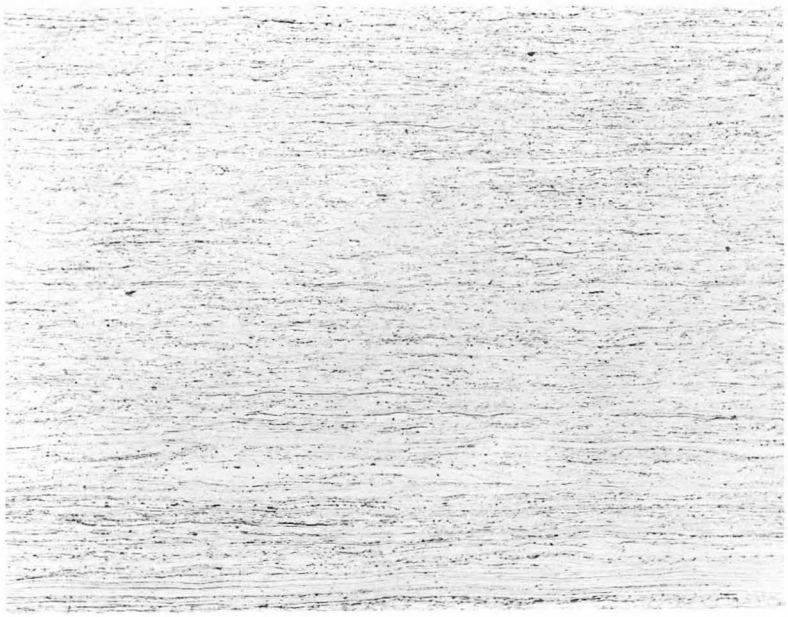
	ASTM Grain Size for 1 Hour Annealing Treatment			
Heat No.	2400°F (1316°C)	2600°F (1426°C)	3000°F (1649°C)	3500°F (1877°C)
70616	7-8	7	5-6	1-3
65079		6-8	5-7	
D-1670			*	
D-1102			5-7	
MCN02A065			6-8	

12. T-222 Heat AL-TA-43 and Heat Ta-43-SF-2

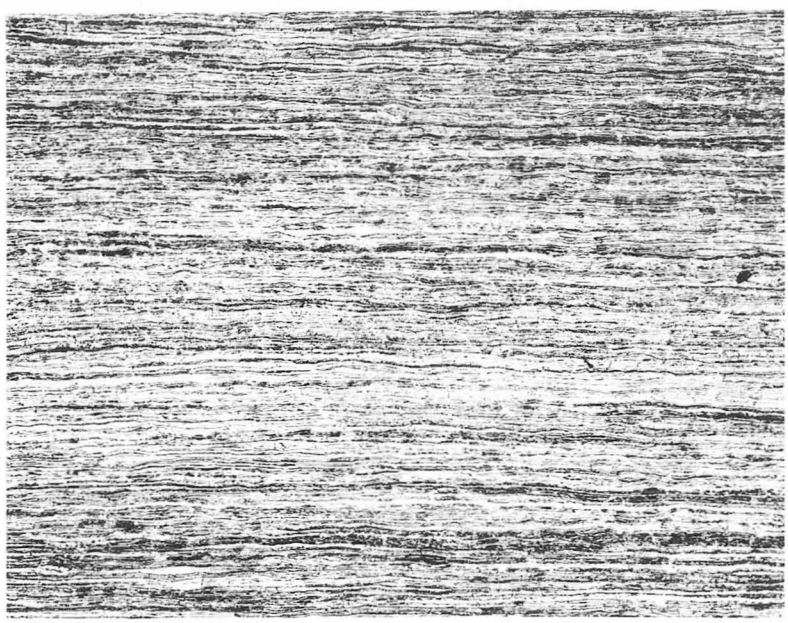
Representative microstructures of the T-222 sheet are shown in Figure 38 for both as-rolled and recrystallized conditions. An increase in grain size was observed with increased recrystallization temperature as shown by Figure 39 and the tabulation below:

Heat	Temperature °F	Temperature °C	ASTM Grain Size
AL-TA-43	2600	1426	6-8
	2800	1538	6-8
	3000	1649	5-6
Ta-43-SF-2	2300	1260	8
	3000	1649	5-6

* Creep tests still in progress. A limited amount of material was available so that metallographic examination must be performed after testing.

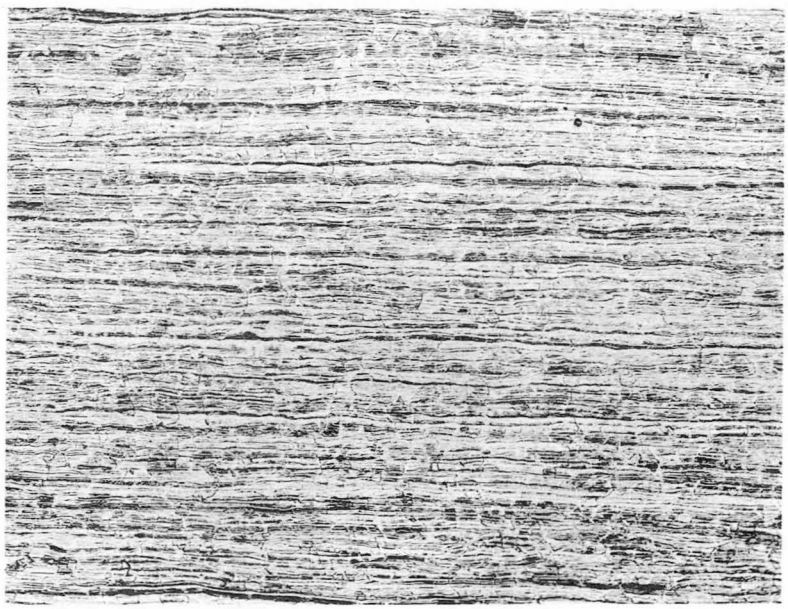


AS-ROLLED

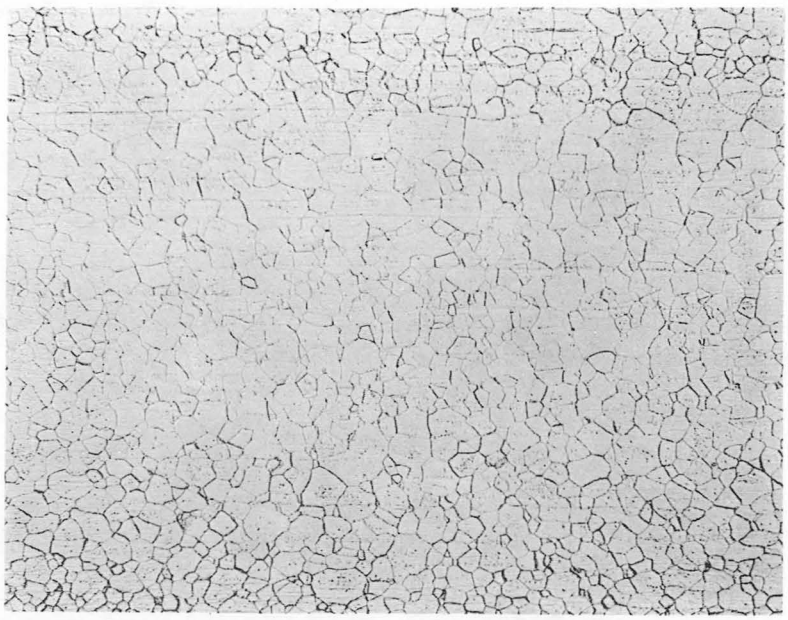


2600°F (1426°C) ONE HOUR

FIGURE 38 T-222 SHEET (HEAT AL-TA-43). ETCHANT: 15% HF, 15% H_2SO_4, 8% HNO_3, 62% H_2O. 100X



2800°F (1538°C) ONE HOUR



3000°F (1649°C) ONE HOUR

FIGURE 39 T-222 SHEET (HEAT AL-TA-43) HEAT TREATED AT TWO TEMPERATURES. ETCHANT: 15% HF, 15% H_2SO_4, 8% HNO_3, 62% H_2O. 100X

13. Astar 811C

Only a limited quantity of this material was received and all was used to fabricate creep specimens. Pre-test metallographic analysis was therefore not performed.

14. Arc-Melted Tungsten Heat KC1357

Figure 40 shows the microstructure of the as-received tungsten sheet. Annealing for one hour at 2800°F (1538°C) or 3200°F (1760°C) caused recrystallization to equiaxed grains, Figures 41 and 42. At the lower temperature well defined grain boundaries were absent suggesting that the recrystallization process was not complete.

15. Vapor Deposited Tungsten

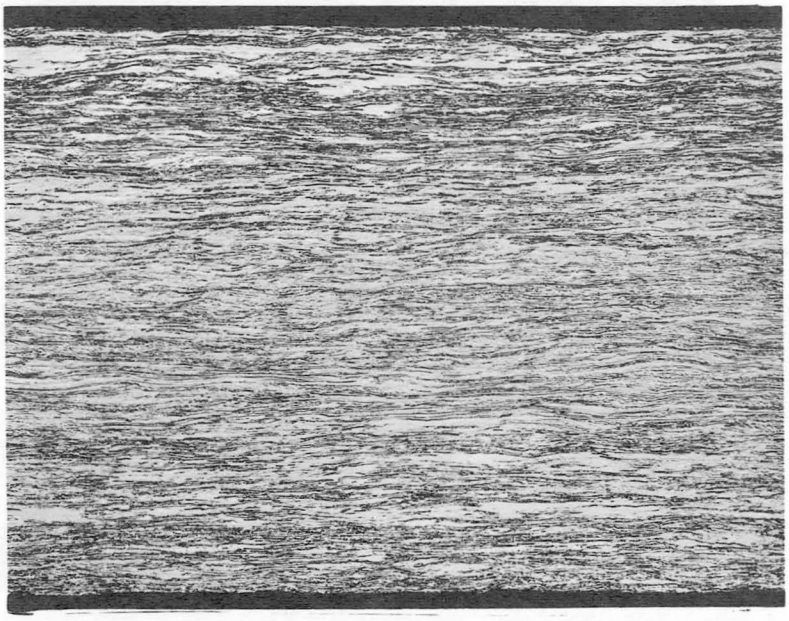
Vapor deposited tungsten was received in the form of a creep specimen. For this reason it was impossible to perform metallographic examination prior to testing.

16. Arc-Melted Tungsten-25% Rhenium, Heat 3.5-75002

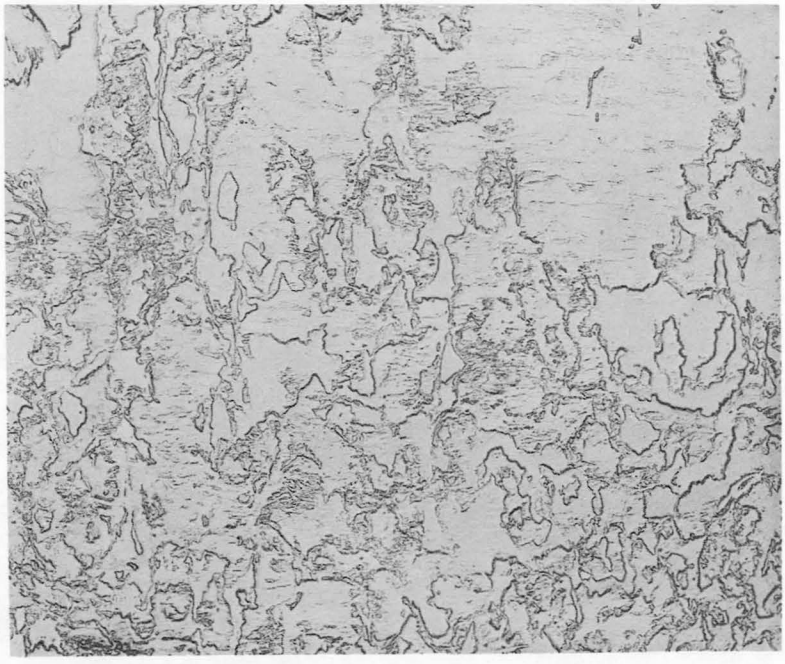
Figure 43 shows the microstructure of the as-received alloy. Annealing one hour at 3200°F (1760°C) produced recrystallization, Figure 44. A comparison of Figures 44 and 42 indicates that the tungsten-25% rhenium alloy possessed reduced grain growth tendencies relative to pure tungsten.

17. Sylvania A

Figure 45 shows the structure of the alloy Sylvania A in the as-received condition and after recrystallization at 3200°F (1760°C) for one hour. The section selected from the original stock was taken through a delaminated area. While Sylvania A alloy is fully recrystallized at 3200°F (1760°C), it exhibits a finer grain size as compared to both tungsten and tungsten-25% rhenium.



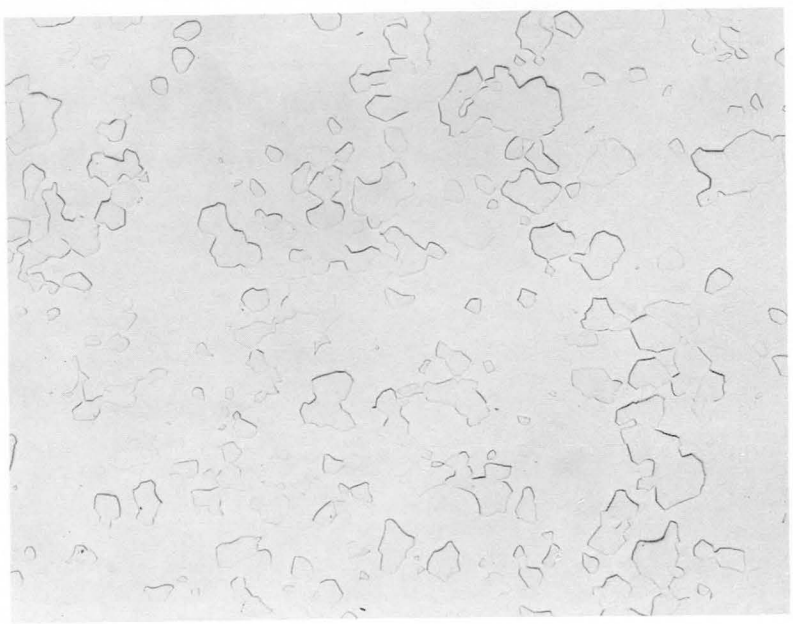
EDGE PERPENDICULAR TO ROLLING DIRECTION



SURFACE OF SHEET

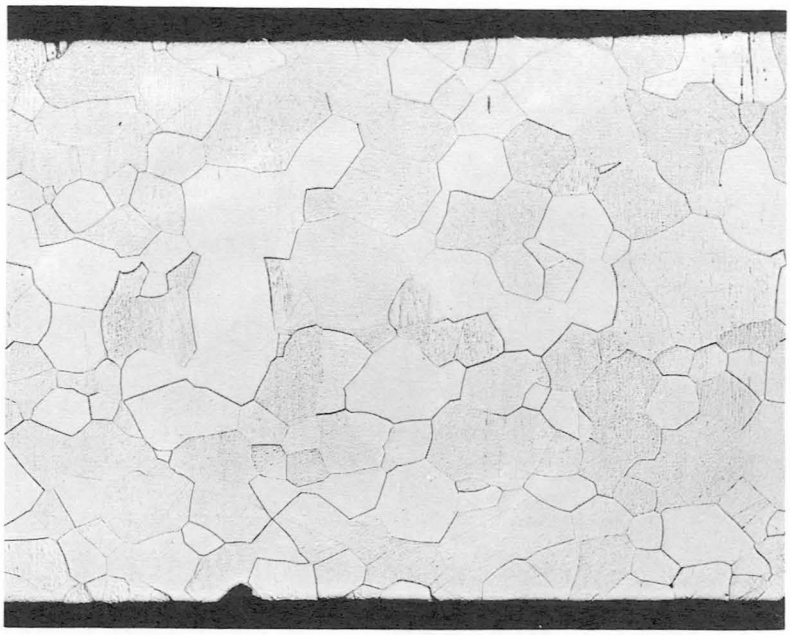
FIGURE 40 MICROSTRUCTURE OF TUNGSTEN SHEET (HEAT KC 1357) IN AS-RECEIVED CONDITION.
ETCHANT: 15% HF, 15% H_2SO_4, 8% HNO_3, 62% H_2O.
100 X

EDGE PARALLEL TO ROLLING DIRECTION

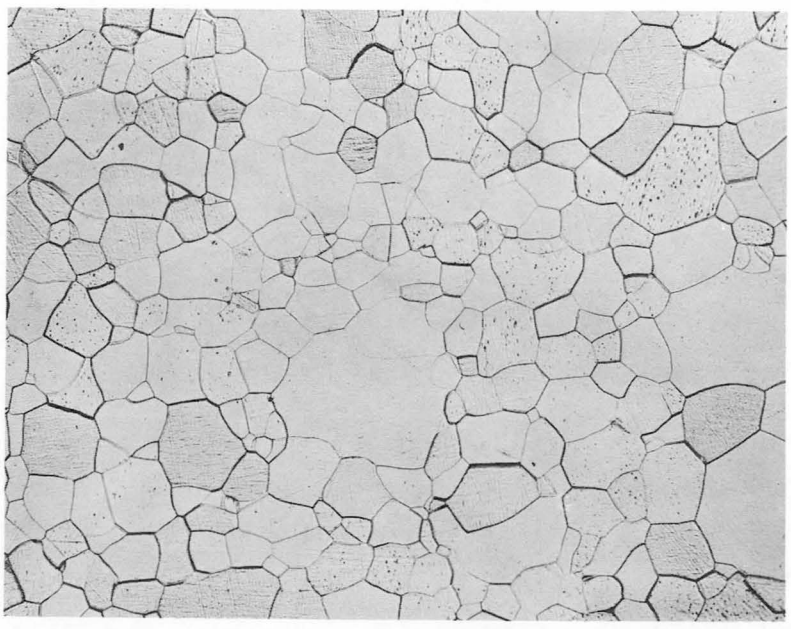


SURFACE OF SHEET

FIGURE 41 MICROSTRUCTURE OF TUNGSTEN SHEET (HEAT KC 1357) ANNEALED ONE HOUR 2800°F (1538°C).
ETCHANT: 15% HF, 15% H_2SO_4, 8% HNO_3, 62% H_2O.
100X

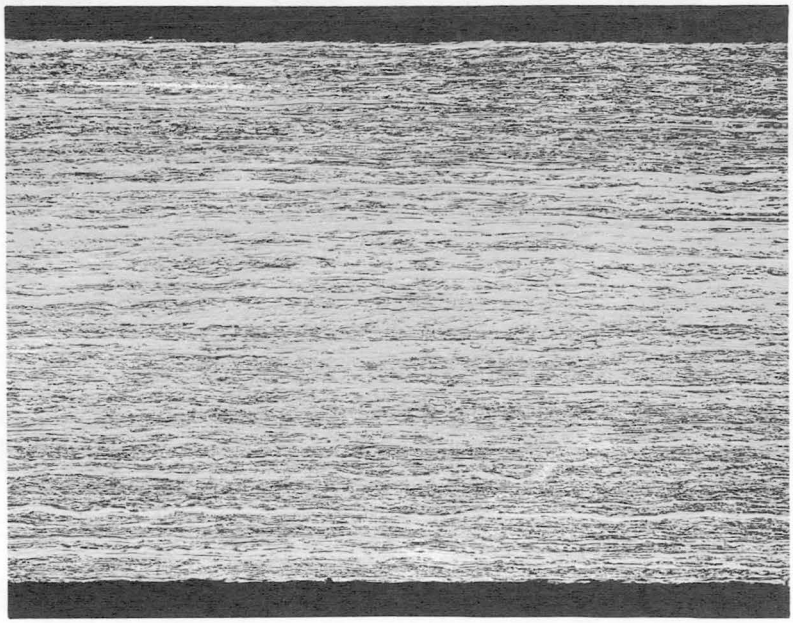


EDGE PERPENDICULAR TO ROLLING DIRECTION

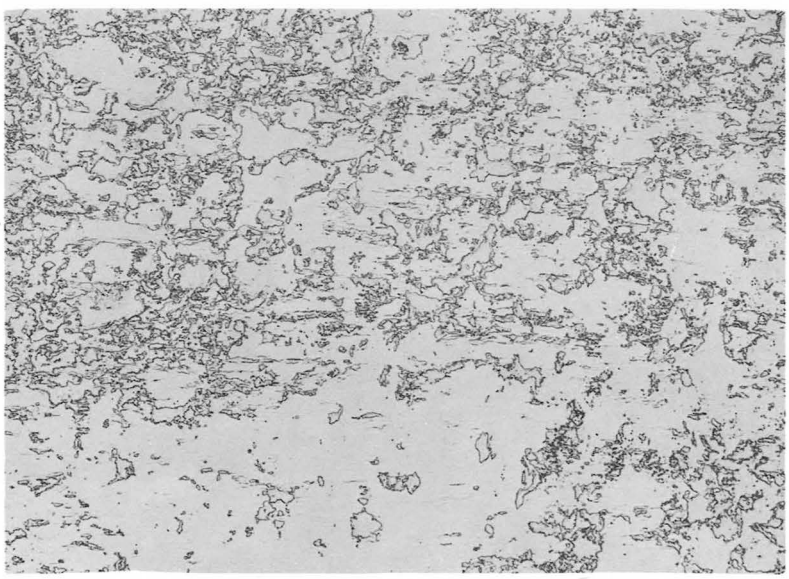


SURFACE OF SHEET

FIGURE 42 MICROSTRUCTURE OF TUNGSTEN SHEET (HEAT KC 1357) ANNEALED ONE HOUR 3200°F (1760°C).
ETCHANT: 15% HF, 15% H_2SO_4, 8% HNO_3, 62% H_2O.
100 X



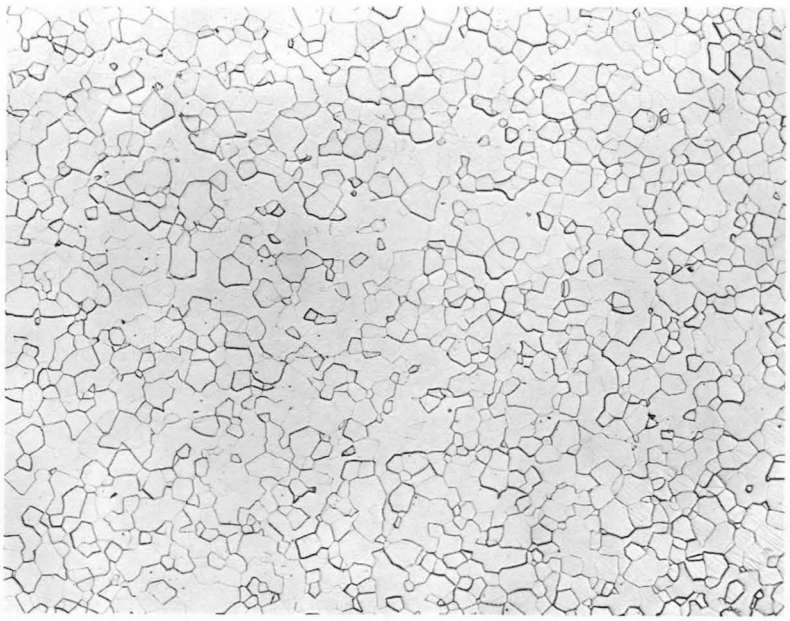
EDGE PERPENDICULAR TO ROLLING DIRECTION



SURFACE OF SHEET

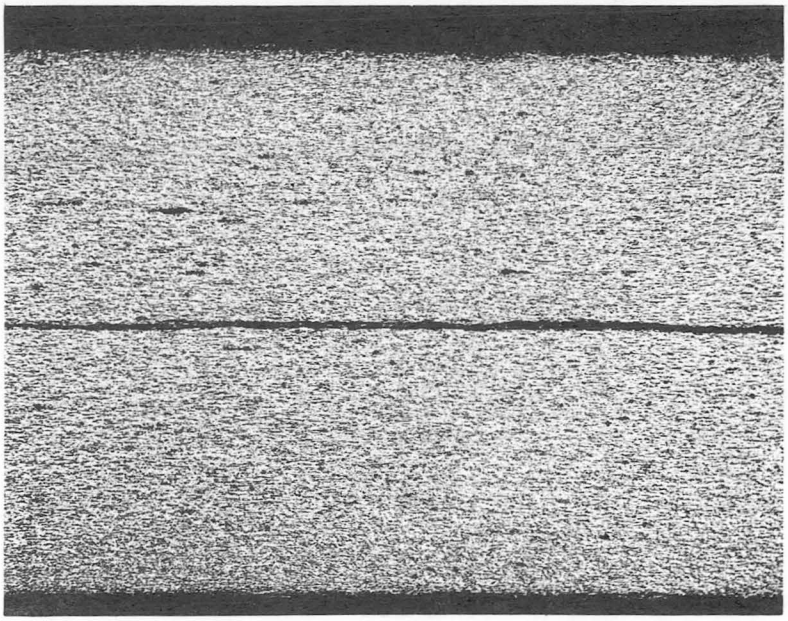
FIGURE 43 MICROSTRUCTURE OF TUNGSTEN - 25% RHENIUM ALLOY SHEET (HEAT 3.5-75002) IN AS-RECEIVED CONDITION.
ETCHANT: 15% HF, 15% H_2SO_4, 8% HNO_3, 62% H_2O. 100X

EDGE PERPENDICULAR TO ROLLING DIRECTION



SURFACE OF SHEET

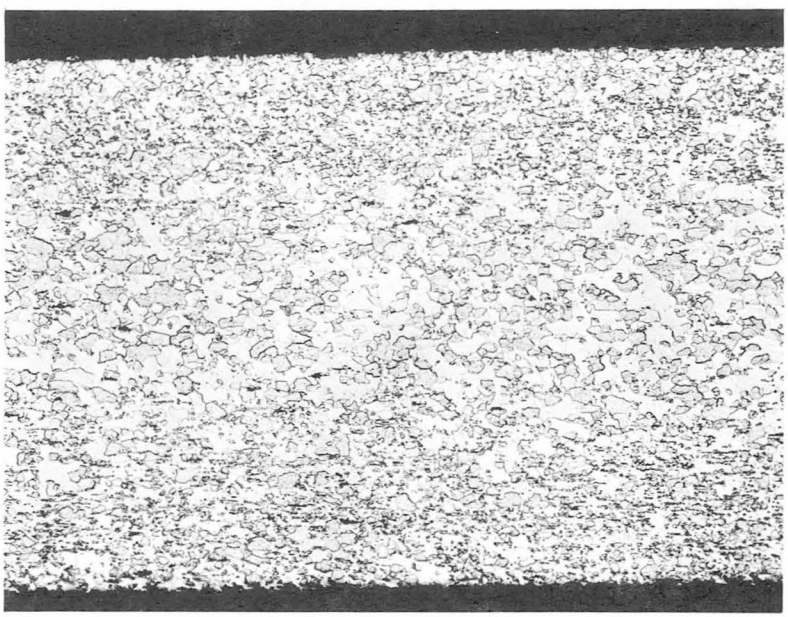
FIGURE 44 MICROSTRUCTURE OF TUNGSTEN - 25% RHENIUM ALLOY SHEET (HEAT 3.5-75002) ANNEALED ONE HOUR 3200°F (1760°C). ETCHANT: 15% HF, 15% H_2SO_4, 8% HNO_3, 62% H_2O. 100X



Delamination

(see text)

EDGE PERPENDICULAR TO ROLLING DIRECTION
AS-RECEIVED



EDGE PERPENDICULAR TO ROLLING DIRECTION
RECRYSTALLIZED ONE HOUR 3200°F (1760°C)

FIGURE 45 MICROSTRUCTURE OF SYLVANIA-A IN AS-RECEIVED AND RECRYSTALLIZED CONDITIONS. ETCHANT: 15% HF, 15% H_2SO_4, 8% HNO_3 62% H_2O. 100X

D. Hardness of Materials

The hardness of all materials was taken as a routine part of metallographic examination. The procedure employed was to obtain the data with metallographic specimens using a Kentron hardness tester. A compilation of the results, given in Table 5, will be used for reference in subsequent discussions. In the case of the TZC alloys, additional comprehensive hardness testing was performed. The results with TZC Heat M-80 showed the following:

1. The hardness of the as-rolled plate varied widely from one location to another, the range being 284 to 338 DPH.

2. Annealing at 3092°F (1700°C) for one hour caused partial recrystallization. The recrystallized grains had a uniform hardness of 251 to 254 DPH.

3. The mean hardness of the unrecrystallized areas, 292 DPH, was statistically lower than the mean hardness of the as-rolled plate, 308 DPH.

TRW Heat M-91 also showed a wide range of hardness in the as-rolled condition, 288 to 390 DPH. Annealing at 3092°F (1700°C) for one hour caused full recrystallization and a substantial decrease in hardness (355 to 240 DPH). When the as-rolled plate was stress relieved at 2500°F (1317°C) for one hour, a small decrease in hardness occurred (335 to 303 DPH).

Because of the range of hardness data, it may be concluded that any change in hardness of TZC due to creep testing may be difficult to significantly identify with creep effects. This is particularly true in the case of stress-relieved material.

T-111 Heats 70616 and 65079 when annealed at 3000°F (1649°C) for one hour had a greater hardness than after a similar treatment at 2600-2800°F (1427-1538°C). Ammon, Filippi, and Harrod (6) have shown that for the similar alloy T-222 these temperatures lie within the solution-precipitation range. Such effects may explain the observed behavior for T-111.

TABLE 5

Hardness of Materials

Material	Heat No.	Heat Treatment °F	Heat Treatment °C	Time Hours	DPH (100 gm load)
AS-30	C5	As Rolled			293
Cb-132M	KC1454	As Rolled			336
	KC1454	3092	1700	1	297
TZC	M-80	As Rolled			308
	M-80	3092	1700	1	240 - 296
	M-91	As Rolled			335
	M-91	3092	1700	1	240
	M-91	+ 3092	1700	1	
		2400	1315	5	280 - 319
	M-91	+ 3092	1700	1	264
		2400	1315	10	
	M-91	2500	1371	1	303
	4345	2400	1315	1	319 - 373
	4345	2500	1371	1	
TZM	7502	2200	1204	1	266 - 342
	7502	2850	1566	1	209
	KDTZM-1175	2300	1260	1	297 - 335
	7463	2250	1232	0.5	300 - 330
	Cb-modified 4305-4	2500	1371	1	278
	"	2800	1538	1	245
	"	3000	1649	1	279
T-111	70616	2400	1316	1	216 - 368
	70616	2600	1427	1	211 - 266
	70616	3000	1649	1	260 - 287
	70616	3000	1649	16	237 - 271
	70616	3500	1877	1	234 - 242
	65079	2800	1538	1	236
	65079	3000	1649	1	253 - 296
	D-1670	As Received			323 - 342
	D-1670	3000	1649	1	
	D-1102	As Received			
	D-1102	3000	1649	1	
	MCN02A065	Not Available			

TABLE 5 (Continued)

Material	Heat No.	Heat Treatment °F	°C	Time Hours.	DPH (100 gm load)
T-222	AL-TA-43	As Rolled			413
	AL-TA-43	2600	1426	1	314
	AL-TA-43	2800	1538	1	317
	AL-TA-43	3000	1649	1	357
	Ta-43-SF-2	2300	1260	1	309 - 353
	Ta-43-SF-2	3000	1649	1	357
Tungsten	KC1357	As Rolled			487
	KC1357	2800	1538	1	421
	KC1357	3200	1760	1	407
W-25%Re	3.5-75002	2375	1302	1	639
	3.5-75002	2550	1399	1	639
	3.5-75002	3200	1760	1	568
Sylvania A		2732	1500	5 min.	579
		3200	1760	1	453

E. Material Tensile Strength

Tensile tests were performed on samples of TZC Heats M-80, M-91, and 4345 as well as on samples of T-111 alloy Heat 65079. Where elevated temperature tests were made, the ambient vacuum was in the 10^{-6} torr range. The data obtained are given in Tables 6 and 7.

TZC Heat M-80 lacked ductility at room temperature. A 25-hour treatment at 2750°F (1510°C) alleviated this condition while two other treatments failed to improve the ductility. One of the two unsuccessful treatments was a 1 hour anneal at 3092°F (1700°C) and the other was a 1 hour anneal at 3092°F (1700°C) followed by a 1 hour anneal at 2400°F (1316°C).

TZC Heat M-91 was ductile at room temperature due to the greater severity of reduction during processing. Stress relieving at 2500°F (1371°C) for one hour had little effect on the tensile properties while recrystallization at 3092°F (1700°C) for one hour produced a substantial decrease in strength. Aging at 2400°F (1316°C) after recrystallizing had little effect on the tensile properties of TZC Heat M-91. TZC Heat 4345 appeared to have slightly better tensile properties than Heat M-91.

T-111 Heat 65079 was the only lot of T-111 alloy selected for tensile testing. As indicated in Table 7, a comparison was made between specimens with tensile axes both parallel and perpendicular to the direction of rolling. The data indicate that the tensile properties of this particular heat of T-111 are independent of the principal directions to rolling.

TABLE 6

TZC Tensile Properties

Heat No.	Condition	Test Temp, °F	Strength, ksi Ultimate	Yield	%El 1"G.L.	%R. A.
M-80 (1)	2750°F-25 Hrs.	R. T.	100	68	7	10
	3092°F- 1 Hr.	R. T.	69	68	1	0
	3092°F- 1 Hr. 2400°F- 1 Hr.	R. T.	95	90	1	0
M-91 (2)	As Rolled	R. T.	141	99	7	7
	2500°F- 1 Hr.	R. T.	106	99	4	7
	3092°F- 1 Hr.	R. T.	85	49	7	7
	3092°F- 1 Hr.	1800°F	45	29	34	88
	3092°F- 1 Hr.	2000°F	41	25	43	88
	3092°F- 1 Hr.	2200°F	35	24	42	88
	3092°F- 1 Hr. 2400°F- 5 Hrs.	R. T.	75	45	8	8
	3092°F- 1 Hr. 2400°F-10 Hrs.	R. T.	72	50	4	6
4345 (3)	2500°F- 1 Hr.	R. T.	123	117	12	19
	2500°F- 1 Hr.	1800°F	77	75	17	61
	2500°F- 1 Hr.	2200°F	62	61	18	61
	3092°F- 1 Hr.	R. T.	88	50	19	17
	3092°F- 1 Hr.	1800°F	63	27	29	68
	3092°F- 1 Hr.	2000°F	61	24	28	70
	3092°F- 1 Hr.	2200°F	54	25	29	66

(1) M-80 Extruded 2.3:1 at 3092°F (1700°C) and cross-rolled at 2925°F (1585°C) with 4% reduction per pass.

(2) M-91 Extruded 2.3:1 at 3092°F (1700°C) and cross-rolled with large deformations per pass, finishing at 2372°F (1300°C).

(3) 4345 Extruded at 3000°F (1649°C), upset forged 40% at 2400°F (1316°C), broad forged to 0.825" at 2400°F (1316°C), vacuum annealed at 2400°F (1316°C).

TABLE 7

T-111, Heat No. 65079, Tensile Properties

Condition	Test Temp, °F	Strength, ksi Ultimate	Yield	%El. 1" G. L.
3000°F-1 Hr.	R. T.	94 (93)*	74 (72)	35 (34)
3000°F-1 Hr.	1800	74 (76)	34 (34)	23 (24)
3000°F-1 Hr.	2200	49 (51)	32 (32)	30 (27)
3000°F-1 Hr.	2600	29 (29)	25 (26)	50 (50)

* ( ) Indicates specimen axis perpendicular to rolling direction. Otherwise specimen axis parallel to rolling direction.

V RESULTS AND DISCUSSION

A. Methods of Presenting the Creep Data

The specific creep data for each test will be presented as total specimen extension plotted as a function of time under load at the selected test temperature. At periodic intervals on the creep curves, the system pressure will be noted to indicate any variations in test environment. To simplify comparisons between different material or material conditions and to aid in design predictions, the data are also presented in the form of Larson-Miller and Manson-Haferd plots (8, 9). Although more generalized parametric equations, which combine the time, temperature, and variables are available (8) with limited data they do not provide any improvement in prediction capability.

B. Columbium-Base Alloys AS-30 and Cb-132M

Three creep tests were made using the as-rolled columbium-base alloy AS-30 and the data are presented in Figure 46. The creep curves show consistent discontinuities which are real effects and not due to scatter in the extension readings. Although similar effects, observed with numberous commercial alloys (9-12), have been associated with an isothermal instability of the material under study no attempt was made to evaluate whether such instabilities existed in the AS-30 alloy.

The creep curves for the Cb-132M alloy which was annealed at 3092°F (1700°C) for one hour prior to testing, are presented in Figure 47. A summary of the pertinent data for both AS-30 and Cb-132M is given in Table 8. Post-test examination indicated that no change in hardness or structure occurred in either alloy as a result of creep exposure.

A comparison of the AS-30 and Cb-132M alloy is presented in Figure 48 on the basis of the Larson-Miller parameter. The results indicate that the Cb-132M has significantly greater creep strength.

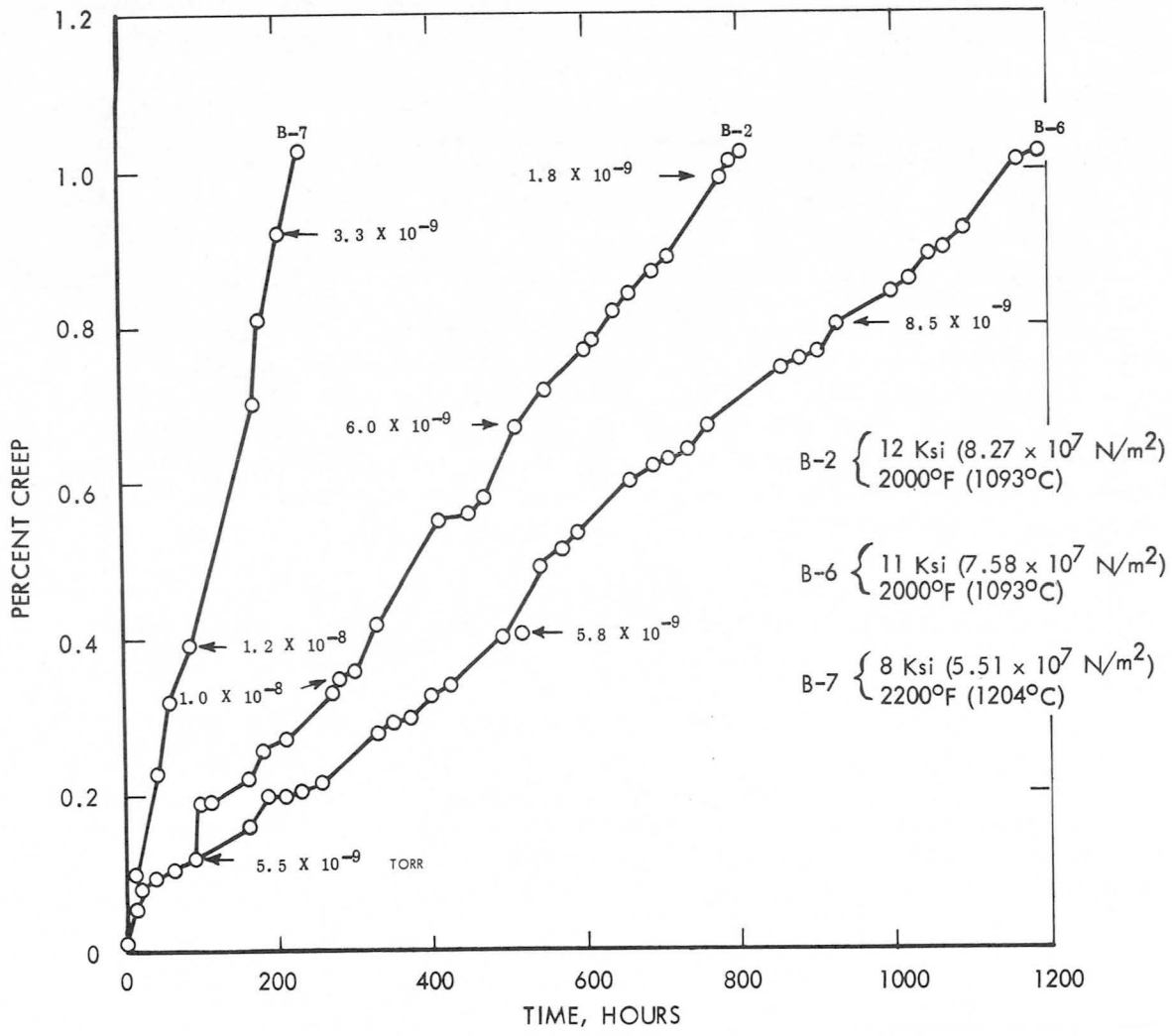


FIGURE 46 CREEP DATA FOR AS-30 PLATE (HEAT C-5) IN AS-ROLLED CONDITION. TESTED IN VACUUM ENVIRONMENT $< 1 \times 10^{-8}$ TORR.

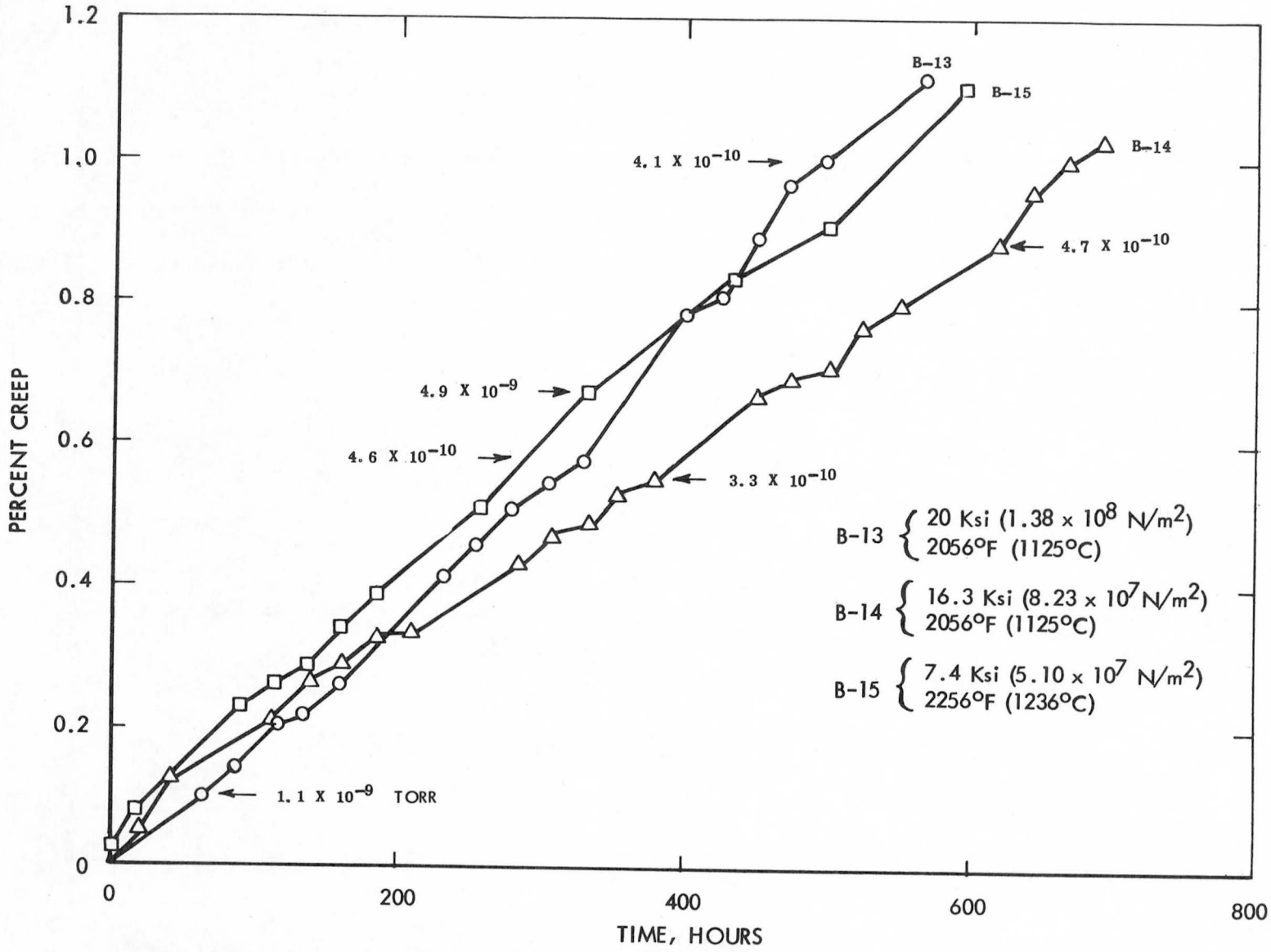


FIGURE 47 CREEP DATA FOR Cb-132M PLATE (HEAT KC-1454) ANNEALED 3092°F (1700°C), 1 HOUR. TESTED IN VACUUM ENVIRONMENT $<1 \times 10^{-8}$ TORR.

TABLE 8

Summary of Creep Data for Columbium-Base Alloys

Spec. No.	Test Temperature °F	°C	Stress ksi	N/m^2	Creep Hours 0.5%	1%	Larson-Miller Parameter $T°R\,(15 + \log t) \times 10^{-3}$ 0.5%	1.0%	Creep Rate $in\text{-}in^{-1}\text{-}hr^{-1}$
AS-30 Heat C5									
B-2	2000	1093	12	8.27×10^7	390	790	43.3	44.0	1.3×10^{-5}
B-6	2000	1093	11	7.58×10^7	450	1170	43.5	44.5	8.5×10^{-6}
B-7	2200	1204	8	5.51×10^7	115	225	45.4	46.1	4.5×10^{-5}
Cb-132M Heat KC1454									
B-13	2056	1125	20	1.38×10^8	275	495	43.8	44.4	2.0×10^{-5}
B-14	2056	1125	16.3	8.23×10^7	340	670	44.0	44.7	1.5×10^{-5}
B-15	2256	1236	7.4	5.10×10^7	250	550	47.2	48.1	1.8×10^{-5}

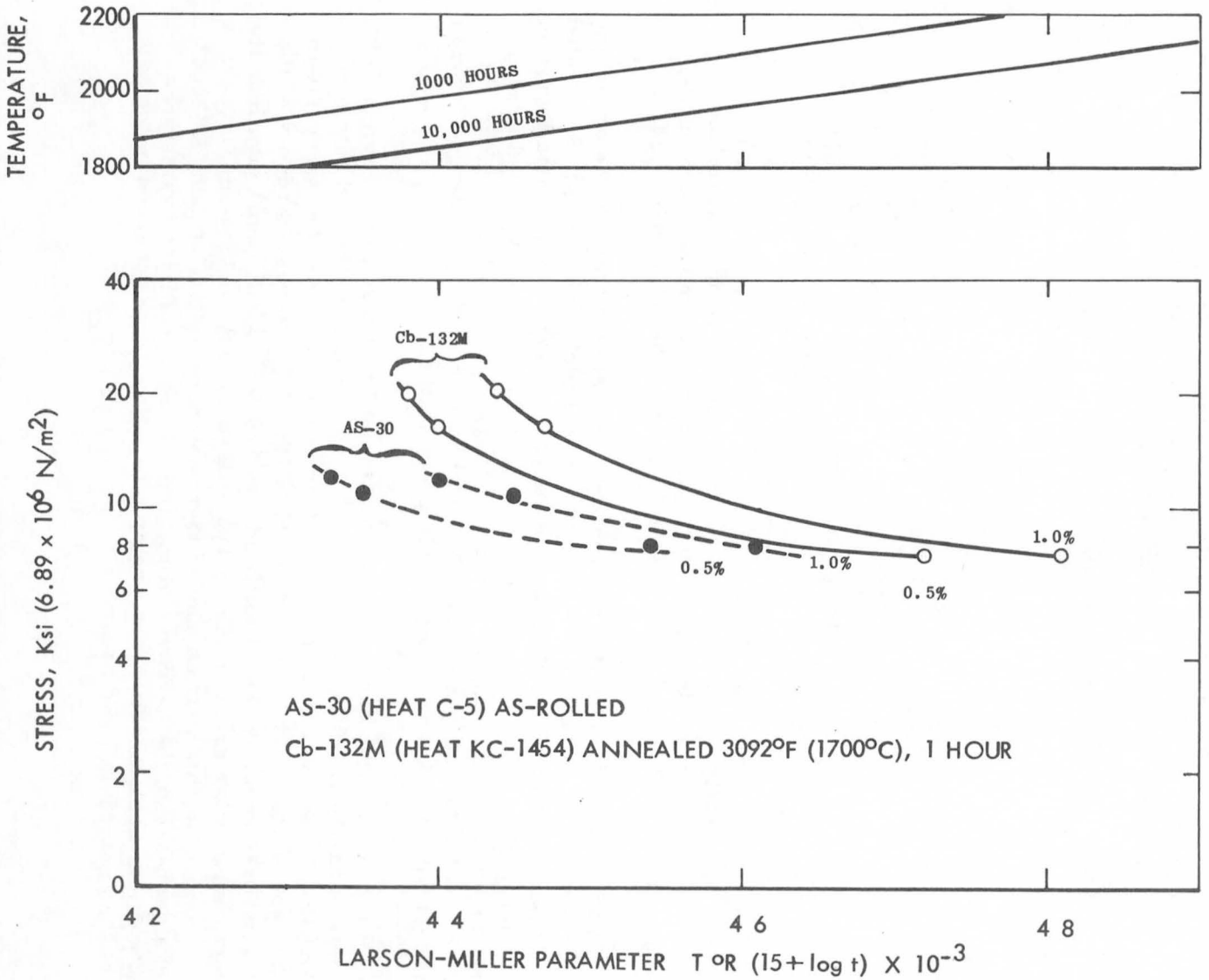


FIGURE 48 LARSON-MILLER PLOT FOR 0.5 AND 1.0% CREEP IN AS-30 AND Cb-132M PLATE. TESTED IN VACUUM ENVIRONMENT $<1 \times 10^{-8}$ TORR.

C. Molybdenum-Base Alloys

1. TZC Heat M-80

Eight creep tests were made with specimens of TZC Heat M-80 recrystallized at 3092°F (1700°C) for one hour prior to evaluation. The test conditions are shown below and the creep data are plotted in Figures 49 and 50.

Specimen	Test Temperature °F	Test Temperature °C	Stress ksi	Stress N/m^2
B-5	2000	1093	14	9.65×10^7
B-5A	2000	1093	20	1.38×10^8
B-5B	2000	1093	22	1.52×10^8
B-8A	2200	1204	18	1.24×10^8
B-9	2000	1093	20	1.38×10^8
B-10	2200	1204	17	1.17×10^8
B-11	1856	1013	25	1.72×10^8
B-12	2056	1125	19	1.31×10^8

Tests B-5, B-5A, and B-5B were not plotted because the tests were conducted sequentially on the same specimen to establish approximate condition for further testing. In Figure 50 the discontinuties in the creep curve do not appear after 5000 hours due to stabilization of the microstructure.

In an effort to identify the microstructural changes that may be occurring during creep testing, a detailed examination was made of specimen B-8A, tested for 2128 hours at 2200°F (1204°C) and 18 ksi (1.24×10^8 N/m^2). Metallographic examination showed that the gage section surface was thermally etched following testing, Figure 51. The microstructure of the gage cross section, Figure 51, indicated that the partially-recrystallized structure remained unchanged throughout the test with the exception of possible precipitation.

A comprehensive hardness examination was also made of specimen B-8A. The average hardness of the recrystallized grains in the unstressed button head was 242 DPH. This was 11 points lower than the hardness of similar grains in a sample recrystallized but not tested, 253 DPH. The average hardness of the unrecrystallized areas in the button head was 289 DPH which is not significantly different than similar areas in the recrystallized but untested specimens, 292 DPH. From these data it is concluded that prolonged heating at 2200°F (1204°C) caused a decrease in the hardness of the original recrystallized grains presumably due to agglomeration of a precipitate. No change occurred in the hardness of the unrecrystallized areas.

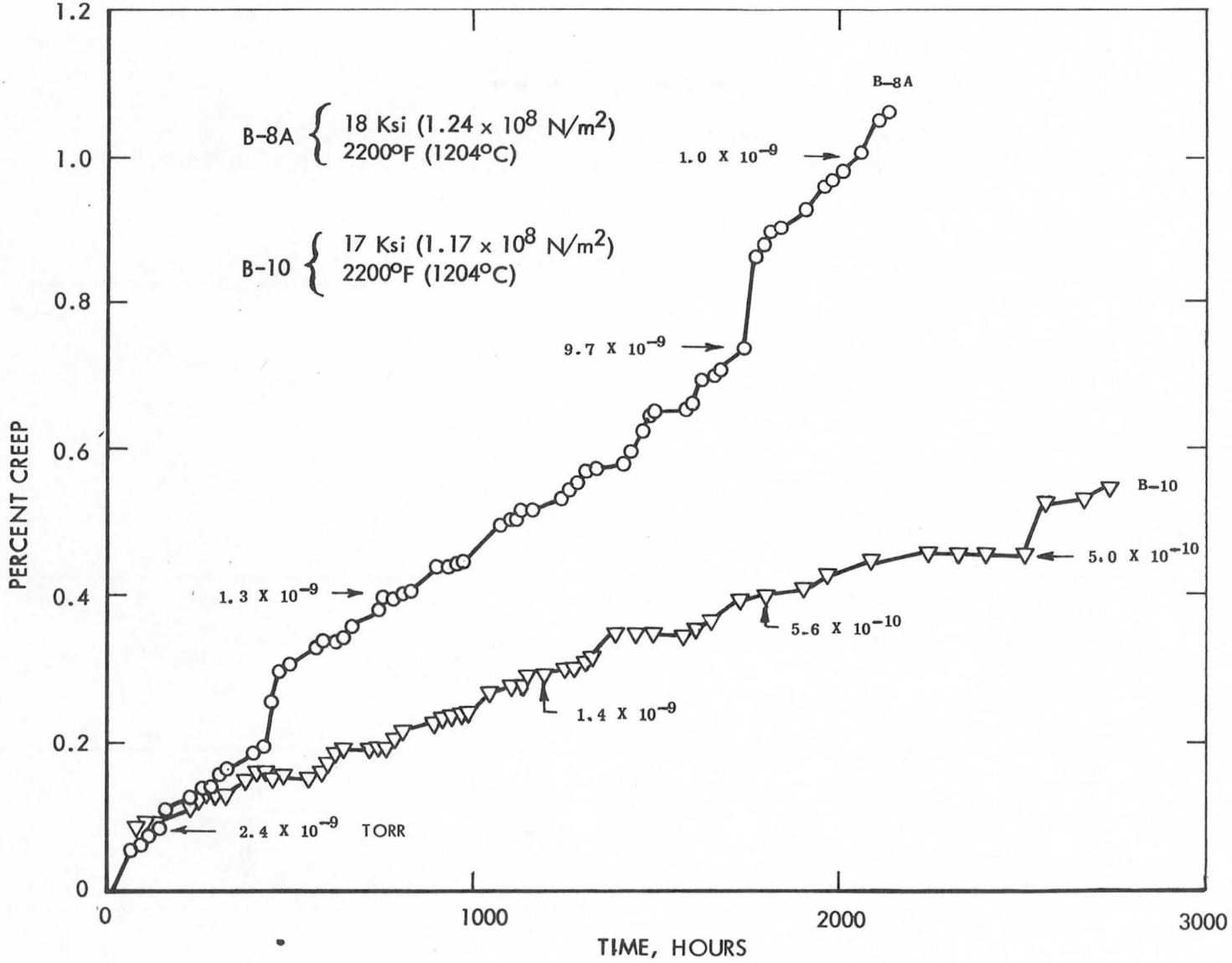


FIGURE 49 CREEP DATA TZC PLATE (HEAT M-80) RECRYSTALLIZED ONE HOUR AT 3092°F (1700°C). TESTED IN VACUUM ENVIRONMENT $< 1 \times 10^{-8}$ TORR.

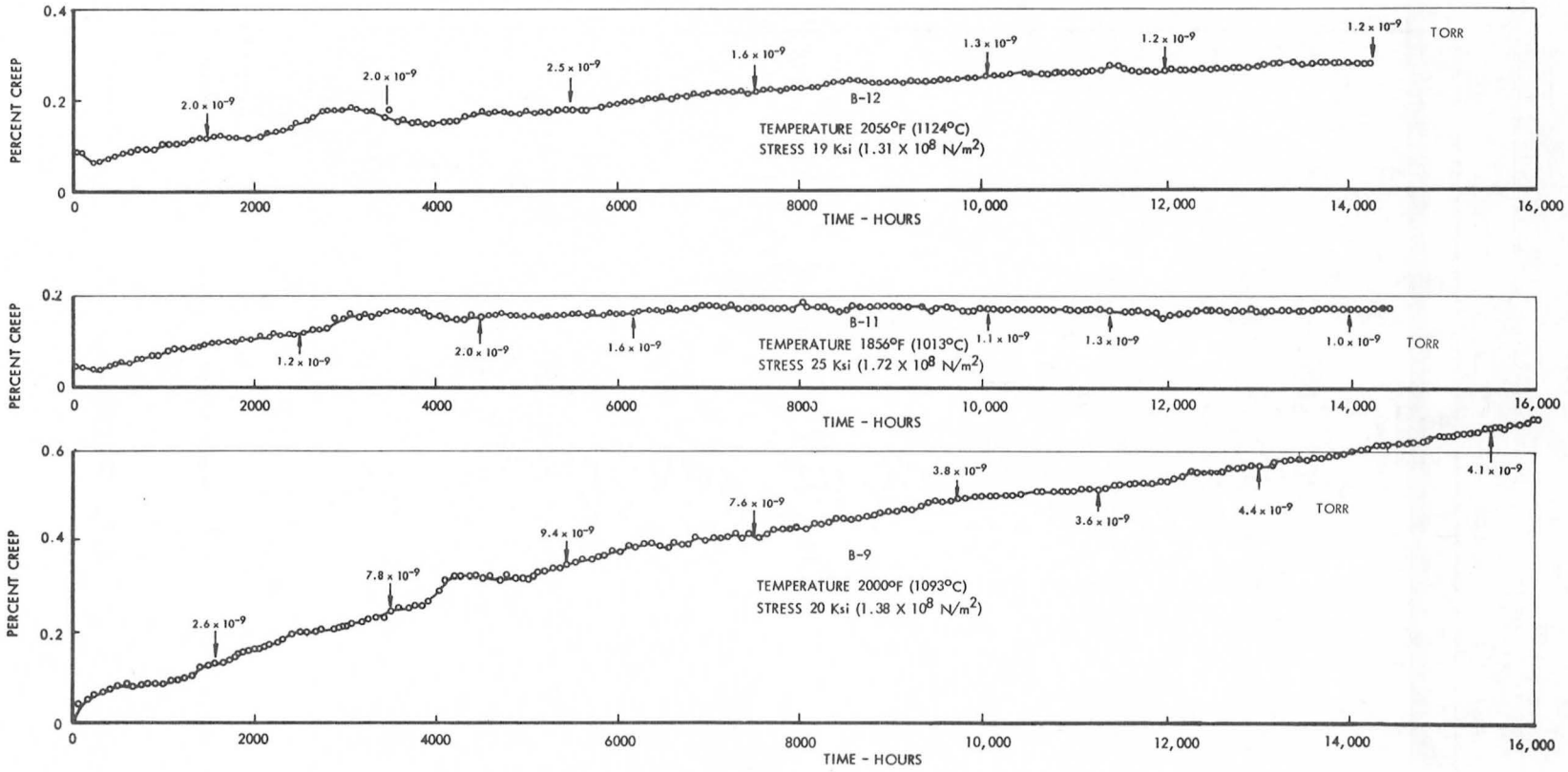
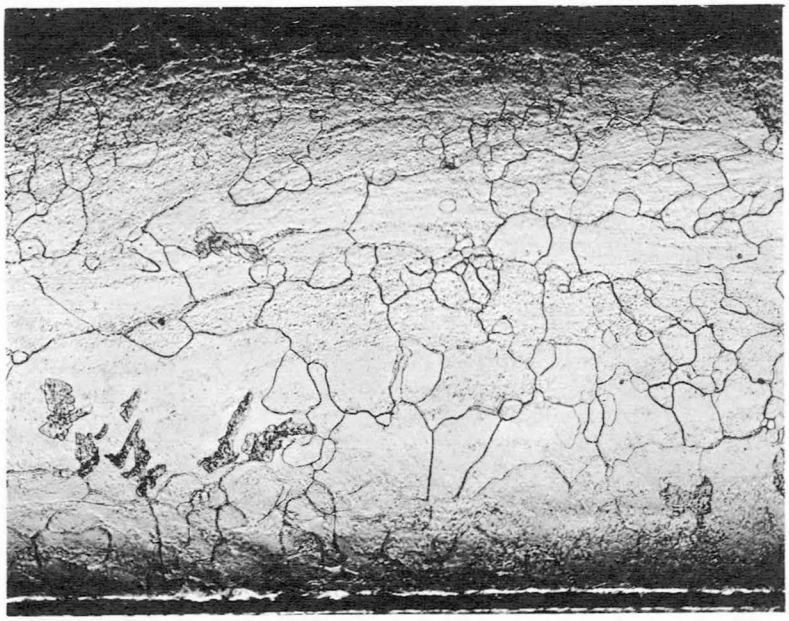


FIGURE 50 CREEP TEST DATA, TZC PLATE (HEAT M-80), ANNEALED 3092°F (1700°C), 1 HOUR.TESTED IN VACUUM ENVIRONMENT $<1 \times 10^{-8}$ TORR.



TENSILE AXIS

THERMAL ETCHED SURFACE OF CREEP SPECIMEN IN GAGE SECTION

⊕ TENSILE AXIS

CROSS-SECTION OF GAGE SECTION

FIGURE 51 TZC PLATE (HEAT M-80) SPECIMEN B-8A. TESTED 2128 HOURS AT 2200°F (1204°C).
ETCHANT: 15% HF, 15% H_2SO_4, 8% HNO_3, 62% H_2O. 100X

Examination of the recyrstallized grains in the gage section showed a hardness of 264 DPH compared to 242 DPH for the unstressed button head. This would indicate that work hardening occurred in the stressed area during testing. The unrecrystallized areas showed no appreciable hardness change which suggests that the major creep contribution occurred preferentially in the recrystallized grains.

Specimen B-9 tested for 16,002 hours to 0.670% elongation at 2000°F (1093°C) was tensile tested at room temperature. The results were identical to a heat treated but untested specimen with the exception of a slight increase in the percent elongation and reduction of area.

2. TZC Heat M-91

Eight creep tests were made with specimens of TZC Heat M-91 subjected to various heat treatments prior to testing.

Spec. No.	Test Temperature °F	°C	Stress ksi	N/m^2	Heat Treatment °F	°C	Hrs.
B-20	2000	1093	20	1.38×10^8	3092	1700	1
B-31	2200	1204	14	9.65×10^7	3092	1700	1
B-22	2000	1093	20	1.38×10^8	3092	1700	1
					2400	1316	5
B-30	2200	1204	22	1.52×10^8	2500	1371	1
B-32	1935	1057	20	1.38×10^8	2500	1371	1
B-33	1900	1038	22	1.52×10^8	2500	1371	1
B-19	1800	982	44	3.03×10^8	2300	1260	1
B-28	2000	1093	28	1.93×10^8	2300	1260	1

The creep data plotted in Figures 52, 53, and 54 show many indications of non-uniform specimen extension. These deviations in the creep curve are real as they are greater than the scatter due to errors in measuring extension.

The results obtained with specimen B-22, annealed and aged prior to testing are open to question. Post-test examination of this specimen revealed a longitudinal crack in the gage section, Figure 55, and visual examination showed a high degree of localized distortion had occurred in some areas which may have influenced the average creep readings. The area of extensive deformation, shown at 500X in Figure 56, consists of grains exhibiting pronounced slip. A review of the creep curve, Figure 54, gives no indication that cracking occurred during test. Since cracking during heat treatment has been reported for the same heat of material (13), it was concluded that the specimen cracked during the heat treatment which was performed in situ prior to creep testing.

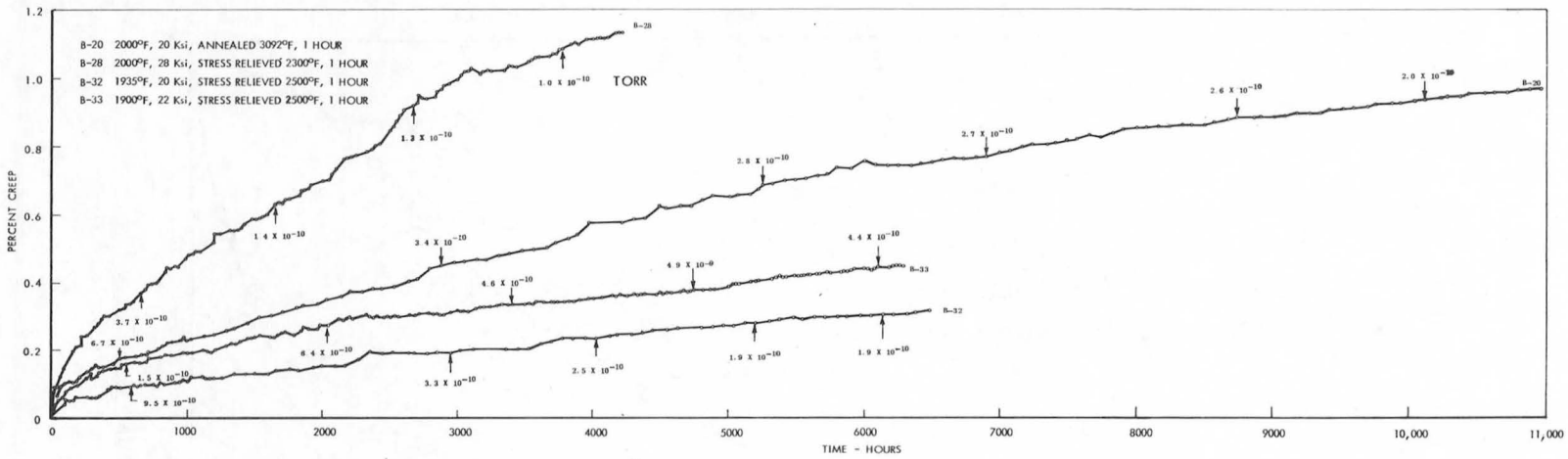


FIGURE 52 CREEP DATA, TZC (HEAT M-91) TESTED IN VACUUM ENVIRONMENT <1 X 10^{-8} TORR.

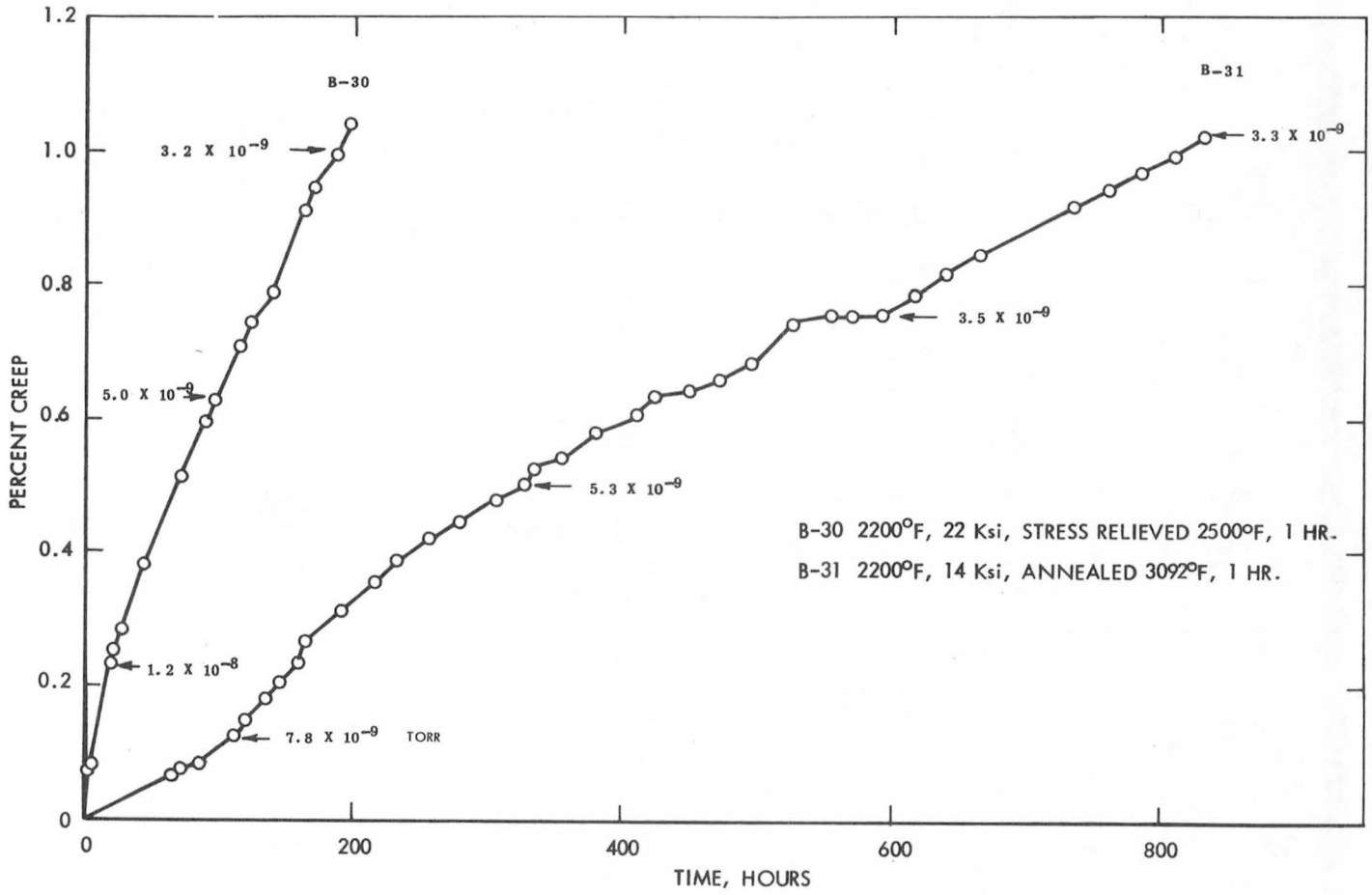


FIGURE 53 CREEP DATA FOR TZC PLATE (HEAT M-91). TESTED IN VACUUM ENVIRONMENT $<1 \times 10^{-8}$ TORR.

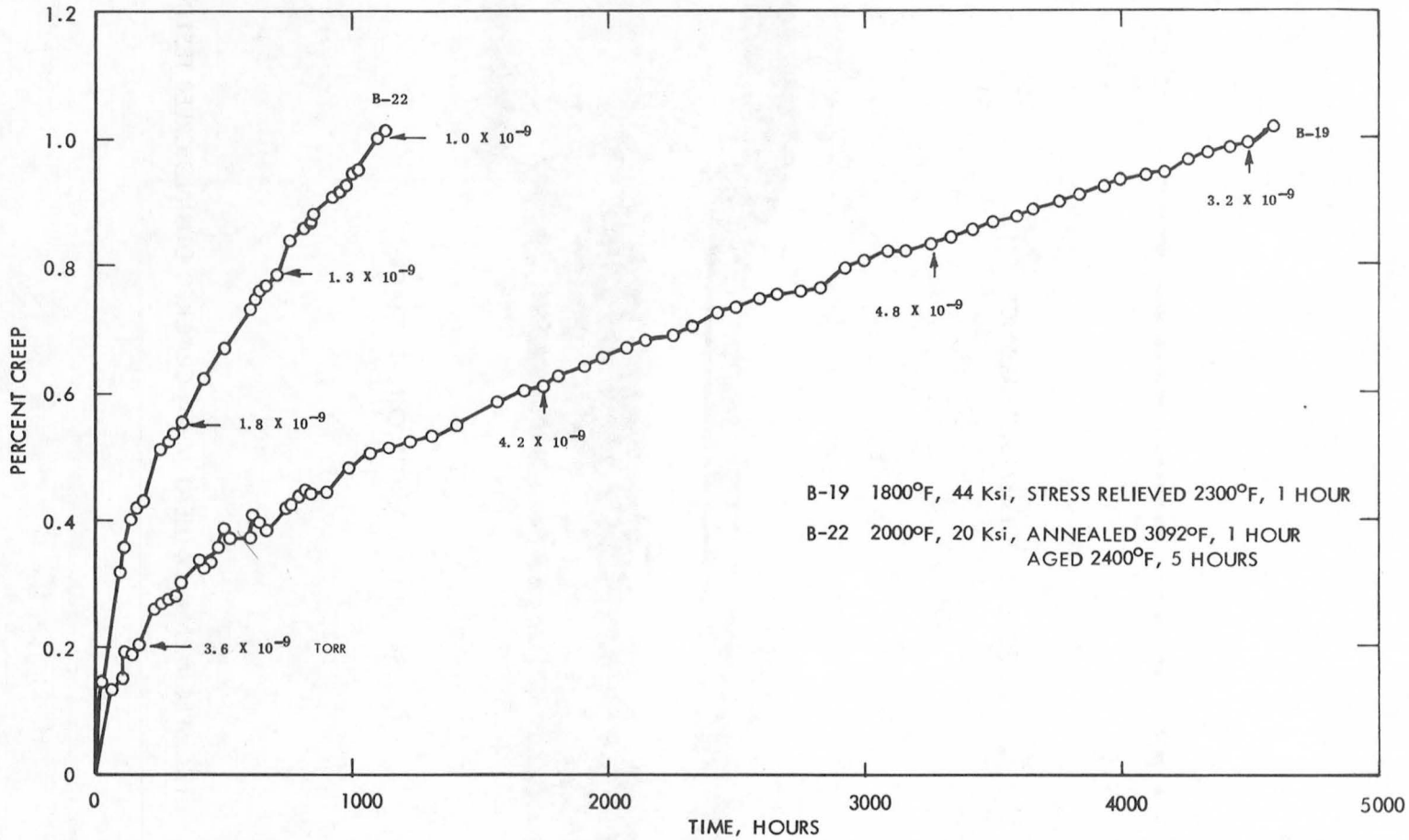
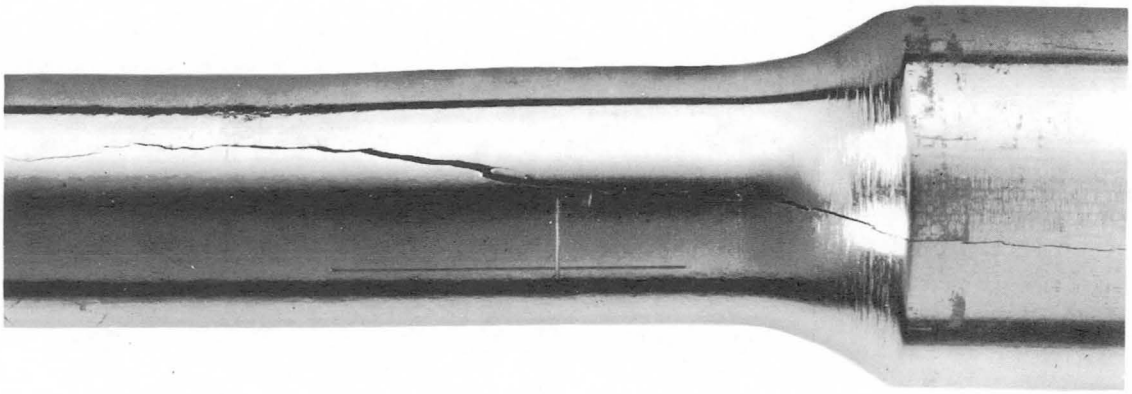


FIGURE 54 CREEP DATA TZC PLATE (HEAT M-91) TESTED IN VACUUM ENVIRONMENT $<1 \times 10^{-8}$ TORR.

OVERALL SPECIMEN APPEARANCE 1.5X



CLOSE-UP OF LONGITUDINAL CRACK 3X

FIGURE 55 TZC SPECIMEN B-22 (HEAT M-91) CRACKED DURING CREEP TESTING

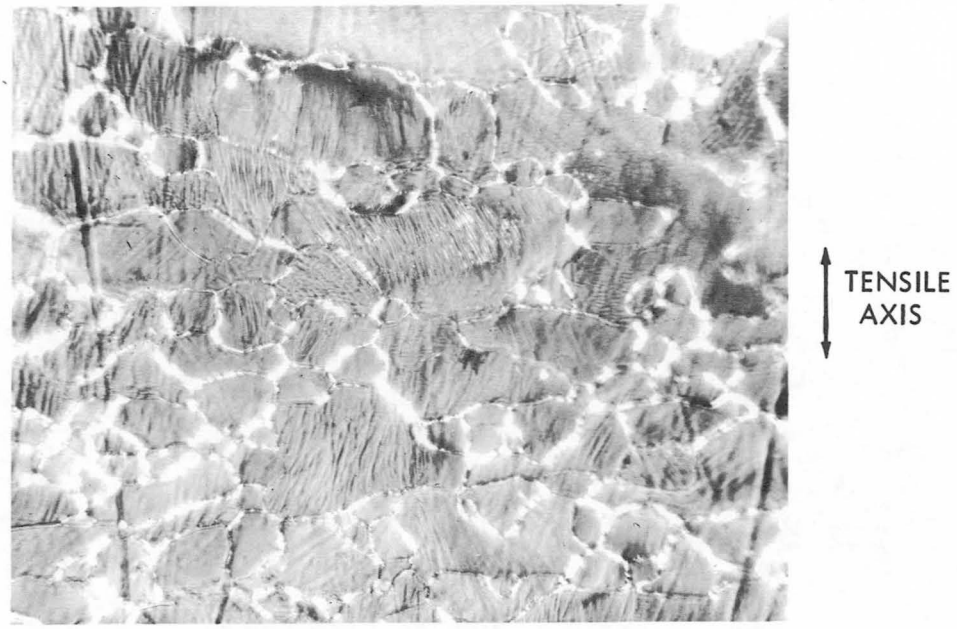


FIGURE 56 TZC SPECIMEN B-22 (HEAT M-91). MICROGRAPH SHOWS LOCAL AREA OF SLIP AS SEEN ON SURFACE OF SPECIMEN AFTER TESTING, POLARIZED LIGHT. 500X

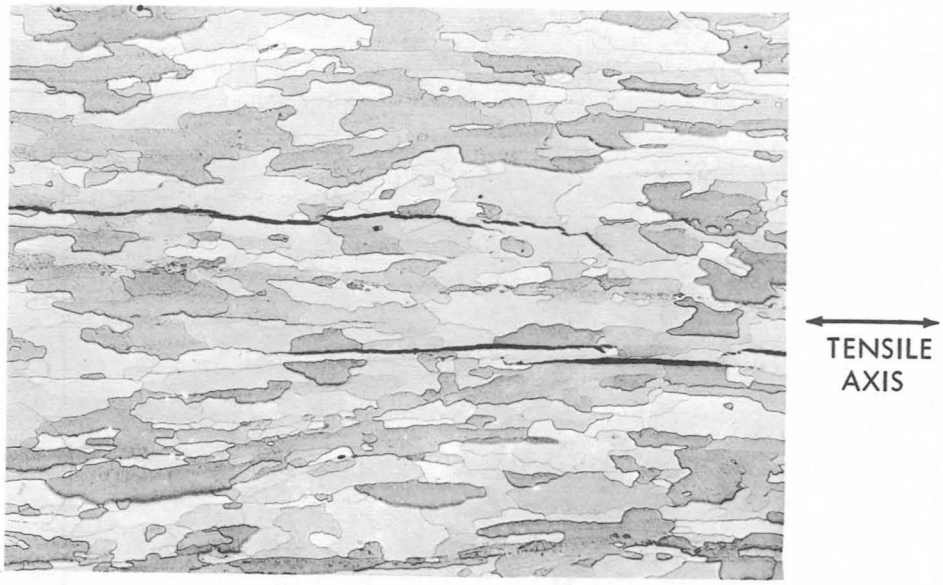
The microstructure of B-22 was examined in an effort to identify the mode of cracking. Figure 57 which shows the defect at 100 and 500X indicates that the crack was preferentially located in the grain boundaries. Since the chemistry of Heat M-91 did not exhibit any abnormalities and since cracks of a similar type were not observed in Heat M-80, it appears that the high degree of warm working applied to Heat M-91, rendered the alloy susceptible to cracking during the recrystallization operation.

Post-test examination of Heat M-91 specimens showed no significant change in the microstructure. A comprehensive hardness examination of stress relieved specimen B-28, tested for 4214 hours at 2000°F (1093°C), showed no difference in hardness between button heat (345 DPH) and gage section (342 DPH). Both of these values were not significantly different than that of the original material, 335 DPH. Specimen B-31 recrystallized at 3092°F (1700°C) and tested for 912 hours at 2200°F (1204°C) had a button head and gage hardness of 226 and 228 respectively. This is not significantly different than the alloy recrystallized and untested (240 DPH).

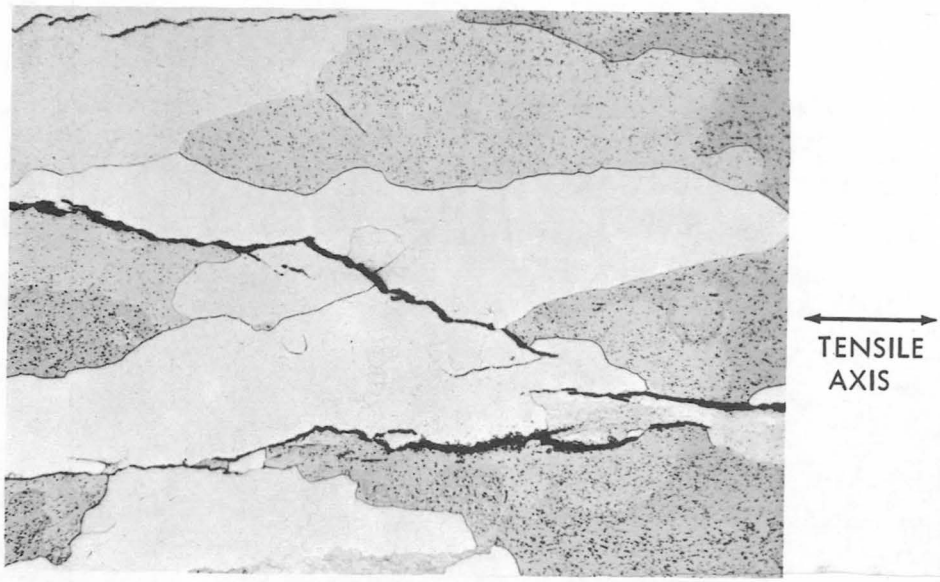
3. TZC Heat 4345

TZC Heat 4345 was received late in the program and the first specimen is on test at 2000°F (1093°C) and 22 ksi (1.52×10^8 N/m^2) (see Figure 58). The heat treatment applied prior to testing was one hour stress relief at 2500°F (1371°C).

A summary of all creep data from Heats M-80, M-91 and 4345 is given in Table 9. To compare the creep strength of the three heats, Larson-Miller and Manson-Haferd parameters were calculated for 0.5% creep and the values are listed in Table 9. The constants used in preparing the parameters were determined from Heat M-80 creep data using a computer program to obtain the parameters which would yield the best fit. The results plotted in Figure 59 show that the Heat M-80 recrystallized at 3092°F (1700°C), Heat M-91 stress relieved at 2300°F (1260°C) and Heat 4345 stress relieved at 2500°F (1371°C) have comparable creep strengths. Heat M-91 fully recrystallized by annealing one hour at 3092°F (1700°C) had comparatively the poorest creep strength.



100X



500X

FIGURE 57 PHOTOMICROGRAPHS OF CRACK IN TZC (HEAT M-91) SPECIMEN B-22. ETCHANT: 15% HF, 15% H_2SO_4, 8% HNO_3, 62% H_2O.

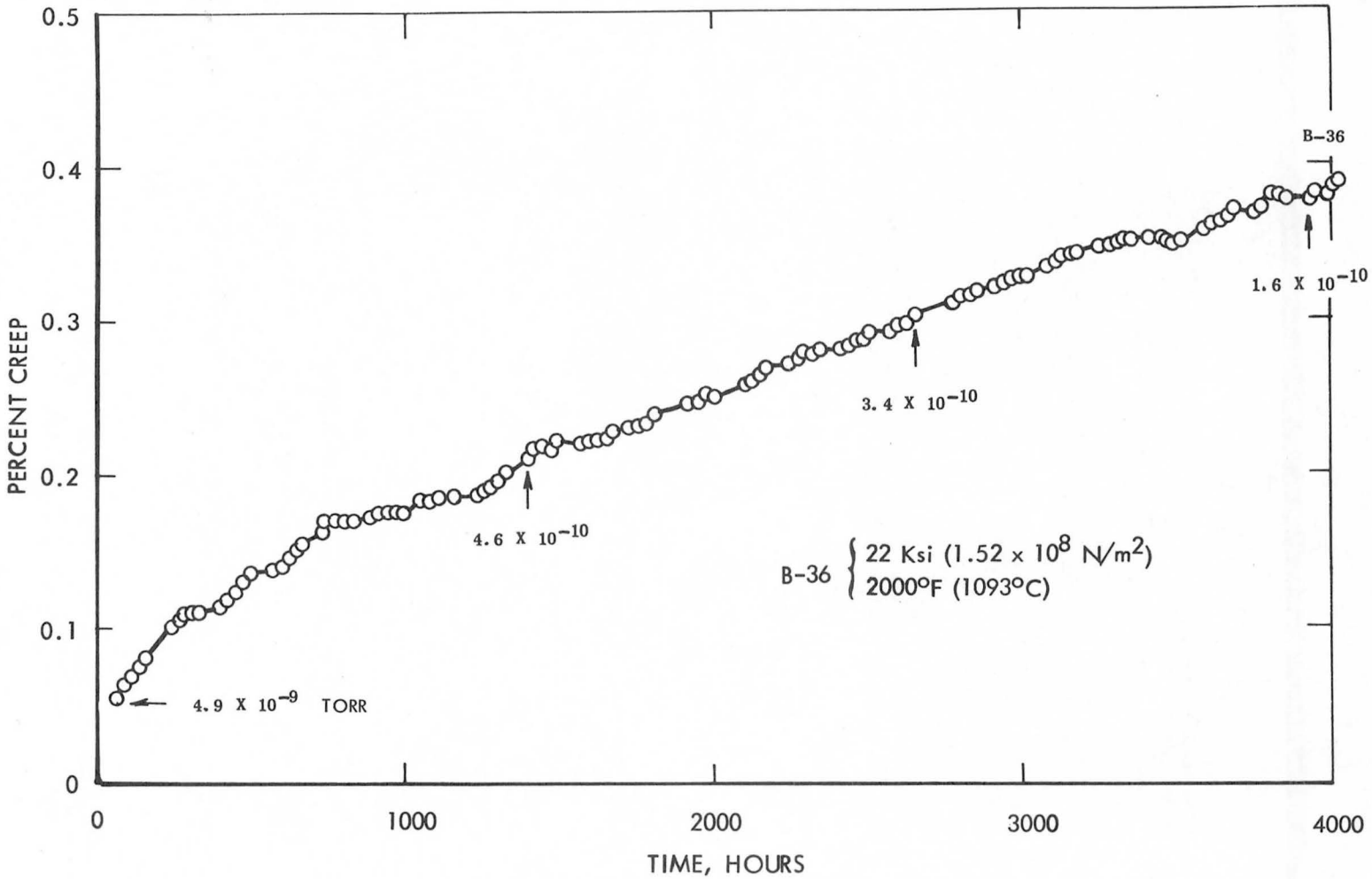


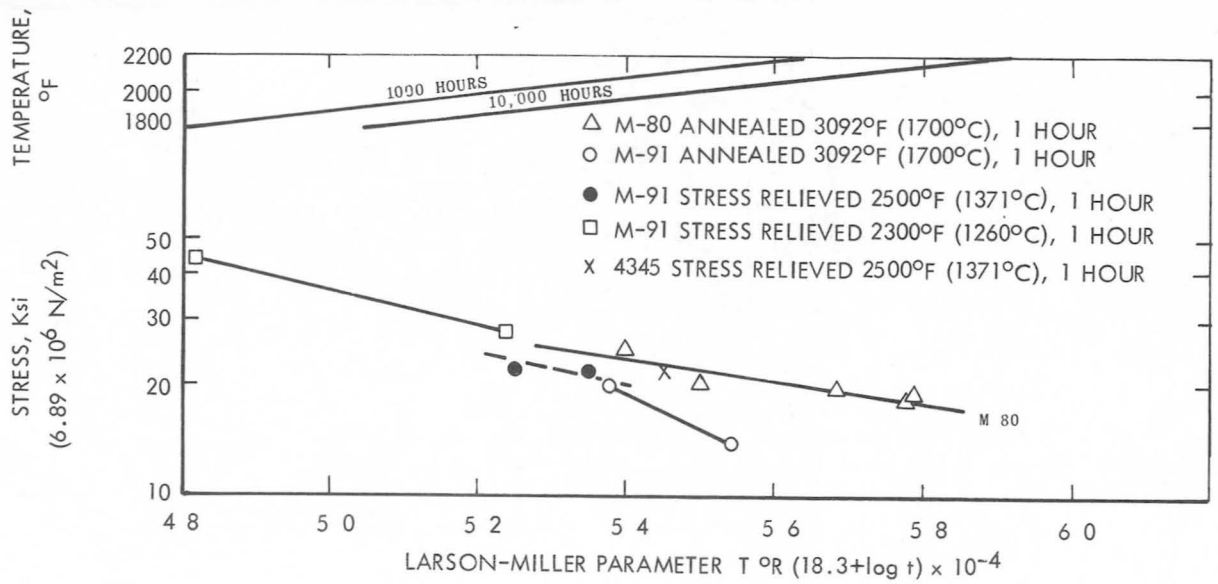
FIGURE 58 CREEP DATA FOR TZC PLATE (HEAT 4345) STRESS RELIEVED 2500°F (1371°C). TESTED IN VACUUM ENVIRONMENT $<1 \times 10^{-8}$ TORR.

TABLE 9

Summary of Creep Data for TZC

Heat No.	Specimen No.	Heat Treatment	Test Temp, °F	Stress ksi	Hours to 0.5% Creep	Larson-Miller Parameter 0.5% Creep $T°R\,(18.3+\log t)\times 10^{-3}$	Manson-Haferd Parameter 0.5% Creep $\frac{\log t - 22.8}{T}\times 10^{3}$	Creep Rate $in\text{-}in^{-1}\text{-}hr^{-1}$
M-80	B-8A	3092°F- 1 Hr.	2200	18	1,100	56.9	-9.00	4.6×10^{-6}
	B-9	3092°F- 1 Hr.	2000	20	10,400	55.0	-9.41	4.8×10^{-7}
	B-10	3092°F- 1 Hr.	2200	17	2,500	57.8	-8.84	2.0×10^{-6}
	B-11	3092°F- 1 Hr.	1856	25	120,000*	54.2	-9.57	4.2×10^{-8}
	B-12	3092°F- 1 Hr.	2056	19	46,000*	57.9	-8.84	1.1×10^{-7}
M-91	B-20	3092°F- 1 Hr.	2000	20	3,650	53.8	-9.62	1.4×10^{-6}
	B-31	3092°F- 1 Hr.	2200	14	325	55.4	-9.23	1.5×10^{-5}
	B-30	2500°F- 1 Hr.	2200	22	70	53.5	-9.52	7.2×10^{-5}
	B-32	2500°F- 1 Hr.	1935	20	14,500*	53.8	-9.64	3.4×10^{-7}
	B-33	2500°F- 1 Hr.	1900	22	8,500*	52.5	-9.93	5.9×10^{-7}
	B-19	2300°F- 1 Hr.	1800	44	1,060	48.2	-10.98	4.7×10^{-6}
	B-28	2300°F- 1 Hr.	2000	28	1,100	52.5	-9.88	4.6×10^{-6}
4345	B-36	2500°F- 1 Hr.	2000	22	7,500*	54.5	-9.96	6.7×10^{-7}

* Extrapolated Value



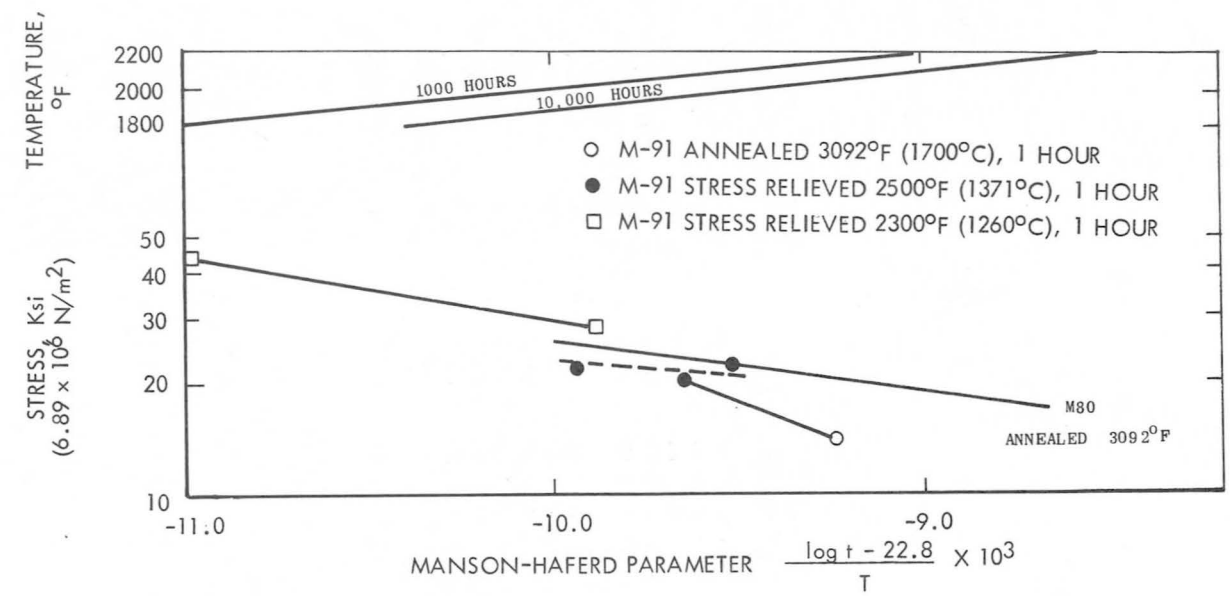


FIGURE 59 PARAMETRIC PLOTS OF 0.5% CREEP DATA FOR TZC PLATE. TESTED IN VACUUM ENVIRONMENT <1 X 10^{-8} TORR.

4. TZM Heat 7502

TZM Heat 7502 was an eleven inch diameter disc forging with radial cut specimens. All but one specimen was tested in the as-received condition, i. e., stress relieved one hour at 2200°F (1204°C). The single exception was specimen B-4 which was recrystallized for one hour at 2850°F (1566°C). The conditions evaluated are listed below:

Specimen No.	Test Temperature °F	°C	Stress ksi	N/m^2
B-1	2000	1093	12	8.27×10^7
B-3	2000	1093	10	6.89×10^7
B-4	2000	1093	10	6.89×10^7
B-29	2000	1093	41	2.82×10^8
B-35	1800	982	44	3.03×10^8

The creep data from these tests are plotted in Figures 60, 61, and 62. Specimens B-3 and B-4, Figure 60, constitute a comparison of the 2200°F (1204°C) stress relief and 2850°F (1566°C) recrystallization heat treatments. The creep curves show that the recrystallized material underwent a rapid extension of almost 0. 3% during the first 500 hours, and then exhibited a relatively constant creep rate of 8.0×10^{-8} in-in^{-1}-hr^{-1}. While the stress relieved material showed no initial extension but a greater steady state creep rate of 2.67×10^{-7} in-in^{-1}-hr^{-1}. The net effect was that the two tests reached approximately the same amount of extension after 10, 000 hours. The appearance of some negative creep in the recrystallized specimen B-4 suggests that precipitation may be occurring during testing of materials so pre-treated.

Post-test examination showed that thermal etching of the specimen occurred even at 2000°F (1093°C) and less than 650 hours, however, no significant change was noted in either the microstructure or hardness.

The test with specimen B-29 was continued to slightly more than 6% extension to evaluate the mode of creep deformation as shown in Figure 63. The deformation at 2000°F (1093°C) and 41 ksi (2.82×10^8 N/m^2) proceeds principally by transgranular slip. This is further confirmed by the absence of grain boundary slip which would be evident by localized displacement of the scribe marks.

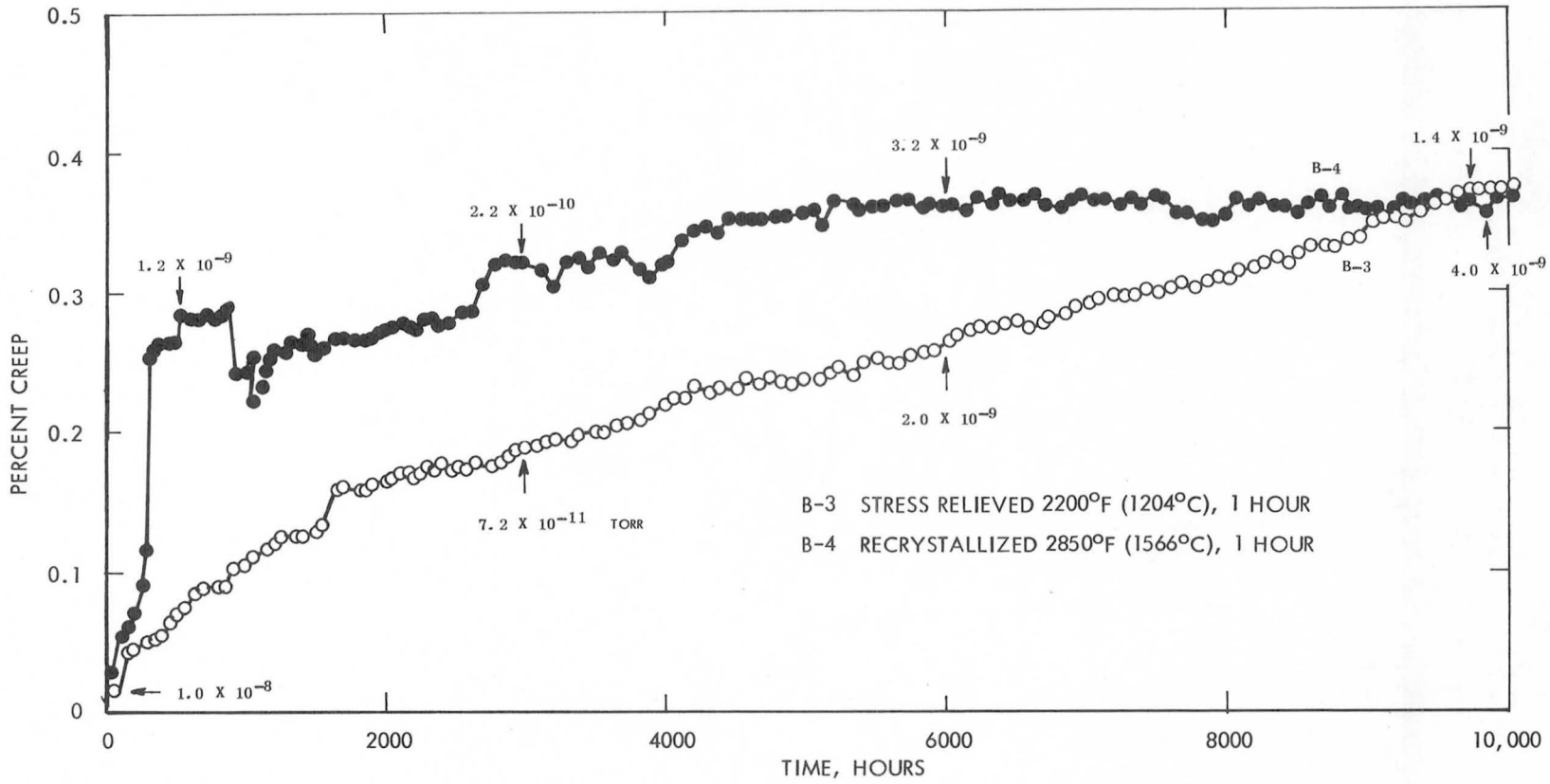


FIGURE 60 CREEP DATA TZM FORGED DISC (HEAT 7502). TESTED AT 2000°F (1093°C) AND 10 Ksi (6.89×10^7 N/m^2) IN VACUUM ENVIRONMENT $<1 \times 10^{-8}$ TORR.

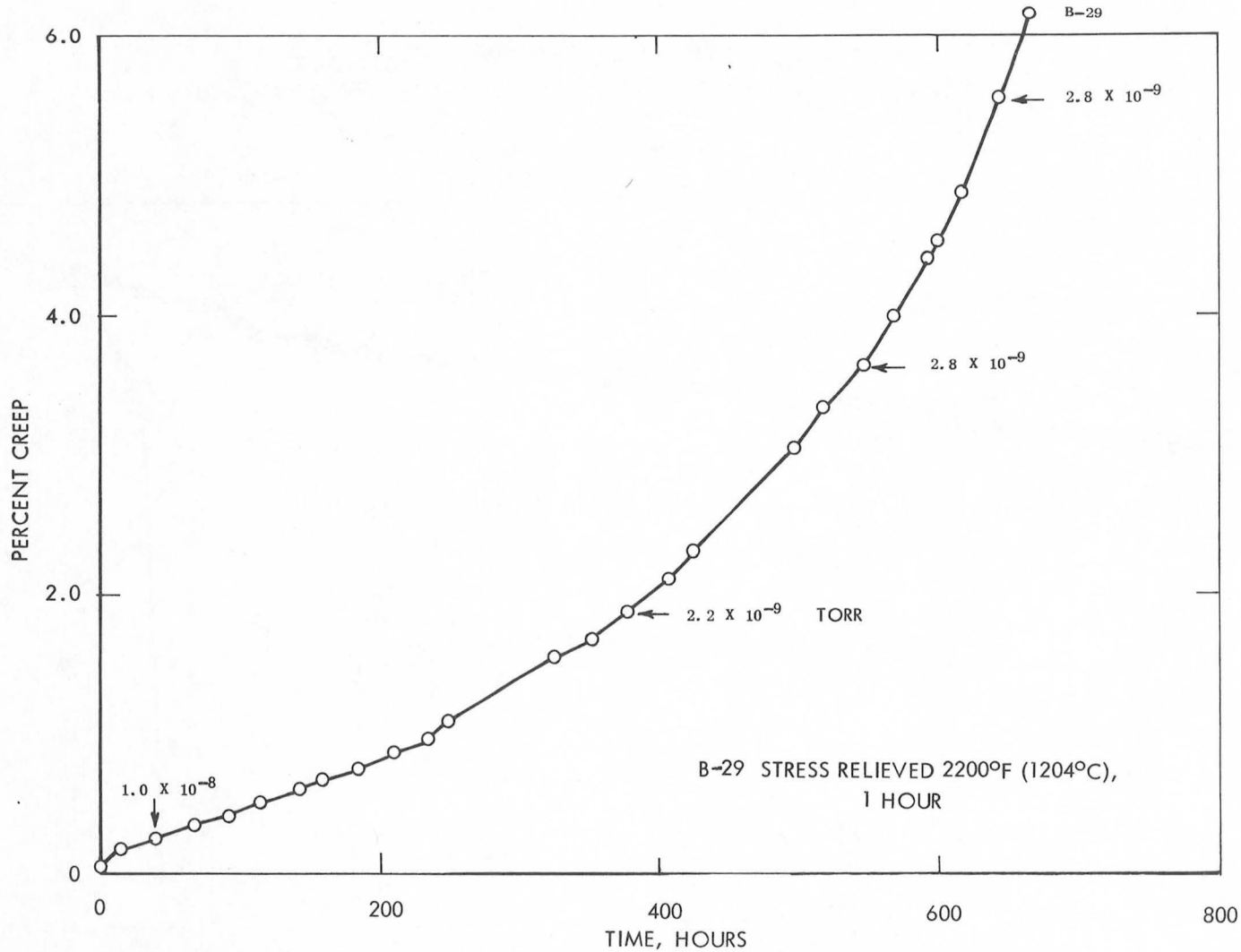


FIGURE 61 CREEP DATA TZM FORGED DISC (HEAT 7502). TESTED AT 2000°F (1093°C) AND 41 Ksi (2.82×10^8 N/m^2) IN VACUUM ENVIRONMENT $<1 \times 10^{-8}$ TORR.

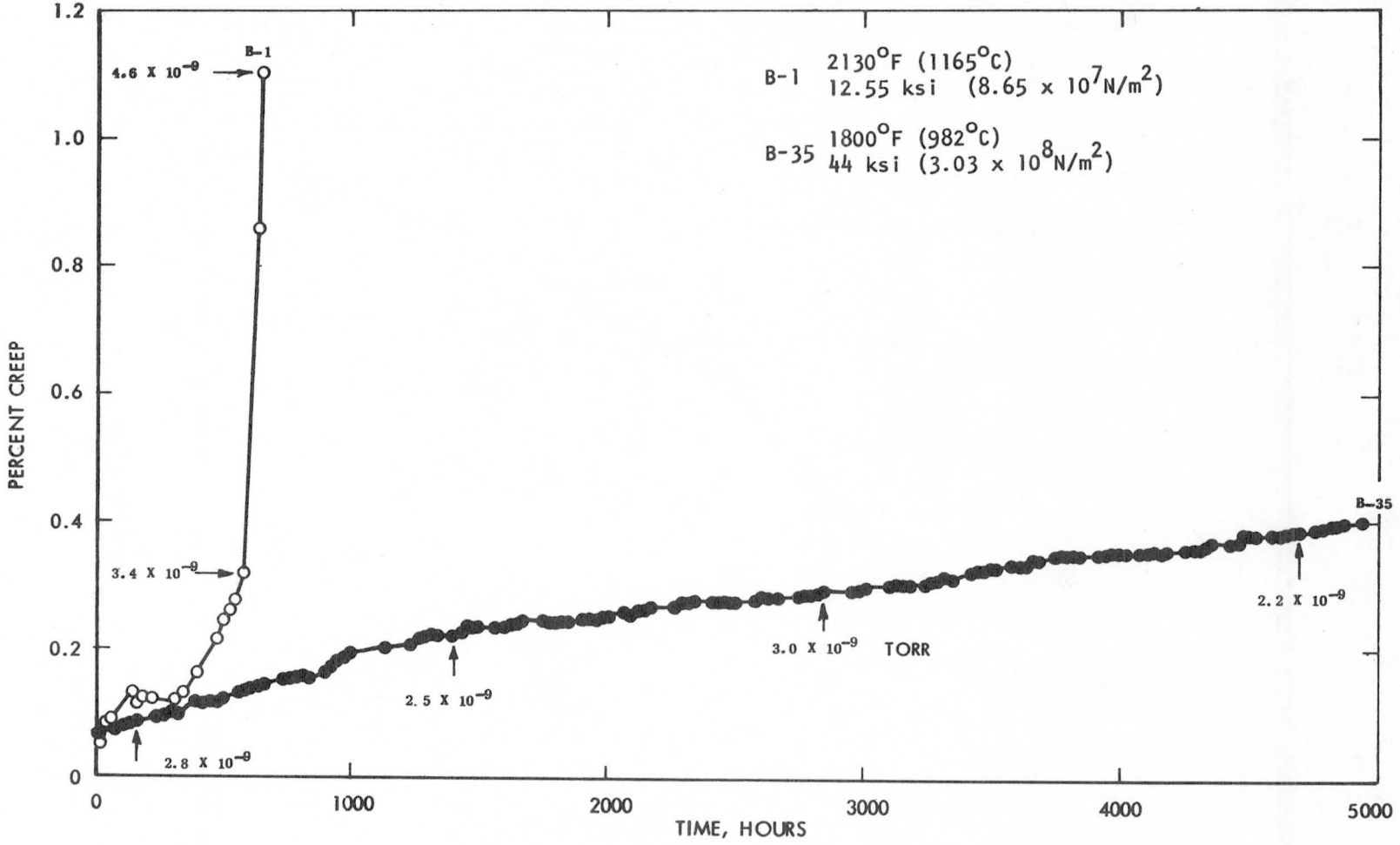
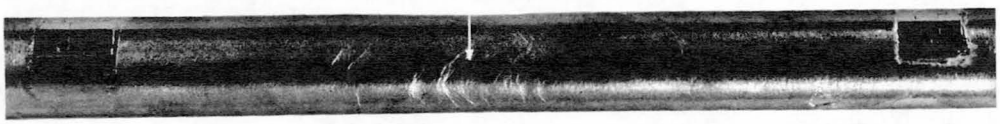


FIGURE 62 CREEP DATA TZM FORGED DISC (HEAT 7502) STRESS RELIEVED 2200°F (1204°C) ONE HOUR. TESTED IN VACUUM ENVIRONMENT $< 1 \times 10^{-8}$ TORR.

TRANS GRANULAR
SLIP LINES



2.5 X

FIGURE 63 TZM FORGED DISC (HEAT 7502) STRESS RELIEVED 2200°F (1204°C). TESTED AT 2000°F (1093°C) AND 41 Ksi IN VACUUM ENVIRONMENT $<1 \times 10^{-8}$ TORR. 6.215% CREEP OBTAINED IN 664.3 HOURS. NOTE THE LINES INDICATING TRANSGRANULAR SLIP.

5. TZM Heat KDTZM-1175

Four tests, listed below, were made using Heat KDTZM-1175 in the as-received condition, i. e. stress relieved at 2300°F (1260°C) for one hour.

Specimen No.	Test Temperature °F	°C	Stress ksi	N/m^2
B-16	1855	1013	23.4	1.61×10^8
B-18	1600	871	55	3.79×10^8
B-21	1600	871	65	4.48×10^8
B-25	1800	982	44	3.03×10^8

The preparation of specimens of this heat were different in that all were electro-polished using Globe Chemical Co. electropolishing solution No. 4 in a lead-lined pyrex container with a C. P. Lead cathode. Polishing was accomplished with a current density of 10 amps/in^2 at 14 volts.

The creep data for the four tests, plotted in Figure 64, show that at 1600°F (871°C) essentially no creep occurred even at stresses as high as 65 ksi (4.48×10^8 N/m^2).

Post-test examination of specimens from Heat KDTZM-1175 indicated that no significant changes in either the microstructure or hardness occurred as a result of the test exposure.

6. TZM Heat 7463 (Commercial Bar)

A single specimen from 5/8 inch diameter commercial TZM bar, Heat 7463, was tested in the as-received condition, i. e. stress relieved at 2250°F (1232°C) for 1/2 hour. The data obtained for the test temperature of 2000°F (1093°C) and a stress of 41 ksi ($2.82 \times 10^8 N/m^2$) are plotted in Figure 65.

A summary of creep data from TZM Heats 7502, KDTZM-1175, and 7463 is given in Table 10. Also included are the times for 0.5% creep, the creep rate and the Larson-Miller parameter based on a constant of 20.1 determined by computer procedures. The parametric comparison made in Figure 66 shows thats Heats KDTZM-1175 and 7463 have greater creep strengths than Heat 7502. Since the chemistry of Heats 7502 and 7463 are essentially the same, it is concluded that the method of processing the molybdenum-base TZM bar alloy has a significant effect on the creep strength.

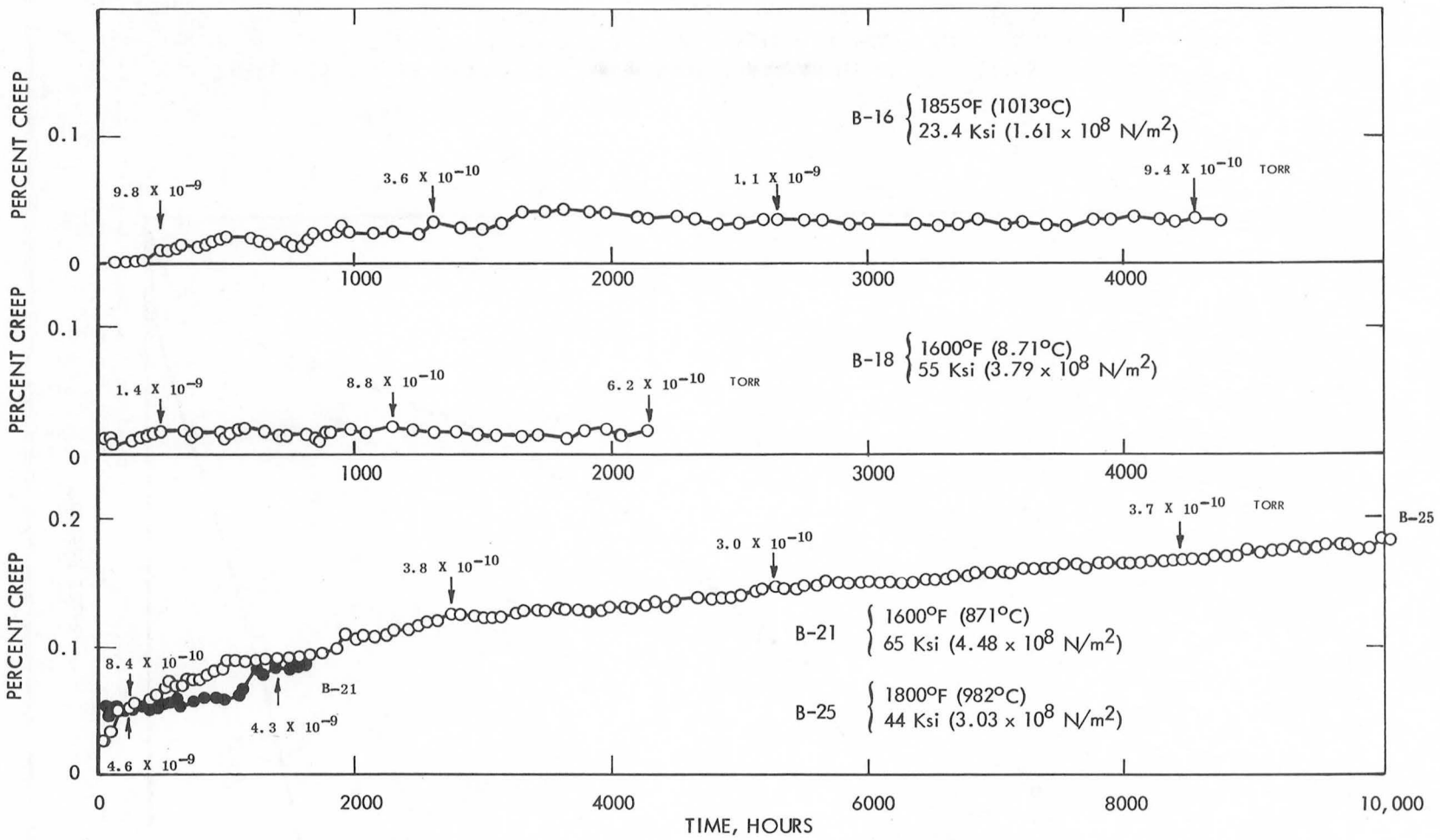


FIGURE 64 CREEP DATA TZM FORGED DISC (HEAT KDTZM-1175) STRESS RELIEVED 2300°F (1260°C) ONE HOUR. TESTED IN VACUUM ENVIRONMENT $< 1 \times 10^{-8}$ TORR.

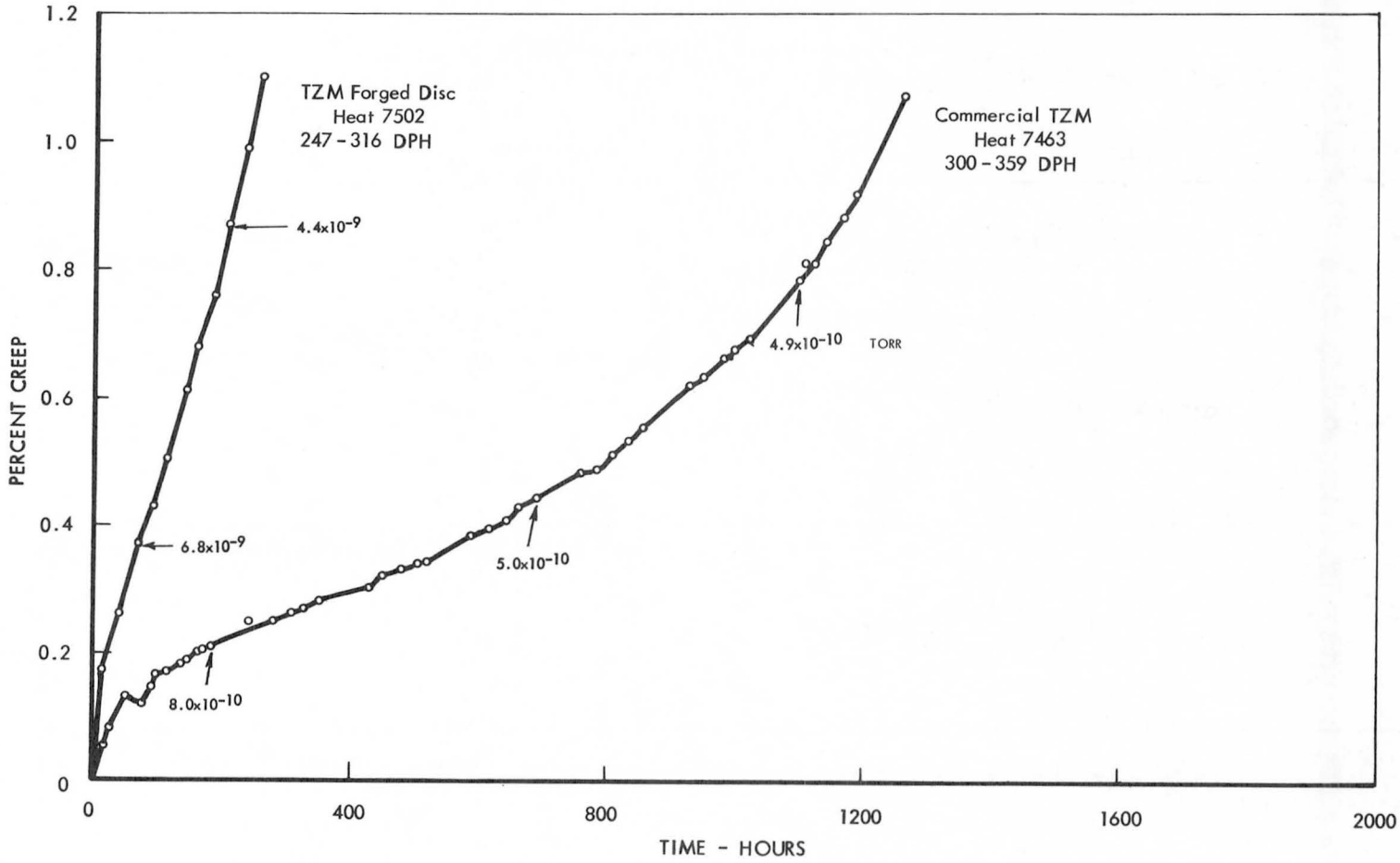


FIGURE 65 CREEP DATA FOR TZM COMMERCIAL BAR (HEAT 7463) STRESS RELIEVED AT 2250°F (1232°C) 1/2 HOUR. TESTED AT 2000°F (1093°C) AND 41 Ksi (2.82×10^8 N/m^2) IN VACUUM ENVIRONMENT $< 1 \times 10^{-8}$ TORR.

TABLE 10

Summary of Creep Data for TZM

Heat	Specimen No.	Heat Treatment	Test Temperature °F	Test Temperature °C	Stress ksi	Stress N/m^2	Hours to 0.5% Creep	Larson-Miller Parameter 0.5% Creep $T^{\circ}R(20.1+\log t) \times 10^{-3}$	Creep Rate $in\text{-}in^{-1}\text{-}hr^{-1}$
7502	B-1	2200°F - 1 Hr.	2130	1165	12.55	8.65×10^7	605	59.3	8.3×10^{-6}
	B-3	2200°F - 1 Hr.	2000	1093	10	6.89×10^7	14,200*	59.6	3.5×10^{-7}
	B-4	2850°F - 1 Hr.	2000	1093	10	6.89×10^7	25,000*	60.3	2.0×10^{-7}
	B-29	2200°F - 1 Hr.	2000	1093	41	2.82×10^8	115	54.5	4.4×10^{-5}
	B-35	2200°F - 1 Hr.	1800	982	44	3.03×10^8	7,000*	54.1	7.1×10^{-7}
1175	B-16	2300°F - 1 Hr.	1855	1013	23.4	$1.6. \times 10^8$	62,500*	57.5	8.0×10^{-8}
	B-18	2300°F - 1 Hr.	1600	871	55	3.79×10^8	60,000*	51.3	8.3×10^{-8}
	B-21	2300°F - 1 Hr.	1600	871	65	4.48×10^8	9,600*	49.6	5.2×10^{-8}
	B-25	2300°F - 1 Hr.	1800	982	44	3.03×10^8	50,000*	56.0	1.0×10^{-7}
7463	B-34	2250°F - ½ Hr.	2000	1093	41	2.82×10^8	800	56.5	6.2×10^{-6}

* Extrapolated Values

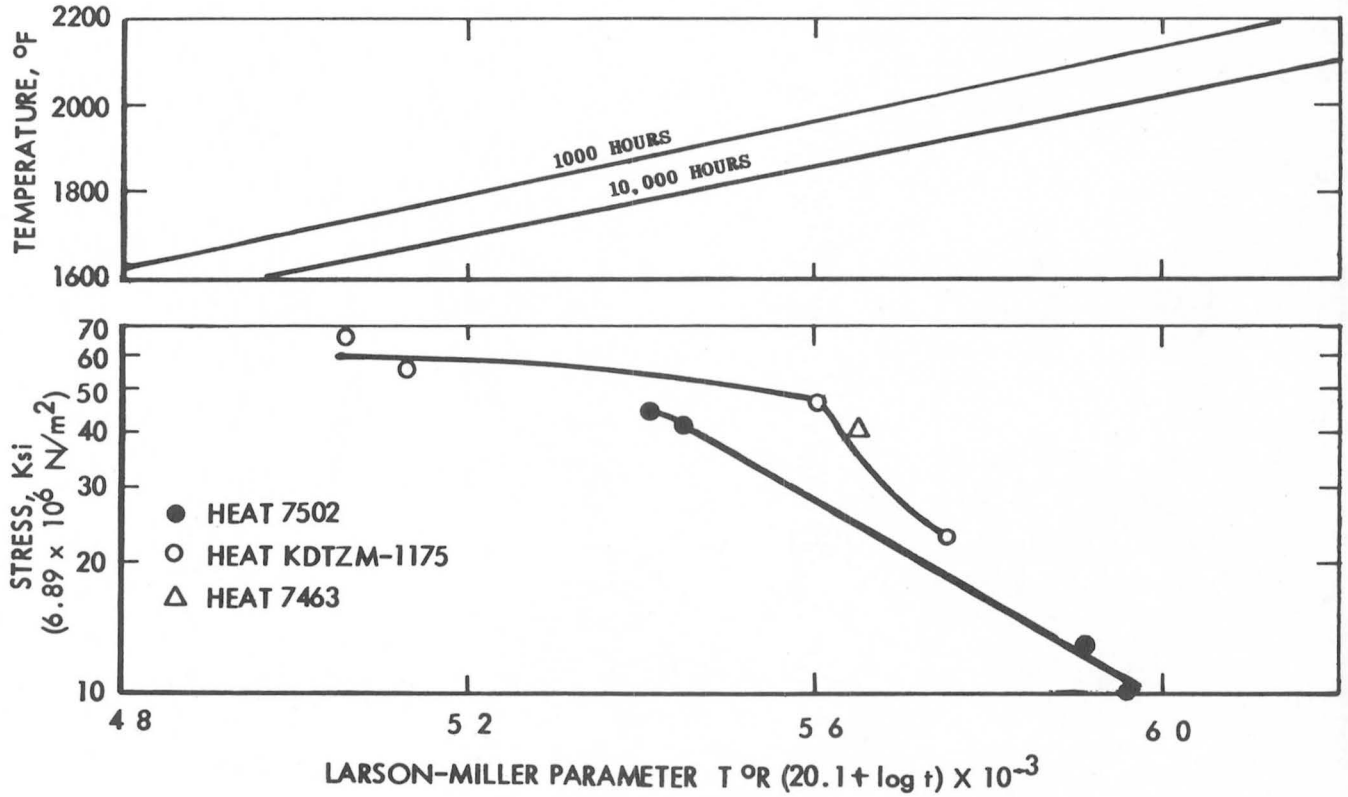


FIGURE 66 LARSON-MILLER PLOT OF 0.5% CREEP DATA FOR TZM STRESS RELIEVED 2200-2300°F (1204-1260°C). TESTED IN VACUUM ENVIRONMENT $< 1 \times 10^{-8}$ TORR.

7. Columbium Modified TZM Heat 4305-4

Two specimens of columbium-modified TZM Heat 4305-4 were tested in the as-received state, i.e., stress relieved one hour at 2500°F (1371°C).

Specimen No.	Test Temperature °F	°C	Stress ksi	N/m^2	Time Hours	%Creep
B-23A	2000	1093	20	1.38×10^8	686	0.032
B-23B	2000	1093	28	1.93×10^8	307	0.028
B-23C	2000	1093	40	2.76×10^8	185	0.188
B-23D	1800	982	46	3.17×10^8	403	0.078
B-23E	2100	1149	34	2.34×10^8	329	0.170
B-27	2000	1093	41	2.82×10^8	1587	1.040

The purpose of the first five tests made on the same specimen (B23A-E) was to accumulate enough data to estimate the time required for 0.5% creep. From this an approximate parametric creep curve was obtained with one specimen. Because of the short times involved, the creep data for these tests are not plotted; however, the creep curve for specimen B-27 tested at 2000°F (1093°C) and 41 ksi ($2.82 \times 10^8 N/m^2$) is plotted in Figure 67. Included with Figure 67 are the creep curves for TZM Heats 7502 and 7463 tested under identical conditions. Although a significant part of the improved creep strength of the columbium modified TZM is due to the method of processing, the comparison with the data obtained from the TZM bar stock indicates that the columbium addition improved creep resistance.

Table 11 and Figure 68 show the Larson-Miller creep data based on the general constant of 15. The curve should be used with some discretion, since it is approximate and based on extrapolated values obtained from a series of tests on a single specimen.

D. Tantalum-Base Alloys

1. T-222 Heat AL-TA-43

Three tests were made with T-222 Heat AL-TA-43 using the two heat treatments shown below. The creep data plotted in Figures 69 and 70 show that the higher recrystallization temperature may provide better creep strength.

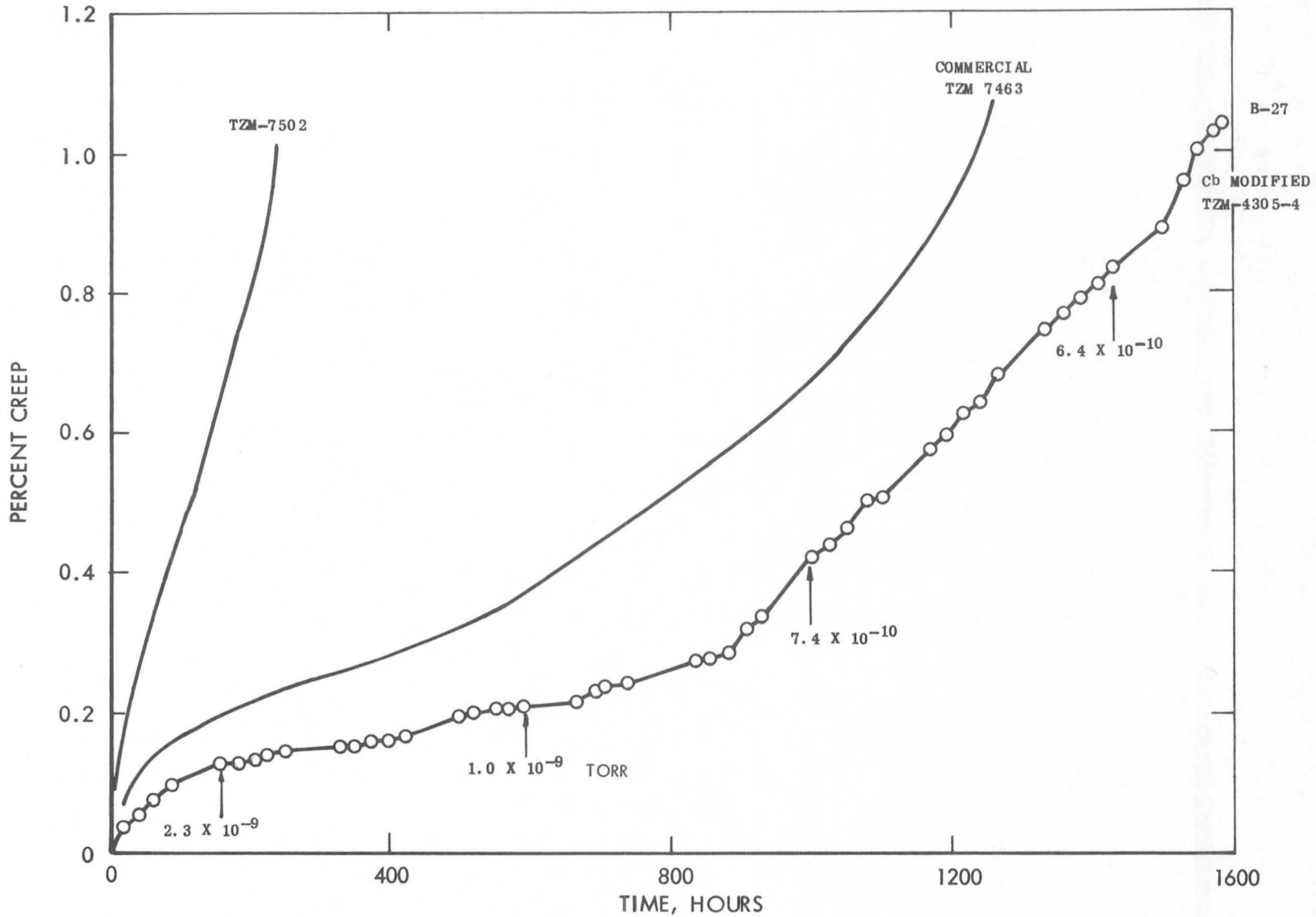


FIGURE 67 CREEP DATA FOR COLUMBIUM MODIFIED TZM (HEAT 4305-4). TESTED AT 2000°F (1093°C) AND 41 Ksi (2.82×10^8 N/m^2) IN VACUUM ENVIRONMENT $< 1 \times 10^{-8}$ TORR.

TABLE 11

Summary of Creep Data for Columbium-Modified TZM Heat 7503-4

Specimen No.	Test Temperature °F	°C	Stress ksi	N/m^2	Hours to 0.5% Creep	Larson-Miller Parameter 0.5% Creep $T°R\ (15.0+\log t) \times 10^{-3}$
B-23A	2000	1093	20	1.38×10^8	20,000*	47.5
B-23B	2000	1093	28	1.93×10^8	10,000*	46.7
B-23C	2000	1093	40	2.76×10^8	630*	43.8
B-23D	1800	982	46	3.17×10^8	4,000*	42.0
B-23E	2100	1149	34	2.34×10^8	1,000*	46.1
B-27	2000	1093	41	2.82×10^8	1,090	44.5

* Extrapolated Values

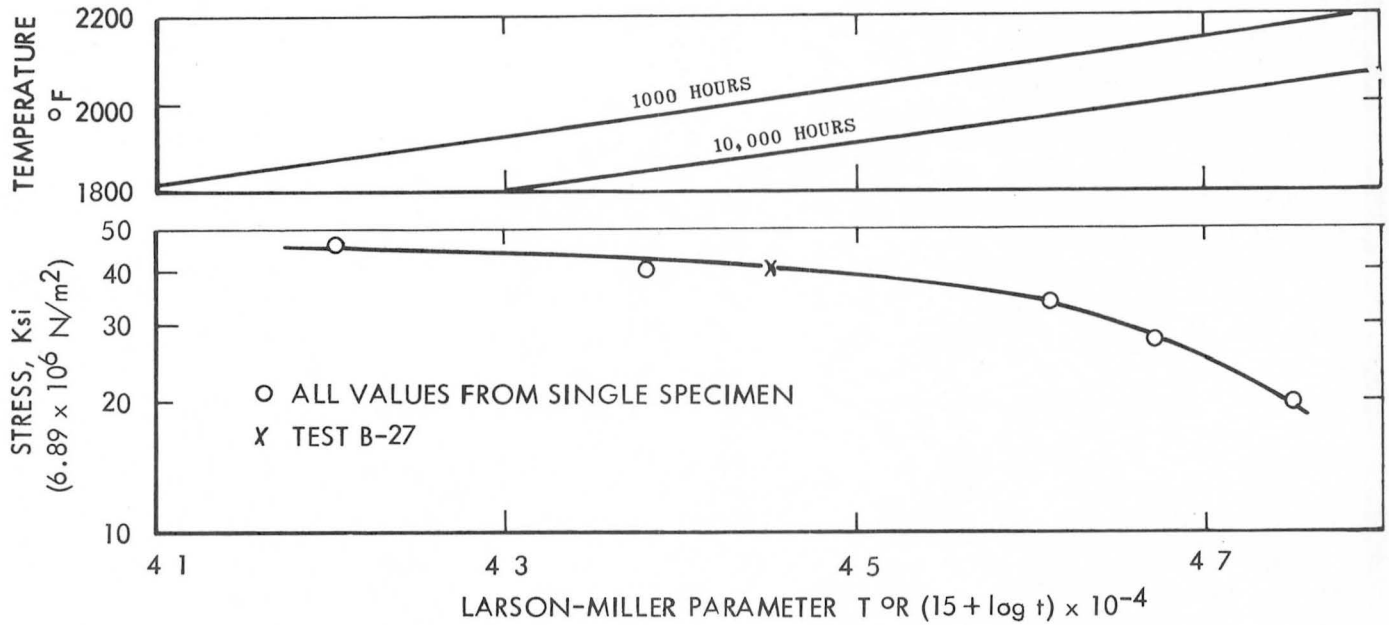


FIGURE 68 LARSON-MILLER PLOT OF 0.5% CREEP DATA FOR COLUMBIUM MODIFIED TZM (HEAT 7503-4) STRESS RELIEVED 2500°F (1371°C) ONE HOUR. TESTED IN VACUUM ENVIRONMENT $<1 \times 10^{-8}$ TORR.

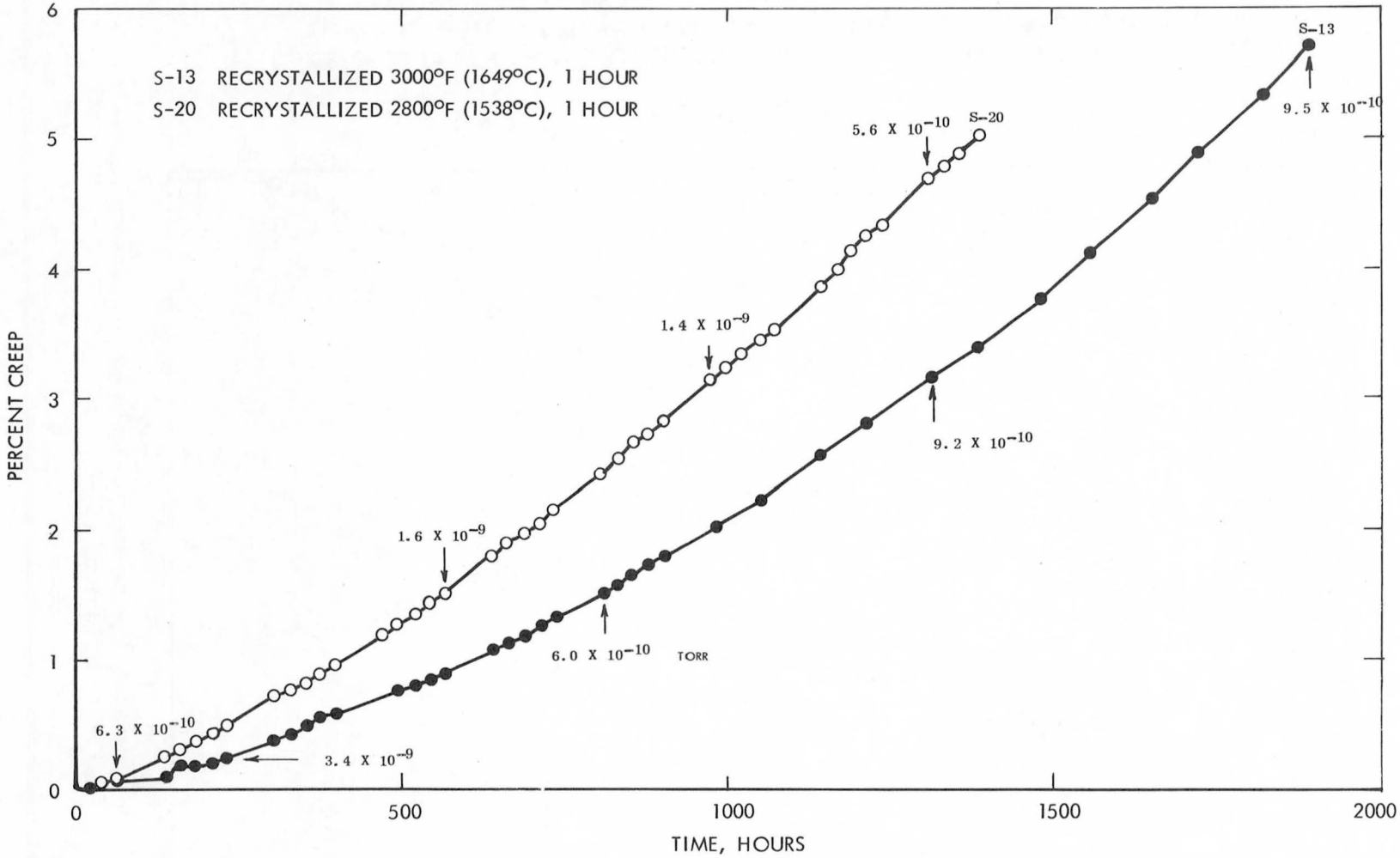


FIGURE 69 CREEP DATA FOR T-222 SHEET (HEAT AL-TA-43). TESTED AT 2200°F (1204°C) AND 12 Ksi (8.26×10^7 N/m^2) IN VACUUM ENVIRONMENT $<1 \times 10^{-8}$ TORR.

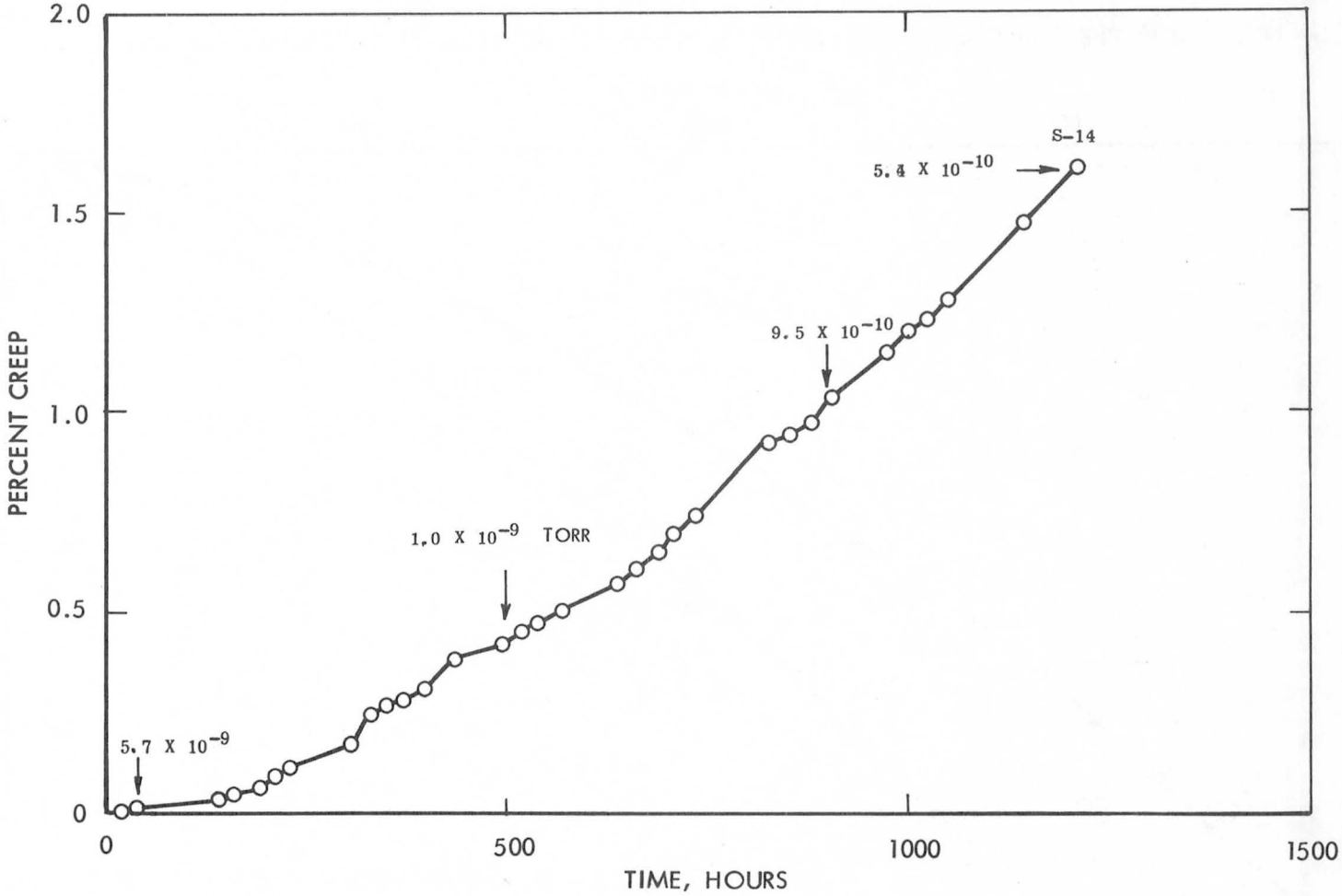


FIGURE 70 CREEP DATA FOR T-222 SHEET (HEAT AL-TA-43) RECRYSTALLIZED ONE HOUR, 3000°F (1649°C). TESTED AT 2056°F (1124°C) AND 19.2 Ksi (1.32×10^8 N/m^2) IN VACUUM ENVIRONMENT $<1 \times 10^{-8}$ TORR.

Specimen No.	Heat Treatment °F	°C	Hrs.	Test Temperature °F	°C	Stress ksi	N/m^2
S-13	3000	1649	1	2200	1204	12	8.26×10^7
S-14	3000	1649	1	2056	1124	19.2	1.32×10^8
S-20	2800	1538	1	2200	1204	12	8.26×10^7

Post-test examinations of the specimens showed that the scribe marks used for creep measurement had been displaced at the thermally etched grain boundaries (see Figure 71). Under polarized light, fine slip lines could be found within the grains of the same area, Figure 72. These observations indicate that creep proceeds in this test by grain boundary sliding as well as by transgranular slip.

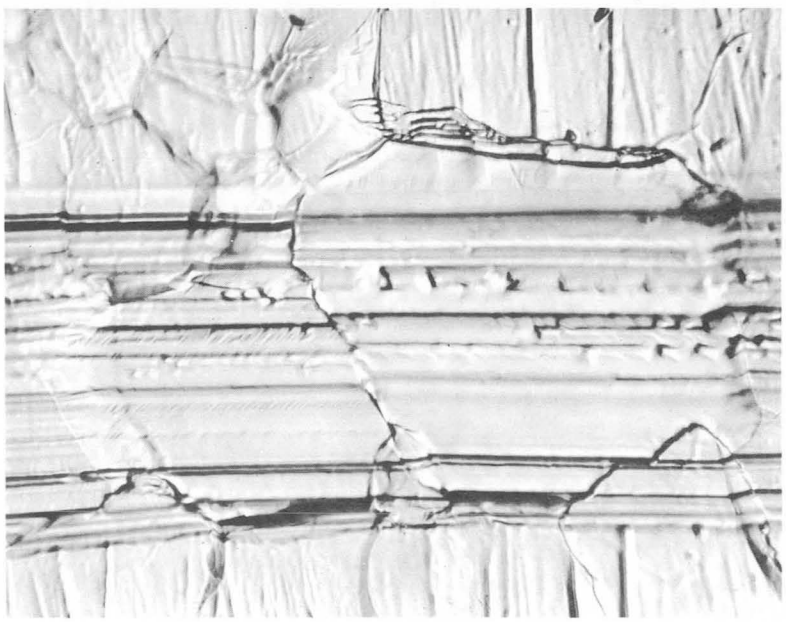
T-222 Heat AL-TA-43 after recrystallization for one hour at 3000°F (1649°C) had a hardness of 357 DPH. Specimen S-13 after testing for 1890 hours at 2200°F (1204°C) had a hardness of 236 DPH indicating that softening took place during testing. Specimen S-14 also exhibited a hardness decrease to 264 DPH after 3229 hours at a lower test temperature of 2056°F (1124°C).

2. T-111 Heat 70616

Several heats of tantalum-base T-111 were tested with Heat 70616 being the most extensively investigated.

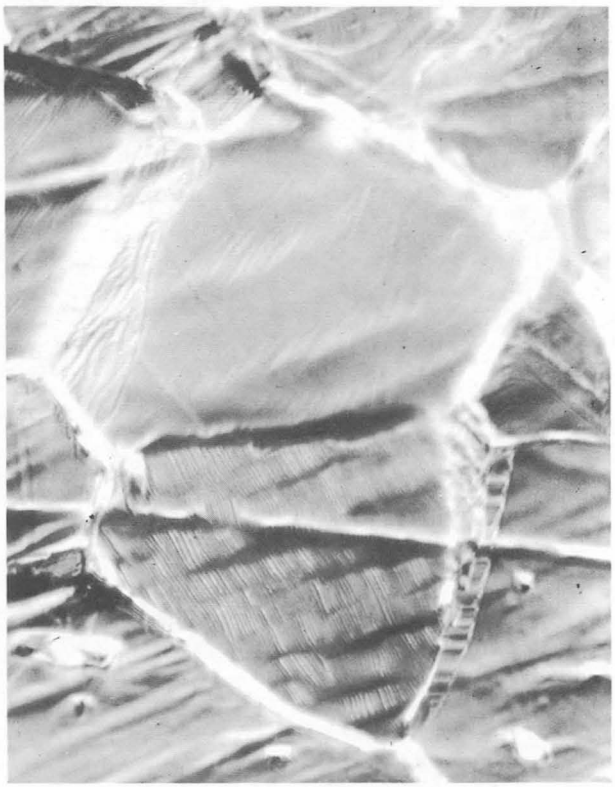
Specimen No.	Heat Treatment °F	°C	Hrs.	Test Temperature °F	°C	Stress ksi	N/m^2
S-16	2600	1427	1	2200	1204	8	5.15×10^7
S-19	3000	1649	1	2200	1204	8	5.15×10^7
S-21	3000	1649	1	2200	1204	12	8.26×10^7
S-22	3000	1649	1	2000	1093	20	1.38×10^8
S-23	3000	1649	1	2120	1160	12	8.26×10^7
S-24	3000	1649	1	1860	1016	20	1.38×10^8

The first two tests, S-16 and S-19, were made to determine the effect of the recrystallization temperature on the creep strength of the alloy. The creep data for the specimens recrystallized at 2600°F (1427°C) and 3000°F (1649°C) are plotted in Figure 73. The results clearly show that the higher recrystallization temperature provided the better creep strength. The two tests at 12 ksi ($8.26 \times 10^7 N/m^2$) are plotted in Figure 74 and those at 20 ksi ($1.38 \times 10^8 N/m^2$) are shown in Figure 75. It will be noted in the three figures that all curves for the material recrystallized at 3000°F (1649°C) exhibit a pronounced upward curvature.



750 X

FIGURE 71 SURFACE OF T-222 SHEET SPECIMEN AFTER TESTING AT 2200°F (1204°C), 12 Ksi ($8.26 \times 10^7 N/m^2$) FOR 1890 HOURS. 5.72% TOTAL EXTENSION. PHOTOMICROGRAPH SHOWS GRAIN BOUNDARY SLIDING. THERMALLY ETCHED SURFACE°



1000 X

FIGURE 72 SURFACE OF T-222 SHEET SPECIMEN AFTER TESTING AT 2200°F (1204°C), 12 Ksi ($8.26 \times 10^7 N/m^2$) FOR 1890 HOURS. 5.72% TOTAL EXTENSION. PHOTOMICROGRAPH ILLUSTRATES FINE SLIP PRESENT IN THE GRAIN. POLARIZED LIGHT.

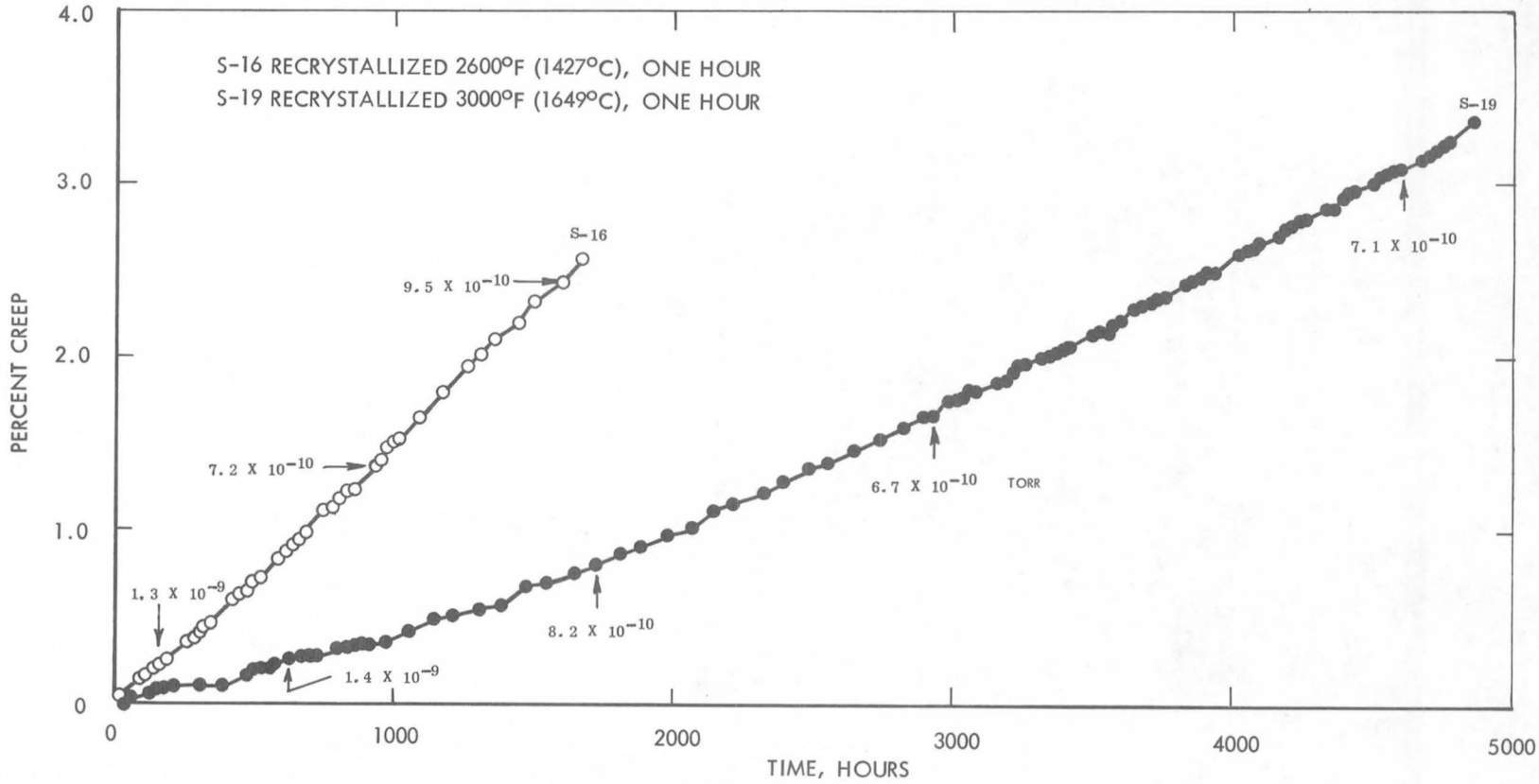


FIGURE 73 CREEP DATA FOR T-111 SHEET (HEAT 70616) TESTED AT 2200°F (1204°C) AND 8 Ksi (5.51×10^7 N/m^2) IN VACUUM ENVIRONMENT $< 1 \times 10^{-8}$ TORR.

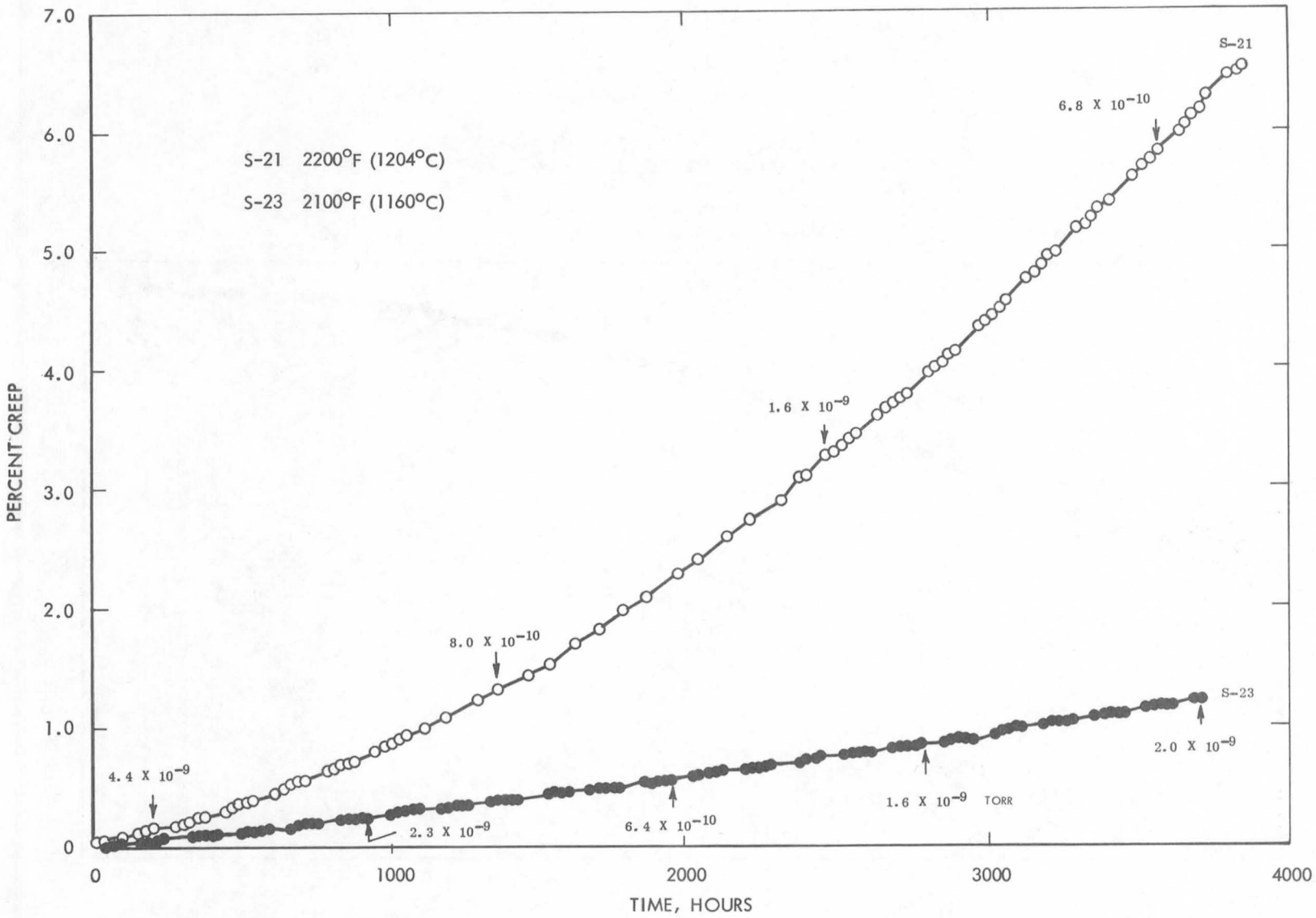


FIGURE 74 CREEP DATA FOR T-111 SHEET (HEAT 70616). TESTED AT 12 Ksi (8.26×10^7 N/m^2) IN VACUUM ENVIRONMENT $< 1 \times 10^{-8}$ TORR.

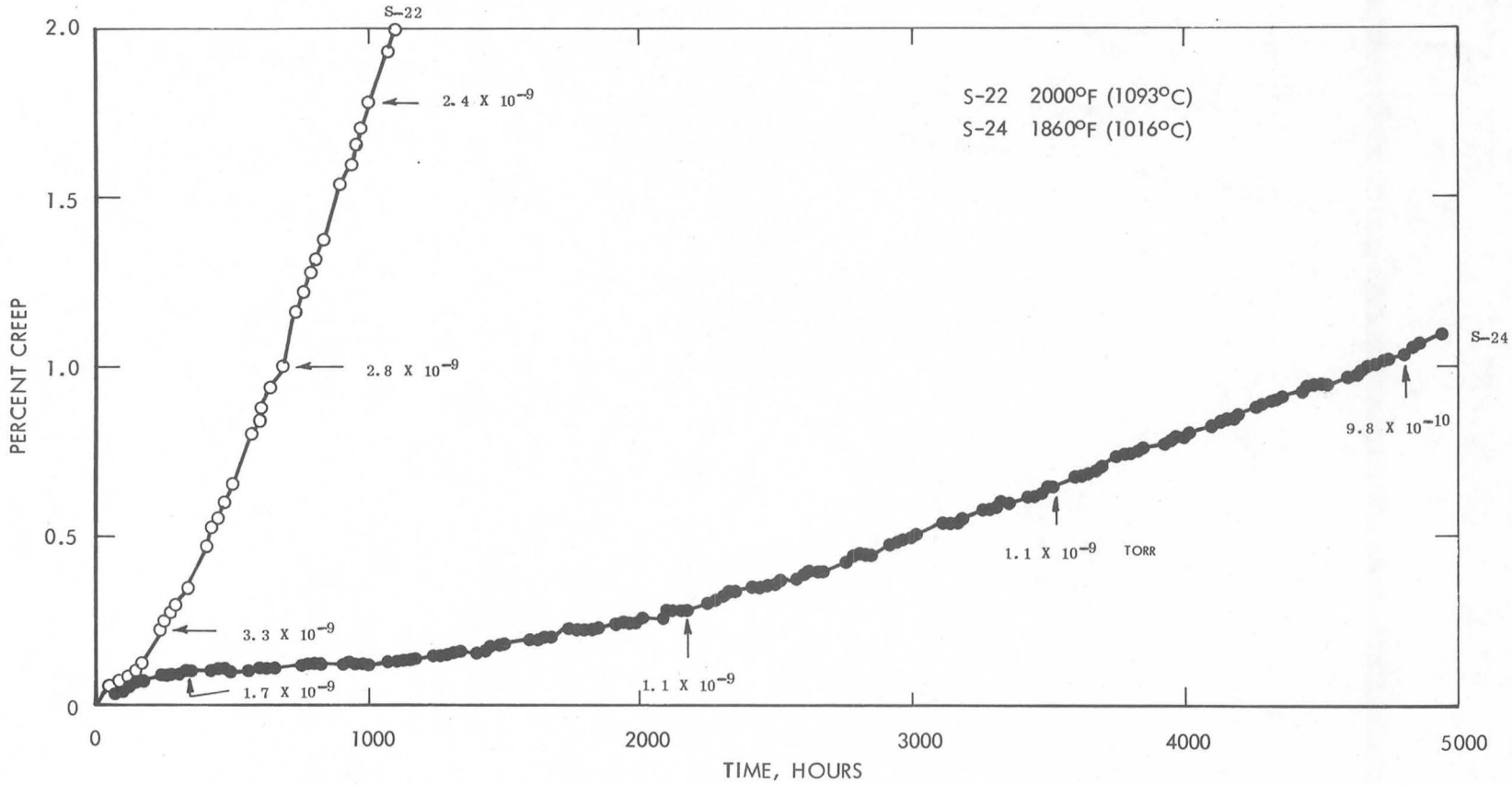


FIGURE 75 CREEP DATA FOR T-111 SHEET (HEAT 70616). TESTED AT 20 Ksi (1.38×10^8 N/m^2) IN VACUUM ENVIRONMENT $<1 \times 10^{-8}$ TORR.

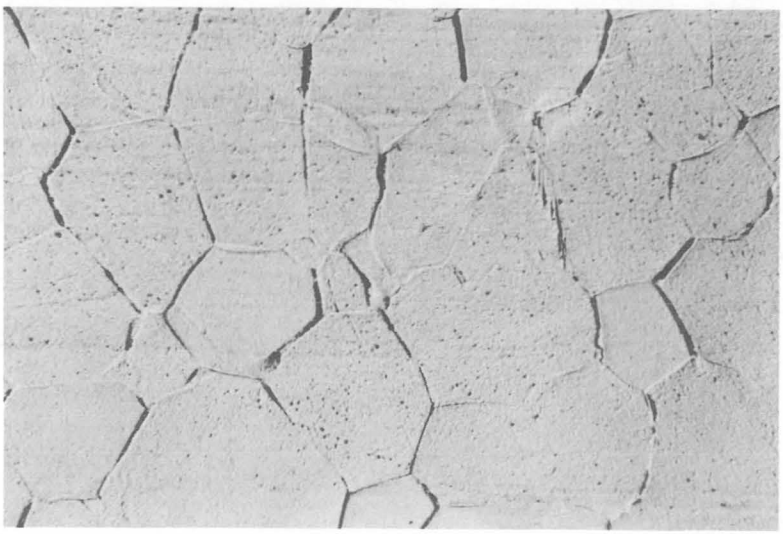
Post-test metallography of the specimens tested to 3-6% extension showed the presence of small grain boundary manifestations which appeared to be voids (Figure 76). However, an electron microprobe analysis on Specimen S-19, tested to 3% elongation at 2200°F (1204°C) indicated these areas to be rich in hafnium (Figure 77). Similar areas could not be detected in the untested material. Titran and Hall (14) observed a similar structure and interpreted it as a grain boundary precipitate. The true nature of the manifestation is thus not clear at the present time. Examination of the specimens after testing showed the presence of both grain boundary and transgranular slip as was observed with T-222 alloy. Post-test hardness measurements of T-111 Heat 70616 showed that prolonged heating during test caused the hardness of the original recrystallized material (260-287 DPH) to decrease to 226-230 DPH.

3. T-111 Heat 65079

Three tests of Heat 65079 were conducted with the specimens being taken both parallel and perpendicular to the direction of rolling. All specimens were recrystallized at 3000°F (1649°C) for one hour prior to testing. Two tests were made at the same temperature and stress to determine whether the creep strength varied as a function of orientation even though there was no difference in tensile properties in the two directions.

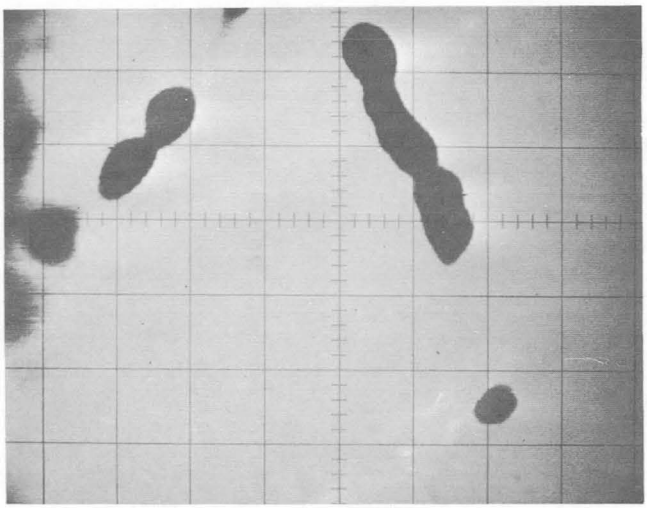
Specimen No.	Test Temperature °F	°C	Stress ksi	N/m^2	Axis of Specimen
S-30	2400	1316	3.5	2.41×10^7	Perpendicular to rolling
S-31	2200	1204	5	3.44×10^7	Perpendicular to rolling
S-35	2200	1204	5	3.44×10^7	Parallel to rolling

The creep data for the two specimens S-31 and S-35 tested at 2200°F (1204°C) and 5 ksi (3.44×10^7 N/m^2) are shown in Figure 78. While these tests are still in progress a small difference in creep resistance can be observed with the creep strength of the sample perpendicular to rolling direction being slightly greater than that parallel to rolling. The creep curve for the specimen tested at 2400°F (1316°C) is plotted in Figure 79.

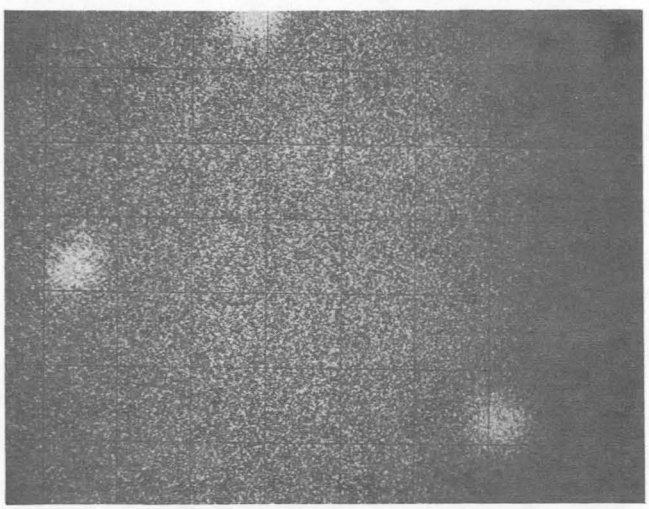


500 X

FIGURE 76 T-111 SHEET SPECIMEN S-21, TESTED AT 2200°F AND 12 Ksi 3840 HOURS. 6.55% CREEP. ETCHANT: 75% HF, 25% H_2O.



SPECIMEN CURRENT IMAGE
2400 X



Hf X-RAY IMAGE
2400X

FIGURE 77 ELECTRON PROBE X-RAY MICROGRAPH OF T-111 SHEET SPECIMEN S-19 SHOWING HAFNIUM CONCENTRATION.

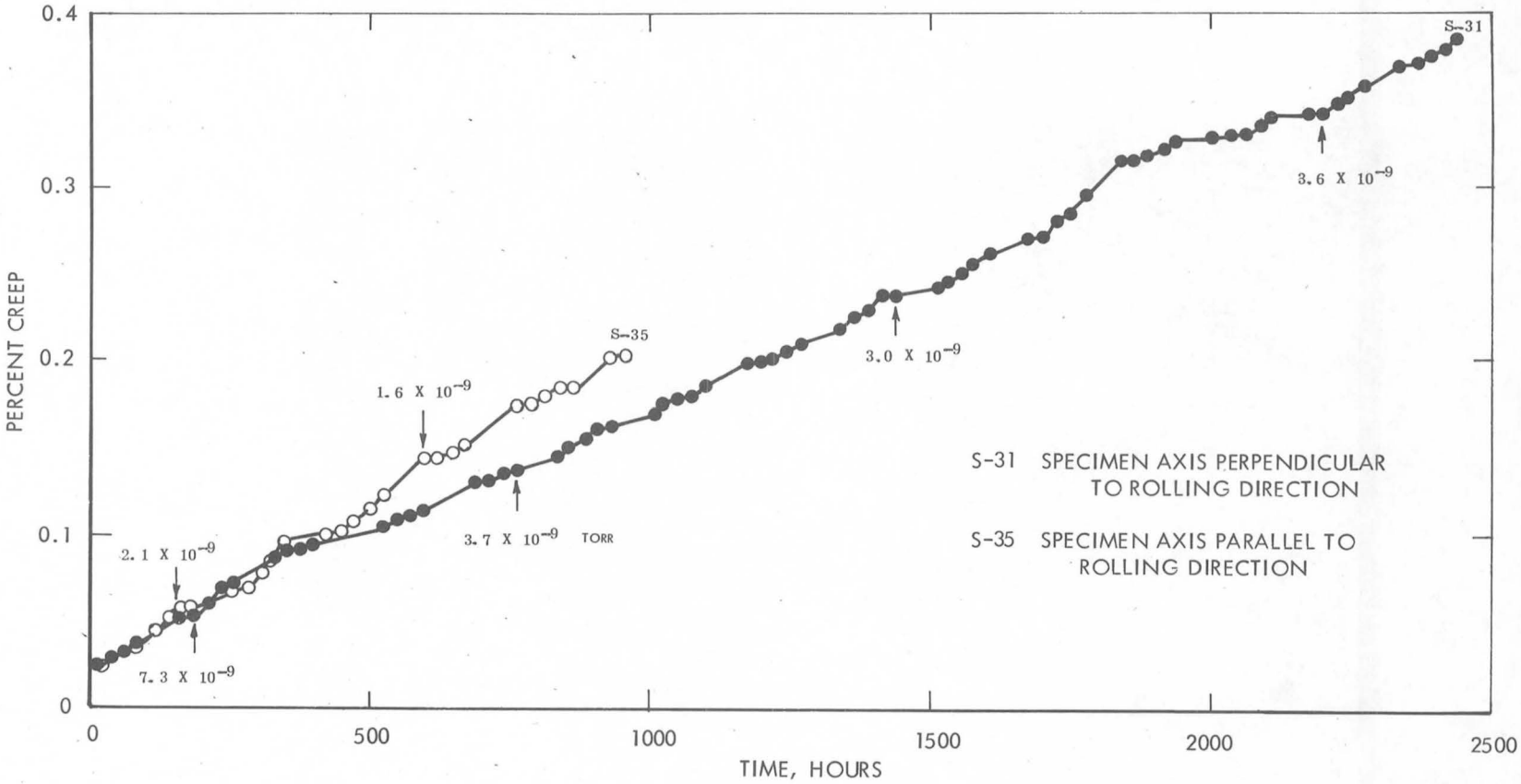


FIGURE 78 CREEP DATA FOR T-111 SHEET (HEAT 65079). TESTED AT 2200°F (1204°C) AND 5 Ksi (3.44×10^7 N/m^2) IN VACUUM ENVIRONMENT $< 1 \times 10^{-8}$ TORR.

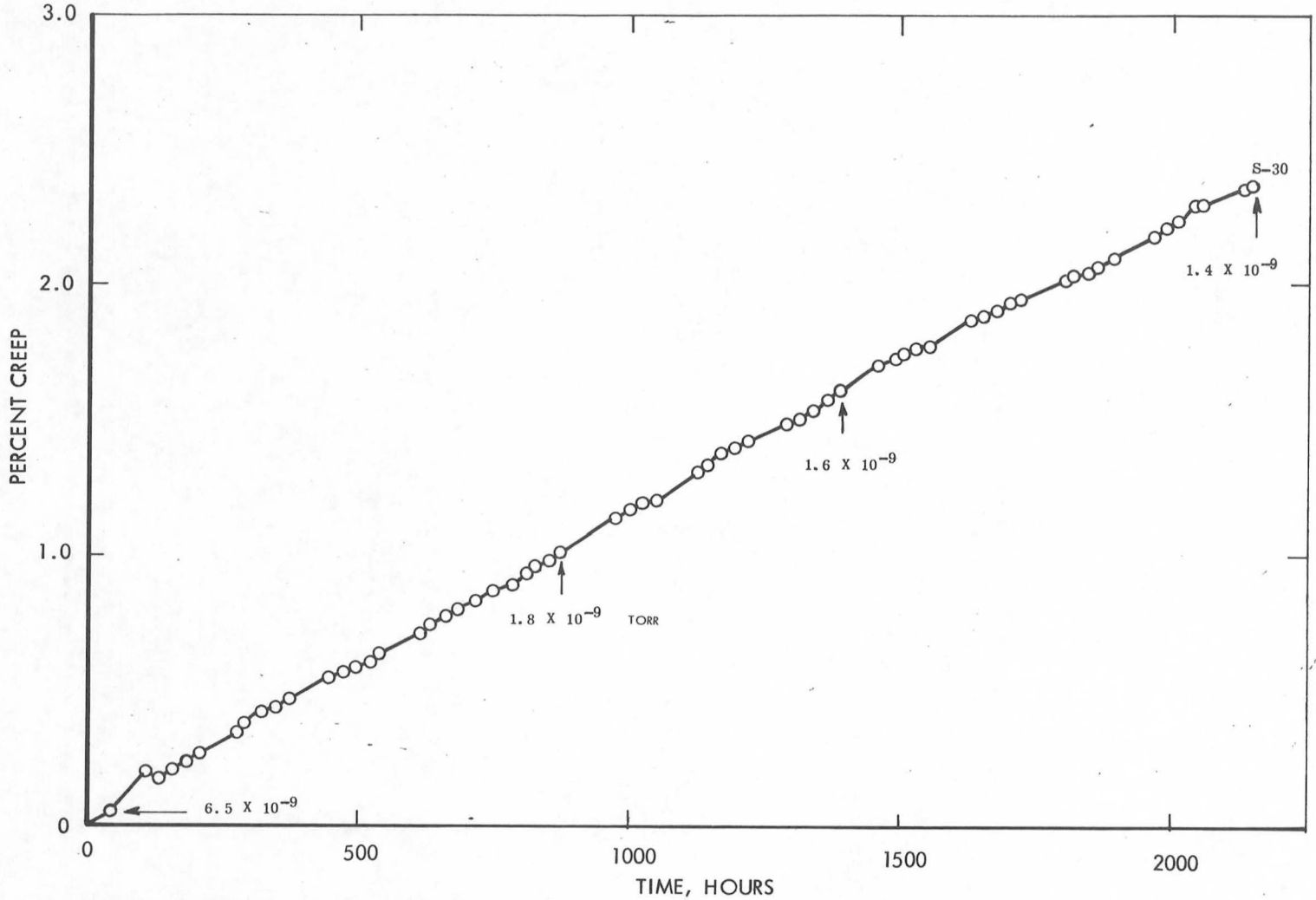


FIGURE 79 CREEP DATA FOR T-111 SHEET (HEAT 65079) SPECIMEN AXIS PERPENDICULAR TO ROLLING DIRECTION. TESTED AT 2400°F (1316°C) AND 3.5 Ksi (2.41×10^7 N/m^2) IN VACUUM ENVIRONMENT $< 1 \times 10^{-8}$ TORR.

4. T-111 Heat D-1670

Four creep tests were made with T-111 Heat D-1670 recrystallized one hour at 3000°F (1649°C).

Specimen No.	Test Temperature °F	°C	Stress ksi	N/m^2
S-25	2000	1093	15	1.03×10^8
S-25A	2600	1427	1.5	1.03×10^7
S-26	1800	982	17	1.17×10^8
S-28	2600	1427	0.5	3.44×10^6

Tests S-25 and S-25A were made using the same specimen. Before each test the specimen was recrystallized at 3000°F (1649°C) for one hour. The creep data for all tests of Heat D-1670 are plotted in Figures 80, 81, and 82. While the test at 1800°F (982°C), Figure 81, exhibited a time dependent increase in creep rate, the other three did not. In fact both Test B-25A and B-28 at 2600°F (1427°C), Figures 80 and 82 respectively, showed a marked tendency for the creep rate to decrease with time.

5. T-111 Heat D-1102

Two creep tests of specimens from Heat D-1102 were run and the creep data are plotted in Figure 83.

Specimen No.	Test Temperature °F	°C	Stress ksi	N/m^2
S-27	2000	1093	13	8.95×10^7
S-32	2200	1204	5	3.44×10^7

The test parameters used with specimens S-32 (Heat D-1102) and S-35 (Heat 65079) were identical, i.e., 2200°F (1204°C) and 5 ksi ($3.44 \times 10^7 N/m^2$). A comparison of the creep data for these tests, Figures 78 and 83, shows that Heat 65079 had greater creep resistance.

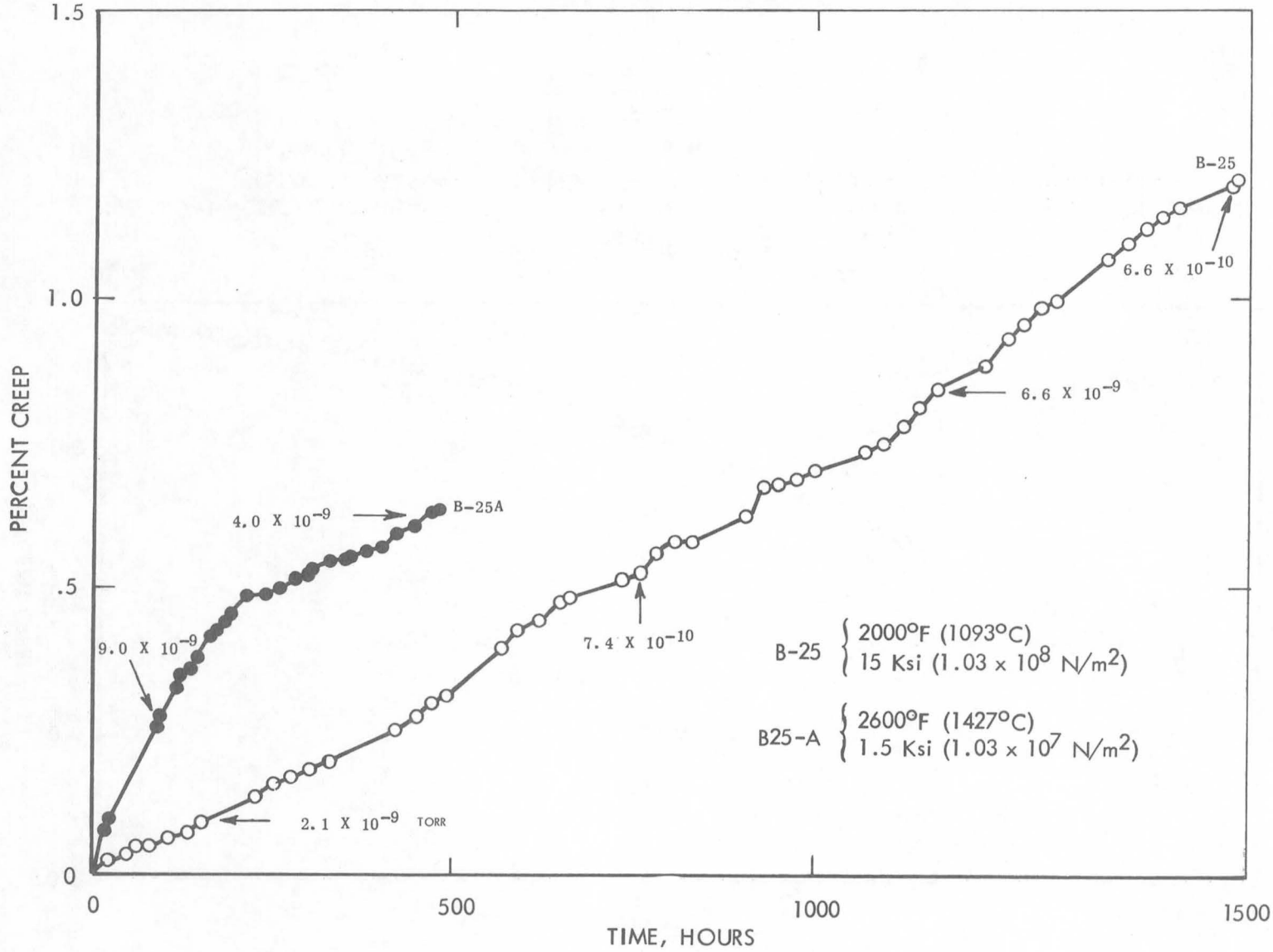


FIGURE 80 CREEP DATA FOR T-111 SHEET (HEAT D-1670) RECRYSTALLIZED ONE HOUR, 3000°F (1649°C). TESTED IN VACUUM ENVIRONMENT $< 1 \times 10^{-8}$ TORR.

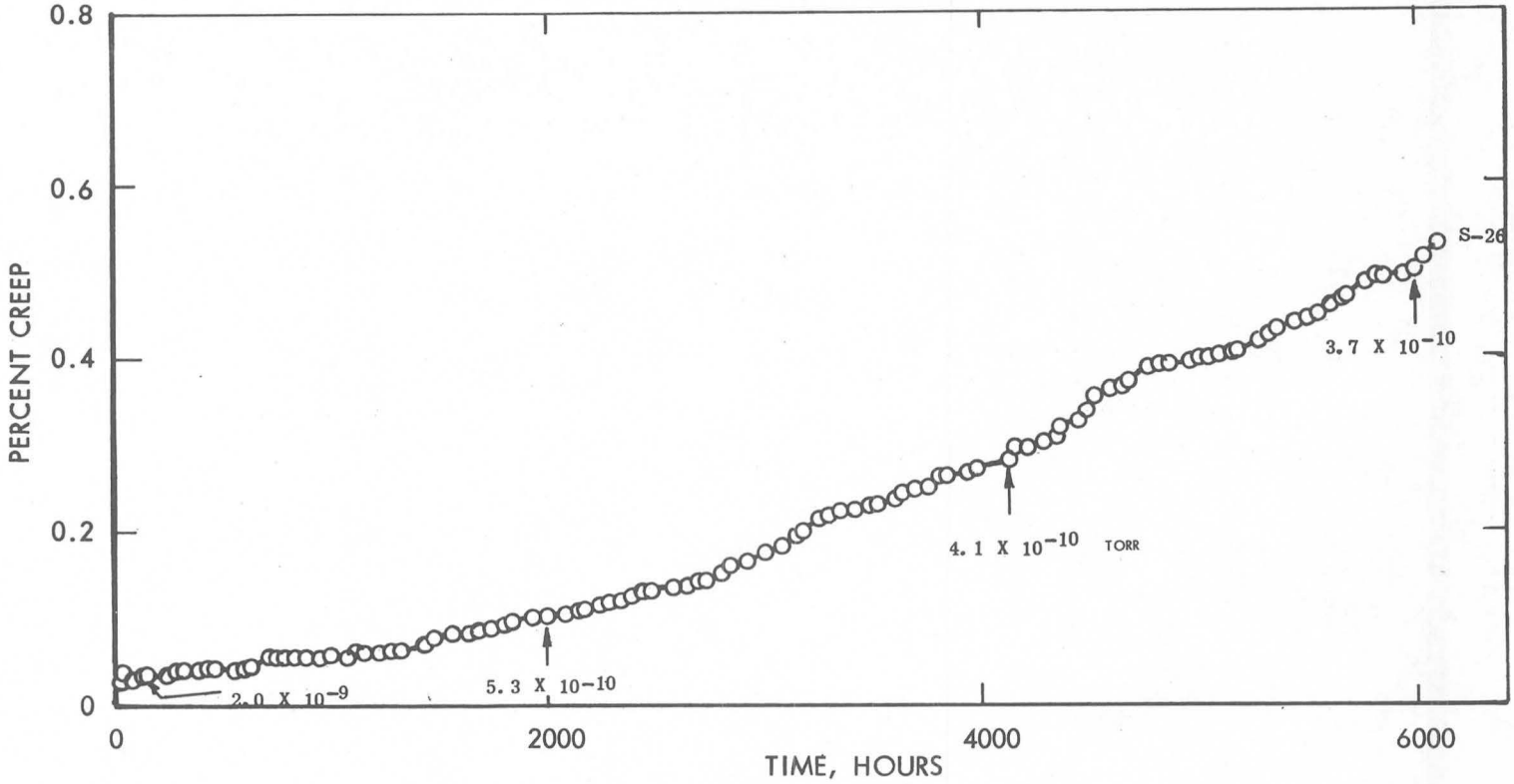


FIGURE 81 CREEP DATA FOR T-111 SHEET (HEAT D-1670) RECRYSTALLIZED ONE HOUR, 3000°F (1649°C). TESTED AT 1800°F (982°C) AND 17 Ksi (1.17×10^8 N/m^2) IN VACUUM ENVIRONMENT $<1 \times 10^{-8}$ TORR.

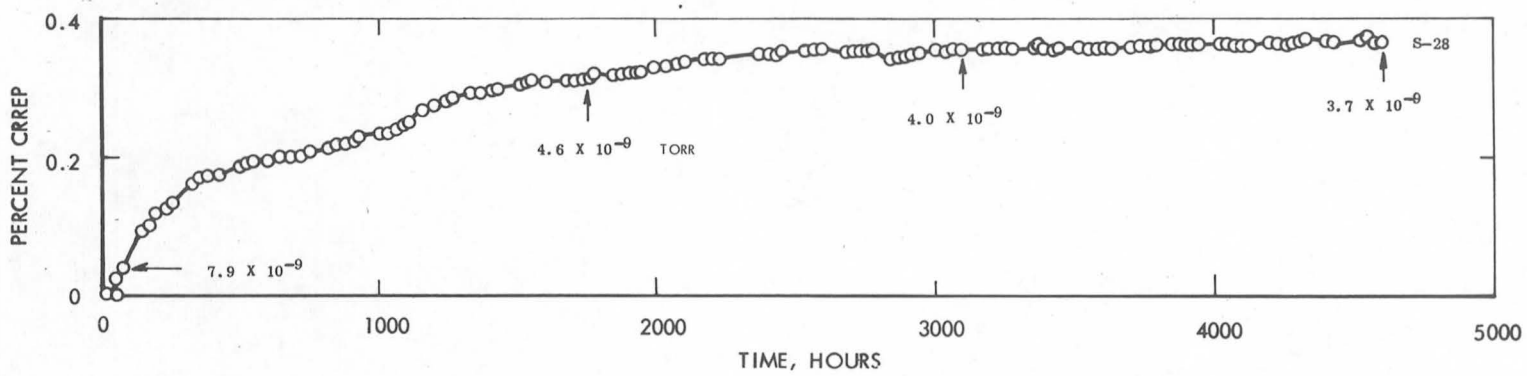


FIGURE 82 CREEP DATA FOR T-111 SHEET (HEAT D-1670) RECRYSTALLIZED ONE HOUR 3000°F (1649°C). TESTED AT 2600°F (1427°C) AND 0.5 Ksi (3.44×10^6 N/m^2) IN VACUUM ENVIRONMENT $< 1 \times 10^{-8}$ TORR.

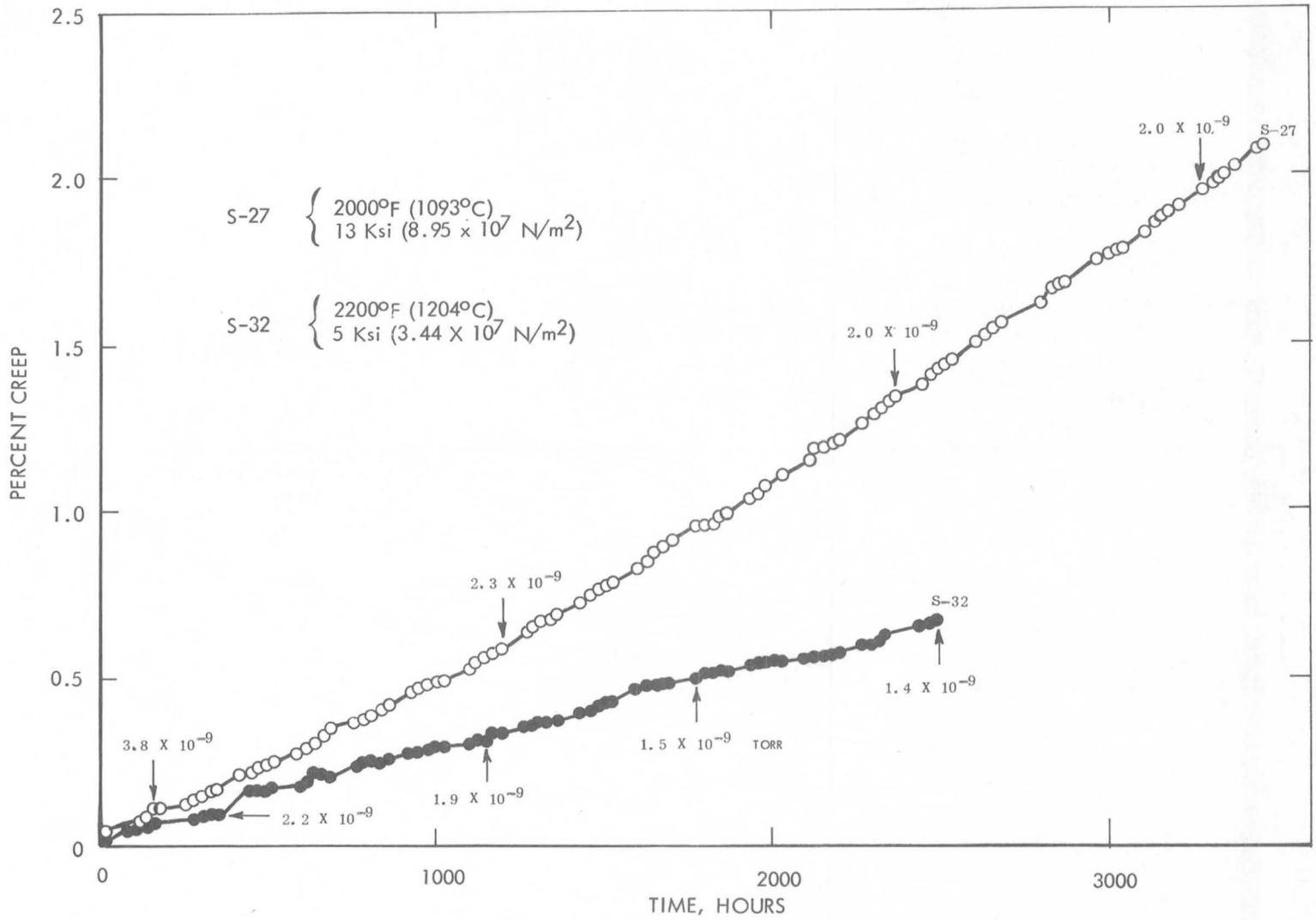


FIGURE 83 CREEP DATA FOR T-111 SHEET (HEAT 1102) RECRYSTALLIZED ONE HOUR AT 3000°F (1649°C). TESTED IN VACUUM ENVIRONMENT $< 1 \times 10^{-8}$ TORR.

6. T-111 Heat MCN02A065

Two tests on specimens of T-111 from Heat MCN02A065 are now in progress and the creep data are shown in Figure 84. A comparison of test S-33 at 2200°F (1204°C) and 8 ksi (5.51 x 10^7 N/m^2) with test S-19 for Heat 70616 (see Figure 73) indicates that Heat MCN02A065 may be more creep resistant than Heat 70616.

A summary of the creep data from all heats of T-111 is given in Table 12. The data for tantalum-base T-222 are also included for comparative purposes. The constant of 11.1 used in computing the Larson-Miller parameters shown in Table 12 and plotted in Figure 85 was obtained by a computer fit of the data. In solving for constants for the Manson-Haferd parameters, four tests (S-21, S-22, S-23 and S-24) run at two different stresses were used. The constants determined from an exact solution of the parametric equation were then used to plot all the data shown in Figure 85. The results from tests S-16 and S-20 have not been plotted since the material was not recrystallized at 3000°F (1649°C). The results show that T-111 heats 70616 and MCN02A065 have creep strengths comparable to that of T-222 Heat AL-TA-43. The other heats of T-111 (Heats 65079, D-1670, and D-1102) exhibited a lower creep resistance. The results indicate the difference in creep strength which can occur between various heats of T-111 alloy with the same nominal composition.

7. Astar 811C

A single test of this alloy is now in progress at 2600°F (1427°C) and 2 ksi (1.38 x 10^7 N/m^2). The creep data for the material recrystallized 1/2 hour at 3600°F (1982°C) are given in Figure 86. The extrapolated time to reach 1% creep of approximately 16,500 hours is indicative of a creep strength considerably greater than that of T-111 (see Figure 87).

E. Tungsten and Tungsten Alloys

1. Arc-Melted Tungsten Heat KC-1357

Five tests were made with arc-melted tungsten sheet recrystallized for one hour at the test temperature.

Specimen No.	Test Temperature °F	°C	Stress ksi	N/m^2
S-5	3200	1760	3	2.07 x 10^7
S-7	3200	1760	0.4	2.76 x 10^6
S-9	3200	1760	1	6.89 x 10^6
S-17	2800	1538	4	2.76 x 10^7
S-18	2800	1538	3	2.07 x 10^7

The creep data for these tests are given in Figure 88.

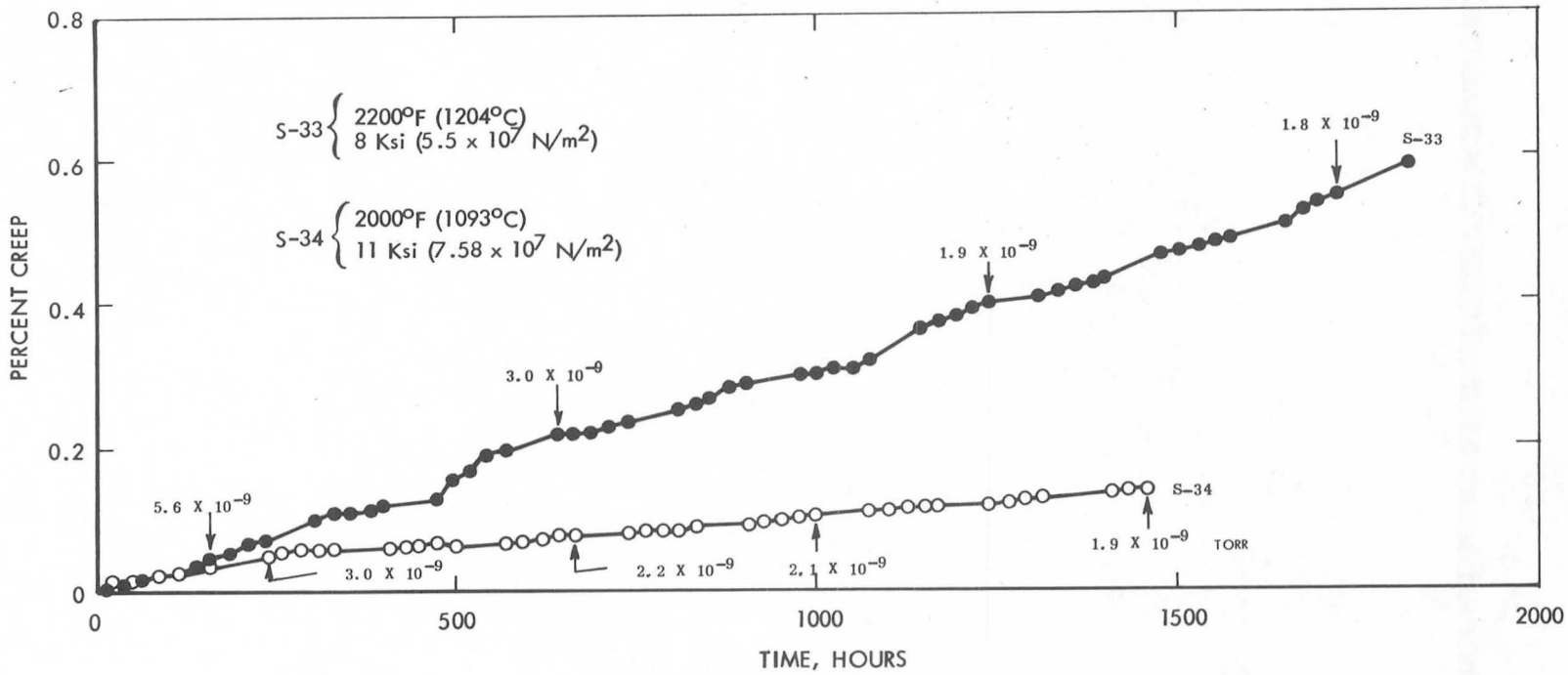


FIGURE 84 CREEP DATA FOR T-111 SHEET (HEAT MCN 02A065) RECRYSTALLIZED ONE HOUR AT 3000°F (1649°C). TESTED IN VACUUM ENVIRONMENT $< 1 \times 10^{-8}$ TORR.

TABLE 12

Summary of Creep Data for Tantalum-Base T-111 and T-222 Alloys, Recrystallized 3000°F (1649°C), 1 Hour

Specimen No.	Test Temperature °F	°C	Stress ksi	N/m^2	Hours to 1% Creep	Manson-Haferd Parameter 1% Creep $\frac{\log t - 17.5}{T + 417} \times 10^3$	Creep Rate $in\text{-}in^{-1}\text{-}hr^{-1}$	Larson-Miller Parameter $T(°R)(11.1 + \log t) \times 10^{-3}$
T-111 Heat 70616								
S-16 (1)	2200	1204	8	5.15×10^7	700	-5.60	1.4×10^{-5}	
S-19	2200	1204	8	5.15×10^7	2,000	-5.43	5.0×10^{-6}	38.4
S-21	2200	1204	12	8.26×10^7	1,140	-5.53	8.8×10^{-6}	37.6
S-22	2000	1093	20	1.38×10^8	670	-6.08	1.7×10^{-5}	34.2
S-23	2120	1160	12	8.26×10^7	3,150	-5.53	3.2×10^{-6}	37.6
S-24	1860	1016	20	1.38×10^8	4,730	-6.08	2.1×10^{-6}	34.2
T-111 Heat 65079								
S-30 (2)	2400	1316	3.5	2.41×10^7	860	-5.18	1.2×10^{-5}	40.1
S-31 (2)	2200	1204	5	3.44×10^7	5,600*	-5.26	1.8×10^{-6}	39.7
S-35	2200	1204	5	3.44×10^7	4,500*	-5.30	2.2×10^{-6}	39.6
T-111 Heat D-1670								
S-25	2000	1093	15	1.03×10^8	1,340	-5.95	7.5×10^{-6}	35.0
S-25A	2600	1427	1.5	1.03×10^7	1,100*	-4.80	9.1×10^{-6}	43.3
S-26	1800	982	17	1.17×10^8	8,400*	-6.13	1.2×10^{-6}	34.0
S-28	2600	1427	0.5	3.44×10^6	95,000*	-4.15	1.1×10^{-7}	46.4
T-111 Heat D-1102								
S-27	2000	1093	13	8.95×10^7	1,880	-5.90	5.3×10^{-6}	35.4
S-32	2200	1204	5	3.44×10^7	3,800*	-5.33	2.6×10^{-6}	39.2
T-111 Heat MCN02A065								
S-33	2200	1204	8	5.51×10^7	3,000*	-5.37	3.3×10^{-6}	38.7
S-34	2000	1093	11	7.58×10^7	12,300*	-5.55	7.8×10^{-7}	37.2
T-222 Heat AL-TA-43								
S-13	2200	1204	12	8.26×10^7	620	-5.63	1.6×10^{-5}	37.0
S-14	2056	1124	19.2	1.32×10^8	900	-5.89	1.1×10^{-5}	35.3
S-20 (3)	2200	1204	12	8.26×10^7	420	-5.92	2.4×10^{-5}	

* Extrapolated Value; (1) - Recrystallized one hour 2600°F (1427°C) (2) - Specimen Axis perpendicular to rolling direction (3) - Recrystallized one hour 2800°F (1538°C).

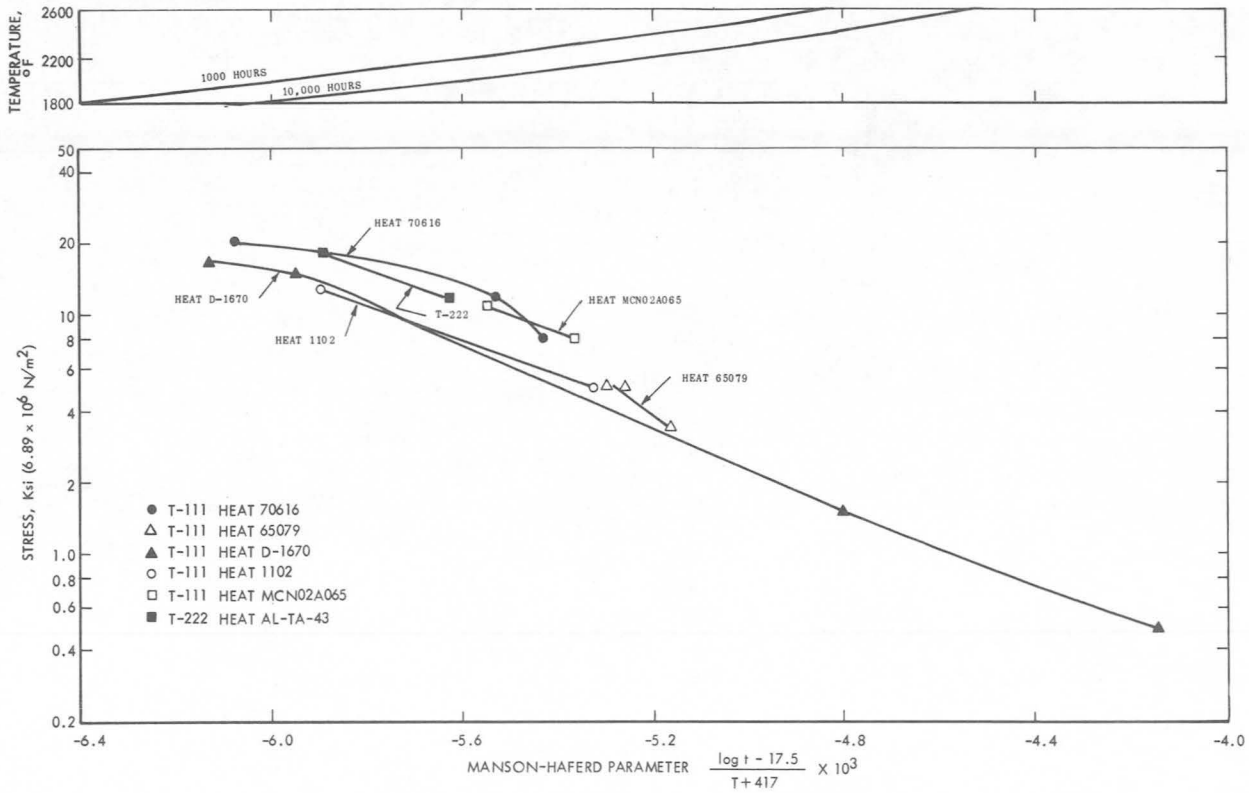


FIGURE 85A PARAMETRIC PLOT OF 1% CREEP DATA FOR T-111 AND T-222 SHEET RECRYSTALLIZED ONE HOUR AT 3000°F (1649°C). TESTED IN VACUUM ENVIRONMENT $< 1 \times 10^{-8}$ TORR.

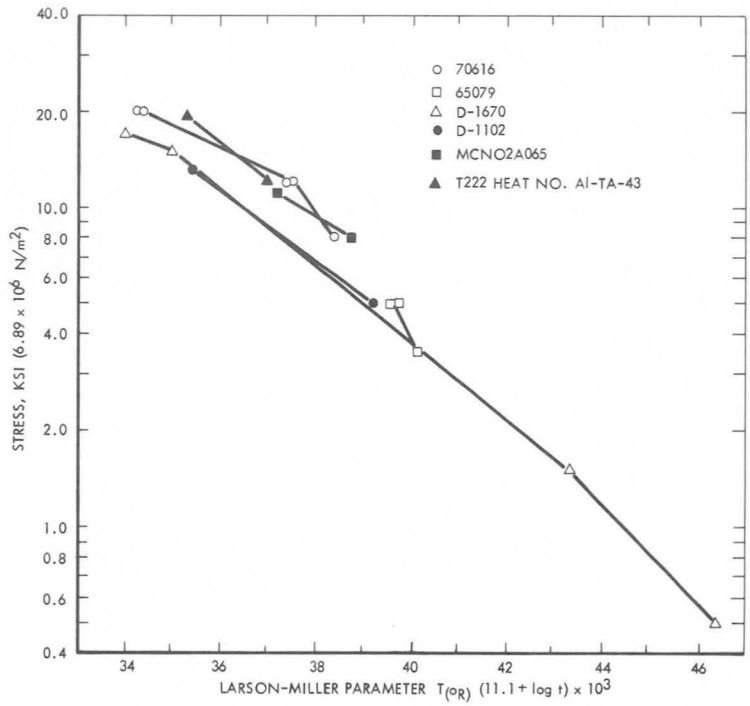


FIGURE 85B PARAMETRIC PLOT OF 1% CREEP DATA FOR T-111 AND T-222 RECRYSTALLIZED ONE HOUR AT 3000°F (1649°C). TESTED IN VACUUM ENVIRONMENT $< 1 \times 10^{-8}$ TORR.

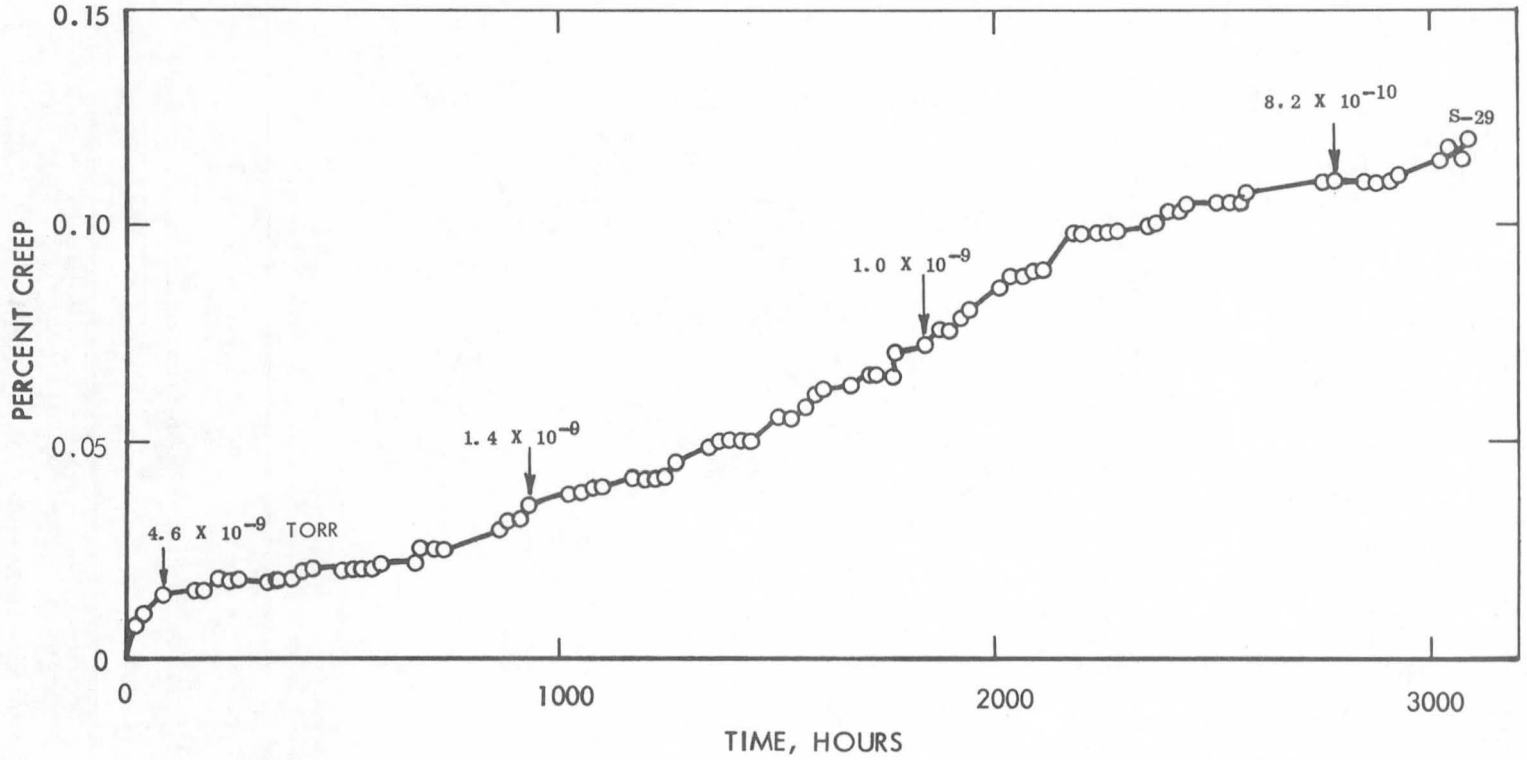


FIGURE 86 CREEP DATA FOR ASTAR 811C SHEET RECRYSTALLIZED ONE-HALF HOUR AT 3600°F (1982°C). TESTED AT 2600°F (1427°C) AND 2 Ksi (1.38×10^7 N/m^2) IN VACUUM ENVIRONMENT $<1 \times 10^{-8}$ TORR.

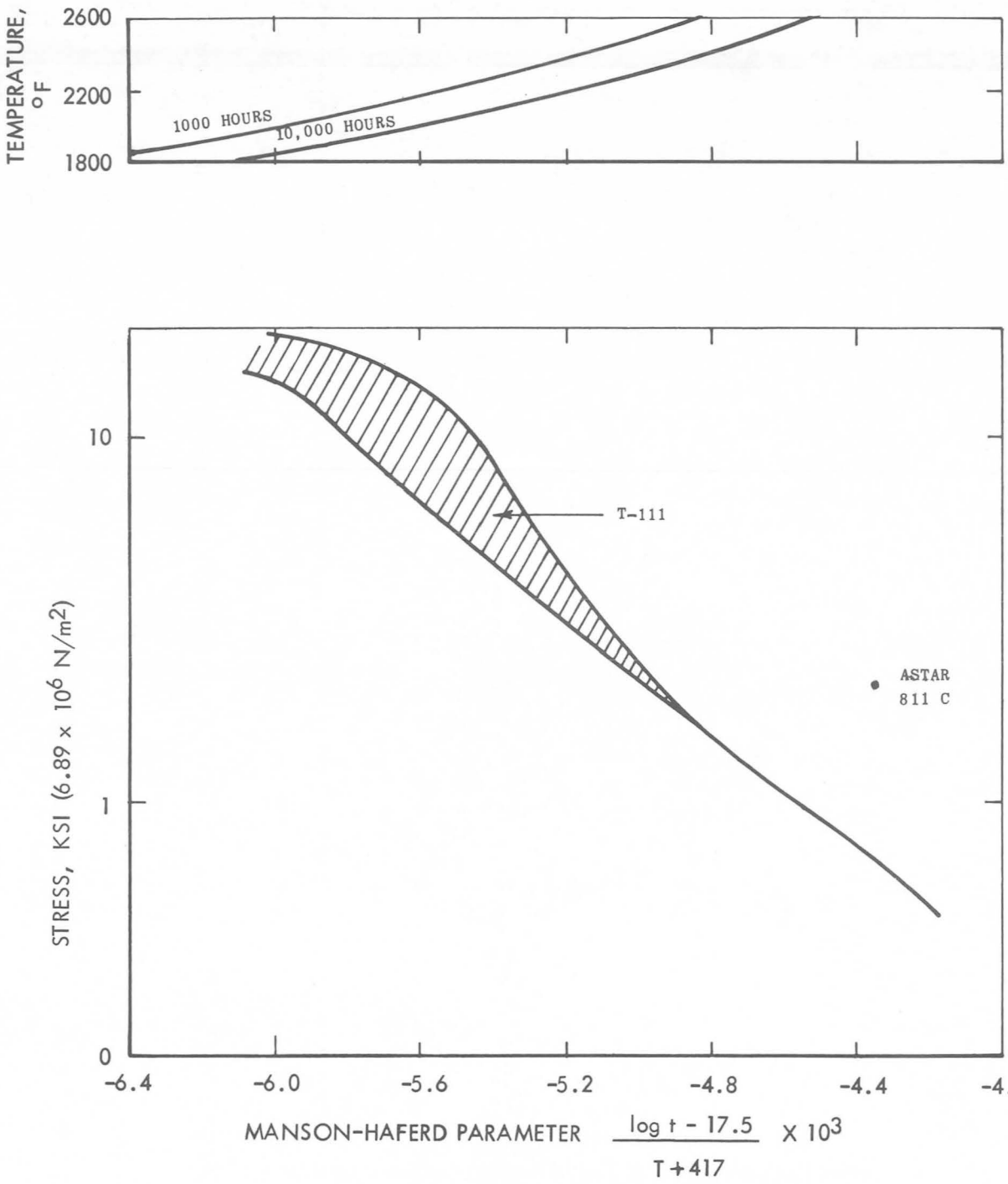


FIGURE 87 PARAMETRIC PLOT OF 1% CREEP DATA FOR ASTAR 811C SHEET RECRYSTALLIZED ONE-HALF HOUR AT 3600°F (1982°C). TESTED IN VACUUM ENVIRONMENT $< 1 \times 10^{-8}$ TORR.

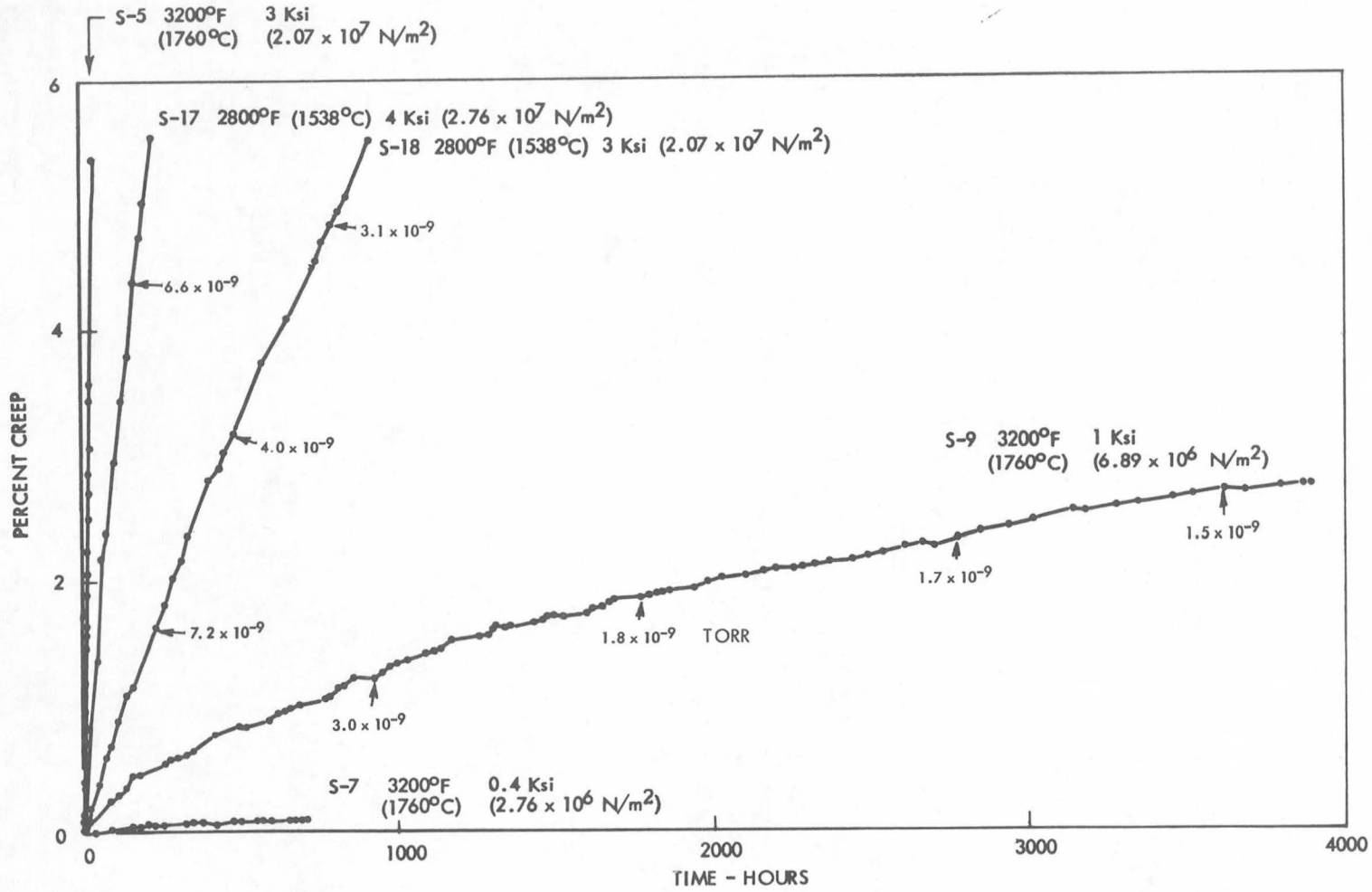


FIGURE 88 CREEP DATA FOR ARC MELTED TUNGSTEN SHEET (HEAT KC-1357) RECRYSTALLIZED 1 HOUR AT TEST TEMPERATURE. TESTED IN VACUUM ENVIRONMENT $< 1 \times 10^{-8}$ TORR.

Post-test examination showed that grain growth was a dominant feature for test S-9 shown in Figure 89. Visual examination indicated that some grains had completely transversed the 0.030 inch thick specimen. Examination of the thermally etched specimen surface failed to show evidence of creep occurring by grain boundary sliding but grain boundary migration associated with grain growth was observed as shown by Figure 90.

2. Vapor Deposited Tungsten

Two vapor-deposited tungsten specimens were tested and the creep data are plotted in Figure 91. Post-test photomicrographs of vapor-deposited tungsten specimen B-24 are shown in Figure 92. This specimen was tested at 2800°F (1538°C) and 2 ksi ($1.38 \times 10^7 N/m^2$) for 6812 hours. During that time, 3.71% extension occurred. The unstressed end of the specimen exhibited columnar grains characteristic of the vapor-deposited material. The stressed gauge section had a similar structure and in addition showed grain boundary porosity probably due to vacancy condensation during test.

A longitudinal section of specimen B-17 taken from the gage area exhibited fine grains alone one edge, Figure 93. Presumably this was the interface between the forming mandral and the tungsten. This large variation of grain size is an intrinsic characteristic of the vapor deposition process, and will be present in the creep specimen to varying degrees depending on how the sample blanks are cut from the as-deposited material. Porosity was apparent, and the grains were equiaxed rather than columnar. The hardness of the vapor-deposited tungsten after test was 413 DPH, identical to arc-melted tungsten recyrstallized at 3200°F (1760°C).

3. Arc-Melted Tungsten-25% Rhenium, Heat 3.5-75002

Four tests were made with the tungsten-rhenium alloy in the recrystallized condition and the creep data are presented in Figure 94.

Specimen No.	Test Temperature °F	°C	Stress ksi	N/m^2
S-3	3200	1760	5	3.44×10^7
S-4	3200	1760	3	2.07×10^7
S-6	3200	1760	0.5	3.44×10^6
S-8	3200	1760	1.5	1.03×10^7

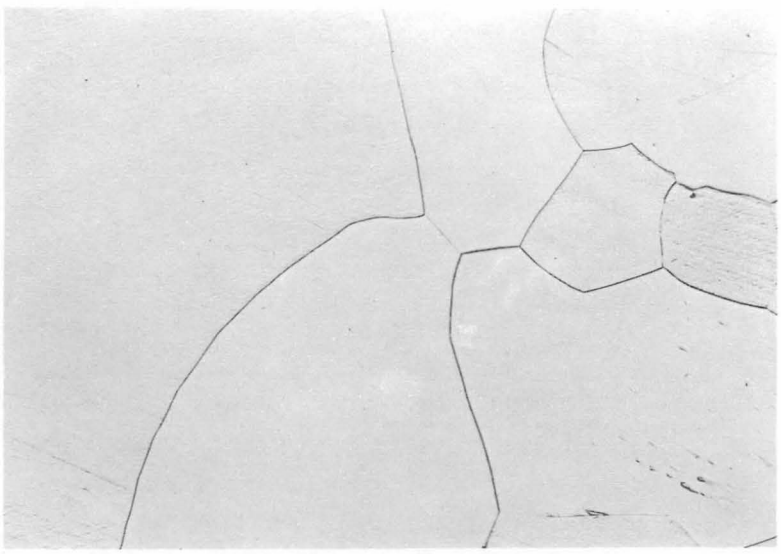


FIGURE 89 GRAIN GROWTH IN ARC MELTED TUNGSTEN CREEP SPECIMEN S-9 TESTED 3886 HOURS AT 3200°F (1670°C).
ETCHANT: 15% HF, 15% H_2SO_4, 8% HNO_3, 62% H_2O. 100X

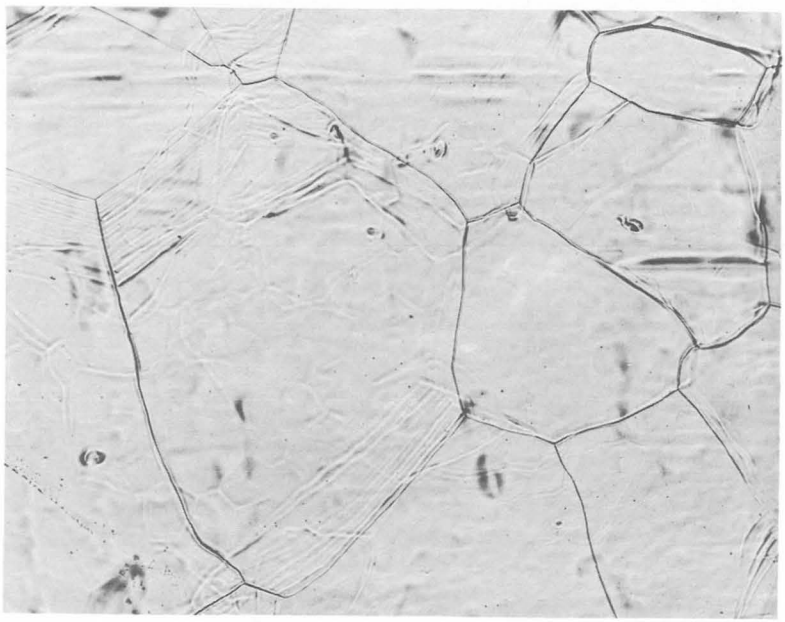


FIGURE 90 ARC-MELTED TUNGSTEN SHEET SPECIMEN S-7 TESTED 720 HOURS AT 3200°F (1670°C) SHOWING GRAIN BOUNDARY MIGRATION. THERMALLY ETCHED DURING TESTING. 100X

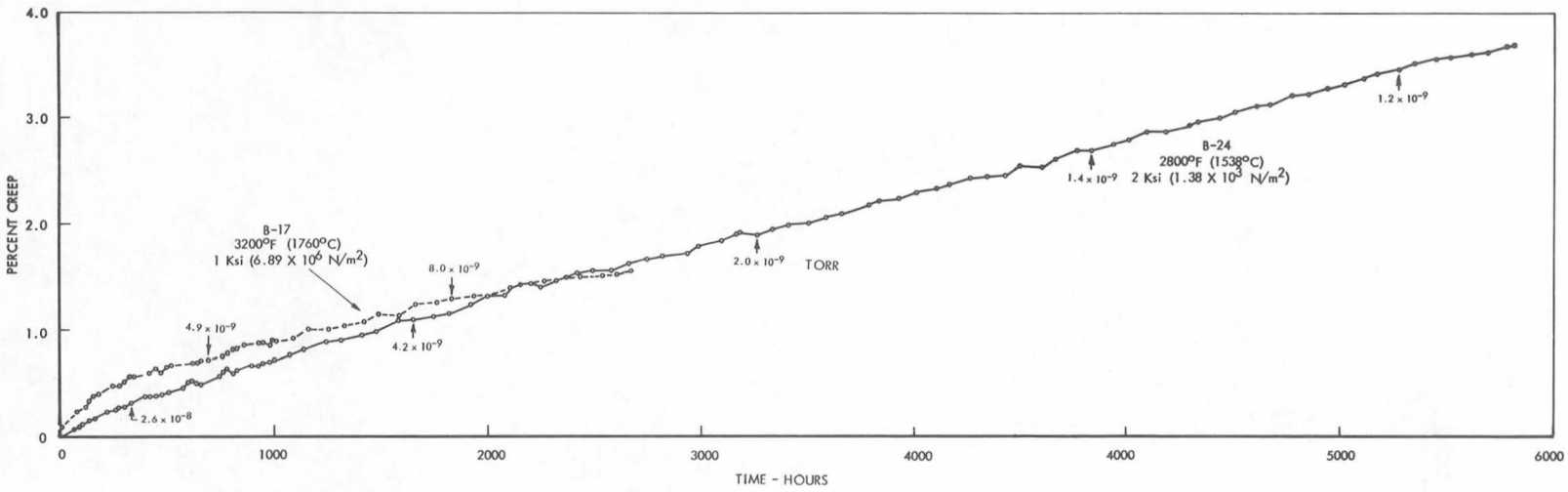
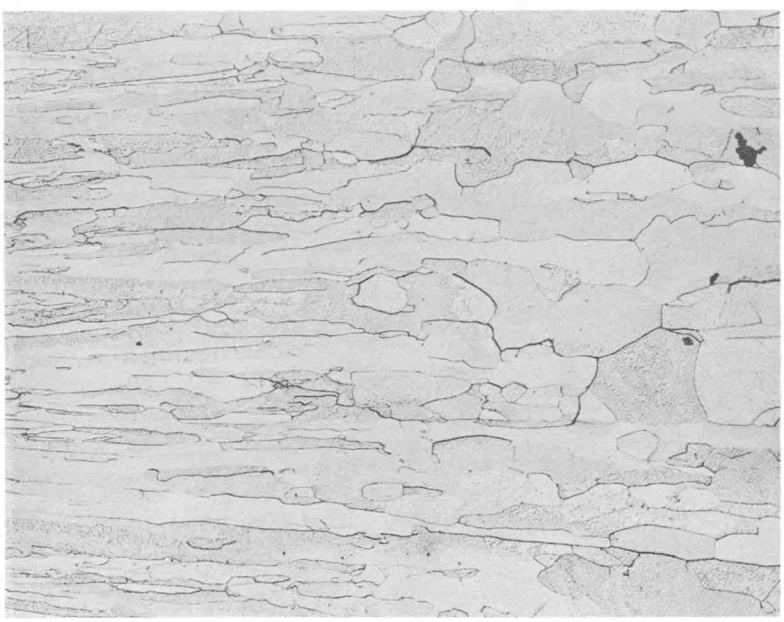


FIGURE 91 CREEP DATA FOR VAPOR-DEPOSITED TUNGSTEN. TESTED IN VACUUM ENVIRONMENT $< 1 \times 10^{-8}$ TORR.

⊕ TENSILE AXIS

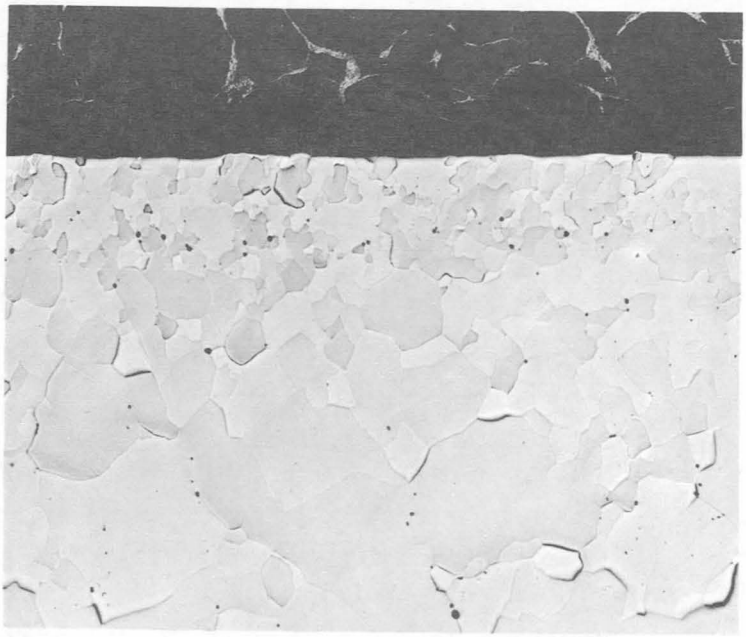
GAGE SECTION PERPENDICULAR TO SPECIMEN AXIS



⊕ TENSILE AXIS

HEAD SECTION PERPENDICULAR TO SPECIMEN AXIS

FIGURE 92 VAPOR DEPOSITED TUNGSTEN MICROSTRUCTURE AFTER TEST. ETCHANT: MURAKAMI'S. 100X



TENSILE
AXIS

413 DPH

100X

FIGURE 93 VAPOR DEPOSITED TUNGSTEN, CREEP TESTED 2863 HOURS AT 3200°F (1760°C) AND 1000 PSI (6.89×10^6 N/m^2), UNETCHED

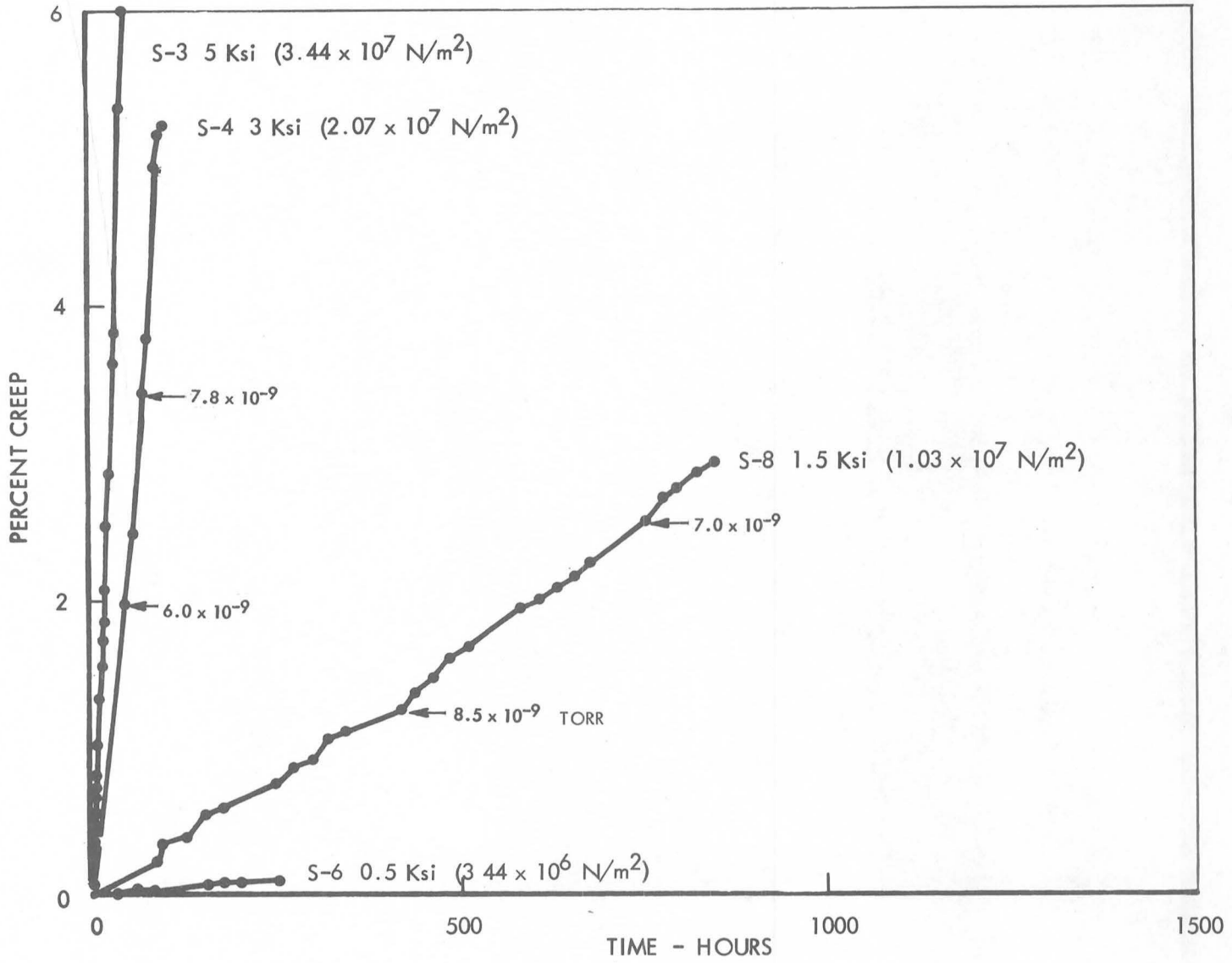


FIGURE 94 CREEP DATA FOR ARC MELTED TUNGSTEN - 25% RHENIUM SHEET (HEAT 3.5 -75002) RECRYSTALLIZED 1 HOUR AT 3200 F (1760 C). TESTED AT 3200°F (1760°C) IN VACUUM ENVIRONMENT $< 1 \times 10^{-8}$ TORR.

Post-test examination of the thermally etched surface of specimen S-4, Figure 95, showed that grain boundary migration had occurred just as with pure tungsten. Comparison of the grain size of specimens S-7 and S-8 revealed a considerable difference despite the similarity of the test time and temperature. If grain boundary migration contributes to creep it would appear that the contribution of the rhenium addition is in restricting grain growth.

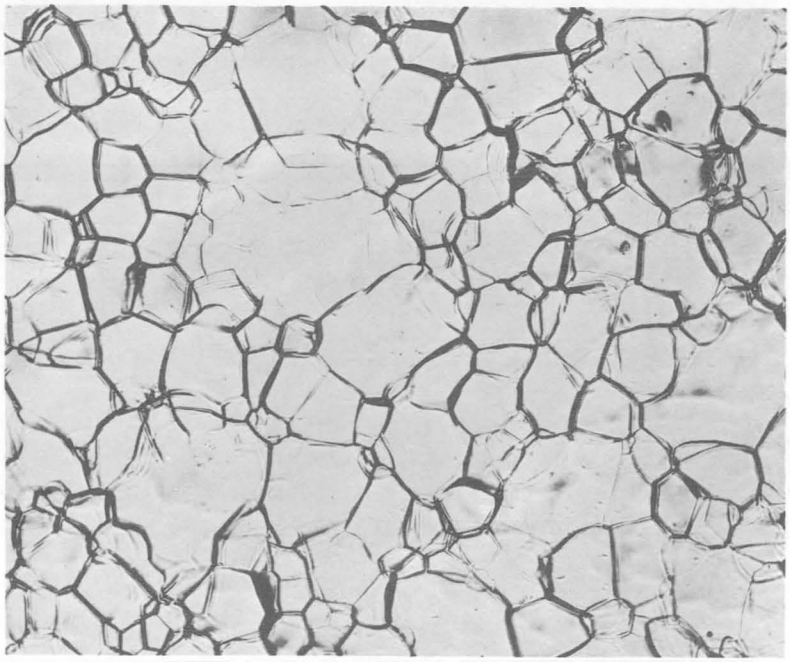
4. Sylvania A

Two tests at 3200°F (1760°C) were made with the Sylvania A in the recrystallized condition and the creep data are presented in Figure 96.

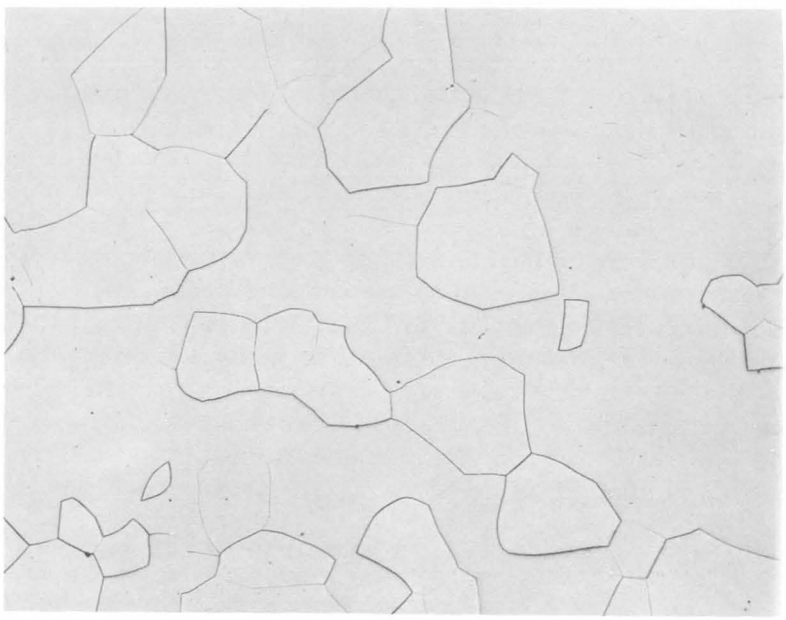
Post-test examination of specimen S-12 revealed a fine-grained microstructure, Figure 97. The second phase observed at 500X was probably responsible in part for the grain boundary pinning and consequent smaller grain size as compared to tungsten or tungsten-25% rhenium materials.

A summary of all creep data for tungsten, tungsten-25% rhenium, and Sylvania A is given in Table 13. The Larson-Miller values for 1% creep in the table are based on a constant of 15. Other results, plotted in Figure 98, show that arc-melted and vapor deposited tungsten have comparable creep strength. The tungsten-25% rhenium alloy had a greater creep strength only at the higher stress levels. While Sylvania A had a superior creep strength to both tungsten and tungsten-25% rhenium, the tendency for Sylvania A to be brittle offsets the higher creep strength.

The results obtained on the arc-melted and vapor-deposited tungsten were compared in Figure 99 to published creep data for pure tungsten (15, 16) using a Larson-Miller parameter for 1% creep. The results of Schmidt and Ogden (15) were obtained with a powder metallurgy product and tests were performed in an argon environment. A powder metallurgy product was also used by the SRI (16), but the test environment was a 10^{-4} torr vaccuum. The creep strength of the arc-melted and vapor-deposited tungsten is definitely inferior to that of the powder metallurgy product of Reference (15), however, the data from Reference (16) appear to be an extension of that obtained with the arc-melted and vapor-deposited material. In view of the fact that References (15) and (16) do not provide quantitative data relative to interstitial content of the material before or after test, the specific causes for the difference observed cannot be determined. However, it is interesting to note that the tests in argon produced higher creep strengths than the tests conducted in vacuum.



S-4 3 Ksi (2.07×10^7 N/m^2) THERMALLY ETCHED



S-8 1.5 Ksi (1.03×10^7 N/m^2)
ETCHANT: 15% HF, 15% H_2SO_4, 8% HNO_3, 62% H_2O

FIGURE 95 TUNGSTEN - 25% RHENIUM ALLOY AFTER CREEP TESTING AT 3200 F (1760 C) IN VACUUM ENVIRONMENT $<1 \times 10^{-8}$ TORR. 100X

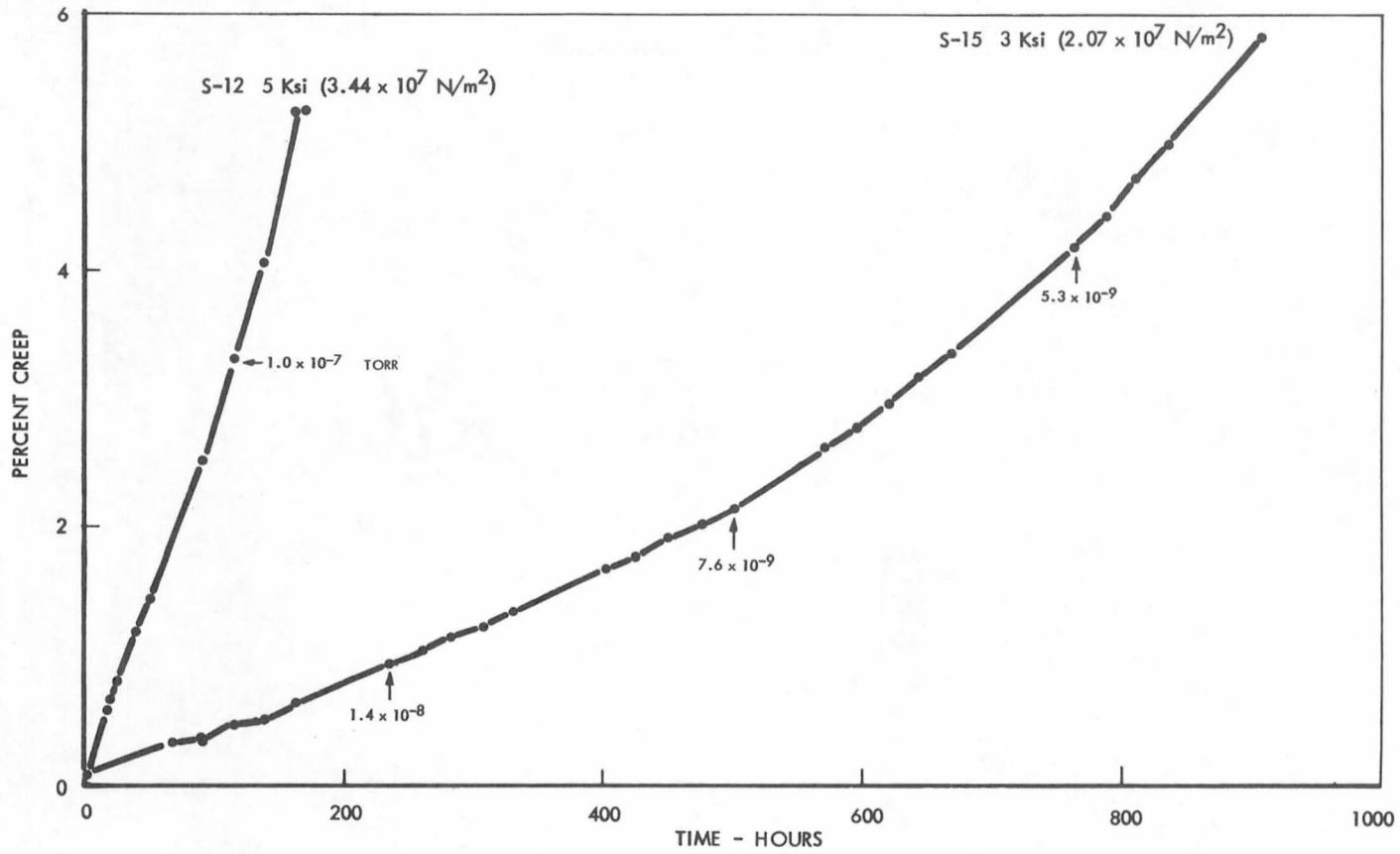
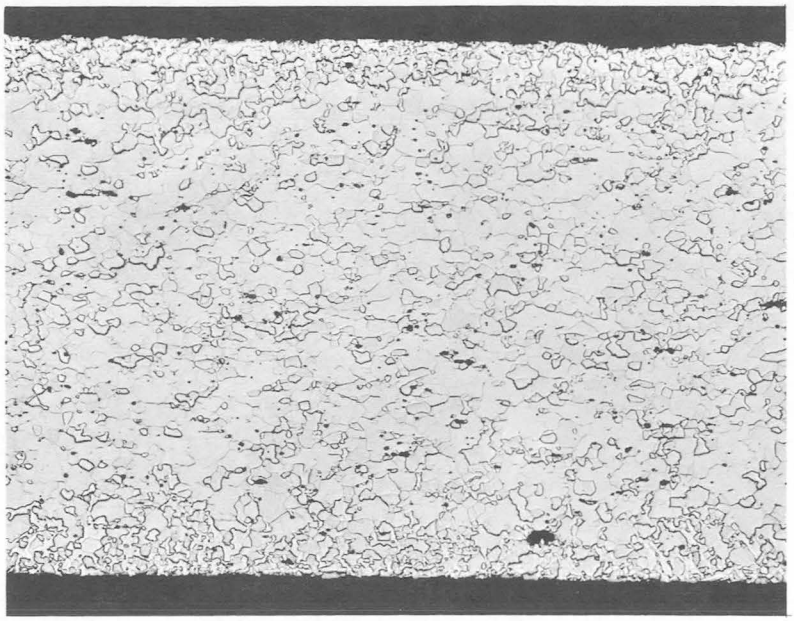
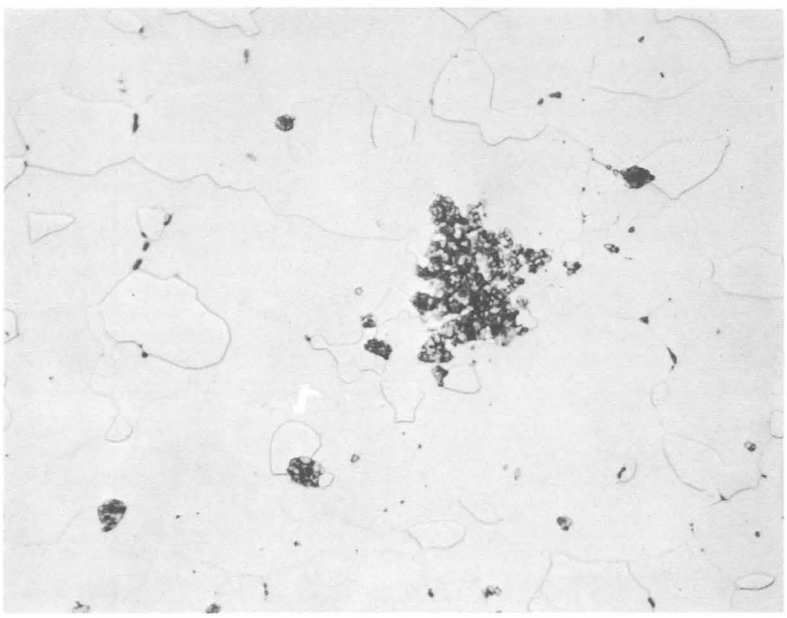


FIGURE 96 CREEP DATA FOR SYLVANIA-A ALLOY SHEET RECRYSTALLIZED 1 HOUR AT 3200°F (1760°C). TESTED AT 3200°F (1760°C) IN VACUUM ENVIRONMENT $< 1 \times 10^{-8}$ TORR.



⊕ TENSILE AXIS

CROSS-SECTION 100X



⊕ TENSILE AXIS

CROSS-SECTION 500X

FIGURE 97 GAUGE SECTION SYLVANIA-A ALLOY SHEET AFTER TESTING 170 HOURS AT 3200°F (1760°C) AND 5 Ksi (3.44×10^7 N/m^2) IN VACUUM ENVIRONMENT $< 1 \times 10^{-8}$ TORR. ETCHANT: 15% HF, 15% H_2SO_4, 8% HNO_3, 62% H_2O.

TABLE 13

Summary of Creep Data for Tungsten and Tungsten-Base Alloys

Spec. No.	Test Temperature °F	°C	Stress ksi	N/m^2	Hours to 1% Creep	Larson-Miller Parameter 1% Creep $T°R\ (15 + \log t) \times 10^{-3}$	Creep Rate $in\text{-}in^{-1}\text{-}hr^{-1}$
Tungsten Heat KC-1357							
S-5	3200	1760	3	2.07×10^7	6	57.8	1.7×10^{-3}
S-7	3200	1760	0.4	2.76×10^6	*	*	1.7×10^{-6}
S-9	3200	1760	1	6.89×10^6	675	65.4	1.5×10^{-5}
S-17	2800	1538	4	2.76×10^7	20	53.1	5.0×10^{-4}
S-18	2800	1538	3	2.07×10^7	125	55.8	8.0×10^{-5}
Vapor Deposited Tungsten							
B-17	3200	1760	1	6.89×10^6	1140	66.0	8.8×10^{-6}
B-24	2800	1538	2	1.38×10^7	1500	59.2	6.7×10^{-6}
Tungsten-25% Rhenium Heat 3.5-75002							
S-3	3200	1760	5	3.44×10^7	12	58.9	8.3×10^{-4}
S-4	3200	1760	3	2.07×10^7	25	60.0	4.0×10^{-4}
S-6	3200	1760	0.5	3.44×10^6	*	*	3.6×10^{-6}
S-8	3200	1760	1.5	1.03×10^7	315	64.0	3.2×10^{-5}
Sylvania A							
S-12	3200	1760	5	3.44×10^7	35	60.6	2.9×10^{-4}
S-15	3200	1760	3	2.07×10^7	250	63.7	4.0×10^{-5}

* Too little creep to extrapolate

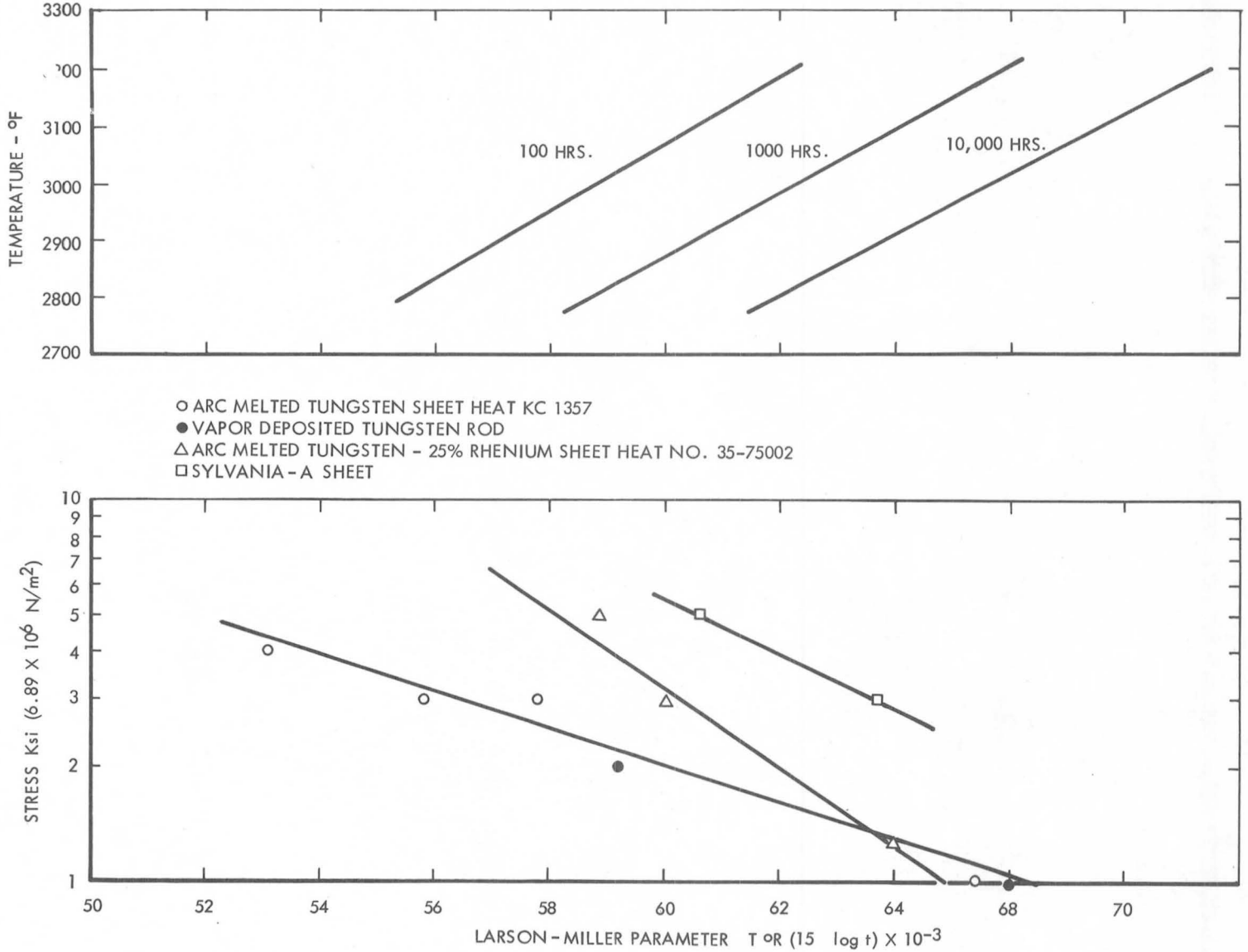


FIGURE 98 LARSON-MILLER PLOT OF 1% CREEP DATA FOR TUNGSTEN AND TUNGSTEN BASE ALLOYS. TESTED IN VACUUM ENVIRONMENT $< 1 \times 10^{-8}$ TORR.

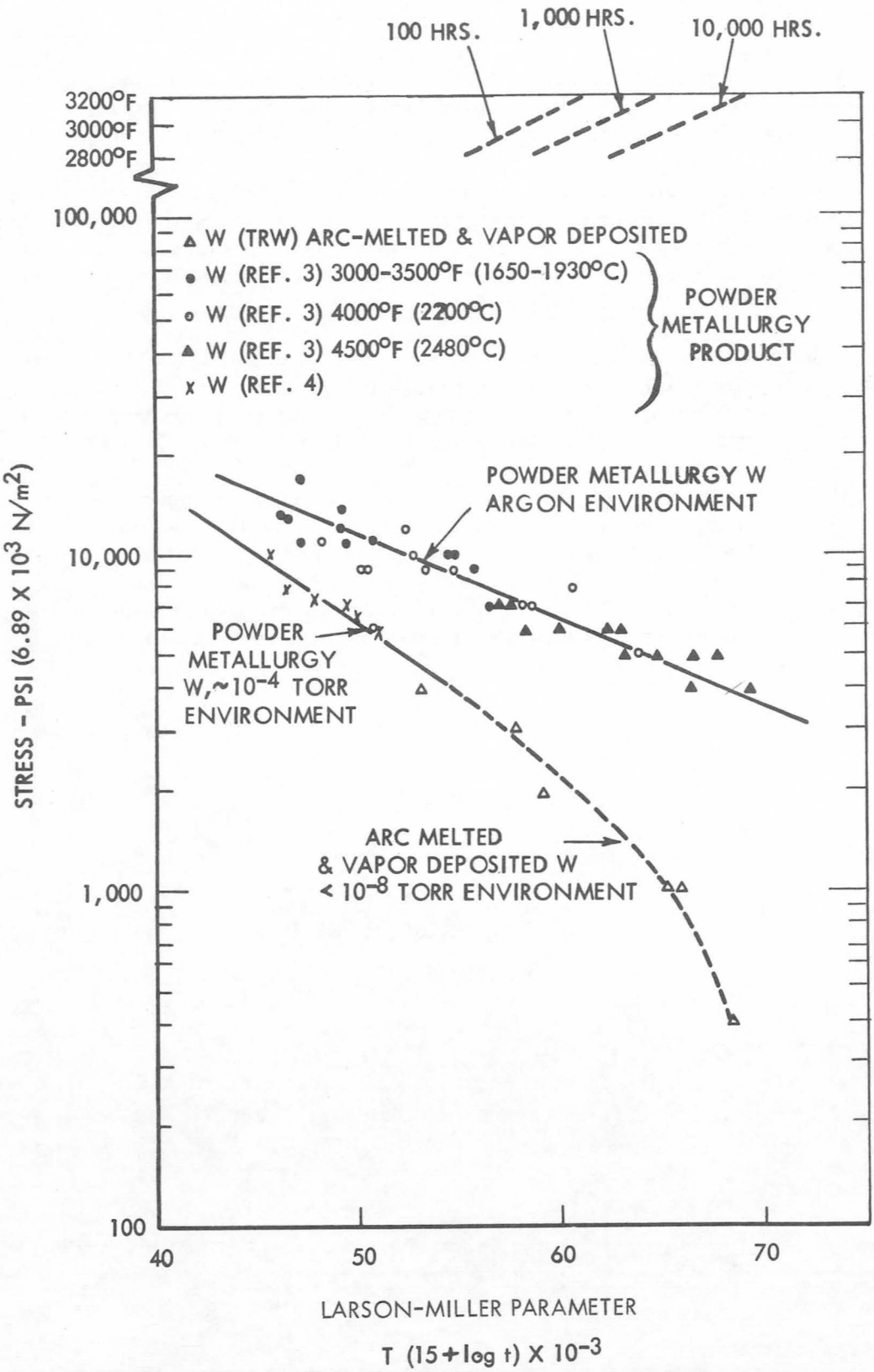


FIGURE 99 LARSON-MILLER PLOT OF TUNGSTEN 1% CREEP DATA.

F. Comparison of Various Refractory Alloys

The creep properties of the various classes of materials are compared in Figures 100 and 101. In Figure 100a the potential turbine alloys are presented on a Larson-Miller plot of 0.5% creep using a constant of 20.1.

The results show that molybdenum-base TZC and TZM alloys have creep strengths superior to Cb-132M. However, comparison of these alloys on the basis of strength-to-weight ratios (Figure 100b) tends to minimize this difference. Figure 100 also indicates that molybdenum-base TZM Heat KDTZM-1175 was more creep resistant than TZC Heat M-91.

Four possible cladding materials are compared in Figure 101; tantalum-base T-111, Astar 811C, tungsten and tungsten-25% rhenium. The parametric comparison for 1% creep, shown in Figure 101, is based on the Manson-Haferd constants previously used for tantalum-base T-111 alloy.

Both tungsten and tungsten-25% rhenium are more creep resistant that T-111. The extrapolated data for a single test of the tantalum-base alloy Astar 811C indicates that at 2600°F (1427°C) the creep properties are comparable to tungsten. Although a comparison of these alloys on the basis of strength-to-weight ratios (Figure 101b) tends to minimize the difference between the tungsten and the tantalum alloys, the tungsten base materials still exhibit superior creep resistance.

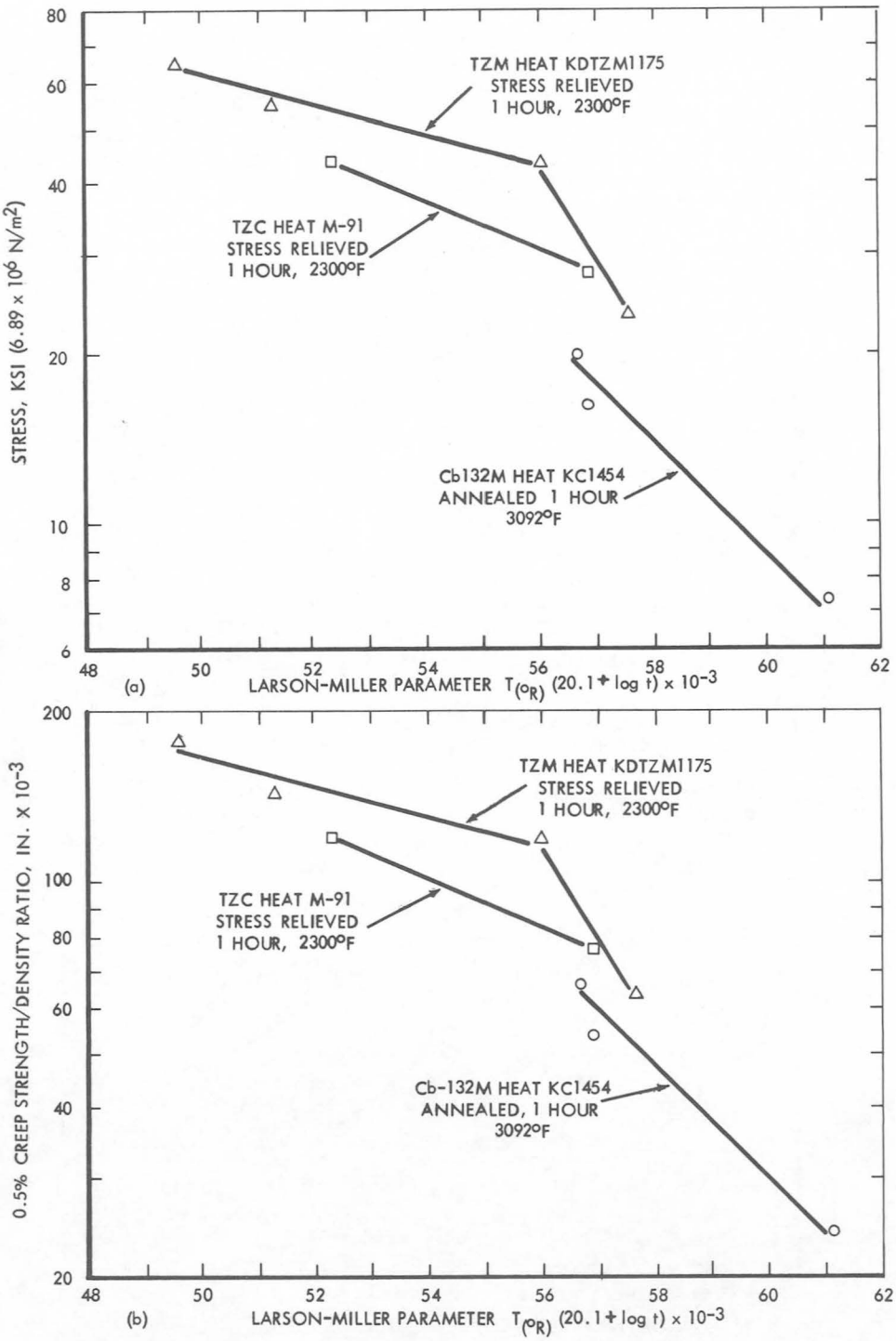


FIGURE 100 PARAMETRIC COMPARISON OF TZM FORGED DISK WITH TZC PLATE AND Cb 132M PLATE; a) 0.5% CREEP STRENGTH, b) 0.5% CREEP STRENGTH-TO-DENSITY RATIO.

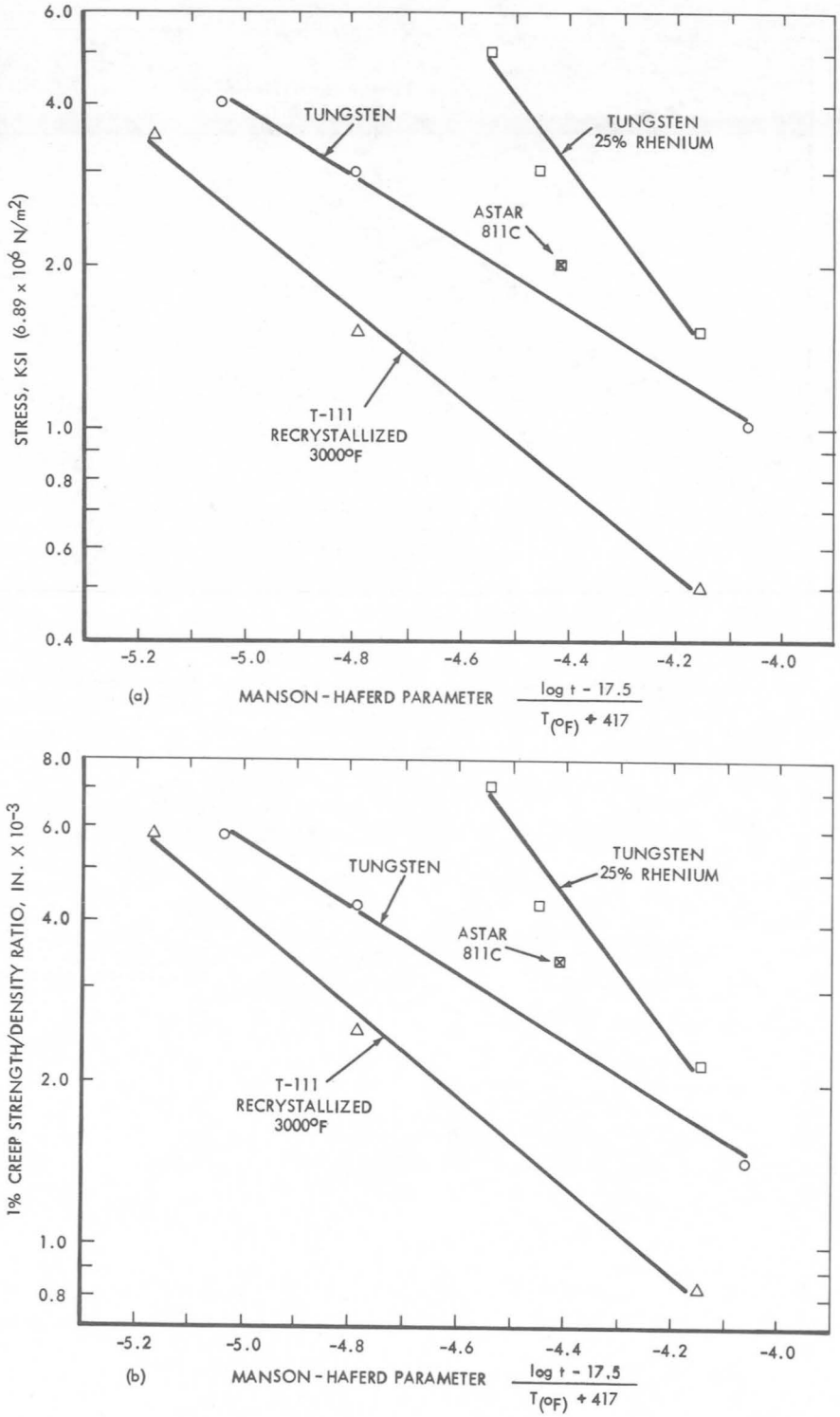


FIGURE 101 PARAMETRIC COMPARISON OF ASTAR 811C, ARC MELTED TUNGSTEN, TUNGSTEN-25% RHENIUM AND T-111 SHEET; a) 1% CREEP STRENGTH, b) 1% CREEP STRENGTH-TO-DENSITY RATIO.

VI SUMMARY AND CONCLUSIONS

Creep tests were conducted with the purpose of defining design properties for refractory metal alloys for times to 10,000 hours in a vacuum environment of less than 1×10^{-8} torr. The specific application would ultimately involve space electric power systems. The tests were performed in specially designed creep units which included methods for accurate temperature and extension measurements.

The following classes of alloys were evaluated in the program:

1. Columbium-base (AS-30, Cb132M),
2. Molybdenum-base (TZC, TZM, Cb-modified TZM),
3. Tantalum-base (T-111, T-222, Astar 811C), and
4. Tungsten-base (W, W-25Re, Sylvania A)

Considering the columbium and molybdenum-base materials as representing potential turbine alloys, the stress-relieved TZM and TZC possess the best creep resistance. Typical values indicated that 0.5% creep would be attained at 2000°F (1093°C) in 10,000 hours at a 20 ksi ($1.38 \times 10^8 N/m^2$) stress level.

A large number of creep tests were performed with the T-111 alloy which is a potential tubing material. Several heats were used to provide an indication of the inherent variability for materials with the same nominal composition. Although the data are limited, the Astar 811C showed the best creep resistance of the tantalum-base alloys.

The creep strengths of arc-melted and vapor-deposited tungsten were similar at 2800°F (1538°C). The tungsten-25% rhenium was superior to the arc-melted tungsten only at the lower stress values (longer test times). Sylvania A had the best creep resistance of the tungsten-base materials; however, the alloy was extremely brittle. Extensive post-test evaluations were made of the materials to define significant property or chemistry changes The tests indicated that prolonged heating in a vacuum environment of $< 1 \times 10^{-8}$ torr does not produce any significant interstitial contamination.

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APPENDIX I

Processing History of Test Materials

Table No.	Processing History Of:
	Columbium-Base
I	AS-30 C5
II	Cb-132M KC1454
	Molybdenum-Base
III	TZC M-80
IV	TZC M-91
V	TZC 4345
VI	TZM Disc 7502
VII	TZM Disc KDTZM-1175
VIII	TZM Bar 7463
IX	Columbium-Modified TZM 4305-4
	Tantalum-Base
X	T-111 70616
XI	T-111 65079
XII	T-222 AL-TA-43
XIII	T-222 Ta-43-SF-2
	Tungsten-Base
XIV	Arc-Melted Tungsten KC1357
XV	Tungsten-25% Rhenium 3.5-75002
XVI	Sylvania A

TABLE I

PROCESSING HISTORY OF AS-30 PLATE
Heat C5

Processing History:

1. Vacuum arc melt ingot 5.4" diameter.
2. Machine to 4.8" diameter.
3. Jacket in molybdenum.
4. Extrude 3.25:1 ratio at 2825°F (1552°C) to 4" x 1.625" sheet-bar.
5. Cross-roll at 2100°F (1149°C) to 0.790", argon atmosphere.
6. Acid etch to remove molybdenum jacket.
7. Abrasive saw to final width and length.

Hardness:

293 DPH (29Rc)

TABLE II

PROCESSING HISTORY OF Cb-132M PLATE

Heat KC1454

Processing History:

1. Electron-beam melt, electrode diameter 2-1/2", mold size 3-7/8"
2. Vacuum arc melt, then can in Mo-0.5Ti.
3. Extrude at 3130°F (1721°C) to 1-1/2" diameter.
4. Cross-roll from 2400°F (1316°C) in three passes yielding reductions of 20, 10, and 10%. Reheat between each pass. Final thickness 3/4".
5. Remove jacket, ship in as-rolled condition.

Hardness:

336 DPH (34 Rc)

TABLE III

PROCESSING HISTORY OF TZC PLATE
Heat M-80

Processing History:

1. Vacuum arc melt ingot 5.88" diameter.
2. Machine to 5" diameter.
3. Extrude 2.30:1 ratio at 3092°F (1700°C) to 4-1/8" x 2.22" plate.
4. Cross-roll on small mill (12" diameter) at 2925°F (1585°C) in 4-1/8" direction to 0.740", hydrogen atmosphere, 4% reduction per pass.
5. Grit blast and cut to final length with abrasive saw.

Hardness:

308 DPH (29 Rc)

TABLE IV

PROCESSING HISTORY OF TZC PLATE
Heat M-91

Processing History:

1. Vacuum arc melt ingot 5.88" diameter.
2. Machine to 5" diameter.
3. Extrude 2.30:1 at 3092°F (1700°C) to 4-1/8" x 2.22" plate.
4. Cross-roll on large mill (28" diameter) to produce relatively large degree of deformation per pass and a finishing temperature as low as 2372°F (1300°C).
5. Grit blast and cut to final length with abrasive saw.

Hardness:

335 DPH (34 Rc)

TABLE V

PROCESSING HISTORY OF TZC PLATE
Heat 4345

Processing History:

1. Machine vacuum arc melted ingot to 5.85" diameter.
2. Extrude to 3" diameter.
3. Heat treat in vacuum 3000°F (1649°C).
4. Machine to 2.4 - 2.8" diameter.
5. Upset forge 40% at 2400°F (1316°C).
6. Broad forge to 0.825" at 2400°F (1316°C).
7. Heat treat in vacuum 2400°F (1316°C), 1 hour.
8. Machine to 0.70".

Hardness:

319-373 DPH (28-36 Rc)

TABLE VI

PROCESSING HISTORY OF TZM FORGED DISC
Heat 7502

Processing History:

1. Vacuum arc melt ingot 11-1/2" diameter.
2. Machine to 10-3/4" diameter.
3. Extrude to 6-1/4" diameter.
4. Heat treat at 2700°F (1482°C).
5. Upset forge at 2200°F (1204°C).
6. Stress relieve at 2200°F (1204°C).

Hardness:

266-342 DPH (25-35 Rc)

TABLE IX

PROCESSING HISTORY OF COLUMBIUM-MODIFIED TZM
Heat 4305-4

Processing History:

1. 3-1/8" diameter machine casting.
2. Extrude to 1-3/8" diameter.
3. Recrystallize.
4. Swage to 5/8" diameter
5. Stress relieve 2500°F (1371°C), 1 hour.

Hardness:

278 DPH (27 Rc)

TABLE X

PROCESSING HISTORY OF T-111 SHEET
Heat 70616

Processing History:

1. Electron beam melt.
2. Arc-cast 5-1/2" ingot.
3. Forge to 1-1/2" thick sheet bar 2200°F (1204°C).
4. Vacuum anneal 2400°F (1316°C).
5. Warm roll 800°F (427°C) to 200 mil thick.
6. Vacuum anneal 2400°F (1216°C).
7. Cold roll to final thickness.
8. Vacuum anneal 2400°F (1316°C).

Hardness:

216-368 DPH (95 R_B - 37 Rc)

TABLE XI

PROCESSING HISTORY OF T-111 SHEET
Heat 65079

Processing History:

1. Forge 6-1/2" diameter arc-melted ingot to 1-1/2" sheet bar at 2200°F (1204°C).
2. Vacuum anneal 2800°F (1538°C).
3. Warm roll 800°F (427°C) to 1/4" thick.
4. Vacuum anneal 2800°F (1538°C).
5. Cold roll to final thickness.
6. Vacuum anneal (1×10^{-4} torr) 2800°F (1538°C), 1 hour.

Hardness:

236 DPH (20 Rc)

TABLE XII

PROCESSING HISTORY OF T-222 SHEET
Heat Al-Ta-43

Processing History:

1. Ingot 4-1/2" diameter x 4-1/2" long.
2. Side forge to 1-1/2" thick (final forging 1-1/2" x 4-1/2" x 5-1/2").
3. Roll to 5-1/2" direction to 0.700" thick.
4. Cut to 0.700" x 4" x 9" size, final rolling direction parallel to 9 " dimension.
5. Roll to 0.030" thickness.

Hardness:

405 DPH (41 Rc)

TABLE XIII

PROCESSING HISOTRY OF T-222 SHEET
Heat Ta-43-SF-2

Processing History:

1. Double consumable arc melt 4" diameter x 8" ingot.
2. Condition.
3. Dynapak broad forge to 1-5/8" thick.
4. Vacuum anneal 2750°F (1510°C), 2 hours.
5. Roll to 0.300" thick.
6. Vacuum anneal 2750°F (1510°C), 2 hours.
7. Cross-roll to 0.100" thick.
8. Vacuum anneal 2750°F (1510°C), 1 hour.
9. Roll to 0.060" thick.
10. Stress-relieve 2300°F (1260°C), 1 hour.
11. Finish roll to 0.040" thick.

Hardness:

339 DPH (34 Rc)

TABLE XIV

PROCESSING HISTORY OF TUNGSTEN SHEET
Heat KC1357

Processing History:

1. Extrude 4:1 ratio 3100°F (1705°C) TRW Inc.
2. Forge open die 2200°F (1204°C).
3. Roll
   a) Initial 2300°F (1260°C)
   b) Intermediate 1800°F (982°C)
   c) Final 1400°F (760°C)
4. Stress-relieve 1700°F (927°C).

Hardness:

487 DPH (48 Rc)

TABLE XV

PROCESSING HISTORY FOR TUNGSTEN-25% RHENIUM SHEET
Heat 3.5-75002

Processing History:

1. Stress relieve 0.055" sheet 1 hour 2375°F (1301°C).
2. Roll to 0.035".
3. Stress-relieve
   a) Small sheet - 2375°F (1301°C).
   b) Large sheet - 2550°F (1399°C).

Hardness:

639 DPH

TABLE XVI

PROCESSING HISTORY OF SYLVANIA "A" SHEET

Processing History:

1. Rolling slabs were made by isostatically pressing powder.
2. Roll slabs at 2732-3452°F (1500-1900°C) to 0.032". Total reduction 90%.
3. Intermediate annealing - none.
4. Final stress relief - five minutes at 2732°F (1500°C).
5. Trim sheet with abrasive saw and chemically clean.

Hardness:

579 DPH (54 Rc)

APPENDIX II

Chemical Analysis

Original Material

Table I - Columbium-Base AS-30 and Cb-132M
Table II - TZC
Table III - TZM and Cb Modified TZM
Table IV - T-111
Table V - T-222
Table VI - Tungsten, Tungsten-25% Rhenium, and Sylvania A

Post-Test Analysis

Table VII - Columbium-Base AS-30 and Cb-132M
Table VIII - Molybdenum-Base TZC
Table IX - Molybdenum-Base TZM
Table X - Tantalum-Base T-111
Table XI - Tungsten
Table XII - Tungsten-25% Rhenium

TABLE I

Columbium-Base

Element	Percent	
	AS-30 Heat C5	Cb-132M Heat KC1454
C	0.09	0.16
Cb	Balance	Balance
Co	0.010	0.010
Cu	0.010	0.010
Fe	0.010	0.010
H_2	0.0015	0.0004
Mn	0.010	0.010
Mo	0.020	4.72
N_2	0.010	0.0024
Ni	0.020	0.020
O_2	0.0060	0.0004
Pb	0.005	0.001
Si	0.020	0.0015
Ta	-	19.7
Ti	0.030	0.010
V	0.015	0.015
W	21.0	15.6
Zr	1.04	2.10

TABLE II

TZC

Element	Percent Heat M-80	Heat M-91	Heat 4345
Al	0.008	0.008	0.008
C	.127	.113	0.075
Cb	0.010	-	0.010
Co	0.0025	0.0025	0.0025
Cr	0.0025	0.0025	0.0025
Cu	0.0025	0.0025	0.0025
Fe	0.005	0.005	0.005
H_2	0.0010	0.0010	0.0002
Mg	0.0025	0.0025	0.0025
Mn	0.005	0.005	0.005
Mo	Balance	Balance	Balance
N_2	0.0018	0.0034	0.0009
Ni	0.005	0.005	0.005
O_2	0.0041	0.0037	0.0019
Pb	0.005	0.005	0.005
Si	0.005	0.005	0.005
Sn	-	0.010	0.010
Ti	1.02	1.17	1.19
V	0.005	0.005	0.005
Zr	0.17	0.27	0.16

TABLE III

TZM

Element	Percent			
	Heat 7502	Heat KDTZM-1175	Heat 7463	Cb Mod.
Al	0.008	0.008	-	0.008
C	0.010	0.035	0.016	0.015
Cb	0.010	0.010	-	1.00
Co	0.0025	0.0025	-	0.0025
Cr	0.0025	0.0025	-	0.0025
Cu	0.0025	0.0025	-	0.0025
Fe	0.005	0.005	0.001	0.005
H_2	0.0007	0.0009	0.0001	0.0004
Mg	0.0025	0.0025	-	0.0025
Mn	0.005	0.005	-	0.005
Mo	Balance	Balance	Balance	Balance
N_2	0.0010	0.0031	0.0001	0.0030
Ni	0.005	0.005	0.001	0.005
O_2	0.002	0.0034	0.0002	0.0039
Pb	0.005	0.005	-	0.005
Si	0.005	0.005	0.001	0.005
Sn	0.010	0.010	-	0.010
Ti	0.44	0.61	0.48	0.48
V	0.005	0.005	-	0.005
Zr	0.10	0.12	0.080	0.095

TABLE IV

T-111

Element	Percent				
	Heat 70616	Heat 65079	Heat D-1670	Heat MCN02A065	Heat D-1102
Al	0.005	-	-	10	-
C	0.0044	0.003	0.001	0.004	0.0034
Cb	0.010	0.05	0.0130	0.06 - 0.07	0.0140
Co	0.0025	0.005	0.001	0.0005	0.001
Cr	0.0025	0.0010	-	0.001	-
Cu	0.0025	0.0020	-	0.002	-
Fe	0.005	0.005	0.002	0.002	0.001
H_2	0.0006	0.0004	0.0005	0.00026	0.0003
Hf	2.30	2.30	2.17	2.3 - 3.3	2.21 - 2.32
Mg	0.0025	-	-	-	-
Mn	0.005	-	-	-	-
Mo	0.010	0.015	0.0150	0.001	0.013
N_2	0.0020	0.005	0.0020	0.0020	0.0013
Ni	0.005	0.005	0.001	0.001	0.001
O_2	0.0055	0.013	0.0072	0.010	0.0020
Pb	-	-	-	-	-
Si	0.005	0.002	-	0.002	-
Sn	0.010	-	-	-	-
Ta	Balance	Balance	Balance	Balance	Balance
Ti	0.010	0.002	-	0.002	-
V	0.005	0.002	0.001	0.001	0.001
W	8.50	8.70	7.9	8.55 - 8.65	7.78 - 8.20

TABLE V

T-222

Element	Al-TA-43	Ta-43-SF-2
Al	0.005	0.005
C	0.012	0.012
Cb	0.010	0.010
Co	0.0025	0.0025
Cr	0.0025	0.0025
Cu	0.0025	0.0025
Fe	0.005	0.005
H_2	0.0011	0.0007
Hf	2.93	2.53
Mg	0.0025	0.0025
Mn	0.005	0.005
Mo	0.010	0.010
N_2	0.0026	0.003
Ni	0.005	0.005
O_2	0.0035	0.0038
Pb	-	0.005
Si	0.005	-
Sn	0.010	0.010
Ti	-	0.010
V	0.005	-
W	9.57	8.77

TABLE VI

Tungsten, Tungsten-25% Rhenium, Sylvania A

Element	Percent		
	Tungsten KC 1357	W-25% Re 3.5-75002	Sylvania A
Al	0.005	0.002	0.002
C	0.0058	0.007	0.030
Cb	0.010	0.010	0.010
Co	-	0.010	0.001
Cr	0.010	0.0025	0.001
Cu	0.001	0.001	0.001
Fe	0.0025	0.0025	0.0025
Hf	-	-	0.52
H_2	0.0003	0.0013	0.0003
Mg	0.001	0.001	0.001
Mn	0.001	0.0015	0.0015
Mo	0.010	0.010	0.010
N_2	0.0016	0.0007	0.0017
Ni	0.001	0.010	0.001
O_2	0.0009	0.0061	0.002
Pb	0.002	0.002	0.002
Re	-	24.88	-
Si	0.0025	0.0025	0.0025
Sn	0.004	0.004	0.004
Ti	0.002	0.002	0.002
V	0.003	0.003	0.003
W	Balance	Balance	Balance

TABLE VII

Columbium-Base

Element	Percent	
	AS-30 Test B-6 1193 Hours 2000°F (1093°C)	Cb-132M Test B-13 568 Hours 2056°F (1225°C)
C	0.07	0.15
Cb	Balance	Balance
Co	0.001	0.001
Cu	0.001	0.005
Fe	0.003	0.003
H_2	0.0009	0.0002
Mn	0.010	0.001
Mo	0.001	4.73
N_2	0.0017	0.0020
Ni	0.001	0.001
O_2	0.0040	0.0003
Pb	0.001	0.001
Si	0.003	0.0015
Ta	-	19.5
Ti	-	0.001
V	0.010	0.001
W	20.48	15.6
Zr	1.16	2.40

TABLE VIII

TZC Heat M-80

Element	Percent		
	Test B-5A, B, 1985 Hours 2000°F (1093°C)	Test B-9 16,002 Hours 2000°F (1093°C)	Test B-12 14,239 Hours 2056°F (1225°C)
Al	0.008	0.008	0.008
C	0.15	0.050	0.11
Cb	0.010	0.010	0.010
Co	0.0025	0.0025	0.0025
Cr	0.0025	0.0025	0.0025
Cu	0.0025	0.0025	0.0025
Fe	0.005	0.005	0.005
H_2	0.0005	0.0001	0.0002
Mg	0.0025	0.0025	0.0025
Mn	0.005	0.005	0.005
Mo	Balance	Balance	Balance
N_2	0.0024	0.0010	0.0007
Ni	0.005	0.005	0.005
O_2	0.0023	0.0023	0.0027
Pb	0.005	0.005	0.005
Si	0.005	0.005	0.005
Sn	0.010	0.010	0.010
Ti	1.02	1.10	1.15
V	0.005	0.005	0.005
Zr	0.16	0.16	0.17

TABLE IX

TZM Heat 7502

Element	Test B-3 10,048 Hours 2000°F (1093°C)
Al	0.008
C	0.012
Cb	0.010
Cr	0.0025
Cu	0.0025
Fe	0.005
H_2	0.0007
Mg	0.0025
Mn	0.005
Mo	Balance
N_2	0.0040
Ni	0.005
O_2	0.0031
Pb	0.005
Si	0.005
Sn	0.010
Ti	0.50
V	-
Zr	0.05

TABLE X

T-111 Heat 70616

Element	Percent	
	Test S-16 1675 Hours 2600°F (1426°C)	Test S-19 4870 Hours 2200°F (1204°C)
Al	0.005	-
C	0.005	0.002
Cb	0.010	-
Co	0.0025	0.005
Cr	0.0025	-
Cu	0.0025	-
Fe	0.005	0.005
H_2	0.0010	0.0003
Hf	2.40	2.32
Mg	0.0025	-
Mn	0.005	-
Mo	0.010	0.020
N_2	0.0040	0.0006
Ni	0.005	0.005
O_2	0.0010	0.0028
Pb	0.005	-
Si	0.002	-
Sn	0.010	-
Ta	Balance	Balance
Ti	0.010	-
V	0.005	0.005
W	8.45	8.79

TABLE XI

Tungsten Heat KC 1357

Element	Percent Test S-5 32 Hours 3200°F (1760°C)	Test S-9 3886 Hours 3200°F (1760°C)
Al	0.005	0.002
C	0.0033	0.004
Cb	0.010	0.010
Co	-	0.001
Cr	0.010	0.001
Cu	0.001	0.001
Fe	0.0025	0.0025
H_2	0.0005	0.0003
Mg	0.015	0.001
Mn	0.0015	0.0015
Mo	0.010	0.010
N_2	0.0010	0.0006
Ni	0.010	0.001
O_2	0.0010	0.0037
Pb	0.002	0.002
Si	0.0025	0.0025
Sn	0.004	0.004
Ti	0.002	0.002
V	0.003	0.003
W	Balance	Balance

TABLE XII

Tungsten-25% Rhenium - Heat 3.5-75002

Element	Test S-4 97 Hours 3200°F (1760°C)	Test S-8 1306 Hours 3200°F (1760°C)
Al	0.005	0.002
C	0.006	0.008
Cb	0.010	0.010
Co	-	0.001
Cr	0.010	0.001
Cu	0.010	0.001
Fe	0.010	0.0025
H_2	0.0005	0.0011
Mg	0.005	0.001
Mn	0.010	0.0015
Mo	0.020	0.010
N_2	0.0010	0.0005
Ni	0.010	0.001
O_2	0.0010	0.0039
Pb	0.005	0.002
Re	24.90	24.56
Si	0.015	0.0025
Sn	0.005	0.004
Ti	0.005	0.002
V	0.015	0.003
W	Balance	Balance

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