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

**MID-CONTRACT REPORT**

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**TRW EQUIPMENT LABORATORIES**  
CLEVELAND, OHIO

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## MID-CONTRACT REPORT

For

17 March 1967 to 1 August 1967

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

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Prepared for:

National Aeronautics and Space Administration  
Contract No. NAS 3-9439

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August 1967


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FOREWORD

The work described herein is being performed by TRW Inc. under the sponsorship of the National Aeronautics and Space Administration under Contract NAS 3-9439. The purpose of this study is to obtain design creep data on refractory metal alloys for use in advanced space power systems.

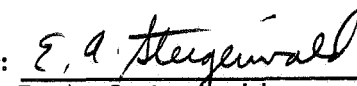
The program is administered for TRW Inc. by E. A. Steigerwald, Program Manager, J. C. Sawyer is the Principal Investigator, and R. R. Ebert contributed to the program. The NASA technical director is Paul E. Moorhead.

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ABSTRACT

Molybdenum-base TZC and TZM alloys and tantalum-base T-111 and Astar 811C are being creep tested in a vacuum environment of  $1 \times 10^{-8}$  Torr. Test temperatures range from 1600 to 2600°F (871 to 1427°C) and stresses from 0.5 to 65 Ksi ( $3.44 \times 10^6$  to  $4.48 \times 10^8$  N/m<sup>2</sup>). Test parameters were selected to provide 0.5-1.0% creep in 5,000 to 15,000 hours.

Test results with molybdenum-base TZC and TZM show the effects of variations in chemistry and method of processing.

Tests with tantalum-base T-111 reveal that significant variations in creep strength can occur between heats of materials. Data are presented to suggest that low creep strength of T-111 alloy may be the result of fine grain size.

Comparison of tantalum-base Astar 811C with T-111 shows the superior creep properties of the Astar 811C alloy.

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## I INTRODUCTION

Space electric power systems depend upon the use of refractory metals in a variety of component areas. A critical property in the design of both Rankine and Brayton systems is the long-time creep strength at elevated temperatures. While turbine, capsule, and clad components of space power systems are not necessarily exposed to the vacuum of space, they do operate in environments of helium, metal vapor, or liquid metals where the partial pressure of reactive gases is extremely low. Consequently, to obtain meaningful creep data it is necessary to perform tests in a vacuum environment of  $< 1 \times 10^{-8}$  torr which substantially eliminates interstitial contamination over long periods of time.

The work of this program is a continuation of a previously established contract, (NAS 3-2545). The common objective of both programs is the generation of nominally 10,000 hour creep data on selected refractory alloys which have potential for use in advanced space electric power systems. The materials examined are in the form of rolled sheet and forged or rolled plate, with the sheet being representative of material for tubing and capsule applications and the plate for turbine components. Tantalum-base sheet alloys T-111, Astar 811C and Ta-10W and molybdenum-base turbine alloys TZC and TZM are the materials of current interest.

The use of encapsulated radioactive isotopes as a space-power heat source has generated a need for creep data under conditions of increasing stress and decreasing temperature. Therefore, some of the creep tests in this program are being made with varying loads and/or varying temperatures. It is recognized that stresses in these capsules are biaxial, but more data is needed on the effect of varying stress and temperature than can be obtained with existing biaxial creep test apparatus.

## II MATERIALS AND PROCEDURE

The composition of the various alloys discussed in this report are presented in Table 1. The material form, the range of test temperatures, and the heat treatments are given in Table 2. A detailed review of the available processing histories of the alloys currently under evaluation is presented in Appendix I.

The geometries of the test specimens are shown in Figures 1 and 2. The particular orientation of the specimens with respect to the working direction is given below:

<u>Material Form</u>	<u>Specimen Axis Parallel To</u>
Disc forging	Radius
Plate	Extruding direction
Sheet	Rolling direction (except where indicated)

The TZM alloy was obtained from two different sources. One lot of material, designated as Heat 7502, was purchased from Climax Molybdenum of Michigan in the form of 11 inch diameter disc forgings. The second lot of material (Heat KDTZM-1175 Disc #3) was a section of a disc forging obtained from AiResearch. The latter material was processed by Universal Cyclops to produce improved creep resistance<sup>(1)\*</sup> through the development of a fine carbide dispersion. A carbon level above 0.02% is necessary in order to produce this effect.

To date two different heats of TZC have been tested. Heat M-91 was produced by the General Electric Co. by rolling a 2" x 4" sheet bar at 2372°F (1300°C) with relatively large reductions on each pass. TZC Heat 4345 was prepared by Climax Molybdenum of Michigan by broad forging 3 inch diameter extruded stock at 2400°F (1316°C).

Six heats of T-111 alloy were obtained from two different sources. Four heats were produced by Wah Chang Corporation (Heats 70616, 65079, 65080, and MCN 02A065), and two were obtained from Fansteel Corporation (Heats D-1670 and D-1102). All heats are being evaluated after recrystallization at 3000°F (1649°C) for one hour.

The specimen of Astar 811C, a relatively new tantalum-base alloy developed under Contract NAS 3-2542, was obtained from the Westinghouse Electric Corporation through NASA Lewis. This alloy was supplied in the form of 0.030" thick sheet in the as-rolled condition.

-----  
\* Numbers in parentheses refer to references in the Bibliography

Table 1

Chemical Composition of Alloys Being Evaluated in Creep Program (Weight %)<sup>(1)</sup>

Material	W	Re	Cb	Mo	Ta	Hf	C	Ti	Zr	ppm		
										N <sub>2</sub>	O <sub>2</sub>	H <sub>2</sub>
TZM (Heat 7502) (Heat KDTZM-1175)				Bal.			.010	.51	.091	100	20	7
				Bal.			.035	.61	.120	43	34	9
TZC (Heat M-91) (Heat 4345)				Bal.			.046	1.17	.270	34	37	10
				Bal.			.075	1.19	.16	9	19	2
T-111 (Heat 70616) (Heat 65079) (Heat 65080) (Heat MCN02A065) (Heat D-1102) (Heat D-1670)	8.5				Bal.	2.30	.0044			20	55	6
	8.7				Bal.	2.30	.003			50	130	4(2)
	8.9				Bal.	1.9	.003			12	130	4(2)
	8.6				Bal.	1.95	.004			20	100	3(2)
	7.9				Bal.	2.28	.003			34	20	3(2)
	7.9				Bal.	2.17-2.44	◀.001			20	72	◀5(2)
Astar 811C	8.0	1.0			Bal.	0.7	.250			-	-	-(3)

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

Table 2

## Summary of Material Variables Being Evaluated in Creep Program

Material	Form	Test Temperature	Test Condition
TZM (Heat 7502)	"Pancake" Forging	2200°F (1093°C)	Stress relieved 2200°F (1204°C)
(Heat KDTZM-1175)	"Pancake" Forging	1600-2000°F (871-1093°C)	Stress relieved 2300°F (1260°C), 1 hour
TZC (Heat M-91)	3/4" Plate	1800-2000°F (982-1093°C)	Recrystallized 3092°F (1700°C), 1 hour
Heat 4345	3/4" Plate		Stress Relieved 2300-2500°F (1260-1371°C), 1 hour
T-111 (Heat 70616) (Heat D-1670) (Heat 1102) (Heat 65079) (Heat MCN02A065) (Heat 65080)	0.030" Sheet	1800-2600°F (982-1427°C)	Recrystallized 2600°F (1426°C) and 3000°F (1649°C), 1 hour
Astar 811C	0.030" Sheet	2600°F (1427°C)	Recrystallized 3600°F (1982°C), 1/2 hour

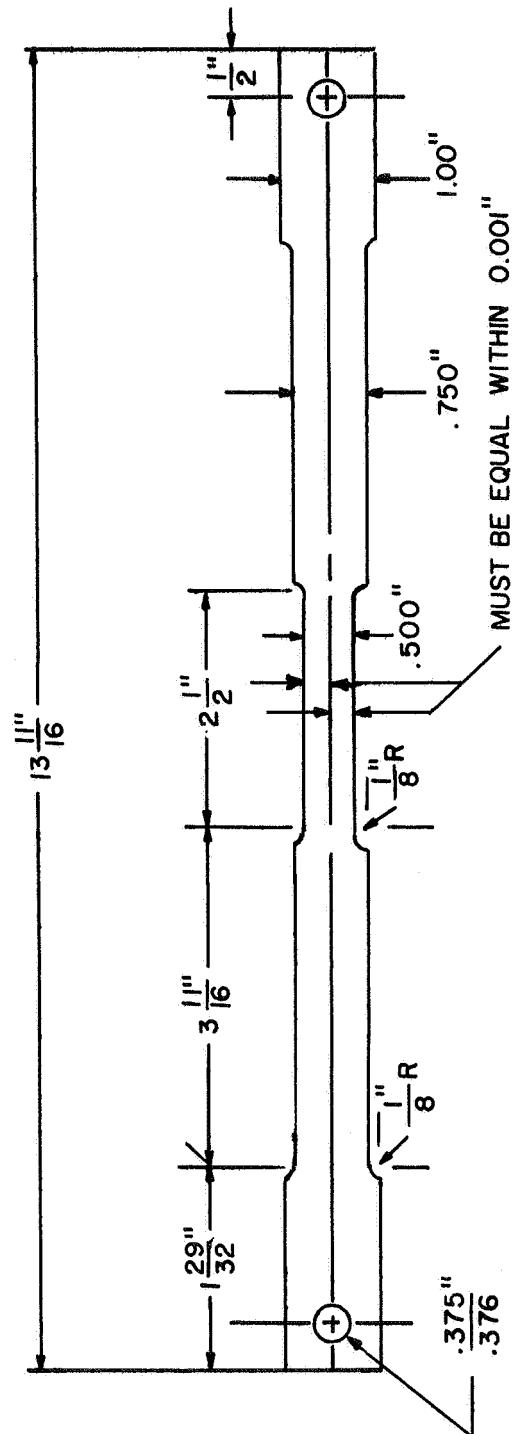


FIGURE 1 CREEP SPECIMEN USED FOR SHEET STOCK



FIGURE 2 CREEP SPECIMEN USED FOR PLATE STOCK

The creep test procedure involves obtaining a vacuum of  $5 \times 10^{-10}$  torr or better at room temperature, then heating the specimen at such a rate that the pressure never rises above  $1 \times 10^{-6}$  torr. The pre-test heat treatments are performed in situ, prior to load application. After heat treatment the specimens are cooled to 600°F (316°C) or lower and then reheated to the test temperature which is maintained for two hours prior to loading in order to insure thermal and structural equilibrium in the test specimen. During testing, the pressure is always less than  $1 \times 10^{-8}$  torr and decreases with test time.

Specimen extension is determined over a two inch gage length by using an optical extensometer to measure the distance between two scribed reference marks with an accuracy of 50 microinches. The program plan involves testing plate and forged alloys at temperatures between 1600 and 2250°F (871 and 1235°C) until 1% total extension is achieved. The tantalum-base materials are being evaluated at 1800 to 2600°F (982 to 1427°C) to elongations of approximately 1 to 5%. In most cases the applied stress levels were selected with the goal of obtaining creep data over total test times between 5000 and 15,000 hours.

### III RESULTS AND DISCUSSION

Results of the creep tests completed, in progress, or initiated during the first four and one-half months of the program are graphically displayed with percent elongation plotted as a function of test time. Reference marks placed on each curve indicate the chamber pressure at various intervals during the test.

#### Molybdenum-Base TZC and TZM

Three tests were conducted on Heat M-91 molybdenum-base TZC alloy. Results of these tests plotted in Figure 3, show a decreasing creep rate with time, indicative of a time dependent strengthening process. Post-test tensile data obtained from the gage section of specimen B-20 (Table 3), which had been creep tested for 12,800 hours at 2200°F (1093°C) and 20 Ksi ( $1.38 \times 10^8$  N/m<sup>2</sup>), show a significant increase in yield strength as a result of the 1% creep strain. This observed strengthening is consistent with the decrease in creep rate which occurred during testing.

Table 3

Tensile Data for TZC Heat M-91 Recrystallized 3092°F  
(1700°C) One Hour

	<u>Ultimate Strength Ksi</u>	<u>0.2% Offset Yield Strength Ksi</u>	<u>Elongation %</u>	<u>Reduction Area %</u>
Before Test	85.0	49.0	7.0	7.0
After Test	88.2	57.0	9.4	7.8

Two creep tests of molybdenum-base TZC Heat 4345 were conducted at 2000°F (1093°C) and 22 Ksi ( $4.52 \times 10^8$  N/m<sup>2</sup>). The singular difference between these two tests was in the stress relief temperatures of 2400 and 2500°F (1316 and 1371°C). The results shown in Figure 4 indicate that the two stress relief treatments provide no significant difference in creep behavior.

The 0.5% creep life for current and previous tests on the two heats of TZC are given in Table 4. Also given are Larson-Miller parameters based on a constant of 14.3 determined by computer fit of the data. These data, plotted in Figure 5, show that Heat M-91 recrystallized at 3092°F or stress relieved at 2500°F had creep properties which were slightly below those obtained with the other variables examined.

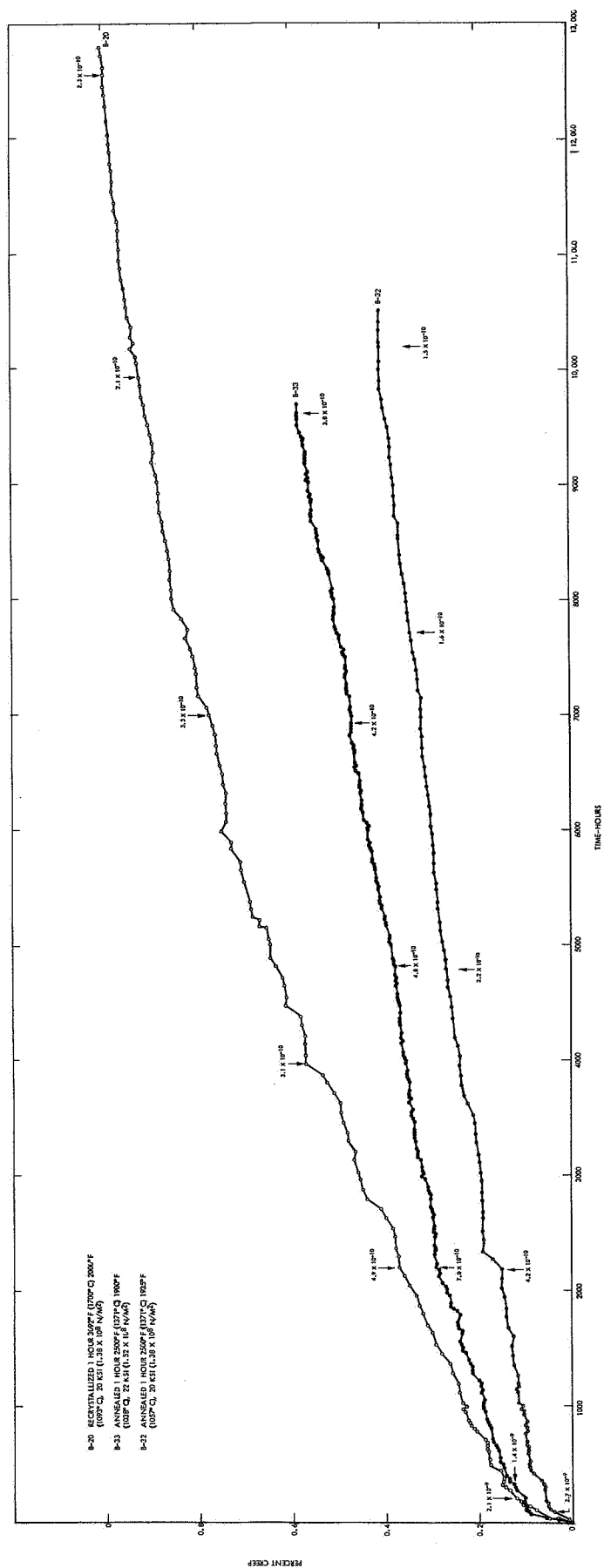


FIGURE 3. CREEP TEST DATA FOR MOLYBDENUM BASE TZC HEAT M-91 TESTED IN VACUUM ENVIRONMENT  $< 1 \times 10^{-8}$  TORR

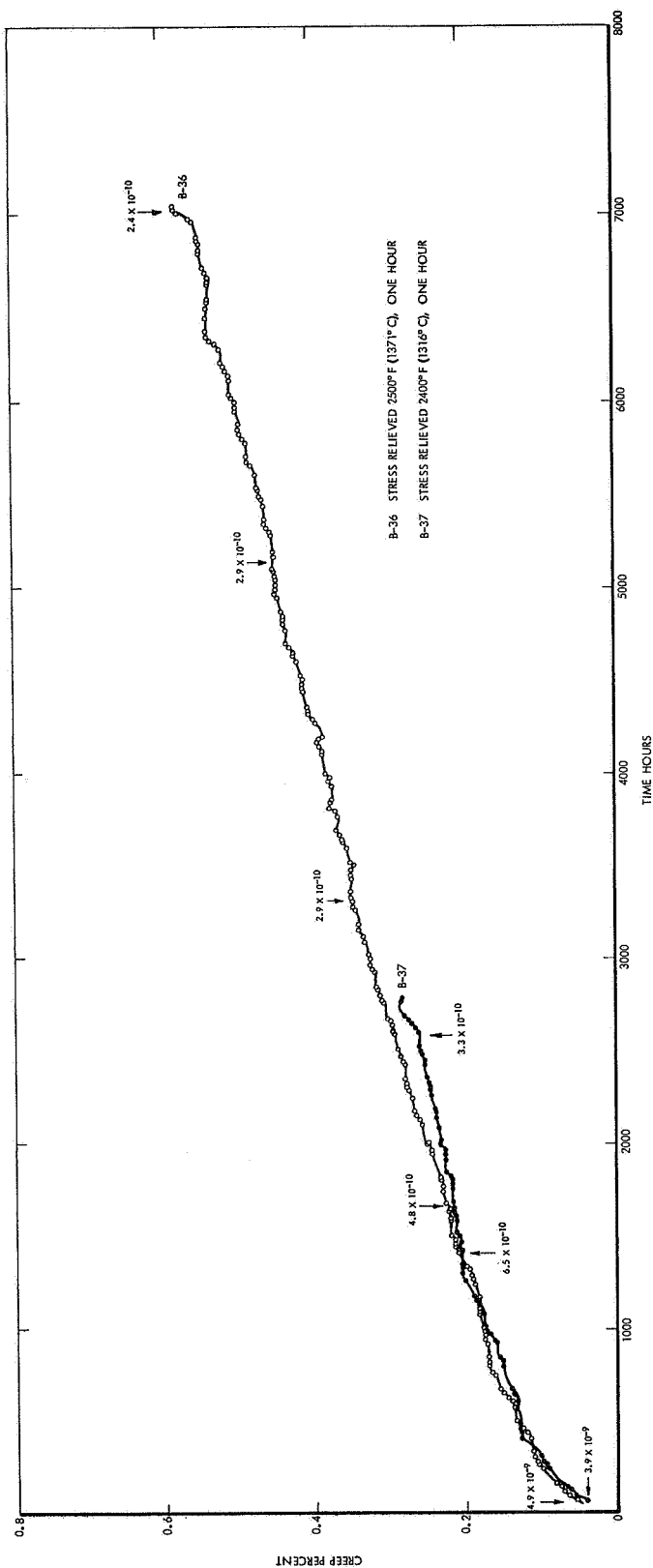


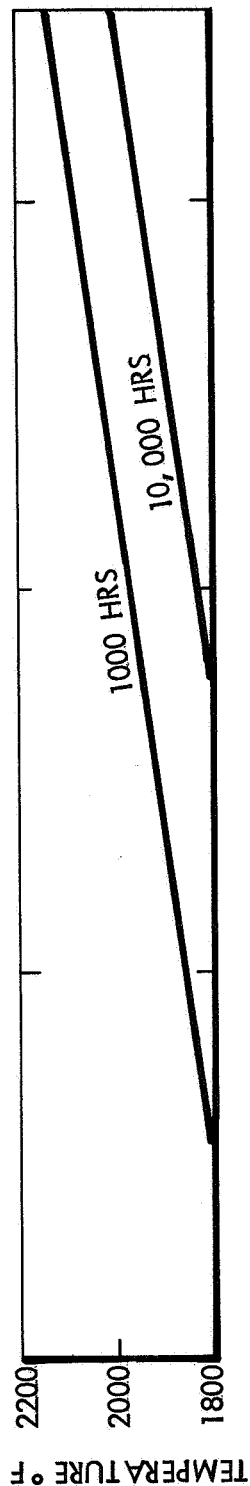
FIGURE 4. CREEP TEST DATA FOR TZC HEAT 4345 TESTED AT 2000°F (1093°C) AND 22 KSI (1.52 X 10<sup>8</sup> N/M<sup>2</sup>) IN VACUUM ENVIRONMENT < 1 X 10<sup>-8</sup> TORR

Table 4

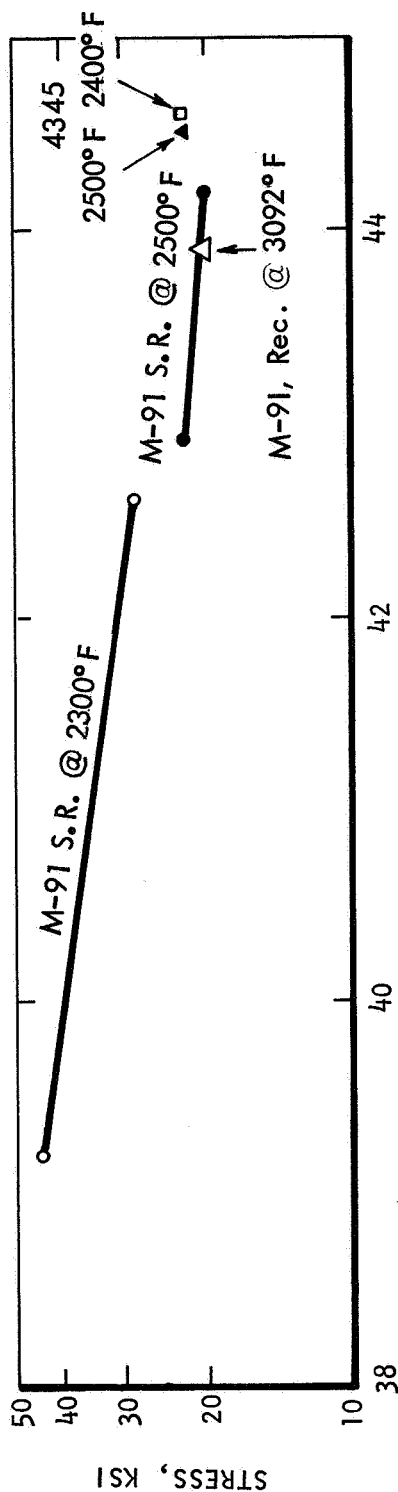
0.5% Creep Test Data for Molybdenum-Base TZC  
Heats M-91 and 4345

Specimen No.	Test Temperature		Ksi	Stress N/m <sup>2</sup> x 10 <sup>8</sup>	Hours for 0.5% Creep	Larson-Miller Parameter T °R(14.3+logt)x10 <sup>-3</sup>
	°F	°C				
<u>Heat M-91, Recrystallized 3092°F (1700°C), 1 Hour</u>						
B-20	2000	1093	20	1.38	3650	43.9
<u>Heat M-91, Stress-Relieved 2300°F (1260°C), 1 Hour</u>						
B-19	1800	982	44	3.03	1075	39.2
B-28	2000	1093	28	1.93	1100	42.6
<u>Heat M-91, Stress-Relieved 2500°F (1371°C), 1 Hour</u>						
B-30	2200	1204	22	1.52	70	42.9
B-32	1935	1057	20	1.38	14,400*	44.2
B-33	1900	1038	22	1.52	7720	42.9
<u>Heat 4345, Stress-Relieved 2500°F (1371°C), 1 Hour</u>						
B-36	2000	1093	22	1.52	5940	44.5
<u>Heat 4345, Stress-Relieved 2400°F (1316°C), 1 Hour</u>						
B-37	2000	1093	22	1.52	6500*	44.6

\* Extrapolated Values



- Heat M-91, Stress Relieved 2300°F (1260°C), One Hour
- Heat M-91, Stress Relieved 2500°F (1371°C), One Hour
- ▲ Heat M-91, Recrystallized 3092°F (1700°C), One Hour
- ▲ Heat 4345, Stress Relieved 2500°F (1371°C), One Hour
- Heat 4345, Stress Relieved 2400°F (1318°C), One Hour



LARSON-MILLER PARAMETER  $T^{\circ}R (14.3 + \log t) \times 10^{-3}$

FIGURE 5. LARSON-MILLER PLOT OF 0.5% CREEP DATA FOR TZC HEATS M-91 AND 4345 TESTED IN VACUUM ENVIRONMENT  $< 1 \times 10^{-8}$  TORR.

Three tests of molybdenum-base TZM were made using specimens prepared from forged disc Heats 7502 and KDTZM-1175. The creep data for these three tests given in Figure 6 clearly show the superior creep strength of Heat KDTZM-1175 at 1800°F (982°C) and 44 Ksi ( $3.03 \times 10^8$  N/m<sup>2</sup>). The new test B-38 with Heat KDTZM-1175 has not progressed far enough to extrapolate the curve to longer times. The post-test room temperature tensile strength of the gage section of specimen B-25, Table 5, is not significantly different from the strength of the original material. This is consistent with the creep results, which showed no tendency for a decreasing creep rate due to strengthening during the test.

The TZM 0.5% creep life and Larson-Miller parameters (based on a constant of 14.3) are presented in Table 6. This particular constant was chosen to allow a comparison of creep data for TZM and TZC. The parametric comparison, Figure 7, shows that TZM Heat KDTZM-1175, stress relieved at 2300°F (1260°C) for one hour has the best creep resistance, surpassing that of TZC Heat M-91. TZM Heat 7502, stress relieved at 2200°F (1204°C) one hour exhibits intermediate properties, being more creep resistant than TZC only at higher stresses. Kovacevich<sup>(2)</sup> reported that at a stress of 41.5 Ksi ( $2.86 \times 10^8$  N/m<sup>2</sup>) and a temperature of 1900°F (1038°C) a specimen from a TZM disc forging stress relieved at 2300°F (1260°C) for one hour produced 0.5% creep in 1000 hours. Comparison of these data with Figure 7 shows that the creep strength of the TZM used by Kovacevich is similar to that of TZM Heat 7502. It must be noted however, that the tests of Kovacevich were made at a pressure of  $1 \times 10^{-4}$  -  $1 \times 10^{-5}$  torr as compared to  $1 \times 10^{-8}$  torr used in the current studies. Unfortunately the available data are insufficient to establish the effect of the higher environmental pressure upon creep life.

Following 10,152 hours of test, the surface of specimen B-25 (TZM Heat KDTZM-1175) which had thermally etched during testing, was examined at 15,000X. The surface, shown in Figure 8, reveals fine striations suggesting that creep proceeds by slip of the bulk material. The fine pits appearing on the surface are characteristic of all areas examined.

#### Tantalum-Base T-111

Various heats of tantalum-base T-111 are being creep tested following a 3000°F (1649°C) one hour anneal in situ. The tests completed or in progress during the reporting period are tabulated below, and the elongation versus time data are plotted in Figures 9 thru 14.



Table 5Tensile Data for TZM Heat KDTZM-1175  
Stress Relieved 2300°F (1260°C), 1 Hour

	<u>Ultimate Strength Ksi</u>	<u>0.2% Offset Yield Strength Ksi</u>	<u>Elongation %</u>	<u>Reduction Area %</u>
Before Test	120	111	17.4	29.1
After Test	121.5	109.6	14.7	29.6

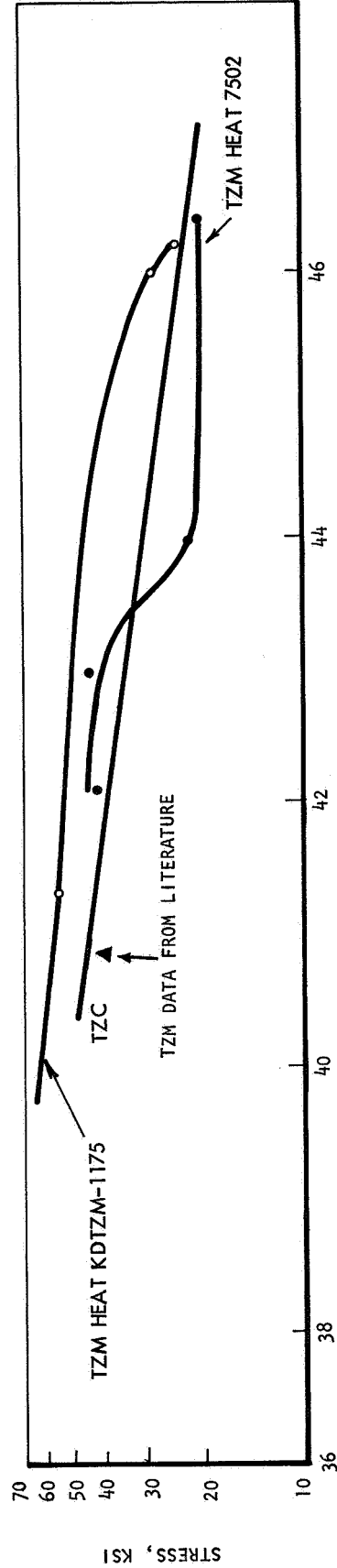
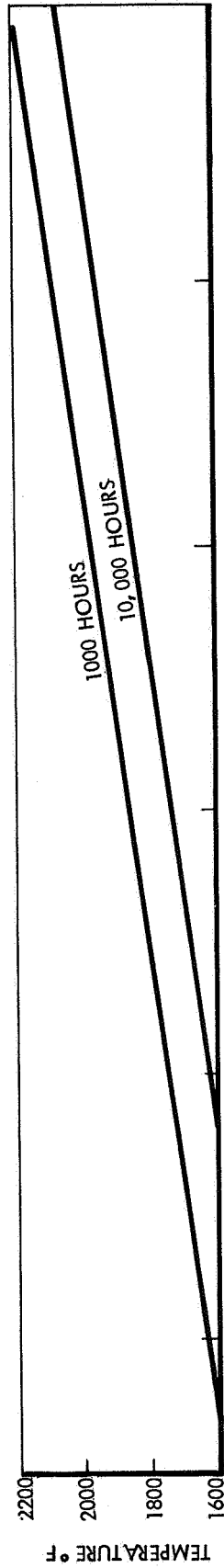
Table 6

0.5% Creep Test Data for Molybdenum-Base TZM  
Heats 7502 and KDTZM-1175

Heat	Specimen No.	Heat Treatment	Test Temperature °F	Test Temperature °C	Stress Ksi	Stress N/m <sup>2</sup>	Hours to 0.5% Creep	Larson-Miller Parameter $T^{\circ}R(14.3+\log t) \times 10^{-3}$
7502	B-1	2200°F - 1 hour	2000	1093	12	$8.27 \times 10^7$	605	42.0
	B-3	2200°F - 1 hour	2000	1093	10	$6.89 \times 10^7$	14,200*	45.4
	B-29	2200°F - 1 hour	2000	1093	41	$2.82 \times 10^8$	115	40.2
	B-35	2200°F - 1 hour	1800	982	44	$3.03 \times 10^8$	7000	41.0
1175	B-16	2300°F - 1 hour	1855	1013	23.4	$1.61 \times 10^8$	62,500*	44.2
	B-18	2300°F - 1 hour	1600	871	55	$3.79 \times 10^8$	60,000*	39.3
	B-21	2300°F - 1 hour	1600	871	65	$4.48 \times 10^8$	9600*	37.7
	B-25	2300°F - 1 hour	1800	982	44	$3.03 \times 10^8$	50,000*	42.9
	B-38	2300°F - 1 hour	2000	1093	22	$1.51 \times 10^8$	**	-

\* Extrapolated Values

\*\* Test time too short to extrapolate



LARSON-MILLER PARAMETER  $T^{\circ}R (14.3 + \log t) \times 10^{-3}$

FIGURE 7. PARAMETER COMPARISON OF STRESS RELIEVED TZM HEATS 7502 AND KDTZM-1175 WITH STRESS RELIEVED TZC HEATS M-91 AND 4345

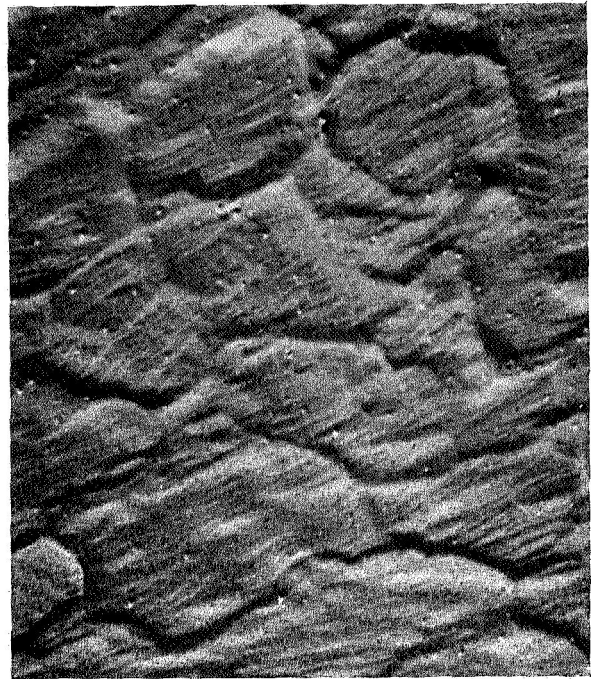
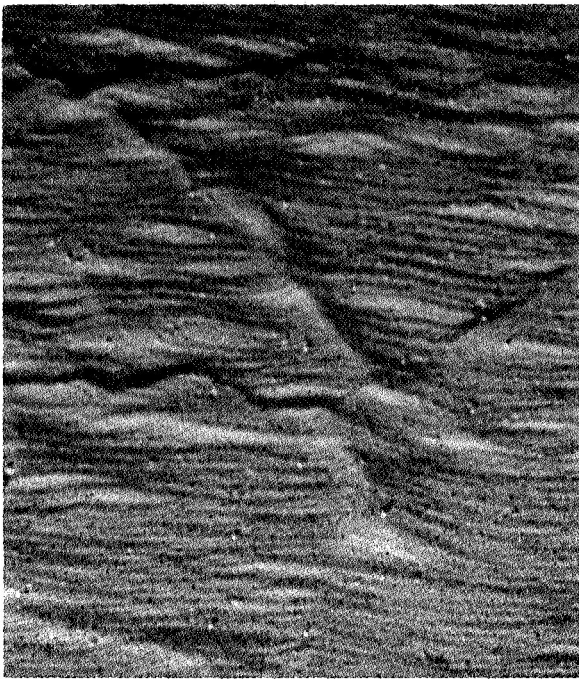


FIGURE 8. SLIP LINES IN THERMALLY ETCHED SURFACE OF SPECIMEN B-25 AFTER 10, 152 HOURS OF TEST. MAGNIFICATION 15,000X. TZM KDTZM-1175.

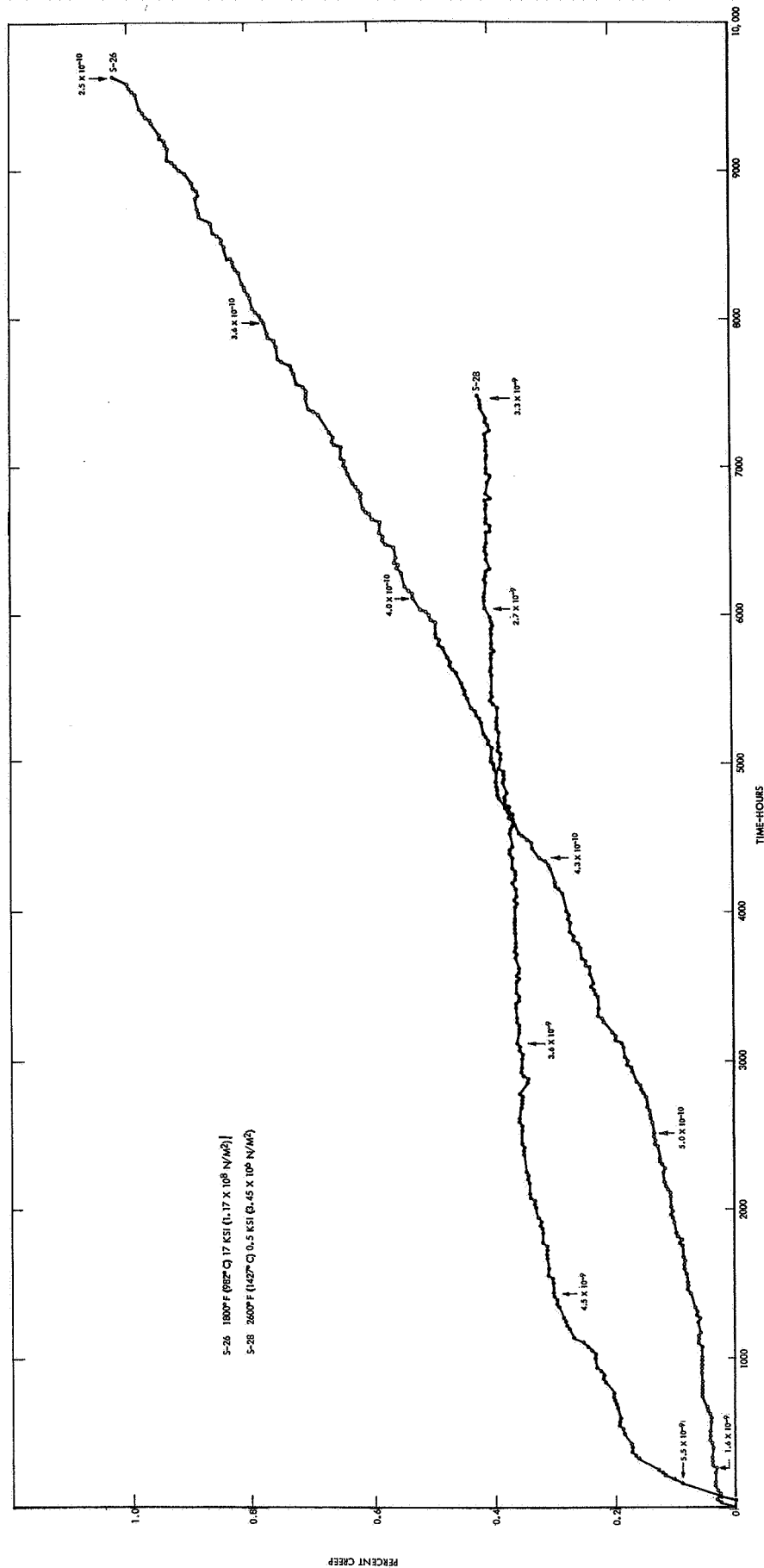


FIGURE 9. CREEP TEST DATA, T-111 HEAT D-1670, ANNEALED 3000°F (1649°C) ONE HOUR, AND TESTED IN VACUUM ENVIRONMENT  $< 1 \times 10^{-8}$  TORR

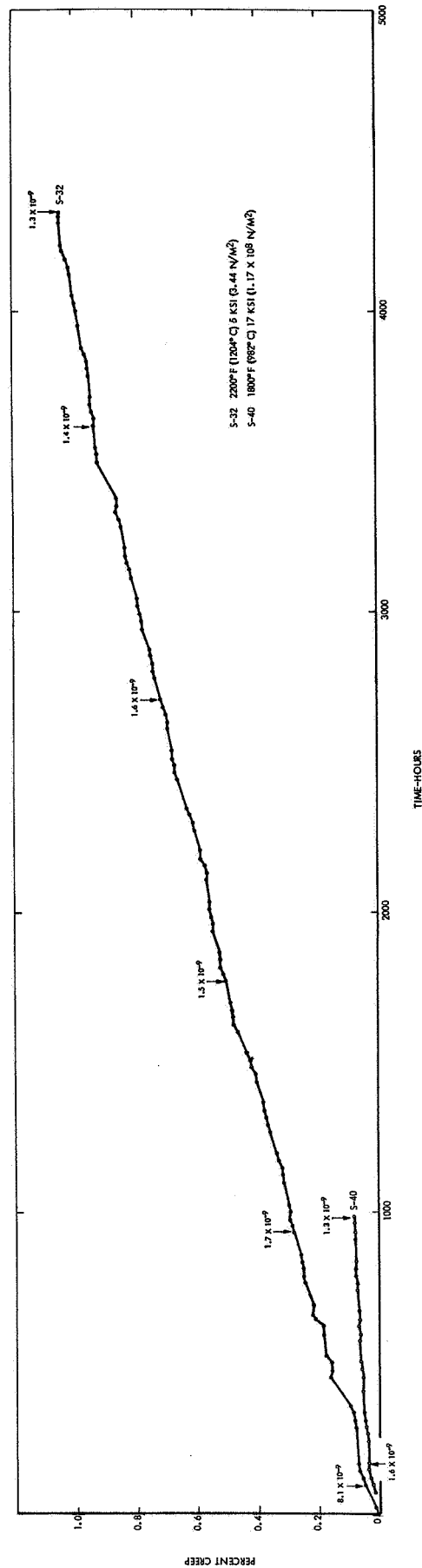


FIGURE 10. CREEP TEST DATA, T-111 HEAT D-1102, ANNEALED 3000°F  
ONE HOUR, AND TESTED IN VACUUM ENVIRONMENT < 1 X 10<sup>-8</sup> TORR

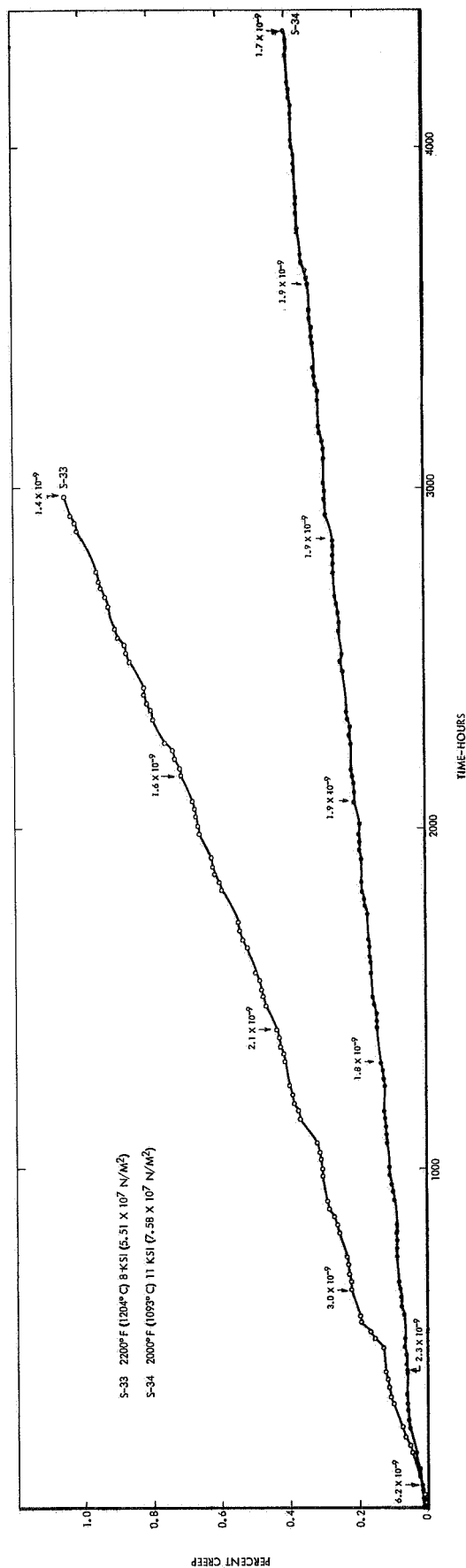


FIGURE 11. CREEP TEST DATA, T-111 HEAT MCN02A065, RECRYSTALLIZED 3000°F (1649°C) ONE HOUR, AND TESTED IN VACUUM ENVIRONMENT  $< 1 \times 10^{-8}$  TORR

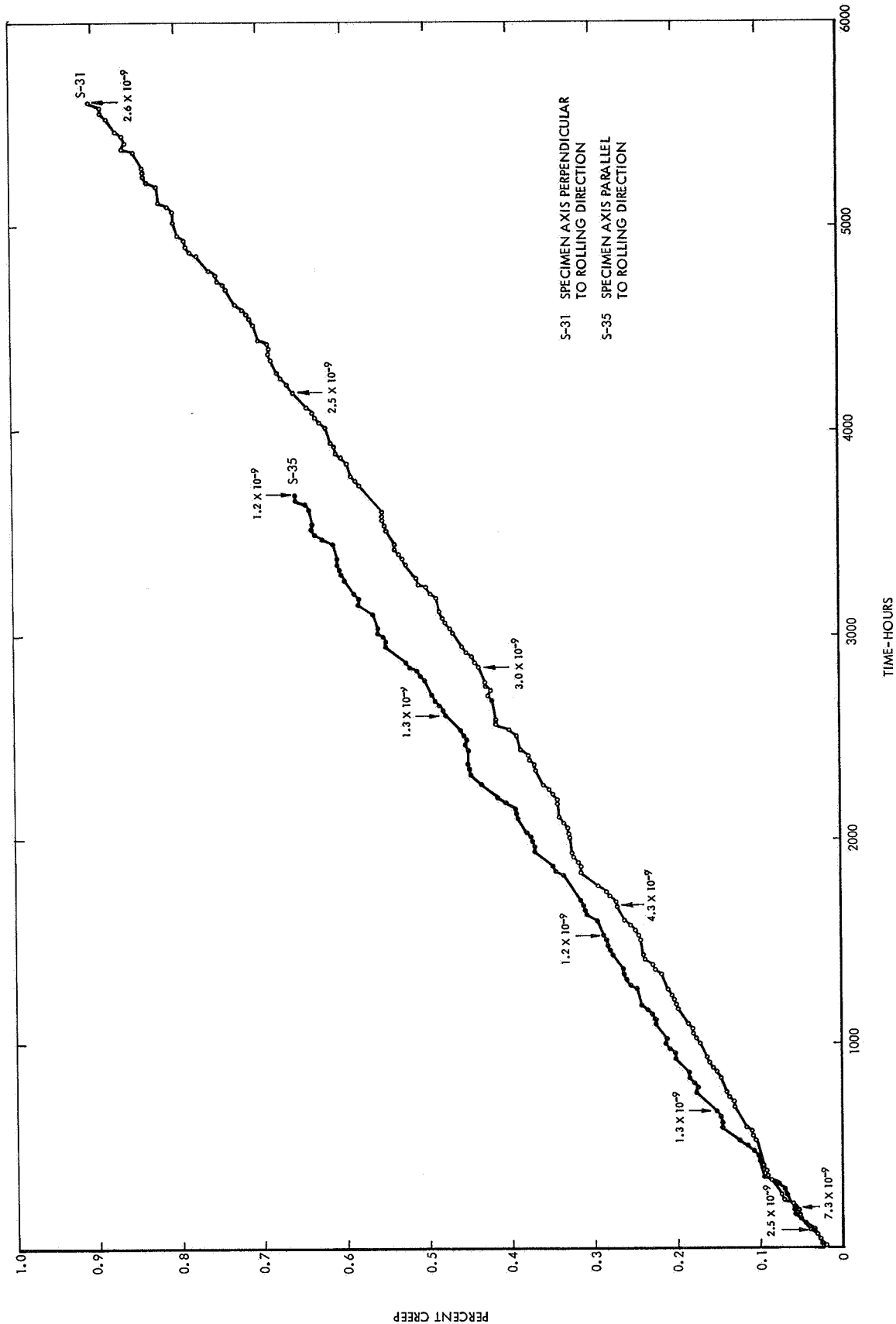


FIGURE 12. CREEP TEST DATA, T-111 HEAT 65079, ANNEALED 3000°F (1649°C) ONE HOUR, TESTED AT 2200°F (1204°C), AND 5 KSI (3.44 X 10<sup>7</sup> N/M<sup>2</sup>) IN VACUUM ENVIRONMENT < 1 X 10<sup>-8</sup> TORR

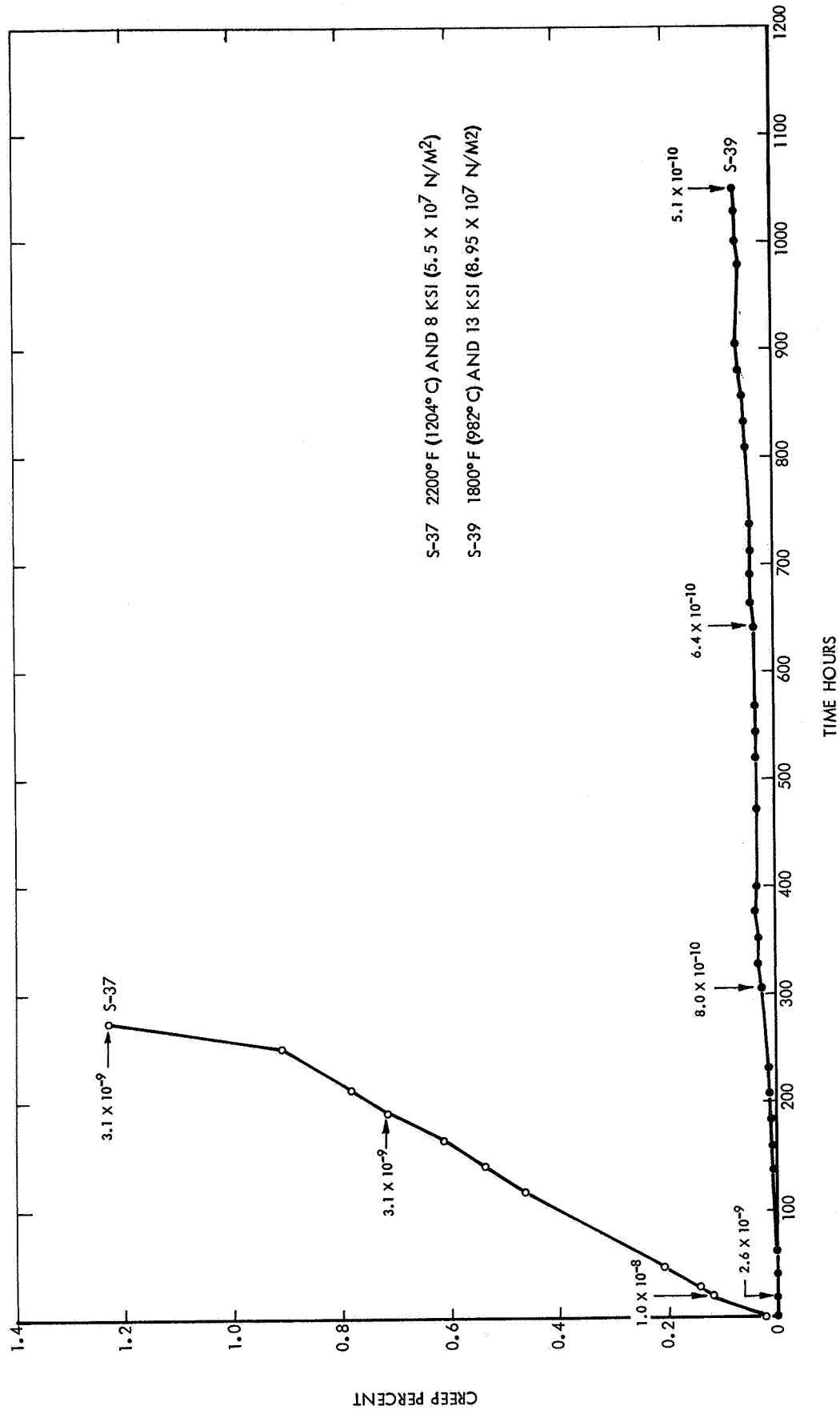


FIGURE 13. CREEP TEST DATA, T-111 HEAT 65080, ANNEALED 3000°F (1649°C) ONE HOUR. TESTED IN VACUUM ENVIRONMENT < 1 X 10<sup>-8</sup> TORR

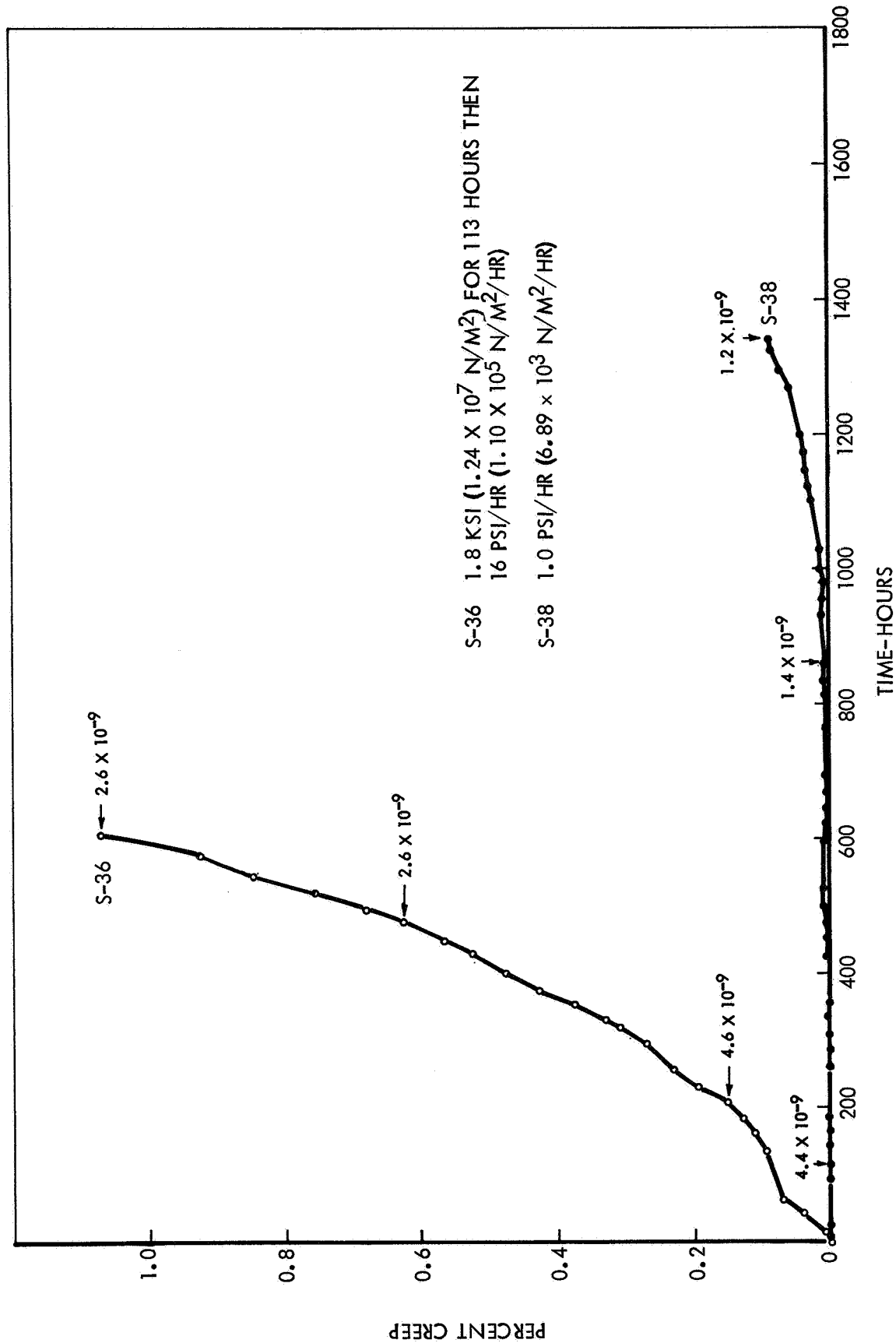


FIGURE 14. CREEP TEST DATA, T-111 HEAT 65080, ANNEALED 3000° F (1649° C), ONE HOUR,  
TESTED AT 2200° F (1204° C) WITH PROGRESSIVE LOAD IN VACUUM ENVIRONMENT  
< 1 x 10<sup>-8</sup> TORR

Table 7

T-111 Creep Tests

Specimen No.	Heat No.	Temperature °F   °C		Stress Ksi	$(N/m^2) \times 10^7$
S-26	D-1670	1800	982	17	11.7
S-28	D-1670	2600	1427	0.5	0.344
S-31	65079	2200	1204	5	3.44
S-32	D-1102	2200	1204	5	3.44
S-33	MCN02A065	2200	1204	8	5.51
S-34	MCN02A065	2000	1093	11	7.58
S-35	65079	2200	1204	5	3.44
S-36	65080	2200	1204	16 psi/hour	Loading Rate
S-37	65080	2200	1204	8	5.51
S-38	65080	2200	1204	1 psi/hour	Loading Rate
S-39	65080	1800	982	13	8.95
S-40	D-1102	1800	982	17	11.7

It has generally been observed that the creep curves for T-111 annealed at 3000°F (1649°C) for one hour are linear or slightly concave upward. Specimen S-28 tested at 2600°F (Figure 9) is an exception, since a decreasing creep rate is observed.

Tests S-31 and S-35 (Figure 12) compare the effect of orientation on the creep properties of Heat 65079 by using specimens whose axes are respective perpendicular to and parallel to the rolling direction. In view of the fact that the tensile properties of Heat 65079 (Table 8) show essentially no directional dependence, the slight difference in creep strength probably represents a statistical variation of test results.

Table 8

Tensile Properties T-111, Heat 65079  
Recrystallized 3000°F (1649°C), 1 Hour

Test Temperature °F	Strength, Ksi		Elongation %
	Ultimate	Yield	
R.T.	94 (93)*	74 (72)	35 (34)
1800	74 (76)	34 (34)	23 (24)
2200	49 (51)	32 (32)	30 (27)
2600	29 (29)	25 (26)	50 (50)

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

Tests S-37 (Figure 13) and S-33 (Figure 11) were conducted at identical temperature and stress, but with different heats of T-111 alloy. Specimen S-37 was taken from Heat 65080 and specimen S-33 from Heat MCN02A065. Comparison of the creep life shows that the specimen from Heat 65080 reached 1% strain in 260 hours while the Heat MCN02A065 specimen required 2850 hours to reach this same extension. A comparison of the two heats (Table 9) shows no significant difference which could explain the lower strength of Heat 65080. Comparison of the tensile properties of the two heats (Tables 8 and 10) again reveal no differences of a magnitude which could account for the observed difference in creep strength. Comparison of the processing methods (Appendix 1, Tables V and VII) shows that both heats were produced by the same vendor, with the only differences being the recrystallization temperature used and the thickness before final reduction. To date the factors of processing temperature and size before final reduction have not been related to the difference in strength; however, the effect of recrystallization temperature prior to annealing the specimen at 3000°F (1649°C) in situ is now being subjected to further examination. Since grain size is known to influence of creep strength of T-111 alloy, the grain sizes of various heats of T-111 were measured and compared to the 1.0% creep life in Figure 15. The results show an apparent influence of grain size upon creep strength. These data indicate that the low creep strength of Heat 65080 might be in part be attributed to a smaller grain size being present after annealing one hour at 3000°F (1649°C). The underlying nucleation and growth phenomenon responsible for the variation in grain size remain unknown.

The 1.0% creep life data for various heats of T-111 are given in Table 11 together with Larson-Miller and Manson HaFord parameters. In both cases the parametric constants are based on the data of Heat 70616 and were obtained from exact or numerical computer solutions of the parametric equations. These data plotted in Figures 16 and 17 show a distinction between the various heats of T-111, with Heats 70616 and MCN02A065 being the most creep resistant and Heat 65080 exhibiting the poorest properties.

Two tests were made with T-111 Heat 65080 employing a load which was increased uniformly with time. Test S-36 was run at a steady stress of 1.8 Ksi ( $1.24 \times 10^7$  N/m<sup>2</sup>) for the first 113 hours, after which the stress was uniformly increased at the rate of 16 psi/hour ( $1.10 \times 10^5$  N/m<sup>2</sup>/hour). Test S-38 was loaded at a lower rate of 1 psi/hour ( $6.89 \times 10^3$  N/m<sup>2</sup>/hour) beginning at zero stress. The pronounced upward curvature of the creep curves from these tests is a result of the constantly increasing stress.

The purpose of the tests involving increasing stress is to determine whether data obtained at a constant stress can be incrementally summed to predict varying stress behavior. The computer program developed by Nichols and Winkler<sup>(3)</sup> to

Table 9

Chemistry of T-111 Heats 65080 and 65079  
(Weight Percent)

<u>Element</u>	<u>Specification</u>	<u>Heat MCN02A065</u>	<u>Heat 65080</u>
Al*	-	<0.0010	<0.0010
C	<0.0050	0.0040	0.0030
Cb *	<0.1000	0.065	0.080
Ca *	<0.0050	<0.0005	<0.0005
Cr *	-	<0.0010	<0.0010
Cu *	-	<0.0020	<0.0020
Fe	<0.0050	0.0020	0.0030
H <sub>2</sub>	<0.0010	0.0003	0.0003
Hf	1.8-2.4	1.95	1.91
Mo *	<0.0200	<0.0010	0.0020
N <sub>2</sub>	<0.0075	0.0020	0.0030
Ni *	<0.0050	<0.0010	<0.0010
O <sub>2</sub> *	<0.0150	0.0100	0.0090
Si *	-	<0.0020	<0.0040
Ta *	Balance	Balance	Balance
Ti	-	<0.0020	<0.0020
V *	<0.0020	<0.0010	<0.0020
W	7.0-9.0	8.60	8.88

\* Determined Spectrographically

Table 10

Tensile Properties T-111, Heat 65080  
Recrystallized 1 Hour 3000°F (1649°C)

Test Temperature °F	Strength, Ksi		Elongation %
	Ultimate	(0.2% Offset) Yield	
R.T.	95.9 (95.0)*	90.0 (85.3)	31.8 (32.0)
2000	62.2 (67.0)	35.5 (38.7)	23.0 (13.2)
2400	32.8	28.2	40.2
2600	28.5	27.4	63.8

\* ( ) - Denotes specimen axis perpendicular to rolling direction.

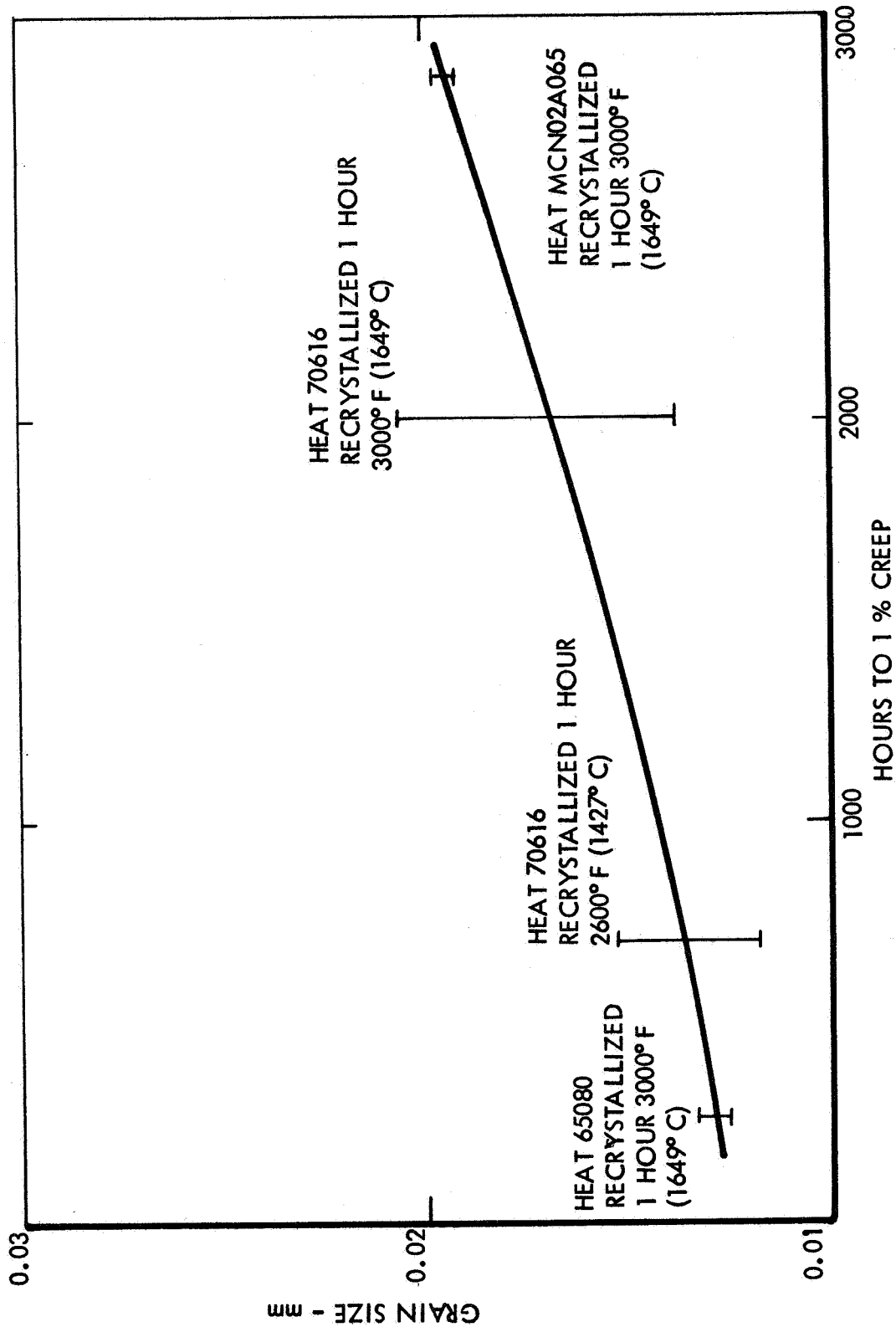


FIGURE 15 EFFECT OF GRAIN SIZE ON 1% CREEP LIFE OF T-111 ALLOY TESTED AT 2200° F (1204° C) AND 8 Ksi (5.51 X 10<sup>7</sup> N/m<sup>2</sup>) IN VACUUM ENVIRONMENT < 1 X 10<sup>-8</sup> TORR

**Table II**

1% Parametric Creep Data for T-111 Tantalum-Base Alloy  
Recrystallized 1 Hour 3000°F (1649°C)

Specimen No.	Temperature		Stress		Hours for 1.0% Creep	Larson-Miller T°R(11.1+logt) x 10 <sup>-3</sup>	Manson-Haferd logt-17.5 T+417 x 10 <sup>3</sup>
	°F	°C	Ksi	N/m <sup>2</sup> x 10 <sup>8</sup>			
Heat 70616							
S-19	2200	1204	8	0.551	2000	38.3	-5.42
S-21	2200	1204	12	0.826	1140	37.6	-5.52
S-23	2120	1160	12	0.826	3150	37.7	-5.52
S-22	2000	1093	20	1.380	670	34.2	-6.07
S-24	1860	1016	20	1.380	4730	34.3	-6.07
Heat D-1670							
S-25	2000	1093	15	1.030	1340	35.0	-5.95
S-26	1800	982	17	1.170	9540	34.1	-6.10
S-25A	2600	1427	1.5	0.103	1100	43.3	-4.79
S-28	2600	1427	0.5	0.0344	40,000	48.0	-4.28
Heat D-1102							
S-27	2000	1093	13	0.895	1880	35.4	-5.88
S-32	2200	1204	5	0.344	4050	39.1	-5.31
S-40	1800	982	17	1.170	18,000*	34.7	-5.97
Heat 65079							
S-30**	2400	1316	3.5	0.241	860	40.1	-5.17
S-31**	2200	1204	5	0.344	6400*	39.6	-5.23
S-35	2200	1204	5	0.344	6000*	39.6	-5.24
S-42	2300	1260	3.5	0.241	7000*	41.2	-5.02
Heat MCN02A065							
S-33	2200	1204	8	0.551	2850	38.7	-5.37
S-34	2000	1093	11	0.758	11,300	37.3	-5.56
Heat 65080							
S-37	2200	1204	8	0.551	260	35.9	-5.76
S-39	1800	982	13	0.895	13,700*	34.4	-6.03

\* Extrapolated Data

\*\* Specimen Axis Perpendicular to Rolling Direction

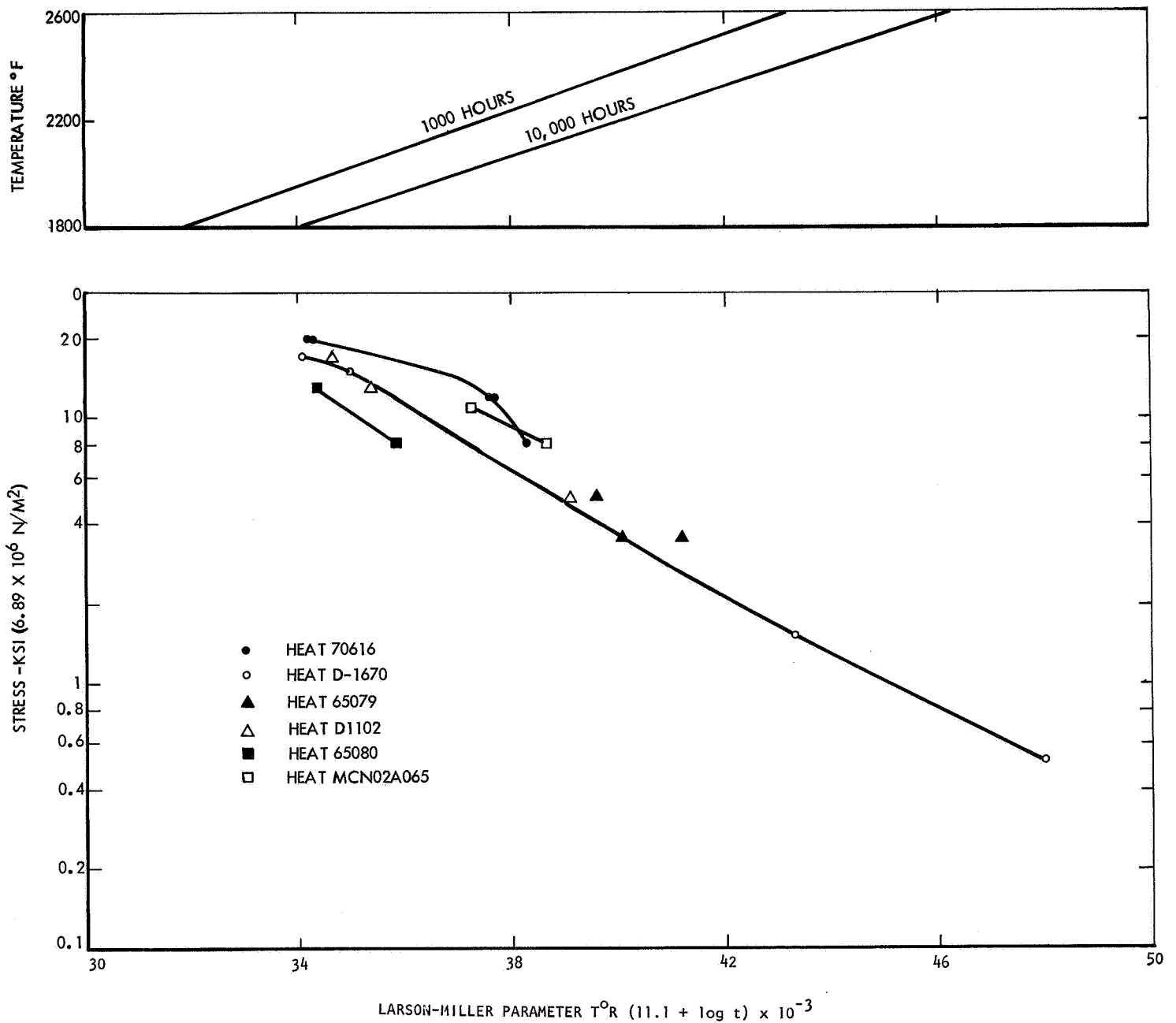


FIGURE 16. LARSON-MILLER PLOT OF CREEP DATA FOR TANTALUM-BASE T-111 ALLOY ANNEALED 3000°F (1649°C) ONE HOUR AND TESTED IN VACUUM ENVIRONMENT  $< 1 \times 10^{-8}$  TORR

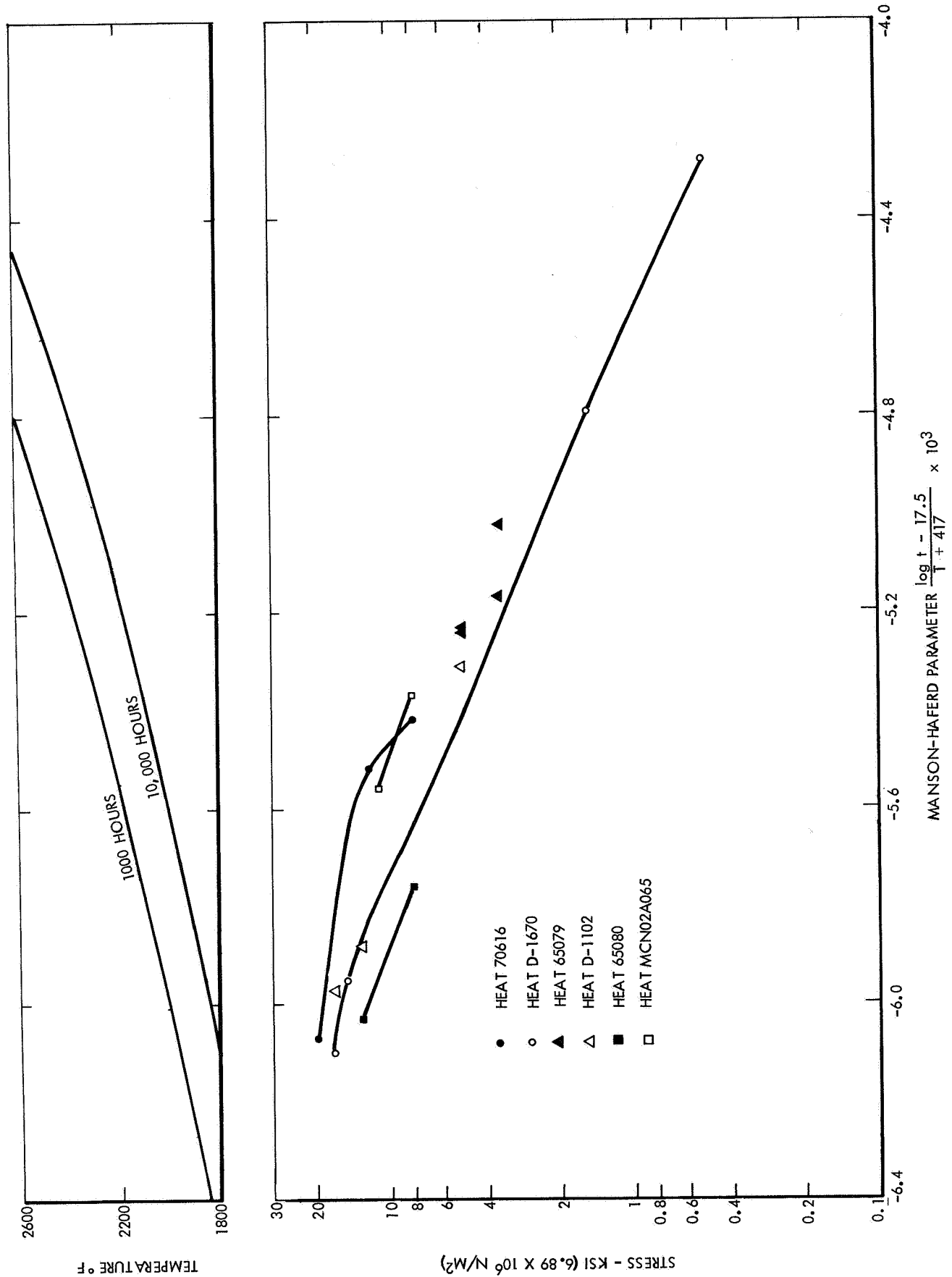


FIGURE 17. MANSION-HAFERD PLOT OF CREEP DATA FOR TANTALUM-BASE T-111 ALLOY  
ANNEALED 3000°F (1649°C) ONE HOUR AND TESTED IN VACUUM ENVIRONMENT  
< 1 X 10<sup>-8</sup> TORR

perform the summation is currently being adapted to the TRW computer facilities. McCoy<sup>(4)</sup> has also developed a procedure for calculating varying stress creep life and using this method a 1% creep life of 4570 hours has been predicted for test S-38 shown in Figure 14.

#### Tantalum-Base Astar 811C

A single test of Astar 811C recrystallized at 3600°F (1982°C) for 0.5 hour is being made at 2600°F (1427°C) and 2 Ksi ( $1.38 \times 10^7$  N/m<sup>2</sup>) (Figure 18). At the current rate of creep, 1% extension should be obtained in approximately 24,000 hours. This predicted creep life is considerably greater than the 400 hours for 1% creep calculated for T-111 alloy if tested at a similar temperature and stress.

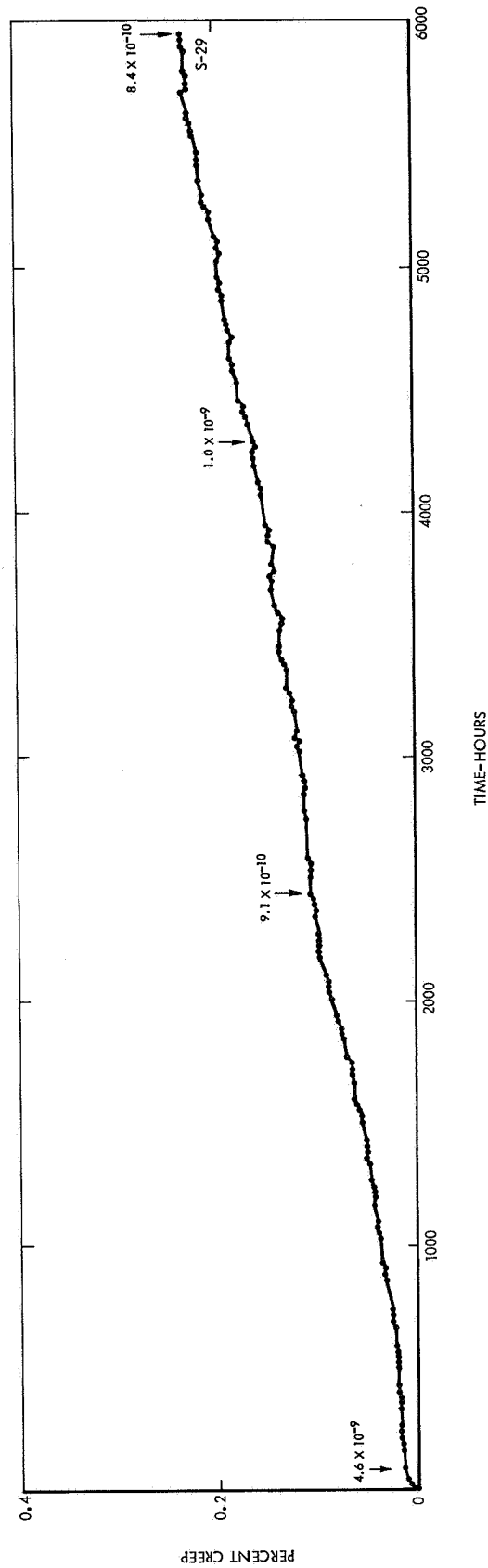


FIGURE 18. CREEP DATA, ASTAR 811C, RECRYSTALLIZED 3600°F (1982°C) 0.5 HOUR, TESTED AT 2600°F (1427°C), AND 2 KSI ( $1.38 \times 10^7$  N/M<sup>2</sup>) IN VACUUM ENVIRONMENT  $< 1 \times 10^{-8}$  TORR

#### IV SUMMARY

Tests of molybdenum-base TZC have shown that extruded and broad forged material has a creep strength equivalent to that of cross-rolled plate.

Tests of molybdenum-base TZM show that the creep life is significantly influenced by the carbon content and method of processing. In the case of Heat KDTZM-1175, the creep strength at high stress is superior to that of molybdenum-base TZC alloy.

Tantalum-base T-111 alloy has been found to exhibit considerable variation in creep strength despite similarities in chemistry and tensile properties. The cause of this variation has not been determined.

Tantalum-base Astar 811C is much more creep resistant than the T-111 tested to date.

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Appendix I

PROCESSING HISTORY OF MATERIALS

TABLE IPROCESSING HISTORY OF TZC PLATEVendor:

General Electric Company  
Refractory Metals Plant  
Cleveland, Ohio  
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 IIPROCESSING HISTORY OF TZC PLATEVendor:

Climax Molybdenum Company of Michigan  
Coldwater, Michigan  
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 IIIPROCESSING HISTORY OF TZM FORGED DISCVendor:

Climax Molybdenum Company of Michigan  
Coldwater, Michigan  
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 IVPROCESSING HISTORY OF TZM FORGED DISCVendor:

AiResearch (Universal Cyclops)  
Disc. No. 3  
Heat KDTZM-1175

Processing History:

- 1) 11-3/4" diameter ingot, machine to 10-3/4" diameter.
- 2) Extrude to 6-1/2" diameter at 2250°F (1232 °C).
- 3) Recrystallize at 2800°F (1538°C) for 4 hours.
- 4) Forge to 4" diameter billet 3400°F to 2800°F (1871 to 1538°C).
- 5) Recrystallize at 2950°F (1621°C) for 2 hours.
- 6) Forge to flat disc 3/4" thick, 2800°F (1538°C) starting temperature 11 blows, finish temperature 2160°F (1182°C).
- 7) Stress relieve at 2300°F (1260°C) for 1 hour.

Hardness:

297-335 DPH (29-34 Rc)

TABLE VPROCESSING HISTORY OF T-111 SHEETVendor:

Wah Chang Corporation  
Albany, Oregon  
Heat 65076 (MCN02A065)

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 3000°F (1649°C).
- 3) Warm roll 800°F (427°C) to 1/4" thick.
- 4) Vacuum anneal 3000°F (1649°C).
- 5) Cold roll to final thickness.
- 6) Vacuum anneal ( $1 \times 10^{-4}$  torr) 3000°F (1649°C), 1 hour.

Hardness:

262 DPH (25 Rc)

TABLE VI  
PROCESSING HISTORY OF T-111 SHEET

Vendor:

Wah Chang Corporation  
Albany, Oregon  
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 ( $1 \times 10^{-4}$  torr) 2800°F (1538°C), 1 hour.

Hardness:

236 DPH (20 Rc)

TABLE VIIPROCESSING HISTORY OF T-111 SHEETVendor:

Wah Chang Corporation  
Albany, Oregon  
Heat 65080

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 .260 or 0.060" thick.
- 4) Vacuum anneal 2800°F (1538°C), 2 hours.
- 5) Cold roll to final thickness.
- 6) Vacuum anneal (  $1 \times 10^{-4}$  torr) 2800°F (1538°C), 2 hours.

Hardness:

248 DPH (22 Rc)

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