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Creep Properties of Selected Refractory Alloys for Potential Space Nuclear Power Applications

H. E. McCoy

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CREEP PROPERTIES OF SELECTED REFRACTORY ALLOYS FOR POTENTIAL SPACE NUCLEAR POWER APPLICATIONS

H. E. McCoy

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CONTENTS

ABSTRACT 1
INTRODUCTION 1
EXPERIMENTAL DETAILS 2
TEST MATERIALS 2
TEST SPECIMENS 4
CREEP MACHINES 5
TEMPERATURE MEASUREMENT 7
STRAIN MEASUREMENT 8
QUALIFICATION OF CREEP TEST FACILITIES 9
CREEP TEST RESULTS FOR Nb–1% Zr 9
CREEP DATA FOR PWC-11 19
CREEP TEST RESULTS FOR Mo-Re ALLOYS 24
CREEP RESULTS FOR CVD-W 28
METALLOGRAPHIC STUDIES 30
DISCUSSION OF RESULTS 45
CONCLUSIONS 47
ACKNOWLEDGMENTS 48
REFERENCES 49
Appendix. CREEP CURVES 51

CREEP PROPERTIES OF SELECTED REFRACTORY ALLOYS FOR POTENTIAL SPACE NUCLEAR POWER APPLICATIONS*

H. E. McCoy

ABSTRACT

Existing equipment and previous test techniques were requalified for conducting high-vacuum, high-temperature creep tests on refractory metals. The creep facilities were requalified on the basis of minimal interstitial pickup and reproducible creep results using Nb-1% Zr and T-111 (Ta-8% W-2% Hf) specimens. After requalification was completed, tests were run on three lots of Nb-1% Zr, one lot of PWC-11 (Nb-1% Zr-0.1% C), molybdenum alloys containing 9, 11, and 13% Re, and two lots of chemical vapor deposition tungsten (CVD-W). The creep strength of Nb-1% Zr was equivalent to that reflected in previously correlated data. Our lot of PWC-11 was thought to have been extruded at a lower-than-optimum temperature, so it was weaker. The duplex anneal used increased the strength slightly. The test results on the Mo-Re alloys were inconclusive because of the multiple stresses and temperatures used on each specimen. It was apparent that the Mo-9% Re alloy was the weakest and that the Mo-11% Re and Mo-13% Re alloys, which had about equal properties, were the strongest. The work on CVD-W has just begun; the creep strength appears equivalent to that measured in previous tests under similar conditions.

INTRODUCTION

The implementation of proposed nuclear power concepts requires the use of refractory alloys based on niobium, molybdenum, tantalum, or tungsten. These materials have melting points above those of iron, nickel, and other common structural materials but have not been used nearly as often. Thus, for refractory alloys, which offer the potential for use at elevated temperatures, designers lack the data and experience which would allow them to proceed with confidence in hardware design.

*Work sponsored by the Division of Defense Energy Projects, U.S. Department of Energy, under contract No. DE-AC05-84OR21400 with Martin Marietta Energy Systems, Inc.

Also unfortunate is the fact that the interest in these materials for nuclear applications is restricted largely to the U.S. government, which ceased work on these materials in 1972. A little additional work has been done by small commercial companies since that time.

The renewed interest in the refractory metals for the SP-100 Program brings about the need to (1) look critically at the existing good data for candidate alloys and (2) initiate a testing program which will test newly manufactured alloys in order to fill in the voids in the old data bases.

Horak et al.[1] at Oak Ridge National Laboratory (ORNL) have been very active in reviewing past data, while both ORNL and the National Aeronautics and Space Administration (NASA) Lewis Research Center have initiated efforts to requalify existing high-vacuum, high-temperature creep facilities and to initiate testing in these facilities. The purpose of this report is to describe progress to date at ORNL in (1) requalifying creep test facilities and operators and (2) performing creep tests on candidate alloys under consideration by the SP-100 Program.

EXPERIMENTAL DETAILS

TEST MATERIALS

The test materials used in this study are listed in Table 1 along with several pertinent details. The first material (Nb-1% Zr), manufactured several years ago and in storage at ORNL, was reanalyzed and found to have a reasonable concentration of interstitial elements. The material was worked from a thickness of 0.16 cm to a thickness of 0.10 cm. The second and third heats of Nb-1% Zr, obtained in the last year from Wah Chang, were provided in the annealed condition. Thus, the last two heats of material are representative of currently produced material.

The PWC-11 material, which had been stored at ORNL for approximately 15 years, had the physical form of a tube shell. It was about 0.5 cm thick and was cold worked to a 0.10-cm sheet for making specimens.

The molybdenum-rhenium alloys were sent to ORNL directly from American Metal Climax, Inc. (AMAX) under instructions from Los Alamos

Table 1. Characterization of test materials

Alloy	Designation	Form	Chemical composition (ppm unless noted %)
Nb-1 Zr	12R-1614	0.10-cm sheet - CW	ORNL analysis - C (21), O (205), N (66), H (2)
Nb-1 Zr	530860	0.10-cm sheet - annealed	Ingot analysis - W (70-150), C (40-50), Zr (0.88-0.90%), Ta (0.885-0.875%), O (140-170), N (43-50) ORNL analysis - C (20), O (202), N (31), H (1)
Nb-1 Zr	530870	0.10-cm sheet - CW	Ingot analysis - W (210-270), C (30-40), Ti (49-174), Ta (940-990), Zr (1.1-1.2%), Mo (50), O (50-60), N (40-42) ORNL analysis - C (43), O (207), N (31), H (4)
PWC-11	RS-8712	0.10-cm sheet - CW	ORNL analysis - C (890), H (11), N_2 (60), O (110), Zr (1.04%)
T-111	111684	0.10-cm sheet - CW	ORNL analysis - C (60), O (41), N (11)
Mo-9 Re	H-390	0.32-cm sheet - CW	
Mo-11 Re	H-389	0.32-cm sheet - CW	
Mo-13 Re	H-395	0.32-cm sheet - CW	AMAX analysis - C (75)
CVD-W	117	Material produced at ORNL several years ago	
CVD-W	5235-141	Material produced recently by GA Technologies	

National Laboratory (LANL). Some of the material, which had rather rough surfaces, was removed from the test section of each specimen.

Two lots of chemical vapor deposition tungsten (CVD-W), which is very brittle and difficult to fabricate, were involved in our test program. The first lot (117) was made at ORNL several years ago. It was used to reactivate the various steps used in fabricating and testing this material. The second lot (5235-141) was produced by GA Technologies and is representative of currently produced material. Both materials contain about 5 ppm fluorine.

TEST SPECIMENS

The specimens used for the Nb-1% Zr, PWC-11, and T-111 tests are shown in Fig. 1. Niobium- and tantalum-base alloys were machined into this configuration by milling, and tungsten-base materials were machined by the electric discharge machining (EDM) method. To reinforce the holes in each end, reinforcing pads of the same or a similar material are welded

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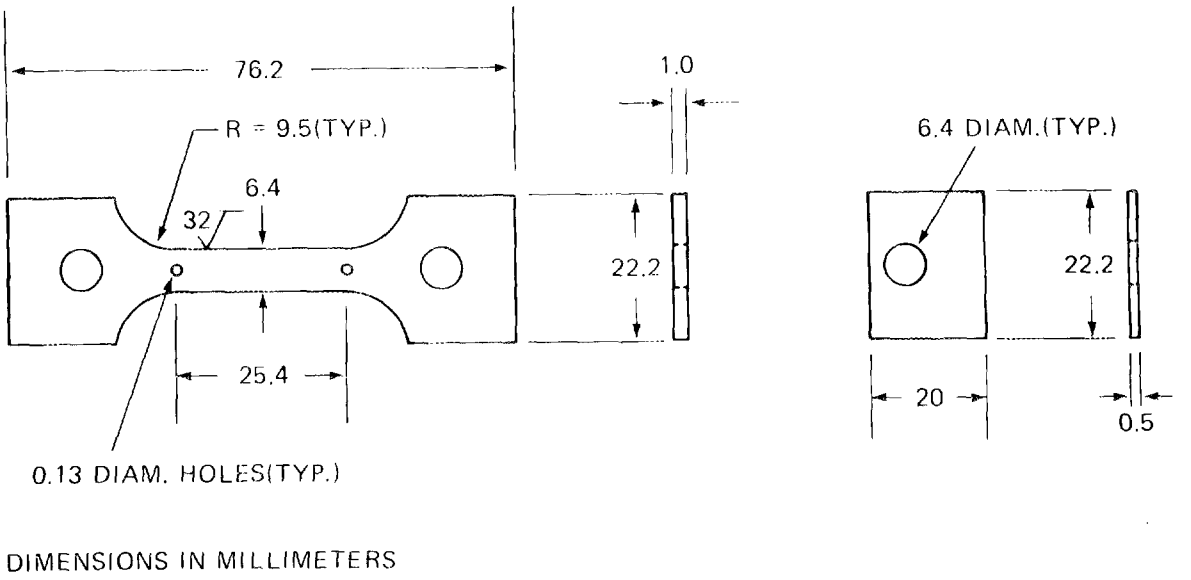

Fig. 1. Schematic of test specimen and support pad.

to the ends of the sample. Two small holes are machined by the EDM method at the extremes of the gage length. These holes are sighted with a special measuring microscope, and their relative movement is used as the measure of strain.

The molybdenum-rhenium specimens had a gage length 6.4 mm wide × 31.8 mm long and a thickness of 3.2 mm. The surface was rough, however, so about 0.8 mm was sanded from each side of the gage section. This made the ends thicker, making it unnecessary to weld support pads to each end. Holes for measuring the strain were 25.4 mm (1 in.) apart even though the gage length was longer.

The geometry of the CVD-W specimen is shown in Fig. 1. The material was hard and brittle and had to be machined by the EDM method. (This method introduces shallow intergranular cracks, so all machined surfaces must either be ground or lapped to remove the cracked material.) Techniques were developed for depositing CVD-W in the form of a thin-walled box 5 × 5 cm in cross section × 50 cm long.[2] Numerous variations of this technique have been developed, but they basically involve the reduction of WF_6 by H_2 with the deposition of tungsten on a substrate.

CREEP MACHINES

The three types of creep machines at ORNL are shown in Figs. 2 through 4. All of the machines are of the dead-load type, and all machines utilize wattage transducers for temperature control. We have two machines of the type shown in Fig. 2. These units have tungsten-mesh heating elements and tungsten shields; they are oil-pumped with liquid nitrogen cold traps. The vacuum in the test chamber is usually of the order of 10^{-8} torr, depending on the test temperature. We have three units of the type shown in Fig. 3. These systems have ion pumps; the heating elements and shields are made of tantalum. We have two machines of the type shown in Fig. 4. These test units have ion pumps and tungsten heaters and heat shields.

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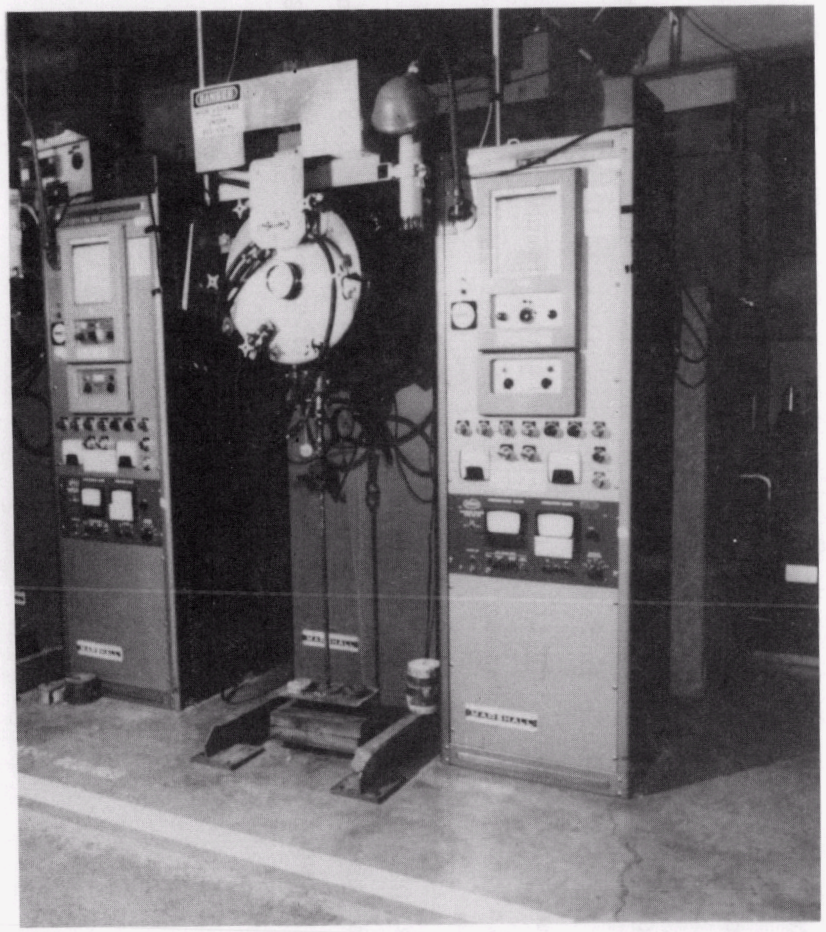

Fig. 2. Vacuum creep machines with oil-diffusion pumps and liquid nitrogen cold traps.

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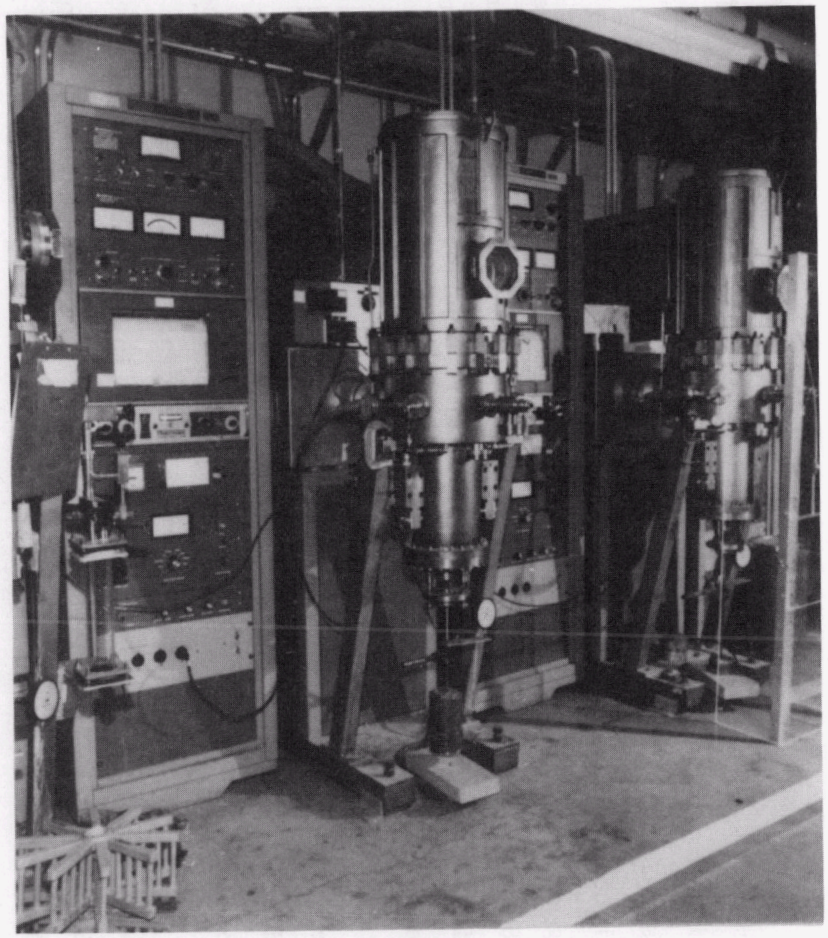

Fig. 3. Three creep machines, using ion pumps, available for testing.

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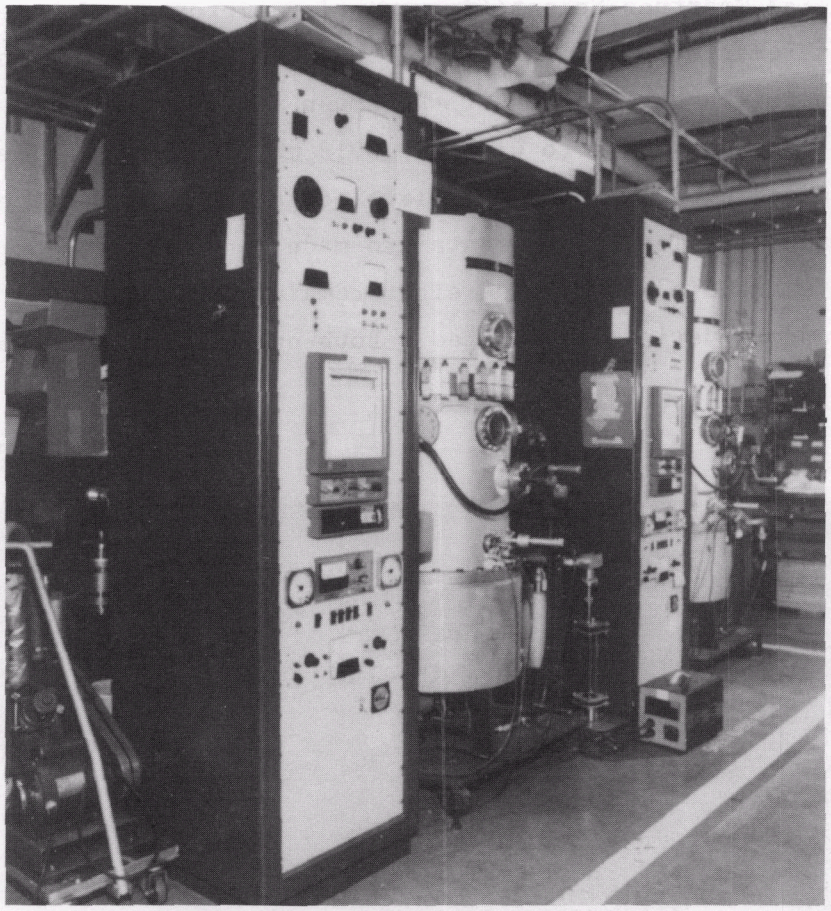

Fig. 4. Two vacuum creep machines with ion pumps and tungsten heating chambers.

TEMPERATURE MEASUREMENT

The temperature in each creep unit is measured by means of a black body and an optical pyrometer. The optical pyrometer used in each machine is calibrated by our Rheology Laboratory using a standard from the National Bureau of Standards. The calibration is performed individually for each machine with the specific sight glass in place. Thus, for each machine there is a calibration curve that incorporates errors associated with the optical pyrometer and the sight glass. This calibration is repeated at least annually.

STRAIN MEASUREMENT

A special Gaertner Scientific measuring microscope is used for making strain measurements. The microscope, shown in Fig. 5, is sighted on two small holes in the gage length of the creep sample. Relative movement of the two holes is measured to within 0.0013 mm (50 μ-in.). The table on which the microscope is mounted is leveled, and the microscope is sighted for each machine. Ten measurements are made, and the highest and lowest measurements are discarded. The eight remaining measurements are averaged and the standard deviation computed. Both numbers are recorded.

ORNL PHOTO 1288-86

Fig. 5. Technician using optical strain-measuring device. Specimen being measured is to the right of technician. Microscope is sighted on two small holes in the specimen gage length (shown in Fig. 1). The change in the distance between the two holes is the strain.

QUALIFICATION OF CREEP TEST FACILITIES

Specimens of T-111 and Nb-1% Zr were tested to qualify the seven creep machines and the operating personnel. As a result, two operators qualified to use the optical extensometer, and they routinely make groups of ten strain readings having a standard deviation of 0.0026 to 0.0078 mm (100 to 300 μ-in.), which is equivalent to 0.01 to 0.03% strain in a 25.4-mm (1-in.) gage length.

Creep testing of the Nb-1% Zr and T-111 specimens was discontinued in July 1984. A summary of results from these tests is provided in Table 2. Comparison of recent Nb-1% Zr and T-111 creep test results with the analysis performed by Horak on previous T-111 and Nb-1% Zr creep results provides reasonable agreement (Figs. 6 and 7).

An additional concern associated with the creep testing of refractory alloys is interstitial contamination of the test specimen from impurities in the test chamber. Table 3, which summarizes the results of chemical analysis of selected Nb-1% Zr and T-111 test specimens, reveals only modest increases in interstitial content during testing in ion-pumped systems. The carbon and oxygen contents of the Nb-1% Zr specimen 24011 and the T-111 specimen 24012 increased markedly because these specimens were tested in a diffusion oil-pumped chamber. Based on these observations, the creep testing facilities and operating procedures are considered qualified. The two diffusion oil-pumped chambers are qualified for creep tests in which the specimens are wrapped with a protective foil.

CREEP TEST RESULTS FOR Nb-1% Zr

The results of creep tests on Nb-1% Zr are summarized in Table 4, and the creep curves are presented in the Appendix. The variables involved include three heats of material, three pretest anneals, base metal specimens, and welded specimens. Most of the tests were discontinued before the materials ruptured, and some tests are still in progress.

The test results are few in number; consequently, the data cannot be analyzed by some of the standard correlations. Horak et al. established the line shown in Fig. 8 on the basis of the existing usable minimum creep

Table 2. Summary of creep test data obtained to date

Material	Test	Pretest heat treatment[a]	Stress		Temperature		Type system[b]	Test time, (h)					Minimum creep rate (%/h)	Total strain (%) at termination of test
								Indicated strain			Tertiary creep	Test termination		
			MPa	ksi	°C	K		1%	2%	5%				
Nb-1% Zr	24011	1	14	2	1000	1273	A					1914	NA	NA
			28	4	1000	1273						649	NA	NA
			28	4	1100	1373						183	NA	NA
			28	4	1200	1473		NA	NA	NA	NA	246[c]	NA	2.0
Nb-1% Zr	24009	1	14	2	1000	1273	B					1989	<5.0E-5	<0.1
			28	4	1000	1273						624	1.0E-4	0.14
			28	4	1100	1373		75	135	302	132	383[d]	1.2E-2	6.0
Nb-1% Zr	24021	1	7	1	1000	1273	B					1158		~0
			7	1	1400	1673		65	130	260	200	549[e]	1.6E-2	10.5
Nb-1% Zr	24020	1	7	1	1200	1473						1098	~0	~0
			7	1	1600	1873	B	NA	NA	NA	NA	262[e]	NA	10
T-111	24012	2	28	4	1200	1473	A					1865	NA	NA
			103	15	1200	1473						836	NA	NA
			103	15	1300	1373		NA	NA	NA	NA	167[c]	NA	1.7
T-111	24000	2	41	6	1000	1273						2051	~0	~0
			172	25	1000	1273	B					278	1.1E-5	<0.1
			172	25	1100	1373		383				383[c]	2.2E-3	~1
T-111	24008	2	28	4	1200	1473	B					1931	~0	~0
			103	15	1200	1473						32	1.7E-4	0.1
			103	15	1300	1573		no readings				70[d]	5.5E-3	2.7

[a]Summary of heat treatments: 1 = 1 h at 1400°C; 2 = 1 h at 1659°C.

[b]A = oil diffusion pump, B = vacuum ion pump. Nominal vacuum levels are 10^{-8} torr.

[c]Discontinued prior to failure.

[d]Specimen ruptured.

[e]Failed shortly before this time.

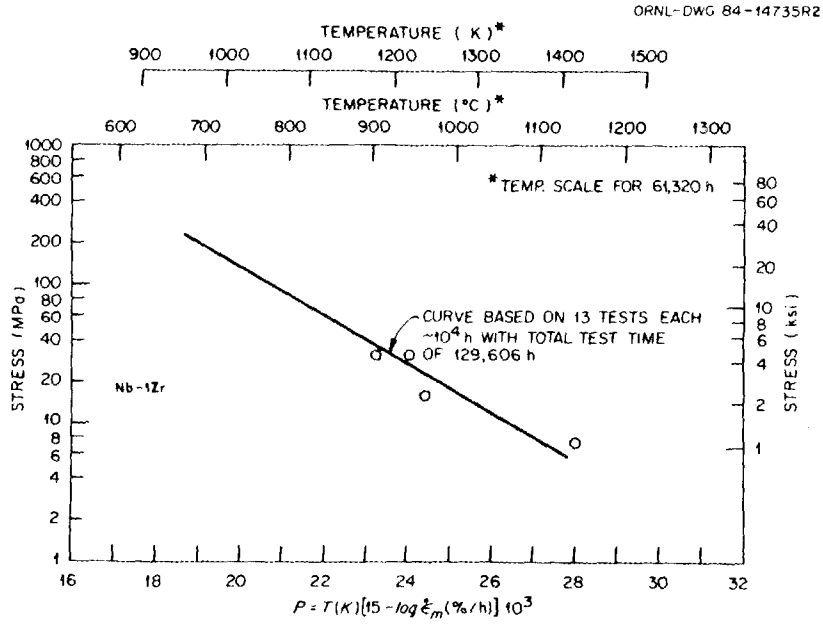

Fig. 6. Comparison of the results of four recent creep tests of Nb-1% Zr specimens performed at ORNL (represented by circles) with the predicted creep properties of Nb-1% Zr (represented by line).

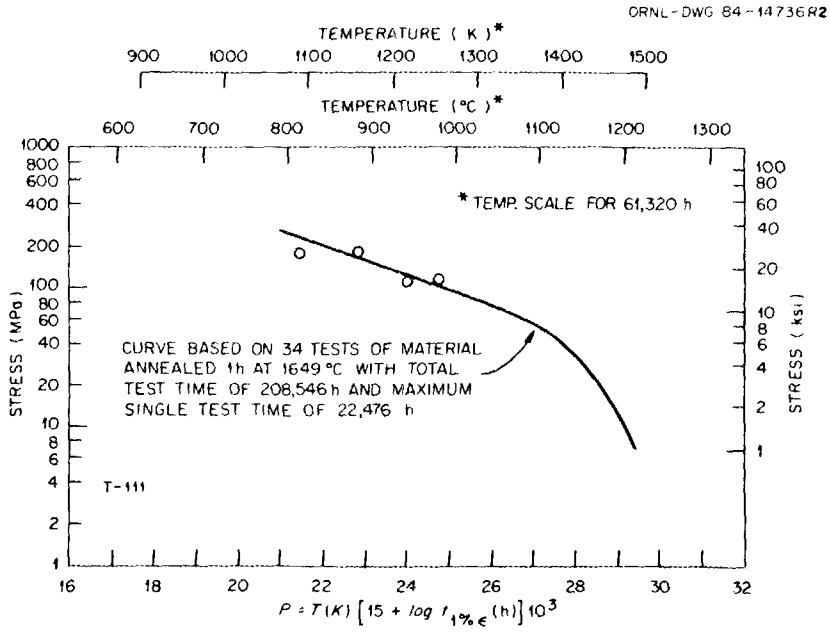

Fig. 7. Comparison of the results of four recent creep tests of T-111 specimens performed at ORNL (represented by circles) with the predicted creep properties of T-111 (represented by curve).

Table 3. Summary of chemical analysis of creep specimens

Type system[a]	Alloy	Test conditions			Chemical analysis (ppm)			
		Test	Cumulative test time (h)	Range of test temperature [°C (K)]	Oxygen	Nitrogen	Hydrogen	Carbon
NA	NB-1% Zr (as received)	NA	NA	NA	205	66	2	21
B	Nb-1% Zr	24021	1700	1000-1400 (1273-1673)	380	70	<1	44
B	Nb-1% Zr	24009	3000	1000-1100 (1273-1375)	239	54	<1	15
A	Nb-1% Zr	24011	2900	1000-1100 (1273-1373)	281	61	<1	195
NA	T-111	NA	NA	NA	41	11	<1	60
B	T-111	24008	2033	1200-1300 (1473-1573)	87	16	<1	62
A	T-111	24012	2900	1200-1300 (1473-1573)	262	12	<1	348

[a]A = oil diffusion pump; B = vacuum ion pump. Nominal vacuum levels are 10^{-8} torr. NA = not applicable.

Table 4. Creep tests on Nb–1% Zr

Test	Pretest anneal (h/K)	Type specimen[a]	Heat	Stress (MPa)	Temperature (K)	Time to indicated strain (h)					Minimum creep rate (%/h)	Creep strain (%)
						1%	2%	5%	t[b]	R		
24724	1/1673, 2/1477	B	1614	48.3	1250	1160	2240			1940[c]	9.9 E-4	2.9
24665	1/1673	B	1614	48.3	1250	770	1000	1380	680	1461[c]	7.1 E-4	5.7
24556	1/1673	B	1614	17.2	1350	940	1500		1500	2076[c]	1.3 E-3	3.5
24561	1/1673, 2/1477	B	1614	17.2	1350	2000	4000			4600[c]	5.1 E-4	2.1
24557	1/1673	B	1614	48.3	1350	40	70	111	64	145[c]	2.5 E-2	8.5
24514	1/1673, 2/1477	B	530860	34.5	1250	1260	1860		1000	2070[c]	4.8 E-4	2.4
24360	1/1673	B	530860	48.3	1250	1270	1520		980	1861[c]	1.5 E-4	4.4
24284	1/1673	B	530860	48.3	1250	1000	1190	1800	800	2300[c]	2.5 E-4	10.6
24477	1/1673, 2/1477	B	530860	48.3	1250	790	990		690	1283[c]	5.8 E-4	3.3
24485	1/1673, 2/1477	B	530860	69.0	1250	80	98	143	61	214[c]	2.0 E-3	12.8
24267	1/1673	B	530860	34.5	1350	200	280	445	230	668[c]	7.3 E-3	13.9
24282	1/1673	B	530860	17.2	1350	1650	2775		2125	3600[c]	5.5 E-4	3.6
24486	1/1673, 2/1477	B	530860	17.2	1350	1300	2300		2030	2604[c]	7.6 E-4	2.1
24398	1/1673	LW	530860	17.2	1350	2390	4000		3900	4385[c]	4.4 E-4	2.3
24268	1/1673	TW	530860	34.5	1350	75	135	195	120	384[c]	1.1 E-2	20.6
24283	1/1673	TW	530860	17.2	1350	420	650	1360	600	2335[d]	2.4 E-3	10.4
24683	1/773	B	530870	48.3	1250	1070	1360		900	1465[c]	1.9 E-4	2.5
24681	1/1673	B	530870	48.3	1250	1180	1860		1000	1202[c]	3.0 E-4	3.3
24748	1/1673, 2/1477	B	530870	17.2	1350	600	950		780	1485[c]	1.7 E-3	3.9
24680	1/1673	B	530870	17.2	1350	2600				2672[d]	1.6 E-4	1.0

[a]B = base, TW = transverse weld, LW = longitudinal weld.

[b]Tertiary creep (based on 0.2% offset from minimum creep rate).

[c]Discontinued prior to failure.

[d]In progress.

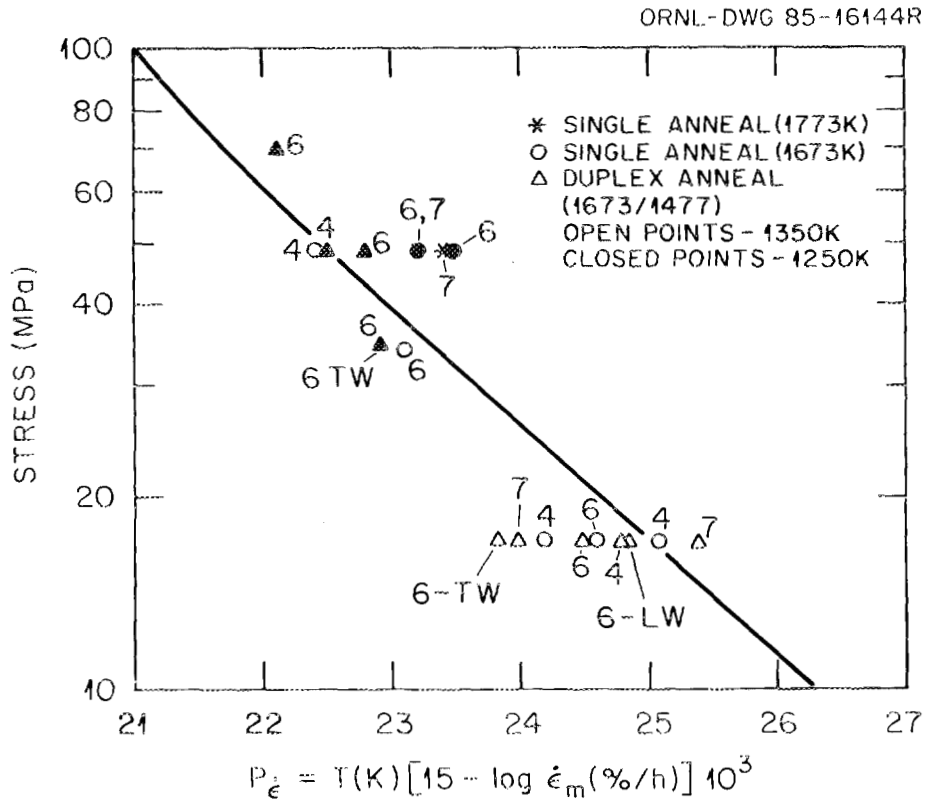

Fig. 8. Comparison of present Nb-1% Zr creep test results with correlation (line) based on minimum creep rate and Larson-Miller parameter established by Horak. Numbers denote last nonzero digit in heat number. Symbols TW and LW refer to transverse and longitudinal welds, respectively.

rate data by applying the Larson-Miller correlation using a constant of 15 (ref. 1). The data from the present study are shown with this line. The established line seems to represent the data average well, but there is considerable scatter about the line. One weld sample falls to the left of the line, but the other two weld samples fall very near the line. The data obtained at 1250 K and 48.3 MPa on two samples with a single anneal fall to the right of the line, but a sample with the duplex anneal comes closer to falling on the line. At a test temperature of 1350 K the scatter is less, and there seems to be less difference in effect between the samples receiving single anneal and samples receiving duplex anneal before the test. The properties of the three heats of Nb-1% Zr do not seem detectably different.

The data for the time to 1% strain are shown in Fig. 9 in a Larson-Miller correlation with a selected constant of 15. The one transverse weld sample tested at 1350 K and 17.2 MPa falls short of the line, but the other data fall very close to the line. There does not seem to be a significant difference between the strength of the material receiving the single anneal and that receiving the duplex anneal.

The correlation in Fig. 10 is plotted to give some idea of the measured time to 1% strain and the time computed from the minimum creep rate. The time-dependent strain through primary and secondary creep is given by

$$\epsilon = at^n + t\dot{\epsilon} \ , \tag{1}$$

where

ϵ = total creep strain,

t = time,

$\dot{\epsilon}$ = minimum creep rate, strain per unit of time,

a, n = constants.

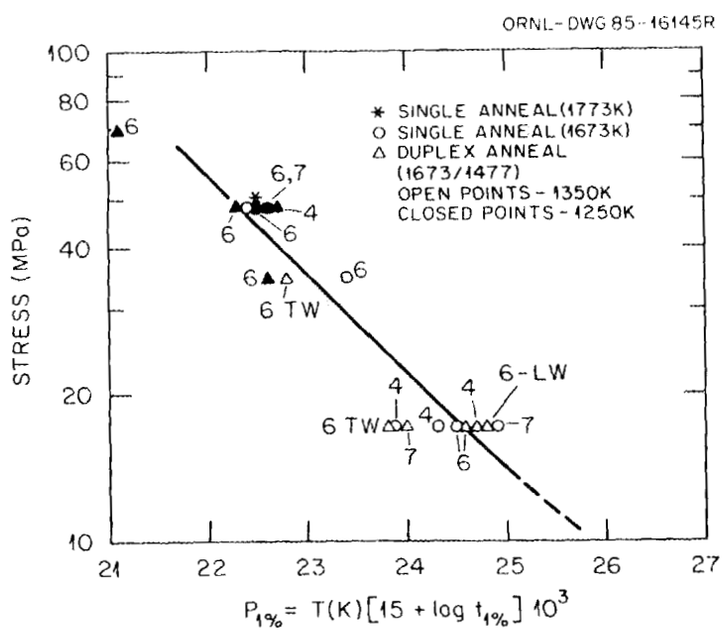

Fig. 9. Representation of test results for 1% creep strain in Nb-1% Zr based on Larson-Miller parameter. Numbers by points denote last nonzero digit in heat number. Symbols TW and LW refer to transverse and longitudinal welds, respectively.

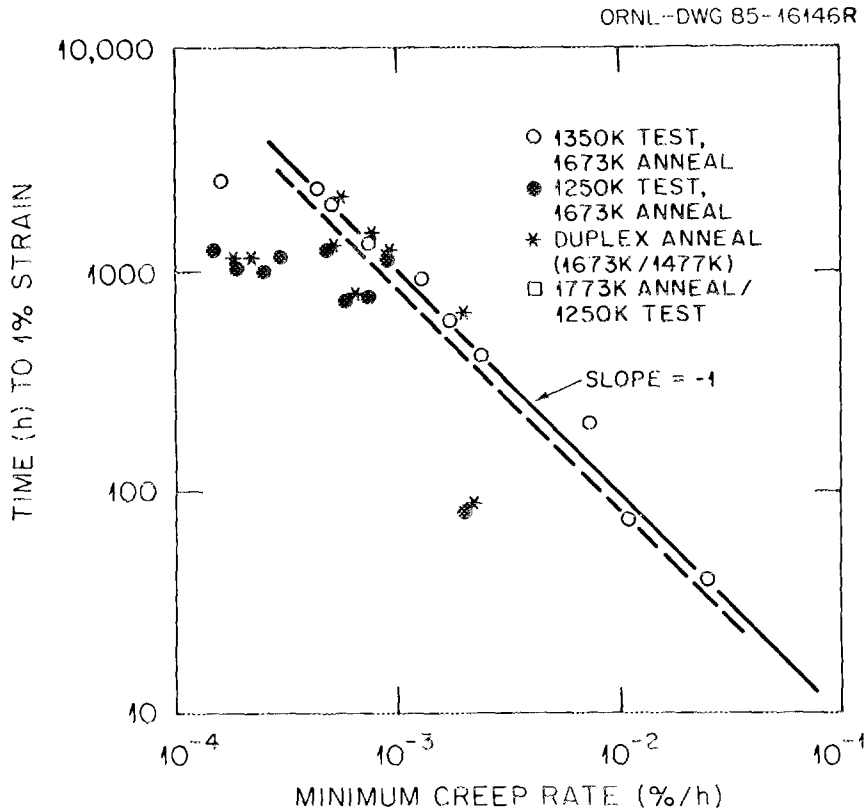

Fig. 10. Creep correlations for Nb-1% Zr.

The first term is the primary creep, and the second term is the minimum or steady-state creep. If the primary creep is negligible and the strain limit of interest is 1%, then Eq. (1) reduces to

$$t_1 \propto 1/\dot{\epsilon} \ , \tag{2}$$

where t_1 = time to 1% strain. Equation (2) can also be represented by

$$\log t_1 \propto \log \dot{\epsilon} \ . \tag{3}$$

These two factors are plotted in Fig. 10. The data for 1350 K are described well by the line with a slope of −1, so the assumption of the primary creep being negligible seems valid at this temperature. At 1250 K most of the data are not described well by the line with a slope of −1, so it appears that primary creep cannot be ignored in predicting the time to 1% strain.

Based on the results in Table 4 and the creep curves in the Appendix, the creep behavior at 1250 K appears to differ from that at 1350 K. At 1250 K there is a period of primary creep, a period of relatively slow creep, and a final period of dramatically increasing rate of creep. At 1350 K there is little if any primary creep, followed by a period of gradually increasing rate of creep which terminates in failure. This description of the differences in creep behavior is qualitative, and the exact differences vary with stress level as well as with test temperature.

Several creep samples were analyzed after testing; the results are given in Table 5. Since the samples were all tested in ion-pumped systems, the contamination level should have been and was very low. The three weld samples show that the weld deposit was contaminated with carbon in all cases and with oxygen in one weld.

The data are also analyzed in terms of the Orr-Sherby-Dorn parameter.[3] Horak[4] applied this parameter to a data base of 13 Nb-1% Zr tests run in lithium and obtained the best-fit equation:

$$\dot{\epsilon}_m = 1.7\sigma^{2.7}e^{-326,715(\mathrm{J/mol})/RT},$$

where

$\dot{\epsilon}_m$ = minimum creep strain rate (in./in./s),
R = gas constant (8.314 J/mol/K),
T = absolute temperature (K).

The line shown in Fig. 11 is from Horak's correlation, and the points are for the data in Table 4. The data fit the line reasonably well, but there is considerable scatter.

The data from this study determined at 1350 K correspond closely to the line developed by Horak, but the three data points determined at 1545 K fall significantly off the line. Thus the data from the present study likely correlate well by the Orr-Sherby-Dorn parameter, but the constants need to be different from those determined by Horak from his data set.

Several samples were analyzed for interstitials following testing, and the results are given in Table 5. The interstitial variations are within reasonable limits.

Table 5. After-test chemical analyses of Nb-1% Zr (ppm)

Test	Temperature (K)	Time (h)	Heat	Carbon	Oxygen	Nitrogen	Hydrogen
As received			12R-1614	21	205	66	2
24557	1350	145	12R-1614	46	220	62	<1
24556	1350	2076	12R-1614	39	249	57	5
24665	1250	1461	12R-1614	34	237	66	<1
24561	1350	4600	12R-1614	46	274	73	1
As received			530860	20	202	31	<1
24360	1250	1861	530860	46	278	31	1
24282	1350	3600	530860	55,53	221,215	54,33	1,1
24267	1350	668	530860	78	218	27	<1
24268	1350	384	530860 weld	163	230	20	<1
24485	1250	214	530860	68	260	38	1
24477	1250	1283	530860	79	312	40	1
24284	1250	2300	530860	81	239	34	1
24283	1350	2335	530860 weld	200	378	34	2
24514	1250	2070	530860	24	286	32	<1
24398	1350	4485	530860 weld	97	213	22	4
24398	1350	4485	530860 base	39	249	57	5
As received			530870	43	207	31	4
24748	1350	1485	530870	61	225	33	1
24683	1250	1465	530870	24	195	27	<1
24681	1250	2303	530870	34	231	30	<1

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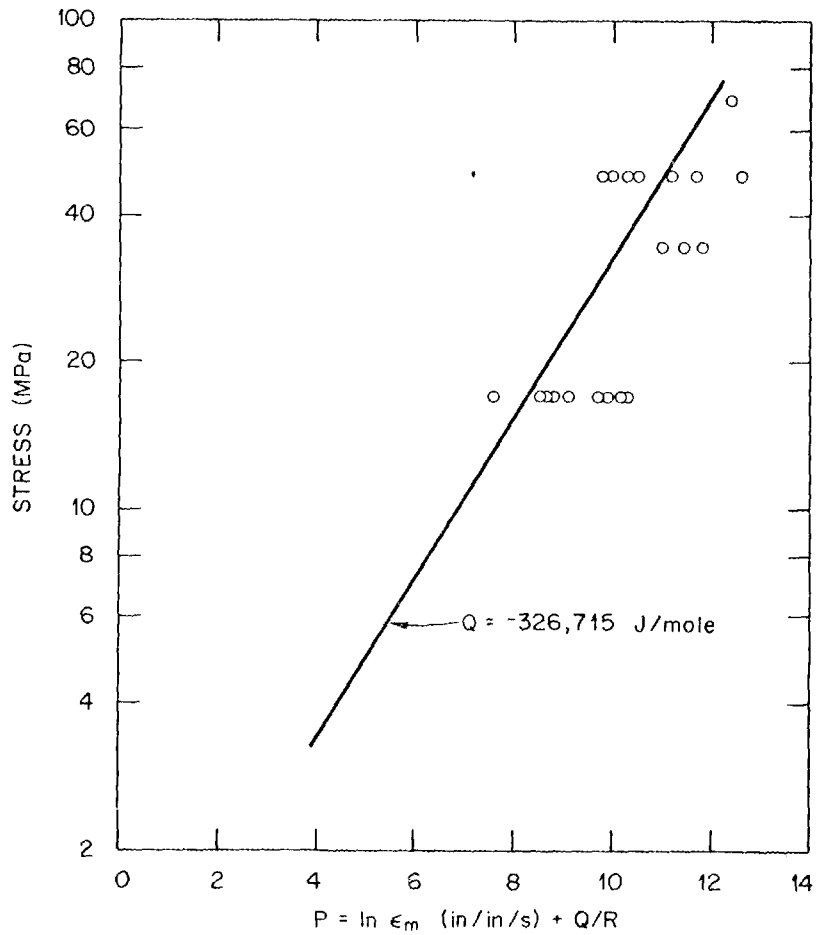


Fig. 11. Test results for Nb-1% Zr correlated using the Orr-Sherby-Dorn parameter. Line was determined by Horak from data in the literature.

CREEP DATA FOR PWC-11

Several samples were tested from a single heat of PWC-11; the results are summarized in Table 6, and individual creep curves for each test are contained in the Appendix. All tests were conducted in systems operating at 10^{-8} to 10^{-9} torr. Two anneals were investigated: a single anneal of 2 h at 1477 K and a duplex anneal of 1 h at 1755 K and 2 h at 1477 K. Two samples with a longitudinal electron-beam fusion weld were tested. Figure 12 shows the data from our tests and compares them with the line determined by Horak[1] for all available and reliable data. The comparison is made on the basis of the Larson-Miller parameter, and our results fall below those for other heats. According to private discussions, these

Table 6. Creep tests on PWC-11

Test	Pretest anneal (h/K)	Type specimen[a]	Stress (MPa)	Temperature (K)	Time to indicated strain (h)					Minimum creep rate (%/h)	Creep strain (%)
					1	2	5	t[b]	R		
24095	2/1477	B	68.9	1350	21	53	127	110	297	3.1 E-2	51.3
24104	1/1755, 2/1477	B	68.9	1350	102	147	205	108	347	6.9 E-3	48.7
24116	1/1755, 2/1477	B	48.3	1350	2150	3950		4000	5750[c]	4.6 E-4	4.0
24399	1/1755, 2/1477	LW	48.3	1350	760	1480	2320	1500	2594[c]	1.1 E-3	9.7
24742	1/1755, 2/1477	LW	75.0	1350	340	520	750	400	804	1.8 E-3	19.0
24093	2/1477	B	17.2	1545	65	135	265	200	648[c]	1.5 E-2	67.8
24094	1/1755, 2/1477	B	17.2	1545	90	210	322	140	646[c]	8.6 E-3	10.0
24117	1/1755, 2/1477	B	10.3	1545	420	825	1760	1100	2636[c]	2.2 E-3	11.2

[a]B = base, LW = longitudinal weld.

[b]Tertiary creep (based on 0.2% strain offset from minimum creep rate).

[c]Discontinued prior to failure.

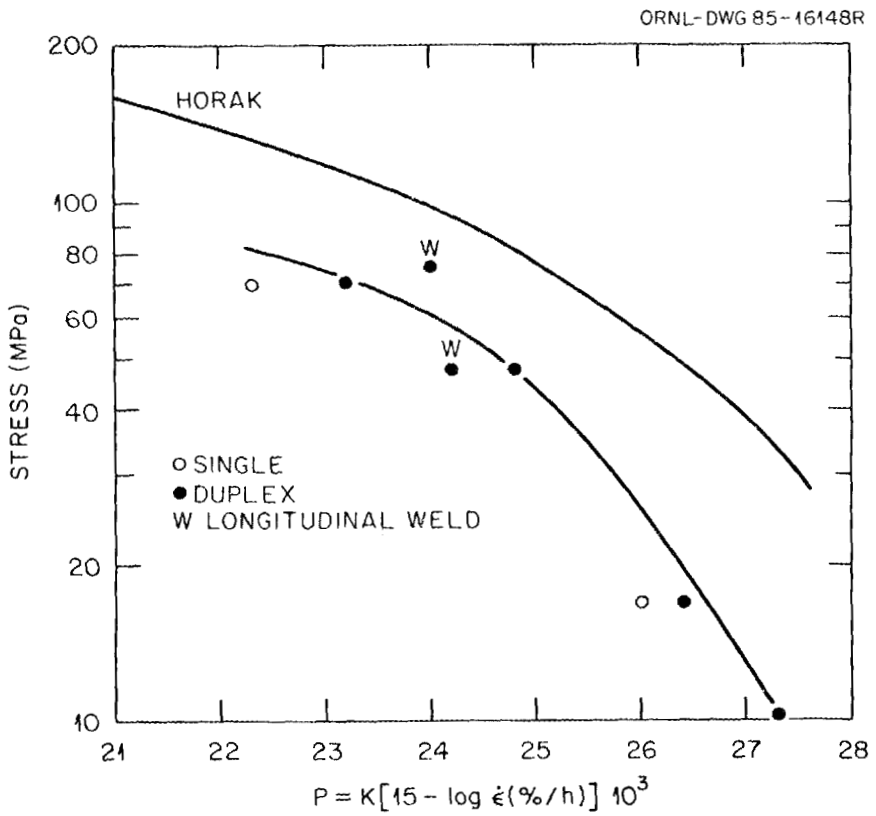


Fig. 12. Larson-Miller correlations for PWC-11 comparing Horak correlation with data from present study.

results are probably attributable to the fact that our material was extruded at a lower temperature than that used for other tests. The duplex anneal offers some strength increase over the single anneal. Our data for the time to 1% strain are correlated with the Larson-Miller parameter in Fig. 13. The data correlate well, but the two specimens with a single anneal and one of the weld samples fall short of the line from the other data.

The minimum creep rate and the time to 1% strain are correlated in Fig. 14. These data fit a line having a slope of −1 very well, so Eq. (3) would indicate that the contribution of primary creep is negligible at the 1% strain level.

The data were also analyzed in terms of the Orr-Sherby-Dorn parameter[3] (Fig. 15). Horak[4] applied this parameter to a base of 18 tests run in lithium and obtained the best-fit equation of:

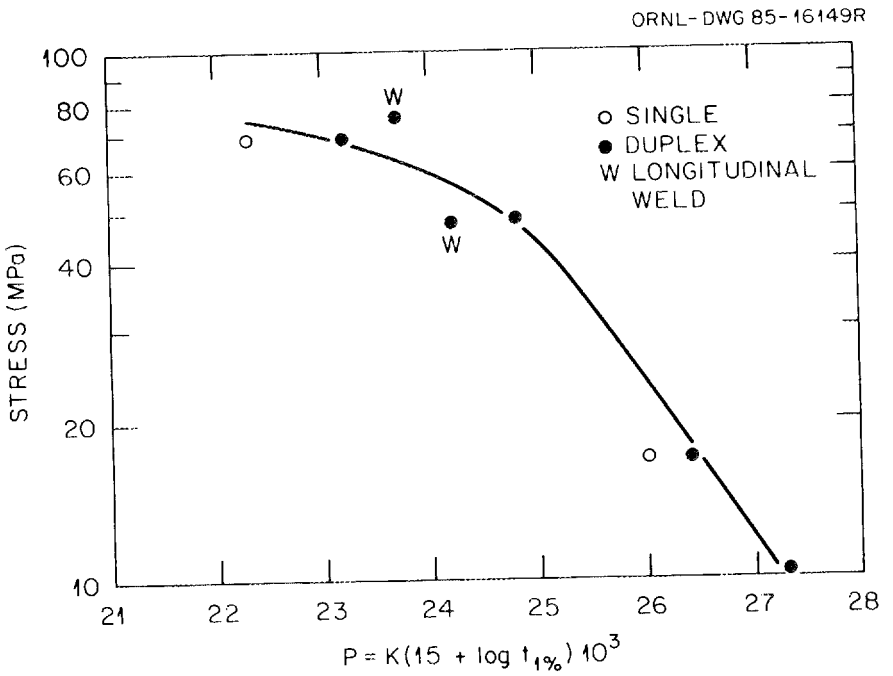


Fig. 13. Larson-Miller correlation for PWC-11 data from present study.

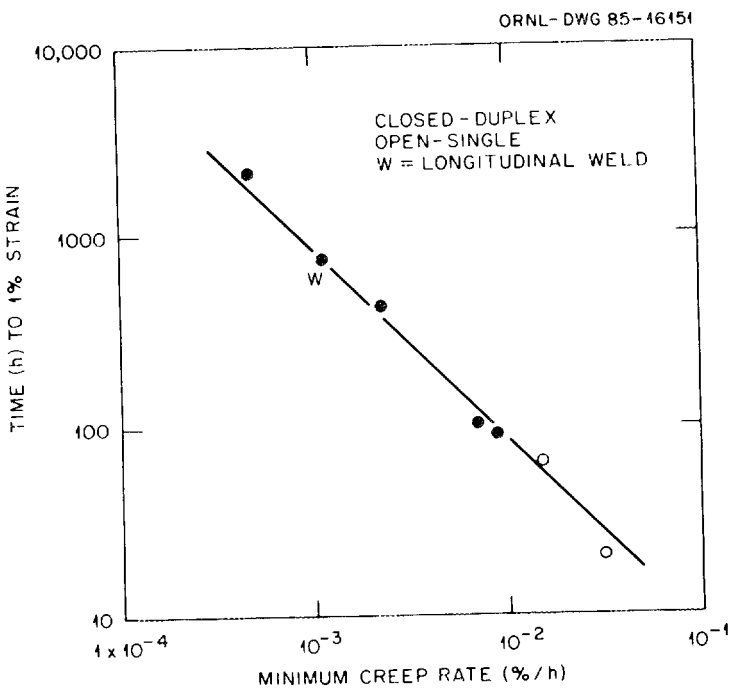


Fig. 14. Creep correlation for PWC-11 involving minimum creep rate and time to 1% strain.

$$\dot{\epsilon}_m = (3 \times 10^{-7})\sigma^{3.7} e^{-212,290(\mathrm{J/mol})/RT} ,$$

where

$\dot{\epsilon}_m$ = minimum creep strain rate (in./in./s),
R = gas constant (8.314 J/mol/K),
T = absolute temperature (K).

The results of posttest chemical analyses of tested specimens are given in Table 7. The results are somewhat erratic. The carbon content was generally lower after the test than before, but several samples were analyzed in the as-received condition and the carbon content varied from 550 to 900 ppm. Such a survey was not made with regard to oxygen content, so it is not known whether or not oxygen was distributed homogeneously within the starting material. One sample has significantly low oxygen content. It appears that the pickup of oxygen and carbon during the creep test is very small.

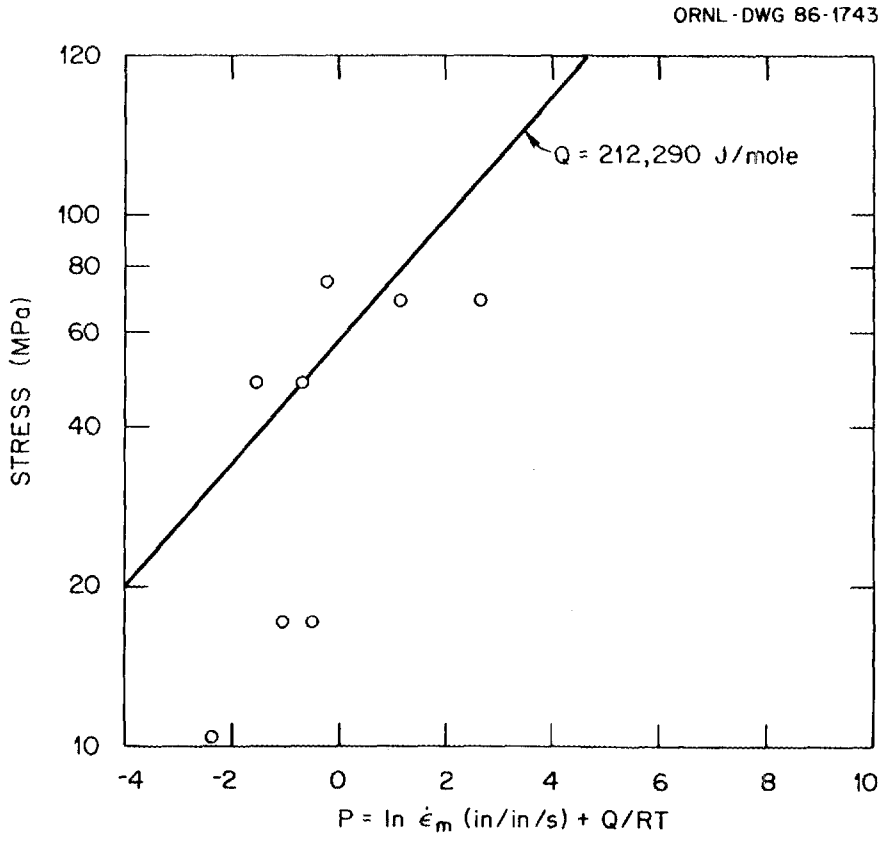


Fig. 15. Test results for PWC-11 correlated using the Orr-Sherby-Dorn parameter. Line was determined by Horak from data in the literature.

Table 7. Posttest chemical analyses of PWC-11 heat RS-8712 (ppm)

Test	Temperature (K)	Time (h)	Carbon	Oxygen	Nitrogen	Hydrogen
As received			890	110	60	11
24116	1350	5750	611,635	150,138	47,45	1,<1
24117	1545	2636	560	44	32	<1
24104	1350	347	740	122	29	<1
24093	1545	648	613	80	29	<1
24095	1350	297	597	159	35	<1
24742[a]	1350	804	706	196	45	<1

[a]Specimen had a longitudinal weld. Test was run in an oil-pumped system, and specimen was wrapped with Nb-1% Zr.

CREEP TEST RESULTS FOR Mo-Re ALLOYS

Three alloys were prepared by AMAX for LANL (these alloys were described in Table 1). Several tests were run on each of these alloys; the test results are summarized in Table 8, and selected creep curves are shown in the Appendix. All but the last two tests were run under multiple test conditions. Each test was either started at a fixed load and had the temperature increased or started at a fixed temperature and had the load increased. The cue to make a change was the specimen's creeping 1% or being at the same test conditions for 1000 h. The data in Table 8 show clearly that the two situations—incrementally increasing the stress or the temperature—result in different creep rates. In fact, the resulting minimum creep rates vary by as much as an order of magnitude. One possible explanation is that the first test condition determines substructures and that these substructures are not altered by further changes in the test conditions.

Figure 16 shows the results of tests using the minimum creep rate and the Larson-Miller parameter with a constant of 15. Only one specimen containing 9% Re was run. This specimen was held at a constant temperature and the stress was incremented. The three points for this test define the location of the left side of the scatterband in Fig. 16. Two tests were

Table 8. Creep tests on Mo-Re alloys annealed 1 h at 1650 K

Test	Alloy[a]	Stress (MPa)	Temperature (K)	Time to indicated strain (h)					Minimum creep rate (%/h)	Creep strain (%)
				1	2	5	t	R		
24141	Mo-9Re	13.8	1500	340				338[b]	2.8 E-3	1.0
		20.7	1500	97	230			246[b]	7.5 E-3	2.1
		27.6	1500	27	62	165	290	602	2.9 E-2	22.7
24098	Mo-11Re	13.8	1500					1004[b]	2.9 E-4	0.46
		20.7	1500					1004[b]	8.2 E-4	0.83
		27.6	1500					2059[c]	5.9 E-4	0.94
24127	Mo-11Re	20.7	1400	800				873[b]	1.1 E-3	1.1
		20.7	1500	200				214[b]	4.6 E-3	1.1
		20.7	1600	40	80	198	340	592[c]	2.5 E-2	16.4
24102	Mo-13Re	13.8	1500					792[b]	≤1.3 E-4	0.01
		20.7	1500					1007[b]	1.1 E-4	0.06
		27.6	1500	1640				1795[c]	6.2 E-4	1.1
24124	Mo-13Re	20.7	1400					1003[b]	3.1 E-4	0.3
		20.7	1500	900				973[b]	1.1 E-3	1.1
		20.7	1600	185	330	800	1580	2227[c]	6.2 E-3	16.7
24300	Mo-11Re	20.7	1400					4193[c]	3.6 E-5	0.7
24301	Mo-13Re	20.7	1400					4430[c]	1.3 E-4	0.5

[a]Mo-9 Re, H 390; Mo-11Re, H 389; Mo-13Re, H 375.

[b]Incremented to next step.

[c]Discontinued.

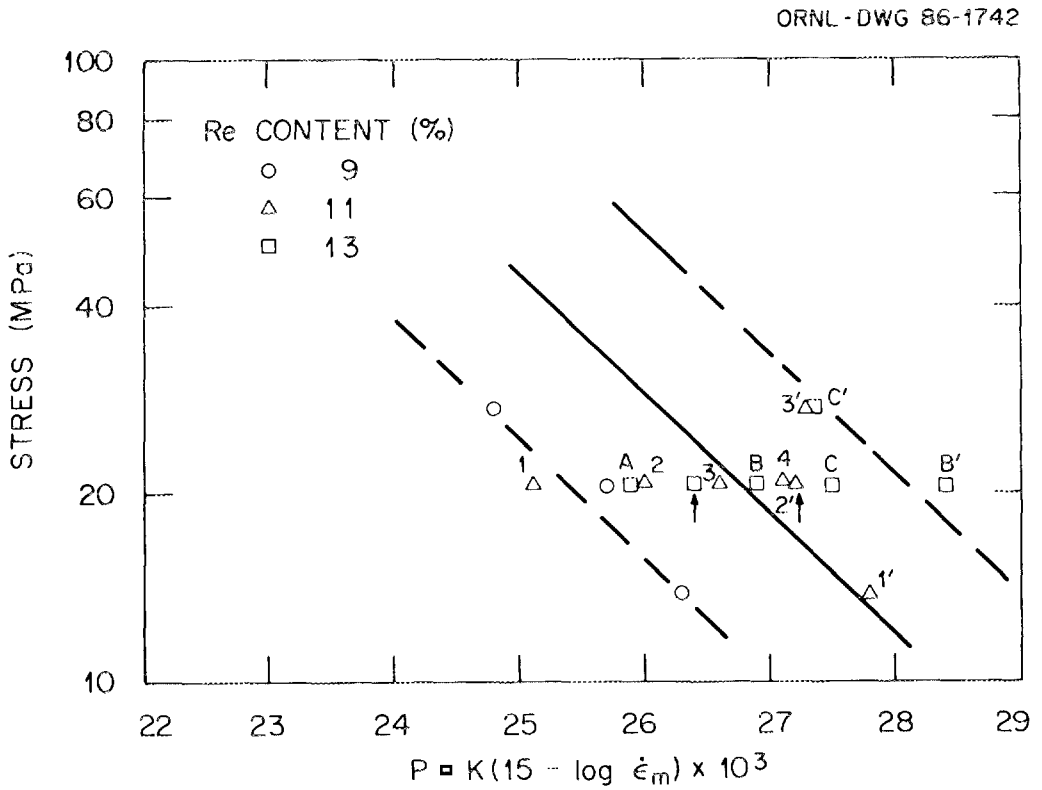


Fig. 16. Larson-Miller correlation for the minimum creep rate of Mo-Re alloys.

run on the alloy containing 11% Re. The triangular points noted 1, 2, and 3 represent stages of the test in which the stress was held constant and the temperature increased. The triangular points noted 1′, 2′, and 3′ are stages of a second test in which the temperature was held constant and the stress increased. Points 2 and 2′ each represent the stages of two tests at 20.7 MPa and 1500 K. Note in Table 8 that the creep rates for points 2 and 2′ vary by a factor of 5.

A similar series of tests are shown in Fig. 16 for the alloy containing 13% Re. The square points noted A, B, and C represent constant load and increasing temperature. The square points noted B′and C′ are for constant temperature and increasing load. Points B′ and B are for test conditions of 20.7 MPa and 1500 K. The results in Fig. 16 show that the same parameter was not obtained, and the data in Table 8 show that the creep rate actually varied by a factor of 10.

The last two tests shown in Table 8 were run under a constant stress of 20.7 MPa and 1400 K. The results for these tests are shown in Table 8 and on Fig. 16 with an arrow. The triangular points with a "1" and an arrow (↑) are for the same test condition for the 11% Re alloy, and the square points with an "A" and an arrow (↑) are for a 13% Re alloy with the same test conditions. There is considerable difference in both cases between the creep rates obtained by the two means of obtaining the same test conditions. Since 1 and A represent the first stages of the stepped testing program, it is very likely that the stepped tests were not run long enough to obtain the true minimum creep rate. However, these tests as a group show clearly that the creep response of the Mo-Re alloys is dependent upon the mechanical-thermal history.

Several of the tests in Table 8 reached 1% strain, so the Larson-Miller parameter is used in Fig. 17 to correlate these results. There are not as many test points as for the minimum creep rate, but the same trends apply as those just discussed for Fig. 16.

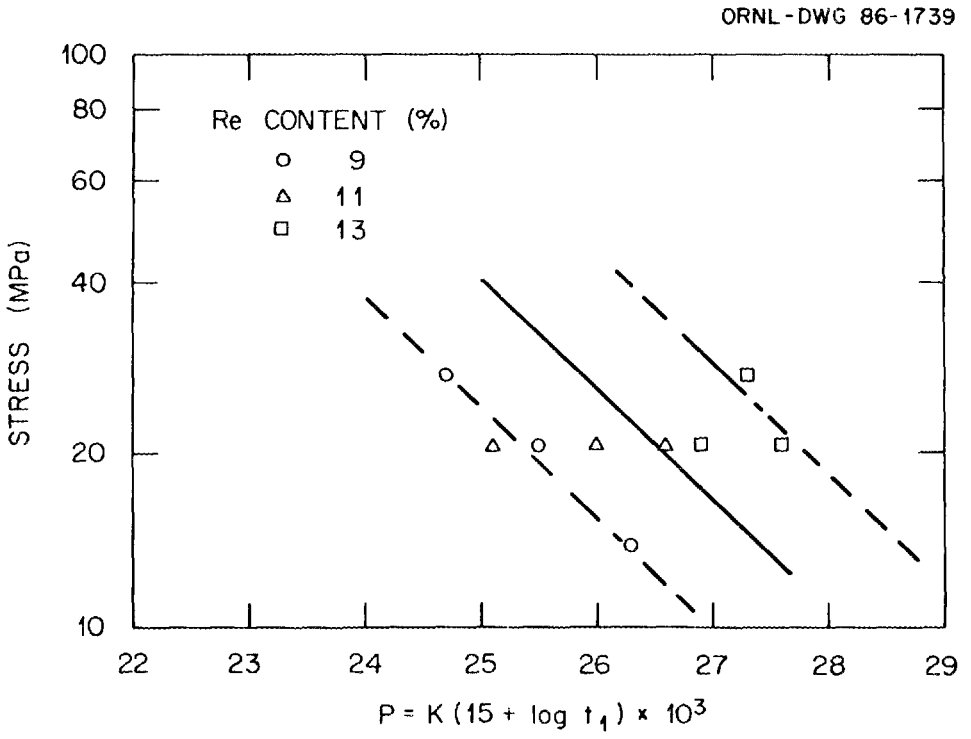


Fig. 17. Larson-Miller correlation for the time to 1% strain of Mo-Re alloys.

Another correlation of the Mo-Re data is shown in Fig. 18. The data for all three alloys seem to fall on lines having a slope of -1, so primary creep does not contribute significantly to the first 1% strain. The alloy containing 9% Re is represented by one line, and the data for alloys containing 11 and 13% Re are represented by another line.

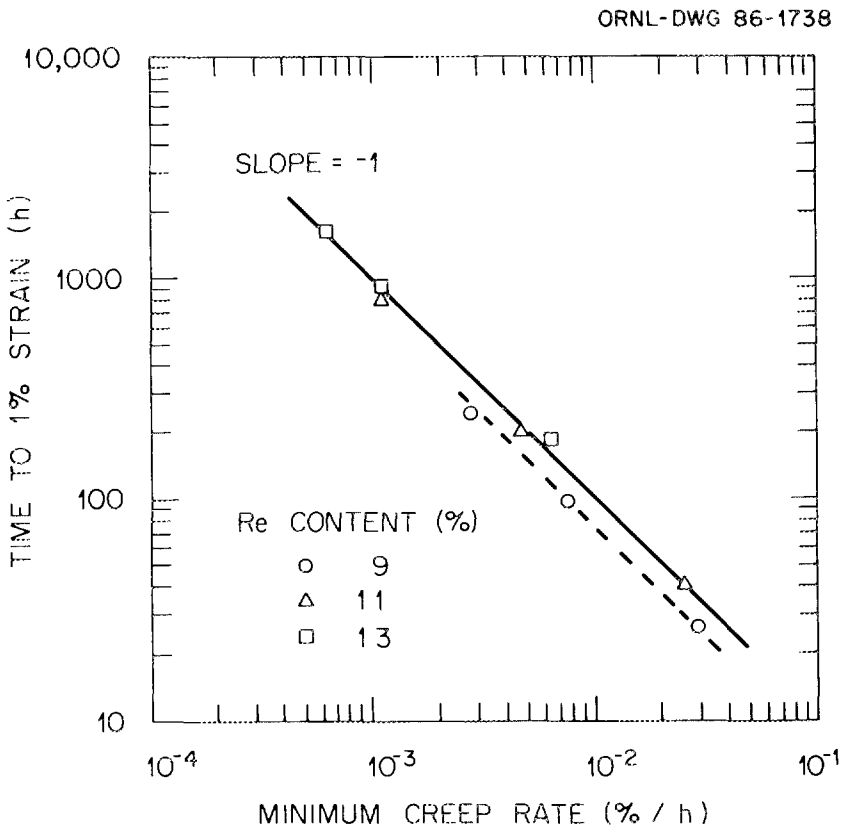


Fig. 18. Correlation of time to 1% strain with the minimum creep rate for Mo-Re alloys.

CREEP RESULTS FOR CVD-W

Five creep tests were run on CVD-W; the results are summarized in Table 9. This material is very hard and brittle, and special test techniques must be developed. Having tested much of this material in past years, we ran the tests described in Table 9 to renew our abilities in this test field. CVD-W exhibits very little primary creep; therefore, there is not much error in comparing the results on the basis of the

Table 9. Creep tests on CVD-W – pretest anneal of 1 h at 2000 K

Test	Lot	Stress (MPa)	Temperature (K)	Time to 1% strain (h)	Minimum creep rate (%/h)	Test time (h)	Creep strain (%)	Comments
24634	117	20.7	1873		5.5 E-4	603[a]	0.4	
24633	117	55.2	1673		8.3 E-4	738	0.85	Failed at flaw
24668	5235-141	20.7	1873	410	1.5 E-3	975[a]	10.6	
24671	5235-141	55.2	1673	52	1.5 E-2	216	12.6	
24701	5235-141	34.5	1673		4.2 E-5	2410[b]	0.7	

[a]Discontinued prior to failure.

[b]In progress.

Larson-Miller parameter with time to 1% strain either measured or calculated from the minimum creep rate. This comparison is made in Fig. 19 with the line coming from Horak's analysis[1] and our five test points superimposed for comparison. Our five samples were annealed at 2000 K before testing, and most of the test samples in Horak's analysis[1] were unannealed. However, our results are described well by the line constructed by Horak.[1]

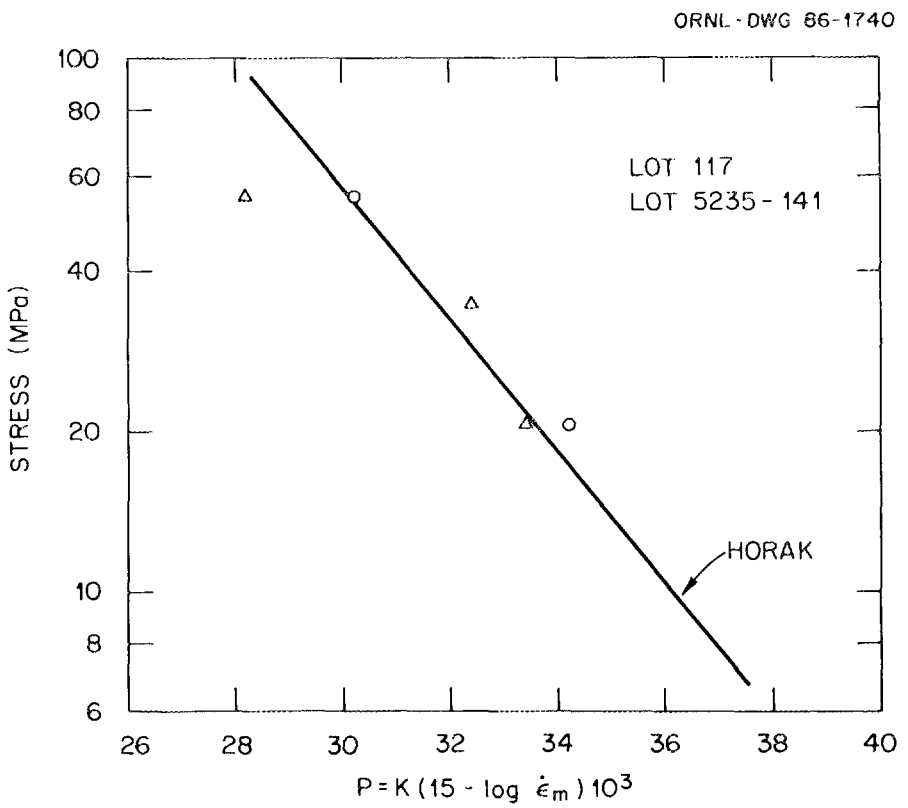
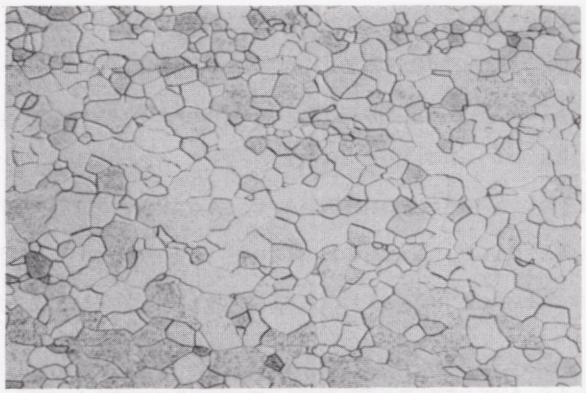


Fig. 19. Larson-Miller correlation for CVD-tungsten. Points are from this study; line is from evaluation of literature by Horak.

METALLOGRAPHIC STUDIES

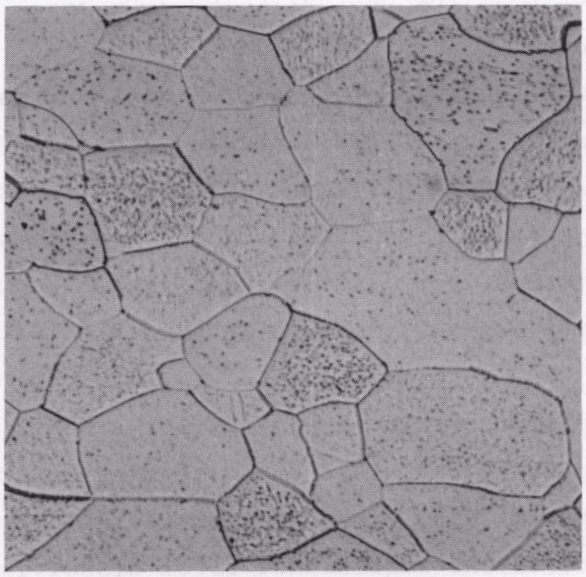
Several samples were examined metallographically. Heat 1614 (Nb-1% Zr) is shown in Fig. 20 after the material was annealed at 1673 K and tested at a range of temperatures varying from 1273 to 1473 K. The photomicrograph taken at the lower magnification shows that the material is reasonably fine-grained; specifically, the grain size number is 6.7.

Y-200475



(*a*) 200 μm

Y-200476



(*b*) 40 μm

Fig. 20. Heat 1614 of Nb–1% Zr annealed 1 h at 1673 K and creep tested about 3000 h at temperatures from 1273 to 1473 K.

The sample, used in one of the first tests in which the vacuum was not as low as desired, increased in oxygen content by about 100 ppm. The fine precipitate in the photomicrograph taken at the higher power of magnification of Fig. 20 is likely zirconium-interstitial compounds.

Photomicrographs of another Nb-1% Zr sample are shown in Fig. 21. This sample is of heat 530860 and was tested at 1250 K and 48 MPa for 2300 h. The test was discontinued after 10% strain. The grain size number is 6.5. The higher magnification view shows that some precipitation occurred.

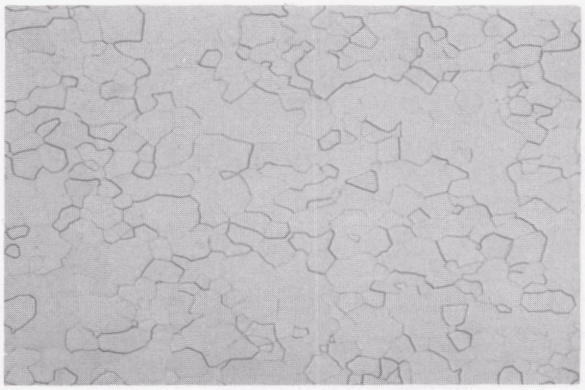
An electron beam fusion pass was run across a tab of Nb-1% Zr (heat 530860); typical photomicrographs are shown in Fig. 22. The fused material has a grain size number of about 2. The base metal has a grain size number of about 6.5, the heat-affected zone seems very narrow, and the weld appears sound.

Figure 23 shows photomicrographs of an Nb-1% Zr sample with a transverse weld. The sample was welded, annealed 1 h at 1673 K, and tested at 1350 K and 12.2 MPa. After 2337 h and 10% strain the test was discontinued. The base metal has a grain size number of 6.0 and the weld metal a grain size number of 1.6; thus, very little grain growth occurred during the testing (compare with Fig. 22). There is some precipitation, particularly in the weld metal.

Photomicrographs are shown in Fig. 24 for a Nb-1% Zr specimen following annealing for 1 h at 1773 K and testing at 1250 K and 48 MPa. The grain size number is 4.6. There is considerable precipitate formation, probably due to the sequence of first dissolving interstitial impurities at 1773 K and then reprecipitating them during testing at 1250 K.

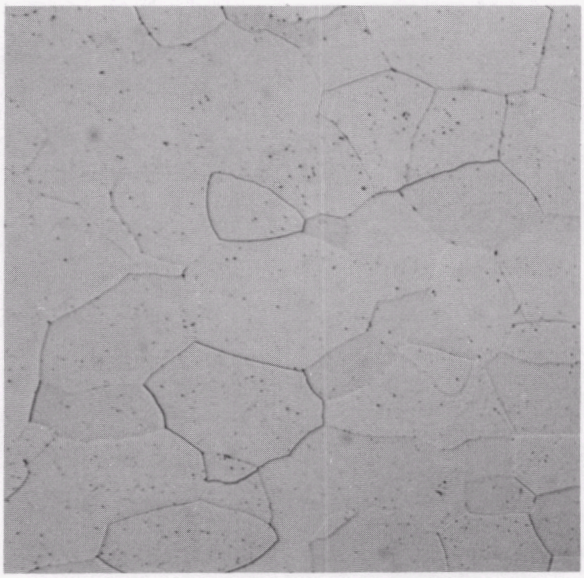
Photomicrographs of the PWC-11 used in our study are shown in Fig. 25. The material is heavily cold-worked, and although there are numerous stringers, probably carbides, there is no evidence of voids or other defects. The sample shown in Fig. 26 was annealed 2 h at 1477 K and creep tested at 1250 K and 69 MPa. The grains are elongated due to the 51% strain that occurred during the test. The grain size number is 7.8. The precipitates in this sample are very fine, as are those noted in Nb-1% Zr. The sample shown in Fig. 27 was given a duplex anneal prior to testing

Y203640



200 μm

Y203639



40 μm

Fig. 21. Heat 530860 of Nb–1% Zr following an anneal of 1 h at 1673 K and creep testing 2300 h at 1250 K and 48 MPa.

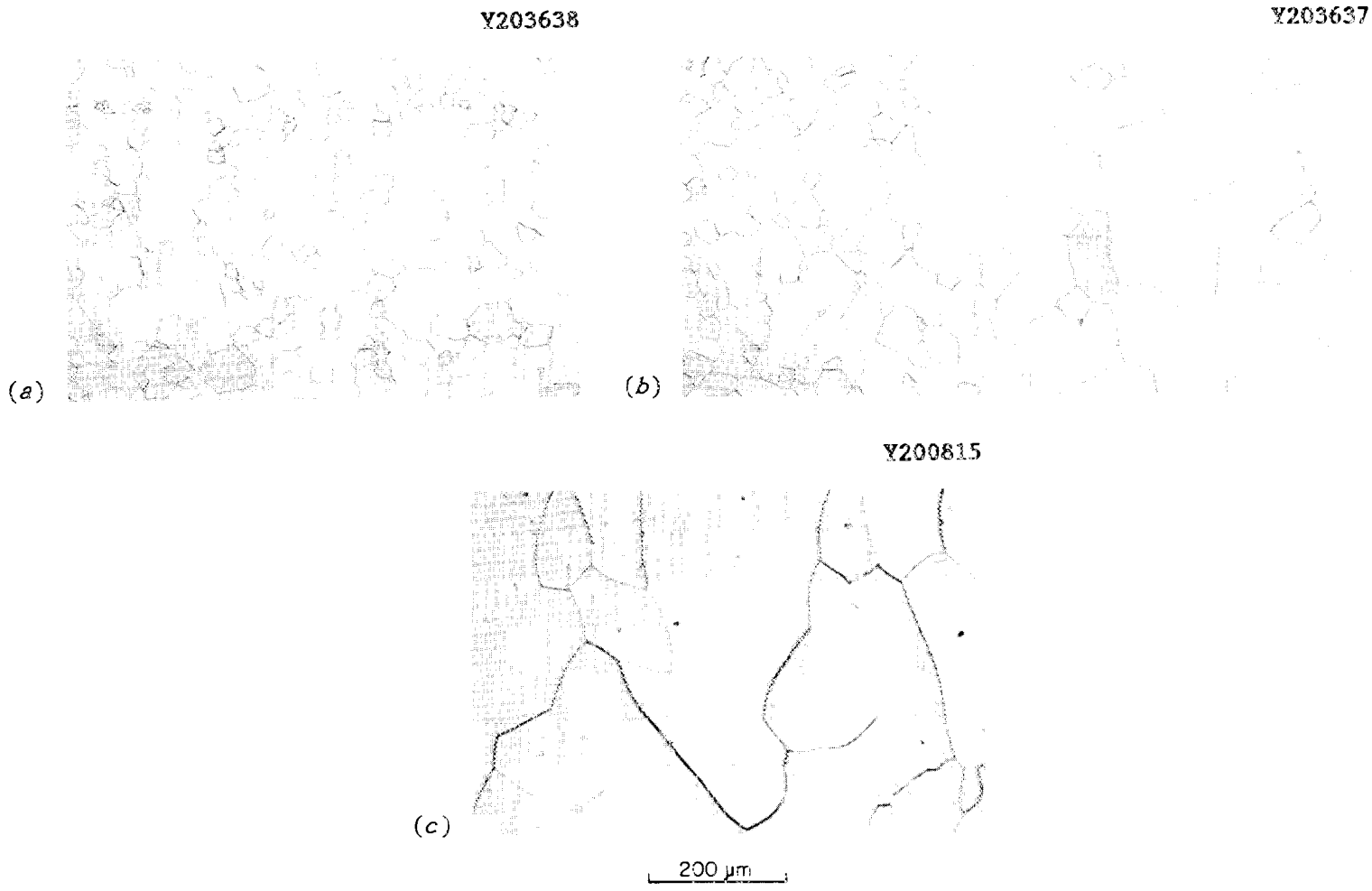


Fig. 22. Heat 530860 of Nb–1% Zr electron-beam-welded and annealed 1 h at 1673 K. (*a*) Base metal. (*b*) Heat-affected zone. (*c*) Weld metal.

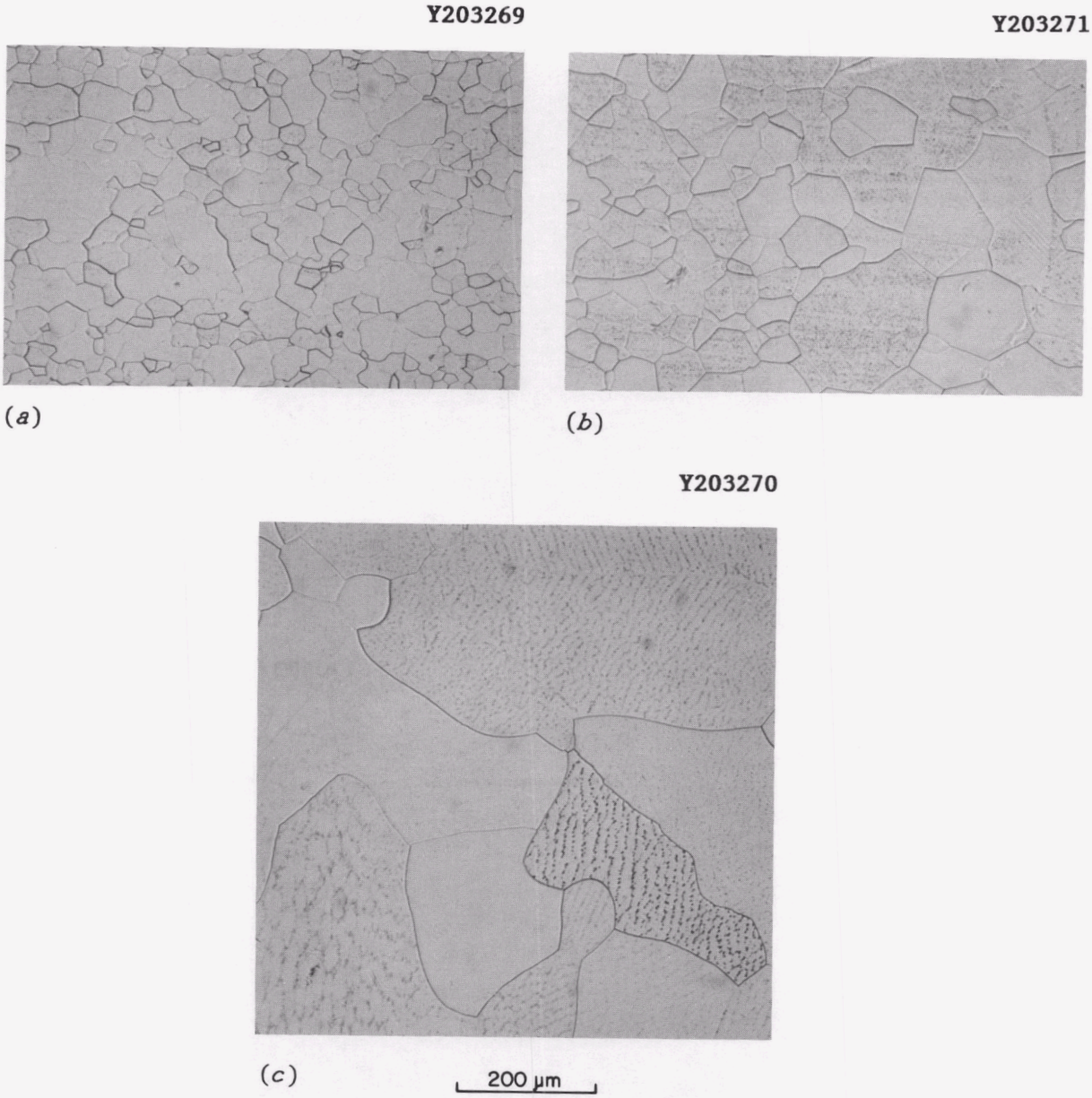
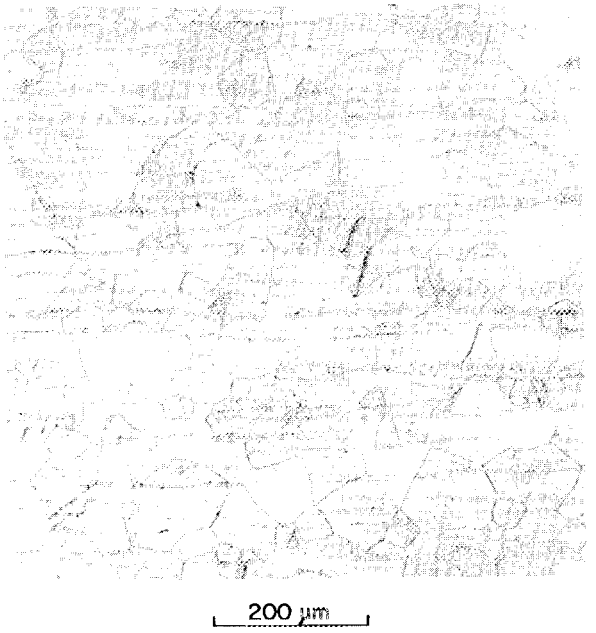


Fig. 23. Heat 530860 of an Nb-1% Zr sample with a transverse electron-beam weld annealed 1 h at 1673 K and tested at 1350 K and 17.2 MPa. Test was discontinued with 10% strain after 2337 h. (*a*) Base metal. (*b*) Heat-affected zone. (*c*) Weld metal.

Y204836



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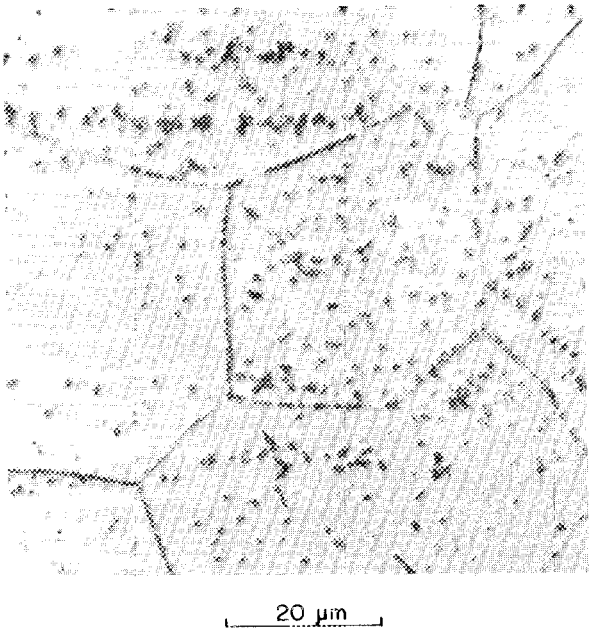


Fig. 24. Photomicrographs of Nb–1% Zr (heat 530870) after being annealed 1 h at 1773 K and tested at 1250 K and 48 MPa. Discontinued after 1365 h and 2.5% strain. Etched.

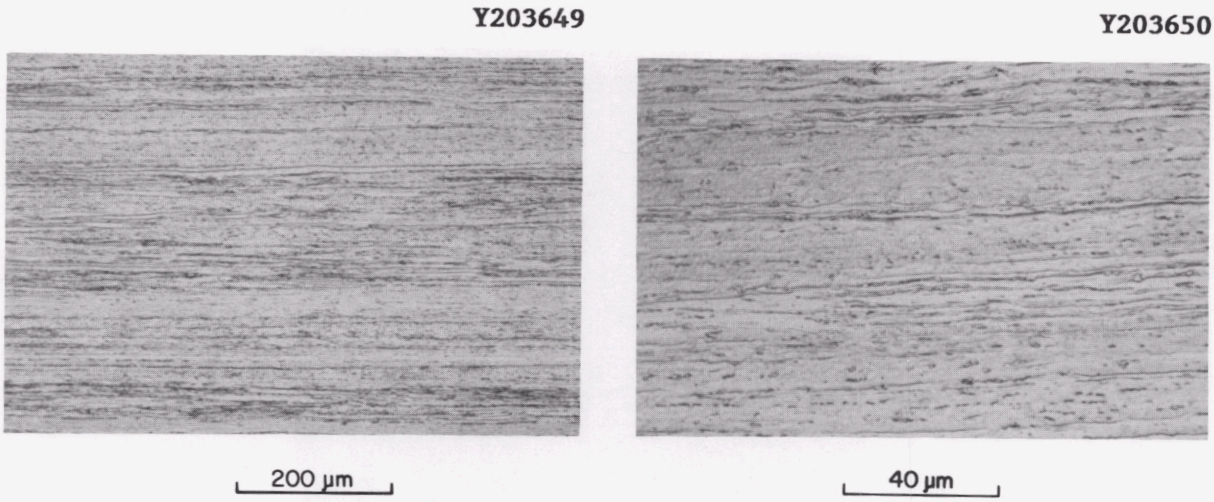


Fig. 25. Longitudinal view of PWC-11 in the as-received condition.

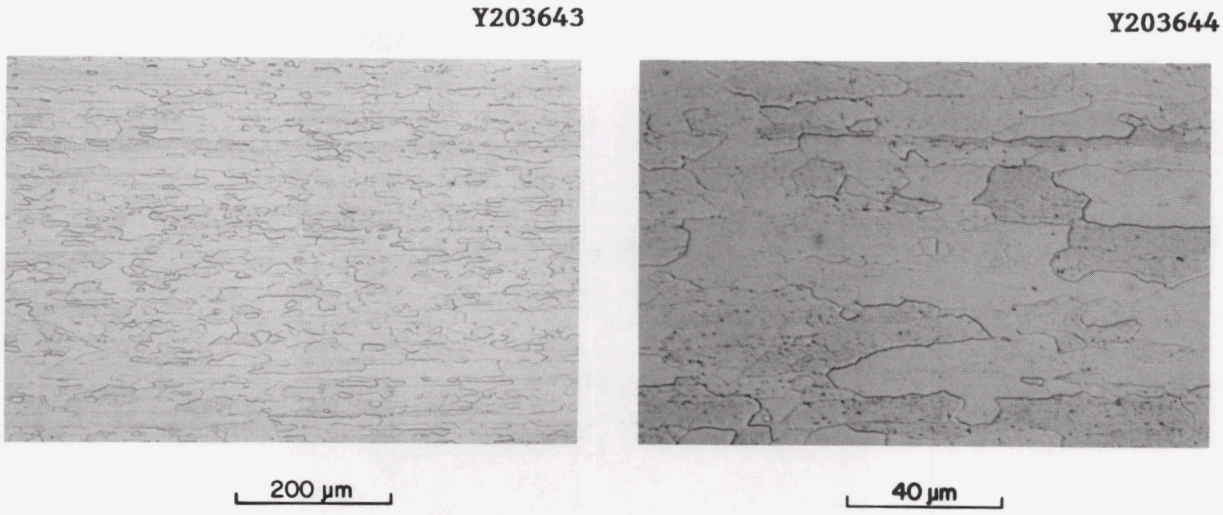
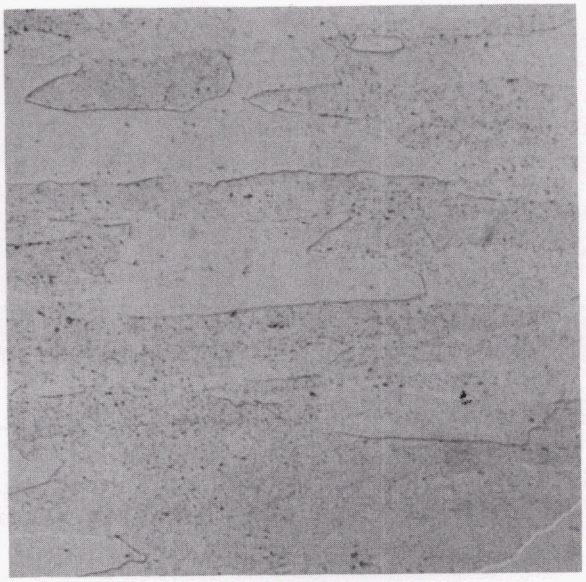


Fig. 26. PWC-11 following an anneal of 2 h at 1477 K and creep testing at 1250 K and 69 MPa. Test sample failed after 297 h with 51% strain.

Y203645

200 μm

Y203646



40 μm

Fig. 27. PWC-11 following an anneal of 1 h at 1755 K and 2 h at 1477 K and creep testing at 1250 K and 69 MPa. Test sample failed after 347 h with 47% strain.

consisting of 1 h at 1755 K and 2 h at 1477 K. The sample was tested at 1250 K and 69 MPa and failed after 347 h with 49% strain. The grain size number is 7.1, and the precipitate is very fine.

Figure 28 shows photomicrographs of a PWC-11 sample given the duplex anneal and tested for a long time at 1250 K and 48 MPa. The test was discontinued after 5750 h with 4% strain. The grain size number is 7.5. The precipitates vary in size but are all reasonably fine. The sample shown in Fig. 29 was given the duplex anneal and tested at 1545 K and 10.3 MPa. The test was discontinued after 2636 h with 11.2% strain. Under these test conditions, the grains became more equiaxial, and the grain size is 7.1. The precipitates are coarser than those noted previously in Figs. 26 through 28 for a lower test temperature.

A macrograph is shown in Fig. 30 of the PWC-11 sample from test 24399. The longitudinal electron beam weld was made over a length of about 20 mm. The weld region is wider than the adjacent material, indicating that the weld deformed more slowly than the adjacent material. The data in Table 6 show that the welded sample crept more rapidly than a base metal specimen tested at the same conditions. This seems to indicate that the heat-affected regions on each end of the weld creep faster than the weld metal or the normal base metal.

The Mo-Re alloys were provided by AMAX in the form of 3-mm-thick sheets and were annealed for 1 h at 1650 K. Small tabs were taken for metallographic examination. The grains were elongated slightly in one direction, which was referred to as the longitudinal direction. Typical photomicrographs are shown in Figs. 31 through 33 for the samples containing 9, 11, and 13% Re. The grain size numbers measured for those three samples were 4.7, 5.7, and 4.8, respectively. The alloys containing 9 and 11% Re were quite homogeneous, and all constituents appeared to be dissolved. The alloy containing 13% Re also contained 75 ppm carbon; therefore, the fine precipitates shown in the higher-magnification photomicrograph in Fig. 33 are probably carbides.

The three Mo-Re alloys were also viewed with the scanning electron microscope. The alloys appeared to be quite homogeneous, and the measured concentrations of rhenium were quite close to the nominal values. No further chemical studies were performed on these samples.

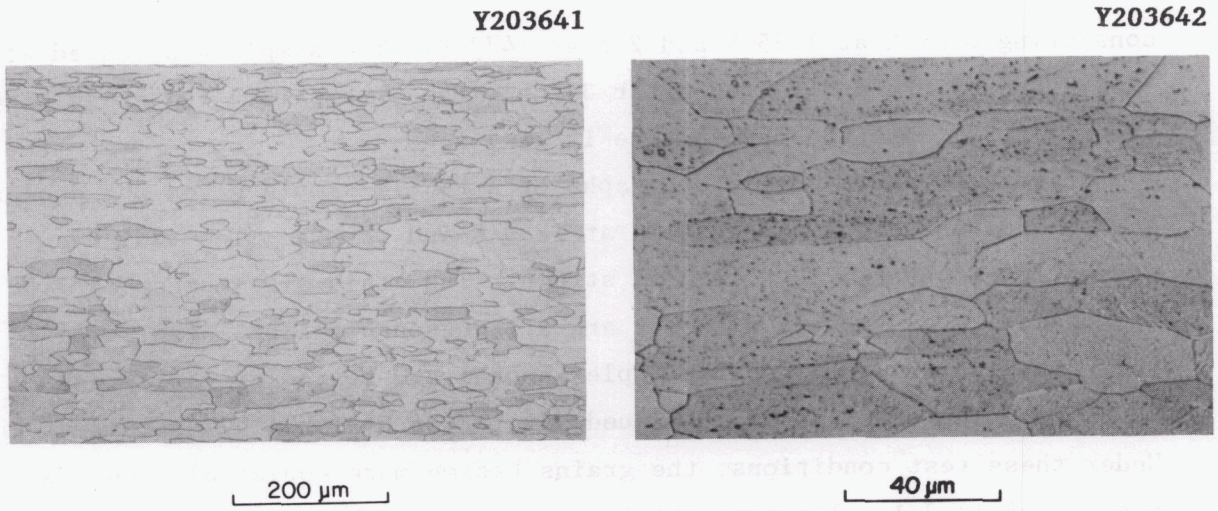


Fig. 28. PWC-11 following an anneal of 1 h at 1755 K and 2 h at 1477 K and creep testing at 1250 K and 48 MPa. Test discontinued after 5750 h and 4% strain.

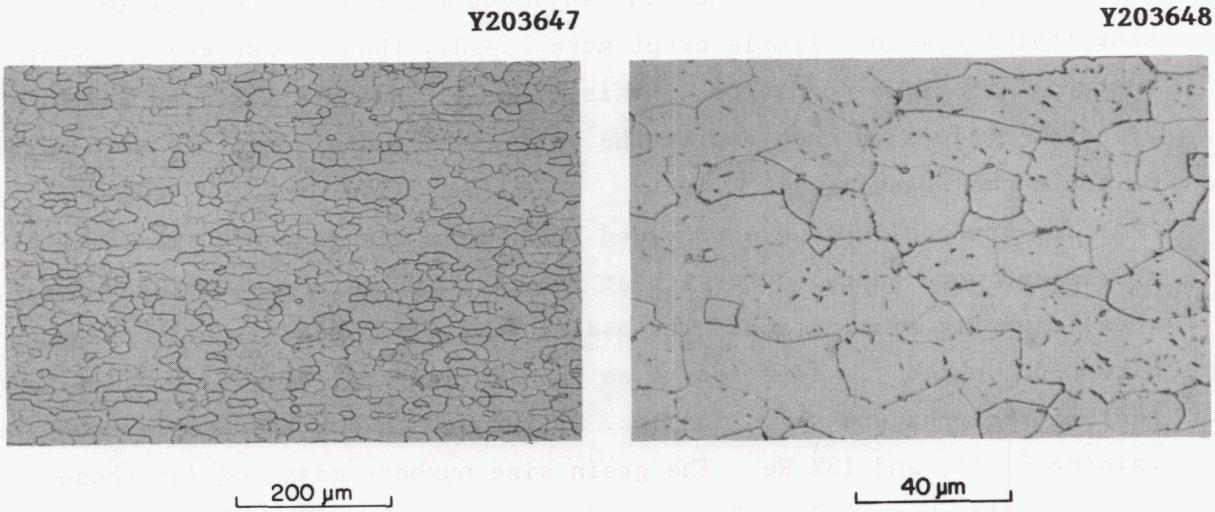


Fig. 29. PWC-11 following an anneal of 1 h at 1755 K and 2 h at 1477 K and creep testing at 1545 K and 10.3 MPa. Test discontinued after 2636 h and 11.2% strain.

YP91

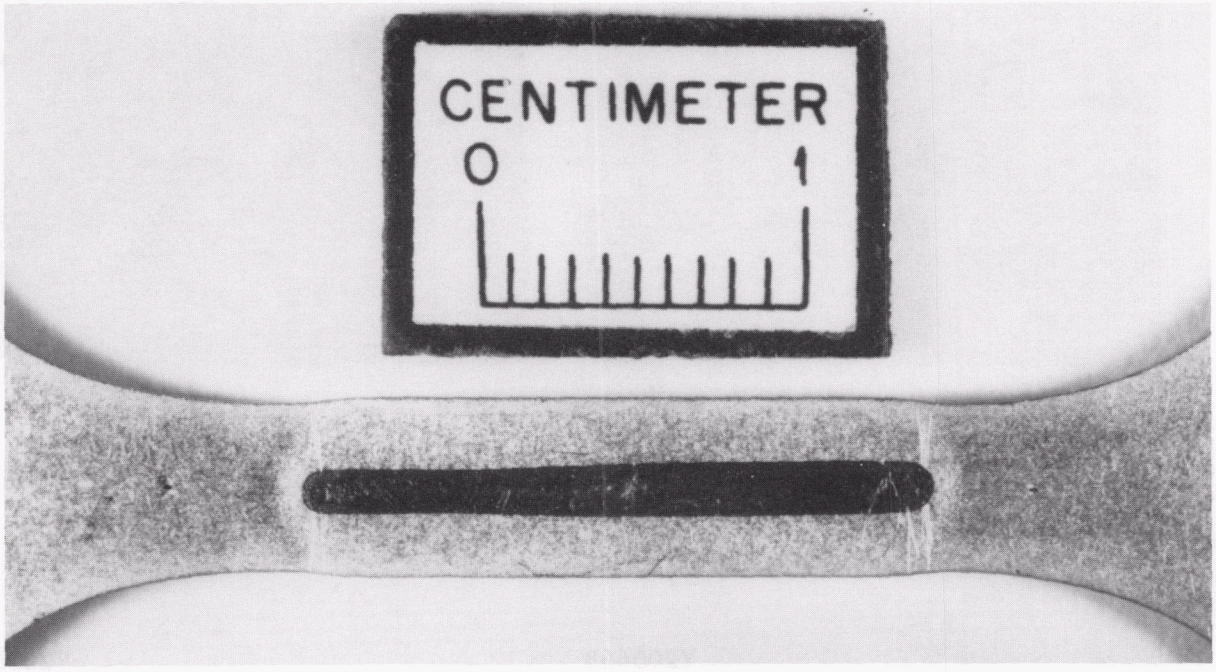


Fig. 30. Macrophotograph of PWC-11 specimen from test 24399. Width of specimen is about 6 mm, length of weld is about 20 mm, and the distance between the holes used to measure the strain is 25 mm. Note that the sample is wider where welded, indicating that the weld is stronger than the base metal.

Photomicrographs of one lot of CVD-W evaluated in our studies are shown in Figs. 34 through 36. These are views across the thickness direction. The top of the deposit was in contact with the molybdenum mandrel, and the lower end was in contact with the reacting gases. The structure in Fig. 34 is typical of this product, with small grains near the mandrel changing rapidly to large columnar grains. The sample shown in Fig. 35 was annealed 1 h at 2000 K; the main change is the reduction in the number of the very small grains. Annealing 16 h at 2000 K (Fig. 36) has very little effect on grain size over that observed in Fig. 35 after 1 h. This material has about 5 ppm fluorine, but there is no evidence that bubbles have formed in the material while annealing in the unstressed condition. In testing, the samples are oriented so that the stress is applied in a direction perpendicular to the long axis of the columnar grains.

Y203666 Y203667

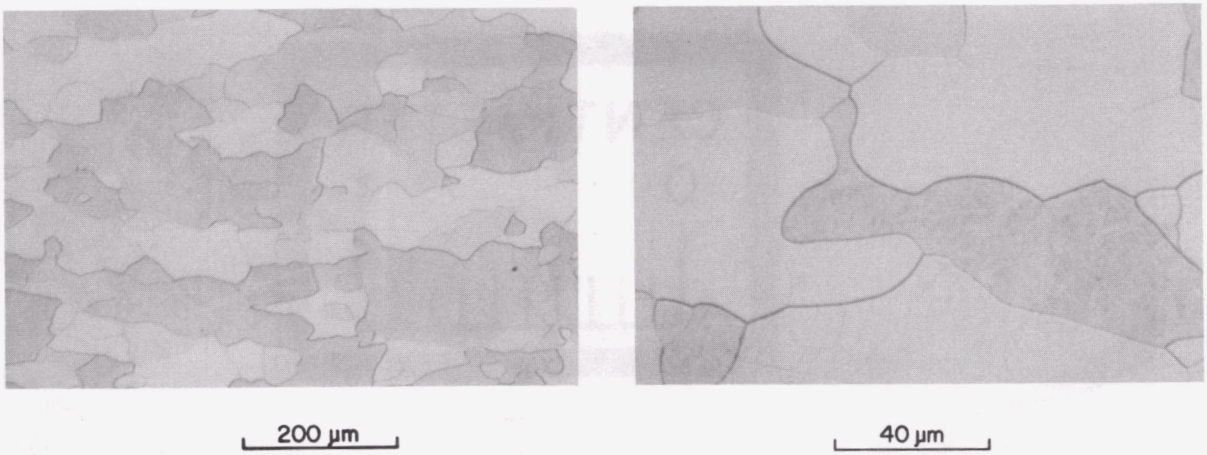


Fig. 31. Transverse section of Mo-9Re (lot 390) annealed 1 h at 1650 K.

Y203668 Y203669

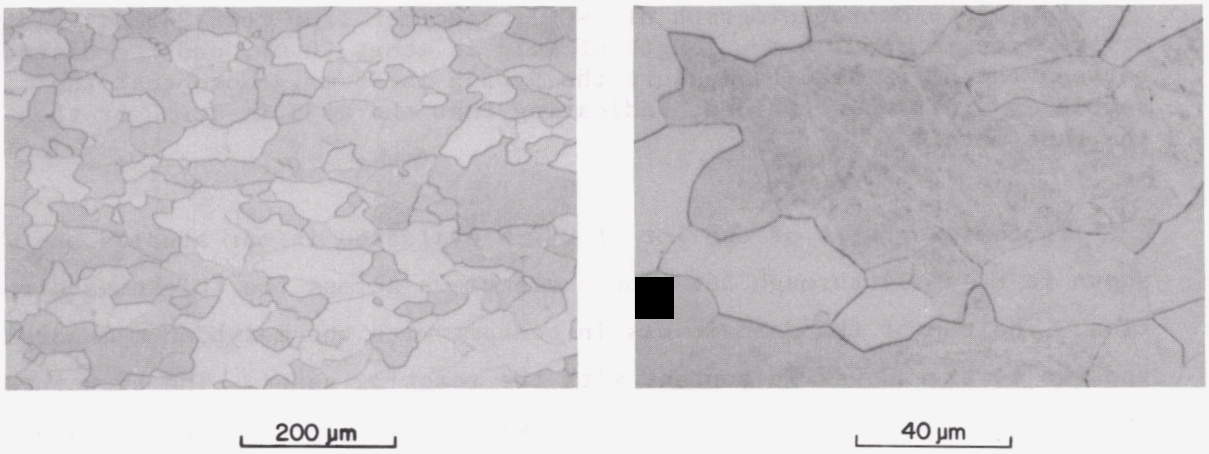
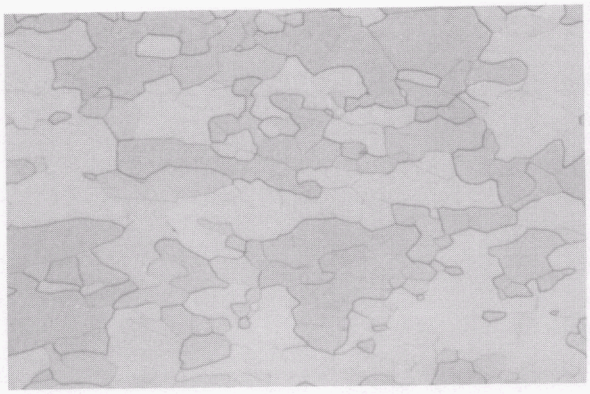


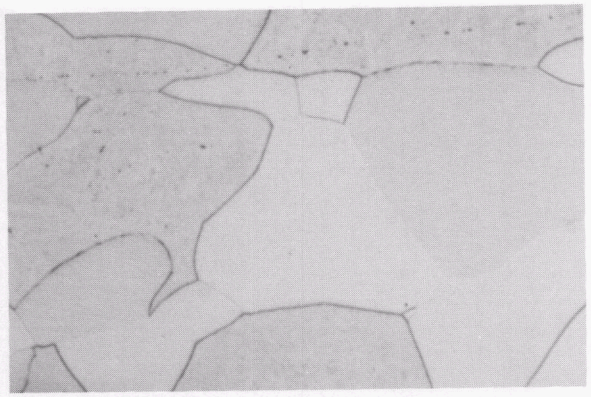
Fig. 32. Transverse section of Mo-11Re (lot 389) annealed 1 h at 1650 K.

Y203670



200 μm

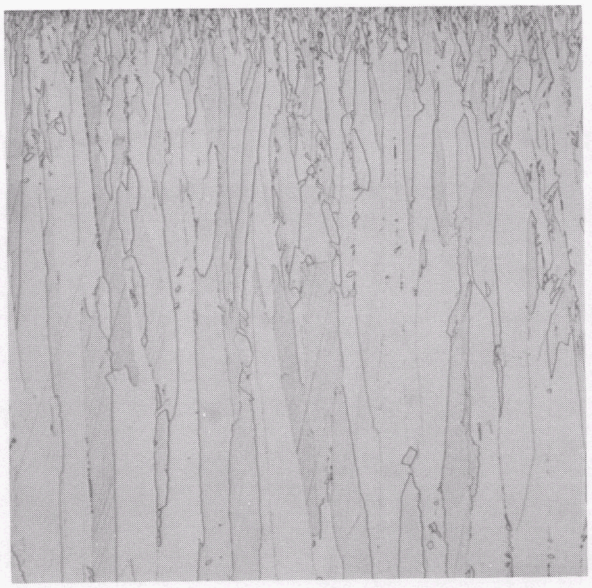
Y203671



40 μm

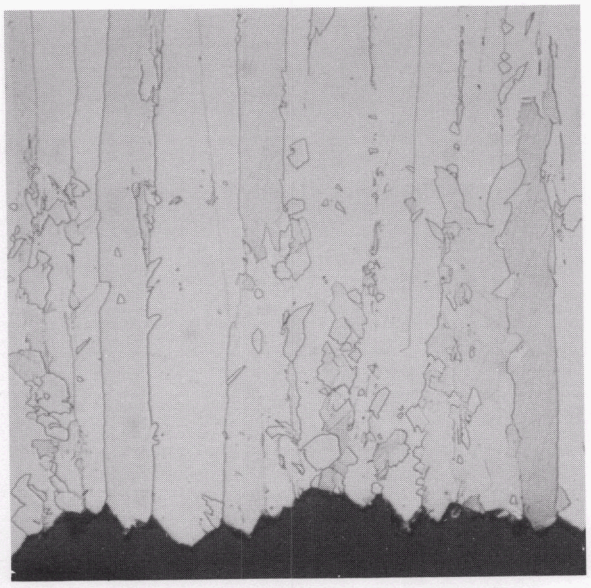
Fig. 33. Transverse section of Mo-13Re (lot 395) annealed 1 h at 1650 K.

Y203628



200 μm

Y203629



200 μm

Fig. 34. Transverse section of CVD-W (lot 117) in the as-deposited condition.

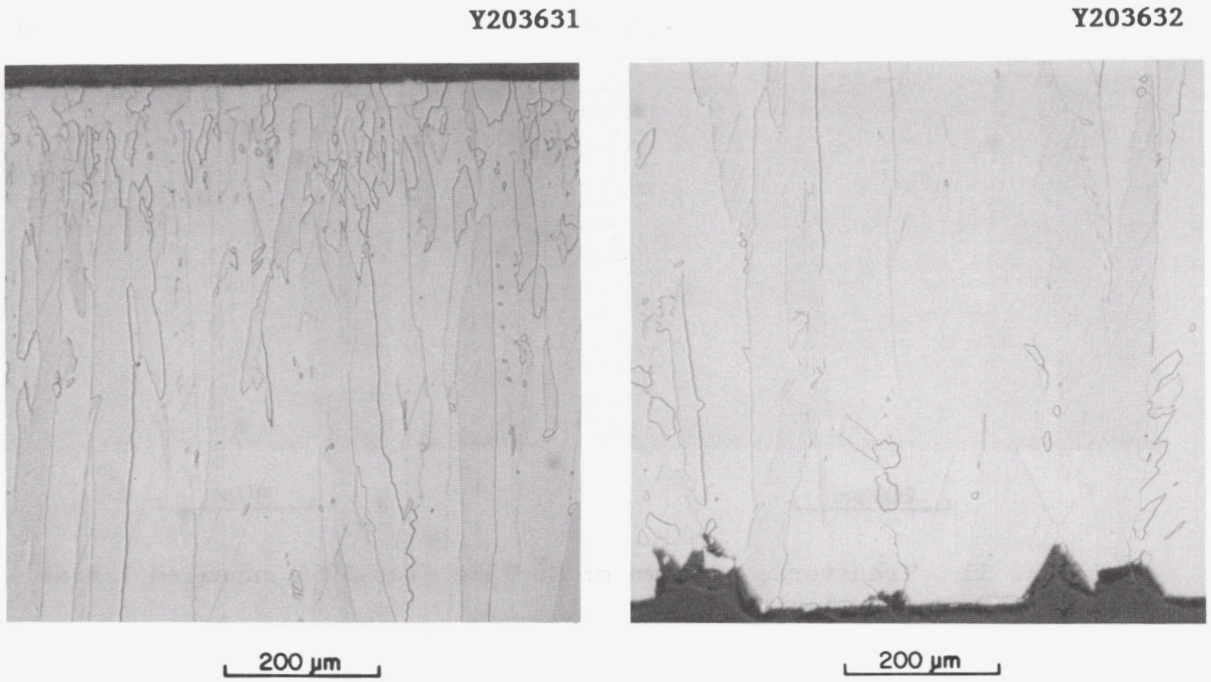


Fig. 35. Transverse section of CVD-W (lot 117) annealed 1 h at 2000 K.

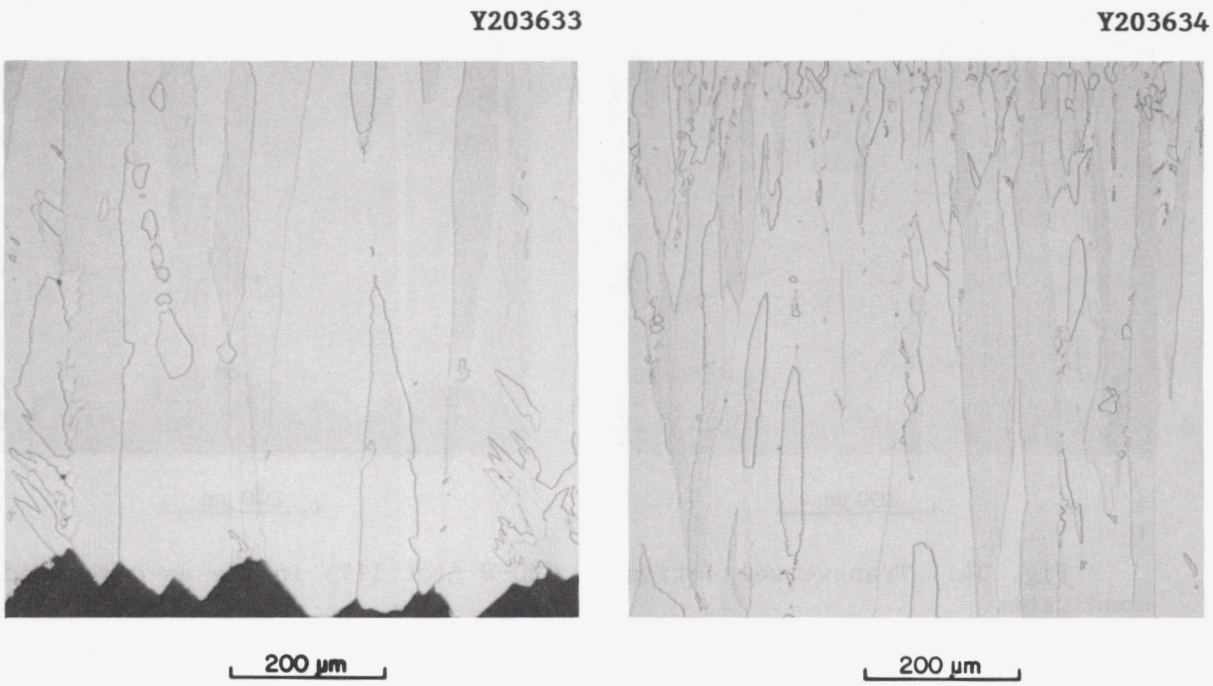


Fig. 36. Transverse section of CVD-W (lot 117) annealed 16 h at 2000 K.

DISCUSSION OF RESULTS

This study has involved four materials: Nb-1% Zr, PWC-11, Mo-Re alloys, and CVD-W. The principal property evaluated was the high-temperature creep strength. Five ion-pumped systems and two oil-pumped and liquid-nitrogen-trapped systems were placed back in service for this work. PWC-11 and Nb-1% Zr were tested in ion-pumped systems, and the molybdenum and tungsten materials were tested mostly in the oil-pumped systems.

Three heats of Nb-1% Zr were studied: one had been produced several years ago and the other two in the last year. The creep properties of all heats seemed equivalent. Although Nb-1% Zr was one of the refractory alloys most studied, it still lacks the type of data base desired for engineering applications. The two main shortcomings are (1) that the influence of pretest heat treatment has not been evaluated and (2) that the properties of welds are not known for the various welding processes that might be used in fabricating a complex system.

Much of the old data on Nb-1% Zr cannot be used with confidence. Testing techniques evolved slowly and samples were quite often contaminated with interstitials during testing.

A brief study[5] performed several years ago showed that the creep strength could be influenced by the pretest anneal. In the present study the samples were annealed at 1673 K for 1 h. Creep tests at 1250 K exhibited behavior in which the initial creep stage proceeded at a very low rate but then accelerated rapidly to failure. The duplex anneal was then evaluated under conditions in which the samples were given an anneal of 1 h at 1673 K and 2 h at 1477 K. This anneal gave slightly different creep behavior but did not eliminate the creep acceleration at 1250 K.

The shapes of the creep curves were evaluated through several correlations. At a test temperature of 1250 K, primary creep was a significant contributor to the first 1% strain, so there is some error in using the minimum creep rate to calculate the time to 1% strain. The time to 2% strain is about 1.5 times the time to 1% strain. The time to tertiary creep is generally slightly less than the time to 2% strain. At 1350°C, primary creep contributes a negligible amount to the first 1% strain. The

creep properties of welds in Nb-1% Zr were similar to those of the base metal; however, the weld metal per se was slightly enriched in interstitials during welding and appeared to be stronger. The adjacent base metal crept faster; consequently, the composite creep rate of weld samples was about equal to that of base metal.

The lot of PWC-11 studied was thought to have been extruded at some relatively low temperature that resulted in weaker creep strength. This point was shown in Fig. 12 where the curve developed by Horak[1] represents the stronger material and the other curve represents our weaker material. Our test material exhibited very little primary creep (Fig. 14), and time to 2% strain was about double the time to 1% strain. The time to tertiary creep was generally less than that to 2% strain.

The creep of the one PWC-11 weld tested was very similar in behavior to that of the Nb-1% Zr. The weld metal itself was stronger than the base metal, but the adjacent material (Fig. 30) crept faster so that the composite creep rate was about equivalent to that for base metal.

The creep results from most of the Mo-Re alloy testing were generally inconclusive because of the increments in temperature and stress performed on most specimens. It was apparent that the alloy containing 9% Re was weaker than the other two, and the alloys containing 11 and 13% Re had very similar creep strengths. Metallographic studies indicated that all three alloys were homogeneous and that the rhenium concentrations were very close to the nominal composition of each alloy.

The CVD-W creep studies are in initial stages. The samples for this study were annealed 1 h at 2000 K, and those from most previous tests were unannealed. As shown in Fig. 19, the present data appear about equal to those results represented by the Horak correlation.[1] However, the present results are inconclusive regarding the matter of relative strengths.

The correlations based on the sketchy test results available to date were used to construct the comparison of strengths shown in Fig. 37. The results for PWC-11 are weaker than those used by Horak in developing his correlation. However, the difference in slope between Nb-1% Zr and PWC-11 indicates that the strengths of the two alloys approach each other at very low stresses. The average line for Mo-11 and -13% Re was used in Fig. 37. The strength seems only slightly higher than that of PWC-11, and the slope matches that for Nb-1% Zr. CVD-W is much stronger than the other alloys.

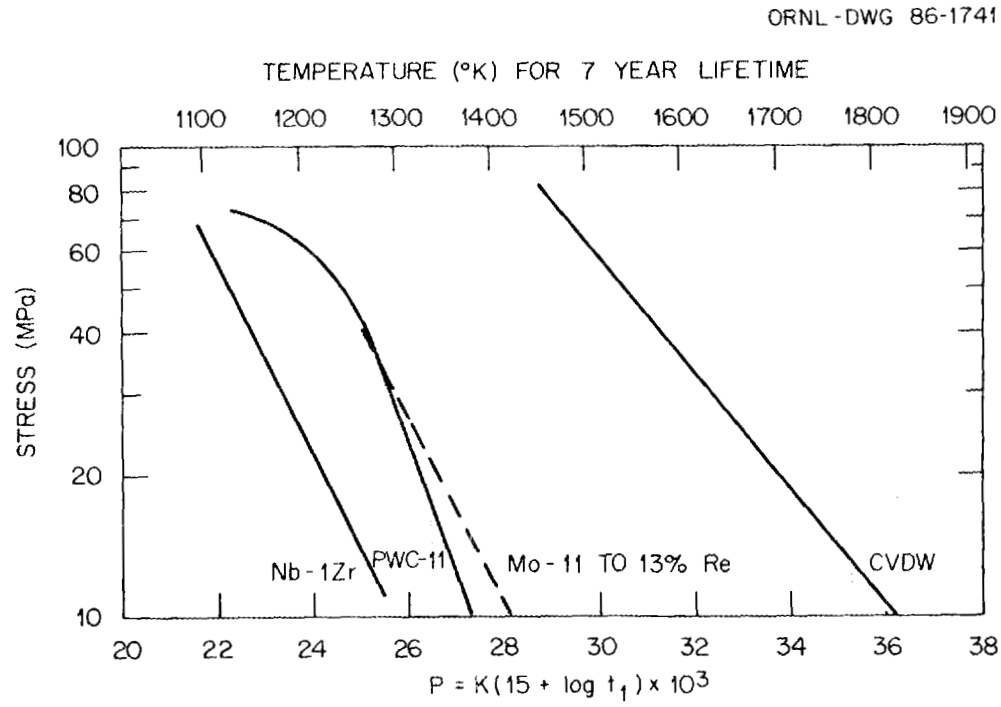


Fig. 37. Larson-Miller correlation for several alloys based on data from this study.

The question of a design criterion that does not damage the metal still remains. In many cases the creep strain limit is set by design (e.g., a close-fitting moving part cannot creep much before seizing). Several creep samples of Nb–1% Zr and PWC-11 were discontinued at about 3% strain, and there was no evidence of internal triple point cracks or voids. Thus these materials can probably be strained to levels of 3% or more before damage occurs. The Mo-Re alloys were not examined after testing. CVD-W has a high tendency to form cracks because of its very brittle large columnar grains. Since this material contains residual gases (F_2 or Cl_2), bubbles can form and grow under stress. This may in fact impose a stress limit as well as a strain limit on this material.

CONCLUSIONS

Suitable testing techniques were established for the very reactive refractory metals. Tests were run on three heats of Nb–1% Zr, and the creep strength of this material agreed well with the correlation developed

by Horak[1] for existing data. Some effort was made to optimize the properties through heat treatment, but this work is not complete. One lot of PWC-11 was tested, but its fabrication history involved a low-temperature extrusion. The correlation developed by Horak[1] was for material extruded at a high temperature, and our data are consistently weaker. A duplex pretest anneal improved the strength, but not to the level of the data summarized by Horak. Welds of Nb-1% Zr and PWC-11 behaved similarly. Chemical analyses show that the weld metal was contaminated slightly by interstitials, so this contamination strengthened the weld deposit. The material around the weld crept faster than the base metal, and the composite weldment crept at about the same rate as a base-metal sample. The welds were free of voids and had good ductility.

The Mo-Re alloy test results are very difficult to interpret because of the steps in temperature and stress that were built into the test matrix. The test results did indicate that the Mo-9Re alloy was the weakest, while Mo-11Re and Mo-13Re, about equal in strength, were stronger than Mo-9Re.

Techniques were re-established for preparing and testing CVD-W. Five tests have been run or are in progress. The test results are close to the correlation developed by Horak,[1] even though our test specimens were given a pretest anneal of 1 h at 2000 K. The specimens used in the Horak correlation were tested in the as-received condition.

ACKNOWLEDGMENTS

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1. J. A. Horak and R. W. Swindeman, "Analysis of Existing Creep Data for Refractory Alloys – Second Interim Progress Report," unpublished ORNL data, 1985.

2. R. L. Heestand, J. I. Federer, and C. F. Leitten, Jr., *Preparation and Evaluation of Vapor Deposited Tungsten*, ORNL-3662, August 1964.

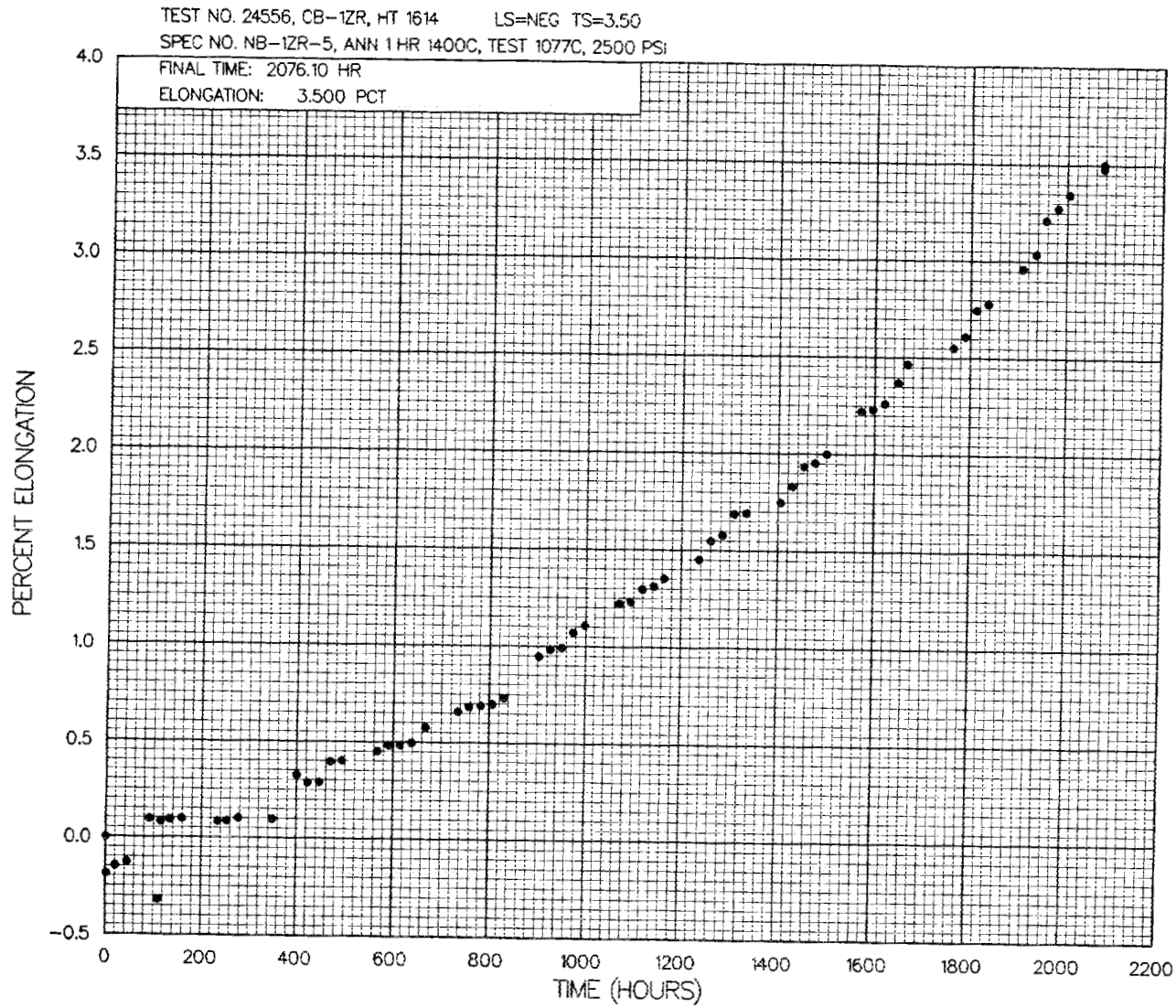
3. R. L. Orr, O. D. Sherby, and J. E. Dorn, "Correlation of Rupture Data for Metals at Elevated Temperatures," *Trans. ASM* **46**, 113–28 (1954).

4. J. A. Horak, unpublished ORNL data, 1985.

5. H. E. McCoy, "Creep Properties of the Nb–1% Zr Alloy," *J. Less-Common Met.* **8**, 20–35 (1965).

Appendix

CREEP CURVES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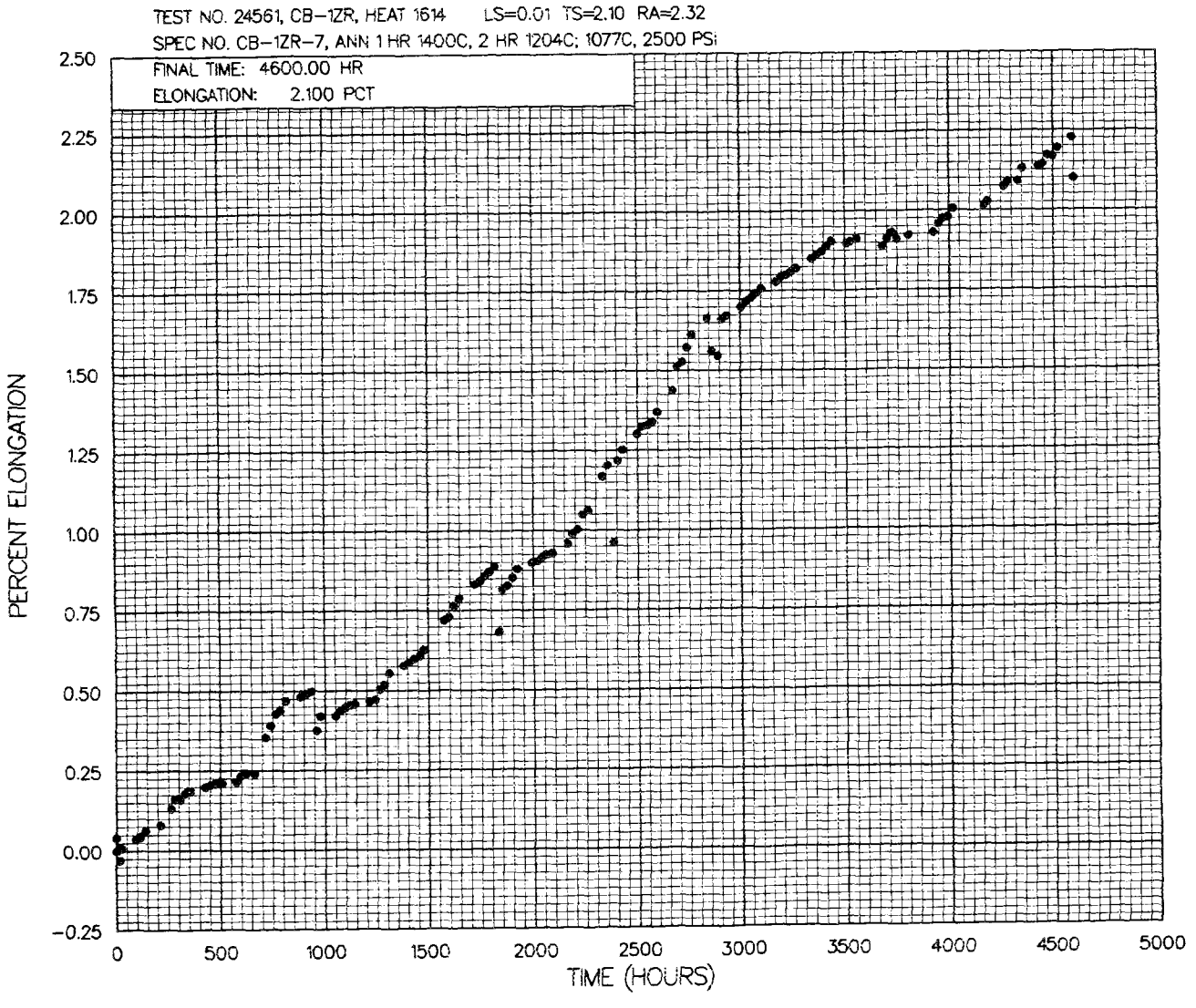
ELONGATION: 3.500 PCT
PERCENT ELONGATION
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
0
200
400
600
800
1000
1200
1400
1600
1800
2000
2200
TIME (HOURS)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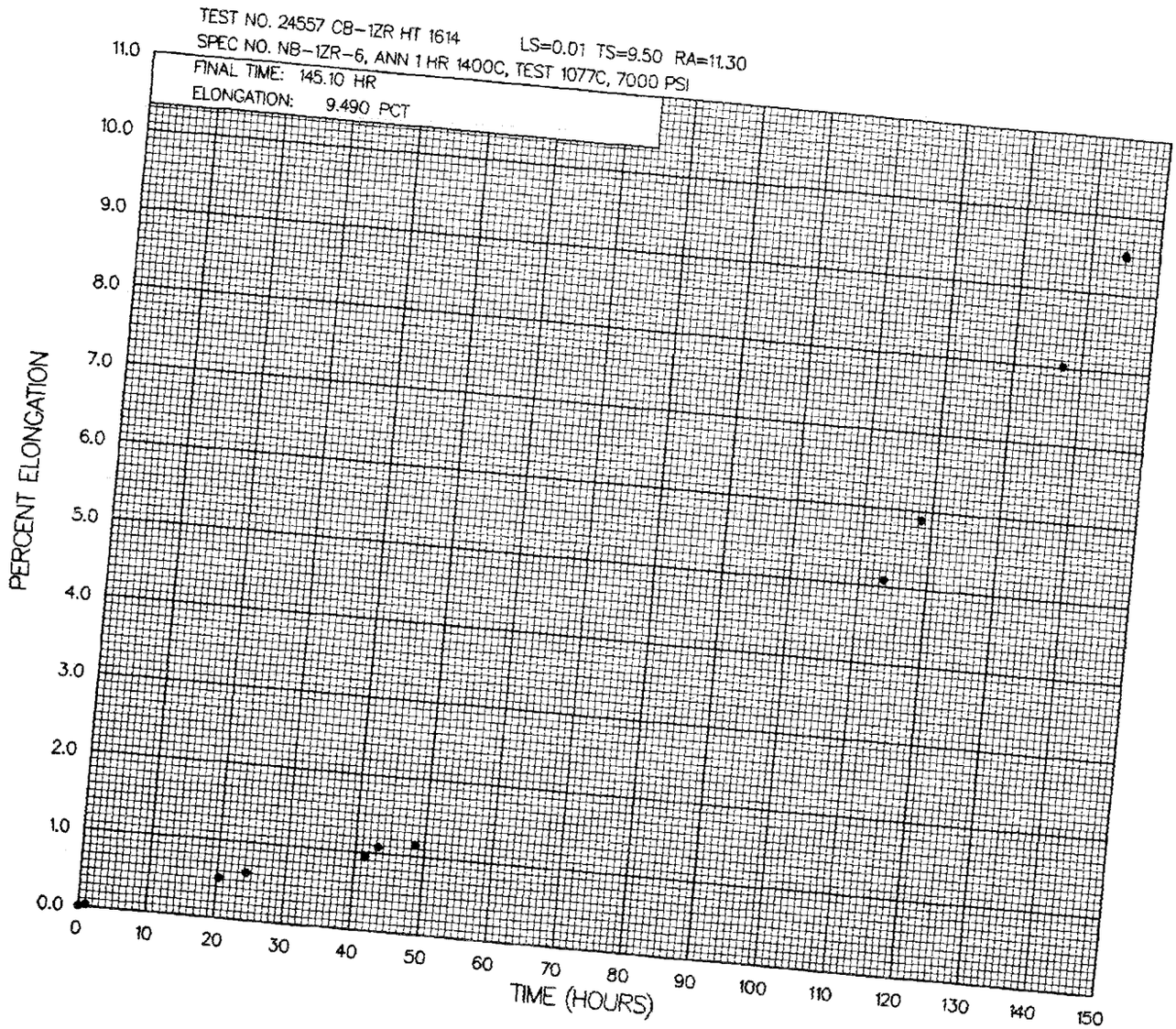
ELONGATION: 2.100 PCT
PERCENT ELONGATION
2.50
2.25
2.00
1.75
1.50
1.25
1.00
0.75
0.50
0.25
0.00
-0.25
0
500
1000
1500
2000
2500
3000
3500
4000
4500
5000
TIME (HOURS)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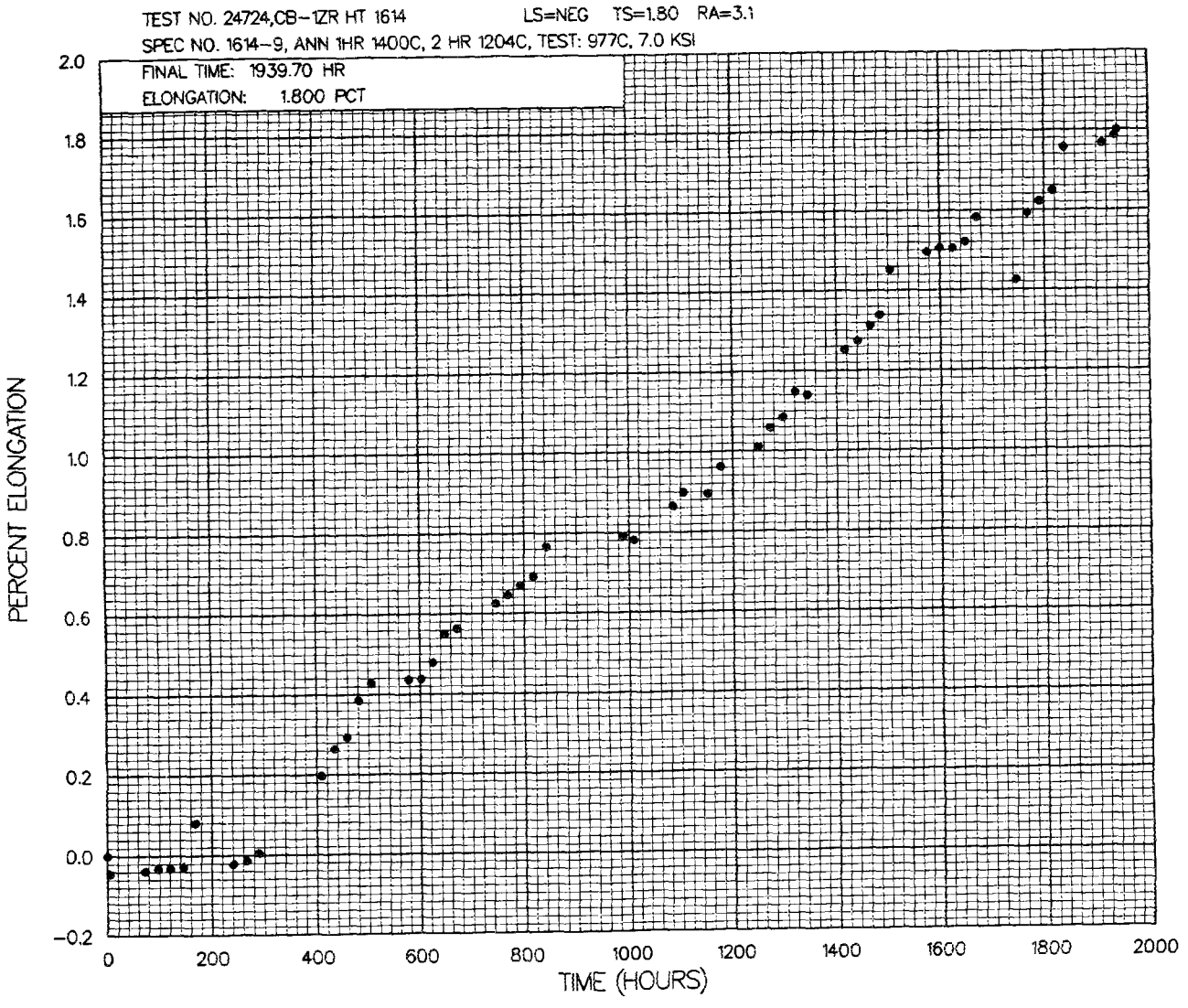
SPEC NO. NB-1ZR-6, ANN 1 HR 1400C, TEST 1077C, 7000 PSI
FINAL TIME: 145.10 HR
ELONGATION: 9.490 PCT
PERCENT ELONGATION
11.0
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
0
10
20
30
40
50
60
70
80
90
100
110
120
130
140
150
TIME (HOURS)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LS=NEG TS=1.80 RA=3.1
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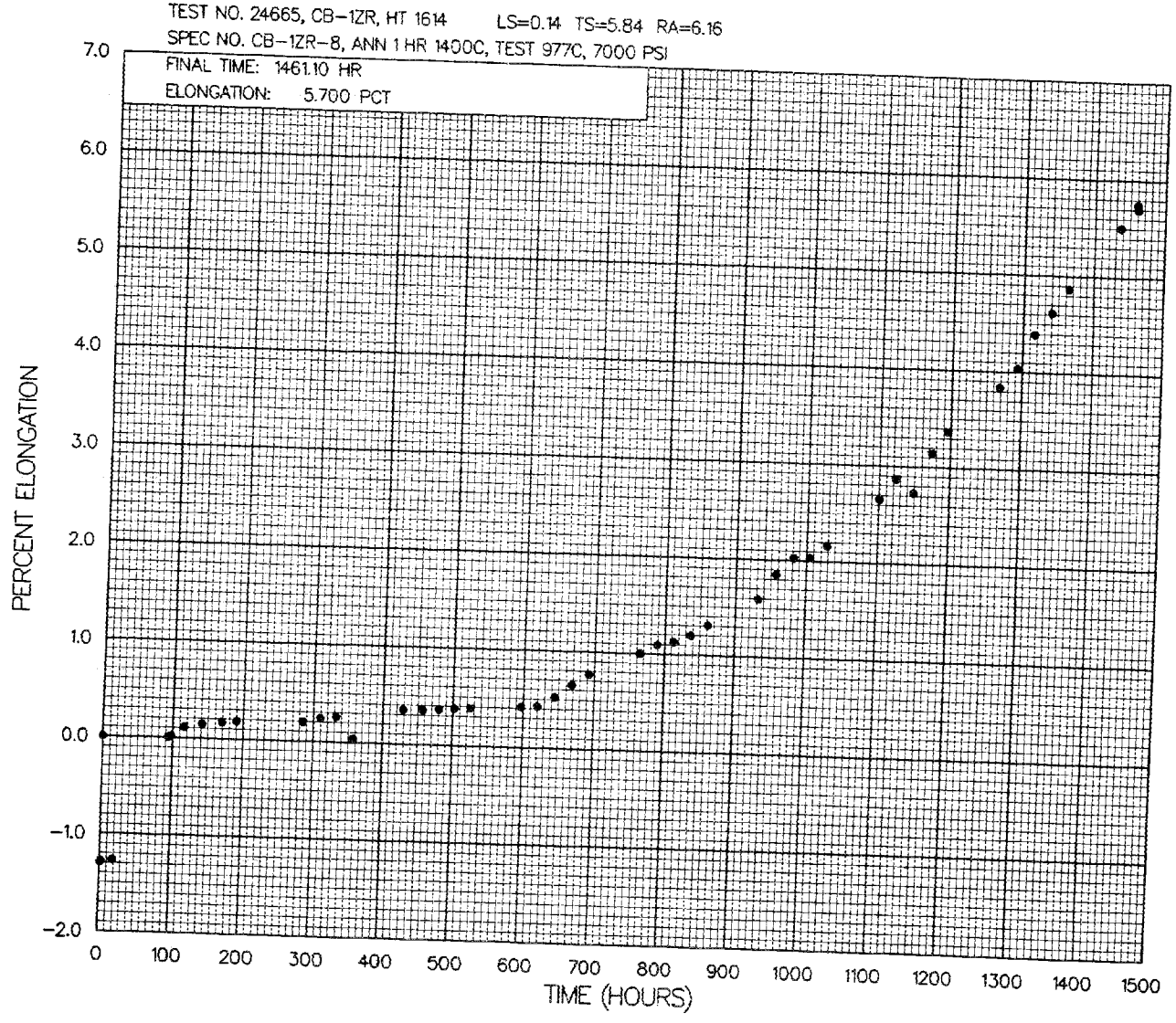
FINAL TIME: 1939.70 HR
ELONGATION: 1.800 PCT
PERCENT ELONGATION
2.0
1.8
1.6
1.4
1.2
1.0
0.8
0.6
0.4
0.2
0.0
-0.2
0
200
400
600
800
1000
1200
1400
1600
1800
2000
TIME (HOURS)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ELONGATION: 5.700 PCT
PERCENT ELONGATION
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6.0
5.0
4.0
3.0
2.0
1.0
0.0
-1.0
-2.0
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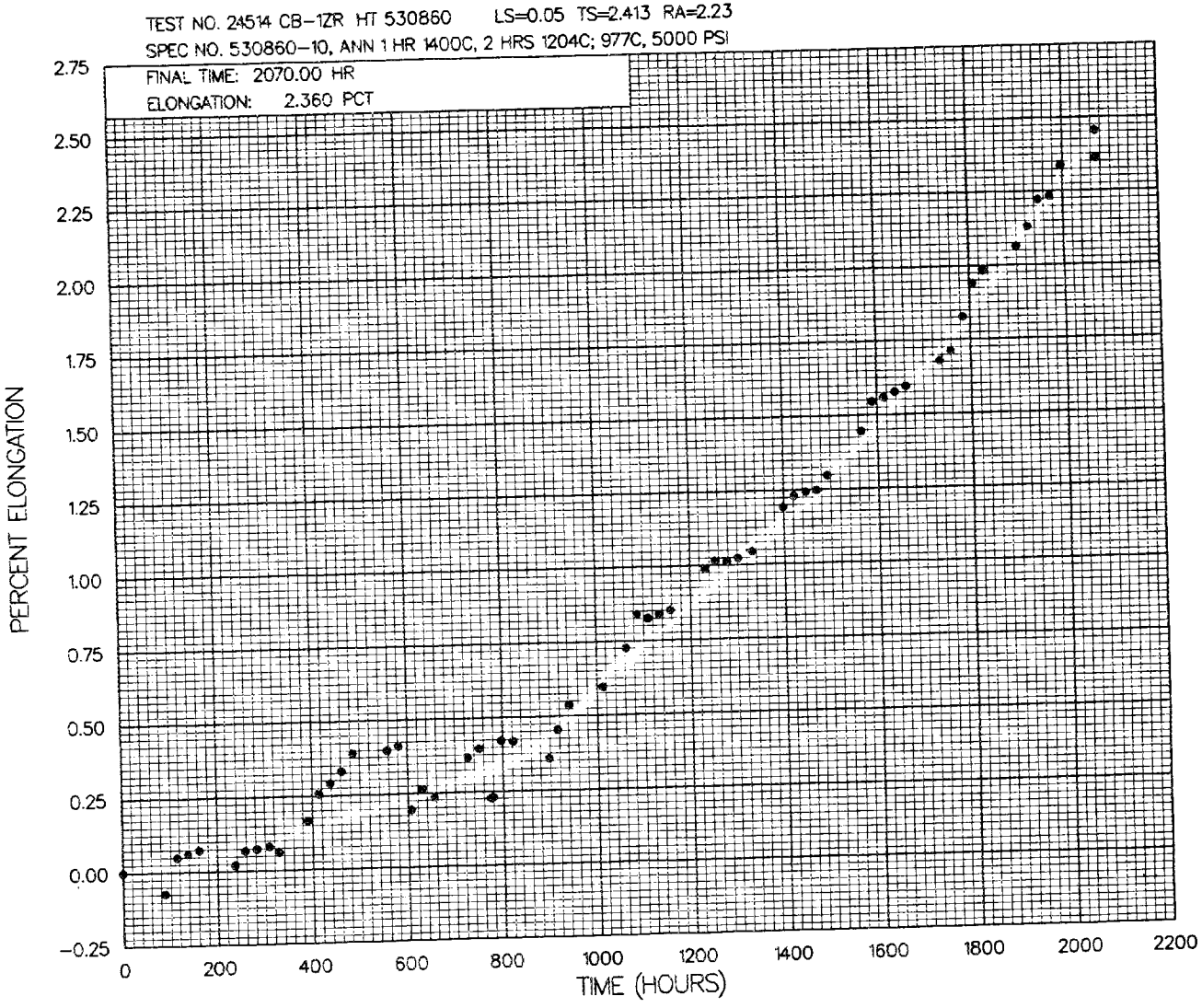
300
400
500
600
700
800
900
1000
1100
1200
1300
1400
1500
TIME (HOURS)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ELONGATION: 2.360 PCT
PERCENT ELONGATION
2.75
2.50
2.25
2.00
1.75
1.50
1.25
1.00
0.75
0.50
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400
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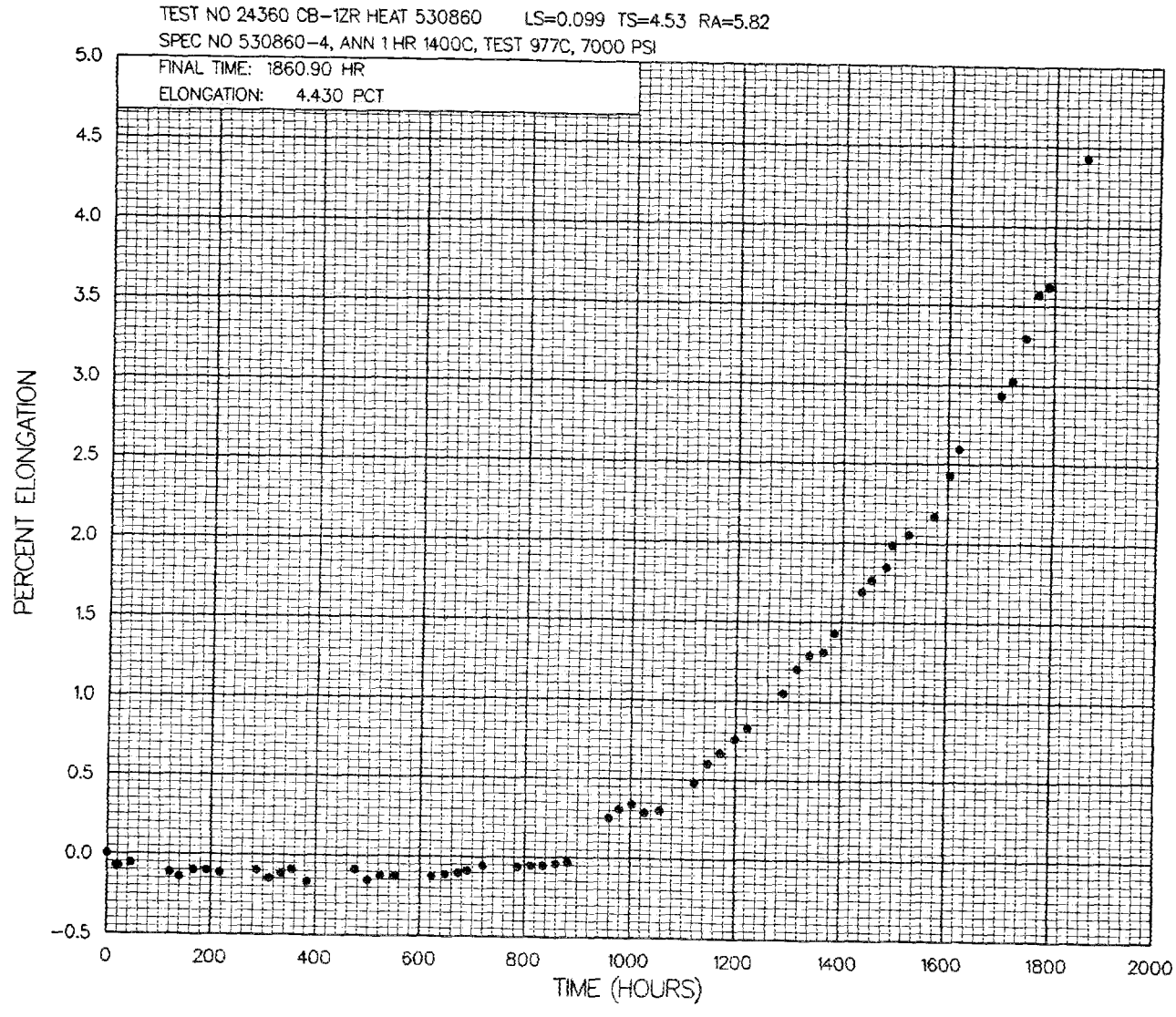
800
1000
1200
1400
1600
1800
2000
2200
TIME (HOURS)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PERCENT ELONGATION
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
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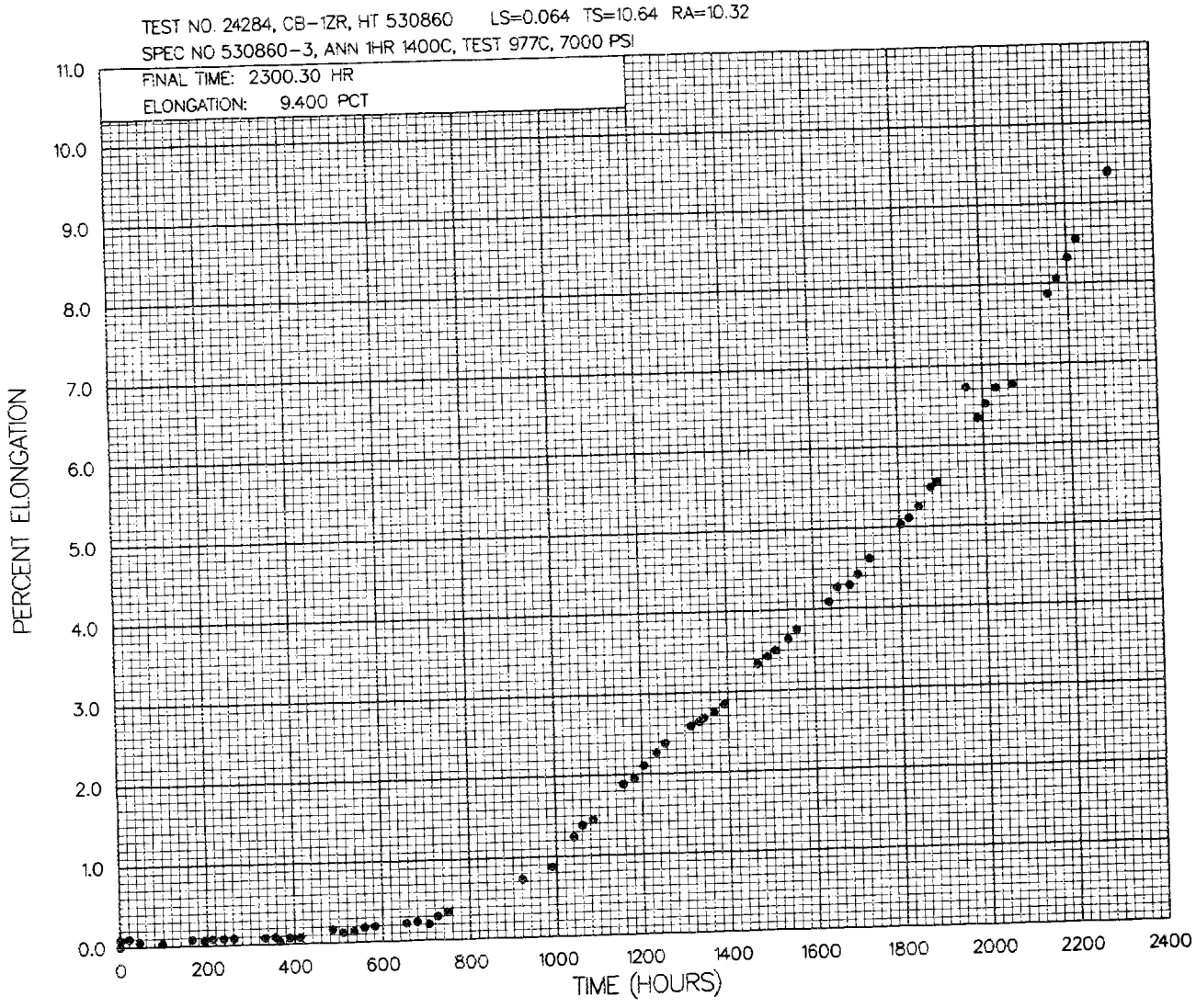
200
400
600
800
1000
1200
1400
1600
1800
2000
TIME (HOURS)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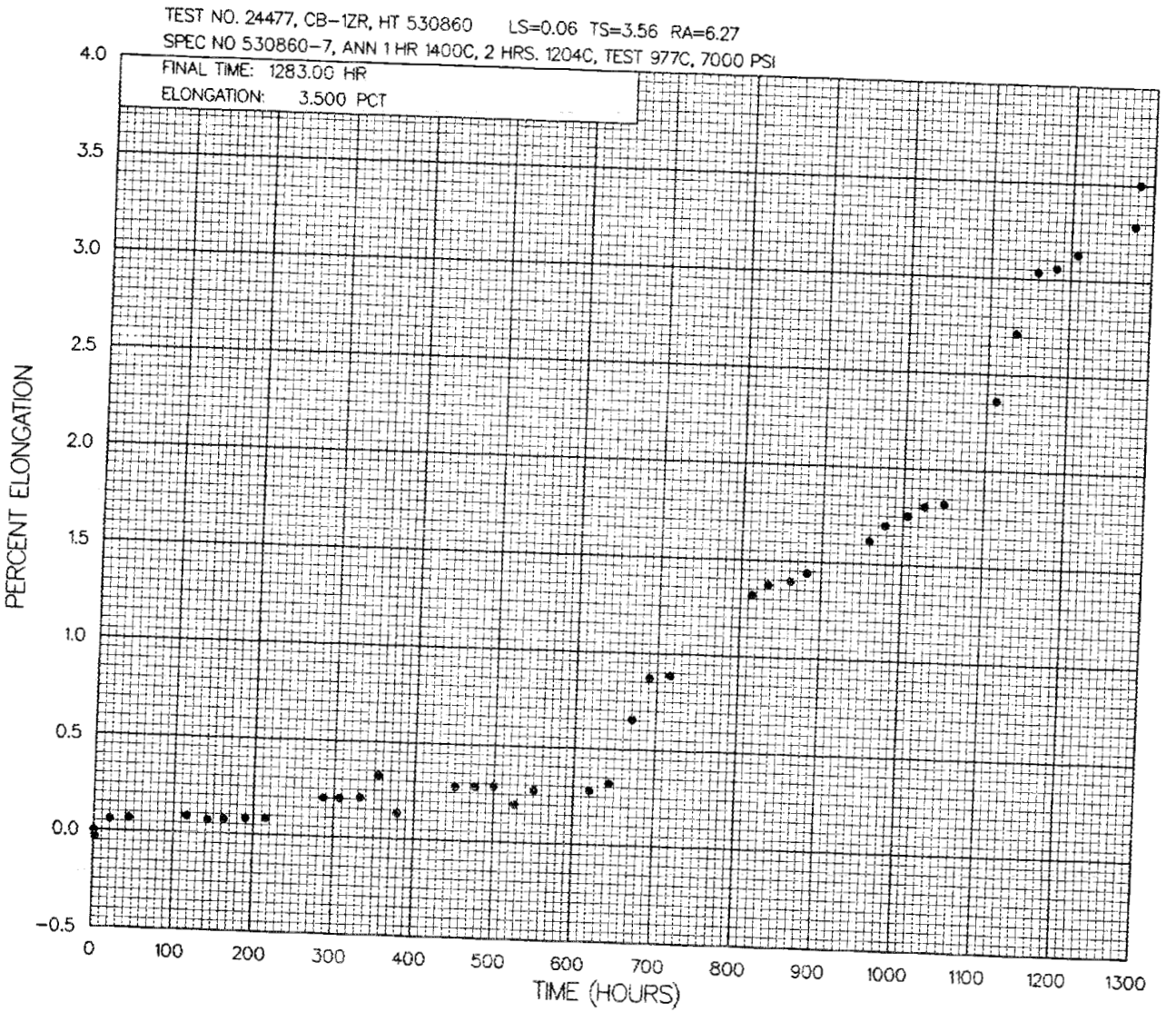
PERCENT ELONGATION
11.0
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
0
200
400
600
800
1000
1200
1400
1600
1800
2000
2200
2400
TIME (HOURS)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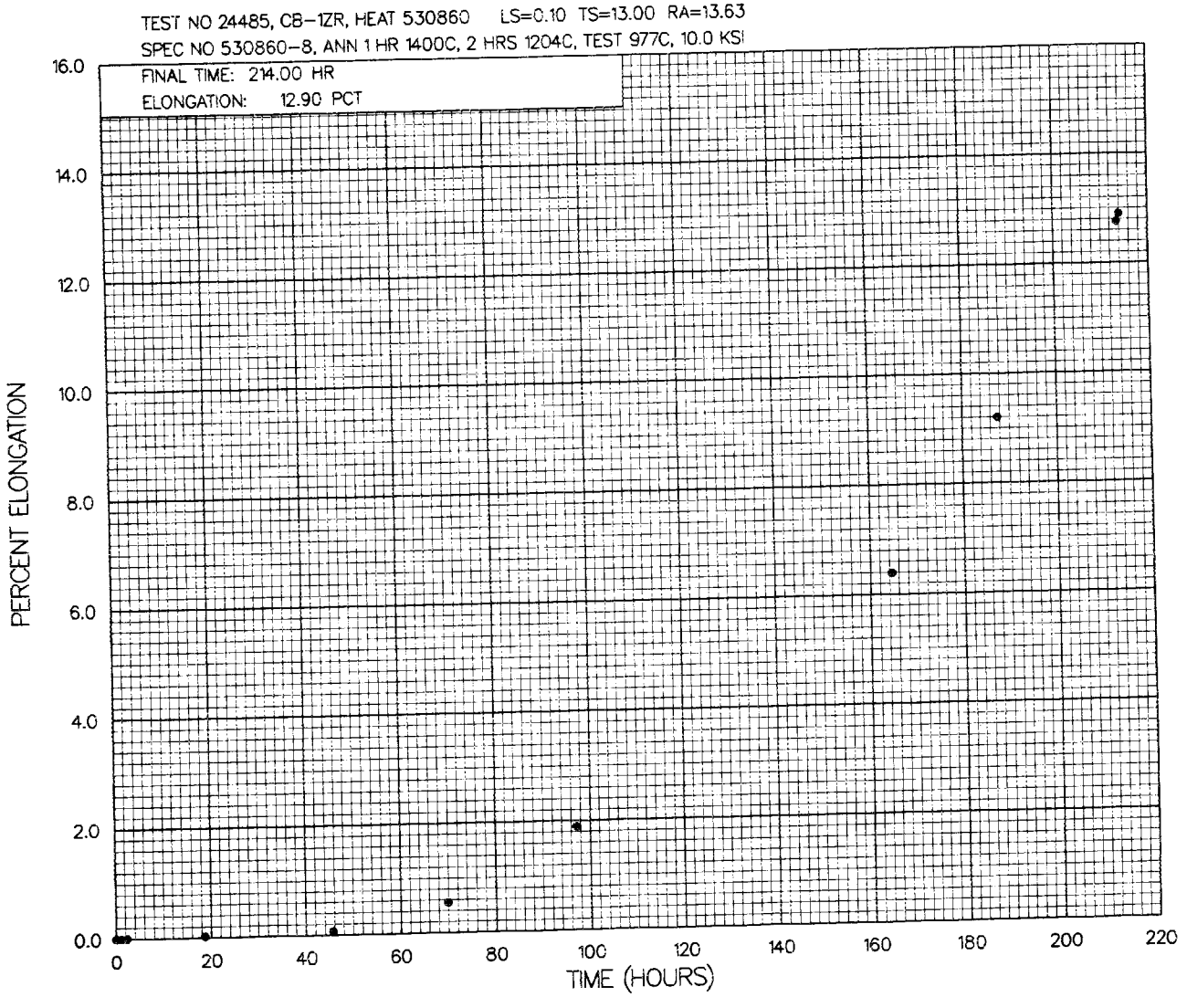
ELONGATION: 3.500 PCT
PERCENT ELONGATION
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
0
100
200
300
400
500
600
700
800
900
1000
1100
1200
1300
TIME (HOURS)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ELONGATION: 12.90 PCT
PERCENT ELONGATION
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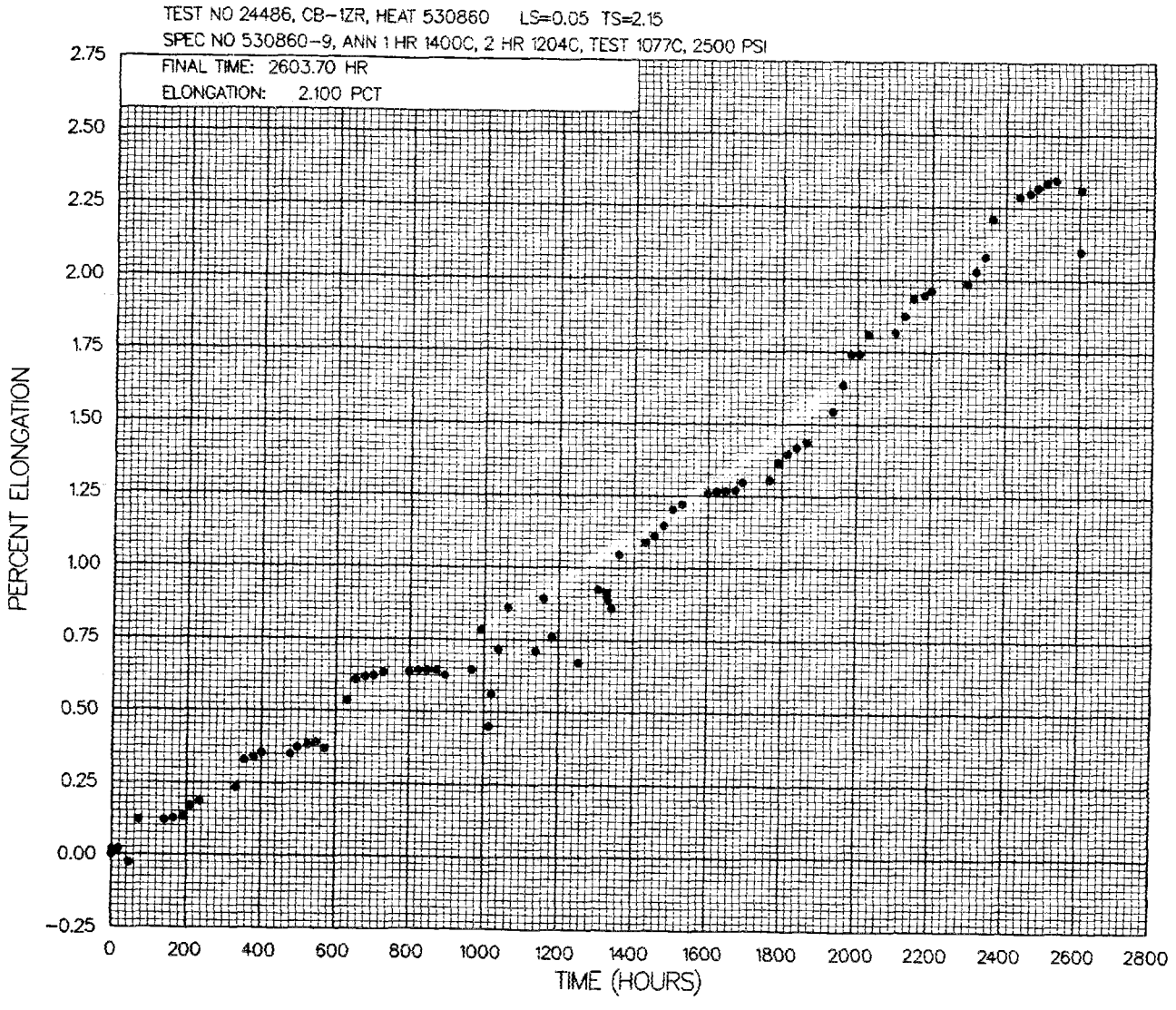
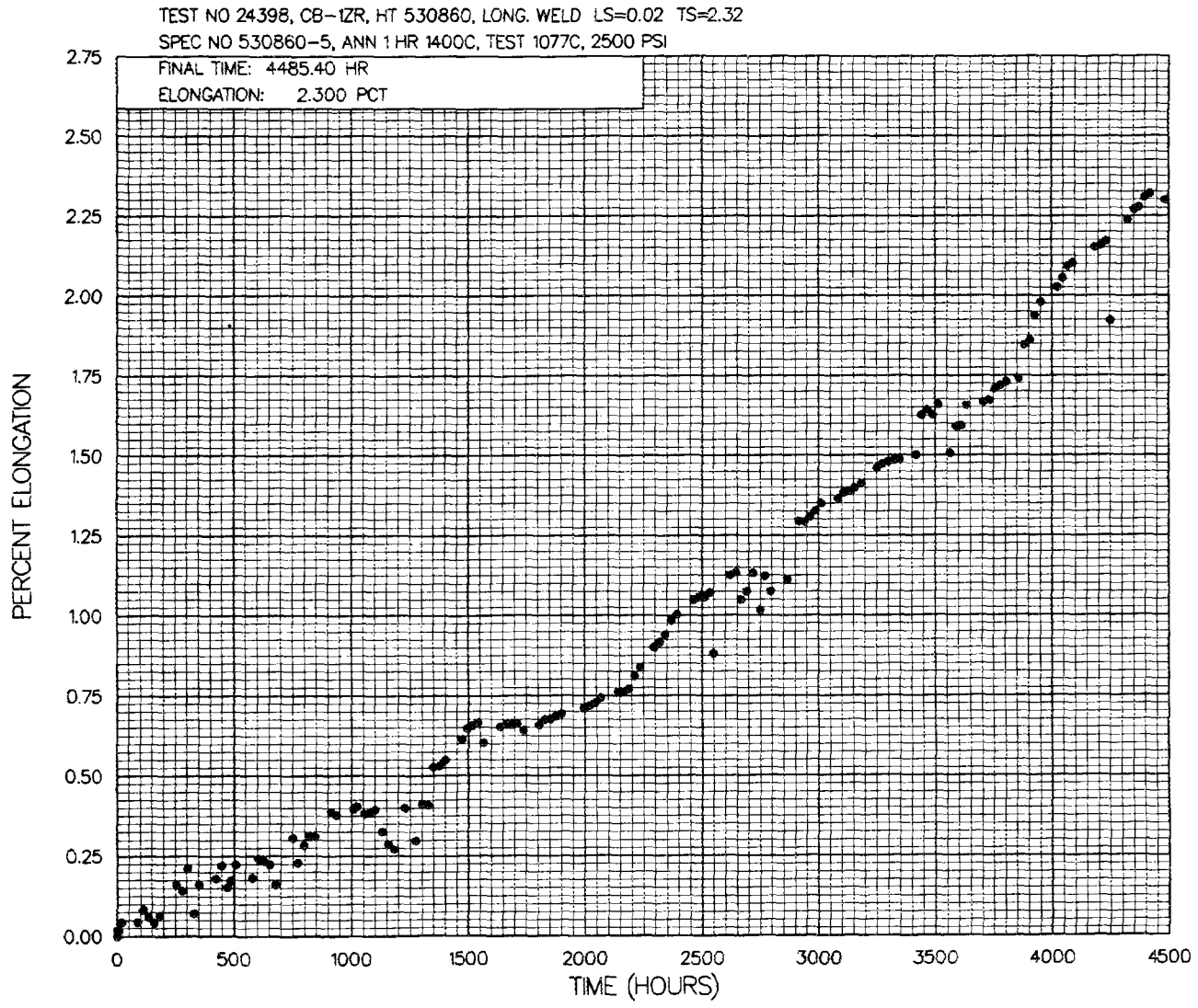
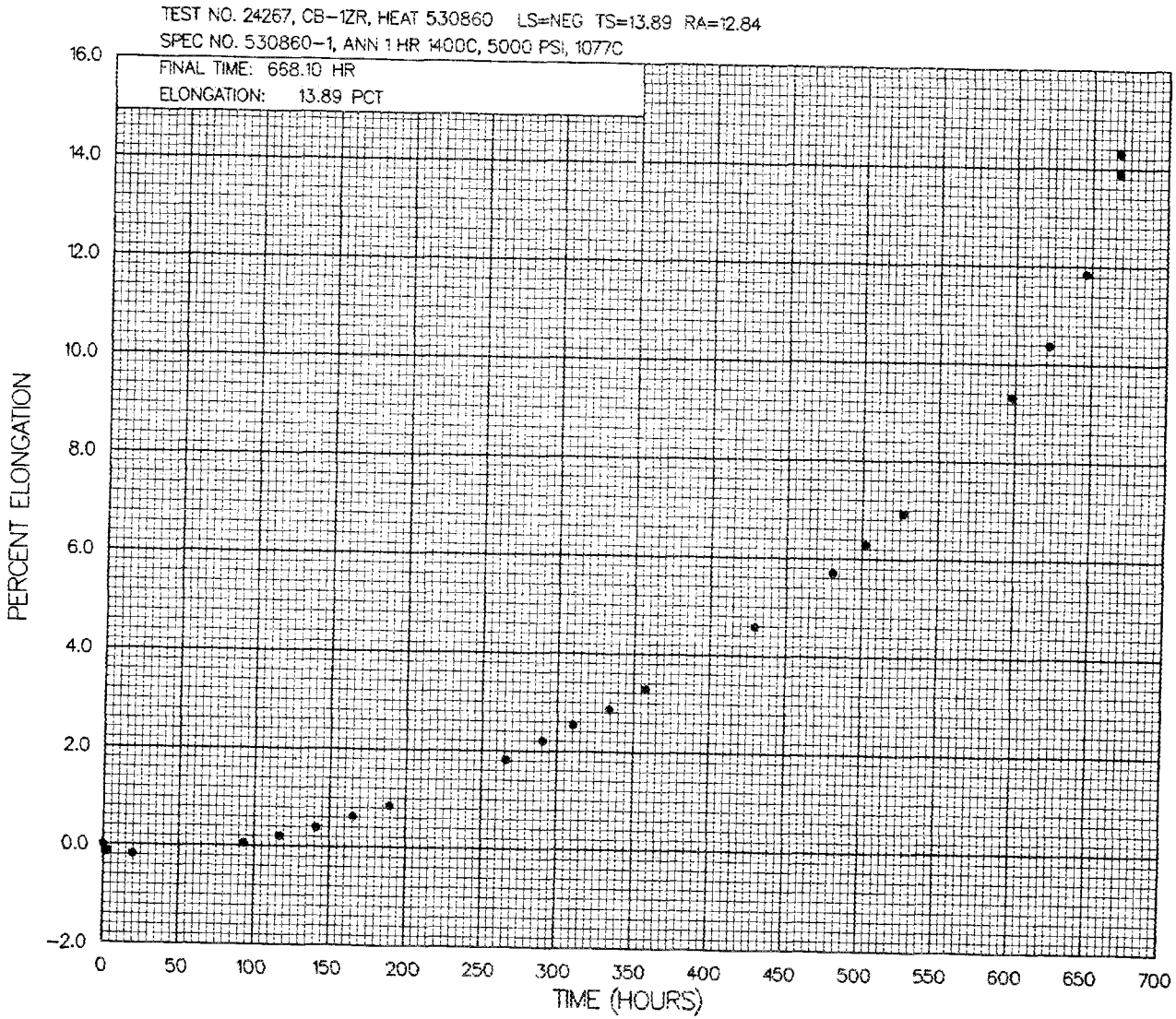
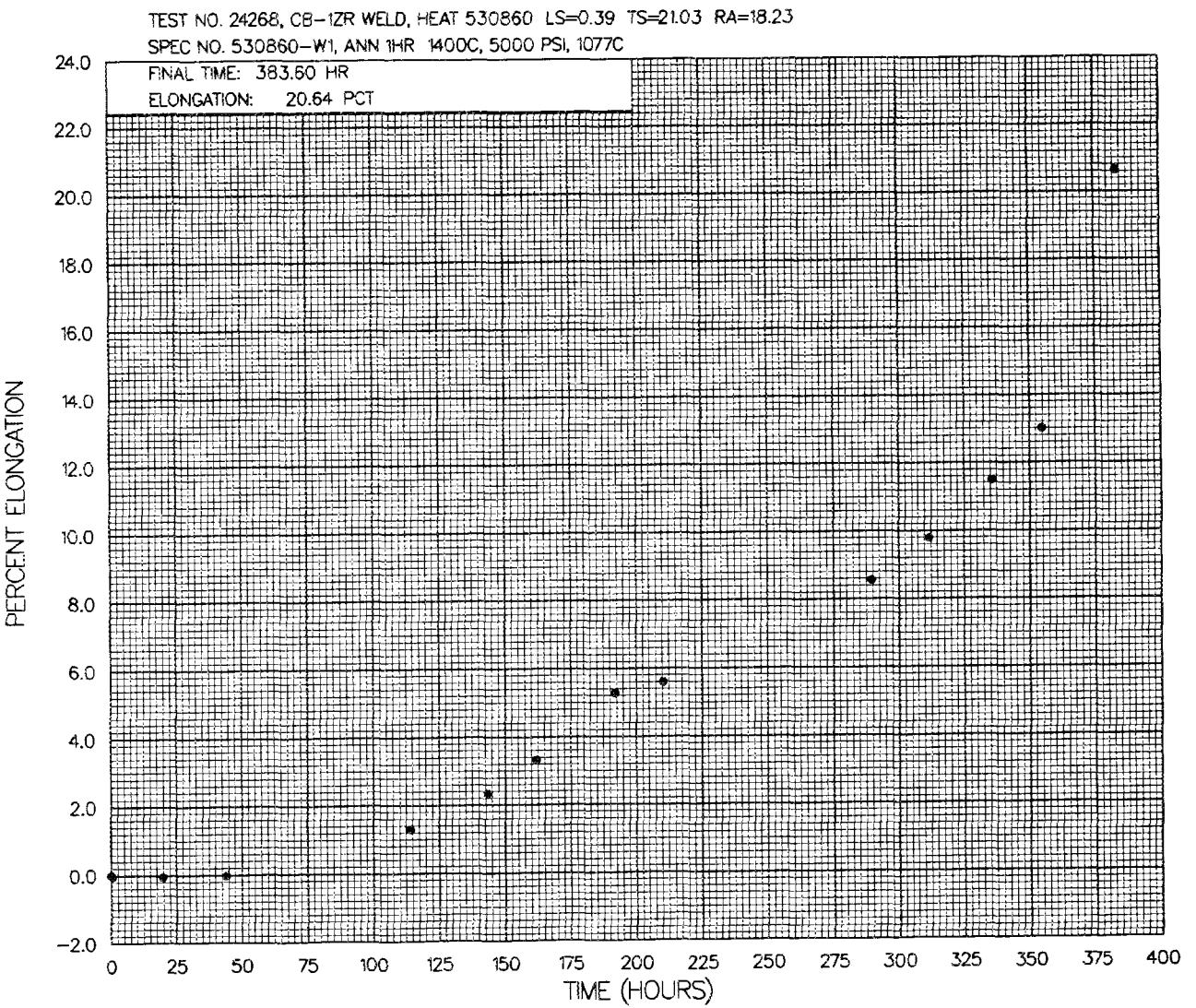
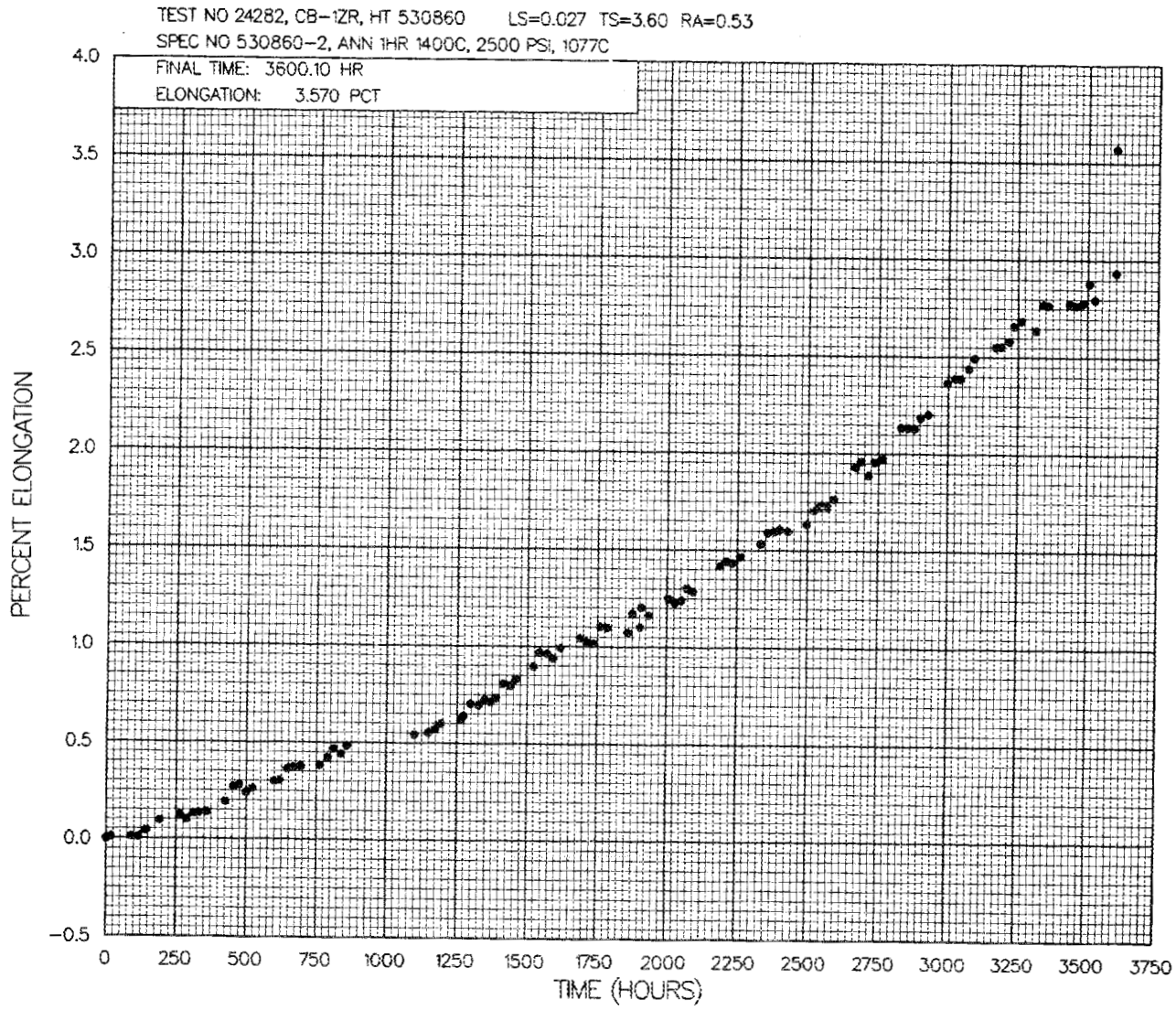
14.0
12.0
10.0
8.0
6.0
4.0
2.0
0.0
0
20
40
60
80
100
120
140
160
180
200
220
TIME (HOURS)

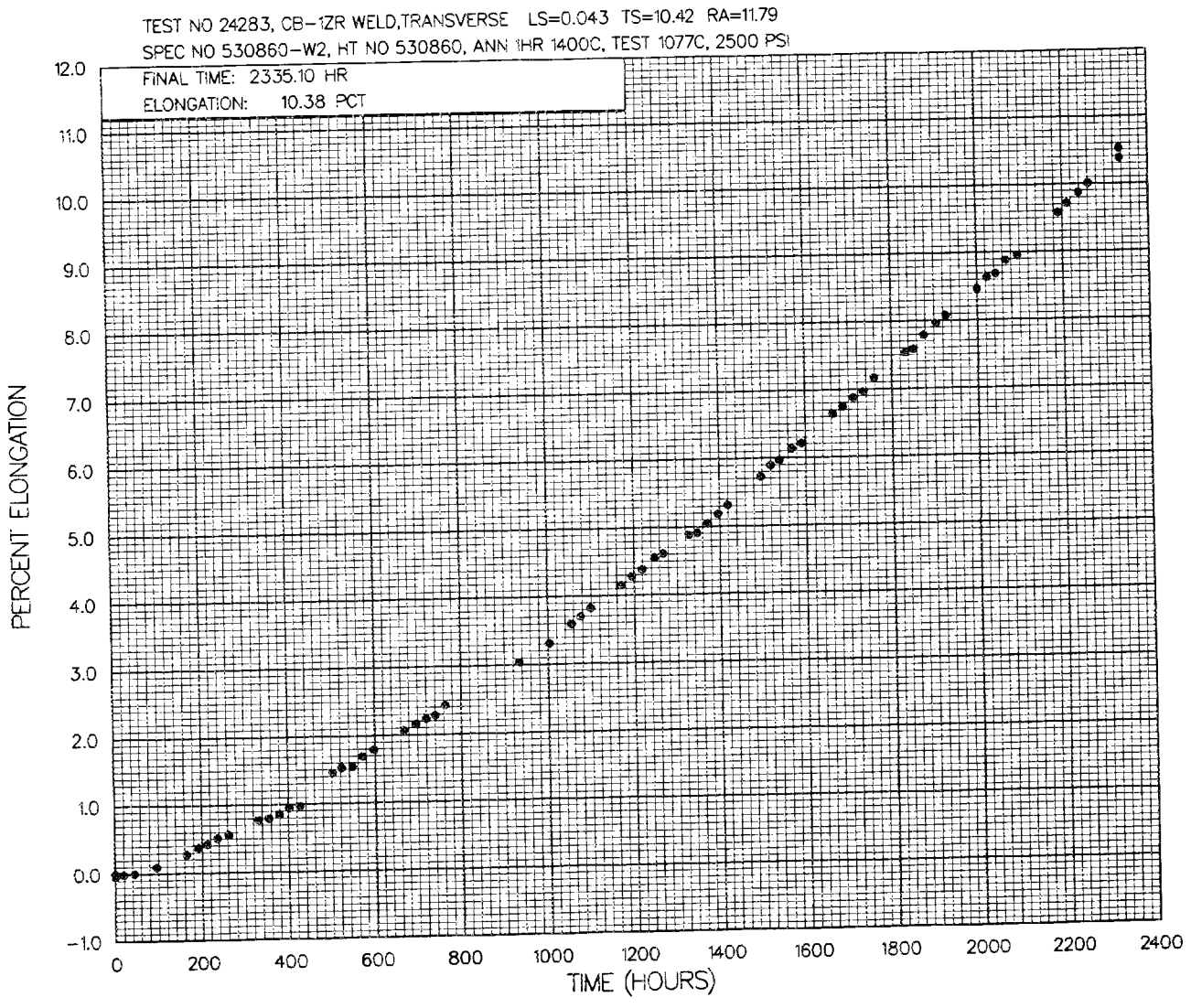

TEST NO 24486, CB-1ZR, HEAT 530860 LS=0.05 TS=2.15
SPEC NO 530860-9, ANN 1 HR 1400C, 2 HR 1204C, TEST 1077C, 2500 PSI
FINAL TIME: 2603.70 HR
ELONGATION: 2.100 PCT
PERCENT ELONGATION
2.75
2.50
2.25
2.00
1.75
1.50
1.25
1.00
0.75
0.50
0.25
0.00
-0.25
0
200
400
600
800
1000
1200
1400
1600
1800
2000
2200
2400
2600
2800
TIME (HOURS)

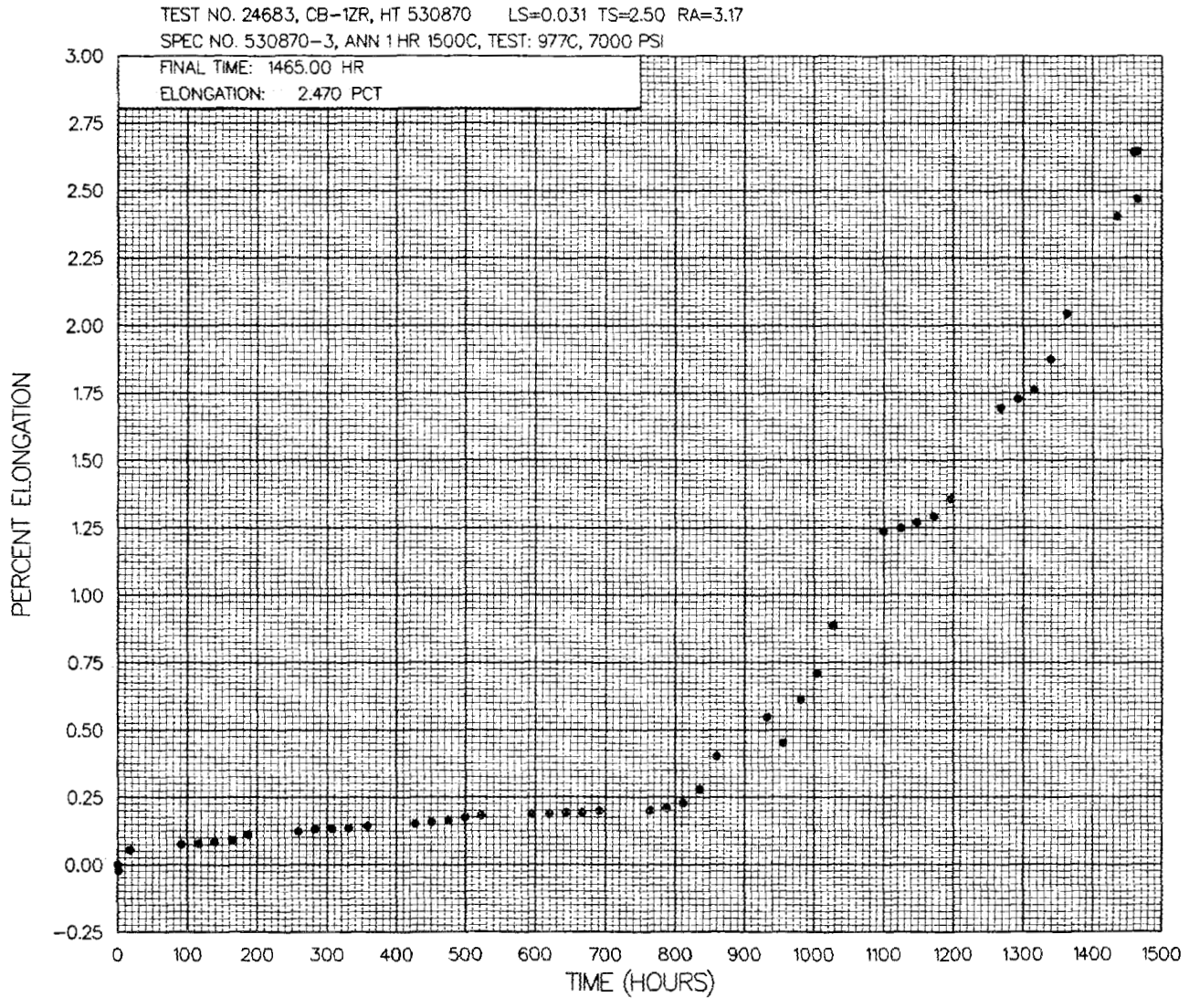

TEST NO 24398, CB-1ZR, HT 530860, LONG. WELD LS=0.02 TS=2.32
SPEC NO 530860-5, ANN 1 HR 1400C, TEST 1077C, 2500 PSI
FINAL TIME: 4485.40 HR
ELONGATION: 2.300 PCT
PERCENT ELONGATION
2.75
2.50
2.25
2.00
1.75
1.50
1.25
1.00
0.75
0.50
0.25
0.00
0
500
1000
1500
2000
2500
3000
3500
4000
4500
TIME (HOURS)

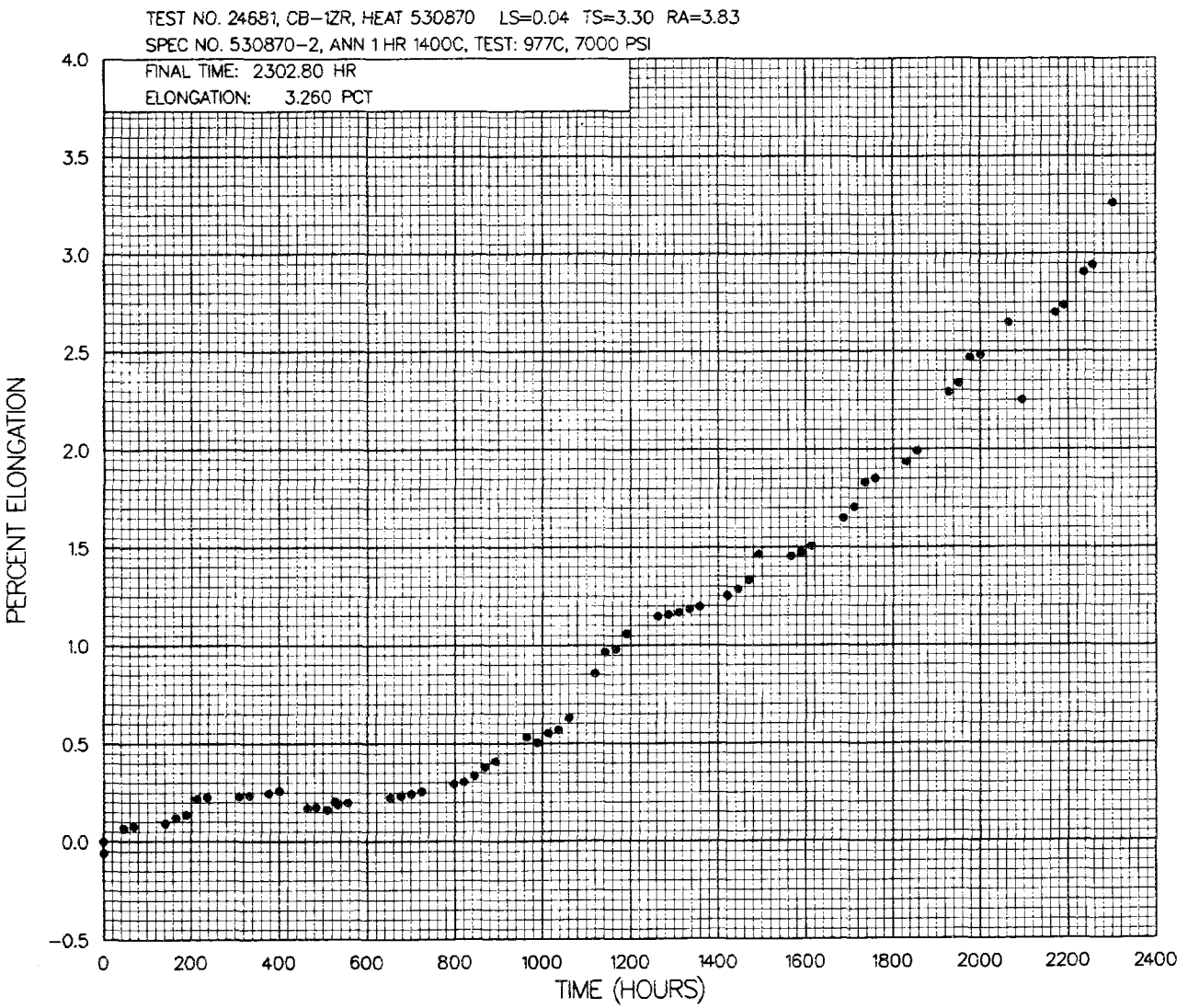

TEST NO. 24267, CB-1ZR, HEAT 530860 LS=NEG TS=13.89 RA=12.84
SPEC NO. 530860-1, ANN 1 HR 1400C, 5000 PSI, 1077C
FINAL TIME: 668.10 HR
ELONGATION: 13.89 PCT
PERCENT ELONGATION
16.0
14.0
12.0
10.0
8.0
6.0
4.0
2.0
0.0
-2.0
0
50
100
150
200
250
300
350
400
450
500
550
600
650
700
TIME (HOURS)

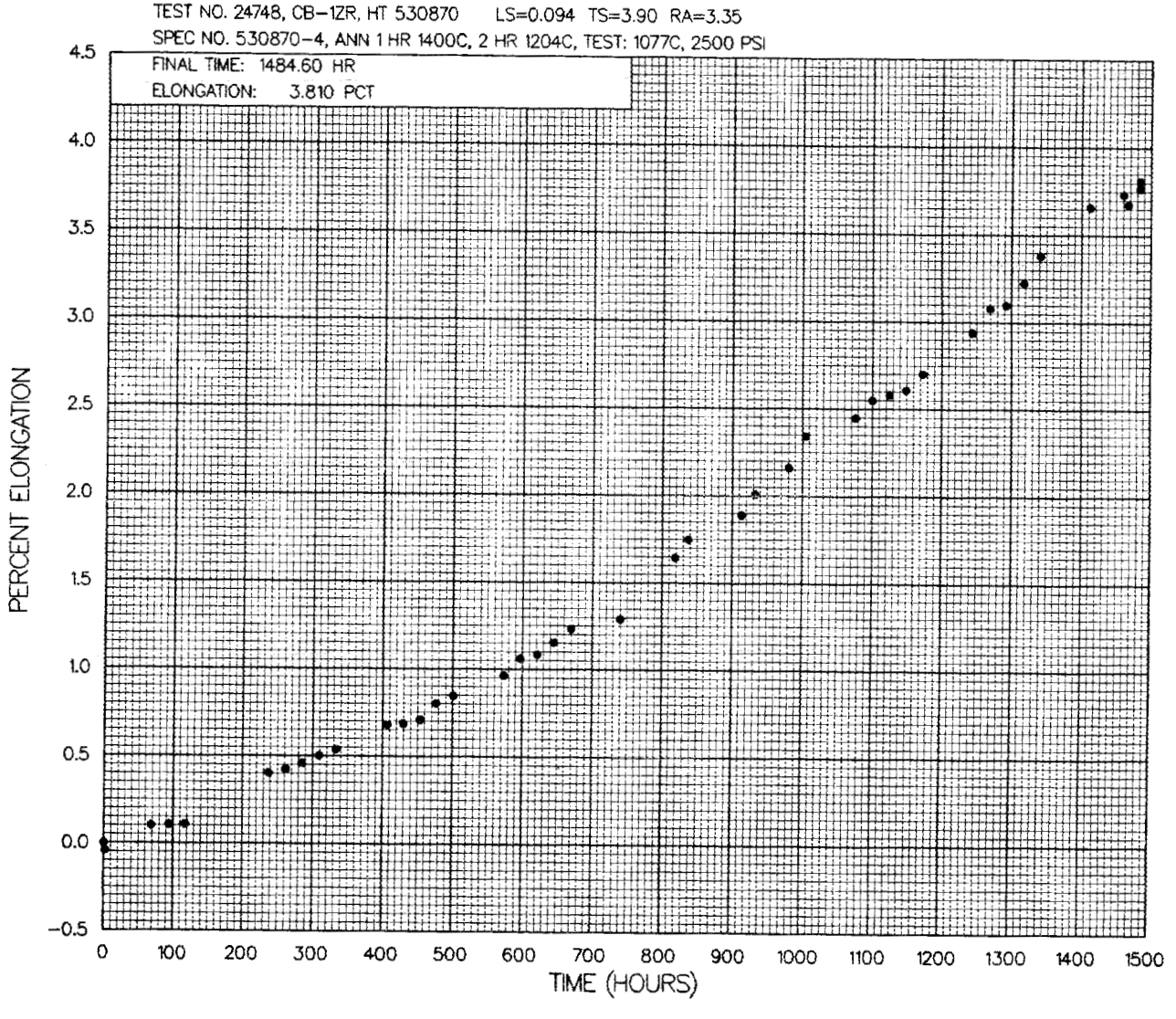

TEST NO. 24268, CB-1ZR WELD, HEAT 530860 LS=0.39 TS=21.03 RA=18.23
SPEC NO. 530860-W1, ANN 1HR 1400C, 5000 PSI, 1077C
FINAL TIME: 383.60 HR
ELONGATION: 20.64 PCT
PERCENT ELONGATION
24.0
22.0
20.0
18.0
16.0
14.0
12.0
10.0
8.0
6.0
4.0
2.0
0.0
-2.0
0
25
50
75
100
125
150
175
200
225
250
275
300
325
350
375
400
TIME (HOURS)

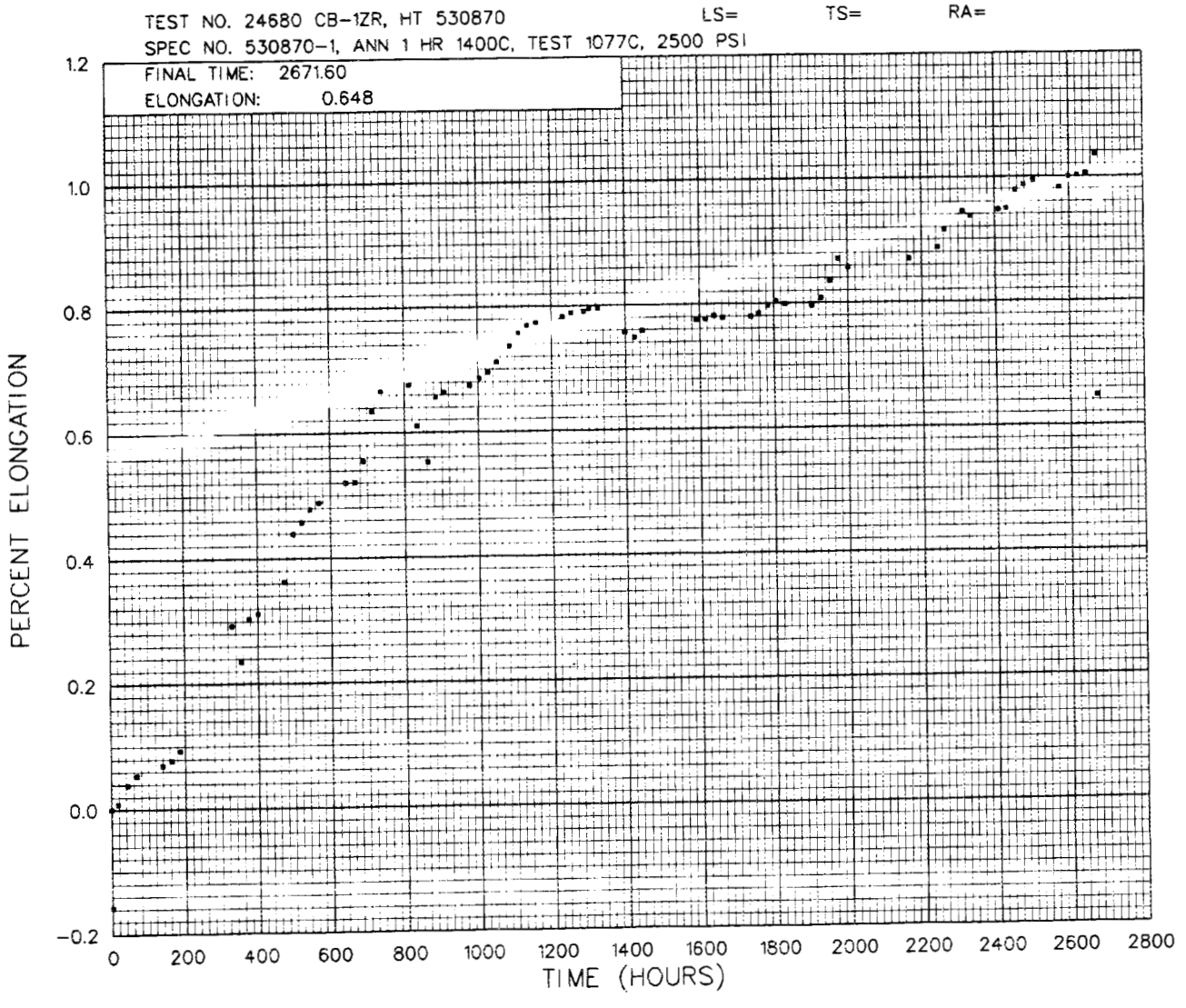

TEST NO 24282, CB-1ZR, HT 530860 LS=0.027 TS=3.60 RA=0.53
SPEC NO 530860-2, ANN 1HR 1400C, 2500 PSI, 1077C
FINAL TIME: 3600.10 HR
ELONGATION: 3.570 PCT
PERCENT ELONGATION
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
0
250
500
750
1000
1250
1500
1750
2000
2250
2500
2750
3000
3250
3500
3750
TIME (HOURS)

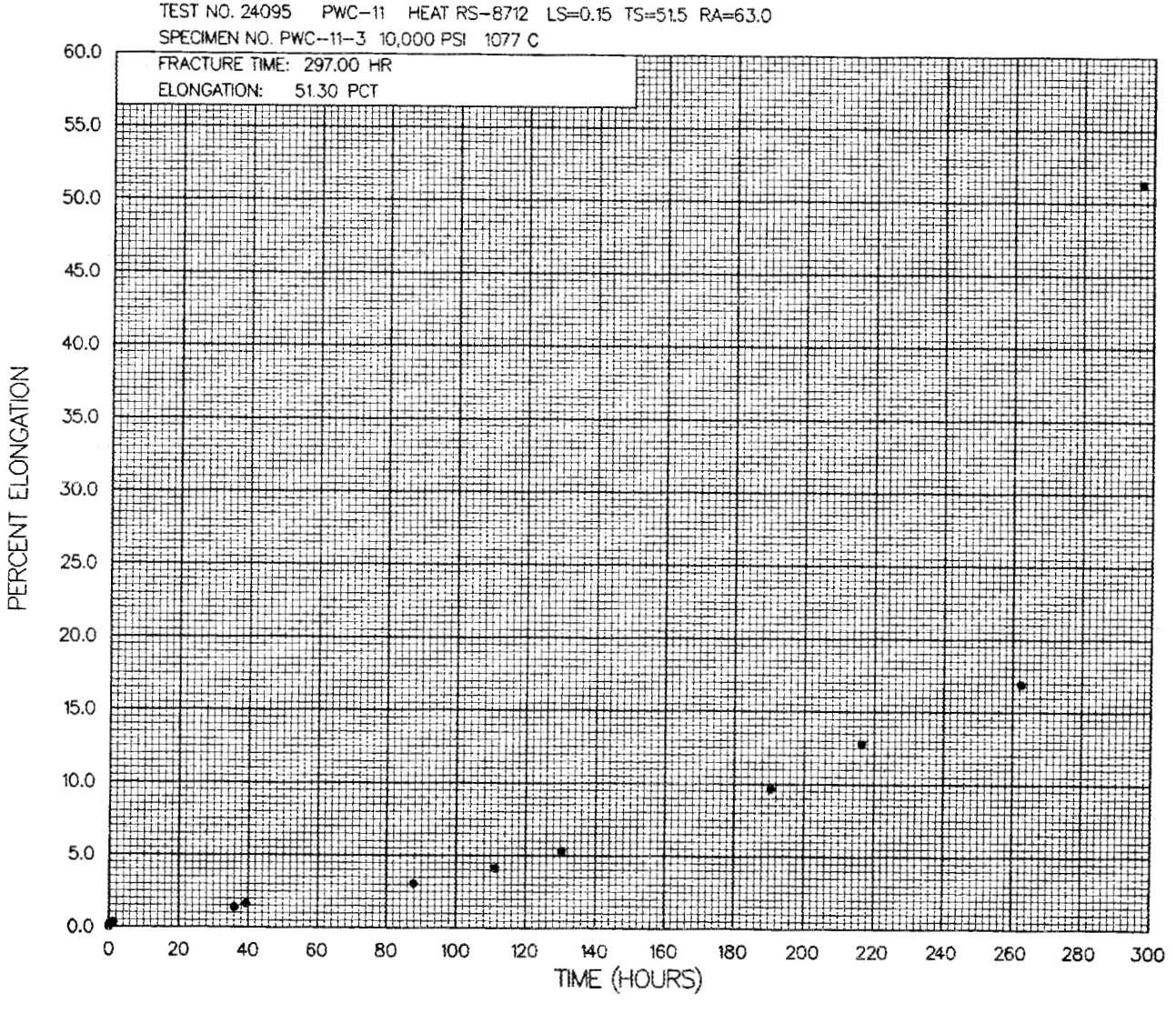

TEST NO 24283, CB-1ZR WELD,TRANSVERSE LS=0.043 TS=10.42 RA=11.79
SPEC NO 530860-W2, HT NO 530860, ANN 1HR 1400C, TEST 1077C, 2500 PSI
FINAL TIME: 2335.10 HR
ELONGATION: 10.38 PCT
PERCENT ELONGATION
12.0
11.0
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
-1.0
0
200
400
600
800
1000
1200
1400
1600
1800
2000
2200
2400
TIME (HOURS)

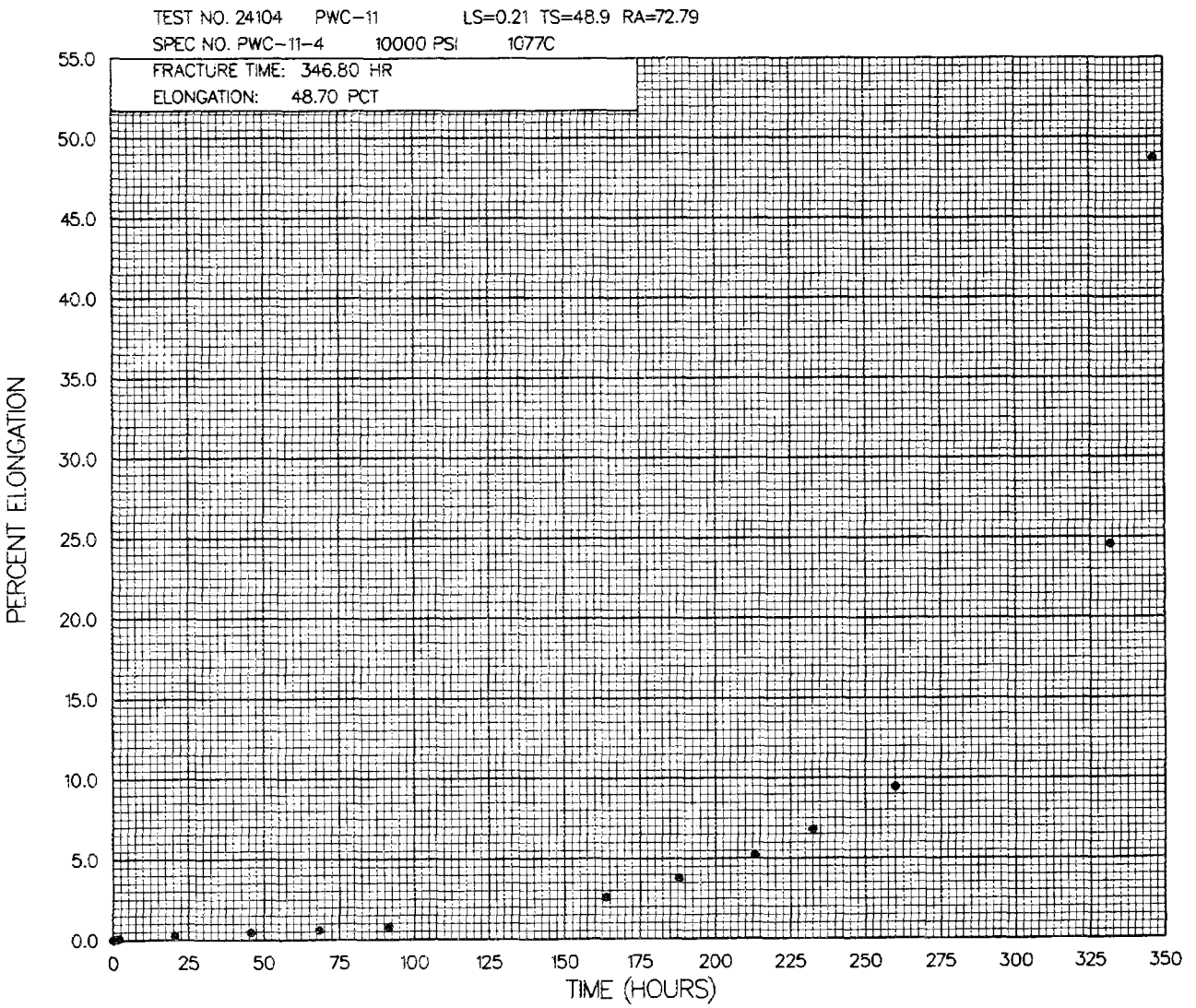

TEST NO. 24683, CB-1ZR, HT 530870 LS=0.031 TS=2.50 RA=3.17
SPEC NO. 530870-3, ANN 1 HR 1500C, TEST: 977C, 7000 PSI
FINAL TIME: 1465.00 HR
ELONGATION: 2.470 PCT
PERCENT ELONGATION
3.00
2.75
2.50
2.25
2.00
1.75
1.50
1.25
1.00
0.75
0.50
0.25
0.00
-0.25
0
100
200
300
400
500
600
700
800
900
1000
1100
1200
1300
1400
1500
TIME (HOURS)

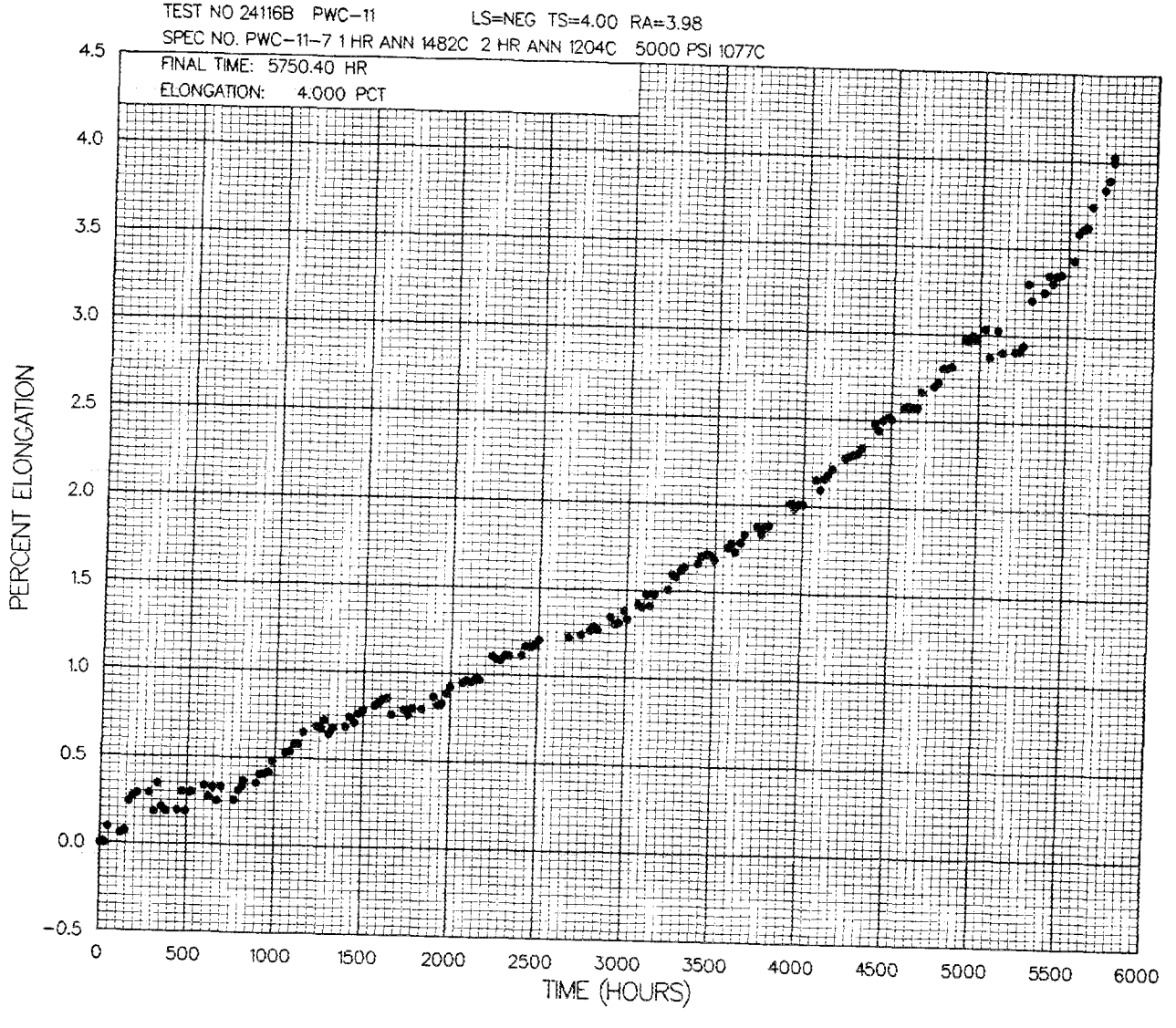

TEST NO. 24681, CB-1ZR, HEAT 530870 LS=0.04 TS=3.30 RA=3.83
SPEC NO. 530870-2, ANN 1 HR 1400C, TEST: 977C, 7000 PSI
FINAL TIME: 2302.80 HR
ELONGATION: 3.260 PCT
PERCENT ELONGATION
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
0
200
400
600
800
1000
1200
1400
1600
1800
2000
2200
2400
TIME (HOURS)

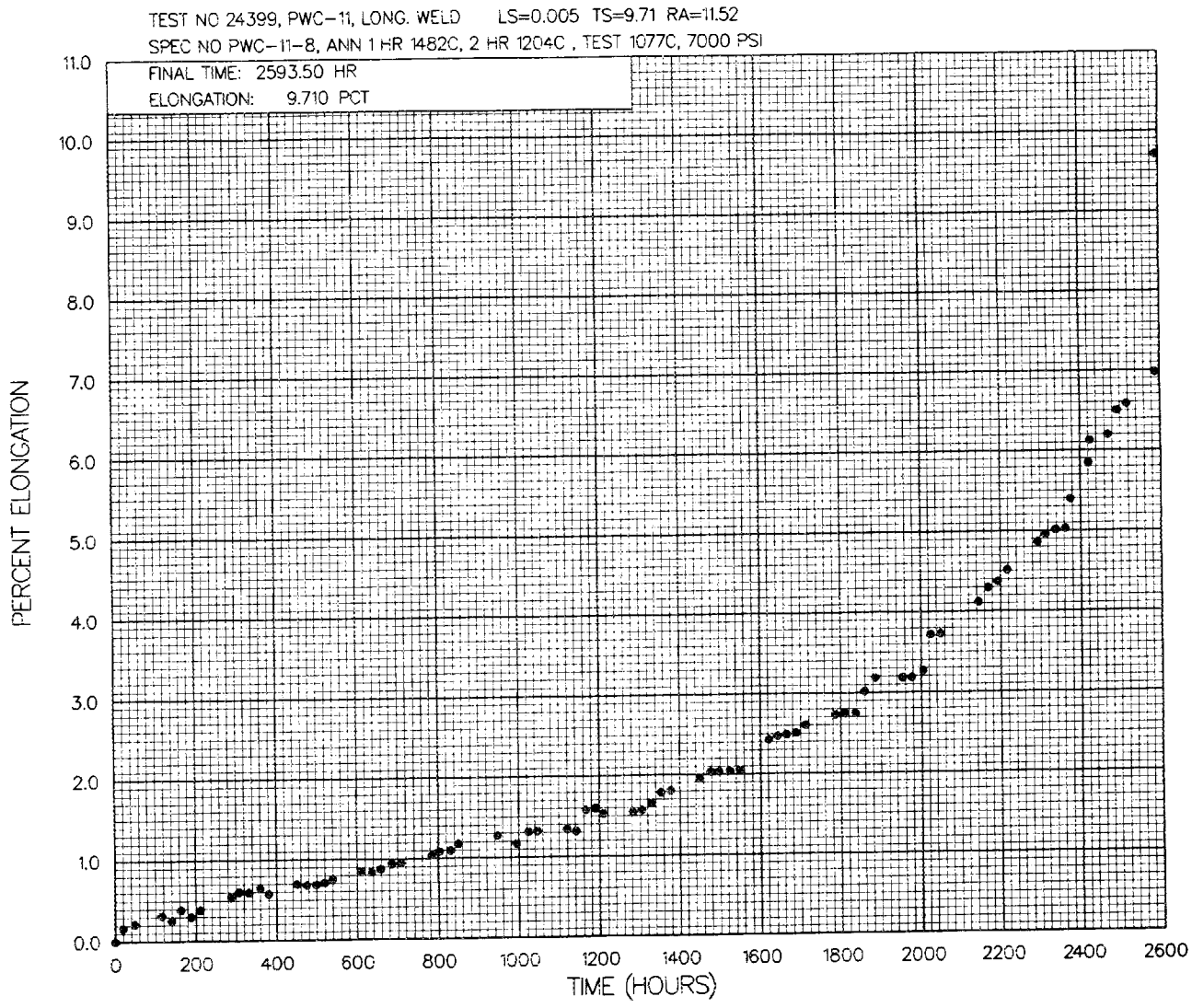

TEST NO. 24748, CB-1ZR, HT 530870 LS=0.094 TS=3.90 RA=3.35
SPEC NO. 530870-4, ANN 1 HR 1400C, 2 HR 1204C, TEST: 1077C, 2500 PSI
FINAL TIME: 1484.60 HR
ELONGATION: 3.810 PCT
PERCENT ELONGATION
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
0
100
200
300
400
500
600
700
800
900
1000
1100
1200
1300
1400
1500
TIME (HOURS)

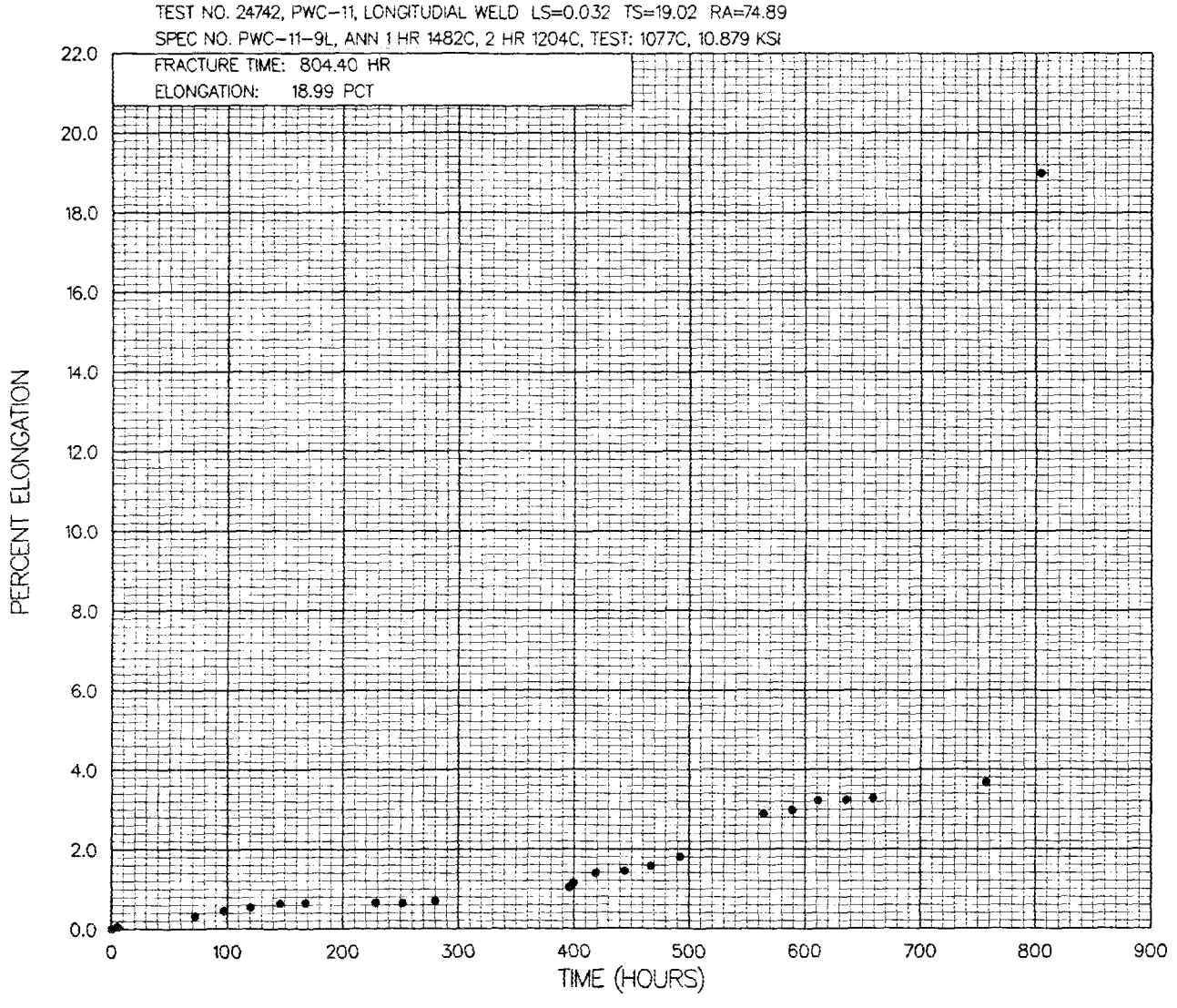

TEST NO. 24680 CB-1ZR, HT 530870
LS=
TS=
RA=
SPEC NO. 530870-1, ANN 1 HR 1400C, TEST 1077C, 2500 PSI
FINAL TIME: 2671.60
ELONGATION: 0.648
PERCENT ELONGATION
1.2
1.0
0.8
0.6
0.4
0.2
0.0
-0.2
0
200
400
600
800
1000
1200
1400
1600
1800
2000
2200
2400
2600
2800
TIME (HOURS)

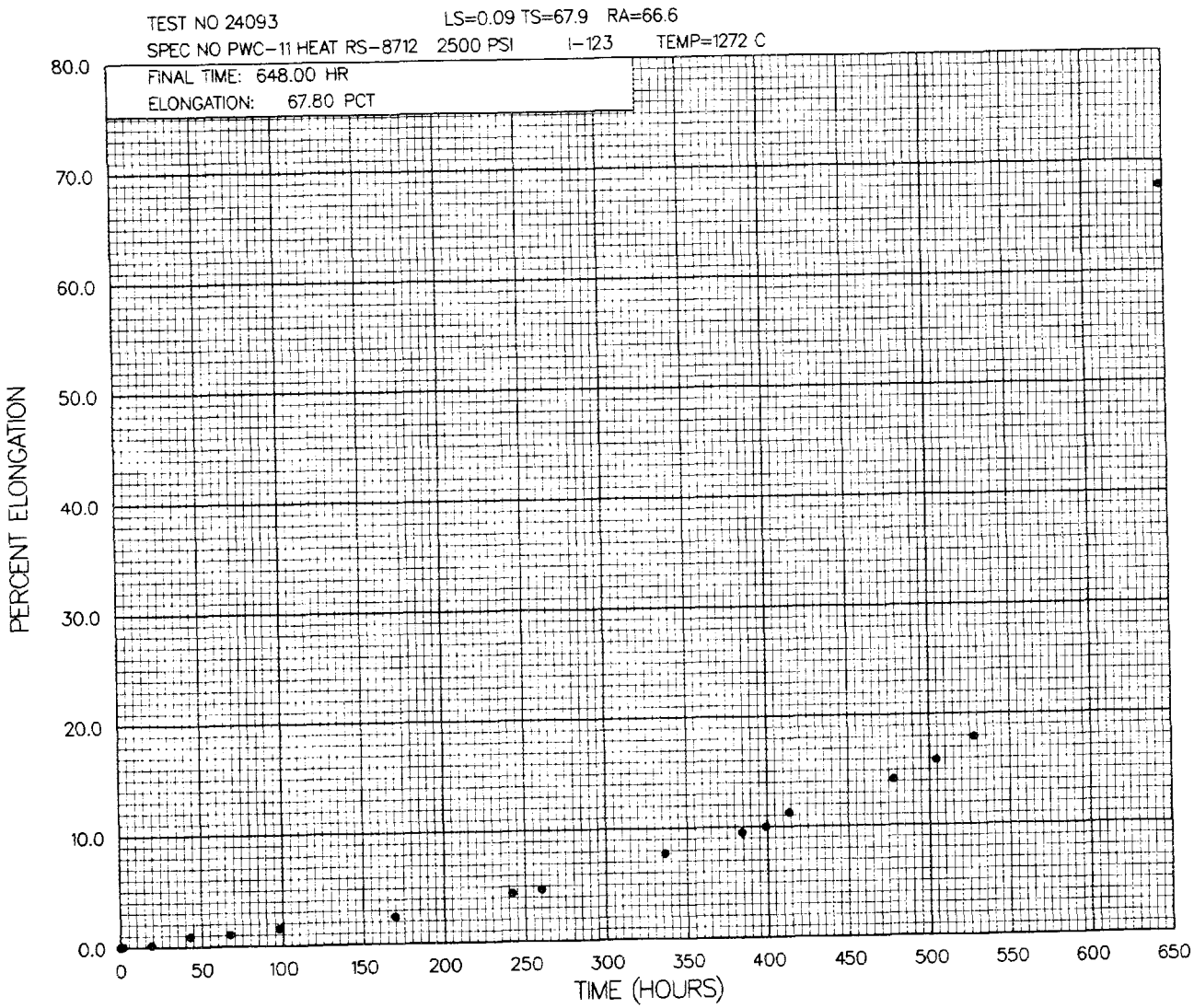

TEST NO. 24095 PWC-11 HEAT RS-8712 LS=0.15 TS=51.5 RA=63.0
SPECIMEN NO. PWC-11-3 10,000 PSI 1077 C
FRACTURE TIME: 297.00 HR
ELONGATION: 51.30 PCT
PERCENT ELONGATION
60.0
55.0
50.0
45.0
40.0
35.0
30.0
25.0
20.0
15.0
10.0
5.0
0.0
0
20
40
60
80
100
120
140
160
180
200
220
240
260
280
300
TIME (HOURS)

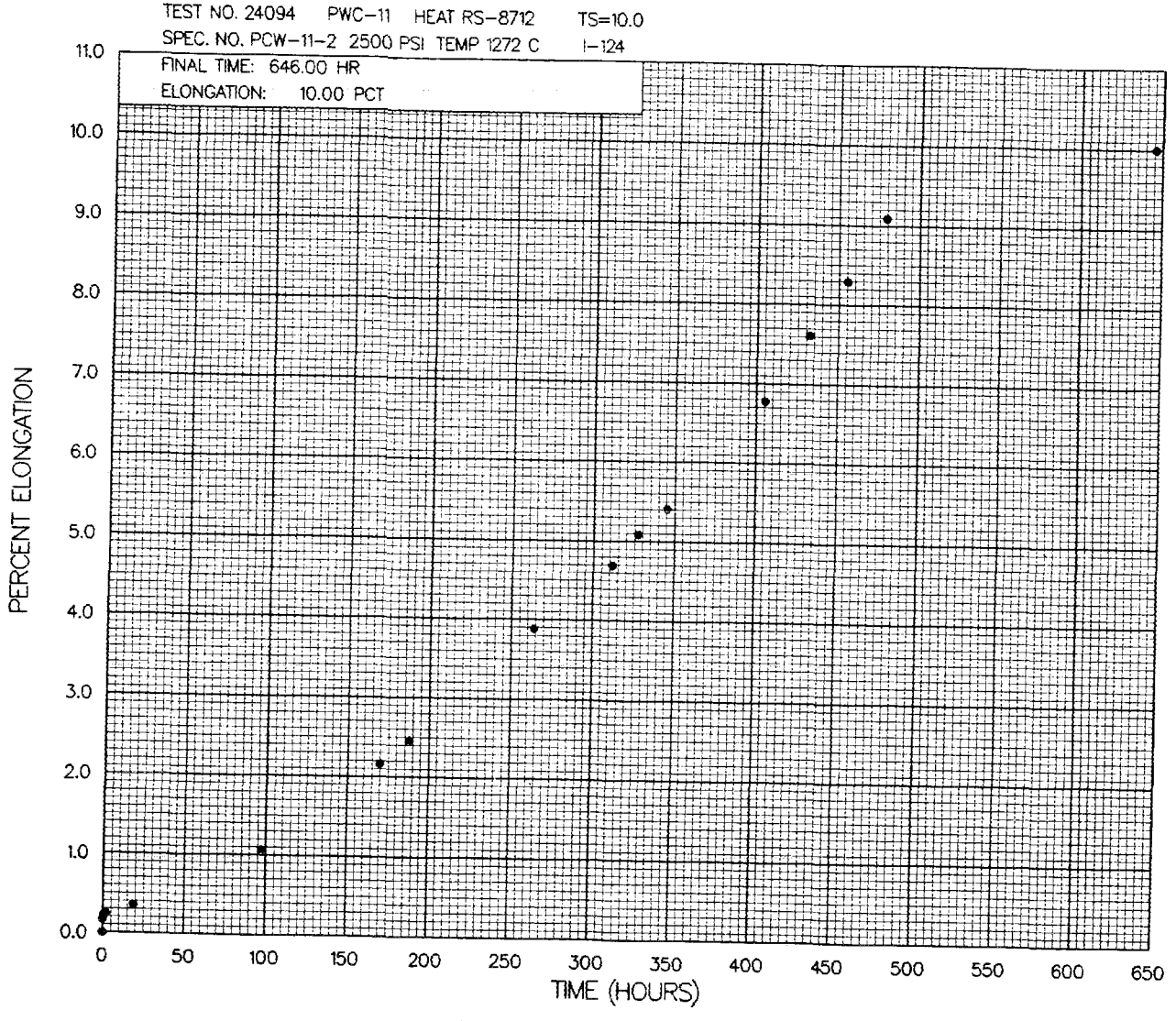

TEST NO. 24104 PWC-11 LS=0.21 TS=48.9 RA=72.79
SPEC NO. PWC-11-4 10000 PSI 1077C
FRACTURE TIME: 346.80 HR
ELONGATION: 48.70 PCT
PERCENT ELONGATION
55.0
50.0
45.0
40.0
35.0
30.0
25.0
20.0
15.0
10.0
5.0
0.0
0
25
50
75
100
125
150
175
200
225
250
275
300
325
350
TIME (HOURS)

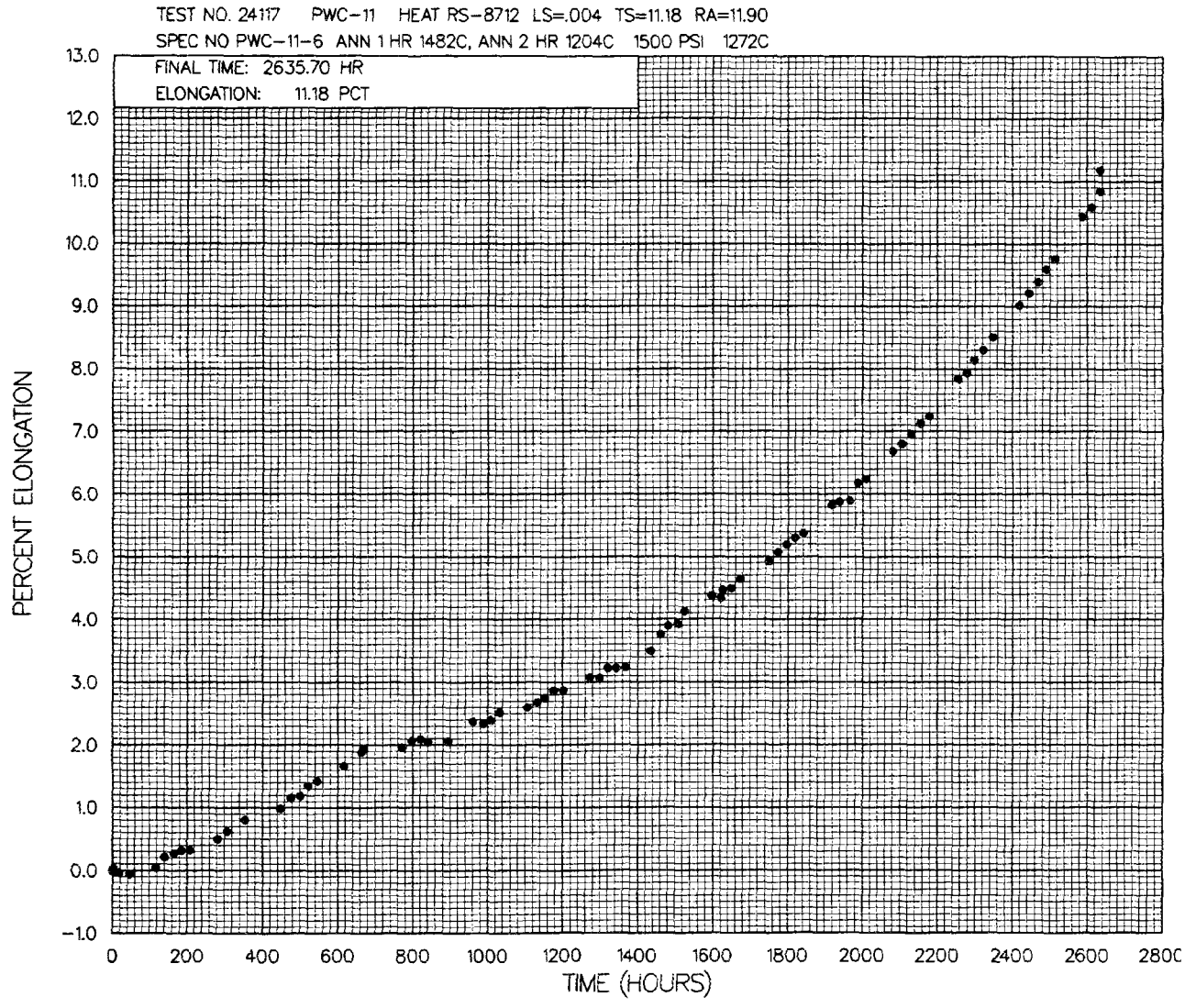

TEST NO 24116B PWC-11
LS=NEG TS=4.00 RA=3.98
SPEC NO. PWC-11-7 1 HR ANN 1482C 2 HR ANN 1204C 5000 PSI 1077C
FINAL TIME: 5750.40 HR
ELONGATION: 4.000 PCT
PERCENT ELONGATION
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
0
500
1000
1500
2000
2500
3000
3500
4000
4500
5000
5500
6000
TIME (HOURS)

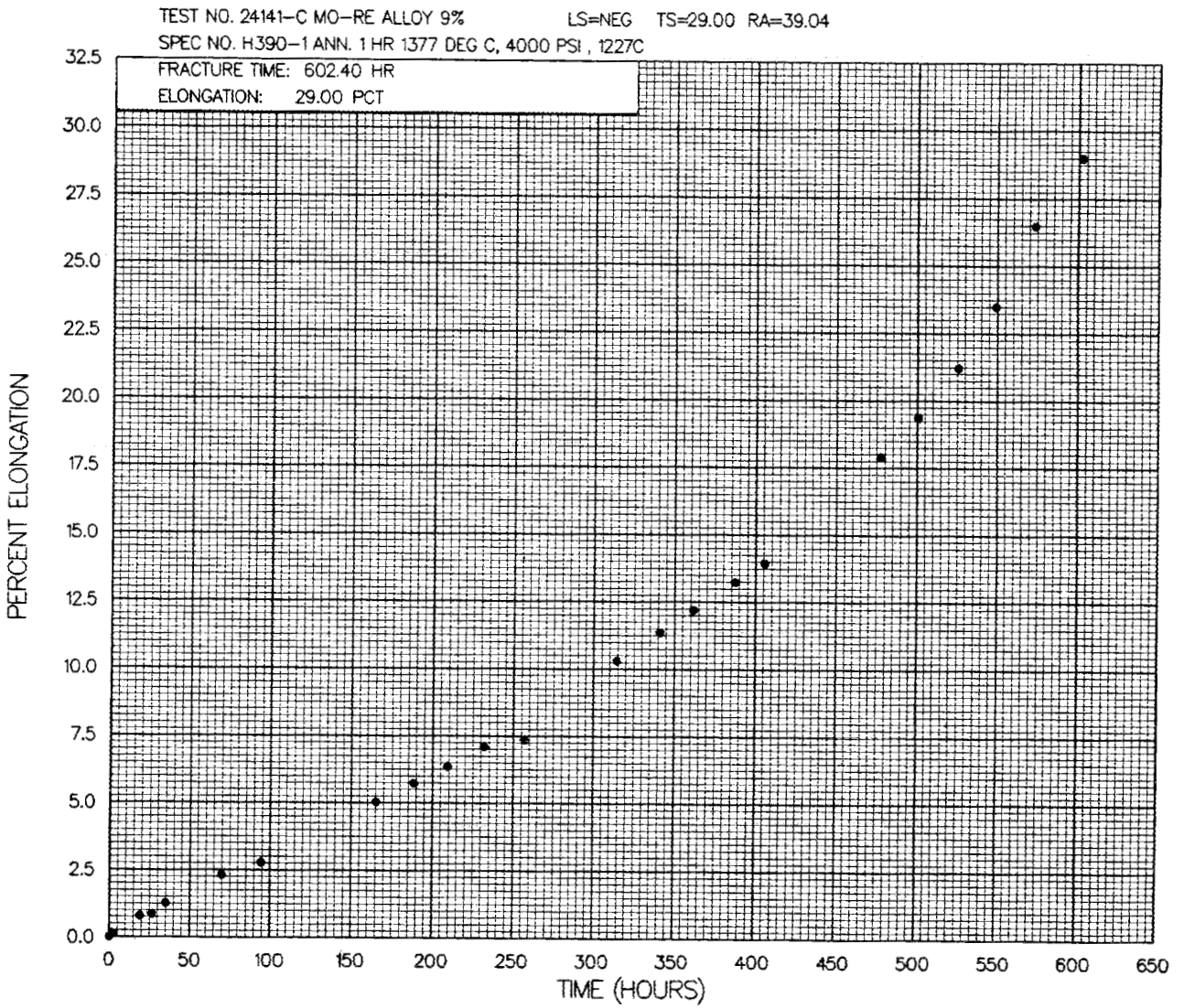

TEST NO 24399, PWC-11, LONG. WELD LS=0.005 TS=9.71 RA=11.52
SPEC NO PWC-11-8, ANN 1 HR 1482C, 2 HR 1204C , TEST 1077C, 7000 PSI
FINAL TIME: 2593.50 HR
ELONGATION: 9.710 PCT
PERCENT ELONGATION
11.0
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
0
200
400
600
800
1000
1200
1400
1600
1800
2000
2200
2400
2600
TIME (HOURS)

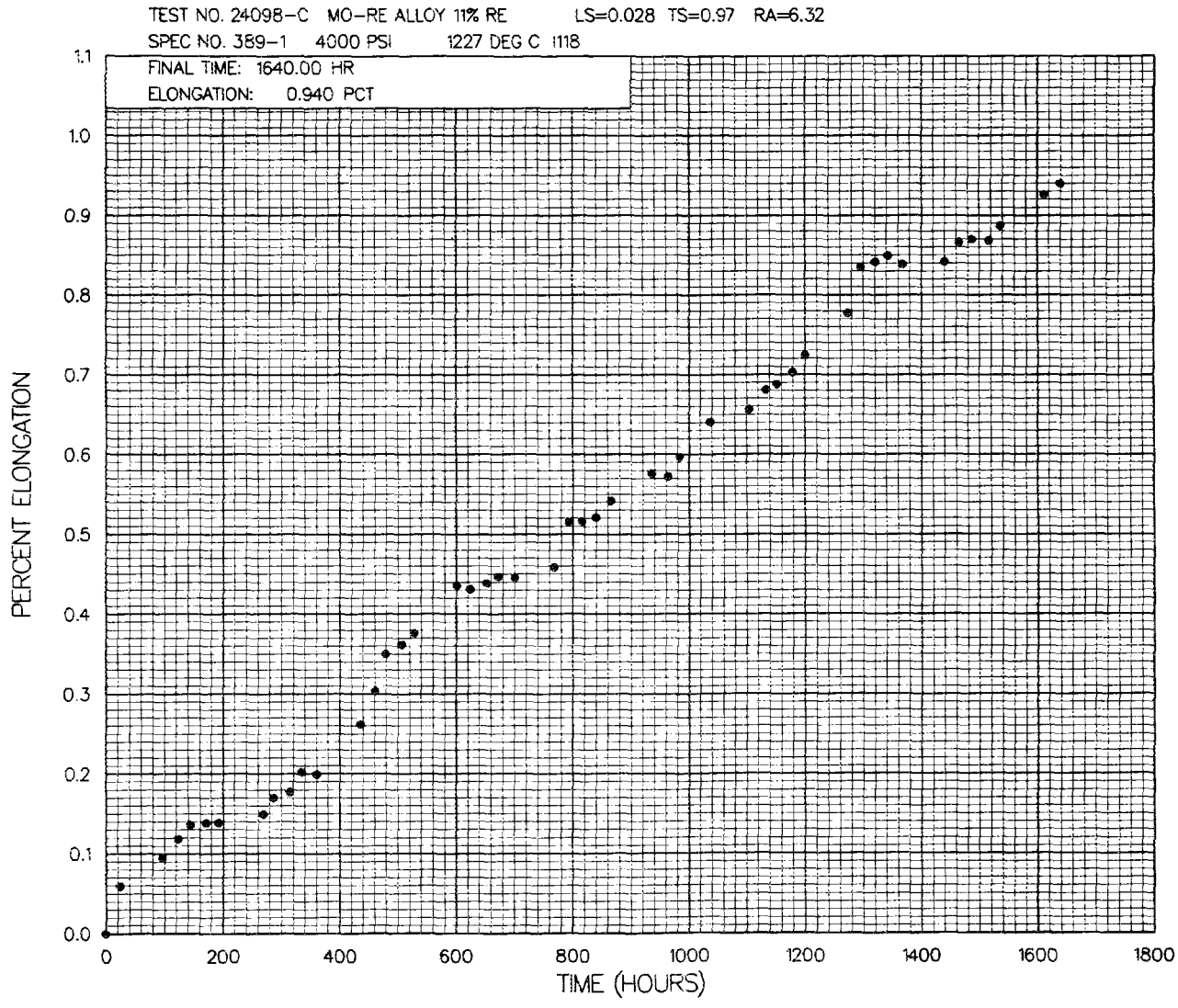

TEST NO. 24742, PWC-11, LONGITUDIAL WELD LS=0.032 TS=19.02 RA=74.89
SPEC NO. PWC-11-9L, ANN 1 HR 1482C, 2 HR 1204C, TEST: 1077C, 10.879 KSI
FRACTURE TIME: 804.40 HR
ELONGATION: 18.99 PCT
PERCENT ELONGATION
22.0
20.0
18.0
16.0
14.0
12.0
10.0
8.0
6.0
4.0
2.0
0.0
0
100
200
300
400
500
600
700
800
900
TIME (HOURS)

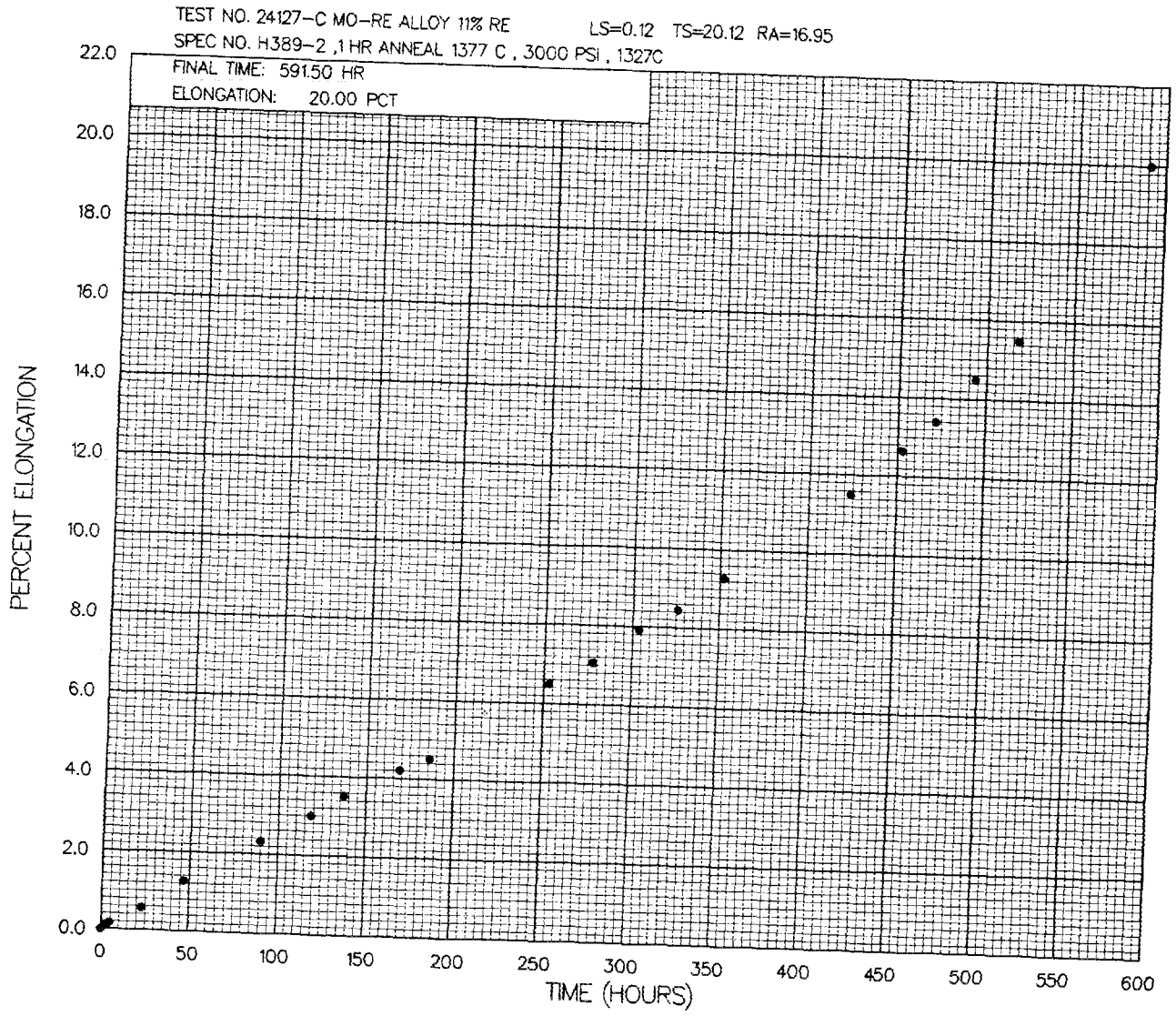

TEST NO 24093
LS=0.09 TS=67.9 RA=66.6
SPEC NO PWC-11 HEAT RS-8712 2500 PSI I-123 TEMP=1272 C
FINAL TIME: 648.00 HR
ELONGATION: 67.80 PCT
PERCENT ELONGATION
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
0.0
0
50
100
150
200
250
300
350
400
450
500
550
600
650
TIME (HOURS)

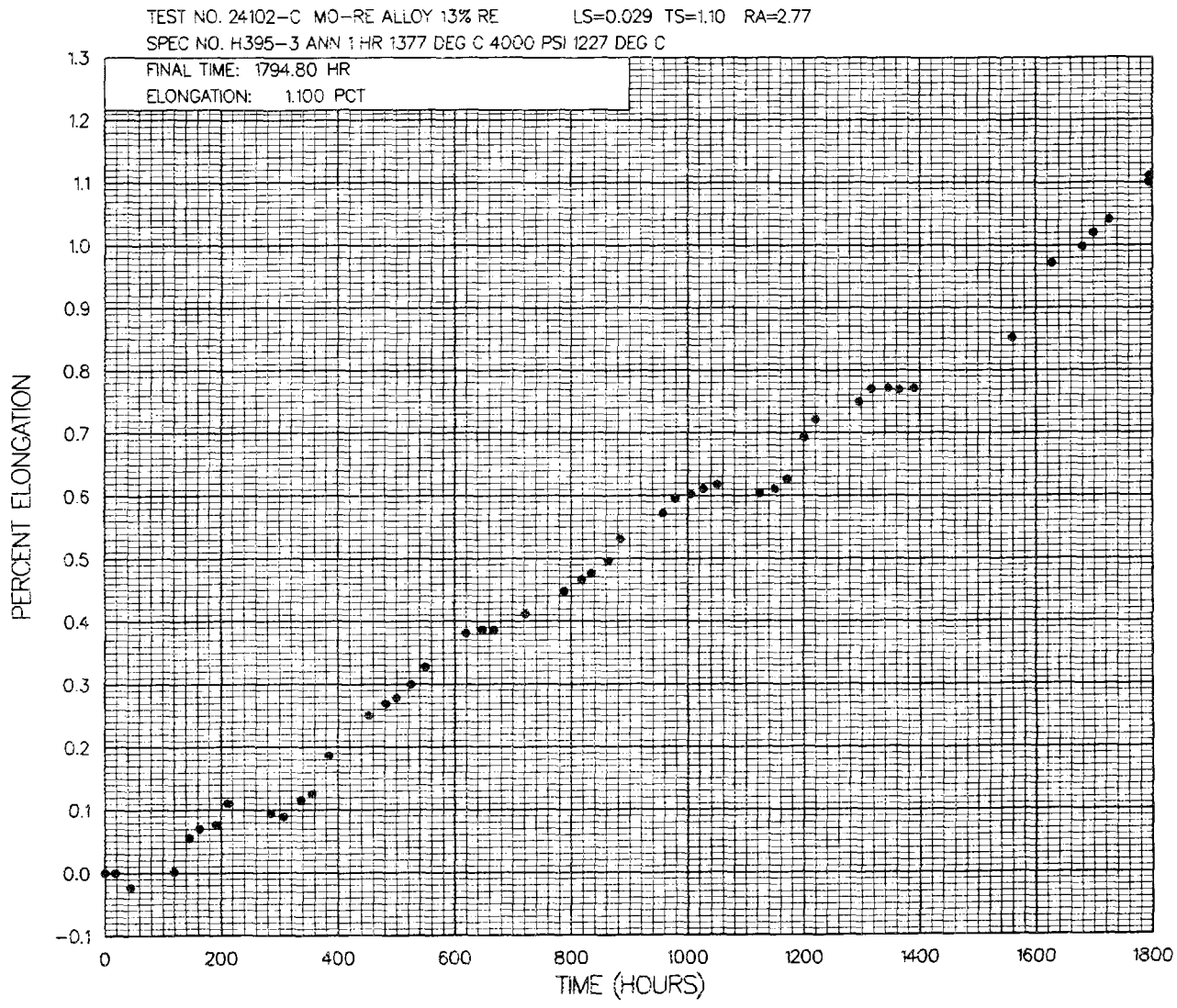

TEST NO. 24094 PWC-11 HEAT RS-8712 TS=10.0
SPEC. NO. PCW-11-2 2500 PSI TEMP 1272 C I-124
FINAL TIME: 646.00 HR
ELONGATION: 10.00 PCT
PERCENT ELONGATION
11.0
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
0
50
100
150
200
250
300
350
400
450
500
550
600
650
TIME (HOURS)

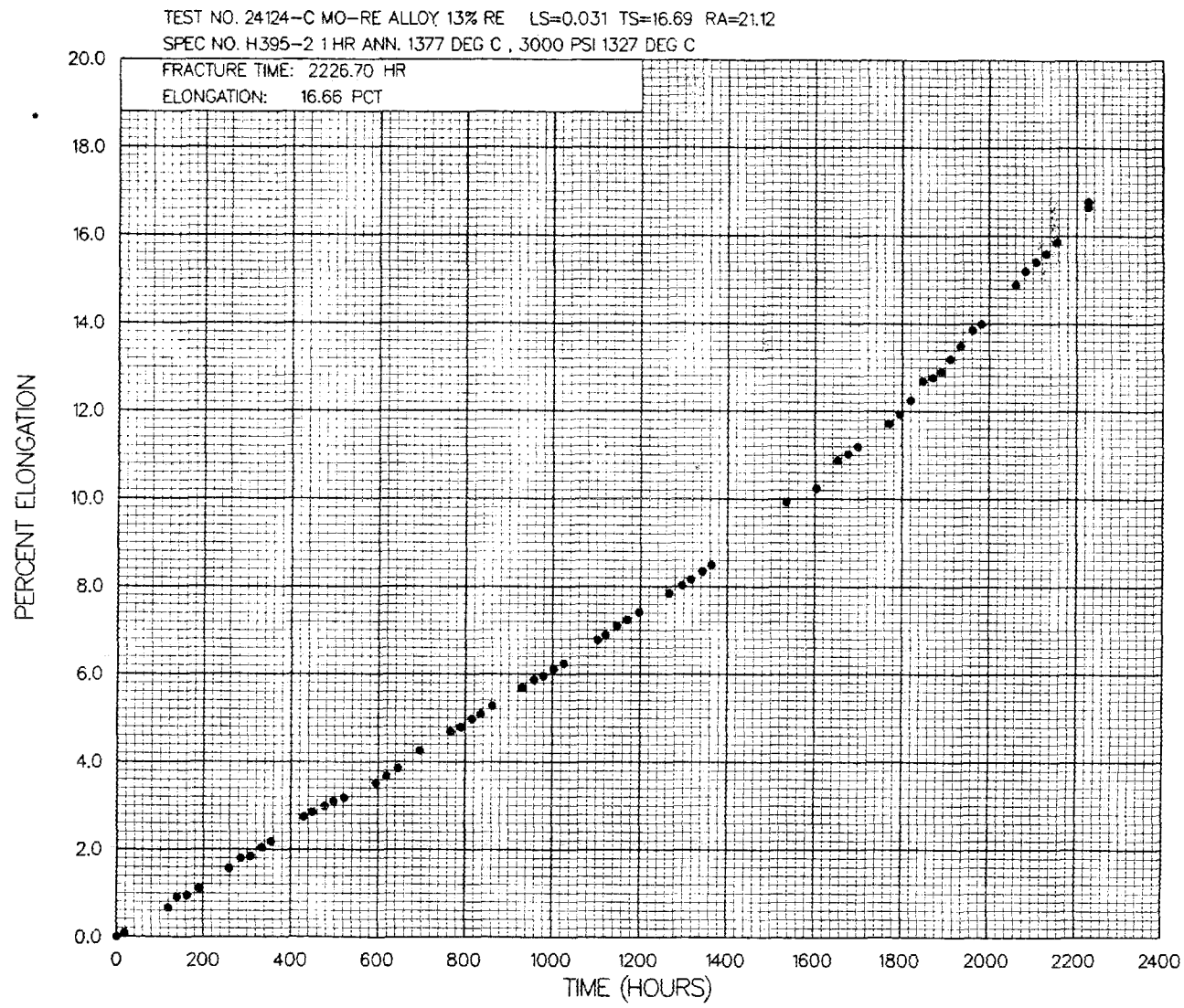

TEST NO. 24117 PWC-11 HEAT RS-8712 LS=.004 TS=11.18 RA=11.90
SPEC NO PWC-11-6 ANN 1 HR 1482C, ANN 2 HR 1204C 1500 PSI 1272C
FINAL TIME: 2635.70 HR
ELONGATION: 11.18 PCT
PERCENT ELONGATION
13.0
12.0
11.0
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
-1.0
0
200
400
600
800
1000
1200
1400
1600
1800
2000
2200
2400
2600
2800
TIME (HOURS)

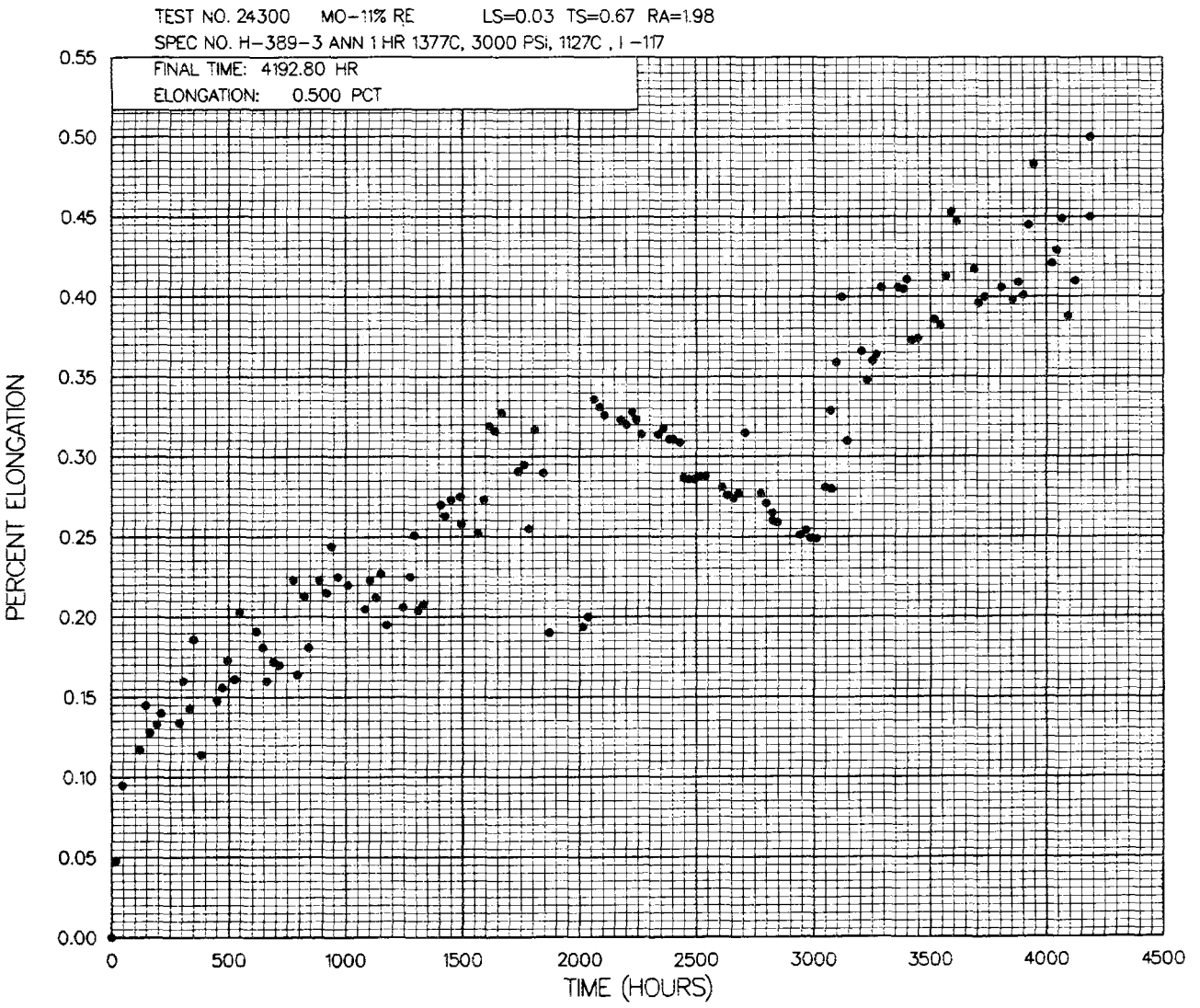

TEST NO. 24141-C MO-RE ALLOY 9%
LS=NEG TS=29.00 RA=39.04
SPEC NO. H390-1 ANN. 1 HR 1377 DEG C, 4000 PSI , 1227C
FRACTURE TIME: 602.40 HR
ELONGATION: 29.00 PCT
PERCENT ELONGATION
32.5
30.0
27.5
25.0
22.5
20.0
17.5
15.0
12.5
10.0
7.5
5.0
2.5
0.0
0
50
100
150
200
250
300
350
400
450
500
550
600
650
TIME (HOURS)

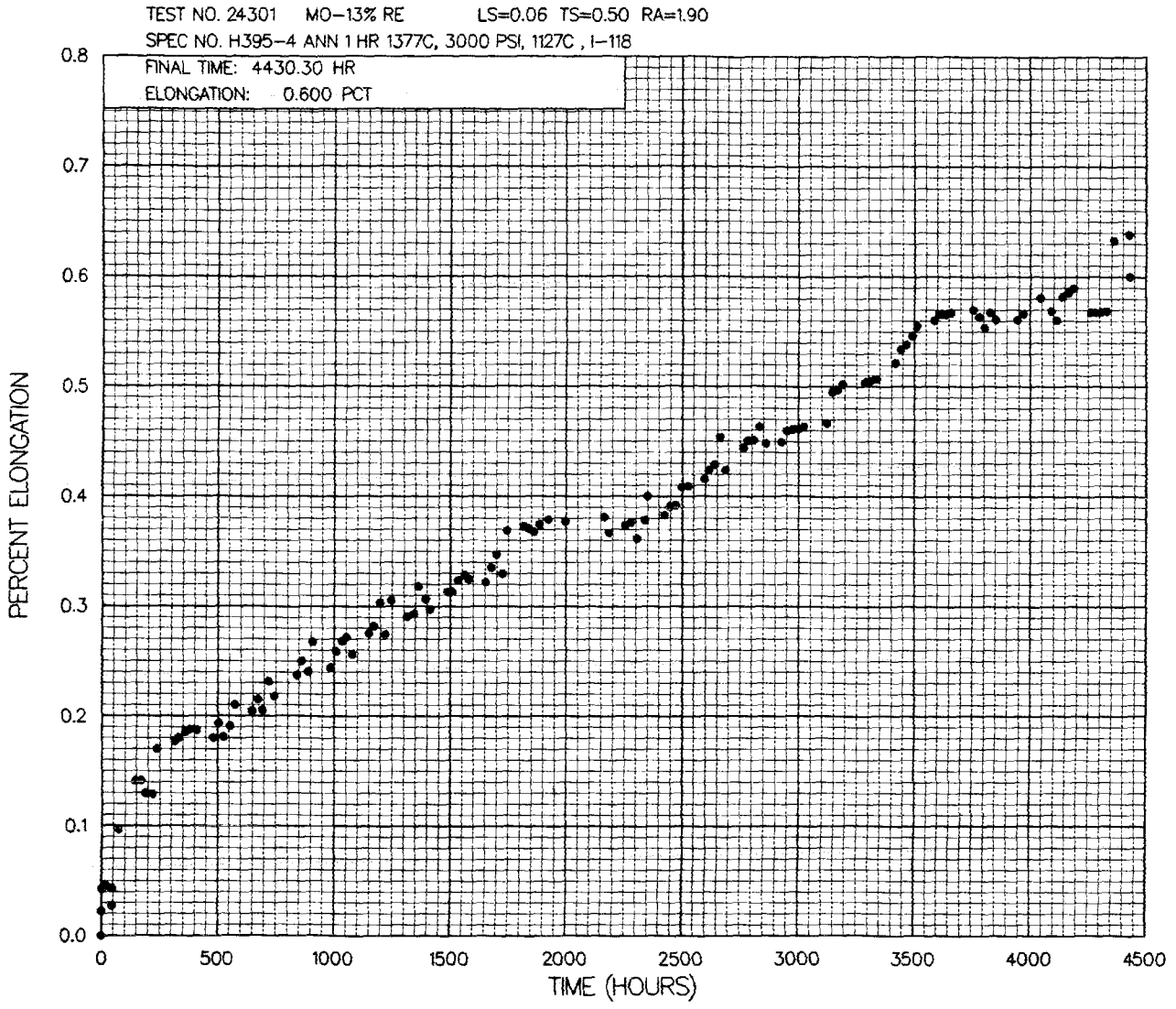

TEST NO. 24098-C MO-RE ALLOY 11% RE LS=0.028 TS=0.97 RA=6.32
SPEC NO. 389-1 4000 PSI 1227 DEG C 1118
FINAL TIME: 1640.00 HR
ELONGATION: 0.940 PCT
PERCENT ELONGATION
TIME (HOURS)
0.0
0.1
0.2
0.3
0.4
0.5
0.6
0.7
0.8
0.9
1.0
1.1
0
200
400
600
800
1000
1200
1400
1600
1800

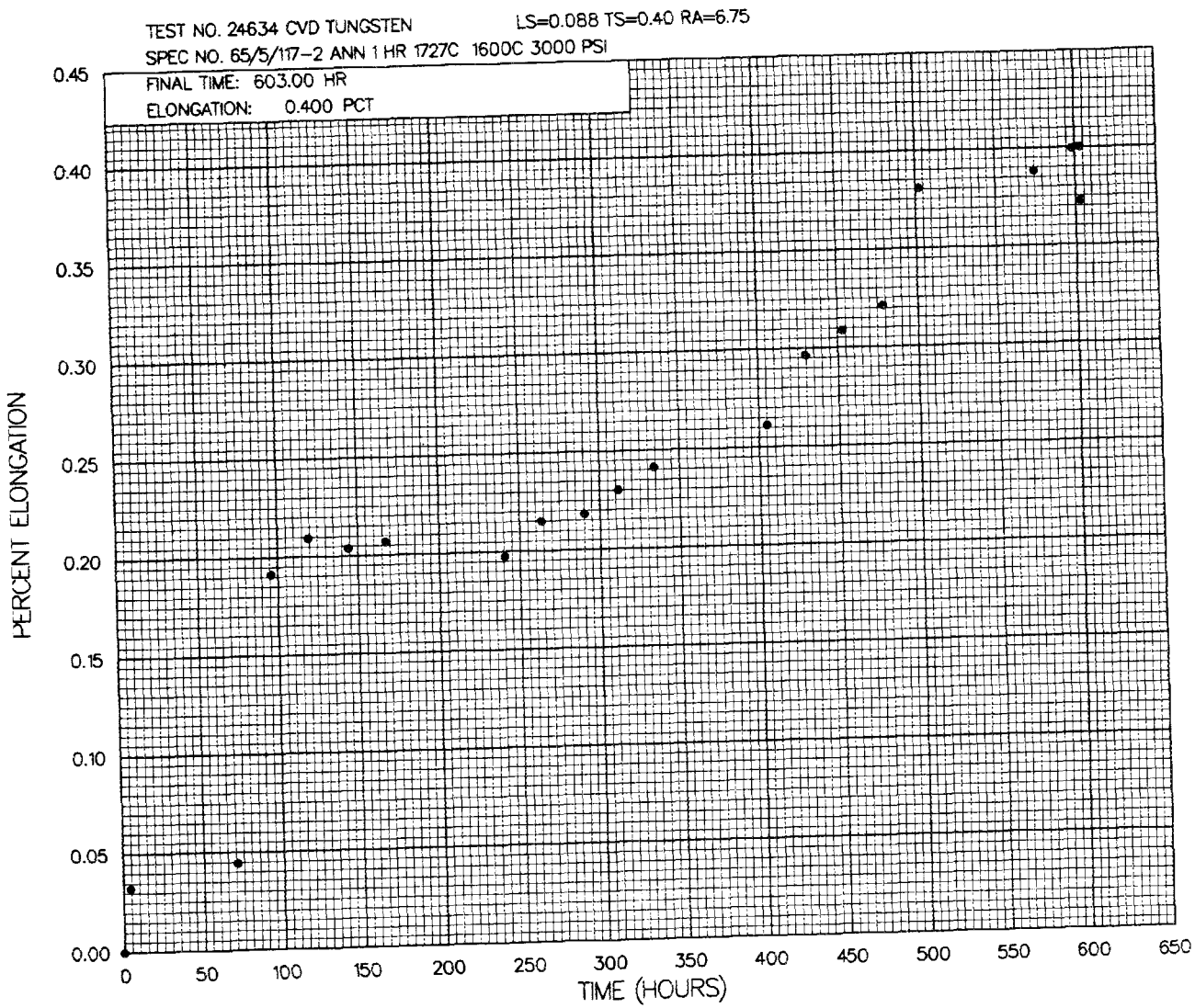

TEST NO. 24127-C MO-RE ALLOY 11% RE
LS=0.12 TS=20.12 RA=16.95
SPEC NO. H389-2 ,1 HR ANNEAL 1377 C , 3000 PSI , 1327C
FINAL TIME: 591.50 HR
ELONGATION: 20.00 PCT
PERCENT ELONGATION
22.0
20.0
18.0
16.0
14.0
12.0
10.0
8.0
6.0
4.0
2.0
0.0
0
50
100
150
200
250
300
350
400
450
500
550
600
TIME (HOURS)

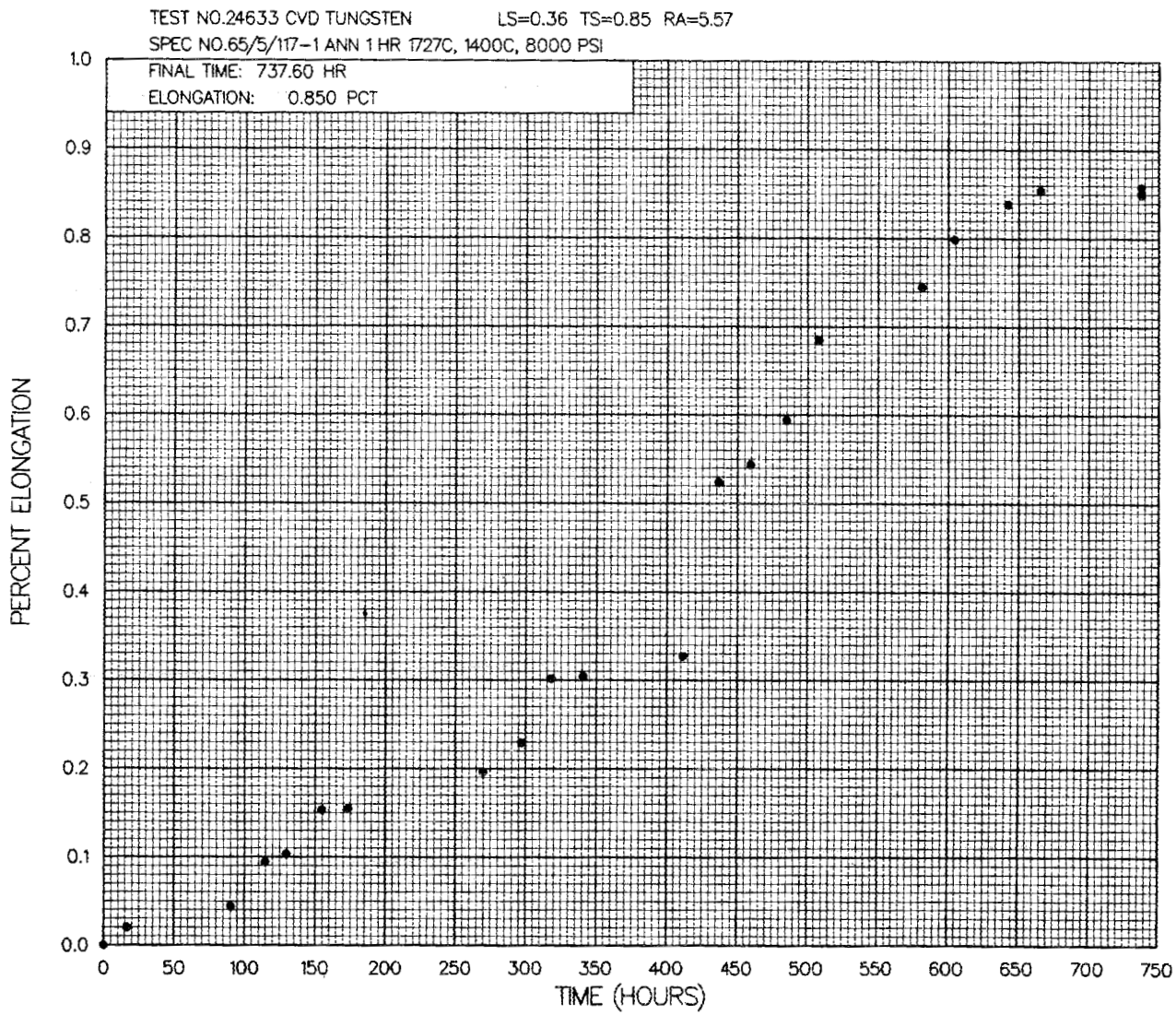

TEST NO. 24102-C MO-RE ALLOY 13% RE LS=0.029 TS=1.10 RA=2.77
SPEC NO. H395-3 ANN 1 HR 1377 DEG C 4000 PSI 1227 DEG C
FINAL TIME: 1794.80 HR
ELONGATION: 1.100 PCT
PERCENT ELONGATION
1.3
1.2
1.1
1.0
0.9
0.8
0.7
0.6
0.5
0.4
0.3
0.2
0.1
0.0
-0.1
0
200
400
600
800
1000
1200
1400
1600
1800
TIME (HOURS)

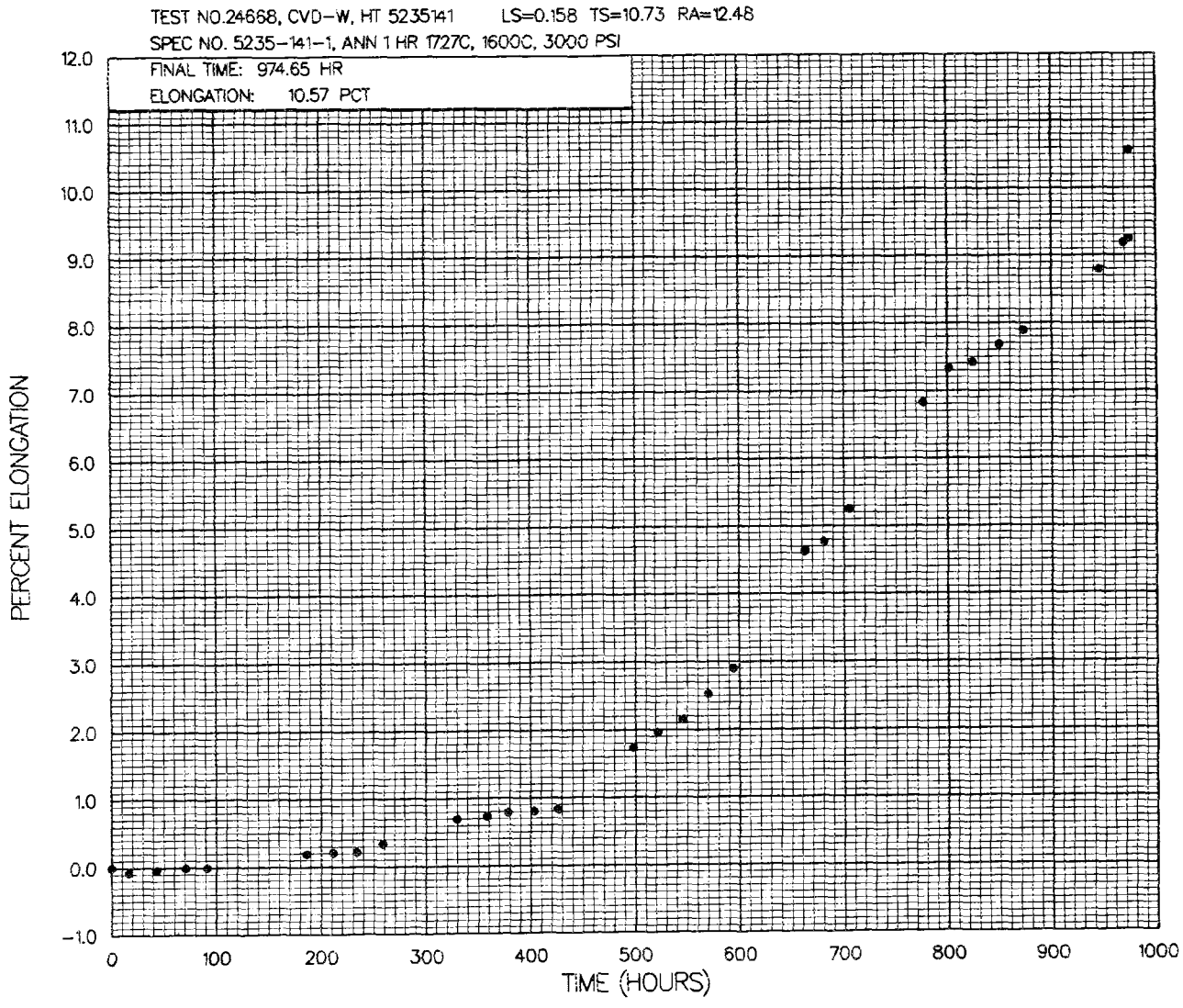

TEST NO. 24124-C MO-RE ALLOY 13% RE LS=0.031 TS=16.69 RA=21.12
SPEC NO. H395-2 1 HR ANN. 1377 DEG C , 3000 PSI 1327 DEG C
FRACTURE TIME: 2226.70 HR
ELONGATION: 16.66 PCT
PERCENT ELONGATION
20.0
18.0
16.0
14.0
12.0
10.0
8.0
6.0
4.0
2.0
0.0
0
200
400
600
800
1000
1200
1400
1600
1800
2000
2200
2400
TIME (HOURS)

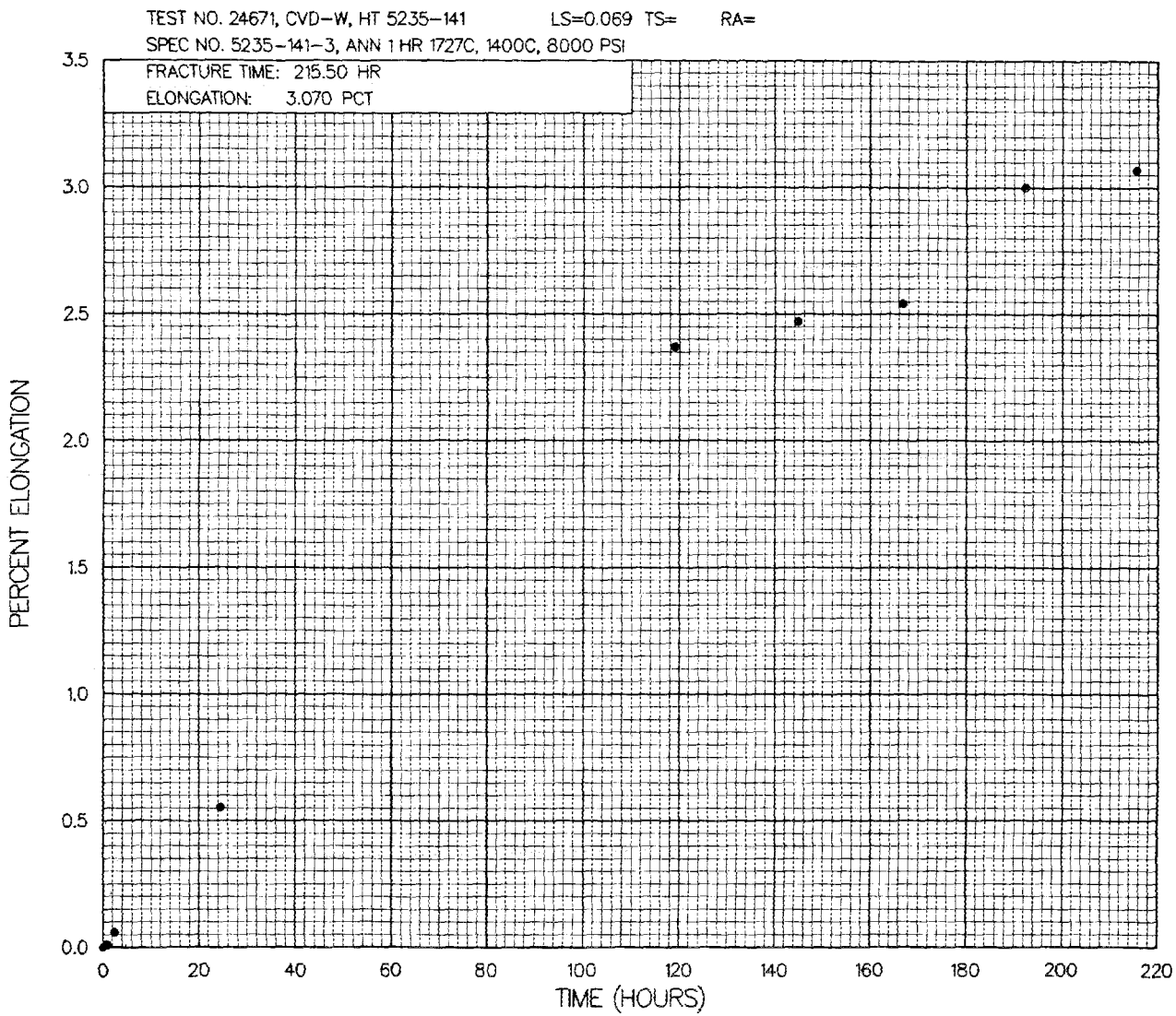

TEST NO. 24300 MO-11% RE LS=0.03 TS=0.67 RA=1.98
SPEC NO. H-389-3 ANN 1 HR 1377C, 3000 PSi, 1127C , I -117
FINAL TIME: 4192.80 HR
ELONGATION: 0.500 PCT
PERCENT ELONGATION
0.55
0.50
0.45
0.40
0.35
0.30
0.25
0.20
0.15
0.10
0.05
0.00
0
500
1000
1500
2000
2500
3000
3500
4000
4500
TIME (HOURS)

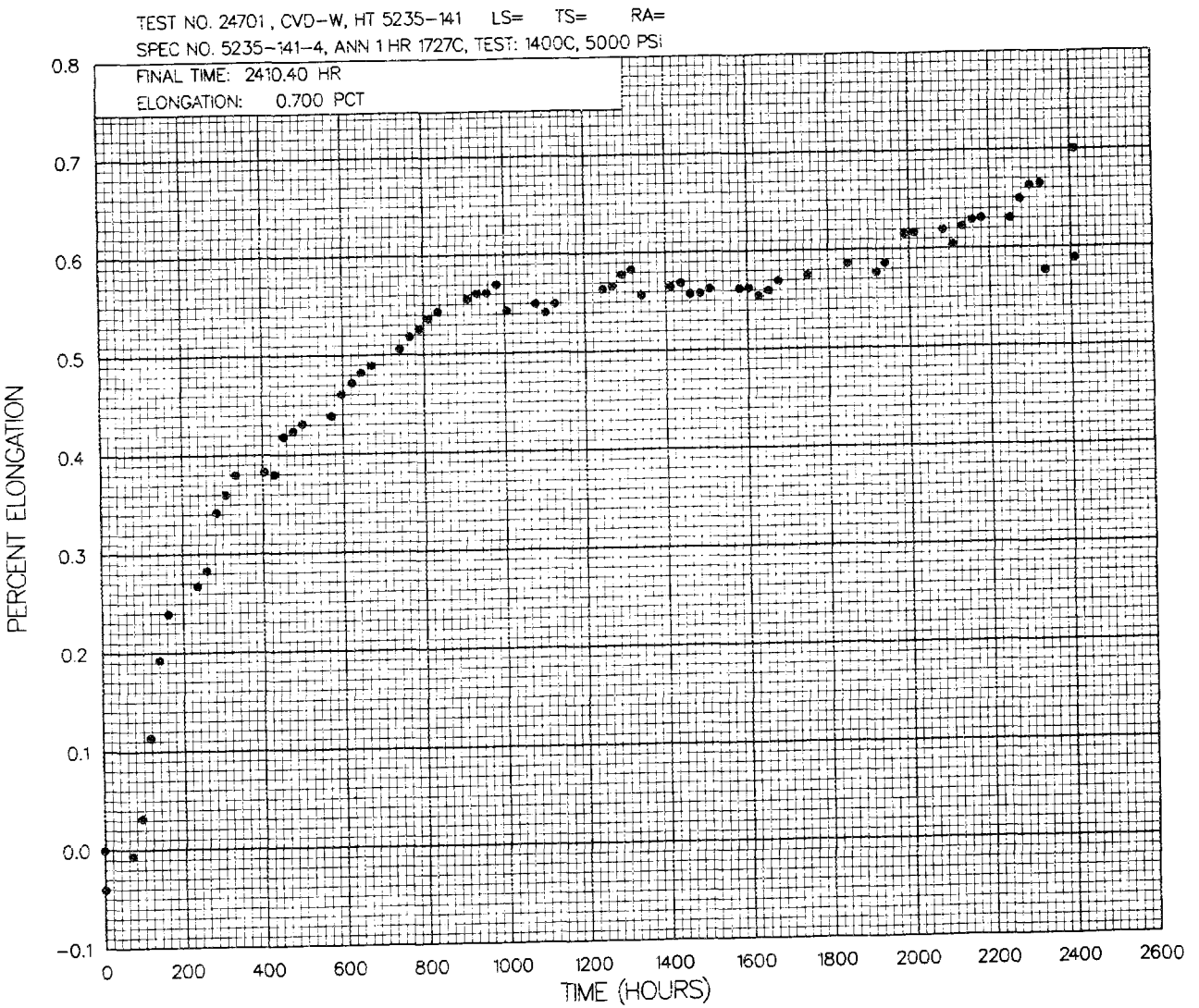

TEST NO. 24301 MO-13% RE LS=0.06 TS=0.50 RA=1.90
SPEC NO. H395-4 ANN 1 HR 1377C, 3000 PSI, 1127C , I-118
FINAL TIME: 4430.30 HR
ELONGATION: 0.600 PCT
PERCENT ELONGATION
0.0
0.1
0.2
0.3
0.4
0.5
0.6
0.7
0.8
0
500
1000
1500
2000
2500
3000
3500
4000
4500
TIME (HOURS)


TEST NO. 24634 CVD TUNGSTEN
LS=0.088 TS=0.40 RA=6.75
SPEC NO. 65/5/117-2 ANN 1 HR 1727C 1600C 3000 PSI
FINAL TIME: 603.00 HR
ELONGATION: 0.400 PCT
PERCENT ELONGATION
0.45
0.40
0.35
0.30
0.25
0.20
0.15
0.10
0.05
0.00
0
50
100
150
200
250
300
350
400
450
500
550
600
650
TIME (HOURS)


TEST NO.24633 CVD TUNGSTEN
LS=0.36 TS=0.85 RA=5.57
SPEC NO.65/5/117-1 ANN 1 HR 1727C, 1400C, 8000 PSI
FINAL TIME: 737.60 HR
ELONGATION: 0.850 PCT
PERCENT ELONGATION
1.0
0.9
0.8
0.7
0.6
0.5
0.4
0.3
0.2
0.1
0.0
0
50
100
150
200
250
300
350
400
450
500
550
600
650
700
750
TIME (HOURS)


TEST NO.24668, CVD-W, HT 5235141 LS=0.158 TS=10.73 RA=12.48
SPEC NO. 5235-141-1, ANN 1 HR 1727C, 1600C, 3000 PSI
FINAL TIME: 974.65 HR
ELONGATION: 10.57 PCT
PERCENT ELONGATION
12.0
11.0
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
-1.0
0
100
200
300
400
500
600
700
800
900
1000
TIME (HOURS)


TEST NO. 24671, CVD-W, HT 5235-141
LS=0.069 TS=
RA=
SPEC NO. 5235-141-3, ANN 1 HR 1727C, 1400C, 8000 PSI
FRACTURE TIME: 215.50 HR
ELONGATION: 3.070 PCT
PERCENT ELONGATION
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
0
20
40
60
80
100
120
140
160
180
200
220
TIME (HOURS)


TEST NO. 24701 , CVD-W, HT 5235-141 LS= TS= RA=
SPEC NO. 5235-141-4, ANN 1 HR 1727C, TEST: 1400C, 5000 PSI
FINAL TIME: 2410.40 HR
ELONGATION: 0.700 PCT
PERCENT ELONGATION
0.8
0.7
0.6
0.5
0.4
0.3
0.2
0.1
0.0
-0.1
0
200
400
600
800
1000
1200
1400
1600
1800
2000
2200
2400
2600
TIME (HOURS)

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