MSAR 64-14

PROGRESS REPORT 41
January 1964

to

U. S. Atomic Energy Commission
Chicago Operations Office
Lemont, Illinois

EFFECT OF HIGH TEMPERATURE SODIUM ON AUSTENITIC AND FERRITIC STEELS

Physical Properties of Materials

Contract AT(11-1)-765
Modification No. 1

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February 18, 1964

MSA Research Corporation

Subsidiary of Mine Safety Appliances Company

Callery, Pennsylvania

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DISCLAIMER

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Progress Report 41

EFFECT OF HIGH TEMPERATURE SODIUM ON AUSTENITIC AND FERRITIC STEELS

PHYSICAL PROPERTIES OF MATERIALS

SUMMARY

The physical properties research program was initiated for the purpose of determining the effects of high temperature reactor grade sodium, and normally anticipated contaminants, upon Type 316 stainless steel and 2 1/4 Cr-1 Mo steel. For comparison, the physical properties of these same materials were to be determined in air and helium. The test temperatures selected were 1200 F for the austenitic and 1100 F for the ferritic. The contaminants to be intentionally introduced into the sodium after the reactor grade sodium tests were completed would be oxygen, carbon, and nitrogen. The types of test selected for revealing any possible effects of the environments upon the materials were tensile, creep, creep-to-rupture, and fatigue.

TEST 1 - STAINLESS STEEL (316) SPECIMENS IN 1200 F, LOW OXYGEN (30 ppm) SODIUM, AIR AND HELIUM

This test has been completed. A topical report is being written which will include the results of the environmental runs and will attempt to correlate these results with the analytical and metallurgical findings.

TEST 2 - 2 1/4 Cr-1 Mo STEEL SPECIMENS IN 1100 F, LOW OXYGEN (30 ppm) SODIUM, AIR AND HELIUM

The status of this test is the same as TEST 1 above.

TEST 3 - STAINLESS STEEL (316) SPEICMENS IN 1200 F, HIGH CARBON SODIUM

The overall complexity of the sodium-carbon-stainless steel system, coupled with the current "state of the art" of this same system,has delayed the actual testing of specimens. Many hours of studies have been achieved with this system to date. These studies include the problems associated with saturating the system with carbon, the sampling and analyses of a dynamic system for carbon, the evaluation of the carburization of metal tabs in 1, 4, 6, 11 and 20 day tests, and the effects of high carbon concentrations on cold trapping and plugging indicator operations.

The AEC has requested a re-evaluation of TEST 3 along with the remaining TESTS in the program. The AEC Ad Hoc Sub-committee will meet at MSAR on February 4 to conduct this review. Meanwhile the system is in operation, ready to receive test specimens, but temporarily we are studying the effects of cold trapping on carbon until the test program has been clarified.

TEST 4 - 2 1/4 Cr-1 Mo STEEL SPECIMENS IN 1100 F, HIGH (300 ppm) OXYGEN SODIUM

The creep, creep-to-rupture and fatigue tests in sodium on both original and exposed specimens have been completed. Exposed specimens have been sent to the University of Michigan for creep-rupture and tensile tests in helium. The creep-rupture tests show a shorter life of both the original and exposed specimens in 1100 F sodium with 300 ppm as compared to sodium with 30 ppm. The fatigue tests showed no difference in fatigue life of 2 1/4 Cr-1 Mo steel in sodium with 300 ppm or 30 ppm oxygen.

The creep tests show the stress for a minimum creep rate of 1% in 10,000 hours to be higher (lower creep rate) in sodium with high oxygen than in sodium with low oxygen, but the creep rate is still higher than in air or helium.

Preliminary results indicate faster decarburization of 2 1/4 Cr-1 Mo steel specimens in sodium contaminated with oxygen than in clean sodium.

Progress Report 41

EFFECT OF HIGH TEMPERATURE SODIUM ON AUSTENITIC AND FERRITIC STEELS

PHYSICAL PROPERTIES OF MATERIALS

1. INTRODUCTION

TESTS 1 and 2 have been completed. These programs are covered under AEC Contract AT(11-1)-765 and modified by letters from F. C. Mattmueller (Director, Contracts Division) to Dr. R. C. Werner (MSAR), dated February 12, 1962, and to C. H. Staub (MSAR) on October 24, 1962, and January 8, 1963. Topical Report No. 1. "Test Facility Design and Operation Procedures," (MSAR 63-161) has been recently issued. A second topical report presenting the results of TEST 1 is in the final stages of completion and will be issued this month. The examination of specimens generated from TEST 2 is nearing completion.

TESTS 3 and 4 are covered under this contract, as outlined in a letter from F. C. Mattmueller to C. H. Staub, dated October 24, 1962, and revised by letter from F. C. Mattmueller to C. H. Staub, dated January 8, 1963.

The preliminary operations of TEST 3 have been temporarily interrupted after completion of the tab tests. High rates of carburization during these tests and difficulty in operation of the oxide indicating and control system under these conditions have been responsible for this delay. The conditions of TEST 3 are to be re-evaluated in view of the experiences and results obtained to date from the tab tests. A meeting at MSAR of the Ad Hoc Subcommittee and MSAR representatives on February 4 and 5 will re-evaluate these conditions, as well as the remainder of the physical properties test schedule.

The sodium phase of TEST 4 at MSAR has been completed and metallographic examination of the specimens generated during the run is in progress. The testing in helium of the specimens exposed for 4000 hours in 1100 F high oxide (300 ppm) sodium has been initiated at the University of Michigan under the direction of Dr. J. W. Freeman.

2. OPERATION

2.1 TEST 3 - STAINLESS STEEL (316) SPECIMENS IN 1200 F, HIGH CARBON SODIUM

The operation of Loop 1 with metal tabs for determining the carburization potential of the sodium with a carbon source has been completed. The results of these preliminary runs were presented in Progress Report 40.

The operation sequence of Loop 1 for this report period is as follows:

1. As reported last period, a plug had occurred in the OCI system. At that time several of the OCI components had been removed from the system to determine if the plug could be located and analyzed. Washings from several places suspected of containing plugs are being analyzed.

2. On January 3, 1964, the complete system was drained in order to remove the OCI inlet valve which appeared to be plugged.

3. By January 9, 1964, all components had been washed, dried and installed back into the loop. The main system was then charged and flow was established in the main system, the carbon bed being isolated.

4. After cold trapping the main system, flow was established through the carbon bed on January 9, 1964, and cold trapping continued. Following several hours of cold trapping, there was an indication of a plug being formed in the OCI system; therefore the carbon bed was isolated.

5. The obstruction in the OCI system disappeared and flow was again established through the carbon bed on January 13, 1964. Again, after several hours, the OCI system began plugging and again the carbon bed was isolated.

6. On January 21, 1964, Mr. Purcell (AEC) advised MSAR not to initiate TEST 3 until after the meeting with the Sub-committee on February 4 and 5.

7. From January 21, 1964 to the end of this report period, the system has been operated with flow confined to the main system (i.e., the carbon bed isolated). During this interim, the plugging valve bellows failed and was replaced. The effects of cold trapping on the carbon concentration are being studied.

As reported earlier, the addition of carbon affects the operation of the plugging indicator such that saturation values are considerably higher than oxide saturation temperatures obtained by chemical analysis. Although it is difficult to determine if the carbon concentration is affected by cold trapping due to scatter in the data, these plugging runs appear to be lowered by cold trapping. It is possible, however, that this lowering could be caused by loss of carbon from the sodium by carburization of the stainless steel in the system.

Since this system was not originally designed to carry out an oxygen-carbon-sodium-stainless steel study, it is difficult to determine and control all parameters that may affect this relationship. However, we hope to show that the carbon potential of the system can be determined with the plugging indicator and the oxygen level can be successfully controlled with the cold trap under high carbon concentrations. These are the parameters that must be monitored and controlled for TEST 3.

2.2 TEST 4 - 2 1/4 Cr-1 Mo STEEL SPECIMENS IN 1100 F HIGH OXYGEN (300 ppm) SODIUM

The sodium phase of TEST 4 at MSAR has been completed and the operation of Loop 2 suspended.

The sodium phase of TEST 4 was completed on January 2, 1964. At that time all test units were drained. TEST 6, the next test scheduled for Loop 2, also involves Cr-Mo specimens in sodium with a low oxide (30 ppm) content. Therefore, after draining the units, the main system was cold trapped for several days to lower the oxide concentration in this loop. The system was then drained. The cold trap was drained and washed out.

Several valve bellows that had failed during TEST 4 were replaced. In some instances, the corrosion on the valve body made it necessary to replace the complete valve. The system is being made ready for TEST 6. This involves calibration of instruments and general repairs to the system.

The progress schedule for TEST 4 is shown below. The operation history of Loop 2 (TEST 4), including estimated completion dates, is shown graphically in Fig. 1.

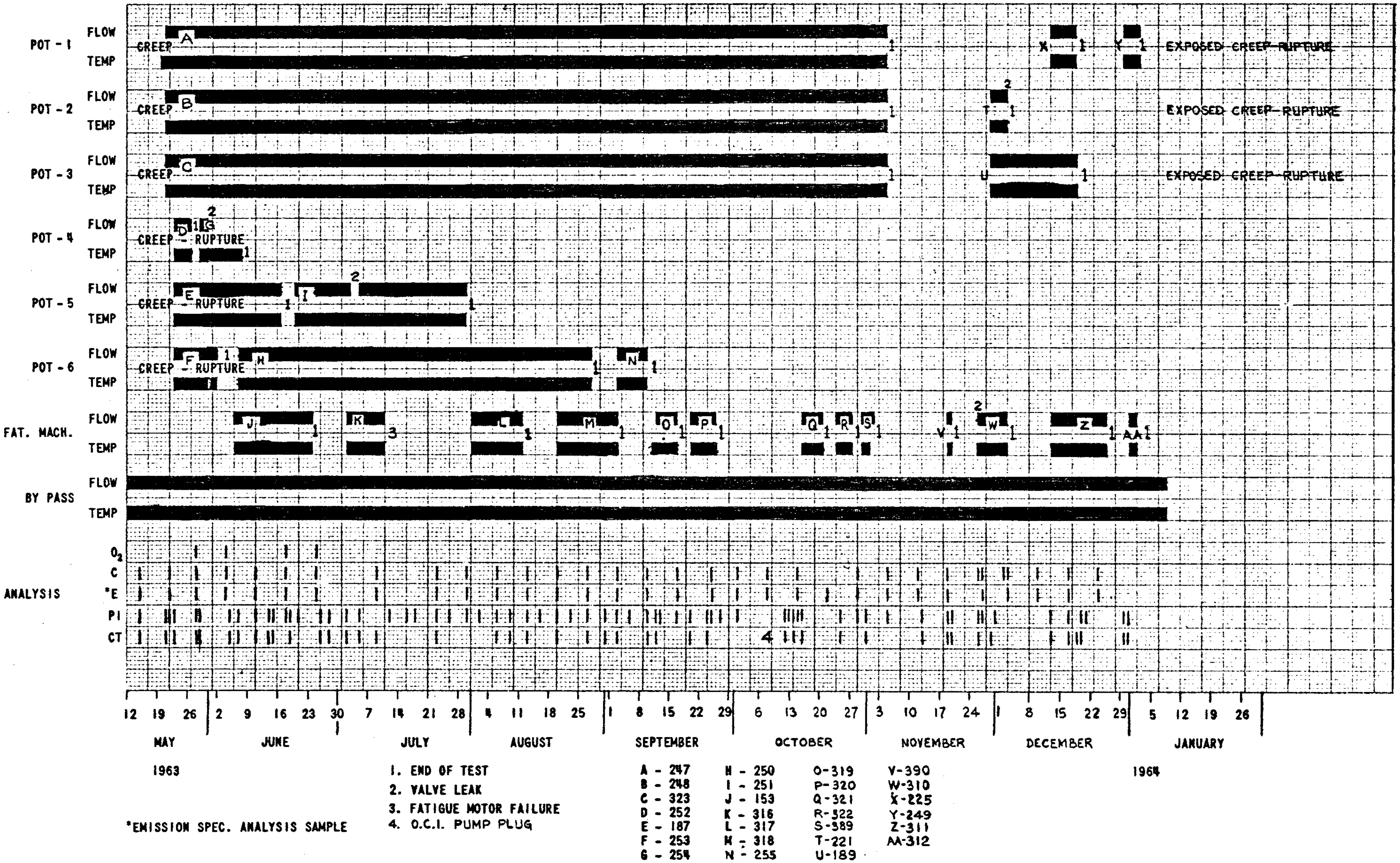
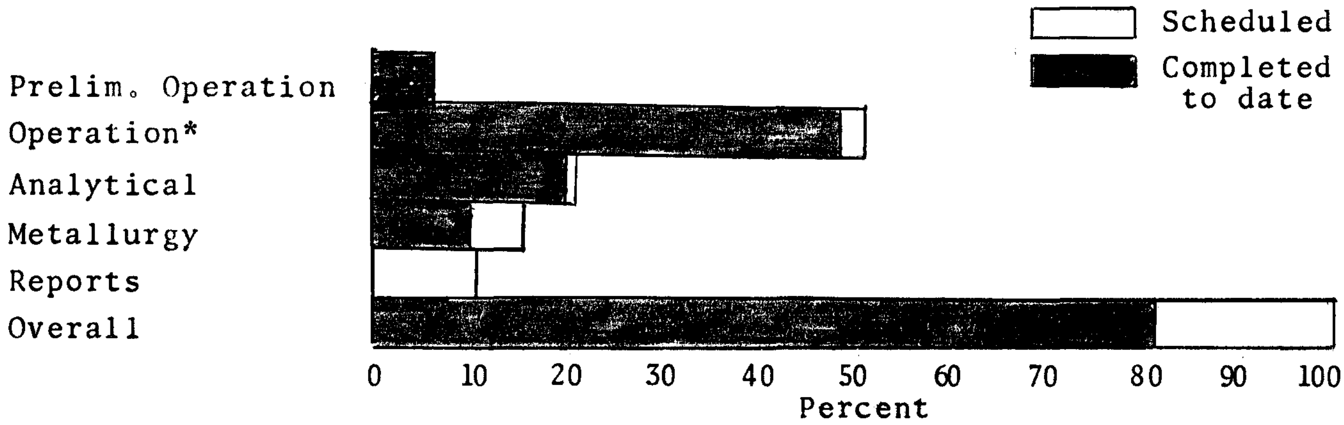


FIG. 1 - OPERATIONAL HISTORY OF LOOP 2 - TEST 4 (Cr-Mo TEST SPECIMENS)



* Based on scheduled hours of testing at MSAR and the University of Michigan.

PROGRESS SCUEDULE - TEST 4

3. TEST RESULTS

3.1 TEST 1 - STAINLESS STEEL (316) SPECIMENS IN 1200 F, LOW (30 ppm) OXYGEN SODIUM, AIR AND HELIUM

All phases of TEST 1 have been completed. Preliminary conclusions and tabulated data were reported in Progress Report 34.

A topical report on TEST 1 is nearing completion and is expected to be completed during the next report period.

3.2 TEST 2 - 2 1/4 Cr-1 Mo STEEL SPECIMENS IN 1100 F, LOW (30 ppm) OXYGEN SODIUM, AIR AND HELIUM

All phases of TEST 2 have also been completed with preliminary conclusions and tabulated data appearing in Progress Report 34.

Upon completion of the topical report on TEST 1, a similar report will be initiated for TEST 2.

3.3 TEST 4 - 2 1/4 Cr-1 Mo STEEL FATIGUE RUNS

All fatigue runs scheduled for TEST 4 have been completed.

Table 1 shows the tabulated results of the completed fatigue tests. Fig. 2 shows the averaged results of TEST 4 compared with the results obtained from TEST 2. These results show good agreement between TEST 2 and TEST 4 indicating no difference in fatigue properties of 2 1/4 Cr-1 Mo in 1100 F sodium with high or low oxygen.

3.4 TEST 4 - 2 1/4 Cr-1 Mo STEEL CREEP-RUPTURE RUNS

The creep-rupture runs on original material have been completed.

Runs in helium on specimens exposed for 4000 hours to 1100 F high oxide (300 ppm) sodium have been initiated. These tests are being conducted by the University of Michigan. One specimen at a load of 13,720 psi failed after 18.0 hours, while a second specimen at a load of 12,000 psi failed after 68.3 hours. A third specimen at a load of 10,000 psi is still in progress after 480 hours of testing.

The results of the runs in the high oxide sodium environment are shown in Table 2 and are plotted in Fig. 3 with curves from TEST 2 for comparison. The elongations are shown as a function of time in Fig. 4. The results of the creep-rupture tests in high oxygen sodium show a consistently shorter life of both the exposed and original material than in low oxygen sodium, which may be attributed to a corrosion layer observed on the creep-rupture specimens from TEST 4.

The results of the runs in helium are also shown in Table 2 and are plotted in Fig. 3. The exposed specimens are showing shorter rupture times in helium than the equivalent runs in sodium, but it is too early to attempt any type of interpolation.

3.5 TEST 4 - 2 1/4 Cr-1 Mo CREEP RUNS

The determination of the minimum creep rates of three 2 1/4 Cr-1 Mo specimens at loads of 7000 psi, 6000 psi and 4500 psi is complete. A fourth specimen (a duplication of the original 7000 psi run) was terminated after approximately 1900 hours. The minimum rates vs time for these runs are shown in Figs. 5, 6 and 7, and a curve of minimum rate vs stress is shown in Fig. 8.

The original 7000 psi run was not consistent with data from the rest of the runs. Examination of the test rig, upon removal of the specimen, revealed a deposit of sodium oxide between the pull rod and the bellows which would have influenced the stress being applied to the specimen.

TABLE 1 - FATIGUE TEST DATA SUMMARY
2-1/4 Cr-1 Mo STEEL - TEST 4 - HIGH OXIDE SODIUM

Specimen No.	Condition	Specimen Thickness in.	% Cyclic Strain	Cycles to Failure	Time at Temp. Hrs
153	High Oxide Na-1100 F	0.0672	.560	63,270	405.5
317	High Oxide Na-1100 F	0.0670	.558	45,890	284.5
318	High Oxide Na-1100 F	0.0655	.545	48,551	333.5
319	High Oxide Na-1100 F	0.0655	.969	12,612	134.0
320	High Oxide Na-1100 F	0.0655	.969	15,295	142.0
321	High Oxide Na-1100 F	0.0655	.969	9,500	121.0
322	High Oxide Na-1100 F	0.0655	2.1	3,150	70.5
389	High Oxide Na-1100 F	0.066	2.1	1,250	20.0
390	High Oxide Na-1100 F	0.066	2.1	2,650	64.0
307	Helium - 1100 F (PE)	0.0667	2.1	1,545	22.0
308	Helium - 1100 F (PE)	0.0668	2.1	1,221	16.0
161	Helium - 1100 F (PE)	0.0672	2.2	1,118	8.0
164	Air - 1100 F (PE)	0.0672	2.2	295	15.0
165	Air - 1100 F (PE)	0.0670	2.1	534	4.0
309	Air - 1100 F (PE)	0.0668	2.1	276	2.5
310	High Oxide Na-1100 F (PE)	0.0665	2.1	3,121	90.0
311	High Oxide Na-1100 F (PE)	0.0668	2.1	1,300	140.0
312	High Oxide Na-1100 F (PE)	0.0665	2.1	2,325	37.5

(PE) - Pre-exposed to 1100 F sodium with 300 ppm oxygen for 400 hrs.

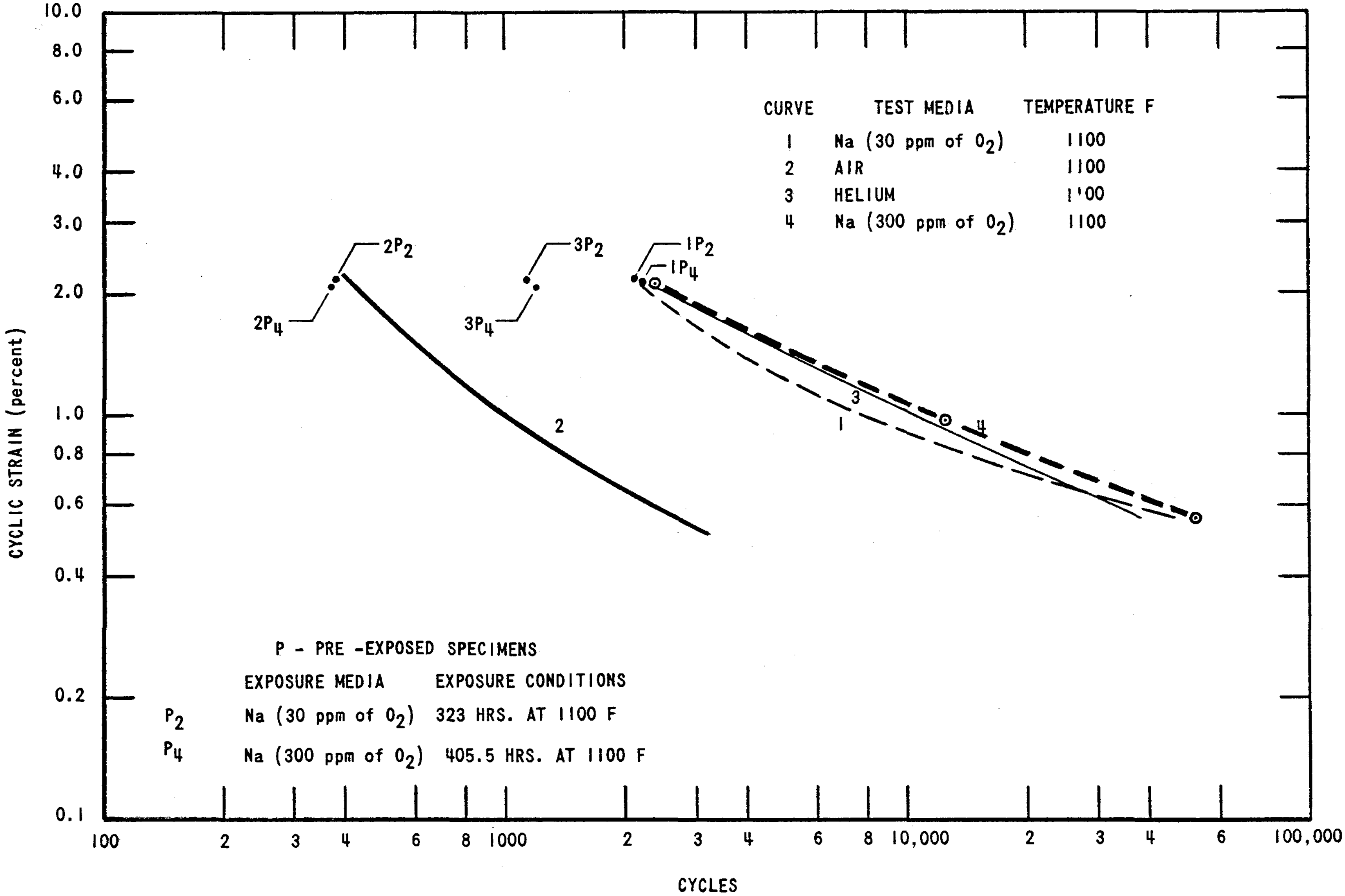


FIG. 2 - FATIGUE CURVES OF AVERAGED DATA FOR 2 1/4 Cr-1 Mo CARBON STEEL FATIGUE TESTS
(Data points for current TEST 4 are shown as ⊙)

TABLE 2 - CREEP-RUPTURE TEST DATA SUMMARY
2-1/4 Cr-1 Mo STEEL - TEST 4 - HIGH OXIDE SODIUM

Specimen No.	Condition	Stress Psi	Elong %	Reduction of Area %	Rupture Time (Hrs)
252	High Oxide Na-1100 F	18,000	73	38	48.5
254	High Oxide Na-1100 F	16,000	60	50	(1)234.4
253	High Oxide Na-1100 F	14,000	66	52	240.6
187	High Oxide Na-1100 F	12,000	51	42	588.7
251	High Oxide Na-1100 F	10,500	.4	16	(2)973.6
250	High Oxide Na-1100 F	9,500	(3)	17	1846.6
255	High Oxide Na-1100 F	16,000		55	141.5
221	High Oxide Na-1100 F (PE)	14,000	60	44	84.9
225	High Oxide Na-1100 F (PE)	12,000	36	25	105.4
189	High Oxide Na-1100 F (PE)	10,000	31	23	353.8
249	High Oxide Na-1100 F (PE)	14,000	54	40	42.8
222	Helium-1100 F (PE)	13,720	41	45	18.0
188	Helium-1100 F (PE)	12,000	32	32	68.3
223	Helium-1100 F (PE)	10,000			(480)*IP

(1) Loss of flow after 65 hours - Temperature gradient across specimen
*IP Test in progress
(2) Loss of flow for 56 hours - Temperature gradient across specimen
(3) Broke at gage mark - elongation not available
(PE) Exposed 4000 hours to 1100 F-High Oxide (300 ppm) sodium

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CALLERY, PA., U.S.A.
SEPTEMBER 3, 1963

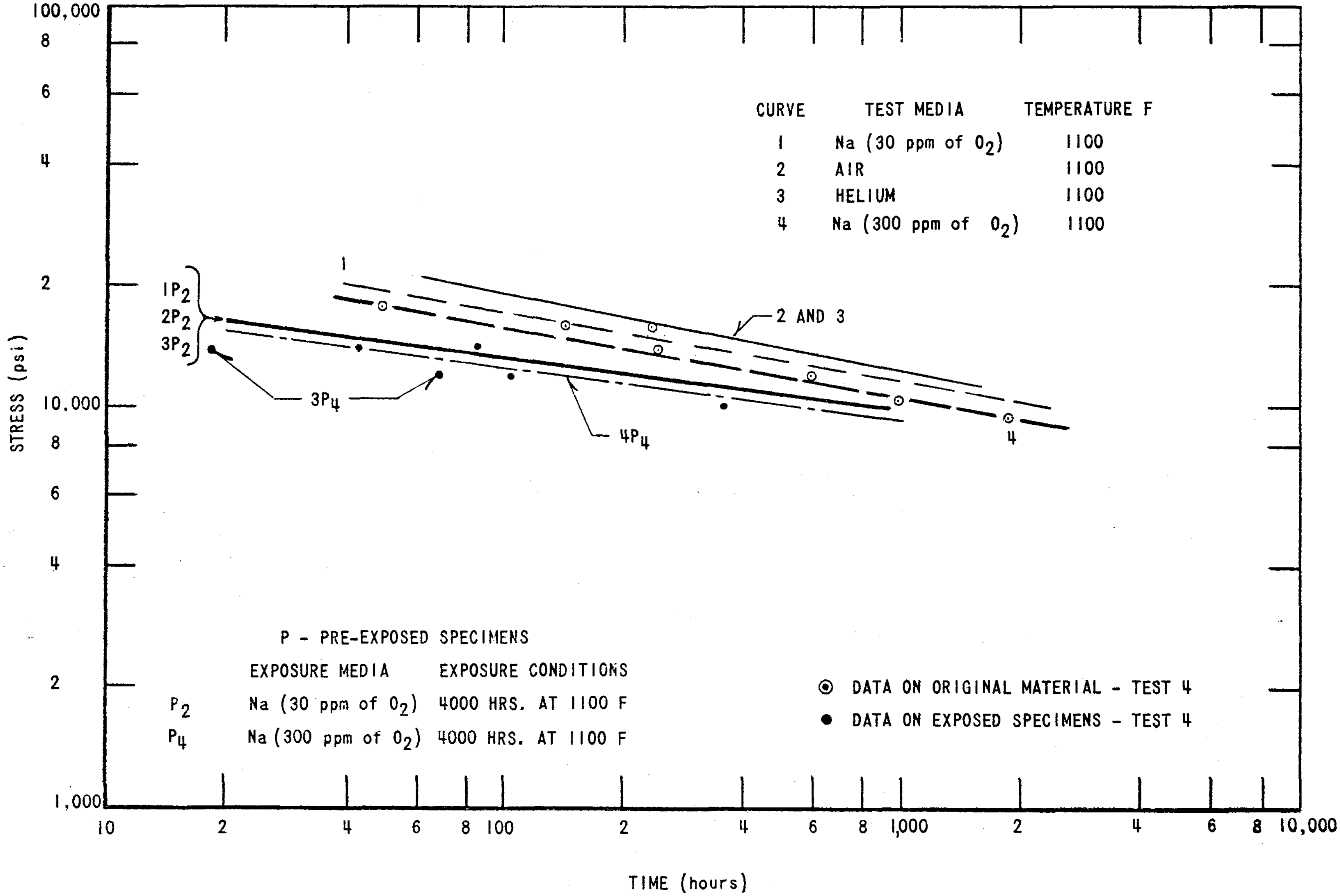

FIG. 3 - CREEP TO RUPTURE OF 2 1/4 Cr-1 Mo CARBON STEEL SPECIMENS

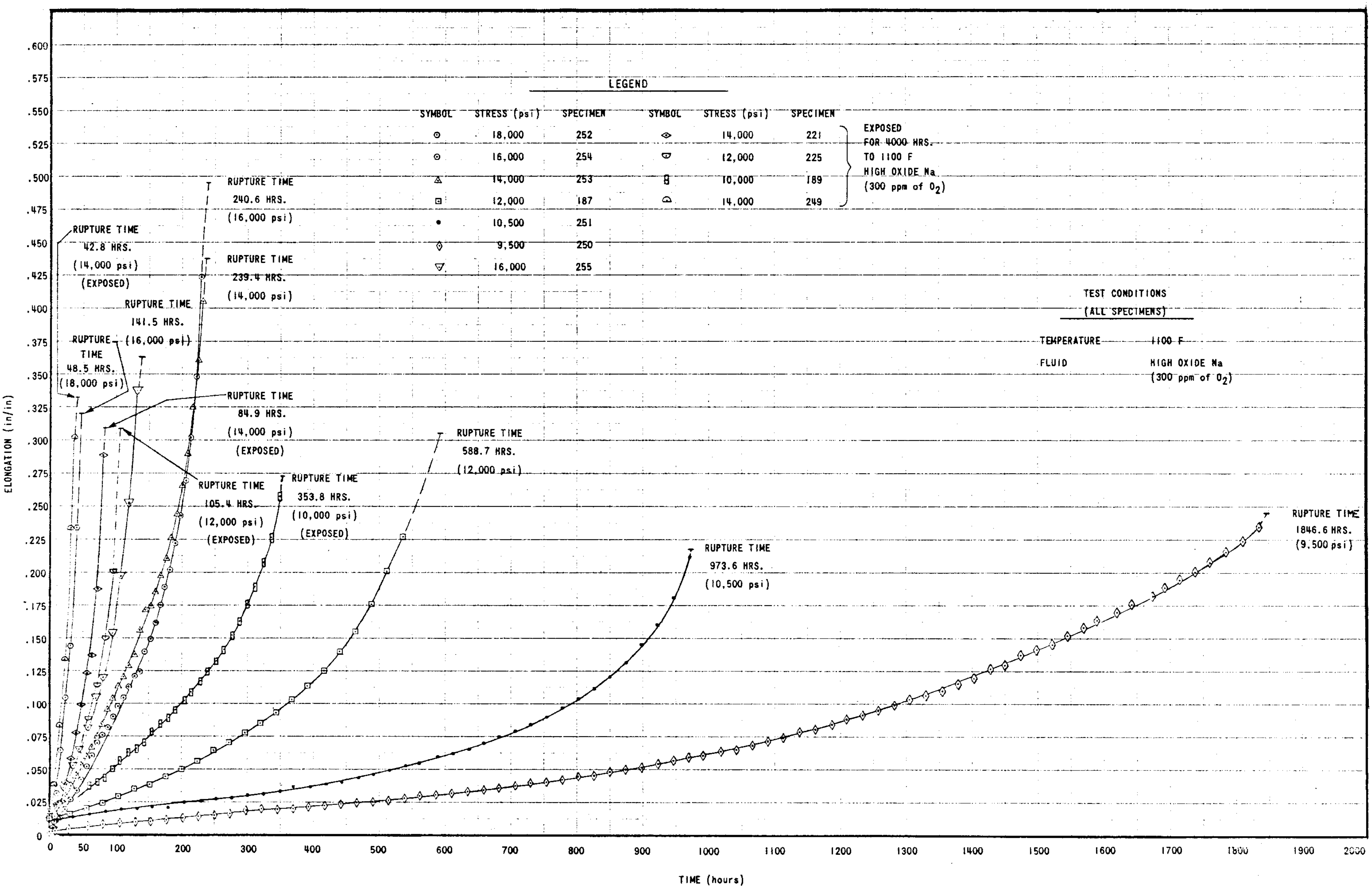

FIG. 4 - ELONGATION OF 2 1/4 Cr-1 Mo STEEL - CREEP RUPTURE SPECIMENS

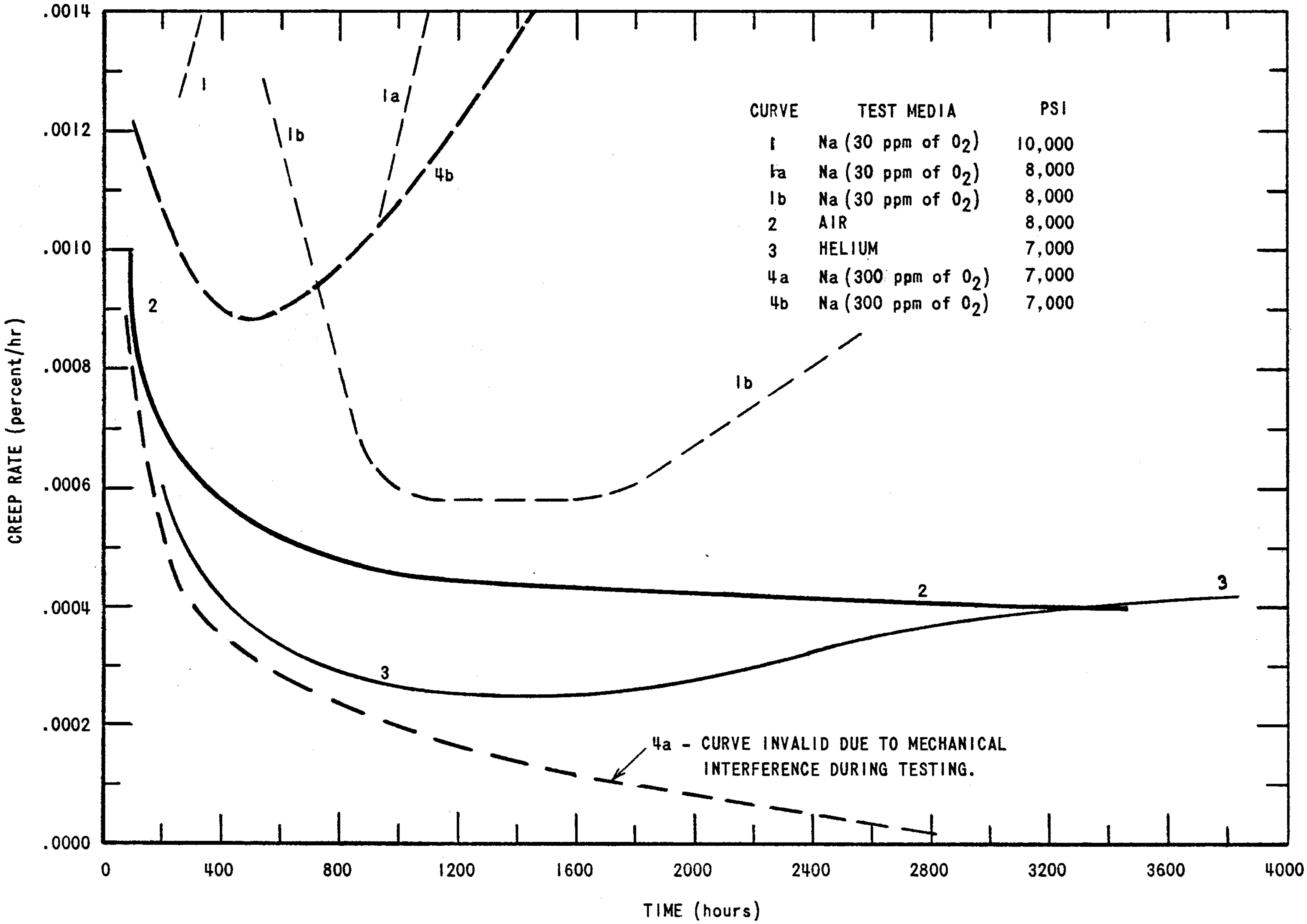

FIG. 5 - CREEP RATES, 2 1/4 Cr-1 Mo CARBON STEEL - 1100°F

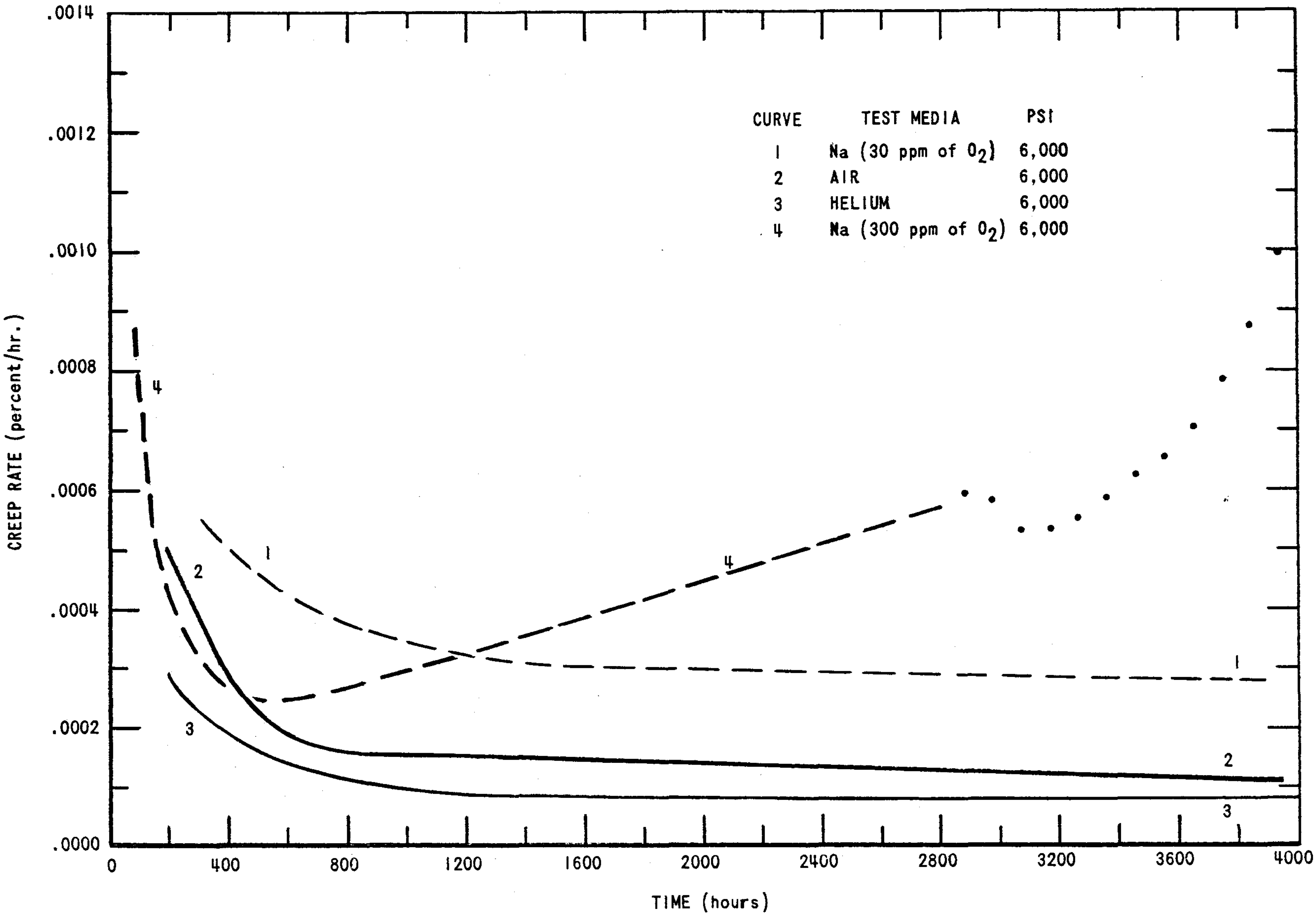

FIG. 6 - CREEP RATES, 2 1/4 Cr-1 Mo CARBON STEEL - 1100°F

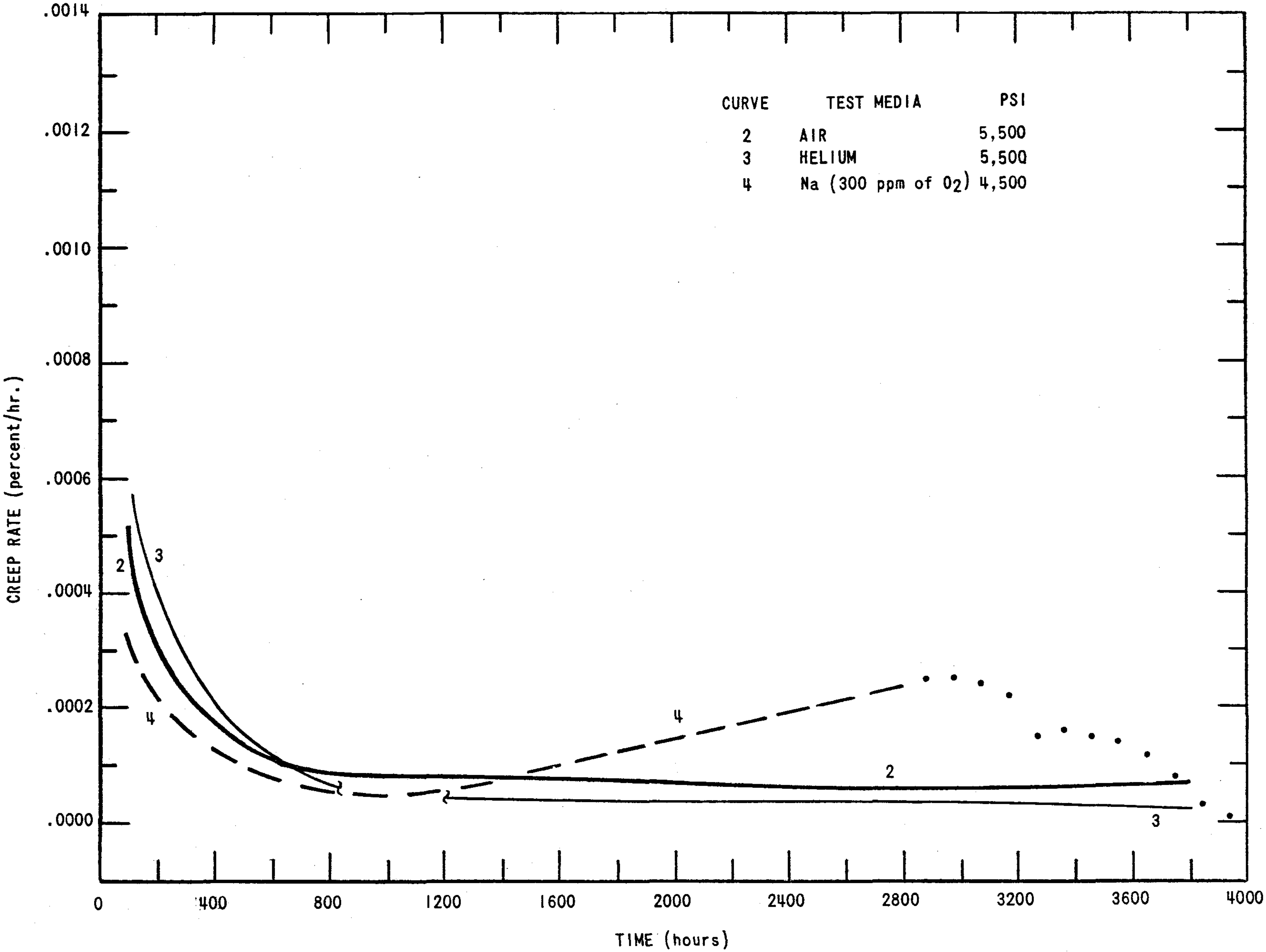

FIG. 7 - CREEP RATES, 2 1/4 Cr-1 Mo CARBON STEEL - 1100°F

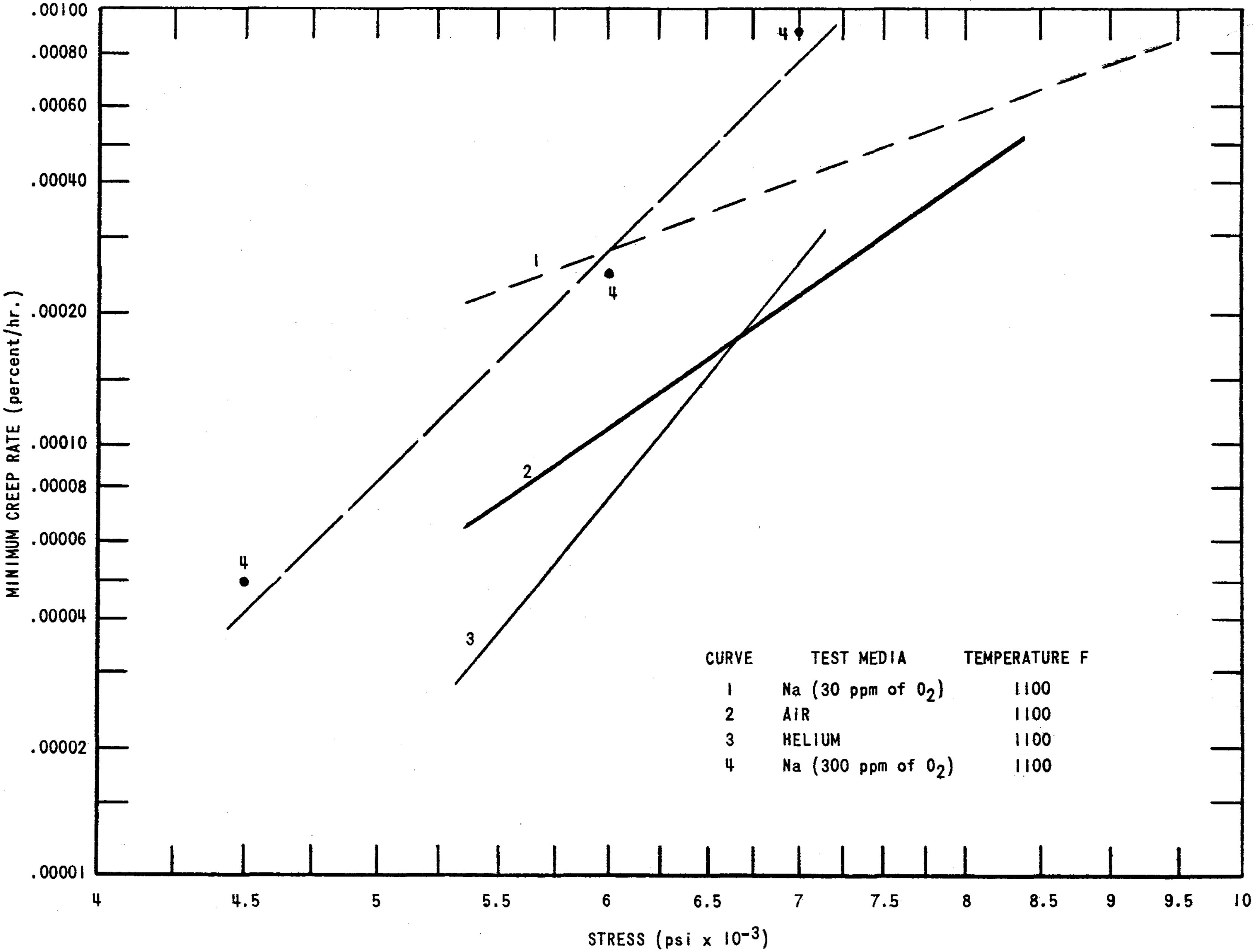

FIG. 8 - MINIMUM CREEP RATE vs STRESS, 2 1/4 Cr-1 Mo CARBON STEEL SPECIMENS

The last 1200 hours of testing of the 6000 and 4500 psi runs indicated that some interference was developing in these units also. However, the minimum creep rates had already been reached on these units and it is believed this interference had no effect on these results. Post test examination of these units showed indications of sodium deposits between the pull rods and bellows.

The creep tests show that the creep rate was lower in 1100 F sodium with high oxygen than with low oxygen. However, the creep rate is still higher in sodium than in air or helium.

3.6 TEST 4 - 2 1/4 Cr-1 Mo TENSILE RUNS

Specimens having been exposed to 1100 F high oxide (300 ppm) sodium for 4000 hours have been sent to the University of Michigan for tensile testing in helium.

The partial results of three tests completed to date are shown in Table 3.

4. METALLURGICAL RESULTS

4.1 TEST 2

Selected Cr-Mo stress-rupture specimens that had been exposed to 1100 F sodium in TEST 2 were sectioned for carbon analysis. Table 4 shows the results of carbon analysis of sections from within the gauge length. Decreasing carbon content with increasing exposure time is seen from the data.

4.2 TEST 3

Failure of the plugging meter valve bellows occurred in the preliminary operations of TEST 3. This valve was exposed to the sodium stream only during oxygen determination runs and was also used throughout TEST 1. The bellows design in this valve has a history of failures throughout this program due to being over-stressed.

Carbon analysis of the foil constituting the portion of the bellows exposed to sodium resulted in a value of 0.57% carbon. It is concluded that the bellows failed as a result of carbon embrittlement under a high stress.

TABLE 3 - TENSILE TEST DATA SUMMARY
2-1/4 Cr-1 Mo STEEL - TEST 4 - HIGH OXIDE SODIUM

Specimen No.	Condition	Tensile Stress (Psi)	0.2% Offset Yield Strength (Psi)	Elong %	Reduction of Area %
124	Helium-1100 F (PE)	23,800			
125	Helium-1100 F (PE)	20,620			
126	Helium-1100 F (PE)	20,740			

(PE) Exposed 4000 hours to 1100 F high oxide (300 ppm) sodium.

TABLE 4 - CARBON CONTENT OF Cr-Mo RUPTURE SPECIMENS DURING TEST 2

Specimen No.	Stress (KPSI)	Rupture Time (hrs)	Avg. Carbon (ppm)
Original	-	-	810
183	14.0	299	681
184	12.0	1116	567
185	10.0	2107	538
Pre-exposed 4000 hours in 1100°F sodium			
196	14.0	72	429
212	10.0	952	332

4.3 TEST 4

Selected creep-rupture specimens that had been exposed to 1100 F sodium in TEST 4 were sectioned for carbon analysis, with the results as shown in Table 5. Fig. 9 shows the average carbon content of Cr-Mo specimens as a function of time and includes selected samples from TEST 2 (low oxygen) and TEST 4 (high oxygen). While only a portion of the specimens from each test has been analyzed for carbon, the results to date indicate more rapid decarburization with high oxygen, particularly within the first 1000 hours where there is significant scatter. More extensive carbon analyses are planned in the attempt to determine the effect of stress on decarburization.

5. ANALYTICAL RESULTS

5.1 LOOP SAMPLES

The results of sodium samples extracted from Loop 2 during TEST 4 for carbon analyses are listed in Table 6, and are presented graphically in Fig. 10. Results of emission spectrograph samples are shown in Table 7.

The carbon analyses from Loop 1 during the preliminary runs for TEST 3 are shown in Table 8 and graphically in Fig. 11. Emission spectrograph analyses for the same period are listed in Table 9. Oxygen analyses, along with plugging indicator runs taken during the month of January are shown below:

Date	Plugging Temp.-°F	Saturation Temp. by Analysis - °F
1/9/64	600	-
1/10/64	740	-
1/14/64	900	-
	940	730
1/16/64	-	210
1/20/64	-	210*
1/27/64	970	-
	680	-
1/28/64	-	440*
1/29/64	550	210*
1/30/64	535	325*
1/31/64	520	-

* Cold trapping

TABLE 5 - CARBON CONTENT OF Cr-Mo RUPTURE SPECIMENS DURING TEST 4

Specimen No.	Stress (KPSI)	Rupture Time	Carbon Content
Original	-	-	810
252	18.0	48.5	675
255	16.0	141.5	533
254	16.0	234.4	713
253	14.0	240.6	600
187	12.0	588.7	755
251	10.5	973.6	434
250	9.5	1847	398
Pre-exposed 4008 hrs in 1100°F sodium (200-300 ppm O)			
249	14.0	42.8	340
221	14.0	84.9	302
225	12.0	105.4	381
189	10.0	353.8	239

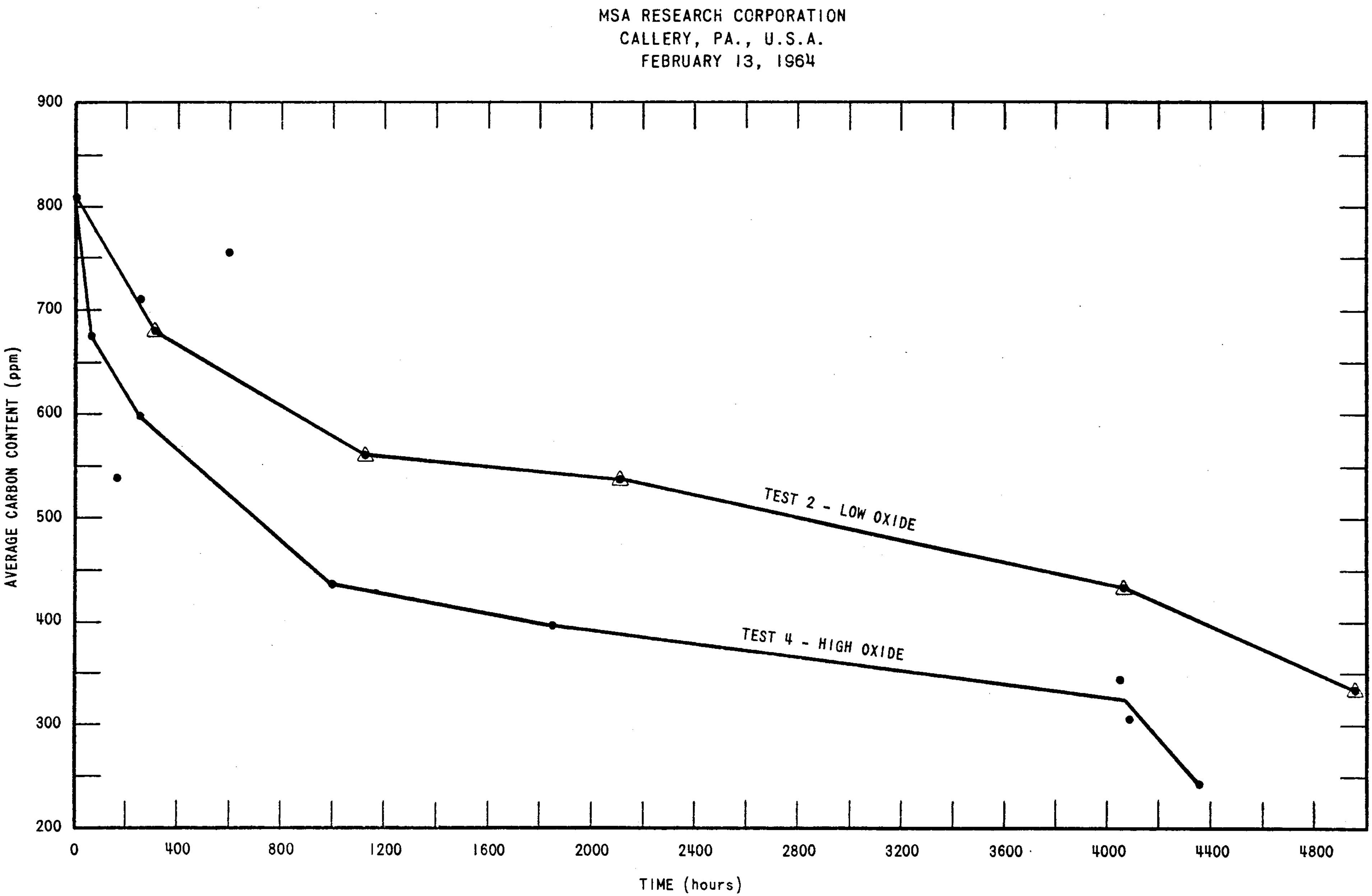

FIG. 9 - DECARBURIZATION OF 2 1/4 Cr-1 Mo STEEL IN 1100°F SODIUM

TABLE 6 - CARBON CONCENTRATION - LOOP 2
(TEST 4 - SODIUM 300 ppm O_2)

Date	Carbon Content (ppm C)
3-22-63	22
3-25-63	34
4-1-63	48
4-9-63	25
4-15-63	20
4-24-63	35
4-30-63	25
5-7-63	18
5-22-63	130
5-28-63	63
6-4-63	52
6-11-63	59
6-18-63	91
6-25-63	123
7-9-63	170
7-16-63	137
7-23-63	94
7-30-63	54
8-6-63	81
8-13-63	70
8-20-63	50
8-27-63	84
9-3-63	81
9-10-63	60
9-13-63	84
9-25-63	49
10-4-63	88 81
10-7-63	59 67
10-8-63	71
10-15-63	97
10-22-63	35
10-29-63	39

TABLE 6 - CARBON CONCENTRATION - LOOP 2
(TEST 4 - SODIUM 300 ppm O_2)
(Continued)

Date	Carbon Content (ppm C)
11-5-63	68
11-12-63	50
11-19-63	94
11-27-63	164
12-2-63	44 70
12-3-63	82
12-10-63	63
12-17-63	42
12-24-63	41

Table 7 - Chemical Analysis of Sodium From Test 4 (Cr-Mo Test Specimens) - in ppm

Date	Fe	B	Co	Mn	Al	Mg	Sn	Cu	Pb	Cr	Si	Ti[1]	Ni	Mo	V	Be	Ag	Zr	Li	Ca
3-22-63	<1	<5	<5	<1	<1	<1	<5	1	<5	<1	<10	<5	<1	<5	<1	<1	<1	<10	<1	2
3-25-63	5	<5	<5	<1	<1	<1	<5	2	<5	1	15	<5	<1	<5	<1	<1	<1	<10	<1	3
4-1-63	<1	<5	<5	<1	<1	<1	<5	1	<5	<1	15	<5	<1	<5	<1	<1	<1	<10	<1	<1
4-9-63	<1	<5	<5	<1	<1	<1	<5	<1	<5	<1	<10	<5	<1	<5	<1	<1	<1	<10	<1	<1
4-15-63	5	<5	<5	<1	<1	3	<5	2	<5	2	<10	<5	<1	<5	<1	<1	<1	<10	<1	20
4-24-63	<1	<5	<5	<1	<1	<1	<5	<1	<5	<1	10	<5	<1	<5	<1	<1	<1	<10	2	<1
4-30-63	<1	<5	<5	<1	2	3	<5	<1	<5	<1	<10	<5	<1	<5	<1	<1	<1	<10		6
5-7-63	1	<5	<5	<1	2	4	<5	<1	<5	<1	12	5	<1	<5	<1	<1	<1	<10		6
5-15-63	2	<5	<5	<1	1	2	<5	5	<5	<1	<10	20	<1	<5	<1	<1	<1	<10		6
5-22-63	3	<5	<5	1	1	2	<5	3	<5	<1	10	<5	<1	<5	<1	<1	<1	<10		5
5-28-63	2	<5	<5	1	2	5	<5	<1	<5	<1	11	<5	<1	<5	<1	<1	<1	<10		7
6-4-63	2	<5	<5	<1	1	3	<5	<1	<5	<1	10	<5	<1	<5	<1	<1	<1	<10		7
6-11-63	1	<5	<5	<1	2	2	<5	1	<5	<1	10	<5	<1	<5	<1	<1	<1	<10		3
6-18-63	<1	<5	<5	<1	2	1	<5	<1	<5	<1	10	<5	<1	<5	<1	<1	<1	<10		2
6-25-63	<1	<5	<5	<1	2	1	<5	<1	<5	<1	<10	<5	<1	<5	<1	<1	<1	<10		3
7-9-63	<1	<5	<5	<1	2	1	<5	<1	<5	<1	<10	<5	<1	<5	<1	<1	<1	<10		1
7-16-63	<1	<5	<5	<1	2	2	<5	<1	<5	<1	10	<5	<1	<5	<1	<1	<1	<10		2
7-23-63	2	<5	<5	<1	2	2	<5	<1	<5	<1	10	5	<1	<5	<1	<1	<1	<10		6
7-30-63	1	<5	<5	<1	1	1	<5	<1	<5	<1	10	<5	<1	<5	<1	<1	<1	<10		5
8-6-63	2	<5	<5	<1	2	5	<5	<1	<5	<1	12	<5	<1	<5	<1	<1	<1	<10		12
8-13-63	<1	<5	<5	<1	<1	2	<5	<1	<5	<1	<10	<5	<1	<5	<1	<1	<1	<10		2
8-20-63	<1	<5	<5	<1	<1	<1	<5	<1	<5	<1	<10	<5	<1	<5	<1	<1	<1	<10		1
8-27-63	1	<5	<5	<1	<1	1	<5	<1	<5	<1	<10	<5	<1	<5	<1	<1	<1	<10		2
9-3-63	<1	<5	<5	<1	<1	10-2	<5	<1	<5	<1	10-25	<5	<1	<5	<1	<1	<1	<10		4
9-10-63	<1	<5	<5	<1	<1	<1	<5	<1	<5	<1	15	25	<1	<5	<1	<1	<1	<10		2
9-17-63	<1	<5	<5	<1	<1	<1	<5	<1	<5	<1	100-15	<5	<1	<5	<1	<1	<1	<10		2
9-25-63	<1	<5	<5	<1	<1	<1	<5	<1	<5	<1	<10	<5	<1	<5	<1	<1	<1	<10		<1
10-1-63	<1	<5	<5	<1	<1	<1	<5	<1	<5	<1	<10	<5	<1	<5	<1	<1	<1	<10		<1
10-8-63	<1	<5	<5	1	<1	<1	<5	2	<5	<1	<10	<5	<1	<5	<1	<1	<1	<10		<1
10-15-63	<1	<5	<5	<1	<1	<1	<5	<1	<5	<1	<10	100	<1	<5	<1	<1	<1	<10		2
10-20-63[2]	4	150	<5	<1	25	1	<5	<1	<5	2	1500	125	<1	<5	<1	<1	<1	<10		2
10-29-63	1	<5	<5	<1	2	1	<5	<1	<5	<1	<90	100	<1	<5	<1	<1	<1	<10		2

TABLE 7 - CHEMICAL ANALYSIS OF SODIUM FROM TEST 4 (Cr-Mo TEST SPECIMENS) - in ppm
(Continued)

Date	Fe	B	Co	Mn	Al	Mg	Sn	Cu	Pb	Cr	Si	Ti(1)	Ni	Mo	V	Be	Ag	Zr	Li	Ca
11-5-63	1	<5	<5	<1	1	1	<5	1	<5	<1	<25	<5	<1	<5	<1	<1	<1	<10		2
11-12-63	1	<5	<5	<1	<1	<1	<5	2	<5	<1	<25	<5	<1	<5	<1	<1	<1	<10		2
11-19-63(2)	5	125	<5	<1	75	1	<5	2	<5	<1	250	>.1%	<1	<5	<1	<1	<1	<10		2
11-27-63	<1	<5	<5	<1	1	<1	<5	<1	<5	<1	<25	<5	<1	<5	<1	<1	<1	<10		1
12-3-63	2	<5	<5	<1	1	1	<5	<1	<5	<1	<25	10	<1	<5	<1	<1	<1	<10		3
12-10-63	<1	<5	<5	<1	<1	<1	<5	<1	<5	<1	<25	15	<1	<5	<1	<1	<1	<10		1

(1) Titanium sample buckets used.
(2) Sample contaminated with glass during preparation for analysis.

Note: Where duplicate shots of samples do not agree, both results are recorded.

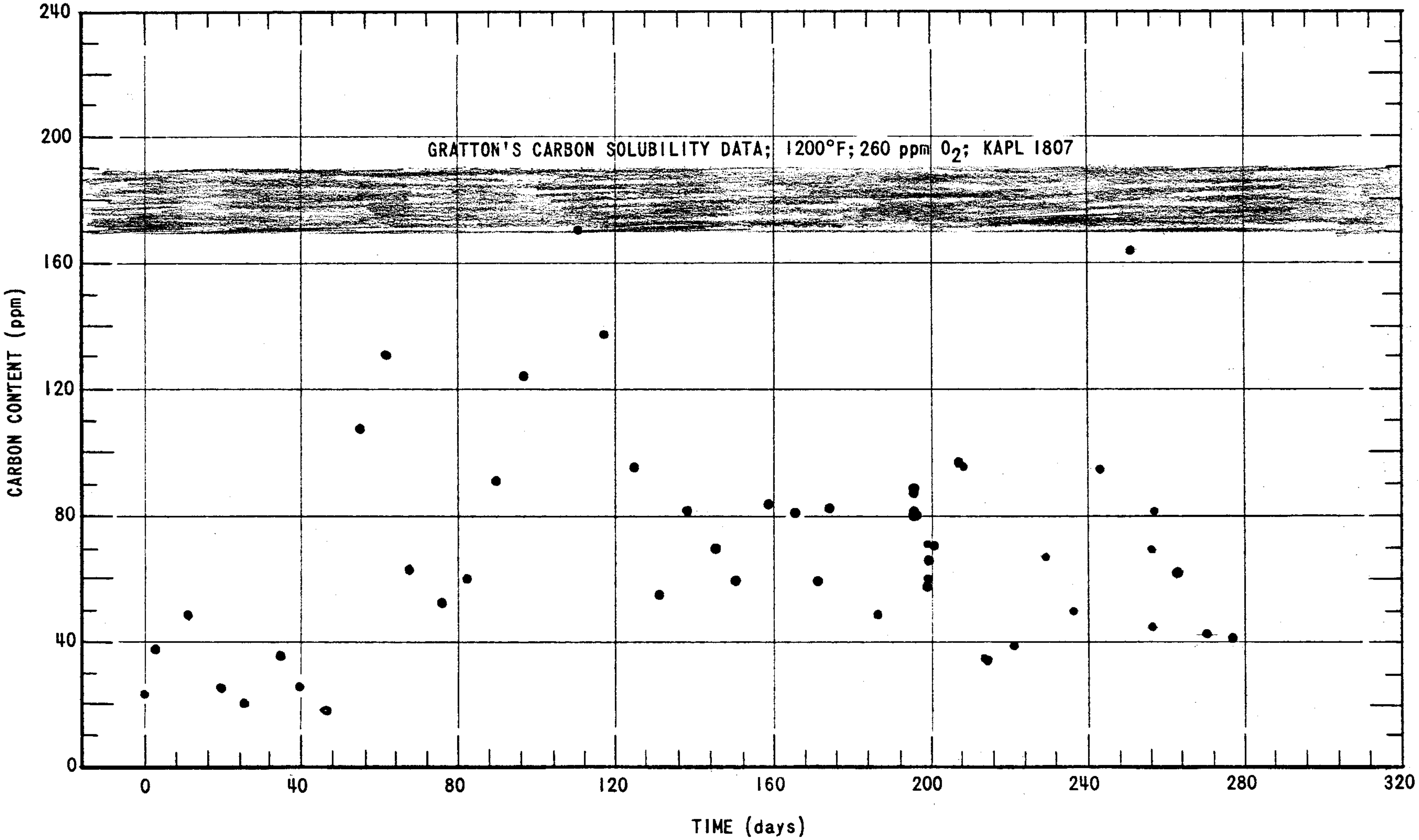

FIG. 10 - CARBON CONTENT, LOOP 2, TEST 4
(Cr-Mo TEST SPECIMENS)

TABLE 8 - CARBON CONCENTRATION - LOOP 1
TEST 3 - PRELIMINARY RUNS

Date	Carbon Content (ppm C)
6-17-63	77*
6-18-63	85
	232
	114
	88
6-19-63	88
	119
	113
6-25-63	52
6-28-63	228
	112
7-2-63	150
7-9-63	115
7-16-63	114
7-23-63	148
7-24-63	62
7-25-63	37
7-26-63	43
7-29-63	86**
7-30-63	76**
8-7-63	63
8-8-63	54
8-9-63	128
8-12-63	67
8-13-63	44
8-14-63	144
8-15-63	184
8-16-63	56
8-19-63	127
8-20-63	70
8-21-63	30
8-23-63	220
8-26-63	43
8-27-63	182

* Circulation through the carbon bed did not begin until 10-22-63

** Sampled at 600° F

TABLE 8 - CARBON CONCENTRATION - LOOP 1
(Continued)

Date	Carbon Content (ppm C)
8-28-63	160
	124 (cored sample)[1]
	185 (cored sample)[1]
	133 (cored sample)[1]
8-30-63	74
9-3-63	85
9-4-63	88
9-5-63	167
	99
	165 (cored sample)[1]
	144 (cored sample)[1]
9-6-63	125
9-9-63	94
9-10-63	288
Creep test pots drained	
9-12-63	83
9-13-63	92
	48
9-17-63	42
9-19-63	87
9-20-63	109
9-23-63	64
9-25-63	67
	71 (samples taken from low
	46 side of expansion tank)
9-26-63	30 (Exp. tank increased to 1220 F)
10-1-63	47
10-2-63	72
10-4-63	82

(1) These samples are explained in a later discussion

TABLE 8 - CARBON CONCENTRATION - LOOP 1
TEST 3 - PRELIMINARY RUNS
(Continued)

Date	Carbon Content (ppm C)	
10-7-63	50	
10-8-63	71	
10-11-63	77	
10-17-63	37	
	147	
10-18-63	52	
10-19-63	76	
	62	
10-21-63	90	
	80	
10-22-63	48	Started Circulation
	74	Through
	119	Carbon Bed
	89	
10-23-63	81	
	197	
	50	
	94	
10-24-63	94	
	45	
	138	
10-25-63	129	
	54	
	77	
	93	
10-26-63	46	
	65	
10-28-63	121	
	75	
10-29-63	60	
	41	
10-30-63	85	
	77	
10-31-63	104	
11-1-63	50	Stopped Circulation
	55	Through Carbon Bed
11-2-63	81	
	68	

TABLE 8 - CARBON CONCENTRATION - LOOP 1
TEST 3 - PRELIMINARY RUNS
(Continued)

Date	Carbon Content (ppm C)	
11-4-63	48	
11-5-63	44	
	82	
11-6-63	86	Started Circulation
	88	Through Carbon Bed
11-13-63	68	
	76	
11-14-63	131	
	50	
11-15-63	32	
	74	
11-18-63	52	
	47	
11-19-63	45	
	44	
11-20-63	63	
	48	
11-21-63	30	
	96	
11-22-63	86	
	35	
11-25-63	79	
11-26-63	77	
	138	
12-23-63	75	Stopped Circulation Through Carbon Bed
1-2-64	124	
1-15-64	90	Carbon bed operated inter-
	92	mittently from 1-10-64
1-16-64	64	
	56	
1-17-64	38	
	46	
1-20-64	53	
	96	

TABLE 8 - CARBON CONCENTRATION - LOOP 1
TEST 3 - PRELIMINARY RUNS
(Continued)

Date	Carbon Content (ppm C)
1-21-64	124 (Contaminated)
	47
1-22-64	35
	57
1-23-64	58
	33
1-27-64	33
	60
1-28-64	36
	126
1-29-64	41
	38
1-30-64	30
	51
1-31-64	42
	62

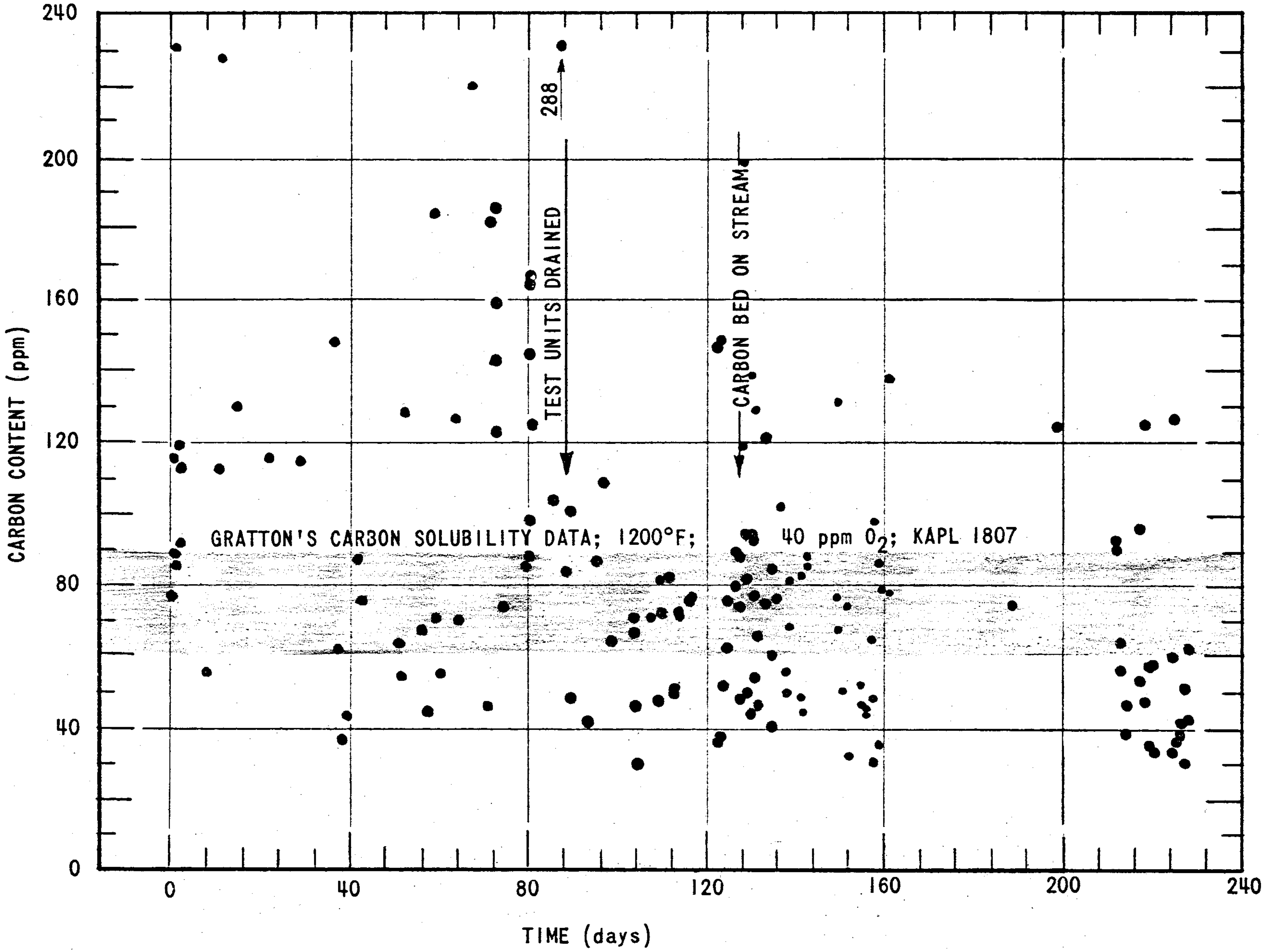

FIG. 11 - CARBON CONTENT, LOOP I, TEST 3 (PRELIMINARY OPERATIONS)

TABLE 9 - EMISSION SPECTROGRAPH ANALYSES - LOOP 1 (ppm)
TEST 3 - PRELIMINARY OPERATIONS

Date	Fe	B	Co	Mn	Al	Mg	Sn	Cu	Pb	Cr	Si	Ti	Ni	Mo	V	Bc	Ag	Zr	Sr	Ba	Ca
6-18-63	<1	<5	<5	<1	2	5	<5	2	<5	<1	<10	<5	<1	<5	<1	<1	<1	<10	<1		4
6-25-63	<1	<5	<5	<1	3	7	<5	<1	<5	<1	10	<5	<1	<5	<1	<1	<1	<10	<1		5
7-2-63	1	<5	<5	<1	2	4	<5	3	<5	<1	<10	<5	<1	<5	<1	<1	<1	<10	<1		4
7-9-63	<1	<5	<5	<1	3	3	<5	<1	<5	<1	<10	<5	<1	<5	<1	<1	<1	<10	<1		3
7-16-63	<1	<5	<5	<1	2	2	<5	3	<5	<1	<10	<5	<1	<5	<1	<1	<1	<10	<1		3
7-23-63	<1	<5	<5	<1	2	4	<5	<1	<5	<1	<10	<5	<1	<5	<1	<1	<1	<10	<1		5
7-30-63	<1	<5	<5	<1	2	3	<5	2	<5	<1	<10	<5	<1	<5	<1	<1	<1	<10	<1		5
8-7-63	1	<5	<5	<1	2	4	<5	<1	<5	<1	<10	<5	<1	<5	<1	<1	<1	<10	<1		6
8-13-63	1	<5	<5	<1	<1	1	<5	2	<5	<1	<10	<5	<1	<5	<1	<1	<1	<10	<1	<3	2
8-20-63	<1	<5	<5	<1	1	<1	<5	<1	<5	<1	<10	<5	<1	<5	<1	<1	<1	<10	<1	<3	1
8-27-63	1	<5	<5	<1	2	<1	<5	<1	<5	<1	<10	<5	<1	<5	<1	<1	<1	<10	<1	<3	1
9-3-63	3	<5	<5	<1	<1	2	<5	<1	7	2	10	25	<1	<5	<1	<1	<1	<10	<1	<3	4
9-10-63	<1	<5	<5	<1	1-2	1-4	<5	<1	<5	<1	<10-15	<5	<1	<5	<1	<1	<1	<10	<1	<3	4
9-17-63	<1	<5	<5	<1	<1	<1	<5	<1	<5	<1	<10	10	<1	<5	<1	<1	<1	<10	<1	<3	3
9-25-63	<1	<5	<5	<1	1	<1	<5	1	<5	<1	<10	<5	<1	<5	<1	<1	<1	<10	<1	<3	1
10-1-63	1	<5	<5	<1	<1	<1	<5	5-2	<5	<1	<10	<5	<1	<5	<1	<1	<1	<10	<1	<3	<1
10-8-63	1-5	<5	<5	<1	1	<1	<5	2	<5	<1	<10	<5	<1	<5	<1	<1	<1	<10	<1	<3	<1
10-22-63	2	<5	<5	<1	2	<1	<5	<1	<5	<1	<10	<25	<1	<5	<1	<1	<1	<10	<1	<3	2
10-29-63	3	<5	<5	<1	1	<1	<5	<1	<5	<1	<10	<25	<1	<5	<1	<1	<1	<10	<1	<3	2
11-5-63	<1	<5	<5	<1	2	<1	<5	<1	<5	<1	<10	<25	<1	<5	<1	<1	<1	<10	<1	<3	2
11-12-63	<1	<5	<5	<1	2	1	<5	1	<5	<1	<25	<5	<1	<5	<1	<1	<1	<10	<1	<3	3
11-19-63	<1	<5	<5	<1	1	<1	<5	1	<5	<1	<25	5	<1	<5	<1	<1	<1	<10	<1	<3	1
11-26-63	<1	<5	<5	<1	1	1	<5	<1	<5	<1	<25	<5	<1	<5	<1	<1	<1	<10	<1	<3	2

Note: Where duplicate shots of samples do not agree, both results are recorded.

These runs illustrate the disagreement between the plugging indicator saturation temperature and the corresponding saturation temperatures obtained from sample analysis for oxygen. It is believed that the plugging indicator is reflecting carbon concentration rather than oxygen concentration under these conditions.