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

MASTER

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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Callery, Pennsylvania

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PROGRESS REPORT 40
December 1963

to

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Chicago Operations Office
Lemont, Illinois

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Physical Properties of Materials

January 21, 1964

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Progress Report 40

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 tests 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) SPECIMENS IN 1200 F, HIGH CARBON SODIUM

Upon completion of the preliminary tab tests, the initiation of the main test was attempted several times but operational difficulties, climaxed with the plugging of the OCI system, forced the delay of this test. Start-up is now scheduled for the second week in January.

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

The creep, creep-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 1200 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.

Progress Report 40

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 is to be issued in the near future. The examination of specimens generated from TEST 2 is nearing completion.

TESTS 3 and 4 are in progress. These tests 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. Preliminary operation with metal tabs for TEST 3 has been completed and examination of these specimens is in progress. Difficulty in the operation of the oxide control and indicating circuit has delayed the initiation of TEST 3 but it is expected that this test will begin during the next report period. The sodium phase of TEST 4 has been completed and metallographic examination of the specimens is in progress. After receiving funds for the next contract period, MSAR sent TEST 4 specimens exposed to sodium to the University of Michigan for tensile and creep-rupture tests in helium. Preparation of Loop 2 for TEST 6 is now in progress.

2. OPERATION

2.1 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.

2.2 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. At this time, it is scheduled for completion by January 31, 1964.

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

The operation in Loop 1 with metal tabs of Armco Iron, 2 1/4 Cr-1 Mo, 316 ss and 304 ss for 1, 4, 6, 11, and 20 days has been completed.

The sequence of events during the operation of Loop 1 for this report period was:

1. From December 2 to December 13, repairs and additions were made to the loop. The No. 2 creep machine, which was plugged and could not be drained, was removed from the system. Plugs were found in the inlet and drain valves which necessitated their removal. The plugs were removed by washing with alcohol. The inlet line (flexible hose) was plugged and this line was replaced.

   Sections of the system piping were removed for metallurgical examination since the tab test indicated high carburization rates.

   The main heater thermocouple well was relocated to give better control during operation of the carbon bed circuit.

2. On December 13, the system was charged and cold trapping initiated.

3. Line heaters on the carbon circuit failed while establishing flow through the carbon pot on December 16th and 19th.

4. On December 19, creep-rupture specimens were installed in creep machines 4 through 6.

5. On December 20, a faulty probe caused over-charging of a test unit, delaying the start of the creep-rupture tests. However, flow was established through the carbon bed and cold trapping of the

system was begun to obtain TEST 3 conditions. Additional creep-rupture specimens were installed in creep machines 1 through 3 to initiate the rupture tests, since the originally scheduled test units had to be cleaned from the spillage due to overcharging.

6. On December 23, the OCI system plugged, postponing start of the tests.

7. Since operation of the system is with sodium saturated with carbon, it is probable that the plug in the OCI system is due to some interaction of the carbon. To locate, remove and analyze this plug, several components of the OCI system were isolated and then carefully drained and washed. Indications of plugs were observed in the shell side of the economizer and the inlet isolation valve. Washings from these areas are being analyzed. The OCI system has been washed, dried and returned to the loop.

   Since test conditions cannot be maintained without the use of the OCI system, initiation of TEST 3 has been delayed until it is certain the OCI circuit is operable.

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

Operation of Loop 2, in which TEST 4 is being conducted, continued. Testing of the exposed creep-rupture specimens was completed as were the tests on the exposed fatigue specimens. Operation of the sodium phase of TEST 4 ended with the termination of the fourth creep run, a duplication of an original creep run.

The sequence of events during this report period for Loop 2 was as follows:

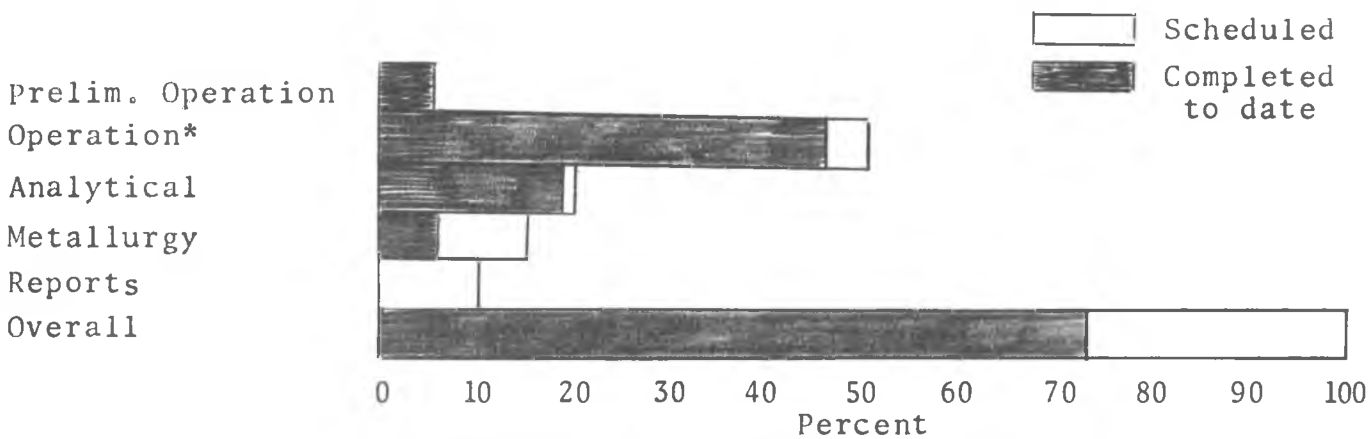
1. Testing of exposed creep-rupture and fatigue specimens continued.

2. On December 9, it was discovered that the OCI pump bus bars had broken loose from the pumping section. It was felt that this was due to overheating in trying to establish OCI flow. This failure was not detected until the pump was removed for washout. A new, larger capacity pump was installed in a new location such that the pump discharge pressure was against the cold trap rather than the suction pressure.

3. Both the inlet and drain valves to the fatigue machine were found to be plugged on December 19. This section of piping was removed, washed, and then replaced.

4. On January 2, 1964, all tests were completed and machines drained. The system will be cold trapped down to 250 F prior to draining for repairs and alterations in preparation for TEST 6.

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



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

PROGRESS SCHEDULE - TEST 4

2.4.1 2 1/4 Cr-1 Mo Steel Fatigue Runs

All fatigue runs scheduled for TEST 4 have been completed.

The two specimens exposed for 405 hours to 1100 F sodium and then fatigue tested in high oxide (300 ppm) sodium at the high cyclic strain condition failed in 1300 and 2325 cycles respectively.

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.

2.4.2 2 1/4 Cr-1 Mo Steel Creep-Rupture Runs

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

The second and third runs of specimens exposed 4000 hours to 1100 F high oxide (300 ppm) sodium were completed during this report period. The specimen at a stress of 10,000 psi failed after 354 hours and the specimen at a load of 12,000 psi failed after 105 hours. Since there was some scatter in this data, a fourth specimen at a load of 14,000 psi was run. This failed after 43 hours. This completes the creep-rupture sodium phase of TEST 4.

These results 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 later observed on the creep-rupture specimens from TEST 4.

Specimens having been exposed 4000 hours to 1100 F high oxide (300 ppm) sodium have been sent to the University of Michigan for testing in helium. No results have been obtained.

2.4.3 2 1/4 Cr-1 Mo Creep Runs

The determination of the minimum creep rates of three 2 1/4 Cr-1 Mo specimens has been completed. These are at loads of 7000 psi, 6000 psi and 4500 psi. A fourth specimen (a duplication of the original 7000 psi load 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.

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.

2.4.4 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. No results have been obtained.

3. METALLURGICAL EFFECTS

3.1 CARBURIZATION OF 316 STAINLESS - TEST 3

Weight change and carbon analysis data shown in the last monthly report indicated rapid carburization of stainless steels when exposed to 1200 F sodium saturated with carbon. Figs. 9, 10, and 11 show the surfaces of the 316 ss tabs exposed to carbon-saturated sodium for respective durations of 6, 11 and 20 days. The adjusted exposure times for these samples are 2, 7, and 16 days. The measured case depths are respectively 0.8, 1.4 and 2.7 mils. Grain boundary carbide precipitation is evident to a depth approximately twice as great as the heavily carburized case.

The results obtained by exposure of stainless steel tabs (304 and 316) to 1200 F sodium are very similar to those reported by Anderson and Sneesby[1] on 304 in terms of case depth as a function of time.

After completion of the foil tests, one inch pipe specimens (Type 316) that had originally been utilized in construction of the loop were sectioned from the inlet and outlet manifolds to the test units. Test pipe nipples (1 in. IPS, 316) that had been inserted in the inlet and outlet portions just prior to TEST 3 operation were also removed.

Fig. 12 is a photomicrograph of a test nipple sectioned from the outlet side, while Fig. 13 is a section from the original piping used in loop construction which had also been removed at the outlet to the test pots. The case thickness on the test nipple, which had been exposed to carbon-saturated sodium for approximately 36 days, approximates that case apparent on the 20-day tab (Fig. 11). The case on the pipe in Fig. 13, which had been exposed to "low" carbon 1200 F sodium for approximately 6500 hours prior to TEST 3, is approximately twice as thick as that on the test nipple.

The internal surface of test nipples and loop pipe specimens were reamed to obtain surface sections for carbon analysis. Table 3 shows the results of carbon analysis. Higher surface carbon contents and deeper penetration are evidenced in sections originally present in the loop. In the case of the test nipples, as well as original loop sections, carburization has proceeded to a greater

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1. NAA-SR-5282 (September 1960).

extent at the outlet sections as compared to inlet sections. Examination of the data presented by Anderson and Sneesby suggests large increases in carburization rate with small temperature difference. Temperature data of these samples was not measured. Data will be obtained during subsequent operations.

In view of the results of the tab tests, the operation of the loop with carbon-saturated sodium at 1200 F for full completion of TEST 3 as originally planned (estimated total operation time = 6500 hours) may require additional thinking before starting.

3.2 CARBURIZATION OF 2 1/4 Cr-1 Mo - TEST 3

Table 4 shows the results of carbon analysis of croloy tabs that had been exposed to 1200 F liquid sodium saturated with carbon. The results tend to confirm weight change data obtained for these tabs. There appears to be an incubation period of approximately four days before carburization proceeds rapidly, and then the rate begins to approach that of the austenitic steels.

3.3 CARBURIZATION DURING TAB TESTS - TEST 4

Tabs of 316, 304 and 2 1/4 Cr-1 Mo which had been exposed to 1100 F sodium containing 200-300 ppm oxygen for 4000 hours were analyzed for carbon. The average carbon values for 316 ss is 703 ppm; for 304 ss, 812 ppm; and for 2 1/4 Cr-1 Mo, 331 ppm. While evaluation of TESTS 2 and 4 are as yet incomplete, carbon transfer does not appear to have been grossly affected when oxygen is added to sodium containing low carbon.

4. ANALYTICAL RESULTS

4.1 LOOP SAMPLES

Results of samples extracted from Loop 2, TEST 4, for carbon analysis are listed in Table 5 and are presented graphically in Fig. 14. Results of emission spectrograph samples are shown in Table 6.

Results of samples extracted from Loop 1 for carbon analysis are shown in Table 7 and presented graphically in Fig. 15. Because of down-time and varying operation in December, only one sample for carbon analysis was taken. When the system is reactivated and the testing program is initiated, duplicate samples for carbon analysis will be taken once per week. Emission spectrograph results are shown in Table 8. Two samples for oxygen analysis were taken during December and results indicated 79 ppm O_2 (505 F) on 20 December and 61 ppm O_2 (455 F) on 23 December. These values are lower than those determined by plugging indicator runs (680 F) which has been generally true since operating through the carbon bed. The results of the plugging indicator are believed to be corresponding to carbon solubility rather than oxygen.

4.2 EVALUATION OF THE BEHAVIOR OF CARBON IN SODIUM

Additional samples have been extracted from the hot trapping vessel and analyzed for sodium. A summary of the results is shown in Table 9. Through 12 December, the vessel was held at 400 F; it was then heated to 1600 F to determine if carbon could be hot trapped from the sodium charge. The results indicate no significant reduction in carbon. Further work with the hot trapping vessel will be discontinued for the present. An extrusion device of the type used by ORNL and NASA is being fabricated. When construction of the device is completed, samples will be extracted from the vessel with a dip tube. Reproducibility of samples from the same tube will be determined and a comparison of different tubes will be made in an attempt to elucidate the effect of sampling techniques on carbon results.

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.

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			141.5
221	High Oxide Na-1100 F (PE)	14,000			84.9
225	High Oxide Na-1100 F (PE)	12,000			105.4
189	High Oxide Na-1100 F (PE)	10,000			353.8
249	High Oxide Na-1100 F (PE)	14,000			42.8

(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

TABLE 3 - CARBON ANALYSIS OF SECTION OF EXPOSED SURFACE OF 316 PIPE

	Carbon Content (%) With Distance From Surface		
	0-5 mils	5-10 mils	10-15 mils
Test Nipple			
Inlet	0.286	0.156	0.064
Outlet	0.689	0.319	0.143
Original Pipe			
Inlet	0.912	0.514	0.296
Outlet	0.902	0.599	0.545

TABLE 4 - CARBON CONTENT OF 2-1/4 Cr-1 Mo TABS
EXPOSED TO CARBON SATURATED SODIUM AT 1200 F

Days Exposed	Average Carbon (%)	
	0.062" Tabs	0.012" Tabs
1	0.088	
4	0.090	0.114
6	0.103	0.172
11	0.146	0.219
20	0.294	0.480

TABLE 5 - 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 5 - 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 6 - 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 6 - 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.

TABLE 7 - 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 7 - 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 7 - 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 7 - 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

TABLE 8 - 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	Be	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.

TABLE 9 - CARBON CONCENTRATION - HOT TRAPPING POT

Date	Carbon Content (ppm C)
9-28-63	59 Vessel at 1200 F - O_2 = 18 ppm
	75 Vessel at 1200 F - O_2 = 18 ppm
9-30-63	132 Vessel at 1600 F
10-1-63	66
	64
10-2-63	53
10-3-63	26
10-4-63	44
	33
	37
10-9-63	51
10-10-63	80
	60
10-11-63	45
	106
10-14-63	27 O_2 = 9 ppm
	50
10-15-63	78
	120
10-16-63	181
	83
10-17-63	60
	52
10-18-63	62
	44
10-19-63	95
	83
10-21-63	61
	91
10-22-63	67
	113
	42

TABLE 9 - CARBON CONCENTRATION - HOT TRAPPING POT
(Continued)

Date	Carbon Content (ppm C)
10-23-63	29
10-24-63	48
	47
10-25-63	57
	74
10-28-63	73
10-29-63	60
	82
10-30-63	64
10-31-63	71 Vessel cooling to 400 F
	81
11-1-63	35
11-6-63	62
	77
11-7-63	107
11-11-63	41
	57
11-14-63	45
	68
11-15-63	61
	87
11-20-63	53
11-21-63	55
	60
11-22-63	59
11-29-63	76
	79
12-2-63	69
	56
	97
12-4-63	50
	65
	83

TABLE 9 - CARBON CONCENTRATION - HOT TRAPPING POT
(Continued)

Date	Carbon Content (ppm C)
12-6-63	68
	57
	34
	36
12-9-63	67
	63
	44
	45
	51
12-11-63	33
	35
	42
12-12-63	63 Vessel heated to 1600 F
12-13-63	41
	66
	73
12-16-63	47
	40
12-18-63	51
	70
	87
12-20-63	44
	62
12-30-63	68

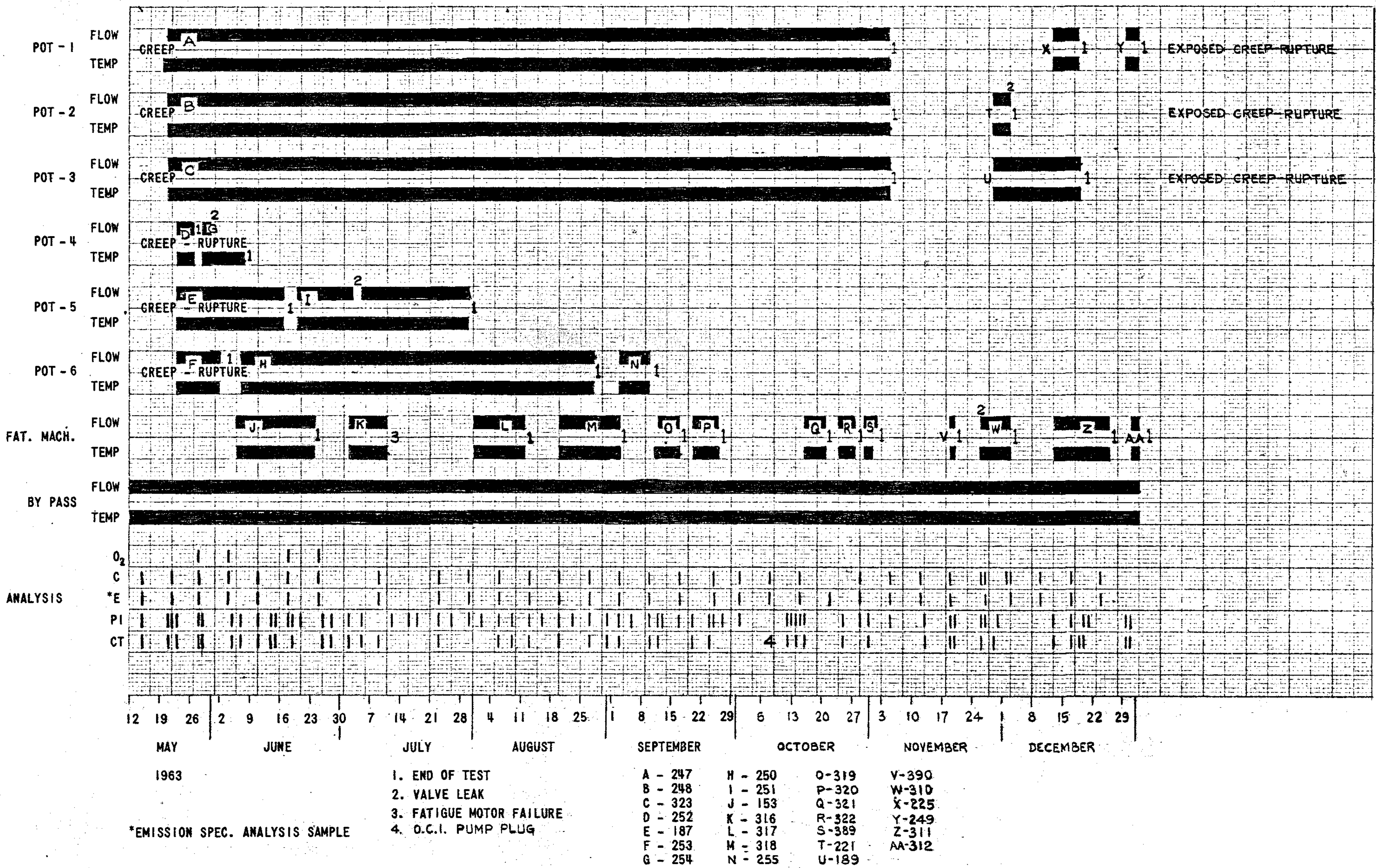

FIG. 1 - OPERATIONAL HISTORY OF LOOP 2 - TEST 4 (Cr-Mo TEST SPECIMENS)

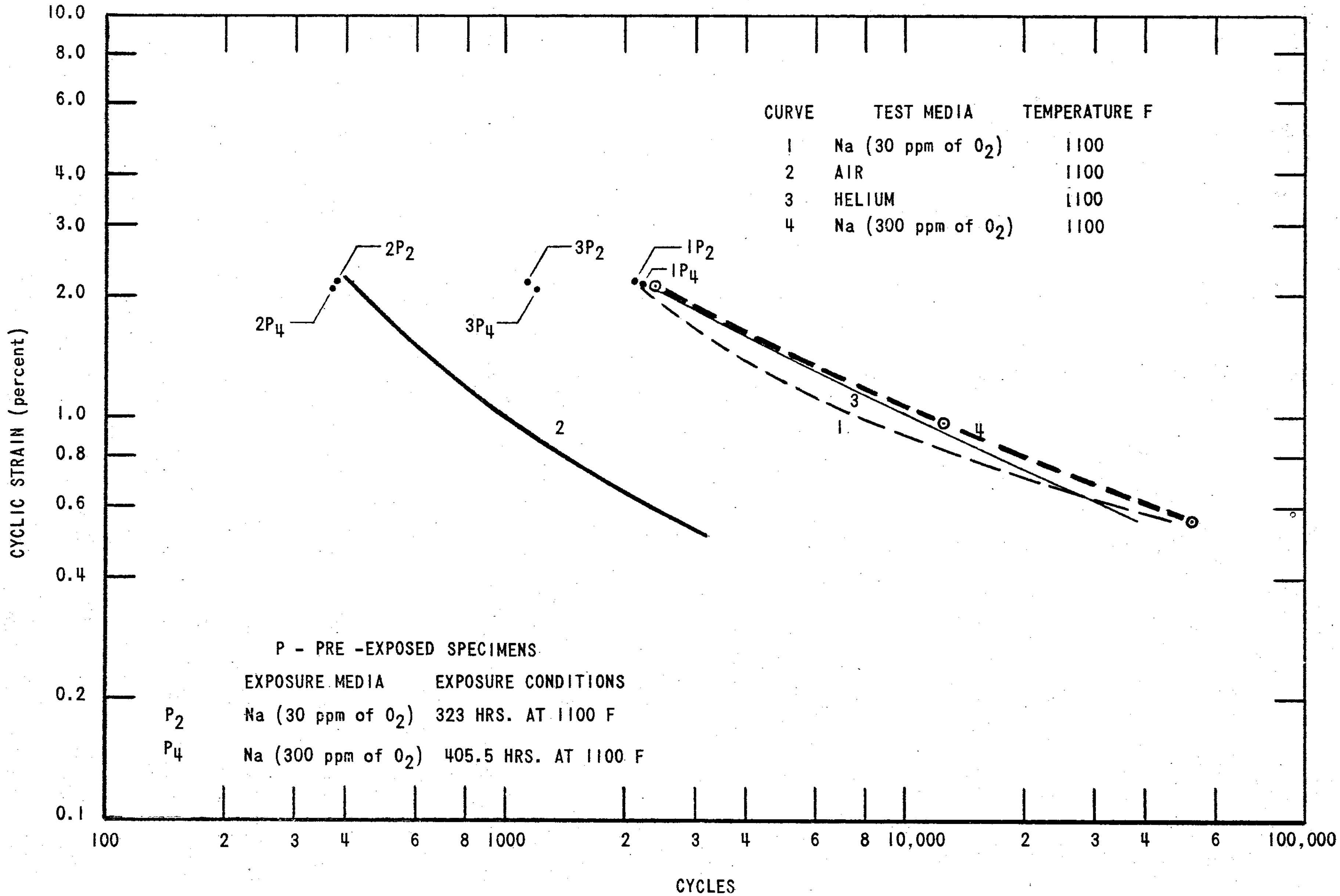

FIG. 2 - FATIGUE CURVES OF AVERAGED DATA FOR 2 1/4 Cr-1 Mo CARBON STEEL FATIGUE TESTS
(Data points for current TEST 4 are shown as ⊙)

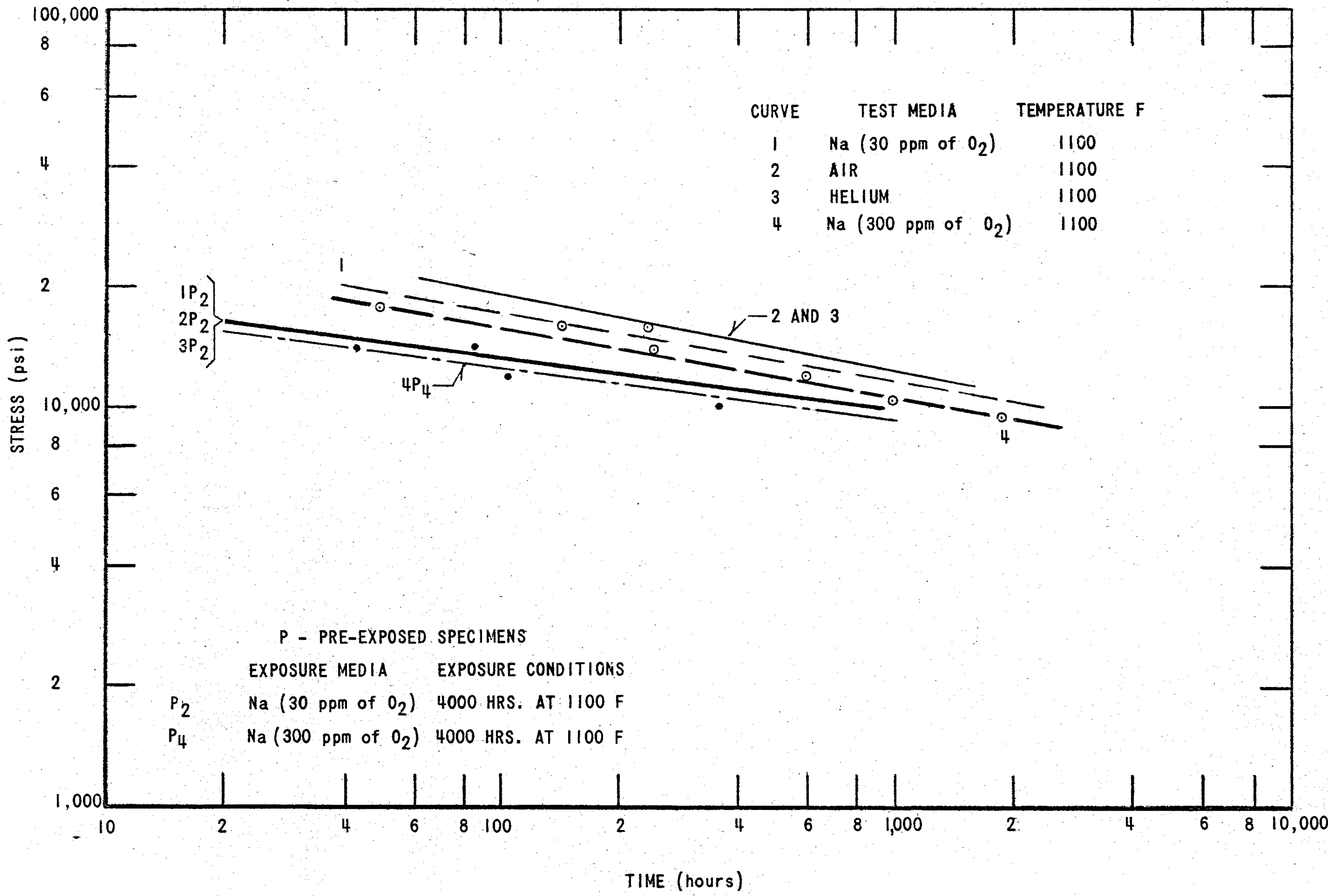

FIG. 3 - CREEP TO RUPTURE OF 2 1/4 Cr-1 Mo CARBON STEEL SPECIMENS
(Data points from TEST 4 shown)

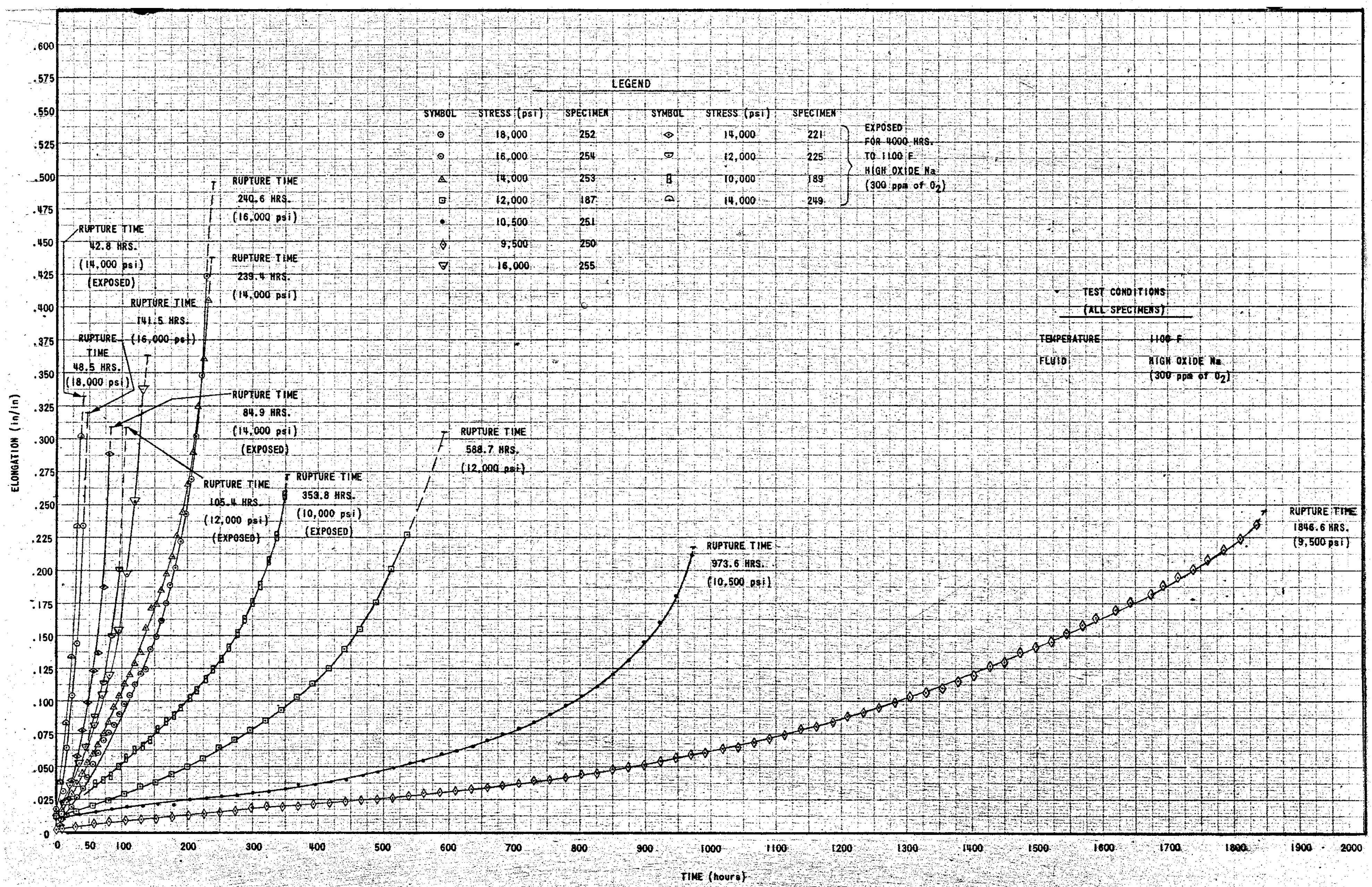

FIG. 4 - ELONGATION OF 2 1/4 Cr-1 Mo STEEL - CREEP RUPTURE SPECIMENS
TEST 4

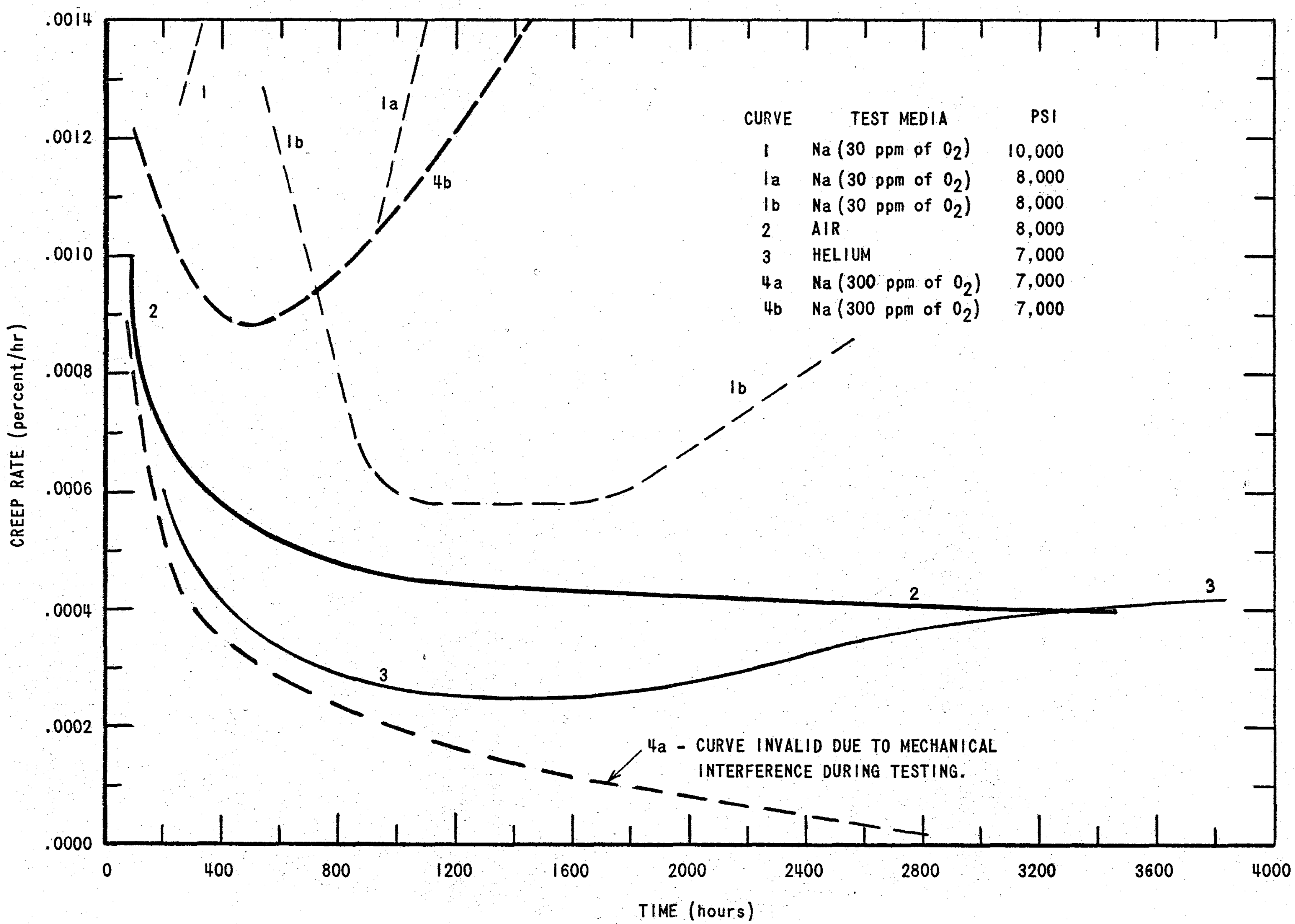

FIG. 5 - CREEP RATES, 2 1/4 Cr- 1 Mo CARBON STEEL 1100 F

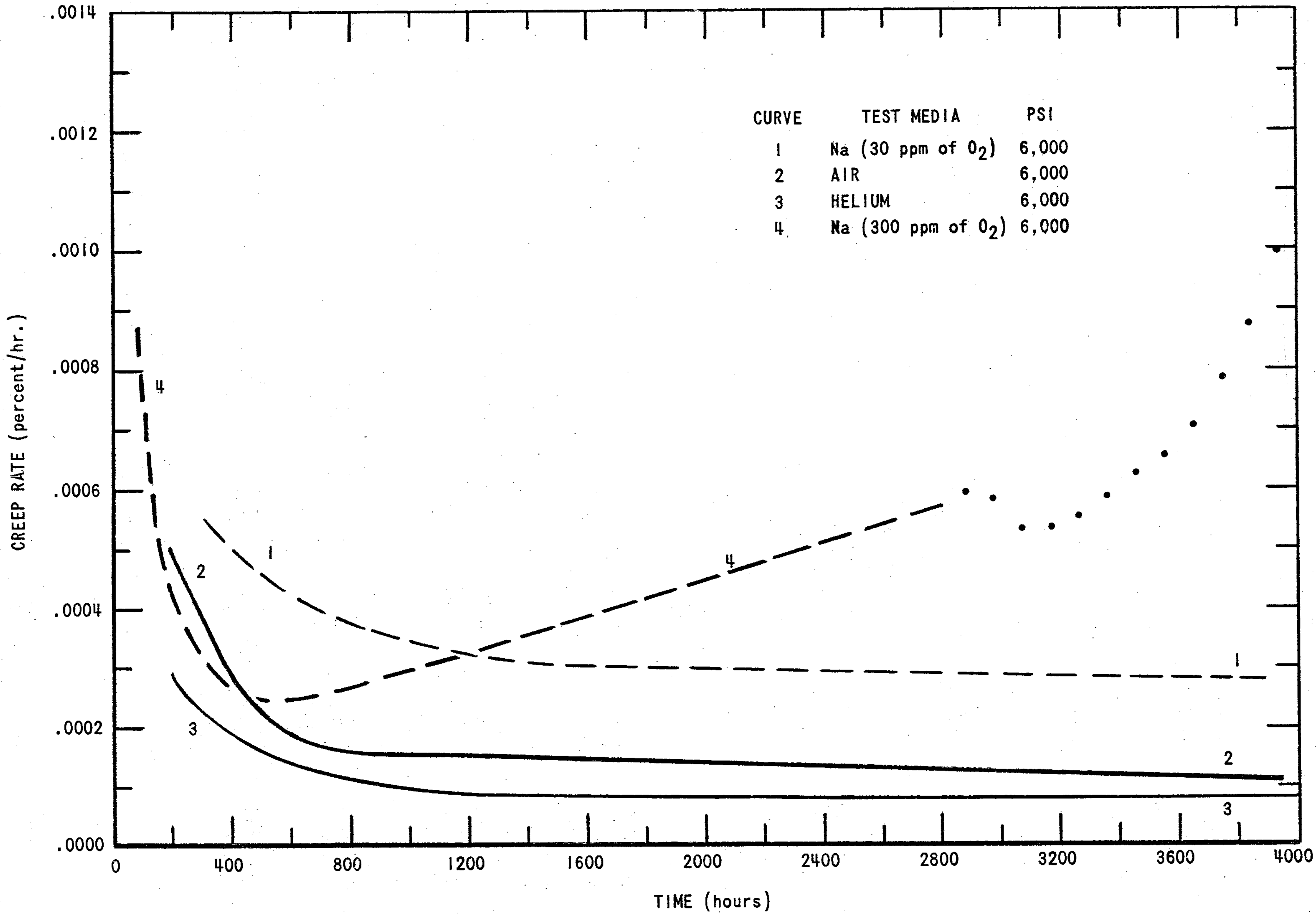

FIG. 6 - CREEP RATES, 2 1/4 Cr-1 Mo CARBON STEEL - 1100 F

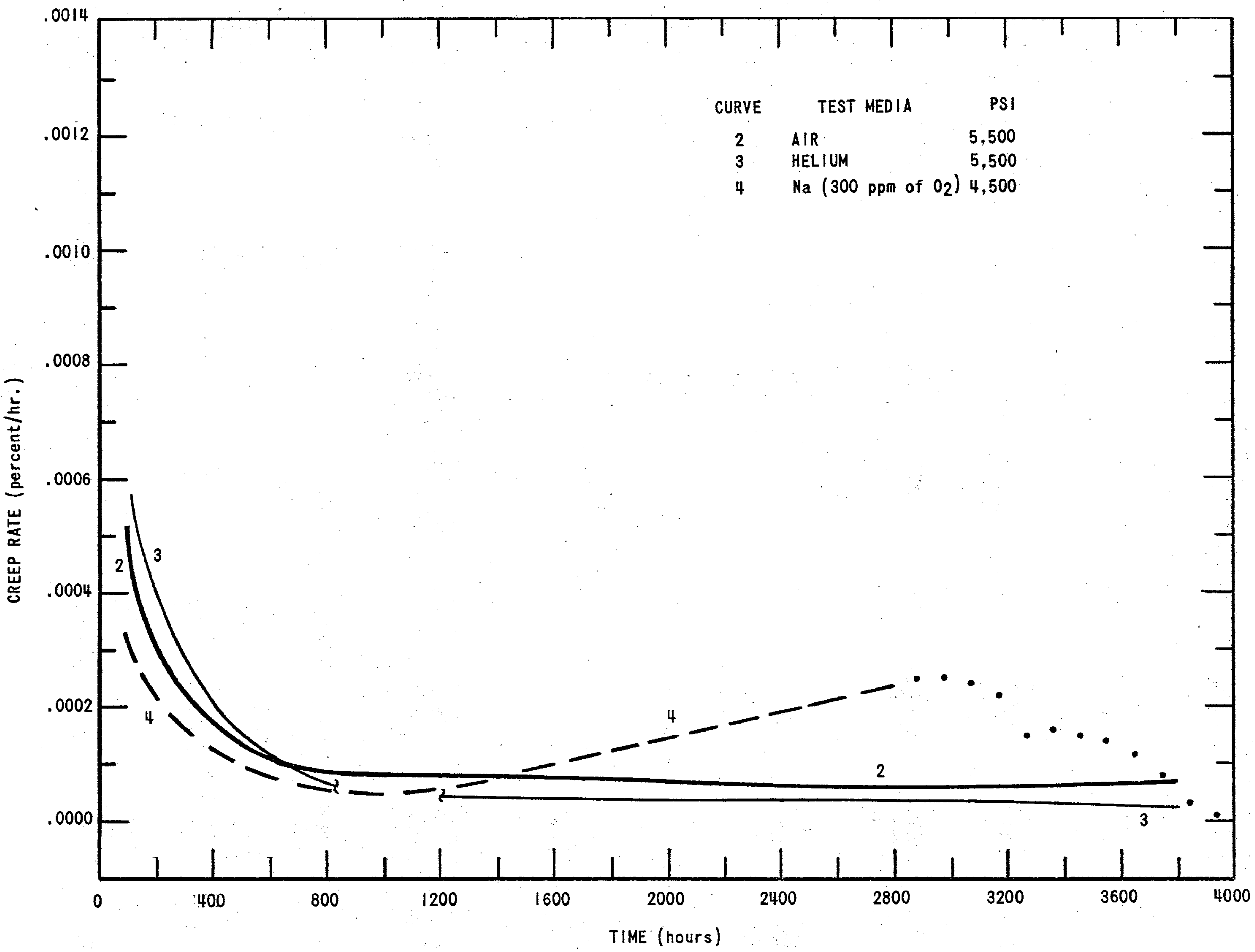

FIG. 7 - CREEP RATES, 2 1/4 Cr-1 Mo CARBON STEEL - 1100 F

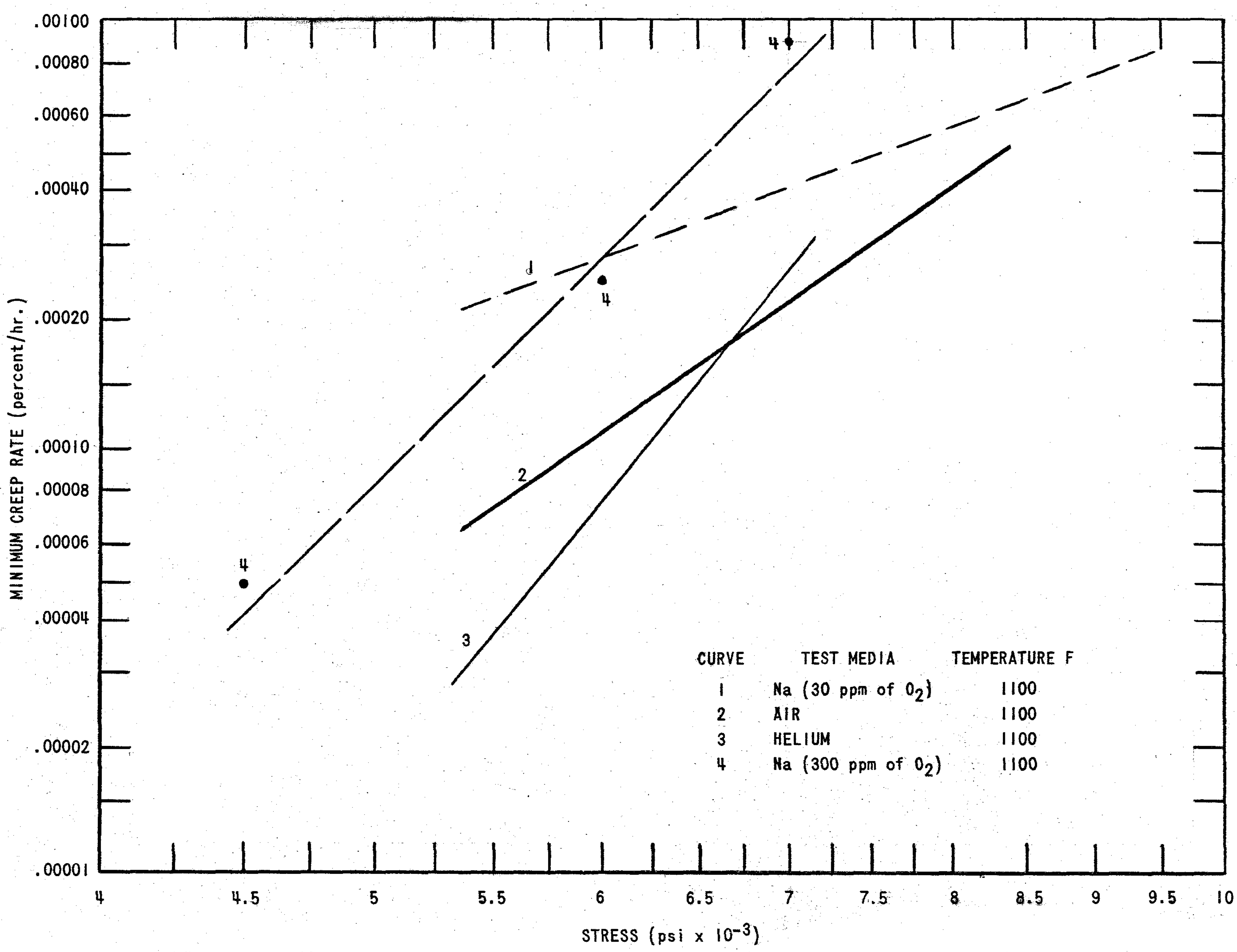

FIG. 8 - MINIMUM CREEP RATE vs STRESS, 2 1/4 Cr-1 Mo CARBON STEEL SPECIMENS
(Data points from TEST 4 shown)

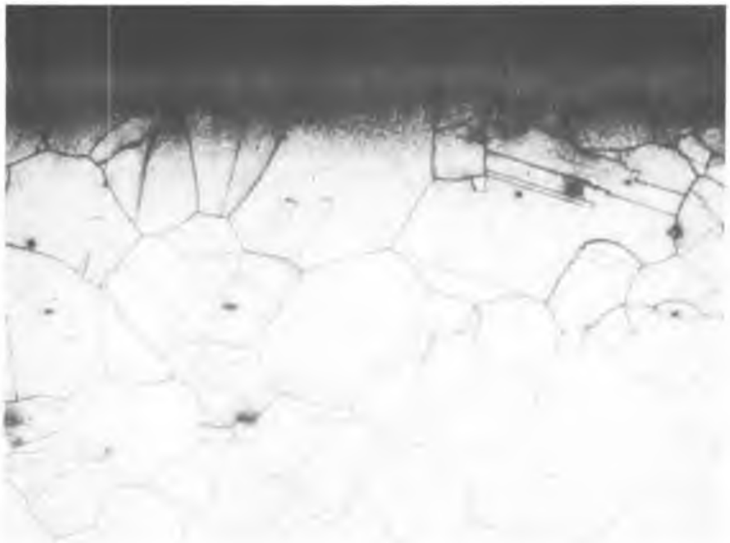

FIG. 9 - 2 DAY
Corrected Exposure Time

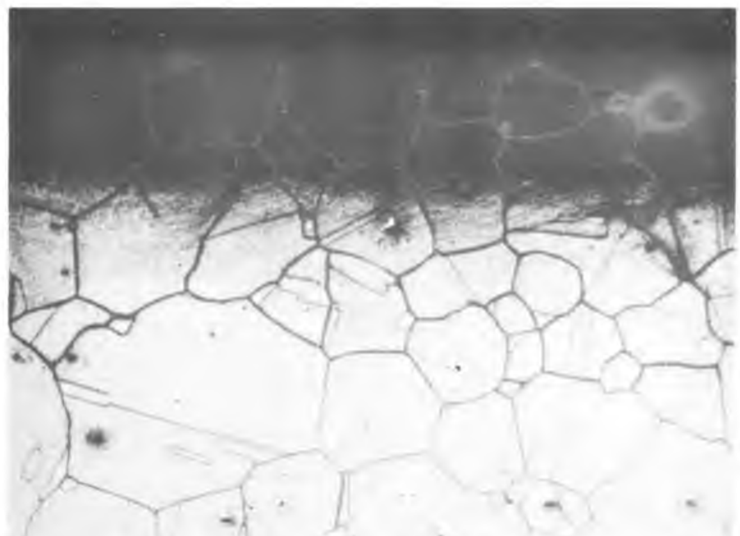

FIG. 10 - 7 DAY
Corrected Exposure Time

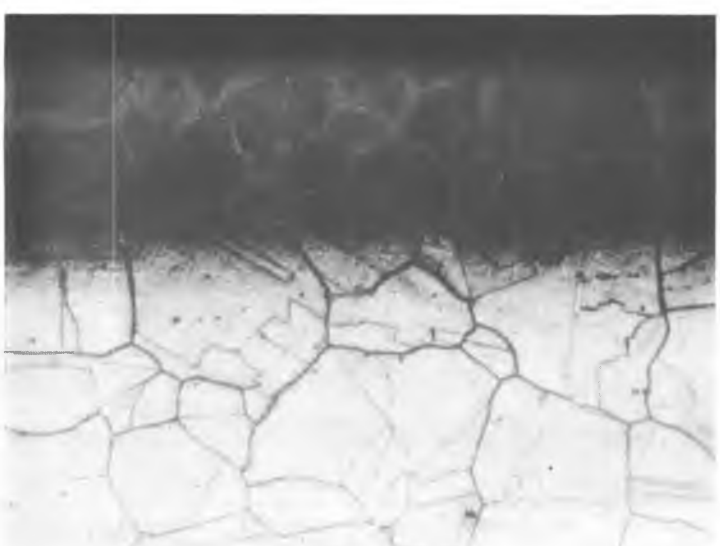

FIG. 11 - 16 DAY
Corrected Exposure Time

CARBURIZATION OF 316 IN 1200°F SODIUM SATURATED
WITH CARBON
(Mag. - 266X) (Etchant-Ferric Chloride)

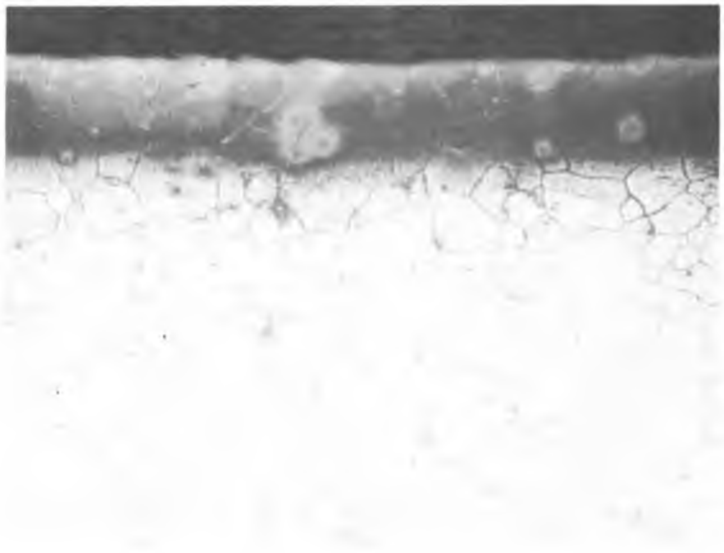

FIG. 12 - OUTLET TEST NIPPLE
133X Etchant: Ferric Chloride

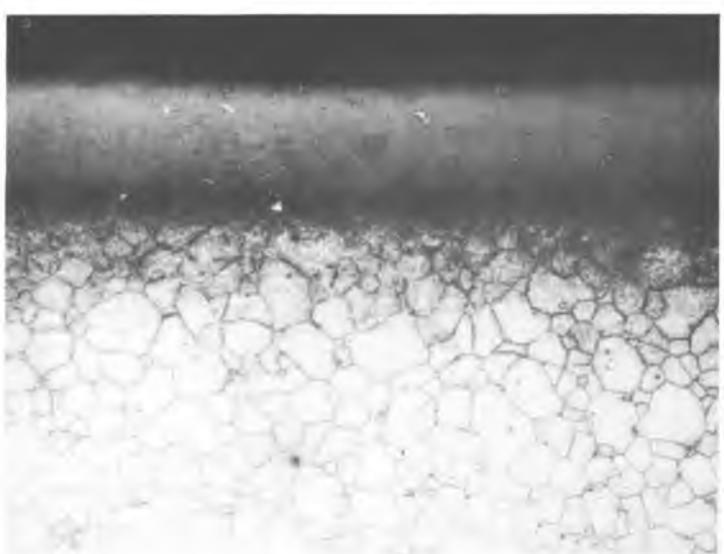

FIG. 13 - OUTLET PIPE
133X Etchant: Ferric Chloride

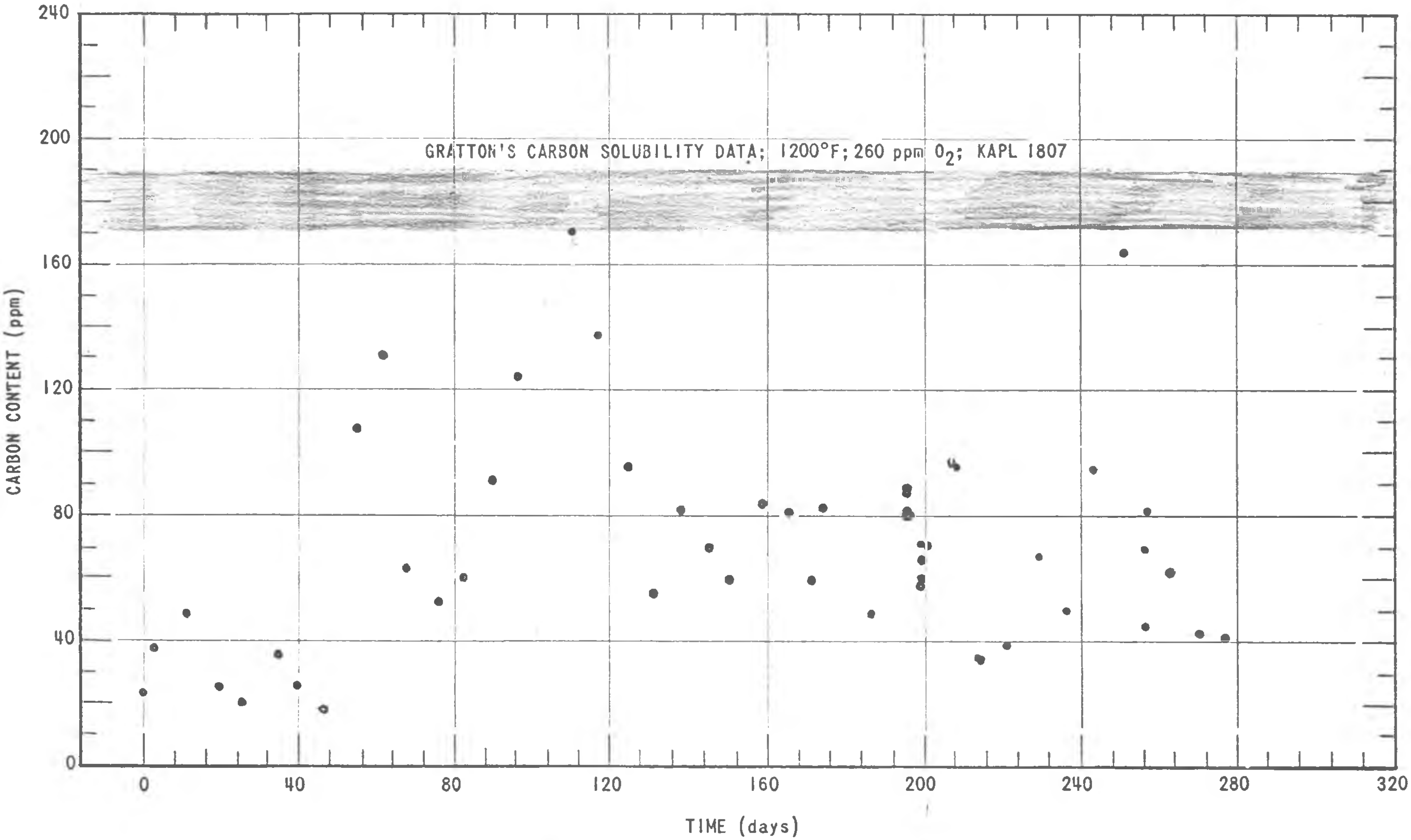

FIG. 14 - CARBON CONTENT, LOOP 2, TEST 4
(Cr- Mo Test Specimens)

R-1611

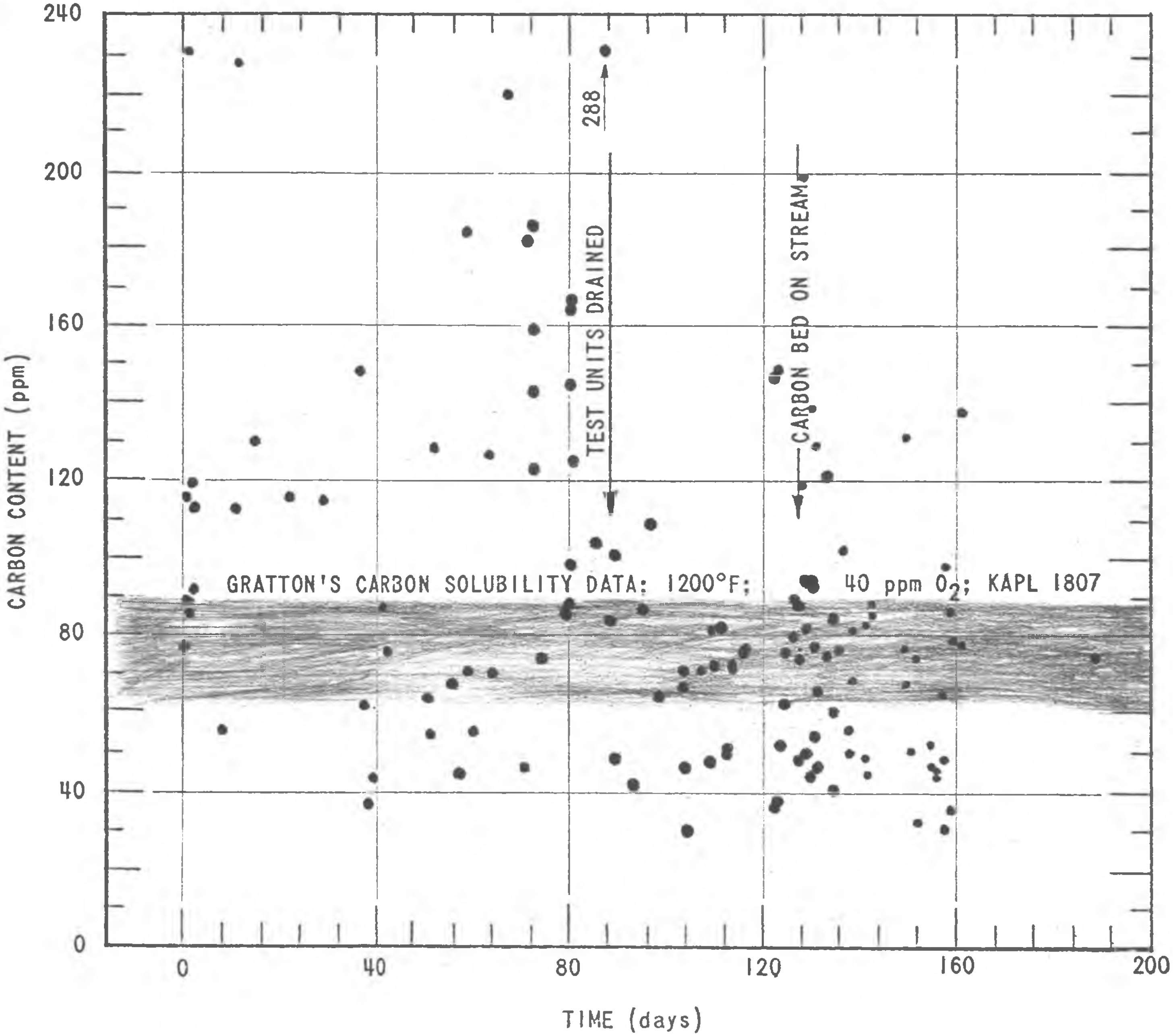

FIG. 15 - CARBON CONTENT, LOOP 1, TEST 3 (PRELIMINARY OPERATIONS)