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PROGRESS REPORT 37
September 1963

to

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

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EFFECT OF 1200°F SODIUM
ON AUSTENITIC AND FERRITIC STEELS
PHYSICAL PROPERTIES OF MATERIALS

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

MASTER

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October 16, 1963

MSA Research Corporation

Subsidiary of Mine Safety Appliances Company

Callery, Pennsylvania

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

EFFECT OF 1200°F SODIUM
ON AUSTENITIC AND FERRITIC STEELS
Physical Properties of Materials

October 16, 1963

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TABLE OF CONTENTS

	Page No.
SUMMARY	i
1. INTRODUCTION	1
2. OPERATION	1
2.1 TEST 1 - STAINLESS STEEL (316) SPECIMENS IN 1200°F, LOW (30 ppm) OXYGEN SODIUM, AIR AND HELIUM	1
2.2 TEST 2 - 2 1/4 Cr-1 Mo STEEL SPECIMENS IN 1100°F, LOW (30 ppm) OXYGEN SODIUM, AIR AND HELIUM	2
2.3 TEST 3 - STAINLESS STEEL (316) SPECIMENS IN 1200°F, HIGH CARBON SODIUM	3
2.4 TEST 4 - 2 1/4 Cr-1 Mo STEEL SPECIMENS IN 1100°F HIGH OXYGEN (300 ppm) SODIUM	3
2.4.1 2 1/4 Cr-1 Mo Steel Fatigue Runs	4
2.4.2 2 1/4 Cr-1 Mo Steel Creep-Rupture Runs	4
2.4.3 2 1/4 Cr-1 Mo Creep Runs	5
2.4.4 2 1/4 Cr-1 Mo Tensile Runs	5
3. ANALYTICAL RESULTS	6
3.1 LOOP SAMPLES	6
3.2 EVALUATION OF THE BEHAVIOR OF CARBON IN SODIUM	6
3.3 ANALYSIS OF SODIUM FOR CO FORMATION	8
3.4 SUMMARY	9
4. METALLURGICAL RESULTS	9
4.1 TEST 1 - STAINLESS STEEL (316) SPECIMENS IN 1200°F, LOW (30 ppm) OXYGEN SODIUM	9
4.2 TEST 2 - 2 1/4 Cr-1 Mo STEEL SPECIMENS IN 1100°F, LOW (30 ppm) OXYGEN SODIUM	10
4.3 TEST 4 - 2 1/4 Cr-1 Mo STEEL SPECIMENS IN 1100°F, HIGH (300 ppm) OXYGEN SODIUM	10
TABLES	12
FIGURES	21

Progress Report 37

EFFECT OF 1200°F 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 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

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

These tests have been completed. Topical reports are being written which will include the results of all the environmental runs and will attempt to correlate these results with the analytical and metallurgical findings.

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

The final phases of the shakedown operation have been reached prior to running the foils and test specimens in sodium with a high carbon concentration. During the next report period, the loop will be operated with the carbon source in stream, followed by operation with foils in the test units. Carbon samples have been taken almost daily with the results ranging from 43 to 220 ppm. Increasing the temperature of the sampling tank is being studied for the effects on carbon fluctuation in the loop. A portion of sodium removed at temperature from the expansion tank is being gettered at 1600°F and sampled for carbon. The carbon concentration is steadily decreasing.



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

The creep tests in sodium have been in operation over 2800 hours. Some fatigue runs have been completed with the results showing fatigue life in high oxygen sodium to be about the same as in low oxygen sodium and helium.

The creep-rupture runs on the original materials are completed and the results show a consistently lower life, but not of a significant amount, in high oxygen sodium compared to low oxygen sodium.

Progress Report 37

EFFECT OF 1200°F SODIUM ON AUSTENITIC AND FERRITIC STEELS
PHYSICAL PROPERTIES OF MATERIALS

1. INTRODUCTION

The University of Michigan is in the process of completing the final helium tests and MSAR is examining the specimens generated under Tests 1 and 2. At the same time, Test 3 is in its shakedown operations and Test 4 is in progress.

The test program for Tests 1 and 2 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, is summarized in Progress Report 33 (Table 1).

The test program for Tests 3 and 4 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, is summarized in Progress Report 33 (Table 2).

2. OPERATION

L. H. Kirschler
J. W. Freeman (University of Michigan)

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

The following tests have been completed:

1. All air tests
2. All sodium tests
3. All fatigue tests
4. All tensile tests
5. All creep-rupture tests

The 11,000 psi creep test being conducted in helium was terminated after 4002 hours of testing. The minimum creep rate was 0.18%/1000 hours from 1000 to 2400 hours of testing. It then increased to a termination rate of 0.24%/1000 hours.

The 10,500 psi creep test is still in progress after 3900 hours. It reached a minimum creep rate of 0.08%/1000 hours after

800 hours of testing, then increased to a present rate of 0.11%/1000 hours. This test will be terminated during the next report period completing all phases of TEST 1.

Preliminary conclusions and tabulated data are reported in Progress Report 34. A graphic illustration of the operational history is shown in Fig. 1. The latest creep rate results are shown in Figs. 2, 3 and 4, with Fig. 5 summarizing the minimum creep rate vs stress for the three environments. The tests just terminated or the test still in progress will not alter the test results since all of the minimum rates had been reached previously. Therefore, the results as discussed in previous reports are not affected.

A topical report on TEST 1 is in progress and is expected to be issued in the near future. Since this report will finalize the findings of TEST 1, no further discussion will be made concerning TEST 1 in future progress reports.

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

The following tests have been completed and data from these tests have been summarized in Table 1 of Progress Report 34:

1. All air tests
2. All sodium tests
3. All helium tests

The operational history of TEST 2 is graphically illustrated in Fig. 6.

The final 2 1/4 Cr-1 Mo creep test in helium was terminated after 4001 hours of testing. Under a load of 7000 psi, this specimen had a minimum creep rate of 0.24% which was reached at 1400 hours and increased to 0.415% at termination. This test completes all phases of TEST 2.

The tabulated data from TEST 2 is summarized in Progress Report 34, Table 1, and the results were discussed in Progress Report 33.

The final creep rates are shown in Figs. 7, 8, and 9 as the creep rates vs time, with Fig. 10 summarizing the minimum creep rates vs stress for the environmental tests.

The results and conclusions of TEST 2 will be finalized in a topical report. Since further data will not be generated from this test, no further discussion will be made concerning TEST 2 in future progress reports.

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

Loop 1 will be utilized for this test and is in operation but without specimens. The fluctuation of the carbon level has been to such a degree (less than 100 ppm to over 200 ppm) that start of the actual test is being delayed until there is a better understanding of the variables involved. The analytical and engineering groups are actively engaged in the clarification of the complete carbon picture. Various methods are being employed to resolve this matter and are discussed under Section 3.

Daily sodium samples are being taken for carbon analyses. Flow was maintained through all test units and bypass until a sodium leak developed in the fatigue-machine test-pot flange necessitating the isolation and draining of this unit. The design of the test units are such that the flange is in the cover-gas space out of the liquid metal. Upon investigating the reason for the high level in the fatigue unit, sodium was discovered in several cover-gas lines to other test units. All test pots were drained, while maintaining flow in the bypass. The cover-gas manifold was removed from the system, washed out, dried and replaced. Sampling continued with operation confined to the bypass only, to determine if carbon can be introduced by air back diffusing through the flanges of the test pots. Analyses during this period were inconclusive.

Two to three pounds of sodium were removed from the Loop 1 expansion tank while circulating at 1200°F. This charge was put into a small pot for further carbon studies. The charge has been heated to 1600°F with a zirconium getter to determine if consistent carbon samples can be obtained. This program is in progress. (See Section 3)

A large effort is being undertaken during the next report period to resolve the carbon situation and make recommendations as to the initiation and test procedure for TEST 3.

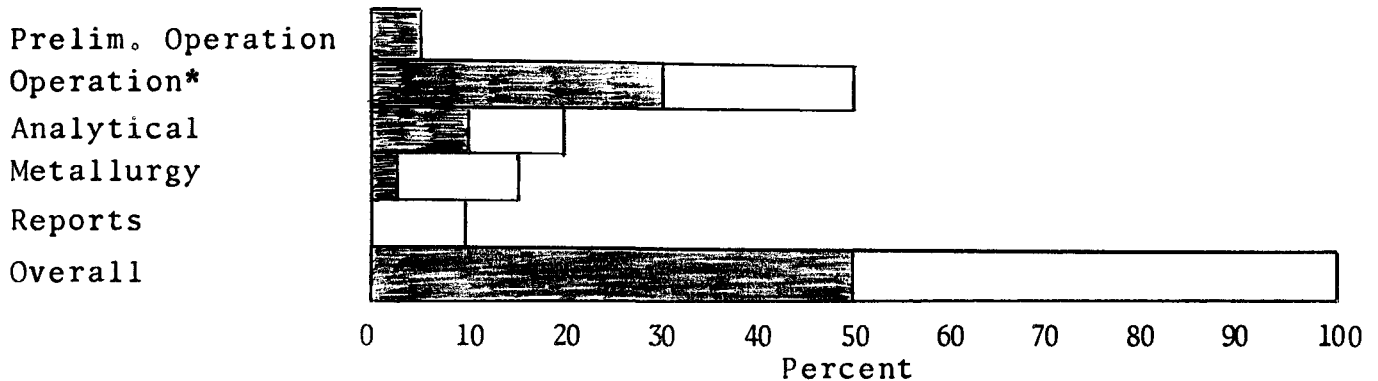
The free energies of formation of carbon compounds at 1100°F are being tabulated, but the data is not sufficiently complete to include at this time.

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

The operation in sodium on the original material is well underway. The three creep tests have been in operation for over 2800 hours. All the creep-to-rupture tests on original material have been completed. Four fatigue tests, one at intermediate strain and three at high strain, on original material are yet to be run. The various tests on exposed specimens will not begin until after the creep tests.

Upon completion of the low cyclic strain fatigue tests, the test mandrels were changed and the intermediate cyclic strain tests initiated. Two tests were completed during this report period.

The progress schedule for TEST 4 is shown below and the operational history of Loop 2 (TEST 4), including estimated completion dates, is shown graphically in Fig. 11.



* 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

Two fatigue runs were made at the intermediate cyclic strain conditions. One specimen failed after 12,612 cycles and the second after 15,295 cycles. A third specimen will be tested during the next report period completing this series of runs and the high cyclic strain tests initiated.

Table 1 shows the results of these runs as well as those obtained from the low cyclic strain runs. Upon completion of the high cyclic strain runs, curves comparing the high oxygen runs (TEST 4) vs the low oxygen runs (TEST 2) will be included. Thus far the results show specimen failures in the same range as the helium and low oxygen sodium tests.

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

The creep-rupture runs on original material have been completed. These results are found in Table 2 and are plotted in

Fig. 12 with curves from TEST 2 for comparison. The results of the creep-rupture tests in high oxygen sodium show a consistently shorter life, but the actual difference based on the accuracy of the tests does not appear to be significant.

Creep-rupture runs on material exposed to 1100°F high oxygen (300 ppm) sodium will be initiated upon completion of the 4000 hour creep tests.

2.4.3 2 1/4 Cr-1 Mo Creep Runs

The determination of creep rates of the three 2 1/4 Cr-1 Mo specimens continued with stresses of 7000 psi, 6000 psi and 4500 psi. These runs have been in progress approximately 3000 hours.

The creep rate of the 7000 psi specimen has decreased to 0.01%/1000 hours at 2800 hours and is still decreasing. The preliminary curve is plotted in Fig. 7 with results from TEST 2 for comparison.

The creep rate of this specimen is difficult to comprehend. It has a lower creep rate than the 6000 or 4500 psi specimens and lower than any of the specimens in air or helium. It is not believed to be a realistic rate and the test unit has been examined externally for possible explanations without results. The test is continuing but another specimen under the same stress will begin during the next report period. Since only three creep runs are made per TEST it is important for data development to have three good runs. Based on the creep rates of the other specimens the new 7000 psi specimen should reach its minimum creep rate before the end of the scheduled runs for TEST 4.

The creep rate of the 6000 psi specimen decreased to 0.25%/1000 hours after 400 hours and now has increased to 0.57%/1000 hours after 2800 hours. The preliminary curve is plotted in Fig. 8 with results from Test 2 for comparison.

The creep rate of the 4500 psi specimen decreased to 0.05%/1000 hours after 1000 hours and has increased to 0.24%/1000 hours after 2800 hours, as shown in Fig. 9.

2.4.4 2 1/4 Cr-1 Mo Tensile Runs

The 2 1/4 Cr-1 Mo tensile tests are not scheduled until completion of the creep runs.

3. ANALYTICAL RESULTS

S. J. Rodgers

3.1 LOOP SAMPLES

Samples have been extracted from Loop 2, TEST 4, on a weekly basis. Results on carbon are shown in Table 3 and graphically in Fig. 13. Results of emission spectrograph samples are shown in Table 4.

Samples have been extracted on a weekly basis from Loop 1 for emission spectrograph analysis and these results are shown in Table 5. Carbon results are shown in Table 6 and graphically in Fig. 14. During the latter part of August and the early part of September, the carbon results were erratic. After the creep test pots were drained, the carbon level appeared to become more stable. This suggests that particulate carbon is the cause of the erratic results which have been obtained on carbon analyses.

3.2 EVALUATION OF THE BEHAVIOR OF CARBON IN SODIUM

In an effort to elucidate the behavior of particulate carbon in sodium, three additional studies were made:

1. Effect of centrifugation.
2. Migration of carbon in sodium during freezing.
3. Hot trapping.

If carbon is present in particulate form, it was felt that centrifugation would cause the carbon particles to migrate; the following experiment was run to determine if such is the case. Two 5/16 in. OD thin wall stainless steel tubes 4 1/2 in. long were flattened and seal welded at one end. Sodium was charged to the capsule under argon purge, the open end crimped and seal welded. These capsules were then centrifuged (~ 280 g) in a gas-fired furnace at a temperature of 1200/1250°F for one hour. This was accomplished by suspending the specimens from a vertical shaft connected to a 1750 rpm motor. The capsules were suspended on the shaft, the furnace lit and the centrifuge motor started. The temperature was brought up to 1200°F in approximately 15 minutes and then manually controlled for one hour. At the end of this time, the motor was stopped and disconnected from the capsule shaft. The shaft with capsules was removed from the furnace and the lower end of the capsules squeezed to separate the sodium into two sections, preventing the migration of carbon from one end to the other during cooling.

The results of this experiment were as follows:

<u>Tube No.</u>	<u>Sample Location</u>	<u>Carbon Content (ppm C)</u>
3	Top	386
	Bottom	504
7	Top	256
	Middle	385
	Bottom	123

On the basis of these samples, no conclusions can be drawn on the effect of centrifugation. The reason for the unusually high carbon values is not immediately obvious. Improper cleaning of tubes is always a potential source of contamination but the pre-treatment which included degreasing with a solvent, etching with HNO_3 and rinsing with distilled water should have given a clean surface. Contamination during welding, which was not performed in an inert gas box, is also a possibility. A duplication of these runs is planned for the future under more aseptic conditions.

In an effort to determine migration of carbon during freezing of sodium, samples were extracted from Loop 1, allowed to freeze and then a section of sodium was removed from the center of the dip bucket as one sample and the remainder of the sodium was analysed as a second sample. Results were as follows:

<u>Date</u>	<u>Sample No.</u>	<u>Carbon Content (ppm C)</u>		
		<u>Core</u>	<u>Bucket</u>	<u>Total</u>
8/28/63	1	485	96	133
8/28/63	2	141	121	124
8/28/63	3	327	160	185
9/5/63	4	57	166	144
9/5/63	5	163	167	165

There appears to be a segregation of carbon when sodium freezes. However, on the basis of these few samples, no conclusions can be made on which conditions cause migration to either the hot or cold zone. In the MSAR method for carbon in sodium where the whole sample is analysed, this migration would not have any effect on the total carbon result -- this is verified in comparing daily samples analysed by both coring and the standard method. In samples where sodium is extruded from a tube, unrealistic results might be expected.

In a further attempt to demonstrate that variations in the apparent carbon content of sodium are due to particulate material, a stainless steel vessel containing zirconium chips was filled with sodium which was piped directly from Loop 1. The object of the procedure was to reduce the carbon content of the sodium by hot trapping (soluble carbon should be removed by gettering providing

a new sink for the solution of particulate carbon) and at the same time provide a non-circulating, relatively quiescent sodium source. The pot is now operating at 1600°F and samples are being extracted daily. Results to date are as follows:

<u>Date</u>	<u>Carbon Content (ppm C)</u>	
9/28/63	59	Vessel at 1200°F -- O ₂ 18 ppm
	75	" " " "
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	

On the basis of these few results, it appears that the carbon level is being reduced and that the problem of particulate carbon in these samples had been eliminated. Work will continue with this vessel until sufficient data is collected.

3.3 ANALYSIS OF SODIUM FOR CO FORMATION

It has been reported by General Electric that the off-gas is primarily CO during dissolution and acidification of sodium samples for carbon analysis. To determine whether the procedure used at MSAR failed to account for all of the carbon in a sample (i.e., that which could be lost as CO), it was decided to again examine the off-gas for CO. This time an alternate method was used -- a colorimetric indicator capable of a theoretical detection limit of ~6 ppm CO based on a 1 gram sodium sample. Interference by H₂ raised the practical detection limit to ~24 ppm CO. The off-gases formed during the analysis were purged through tubes containing the colorimetric reagent. Results were as follows:

<u>Sample Material</u>	<u>Treatment</u>	<u>CO Content of Sample (ppm CO)</u>
Sodium	H ₂ O	< 24
	HCl	< 24
Sodium	H ₂ O	< 24
	Van Slyke Solution	< 6
Hydrogen	500 cc	< 24
KC ₈	H ₂ O	< 12
	H ₂ O	< 6
	HCl	< 12

The interference of H_2 necessitates that the CO levels in sodium be reported as a less-than value. The values found with sodium were equivalent to the H_2 background, thus the CO level should be essentially zero. Potassium graphite (KC_8) was also run; this compound has a high carbon-to-alkali metal ratio and, because of the poorer heat transfer compared with sodium, it should result in higher localized temperatures, a condition which could lead to the formation of CO. However, the results indicate that little, if any, CO is produced with the values which were found easily attributable to H_2 . Values with KC_8 were lower than with sodium; the free alkali metal available to produce H_2 is lower in the compound.

3.4 SUMMARY

The following comments are made to summarize the work during the past month:

1. The variation in carbon results is likely due to the presence of particulate carbon. Isolation of the creep test pots from the expansion tank has resulted in better agreement among carbon samples; no source of particulate carbon is immediately evident in the creep test pots.
2. Additional evaluation of the effect of centrifugation seems to be in order.
3. Sampling of the hot trapping vessel will continue until a consistent carbon level is attained.
4. Little, if any, CO is formed during dissolution and acidification of the sodium in Loop 1 and Loop 2.

4. METALLURGICAL RESULTS

4.1 TEST 1 - STAINLESS STEEL (316) SPECIMENS IN 1200°F, LOW (30 ppm) OXYGEN SODIUM

The surfaces of selected stainless steel specimens (No. 39 and No. 99) were milled and the millings were analyzed for carbon conductimetrically in order to provide semi-quantitative carbon profiles. Table 7 shows the results of carbon analysis as a function of distance from the surface.

Examination of the data in Table 7 verifies the conclusions observed earlier in microphotographic examination and microhardness profile data. In the case of the stressed sample (No. 39) carburization of a depth of at least 0.006 in. is evident, while the unstressed sample shows the diffusion layer to be less than 0.002 in.

thick. The results of carbon analysis confirm the effects observed, where the diffusion of carbon appears to be related to stress.

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

A sample of 2 1/4 Cr-1 Mo that had been stressed to rupture in 1100°F sodium (rupture time = 6632.8 hrs) was surface milled, and the millings were analyzed for carbon. Carbon contents as a function of distance from the surface are 0-2 mil (435 ppm), 2-4 mil (284 ppm), 4-8 mil (376 ppm), 8-12 mil (421 ppm), 12-16 mil (420 ppm). It is apparent that croloy has not been fully decarburized. Further carbon analyses are necessary to define the nature of the carbon profile in croloy when samples are exposed to sodium.

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

A series of tabular specimens has been exposed to 1100°F sodium in Loop 2 prior to determination of the mechanical properties of croloy in oxygen-contaminated sodium. Tabular materials of four alloys (316, 304, 2 1/4 Cr-1 Mo, and Armco Iron) were suspended in the liquid phase, the cover gas above the liquid metal level, and at the liquid-vapor interface. Tabs of two thicknesses (0.062 in. and 0.010 in.) of each material were exposed at each of the three sites in the loop. A sufficient number of tabs were prepared to provide exposures of 1, 3, 6 and 10 days duration. The oxygen content of the 1100°F sodium throughout the duration of the run ranged between 200 and 300 ppm oxygen.

At this writing, carbon analysis of specimens completely immersed in the liquid is near completion and these results are shown in Table 8. Carbon analyses of Armco Iron samples have not been completed. Also shown are the carbon contents of selected specimens exposed solely to vapor. Fig. 15 shows the carbon content of the alloys as a function of time. The one-day test results are not included in plotting the data, since they proved to be erratic. The erratic nature of one-day test exposures may be due to the time required (generally one day) to equilibrate the oxygen content of the sodium stream after the samples have been immersed.

The raw data plots shown in Fig. 15 suggest the following conclusions:

1. Quantitative carbon content relationships cannot be derived with the limited number of test durations performed.

2. In the case of the two stainless steels, carburization is a function of tab surface to weight or volume ratio. With the 0.010 in. tabs, the carbon increase approaches 200% in ten days, while the 0.062 in. tabs had carbon increases of approximately 15% (316) and 10% (304) in the same time period.
3. While the carbon results of 2 1/4 Cr-1 Mo samples are more erratic than in the case of stainless steel, it is apparent that the decarburization rate of 2 1/4 Cr-1 Mo is low over the ten days of test exposure.
4. The few carbon analyses that had been performed on samples exposed solely to the vapor suggest carburization of stainless steel in the vapor phase.

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

<u>Specimen No.</u>	<u>Condition</u>	<u>Specimen Thickness in.</u>	<u>% Cyclic Strain</u>	<u>Cycles to Failure</u>	<u>Time at Temp. Hrs</u>
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

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

<u>Specimen No.</u>	<u>Condition</u>	<u>Stress Psi</u>	<u>Elong %</u>	<u>Reduction of Area %</u>	<u>Rupture Time (Hrs)</u>
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			(2)973.6
250	High Oxide Na-1100 F	9,500			1846.6
255	High Oxide Na-1100 F	16,000			141.5

- (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

TABLE 3 - CARBON CONCENTRATION - LOOP 2
(TEST 4 - SODIUM 300 ppm O_2)

<u>Date</u>	<u>Carbon Content (ppm C)</u>
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	Analysis not complete
10-4-63	88
	81
10-7-63	59
	67

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

<u>Date</u>	<u>Fe</u>	<u>B</u>	<u>Co</u>	<u>Mn</u>	<u>Al</u>	<u>Mg</u>	<u>Sn</u>	<u>Cu</u>	<u>Pb</u>	<u>Cr</u>	<u>Si</u>	<u>Ti</u>	<u>Ni</u>	<u>Mo</u>	<u>V</u>	<u>Be</u>	<u>Ag</u>	<u>Zr</u>	<u>Li</u>	<u>Ca</u>
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

TABLE 5 - EMISSION SPECTROGRAPH ANALYSES - LOOP 1 (ppm)

<u>Date</u>	<u>Fe</u>	<u>B</u>	<u>Co</u>	<u>Mn</u>	<u>Al</u>	<u>Mg</u>	<u>Sn</u>	<u>Cu</u>	<u>Pb</u>	<u>Cr</u>	<u>Si</u>	<u>Ti</u>	<u>Ni</u>	<u>Mo</u>	<u>V</u>	<u>Bc</u>	<u>Ag</u>	<u>Zr</u>	<u>Sr</u>	<u>Ba</u>	<u>Ca</u>
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

TABLE 6 - CARBON CONCENTRATION - LOOP 1

<u>Date</u>	<u>Carbon Content (ppm C)</u>
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

* Sampled at 600° F

TABLE 6 - CARBON CONCENTRATION - LOOP 1
(Continued)

<u>Date</u>	<u>Carbon Content (ppm C)</u>
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 PROFILES OF 316 STAINLESS
EXPOSED TO 1200 F SODIUM

Sample No.	39 (3DLX3)	99
Exposure Time, hrs	2489.5	4000
Stress Level, psi	18,500	0
Average Carbon, ppm		
0-2 mil	3211	1063
2-4 mil	1516	491
4-6 mil	686	561
6-8 mil	806	471
As-Received Carbon		
Content, ppm	458	458

TABLE 8 - CARBON CONTENT OF ALLOYS IMMERSSED IN 1100 F
SODIUM CONTAINING 200-300 ppm OXYGEN

Average Carbon Content in ppm ¹					
Alloy	As Received	Exposure Time, Days			
		1	3	6	10
<u>2-1/4 Cr-1 Mo</u>					
0.062	991	1038	898	839	882 (1067) ²
0.010	1107	1159	1110	815	1109
<u>316 ss</u>					
0.062	481	450	545	557	565 (700) ²
0.010	579	622	738	1244	1675 (751) ²
<u>304 ss</u>					
0.062	667	668	619	764	739
0.010	640	913	840	1645	1681
<u>Armco Iron</u>					
0.062	127				
0.010	220				

1 - Average of at least two analyses

2 - Exposed solely to vapor

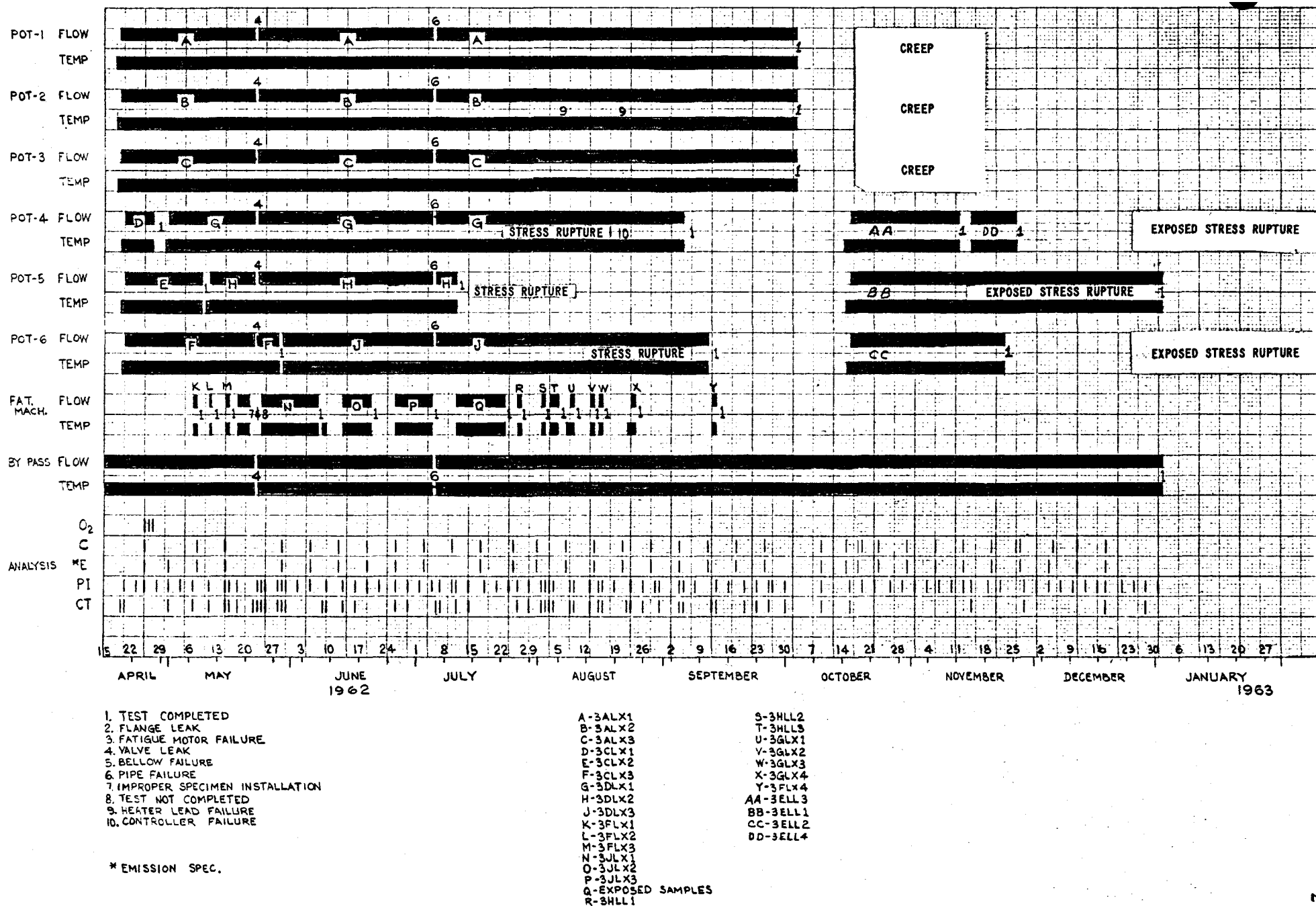


FIG. 1 - OPERATIONAL HISTORY OF LOOP 1 DURING TEST 1 IN SODIUM (30 ppm O₂)

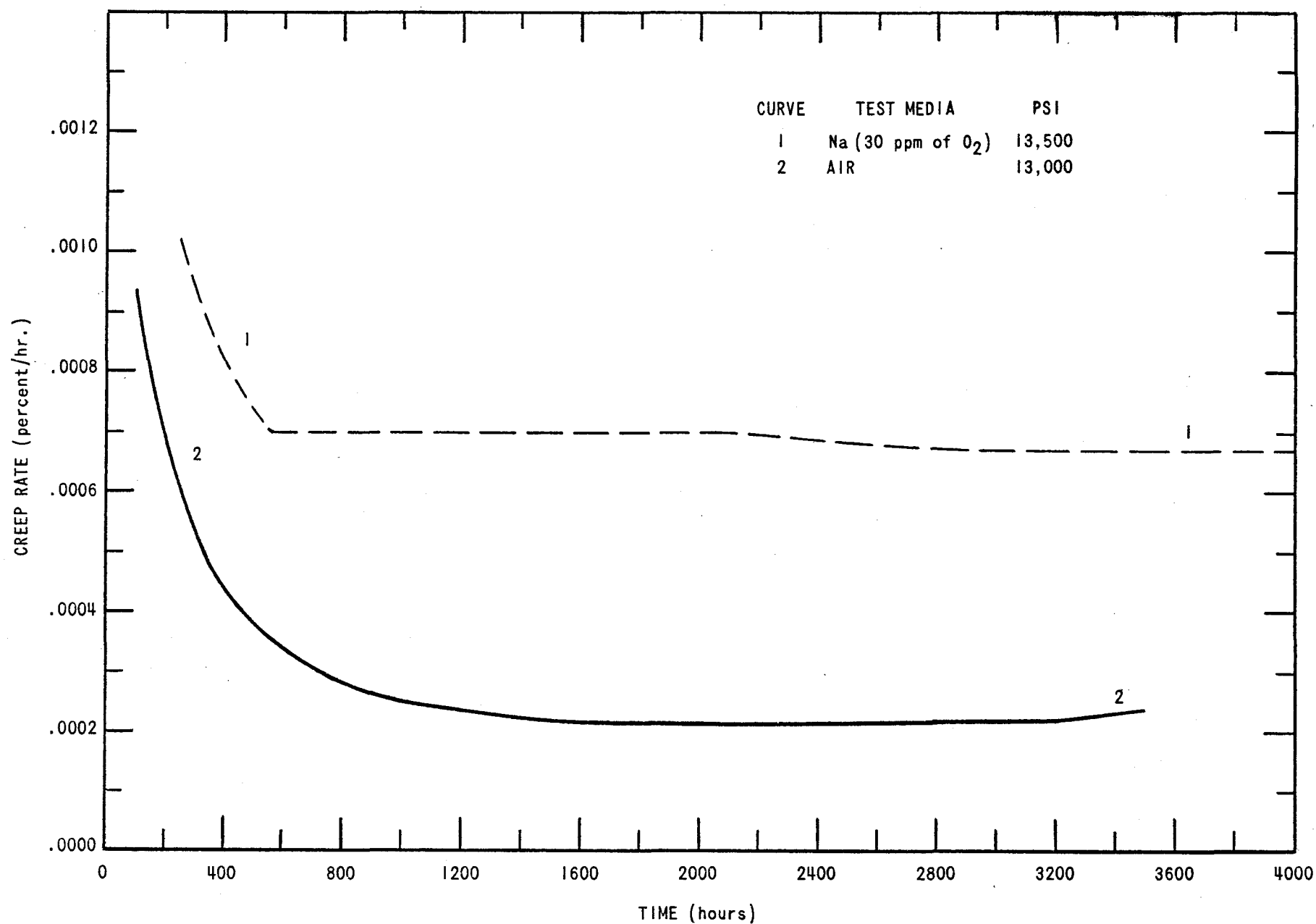


FIG. 2 - CREEP RATES, 316 STAINLESS STEEL SPECIMENS - 1200°F

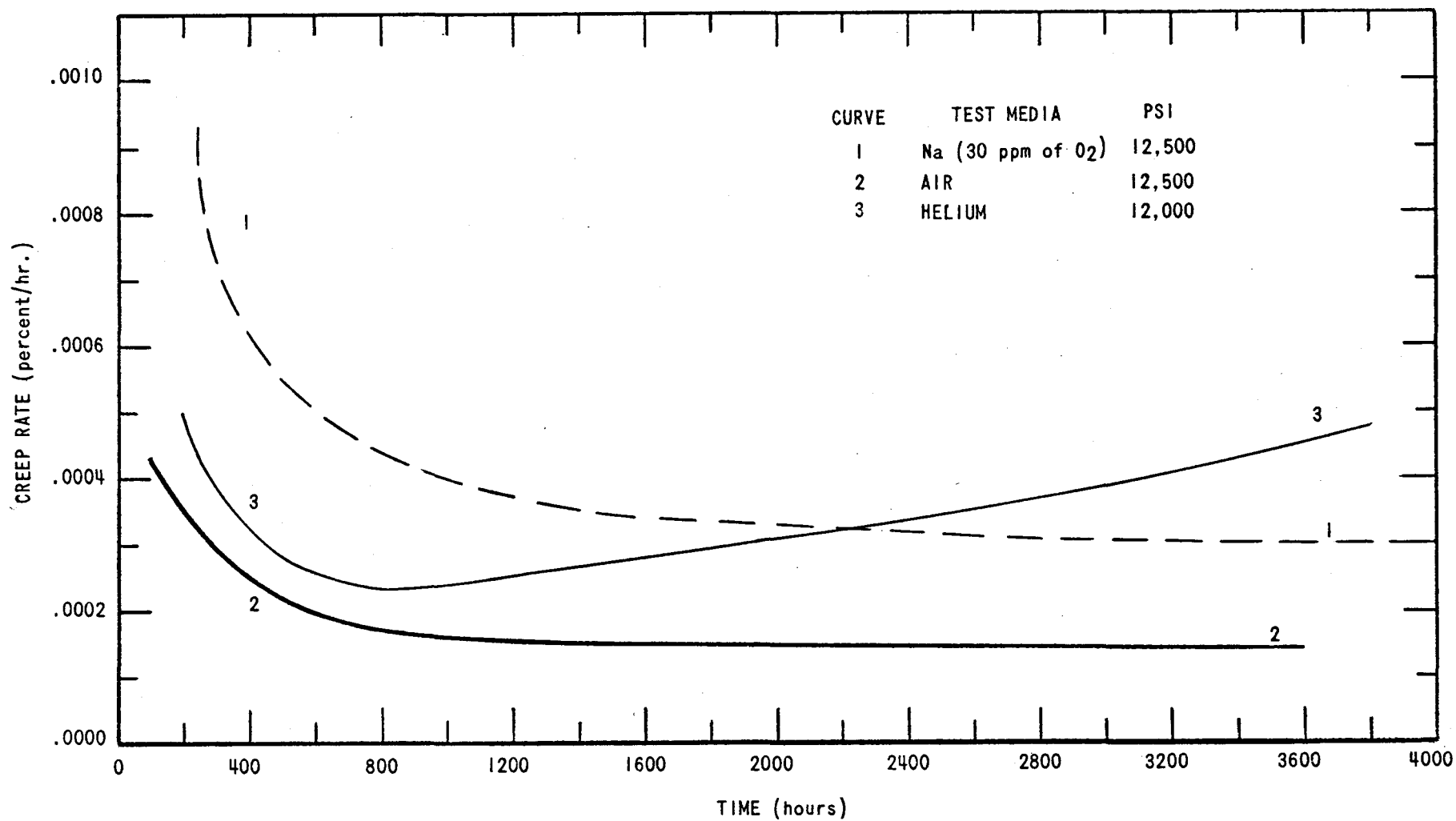


FIG. 3 - CREEP RATES, 316 STAINLESS STEEL SPECIMENS - 1200°F

R-1599

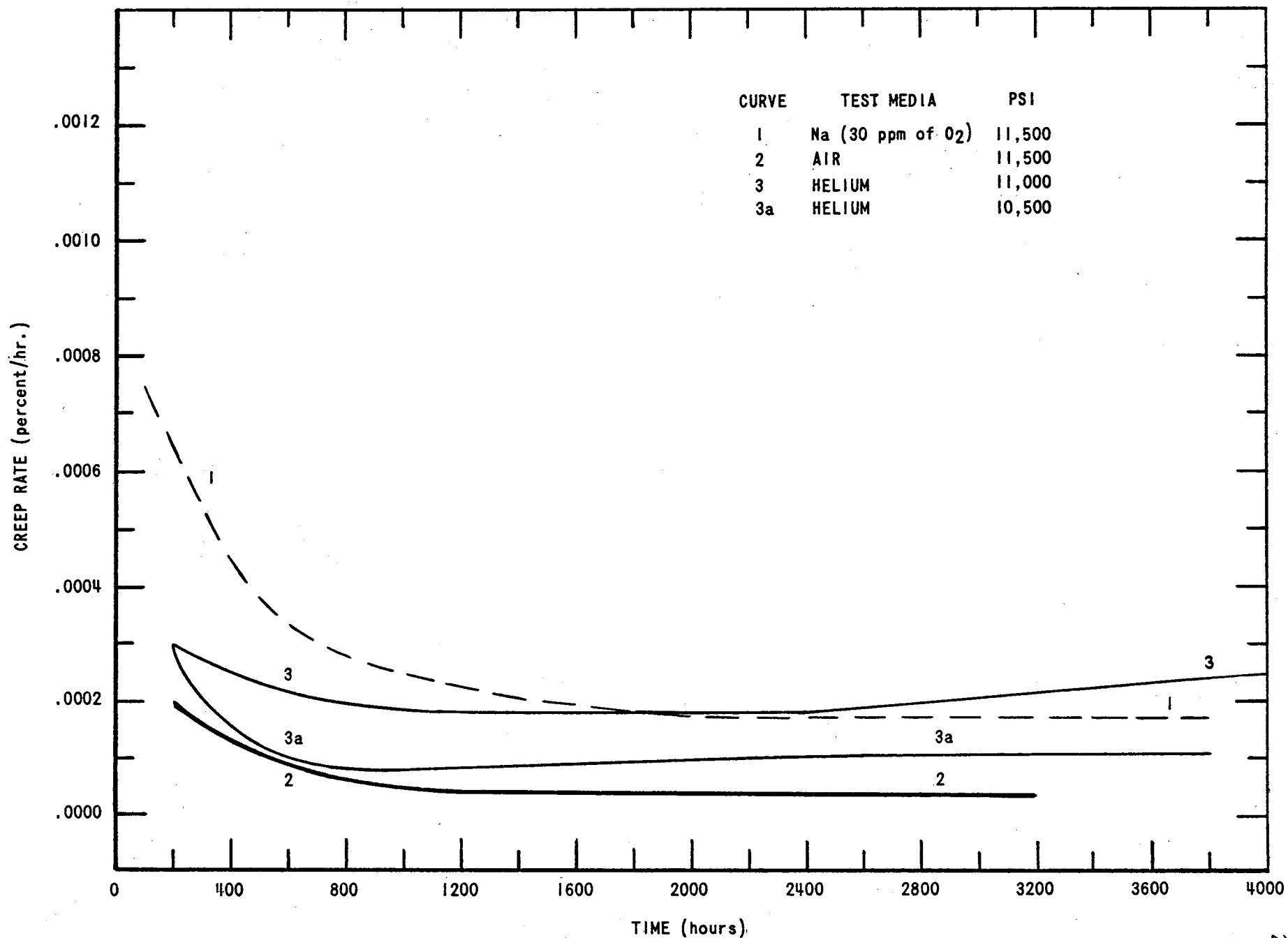


FIG. 4 - CREEP RATES, 316 STAINLESS STEEL SPECIMENS - 1200°F

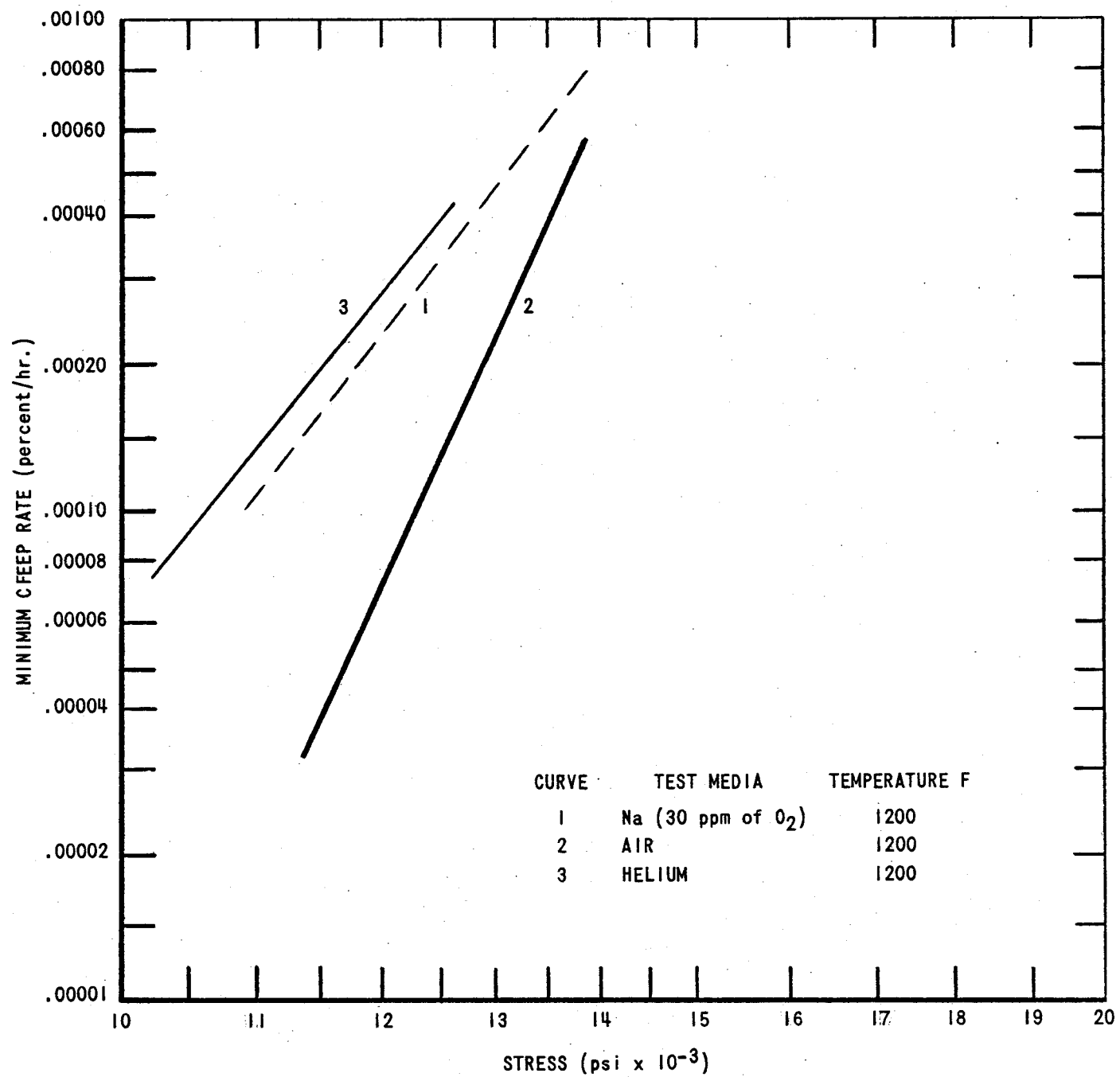


FIG. 5 - MINIMUM CREEP RATES vs STRESS, 316 STAINLESS STEEL SPECIMENS

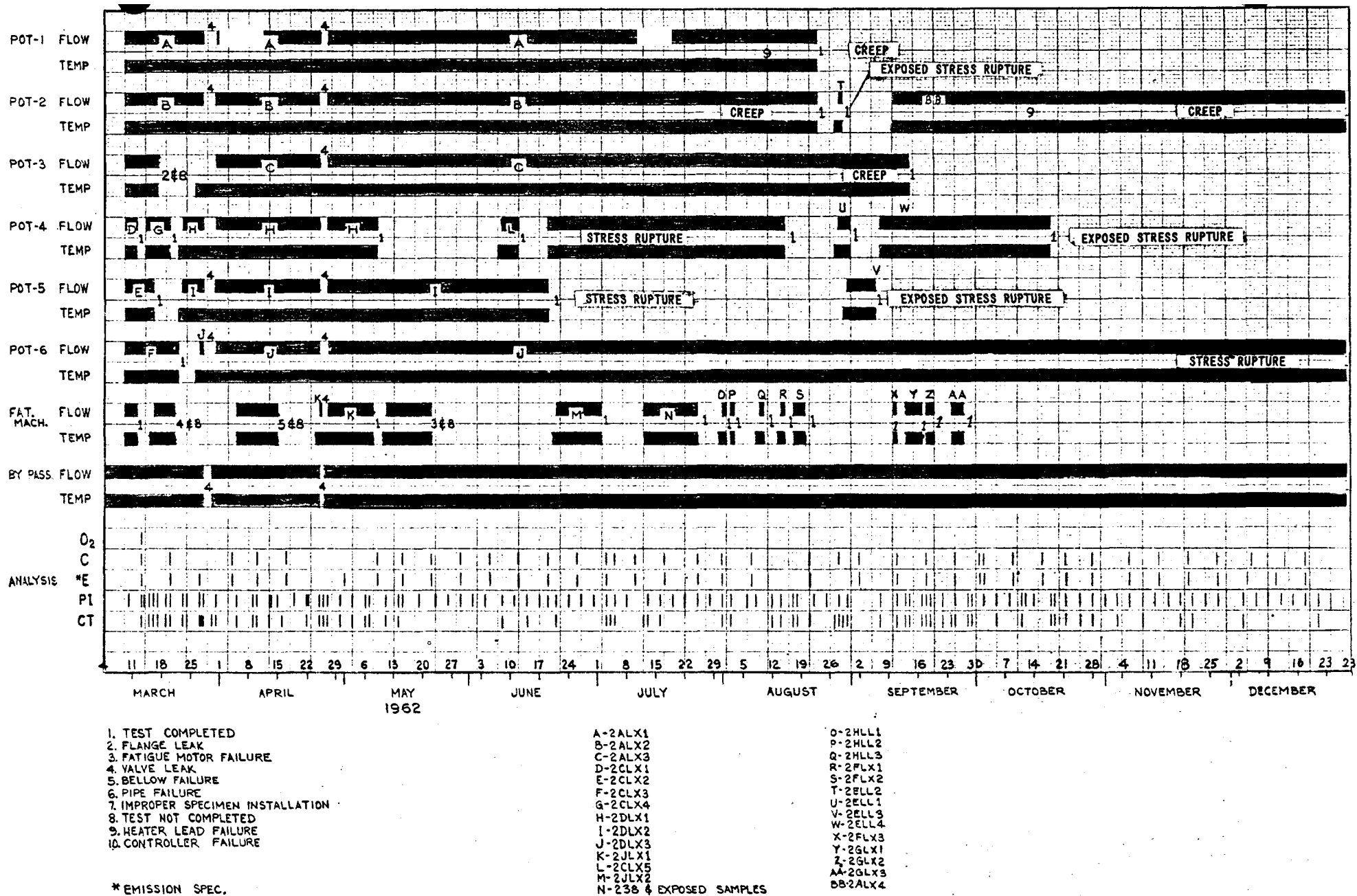


FIG. 6 - OPERATIONAL HISTORY OF LOOP 2 DURING TEST 2
(Cr-Mo Test specimens in Sodium 30 ppm O₂)

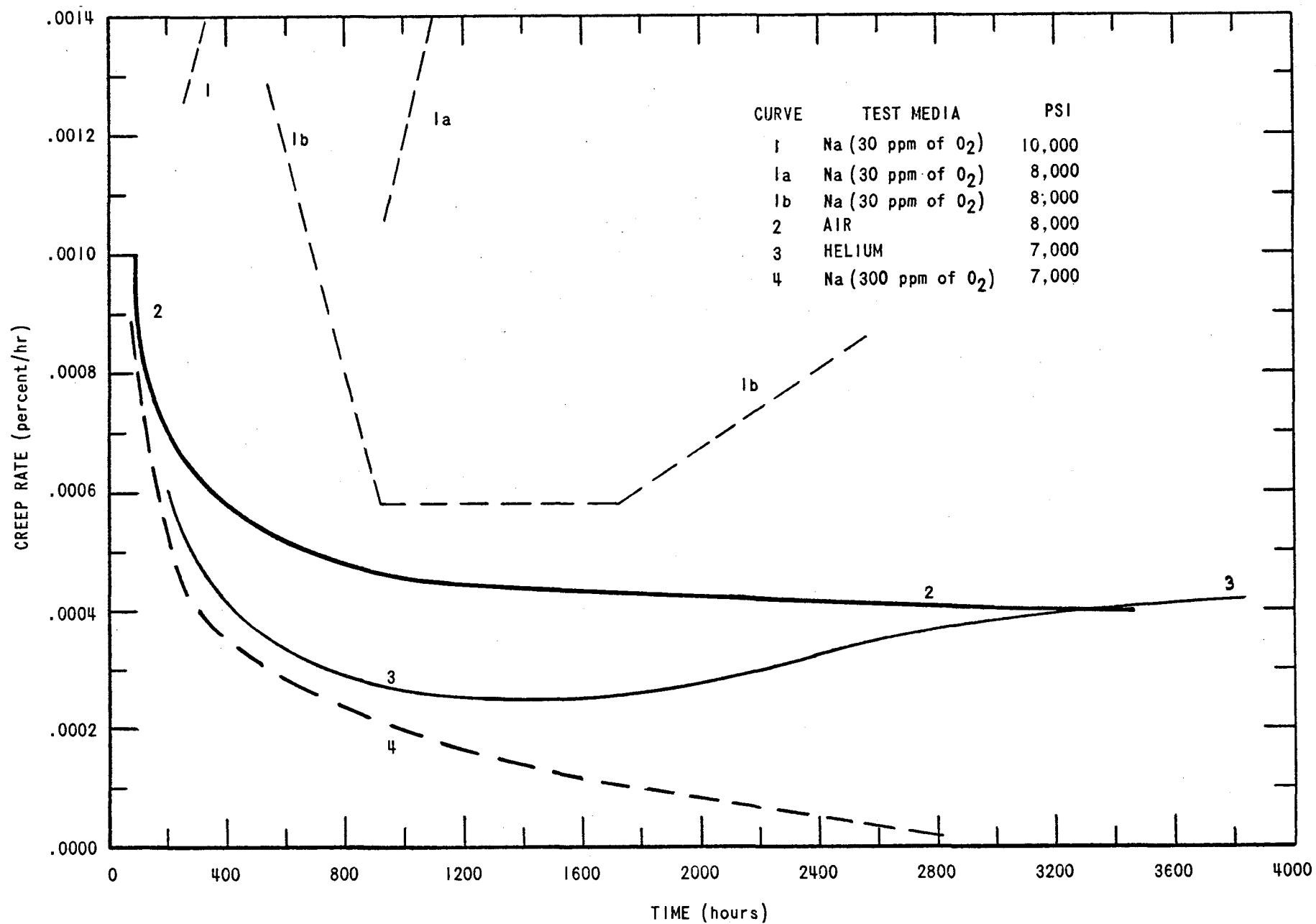


FIG. 7 - CREEP RATES, 2 1/4 Cr-1 Mo CARBON STEEL - 1100°F

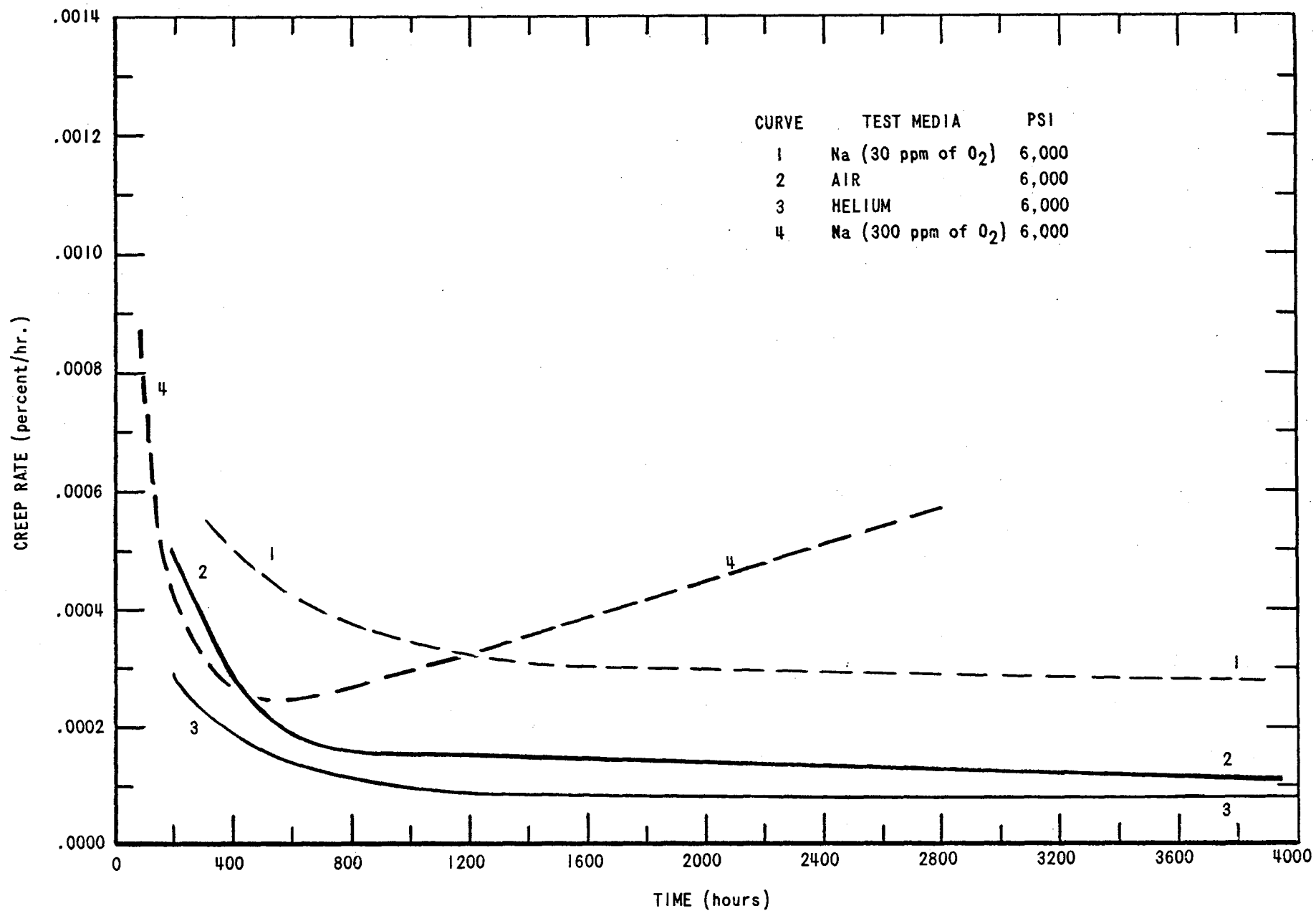


FIG. 8 - CREEP RATES, 2 1/4 Cr-1 Mo CARBON STEEL - 1100°F

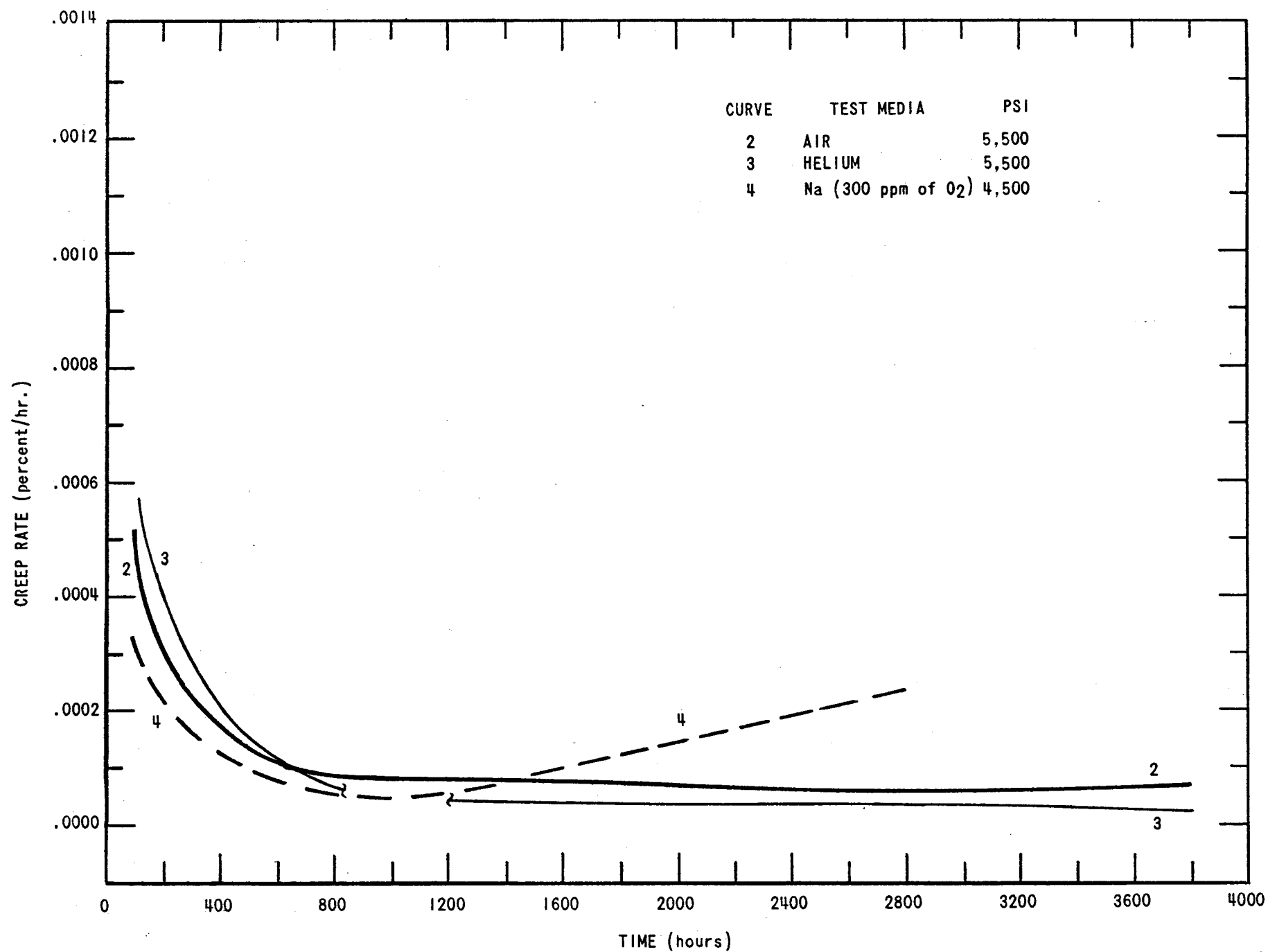


FIG. 9 - CREEP RATES, 2 1/4 Cr-1 Mo CARBON STEEL - 1100°F

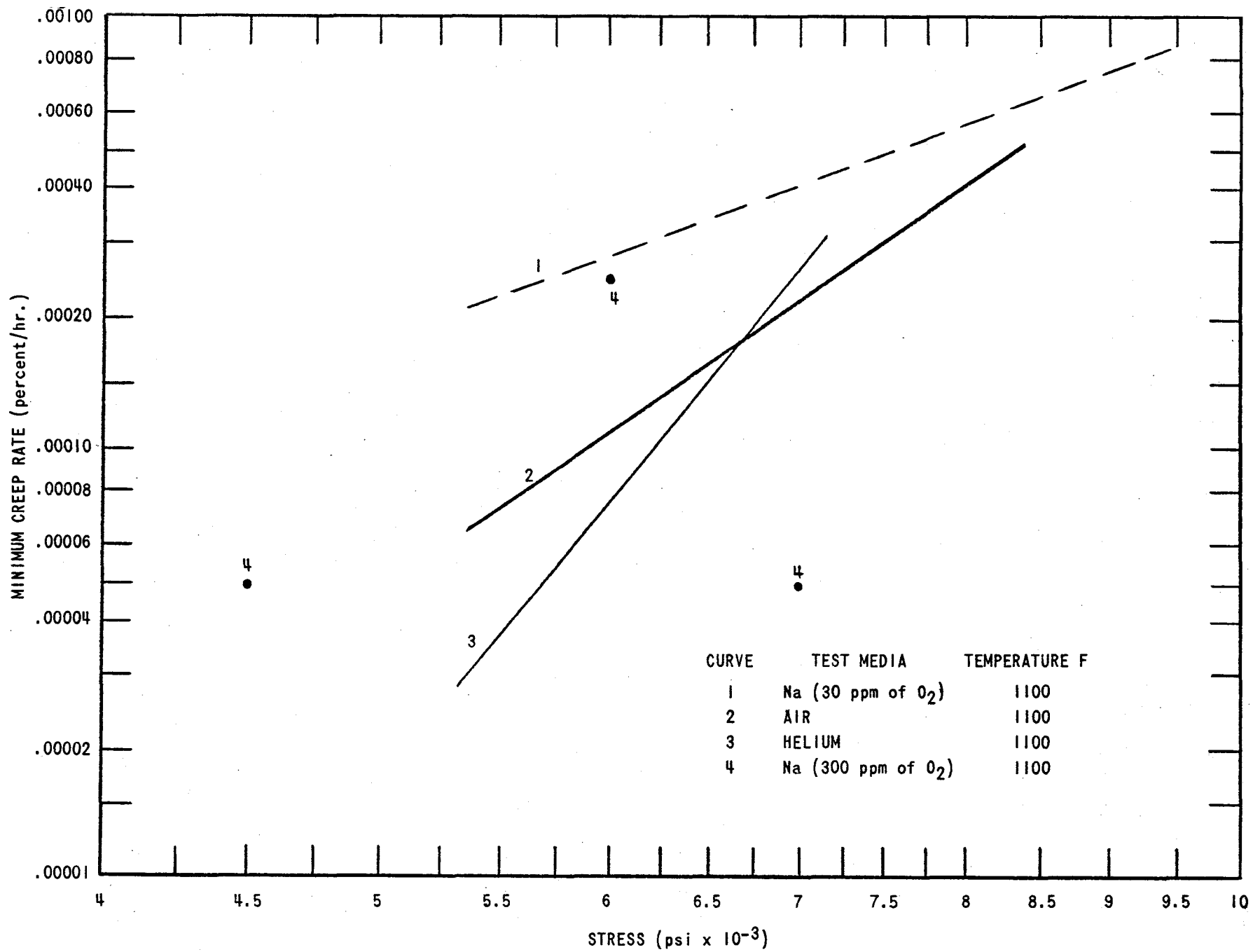


FIG. 10 - MINIMUM CREEP RATE vs STRESS, 2 1/4 Cr-1 Mo CARBON STEEL SPECIMENS

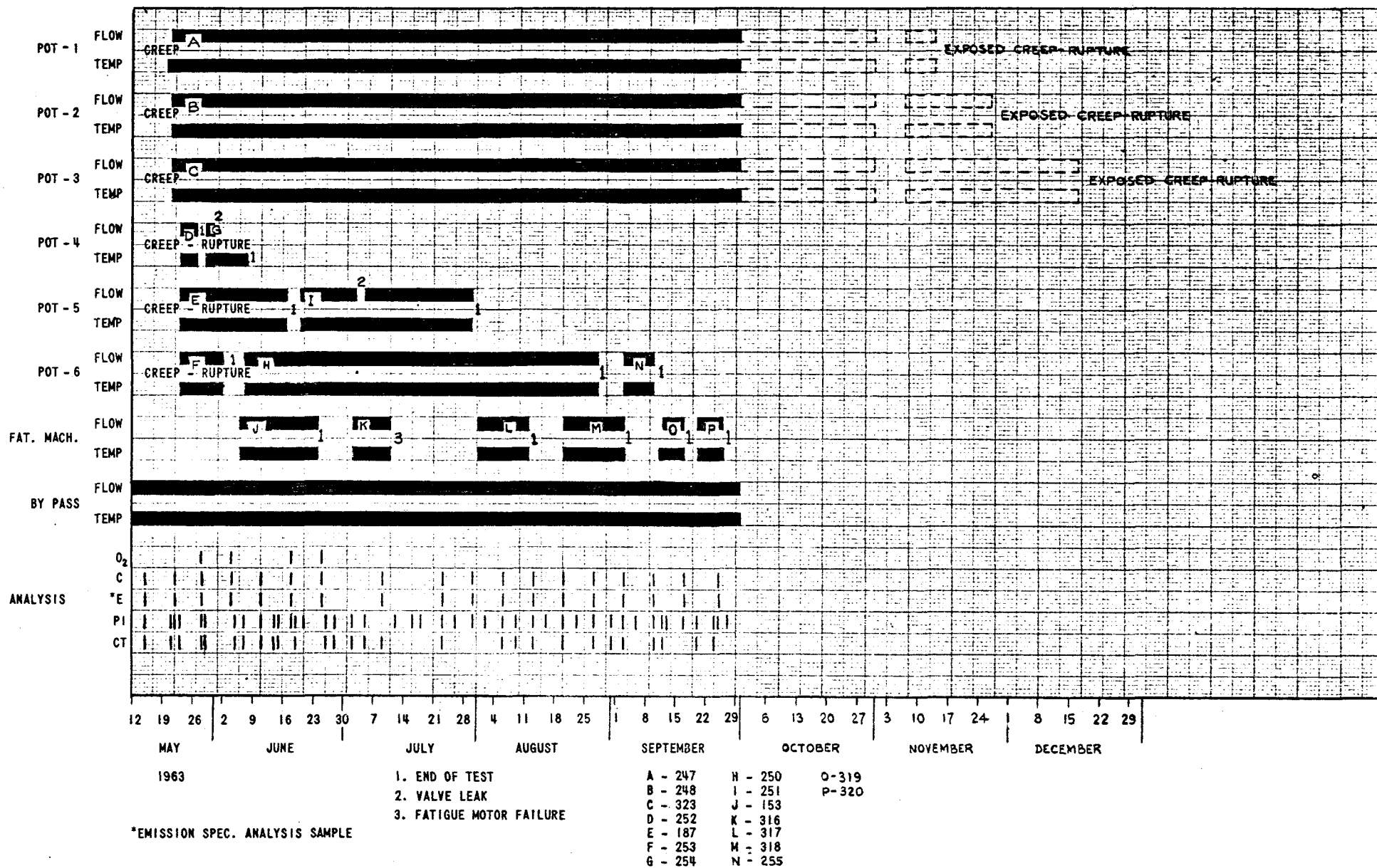


FIG. 11 - OPERATIONAL HISTORY OF LOOP 2 - TEST 4 (Cr-Mo TEST SPECIMENS)

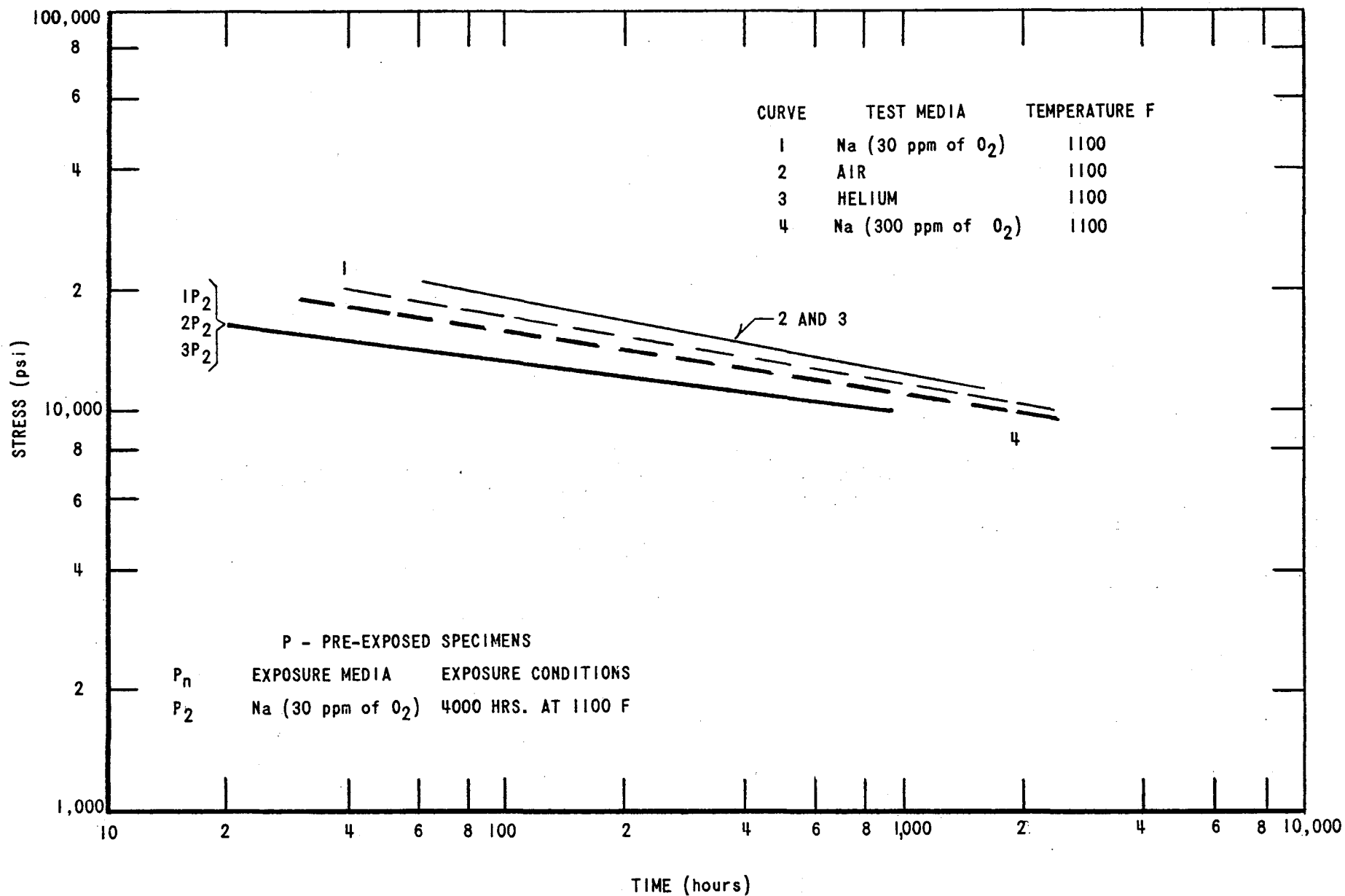


FIG. 12 - CREEP TO RUPTURE OF 2 1/4 Cr-1 Mo CARBON STEEL SPECIMENS

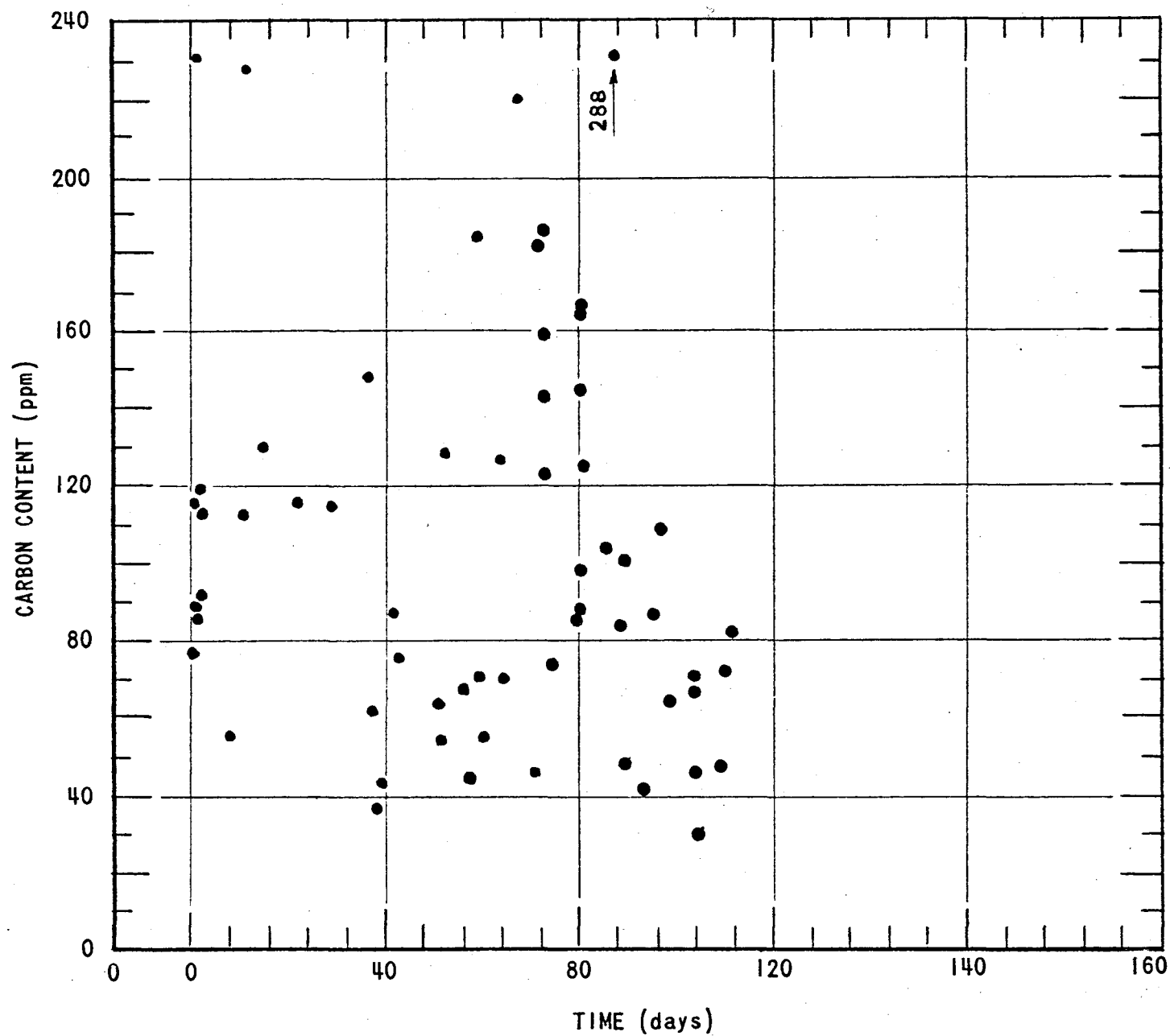


FIG. 13 - CARBON CONTENT, LOOP 1, TEST 3
(SS TEST SPECIMENS)

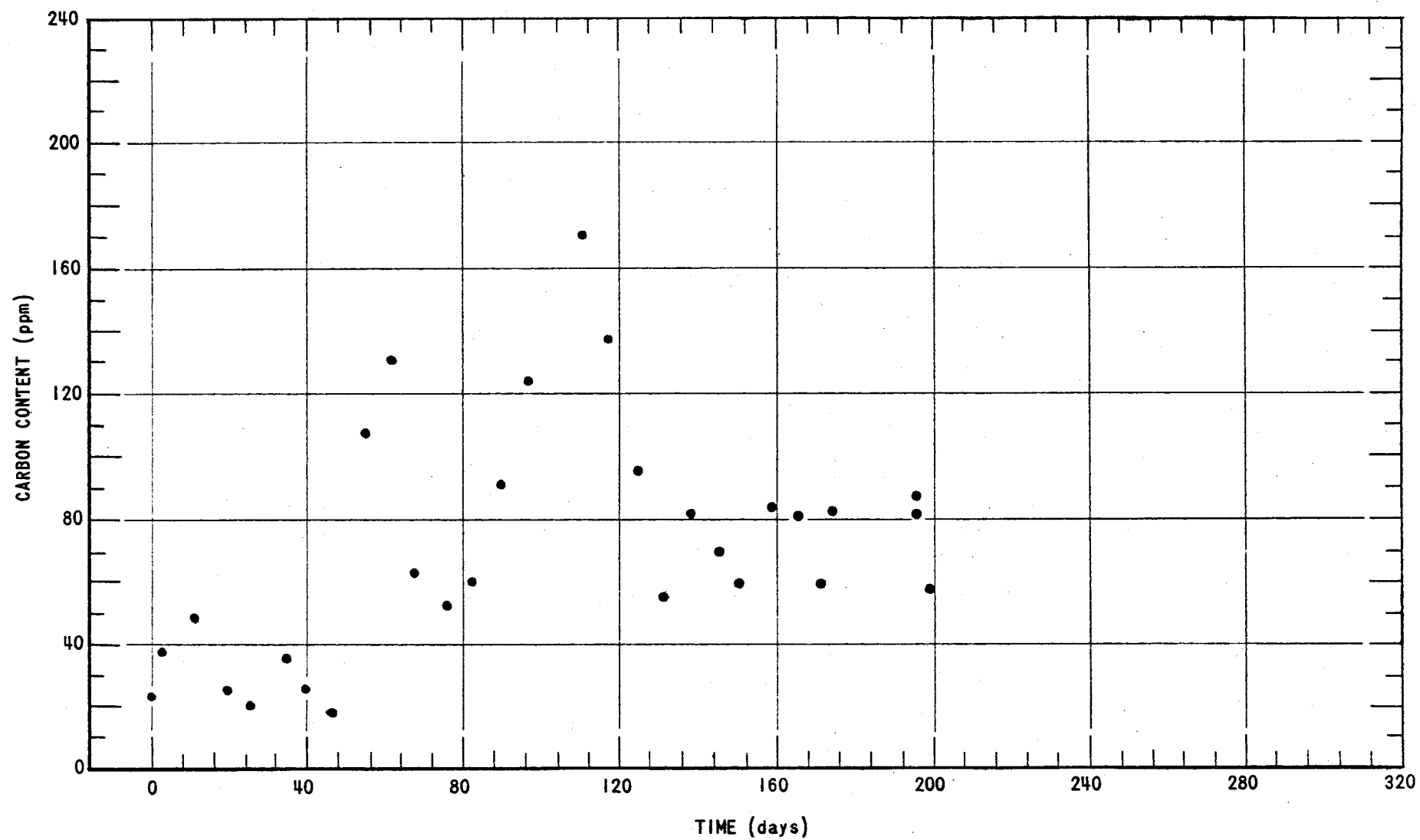


FIG. 14 - CARBON CONTENT, LOOP 2, TEST 4 (Cr-Mo TEST SPECIMENS)

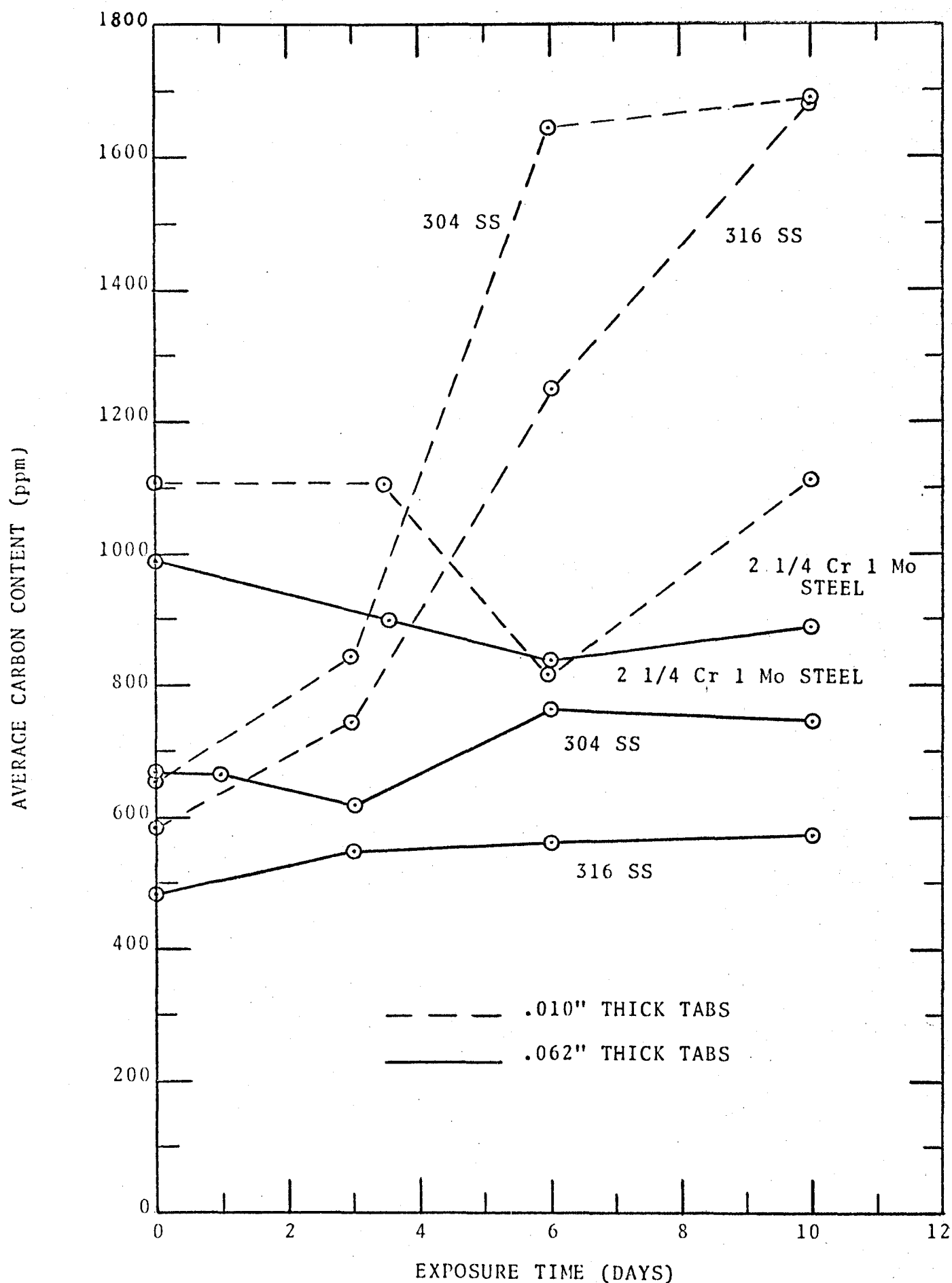


FIG 15 - CARBON CONTENT OF TABS EXPOSED TO 1100°F SODIUM (200-300 ppm oxygen)