MSAR 63-105

PROGRESS REPORT 34
June 1963

to

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

EFFECT OF 1200°F SODIUM ON AUSTENITIC AND FERRITIC STEELS

Physical Properties of Materials

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

July 24, 1963

MSA Research Corporation

Subsidiary of Mine Safety Appliances Company

Callery, Pennsylvania

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July 24, 1963

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SUMMARY

Tests 1 and 2, which consisted of the determination of the physical properties of Type 316 ss and 2 1/4 Cr-1 Mo steel specimens in low oxide sodium, helium and air at 1200 F and 1100 F respectively, have been completed except for several tests in helium.

Metallurgical examination of the specimens generated under Tests 1 and 2 is continuing. Photomicrographs and discussion of stainless steel fatigue and creep-to-rupture specimens included in this report indicate that while massive oxidation products on stainless steel fatigue specimens are not apparent, as in the case of the Cr-Mo specimens, surface attack is thought to reduce fatigue life. In the case of the stainless steel stress rupture specimens, evidence is shown that carburization is greater in short time stressed specimens than in 4000 hour exposed unstressed specimens. A topical report on the findings of Tests 1 and 2 is in progress.

Test 3, the determination of the physical properties of stainless steel in 1200 F sodium saturated with carbon is in its shakedown stage. Trouble has been experienced in establishing a constant carbon operating level prior to evaluating the effectiveness of the carbon saturating bypass system.

Test 4, the measurement of the physical properties of 2 1/4 Cr-1 Mo steel specimens in sodium containing 200 to 300 ppm sodium oxide, is in progress with several of the short time creep-to-rupture tests completed. Very preliminary evaluation of the test to date indicates a lower creep-to-rupture strength and a lower creep rate in this environment compared to sodium with low oxide.

Progress Report 34

EFFECT OF 1200 F SODIUM ON AUSTENITIC AND FERRITIC STEELS

PHYSICAL PROPERTIES OF MATERIALS

1. INTRODUCTION

MSA Research Corporation and the University of Michigan are in the process of completing the final helium tests and examining the specimens generated under Tests 1 and 2. At the same time, Test 3 began 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 OXIDE SODIUM, AIR AND HELIUM

The following tests have been completed:

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

A creep-to-rupture test of a 316 ss specimen in helium at a load of 17,750 psi is still in progress at 2609 hours.

Two creep tests are still in progress in helium. The test at 11,000 psi has decreased to a rate of 0.18%/1000 hrs after 1600 hours of testing, while the test at 10,500 psi has decreased to a rate of 0.08%/1000 hrs after 1600 hours of testing. A third test at 12,000 psi has been terminated after 4008 hours. It exhibited a rate of 0.23%/1000 hrs at 800 hours, then began increasing to a termination rate of 0.48%/1000 hrs.

All data from Test 1 is found in Table 1. Preliminary conclusions, along with plots of the test results, were included in last month's progress report (No. 33).

Results of part of the metallurgical examination of stainless steel specimens from Test 1 are included under Section 4.1 of this report.

2.2 TEST 2 - 2 1/4 Cr-1 Mo STEEL SPECIMENS IN 1100 F, LOW OXIDE SODIUM, AIR AND HELIUM

The following tests have been completed:

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

Two creep tests in helium are still in progress. The test at 5500 psi has decreased to a rate of 0.035%/1000 hrs after 2800 hours. The test at 7000 psi has decreased to a rate of 0.245%/1000 hrs after 1800 hours of test.

The tensile data for specimens 2BHX-3, -4 and -5 were reported erroneously in Progress Report 33, Table 3. This correction and the additional data to date are found in Table 1. Preliminary conclusions, plots of the test results and the results of the metallurgical examinations of the Cr-Mo specimens were included in last month's progress report (No. 33).

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

Circulation continued this month through all but one test pot. The outlet line heater for this unit burned out and appeared to have shorted out to the flexible line. It was decided to isolate this pot until this line could be replaced and continue shakedown runs with the rest of the system.

The oxide level was lowered to below a saturation temperature of 300 F and several sodium samples were taken to determine the carbon level of the system prior to circulation through the bypass carbon tank. During this time, periodic checks of the oxide content indicated that the saturation temperature was increasing from less than 300 F to 600-700 F in a three or four day period. The scatter in the carbon analysis was too great to indicate a satisfactory starting point, so the system was cold trapped continuously for 20 hours in an attempt to clean up the system. Additional sodium samples have been taken for carbon analysis. The results and discussion of the analytical results are found under Section 5.

Upon determination of the carbon level in the system, the carbon bypass tank will be put into operation and the test conditions obtained. Foil samples will then be installed for a 2-3 week period. After data obtained from these foils has been correlated, Test 3 will be initiated.

2.4 TEST 4 - 2 1/4 Cr-1 Mo STEEL SPECIMENS IN 1100 F, HIGH OXIDE SODIUM

Testing of the three creep specimens and the creep-rupture specimens continued. Fatigue tests were initiated during this report period. The operational history of Loop 2 during Test 4 is shown to date in Fig. 1.

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

A 2 1/4 Cr-1 Mo steel fatigue specimen was installed in the test machine in which a 12 in. radius mandrel was used for a low cyclic strain test. This specimen failed after 63,270 cycles. This one test is in good agreement with the fatigue tests in low oxide sodium.

Twelve fatigue specimens were exposed to 1100 F, high oxide sodium for 406 hours for future testing in air, helium and high oxide sodium.

Additional fatigue tests will be run at the low strain level during the next report period. Detailed data from the fatigue tests, along with graphs, will be presented as more data is generated.

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

Two additional creep-to-rupture tests in the high oxide sodium failed during this report period. The specimen at a load of 12,000 psi failed after 588.7 hours. The test pots in which a specimen was loaded at 16,000 psi developed a leak in the bellows of the outlet valve after 65 hours of testing. The test pot was isolated from the rest of the system and, in an attempt to complete the run, the test was continued. However, with no flow, a temperature drop of 40-50 degrees from the 1100° test temperature developed across the test specimen. The test was continued until failure at 234.4 hours, which is not in agreement with other tests of this series. Consideration will be given to repeating this test at a later date.

Based on the test results to date, a preliminary curve was made and the final two stress values of 10,500 psi and 9500 psi were chosen. These two final creep rupture tests are now in progress.

Data to date is found in Table 2 and is shown in Fig. 2 plotted against curves obtained from Test 2 (low oxide sodium) for comparison. From the three points to date, it appears that the creep-rupture curve for high oxide sodium will be parallel but lower than the low oxide curve determined in Test 2. The specimen elongations, as taken from externally mounted dial gages, are plotted against time in Fig. 3.

2.4.3 2 1/4 Cr-1 Mo Steel Creep Tests

The creep tests of the three 2 1/4 Cr-1 Mo specimens continued at loads of 7000 psi, 6000 psi and 4500 psi. These tests have been in progress approximately 1000 hours.

The creep rate of the 7000 psi specimen decreased to 0.29%/1000 hrs at 400 hours and has held constant since that time. The preliminary curve is plotted in Fig. 4 and 5 with data obtained from Test 2 for comparison.

The creep rate of the 6000 psi specimen has decreased to 0.25%/1000 hrs after 400 hours and has decreased slightly since then. The preliminary curve is plotted in Fig. 5 with data obtained from Test 2 for comparison.

The creep rate of the 4500 psi specimen has had a general decrease to 0.05%/1000 hrs at the present time and may decrease further. The preliminary curve is plotted in Fig. 6 with data obtained from Test 2 for comparison.

From the preliminary data, 2 1/4 Cr-1 Mo steel tested in high oxide sodium exhibits a lower creep rate for short time tests than those tested in low oxide sodium. These creep rates may change as the test progresses to the 4000 hour time limit. Although the creep rates in high oxide sodium are lower than in low oxide sodium, they are still higher than in air or helium.

2.4.4 2 1/4 Cr-1 Mo Tensile Tests

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

3. THERMODYNAMIC CONSIDERATIONS

T. Ciarlariello

Whether oxidation of a metal in a given environment can proceed can be found by computing the free energy change of the oxidation reaction. If the free energy change is negative, the reaction can occur; if the change is positive, the reaction will not occur. It should be noted that the free energy change does not indicate the rapidity of oxidation, but only indicates if the oxidation is possible. Temperature and the presence of catalysts can greatly affect reaction rates. The nature of the oxidation products also changes the reaction rates. Metals such as stainless steel have a tight thin adherent oxide coating. Steel, by contrast, has a looser oxide coat. This difference is responsible for the more rapid oxidation of iron and the slower oxidation of stainless steel.

The reaction will proceed until the quantity of oxygen in the oxidizing media drops to a low value. If the media contains but a small quantity of oxygen, the oxide produced will be vanishingly small.

The free energy of formation of various metals in the standard state can be found in Coughlin.[1] If the products and reactants are not present in the standard state, the free energies will be different. In order to calculate the true free energy of formation, some assumptions have to be made about the state of the metals and oxides. The following assumptions were made in preparing Table 3:

A. The metals form ideal solutions, miscible in all proportions.

B. The oxides form discrete phases, with zero solubility in the metal, and with no metal dissolved in the oxide.

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1. Coughlin, James P., Contributions to the Data on Theoretical Metallurgy, Bulletin 542, U. S. Bureau of Mines

In accordance with the first assumption, the free energies can be corrected by the equation:

$$\Delta F_m = -\frac{RT}{N} \ln C \qquad (1)$$

where

R = gas constant
T = temperature
N = number of atoms of oxygen in metal oxide
C = metal concentration, mole fraction
ΔF_m = metal correction to the free energy, per gm atom of oxygen

The solid metal oxides require no correction since they are in the standard state. The gaseous metal oxides require a correction if they are not present at a 1 atmosphere partial pressure.

$$\Delta F_p = -\frac{RT}{N} \ln \frac{1}{P} \qquad (2)$$

where

ΔP = partial pressure of product, atmospheres

Equation 2 can also be used to calculate the free energy change if the oxygen is not present at 1 atmosphere pressure.

For Na_2O in sodium, the change in free energy due to dilution is given by

$$\Delta F_{O_2} = -RT \ln \frac{C}{C_s}$$

where

C = mole fraction of Na_2O in Na
C_s = mole fraction of Na_2O in saturated Na

This change can then be algebraically added to the free energy of Na_2O to obtain the free energy of oxygen in dilute Na_2O. The ΔF change due to the sodium is negligible because the Na_2O is dilute.

The free energy of reaction of a metal becoming metal oxide can then be found by summing the free standard state free energy and the corrections given by equations (1) and (2).

$$\Delta F = \Delta F^\circ + \Delta Fm + \Delta Fp$$

where

ΔF° is the standard state free energy.

The free energies of metal and oxygen can then be listed in order of descending free energy (on a gm atom of oxygen basis). Oxides listed high in the table will oxidize metals low in the table, and the ΔF of reaction will be the difference between the two listed free energies of the table.

Caution should be used when the two values of free energy are close. The standard free energies of formation given by Coughlin are given to $\pm$ 1 K cal. Therefore, the error in Table 3 is $\pm$ 1 K cal, and when two numbers are subtracted, the error can be $\pm$ 1.4 K cal. Therefore, for free energy differences of the order of -1.4 K cal, the reaction may not proceed. Similarly, for differences of the order of +1.4 K cal, the reaction may still proceed.

4. METALLURGICAL RESULTS

F. Tepper
K. W. Reber
J. W. Freeman (University of Michigan)

4.1 STAINLESS STEEL FATIGUE SPECIMENS - TEST 1

Figures 7 and 8 show respectively specimens tested in air and helium at the same cyclic strain. Surface initiated fatigue cracking is evident in both photomicrographs. These cracks appear to be predominantly transgranular, and are similar in nature to fatigue cracking observed in 2 1/4 Cr-1 Mo air tests. The presence of massive air corrosion product observed within fatigue cracks associated with croloy air tests are not visible in stainless steel air fatigue tests. Microcracks are also apparent in both figures. These microcracks which exist throughout the matrix appear to be intergranular and generally parallel to the stress direction.

Figure 9 shows fatigue cracking in a sample that had been pre-exposed to sodium, water washed and tested in air. Cracking appears to be more intergranular than in the case of specimens not pre-exposed and suggests caustic attack. Incomplete alkali removal during the water wash step is a possible cause of caustic attack and subsequent reduction in cycle life (approximately 50%) of pre-exposed air test specimens as compared to unexposed air test specimens.

Figures 10, 11, and 12 show specimens fatigue tested respectively in air, helium and sodium at low cyclic strain. Surface cracking is apparent in the air test, and absent in helium or sodium tests. Surface cracking becomes less predominant with decreasing cyclic strain when specimens are tested in air, and become virtually non-existent in He and Na tested specimens.

While photomicrographs of ruptures are not shown, a small degree of plastic deformation is evidenced in short term high cyclic strain specimens. The plastic deformation at the rupture face is more shallow (2-3 grain diameters from fracture) and not as pronounced as that observed with croloy specimens.

Surface-initiated fatigue cracking is apparent after testing in each of the environments, but is more pronounced in air tests and at high cyclic strain. Evaluation of fatigue test data shows, in general, increasing fatigue resistance from air to sodium or helium. While massive oxidation products on stainless steel are not apparent, as in the case of croloy, air oxidation is thought to reduce fatigue life. Comparison of thermodynamic values for metal oxides would suggest that stainless steel is slightly more effective than croloy as an oxygen scavenger for sodium. Thus, oxide coatings are prevented from forming on croloy but superficial oxide coatings are known to occur on stainless steel when immersed in sodium. These factors could explain the similarity of sodium and helium fatigue data, while the air life is significantly reduced.

4.2 STAINLESS STEEL STRESS TO RUPTURE - TEST 1

Figure 13 shows a longitudinal section at the failure of a stainless specimen stressed to rupture in air at 1200°F. Examination of the intergranular separation near fracture shows greater air oxidation production than surfaces where cracking did not occur. Figures 14, 15, and 16 show longitudinal sections of the failure for air, helium and sodium tests. An intergranular mode of fracture is evidenced in each case. Twinning is particularly pronounced in Figure 15 which had undergone more strain than the sample shown in Figures 14 or 16.

Figures 17-21 show longitudinal sections of stainless samples stressed to rupture in 1200°F sodium. Each of the samples were etched with ferric chloride to define carbides. An increase in the carbide case is evident with increasing time of exposure to sodium. Table 4 describes test conditions for each of the samples and shows results of carbon analysis for each sample. Also included are optical measurements of carbide affected zones.

Examination of this series of photomicrographs shows an expected increase in grain elongation with increasing stress. Intergranular cracks are evident in each case, and in Figures 19 and 20 considerable carburization is evident in the region of a crack. The large crack shown in Figure 21 apparently originated at a time near complete failure of the specimen. Carbide precipitation appears to have occurred both at grain boundaries and along dislocation concentrations. Carbide precipitation is also pronounced at twin planes. A phase, as yet unidentified, is evident at grain boundaries in the heavily carburized region (Figures 20 and 21).

Figure 22 shows a sample exposed to 1200°F sodium for 4000 hours with no stress applied. A carbide case is hardly apparent. Figures 23-26 show longitudinal sections of stainless steels pre-exposed to 1200°F sodium for 4000 hours and then stressed to rupture in 1200°F sodium. Table 4 also describes test conditions for this series of samples.

A striking comparison can be made of carbide penetration between stressed samples and those pre-exposed and subsequently stressed. The extent of visible carburization appears to be essentially dependent upon the duration that a sample is under stress. A sample under stress for as little as 144 hours appears to be carburized to the same extent as a sample exposed to sodium for 4000 hours with no stress.

Carbon analyses of specimens shown in Figures 17-26 were performed and are shown in Table 4. The specimens for carbon analyses were removed above the gage length and represent areas at stress levels 40% of the stress applied to the gage length. Analyses were performed without attempting to remove the carburized layer from the specimen which was originally 0.062 in. thick.

Figure 27 is a plot of carbon content versus exposure time, and the effect of stress on increase of carburization rate is immediately apparent. In samples tested without prior sodium exposure, the carburization rate increases with time and no maximum carbon value is attained over the range examined. The increase in carbon content for a sample exposed unstressed is very low, but upon initiation of stressed conditions the carburization rate is most rapid, reaches a peak, and decreases.

While the data shown in Figure 27 represents carbon content at stress levels lower than that in the gage length, metallographic evaluation within the gage length augments the trends suggested by carbon analyses. The evidence suggests that stress significantly affects carburization rate. Quantitative evaluation of this effect and explanation of the anomaly apparent in Figure 27, can only be resolved by carbon analysis of specimens within the gage length. Further evaluation of this effect is planned, which will include repeat analyses of 4000 hour unstressed specimens.

5. ANALYTICAL RESULTS

S. J. Rodgers
J. C. Gerken

5.1 LOOP ANALYSIS

Extraction of samples from Loop 2, Test 4, on a weekly basis has continued. Analyses have been run for carbon, oxygen and for other elements by emission spectrograph. The results of emission spectrograph samples are shown in Table 5, and the carbon and oxygen results are as follows:

Date	Carbon Content (ppm C)	Oxygen Content (ppm O)
*3/22/63	22.2	
*3/25/63	33.9	
*4/1/63	48.2	
*4/9/63	25.4	
*4/5/63	20.2	
*4/24/63	35.4	
*4/30/63	25.0	
*5/7/63	18.0	
*5/15/63	--	
*5/22/63	130	
5/28/63	63	2454 (sample contaminated)
6/4/63	52	424
6/11/63	59	252
6/18/63	--	356
6/25/63	--	319

* Previous results.

Results of oxygen samples analyzed by the amalgamation procedure agree well with values determined during plugging indicator runs. Additional samples for oxygen analyses are not planned but samples will be taken if loop operation should require them. Results of emission spectrograph analyses will be reported when received.

Samples have been extracted from Loop 1 for carbon analysis and the results are as follows:

Date	Carbon Content (ppm C)
6/17/63	77
6/18/63	85
	232
	114
	88
6/19/63	88
	119
	113

Results have shown a wide variation in carbon content from sample to sample. Black particles have been observed on many of these samples. It is recommended that loop operation not be initiated until the carbon level has stabilized.

5.2 PARTICULATE CARBON

Additional filtered samples from the sodium pot are planned. Another filter and tube for the extraction of a sample are being prepared and the sample will be extracted and analyzed in July.

5.3 ROUND ROBIN CARBON

Analysis of sodium from Set 1A which represents as-received sodium which has been cold trapped to a plugging temperature of lower than 300°F has been completed. The sample tube which was received was cut into 10 sections and numbered consecutively 10 through 19. Results are as follows:

Sample Number	Carbon Content (ppm C)
10	78.0
11	50.8
12	32.4
13	70.7
14	26.1
15	45.0
16	168.0
17	62.8
18	35.2
19	450.0

Results from this cold trap sodium, as compared to the first set of samples which were hot trapped, show a considerably greater spread of values. It is felt that this spread is due to characteristics of the sample rather than analytical variations, but another sample has been requested and will be analyzed in the same fashion.

6. FUTURE WORK

Examination and evaluation of the specimens generated under Tests 1 and 2 will continue. Topical reports covering this period of the work will be forthcoming.

Stabilization of the carbon concentration in Loop 1 for the start of Test 3 will be given a high priority during the next report period.

Test 4 will continue as scheduled.

Table 1 - Test Data Summary
Tests 1 and 2

A. Fatigue Tests

316 ss	Condition	Specimen Thickness In.	% Cyclic Strain	Cycles to Failure	Time at Temperature Hrs
3FAX2	Air-1200 F	.0680	2.18	599	14.7
3FAX3	Air-1200 F	.0680	2.18	600	15.5
3FAX4	Air-1200 F	.0680	2.18	570	16.6
3HAL1	(1)Air-1200 F	.0660	2.11	296	1.8
3HAL2	(1)Air-1200 F	.0663	2.12	302	2.5
3HAL3	(1)Air-1200 F	.0662	2.12	326	2.0
3FHX4	He-1200 F	.0665	2.13	1325	17.7
3FHX5	He-1200 F	.0685	2.19	848	5.5
3FHX6	He-1200 F	.0670	2.14	1122	7.5
3HHL1	(1)He-1200 F	.0665	2.13	1116	16.8
3HHL2	(1)He-1200 F	.0675	2.16	1233	7.0
3HHL3	(1)He-1200 F	.0660	2.11	749	4.5
3FLX1	Na-He-1200 F	.0675	2.16	615	17.5
3FLX2	Na-He-1200 F	.0680	2.18	275	2.0
3FLX3	Na-He-1200 F	.0678	2.16	492	20.0
3FLX4	Na-He-1200 F	.0675	2.16	475	14.0
3HLL1	(1)Na-He-1200 F	.0680	2.18	700	21.8
3HLL2	(1)Na-He-1200 F	.0675	2.16	751	42.0
3HLL3	(1)Na-He-1200 F	.0675	2.16	545	65.5
3GAX7	Air-1200 F	.0680	1.00	1498	8.5
3GAX8	Air-1200 F	.0680	1.00	1663	9.5
3GAX9	Air-1200 F	.0675	1.00	1500	8.5
3GHX2	He-1200 F	.0680	1.00	9693	63.0
3GHX3	He-1200 F	.0670	0.99	9058	51.5
3GHX4	He-1200 F	.0678	1.00	7816	59.5
3GLX1	Na-He-1200 F	.0665	.985	2385	32.5
3GLX2	Na-He-1200 F	.0675	1.00	2110	22.5
3GLX3	Na-He-1200 F	.0672	.995	3914	23.5
3GLX4	Na-He-1200 F	.0682	1.01	4112	44.0

(1) Specimen pre-exposed to 1200 F sodium for 286 hours

Table 1 - Test Data Summary - Cont'd

316 ss	Condition	Specimen Thickness In.	% Cyclic Strain	Cycles to Failure	Time at Temperature Hrs
3JAX5	(4) Air-1200 F	.0665	.554	8956	49.8
3JAX6	(4) Air-1200 F	.0682	.568	8060	44.8
3JAX7	Air-1200 F	.0678	.565	8556	48.0
3JHX1	He-1200 F	.0670	.558	50,004	277.8
3JHX2	He-1200 F	.0685	.571	38,804	217.5
3JHX3	He-1200 F	.0688	.573	37,011	230.5
3JLX1	Na-He-1200 F	.0680	.565	55,925	333.5
3JLX2	Na-He-1200 F	.0668	.556	29,400	160.0
3JLX3	Na-He-1200 F	.0670	.558	33,055	212.5
2-1/4 Cr-1 Mo					
2FAX1	Air-1100 F	.0685	2.15	474	16.5
2FAX2	Air-1100 F	.0675	2.16	392	16.5
2FAX3	Air-1100 F	.0678	2.17	365	28.2
2HAL1	(3) Air-1100 F	.0670	2.14	353	2.5
2HAL2	(3) Air-1100 F	.0670	2.14	371	2.5
2HAL3	(3) Air-1100 F	.0670	2.14	449	2.8
2FHX5	He-1100 F	.0670	2.14	1725	15.8
2FHX6	He-1100 F	.0672	2.15	2360	10.5
2FHX7	He-1100 F	.0670	2.14	2440	14.3
2HHL1	(3) He-1100 F	.0675	2.16	843	5.0
2HHL2	(3) He-1100 F	.0675	2.16	2129	11.8
2HHL3	(3) He-1100 F	.0675	2.16	196	2.0
2HHL4	(3) He-1100 F	.0672	2.15	1614	20.0
2HHL5	(3) He-1100 F	.0668	2.14	999	7.5
2FLX1	Na-He-1100 F	.0672	2.15	1931	34.0
2FLX2	Na-He-1100 F	.0675	2.16	2134	41.5
2FLX3	Na-He-1100 F	.0672	2.15	2280	17.5
2HLL1	(3) Na-He-1100 F	.0675	2.16	2448	33.8
2HLL2	(3) Na-He-1100 F	.0672	2.15	1770	21.5
2HLL3	(3) Na-He-1100 F	.0672	2.15	2140	25.5

(3) Specimen pre-exposed to 1100 F sodium for 323 hours.
(4) Specimens 3JAX3 and 3JAX4 tested on different mandrel design have been voided.

Table 1 - Test Data Summary - Cont'd

2-1/4 Cr-1 Mo	Condition	Specimen Thickness In.	% Cyclic Strain	Cycles to Failure	Time at Temperature Hrs
2GAX1	Air-1100 F	.0675	1.00	998	5.8
2GAX2	Air-1100 F	.0675	1.00	1011	5.5
2GAX3	Air-1100 F	.0672	.995	1017	6.0
2GHX1	He-1100 F	.0675	1.00	12,400	101.5
2GHX2	He-1100 F	.0672	.997	10,388	58.3
2GHX3	He-1100 F	.0665	.985	9,878	63.0
2GLX1	Na-He-1100 F	.0672	.995	6727	94.0
2GLX2	Na-He-1100 F	.0672	.995	7300	42.0
2GLX3	Na-He-1100 F	.0672	.995	9249	64.5
2JAX1	Air-1100 F	.0668	.556	2710	16.5
2JAX2	Air-1100 F	.0665	.554	2768	16.5
2JAX3	Air-1100 F	.0665	.554	2850	16.0
2JHX1	He-1100 F	.0675	.562	18,454	104.0
2JHX2	He-1100 F	.0675	.562	32,390	185.8
2JHX3	He-1100 F	.0665	.554	56,522	326.0
2JHX4	He-1100 F	.0665	.554	43,200	254.0
2JLX1	Na-He-1100 F	.0672	.560	44,458	305.0
2JLX2	Na-He-1100 F	.0668	.556	42,555	256.0
2JLX3	Na-He-1100 F	.0672	.560	53,400	323.0

B. Tensile Tests

316 ss	Condition	Tensile Str. (Psi)	0.2% Offset Yield Strength (Psi)	Elong %	Reduction of Area %
3BAX4	Air-1200 F	49,400	26,100	43	47
3BAX3	Air-1200 F	50,250	25,650	41	48
3BAX1	Air-1200 F	51,200	26,300	47	44
3BHX1	He-1200 F	47,200	26,600	46	58
3BHX2	He-1200 F	47,500	25,900	52	54
3BHX3	He-1200 F	48,600	25,300	44	53
3BHL1C	(5)He-1200 F	48,850	24,500	39	46
3BHL2C	(5)He-1200 F	46,900	24,800	52	52
3BHL3C	(5)He-1200 F	50,000	26,200	41	46

Table 1 - Test Data Summary - Cont'd

316 ss	Condition	Tensile Str. (Psi)	0.2% Offset Yield Strength (Psi)	Elong %	Reduction of Area %
3BHL1U	(11)He-1200 F	49,100	26,750	41	56
3BHL2U	(11)He-1200 F	46,400	25,200	46	55
3BHL3U	(11)He-1200 F	49,900	25,650	46	51
3BHX4	(5)He-RT	93,200	44,600	47	46
3BHX5	(5)He-RT	92,000	43,750	48	50
3BHX6	(5)He-RT	92,500	42,000	46	40
3BHX7	(5)He-RT	91,400	43,700	49	52
3BHX8	(5)He-RT	92,500	43,900	49	52
3BHX9	(5)He-RT	95,500	45,600	46	40
3BHX10	He-RT	89,500	49,700	65	59
3BHX11	He-RT	88,500	49,400	66	59
2-1/4 Cr-1 Mo					
2BAX4	Air-1100 F	49,170	25,800	28	64
2BAX1	Air-1100 F	56,800	28,800	32	57
2AAX4	Air-1100 F	59,400	29,370	32	62
2BHX3	He-1100 F	39,700	25,000	26	63
2BHX4	He-1100 F	38,120	22,600	31	44
2BHX5	He-1100 F	37,820	23,500	41	74
2BHL1C	(6)He-1100 F	26,620	17,200	45	54
2BHL2C	(6)He-1100 F	25,230	(10) --	46	55
2BHL3C	(6)He-1100 F	26,580	16,800	47	41
2BHL4C	(6)He-1100 F	26,160	17,300	41	50
2BHL1U	(12)He-1100 F	25,800	16,150	40	65
2BHL2U	(12)He-1100 F	26,410	17,950	42	56
2BHL3U	(12)He-1100 F	29,200	19,400	38	57

Table 1 - Test Data Summary - Cont'd

C. Creep Tests

316 ss	Condition	Stress Psi	Rate %/1000 hrs
3AAX2	Air-1200 F	13,000	0.215 Terminated 3692 hrs
3BAX2	Air-1200 F	12,500	0.15 Terminated 4242 hrs
3AAX3	Air-1200 F	11,500	0.035 Terminated 3986 hrs
*3AHX1	He-1200 F	12,000	0.48 Terminated 4008 hrs
*3AHX2	He-1200 F	11,000	0.18 (IP 1914 hrs)
*3AHX3	He-1200 F	10,500	.080 (IP 1769 hrs)
3ALX1	Na-He-1200 F	13,500	0.67 Terminated 4000 hrs
3ALX2	Na-He-1200 F	12,500	0.31 Terminated 4000 hrs
3ALX3	Na-He-1200 F	11,500	0.17 Terminated 4000 hrs

2-1/4 Cr-1 Mo	Condition	Stress Psi	Rate %/1000 hrs
2BAX2	Air-1100 F	8,000	.395 Terminated 3957 hrs
2AAX2	Air-1100 F	6,000	.12 Terminated 4010 hrs
*2AAX1	Air-1100 F	5,500	.07 Terminated 4000 hrs
2AHX1	He-1100 F	6,000	.085 Terminated 4009 hrs
*2AHX2	He-1100 F	5,500	.035 (IP 2921 hrs)
*2AHX3	He-1100 F	7,000	.245 (IP 1913 hrs
2ALX1	Na-He-1100 F	10,000	**Terminated 1839 hrs
2ALX2	Na-He-1100 F	8,000	**Terminated 4000 hrs
2ALX3	Na-He-1100 F	6,000	.28 Terminated 4000 hrs
2ALX4	Na-He-1100 F	8,000	.57 Terminated 2587 hrs

D. Stress-Rupture Tests

316 ss	Condition	Stress (Psi)	Elong %	Reduction of Area %	Rupture Time (Hrs)
3CAX10	Air-1200 F	27,500	61	45	173.6
3CAX2	Air-1200 F	27,500	58	49	152.8
3CAX9	Air-1200 F	27,500	52	46	146.5
3CAX3	Air-1200 F	27,500	63	50	143.0
3DAX4	Air-1200 F	24,000	40	38	476.2
3CAX6	Air-1200 F	24,000	60	43	426.2
3CAX7	Air-1200 F	24,000	44	39	349.7
3CAX1	Air-1200 F	22,000	33	31	863.0
3CAX8	Air-1200 F	21,500	33	28	1000.6
3CAX5	Air-1200 F	21,500	35	38	971.0
3DAX3	Air-1200 F	20,500	35	29	1387.8
3DAX1	Air-1200 F	18,500	34	29	2363.0

Table 1 - Test Data Summary - Cont'd

316 ss	Condition	Stress (Psi)	Elong %	Reduction of Area %	Rupture Time (Hrs)
3DAX2	Air-1200 F	17,750	39	20	1969.5(9)
3AAX4	Air-1200 F	17,750	18	18	3695.1
3EAL1	(5)Air-1200 F	27,500	62	46	144
3EAL2	(5)Air-1200 F	24,000	54	44	574
3EAL3	(5)Air-1200 F	21,500	49	42	985.2
3CAX4	He-1200 F	27,500	63	46	166.3
3CHX2	He-1200 F	27,500	53	49	152.8
3CHX3	He-1200 F	24,000	48	49	697.7
3DHX3	He-1200 F	24,000	65	54	483.1
3CHX1	He-1200 F	21,500	52	47	894.3
3DHX1	He-1200 F	20,000	34	37	1509.5
3DHX2	He-1200 F	18,000	44	32	2619.9
*3DHX4	He-1200 F	17,750			IP (2609)
3EHL1	(5)He-1200 F	27,500	57	52	57.6
3EHL2	(5)He-1200 F	24,000	48	44	675.0
3EHL3	(5)He-1200 F	21,500	49	42	822.2
3CLX1	Na-He-1200 F	27,500	74	50	144.6
3CLX2	Na-He-1200 F	24,000	62	49	445.0
3CLX3	Na-He-1200 F	21,500	70	46	890.7
3DLX2	Na-He-1200 F	20,000	53	36	1437.0
3DLX3	Na-He-1200 F	18,500	33	17	2489.5
3DLX1	Na-He-1200 F	17,750	27	16	2942.2
3ELL3(7)	(5)Na-He-1200 F	24,000	73	53	632.0
3ELL2	(5)Na-He-1200 F	21,500	73	53	902.5
3ELL1(7)	(5)Na-He-1200 F	20,000	65	44	1869.7
3ELL4	(5)Na-He-1200 F	27,500	71	45	236.0
2-1/4 Cr-1 Mo					
2CAX4	Air-1100 F	20,000	64	58	90.8
2CAX2	Air-1100 F	17,500	42	36	139.2
2CAX3	Air-1100 F	15,000	41	32	302.3
2CAX1	Air-1100 F	12,000	23	15	1390.5
2EAL1	(6)Air-1100 F	10,000	22	11	1209
2CHX1	He-1100 F	20,000	55	55	108.0
2CHX3	He-1100 F	20,000	56	40	86.5
2DHX1	He-1100 F	16,500	47	41	246.6
2DHX3	He-1100 F	16,500	47	54	98.4
2DAX1	He-1100 F	14,500	36	41	303.8
2DAX4	He-1100 F	12,500	24	27	1545.0
2DAX5	He-1100 F	10,500	22	27	624.0(8)
2CHX4	He-1100 F	10,500	27	28	1398
2EHL1	(6)He-1100 F	14,000	36	31	78.8
2EHL2	(6)He-1100 F	12,500	31	21	180.4
*2EHL3	(6)He-1100 F	10,000	27	27	1078.4

Table 1 - Test Data Summary - Cont'd

2-1/4 Cr-1 Mo	Condition	Stress (Psi)	Elong %	Reduction of Area %	Rupture Time (Hrs)
2CLX1	Na-He-1100 F	20,000	88	66	30.1
2CLX4	Na-He-1100 F	20,000	68	60	71.7
2CLX5	Na-He-1100 F	20,000	77	60	41.9
2CLX2	Na-He-1100 F	16,500	71	58	146.6
2CLX3	Na-He-1100 F	14,000	53	58	298.8
2DLX1	Na-He-1100 F	12,000	34	36	1116.0
2DLX2	Na-He-1100 F	10,000	36	28	2106.6
2DLX3	Na-He-1100 F	7,500			6623.8(Term)
2ELL2	(6)Na-He-1100 F	16,500	67	53	20.0
2ELL1	(6)Na-He-1100 F	14,000	53	42	71.7
2ELL3	(6)Na-He-1100 F	12,000	56	41	159.7
2ELL4	(6)Na-He-1100 F	10,000	52	34	952.2

* - Results obtained since last report
** - Creep rate too high to determine a minimum rate
IP - Indicates tests in progress for indicated time
(5) - Specimen pre-exposed to 1200 F sodium for 4000 hours
(6) - Specimen pre-exposed to 1100 F sodium for 4000 hours
(7) - Specimen code corrected
(8) - Specimen failed prematurely at gage mark
(9) - Molybdenum sheet used accidentally as shielding.
(10) - Operational strain measurement difficulties, stress-strain data not available.
(11) - Specimen pre-exposed to 1200 F sodium for 4000 hrs - not washed.
(12) - Specimen pre-exposed to 1100 F sodium for 4000 hrs - not washed.

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			*IP (275)
250	High Oxide Na-1100 F	9,500			*IP (561)

(1) Loss of flow after 65 hours - Temperature gradient across specimen.
*IP Test in progress

TABLE 3 - FREE ENERGIES OF FORMATION AT 1100°F

Constituents	Initial State	Final State	ΔF K cal per gm-atom of Oxygen
Oxygen	1 atmosphere	1 atmosphere	0.0 (Base)
Oxygen	1 atmosphere	Air (21 v/o) 1 atmosphere pressure	-1.4
Oxygen	1 atmosphere	10 ppm O_2/He	-9.9
Oxygen	1 atmosphere	1 ppm O_2/He	-11.8
Oxygen	1 atmosphere	0.1 ppm O_2/He	-13.8
Phosphorus	Pure	PO(g) 1 atmosphere pressure	-23.0
Nickel	12.57 W/O in iron	NiO	-34.6
Carbon	0.092 W/O in iron	CO(g) 1 atmosphere pressure	-37.8
Nickel	Pure	NiO	-38.0
Carbon	Pure	CO(g) 1 atmosphere	-45.1
Molybdenum	1% in iron	MoO_2	-46.4
Iron	Pure	FeO	-49.9
Molybdenum	Pure	MoO_2	-50.8
Chromium	2.2 W/O in iron	Cr_2O_3	-65.9
Hydrogen	1 atmosphere	H_2O at 0.00265 psia (H_2 gas saturated with H_2O at 2000 psig	-67.63
Manganese	0.6 W/O in iron	MnO	-68.1
Chromium	Pure	Cr_2O_3	-70.2
Manganese	Pure	MnO	-76.9
Sodium	Pure	Na_2O 30 ppm in sodium	-77.8
Silicon	Pure	SiO_2	-85.9

TABLE 4 - STRESS TO RUPTURE OF STAINLESS STEEL IN SODIUM

Sample No.	Fig. No.	Pre-Exposed 4000 Hours	Stress Level (psi)	Percent Elongation	Rupture Time (hrs)	Overall Carbon Content* (ppm)	Estimated Case Thickness (microns)
3CLX1	17	No	27,500	74	144.6	483	3
3CLX3	18	No	21,500	70	890.7	539	30
3DLX2	19	No	20,000	53	1437.0	598	80
3DLX3	20	No	18,500	53	2489.5	875	100
3DLX1	21	No	17,750	27	2942.2	1180	100
99	22	Yes	Unstressed			581	6
3ELL4	23	Yes	27,500	71	236.0	903	40
3ELL3	24	Yes	24,000	73	632.0	922	40
3ELL2	25	Yes	21,500	73	902.5	712	30
3ELL1	26	Yes	20,000	65	1869.7	674	20

* as-received carbon = 457 ppm.

Note: Sections of specimens analyzed for carbon were removed from 1/2 inch above gage length, where the stress level was 40% of listed values.

Table 5 - 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	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

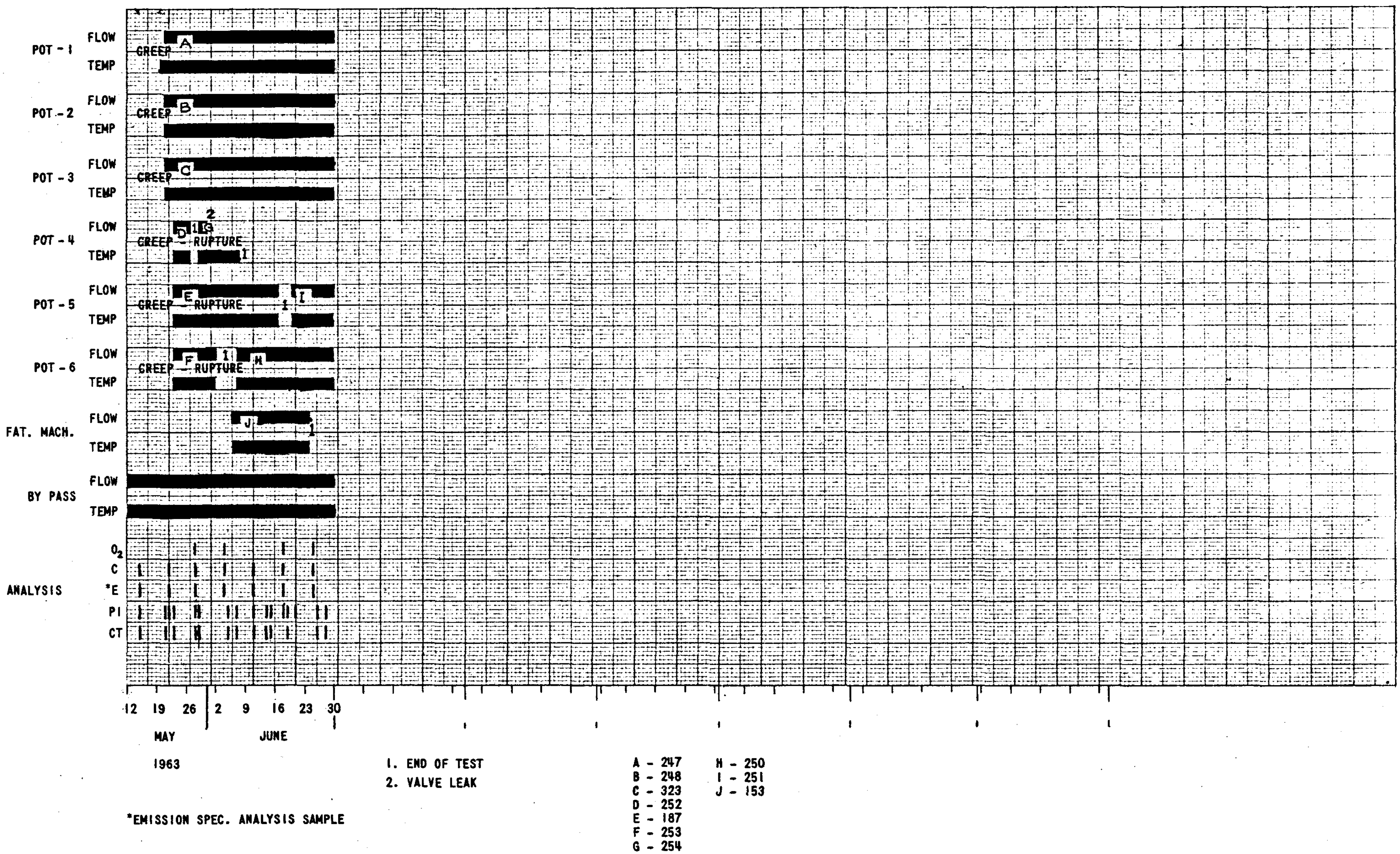


FIG. 1 - OPERATIONAL HISTORY OF LOOP 2 - TEST 4 (Cr-Mo Test Specimens)

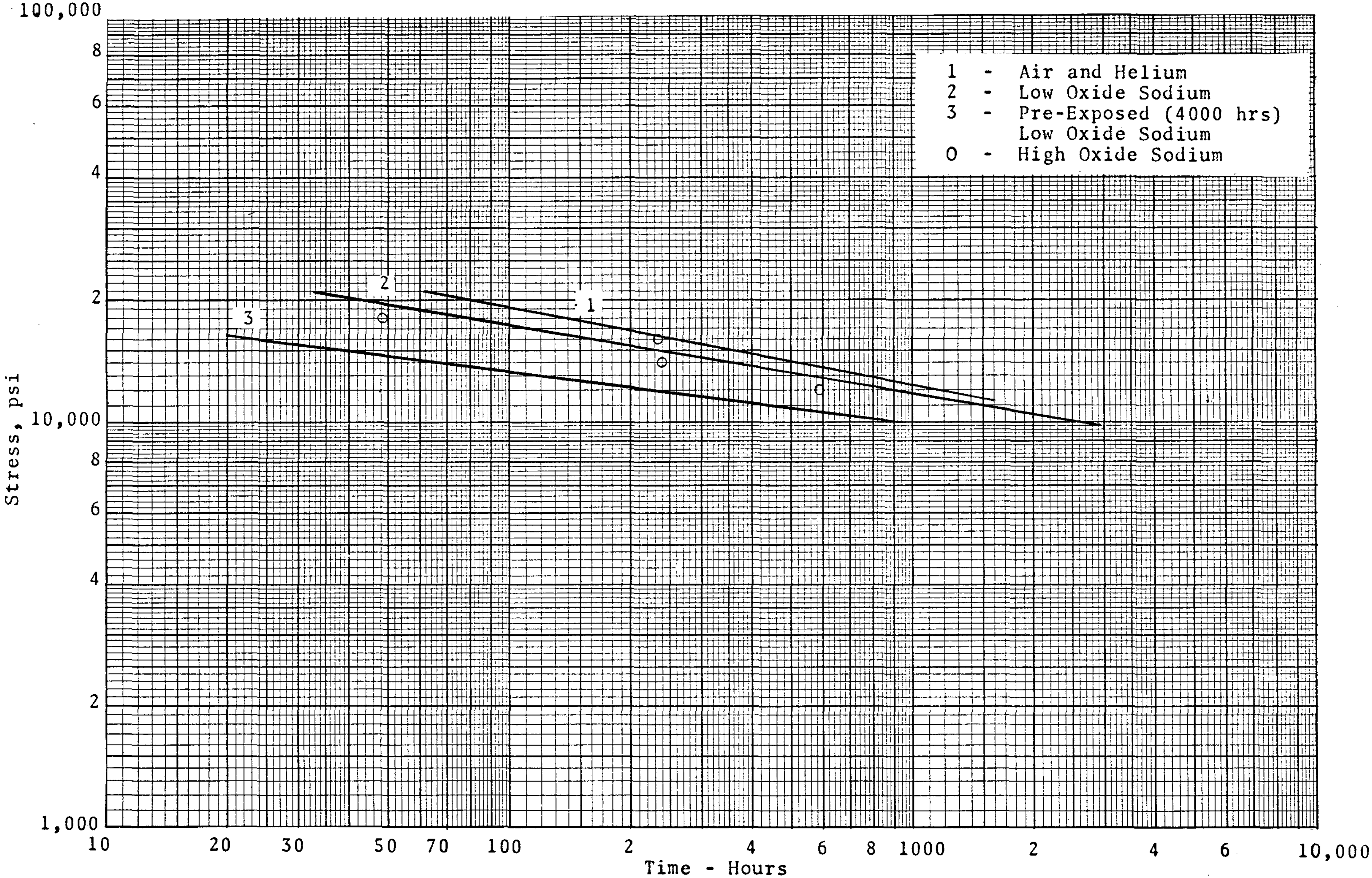


FIG. 2 - CREEP TO RUPTURE OF 2 1/4 Cr-1 Mo CARBON STEEL SPECIMENS - 1100 F

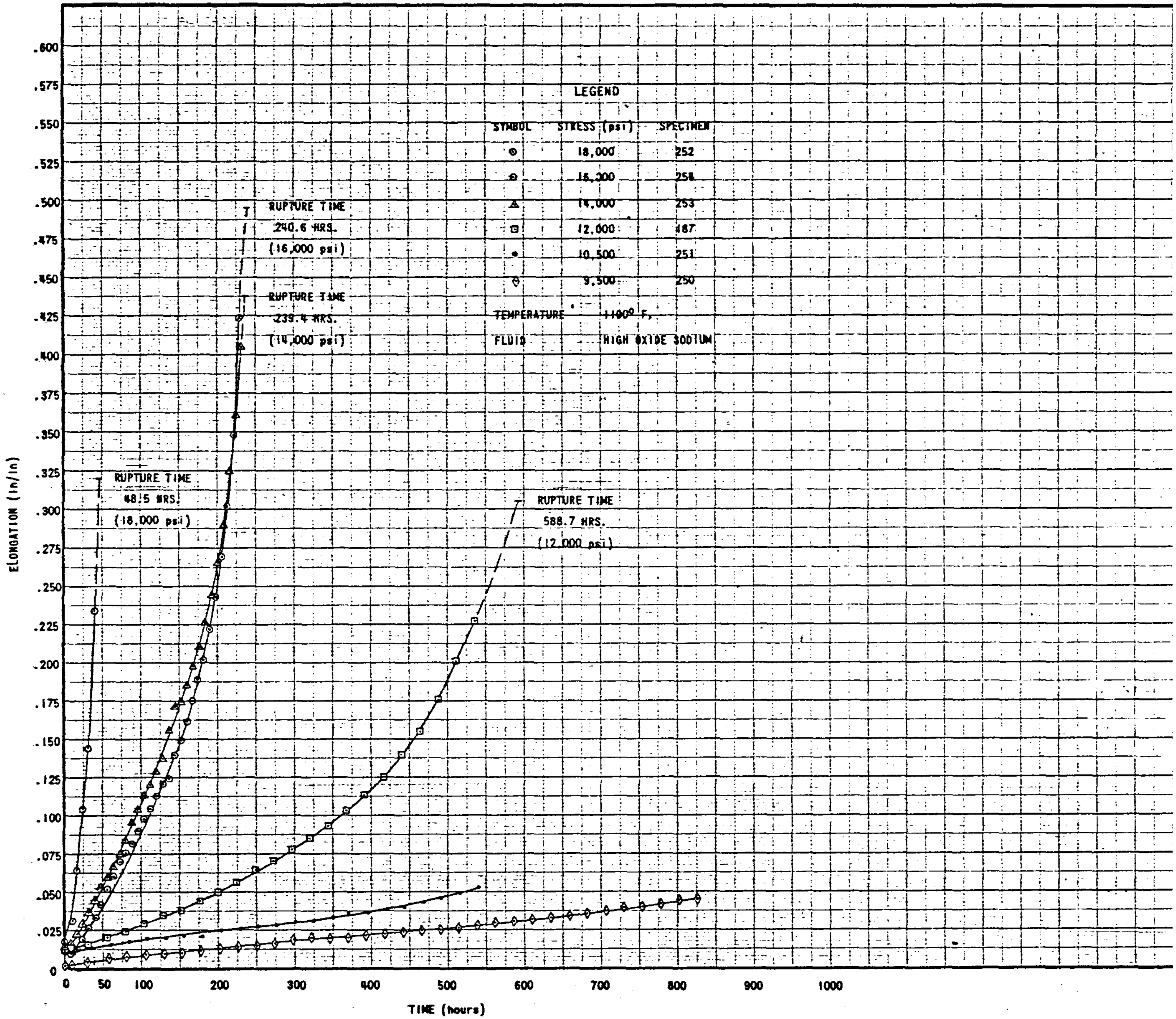


FIG. 3 - ELONGATION OF 2 1/4 Cr-1 Mo STEEL - CREEP RUPTURE SPECIMENS

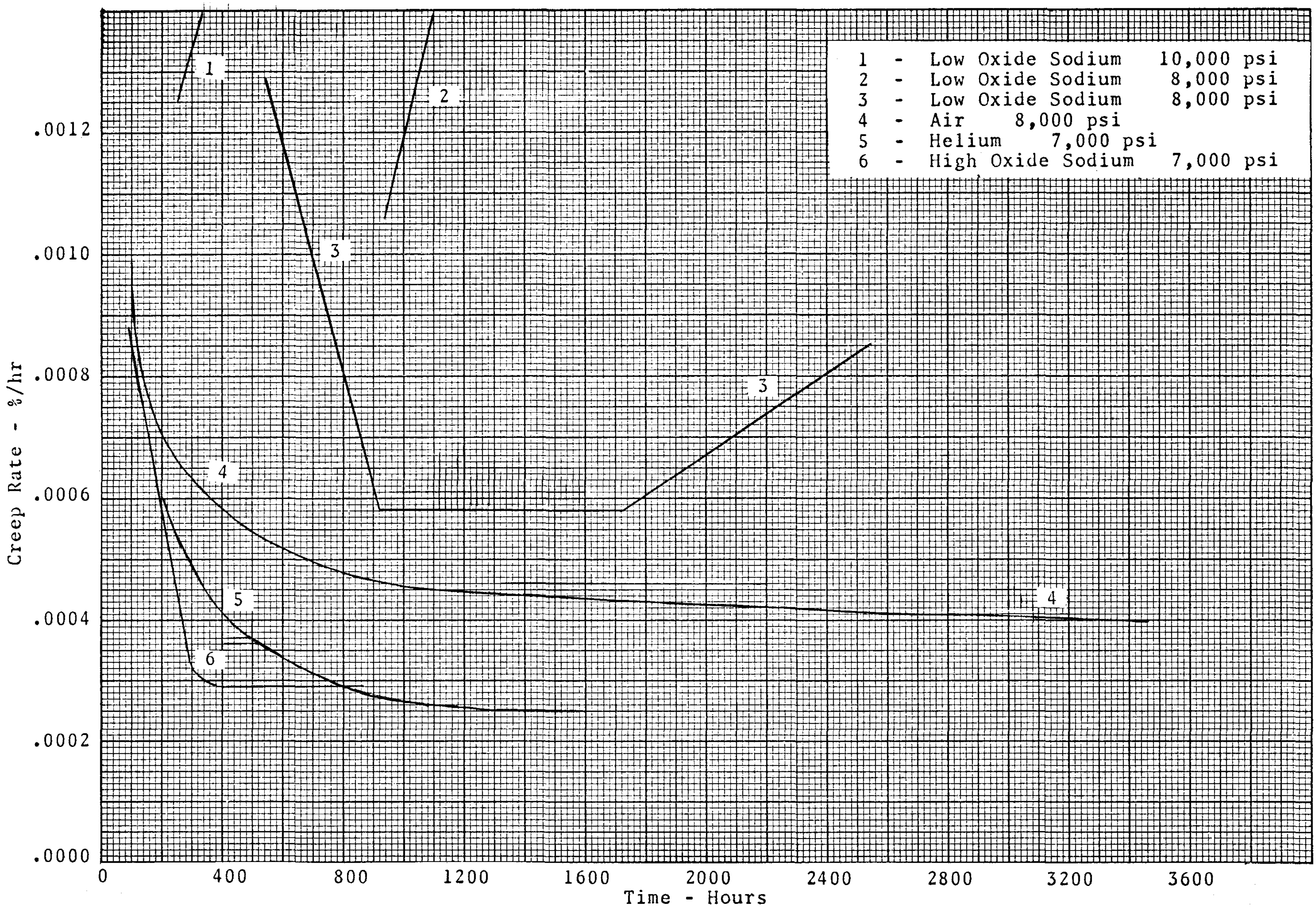


FIG. 4 - CREEP RATES OF 2 1/4 Cr-1 Mo CARBON STEEL - 1100 F

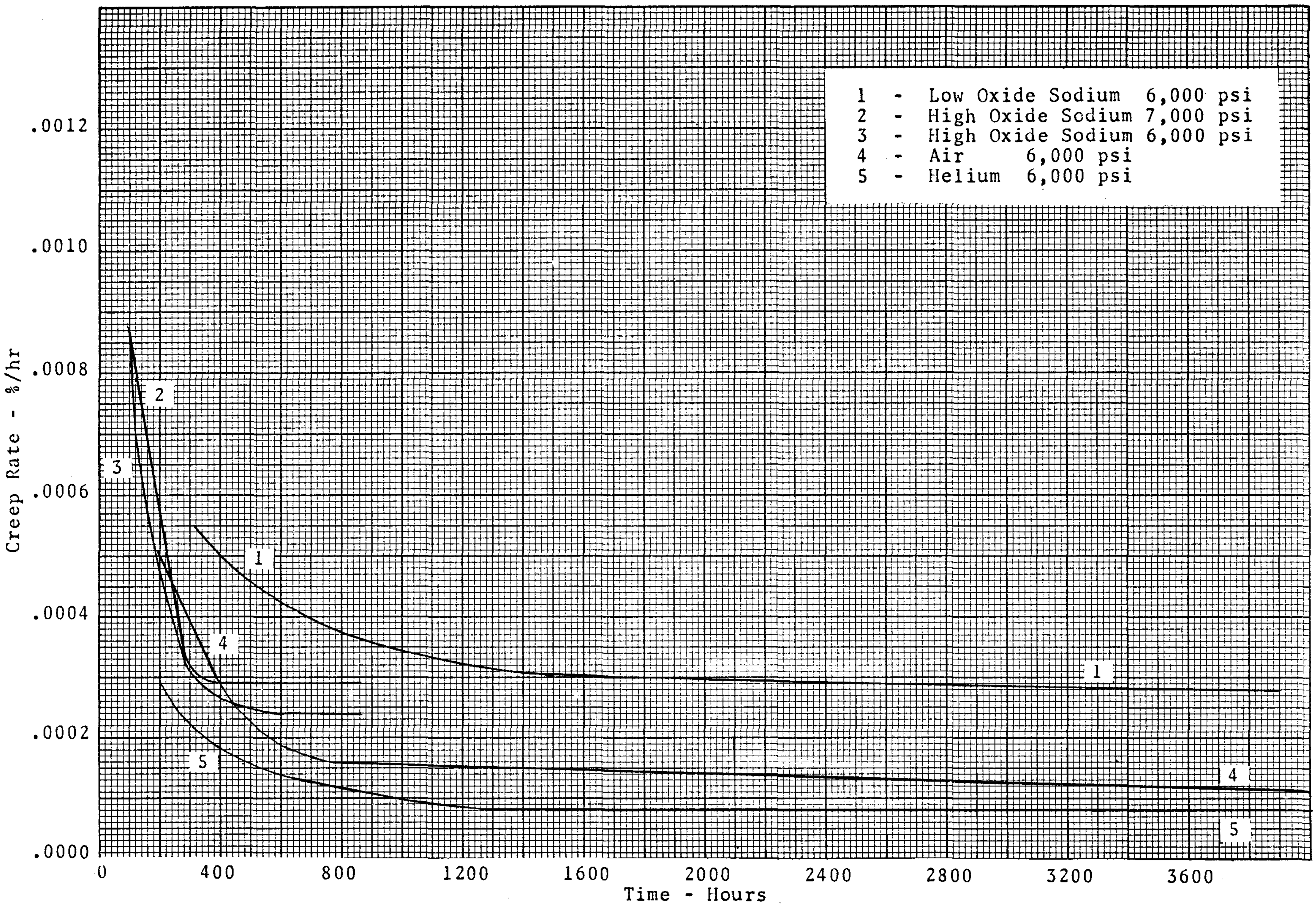


FIG. 5 - CREEP RATES OF 2 1/4 Cr-1 Mo CARBON STEEL - 1100 F

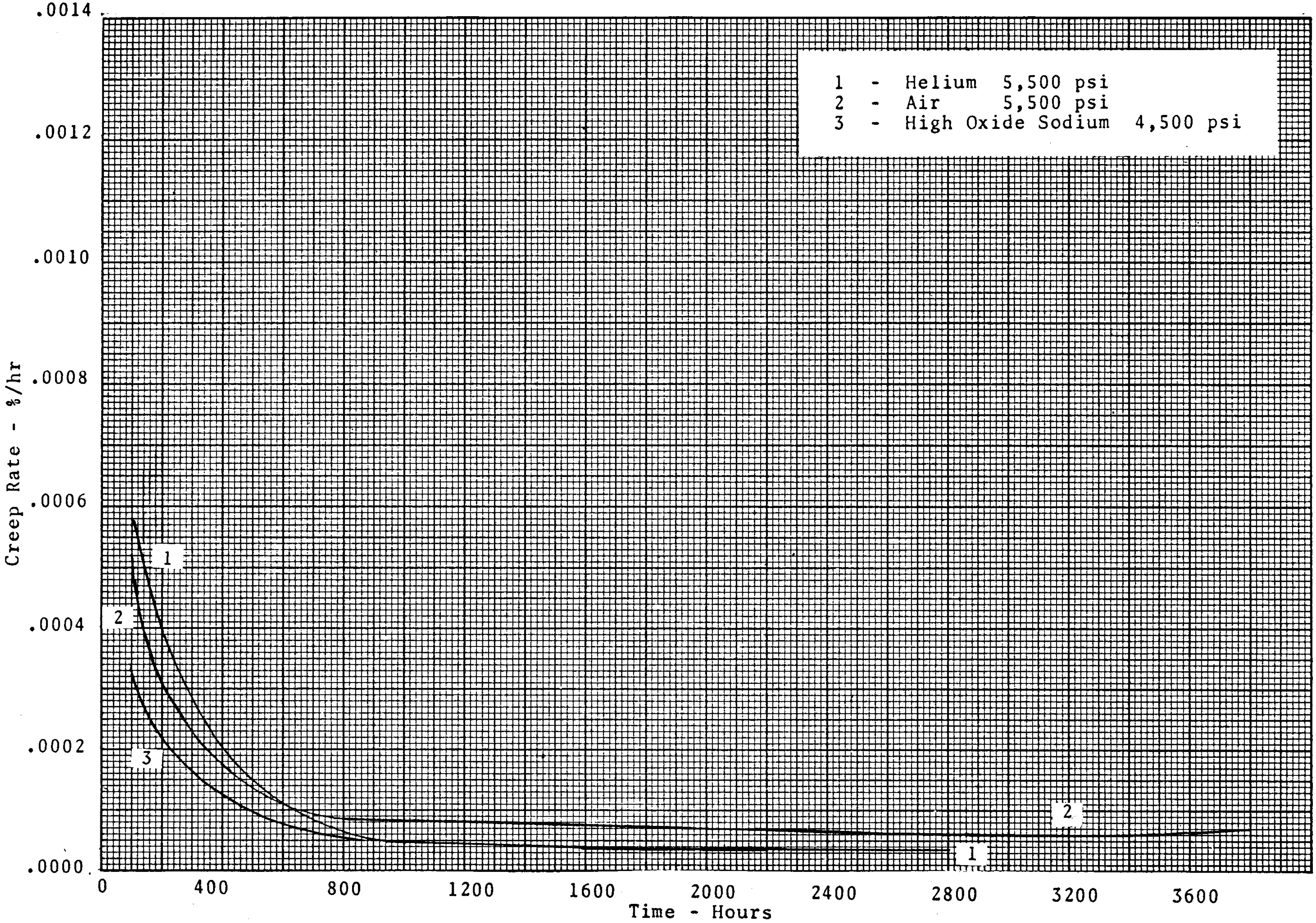


FIG. 6 - CREEP RATES OF 2 1/4 Cr-1 Mo CARBON STEEL - 1100 F

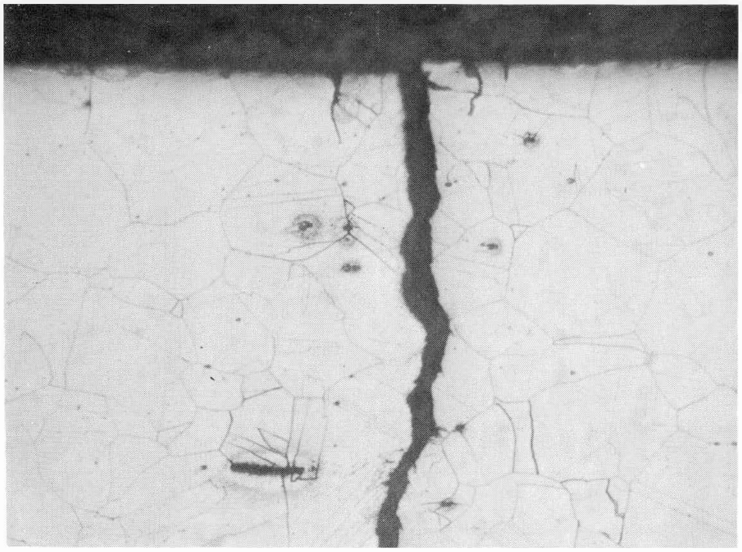


FIG. 7

316 ss FATIGUED IN 1200°F AIR
(3FAX2)
Cyclic Strain = 2.18%
Cycles to Failure = 598

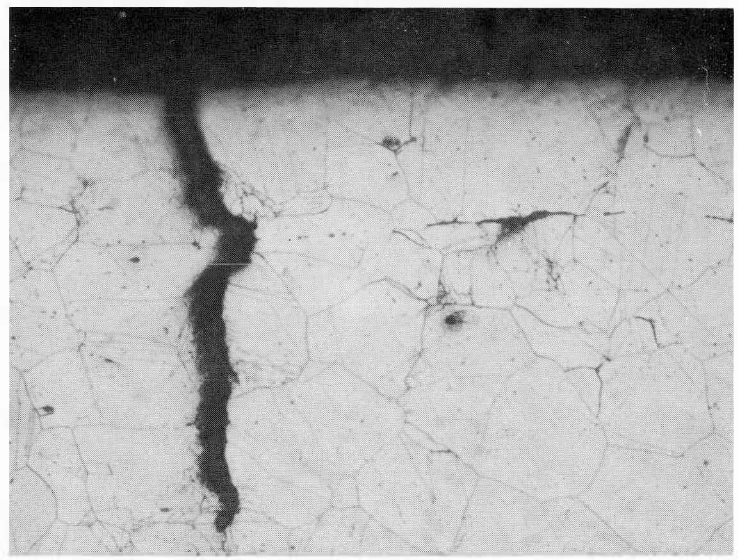


FIG. 8

316 ss FATIGUED IN 1200°F He
(3FHX4)
Cyclic Strain = 2.13%
Cycles to Failure = 1325

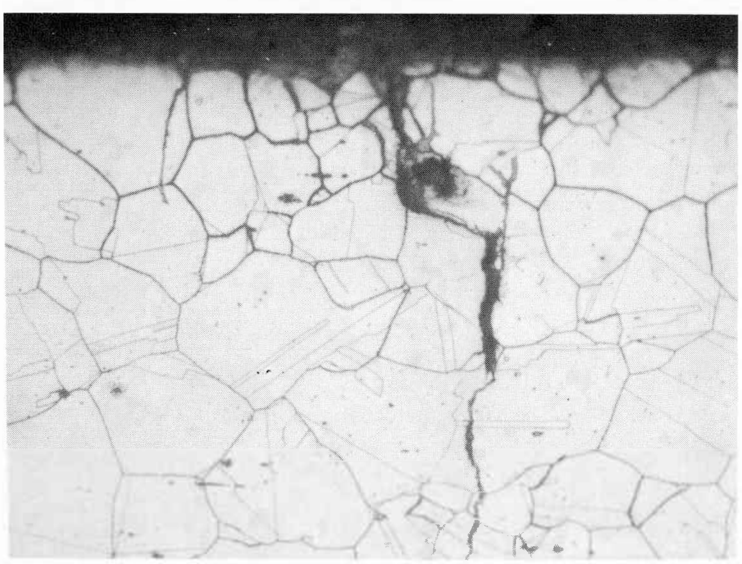


FIG. 9

316 ss PRE-EXPOSED TO 1200°F Na
(286 hrs) and FATIGUED IN
1200°F AIR
(3HAL2)
Cyclic Strain = 2.12%
Cycles to Failure = 302

Note: Specimens etched with ferric chloride - 266X of longitudinal sections.

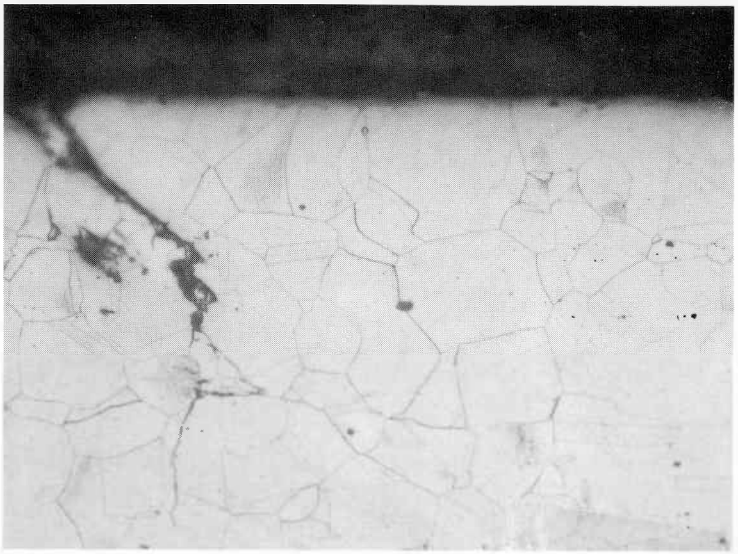


FIG. 10

316 ss FATIGUED IN 1200°F AIR
(3JAX7)
Cyclic Strain = 0.565%
Cycles to Failure = 8556

FIG. 11

316 ss FATIGUED IN 1200°F He
(3JHX2)
Cyclic Strain = 0.571%
Cycles to Failure = 38,804

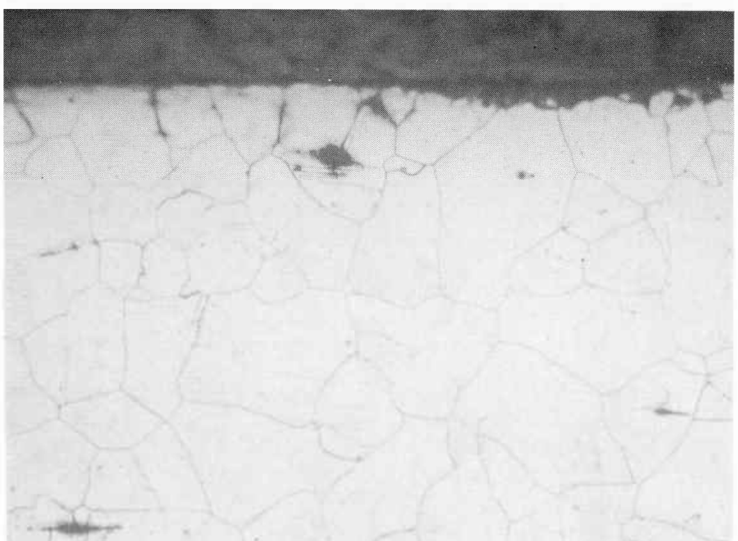


FIG. 12

316 ss FATIGUED IN 1200°F
SODIUM
(3JLX3)
Cyclic Strain = 0.558%
Cycles to Failure = 33,055

Note: Specimens etched with ferric chloride - 200 X of longitudinal sections.

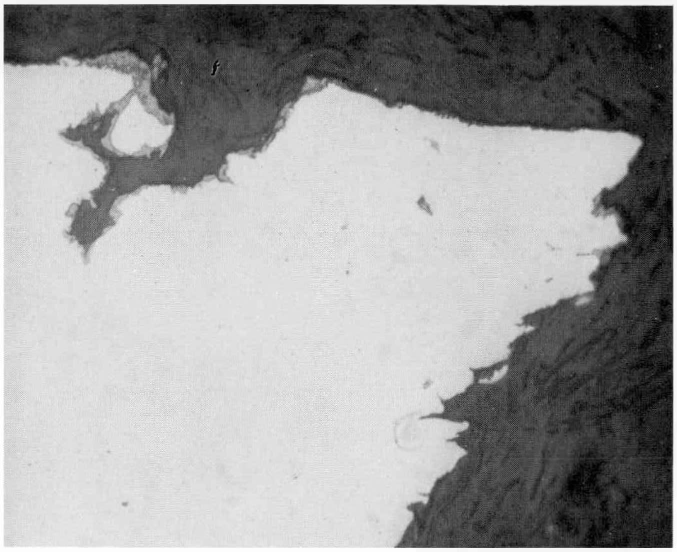


FIG. 13

316 STRESS-TO-RUPTURE IN 1200°F AIR
(3CAX10)
Stress = 27,500
Rupture Time = 173.6 hrs
Elongation = 61%
As polished - 66X

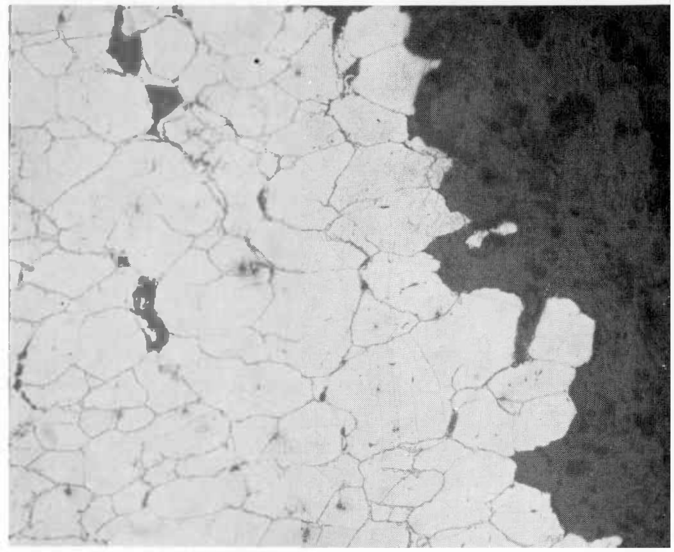


FIG. 14

316 STRESS-TO-RUPTURE IN 1200°F AIR
(3AAX4)
Stress = 17,750
Rupture Time = 3695 hrs
Elongation = 18%
Ferric chloride etch - 133X at fracture

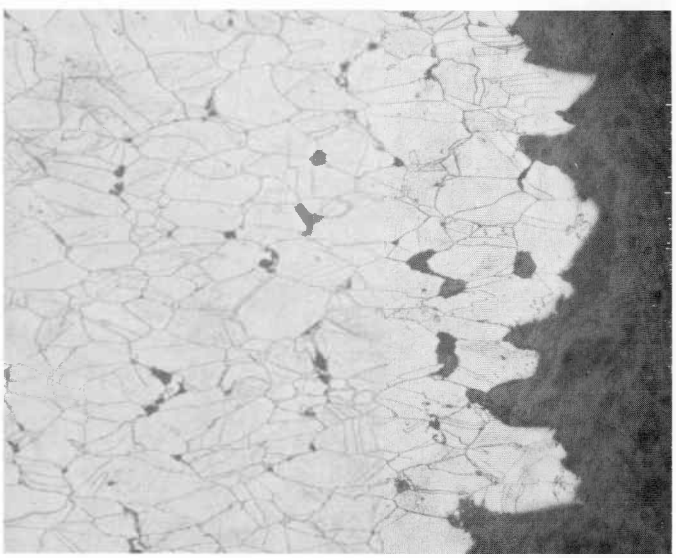


FIG. 15

316 STRESS-TO-RUPTURE IN 1200°F He
(3DHX2)
Stress = 18,000
Rupture Time = 2620 hrs
Elongation = 44%
Ferric chloride etch - 133X at fracture

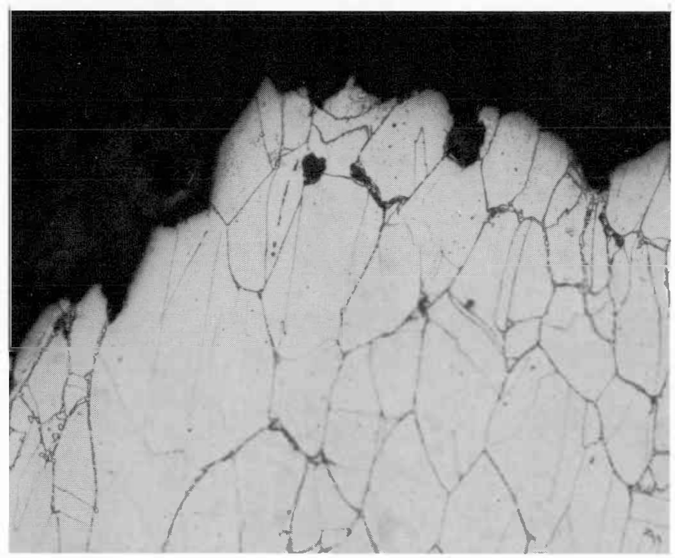
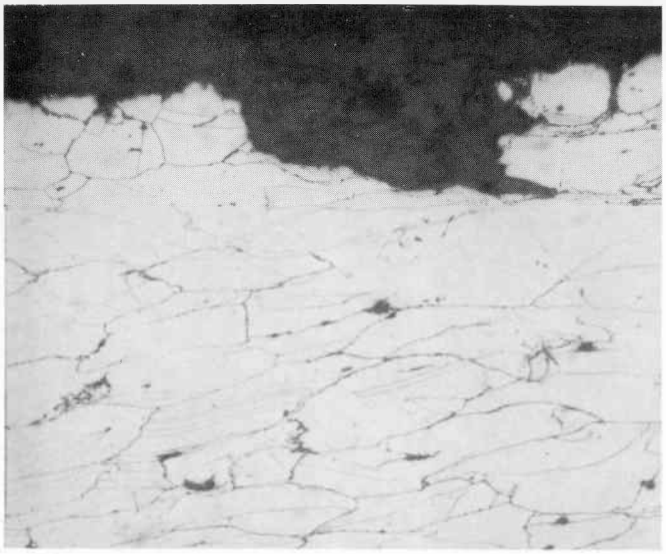
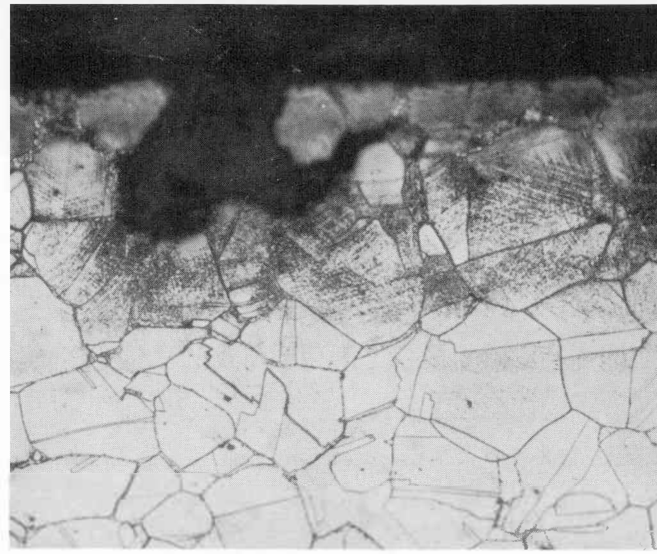
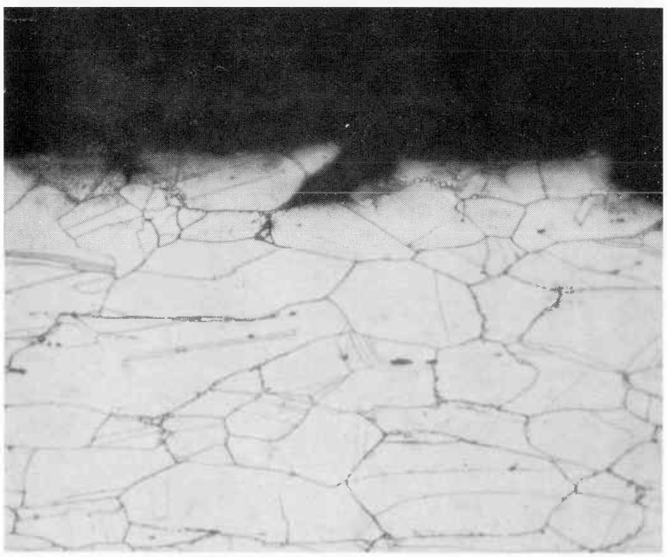


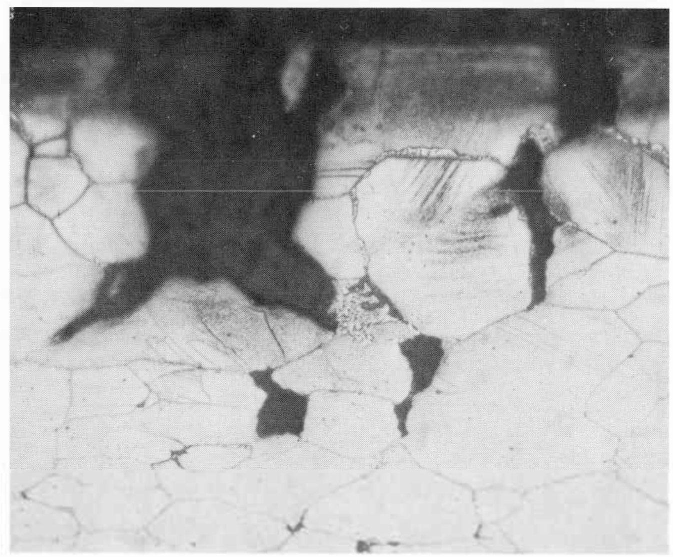
FIG. 16

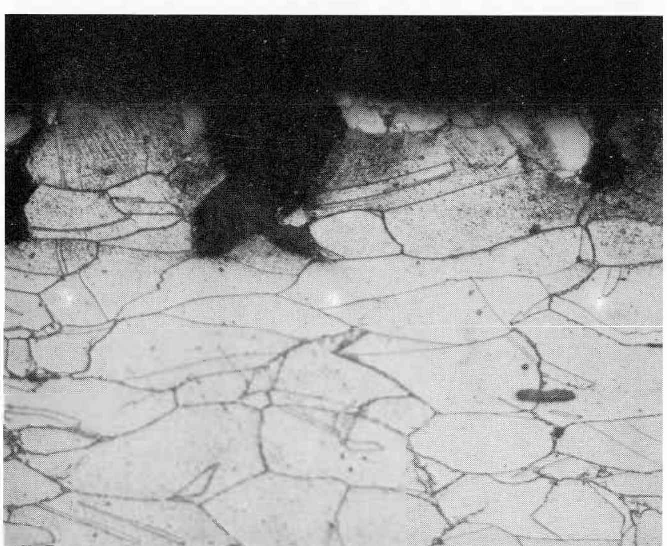
316 STRESS-TO-RUPTURE IN 1200°F Na
(3DLX3)
Stress = 18,500
Rupture Time = 2490 hrs
Elongation = 33%
Ferric chloride etch - 266X at fracture







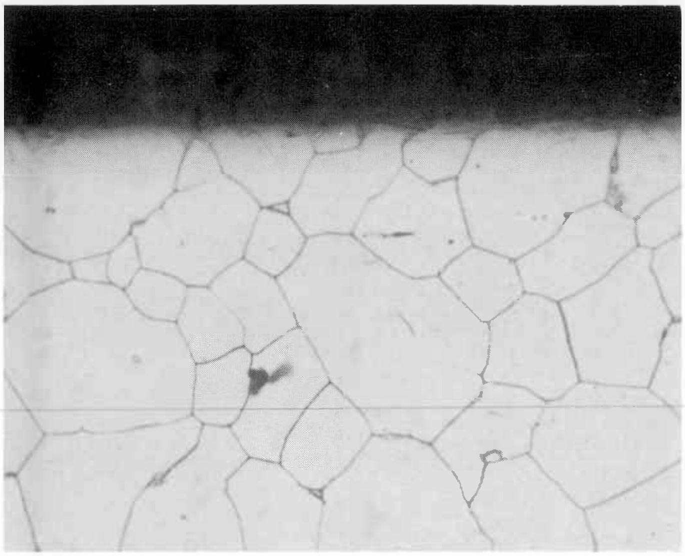


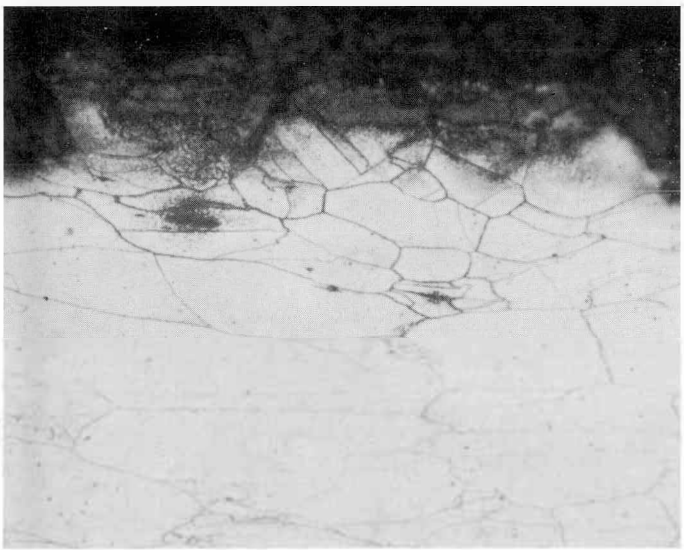


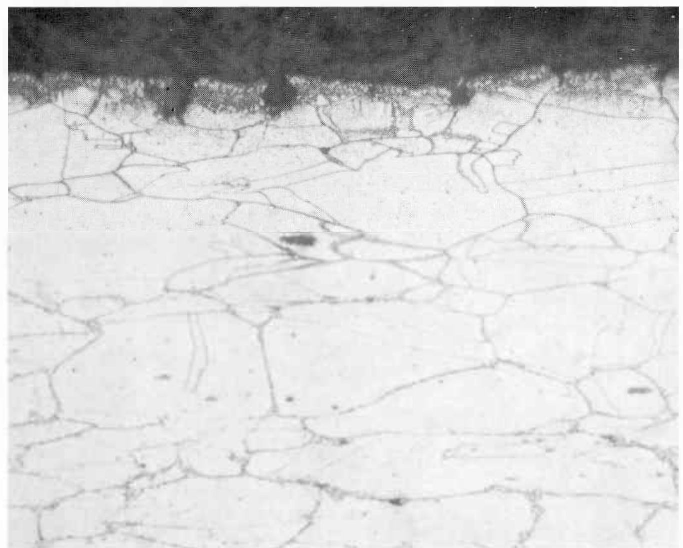
		Rupture Time, hrs
FIG. 17	- Top Left	144.6
FIG. 18	- Middle Left	890.7
FIG. 19	- Bottom Left	1437.0
FIG. 20	- Top Right	2489.5
FIG. 21	- Bottom Right	2942.2

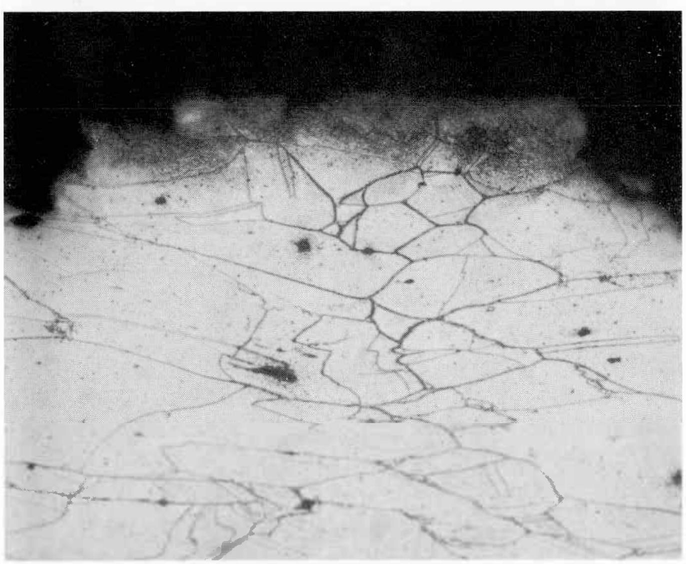
STAINLESS STEEL
STRESS-TO-RUPTURE IN Na

Ferric Chloride etch - 266X









		Rupture Time, hrs
FIG. 22	- Top Left	Unstressed (4000 hr)
FIG. 23	- Middle Left	236.0
FIG. 24	- Bottom Left	632.0
FIG. 25	- Top Right	902.5
FIG. 26	- Bottom Right	1869.7

PRE-EXPOSED STAINLESS STEEL
STRESS-TO-RUPTURE IN Na

Ferric chloride etch - 266X

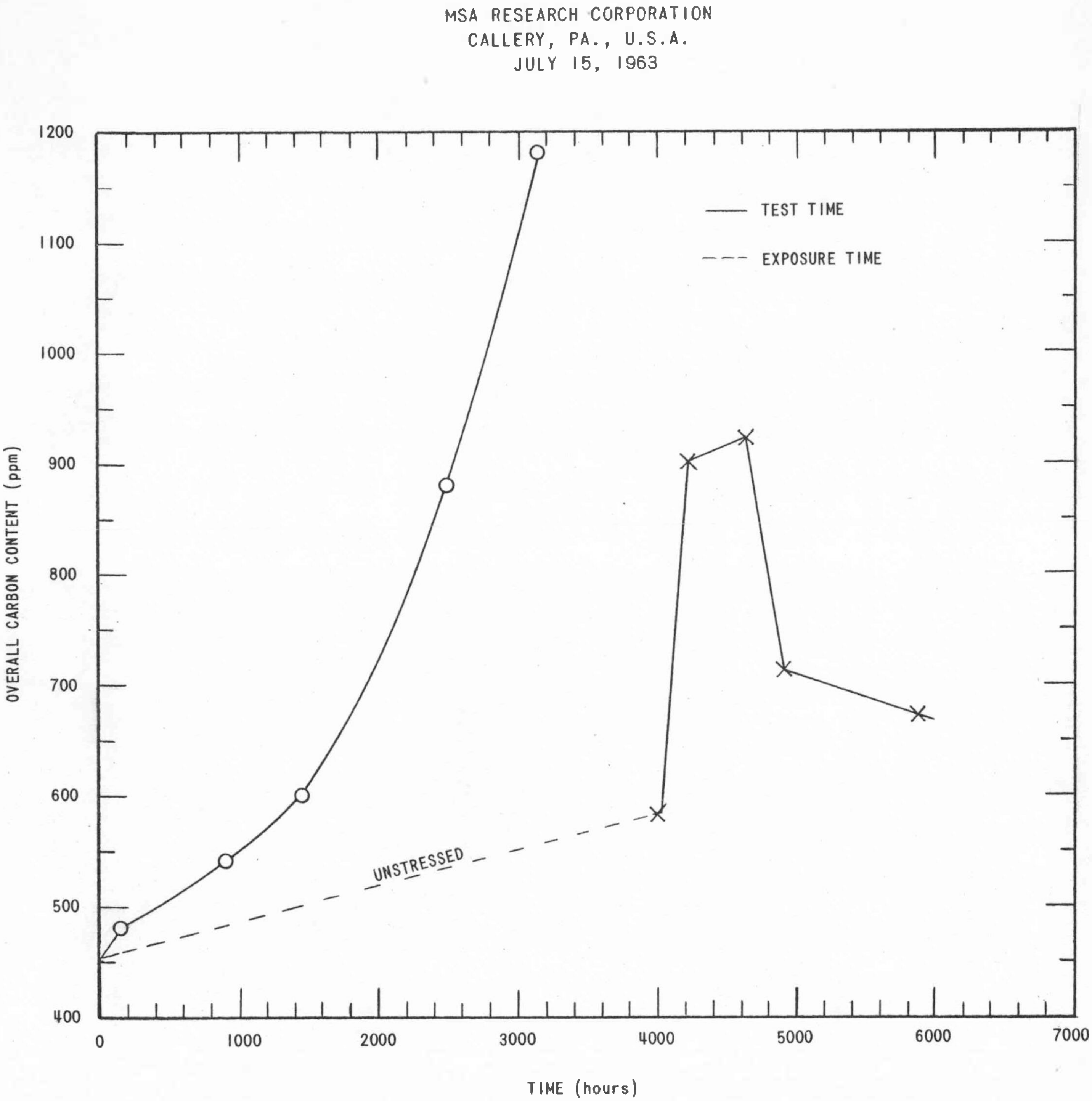


FIG. 27 - CARBURIZATION DURING STRESS RUPTURE OF 316 IN SODIUM - TEST 1