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MSAR 64-18

TOPICAL REPORT NO. 2
RESULTS OF PHYSICAL PROPERTY TESTS OF 316 SS SPECIMENS IN 1200 F SODIUM WITH LOW OXYGEN

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to

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

EFFECT OF HIGH TEMPERATURE SODIUM ON AUSTENITIC AND FERRITIC STEELS

Physical Properties of Materials

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

MASTER

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Available from the
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March 1964

MSA Research Corporation

Subsidiary of Mine Safety Appliances Company

Callery, Pennsylvania

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to

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ON AUSTENITIC AND FERRITIC STEELS

Physical Properties of Materials

March 1964

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ACKNOWLEDGEMENTS

The authors gratefully acknowledge the special skills and efforts of the various groups at MSAR whose combined contributions made this program possible. We particularly wish to recognize those in charge of operations (L. Kirschler), the analytical work (S. Rodgers) and the metallurgical studies (F. Tepper), as well as the technicians and operators who maintained the test conditions and schedules throughout the tests.

We also wish to acknowledge the special help and consultation of Professor J. W. Freeman of the University of Michigan and Professor G. T. Horne of Carnegie Institute of Technology.

ABSTRACT

Operation of nuclear power plants cooled with sodium necessitates additional information regarding the effects on the physical properties of the materials of construction. A program is in progress at MSAR under a U. S. Atomic Energy Commission contract in which the physical properties of 316 stainless steel and 2 1/4 Cr-1 Mo steel are compared in dynamic sodium systems at 1200 F and 1100 F respectively. This report presents the results of the first test series in which the fatigue, tensile, creep and creep-to-rupture properties of Type 316 ss were compared in 1200 F sodium, air and helium environments. Future scheduled tests will reveal the effects of sodium containing various contaminants, such as oxygen, carbon and nitrogen.

Using test specimens from the same heat, MSAR conducted creep, creep-to-rupture, and cycle strain tests in sodium. MSAR also conducted cyclic strain tests in air and helium. The University of Michigan, under subcontract to MSAR, conducted creep, creep-rupture and tension tests in air and helium. The results of these tests revealed:

1. The stainless steel specimens to be used throughout the program were representative of an average Type 316 ss heat.

2. The high temperature (1200 F) fatigue life of 316 ss in helium is longer than in air. The fatigue life of 316 ss in 1200 F sodium is the same as in air at high cyclic strains but is the same as in 1200 F helium at the low cyclic strains. Short time exposure to 1200 F sodium prior to high cyclic strain tests did not appreciably effect the life in sodium and helium but shortened the life in air.

3. No significant differences in the creep-rupture properties of 316 ss were discernable in air, sodium or helium at 1200 F. Specimens exposed for 4000 hours (unstressed) in 1200 F sodium also showed no significant change in creep-rupture properties.

4. The creep rates of 316 ss appear to be consistently higher in 1200 F helium and sodium compared to 1200 F air; however, the results show the stresses to produce a minimum creep rate of 1% in 10,000 hours in all three environments to be within a 15% spread.

5. Tensile properties showed no significant differences when tested in air or helium regardless of whether the specimens were exposed for 4000 hours to sodium or whether the exposed specimens were washed or unwashed prior to testing.

This is the second of a series of topical reports on this project. The first report, MSAR Topical Report 1 - Test Facility Design and Operation Procedures (MSAR 63-161), presented the details of the design and operation of the test equipment and facilities involved in the performance of these tests. The results of the effect of 1100 F sodium on the physical properties of 2 1/4 Cr-1 Mo steel will be reported in a future report. Following the issuance of that report there will be others giving the results of tests using sodium under the same conditions but with various contaminants.

TOPICAL REPORT 2

RESULTS OF PHYSICAL PROPERTY TESTS OF 316 SS SPECIMENS IN 1200 F SODIUM WITH LOW OXYGEN

1. INTRODUCTION

Since the development of sodium cooled reactor systems, it has been apparent that greater thermal efficiencies would be reached at higher operating temperatures helping to offset the higher equipment and operating costs of these systems.

The ASME Code provides allowable stress values for the austenitic and ferritic steels at temperatures above 1000 F for service in air environments but does not cover other environments. A considerable amount of design data is available up to 1000 F, but very little information is readily obtainable above this temperature as to the effects of various environments on the physical properties of the anticipated structural materials.

The Sodium Components Group of the Atomic Energy Commission undertook several programs to establish a design criteria for structural materials in sodium cooled reactor systems. The Physical Properties Research Program was developed to determine the effects of reactor grade sodium, and normally anticipated contaminants, on representative types of structural steels of interest for the construction of sodium systems. The materials to be tested and the test conditions for this program were selected by the Sodium Components Group so that the results could be interpreted and compared with other programs in related fields.

The materials selected for this program were Type 316 stainless steel as representative of the austenitic steels and 2 1/4 Cr-1 Mo steel as representative of the ferritic steels. The temperatures chosen were 1200 F for the Type 316 tests and 1100 F for the 2 1/4 Cr-1 Mo tests. To serve as a basis for comparison, all tests were to be duplicated in air and helium at the respective test temperatures, as well as in reactor grade sodium with various contaminants. The contaminants to be intentionally introduced into the sodium during various phases of the program were oxygen, carbon and nitrogen.

The type of tests to be employed to determine the environmental effects were to include tensile, creep, creep-rupture and high temperature cyclic strain tests.

The creep, creep-rupture and tension tests were carried out to the best of our abilities in accordance with ASTM E-139 (Recommended Practice for Conducting Creep and Time for Rupture Tension Tests of Materials) and ASTM E-21 (Short-Time Elevated Temperature Tension Tests of Materials). One noted exception was the one inch gage length of the creep-rupture specimens. The standard rectangular test specimen has a two-inch gage length.

The ASTM Manual on Fatigue Testing was used as a guide when applicable during the cyclic strain testing program.

In addition to these physical tests, appropriate chemical analyses and metallography techniques were to be used in assisting in the interpretation of the data obtained from these tests.

To fulfill the Physical Properties Research Program, Contract AT(11-1)-765 was awarded to MSA Research Corporation (MSAR). Under this contract, MSAR was to design, fabricate, operate test equipment and interpret results from the test program. All the test phases in sodium were to be conducted at MSAR.

In designing the test facilities, it was determined that standard air equipment for tensile, creep, and creep-rupture testing could be readily adapted for testing in an environment of helium. Since several such test facilities were already in existence, it was economical to use these existing facilities rather than to acquire these facilities at MSAR. Therefore, MSAR awarded subcontract AEC-765-1 under Prime Contract AT(11-1)-765 to the University of Michigan for this portion of the program. Under this contract, the University of Michigan was to conduct, under the direction of MSAR, the tensile, creep and creep-rupture tests in air, modify their equipment for tests in helium, and carry out these same tests in helium. All the work at the University of Michigan was under the direction of Professor J. W. Freeman who is also a consultant to MSAR on all phases of the Physical Properties Program. Since a special fatigue apparatus had to be designed and fabricated, MSAR conducted the fatigue tests in air and helium as well as in sodium.

This report covers the results of TEST 1 designated as that phase of the Physical Properties Program which involves the testing of Type 316 stainless steel in air, helium and reactor grade "as is" (no added contaminants) sodium. The oxygen content of the sodium was maintained at 30 ppm (saturation temperature of 300 F) or less throughout the testing. Other phases of the Physical Properties Program will be covered in future topical reports.

Detailed discussions of the design and operation of the test equipment and facilities are minimized in this report since <u>MSAR Topical Report 1 - Test Facility Design and Operation Procedures (MSAR 63-161)</u> discusses these areas in detail.

2. TEST PROGRAM

There are eight different tests involving 316 ss and 2 1/4 Cr-1 Mo steel specimens scheduled under this contract. These tests and their parameters are listed in Table 1. This report covers the procedures that were followed and the results that were obtained under TEST 1, the scope of which included tensile, creep, creep-rupture and fatigue testing of Type 316 stainless steel at 1200 F in air, helium and low (30 ppm) oxide reactor grade sodium. Future tests will be conducted in reactor grade sodium with intentionally introduced contaminants, namely carbon and nitrogen.

Test specimens consisted of both the original material and unstressed specimens exposed for 4000 hours to the 1200 F sodium. The various mechanical tests with the breakdown of the number of specimens scheduled per test are shown in Table 2. These tests and specimens are further described in the following sections.

2.1 TENSILE TESTS

To serve as a basis for comparison of tensile strengths, triplicate original material specimens were tested in air at 1200 F. In helium, triplicate original material specimens were tested at both 1200 F and at room temperature. To determine the effects of exposing Type 316 stainless steel (unstressed) to 1200 F low (30 ppm) oxide sodium for a period of 4000 hours, six exposed specimens were tested in helium at 1200 F. Three of these specimens were washed in alcohol and water after exposure, while three were tested unwashed from the exposure pot. Six similarly exposed and washed specimens were tested in helium at room temperature.

2.2 CREEP TESTS

Three creep specimens were run at 1200 F in each of the environments: air, helium and low (30 ppm) oxide sodium. The three specimens were run at three different stress levels that permitted the determination of a minimum creep rate of 0.1%/1000 hours. The creep tests were terminated at the end of 4000 hours.

Twenty-one additional specimens were exposed, stress free, for 4000 hours during the sodium creep tests for later tensile and creep-rupture tests.

TABLE 1 - COMPLETE TEST PROGRAM

Test	Material	Temp.	Sodium	Contamination	Cover Gas	Sodium-Oxide
1	316 ss	1200 F	Reactor Grade	"As Is"	He	30 ppm
3	316 ss	1200 F	Reactor Grade	Saturated C	He	30 ppm
5	316 ss	1200 F	Reactor Grade	"As Is"	N_2	30 ppm
7	316 ss	1200 F	Reactor Grade	Saturated C	N_2	30 ppm
2	2-1/4 Cr-1 Mo	1100 F	Reactor Grade	"As Is"	He	30 ppm
4	2-1/4 Cr-1 Mo	1100 F	Reactor Grade	"As Is"	He	200-300 ppm
6	2-1/4 Cr-1 Mo	1100 F	Reactor Grade	"As Is"	N_2	30 ppm
8	2-1/4 Cr-1 Mo	1100 F	Reactor Grade	Saturated C	He	30 ppm

TABLE 2 - SPECIMEN AND TEST SCHEDULE FOR TEST 1
(316 ss at 1200 F)

Environment	Creep Test			Tensile Test				Creep to Rupture												High Temperature Fatigue			
	UE			UE	PE	UE RT	PE RT	PE			UE*			UE						Cyclic Strain 0.56%	Cyclic Strain 1%	Cyclic Strain 2.1%	Cyclic Strain 2.1% PE
	#1	#2	#3					#1	#2	#3	#1	#2	#3	#1	#2	#3	#4	#5	#6				
Na, with 30 ppm O_2	1	1	1					1	1	1				1	1	1	1	1	1	3	3	3	3
Air	1	1	1	3				1	1	1	2	2	2	1	1	1	1	1	1	3	3	3	3
Helium	1	1	1	3	6	3	6	1	1	1				1	1	1	1	1	1	3	3	3	3

UE - Unexposed
RT - Room Temperature
PE - Pre-exposed
\# - Stress Values
* - Special Check for Uniformity

2.3 CREEP-RUPTURE TESTS

Six creep-to-rupture specimens at six different stress levels were run at 1200 F in air, helium and low (30 ppm) oxide sodium. The six stress levels were chosen to provide a curve for each environment from short time rupture to 3000 hours rupture time.

Nine creep-rupture tests were also conducted at 1200 F in air, helium and sodium on specimens previously exposed for 4000 hours to 1200 F sodium with low oxide. The three highest stresses used during the tests on the original stock were selected as the operating stresses for these exposed tests.

Six additional specimens of the original material, two at each of the three highest stresses, were run in air at 1200 F. The purpose of the latter runs was to establish the degree of uniformity of the specimens.

2.4 FATIGUE TESTS (HIGH TEMPERATURE CYCLIC STRAIN TESTS)

The fatigue tests conducted throughout the program were based on bending test specimens over mandrels having a machined radius. Using this technique, the radius of the mandrel controlled the cyclic strain.

The cyclic tests performed in this phase of the program are referred to throughout this report as "fatigue tests." Fatigue testing is normally conducted at ambient temperature or slightly above where the creep rate of polycrystalline steels is negligible. Under the temperature conditions employed in this test program (1100 and 1200 F), the specimens are exposed to cyclic stress conditions where creep can be brought into play. Thus, "high temperature cyclic testing" more adequately defines the conditions employed than does "fatigue" testing.

In each of the test environments: air, helium and low (30 ppm) oxide sodium, triplicate specimens were tested at three different cyclic strain levels. The averaged data points were used to develop curves for cyclic strain vs cyclic failure for each environment. The cyclic strain levels were selected to cause failures in approximately 400, 5000 and 60,000 cycles.

During one of the 60,000 cycle tests in sodium, nine specimens were exposed stress free. These specimens were then tested in triplicate at the high cyclic strain in the three environments.

3. TEST FACILITIES

3.1 SODIUM TEST LOOP NO. 1

The environmental tests in low oxide sodium were performed at MSAR in one of two identical dynamic loops constructed of Type 316 stainless steel. (The second loop was used for the testing of 2 1/4 Cr-1 Mo steel specimens.) The major loop statistics, such as sodium volumes, flow rates and surface areas, are listed in Table 3. As shown schematically in Fig. 1, and isometrically in Fig. 2, the main flow was pumped through a resistance type electrical immersion heater which heated the system to approximately 1200 F. From this heater, the sodium flow was divided into seven parallel circuits each containing a complete test unit. Three of the test units were used for measuring creep rates, three for conducting creep-rupture tests, and one for the fatigue tests. After the sodium entered the bottom of a test unit, the temperature was adjusted to 1200 F ±3°F by an immersion type heater. Flow was then directed by baffles across the test specimen and out of the test unit into a common return manifold. Flow to each test unit was monitored by an individual magnetic flowmeter. Each test unit could be isolated, drained or charged independently. The test units will be described in detail under the individual test discussions. Flow from the return manifold entered the side of an instream expansion tank which was equipped for extraction of representative sodium samples for various analyses. From here the sodium was gravity fed back to the electromagnetic pump.

An eighth parallel circuit included the necessary components for monitoring and controlling the oxide concentration of the sodium. Flow entered this circuit through a throttling valve, magnetic flowmeter, an economizer and a finned air cooler. Upon leaving the cooler, the flow was split by means of control and shut-off valves to allow a choice of flow through either the plugging indicator, for determining the oxide concentration, or through the cold trap, for controlling the oxide concentration. This complete circuit is referred to as the Oxide Control and Indicating (OCI) circuit.

Test temperatures were automatically maintained through the use of West Guardsman Digital Set Point Controllers and saturable core reactor systems that actuated the immersion heaters. The system temperatures pertinent to the test were measured with calibrated thermocouples and monitored on a Brown multipoint recorder which augmented the manual data logging.

TABLE 3 - TEST LOOPS - STATISTICS

I. Circulating Section (Heater Expansion Tank & 1 in. Pipe)

Na Volume: 2.206 Ft^3
Na Weight: 110#
Contact Area: 32.7 Ft^2

II. Creep or Stress Machine (Pot only)

Na Volume: 0.1758 Ft^3
Na Weight: 8.85#
Velocity (1/2 gpm) Past Specimen: 0.045 Ft/sec.

III. Creep or Stress Machine (Including Inlet & Outlet Lines)

Na Volume: 0.1858 Ft^3
Na Weight: 9.35#
Contact Area: 3.82 Ft^2

IV. Fatigue Machine (Pot only)

Na Volume: 0.552 Ft^3
Na Weight: 27.8#
Velocity (1/2 gpm) Past Specimen: 0.0055 Ft/sec.

V. Fatigue Machine (Including Inlet & Outlet Lines)

Na Volume: 0.585 Ft^3
Na Weight: 29.4#
Contact Area: 6.77 Ft^2

VI. Cold Trap (Pot only)

Na Volume: 0.87 Ft^3
Na Weight: 43.6#

VII. OCI System (Including Inlet & Outlet Lines)

Na Volume: 0.973 Ft^3
Na Weight: 49#
Contact Area: 14.16 Ft^2

VIII. Total System

Na Volume: 4.8788 Ft^3
Na Weight: 197.75#
Contact Area: 76.55 Ft^2

IX. Fatigue Cycle	Up	Hold	Down	Hold
3-1/8" Radius Mandrel	5 sec.	5 sec.	5 sec.	5 sec.
6-3/4" Radius Mandrel	3 sec.	7 sec.	3 sec.	7 sec.
12" Radius Mandrel	5 sec.	5 sec.	5 sec.	5 sec.

X. Fatigue Specimen Area - 6.858 in.^{2} or 0.0475 Ft^2
Creep Specimen Area - 13.765 in.^{2} or 0.0955 Ft^2
Stress Specimen Area - 16.015 in.^{2} or 0.111 Ft^2

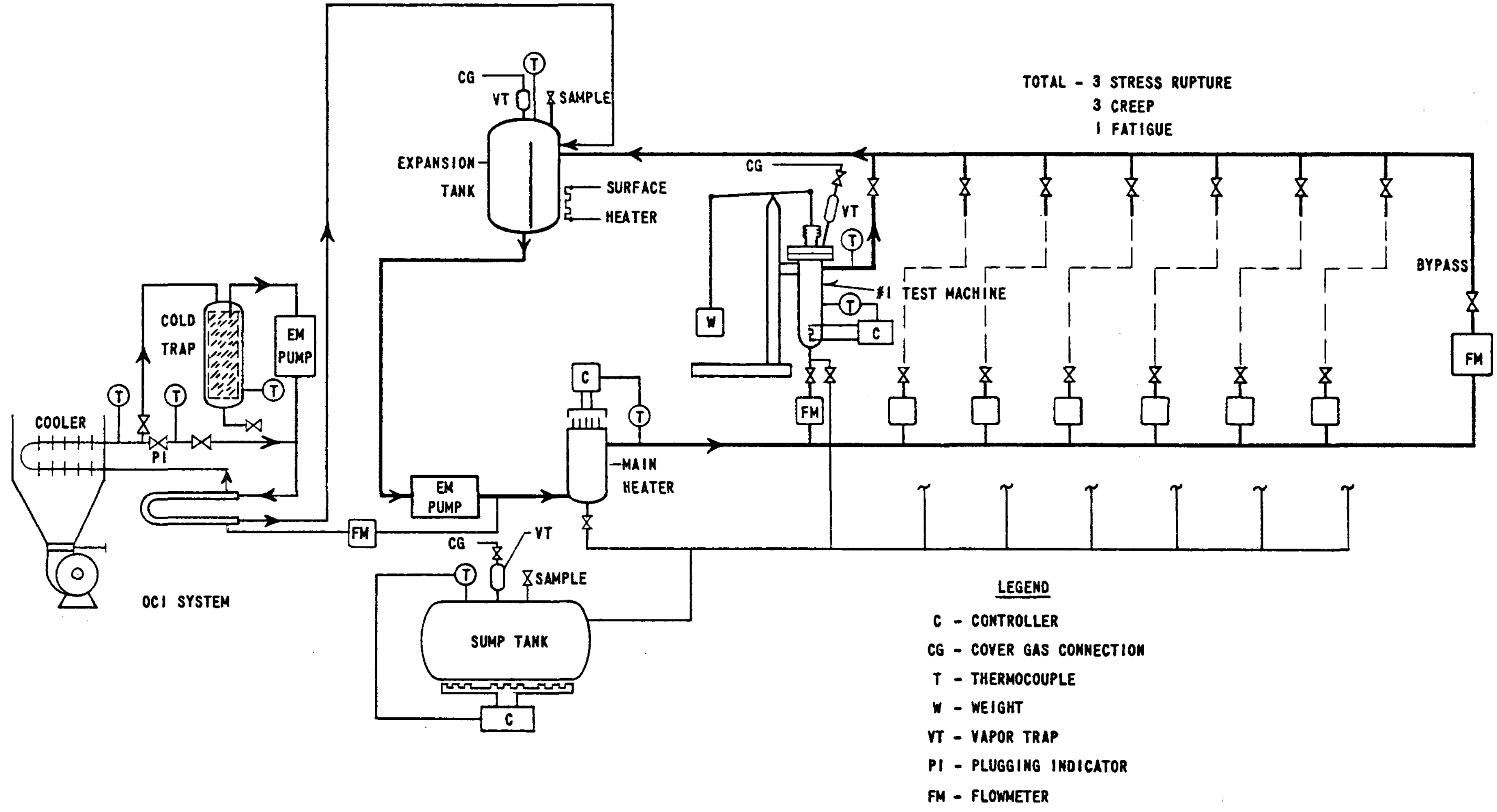


FIG. 1 - FLOW SHEET OF PHYSICAL PROPERTIES TEST LOOP

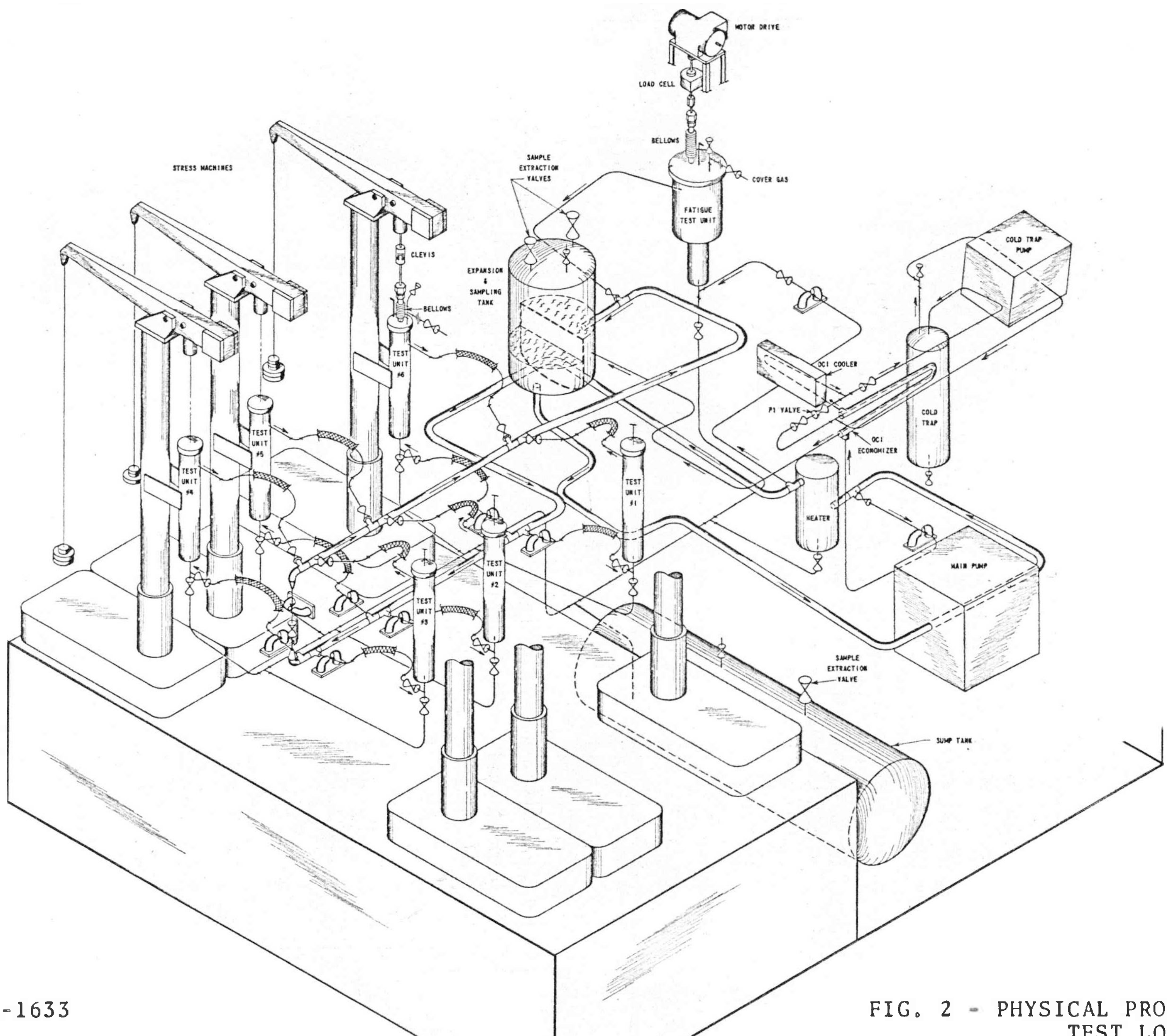


FIG. 2 - PHYSICAL PROPERTIES TEST LOOP (Isometric)

3.2 AIR AND HELIUM TEST FACILITIES

The air and helium phases of TEST 1, with the exception of fatigue tests (see Section 5), were carried out at the University of Michigan under subcontract to MSAR. Dr. James Freeman directed this program and acted as general consultant throughout the overall program. The air tests were carried out in existing standard test machines which were later converted to accommodate the helium tests. The standard test machine was modified by inserting a tubular chamber inside the heating furnace of the air test machine. The test specimens were installed in the chamber and the chamber was heated, during which time a vacuum was maintained to outgas the chamber and specimen. Standard tank helium was then introduced after being passed through a NaK purification system to remove any traces of oxygen and moisture.

4. TEST SPECIMENS

4.1 TEST SPECIMEN DESIGN AND FABRICATION

All specimens were obtained from sheet stock and had a nominal thickness of 0.065 in. The number of specimens required for the entire program; i.e., TESTS 1 through 8, was considered and sufficient stock was procured from the same heat to provide for this need.

The Type 316 stainless steel test specimens were fabricated from fully annealed 16 gage stock with an ASTM grain size number 5-7. All the specimens were taken from the sheet stock in the direction of rolling.

The specimens for the creep and tensile tests were identical with gage lengths of 2 1/2 in. x 1/2 in. The total elongation of the creep-rupture specimens forced the reduction of this specimen's gage length to 1 in. x 1/2 in. in order to contain the elongation within the range of the stress machine. The fatigue specimens were 6 in. x 1/2 in. The detailed dimensions of the test specimens are shown in Figs. 3 and 4.

Blanks 7 5/8 in. x 1 3/8 in. for creep, creep-rupture and tensile tests were cut from stock. Blanks 6 1/8 in. x 5/8 in. were cut for the fatigue tests. All edges and surfaces were milled equally from both sides. The blanks were milled to within 0.010 in. of the finished dimensions. The final 0.010 in. over the gage length were removed by wet grinding techniques in decrements less than 0.005 in. per pass to insure stress-free surfaces. The resultant surface finish was 32 RMS or better.

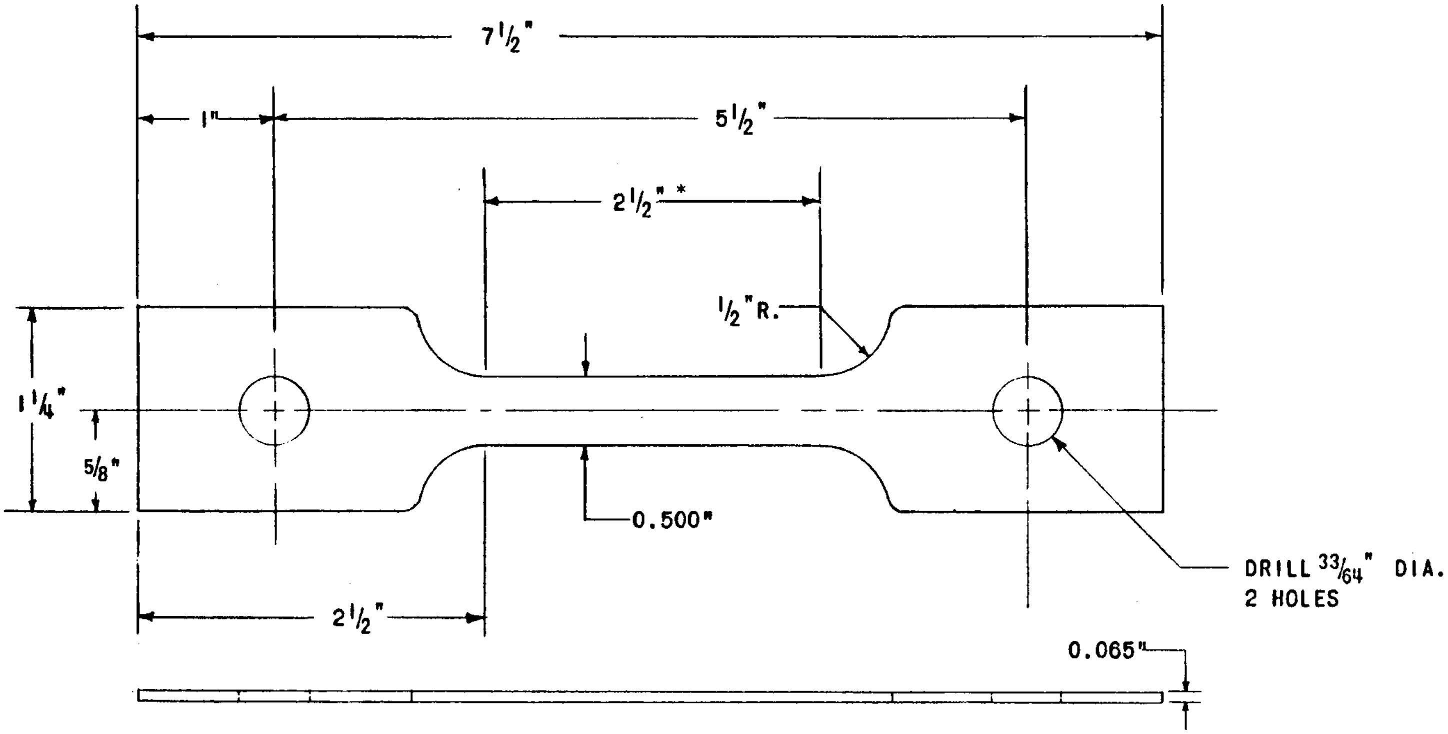


FIG. 3 - CREEP, TENSILE AND CREEP-RUPTURE SPECIMEN
(Creep-to-rupture gage lengths are 1 inch)

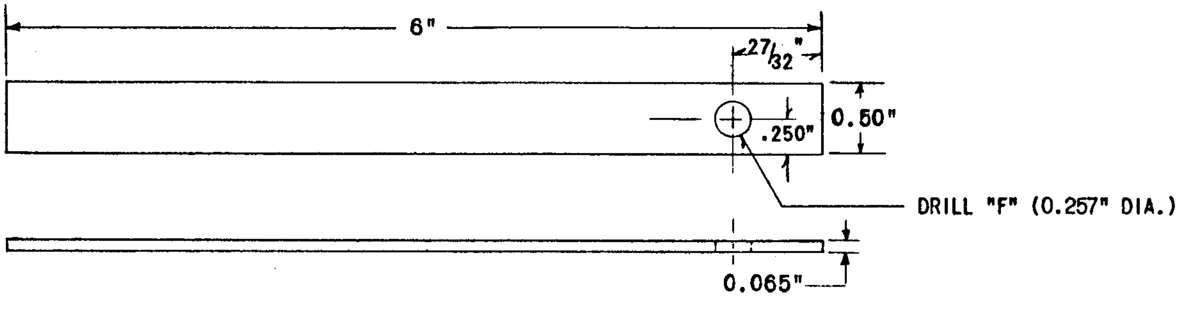


FIG. 4 - FATIGUE SPECIMEN

4.2 TEST SPECIMEN IDENTIFICATION

Each test specimen was numbered consecutively for identification. In addition to the consecutive numbers, a code system was originally used to identify a specimen with a particular test. This cross reference system proved to be both cumbersome and restricting and has been dropped in succeeding tests. Consecutive numbers only are used. A card file system is maintained in which the detailed dimensions and history of each specimen is logged.

4.3 TEST SPECIMEN HANDLING PROCEDURES

The overall specimen handling procedure is pictured graphically in Fig. 5. The major handling steps are described in detail in the following sections.

4.3.1 Pre-Test Cleaning

After fabrication, the specimens were washed thoroughly in acetone and rinsed in methyl alcohol, dried and placed in a desiccator until use. Topographic photographs were taken of each specimen prior to testing. After cleaning, all handling was performed with clean, white, lint-free gloves.

4.3.2 Pre-Test Measurements

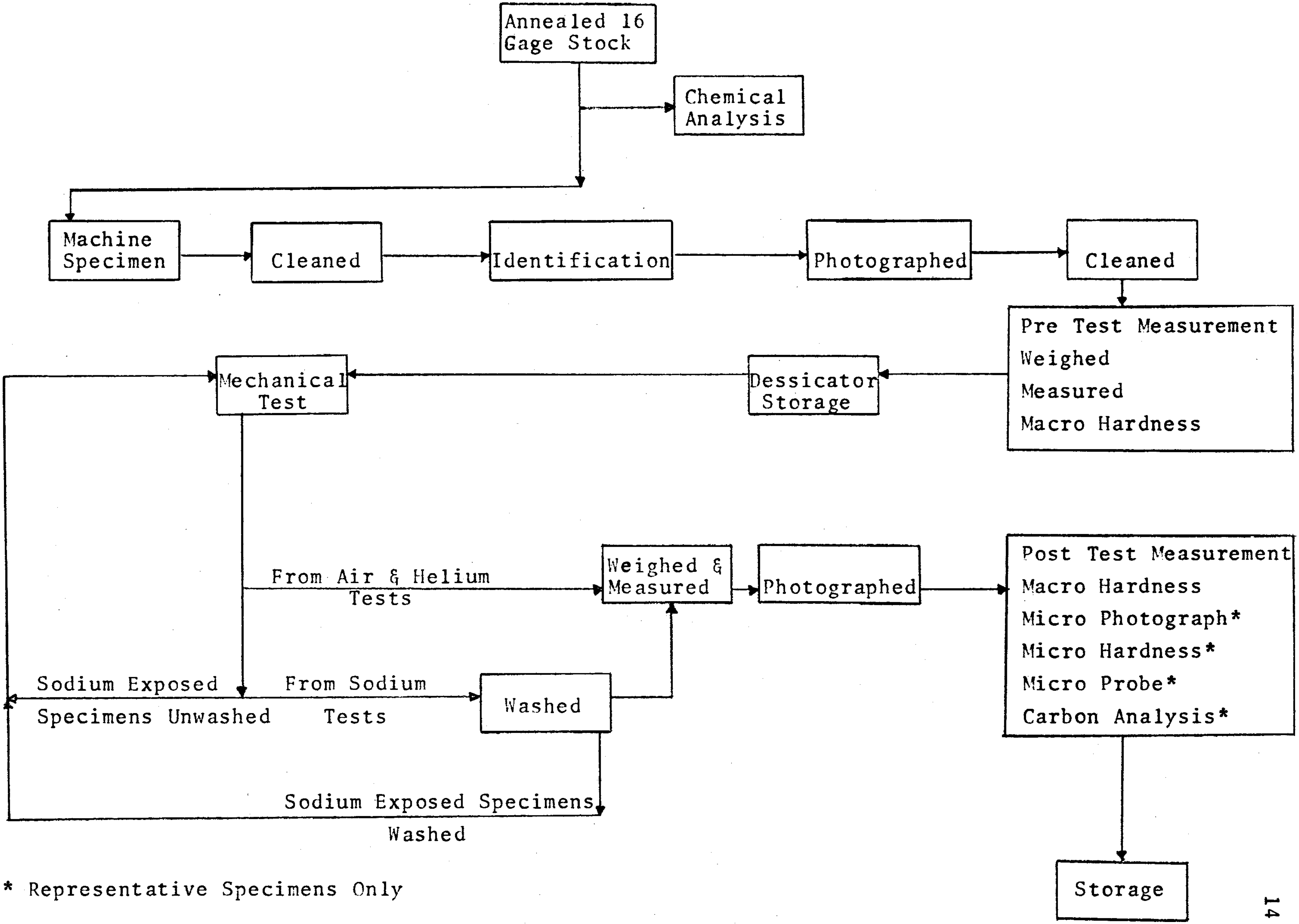
After cleaning, the test specimens were weighed to the nearest milligram. Gage lengths were scribed on the creep, creep-rupture and tensile specimens. This length,along with the gage width and thickness, was measured. Macro-hardness was run outside the gage length on each specimen. Visual examinations were made to detect any flaws or surface defects. All data was recorded on index dards for each test specimen.

4.3.3 Post-Test Handling

The air and helium test specimens were kept in a desiccator as received from the University of Michigan.

The sodium test specimens (with the exception of the exposed, uncleaned tensile test specimens) were taken from the test pots and immersed immediately in alcohol. They were then washed with water, rinsed with distilled water, and washed with alcohol again before being dried and placed in a desiccator.

The exposed, uncleaned specimens were removed from the test pots under cover gas, sealed in plastic bags and placed in a desiccant for shipment to the University of Michigan for tensile testing.



* Representative Specimens Only

Fig. 5 - Specimen Flow Sheet

4.3.4 Post-Test Measurements

Upon completion of a test, the test specimens were again weighed when possible and compared to the original weights. This could not be done in all cases. In the air and helium runs some of the metal was removed in attaching the elongation measurement devices. All of the creep specimens required stiffeners around the pin hole to prevent elongation at this location. These stiffeners were welded to the specimens and made it impossible to determine weight changes of these specimens.

Final measurements were taken and compared to original measurements for most specimens. Final macro hardness was determined for each specimen and compared with the original.

Various metallographic and chemical examinations were made on representative specimens. These varied from micro hardness, carbon analyses, or micro probe analyses depending on the particular interest. Detailed analytical and metallographic studies made on these specimens are discussed under their respective sections.

5. FATIGUE TESTS*

The fatigue testing portion of this program is involved with the determination of the effects of environments on the fatigue properties of the test materials. With this purpose in mind, the controlled radius bending test was selected because this method tends to minimize data scatter and lends itself to relatively short time tests. It was not possible to determine the elastic strain from these tests; therefore, the cyclic strain values are actually a total strain range. Since the purpose was to determine the environmental effects on fatigue life rather than fatigue values, this method appeared justified.

The test procedure requires the clamping of a flat bar type test specimen between two curved surfaces with carefully machined radii. Only one end of the specimen is clamped while the other end extends out beyond the controlled bending surfaces. This free end is gripped in a movable barrel-like holder attached to an actuator rod which, in turn, is attached to a motor drive. The motor, a reversible, capacitor type containing an adjustable eccentric, provides a vertical movement to the actuator rod. The linear stroke of the actuator rod alternately bends the specimen, first over one controlled surface and then the other. The use of this technique is based on the assumptions that (1) the contact between the test specimen and the mandrel, at the compression face of the specimen in bending, will not affect its fatigue life; and (2) the strain distribution in bending will be predicted by fundamentals of strength of materials. The basis for obtaining the cyclic strain as a function of specimen thickness and radius of the

---

* As defined under Section 2.4.

mandrel may be found in "Elements of Strength of Materials."[1] Here the strain is depicted as Y/ρ where:

Y = 1/2 the thickness of the specimen

ρ = radius of curvature.

Where bending is reversed, using two mandrels, the cyclic strain is the sum of the positive and negative strains. Therefore the result is:

$$\text{Cyclic strain} = 2\,\frac{t/2}{\rho} = \frac{t}{\rho}$$

where:

t = test specimen thickness

ρ = radius of mandrel

The results of the fatigue tests to date indicate the above assumptions to be valid and the tests show good agreement with triplicate specimens.

A drawing of the complete test assembly, as finally conceived and constructed, is shown in Fig. 6.

The first section of this unit contained an immersion heater while the second section contained the test specimen. The second section was fabricated from a length of 10 in. Sch 40 pipe, a 10 in. buttweld cap, and a 10 in. I.P.S. weld neck quick disconnect temperature compensating coupling. Sodium leaving the heater section passed through a stainless steel wire screen and over the control thermocouple. The screen was used to prevent pieces of the fatigue specimen from falling into the heater section after fracture. The sodium outlet was located over the baffle in the test section but below the disconnect coupling seal level.

An inner shell welded to the under side of the blind flange was a length of 6 in. Sch 40 pipe with a baffle between the inner and outer shell to insure flow over the test specimen. Sodium outlet holes were provided in this inner shell. The actuator rod guide, mandrel holder and pins for holding specimens during exposure were welded to the lower portion of this inner section. A bellows assembly, welded to the outside of the blind flange, in conjunction with a packing gland seal, permitted passage of the actuator rod through the top flange to the motor drive. A probe and cover gas nozzle were also located in the flange. The DSD Manufacturing Company quick disconnect flange with the actual test assembly attached to the blind portion of this flange permitted removal of the entire mechanical unit for assembly and disassembly.

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[1] Timoshenki and MacCullough, 3rd Ed., 1949, page 119.

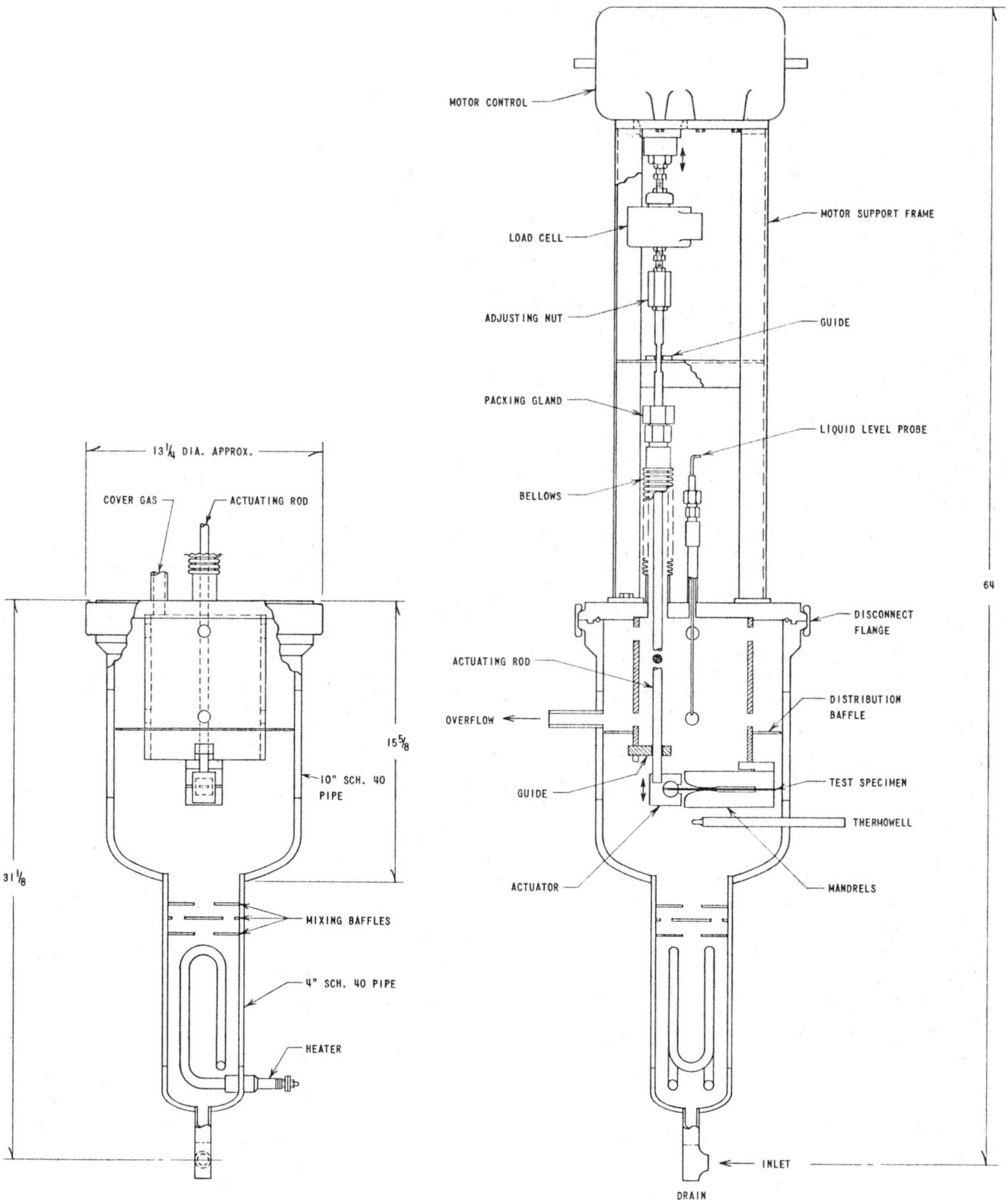

FIG. 6 - FATIGUE TEST UNIT

One such fatigue unit was required for each of the test loops and two additional fatigue units were eventually built and used to conduct the fatigue tests in air and helium environments. These two final units were different from the one in the test loop, inasmuch as the heat source was from electrical resistant radiant type units mounted on the O.D. of the shell of the test unit. During the air tests, air entered at a flow rate of 0.2 CFM through the top flange down through the annulus between the inner 6 in. and the outer 10 in. pipes, over the test specimen, up through the 6 in. pipe and out the top. A thermocouple attached directly to the test specimen mandrel measured and controlled the temperature during testing.

The helium tests were conducted under a static 15 psig pressure of helium gas. The test assembly was heated, evacuated and flushed several times with helium gas before each test run. The helium pressure was maintained at 15 psig from standard helium cylinders. Prior to entering the test unit, the helium was passed through a purification system consisting of a train of hot and cold containers of NaK and knock-out tanks. As a check on the purity of the helium, a series of tests were made using a Minoxo oxygen indicator capable of measuring traces of oxygen (0-100 ppm) in inert gases. The instrument has two ranges, a high range of 0 to 100 ppm and a low range of 0 to 10 ppm. The sensitivity is 0.5 ppm with an accuracy of ±5% of full scale over a 24 hour period. The helium supply for the fatigue machine, when tested directly from the supply cylinder, indicated 2.1 ppm of oxygen present. The helium effluent from the NaK purification unit over a four-hour period showed a decrease to 0.8 ppm. During an actual fatigue test the effluent gas from the fatigue machine was monitored continuously for eight days with the average oxygen concentration of less than 0.4 ppm. These tests indicated the purification system to be both effective and reliable.

The detection of specimen failure is complicated since visual observation of the test specimen during the fatigue tests in sodium is not possible. Even in the cases of tests in air and helium, where windows might have been possible, visual observation would not have helped unless the test specimen could be made to separate at failure. One detection system suitable for all three environments is desirable to reduce variables between tests. The method being used is based on a Baldwin-Lima-Hamilton load cell as an integral section of the actuator rod, located between the specimen holder and motor drive. This unit measures the force being applied to the test specimen during the bending cycle. This force is monitored at the control panel and a sudden reduction of this signal indicates specimen failures. With some exceptions, this failure detection system has been good. The longest fatigue tests are under low cyclic strains, meaning that the mandrels have large radii requiring small forces during bending. The signal from the load cell during these tests is low and in several instances the indication of specimen failures was not as discernible as one would like for obtaining data with a minimum of scatter.

The fatigue tests were based on comparing the results from the three test environments at three different cyclic strain levels (2.1%, 1.0% and 0.56%) from mandrels having radii of curvatures of 3 1/8 in., 6 3/4 in., and 12 in. Fatigue failures were in the ranges of 400, 5000 and 60,000 cycles respectively.

The procedures used for the fatigue runs in sodium were as follows: The test specimen was installed in the mandrel and the test apparatus assembled. The test unit was evacuated, purged with helium, preheated to ~600 F and charged with sodium from the sump tank. The immersion heater was turned on and the temperature of the test unit raised to 1200 F. The OCI system was put into operation, since the oxygen level in the sodium system increased each time a test unit was put on stream. After operating temperatures were reached in the test unit, flow was established and cycling of the test specimen was initiated. Cycling continued until the load cell indicated specimen failure.

Upon failure, the test unit was isolated from the remainder of the system and drained at the test temperature. The unit was cooled, disassembled and washed with alcohol and water. The specimen was washed with alcohol and water, dried, and placed in a desiccator.

A similar procedure was used for the air and helium tests. In the case of helium, after assembly a vacuum (<1 mm Hg) was pulled on the test unit while the temperature was increased to 600 F. Outgassing was permitted for at least one hour and then the vacuum was broken with helium. The temperature was increased to 1200 F and cycling was initiated. Most of the specimens did not retain their original bright finish throughout the testing in helium. Therefore, approximately midway through the program, zirconium chips were placed in the test unit to act as a scavenger for any oxygen that might diffuse into the test unit. No consistancy was ever achieved in maintaining the bright finish of the specimens throughout the tests; however, the degree of discoloration could never be correlated with failure times.

During the preliminary phases of the fatigue test program, several mandrels of various radii were used before arriving at the proper ones for the various levels of strain desired. While examining the feasibility of 100,000 cycle tests, it was found that, although the bellows purchased had an estimated cycle life of 30,000-150,000 cycles, most bellows failed below 100,000 cycles. Also, the load required to deflect the test specimen over this mandrel was so small, it was difficult to separate this strain on the load cell from that required to deflect the bellows, making it extremely difficult to detect failure. Furthermore, the effects of creep on the long-time cyclic test could become pronounced. It was on these bases that the low strain level was raised to promote failure in approximately 50,000 cycles rather than 100,000 cycles.

The fatigue cycles for the different strain levels were:

Cyclic Strain %	Mandrel Radius In.	Cycle Time - Seconds Up	Hold	Down	Hold
2.1	3 1/8	5	5	5	5
1.0	6 3/4	3	7	3	7
0.56	12	5	5	5	5

5.1 FATIGUE TEST RESULTS

As described in earlier sections, the fatigue tests were conducted at three different levels of strain (2.1, 1.0 and 0.56%) on original material in each of the three environments (air, helium and sodium) with triplicate specimens being run at each condition. In addition, triplicate specimens were run at the highest strain level (2.1%) in each environment after being exposed to 1200 F low oxide (30 ppm) sodium for 286 hours.

The results of these tests are tabulated in Table 4. Fig. 7 is a plot of the data on original material as per cent cyclic strain (as defined earlier) vs the number of cycles to failure. Fig. 8 shows the same curves minus the data points for the original material, but with the data points of the exposed specimens shown for comparison.

The conclusions that can be drawn from the results of the fatigue tests are:

a. The fatigue life of 316 ss in 1200 F helium is considerably longer than in 1200 F air throughout the range of cyclic strains tested. The increase in fatigue life varies by a factor of 1.9 at the high cyclic strain to 4.9 at the low cyclic strain.

b. The fatigue life of 316 ss in 1200 F sodium is the same as in air at high cyclic strains, but is the same as in 1200 F helium at the low cyclic strains. The relative standard deviations of the triplicate specimens were higher in sodium than the other two mediums.

c. The general shape of the 316 ss fatigue curves indicates the possibility that at very low cyclic strains the differences between the three environments may not be significant. However, with results from only three cyclic stress levels, it is difficult to extrapolate the data beyond the limits of this test program.

TABLE 4 - HIGH TEMPERATURE CYCLIC STRAIN TEST DATA

316 ss Specimen	Condition (1200 F)	% Cyclic Strain	Cycles to Failure	Average Cycles to Failure	Standard Deviations
3FAX2	Air	2.18	599		
3FAX3	Air	2.18	600	590	17
3FAX4	Air	2.18	570		
3HAL1	Air*	2.11	296		
3HAL2	Air*	2.12	302	308	16
3HAL3	Air*	2.12	326		
3FHX4	He	2.13	1325		
3FHX5	He	2.19	848	1098	239
3FHX6	He	2.14	1122		
3HHL1	He*	2.13	1116		
3HHL2	He*	2.16	1233	1033	253
3HHL3	He*	2.11	749		
3FLX1	Na-He	2.16	615		
3FLX2	Na-He	2.18	275	464	141
3FLX3	Na-He	2.16	492		
3FLX4	Na-He	2.16	475		
3HLL1	Na*-He	2.18	700		
3HLL2	Na*-He	2.16	751	665	107
3HLL3	Na*-He	2.16	545		
3GAX7	Air	1.00	1498		
3GAX8	Air	1.00	1663	1554	95
3GAX9	Air	1.00	1500		
3GHX2	He	1.00	9693		
3GHX3	He	0.99	9058	8856	995
3GHX4	He	1.00	7816		
3GLX1	Na-He	.985	2385		
3GLX2	Na-He	1.00	2110	3130	1029
3GLX3	Na-He	.995	3914		
3GLX4	Na-He	1.01	4112		
3JAX5	Air	.554	8956		
3JAX6	Air	.568	8060	8524	449
3JAX7	Air	.565	8556		
3JHX1	He	.558	50,004		
3JHX2	He	.571	38,804	41,940	7041
3JHX3	He	.573	37,011		
3JLX1	Na-He	.565	55,925		
3JLX2	Na-He	.556	29,400	39,460	11,698
3JLX3	Na-He	.558	33,055		

* Specimen pre-exposed to 1200 F sodium for 286 hours.

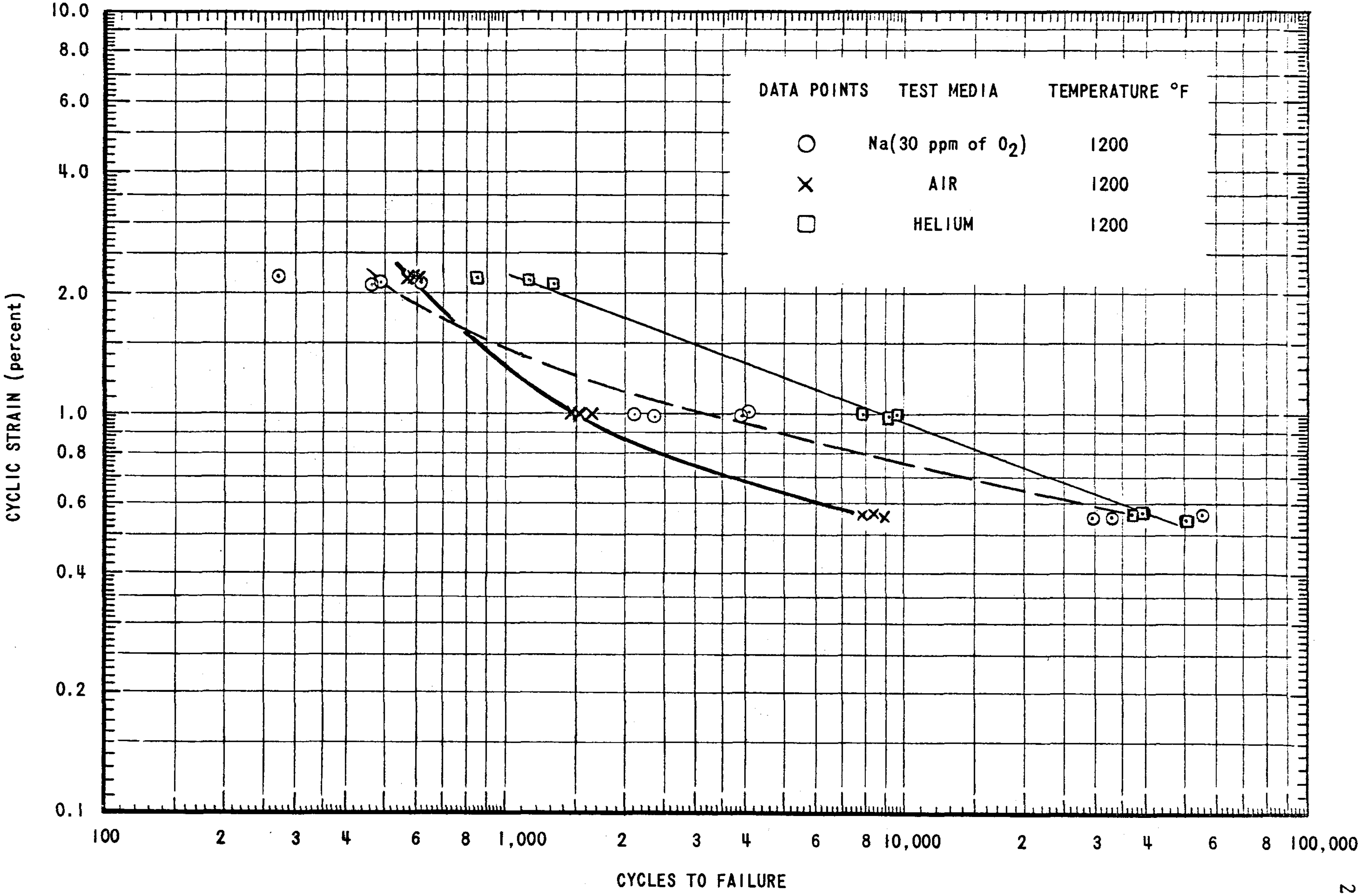

FIG. 7 - HIGH TEMPERATURE CYCLIC STRAIN (FATIGUE) TESTS

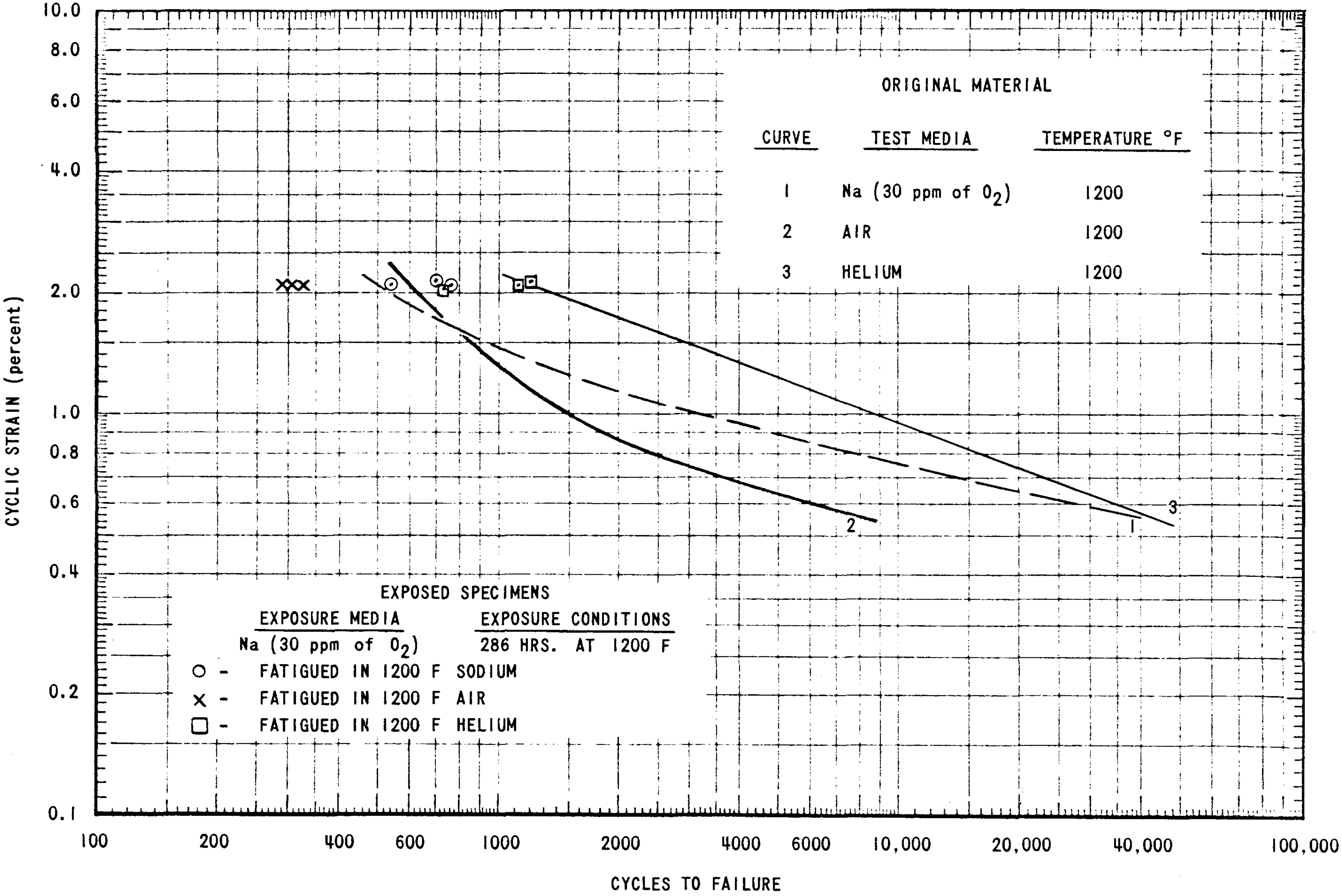

FIG. 8 - HIGH TEMPERATURE CYCLIC STRAIN (FATIGUE) TESTS - Comparison of Exposed Specimens Failure Points at High Cyclic Strain with Fatigue Test Curves

d. The 316 ss specimens exposed for 286 hours in 1200 F sodium and then tested in air, helium and sodium showed generally:

1. A shorter life when tested in 1200 F air.

2. A trend is suggested toward longer life in 1200 F sodium but, considering the standard deviations for both the exposed and original material, the existence of such a trend would be difficult to substantiate.

3. The exposed specimens indicated no change when tested in 1200 F helium.

5.2 METALLOGRAPHIC ANALYSIS OF FATIGUE SPECIMENS

Metallographic examination has been the primary tool utilized in evaluation of fatigue failure of 316 stainless steel. Fatigue is that property that has been measured in the program that is thought to be most sensitive to environmental effects. Metallographic examination of fatigue specimens has been performed at several magnifications to enable evaluation of semi-micro as well as micro effects. In all of the following photomicrographs, ferric chloride was used as an etchant.

Figs. 9, 10 and 11 show longitudinal sections of the failure of specimens fatigued in 1200 F air, in the order of decreasing stress levels with resulting increase in cycle life. Surface cracking is less apparent with decreasing strain, although surface cracks are still apparent at the lowest strain level. Internal voids are evidenced with increasing strain.

Fig. 12 shows a specimen fatigue tested in a 1200 F helium environment, while Fig. 13 shows a specimen fatigued to rupture in 1200 F sodium. Both of these specimens were tested at a cyclic strain approximately equivalent to that shown in Fig. 9. A few surface cracks exist in the helium tested specimen while none are apparent in the sodium tested specimen. Void formation is apparent in both of these samples.

Fig. 14 shows a specimen that had been exposed to 1200 F sodium for 286 hours, was subsequently washed in water, dried, and fatigue tested in 1200 F air. Surface cracking is more prevalent than the air test sample (Fig. 9) not previously exposed to sodium.

Fig. 15 shows a fatigue crack in an air tested sample. The fatigue crack is transgranular and shows no branching. A grey deposit, which is thought to be air oxidation product exists within the crack system near the crack-tip. Such fatigue cracks,which were found to be prevalent in air-test specimens, were only apparent

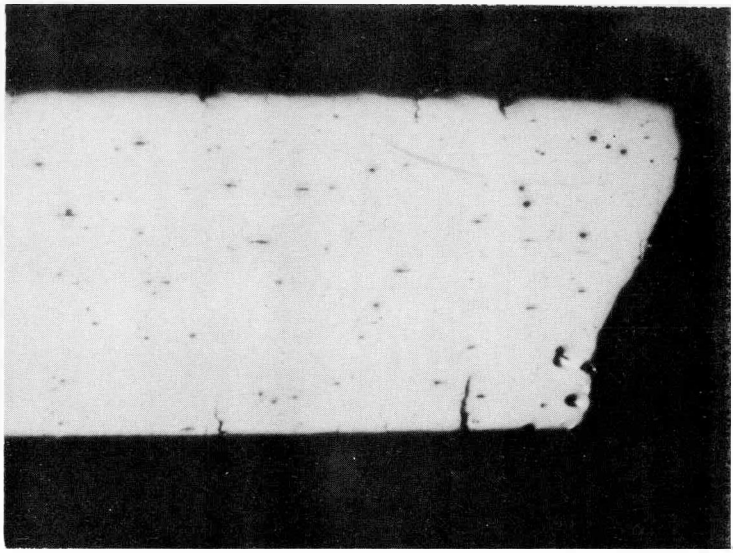

FIG. 9 - FATIGUE IN 1200 F AIR (3FAX2)
Cyclic Strain - 2.18%
Cycles to Failure - 599
Mag. - 20X

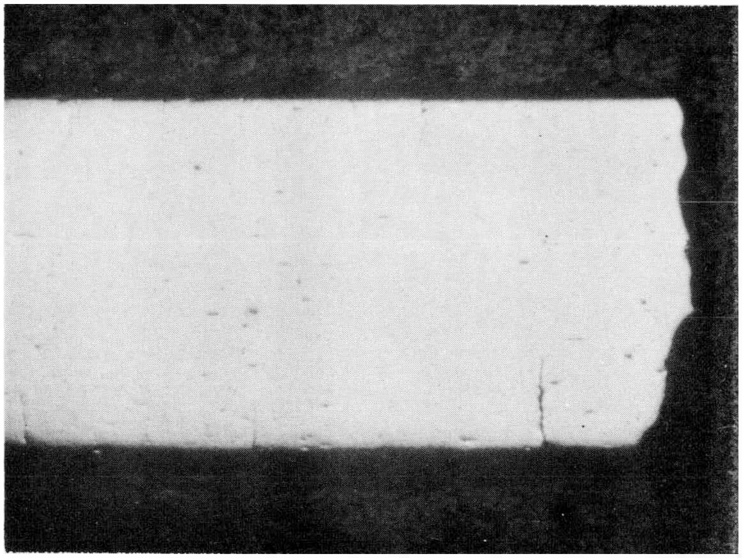

FIG. 10 - FATIGUE IN 1200 F AIR (3GAX9)
Cyclic Strain - 1.00%
Cycles to Failure - 1500
Mag. - 20X

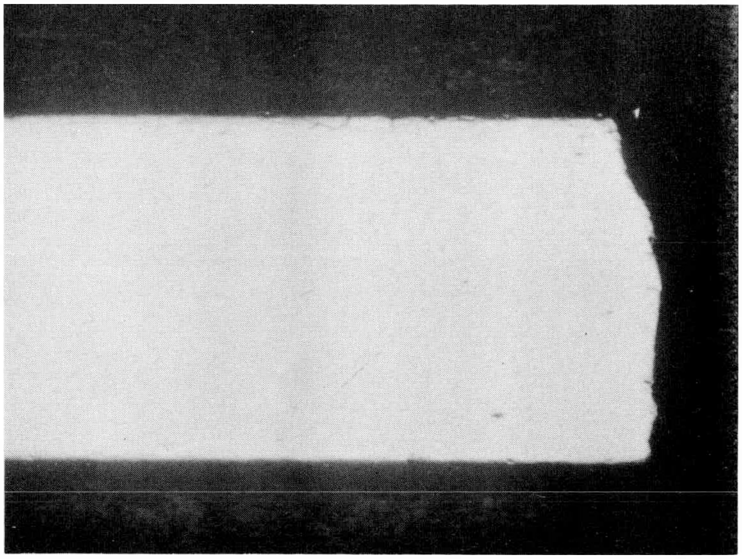

FIG. 11 - FATIGUE IN 1200 F AIR (3JAX7)
Cyclic Strain - 0.57%
Cycles to Failure - 8556
Mag. - 20X

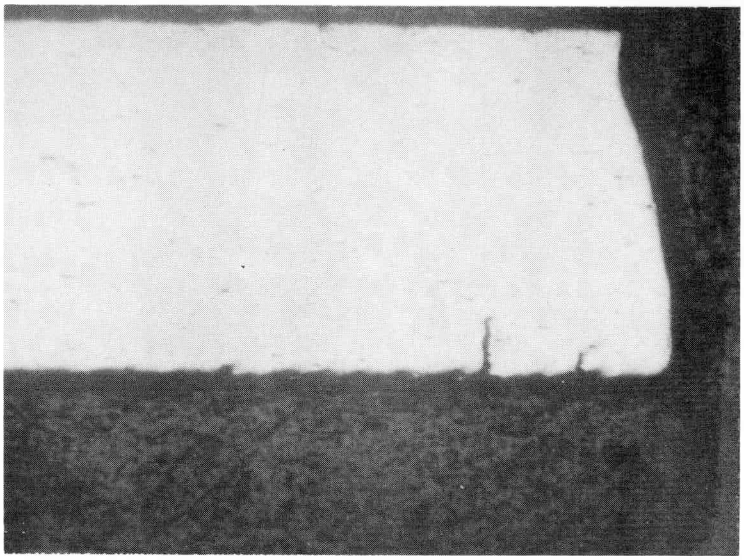

FIG. 12 - FATIGUE IN 1200 F He
(3FHX4)
Cyclic Strain - 2.13%
Cycles to Failure - 1325
Mag. - 20X

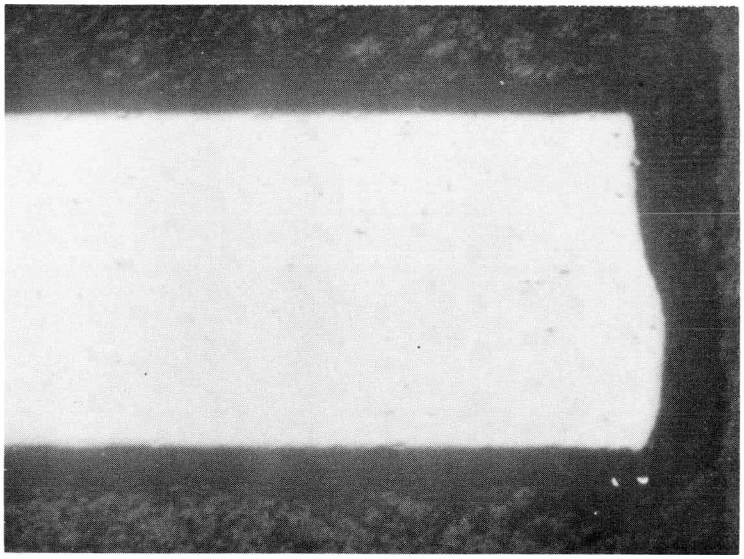

FIG. 13 - FATIGUE IN 1200 F Na
(3FLX3)
Cyclic Strain - 2.16%
Cycles to Failure - 492
Mag. - 20X

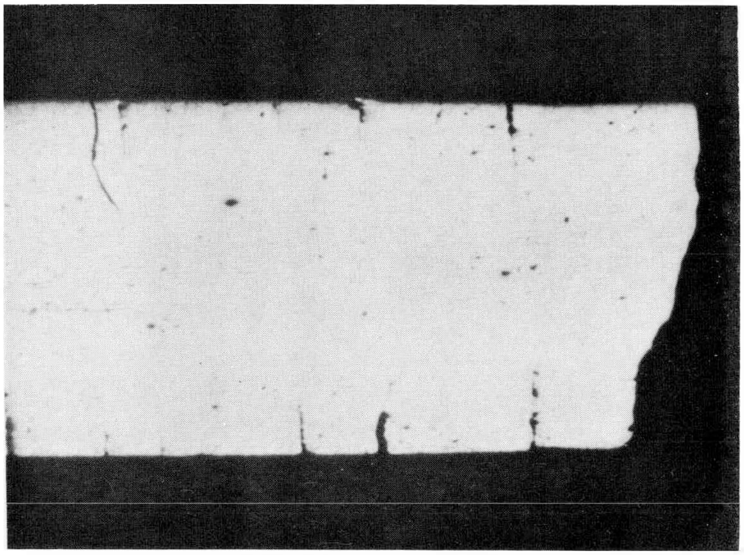

FIG. 14 - EXPOSED TO 1200 F Na
(286 hours) AND FATIGUE
TESTED IN 1200 F AIR
(3HAL2)
Cyclic Strain - 2.12%
Cycles to Failure - 302
Mag. - 20X

in examination of longitudinal sections. The voids that had been revealed at 20X magnification are seen as cracks which are parallel to the stress direction and normal to fatigue cracks. Examination of such specimens suggests that the majority of such voids are formed at grain boundaries.

Fig. 16 shows a section of a specimen that had been pre-exposed to sodium, thoroughly washed with alcohol and water, and subsequently fatigued in 1200 F helium. Sensitivization of the stainless to a depth of approximately 1 grain has occurred. The specimen, shown in Fig. 17, received the same pre-exposure treatment as that in Fig. 16, and was subsequently fatigued in air. Sensitivization to a depth of 1 grain is again evident. Three transgranular cracks are seen in this section, with two of the cracks filled with corrosion product. Some branching of cracks has occurred.

Fig. 18 shows a section that had been fatigued in 1200 F sodium without any prior pre-exposure treatment, while Fig. 19 shows a specimen fatigued in helium at the same stress level. No surface cracking is evident in these photomicrographs and none was observed in either sample. Internal voids are apparent, while sensitivization at the surface is not apparent. A very shallow surface phase is found in the sodium test, which is thought to be predominantly carbide formed during 212 hours of sodium exposure.

Fig. 20 shows a longitudinal section of a fatigue specimen at the fracture. A slight degree of plastic deformation is evident at the upper portion of the fracture. No signs of plastic deformations were indicated in specimens fatigued at cyclic stresses less than 2%. At a cyclic stress level of 2% failure is initiated by fatigue cracks, while final separation occurs plastically.

Microhardness measurements were made on several of the fatigue specimens. Hardness traverses of all the specimens examined were uniform with the exception of pre-exposed samples, where a slight increase in surface hardness ($\Delta H_V = < 10$ units) was noted in those specimens pre-exposed to sodium for 286 hours. Slight increases in hardness were noted in regions immediately adjacent to fatigue cracks and at the fracture face.

Conclusions

The mode of initiation of fatigue fracture and the mechanisms associated with extension of fatigue cracks is not well understood. Models for fatigue cracking has been produced by many, such as Orowan[2] and Freudenthal[3]. Orowan proposes highly localized

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[2] Orowan, E., Proceedings of the London Royal Society, Vol. 171A, pp 79-105 (1939).

[3] Freudenthal, A. M. and Dolan, T. J., Fourth Progress Report, ONR, Contract N6-ori-71, Task IV (1948).

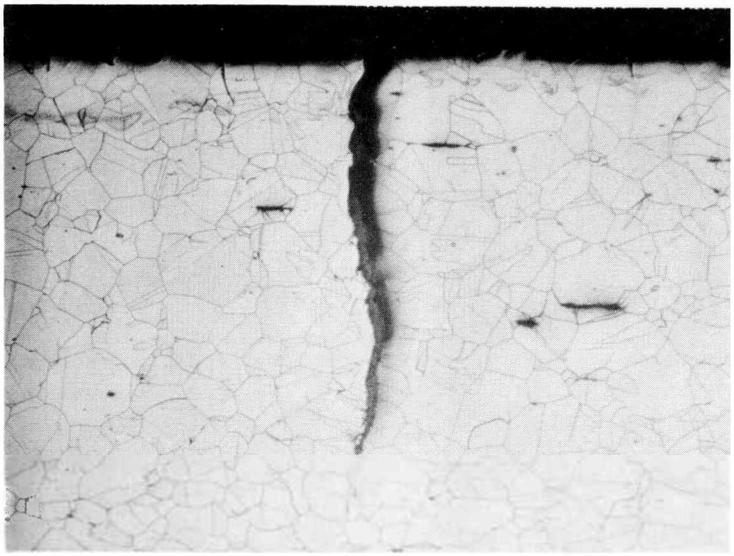

FIG. 15 - FATIGUE IN 1200 F AIR
100X Mag. of Sample
in Fig. 9
Note grey air oxidation
product near tip of fatigue
crack.

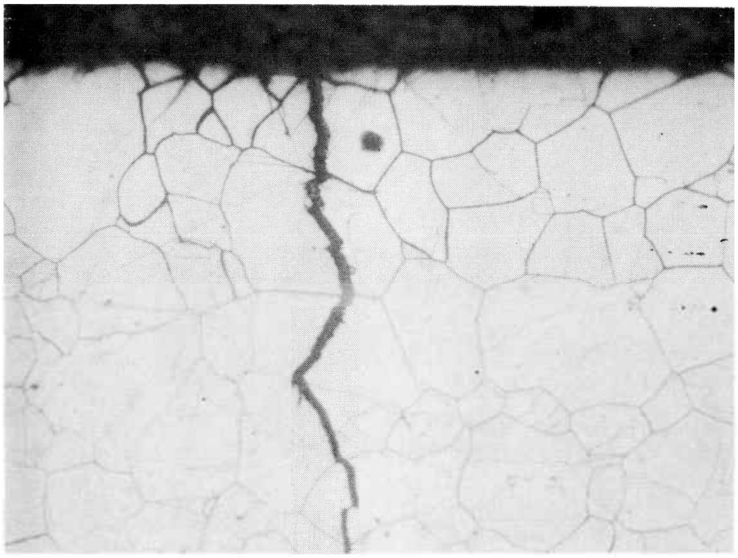

FIG. 16 - EXPOSED TO 1200 F Na
(286 hours) AND FATIGUE
TESTED IN 1200 F HELIUM
Cyclic Strain - 2.11%
Cycles to Failure - 749
Mag. - 200X

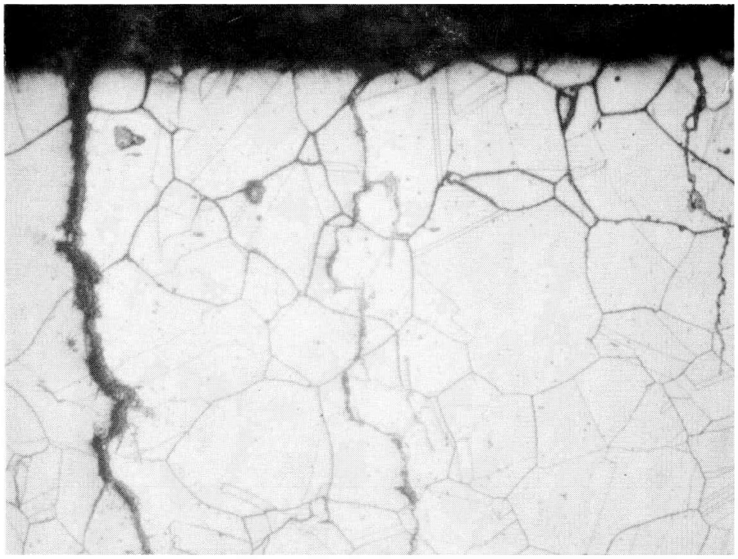

FIG. 17 - EXPOSED TO 1200 F Na
(286 hours) AND FATIGUE
TESTED IN 1200 F AIR
200X Mag. of Sample
in Fig. 14

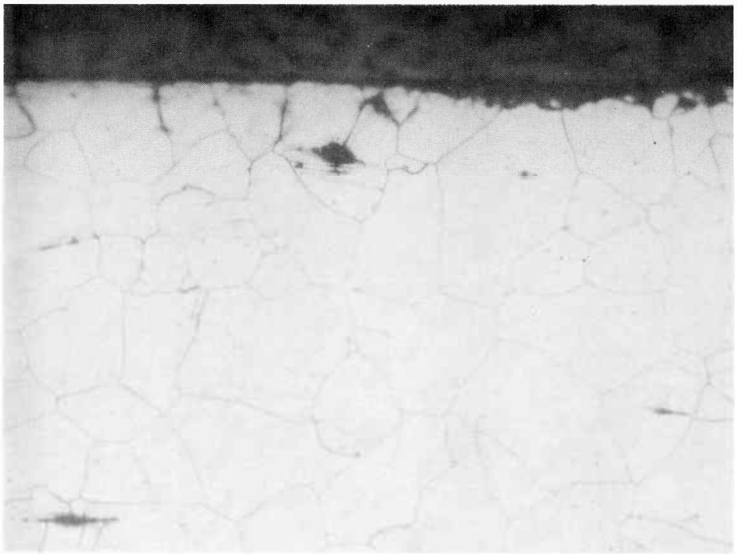

FIG. 18 - FATIGUE IN 1200 F Na
Cyclic Strain - 0.56%
Cycles to Failure - 33,055
Mag. - 200X

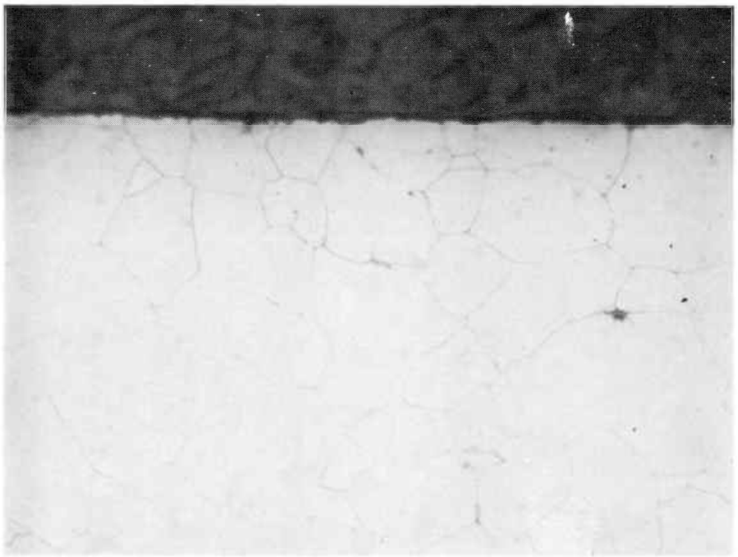

FIG. 19 - FATIGUE IN 1200 F He
Cyclic Strain - 0.57%
Cycles to Failure - 38,804
Mag. - 200X

FIG. 20 - EXPOSED TO 1200 F Na
(286 hours) AND FATIGUE
TESTED IN 1200 F HELIUM
Cyclic Strain - 2.13%
Cycles to Failure - 1116
Mag. - 200X

plastic inhomogeneities that result in cumulative deformation under succeeding cycles until a self-propagatory crack is formed. Freudenthal proposes a mechanism which involves fragmentation of individual crystallytes.

While the mechanisms of fatigue failure are little understood, some of the causes resulting in fatigue failure include:

1. Surface imperfections.
2. Notches in the specimen which can be defined as macroscopic surface imperfections.
3. Non-uniform loading resulting in localized stresses.

Mechanical fatigue cracking occurs by transgranular extension of surface cracks into the body of the metal until two opposite crack systems reduce the effective cross-sectional area of the body until plastic fracture occurs. Thus, fatigue failures appear to have both a smooth or rubbed surface where transgranular fatigue cracks had existed and a jagged surface texture where plastic deformation had occurred.

The effects observed in this study appear to be those normally observed in mechanical fatigue failure, even though the test was performed at temperatures where creep can affect the cyclic test. Superimposed over such cracking, environmental effects have been observed and these effects appear to be responsible for accelerated failure. Environmental effects become particularly marked under the conditions of fatigue testing employed in this study. The conditions include maintaining the specimen in the flexed position for a considerable part of the overall cycle. For instance, at low cyclic strains (0.5%) the surface under tension is maintained in the flexed position for 7-8 seconds, or approximately 40% of the duration of the total 20 second cycle. Where surface cracking has been initiated and there can be reaction of the environment with freshly formed metal surfaces, maintaining an incipient crack under tension can permit extensive reaction of the environment with freshly formed metal surfaces at the crack-tip.

In the use of metals as structural materials in sodium heat transfer systems, the service conditions that are likely to be encountered are such that vibrations result in flexure of the structural material at low amplitude and for cycles in excess of 10^4. Thus, examination of the experimental data at low cyclic stresses becomes more important than that data for high cyclic stresses. It is under such conditions that environmental effects become most marked.

Metallographic examination of specimens points to a direct correlation between the extent of surface cracking and the cycle life of a specimen. In each of the environments, the extent of surface cracking decreases with decreasing strain. At any stress level surface cracking is more extensive when the specimen is tested in air as compared to helium or sodium environments.

Where specimens have been pre-exposed to a sodium environment, and were subsequently washed and tested in either helium or sodium, the mechanical fatigue cracks which normally result appear to be accompanied by a form of stress-corrosion. It is felt that alkali metal removal after sodium exposure is not complete. Weeping of caustic from steels that had been exposed to sodium and were subsequently washed is a known phenomena. Incomplete caustic removal in the water wash step is thought to be the result of surface irregularities. Retained caustic appears to contribute to extension of mechanical fatigue cracks into the specimen, and is observed with pre-exposed helium specimens (Fig. 16) as well as pre-exposed air specimens (Fig. 17).

A mechanism is proposed for the lower fatigue life (at low cyclic stresses) of Type 316 ss in air as compared to either a helium or sodium environment. As seen in Fig. 15, air corrosion products can be formed within a fatigue crack when the specimen is cycled in 1200 F air. When the surface at such a crack is under compression, the presence of oxides results in a wedge. Highly localized stresses are thought to result at the crack-tip which results in tearing of metal at the tip and extension of the crack system. When this surface is maintained under tension, the freshly formed metallic surfaces can be oxidized and more corrosion products are formed within the crack.

6. CREEP-RUPTURE TESTS

The creep-rupture tests were conducted in air, helium and sodium at 1200 F.

All creep-rupture tests in air and helium were run on standard test machines located at the University of Michigan. For these tests, a modified Martens-type extensometer was used. Pairs of extension rods were attached to the upper and lower extremities of the gage section on both sides of the specimen. These rods extended out of the furnace. A mirror was mounted between each pair of extension rods outside of the furnace. The relative movement of the rods attached to the upper and lower extremities of the gage length caused the mirror to rotate. The change in scale readings was read with a telescope. A high degree of sensitivity was obtained from the multiplication of the optical beam. The degree of sensitivity could be controlled by the size of the mirror mounted between the extension rods. The sensitivity could, therefore, be controlled to the requirements of each test with a maximum sensitivity of three millionths of an inch per inch of gage length for a 2-inch gage length. For tests in helium, the extensometer was inside a chamber with a window for sighting on the mirrors. The extensometer has been standard equipment at the University of Michigan for many years with entirely satisfactory results.

All the tests in sodium were conducted in test units as shown in Fig. 21. The necessity of installing and removing test specimens at various intervals suggested the use of a mechanical joint which permitted the removal of the top of the test units. To locate the mechanical joint out of the sodium meant that each test unit had a cover gas space. The test units were therefore designed and located such that the sodium entered the bottom of the units and flowed upward and out a side nozzle. The sodium flowed by gravity out of the test unit into the instream expansion tank.

Six such test units in parallel were required to complete the tests in the required time schedule. Fig. 22 shows these test units as they appeared during the construction of the loop.

The various lengths in testing times indicated that each of the test units should be capable of complete isolation from each other. The test units were charged and drained independently without disturbing the other units. Separate fill and drain lines, along with isolation valves, were provided.

A Type 316 ss metallic bellows was used in conjunction with a Conax mechanical packing gland to seal the 1/2 in. diameter pull rod which connects the test specimen through the top of the test unit to the stress machine. The bellows had an averaged spring constant of 100 pounds per inch of travel and, in order to eliminate this force from the pull rod, the elongation of the bellows was measured and the resulting stress compensated for throughout the various tests. During the creep-to-rupture tests, the bellows were readjusted to the neutral point after 1/16 inch of travel.

The temperature condition of 1200 F within an accuracy of ±3°F at the test specimen was not an easy condition to meet. The major heat to the main system was supplied by means of a centrally controlled immersion heater unit. However, to eliminate the effects of heat losses and control lags between this heater and the test units, a separate and individually controlled immersion heater was located in the test unit itself. Three baffles in series were located downstream of the immersion heaters to insure good mixing. A dual thermocouple between the last baffle and the test specimen recorded and controlled the sodium temperature.

The test machine itself was a standard Arcweld* Model K, 6000 lb capacity creep-rupture machine with a 10:1 ratio lever arm equipped with a load weight elevator for manual operation and a switch assembly for determination of elevator position with respect to load weights. The test machines were calibrated by a factory representative prior to each test series.

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* The company name has since been changed to Satec Corporation.

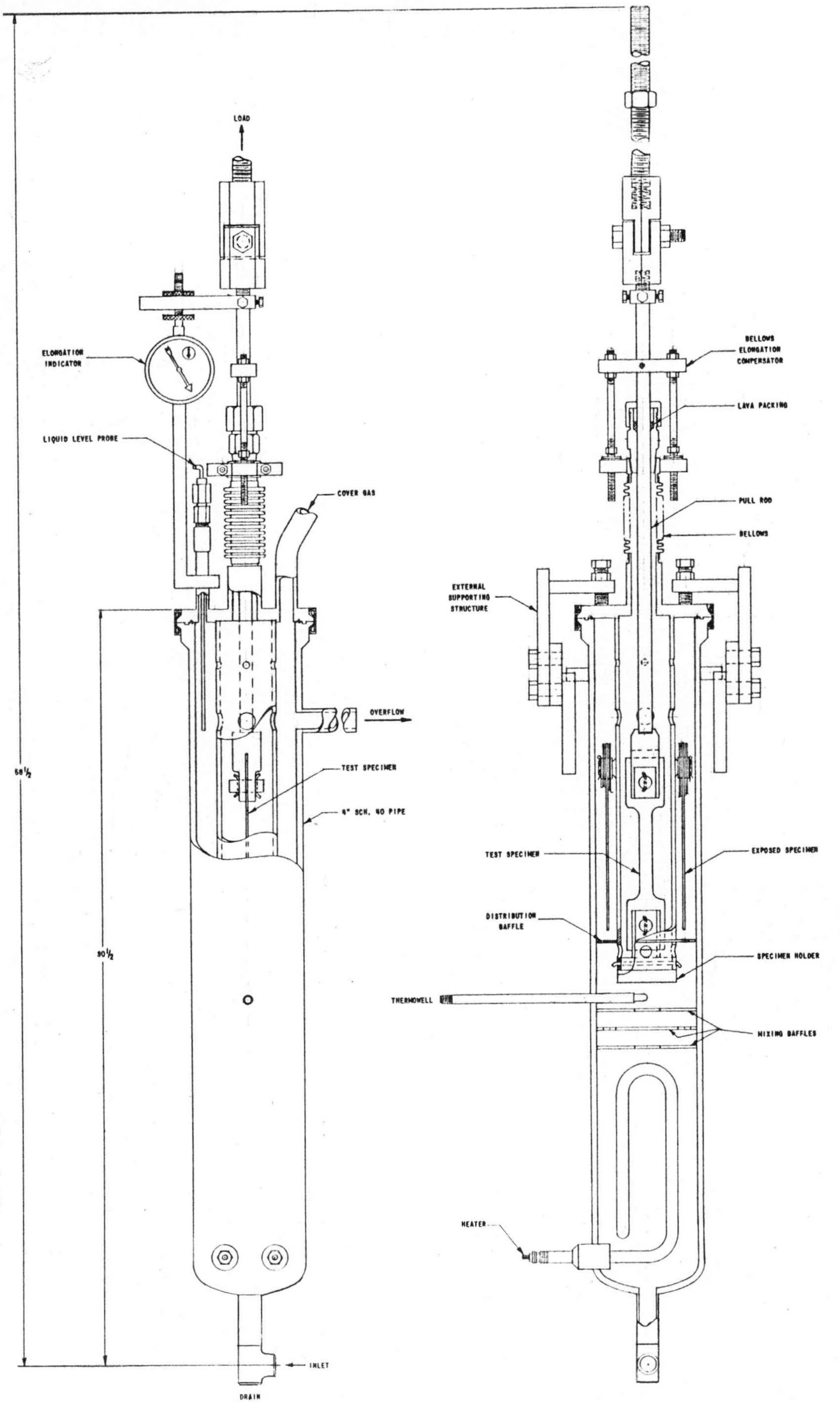


FIG. 21 - CREEP AND CREEP-TO-RUPTURE TEST UNIT

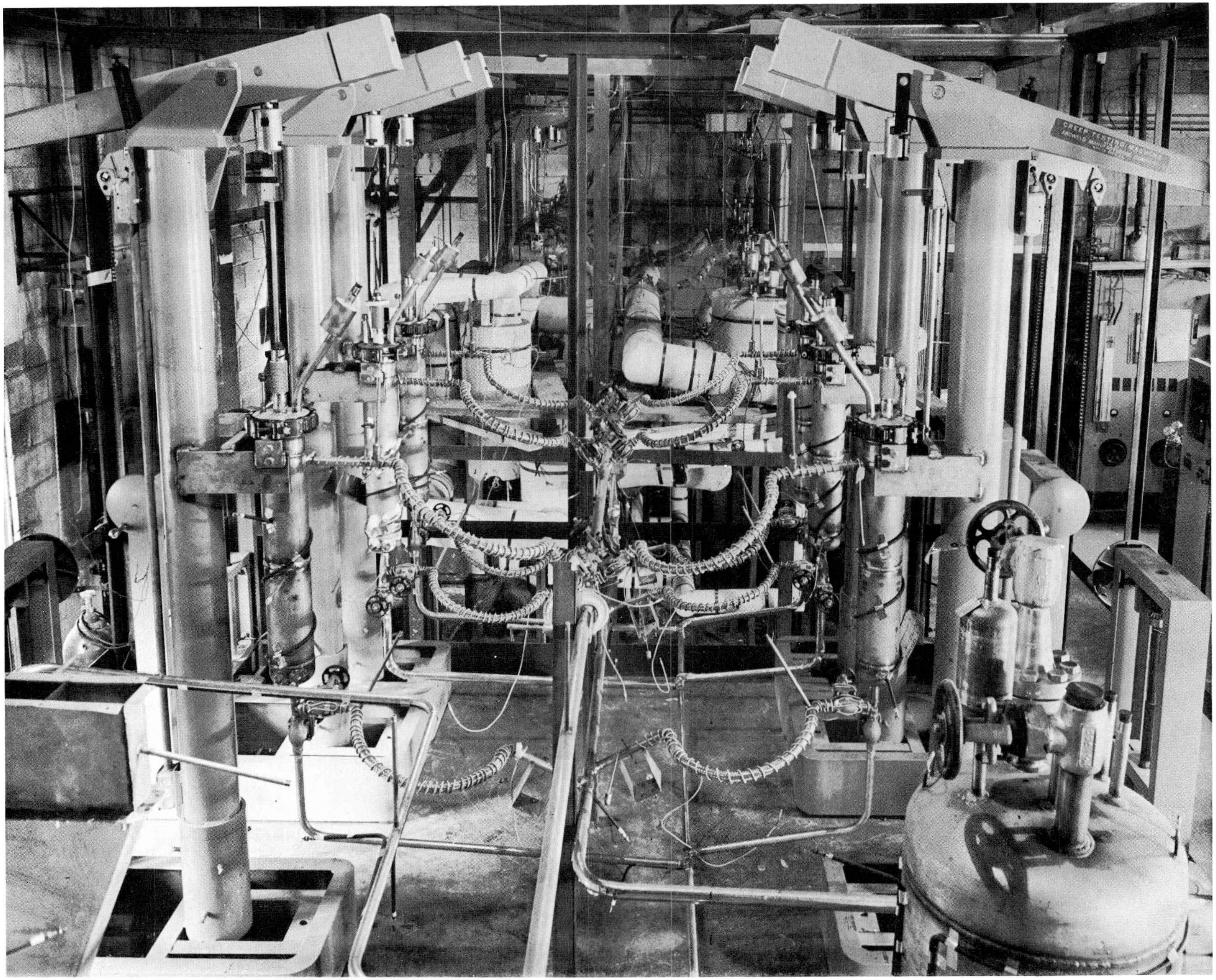


FIG. 22 - PHYSICAL PROPERTIES TEST LOOP DURING CONSTRUCTION

After assembling the test apparatus, with the specimens still unstressed, the test chamber was put under a helium atmosphere by alternately pulling a vacuum and purging with helium. The unit was then preheated to approximately 600 F and charged with sodium from the sump tank.

While the test unit temperature was being increased to test conditions, the OCI system was activated and the inlet and outlet lines of the test unit were preheated. When test conditions were reached in the test unit, flow was established by opening the inlet valve and equilibrium at the desired test conditions attained in the unit. At this time, the stress load was applied to the test specimen in a pre-determined series of steps.

6.1 CREEP-RUPTURE TEST RESULTS

Single rupture tests at six different stress levels were run on original material in the three test environments at 1200 F. In addition, duplicate tests were run in air at three stress levels to establish the uniformity of the test specimens. Single rupture tests were also performed in air, helium and sodium at three stress levels on 316 ss specimens after exposure for 4000 hours to low oxide (30 ppm) 1200 F sodium.

The results of these tests are summarized in Table 5. All the data on the original material is shown graphically in Fig. 23 as the stress load against the rupture time. The stress load is based on the initial cross-sectional area of the test specimen. Examination of this data indicates the results to be in such close agreement that there appears to be no significant difference in the creep-rupture properties of Type 316 ss in 1200 F air, helium or sodium throughout the stress range tested.

The data from the creep-rupture tests on specimens exposed unstressed for 4000 hours to 1200 F sodium is shown in Fig. 24 plotted against the stress-rupture curve drawn through the data on the original material. Data points for the results of the tests on the original material are not shown in this figure. These specimens that had been exposed for 4000 hours in 1200 F sodium showed no significant change in creep-rupture properties when tested in 1200 F air. There may be a trend for the exposed specimens to have extended life when tested in 1200 F sodium. The exposed specimens showed more scatter when tested in 1200 F helium but no trends were noted. The results were sufficiently close to the original material so that no definitive environmental effects could be observed.

Examination of elongation and reduction of area data of the specimens was not very revealing. Trends, if any, were difficult to see through the overall scatter of this portion of the data. There appeared to be a trend for the specimens tested in sodium at high stresses to have a significantly greater elongation, but this observation is based on a very limited number of specimens in sodium at the high stresses.

TABLE 5 - CREEP-RUPTURE TEST RESULTS FROM TEST 1

SS Test Specimen	Condition 1200 F	Stress (Psi)	Elong %	Reduction Of Area %	Rupture Time (Hrs)
3CAX10	Air	27,500	61	45	173.6
3CAX2	Air	27,500	58	49	152.8
3CAX9	Air	27,500	52	46	146.5
3CAX3	Air	27,500	63	50	143.0
3DAX4	Air	24,000	40	38	476.2
3CAX6	Air	24,000	60	43	426.2
3CAX7	Air	24,000	44	39	349.7
3CAX1	Air	22,000	33	31	863.0
3CAX8	Air	21,500	33	28	1000.6
3CAX5	Air	21,500	35	38	971.0
3DAX3	Air	20,500	35	29	1387.8
3DAX1	Air	18,500	34	29	2363.0
3DAX2	Air	17,750	39	20	1969.5**
3AAX4	Air	17,750	18	18	3695.1
3EAL1	Air*	27,500	62	46	144
3EAL2	Air*	24,000	54	44	574
3EAL3	Air*	21,500	49	42	985.2
3CAX4	He	27,500	63	46	166.3
3CHX2	He	27,500	53	49	152.8
3CHX3	He	24,000	48	49	697.7
3DHX3	He	24,000	65	54	483.1
3CHX1	He	21,500	52	47	894.3
3DHX1	He	20,000	34	37	1509.5
3DHX2	He	18,000	44	32	2619.9
3DHX4	He	17,750	44	68	3068.1
3EHL1	He*	27,500	57	52	57.6
3EHL2	He*	24,000	48	44	675.0
3EHL3	He*	21,500	49	42	822.2
3CLX1	Na-He	27,500	74	50	144.6
3CLX2	Na-He	24,000	62	49	445.0
3CLX3	Na-He	21,500	70	46	890.7
3DLX2	Na-He	20,000	53	36	1437.0
3DLX3	Na-He	18,500	33	17	2489.5
3DLX1	Na-He	17,750	27	16	2942.2
3ELL4	Na-He*	27,500	71	45	236.0
3ELL3	Na-He*	24,000	73	53	632.0
3ELL2	Na-He*	21,500	73	53	902.5
3ELL1	Na-He*	20,000	65	44	1869.7

* Pre-exposed to 1200 F for 4000 hours

** Molybdenum sheet used accidentally as shielding.

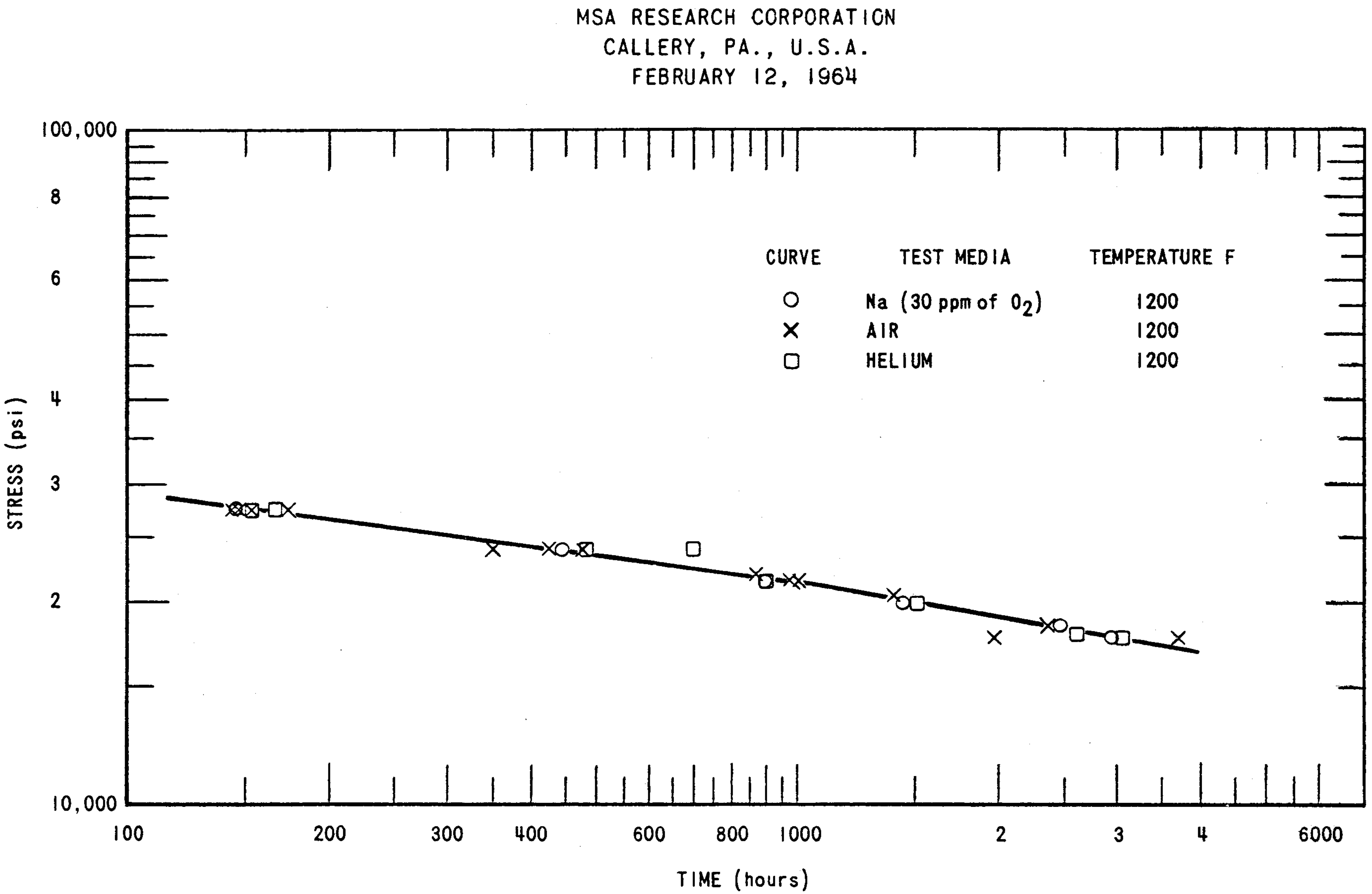


FIG. 23 - CREEP TO RUPTURE OF 316 STAINLESS STEEL SPECIMENS IN AIR, HELIUM AND SODIUM AT 1200 F

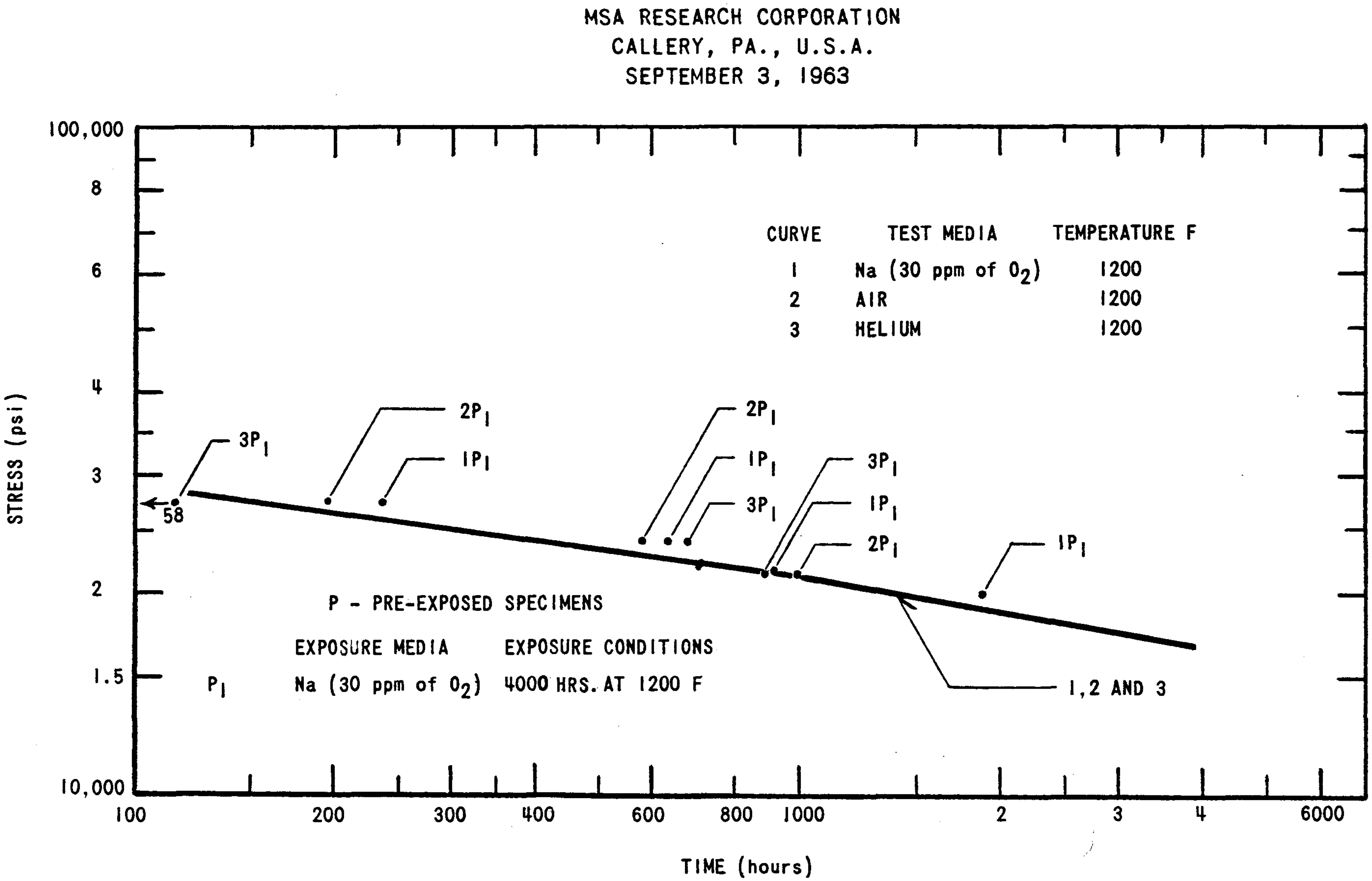


FIG. 24 - CREEP TO RUPTURE OF 316 STAINLESS STEEL SPECIMENS - Comparison of Exposed Specimens Failure Points with Original Material Creep Rupture Curves

6.2 STATISTICAL ANALYSIS OF CREEP-RUPTURE DATA

The creep rupture data for the 27,500, 24,000, 17,750 and 21,500 stress levels were statistically analyzed. The data at 20,000, 18,000 and 22,000 psi was not correlated because these points were not duplicated.

The rupture time variations between air, helium and sodium environments were found to be within normal statistical deviations. Similarly, no effect of the pre-treatment was noted.

This analysis does not rule out a possible difference in rupture times but this can only be shown by making more duplicate runs. For the few samples noted, the differences could have occurred by chance alone.

27,500 Psi Runs

The first analysis made compared the four air and two helium data points. The pooled standard deviation of the data points was 12.3 hours. The deviation of a four-point mean and a two-point mean was found to be 10.65 hours. At four degrees of freedom, the "Student t" test had a value of 0.516 which would occur by chance alone over 50% of the time. Therefore, it was concluded that there was no statistical difference between the air and helium environments.

The air and helium runs were then averaged together so that a more efficient comparison could be made with the sodium environment run.

The comparison of the six helium and air points with the one sodium point had a pooled deviation of 12 hours. The deviation of a six-point mean and a one-point mean had a deviation of 13 hours. The "Student t" value was 0.865. Since this would occur 40 to 50% of the time by chance alone, the sodium environment should be considered to be no different than air or helium.

The air, helium and sodium runs were then averaged to obtain a more sensitive comparison with the specimens exposed for 4000 hours.

Next, each exposed data point was compared individually with the mean of the seven air, helium and sodium data points. The exposed air point was not significant. The observed result would occur 40 to 50% of the time by chance. However, the exposed helium and sodium runs showed a significant difference at the 0.1% level.

Exposing specimens seemed to increase the rupture time of the sodium test and lower the rupture time of the helium test. However, since the exposed data points were only isolated points, the pooled deviation does not consider the spread of the exposed data but instead assumes that the pooled deviations equal the deviation of the helium, sodium and air samples.

Therefore, a comparison was made between the mean of the seven air, helium and sodium points and the mean of the three exposed helium, air and sodium points. The difference was not significant at the 50% level. It is felt that there is indeed no statistical difference between original and exposed specimens.

24,000 Psi Runs

The first analysis compared the three air data points with the two helium points. The "Student t" value was 1.86 and this would occur between 10 and 20% of the time due to chance alone. Therefore, there is no statistical difference between helium and air at 24,000 psi.

The air and helium data points were pooled so that five data points could be compared with the sodium data point. The use of five points rather than four gives greater efficiency.

The sodium data point was not significantly different from the helium and air data points. The difference would occur over 50% of the time by chance alone.

Next, the helium, air and sodium data were pooled and the exposed points were compared. The exposed helium value would occur 10 to 20% by chance. The exposed sodium value would occur 20 to 30% of the time and the exposed air would occur 40 to 50% by chance alone.

The three exposed specimens were compared with the six helium, air and sodium points. The "Student t" was 2.07, which would occur between 5 and 10% of the time by chance alone. More data points are necessary to indicate whether there is a correlation. Any conclusions made before obtaining additional data would be speculation.

21,500 Psi Runs

The difference between the mean of the two air data points and the helium would occur 10 to 20% of the time.

The air and helium points were pooled and compared with the sodium point. The difference would occur 40 to 50% of the time.

The helium, air and sodium points were pooled and compared with the exposed points. The exposed helium difference would occur 10 to 20% of the time. The exposed air and sodium were even less significant.

The pooled exposed points were compared with the pooled air, helium and sodium points and were found not to be significant at the 50% level.

17,750 Psi Run

Since the 18,500 psi air point lies between the two 17,750 points, it is included in the analysis. This gives three rather than two air data points and increases the sharpness of the analysis.

The helium and air data agree at the 50% probability level. The pooled helium and air data agree with the sodium data at the 50% level.

Sign Test Analysis

Even though the deviation at each stress level is dependent on stress and therefore all the stress data cannot be directly pooled, it is still possible to compare the sign of the difference between the air mean and each helium point.

For seven helium points there were four plus three minus, which is not statistically significant. For the five sodium points, there were three plus and two minus, which is also not significant.

There were not enough exposed values to check the pre-tested air, helium and sodium individually. However, for the nine pre-tested points there were five plus and four minus, which again is not significant.

6.3 METALLURGICAL ANALYSIS OF CREEP-RUPTURE SPECIMENS

Statistical examination of creep rupture data of 316 ss in 1200 F air, helium and sodium shows no discernible environmental effects, irrespective of whether the specimens had been pre-exposed to 1200 F sodium (4000 hours) or not. Metallurgical evaluations of this series of tests was primarily directed at eludication of carbon and metal transfer. The metallurgical examination of the creep and creep-to-rupture specimens were correlated and the findings are discussed in Section 7 under the results of the creep tests.

7. CREEP TESTS

The creep tests were conducted in air, helium and sodium. The air and helium tests were conducted on the same machines, using the same procedures as described for the creep-rupture tests. The sodium creep test machines were identical to the sodium creep-rupture machines shown in Fig. 21. One exception is noted. This was the method used in compensating for the spring constant of the bellows. During the rupture tests, the elongation of the bellows was adjusted periodically to keep the error in the load to the specimen at a minimum. This method was necessary since the total elongation of the specimen was greater than could be tolerated by the bellows. During the creep tests, weights were added to the test machine to compensate for the spring constant of the bellows. These weights were added in 1/2 pound increments for each 1/16 inch of bellows elongation. The initiation of the creep test was identical to that previously described for the rupture tests.

One of the more perplexing problems connected with this program was determining the method to be used for the measurement of specimen elongation during the creep tests in sodium. Many methods and designs were considered but almost all had some untried or untested areas with dubious reliability or accuracy. The use of any of these methods without a development and testing program was not considered wise. Such a program would not only have increased the cost but would have delayed the overall program for an unknown length of time.

The use of dial gages, mounted externally but directly to the pull rod, was seen to be a method with faults, but here at least the problem areas were recognized and understood. The major disadvantage to the use of dial gages is their relative location to the actual test specimen, leaving ample room for errors through connections and temperature gradients. Recognizing these factors, the dial gages were used and, as predicted, proved to be sensitive to ambient temperature and small liquid level changes. However, the data from the gages appears quite adequate for measuring the creep rates. Fully jeweled dial gages with a dial range of 0.100 inches with 0.0001 inch divisions were used.

Following the charging of the test unit and loading of the specimen, as described in Section 6 for the creep-rupture specimens, the dial gage settings were adjusted to zero. Throughout the 4000 hour test, the dial gage reading for each specimen was recorded every two hours. The actual total elongation readings were averaged over a 25-hour period and divided by the gage length of the specimen. The elongation (inch per inch of specimen gage length) was then plotted against the test time. An example of this data is shown in Fig. 25 where a portion of the data for the 11,500 psi creep test in sodium is shown.

MSA RESEARCH CORPORATION
CALLERY, PA., U.S.A.
JANUARY 7, 1964

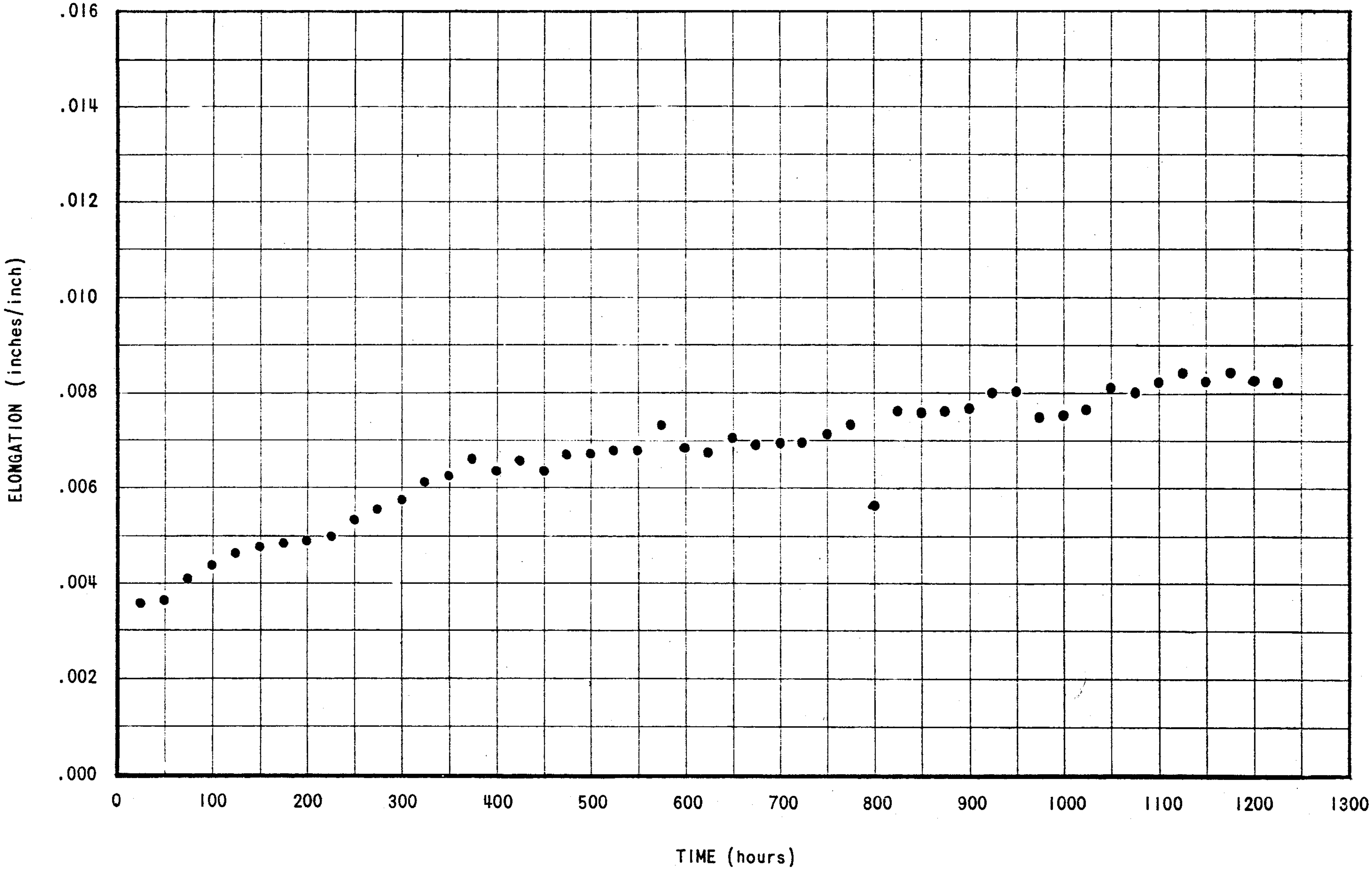


FIG. 25 - CREEP OF STAINLESS STEEL SPECIMEN AT 11,500 psi STRESS IN 1200 F SODIUM

7.1 CREEP TEST RESULTS

The creep rate per hour for each specimen was derived by determining the slope of the curve drawn through the data points of the elongation (inch/inch) versus time (hours) plot. The creep rate was determined every 200 hours and plotted against the test time. The creep rates of the three environmental tests at the various stress levels are shown in Figs. 26, 27 and 28. For comparable stress levels, the creep rates in helium and sodium were consistently higher than the creep rates in air.

Comparing the minimum creep rates obtained for each stress level shows the sodium and helium runs with comparable minimum rates while those for air were lower. Fig. 29 is a plot of the resulting minimum rates versus the test stress. Using this plot, the stress necessary to produce a minimum creep rate of 0.1% per 1000 hours, a condition of the creep test, was determined for each environment. These values are as follows:

Environment	Stress to Produce 0.1% Min. Rate in 1000 hours
1200 F Air	12,300 psi
1200 F Sodium	10,900 psi
1200 F Helium	10,650 psi

While the creep rates in helium and sodium proved to be consistently higher than in air, the results indicate the stress to produce an 0.1% minimum creep rate in 1000 hours to be within 15% of the corresponding stress for the same creep rate in air.

7.2 METALLURGICAL ANALYSIS OF THE CREEP AND CREEP-RUPTURE SPECIMENS

Fig. 30 shows an unstressed 316 ss specimen that had been exposed to 1200 F sodium for 4000 hours, where the oxygen content of the sodium was maintained at a level of 30 ppm or less. The carbon content of the sodium throughout the duration of the run averaged between 40 and 50 ppm. No microstructural changes appear to have resulted from sodium exposure, with the exception that a carbide-like case approximately 3 microns thick is seen at higher magnification. Fig. 31 shows the 316 specimen that had been creep tested in 1200 F at 13,500 psi sodium over 4000 hours. With the exception of a slight degree of sensitization at the surface, the specimen resembled that shown in Fig. 30.

Figs. 32 through 36 show creep-rupture specimens in 1200 F sodium. These specimens were tested in the same sodium environment as those shown in Figs. 30 and 31. The specimens are shown in the order of decreasing stress levels with resulting increase of rupture lives. The gross surface imperfections apparent in the photomicrographs are intergranular surface fractures occurring during

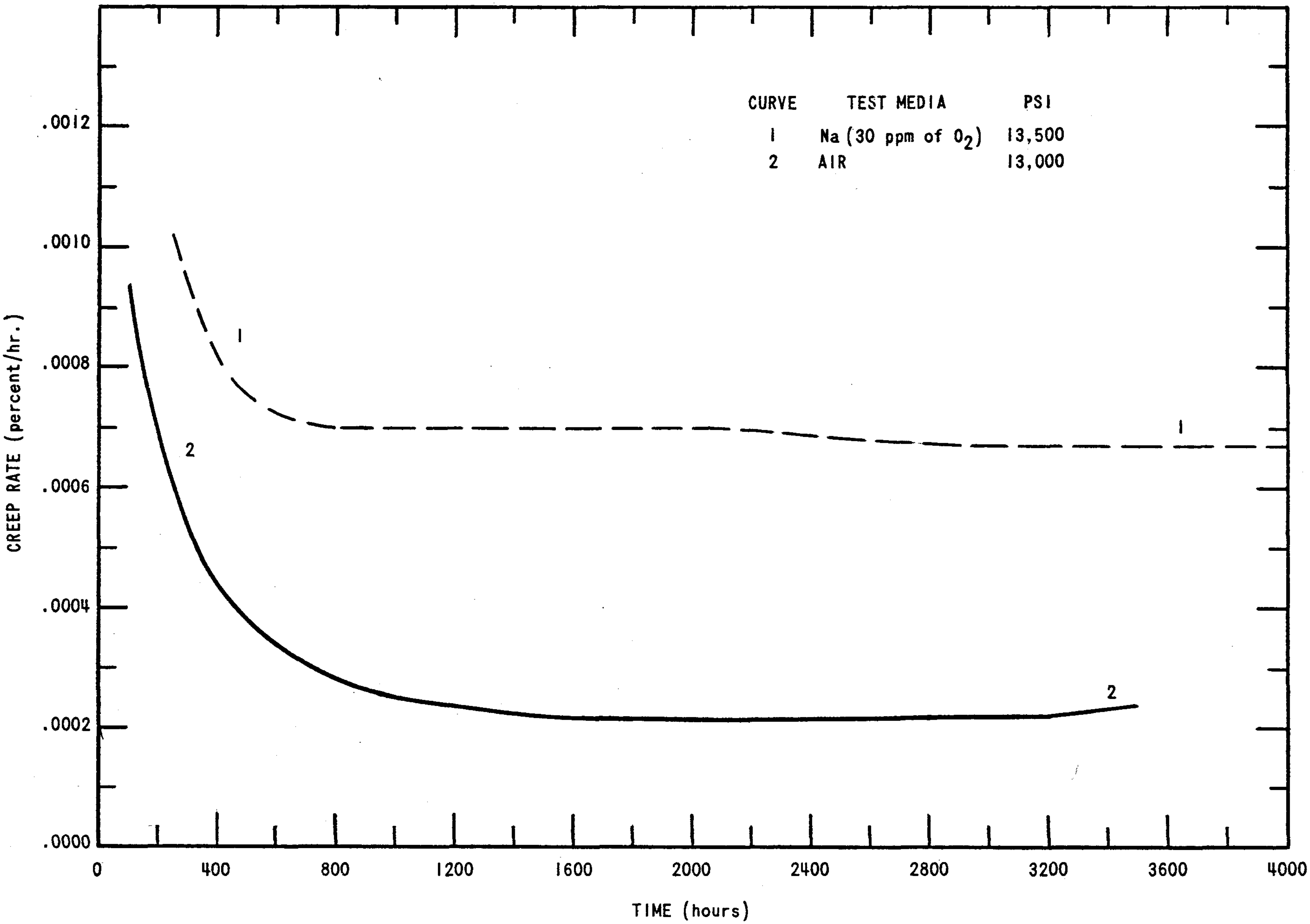


FIG. 26 - CREEP RATES, 316 STAINLESS STEEL SPECIMENS - 1200 F

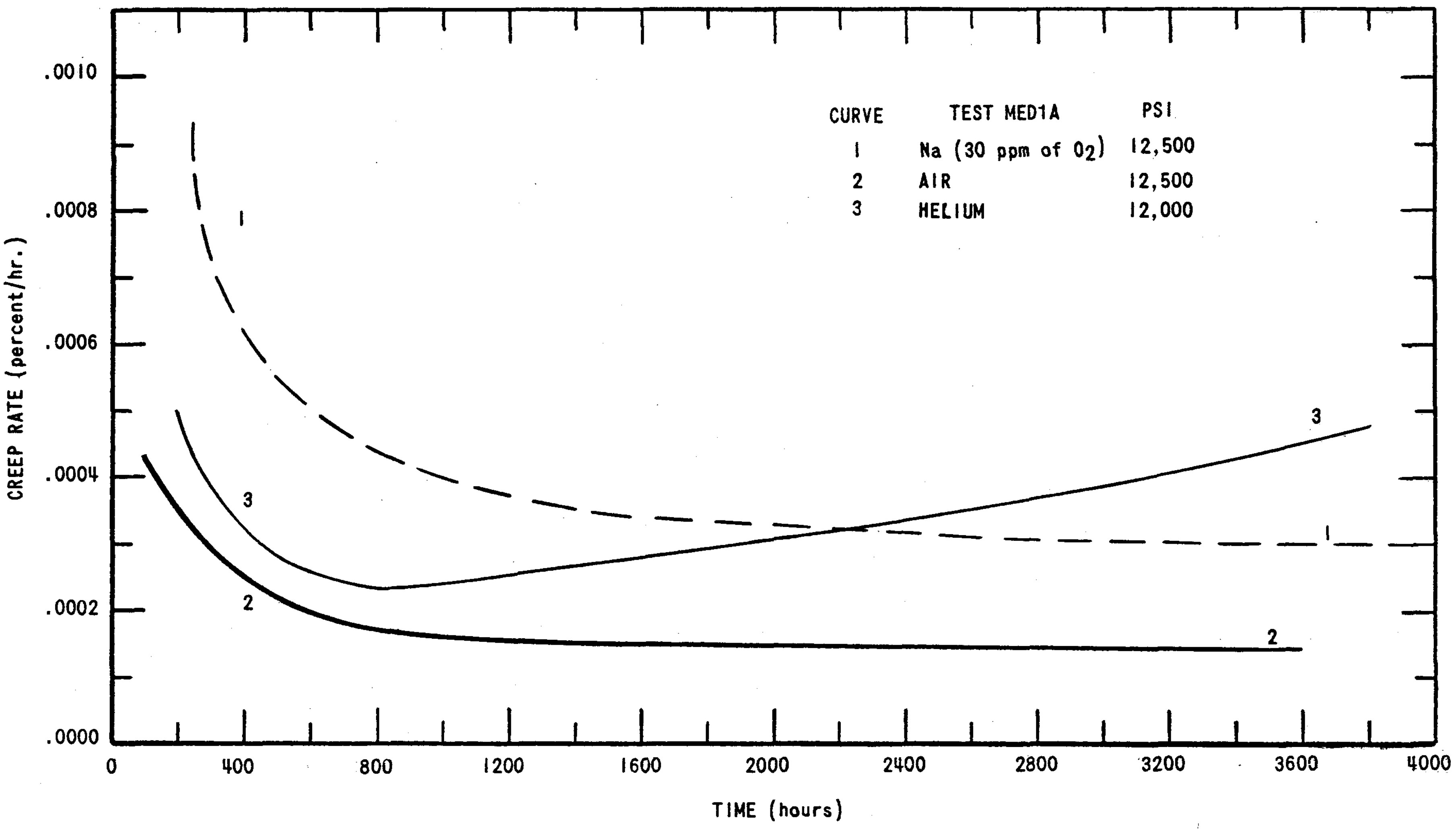


FIG. 27 - CREEP RATES, 316 STAINLESS STEEL SPECIMENS - 1200 F

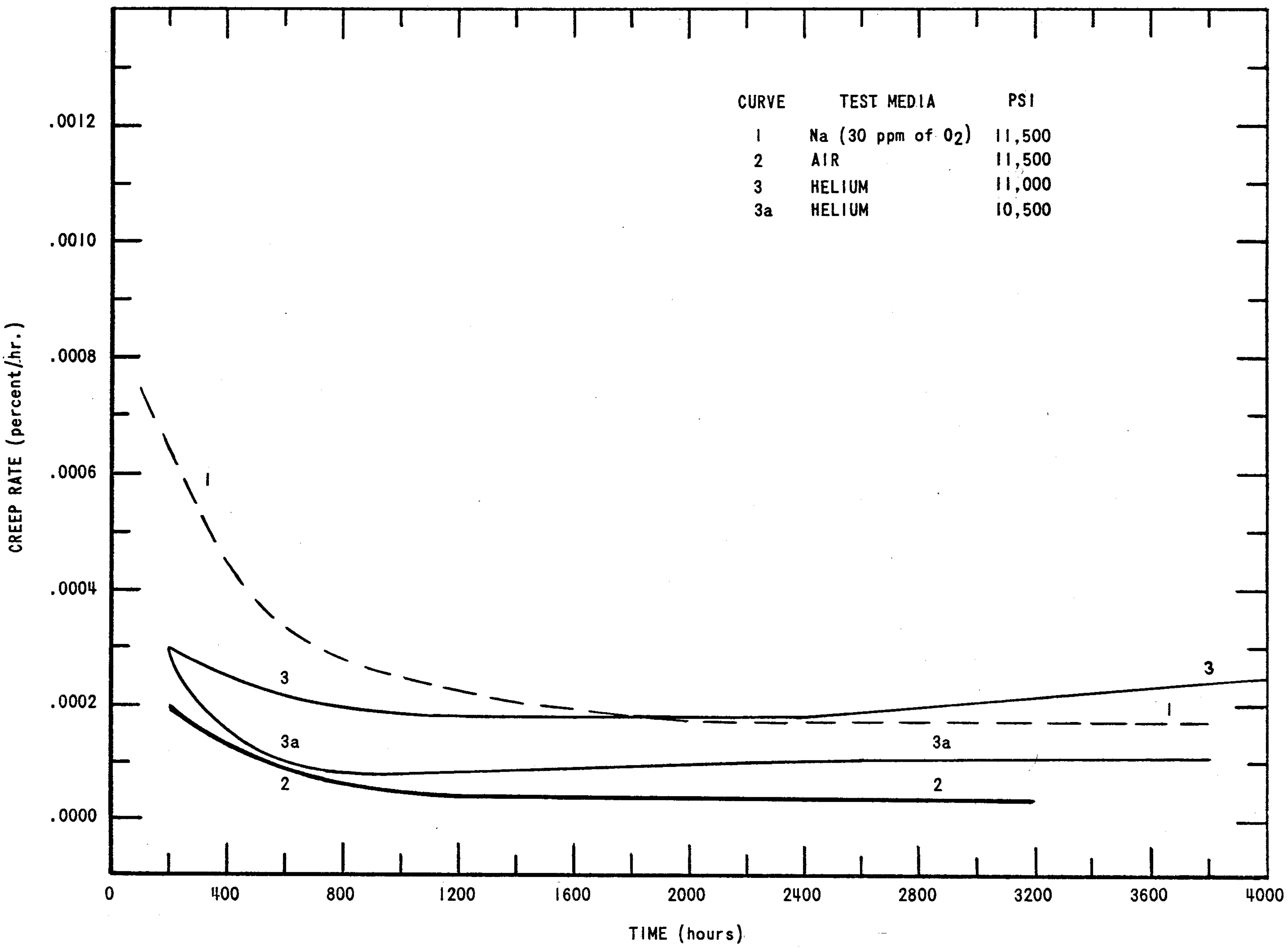


FIG. 28 - CREEP RATES, 316 STAINLESS STEEL SPECIMENS - 1200 F

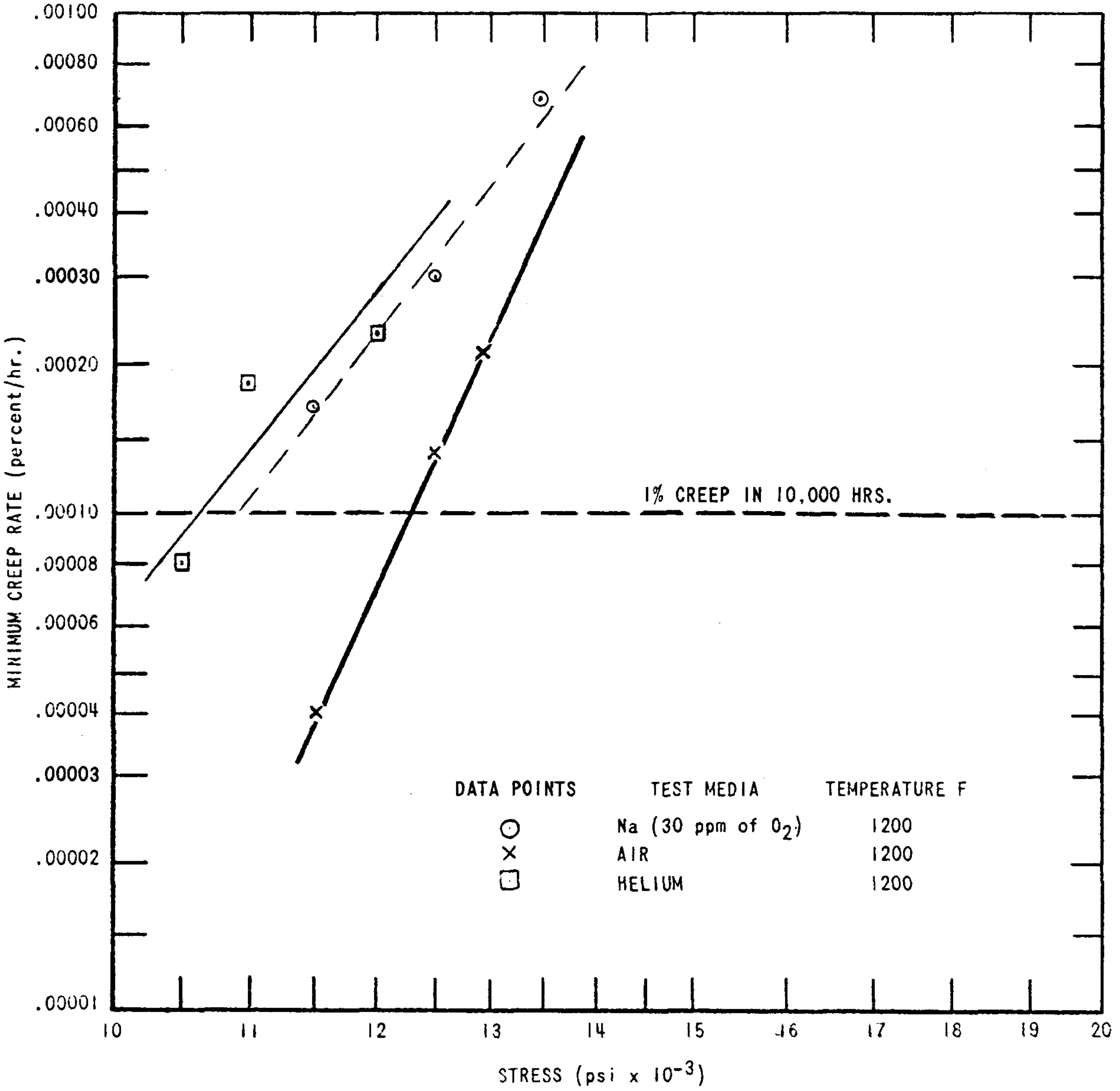


FIG. 29 - MINIMUM CREEP RATE vs STRESS, 316 STAINLESS STEEL SPECIMENS

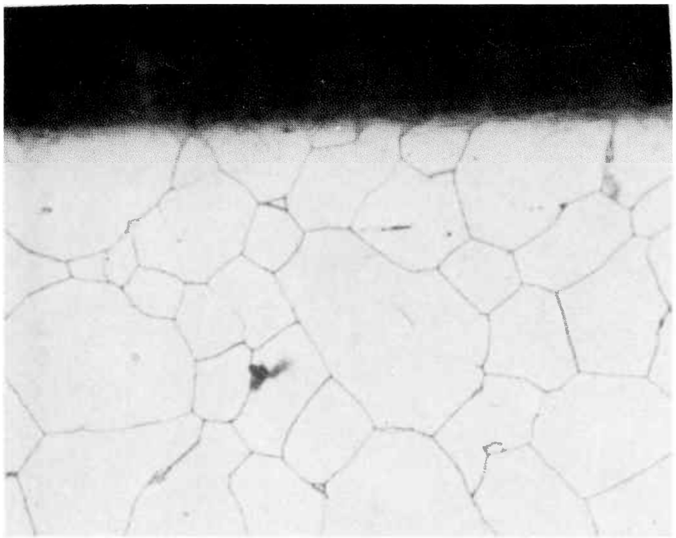


FIG. 30 - STAINLESS (316) EXPOSED TO 1200 F Na
FOR 4000 HOURS
Sample No. 99
$FeCl_3$ etch - 266X

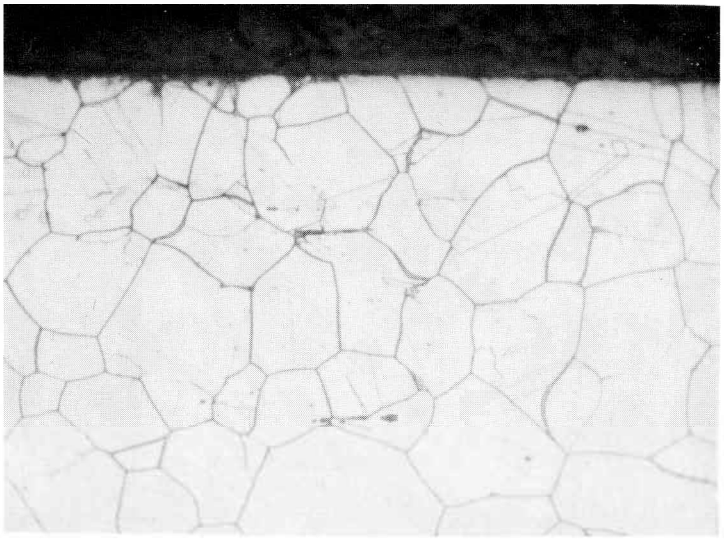
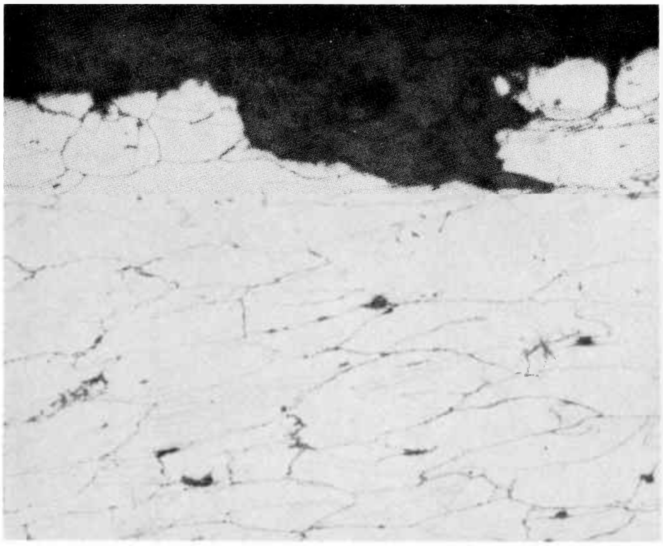
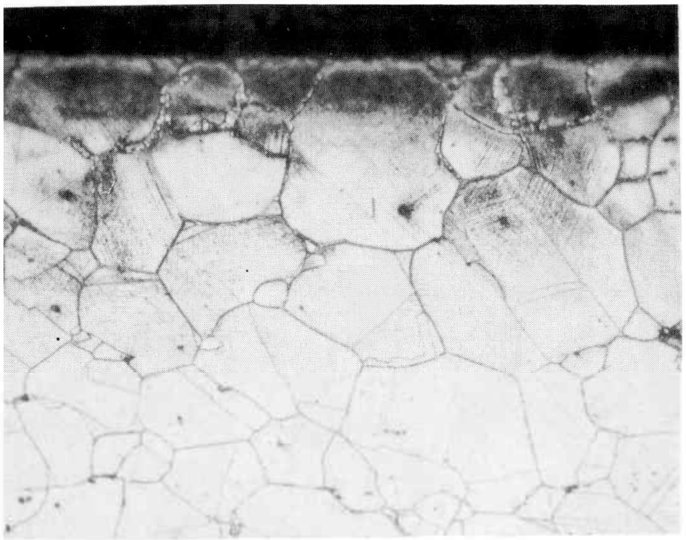
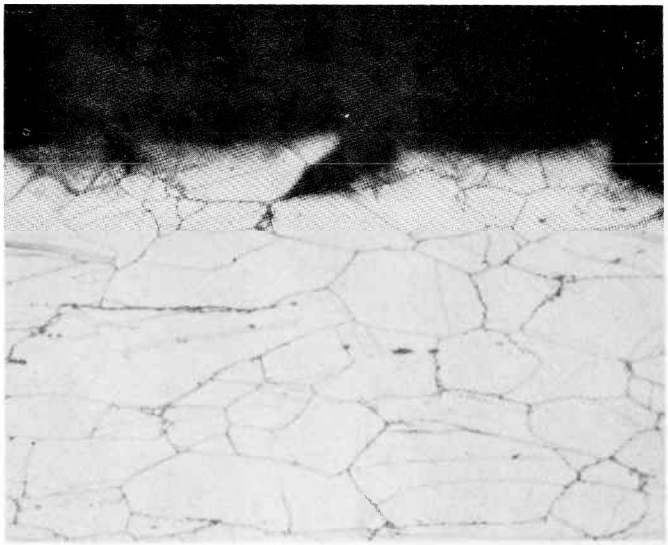
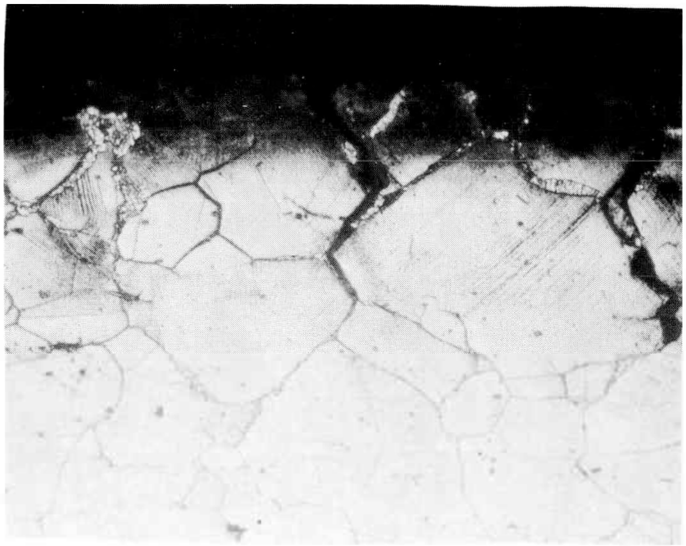


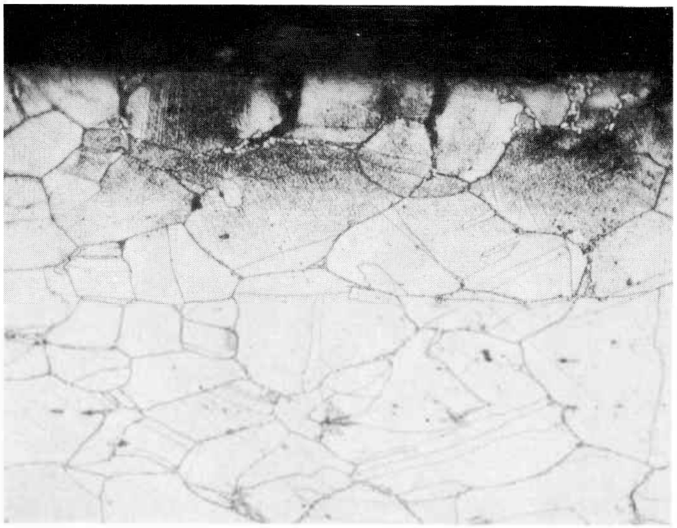
FIG. 31 - STAINLESS (316) CREEP IN 1200 F Na
FOR 4000 HOURS
Specimen 3ALX1
$FeCl_3$ etch - 266X











		Rupture Time, hrs
FIG.32	- Top Left	144.6
FIG.33	- Middle Left	890.7
FIG.34	- Bottom Left	1437.0
FIG.35	- Top Right	2489.5
FIG.36	- Bottom Right	2942.2

STAINLESS STEEL
CREEP-TO-RUPTURE in Na

Ferric Chloride etch - 266X

elongation. A carbide-like case is apparent in Figs. 33 through 36 and appears to be thicker with increasing rupture life. Figs. 34 through 36 suggest carbide precipitation at dislocations, with such precipitation being more marked at higher stress levels. Comparison of this set of photomicrographs with that of Figs. 30 and 31 suggest that these specimens had been carburized to a greater extent than unstressed or lightly stressed (creep) specimens.

Figs. 37 through 40 show specimens that had been exposed to sodium for 4000 hours in the same fashion as the specimen shown in Fig. 30, but were subsequently stressed to rupture in 1200 F sodium. In each of these specimens, a carbide case thicker than that shown in Fig. 30 is again apparent. However, the photomicrographs indicate decreasing carburization with increasing time.

A series of microhardness measurements were made on selected specimens shown in the previous photomicrographs. Fig. 41 shows a microhardness profile of an unstressed specimen and three sodium creep-to-rupture specimens. The microhardness data appears to approximate the case thicknesses observed in the photomicrographs. The nature of the microhardness profile resulting from carburization could not be quantitatively described since hardness increase effects were very shallow. Fig. 42 shows microhardness profiles of those specimens which were first exposed in sodium for 4000 hours and then subsequently stressed to rupture. The hardness measurements again concur with the metallography, which, in the case of the exposed specimens, showed decreasing carburization with increased exposure time.

Each of the specimens shown in the above photomicrographs was analyzed for carbon by conductiometric analysis of the CO_2 produced during oxidation. Sections for carbon analysis were removed both from the gage length and the shoulder, where the stress level is significantly less but is more non-uniform than the gage length.

Table 6 shows the average (of at least two replicas) carbon content of each section, as well as the test conditions and optically measured case thickness. Carbon analysis of the shoulder appears to follow the trends observed in metallography and microhardness determinations. With the exception of one gage length specimen (3DLX1), the carbon analysis of gage length sections resembled carbon diffusion results by the other two means. In the light of conflicting microhardness and metallographic data, this low carbon value on this sample has been discarded.

Table 7 shows the results of carbon analysis of surface sections of an unstressed stainless sample and a long-term stress-to-rupture sample. The carbon profile, although incomplete, tends to confirm the carbon gradient indicated in the photomicrographs. In the case of the unstressed specimen, the bulk of the carbon increase is indicated within less than 2 mils from the surface. If it is assumed that this carbon increase occurs only within the optically measured case depth (6 microns), the carbon content of this case would be approximately 1.3%.

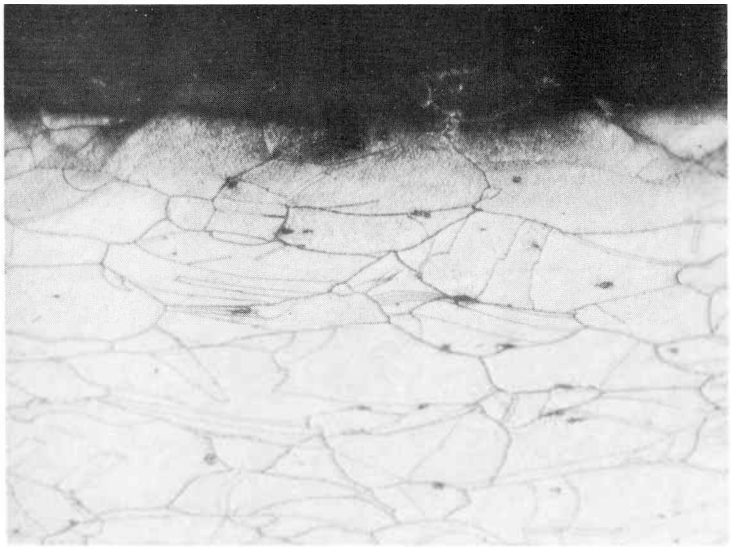


FIG. 37 - PRE-EXPOSED 316 ss
CREEP RUPTURE IN SODIUM
Rupture Time - 236 hrs

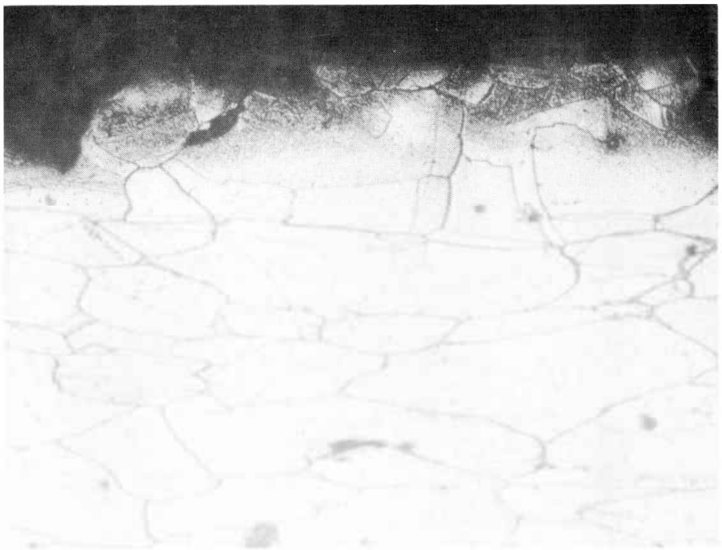


FIG. 38 - PRE-EXPOSED 316 ss
CREEP RUPTURE IN SODIUM
Rupture Time - 632 hrs

FIG. 39 - PRE-EXPOSED 316 ss
CREEP RUPTURE IN SODIUM
Rupture Time - 903 hrs

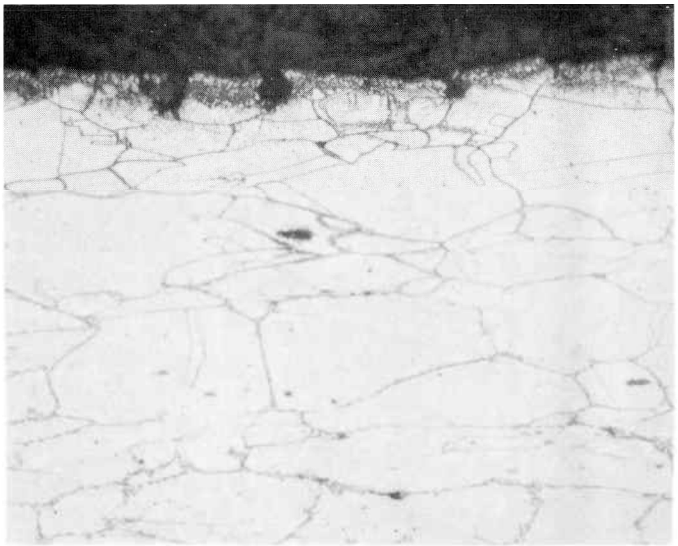


FIG. 40 - PRE-EXPOSED 316 ss
CREEP RUPTURE IN SODIUM
Rupture Time - 1870 hrs

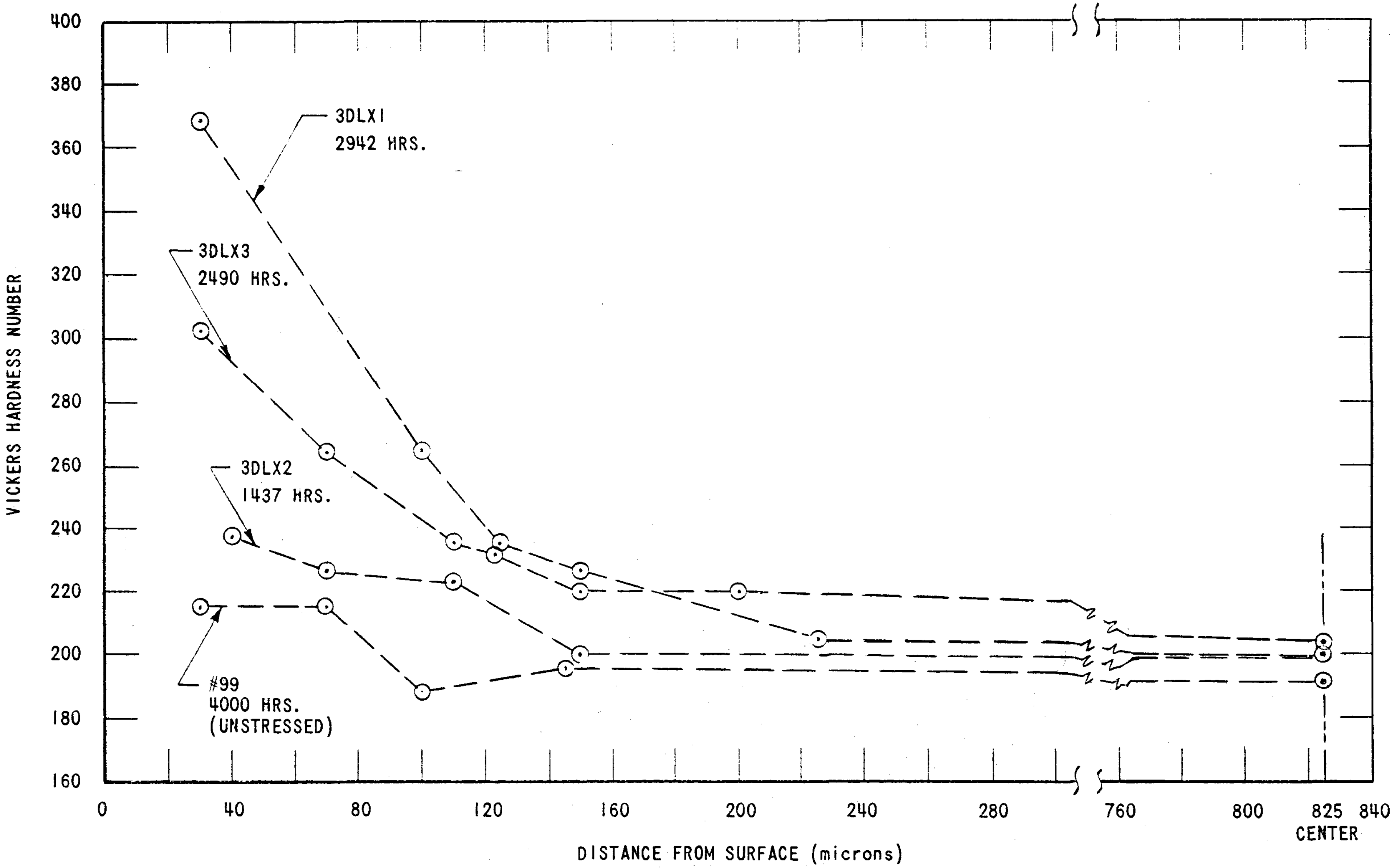


FIG. 41 - HARDNESS PROFILES OF 316 CREEP-RUPTURE SPECIMENS

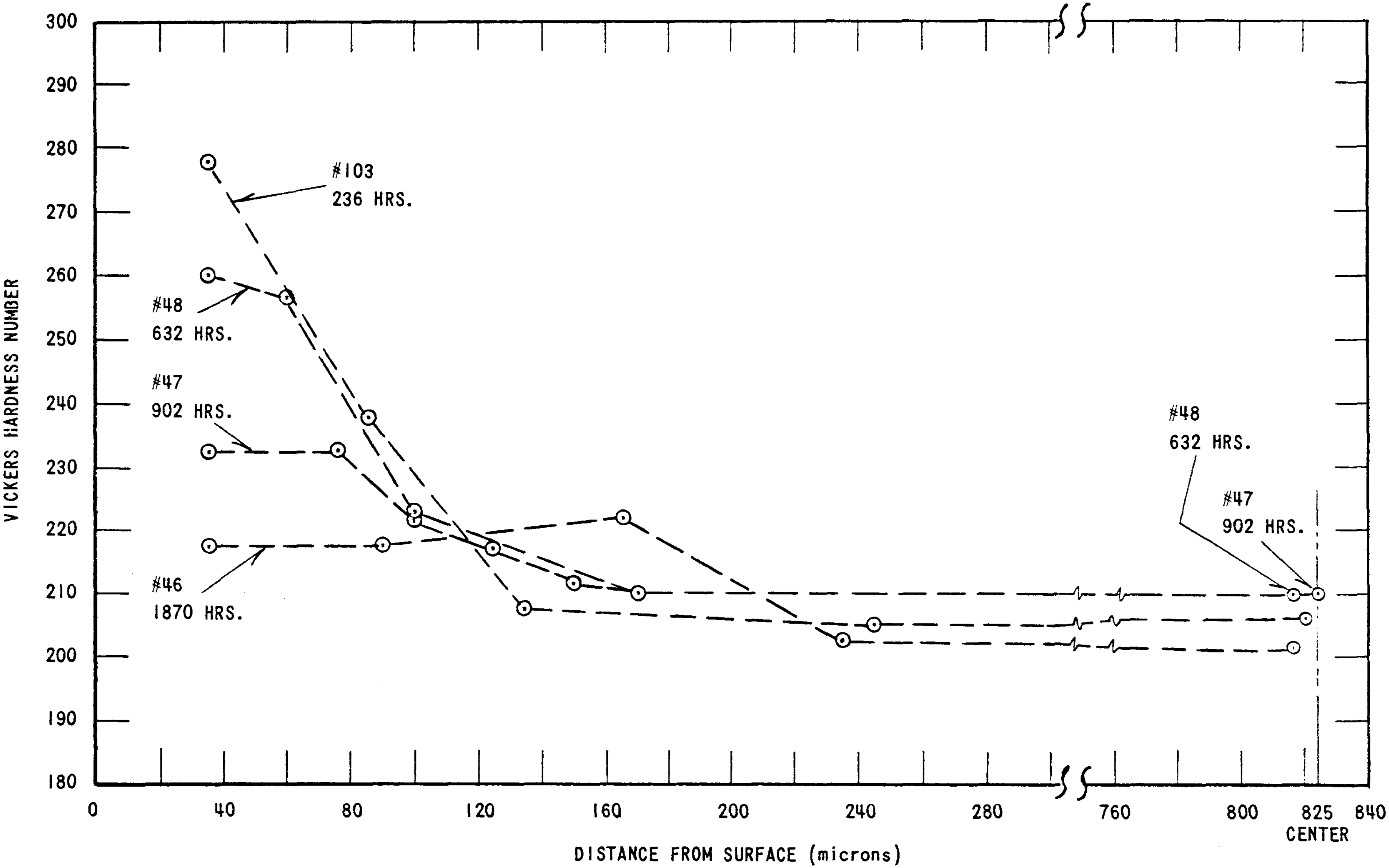


FIG. 42 - HARDNESS PROFILES OF 4000 hr EXPOSED 316 CREEP_RUPTURE SPECIMENS

TABLE 6 - CARBON CONTENT OF 316 SS IN 1200 F SODIUM

Sample No.	Type of Test	Stress Applied (K Psi)	Exposure or Rupture Time (Hrs)	Elongation (%)	Case Thickness (Microns)	Carbon Content of Shoulder (ppm)	Carbon Content of Gage Length (ppm)
99	Unstressed	0	4000	0	6	600	600
3ALX1	Creep	13.5	4000	2.5	6	-	600
3ALX2	Creep	12.5	4000	1.7	-	-	554
3ALX3	Creep	11.5	4000	0.4	-	-	520
3CLX1	Creep-Rupture	27.5	145	74	3	483	534
3CLX2	Creep-Rupture	24.0	445	62	-	-	518
3CLX3	Creep-Rupture	21.5	891	70	30	539	572
3DLX2	Creep-Rupture	20.0	1437	53	80	598	594
3DLX3	Creep-Rupture	18.5	2490	33	100	875	970
3DLX1	Creep-Rupture	17.75	2942	27	100	1180	500-900
3ELL4	Creep-Rupture*	27.5	236	73	40	903	934
3ELL3	Creep-Rupture*	24.0	632	73	40	922	858
3ELL2	Creep-Rupture*	21.5	902	65	30	712	699
3ELL1	Creep-Rupture*	20.0	1870	71	20	674	731

* Exposed 4000 hours in 1200 F sodium

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

Examination of carbon analysis, metallographic and microhardness data indicates a striking difference in carburization rates of unstressed or lightly stressed specimens with those specimens undergoing significant deformation. The extent to which any of the specimens have been carburized has been sufficiently low so as not to affect the mechanical properties of the test specimen. It is highly unlikely that stainless components of an operating loop would be exposed to such high deformation for extended periods. However, observations of the parameter of stress on carburization rate at low carbon levels may be useful in interpreting carbon diffusion in 316 ss when exposed to high carbon environments.

The nature of the stress parameter on carburization rates is not adequately defined on the basis of the limited number of sodium stress-rupture tests that have been performed. Analysis of the data is made more complex because of variations in elongation rate during exposure. One possible interpretation of the data is that the increase in carburization is proportional to some function of strain and time at the unit temperature. Those samples that showed the greatest carbon gains were stressed at moderate levels. If it can be assumed that the carbon level of pre-exposed samples was 600 ppm after 4000 hours sodium exposure, then the pre-exposed samples show the greatest carburization rate in the stressed condition.

It is concluded that a mechanism exists for increase of carburization by application of a stress. One suggested mechanism is that in which the formation of dislocations on the surface act as channels down through which carbon diffuses more readily than in unstressed material. Increase in the diffusion rate of carbon would tend to reduce the carbon activity at the liquid sodium-stainless interface and the affinity of the stainless steel surface for carbon is increased. In the case of the pre-exposed and subsequently stressed samples, carbon diffusion in the stainless is minimal and the surface attains a high carbon activity. Application of high stresses with resulting high strains accelerates carbon diffusion and the carbon activity at the surface is rapidly diminished.

A second possible mechanism is one where the strain can alter (increase) the thermodynamic potential of the stainless for carbon. Yang, Horne and Pound[4] describe a somewhat related effect where elastic strain has altered the electrode potential of metals.

A third, and perhaps the most valid proposed mechanism is one where the reaction rate of the carbon dissolved in the sodium in a stainless steel system is greatly increased by strain of the lattice at the liquid-solid interface.

---

[4] Physical Metallurgy of Stress-Corrosion Fracture, AIME Meeting, New York, 1959.

Unfortunately, an insufficient number of unstressed samples had been exposed to the high purity sodium environment with varying durations of exposure that can permit a quantitative comparison of the stressed to unstressed conditions. Furthermore, it is thought that an insufficient number of stressed samples had been generated to quantitatively define this parameter. A complete test series is presently underway that could shed light on this parameter. This test series involves determination of the mechanical properties of 316 ss when exposed to 1200 F sodium saturated with carbon.

Microprobe analysis of two stainless steel specimens that had been exposed to high purity sodium was performed at the Mellon Institute, Pittsburgh, Pa. Sample No. 3ALX1 was a 316 specimen that had been creep tested at a stress of 13,500 psi for 4000 hours in 1200 F high purity sodium. Sample No. 99 was exposed to 1200 F sodium for 4000 hours in an unstressed condition.

Fig. 43 is an iron image at the surface of sample No. 99. Each of the grid squares shown in Fig. 43 is 1.77 mils on edge, and the spacing between center grid markings is 0.35 mils. The iron content within the field is quite homogeneous, although the lower left corner appears to show a lower iron content. Examination of all microprobe photographs shows the same low density effect in the same portion of the field, indicating that this effect is a characteristic of the instrument.

Figs. 44 and 45 show respectively the chromium and nickel images of the same sample, which also indicate no significant inhomogeneities with respect to these elements. Examination of Ni, Cr and Fe images (not shown) of as-received 316 did not show any inhomogeneities and appeared to be identical to images of the creep specimen (not shown) as well as those of sample No. 99. Fig. 46 shows a molybdenum image at the surface of the creep specimen. No inhomogeneities appear to exist for molybdenum.

Quantitative line profiles of Fe, Cr and Ni composition from the matrix to the exposed surface were obtained for both exposed samples. Fig. 47 is a plot of metallic compositions versus distance from the surface. The iron, chromium and nickel values were found to be uniform on both samples, with the exception of a surface-affected layer approximately 8 microns (0.0003 in.) thick. The matrix chromium content was found to be 23% as compared to a chemical Cr analysis of 18.1%, while microprobe Ni analysis was approximately 10.5% as compared to a chemical analysis of the heat of 12.7%. The differences between microprobe results versus those obtained by chemical analysis are thought to be the result of interferences of foreign elements in microprobe analysis.

The microprobe line profiles show a depletion of chromium, which is not apparent on the qualitative photographs shown in Fig. 44. A slight enrichment of iron, probably as a result of chromium removal is also apparent in Fig. 47.

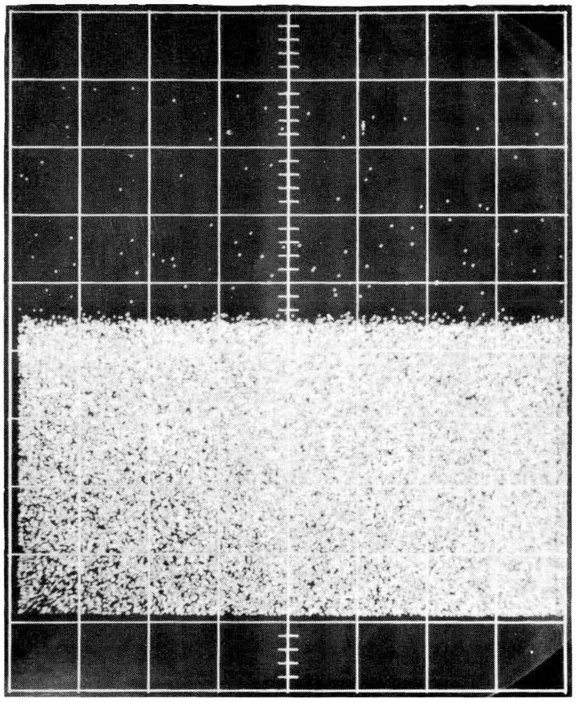


FIG. 43 - IRON IMAGE
Type 316 ss - Specimen No. 99

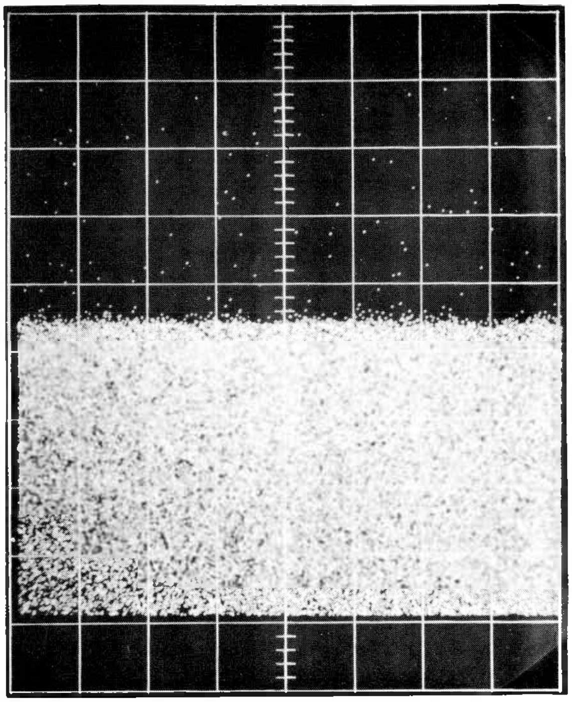


FIG. 44 - CHROMIUM IMAGE
Type 316 ss - Specimen No. 99

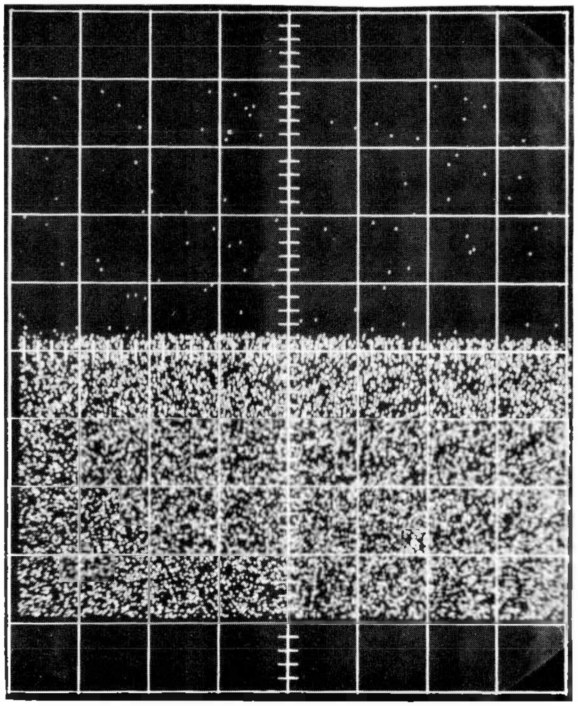


FIG. 45 - NICKEL IMAGE
Type 316 ss - Specimen No. 99

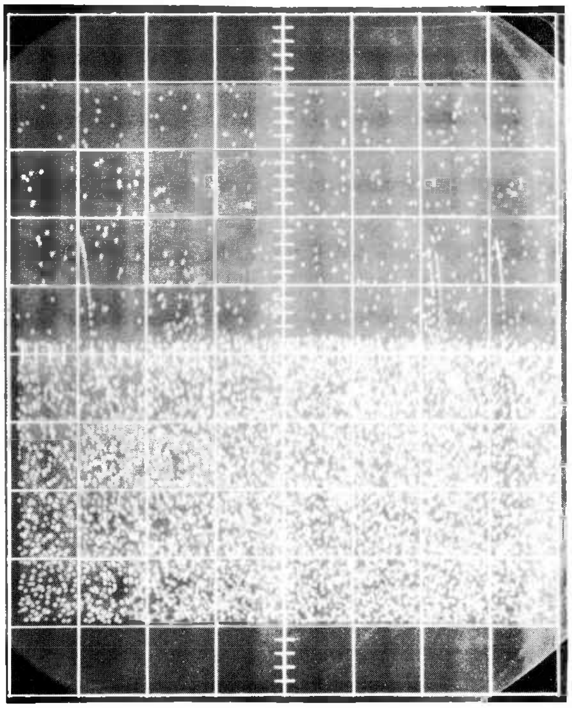


FIG. 46 - MOLYBDENUM IMAGE
Type 316 ss - SPECIMEN 3ALX2

Note: Field is 360 microns (approximately 0.014 in.) wide.

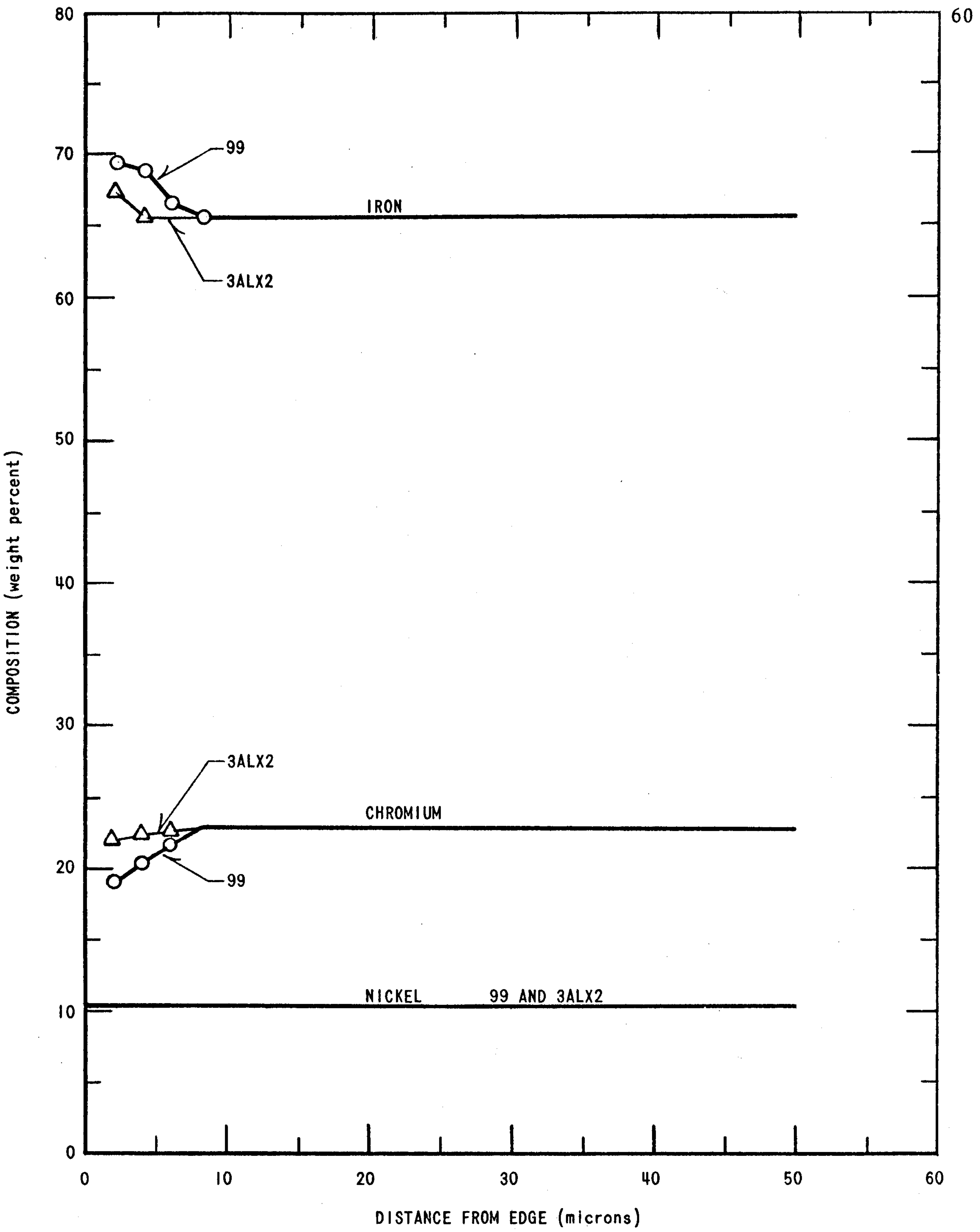


FIG. 47 - MICROPROBE LINE PROFILE FOR TYPE 316 STAINLESS STEEL SPECIMENS 99 and 3ALX2

It is concluded that metallic mass transfer is not significant in 4000 hours under the relatively isothermal conditions and low oxygen content existing in the systems. Chromium removal with subsequent enrichment of the remainder of the constituents of the surface has occurred, but the affected zone is significantly less than 1 mil. The application of a load to the Type 316 ss specimens did not markedly affect mass transfer. The differences in removal of chromium from the stressed versus unstressed sample is thought to be within experimental error in determination of the exact location of the surface.

8. TENSILE TESTS

All tensile tests were run at the University of Michigan in air on standard tensile test machines and on modified standard machines for the helium tests.

Standard operating and test procedures were used for the tests in air. The helium test went through a period of evolution in an effort to prevent oxidation of the test specimen even for the short time required to run these tests. The final procedure used was to install the specimen into the test chamber and begin outgassing. A vacuum of less than 10 microns was maintained with a maximum leak rate of 10 μ/minute during which time the temperature was increased to test conditions (1200 F). This vacuum was then broken with helium after first passing it through a NaK bubbler to remove any traces of oxygen or moisture.

8.1 TENSILE TEST RESULTS

Triplicate specimens were tested in air and helium at 1200 F to serve as a basis for comparison. Tests were also run in helium at room temperature. Twelve specimens were exposed to 1200 F, low oxide (30 ppm) sodium for 4000 hours. Three of these were then tested without washing in helium at 1200 F. Three were run at 1200 F in helium after first being washed with alcohol and water. The remaining six were washed and then tested in helium at room temperature.

The results of the tensile tests are listed in Table 8. Comparison of the averaged tensile strengths at 1200 F in air and helium show variations of 5% or less regardless of whether the specimen was first exposed in sodium at 1200 F or whether the exposed specimens were washed or unwashed prior to testing. Any difference in tensile strengths for the various test conditions was considered indiscernible.

Similarly, the averaged yield strengths for all the environments and test conditions at 1200 F showed variations of

TABLE 8 - TENSILE TEST RESULTS

Specimen	Condition	Tensile Stress (Psi)	Averaged Tensile (Psi)	0.2% Offset Yield (Psi)	Averaged Yield (Psi)	Yield Standard Deviation (Psi)	Elongation %	Reduction Of Area %
3BAX4	Air-1200F	49,400		26,100			43	47
3BAX3	Air-1200F	50,250	50,280	25,650	26,020	330	41	48
3BAX1	Air-1200F	51,200		26,300			47	44
3BHX1	He-1200F	47,200		26,600			46	58
3BHX2	He-1200F	47,500	47,770	25,900	25,930	650	52	54
3BHX3	He-1200F	48,600		25,300			44	53
3BHL1C	He-1200F*	48,850		24,500			39	46
3BHL2C	He-1200F*	46,900	48,580	24,800	25,170	910	52	52
3BHL3C	He-1200F*	50,000		26,200			41	46
3BHL1U	He-1200F**	49,100		26,750			41	56
3BHL2U	He-1200F**	46,400	48,470	25,200	25,870	800	46	55
3BHL3U	He-1200F**	49,900		25,650			46	51
3BHX4	He-RT*	93,200		44,600			47	46
3BHX5	He-RT*	92,000		43,750			48	50
3BHX6	He-RT*	92,500		42,000			46	40
3BHX7	He-RT*	91,400	92,850	43,700	43,930	1190	49	52
3BHX8	He-RT*	92,500		43,900			49	52
3BHX9	He-RT*	95,500		45,600			46	40
3BHX10	He-RT	89,500		49,700	49,550	210	65	59
3BHX11	He-RT	88,500	89,000	49,400			66	59

* Pre-exposed for 4000 hours in 1200 F sodium. Specimen washed after exposure.

** Pre-exposed for 4000 hours in 1200 F sodium. Specimen unwashed after exposure.

less than 6%. The agreement among the triplicate specimens was good. For example, the relative deviation between specimens based on the yield data ranged from 1.3 to 3.6%. No effect was therefore noted for the yield strengths under these conditions.

The comparison of the tensile and yield strengths in helium at room temperature between exposed and original material showed a 4% reduction in the ultimate tensile and a corresponding increase of 11% in the yield strength.

Fig. 48 shows the surface of a stainless specimen that had been exposed to sodium for 4000 hours and was subsequently tensile tested in helium at 1200 F. A carbide case is barely evident on this sample and carbon analysis confirmed that little carburization has occurred.

In summary, the mechanical property data indicates little effect of environment on short-term 1200 F tensile properties of 316, even though such samples may have been exposed to sodium for 4000 hours. Metallographic examination of selected samples shows no surface coatings or reaction zones.

9. ANALYTICAL CONTROL

9.1 GENERAL CONTAMINANTS

Sufficient sodium has been purchased to supply the needs throughout this program as it is scheduled today. The sodium was purchased in seventeen cast drums from the DuPont Company and is designated as Lot No. 52, which is the same lot that supplied the sodium for the mass transfer program at General Electric (San Jose). The sodium is designated as "reactor grade" and the certificate of quality lists the calcium and chloride contents as being less than 5 ppm and 6 ppm respectively.

The initial analysis of this sodium, as well as the analyses of sodium samples removed from the instream expansion tank at 1200 F on a weekly basis throughout TEST 1, are tabulated in Table 9. All of the results shown were obtained by emission spectrographic analyses.

These samples were removed initially in nickel buckets but, as seen, this influenced the nickel concentration of the sodium samples. Titanium buckets were substituted to avoid nickel contamination and the subsequent variation in titanium content is not of interest since titanium is not a natural constituent of 316 ss.

The use of glassware in the digestion stage of the analysis was believed to be the cause of some of the high silicon values. The use of Teflon beakers during the digestion stage helped to reduce this contamination.

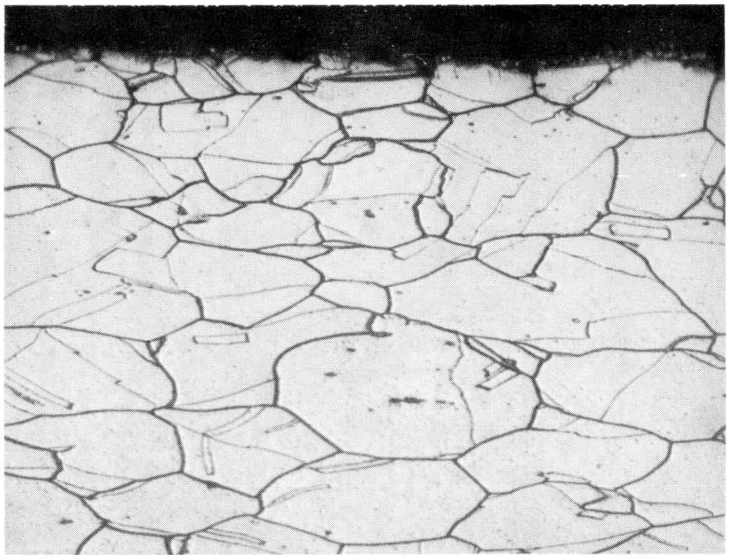


FIG. 48 - 316 ss TENSILE IN 1200 F HELIUM

Sample - BHL3C
Exposed - 4000 hrs in 1200 F Sodium
Tensile - 5000 psi
% Elongation - 41
R/A % - 46

Ferric Chloride Etch 266X

Table 9 - Chemical Analysis of Sodium From TEST 1 (ss Test Specimens) - in ppm

Date	Fe	B	Co	Mn	Al	Mg	Sn	Cu	Pb	Cr	Si	Ti	Ni	Mo	V	Ca	Ag	Be	Ba	Sr	Zr	Li
3-15-62	3	<5	<5	<1	<1	<2	<5	<2	<5	<1	<10	<5	<1	<5	<1	2	<1	<1	<2	<1	<10	
4-25-62	8	<5	<5	1	<1	5	<5	3	<5	<1	35	<5	300	<5	<1		<1	<1			<10	
5-1-62	7	<5	<5	<1	5	4	<5	1	<5	<1	30	<5	40	<5	<1		<1	<1			<10	
5-8-62	4	<5	<5	1	<1	<2	<5	1	<5	<1	30	<5	100	<5	<1		<1	<1			<10	
5-15-62	6	<5	<5	1	2	3	<5	5	<5	<1	30	<5	60	<5	<1		<1	<1			<10	
5-29-62	1	<5	<5	<1	4	5	<5	2	<5	<1	25	<5	<1	<5	<1			<1			<10	
6-5-62	2	<5	<5	3	3	2	<5	50	<5	<1	20	<5	.1%	<5	<1			<1			<10	
6-12-62	1	<5	<5	1	2	4	<5	50	<5	<1	25	<5	8	<5	<1			1			<10	
6-14-62	<1	<5	<5	<1	1	<1	<5	9	<5	<1	10	<5	<1	<5	<1	<1	<1	<1	<3	<1	<10	
6-26-62	<1	<5	<5	<1	<1	<1	<5	3	<5	<1	<10	<5	<1	<5	<1	<1	<1	<1	<3	<1	<10	
7-3-62	1	<5	<5	<1	1	<1	<5	10	<5	<1	12	<5	<1	<5	<1	<1	<1	<1	<3	<1	<10	
7-10-62	<1	<5	<5	<1	1	<1	<5	<1	<5	<1	30	<5	2	<5	<1	<1	<1	<1	<3	<1	<10	
7-17-62	25	<5	<5	50	<1	2	<5	5	<5	<1	10	<5	<.1%	<5	<1		<1	<1			<10	
7-25-62	1	<5	<5	3	<1	1	<5	12	<5	<1	10	<5	150	<5	<1		<1	<1			<10	
7-31-62	<1	<5	<5	<1	6	5	<5	<1	<5	<1	25	5	<1	<5	<1		<1	<1			<10	
8-7-62	2	<5	<5	1	<1	1	<5	<1	<5	<1	10	<5	<1	<5	<1		<1	<1			<10	
8-14-62	1	<5	<5	<1	5	30	<5	<1	<5	<1	75	7	110	<5	<1		<1	<1			<10	
8-21-62	1	<5	<5	<1	4	3	<5	<1	<5	<1	15	80	<1	<5	<1		<1	<1			<10	
8-28-62	1	8	<5	<1	4	4	<5	1	<5	<1	15	50	<1	<5	<1		<1	<1			<10	
9-4-62	2	<5	<5	1	2	4	<5	<1	<5	1	15	20	<1	<5	<1		<1	<1			<10	
9-11-62	2	5	<5	<1	2	2	<5	<1	<5	<1	8	110	<1	<5	<1		<1	<1			<10	
9-17-62	2	10	<5	<1	1	2	<5	<1	<5	1	15	10	1	<5	<1		<1	<1			<10	
9-25-62	1	<5	<5	<1	2	3	<5	<1	<5	<1	10	120	1	<5	<1		<1	<1			<10	
10-1-62	2	12	<5	<1	3	15	<5	<1	<5	<1	100	110	<1	<5	<1		<1	<1			<10	
10-9-62	2	5	<5	<1	2	10	<5	<1	<5	<1	7	10	<1	<5	<1		<1	<1			<10	
10-16-62	1	<5	<5	<1	2	45	<5	<1	<5	<1	25	15	<1	<5	<1	30	<1	<1			<10	<1
10-23-62	<1	<5	<5	<1	<1	40	<5	<1	<5	<1	15	<5	<1	<5	<1	25	<1	<1			<10	<1
10-30-62	<1	<5	<5	<1	<1	2	<5	<1	<5	<1	10	20	<1	<5	<1	<1	<1	<1			<10	<1
11-6-62	<1	<5	<5	<1	<1	1	<5	1	<5	<1	<10	<5	<1	<5	<1	<1	<1	<1			<10	<1
11-13-62	<1	<5	<5	<1	<1	2	<5	1	<5	<1	10	20	<1	<5	<1	<1	<1	<1			<10	<1
11-20-62	<1	<5	<5	<1	<1	<1	<5	<1	<5	<1	<10	<5	<1	<5	<1	2	<1	<1			<10	<1
11-27-62	<1	<5	<5	<1	<1	<1	<5	<1	<5	<1	<10	<5	<1	<5	<1	4	<1	<1			<10	<1
12-4-62	<1	<5	<5	<1	<1	<1	<5	2	<5	<1	<10	<5	<1	<5	<1	<1	<1	<1			<10	<1
12-11-62	<1	<5	<5	<1	<1	<1	<5	3	<5	<1	<10	<5	<1	<5	<1	1	<1	<1			<10	<1
12-19-62	<1	<5	<5	<1	<1	1	<5	<1	<5	<1	<10	<5	<1	<5	<1	2	<1	<1			<10	<1

The results of 35 samples taken from the 1200 F isothermal sodium system over a nine-month period of continuous operation showed little, if any, of the structural materials to be in solution. This would be expected in an isothermal system where mass transfer rates are at a minimum.

9.2 OXYGEN

The oxygen level was controlled as one of the test parameters at an equivalent saturation temperature of 300 F. Using the oxygen saturation solubility curve as shown in Fig. 49, this saturation temperature corresponds to an oxygen concentration of 30 ppm. Periodical (a maximum of every three days) plugging indicator tests determined the oxygen level and, if this level exceeded 300 F, cold trapping was initiated until the saturation temperature was 300 F or lower. In addition to the regular schedule, the cold trap was operated each time a test unit was put back on stream following replacement of the specimens. The oxygen level was within specifications usually within four hours. No difficulties were experienced in maintaining this saturation level at 300 F or lower for two or three week periods without cold trapping, if test units were not being put on stream during this period. The operational history graph (Fig. 50) shows the frequency of plugging runs and cold trap operations.

9.3 CARBON

Considerable attention was given to the carbon concentration since the future program calls for a series of tests in 1200 F sodium saturated with carbon. Low concentrations of carbon are difficult to determine and considerable time has been spent establishing a reliable technique. Samples are dripped from the flowing sodium stream and transferred to a reaction flask where the sodium is reacted with water at 0°C. After reaction, the total sample is acidified to remove carbonate and evaporated to dryness. When carbonate levels are of interest, the off-gas is collected during the acidification step. The dried sample is heated to 1200 F for 40 minutes under an atmosphere of purified oxygen. This procedure oxidizes carbon to CO_2 which is collected in a U-tube cold trap. The CO_2 content is measured with a mass spectrometer and the values converted to carbon concentration in the sodium.

Van Slyke oxidizing solution was used originally as the oxidizing medium. However, difficulties in reducing and maintaining the carbon background at a low level made this technique unattractive. Oxygen purification has proven less demanding than Van Slyke purifications. Samples of Na_2SO_4 spiked with known amounts of carbon have shown that direct oxidation by heating in an oxygen atmosphere quantitively oxidizes carbon to CO_2.

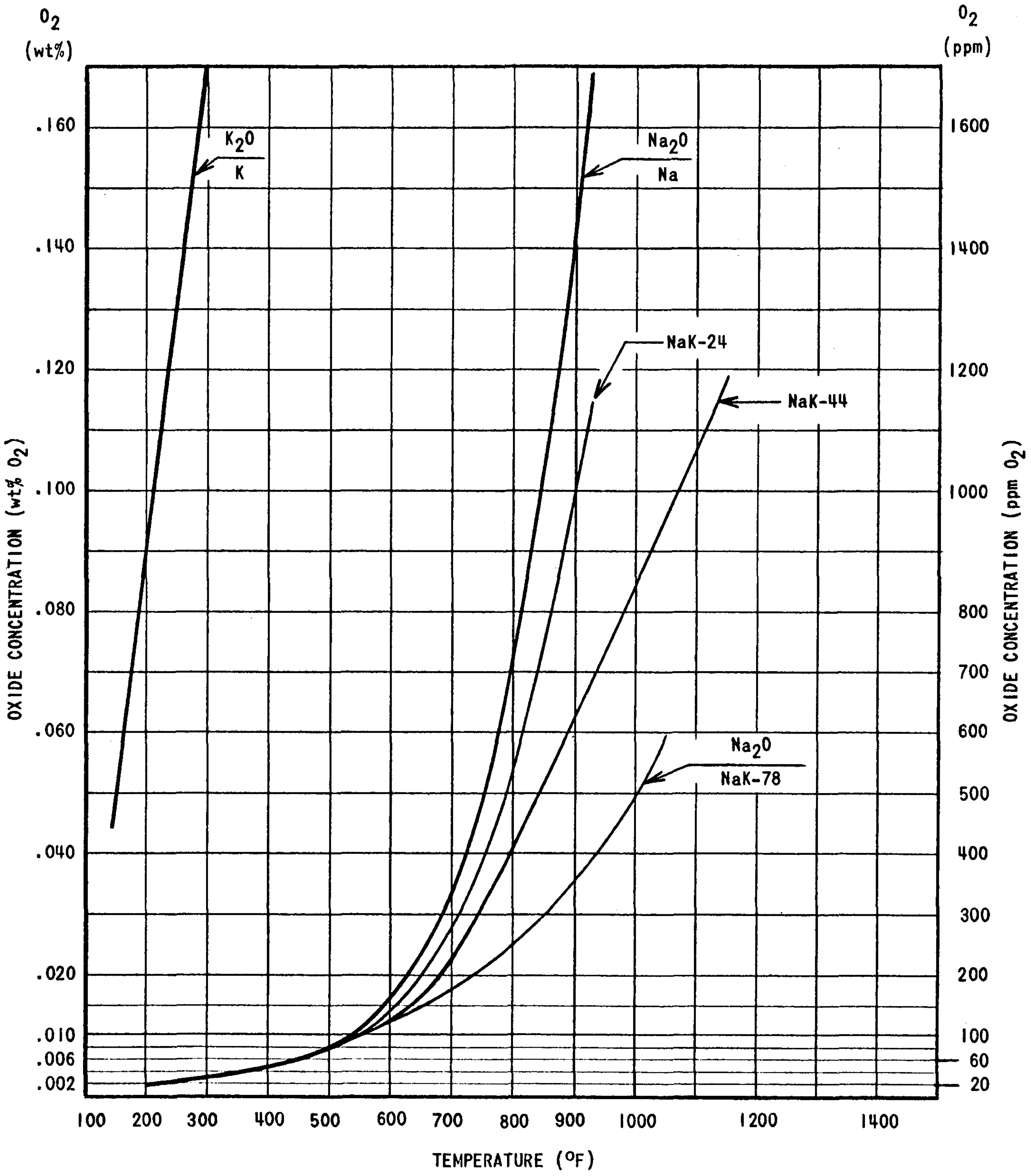


FIG. 49 - OXIDE SATURATION SOLUBILITIES

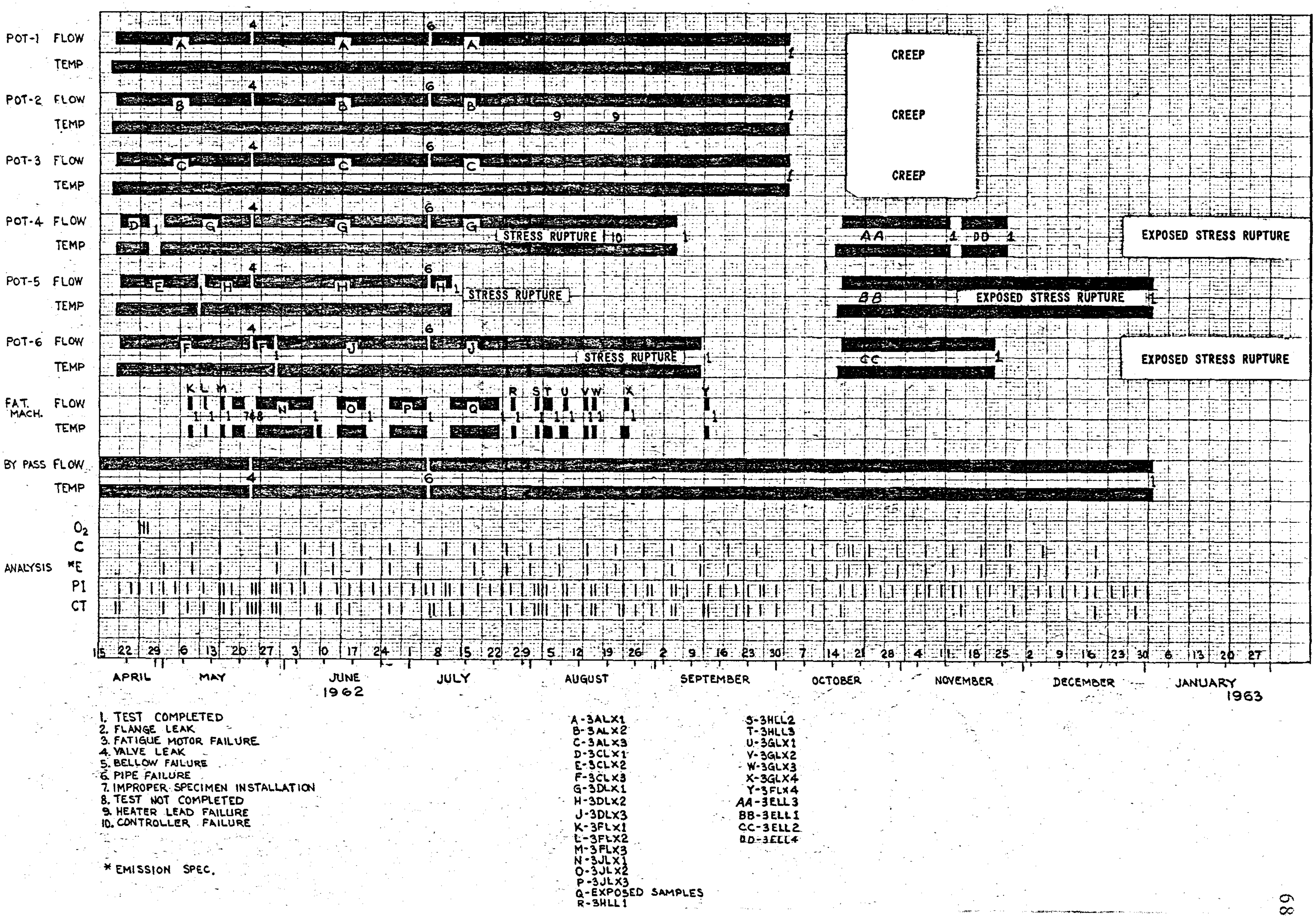


FIG. 50 - OPERATIONAL HISTORY OF LOOP 1 DURING TEST 1 IN SODIUM (30 ppm O_2)

We believe variations in carbon content of sodium are a result of sampling rather than analyses.

The tabulated carbon results are shown in Table 10 and also in Fig. 51 where the results are shown graphically in comparison with Gratton's data.[5] The results show considerable scatter but in general compare favorably with the scatter of Gratton's carbon solubility data. As shown in Fig. 51, the sodium system ran for the majority of TEST 1 below the solubility levels given in Gratton's data.

10. SODIUM LOOP OPERATION

The operational history of Loop 1 during TEST 1 is shown as a chart in Fig. 49. This chart indicates the periods that test temperatures and flows were maintained through each of the test units and manifold bypass. The times at which sodium samples for analyses were taken, and operation of the OCI system are also indicated in this figure. System failures are indicated by notes and only on two occasions was it necessary to interrupt operations. In both cases, the test units were isolated and temperatures maintained while repairs were made.

The first interruption was due to failure in a 1/2 in. bellows sealed valve. The failure was in the bellows and proved to be due to an improper bellows design. The second interruption was a failure of a 1/2 in. Sch 40 stainless steel OCI inlet pipe. This line was maintained continuously at 1200 F by external resistance heaters regardless of whether the OCI system was in operation or not. The failure was initially attributed to the short-out of the electrical trace heater, but a second failure in the same general location in a later program indicated the failures were due to localized overheating. Checking the monitoring surface thermocouple for this area showed this thermocouple was not indicative of the temperature in the pipe at the site of the failures and severe overheating had occurred. The heating and thermocouple systems were modified to correct this situation.

One operating problem encountered that did not require interruption of the test was the plugging of the cover gas lines to the test units and to the expansion tank. Although insulation and trace heaters were installed on all the lines back to the cover-gas manifold and maintained above the sodium-melting temperature, sodium plugs would still form in some of the lines. The cover gas was analyzed for traces of oxygen or moisture in the belief that either of these in contact with the sodium vapors diffusing into

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5. KAPL Report 1807, Solubility of Carbon in Sodium at Elevated Temperatures, by J. G. Gratton, June 30, 1957.

Table 10 - Carbon Content of Sodium Samples Removed From Loop 1 (ss Specimens)

Date	Carbon Content - ppm
5-1-62	43
5-8-62	127
5-15-62	61
5-29-62	40
6-5-62	123
6-12-62	64
6-19-62	48
6-26-62	56
7-3-62	47
7-10-62	10
8-7-62	43
8-14-62	152
8-21-62	> 59
8-28-62	50
9-4-62	41
9-11-62	29
9-17-62	65
10-12-62	29
10-15-62	33
10-15-62	31
10-16-62	27
10-16-62	51
10-18-62	38
10-18-62	20
10-19-62	31
10-19-62	61
10-23-62	30
10-30-62	45
11-6-62	32
11-13-62	69
11-20-62	80
11-26-62	38
11-27-62	76
12-5-62	31
12-11-62	34
12-19-62	42

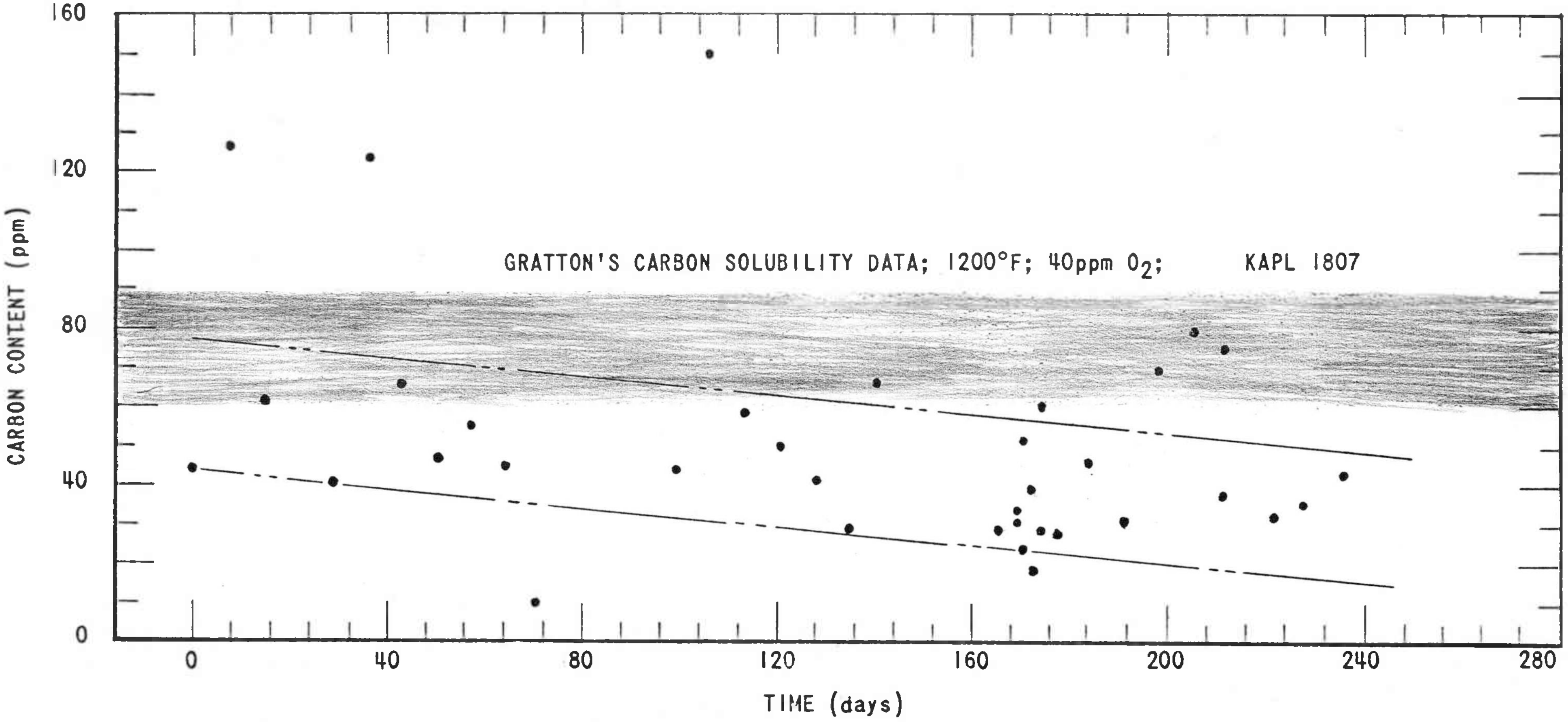

FIG. 51 - CARBON CONTENT, LOOP I, TEST I
(SS TEST SPECIMENS)

these lines were forming plugs. However, these analyses showed less than 1 ppm oxygen or moisture present in these gases. A second possibility was back diffusion of air through the flanges sealing the tops of the test units. These flanges incorporated a stainless steel O-ring for a seal. Upon opening these flanges, at the completion of the tests, some evidence of sodium vapor leaking through this seal was observed. The cover gas pressure in the test units was maintained at 2 in. H_2O pressure and it is possible that some back diffusion of air was taking place, although this could never be verified.

Flexible metal hoses with stainless steel overbraid were used for the inlet and outlet lines to six of the test units. These hoses were used to eliminate the need for complex rigid piping in maintaining low piping stresses. No difficulties were encountered with these units during this program.

Metallic bellows were used in the cover gas phase of the test units as a flexible seal between the pull rods and the atmosphere. The bellows on the fatigue machine were changed periodically since the design life was not expected to exceed 50,000 cycles with any reliability. There were no failures in the fatigue bellows during testing. The bellows on the 4000-hour creep tests were found to be cracked, upon completion of testing, although they had not been extended beyond their design limit.

The OCI system was the eighth parallel circuit in the test loop. The inlet to this system was ahead of the pump and the return line re-entered the main system at the expansion tank. The purpose of this circuit was to monitor and control the saturation temperature of the sodium system at an equivalent of 30 ppm oxygen or less.

The oxygen level of the system was checked at least twice weekly. The oxygen control section (cold trap) was activated when the oxygen level of the system exceeded 30 ppm or just prior to establishing flow through a test unit. Experience showed that putting a test unit on stream temporarily increased the oxygen concentration of the system. Although it was possible to run the cold trap continuously, this would set up an undesirable ΔT in the system. Therefore, it was activated only as needed. No unusual problems arose concerning the operation of this system. An EM pump was added to the circuit to provide additional driving force through the cold trap as the test progressed and the cold trap became more concentrated with oxides.

The use of the plugging indicator valve proved to be a reliable method of determining the oxygen saturation temperature of the system.

11. CONCLUSIONS

Figs. 52 and 53 compare the properties of the 316 ss test materials used in this program against several typical 316 ss heats. Comparing the tensile, creep and creep-rupture tests in 1200 F air shows these properties to be average and, therefore, the results could be expected to be representative of Type 316 ss.

In summarizing the results of the fatigue, creep-to-rupture, creep and tensile tests on 316 stainless steel specimens in 1200 F sodium with 30 ppm or less of oxygen, the following conclusions have been drawn:

11.1 CYCLIC STRAIN (FATIGUE) TESTS

The fatigue life of 316 ss in 1200 F helium is considerably longer than in 1200 F air throughout the range of the cyclic strains tested. The increase in fatigue life varies by a factor of 1.9 at the high cyclic strain to 4.9 at the low cyclic strain.

The fatigue life of 316 ss in 1200 F sodium is the same as in air at high cyclic strains, but is the same as in 1200 F helium at the low cyclic strains.

The 316 ss specimens exposed for relatively short times in 1200 F sodium and then tested in air, helium and sodium showed generally, when compared to the original material in the same environment:

a. Shorter life when tested in 1200 F air.

b. A trend toward longer life in 1200 F sodium.

c. No change when tested in 1200 F helium.

11.2 CREEP-RUPTURE TESTS

The test results are in such good agreement that there appears to be no significant differences in the creep-rupture properties of 316 ss in air, sodium or helium at 1200 F.

Specimens exposed for 4000 hours (unstressed) in 1200 F sodium showed no significant change in creep-rupture properties when tested in 1200 F air and helium. There appears to be a trend for the exposed specimens to have extended life when tested in 1200 F sodium.

MSA RESEARCH CORPORATION
CALLERY, PA., U.S.A.
FEBRUARY 17, 1964

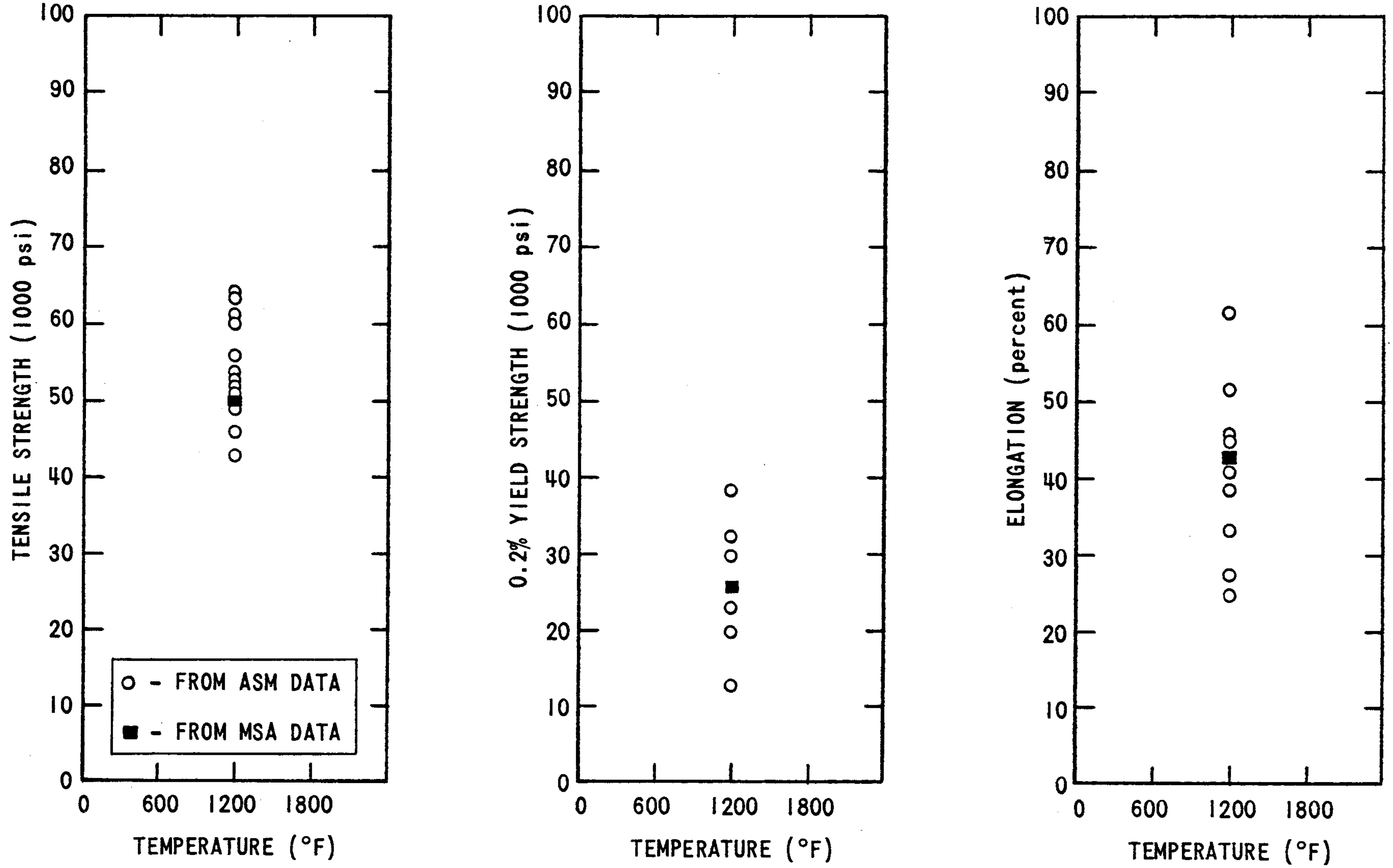

FIG. 52 - COMPARISON OF MSAR AIR DATA WITH OTHER 316 STAINLESS STEEL HEATS
(Data from Metals Handbook - Vol. I, Properties and Selection of Metals - American Society for Metals)

MSA RESEARCH CORPORATION
CALLERY, PA., U.S.A.
FEBRUARY 14, 1964

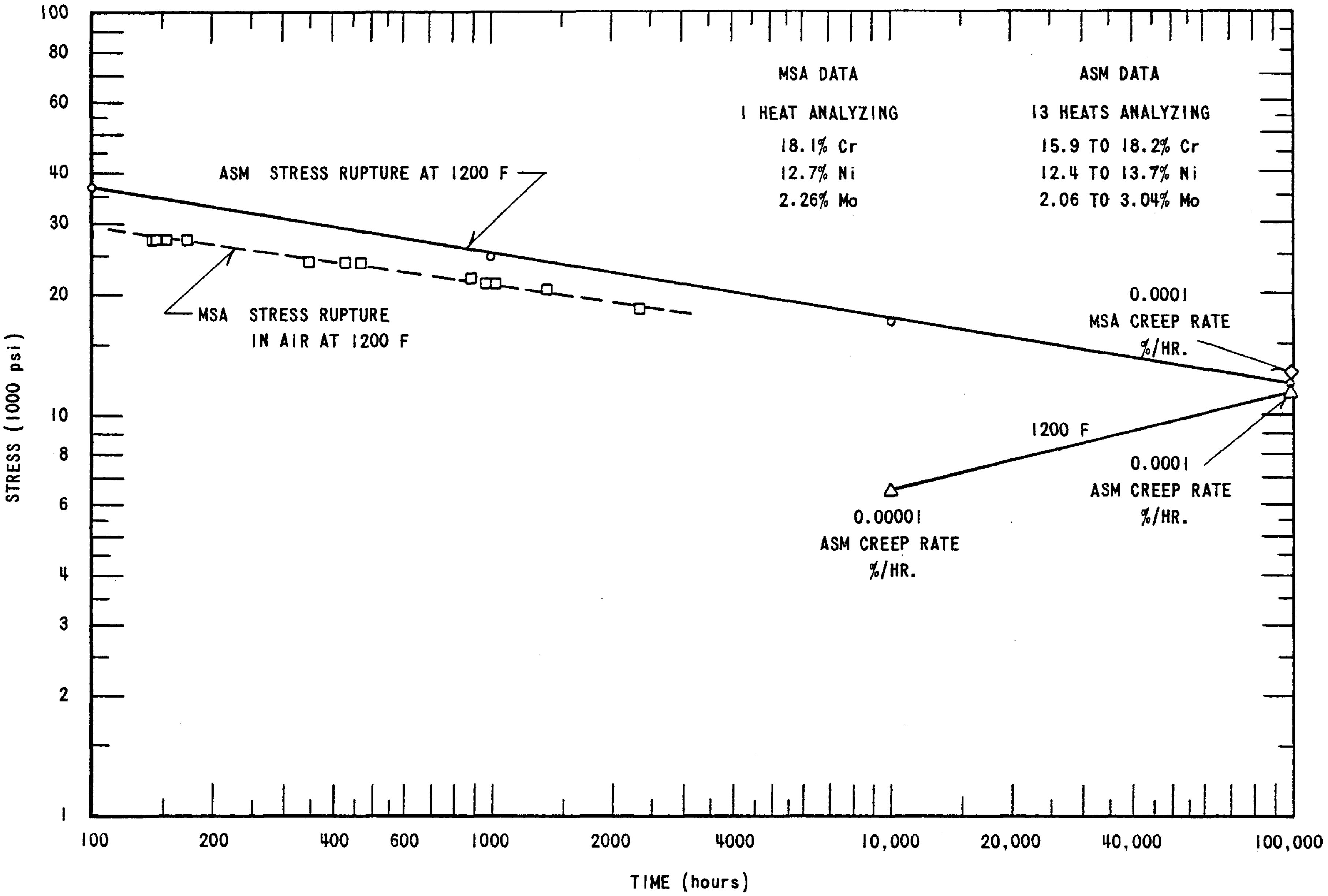

FIG. 53 - COMPARISON OF MSAR AIR RUPTURE AND CREEP DATA WITH OTHER 316 ss HEATS
(Data from Metals Handbook - Vol. I, Properties and Selection of Metals - American Society for Metals)

11.3 CREEP TESTS

The creep rates of 316 ss appear to be consistently higher in 1200 F helium and sodium compared to 1200 F air; however, the results show the stresses to produce a minimum creep rate of 1% in 10,000 hours to be: 12,300 psi for air, 10,900 psi for sodium, and 10,650 psi for helium. This is less than a 15% variation.

11.4 TENSILE TESTS

There were no significant differences in the tensile properties when tested in air or helium at 1200 F.

Specimens exposed for 4000 hours in 1200 F sodium indicated no significant tensile property change when tested in 1200 F helium regardless as to whether the specimens were washed or unwashed after exposure.

Specimens exposed for 4000 hours in 1200 F sodium indicated no significant change in tensile property when tested in room temperature helium and compared to original material tested under the same conditions.