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QUARTERLY PROGRESS REPORT
January-February-March 1968

Sponsored by
U. S. Atomic Energy Commission
Chicago Operations Office
Argonne, Illinois

MASTER

EFFECT OF HIGH TEMPERATURE SODIUM
ON THE
MECHANICAL PROPERTIES
OF
CANDIDATE ALLOYS
FOR THE
LMFBR PROGRAM

May 1968

MSA RESEARCH CORPORATION
DIVISION OF MINE SAFETY APPLIANCES COMPANY
EVANS CITY, PENNSYLVANIA 16033

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Contract AT(11-1)-765

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Signed:

R. C. Andrews
R. C. Andrews
Project Engineer

Signed:

K. R. Barker
K. R. Barker
Project Supervisor

Approved:

R. C. Werner
R. C. Werner
Associate Director
Engineering and
Development

MSA RESEARCH CORPORATION
Evans City, Pennsylvania 16033

leg

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SUMMARY

The present status of the Mechanical Properties Test Program facilities is as follows:

- LOOP 1 - Operating and performing excursions with high carbon-sodium on 15 and 60 mil, 304 and 316 ss specimens at 1200 F.
- LOOP 2 - Operating and performing excursions in sodium with 50 ppm oxygen on 60 mil, 304 and 316 ss specimens at 1200 F and 1400 F.
- LOOP 3 - Operating and performing creep-rupture tests on specimens from Loop 2 at 1200 F with a velocity of 20 ft/sec across the test specimens in 'clean' sodium (less than 10 ppm oxygen) and a system ΔT of 150 F.

STATIC RETORTS

- Unit 1 - Long-term (minimum 10,000 hours) exposure of 2 1/4 Cr-1 Mo at 1050 F is in progress.
- Units 2 and 3 - Screening tests of 1000-, 2000-, and 3000-hour exposures at 1200 F of six ferritic steels are in progress.
(The 1000-hour exposure has been completed.)
- Unit 4 - Long-term (minimum 10,000 hours) exposure in sodium (<10 ppm O_2) of 304 and 316 ss at 1200 F is in progress.

The program work performed during this report period is as follows:

PHASE I, TASK 1 - AUSTENITIC STAINLESS STEEL - SODIUM (50 ppm O_2) EXCURSION

The 500-hour excursions of 15 mil and 60 mil, 304 and 316 ss specimens were completed during this report period at both 1200 F and 1400 F. Excursions of similar specimens sealed in helium cans and exposed to the same thermal history as the above

were also completed. The oxygen concentration was maintained at 50 ppm by control of the cold trap at approximately 550 F throughout these excursions.

Test specimens from these excursions were tensile tested at room temperature, 1200 F and 1400 F. All changes due to the excursions were relatively small and the thin material usually accentuated the changes in yield strength and ductility. Recognizing the over-all changes as being small, the following summary attempts to point out trends by comparing the sodium versus helium properties.

304 ss Exposed to Na (50 ppm O₂) for 500 Hours at 1200 F

1. Increased 1200 F and 1400 F yield strength.
2. No relative change in tensile strengths.
3. Decreased room-temperature, 1200 F and 1400 F ductility, except the 60 mil room-temperature ductility showed a slight increase.

316 ss Exposed to Na (50 ppm O₂) for 500 Hours at 1200 F

1. Increased room-temperature, 1200 F and 1400 F yield strength except the 60 mil 1400 F yield strength showed a slight decrease.
2. No relative change in tensile strengths. The 1400 F 15 mil tensile strengths showed the largest change (increased).
3. Decreased room-temperature, 1200 F and 1400 F ductility.

304 ss Exposed to Na (50 ppm O₂) for 500 Hours at 1400 F

1. Decreased room-temperature, 1200 F and 1400 F yield strengths of 15 mil specimens. Little change in 60 mil specimens.
2. Decreased room-temperature, 1200 F and 1400 F tensile strengths of 15 mil specimens with very slight changes in 60 mil specimens.
3. Decreased room temperature, 1200 F and 1400 F ductility, except room-temperature 60 mil tensile strengths showed slight increase.

316 ss Exposed to Na (50 ppm O₂) for 500 Hours at 1400 F

1. Decreased room-temperature, 1200 F and 1400 F yield strengths of 15 mil specimens. Little change in 60 mil specimens except the 1400 F ductility decreased.
2. All tensile strengths decreased except 15 mil 1200 F tensile strengths which increased slightly.
3. Ductility decreased at all temperatures except the room-temperature 60 mil which showed no change.

PHASE I, TASK 2 - HIGH CARBON EXCURSION OF 304 and 316 SS AT 1200 F

Three different excursions were conducted on 15 mil specimens in order to obtain a variety of total carbon values in the test material. Subsequent measurement of tensile strengths following these excursions indicated the effects of these carbon increases. As a result of these excursions, the total carbon values for the 304 ss 15 mil material in 100 hours were 1531 ppm, 1900 ppm and 2875 ppm. Carburized 316 ss material of the same thickness and exposure time showed total carbon values of 1348 ppm, 1625 ppm and 3000 ppm.

The general results of the tensile tests conducted in the carburized specimens and compared to the helium exposed specimens are summarized as follows:

304 ss Exposed Na (Carbon) at 1200 F - 15 mil Specimens

1. The yield strengths increased at room temperature, 1200 F and 1400 F.
2. The tensile strengths were equal at room temperature for two out of the three excursions and showed general increases at 1200 F and 1400 F.
3. In all cases the ductility decreased.

316 ss Exposed Na (Carbon) at 1200 F - 15 mil Specimens

1. The yield strengths increased at room temperature, 1200 F and 1400 F and appeared more pronounced than in the 304 ss material.
2. The tensile strengths increased at room temperature, 1200 F and 1400 F.
3. In all cases the ductility decreased.

The results also indicated that as the total carbon content increases in the specimen, the tensile and yield strengths at room temperature increased, with a subsequent decrease in ductility, -- with one exception: the 304 ss room temperature tensile strength did not show an increase with carbon content. The tensile tests at 1200 F showed an increase in tensile and yield strengths for both materials and a decrease in ductility. The decrease in ductility is not as great at 1200 F as at room temperature. At 1400 F, the tensile and yield strengths continued to increase with increasing carbon content and with a decrease in ductility.

PHASE II - FERRITIC STEELS

Task 1 - Creep Rupture Properties in Clean Sodium

The creep-rupture tests are preceded by a screening test involving the static exposure of the six ferritic steels for 1000, 2000 and 3000 hours in stainless steel retorts at 1200 F. The sodium chemistry of the static sodium was characterized and the 1000 and 3000-hour exposures initiated.

Task 2 - Long-term Static Exposures at 1050 F

Following the characterization of the sodium, specimens of 2 1/4 Cr-1 Mo steel were immersed at 1050 F and are scheduled for 10,000 and 20,000 hours.

PHASE III - OTHER CANDIDATE ALLOYS

Contacts have been made with Westinghouse and the AEC to obtain test material from their vanadium-base alloy programs when it becomes available.

PHASE IV - HIGH TEMPERATURE EXTENSOMETER

The model extensometer and electrical system under test with the extensometer unit in 1200 F failed at the end of 820 hours. The cause of failure was the deterioration of the Constantan-wire core. New material (Inconel X) has been ordered to replace this coil. The new coil and testing is scheduled for July.

PHASE V - ANALYTICAL TECHNIQUES

The surface tension of sodium contaminated with oxygen is failing to generate reproducible results that would permit positive conclusions. Based on the results of surface tension measurements in a static sodium system with various cold-trap temperatures, the test machine revealed sufficient inadequacies which prevent definite conclusions from being drawn.

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1. INTRODUCTION

MSAR's initial efforts under AEC Contract AT(11-1)-765 were to determine the effects of sodium and its normally anticipated contaminants on the mechanical properties of structural materials anticipated for use in nuclear power plants cooled with sodium and operating at a maximum temperature of 1200 F. The original materials considered were 316 ss and 2 1/4 Cr-1 Mo with temperature limits of 1200 F and 1100 F respectively. Later, the program was expanded to include 304 ss as a candidate material for use at 1200 F. The environmental effects were to be evaluated through several mechanical properties tests (creep, creep-rupture, fatigue, tensile and impact tests) and metallographic examinations. Environmental effects of 'clean' sodium, oxygen-contaminated sodium, and carbon-contaminated sodium were evaluated by comparison of data from tests in these respective environments with data from similar tests on the same heats in air and helium. This information was disseminated in the several topical reports as listed in Table 1.

In the fall of 1966, the program was redirected towards support of the LMFBR Program where several additional candidate alloys and alloy classes were being considered for use. In addition to the two existing 316 ss dynamic sodium systems (Loops 1 and 2) used in the earlier portions of the program, a third dynamic system (Loop 3) and four static exposure retorts were constructed. The Loop 3 system is being operated with a 'clean' sodium (<10 ppm O₂) environment with added parameters of ΔT (50 to 150 F), velocities (15 to 30 ft/sec), temperature (1400 F maximum), and variations in specimen thickness (15 to 60 mil). The static retorts are exposing, simultaneously, specimens in both a helium and 'clean' sodium (150-pound capacity) environment for extended lengths of time (in excess of 10,000 hours) with the necessary hardware for extracting sodium samples for characterization of the sodium chemistry throughout the exposure. Sodium chemistry of all the sodium systems is monitored, using updated state-of-the-art sampling methods and analytical procedures. Loop 3 is the most complete, including a bypass sampler, hot trap and two UNC oxygen meters. Loop 1 has a UNC carbon meter. Post-test evaluations of the effects of stress and corrosion on the candidate materials are made by metallographic and elemental analyses. Microhardness measurements, microprobe and X-ray diffraction analyses are employed to aid in the delineation of chemistry and phase changes occurring during testing and/or exposures.

Additional areas being investigated under this program include a method for the direct measurement of creep rates in a high

TABLE 1 - MSAR TOPICAL REPORTS GENERATED UNDER AT(11-1)-765

Report No.	Title	MSAR Report No.
1	TEST FACILITY DESIGN AND OPERATION PROCEDURES, November 1963	63-161
2	RESULTS OF MECHANICAL PROPERTIES TEST OF 316 SS SPECIMENS IN 1200 F SODIUM WITH LOW OXYGEN, March 1964	64-18
3	RESULTS OF MECHANICAL PROPERTIES TEST OF 2 1/4 Cr-1 Mo STEEL SPECIMENS IN SODIUM WITH HIGH AND LOW OXYGEN, AIR, AND HELIUM ENVIRONMENTS AT 1100 F, July 1964	64-81
4	RESULTS OF MECHANICAL PROPERTIES TESTS OF 316 SS SPECIMENS IN 1200 F SODIUM CONTAMINATED WITH CARBON, December 1965	65-194
5	RESULTS OF MECHANICAL PROPERTIES TESTS OF 316 SS SPECIMENS IN 1200 F SODIUM CONTAMINATED WITH OXYGEN (~300 ppm), May 1966	66-78
6	EFFECT OF CARBURIZATION ON THE PHASES IN AUSTENITIC STAINLESS STEEL, June 1966	66-119
7	DESIGN PROPERTIES OF ONE HEAT OF 2 1/4 Cr-1 Mo AND 316 STAINLESS STEEL IN HIGH TEMPERATURE AIR, HELIUM AND SODIUM ENVIRONMENTS, September 1966	66-174
8	RESULTS OF MECHANICAL PROPERTIES TESTS OF 304 SS SPECIMENS CONDUCTED IN: 1. 1200 F SODIUM CONTAMINATED WITH OXYGEN (~300 ppm); and 2. 1200 F SODIUM CONTAMINATED WITH CARBON, July 1967	67-103
9	ANALYSIS OF HIGH OXYGEN AND HIGH CARBON COLD TRAPS, December 1967	67-203
10	A LIMITED COMPARISON OF THE RELATIVE MERITS OF 304 VERSUS 316 STAINLESS STEEL FOR LIQUID METAL SERVICE	67-216

temperature liquid metal environment and the development of techniques for the measurement of impurities in sodium. Analyses of two cold traps used in mechanical properties studies, -- one having been exposed to a high oxygen-sodium environment and the other to a high carbon-sodium environment -- were presented in Topical Report No. 9 (see Table 1).

2. SCOPE OF THE MECHANICAL PROPERTIES PROGRAM

The present program in direct support of the LMFBR program is centered around the instream testing of candidate alloys. In the earlier testing of 304 and 316 ss, parameters such as ΔT , velocity and the material thickness were minimized to determine environmental effects. Under this program these parameters approach the conditions anticipated in a reactor system. Similarly, the pre-test excursions are in environments contaminated with oxygen or carbon as might be encountered in a reactor system. These excursions are limited to less than 1000 hours which is believed to be reasonable in actual reactor operation. Exposures in the static retorts under 'clean' sodium conditions are used for support of the instream testing in two manners. The first are short-time (1000-3000 hours) screening exposures where one or two alloys from several alloys of a particular class are selected for further instream testing. An example of these are the short-time exposures of five different ferritic steels in a decarburizing environment. The second type of exposures are the long-term (in excess of 10,000 hours) exposure in a 'clean' sodium environment (<10 ppm) of actual candidate alloys such as 304, 316 and 2 1/4 Cr-1 Mo. These exposures would then be followed by mechanical testing to provide comparative data.

The following outline details the tests being conducted under the program, including the purpose of each task.

PHASE I - AUSTENITIC STEELS

- TASK 1 - High oxygen sodium excursions of 304 and 316 ss at 1200 F and 1400 F followed by creep-rupture testing in flowing 'clean' sodium and by tensile testing in air

Type 304 and 316 ss specimens, 15 mil and 60 mil, will undergo 500-hour excursions, stress-free, in 1200 F and 1400 F sodium contaminated with oxygen at the 50 ppm level to simulate an oxygen excursion in a reactor system. Loop 2, formerly used for the high oxygen sodium testing, will be used to conduct these excursions. The specimens from these excursions will then be creep-rupture tested in Loop 3 under parameters of 'clean' sodium

(<10 ppm), ΔT (150 F maximum), velocity (20 ft/second) and temperature of 1200 F. This data will then be compared with previous test data to determine if the added parameters indicated above had additional effects on the mechanical properties. Tensile tests in air will be conducted from room temperature to 1400 F to aid in this determination.

TASK 2 - High carbon sodium excursion of 304 and 316 ss at 1200 F followed by creep-rupture testing in flowing 'clean' sodium and by tensile testing in air

Type 304 and 316 ss specimens, 15 mil thick, will undergo a 1200 F high carbon excursion in Loop 1 to simulate a carbon incident in a reactor system. The duration of the excursion will be such so as to provide a 0.2-0.3% carbon concentration in the 15 mil specimens. These specimens will then be tensile tested in air from room temperature to 1400 F, thereby providing a more definite picture of the ductility-carburization relationship of this alloy.

Thicker specimens (60 mil) of 304 and 316 ss will undergo a similar excursion. However, the duration of this excursion will be controlled to provide a 5-10 mil carburized case depth in the 60 mil specimens rather than any particular carbon level. The evaluation of the effect of such an excursion on the material strength is expected to be of prime importance to the designer. These specimens will subsequently be creep-rupture tested in Loop 3 and tensile tested in air, as described under Task 1. This data will provide information by comparing the less carburized specimens from these tests, including the additional parameters imposed by Loop 3 test conditions, with the more heavily carburized tests of the previous program.

TASK 3 - Long-term (10,000 to 30,000 hours) exposures of 304 and 316 ss in 'clean' sodium followed by creep-rupture tests in 'clean' sodium and by tensile tests in air

Type 304 and 316 ss specimens, 60 mil thick, will be exposed in a static retort. This retort has been fabricated with a diffusional-type cold trap which is maintained at 250 F or less for controlling the O₂ content at <10 ppm. A sodium sampling valve is centrally located in the top head from which bucket-type samples can be removed for analyses to fully characterize the sodium throughout the exposure period. The specimens are immersed in the sodium, suspended from the periphery of a helium-filled can inside of which are control specimens seeing the identical thermal history. The helium-exposed specimens will be used to differentiate between the thermal-aging effects and environmental effects that may appear in subsequent mechanical tests.

Specimens from this exposure will also undergo creep-rupture tests in Loop 3 and tensile tests in air, as outlined under Task 1. It is hopeful that data from these tests will help decide if long-term mechanical testing in sodium will be necessary.

TASK 4 - High oxygen and high carbon excursion of modified austenitic steels followed by creep-rupture tests in 'clean' sodium

At the present time, Oak Ridge National Laboratory is modifying medium carbon grades of 304 and 316 ss to increase their resistance to radiation damage. Task 4 will be similar to the testing of standard grades of 304 and 316 ss as outlined under Tasks 1 and 2. The data is intended to show the compatibility of these modified austenitics with sodium and its normal contaminants.

TASK 5 - Synergistic studies of high carbon-oxygen sodium exposures on 304 ss followed by creep-rupture testing in 'clean' sodium

During previous work with 304 ss in high oxygen and high carbon sodium, several synergistic effects were noted in metallographic examinations subsequent to mechanical testing. However, the effects of these changes on the strength properties were not determined. Under Task 5, 60 mil 304 ss specimens will undergo a high carbon sodium excursion in Loop 1 to form a carbide case depth of 5-10 mil. This will be followed by a 500-hour high oxygen sodium excursion in Loop 2 with the oxygen content of the sodium being at the 50 ppm level. A reverse-type exposure will also take place (high oxygen sodium first, followed by high carbon sodium). These specimens will then be creep-rupture tested in Loop 3 and tensile tested in air, as outlined in Task 1. From this data, an effort will be made to delineate the synergisms noted in earlier work.

PHASE II - FERRITIC STEELS

TASK 1 - Exposure of candidate ferritic alloys in a decarburizing environment followed by creep-rupture tests in 'clean' sodium

The potential stress-corrosion problems between the austenitics and water at elevated temperatures recently led to changes in the design studies of the steam generator in the LMFBR program from a combination of austenitics and ferritics to all-ferritic generator. Similarly, there has been widespread use of ferritics in the Euratom fast breeder reactor program. Past work with 2 1/4 Cr-1 Mo, -- one of the leading ferritics -- has shown it will readily undergo decarburization and subsequent loss of strength in the 1100 F temperature range in short-time periods (4000 hours).

Under Task 1, several leading ferritics -- some of which have been stabilized in hopes of decreasing the decarburization process -- will be exposed in all-stainless-steel retorts in which the austenitic retort itself will act as a carbon sink. The 'clean' sodium of the retort will act as the transport media. The alloys selected to date are listed in Table 2. A standard heat of 2 1/4 Cr-1 Mo will be used as a control during this exposure. A 1200 F exposure temperature was chosen to enhance decarburization over the excursion periods of 1000, 2000 and 3000 hours. These specimens will then be tensile tested in air. From this data, one or two of the most promising alloys will be selected for further testing in 'clean' sodium similar to that under Phase I, Task 1. Essentially, the first work under this task is a screening-type program whereby the more complete test program will be limited to one or two alloys.

TASK 2 - The long-time exposures (10,000 and 20,000 hours) of candidate ferritic alloys followed by creep-rupture tests in 'clean' sodium and tensile tests in air

As discussed under Task 1, there is a definite use of ferritic alloys in the LMFBR program which will require long-term exposure data for use at temperatures up to 1050 F. Under Task 2, 60 mil 2 1/4 Cr-1 Mo specimens and specimens of the most promising alloys from Task 1 will be exposed in a stainless steel retort at 1050 F for times in excess of 10,000 hours. As under Task 1, 'clean' sodium will be the transport media and the austenitic vessel will be the carbon sink to promote decarburization. The sodium chemistry will be monitored throughout the exposure period. Specimens will be exposed in both the sodium environment and a helium environment to separate thermal aging effects from the sodium corrosion effects in the subsequent creep-rupture tests in 'clean' sodium and tensile tests in air. This data will provide long-term decarburization effects and mechanical properties of ferritic steels.

PHASE III - OTHER CANDIDATE ALLOYS

TASK 1 - Creep-rupture tests in clean sodium

Recent work at MSAR and other investigators have demonstrated the limited use of 304 and 316 ss, particularly in the areas of fuel cladding where both decarburization and irradiation destroy the ductility of these materials. Several alloys are presently being developed to serve as backup materials. Such alloys include the vanadium alloys being developed by Westinghouse and Argonne National Laboratory; cobalt alloys being tested by Brookhaven National Laboratory for corrosion resistance and stability; and other Nb and Co alloys.

TABLE 2 - COMPOSITION OF FERRITIC TEST MATERIALS
FOR THE MSAR-LMFBR STRUCTURAL MATERIALS PROGRAM

Type of Material	Supplier	Heat No.	Cr	Mo	C	Mn	P	S	Si	Other
Mod. 2 1/4 Cr-1 Mo	U. S. S.	W8815	2.26	1.02	0.14	0.44	0.013	0.018	0.16	V = 0.62, Cb = 0.094, Al = 0.025
Stab. 2 1/4 Cr-1 Mo	B & W	--	2.25	1.01	0.10	0.45	0.023	0.011	0.49	Ti = 0.20, V = 0.20, Zr = 0.14, B = 0.005, Ni = 0.10, Co = 0.09
Rheinrohr 3HK5S	Thyssen Röhrenwerke		2.22	0.97	0.048	0.72	0.017	0.011	0.23	Ni = 0.61, Nb/Ta = 0.56
Croloy 5	B & W	47295	5.04	0.63	0.10	0.48	0.016	0.014	0.30	Ni = 0.23, Cu = 0.11
Croloy 5 Ti	B & W	10339	4.99	0.51	0.09	0.48	0.014	0.015	0.23	Ni = 0.24, Cu = 0.12, Ti = 0.45

Under Task 1, a limited program is scheduled for creep-rupture testing in 'clean' sodium at 1400 F of the most promising of these alloys. Initially, screening-type testing will guide the program until data indicate a more complete test program is warranted.

TASK 2 - High oxygen sodium excursions followed by creep-rupture testing in 'clean' sodium and tensile testing in air

Task 2 is more or less a continuation of the screening tests under Task 1. Here, 10-20 mil specimens will undergo short-time excursions (100 hours) in high oxygen sodium (20-30 ppm) at 1400 F prior to their testing in clean sodium. Particular attention will be given to the carbon, oxygen and nitrogen contents of these alloys.

PHASE IV - EXTENSOMETER DEVELOPMENT

A linear variable differential transformer (LVDT) type of extensometer will be tested in 1200 F sodium. The performance goal is the capability of measuring a strain rate of 0.0001%/hr at 1200 F. The instrument must also be usable after 1000 hours of exposure to 1200 F sodium.

PHASE V - ANALYTICAL TECHNIQUES

This task is comprised of research and development on the various, promising analytical techniques. At the present time, only work on the use of surface tension as a means of detecting oxygen content in sodium is being studied. It is anticipated that the studies of the UNC oxygen and carbon meters will be conducted under this portion of the contract.

Summary of the Mechanical Properties Program

The scope of the MSAR-LMFBR program is summarized in Table 3. The program is directly involved in the material-environmental problem areas of the LMFBR program, and the data generated is expected to be of considerable value in making material selections and, in some instances, contributing to design values. The program is also intended to clarify avenues in which additional investigations and efforts will lead to optimization of material selections and code values.

TABLE 3 - SUMMARY OF MSAR-LMFBR STRUCTURAL MATERIAL TEST PROGRAM

Phase	Program	Facility
PHASE I - AUSTENITIC STEELS		
Task 1	Oxygen excursions of 316 ss and 304 ss at 1200 F and 1400 F followed by creep-rupture tests in clean sodium.	High oxygen sodium (50 ppm) loop and clean sodium loop.
Task 2	Carbon excursion of 316 ss and 304 ss at 1200 F and 1400 F followed by creep-rupture tests in clean sodium.	High carbon sodium loop and clean sodium loop.
Task 3	Long-term (10,000 and 20,000 hrs) exposures of 316 ss and 304 ss in clean sodium followed by creep-rupture tests in clean sodium.	Static pots and clean sodium loop.
Task 4	Oxygen and carbon excursions on modified austenitic steel followed by creep-rupture tests in clean sodium.	High oxygen, high carbon and clean sodium loops.
Task 5	Synergistic studies of carbon-oxygen exposures followed by creep-rupture tests in clean sodium.	High carbon, high oxygen and clean sodium loops.
PHASE II - FERRITIC STEELS		
Task 1	Exposure in decarburizing environment followed by creep-rupture tests in clean sodium.	Static pots and clean sodium loop.
Task 2	Long-time exposures (10,000 and 20,000 hrs) followed by creep-rupture tests in clean sodium.	Static pots and clean sodium loop.
PHASE III - OTHER CANDIDATE ALLOYS		
Task 1	Creep-rupture tests in clean sodium.	Clean sodium loop.
Task 2	Oxygen excursion followed by mechanical testing in clean sodium.	High oxygen loop and clean sodium loop.
Task 3	Carbon excursion followed by mechanical testing in clean sodium.	High carbon loop and clean sodium loop.
PHASE IV	Extensometer development.	
PHASE V	Development of techniques for measurement of impurities in sodium.	

3. PROGRAM PROGRESS

PHASE I - AUSTENITIC STAINLESS STEELS

PHASE I - TASK 1: 500-HOUR EXCURSION OF 304 and 316 ss in 1200 F and 1400 F HIGH OXYGEN SODIUM

1200 F. Excursion

The parameters selected for the high oxygen excursion of 304 and 316 ss prior to their mechanical testing in air and 'clean' sodium (Loop 3) were to include an oxygen content of 50 ppm, an exposure temperature of 1200 F, an exposure duration of 500 hours, and a minimum system ΔT of 50 F. All of these parameters were to take place in a low velocity (< 1 ft/sec) dynamic sodium system. These operating conditions were obtained in Loop 2 during this report period.

Four Loop 2 exposure units were assembled with a combination of 15 mil and 60 mil, 304 and 316 ss specimens, totaling 30 specimens per unit. A fifth unit was assembled whereby a total of 32 specimens of the aforementioned combination were inserted into a stainless steel can under a helium atmosphere to provide thermally aged specimens as controls. These five units were put on-stream between January 7 and January 11, 1968, initiating 500-hour excursion periods.

Figure 1 provides pertinent flow and temperature data maintained throughout the excursion period.

The oxygen concentration (50 ppm) was maintained by control of the cold trap (~ 500 F).

The sodium chemistry was monitored throughout this period by use of a calibrated plugging indicator and via several sodium samples. A table of these results follows:

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EVANS CITY, PA., U.S.A.
MARCH 25, 1968

FLOW RATES—	GPM
UNITS 1-5	0.50
COLD TRAP	1.75
BY-PASS	1.00
TOTAL	5.25

TEMPERATURES	
T1	1150°F MAX
T2	1200°F ± 5
T3	530°F ± 10

FIG. 1 - AVERAGED LOOP CONDITIONS DURING 1200 F HIGH OXYGEN (50 ppm) EXCURSION
OF 304 and 316 STAINLESS STEEL SPECIMENS
Conducted January 7, 1968
PHASE I, TASK 1

Date	Saturation Temp., °F	PI Runs	Analytical Determinations				
		Equivalent O ₂ Content (1), ppm	ppm				
			O ₂	C	H ₂	C ₂ H ₂	CO ₂
1/9	520	62	57	-	-	-	-
1/11	515	58	-	-	-	-	-
1/12	-	-	-	59	11	-	-
1/16	480	37	33	-	-	4	57
1/18	525	66	-	-	-	-	-
1/19	510	55	-	-	-	-	-
1/22	520	62	(2)	-	-	-	-
1/23	500	48	(2)	-	-	-	-
1/26	510	55	-	-	-	-	-
1/31	520	62	-	-	-	-	-
2/1	-	-	70	-	-	-	-

(1) MSAR Loop 2 Calibration Curve, MSAR 68-14.

(2) Samples extracted. Failure in analytical equipment.

In addition, two samples extracted on January 15 and January 22, 1968, analyzed for metallic constituents by emission spectrometry did not reveal any abnormalities.

The carbon activity was monitored by exposing 5 mil 304 ss tabs in the instream expansion tank on a weekly basis with analysis of the tab for total carbon content. A table of the results prior to this excursion period follows:

5 Mil 304 ss Carbon Monitoring Tab Results
(Instream Expansion Tank - 1200 F)

Dates	Tab No.	Exposure Time, Hrs	Total Carbon*, ppm
1/6 - 1/12	4-304	147	502
1/13 - 1/22	4-306	215	375
1/22 - 1/29	4-316	170	341

* Original Total Carbon, ppm = 545 ± 50

These results indicate that the carbon activity in the sodium prior to starting this excursion was sufficiently low to prevent carburization of the 304 ss monitoring tabs. If anything, there may have been a slight decarburization of the tabs.

The high oxygen excursions were concluded between January 29 and February 1, 1968. The specimens were carefully washed with alcohol and water and stored in a vacuum desiccator until required for mechanical testing.

Test specimens from this excursion were tensile tested at room temperature, 1200 F and 1400 F. Table 4 shows the tensile strengths of the as-received material; Table 5, the strengths of the helium-exposed specimens accompanying the sodium-exposed specimens; and Table 6, the strengths following the 500-hour excursion in the high oxygen sodium.

Figures 2, 3 and 4 graphically compare the tensile strength results of 304 ss for the three environments, while Figures 5, 6 and 7 show the same comparison for the 316 ss tests. In each figure, typical 304 or 316 tensile data is given as published in the United States Steel Bulletin, "Steels for Elevated Temperature Service."

Discussion of Results of 500-hour 1200 F Excursion of 304 and 316 ss in High Oxygen Sodium

As-received

Single heats of 304 and 316 ss were used in this program. The suppliers' chemical composition of these materials is as follows:

Type of Material	Heat No.	Cr	Ni	C	Mn	P	S	Si	Mo
304	322891	18.26	9.36	0.057	1.20	0.023	0.010	0.52	0.15
316	19295	17.64	12.72	0.048	1.68	0.019	0.011	0.56	2.85

Comparison in the preceding figures of the tensile data of these heats with the typical tensile data of respective materials shows the 304 to give values slightly higher than average, whereas the 316 values are closer to the average.

Helium-exposed

Thermal effects, as demonstrated in the short-time tensile tests of the helium-exposed specimens were normal. Tensile strength decreases linearly with temperature to approximately 1000 to 1100 F, at which point the slope begins to become steeper, reaching a sharp angle at about 1400 F. The yield strength behaves similarly until about 1100 F. Between 1100 and 1300 F, where carbide effects occur, there is an inversion. Beyond 1300 F, carbide solution takes over and yield strength again decreases. The ductility data is at the minimum at 1200 F; however, no tests were conducted between ambient temperature and 1200 F. Since the data appear normal in all other respects, it is expected that the ductility increases gradually from ambient temperature to 800-900 F, at which point it begins to decrease due to the carbide effects. It reaches a minimum between

TABLE 4 - 304 and 316 ss TENSILE DATA - AS-RECEIVED MATERIAL
PHASE I, TASK 1

Spec. No.	Material	Nominal Thickness, In.	Test Temp., °F	Yield Strength, psi	Average Y.S., psi	Tensile Strength, psi	Average T.S., psi	Elong. %	Average Elong., %	Reduction of Area, %
A1	304	0.060	P.T.	46,150		96,500		69		64
A2	"	"	R.T.	45,300	45,066	96,300	98,730	72	73	64
A5	"	"	R.T.	43,750		104,400		78		70
A66	"	"	1200	24,000		46,400		43		50
A3	"	"	1200	25,810	23,905	48,830	47,615	45	44	49
A67	"	"	1400	18,610		25,000		67		55
A68	"	"	1400	18,170		25,030		75		40
A67	"	"	1400	17,330	18,195	22,770	23,842	76	72	60
A70	"	"	1400	18,670		22,570		73		60
L62	"	0.015	R.T.	45,710		93,570		62		49
L63	"	"	R.T.	35,000	40,355	92,860	93,215	70	66	55
L58	"	"	1200	20,930		43,710		39		40
L59	"	"	1200	21,430	21,180	45,000	44,355	40	40	43
L60	"	"	1400	17,430		28,570		47		47
L61	"	"	1400	18,280	17,855	25,860	27,215	51	49	48
B1	316	0.060	R.T.	45,000		86,330		38		64
B41	"	"	R.T.	47,000		90,170		65		67
B40	"	"	R.T.	44,280	46,570	89,660	88,232	65	59	65
B42	"	"	R.T.	--		86,670		65		59
B43	"	"	R.T.	50,000		88,330		63		61
B61	"	"	1200	27,100		54,900		45		53
B62	"	"	1200	27,660	27,380	57,060	55,980	43	44	53
B63	"	"	1400	23,470		33,000		83		70
B64	"	"	1400	23,500	23,485	33,330	33,165	83	83	73
M62	"	0.015	R.T.	50,000		87,430		53		48
M63	"	"	R.T.	--	46,355	88,860	88,193	58	56	49
M64	"	"	R.T.	42,710		88,290		58		51
M58	"	"	1200	25,000		55,140		40		43
M59	"	"	1200	27,860	26,430	50,710	52,925	40	40	43
M60	"	"	1400	19,290		31,640		61		54
M61	"	"	1400	24,280	21,785	33,710	32,675	58	59	51

TABLE 5 - 304 and 316 ss TENSILE DATA - 500-HOUR HELIUM EXPOSED AT 1200 F
PHASE I, TASK 1

Spec. No.	Material	Nominal Thickness, In.	Test Temp., °F	Yield Strength, psi	Average Y.S., psi	Tensile Strength, psi	Average T.S., psi	Elong. %	Average Elong., %	Reduction of Area, %
A6	304	0.060	R.T.	36,900		91,720		60		65
A7	"	"	R.T.	41,670	39,285	95,830	93,775	60	60	66
A8	"	"	1200	25,520		47,310		44		58
A9	"	"	1200	23,780	24,650	46,340	46,825	45	45	55
A11	"	"	1400	21,670		28,270		76		70
A12	"	"	1400	20,550	21,110	30,210	29,240	74	75	72
L2	"	0.015	R.T.	41,160		95,650		60		46
L3	"	"	R.T.	40,000	40,580	94,860	95,255	60	60	54
L4	"	"	1200	20,290		44,060		42		42
L5	"	"	1200	23,910	22,100	44,930	44,495	40	41	48
L6	"	"	1400	20,290		28,400		61		54
L7	"	"	1400	22,140	21,215	27,140	27,770	55	58	51
B2	316	0.060	R.T.	46,550		95,690		48		56
B3	"	"	R.T.	46,670	46,610	95,000	95,345	50	49	63
B4	"	"	1200	28,000		53,330		43		56
B5	"	"	1200	30,000	29,000	54,130	53,730	40	42	56
B6	"	"	1400	27,930		36,760		74		69
B7	"	"	1400	29,180	28,555	35,990	36,375	69	72	67
M1	"	0.015	R.T.	42,030		89,850		44		46
M2	"	"	R.T.	46,380	44,205	95,650	92,750	43	44	46
M3	"	"	1200	30,580		54,640		28		38
M4	"	"	1200	30,710	30,645	52,570	53,605	22	25	40
M5	"	"	1400	25,710		33,860		54		51
M6	"	"	1400	25,360	25,535	34,930	34,395	60	57	52

TABLE 6 - 304 and 316 ss TENSILE DATA - 500-HOUR HIGH OXYGEN SODIUM EXPOSED AT 1200 F
PHASE I, TASK 1

Spec. No.	Material	Nominal Thickness, In.	Test Temp., °F	Yield Strength, psi	Average Y.S., psi	Tensile Strength, psi	Average T.S., psi	Elong. %	Average Elong., %	Reduction of Area, %
A20	304	0.060	R.T.	40,000		95,670		62		63
A21	"	"	R.T.	42,240	41,120	97,930	96,800	63	63	61
A22	"	"	1000	24,830		54,480		34		58
A23	"	"	1000	24,830	24,830	57,590	56,035	30	32	58
A14	"	"	1200	26,900		47,590		39		58
A15	"	"	1200	26,210	26,555	45,100	46,345	40	40	62
A16	"	"	1400	23,330		30,800		68		67
A17	"	"	1400	23,080	23,205	30,430	30,615	63	66	63
L18	"	0.015	R.T.	42,030		93,480		47		45
L19	"	"	R.T.	42,030	42,030	92,160	92,820	53	50	43
L20	"	"	1000	25,710		54,570		30		37
L21	"	"	1000	23,190	24,450	51,300	52,935	28	29	37
L8	"	"	1200	28,670		43,600		21		37
L11	"	"	1200	33,140	30,905	48,000	45,800	23	22	34
L12	"	"	1400	27,680		31,450		22		33
L13	"	"	1400	25,000	26,340	30,130	30,790	24	23	39
B18	316	0.060	R.T.	48,330		95,330		48		60
B19	"	"	R.T.	48,330	48,330	95,170	95,250	48	48	60
B20	"	"	1000	30,000		65,860		30		51
B21	"	"	1000	32,000	31,000	66,000	65,930	30	30	56
B10	"	"	1200	33,330		56,000		40		56
B11	"	"	1200	31,720	32,525	56,550	56,275	36	38	55
B12	"	"	1400	27,610		36,700		63		68
B13	"	"	1400	27,330	27,470	34,670	35,685	60	62	70
M17	"	0.015	R.T.	50,570		94,570		38		38
M18	"	"	R.T.	44,710	47,640	93,710	94,140	32	34	44
M19	"	"	1000	30,720		61,600		20		27
M20	"	"	1000	32,860	31,790	65,710	63,655	22	21	34
M9	"	"	1200	36,960		53,620		20		24
M10	"	"	1200	38,400	37,680	57,250	55,435	25	23	27
M11	"	"	1400	32,860		38,860		26		43
M12	"	"	1400	31,880	32,370	41,300	40,080	32	29	35

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* FROM USS BULLETIN "STEELS FOR ELEVATED TEMPERATURE SERVICE"

FIG. 2 - 0.2% OFFSET STRENGTH OF 304 SS
15 mil and 60 mil SPECIMENS AFTER 500-HOUR EXPOSURE
TO 1200 F HELIUM AND SODIUM (50 ppm O₂)
PHASE I, Task 1

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* FROM USS BULLETIN, "STEELS FOR ELVATED TEMP. SERVICE"

FIG. 3 - TENSILE STRENGTH OF 304 ss 15 mil and 60 mil SPECIMENS
AFTER 500-HOUR EXPOSURE
TO 1200 F HELIUM AND SODIUM (50 ppm O₂)
PHASE I, Task 1

R-2349

* FROM USS BULLETIN "STEELS FOR ELEVATED TEMP. SERVICE"

FIG. 4 - DUCTILITY OF 304 ss 15 mil and 60 mil SPECIMENS
AFTER 500-HOUR EXPOSURE
TO 1200 F HELIUM AND SODIUM (50 ppm O₂)
PHASE I, Task 1

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R-2350

* FROM USS BULLETIN, "STEELS FOR ELEVATED TEMP. SERVICE"

FIG. 5 - 0.2% OFFSET YIELD STRENGTH OF 316 SS
15 mil and 60 mil SPECIMENS AFTER 500-HOUR EXPOSURE
TO 1200 F HELIUM AND SODIUM (50 ppm O₂)
PHASE I, Task 1

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* FROM USS BULLETIN, (STEELS FOR ELEVATED TEMPERATURE SERVICE)

FIG. 6 - TENSILE STRENGTH OF 316 ss 15 mil and 60 mil SPECIMENS
AFTER 500-HOUR EXPOSURE
TO 1200 F HELIUM AND SODIUM (50 ppm O₂)
PHASE I, TASK 1

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* FROM USS BULLETIN "STEELS FOR ELEVATED TEMP. SERVICE"

FIG. 7 - DUCTILITY OF 316 ss 15 mil and 60 mil SPECIMENS
AFTER 500-HOUR EXPOSURE
TO 1200 F HELIUM AND SODIUM (50 ppm O₂)
PHASE I, Task 1

1000 and 1200 F, and thence increases with increasing test temperature as carbide dissolution occurs. In this respect, it is a mirror image of yield strength.

High Oxygen Sodium Excursion

Analysis of the data from the high oxygen sodium-exposed specimens shows a distinct deviation from that data obtained with the helium-exposed specimens. As had been defined in previous work¹, oxidation effects are limited to the surface. As such, they have a greater effect on those properties which are influenced by the surface or by the surface-to-volume ratio. Comparing the data, the tensile strengths are reasonably consistent between the two exposure systems. This is to be expected since tensile strength is primarily a volume property. The yield strength, however, is controlled somewhat by the surface, and the data show that sodium-exposed materials have yield strength slightly higher than that of the helium-exposed materials at all test temperatures. The difference is small. The controlling influence in all of these specimens as far as strength is concerned is the carbide distribution and location. The surface embrittlement due to oxidation is only an add-on feature.

Ductility exhibits a significantly different response to the oxidation than does strength. On a broad, general basis, it can be observed that sodium-exposed specimens always have lower ductilities than commensurate helium-exposed specimens. The effect is more pronounced in elongation than in reduction of area and in the 15 mil than in the 60 mil specimen.

The value of elongation is primarily derived from the uniform extension of the specimens during loading. The elongation occurring during necking is only a small contributor to the over-all value. Any surface hardening has a pronounced effect on the ability of the material to elongate during the initial stages of loading. Thus, this hardening (oxidation) is more observable in elongation values than it is in other ductility measurements.

The value of reduction of area is associated with the necking of the specimen after maximum load has been achieved. Materials which have a brittle case usually initiate failure by cracking in this case. The crack continues to propagate until sufficient deformation occurs at the root of the crack that work-hardening overcomes the stress and the crack stops. From that point to final failure, the behavior is ductile. The effect of surface hardening on reducing the value of reduction of area is

1. Topical Report No. 5, MSAR 66-78, and Topical Report No. 8, MSAR 67-103.

directly proportional to the ratio of the hardened material to the total thickness. In the specimens in question, this surface effect is to only a shallow depth and, as a consequence, the effect on the value of reduction of area is small compared to the effect on elongation.

All of the above observations can, in turn, be applied to explaining the more pronounced effect of oxidation on the ductility in 15 mil specimens than in 60 mil specimens. The 60 mil materials with the larger unaffected-to-affected material ratio show considerably less decrease in ductility due to the oxidation than do the thinner materials. In making these observations, it must also be noted that the thicker materials make a greater recovery of their ductility as temperature is increased beyond the sensitization range. Thus, at 1400 F, the 60 mil material shows less disparity between helium and sodium-exposed specimens than do the 15 mil specimens.

In analyzing the 1400 F data, with comparison to that of 1200 F, it is possible to observe the effects of the two embrittling phenomena, -- sensitization and oxidation. The helium-exposed specimens which are only undergoing sensitization embrittlement show recovery of ductility values at 1400 F to equal or greater than that observed at room temperature. The oxidized specimens, although they show a recovery of some ductility on a percentage basis, it is considerably smaller than that achieved by the helium specimens, and is a relative measure of the embrittling effect of the oxidation. Comparing the recovery of 60 mil specimens with that of 15 mil specimens adequately demonstrates the more profound effect of surface embrittling phenomenon on the thinner wall materials. It would appear that some emphasis could be made to this point to caution against the indiscriminate use of mechanical property data out of sodium for all material thicknesses.

Metallographic analyses following the tensile tests of the high oxygen-exposed specimens and accompanying 5 mil tabs show effects in the form of a thin surface scale with additional penetration of the oxide into the grain boundaries. The following table compares the depths of penetration of this scale:

Penetration of Oxide Scale

<u>Material</u>	<u>Material Thickness, mils</u>	<u>Average Penetration, μ</u>	<u>Maximum Penetration μ</u>
304	5	15	-
304	15	15	25
304	60	15	25
316	15	10	15
316	60	10	15

This shows that the degree of penetration of the oxide is independent of specimen thickness whether it be 15 mil or 60 mil and that the penetration into the 304 ss is greater than into the 316 ss. In both the helium-exposed and high oxygen sodium-exposed specimens, the carbide precipitation is well defined. These analyses support the two effects observed in the tensile tests, -- one due to carbide sensitization; and the second, to the surface oxidation.

To ascertain whether carbon was influencing the test results, carbon analyses were made of the 5 mil 304 ss monitoring tabs from each of the excursion units. Representative test specimens were also analyzed. These results are given in the following table.

304 ss Carbon Analyses Results
(500-hour Excursion Units - 1200 F)

<u>Excursion Dates</u>	<u>Excursion Unit</u>	<u>Tab or Spec. No.</u>	<u>Material Thickness, mil</u>	<u>Excursion Time, hrs</u>	<u>Final Carbon, ppm</u>	<u>Original Carbon, ppm</u>
1/9-1/29	1	4-295	5	500	412	545
1/9-1/29	1	L-18	15	500	666	667
1/9-1/29	1	A-20	60	500	625	615
1/11-2/1	2	4-292	5	502	2832	545
1/11-2/1	2	L-8	15	502	1708	667
1/11-2/1	2	A-14	60	502	878	615

As seen in the 5 mil tab data, there was a carburizing environment present in Excursion Unit No. 2, the source of which could not be determined. Analyses of test specimens from this unit showed only a slight degree of carburization and the effects, if any, are not apparent in the tensile data.

Summary

In summary, it can be stated that the test material is typical of standard heats of 304 and 316 ss and that the material responded in a normal manner when thermally aged, there being a slight increase in tensile and yield strengths with corresponding loss in ductility from carbide sensitization of both materials. The high oxygen-sodium excursion caused a deviation from the data of the helium thermally-aged specimen results. There was little change in tensile strength. The yield strength showed small increases in the sodium-exposed tests. In all cases, the controlling influence on the strengths appeared to be the carbide location and distribution.

Ductility exhibits a significantly different response to oxidation than do the strength properties. It was observed that the sodium-exposed specimens always have lower ductilities than commensurate helium-exposed specimens. The effect is more pronounced in elongation than in reduction of area and in 15 mil than in 60 mil specimens.

Analyses of the 1400 F tensile data make possible the separation of the two observed embrittling phenomena, -- sensitization and oxidation. The helium-exposed specimens show recovery of ductility values, while the oxidized sodium-exposed show only partial recovery. Comparison of this recovery between the 15 mil and 60 mil specimens shows that care has to be taken in the indiscriminate use of mechanical property data from sodium for all material thicknesses.

Metallographic analyses of the oxidized specimens show no difference in the degree of oxidation between the 15 and 60 mil specimens and support the two observed effects (sensitization and oxidation) noted in tensile-test data. In some instances, random carburization took place but to such a small degree that it did not appear to influence the test results.

1400 F Excursion

The temperature of Loop 2 was slowly increased to 1400 F, following the 1200 F excursion, while all other conditions were maintained similarly to those during the 1200 F excursion, -- this being a dynamic sodium system with a minimum ΔT of 50 F and a sodium oxide content of 50 ppm.

Once these conditions were attained, Loop 2 was charged with a number of specimens equivalent to that of the 1200 F excursion. These specimens again were a combination of 304 and 316 ss of 15 and 60 mil thickness and from the same heats used in the earlier excursions.

On February 20 and 21, 1968, the 1400 F-500 hour high oxygen sodium excursion was initiated. During the course of this excursion, three failures occurred in bellows-sealed valves. (The valves are Powell Fig. 2475, 1/2 in. IPS with thin 2-ply bellows.) While it is unusual to experience this many failures in a 500-hour period, other similar failures have occurred during the previous 20,000 hours of loop operations. All the valves had seen prior service during the 1200 F excursion and, therefore, it would be impossible to say that the failures were initiated by the sodium conditions imposed on the valves by this 1400 F excursion.

On February 22, the first failure of a bellows-sealed sodium valve occurred. This necessitated draining the main system after isolating sodium flow through all the exposure units. The

units were held at 1400 F under static conditions for ~40 hours until the faulty valve was replaced and operating conditions were regained. Flow was then established through the exposure units and the excursion continued. On March 6, the excursion in No. 3 unit was terminated prematurely after 332 hours of exposure when a second bellows-sealed valve failed. On March 10, a third valve failure occurred on No. 5 unit, necessitating the isolation of flow through this unit. The final 67 hours of the excursion were completed under static sodium conditions. Figure 8, with included table, provides the pertinent flow and temperature-data averages during the course of the excursion period which ended March 13, 1968.

Sodium samples were extracted from the system during the excursion period and analyzed for various constituents. The results of these analyses and saturation temperatures determined by the plugging indicator are listed in the following table.

Date	P.I. Results		Analytical Determinations, ppm			
	Sat. Temp. °F	Equivalent O ₂ Content (1) ppm	O ₂	C	H ₂	Off-gas
2/22	510	55	(2)	-	22	-
2/26	510	55	(2)	-	-	-
2/29	-	-	-	29	-	-
3/1	515	58	-	-	-	-
3/4	510	55	86	27	-	-
3/11	510	55	-	-	-	-
3/12	515	58	66	-	-	-

(1) From P.I. calibration data - MSAR 68-114, Quarterly Report for October-November-December, 1967.

(2) Samples extracted. Failure in analytical equipment.

Three additional samples extracted during this period that were analyzed by emission spectrography did not reveal any abnormalities in the metallic constituents. The oxygen sample analyzed on March 4 was not in agreement with the P.I. data. Investigation showed that, at the higher operating temperature of 1400 F, there was a large increase in the amount of sodium vapors condensed on the sampling rod during extraction of the sample. This condensed vapor could then be a source of contamination during the transfer of the sample to the analytical glassware.

The carbon activity of the sodium during the excursion period was monitored by analyses of 5 mil 304 ss tabs suspended, on a weekly basis, in the main stream sodium. Results of these analyses are given in the following table.

MSA RESEARCH CORPORATION
EVANS CITY, PA., U.S.A.
APRIL 15, 1968

FLOW - gpm
TEST UNIT 0.45-0.5
COLD TRAP 0.46-0.47
BY PASS 1.8-2.2
TOTAL SYSTEM ~ 5

TEMPERATURES °F
T1 530-535
T2 1400
T3 1330-1350
 ΔT 50-70

FIG. 8 - TEMPERATURE AND FLOW DATA DURING THE 500-HOUR 1400 F EXCURSION
OF 304 ss and 316 ss
15 and 60 mil specimens
PHASE I, Task 1

304 ss 5 mil Carbon Monitoring Tab Results
(Expansion Tank - 1400 F)

<u>Dates</u>	<u>Tab No.</u>	<u>Exposure Time, Hrs</u>	<u>Total Carbon Content, ppm</u>
2/13 - 2/20	4-332	168	368
2/20 - 2/22	4-339	57	364
2/26 - 3/5	4-342	191	334
3/5 - 3/12	4-352	162	560

Original Carbon Content = 545 ppm

Again, all the results indicated that a decarburization of the monitoring tabs took place prior to and during this excursion period with the exception of the tab during the final week of the excursion. This tab showed little change in carbon content. No reason for this apparent change in carbon activity of the sodium could be found.

This excursion period was ended on March 13, 1968, at which time the exposure units were drained, the specimens removed from the units and washed with alcohol and water. They were then stored in a vacuum desiccator until required for mechanical testing.

The 5 mil 304 ss monitoring tabs accompanying the mechanical test specimens in each unit were also analyzed for carbon changes to determine if carburization had taken place in any of the exposure units. The results of these analyses are given in the following table.

304 ss 5 mil Carbon Monitoring Tabs
(Accompanying Tensile Specimens during the 1400 F Excursion)

<u>Dates</u>	<u>Exp. Unit</u>	<u>Tab No.</u>	<u>Exposure Time, Hrs</u>	<u>Total Carbon, ppm</u>
2/20 - 3/12	#1	4-333	500	548
2/20 - 3/12	#2	4-334	500	703
2/21 - 3/13	#4	4-335	500	582
2/21 - 3/13	#5	4-336	500	520

Original Carbon = 545 ± 50

While the tabs from the expansion tank showed decarburi-
zation throughout most of the excursion period, these tabs showed little change in carbon content with the exception of the tab from the exposure unit No. 2. This tab indicated that a slight amount of carburization had taken place. No explanation for this difference can be made at this time.

Test specimens from this excursion were tensile tested at room temperature, 1200 F and 1400 F. The results of these tests are given in Table 7 (helium exposed) and Table 8 (high oxygen sodium exposed).

Figures 9, 10 and 11 graphically compare the results of the tensile tests of 304 ss for as-received material (see Table 4, page 14, under "1200 F Excursion"); for 1400 F helium exposed material; and for 1400 F high oxygen sodium-exposed material. Figures 12, 13 and 14 make the same comparison for the 316 ss data. In each figure, typical 304 and 316 ss tensile data are given as published in the United States Steel Bulletin, "Steels for Elevated Temperature Service."

Discussion of Results of 500-hour 1400 F Excursion of 304 and 316 ss

As-received

Comparison of this data was discussed under the 1200 F excursion (see Page 13).

Helium-exposed

Normal thermal effects, similar to those shown during the 1200 F excursion, were again demonstrated in the 1400 F helium exposed tensile tests. The tensile strength decreased with increasing test temperatures. There was a similar decrease in the yield strength with increasing test temperatures. The ductility is at the minimum during the 1200 F tests, which is normal for this type of material where the carbide precipitation causes loss in ductility between 800 and 1200 F, and then carbide dissolution causes an increase in ductility above 1200 F. All material, regardless of thickness (15 mil or 60 mil) and regardless of the type of material (304 or 316), reacted in a similar fashion.

High oxygen-exposed

Comparison of data from the high oxygen-exposed 60 mil specimens with the data from helium-exposed specimens shows the strengths are essentially equivalent at all test temperatures. The ductility data, however, show it is equivalent at room temperature with significant deviation at 1200 F and a lessening of the difference at 1400 F where some recovery in ductility occurs. The lower

TABLE 7 - 304 and 316 ss TENSILE DATA - 500-HOUR HELIUM EXPOSED AT 1400 F
PHASE I, TASK 1

Spec. No.	Material	Nominal Thickness, In.	Test Temp., °F	Yield Strength, psi	Average Y.S., psi	Tensile Strength, psi	Average T.S., psi	Elong. %	Average Elong., %	Reduction of Area, %
A136	304	0.060	R.T.	37,130	37,900	87,670	88,000	62	61	66
A137	"	"	R.T.	38,670		88,330		60		63
A138	"	"	1200	21,380	21,525	45,170	44,420	43	45	65
A139	"	"	1200	21,670		43,670		47		63
A140	"	"	1400	19,000	18,000	28,130	26,900	68	66	63
A141	"	"	1400	17,000		25,670		64		67
L86	304	0.015	R.T.	36,960	40,270	93,040	93,260	68	67	54
L87	"	"	R.T.	40,580		93,480		66		54
L88	"	"	1200	20,010	20,150	42,750	38,620	55	53	51
L89	"	"	1200	20,290		34,490		50		43
L90	"	"	1400	19,050	19,235	25,790	26,780	63	63	52
L91	"	"	1400	19,420		27,770		63		52
B126	316	0.060	R.T.	41,660	41,595	94,160	94,455	45	45	55
B127	"	"	R.T.	41,530		94,750		45		50
B128	"	"	1200	24,670	25,000	52,000	53,000	49	47	60
B129	"	"	1200	25,330		54,000		45		57
B130	"	"	1400	22,000	24,335	30,670	32,000	78	76	70
B131	"	"	1400	26,670		33,330		73		73
M87	316	0.015	R.T.	40,580	41,885	93,910	93,910	44	45	44
M88	"	"	R.T.	43,190		93,910		46		45
M89	"	"	1200	25,360	24,275	47,830	47,830	38	43	39
M90	"	"	1200	23,190		47,830		47		51
M91	"	"	1400	23,290	23,465	36,860	35,265	59	60	51
M92	"	"	1400	23,640		33,670		60		52

TABLE 8 - 304 and 316 ss TENSILE DATA - 500-HOUR HIGH OXYGEN SODIUM EXPOSED AT 1400 F
PHASE I, TASK 1

Spec. No.	Material	Nominal Thickness, In.	Test Temp., °F	Yield Strength, psi	Average Y.S., psi	Tensile Strength, psi	Average T.S., psi	Elong. %	Average Elong. %	Reduction of Area, %
A112	304	0.060	R.T.	38,300	37,605	90,170	89,715	63	62	63
A120	"	"	R.T.	36,910		89,260		60		62
A106	"	"	1200	21,720	22,195	44,140	43,735	38	39	59
A121	"	"	1200	22,670		43,330		40		53
A107	"	"	1400	17,170	17,920	23,000	24,835	63	60	58
A113	"	"	1400	18,670		26,670		56		62
L64	304	0.015	R.T.	32,320	32,825	84,780	85,505	63	63	52
L78	"	"	R.T.	33,330		86,230		63		52
L72	"	"	1200	20,290	19,470	34,930	35,910	33	34	30
L79	"	"	1200	18,650		36,890		35		36
L65	"	"	1400	16,220	16,680	22,980	22,635	33	36	39
L73	"	"	1400	17,140		22,290		38		37
B104	316	0.060	R.T.	40,400	41,865	92,930	93,550	45	45	52
B134	"	"	R.T.	43,330		94,170		45		51
B113	"	"	1200	25,520	25,095	49,650	50,825	45	44	59
B135	"	"	1200	24,670		52,000		43		53
B105	"	"	1400	21,480	21,420	29,700	30,750	63	63	63
B114	"	"	1400	21,360		31,800		62		63
M79	316	"	R.T.	39,860	41,305	89,130	88,380	40	40	39
M95	"	"	R.T.	42,750		87,630		40		46
M71	"	"	1200	22,460	22,825	52,170	48,985	30	30	30
M96	"	"	1200	23,190		45,800		30		36
M72	"	"	1400	22,460	19,925	30,430	29,710	38	38	29
M80	"	"	1400	17,390		28,990		38		36

* FROM USS BULLETIN "STEELS FOR ELEVATED TEMPERATURE SERVICE"

Fig. 9- 0.2% Offset Yield Strength of 304SS - 15 Mil and 60 Mil Specimens after 500 Hours Exposure to 1400F Helium and Na(50ppm O₂).

Phase I, Task I

TENSILE TEST TEMPERATURE

* FROM USS BULLETIN "STEELS FOR ELEVATED TEMPERATURE SERVICE"

Fig. 10 - Tensile Strength of 304SS-15 Mil and 60 Mil Specimens
 after 500 Hours Exposure to 1400F Helium and Na(50 ppm O₂)

Phase I, Task I

TENSILE TEST TEMPERATURE

FROM USS BULLETIN "STEELS FOR ELEVATED TEMPERATURE SERVICE"

Fig. 11 - Ductility of 304SS - 15 Mil and 60 Mil Specimens after 500 Hours Exposure to 1400F Helium and Na(50ppm O₂).

Phase I, Task I

* FROM USS BULLETIN "STEELS FOR ELEVATED TEMPERATURE SERVICE"

FIG. 12 - 0.2% OFFSET YIELD STRENGTH OF 316 SS -
 15 MIL AND 60 MIL SPECIMENS AFTER 500 HOURS OF EXPOSURE
 TO 1400 F HELIUM AND Na (50 ppm O₂)
 PHASE I, TASK 1

TENSILE TEST TEMPERATURE

* FROM USS BULLETIN "STEELS FOR ELEVATED TEMPERATURE SERVICE"

Fig. 13—Tensile Strength of 316SS— 15 Mil and 60 Mil Specimens after 500 Hours Exposure to 1400F Helium and Na(50ppm O₂).

Phase I, Task I

* FROM USS BULLETIN "STEELS FOR ELEVATED TEMPERATURE SERVICE"

Fig. 14-Ductility of 316SS— 15 Mil and 60 Mil Specimens
 after 500 Hours Exposure to 1400F Helium and
 Na(50ppm O₂).

Phase I, Task I

ductility for the sodium-exposed specimens is not so surprising insomuch as the oxygen absorption on the surface must be considered an embrittling phenomenon. What is interesting is that there is no difference in yield strength data and only a slight difference in tensile strength data. It would be expected that the embrittling oxidation would increase the yield strength above that of the helium-exposed specimens. However, since this did not occur, a more complex mechanism must be involved. While metallography (discussed in a latter section) did indicate decarburization, -- which is a softening mechanism that would counteract the oxidation-strengthening mechanism -- during the 1400 F excursion, it does not explain the variations in ductilities. The loss of carbon should cause increases in ductilities rather than the loss of ductilities, as noted. Further evaluation of the specimens will be necessary to more fully describe the oxidation mechanisms taking place during the 1400 F excursions and the morphological changes that occur which have a direct bearing on the mechanical properties. There is microscopic evidence that sigma is forming in the carbon-depleted areas but, due to the small amount formed, X-ray diffraction could not detect it.

The 15 mil specimens follow the same pattern set by the 60 mil specimens; however, due to the thinner cross-section, the differences noted in all the tensile data are much more evident.

The appearance of the specimens removed from the 1400 F excursion differed significantly in appearance from those of the 1200 F excursion. On these specimens, a green rather than a gray surface coloration existed. X-ray diffraction of the surface shows that both scales have the same crystal structure and similar lattice parameters. The only significant difference in each X-ray pattern is in the lines from the metal phase. The X-ray patterns from the 1200 F gray scale show sodium chromite lines along with those for alpha iron. The gamma lines are very weak and, in most cases, non-existent. The green, 1400 F scale, however, shows very weak alpha lines with an intense, complete gamma pattern.

X-ray fluorescent analyses were run on the scaled surface of two typical test specimens, -- one from 1200 F and one from 1400 F exposure. These analyses were strictly qualitative since the scale in both cases was not thick enough to prevent excitation of the underlying base metal. The analyses did show the greater concentration of chromium in the green scale, as indicated by the color. The reflections from the 1200 F sample show an Fe/Cr ratio of $K_{\alpha 1}$ peak heights of 3 to 1. With the 1400 F sample, the $K_{\alpha 1}$ peak height ratio for Fe/Cr is only 1.3 to 1.

Metallographic investigation of the 1400 F samples shows a thin oxide film fairly continuously, with no sub-surface grain boundary oxide penetration normally seen in the 1200 F excursion samples. The small ferritic iron islands characteristic at 1200 F are missing at 1400 F. The question arises as to whether the differences are strictly thermal or whether they are due to an

increase in surface erosion which does not allow time for grain boundary penetration. Measurements of specimens before and after the 1400 F excursion show no measurable change in thickness.

On the basis of the limited data, the most plausible explanation seems to lie with enhanced iron transport at the 1400 F excursion temperatures. The absence of the ferritic iron islands in the green scale and the greater concentration of chromium in the scale strongly support this. The absence of grain boundary penetration of oxide can be attributed to either or both of the following phenomenon.

First, at these temperatures, a more perfect scale may develop which limits oxygen diffusion to the stainless steel surface. Second, a higher temperature may allow a greater solubility and greater diffusivity of the oxygen into the metal structure. Metallographic evidence shows that the second mechanism is operative; whether it is joined by the first is not certain. Figure 15 shows the surface of a 60 mil tensile specimen after 500 hours at 1400 F in Loop 2. In the unetched condition, the thin surface scale is visible, with some minor evidence of penetration into the grain boundaries. Upon etching (see Figure 16), an artifact is developed at the grain boundaries which is indicative of a hardening process close to these boundaries. Since no carbide precipitation is evident, the conclusion to be drawn is that this hardening is due to oxygen dissolved into the lattice at those points.

Summary

The metallographic results show a change in mechanism of oxidation of stainless steel in high oxygen sodium at 1400 F from that which has heretofore been observed at 1200 F. In addition to this change, the higher temperature results in a minimization of carbide sensitization which also is a prominent factor at 1200 F. The properties reflect these variations in behavior due to the higher temperature. In addition, some scatter was observed which is greater than had previously been encountered. Much of this can be due to the fact that the cool-down period for each specimen may vary somewhat, particularly with respect to the time of passage from 1300 F to 1100 F where carbide sensitization would reoccur. Those specimens tested at 1200 F encountered re-precipitation of carbide, the magnitude of which will vary with the time at temperature prior to the initiation of testing.

The materials, for the most part, however, follow an accepted pattern, with the 1400 F high oxygen-exposed specimens having lower strengths and higher ductilities than the helium-exposed specimens.

There is some evidence of surface decarburization, as seen in Figure 16 and confirmed by carbon analyses of specimens, which more than offsets the strengthening effect of the dissolved oxygen,

Unetched

X533

FIG. 15 - SURFACE SCALE AND SCALE PENETRATION
AFTER 500 HOURS IN LOOP 2 AT 1400 F
Specimen A112

Etched

X533

FIG. 16 - MICROSTRUCTURE OF SPECIMEN
AFTER 500 HOURS IN LOOP 2 AT 1400 F
SHOWING SURFACE OXYGEN EFFECTS
Specimen A112

as evidenced by the lower strengths of the sodium-exposed specimens than of the helium-exposed specimens. This is not the complete story, however, since ductility differences are not fully amenable with softening due to decarburization. The oxidation effects on the 15 mil specimens are quite evident, while the 60 mil specimens reflect only the thermal-aging effects.

A direct comparison of the tensile data from the 1200 F and 1400 F excursions is not truly valid. The change in oxidation mechanisms in combination with a diminution of carbide sensitization produced a marked softening during the 1400 F excursion. It is quite probable, however, that much of the strength and ductility changes occurring during the 1400 F excursion could be recovered by re-aging at 1200 F to regain the effect of carbide precipitation. At 1400 F, there appears to be an enhanced mass transfer of iron and there is evidence of decarburization. Additional effects must be present, however, since the ductility and strength values at 1400 F are not fully consistent with microstructural changes defined to date.

Future Work Scheduled Under Phase I, Task 1

Additional testing of 304 and 316 ss specimens from both the 1200 F and 1400 F excursions is to be initiated in the near future. This work will be creep-rupture testing in sodium under parameters of high velocity (20 ft/sec), ΔT (~ 150 F), and low oxygen (< 10 ppm). These additional tests will complete the work statements under Phase I, Task 1, of the program. A topical report will summarize all future data generated, as well as the data discussed in this report. Loop 2 will then be used to enter other phases of the program, such as high oxygen excursions of vanadium-base alloys, modified austenitics and special alloys.

PHASE I, TASK 2: HIGH CARBON EXCURSIONS OF 304 and 316 SS

1200 F Excursions - 15 mil Specimens

MSAR's previous carburization work with 304 ss and 316 ss was conducted in a sodium system in which there was an extremely high carbon activity and exposure times were as long as 4000 hours.² In these tests, the heavily carburized materials experienced almost total loss of ductility. This portion of the program is directed towards more controlled rates to better define the effects of more moderate carburized levels. The basic test in this task is to conduct tests on materials carburized to depths of 5-10 mils in a 60-mil specimen. Meanwhile, this 'allowable' degree of carburization of thinner specimens became a point of discussion. MSAR agreed to carburize 15 mil specimens to values up to 0.5% and to evaluate the effect on mechanical strength. Efforts during this report period were therefore directed towards obtaining a carbon activity in Loop 1 to where a 15 mil specimen would carburize to a total carbon content of 0.3 to 0.4% total carbon in 100 hours.

To reach this carburizing potential, Loop 1 was cold-trapped continuously for an extended period of time without flow through the carbon bed. The carbon activity was monitored by use of the 5 mil 304 ss tabs described under Phase I, Task 1, of this report. The results of the analyses of these tabs are given in the following table:

5 mil 304 ss Carbon Monitoring Tab Results
(Instream Expansion Tank - 1200 F)

<u>Dates</u>	<u>Tab No.</u>	<u>Exposure Time, Hrs</u>	<u>Total Carbon, ppm</u>
12/20 - 12/25	4-294	120	4210
12/26 - 1/2	4-291	168	4040
1/2 - 1/9	4-301	161	3800

Original Carbon = 545 ppm

It was believed that this carbon activity would produce the desired carburization of the 15 mil tabs. To verify this, a combination of 15 mil 304 and 316 ss specimens were exposed in three separate units. Between January 15 and January 23, 1968, excursions of 48, 96 and 166 hours were conducted. The pertinent flow and temperature data during this period are shown in Figure 17.

2. Topical Report No. 4, MSAR 65-194; and Topical Report No. 8, MSAR 67-103.

FLOW RATES GPM	
CARBON BED	0.0-0.25
UNIT 1-3	0.5
COLD TRAP	0.7
BY PASS	3.3
TOTAL	4.75

T1	1150°F MAX
T2	1200°F ± 5°
T3	250°F ± 10°
T4	1100°- 1200°F

FIG. 17 - AVERAGED LOOP CONDITIONS DURING 1200 F HIGH CARBON EXCURSION
OF 15 mil 304 and 316 ss SPECIMENS
CONDUCTED BETWEEN JANUARY 15 and 23, 1968
PHASE 1, Task 2

Analyses of the 15 mil test specimens and the 5 mil monitoring tab accompanying the test specimens during this first excursion revealed that the level of carburization (~ 1500 ppm) was less than had been anticipated. Therefore, a second and third excursion (100-hour duration each) were conducted, each time with an increased carburizing potential. These changes were accomplished by flowing through the carbon bed between 1100 and 1200 F and with a flow rate between 0 and $1/4$ gpm. The second excursion was conducted between February 29 and March 4, 1968. During this period, the 5 mil 304 ss weekly (~ 160 hour) monitoring tab indicated a total carbon content of 6835 ppm with the test specimens achieving a level of approximately 1800 ppm.

The third excursion took place between March 18 and March 23, 1968. The monitoring tab analysis indicated a total carbon content of 8000 ppm with the 15 mil specimens reaching a level of 3000 ppm.

Sodium samples were being removed periodically throughout these excursions. Table 9 lists the results of the various analytical determinations made during these periods.

Sodium samples from Loop 1, as well as Loop 2 and the Static Retorts, were analyzed for metallic impurities by emission spectrography and, except for calcium values, did not reveal any abnormalities. All samples show relatively high calcium (8-12 ppm) concentrations. The samples were taken at different times but were prepared and analyzed at the same time. Since there is no logical reason for the calcium concentration to increase in all these isolated systems, it was concluded that contamination of the sample occurred either during preparation or during the analyses.

The carburizing potential of Loop 1 sodium is monitored on a weekly basis by exposing a 5 mil 304 ss tab in the instream sump tank and then analyzing for change in total carbon content. The results of these analyses are given in Table 10 for the period during which the three excursions were conducted.

Test Results

Following each excursion, the test specimens were removed from the exposure units, thoroughly washed with alcohol and water, and stored for subsequent tensile tests in air from room temperature to 1400 F. During the final excursion, a similar combination of test specimens were exposed to a helium atmosphere and subsequently tensile tested to provide data on thermal aging effects. All the tensile data from these tests is summarized in Tables 11, 12 and 13. The data is shown graphically in Figures 18, 19 and 20, where a comparison is made between the as-received material, the helium-exposed material, and the material that has undergone the three high carbon sodium excursions. Typical 304 and 316 ss tensile data is given in each figure as published in the United States Steel Bulletin, "Steels for Elevated Temperature Service."

TABLE 9 - LOOP 1 SODIUM ANALYSES RESULTS
DURING CARBON EXCURSIONS 1, 2 AND 3

Dates	P.I. Runs		Analytical Determinations, ppm				
	Saturation Temp., °F	Equivalent O ₂ Content (1)	O ₂	C	H ₂	C ₂ H ₂	CO ₂
1/15	-	-	-	41	10	1.7	960
1/16	-	-	-	-	-	-	-
1/17	<300	<10	33	-	-	-	-
1/22	<300	<10	-	-	-	-	-
1/24	<300	<10	<10	-	-	-	-
1/25	-	-	-	68	-	-	-
1/29	<300	<10	<10	-	-	-	-
2/1	-	-	-	49	-	2.0	210
2/5	-	-	-	70	-	-	-
2/9	300	<10	<10	-	-	-	-
2/12	300	<10	<10	-	-	-	-
2/23	300	<10	<10	-	13	-	-
2/27	<300	<10	28	22	-	-	-
2/28	-	-	-	-	-	2.0	9.2
3/1	-	-	-	-	-	3.8	12.6
3/5	<300	<10	<10	-	-	2.5	132
3/6	-	-	-	48	-	5.8*	11.0*
3/13	-	-	-	-	-	2.9	366
3/14	<300	<10	<10	-	-	2.4*	4.9*
3/20	300	<10	12	-	-	-	-

(1) MSAR Loop 2 Calibration Curve, MSAR 68-14.

* Values determined from filtered sample. All other values determined on unfiltered bucket samples.

TABLE 10 - WEEKLY 5 MIL 304 SS CARBON MONITORING TAB RESULTS
(Instream Expansion Tank - 1200 F)

<u>Dates</u>	<u>Tab No.</u>	<u>Exposure Time, Hrs</u>	<u>Total Carbon, ppm</u>
1/9 - 1/17	4-305	165	3161
1/17 - 1/24	4-313	168	2777
1/24 - 1/29	4-318	114	2015
1/29 - 2/5	4-322	162	7766
2/5 - 2/12	4-326	164	5943
2/12 - 2/19	4-331	166	5070
2/19 - 2/26	4-338	167	10550
2/26 - 3/4	4-341	169	6835
3/4 - 3/11	4-349	161	8002
3/11 - 3/18	4-355	166	9487
3/18 - 3/23	4-356	125	8004

Original Carbon Content = 545 ppm

TABLE 11 - 304 and 316 ss TENSILE DATA - FIRST HIGH CARBON SODIUM EXCURSION AT 1200 F
15 mil Specimens
Phase I, Task 2

Specimen No.	Material	Exposure Time, Hrs	Test Temp., °F	Yield Strength, psi	Average Y.S., psi	Tensile Strength, psi	Average T.S., psi	Elong. %	Average Elong. %	Reduction of Area, %
L-46	304	48	R.T.	45,710	44,285	95,710	96,440	38	44	39
L-47	304	48	R.T.	42,860		97,170		50		41
L-52	304	96	R.T.	46,430	46,500	93,570	93,285	28	28	37
L-53	304	96	R.T.	46,570		93,000		28		37
L-40	304	166	R.T.	42,860	45,645	90,290	88,000	37	30	40
L-41	304	166	R.T.	48,430		85,710		22		36
L-48	304	48	1200	24,290	24,075	48,710	47,570	37	40	40
L-49	304	48	1200	23,860		46,430		43		40
L-54	304	96	1200	27,110	26,600	47,100	44,855	30	31	37
L-55	304	96	1200	26,090		42,610		31		37
L-42	304	166	1200	27,860	25,860	49,430	45,430	31	36	39
L-43	304	166	1200	23,860		41,430		40		48
M-46	316	48	R.T.	48,570	47,285	92,710	90,640	30	31	37
M-47	316	48	R.T.	46,000		88,570		31		37
M-39	316	96	R.T.	53,570	56,785	93,190	97,665	22	23	33
M-40	316	96	R.T.	60,000		102,140		23		33
M-52	316	166	R.T.	54,000	48,430	102,000	99,785	30	28	37
M-53	316	166	R.T.	42,860		97,570		26		37
M-48	316	48	1200	27,570	29,715	56,710	57,785	36	34	40
M-49	316	48	1200	31,860		58,860		31		38
M-42	316	96	1200	29,710	29,997	57,240	55,190	30	33	37
M-43	316	96	1200	30,285		53,140		36		38
M-54	316	166	1200	32,860	33,785	51,000	52,285	32	33	46
M-55	316	166	1200	34,710		53,570		34		39

TABLE 12 - 304 and 316 ss TENSILE DATA - SECOND HIGH CARBON SODIUM EXCURSION
 FOR 100 HOURS AT 1200 F
 15 ml Specimens
 Phase I, Task 2

Specimen No.	Material	Test Temp. °F	Yield Strength, psi	Average Y.S., psi	Tensile Strength, psi	Average T.S., psi	Elong. %	Average Elong. %	Reduction of Area, %
L-101	304	R.T.	50,000	50,360	92,750	94,925	28	31	43
L-103	304	R.T.	50,720		97,100		34		45
L-104	304	1200	26,350	27,450	46,350	46,220	27	28	42
L-105	304	1200	28,550		46,090		28		37
L-106	304	1400	21,350	21,545	29,190	28,725	35	37	49
L-107	304	1400	21,740		28,260		39		39
M-103	316	R.T.	56,250	57,110	99,060	98,445	17	17	28
M-104	316	R.T.	57,970		97,830		16		29
M-105	316	1200	36,230	36,475	54,060	57,905	24	22	36
M-106	316	1200	36,720		61,750		20		(1)
M-107	316	1400	32,320	29,930	39,570	37,030	40	38	45
M-108	316	1400	27,540		34,490		35		(1)

(1) Broke outside gage mark.

TABLE 13 - 304 and 316 TENSILE DATA - THIRD HIGH CARBON SODIUM AND HELIUM EXCURSION
FOR 100 HOURS AT 1200 F - 15 mil Specimens
Phase I, Task 2

Specimen No.	Material	Exposure Environment	Test Temperature	Yield Strength, psi	Average Y.S., psi	Tensile Strength, psi	Average T.S., psi	Elong. %	Average Elong. %	Reduction of Area, %
L-114	304	He	R.T.	39,400	42,035	95,270	95,545	64	64	50
L-115	304	He	R.T.	44,670		95,820		63		52
L-116	304	He	1200	24,640	24,465	42,750	44,445	45	42	43
L-117	304	He	1200	24,290		46,140		38		43
L-108	304	Na	R.T.	62,860	63,710	88,570	92,350	5	7	24
L-109	304	Na	R.T.	64,560		96,130		9		25
L-110	304	Na	1200	33,810	34,050	56,110	56,555	15	17	19
L-111	304	Na	1200	34,290		57,000		19		29
L-112	304	Na	1400	29,450	28,715	36,400	35,990	22	20	35
L-113	304	Na	1400	27,980		35,580		18		28
M-113	316	Na	R.T.	68,150	71,220	103,300	105,650	11	11	24
M-114	316	Na	R.T.	74,290		108,000		10		24
M-115	316	Na	1200	38,240	41,060	67,820	64,845	16	18	27
M-116	316	Na	1200	43,880		61,870		19		28
M-117	316	Na	1400	36,590	37,805	45,340	45,410	20	20	28
M-118	316	Na	1400	39,020		45,480		20		35
M-109	316	He	R.T.	45,000	44,740	94,000	92,195	50	50	44
M-110	316	He	R.T.	44,480		90,390		50		45
M-111	316	He	1200	25,110	27,240	52,080	51,110	42	41	40
M-112	316	He	1200	29,370		50,140		40		41

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EVANS CITY, PA., U.S.A.
MAY 6, 1968

* FROM USS BULLETIN "STEELS FOR ELEVATED TEMPERATURE SERVICE"
Fig.18-0.2% Offset Yield Strength of 304SS and 316SS
15 Mil Specimens after 100 Hour Excursions in
Helium and High Carbon Sodium at 1200F.

* FROM USS BULLETIN "STEELS FOR ELEVATED TEMPERATURE SERVICE"

Fig. 19 Tensile Strength of 304SS and 316SS 15 Mil Specimens
After 100 Hour Excursions in Helium and High Carbon
at 1200F.

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* FROM USS BULLETIN "STEELS FOR ELEVATED TEMPERATURE SERVICE"
Fig.20 Ductility of 304 and 316SS 15 Mil Specimens after
100 Hour Excursions in Helium and High Carbon
Sodium at 1200F.

Representative test specimens (following tensile testing) and 5 mil monitoring tabs accompanying the test specimens during the excursion period were analyzed for total carbon content for each of the three high carbon sodium excursions. The results of these analyses are in Table 14.

These carbon content values were then averaged and the tensile properties at the three different test temperatures (room temperature, 1200 F and 1400 F) were plotted as a function of carbon content, as shown in Figures 21, 22 and 23.

Discussion of Results

As-Received and Helium Exposed

This discussion is found in the foregoing section:
PHASE I, Task 1.

First Excursion (48, 96, and 166 hours)

The carburization data from this excursion indicated that little change would be seen in the tensile properties, which was the case. The carbon contents of the specimens from this excursion ranged between 1000 and 1500 ppm total carbon. There was a slight increase in yield strength, little change in the tensile strength, and slight loss of ductility as noted.

Second Excursion (100 hours)

The carbon content of the specimens from this excursion varied between 1600 and 1900 ppm total carbon and significant changes in the tensile properties were anticipated. There was a large increase in tensile and yield strength for both the 304 and 316 ss materials at all test temperatures. The tensile strengths for both materials showed only slight increases in the room temperature tests and insignificant changes in the elevated temperature tests. The ductility losses in these materials were more pronounced than in the materials from the first excursion, but still showed some ductility (>15%).

Third Excursion (100 hours)

The carbon levels attained during this excursion ranged from 2800 to 3500 ppm total carbon which showed corresponding changes in tensile properties from those of the previous excursions. The surface carbon content of these specimens is quite high, approaching 2 w/o. The visual depth of penetration is between 1.0 and 1.5 mils per side. The microprobe data show enhanced carbon for 8 microns (3 mils) per side. The carbon increase is greater

TABLE 14 - RESULTS OF CARBON ANALYSES OF 304 and 316 SS TEST SPECIMENS
AND ACCOMPANYING MONITORING TABS
PHASE I, Task 2

<u>Excursion Dates</u>	<u>Excursion Unit</u>	<u>Tab or Spec. No.</u>	<u>Material</u>	<u>Material Thickness, mils</u>	<u>Exposure Time, Hrs</u>	<u>Total Carbon, ppm</u>
<u>FIRST EXCURSION</u>						
1/16-1/18	2	4-311	304	5	48	1119
		L-47	304	15	48	983
		M-46	316	15	48	991
1/15-1/18	1	4-310	304	5	96	2068
		L-52	304	15	96	1531
		M-39	316	15	96	1348
1/16-1/23	3	4-312	304	5	166	2211
		L-40	304	15	166	1510
		M-52	316	15	166	1348
<u>SECOND EXCURSION</u>						
2/29-3/4	2	4-30	304	5	100	3432
		L-102	304	15	100	1926
		L-103	304	15	100	1870
		M-103	316	15	100	1615
		M-104	316	15	100	1638
<u>THIRD EXCURSION</u>						
3/19-3/23	5	4-357	304	5	100	5380
		L-108	304	15	100	2854
		L-109	304	15	100	2904
		M-113	316	15	100	2771
		M-114	316	15	100	3486

Original Carbon Content Values: 5 mil 304 ss = 421 ppm
15 mil 304 ss = 615 ppm
15 mil 316 ss = 468 ppm

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FIG. 21 - TENSILE PROPERTIES VS CARBON CONTENT
 0.015 in. Thick 304 and 316 Tensile Specimens Exposed to High
 Carbon Sodium
 Test Temperature: Room Temperature

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FIG. 22 - TENSILE PROPERTIES VS CARBON CONTENT
 0.015 in. Thick 304 and 316 Tensile Specimens
 Exposed to High Carbon Sodium
 Test Temperature: 1200 F

R-2372

FIG. 23 - TENSILE PROPERTIES VS CARBON CONTENT
 0.015 in. Thick 304 and 316 Tensile Specimens
 Exposed to High Carbon Sodium
 Test Temperature: 1400 F

for 316 than for 304, which is consistent with prior work³ where carbon increases in 316 ss are always greater during short exposures (~100 hours).

The effect of carburization on the tensile properties was as expected with higher tensile and yield strengths and lower ductilities.

Summary

It was well demonstrated that different levels of carburization can be obtained in a sodium system. This was done both by varying the time of exposure, as was the case in the first excursion, or by varying the carbon activity of the sodium, as was the case in the second and third excursions.

Figures 18, 19 and 20 compare the data obtained from the as-received material, helium-exposed material, and the three sets of carbon-exposed materials. Due to the limited data, no great weight should be placed on any one test; however, trends can be noted. The most notable and discerning trend is the major loss of ductility with minor increases in carbon contents. Figures 21, 22 and 23 show the change in tensile properties as a function of the total carbon content.

It is obvious that total carbon values of 0.5 w/o in 100 hours are not acceptable. The question arises as to what would be an acceptable level of carbon in sodium. The answer appears to be that there is no acceptable level, -- only a tolerable level. The original approach by AI assumed that an equilibrium is established between the stainless steel and the carbon in the sodium. One equilibrium is already set; that is, the one which exists between the carbon in solution in the austenitic lattice and that in the carbide. If this is upset, more carbide forms until it is re-established. MSAR data show that the chemistry of the carbide will change to maintain the basic equilibrium. Thus, it appears that the only acceptable, continuous carbon level is that which is in equilibrium with the carbon in solid solution in the austenitics. Any higher value results in carbon absorption and precipitation of carbide. The data so far gathered say that there is no new higher carbon activity in sodium acceptable to the stainless under any condition short of complete precipitation of all the chromium.

The data which appear necessary are the interrelation between carbon activity in the sodium, carbon absorption by the stainless, and the time at temperature. These data could define in practical terms the life of cladding or plumbing under the conditions imposed or the deterioration of properties (life) due to a specific carbon excursion. Short-duration carbon excursions could probably be tolerated, but current data do not allow specific definition as to carbon levels or excursion durations.

3. Topical Report No. 4, MSAR 65-194; and Topical Report No. 8, MSAR 67-103.

Future Work Scheduled Under PHASE I, Task 2

Further excursions of 304 and 316 ss material will be done under this phase of the program. A carbon case depth of 5-10 mils will be formed on 60 mil specimens in Loop 1. These specimens will then be tensile tested in air from room temperature to 1400 F and creep-rupture tested in the clean sodium conditions of Loop 3 as outlined under Phase I, Task 1, in the preceding section.

PHASE I, TASK 3: LONG-TERM EXPOSURES OF 304 and 316 ss in
1200 F 'CLEAN' SODIUM IN STATIC RETORTS

The long-term effects of 'clean' sodium on the mechanical properties of 304 and 316 ss are to be determined. The parameters set for these determinations are exposures of 60 mil 304 and 316 ss in a static sodium environment for periods in excess of 10,000 hours, the oxygen content of which is to be maintained at <10 ppm by means of a diffusional-type cold trap. This exposure is then to be followed by creep-rupture testing in 'clean' sodium with test parameters of high velocity (~ 20 ft/sec), test temperature of 1200 F, and system ΔT of 150 F (maximum).

The design of the static retort is shown in Figure 24. Each retort is equipped with four flanged ports through which specimens are exposed and removed, a diffusional-type cold trap for control of the oxygen content, and a sampling port for removal of sodium for analyses. (Four identical retorts were constructed to serve other phases of the program in addition to Phase I, Task 3.)

During the previous report period, Static Retort No. 4 had been charged with ~ 150 lb of sodium, brought to the operating temperature of 1200 F, and was in progress of having the sodium characterized. While characterization was being completed, a helium-gas system was installed to permit simultaneous exposure of canned test specimens in a helium environment with those in the 'clean' sodium environment. These helium-exposed specimens will be used to differentiate thermal-aging effects from corrosion effects. Both the sodium characterization and installation of the gas system have been completed.

The results of the sodium characterization analyses for Static Retort No. 4 are given in the table below:

Static Retort No. 4 Sodium Analyses Results

		Analytical Determinations, ppm							
		<u>O₂</u>	<u>C</u>	<u>H₂</u>	<u>C₂H₂</u>	<u>CO₂</u>	<u>Mass 43</u>		
Sample No. 1		< 10	29	< 2	2	23	4		
Sample No. 2		< 10	48	10	2	24	4		
		<u>Fe</u>	<u>Mg</u>	<u>Cu</u>	<u>Cr</u>	<u>Al</u>	<u>Si</u>	<u>Ti (1)</u>	<u>Ca</u>
Sample No. 1		2	3	2	1	< 1	< 10	200	3
Sample No. 2		4	3	4	3	4	10	120	5

(1) Sampling bucket used in extracting samples is titanium.

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FIG. 24 - MSAR STATIC EXPOSURE RETORT UNIT

All other metallic impurities were below limits of detection.

Two 5 mil 304 ss carbon-monitoring tabs were suspended in the sodium for ~160 hours each. The analytical results of these tabs showed total carbon contents of 477 and 320 ppm total carbon. The original carbon content of these tabs is 545 ppm, which indicates a low carbon activity.

Each of the four specimen ports were charged with ten each of 304 and 316 ss 60 mil specimens for immersion into the sodium, and a combination of the same specimens (totalling seven) in canned helium atmosphere, making totals of 80 specimens (40 each 304 ss and 316 ss) being exposed in the sodium environment and 28 specimens (14 each 304 ss and 316 ss) being exposed in the canned helium environment of Static Retort No. 4.

On February 7, 1968, the long-term excursion (10,000 and longer) of these specimens was initiated and has now exceeded 1260 hours of exposure.

Sodium samples have been removed periodically during this time for various analyses. These results are given in the following table:

Static Retort No. 4 Sodium Analyses Results

<u>Date</u>	<u>Analytical Determinations, ppm</u>				
	<u>O₂</u>	<u>C</u>	<u>H₂</u>	<u>C₂H₂</u>	<u>CO₂</u>
2/6	<10	-	-	-	-
2/7	-	37	-	-	-
2/20	-	-	11	-	-
2/26	<10	-	-	2	151
3/12	-	27	-	-	-
3/14	<10	-	-	-	-
3/22	-	-	12	-	-
3/25	<10	-	-	-	-
3/27	-	-	-	3	74
3/28	-	27	-	-	-

Two samples were taken on February 19 and March 14, 1968, for analyses of metallic impurities. The results of both samples did not show any abnormalities.

This exposure will continue for a minimum of 10,000 hours, at which time a portion of the test specimens will be removed. These specimens will then undergo extensive mechanical property testing as well as metallographical examinations. The mechanical testing will include tensile tests as well as in-stream creep-rupture tests in 'clean' sodium.

PHASE I, TASK 4: 500-HOUR EXCURSION OF MODIFIED AUSTENITICS IN
1200 F and 1400 F HIGH OXYGEN SODIUM AND IN
1200 F HIGH CARBON SODIUM

This part of the program is a duplication of Phase I, Tasks 1 and 2, utilizing modified austenitics being developed at Oak Ridge National Laboratory. Contact has been made with James Weir (ORNL) to obtain a quantity of this material for our test program. To date, we have obtained a quantity of Ti-stabilized 304 ELC in the form of 11-13 mil sheet. Test specimens and corrosion tabs have been made from this material. Some preliminary work in the high oxygen sodium environment of Loop 2 is scheduled for the next report period. However, the latest correspondence with Mr. Weir indicated that medium carbon heats of 304 and 316 ss were being modified with titanium. These materials are not available at the present time.

PHASE I, TASK 5: SYNERGISTIC STUDIES OF HIGH CARBON-OXYGEN
SODIUM EXPOSURES ON 304 ss FOLLOWED BY
CREEP-RUPTURE TESTING IN 'CLEAN' SODIUM

This work is not scheduled for this fiscal year.

PHASE II - FERRITIC STEELS

PHASE II, TASK 1: CREEP-RUPTURE TESTING OF CANDIDATE FERRITIC ALLOYS IN 'CLEAN' SODIUM AT 1200 F

This portion of the program will result in detailed evaluation of one or more ferritic alloys for possible use in the LMFBR systems. Stabilized ferritic steels are to be exposed to sodium contained by stainless steel. Maintenance of high temperature strength via resistance to decarburization is the property being sought. Several alloys were initially selected, the compositions of which are listed in Table 15.

Specimens will be exposed in a stainless steel retort (as described in Phase I, Task 3) at 1200 F for periods of 1000, 2000 and 3000 hours, followed by tensile testing at several temperatures. Results of these tests would help eliminate all but the most promising alloys. The remaining alloys will then be taken into a larger scale tensile and creep-rupture test program.

Prior report periods saw Static Retorts No. 2 and 3 charged with 154 pounds of sodium and brought to the operating temperature of 1200 F. During this report period, characterization of the sodium in each of the retorts was completed, the results of which are given in the table below:

Static Retorts No. 2 and 3 Sodium Analyses Results

	Retort No.	Analytical Determinations, ppm					
		<u>O₂</u>	<u>C</u>	<u>H₂</u>	<u>C₂H₂</u>	<u>CO₂</u>	<u>Mass 43</u>
Sample No. 1	2	10	34	16	1	22	
Sample No. 2	2	-	62	12	2	14	
Sample No. 1	3	10	38	15	4	14	3
Sample No. 2	3	10	51	12	2	17	5

Duplicate samples taken from each retort and analyzed for metallic constituents gave results below the limits of detection for each constituent.

The 304 ss 5 mil tab suspended in Static Retort No. 2 for 174 hours analyzed at 477 ppm total carbon, as compared to the original material value of 616 ppm total carbon. Similarly, Static Retort No. 3 had monitoring tabs on two different occasions, -- first, 160 hours, where the final total carbon was 325 ppm; and a second time, for 168 hours, where the final total carbon was 389 ppm. (Original carbon = 545 ppm) Monitoring tabs from different heats were used in Static Retorts No. 2 and 3.

TABLE 15 - CANDIDATE FERRITIC ALLOYS

Alloy	Supplier*	Composition, %							Other
		Cr	Mo	C	Mn	P	S	Si	
2 1/4 Cr-1 Mo	1	2.26	1.02	0.14	0.44	0.013	0.018	0.16	V = 0.62, Cb = 0.094 Al = 0.025
2 1/4 Cr-1 Mo	2	2.25	1.01	0.10	0.45	0.023	0.011	0.49	Ti = 0.20 V = 0.20 Zr = 0.14 B = 0.005 Ni = 0.10 Co = 0.09
3HK5S	3	2.22	0.97	0.048	0.72	0.017	0.011	0.23	Ni = 0.61 Nb/Ta = 0.56
Croloy 5	2	5.04	0.63	0.10	0.48	0.016	0.014	0.30	Ni = 0.23 Cu = 0.11
Croloy 5 Ti	2	4.99	0.51	0.09	0.48	0.014	0.015	0.23	Ti = 0.45 Ni = 0.24 Cu = 0.12
2 1/4 Cr-1 Mo	4	2.17	1.01	0.097	0.56	0.007	0.022	0.33	

* 1. United States Steel Corporation

3. Thyssen Rohrenwerke

2. Babcox and Wilcox

4. McInnes Steel Corporation

Static Retort No. 2 was charged with a combination of the six alloys. Six specimens of each alloy were submerged in sodium while a minimum of four specimens were placed in the canned helium environment. These specimens will undergo a 3000-hour excursion which was initiated on March 7, 1968, and has now exceeded ~560 hours of exposure.

Static Retort No. 3 was charged with a similar number of specimens. These specimens will undergo a 1000-hour excursion which was initiated on March 6, 1968, and has now exceeded ~590 hours of exposure.

Sodium samples will be removed from each of these retorts at periodic intervals during the exposure and will undergo various analyses. The results of the analyses completed to date appear in the following table:

Static Retorts No. 2 and 3 Sodium Analyses Results

<u>Date</u>	<u>Analytical Determinations, ppm</u>				
	<u>O₂</u>	<u>C</u>	<u>H₂</u>	<u>C₂H₂</u>	<u>CO₂</u>
Retort No. 2:					
3/8	19	-	-	-	-
3/18	29	-	-	-	-
3/19	-	32	-	-	-
3/20	-	-	-	3	46
3/26	-	-	2	-	-
3/29	29	-	-	-	-
Retort No. 3:					
3/6	38, 34	-	-	-	-
3/7	30	-	-	-	-
3/8	26	-	-	-	-
3/11	39	-	-	-	-
3/13	20	-	-	-	-
3/15	35	-	-	-	-
3/18	40	-	-	-	-
3/22	-	-	15	5	9
3/27	10	-	-	-	-

An additional sample was taken from Static Retorts No. 2 and 3 on March 15 to be analyzed for metallic constituents. They did not show any abnormalities.

It is interesting to note the immediate increase in oxygen content upon submerging the ferritic alloys into the sodium. Both retorts had analyzed at less than 10 ppm, but increased immediately to >20 ppm and remained at or above this level for

several days even though the diffusional cold traps were operating at ~ 250 F. This same phenomenon took place upon the charging of Static Retort No. 1, as will be seen in the following section. However, in Static Retort No. 4, which was charged with austenitic alloys, the oxygen level did not rise above the original <10 ppm.

The 1000-hour exposure in Static Retort No. 3 will be completed in mid-April. The specimens will be removed and tensile tested at various temperatures in air. Upon removal from the retort, an identical number of specimens will initiate the 2000-hour exposure. Both the 2000-hour specimens and the 3000-hour specimens from Static Retort No. 2 will be removed and tensile tested in air at various temperatures.

Upon the basis of the tensile test data and supporting metallography, the number of candidate alloys will be reduced to the one or two of the most promising for further tensile and creep-rupture testing.

PHASE II, TASK 2: LONG-TERM EXPOSURES OF FERRITIC STEEL IN 1050F
'CLEAN' SODIUM IN STATIC RETORTS

The long-term effects of 'clean' sodium on the mechanical properties of selected ferritic steels is to be determined. At the present time, only the 2 1/4 Cr-1 Mo alloy has been selected for this exposure. This alloy was selected because it is a 'standard' ferritic alloy being considered for use today. Other alloys will be selected with help of the screening tests as described under Phase II, Task 1.

The parameters for this exposure are to be in static sodium at 1050 F in excess of 10,000 hours, the oxygen content of which is to be <10 ppm controlled by use of a diffusional cold trap. This exposure is then to be followed by tests similar to those outlined under Phase I, Task 3, in the preceding section.

No. 1 Retort used in this exposure is identical to that described in Phase I, Task 3. This retort was also characterized at 1200 F, as were the previous three units. The results of these sodium analyses are given in the following table:

Static Retort No. 1 Sodium Analyses

	<u>O₂</u>	<u>C</u>	<u>H₂</u>	<u>C₂H₂</u>	<u>CO₂</u>
Sample No. 1	<10	49	<2	5	106
Sample No. 2	<10	23	<2	2	38

Two samples analyzed for metallic constituents gave results below the limits of detection for each constituent.

The retort temperature was then lowered to 1050 F and the carbon activity determined by use of a 304 ss monitoring tab. After 160 hours of exposure, the total carbon content was 479 ppm, as compared to the original content of 421 ppm, which showed a low level of carbon activity.

A new heat of 2 1/4 Cr-1 Mo (than was used in previous MSAR programs) is being used in this exposure to insure a sufficient quantity of material will be available in the event a full-scale program is desired. The chemical analysis of this heat is as follows:

Chemical Analysis of Long-term Excursion 2 1/4 Cr-1 Mo

<u>Type</u>	<u>Supplier</u>	<u>Cr</u>	<u>Mo</u>	<u>C</u>	<u>Mn</u>	<u>P</u>	<u>S</u>	<u>Si</u>
2 1/4 Cr-1 Mo	McInnes Steel	2.22	0.99	0.11	0.50	0.009	0.022	0.21

A total of thirty 60 mil specimens of this material were installed in two loading ports of Static Retort No. 1 and immersed in sodium, while a total of fifteen were placed in the canned helium environment. This exposure was initiated on March 7, 1968, and now has undergone ~550 hours of exposure.

The stock thickness of this heat as received from the supplier was 1/4 in. Additional material is being rolled from which specimens will be fabricated and inserted, during the next report period, in the two remaining ports of Static Retort No. 1. (Once the ferritic screening of alloys under Phase I, Task 1, has been completed, Static Retorts No. 2 and 3 will be available for long-term exposures of other selected ferritics or other alloys.) Here again, sodium samples will be removed for various types of analyses during the exposure period. Those analyses completed to date are shown in the table below.

Static Retort No. 1 Sodium Analyses Results

<u>Date</u>	<u>Analytical Determinations, ppm</u>				
	<u>O₂</u>	<u>C</u>	<u>H₂</u>	<u>C₂H₂</u>	<u>CO₂</u>
3/8	24	-	-	-	-
3/19	20	39	-	2	64
3/26	-	-	2	-	-
3/28	21	-	-	-	-

Another sample was taken on March 15. Analysis for metallic constituents did not reveal any abnormalities.

As noted in the other units involved with ferritic specimens, there was an oxygen increase in this unit upon immersion of the ferritic test specimens. The cause of this increase has not been determined.

The excursion will continue for a minimum of 10,000 hours with sodium samples being removed on a periodic basis and analyzed for the various contaminants. At the completion of the excursion, specimens will be removed and will undergo extensive mechanical and metallurgical tests.

PHASE III - OTHER CANDIDATE ALLOYS

PHASE III, TASK 1: CREEP-RUPTURE TESTING OF CANDIDATE VANADIUM-BASE ALLOYS IN 'CLEAN' SODIUM AT 1200 F

This portion of the program requires the determination of mechanical properties in 'clean' sodium at 1200 F on the best vanadium-base alloys from the Westinghouse Development Program. MSAR is presently in the process of making arrangements with Westinghouse to obtain a quantity of vanadium alloy. At the present time, no schedule for receipt of the material has been set.

PHASE III, TASK 2: 500-HOUR EXCURSION OF CANDIDATE VANADIUM-BASE ALLOYS IN 1200 F HIGH OXYGEN SODIUM

The selected alloy under Phase III, Task 1, is to be exposed in 1200 F high oxygen sodium and then to be mechanically tested in 'clean' sodium at conditions similar to those under the austenitic tests of Phase I, Task 1. No schedule has been defined for this portion of the program.

PHASE IV - DEVELOPMENT OF HIGH TEMPERATURE EXTENSOMETER
(E. King and D. Green)

An extensometer is a device used to remotely measure the strain of a specimen under stress, when it is impossible to make the measurement directly due to the physical arrangement or environment. Most of the devices employ an extension rod that is clamped to the specimen. The rod moves as the strain increases and the movement of the rod is measured as an indication of the strain change in the specimen. The major difficulties encountered are the complexities in transmitting the motion from the stressed area at high temperature to a remote measuring device at room temperature. The thermal effects on the rod and the inability to accurately transmit the motion from the stressed area can cause wide scatter bands in test results. The motion, as strain, is generally measured with a Linear Variable Differential Transformer (LVDT) located outside the test environment.

MSAR proposed to place the LVDT in the high temperature sodium environment to eliminate the problems of the extension rod. The performance goal is the capability of measuring a strain rate of 0.0001%/hr in 1200 F. The instrument must also be usable after 1000 hours of exposure to 1200 F sodium.

Previous reports have described the current progress in the development of a high temperature Linear Variable Differential Transformer to be used as a transducing component for an extensometer operating in an atmosphere of hot liquid metals. The experimental work has been sufficiently successful to predict optimum operation within the 1200 F to 1300 F range.

The LVDT (wound with #28 Constantan wire on a ceramic tube and encased in a stainless steel housing) was tested for sensitivity, linearity, stability of output, and life. Four assemblies were wound and two of these were housed in stainless steel. The characterization of these units is described below.

Input - 3 V @ 1650 Hz to a 50 ohm primary coil

Rate - 0.02 in./mV AC linear; 0.3 in. each side
of null (cold, housed, 45 ohm/sec)

Core - Hyperco 27 (0.90 x 0.25 dia)

(All four were identical within small limits.)

From repeated hot tests (one to two hours at 1200-1300 F), two major problems were encountered, -- instability from the demodulator; and no means of relating core position to output during the heating and cooling cycle.

A battery-operated oscillator-amplifier was built to produce 8.5 V @ 1600 Hz into a 50 ohm coil. [Rate (cold, housed) - 0.0086 in./mV AC - a proportional increase in sensitivity.]

A battery-operated receiver-amplifier was used to eliminate power fluctuations. Tests now showed good stability at any fixed temperature setting and a predictable change upon heating and cooling, thereby resolving the first major problem area.

Long-term life tests were begun on February 20, 1968. One of the housed LVDT's was placed in a muffle furnace and heated to 1200 F. The temperature was held constant with a Pyrovane thermocouple and saturable reactor system, and the temperature was recorded on a strip chart for the duration of the test. A battery (12 V automobile storage battery) operated the oscillator and the receiver was connected to the unit. In checking core accuracy, adjustments were made by inserting 1/16 in. thick disks (shims) between a collar clamped to the core extension rod (3/32 in. stainless) and a guide tube leading to the LVDT.

The collar was set to place the core at null so each disk represented 1/16 in. displacement off null. The test routine consisted of taking strip-chart recordings at different core settings for as long as the battery voltage remained up. The first weeks produced excellent results. The output recording did, however, show variations when the battery charger was in play. During the latter seventy hours of the test, a different receiving amplifier, with more gain, was substituted. A distinct cycle of approximately ten minutes in length was attributed to the Pyrovane oscillator inducing a current in the receiving amplifier.

The test was run continuously for 894 hours and a recognized breakdown occurred at 820 hours, but deterioration had begun as early as 350 hours.

The LVDT was opened for examination. The insulation was intact, showing no signs of deterioration, but the Constantan wire had oxidized throughout the length of the coil and eventually open-circuited. At this writing, it is not known just what caused this deterioration. The core of Hyperco had a very thin scale of what appeared to be an oxide.

Summary

The long-term tests in 1200 F air, although shorter than anticipated, provided good data as summarized below.

1. The transformer design is satisfactory.
2. The oscillator-amplifier and the receiver-amplifier, each with its voltage-regulated power supply, will produce a stable and sensitive recording.

3. The signal output from the LVDT to the receiver-amplifier and to the recorder must be well shielded.
4. The wire and coil insulation is satisfactory.
5. The use of Constantan wire is not satisfactory.

Inconel X wire, #28 Ga, has been ordered, with delivery due about July 1, 1968. New LVDT's will be wound and tested with the new material. In addition, several test samples of Constantan will be heated in stainless steel tubing with and without a purge gas to determine the reasons for the deterioration of the Constantan.

PHASE V - ANALYTICAL TECHNIQUES
(F. Roehlich)

As part of the Development of Techniques for the Measurement of Impurities in Sodium Program, the surface tension of sodium is under study. The measurements are being conducted by means of the maximum bubble-pressure method, and oxygen content of the sodium is controlled by temperature regulation of a diffusional cold trap. Previous results and apparatus design have been covered in past reports.

Forty sets of surface tension measurements were taken during the report period at temperatures ranging up to approximately 600 F. Results showed that changes in dissolved-oxygen content did not produce clearly defined corresponding changes in the apparent sodium surface-tension values obtained with the present apparatus. A series of low surface-tension values was obtained, however, in the presence of insoluble oxides (dark oxide surface film). The difficulty in measuring small changes in apparent surface tension may be attributed partially to an inherent characteristic of the experimental apparatus. The liquid level of the sodium in the chamber could not be determined or maintained with adequate accuracy due to the presence of the 'cold trap' and insoluble surface films. Location of the liquid level is required because of the necessity in knowing the precise liquid head over the end of the capillary tube immersed in the sodium. An error of 0.005 in. in the assumed immersion depth of the capillary tubes produces a difference of one dyne/cm in the calculated surface tension. Reproducibility of depth measurement was often no better than 0.025.

A method which has been employed previously to alleviate this problem involves the use of tubes of widely different effective bubble radii mounted in a common plane beneath the liquid level. The disadvantage of this technique lies in the fact that the difference in radii must be large in order to yield significantly different maximum bubble pressures over the temperature range covered; hence the bubble on at least one of the tubes would be severely distorted.

Initial measurements with a fresh batch of sodium indicated that surface tension values taken with the cold trap and chamber both at the same temperature were higher than those taken with the cold trap frozen. Investigation showed, however, that these results may have been due to a contaminant surface layer which prevented proper determination of the actual sodium liquid level. Subsequent experiments yielded data which was quite low. (For example, 110 dynes/cm vs 207 for pure sodium at 302 F with the trap partially frozen.) The entire batch of sodium (trap and chamber) was then heated to 800 F. This temperature was maintained for about sixty hours, during which time the impurity layer dissolved.

The chamber was then equilibrated at 275 F with the center of the trap maintained at 108 F. The resulting bubble-pressure data

were stable and produced a calculated result of 220 dynes/cm which is about 5% greater than the results previously obtained for gettered 'pure' sodium. ('Pure' sodium data is shown in Figure 25.) An amalgamation sample was then withdrawn from the chamber which yielded a value of 6 ppm oxygen. The system was then heated and equilibrated at a chamber temperature of 601 F with the cold trap at 398. Results were 177 dynes/cm, which is about 5% lower than the 'pure' sodium regression line. The cold trap temperature was then lowered to 153 F. Subsequent data showed no significant change in apparent surface tension.

The chamber temperature was then returned to 293 F with the cold trap now maintained at 285 F (midpoint). Surface tension data then taken ranged from 215 dynes/cm to 221 dynes/cm over a five-day period. Hence, the effect of dropping the cold-trap temperature from 285 to 108 F with the chamber sodium at 275-295 F produced no noticeable change in surface tension. Similarly, a drop in cold-trap temperature from 398 to 153 with the chamber sodium at ~600 F had no significant effect on surface tension.

Further experimentation was halted by a temperature excursion which resulted when a mercury relay on the cold trap heater jammed. The resulting expansion of the chamber sodium completely plugged the capillary tubes. The tubes were opened again, however, by heating the upper portion of the chamber and applying 30 psig of argon cover gas.

Experiments were resumed with the chamber and cold trap both between 324 F and 335 F. Resulting surface tension values were in the range of 160-170 dynes/cm, which is 15% lower than the 'pure' sodium value at that temperature. Another amalgamation sample showed 52 ppm oxygen. The solubility curve for oxygen in sodium indicates a value of less than 5 ppm at this cold-trap temperature. The apparent return of insoluble oxygen was attributed to unstable convection currents resulting from the temperature excursion. Surface tension data are presented in Table 16. Each value listed is the average of duplicate readings.

It may be concluded that soluble oxygen changes within the solubility limits have no measurable effect on apparent surface tension as determined by the maximum bubble pressure apparatus employed.

Due to inherent limitations of the experimental device, it is difficult to compare results of clean versus contaminated sodium. However, the results do indicate that undissolved oxygen, either in the sodium or on the sodium surface, does result in a significant decrease in apparent surface tension.

MSAR had a brief opportunity to review this program with Dr. A. W. Thorley (Culcheth, England) during his recent visit. The studies they had made in this area and reported at the international symposium held at Nottingham in 1966 would tend to indicate they

FIG. 25 - 'PURE' SODIUM SURFACE TENSION

TABLE 16 - SURFACE TENSION DATA

<u>Data Point No.</u>	<u>Chamber Temperature (°F)</u>	<u>Cold Leg Temperature (°F)</u>	<u>Surface Tension (dy/cm)</u>
1	290	75	180
2	290	107	189
3	330	327	223
4	335	329	214
5	325	318	220
6	290	105	178
7	302	132	110
8	322	321	129
9	275	108	220
10	590	153	177
11	590	153	165
12	293	285	221
13	295	282	215
14	295	282	220
15	331	324	168
16	335	328	166
17	331	324	160
18	510	507	169
19	514	508	198
20	352	348	213

might be in conflict with this program. However, when the method and purpose of this program were explained, Thorley predicted that there would be a decrease in surface tension with increases in oxygen content by this method. However, at the same time, he stated that by any method, whether it be based on wetting, contact angles, bubble pressure, or others, surface tension is a difficult physical property to measure quantitatively. MSAR has found this to be true in as much as the test machines never seem to adequately measure changes which would revolve the problem decisively.

No conclusions can be drawn on the effects of dissolved oxygen on surface tension with the experimental apparatus used in this study. Additional work on the effect of oxygen on surface tension has been discontinued during the remainder of this fiscal year because of limited fundings. Future work on the effects of oxygen on surface tension would require that the experimental device be redesigned to avoid the effects of temperature and liquid-level changes.

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