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SODIUM MASS TRANSFER: II
SCREENING TEST DATA AND ANALYSIS

Volume 2 — Metallurgy

By
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June 1962

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SODIUM MASS TRANSFER: II
SCREENING TEST DATA AND ANALYSIS
VOLUME 2-METALLURGY

by

F. J. Hetzler and R. S. Young

Volume 1 - Mass Transfer Results
Volume 2 - Metallurgy
Volume 3 - Corrosion Sample Data

June, 1962

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GENERAL ELECTRIC

SAN JOSE, CALIFORNIA

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I. INTRODUCTION

In 1959 the U. S. Atomic Energy Commission contracted with the General Electric Company to carry out a Sodium Mass Transfer investigation with the objective of evaluating three prospective steel construction materials for utilization in liquid sodium-cooled reactor systems operated in the temperature range of 600 to 1300 F. Six liquid sodium test loops were designed and built to implement the study of the three selected materials: AISI 316 stainless steel (316SS); $2\frac{1}{4}$ percent Chromium - 1 percent Molybdenum steel ($2\frac{1}{4}\text{Cr}-1\text{Mo}$), and 5 percent Chromium - $\frac{1}{2}$ percent Molybdenum - $\frac{1}{2}$ percent Titanium steel ($5\text{Cr}-\frac{1}{2}\text{Mo}-\text{Ti}$). The latter two were chosen because of their extensive service in central station power plant applications. A complete description of the design and fabrication of the test loops has been included in GEAP-3725 (1).^{*} Operation of the first loop began in June of 1960. The operational data, corrosion analysis and general program objectives have been included in Volume 1 of this report, GEAP 3726 (2); Volume 3 contains the corrosion sample data.

This document reports and interprets extensive observations of the metallurgical effects noted after exposure of the materials to liquid sodium. This exposure was made under dynamic conditions in test runs of up to 3000 hours from the initial startup through December 1961. The test program is continuing to obtain results under other conditions of the primary test parameters. Effects reported herein are under further investigation to gain improved understanding of the mechanisms of their occurrence and the possible effects on mechanical properties.

^{*} Note

Numbers in parentheses () correspond to items in bibliography.

II. SUMMARY AND CONCLUSIONS

Results obtained in these initial screening runs are summarized by materials. Where results depend upon bimetallic material combinations the specific combination is indicated. Reference to Table III-1 and Figure III-1 will clarify the material systems mentioned.

A. 2 $\frac{1}{4}$ Cr-1Mo

The material decarburized at 1000-1200 F in both monometallic and in bimetallic 316SS-2 $\frac{1}{4}$ Cr-1Mo systems. The decarburization process was found to increase with increasing oxygen levels in the sodium and by providing a carbon sink.

Limited tensile data indicated that strength of the material significantly decreased with the loss of carbon. The shape of the carbides probably was significant since higher tensile strengths were encountered when the carbides were formed in a pearlite phase than when the carbides existed in spherodized form. A reduction of approximately 30% in room temperature tensile strength was indicated by the data.

Carbide stabilized 2 $\frac{1}{4}$ Cr-1Mo alloys, 4S4 and 4S8, special alloys developed by Atomics International and U. S. Steel Corp., were approximately neutral to carbon transport in 700 hours in a bimetallic 316SS-2 $\frac{1}{4}$ Cr-1Mo system.

No serious localized effects were observed in this alloy group.

B. 5Cr- $\frac{1}{2}$ Mo-Ti

Intergranular penetration of corrosion products was observed in 1000-1200 F temperature regions to a maximum depth of 0.001-inch in approximately 2800 hours. The layer where the phenomenon occurred was found to be chromium depleted. This penetration can result in lower strength properties particularly in the case of components with stress-concentrations. Depths of penetration are minor so far but longer times than 2800 hrs. at low sodium velocities might enhance the effect and cause significant reductions in stress-rupture strength and hasten inter-crystalline cracking.

The alloy was approximately neutral to carbon transport, both in a monometallic and in a bimetallic 316SS-5Cr- $\frac{1}{2}$ Mo-Ti system.

C. 316 Stainless Steel

The alloy was approximately neutral to carbon transport in a monometallic system, but carburized in a bimetallic 316SS-2 $\frac{1}{4}$ Cr-1Mo system. The degree of carburization was limited by the apparent inability of 316 stainless steel to accept carbon as readily as the ferritic alloy lost carbon. An increase in carbon content slightly in excess of twice the as-received value of 0.045% was observed. This can be expected to cause some increase in mechanical properties, tensile and yield strength. The effects on microstructure of carbon increase thus far in this work have not been completely evaluated; however, in areas of worked structure heavier carbide cases and precipitation of carbides along preferred crystallographic planes, termed Widmanstätten structure, have been observed. These effects indicate possible susceptibility to local embrittlement.

An austenitic-to-ferritic transformation occurred at the surface at temperatures of 1100-1300 F. This transformation was observed to begin in 700 hours and was enhanced in regions of deformed structure. A surface structure anomaly was also observed at 1000 F in 700 hours which may be the initiation of the transformation. Chromium depletion was observed adjacent to the surface in the area where these effects took place.

III. LOOP DESCRIPTION AND SAMPLE PREPARATION

A. SODIUM LOOPS

The six test loops constructed for the Sodium Mass Transfer project were identical in design and construction with the exception of materials which varied as indicated in Table III-1.

TABLE III-1

LOOP DESIGNATIONS AND MATERIALS

<u>Loop</u>	<u>Material</u>	
	<u>Hot Leg*</u>	<u>Cold Leg*</u>
1	316 SS	316 SS
2	316 SS	2 $\frac{1}{4}$ Cr-1Mo
3	316 SS	2 $\frac{1}{4}$ Cr-1Mo
4	316 SS	5Cr- $\frac{1}{2}$ Mo-Ti
5	2 $\frac{1}{4}$ Cr-1Mo	2 $\frac{1}{4}$ Cr-1Mo
6	5Cr- $\frac{1}{2}$ Mo-Ti	5Cr- $\frac{1}{2}$ Mo-Ti

Loops of a single material are referred to as monometallic, while loops of two alloys are termed bimetallic. A monometallic system, for all practical purposes, contains no other materials of construction than that specified.

Bimetallic systems were constructed with a "hot-leg"* of 316SS and a "cold-leg" of the designated chrome-molybdenum alloy. The isometric diagram (Figure III-1) provides a simplified view of a loop and indicates the seven sample holders with their usual operating temperatures.

* Hot Leg: That portion of a loop where increasing temperatures are experienced, samples are generally at 1000, 1100 and 1200 F.

Cold Leg: That portion of a loop where decreasing temperatures are experienced, samples are generally at 1100, 1000 and 700 F.

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FIGURE III-1 SODIUM MASS TRANSFER LOOP ISOMETRIC WITH NOMINAL OPERATING TEMPERATURES OF STANDARD CORROSION SAMPLE HOLDERS

Sodium loop construction details and a comprehensive report of operational data for each run period can be found in GEAP 3725 and Volume 1 of this report. (1), (2) A summary of test conditions for the runs from which data were obtained for this report is contained in Appendix A.

Four types of samples were used in the experiment. These are illustrated in Figure III-2.

The "standard corrosion sample" provided the bulk of information with respect to both corrosion and metallographic data. "In-pipe samples" produced similar information in limited quantities. The "0.006-inch thick foil sample" was primarily designed as a radioactive tracer tab. Metallographic data from the tabs were not satisfactory because of the small cross section. They were generally given only a cursory inspection.

B. SAMPLE PREPARATION

All corrosion samples were fabricated from the same steels utilized in the loop sections in which they were to be exposed. Prior to operation of the six test loops, a metallographic examination was carried out on each lot of the construction materials, to establish a basis for sample comparison after exposure to liquid sodium, and provide standards for test specimen preparation. Samples of 316SS, $2\frac{1}{4}$ Cr-1Mo steel and 5Cr- $\frac{1}{2}$ Mo-Ti steel were cut from the following stock: $\frac{1}{2}$ -, 1-, 4- and 8-inch schedule 40 pipe; 3/8- and 5/8-inch tube; 1-inch bar and $3\frac{1}{2}$ - to 4-inch bar. Examinations revealed structures normal to wrought products of these alloys.

Material for the $2\frac{1}{4}$ Cr-1Mo and 5Cr- $\frac{1}{2}$ Mo-Ti samples was obtained by cutting pipe from the above stock, flattening and rolling to 1/16-inch thick sheet. 316SS test sample stock was initially obtained by rolling 1/8-inch plate to a 1/16-inch thick sheet. To eliminate the effects of rolling operations all samples were heat treated.

The initial heat treatments, employed to bring the micro-structures into conformance with structures observed in as-received loop steels, were as follows:

FIGURE 111-2 CORROSION SAMPLES

Left to Right: In- pipe, standard corrosion,
0.006-inch foil and 1/2-inch pipe samples.

2 $\frac{1}{4}$ Cr-1Mo - $\frac{1}{2}$ hour at 1575 F, furnace cooled to 1200 F and oil quenched.

5Cr- $\frac{1}{2}$ Mo-Ti - $\frac{1}{2}$ hour at 1575 F, furnace cooled to 1200 F and oil quenched.

316SS - $\frac{1}{2}$ hour at 2000 F and oil quenched.

Variations in heat treating, such as the use of a dry-hydrogen atmosphere for 2 $\frac{1}{4}$ Cr-1Mo and argon for 5Cr- $\frac{1}{2}$ Mo-Ti, and changes in holding and cooling times were later employed. These are indicated in the examinations section of the report. For expediency, the 316 stainless steel sample stock was used for standard corrosion samples in the as-rolled condition in the first check-out run (1P) of loop #1 and for original "in-pipe" samples installed in loops #1, #2, #3, and #4. Material in the annealed condition was subsequently incorporated in all runs requiring 316SS. Early problems with duplex grain structure were alleviated by using 316SS sheet purchased to specification.

Table III-2 shows the compositions, specifications and material sources of the three primary test steels. The structures characteristic of the loop materials and representative of good sample stock are shown in Figures III-3, III-4, and III-5. Two variations of 2 $\frac{1}{4}$ Cr-1Mo (4S4 and 4S8 alloys) were tested at the request of Atomics International and the Atomic Energy Commission for a short time in run 2 of loop #3. Their compositions are shown in Table III-3.

Problems encountered in sample fabrication in the early phases of testing were cold working and excessive heating caused by cutting and grinding operations. The effects of these operations were subsequently alleviated to some extent by care in cutting and machining and wet-grinding to finish dimensions. Macroinspection, Rockwell "B" hardness, and X-ray fluorescence measurements were taken of representative samples to minimize variations.

The specimens were cleaned as follows:

1. The chromium-molybdenum alloys were vapor blasted with No. 220 grit silicon carbide and rinsed in a dilute aqueous solution of sulfuric acid.
2. The 316SS was degreased with a detergent and passivated with a 20 per cent nitric acid - 2 per cent sodium dichromate solution at 110-120 F for 20 minutes.

TABLE III-2
CHEMICAL COMPOSITIONS OF THE PRIMARY TEST STEELS*

ALLOY	$2\frac{1}{4}\text{Cr}-1\text{Mo}$		$5\text{Cr}-\frac{1}{2}\text{Mo}-\text{Ti}$		AISI 316	
ASTM Specification (3)	A335-P22		A335-P5c		A240-316-2B	
Analysis	ASTM	Certified 1-Inch Pipe	ASTM	Certified 2-Inch Pipe	ASTM	Certified 1/16-Inch Sheet
Carbon, %	0.15 max.	0.105	0.12 max.	0.09	0.08 max.	0.05
Manganese, %	0.30 to 0.60	0.45	0.30 to 0.60	0.48	2.00 max.	1.61
Phosphorus, %	0.030 max.	-	0.030 max.	--	0.045 max.	-
Sulfur, %	0.030 max.	-	0.030 max.	--	0.030 max.	-
Silicon, %	0.50 max.	0.24	0.50 max.	0.40	1.00 max.	0.55
Chromium, %	1.90 to 2.60	2.28	4.00 to 6.00	4.76	16.00 to 18.00	16.80
Molybdenum, %	0.87 to 1.13	0.96	0.45 to 0.65	0.52	2.00 to 3.00	1.95
Titanium, %	--	--	4xC min. 0.70 max.	0.48		-
Nickel, %	--	--	--	--	10.00 to 14.00	12.50

* Material Source

$2\frac{1}{4}\text{Cr}-1\text{Mo}$, Gately Stainless and Alloy Co. Heat No. 657266

$5\text{Cr}-\frac{1}{2}\text{Mo}-\text{Ti}$, Babcock and Wilcox Co. Heat No. 15602

AISI 316, Commercial Supply

250X
2 1/4 Cr - 1 Mo

PICRAL
STANDARD B33M

FIGURE III-3

Sample hot-rolled from flattened 1-inch pipe to 1/16-inch thickness, then annealed in dry-hydrogen at 1575 F. ASTM grain size 8. Carbide preference to grain boundaries.

250X
5 Cr - 1/2 Mo - Ti

NITAL-PICRAL
STANDARD C11M

FIGURE III-4

Sample hot-rolled from 2-inch flattened pipe to 1/16-inch thickness, and annealed in argon at 1575 F. ASTM grain size 6. Carbides spheroidized, with 2-5 micron particle size.

75X
316SS

10% OXALIC-ELECTROLYTIC
STANDARD A14M

FIGURE III-5

Typical annealed structure of austenitic grade stainless steels. The sample had been fabricated from annealed sheet stock of proper thickness dimension. ASTM austenitic grain size 5.

3. All samples were then flushed with hot water, thoroughly rinsed with distilled water, acetone rinsed, dried in a 250 F oven, then weighed.

TABLE III-3

CHEMICAL COMPOSITION OF 4S4 and 4S8 ALLOYS

(Developed and supplied by Atomics International
and U. S. Steel Corporation (4))

	<u>4S4*</u>	<u>4S8**</u>		<u>4S4*</u>	<u>4S8**</u>
C, %	0.14	0.14	Cr, %	2.34	2.23
Mn, %	0.46	0.39	Mo, %	1.08	0.99
P, %	0.006	0.003	Nb, %	-----	0.10
S, %	0.024	0.007	V, %	0.60	0.60
Si, %	0.20	0.25			

* Experimental Heat #L5066

** Experimental Heat #L6015

IV. EXAMINATIONS AND RESULTS

After exposure, the specimens were weighed and visually examined by means of a low power microscope for color and amount of surface deposits and localized effects. Photographs and hardness measurements were made of representative samples. The samples were cut into three sections. One section was metallographically mounted and subjected to a thorough micro-examination, the second utilized for carbon analysis and the third placed in permanent storage. X-ray fluorescence* and diffraction measurements were performed on selected samples to aid in interpretation of the exposure effects. A limited number of room-temperature tensile tests were carried out to note any change in mechanical properties resulting from exposure.

Approximately 428 samples were examined during the period June, 1960 to December 31, 1961. Appendix A summarizes the work in historical fashion by test-runs and includes associated average operating conditions with significant examination results. These data are discussed below in a generalized manner under each specific material designation.

A. 2 $\frac{1}{4}$ Cr-1Mo STEEL

Illustrations presented of the 2 $\frac{1}{4}$ Cr-1Mo samples, in Figures IV-1 through IV-8, are representative of the significant microstructural changes. Temperatures of the liquid sodium produced the most pronounced observable effect on the samples. Effects created by time, oxygen content of the sodium, and velocity and activity due to composition gradients of monometallic or bimetallic systems were difficult to observe by metallographic inspection. These effects were real, however, and appeared in the carbon analysis.** In some cases fabrication practice, such as hot rolling, caused variations in microstructure, (grain distortion and carbon loss) which were taken into account in the analysis.

- - - - -
* X-ray fluorescence data is being subjected to a separate analysis and will be reported at a later date.

** Carbon analysis results are contained in Appendix C.

250X
2 1/4 Cr - 1 Mo

PICRAL
STANDARD B11M

FIGURE IV-1

Distorted structure, typical of samples fabricated by flattening and hot-rolling 1-inch pipe. Note the severe grain elongation and massive carbides.

250X
2 1/4 Cr - 1 Mo

PICRAL
STANDARD B11M

FIGURE IV-2

The sample was fabricated from the material illustrated in Figure IV-1, then annealed at 1575 F and cooled at a rate of 150 F per hour.

250X
2 1/4 Cr - 1 Mo

PICRAL
STANDARD B11M

FIGURE IV-3

Figure IV-3 represents the sample illustrated in Figure IV-2. The edge view has a decarburized layer of 0.004 inch. Decarburization was partially created by the annealing environment and partially by a previously carbon depleted surface present in the 1-inch pipe used for the material source.

250X
2 1/4 Cr - 1 Mo

PICRAL
STANDARD B33M

FIGURE IV-4

This sample was also fabricated from material illustrated in Figure IV-1. A slightly longer holding period (20 minutes) at 1575 F was incorporated and the cooling rate limited to 10 - 50 F per hour to 1300 F. The carbides formed spheroidized particles with a tendency toward grain boundary locations. This structure is nearly identical to the parent 1-inch pipe. Surface decarburization did not appear in the sample.

250X
2 1/4 Cr - 1 Mo
LOOP #2, RUN 1

PICRAL
2902

10 PPM O₂ SODIUM
700 °F - 700 HOURS

FIGURE IV-5

This sample was comparative to standard B11M. (Figure IV-2). No significant changes in microstructure were recorded. Samples exposed to the same temperature appeared similar. Oxygen level, velocity, and time did not appreciably alter other samples from the above figure.

FIGURE IV-6

Decomposition of the pearlite phase can be noted. Carbon analysis (0.105% as received - 0.090% after exposure) indicated carbide loss. This sample was representative for time-temperature periods of similar magnitude. Compare to Figure IV-2.

250X
2 1/4 Cr - 1 Mo
LOOP #5, RUN 1

PICRAL
5101

50 PPM O₂ SODIUM
1000 °F - 1400 HOURS

250X
2 1/4 Cr - 1 Mo
LOOP #3 RUN 1
1100 °F - 2800 HOURS

PICRAL
3600
50 PPM O₂ SODIUM

FIGURE IV-7

Standard sample B11M, Figure IV-2, was used as the standard of comparison. Isothermal carbide decomposition had proceeded from a pearlite stage to spheroidized particles. Carbide redistribution extended to the surface thereby negating the original decarburized layer.

250X
2 1/4 Cr - 1 Mo
LOOP #5, RUN 1
1200 °F - 2800 HOURS

PICRAL
5500
50 PPM O₂ SODIUM

FIGURE IV-8

Temperature and environment effects had removed the major portion of original carbides noted in B11M, Figure IV-2. Ferrite structure with dispersed spheroidized carbides remained. The carbon content had changed from 0.105 - 0.050%. Basic grain size had not been altered.

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Examination of the carbon data for the first runs of loops #2, #3, and #5 indicated significant variations. Carbon content of samples at the same temperature and environmental conditions, but in different loop positions, were found to be within limits of ± 0.010 percent at the 95 percent tolerance level. When these data were fitted to a standard Arrhenius equation of the form $K \exp - Q/RT$ where Q is the energy of activation for a process, R is the gas constant, T is temperature in degrees Kelvin and K a frequency factor, it appeared that carbon diffusion behaved as a function of temperature, oxygen level and activity produced by providing a carbon sink. Table IV-1 tabulates the calculated Arrhenius equation coefficients.

TABLE IV-1
CALCULATED ARRHENIUS EQUATION COEFFICIENTS FOR CARBON DIFFUSION
in $2\frac{1}{4}\text{Cr}-1\text{Mo}$

LOOP-RUN	O_2^* , ppm	MATERIAL COMBINATIONS	k	Q
3-1	50	316SS- $2\frac{1}{4}\text{Cr}-1\text{Mo}$	2.2×10^{-2}	$4,000 \pm 3,000$
2-1	10	316SS- $2\frac{1}{4}\text{Cr}-1\text{Mo}$	3.6×10^{-3}	$8,000 \pm 2,000$
5-1	50	$2\frac{1}{4}\text{Cr}-1\text{Mo} - 2\frac{1}{4}\text{Cr}-1\text{Mo}$	8.6×10^{-3}	$22,000 \pm 6,000$

* Based on KAPL Oxygen Saturation Data, TID 5277, p. 7.

Comparison of the activation energies Q indicated that differences were encountered with oxygen levels between runs 2-1 and 3-1 and that run 5-1 without 316 SS as a carbon sink required a much larger activation energy than either. Fick's second law of diffusion, (5) accounts for time as a function of its square root. The data obtained did not have a sufficient time spread for this factor to be readily computed for operating periods encountered. A second likely possibility can also be theorized for the lack of a time-variable separation. Since the samples have a thickness dimension of 1/16-inch, the carbon content of the samples reduces significantly in the first 700-hour period. As the carbon content approaches equilibrium, diffusion rates decrease preventing significant changes for

periods longer than 700 hours. These are conditions that will be separated in the future test program.

Maximum system temperatures, as well as over-all temperature differentials, no doubt would have major effects on the decarburization process. To date these test conditions have not been varied; however, future experiments will provide additional data. Results presented correlate with Epstein(6) and those of Hayes and Shepard.(7) The latter investigation predicts that the equilibrium carbon content of $2\frac{1}{4}\text{Cr-1Mo}$ alloy in a 304 stainless steel system is somewhat less than 0.02 percent. Epstein predicted the Arrhenius equation for movement of low chromium complex carbide in ferrite would be $0.2/\sqrt{t} (\exp - 20,000/RT)$, or for a period of 1000 hours $7 \times 10^{-3} \exp - 20,000/RT$ which compares favorably with results obtained for $2\frac{1}{4}\text{Cr-1Mo}$ in the monometallic system.

A limited number of room-temperature tensile tests were made on corrosion samples from early runs. It is recognized that the corrosion samples did not provide standard size ASTM tensile test specimens; however, the results were interesting and therefore are reported in Table IV-2.

The results correlate with the discussion by Babcock and Wilcox(8),(9) on the effect of carbon as an alloying element, where not only the amount of carbon present but the form that it assumes, provides tensile strengths of varying magnitude.

Limited data were obtained for the 4S4 and 4S8 (Carbide Stabilized $2\frac{1}{4}\text{Cr-1Mo}$) alloys since only a total of 12 samples had been exposed to test conditions. The single test period of 700 hours did not allow a time-effect correlation to be made. Carbon analysis indicated that both alloys decarburized at 700, 1000, and 1100 F (from 0.14 percent to 0.13 percent); however, it was possible that the analyses may have provided deviation of a magnitude to account for the change. Chemical analyses tabulated in Table III-3 indicate the carbide stabilizing additions of vanadium or columbium-vanadium. A discussion of the stabilized ferritic steels has been presented by Hayes and Birkle(4) who describe the possibilities of these alloys for sodium service.

TABLE IV-2

ROOM TEMPERATURE TENSILE STRENGTHS OF $2\frac{1}{4}$ Cr-1Mo

<u>Sample Number</u>	<u>Loop-Run</u>	<u>Exposure Temp., °F</u>	<u>Exposure Time, Hrs.</u>	<u>Percent C</u>	<u>Yield Strength psi</u>	<u>Ultimate Strength psi</u>
Standard	---	---	---	0.105	53,000	98,000
Standard	---	---	---	0.105	52,000	100,000
Standard	---	---	---	0.105	52,000	97,000
3906	3-1	700	700	0.090	63,000	89,000
5902	5-1	700	700	0.085	66,000	92,000
5903	5-1	700	700	0.085	63,000	90,000
2904	2-1	700	1400	0.090	59,000	87,000
2902	2-1	700	700	0.095	61,000	90,000
3702	3-1	1000	2800	0.100	59,000	85,000
5101	5-1	1000	1400	0.090	51,000	80,000
2600	2-1	1100	700	0.040	35,000	68,000
2601	2-1	1100	1400	0.040	30,000	61,000
5501	5-1	1200	700	0.050	32,000	63,000

Summary of $2\frac{1}{4}$ Cr-1Mo Steel Examinations.

The most significant effect of the sodium environment on $2\frac{1}{4}$ Cr-1Mo was decarburization, with a possible exception in the case of the carbide-stabilized 4S4 and 4S8 alloys. A set of Arrhenius equations indicate that the movement of carbon is a function of temperature, oxygen level and materials incorporated in the loop. These were derived from an equation suggested by Epstein. A limited amount of tensile data was tabulated which indicated that yield strengths, of samples exposed at 1200 F for 700 hours in the liquid sodium environment, dropped from 52,000 to 32,000 psi and ultimate strengths dropped from 98,000 to 63,000 psi.

B. 5Cr- $\frac{1}{2}$ Mo-Ti STEEL

Fabrication of the 5Cr- $\frac{1}{2}$ Mo-Ti samples was accomplished by flattening 2-inch pipe sections then hot-rolling to the specified 1/16-inch dimension.

Figure IV-9 illustrates the distorted structure obtained by hot-rolling.

Figure IV-10 indicates the improved structure obtained with an annealing treatment. Carbides were distributed evenly and any indication of a decarburized surface zone was not observed. To prevent possible hydriding of the titanium, argon was employed for the annealing atmosphere in place of dry hydrogen used for the 2 $\frac{1}{4}$ Cr-1Mo.

Intergranular penetration to approximately 0.001-inch was observed on some samples exposed to the liquid sodium environment at temperatures of 1000 F and greater. Figure IV-11 illustrates the surface condition of a non-exposed (IV-11A) and the nature of the penetration in an exposed (IV-11B) sample.

Table IV-3 notes the intergranular attack with respect to frequency of occurrence, depth and loop conditions. The increasing frequency by which the intergranular penetration appeared at temperatures of 1000-1200 F, and a recheck of pre-exposure control samples, indicated that the effect was real and not created by pre-exposure sample treatment.

X-ray diffraction examinations of a $\frac{1}{2}$ -inch-in-pipe sample with intergranular penetration to the extent of 0.001-inch were made. Three successive layers of material were removed from the inside surface of the 5Cr- $\frac{1}{2}$ Mo-Ti section and examined by X-ray diffraction. Each layer was approximately 0.001-inch thick. All layers removed had a body-centered cubic structure. They differed only in the size of the unit cell. The first two layers were single phase α Fe as determined by measurement of the unit cell. The third layer removed was found to be single phase, but the lattice parameter increased to a value that indicated about a 4 percent solid solution of Cr in α Fe. The examination, therefore, indicated a chromium depleted surface in which the penetration appeared.

250X
5 Cr - 1/2 Mo - Ti

NITAL-PICRAL
STANDARD C1M

FIGURE IV-9

As-rolled condition of the 5Cr-1/2Mo-Ti. Note elongated grain structure. Surface decarburization to approximately 0.003 inch was also observed.

250X
5 Cr - 1/2 Mo - Ti

NITAL-PICRAL
STANDARD C11M

FIGURE IV-10

Same material as illustration in Figure IV-9 after 1575 F anneal with a 10 - 50 F per hour cooling rate. Argon protective atmosphere. ASTM grain size 5.

250X
5 Cr - 1/2 Mo - Ti

NITAL-PICRAL
STANDARD C11M

FIGURE IV-11A

Edge view of a non-exposed sample, typical appearance of samples exposed in the 700 F temperature region.

250X
5 Cr - 1/2 Mo - Ti
LOOP #4 RUN 1

NITAL-PICRAL
4600
50 ppm O₂

1100 °F - 2800 HRS.

FIGURE IV-11B

Intergranular penetration to 0.0008-inch appears at surface as viewed on the cross-section of the sample.

TABLE IV-3

INTERGRANULAR PENETRATION OF 5Cr- $\frac{1}{2}$ Mo-Ti STEEL

<u>Penetration Frequency</u> Penetration inches X 10^{-3}												
Temp. Deg. F	Time Hrs.	None	0.1	0.2	0.3	0.4	0.5	0.6	0.7	0.8	0.9	1.0
700 F	700	6 (4)	(1)									
	1400	6 (5)	(1)									
	2100	4 (1)	(1)									
	2800	4 (8)										
1000 F	700	1 (1)										
	1400	(3)										
	2100											
	2800	(6)	(1)	(3)		1			1	1		
1100 F	700	2		(1)								
	1400			1 (1)	(1)		(1)					1
	2100					1						
	2800	(2)	(2)	1 (8)	1	(3)		1		1	1	2
1200 F	300		(2)									
	700	(3)	(1)	(1)								
	1400	(3)	(1)	(1)					(1)			
	2100	(1)		(1)				(1)				
	2800	(4)	(1)	(1)								

Note: Quantities shown in parentheses, (), are samples from the monometallic loop. Remaining quantities are samples from the bimetallic loop.

Carbon migration experienced with $2\frac{1}{4}\text{Cr}-1\text{Mo}$ was not apparent in the $5\text{Cr}-\frac{1}{2}\text{Mo}-\text{Ti}$. The alloy remained approximately neutral to carbon activity, although slight (0.09-0.12 percent) gains were recorded. The gain occurred regardless of oxygen levels or whether monometallic or bimetallic systems were employed in the experiments. Results of carbon analyses are listed in Table IV-4.

TABLE IV-4

CARBON CONTENT OF THE $5\text{Cr}-\frac{1}{2}\text{Mo}-\text{Ti}$ CORROSION SAMPLES

	<u>Minimum Values</u> <u>Percent C</u>	<u>Maximum Values</u> <u>Percent C</u>
Unexposed samples	0.088	0.097
Samples exposed in a monometallic system (Loop 6)	0.094	0.134
Samples exposed in a bimetallic system (Loop 4)	0.089	0.114

These data are partially substantiated by Hayes and Shepard(7) who reported that $5\text{Cr}-\frac{1}{2}\text{Mo}-\text{Ti}$ remained approximately neutral to carbon transport in a 304 stainless steel loop. Their experiment was performed with 1025 F sodium, 13.5 ft/sec velocity, and 36 parts-per-million oxygen in the sodium. No substantial temperature gradient was employed.

Eight room-temperature tensile tests were performed on $5\text{Cr}-\frac{1}{2}\text{Mo}-\text{Ti}$ corrosion samples from both monometallic and bimetallic loops. There was no significant variation in the room-temperature tensile properties of exposed samples versus nonexposed.

Summary of $5\text{Cr}-\frac{1}{2}\text{Mo}-\text{Ti}$ Examinations.

The $5\text{Cr}-\frac{1}{2}\text{Mo}-\text{Ti}$ remained approximately neutral to carbon migration for the experimental conditions. Slight intergranular penetration appeared in a

chromium depleted surface of the alloy. Penetration did not exceed 0.001-inch in a period of 2800 hours and was observed primarily in samples exposed to the 1000-1200 F temperature regions.

C. AISI 316 STAINLESS STEEL

Loop #1, the monometallic 316 stainless steel loop, was the first system to be used in the Sodium Mass Transfer experiment. Figure IV-12 illustrates the as-received stainless steel used for samples in Run 1P of Loop #1. The result of a 400-hour exposure of this material at 1300 F is shown in Figure IV-13. Figure IV-14 is illustrative of the results of annealing the hot-rolled material shown in Figure IV-12 to obtain an improved structure representative of the as-received 316 stainless steel pipe used in the sodium loop construction.

The photomicrographs in Figures IV-15 through IV-19 are those of samples from Loop #2, Run 1. This was a normal run in a bimetallic loop (316SS-2 $\frac{1}{4}$ Cr-1Mo) which produced nominal 700-, 1400-, 2100-, and 2800-hour exposure periods with an oxygen level in the sodium of about 10 parts-per-million. Sample material was similar to that observed in ALOM, Figure IV-14; some duplexing of the grain size, attributed to fabrication and annealing practice, can be observed in exposed samples. The environmental effects on the 316SS are clearly evident in the photomicrographs and are representative of those observed in other loops.

The samples that have been illustrated in Figures IV-15 through IV-19 are repeated in Appendix B. The appendix compares the previous figures, produced with bright field illumination, to those photographed with polarized illumination. There is also an accompanying set of photomicrographs magnified 750X to indicate the nature of the carbide formation. The highly magnified set of photomicrographs incorporated a chemical etchant so that etching artifacts, which are occasionally associated with electrolytic etchants would not be present.

Following the initial start-up of the sodium loops, AISI 316 stainless steel sheet 1/16-inch thick was procured to ASTM specifications and used for subsequent sample manufacture. The microstructure of this material

75X
316SS

10% OXALIC-ELECTROLYTIC
STANDARD A1M

FIGURE IV-12

Sample representative of material in Loop #1 for run 1P. Material was hot-rolled from 1/8-inch plate which produced the severe elongated grain structure. The rolling temperatures were responsible for the sensitization.

75X
316SS

10% OXALIC-ELECTROLYTIC
1400

LOOP #1, RUN 1P

50 PPM O₂ SODIUM

1300 °F - 400 HOURS

FIGURE IV-13

Corrosion sample from material illustrated in Figure IV-12. The microstructure appeared similar to the standard, however, approximately 0.001-inch of ferrite had developed at the surface.

75X
316SS

10% OXALIC-ELECTROLYTIC
STANDARD A10M

FIGURE IV-14

This figure represents the microstructure of samples provided with an anneal. Carbon 0.045%. Previously the material was in a condition similar to that of Figure IV-12.

75X
316SS

10% OXALIC-ELECTROLYTIC
2100

LOOP #2, RUN 1

10PPM O₂ SODIUM

1000°F - 651 HOURS

FIGURE IV-15

Sample was moderately sensitized. Note the zone of microstructure at the surface. Carbon 0.070%. Compare to as-received annealed structure, Figure IV-14.

75X
316SS

10% OXALIC-ELECTROLYTIC
2101

LOOP #2, RUN 1

10 PPM O₂ SODIUM

1000 °F - 1438 HOURS

FIGURE IV-16

Sensitization in the sample was increased over that of the previous sample. The band at the surface has increased slightly with increased time. Carbon 0.075%. Compare to Figure IV-14.

75X 10% OXALIC-ELECTROLYTIC
 316SS 2175
 LOOP #2, RUN 1 10PPM O₂ SODIUM
 1000°F - 2728 HOURS

FIGURE IV-17

Microstructure was similar to the previous sample Figure IV-16. The surface band has increased with the exposure time. Carbon 0.085%. Compare to Figure IV-14.

75X 10% OXALIC-ELECTROLYTIC
 316SS 2200
 LOOP #2, RUN 1 10 PPM O₂ SODIUM
 1100 °F - 2728 HOURS

FIGURE IV-18

A continuous intergranular carbide network had formed. Spherodized clusters of carbides existed and a Widmanstätten structure appeared adjacent to the surface. Incipient ferrite development appeared at the surface, but the band observable in the 1000 F samples was not visible. Carbon 0.09%. Compare to Figure IV-14.

277-12

FIGURE IV-19

The above 4 photomicrographs indicate the effects of increasing times at 1200 F in Loop #2, Run 1, with 10 ppm O_2 in sodium. Carbide formations appeared more heavily near the surfaces in the first pair of photomicrographs, (a) and (b). These two samples were in a physical position so that the sodium stream passed over them before reaching the samples indicated in (c) and (d). Compare to Figure IV-14.

is the same as shown in Figure III-5. The 2B surface finish supplied by the vendor was preserved on finished samples, while wet-grinding was employed to partially remove the effects of shearing on sample edges. The result of exposure of this material in a bimetallic 316SS-2 $\frac{1}{4}$ Cr-1Mo loop is shown in Figure IV-20.

The photomicrographs presented for 316SS are adjudged to be typical of results obtained to December 31, 1961. Several effects were apparent in the illustrations and subsequently discussed. Noted were carburization, a band at the surface of 1000 F samples which is lighter in appearance, and a ferritic surface phase. Limited tensile data were taken early in the program and are included in graphic form.

The lighter band appearing in the samples of Figures IV-15, IV-16, and IV-17 has been termed "surface structure anomaly". This gradient appears real, since it can be reproduced with various etchants, and only appears in samples exposed to 1000 F temperature. Figure IV-21 plots the depth of penetration of the gradient versus time. A diffusion value of $D = 5.7 \times 10^{-11} \text{ cm}^2/\text{sec}$ was placed on the anomaly by incorporating metallographic measurements according to Fraenkel and Houben.⁽¹⁰⁾ The basic equation was $x = k(DT)^{\frac{1}{2}}$, where x is a metallographically measurable distance, k a frequency factor, D the diffusion rate in cm^2/sec , and T the time in seconds. Limited examinations incorporating X-ray fluorescence and X-ray diffraction indicated a chromium depletion at the surface.

An interesting observation is that ferrite is seldom found at the surface and then only in minute amounts at 1000 F. It may be possible then that the phenomenon is connected with the austenitic-to-ferritic transformation described below.

Surface phenomena at temperatures exceeding 1000 F, which involved transformation of the austenitic 316 stainless steel to ferrite, were encountered. The illustrations in Figures IV-22 through IV-25 indicate the effects of temperature and cold-work on the phenomenon. Precautions have

75X
316SS
LOOP #3, RUN 6
1200°F - 1527 HOURS

10% OXALIC-ELECTROLYTIC
3280
12PPM O₂ SODIUM

FIGURE IV-20

Sample from material illustrated in Figure III-5. Moderately sensitized, incipient ferritic layer. Intergranular ferrite to depth of 1 grain. Note that a Widmanstätten structure with heavier ferrite had developed at area with localized cold-work.

277-15

FIGURE IV-21 SURFACE STRUCTURE ANOMOLY 316 STAINLESS STEEL EXPOSED AT 1000°F

250X 10% OXALIC-ELECTROLYTIC
 316SS 3104
 LOOP #3, RUN 1 50PPM O₂ SODIUM
 1000°F - 700 HOURS

FIGURE IV-22

A slight Widmanstätten structure had developed at the locally deformed section on the upper portion. This had been caused by a shearing operation which had not had sufficient material machined away to remove all deformation. A very minute amount of ferrite appeared at the surface. Compare to standard sample A10M, Figure IV-14.

250X 10% OXALIC-ELECTROLYTIC
 316SS 3200
 LOOP #3, RUN 1 50PPM O₂ SODIUM
 1100°F - 2728 HOURS

FIGURE IV-23

A dark layer appeared at the surface to a depth of approximately 0.0012 inch. Carbide growth was massive intergranularly, however, localized attack was not present. An incipient ferritic layer appears at the extreme surface. Compare to Figure IV-14.

277-16

250X 10% OXALIC-ELECTROLYTIC 1401
 316SS
 LOOP #1, RUN 1P 50PPM O₂ SODIUM
 1300°F - 400 HOURS

FIGURE IV-24

A 0.0008 inch ferritic layer had developed on the sample. Micro-emission X-ray spectrometer analysis of a similar sample (1200) indicated chromium depletion at the outer surface with a corresponding increase in iron and nickel. X-ray diffraction (Debye-Scherrer) patterns of the surface layer indicated a body-centered cubic lattice of a more perfect nature than generally encountered in a low alloy ferritic steel. Note the cold worked structure associated with samples fabricated from material illustrated in Figure IV-12.

250X 10% OXALIC-ELECTROLYTIC 2300
 316SS
 LOOP #2, RUN 1 50PPM O₂ SODIUM
 1200°F - 2728 HOURS

FIGURE IV-25

Cold worked sample of material illustrated in Figure IV-12. A ferritic layer of about 0.0003-inch had developed.

277-17

been applied to prevent cold-work in the samples, however, it has been extremely difficult to insure because of the necessary fabrication procedures. The sheet stock as received has been subjected to a light, cold-rolled pass, on polished rolls in accordance with specification ASTM A240-2B finish (3). The major portion of cold-work has been found on the narrow dimension of samples resulting from subsequent fabricating methods. Dependency of the transformation upon the degree of cold-work is illustrated in Figure IV-25 where local deformation occurred, and in Figure IV-24 where deformation was found throughout the sample. Attempts to place values on the quantity of ferrite formed provided differing values in accordance with the oxygen level. The equations were in the Arrhenius form $k \exp - Q/RT$, where k is the frequency factor, Q the activation energy for a process in calories per mole, R the gas constant and T temperature in degrees Kelvin. For an oxygen level in the sodium of 50 parts-per-million, it was found that the thickness of the ferritic layer fitted the equation $5.6 \times 10^3 \exp - 51,000/RT$ cm, while at a level of 10 parts-per-million oxygen layer thickness was represented by $3.1 \times 10^3 \exp - 50,000/RT$ cm. Time dependency was not established. It is interesting to note that the frequency factor differed but the activation energy remained relatively constant for both equations. This would indicate diffusion dependency which corresponds to Epstein's conception of the process (6). Observations correspond to those of McKee (11) who found the transformation in his experiments with liquid sodium at 1500 F, and by others both in liquid sodium and other liquid metal environments (12), (13), (14). The austenitic-to-ferritic transformation is attributed to the selective leaching of elements. Elements selectively leached, normally permit the thermodynamically metastable austenite to exist at low temperatures.

Sigma phase was expected to develop in the 316 stainless steel as reported by the American Society for Metals Handbook (15); however, the phase has been found in only one sample. The sample (1300) was in the hot-rolled condition and had been exposed to liquid sodium environment at 1300 F for a period of approximately 4200 hours.

Intergranular attack has not been present in samples, however, massive carbide formations appearing intergranularly, adjacent to the surface, have allowed ferrite to form in these areas in some instances.

Carbon analysis of samples indicated that 316 stainless steel experienced significant carburization when a carbon source was provided. Both the monometallic 316SS Loop (#1) and the bimetallic 316SS-5Cr- $\frac{1}{2}$ Mo-Ti Loop (#4) have shown no significant carburization of the 316SS.

Carburization has occurred in the bimetallic 316SS-2 $\frac{1}{4}$ Cr-1Mo Loops #2 and #3. The inability of 2 $\frac{1}{4}$ Cr-1Mo to retain carbon, in the sodium environments employed at elevated temperatures, has been realized and described in the examination of the 2 $\frac{1}{4}$ Cr-1Mo alloy.

Carbon data from run 1 of Loops #2 and #3 were fitted to the Arrhenius equation, $k \exp(-Q/RT)$. The equation coefficients based on the limited data available are listed in Table IV-5.

TABLE IV-5

CALCULATED ARRHENIUS EQUATION COEFFICIENTS FOR CARBON DIFFUSION
IN BIMETALLIC LOOPS

Loop	O ₂ , ppm	Material	k	Q
2	10	2 $\frac{1}{4}$ Cr-1Mo	3.6×10^{-3}	$8,000 \pm 2,000$
		316SS	1.4×10^{-2}	$5,000 \pm 1,000$
3	50	2 $\frac{1}{4}$ Cr-1Mo	2.2×10^{-2}	$4,000 \pm 3,000$
		316SS	2.4×10^{-3}	$12,000 \pm 5,000$

Values obtained indicated the inability of the 316 stainless steel to accept carbon as rapidly as it was made available by the 2 $\frac{1}{4}$ Cr-1Mo. This was further substantiated by known diffusion rates of interstitial atoms in both austenite and ferrite (16), and by direct observation of increased deposition products in Loop #2. Correlation of the data checks fairly well with those of Hayes and Shepard (7) and Anderson and Sneesby (17). The

former describe the role of carbon activity in an austenitic-ferritic-sodium system, and the latter describe the carburization of austenitic stainless steel with carbon-saturated sodium.

Twenty-six tensile tests were performed on samples from Loop #1, Run 1P, the hot-rolled material illustrated in Figure IV-12. Results are presented in Figure IV-26 and indicate the decreasing strengths with increasing temperatures. It should be noted, and the data viewed accordingly, that the cold work provided high values for the unexposed materials.

FIGURE IV-26 ROOM TEMPERATURE TENSILE STRENGTH OF EXPOSED 316 SS. VS. 400 HOUR EXPOSURE AT TEMPERATURE LOOP 1 RUN 1P

Summary of 316 Stainless Steel Examinations.

The 316SS was subject to carburization when exposed in a bimetallic 316SS $2\frac{1}{4}$ Cr-1Mo System. Additional driving force for the process was provided with higher oxygen levels in the sodium environment. Surface effects were encountered whereby an austenitic-to-ferritic transformation was evident to minor depths at temperatures of 1100-1300 F. An apparent surface gradient anomaly was encountered with samples exposed at 1000 F. The surface of the samples indicated a 0.001 to 0.002-inch layer of chromium depletion which may be the first stages of the austenitic-ferritic transformation. No serious localized effects were observed.

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APPENDIX A

SUMMARY OF THE METALLURGICAL EXAMINATIONS OF SAMPLES FROM TEST RUNS COMPLETED BY 12/31/61

Results of the metallurgical examinations of the samples from each test run are tabulated in this appendix. The results are presented in numerical order of the test run number. Other pertinent run information such as loop material, $T_{\max}$, ΔT , oxygen level and the number of samples examined are presented. The figures and tables referenced herein are presented in the text of this report.

Appendix B presents some comparative photomicrographs of 316SS samples exposed to sodium. Appendix C presents a tabulation of the carbon analyses of samples exposed in runs reported in this appendix.

APPENDIX A

SUMMARY OF THE METALLURGICAL EXAMINATIONS OF SAMPLES FROM TEST RUNS COMPLETED BY 12/31/61

<u>Loop-Run</u>	<u>Material</u>	<u>T, max, °F</u>	<u>ΔT, °F</u>	<u>*Oxygen ppm</u>	<u>Samples Examined</u>	<u>Samples Removed 100's Hours</u>
1-1P	316SS	1300	500	50	25	4
<p>Samples installed in the loop were severely cold-worked. Grain structure elongation and sensitization was observed in all samples. Figure IV-12 is a photomicrograph of structure typical to the run. Figure IV-24 illustrates the ferritic layer developed on the cold-worked structure. The carbon content ranged from 0.040 to 0.065 percent.</p>						
1-1	316SS	1300	500	50	36 2($\frac{1}{2}$ -Inch pipe)	7,14,28
<p>Standard corrosion sample AlOM (Figure IV-14) represented the material condition of the samples with exception to samples 1107, 1478, 1607, 1914B, and 1915 which were represented by Al4M (Figure III-5). Samples associated with AlOM had a duplex grain structure, developed by an annealing operation on the 1/16-inch sheet stock. Al4M represents material in the as-received 1/16-inch sheet stock. Ferritic development at the surface of the samples decreased from the previous run and was incipient only at temperatures of 1100-1300 F. Ferrite was present at cold-worked edges of the samples where deformation had been caused by shearing. Nominal carbon content of the corrosion samples ranged from 0.040 to 0.065 percent with maximums at the 1100 F region.</p>						
1-1F	316SS	1300	500	50	2	4.8
<p>The run was initiated with a high plugging temperature in an attempt to trap crud in a filtering system. Run was terminated upon plugging of the loop. Too few samples were obtained to determine the significance of the higher oxygen level.</p>						

* Based on KAPL data, TID 5277, p. 7.

<u>Loop-Run</u>	<u>Material</u>	<u>T,max, °F</u>	<u>ΔT, °F</u>	<u>Oxygen ppm</u>	<u>Samples Examined</u>	<u>Samples Removed 100's Hours</u>
-----------------	-----------------	------------------	---------------	-----------------------	-----------------------------	--

1-2	316SS	1200	500	12	10	7,14,22
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Standard sample for this run was A24M (equivalent to A14M, Figure III-5. Ferrite formed at surfaces in 1100 to 1200 F regions to an approximate depth of 0.6 mils in 2165 hours. Also had 0.6 mil-thick ferrite (no positive identification but appearance same) layer in cold zone at 700 F.

2-1	316SS 2 $\frac{1}{4}$ Cr-1Mo	1200	500	10	21(316SS) 19(2 $\frac{1}{4}$ Cr-1Mo) 2($\frac{1}{2}$ -Inch pipe)	7,14,28
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316SS - A10M (Figure IV-14) provided the standard corrosion sample for the 316SS except the pipe sample which was compared to A1M (Figure IV-12). Ferritic development was incipient even at the upper temperature range. Carburization was more evident than experienced in the monometallic 316 loop and the nominal carbon content varied from 0.045 percent in the unexposed material to a maximum of 0.100 percent in samples from 1100-1200 F region. Greatest sensitization was observed in H2 and H3 samples, however, effects that could be termed catastrophic were not present.

2 $\frac{1}{4}$ Cr-1Mo - Standard corrosion sample was B11M (Figure IV-2) except B1M (Figure IV-1) served as standard for pipe sample 2800. The unexposed material, as well as samples from the 700 F region presented a pearlitic structure in a ferritic matrix with surface decarburization to ~4 mils. Samples from the 1000-1100 F region had undergone an isothermal pearlitic decomposition so that carbides were present only in a spheroidized form. Carbon content varied from 0.105 percent in the standard to 0.035 percent in the 1100 F region. Tensile data on two samples from the 1100 F region and one from the 700 F region are reported in Table IV-2.

2-2	316SS 2 $\frac{1}{4}$ Cr-1Mo	1200	500	15	3(316SS) 3(2 $\frac{1}{4}$ Cr-1Mo)	7
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This run was initiated to study the effects of high velocity (20 ft/sec) on the corrosion rate as planned by the statistical test design. Metallographic observations were based on standard sample A12M (similar to

<u>Loop-Run</u>	<u>Material</u>	<u>T,max, °F</u>	<u>ΔT, °F</u>	<u>Oxygen ppm</u>	<u>Samples Examined</u>	<u>Samples Removed 100's Hours</u>
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2-2 (continued)

Al4M, (Figure III-5) for the 316SS and B33M (Figure III-3) for the $2\frac{1}{4}$ Cr-1Mo. Results were comparable with the previous run in that the major phenomenon was carbon transport with the $2\frac{1}{4}$ Cr-1Mo providing the source and the 316SS acting as a sink.

2-3	316SS $2\frac{1}{4}$ Cr-1Mo	1200	500	13	3(316SS) 3($2\frac{1}{4}$ Cr-1Mo)	7
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The run was provided to produce data in the manner dictated by the statistical replicate program for low velocities (9 ft/sec). Metallographically the results were very similar to the previous run.

2-4	316SS $2\frac{1}{4}$ Cr-1Mo	1200	500	11	3(316SS) 3($2\frac{1}{4}$ Cr-1Mo)	28
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316SS - Ferrite formation on surface at 1100 and 1200 F with maximum thickness at 1200 F of 0.4 mils. Light sensitization at 1000 F; heavily sensitized throughout at 1100 and 1200 F.

$2\frac{1}{4}$ Cr-1Mo - Little change noted metallographically. Carbides spherodized.

3-1	316SS $2\frac{1}{4}$ Cr-1Mo	1200	500	50	17(316SS) 16($2\frac{1}{4}$ Cr-1Mo) 2($\frac{1}{2}$ -Inch pipe)	7, 14, 28
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316SS - AlOM (Figure IV-14) provided the standard corrosion sample for the 316SS except that the pipe sample was compared to AIM (Figure IV-12). The surface ferrite was measurable in some instances (Figure IV-23) but generally appeared incipient. Duplex grain size and sensitization were noted as in run 2-1. Sensitization occurred to a much greater extent in areas where shearing had caused localized deformation and a Widmanstätten structure was formed. Nominal carbon content ran from 0.045 percent in the standard material to 0.105 percent for a 2800 hour - 1100 F high velocity sample. Serious effects such as intergranular attack were not observed.

<u>Loop-Run</u>	<u>Material</u>	<u>T,max. °F</u>	<u>ΔT. °F</u>	<u>Oxygen ppm</u>	<u>Samples Examined</u>	<u>Samples Removed 100's Hours</u>
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3-1 (continued)

2 $\frac{1}{4}$ Cr-1Mo - Both heat-treated and cold-worked samples were run. The heat-treated samples were compared to standard corrosion sample B1M (Figure IV-2) and therefore were of a pearlitic nature with surface decarburization. Samples 5700, 5701, 3702, 5900, 5901, 5903 and 3905 were of the original cold-rolled stock and were represented by standard sample B1M (Figure IV-1). Nominal carbon content ranges were 0.105-0.053 percent. A small number of tensile tests were performed. Results are indicated in Table IV-2. No localized serious effects were observed.

3-2	316SS 4S4/4S8	1200	500	10	14(316SS) 6(4S4)/6(4S8)	7
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Two small sheets of experimental, carbide-stabilized 2 $\frac{1}{4}$ Cr-1Mo alloy were received for testing. The chemical composition is shown in Table III-3. Samples of the 4S4 alloy were installed in the 700 and 1000 F region of the cold leg while the 4S8 was exposed to temperatures of 700 and 1100 F in the cold leg.

316SS - Metallographic examination indicated sensitization to a greater extent than observed in the previous run. Standard sample A14M (Figure III-5) was representative of the unexpressed microstructure. Carbon content of the samples ranged between 0.071-0.101 percent whereas the material originally contained 0.045 percent carbon.

4S4 and 4S8 - The only change noted in samples from the 4S4 and 4S8 alloys was a slight growth in the carbides. Nominal carbon content in the 4S4 was 0.126-0.129 percent whereas the original content had been 0.14 percent. The 4S8 was similar with a 0.124-0.130 percent carbon content and had an original analysis of 0.14 percent.

<u>Loop-Run</u>	<u>Material</u>	<u>T, max, °F</u>	<u>ΔT, °F</u>	<u>Oxygen ppm</u>	<u>Samples Examined</u>	<u>Samples Removed 100's Hours</u>
3-3	316SS 2 $\frac{1}{4}$ Cr-1Mo	1200	500	10	3(316SS) 3(2 $\frac{1}{4}$ Cr-1Mo)	7

A seven-sample (six corrosion samples, one 6-mil foil) run to correlate the effects of low (7 ft/sec) velocity. Standards for the samples were comparable to Al4M (Figure III-5) for the 316SS and B33M (Figure IV-4) for the 2 $\frac{1}{4}$ Cr-1Mo. Observations of metallographic samples indicated a carbon shift from the 2 $\frac{1}{4}$ Cr-1Mo to the 316SS. No serious localized effects were noted.

3-4	316SS 2 $\frac{1}{4}$ Cr-1Mo	1200	500	10	3(316SS) 3(2 $\frac{1}{4}$ Cr-1Mo)	7
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A seven-sample run to check the effects of high (20 ft/sec) velocity. Results were comparable to the previous run.

3-5	316SS 2 $\frac{1}{4}$ Cr-1Mo	1200	500	10	3(316SS) 3(2 $\frac{1}{4}$ Cr-1Mo)	7
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Run conditions and results very similar to the previous run.

3-6	316SS 2 $\frac{1}{4}$ Cr-1Mo	1200	500	10	4(316SS) 4(2 $\frac{1}{4}$ Cr-1Mo)	15
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The run was similar to 3-4 and 3-5 with exception to time. Six corrosion samples were examined with results similar to the three previous runs. A localization of carbides at the grain boundaries of the 2 $\frac{1}{4}$ Cr-1Mo was slightly more prevalent than observed in the 700-hour runs. Two pipe samples, one of each material, were removed after an accumulated test exposure of 5244 hours. The 316SS and 2 $\frac{1}{4}$ Cr-1Mo pipe samples were of the cold-worked structure comparable to the standard AIM (Figure IV-12) and BIM (Figure IV-1) respectively. No serious effects were observed in either sample; carbon shift was the only significant change.

4-1	316SS 5Cr- $\frac{1}{2}$ Mo-Ti	1200	500	50	17(316SS) 16(5Cr- $\frac{1}{2}$ Mo-Ti) 2($\frac{1}{2}$ -Inch pipe)	7,14,28
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The run was standard with respect to test conditions. Carbon transport was almost negligible when compared to the 316SS-2 $\frac{1}{4}$ Cr-1Mo loops.

<u>Loop-Run</u>	<u>Material</u>	<u>T,max, °F</u>	<u>ΔT, °F</u>	<u>Oxygen ppm</u>	<u>Samples Examined</u>	<u>Samples Removed 100's Hours</u>
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4-1 (continued)

316SS - Samples were of the type associated with standard A10M (Figure IV-14), the duplex grained material. Sensitization normal to temperatures involved was observed as well as ferritic transformation of deformed areas. No serious localized effects were noticed. Little carburization was encountered as shown by the range 0.045-0.055 percent.

5Cr- $\frac{1}{2}$ Mo-Ti - Samples were of the type associated with standard sample C11M (Figure III-4). No apparent changes in microstructure were observed, however, intergranular penetration, dependent on temperatures exceeding 700 F was present. The penetration extended to a maximum depth of 1.0 mil after a four-month exposure at 1100 F and seemed to be arrested at that point. Table IV-3 tabulates the penetration values.

4-2	316SS	1200	500	10	17(316SS)	7,14,28
	5Cr- $\frac{1}{2}$ Mo-Ti				18(5Cr- $\frac{1}{2}$ Mo-Ti)	

Test conditions were similar to the previous run with exception of the addition of a radio-active tracer tab. Condition of the 316SS samples was improved in that the standard for comparison was similar to A14M (Figure III-5).

316SS - Samples exposed in the 1100-1200 F were heavily sensitized. 2800-hour samples from that temperature region had undergone sensitization until some intergranular ferrite had developed (Notably sample 4276, 2803 hours at 1100 F had developed intergranular ferrite throughout.)

5Cr- $\frac{1}{2}$ Mo-Ti - As experienced in the previous run, no changes in microstructure were detected. Intergranular penetration was present in the 1000-1100 F samples to a maximum depth of 1.0 mil.

4-3	316SS	1200	500	10	0	7
	5Cr- $\frac{1}{2}$ Mo-Ti					

The run was designed to study the "down-stream effect". All sample holders in the hot leg were held at a temperature of 1200 F while the cold leg holders were run at 700 F. Examination was not performed on the samples.

<u>Loop-Run</u>	<u>Material</u>	<u>T,max, °F</u>	<u>ΔT, °F</u>	<u>Oxygen ppm</u>	<u>Samples Examined</u>	<u>Samples Removed 100's Hours</u>
5-1	2 $\frac{1}{4}$ Cr-1Mo	1200	500	50	37 2($\frac{1}{2}$ -Inch pipe)	7,14,28

The run was basically standard and the standard sample for comparison was B11M (Figure IV-2). Significant changes in samples were isolated to an isothermal decomposition of pearlite to spheroidized carbides and the accompanying carbon transport. Upon spheroidization of the carbides, a tendency for preferred grain boundary location occurred -- see Figure IV-8. Nominal carbon content of the samples ranged between 0.115-0.035 percent with the lowest value being recorded in the 1200 F region of the loop.

5-2	2 $\frac{1}{4}$ Cr-1Mo	1200	500	10	12	7,14,30
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A run duplicating conditions set for the previous run with exception to oxygen. The run had not been completed at the date of this report thereby excluding 3000-hour data. Standard sample was comparable to B33M (Figure IV-4). Results of the run were again limited to observance of the carbon shift previously noted.

6-1	5Cr- $\frac{1}{2}$ Mo-Ti	1200	500	50	35 2($\frac{1}{2}$ -Inch pipe)	7,14,28
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The run was designed as a standard four-month test. Sample comparison was established with C11M (Figure 111-4). Little effect of the environment on the microstructure was noted, however, intergranular penetration developed in the 1100-1200 F temperature region. Table IV-3 tabulated depth of the intergranular attack, and Figure 11-A and 11-B illustrated the effect. No significant carbon shift was noted.

6-2	5Cr- $\frac{1}{2}$ Mo-Ti	1200	500	10	45	7,14,28
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A duplication of the previous run with exception to a lower oxygen level. Results were comparable to those of the previous run. The lower oxygen level did not appreciably alter the effect of intergranular penetration.

APPENDIX B

EFFECTS OF EXPOSURE OF 316 STAINLESS STEEL TO LIQUID SODIUM

Appendix B contains photomicrographs showing effects of exposure of 316SS to liquid sodium in which bright field illumination, polarized light and higher magnification are used to better illustrate growth of the anomolous surface band at 1000 F, and carbon pick-up. The samples photographed for this appendix are also illustrated as Figures IV-14, IV-15, IV-16, IV-17, IV-18, and IV-19 (a, b, c, and d) together with the results of exposure, in the report section of this document.

The illustrations presented herein have been reduced 54.5% from the original magnification for the purposes of reproduction and inclusion in this report.

EFFECTS OF EXPOSURE OF 316 STAINLESS STEEL TO LIQUID SODIUM (10PPM O₂)

SAMPLE NO.
TIME
TEMPERATURE

75X
BRIGHT FIELD
ILLUMINATION

75X
POLARIZED
ILLUMINATION

750X
BRIGHT FIELD
ILLUMINATION

A10M
AS RECEIVED
CARBON 0.045%

2100
651 HOURS
1000°F
CARBON 0.070%

2101
1438 HOURS
1000°F
CARBON 0.075%

2175
2728 HOURS
1000°F
CARBON 0.085%

10% OXALIC-ELECTROLYTIC

REDUCED 54.5%

MODIFIED MURAKOMI'S
REAGENT

277-1

EFFECTS OF EXPOSURE OF 316 STAINLESS STEEL TO LIQUID SODIUM (10PPM O₂) - CONT.

SAMPLE NO.
TIME
TEMPERATURE

75X
BRIGHT FIELD
ILLUMINATION

75X
POLARIZED
ILLUMINATION

750X
BRIGHT FIELD
ILLUMINATION

2200
2728 HOURS
1100°F
CARBON 0.09%

2475
651 HOURS
1200°F
CARBON 0.075%

2476
1437 HOURS
1200°F
CARBON 0.075%

2503
2131 HOURS
1200°F
CARBON 0.055%

2502
2728 HOURS
1200°F
CARBON 0.065%

10% OXALIC-ELECTROLYTIC

MODIFIED MURAKAMI'S
REAGENT

REDUCED 54.5%

277-2

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APPENDIX C

CARBON ANALYSIS OF EXPOSED SAMPLES

Average carbon content of the exposed samples has been tabulated. The table indicates time and temperature of exposure, velocities, materials, increasing and decreasing temperatures and oxygen levels in the sodium. The analyses were performed with a Leco Semi-Automatic Gaseometric Analyzer, calibrated 0.005 to 1.500 percent. Check analysis indicated an accuracy of ± 0.003 percent, however, values have been reported to 0.001 percent. Analysis was not completed for Run 3 of Loops 3 and 4 and are therefore not included in the table.

APPENDIX C
PERCENT CARBON (AVERAGE)
OF SAMPLES EXPOSED TO FLOWING SODIUM

TEMP. OXYGEN VEL. TIME				OXYGEN VEL. TIME				TEMP. OXYGEN VEL. TIME				TEMP. OXYGEN VEL. TIME								
°F PPM FPS HRS.				PPM FPS HRS.				°F PPM FPS HRS.				°F PPM FPS HRS.								
LOOP NO. 1				RUN P 400				RUN 1 800 1500 2200 3000				RUN 2 700 1400								
316SS (.045)	1100	50	18/6		50	12/4			.054	.052	.042	.056	1000	12	21/7	.055	.051			
	1200		18/6	.058		12/4				.059	.054		1100		21/7					
	1300		18/6	.046		12/4		.048	.038	.048	.051		1200		21/7	.057	.048			
316SS (.045)	1200		6	.065		4		.050	.051	.045	.045		1100		7					
	1100		6	.060		4				.061	.046		1000		7					
	800		6	.065		4		.045	.046	.050	.044		700		7	.049	.050			

°F OXYGEN VEL. TIME HRS.				OXYGEN VEL. HRS.				OXYGEN VEL. HRS.			
LOOP NO. 2				RUN 1 700 1400 2100 2800				RUN 2 700			
316SS (.045)	1000	10	21/7	.070	.075	.085	.090	15	20	.074	
	1100		21/7			.090	.092			.082	
	1200		21/7	.075	.065	.060	.055				.065
2 1/4 Cr - 1 Mo (.105)	1100		7		.040	.035	.035				.078
	1000		7				.065				.107
	700		7	.090	.090	.092	.098			.113	.111

		°F OXYGEN VEL.		TIME HRS.				OXYGEN VEL. HRS.				HRS. OXYGEN VEL.				HRS. OXYGEN VEL. HRS.			
LOOP NO. 3		RUN 1		700	1350	2000	2700	RUN 2		700	700	RUN 4		700	RUN 5		700		
316SS (.045)	1000	50	15/5				.100 ↓	.092	10	17/6	.072 ↓		.072 ↓	10	20	.083	12	27	.081
	1100		15/5				.105 ↓	.090		17/6	.088 ↓		.088 ↓			.086			.089
	1200		15/5	.065 ↓	.065 ↓	.055 ↓	.080 ↓	.065	THIS RUN	17/6	.101 ↓	THIS RUN	.101 ↓			.093			.089
2 1/4 Cr - 1 Mo (.105)	1100		5				.053 ↓		458	6	.127	454			.058			.063	
	1000		5				.108 ↓		(.14)	6		(.14)	.129			.095			.103
	700		5	.100 ↓	.100 ↓	.085 ↓	.108 ↓			6	.129		.130			.111			.114

APPENDIX C CONT.

LOOP NO. 4	°F OXYGEN VEL.			TIME HRS.				OXYGEN VEL.			TIME HRS.				°F OXYGEN VEL. HRS.		
	RUN 1			700	1400	2100	2800	RUN 2			700	1400	2100	2800	RUN 4		
316SS (.045)	1000	50	12/4				.055 / .052	10	11/4			.052 / .048	.057 / .041		1200	10	4 .055
	1100		12/4				.055 / .050		11/4				.051 / .053		1200		.053
	1200		12/4	.045 / .050	.045 / .045	.040 / .040			11/4		.054 /		.050 / .050		1200		.053
5 Cr - 1/2 Mo - Ti (.094)	1100		4				.110		4		.093 / .095	.100 /			800		.108
	1000		4				.103		4				.094 /		700		.102
	700		4	.110	.098	.103	.100		4		.093 / .090	.087 /			700		.102

LOOP NO. 5	°F OXYGEN VEL.			TIME HRS.				OXYGEN VEL.			TIME HRS.				°F OXYGEN VEL. HRS.		
	RUN 1			600	1350	2100	2700	RUN 2			700	1400					
2 1/4 Cr - 1 Mo (.105)	1000	50	12/4		.092 /		.110 / .110	10	15/5		.115 / .119						
	1100		12/4				.105 / .098		15/5								
	1200		12/4	.035 / .050	.035 / .050	.055 / .050			15/5	.055 / .062	.032 / .048						
2 1/4 Cr - 1 Mo (.105)	1100		4		.115		.125		5	.120 / .122							
	1000		4				.102		5								
	700		4		.083	.085	.083		5	.117 / .113							

LOOP NO. 6	°F OXYGEN VEL.			TIME HRS.				OXYGEN VEL.			TIME HRS.				°F OXYGEN VEL. HRS.		
	RUN 1			700	1400	2100	2800	RUN 2			350	700	1100	1400	2100	2800	
5Cr - 1/2 Mo - Ti (.094)	1000	50	15/5		.102 /		.110 / .105	10	13/4		.100 / .096			.099 / .094	.107 / .107		
	1100		15/5				.105 / .098		13/4						.119 / .125		
	1200		15/5	.105 / .125	.115 / .112	.110 / .120	.120 / .120		13/4	.103 / .094	.119 / .109	.106 / .116	.116 / .119	.122 / .117	.117 / .117		
5 Cr - 1/2 Mo - Ti (.094)	1100		5		.105		.112		4	.092 / .105	.094 / .112						
	1000		5				.105		4						.105		
	700		5	.102	.094	.095	.095		4	.096 / .096			.098 /				

As received carbon is shown below each alloy designation; temperature of exposure is given in order of occurrence in each loop; three temperatures in the hot-leg sample holders are listed first followed by the three cold-leg sample holder temperatures; vertical columns represent successive down-stream positions; the nominal oxygen content in PPM of the sodium is given for each run; sodium velocities are given in feet per second; values at different velocities are separated by a slant-line with the higher velocity value given on the upper-left.