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**SODIUM TECHNOLOGY DEVELOPMENT PROGRAM
MASS TRANSFER INVESTIGATION
IN LIQUID METAL SYSTEMS
QUARTERLY PROGRESS REPORT NO. 13
MARCH 1970 - MAY 1970**

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**SODIUM TECHNOLOGY DEVELOPMENT PROGRAM
MASS TRANSFER INVESTIGATIONS IN LIQUID METAL SYSTEMS**

Quarterly Progress Report No. 13
March 1970 - May 1970

Approved:

R. S. Young
R. S. Young, Project Engineer
Sodium Mass Transfer Program

Approved:

E. L. Zebroski
E. L. Zebroski, Manager
Development Engineering Section

This document is
PUBLICLY RELEASABLE

James A. Kelly
Authorizing Official

Date: 9/12/07

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1. INTRODUCTION AND PROGRAM SUMMARY

1.0 TECHNICAL HIGHLIGHTS

Changes in mechanical properties of cladding due to exposure to flowing sodium are about as big as the changes due to radiation effects. Data on such changes must be obtained and taken into account quantitatively in rational fuel design. Similar data is not available from fuel testing in EBR-II.

The results of a series of long-term stress-rupture tests of Types-304 and 316 stainless steel in 1150°F and 1300°F sodium are given in Section 4. The projected reduction in rupture strength for Types 304 and 316 stainless steel after 30,000 hours in 1300°F sodium compared to argon are 60 and 53%, respectively. Rupture ductilities have been reduced by 50% after 3500 hours exposure, although Type-304 stainless steel shows a large reduction in rupture strength at 1300°F, and a smaller reduction at 1150°F.

Major Transport of Carbon, Nitrogen, and Silicon is Observed in All Austenitic Systems

A gross loss of interstitials, particularly carbon is found (up to 73% bulk loss for Type 304 stainless steel and 48% loss for Type 316 stainless steel after 4800 hours at 1300° 400°F ΔT). No loss of carbon was recorded from stabilized grades of stainless exposed in the same loops. These findings contribute to the selection of stabilized stainless grades (Types 321 and 347 stainless steel) as prime candidates for core components in the demonstration plant.

Particulates in stainless steel loops are found to be mostly under 10 microns in size in the cold leg. These data provide a conceptual basis for design of cleanup systems that may be applicable to reactor circuits, and which may reduce deposition significantly.

Investigation of the effect of pore size of in-stream micrometallic filters in the range 5 to 10 microns on efficiency of pick up of particles from the sodium is reported in sections 2.5 and 3.1. The 10 micron pore size filters collected almost no material in the low temperature region of the loop (600°F) compared to about 1 gram of material normally found in the smaller pore size filters. Particulates measured on surfaces at 1100°F are 2 to 3 microns in size.

A device was completed for calibrating oxygen and hydrogen meters. This will be the first device available within the National Program for oxygen-meter calibration capable of accounting for the interaction of hydrogen with oxygen and correlating both with cold trap and plugging temperatures.

Preliminary work supporting test loop operations that requires oxygen control (within ± 2 ppm) in the 2 to 20 ppm range is reported. Two air-reference oxygen cells and a hydrogen meter (vacuum type)* are mounted in a magnetically stirred isothermal pot with accessories for calibration and gettering (see Figures 2 and 3).

Extensive calibrations of oxygen meters have been carried out. The results of successive calibration runs on an oxygen meter in loop 8 are given in section 2.9. Significant changes in calibration continue to occur with Cu-Cu₂O, paralleling results with air-reference electrodes.

1.1 PROGRAM DESCRIPTION AND BACKGROUND

Corrosion of candidate structural materials, the influence of corrosion and of impurities in the sodium on engineering properties of materials, are being studied in test loops designed to simulate typical conditions of sodium cooled reactors. The program is designed to obtain out-of-pile test data that will support engineering design selections (when used in conjunction with irradiation data) and the establishment of specifications for impurity monitoring and control for large plants. The work is in support of the Liquid Metal Fast Breeder Reactor (LMFBR) and the Fast Flux Test Facility (FFTF) development programs.

In an earlier 6-year phase of the program ending in 1965, corrosion and mass transfer of Type 316 stainless steel and 2-1/4Cr-1Mo and 5Cr-1/2Mo-1/2Ti ferritic steels were studied in depth at sodium temperatures to 1200°F. More than 2500 samples of these materials were exposed to sodium during a total of 140,000 hours of loop operations. Average corrosion rates were correlated by statistical analysis from which quantitative relations were derived. The data were correlated with the variables of temperature, sodium velocity, sodium impurity content under cold trap control, and type of material.

In the current phase of the program, the number of materials studied has been extended to include cladding candidates for oxide fuel (Types 304, 316, and 347 stainless steels and Incoloy 800). The temperature range of testing has been

* Discovered in this program.

increased to 1300°F, and temperature profiles, loop geometry, and sodium purity controls have been established to represent environments of large fast reactors in an improved way.

Increasing emphasis has been placed on improving sodium impurity monitoring and control. In-line devices are used to obtain rapid and, where possible, continuous indications of impurity changes. These include chromatography for cover gas, replaceable flow-through sodium samplers, electrochemical cells for oxygen activity and diffusion cells for hydrogen concentration in sodium. Provisions are being made for electrochemical cells for carbon.

Cold traps for control of sodium purity in test loops have been modified with electromagnetic stirring devices to increase cleanup rate and automatic controls are being added for continuous and unattended operation.

1.2 SUMMARY OF TASKS

The sodium Mass Transfer Program is organized into five tasks. These tasks and their objectives are as follows:

Task I: Corrosion and Deposition

Objectives are improvements of empirical and rational models for the accurate prediction of corrosion and mass transport for design of primary and secondary sodium systems of large plants.

Task II: Sodium Impurities Determinations and Particulate Impurity Identification

This task has the objectives of developing improved methods for sampling of sodium systems, impurity monitoring, collection and identification of particulate material, and chemical and structural analysis. An operating objective is to provide well-controlled operation of loops and to support the development of rational models.

Task III: Inactive

Task IV: Corrosion and Mass Transport Effects on Composition, Structure, and Structural Integrity of Candidate Alloys for LMFBR's and Localized Attack

The objective is to obtain exposures of specific LMFBR candidate materials, including Types 304 and 316 stainless steel tubing, and to determine correlations between mass transfer and sodium impurity effects which affect long term stress rupture properties.

Task V: Large System Correlations

The purpose of this task is the correlation of corrosion data from a large sodium system with predictions from corrosion models developed in Task I. Sample holder assemblies are installed in the Sodium Components Test Installation (SCTI) for this purpose.

2. TASK I: CORROSION-DEPOSITION STUDIES

2.1 GENERAL

This work is directed toward the following:

- experimental study of the corrosion of steel, including Types 304 and 316 stainless, and deposition occurring in pumped sodium systems at top sodium temperatures to 1150°F and 1300°F and at a ΔT of 400°F to 500°F similar to large reactor systems
- development of an understanding of the mechanisms involved in the phenomena of corrosion, mass-transport, and deposition in small test loops so that extrapolations can be made to larger systems
- monitoring of the test systems for localized attack
- establishment of relationships between sodium impurities, loop operations, and metallurgical effects
- using heaters employing heat flux of 0.5 to 1.0×10^6 Btu/h-ft² peak in sodium to measure the effects of reactor heat flux on materials

The work is carried out in test loops constructed to simulate corrosion conditions of a 1000 MW coolant system and a closed loop system for FFTF.

2.2 OPERATION OF TEST FACILITIES

A summary of loop operations including conditions of tests and operating times is shown in Table 1 for the 3-month period ending February 20, 1970 through May 22, 1970.

2.3 LOOP 8 PARAMETER STUDIES (Run 8-6)

The purpose of this run is to determine corrosion and mass transfer data, (especially carbon movement), at high oxygen levels and to obtain the following information relative to the chemistry of the system:

1. Hydrogen oxygen interaction effects
2. Cold trap efficiency with respect to oxygen removal
3. Oxygen meter calibration at high oxygen levels in sodium and comparison of results with on-line vacuum distillation results.

During this period, the following activities were carried on in connection with this loop:

1. Calibrated and checked out on-line monitoring equipment while operating loop at 900°F maximum. This included on-line distillation, oxygen cell, and plugging meter. On-Line chromatograph was arranged to sample the cover gas alternately from Loops 8 and 9 cold leg surge tanks.
2. A test run (Run 8-6P) was completed after 604 hours at 1300°F using Type 316 stainless steel samples as monitors for continued carbon activity subsequent to Run 8-5. At the conclusion of this interval, nine selected tabs were removed for examination and replaced with fresh tabs of the same material. Preliminary examination of samples shows slight carbon decoration of slip planes but no evidence of the high carbon activity which appeared in Run 8-5.

2.4 LOOP 9 OPERATION

The purpose of the current test (Run 9-2A) is to obtain corrosion data at higher oxygen levels (~425°F) and to determine relationships between cold trap operation and changes in the chemistry of the system.

Operations at Run 9-2A temperature and flow conditions were carried out from February 20, 1970 to April 13, 1970, when a valve leak required shutting down the loop for repair. Operation at reduced temperature has been maintained since April 28, 1970, while adjusting the loop impurity level to a low value prior to moisture addition tests. During this 3-month period, the following activities were carried out:

1. Cold trap efficiency studies were made of effect of increased Reynolds number with respect to removal of hydrogen and oxygen species introduced in discrete steps—such as via cold trap, teabag, or cover gas additions. Figure 1 shows the installation of the electromagnetically stirred cold trap (cold trap No. 2) in parallel with the original (unstirred) trap. As reported previously in Quarterly 12, the improvement in impurity removal rate was monitored with a diffusion tube hydrogen detector and an electrochemical oxygen cell.
2. Equipment was installed and checked out to provide continuous addition of moisture to loop cold leg. The purpose of this is to establish constant O₂ and H₂ concentrations in the loop for further cold trap effectiveness studies. At conclusion of these studies, the present run will be terminated.
3. Preparations for Run 9-3 were begun, including:
  - a. Modification of the design for the high flux heater test section.
  - b. Fabrication of approximately 60% of the corrosion samples.
  - c. Initiation of steps for procuring spare heaters and test section materials.

The main objective of Run 9-3 is to obtain high precision, definitive measurements of the effect of high heat flux (0.8×10^6 Btu/hr/ft²) on corrosion at low oxygen conditions. Higher precision measurements involve:

- a. Close control of oxygen (± 2 ppm) measured by chemical analysis and monitored by the O₂ meter and plugging indicator.
- b. Corrosion determination by weight change of removable samples placed over the heated zone (i.e., heater anode).
- c. Comparison of high flux corrosion sample weight and material changes with that of unheated specimens exposed to sodium in an isothermal zone at comparable temperatures and velocity.

Table 1

SUMMARY OF LOOP OPERATIONS

February 20, 1970 to May 22, 1970

	Temp (°F)	ΔT (°F)	Flow (gpm)	T_s or CT (°F)*	Time (hr)
Run 8-6	1300	400	5.7	< 470	2,000
Three-Month Operation					1,610
Cumulative, Run 8-6P				< 300	867
Total Loop Age					24,998
Run 9-2A	1300	500	5.0	425	~4,000
Three-Month Operation					1,821
Cumulative, Run 9-2A					4,125
Total Loop Age					18,357
Run 1R-3	1300	500	2.0	400	2,000
Three-Month Operation					-0-
Cumulative, Run 1R-3					-0-
Total Loop Age					6,027
Run 4R-2	1300	500	6.5	< 300	10,000
Three-Month Operation					2,183
Cumulative, Run 4R-2			—	1000°F and above 1300°F and pressure applied	10,955 10,569
Total Loop Age					12,856
Run 5R-5	1300	500	10.0	< 300	5,000
Three-Month Operation					1,031
Cumulative, Run 5R-5			—	1000°F and above 1300°F and pressure applied	3,037 2,850
Total Loop Age					18,298
Run 6R-4	1150	400	7.0	< 300	10,000
Three-Month Operation					2,184
Cumulative, Run 6R-4			—	1000°F and above 1300°F and pressure applied	6,958 6,620
Total Loop Age					13,696

• — Plugging Temperature

CT — Temperature of Cold Trap

AIR REFERENCE
OXYGEN CELL

ORIGINAL COLD
TRAP (NO. 1)

STIRRED COLD
TRAP (NO. 2)

ELECTROMAGNETIC
STIRRER

Figure 1. Stirred Cold Trap—Loop 9

2.5 LOOP 1R PARTICULATE STUDY

The objective of this test is to generate, collect, and analyze relatively large quantities of particles of corrosion products under controlled conditions, and to develop a basic understanding of their role in sodium mass transfer.

During the last run (1R-2), the 2-5 micron micrometallic filter was replaced with a 7-10 micron pore size filter. After ~1200 hours of operation, the filter was removed for examination. The filter did not collect any particulates. This observation implies that the size distribution of the particles in suspension in sodium is less than 10 microns at 600°F. The size of particles deposited in higher temperature regions (~1100°F) was about 2 to 3 microns.

During this period, the loop underwent modifications in preparation of Run 1R-3 at high oxygen. These included:

1. Design of an oxygen meter loop containing two cells. This loop will be located in parallel with the cold leg
2. Fabrication of a new load of corrosion samples
3. Procurement of Marman flanges and their installation on the sample zone piping in the hot leg
4. Removal of cold trap, its cleanout and subsequent installation in the loop with excess Na₂O inventory for maintaining high oxygen level during the run
5. Replacement of defective trim heater
6. Fabrication of parts for the oxygen meter and loop preparations for its installation
7. Procurement of filters (5 micron, sintered nickel)
8. Design of controls and instrumentation for the O₂ meters (temperature and flow) and procurement of hardware for this purpose.

NOTE: It is planned to employ instruments being obtained from APDA for indicating and monitoring the cell output.

2.6 H₂-O₂ EQUILIBRIA STUDY

The objective of this study is to determine the effects of hydrogen in sodium on oxygen activity (and vice versa). The overall aim is to obtain information which will enable better interpretation of data obtained from on-line instrumentation and samplers (O₂ meter, H₂ meter, carbon meter, plugging indicator, chromatograph, and distillation), and better application of these devices, as well as purity control devices (cold trap and hot trap), for establishing and maintaining the levels of these impurities in sodium loops.

During this period, the stirred isothermal pot was completed, (Figures 2 and 3). Connections to Loop 6R were made for initial fill and conditioning prior to conducting the objective tests. During this conditioning period, measurements of oxygen activity in Loop 6R will be determined and comparison made later with output of a permanently installed oxygen meter to be installed in the loop purification leg.

On May 22, 1970, the initial sodium fill of the pot was made.

2.7 LOOP PURITY CONTROL

Loop cold traps have been controlled with semiautomatic temperature control equipment originally developed in Phase 1 for standardizing plugging runs (see GEAP-4837, pp. 121-122). This equipment was an improvement over the prior methods of cold trap control since it enabled full time operation within narrower limits than was formerly possible by manual adjustments. Nevertheless, the equipment lacked features necessary for precise control of loop purity.

Close control of loop oxygen is of utmost importance to reduce the error in corrosion data. As a consequence, a new controller system has been developed to overcome the following disadvantages in the existing (older version) equipment:

1. Lag in response time causing temperature overshoot and undershoot.
2. Wide deadband. For the overall system, this amounts to ±10°F, partly due to mechanical component wear and backlash in the recorders, which contain feedback slidewires.
3. Cold trap temperature excursions from failure of brush-type motors during unattended operation. These were originally selected to provide variable capacity blowers in order to accommodate the wide flow range in cooling plugging indicator and cold trap (same units are used for both operations).
4. Inability to ramp temperature from an initial high value to a lower specified (Run Plan) value without operator attention.
5. Limited capability to control auxiliary apparatus in response to inadvertent temperature (or flow) excursions.

Figure 2. Stirred Isothermal Pot for Meter Calibration

FIGURE 3. Stirred Isothermal Pot for Meter Calibration, Completed Assembly

The new system overcomes these disadvantages and has features which increase its flexibility while providing closer control and surveillance of cold trap temperature. Some of these are:

1. The new system is basically all solid state, except for the ramp timers, thereby providing greater freedom from mechanical failures and component wear and backlash (slidewires, indicators, cam-type switches, etc.).
2. A recorder monitors temperature and flow and is not part of the control system. All limit and feedback circuitry is external to the recorder. This overcomes mechanical problems mentioned above.
3. The controller deadband is $\pm 1^\circ\text{F}$.
4. The new system has a variable ramp-down and ramp-up function which is adjustable from 5°F/hr to 50°F/hr .
5. Temperature controllability is $\pm 2^\circ\text{F}$ over a range from 0°F to 1000°F . System oscillation is expected to be less than $\pm 1^\circ\text{F}$ about the set point.
6. Adjustable range relays on both the temperature and flow channels provide logic for controlling the application of heaters or isolating valves in the event of excursions beyond pre-set values. (This means that there is, for example, built-in capability for isolating the cold trap from the loop by closing the sodium valves, in the event of an inadvertent upward temperature excursion.)

The first of these systems has been installed in Loop 8 and is now controlling cold trap No. 1. These systems will be extended to other loops as soon as practical.

2.8 CARBON METER

An electrochemical carbon meter was ordered from Brookhaven National Laboratory (April 2, 1970).

Installation of the meter is planned for Loop 8. Preliminary design of the installation has been completed and drawings are being made. Instrumentation has been ordered.

2.9 LOOP 8 OXYGEN METER

The daily readings of oxygen meter voltage and cold trap temperature for Run 8-6 correlated fairly well. The oxygen concentration plotted as ppm O (Figure 4), was obtained by converting the cold trap temperature of ppm O by the Eichelberger solubility curve.* It was necessary to assume that the cold trap temperature was the Na_2O saturation temperature. The least squares equation obtained, $\text{EMF} = 1.150 - 0.035 \log C$, compares fairly well with the previous calibration,** $\text{EMF} = 1.179 - 0.051 \log C$, considering that the present correlation covers a very small range of the cold trap temperatures covered when the previous calibration** was made.

The results show a distinct change in calibration between Runs 8-5 and 8-6. Otherwise, the cell has given reasonably stable signals at constant cold trap temperature.

2.10 LOOP 3R - RATIONAL MODEL EXPERIMENT

The purpose of this test is:

1. To simulate velocity profiles close to walls of typical sodium system components, and to study how their shape may influence the corrosion process.
2. To test the performance of an electrochemical oxygen meter with an air reference electrode.
3. To determine how different oxygen impurity levels will influence the leaching out of the different alloy elements on a sodium exposed stainless steel surface.

The following activities were carried out during this period:

1. Assembly of the loop and associated control and instrumentation was completed. Figures 5 and 6 show details of the sodium agitator, drive and sample assembly. Predetermined velocity profiles are produced on the i.d. of samples by varying annular gap between agitator and samples and agitator rotational velocity.
2. Checkout of controls and instruments, preheaters and thermocouples, dc pump and power supply, sodium agitation drive, and ventilation system was completed.

*R. L. Eichelberger, AI-AEC-12685 (1968)

**GEAP-10036, p 19.

Figure 4. Oxygen Meter Calibration Curve

Figure 5. Sample Holder and Components of Rotating Mechanism for Loop 3R

Figure 6. Sodium Agitator Assembly and Sample Holder for Loop 3R

3. Plans were completed for measuring the oxygen removed from fresh stainless steel surfaces during loop startup. A Fixture containing fresh, cleaned surfaces which can be valved into the loop via normal flow through sampler ports was fabricated. Measurements of the oxygen increase will be made by oxygen cells (two), plugging indicator, and chemical sampling.

2.11 OBSERVATIONS CONCERNING EFFECT OF OXYGEN ON MASS TRANSFER

Further analysis of the unusually high corrosion rates reported for Run 6R-3 in PA-15 Quarterly Progress Report No. 12 reveal that the oxygen content would not have to be extremely high to obtain these rates.

It was shown for the 300-series austenitics that the increase in corrosion rate for Run 6R-3 over the standard rates was ~7X, Table 2. If it can be assumed that the standard rates are actually obtained at a range of 2 to 4 ppm oxygen, rather than the < 10 ppm usually reported; a 7X increase in corrosion rate could be effected at a range of 10 to 20 ppm oxygen. Investigating the literature^{1,2} for the effects of oxygen on the corrosion rates of Types 304 and 316 SS at 1300°F reveals the results shown in Table 3.

Table 2

CORROSION RESULTS IN 1300°F SODIUM

Material	6R-3	Standard
316	2.63	0.35
304	3.02	0.55
347	3.04	0.64

Table 3

CORROSION RATES IN 1300°F SODIUM AS A FUNCTION OF OXYGEN

Mils/Year					
Material	25 ppm	6R-3	10-12 ppm	4 (< 5) ppm	Standard
316	4.0 ¹	2.63	1.2 ¹	0.35 ¹	0.35
304	—	3.02	2.2 ²	0.50 ²	0.55

It can be seen that the agreement with the above assumption is reasonable.

2.12 EFFECTS OF HEAT FLUX

The objective is to study the effects of heat transfer on corrosion and mass transfer. Previously, electron bombardment heaters (EB heaters) have operated for times up to 3700 hours³ in the heat flux range of 0.5 to 1.0 X 10⁶ Btu/hr-ft². EB heater E-EB-4-3 has recently completed 4587 hours of exposure. The Type 316 SS anode was exposed for 3264 hours to nonisothermal sodium (system $\Delta T = 440^\circ\text{F}$) flowing at 15 fps in the temperature range of 1122 to 1235°F (ΔT of heat transfer section), at an average heat flux of 0.5 X 10⁶ Btu/hr-ft² (11 kW).

The EB heater anode has been metallurgically evaluated to ascertain the effects of heat transfer on the corrosion characteristics in sodium. The evaluation consisted of optical metallography, surface X-ray fluorescence (XRF), scanning electron microscopy (SEM), and surface roughness (profilometry). To further aid the analysis, a Type 316 SS anode exposed for 1228 hours to nonisothermal sodium (system $\Delta T \approx 525^\circ\text{F}$), flowing at 15 fps with no heat flux at an average temperature of ~1300°F (1319°F inlet to 1280°F outlet) was also evaluated. The two anodes were exposed in the same test facility and the same test location.

2.13 RESULTS OF METALLURGICAL EXAMINATION

The Type 316 SS anodes were seamless and annealed 0.438 in. o.d. X 0.049 in. wall tubing. The tubing was centerless ground to 0.394 in. o.d. X 0.027 in. wall. The chemical composition is shown in Table 4.

Table 4

CHEMICAL COMPOSITION OF TYPE 316 SS ANODE MATERIAL, WT.%

C	Si	Mn	Cr	Ni	Fe	Mo	P	S
0.06	0.58	1.64	17.4	13.4	Bal.	2.32	0.022	0.011

2.13.1 Anode With Heat Flux

Sixteen metallographic and XRF samples were evaluated. The location of the samples is shown in Figure 7. The changes in o.d. surface composition are shown in Table 5. The i.d. of the samples was coated with varying amounts of tungsten, barium, zinc, and copper. The significant changes in metallographic response are shown in Figure 8.

2.13.2 Anode Without Heat Flux

The EB heater experienced an internal short after a brief period of startup. Metallographic evaluation and XRF analysis were performed on four samples from the anode. The changes in o.d. surface composition were minor (2.5 to 3.0% for both chromium and nickel), and consequently no metallographic surface changes were noted.

2.14 DISCUSSION

2.14.1 Compositional/Structural Effects

The o.d. surface compositional and structural changes for the anode with heat flux are correlated with the bulk sodium temperature profile along the anode in Figure 9. The compositional change follows the sodium temperature profile quite well. There is an apparent initial deposition zone (primarily chromium) between anode samples 1 and 5. This was not seen in the analysis of the previous anodes.³ However, the sodium temperature appears to decrease slightly between the inlet to the EB heater test section (1122°F) and the start of the heated zone (1113°F). The slight temperature drop could cause deposition, especially of selective elements, i.e., chromium, if the sodium in the loop were near the saturation level for these elements. After anode sample 5, the surface composition roughly follows the increasing sodium temperature profile until the heated zone is passed. Downstream of the heated zone, the surface composition approaches the base composition. This occurs even though the bulk sodium temperature remains relatively constant. It has previously been stated⁴ that the effect of heat transfer on corrosion rate appears to be adequately described as an increase in rate corresponding to an increase in metal surface temperature, in this case 15 to 20°F, over the bulk sodium temperature. Therefore, a reduction in the metal surface temperature, to approximately that of the bulk sodium, combined with loop position (further downstream) results in the decrease in surface compositional effects downstream of the heated zone.

The correlation of the o.d. surface structural response to the compositional changes in Figure 9 requires further explanation. The formation of a white surface layer, followed by a subsequent change to a grey layer, has been previously seen.³ However, due to the large number of metallographic samples in the present evaluation, it can be seen that the white layer actually disappears prior to the formation of the grey layer. The two distinct layers result from two different phenomena associated with mass transfer processes. The white layer is a result of decarburization. This is evidenced by the gradual removal of the carbides decorating the o.d. surface slip lines, anode samples 2 and 5 in Figure 8 and the relatively minor selective leaching of chromium and nickel in these samples, Figure 9. As the temperature is increased, the amount of carbon removal and selective oxidation (mass transfer) increases. The decarburization eventually results in the complete removal of the carbides from the slip lines, which are no longer visible in anode sample 10, Figure 8. Also, the white layer has disappeared on anode sample 10, probably removed by corrosion as indicated by the roughening or pitting shown on anode sample 6. As the extent of decarburization increases in depth into the material, grain boundary sigma particles form and grow, as shown on anode samples 10 through 16. Up to and including anode sample 10, the selective oxidation of chromium

Figure 7. Sketch Showing the Areas of Metallographic and X-Ray Examination

Figure 8. Structural Variations of Type 316 SS EB Heater Anode
(Photos reduced 60% for Publication.)

Figure 9. Compositional and Structural Effects of Type 316 SS Anode as a Function of Temperature and Loop Position

Table 5
COMPOSITIONAL CHANGE ON THE O.D. OF THE HEATER ANODE

Based on XRF

Specimen	Cr	Ni	Mn	Mo
1	0	+1.80*	0.26	0.03
2	+1.87	+0.84	0.16	+0.07
3	+4.57	1.20	0.06	+0.02
4	+2.04	2.64	0.16	+0.07
5	+0.17	3.24	0.26	+0.12
6	1.70	3.84	0.38	+0.10
7	3.23	4.32	0.54	+0.10
8	4.42	6.84	0.66	+0.02
9	4.42	6.84	0.66	+0.07
10	4.59	7.44	0.72	+0.15
11	5.10	7.80	0.76	+0.17
12	6.12	8.76	0.90	+0.22
13	6.29	9.12	0.98	+0.25
14	6.29	8.40	0.90	+0.25
15	6.80	9.24	0.96	+0.25
16	0	2.64	0.54	0.03

* Positive value represented an increase over the base composition.

and nickel (also manganese) has been insufficient (12.4 Cr-4.6 Ni alloy) to produce a surface phase change from austenite to ferrite. However, starting with anode sample 11 (11.9 Cr-4.2 Ni) and continuing through anode sample 15 (10.2 Cr-2.8 Ni), a ferritic surface phase (grey layer) is formed which grows to a maximum of 0.25 mil. The ferritic phase disappears downstream of the heated zone as the selective oxidation is reduced to almost zero on anode sample 16.

The general microstructure shown in Figure 8 is typical for Type 316 SS in the temperature range of 1100 to 1250°F. At 1100 to 1125°F, the structure is predominantly carbide precipitation in the grain boundaries, anode sample 2. As the temperature is increased, precipitates form intragranularly and sigma particles appear in the grain boundaries. The sigma particles are primarily a result of decarburization, but are also due to the increasing temperature. The structure near the i.d. surface of the anode is generally similar to that along the o.d. surface until anode sample 11, when massive sigma particles become evident, and remain present through anode sample 13. In this case, the large sigma particles are indicative of a significantly higher temperature³ at the i.d. surface of this region (area of highest heat flux). This agrees with the temperature profile shown in Figure 9, where the sodium temperature rises rapidly and reaches a peak in the vicinity of anode sample 11.

The o.d. surface compositional changes, initiation of grain boundary sigma particles, and absence of a ferritic surface phase on the anode without heat flux are indicative of Type 316 SS exposed isothermally to 1300°F sodium flowing at 15 fps for ~1000 hours.⁵

2.14.2 Effects of Heat Transfer on the Corrosion Rate

To determine whether heat flux per se alters the corrosion rate response of Type 316 SS in flowing sodium, the following technique is presently available: Compare the metallurgical characteristics of the present anode (compositional and structural responses) to the established isothermal and heat transfer characteristics in the literature.

Isothermal exposures of Type 316 SS to flowing sodium containing < 10 ppm oxygen^{1,6} have resulted in the various corrosion rates as shown in Table 6. Exposures at 1300°F and 10 fps⁵ show the following structural boundary conditions based on test loop position (downstream) and the corresponding corrosion rates:

Table 6

**ISOTHERMAL CORROSION RATE DATA
FOR TYPE 316 SS IN SODIUM**

Sodium Temperature (F)	Velocity (fps)	Corrosion Rate (mils/yr)*
1202 ¹	27.5	0.62
1300 ⁶	28.0	0.75
1300 ⁶	16.0	0.68
1300 ⁶	6.0	0.32
1300 ⁶	3.0	0.22

* Rates based on exposure times of 3000 hours or longer.

- (1) Upstream position, 0.39 mils/year.
  - (a) 1000 hours, no ferritic surface phase,
  - (b) 1750 hours, ferritic surface phase;
- (2) Downstream position, 0.15 mils/year.
  - (a) 4850 hours, no ferritic surface phase.

Therefore, the structural response is not only dependent on the corrosion rate, which is a function of temperature, velocity, loop position, etc., but is also dependent on the exposure time at a particular rate.

Nonisothermal exposures of Type 316 SS to flowing sodium containing < 10 ppm oxygen^{7,8} have resulted in the mass transfer characteristics as shown in Table 7. The recent data obtained by Atomics International (AI)⁸ have been aided by weighable samples, as indicated by the corrosion rates in Table 7.

Table 7

NONISOTHERMAL DATA FOR TYPE 316 SS IN SODIUM

Exposure Time (hr)	Bulk Sodium Temp. (F)	Metal Surface Temp. (F)	Velocity (fps)	Corrosion Rate (mils/yr)	Ferritic Layer (mils)
351 ⁸	1230	1266	20.5	0.72	0.20
361 ⁸	1220	1255	19.1	0.60	N.A.
589 ⁷	1225	1260	20.0	—	0.40
344 ⁸	1225	1250	10.1	0.58	0.12
3264*	1223	1240	15.0	—	0.25

* Evaluation contained herein.

The effect of velocity on the corrosion rate of Type 316 SS in 1300°F isothermal sodium using the data from Table 6 is shown in Figure 10. There is general agreement that the velocity effect is negligible after 15 fps.^{1,6} The effect of temperature is indicated in the figure by the inclusion of the United Kingdom (UK) data in 1202°F isothermal sodium. For comparison purposes, the AI heat flux data which are within the temperature and velocity ranges of the isothermal data are also included. Within the limits of the data, it can be seen that there is no apparent effect of heat transfer on the corrosion rates. This is further illustrated in Figure 11 where the effect of temperature (over a limited range) on the corrosion rate is shown assuming a minimal effect of velocity in the 20 to 30 fps range.

Figure 10. Corrosion Rate of Type 316 SS as a Function of Velocity in 1300F Isothermal Sodium

Figure 11. Corrosion Rate of Type 316 SS as a Function of Temperature in Sodium Flowing at 20 - 30 fps

The data in Table 7 shows the existence of a ferritic surface phase for the various Al exposure conditions, even though the exposure times are relatively short. It was stated earlier in this report that for corrosion rates lower than those in Table 7, the formation of the ferritic surface phase is time dependent in addition to being corrosion rate dependent. If one assumes, within experimental error, equivalent corrosion rates for the first three data points in Table 7 (temperatures and velocities are similar), then there is an apparent time effect on the growth of the surface phase during the first 1000 hours (0.2 to 0.4 mils, 351 to 589 hours). There is the further effect of corrosion rate (produced by decreased velocity) on the surface phase thickness at equivalent exposure times (0.12 mils at 344 hours and 0.20 mils at 351 hours). The above effects are explained by the observation that steady-state corrosion rates are generally not achieved before 500 to 1000 hours.⁵ Since the initial corrosion rate is higher than the steady-state rate, the higher selective leaching rates of chromium, nickel, and manganese produce a growing ferritic surface layer with time during the initial corrosion rate stage. When the steady-state corrosion rate is achieved, the ferritic surface phase has reached its limiting thickness. This leads to the conclusion that the reported Al corrosion rates are somewhat high because they reflect heavily the initial corrosion rate due to the short exposure times.

It is virtually impossible to assign corrosion rates to the anode under heat flux on the basis of comparisons to known corrosion rate samples because of the differences in temperatures and exposure times. To adequately define the possible effects of heat transfer on the corrosion rate, the following two requirements must be satisfied; (1) weighable heat transfer samples, and (2) comparable isothermal samples exposed at a sodium temperature equal to the maximum metal temperature of the heat transfer samples. This is planned for Run 9-3.

2.14.3 Surface Characteristics

AI has examined the surface characteristics of their heat transfer samples with the use of electron-micrograph stereograms.^{7,8} Profile measurements were made from the stereograms, and it was found that on an as-received 21 rms surface, the heated surfaces were 12 rms, and the unheated surfaces were 25 rms after exposure.⁷ This indicates an increase in roughness of the unheated zone and a decrease in the roughness of the heated zone. The grain boundaries in the heated zone were preferentially attacked, leaving the grains highly polished. Two-stage carbon replicas of the surfaces show differences in surface appearance depending upon location.⁸ Upstream of the heated zone, the surface is uniformly covered with particles, a fraction of a micron in size. No particles were observed in the heated zone where the grain boundaries were delineated. Downstream of the heated zone, the grain boundaries were still clearly visible, but sparsely covered with particles.

Profilometry measurements were made on the heat flux anode and are shown in Table 8. Scanning electron micrographs of representative regions of the anode show the following: (1) upstream of the heated zone, the surface is uniformly covered with particles, ~1 micron in size; (2) In the heated zone, the grain boundaries were in evidence along with a few particles; and (3) downstream of the heated zone, the grain boundaries were still visible, but there was a heavier coating of particles than in the heated zone. The GE and AI results are in basic agreement with the exception of the surface roughness measurements, which show opposing trends.

Table 8

SURFACE ROUGHNESS OF HEATED ANODE

Sampler	RMS
As-Received	12
Upstream of Heated Zone	7
Heated Zone	10
Downstream of Heated Zone	11

The General Electric data show a decrease in roughness in the unheated zone, and a gradual increase in roughness (approaching the as-received) in the heated zone of the anode. However, the AI surface roughness data were achieved by optical measurement which, if used in an area of deposition (unheated zone), will give a fictitiously higher value. The deposits are rather loose (based on appearance and past knowledge) and should not affect a physical measurement of surface roughness, which will then show the actual roughness of the underlying metal substrate. Therefore, what actually appears to

be happening is that, at the lower corrosion rates, the metal surface is being polished, and, at the higher rates, the grain boundaries are being selectively attacked resulting in a rougher surface. This is of importance in heat transfer calculations concerned with fluid flow past a roughened surface.

2.14.4 Conclusions

The mass transfer characteristics of Type 316 SS in 1200 to 1300°F flowing sodium do not appear to be affected by heat transfer. The effect of heat transfer can be described as an increase in corrosion rate corresponding to an increase in metal surface temperature over the bulk sodium temperature. However, weighable heat transfer and isothermally exposed samples, tested at equivalent test conditions (sodium velocities, loop positions, temperatures, and exposure times), are necessary to adequately define the corrosion rates under the influence of heat transfer and the effects, if any, of heat flux on mass transfer.

Type 316 SS experiences a surface polishing from the as-ground condition of 12 rms at low corrosion rate conditions, i.e., isothermal 1125°F sodium. As the corrosion rate increases (1125 to 1235°F sodium with heat transfer), the surface roughens, due to preferential attack at the grain boundaries, and approaches the as-ground condition.

3. TASK II: SODIUM IMPURITIES DETERMINATIONS AND PARTICULATE IMPURITY IDENTIFICATION

3.1 SEPARATION AND IDENTIFICATION OF PARTICULATES

3.1.1 Loop 1 Filters

Examination of two additional filters from Loop 1R have been completed. During Loop Run 1R-2, a filter with a 10 micron porosity was tested in place of the five micron ones used in previous runs. The temperature of collection averaged 513°F. Essentially nothing was collected. This compares to the 3 grams of material that have usually been obtained indicating that most of the loop particulates are below 10 micron in size.

During Loop Run 1R-2A, a 5 micron filter was placed in-stream for 175 hours at an average sodium temperature of 724°F. Again, only a few milligrams of materials were obtained. All previous collections were made at temperatures of about 500°F. The temperature of collection in the present experiment is higher than that at which hydrogen compounds (NaH, NaOH) have been found to precipitate in the loops. It has been assumed that the sodium oxide in the previous samples had been due to both the decomposition of this hydroxide during distillation of the filter and to contamination during sampling. The present results indicate that contamination has been only a minimal contributor and the high quantities of oxide observed previously were due to sodium hydroxide decomposition. The data also tends to show that the oxides of silicon believed to be present are also soluble at the 700°F sampling temperature.

3.2 INTERACTION STUDIES, COMPOUND IDENTIFICATION, ANALYTICAL CHEMISTRY

3.2.1 Interaction Studies

The first set of capsule experiments designed to study sodium-oxygen-transition metal interaction have been completed. Experimental details were provided in the previous quarterly.

Wires of iron, nickel, chromium and molybdenum were exposed to sodium containing 100 ppm oxygen for 300 hours at 600°C. At the completion of the test, α Fe₂O₃ was found on the iron wire while traces of Cr₇C₃ were found on the chromium wire and Mo₂C was deposited on the molybdenum wires. The source of carbon is the impurity normally found in sodium.

Analysis of the sodium from several of the capsules showed a depletion in oxygen to 20 ppm from the original 100 ppm, while the X-ray data gave no indication of oxide on the Cr and Mo wires. This fact, coupled with the observation that the alumina capsules in which the reaction took place, appeared discolored leads to the tentative conclusion that the oxygen activity was reduced because of reaction of the sodium oxide with the alumina capsule or impurities therein. Further tests, in different capsules, are underway.

3.2.2 Accumulation of X-Ray Diffraction Data

β -sodium ferrite, NaFeO_2 , has been prepared by the reaction of sodium carbonate with iron oxide (Fe_2O_3). An attempt to convert this to the α -form is in progress. The latter compound, $\alpha\text{-NaFeO}_2$, should resemble NaCrO_2 structurally and it is thus, important to determine whether the two species can be distinguished by X-ray analysis. (The chromite has been detected in loop particulates).

The X-ray pattern for the β -form is in good agreement with that of Thery.⁹

3.3 SAMPLING OF LOOPS AND DETERMINATION OF IMPURITY LEVELS

3.3.1 Total Alkalinity Data

A summary of total alkalinity data obtained over the past year has been prepared and is presented in Table 9.

This determination reflects the alkaline impurities in the loop, i.e., Na_2O , Na_2CO_3 , NaH . At least in a qualitative sense, the data obtained mirrors the oxygen activity and corrosion potential in the loops. The data in Table 9 clearly shows that the sodium in Loop 1R during Run 1 was considerably less pure than is normal for the mass transfer systems. Based on the presence of high first break temperatures in the plugging indicator runs, the impurities causing the high values are probably hydrogen compounds.

The precision of the alkalinity determination (analysis and sampling error) is on the order of ± 2 ppm. (one standard deviation) The data indicates that loop control is only slightly less precise, ± 3 to 4 ppm. Thus, any efforts to improve purification procedures will also require improvements in the analytical technique if the changes are to be measured. Current effort in this region includes revision of the procedure to make correction for hydride impurity and attempts to lower the blank by equilibration of the amalgamation vessel in the glove box.

The most recent data on alkaline impurities (amalgamation determination performed on flow through samples) is shown in Table 10.

3.3.2 Determination of Carbon

Carbon analyses were performed on a number of samples of Loop 8 sodium in support of the investigation of high carburization rates during Run 5. The data obtained is listed in Table 11.

The data for total carbon from the flow through samples taken during the run show no significant increases over those values experienced in the loops in which extensive carburization is not taking place. The Loop 8 data gives an average carbon content of 37 ppm while data compiled for several runs from the other loops gives a mean value of 38 ppm. There is some indication that readily hydrolyzable carbon forms are somewhat higher in the Loop 8 run but the error in this portion of the analysis is too high to make a conclusive comment.

The samples from the dump tanks give no clarification of the problem, but the data does show that some settling of carbon occurs in these more stagnant areas, i.e., higher results are observed near the bottom of the tank.

Analysis of the contents of the cold trap from the system are in progress. Preliminary data show significant correlation with the carburization problem.

Two areas near the bottom of the trap are under study. The upper one is fairly soft. Hydrolysis of this sodium liberates black particles which are about 50% carbon. Analysis of the samples by the usual carbon determination indicates that the total carbon content of the sodium is about 500 ppm with 200 ppm being present in the water acid form.

Below this section is a very hard plug, i.e., material with a low metallic sodium content. Sodium was removed by distillation and the residue analyzed for carbon species. The data is shown in Table 12.

The most striking observation is the high quantity of carbonate in this system.

In previous cold trap examinations,¹⁰ the major carbon species in an austenitic system cold trap was elemental carbon present at a ratio of about 4 to 1 to the carbonate carbon. In the present examination, the ratio is reversed—the carbonate carbon being about six times higher than the elemental carbon. The present data can also be compared to the previous results in a ferritic system wherein cold trap deposits contained carbonate as a major species but at only a 1.2 to 1 ratio to the elemental carbon.

Table 9

**SUMMARY OF TOTAL ALKALINITY
DETERMINATIONS (AMALGAMATION)**

Sample Source	Number Of Samples	Total Alkalinity (ppm)
Loop 1R Run 1	6	19.3 ± 3.7
Loop 1R Runs 2, 2R	8	12.9 ± 1.6
Loop 4 Run 2	33	14.1 ± 4.2
Loop 5 Runs 4, 5		
Loop 6 Runs 3, 4		
Loop 8 Runs 5, 6	17	12.4 ± 3.3
Loop 9 Runs 2, 2A		

Table 10

**TOTAL ALKALINE IMPURITIES IN LOOP
SODIUM (AMALGAMATION DETERMINATION)**

Sample Number	Date Taken	Total Alkalinity
1-2A-2	1-26-70	10, 15*
4R-2-12A	11-19-69	8
4R-2-14A	1-19-70	14
4R-2-15A	2-11-70	16
4R-2-16A	3-3-70	8
4R-2-17A	3-24-70	8
6R-4-6A	12-13-69	16
6R-4-7A	1-13-70	10
6R-4-8A	1-29-70	11, 15
6R-4-10A	3-17-70	12
6R-4-12A	5-17-70	14
8-6-1A	12-23-69	11
8-6-2A	2-3-70	14
8-6-3A	2-20-70	24
8-6-4A	2-26-70	13
8-6-11A	4-28-70	11, 7
9-2A-4A	11-6-69	10
9-2A-8A	3-18-70	15

* Reported previously.

Table 11

DETERMINATION OF CARBON IN LOOP 8 SODIUM

Sample (Flow through samples)	Water Acid Carbon* ppm	Total Carbon ppm
8R-5-1A	7	42
8R-5-2A	10	31
8R-5-4A	7	29
8R-5-5A	11	28
8R-5-6A	32	57
8R-5-7A	16	39
8R-5-8A	20	38
8R-5-9A	5	44
8-6-7A	7	27
8-6-8A	6	32
8-6-9A	3	40
8-6-10A	4	40
(Tank Samples - glass thief tube)		
8-6-5B (FWD Tank)	2	13
8-6-5A (Aft Tank)	6	24
Holding Tank	5	22
(Tank Samples - metal thief tube)		
Sodium heel - Rear Original		
Dump Tank	11	83
Sodium heel - Forward		
Dump Tank	14	71

* Carbon liberated by treatment with water and acid, e.g., of carbides, carbonate or possible attack on elemental carbon.

Table 12

CARBON CONTENT IN HARD DEPOSITS
IN LOOP 8 COLD TRAP

Species Evolved in Dissolution	Carbon (ppm)
Ethane	4
Acetylene	30
Carbon Dioxide	2000
"Elemental" Carbon	300

It is uncertain whether sodium carbonate is a cause of carburization or a by-product. That is, carbonate entering the system presumably from the atmosphere should be reduced to elemental carbon via the reaction:

and the carbon so formed could be carburizing. On the other hand, if some other source of carbon, e.g., pump oil caused the carburization, metallic carbides so formed may react with Na_2O in the system to form Na_2CO_3 :

Alternatively, the carbonate may have no part in the phenomenon at all. Further study of the effect of carbonate seems warranted.

4. TASK IV: CORROSION AND MASS TRANSPORT EFFECTS ON COMPOSITION, STRUCTURE, AND STRUCTURAL INTEGRITY OF CANDIDATE ALLOYS FOR LMFBR'S AND POSSIBLE LOCALIZED ATTACK

4.1 COMPOSITION AND STRUCTURAL EFFECTS ON MECHANICAL PROPERTIES

4.1.1 Loop 4R – Corrosion Effect on Stress Rupture, Run 4R-2, 1300°F

The loop operated at test conditions during the report period. The run reached 10,569 hours at 1300°F with the pressure applied, as of May 22. There were no failures during the report period. Twelve of the original sixteen samples have failed. The results to date are shown in Table 13.

4.1.2 Loop 5R – Corrosion Effect on Stress Rupture, Run 5R-5, 1300°F

The loop was restarted during the report period after replacement of the main loop heater. The run reached 2850 hours at 1300°F with the pressure applied as of May 22. There have been no failures of the original sixteen samples.

4.1.3 Loop 6R – Corrosion Effect on Stress Rupture, Run 6R-4, 1150°F

The loop operated at test conditions during the report period. The run reached 6620 hours at 1150°F with the pressure applied, as of May 22.

There were two failures during the report period. Nine of the original sixteen samples have failed. The results to date are shown in Table 14.

The long-term stress-rupture tests of Type 304 SS in 1150°F sodium have been concluded with the two recent failures. The results are illustrated in Figure 12, accompanied by the previously obtained 1300°F sodium results. Two observations can be made: (1) the drastic differences in relative slope of the stress-rupture curves at the two temperatures; and (2) the significant increase in rupture strength associated with the carbide agglomerated treatment at 1150°F; whereas, no difference was noted at 1300°F.

4.1.4 Stress Rupture Control Tests

Stress-rupture tests are being run at 1300°F in argon on mill-annealed and carbide-agglomerated Types 304 and 316 SS. The tests are being run for the purpose of supplying comparison (control) data for the tests in flowing sodium. The same heats of materials are being tested in both environments; whereby, the environmental effect(s) can be delineated. The results to date are shown in Table 15.

The results, plus those for Run 4R-2 and the previously reported data from Loop 5R¹¹ are plotted in Figures 13 and 14. On the basis of comparison with the control tests, there is a definite decrease in long-term stress-rupture properties due to exposure in nonisothermal flowing sodium. The loss in rupture strength is increasing with exposure time and is quite dramatic for Type 304 SS. There was no consistent difference in the pattern of results between the two structures as was noted previously for Type 304 SS at 1300°F, Figure 12.

Bulk carbon determinations show that Types 304 and 316 SS lose 73 and 48%, respectively, after 4800 hours exposure to 1300°F sodium.¹² The loss of rupture strength shown in Figures 13 and 14 can be tied directly to the substantial losses of carbon.

Figure 12. Biaxial Stress-Rupture Properties of Type 304 SS in Sodium

Figure 13. 1300F Biaxial Stress-Rupture Properties of Type 316 SS in Sodium and Argon

Figure 14. 1300F Biaxial Stress-Rupture Properties of Type 304 SS in Sodium and Argon

Table 13

1300°F BIAXIAL STRESS—RUPTURE DATA FROM RUN 4R-2

Material	Initial Hoop Stress, psi	Time-To-Rupture, hr
304CA	7000	2188
304MA	7000	2198
304MA	7000	2237
304CA	7000	2263
316MA	8000	3822
316MA	8000	4415
316CA	8000	5162
316CA	8000	5175
304CA	5000	4500
304MA	5000	5187
304CA	5000	6228
304MA	5000	6546

MA — mill annealed

CA — carbide agglomerated, 1650°F for 24 hours.

Table 14

1150°F BIAXIAL STRESS—RUPTURE DATA FROM RUN 6R-4

Material	Initial Hoop Stress, psi	Time-To-Rupture, hr
304MA	20,000	514
304MA	20,000	559
304CA	20,000	1662
304CA	20,000	4006
316MA	20,000	639
304MA	18,000	1710
304MA	18,000	1947
304CA	18,000	5391
304CA	18,000	6235

Table 15
1300°F BIAXIAL STRESS—RUPTURE DATA IN ARGON

Material	Initial Hoop Stress, psi	Time-To-Rupture, hr
316CA	15,000	925
316MA	15,000	1375
316CA	13,000	1584
316MA	13,000	2415
316MA	11,000	3183
316MA	11,000	4551
316CA	11,000	4874
304CA	11,000	718
304MA	11,000	829
304MA	11,000	901
304MA	9,000	2511
304CA	9,000	2727
304CA	9,000	2895
304MA	9,000	3373
304CA	9,000	3389
304MA	9,000	4374
304MA	8,000	3380
304CA	8,000	4165

Consistent with the loss of carbon and rupture strength would be an expected increase in rupture ductility. However, the rupture ductilities for Types 304 and 316 SS in 1300°F sodium are ~50% lower than in 1300°F argon, Table 16. The loss of carbon has promoted the formation of massive sigma particles in the grain boundaries, Figures 15 and 16. Assuming the prevailing mechanism for creep at 1300°F to be grain boundary sliding, the loss in rupture ductility can be explained on the basis of the grain boundary sigma particles impeding the sliding motion. This results in premature cracking in the grain boundaries at the sigma particles, and a reduction in load (stress) transmittance to the grains. Therefore, the grains do not deform as much and the resultant rupture ductilities are reduced.

Samples of Types 304 and 316 SS, 0.250 in. o.d. X 0.015 in. wall seamless tubing, have been exposed to 1300°F sodium for various lengths of time in the unstressed condition. The biaxial stress-rupture testing of these samples are shown in Figures 17 and 18 compared to the argon results for previously unexposed material. The rupture strengths of the sodium exposed samples have been reduced, and the reduction is not time dependent. The longer the sodium exposure, the greater the strength reduction, although it can be seen that the shorter exposure times become difficult to distinguish between, Figure 17. The results are predictable on the basis of increased bulk decarburization with increasing sodium exposure. The results point up the fallacy of attempting to predict the mechanical properties of Types 304 and 316 SS after sodium exposure on the basis of testing decarburized material. The material is constantly changing due to the sodium exposure and hence, the properties are constantly changing. Therefore, it is not possible to select a reasonable starting condition to predict the properties at a future point in time of 1 to 3 years.

SODIUM

ARGON

500X

GLYCEREGIA

Figure 15. Type 316 SS Exposed at 1300F

SODIUM

ARGON

500X

GLYCEREGIA

Figure 16. Type 304 SS Exposed at 1300F

Figure 17. 1300F Biaxial Stress-Rupture Properties of Type 316 SS in 1300F Argon

Table 16

EFFECT OF ENVIRONMENT ON THE
RUPTURE DUCTILITY AT 1300°F

		Hoop Stress, psi	$\Delta D/D$ %
316 SS	Sodium	12,000	5.6
		9,000	4.4
	Argon	15,000	13.6
		13,000	14.8
		11,000	12.3
304 SS	Sodium	9,000	9.2
		7,000	6.8
	Argon	11,000	15.6
		9,000	15.6
		8,000	16.8

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PA-15 PROGRAM CONTRIBUTORS

TASK I – CORROSION/DEPOSITION STUDIES

P. Roy – Task Leader

J. T. Cochran	L. E. Pohl
G. D. Collins	M. K. Schad
M. F. Gebhardt	G. P. Wozadlo

TASK II – SODIUM IMPURITIES AND PARTICULATES

D. Dutina – Task Leader

E. A. Aitken	W. D. Reynolds
G. D. Collins	H. S. Rosenbaum
J. E. Lewis	P. Roy
L. E. Pohl	J. L. Simpson

TASK IV – CORROSION AND MASS TRANSPORT EFFECTS

G. P. Wozadlo – Task Leader

G. D. Collins	L. E. Pohl

GENERAL PROGRAM AND CONSULTING

C. N. Spalaris
R. S. Young
E. L. Zebroski

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Asst. Director for Project Management Division of Reactor Development & Technology United States Atomic Energy Commission Washington, D. C. 20545	1	Chief, Liquid Metal Projects Branch Division of Reactor Development & Technology United States Atomic Energy Commission Washington, D. C. 20545	1
Asst. Director for Plant Engineering Division of Reactor Development & Technology United States Atomic Energy Commission Washington, D. C. 20545	1	Chief, Fuel Engineering Branch Division of Reactor Development & Technology United States Atomic Energy Commission Washington, D. C. 20545	1
Asst. Director for Reactor Engineering Division of Reactor Development & Technology United States Atomic Energy Commission Washington, D. C. 20545	1	LMFBR Program Manager Division of Reactor Development & Technology United States Atomic Energy Commission Washington, D. C. 20545	1
Asst. Director for Reactor Technology Division of Reactor Development & Technology United States Atomic Energy Commission Washington, D. C. 20545	1	Director, LMFBR Program Office Argonne National Laboratory 9700 South Cass Avenue Argonne, Illinois 60439	1
Asst. Director for Program Analysis Division of Reactor Development & Technology United States Atomic Energy Commission Washington, D. C. 20545	1	Director, Contracts Division United States Atomic Energy Commission San Francisco Operations Office 2111 Bancroft Way Berkeley, California 94704	1
Chief, Coolant Chemistry Branch Division of Reactor Development & Technology United States Atomic Energy Commission Washington, D. C. 20545	3	Chief, California Patent Group United States Atomic Energy Commission San Francisco Operations Office P. O. Box 808 Livermore, California 94550	1
Chief, Fuels & Materials Branch Division of Reactor Development & Technology United States Atomic Energy Commission Washington, D. C. 20545	1	United States Atomic Energy Commission Division of Technical Information Extension P. O. Box 62 Oak Ridge, Tennessee 37380 Attention: T. W. Laughlin	90