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DEVELOPMENT REPORT
OCTOBER 1968

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**SODIUM COMPONENTS DEVELOPMENT PROGRAM
MASS TRANSFER INVESTIGATIONS
IN LIQUID METAL SYSTEMS
QUARTERLY PROGRESS REPORT
NO. 6
JUNE-AUGUST 1968**

U.S. ATOMIC ENERGY COMMISSION
CONTRACT AT(04-3)-189
PROJECT AGREEMENT 15

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SODIUM COMPONENTS DEVELOPMENT PROGRAM

MASS TRANSFER INVESTIGATIONS IN LIQUID METAL SYSTEMS

Quarterly Progress Report No. 6

June - August 1968

R. S. Young

E. L. Zebroski

Prepared for the
U. S. Atomic Energy Commission
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SECTION I

INTRODUCTION AND PROGRAM SUMMARY

1.1 GENERAL

Corrosion of candidate structural material, mechanical properties effects, and sodium impurities 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 permit optimum material selections (when used in conjunction with irradiation data) and the establishment of corrosion and sodium purity specifications for large plants. The work is in support of the LMFBR and FFTF development programs.

In an earlier 6-year phase of the program ending in 1965, corrosion and mass transfer of Type 316 stainless, 2-1/4 Cr-1 Mo, and 5 Cr-1/2 Mo-1/2 Ti ferritic steels were studied in depth at sodium temperatures to 1200°F. More than 2500 samples of these materials were exposed to sodium during 140,000 hours of loop operations. Average corrosion rates were correlated by statistical analysis from

which a quantitative relation was 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 materials have been extended to include cladding candidates for fast oxide fuel [Types 304, 316, and 347 stainless steels (SS), and Incoloy 800]. The temperature range of testing has been increased to 1300°F and temperature profiles, loop geometry, and sodium purity controls established to represent environments of large fast reactors in an improved way. Further tasks in sodium reactor technology were added this year. These included test loop simulation and sodium vapor control study in support of the FFTF program.

1.2 SUMMARY

The Sodium Mass Transfer Program was organized into six Tasks in July to conform to requirements of program planning for Fiscal Year

1969. These Tasks and their relationship to previous work are as follows:

Task 1 LMFBR/FFTF Corrosion Parameter Studies

This Task is a continuation of Task 1 of the FY-1968 program. Objectives are determination of empirical and rational models for accurate prediction of corrosion and mass transport in primary and secondary systems of large plant designs.

Task 2 Sodium - Impurities Interactions and Identification of Particulate Matter

This Task continues the effort carried out in Task 3, Sodium Impurities, during FY-1968. The scope has been modified to include the three development efforts: sodium system sampling, analytical chemistry, and collection and identification of particulate material. Objectives are to insure well controlled operation of loops

and to support development of rational models.

Task 3 Mechanical Properties of Ferritic Materials in Bimetallic Systems

The objective of this Task is to determine effects on design properties of 2-1/4 Cr-1 Mo as a result of carbon transport in a bimetallic system. The initial phase is limited to review of available data and determination of test objectives and experimental requirements. Samples to measure property changes are available from loop components from phase 1 of the program.

Task 4 Mechanical Properties Limits for Cladding Materials and Localized Attack

This Task is a continuation of work carried out under Task 2 of the FY-1968 Program. The objective is to obtain data, long term stress rupture to 10,000 hours, and stress fatigue data to 6,000 hours for LMFBR cladding candidates.

Task 5 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 1. Sample holder assemblies were installed in SCTI under Task 4 of the FY-1967 and 1968 program. Selected samples will be removed for examination during fiscal year 1969-1970.

Task 6 LMFBR Fuel Examination Facility and Fuel Handling and Radioactive Maintenance System Development

The objectives of this Task are to evaluate sodium cleaning methods for fuel, methods of measurements, and control of sodium vapor and to develop and evaluate

refueling cell techniques and equipment application to FFTF reference and backup designs.

1.3 REPORTS

Monthly report letters are prepared reporting progress in all tasks. Progress letters No. 1 through 104 covering work from July 1, 1959 through July 31, 1968 have been issued.

In addition to the progress report letters, 28 topical reports and 5 Quarterly Progress Reports covering test results have been issued (see Section V).

This is the sixth quarterly progress report issued and covers the main results obtained in each task. The objective is to report the results of data analysis and examinations in those tasks that have reached a meaningful stage of development.

SECTION II

TASK I - LMFBR/FFTF

CORROSION PARAMETER STUDIES

The objective of this work is to determine corrosion and mass transfer rates and correlations with sufficient accuracy for design purposes of large scale systems. Conditions covered are primarily those of system design interest to large LMFBR's and FFTF.

2.1. SUB-TASK 1.1 - EMPIRICAL CORROSION MODELS

Mockup studies related to the PA-15 mass transfer program involve: (a) experimentally determining mass transfer, corrosion and exposure effects in steel exposed to flowing sodium at bulk temperatures up to 1300°F, and (b) re-evaluating the mass transfer/corrosion model on the basis of scaleup or geometric parameters to permit prediction of mass transfer corrosion in large scale systems, (c) monitoring for localized attack, and (d) generating information which will permit establishing relationships between sodium impurities, loop operations, and metallurgical effects.

Loops 8 and 9 which were constructed to simulate corrosion conditions of a 1000 MWe coolant system, provide the major sources of data for the mockup studies. Loop 8 is scheduled to complete a series of four parametric runs to study the effects on mass transfer rates of sodium velocity, mass flow rate, and downstream. The fourth run of the series is currently underway. Loop 9 has completed one run to study the range of velocity effect on mass transfer rates. A second run using operating conditions estimated for FFTF closed loop design is being prepared.

2.1.1 Empirical Equations for Hot Leg Corrosion Rates

Corrosion results (weight change for the duration of the particular run) for the isothermal hot-legs for runs 8-1, 8-2, 8-3, and 9-1, along with the down-stream parameters, velocity, etc., were fit to the following model:

$$\ln CR + b_0 + b_1 \ln V + b_2 (L/D)_1 + b_3 \ln (L/D)_2 + \frac{b_4}{t+1}$$

where,

t = Exposure time, hours

CR = Average corrosion rate,
mg/dm² - month.

b_0, b_1 , etc. = Constants.

V = Sodium velocity, fps.

$(L/D)_1$ = Number of hydraulic
diameters downstream in an
isothermal region.

$(L/D)_2$ = Number of hydraulic
diameters downstream in
an isovelocity region.

Evaluation of the equation format
and an analysis of the coefficients
between the four runs have been
partially completed. The following
equations have been obtained for
each run at 1300°F and oxygen <10 ppm:

Run 8-1

$$(a) \ln CR = 3.25 - 0.0008 (L/D)_1 - 0.086 \ln (L/D)_2 + 0.34 \ln V + 1.035/(t + 1)$$

$$(b) \ln CR = 3.54 - 0.0007 (L/D)_1 - 0.087 \ln (L/D)_2 + 0.35 \ln V$$

Run 8-2

$$\ln CR = 3.72 - 0.0009 (L/D)_1 - 0.083 \ln (L/D)_2 + 0.329 \ln V$$

Run 8-3

$$(a) \ln CR = 4.81 - 0.0020 (L/D)_1 - 0.074 \ln (L/D)_2 + 0.158 \ln V + 0.6000/(t + 1)$$

$$(b) \ln CR = 5.14 - 0.0030 (L/D)_1 - 0.095 \ln (L/D)_2 + 0.169 \ln V$$

Run 9-1

$$\ln CR = 3.76 - 0.0017 (L/D)_1 - 0.084 \ln (L/D)_2 + 0.480 \ln V$$

Evaluation of the coefficients obtained indicates that the present format of the corrosion equation is too simple to describe in detail all of the possible corrosion patterns in a complex loop. At least part of the variation in the coefficients, however, is due to the difficulty of maintaining constant and comparable oxygen levels to the same level of accuracy which is now available for the corrosion rate measurements.

The $\frac{b_4}{t+1}$ term in the equation was originally introduced to account for an initial high corrosion rate in the phase 1 results where data were derived from three different temperature zones at 1000, 1100 and 1200°F. The present corrosion data from loops 8 and 9 along with the data from loops 5 and 6 are all obtained in an isothermal zone at 1300°F. Since nearly all of the weight change data extrapolates fairly closely to zero at zero time, steady state corrosion rate is attained rapidly (<100 hr) at 1300°F. The time dependent term, accordingly can be eliminated at 1300°F within the accuracy of the data as shown by the

equations (a) and (b) for runs 8-1 and 8-3.

The effect of sodium velocity on the corrosion rate for specimens in the same downstream positions is shown in Figure 1. It suggests that corrosion rate becomes nearly independent of the velocity at high velocities (>15 fps). This requires modification of the equations developed for phase 1, in which only two levels of velocity that bracketed the lower, nearly linear range, of velocities were covered. The trend of the effect of velocity on the corrosion rate is in qualitative agreement with published UK⁽¹⁾ results to date, but the absolute magnitude of the rates continues to be somewhat lower for this work than that reported in the UK.

2.1.2 Empirical Equation for Cold Leg Deposition Rates

The data, including weight change, velocity, and downstream, from the isothermal cold-leg sections were fit to the model and the following equations obtained:

FIGURE 1. EFFECT OF VELOCITY ON CORROSION RATE AT 1300° F

Run 8-1

$$\ln DR = 1.99 - 0.0013 (L/D)_1 + 0.004 \ln (L/D)_2 + 0.106 \ln V$$

Run 8-2

$$\ln DR = 2.48 - 0.0030 (L/D)_1 - 0.137 \ln (L/D)_2 + 0.822 \ln V$$

Run 8-3

$$\ln DR = 1.34 - 0.0005 (L/D)_1 - 0.165 \ln (L/D)_2 + 1.074 \ln V$$

Run 9-1

$$\ln DR = 2.50 - 0.0025 (L/D)_1 - 0.0093 \ln (L/D)_2 + 0.806 \ln V$$

The velocity dependence of the deposition rate for specimens in similar downstream positions is shown in Figure 2. From the present results, the deposition rate increases linearly with velocity up to 15 fps.

2.1.3 Analysis of Samples from Run 8-1

Samples were selected from the isothermal hot-leg (316 SS, cartridge FB and 304 SS, cartridge GB), cooler (316 SS, cartridge JC), and isothermal

cold-leg (304 SS, cartridge NA). The samples were all tubes, which were exposed to flowing sodium on i.d. surfaces. The elemental composition and distribution was determined using the electron microprobe and x-ray fluorescence (XRF), and the precipitate phases were determined by x-ray diffraction (XRD). The microprobe analyses were performed on cross-sections of the tube walls and the x-ray analyses were performed on tube wall surfaces and electrolytic extractions (selective dissolution) of tube specimens.

FIGURE 2. EFFECT OF VELOCITY ON DEPOSITION RATE AT 1300° F

The x-ray data from these precipitates were obtained employing 114.6 mm Debye-Sherrer powder cameras and XRD-5 General Electric x-ray diffractometer and spectrometer. The fluorescence analyses were not quantitative, because in many of the precipitate extractions, an insufficient amount of material was obtained to give reliable and accurate measurements. Chromium, $\text{K}\alpha_1$, $\text{K}\alpha_2$, and $\text{K}\alpha$ radiation was used for all of the XRD analyses while platinum radiation using a LiF analyzing crystal was used for XRF analyses. No XRF analyses were performed to detect elements less than atomic number 20.

2.1.3.1 Isothermal Hot Leg, Type 316 Stainless Steel

Samples were exposed 3000 hours in sodium at 1305°F, 3 fps. An example of a specimen current image of one of the samples (81FB52) is shown in Figure 3, where the section includes the i.d. surface and some precipitates. The light to dark contrast is caused by variations in average atomic number of constituents. The lighter the area, the higher the average atomic number. To study the distribution of alloying elements and precipitates, the sample was

examined by the x-ray image technique. Figure 4 through 8 show the relative distribution of Cr, Fe, Ni, Mo and Mn by this technique.

A number of grain boundaries to within about 5 microns of the i.d. surface were analyzed for sodium. No evidence of sodium was found at depths greater than 5 microns within the limits of sensitivity of the microprobe (~2-4%).

The results of the electron microprobe point counting, for the determination of compositional gradients with distance from the surface, are shown in Figures 9 and 10.

The x-ray results of the selective dissolution of the precipitates from two samples (81FB51 and 81FB53) are shown in Tables 1 and 2. The NaCl compound is believed to be a result of reaction with residual sodium on the sample and the acid solution. The difference in relative amounts of phases detected by the two extraction solutions is because the phosphoric acid solution dissolves more of the matrix than the hydrochloric acid solution.

A section of sample 81FB52 was examined on the inside diameter (i.d.)

TABLE 1

X-RAY DIFFRACTION IDENTIFICATION OF PRECIPITATES EXTRACTED FROM
SAMPLES EXPOSED TO SODIUM FOR 3000 HOURS IN RUN 8-1

** Identifi- cation	10 Percent HCL-Methanol Extraction				10 Percent $H_3PO_4-H_2O$ Extraction			
	Major	Minor	Trace	Weak Trace	Major	Minor	Trace	Weak Trace
81FB51 316 SS	σ -phase	$M_{23}C_6$	NaCl	Matrix Unidentified	Matrix	----	σ -phase	$M_{23}C_6$ Unidentified
81FB53 316 SS	σ -phase	A_2B Laves phase $M_{23}C_6$	Matrix NaCl	Unidentified	Matrix	-----	σ -phase $M_{23}C_6$	Unidentified
81GB55 304 SS	$M_{23}C_6$	----	Unidentified similar to Cr_7C_3 , Cr_3C_2 and Fe_3C .	M_3O_4 spinel	Matrix	M_3O_4 , close to composition of Fe_3O_4 .	$M_{23}C_6$ and unidentified similar to Cr_7C_3 , Cr_3C_2 , and Fe_3C .	----
81JC51 316 SS	$M_{23}C_6$	A_2B Laves phase	σ -phase	Unidentified	Matrix	----	----	$M_{23}C_6$ σ -phase
81JC53 316 SS	$M_{23}C_6$	A_2B Laves phase	----	Unidentified	Matrix	-----	M_3O_4 spinel σ -phase	$M_{23}C_6$
81NA55 304 SS	$M_{23}C_6$	----	$M_3C_2?$	Unidentified	Matrix	$M_{23}C_6$	Unidentified	----
81NA57 304SS	$M_{23}C_6$	M_3O_4	$M_7C_3?$	Unidentified	$M_{23}C_6$	Unidentified*	$Fe_3C ?$	----

*Consists of more than four weak lines.

**81FB51) 1305F 3 FPS
 81FB53)
 81GB55 1300F 1 FPS
 81JC51) 1200F 2 FPS
 81JC53)
 81NA55) 800F 2 FPS
 81NA57)

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TABLE 2

SEMIQUANTITATIVE X-RAY FLUORESCENCE DATA FOR PRECIPITATES EXTRACTED
FROM SAMPLES EXPOSED TO SODIUM FOR 3000 HOURS IN RUN 8-1

			10 Percent HCL-Methanol Extraction							10 Percent H ₃ PO ₄ -H ₂ O Extraction								
Identification			MAJOR	STRONG MINOR	MINOR	WEAK MINOR	STRONG TRACE	TRACE	WEAK TRACE	POSSIBLE TRACE	MAJOR	STRONG MINOR	MINOR	WEAK MINOR	STRONG TRACE	TRACE	WEAK TRACE	POSSIBLE TRACE
Temperature	Na	Velocity																
81FB51	316	SS	Fe	Cr	Ni	Mo	--	--	Ti	--	Fe	--	Ni	--	--	Mo	--	--
1305°F	3 FPS		--	--	--	--	--	--	--	--	--	--	Cr	--	--	--	--	--
81FB53	316	SS	Fe	--	Cr	Mo	--	--	Ti	--	Fe	Cr	Ni	--	--	Mo	--	--
1305°F	3 FPS		--	--	Ni	--	--	--	--	--	--	--	--	--	--	--	--	--
81GB55	304	SS	Cr	Fe	Ni	--	Mn	--	--	Mo	Fe	Cr	Ni	--	Mn	--	--	Mo
1300°F	1 FPS		--	--	--	--	--	--	--	Nb	--	--	--	--	--	--	--	Cu
81JC51	316	SS	Fe	Cr	--	Ni	--	--	--	--	Fe	--	Cr	--	--	Mo	--	--
1200°F	2 FPS		--	--	--	Mo	--	--	Ti	--	--	--	Ni	--	--	--	--	--
81JC53	316	SS	Fe	--	Ni	Mo	--	--	Ti	--	Fe	Cr	Ni	--	Mo	--	--	Cu
1200°F	2 FPS		Cr	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--
81NA55*	304	SS	Cr	--	Fe	Ni	--	--	--	Mo	Fe	--	Cr	Mn	--	--	Mo	--
800°F	2 FPS		--	--	--	Mn	--	--	--	--	--	--	Ni	--	--	--	Cu	--
81NA57*	304	SS	Cr	--	Mn	Fe	--	--	--	Mo	Not enough material for analysis							
800°F	2 FPS		--	--	--	Ni	--	--	--	--								

*Very small amounts of material used for analysis.

NOTE: Elements with atomic numbers less than 20 could not be detected with this x-ray fluorescence technique.

FIGURE 3. TYPE 316 STAINLESS STEEL SPECIMEN CURRENT IMAGE SHOWING SECOND PHASES. SAMPLE 81FB52 INSIDE DIAMETER SURFACE ~ 1700X.

FIGURE 4. X-RAY IMAGE SHOWING DISTRIBUTION OF Cr IN SAME AREA AS FIGURE 3.

FIGURE 5. X-RAY IMAGE SHOWING DISTRIBUTION OF Fe IN SAME AREA AS FIGURE 3

FIGURE 6. X-RAY IMAGE SHOWING DISTRIBUTION OF Ni IN SAME AREA AS FIGURE 3

FIGURE 7. X-RAY IMAGE SHOWING DISTRIBUTION OF Mo IN SAME AREA AS FIGURE 3

FIGURE 8. X-RAY IMAGE SHOWING DISTRIBUTION OF Mn IN SAME AREA AS IN FIGURE 3

FIGURE 9. PERCENT COMPOSITION VERSUS DISTANCE FROM THE INSIDE DIAMETER SURFACE FOR SAMPLE 81FB52 (316 SS)

FIGURE 10. PERCENT COMPOSITION VERSUS DISTANCE FROM THE OUTSIDE DIAMETER SURFACE FOR SAMPLE 81FB52 (316 SS)

and outside diameter (o.d.) surfaces by XRD. The results are shown in Table 3.

The microprobe data show that the precipitates are composed primarily of Cr and Mo in varying compositions with some Fe. Those precipitates closest to the surface are Mo rich, but as the distance from the surface increases, the Cr content increases. Other observations are that Fe and Ni are depleted in the Mo-Cr rich areas and that the concentration of Mn decreases as the surface is approached.

The XRD analyses reveal the presence of a sigma phase and a trace of a body-centered-cubic structure. Cr and Mo form a continuous b.c.c. solid solution; however, the measured lattice parameter is too low except for a very low percent of Mo and almost pure Cr. It must therefore be concluded that the Cr-Mo particles are actually sigma Cr-Fe and sigma Mo-Fe. These sigma phases are reported to form a continuous solid solution⁽²⁾. In addition, a "Laves" phase, probably Fe_2Mo , and the carbide M_{23}C_6 , probably $(\text{Fe, Cr, Ni})_{23}\text{C}_6$, also exist.

2.1.3.2 Isothermal Hot Leg, Type 304 Stainless Steel

Samples were exposed 3000 hours in sodium at 1300°F, 1 fps. The electron microprobe could not detect any second phases, precipitates, or sodium at or near the inside diameter surface of sample 81GB56. A quantitative measurement of composition versus distance from the surface was made. The variation in the concentrations of the primary elements were minor.

The x-ray results of the dissolved precipitates from sample 81GB55 are shown in Tables 1 and 2.

A section of sample 81GB56 was examined on the (o.d.) surface by XRD, Table 3. The presence of a ferritic or martensitic phase or both could be a result of either; (a) elemental perturbation, Cr increase and Mn depletion at the surface or (b) the transformation of γ to α' (strain-induced martensite) by cold work, which could have occurred when the sample was cut into segments for the x-ray analysis.

TABLE 3

X-RAY DIFFRACTION IDENTIFICATION OF PHASES DETECTED ON OR NEAR THE
SURFACE OF THE SAMPLES EXPOSED TO SODIUM FOR 3000 HOURS IN RUN 8-1

Identification			Outside Diameter Surface			Inside Diameter Surface		
Temperature	Na	Velocity	Major	Minor	Trace	Major	Minor	Trace
81FB52	316	SS	Sigma phase and f.c.c. matrix	--	--	f.c.c. matrix ferrite		
1305°F	3	FPS						
81GB56	304	SS	f.c.c. matrix	--	Ferrite or Martensite	Not analyzed because of physical shape		
1300°F	1	FPS						
81JC52	316	SS	f.c.c. matrix	Sigma phase	--	f.c.c. matrix	Sigma phase and $M_{23}C_6$	Possible trace; one weak line (Cr_2O_3 or Fe_2O_3)
1200°F	2	FPS						
81NA56	304	SS	f.c.c. matrix	--	--	f.c.c. matrix	--	--
800°F	2	FPS						

The microprobe data show an o.d. surface phase that is Cr rich and depleted in Mn. No other phases or precipitates were noted. The XRD analyses revealed several carbide phases and a M_3O_4 spinel structure, probably a $(Cr, Fe)_3O_4$.

2.1.3.3 Cooler #1, Type 316 Stainless Steel

Samples were exposed 3000 hours in sodium at 1200°F, 2 fps. A specimen image of sample 81JC52, including the i.d. surface and precipitates, is shown in Figure 11. The x-ray images for the major alloying elements show the surface to be rich in chromium. The x-ray image showing the distribution of chromium, the same area as in Figure 11, is shown in Figure 12. The x-ray images for Fe, Ni, Mo, and Mn gave relatively uniform distributions in contrast to chromium. The same area as in Figure 11 is shown at higher magnification in Figure 13. Line profiles along the same trace for Cr, Fe, Ni, Mo, and Mn K α x-rays are shown in Figures 14 through 18.

The results of the electron microprobe point counting, for the

determination of compositional gradients with distance from the i.d. surface, are shown in Figure 19. No detectable sodium was found associated with the grain boundaries.

The x-ray results of the selective dissolution of the precipitates from two of the samples (81JC51 and 81JC53) are shown in Tables 1 and 2. A section of sample 81JC52 was analyzed on the i.d. and o.d. surfaces by XRD. The results are shown in Table 3.

The microprobe data show that the i.d. surface is rich in Cr. The Cr appears to extend past the surface, indicating the presence of a Cr rich deposit, possibly an oxide. The concentration of Mn decreases as the surface is approached. The precipitates seen near the surface are Cr and Mo rich, with depleted areas of Fe, and Ni rich areas surrounding them. The observations described for the i.d. surface are also true for the o.d. surface area with the one exception of less precipitates. The XRD analyses reveal the presence of a sigma phase. Contrary to the 316 stainless-steel hot-leg sample, the sigma phase was only a trace

FIGURE 11. SPECIMEN CURRENT IMAGE OF COOLER SAMPLE TYPE 316 STAINLESS STEEL, SAMPLE 81JC52 INSIDE DIAMETER AREA ~1700X

FIGURE 12. X-RAY IMAGE SHOWING DISTRIBUTION OF Cr IN SAME AREA AS IN FIGURE 11, 316 STAINLESS STEEL, SAMPLE 81JC52 INSIDE DIAMETER AREA

FIGURE 13. SPECIMEN CURRENT IMAGE. MAGNIFIED SECTION OF FIGURE 11. $\sim 2800\times$

FIGURE 14. LINE PROFILE OF $\text{Cr K}\alpha$ X-RAYS SUPERIMPOSED ON SPECIMEN CURRENT IMAGE. PATH OF ELECTRON BEAM IS SHOWN BY THE STRAIGHT LINE TRACE $\sim 2800\times$. SAMPLE 81JC52 INSIDE DIAMETER

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FIGURE 17. LINE PROFILE OF $\text{Mo K}\alpha$ X-RAYS SUPERIMPOSED ON SPECIMEN CURRENT IMAGE, ALONG SAME TRACE AS IN FIGURE 14. SAMPLE 81JC52 INSIDE DIAMETER $\sim 2800\times$

FIGURE 18. LINE PROFILE OF $\text{Mn K}\alpha$ X-RAYS ALONG SAME TRACE AS IN FIGURE 14. SAMPLE 81JC52 INSIDE DIAMETER

FIGURE 19. PERCENT COMPOSITION VERSUS DISTANCE FROM THE INSIDE DIAMETER SURFACE FOR SAMPLE 81JC52 (316 SS)

phase. Also present were the carbide $M_{23}C_6$, probably $(Fe, Cr)_{23}C_6$, and a "Laves" phase, which agreed well with Fe_2Mo . The designation $(Fe, Cr)_{23}C_6$, indicates a Fe rich carbide. This is inferred from the XRF results shown in Table 2. A trace phase of the M_3O_4 spinel, most likely Fe_3O_4 , was also found. XRD of the i.d. surface also showed the sigma phase, $M_{23}C_6$ carbide, and a weak line matching the Cr_2O_3 and Fe_2O_3 compound. This agrees with the Cr rich deposit found on the i.d. surface by the microprobe.

2.1.3.4 Isothermal Cold Leg, Type 304 Stainless Steel

Samples were exposed 3000 hours in sodium at 800°F, 2 fps. The electron microprobe could not detect any second phases, precipitates, or sodium at or near the surfaces of sample 81NA56. A quantitative measurement of composition versus distance from the surfaces was made. No significant variations were seen.

The x-ray results of the dissolved precipitates from samples 81NA55 and 81NA57 are shown in Tables 1 and 2.

A section of sample 81NA56 was analyzed by XRD on both the i.d. and o.d. surfaces. The results are shown in Table 3. The sample had an extremely thin oxide layer on both surfaces. The XRD analysis could not detect any phases for the oxide. A weak trace of $M_{23}C_6$ was found on the i.d. surface. After removing the oxide layer by electropolishing, only the matrix could be identified, indicating the $M_{23}C_6$ was present as a thin layer at or near the surface.

The microprobe data did not reveal any second phase, precipitates, or compositional variations.

The x-ray examinations showed the major phase, other than the matrix, to be $M_{23}C_6$, probably $(Cr, Fe)_{23}C_6$. The designation $(Cr, Fe)_{23}C_6$ indicates a Cr rich carbide. This is inferred from the XRF results shown in Table 2. There were trace indications of numerous other carbides, such as M_7C_3 , M_3C_2 , and Fe_3C . These were not positively identified, but their lines agreed somewhat with the extra lines present. A minor phase of M_3O_4 was also found, which appeared to be close to the composition of Fe_3O_4 .

2.1.3.5 Summary

In general, the observations of this study either substantiate or extend those found and concluded from the surface XRF and metallographic examinations discussed in Section II, Subtask 1.1, 2.2, of Quarterly Progress Report No. 5. (2)

The major observations are listed below:

- a. The identification of sigma phase and its Cr-Mo-Fe constituents in the 316 SS isothermal hot-leg samples. The identification of a Fe_2Mo "Laves" phase and the $(\text{Fe}, \text{Cr}, \text{Ni})_{23}\text{C}_6$ carbide in the same samples. The "Laves" phase has not been previously observed.
- b. The findings of a $(\text{Cr}, \text{Fe})_3\text{O}_4$ oxide on the 304 stainless steel isothermal hot-leg sample is not too surprising, however, since the deposition of Cr, and probably Fe, is known for this region of the isothermal hot-leg.
- c. The identification of a Cr-Mo sigma phase layer on the i.d. and o.d. surfaces, and a Cr rich i.d. surface deposit on the samples from the Zone J cooler. The identification of the $(\text{Fe}, \text{Cr})_{23}\text{C}_6$ carbide in the same samples. The finding of a Fe_2Mo Laves phase and the identification of the Cr deposit as possibly Cr_2O_3 on these samples is an addition to previous observations.
- d. The depletion of Mn near the surfaces of the isothermal hot-leg (316 and 304 stainless steel) and Zone J cooler samples.
- e. The identification of the $(\text{Cr}, \text{Fe})_{23}\text{C}_6$ carbide and numerous trace carbides in the 304 stainless steel isothermal cold-leg samples. This observation was postulated but not substantiated previously, and is significant since it definitely indicates cold-leg carburization in a monometallic (austenitic) system. The identification of carbides in 304 stainless steel after 3000 hours exposure at 800°F is not commonly observed or reported in the literature. Also the carbides were concentrated at the surfaces, typical of carburization. The

identification of the Fe_3O_4 oxide on the surfaces of the isothermal cold-leg samples is an extension of the previous observations.

2.1.4 Metallurgical Evaluation of Results from Run 9-1

The test facility used was prototype mock-up loop 9, designed to simulate the temperature and corrosion effects of the coolant throughout a LMFBR primary system. As shown in Figure 20, the core test section, Zone F, corresponds to the core fuel and the upper blanket and gas plenum of the reactor core. Zone G corresponds to the reactor vessel upper plenum and Zone H to the primary system outlet pipe. Zones J, K, and M correspond to the heat exchanger, inlet to outlet. Zones N and R correspond to the primary system return piping, and Zone B to the primary system pump or extreme downstream position. Loop 9 is essentially the same as prototype mock-up loop 8.⁽³⁾ Operating parameters, such as maximum temperature and oxygen level, simulate the hot channel values of an LMFBR.

The loop capacity and test sample complement were designed for a study of geometry effects, such as sodium velocity, loop position and sample shape flow restriction, on the corrosion rate. The corrosion phenomena were monitored by ring and tube samples, Figure 21. A modular form of sample holder was employed that provided for sample exposure under the following conditions:

- a. Parallel sample for multiple data at the same loop position.
- b. Samples in series to study loop position, "downstream" (L/D), effects.
- c. Varied sample sizes to provide different flow cross section (velocities).
- d. Samples throughout the test facility heated, cooled and isothermal areas.
- e. Sample removal without disturbing the exposed samples.

In addition, the deposition phenomena were monitored by impinge-

FIGURE 20. SODIUM MASS TRANSFER LMFBR CORROSION MOCKUP LOOP 9

ASSEMBLED

CARTRIDGE CASE

RING SAMPLES OVER CENTRAL MANDREL

TUBE SAMPLES TO FIT IN CARTRIDGE CASE

ment samples, Figure 22. These were designed to increase the amount of deposit in major deposition areas for greater sensitivity in detecting compound species.

Cartridges of these samples were placed in Zones J and M, one at the entrance and exit of each zone.

2.1.4.1 Loop 9 Operating Parameters

The nominal operating conditions for run 9-1 were 3000 hours at 1300°F

$T_{\max}$, 800°F $T_{\min}$, a flow of 6 gpm, and an oxygen content ≤ 10 ppm based on a plugging temperature (T_s) of 260 to 280°F.

The determination of an "average" set of operating conditions for run 9-1 was straightforward. The loop operated for less than 24 hours below rated conditions. The average exposure conditions used for analysis are shown below:

Exposure Time, Hours	Flow gpm	T_s °F	Zone and Temperature, °F							
			F	G	H	J _{out}	M _{in}	N	R	B
3048	5.6	280	1303	1301	1293	1216	1076	983	916	887

2.1.4.2 Loop 9 Test Results

The materials tested were Type 316 stainless steel in Zones F, J, and M, and Type 304 stainless steel in Zones, G, H, N, R, and B. The test materials correspond to those originally proposed for use in the primary system of the LMFBR.

After exposure, the sample cartridges were removed to an inert

atmosphere (argon) chamber and transferred to an inert atmosphere glove box, where the samples were removed from the cartridges. The samples were then cleaned (reaction of sodium with alcohol and water), dried, and weighed to determine weight changes.

The impingement cartridges from Zones J & M and the cartridges from Zone B were cleaned by vacuum distillation of the sodium at 750°F

FIGURE 22. TYPICAL CAPSULE AND SPECIMENS LOADED INTO ZONE J OF LOOP 9 FOR RUN 9-1. SHOWN ARE STANDARD CORROSION AND IMPINGEMENT SAMPLES.

for two days. After distillation, the samples were removed from the cartridges, rinsed with alcohol, dried, and weighed. The deposits were scraped from some of the samples for analysis by emission spectrography, x-ray fluorescence (XRF), and x-ray diffraction (XRD). The analytical results of the examination of the deposits removed are shown in Tables 4 and 5. The material on the specimens in cartridge MA was dark in color. It was quite adherent and could be removed only by scraping except where the deposit was heavy and the outer layers could be removed by tapping. A large part of it was magnetic. Under 100X magnification, it appeared crystalline. The crystals were clumped together, probably because of their magnetic properties. A total carbon analysis showed that 2.1% of this element was present in the residue. Cyanide was detected, but only at the 1 ppm level. Complex cyanides could have escaped detection. Some of the material was mounted on a slide and covered with oil. Microscopic examination (in air) at 1,000X provided the following information: Two crystalline forms were present, one enveloping the other. The outer material was fairly clear and light brown in color;

the enclosed material was black and opaque. The clear material was present in all crystals, but occasionally the dark center material was absent. Three separate slides of the material were examined; all looked similar. Microprobe analyses of one crystal showed Cr, Mn, and Na to be present. Analysis of a small clump of crystals indicated that Fe, Ni, and Mn, were the predominant species present. XRD analysis of sample deposits from cartridge BA have shown a pattern identified as iron carbide. The deposit appeared as a black flake. An orange deposit from the same areas did not show a pattern.

As stated previously, the standard samples are designed tubes and rings. The tube samples have an i.d. surface exposure with the o.d. surface forming a slip-fit flow restricting annulus with the cartridge itself (Figure 21). The ring samples have an o.d. surface exposure with the i.d. surface forming a slip fit with the mandrel (Figure 21). The weight change data (mg) are converted into standard corrosion terminology (mg/dm^2) using only the respective tube and ring sample surface areas exposed to the flowing sodium. The weight change

TABLE 4

METALLIC COMPOSITION OF THE DEPOSITS REMOVED
FROM THE IMPINGEMENT SAMPLES IN RUN 9-1

Metal (%)	Test Cartridges			
	ZONE			
	JA	JE	MA	ME
Fe	60	(40)	10 (5)	25
Cr	20	(50)	35 (30)	15
Ni	10	(10)	35 (50)	25
Mn	10	(Trace)	20 (15)	25
Mo	2	---	0.5	0.5
Na	0.2	---	2	<0.05

()'s - x-ray fluorescence. All other data obtained by emission spectrography.

results, loop positions, and velocities for run 9-1 are shown in Table 6.

After weighing, the samples were sectioned for XRF analysis of both the o.d. and i.d. surfaces, and metallography. This included the samples cleaned by vacuum distillation, which did not have their deposits removed. The plot of weight change and corresponding XRF versus loop position downstream is shown

in Figure 23. The plot of the crevice-side surface XRF versus loop position is shown in Figure 24. The comparison of the weight change characteristics exhibited in run 9-1 versus those of runs 8-1 and 8-2⁽²⁾ are shown in Figure 25.

The typical microstructural changes observed in Type 304 and 316 stainless steel in run 9-1 are shown in Figure 26. A more detailed look at the deposits from the various

TABLE 5

X-RAY DIFFRACTION ANALYSIS OF THE DEPOSITS
REMOVED FROM THE IMPINGEMENT SAMPLES IN RUN 9-1

Test Cartridge

<u>MA</u>		<u>JE</u>
δ Fe (major)		$M_{23}C_6$ (major)
C Fe_3C $M_{23}C_6$ M_7C_3 $NaNiO_2$	$\left. \begin{array}{c} \\ \\ \\ \\ \end{array} \right\}$ (minor)	$\sigma FeCr$ Cr
α Fe α Mn unidentified amorphous substance	$\left. \begin{array}{c} \\ \\ \\ \\ \end{array} \right\}$ (trace)	

samples in the impingement cartridges is shown in Figure 27.

2.1.4.3 Discussion

The primary objective of metal-lurgical analysis of run 9-1 test results was to study the effects of impingement samples (extreme local

sodium flow disturbances) on the deposition characteristics of particulate material from flowing sodium.

The areas of the loop chosen for the study were those where the deposition was known from previous runs to be at a maximum. Accompanying

TABLE 6

WEIGHT CHANGE, LOOP POSITION, AND VELOCITY OF RUN 9-1AFTER 3048 HOURS

Test Zone	Weight Change, mg dm ⁻²		Velocity, fps	Downstream (L/D)
	Tube	Ring		
FA*	-563	-555	16.2	53.4 **
	-492	-542	↓	↑
	-492	-509		↓
	-476	-515		
	-451	-504		
	-439	-493		
	-412	-472		117.9
FB	-274	-292	5.6	131.4
	-221	-282	↓	↑
	-198	-275		↓
	-189	-270		
	-222	-262		
	-203	-258		
	-209	-280		150.3
FC	-247	-333	8.0	160.9
	-210	-318	↓	↑
	-222	-332		↓
	-206	-322		
	-209	-308		
	-210	-309		
	-224	-351		190.4
FD	-312	-358	23.1	207.2
	-323	-351	↓	↑
	-301	-334		↓
	-304	-352		
	-282	-323		
	-277	-314		
	-268	-306		301.0

*The first letter represents the test zone as shown in Figure 30. The second letter represents the cartridge, i.e., A is the first or upstream cartridge.

**The hot-leg downstream calculation starts at the inlet to the low-flux heater.

TABLE 6 (Cont'd)

Test Zone	Weight Change, mg dm ⁻²		Velocity, fps	Downstream (L/D)
	Tube	Ring		
FE	-334	---	3.9	316.0
	-289	---	↓	↑
	-244	---		
	-240	---		
	-246	---		
	-234	---		
	-230	---		323.8
FF	-230	-291	11.4	334.7
	-201	-278	↓	↑
	-204	-272		
	-192	-280		
	-196	-274		
	-213	-273		
	-194	-289	↓	378.8
FG	-282	---	3.9	389.7
	-244	---	↓	↑
	-230	---		
	-247	---		
	-235	---		
	-220	---		
	-221	---	↓	397.5
FH	-204	-250	16.2	410.1
	-217	-258	↓	↑
	-205	-252		
	-204	-253		
	-200	-242		
	-218	-243		
	-217	-245	↓	474.6
GA	-142	---	2.4	494.5
	-99	---	↓	↑
	-93	---		
	-92	---		
	-94	---		
	-93	---		
	-88	---		
	-102	---		500.7
GB	-148	---		506.3
	-104	---		↑
	-94	---		
	-89	---		
	-86	---		
	-82	---		
	-81	---	↓	512.4
	-97	---		

TABLE 6 (Cont'd)

Test Zone	Weight Change, mg dm ⁻²		Velocity, fps	Downstream (L/D)
	Tube	Ring		
HA	-68	---	1.3	536.7
	-65	---		↓
	-66	---		
	-58	---		
	-61	---		
	-66	---		
	-65	---		541.2
HB	-67	---	1.3	546.7
	-61	---		↓
	-59	---		
	-59	---		
	-51	---		
	-55	---		
*	-58	---	1.3	551.2

Test Zone	Tube	Washer**	Disk**	Velocity, fps	Downstream (L/D)
JA		44		14.3	↑
	38			3.5	
			52	14.9	
	39			3.5	
		47		14.3	
	38			3.5	
			43	14.9	
	47			3.5	
		66		14.3	
	34			3.5	
			55	14.9	
	61			3.5	
		83		14.3	
	46			3.5	
			72	14.9	
	70			3.5	
		---		14.3	
					33.8

* The cold-leg downstream calculation starts at the inlet to Zone J (first cooler).

** Flow restriction samples from the impingement cartridges, shown in Figure 22.

TABLE 6 (Cont'd)

Test Zone	Tube	Ring	Velocity, fps	Downstream (L/D)
JB	22	---	3.5	43.0
	22	---		
	13	---		
	12	---		
	23	---		
	20	---		
	40	---		50.4
JC	68	---		59.4
	52	---		
	54	---		
	47	---		
	55	---		
	48	---		
	55	---		66.8
JD	85	---	3.5	75.8
	77	---		
	79	---		
	72	---		
	76	---		
	76	---		
	84	---		83.2
JE	<u>Tube</u>	<u>Washer</u>	<u>Disk</u>	
	---	342	---	14.3
	---		---	3.5
	---		---	14.9
	427			3.4
	---	359		14.3
	---			3.5
	---		299	14.9
	368			3.5
	---	366		14.3
	292			3.5
	---		274	14.9
	---			3.5
	---	328		14.3
	302			3.5
	---		294	14.9
	---			3.5
	---	389		14.3
				102.3

TABLE 6. (Cont'd)

Test Zone	Tube	Washer	Disk	Velocity, fps	Downstream (L/D)
MA		1184		14.3	233.7
	---			3.5
			---	14.9	
	2002			3.5	
		1450		14.3	
	---			3.5	
			1381	14.9	
	---			3.5	
		1291		14.3	
	---			3.5	
			---	14.9	
	1287			3.5	
		---		14.3	
	878			3.5	
MB			---	14.9
	1046			3.5	
		---		14.3	
MC
MD

TABLE 6 (Cont'd)

Test Zone	Tube	Washer	Disk	Velocity, fps	Downstream (L/D)
ME		247		14.3	301.6
	358			3.5	
			366	14.9	
	429			3.5	
		296		14.3	
	251			3.5	
			254	14.9	
	310			3.5	
		261		14.3	
	209			3.5	
			216	14.9	
	313			3.5	
		257		14.3	
	204			3.5	
NA			208	14.9	
	278			3.5	
		274		14.3	312.2
NB					
NC					
ND					

TABLE 6 (Cont'd)

Test Zone	Tube	Ring	Velocity, fps	Downstream (L/D)
NE	18	51	3.9	469.8
	12	40		↑
	26	28		↓
	27	32		↓
	7	32		↓
	21	28		475.5
NF	52	36	11.4	484.9
	61	33		↑
	34	33		↓
	35	33		↓
	54	36		↓
	54	37		504.1
RA	14	---	1.3	616.7
	9	---		↑
	12	---		↓
	8	---		↓
	6	---		↓
	9	---		↓
RB	4	---		621.2
	12	---		628.9
	6	---		↑
	8	---		↓
	3	---		↓
	8	---		↓
RC	5	---		633.4
	10	---		↓
	12	---		641.1
	9	---		↑
	4	---		↓
	9	---		↓
BA	9	---	99.3	645.6
	5	---		↓
	13	---		768.3
	138	---		↑
	188	---		↓
	100	---		↓
	162	---		786.4
	137	---		↓
	212	---		↓
	---	---		↓
	288	---		↓
	150	---		↓
	---	---		↓
	162	---		↓
	150	---		↓

FIGURE 23. WEIGHT CHANGE AND EXPOSED SURFACE X-RAY FLUORESCENCE DATA VERSUS LOOP POSITION FOR RUN 9-1 AFTER 3000 HOURS EXPOSURE

FIGURE 24. CREVICE SURFACE X-RAY FLUORESCENCE DATA VERSUS LOOP POSITION FOR RUN 9-1 AFTER 3000 HOURS EXPOSURE

FIGURE 25. COMPARISON OF THE WEIGHT CHANGE DATA VERSUS LOOP POSITION FOR RUNS 8-1, 8-2, AND 9-1.

(REDUCED TO 85% FOR REPRODUCTION PURPOSES)

FIGURE 26. TYPICAL MICROSTRUCTURAL EFFECTS OF TUBE SAMPLES (INSIDE DIAMETER EXPOSURES) AFTER 3000 HOURS EXPOSURE TO FLOWING SODIUM DURING RUN 9-1

WASHERS

DISKS

(REDUCED TO 95% FOR
REPRODUCTION
PURPOSES)

FIGURE 27. SURFACE MICROSTRUCTURES AND DEPOSIT
CHARACTERISTICS OF THE IMPINGEMENT
SAMPLES FROM RUN 9-1

the study of impingement effects on deposition was the study of the effects, if any, on corrosion. A secondary objective of the analysis was to look further at the effects of velocity on corrosion.

The general dependency of mass transfer (corrosion and deposition) on loop position (downstream), velocity, mass flow, etc., reported previously⁽³⁾, are again evident from the run 9-1 results. The downstream effect of decreasing corrosion and deposition with position change in an isothermal region can be seen in Table 6 and Figure 23. The increase of corrosion and deposition with increasing velocity can also be seen in Table 6 and Figure 23.

The higher corrosion and deposition of the first sample or samples in a cartridge "entrance effect" is shown in Table 6. All of these characteristics of a non-isothermal flowing sodium system have been discussed previously.⁽³⁾

The consistently observed relationship⁽³⁾ of higher corrosion values for the tube samples versus the ring samples has been reversed in Zone F, Table 6 and Figure 23. The

effect can be a result of using only the surface area exposed to flowing sodium for the corrosion calculation. However, the cause of this effect might be an additional one; appreciable corrosion in the low velocity flow passage (crevice) between the ring sample and the mandrel (slip fit) as compared to the tube sample. This hypothesis is supported by considering construction details of cartridges. Tubing of a larger wall thickness (than previously encountered in Loop 8) was used to obtain the various velocities through Zone F. Cartridge end plug or cones did not completely cover the edge of the first and last ring samples in the Zone F cartridges. It is possible to envision the incoming sodium having a direct path along the crevice side of the ring samples. Further, if the crevice corrosion contribution is kept constant for all Zone F cartridges, the effect on weight change-per unit area would be expected to be the greatest of the lower velocity cartridges because of the lower corrosion on the flowing side of the samples. This is the case as can be seen in Figure 23, where cartridges FB, FC, and FF (low velocities) show greater effect than cartridges FA, FD, and FH (high velocities). The rate calculation using only the surfaces

exposed to flowing sodium is chosen for the present as a conservative (upper limit) value of the corrosion rates.

2.2 SUB-TASK 1.2 - RATIONAL CORROSION, DEPOSITION MODELS

The objective of this task is to develop the physical and chemical models describing mechanisms of mass transport and corrosion in order to extrapolate the empirical observations on small loops to large systems.

2.2.1 Surface Chemical Reaction Kinetics and Its Dependence on Velocity, Temperature, Oxygen Activities, and Reaction Compounds

The relationship between the diffusion in the sodium, diffusion in the solid metal, and the kinetics of chemical reactions at the surface are being evaluated with respect to the corrosion and deposition process at the present time. The effect of sodium velocity on corrosion rate (Figure 1) suggests that the rate determining step in the corrosion process is velocity dependent. The phase II and UK data show that between 0 to ~ 15 fps, the diffusion of solutes (corrosion products)

across the liquid boundary layer is rate determining. At higher velocities, the liquid boundary layer diminishes and either the atomic dissolution or the kinetics of some chemical reaction e.g., formation of a complex oxide at the surface prior to dissolution, becomes the rate controlling step.⁽⁴⁾ The sketch below illustrates one of the possible mechanisms.

It is believed that at low velocity, reactions (1) and (2) are rate determining. Reaction (1) is the transfer of oxygen from the sodium to the corroding surface, and reaction (2) is the transfer of corrosion product "MO" from the surface into the sodium through the liquid boundary layer. At higher velocities, greater than 12 to 15 fps for example, the liquid boundary layer diminishes, and reactions (3) and (4) become rate controlling. Reaction (3) is the formation of

"MO" at the surface, and reaction (4) is the diffusion of alloy constituents in the solid.

Possible surface reactions based on oxygen activities, temperature, and thermodynamic stabilities of various sodium complexes are being evaluated at the present time and are being correlated with observations of the deposition behavior at lower temperature in cold-legs of loops. Although there may be some diffusion into the solid metal surfaces after deposition, the chemical activity of the solid does not play a very significant role in the kinetics of deposition. It is postulated that either the nucleation and growth of the corrosion products in liquid sodium or the transfer across the liquid boundary layer to the depositing surface is rate determining. Deposition rates (Figure 2) within the experimental uncertainties, seem to indicate that diffusion of the nucleated particles across the boundary layer is rate controlling, at least up to 15 fps. It can also be explained by the degree of turbulence, increasing with increased velocity, producing an increase in the number of particles colliding with the deposition surface, con-

sequently increasing the probability of deposition.

2.2.2 Carbon Transport in Austenitic Stainless Steel Loops

Carburization of the 316 stainless steel hot-leg and decarburization of the 2 1/4 chromium-ferritic cold-leg in bimetallic systems has been observed on many occasions. Metallographic examinations of samples from runs 8-1, 8-2, 8-3, and 9-1 indicate that unlike bimetallic loops, carbon is transferred from the hot-leg to the cold-leg in monometallic (austenitic) loops. Metallographic observations were further verified by electrolytic extraction and subsequent x-ray analysis of hot and cold-leg samples from run 8-1 (see Section 2.1.3).

The total carbon analysis of the samples from run 8-3 for the determination of carbon transport is in progress.

The above observations concerning carbon transport can create serious problems in the design and operation of an Intermediate Heat

Exchanger (IHX). The IHX tubes will not only pick up carbon from the secondary side of the ferritic steam generator but may also be carburized on the primary side. This may significantly impair the mechanical properties and expected lifetime of the IHX tubes.

One of the explanations for the carbon transport in a bimetallic loop is that the activity coefficients of carbon (γ_c) in plain carbon or low-alloy ferritic steels are much higher than the γ_c in austenitic stainless steels. Hence, as a first approximation, if one neglects the effect of the temperature coefficients of relative partial excess free energies for carbon steel ($\bar{\Delta} G_c^{xs}$, in other words, the entropy effects) the transfer of carbon from ferritic steels to austenitic stainless steels bridged by liquid sodium is necessary to bring the entire system in equilibrium with respect to carbon. Unfortunately, the carbon transport from hot-leg to cold-leg in a monometallic loop cannot be explained by the same argument. Carbon in solution in austenitic stainless steels shows a relatively large positive deviation from ideality, which tends to become

more ideal as the temperature is increased. In other words, the activity coefficients for carbon in austenitic stainless steel (γ_c) decreases with increasing temperature. From equilibrium considerations, the hot-leg should carburize and the cold-leg should decarburize as in the case of the bimetallic loops. However, it must be noted that the difference of γ_c between ferritic steels and austenitic stainless steels is much greater than the difference of γ_c in austenitic stainless steel at two different temperatures (only the entropy effect). Therefore, one may expect that the carbon movement in austenitic stainless steel systems will be much less, but a complete reversal of the carbon transport cannot be explained solely on the activity coefficient considerations. In order to explain rationally the mode of carbon transport in a monometallic stainless steel system, one must take into consideration the variation of γ_c in liquid sodium throughout the entire system. It is reasonable to assume that γ_c in sodium will depend on the other solute elements, e.g., Cr, Mo, Mn, Fe. Stable carbides of Cr, Mo, and Mn (order of decreasing stability) are formed

at these temperatures, and one may expect the interaction parameter for carbon

$$\frac{\delta \ln \gamma_c \text{ in liquid Na}}{\delta x_i}$$

Where $i = \text{Cr, Mo, Mn, Fe, etc.}$, to be negative.

The presence of carbide forming elements in solution in liquid sodium will tend to decrease the activity coefficients of carbon. From this, one can postulate that the presence of corrosion products in the hot leg, especially Cr (forms most stable carbide), will decrease the γ_c in liquid sodium in the hot-leg. Consequently,

more carbon will be transferred to the sodium. When Cr deposition begins in the coolers (as observed from x-ray fluorescence data), the γ_c in the sodium increases. This increase coupled with the decreasing temperature causes the cold-leg specimens to carburize. Preliminary analysis of samples from run 8-3 tends to substantiate the hypothesis.

A new design for a particulate collection loop has been completed in order to locate the loop position of the precipitates and subsequently identify them and to obtain quantitative data on the contribution of particulates to mass transport rates.

SECTION III

TASK 2 - SODIUM-IMPURITIES INTERACTIONS AND IDENTIFICATION OF PARTICULATE MATTER

The sodium impurities task covers the chemical monitoring of test loops. Non-contaminating sampling methods, along with sensitive methods of detection and

analysis, are used for concentrations, separation, and identification of compounds at ppm levels. Sodium cover gas and deposited materials (surfaces, plugging indicators, and

cold traps) are analyzed. The long-range objective is to determine the control and levels of impurities that affect corrosion mechanisms.

3.1 SUB-TASK 2.1 - SAMPLING OF SODIUM SYSTEMS

The objective of this task is to utilize sensitive means for determining impurities in sodium in the various test loops and to identify and eventually control the species affecting mass transfer and mechanical properties.

3.1.1 Sodium Distillation

Distillation equipment is in the process of being checked out and connected to Loop 8 for continuous, automatic operation. A description of the station and its designed functions was given in GEAP-5602, Quarterly Progress Report No. 4, December 1967 to February 1968.

Modifications were made on the weighing transducer support mechanism in an attempt to eliminate a "chatter" at the low signal output voltage. Inverting the transducer to provide a constant load at all times has made some improvement. Further steps are being taken to desensitize the

amplifier which is expected to reduce or eliminate the "chatter."

An argon gas system was added to eliminate potential interface problems with other tests being conducted in the same loop by the introduction of helium, the normal cover gas used during distillation. The argon will be introduced during initial sodium circulation steps when the lines are being cleaned and brought up to isothermal conditions prior to distillation and during transfer of the distillate from the station to the loop.

3.1.2 Oxygen Meter

The design of an installation of a UNC oxygen meter on Loop 8 for operation in parallel with the distillation equipment is complete. Figure 28 is a sketch describing the basic installation package. The source of sodium will be initially the sampling manifold in the cold leg of Loop 8 at about 875° to 900°F that is also supplying the distillation rig. The unit contains the sensing instrument, an economizer plus cooler (to bring the sodium temperature at 1 gpm from a maximum 900°F down to 600°F), a flowmeter and the control and read-out instruments.

FIGURE 28. OXYGENMETER INSTALLATION FOR LOOP 8

It is independent of any loop and can be connected with its flanges in any desired location.

The oxygen-meter control and recording system was redesigned during the past quarter since the oxygen-meter signal is strongly influenced by ambient-temperature fluctuations. The design was modified to record the oxygen-meter signal as well as the environmental factors that influence it. The oxygen-meter sensor is now contained in an insulated box which will be supplied with air at a constant temperature of $72^{\circ}\text{F} \pm 2^{\circ}\text{F}$. The oxygen-meter signal, the sodium temperature, ambient air temperature, and the sodium flow rate will be recorded.

3.1.3 Cover Gas Monitoring by Gas Chromatography

The equipment for automatic gas sampling and analysis (initially for hydrogen and nitrogen) has been described in GEAP-5602, Quarterly Progress Report No. 4. Gas sampling lines and the chromatograph were moved to a position adjacent to loop 8 and steps taken to provide constant ambient temperature control.

3.1.4 Surveillance Analyses

The preceding Quarterly Report No. 5, GEAP-5648, described the sampling and analytical techniques used in routine analyses of the coolant from test loops. During the current reporting period, the flow-through pipe technique has been extended to sampling sodium from the loop-9 hot leg.

Total alkalinity analysis have been performed on one of these samples and results are shown in Table 7. Shown also are results obtained on a sample taken from the cold leg of the same loop at about the same time.

The agreement of the results from the two sampling points within experimental error leads to the tentative conclusions that the extension of the technique to the higher temperature regions has been successful (i.e., the hot sample), that it is representative of the system, and that contamination by the atmosphere is no greater than when sampling the cold leg.

TABLE 7

COMPARISON OF TOTAL ALKALINITY BETWEEN
HOT AND COLD LEG SAMPLE RESIDUES

<u>Sample No.</u>	<u>Date Taken</u>	<u>Sampling Temperature</u>	<u>Total* Alkalinity (ppm)</u>
9-2-6C	6/14/68	750°F	12.2
(Cold Leg)			10.8
9-2-6D	6/14/68	1100 - 1200°F	15.5
(Hot Leg)			15.4

*Not corrected for hydride. This correction will be made if hydrogen analyses indicate total alkalinity to be lower.

3.2 SUB-TASK 2.2 - ANALYTICAL CHEMISTRY

procedure has been completed and is reported in Table 8.

3.2.1 Cold Trap Examination (Loop 1)

The previous report (GEAP-5648) described the nature and distribution of carbon containing deposits found in a cold trap which had seen service in an austenitic loop. (Loop 1, 316 stainless steel, phase 1).

The determination of total alkalinity by the amalgamation

The zones refer to sections of the trap along its length from inlet to outlet corresponding roughly to those examined in the earlier cold trap study (Quarterly Progress Report No. 1, GEAP-5508).

No attempt has been made as yet to distinguish among the alkaline species present, i.e., oxide, hydride, hydroxide, and carbonate, but on the basis of previous work it can be

assumed that sodium oxide is the major species. Based on this assumption an examination of the data indicates that only a small fraction of the trap capacity for sodium oxide has been depleted even after a 20,000

hour operational period for the loop at the end of which time it was still possible to maintain oxygen levels so that plugging indicator breaks of about 300°F (≈ 10 ppm O) could be attained.

TABLE 8

TOTAL ALKALINITY IN THE LOOP 1 COLD TRAP

<u>Zone</u>	<u>Total Alkalinity*</u> <u>(ppm)</u>
1	100
2	2,060
3	10,300
4	4,200

*Calculated on the basis of Na_2O , after sodium removal.

Total hydrogen results were also obtained for several of the samples and are shown in Table 9.

These results confirm the earlier (Loop 5) examination data which showed the trap to be a deposition zone for hydrogen compounds.

3.2.2 Round Robin Analyses -
Oxygen in Sodium by
Amalgamation

Work was completed and reported on the analyses of 12 samples of sodium prepared by APDA for the

TABLE 9

TOTAL HYDROGEN IN LOOP 1 COLD TRAP SAMPLES

<u>Zone</u>	<u>Total Hydrogen</u> <u>(ppm)</u>
1	104
2	232
4	110
5	38

Atomic Energy Commission to test the proposed ASTM standard method for measuring oxygen by the amalgamation technique. Both the alkalinity and the sodium content of the residue separated by amalgamation were

measured in assessing the oxide content. The precision obtained for six replicate runs at each of two oxygen levels was quite satisfactory, as is shown in Table 10.

TABLE 10

ROUND ROBIN OXYGEN DETERMINATIONS

<u>Sample</u>	<u>Oxygen (ppm)</u>
Low Level Oxygen	5.42 ± 0.36 (titration)
	5.82 ± 0.83 (flame photometry)
High Level Oxygen	26.78 ± 2.10 (titration)
	25.33 ± 1.79 (flame photometry)

3.2.3 Particulate Identification

Specific measurements for the elemental constituents of the residues obtained in the impingement study (Loop 9 Run 9-1) and the normal

corrosion deposits from corresponding locations in Loop 8 have been completed.

The results are shown in Table 11.

TABLE 11

ELEMENTAL COMPOSITION OF COLD LEG CORROSION DEPOSITS

<u>Element</u>	<u>Loop 8</u> <u>(%)</u>	<u>Loop 9</u> <u>(%)</u>
Cr	22.6 ⁽¹⁾	35.2
Fe	9.3	6.0
Ni	19.0	25.4
Mn	8.7	7.3
Na ⁽²⁾	9.7	10.0
C ⁽³⁾	3.0	2.0

(1) Results good to $\pm 5\%$ except carbon $\pm 15\%$.

(2) Na analysis by flame photometry.

(3) C analysis by wet combustion.

The first four elements appear to be in the concentration ranges expected, based upon the x-ray fluorescence measurements performed on the surfaces of corrosion specimens exposed in the same loop positions.

The carbon levels show increases by factors of 20 and 30 over the concentration of this element in the parent metal. The release of hydrocarbon gases upon the addition of water and dilute acid was observed and indicates that carbides are

present. These results confirm the previously reported x-ray diffraction results which showed that patterns for carbides of the types $M_{23}C_6$ and M_7C_3 were present.

3.3 SUB-TASK 2.3 - STUDY OF PARTICULATE MATERIAL

Design revisions for an experiment in Loop 1 have been completed. The purpose of the test is to generate corrosion particles under known conditions and to collect them and analyze their chemical compositions and physical properties. A schematic

diagram for the test is shown in Figure 29.

The sodium entering the heated leg will be filtered so no particles greater than 5 microns can be carried in by the sodium. After the filter, the sodium is heated from 500°F to 1300°F and sent through a sample section for generating corrosion products. The sodium is then cooled to 1000°F in the shortest possible time and passed through a magnetic field to separate magnetic particles. This step is repeated after the sodium is cooled to 600°F in a second cooler section.

SECTION IV

TASK 3 - MECHANICAL PROPERTIES

OF FERRITIC MATERIALS IN BI-METALLIC SYSTEMS

4.1 TEST OBJECTIVES AND EXPERIMENTAL REQUIREMENTS

The objective of this Task is to determine effects on design properties of 2-1/4 Cr-1 Mo as a result of carbon transport in a bimetallic system. The initial phase is limited to review of available data and determination of test

objectives and experimental requirements. Samples to measure property changes are available from loop components from phase 1 of the program.

The test objectives and requirements for testing 2-1/4 Cr-1 Mo have been determined and forwarded to RDT by letter. (5)

FIGURE 29. PRELIMINARY DESIGN FOR "PARTICULATE" STUDY

SECTION V

TASK 4 - MECHANICAL PROPERTIESLIMITS FOR CLADDING MATERIALS5.1 DESIGN CRITERIA FOR CLADDING
CANDIDATES

Work under this sub-task includes a screening phase of several candidate materials for LMFBR fuel cladding (Type 304, 316, 347 stainless steel and Incoloy 800) in preparation for obtaining design data, long-term stress-rupture (to 10,000 hours), and stress fatigue data (to 6,000 hours). Present emphasis is on test conditions of 1300°F, sodium velocities of 17 fps and controlled sodium purity corresponding to < 300°F plugging temperatures.

5.1.1 Summary of Stress-Rupture
Screening Runs in Loop 5R

The purpose of the stress-rupture screening phase was to reduce 12 material-structural combinations to the four best combinations for in-depth stress-rupture data runs. The test facility used was loop 5R, described previously.⁽⁶⁾ The

nominal test conditions of the loop were 1300°F $T_{\max}$, $\Delta T = 425$ F, $T_s \approx 280^\circ\text{F}$ ($O_2 < 10$ ppm), and a sodium flow of 10 gpm.

Statically pre-pressurized pins⁽⁷⁾ of Types 304, 316, 347 stainless steel and Incoloy 800 in the mill-annealed, carbide agglomerated, 20% cold reduced conditions were exposed for 1745, 2085, and 3044 hours in 1300°F sodium. Some additional samples were tested at 1300°F for 1008 hours in loop 6R, the stress cycle fatigue facility, which is identical to Loop 5R. The material used for the pressurized pins was 0.250 inch o.d. X 0.015 inch wall weldrawn tubing, shown in Figure 30.

The use of a pre-pressurized sample does not allow for failure monitoring in a flowing sodium system. Therefore, the results of the tests, Tables 12 through 15, are failure no-failure information with corresponding $\Delta D/D$'s.

TABLE 12

TYPE 316 SS BIAXIAL STRESS-RUPTURE RESULTS IN 1300°F SODIUM
(0.250 IN. O.D. X 0.015 IN. WALL WELDRAWN TUBING)

} HOOP STRESS, psi () % $\Delta D/D$
 >NO FAILURE
 <FAILURE

Structural

Condition	7000	9000	10,000	11,000	12,000	13,000	14,000	16,000	20,000
Mill Annealed	J2085	>1008(0) >2085(0) >3044(1)	>1745(1) <3044(9)	>1745(2) <3044(8)	>1008(2) <3044(10)		>2085(8)		
Carbide Agglomerated*	>2085(0)	>2085(0)	>1745(3) >3044(6)	>1745(4) <3044(16) >3044(6)	<3044(15)	<3044(14)	<2085(10)		
Cold** Reduced					>3044(0)	>1008(0) >3004(0)	<1745(1) <3044(0)	<3044(0)	>1008(0)

*Mill annealed material heat treated at 1650°F for 24 hours.

** 20%

NOTES:

1. The specimens at 1745 and 2085 hours contained filler plugs to reduce the gas volume. Because of this, the hoop stress drops off rapidly with expansion. This is especially critical at hi-stress, short failure time exposures where the expansion is the greatest.
2. The specimens at 1008 and 3044 hours had no filler plugs. With this configuration the hoop stress is nearly constant with expansion.

TABLE 13

TYPE 304 SS BIAXIAL STRESS-RUPTURE RESULTS IN 1300°F SODIUM
(0.250 IN. O.D. X 0.015 IN. WALL WELDRAWN TUBING)

}>No Failure
 }<Failure
 HOOP STRESS, psi ()% ΔD/D

Structural Condition	4000	5000	6000	7000	8000	9000	10,000	11,000	12,000	13,000	14,000	20,000	21,750
Mill Annealed	>3044 (2)	>3044 (4) <3044 (8)	>3044 (2)	>1745 (4) >2085 (5) <2085 (7) <3044 (8)	<1745 (6)	>1008 (4) <2085 (8)			<1008 (2)		<2085 (9)		
Carbide Agglomerated*	>3044 (1)	>3044 (4) <3044 (5)	<3044 (6)	>1745 (3) >2085 (4) <2085 (2) >3044 (2)	>1745 (6)	<2085 (9)					<2085 (11)		
Cold Reduced**						>3044 (0)	>3044 (1) <3044 (2)	<1745 (2) <3044 (2)		>1008 (0)	<2085 (1)	>1008 (0)	<2085 (1)

*Mill annealed material heat treated at 1650°F for 24 hours

** 20%.

NOTES:

1. The specimens at 1745 and 2085 hours contained filler plugs to reduce the gas volume. Because of this, the hoop stress drops off rapidly with expansion. This is specially critical at hi-stress, short failure time exposures where the expansion is the greatest.
2. The specimens at 1008 and 3044 hours had no filler plugs. With this configuration, the hoop stress is constant with expansion.

TABLE 14

TYPE 347 SS BIAxIAL STRESS-RUPTURE RESULTS IN 1300°F SODIUM

(0.250 IN. O.D. X 0.015 IN. WALL WELDRAWN TUBING)

>No Failure }
 <Failure } HOOP STRESS, psi ()% $\Delta D/D$

Structural Condition	7000	9000	10,000	12,000	13,000	14,000	20,000	21,750
Mill Annealed	>2085(0)	>1008(0) >1745(2) <1745(2) <2085(2)	<1745(2)	<1008(2)		<2085(1)		
Carbide Agglomerated	>2085(2)	<1745(5) <2085(4)	<1745(6)			<2085(2)		
Cold Reduced					<1008(2) <1745(1)	<2085(1)	<1008(10)	<2085(1)

*Mill annealed material heat treated at 1650°F for 24 hours.

** 20%.

Notes:

1. The specimens at 1745 and 2085 hours contained filler plugs to reduce the gas volume. Because of this, the hoop stress drops off rapidly with expansion. This is especially critical at hi-stress, short failure time exposures where the expansion is the greatest.
2. The specimens at 1008 and 3044 hours had no filler plugs. With this configuration, the hoop stress is constant with expansion.

TABLE 15

INCOLOY-800 BIAxIAL STRESS-RUPTURE RESULTS IN 1300°F SODIUM
(0.250 IN. O.D. X 0.015 IN. WALL WELDDRAWN TUBING)

Structural Condition	<div> <div>>No Failure <Failure</div> <div>HOOP STRESS, psi ()% ΔD/D</div> </div>								
	6000	7000	8000	9000	11,000	12,000	14,000	20,000	21,750
Mill Annealed	<3044(4)	>2085(0)	>1745(0)	>1008(0) >1745(0) <1745(0) >2085(0) <3044(2)		<1008(0)	<2085(1)		
Carbide Agglomerated		>2085(0)	>1745(0)	>1745(0) <1745(1) >2085(0) <3044(8)	<3044(8)		<2085(1)		
Cold Reduced						<1745(0) <3044(1)	<2085(0) <3044(2)	>1008(0)	<2085(0)

*Mill annealed material heat treated at 1650°F for 24 hours.

** 20%

NOTES:

1. The specimens at 1745 and 2085 hours contained filler plugs to reduce the gas volume. Because of this, the hoop stress drops off rapidly with expansion. This is especially critical at hi-stress, short failure time exposures where the expansion is the greatest.
2. The specimens at 1008 and 3004 hours had no filler plugs. With this configuration, the hoop stress is constant with expansion.

The mill annealed and carbide agglomerated specimens at 3044 hours were of a different heat of material.

The typical stress-rupture characteristics are shown in Figures 31 through 34, which also show the typical effects of the sodium exposure on the surface microstructure exclusive of stress.

The Type 347 stainless steel and Incoloy 800 samples all failed in the tube seam welds, with the creep localized in these areas (see Figure 33 and 34). Metallographic examination of the welds (Figure 30) show that the 347 stainless steel had the most homogeneous weld. Type 316 stainless steel exhibited the poorest appearing weld, but the best behavior. Subsequent scoping tests on two different heats of the four test alloys in 1300°F argon showed the weld performance to be a function of the heat of material and not a basic property of a particular alloy. (8) The 20% cold reduced 304 and 316 stainless steel also failed in the seam welds. Due to the inability to determine the "quality" of incoming weldrawn tubing (on the basis of metallography, chemical composition, etc.), seamless tubing will be used exclusively for future stress-rupture data runs.

Comparing the microstructures of the pressurized pins exposed to

1300°F flowing sodium and their companion unstressed samples (Figures 31 through 34) leads to the conclusion that no effect of applied stress on corrosion characteristics occurred in 3000 hours. It has been previously assumed that the effects of sodium mass transfer on the mechanical properties would be quite subtle. With the development of compositionally modified zones a few mills thick in 3000 hours, it is evident that tests in sodium approaching 10,000 hours must be undertaken if the sensitivity of properties to flowing sodium environment is to be measured quantitatively.

The best-fit stress-rupture curves for 304 and 316 stainless steel are shown in Figure 35. The slope of these curves is arbitrary due to the "go no-go" nature of the data. The information gathered on the various structural conditions of the two alloys, however, can be compared relative to each other with these curves. The observations are: (a) Carbide agglomerated 316 stainless steel has better stress-rupture properties than annealed 316 stainless steel; (b) the 20% cold reduced materials have higher stress-rupture strengths up to 3000 hours; and (c)

TYPE 316 STAINLESS STEEL

TYPE 304 STAINLESS STEEL

25 μ

TYPE 347 STAINLESS STEEL

INCOLOY - 800

FIGURE 30. MATERIAL USED FOR SCREENING RUNS IN LOOPS 5R AND 6R
(Weldrawn Pressure Tubing - Weld Areas 0.250 in. o.d. x 0.015 in. Wall)

2083 HOURS
H.S. = 14,000 psi GLYCEREGIA 500X

2083 HOURS UNSTRESSED
GLYCEREGIA 500X

1745 HOURS UNSTRESSED
GLYCEREGIA 500X

NOTE: H.S. = HOOP STRESS

(REDUCED TO 75% FOR REPRODUCTION PURPOSES)

FIGURE 31. TYPE 316 STAINLESS STEEL – BIAXIAL STRESS RUPTURE VERSUS MASS
TRANSFER STRUCTURAL EFFECTS IN 1300°F FLOWING SODIUM

2083 HOURS
H.S. = 7,000 psi HCl - H₂O₂ 25X

2083 HOURS
H.S. = 14,000 psi HCl - H₂O₂ 250X

2083 HOURS
H.S. = 14,000 psi HCl - H₂O₂ 500X

1745 HOURS UNSTRESSED
GLYCERIA 500X

NOTE: H.S. = HOOP STRESS

(REDUCED TO 75% FOR REPRODUCTION PURPOSES)

FIGURE 32. TYPE 304 STAINLESS STEEL - BIAxIAL STRESS RUPTURE VERSUS MASS TRANSFER STRUCTURAL EFFECTS IN 1300°F FLOWING SODIUM

2083 HOURS
H.S. = 14,000 psi 10% OXALIC 100X

2083 HOURS
H.S. = 9,000psi 200X

2083 HOURS 200X
H.S. = 14,000 psi

2083 HOURS
H.S. = 14,000 psi 10% OXALIC 500X

2083 HOURS UNSTRESSED
10% OXALIC 500X

1745 HOURS UNSTRESSED
GLYCEREGIA 500X

(REDUCED TO 75% FOR REPRODUCTION PURPOSES)

NOTE: H.S. = HOOP STRESS

**FIGURE 33. TYPE 347 STAINLESS STEEL – BIAXIAL STRESS RUPTURE VERSUS MASS TRANSFER
STRUCTURAL EFFECTS IN 1300°F FLOWING SODIUM**

2083 HOURS

H.S. 14000 psi 10% OXALIC

100X

2083 HOURS, UNSTRESSED

10% OXALIC

500X

2083 HOURS

H.S. 14,000 psi 10% OXALIC

500X

1745 HOURS, UNSTRESSED

GLYCERIC

500X

(REDUCED TO 75% FOR REDUCTION)

NOTE: H.S. = HOOP STRESS

**FIGURE 34. INCOLOY-800 – BIAxIAL STRESS RUPTURE VERSUS MASS TRANSFER
STRUCTURAL EFFECTS IN 1300°F FLOWING SODIUM**

FIGURE 35. B AXIAL STRESS RUPTURE CURVES AT 1300° F

no effect of annealed versus carbide agglomeration could be noted for 304 stainless steel. Data obtained in 1300°F vacuum for annealed and carbide agglomerated under constant pressure conditions (Table 16 and Figure 35), however, show a decrease in stress-rupture properties for the carbide agglomerated material. It had been noted previously that the microstructural response of 304 stainless steel to the heat treatment was nil, while 316 stainless steel showed a definite occurrence of agglomerated carbides in the grain boundaries and at triple points. An examination of the 304 stainless steel carbon isoplethal section shows that a 304 stainless steel with a lower level of carbon (0.04%) will not respond at 1650°F in 24 hours.

The stress rupture data in sodium at 1300°F for 304 and 316 stainless steel are compared to the Atomic International (AI) data⁽⁹⁾ at 1200 and 1400°F in Figures 36 and 37. The Reference 9 specimens were pressure controlled and exposed in static sodium autoclaves. It should be noted that the 1300°F data reported here fall within the 1200 and 1400°F Reference 9 data, the only

other set of data reported in the literature which can be compared with the results reported here. Only qualitative comparison can be made since the two sets of data were obtained under different environmental conditions, as well as different chemistry. (Stagnant sodium with no ΔT for the work in Reference 9, versus flowing sodium with about 350°F ΔT for this work). The main expected difference is the mass transfer of carbon. The data with no ΔT in stagnant sodium is easier to obtain but the application to practical systems is less clear. The error bands reported in Figures 37 and 38 can be reduced only when more data become available.

One final comparison is made in Figure 38, where the annealed 304 stainless steel data in 1300°F sodium (pre-pressurized) and vacuum (constant pressure) versus the Reference 9 data in 1400°F sodium (constant pressure) are shown. The point of interest is the parallel position of the two constant pressure tests. The apparent decrease of stress-rupture properties in sodium versus vacuum at 1300°F cannot be substantiated at the present time.

FIGURE 36. TYPE 316 SS BIAXIAL STRESS RUPTURE PROPERTIES IN SODIUM

FIGURE 37. TYPE 304 SS BIAXIAL STRESS RUPTURE PROPERTIES IN SODIUM

FIGURE 38. BIAXIAL STRESS RUPTURE PROPERTIES (Type 304 SS in Vacuum versus Sodium)

TABLE 16

BIAXIAL STRESS-RUPTURE DATA AT 1300°F IN VACUUM
FOR 304 SS

<u>Structural</u> <u>Condition</u>	<u>Hoop Stress*, psi () $\Delta D/D, \%$</u>	
	<u>10,000</u>	<u>12,000</u>
Mill Annealed	2533 hours (11)	1002 hours
Carbide Agglomerated	1664 hours (13)	550 hours

*These tests were performed at constant pressure. Therefore,
the hoop stress increases as expansion occurs.

SECTION VI

LIST OF SODIUM MASS
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