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AEC RESEARCH AND
DEVELOPMENT REPORT
JANUARY 1969

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**SODIUM COMPONENTS DEVELOPMENT PROGRAM
MASS TRANSFER INVESTIGATIONS
IN LIQUID METAL SYSTEMS
QUARTERLY PROGRESS REPORT
NO. 7
SEPTEMBER-NOVEMBER 1968**

U.S. ATOMIC ENERGY COMMISSION
CONTRACT AT(04-3)-189
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SODIUM COMPONENTS DEVELOPMENT PROGRAM
MASS TRANSFER INVESTIGATIONS IN LIQUID METAL SYSTEMS

Quarterly Progress Report No. 7

September - November 1968

R. S. Young

R. S. Young, Project Engineer
SMT Program, P. A. 15

E. L. Zebroski

E. L. Zebroski, Manager
Sodium Reactor Technology Section

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SECTION I

INTRODUCTION AND PROGRAM SUMMARY

1.1 GENERAL

Corrosion of candidate structural materials, 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 steel and 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. An excess of 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 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 were established to represent environments of large fast reactors in an improved way.

1.2 SUMMARY

The Sodium Mass Transfer Program is currently organized into five Tasks. 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 mechanistic 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.

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 stress-rupture data 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 SCT1 under Task 4 of the FY-1967 and 1968 programs. Examination of samples from SCT1 is planned during FY-1969.

1.3 REPORTS

Monthly report letters are prepared reporting progress in all tasks. Progress letters No. 1 through No. 106 covering work from July 1, 1959 through October 31, 1968 have been issued.

In addition to the progress letters, 28 topical reports and 6 Quarterly Progress Reports covering test results have been issued (see Section V).

This is the seventh 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 1 - 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 - Corrosion Testing

Mockup studies related to the P. A. 15 mass transfer program involve:

- Experimentally determining mass transfer, corrosion, and exposure effects in steel exposed to flowing sodium at bulk temperatures up to 1300°F.
- Developing an understanding of the mechanisms involved in corrosion-mass transport-deposition processes so that extrapolations can be made to other engineering conditions.
- Monitoring for localized attack.
- 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 source of data for the mockup studies. Loop 8 has completed a series of four parametric runs to study the effects on mass transfer rates of sodium velocity, mass flow rate, and downstream. Loop 9 has completed one run to study the range of velocity effect on mass transfer rates. A second run utilizing operating conditions estimated for FFTF closed loop design is being prepared.

2.1.1 Corrosion and Deposition Patterns in Run 9-1

In run 9-1, deposition occurred at the inlet of the first cooler, Zone J, (Figure 1), which was an impingement cartridge.⁽¹⁾ In runs 8-1 and 8-2, corrosion (weight loss) occurred into the first cartridge. The deposition indicates that the sodium was saturated at 1300°F. However, examination of the XRF results (Figure 2) show that the sodium is apparently saturated in chromium only, and that nickel and manganese are being selectively removed from steel surfaces. This agrees with the results of runs 8-1 and 8-2. The effect is two-fold: the decreasing temperature supplies the activation energy for Cr precipitation; and the flow disturbances (turbulence), produced by the impingement samples, increases the amount of material precipitated. This increased deposition of Cr then reverses the slight amount of total corrosion (weight loss) seen in runs 8-1 and 8-2 to a weight gain. The deposition pattern through the remainder of cooler J is comparable with that in runs 8-1 and 8-2 until the outlet (impingement) cartridge. The amount of deposition is substantially increased in the outlet impingement cartridge (Figure 2). This effect can again be attributed to the change in the flow characteristics. The XRF results show the increase in deposition to be predominantly Cr. This is expected since the Zone J cooler

is a known Cr deposition area.⁽²⁾ It was noted in runs 8-1 and 8-2 that the Ni corrosion in the Zone J cooler increased when Cr precipitated. In Figure 2, it can be seen that the same effect occurs at the entrance to the cooler. Additionally, a reversal from slight deposition to substantial corrosion (removal) of Ni occurs in the outlet impingement cartridge of the Zone J cooler when the Cr deposition rises substantially. Again, the apparent change (decrease) in the activity of Ni in sodium occurs when Cr precipitates from solution.

The region for maximum deposition and Ni precipitation monitored in loop 8 was the Zone M cooler. This was again true for run 9-1. However, the deposition pattern through the cooler was changed. The inlet and outlet cartridges (impingement), primarily the inlet cartridge, exhibited very large amounts of deposits (Figure 2). The intermediate cartridges had less deposits than those in runs 8-1 and 8-2 (Figure 3). Apparently the excess material removed in the inlet impingement cartridge, due to the changed flow characteristics, lowered the amount available for precipitation because of decreasing temperature in the following cartridges. The XRF data show that Ni is again the major element precipitating in the Zone M cooler. The drop of deposition in the intermediate cartridges is primarily a result of Cr and Mn decreases. The Ni deposition is relatively stable.

The effect of the impingement cartridges, primarily Zone M, on the isothermal cold-leg Zone N is to shift the Cr, Ni, and Mn from deposition (runs 8-1 and 8-2), to corrosion (Figure 2). However, the amount of total deposition is comparable to that of run 8-2 (Figure 3), which had similar velocity conditions. Therefore, the deposit in the isothermal cold-leg of run 9-1 is predominantly Fe or Fe compounds.

The effect of velocity on deposition is shown in Zone B (Figures 2 and 3). Deposition was promoted by a substantial increase in velocity, where under similar conditions of downstream position and temperature (run 8-2) deposition was minor. However, the Ni, Cr, and Mn deposition was quite similar to that of runs 8-1 and 8-2.

The major effect of the impingement cartridges (samples) can be concluded to be one of increased local deposition. This increased deposition then changed the deposition characteristics in immediately adjacent areas (intermediate cartridges of Zone M) and the deposition patterns of the major elements in the isothermal cold-leg region immediately following. In other words, the occurrence of a Fe-rich deposit has been promoted to a higher temperature region by the increased removal of Cr, Ni, and Mn in the J and M coolers. However, the effect on the downstream deposition patterns (total weight of deposit) is minimal, if any, indicating more Fe in solution than in runs 8-1 and 8-2. If this is true, a higher hot-leg corrosion rate is indicated, which in fact is the case (Figure 3). However, the increase in corrosion rate is minimal.

Fluorescence data from the crevice or blind surface (Figure 4) shows a significant amount of Cr, Ni, and Mn movement throughout the system. The hot-leg changes are similar to those for run 8-2,⁽²⁾ which had similar flow and velocity conditions. Based on the previous data concerning higher corrosion rates of ring samples, one would expect perturbation of the crevice surface of the ring samples to be greater than for the

FIGURE 1. SODIUM MASS TRANSFER LMFBR CORROSION MOCKUP LOOP 9

FIGURE 2. WEIGHT CHANGE AND EXPOSED SURFACE X-RAY FLUORESCENCE DATA VERSUS LOOP POSITION FOR RUN 9-1 AFTER 3000 HOURS EXPOSURE

FIGURE 3. COMPARISON OF THE WEIGHT CHANGE DATA VERSUS LOOP POSITION FOR RUNS 8-1, 8-2, AND 9-1

FIGURE 4. CREVICE SURFACE X-RAY FLUORESCENCE DATA VERSUS LOOP POSITION FOR RUN 9-1 AFTER 3000 HOURS EXPOSURE

tube samples, which is not the case. The cold-leg changes are much more severe than for run 8-2⁽²⁾. The crevice effects for run 9-1 agree with the flowing-surface changes for run 9-1, which, as stated previously, were apparently a result of the upstream depletion of Cr, Ni, and Mn in the sodium. There does not appear to be a continuous velocity pattern or dependence for the crevice surface changes as was the case for runs 8-1 and 8-2.

The effect of increased corrosion rate in the isothermal hot-leg, primarily the first three cartridges in Zone F, on the surface microstructure can be seen in Figure 5. A uniform ferritic surface layer decreasing in thickness with downstream position can be seen for cartridges FA, FB, and FC. The formation of a ferritic surface layer versus a ferritic phase in the grain boundaries and their relationship to the corrosion rate was discussed previously.⁽²⁾ A ferritic surface layer will form at higher corrosion rates, which substantiates the previous observation based on weight change.

The presence of sigma phase in Zones F and J is again evident (Figure 5), indicating the decarburization of the 1300°F, isothermal hot-leg.

The various deposition characteristics through the coolers and isothermal cold-leg sections of the system can be seen in Figures 5 and 6. The deposit appearance changes from Zone J (high Cr) to Zone M (high Ni) to Zones N, R, and B (high Fe) (Figure 5). The metallographically retained thickness of the deposit is relatively constant, although the amounts (from weight change data) vary from 400 mg/dm² in cartridge JE to 2000 mg/dm² in cartridge MA. This is a result of the normal wet polishing techniques used in metallographic preparation. With the use of completely dry polishing techniques, the deposits can be retained in their appropriate thicknesses. The cooler deposits can be better seen in Figure 6 where the impingement samples are shown.

There is further evidence for the decarburization-carburization patterns of the monometallic (austenitic) system. The lack of decoration of slip lines on the surfaces of the Zone J impingement samples that show cold work effects (Figure 6) indicates decarburization. Conversely the heavy decoration of slip lines of the Zone M impingement samples indicates carburization. Further cold-leg carburization indications are the slip marking decorations of the Zone N (ring samples) and B samples (Figure 5). Also as mentioned previously, iron carbide was identified in the deposits removed from the Zone B samples.

XRD analysis was the primary tool used in characterizing the material removed from the impingement cartridges. Sufficient material for analysis was obtained only from cartridges JE and MA, Table 1. The removed material was sealed in quartz capsules for the examination.

In cartridge MA, the predominant pattern observed was of an austenitic (fcc) steel. As shown in Table 2, the major elements present was Ni, Cr, Mn, and Fe. Minor amounts of free carbon, iron carbide, and a material isostructural to sodium nickelate were also identified. A trace of some unidentified substance as well as a halo in the pattern (denoting some amorphous substance) were also noted. A second portion of the

(REDUCED TO 85% FOR REPRODUCTION PURPOSES)

FIGURE 5. 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 6. SURFACE MICROSTRUCTURES AND DEPOSIT CHARACTERISTICS OF THE IMPINGEMENT SAMPLES FROM RUN 9-1

sample, which had been exposed to air, was also examined. This time, in addition to the austenite, minor carbide phases of the forms $M_{23}C_6$ and M_7C_3 were seen. Traces of α Fe and α Mn were also identified. The carbon, Fe_3C , and $NaNiO_2$ were not detected and, although an unidentified trace constituent was present, it was not the same as the one present in the first sample analyzed. Some of the differences between samples may be attributable to the effects of air exposure, but it seems more likely that a large portion of the difference is due to sample inhomogeneity.

Less work was performed on cartridge JE because of the smaller quantities and greater adherency of the material. Some deposit was removed by scraping a specimen with a glass slide in air. The major species present was designated as a carbide of the $M_{23}C_6$ type. The carbide phase had a large face-centered cubic structure with a calculated lattice parameter of $10.625\text{\AA} \pm 0.005$. Minor phases of σ FeCr and Cr were also detected.

Two observations of interest should be noted from this work: (a) the concentration of carbon in the deposits up to 2% from an initial level of $\sim 0.05\%$ in the steel, and its presence in the form of complex metal carbides; (b) the degree of tenacity between deposits in different areas of the system (JE and MA). Both of these observations have been noted in different ways and discussed previously.⁽¹⁾ The carburization of MA samples and the metallographic appearance of the various deposits are examples.

TABLE 1

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

Test Cartridge

<u>MA</u>	<u>JE</u>
γ Fe (major)	
C	$M_{23}C_6$ (major)
Fe_3C	
$M_{23}C_6$ (minor)	σ FeCr
M_7C_3	Cr (minor)
α Fe	
α Mn	
Unidentified (trace)	
amorphous substance	

TABLE 2

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

<u>Metal (%)</u>	<u>Test Cartridge</u>			
	<u>JA</u>	<u>JE</u>	<u>MA</u>	<u>ME</u>
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

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

Concerning the reporting of the compound similar in structure to sodium nickelate (Table 1), it is unlikely that this compound is present. Additional work with the deposits showed that the structure did not change with exposure to air. If sodium nickelate were present, it would be converted to the carbonate as a result of exposure.

The compositional comparison of the impingement cartridge deposits obtained by emission spectrography and XRF of the removed deposits (Table 2) and XRF of the samples are shown in Table 3.

TABLE 3

DEPOSITS AND SAMPLE SURFACE COMPOSITION

<u>Metal (%)</u>	<u>Test Cartridge</u>			
	<u>JA</u>	<u>JE</u>	<u>MA</u>	<u>ME</u>
Cr	20* 20**	(50) 43	35 23	15 24
Ni	10 10	(10) 10	35 21	25 21
Mn	10 2	(trace)3	20 4	25 6
Mo	2 3	--- 3	0.5 3	0.5 3
Fe	60 65	(40) 41	10 49	25 46

* Removed deposit - emission spectrography

** Deposit on samples - XRF

() XRF

The agreement between the two sets of data is generally very good. A major problem area appears to be in obtaining representative Mn results with emission spectrography, cartridges JA, MA, and ME. The other area of major disagreement is in cartridge MA where the Cr and Ni in the deposit retained on the sample are low compared to the analysis of the removed deposit. As noted previously, the deposits in cartridge MA can be removed easily as compared to those in cartridge JE. The possibility exists that much of the cartridge MA sample deposit was lost during preparation for XRF, much as it is lost during metallographic preparation, leaving an Fe-rich metal surface for the XRF analysis. However, both sets of data reaffirm the observation: Of the major alloying elements, Cr deposits first, followed by Ni, and then Mn, with Fe supplying the remainder to ~ 100% as the sodium temperature is decreased.

Recently, the removed deposits from cartridge MA have been reanalyzed, in conjunction with deposits removed from cartridge MA in run 8-3, using wet chemical techniques (colorimetric and flame photometry). The run 9-1 results are compared below with the previous two sets of data:

	<u>Elemental Percent</u>						
	<u>Cr</u>	<u>Ni</u>	<u>Mn</u>	<u>Mo</u>	<u>Fe</u>	<u>Na</u>	<u>C</u>
Emission spectrography	35	35	20	0.5	10	2	--
XRF	23	21	4	3	49	--	--
Wet Chemistry	35	25	7	--	6	10	2

2.1.2 Test Results and Evaluation of Run 8-3

The test facility used was prototype mockup loop 8 which has been described previously.⁽²⁾ A special corrosion cartridge (Figure 7) was used in Run 8-3 to determine the effect of mass flow on corrosion rate.

2.1.2.1 Loop Operating Conditions

The average operating conditions for Run 8-3 were 3000 hours, 1300°F T_{max} , 800°F T_{min} , and oxygen content < 10 ppm based on plugging temperatures of 270-280°F. The sodium velocities in the hot leg were 6 and 28 fps, whereas in the cold leg and coolers it varied from 1.3 fps to 15.3 fps. Figure 8 shows the velocity and temperature distribution in Run 8-3.

The loop was cold trapped during the run at 270°F to 280°F and monitored with a plugging indicator. It is difficult to assign an exact value for oxygen level in sodium, based solely on plugging temperatures⁽³⁾; however, it is estimated that the oxygen level during the run was between 5 ppm and 10 ppm.

2.1.2.2 Test Results

The materials tested were Type-316 SS in Zones C, E, F, J, and M and Type-304 in Zones G, H, N, R, and B. After exposure, the samples were removed under argon

cover, the excess sodium was cleaned with alcohol and water, and weighed to determine the weight change.

The corrosion and deposition behavior for Run 8-3 is shown in Table 4 and Figure 9.

2.1.2.3 Discussion

The separation of the various parameters is complicated by the presence of "crevice side" corrosion discussed previously⁽²⁾. A special corrosion cartridge was placed in the hot leg (Figure 7) to determine the effect of mass flow. The standard corrosion cartridge consists of rings and tubes. The tube outside diameter is flush against the holder, and the annular ring's inside diameter, flush against a concentric mandrel running through the center. It is assumed that the outside diameter of the tube and inside diameter of the ring (crevice surfaces) do not see flowing Na; hence, the corrosion rate per unit area is determined by the inside diameter surface of the tube and the outside diameter surface of the ring. The special cartridge in Run 8-3 had another specimen placed between the outer and inner specimens, which was in contact with flowing sodium on both inside diameter and outside diameter surfaces (Figure 10). The sodium velocity was designed to be the same in both (A and B) annular sections. However, the flow in Section A was 4.4 gpm and Section B was 1.3 gpm. The corrosion rate (CR, mg/dm² - month) for the standard i. d. surface sample (tube) was about three times less than for the o. d. surface sample (ring). The samples placed between the tube and the ring saw 1.3 gpm Na flow on o. d. and 4.4 gpm Na flow on i. d.; so one should expect that the CR, mg/dm² - month, for those samples to be somewhere between the rings and the tubes. However, it was found to be lower than both of them. The conclusion derived from this observation is that in the tube and the ring samples, the crevice side corroded to some extent, although it was not taken into consideration in the calculation of rate per unit area. The corrosion rate on the crevice sides will depend on the velocity. At very low velocities, the corrosion product will tend to remain stagnant; whereas, at higher velocities, it may continue to corrode at a finite rate.

If it is assumed that the corrosion data for the specimens (FB51_s) which saw flowing sodium on both sides to be correct, it can be seen that the FB-51 tube data should be above the FB51_s data, in other words, less than 35 mg/dm² (Figure 10). Since we have not included the crevice side corrosion, the corrosion rate for FB51 is ~ 57 mg/dm² - month.

The results of the special corrosion cartridge (FB) indicate that the corrosion rates, which are being used to determine the coefficients in the equation, may vary as much as a factor of two, and will not remain constant since the crevice side corrosion will depend on the velocity, actual construction of the samples, etc. This is confirmed by the x-ray fluorescence analysis of the crevice sides of the hot leg samples. Samples from runs 8-2 and 9-1 show a considerable amount of depletion of Cr, Ni, Mn in the F section. In run 8-1, this was not true. This can be explained by runs 8-2 and 9-1 having a higher flow rate and the samples in general saw higher velocities than run 8-1.

FIGURE 7. VARIATION OF CORROSION CARTRIDGES IN RUN 8-3

FIGURE 8. TEMPERATURE AND VELOCITY DISTRIBUTION FOR RUN 8-3

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TABLE 4

WEIGHT CHANGE, LOOP POSITION AND VELOCITY OF RUN 8-3

<u>TIME (HOURS)</u>	<u>TEST ZONE</u>	<u>WEIGHT CHANGE (mg/dm²)</u>		<u>VELOCITY (fps)</u>	<u>DOWNSTREAM (L/De)</u>
		<u>TUBES</u>	<u>RINGS</u>		
1093	FA	-206	-166	28.0	175.3
		-180	-150		
		-174	-147		
		-174	-143		
		-147	-128		
		-166	-123		
		-148	-121		
		-156	-119		
	FC	-152	-109	28.0	362.3
		-150	-105		
		-140	-109		
		-137	-101		
		-115	-95		
		-128	-89		
		-89	-59		
		-70	-58		
	FE	-67	-57	6.0	605.6
		--	-54		
		-61	-63		
		-62	-59		
		-59	-67		
		-23	--		
		-26	--		
		-22	--		
	JA	-21	--	3.60	2.39
		-18	--		
		-14	--		
		-10	--		
		66	--		
		47	--		
		40	--		
		40	--		
	ME	37	--	3.60	230.9
		36	--		
		35	--		
		-366	-305		
		-341	-277		
		-306	-255		
		-328	-253		
		-307	-241		
1920	FA	-297	-216	28.0	175.3
		-279	-218		
					284.4

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<u>TIME (HOURS)</u>	<u>TEST ZONE</u>	<u>WEIGHT CHANGE (mg/dm²)</u>		<u>VELOCITY (fps)</u>	<u>DOWNSTREAM (L/De)</u>
		<u>TUBES</u>	<u>RINGS</u>		
(cont)					
1920	FC	-246	-189	28.0	362.3
		-241	-188		
		-245	-180		
		-218	-174		
		-230	-166	28.0	362.3
		-232	-164		
		-214	-161		471.4
	FE	-120	-100	6.0	605.6
		-107	-96		
		-102	-95		
		-96	-84		
		-99	-85		
		-91	-92		
		-92	-107		625.2
	JA	-34	--	3.60	2.39
		-27	--		
		-28	--		
		-26	--		
		-29	--		
		-19	--		
		-15	--		9.84
	ME	115	--	3.60	230.9
		80	--		
		77	--		
		77	--		
		90	--		
		95	--		
		93	--		238.3
3010	FB ₅₁	-241	--	6.20	288.9
		-223	--		
		-198	--		
		-187	--		
		-174	--		
		-157	--		397.9
		-145	--		
	FB ₀₁	--	-680	6.20	288.9
		--	-511		
		--	-627		
		--	-498		
		--	-565		
		--	-614		
		--	-600		311.6

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<u>WEIGHT CHANGE (mg/dm²)</u>					
<u>TIME (HOURS)</u>	<u>TEST ZONE</u>	<u>TUBES</u>	<u>RINGS</u>	<u>VELOCITY (fps)</u>	<u>DOWNSTREAM (L/De)</u>
(cont) 3010					
SPECIAL	FB51s	-157	--	6.20	288.9
CARTRIDGE		-140	--		
		-137	--		
		-139	--		
		-130	--		
		-129	--		
		-126	--		326.6
	FD	-294	-228	28.0	492.8
		-277	-205		
		-265	-200		
		-235	-191		
		-225	-179	28.0	492.8
		-233	-191		
		-221	-175		601.9
	FF	-185	-158	28.0	628.9
		-162	-145		
		-177	-135		
		-165	-131		
		-146	-124		
		-133	-103		
		-132	- 99		738.0
	GA	-110	--	2.40	749.9
		-151	--		
		-122	--		
		-114	--		
		-120	--		
		-142	--		
		-127	--		
		-132	--		755.9
	GB	-137	--	2.40	759.5
		-148	--		
		-153	--		
		-123	--		
		-106	--		
		-136	--		
		-104	--		
		-97	--		764.8
	HA	-106	--	1.30	772.0
		-93	--		
		-88	--		
		-83	--		
		-82	--		
		-75	--		
		-87	--		776.5

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<u>WEIGHT CHANGE (mg/dm²)</u>					
<u>TIME (HOURS)</u>	<u>TEST ZONE</u>	<u>TUBES</u>	<u>RINGS</u>	<u>VELOCITY (fps)</u>	<u>DOWNSTREAM (L/De)</u>
(cont) 3010	HB	-103	--	1.30	779.5
		-86	--		
		-79	--		
		-74	--		
		-73	--		
		-70	--		
		-80	--		
					784.0
	JA	(See 1093 & 1920 hrs)			
	JB	-0.61	--	3.60	13.5
		-13.5	--		
		-14	--		
		-12	--		
		-7	--		
		-2	--		
		+8	--		
					20.9
	JC	41	--	3.60	24.6
		26	--		
		32	--		
		34	--		
		38	--	3.60	24.6
		48	--		
		61	--		
					32.0
	JD	97	--	3.60	35.6
		81	--		
		81	--		
		82	--		
		93	--		
		106	--		
		113	--		
					43.1
	JE	123	--	3.60	49.1
		131	--		
		116	--		
		112	--		
		118	--		
		122	--		
		123	--		
					56.5
	MA	191	--	3.60	186.5
		228	--		
		152	--		
		166	--		
		161	--		

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<u>WEIGHT CHANGE (mg/dm²)</u>					
<u>TIME (HOURS)</u>	<u>TEST ZONE</u>	<u>TUBES</u>	<u>RINGS</u>	<u>VELOCITY (fps)</u>	<u>DOWNSTREAM (L/De)</u>
(cont)3010	RA	15	--	1.30	601.0
		14	--		
		11	--		
	RB	15	--	1.30	605.5
		13	--		607.9
		11	--		
		11	--		
		8	--		
		5	--		
		8	--		
		10	--		612.4
	RC	13	--	1.30	614.7
		12	--		
		11	--		
		15	--		
		14	--		
		8	--		
		10	--		619.2
	BA	20	--	1.30	741.5
		17	--		
		12	--		
		14	--		
		14	--		
		16	--		
		16	--		746.0
	MA	145	--	3.60	186.5
		140	--		194.0
	MB	388	--	3.60	197.6
		217	--		
		174	--		
		152	--		
		140	--		
		140	--		
		139	--		205.1
3010	MC	298	--	3.60	208.7
		167	--		
		134	--		
		113	--		
		106	--		
		103	--		
		117	--		216.1

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WEIGHT CHANGE (mg/dm ²)						
TIME (HOURS)	TEST ZONE	TUBES	RINGS	VELOCITY (fps)	DOWNSTREAM (L/De)	
(cont) 3010	MD	257	--	3.60	219.8	
		146	--			
		112	--			
		102	--			
		98	--			
		93	--			
		91	--			
	ME	(see 1093 & 1920 hrs)				227.2
	NA	456	383	15.3	247.3	
		434	384			
		289	244			
		261	198			
		214	174			
		188	151			
		154	128			
	NB	47	43	4.60	271.1	
		43	39			
		42	41			
		41	39			
		39	38			
		38	39			
		43	39			
	NC	90	84	15.3	420.9	
		80	80			
		68	74			
		73	75			
		66	66			
		65	65			
		60	66			
	RA	21	--	1.30	522.6	
		14	--			
		16	--			
						601.0

FIGURE 9. CORROSION AND DEPOSITION BEHAVIOR IN RUN 8-3

The corrosion data from the special cartridge suggests that at a constant velocity, an increase in mass flow increases the corrosion rate. In Figure 10, the ring samples in Section A encountered about three times higher flow rate (4.4 gpm) than tube samples in Section B (1.3 gpm). The corrosion of the ring samples was about three times higher than tube samples. However, the conclusion of increased corrosion rate because of increased mass flow, per se, may be erroneous. According to the UK⁽⁴⁾ observations, the corrosion rate becomes independent of velocity or mass flow at Reynolds numbers greater than 60,000 for a particular bore size. In the special cartridge the Reynolds numbers for Sections A and B were approximately 10,000 and 50,000, respectively. In this region of turbulent flow, the present observation is in qualitative agreement with the UK⁽⁴⁾ results.

The x-ray fluorescence analysis of the change in the surface composition is shown in Figure 11. The figure shows a similar trend as in runs 8-1, 8-2, and 9-1 of selective dissolution and deposition.

Metallographic observations of hot leg and cold leg specimens from run 8-3 indicates that hot leg samples are decarburized, whereas cold leg samples are carburized. Formation of sigma phase in hot leg specimens and existence of carbides in cold leg specimens in run 8-1 also indicates that carbon is transferred from hot to cold zones. Analysis of the bulk carbon concentration of run 8-3 samples (Figure 12) verifies the previous observations.

2.1.2.4 Summary and Conclusions

As in the case of previous runs, the general trend of decrease in corrosion rate of Type-316 austenitic stainless steel at 1300°F with increase in downstream position has been observed.

Considerable corrosion has been observed on the crevice side of the specimens which results in some uncertainties in the corrosion data. Because of the crevice side corrosion it becomes more difficult to represent the corrosion data by an empirical equation.

Metallographic and chemical analysis of the hot and cold leg samples show a decarburization of the hot leg and carburization of the cold leg.

A definite pattern of Cr and carbon dissolution and deposition has been observed indicating that carbon transport depends upon the movement of Cr in the system.

2.2 Subtask 1.2 Rational Corrosion Models — Mechanistic Studies

2.2.1 Effect of Downstream on the Corrosion of Austenitic Stainless Steels

It has been observed that the corrosion rate of stainless steel in an isothermal region decreases with the increase of downstream position. Sodium entering the hot leg has the highest corrosion potential, hence the corrosion rate at the entrance of the hot leg is the highest. As sodium becomes saturated with solute elements, the activities of

FIGURE 10. CORROSION BEHAVIOR IN THE SPECIAL CARTRIDGE FROM RUN 8-3

FIGURE 11 X-RAY FLUORESCENCE DATA ON RUN 8-3 SAMPLES

FIGURE 12. CARBON TRANSPORT IN RUN 8-3

the solute elements increases, which in turn decreases the corrosion potential,

$$\Delta (a_i^{\text{Na}} - a_i^{\text{Solid}}),$$

where: a_i^{Na} = activity of i^{th} component in the Na

and: a_i^{Solid} = activity of i^{th} component in solid.

The decrease in corrosion potential decreases the corrosion rate of the samples in downstream positions. The effect of downstream is a complex function of velocity, geometry, etc. The decrease of corrosion rate versus downstream ($\propto \frac{L}{D}$, where; L = length and D = hydraulic diameter) at various velocities is shown in Figure 13. The figure indicates that an increase of velocity increases the downstream effect (slope of $\frac{\delta \Delta W}{\delta t}$ vs L/D plot). At higher velocities, 16 and 28 fps, where the reaction at the surface is the rate controlling step in the corrosion process, the corrosion rate decreases from 0.75 mils/year to 0.3 mils/year over L/D = 600. At lower velocities, 3 and 6 fps, where diffusion of solutes across the liquid boundary layer is the rate controlling step in the corrosion process, the rate decreases from 0.3 mil/year to 0.2 mil/year over the same downstream.

2.2.2 Effect of Composition on the Corrosion of Austenitic Stainless Steels

For comparative purposes, the corrosion of Types 304, 316, 347 SS and incoloy-800 was studied. The alloys were exposed together in the same test facility. The samples were placed in parallel flow channels, avoiding the effect of downstream. The results of the corrosion tests at 1300°F, $O_2 < 10\text{ppm}$, are shown in Figure 14. The data show that the corrosion rate for Incoloy-800 is about 3 to 4 times higher than for Type-316 SS under identical conditions of loop operation. The present observation is in qualitative agreement with the UK⁽⁵⁾ and BNL⁽⁶⁾ data.

The effect of composition on corrosion rate depends on sodium velocity, sodium temperature, and the oxygen concentration of the sodium. The major constituents of austenitic stainless steels are iron, chromium, and nickel. Therefore, the relative effects of oxygen content, velocity, and temperature on the three principle elements will determine the corrosion rate of the various alloys. At higher velocities (> 15 fps), where the reaction rate constant at the surface is believed to be rate controlling, the reaction rate constants govern the total corrosion.

It has been observed⁽⁵⁾ that the corrosion rate of the high Ni base alloy Nimonic 80A (75% Ni) does not depend on the oxygen level in the sodium, between < 10 to 30 ppm O_2 . From this observation, one can conclude that for these oxygen levels, the dissolution process of Ni does not involve oxygen transfer. This is because no stable nickel-sodium complex oxides can exist at these temperatures and oxygen levels.

It has been established that the corrosion of austenitic stainless steel depends strongly on the oxygen level in sodium. This observation implies that the dissolution process for either Fe or Cr involves oxygen transfer. Both Fe and Cr form stable

FIGURE 13. THE EFFECT OF DOWNSTREAM ON THE CORROSION RATE AS A FUNCTION OF VELOCITY IN 1300°F SODIUM CONTAINING < 10 ppm O₂

FIGURE 14. CORROSION RATES OF MILL ANNEALED MATERIALS IN 1300°F SODIUM

complex oxides with sodium, e.g., $\text{Fe}(\text{Na}_2\text{O}_2)$ and NaCrO_2 . However, the thermodynamic stabilities of these complex oxides indicates that at 1200°F , $\text{Fe}(\text{Na}_2\text{O}_2)$ can only exist in sodium at an oxygen level greater than 1600 ppm⁽⁷⁾, whereas, the stability of NaCrO_2 ⁽⁸⁾ indicates that it can exist in sodium containing 2 to 5 ppm oxygen at 1300°F . From the above thermodynamic considerations, one can conclude that the effect of oxygen on the corrosion of Cr will be much stronger than that of Fe. Therefore, the major reason for the increased corrosion of austenitic stainless steels with increasing oxygen, between 5 to 30 ppm, is probably caused by the increased corrosion rate for Cr. At a constant velocity, and a low oxygen level, it is feasible for the reaction rate constant for Ni to be higher than that of Cr. As a consequence, a high Ni alloy can corrode at a higher rate. If the oxygen level is increased, the reaction rate constant for Cr is increased, whereas it remains the same for Ni. It is very likely that the corrosion rate of the 300-series stainless steels will supersede the higher nickel alloys at higher oxygen levels. Figure 15 illustrates the above possibility:

FIGURE 15. COMPARISON OF REACTOR RATES OF CHROMIUM AND NICKEL WITH INCREASING OXYGEN

It should be pointed out that the relative rates will be strongly dependent on sodium velocity and temperature. For example, the activation energy for atomic dissolution, e.g., Ni, is expected to be considerably lower than the activation energy for Cr dissolution which involves oxygen transfer. Therefore, increasing temperature will affect the Cr corrosion to a greater degree than the nickel corrosion.

2.2.3 Carbon Transport in Bimetallic Loops

The possibility of the carburization of the Type-316 stainless steel IHX and decarburization of the 2-1/4 Cr-1 Mo superheater tubes in a bimetallic secondary

system has been observed from the thermodynamic prediction of carbon activity in the solid state. Better control of carbon transfer can be obtained by noting the type of carbides precipitating in the IHX and superheater tubes for long times at operating temperatures and subsequently altering the as-received microstructure of the normalized 2-1/4 Cr-1 Mo by heat treatments. Typical material specifications are:

- Solution annealed Type 316 stainless steel operating at approximately 1000°F for the IHX; and
- Normalized 2-1/4 Cr - 1 Mo steel operating at approximately 950°F for the superheater tubes.

The as-received structures are austenite and bainite, respectively.⁽⁹⁾

The time-temperature sensitization diagram for Type 316-L stainless steel shows that the precipitation of $M_{23}C_6$ begins in 35 hours at 1000°F.⁽¹⁰⁾ The $M_{23}C_6$ carbide is a chromium-rich carbide with a complex cubic (defect) structure, in which M denotes a mixture of iron, chromium, nickel, and manganese atoms. The time of incipient precipitation for Type-316 stainless steel should be shorter because of a higher carbon content than the Type-316-L SS. X-ray identification of phases in Type-316 stainless steel subjected to stress to cause creep rupture in short time periods, suggests that stress accelerates the precipitation of $M_{23}C_6$ and sigma.⁽¹¹⁾ Therefore, a low carbon activity is established rapidly at operating conditions in the IHX.

The bainitic structure of 2-1/4 Cr - 1Mo will temper to a mixture of alloy carbides. The effect of time and temperature on the type of alloy carbide is illustrated in Figure 16.⁽⁹⁾ The activity of carbon in 2-1/4Cr - 1Mo will decrease as the metastable carbide, cementite (M_3C), redissolves to form Mo_2C , or transform "in situ" to M_7C_3 . The M_7C_3 is a chromium rich carbide with a hexagonal structure, in which M denotes a mixture of iron and chromium atoms. The carbon activity of 2-1/4Cr - 1Mo will be substantially lower after cementite redissolves, and the stable alloy carbides, M_7C_3 , Mo_2C , $M_{23}C_6$, and M_6C precipitate. Extrapolation of the curves in Figure 15 suggests that the above processes occur at times much greater than those for precipitation of $M_{23}C_6$ in the Type-316 stainless steel IHX. Therefore, a large activity gradient will exist for a long time, which will cause decarburization of the superheater tubes and carburization of the IHX when the 2-1/4Cr - 1Mo is used in the normalized condition.

A modified ferritic 2-1/4Cr - 1Mo steel, doped with either Ti or Nb, has been found to stop carbon transport and not jeopardize mechanical properties⁽¹²⁾. At the present time, this steel is not available for commercial use in the United States. Another approach is to alter the as-received structure of the 2-1/4Cr - 1Mo steel. Isothermal transformation studies of Mo steels, with a Mo to carbon ratio approximately equal to those for low carbon 2-1/4Cr - 1Mo steels, show that alloy carbides, $M_{23}C_6$ and Mo_2C , precipitate in 24 hours at 700°C⁽¹³⁾. The $M_{23}C_6$ carbide in a ferritic steel will differ in chromium content from that of the $M_{23}C_6$ carbide in Type-316 stainless steel. Therefore, the carbon activity of the stainless steel will be lower; however, the difference will not be as great as when cementite is present in the ferritic steel. This

FIGURE 16. ISOTHERMAL DIAGRAM SHOWING THE SEQUENCE OF CARBIDE FORMATION ON TEMPERING OF NORMALIZED 2-1/4 Cr, 1 Mo STEEL

suggests that the embrittlement of the IHX can be reduced by installing 2-1/4Cr - 1Mo superheater tubes with a heat-treated structure, containing alloy carbides, as apposed to a baintic structure containing carbide and cementite. However, such a treatment may be a compromise between reducing the embrittlement of the IHX and maintaining the creep properties of the superheater tubes. The heat-treatment is likely to agglomerate the alloy carbides, resulting in an increase in the resistance to decarburi- zation at an expense of creep strength. To achieve both creep strength and carbide stability in a bimetallic loop, a 2-1/4Cr - 1 Mo steel doped with Ti or Nb is needed. The carbon activities for 2-1/4Cr - 1Mo in the normalized and heat-treated conditions and Type-316 stainless steel as a function of time at 1000⁰F are shown in Figure 17.

2.2.4 Mechanism For Carbon Transport

Components from Loop 5 of the Phase I SMT program are being examined to determine the extent of carbon movement in a monometallic ferritic system. The knowledge of the carbon gradients through the walls of the low alloy (2-1/4Cr - 1Mo) components, rather than the bulk carbon contents, should provide more insight into the mechanisms of carbon transport.

The Na-exposed surfaces, shown in Figure 18, exhibit 8-10 mils of grain growth in the heater shells, with no grain growth in the coolers. This might be from surface cold work due to the different methods of producing the large diameter (6 inch) heater shell versus the small diameter (1 inch) cooler. The bulk carbon contents, shown in Table 5, do not correlate very well with the metallography of the i. d. surfaces. Therefore, carbon analysis of layers removed from these surfaces will be made.

TABLE 5

CARBON ANALYSIS OF LOOP 5 COMPONENTS

Component	Measured Carbon Level (%C)	
	<u>Inlet</u>	<u>Outlet</u>
3rd Heater Shell	0.106	0.031
Sampling Port	0.086	0.106
1st Cooler	0.087	0.098
2nd Heater Shell	0.109	0.115
1st Heater Shell	0.110	0.119
2nd Cooler	0.130	0.126

FIGURE 17. RELATIVE CARBON ACTIVITIES AS A FUNCTION OF TIME AT 1000°F

ALL SAMPLES HEATED AT 1650°F FOR 35 MINUTES, COOLED TO 1100°F IN ONE HOUR AND AIR-COOLED

100X

FIGURE 18. INNER SURFACES OF LOOP 5 COMPONENTS WITH CORRESPONDING BULK CARBON CONTENT

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 concentration, 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 which 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 Carbon Distribution

Experiments have been underway to determine whether the flow-through system in a pipe provides a representative sample for the determination of carbon in sodium. Sodium was cored from the center two-thirds of a section of the pipe to provide one sample for analysis. Most of the remaining sodium was scraped from the pipe to provide a second analytical sample. Three such pairs of samples were taken along a single sampler. Results are shown in Table 6.

The results indicate that the total carbon concentration in the core samples is slightly lower than the concentration in the sodium closer to the walls.

Our normal analytical sample is the sum of these two fractions, thus minimizing the potential sampling error. However, since the scraping procedure does not completely strip the sodium from the pipe, the possibility remains that some carbon escapes detection if it deposits directly on the walls. Experiments are underway to test this point. They consist of washing all the sodium from the tube surfaces and analyzing the resulting solution for carbon.

It is clear, however, even from the limited data obtained to date, that extrusion techniques which are commonly used for sample removal will lead to low results unless a large fraction of the sodium can be removed.

3.1.2 Hot Leg Sampling

The last quarterly⁽¹⁾ reported the first data obtained for the analysis of sodium sampled from the hot leg of Loop 9. Two more pairs of hot and cold samples have been analyzed for total alkalinity and total hydrogen. Carbon analyses have been obtained for three of the samples to date. All the available data are summarized in Table 7.

TABLE 6

ANALYSIS FOR CARBON IN SODIUM FROM THREE
SECTIONS OF FLOW-THROUGH SAMPLE 5R-3-6A
 (Carbon - ppm)

<u>Section</u>	<u>CORE</u>			<u>WALL</u>		
	<u>Water-Acid</u>	<u>Van Slyke</u>	<u>Total</u>	<u>Water-Acid</u>	<u>Van Slyke</u>	<u>Total</u>
1	2.9	8.9	11.8	4.2	23.0	27.2
2	2.3	15.7	18.0	5.1	18.5	23.6
3	3.0	18.1	21.0	6.0	21.0	27.0

TABLE 7

COMPARISON OF ANALYSES BETWEEN
HOT AND COLD LEG SAMPLES

<u>Sample Number</u>	<u>Temperature (°F)</u>	<u>Total Alkalinity (ppm)</u>	<u>Total Hydrogen (ppm)</u>	<u>Total Carbon</u>
9-2-6C	750	12.2, 10.8	2.2	164
9-2-6D	1100	15.5, 15.4	2.3	240
9-2-8C	755	7.8	1.8	---
9-2-8D	1125	8.5	1.8	---
9-2-9B	~ 750	11.2	1.7	---
9-2-9A	~ 1100	13.4	2.4	12

The total alkalinity and hydrogen values are in good agreement within the precision of the methods, confirming the validity of the high temperature sampling. It was expected that the hot leg samples would have less hydrogen, but this does not appear to be the case. However, the precision for the analysis (± 0.5 ppm) may not be sufficient to detect small actual differences.

There are further comments on the sampling for carbon in the following section.

3.1.3 Surveillance Analyses

The fifth quarterly⁽²⁾ discussed the loop surveillance program and presented data for Loop Runs 9-1, 8-2, and 8-3. Additional data obtained in a continuation of the program are provided in Tables 8 and 9. Values obtained for total hydrogen and for alkalinity for Loop Runs 4-1, 5-3, 5-4, 6-2, 8-4, and 9-2 are presented in Table 8; carbon values are reported in Table 9.

No corrections have been made in the alkalinity values for hydride hydrogen. Based upon past experience, however, the alkalinity values are lower by about 1.5 ppm for each ppm total hydrogen observed. Since the analysis for forms of hydrogen can be performed only sporadically because of the length of time involved and because the corrections are imprecise, it seems preferable for consistency to report the alkalinity values without correction.

The hydrogen values, with one exception are in the 1 to 2 ppm range, not far from the limits of detection. One high value was observed, but this was during a period of isothermal operation.

The total alkalinity values range from about 5 to 15 ppm. Some higher values were observed. These were usually associated with early steps in loop operation.

The good agreement for carbon for the three sections of Sample 5R-3-6A and the two sections of Sample 5R-3-3A indicates that segregation along the tube is not serious. The cause of the extremely high values for this element in Samples 9-2-6C and 9-2-6D is not known, nor can the discrepancy among the three analyses for 9-2-6C be explained.

3.1.4 Summary of Plugging Indicator Investigation on Loops 8 and 9

John C. Biery and Charles C. McPheeters of Los Alamos Scientific Laboratory visited General Electric November 19th and 20th for the purpose of applying techniques of operating plugging indicators partially plugged, in use at LASL, to the mass transfer equipment. The following summary of results is transcribed from notes by these investigators.

Plugging meter runs were made on mass transfer loops 8 and 9 on November 19 and 20, 1968. Loop 8 indicated only one precipitable impurity and the behavior of the impurity was studied in some detail. Loop 9 probably contained more than one impurity and the one which was studied exhibited equilibrium temperatures much above the cold

TABLE 8
SURVEILLANCE DATA: TOTAL ALKALINITY

Sample Number	Date Sampled	Run Test Hours	Sampling Temperature (°F)	Plugging Temperature (°F)	Cold Trap Temperature (°F)	Total Alkalinity (ppm O)	Hydrogen (ppm)
4R-1-1A	07-24-68	530	865	278	Secured	15.0	
5R-3-3A	02-02-68	2058	875	262,274,260	Secured	19.0	1.0, 1.2
5R-3-4A	02-19-68	2466	875	260,242	Secured	14.2	0.7, 1.0
5R-3-6A	03-15-68	3020	885	255	Secured	7.2	1.5
5R-4-2A	09-09-68	70	850	260	280	8.5	
6R-2-1A	05-23-68	0	750	275,280	290	3.9	
8-4-1A	05-24-68	46	785	289	250	30.8, 37.4	1.7
8-4-4A	06-07-68	346	760	260,330	310	19.0	2.9
8-4-5A	06-24-68	754	750	260	280	6.5	1.3
8-4-7A	07-05-68	1300	750	280	300	8.6	
9-2-2A	04-26-68	0	685	243,247	280	9.5	1.6
9-2-3C	05-07-68	Isothermal (750 °F)	659	252,255	280	13.5	7.1
9-2-4C	05-21-68	0	654	255	280	17.0	lost
9-2-6C	06-14-68	70	750,*	250	270	12.2, 10.8	2.2
9-2-6D	06-14-68	70	1100,*	250,240	270	15.5, 15.4	2.3
9-2-8C	07-24-68	723	735,*	255	290	7.8	1.8
9-2-8D	07-24-68	725	1195,*	265	290	8.5	1.8
9-2-9A	08-16-68	1277	1190,*	265	290	13.4	
9-2-9B	08-16-68	1277	750,*	265	290	11.2	

* Samples taken from hot leg and cold leg at the same time

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TABLE 9

CARBON ANALYSES (ppm)

<u>Sample Number</u>	<u>Water-Acid</u>	<u>Van Slyke</u>	<u>Total</u>
5R-3-3A	11.5	27.	38.5
	16.6	27.4	43.
5R-3-4A	3.4	8.0	11.4
5R-3-6A	3.5	16.2	19.7
	3.6	17.0	20.6
	3.1	19.3	22.4
8 -4-1A	12.4	34.2	46.6
8 -4-4A	23.2	66.5	89.7
9 -2-2A	10.4	14.2	24.6
9 -2-6C	22.2	218.	240.
	12.6	134.	147.
	9.2	33.4	42.6
9 -2-6D	4.0	160.	164.
9 -2-9A	7.8	4.7	12.5

trap temperature of the loop. (Cold trap temperature = 280°F , equilibrium temperature varied from 430° to 700°F .)

3.1.4.1 Summary of Loop 8 Plugging Indicator Behavior (Cold Trap Temperature = 320°F)

a. Oscillating Runs (Partially Plugged): Five sets of five minima and maxima were run. The average values of the indicated saturated temperature were as follows: 320°F , 322°F , 320°F , 317°F , 311°F , 309°F . The last two sets were obtained after a set point change which increased the amount of plug and lowered the flow. (The 320°F temperature is equivalent to 3.2 ppm of oxygen from the Rutkauskas curve.)

Note: The reason for the above behavior of changing saturation temperature is not clearly understood. However, the probable reasons may be the trapping of Na_2O in the flow reversal region of the P.I. A rough calculation was made, and the result indicated that there was sufficient area in the cold region of the P.I. to significantly affect the concentration in the 12 gallon system. The above saturation temperature change is equivalent to 0.3 ppm oxygen change (Rutkauskas curve), and the calculations indicated that a change of 1 to 10 ppm might be expected.

Mass transfer coefficients were calculated from portions of the partially plugged runs. The results are as follows:

<u>Velocity (cm/sec)</u>	<u>Coefficients</u> (gm $\text{O}/\text{cm}^2\text{-h-ppm}$)
557	1.04×10^{-2}
690	1.12×10^{-2}
810	1.30×10^{-2}
1000	1.96×10^{-2}

The Rutkauskas solubility curve was used to calculate the ΔC values in the mass transfer calculations. These results are in very good agreement with similar calculations run on partially plugged dynamic data obtained from a plugging meter at LASL. The investigators feel that the impurity being studied was probably Na_2O and was the same impurity observed in the LASL studies.

b. Cooling Rate Effect on Bare Orifice Nucleation: Normal plugging runs were made at varying cooling rates. The saturation temperature was 320°F and the indicated plugging temperatures required to nucleate a detectable quantity of precipitate are indicated below:

<u>Cooling Rate ($^{\circ}\text{F}/\text{min}$)</u>	<u>Plugging Temperature ($^{\circ}\text{F}$)</u>	<u>Temperature Depression ($^{\circ}\text{F}$)</u>
2.29	279	41
4.7	275	45
6.3	265	55
12.0	262	58

c. Effect of temperature depression was studied.

<u>Time to Plug Detection (min)</u>	<u>Constant Orifice Temperature (^oF)</u>	<u>Temperature Depression (^oF)</u>
10.0	283	37
8.25	274	46

3.1.4.2 Summary of Loop 9 Experience

No dynamics were calculated; however, the meter was run overnight in the oscillating mode on the high temperature impurity. The flow change was very erratic and temperature changes were quite variable. The indicated saturation temperature varied from 470^oF down to 430^oF overnight. When the temperature was raised to dissolve the plug off, the plug size increased. The reason was obscure, but may have been because of extensive upstream precipitation in the meter itself.

The investigators feel that the flow rates in both the cold trap and P.I. of loop 9 are much too low.* The low flow rate does not permit the cold trap to control the upper impurity. There is probably a large source of the upper temperature impurity, and this rate is greater than the removal rate of the cold trap.

*Loop 9 is scheduled to be dumped for changing the high heat flux heater. During restarting operations in early December it is planned to re-establish a 0.25 gpm minimum flow through the purification leg vs. ~ 0.1 gpm during this investigation. Experience at this level of flow and at low temperatures during start-up has been the observance of single, low-temperature breaks. Raising the hot leg temperature above 600^oF to 1300^oF will normally result in return of a high temperature break at about 600^oF in the plugging trace.

SECTION IV

TASK 4 - MECHANICAL PROPERTIES LIMITS FOR CLADDING MATERIALS

4.1 Subtask 4.1 Mechanical Property Limits For Cladding Materials-Design Criteria

4.1.1 Stress Rupture Screening Runs in Loop 5R-Additional Evaluation of the Data4.1.1.1 Biaxial Stress-Rupture

In the previously reported⁽¹⁾ summary of the Loop 5R screening runs, biaxial stress-rupture curves were shown and compared to those of other investigators. The use of the term biaxial stress-rupture, to connote constant hoop stress to rupture, may have been misleading based on the specimen design.⁽¹⁴⁾ The use of a filler plug, originally for the purpose of reducing possible unstable rupture conditions, produced a decreasing hoop stress with deformation (creep). The ratio of initial gas volume to specimen volume for specimens is approximately 0.38. A consideration of the variations in pressure and hoop stress as functions of this ratio is shown in Figure 19. A sample expansion of 5% to 10% can result in a 10% to 20% decrease in hoop stress. This variation in hoop stress will alter the creep rates during exposure, and consequently, significantly increase the rupture time. As shown in Figure 19, the hoop stress will remain constant in an expanding tube filled with ideal gas for the case of a hollow tube where the initial specimen volume and initial gas volume are equal. The development of the above statement is shown below:

The longitudinal strain rate⁽¹⁵⁾ and consequently the strain in the longitudinal direction is zero.

Therefore: $2 \pi R t = K$ since the volume of metal remains constant (1)

Also: $PV = K$, at constant temperature (Boyles' Law) or
 $P \pi R^2 L = K$ (2)

For thin walled tubes:

$$\sigma = PR/t \quad (3)$$

In terms of conditions at times 1 and 2, Equations (1), (2), and (3) can be written:

$$R_2 = R_1 t_1/t_2 \quad (1A)$$

$$P_2 = \frac{P_1 R_1^2}{R_2^2} = \frac{P_1 t_2^2}{t_1^2} \quad (2A)$$

$$\sigma_1 = \frac{P_1 R_1}{t_1} = \frac{P_2 R_2}{t_2} \quad (3A)$$

Substituting (1A) and (2A) into the equation for σ_2 :

$$\sigma_2 = \frac{P_1 R_1}{t_1}$$

Therefore:

$$\sigma_2 = \sigma_1 = K$$

Nomenclature:

R =	mean radius
t =	wall thickness
P =	internal pressure
V =	tube volume
l =	tube length
σ =	hoop stress

The biaxial stress-rupture data has been adjusted using Figure 19. The "best-fit" biaxial stress-rupture curves for the four alloys in the annealed condition are shown in Figure 20. Uniaxial stress-rupture tests in air at 1300°F show a similar relative performance of the alloys^(16,17). The biaxial stress-rupture curves for Types-304 and 316 SS in all structural conditions are shown in Figure 21. A comparison of the curves in Figure 21 and the available biaxial stress-rupture data for Types 304 and 316 SS is shown in Figures 22 and 23. The AI tests⁽¹⁸⁾ were performed in static sodium pots with the samples under constant pressure control. The material was seamless in the 10% to 15% cold reduced material. The UK tests⁽¹⁹⁾ were performed in a vacuum furnace with the samples under constant pressure control. The material was a British version of Type-316 SS, vacuum melted, in the 20% cold reduced condition and after an anneal (1922°F for thirty minutes) of the cold reduced material.

The typical failure characteristics and post-exposure microstructures of the four alloys tested are shown in Figures 24 through 27.

4.1.1.2 Unstressed Corrosion Samples

The weight change data and positions of the corrosion samples from Zone F of Loop 5R⁽²⁰⁾ are shown in Table 10. It can be seen that the positioning of the samples results in a reasonably good overlap of downstream positions in Zone F. Also, the "downstream effect", although present, is not pronounced and has considerable scatter. Based on this, the corrosion data can be treated with average values and are shown in Table 11 and Figure 28.

The average weight change data for the Type-316 SS corrosion cartridge samples in Zones E, G, and J, of Loop 5R⁽²⁴⁾ are shown in Table 12 and Figure 29.

The results of x-ray fluorescence (XRF) and electron microprobe analyses of the Zone F corrosion samples from Loop 5R are shown in Figures 30 through 33.

The results of XRF analysis on selected corrosion cartridge samples from Zones E, G, and J of Loop 5R are shown in Table 13. The metal loss

FIGURE 19. RATIO OF FINAL TO INITIAL HOOP STRESS VERSUS RATIO OF FINAL TO INITIAL RADIUS AS A FUNCTION OF THE RATIO OF INITIAL GAS VOLUME TO INITIAL SPECIMEN VOLUME (α)

FIGURE 20. RELATIVE BIAxIAL STRESS-RUPTURE PERFORMANCE OF MILL ANNEALED CANDIDATE ALLOYS IN 1300°F SODIUM

FIGURE 21. BIAXIAL STRESS-RUPTURE PROPERTIES OF TYPES 304 AND 316 STAINLESS STEEL IN 1300°F SODIUM AS A FUNCTION OF STRUCTURE

FIGURE 21. BIAxIAL STRESS-RUPTURE PROPERTIES OF TYPES 304 AND 316 STAINLESS STEEL IN 1300°F SODIUM AS A FUNCTION OF STRUCTURE

FIGURE 22. BIAxIAL STRESS-RUPTURE PROPERTIES OF TYPE 316 STAINLESS STEEL: EFFECTS OF TEMPERATURE, ENVIRONMENT, AND STRUCTURE

TABLE 10

WEIGHT CHANGE DATA AND POSITIONS OF THE ZONE F CORROSION SAMPLES FROM LOOP 5R

Zone F Channel	Run	Direction of Sodium Flow → Position			
		1	2	3	4
1	5R-1	800 - A (381)	800 - A (342)	800 - H (297)	800 - A (360)
	5R-2	800 - A (309)	304 - A (171)	316 - S (74)	347 - H (237)
	5R-3	800 - A (686)	800 - S (556)	800 - S (563)	800 - A (664)
2	5R-1	304 - A (224)	304 - A (227)	304 - H (235)	304 - A (190)
	5R-2	304 - H (254)	800 - S (400)	347 - A (180)	316 - A (94)
	5R-3	304 - A (350)	304 - S (345)	304 - S (349)	304 - A (279)
3	5R-1	316 - A (153)	316 - A (136)	316 - H (227)	316 - A (128)
	5R-2	316 - A (124)	347 - A (192)	800 - H (310)	304 - S (117)
	5R-3	316 - A (206)	316 - S (176)	316 - S (122)	316 - A (142)
4	5R-1	347 - A (276)	347 - A (263)	347 - H (314)	347 - A (238)
	5R-2	347 - S (184)	316 - H (162)	304 - A (179)	800 - A (394)
	5R-3	347 - A (383)	347 - S (331)	347 - S (306)	347 - A (322)
5	5R-1*	800 - S (171)	800 - S (63)	800 - H (48)	800 - S (52)
	5R-2	800 - A (381)	304 - S (134)	316 - S (77)	347 - H (230)
	5R-3	800 - H (855)	800 - G (791)	800 - G (694)	800 - H ---
6	5R-1	304 - S (255)	304 - S (222)	304 - H (229)	304 - S (206)
	5R-2	304 - H (253)	800 - S (382)	347 - S (154)	316 - A (91)
	5R-3	304 - H (378)	304 - G (448)	304 - G (390)	304 - H (321)
7	5R-1	316 - S (170)	316 - S (168)	316 - H (222)	316 - S (121)
	5R-2	316 - S (89)	347 - A (193)	800 - H (299)	304 - S (114)
	5R-3	316 - H (277)	316 - G (294)	316 - G (271)	316 - H (244)
8	5R-1	347 - S (207)	347 - S (210)	347 - H (319)	347 - S (205)
	5R-2	347 - S (164)	316 - H (171)	304 - A (169)	800 - S (395)
	5R-3	347 - H (378)	347 - G (485)	347 - G (472)	347 - H (400)

A - mill annealed
H - 20% cold reduced

S - carbide agglomerated
G - Garafalo heat treatment

() - Weight loss, mg/dm²

*Inlet pressurized specimen burst, restricting or blocking the flow in this channel, consequently reducing and negating the corrosion data.

TABLE 11
AVERAGE CORROSION RESULTS OF THE ZONE F SAMPLES FROM LOOP 5R

<u>Material</u>	<u>Condition</u>	<u>Weight Loss, mg/dm²</u>		
		<u>1745 hours</u>	<u>2083 hours</u>	<u>3044 hours</u>
316 SS	Mill	103	139	174
	Annealed			
	Carbide	80	153	149
	Agglomerated			
	Cold Reduced	167	225	260
	Garafalo	---	---	282
304 SS	Mill	173	214	314
	Annealed			
	Carbide	121	228	347
	Agglomerated			
	Cold Reduced	254	232	350
	Garafalo	---	---	419
347 SS	Mill	188	259	352
	Annealed			
	Carbide	168	207	318
	Agglomerated			
	Cold Reduced	234	317	389
	Garafalo	---	---	476
Incoloy- 800	Mill	309	381	675
	Annealed			
	Carbide	392	---	560
	Agglomerated			
	Cold Reduced	305	297	855
	Garafalo	---	---	742

TABLE 12

AVERAGE CORROSION RESULTS OF THE ZONE E, G, AND J — TYPE 316 STAINLESS STEELSamples from Loop 5RWeight Loss, mg/dm²

<u>Test Zone</u>		<u>1745 hours</u>		<u>2083 hours</u>		<u>3044 hours</u>	
		<u>Tube</u>	<u>Ring</u>	<u>Tube</u>	<u>Ring</u>	<u>Tube</u>	<u>Ring</u>
E	Sodium	145	134	173	165	327	274
G	Flow	82	69	99	110	151	130
J	↓	69	79	77	89	126	117

FIGURE 23. BIAxIAL STRESS-RUPTURE PROPERTIES OF TYPE 304 STAINLESS STEEL: EFFECTS OF TEMPERATURE, ENVIRONMENT AND STRUCTURE

ANNEALED; $\sigma_H = 11,000$ psi

I 1 mil

20% COLD REDUCED; $\sigma_H = 12,000$ psi

I 1 mil

GLYCERIN

FIGURE 24. TYPE 316 STAINLESS STEEL—SURFACE STRUCTURE CHANGES AS A RESULT OF BIAxIAL STRESS RUPTURE TESTS IN 1300°F FLOWING SODIUM (3000 hours)

ANNEALED; $\sigma_H = 8,000$ psi

1 mil

20% COLD REDUCED; $\sigma_H = 11,000$ psi

1 mil

GLYCERINE

FIGURE 25. TYPE 304 STAINLESS STEEL—SURFACE STRUCTURE CHANGES AS A RESULT OF BIAxIAL STRESS RUPTURE TESTS IN 1300°F FLOWING SODIUM (1750 hours)

ANNEALED; $\sigma_H = 10,000$ psi

I 1 mil

20% COLD REDUCED; $\sigma_H = 13,000$ psi

I 1 mil

GLYCEREGIA

FIGURE 26. TYPE 347 STAINLESS STEEL—SURFACE STRUCTURAL CHANGES AS A RESULT OF BIAxIAL STRESS RUPTURE TESTS IN 1300°F FLOWING SODIUM (1750 hours)

ANNEALED, $\sigma_H = 9,000$ psi

I 1 mil

20% COLD REDUCED, $\sigma_H = 12,000$ psi

I 1 mil

GLYCERINE

FIGURE 27. INCOLOY-800—SURFACE STRUCTURAL CHANGES AS A RESULT OF BIAxIAL STRESS-RIPTURE IN 1300°F FLOWING SODIUM (3000 hours)

FIGURE 28. CORROSION RATES OF MILL ANNEALED MATERIALS IN 1300°F SODIUM

FIGURE 29. CORROSION RATES OF MILL ANNEALED TYPE 316 STAINLESS STEEL VERSUS LOOP POSITION IN 1300°F SODIUM

versus loop downstream position with the corresponding chromium and nickel changes is shown in Figures 34 through 36.

The typical microstructural effects of 1300°F sodium exposure on the corrosion samples are shown in Figures 37 through 40.

4.1.1.3 Discussion

Although the screening of the four alloys relative to their biaxial stress-rupture properties was not completely definitive, the results achieved were analyzed and weighed along with input from other materials programs. Based upon these considerations, mill annealed and carbide agglomerated Types-304 and 316 SS and Type-316 SS cold reduced 20% were recommended for further in-depth testing.

TABLE 13

X-RAY FLUORESCENCE ANALYSIS OF ZONE E, G, AND J CORROSION CARTRIDGE

SAMPLES FROM LOOP 5R

Test Zone	Sample	Surface	Cr			Ni			Mn			Mo		
			5R-1	5R-2	5R-3	5R-1	5R-2	5R-3	5R-1	5R-2	5R-3	5R-1	5R-2	5R-3
Standard	Ring*	o.d.	17.6	16.2	--	12.8	12.2	--	1.66	2.63	--	2.35	2.13	--
		i.d.	17.5	16.0	--	11.9	11.6	--	1.52	2.57	--	2.05	2.11	--
	Tube**	o.d.	18.2	17.5	17.6	14.0	14.2	6.4	2.00	2.77	1.65	3.30	2.46	2.18
		i.d.	18.2	17.8	17.2	13.7	14.7	13.1	1.90	2.90	1.54	3.75	2.61	2.17
E	Ring	o.d.	11.0	9.3	--	5.1	--	--	0.53	0.62	--	3.00	2.27	--
		i.d.	18.2	13.4	--	13.8	7.4	--	1.65	1.56	--	2.99	1.93	--
		o.d.	11.8	11.2	--	7.2	--	--	0.53	0.88	--	2.52	2.31	--
		i.d.	18.2	13.5	--	14.0	7.7	--	1.90	1.75	--	2.90	1.95	--
	Tube	o.d.	17.8	15.2	16.9	12.3	8.6	12.5	0.63	0.84	0.89	3.60	2.68	2.17
		i.d.	13.8	11.8	5.3	7.8	--	6.3	0.79	1.00	0.81	3.35	2.79	2.15
G	Ring	o.d.	17.9	13.6	14.2	12.9	5.7	11.2	0.66	0.57	0.84	3.59	2.56	2.26
		i.d.	16.1	12.1	6.6	11.8	2.1	6.7	1.10	1.22	0.88	3.50	2.57	2.17
	Tube	o.d.	15.8	12.8	--	10.9	4.9	--	0.69	0.96	--	2.65	2.09	--
		i.d.	18.2	14.6	--	13.8	9.1	--	1.60	1.86	--	2.90	1.96	--
		o.d.	17.5	13.8	--	12.7	9.1	--	1.20	1.72	--	2.62	2.07	--
		i.d.	18.2	15.1	--	13.6	10.7	--	1.73	2.08	--	2.95	2.03	--
	Tube	o.d.	19.1	14.6	14.9	13.7	6.5	11.4	1.00	0.74	0.83	3.59	2.55	2.29
		i.d.	17.5	13.7	13.9	12.8	7.9	12.2	1.27	1.76	1.21	3.48	2.42	2.19
		o.d.	19.2	15.0	16.6	13.2	8.1	12.2	0.92	0.70	0.86	3.50	2.39	2.21
		i.d.	17.6	13.9	13.5	12.5	7.7	12.0	1.20	1.54	1.17	3.35	2.37	2.19

TABLE 13 cont.

Test Zone	Sample	Surface	Cr			Ni			Mn			Mo		
			5R-1	5R-2	5R-3	5R-1	5R-2	5R-3	5R-1	5R-2	5R-3	5R-1	5R-2	5R-3
J	Ring	o.d.	16.9	13.2	--	11.0	3.7	--	0.67	0.72	--	2.90	2.23	--
		i.d.	18.0	14.2	--	13.2	8.4	--	1.40	2.04	--	2.93	1.89	--
		o.d.	17.5	14.8	--	12.7	5.5	--	1.20	0.84	--	2.70	0.78	--
		i.d.	18.2	15.4	--	14.1	10.8	--	1.45	2.12	--	2.90	2.02	--
	Tube	o.d.	18.2	14.8	16.6	12.9	7.0	12.2	0.69	0.98	0.85	3.59	2.51	2.22
		i.d.	17.8	15.0	14.7	12.8	10.4	12.1	1.25	1.81	1.23	3.50	2.61	2.17
		o.d.	18.2	17.1	17.9	13.1	13.0	12.6	0.65	1.14	0.87	3.55	2.67	2.19
		i.d.	17.7	15.3	14.9	12.8	11.6	12.3	1.30	1.80	1.21	3.49	2.68	2.21

* o.d. exposure

** i.d. exposure

Fe X-RAY IMAGE

Cr X-RAY IMAGE

Ni X-RAY IMAGE

Mo X-RAY IMAGE

1 mil

FIGURE 30. MODIFIED COMPOSITIONS OF GRAIN BOUNDARIES AND SURFACE LAYERS: TYPE 316 STAINLESS STEEL AFTER 2000 HOURS EXPOSURE TO 1300°F FLOWING SODIUM

1 mil

Fe X-RAY IMAGE

Cr X-RAY IMAGE

1 mil

Ni X-RAY IMAGE

Mn X-RAY IMAGE

FIGURE 31. MODIFIED COMPOSITIONS OF GRAIN BOUNDARIES AND SURFACE LAYERS: TYPE 304 STAINLESS STEEL AFTER 2000 HOURS EXPOSURE TO 1300°F FLOWING SODIUM

FIGURE 32. MODIFIED COMPOSITION OF GRAIN BOUNDARIES AND SURFACE LAYERS: TYPE 347 STAINLESS STEEL AFTER 2000 HOURS EXPOSURE TO 1300°F FLOWING SODIUM

FIGURE 33. MODIFIED COMPOSITION OF GRAIN BOUNDARIES AND SURFACE LAYERS FOR INCOLOY 800 AFTER 2000 HOURS EXPOSURE TO 1300°F FLOWING SODIUM

FIGURE 34. CORROSION AND CHANGE IN SURFACE COMPOSITION OF MILL ANNEALED TYPE 316 STAINLESS STEEL VERSUS LOOP POSITION AFTER 2083 HOURS IN 1300°F SODIUM

FIGURE 35. CORROSION AND CHANGE IN SURFACE COMPOSITION OF MILL-ANNEALED TYPE 316 STAINLESS STEEL VERSUS LOOP POSITION AFTER 1745 HOURS IN 1300°F SODIUM

FIGURE 36. CORROSION AND CHANGE IN SURFACE COMPOSITION OF MILL ANNEALED TYPE 316 STAINLESS STEEL VERSUS LOOP POSITION AFTER 3044 HOURS IN 1300°F SODIUM

ANNEALED; 1750 hours

ANNEALED; 3000 hours

1 mil

CARBIDE AGGLOMERATED; 1750 hours

20% COLD REDUCED; 3000 hours

GLYCEREGIA

FIGURE 37. TYPE 316 STAINLESS STEEL—STRUCTURAL EFFECTS IN SURFACE LAYERS AFTER 1750 AND 3000 HOURS EXPOSURE IN 1300°F FLOWING SODIUM

FIGURE 38. UPPER: TYPE 303 STAINLESS STEEL; LOWER: TYPE 347 STAINLESS STEEL — STRUCTURAL EFFECTS IN SURFACE LAYERS AFTER 1750 HOURS EXPOSURE IN 1300°F FLOWING SODIUM

ANNEALED HEAT No 1

INTERGRANULAR
DISSOLUTION

1 mil

ANNEALED HEAT No. 2

20% COLD REDUCED

INTERGRANULAR
DISSOLUTION

1 mil

ANNEALED HEAT No 2 UNETCHED

GLYCEROL

FIGURE 39. INCOLOY-800—STRUCTURAL EFFECTS IN SURFACE LAYERS AFTER 3000 HOURS EXPOSURE IN 1300°F FLOWING SODIUM

ZONE E: ENTRANCE

ZONE G: 52 in. DOWNSTREAM

1 mil

ZONE J 100 in. DOWNSTREAM

GLYCEREGIA

FIGURE 40. TYPE 316 STAINLESS STEEL—SURFACE COMPOSITION AND STRUCTURE CHANGES VERSUS LOOP POSITION IN 1300°F FLOWING SODIUM AFTER 3000 HOURS

GEAP-5719

The purpose of the carbide agglomeration heat treatment was to improve the stress-rupture properties of the materials by precipitating large carbides in the grain boundaries, primarily at triple points thereby retarding the creep processes involving grain boundary sliding. This effect has apparently been achieved at 1300°F for Type-316 SS, but not for Type-304 SS (Figure 21). The Type-304 SS did not respond to the carbide agglomeration heat treatment (1650°F) as observed by optical metallography. The explanation for this is that the Type-304 SS was at the minimum specification for carbon content, 0.04%. It has been shown⁽²¹⁾ that a 18Cr - 8Ni stainless steel has a solubility limit of 0.05% C at 1650°F. The low carbon content of the Type-304 SS is a probable explanation for the relatively low 1300°F stress-rupture properties shown in Figure 20. Austenitic stainless steels decarburize rapidly at 1300°F in a monometallic non-isothermal flowing sodium system⁽¹⁴⁾ and Type-304 SS decarburizes at a faster rate than Type-316 SS^(26, 27) —this contributes to the low stress-rupture properties. The relative slopes (greater decrease for Type-304 SS) of the stress-rupture curves in Figure 21 indicate a faster loss of carbon in the Type-304 SS.

The ductilities at failure ($\Delta D/D_1$ %) of the mill annealed Types-304 and 316 SS are quite similar.⁽¹⁾ The ductility of the Type-316 SS is substantially increased by the carbide agglomeration heat treatment, while for the Type-304 SS, the ductility is unchanged, or possibly reduced, by the heat treatment⁽¹⁾.

The application of a 20% cold reduction to the annealed material appears to improve the 1300°F stress-rupture properties of Types-304 and 316 SS, at least for times up to 3000 hours (Figure 21). The rupture ductilities of the 20% cold reduced materials are low, as would be expected, and as reported for Type-316 SS⁽¹⁹⁾.

Biaxial stress-rupture data obtained on Types-304 and 316 SS in environments other than non-isothermal flowing sodium are shown in Figures 22 and 23 and are compared to the data from Figure 21. The obvious difference in the data is that the cold reduced condition results in a "drop-off" of stress-rupture properties as compared to the annealed condition in 1200°F and 1400°F static sodium and 1300°F vacuum. An examination of the AI biaxial stress-rupture data in static^(18, 24) and isothermal flowing⁽²⁵⁾ sodium clarifies this difference:

- Cold work reduced the long-term stress-rupture properties of Types-304 and 316 SS below that of the annealed material. The effect increased with temperature (1200°F to 1400°F) and was more pronounced for Type 304 SS⁽¹⁸⁾.
- Sigma phase was predominant in the cold worked material. The formation of the sigma phase and the loss of grain boundary carbides was a result of prior cold work and high temperature deformation⁽¹⁸⁾.
- The reduction of the cold worked stress-rupture properties as associated with the initiation of voids at the sigma particles⁽¹⁸⁾.
- The microstructural effects in high purity static sodium were the same as in helium⁽¹⁸⁾.

- No degradation of Types-304 and 316 SS stress-rupture properties was observed in a high purity static sodium environment as compared to helium⁽¹⁸⁾.
- Annealed Types 304 and 316 decarburized when exposed to 1300°F and 1400°F zirconium gettered static sodium. A 1000 hour exposure produced a 30% to 50% loss of bulk carbon, the formation of sigma phase particles throughout the grain boundaries of the Type-304 SS, and a decarburized layer which transformed to sigma on the Type-316 SS⁽²⁴⁾.
- The exposure to zirconium gettered static sodium caused a 25% reduction in the stress-rupture properties of Type-304 SS as compared to tests in helium. Limited data on the Type-316 SS suggested a greater stability⁽²⁴⁾.
- The biaxial stress-rupture data in isothermal flowing sodium agrees with the data obtained in static sodium. The stress-rupture results in helium, static sodium, and isothermal flowing sodium support the concept that the sodium environment does not affect the life of cladding under isothermal test conditions⁽²⁵⁾.
- The absence of sigma phase after the exposure to isothermal flowing sodium was noted⁽²⁵⁾.

Therefore, the AI results can be summarized as follows:

- The formation of sigma in the cold worked material because thermal exposure was tied to the loss of stress-rupture properties, as compared to the annealed material which did not form sigma.
- The exposure to isothermal sodium produced no loss of stress-rupture properties as compared to controls in helium. The exposure to zirconium gettered sodium produced sigma in the annealed materials with a subsequent reduction in stress-rupture properties.

The reason for the agreement between the AI results and the UK (vacuum) results is as stated above; the formation of sigma in the cold worked material is due strictly to thermal exposure. The GE, PA-15 exposures in non-isothermal flowing sodium result in decarburization, as discussed previously, and the formation of sigma in both the mill annealed and 20% cold reduced Types-304 and 316 SS materials (Figures 23, 24, 36, and 37). Therefore, the absence of a loss in stress-rupture properties for the cold reduced material compared to the annealed material is realistic, since both structures decarburize and contain grain boundary sigma particles. The important factor is that in studying the effects of sodium on the mechanical properties of austenitic stainless steels, the tests must be performed in a non-isothermal flowing sodium environment.

The "crossover" of the cold worked and annealed stress-rupture properties described above can also be partially attributed to recovery or relaxation of internal elastic stresses in the cold worked material. It is reasonable to surmise that the

"crossover" is a function of (a) recovery and recrystallization of the cold worked material and (b) the precipitation and subsequent growth of second-phase particles (carbide and sigma) on grain boundaries, since all fissures found were associated with carbide and sigma particles⁽¹⁸⁾. The disappearance of the slip bands would be indicative of recrystallization, and an increase in rupture ductility with time would be attributed to a recovery process. However, neither of these changes occurred in the PA-15 tests on 20% cold reduced material at 1300°F for times up to 3000 hours.

The metallography of the Type-316 SS stress-rupture samples is shown in Figure 24. In addition to the extensive carbide precipitation on slip bands (20% cold reduced), twin boundaries, and grain boundaries, sigma phase is also found randomly in the grain boundaries. There is a thin ferritic surface phase (~ 1/8 mil) on both the annealed and cold worked material. The annealed material showed extensive necking, with large grain boundary voids, most of which were associated with sigma particles and some with triple points. The carbide agglomerated material (not shown) was similar. The cold worked material showed no necking, with a high density of small grain boundary voids, primarily associated with sigma particles.

The metallography of the Type-304 SS stress-rupture samples is shown in Figure 25. The structure is predominantly one of sigma particles in the grain boundaries and a 1-2 mil metastable surface phase. The grain boundaries and slip bands are rapidly denuded of carbides. The cold worked material has a higher density of grain boundary sigma particles than the annealed material. The annealed (also carbide agglomerated) material showed extensive necking with large grain boundary voids, most of which were associated with triple points, some with the sigma particles. The cold worked material showed no necking, with a high density of small grain boundary voids, primarily associated with sigma particles. The interconnection of these small voids is shown in Figure 25.

The metallography of the Type-347 SS stress-rupture samples is shown in Figure 26. The structure is predominantly one of grain boundary sigma particles (grain boundary delineation is lost), a 1 mil ferritic surface phase, and a 1 mil metastable surface phase below the ferritic phase. The cold worked material has a higher density of grain boundary sigma particles than the annealed material. Neither the annealed (also carbide agglomerated) or the cold worked materials exhibited necking, except in the seam weld areas. The annealed material had a low density of large grain boundary voids, primarily associated with triple points. The cold worked material had a high density of small grain boundary voids, primarily associated with the sigma particles.

The metallography of the Incoloy-800 stress-rupture samples is shown in Figure 27. The annealed material shown in the figure is from a heat that did not fail in the seam weld. The annealed structure (also carbide agglomerated) is predominantly one of what appears to be grain boundary sigma particles, the grain boundary delineation disappearing, and some grain boundary carbides, and 2 mils of intergranular dissolution after 3000 hours exposure. The cold worked structure has a mixture of carbide and sigma particles in the grain boundaries, carbides on slip bands, and 2 mils of intergranular dissolution after 3000 hours exposure. The depth of the intergranular

dissolution does not appear to be affected by the applied stress since the attack is comparable in the large exposed failure area and on the exposed surface (Figure 27). The annealed and carbide agglomerated material showed extensive necking, with a very low density of grain boundary (internal) voids. The failures appeared to be initiated by the surface attack, localized at one point and continuing through the cross-section, with little grain boundary fissuring elsewhere. The cold worked material showed no necking, with a high density of small grain boundary voids, primarily associated with sigma particles.

In summary, the annealed materials exhibit some grain deformation and a low density of grain boundary voids, primarily at triple points. The cold worked materials exhibit no grain deformation and extensive grain boundary fissures, primarily at carbide and sigma particles. The difference between the annealed and cold worked austenitic stainless steels can be attributed to the effects of grain hardening reducing the deformability of the cold worked material and to the weakening of the grain boundary by prior cold work where the voids are nucleated at precipitates by grain boundary sliding.

The corrosion results of the four test materials (Table 11) show that Type-316 SS has the lowest corrosion rate in all structural conditions, followed by Type-304 SS, Type-347 SS, and Incoloy-800, in order of increasing corrosion. The difference between the mill annealed and carbide agglomerated corrosion rates of the materials is not readily discernable. The data oscillate between the two structures, with the exception of Type-347 SS, where the carbide agglomerated structure has a consistently lower corrosion rate. For the 300-series austenitic stainless steels, the cold worked structure produces the highest corrosion rate. This is explainable on the basis of increased diffusion through a cold worked structure as compared to an annealed structure, where the primary paths of diffusion are the grain boundaries. The cold worked Incoloy-800 data have a high degree of scatter (also the annealed data), which is typical of a material undergoing grain boundary attack, where grains can randomly fall out of the material. The annealed data for the four test materials are shown in Figure 28. The data can be easily extrapolated to zero time-zero corrosion, indicating that linear corrosion rates (for all practical purposes) are operable in non-isothermal, 1300°F flowing sodium. A detailed discussion of linear corrosion rates in sodium has been presented previously⁽²⁾.

The mill annealed Type-316 SS corrosion cartridge data (Table 11) show the "downstream effect" of decreasing corrosion with downstream position, which has been described and discussed in detail previously⁽²⁾. The normal trend of tube sample corrosion higher than ring sample corrosion, explained previously⁽²⁾, is almost completely dominant. A plot of the data (Figure 29) shows that the linear extrapolation holds to the lowest corrosion rate area monitored (Zone J). The Type-316 SS data from the test section (Zone F) is shown in Figure 29 for comparison purposes. The sodium velocity in Zone F is 17 fps compared to 10 fps in the corrosion cartridges. Although the sodium mass flow rate in Zone F is only 1.2 gpm compared to 9.6 gpm in the corrosion cartridges, it is questionable whether the sodium mass flow rate has a major effect on the corrosion rates. Therefore, the effect of increased sodium velocity in Zone F should be to push the corrosion rate curve toward the inlet corrosion cartridge, Zone E,

and away from Zone G, where the loop positions, downstream (Zones F and G) are quite close. This appears to be the case, since the effect of increasing downstream position, decreasing the corrosion, is not a linear function, but logarithmic, which would force the Zone F corrosion curve further toward the Zone G corrosion curve. The opposite situation is observed.

The metal loss and corresponding Cr and Ni surface concentration changes of the Type-316 SS corrosion cartridge samples versus loop position are shown in Figures 34, 35, and 36. It can be seen that as the corrosion decreases, the Ni and Cr surface losses decrease. This agrees with the accepted theory⁽²⁾. The Ni and Cr surface losses in percent change appear to be parallel to the metal losses for each run. The fact that the curves are parallel is not important, but that the surface XRF and corrosion data directly correlate is important and encouraging to the support of the sodium corrosion model at 1300°F⁽²⁾. The interesting point to note about the surface compositional changes in Cr and Ni is that the relative losses of Cr and Ni oscillate with the various runs. For the 2083 hour time point, the Ni loss is higher than the Cr loss. This is also the case for the 1745 hour time point, but the Ni and Cr surface losses are substantially higher than in the 2083 hour run, especially the Ni. In the 3044 hour run, the Cr loss is higher than the Ni loss. Based on the theory that Cr forms an oxide which then goes into the sodium, and Ni goes into the sodium as atomic Ni,⁽²⁶⁾ it appears that the relative oxygen level of a particular run might be monitored by the surface XRF and metal loss data. In other words, when the Ni surface loss exceeds the Cr surface loss, the relative oxygen content of the run is less than when the opposite situation exists, since Cr corrosion is dependent on oxygen. Also, examining the Cr corrosion as a function of the chronological order of the runs, gives the impression that the oxygen is building-up in loop 5R as the total time of loop exposure increases.

The metallography of the Type-316 SS, Zone F corrosion samples is shown in Figure 37. The microstructures are the same as those discussed previously for the stress-rupture samples, indicating no effect of applied stress on the microstructural changes in 1300°F flowing sodium. The results are substantiated by the microprobe data (Figure 30) which show a loss of Cr and Ni up to 1.5 mils from the surface and an increase of Fe up to approximately 4 mils. The x-ray image for Fe shows a gradual increase in the outer 1 mil and a heavy concentration at the surface and in an apparent grain boundary (light line running almost horizontal in the center of the image). The x-ray image for Cr shows an absence at the surface and in the apparent grain boundary mentioned above. Nickel is also absent at the surface while Mo appears to be concentrated at local spots indicating high Mo sigma particles. It should be noted that the formation of the grain boundary sigma particles (decarburization) is relatively slow in annealed Type-316 SS, (Figure 37) as shown by the increased depth from 1750 to 3000 hours. Also, the carbide agglomerated material forms a continuous sigma phase grain boundary network. This is reasonable, since the heat treatment produces large carbides in the grain boundaries prior to exposure, effectively reducing the grain boundary carbon content (decarburization).

The metallography of the Types-304 and 347 SS, Zone F corrosion samples is shown in Figure 38. The microstructures are identical to those described previously

for the stress-rupture samples, again indicating no effect of the applied stress on the microstructural changes in 1300°F flowing sodium. The Type-304 SS microprobe data (Figure 31) show a loss of Cr and Ni up to 3 or 4 mils from the surface, Mn up to 2 mils, and an increase of Fe up to 4 mils. The x-ray images cover one of the large surface phase areas and show a concentration of Fe, and an absence of Cr, Ni, and Mn. The decrease of the alloying elements (Cr, Ni, and Mn) was not sufficient to produce a phase shift from fcc (γ) to bcc (α). However, the decrease apparently promoted a shear transformation of γ to α' as a result of the cold work introduced by vibratory polishing of the metallographic samples. The Type-347 SS microprobe data (Figure 32) show a substantial loss of Cr and Ni up to 0.5 mil from the surface with a total loss of Nb. The Fe exhibits a dip at approximately 2 mils from the surface, followed by an increase out to the surface. The x-ray images show a slight concentration of Fe and an absence of Cr and Ni in the 0.5 mil surface band. The x-ray image for Nb shows a concentration of this element in an area of no other apparent alloy presence. It is possible that this area is a particle high in Nb, probably NbC. The decrease of Cr and Ni in the 0.5 mil surface band accounts for the presence of the continuous ferritic phase in this area. The presence of the metastable grain boundary phase or the retardation of the sigma phase, or both, in the 2 mil band is not explained by the microprobe data.

The metallography of the Incoloy-800, Zone F corrosion samples is shown in Figure 39. Again the microstructures are the same, including the depth of intergranular dissolution, as those shown previously for the stress-rupture samples, indicating no effect of applied stress on the microstructural response in 1300°F flowing sodium. A substantial variation of grain size between the two heats of annealed material is shown to have no effect on the rate of intergranular dissolution. The microprobe data (Figure 33) show substantial losses of Cr and Ni, primarily Ni, up to 0.5 mil from the surface. However, the losses are not sufficient to produce a phase shift since a 20 Cr-14Ni alloy (surface composition) is fully austenitic. The microprobe traces also show the effect of crossing a "denuded" grain boundary area approximately 1 mil from the surface, where the loss of Cr and Ni, primarily Ni, can be seen. The x-ray images cover several grain boundary areas in the outer 1.25 mils. They show a concentration of Fe at the surface and in the grain boundaries and an absence of Cr, Ni, and Mn in these areas. The high loss of Ni (approximately 14%) appears to account for the intergranular "voiding" seen by metallography.

In summary, it can be said that although applied stress does not appear to affect the compositional changes in 1300°F non-isothermal flowing sodium, the compositional changes will certainly affect the mechanical properties in 1300°F flowing sodium, especially the long-term ($> 10,000$ hours) properties.

The microstructural changes of the mill annealed Type-316 SS corrosion cartridge samples versus loop position is shown in Figure 40. The change in structure, primarily surface structure, agrees with the corrosion and surface XRF data discussed previously (Figures 34, 35, and 36). The highest corrosion rate position (highest elemental surface loss) exhibits a ferritic surface phase. The ferritic surface phase disappears as the corrosion rate decreases, and, consequently, the elemental surface loss diminishes to the point where a phase shift is not possible.

4.1.1.4 Summary and Conclusions

- a. The use of weldrawn pressure tubing for stress-rupture testing is unacceptable because of localized deformation in the weld region.
- b. For the heats of material tested, Type-316 SS exhibits better stress-rupture properties than Type-304 SS in 1300°F non-isothermal flowing sodium at times up to 3000 hours.
- c. The carbide agglomeration heat treatment improves the stress-rupture properties (rupture strength and ductility) of Type-316 SS in 1300°F non-isothermal flowing sodium. The results for Type-304 SS were not conclusive because of the lack of a response to the heat treatment.
- d. Types-304 and 316 SS in the 20% cold reduced condition exhibit improved stress-rupture properties (rupture strength) in 1300°F non-isothermal flowing sodium as compared to the mill annealed material for times up to 3000 hours. The rupture ductilities of the cold worked material are significantly lower.
- e. In the stress-rupture tests, the annealed austenitic stainless steels exhibit grain deformation and a low density of grain boundary fissures, primarily at triple points, and the cold worked materials exhibit no grain deformation with extensive grain boundary fissures, primarily at precipitate phases.
- f. The exposure of austenitic stainless steels to 1300°F non-isothermal flowing sodium results in decarburization and the subsequent formation of grain boundary sigma particles in all structural conditions (mill annealed, carbide agglomerated, and 20% cold reduced).
- g. The evaluation of the effects of sodium on the mechanical properties (stress-rupture) of austenitic stainless steels must be performed in non-isothermal flowing sodium test facilities.
- h. The finite effect of exposure to 1300°F non-isothermal flowing sodium on the stress-rupture properties of the austenitic stainless steels cannot be determined on the basis of the sodium exposures alone. Concurrent stress-rupture tests in an inert environment on identical heats of material are necessary.
- i. Type-316 SS in the mill annealed, carbide agglomerated, and 20% cold reduced conditions exhibits the lowest corrosion rates in 1300°F non-isothermal flowing sodium of the materials tested. The corrosion rates for the annealed materials are: Type-316 SS — 48 mg/dm² - month Type-304 SS — 74 mg/dm² - month Type-347 SS — 85 mg/dm² - month Incoloy-800 — 165 mg/dm² - month (167 mg/dm² - month ≈ 1 mil/yr).
- j. The 20% cold reduced materials have a slightly higher corrosion rate in 1300°F non-isothermal flowing sodium than the mill annealed materials.
- k. The "general" corrosion characteristics of austenitic stainless steels in non-isothermal flowing sodium were observed: hot-leg metal loss, corrosion rate decreasing with "downstream" position and selective removal of Cr, Ni, and Mn resulting in surface phase changes.
- l. Incoloy-800 exhibits a moving front of intergranular attack (dissolution) which has progressed 2 mils after 3000 hours exposure to 1300°F non-isothermal flowing sodium. The intergranular attack is a result of high losses of Cr, Ni, and Mn from the grain boundary regions to the sodium.

- m. Applied stress does not appear to affect the compositional and microstructural perturbations produced by exposure to 1300°F non-isothermal flowing sodium. However, the effects of the compositional and microstructural changes on the mechanical properties of the austenitic stainless steels should be appreciated.

4.1.2 Intergranular Attack

A test was performed to evaluate steam as an agent for the post-exposure cleaning of fuel cladding. Two tests were run for 500 hours at 1175°F in a pot of static sodium. The test samples (2-1 per test) were 0.281-inch o. d. x 0.015-inch wall x 8-inches long annealed Type-316 SS sheaths swaged over 230V-1000W Firerod* heaters. After the first test, the sample was removed into an inert environment, heated to 400°F, and steam cleaned. In the second test, the sample was removed into an inert environment and cleaned in alcohol and water. The heater was present in the second test to obtain the same stress configuration during the sodium exposure as was present in the steam cleaning test.

The exposures to the static sodium resulted in varying degrees of attack, none of which could be attributed to the cleaning procedures. The sodium in the pot was not cold or hot trapped. Analyses of the sodium were performed for oxygen (amalgamation), hydrogen (diffusion), and carbon (wet combustion). The results ranged as high as 150 ppm O₂, 14 ppm H₂, and 46 ppm C.

The 8-inch Type-316 SS heater sheaths were immersed in the sodium to 5 or 6 inches of their length. A 2 psig argon cover gas was maintained over the sodium, and the remaining 2 to 3 inches of the heater sheath was exposed to this vapor phase.

The attack observed in the sodium was intergranular oxidation (Figure 41). The oxidation also occurred along slip bands which resulted from the swaging. An end plug was welded to the bottom of the heater sheath and the weld ground off. In this area of extensive surface cold work, the slip band oxidation was severe (Figure 42). Intergranular attack and the formation of an oxide in high-oxygen sodium has been noted previously.

The attack of major interest and concern occurred in the material exposed to sodium vapor. The Type-316 SS surface suffered general oxidation with some leading intergranular penetrations (Figure 43). This type of attack in the vapor phase has not been seen before and the cause is unknown at the present time.

*Trademark of the Watlow Electric Manufacturing Co.

500X

10% OXALIC

500X

FIGURE 41. TYPE-316 STAINLESS STEEL HEATER SHEATH EXPOSED TO 1175°F STATIC SODIUM FOR 500 HOURS

10% OXALIC

500X

FIGURE 42. TYPE-316 STAINLESS STEEL HEATER SHEATH (SURFACE GROUND) EXPOSED TO 1175°F STATIC SODIUM FOR 500 HOURS

10% OXALIC

1000X

500X

FIGURE 43. TYPE-316 STAINLESS STEEL HEATER SHEATH EXPOSED TO THE VAPOR PHASE IN A 1175°F POT OF STATIC SODIUM FOR 500 HOURS

SECTION V

LIST OF SODIUM MASS TRANSFER PROJECT REPORTS

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PROGRAM CONTRIBUTORS

TASK 1 — LMFBR/FFTF CORROSION PARAMETERS STUDIES

R. Arnold
J. T. Cochran
M. Gebhardt
A. Montes
M. D. Moore
P. Roy
M. K. Schad
W. Wagner

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

L. G. Butler
D. Dutina
J. L. Simpson
A. G. Steamer

TASK 4 — MECHANICAL PROPERTIES FOR CLADDING MATERIALS AND LOCALIZED ATTACK

G. G. Gaul
A. Montes
W. Wagner
G. P. Wozadlo

GENERAL PROGRAM AND CONSULTING

G. D. Collins
F. A. Comprelli
R. W. Lockhart
L. E. Pohl
W. W. Sabol
C. N. Spalaris
R. S. Young
E. L. Zebroski

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Glen W. Wensch, Chief	2	M. J. Whitman	1
Liquid Metal Project Branch		Assistant Director for Program	
Div. of Reactor Development & Tech.		Analysis	
AEC Headquarters		Div. of Reactor Development & Tech.	
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for Nuclear Safety		Washington D. C. 20545	
Div. of Reactor Development & Tech.			
AEC Headquarters			
Washington, D. C. 20545			
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Washington, D. C. 20545		Washington, D. C. 20545	
N. Grossman, Chief	1	J. J. Morabito, Special Project	1
Engineering Development Branch		Branch	
Div. of Reactor Development & Tech.		FFTF Project	
AEC Headquarters		Div. of Reactor Development & Tech.	
Washington, D. C. 20545		AEC Headquarters	
		Washington, D. C. 20545	

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E. E. Sinclair	1	W. R. Simmons	1
Asst. Director for Reactor Tech.		Argonne National Laboratory	
Div. of Reactor Development & Tech.		9700 South Cass Avenue	
AEC Headquarters		Argonne, Illinois 60439	
Washington, D. C. 20545			
A. Giambusso	1	R. Bane	1
Asst. Director for Project		Argonne National Laboratory	
Management		9700 South Cass Avenue	
Div. of Reactor Development & Tech.		Argonne, Illinois 60439	
AEC Headquarters			
Washington, D. C. 20545		R. Humphreys, LMFBF Program	1
M. Booth, Chief	2	Office	
Systems Engineering Branch		Argonne National Laboratory	
Div. of Reactor Development & Tech.		9700 South Cass Avenue	
AEC Headquarters		Argonne, Illinois 60439	
Washington, D. C. 20545			
Chief, Administrative Budget and	1	L. Kelman	1
Program Analysis		LMFBF Program Office	
Div. of Reactor Development & Tech.		Argonne National Laboratory	
AEC Headquarters		9700 South Cass Avenue	
Washington, D. C. 20545		Argonne, Illinois 60439	
J. H. DeVan	1	W. R. Wykoff	1
Oak Ridge National Laboratory		Los Alamos Scientific Laboratory	
P. O. Box Y		Los Alamos, New Mexico 87544	
Oak Ridge, Tennessee 37830			
J. White	1	G. Waterbury	1
Oak Ridge National Laboratory		Los Alamos Scientific Laboratory	
Oak Ridge, Tennessee 37830		Los Alamos, New Mexico 87544	
R. E. MacPherson	1	L. E. Whinery	1
Oak Ridge National Laboratory		Los Alamos Scientific Laboratory	
Oak Ridge, Tennessee 37830		Los Alamos, New Mexico 87544	
D. Gurinsky	1	E. R. Astley, FFTF Project	3
Brookhaven National Laboratory		Manager	
Upton, Long Island		Battelle-Pacific Northwest	
New York 11973		Laboratory	
Sherman Greenberg	1	Richland, Washington 99352	
Argonne National Laboratory		Supervisor of Configuration	2
9700 South Cass Avenue		Control	
Argonne, Illinois 60439		FFTF Project	
F. Smith	1	Battelle-Pacific Northwest	
Argonne National Laboratory		Laboratory	
9700 South Cass Avenue		Richland, Washington 99352	
Argonne, Illinois 60439		R. W. Dickinson, Director, LMEC	1
		Atomics International	
		P. O. Box 309	
		Canoga Park, California 91304	

J. Droher, Director LMIC Atomics International P. O. Box 309 Canoga Park, California 91304	1	RDT Senior Site Representatives Pacific Northwest Laboratory Richland, Washington 99352 Attn: P. G. Holsted	1
C. E. Weber Atomics International Canoga Park, California 91304	3	RDT Senior Site Representatives Atomic Power Development Assc., Inc. Detroit, Michigan 48226 Attn: J. D. Wille	1
Technical Director Atomic Power Development Assc., Inc. 1911 First Street Detroit, Michigan 48226	2	Division of Technical Information Ext. U. S. Atomic Energy Commission P. O. Box 62 Oak Ridge, Tennessee 37831	50
R. C. Werner Mine Safety Appliances Research Corp. Callery, Pennsylvania	1	J. Bishop Advance Research Division Westinghouse Corporation P. O. Box 159 Madison, Pennsylvania 15663	1
Phillip S. Otten Baldwin-Lima-Hamilton Corporation Eddystone, Pennsylvania 19013	1	Los Alamos Scientific Laboratory P. O. Box 1663 Los Alamos, New Mexico 87544 Attn: Reports Librarian	1
J. H. Wright Westinghouse Corporation Atomic Power Division P. O. Box 355 Pittsburgh, Pennsylvania 15230	1	S. Golan, Project Manager 1000 MWe LMFBR Follow-On Study Atomics International P. O. Box 309 Canoga Park, California 91304	2
Paul E. Probert, Project Engineer Babcock and Wilcox Company Barberton, Ohio 44203	1	M. W. Croft, Project Manager 1000 MWe LMFBR Follow-On Study The Babcock and Wilcox Company P. O. Box 1260 Lynchburg, Virginia 24505	2
C. A. Barrett National Aeronautics and Space Administration Lewis Flight Propulsion Laboratory Cleveland, Ohio 44135	1	W. P. Staker, Project Manager 1000 MWe LMFBR Follow-On Study Combustion Engineering, Inc P. O. Box 500 Windsor, Connecticut 06095	2
Kurt Goldmann United Nuclear Corporation White Plains, New York 10601	1	W. Chernock Combustion Engineering, Inc. P. O. Box 500 Windsor, Connecticut 06095	2
RDT Senior Site Representatives Atomics International Canoga Park, California 91305 Attn: R. L. Morgan	1		
RDT Senior Site Representatives General Electric Company Sunnyvale, California 94086 Attn: J. V. Levy	1		

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C. A. Anderson, Project Manager 2
1000 MWe LMFBR Follow-On Study
Westinghouse Electric Corporation
Advanced Reactors Division
Waltz Mill Site, P. O. Box 158
Madison, Pennsylvania 15663

L. W. Fromm, Manager 2
1000 MWe LMFBR Follow-On Study
Building 208
Argonne National Laboratory
9800 South Cass Avenue
Argonne, Illinois 60439