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GEAP-5648
AEC RESEARCH AND
DEVELOPMENT REPORT
JULY 1968

MASTER

**SODIUM COMPONENTS
DEVELOPMENT PROGRAM
MASS TRANSFER INVESTIGATIONS
IN LIQUID METAL SYSTEMS
QUARTERLY PROGRESS REPORT
NO. 5
MARCH - MAY 1968**

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

ADVANCED PRODUCTS OPERATION

GENERAL ELECTRIC

SUNNYVALE, CALIFORNIA

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SODIUM COMPONENTS DEVELOPMENT PROGRAM
MASS TRANSFER INVESTIGATIONS IN LIQUID METAL SYSTEMS

Quarterly Progress Report No. 5

March—May 1968

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Prepared for the
U. S. Atomic Energy Commission
Contract No. AT(04-3)-189
Project Agreement 15

*Printed in U. S. A. Available from the
Clearing House for Federal Scientific and Technical Information
National Bureau of Standards, U. S. Department of Commerce
Springfield, Virginia
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TABLE OF CONTENTS

		<u>Page</u>
	ABSTRACT	ix
SECTION I	INTRODUCTION AND PROGRAM SUMMARY	1
	1.1 General	1
	1.2 Reports	3
SECTION II	MASS TRANSFER AND CORROSION SPECIFICATIONS	4
	2.1 Subtask 1.1—Reactor Corrosion Mockup Loop Testing	4
	2.2 Subtask 1.3—Development of High Heat Flux Heaters	37
SECTION III	CLADDING MATERIALS TESTING	56
	3.1 Subtask 2.1—Mechanical Properties Test of Cladding Candidates	56
SECTION IV	TASK 3—SODIUM CHEMISTRY	62
	4.1 Subtask 3.1—Sampling of Sodium Systems	62
	4.2 Subtask 3.2—Impurities Identification in Sodium Systems	63
SECTION V	LIST OF SODIUM MASS TRANSFER PROJECT REPORTS	70
	REFERENCES	72
	CONTRIBUTORS	73
	DISTRIBUTION	74

LIST OF ILLUSTRATIONS

<u>Figure</u>	<u>Title</u>	<u>Page</u>
2-1	Sodium Mass Transfer LMFBR Corrosion Mockup Loop 8	6
2-2	Corrosion Cartridge Assembly Showing the Typical Ring and Tube Specimens	7
2-3	Weight Change and Exposed Surface X-Ray Fluorescence Data versus Loop Position for the Tube Specimens from Run 8-1 after ~ 3000 Hours Exposure	19
2-4	Weight Change and Exposed Surface X-Ray Fluorescence Data versus Loop Position for the Tube Specimens from Run 8-2 after ~ 3000 Hours Exposure	21
2-5	Crevice Surface X-Ray Fluorescence Data versus Loop Position for the Tube Specimens from Run 8-1 after ~ 3000 Hours Exposure	23
2-6	Crevice Surface X-Ray Fluorescence Data versus Loop Position for the Tube Specimens (Ring Specimens included in Zone F) from Run 8-2 after ~ 3000 Hours Exposure	25
2-7	Run 8-1 Tube Specimens Showing the Surface Discoloration after Sodium Exposure	27
2-8	Typical Microstructural Effects of the Tube Specimens from Run 8-1 after ~ 3000 Hours Exposure	29
2-9	Typical Microstructural Effects of the Tube Specimens from Run 8-2 after ~ 3000 Hours Exposure	31
2-10	Sketch Showing the Areas of Metallographic and X-Ray Examination of the Type 316 Stainless Steel Electron Bombardment Heater Anode	39
2-11	Outside Diameter Surface and General Microstructure of the Type 316 Stainless Steel Anode—Metallographic Specimen No. 1	40
2-12	Outside Diameter Surface and General Microstructure of the Type 316 Stainless Steel Anode—Metallographic Specimen No. 5	40
2-13	Outside Diameter Surface and General Microstructure of the Type 316 Stainless Steel Anode—Metallographic Specimen No. 8	41
2-14	Outside Diameter Surface and General Microstructure of the Type 316 Stainless Steel Anode—Metallographic Specimen No. 9	41
2-15	Surface Composition Change and Ferritic Layer Growth of the Type 316 Stainless Steel Anode	42
2-16	Peak Surface Temperature Profiles of the Type 316 Stainless Steel Anode	43
2-17	Structural Variation Across the Wall of the Type 316 Stainless Steel Anode—Metallographic Specimens No. 2 and 3	49
2-18	Structural Variation Across the Wall of the Type 316 Stainless Steel Anode—Metallographic Specimens No. 7 and 8	49
2-19	Typical Inside Diameter Surface Deposit of the Type 316 Stainless Steel Anode at the Ends of the Emitter Zone	50
2-20	Structural Variation Across the Wall of the Type 304 Stainless Steel Anode—Metallographic Specimens No. 2 and 4	50
2-21	Detail of Heater Construction	51
2-22	Electron Bombardment Heater Control Circuit	53
2-23	Temperature Rise Along HHFH	54
2-24	Temperature Rise Along HHFH	55
3-1	Corrosion Rates of Annealed Materials in 1300°F Sodium	58
3-2	Corrosion Rates of Annealed Type 316 Stainless Steel versus Loop Position in 1300°F Sodium	59
3-3	Biaxial Stress Cycle Fatigue of Type 316 Stainless Steel in 1300°F Sodium	61
4-1	Contents of Loop 1 (Cold Trap Sectioned into Four Pieces)	65

.

9

,

4

1

LIST OF TABLES

<u>Table</u>	<u>Title</u>	<u>Page</u>
2-1	Runs 8-1 and 8-2 Operating Conditions	8
2-2	Weight Change, Loop Position, and Velocity of Run 8-1	9
2-3	Weight Change, Loop Position, and Velocity of Run 8-2	14
2-4	X-Ray Analysis of the Type 316 Stainless Steel Electron Bombardment Heater Anode	44
2-5	X-Ray Analysis of the Type 304 Stainless Steel Electron Bombardment Heater Anode	45
3-1	Effect of Alloy Composition and Structural Condition on Corrosion Weight Loss, mg/dm ²	57
3-2	Effect of Loop Position on the Corrosion of Type 316 Stainless Steel Weight Loss, mg/dm ²	57
3-3	Biaxial Stress Cycle Fatigue Results from Run 6R-1 in 1300 °F Sodium	60
4-1	Concentration of Carbon in Cold Trap Sodium	64
4-2	Forms of Carbon in Cold Trap Deposits	66
4-3	Hydrogen, Alkalinity, and Plugging Temperature Measurements for Loop 9	68
4-4	Hydrogen, Alkalinity, and Plugging Temperature Measurements for Loop 8	68
4-5	Analyses for Carbon—Loops 8 and 9	69
4-6	Metal Impurities—Loops 8 and 9	70

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1

1

1

1

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ABSTRACT

Work is reported on investigations of materials corrosion, mechanical properties, and sodium impurities in pumped sodium systems operating under controlled purity conditions to maximum temperatures of 1300°F

Empirical equations representing hot leg corrosion measured in runs 8-1, 8-3, and 9-1 are presented for comparison. Metallurgical evaluation of results of runs 8-1 and 8-2 and metallographic examinations of high heat flux heaters were completed. Significant observations include decarburization in the 1200°—1300°F region of an austenitic system and carburization within the same system at lower sodium temperatures, and a higher corrosion rate of Type 316 stainless steel in sodium at 1200°—1300°F under heat transfer conditions compared to equivalent isothermal conditions.

The analysis of corrosion data obtained for Types 316, 304 and 347 stainless steel and Incoloy 800 during screening tests in loops 5R and 6R is reported. The corrosion rates observed are linear. A greater rate is found for Incoloy 800 than for the austenitic grades of stainless steel. Low corrosion rates observed in the test sections for mechanical properties are attributed to low mass flow rates. It is suggested that a Reynolds Number encompassing velocity, mass flow, and hydraulic diameter should be included in further development of corrosion models.

Preliminary results of examination of a cold trap from an austenitic (Type 316 stainless steel) system are given and compared to results from a cold trap from a ferritic system. A factor of three less total carbon was found in the austenitic system. The carbonate and cyanide anions were the major carbon containing forms in the residues from the ferritic system. These were reduced sevenfold in the austenitic loop deposits. A compound yielding acetylene upon hydrolysis of the ferritic loop deposits was not detected in the austenitic loop.

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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, 2-1/4Cr-1 Mo and 5 Cr-1/2 Mo-1/2Ti ferritic steels were studied in depth at sodium temperatures to 1200°F. In 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 examined under the variables of temperature, sodium velocity, sodium impurity content under cold trap control, and type of material.

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

control in support of the FFTF program. The current scope of work is divided into the following tasks and subtasks.

Task 1—Mass Transfer and Corrosion Specifications

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 to FFTF.

Subtask 1.1—Reactor Corrosion Mockup Loop Testing

The ranges of test parameters covered in Phase I of the Project have been extended. Test Loops 8 and 9 were designed to simulate the expected range of temperatures, velocities, sodium purity, and corrosion potentials which might apply to a 1000 MW reactor system. A series of four runs of 3000 hours each is being conducted in Loop 8 to study the effects (two levels each) of sodium velocity, position (downstream measured in L/D units), and flow (gpm). Test materials are Types 316 and 304 stainless steel. Top temperatures of 1300°F are being utilized and sodium purity is controlled by cold trap to <300°F plugging temperatures. Replicate data and the effects of a wider range of velocity, several steps from 0.7 to 24 fps, have been obtained in Loop 9.

Three runs of 3000 hours each have been completed in Loop 8. After completion of the fourth run in early fiscal year 1969, refinement of empirical corrosion models can be made.

Subtask 1.2—FFTF Closed Loop Simulation

The initial objective of this task was to determine the corrosion, mass transfer, and deposition phenomena associated with the use of Type 304 stainless steel in a system with bulk sodium temperatures to 1300°F. The hot leg of Loop 9 and cartridge sample holders were modified during fiscal year 1968 to mock up temperature profiles and corrosion potentials to bracket the range of calculated corrosion and deposition rates of a 5 MW closed loop design. Type 304 stainless steel was utilized in all modifications. Provisions have been included for utilizing heat fluxes of 0.5 to 1.0×10^6 Btu/h-ft² for some material tests (with resultant increased surface temperatures).

Subtask 1.3—Development of High Heat Flux Heaters

The objective of this task is the development of high heat flux heaters capable of operating for times to ~3000 hours to obtain effects of increased temperature at metal surfaces caused by heat flux in the range of 1×10^6 Btu/h-ft². The work is in direct support of Subtask 1.1 and 1.2. Electron bombardment heating is currently being tested.

Subtask 1.4—FFTF Support, Refueling Cell Mockup

The refueling cell mockup, initially built for SEFOR, was used for sodium vapor deposition and vapor transport experiments during July 1967. A series of tasks were performed in the cell in an argon atmosphere above a 4-foot diameter pool of liquid sodium. Results are reported in GEAP-5560.

Task 2—Cladding Materials Testing

The objectives of this task are to develop mechanical property design data

for candidate materials for fast oxide cladding, and to investigate local corrosion phenomena resulting in intergranular attack

Subtask 2 1—Mechanical Properties Tests of Cladding Candidates

Work under this task includes screening of several candidate materials for LMFBR fuel cladding in preparation for obtaining design data, long term stress-rupture (to 10,000 hours), and stress fatigue data (to 4000 hours). Present emphasis is on test conditions of 1300°F, sodium velocities of 12 to 15 fps, and controlled sodium purity corresponding to <300°F plugging temperatures.

Types 304, 316, and 347 stainless steels, and Incoloy 800 are currently being screened in Loop 5R under the above conditions at various static stress levels. Equipment for testing under conditions of alternating stress levels has been completed (Loop 6R). Loop 4R is being placed in operation for long-term stress-rupture.

The investigation of localized intergranular phenomena is also underway as part of this task. Statically pressurized tube specimens are being exposed in regions of test loops in an attempt to discover sodium environmental and material conditions that result in intergranular attack

Task 3—Sodium Impurities

The sodium impurities task covers the chemical monitoring of test loops. Sensitive noncontaminating sampling methods 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 and control the levels of impurities which affect corrosion mechanisms

Subtask 3.1—Sodium System Sampling and Impurities Detection

The objectives of this task are to develop and apply sensitive means for determining impurities in sodium in the various test loops and to identify and eventually control the species affecting corrosion rates and mechanical properties.

Solid material collectors and modified plugging indicators have been utilized to collect and identify hydrogen compounds precipitating at 500°–600°F. Single tubes and flow-through pots of 1.3-liter capacity are being utilized to collect sodium from operating loops to supply samples for analysis and concentration of impurities.

Major effort is currently being applied in measuring impurities in both the cover gas and in the liquid sodium on a continuous basis during test runs. A gas chromatograph has been connected to Loop 8 hot and cold leg surge tanks to measure and record continuously hydrogen and nitrogen concentration of the cover gas. In parallel with this, a sodium distillation station designed to distill 50 gm batches of sodium from the loop is being readied for connection to Loop 8. Program and weight recording equipment will control the process to concentrate impurities in 1 kg of sodium in any one run. A UNC oxide meter installation is being designed for operation along with the still. Data from each unit will be correlated. A special test is being designed to trap magnetic particles of the type observed from the impingement cartridges of Loop 9 (run 9-1).

Subtask 3.2—Sodium System Impurities Identification

The current objective of this task is to utilize the refined chemical techniques for impurity concentration and analysis to characterize sodium in operating loops and contents of cold traps. Sampling plans for loop cover gas and sodium have been devised to provide data for all phases of loop operations.

Task 4—Offsite Monitoring

This task monitors large sodium systems for corrosion and mass transfer. The objective is to obtain verification of corrosion models and equation constants developed in Task 1. Nine sample holder assemblies were designed and built and have been installed in the SCTI test facility of LMEC.

1.2 REPORTS

Monthly report letters are prepared reporting progress in all tasks. Progress letters No. 1 through 102 covering work from July 1959 through April 30, 1968 have been issued.

In addition to the progress letters, 28 topical reports and 4 quarterly progress reports covering test results have been issued (see Section V).

This is the fifth quarterly progress report issued and covers significant results obtained in project task areas. The objective is to report the results of data analysis and examinations in those tasks that have reached a meaningful stage of development. It is expected that this will make information available at an earlier date than permitted by the topical report system. The material in the following sections is identified according to the task it supports.

SECTION II

MASS TRANSFER AND CORROSION SPECIFICATIONS2.1 SUBTASK 1.1—REACTOR CORROSION
MOCKUP LOOP TESTING

Mockup studies related to the PA-15 mass transfer program involve (1) experimentally determining mass transfer, corrosion, and exposure effects in steel exposed to flowing sodium at bulk temperatures up to 1300°F, (2) reevaluating the mass transfer corrosion model on the basis of scaleup or geometric parameters to permit prediction of mass transfer corrosion in full-size systems, (3) monitoring for localized attack, and (4) generating information which will permit establishing relationships between sodium impurities, loop operations, and metallurgical effects.

Loops 8 and 9, which were constructed to simulate corrosion conditions of a 1000 MWe coolant system, provide the major sources of data for the mockup studies. Loop 8 is scheduled for a series of four parametric runs conceived to study the effects of sodium velocity, mass flow rate, and downstream on mass transfer rates. Loop 9 has completed one run to study the range of velocity effect on mass transfer rates.

2.1.1 Corrosion Rate Prediction Equations
for the Hot-Leg Based on Data from
Runs 8-1, 8-2, 8-3, and 9-1

The raw data, including weight change, temperature, time, and flow from the isothermal hot-leg sections, F, G, and H, in runs 8-1, 8-2, 8-3, and 9-1 are in the process of being transformed into the following terms of the empirical model for corrosion rate

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

where

CR = average corrosion rate,
mg/dm²-month

V = sodium velocity, ft/sec

(L/D)₁ = downstream factor, number of
hydraulic diameters downstream
in an isothermal region

(L/D)₂ = downstream factor, number of
hydraulic diameters downstream
in an isovelocity region (single
corrosion cartridge)

t = exposure time, months

b₀, b₁, etc. = constants

Reevaluating of the equation format and statistical analysis of the coefficients (constants) between the four runs is in progress. The oxygen (1.156 ln Ox) and temperature $\left(\frac{-23,827}{t+460}\right)$ variables previously

estimated in Phase I have been held constant in current tests and will not be included in the above equation. It will suffice for the present model treatment to note that the temperature is 1300°F and the oxygen, <10 ppm. The following equations have been obtained based upon analysis of the data for individual runs.

Run 8-1

$$\ln CR = 3.25 + 0.340 \ln V - 0.008 (L/D)_1 \\ - 0.086 \ln (L/D)_2 + 1.035/t + 1$$

$$\ln CR = 3.54 + 0.350 \ln V - 0.0007 (L/D)_1 \\ - 0.087 \ln (L/D)_2$$

Run 8-3

$$\ln CR = 4.81 + 0.158 \ln V - 0.0020 (L/D)_1 \\ - 0.074 \ln (L/D)_2 + 0.600/t + 1$$

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

Run 9-1

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

The absence of a time term in the run 9-1 equation is due to only one time point for the run. For this reason, runs 8-1 and 8-3 were also compared without the time term. It should be noted for runs 8-1 and 8-3 that the absence of the time term causes a change in all the coefficients, with the constant, b_0 , being affected most. The development of the run 8-2 equation is in process.

2.1.2 Metallurgical Evaluation of the Results from Runs 8-1 and 8-2

The test facility, mockup Loop 8, is designed to simulate corrosion effects of the coolant throughout the primary system of a Liquid Metal Fast Breeder Reactor (LMFBR). As shown in Figure 2-1, Zones C and E correspond to the core fuel, and Zone F to the upper blanket and gas plenum of the reactor core. Zone G corresponds to the reactor vessel upper plenum and Zone H to the primary system outlet pipe. Zones J, K, and M correspond to the heat exchanger, inlet to outlet. Zones N and R correspond to the

primary system return piping and Zone B to the primary system pump or extreme downstream position. Operating parameters, such as temperature and oxygen level, duplicate the hot channel values of an LMFBR.

The loop capacity and test sample complement are designed for a parametric study of geometry effects such as sodium velocity, loop position, and mass flow rate, on the corrosion rate. The mass transfer (corrosion-deposition) is monitored by ring and tube samples, Figure 2-2. A modular form of sample holder is employed which provides for sample exposure under the following conditions:

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

2.1.2.1 Loop Operating Parameters

The nominal operating conditions for runs 8-1 and 8-2 were 3000 hours at 1300°F T_{max} , 800°F T_{min} , and an oxygen content ≤ 10 ppm based on a plugging temperature (T_s) of 260°–280°F. The flow in run 8-1 was specified at 3 gpm and for run 8-2, 6 gpm. Several corrosion cartridges were removed at an intermittent time point, 2000 hours for 8-1 and 1000 hours for 8-2, resulting in a mixture of 1000-, 2000-, and 3000-hour data. The actual exposure conditions used in this evaluation are shown in Table 2-1.

FIGURE 2-1. SODIUM MASS TRANSFER LMFBR CORROSION MOCKUP LOOP 8

FIGURE 2-2. CORROSION CARTRIDGE ASSEMBLY SHOWING THE TYPICAL RING AND TUBE SPECIMENS

TABLE 2-1
RUNS 8-1 AND 8-2 OPERATING CONDITIONS

Run	Exposure Times, hours	Flow, gpm	T _s , °F	<u>E</u>	<u>F</u>	<u>G</u>	<u>H</u>	<u>J</u> (out)	<u>M</u> (in)	<u>N</u>	<u>R</u>	<u>B</u>	<u>C</u>
8-1	1042	3.1	~275**	1332	1307	1289	1286	1137	884	-	-	778	-
	1995	3.0	~275	1320	1302	1300	1298	1132	873	-	-	778	-
	3037*	3.1	~275	1323	1304	1296	1296	1133	876	801	790	778	794
8-2	1083	5.9	270	1304	1309	1295	1290	1201	1045	965	913	876	885
	1867	5.7	293	1303	1308	1294	1289	1186	1031	964	923	886	884
	2950*	5.8	286	1303	1308	1295	1290	1192	1035	964	919	882	884

*The flow and temperature values for this time point are actually the averages of the other two time points.

**The loop was cold trapped during the entire run at 270°—280°F. In view of the uncertainty in interpreting the oxide plugging break, which appeared to yield exceptionally low values for T_s, the oxide value corresponding to the average cold trap temperature (~275°F) is probably the most realistic.

2.1.2.2 Test Results

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

After exposure, the sample cartridges are removed to an inert atmosphere (argon) chamber and transferred to an inert atmosphere glove box, where the samples are removed from the cartridges. The samples are then cleaned (reaction of sodium with alcohol and water), dried, and weighed to determine weight changes. As stated previously, the samples are designated tubes and rings. The tube samples have an inside diameter surface exposure with the outside diameter surface forming a crevice with the cartridge itself, Figure 2-2. The ring samples have an outside diameter surface exposure with the inside surface

forming a crevice with the mandrel, Figure 2-2. The weight change data (mg) is converted to standard corrosion terminology (mg/dm²) using only the surface area exposed to the flowing sodium, tube inside diameter, and ring outside diameter. The weight change results, loop position, and velocity are shown for runs 8-1 and 8-2 in Tables 2-2 and 2-3. Sample data from sections C and E were not analyzed since exposure conditions were complicated by operational problems. Tube samples in section E were exposed during runs 8-1P, an initial seasoning run, and 8-1. Difficulties were experienced in removing samples from sections C and E because loop design prohibited adequate gas blanketing of preheated samples and over heating resulted. Also, ring samples in these sections were located on heated mandrels with close ring-heater tolerances that interfered with removal causing damage (scraping) of the samples and error in weight change data. Partial examinations of samples from these sections will be attempted prior to completing the Loop 8 test series.

TABLE 2-2

WEIGHT CHANGE, LOOP POSITION, AND VELOCITY OF RUN 8-1

Time (hours)	Test Zone	Weight Change (mg/dm ²)		Velocity (ft/sec)	Downstream (L/D)
		Tube	Ring		
1995	FA*	-133	-	3.1	167.7**
		-129	-		↓
		-109	-		
		-104	-		
		-101	-		
		-114	-		
		-115	-		187.3
	FB	-131	-102	↓	191.8
		-114	-86		↓
		-99	-87		
		-106	-81		
		-99	-82		
		-110	-79		
		-109	-83		211.4
	FC	-210	-163	14.6	232.8
		-194	-152		↓
		-181	-139		
		-170	-130		
		-167	-126		
		-158	-116		
		-148	-109		355.8
	FD	-180	-138	↓	377.3
		-169	-123		↓
		-134	-118		
		-137	-109		
		-136	-108		
		-144	-94		
		-139	-90		500.3
	FE	-88	-72	3.1	504.0
		-74	-68		↓
		-66	-64		
		-60	-46		
		-72	-50		
		-78	-		
		-78	-		523.6
	FF	-81	-65	↓	527.3
		-81	-71		↓
		-69	-77		
		-78	-61		
		-80	-63		
		-132	-61		
		-108	-52		546.9
	GA	-93	-	1.2	559.6
		-82	-		↓
		-77	-		
		-72	-		
		-66	-		
		-64	-		
		-62	-		
	GB	-89	-		565.7
		-76	-	↓	568.4
		-79	-		↓
		-73	-		
		-67	-		
		-70	-		
		-76	-		
		-67	-		
		-64	-	1.2	574.5

TABLE 2-2 (Continued)

Time (hours)	Test Zone	Weight Change (mg/dm ²)		Velocity (ft/sec)	Downstream (L/D)	
		Tube	Ring			
1995	HA	-56	-	0.7 ↓	581.7	
		-42	-			
		-44	-			
		-38	-			
		-39	-			
		-51	-			
		-54	-		586.2	
	HB	-58	-		589.2	
		-40	-			
		-41	-			
		-41	-			
		-35	-			
		-41	-			
		-42	-		594.0	
	JA	-18	-		1.9	609.8
		-7	-			
		-14	-			
	***		-7		-	
1042	ME	3	-	3.2 ↓	7.4	
		6	-		9.8	
		-	-			
		12	-		228.5	
		26	-			
		17	-			
		30	-			
		22	-			
		15	-			
		23	-		236.0	
	FA	-77	-60		3.2	167.7
		-66	-39			
		-74	-54			
		-68	-46			
		-64	-37			
		-58	-			
		-74	-44			187.3
	FC	-112	-		15.0	232.8
		-96	-			
		-97	-46			
		-84	-49			
-88		-41				
-80		-51				
-83		-47	355.8			
FE	-61	-43	3.2	504.0		
	-45	-37				
	-47	-13				
	-48	-37				
	-46	-40				
	-55	-35				
	-54	-32		523.6		
JA	-10	-	2.0	609.8		
	-1	-				
	-12	-				
	-7	-				
	-2	-		614.9		
		6	-	↓	8.6	
		4	-		9.8	

TABLE 2-2 (Continued)

Time (Hours)	Test Zone	Weight Change (mg/dm ²)		Velocity (ft/sec)	Downstream (L/D)
		Tube	Ring		
1042	ME	36	-	2.0	228.5
		22	-	↓	↓
		23	-	↓	↓
		17	-	↓	↓
		18	-	↓	↓
		18	-	↓	↓
		16	-	↓	236.0
3037	FB	-163	-134	3.2	191.8
		-138	-106	↓	↓
		-119	-110	↓	↓
		-123	-104	↓	↓
		-118	-106	↓	↓
		-132	-104	↓	↓
		-134	-104	↓	211.4
	FD	-187	-170	15.0	377.3
		-209	-121	↓	↓
		-168	-140	↓	↓
		-162	-124	↓	↓
		-160	-128	↓	↓
		-169	-70	↓	↓
		-166	-112	↓	500.3
	FF	-105	-84	3.2	527.3
		-104	-88	↓	↓
		-107	-95	↓	↓
		-101	-77	↓	↓
		-100	-80	↓	↓
		-155	-57	↓	↓
		-126	-67	↓	546.9
	GA	-108	-	1.3	559.6
		-97	-	↓	↓
		-90	-	↓	↓
		-84	-	↓	↓
		-80	-	↓	↓
		-92	-	↓	↓
		-80	-	↓	↓
	GB	-104	-	↓	565.7
		-90	-	↓	568.4
		-91	-	↓	↓
		-85	-	↓	↓
		-78	-	↓	↓
		-78	-	↓	↓
		-89	-	↓	↓
	HA	-74	-	↓	↓
		-76	-	↓	574.5
		-67	-	0.7	581.7
		-51	-	↓	↓
		-46	-	↓	↓
		-45	-	↓	↓
		-44	-	↓	↓
	HB	-	-	↓	↓
		-57	-	↓	586.2
		-64	-	↓	589.2
		-50	-	↓	↓
		-50	-	↓	↓
		-46	-	↓	↓
		-42	-	↓	↓
		-47	-	↓	↓
		-51	-	↓	594.0

TABLE 2-2 (Continued)

Time (hours)	Test Zone	Weight Change (mg/dm ²)		Velocity (ft/sec)	Downstream (L/D)
		Tube	Ring		
3037	JB	46	-	2.0	13.5
		42	-		↓
		31	-		
		26	-		
		39	-		
		61	-		
	JC	52	-		20.9
		83	-		24.5
		77	-		↓
		58	-		
		50	-		
		55	-		
		77	-		
		75	-		32.0
	JD	119	-		35.6
		114	-		↓
		80	-		
		68	-		
		74	-		
		86	-		
	JE	76	-		43.1
		136	-		47.8
		121	-		↓
		88	-		
		80	-		
		80	-		
		107	-		
		92	-		55.2
	MA	50	-		185.2
		286	-		↓
		112	-		
		71	-		
		75	-		
		81	-		
	MB	80	-		192.7
		28	-		196.3
		123	-		↓
		102	-		
		83	-		
		80	-		
	MC	83	-		
		84	-		202.7
		178	-		206.4
		-	-		↓
		112	-		
		99	-		
	MD	79	-		
		73	-		
		91	-		213.8
		198	-		217.4
		132	-		↓
		69	-		
		75	-		
		64	-		
		73	-		↓
		66	-		224.9

TABLE 2-2 (Continued)

Time (hours)	Test Zone	Weight Change (mg/dm ²)		Velocity (ft/sec)	Downstream (L/D)
		Tube	Ring		
3037	NA	26	18	2.5	244.9
		47	20		
		28	21		
		29	20		
		23	21		
		38	18		
		31	20		273.6
	NB	21	20	8.2	295.7
		27	22		
		20	21		
		19	19		
		18	19		
		27	20		
		22	20		397.5
	NC	37	21	2.5	418.6
		27	14		
		27	25		
		31	28		
		27	27		
		31	24		
		28	25		447.3
	RA	14	-	0.7	525.7
		13	-		
		16	-		
		14	-		
		14	-		
		16	-		
		11	-		530.2
	RB	14	-		532.5
		17	-		
		12	-		
		11	-		
		12	-		
		15	-		
		13	-		537.0
	RC	-	-		539.4
		16	-		
		13	-		
		11	-		
		11	-		
		11	-		
		12	-		543.8
	BA	-	-		992.3
		11	-		
		9	-		
		7	-		
		8	-		
		10	-		996.8
		6	-		

*The first letter represents the test zone as shown in Figure 2-1. The second letter represents the cartridge, i. e., A is first or upstream cartridge.

**The hot-leg downstream calculation starts at the outlet of Zone C (first heater).

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

TABLE 2-3

WEIGHT CHANGE, LOOP POSITION, AND VELOCITY OF RUN 8-2

Time (hours)	Test Zone	Weight Change (mg/dm ²)		Velocity (ft/sec)	Downstream (L/D)
		Tube	Ring		
1083	FA	-102	-90	6.0 ↓	167.7 ↓
		-86	-83		
		-94	-82		
		-93	-72		
		-82	-76		
		-92	-68		
		-92	-68		
	FC	-152	-98	28.6 ↓	187.3 ↓
		-138	-85		
		-135	-79		
		-120	-80		
		-123	-		
		-107	-76		
		-106	-74		
	FE	-83	-62	6.0 ↓	232.8 ↓
		-67	-64		
		-64	-56		
		-71	-50		
		-64	-53		
		-63	-		
		-61	-		
	JA	-28	-	3.7 ↓	355.8 ↓
		-23	-		
		-28	-		
		-23	-		
		-26	-		
		-20	-		
		-15	-		
	ME	72	-	↓	523.6 ↓
		39	-		
		34	-		
		31	-		
		32	-		
		34	-		
		29	-		
1867	FA	-186	-174	5.8 ↓	609.8 ↓
		-157	-140		
		-145	-138		
		-140	-139		
		-138	-136		
		-139	-143		
		-185	-		
	FC	-301	-247	27.6 ↓	617.4 ↓
		-315	-236		
		-314	-199		
		-263	-180		
		-296	-177		
		-259	-163		
		-252	-157		
	FE	-115	-102	5.8 ↓	228.5 ↓
		-112	-94		
		-92	-93		
		-95	-93		
		-91	-88		
		-89	-89		
		-91	-96		
				↓	236.0 ↓
				↓	167.7 ↓
				↓	504.0 ↓
				↓	523.6 ↓

TABLE 2-3 (Continued)

Time (hours)	Test Zone	Weight Change (mg/dm ²)		Velocity (ft/sec)	Downstream (L/D)
		Tube	Ring		
1867	JA	-15	-	3.6	609.8
		-20	-		
		-16	-		
		-15	-		
		-12	-		
		-4	-		
		-3	-		
	ME	107	-	↓	617.4
		71	-		↓
		63	-		
		60	-		
		56	-		
		55	-		
		59	-		
2950	FB	-248	-205	5.9	191.8
		-210	-		
		-191	-173		
		-181	-161		
		-182	-158		
		-193	-160		
		-201	-158		
					211.4
	FD	-358	-227	28.1	377.3
		-322	-204		
		-293	-196		
		-287	-180		
		-253	-176		
		-251	-170		
		-280	-153		
					500.3
	FF	-170	-125	5.9	527.3
		-160	-132		
		-137	-116		
		-136	-126		
		-126	-115		
		-130	-113		
		-143	-108		
					546.9
	GA	-144	-	2.4	559.6
		-142	-		
		-137	-		
		-138	-		
		-132	-		
		-146	-		
		-136	-		
		-136	-		
	GB	-133	-	↓	565.7
		-132	-		↓
		-130	-		
		-136	-		
		-129	-		
		-120	-		
		-136	-		
		-133	-		
	HA	-131	-	1.3	581.7
		-96	-		
		-96	-		
		-89	-		
		-89	-		
		-85	-		
		-89	-		
					586.2

TABLE 2-3 (Continued)

Time (hours)	Test Zone	Weight Change (mg/dm ²)		Velocity (ft/sec)	Downstream (L/D)
		Tube	Ring		
2950	HB	-101	-	1.3	589.2
		-92	-		
		-79	-		
		-80	-		
		-79	-		
		-82	-		
		-86	-		594.0
	JB	10	-	3.6	619.8
		-6	-		
		-4	-		622.2
	JC	6	-		17.2
		6	-		
		13	-		
		11	-		20.9
		59	-		24.5
		37	-		
		32	-		
		35	-		
		33	-		
		37	-		
		39	-		32.0
	JD	75	-		35.6
		59	-		
		64	-		
		61	-		
		60	-		
		61	-		
	JE	128	-		43.1
		134	-		47.8
		76	-		
		64	-		
		63	-		
		68	-		
	MA	61	-		55.2
		58	-		
		563	-		185.2
		415	-		
		302	-		
		268	-		
	MB	219	-		
		203	-		
		231	-		192.7
		620	-		196.3
		304	-		
		226	-		
	MC	196	-		
		175	-		
		152	-		
		158	-		202.7
		472	-		206.4
		153	-		
	MD	140	-		
		74	-		
		119	-		
		106	-		
		126	-		213.8
		394	-		217.4
		191	-		
		123	-		
		109	-		
		108	-		
		99	-		
		108	-		224.9

TABLE 2-3 (Continued)

Time (hours)	Test Zone	Weight Change (mg/dm ²)		Velocity (ft/sec)	Downstream (L/D)
		Tube	Ring		
2950	NA	102	72	4.6	244.9
		83	64		
		80	62		
		78	62		
		79	54		
		73	64		
		70	60		
	NB	270	64	15.3	295.7
		243	166		
		148	109		
		137	76		
		106	78		
		88	73		
		86	73		
	NC	39	19	4.6	418.6
		32	19		
		30	18		
		30	19		
		29	17		
		28	17		
		28	20		
	RA	17	-	1.3	525.7
		12	-		
		10	-		
		11	-		
		11	-		
		10	-		
		14	-		
	RB	14	-		530.2
		13	-		
		9	-		
		8	-		
		8	-		
		8	-		
		8	-		
	RC	13	-		537.0
		11	-		
		13	-		
		13	-		
		11	-		
		9	-		
		10	-		
	BA	9	-		543.8
		5	-		
		4	-		
		2	-		
		3	-		
		2	-		
		3	-		
					992.3
					996.8

After weighing, the samples are sectioned for x-ray fluorescence (XRF) analysis of both the outside and inside diameter surfaces, and metallography. The plots of weight change and corresponding XRF versus total loop position downstream are shown in Fig-

ures 2-3 and 2-4. Plots of the crevice-side surface XRF versus loop position are shown in Figures 2-5 and 2-6. The data indicated essentially no movement (corrosion or deposition) of molybdenum. This agrees with the Phase I results where Mo was found to be relatively stable.

The visual appearance of the samples after sodium exposure is shown in Figure 2-7. The hot-leg samples, Zones F, G, and H, have a bright matte finish. The coolers and cold-leg samples, Zones J, M, N, R, and B, are discolored with the discoloration increasing with the weight gain.

The typical microstructural effects through the loop for runs 8-1 and 8-2 are shown in Figures 2-8 and 2-9, respectively.

2.1.2.3 Discussion

The precision and reproducibility of the mass transfer studies in the Phase I work of PA-15 were not sufficient to establish geometry effects unequivocally. Runs 8-1 and 8-2 were therefore primarily concerned with the study of geometry effects. The isothermal hot-leg, primarily Zone F, of runs 8-1 and 8-2 is of special interest in evaluating the effects of velocity and downstream. The downstream effect of decreasing corrosion with position change in an isothermal region can be seen for both runs in Figures 2-3 and 2-4 and Tables 2-2 and 2-3. The increase of corrosion with

increasing velocity can be seen in the data for each run and in the comparison of run 8-1 data to that of run 8-2.

The downstream position effect can be interpreted in the following manner. Mass transfer (corrosion and deposition) is a result of the relative activities of the Fe, Cr, Ni, Mn, Mo, and C in the solid metal and the liquid sodium. The difference in activities sets up a corrosion potential. As the sodium flows downstream, it contains more corrosion products. The "loading" of the sodium results in the reduction of the corrosion potential and correspondingly the corrosion rate. If the isothermal hot-leg were infinite in length, the sodium would eventually become saturated and corrosion at the infinite position would stop. The effect of velocity increasing the corrosion rate is also associated with relative activities and corrosion potential. The effect is one of a change in concentration profiles of the solutes in the sodium boundary layer immediately adjacent to the metal. The velocities of runs 8-1 and 8-2 are in the turbulent flow range. As the velocity increases, the degree of turbulence increases producing the effect shown in the sketch below:

FIGURE 2-3. WEIGHT CHANGE AND EXPOSED SURFACE X-RAY FLUORESCENCE DATA VERSUS LOOP POSITION FOR THE TUBE SPECIMENS FROM RUN 8-1 AFTER ~3000 HOURS EXPOSURE

FIGURE 2-4. WEIGHT CHANGE AND EXPOSED SURFACE X-RAY FLUORESCENCE DATA VERSUS LOOP POSITION FOR THE TUBE SPECIMENS FROM RUN 8-2 AFTER ~3000 HOURS EXPOSURE

FIGURE 2-5. CREVICE SURFACE X-RAY FLUORESCENCE DATA VERSUS LOOP POSITION FOR THE TUBE SPECIMENS FROM RUN 8-1 AFTER ~3000 HOURS EXPOSURE

FIGURE 2-6. CREVICE SURFACE X-RAY FLUORESCENCE DATA VERSUS LOOP POSITION FOR THE TUBE SPECIMENS (RING SPECIMENS INCLUDED IN ZONE F) FROM RUN 8-2 AFTER ~3000 HOURS EXPOSURE

FIGURE 2-7. RUN 8-1 TUBE SPECIMENS SHOWING THE SURFACE DISCOLORATION AFTER SODIUM EXPOSURE

The velocity effect is particularly noticeable at high corrosion potentials (upstream). As the sodium becomes saturated, the effect continually decreases because of the lower corrosion potentials involved. The velocity effect as shown above is based on the assumption that diffusion through the liquid is the rate controlling step. If, however, either bulk diffusion in the solid or diffusion across the intermediate ferritic phase is the rate controlling step, the corrosion rate will be independent of sodium velocity. This may occur at high velocities.

An additional effect, although not readily discernible on the basis of runs 8-1 and 8-2, is that of mass flow rate on corrosion rate. An increase in mass flow allows for a greater mass of sodium and, therefore, a greater sink for soluble elements and reaction products, to be available for reaction with the metal at a particular point and corrosion potential, thereby increasing the corrosion rate. Since the isothermal downstream term describes the saturation of the flowing sodium (saturation increases with downstream in a corrosion region), it is reasonable to conclude that the sensitivity of corrosion (also deposition) rate to loop position becomes more pronounced as the mass flow rate increases. Since velocity is dependent on mass flow rate for a constant geometry system, one might say that the important parameter when studying the geometry effects is the mass flow rate, with the corrosion (deposition) rate becoming more sensitive to geometric parameters as the mass flow rate is increased. A better insight into the effect of mass flow rate must await further investigation as more data are generated.

There is an additional geometry effect on corrosion, noted from the Loop 8 data, concerning the local downstream position in the separate cartridges. The corrosion rates are significantly higher at the entrance (first sample) to a cartridge, Tables 2-2 and 2-3, than on the other samples. This effect is not concerned with downstream position,

per se, but with the sodium mass flow and velocity characteristics (turbulence) on the corrosion potential in the boundary layer. There is a velocity increase as the sodium enters each cartridge. The entrance effect is dependent on the relative velocities, the higher the cartridge velocity (velocities between cartridges are fairly constant), the greater the weight change difference, although the relative weight change ratios are constant at ~ 1.5 . The above is particularly noticeable at high corrosion potentials (upstream position). Since the effect is a result of flow characteristics on localized corrosion potential, it diminishes with decrease in bulk corrosion potential much as the bulk velocity effect decreased.

The effects of downstream and velocity on corrosion rate as discussed above are necessarily oversimplified by treatment of the corrosion potential considerations as for a pure metal. In the multicomponent material such as austenitic stainless steel, the various components in the steel and in sodium can cause unknown shifts in the relative activities and the elemental solubilities in sodium which will affect the corrosion potential. Also, the form of the elements in the sodium, in solution or as particulates, will produce shifts. There is much experimental work needed before adequate handling of corrosion (deposition) mechanisms can be attempted.

The corrosion cartridges in Zone F consist of both tube and ring samples. There is basically good agreement between the samples, Tables 2-2 and 2-3, concerning the geometry effects discussed above. However, the ring samples have a consistently lower corrosion rate than the tube samples. This can be shown to be a result of using only the surface area exposed to flowing sodium for the corrosion calculation. It is known (discussed later) that corrosion also occurs on the crevice sides of the samples (amount dependent on velocity). If the total sample surface is used in the corrosion calculation, the values between tubes and rings approach each other.

FIGURE 2-8. TYPICAL MICROSTRUCTURAL EFFECTS OF THE TUBE SPECIMENS FROM RUN 8-1 AFTER ~3000 HOURS EXPOSURE

FIGURE 2-9. TYPICAL MICROSTRUCTURAL EFFECTS OF THE TUBE SPECIMENS FROM RUN 8-2 AFTER ~3000 HOURS EXPOSURE

The tubes are divided by an additional value which is larger than the original (outside versus inside diameter) and the rings by a value which is smaller (inside versus outside diameter). The use of only the surface exposed to flowing sodium results in a conservative estimate of the corrosion rates.

In the cooler sections (Zones J and M) of Loop 8, the corrosion/deposition patterns are studied as a function of temperature (downstream to a lesser extent) with the velocity remaining constant. In both runs 8-1 and 8-2, corrosion occurred into the first cartridge of Zone J, indicating the sodium was not saturated at 1300°F under the conditions for these runs. As the sodium cooled further, it reached its saturation point (the saturation temperature for the various elements in solution varies) and selective deposition occurred. The rate of deposition increased with temperature decrease as a result of supersaturation. Deposition also occurred in the isothermal cold-leg sections, but to a much lesser degree. The reasons for this are twofold, (1) the sodium has been substantially unloaded by the time the isothermal cold-leg sections are reached, and (2) the driving-force for deposition is very low in an isothermal area since an activation energy is needed. (If the sodium is saturated at a particular temperature, some nucleation site is necessary for deposition.) The amount of deposition occurring in the isothermal cold-leg sections is dependent on downstream position and velocity much as corrosion is dependent in the isothermal hot-leg sections. The causes of these effects are the reverse of the hot-leg, (1) deposition potential decreasing with downstream position as a result of a smaller amount of corrosion products in the sodium, and (2) increasing the velocity increases the degree of turbulence, resulting in more corrosion products contacting the pipe or sample wall and precipitating. Entrance effects are noted for deposition areas, much as for corrosion areas,

and the magnitude of the effects decreases with decreasing deposition potential. The effect is analogous to the velocity effect on flow characteristics described above.

The x-ray fluorescence analysis provides a better insight into the relative corrosion and deposition characteristics of the various components. In Figures 2-3 and 2-4, it can be seen that chromium, nickel, and manganese are selectively leached from the austenitic stainless steel in the corrosion areas of the loops. The thought here is that the selective leaching process occurs during an initial nonequilibrium corrosion rate stage and that once steady-state corrosion rates are achieved, stoichiometric dissolution of the resultant surface alloy composition occurs. This relationship is shown in the sketch below.

At conditions producing low corrosion rates, as in A above, the time to reach steady-state conditions is longer, the driving force for selective leaching is reduced, and the alloy perturbation at the surface is shallow. At conditions producing higher corrosion rates (B, C, D), the curve approaches linearity, but never actually reaches it because of the leaching process

which occurs initially over a relatively short period of time. Because of the greater driving force for selective leaching associated with higher corrosion rates, the amount of alloy surface perturbation increases, 8-1 versus 8-2 (Figures 2-3 and 2-4), and for the case of austenitic stainless steels, a transformation to a ferritic phase can occur.

The fluorescence results show that while the bulk sodium is apparently not fully saturated, it is saturated with chromium which precipitates before the first cooler is reached. The source of the activation energy for the chromium deposition is the slight degree of cooling that occurs in the H. zone. The bulk corrosion into the first cartridge of Zone J is then primarily a result of nickel and manganese corrosion. At some temperature below 1300°F, nickel and manganese achieve saturation in the sodium and further cooling produces precipitation of these elements. Two observations of interest concerning the corrosion are, (1) as chromium precipitates from solution, it appears that the activity of nickel in sodium decreases, resulting in an increase in nickel corrosion, and (2) as the corrosion rate increases, 8-1 versus 8-2, the corrosion potential for nickel increases, resulting in increased nickel corrosion over and above that expected just on the basis of the increased corrosion rate.

The fluorescence results through the coolers (Zones J and M) and the isothermal cold-leg sections (Zones N, R, and B) are difficult to interpret. The major difficulty comes from the absence of sample coverage in the two coolers between J and M, and the temperature gaps between run 8-1 and 8-2. The entire Zone M cooler and isothermal cold-leg from run 8-2 lies in the nonsampled areas of run 8-1. Conversely, the Zone M cooler and isothermal cold-leg from run 8-1 lies below the T_{min} region of run 8-2. The major observation to be made is that, although deposition is the mode of weight change

measured, there is apparently an interchange of corrosion and precipitation kinetics between the various elements. The apparent first-order driving force for deposition is the solubility versus temperature consideration (saturation effects) of the various elements. A complicating second-order driving force is the effect of the precipitation of one element on the solubilities of the other elements.

The crevice surface fluorescence data show the same general trends as the surfaces exposed to flowing sodium, Figures 2-5 and 2-6. However, there is a shift on the elemental change axis. The crevice surface corrosion is directly dependent on velocity as would be expected. At high bulk stream velocities, fairly representative corrosion rates are indicated. At lower bulk stream velocities, deposition (amount not known, but assumed to be small) is indicated. In the cooler and cold-leg sections, velocity promotes deposition. The major observation here is that corrosion and deposition occur on the crevice surface and can be substantial, depending on bulk stream velocity. The major dependence on velocity, however, makes it extremely difficult to relate the crevice surface contribution to the total measured weight change.

The effects of mass transfer on the microstructure, primarily during the elemental leaching period as mentioned previously, can be observed metallographically. The formation of a surface phase, in the case of austenitic stainless steels, a ferritic phase, can be seen for the corrosion Zones F, G, and H, in Figures 2-8 and 2-9. The ferritic phase is primarily associated with the grain boundaries due to the higher diffusion rates of the preferentially leached elements at these areas through an annealed structure. At higher corrosion rates, a surface layer of the ferritic phase will form. The phase change in the Type 304 stainless steel, Zones G and H, has spread out substantially into the grains and has a feathery appearance. Apparently, the compositional change was not sufficient

to cause the allotropic phase transformation of γ to α , and a transformation of γ to α' (strain-induced martensite) occurred from the vibratory polishing. It is known that Type 304 stainless steel is metastable and that slight degrees of cold work can force this transformation without a compositional change.

The formation of sigma phase, seen in Figures 2-8 and 2-9, for the Type 316 stainless steel in Zones F and J, and to a much lesser degree for Type 304 stainless steel in Zones G and H is an indication of decarburization in addition to the removal (leaching) of Ni and Mn. It has been shown⁽¹⁾ that lowering the carbon content (also Ni and Mn) will promote sigma formation. The substantial decrease in Cr content acts in reverse. In general, the loss of Cr counteracts the loss of Ni and Mn, making the loss of C the major driving force for sigma formation. The Type 316 and 304 stainless steel under test will not form sigma phase upon heating in air at 1300°F for 3000 hours. The driving force for carbon movement is the same as for the other elements, namely, concentration gradient between metal and the sodium in contact with the metal. The same downstream (sodium saturation) effect will exist for carbon movement also. For the decarburization process to continue, a carbon "sink" is necessary much as deposition perpetuates the general mass transfer process. It will be shown that the carbon "sink" is the cold-leg, primarily the crevice surfaces.

The depth of sigma phase formation in Type 316 stainless steel decreases with downstream position and temperature, run 8-1, Zone F (1300°F), inlet Zone J (1250°F), and run 8-2, Zone F (1300°F), outlet Zone J (1200°F). The effect of downstream was explained above. The continued decarburization with decreasing temperature (although at a lower rate) indicates that the sodium is not saturated with carbon much as the case

for nickel and manganese. The degree of sigma formation (decarburization) in Type 304 stainless steel is very slight. However, it has been shown^(2, 3) that carbon diffuses faster in Type 304 stainless steel than in Type 316 stainless steel. Therefore, the effect is one of downstream position (lower corrosion potential). Further, it has been shown⁽⁴⁾ that given the same corrosion potential, Type 304 stainless steel will form a greater amount of sigma phase than Type 316 stainless steel.

The absence of carbide decoration on grain boundaries and slip markings and a nearly continuous layer of sigma phase on the crevice surface of Zones F, G, and H, (Figure 2-8), indicates decarburization in these areas. Conversely, the heavy grain boundary and slip marking decoration on the crevice surface of Zone J, Figure 2-8, reveals one area of the carbon "sink." Another area can be seen for run 8-2, Zone N, Figure 2-9, where in cold-leg regions, if the surface exposed to flowing sodium has been machined (slip markings), the disturbed metal surface will contain heavy concentrations of precipitated carbides. It takes substantial carburization of an annealed structure for metallographic observation (~0.31 wt %).⁽⁵⁾ However, with numerous sites available for carbide precipitation, a cold worked structure reveals carburization readily at lower carbon levels.

It has been found that no matter what the calculated thickness of deposit (from weight change data), metallography always reveals approximately the same thickness. Recently, it has been found that by using a completely dry metallographic preparation technique, the appropriate amounts of deposit can be retained on a sample. However the dry techniques do not allow for a metallographic specimen with good viewing characteristics (scratched)

since the final polishing procedures are wet techniques. There are two observations which can be made about the nature of the deposits (1) the high chromium deposition areas (Zone J) have a different appearance than the high nickel deposition areas (Zone M), and (2) the high chromium deposition areas (small amounts of deposit) metallographically show a greater thickness of retained deposit than the high nickel deposition areas (large amounts of deposit). The second observation in conjunction with the inability to retain the deposits by wet metallographic techniques suggests that the porosity of the deposit and its tenacity (adherence) are a function of the major element present, chromium or nickel.

2.1.2.4 Summary and Conclusions

Following is a restatement of current views of the basic phenomena of corrosion and deposition:

- a. The corrosion rate of austenitic stainless steels in 1300°F sodium is decreased with downstream position. The effect is one of sodium saturation and the resulting decrease of corrosion potential.
- b. The corrosion rate of austenitic stainless steels in 1300°F sodium increases with increasing velocity. The effect is one of sodium flow characteristics (turbulence) increasing the corrosion potential in the metal surface/sodium boundary layer.
- c. A localized effect of flow characteristics (degree of turbulence) known as "entrance effect" of a corrosion cartridge, has been observed, which increases the corrosion rate locally for an austenitic stainless steel exposed in 1300°F sodium.
- d. The corrosion process in 1300°F sodium is comprised of two rate controlling steps (1) selective leaching of chromium, nickel, and manganese, and (2) stoichiometric dissolution of the resulting surface alloy composition with diffusion of Cr, Ni, and Mn in the iron-rich surface layer. The factors affecting corrosion rate, i. e., temperature, velocity, etc., determine the time dependence and magnitude of the first step.
- e. A corrosion rate comparison of Types 304 and 316 stainless steels could not be made because of the extreme differences in downstream position and velocity.
- f. The deposition rate in sodium is dependent on downstream position, velocity, and "entrance effects" for analogous reasons to the corrosion rate dependency. However, the primary driving force for deposition is a ΔT , but not to be construed to any dependency on magnitude of ΔT of the system. The ΔT effect is simply the relative change in solubilities of the corrosion products in sodium with decreasing temperature.
- g. The temperature range of deposition of individual elements is dependent on the relative solubilities of the elements in sodium and amount of each element entering the sodium during the corrosion process. For example, chromium is depositing in a region where nickel and manganese are corroding, indicating saturation of chromium in sodium. The nickel and manganese deposit after the temperature is lowered to the point of their saturation in sodium.
- h. There is direct evidence in a monometallic austenitic system of decarburization in the 1200°—1300°F

sodium temperature range and carburization at lower sodium temperatures. The decarburization is analogous to the selective leaching process of the major alloying elements.

2.2 SUBTASK 1.3—DEVELOPMENT OF HIGH HEAT FLUX HEATERS

2.2.1 Electron Bombardment Heater

The objective of this task is the development of heaters capable of operating for times up to 3000 hours in sodium at temperatures up to 1300°F to determine effects of high heat flux typical of LMFBR fuel cladding ($0.5 - 1 \times 10^6$ Btu/h-ft²). The work is in direct support of Subtasks 1.1 and 1.2. The heater development will facilitate the study of the possible effects of heat transfer on corrosion and mass transfer. The electron bombardment heater (EB heater) is one of the concepts presently under test.

The EB heater has recently undergone two successful tests. In the first test, the anode material was Type 316 stainless steel. The heater operated in sodium for ~3700 hours, of which 1725 hours were at average heat fluxes $\geq 0.8 \times 10^6$ Btu/h-ft² (~20 kW) and corresponding local peak heat fluxes of 3×10^6 Btu/h-ft². During the 1725 hours, the sodium temperature averaged 1000°F at the inlet to the heater and 1200°F at the outlet, although the outlet sodium temperature reached 1300°F at times. The anode was removed for metallographic examination and the feasibility of attaching a new anode to the cathode assembly was successfully demonstrated.

The replacement anode was Type 304 stainless steel. It was purposely operated above rating—40 hours at 24 kW and 35 minutes at 27 kW. The operation was terminated by a cathode-to-anode short.

2.2.2 Conclusions

- a. The corrosion-rate of Type 316 stainless steel in sodium at 1200°—1300°F is accelerated under heat transfer conditions when compared to equivalent isothermal conditions.
- b. The formation of a ferritic surface phase (layer), selective removal of Cr, Ni, and Mn, is dependent on the initial corrosion/elemental removal rate. For equivalent corrosion-rates, the surface layer formation will be similar whether formed under heat transfer or isothermal conditions.
- c. The heat flux, for the present cathode emitter design, is concentrated at the ends of the emitting zone.

2.2.3 Post-Test Examination Results

The Type 316 and 304 stainless steel anodes were both weld drawn, annealed, and surface ground 0.394-inch outside diameter $\times$ 0.027-inch wall tubing. The heater assemblies are held in place by a previously described⁽⁶⁾ support sleeve.

The cathode was Mo-Re with a 10-inch emitting zone (heated length) of impregnated (barium, calcium, and aluminum) tungsten.

2.2.3.1 Type 316 Stainless Steel Anode

The Type 316 stainless steel anode was visually examined and observed to be slightly bowed, with no surface indentations other than those made by the set screws in the support sleeve. The anode had the matted surface finish typical of stainless steel after sodium corrosion.

The anode was sectioned for metallography and x-ray examination as shown in Figure 2-10. The metallographic examination

revealed a structural variation, both on the outside diameter surface and in bulk structure, along the length of the anode, Figures 2-11 through 2-14. The outside diameter surface microstructure varied from a decarburized layer at the sodium inlet (specimens 1, 2, and 3), Figure 2-11, to the formation and growth of a ferritic surface phase as the temperature increased (specimens 4, 5, 6, 7, and 8), Figures 2-12 and 2-13. The surface layer decreased substantially downstream of the 10-inch emitter zone, Figure 2-14. The x-ray fluorescence (XRF) analysis of the outside and inside diameter surfaces is shown in Table 2-4 and Figure 2-15. The growth of the surface layer is also shown in Figure 2-15. The change in general microstructure from sodium inlet to outlet indicated an increase in the bulk sodium temperature. An attempt is made to plot the outside diameter surface temperature profile, Figure 2-16, based on sodium exposed Type 316 stainless steel microstructures at known temperatures.

The examination of the cross section revealed that only metallographic specimens No. 2, 3, 7, and 8 had a major structural change from the outside to the inside diameter surface, Figures 2-17 and 2-18. These specimens were at the ends of the emitter zone. The variation existed around the entire circumference. Accompanying the structural variation was an inside diameter surface deposit, Figure 2-19, found only on the four specimens listed above. The x-ray diffraction (XRD) analysis showed that deposit to be primarily Ni_2Si with a trace of tungsten at the highest temperature end of the emitter zone, Table 2-4. An estimate of the inside diameter surface temperature profile is shown in Figure 2-16.

2.2.3.2 Type 304 Stainless Steel Anode

The visual examination of the Type 304 stainless steel anode showed collapse of the anode against the cathode at both ends of the

10-inch emitter zone causing a short-circuit and termination of the test. There were no other indentations except for those caused by the support sleeve. The matted surface finish was observed as for the Type 316 stainless steel anode.

The anode was sectioned for metallography and x-ray examination. The metallographic examination revealed a general structural variation from sodium inlet to outlet. The outside diameter surface showed an increase in decarburization along the length of the emitter zone, followed by a decrease immediately after the zone. There was no indication of a compositionally disturbed surface layer. The XRF analysis of the outside and inside diameter surfaces is shown in Table 2-5.

The examination of the cross section again showed a structural variation only at the ends of the emitter zone, Figure 2-20. The variation existed around the entire circumference. Because of the operation of the heater above rating, the inside diameter of the anode had reached $1900^{\circ}\text{--}2100^{\circ}\text{F}$, depending on the time interval, resulting in carbide re-solution and grain growth. In addition, the local overheating (thermal stresses) combined with mechanical restraint (support sleeve) caused a collapse with intergranular tearing of the anode at the inside diameter surface, Figure 2-20. The thermal stress in the $1900^{\circ}\text{--}2100^{\circ}\text{F}$ temperature range (3000—4000 psi) is $\geq$ the materials yield (3000—1500 psi) and tensile (4500—2500 psi) strength at these temperatures. Similar behavior, although a result of filament misalignment, was noted on a previous anode.⁽⁶⁾ There was an inside diameter surface deposit at the ends of the emitter zone, which was noted upon visual examination, but was not retained during metallographic preparation. The XRD analysis showed the deposit to be tungsten, Table 2-5.

FIGURE 2-10. SKETCH SHOWING THE AREAS OF METALLOGRAPHIC AND X-RAY EXAMINATION OF THE TYPE 316 STAINLESS STEEL ELECTRON BOMBARDMENT HEATER ANODE

GLYCEREGIA

500X

FIGURE 2-11. OUTSIDE DIAMETER SURFACE AND GENERAL MICROSTRUCTURE OF THE TYPE 316 STAINLESS STEEL ANODE-METALLOGRAPHIC SPECIMEN No. 1

GLYCEREGIA

500X

FIGURE 2-12. OUTSIDE DIAMETER SURFACE AND GENERAL MICROSTRUCTURE OF THE TYPE 316 STAINLESS STEEL ANODE-METALLOGRAPHIC SPECIMEN No. 5

GLYCEREGIA

500X

FIGURE 2-13. OUTSIDE DIAMETER SURFACE AND GENERAL MICROSTRUCTURE OF THE TYPE 316 STAINLESS STEEL ANODE-METALLOGRAPHIC SPECIMEN No. 8

GLYCEREGIA

500X

FIGURE 2-14. OUTSIDE DIAMETER SURFACE AND GENERAL MICROSTRUCTURE OF THE TYPE 316 STAINLESS STEEL ANODE-METALLOGRAPHIC SPECIMEN No. 9

FIGURE 2-15. SURFACE COMPOSITION CHANGE AND FERRITIC LAYER GROWTH OF THE TYPE 316 STAINLESS STEEL ANODE

FIGURE 2-16. PEAK SURFACE TEMPERATURE PROFILES OF THE TYPE 316 STAINLESS STEEL ANODE

TABLE 2-4

X-RAY ANALYSIS OF THE TYPE 316 STAINLESS STEEL
ELECTRON BOMBARDMENT HEATER ANODE

<u>Specimen</u>	<u>XRF</u>				<u>XRD</u>
	<u>Cr</u>	<u>Ni</u>	<u>Mn</u>	<u>Mo</u>	
Standard o. d.	15.9	11.5	1.55	2.00	-
Standard i. d.	16.2	11.8	1.55	2.38	-
No. 1 o. d.	13.6	11.6	2.02	2.31	-
No. 1 i. d.	15.2	11.0	2.35	2.49	γ , Ni ₂ Si
No. 2 o. d.	12.2	10.6	1.75	2.18	-
No. 2 i. d.	13.8	11.6	2.65	3.13	γ , Ni ₂ Si
No. 3 o. d.	12.4	8.0	1.59	2.43	-
No. 3 i. d.	15.4	12.1	2.29	2.84	γ , Ni ₂ Si
No. 4 o. d.	10.2	6.4	1.40	2.20	-
No. 4 i. d.	15.4	12.2	2.41	2.70	γ , Ni ₂ Si
No. 5 o. d.	10.4	6.2	1.45	2.28	-
No. 5 i. d.	15.3	12.0	2.35	2.68	γ , Ni ₂ Si
No. 6-7 o. d.	8.6	4.9	1.20	2.12	-
No. 6-7 i. d.	15.1	11.6	2.34	2.81	γ , Ni ₂ Si
No. 8 o. d.	6.3	3.2	0.85	2.11	-
No. 8 i. d.	13.8	12.5	2.42	5.84	γ , Ni ₂ Si, W
No. 9 o. d.	10.0	6.6	1.27	2.25	-
No. 9 i. d.	15.3	11.2	2.30	2.59	γ , Ni ₂ Si

TABLE 2-5
X-RAY ANALYSIS OF THE TYPE 304 STAINLESS STEEL
ELECTRON BOMBARDMENT HEATER ANODE

<u>Specimen</u>	<u>XRF</u>			<u>XRD</u>
	<u>Cr</u>	<u>Ni</u>	<u>Mn</u>	
Standard o. d.	17.8	8.9	1.70	-
i. d.	17.9	8.9	1.70	-
Inlet Emitter—No. 1 o. d.	17.5	8.8	1.09	-
i. d.	16.7	9.0	0.81	W
Inlet Emitter—No. 2 o. d.	17.9	8.9	1.60	No
i. d.	18.0	8.9	1.72	Pattern
Outlet Emitter No. 2 o. d.	17.8	8.8	1.41	-
i. d.	16.8	9.0	0.86	W

2.2.4 Discussion of Results

A preliminary discussion concerning corrosion rates in sodium and the variables affecting the rates is necessary. The primary variables affecting corrosion rate are temperature, velocity, loop position (downstream—sodium saturation effect), system geometry (flow patterns, etc.), and now, heat transfer. In general, increasing temperature and velocity increases the corrosion rate. The effect of increasing downstream position is to reduce the corrosion rate. The effect of system geometry is such that local disruption of the sodium flow (turbulence) increases the corrosion rate.

Recent corrosion data at 1300°F have shown corrosion rates that are approximately linear (reported under subtask 2.1). With this as a basis, a corrosion rate comparison can be made between isothermal and heat transfer conditions without regard to deline-

ation of nonequilibrium and steady-state rates as exist for parabolic, cubic, and logarithmic rate laws.

The examination of the EB heater anodes revealed several items of interest:

- a. The effect of heat transfer was to increase the corrosion-rate,
- b. The metal/sodium temperature increased along the heater, and
- c. The heat flux was concentrated at the ends of the 10-inch emitter zone.

The first and second observations were noted by the change in microstructure, both outside diameter surface and general, of the two anodes. The general structure indicated a metal, and consequently a sodium, rise in temperature along the emitting zone of the heater, Figures 2-11 through 2-13.

The structure also indicated that the metal/sodium temperature downstream of the emitter zone was essentially equal to the peak temperature in the zone, Figures 2-14 and 2-16. The Type 304 stainless steel anode did not show this. However, the exposure to high power levels (change in temperature profile) might have caused the different effect. The change in outside diameter surface structure of the Type 316 stainless steel anode showed the formation and growth of a ferritic surface phase along the emitter zone, Figures 2-11 through 2-13, and a substantial decrease in the surface layer immediately downstream of the zone, Figures 2-14 and 2-15. The general structure indicated the same or similar metal/sodium temperature for both locations. Therefore, the conclusion to be made is that the application of heat transfer increased the corrosion-rate of the EB heaters.

An additional observation can be made to substantiate the above conclusion. The isothermal hot-leg corrosion-rates of Type 316 stainless steel, experienced after 3000 hours at 1300°F (reported under subtask 1.1), produce a chromium and nickel elemental change of 2%—6% which is not sufficient to form a ferritic surface phase. This is similar to the results observed for metallographic specimen No. 9, Figure 2-15. However, when the isothermal corrosion-rates at 1300°F are increased by the use of impingement samples (deposition promoters), the chromium and nickel elemental change is 8%—12% and a ferritic surface phase ~0.2-mil thick was formed. This is similar to the results observed for metallographic specimen No. 8, Figure 2-15. These results show that if the corrosion rates under heat transfer and isothermal conditions are equivalent (by the adjustment of other parameters), the surface effects will be the same. Conversely, if the test parameters under heat transfer and isothermal conditions are equivalent, heat transfer will

increase the corrosion rate. Therefore, the formation of the ferritic surface phase is a result of the initial corrosion-rate and not a function of the amount of corrosion, per se. That is to say, at linear corrosion rates, the elemental concentration at the surface is established very rapidly and does not change with time or subsequent metal loss. Under constant test conditions, if a layer forms, it does not grow. If the layer is absent, it will not appear with additional corrosion. In general, if the rate of corrosion (nonstoichiometric removal of alloying elements) is low, the bulk diffusion rate of alloying elements to the corroding surface is sufficient to reduce the surface compositional gradient. If this occurs, a compositionally disturbed layer is not observed. The reverse is true at high rates of corrosion.

The above discussion leads to one additional observation, an estimate of the corrosion-rates under heat transfer conditions is possible by comparing the thickness of the surface layer formed to that formed under isothermal conditions. The one requirement is that isothermal corrosion-rates of sufficient magnitude be obtained to define the effect on the surface layer thickness. The use of a weighable (replaceable) anode could, of course, reduce the need for the above comparison. However, a replaceable anode may prove to be a difficult development problem.

The XRF of the Type 316 anode substantiated the nucleation and growth of a ferritic phase from anode positions 4 through 9, Table 2-4 and Figure 2-15. XRF and surface layer growth, Figure 2-15, also confirm the shape of the temperature profile, Figure 2-16. The Type 304 anode cannot be used to verify the above conclusion due to short-time exposure with lack of a compositionally disturbed layer. The outside diameter surface

degree) on the Type 304 stainless steel anode are a result of the cathode (tungsten) and related materials depositing on the anode, primarily in the areas of high heat flux. The XRD results have confirmed the deposition of tungsten in the high heat flux regions. The occurrence of Ni_2Si on the entire inside diameter surface of the Type 316 stainless steel heater is more of a mystery. It is possible that the Ni_2Si compound is a part of the braze alloy used to cap-off the tungsten section of the cathode.

2.2.5 M-EB-1-2 Heater Construction Details

Based on the data collected from the operation of E-EB-3-1 and E-EB-3-2, small changes were made in the construction of heater M-EB-1-2.* With reference to Figure 2-21, the changes made were as follows:

* In view of the fact that several heaters had been made and used, and that several more were being made for use in Loop 9, a numbering system was devised to designate individual heaters. The numbering system is as follows:

X - EB - X - X
 └───┬────────┘ Anode Number
 └───┬────────┘ Heater Number
 └──────────┘ Manufacturer's Initial
 (E = Eumac, M = Monolith)

Therefore, the third heater by Eimac, with the second anode in use, would be designated E-E3-3-2. The 6557-hour run terminated on January 13, 1968, was made with heater E-EB-3-1.

- a. The wires, Part 8, that hold the filament rod spacers in place were platinum on E-EB-3-1. They melted during a test to determine maximum power capability, allowing the spacers to drop to the bottom of the filament rod, causing malfunctioning of the filament circuit. Tantalum wire was used in the new heater because it has a higher melting point than platinum.
- b. The lower spacing insulator on the cathode rod, Part 3, was redesigned to make it unnecessary to machine a retaining notch in the cathode support tube. An insulator retainer was designed and installed at the bottom end of the anode assembly. These changes were made to make assembly easier and to strengthen the cathode assembly.
- c. The filament insulators used in the fabrication of E-EB-3-1 were made of Al_2O_3 and appeared to have operated satisfactorily; however, BeO insulators were used in the construction of M-EB-1-2 for both filament and cathode-to-anode, because of the superior high temperature characteristics of BeO .

2.2.5.1 Anode Material Change

The first anode material assigned to M-EB-1-1 was Type 304 stainless steel. A new anode, made of Type 316 stainless steel, was substituted pursuant to agreement with PNL and the heater designation changed to M-EB-1-2 to reflect the change.

2.2.5.2 Delivery of M-EB-1-2

EB heater M-EB-1-2 was installed in Loop 9 April 9, 1968. Cathode activation continued until the time of this report.

2.2.5.3 Instrumentation

A Mark 200 Brush recorder has been installed to record the operational data. The data being recorded are as follows:

Plate Voltage (E_p)

Plate Current (I_p)

Vacuum Pump Pressure (P)

Filament Current (I_f)

Sodium Inlet Temperature

Sodium Outlet Temperature

Six temperatures starting at bottom end of cathode, ending at upper end, spaced 2 inches apart. These eight temperatures are recorded on one recorder channel through the use of a thermocouple sequencer.

2.2.5.4 Computational Techniques

A computer code, using BASIC language, has been written and debugged, using the time-share computer facility. The code is based on the assumptions that there is no axial energy-transfer, and that the electron emission from the cathode is a function of cathode temperatures, and the electrons available to it from the plate current power supply. The heater power supply is such that cathode heating is primarily by means of the alternating current filament circuit, and secondarily, by flow to the direct plate current, which is fed from both ends of the cathode (see Figure 2-22).

GLYCEREGIA

o.d.

i.d.

150X

FIGURE 2-17. STRUCTURAL VARIATION ACROSS THE WALL OF THE TYPE 316 STAINLESS STEEL ANODE-METALLOGRAPHIC SPECIMENS No. 2 AND 3

GLYCEREGIA

o.d.

i.d.

150X

FIGURE 2-18. STRUCTURAL VARIATION ACROSS THE WALL OF THE TYPE 316 STAINLESS STEEL ANODE-METALLOGRAPHIC SPECIMENS No. 7 AND 8

UNETCHED

500X

FIGURE 2-19. TYPICAL INSIDE DIAMETER SURFACE DEPOSIT OF THE TYPE 316 STAINLESS STEEL ANODE AT THE ENDS OF THE EMITTER ZONE

o.d.

i.d.

HCl - H₂O₂

125X

FIGURE 2-20. STRUCTURAL VARIATION ACROSS THE WALL OF THE TYPE 304 STAINLESS STEEL ANODE-METALLOGRAPHIC SPECIMENS No. 2 AND 4

- 1 NONEMITTING CATHODE SUPPORT STRUCTURE-MOLYBDENUM
- 2 ANODE-TYPE 316 STAINLESS STEEL
- 3 INSULATOR-BERYLLIA
- 4 NONEMITTING SPACER-MOLYBDENUM
- 5 EMITTER-IMPREGNATED TUNGSTEN
- 6 FILAMENT ROD-TUNGSTEN
- 7 INSULATOR SPACER-ALUMINA
- 8 HOLDING RINGS-TANTALUM

SCALE 3 in = 1 in

(REDUCED TO 75% FOR REPRODUCTION)

FIGURE 2-21 DETAIL OF HEATER CONSTRUCTION

The given parameters are:

- a. Heater component physical dimensions.
- b. Material physical properties as a function of temperature:
 1. Electrical resistivity.
 2. Thermal conductivity.
 3. Thermal emissivity.
 4. Electron emission (for the cathode)
- c. Sodium inlet temperature, flow rate, surface heat transfer coefficient from the anode, and sodium flow cross-sectional area.
- d. Electrical parameters are filament current, plate current flow in the two ends of the cathode, and plate voltage. The calculation starts at the end of the cathode nearest the sodium inlet and proceeds in the following steps.
 1. For the first elemental length of heater, the temperatures of the filament, cathode support structure, and cathode are assumed. Based on these numbers, the internal heat generation in the various elements by resistance heating is calculated as in the electron emission from the element of the cathode under consideration. An energy balance is then performed on the element, considering both radiant heat transfer and electron flow. The initial assumed filament temperature is then tested against the calculated filament temperature, and if the difference exceeds 0.1%, the calculated temperatures are used as a second assumed set and the calculation is repeated until closure is obtained.
 2. When an energy balance is achieved on the first elemental length, the

energy input to the sodium is used to calculate the rise in temperature of the sodium flowing by the element, and the plate current available for the succeeding elements is reduced by the amount of plate current flowing to the anode in the first element.

3. These steps are repeated for each succeeding element until the plate current supplied at the cathode end is exhausted, or until the active length of the heater is used up. In a particular calculation for the case where half the plate current flow is at each end of the cathode, the plate current from the cathode end will be exhausted at or before the midpoint of the cathode.
4. When the cathode end plate current is exhausted, the operations in steps 1 and 2 are continued, except that in step 3, the elements of plate current are now added, and are called filament end plate current. The sequential calculation is continued with the increasing value of filament end plate current until either the cathode length is exceeded or the calculated filament end plate current exceeds the input value. When the calculated cathode length equals the actual length, the calculation is stopped. If, however, the calculated filament end plate current equals the input value before the cathode length is exceeded, then the calculation returns to the end of the cathode and calculation insert elements which have no emission, then repeats the filament end calculation. This procedure is repeated until the plate current calculated at the filament end of the cathode is nearly the same as the given value and the full length of active cathode is used up.

2.2.5.5 Comparison of Results

Calculations have been performed for two cases taken from the operational data obtained during testing of heater E-EB-3-1. Figures 2-23 and 2-24 are a comparison of the calculated sodium temperature along the heater with the measured values of sodium temperature. Also shown on these curves are the calculated filament, cathode surface, and anode inside surface temperatures. The cathode and filament temperatures are in degrees centigrade, while the sodium and anode temperatures are given in degrees fahrenheit. It should be noted that the calculated sodium temperature rise in these figures is greater than the measured temperature rise which implies the lack of an energy balance. This discrepancy is explained by the fact that in the tests performed, the measured temperature continued to rise for 2 inches downstream from the end of the heated length. When this rise is taken into account, an energy balance between the measured and the computed case is achieved.

In both the calculated and measured sodium temperature profiles along the heater, there is a change of slope with position; the rate of sodium temperature rise being greater at both ends than in the middle. In the case of the measured data, this slope change is displaced downstream as is the point of maximum temperature. Exact quantitative comparisons between calculated and test results have not been possible, because of incomplete test temperature data, but the similarity of trends lends credence to conclusions based on the analysis.

Based on this analysis, it can be concluded that power generation is greatest at the ends of the heater and that there can be a dead zone in the center, which becomes wider as the filament temperature is increased, and conversely improved by lowering filament temperature. Uniform power distribution at the highest power levels can therefore be attained by reducing the emission of the ends of the cathode by suitable design of emitter end segments.

FIGURE 2-22. ELECTRON BOMBARDMENT HEATER CONTROL CIRCUIT

FIGURE 2-23. TEMPERATURE RISE ALONG HHFH

$$E_p = 595$$

$$I_1 = I_f = 68.0 \text{ A}$$

$$I_2 = I_3 = 0.5 I_p = 20.5 \text{ A}$$

INLET TEMP. = 1057°F
3.5 gpm

DATA FROM DECEMBER 18, 1967

FIGURE 2-24. TEMPERATURE RISE ALONG HHFH

SECTION III

CLADDING MATERIALS TESTING3.1 SUBTASK 2.1—MECHANICAL
PROPERTIES TEST OF CLADDING
CANDIDATES3.1.1 Loops 5R and 6R—Corrosion Rates
of Candidate Cladding Materials

The cladding materials screening runs in Loops 5R (runs 5R-1, 5R-2, and 5R-3) and 6R (run 6R-1) have contained specimens to monitor the effects of sodium (mass transfer) at 1300°F in the absence of stress. The results indicate, for all practical purposes, linear corrosion rates.

3.1.1.1 Effect of Alloy Composition

Corrosion samples of Types 304, 316, 347 stainless steels, and Incoloy 800 have been exposed in Loop 5R for 1745, 2083, and 3044 hours. The materials were in the as-received (mill-annealed), carbide agglomerated (1650°F, 24 hours), and cold-reduced (20%) conditions. The average corrosion results are shown in Table 3-1. The specimens were all at 1300°F, sodium velocity of 17 ft/sec, and an essentially constant downstream position, which facilitates the plotting of the data. The results are plotted in Figure 3-1 for the annealed materials, and show curves that can be readily extrapolated through zero time and weight change.

The data show a distinct difference among Types 316 and 304 (347) stainless steel, and Incoloy 800 in the mill-annealed condition. The effect of the 1650°F heat treatment on the corrosion of Types 304 and 316 stainless steel, and Incoloy 800 is completely masked by the scatter in the data. However, for Type 347 stainless steel, there is a definite trend toward lower corrosion for the heat-treated material. Type 347 stainless steel is an alloy

designed to respond to this heat treatment by tying up the carbon intragranularly in the form of niobium carbides. The effect this may have on the corrosion properties (primarily the initial corrosion effects) is not known. The effect of cold work can be generally said to increase the corrosion (primarily the initial corrosion effects) of the alloys. This can be explained on the basis of increased (more uniform) diffusion of the selectively leached elements during the initial corrosion period, prior to the establishment of steady-state conditions. Although the initial corrosion period is not seen in the plots, it exists, but occurs very rapidly under the test parameters of concern here. The data for cold-worked Incoloy 800 (annealed also) are badly scattered. This can be explained on the basis of the intergranular dissolution that occurs in this alloy, noted previously, ⁽⁷⁾ which also results in grain dropping.

3.1.1.2 Effect of Loop Position

Corrosion cartridges containing annealed Type 316 stainless steel were exposed in Loop 5R for 1745, 2083, and 3044 hours, and in Loop 6R for 1008 hours. One of the cartridges in each run was upstream of the data reported in subsection 3.1.1.1, and two cartridges were in downstream positions. All of the cartridges were at 1300°F with a sodium velocity of 8 to 10 ft/sec. The results are shown in Table 3-2 and Figure 3-2. The data for the various loop (downstream) positions can be readily extrapolated through zero time and weight change.

The effect of loop position, discussed in Subtask 1.1, can be clearly observed from the above data. The interesting observation however, is the dotted curve in Figure 3-2 representing the Type 316 stainless steel data from the test section. While the downstream position

of these data is as shown in the plot, the velocity is 17 ft/sec as compared to ~9 ft/sec for the three corrosion cartridges. The results from Subtask 1.1, Figures 2-3 and 2-4, show that an increase in velocity will overshadow the downstream effect (compare test zones FB and FD in both figures). In other words, even though the test section has an intermediate downstream position, the corrosion rate should be as high or higher than the first corrosion

cartridge due to the higher velocity. The explanation for this effect is the differences in mass flow rate. The corrosion cartridges had a mass flow of ~10 gpm and the test section (eight parallel channels) a mass flow of ~1.2 gpm. This trend in the data suggests that possibly the corrosion rate model development should contain Reynolds Number as a variable to encompass velocity, mass flow, and hydraulic diameter into one term.

TABLE 3-1

EFFECT OF ALLOY COMPOSITION AND STRUCTURAL CONDITION
ON CORROSION WEIGHT LOSS, mg/dm²

<u>Material</u>	<u>Condition</u>	<u>1745 hours</u>	<u>2083 hours</u>	<u>3044 hours</u>
Type 316 stainless steel	Annealed	103	139	174
	Carbide	80	153	149
	Agglomerated			
	Cold-worked	167	225	260
Type 304 stainless steel	Annealed	173	214	314
	Carbide	121	228	347
	Agglomerated			
	Cold-Worked	254	232	350
Type 347 stainless steel	Annealed	188	259	352
	Carbide	168	207	318
	Agglomerated			
	Cold-Worked	234	317	389
Incoloy 800	Annealed	309	381	675
	Carbide	392	---	560
	Agglomerated			
	Cold-Worked	305	297	855

TABLE 3-2

EFFECT OF LOOP POSITION ON THE CORROSION
OF TYPE 316 STAINLESS STEEL WEIGHT LOSS, mg/dm²

	<u>1008 hours</u>	<u>1745 hours</u>	<u>2083 hours</u>	<u>3044 hours</u>
Sodium	66	145	173	327
Flow	45	82	99	151
↓	34	69	77	126

FIGURE 3-1. CORROSION RATES OF ANNEALED MATERIALS IN 1300°F SODIUM

FIGURE 3-2. CORROSION RATES OF ANNEALED TYPE 316 STAINLESS STEEL VERSUS LOOP POSITION IN 1300°F SODIUM

3.1.2 Loop 6R, Run 6R-1

The experimental concept of run 6R-1 and the specimen test parameters that have been reported, ⁽⁸⁾ were in error because of the mislocation of the specimens during fabrication. Because of the above error, companion specimens (unfailed) to the failed specimens were not terminated at failure.

3.1.2.1 Test Results

Run 6R-1 was terminated after 1008 hours of operation in 1300°F, T_{max} , flowing sodium. The 16 biaxial stress cycle fatigue specimens (annealed Type 316 stainless steel) have been removed from the loop and examined. The results of the test are shown in Table 3-3 and Figure 3-3.

The data show a smooth curve of maximum hoop stress versus time-to-failure. However, the data vary in such a way that no conclusion can be drawn concerning the relative failure times of constrained and unconstrained specimens. At the highest and lowest

stress levels, the constrained specimens appear to have longer life-times. However, the scatter at the middle stress levels shows data for the constrained specimens at both shorter and longer failure times compared to the unconstrained specimens.

Macroexamination shows the unconstrained specimens to expand to the limits of the test channel (0.250-inch outside diameter to ~0.300-inch outside diameter) and failing in a manner suggestive of a burst test. This is understandable since the maximum hoop stress levels are at or above the yield stress at 1300°F (~20,000 psi) for Type 316 stainless steel. The constrained specimens show cracks on the outside diameter surface ~35°—90° circumferentially removed from the wire constraints. The maximum deformation is less than for the unconstrained specimens and the cracks are small and typical of biaxial stress-rupture failures. However, the local deformation (strain) in the failure areas may be significantly greater for the constrained specimens. This value must await calculation from macrophotos of selected cross sections.

TABLE 3-3

BIAXIAL STRESS CYCLE FATIGUE RESULTS FROM RUN 6R-1 IN 1300°F SODIUM

Stress Level (psi)	Specimen Configuration	Exposure Time (hours)	Number of Cycles
25,000 maximum 7,000 minimum	Unconstrained	49.1* Failed	491
	Constrained	49.1*	491
23,000 maximum 7,000 minimum	Unconstrained	87.3 95.5 Failed	873 955
	Constrained	87.3 Failed 95.5	873 955
21,000 maximum 7,000 minimum	Unconstrained	241.5 309.8 Failed	2415 3098
	Constrained	241.5 Failed 309.8	2415 3098
19,000 maximum 7,000 minimum	Unconstrained	672.8 Failed	6728
	Constrained	711.8 Failed	7118

* Two samples at each condition showed the same results.

FIGURE 3-3. BIAxIAL STRESS CYCLE FATIGUE OF TYPE 316 STAINLESS STEEL IN 1300°F SODIUM

Preliminary metallographic examination reveals the following apparent modes of failure:

Unconstrained—General outside diameter and inside diameter grain boundary separations followed by local "necking," and subsequent rapid failure.

Constrained—Large inside diameter (small outside diameter) grain boundary separations, aided by localized outside diameter (large) separations away from the wire area, and subsequent failure in these localized areas.

The verification and possible explanation of the above observations await a more detailed metallographic examination.

SECTION IV

TASK 3—SODIUM CHEMISTRY

4.1 SUBTASK 3.1—SAMPLING OF SODIUM SYSTEMS

The installation of an on-line gas chromatograph on Loop 8 has been successfully completed. The chromatograph was operated for 1 week during prerun test trials. A sample was taken every hour, alternately from the hot and cold leg surge tanks. Direct readout in parts per million for both hydrogen and nitrogen were reproducible to $\pm 5\%$. Minimum sensitivities of 2 ppm for hydrogen and 50 ppm for nitrogen were achieved. These are based on taking a 10 cc sample at 30 psig.

The gas chromatograph operates at a temperature of 50°C for both the oven and the detectors. The argon carrier gas flows at a rate of 80 cc/min with a column pressure of 30 psig. The 100k thermistor detectors are used at 1.75 milliamperes. Sampling intervals can be varied from 15 minutes (the time required to complete an

analysis) to 5 hours. The signal created at the detector by the column effluents is displayed as a peak on a multirange (0—1, 0—10 mV) recorder. This signal is also transmitted to a Vidar, which integrates the area under the peak. The area in the form of a totalized count from the voltage to frequency converter is then sent to either the hydrogen or nitrogen readout recorders, which are calibrated in parts per million.

The entire system is operated automatically once the sample interval is selected to achieve 24-hour surveillance of the surge tank cover gas.

The on-line chromatograph was first employed to measure the response of hydrogen and nitrogen to cold trapping during the startup phase of run 4 in Loop 8. Curves showing the trends of these impurities in two loop positions are shown in the following drawing.

4.1.1 Observations

- a. The on-line chromatograph demonstrated that the cold trap is effective in controlling the hydrogen, but appears to have no observable effect on controlling nitrogen.*
- b. Operational effects, such as cold trapping to reduce loop hydrogen or contamination of the cover gas, have been effectively monitored.
- c. The chromatograph has demonstrated the ability to detect cover gas impurities alternately from two positions on an hourly basis and display the absolute levels directly.

4.2 SUBTASK 3.2—IMPURITIES IDENTIFICATION IN SODIUM SYSTEMS

4.2.1 Cold Trap Examination

A previous report⁽⁹⁾ described the results of analyses of materials deposited in a cold trap which had seen service in a ferritic

(2-1/4 Cr-1 Mo) loop. The loop (Loop 5) operated for a total of 20,000 hours at controlled oxygen levels, corresponding to plugging temperatures of 290°F (about 10 ppm oxygen) and 450°F (about 50 ppm oxygen). Loop temperatures were in the range 600° to 1200°F.

Significant findings were: the direct observation (by x-ray diffraction) of sodium hydride; evidence for the presence of at least five separate forms of carbon and, specifically, the presence of high concentrations of the cyanide and carbonate anions; and unexpectedly low concentrations of sodium monoxide.

Recently, examination has been under way of a cold trap which had operated in an austenitic (Type 316 stainless steel) system. This loop (Loop 5) had functioned for about the same period of time and in about the same temperature range as the ferritic loop studied previously. (In one run, the maximum temperature was 1300°F.) Two oxygen levels had also been maintained: i.e., plugging temperatures of 450°F (about 50 ppm) and 300°F (about 10 ppm).

* The rise in N₂ level after onset of cold trapping is believed to have resulted from a leak in a piping connection, which later developed into a sodium leak.

The contents of the trap, sectioned into four pieces, are shown in Figure 4-1. Initial examination has shown that hydrogenous species

have deposited in this trap also. The oxygen concentration in the region of greatest deposition appears to be about 1% (about 4% Na_2O) which, though roughly five times that found in the Loop 5 trap, is still well below that expected.

The major difference observed to date in the deposits from the two systems was in the quantity and in the nature of the carbon species present. There was a factor of three less total carbon in the austenitic system residues which was primarily caused by a depletion in the concentration of carbon present in compound form, relative to that found in the elemental state. The carbonate and cyanide anions, for example, which were the major carbon-containing forms in the ferritic system residues, were reduced in concentration sevenfold in the austenitic loop deposits. Further, a compound yielding acetylene upon hydrolysis of the ferritic loop deposits was undetected in the stainless steel loop.

Total carbon was determined using a wet combustion procedure.⁽¹⁰⁾ This method also permits distinguishing between carbon species which are converted to gases by treating the sample with water and then dilute acid (carbonates, cyanides, and carbides) and those which require oxidation to effect the transformation (largely elemental carbon). The results of these measurements are shown in Table 4-1.

It is clear from the data that the reduced carbon level in the Type 316 stainless steel system is due largely to the decrease in species which are readily volatilized.

Several techniques were used to discriminate the carbon forms still further (1) Impurities were concentrated from samples of the cold trap contents by removing sodium by vacuum distillation, amalgamation, or liquid ammonia dissolution.⁽¹¹⁾ Portions of the impurities thus isolated were examined by x-ray diffraction and infrared spectroscopy. Separate fractions were hydrolyzed and acidified the volatile products were identified by gas chromatography. (2) The gases released upon heating sodium from the trap in vacuum and those evolved during amalgamation were caught and characterized. (3) Specific determinations for cyanide (spectrophotometry) and carbonate (gas chromatography) were also performed on the sodium. The results of these determinations are summarized in Table 4-2.

The concentration of elemental carbon (repeated from Table 4-1 for convenience) is clearly of the same order of magnitude in the two systems. On the other hand, carbon in the form of cyanide or carbonate is much lower in the stainless steel residues. There is also a reduction in this system, although of lower magnitude, in the carbon present in a form readily evolved (as methane) upon heating. Finally, it was observed that the species which produced acetylene upon hydrolysis of the 2-1/4 Cr-1 Mo loop deposits was absent in the austenitic system. This behavior is characteristic of carbides, and particularly of those of the alkali metals and alkaline earths.

TABLE 4-1

CONCENTRATION OF CARBON IN COLD TRAP SODIUM

<u>Carbon (ppm)</u>	<u>Austenitic Loop</u>	<u>Ferritic Loop</u>
Volatilized by water, acid	90	640
Volatilized by oxidation	220	280
Total	310	920

FIGURE 4-1. CONTENTS OF LOOP 1 (COLD TRAP SECTIONED INTO FOUR PIECES)

TABLE 4-2

FORMS OF CARBON IN COLD TRAP DEPOSITS

<u>Form of Carbon</u>	<u>Austenitic System (Carbon, ppm)</u>	<u>Ferritic System (Carbon, ppm)</u>
"Carbide" (acetylene production)	Not detected (< 1)	25
Evolved upon heating (largely CH ₄)	10	25
Carbonate	50	350
Cyanide	45	280
"Elemental"	220	280

The metallurgical examinations⁽¹²⁾ of these two loops indicated that carbon activity (carburization-decarburization reactions) was negligible in the Type 316 stainless steel loop, while the ferritic loop experienced a decarburization (at a low rate). In light of these observations, the cold trap analyses imply that it is the complex carbon species that participate in the movement of carbon. Moreover, there is the strong suggestion that the carbide forms may be the most important of these. There is confirmation of this view in the recent work of Wildeman, et al.,⁽¹³⁾ which indicates that acetylene is the major gaseous species released upon hydrolysis of cold trap deposits from a sodium system containing deliberately added high levels of carbon.

Work is continuing on the cold trap residues with the objectives of determining the extent and distribution of oxygen and hydrogen impurities and to characterize other species which may have deposited.

4.2.2 Study of Particulate Matter in Sodium

Two previous reports in this series discuss the characterization of material deposited from Loop 9 sodium on so-called impingement samples (corrosion specimens in the form of baffles and washers instead of the usual ring configuration).

In a continuation of this work, cartridges of conventional corrosion samples, exposed to sodium in the cold zone of Loop 8, were examined in an analogous manner to that used in the Loop 9 study: Sodium was removed by vacuum distillation, the cartridges were dismantled in a glove box, and corrosion deposits were removed and encapsulated for x-ray diffraction and fluorescence analyses.

Preliminary results indicate that the Loop 8 corrosion deposits are similar to those obtained from a roughly equivalent temperature region of Loop 9. The major phase in the x-ray patterns was due to a face-centered cubic structure ($a_0 = 3.580 \text{ \AA}$). Nickel is the major element present in both samples, but is at a higher relative concentration in the Loop 8 material. (Appreciable amounts of iron, chromium, and manganese are also present.) The Loop 8 deposit's diffraction pattern contains lines probably from metallic nickel. These were not detected in the earlier examination (Loop 9).

A rough determination of the curie temperature indicated that the material was magnetic in the range ambient to 700°F. (The Loop 9 deposits were magnetic, but no attempt was made to determine the temperature range.)

A strong minor phase of the general form $M_{23}C_6$, $a_0 = 10.622 \text{ \AA}$, was found in both samples. Acidification of the samples released a variety of hydrocarbons, thus confirming the presence of carbides. The same process led to the release of CO_2 , indicating the presence of some carbonate in the samples.

The minor phase with the sodium nickelate structure was observed again. The compound is apparently stable in air, thus precluding its actually being sodium nickelate, which is converted to nickel carbonate under these conditions.

4.2.3 Surveillance Analyses

During the past year, there has been a gradual resumption of the routine analyses of the coolant from the mass transfer loops. In Phase I of the program, metals and carbon were determined regularly over an extended period, but they were discontinued when the results were not found to be useful in correlating with the corrosion and mass transfer processes occurring in the loops. The measurements were reinstituted recently, on an infrequent schedule, because of their more direct effect on mechanical properties, and to warn of the inadvertent variations in the concentrations of these contaminants. Carbon is determined by a wet combustion technique,⁽¹⁰⁾ while emission spectrograph is used for monitoring the metallic impurities.

The new surveillance program emphasizes the laboratory analysis for oxygen because of its critical role in corrosion and for hydrogen because of its ubiquity in our systems. In the early work, oxygen was determined only by plugging indicator measurements, and the laboratory hydrogen method was not established until the program had virtually been completed. Progress on establishing these methods and in developing an adequate means of sampling (flow-through sampler) has been described in the previous reports in this series.

The first application of these techniques has been to samples from Loops 8 and 9. These results are tabulated and discussed below. Table 4-3 provides the data for hydrogen alkalinity and plugging indicator measurements for Loop 9.

The first four samples were taken at random times during the operational cycle and, thus, should contain sodium representative of the cold leg (sampling temperature, $860^\circ - 870^\circ\text{F}$). The lowest temperature flow breaks in the plugging indicator traces (ascribed to the precipitation of Na_2O) are in good agreement ($\pm 10^\circ\text{F}$) with the prevailing cold trap temperatures, 250°F to 270°F . Flow breaks at higher temperatures were also observed (last column, Table 4-3). These, are believed on the basis of earlier work, to be due to the precipitation of hydrogenous compounds. There is also apparent agreement between the alkalinity value and the solubility for Na_2O at the corresponding cold trap temperature. This is probably fortuitous, since the "oxygen" calculated from alkalinity includes $NaOH$ and Na_2CO_3 , as well as Na_2O and moreover some preliminary data indicate that the former two species comprise the major portion of the total alkalinity. Further, the validity of the Claxton⁽¹⁴⁾ curve, based as it is upon composite data of several investigators, is also questionable. The most recent determination of Na_2O solubility⁽¹⁵⁾ indicates values approximately one-third that of Claxton for corresponding temperatures in the range of interest.

The last three values were part of a test determining the effects of raising the cold trap temperature on the plugging indicator flow breaks. Results have been discussed in an earlier report.

The Loop 8 data are shown in Table 4-4. Plugging runs could not be made each time samples were taken for chemical analysis because of operation of the plugging compound collectors, but the few results shown and additional data obtained at other times during the

TABLE 4-3

HYDROGEN, ALKALINITY, AND PLUGGING TEMPERATURE
MEASUREMENTS FOR LOOP 9

Run and Sample Number	Date	Hydrogen ⁽¹⁾ (ppm)	Alkalinity ⁽²⁾ (ppm)	Cold Trap ⁽³⁾ Temperature (°F)	Plugging Indicator Flow Breaks	
					[Temperature (°F)]	
					"Oxide" Break ⁽⁴⁾	Other Breaks
9-1-2A	4/22/67	1.6	7.8	Valved Off	278, 260	525, 495
9-1-4A	5/01/67	1.8	3.4	270 (6.7)	258, 277	530, 605
9-1-5A	5/23/67	5.0	5.7	250 (5)	250, 260	405, 440
9-1-6A	6/15/67	7.1	3.5	270 (6.7)	255, 260	435—515, 470
9-1-7A	6/16/67	8.2	5.9	315 (12)	363, 355	450, 640—460
9-1-8A	6/17/67	8.3	6.4	350 (6)	360, 365	670—500—435, 675—430
9-1-10A	6/23/67	14.3	50.0	400 (31)	None Discernible	700

(1) By diffusion technique. At least two analyses run on each sample. Results are good to ± 0.5 ppm.

(2) A measure of the oxygen concentration determined by amalgamation/titrimetry; correction made for sodium hydride; calculated on the assumption that Na_2O is only alkaline species remaining. At least two analyses run on each sample. Results are good to $\pm 20\%$.

(3) Value in parentheses is the oxide saturation solubility at the temperature calculated from the data of Claxton. ⁽¹⁴⁾

(4) The values represent duplicate runs.

TABLE 4-4

HYDROGEN, ALKALINITY, AND PLUGGING TEMPERATURE
MEASUREMENTS FOR LOOP 8

Run and Sample Number	Date	Hydrogen ⁽¹⁾ (ppm)	Alkalinity ⁽²⁾ (ppm)	Cold Trap ⁽³⁾ Temperature (°F)	Plugging Indicator Flow Breaks	
					[Temperature (°F)]	
					"Oxide" Break ⁽⁴⁾	Other Breaks
8-2-1A	5/8/67	2.2	3.0	Valved Off	None Observed	590—470, 565—445
8-2-3A	5/24/67	3.5	4.7	280 (7.7)	Not Operated	-
8-3-1A	8/22/67	4.3	5.1	285 (8.2)	240, 240	385, 340
8-3-6B	9/08/67	11.1	16.2	290 (8.7)	Not Operated	-
8-3-7B	10/05/67	10.0	23.5	270 (6.7)	248, 250	385, 400

(1) By diffusion technique. At least two analyses run in each sample. Results are good to ± 0.5 ppm.

(2) A measure of the oxygen concentration determined by amalgamation/titrimetry; correction made for sodium hydride; calculated on the assumption that Na_2O is only alkaline species remaining. At least two analyses run on each sample. Results are good to $\pm 20\%$.

(3) Value in parentheses is the oxide saturation solubility at the temperature calculated from the data of Claxton. ⁽¹⁴⁾

(4) The values represent duplicate runs.

runs indicate a spread of about 40°F between the temperature of the "oxide" break and that of the cold trap (the cold trap being higher). An explanation for this deviation is not available.

For the first three samples, the alkalinity "oxygen" values show reasonable agreement with oxide solubilities corresponding to the cold trap temperatures. The chemical determinations show an appreciable increase in alkalinity for the subsequent samples, but there is not a corresponding rise in the temperature of the lowest flow interruption. Since the total hydrogen in these samples also increased, it is surmised that it was an increase in hydroxide rather than oxide concentration which caused the change in alkalinity. An excursion in cold trap temperature to 320°F a few days prior to taking Sample 8-3-6B may explain the introduction of sodium hydroxide,

but no explanation of the high alkalinity of the last sample is available.

Tables 4-5 and 4-6 provide data on the analyses for carbon and metal impurities. The total carbon appears to be in the expected range, 25 to 50 ppm. However, the ratio of water-acid to total carbon seems high in several cases. It should be noted that one of the components of the water-acid fraction is carbonate and this species would also cause higher than expected "oxygen" values when "oxygen" is determined by alkalinity measurements. There are insufficient data to draw any conclusions at this time but this trend (high water-acid carbon) will receive special attention in future runs.

The metal analyses are in the normal range. Silicon is probably from the etching of the glass beaker used for sample preparation.

TABLE 4-5

ANALYSES FOR CARBON—LOOPS 8 AND 9

<u>Sample No.</u>	<u>"Water-Acid" Carbon^(a) (ppm)</u>	<u>Total Carbon^(b) (ppm)</u>
8-3-1A	4	26
8-3-11A	10	30
8-3-24A	17	54
9-1-2A	4	24
9-1-5A	18	53
9-1-8A	7	41

* "Water-acid" carbon is defined as the carbon converted to gaseous forms when water is used to dissolve a sodium sample and when the resulting solution is acidified.

(a) Results good to ± 2 ppm.

(b) Results good to ± 5 ppm.

TABLE 4-6

METAL IMPURITIES—LOOPS 8 AND 9

<u>Metal</u>	<u>Sample Number</u>	
	<u>8-3-11A</u> (ppm)	<u>9-1-2A</u> (ppm)
Fe	5	5
Cr	3	3
Mn	0.5	0.5
Al	1	1
Mg	1	3
Si	-	20

Results good to $\pm$ a factor of two. Other metals undetected; < 5 ppm.

Based upon these initial observations, it appears that:

- a. The "oxygen" content of Loop 9 appears to be in good control, based upon the agreement between cold trap and plugging indicator temperatures. There is also apparent agreement in the "oxygen" cal-

culated from alkalinity measurements, although this is probably fortuitous.

- b. Loop 8 does not appear to be in as close "oxygen" control as Loop 9. Agreement between plugging and cold trap temperatures is not as good. Alkalinity determinations yield higher values than expected, and this is attributed to the presence of NaOH and Na_2CO_3 .
- c. Preliminary results indicate that the major fraction of the total oxygen impurity in the loops may be in the form of hydroxide and carbonate rather than oxide. The investigation of this point will be pursued in future runs and will be abetted by data obtained with an in-line distillation unit and an oxygen activity meter being installed in Loop 8.
- d. Analyses for total carbon on metals show these impurities to be in the expected ranges. There is some evidence that carbonate may be higher than normal.

SECTION V

LIST OF SODIUM MASS TRANSFER PROJECT REPORTS

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2. GEAP-3726—Lockhart, R. W., et al., "Sodium Mass Transfer: II Screening Test Data and Analysis - Mass Transfer Results," Volume I, April 1962.
3. GEAP-3726—Hetzler, F. J., and Young, R. S., "Sodium Mass Transfer: II Screening Test Data and Analysis - Metallurgy," Volume II, June 1962.
4. GEAP-3726—Lockhart, R. W., "Sodium Mass Transfer: I Screening Test Data and Analysis - 1961 Corrosion Sample Data," Volume 3, May 1962.
5. GEAP-4006—Alter, H. W., and McManus, P. A., "Sodium Mass Transfer: III The Application of Liquid Ammonia as a Leaching Agent," April 1962.
6. GEAP-4181—Pohl, L. E., "Sodium Mass Transfer: IV 1962 Corrosion Sample Data," January 1963.

7. GEAP-4182—Lockhart, R. W., "Sodium Mass Transfer: V 1962 Test Run Reports," June 1963.
8. GEAP-4183—Dunn, E. L., "Sodium Mass Transfer: VI Statistical Correlation of 1961 - 1962 Corrosion Data," February 1964.
9. GEAP-4178—Sabol, W. W., and Lockhart, R. W., "Sodium Mass Transfer: VII Methods for Determining Impurities in Sodium Samples," September 1963.
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