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**SODIUM COMPONENTS
DEVELOPMENT PROGRAM
MASS TRANSFER INVESTIGATIONS
IN LIQUID METAL SYSTEMS
QUARTERLY PROGRESS REPORT
NO. 4
DECEMBER 1967-FEBRUARY 1968**

U.S. ATOMIC ENERGY COMMISSION
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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. 4
December 1967—February 1968

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

E. L. Zebroski, Manager
Sodium Reactor Technology Section

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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 are given (Task 1.1) representing hot leg corrosion and cold leg deposition observed in run 8-1 of 3000 hours.

Test conditions were selected (Task 1.2) for Loop 9, modeling FFTF 5 MW closed loop corrosion conditions. The temperature profile of the hot leg section corresponding to the reactor tube was adjusted to permit operation of the electron bombardment heater at 20 KW (800,000 Btu/h-ft² average).

The replacement of an anode tube (simulating reactor fuel cladding) on an electron bombardment heater was successfully accomplished (Task 1.3). This demonstrated the

ability to expose cladding (1 cm diameter × 25.4 cm length in this case) to high heat flux in sodium, remove the cladding for examination, replace or renew the test cladding, and resume test operations.

A new fast acting (~10 microseconds) solid-state crowbar circuit was developed and tested to divert cathode to anode arcs in the electron beam heaters.

The analysis of 6R-1P cladding specimens (Task 2.1) was continued. Bending stresses associated with pressurized specimens were analyzed for samples which simulate fuel spacers.

The flow blockage in a plugging indicator was related to hydrogen additions to a loop (Task 3.1). The cold trap was identified as a major source of hydrogen during some operations.

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, 2½Cr-1 Mo and 5Cr-½Mo-½Ti 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. Two types of heating are under test: (1) electron bombardment heating, and (2) resistance element heating.

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). Rebuilding Loop 4 is under way to permit the start of long-term stress rupture tests in fiscal year 1968.

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 fabricated in 1966 and five have been installed in the SCTI test facility of LMEC by Atomics International.

1.2 REPORTS

Monthly report letters are prepared reporting progress in all tasks. Progress letters No. 1 through 101 covering work from July 1, 1959 through March 31, 1968 have been issued.

In addition to the progress letters, 28 topical reports and 3 quarterly progress reports covering test results have been issued (see Section V).

This is the fourth 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 SPECIFICATIONS

2.1 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) re-evaluating 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 sched-

uled 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 the velocity effect on mass transfer rates. The parameters and experimental conditions for these runs are summarized in Table 2-1.

2.1.1 Corrosion Rate Equation for the Hot Leg Based on Data from Run 8-1

The data, including weight change, temperature, time, and flow, from the isothermal hot-leg sections, F, G, and H, in run 8-1, were transformed into the following terms to fit the previously determined model for the corrosion rate equation: $\ln(V)$, $(L/D)_1$, $\ln(L/D)_2$, $1/(t+1)$, and $\ln CR$. The combined analysis of data, again including all the parametric run variables, collected for the 1000-, 2000-, and 3000-hour time points yielded the following empirical equation:

TABLE 2-1
RUN PARAMETERS FOR LOOPS 8 AND 9

Run	Velocity (fps)		Downstream L/D		Flow (gpm)	Samples		
	Hot Leg	Cold Leg	Hot Leg	Cold Leg		Tube	Ring	Pressurized
8-1	0.7-14.3	0.7-8.1	450	980	3.0	189	63	4
8-2	1.3-26.3	1.3-14.9	450	980	5.7	189	63	12
8-3	1.3-26.3	1.3-14.9	692	1011	5.7	189	63	12
8-4	0.7-14.3	0.7-3.1	692	1011	3.0	189	63	12
9-1	0.7-24.0	0.7-24.0	509	1028	5.7	200	92	12

Common Parameters:

$T_{\max}$ —1300°F, $T_{\min}$ —800°F

Test Duration—3000 hours

Na₂O Impurity— <10 ppm oxygen (260°—280°F plugging)

Test Materials—Types 304 and 316 stainless steel

$$\ln CR = 3.3745 + 0.3193 \ln(V) - 0.0009 (L/D)_1 - 0.0506 \ln(L/D)_2 + 0.7173 (1/t+1)$$

where:

CR = corrosion rate, weight loss per unit time (mg/dm²-mo)

V = sodium velocity (fps)

(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

t = exposure time (months)

The oxygen ($1.156 \ln O_x$) and temperature - $\frac{23,827}{T+460}$ variables previously determined in Phase I are included as constants. The above equation was developed at a temperature of 1300°F and an oxygen level of <10 ppm (best approximation is presently 7 ppm).

2.1.2 Deposition Rate Equation for the Cold Leg Based on Data from Run 8-1

The data from the isothermal cold leg sections, N, R, and B, in run 8-1 can be represented as follows:

$$\ln DR = 2.068 + 0.1390 \ln(V) - 0.0013 (L/D)_1 - 0.0335 \ln(L/D)_2$$

2.2 SUBTASK 1.2—FFTF CLOSED LOOP SIMULATION

Loop 9 modification is complete and all the samples have been fabricated. The instructions for the preparation and installation of the samples have been prepared and the work is in progress. Figure 2-1 shows a view of the modified loop with the thermal insulation in place.

A review of the test plan has indicated the desirability of operating the high flux heater at a power level of 20 kW to produce an average 0.8×10^6 Btu/h-ft² heat flux. The

FIGURE 2-1. LOOP 9 HCT LEG MODIFIED TO SIMULATE TYPICAL CLOSED LOOP CONDITIONS

design reported in Quarterly Progress Report No. 3 was based on a 25 kW power level. The reduction in power level requires that the inlet temperature of the HHF heater be increased to 1045°F. The outlet temperature and the sodium velocity of the HHF heater have been kept the same, 1300°F and 15 fps, respectively. These changes resulted in a corrosion increase in all sections between the inlet of the main heater and the inlets of the HHF heater and the 50 kW heater. To keep the ΔS increase comparable to FFTF and to avoid a change of the already manufactured test samples, the outlet temperature of the 50 kW heater was lowered by 25°, to 1275°F. (See Figure 2-2 for the revised temperatures on the loop, and Table 2-2 for the new ΔS and temperature values.)

2.3 SUBTASK 1.3—DEVELOPMENT OF HIGH HEAT FLUX HEATERS

2.3.1 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 operations in this quarter demonstrated the capability of the electron bombardment heater (designated E-EB-1-1) to perform a test planned in Subtask 1.2, FFTF Closed Loop Simulation. When the test was terminated on January 13 the heater had operated 6557 hours, of which 1625 were at 800,000 Btu/h-ft² or more. Maximum average heat flux attained was 1.13×10^6 .

2.3.2 Replacement of Anode

Since the operating test was successful, it was decided to attempt to replace the anode as a step in developing means of providing a weighable replaceable corrosion sample after high heat flux exposure. The Type 316 stain-

less steel tube, 0.394-inch outside diameter $\times$ 0.027-inch wall thickness, was cut and removed from the heater (Figure 2-3). It was replaced by a Type 304 stainless steel tube of the same dimensions and welded to the stub of the original Type 316 stainless steel tube using a sleeve which overlapped the new tube and the old tube. Two lap welds closed the joint without necking down the diameter. The tube served as the new anode for the heater.

The objectives of the test were to determine whether the emission had been impaired by the brief exposure of the cathode to air and to obtain data relevant to establishing operating limits for this model electron bombardment heater. Anode power was established at 24 kW, 120% of rated, without delay, with the same voltages and currents experienced in the original Type 316 stainless steel anode. Operation at these conditions was about 40 hours. The filament current was raised to 80 amperes, while the plate current was at 44 amperes. The total power to the anode was 28 kW, for an average of 1,130,000 Btu/h-ft² with bulk sodium outlet temperature 1300°F. Operation at 28 kW was performed for 30 minutes. The filament current divider circuit was then bypassed, and within a few hours (less than overnight) a cathode-anode short developed. The test was terminated at this point. The heater was withdrawn for examination (Figure 2-4) and the power supply was moved and installed for the Loop 9 test. The results of the examination of the Type 304 stainless steel anode are given elsewhere in this report.

Disassembly of the internal cathode circuit of the heater showed that some platinum wires which had been attached to the center tungsten rod had melted, allowing spacer insulators to fall down to the connection end of the heater. This provided a confirmation of tube internal operating temperatures using the melting point of

* THESE TEMPERATURES ARE ADJUSTED FROM VALUES GIVEN IN QUARTERLY No. 3.

FIGURE 2-2. LOOP ARRANGEMENT AND SAMPLE LOCATIONS

TABLE 2-2
LOOP 9 CORROSION MODEL DATA

Section		Inlet Temperature (°F)	Outlet Temperature (°F)	Flow Rate (gpm)	Velocity (ft/sec)	Tube Sample Inside Diameter (inch)	Ring Sample Outside Diameter (inch)	Cartridges No.	L/D Total Section	CR (Estimate) (mg/dm ² -month)	ΔS (ppb)	ΣΔS (ppb)
Heater, 110 kW	E D _{in}	800	1045	5	0.0914				38	0.0141	0.00285	0.029
Samples I		1045	1045	5	6.55	0.750	0.500	3	129	0.716	0.0082	0.03185
Valve		1045	1045	2.8	1.04				11	0.132	0.00137	0.165
Connecting Pipe		1045	1045	2.8	1.04				11	0.155	0.00081	
Heater, 50 kW		1045	1275	2.8	0.0544				24	0.0902	0.0163	
Samples II		1275	1275	2.8	1.73	0.805		3	51	3.85	0.0495	
Connection Pipe		1045	1045	2.2	11.7				29	0.635	0.00791	
Valve		1045	1045	2.2	5.55				15	0.257	0.00081	
Flowmeter B		1045	1045	2.2	2.31				39	0.139	0.00117	
High Heat Flux Heater ⁽¹⁾		1045	1300	2.2	15				207	16.45	0.1179	
Connecting Pipe		1300	1300	2.2	0.82				44	1.45	0.0388	
Cooler I	F D _{out}	1285	1250	5	3.45	0.805		4	48	6.25	0.0513	0.2342
Samples IV		1250	1250	5	3.36	1.000	0.625	2	59	5.44	0.05083	0.2851
Samples V		1250	1250	5	2.04	1.000		2	24	2.61	0.01663	
Cooler II		1250	1175	5	7.85	0.805	0.625	5	187	12	0.179	0.2996
Connecting Pipe		1175	1175	5	1.86				10	0.158	0.00042	0.4786
Cooler III		1175	1100	5	3.14				56	2.92/-7.12	0.0131/-0.0342	0.494/0.4579
Connecting Pipe		1100	1100	5	1.86				14	4.61	0.01831	0.4396
Cooler IV		1100	1025	5	1.86				45	4.77	0.061	0.3768
Connecting Pipe		1025	1025	5	1.86				14	3.92	0.0154	0.3632
Cooler V		1025	950	5	3.14	0.805		5	60	5.5	0.0525	0.3107
Sample X	Remaining Loop	950	950	5	0.91	1.334		2	26	2.2	0.0255	0.3048
Connecting Pipe and Main Flowmeter		950	950	5	12.9				28	14.9	0.0376	0.2672
Cooler VI		950	875	5	0.788				30	1.75	0.0337	0.2334
Sample XI		875	875	5	0.91	1.334		3	25	1.88	0.00218	0.2313
Cooler VII		875	800	5	0.788				30	1.515	0.0291	0.2022
Connecting Pipe		800	800	5	0.788				7	1.09	0.00522	0.1969
Pumps		800	800	5	17.8				425	13.65	0.165	0.03297
Connecting Pipe		800	800	5	0.788				17	0.325	0.0036	0.02936

(1) Ring Cross Section: Outside Diameter = 0.475 inch, Inside Diameter = 0.394 inch. Heated Length = 10 inches.

NEG. No. A-6/Z15b

AFTER 1870 HOURS AT $1,000,000 \text{ Btu/h-ft}^2$ OR MORE IN
 $1250^{\circ}\text{--}1300^{\circ}\text{F}$ OUTLET BULK SODIUM TEMPERATURE

FIGURE 2-3. ELECTRON BOMBARDMENT HEATER, FIRST ANODE TYPE 316 STAINLESS STEEL

FIGURE 2-4. ELECTRON BOMBARDMENT HEATER WITH SECOND ANODE AFTER TEST

platinum at 1776°C. Comparison with heat transfer calculations were in good agreement.

2.3.3 Detection of Hydrogen in Sodium by the Electron Bombardment Heater

This model heater was equipped with an 8 liter/sec ion vacuum pump which was operated continually. In the previous quarter it had been noticed that the internal tube pressure would rise in response to increasing the temperature of the cold trap when it was valved open to communicate with the loop. A test was conducted to determine the time constant of this response and further narrow it down to hydrogen (see subsection 4.3, Hydrogen Distribution Studies). An injection of 0.1 cc of water was made into the cover gas of the surge tank of Loop 1, but no response was detected by the heater. Then an injection of 0.2 cc of water was made and the pressure rose about two decades 2 minutes after the injection. The pressure reached 10^{-6} torr for a short time. This pressure corresponds to a steady-state cold trap temperature of 360°F. Since the transient time of the sodium from the surge tank to the heater is on the order of a minute, it is concluded that the heater responds to an increase in hydrogen in a time of the order of a minute.

It is not known why the original injection failed to produce a response, but it was the first attempt to inject moisture and the small quantity could have been adsorbed in the entrance piping and valve structure before reaching the surge tank cover gas, or it might have been blown back out before closing the valve. But the second injection produced a prompt positive response. Further study is necessary to correlate transient pressure rise to the quantity of water injected or measure

the rate of a steady small in-leakage of moisture.

2.3.4 Development of a Weighable Corrosion Sample

Since the Type 316 stainless steel anode was successfully replaced with a Type 304 stainless steel anode by welding, it was decided to test a tubing flare fitting in a capsule assembly as a means for fastening an anode over a cathode assembly. If this is successful, an anode can be removed after test and weighed to correlate high heat flux effects with other samples using weight change. The capsule was fitted with an ion pump to maintain and measure vacuum in the capsule. A test was assembled and put on automatic operation to immerse the capsule in sodium and to draw it into cool cover gas, repetitively. The lower part of the vertical pipe comprising the body of the test rig was electrically heated and contained sodium. The upper part of the pipe was water cooled. The soak time was approximately 4 minutes in each medium. The initial sodium temperature was 280°F. Leakage was noted in the fitting during the first night after initial operation. It was removed, cleaned, and tightened without disassembly and operation resumed. The sodium temperature was raised in steps while cycling was continued. By February 15, 2600 cycles were completed and the sodium was at 690°F. When the fitting is disassembled at the conclusion of the test, mating surfaces and threads will be checked for galling, self-welding, or similar evidence of possible transfer of material from the fixed part of the joint to the removal part of the joint. If none is found, the use of an anode tube attached by a similar fitting will be considered as a weighable corrosion sample on a future heater.

2.3.5 Solid State Crowbar Development

During the demonstration of heater E-EB-1-1, numerous tripouts of plate

power were experienced. The use of a high speed oscilloscope showed that some of these were definitely spurious trips caused by close settings of the thyatron crowbar while performing its function of protecting the heater from internal cathode-anode arcs. A solid state circuit was developed which will sense an arc and switch practically all of the fault energy away from the heater in a few microseconds. It was expected that the solid state SCR would show better repeatability and less drift than the thyatron. The disadvantages of the thyatron were (1) control characteristics showed aging effects and changed with time, (2) a battery was required to provide fixed constant bias for the control grid. As the battery aged and became weaker, the bias potentiometer needed adjustment, and erratic, spurious tripping developed.

The SCR provides reliability, economy, and simplicity of circuit design, although it must be protected from gate currents which could fuse the gate junction. The circuit shown in Figure 2-5 has been shown to meet the objectives.

The operation of the circuit of Figure 2-5 is as follows. An SCR fires (switches to its low impedance state) when some critical voltage is applied at its gate terminal. This critical voltage is always positive with respect to its cathode so no bias battery is required. The resistor, R_s , is the sensing device for the crowbar package. Almost all of the current which flows through the electron bombardment heater flows through R_s . Thus a change in current through the test device will cause a corresponding change in voltage drop across R_s . Resistor R_3 is a 10-turn potentiometer which allows a portion of the voltage developed across R_s to be seen by the gate of the SCR. Resistors R_1 and R_2 are current limiting resistors which protect the gate of the SCR from being

damaged by overcurrent. When the gate voltage reaches a critical value the SCR will change to its low impedance state in a time on the order of 1μ sec. In the low impedance state the voltage drop across the SCR will be less than 3 volts independent of current greater than 0.5 amp. When the SCR begins to conduct, meter relay AMR2 will be activated in about 100 milliseconds to open the main contactors turning off all plate power. Meter relay AMR1 has been included to protect against a characteristic of the power supply used with electron bombardment heater No. 3. This power supply exhibits a sizable voltage spike on automatic reset which caused the SCR to fire on reset. To eliminate this, a relay was placed in the gate circuit to hold the SCR out of the system until the relay coil senses a small current (say 3 to 5 amps) in the heater, at which time the relay closes and the crowbar functions as described above.

The only foreseeable disadvantage to this crowbar is that the SCR is temperature sensitive. A change of about 50°F caused about 10% shift in the firing point. Thus some sort of temperature control may be necessary if wide variations in ambient temperature are expected.

It is well to note that, assuming that temperature is fairly constant, the operation of the crowbar depends only upon the current drawn by the test device. There is no dependence on the voltage of the electron bombardment heater other than the maximum limit of the SCR, 1100 volts.

2.3.6 Third Anode, Type 316 Stainless Steel

An electron bombardment heater (10-inch heated length) contained within a Type 316 stainless steel anode tube of 0.394 inch outside diameter $\times$ 0.027 inch wall was tested in sodium with the goal of

SCR - GE C150 PA (SILICON CONTROLLER RECTIFIER)

$R_1 = 10\Omega$ 1 WATT

$R_2 = 600\Omega$ 1 WATT

$R_3 = 2.5\text{ k}\Omega$ 10 TURN POTENTIOMETER

$R_5 = 0.5\Omega$ 2 kW

(2) OHMI TE No. 2312

D_1 = DIODE GE 1N1692

AMR 1 AND AMR 2 METER RELAYS

FIGURE 2-5. CIRCUIT

producing 10^6 Btu/h-ft² heat flux for a sustained period of time. This heat flux corresponds to ~22 kW power to the heater.

2.3.6.1 Post-Test Examination Results

The heater operated at or above 22 kW for 841 hours and above 0.8×10^6 Btu/h-ft² for 1625 hours. During this time period, the sodium temperature averaged 1000° F inlet to 1200° F outlet.

At the conclusion of the test, the heater anode was visually examined and found to be in good condition. The anode was slightly bowed, but there were no dented or caved-in regions. The anode had a shiny satin-surface finish, typical of surfaces that have seen sodium corrosion.

The heater anode was removed from the cathode for metallographic examination, which subsequently yielded two items of interest:

- a. The heat flux was concentrated at the ends of the 10-inch emitter zone, and
- b. The metal/sodium temperature increased (the type of increase, linear, etc., cannot be determined) along the heater with increasing downstream position.

The first observation was noted by two effects; (1) a structural variation from outside diameter to inside diameter surface that existed only at the limits of the 10-inch emitter zone, Figures 2-6 and 2-7, and (2) an inside diameter surface deposit that existed only at the limits of the emitter zone, Figure 2-8. The second observation was noted by a constant change in microstructure (increase in grain precipitation) from inlet to outlet, Figures 2-9 through 2-12, and a

constant increase in the thickness of the surface layer on the outside diameter within the emitter zone, Figures 2-10 and 2-11.

The formation and growth of the compositionally disturbed layer is a result of corrosion. Two distinct layers formed on the heater anode, one peculiar to the non-heat transfer areas only, Figures 2-9 and 2-12, and the other peculiar to the heat transfer regions, Figures 2-10 and 2-11. Immediately downstream of the emitter zone (heat transfer), the one layer disappeared, although the microstructure indicated a continuing rise in temperature, Figures 2-11 and 2-12.

2.3.7 Fourth Anode, Type 304 Stainless Steel

The Type 316 stainless steel anode was replaced with a 0.394 inch outside diameter $\times$ 0.027 inch wall Type 304 stainless steel anode on the same cathode assembly. The heater was run as noted above.

2.3.7.1 Post-Test Examination Results

The heater anode was visually examined and found to be caved-in at the ends of the emitter zone. The anode was removed and metallographically examined.

The results revealed the same conclusions formed about the Type 316 stainless steel anode. The anode collapse indicated that the heat flux was concentrated at the ends of the 10-inch emitter zone. The metallographic examination showed the structural variation from outside diameter to inside diameter surface at the limits of the emitter zone, Figure 2-13. At the inside diameter surface, the material had resolidated and grain growth had occurred along with intergranular tearing. A similar

FIGURE 2-6. STRUCTURAL VARIATION ACROSS THE WALL OF THE TYPE 316 STAINLESS STEEL HEATER ANODE AT THE INLET OF THE EMITTER ZONE

FIGURE 2-7. STRUCTURAL VARIATION ACROSS THE WALL OF THE TYPE 316 STAINLESS STEEL HEATER ANODE AT THE OUTLET OF THE EMITTER ZONE

UNETCHED

500X

FIGURE 2-8. INSIDE DIAMETER SURFACE DEPOSIT OF THE TYPE 316 STAINLESS STEEL HEATER ANODE AT THE LIMITS OF THE EMITTER ZONE

GLYCEREGIA

500X

FIGURE 2-9. OUTSIDE DIAMETER SURFACE AND GENERAL MICROSTRUCTURE OF THE TYPE 316 STAINLESS STEEL HEATER ANODE IN AN AREA UPSTREAM OF THE EMITTER ZONE

GLYCEREGIA

500X

FIGURE 2-10. OUTSIDE DIAMETER SURFACE AND GENERAL MICROSTRUCTURE OF THE TYPE 316 STAINLESS STEEL HEATER ANODE AT THE MIDPOINT OF THE EMITTER ZONE

GLYCEREGIA

500X

FIGURE 2-11. OUTSIDE DIAMETER SURFACE AND GENERAL MICROSTRUCTURE OF THE TYPE 316 STAINLESS STEEL HEATER ANODE AT THE DOWN-STREAM END OF THE EMITTER ZONE

FIGURE 2-12. OUTSIDE DIAMETER SURFACE AND GENERAL MICROSTRUCTURE OF THE TYPE 316 STAINLESS STEEL HEATER ANODE IN AN AREA DOWNSTREAM OF THE EMITTER ZONE

FIGURE 2-13. STRUCTURAL VARIATION ACROSS THE WALL OF THE TYPE 304 STAINLESS STEEL HEATER ANODE AT THE ENDS OF THE EMITTER ZONE

occurrence was noted on an earlier Type 316 stainless steel heater anode. ⁽¹⁾ There was a visually observed deposit on the inside diameter surface at the ends of the emitter zone, which was not retained during the metallographic preparation. Although the exposure time was short, a change in general and surface microstructure was observed, Figures 2-14, 2-15, and 2-16. The outside diameter surface perturbation increased with downstream in the heat transfer region and changed immediately downstream of the emitter zone, Figures 2-15 and 2-16. The general microstructure indicated a temperature increase in the heat transfer region, but did not indicate a continued rise in temperature downstream of the emitter zone as did the Type 316 stainless steel anode.

2.4 SUBTASK 1.3.2—DEVELOPMENT HIGH HEAT FLUX RESISTANCE HEATERS

Five resistance heaters were fabricated and assembled. Of the five, one was damaged during the initial stages of fabrication and one was damaged and rendered inoperative just prior to final assembly. One heater labeled "BX" was assembled into the Loop 11 adapter flange and commenced operation December 20. The objective of the run plan for this heater was to demonstrate long-term reliability and operational stability at intermediate range heat flux levels (300,000—500,000 Btu/h-ft²). Additional instrumentation was installed in the control circuitry to protect the heater in the event of a high resistance arc.

An additional adapter flange has been fabricated. This modified flange facilitates

heater replacement to the loop without the necessity for cutting and rewelding the main body of the adapter. Two other resistance heaters are available for loop testing after the objectives on heater "BX" are attained.

Resistance heater "BM" operated without difficulty for approximately 500 hours in Loop 11. During this period heat flux at the cladding outside diameter was calculated to be ~450,000 Btu/h-ft² (45% of target output). Test section outlet temperature was programmed upward from 700° to 850° F during the test period.

After approximately 3 hours at 850° F, electrical power to the heater was interrupted by breaker trip, and preliminary investigation indicated the heater ribbon (coil) to be grounded. Calculations based on operating conditions during the test indicated that the heater coil probably did not rise above 1250° F.

Visual observation of the heater after removal from the loop showed that sodium had entered the heater at a point on the cladding at the transition from 0.313 inch outside diameter to 0.250 inch outside diameter. X-ray photographs of the heater showed the coil to be intact.

Some metal "splashes" in the area of the cladding opening indicate that an electric arc may have occurred. Whether an arc from the center electrode to the cladding made the opening, thereby permitting sodium to enter the heater or whether a cladding crack penetrated through, admitting sodium and causing the arc, is unknown.

10% OXALIC

500X

FIGURE 2-14. OUTSIDE DIAMETER SURFACE AND GENERAL MICROSTRUCTURE OF THE TYPE 304 STAINLESS STEEL HEATER ANODE AT THE MIDPOINT OF THE EMITTER ZONE

10% OXALIC

500X

FIGURE 2-15. OUTSIDE DIAMETER SURFACE AND GENERAL MICROSTRUCTURE OF THE TYPE 304 STAINLESS STEEL HEATER ANODE AT THE DOWNSTREAM END OF THE EMITTER ZONE

10% OXALIC

500X

FIGURE 2-16. OUTSIDE DIAMETER SURFACE AND GENERAL MICROSTRUCTURE OF THE TYPE 304 STAINLESS STEEL HEATER ANODE IN AN AREA DOWNSTREAM OF THE EMITTER ZONE

SECTION III

CLADDING MATERIALS TESTING

3.1 SUBTASK 2.1—MECHANICAL PROPERTIES TEST OF CLADDING CANDIDATES

3.1.1 Loop 6R—Experimental Concept, Run 6R-1P

Run 6R-1P, a preliminary run prior to extensive creep fatigue tests, has been described and discussed in detail in the previous two quarterlies.^(2, 3) The specimens were tubular with and without wire constraint, under cyclic and static loading and tested at 1300° F in air. The results indicated that cycling retarded creep on the basis of time at load. It was not possible to evaluate the wire constrained specimen. There was no cracking observed, as previously noted, but the specimens were not deformed to failure as in previous tests. This had led, as stated previously,⁽³⁾ to the present Loop 6 test, run 6R-1, on both constrained and unconstrained specimens.

3.1.1.1 Strain Analysis

The evaluation of the 6R-1P specimens has continued with the analysis of bending stresses. It is necessary to consider bending strain introduced because of geometry changes in the wire constrained specimens to evaluate the peak surface strain encountered.

To supply detailed information on cross-sectional geometry for such analysis, a wire-constrained specimen was sectioned, as shown in Figure 3-1. The scales shown in the vertical and horizontal positions have 1/64-inch divisions and were used to increase the accuracy of scaling from the photograph.

As previously reported,⁽²⁾ certain sections of the cross section were found to closely correspond to arcs of circles as shown in Figure 3-2. In previous observations, it appeared that the tube wall between wires tended to form a circular cylindrical shape tangent to the wires. However, in most cases, there was a small section next to the wire having a smaller radius of curvature as shown in Figure 3-2. Note in Figure 3-3 that two small sectors of significantly different radius of curvature do exist but, in this case, are not adjacent to the wires but in the central region and they have larger rather than smaller radii of curvature compared to the surrounding area. It appears that little or no change in curvature occurred in the larger segments. The bending strain (ϵ_b) calculated from the change in curvatures is indicated in Figure 3-4 (left ordinate scale).

It might be hypothesized that if deformation were to continue, the radii of the four segments would remain about the same but the included angle would decrease as the included angle of the sector with the large radius increased. It would then be possible at some later point in time after more deformation to reach a configuration like that previously observed, Figure 3-2.

The interior surface tensile strains caused by bending approached 50% in the earlier test since that wall was observed to form closely around the small diameter wire. As shown in Figure 3-1, it does not appear that in this case the wall conforms to the more gentle curvature at the oval wires used in these tests or even approaches flattening (flattening produces about 6%

TYPICAL
OVAL
WIRE
POSITION

TEMPERATURE—1300°F
INITIAL HOOP
STRESS—17,500 psi

200 psi PRESSURE
DROP INDICATED AT
108 hours

TOTAL TEST TIME—301 hours
NO FAILURE

IIX

FIGURE 3-1. WIRE CONSTRAINED SPECIMEN CROSS SECTION

WIRE
CONSTRAINT

(NOT TO SCALE)

FIGURE 3-2. CROSS SECTION OBTAINED IN PREVIOUS RESULTS

FIGURE 3-3. SPECIMEN CROSS SECTION PROFILE

FIGURE 3-4. BENDING AND TOTAL SURFACE STRAIN AROUND CIRCUMFERENCE OF THE SPECIMEN

strain). This fact would discount the claim of strain reduction because of the oval wire. It would appear that the curvature of the restraint surface did not matter since the tube did not conform to it anyway. Perhaps after more deformation such as described above, a point would be reached where conformation would occur.

As previously reported, ⁽²⁾ the 7.9% average hoop strain (ϵ_{bulge}) obtained from geometric relations did not account for any uniform expansion required to take up the tolerance between the thick walled test channel, the wire, and the specimen. This additional strain, ϵ_w , can be obtained from $\Delta D/D$ measurement across the specimen under the wires. This dimension taken from the photograph in Figure 3-1 indicates that take up strain would be about 2.8%. Therefore,

$$\text{Total membrane strain } (\epsilon) = \epsilon_{\text{bulge}} + \epsilon_w = 10.7\%$$

This compares closely with the value of 10.6% previously reported for the unrestrained specimen tested at the same stress level.

To produce a curve of total surface strain, ϵ_T , it is necessary to add mean hoop strain, ϵ_m , to the above bending components, ϵ_{bend} , as previously explained. This has the effect of placing a 10.7% bias on the curve of bending strain and is shown as the ordinate to the right in Figure 3-4. It is evident that the largest total surface tensile strain (larger by several percent) occurs at the inner surface under the wire, an area not under the influence of sodium in a sodium loop test.

The study of deformation characteristics will continue on results obtained from specimens in the 1000-hour sodium loop seasoning run 6R-1.

3.1.2 Loop 6R—Experimental Concept, Run 6R-1

Run 6R-1 is a combination-loop seasoning, and specimen checkout test, designed to run at 1300° F sodium temperature for ~1000 hours. The test section (eight parallel tubes) contains 16 specimens of 0.250 inch outside diameter $\times$ 0.015 inch wall annealed Type 316 stainless steel, seamless, dynamically (cyclic) pressurized pins. Four hoop stress levels (4 specimens/level) are being evaluated:

- a. 19,000 psi maximum
7,000 psi minimum
- b. 21,000 psi maximum
7,000 psi minimum
- c. 23,000 psi maximum
7,000 psi minimum
- d. 25,000 psi maximum
7,000 psi minimum

There are two specimens/stress level with wire constraint and two specimens without constraint.

3.1.3 Preliminary Test Results

One high stress (25,000 psi) specimen of each configuration failed after ~49 hours at operating conditions. The leaks were apparently small, because by the time failure was indicated, two failures were found. Therefore, the failure time is the same for all practical purposes. The test is set up to depressurize the companion specimen of any failed specimen, thereby providing a sample for examination (hopefully just prior to possible failure) at the conclusion of the run.

The next failure occurred at ~87 hours in the wire-constrained specimen at 23,000 psi. Following this at ~96 hours, the non-constrained 23,000 psi specimen failed. Since the last failure, ~150 additional hours of operation have been achieved without further failure.

The stress levels chosen for run 6R-1 were based on mean stress for lack of a better base point. This reasoning was outlined in the Quarterly Progress Report No. 3.⁽³⁾ However, to achieve mean stress levels up to 16,000 psi (thought to be necessary for failure in <1000 hours) it was necessary to exceed the 1300° F yield strength of the Type 316 stainless steel (20—21,000 psi). It is obvious from the failures to date that stress-cycling over the yield strength has an adverse effect on the time-to-failure. The two remaining stress levels, being at and below the yield strength, should produce some interesting comparisons.

3.2 SUBTASK 2.1.3—LOOP PRESSURIZATION SYSTEMS

Fabrication of specimens for run 6R-1 (in-sodium) was completed. The construction of the duplex specimens, as shown in Figure 3-5, allows very limited passages for gas flow. To determine that adequate flow would be achieved during cyclic testing, these specimens were pretested, utilizing strain gage methods, to ensure that all specimens responded to gas pressure within 3 seconds. Seven dual (tandem) specimens and one set (pair) with machined-in "defects" were installed in the test section of Loop 6R. The defected specimens were used to test for gas leak detection in-sodium and instrumentation for loop isolation (see Figure 3-6). This

instrumentation was tested by allowing argon gas to leak into the test section through the slow leak "defect" over approximately a 30-minute period. This leak was detected by a low-pressure switch (PS No. 1) on the leak detection manifold and by high pressure switches (PS No. 2, PS No. 3) on both the hot leg and cold leg surge tanks of the sodium system. Level changes in both surge tanks were also noted. The affected pressure switches, operating independently, each acted to isolate the sodium loop from further gas input. In a second test, high pressure argon (3000 psi) was admitted to the sodium loop through the simulated "burst-specimen" defect. Within 2.5 seconds sodium flow was seriously perturbed and pressures in both hot leg and cold leg surge tanks began to rise. Flow signal deviation circuits and surge tank pressure switches, again operating independently, isolated the loop from further gas input. In both tests, gas venting circuits were automatically activated to vent high pressure gas from all specimens down to approximately 100 psi. This minimum pressure is required to prevent sodium from entering the failed specimens.

These tests "in-sodium" indicated that slow gas leaks from failed (cracked) specimens and rapid gas leaks from "blown" or ruptured specimens would be detected and would cause the sodium loop to be isolated from further gas input. It was substantiated that loop integrity would be well protected by the leak detection devices currently installed.

Figure 3-7 shows the complete gas pressurization system, including the control and instrumentation panel, gas connection area to loop, and the high pressure pump and gas plenum tanks.

FIGURE 3-5. DYNAMICALLY PRESSURIZED SPECIMENS

NOTE:

1. FOR DESCRIPTION OF PRESSURIZATION SYSTEM, REFER TO GEAP-5546 SECTION III, PP 38-41

FIGURE 3-6. ADDITION OF PRESSURIZED PIN FAILURE DETECTION AND SHUTDOWN SYSTEM TO LOOP 6R

GEAP-5602

NEG. No. A-68200

FIGURE 3-7. GAS PRESSURIZATION SYSTEM

SECTION IV

TASK 3—SODIUM CHEMISTRY4.1 SUBTASK 3.1.2—IN-LINE
DISTILLATION

The sodium in-line distillation station is shown in Figure 4-1. The station was received in San Jose on January 26 and is being prepared for operational checkout. A functional description of the distillation station follows.

4.1.1 General

The sodium in-line distillation station is capable of receiving sodium from a loop main stream at sodium temperatures up to 1400° F. In its present form it will accept sodium in batches of 50 gm and distill it automatically. It will span 20 cycles distilling a total of 1 kg per operation to concentrate impurities present.

The control console, Figure 4-3, has been designed to permit either manual or automatic operation. The master panel on the console receives signals from the weighing transducer (Figure 4-2), pressure switch and vacuum gauges. The master panel will control the sodium fill valve, vacuum pump, and crucible heater (Figure 4-2). The master panel is capable of being set to permit up to 20 cycles of automatic operation with each cycle capable of distilling 50 gm of sodium.

4.1.2 Preoperational Procedure

Following a thorough cleaning operation and bake out the station will be connected to the sodium loop to be sampled. Helium will be back purged to relieve the vacuum and raise the internal pressure to approximately 1 atmosphere. At this time with the fill nozzle in the circulating position, Figure 4-1, and sodium loop supply and return

valves open, the sodium fill valve may be manually operated and sodium circulated through the system bypassing the crucible, cleaning the lines, and raising the supply lines to isothermal conditions. When these conditions are reached, sodium flow is secured and the fill nozzle switched to discharge into the crucible. The system is isolated from the loop on the return leg and is now ready for automatic operation.

4.1.3 Automatic Operation

All controls are placed in the "on" position and the vacuum valve switch and sodium valve control switch are placed in the "automatic" position. The counter and timer are preset and the system energized. It will then operate through the preset number of cycles and the crucible heater will remain on through and beyond the last cycle to ensure complete distillation. When the timer runs its course the heater will be deenergized. The vacuum system will continue to operate and maintain the distillation pressure until the operator removes the crucible to the glove box and encapsulates it. The operator may now secure the system or prepare for another run.

4.1.4 Operational Sequence

The automatic sequence commences with a helium filled system at atmospheric pressure. The events are as follows: The controls are placed in the automatic mode and the distillation heater is energized and the temperature control adjusted to the operational setting. The unit is then energized and the weighing transducer will signal an empty crucible. This signal will open the sodium fill valve and commence filling the

FIGURE 4-1. SODIUM DISTILLATION UNIT

FIGURE 4-2. BALANCE SYSTEM FOR SODIUM DISTILLATION CONTROL

FIGURE 4-3. CONTROL CONSOLE FOR IN-LINE DISTILLATION

crucible. The weighing transducer will signal a full and commence filling the crucible. The weighing transducer will signal a full crucible, closing the sodium valve and starting the vacuum system. The vacuum system will begin pumping down to the distillation pressure and when it is reached the vacuum pressure gauge will signal the helium bleed valve to open permitting a constant bleed ahead of the pump thereby maintaining a steady distillation pressure. A manually operated throttle valve is located between the bleed valve and the pump to permit adjustments to maintain a steady pressure and avoid oscillations because of a hunting pump. As the sodium distills, the weighing transducer will supply a continuous signal to a strip recorder while concurrently indicating on its control panel. When the "empty" signal comes on, the counter will advance one count, the helium vent valve will open to raise the pressure to atmospheric (to avoid flashing during the next fill cycle), the sodium fill valve will open, and the sequence is repeated. On the final crucible fill cycle, the timer is actuated. On a signal from the weighing transducer that the crucible is empty, the helium vent valve is kept closed by an overriding signal from the timer and the system maintains temperature and pressure until the timer has zeroed out. At this point the signal from the timer interrupts power to the heater, resets the counter and timer, and shifts from the automatic mode of operation. The vacuum pump, vacuum valve, pressure switch, and helium bleed valve continue to function until the operator assumes control of the system for removal of the crucible.

The operator may secure the system following removal of the crucible or insert a new crucible and commence another distillation run.

4.2 SUBTASK 3.1.3—GAS SAMPLING ANALYSIS (CHROMATOGRAPH)

The primary objective is to establish an in-line gas analysis system by chromatography. This in-line system is essentially an adjunct to the operating sodium loops. The main function of the transport system is to continuously deliver loop surge tank cover gas to the gas analyzer (chromatograph). Because of the relatively small cover gas volume (3000 cc) in one surge tank, the transport system is designed to be an extension of the surge tank rather than merely a gas removal system. This requires that the cover gas be circulated continuously in the transport system and then returned to the same surge tank. A small portion of gas, approximately 10 cc, is removed at the chromatograph six-way valve and after analyzation is vented to atmosphere. An increased volume of gas of approximately 5000 cc is attributed to the surge tank vapor traps and tubing necessary to transport the surge tank gas to the analyzer. Hence, the total cover gas volume is increased from 3000 cc to 8000 cc. To ensure that the pumped cover gas within the transport system is typical to surge tank cover gas, the outlet from the surge tank (suction to pump) is placed near the top of the surge tank while the return to the surge tank (pump discharge) is located at no more than 1.0 inch above the highest reading on the surge tank level indicator. Figure 4-4 depicts the transport system. Basic features of the design include (1) complete isolation of gas input sources so that no cross-contamination may occur; (2) sodium loop surge tank pressure protection in the event of equipment malfunction; (3) gas flow and gas pressure monitoring instrumentation to aid in evaluating chromatograph output; (4) complete flexibility for dismantling or

FIGURE 4-4. COVER GAS CIRCULATION AND SAMPLING SYSTEM

removal of any portion of the analysis and transport system without affecting sodium loop operation; and (5) adaptability of the system to utilization of the single chromatograph by many different loops by installation of the "alternate group valves" (Figure 4-4).

Major components of the gas transport system include two vapor traps, "dry" zero leakage pump (60 cc/min) and fail-safe positive pressure protection devices for each surge tank.

The chromatograph itself has been modified to permit detection of nitrogen and hydrogen impurities in the sampled argon cover gas. Capability of the chromatograph may be extended to analyze for methane and carbon monoxide impurities with some alterations of sequencing and readout instrumentation. With present instrumentation, it is possible to analyze pumped cover gas alternately from the Loop 8 hot and cold leg surge tanks once every 20 minutes.

4.3 HYDROGEN DISTRIBUTION STUDIES

The previous reports in this series described the results of experiments which

indicated that hydrogen compounds, namely sodium hydride and hydroxide, were the major constituents in deposits collected in the mass transfer loop solids collector at temperatures of 400° to 700° F.

In a continuation of this study, hydrogen was either added to or removed from the loops in an attempt to corroborate the previous findings. These experiments provide initial information on the distribution and behavior of hydrogen in these systems and suggest control measures which may be used to limit hydrogen's undesirable effects.

4.3.1 Experimental

Hydrogen was deliberately added to Loops 8 and 9 from their respective cold traps, since analyses of cold trap deposits (previously discussed in Quarterly No. 1, GEAP-5508, Section V) have shown it to be a collector of NaH and NaOH. The Loop 9 test consisted of raising the temperature of the cold trap in increments from 275° to 400° F. The effects on the plugging temperatures and on the hydrogen content of the cover gas and sodium are shown in Table 4-1.

TABLE 4-1
EFFECTS OF RAISING LOOP 9 COLD TRAP TEMPERATURE

Cold Trap Temperature (° F)	Hydrogen Content Surge Tank Gas		Hydrogen Content Cold Leg Sodium (ppm)	Plugging Indicator Flowbreaks (° F)
	Cold Leg (vpm)*	Hot Leg (vpm)*		
275	< 2	< 2	6.2	470, 260
315	2	< 2	9.0	640, 460, 355
350	120	5	7.5	675, 430, 365
400	660	110	15.5	700

* vpm = parts per million by volume

The results show a rise in the concentration of hydrogen in both the cover gas and sodium, and the appearance of initial flow reductions through the plugging indicator in the temperature range 600° to 700° F.

In the Loop 8 study, the cold trap was raised from an initial temperature of 310° to 425° F. The analyses of the surge tank cover gas and the temperatures of the flow reduction in the plugging indicator are shown in Table 4-2.

TABLE 4-2
EFFECTS OF RAISING THE LOOP 8 COLD TRAP TEMPERATURE

Cold Trap Temperature (° F)	Hydrogen Content Surge Tank Gas		Plugging Indicator Breaks (° F)
	Cold Leg (vpm)	Hot Leg (vpm)	
310	20	<2	380, 275
350	100	85	385, 270
425	150—250*	100—150*	480

* Results of several samples

These data also indicate an increase in the hydrogen in the cover gas and an increase in the initial flowbreak temperature. Analyses of the sodium have not been completed, but preliminary results show a slight trend for the hydrogen content to increase also.

The results appear to show that the hydrogen content of Loop 8 is lower than that of Loop 9. The reason for this is not yet clear but may be explained after further studies. It is also interesting to note that even at cold trap temperatures of only 310°, hydrogen appears in the cold leg surge tank cover gas. This suggests that methods, in addition to cold trapping, probably will be required to limit hydrogen levels in sodium loops.

In a subsequent phase of the Loop 8 test, the cold trap was valved off and the surge tank cover gas was diluted with pure argon by partial evacuation and backfilling. The effects on the hydrogen content in the

argon and on the flowbreak temperatures are illustrated in Figure 4-5.

The effect of the purging cycles is seen to be a decrease in the hydrogen content and in the initial flow break temperature. (The temperature of the second break, attributed to oxygen, as expected, did not change.) It was noted that the initial purging steps led to a sudden increase in hydrogen content. This is attributed to the release of hydrogen from hydrogenous deposits on the cooler portion of the surge tank wall coming into contact with the hot sodium as a result of change of sodium level because of the argon venting. This effect may be similar to that noted in the Fermi Reactor secondary system where hydrogen in the cover gas was being monitored.

4.3.2 Conclusions

These experiments show that the addition or removal of hydrogen does

FIGURE 4-5. EFFECT OF HYDROGEN REMOVAL ON PLUGGING METER INITIAL FLOW BREAK TEMPERATURE

affect the flow through the plugging indicators (initial break) and corroborates the results obtained by direct examination of deposits found in the solids collectors, i. e., that sodium hydride and hydroxide were present.

The experiments also confirm previous observations that the cold trap is a source of hydrogen and indicate that other cool regions may also contain deposited hydrogen compounds. The potential adverse effects of hydrogen compounds in large systems (for example, flow blockage and confusion of detection of sodium to water leaks) warrant additional study of hydrogen control and elimination.

4.4 IMPINGEMENT SAMPLES

Analyses of the impingement sample residues continued. Total carbon determinations showed that deposits contained more than 2% of this element, as compared to its initial concentration in the steel of 0.05%. X-ray diffraction indicates that at least part of the carbon is present, a metal carbide of the form $M_{23}C_6$, where M is the metal constituent. Attempts to further characterize the carbon species are in progress. Simple cyanides were present, but only at the detection limit, about 1 ppm.

An inconsistency in the data has become apparent. It has been observed that the majority of the material appears to be magnetic; yet x-ray data indicate that the crystals are austenitic. Attempts to reconcile this point continue.

As a control for this study, corrosion samples of normal configuration have been exposed in Loop 8 and are being examined by the same techniques applied to the impingement samples.

4.5 ANALYSES FOR GASEOUS CARBON SPECIES

An early attempt⁽²⁾ to measure carbon-containing species in Loop 5 cover gas was terminated because of a loop pump failure. Recently, a second test was performed. Both methane and carbon dioxide were detected as impurities in the helium cover gas. The presence of large quantities of argon (previously used as a cover gas and incompletely removed) prevented analysis for oxygen, which was to serve as an indicator for leakage of the gas concentration system. For this reason, and particularly because it had been necessary to pressurize the loop with fresh helium shortly before the test, quantitative estimates of the concentration of these two species will require another run.

SECTION V

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8. GEAP-4183—Dunn, E.L., "Sodium Mass Transfer: VI Statistical Correlation of 1961 - 1962 Corrosion Data," February 1964.
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CONTRIBUTORS

The following people contributed work performed on the Sodium Mass Transfer Program during the period ending February 1968.

Task 1—Corrosion Mockup Studies

J. T. Cochran	L. E. Pohl
F. A. Comprelli	M. K. Schad
W. J. Margetanski	A. G. Steamer
L. E. Pohl	G. P. Wozadlo

Task 3— Sodium Chemistry

W.V. Cummings	W. D. Reynolds
D. Dutina	W. W. Sabol
L. Pohl	J. L. Simpson

Task 2—Cladding Materials Testing

C. F. Barrett	L. E. Pohl
F. A. Comprelli	D. J. Vogelhuber
M. D. Dermer	G. P. Wozadlo
D. P. Hines	M. D. Moore
W. J. Margetanski	

General Program and Consulting

G. D. Collins	C. N. Spalaris
L. F. Epstein	R. S. Young
R. W. Lockhart	E. L. Zebroski

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Glen W. Wensch, Chief Liquid Metal Project Branch Div. of Reactor Development & Tech. AEC Headquarters Washington, D.C. 20545	2	N. Grossman, Chief Engineering Development Branch Div. of Reactor Development & Tech. AEC Headquarters Washington, D.C. 20545	1
Ralph H. Jones, Components Branch Div. of Reactor Development & Tech. AEC Headquarters Washington, D.C. 20545	1	W.R. Kornack LMFBR Branch Div. of Reactor Development & Tech. AEC Headquarters Washington, D.C. 20545	1
J.A. Lieberman, Assistant Director for Nuclear Safety Div. of Reactor Development & Tech. AEC Headquarters Washington, D.C. 20545	1	M.J. Whitman Assistant Director for Program Analysis Div. of Reactor Development & Tech. AEC Headquarters Washington, D.C. 20545	1
S.A. Szawlewicz, Chief Research and Development Branch Div. of Reactor Development & Tech. AEC Headquarters Washington, D.C. 20545	1	L. J. Colby Chemistry and Chemical Separations Branch Div. of Reactor Development & Tech. AEC Headquarters Washington, D.C. 20545	1
J.M. Simmons, Chief Fuels and Materials Branch Div. of Reactor Development & Tech. AEC Headquarters Washington, D.C. 20545	1	R. Sweek LMFBR Program Manager, LMFBR Branch Div. of Reactor Development & Tech. AEC Headquarters Washington, D.C. 20545	1

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Assistant Director for Project Management		Los Alamos Scientific Laboratory	
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AEC Headquarters			
Washington, D.C. 20545		G. Waterbury	1
		Los Alamos Scientific Laboratory	
		Los Alamos, New Mexico 87544	
M. Booth, Chief	2	L.E. Whinery	1
Systems Engineering Branch		Los Alamos Scientific Laboratory	
Div. of Reactor Development & Tech.		Los Alamos, New Mexico 87544	
AEC Headquarters			
Washington, D.C. 20545		E.R. Astley, FFTF Project Manager	3
Chief, Administrative Budget and		Battelle-Pacific Northwest Laboratory	
Program Analysis	1	Richland, Washington 99352	
Div. of Reactor Development & Tech.		Supervisor of Configuration Control	2
AEC Headquarters		FFTF Project	
Washington, D.C. 20545		Battelle-Pacific Northwest Laboratory	
		Richland, Washington 99352	
J.H. DeVan	1	R.W. Dickinson, Director, LMEC	1
Oak Ridge National Laboratory		Atomics International	
P.O. Box Y		P.O. Box 309	
Oak Ridge, Tennessee 37830		Canoga Park, California 91304	
J. White	1	J. Droher, Director LMIC	1
Oak Ridge National Laboratory		Atomics International	
Oak Ridge, Tennessee 37830		P.O. Box 309	
		Canoga Park, California 91304	
R.E. MacPherson	1	C.E. Weber	3
Oak Ridge National Laboratory		Atomics International	
Oak Ridge, Tennessee 37830		Canoga Park, California 91304	
D. Gurinsky	1	Technical Director	2
Brookhaven National Laboratory		Atomic Power Development Associates, Inc.	
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