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**SODIUM COMPONENTS
DEVELOPMENT PROGRAM
MASS TRANSFER INVESTIGATIONS
IN LIQUID METAL SYSTEMS
QUARTERLY PROGRESS REPORT
NO.3
SEPTEMBER-NOVEMBER 1967**

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. 3
September - November 1967

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ABSTRACT

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

Preparations were completed for testing of Type 304 stainless steel in a loop modified to simulate typical closed loop conditions of FFTF. The design details and corrosion test conditions are given along with a schematic representation of the modified test loop and specimen locations.

The successful attainment of long-term operation with heat fluxes on the order of 1×10^6 Btu/h-ft² with an electron bombardment heater simulating typical fuel cladding exposure in flowing sodium is reported. Stable heater operation was obtained for bulk sodium temperatures to 1200° F for ~1100 hours, and operation continues.

Work was completed on data analysis and reporting of sodium vapor transport and deposition rates and sodium fog production experiments in a refueling cell mockup containing a 4-foot diameter open pool of liquid sodium.

Short time stress-rupture tests of LMFBR cladding candidates weld drawn and annealed, were conducted to establish probable mode and relative rates of failures of tubing currently in test in sodium loops and irradiation capsules. Results indicate that weld drawn Types 304 and 316 stainless steels should yield representative and uniform properties. Type 347 stainless steel and Incoloy 800 in the weld drawn condition, because of localized seam weld deformation, do not produce homogeneous mechanical property data. Further use of these materials in weld drawn tubing form is discouraged and seamless materials have been procured for future tests.

Plugging mechanism studies in mass transfer loops confirm that hydrogen compounds are responsible for the high temperature break in the plugging indicator.

SECTION I

INTRODUCTION

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, $2\frac{1}{4}$ Cr-1 Mo, and 5 Cr- $\frac{1}{2}$ Mo- $\frac{1}{2}$ Ti 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 oxide impurity content, 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 tem-

perature profiles, loop geometry, and sodium purity controls established to represent large fast reactor environments better. Further tasks in sodium reactor technology have been added this year. These include 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 are being 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 oxide 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.

Subtask 1.2—FFTF Closed Loop Simulation

The initial objective of this task is 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. Peak

heat fluxes of 0.5 to 1.0×10^6 Btu/h-ft² will be used for some of these tests (with resultant increased surface temperatures). Tests will be conducted in Loop 9 containing a rebuilt section for the hot leg with corrosion potentials adjusted to bracket the range of calculated corrosion and deposition rates of closed loop design.

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 1.2. 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. A series of tasks were performed in the cell in an argon atmosphere above a 4-foot diameter pool of liquid sodium. These tasks included:

1. Establishing the heat load imposed on the cell by the open reactor at two sodium pool temperatures.
2. Investigation of the argon gas temperatures at various elevations above the open reactor sodium surface.
3. Performing sodium vapor transport tests at two sodium pool temperatures.
4. Investigation of sodium vaporization (smoking) characteristics versus fuel rod temperature for sodium wetted rods handling in the cell atmosphere.

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 6000 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 underway to permit the start of long-term stress rupture tests in fiscal year 1968.

Subtask 2.2—Localized Attack Studies

The investigation of localized intergranular phenomena is also underway as part of this task. The effect of stress is being evaluated by exposing statically pressurized specimens. A related study has been initiated on the effects of residual sodium contamination (or possible penetration of sodium into metal surfaces) on localized corrosion. Procedures and criteria for the cleaning and storage of samples to avoid post exposure corrosion by sodium oxide or sodium hydroxide, or both, are being established.

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.

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 have been installed in the SCTI test facility of LMEC by Atomics International.

1.1 REPORTS

Monthly report letters are prepared reporting progress in all tasks. Progress letters No. 1 through 98 covering work from July 1, 1959 through October 31, 1967 have been issued.

In addition to the progress letters, 28 topical reports and 2 quarterly progress reports covering test results have been issued (see Section VI).

This is the third quarterly progress report issued and covers 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

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 reactor primary 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 conceived 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.

Run 8-3 is currently operating with completion scheduled in December 1967. Data obtained in runs 8-1 and 8-2 have been analyzed and refined coefficients in the corrosion model reported.⁽¹⁾ Work is continuing to establish estimates of percent uncertainty in corrosion rates predicted by corrosion models developed from all data obtained from Loops 8 and 9 to date. Results of analyses will be given in subsequent reports.

2.1 SUBTASK 1.2—FFTF CLOSED LOOP SIMULATION

The initial objective of this task is 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 and peak heat fluxes in the range from 0.5 to 1.0×10^6 Btu/h-ft².

Loop 9, originally constructed as a mockup of a 1000 MW sodium system, is being modified to simulate a typical FFTF 10 MW closed loop. The mockup is designed on the basis that corrosion processes provide the same amount of change in solute, ΔS , (corroded material) in the sodium at all locations in the mockup as would be expected in the target system. The procedure consists of providing temperature conditions in each section of the mockup identical to the target system and then adjusting surface area of the specimens and velocity such that the calculated amount of material removed or deposited (per unit mass flow of sodium) is the same in the mockup and target. A detailed discussion of the corrosion and deposition process and methods of generating a mockup is contained in GEAP-4881.

The FFTF closed loop being modeled is shown schematically in Figure 2-1 and the system parameters are tabulated in Table 2-2. Type 304 stainless steel and a 1300°F maximum bulk sodium temperature were chosen as representative of the loops by PNL and DRDT.

The Loop 9 mockup of the FFTF closed loop is represented schematically in Figure 2-2 and the corrosion model data for this configuration is shown in Table 2-3 and plotted in Figure 2-3.

All piping modifications to Loop 9 have been made and electron beam heaters of the type described in subtask 1.3 ordered for use in the high heat flux section E₂.

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

TABLE 2-2
ESTIMATED REPRESENTATIVE PARAMETERS FOR
FFTF 10 MW CLOSED LOOP

Section	Temperature (°F)	Velocity (ft/sec)	Surface Area (in. ²)	$\overline{CR}$ * Estimate	$\overline{\Sigma\Delta S}$ * (ppb)
Inlet to D	—	—	—	—	0.029
D _{in} to Test (E ₁)	750—800	20/35	6,250	0.3	—
Test (fuel) (E ₂)	800—1300	~25	3770 total (293 pipe)	80	0.161
Test to D _{out} (F)	1300—1250	20/35	6,250	20	0.210
D _{out} to C _{out}	1250	30	1,150	15	—
C _{out} to B _{out}	1250	11	9,500	10	0.280
B to A (IHX)	1250—750	27	49,200	15	0.290/-0.261
A to B _{in}	750	10.5	5,770	1	—
B _{in} to C _{in}	750	10.5	9,500	0.5	—
C _{in} to D _{in}	750	29	1,150	0.7	-/0.029

* $\overline{CR}$ = average corrosion rate estimated for section in mg/dm²-month.
 $\overline{\Sigma\Delta S}$ = cumulative contributions of corrosion products.

TABLE 2-3
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	Oxide-Level (ppm)	CR (Estimate) (mg/dm ² -month)	ΔS (ppb)	ΣΔS (ppb)
Heater, 110 kW	800	982	5	0.0914				38	5	0.01995	0.00404	0.029
Samples I	982	982	5	6.55	0.805	0.669	3	114	5	1.43	0.0149	0.03304
Valve	982	982	2.8	1.04				11	5	0.092	0.000385	0.04795
Connecting Pipe	982	982	2.8	1.04				23	5	0.0917	0.00077	
Heater, 50 kW	982	1300	2.8	0.0544				24	5	0.1251	0.01695	
Samples II	1300	1300	2.8	1.73	0.75	0.500		40	5	7.34	0.0574	
Flowmeter B	982	1030	2.2	5.55				26	5	0.342	0.00075	
Valve	982	1030	2.2	2.31				14.5	5	0.0847	0.000467	
Connection Pipe	982	1030	2.2	2.31				59	5	0.071	0.00104	
High Heat Flux Heater ⁽¹⁾	982	1300	2.2	15				141	5	15.6	0.0398	
Connecting Pipe	1300	1300	2.2	0.82				89	5	3.4	0.075	
Cooler I	1300	1250	5	3.45	0.75			48	5	10.5	0.081	0.223
Samples IV	1250	1250	5	14	0.805	0.711		119	5	19.2	0.1323	0.3627
Samples V	1250	1250	5	14	0.805	0.711		119	5	7.74	0.055	0.41772
Cooler II	1250	1175	5	2.05	0.805			60	5	3.3	0.0266	0.44432
Connecting Pipe	1175	1175	5	1.31				10	5	0.158	0.00042	0.44852
Cooler III	1175	1100	5	1.31				45	5	1.29/-3.4	0.082/-0.0216	0.43512
Connecting Pipe	1100	1100	5	1.31				14	5	3.47	0.0138	0.42132
Cooler IV	1100	1025	5	1.31				45	5	3.52	0.048	0.37652
Connecting Pipe	1025	1025	5	1.31				14	5	2.92	0.0116	0.36492
Cooler V	1025	950	5	2.05	0.805			60	5	2.67	0.0339	0.33102
Sample X	950	950	5	0.91	1.334			26	5	2.55	0.01346	0.30756
Connecting Pipe and Main Flowmeter	950	950	5	0.788				28	5	1.45	0.01713	0.29043
Cooler VI	950	875	5	0.788				30	5	1.75	0.0337	0.25673
Sample XI	875	875	5	0.91	1.334			25	5	1.368	0.0192	0.23753
Cooler VII	875	800	5	0.788				30	5	1.515	0.0291	0.20843
Connecting Pipe	800	800	5	0.788				7.5	5	1.09	0.00522	0.20321
Pumps	800	800	5	17.8				425	5	13.65	0.1655	0.03771
Connecting Pipe	800	800	5	0.788				17	5	0.33	0.00369	0.03402

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

FIGURE 2-1. CLOSED LOOP DIAGRAM

FIGURE 2-2 LOOP ARRANGEMENT AND SAMPLE LOCATIONS

FIGURE 2-3. CORROSION PRODUCT PROFILE OF LOOP 9

2.2 SUBTASK 1.3—DEVELOPMENT OF HIGH HEAT FLUX HEATERS

The objective of this task is to develop the capability of maintaining about 1.0×10^6 Btu/h-ft² heat flux at a stainless steel to sodium interface with 1300°F bulk sodium temperature for times to 3000 hours.

Two types of heaters are currently in test, and a third (RF) type has been previously tested:

- a. Electron Bombardment—This has now become the reference method for the HHF test section in Loop 9.
- b. Resistance, Swaged—This is a longer range alternate that may be used as a lower cost backup for future tests of both FFTF and LMFBR corrosion characteristics.

RF induction—Preliminary tests with this concept have proven feasible, but high surface temperatures required (~1500°F) for 10^6 heat fluxes, and the high thermal stresses on the external piping make this approach less attractive for unattended operation.

2.2.1 Electron Bombardment Heater Testing Loop 1

The heater in test in Loop 1 (designated E-EB-1-1) reached 3056 hours of power operation as of November 20, 1967. Operating goals have been uniform performance at 1200°—1300°F bulk sodium output at a sodium velocity of 15 ft/sec. About 1083 hours were accumulated at these conditions with an average heat flux of 800,000 Btu/h-ft² or greater over the 10-inch heated length.

This heater is similar to the electron bombardment heater design described in Quarterly Report No. 1 except the cathode support structure was made from molybdenum-rhenium alloy instead of molybdenum.

2.2.2 Operating Characteristics

The electron bombardment heater performance is best characterized by a plot of log plate current versus log plate voltage. Figure 2-4 is typical of such a plot. The figure consists of three zones:

- a. Space charge limited emission zone in which the slope is 3/2. In this mode of operation an electron cloud surrounds the cathode and produces a space charge which limits the number of electrons emitted by the cathode.
- b. The transition zone between space charge limited emission and temperature limited emission. This zone is bounded on one side by the space charge limited zone with a slope of 3/2 and on the other side by the temperature limited zone with a slope of zero.
- c. The temperature limited zone. In this region the plate current is a constant which is independent of plate voltage and depends only on the temperature of the emitting surface. There is no space charge and all the electrons emitted flow immediately to the anode.

The controllable parameter which determines the point at which the heater will depart from space charge limited operation is the emitter temperature. Diode lines were run with constant filament current which produces constant cathode temperature. This requires accounting for both a-c and d-c components of current flowing through the filament. The heater is designed with the filament and cathode electrically continuous. The electrical arrangement is shown schematically in Figure 2-5. Anode to cathode beam current is fed to the cathode through a current divider which sends a portion of the current through the filament and the rest directly to the cathode. At present the current split is set so half of the beam current goes through the filament.

FIGURE 2-4. ELECTRON BOMBARDMENT HEATER FAMILY OF DIODE LINES

FIGURE 2-5 ELECTRON BOMBARDMENT HEATER CONTROL CIRCUIT

Specifically,

where

$$I_{ft} = I_f + 0.5 I_p = \text{Constant}$$

$$I_{ft} = \text{total filament current ac + dc}$$

$$I_f = \text{a-c component of filament current}$$

$$I_p = \text{d-c plate current.}$$

In Figure 2-4 data are plotted for four different values of I_{ft} ranging from 60 to 90 amperes. From these curves it may be seen that at 60 A the heater is operating in the temperature limited emission zone and that at 90 A operation departs from space charge limited operation at about 2.7 kW, but is still in the transition zone at a power level of 22 kW which is just above the nominal heater rating of 20 kW.

2.2.3 Tripout Investigation

Repetitive trips of the beam power circuit were a cause of annoyance though they did not appear to cause any damage to the heater. These trips were occurring at a rate of about seven per day, and two sources appeared to be responsible. First, true arcs between the cathode and the anode could cause activation of the crowbar with subsequent opening of the circuit breaker in response to current meter relay signals. Secondly, power supply line transients could cause activation of the crowbar circuit in the absence of a true arc. A tripout study was performed which showed that arcing in the tube was a secondary cause and that most of the trips were caused by a loose connection in the main three-phase power supply to the plate power rectifier circuit. Since the line was repaired a continuous run of 264 hours has been obtained.

2.2.4 Power Control Circuit

An automatic control system has been designed and built to control the power level to permit continuous unattended operation.

As originally conceived, the control system sensed the heater internal vacuum and raised power to a preset limit if the heater pressure was below a lower set point, and reduced power when the heater pressure exceeded an upper set point. This system did not achieve the desired results because the large vacuum pump installed on the present heater (8 liters/sec) caused the pressure to be nearly independent of power. The control system was then modified to sense plate current and to vary the control signal to the saturable reactor which was the primary source of plate power.

In October the power control circuit was modified to allow control of the plate voltage as well as the plate current. The modification was accomplished by installing a meter relay in the plate voltage circuit. An independent and adjustable upper power limit is provided which prevents applying unlimited power to the heater in the event of a control system malfunction.

2.2.5 Heater Internal Pressure as a Function of Cold Trap Temperature

On two occasions, during overnight operation, the cold trap cooling system in Loop 1 failed causing the cold trap temperature to approach bulk sodium temperature. These occurrences were both accompanied by a rise in the tube pressure of several orders of magnitude.

On the basis of these observations, it is thought that a tube of this type might function as an impurity detector as well as an HHFH. With this in mind, data were taken to determine the relationship between cold trap temperature and tube pressure.

The cold trap is in a bypass leg in a sodium loop through which a portion of the bulk sodium may flow. If the cold trap temperature is raised, some of the precipitated impurities redissolve and begin to enter the sodium system again. This increase in

impurities has been sensed by watching the electron bombardment heater tube pressure as can be seen in Figure 2-6. The data points on this plot were obtained by varying the cold trap temperature from 400° to 260°F in steps, and observing the tube pressure at each step. There is a sizable lag between cold trap temperature change and tube pressure change, so each step in temperature was maintained for about 15 minutes, and pressure was read immediately before each change in temperature.

Similar data taken with power supplied to the tube also show pressure dependence on cold trap temperature but it also indicates some dependence upon bulk sodium temperature and the power level of the tube. In general, heater operating pressure is in the range from 10^{-7} to 10^{-6} torr.

2.2.6 Resistance Element Heater

A swaged construction resistance type heater element is being tested as an alternate design which may have potential usefulness to the FFTF and LMFBR programs. This heater has a 0.25-inch outside diameter, a heated length of 15 inches, and a design rating of 24.7 kW.

Three heaters of this type were procured for preliminary tests to evaluate the concept. A sodium loop has been modified to permit testing of this type heater and evaluation tests have been performed with the available units. One heater failed by cracking of a closure weld which allowed sodium to enter and short out the heater element. The other two units were tested with partial success; each achieved approximately 80 percent of rating or a surface heat flux of 800,000 Btu/h-ft² at a bulk sodium temperature of about 850°F. In both cases the heaters failed by melting of the resist-

ance element. In neither case was the damage extensive enough to breach the ground insulation and cause failure of the heater sheath.

2.3 SUBTASK 1.4—FFTF SUPPORT, REFUELING CELL MOCKUP

The technical report of the work performed under this task has been completed (GEAP-5560). A brief discussion of the results is as follows:

a. Vessel Cover Gas Temperature Profile

A consistent profile of the cover gas temperature was obtained with several sodium pool temperatures. Within 2 inches above the pool the argon cover gas temperature dropped below the freezing point of sodium (a ΔT of ~250°—300°F) and proceeded to cool down another 80°F in the next 5 feet of elevation.

b. Sodium Vapor Deposition Rates

At sodium pool temperatures of 450° and 500°F deposition rates are negligible and are on the order of 10^{-8} milligrams/centimeter square-hour measured following an exposure of the aerosol collectors for 300 hours.

c. Vapor Deposition Rates from a Heated Fuel Bundle Mockup Out-of-Core

The handling of high heat generating fuel elements out-of-core is the largest single source of sodium aerosols. The mockup bundle was inserted and removed from the sodium at surface temperatures precalibrated from 500°—1000°F with maximum surface temperature of 800°F with fully plated rods. The aerosol generation rate was calculated at 1.9 grams/minute.

FIGURE 2-6. HEATER INTERNAL PRESSURE VERSUS SODIUM COLD TRAP TEMPERATURE

d. Heat Power Losses from the Molten Pool to the Cell

The results of this work are influenced by the cell environment. The test cell is a large (8500 ft³) uninsulated steel shell partly sheltered within the test building and partly located outside the building. Ambient environmental conditions had a strong added effect on cell bulk gas temperatures. Future tests will include insulation or shim heaters to permit control of ambient cell temperatures.

e. Sodium Vapor Transport for one Arrangement and Ventilation Pattern

During the majority of the test period argon circulation was provided by the 200 CFM purification flow entering through a single 4-inch pipe at approximately 38 ft/sec. Under these conditions, sodium deposition rates were measured at 10⁻⁸ milligrams/centimeter square-hour following an exposure of 300 hours.

SECTION III

SUBTASK 2.1—MECHANICAL PROPERTIES TESTS OF CLADDING CANDIDATES

3.1 THE SHORT-TIME BIAXIAL STRESS-RUPTURE TEST AS A TOOL FOR DETERMINING WELD DRAWN TUBING ACCEPTABILITY

The pressure tubing specified for mechanical property testing has been weld drawn for better dimensional control (primarily wall thickness). As a result of the seam-weld failures during the initial stress-rupture testing, ⁽¹⁾ seamless tubing has been specified for future mechanical property testing.

Because of the time delay in receiving the seamless tubing, short-time (<500 hours) biaxial stress-rupture tests were run on the annealed 0.250-inch outside diameter × 0.015-inch wall pressure tubing. The knowledge of seam-weld failure tendencies for each weld drawn tube material on-hand was needed to predict failures of tubing already in test, to determine if any of the tubing could be used in further testing, and to examine this test technique as an acceptance criterion for experimental materials.

3.1.1 Test Conditions and Results

The materials (0.250-inch outside diameter × 0.015-inch wall) were weld drawn and annealed Types 304, 316, and 347 stainless steels, and Incoloy 800. Chemical compositions and room-temperature mechanical properties are shown in Tables 3-1 and 3-2.

The tests were conducted in a tube furnace in flowing argon at 1300°F. The argon flow was restricted by the specimen holder design, resulting in air contamination and specimen oxidation. This oxidation did not affect the evaluation of specimen properties. The specimens, Figure 3-1, were internally pressurized and welded under pressure. The samples were visually examined at ~1 week intervals to determine failures. The results are shown in Table 3-3. It can be seen that the hoop stress levels were too high in most cases and the majority of the specimens failed in less than one week.

TABLE 3-1
CHEMICAL COMPOSITION OF THE
0.250-INCH OUTSIDE DIAMETER × 0.015-INCH WALL PRESSURE TUBING

Material	C	Mn	Si	Ni	Cr	Fe	Co	Mo	Cb and Ta	Al	Ti	S	P	Cu
Type 304 Stainless Steel	0.04	1.81	0.44	9.6	18.4	Bal.	-	-	-	-	-	0.030	0.021	-
Type 316 Stainless Steel	0.06	1.81	0.56	12.3	16.7	Bal.	-	2.78	-	-	-	0.010	0.024	-
Type 347 Stainless Steel	0.06	1.77	0.52	9.8	17.5	Bal.	-	-	0.82	-	-	0.013	0.022	-
Incoloy 800	0.04	0.82	0.60	34.3	21.2	Bal.	0.047	-	-	0.113	0.443	0.019	-	0.43

TABLE 3-2
ROOM TEMPERATURE PROPERTIES OF THE ANNEALED
0.250-INCH OUTSIDE DIAMETER × 0.015-INCH WALL PRESSURE TUBING

Material	UTS, kpsi	0.2% YS, kpsi	% E, 2 in.	ASTM Grain Size No.
Type 304 Stainless Steel	87.5	38.4	59	7
Type 316 Stainless Steel	86.5	41.6	50	5.5
Type 347 Stainless Steel	91.6	41.7	48	10
Incoloy 800	82.2	65.8	40	5.5

TABLE 3-3
RESULTS OF THE SHORT-TIME BIAXIAL
STRESS-RUPTURE TESTS IN ARGON AT 1300°F

Material	Hoop Stress kpsi	$\Delta D/D, \%$		Location of Failure and Approximate Time to Failure, hours	
		(At Failure)	(General)		
Type 304 Stainless Steel	12	9.2	5.2	Tube	<166
	15	10.0	5.6	Tube	<166
	18	Burst	-	Tube	<166
Type 316 Stainless Steel	14	17.6	4.8	Tube	460-820
	17	30.4	8.4	Tube	<166
	20	28.8	8.0	Tube	<166
Type 347 Stainless Steel	10	N. F.	1.3		668
	14	3.3	3.3	Weld	336-500
	16	4.2	4.2	Weld	170-336
Incoloy 800	14	1.2	1.2	Weld	168-311
	17	1.6	1.6	Weld	<168
	20	2.8	2.8	Weld	<168

FIGURE 3-1. STATICALLY PRESSURIZED SPECIMEN

3.1.2 Metallurgical Examination

3.1.2.1 Type 304 Stainless Steel

The typical Type 304 stainless steel failure area (tube) is shown in Figure 3-2. There are grain boundary separations throughout the cross section with cracks propagating from the outside diameter. There was carbide precipitation in the grain boundaries and slight air oxidation of the outside diameter surface in the outside diameter grain boundaries, and along the outside diameter stress-rupture cracks, Figures 3-2 and 3-3.

3.1.2.2 Type 316 Stainless Steel

The typical Type 316 stainless steel failure area (tube) is shown in Figure 3-4. There are grain boundary separations throughout the cross section. In general (away from failure area), the cracks propagated from the outside diameter surface. There was precipitation in the grain boundaries (carbide and/or sigma), Figures 3-4 and 3-5, and precipitation in the grains (probably sigma) increasing with exposure time, Figures 3-5 and 3-6. Slight air oxidation effects are observed, Figure 3-4, in the outside diameter surface grain boundaries, and along the outside diameter stress-rupture cracks.

3.1.2.3 Type 347 Stainless Steel

The Type 347 stainless steel stress-rupture failures were in the seam weld, Figure 3-7. The deformation (grain boundary separations) was localized in the weld with the cracks appearing to propagate from the inside diameter, Figure 3-8. There was sigma precipitation in the grain boundaries with an apparent absence of carbide precipitates, Figure 3-7.

3.1.2.4 Incoloy 800

The Incoloy 800 stress-rupture samples all failed in the seam weld, Figure 3-9. The deformation (grain boundary separations) was localized in the weld. The cracks appeared to propagate from the inside diameter. There was carbide precipitation in the grain boundaries and a trace of an apparent substructure, Figures 3-9 and 3-10.

3.1.3 Discussion

The lack of reference stress-rupture data led to the overpressurization of the samples. This did not interfere with the major purpose of the test, namely, determination of failure modes. However, the high pressures did rule out obtaining any meaningful time-to-failure data because 70 percent of the failures occurred during the first time interval.

The failure modes seen after the first stress-rupture test in sodium (run 5R-1)⁽¹⁾ were duplicated exactly in these short-time tests in air/argon, thus relieving sodium as a direct cause. Types 304 and 316 stainless steels failed in the tubes after significant expansion with Type 316 stainless steel showing the greater expansion tendencies. Type 347 stainless steel and Incoloy 800 failed in the tube seam welds with little or no expansion, and all of the creep occurred in the welds. This indicates that the short-time biaxial stress-rupture test can be used as an effective means of determining the transverse properties of weld drawn tubing (seam-weld failure tendencies). Therefore, this relatively rapid test will indicate whether or not the weld and heat-affected zone have mechanical properties representative of the bulk material. It has been shown, using the same materials,⁽²⁾ that dynamic tube-burst tests do not produce the same failure characteristics as the lower strain-rate, stress-rupture test.

10% OXALIC

200X

FIGURE 3-2. STRESS-RUPTURE FAILURE AREA (TUBE) OF TYPE 304 STAINLESS STEEL AFTER EXPOSURE TO 1300°F ARGON

10% OXALIC

500X

FIGURE 3-3. TYPICAL STRUCTURE (TUBE) OF TYPE 304 STAINLESS STEEL AFTER 166 HOURS EXPOSURE TO 1300°F ARGON

10% OXALIC

200X

FIGURE 3-4. STRESS-RUPTURE FAILURE AREA (TUBE) OF TYPE 316 STAINLESS STEEL AFTER EXPOSURE TO 1300°F ARGON

10% OXALIC

500X

FIGURE 3-5. STRUCTURE (TUBE) OF TYPE 316 STAINLESS STEEL AFTER 166 HOURS EXPOSURE TO 1300°F ARGON

10% OXALIC

500X

**FIGURE 3-6. STRUCTURE (TUBE) OF TYPE 316 STAINLESS STEEL
AFTER 820 HOURS EXPOSURE TO 1300°F ARGON**

HCl+H₂O₂

500X

**FIGURE 3-7. STRUCTURE AND STRESS-RUPTURE FAILURE AREA (TUBE-SEAM WELD TRANSITION
ZONE) OF TYPE 347 STAINLESS STEEL AFTER EXPOSURE TO 1300°F ARGON**

UNETCHED

50X

FIGURE 3-8. STRESS-RUPTURE FAILURE AREA (WELD) OF TYPE 347 STAINLESS STEEL AFTER EXPOSURE TO 1300°F ARGON

10% OXALIC

100X

FIGURE 3-9. STRESS-RUPTURE FAILURE AREA (WELD) OF INCOLOY 800 AFTER EXPOSURE TO 1300°F ARGON

10% OXALIC

500X

**FIGURE 3-10. TYPICAL STRUCTURE (TUBE) OF INCOLOY 800
AFTER 168 HOURS EXPOSURE TO 1300° F ARGON**

Type 347 failed in the seam weld with localized weld deformation (creep). The intimation is that the seam welds had much lower stress-rupture properties than the tube. No obvious effects on the failure modes from chemical or mechanical property specifications can be noted. The Type 347 stainless steel welds were completely homogenized, as opposed to the Type 304 and 316 stainless steel welds; therefore, no apparent effect of weld homogenization (grain size effects) is indicated. There was no metallographically discernible difference in the structure between the tube and seam weld that would explain the failures.

The failure characteristics of the Incoloy 800 stress-rupture samples are a composite of the various 300 series stainless steel failures. The failures occurred in the weld with localized weld deformation. The weld was not homogenized. As was the case for the 300 series stainless steels, no obvious effects of the chemical or mechanical property specifications on the failure modes

could be noted. The Incoloy 800 had a duplex grain structure of fine grain surfaces and a large grain center. However, the outside diameter surface was removed for the 5R-1 tests⁽¹⁾ and the results were the same.

A point of interest is the apparent initiation of cracks on the inside diameter surface for both Type 347 stainless steel and Incoloy 800. Hoop stress calculations predict the highest stresses at the inside diameter surface, but this is the first observation of it in stress-rupture testing.

In comparing the chemical compositions and mechanical properties of the respective materials, a consistent and predictable trend is noted. The higher carbon content, and finer grain size (1 → 10); the greater the tensile and yield strengths and the lower the percent elongation. As stated above, however, there is no consistent correlation between these properties and the failure character-

istics. In general, a larger grain size results in better stress-rupture properties. In this case, the grain size had no effect on the failure characteristics. Correspondingly, as noted above, the grain size variations between weld and tube had no apparent effect. Therefore, the inspection of incoming weld drawn tubing as pertains to chemical composition, uniaxial tensile properties, and metallography (structure and grain size), will not predict the occurrence of seam-weld failures under biaxial stress/strain conditions.

The weld drawn Types 304 and 316 stainless steels should yield representative properties in the mechanical property studies. The Type 347 stainless steel and Incoloy 800, because of localized seam weld deformation, will not produce representative mechanical property data and are therefore not useable.

3.2 LOOP 6R—EXPERIMENTAL CONCEPT, RUN 6R-1P

A preliminary run, 6R-1P, briefly outlined in the previous quarterly, ⁽¹⁾ has been completed. The purpose of the run was to compare the specimen configuration (wire constraint versus no wire), stress condition (static versus cyclic) and to scope stress levels for the following 1000-hour seasoning run, 6R-1.

3.2.1 Test Conditions and Results

The test specimens were 0.250-inch outside diameter $\times$ 0.015-inch wall annealed and 1/4-hard Type 316 stainless steel, both under static and cyclic pressure conditions with and without wire constraint. The wire radius of curvature was 0.151 inch as compared to the specimen radius of 0.125 inch. The test was run in air at 1300°F and was terminated after 301 hours. The specimen stress-strain relationships are shown in Table 3-4. The static specimens were

pressurized and valved-off. The pressure switches were set to indicate failure (stoppage of elapsed time indicator) at a 200 psi pressure drop. Because of system leaks (only one sample actually failed), the 200 psi pressure drop occurred at varying times. The specimens were not vented or depressurized and were left in the furnace. This is the reason for the scatter in exposure times for the static specimens.

3.2.2 Static Versus Cyclic Stress Conditions

The major result of the test was that the statically pressurized specimens exhibited predictable expansion and failure characteristics based on the exposure times to stress and temperature. The cyclically-pressurized specimens had little to no expansion in all cases.

These effects are illustrated in Figures 3-11 and 3-12 for the two sets of annealed samples without wire constraints. A range of uncertainty is indicated since, as stated previously, the stress level of the static specimens was unknown after the start of the test. However, there is no method of determining the length of time where a stress existed sufficient to produce deformation, since the specimens were not vented and remained in the thermal environment until the test was terminated. Since it is an oversimplification to assume a linear strain rate, the average strain rates (slopes of curves in Figures 3-11 and 3-12) were calculated for both time limits under static conditions and on the basis of time at maximum load for the cyclic condition.

It can be observed that for the higher stress level, Figure 3-11, cyclic loading retards creep, i.e., creep rates decrease. Static stress creep rates were between three and nine times higher than the cyclic stress creep rate. For the lower stress level, Figure 3-12, the uncertainty range spans the cyclic data. It cannot be con-

cluded from these data whether cyclic loading retards or accelerates creep. Two items should be emphasized; (1) the deformation was small in relation to that expected at failure for annealed material under these conditions and (2) the static specimen lost 200 psi in ~ 70 hours, indicating a rapid dropoff in pressure. If this rapid drop is assumed to continue, the indications are that upper limits of static stress creep rate would be more nearly correct. If this is true, then retardation of creep was also produced by cyclic stressing at the lower stress level.

The selection of hoop stress was made on the basis of reported data⁽³⁾ which equated the time factor because of the doubling of the creep rate by cyclic stressing. This does not appear to be the situation for Type 316 stainless steel at 1300°F. In an attempt to correlate cyclic and static conditions, it was found that on the basis of mean stress level (12,375 psi versus 17,750 psi maximum for the highest stress sample), the results appeared reasonable in relation to previously obtained static data.

TABLE 3-4
RESULTS OF TESTING TYPE 316 STAINLESS STEEL
UNDER VARIOUS STRESS CONDITIONS AT 1300°F IN AIR

Specimen Condition	Hoop Stress		Approximate Time at Conditions, hours	Specimen Configuration	$\Delta D/D, \%$
	Nature	Level, psi			
Annealed	Steady-State	15,750	70	Wire No Wire	0.6* 0.4
	Cyclic	$\frac{15,750}{7,000}$	301	Wire No Wire	0.6* 0.6
	Steady-State	17,750	108	Wire No Wire	12.2* 10.6
	Cyclic	$\frac{17,750}{7,000}$	301	Wire No Wire	1.6* 1.6
1/4 Hard	Steady-State	19,750	131	Wire No Wire	0 0
	Cyclic	$\frac{19,750}{7,000}$	301	Wire No Wire	0 0
	Steady-State	23,500	255	Wire No Wire	1.0* 1.0 Failed (1.8)
	Cyclic	$\frac{23,500}{8,000}$	301	Wire No Wire	0.4* 0

* Opposite wires.

FIGURE 3-11. COMPARISON OF THE AVERAGE STRAIN RATE BETWEEN STATICALLY AND CYCLICALLY PRESSURIZED (HIGH) ANNEALED TYPE 316 STAINLESS STEEL AT 1300° F

GEAP-5582

FIGURE 3-12. COMPARISON OF THE AVERAGE STRAIN RATES BETWEEN STATICALLY AND CYCLICALLY PRESSURIZED (LOW) ANNEALED TYPE 316 STAINLESS STEEL AT 1300° F

3.2.3 Evaluation of Wire Constraint

Sufficient deformation was not obtained to determine whether the possible failure initiating cracks occurred on the inner surface (wire constraint) as was previously observed and reported.⁽¹⁾ Significant deformation was noted on one set of samples, however, the variation in average deformation between the wire constrained and non-constrained sample was negligible.

To compare the average hoop strain of the wire constrained and nonconstrained specimens, it was necessary to develop a method of determining hoop strain from $\Delta D/D$ measurements of the wire constrained specimens. The $\Delta D/D$ for these specimens was measured on the major diameter (bulge) as shown in Figure 3-13. Each bulge was assumed to consist of a circular segment tangent to the wire (see Reference (1) for support of this assumption) with intimate contact between wall and wire in the contact region. The average hoop strain caused by bulging, ϵ_b , was calculated as a function of $\Delta D/D$ through the parameter θ , Figure 3-13, from geometric considerations:

$$\epsilon_b = \left(2 - \frac{\theta}{\pi/2}\right) \frac{1 + \frac{D_W}{D_1} (1 - \sin \theta)}{\sin \theta} + \left(1 - \frac{\theta}{\pi/2}\right) \frac{D_W}{D_1} - 1 \quad (1)$$

$$\frac{\Delta D}{D} = \frac{1 + \frac{D_W}{D_1} (1 + \cos \theta - \sin \theta)}{\sin \theta} \quad (2)$$

where,

D_W = apparent diameter of wire

D_1 = distance between wires

θ = angle, Figure 3-13.

The plot of ϵ_b versus $\Delta D/D$ for the conditions of run 6R-1P is shown in Figure 3-13.

The strain, ϵ_b , determined in the above manner does not account for any uniform expansion required to take up the tolerance among the thick-walled test channel, the wire, and the specimen. This additional strain, ϵ_W , can be obtained directly from $\Delta D/D$ measured across the specimen directly under the wires. Note that in Table 3-4, the annealed constrained specimen with a static stress of 17,750 psi experienced 12.2 percent $\Delta D/D$, which corresponds to 7.9 percent ϵ_b in Figure 3-13. This is 2.7 percent less than the strain in the nonconstrained specimen. However, the take-up strain, ϵ_W , which as yet has not been measured, may bring these values closer together. It should be emphasized that the above is a comparison of average hoop strain. To obtain the peak strain, it is necessary to consider bending strains as the geometry changes. Bending was discussed in detail in the previous quarterly report.⁽¹⁾

3.2.4 Metallurgical Examination

The microstructures of the stress-rupture samples were normal for Type 316 stainless steel exposed in air at 1300°F. There were grain boundary precipitates, heavy outside diameter surface oxidation with associated structural perturbation, and grain boundary separations—primarily outside diameter surface oriented. One interesting observation was the occurrence of inside diameter surface strain markings (slip lines) on the annealed cyclically-stressed specimens as compared to the statically-stressed specimens.

There was little to no metallographically observable effect of the wire constraint, even on the highest statically stressed specimen.

3.2.5 Results of Literature Survey

The reported effects of the addition of cyclic conditions to creep conditions vary considerably, i.e., cyclic conditions can accelerate, retard, or have little effect on creep rate under static loads.

The most reasonable comparison between cyclic and static loading would appear to be to calculate expected creep under cyclic load on the basis of time at stress using relations between creep rate and stress obtained in static load creep tests. That is, strain is integrated strain rate as a function of variable stress with respect to time. Other bases of comparison include mean stress level and maximum stress level. The general behavior of material classes have been classified by Vitovec⁽⁴⁾ and can be seen in relation to the above bases of comparison in Figure 3-14. The three common bases for comparison are indicated by dotted lines. Data to the left of a base indicate accelerated creep and to the right, retarded creep.

In Figure 3-14, curves I, II, and III represent equal creep in equal times for various combinations of alternating and mean stress. Curve I is characteristic of most materials and curve II is representative of some alloys at certain temperatures and frequencies with alternating stress. Stainless steels would appear to fall in the category of curve II. That is, under some conditions, using curve III as a comparison, creep is retarded while for other conditions, creep is accelerated.

For example, Guarnieri⁽³⁾ found, in creep-fatigue tests of Type 321 stainless steel using intermittent loading, creep acceleration by as much as 100 percent at 1200° F and retardation as much as 40 percent at 1350° F. Simmons and Cross⁽⁵⁾ in intermittent loading tests of Type 310 stainless steel at 1500° F observed similar results to those of Guarnieri at 1200° F. In fact, comparison on the basis of constant maximum stress was found to produce good results for their case. Other investigators present equally contradictory results.

It was noted from the literature that ductility and time-to-failure were equally erratic with generally the same characteristics as in Figure 3-14.

Explanation of the observed behavior is generally considered to be involved with (1) increased dislocation mobility caused by the cyclic nature of stress causing acceleration of creep, (2) initiation and/or acceleration of carbide precipitation which can accelerate or retard creep—the controlling factor appearing to be the degree of dispersion and particle size, and (3) the effect of the cyclic stress on cracks in the material generally propagating or accelerating the propagation of cracks, which affects ductility and time-to-failure. The last two items appear to be significant since carbide precipitation is prevalent in the candidate cladding materials and corrosion in sodium can produce cracks.

Many investigators have developed analytical relationships with some producing good correlation on certain materials and conditions. However, there appears to be no acceptable means of analytical correlation. The above mentioned relationships do not allow for reasonable correlation of the observed results from run 6R-1P. As was stated previously, good correlation was obtained (at least for two specimens) on the basis of mean stress level. However, even though the mean stress correlation will influence the stress levels to be used in run 6R-1, there is no real basis for assuming that this correlation will be applicable over wide ranges of conditions. It is proposed to display test results on a stress range diagram similar to Figure 3-14. The correlation of results will be represented by a family of curves representing levels of strain and strain-at-failure.

3.2.6 Present Status

Run 6R-1 (loop 6R—1300° F sodium) will contain 16 annealed Type 316 stainless steel specimens, eight with wire constraint and eight without. There will be four different levels of cyclic stress, the levels will be determined on the basis of mean stress calculations. The results of run 6R-1 will determine the specimen configuration and will scope out stress levels for succeeding runs.

FIGURE 3-13 AVERAGE MEMBRANE STRAIN COMPONENT CAUSED BY WIRE CONSTRAINT VERSUS THE MAJOR DIAMETER CHANGE

FIGURE 3-14 SCHEMATIC STRESS RANGE DIAGRAM SHOWING CURVES FOR EQUAL CREEP AT SOME CONSTANT TIME

SECTION IV

SUBTASK 2.2—LOCALIZED ATTACK STUDIES4.1 SODIUM CLEANING AND
SUBSEQUENT EFFECTS

The possibility of causing localized attack by steam cleaning sodium exposed surfaces has been investigated. The areas of special concern are the occasional removal and cleaning of fuel elements and components for examination and maintenance.

Two specimens, 0.281-inch outside diameter $\times$ 0.015-inch wall Type 316 stainless steel swaged over a Calrod heater, were exposed to 1175° F sodium for 500 hours. The specimens were exposed in two separate tests in a static pot. Two sodium samples have been extracted at 1175° F from the pot in preevacuated metal thief tubes after the second test. The samples will be analyzed for oxygen, etc.

After exposure, the specimens were removed in an inert environment. One specimen was heated to ~400° F (with the Calrod), to simulate gamma heating of a removed fuel

element, and steam cleaned. The second specimen was cleaned in alcohol and water at room temperature. Both specimens were intergranularly attacked up to 1.5 mils. The attack was apparently the result of the level of impurities in the sodium, and not the cleaning procedures, since the alcohol and water cleaning procedure has been used on all previous sodium exposed samples without any observable attack.

An interesting side effect of the exposures was the attack experienced by the specimens in the sodium vapor space of the static pot. The surface was massively penetrated with fine intergranular penetrations extending into the material. The material was sensitized to a lesser degree than in the area of direct sodium contact (lower temperature in vapor space). This may have contributed to the lesser degree of intergranular penetration. The surface areas where little to no attack occurred had a dark surface band of ~0.3 mil similar in appearance to a carburized or nitrided layer.

SECTION V

TASK 3—SODIUM IMPURITIES

During operation of the loops in Phase I of the Sodium Mass Transfer Program, sodium from the systems has been analyzed regularly for carbon and metallic impurities. Despite considerable effort and the development of sensitive techniques for the determination of many of the elemental impurities, there has been little direct correlation between the elemental species found and the extent of mass transfer. Similarly,

these elemental analyses have not led to the identification of the material responsible for the nonoxide breaks observed in plugging indicator curves. It has been surmised that impurities in compound form play a role in these phenomena. Because the existing analytical techniques are primarily based upon analysis for elements, a program has been underway in which the search for complex species and compounds is emphasized.

5.1 SEARCH FOR SPECIES CAUSING HIGH TEMPERATURE BREAKS IN PLUGGING RUNS

The two previous reports in this series⁽¹⁾⁽⁶⁾ have discussed the problem of multiple breaks in plugging indicator rung. The techniques for collecting and analyzing the material causing these breaks have also been described, and some preliminary results have been presented. To date, four samples have been obtained from Loop 8 by means of the plugging compound collectors.

5.1.1 Loop Conditions During Plugging Compound Collection

During all of these sampling periods the main loop hot leg temperature was 1300°F. The cold leg temperature at the inlet to the purification and sampling loop was ~800°F for the first sample and ~890°F for the remainder. Other conditions during these samplings are summarized in Table 5-1.

Sodium was pumped through the devices kept at the temperatures shown in Column 2 of the table until the initial flow was reduced to the percent shown in Column 3. Pertinent cold trap information is shown in Column 4. The characteristics of the plugging indicator run made just before the collection run are summarized in the final column.

Three runs (1, 2, and 4) each showed flow breaks at temperatures well above that of the cold trap. These flow reductions were presumably caused by the species being sought, an attempt was made to plug the collectors at the temperature in the vicinity of these breaks. No flow decreases were observed near the cold trapping temperature, indicating oxide content was below saturation for these temperatures or that the species causing the higher temperature flow break was obscuring the "oxide" break. The loop cold trap had been valved off for a considerable period of time prior to the operation of the third collector. In this case, a barely discernible break at about 340°F may have been signalling the presence of sodium oxide.

5.1.2 Analysis of Collected Material

The primary method of examination of the material deposited in the channels of the collector was direct x-ray diffraction analysis. Amalgamation of samples followed by identification of the released gases was also used in the characterization. In several cases, sodium was removed from the deposits by distillation or ammonia dissolution, and the residue analyzed. Finally, a few elemental analyses were performed.

The significant results are summarized in Table 5-2.

TABLE 5-1
LOOP CONDITIONS DURING PLUGGING COMPOUND COLLECTION

Device Number Date of Service	Collecting Temperature (°F)	Percent Flow*	Cold Trap Temperature (°F)	Plugging Temperature (°F)
1) October 26, 1966 - October 27, 1966	325—310 (90%) >325 (~10%)	12	275	430 No 'oxide' break
2) March 2, 1967 - March 3, 1967	463—300 (90%) 300—230 (~10%)	15	285	395 No 'oxide' break
3) April 6, 1967 - May 1, 1967	600—585 (84%) >600 (~16%)	35	Not Operated	730—735 Possible 'oxide' break (340)
4) September 5, 1967 - September 9, 1967	405—360 (77%) >405 (~23%)	41	318	420—423 No "oxide" break

* $\frac{\text{Final Flow}}{\text{Initial Flow}} \times 100$

TABLE 5-2
ANALYSIS OF PLUGGING COMPOUND COLLECTOR DEPOSITS

Collector Number	Species	Approximate Concentration (%)	Method of Determination
1	Na	Major	x ray
	Na ₂ O	>5	x ray
	NaH	5	x ray, amalgamation
	NaOH	>5	x ray
2	Na	Major	x ray
	NaOH	4.0	x ray
	NaH	0.5	amalgamation
3	Na	Major	x ray
	NaOH	>5	x ray
	NaH	4	amalgamation
4	Na	Major	x ray
	NaOH	50.5**	x ray
	NaH	0.1	amalgamation

** Material from two separate slots.

Other species observed at extremely small concentrations in some, but not all of the devices were sodium carbonate, α Fe, and the cyanide anion.

5.1.3 Conclusions

It is clear that the only species detected which were present at concentrations sufficiently large to create flow stoppage problems are those containing hydrogen.

These data, coupled with previously-reported observations of high concentrations of hydrogen in cover gas and in cold trap deposits, lead to the conclusion that hydrogen compounds are responsible for the high temperature breaks observed in Loop 8 plugging runs and presumably the high temperature breaks in the other loops.

5.2 HYDROGEN MEASUREMENTS

The determination of hydrogen in mass transfer loop sodium is necessary for several reasons. Firstly, such data are required in support of investigations of anomalies in plugging indicator curves, of studies of effects of cold trap temperature variations, and in

explaining changes in cover gas purity. Further, the normal surveillance of the loop sodium would not be complete without knowledge of the hydrogen content. Finally, the concentration of hydrogen species must be known to obtain correct values for the oxygen concentration when the amalgamation technique is used to measure sodium oxide.

5.2.1 Flow-Through Sampling for Hydrogen Determination

The previous quarterly described initial experience with a flow-through pipe sampler. The results presented showed the device to be a satisfactory means of obtaining samples for the analysis of oxygen. During the present reporting period, an investigation has begun to determine whether the device is equally satisfactory for obtaining sodium from the loops for hydrogen measurement.

The analytical technique used for the measuring of hydrogen in sodium is based upon gaseous diffusion and has been described in the project literature⁽³⁾. An initial question was whether to core the sodium from the pipe prior to analysis or analyze sections,

pipe and all, as is done in the oxygen determination. Coring is attractive, since it eliminates concern over blanks caused by insufficient surface cleaning or hydrogen content of the pipes. Use of entire pipe sections, seems preferable in order to avoid loss of hydrogen compounds which may have precipitated on the tube walls during cooling of the sample.

A number of samples were analyzed using both techniques. The results are shown in Table 5-3.

TABLE 5-3
DETERMINATION OF HYDROGEN

Sample Number	Hydrogen (ppm)	
	Coring	Total Sampling
5R-2-1A	2.1	6.4
9-1-5A	2.5	5.0
9-1-6A	7.0 $\pm$ 0.9	7.2 $\pm$ 0.06
9-1-7A	6.0 $\pm$ 0.6	10.3 $\pm$ 1.6
9-1-8A	3.3 $\pm$ 0.6	8.3 $\pm$ 0.8
9-1-10A	7.3 $\pm$ 2.2	17.2 $\pm$ 0.07

It is evident that values obtained by total sampling are higher. Since the determination of blanks caused by hydrogen in the steel or to surface contamination of the pipes has yet to be evaluated, a clear-cut conclusion that precipitation is occurring cannot be made as yet. However, some confirmation that deposition on the walls is occurring appears in the results obtained from Samples 9-1-6A and 9-1-10A. The cold trap temperatures during the respective sampling periods were 270° and 400°F. A large increase in hydrogen content in the cover gas was noted as a result of the heatup. The sodium taken by coring from the high cold trap temperature sample (9-1-10A) did not reflect this change, while the sodium plus pipe sample showed a hydrogen increase of more than twofold.

Work to establish blanks for the pipe section is in progress.

5.2.2 Hydrogen Effects on Oxygen Determination

The effects of hydrogen-containing compounds on the determination of oxygen by amalgamation have been discussed frequently. A detailed description of the problem has recently been published.⁽⁴⁾ As an illustration, consider the effect of sodium hydride. One ppm of hydrogen, present as sodium hydride, will produce an alkalinity equivalent to 8 ppm of reported oxygen. Fortunately, destruction of 80 to 90 percent of this compound to sodium and hydrogen by the heat of amalgamation reduces the problem, and in the actual case the oxygen equivalent is only 1 to 2 ppm for every ppm of hydrogen (as NaH) initially present.

Preliminary results give evidence of this effect. Oxygen results obtained by amalgamation appear to be slightly higher than would be predicted on the basis of plugging indicator breaks and cold trap temperatures by an amount which could be accounted for by the presence of sodium hydride. Data are being accumulated on this subject. Further, a modification has been made in the amalgamation technique to permit determination of hydride hydrogen, which should make a correction possible.

5.2.3 Impingement Samplers

Five special corrosion samplers consisting of a series of baffles and washers, in addition to the usual rings, were installed and run in cold leg areas of Loop 9 during run 9-1. These devices, called impingement samplers, were designed to obtain information on the mechanical sticking of corrosion products to pipe surfaces in sodium systems.

After loop exposure the cartridges, each containing 17 specimens, were removed from the loop and sealed. Next, sodium was removed from each of the devices by vacuum distillation. The cartridge was next transferred to an inert atmosphere for disassembly.

Unusually heavy deposits, 50 to 100 mg, were found on the specimens which were exposed in the lower temperature

regions. Identification of this residue is underway.

Preliminary results obtained by x-ray diffraction and fluorescence analyses indicated that the major species present in the cooler zone deposit had an austenitic steel structure composed of nickel, manganese, iron, and chromium. Also noted is an unidentified material, isostructural to NaNiO_2 , similar to one previously observed in the program,⁽⁵⁾ and some carbon containing species.

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