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**SODIUM TECHNOLOGY
DEVELOPMENT PROGRAM
MASS TRANSFER INVESTIGATIONS
IN LIQUID METAL SYSTEMS
QUARTERLY PROGRESS REPORT
NO. 10
JUNE-AUGUST 1969**

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BREEDER REACTOR DEVELOPMENT OPERATION

GENERAL ELECTRIC

SUNNYVALE, CALIFORNIA

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SODIUM TECHNOLOGY DEVELOPMENT PROGRAM
MASS TRANSFER INVESTIGATIONS IN LIQUID METAL SYSTEMS

Quarterly Progress Report No. 10

June - August 1969

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SECTION 1

INTRODUCTION AND PROGRAM SUMMARY

1.1 GENERAL

Corrosion of candidate structural materials, the effects of corrosion on the mechanical properties, 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 liquid Metal Fast Breeder Reactor (LMFBR) and the Fast Flux Test Facility (FFTF) development programs.

In an earlier 6-year phase of the program ending in 1965, corrosion and mass transfer of Type 316 stainless steel and 2-1/4Cr-1Mo and 5Cr-1/2Mo-1/2Ti ferritic steels were studied in depth at sodium temperatures to 1200°F. More than 2500 samples of these materials were exposed to sodium during a total of 140,000 hours of loop operations. Average corrosion rates were correlated by statistical analysis from which quantitative relations were derived. The data were correlated with the variables of temperature, sodium velocity, sodium impurity content under cold trap control, and type of material.

In the current phase of the program, the materials were extended to include cladding candidates for oxide fuel (Types 304, 316, and 347 stainless steels (SS), and Incoloy-800). The temperature range of testing was increased to 1300°F, and temperature profiles, loop geometry, and sodium purity controls have been established to represent environments of large fast reactors in an improved way.

1.2 SUMMARY OF TASKS

The Sodium Mass Transfer Program is organized into five tasks. These tasks and their objectives are as follows:

TASK I - CORROSION/DEPOSITION

Objectives are improvements of empirical and rational models for accurate prediction of corrosion and mass transport in primary and secondary sodium systems of large plant designs.

TASK II - SODIUM-IMPURITIES DETERMINATIONS AND PARTICULATE IMPURITY IDENTIFICATION

This task includes the development efforts: sampling of sodium systems, impurity monitoring, collection and identification of particulate material, and chemical and structural analysis. An operating objective is to provide well-controlled operation of loops and to support development of rational models.

TASK III - FERRITIC MATERIAL EVALUATION

This task has the objective of defining test needs and experimental requirements for determining the effect of carbon loss on long term mechanical properties of ferritic steels. No work is currently underway on this task.

TASK IV - CORROSION AND MASS TRANSPORT EFFECTS ON COMPOSITION, STRUCTURE, OR MECHANICAL PROPERTIES OF CANDIDATE ALLOYS FOR LMFBR'S AND LOCALIZED ATTACK

The objective is to obtain exposures of specific LMFBR candidate ma-

terials, including Types 304 and 316 stainless steel tubing, and to determine correlation of mass transfer and sodium impurity effects with changes in long term stress rupture properties.

TASK V - LARGE SYSTEM CORRELATIONS

The purpose of this task is the correlation of corrosion data from a large sodium system with predictions from corrosion models developed in Task I. Sample holder assemblies were installed in SCTI under Task IV of the FY-1967 and 1968 program.

TASK VI - EVALUATION OF MASS TRANSFER STATE-OF-THE-ART AND ANALYSIS OF FFTF FUEL CLAD, PIPING, AND CLOSED LOOP MATERIAL DEGRADATION RATES

The objective of this task is to provide the basis for selection of allowances for materials degradation of selected FFTF components over the life of the plant. A report is in preparation.

1.3 SUMMARY OF SIGNIFICANT RESULTS DURING REPORTING PERIOD

TASK I - CORROSION/DEPOSITION STUDIES

Chemistry control experiments were completed during operation of Loops 8 and 9 during the quarter. On-line instrumentation, sampling, and impurity monitors were operated to correlate impurity concentrations with cold trap and plugging temperatures. Cold trap temperatures were varied in the range 300°F to 550°F and corresponding response noted for the oxygen meter, distillation sampler, and a hydrogen diffusion cell. These tests were carried out with the loops operating at 1300°F T_{max} and cold leg temperatures of

800°F. The plugging meter was also used to monitor the effects of injection of a small quantity of water in the cover gas with the loop isothermal at 600°F.

Examination of deposits in the cold leg of a sodium loop indicated that particulates are nucleated in the main stream and subsequently deposited on surfaces. Remarkably distinct pictures of surface deposits of crystallite particles, 2 to 5 microns in size, have been obtained by a scanning electron microscope technique.

TASK II - SODIUM-IMPURITIES DETERMINATIONS AND PARTICULATE IMPURITY IDENTIFICATION

Sodium samples taken routinely from operating loops showed total alkalinity values of ~10 ppm. Analysis for major elements showed average values for nickel 1.5 ppm, iron 6.2 ppm, and chromium 3.8 ppm.

The oxygen meter continued to show correct qualitative response to changes in the impurity level of the sodium. Departure of meter response from the theoretical curve indicates an interaction between oxygen and other impurities such as hydrogen or carbon, especially at 10 to 30 ppm levels of oxygen. The meter response to impurity changes was rapid and reproducible but, for the absolute measurement of oxygen activity, there are serious discrepancies still to be reconciled.

Analysis of particulate residue collected from Loop 1 showed the major metallic elements present to be silicon, manganese, chromium, iron, nickel, calcium, and aluminum. Metallographic and x-ray examina-

tions of water insoluble fractions showed the particles to be complex agglomerates containing both metallic and nonmetallic components. The major phase found by x-ray was alpha iron. An austenitic phase and carbides ($M_{23}C_6$ and M_7C_3) were also detected.

TASK IV - CORROSION AND MASS TRANSPORT EFFECTS ON COMPOSITION, STRUCTURE, OR MECHANICAL PROPERTIES OF CANDIDATE ALLOYS FOR LMFBR'S AND LOCALIZED ATTACK

Examination of corrosion data for Types 304, 316, 347 stainless steel and Incoloy-800 obtained in 1300°F sodium to 4838 hours showed a non-linear response for the alloys with higher corrosion rates. Incoloy-800

exhibited the highest initial and steady state corrosion rate. Type 316 stainless steel continued to show a linear response with the lowest overall corrosion rate.

1.4 REPORTS

Monthly report letters are prepared reporting progress in all tasks. Progress letters through July 31, 1969, have been issued.

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

This is the tenth quarterly progress report and covers the main results obtained in each task.

SECTION 2

TASK I - CORROSION/DEPOSITION STUDIES

2.1 GENERAL

This work is directed toward:

- Experimentally studying the corrosion of steel, including Types 304 and 316 stainless, exposed to flowing sodium at bulk temperatures to 1300°F.
- Developing and using heaters employing heat flux of 0.5 to 1.0×10^6 Btu/h-ft² average in sodium to measure the effects of reactor heat flux on materials.
- Developing an understanding of the mechanisms involved in phenomena of corrosion mass-transport and deposition in small test loops so that extrapolations can be made to other larger systems.

- Monitoring the test systems for localized attack.
- Establishing relationships between sodium impurities, loop operations, and metallurgical effects.

The work is carried out in test loops constructed to simulate corrosion conditions of a 1000 MW coolant system and a closed loop system for FFTF.

2.2 LOOP 8 PARAMETER STUDY

Conditions of test—Run 8-5: 1300°F (T_{max}), 500°F (ΔT), < 300°F, plugging temperature by cold trap control, 2,500 hours duration.

At 0800, August 31, 1969, the operating times were:

Current Quarter (5/31/69- 8/31/69)	- 2,864 hours
Run 8-5	- 3,563 hours
Na has circulated in the loop for	- 21,869 hours

The end of Run 8-5 was extended to allow completion of chemistry experiments.

During this period, the loop operated at test conditions without difficulty.

SUMMARY OF OPERATIONS COMPLETED IN RUN 8-5

- Obtained data to calibrate the on-line electrochemical oxygen meter to oxygen impurities as determined by:
 1. Cold trap temperature and solubility correlations.
 2. The on-line distillation system.
- Distillation of 50-gram batches of sodium (on-line) for determination of total alkalinity values.
- Distillation of 1000 grams of sodium in ~60-gram batches to collect and identify particulate impurities.
- Regular sampling of the sodium via flow-through samplers.
- Operation of plugging indicator to obtain data on sodium plugging temperature and to identify compounds causing the plugging breaks.

The operating parameters of Loop 8 from the beginning of Run 8-5 (February 15, 1969) to August 1, 1969, are given in Appendix 1. Results of data analysis are discussed below and in Task II.

2.3 LOOP 9 PARAMETER STUDY

Conditions of Test - Run 9-2: 1300°F (max), 500°F (ΔT), <300°F, plugging temperature controlled by cold trap, 3,000 hours duration.

Parameters being studied are sodium mass flow, temperature, velocity, system geometry, and heat flux.

At 0800, August 31, 1969, the operating times were:

Current Quarter (5/31/69-8/31/69)	- 1,080 operating hours
Run 9-2 - hours above 1000°F	- 6,339 hours
EB heater at 10kW	- 4,334 hours
Na has circulated in the loop for	13,242 hours.

Operating parameters of Loop 9 for Run 9-2, from its beginning on April 18, 1969, to its termination on July 15, 1969, are given in Appendix 2.

2.3.1 Summary of Work Completed in Run 9-2

During this period the loop operated 4,334 hours with ~500,000 average, 800,000 peak, Btu/h-ft² heat flux at 10 kW total power of the high heat flux heater. Outlet temperature of the heater averaged 1240°F, while the outlet of the low heat flux auxiliary heater averaged 1290°F, to produce a mixed average of 1275°F into the first cooler inlet (simulated upper core test section).

Cold trapping was carried on full time at 270°F with a flow averaging 0.6 gpm. During this period, the nature of the plugging runs varied from a double break system to a single break, and back to a double break again.

On July 8, the power was removed from the EB heater in advance of shutting down the loop to end Run 9-2. Prior to dumping the loop,

three experiments were performed; a cold trap temperature excursion and recovery, and two water injections into the cover gas.

Presently, the samples are being removed and analyzed. Modifications to the loop itself are also in progress; these include attachment of vapor traps and lines from the surge tanks and the chromatograph, and addition of a second cold trap.

2.3.2 Study of Cold Trap Operation and Hydrogen Measurement

During the course of operations in Loop 9 (Run 9-2, see Appendix 2, Operating Parameters), a 3- to 4-decade increase in pressure was recorded on the ion pump for the high heat flux heater (electron bombardment) whenever the cold trap temperature increased. This effect was observed to be reproducible.

The above effect on the electron bombardment heater is attributed to hydrogen diffusion through the heater anode. Observations of release of hydrogen to the loop upon increasing cold trap temperature have been noted in previous reports. In order to study the response of the heater in Loop 9 it was used as a diffusion tube during a preliminary experiment to study cold trap efficiency.

2.3.3 Cold Trap Temperature Excursion and Recovery

The purpose of this experiment was to:

1. Observe the response of the monitoring devices, ion pump - diffusion-tube, and plugging indicator to a controlled excursion in cold trap temperature.
2. To determine the alkalinity and total hydrogen concentration at the maximum cold trap temperature using chemical analysis.

3. To obtain data on the rate of removal of the impurities during the cold trap recovery as an indicator of cold trap efficiency.

On July 10, the cold trap temperature was raised from 300°F to 550°F in three steps. The final step was from 400°F to 600°F. However, the temperature was reduced to 550°F when it became apparent that the hydrogen detector (EB heater pressure) would go off scale with a 600°F cold trap. After approximately 20 minutes at 550°F, a flow-through sampler was started in the cold leg and turned off 4 hours later. The cold trap temperature was then lowered to 270°F at a rate of 5°F per minute.

During this experiment, the upper and lower plugging breaks were monitored by repeated bare-orifice plugging runs. The electrical current reading of the 8 liter/second ion pump on the electron bombardment heater was also recorded. The impurity removal, as indicated by the monitors, lagged the indicated cold trap temperature by about 1 hour.

Figure 1 is a plot of the cold trap temperature and the response of the on-line instrumentation versus time.

2.3.4 Water Injection Experiments

The purpose of this experiment was to:

1. Determine the response of the on-line impurities monitoring devices of Loop 9 (oscillating mode plugging indicator and ion pump-diffusion tube) to an injection of water.
2. Compare the response of these devices to an injection of water with the cold trap operating versus a similar injection without the cold trap operating.
3. Evaluate the rate of recovery after the injection of water, as a measure of cold trap efficiency.

FIGURE 1. RESPONSE OF PLUGGING INDICATOR and HYDROGEN DIFFUSION CELL TO A COLD TRAP TEMPERATURE RISE AND RECOVERY

For the water injections, loop temperatures were set isothermal at approximately 600°F. For the first injection, the cold trap was valved out and 0.8 cc of deionized water was injected into the cover gas. An increase in the plugging temperature (oscillating) was observed 19 minutes later (225°F to 250°F). The plugging temperature continued to increase for about 2 hours to a steady-state value of approximately 295°F. Flow was initiated to the cold trap at a temperature of 320°F at 0.7 gpm. The cold trap temperature was then lowered to 280°F at a rate of 2°F to 3°F per minute. The impurity monitor lagged the indicated cold trap temperature by about 1 hour. The ion pump reached its maximum pressure 1-1/2 hours after the injection.

Figure 2 is a plot of the oscillating mode plugging temperature versus time for this water injection.

A second water injection took place while the cold trap was operating at 290°F and 0.70 gpm. Six minutes later the plugging temperature increased from 225°F to 240°F. The plugging temperature continued to rise for 25 minutes to a maximum of 275°F and then began its descent. The ion pump reached its maximum about an hour after the injection.

Figure 3 is a plot of the oscillating mode plugging temperature versus time for this injection.

2.3.5 Conclusions

Both the plugging indicator and the diffusion tube responded to increased impurity in the loop as shown in Figure 1. Response of the diffusion tube was essentially instantaneous. The plugging indicator was operated in the bare orifice mode at specific time intervals so no conclusion can be made on sensitivity to initial changes in purity. The monitoring equipment showed a return of the system to initial conditions in about 1 hour after lowering cold trap temperature except for the high temperature

break in the plugging curve. This might indicate a longer time required to equilibrate hydrogen in the system to the cold trap temperature.

In the water injection experiments (Figures 2 and 3), the operation of the cold trap was shown to limit the amount of impurity increase in the loop due to an intentional injection. The cold trap operating at 0.7 gpm was thus shown to be capable of effective removal of impurities from Loop 9.

2.4 HIGH HEAT FLUX EFFECTS

As noted above under Loop 9 Operations, the electron bombardment heater ran for 4,334 hours at an average heat flux 500×10^3 Btu/h-ft², (800 x 10³ peak) in the hot leg. The anode, Type 316 stainless steel, exposed during this period was the fourth to be used with this cathode and head structure.

The appearance of the heater and anode surface exposed to flowing sodium immediately after removal from the loop and cleaning is shown in Figure 4. A darker surface layer is apparent at the point where heat flux begins. The heated zone is 10 inches with a 0.394-inch diameter.

The sodium cleaning procedure used on removal consisted of swabbing with cotton and deionized water followed by drying at room temperature. No change in surface appearance or evidence of entrapped sodium can be seen on the exposed surfaces of the anode. Preparations are being made to replace the anode and prepare the exposed one for examination.

2.5 "RATIONAL MODEL" EXPERIMENT, TEST FOR EFFECTS OF HIGH REYNOLD'S NUMBER

Corrosion of stainless steel in sodium is known to be affected by the Reynold's number associated with the system and sample geometry.

FIGURE 2. RESPONSE OF PLUGGING METER DURING INJECTION OF WATER IN COVER GAS

FIGURE 3. PLUGGING TEMPERATURE DURING WATER INJECTION IN LOOP 9 COVER GAS

COMPLETED 4000 HOUR RUN AT 1250° - 1300° F SODIUM OUTLET
AVE. HEAT FLUX 500,000 Btu/h-ft²
PEAK HEAT FLUX 800,000

FIGURE 4. HIGH HEAT FLUX HEATER, ELECTRON BOMBARDMENT

Reactor systems of the LMFBR type will be operating with Reynold's numbers of up to 100,000. Thus, it is essential that corrosion rates be determined at levels higher than 30,000 (Reynold's), which has been the limit previously. To accomplish this, an experiment and test loop was designed to determine the corrosion rates of various steels at Reynold's numbers up to 100,000 and the effect of oxygen activity levels on corrosion. The total sodium inventory of the loop is approximately 5 liters. The high Reynold's number will be obtained by rotating the corrosion samples. The loop has been designed to operate at 1300°F and with a pumped purification system to maintain a constant sodium quality at the position of the samples.

Figure 5 shows a schematic of the experiment design. The test vessel will be supplied by 0.25 gpm sodium, with temperatures up to 1300°F and an oxygen concentration between 2 to 50 ppm. These conditions are achieved and controlled with the sodium supply system which contains an oxygen meter for measuring the oxygen activity electrochemically and an oxygen controller for changing the oxygen concentration.

Both of these components are operated at 700°F. Therefore, they are installed behind a controllable cooler and heater.

Other components of the supply system are:

- A main flowmeter
- An electromagnetic pump
- A filter for remaining particulate material
- A purification system
- A flow-through dump tank by which the sodium level in the test vessel will be set by regulating cover gas pressure

The dump tank is also equipped with a sample port for insertion of small quantities of impurities under inert atmosphere.

The purification system will be equipped with a rapid sampler, a continuously operating cold trap, and a plugging indicator.

Design Specifications of the Experiment

Sodium Volume	5-6 liters
Maximum Flowrate	0.25 gpm
Maximum Temperature	1300°F
Minimum Temperature	500°F
Oxygen Range in Sodium	2-50 ppm
Cover Gas Purity	Argon (NaK bubbled)

2.6 LOOP 1 PARTICULATE COLLECTION

After each filter change, the main system sodium flow has stayed at 5 gpm for a few days, and then gradually decreased to a constant flow of approximately 2 gpm for several hundred hours prior to filter removal and reinsertion of new filter. A new filter was inserted on August 18.

2.7 MECHANISM OF DEPOSITION

Previously, it was observed that activity of an alloy constituent of the substrate does not play a significant role in the deposition process. Furthermore, various elements and compounds as particulates have been identified in cold leg deposits. Lastly, we have observed that increase of turbulence enhances deposition. From these observations one can postulate two mechanisms of deposition:

- a. Nucleation of particulate compounds, e.g., carbides, chromites, in the bulk sodium, prior to deposition, or
- b. Precipitation of individual elements and subsequent recombination, and growth on the substrate.

FIGURE 5. SCHEMATIC OF RATIONAL MODEL EXPERIMENT

The scanning electron microscope picture, taken on the surface of a cold leg sample exposed in the first cooler of loop 8 at $\sim 1200^\circ\text{F}$, tends to verify the first mechanism (Figure 6). The photo shows distinct separate crystalline phases. This implies that these crystals were nucleated in the main stream, and subsequently deposited on the substrate. If the nucleation occurred on the substrate, one should not see such discrete crystalline structure. The size distribution of the particulates is approximately 2 to 5 microns. This observation implies that particulates nucleated in the bulk sodium reach a critical size before they deposit from sodium.

If the above mechanism is correct, one may expect the particles to be loosely adherent to the substrate, provided there is no rapid diffusion type bonding. If the diffusion bonding is slow, then it is possible that the build-up of particulates on the walls of the heat exchanger will not go beyond a certain thickness. This is due to the fact that if the particles are loosely adhered to the surface they will tend to be carried away by an erosion process as soon as the deposit reaches a critical thickness.

2.8 TRANSPORT OF CARBON

The exact nature of the particulates responsible for carbon transfer in all austenitic systems is still open to question. Recently, it has been postulated⁽¹⁾ that the mechanism of carbon transport in a liquid sodium/steel sys-

tem involves the formation of Na_2C_2 . This postulation was based on the study of carbon transfer between sodium and Fe-C alloys, where carbon is essentially in the form of Fe_3C . However, another independent study⁽²⁾ indicates that it is not possible to decompose pure Fe_3C below $550\text{--}650^\circ\text{C}$. The implication of the above experiment is that the Na_2C_2 is unstable at these temperatures, which is based on the ΔG formation of Fe_3C being positive. Hence, it is unlikely that the stainless steel carbides, M_{23}C_6 , M_7C_3 , etc., ($\text{M}=\text{Fe}, \text{Cr}, \text{Mn}$) which are far more stable than Fe_3C , will be reduced by liquid sodium. We believe that the transition metal carbides are involved in the carbon transport of austenitic systems.

The above hypothesis is based on two observations: (1) M_{23}C_6 carbides in the deposits of the colder regions, and (2) the strong relationship between the movement of chromium and of carbon. The selective dissolution of chromium from the steel into the liquid sodium will tend to decrease the activity coefficient of carbon in liquid sodium, which will result in decarburization. As the sodium temperature is reduced, one may expect the precipitation of chromium and carbon to take place in the form of carbides due to the stability of Cr-carbides. The precipitation can occur either by recombination from the dissolved state or as particulate carbides from solution. As soon as the sodium becomes depleted with respect to chromium and carbon, further decarburization takes place.

FIG. 6 PARTICULATE MATERIAL DEPOSITED ON COLD LEG SURFACES AT 1200°F.

SECTION 3

TASK II - SODIUM-IMPURITY INTERACTIONS AND IDENTIFICATION
OF PARTICULATE MATTER

Work in this task encompasses the extensive support in sodium chemistry, sodium and gas sampling from loops, impurities detection, and control required primarily by Task I of this program.

3.1 LOOP MONITORING FOR IMPURITIES

3.1.1 Flow-Through Sampling, In-Line

The method used for obtaining samples on a regular basis, normally biweekly, representative of loop sodium consists of installing "Rapid Pipe Samplers" made up of lengths of stainless steel tubing, 1/2-inch o.d., at appropriate points in a test loop. The design and use of these samplers was initially reported in quarterly report No. 2, GEAP-5546.

3.1.2 Total Alkalinity Measurements

Table 1 provides recent data on the total alkalinity (amalgamation determination) for loop samples. Some additional values for Loop 8 are provided in the discussion of on-line distillation (Table 3).

3.1.3 Analyses For Metals

The previous report gives initial results of experiments performed to evaluate the metal flow-through devices for sampling loop sodium for the analysis of metallic impurities. Analyses for iron were reported. The determination of nickel and chromium have been completed and are shown in Table 2. (The results for iron are repeated for convenience.) The data represent analyses for each of the three elements in the sodium removed at different places along the samples.

The data, particularly that for iron, shows considerable scatter. Some of this may be due to contamination during the cutting of tubes for subsampling. This problem should be avoidable by the use of samplers constructed of a single metal (analyses for the constructional element would not be performed). Samples have been taken in nickel tubes and the completion of analytical work is scheduled for the next quarter.

Where sporadic high values are not encountered, e.g., sample 9-2-11C, it is pre-

TABLE 1

TOTAL ALKALINITY IN LOOP SODIUM- FLOW-THROUGH SAMPLES

<u>Sample No.</u>	<u>Date Taken</u>	<u>Plugging Indicator Temp. (°F)</u>	<u>Total Alkalinity ppm</u>
1R-1-1A	5/14/69	Indistinct	23
4R-2-6A	4/23/69	270	10
4R-2-7A	5/21/69	274	11
6-3-3A	5/16/69	280	10
8-5-3A	5/6/69	300	12, 12

TABLE 2

ANALYSES FOR METAL IMPURITIES—FLOW THROUGH SAMPLES

(Precision $\pm 10\%$)

Sample	Section*	Element (ppm)		
		Ni	Fe	Cr
5R-4-3A	1	0.8	2.1	6.7
	2	5.0	11.2	10.0
	3	0	8.2	6.0
	4	--	2.8	1.0
	Filter **	--	1.2	1.2
5R-4-4A	1	1.7	8.0	4.1
	2	1.2	5.5	0
	4	1.6	10.2	3.1
	Filter **	--	--	1.7
9-2-11C	1	1.1	2.6	2.1
	2	1.0	3.5	2.8
	3	0.9	3.4	2.9
	4	1.5	3.4	2.8
	Filter **	--	--	1.0
9/2/2A	1	3.0	10.5	6.3
	2	1.2	10.0	2.8
	3	0.9	7.6	2.8
	4	0.5	3.5	3.4
	Filter **	--	1.1	1.5

*A section is a piece of one-half inch tubing about 1 inch long.

**Refers to material removed after sodium dissolution and acidification. The solutions for all pipe sections from a given sample were filtered through a single filter.

--Not Analyzed.

sumed that contamination did not occur. These results indicate that position-to-position variation along the length of the tube is not serious. They further indicate that obtaining the samples by melting from the tube yields a representative sample.

An interesting observation was that a measurable fraction, 5% to 15% of the chromium and iron, was present in a form not readily dissolved by the normal procedure, i. e., boiling with moderately strong acid. These results are designated as "filter" data in Table 2. This refractory material was obtained by filtering the solution resulting from the dissolution and

acidification of the sodium sample. The particles were dissolved by a fusion technique. (No attempt was made to filter the solution containing the sample prior to acidification.) These results are compatible with data obtained in the Loop 1 particulate study which show that refractory particles are present in the loops.

The presence of small amounts of particulates could explain the discrepancies observed between solubility data for iron, nickel, and chromium and the values observed in operating systems.

The data in Table 2 should be typical for

TABLE 3OXYGEN METER RESPONSE TO COLD TRAP TEMPERATURE CHANGES

<u>Cold Trap Temperature °F</u>	<u>Date of Measurement</u>	<u>Oxygen Meter Reading, Volts*</u>
300	5-13-69	1.160
	5-27-69	1.160
	6-3-69	1.160
	7-1-69	1.160
	7-27-69	1.160
350	5-15-69	1.141
	6-3-69	1.141
	6-2-69	1.140
	7-2-69	1.142
	7-11-69	1.146
400	5-18-69	1.124
	6-5-69	1.128
	6-12-69	1.124
	7-7-69	1.128
450	6-18-69	1.107
	6-13-69	1.107
	7-8-69	1.109

*Temperature of sodium at sensor 600°F ±3°F

the mass transfer loops under normal operating conditions.

3.2 ON-LINE MONITORING - OXYGEN METER, VACUUM DISTILLATION

The oxygen meter in Loop 8 operated continuously for 2000 hours during Loop Run 8-5. Responses of the meter to changes in sodium and air temperature and to changes in sodium purity caused by elevation of the cold trap temperature were reported in the previous quarterly.

3.2.1 Response of Meter to Cold Trap Temperature

Additional data were obtained on the response of the meter to changes in cold trap temperature. All data obtained during the run are summarized in Table 3.

The data indicate the reproducibility of the instrument over the period of testing. The direction of the change (downward) of the readings with increases in impurity content was the correct one. EMF is stable, reproducible, and

follows the expected direction with increase in cold trap temperature.

3.2.2 Comparison of Meter Response with On-Line Samplers

In addition to observing the variation of oxygen meter readings with temperature, the changes in purity were also monitored by analyzing residue obtained by on-line distillation, by amalgamation of grab (flow-through) samples, and by plugging indicator determinations.

Figure 7 shows the plot of EMF versus log concentration of oxygen (ppm) where the oxygen content is expressed in three different ways. In Curve 1 the EMF is plotted against total oxygen. The scatter in the data points makes it difficult to determine if the relationship is linear as expected by Henry's Law.

In Curve 2 the EMF is plotted against total oxygen less the oxygen which is combined as carbonate. This value is a measure of the oxygen present as Na_2O or as NaOH since appreciable amounts of hydrogen are present in the system. NaOH and Na_2O cannot be distinguished by the distillation method. The data points in this case show a distinct curvature, hence it is suggested that Henry's Law is not applicable.

In Curve 3 the oxygen concentration is calculated from the cold trap temperature assuming that the oxygen concentration follows the solubility curve of Eichelberger.

The dashed line shows the theoretical relationship between EMF and oxygen concentration where Henry's Law constant is the reciprocal of the oxygen concentration for saturation at a sensor temperature of 550°K , again assuming the Eichelberger solubility equation. It is apparent that the theoretical curve falls in between the total oxygen data points curve (1) and the data points obtained from cold trap temperature, Curve 3.

The deviation of Curve 3 from the theoretical implies either that (1) the temperature dependence of the Eichelberger equation is incorrect, or (2) the cold trap temperature does not represent the true saturation temperature for the system, or (3) other constituents such as hydrogen or carbon interact with oxygen to cause a deviation from the ideal behavior. The temperature of the cold trap system was found to be in close agreement with oscillating mode plugging temperatures so that saturation conditions probably exist at the cold trap temperature. It can be shown that a not unreasonable 20% uncertainty in the temperature dependence of the Eichelberger equation is sufficient to account for the displacement of curve 3 from the theoretical curve. Hence, it is difficult to ascribe this deviation to other causes.

The deviation of Curve 2 data points from the theoretical is less pronounced, which would imply that there is good agreement between oxygen concentration in the sensor region and the EMF observed using the idealized Henry's law constant. At high concentration of oxygen, a distinct departure from the theoretical curve is evident which suggests that Henry's Law does not apply over the entire range of oxygen concentration. The curvature indicates that the oxygen solubility is less than expected which may be due to a positive deviation from Henry's Law.

Whether this deviation is due to a mutual interaction between oxygen or due to a coupling reaction between the hydrogen or carbon constituents cannot be discerned from the data. Further work at high oxygen concentrations is needed to understand the relationship between oxygen concentration from distillation and the EMF of the oxygen meter.

Curve 1 shows for a given EMF that the oxygen concentration is higher than expected from the theoretical. Since this curve contains the oxygen which exists presumably as the carbonate, it would be expected that the data

FIGURE 7. COMPARISON OF OXYGEN METER RESPONSE TO DISTILLATION RESULTS

points would be displaced upwards from the theoretical values if the carbonate present was inert with respect to other constituents in the system. If the carbonate concentration exceeded the remaining oxide constituents, this curve would show a behavior similar to the trend that is observed. There is no evidence that oxygen exists as carbonate in sodium or that the carbonate-oxygen interaction occurs so that no conclusions about the relationship of oxygen activity to total oxygen concentration can be reached at this time.

3.2.3 Comparison of Distillation Results and Cold Trap Temperature

The distillation results are plotted against cold trap temperature in Figure 8. Curve 1 represents total oxygen, Curve 2 represents the oxygen present as sodium oxide, and the dashed line represents the theoretical value. (It should be recognized that the "theoretical" solubility curve is a best judgment value based on Eichelberger's evaluation of data obtained from a number of experimenters.)

The sodium oxide curve parallels the theoretical curve at lower temperatures but is positioned above it. This may be due to the presence of hydroxide which is converted to the oxide during distillation. At the higher temperatures, saturation values for oxygen at the given cold trap temperatures are not reached (the oxide value falls below theoretical). It is believed that this is due to an insufficient amount of oxygen in the cold trap. This is not unexpected. At 450°F, the oxide solubility is 25 ppm. For the 40-liter system about 1 gram of oxygen is required. In previously reported work it was found that cold traps from the phase I loops contained only about this much oxygen even after longer periods of operation. An experiment is planned in which a sufficient supply of oxygen will be assured to further test these observations.

The total oxygen value is well above the theoretical curve initially and reflects the fact

that a major portion of the oxygen at the lower temperature is present as the carbonate. However, the carbonate concentration remains constant with temperature and the influence of the increasing amounts of oxide oxygen is evidenced by the convergence of the results toward the theoretical curve. This trend is perhaps more apparent in the tabular presentation of the data as shown in Table 4.

It is clear from this data that the distillation technique is sensitive to changes in sodium purity throughout the range tested. It is also clear that sodium oxide levels in the loop are no higher than 5 ppm at the usual cold trap temperatures, 300°F or less.

Also included in Table 4 are the results of amalgamation determination of total alkalinity performed on grab samples of sodium taken during these experiments.

There is a lack of response of the amalgamation results to warming up the cold trap until the higher temperatures are reached. This is attributed to the poor precision of the techniques, ± 3 ppm at the low levels, and its lack of discrimination among alkaline species.

The plugging indicator runs during these tests gave no breaks when the cold trap temperatures were below 320°F. Above this temperature, up to 400°F, which was the highest temperature tested, operation in the oscillating mode produced flow breaks at temperatures in agreement with that of the cold trap to $\pm 10^\circ\text{F}$. The bare orifice mode of operation gave plugging temperatures about 50°F lower than that of the trap.

In summary, the oxygen meter response to changes in impurity levels effected by changing the cold trap temperature was reproducible, rapid, and sensitive. From a loop control and operational standpoint it appears to be a most useful monitoring tool. From the theoretical standpoint, that is the measurement of

FIGURE 8. COMPARISON OF DISTILLATION RESULTS TO COLD TRAP TEMPERATURES

TABLE 4
RESPONSES TO COLD TRAP WARM-UP

Cold Trap °F	Oxygen Meter Volts	Oxygen As Na ₂ O ppm	Oxygen As Na ₂ CO ₃ ppm	Total Oxygen Na ₂ O+Na ₂ CO ₃ ppm	Sample ^(a) Number	Mercury ^(a) Amalgamation Alkalinity; ppm
300	1.160	5	9	14	--	--
300	1.160	4	17	21	8-5-5A	14
350	1.142	10	15	25	--	--
350	1.146	10	12	22	8-5-6A	11
400	1.124	16	14	30	8-5-7A	17
450	1.109	20	14	34	8-5-8A	25

(a) Rapid (on-line) samples taken for mercury amalgamation analysis for total alkalinity determinations.

absolute oxygen activity, there are discrepancies still to be reconciled.

The distillation unit appears to function satisfactorily. The ability to analyze larger samples (50 grams) has enabled a more precise determination of sodium carbonate. Sodium oxide levels at normal cold trap temperatures are 5 ppm or less. A larger fraction of the oxygen is present as the carbonate than as the oxide under these conditions.

The flow-through sampler and amalgamation technique reflected the cold trap changes poorly in the lower temperature regions.

The plugging indicator, when operated in the oscillating mode, gave flow breaks at temperatures which corresponded well with that of the cold trap at temperatures above 320°F, but plugs could not be maintained at lower temperatures, the region of normal operation.

3.3 ANALYSIS OF LOOP 1 PARTICULATES

Examination of the materials collected on the nickel filters removed from Loop 1 after

600 hours of service was initiated. Progress has been made on establishing the chemical and physical properties of the impurities.

Sample preparation consisted of removing sodium from the filter by distillation. Particles were dislodged by this process and by tapping the filter subsequent to distillation. About 1 gram of a dark, clumped, irregularly shaped particulate residue was obtained from a single filter element.

Analysis by emission spectrography showed the major metallic elements present to be sodium, silicon, manganese, chromium, iron, nickel, calcium, and aluminum. Colorimetric analyses which are capable of greater precision than the spectrography, are being performed to obtain quantitative estimates of these elements. The results obtained to date are shown in Table 5. Fairly severe treatment with acid was required to prepare the sample for these analyses.

X-ray diffraction patterns of this material were obtained but have proven too complex to permit characterization thus far. Consequently, a second separation, this time into water-soluble and insoluble fraction, was performed.

TABLE 5

ANALYSES OF METAL IMPURITIES FILTERED
FROM LOOP 1 (NICKEL FILTER)

<u>Element</u>	<u>Concentration wt. %</u>
Iron	10.5
Manganese	3.3
Chromium	7.0
Nickel	2.0
Sodium	42.0
Silicon	5.2
Calcium	1.0

Two-thirds of the sample proved to be water-soluble and contained the bulk of the sodium, silicon, and calcium associated with the collected material. The source of the silicon and calcium has not been established. Neither was present in the unused filter to an appreciable extent. Neither element is present to a great extent in normal (cold-trapped) sodium. Since the filter was in place during early operation of the loop, these impurities may be associated with the assembly of the loop or the new charge of sodium.

The water insoluble fraction containing most of the remaining metals consisted of a variety of particles. These are shown in Figure 9. The majority of these are dark, powdery, and irregular in shape. Sizes range from less than 20 microns to about 500 microns. The particles are ferromagnetic. Metallographic examination indicates that the particles are complex agglomerates containing both metallic and non-metallic components. X-ray diffraction analyses identify the major phase to be alpha iron (body centered cubic, $a_0 = 2.869\text{\AA}$). In addition, a detectable austenitic phase (face centered cubic, $a_0 = 3.588\text{\AA}$) and carbides of the $M_{23}C_6$ (face centered cubic, $a_0 = 10.623\text{\AA}$) and M_7C_3 (hexagonal $a_0 = 13.98\text{\AA}$, $c_0 = 4.523\text{\AA}$) types were found.

Electron microprobe analyses of unsectioned individual particles show essentially the same ratio for the metallic constituents Fe, Cr, Ni, and Mn as reported in Table 5. In addition, areas of nearly pure carbon and some particles containing moderate concentrations of carbon consistent with metal carbides were observed.

Preliminary analyses for total carbon indicate that concentration is in excess of 2%. No evidence was found in this initial work for readily hydrolyzable carbides such as those of calcium or sodium.

Additional particles — some metallic and containing iron as a major constituent — and other nonmagnetic ones containing aluminum were present in the water insoluble fraction in minor amounts.

In summary, the filters contained a wide variety of materials. The most pertinent of these are magnetic — water insoluble ones containing the stainless steel metals. They are largely alpha iron but contain complex metal carbides as well. These resemble the particles found on the impingement samples which were collected at higher temperature with the exception that the $NaMO_2$ phase found in the

FIGURE 9. WATER INSOLUBLE PARTICLES

earlier work was not observed. Evidence was found for the presence of elemental carbon as well. In addition, the samples contained unexplained debris consisting largely of water soluble calcium and silicon salts as well as metallic chips.

Future work will examine differences in the nature of the collected material as a function of time in the loop. Attempts at in-situ examination of the deposited material by direct x-ray diffraction work on portions of a filter will also be made to avoid possible ambiguities due to the effects of distillation hydrolysis.

SECTION 4

TASK IV - CORROSION AND MASS TRANSPORT EFFECTS ON COMPOSITION, STRUCTURE, OR MECHANICAL PROPERTIES OF CANDIDATE ALLOYS FOR LMFBR'S AND LOCALIZED ATTACK

4.1 GENERAL

The objective is to obtain effects of mass transfer on mechanical properties of alloys in flowing sodium systems with temperature differences. Work under this task includes the testing of Types 304 and 316 SS to obtain long-term stress-rupture (to 10,000 hours) data. The present test conditions are 1300°F T_{max} , sodium velocities to 17 fps, and controlled sodium purity corresponding to plugging temperature <300°F.

In addition to the commercial mill annealed grades of Types 304 and 316 SS, the carbide agglomerated (CA) condition (1650°F for 24 hours) is included in the experiments. The basis for selecting the CA heat treatment stems from observations made in EBR-II irradiations, where higher residual ductilities were noted for the CA structure after 3×10^{21} nvt ($E > 1$ MeV).

4.2 TESTING OF LMFBR CLADDING

4.2.1 Loop 4R - Stress Rupture

Run 4R-2; 10,000 hours, 1300°F (T_{max}), 500°F (ΔT), <300°F (T_s)

During the report period, the loop operated at run conditions except for brief shutdowns as a result of specimen failures and a 17-day shutdown due to a sodium leak. The loop has presently operated at 1300°F with the pressure applied for ~5200 hours. The results to date are shown in Table 6. The results plus previously reported data from loop 5R⁽³⁾ are plotted in Figures 10 and 11, where they are compared with 1300°F stress-rupture data in other environments^(4, 5). Also shown in Figures 10 and 11 are the control tests in progress in argon. On the basis of comparison with the referenced data and the control tests, there appears to be a decrease in long term stress-rupture properties due to exposure in nonisothermal flowing sodium.

4.2.2 Loop 5R - Stress Rupture

Run 5R-5; 5000 hours, 1300°F (T_{max}), 500°F (ΔT), <300°F (T_s)

During the report period, loop repairs and modifications were made, sample loading was completed, and startup achieved. The loop has presently operated at 1300°F with the pressure applied for ~300 hours.

FIGURE 10. BIAXIAL STRESS-RUPTURE PROPERTIES OF TYPE-316 STAINLESS STEEL IN 1300°F SODIUM

FIGURE 11. BIAXIAL STRESS-RUPTURE PROPERTIES OF TYPE-304 STAINLESS STEEL IN 1300°F SODIUM

TABLE 6

1300°F BIAxIAL STRESS-RUPTURE DATA FROM RUN 4R-2

<u>Material</u>	<u>Initial Hoop Stress, psi</u>	<u>Time-to-Rupture, hours</u>
304SS - CA	7000	2188
304SS - MA	7000	2198
304SS - MA	7000	2237
304SS - CA	7000	2263
316SS - MA	8000	3822
316SS - MA	8000	4415
304SS - CA	5000	4500

MA - mill annealed

CA - carbide agglomerated, 1650°F for 24 hours

4.2.3 Loop 6R - Stress-Rupture

Run 6R-4; 10,000 hours, 1150°F (T_{max}), 440°F (ΔT), <300°F (T_s)

During the report period, pressure system modifications were made, sample loading completed, and startup achieved. The loop has presently operated at 1150°F with the pressure applied for ~500 hours.

4.2.4 General Corrosion

A numerical error of about 15% was found in corrosion data reported previously⁽³⁾

for Types 304, 316, 347 SS and Incoloy-800 exposed to 1300°F flowing sodium. The correct value for the longest time point was 4838 hours, (vs. 4178 hours previously). The average corrosion results of the upstream sampling area in Loops 4R, 5R, and 6R are shown in Figure 12.

The previously reported⁽⁶⁾ differences between the alloys remain unchanged. However, the apparent linear corrosion rate exists only for Type 316 SS. A nonsteady state corrosion period is shown for the other three alloys. The materials with higher corrosion rates exhibit a more pronounced nonsteady state effect.

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FIGURE 12. CORROSION RATES OF MILL ANNEALED STAINLESS STEELS IN 1300°F SODIUM

SECTION 5

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APPENDIX 1. OPERATING PARAMETERS OF LOOP 8, RUN 8-5

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