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**SODIUM TECHNOLOGY DEVELOPMENT PROGRAM
MASS TRANSFER INVESTIGATION
IN LIQUID METAL SYSTEMS
QUARTERLY PROGRESS REPORT NO. 14
JUNE 1970—AUGUST 1970**

U.S. ATOMIC ENERGY COMMISSION
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PROJECT AGREEMENT 15

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**SODIUM TECHNOLOGY DEVELOPMENT PROGRAM
MASS TRANSFER INVESTIGATIONS IN LIQUID METAL SYSTEMS**

Quarterly Progress Report No. 14
June 1970 - August 1970

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1. INTRODUCTION AND PROGRAM SUMMARY

1.1 TECHNICAL HIGHLIGHTS

1. The change in mechanical properties of 304 and 316 cladding due to exposure to flowing sodium are found to be about equal to the changes due to irradiation to 10^{23} nvt.

Further results of a series of long-term stress-rupture tests on Types 304 and 316 ss at 1150°F and 1300°F in flowing sodium are reported (Section 4). Reductions in rupture strength (projected to 30,000 hours) for Types 304 and 316 ss in 1300°F sodium are 60% and 53% respectively, compared to tests in argon. Rupture ductilities are reduced 50% after 3500 hours exposure. Effects at 1150°F are less well defined but are less than half as large as at 1300°F.

2. Transport of large fractions of carbon, nitrogen, and silicon is observed in austenitic loops with non-isothermal flowing sodium. The loss of minor elements from thin sections of materials such as fuel cladding indicates that useable mechanical properties will be dominated by these effects over much of the reactor core, and by the combined effect of radiation on the alloy (with composition as modified by exposure to sodium) in the peak flux regions.

Major loss of interstitial elements, particularly carbon, has been observed in the hot-leg of four runs. Up to 73% bulk loss for Type 304 ss and 48% loss for Type 316 ss is found after 4800 hours in 1300°F sodium. Up to 38% bulk loss in nitrogen and 30% loss in silicon are found for 316 ss after 5500 to 6600 hours. Transport of these elements has resulted in lowered stress-rupture properties and loss of ductility. No loss of carbon was recorded from stabilized grades of stainless exposed in the same loops.

3. The oxygen contributed to sodium by fresh stainless steel surfaces (such as new piping or new in-pile capsules) was found to be substantially lower than estimated. Oxygen meter measurements during loop start-up show about one-fifth as much oxygen availability as theoretical estimates based on oxide film thickness.

Oxygen removal studies were conducted on freshly cleaned stainless steel surfaces exposed to sodium during the start-up of new loop 3R (Section 2.8). The data from these studies establish that the levels of oxygen contamination from oxide films in sodium systems are more nearly in agreement with predictions noted in Russian literature than with upper limit estimates of oxide layer thickness. (This is of special significance to the design and start-up operation of large systems with slow cleanup rates and for sodium filled loops).

4. Radioactive buildup predictions appear to require taking account of particle transport and composition. Calculations based only on solution-precipitation models may be seriously incomplete.

Variations in composition were found in particulates collected from cooling zones of test loops. Analyses reported in Section 3 show transition metals, Ni, Cr, Mn concentrated within preferred areas. These observations suggest that deposition is affected by a combination of the local hydraulic conditions which control the adherence of particles, as well as the change of solubilities with temperature.

5. Lower values for amalgamation analysis for oxygen in sodium are not found after equilibrating glassware in dry argon.

Equilibration times ranging from 4 to 168 hours did not lower the determination of total alkalinity below a nominal 13 ppm ± 2 , contrary to observations of lower values made at EBR-II.

1.2 PROGRAM DESCRIPTION AND BACKGROUND

Corrosion of candidate structural materials, the influence of corrosion and of impurities in the sodium on engineering properties of materials, 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 support engineering design selections (when used in conjunction with irradiation data) and the establishment of specifications for impurity monitoring and control for large plants.

Current work is directed to the near-term data or development requirements of sodium technology for application to FFTF and LMFBR demonstration plant designs. Emphasis in scope is placed on deposition studies, design data for materials performance, and the associated requirements and methods for plant chemistry control.

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 number of materials studied has been extended to include cladding candidates for oxide fuel (Types 304, 316 and 347 stainless steels and Incoloy 800). The temperature range of testing has been 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.

Increasing emphasis has been placed on improving sodium impurity monitoring and control. In-line devices are

used to obtain rapid and, where possible, continuous indications of impurity changes. These include chromatography for cover gas, replaceable flow-through sodium samplers, electrochemical cells for oxygen activity and diffusion cells for hydrogen concentration in sodium. Provisions are being made for electrochemical cells for carbon.

Cold traps for control of sodium purity in test loops have been modified with electromagnetic stirring devices to increase cleanup rate and automatic controls are being added for continuous and unattended operation.

1.3 SUMMARY OF TASKS

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

Task I: Corrosion and Deposition

Objectives are improvements of empirical and rational models for the accurate prediction of corrosion and mass transport for design of primary and secondary sodium systems of large plants.

Task II: Sodium Impurities Determinations and Particulate Impurity Identification

This task has the objectives of developing improved

methods for 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 the development of rational models.

Task III: Inactive

Task IV: Corrosion and Mass Transport Effects on Composition, Structure, and Structural Integrity of Candidate Alloys for LMFBR's and Localized Attack

The objective is to obtain exposures of specific LMFBR candidate materials, including Types 304 and 316 stainless steel tubing, and to determine correlations between mass transfer and sodium impurity effects which affect 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 are installed in the Sodium Components Test Installation (SCTI) for this purpose.

2. TASK I: CORROSION-DEPOSITION STUDIES

2.1 GENERAL

Current work is directed toward the following:

- Experimental study of the corrosion, mass transport, and the resultant deposition occurring in pumped sodium systems of Types 304 and 316 stainless steel at top sodium temperatures of 1150°F and 1300°F and at ΔT 's of 400°F to 500°F similar to large reactor systems.
- 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 sodium cooled plants.
- Measurement of effects of hydrogen-oxygen interactions in a sodium system from the standpoint of affect on oxygen activity and monitoring oxygen with electrochemical meters.
- Establishment of 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 ANALYSIS OF DATA AND RESULTS

2.2.1 Depletion of N₂ from 316 SS

Nitrogen loss (similar to that of carbon) has been observed in hot leg corrosion specimens in mass transfer loops. The amount of N₂ loss from 316 SS samples is shown in Table 1.

Table 1

LOSS OF NITROGEN FROM 316 SS*

	Pre-Exposure	Post Exposure**	% Loss
FC51	0.047%	0.029%	38%
FF51	0.047%	0.038%	19%
GD01	0.047%	0.033%	30%

* N₂ analysis (bulk) by Kjeldahl Method

** 6600 hrs, 1300°F, 15 fps, <10 ppm oxygen sodium conditions

The loop was operated under argon cover gas with partial pressures of nitrogen ranging 10⁻³ to 10⁻² atmospheres.

Removal of nitrogen from the stainless steels under such low partial pressures of nitrogen in the cover gas is feasible.

The effect of N₂ loss on mechanical properties is not established. However, it is well known N₂ in solid solution stabilizes the γ -phase in austenitic steels. Hence, loss of N₂ (like carbon) from the steels could enhance formation of ferritic sigma phase, and decrease the ductility of the steels

as it has been observed in loops where degradation of mechanical properties are being studied.

2.2.2 Selective Leaching of Silicon from 316 Stainless Steels

A large amount of silicon as sodium silicate type compounds has been observed in particulates collected in micro-metallic filters. The source of silicon was obscure until the analyses of the hot leg (1300°F) corrosion samples showed between 15-30% of silicon was leached out from the hot leg samples after ~6600 hours of operation.

Analysis of the available thermodynamic data indicates that Na₂O.SiO₂ can exist in liquid sodium below 0.01 ppm of oxygen at the operating temperatures of the loop. Since the nominal oxygen levels of sodium in the particulate collection loop (1R) is about 5-10 ppm, the observation of sodium silicate type compounds in cold leg deposits is consistent with thermodynamic calculations. Considering the high stability of sodium silicates with respect to oxygen partial pressures, under normal loop operating conditions (1-10 ppm oxygen), it will be impossible to retard selective leaching of silicon from the stainless steels.

2.2.3 Hydrogen Monitoring and Cold Trap Efficiency Studies

A 15 mil thick 316 SS diffusion membrane is being used to monitor hydrogen in loop 9 (see Section 2.5). A 8.0 liter/sec capacity ion pump on the vacuum side of the membrane is used to determine the partial pressures of hydrogen permeating through the membrane.

A first attempt was made to calibrate the ion pump pressure with respect to concentration of hydrogen in sodium. The loop sodium was cleaned up with a clean-up cold trap operating at 270°F for several days. The clean up operation lowered the ion pump pressure to <10⁻⁸ torr (mm-Hg). A known amount of a mixture of 2% H₂ and 98% argon was introduced through a tube submerged in sodium in the surge tank at a temperature of 800°F. Due to slow reaction rate of H₂ with liquid sodium at 800°F, most of the H₂ did not dissolve in sodium and escaped into the cover gas, consequently quantitative addition of hydrogen was not successful. However, it was possible to introduce a finite amount of hydrogen via the gas phase. After the introduction of the hydrogen into the sodium, it was removed by cold trapping with a second clean cold trap. The diffusion membrane was then calibrated against various cold trap temperatures. Figure 1 shows the calibration curve at 1300°F, using the solubility data obtained at APDA given by the following equation:

$$\log C_H = 6.52 - \frac{3180}{T^{\circ}K} \quad \text{in PPM}$$

Figure 1. Relationship Between Vac-Ion Current and Concentration of H_2 in Sodium

A second series of experiments was carried out to determine the temperature coefficient of hydrogen permeation at constant concentrations of hydrogen in the sodium. During this experiment, the cold trap (containing NaH) temperature was raised in steps to 350°F (0.28 ppm H), 430°F (1.22 ppm H), 470°F (2.3 ppm H) and 510°F (4.14 ppm H). At each cold trap temperature the temperature dependence of the ion pump pressure was determined and is shown in Figure 2. The activation energy for the hydrogen permeation was found to be ~30 K Ca1 between 1000°F to 1300°F. The activation energy obtained in the present experiment is about 2.5 times higher than the published data of activation energy of diffusion of gaseous hydrogen through stainless steels which is about 12-13 K Ca1.

The present data indicates that diffusion of hydrogen through the stainless steel membrane is not the rate controlling step in this process. The higher activation is thought to be due to a thin layer of tungsten deposited on the inner surface of the diffusion tube from previous use as an anode for an electron bombardment heater. Further experiments are in progress to verify the present results, since they are in apparent disagreement with recently published results (ANL/ST-2), where permeation of hydrogen through a nickel membrane below 1000°F was found to be controlled by diffusion of hydrogen through the membrane.

2.2.4 Cold Trap Efficiency Studies

The results of the clean up studies in loop 9 (Run 9-2a), that were reported in earlier quarterlies, were not quantitative as the membranes were not calibrated. Figure 3 shows a plot of recent hydrogen clean up rate studies.

The figure shows the increase of the removal rate for hydrogen with increase of the Reynolds Number in the cold trap (by electromagnetic stirring). Quantitative studies of cold trap efficiency with respect to removal of oxygen (as monitored by oxygen cells) are being carried out at present.

2.3 OPERATION OF TEST FACILITIES

A summary of loop operations including conditions of tests and operating times is shown in Table 2 for the 3-month period ending August 21, 1970.

2.4 LOOP 8 PARAMETER STUDIES (Run 8-6)

The purpose of this run is to determine corrosion data and movement of interstitials (especially carbon movement), at high oxygen levels and to obtain the following information relative to the chemistry of the system:

1. Hydrogen-oxygen interaction effects.
2. Cold trap efficiency with respect to oxygen removal.
3. Oxygen meter calibration at high oxygen levels in sodium and comparison of results with on-line vacuum distillation results.

The data provided by Run 8-6 will complete the extension of corrosion effects to high oxygen at 1300°F

Table 2

SUMMARY OF LOOP OPERATIONS May 22, 1970 to August 21, 1970

	Temp. (°F)	ΔT (°F)	Flow (gpm)	T _s or CT (°F)*	Time (Hours)
Run 8-6	1300	400	5.7	<432	2,000
Three-Month Operation					477
Cumulative, Run 8-6P				<300	1,205
Total Loop Age					25,475
Run 9-2A	1300	500	5.0	425	~4,000
Three-Month Operation					2,005
Cumulative, Run 9-2A					5,024
Total Loop Age					20,363
Run 1R-3	1300	800	2.0	432	2,000
Three-Month Operation					571
Cumulative, Run 1R-3					336
Total Loop Age					6,598
Run 3R-1	1300	800	0.3	270	500
Three-Month Operation					184
Cumulative, Run 3R-1					0
Total Loop Age					184
Run 4R-2	1300	500	6.5	<300	10,000
Three-Month Operation					2,180
Cumulative, Run 4R-2 – 1000°F & above – 1300°F & pressure applied					13,134
Total Loop Age					12,678
					15,036
Run 5R-5	1300	500	10.0	<300	5,000
Three-Month Operation					1,914
Cumulative, Run 5R-5 – 1000°F & above – 1300°F & pressure applied					4,763
Total Loop Age					4,505
					20,212
Run 6R-4	1150	400	7.0	<300	10,000
Three-Month Operation					2,183
Cumulative, Run 6R-4 – 1000°F & above – 1300°F & pressure applied					7,394
Total Loop Age					7,054
					15,879

* – Plugging Temperature

CT – Temperature of Cold Trap

and establish the oxygen dependence term in the corrosion model. Carbon movement resulting from the high oxygen activity will receive major study. Measurement of bulk sample carbon contents, analysis of particulate material

Figure 2. Hydrogen Permeation Data vs Sensor Temperature

Figure 3. Cold Trap Cleanup

deposited on surfaces, and x-ray fluorescent data on surface layers will determine whether changes in carbon activity between hot and cold legs (expected because of a shift in chromium transport) will effect a different pattern of carbon transport.

During this period, the following activities were carried out:

1. Completed a 1205-hour test for the purpose of determining the residual carbon activity from the previous test. Loop purity was monitored with plugging indicator, UNC oxygen meter, on-line distillation, flow-through samplers, and on-line chromatograph.
2. The corrosion samples were processed for metallurgical (carbon) examination.
3. Samples of loop piping and/or liner were removed for determination of carbon content and to establish a comparison with the tabs taken from adjacent zones. The loop regions sampled included:
 - a. Hot leg — Zone E (liner).
 - b. Hot leg — Zone F to G crossover.
 - c. Cold leg — Zone K outlet (Cooler #2 outlet).
4. Completed fabrication, processing, and cartridge assembly of samples. Began installing samples in the loop on August 18, 1970.
5. Completed cleanup and overhaul of on-line distillation system.
6. Made preparations for carbon meter installation between the outlet of Cooler #2 (cold leg) and the cold leg surge tank. The design of piping and related sodium components has been completed, as has the design of electrical controls and instrumentation for temperature control and readout of carbon meter. The carbon meter connection to the loop is planned to occur after ~1000 hours of Run 8-6.

Startup of high oxygen Run 8-6 is planned for September.

2.5 LOOP 9 OPERATIONS

The purpose of the current test (Run 9-2a) is to obtain corrosion data at higher oxygen levels (~425°F) and to determine relationships between cold trap operation and changes in the chemistry of the system.

The following activities were carried out during the quarter:

1. Cold trap efficiency studies were performed on the effect of increased Reynolds Number, obtained by magnetic stirring, with respect to removal of hydrogen species introduced in dilute gaseous form via the cold trap. The loop was cleaned with cold trap #1 at 270°F prior to addition of the hydrogen.
2. Calibration of the diffusion type (stainless steel tube) hydrogen detector as a function of sodium temperature past the sensor for various cold trap temperatures

was carried out. The sodium is assumed to be saturated with hydrogen based on solubility data by APDA⁽¹⁾.

3. Installed and checked out equipment to provide continuous addition of moisture to loop cold leg. The purpose of this is to establish constant O₂ and H₂ concentrations in the loop for further cold trap effectiveness studies and to obtain some preliminary information on water leak rate criteria. At conclusion of these studies, the present run will be terminated.
4. Initiated preparations for Run 9-3, including:
 - a. Modified design for high flux heater test section.
 - b. Fabrication of corrosion samples.
 - c. Initiated procurements of spare high flux heaters and test section materials.

The main objective of Run 9-3 is to obtain high precision definitive measurements of the effect of high heat flux (0.8×10^6 Btu/hr/ft²) on corrosion at low oxygen conditions. Higher precision measurements involve:

1. Control of oxygen (± 2 ppm) measured by chemical analysis and monitored by oxygen meter and plugging indicator. A new cold trap control system (described in GEAP-13539-13) has been assembled, and is undergoing checkout. It will be applied to the cold trap during this run to maintain cold trap temperature $\pm 2^\circ\text{F}$.
2. Corrosion determination by weight change of removable samples placed over the heated zone (i.e., heater anode).
3. Comparison of high heat flux samples with unheated specimens in a parallel test section exposed to sodium in an isothermal zone at comparable temperatures and velocity. The geometry of the samples will be the same in both locations.

2.6 LOOP 1R PARTICULATE STUDY

The objective of this study is to generate, collect, and analyze relatively large quantities of particles of corrosion products under controlled conditions, and develop a basic understanding of their role in sodium mass transfer, and effects in deposition areas and means for control and removal.

During this period, preparations for Run 1R-3 at high oxygen were completed. These included:

1. Installation of an oxygen meter loop containing two cells. This loop is located in parallel with the cold leg.
 2. Processing and loading of corrosion samples.
 3. Installation of 5 micron filter elements in the main filter housing.
- (1) S. A. Meecham and E. F. Hill, "The Solubility of Hydrogen in Liquid Sodium," Paper #199 presented at New York meeting of Division of Physical Chemistry, American Chemical Society, September 11, 1969.

4. Wiring and checkout of trim heater temperature control system, using proportional control elements for smoother control.

On July 31, 1970, the loop was filled and operated at 750°F maximum for a period of ~115 hours while cold trapping at 270°F, to establish loop purity level and to filter the sodium of particulates which may have been introduced during the prior loop modifications. The loop was then shutdown and the filter was removed and replaced with a new assembly. Following restart of the loop on August 6, 1970, hot leg temperatures were increased to 1300°F and, on August 14, 1970, the cold trap temperature was set to 432°F to establish the high oxygen conditions for this run. (T_s equivalent to 20 ppm based on solubility data by Eichelberger.) A new cold trap control system (see GEAP-13539-13) will be applied to this loop to maintain the close control ($\pm 2^\circ\text{F}$) required.

The oxygen meters will be placed in operation pending completion of the controls and instrumentation for temperature control and cell readout.

Sampling of the main filter is planned at 600-hour intervals and monitoring of the #2 economizer temperatures twice daily is being carried out to establish possible fouling of the surfaces with particulates.

2.7 LOOP 3R — RATIONAL MODEL EXPERIMENT

The Rational Model Experiment was designed to obtain scale-up data from small test loops to large systems with sodium velocity at sample surfaces and oxygen as the primary parameters.

Specific requirements included:

1. Simulate velocity profiles close to walls of typical sodium system components, and study how their shape may influence the corrosion process.
2. Test performance of electrochemical oxygen meters with air reference electrodes.
3. Determine how different oxygen impurity levels will influence the leaching of different alloy elements on a sodium exposed stainless steel surface.

An isometric of the loop is shown in Figure 4. During this period, loop operation began on July 24, 1970.

2.8 LOOP 3R STARTUP OPERATIONS

A test to measure the oxygen removed by sodium from new stainless steel surfaces was completed to 620°F. The measurement was made by exposing 10.1 ft² (0.94 M²) of fresh stainless steel surface to loop sodium and measuring the change with an oxygen meter. The freshly cleaned and dried surface was contained in a fixture inserted in place of the flow-through sampler, Figure 4.

Preliminary results indicate the oxygen reduced by sodium to 620°F from fresh, cleaned stainless steel surfaces is in the range of $0.4\text{--}5.8 \times 10^{-2}$ gm/M² and is in the same range as predicted by Ivanovskii⁽¹⁾. Individual operations and results are summarized as follows:

1. When the specially prepared test fixture was inserted in loop 3R, the change in loop oxygen by air reference oxygen meter measurement was equivalent to 0.7 ppm. This resulted from exposing 0.94 M² of fresh stainless steel surfaces to 620°F sodium for 18 hours. The oxygen contribution to 5500 grams of sodium corresponds to 0.41×10^{-2} gm/M² of stainless steel surface.
2. A comparative measurement of oxygen change was made utilizing the change in oxygen meter reading when the cold trap was inserted in the loop. The change was equivalent to 5.5 ppm oxygen contributed to 5500 grams of sodium, and resulted from exposing ~1.07 M² of acetone cleaned stainless steel surfaces (mostly wire mesh), to 570°F sodium for one hour. The oxygen contributed from the surfaces corresponds to 2.8×10^{-2} gm/M².
3. When the test fixture was inserted in the loop circuit following cold trapping at 250°F, a plugging indicator run was made which indicated two flow breaks. The lower temperature break occurred at approximately 388°F. Based on oxygen solubility data by Eichelberger, the change in oxygen from 250°F, T_s to 388°F, T_s is 10 ppm. This corresponds to an oxygen contribution of 5.8×10^{-2} gm/M² from the 0.94 M² stainless steel surfaces.
4. The data from 1, 2, and 3 above are correlated with each other and with information from other sources in the following tabulation:

Source— Method	Area (M ²)	Sodium Inventory (gms)	Surface Temp. (°F)	Oxygen Change (ppm)	Equivalent Oxygen Film (gms/M ²)
Fixture— Loop 3R (by O ₂ meter)	0.94	5500	620	0.7	0.41×10^{-2}
Cold Trap— Loop 3R (by O ₂ meter)	1.07	5500	570	5.5	2.8×10^{-2}
P.I.—Loop 3R (Na ₂ O solubility data)	0.94	5500	620	10	5.8×10^{-2}
Ivanovskii (Ref. 1)	—	—	Not re- ported	—	$\sim 5 \times 10^{-2}$

(1) Ivanovskii, M. M., et al, "The State and Distribution, Monitoring, and Elimination of Impurities in Circulating Alkali Metal Coolants" (a paper presented at the IAEA Symposium at Vienna, December, 1966).

Figure 4. Sodium Mass Transfer Rational Model Studies — Loop 3R

2.9 H₂-O₂ EQUILIBRIA STUDY

The objective of this study is to determine the effects of hydrogen in sodium on oxygen activity (and vice versa). The overall aim is to obtain information which will enable better interpretation of data obtained from on-line instrumentation and samplers (oxygen meter, hydrogen meter, carbon meter, plugging indicator, chromatograph, and distillation), and better application of these devices, as well as purity control devices (cold trap and hot trap), for establishing and maintaining the levels of these impurities in sodium loops.

During this period, the pot was filled and circulated for cleanup by Loop 6R cold trap. Following this, the pot and associated instrumentation were disconnected from the loop for conduction of tests to calibrate oxygen meters and hydrogen diffusion cells.

2.10 MASS BALANCE LOOP 2R

The purpose of this loop is to study the deposition process of corrosion particles. Experience from Loop 1R and others show that the different alloy elements of stainless steel precipitate at various regions in the cold leg of a loop. It is important to know where in the system the corroded material will be deposited because these will be local areas with degradation in heat transfer properties and subsequently high radioactivity, probably in the intermediate heat exchanger.

A preliminary design was finished and sketches made for the main components. The loop isometric is shown in Figure 5 and the technical design data are given in Table 3.

The flow cross-section of the loop with samples is kept uniform with the exception of the pump and flowmeter. The downstream factor is excluded because there are essentially no isothermal sections. Uniform sample placement and uniform flow stream are used to facilitate locating the area of saturation of the alloy elements along the sodium stream.

The temperature profile around the loop is a sine curve with the upper and lower limit at 1300°F and 800°F respectively. Any other range is possible within this value.

The maximum heat flux in the hot leg, as well as in the cold leg, is 365,000 Btu/ft²hr. With these features, it is possible to approximate the temperature profile and heat flux of a sodium-cooled reactor design.

The sodium flow will be 10 gpm. This flow will be achieved with a modified AC EM pump, employing a one pass pump cell. To achieve the high heat flux rates and the desired temperature profiles eight profiled resistance heaters will be used in the hot leg, and in the cold leg, the heat will be transferred to a secondary sodium system in a sodium-to-sodium profiled heat exchanger.

In the sodium-to-sodium heat exchanger, the stream of 10 gpm in the secondary system is fed in at the center and branched off in a ratio of 7 gpm to 3 gpm at maximum loop capacity to achieve the temperature profile required. Both the primary and the secondary loop will be equipped with a purification system, each one containing oxygen meters, cold traps, and plugging indicators.

Table 3

TECHNICAL DATA OF LOOP 2R

Primary Loop

Maximum Temperature	1300°F
Minimum Temperature	800°F
Sodium Flow	10 gpm
Maximum Heat Flux (Cold and Hot Leg)	365,000 Btu/hr ft ²
Temperature Distribution	Continuous Sinusoidal

Secondary Loop

Maximum Temperature	1250°F
Minimum Temperature	470°F
Main Sodium Flow	10 gpm
Hot Leg Sodium Flow	3 gpm
Cold Leg Sodium Flow	7 gpm
Cooling Air Required	4000 Cfm

Required Power for Total Loop	~300 KW
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Figure 5. Mass Balance Test Loop

3. TASK II: SODIUM IMPURITIES DETERMINATIONS AND PARTICULATE IDENTIFICATION

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 SAMPLING OF LOOPS AND DETERMINATION OF IMPURITY LEVELS

1. Total Alkalinity Data

The total alkalinity measurements from routine loop samples are shown in Table 4.

Table 4

TOTAL ALKALINE IMPURITIES IN LOOP SODIUM (Flow-Through Samples — Amalgamation Determination)

Sample No.	Date Taken	Total Alkalinity (ppm)
4R-2-18A	4-16-70	11
5-5-6A	4-13-70	13
5-5-7A	5-4-70	7
6R-4-11A	4-2-70	12
6R-4-12A	4-17-70	14, 10
6R-4-13A	5-12-70	13
8-6-7A	3-18-70	19
8-6-8A	4-2-70	10

With the exception of one value from loop 8, 19 ppm, the data is in the usual range.

2. Equilibration Equipment

Recent work at EBR II* has shown that long term equilibration in the glove box atmosphere of glassware to be used in the amalgamation determination of "oxygen" yields lower results than data obtained when the glassware is equilibrated for only short periods. An initial test of this modification of the procedure with our system did not yield a similar result. Table 5 shows data obtained for analyses obtained on a single sample for varying equilibration times for the glassware. The data obtained is the same within the usual error.

Table 5

EFFECT OF GLASSWARE EQUILIBRATION IN OXYGEN (Total Alkalinity) Values (ppm)

Glassware Equilibration Time (hours)	Sample 6-4-8A "Oxygen" (ppm)
168	15
20	11
4	14

* ANL 7679, March 1970.

3. Equipment has been assembled for the determination of hydride hydrogen and carbonate in flow-through samples for the purpose of making correction of the amalgamation data to yield a value closer to the true oxide value.

4. Carbon Determination

- A single sample, 6R-4-11A, has been analyzed for total carbon. The result obtained was 27 ppm.
- Two further tests have been made on loop 8 cold trap deposits in search of the cause of the extensive carburization that occurred during loop Run 8-5.

Infrared analyses were made of pyrolyzed black insoluble material obtained by reacting cold trap sodium with water. The volatiles were condensed and examined. No evidence of organic matter was found.

An analysis for cyanide was also performed and showed 10 ppm carbon was present in this form. This is low relative to the amounts found in deposits in a ferritic system (where carburization was taking place) cold trap. This relatively low value leads to the conclusion that this species was not involved in loop carburization.

3.2 SEPARATION AND IDENTIFICATION OF PARTICULATES

1. Residues from Loop 1 Filters

Electron microscopic techniques have been explored as a means for supplementing x-ray diffraction analysis for the characterization of particulates obtained from loop 1. Electron microprobe x-ray fluorescence analysis has been used in conjunction with these studies.

The materials examined were residues obtained after removal of sodium by distillation from filters which had seen service in loop 1. The sampling history and the results of x-ray diffraction and bulk chemical analysis have been presented in the previous quarterlies in this series.

Techniques for sample preparation and loading in a dry, oxygen free atmosphere were developed prior to the application of scanning electron microscopy (SEM), transmission electron microscopy (TEM) and electron diffraction.

The bulk of the particles were large opaque agglomerates with size in the range of 1 to 100 microns. A scanning electron microphotograph is shown in Figure 6. In addition there were smaller, 1 to 10 micron, irregular clusters and individual particles in a variety of shapes. Figure 7 shows a representative

particle size ~60 microns

*Figure 6. SEM Photograph of Agglomerates from Loop 1 Filter
3000 X*

particle size 1-10 microns

*Figure 7. SEM Photograph of Smaller Particle Clusters from Loop 1 Filters
3000X*

selection of these particles. Transmission electron microphotographs of typical large and small particles are shown in Figures 8 and 9 respectively and indicate the presence of both spongy opaque particles and transparent and clearly crystalline species.

It appears possible that the agglomerates are comprised of the smaller particles and it is conceivable some coalescence may occur during the distillation step after sodium has been volatilized.

Although electron diffraction patterns were obtained for a number of particles the data was not sufficiently clear to permit identification of the compounds present.

Electron microprobe examination of some of the larger particles indicated two predominant types: those with high sodium, silicon and oxygen contents (plus low concentrations of transition metals, Cr, Mn, Fe, Ni) and less frequently, those made predominantly of iron with silicon and other trace metals present. The transition metals appear to concentrate in certain preferred areas within each particle while Na, Si and O are more evenly distributed.

It was also noted that some of the particles contracted under the beam and the shrinkage was accompanied by the loss of sodium and oxygen. On the other hand some particles containing high quantities of sodium and silicon were stable under the beam. These observations are consistent with the presence of sodium oxides (volatile?) and silicates (not volatile).

In summary this initial application of electron microscopic techniques has revealed the complex morphology of the impurities in these residues. The electron microprobe analysis has provided some clues as to the compositional characteristics.

Further exploration of these techniques is warranted. Electron diffraction, however, does not appear to provide a useful line of attack.

2. Source of Silicon in Particulates

Because of the high concentration of silicon observed in loop 1 particulates, corrosion samples (rings) were analyzed for silicon content. A sample (316 SS) unexposed to sodium contained 0.42% Si while three samples which had seen sodium flow at temperatures of $\sim 1300^{\circ}\text{F}$ for several thousand hours contained 0.29, 0.35 and 0.29% silicon respectively (the analytical error is on the order of $\pm 0.05\%$). On the basis of these data it appears that silicon is depleted in the steel by exposure to sodium and is the probable source of silicon in the particulates.

3. In-Line Distillation

An in-line distillation of a large sample (500 gram) was attempted. Although some residue was accumulated it was only a fraction of that expected based on the concentration of elemental impurities in the loop 8 sodium. The cause of the loss is being investigated. Nevertheless sufficient residue was obtained to permit x-ray diffraction analysis which showed the presence of a major phase of α -iron and appreciable quantities of Na_2CO_3 . Traces of Cr_7C_3 and NaMO_2 also appeared to be present, but the quantities were too small for positive identification. These data reflect 800°F impurities and resemble data obtained from economizer deposits collected in the $300\text{--}700^{\circ}\text{F}$ region. The data encourages the continued investigation of distillation as a tool for particulate accumulation.

3.3 INTERACTION STUDIES

1. Hydrogen-Oxygen Pot Study, Meter Calibrations (see Section 2.9)

The objective of this work is to improve monitoring of loop purity by assessing the effects of hydrogen-oxygen interactions on the response of activity meters for these species.

The system consists of a 2 liter capacity pot equipped with oxygen meters and a gettering device for oxygen (U foil); a diffusion tube for hydrogen removal coupled with a sputter-ion pump and a residual gas analyzer for hydrogen measurement; a chromatograph system for the analysis of hydrogen in the cover gas; and equipment for the introduction of known quantities of hydrogen and oxygen gas.

The pot has been filled with sodium and transferred to the laboratory with the required instrumentation. Initial installation has begun.

2. Refractory Wires — Oxygen-Sodium Study

The three metal wires, iron, chromium and nickel, which had been suspended in liquid sodium (~ 400 hours, 600°C) containing 100 ppm oxygen failed to show any oxide product. (The nickel wire pattern contained a few lines which did not belong to nickel metal. These matched some lines in the pattern for the high temperature form of sodium nickelate which is unlikely to be present.) Chromium nitride appeared to have deposited on the chromium wire which may be evidence that the boron nitride crucibles had undergone oxidation rather than the wires.

particle size ~60 microns

*Figure 8. Transmission Electron Image of Agglomerates from Loop 1
500X*

2 to 3 microns

*Figure 9. Transmission Image of Small Crystal from Loop 1 Filter
26,000X*

4. TASK IV: CORROSION AND MASS TRANSPORT EFFECTS ON COMPOSITION, STRUCTURE, AND STRUCTURAL INTEGRITY OF CANDIDATE ALLOYS FOR LMFBR'S AND POSSIBLE LOCALIZED ATTACK

4.1 GENERAL

The objective is to obtain effects of mass transfer on corrosion and structural integrity of alloys in flowing sodium systems with temperature differences. Work under this task includes the testing of Types 304 and 316 stainless steel to obtain long-term stress-rupture (to 10,000 hours) data. The present test conditions are 1150°F and 1300°F T_{max} , sodium velocities to 17 ft/sec, and controlled sodium purity corresponding to a plugging temperature <300°F.

In addition to the commercial mill-annealed grades of Types 304 and 316 stainless steel, 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 COMPOSITION AND STRUCTURAL EFFECTS ON MECHANICAL PROPERTIES

4.2.1 Loop 4R — Corrosion Effect on Stress Rupture, Run 4R-2, 1300°F

The loop operated at test conditions during the report period. The run reached 12,510 hours at 1300°F with the pressure applied, as of August 14. There were two failures during the report period. Fourteen of the original sixteen samples have failed. The results to date are shown in Table 6.

Table 6

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

Material	Initial Hoop Stress, psi	Time-To-Rupture, Hrs
304CA	7000	2188
304MA	7000	2198
304MA	7000	2237
304CA	7000	2263
316MA	8000	3822
316MA	8000	4415
316CA	8000	5162
316CA	8000	5175
304CA	5000	4500
304MA	5000	5187
304CA	5000	6228
304MA	5000	6546
316CA	6000	10852
316MA	6000	10882

MA - mill annealed

CA - carbide agglomerated, 1650°F for 24 hours

4.2.2 Loop 5R — Corrosion Effect on Stress Rupture, Run 5R-5, 1300°F

The loop operated at test conditions during much of the report period. A short period of downtime was experienced for replacement of a main loop pump. The run reached 4337 hours at 1300°F with the pressure applied as of August 14. There was one failure during the report period, which was the first failure of the original sixteen samples. The result to date is shown in Table 7.

Table 7

1300°F BIAXIAL STRESS RUPTURE DATA
FROM RUN 5R-5

Material	Initial Hoop Stress, psi	Time-To-Rupture, Hrs
304MA	5500	3939

4.2.3 Loop 6R — Corrosion Effect on Stress Rupture, Run 6R-4, 1150°F

The loop operated at low temperature (~700°F) conditions during most of the report period for operations with the H₂-O₂ monitoring pot. The run reached 6886 hours at 1150°F with the pressure applied, as of August 14.

There were no failures during the report period. Nine of the original sixteen samples have failed. The results to date are shown in Table 8.

Table 8

1150°F BIAXIAL STRESS RUPTURE DATA
FROM RUN 6R-4

Material	Initial Hoop Stress, psi	Time-To-Rupture, Hrs
304MA	20,000	514
304MA	20,000	559
304CA	20,000	1662
304CA	20,000	4006
316MA	20,000	639
304MA	18,000	1710
304MA	18,000	1947
304CA	18,000	5391
304CA	18,000	6235

4.2.4 Stress Rupture Control Tests

Stress-rupture tests are being run at 1300°F in argon on mill-annealed and carbide-agglomerated Types 304 and 316 SS. The tests are being run for the purpose of supplying comparison (control) data for the tests in flowing sodium. The same heats of materials are being tested in both

environments; whereby, the environmental effect(s) can be delineated. The results to date are shown in Table 9.

Table 9

**1300°F BIAXIAL STRESS RUPTURE DATA
IN ARGON**

Material	Initial Hoop Stress, psi	Time-To-Rupture, Hrs
316CA	15,000	925
316MA	15,000	1375
316CA	13,000	1584
316MA	13,000	2415
316MA	11,000	3183
316MA	11,000	4551
316CA	11,000	4874
316CA	11,000	9279
304CA	11,000	718
304MA	11,000	829
304MA	11,000	901
304MA	9,000	2511
304CA	9,000	2727
304CA	9,000	2895
304MA	9,000	3373
304CA	9,000	3389
304MA	9,000	4374
304MA	8,000	3380
304CA	8,000	4165
304CA	8,000	5965

The results, plus those for Runs 4R-2 and 5R-5 and the previously reported data from Loop 5R⁽¹⁾, are plotted in Figures 10 and 11. On the basis of comparison with the control tests, there is a definite decrease in long-term stress-rupture strength due to exposure in 1300°F nonisothermal flowing sodium.

Samples of Types 304, 316, 321, 347 SS, and Incoloy-800, 0.250" O.D. X 0.015" wall seamless tubing, have been exposed to 1300°F sodium for various lengths of time in the unstressed condition. The biaxial stress-rupture testing

of these samples in 1300°F argon has been initiated, and the preliminary results are compared to the argon results for previously unexposed material in Figures 12 through 16.

The rupture strength of the sodium exposed Types 304 and 316 SS has been reduced. The strength reduction increases with increased sodium exposure, although it is difficult to distinguish between 500, 1000, and 1700 hours exposure. However, the stress-rupture curve for a particular sodium exposure has the same slope as the unexposed data. It has been established⁽²⁾ that Types 304 and 316 SS undergo decarburization due to exposure in 1300°F nonisothermal flowing sodium. These results are predictable on the basis of increasing bulk decarburization with increasing sodium exposure. In effect, the results are a series of curves for Types 304 and 316 SS with various carbon contents.

The rupture strength of the sodium exposed Types 321 and 347 SS also shows a reduction. The strength reduction appears to be more dependent on the sodium exposure time, Figure 15. However, it is known, at least for Type 347 SS, that the stabilized grades of austenitic stainless steel do not decarburize in 1300°F nonisothermal flowing sodium. This and the fact there is a significant effect of the carbide agglomeration heat treatment on the rupture properties at 1300°F not noted with Types 304 and 316 SS, indicate that an aging process is occurring, which is drastically reducing the rupture strength.

The statements made concerning Types 321 and 347 SS also apply to Incoloy-800 with one exception; the rupture strength appears to increase after some period of exposure to 1300°F sodium. However, it is well known that Incoloy-800 will age harden in this temperature range, which would increase the rupture strength and tend to offset any other mechanism producing a reduction in rupture strength.

(1) PA-15 "Quarterly Progress Report #9" GEAP-10036, June, 1969.

(2) PA-15 "Quarterly Progress Report #13".

Figure 10. 1300°F Biaxial Stress-Rupture Properties of Type 316 SS in Sodium and Argon

Figure 11. 1300° F Biaxial Stress-Rupture Properties of Type 304 SS in Sodium and Argon

Figure 12. 1300°F Biaxial Stress-Rupture Properties of Type 304 SS in Argon

Figure 13. 1300°F Biaxial Stress-Rupture Properties of Type 316 SS in Argon

Figure 14. 1300°F Biaxial Stress-Rupture Properties of Type 321 SS in Argon

Figure 15. 1300°F Biaxial Stress-Rupture Properties of Type 347 SS in Argon

Figure 16. 1300° F Biaxial Stress-Rupture Properties of Incoloy-800 in Argon

5. LIST OF SODIUM MASS TRANSFER PROJECT REPORTS

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