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JUNE 1969

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**SODIUM COMPONENTS DEVELOPMENT PROGRAM
MASS TRANSFER INVESTIGATIONS
IN LIQUID METAL SYSTEMS
QUARTERLY PROGRESS REPORT
NO. 9
MARCH-MAY 1969**

U.S. ATOMIC ENERGY COMMISSION
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PROJECT AGREEMENT 15

BREEDER REACTOR DEVELOPMENT OPERATION
GENERAL ELECTRIC
SUNNYVALE, CALIFORNIA

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SODIUM COMPONENTS DEVELOPMENT PROGRAM
MASS TRANSFER INVESTIGATIONS IN LIQUID METAL SYSTEMS

Quarterly Progress Report No. 9
March - May 1969

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SECTION I

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 2-1/4 Cr-1 Mo and 5 Cr-1/2 Mo-1/2 Ti ferritic steels were studied in depth at sodium temperatures to 1200°F. More than 2500 samples of these materials were exposed to sodium during 140,000 hours of loop operations. Average corrosion rates were correlated by statistical analysis from which quantitative 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 have been extended to include cladding candidates for oxide fuel (Types 304, 316, and 347 stainless steels (SS), and Incoloy-800). The temperature range of testing has been increased to 1300°F, and temperature profiles, loop geometry, and sodium purity controls have been established to represent environments of large fast reactors in an improved way.

1.2 SUMMARY OF TASKS

Work in Fiscal Year 1969 in the Sodium Mass Transfer Program is organized into five tasks. These tasks and their objectives are as follows:

TASK I - LMFBR/FFTF CORROSION
PARAMETER STUDIES

Objectives are determination 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 INTER-
ACTIONS AND IDENTIFICATION
OF PARTICULATE MATTER

This task includes the three development efforts: sodium system sampling, analytical chemistry, and collection and identification of particulate material. Objectives are to insure 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 - MECHANICAL PROPERTIES
LIMITS FOR CLADDING
MATERIALS AND LOCALIZED
ATTACK**

The objective is to obtain effects of mass transfer and structural changes of materials on usable mechanical properties in flowing sodium systems with temperature differences. Candidates for LMFBR cladding, including Types 304 and 316 stainless-steel tubing, are being tested for stress rupture and stress fatigue lifetimes.

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.

**1.3 SUMMARY OF SIGNIFICANT RESULTS
DURING REPORTING PERIOD**

**TASK I - LMFBR/FFTF CORROSION
PARAMETER STUDIES**

Evaluation of corrosion and deposition data from Run 8-4 at 1300°F showed that adjacent surfaces of pure iron and Type-316 stainless steel developed similar compositions. The results indicated a trend for a quasi-equilibrium to cause all surfaces at equivalent positions (downstream) in a sodium system to reach a uniform surface composition under a steady-state condition.

Examination of patterns of deposition of chromium on surfaces and increase in bulk carbon content of the steel gave further indication that carbon movement in an austenitic system is associated with chromium movement and that the

transferring species is a complex chromium carbide.

A steady operation of 3426 hours of an electron bombardment heater has been attained with 500,000 Btu/h-ft² heat flux in flowing sodium at 1300°F.

**TASK II - SODIUM-IMPURITIES INTER-
ACTIONS AND IDENTIFICATION
OF PARTICULATE MATTER**

Data obtained from distillation of sodium from an operating loop have for the first time provided a straight-forward determination of carbonate because of the large samples (50 grams) obtainable by in-line distillation. The results show that values for total alkalinity, normally ~12 ppm, represent ~6 ppm oxygen as sodium oxide when correction for carbonate is made.

The cold trap was shown to efficiently remove hydrogen from a test loop after excursions caused by increasing temperature of the cold trap. Concentrations of hydrogen to ~6000 ppm in the cover gas, monitored by chromatography, were reduced to <100 ppm in 36 hours.

Calibration curves were obtained for response of an electrochemical oxygen meter to changes in ambient and sodium temperatures. The temperature coefficients measured were -0.30 MV/°F for ambient and +0.26 MV/°F for sodium.

**TASK IV - MECHANICAL PROPERTIES
LIMITS FOR CLADDING
MATERIALS AND LOCALIZED
ATTACK**

Results of tests of mechanical properties at 1300°F in two loops

continue to show that initial microstructural condition (carbide agglomerated vs mill annealed) of Type-304 stainless steel does not appear to have an effect on the stress-rupture properties.

A reduction in long-term stress-rupture properties in nonisothermal sodium at 1300°F is indicated by comparison of the test data to data obtained in other environments.

Evaluation of preliminary data on stress-fatigue from Loop 6R shows a retardation of long-term rupture of 304 and 316 stainless steel. The stress-fatigue results, based on mean stress, compare favorably with constant stress results from Loops 4R and 5R.

Corrosion data comparing the alloy types, 304, 316, and 347 stainless steel, and Incoloy 800, have been obtained to 4178 hours at 1300°F. Types 304 and 316 stainless steel continue to exhibit the lowest corrosion rates of the alloys tested.

1.4 REPORTS

Monthly report letters are prepared reporting progress in all tasks. Progress letters through April 30, 1969 have been issued.

In addition to the progress letters, 28 topical reports and 8 quarterly progress reports covering test results have been issued (see Section V).

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

SECTION II

TASK I - LMFBR/FFTF CORROSION PARAMETER STUDIES

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.
- Determine the effect of changing mass flow (gpm) at constant velocity on corrosion rates.
- Determine carbon transport in an all-austenitic system.
- Determine the effect of alloy composition on corrosion and deposition processes.
- Calibrate a chromatograph in the loop for concentration of hydrogen in the cover gas as a function of temperature of the cold trap.
- Seek correlations between sodium purity and high and low temperature breaks obtained with a standard plugging indicator.
- Observe the nature of the particulates deposited on the walls of the hot leg.
- Check out an in-line distillation unit for determination of oxygen and other impurities in sodium.

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.

The corrosion and weight change data for Run 8-4 are reported in quarterly report No. 8, GEAP-10008.

2.1 ANALYSIS OF CORROSION DATA AND MODEL DEVELOPMENT

The evaluation of all the data from Run 8-4 was completed during this period. The objectives for Run 8-4 follow:

- Determine the effect of velocity on corrosion and deposition in a system of Type-304 stainless steel and Type-316 stainless steel.

2.1.1 Loop Operating Parameters for Run 8-4

The nominal conditions for Run 8-4 were 3356 hours at 1300°F, $T_{\max}$ 800°F, $T_{\min}$ and oxygen content of < 10 ppm based on chemical analysis and plugging temperature (T_g) of 260°F to 300°F. Because of the presence of hydrogen (2-3 ppm) in the sodium, the "nominal" oxygen levels based on plugging temperatures and oxygen solubility data are not considered to be reliable. We prefer the oxygen levels based

on chemical analysis of the sodium by amalgamation and vacuum distillation as these have been proven by experiments to be more meaningful.

2.1.2 Corrosion Mechanism

Effects of velocity on corrosion rates of Type-316 stainless steel are consistent with previous runs as reported in quarterly report No. 6, GEAP-5693. A few samples of pure iron were placed in the hot leg of Run 8-4 to determine if the chemical composition of the surface affected the corrosion-deposition process. All the iron samples exhibited a small weight gain, shown by X-ray fluorescence (XRF) analysis to be due to precipitation of chromium, nickel, and manganese in the hot leg. In contrast, 316 stainless steel adjacent to the iron samples showed loss of nickel, chromium, and manganese. Figure 1 shows the pattern of nickel and chromium movement. It is expected that under a steady state condition, eventually all the samples (in the same downstream position) will have the same surface composition, which will be controlled by the activities of solutes in the flowing sodium. The XRF analysis of the iron samples in the hot leg showed ~6% nickel, ~3% chromium, and ~1.8% manganese in the surface layer. The composition of stainless steel surfaces in adjacent positions was ~13% chromium, ~11% nickel, and ~1% manganese. These results indicate a trend for a quasi-equilibrium to cause all of the samples to achieve a uniform surface composition under a steady state condition.

Corrosion of 316 stainless steel at 1300° F was found to be weakly dependent on mass flow (0.5 to 1.0 gpm) at constant velocity of ~3 fps. The data were obtained from a specially designed cartridge in Run 8-4. The results are reported in quarterly No. 8, GEAP-10008.

2.1.3 Mechanism of Deposition

The deposition pattern in the cold leg (Figure 1) indicates that the composition of the surface on which deposition occurs does not play a significant role in the deposition process. Deposition depends on the local degree of turbulence and on the activities of the alloy constituents in the solution or present as particles in the sodium. This argument is based on the fact that the quantity of Ni, Cr, and Mn depositing on the pure iron samples was about the same as that on the adjacent stainless-steel samples.

2.1.4 Carbon Transport

For comparative purposes, the results of the carbon transport for Runs 8-3 and 8-4 are shown in Figure 2. For both runs, the samples in the cold leg and coolers were carburized, and the samples in the hot leg lost carbon. The loss of carbon in the hot leg is attributed to the activity of carbon in sodium being considerably lower than that in the steel. However, in the cold leg, carbon is deposited from the stream on the substrate as a particulate. In a previous experiment $M_{23}C_6$ type carbides were observed in cold leg deposits, where M was mainly chromium. Figure 2 shows a strong relationship between movement of carbon and chromium. In Run 8-4 the maximum deposition of chromium occurred upstream to that of Run 8-3. This resulted in a shift in the area of maximum carbon deposition in the same direction (upstream). This is a further indication that carbon movement is associated with chromium movement and that the transferring species is a complex chromium carbide.

2.1.5 Effects of High Heat Flux

A total of 3426 hours of continuous operation have been accumulated on the Type-316

FIGURE 1. MOVEMENT OF ALLOY CONSTITUENTS IN TEST RUN 8-4

FIGURE 2. COMPARISON OF CARBON AND CHROMIUM MOVEMENT BETWEEN TEST RUNS

stainless steel anode of the electron bombardment (EB) heater in Loop 9. The heat flux remained relatively stable during the quarter at about 500,000 Btu/h-ft². Flow conditions were ~15 fps and 1300°F bulk sodium outlet.

An additional EB heater was installed and started up in Loop IR for the experiment to collect particulate material. Initial observations of heater pressure and cathode emission appear satisfactory.

2.1.6 Impurity Study (Loop 9)

The run plan and detailed specifications for an experiment to study hydrogen impurity in Loop 9 (Run 9-2a) have been prepared. A special cold trap was designed for this experiment in order to study efficiency of the trap for removal of hydrogenous compounds. The hydrogen in the sodium will be monitored by the pressure changes in the EB heater, and changes in the cover gas composition, by chromatography. Corrosion behavior of small amounts of pure Ni, Fe, Cr, and 2-1/4 Croloy and tantalum will also be observed in the run. An attempt will be made to study the mass transport at higher oxygen levels (~25 ppm).

2.1.7 Methods to Collect Particulates (Loop IR)

This experiment is designed to fulfill the following objectives for conditions of the run:

- To collect particulate material in a flowing sodium system at 1000°F to 600°F by using separations based on the physical size as well as the magnetic properties of these particulates.
- Determine composition of the particulates and identity of main compounds present.

- Determination of the role of particulate material in the corrosion-deposition process and of the factors which afford possible control of this process.

The two methods being used for collection of particulates are (a) passing the sodium through metallic filters with pores 5-10 microns in diameter, and (b) application of relatively strong (2300 gauss) magnetic fields in flowing sodium.

A description and diagram of the test are shown in quarterly report No. 6, GEAP-5693, pp. 65-66.

Following the initial shake-down run with a clean-up filter, two filters were inserted into the system. After about 400 hours of operation, the loop showed reduced flow. One of the upstream filters was removed for subsequent analysis of material collected.

Examination of the first filter used during the shake-down run showed presence of some particulates other than the filter material (pure iron). Sodium was removed from the filter by distillation. Emission spectrography of residue showed, in addition to iron, major constituents of nickel, chromium, and titanium. Iron and nickel determined colorimetrically were 27% and 14% respectively. The powder was magnetic and x-ray diffraction showed patterns for alpha and gamma iron types. The source of titanium is not known. Remaining filter material will be checked as a possible source.

2.1.8 "Rational Model" Experiment

Corrosion of stainless steel in sodium is known to be affected by the Reynolds number associated with the system and sample geometry. Reactor systems of the LMFBR type will be operating with Reynolds numbers of up to 100,000. Thus, it is essential that

corrosion rates be determined at levels higher than 30,000 (Reynolds), which has been the case up till now. To accomplish this, an experiment was designed to determine the corrosion rates of various steels at Reynolds numbers up to 100,000 and the effect of oxygen activity levels on corrosion. The total sodium inventory of the loop is approx-

imately 5 liters. The high Reynolds number will be obtained by rotating the corrosion samples in flowing sodium. A small loop incorporating rotating samples has been designed for operation at 1300 °F and 0.5 gpm to maintain a constant sodium quality at the position of the samples.

SECTION III

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 routine 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 of Loop Sodium

Analyses for total alkalinity were done on samples of the "Rapid Sampler" type taken from all five of the test loops during routine surveillance. Sample identifications and results are given in Table 1. The total alkalinity, assuming only Na_2O present, shown by the table is normally about 12 ppm $\pm$ 5 ppm of oxygen. The exceptions of high values noted in Table 1 were caused by either formation of sodium voids in the sample tubes which allowed contamination by the atmosphere during removal and handling or a cold trap temperature greater than 800° F for a short period of time when failure of a blower motor was detected.

Early results from distillation, shown below in Table 4, using large samples (50 grams) indicate that a large fraction of the total alkalinity as given in Table 1 is due to Na_2CO_3 . The oxygen present as Na_2O is expected to be in a lower range (~4 ppm).

3.1.3 Total Hydrogen of Loop Sodium

Values for total hydrogen taken from a series of flow-through samples are given in Table 2. The results are in the 1-4 ppm range and show no apparent trend.

3.1.4 Analyses for Metallic Impurities

Studies were initiated to evaluate the technique of "Rapid Sampling" for measurement of elemental impurities such as iron, nickel, chromium, etc. The questions to be answered include: do the metallic species deposit uniformly along the tube length; do they deposit on or near tube walls and how can they be removed for analysis without contamination?

Four separate samplers were sectioned to provide ~4 gram samples for analysis in order to establish any variability of results from samples taken at different positions along a tube. The samples were melted out of the sections of tube, and the remaining film was recovered by rinsing with alcohol. Results for iron, determined by a conventional colorimetric technique, are shown in Table 3. Although limited, the data indicate a possible position-to-position variability. Further tests planned include the use of nickel tubes for sampling to check on the effects of deposition on the wall or of contamination.

3.2 ON-LINE INSTRUMENTATION

3.2.1 Gas Chromatograph on Cover Gas

A gas chromatograph is installed to continuously sample and analyze the cover gas for hydrogen and nitrogen from surge tanks in the hot and cold legs of Loop 8. The efficiency of a cold trap for removing hydrogen from a

TABLE 1

TOTAL ALKALINITY IN LOOP SODIUM FLOW-THROUGH SAMPLES

<u>Sample No.</u>	<u>Date Taken</u>	<u>Plugging Indicator Temperature (° F)*</u>	<u>Total Alkalinity, ppm**</u>
4R-2-1A	1-8-69	230	500,160***
4R-2-2A	1-24-69	225	12
4R-2-3A	2-7-69	undiscernible	12
4R-2-4A	3-6-69	undiscernible	73***
4R-2-5A	3-28-69		7
5R-4-7A	11-19-68	286	15, 14
5R-4-8A	12-3-68	280	18
5R-4-9A	12-17-68	282	9
5R-4-10A	1-3-69	250	15
5R-4-11A	1-16-69	275	11
5R-4-12A	2-1-69	undiscernible	11
5R-4-13A	2-18-69	300	13
6R-3-1	1-29-69	260	13
6R-3-2	4-3-69		15
8-4-13A	11-22-68	282	10
8-5-1A	3-12-69	275	15
8-5-2A	3-20-69	300	7
9-2-13C	1-9-69	240	7
9-2-14C	1-23-69	240	14
9-2-14D	1-23-69	240	88***
9-2-15A	2-7-69	not taken	104****
9-2-16A	4-7-69	255	14
9-2-16B	4-8-69	255	7

*Temperature of operation of cold trap varied between 270° F and 320° F.

**Determined by flame photometry; calculated on the basis of Na₂O.

***Sampling tubes had voids.

****Sample was taken during high temperature of the cold trap (>800° F) resulting from loss of cooling when a blower motor failed.

TABLE 2
FLOW THROUGH SAMPLES -
TOTAL HYDROGEN

Sample No.	Total Hydrogen (ppm)
4R-2-3A	1.4
5R-4-1A	1.1
5R-4-5A	3.0
5R-4-8A	2.0
5R-4-12A	0.9
6R-2-2A	0.8
8-4-8A	3.4
8-4-10A	1.1
8-4-13A	1.0
8-4-14A	1.2
9-2-10C	1.0
9-2-10D	3.2
9-2-11C	3.7
9-2-13C	1.2
9-2-14C	0.9

TABLE 3
DETERMINATION OF IRON
IN LOOP SODIUM

Sample No.	Position	Iron* (ppm)
9-2-2A	1	10.5
	2	10.0
	3	7.6
	4	3.5
9-2-11C	1	2.6
	2	3.5
	3	3.4
	4	3.4
5R 4-3A	1	2.1
	2	11.2
	3	8.2
	4	2.8
5R 4-4A	1	8.0
	2	5.5
	3	10.2

*Precision Is On The Order of $\pm 5\%$ to 10%

sodium system was studied in Loop 8 during Run 8-5. High concentrations of hydrogen were produced and monitored in the surge tanks of Loop 8 on two occasions. In the first case > 6000 ppm and > 3000 ppm (by volume) of hydrogen were reached in the cover gas in hot and cold leg surge tanks respectively. This resulted from rise in temperature of the cold trap to 800°F during a brief loss of cooling air. In the second case, a much lower level of hydrogen, 600 ppm (by volume), was reached in the cover gas in the surge tank in the cold leg when flow through the cold trap was reduced from 0.6 to 0.01 gpm.

Figure 3 shows the change in concentration of hydrogen in both surge tanks of Loop 8 when cooling of the cold trap was resumed in the first case. The reduction in total hydrogen with time for both surge tanks was calculated from these data and is shown in Figure 4. The plot indicates efficiency of the trap for removal of hydrogen from the system assuming the equilibria in the gas phase are not rate limiting.

In the second case, where 600 ppm of hydrogen was reached in the cold leg surge tank, the efficiency of the cold trap was shown to be about the same as determined previously. This is shown by the similarity in slope of the plots of removal of hydrogen in Figures 4 and 5.

3.2.2 On-Line Distillation

The on-line distillation unit was used during Run 8-5 to determine oxygen in sodium in a series of 10 runs. Fifty-gram samples were distilled for each run without interrupting loop operation. The sodium temperature was 1000°F for the first 9 runs and 900°F for the last one. The results obtained are shown in Table 4.

Each large sample (50 grams) permits a detection and determination of carbonate by titrimetry. Relatively high concentrations of

FIGURE 3. DECREASE IN HYDROGEN CONCENTRATION IN GAS PHASE DURING COLD TRAPPING (GAS CHROMATOGRAPHY)

FIGURE 4. DECREASE OF TOTAL HYDROGEN IN GAS PHASE AS A RESULT OF COLD TRAPPING

FIGURE 5. REDUCTION IN HYDROGEN IN COLD LEG SURGE TANK AFTER RESUMPTION OF NORMAL FLOW IN COLD TRAP

TABLE 4

DETERMINATION OF OXYGEN AND CARBONATE
BY ON-LINE DISTILLATION AND TITRIMETRY

<u>Date of Run</u>	<u>Cold Trap Temperature (° F)</u>	<u>Oxygen* (ppm)</u>	<u>Carbonate (ppm)</u>
2/19/69	350	230**	--
2/20/69	305	50**	--
3/10/69	300	14**	--
3/15/69	300	5	10
3/20/69	310	8	40
3/25/69	325	4	10
4/2/69	325	7	40
4/4/69	300	6	15
4/29/69	290	4	25
5/6/69	290	8	<5

* Calculation assumes that Na_2O is the only alkaline species present other than Na_2CO_3 .

** High results are due to clean-up of the distillation cups and system.

carbonate were found indicating that values of total alkalinity obtained previously should be reduced by approximately a factor of two to reflect levels of oxygen present as Na_2O .

3.2.3 Oxygen Meter

The first electrochemical cell for oxygen was installed in Loop 8 and operated satisfactorily with some drift and with proper qualitative response to temperature changes of the cold trap. When the temperature of the cold trap rose to $\sim 800^\circ\text{F}$, as described under subsection 3.2.1, flow blockage occurred in areas at lower temperature. The meter circuit (operated at 600°F) plugged at a screen position below the electrode. During attempts to establish flow, the electrode was separated from its housing, removed, and a new assembly installed. Screen openings were enlarged to prevent reoccurrence of plugging in this location.

The second sensor was successfully inserted and has been operating for about 1 month during Run 8-5. During this period some of the common operational characteristics were determined. The variation of sensor response with changes in sodium temperature is shown in Figure 6. The value obtained was $+0.26\text{ MV}/^\circ\text{F}$ which compares favorably to a theoretical value of $+0.21\text{ MV}/^\circ\text{F}$. The temperature of the sodium around the sensor is controlled to $\pm 2^\circ\text{F}$ by equipment independent of main loop controls. The effect of changes in ambient temperature around the housing is shown in Figure 7. The change is equivalent to $-30\text{ MV}/^\circ\text{F}$. The housing area is temperature controlled to $\pm 1^\circ\text{F}$.

Figure 8 also shows a plot of the theoretical relationship between oxygen concentration and emf based on the solubility curve proposed by Eichleberger. ⁽¹⁾ The reading of the oxygen meter shows a higher activity for

FIGURE 6. OXYGEN METER RESPONSE TO SODIUM TEMPERATURE CHANGES

FIGURE 7. OXYGEN METER RESPONSE TO AMBIENT TEMPERATURE CHANGES

FIGURE 8. OXYGEN METER CALIBRATION CURVE

oxygen than the theoretical calculation which is based on the assumption that Henry's Law is obeyed by oxygen in sodium. The discrepancy could be caused by a positive deviation of oxygen activity from Henry's Law or the presence of solute components such as hydrogen, carbon, and transition metals affecting solubility and making existing data of solubility invalid for a multi-component system. From thermodynamic considerations it should be expected that oxygen activity is lowered by the presence of chromium, hydrogen, etc. The data, however, are in the opposite direction. A third alternative could be that the oxygen meter is not measuring activity of oxygen in the system. Experiments are being planned to resolve the current ambiguities.

3.3 ANALYTICAL DEVELOPMENT

3.3.1 Reaction of Sodium, Chromium and Oxygen

An attempt to synthesize sodium chromite at 1000° F by direct reaction of excess sodium metal with sodium oxide in the presence of sodium was not successful. A reac-

tion did occur but the products could not be identified; nickel compounds, from reaction of the sodium oxide and cup, are suspected.

3.3.2 Analysis of Distillation Residue

The reaction of residues with a general reagent to form volatile products which could be identified by gas chromatography has been studied and identification has been made of impurities remaining after distillation. The reagent studied has been trimethyl-chlorosilane and it has been found to react with several common sodium impurities, e.g., sodium hydroxide, sodium carbonate, and sodium oxide, to yield various volatile silyl products. Although the technique looks promising, the amount of development required to refine it to a practical tool appears excessive and the study will be postponed for the present.

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SECTION IV

TASK IV - MECHANICAL PROPERTIES FOR
CLADDING MATERIALS AND LOCALIZED ATTACK

4.1 TESTING OF LMFBR CLADDING

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, and stress-fatigue data to compare the effect of cyclic and noncyclic stress conditions. 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,⁽¹⁾ where higher residual ductilities were noted for the CA structure after 3×10^{21} nvt ($E > 1$ MeV).

4.1.1 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 4R-2 conditions except for brief shutdowns as a result of specimen failures. Up to May 26, the loop operated for ~3600 hours at 1300°F with the pressure applied to the samples. The results to date are shown in Table 5.

4.1.2 Loop 5R - Stress Rupture

Run 5R-4: 1300°F (T_{max}), 425°F (ΔT), < 300°F (T_s)

During the report period, Run 5R-4 was terminated after 4178 hours at 1300°F.

Eleven data points were obtained from the original 16 samples. The results are shown in Table 6.

TABLE 5

1300°F BIAXIAL STRESS-RUPTURE

Material	<u>DATA FROM RUN 4R-2</u>	
	Initial Hoop Stress (psi)	Time-to- Rupture (hours)
304 SS - CA	7000	2188
304 SS - MA	7000	2198
304 SS - MA	7000	2237
304 SS - CA	7000	2263

MA - mill annealed

CA - carbide agglomerated, 1650°F for 24 hours

TABLE 6

1300°F BIAXIAL STRESS-RUPTURE

Material	<u>DATA FROM RUN 5R-4</u>	
	Initial Hoop Stress (psi)	Time-to- Rupture (hours)
304 SS - CA	9,000	1184
304 SS - MA	9,000	1189
304 SS - CA	9,000	1486
316 SS - CA	12,000	1497
316 SS - CA	12,000	1642
304 SS - MA	9,000	1656
304 SS - MA	7,000	2409
304 SS - CA	7,000	2409
304 SS - MA	7,000	2644
304 SS - CA	7,000	3342
316 SS - CA	9,000	3526
316 SS - MA	9,000	4178*
316 SS - CA	9,000	4178*

*Not failed

The results from Runs 4R-2 and 5R-4 show that within experimental error, the initial microstructural condition of Type-304 SS does not appear to have an effect on the stress-rupture properties.

The average results are shown plotted in Figure 9, where they are compared with 1300°F stress-rupture data ^(2,3) in other environments. There appears to be a decrease in long-term stress-rupture properties due to the exposure in nonisothermal flowing sodium. However, it should be noted that the comparison with referenced data may not be directly applicable since the materials are from different heats. Experiments now in progress, testing identical specimens (heats, geometries, etc.) in argon, are expected to provide the desired data for comparison purposes between sodium and nonsodium exposed stress-rupture properties.

The next run in Loop 5R, 5R-5 will be a 5000 to 6000-hour stress-rupture test at 1300°F.

4.1.3 Loop 6R - Stress Fatigue

Run 6R-3: 1300°F (T_{max}), 425°F (ΔT), <300°F (T_s)

During the report period, Run 6R-3 was terminated after ~1700 hours at 1300°F. The results are shown in Table 7.

The results from Run 6R-3, combined with appropriate results from Runs 6R-1 and 6R-2, are shown in Figure 10. The mill annealed material appears to be slightly better than the carbide agglomerated for both Types-304 and 316 SS.

In Figure 11, the stress-rupture and stress-fatigue data are compared. The stress-fatigue data have been plotted as time at the maximum stress level, or one-half the total rupture life.

TABLE 7
1300°F BIAXIAL STRESS-FATIGUE
DATA FROM RUN 6R-3

Material	Initial Hoop Stress (max; psi*)	Time-to- Rupture (hours)
304 SS - CA	15,000	130
304 SS - CA	15,000	185
304 SS - MA	15,000	201
304 SS - MA	15,000	203
316 SS - CA	18,000	354
316 SS - CA	18,000	400
316 SS - MA	18,000	417
316 SS - MA	18,000	499
304 SS - CA	12,000	506
304 SS - CA	12,000	542
304 SS - MA	12,000	582
304 SS - MA	12,000	743
316 SS - CA	15,000	1059
316 SS - MA	15,000	1071
316 SS - MA	15,000	1090
316 SS - CA	15,000	1154

*Minimum hoop stress of 7000 psi for all samples

The slight difference in structural response noted for Type-304 SS in Figure 10 has been ignored for convenience of plotting. Based on these preliminary data, it appears that stress-fatigue retards the long-term rupture of Types-304 and 316 SS in 1300°F flowing sodium. In Figure 12, the average strain rates for stress-rupture and stress-fatigue are compared. There also appears to be a retardation of long-term creep due to stress-fatigue at 1300°F. A definite statement cannot be made due to the lack of similar exposure times to sodium and the previously noted apparent effect of long-term sodium exposure on stress-rupture properties. However, the retardation of creep and rupture has been reported ⁽⁴⁾ for Type-321 SS at 1350°F. As observed previously, ⁽⁵⁾ the stress-fatigue results based on mean stress compare favorably with the constant stress results from Loops 4R and 5R (Figure 13).

FIGURE 9. BIAxIAL STRESS - RUPTURE PROPERTIES OF TYPES 304 AND 316 STAINLESS STEEL IN 1300° F SODIUM

FIGURE 10. BIAxIAL STRESS - FATIGUE PROPERTIES OF TYPES 304 AND 316 STAINLESS STEEL IN 1300°F SODIUM

FIGURE 11. COMPARISON OF BIAxIAL STRESS - RUPTURE AND STRESS - FATIGUE PROPERTIES FOR TYPES 304 AND 316 STAINLESS STEEL IN 1300°F SODIUM

FIGURE 12. EFFECT OF STRESS AND STRESS APPLICATION ON THE AVERAGE STRAIN RATE OF TYPES 304 AND 316 STAINLESS STEEL IN 1300°F SODIUM

FIGURE 13. COMPARISON OF BIAxIAL STRESS - RUPTURE AND STRESS FATIGUE PROPERTIES FOR TYPES 304 AND 316 STAINLESS STEEL IN 1300°F SODIUM

The next run in Loop 6R, 6R-4, will be a 10,000 hour stress-rupture test at 1150°F.

4.1.4 General Corrosion

Types-304, 316, 347 SS, and Incoloy-800 in the mill annealed condition have been isothermally exposed to 1300°F flowing sodium at 10 fps during the mechanical property tests in Loops 4R, 5R, and 6R. Corrosion data have been obtained at times from 538 to 4178 hours. The average corrosion results of the sampling area furthest upstream are shown in Figure 14. Each plotted point represents an average of two to twelve data points. The spread in data ranges from 9% for Type-347 SS to 13% for Incoloy-800 at the longest time point.

The previously reported ⁽⁶⁾ differences in alloy response, in a well controlled sodium environment, and the essentially linear rates have been maintained through 4178 hours. Types-304 and 316 SS continue to exhibit the lowest corrosion rates of the alloys tested. This strengthens the choice of these alloys for the preliminary LMFBR core design.

In addition to the highest corrosion rate in 1300°F flowing sodium, Incoloy-800 also exhibits a progressing intergranular attack, which has reached 2 mils in 3000 hours. ⁽⁶⁾ The attack is a result of the high selective removal rates of Cr and Ni at 1300°F. These data, however, do not rule out the use of Incoloy-800 for lower temperature applications where the corrosion rates will be lower.

All subsequent runs in Loops 4R, 5R, and 6R, including Run 4R-2 presently in progress, will contain mill annealed Type-321 SS, carbide agglomerated Types-304 and 316 SS, and cold reduced Type-316 SS corrosion samples in addition to the four mill annealed alloys.

The cold reduced Type-316 SS has been included in the mass transfer (corrosion) phase of the subtask because of its favorable performance as a fuel cladding reported by the British. Low swelling rates were detected for cold worked Type-316 SS after reactor exposures, but it has also exhibited lower stress-rupture properties as compared to annealed materials. The cold worked grades of stainless steel are now being strongly considered for use in EBR-II fuel element experiments. It is important to establish its structural response to flowing sodium and compare it to that found for the annealed materials.

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FIGURE 14. CORROSION RATES OF MILL ANNEALED STAINLESS STEELS IN 1300°F SODIUM

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