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AEC RESEARCH AND
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APRIL 1970

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**SODIUM TECHNOLOGY DEVELOPMENT PROGRAM
MASS TRANSFER INVESTIGATION
IN LIQUID METAL SYSTEMS
QUARTERLY PROGRESS REPORT NO. 12
DECEMBER 1969 – FEBRUARY 1970**

U. S. ATOMIC ENERGY COMMISSION
CONTRACT NO. AT(04-3)-189
PROJECT AGREEMENT 15

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GEAP 13539 12
AEC Research and
Development Report
April 1970

SODIUM TECHNOLOGY DEVELOPMENT PROGRAM
MASS TRANSFER INVESTIGATIONS IN LIQUID METAL SYSTEMS

Quarterly Progress Report No. 12
December 1969 - February 1970

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1. INTRODUCTION AND PROGRAM SUMMARY

1.1 GENERAL

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. 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 2¼Cr-1Mo and 5Cr-½Mo-½Ti 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 (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.

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.

1.2 SUMMARY OF TASKS

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

Task I: Corrosion 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 Possible 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

This work is directed toward the following:

- Improved quantitative measurements needed for design of the corrosion rates of structural materials steel, including Types 304 and 316 stainless, and of deposition rates occurring in pumped sodium systems at top sodium temperatures to 1150°F and 1300°F and at a ΔT of 400°F to 500°F, similar to large reactor systems.

- Establishment of relationships between sodium impurities, loop operating conditions, and metallurgical effects which change the design-usable properties of the main materials used in Sodium Systems.
- Development of an understanding of the mechanisms involved in the phenomena of corrosion, mass-transport, and deposition in small test loops so that extrapolations can be made to larger systems.
- Monitoring of the test systems for localized attack.

- Using heaters employing heat flux of 0.5 to 1.0 X 10⁶ Btu/h-ft² peak in sodium to measure the effects of reactor heat flux on materials.

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 OPERATION OF TEST FACILITIES

A summary of loop operations including conditions of tests and operating times is shown in Table 1 for the 3-month period ending February 20, 1970.

2.3 LOOP 8 PARAMETER STUDY

The test run (8-6) in preparation for startup is designed to extend the data to higher oxygen levels ($\approx$ 30 ppm) and for study of the effect on formation of particulates and subsequent transport of carbon species from hot-to cold-leg areas.

The following activities were carried out in the current report period:

- Completed Run 8-6 sample loading.
- Completed installation of two cold traps. One had been removed so that a large inventory of Na₂O could be added to it in order to provide adequate oxygen source. The other is a new cold trap, equipped with electromagnetic stirrer, for studies of effect of increasing Reynolds number on improving oxygen removal.
- Operated loop at 900°F for checkout and calibration of on-line monitoring and control devices (oxygen meter, distillation rig, plugging indicator, chromatograph, and cold trap No. 1).
- A pump cell leak on January 17, 1970, was repaired and calibration procedures resumed.

2.4 LOOP 9 CHEMISTRY CONTROL STUDY

The purpose of this run (9-2a) is to obtain corrosion data at oxygen levels of about 30 ppm and to determine relationships between cold-trap operation and changes in chemistry of the system.

Operation at temperature and flow conditions was maintained except for brief shutdowns due to site power outage and to repair sampler valves in the hot leg.

Activities during the period included:

- Operation of cold trap No. 2 in both stirred and un-stirred modes with several different coolant purity variables to evaluate hydrogen and oxygen removal rates with varying local Reynolds number. Variables included different initial loop hydrogen and oxygen levels, cold trap flow rates, cold trap minimum temperatures, and stirring rates.

Table 1
SUMMARY OF LOOP OPERATIONS
November 14, 1969 to February 20, 1970

	Conditions of Test				
	Temp. (°F)	ΔT (°F)	Flow (GPM)	T _s or CT (°F)*	Time (Hours)
Run 8-6	1300	500	5.7	<470	2,000
3-Month Operation					1,519
Total Loop Age					23,388
Run 9-2A	1300	500	5	425	2,000
3-Month Operation					2,292
Cumulative, Run 9-2A					2,887
Total Loop Age					16,536
Run 1R-2	1300	800	2	<300	-----
3-Month Operation					1,510
Cumulative, Run 1R-2					1,534
Run 1R-2A	1300	600	2	<300	100-200
3-Month Operation					175
Cumulative, Run 1R-2A					173
Total Loop Age					6,123
Run 4R-2	1300	500	8	<300	10,000
3-Month Operation					2,340
Cumulative, Run 4R-2					8,772
1000°F and above					
1300°F and pressure					
applied					8,397
Total Loop Age					10,673
Run 5R-5	1300	500	10	<300	5,000
3-Month Operation					796
Cumulative, Run 4R-5					2,143
1000°F and above					
1300°F and pressure					
applied					2,036
Total Loop Age					17,267
Run 6R-4	1150	440	10	<300	10,000
3-Month Operation					2,350
Cumulative, Run 6R-4					4,823
1000°F and above					
1150°F and pressure					
applied					4,501
Total Loop Age					11,512

* T_s - Plugging Temperature

CT - Temperature of Cold Trap

2. Several galvanic cells for oxygen monitoring in the sodium were utilized during loop operations. These included cells with air and Na_2O reference electrodes. Typical values for cell response were +6 millivolts during a change in cold trap temperature from 300°F to 660°F using an air reference and +36 millivolts for a change of 300°F to 600°F in cold trap temperature using a Na_2O reference electrode. The low response of the air reference cell was found to be poor due to an inferior coating of platinum on the inner surface of the ceramic electrolyte. This has been improved substantially by using a technique which provides an adherent gold plating in recently installed cells.

2.5 LOOP 1R PARTICULATE STUDY

Test Run 1R-2 was ended January 16, 1970 with 1534 hours using a sintered nickel filter of 10-micron pore size. This was replaced with a filter of 5-micron pore size and the loop restarted (Run 1R-2a) with the sodium temperature at the filter location (minimum temperature of the cold leg) adjusted to 700°F versus 500°F for the previous test. The objective was two-fold. First, it was desired to obtain a blank on the filters as a result of short term operation (175 hours) and specimen handling. The second objective was to see if NaOH in the filters (eliminated by running at a higher temperature than the precipitation range of NaOH indicated by the plugging temperature first break of $\sim 500^\circ\text{F}$) was responsible for formation of silicates during distillation.* The filters from these tests are being examined. Corrosion samples exposed at 1300°F for 6100 hours were also removed for examination.

Preparations for Run 1R-3, a high oxygen run (25 to 30 ppm) have also been underway, including:

1. Design of a bypass flow circuit for installation of an oxygen meter.
2. Procurement of materials for new samples and construction of a bypass loop for the oxygen meter.
3. Removal of and cleaning of the cold trap in preparation for inserting excess Na_2O inventory and reinstalling in loop.
4. Receipt of and checkout of electrical equipment which will comprise a system to improve control of the loop cold trap under continuous (24-hour) operation. (Identical systems are being set up for Loops 8 and 9.)

Plans for Run 1R-4 involve significant modifications to the loop to obtain better measurements of thermal degradation. In particular, a new economizer with sensitive temperature measuring equipment will replace existing Economizer II. Relocation of the cleanup filter to a position on the shell outlet of Economizer II will make it

possible to collect particulate material of the type found at higher temperature in the loop. Also, installation of pressure measuring apparatus, comprised of Na/NaK pressure transducers and indicating/recording readout, will be effected before startup of that run. Changes in the pressure drop across the filters as particles are collected can then be detected.

2.6 $\text{H}_2\text{-O}_2$ EQUILIBRIA IN SODIUM

The objective of this study is to determine the effects of hydrogen in sodium on oxygen activity. The overall aim is to obtain information which will enable better interpretation of data obtained from on-line instrumentation and samplers (O_2 meter, H_2 meter, plugging meter, chromatograph, distillation) and better application of these devices as well as cold traps for controlling the levels of these impurities in sodium loops.

During this period, the design and drafting was completed for a stirred isothermal pot facility. Materials were procured and fabrication was begun.

2.7 RATIONAL MODEL SCALE-UP STUDIES

Corrosion of stainless steel in a sodium system is a function of velocity or conditions of flow such as fluid boundary layers or Reynolds numbers that are associated with the geometry of the system. Reactor systems of the LMFBR type will operate with Reynolds numbers up to the order of 100,000. Thus, to obtain scale-up test data or extrapolate from small test loops with Reynolds numbers on the order of 30,000, an experiment test loop has been designed to reproduce boundary layer conditions typical of large systems. The high Reynolds numbers will be obtained by rotating specimen surfaces in a flowing stream of sodium with oxygen activity control by electrochemical cells. Basic design parameters of the loop were given in Quarterly Report No. 10 (GEAP)13539).

The assembly of Loop 3R for this work reached 30% completion this period. Generalized equations for flow distribution in the boundary layer region in pipes and outside of cylinders were modified to fit conditions typical of sodium coolant piping and fuel bundles of a large fast reactor. General equations of flow distribution between cylinders in the test loop (inner cylinder rotating) can then be graphically compared to the reference cases in the reactor.

Computer programs were written for these modified equations which compute velocity distributions and provide a graphical plot of the sodium velocity versus distance from the wall. This technique will be used to select the proper diameters and rotational speed so as to produce a velocity profile in the experiment similar to the reactor case. To measure the effect of the different velocity profiles, the inside of the outer stationary cylinder carries removable samples which will be weighed and studied to evaluate the corrosion effects.

*See Task II - Analysis of Particulates.

2.8 COMPARATIVE CORROSION STUDIES

Analysis of weight change data obtained on ring and tube specimens exposed in loop piping during Run 6R-3 showed higher initial rates similar to those sometimes observed on startup of a new loop. A large amount of new surface area was introduced into the loop in connection with replacement of the main test section headers and tubes after Run 6R-2.*

New corrosion samples including Types 304, 316, 321, and 347 stainless steel and Incoloy-800 were also inserted for a total time of exposure of 1770 hours at 1300° F. Normal post exposure procedures included weighing, x-ray fluorescence analysis of surfaces, and metallography.

The corrosion rates were measured as in previous runs at closely similar temperatures and flow conditions, and at "nominally similar" oxygen levels as measured by plugging temperature and amalgamation method assay. As noted in prior work, each of these "nominal" measurements represents the total of several constituents including sodium oxide. Calibrated oxygen meter or distillation of large samples is needed to distinguish oxygen activity from total oxygen. Addition of both an oxygen meter and distillation equipment is scheduled for Run 6R-4.

The short time mechanical properties data reported previously** are not believed to be measurably affected by the higher initial corrosion rates. The material attrition due to corrosion was 0.3 mil or less which is less than one-half of the range of variation of thickness measurements in the as-received tubing. The initial corrosion rates in 6R-3 ranged from two to six times higher than in 5R-4.

2.9 CORROSION AND DEPOSITION ANALYSIS

Run 8-5

Preliminary analyses of samples from Run 8-5 were completed. The weight change data, x-ray fluorescence (XRF) analysis of the surface composition, and bulk carbon analysis are shown in Figures 1 and 2.

Examination of initial weight change data of Sections E and F (hot leg) showed weight loss as expected due to corrosion, while samples from Sections G and H showed weight gain, although XRF analysis of the surfaces of the specimens indicated selective leaching of Ni and Cr. Bulk carbon analyses of the hot and cold leg samples showed that both the hot and cold leg samples were carburized during this run. The weight change data were then corrected for weight gain due to carbon pickup and net corrosion rates were calculated as shown in Figure 1. As can be seen from Figure 1, the corrosion rates are similar to the previous runs for the same velocity and oxygen concentration of sodium. The relative movement of alloying elements (Cr and Ni) is also similar to that of the previous runs. The

carburization observed did not alter the general trend of movement of alloying elements.

Two specimens of 2¼ Croloy ($\approx 0.1\%$ carbon) were placed in the cold leg which did not show any change in carbon concentration. Pure Armco iron with 0.01% carbon also remained unchanged. The results of carburization of the stainless steels, and no change in the ferritic steels, indicate that activity of carbon was at least an order of magnitude higher in Run 8-5 than in previous runs. The high activity of carbon in sodium was unexpected since monitoring of sodium samples during the run showed normal carbon levels ranging from 27 to 44 ppm.

2.10 STUDY OF COLD TRAP EFFICIENCY FOR REMOVAL OF OXYGEN AND HYDROGEN FROM SODIUM

It has been established that an increase of turbulence or Reynolds number of flowing sodium increases the mass transfer coefficient for removal of Na_2O . An investigation was undertaken in Loop 9 to study the mass transfer of Na_2O and NaH or NaOH in the cold trap. The Reynolds number of the sodium flowing through the cold trap was increased by electromagnetic stirring. The cleanup rate of hydrogen was monitored by a diffusion tube connected to a getter-ion vacuum pump. The current generated at the pump is directly proportional to the concentration of hydrogen in sodium. Oxygen removal rate was monitored by an electrochemical galvanic cell with $\text{Na}/\text{Na}_2\text{O}$ as reference electrode. Hydrogen and oxygen were introduced into the system by increasing the cold trap temperature. A second cold trap, with the stirring mechanism, placed in parallel to the first cold trap was used for the efficiency study. After the introduction of the impurities with the first cold trap, it was valved out of the system, and the second cold trap was brought on line. The temperature and flow through the second cold trap was maintained constant (0.2 gpm) when the removal rate was studied with stirred and unstirred mode. The results are shown in Figures 3 and 4.

Figure 3 shows the decrease of pump current with time both in stirred and unstirred mode of the cold trap. The results show that decrease of current (i.e., decrease of hydrogen concentration in Na) is considerably faster with the cold trap operated in a stirred mode condition. This observation implies that stirring action increases the Reynolds number of sodium flowing through the cold trap, which in turn increases the mass transfer coefficient for removal of hydrogen. The removal rate of oxygen is shown in Figure 4. The galvanic cell measures the activity of oxygen in the sodium. In this case, the rate of increase of the emf (i.e., decrease of oxygen activity) is also higher in the case of the stirred cold trap. This observation implies that stirring has increased the cold trap efficiency for removal of oxygen, and that a normal flow through the cold trap results in an efficiency well below 100%.

* A small sodium leak in a thermocouple well in the test section required replacement of the test section.

** GEAP-10036 - Quarterly Report No. 9, March-May 1969.

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FIGURE 1. XRF AND WEIGHT CHANGE DATA RUN 8-5

FIGURE 2. CARBON ANALYSIS RUN 8-5

FIGURE 3. CHANGE IN HYDROGEN CONTENT OF SODIUM AS A FUNCTION OF OPERATING MODE OF COLD TRAP

FIGURE 4. CHANGE IN OXYGEN ACTIVITY AS A FUNCTION OF OPERATING MODE OF COLD TRAP

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 SEPARATION AND IDENTIFICATION OF PARTICULATES

Characterization of the particulates filtered from Loop 1R, Run 1, at three different times has been completed.

The sampling history is shown in Table 2.

Table 2
SAMPLING HISTORY

Sample Designation	Filter Exposure (Hours)	Run Length to Time of Filter Removal (Hours)	Flow Rate (gal/min)
A	600	750	4
B	1100	2700	2
C	1700	4400	2

All filters were sintered nickel of 5-micron porosity. Sampling was carried out at 500°F.

The analytical steps consisted of removing one filter of the three comprising the unit and separating the residual sodium by distillation.

Specific quantitative analyses were performed for the elements shown in Table 3. Several additional metallic species, e.g., Ti, V, and Mo were detected by emission spectrographic techniques; but since they were present at low concentrations, precise analyses were not performed.

Each sample (single filter) yielded about 1 gram of material. A large fraction of the sample (about two-thirds) was water soluble and consisted primarily of sodium and silicon. It is presumed that a sodium silicate was present in the distillation residue on the basis of the chemistry of the product (decomposes to yield an alkaline solution in water; SiO_2 can be precipitated by evaporation with acids) but there is no evidence that silicon was combined in this way in the sodium. A variety of silicates are possible but all are in the form $x \text{Na}_2\text{O} \cdot y \text{SiO}_2$. It is possible to calculate the oxygen present in the residue from the amounts of sodium and silicon present and this has been done to provide the oxygen concentration shown in Table 3. No silicon was found in the water in soluble portion of Sample A, but appreciable amounts were present in the subsequent filters according to emission spectrographic results. Quantitative analyses for the silicon in these residues is not yet complete

but presumably accounts in part for the low mass balance for these latter samples. The discrepancy may also be due to a difference in the composition of oxygen containing compounds in the later samples. The amount of sodium oxide present is in excess of that required for the common silicates and probably arises from contamination of the filter in handling and by the conversion of sodium hydroxide known to precipitate from the loop sodium at these temperatures.

Table 3
ELEMENTAL COMPOSITION OF
DISTILLATION RESIDUE

Element	Concentration % of Residue*		
Filter:	A	B	C
Fe	10.5	3.5	1.3
Cr	7.0	2.0	3.0
Mn	3.3	7.4	7.1
Ni	2.0	2.4	1.5
C	2.0	0.8	0.6
Ca	1.1	2.5	2.2
Na	42.0	36.0	40.0
Si	5.2	8.5	4.4
O**	21.0	23.0	19.0
Total	94.1	86.1	79.1

* Precision $\pm 15\%$ of value reported

** Calculated Value

Of the metallic species, only manganese was found in the water soluble material to any appreciable extent.

Calculation of stainless steel constituents in filterable form, assuming 100% filter efficiency, showed that the filterable material was in the parts per billion (ppb) range, Table 4. It is likely that substantial fractions of particulates were in a size range less than 5 microns and were not collected.

X-ray diffraction patterns were obtained on all residues before exposure to air. In addition, the product remaining after water leaching was also examined. Data is shown in Table 5.

To a large degree, the diffraction patterns within the two sample categories were quite similar. The unidentified phase is undoubtedly due to Na-Si-O compounds. However, the pattern does not match any available in the ASTM compilation. Possibly the occlusion of some of the transition metals, in the silicate, (manganese for example) leads to an unfamiliar pattern.

Table 4

**CONCENTRATION OF FILTERABLE ELEMENTS
COLLECTED FROM LOOP 1R SODIUM***

Element	Concentration (ppb)		
	A	B	C
Fe	0.64	0.24	0.058
Cr	0.45	0.14	0.14
Mn	0.21	0.50	0.32
C	0.13	0.054	0.27
Si	0.36	0.59	0.19
Ni	0.13	0.16	0.067
Total	1.9	1.7	0.8

* Assumes filtering efficiency of 100% for particles greater than 5 microns.

Table 5

**X-RAY DIFFRACTION ANALYSES
OF FILTER RESIDUES**

Sample Designation	Phases-Observed
A (distillation residue)	α Fe; austenite; unidentified; traces of $M_{23}C_6$, M_7C_3
B (distillation residue)	Unidentified; α Fe; austenite; traces of $M_{23}C_6$, M_7C_3
C (distillation residue)	Unidentified, trace carbide
A (water insoluble)	α Fe; austenite; $M_{23}C_6$
B (water insoluble)	α Fe; austenite; $M_{23}C_6$
C (water insoluble)	Unidentified, α Fe, austenite

The attempt to collect particles by magnetic means (~ 2600 gauss) in this run was not successful presumably because the magnetic field was not sufficient to overcome the hydraulic forces carrying the particles in the sodium stream.

In summary, the data obtained thus far in this study indicate the following:

- The particulates removed from Loop 1R by filtration indicate some fraction of the particulates in the sodium is below the 5-micron pore size of the filters.
- The amount of available material decreased with time which is consistent with the expected reduction in corrosion rate after initial loop operation.
- Sodium and silicon oxides comprise a major share of the sample. The former is at least partly due to contamination but some arises from the conversion of sodium hydroxides. The source of the silicon oxide is undetermined but the source of silicon is probably the steel itself.

- The transition elements make up the remainder of the sample. The concentrations of iron and carbon decrease noticeably with time. Alpha iron, austenite, and traces of carbides have been observed.
- The magnetic technique (to 2600 gauss fields) for collection was not powerful enough to collect any of the particulates.

Because of the appearance of silicon in the Loop 1 particulates, several samples of cold trap sodium were examined for this element. (An extensive discussion^{1,2} of the analysis of these traps has been provided previously; no analysis for silicon was performed at the time).

The results indicate that in the ferritic system the silicon concentration was on the order of 400 ppm, while it was about 700 ppm in the austenitic system deposit. Thus, silicon was a major impurity collected in the traps.

A major unidentified diffraction pattern for the residues from the ferritic system cold trap was reported in the previously cited work. Since acetylene was produced on hydrolysis of these residues, the pattern was compared to the one recently obtained for Na_2C_2 . The unknown pattern was not similar to that of Na_2C_2 .

3.2 INTERACTION STUDIES, COMPOUND IDENTIFICATION, ANALYTICAL CHEMISTRY

3.2.1 Capsule Studies of Sodium-Oxygen-Transition Metal Interactions

A technique of x-ray identification of oxidation products formed on wires is being used to facilitate identification of particulates obtained from loop sodium samples. Experiments patterned after those of Addison and Barker are under way. Wires of transition metals, immersed in sodium with a 100 ppm oxygen concentration, and contained in alumina capsules, are being heated at 600°C . At the completion of the tests, direct examination of the wires by x-ray diffraction analysis will be made to determine the nature of any oxidation product formed.

3.2.2 Development of Additional Techniques for the Examination of Particulates

Attempts are in progress to supplement x-ray diffraction analysis of particles filtered from sodium with optical microscopic examinations. It has proven possible to immerse in epoxy, without air exposure, particles from distillation residues. More importantly it has been possible to immerse sodium itself in the epoxy and have it remain

(1) Dutina, D., Simpson, J. L., and Young, R. S., "Determination of Impurities in a Sodium System Cold Trap," Trans. ANS, 10 (1), 145, June 1967.

(2) Dutina, D., Simpson, J. L., and Young, R. S., "Carbon Impurities in Sodium System Cold Traps," ANL-7520, Part 1, November 1968.

shiny for several days. This opens the possibility of simple direct examination of a filter coated with sodium to observe the physical properties of collected material.

Similarly, it is planned to modify the inert atmosphere holder previously used in the program to accept portions of the nickel filter for direct x-ray diffraction analysis. Such direct examinations are needed to eliminate concern about interaction of species during distillation processes.

3.2.3 Accumulation of X-Ray Diffraction Data

a. Sodium Chromite

A communication from M. G. Barker, University of Nottingham, UK, supplied x-ray data for NaCrO_2 which closely matches that reported in a paper¹ presented at the AIME Liquid Metals Corrosion Symposium, Philadelphia, Pennsylvania, October 12 through 16, 1969. The data is shown in Table 6.

Table 6
LATTICE PARAMETER FOR NaCrO_2

	a_0	a_0
	(A)	(A)
Barker Data	2.96	15.94
This Laboratory	2.97 ± 0.01	15.96 ± 0.01

	a_0	a_0
	(A)	(A)
Barker Data	2.96	15.94
This Laboratory	2.97 ± 0.01	15.96 ± 0.01

b. Disodium Acetylide

A sample of disodium acetylide (Na_2C_2) was obtained from J. McCowan of FFTF. X-ray diffraction patterns were cataloged. They matched those calculated from H. Foppl² as supplied by R. V. Schablaske of ANL. The acetylide pattern is not found in the ASTM files.

- (1) "The Relationship of Particulates to Mass Transport Phenomena in Flowing Sodium in Austenitic Systems," Prodyot Roy, Dragomir Dutina, Frank Comprelli.
- (2) H. Foppl, Angew. Chem. 70, 401 (1958).

3.3 SAMPLING OF LOOPS AND DETERMINATION OF IMPURITY LEVELS

3.3.1 Total Alkalinity Measurements

Table 7 provides recent data on total alkalinity measurements (amalgamation determination) for loop samples. The values are fairly typical for the loops. Occasional high values are accounted for by cold trap excursions.

3.3.2 Determination of Chloride

The determination of chloride in loop sodium is shown in Table 8. In this range, the precision of the data is about $\pm$ a factor of two. The results are lower by a factor of five to ten than those reported for a single sample reported previously. The earlier result is believed to be in error. A filtration step was incorporated in the procedure (prior to the addition of the silver precipitant) in the present work. The filter showed appreciable darkening. Such suspended particulates would lead to a positive error in the earlier data for chloride.

3.3.3 Determination of Carbon

Analysis for carbon has been completed on several loop samples. The data is shown in Table 9.

With one exception, the results are in the usual range of 40 ± 20 ppm, which are typical for these systems.

3.3.4 Determination of Hydrogen, Table 10

3.4 ANALYSIS OF RADIOACTIVE SODIUM

Both EBR-II and FFTF were visited to survey analytical techniques being used and being planned for the analysis of radioactive sodium. The observations will help in the formulation of specifications for equipment and methods for handling and analyzing radioactive sodium samples.

An evaluation of available data on the composition of radioactive species in sodium has been initiated.

Table 7
TOTAL ALKALINITY IN LOOP
SODIUM-FLOW THROUGH SAMPLES

Sample Number	Date Taken	Plugging Temperature (°F)	Total Alkalinity (ppm)
1R-1-5A	10/15/69	*	14
1R-1-6A	10/28/69	*	17
1R-1-7A	11/11/69	*	23
1R-2-2A	12/4/69	*	12
1R-2-3A	12/16/69	*	13
1R-2-4A	12/20/69	*	15
4R-2-11A	10/21/69		13
4R-2-13A	12/29/69		18
5R-5-2A *	10/9/69	270	30 ⁽¹⁾
5R-5-3A	10/15/69	*	18
5R-5-4A	11/12/69	*	13
6R-4-4A	10/16/69	*	20
6R-5-5A	11/14/69	*	11
8-5-6A	7/2/69	*	11
8-5-7A	7/7/69	*	17
8-5-8A	7/8/69	450	25 ⁽²⁾
9-2A-1	10/22/69	*	19 ⁽³⁾
9-2A-3A	11/1/69	240	13
9-2A-5A	11/11/69		19
9-2A Special ⁽⁴⁾			

* Plugging temperature indicated as low and undiscernible.

(1) Cold trap in line for 24 hours at 278°F prior to sampling. Purification leg was shutdown in week prior for repairs to plugging indicator.

(2) Cold trap operation at 450°F for calibration of oxygen meter.

(3) Cold trap operation at 495°F for hydrogen measurements.

(4) Sampled from tube containing wires for internal friction measurement.

Table 8
DETERMINATION OF CHLORIDE
IN LOOP SODIUM

Sample	Chloride (ppm)
1-1-2A	0.9
4-2-7A	0.7
5-4-4A	0.8
6-3-3A	0.4
8-5-4A	0.4
9-2-19A	0.4

Table 9
DETERMINATION OF CARBON IN
FLOW-THROUGH SAMPLES
(Wet Combustion Technique)

Sample Number	Total Carbon (ppm)
1R-1-1A	87
1R-1-3A	39
1R-1-5A	35
5R-5-3A	33
6R-3-3A	37
8-5-9A	44
9-2-18A	43

Table 10
DETERMINATION
OF HYDROGEN

Sample Number	(ppm)
4R-2-7A	3.5
4R-2-11A	3.5
5R-5-1A	3.7
5R-5-3A	1.0
6R-3-1A	5.1
6R-3-3A	4.5
6R-4-1A	1.2, 1.5

No significant trends are apparent.

4. TASK IV: CORROSION AND MASS TRANSPORT EFFECTS ON COMPOSITION, STRUCTURE, AND STRUCTURAL INTEGRITY OF CANDIDATE ALLOYS FOR LMFBR 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 TESTING OF LMFBR CLADDING

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 8229 hours at 1300°F with the pressure applied, as of February 13. There were two failures during the report period. Twelve of the original sixteen samples have failed. The results to date are shown in Table 11.

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

The loop operated at test conditions until December 17, when a sodium leak occurred in the main heater. Since that time, the loop has been inoperative for the replacement of the heater. The total run time, as of December 17, at 1300°F with the pressure applied, is 2036 hours. There have been no failures of the original sixteen samples.

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

The loop operated at test conditions during the report period. The run reached 4333 hours at 1150°F with the pressure applied as of February 13.

There was one failure during the report period. Seven of the original sixteen samples have failed. The results to date are shown in Table 12.

4.3 STRESS RUPTURE CONTROL TESTS

Stress-rupture tests are being run at 1300° the environmental effect(s) can be delineated. The results to date are shown in Table 13.

The results, plus those for Run 4R-2 and the previously reported data from Loop 5R,* are plotted in Figures 5 and 6. On the basis of comparison with the control tests, there is a definite decrease in long-term stress-rupture properties due to exposure in nonisothermal flowing sodium.

* Quarterly Report No. 9, GEAP-10036

Table 11

**1300°F BIAXIAL STRESS RUPTURE
DATA FROM RUN 4R2**

Material	Initial Hoop Stress (psi)	Time-to-Rupture (Hours)
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

* CA - carbide agglomerated, 1650°F
for 24 hours

** MA - mill annealed

Table 13

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

Material	Initial Hoop Stress (psi)	Time-to-Rupture (Hours)
316MA	15,000	1375
316CA	13,000	1584
316MA	13,000	2415
316MA	11,000	3183
316MA	11,000	4551
316CA	11,000	4874
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	89,000	3373
304CA	9,000	3389
304MA	9,000	4374

Table 12

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

Material	Initial Hoop Stress (psi)	Time-to-Rupture (Hours)
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

FIGURE 5. 1300°F BIAxIAL STRESS-RUPTURE PROPERTIES OF TYPE 316 SS IN SODIUM AND ARGON

FIGURE 6. 1300°F BIAXIAL STRESS-RUPTURE PROPERTIES OF TYPE 304 SS IN SODIUM AND ARGON

5. LIST OF SODIUM MASS TRANSFER PROJECT REPORTS

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