SODIUM COMPONENTS DEVELOPMENT PROGRAM
MASS TRANSFER INVESTIGATIONS IN LIQUID METAL SYSTEMS

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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

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 INTERACTIONS 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

Corrosion rates were found to be weakly dependent on mass flow for the conditions of 1300°F, approximately 2,000 hours, and 3 ft/sec velocity. Mass flow was varied by a factor of two in a specially designed corrosion cartridge in which two values of mass flow were obtained at the same velocity. Saturation of iron in the sodium was established by negligible weight losses of ARMCO iron samples.

The effect of heat-flux on corrosion was further investigated. An electron-bombardment (EB) heater was operated continuously in loop 9 for 1250 hours at about 172 watts/cm² (~500,000 Btu/h-ft²) heat flux with 1300°F bulk sodium outlet.

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

Hydrogen compounds have been found to be the main cause of low temperature breaks in the plugging curve as well as the high temperature breaks. An unexpected result was the near absence (~5%) of sodium oxide. This work confirms that a plugging temperature is not uniquely related to the oxygen concentration in a sodium system. (The absence of a high temperature break and the presence of a single low temperature break near the cold trap temperature provided opportunity to collect and identify particulates causing plugging of the orifices at low temperature.)

The cyanide concentration was investigated in the hot and cold-legs of loop 9 to determine the degree of saturation. Findings were less than 1 ppm of cyanide in samples removed at temperatures from 755°F to 1230°F (solubility of CN is 100 ppm at 1100°F).

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

Stress-rupture shows that initial microstructural condition of Type-304 SS does not have significant effect on stress-rupture properties for the range of structures tested. (Test conditions were 1300°F for 3600 hours, with rupture data from 14 out of 16 specimens.)

Type-316 SS showed a strong effect due to initial structure. After a carbide agglomeration heat treatment, a 100% increase in time-to-rupture was found compared to mill annealed material (both results from loop 5R).

A 10,000-hour stress-rupture run was started in loop 4R at 1300°F.

1.4 REPORTS

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

In addition to the progress letters, 28 topical reports and 7 Quarterly Progress Reports covering test results have been issued (see Section V).

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

SECTION II

TASK I - LMFBR / FFTF CORROSION PARAMETER STUDIES

This work is directed toward:

- a. Experimentally studying the corrosion of steel exposed to flowing sodium at bulk temperatures to 1300°F.
- b. Developing and using high heat flux heaters for operation at 0.5 to 1.0×10^6 Btu/h-ft² heat flux in sodium to measure the effects of reactor heat flux on candidate materials.
- c. Developing an understanding of the mechanisms involved in corrosion-mass transport and deposition phenomena so that extrapolations can be made to large-scale systems.
- d. Monitoring for localized attack.
- e. Establishing relationships between sodium impurities, loop operations and metallurgical effects.

The work is carried out primarily in loops 8 and 9 constructed to simulate corrosion conditions of a 1000 MWe coolant system.

2.1 CORROSION-DEPOSITION STUDIES

2.1.1 Run 8-4

Run 8-4 was completed during this period. The total run time was 3356 hours. The maximum operating temperature was 1300°F with 500°F (ΔT). The plugging temperature was maintained at < 300°F. The weight losses in the hot leg and weight gain in the coolers and cold leg, at various velocities and temperatures are given in the Appendix.

The corrosion results are in good agreement for comparable conditions with previous runs.

A special corrosion cartridge was inserted for 2338 hours in Run 8-4 to study the effect of mass flow. Figure 1 shows the flow sections of the cartridge and variations in mass flow.

FIGURE 1. CORROSION CARTRIDGE WITH VARIATIONS IN MASS FLOW

The corrosion specimens between channels A and B saw 0.5 gpm flow, whereas the specimens between C and D saw 1.0 gpm flow. The velocities for both specimens were 3 fps. The result as shown in Figure 2 shows that mass flow has little effect on corrosion rate of stainless steels. In other words, if the mass flow (gpm) is changed without changing the Reynolds number, or the velocities, the corrosion rate will be changed little at least for velocities near 3 fps. In the present run the Reynolds numbers for both specimens were approximately the same (8110 for specimen between A and B and 7670 for specimen between C and D). Metallographic analysis, x-ray fluorescence, chemical analysis for all the samples is in progress.

2.1.2 Run 8-5

The Run 8-5 has started during this period. The run will be carried out at $T_{\max} = 1300^{\circ}\text{F}$ with 500°F (ΔT). The purpose of this run is to provide "downstream" conditions at uniform velocity which are similar to those in a reactor core. The run plan also calls for evaluation of performance of an oxygen meter at a hot leg temperature of 1300°F . This will permit a comparison of the effect of hot leg temperature on the measured activity of oxygen.

2.2 RATIONAL MODEL DEVELOPMENT

In order to understand mechanism of corrosion and deposition, several ARMCO iron samples were placed in the hot and cold leg of Run 8-4. The hot leg samples did not show any significant corrosion; e.g., one ARMCO iron sample lost 6.5 mg/dm^2 whereas the 316 SS samples adjacent to ARMCO iron lost 484 mg/dm^2 . The data indicated that sodium in the loop becomes saturated with respect to iron and that α -iron is not efficiently precipitated in the cold leg. Another reason for negligible weight change for iron specimens in the hot leg could be due to selective deposition of Cr and/or Ni on iron samples even in the hot leg. This will be verified by XRF* analysis of the iron samples. A few ARMCO iron samples were also placed in the cold leg of this run. XRF analyses are now being made to determine the effect, if any, of the composition of substrate on selective deposition of corrosion products.

2.3 DEVELOPMENT AND TESTING OF HIGH HEAT FLUX HEATERS

The objective of this task is the development of high heat flux heaters capable of operating for times to 3000 hours in sodium at temperatures to 1300°F to obtain the effects of increased temperature at metal surfaces caused by heat flux typical of fast reactor fuel, namely in the range of 150 to 300 watts/cm^2 ($0.5\text{--}1.0 \times 10^6 \text{ Btu/h-ft}^2$).

During the quarter the electron-bombardment (EB) heater was operated in loop 9 continuously for 1250 hours at about 172 watts/cm^2 at 1300°F bulk sodium outlet temperature. Before the start of this run the anode was changed twice with 3 day turn around times. The first change was to remove pieces of spalled cathode which caused a short between the anode and cathode. The second change was to replace a broken cathode-anode insulator which, it appeared, had cracked during the first operating period.

*X-ray Fluorescence

FIGURE 2. EFFECT OF MASS FLOW ON CORROSION RESULTS

2.3.1 Heater Operation

Operation at 172 watts/cm² heat flux was very stable over the 1250 hours in this period. A typical pattern of temperatures indicated by thermocouples in the sodium stream along the heater is shown in Figure 3. Operation was interrupted on December 1 by a short due to cathode spalling.

The cavity in the cathode was chamfered, and a new anode tube was prepared, assembled, welded, and baked out. Continued operation of this heater was characterized by uniformity of heat flux. The readings of the same thermocouples as shown in Figure 3 are also shown in Figure 4. The anode-cathode stand-off voltage was greatly improved, operation was stable at 525 volts.

On December 24 the heater again shorted, cathode to anode. A new split ring insulator was installed as a replacement and a new anode, the third for this cathode assembly, was installed. Anode voltage was reduced to 360 volts, the electron current was increased and operation has been stable throughout the remainder of the quarter. The last anode has accumulated 1250 hours.

As shown in Figure 5, a test section is being installed in loop 1R in conjunction with the particulate collection experiment being readied in Task II. A new power supply for operation of this second heater is being checked out and tested.

The operating experience in this quarter shows the capability of the heater to meet objectives but also shows need for extremely good quality control to establish the uniformity of electron emission.

2.3.2 Response of EB Heater to Hydrogen

It has been observed that the pressure in the EB heater rises in response to two effects. The smallest effect is noted upon increase of the beam current. The pressure changes by a factor of approximately two over the full usable beam current range. The more important effect is a pressure rise due to diffusion of hydrogen from the sodium through the stainless steel anode tube. This has occurred both in response to an increase in cold trap temperature, and again when small amounts of water were injected into the cover gas of a loop, as reported in GEAP-5582 (Quarterly No. 3, September to November 1967).

During this quarter, operating data were processed and plotted in Figures 6 and 7. Figure 6 is an initial calibration of the heater as an indicator of the steady state content of hydrogen in the sodium at a constant sodium temperature of 1300°F at the heater and at several cold trap temperatures. Figure 7 is a partial record of the transient response to a change in cold trap cooling rate. After a long steady period of 300°F cold trap operation, the cold trap temperature was raised to 350°F for the purpose of a test of the oscillating plugging indicator. The cold trap reference temperature rose to 350°F in about 20 minutes. The response of the EB heater pressure was noted for 4 hours.

Since the heater pressure rise in Figure 7 was slower than the response time of which the heater is capable, the rate of pressure rise was apparently an indication of the rate of introduction

of hydrogen bearing impurities from the cold trap into the loop. The EB heater is potentially a continuous monitor of hydrogenous impurities and may be useful in studying basic cold trapping operation. Furthermore, the EB heater seems to be a prompt indicator of hydrogen introduction such as by a leak from a steam generator or the atmosphere seeping into a sodium system. It is planned to continue these observations in the next quarter.

E-EB-4 1

FIGURE 3. TEMPERATURE RISE OF SODIUM ALONG HIGH HEAT FLUX HEATER

E-EB-4-3

FIGURE 4. TEMPERATURE PATTERN SHOWING MORE UNIFORM EMISSION

FIGURE 5. ELECTRON BOMBARDMENT HEATER TEST SECTION IN LOOP 1R
SHOWING THERMOCOUPLE PENETRATIONS

FIGURE 6. EB HEATER PRESSURE: RESPONSE TO COLD TRAP TEMPERATURE, LOOP 9

FIGURE 7. EB HEATER PRESSURE: FOLLOWING RISE IN COLD TRAP TEMPERATURE

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 AND CONTROL

3.1.1 Characterization of Materials Depositing in Plugging Indicators at Low Temperatures

The Solids Collector⁽¹⁾ was designed to accumulate sufficient quantities of the species precipitating in plugging indicators (P.I.) to permit their identification. Initial work recently reported⁽²⁾ showed that sodium hydroxide and sodium hydride were the compounds depositing in P.I.'s at temperatures considerably in excess of the cold trap temperature. In the final 2000 hours of Run 8-4 these high temperature breaks (about 400°F) disappeared, presumably due to an increased efficiency of the cold trap for the removal of the hydrogenous compounds. This presented the opportunity to collect the impurity causing flow stoppage at low temperatures, i.e., temperatures more nearly corresponding to that of the cold trap. It had been assumed that this impurity was Na₂O.

The Solids Collector was installed in the loop cold leg and operated initially as a plugging indicator. With the cold trap temperature maintained at 325°F, the Solids Collector plugged in the 285 to 300°F region when operated in the oscillating mode, and 265 to 275°F when operated as a bare orifice. The failure of the plugging temperature (oscillating) to correspond to the cold trap temperature is unexplained at this time. Finally the collector was allowed to plug in the bare mode (265 to 275°F), cooled to room temperature, disconnected from the loop, and dismantled in a glove box.

Analyses of deposits from two of the channels of the device showed sodium hydride to be present in the concentration range of 5 to 10%. (The bulk of the material in the channel is sodium metal.) Sodium hydroxide was present on one of the samples at barely detectable levels, about 5%. Of significance is the absence of detectable (<5%) amounts of sodium oxide. The data thus indicates that it is the hydrogen compounds which are causing the low temperature flow breaks as well as the high temperature ones. Although unexpected, this result is not altogether surprising. Once precipitation of the hydrogen compound begins, it should continue as the temperature decreases. Further it is important to note that in the temperature regions under study, hydrogen is less soluble than oxygen in sodium. Thus the presence of hydrogen in the plug can be explained.

Subject to further investigation, however, is the question of why hydrogen compounds persist in loop sodium at temperatures greater than that of the cold trap. A broader question still, is learning the effect of these hydrogenous species on the oxygen activity in the system.

TABLE 1

FLOW THROUGH SAMPLERS—TOTAL ALKALINITY

<u>Sample No.</u>	<u>Date Taken</u>	<u>Sample Temperature F</u>	<u>Lowest P.I. Temperature F</u>	<u>Cold Trap Temperature F</u>	<u>Total Alkalinity ppm***</u>
5R-4-1A	8-6-68	675	245	280	7
5R-4-3A	9-19-68	845	240	280	9
5R-4-4A	10-3-68	825	256	-**	10
5R-4-5A	10-17-68	820	274	-**	8
5R-4-6A	10-31-68	850	287	-**	10
6R-2-2A	8-1-68	940	-*	260	8
8-4-8A	9-6-68	750	-*	280	7
8-4-9A	8-4-68	755	-*	310	10
8-4-10A	10-10-68	712	269	325	12
8-4-11A	11-4-68	730	265	320	9
8-4-12A	11-8-68	710	284	320	9
9-2-10C	11-16-68	765	265	290	11
9-2-10D	11-16-68	1230	265	290	8
9-2-11C	11-20-68	760	-*	295	10

* No flow break discernible. High temperature flow breaks observed in most samples.

** Cold trap not operated.

*** By amalgamation. Calculated on the basis of Na_2O . No corrections for other alkaline species.

This data re-emphasizes the fact that a plugging indicator is a "something meter" and gives a measure of overall behavior with respect to precipitable impurities, but is not an explicit indicator of the oxygen concentration in the system.

3.1.2 Flow Through Samplers

Analyses for total alkalinity of 14 samples taken from four loops were completed during the reporting period. The results are reported in Table 1 which also includes data in plugging indicator, cold trap and sampling conditions. The data show that for all loops the alkalinity was in the range 10 ± 3 ppm, which is within the combined sampling and analysis error, thus indicating a consistency in the loop purity control.

Analyses for metallic impurities in samples taken in earlier loop runs are shown in Table 2. The results appear to be in the usual range.

TABLE 2

ANALYSES FOR METAL IMPURITIES

<u>Sample No.</u>	<u>Impurity (ppm)</u>				
	Cr	Fe	Mn	Ni	Ca
G-2-1A	1	10	2	< 1	4
4R-1-2A	2	-	3	2	5
5R-4-2A	< 1	12	2	< 1	5
8-4-5A	< 1	8	2	< 1	5
5R-3-4A	2	12	2	1	4

Plugging temperatures were obtained using the bare orifice technique. These generally were 25 to 50 degrees (F) lower than the cold trap temperature.

3.1.3 Oxygen Meter

Installation of the electrochemical cell for detecting oxide activity was completed in loop 8 except for final connection of power supply. Location of the components of the oxygen meter system in respect to loop 8 is shown in Figure 8.

3.2 PARTICULATE STUDIES

3.2.1 Compound Synthesis

Preparation is continuing for the examination of material which is to be collected in the particulate study. One phase of this work is the synthesis of compounds which may be present in these corrosion deposits. The study includes the determination of the chemical behavior of such compounds and the cataloging of x-ray diffraction patterns.

FIGURE 8. OXYGEN METER COMPONENTS DURING INSTALLATION

The first compound under investigation is sodium chromite, NaCrO_2 . It has been prepared by the reaction of Na_2CO_3 with Cr_2O_3 at 1650°F . Its x-ray diffraction pattern contained a hexagonal structured single phase with a lattice parameter $a_0=2.96 \text{ \AA}$, $c_0=15.98 \text{ \AA}$ which checked literature data ⁽³⁾. The product was soluble in concentrated sulfuric acid shortly after preparation. However, on standing, it could not be dissolved in this nor other strong acids. The x-ray pattern did not change with time.

3.2.2 Loop 1R Experiment to Collect Particulate Material

Loop 1R was rebuilt to operate at $1300^\circ\text{F } T_{\text{max}}$ and a 500 to $800^\circ\text{F } \Delta T$ with both micrometallic filters ($5\text{-}10 \mu$ size) and magnetic particle collectors. Figure 9 shows the main features of the test including pipe locations for magnetic collectors and filters. The experiment is scheduled to operate in April.

3.2.3 Loop 8 Cold Trap—Economizer Examination

Neutron radiographs revealed possible plugs (high impurities regions) in both the inlet and outlet lines to the trap. When the lines were opened at these points, hard, brittle deposits were found, confirming the neutrograph information. Analyses of the deposits both by x-ray and chemical techniques showed the material to consist of the species shown in Table 3.

TABLE 3
COMPOSITION OF COLD TRAP PLUG

<u>Species</u>	<u>Concentration (%)</u>
Na_2O	88
Na	6
NaH	5
NaOH	1

The location of the plug suggests some cold trap design modifications (e.g., increases in the inlet pipe diameter, more heat in this region, etc.) which should avoid recurrence.

The economizer has been sectioned, and the sodium removed by distillation. Analyses of the residual particles are underway.

3.3 ANALYTICAL CHEMISTRY

The study of the behavior of cyanide in sodium and its potential for carburization of steel in this medium has been of interest because of its ease of formation from impurities commonly found in sodium, ⁽⁴⁾ its presence in high concentration in sodium system cold traps, ⁽⁵⁾ and its high solubility in sodium ⁽⁶⁾.

The study of cyanide behavior is not a major part of the present program, however the characterization of system impurities has included the analysis for this anion. It was previously

FIGURE 9. LOOP 1R, PARTICULATE COLLECTION EXPERIMENT

reported⁽¹⁾ that cyanide had been found at a concentration of 10 ppm in a sample from loop 8. Since this value is of the same order of magnitude as the reported solubility at this temperature, speculation was aroused as to whether the system was saturated with respect to this species with consequent precipitation throughout the system. Further study of the question was made possible by the recently added capability of sampling the hot, as well as the cold, leg sodium of loop 9. Several samples from the loop were analyzed with results as shown in Table 4.

TABLE 4
CYANIDE CONCENTRATION IN LOOP 9 SODIUM

<u>Sample No.</u>	<u>Sampling Temperature °F</u>	<u>Cyanide ppm</u>
9-2-6D	1100	0.2
9-2-8D	1125	0.6
9-2-10D	1230	0.1
9-2-8C	755	0.05
9-2-11C	760	<0.05

The results show that cyanide concentrations are generally less than 1 ppm at either hot or cold leg. Hot leg cyanide concentrations are about a factor of 10 higher than those for the cold leg (based on a limited amount of data).

Since the solubility of cyanide is > 100 ppm at 1100°F and about 1 ppm at 750°F , it is clear from the data that the system is not saturated with cyanide. Both nitrogen and carbon are available in the system and the reaction should take place at hot leg temperatures. That it does so only to a relatively small extent may be due to slow kinetics of the process. An alternate explanation may lie in the current speculation concerning the carbon-oxygen-chromium system. In very qualitative terms, at the low oxygen level in the systems, chromium is available to tie up the carbon activity, thus limiting the availability of carbon for cyanide formation. A possible test of this hypothesis could be obtained by determining whether cyanide concentration changes with increased oxygen level.

3.4 PARTICIPATION IN STANDARDS ACTIVITIES

During 1968 PA-15 personnel participated in an RDT-sponsored round robin to test the proposed ASTM amalgamation procedure for the determination of "oxygen" in sodium. Two dozen samples, prepared by APDA, were analyzed. Results were reported to APDA for compilation.

In November, in conjunction with the ANL conference, D. Dutina chaired two meetings of the ASTM group; one of the participants in the round robin, the second of the entire ASTM E-3, Division N sodium task force. The primary subject at both sessions was a discussion of the round robin results. In addition, there was some discussion on future plans related to sampling and to standardization of the distillation technique. The G-1, Sub. VIII, Task Group B (Sodium Corrosion) meeting was attended during the same period.

SECTION IV

TASK IV - MECHANICAL PROPERTIES FOR CLADDING MATERIALS AND
LOCALIZED ATTACK

4.1 TESTING OF LMFBR CLADDING

Longer range testing of stress-rupture properties to 10,000 hours has started with concentration on Types 304 and 316 stainless steel in the annealed and carbide agglomerated conditions.

4.1.1 Loop 5R—Stress Rupture

Run 5R-4 1300°F ($T_{\max}$), 425°F (ΔT).

During the report period, the loop operated at these conditions except for brief automatic shutdowns as a result of specimen failures and the failure of a heater. The loop has operated at 1300°F with the pressure applied for about 3650 hours.

As of the end of February, fourteen of the original sixteen samples have failed. The results are shown in Table 5 and Figure 10. The results show that within experimental error, the initial microstructural condition of the Type 304 SS did not have an effect on the stress-rupture properties.

Based on the sample failures to date the Type 316 SS has apparently responded more favorably to the carbide agglomeration heat treatment than the Type 304 SS. At an initial hoop stress of 12,000 psi, there was a 100% improvement in time-to-rupture as compared to the mill annealed material. However, scatter in the results, similar to that noted for Type 304 SS, is appearing at an initial hoop stress of 9000 psi.

The stress-rupture results⁽⁷⁾ of the pre-pressurized (weldrawn) samples indicated an increase in rupture ductility for the Type 316 SS carbide agglomerated material. However, the rupture times were not known. In run 5R-4, the rupture ductilities will not be known until the run is terminated. If the agglomeration of carbides in the Type 316 SS increased both time-to-rupture and the rupture ductility, it would indicate comparable strain rates (with improved properties) to the mill annealed material. However, subsequent screening of the unfailed weldrawn, pressurized samples⁽⁷⁾ in pressure controlled stress-rupture tests (in air), have indicated a reduction in rupture time accompanying the increased rupture ductility for the carbide agglomerated material versus the mill annealed material.

Both the 5R-4, Type 316 SS seamless material and the previously tested weldrawn material responded metallographically to the carbide agglomeration heat treatment. It appears that if the benefits of carbide agglomeration are to be utilized reliably, more extensive study of the structure is desirable.

The seamless Type 304 SS (used in Run 5R-4) showed a metallographically obvious response to the carbide agglomeration heat treatment. The weldrawn Type 304 SS used for the pre-pressurized

TABLE 5

BIAXIAL STRESS-RUPTURE PROPERTIES OF MILL ANNEALED AND CARBIDE
AGGLOMERATED TYPES 304 AND 316 SS IN 1300°F FLOWING SODIUM

<u>Material</u>	<u>Initial Hoop Stress, psi</u>	<u>Failure Time, hours</u>
304SS - MA	7,000	2409
	7,000	2644
304SS - CA	7,000	2409
	7,000	3342
304SS - MA	9,000	1189
	9,000	1656
304SS - CA	9,000	1184
	9,000	1486
316SS - MA	9,000	3098
	9,000	NF
316SS - CA	9,000	3526
	9,000	NF
316SS - MA	12,000	830
	12,000	830
316SS - CA	12,000	1497
	12,000	1642

MA - mill annealed.

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

NF - not failed.

- 23 -

samples did not. Neither group of heat treated Type 304 SS appeared to alter the stress-rupture properties. The pre-pressurized samples did not show a change in rupture ductility.

4.1.2 Loop 4R—Stress Rupture

Run 4R-2, 10,000 hours 1300°F ($T_{\max}$), 500°F (ΔT).

During the report period, the samples were fabricated and the run started on December 31, 1968. The loop has presently operated at 1300°F with the pressure applied for about 1300 hours. As of February 28, no rupture had been recorded

4.1.3 Loop 6R—Stress Fatigue

Run 6R-3, 2000 hours 1300°F ($T_{\max}$), 425°F (ΔT).

During the report period, the samples were fabricated and the run started on January 23, 1969. Pressure was applied continuously starting February 10, 1969. The loop has presently operated at 1300°F with the pressure applied for about 350 hours. A diagram showing the pressure cycle is shown in Figure 11. Two Type 304 SS carbide agglomerated samples at initial hoop stresses of 15,000 psi (max) and 7000 psi (min) have failed at 130 and 185 hours. An extrapolation of the stress-rupture data from Run 5R-4, indicates an expected rupture time of 750 hours if there were no structural effects due to cycling

4.2 LOCALIZED ATTACK

The results of two tests in a pot of static sodium to evaluate steam cleaning⁽⁷⁾ indicated that the sodium in the pot was sufficiently contaminated to produce start of intergranular attack of austenitic stainless steels. It was thought that a subsequent loop exposure of these materials (including carbon steel to austenitic stainless steel), might provide some information concerning the mechanism of localized attack.

Plain carbon, low-alloy, ferritic stainless, and austenitic stainless steels, listed in Table 6, were exposed simultaneously in an austenitic stainless steel pot containing static sodium (100 ppm oxygen) at 1175°F for 596 hours. The microstructural appearance of the intergranular attack reported in Phase I of PA-15 was duplicated in this test. The type and degree of attack is outlined in Table 7

A rapid rate of decarburization can result in localized (intergranular) attack in plain carbon steels. The initiation and subsequent depth of attack is directly related to the initial carbon content (higher percentage C leads to greater attack). The mechanism of decarburization in sodium in a static stainless steel pot is difficult to predict. In an austenitic system, one may expect the sodium to be nearly saturated with chromium which forms stable carbides. The presence of chromium in a solution will decrease the activity of carbon in the sodium. The decrease of carbon activity is due to formation of particulate carbides either as a separate phase or in a solvated state. Hence the presence of chromium will increase the rate of decarburization and one can postulate the following thermodynamically favorable reaction as a possible mechanism of decarburization.

FIGURE 11. STRESS - FATIGUE TEST TECHNIQUE.

TABLE 6

CHEMICAL COMPOSITIONS OF THE MATERIALS EXPOSED TO 1175°F STATIC SODIUM

<u>Material</u>	<u>Composition, wt %</u>						
	<u>C</u>	<u>Cr</u>	<u>Ni</u>	<u>Mo</u>	<u>Mn</u>	<u>Fe</u>	<u>Others</u>
AISI C1008	0.06	-	-	-	0.43	Balance	
ASTM A285	0.20	-	-	-	0.55	Balance	
USS Cor Ten*	0.08	0.76	0.28	-	0.45	Balance	
ASTM A335	0.09	2.27	-	0.96		0.52	
AISI Type 502	0.07	5.00	-	0.54		0.53	
AISI Type 410	0.10	12.09	-	-	0.35	0.53	
AISI Type 430	0.08	16.50	-	-	0.44	0.53	
USS Stainless* "W"	0.07	16.80	6.60	-	0.67	0.53	0.85Ti 0.36Al
USS Tenelon*	0.07	17.47	-	-	14.03	0.53	0.46N
AISI Type 202	0.10	17.85	4.72	-	7.48	0.53	0.13N
AISI Type 304	0.06	18.80	9.40	-	1.60	0.53	

* Registered trademarks of United States Steel.

TABLE 7

SURFACE EFFECTS OF THE MATERIALS EXPOSED TO 1175°F STATIC SODIUM
FOR 596 HOURS

<u>Material</u>	<u>Electropolished</u>	<u>Surface Ground</u>
AISI C1008	No significant effect	
ASTM A285	2 mils I. G. * Slight surface oxide	0.5 mil I. G.
USS Cor Ten	2 mils I. G. With slight surface oxide	
ASTM A335	1.5 mils I. G. With heavy oxide associated with the G. B. **	
AISI Type 502	1.5 mils I. G. Heavy oxide associated with the G. B.	1 mil I. G.
AISI Type 410	1 mil I. G. With heavy surface oxide associated with the G. B.	
AISI Type 430	1 mil I. G. With heavy surface oxide associated with the G. B.	
USS Stainless "W"	1 mil attack With surface oxide	
USS Tenelon	0.75 mil attack With slight surface oxide	
AISI Type 202	1 mil attack Slight surface oxide	0.25 mil attack
AISI Type 304	1 mil I. G. Containing oxide and slight surface oxide	

* I. G.—Intergranular attack.

** G. B.—Grain Boundary.

The addition of chromium to a plain carbon steel results in formation of stable carbides in the alloy. Formation of stable carbides decreases the activity of carbon in the alloy, which in turn, decreases the rate of decarburization and the depth of intergranular attack.

The situation is further affected by the presence of high oxygen in the sodium, which will tend to promote oxidation of the chromium in the alloy. It is noted that there is no means presently available to observe most of the species in sodium directly, so the modular formulas given are only typical of the expected thermodynamic state. The oxidation and decarburization (intergranular due to the faster diffusion at the grain boundaries) of the low alloy ferritic steels can be represented by the following reaction:

Whether decarburization precedes oxidation, or the reactions occur simultaneously (as shown above) cannot be predicted from the results of this test. As the chromium content in the steel is increased, the Fe_3C carbide is replaced by the $(\text{Fe}, \text{Cr})_7\text{C}_3$ carbide. An alternative attack process that can then occur for the ferritic steels in high oxygen sodium, is the direct oxidation of the alloy carbide by the following reaction:

As the chromium content of the low-alloy ferritic steels increases, the depth of intergranular attack decreases, and the mode of attack changes toward general oxidation of the chromium. The attack remains centered around the grain boundaries. However, the chromium oxidation rate increases as the chromium content increases (increased Cr activity) resulting in the oxidation proceeding into the grains.

The effect of exposing ferritic and austenitic stainless steels to the conditions described above is one of chromium oxidation to sodium chromite or chromium oxide by the following reactions:

The oxidation tends to be intergranular, for the reason discussed above. The ferritic stainless steels were attacked to a greater extent (general and grain boundary) than the austenitic stainless steels. This is due to the higher chromium activity in ferrite.

SECTION V

LIST OF SODIUM MASS TRANSFER PROJECT REPORTS

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9. GEAP-4178—Sabol, W. W., and Lockhart, R. W., "Sodium Mass Transfer: VII Methods for Determining Impurities in Sodium Samples," September 1963.
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11. GEAP-4436—Lockhart, R. W., et al., "Sodium Mass Transfer: IX The Effect of Carbon and Carbonate Additions in a Sodium Test Loop," December 1964.
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19. GEAP-4833—Dutina, D., and Simpson, J. L., "Sodium Mass Transfer: XVII The Determination of Hydrogen in Loop Sodium and Cover Gases," June 1965.
20. GEAP-4834—Plumlee, D. E., Lockhart, R. W., and Pohl, L. F., "Sodium Mass Transfer: XVIII Studies of Carbon Movement in Pumped Sodium Test Loops," June 1965.
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7. PA-15 Quarterly Progress Report No. 7, September through November 1968, GEAP-5719.

APPENDIX

RUN 8-4 CORROSION AND WEIGHT CHANGE DATA*

Test** Zone	Weight Change (mg/dm ²)		Temperature (°F) and Velocity (fps)	Downstream (L/D)
	Tubes	Rings		
E01	-	-563	4.4	~ 149.0***
	-	-563	1309°F	↓
	-	-514		
	-	-504		
	-	-470		
	-	-526		
	-	-578		
	-	-519		
	-	-540		
	-	-514		
	-	-545		
	-	-493		
	-	-490		
	-	-444		
	-	-397		
	-	-356		
	-	-346		
E51	-488	-	4.4	~ 162.0
	-320	-	1309°F	↓
	-364	-		
	-289	-		
	-274	-		
	-268	-		
	-257	-		
	-250	-		
	-246	-		
	-252	-		
	-252	-		
	-247	-		
	-249	-		
	-236	-		
	-239	-		
FA01	-236	-	14.3	~ 162.0
	-	-560	1303°F	↓
	-	-523		
FA05	-	-484		
	-	-452	↓	↓
	-	-417		
	-	-373	↓	284.4

Appendix (Continued)

Test ** Zone	Weight Change (mg/dm ²)		Temperature (°F) and Velocity (fps)	Downstream (L/D)
	Tubes	Rings		
FA51	-633	-	14.3	175.3
	-605	-	1303°F	↓
	-581	-		
	-512	-		
	-494	-		
	-491	-		
FB01	-	-123	3.1	284.4
	-	-97	1303°F	↓
	-	-97		
	-	-95		
	-	-92		
	-	-92		
	-	-95		
FB51	-231	-	3.1	358.0
	-178	-	1303°F	↓
	-167	-		
	-160	-		
	-153	-		
	-155	-		
	-177	-		
FC31T (2338 h)	-	-258	~3	288.9
	-	-224	1303°F	↓
	-	-219		
	-	-214		
	-	-205		
	-	-203		
	-	-193		
FC51P (2338 h)	-	-195	~3	362.3
	-266	-	1303°F	↓
	-239	-		
	-234	-		
	-220	-		
	-219	-		
	-219	-		
FD01	-217	-	1303°F	↓
	-216	-		
	-	-208		
	-	-190		
	-	-192		
	-	-179		
	-	-178		
	-	-171	1303°F	↓
	-	-175		
	-	-	14.3	471.4
				492.8
				↓
				601.9

Appendix (Continued)

Test** Zone	Weight Change (mg/dm ²)		Temperature (° F) and Velocity (fps)	Downstream (L/D)
	Tubes	Rings		
FD51	-303	-	14.3	492.8
	-264	-	↓ 1303° F	↓
	-223	-		
	-212	-		
	-201	-		
FD56	-187	-		
	-169	-		605.6
FE01	-	-72	3.1	605.6
	-	-74	↓ 1303° F	↓
	-	-68		
	-	-69		
	-	-67		
	-	-69		
FE51	-124	-		
	-109	-		605.6
	-106	-	↓ 1303° F	↓
	-125	-		
	-103	-		
	-112	-		
FF01	-	-149	14.3	625.2
	-	-157	↓ 1303° F	↓
	-	-155		
	-	-141		
	-	-133		
	-	-140		
	-	-132		
FF51	-227	-		
	-170	-	↓ 1303° F	↓
	-222	-		
	-157	-		
	-228	-		
	-164	-		
	-158	-		
	-158	-		
GA51	-48	-	1.3	738.0
	-44	-	↓ 1286° F	↓
	-9	-		
	-48	-		
	-35	-		
	-38	-		
	-25	-		
	-29	-		
	-29	-		

Appendix (Continued)

Test ** Zone	Weight Change (mg/dm ²)		Temperature (°F) and Velocity (fps)	Downstream (L/D)
	Tubes	Rings		
GB51	-44	-	1.3	759.5
	-43	-		
	-39	-		
	-42	-		
	-42	-	1286° F	
	-38	-		
	-21	-		
	-34	-		764.8
HA51	-25	-	0.7	771.9
	-31	-		
	-7	-		
	-4	-	1286° F	
	-10	-		
	-5	-		
	-6	-		776.5
	-13	-	0.7	779.5
HB51	-2	-		
	3	-		
	7	-	1286° F	
	18	-		
	21	-		
	-5	-		783.9
	69	-	1.9	2.39****
	45	-		
JA51	41	-	1139° F	
	28	-		
	45	-		
	51	-		9.84
	145	-	1.9	13.5
	98	-		
	82	-	1139° F	
	89	-		
JB51	84	-		
	91	-		
	90	-		20.9
	188	-	1.9	24.6
	130	-		
	119	-		
	111	-	1139° F	
	105	-		
JC51	119	-		
	49	-		32.0

Appendix (Continued)

Test Zone**	Weight Change (mg/dm ²)		Temperature (°F) and Velocity (ips)	Downstream (L/D)
	Tubes	Rings		
JD51	264	-	1.9	35.6
	168	-	1139° F	↓
	149	-		
	129	-		
	129	-		
	131	-		
	133	-		
JE51	318	-	1.9	43.1
	188	-	1139° F	49.1
	176	-		↓
	153	-		
	168	-		
	170	-		
	202	-		
KA51	431	-	1.9	56.5
	233	-	1006° F	~60
	193	-		↓
	169	-		
	165	-		
	172	-		
	169	-		
KB51	423	-	1.9	~67
	187	-	1006° F	~73
	162	-		↓
	155	-		
	142	-		
	134	-		
	150	-		
KC51	398	-	1.9	~80
	181	-	1006° F	~86
	148	-		↓
	141	-		
	139	-		
	138	-		
	138	-		
KD51	378	-	1.9	~93
	186	-	1006° F	~96
	156	-		↓
	139	-		
	129	-		
	124	-		
	132	-		103

Appendix (Continued)

Test** Zone	Weight Change (mg/dm ²)		Temperature (°F) and Velocity (fps)	Downstream (L/D)
	Tubes	Rings		
KE51	315	-	1.9	~109
	156	-	1006°F	↓
	132	-		
	125	-		
	122	-		
	121	-		
	128	-		~116
MA52	89	-	1.9	187.5
	74	-	812°F	↓
	69	-		
	73	-		
MB51	77	-	812°F	194.0
	81	-		197.6
	71	-		↓
	57	-		
	66	-		
	59	-		
	70	-		
MC51	82	-		205.1
	165	-	1.9	208.7
	106	-	812°F	↓
	109	-		
	80	-		
	75	-		
	64	-		
MD51	98	-		216.1
	185	-	1.9	219.8
	130	-	812°F	↓
	85	-		
	95	-		
	91	-		
	103	-		
ME51	100	-	1.9	227.2
	265	-	812°F	230.9
	137	-		↓
	130	-		
	78	-		
	86	-		
	97	-		
	87	-		238.3

Appendix (Continued)

Test** Zone	Weight Change (mg/dm ²)		Temperature (°F) and Velocity (ips)	Downstream (L/D)
	Tubes	Rings		
NA01	-	46	8.1	247.3
	-	28	804° F	↓
	-	40		
	-	32		
	-	58		
	-	25		
	-	16		
NA51	45	-	8.1	249.0
	48	-	804° F	↓
	26	-		
	58	-		
	34	-		
	32	-		
	23	-		
NB01	-	13	2.5	249.0
	-	15	804° F	↓
	-	12		
	-	13		
	-	12		
	-	11		
	-	13		
NB51	27	-	2.5	271.1
	23	-	804° F	↓
	21	-		
	19	-		
	22	-		
	21	-		
	23	-		
NC01	-	-	8.1	399.8
	-	19	804° F	↓
	-	23		
	-	17		
	-	20		
	-	15		
	-	33		
NC51	14	-	8.1	420.9
	26	-	804° F	↓
	26	-		
	26	-		
	24	-		
	38	-		
	23	-		
			522.6	

Appendix (Continued)

Test** Zone	Weight Change (mg/dm ²)		Temperature (°F) and Velocity (fps)	Downstream (L/D)
	Tubes	Rings		
RA51	18	-	0.7	601.0
	20	-	753°F	↓
	17	-		
	17	-		
	20	-		
	18	-		
	16	-		
RB51	19	-	0.7	605.5
	15	-	753°F	↓
	16	-		
	16	-		
	20	-		
	18	-		
	17	-		
RC51	21	-	0.7	612.4
	18	-	753°F	↓
	18	-		
	17	-		
	15	-		
	17	-		
	21	-		
BA51	10	-	0.7	619.2
	10	-	769°F	↓
	7	-		
	6	-		
	7	-		
	10	-		
	10	-		
C01	-	0.2	4.4	741.5
	-	6	770°F	↓
	-	1		
	-	0		
	-	-5		
	-	2		
	-	-2		
	-	1		
	-	4		
	-	4		
	-	-2		
	-	0		
	-	0		
	-	-2		
			745.9	
			~ 757.0	

Appendix (Continued)

Test ** Zone	Weight Change (gm/dm ²)		Temperature (°F) and Velocity (fps)	Downstream (L/D)
	Tubes	Rings		
C01 (cont)	-	0	4.4 (cont)	~ 770.0 (cont)
	-	3	↓ 770° F (cont)	↓ ~ 770.0
	-	5		
C51	7	-	4.4	~ 757.0
	9	-	↓ 770° F ↓	↓
	9	-		
	8	-		
	7	-		
	5	-		
	9	-		
	9	-		
	7	-		
	7	-		
	11	-		
	9	-		
	10	-		
	6	-		
	8	-		
	9	-		~ 770.0

-
- * Length of Run - 3356 hours except for samples in Zones FC31T and FC51P which were exposed for 2338 hours.
- ** Letters represent test zone in the loop (first letter) and consecutive position within each zone (second letter). Sample numbers are consecutive with the first one only being given for each cartridge.
- *** Downstream is distance along flow path divided by hydraulic diameter. The hot-leg calculation starts at the outlet of Zone C (first heater).
- **** The cold-leg calculation starts at the inlet of Zone J (first cooler).

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