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SNAP-8 CORROSION PROGRAM QUARTERLY PROGRESS REPORT
FOR PERIOD ENDING MAY 31, 1965

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OAK RIDGE NATIONAL LABORATORY
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SNAP-8 CORROSION PROGRAM QUARTERLY PROGRESS REPORT
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INTRODUCTION

This report completes the series of quarterly progress reports for the work authorized to date under Interagency Agreement 40-14-63, NASA Order No. C-220-A, which initiated a corrosion-study program at the Oak Ridge National Laboratory in support of the SNAP-8 electrical generating system. A summary report is being prepared. The primary objectives of this corrosion-study program were (1) to evaluate the compatibility of the proposed structural materials with the proposed reactor coolant, NaK containing hydrogen, and (2) to provide information on the behavior, control, and disposition of the hydrogen present in the SNAP-8 primary coolant, particularly with respect to the diffusion of hydrogen from the SNAP-8 primary circuit through Croloy 9M boiler tubes into the power conversion circuit.

The program initially provided for 12 loop experiments, with time and space available in the schedule for a thirteenth loop. The 12 initial experiments included operation of eight loops to obtain information for a factorial replicate study of three variables at two levels each, two loops for a time-series study at SNAP-8 conditions, and two (or three) additional loops for investigating means of limiting hydrogen egress through the Croloy 9M cooler section of the loop. In addition, the program included a study of hydrogen permeability for three SNAP-8 container materials with and without NaK present and an investigation of the relationships between hydrogen solubility and partial pressure in NaK as a function of temperature.

Several modifications were made to the original loop program. Three "hot-spot" loops were added; in each of these a redesigned hot section more closely simulated the SNAP-8 hot-spot condition than was achievable with the loops as originally designed. Two of these loops were used to study hydrogen egress and control. Operation of four of the original loops (see Table 1) and one of the hot spot loops was deferred pending a more complete evaluation of the results from the existing loops. Finally,

two isothermal loops were operated to decarburize specimens of Croloy-9M for mechanical-property tests, and a study was made to ascertain the general effects of decarburization as found in the corrosion loops on the mechanical properties of both standard and modified Croloy 9M.

Previous reports in this series described in detail the program plan, the experiments to be conducted, and the test equipment to be used to simulate the SNAP-8 primary circuit at reduced scale. Startup and operating experience for ten loops, including the first "hot-spot" loop, were also described, including the difficulties encountered in maintaining and measuring oxygen and hydrogen levels in the NaK. Operation of eleven loops was completed.

Cold trapping was effective in controlling contaminants, and it reduced hydrogen effluent from the loops at cold trap temperatures as high as 550°F. Plugging indicators used to determine oxide levels in the loops consistently showed multiple responses that indicated the presence of contaminants other than Na₂O. Sampling devices for isolating the other contaminants showed only argon, K₂O, and Na₂O and no evidence of corrosion products or carbon compounds. Characterization of the behavior of hydrogen in the loops was studied in the light of the discovery of extraneous sources of hydrogen, and monitoring of loop hydrogen effluents was revised to overcome deficiencies found in the argon-sweep-gas and thermal-conductivity-cell technique used initially.

Procedures for post-run examination of corrosion loops were developed, and results from the examination of completed loop experiments showed some degree of corrosion and mass transfer in each loop. Low-oxygen-content loops had very little corrosion, but high-oxygen-content loops had significantly greater amounts. The oxygen level was shown to have a greater effect on the corrosion of iron-base alloys than on that of nickel-base alloys. Carbon migration was severe in all loops and was apparently independent of the hydrogen and oxygen content of the NaK at the oxygen levels investigated. Extensive decarburization was observed in the hot sections of the Croloy 9M, and, in some cases, the decarburization was accompanied by grain growth that produced very large grains. Carbon pickup was observed throughout the rest of the loop. Hydrogen at

the low levels experienced in the loops did not adversely affect corrosion and mass transfer.

It was found that chromium enrichments of the surfaces of chromized Hastelloy N piping and tubing typical of SNAP-8 fuel element cladding varied in a random fashion from 0 to 75% chromium and to a maximum depth of 0.0015 in. Such variation is not expected to have a significant effect on corrosion.

Hydrogen permeability of Hastelloy N was measured both in the presence and absence of NaK, as was the hydrogen permeability of type 316 stainless steel and Croloy 9M in the absence of NaK, as reported previously. An equation for the solubility of hydrogen in NaK was determined:

$$\log_{10} \frac{X}{P^{1/2}} = 0.0756 + \frac{274}{T},$$

where

X = hydrogen concentration, cc (STP) per g of NaK,

P = hydrogen partial pressure, atm,

T = temperature, °K.

In the absence of cold trapping, the hydrogen partial pressure in the SNAP-8 primary NaK system at 1100°F should be about 2.4×10^{-5} atm. Cold trapping at about 100°F would be needed to reduce this concentration by a factor of 10^4 and the hydrogen diffusion through the Croloy 9M by a factor of 10^2 . Solid getters for hydrogen, such as zirconium or yttrium, might also effect a similar reduction in concentration, but use of a soluble getter, such as lithium, accompanied by cold trapping at temperatures around 300°F appears to have the most promise of effective hydrogen control. Diffusion windows appear to be impractical.

This report covers progress during the eighth quarterly period of the program, March 1, 1965 to May 31, 1965. Previous reports in this series were:

ORNL-3538	For Period Ending August 31, 1963
ORNL-3604	For Period Ending November 30, 1963
ORNL-3618	For Period Ending February 29, 1964
ORNL-3671	For Period Ending May 31, 1964

ORNL-3730	For Period Ending August 31, 1964
ORNL-3784	For Period Ending November 30, 1964
ORNL-3823	For Period Ending February 28, 1965

SUMMARY

Operation of the final loop in the present phase of the corrosion program was completed during this reporting period. This loop was basically a "hot spot" loop operated with continuous cold trapping and with deuterium injection. The objectives were to ascertain differences in corrosion and mass transfer rates resulting from continuous cold trapping and to evaluate the effectiveness of cold trapping in reducing the hydrogen concentration in the loop NaK and thus the hydrogen effluent from the loop. Two hydride cold traps were provided, one for the collection of hydrogen from extraneous sources while no hydrogen was being injected into the loop, and one to collect deuterium to permit characterization of the behavior of hydrogen in the SNAP-8 primary circuit when free of effects of hydrogen from extraneous sources.

The loop operated for 2659 hr at design temperature prior to shutdown. The hydride trap for collecting extraneous hydrogen operated for 500 hr, and the deuterium trap operated for 676 hr. Extraneous hydrogen was observed with both an on-stream mass spectrometer and gas sampling techniques; however, the rate of extraneous H₂ flow diminished with time from a level of 10⁻⁵ scc/sec to 2 × 10⁻⁶ scc/sec. This level was reduced to below 10⁻⁸ scc/sec when the loop temperature was reduced to room temperature.

The introduction of deuterium into the loop successfully served to distinguish between extraneous hydrogen and that injected into the loop. The deuterium data indicated that the equilibrium pressure in the loop is approximately the same as the calculated hydrogen partial pressure for the SNAP-8 primary system in space (2.4 × 10⁻⁵ atm). Cold trapping at 120°F reduced the D₂ outflow from the loop by a factor of 2500, while cold trapping at 260°F, a temperature more appropriate to the SNAP-8 system, reduced the D₂ outflow by a factor of 6.

Analytical examinations were completed on all but the final loop (14-4). Results of these examinations in general confirmed previous observations. Very little metal migration occurred in low-oxygen-content NaK at both temperature levels investigated; however, carbon migration was quite severe. Corrosion of the iron-base alloys in the low-oxygen-content NaK was very low, as compared with corrosion of the chromized Hastelloy N, which ranged from three to seven times as great. Increasing the oxygen content of the NaK accelerated the corrosion rate on all materials; however, the effect on the corrosion rate of Hastelloy N was much less than that experienced by iron-base alloys. High corrosion rates were observed in some loops run with low oxide content. The results are consistent with a hypothesis that intermittent high-oxide-content situations in an otherwise low-oxide-content regime are as damaging as continuous high oxide contents. Additions of hydrogen to the NaK did not produce any discernible effects on the mass transfer rates. Furthermore, the extent of carbon migration apparently was not affected by hydrogen or oxygen levels in the NaK.

Because of the extensive decarburization of Croloy 9M in the SNAP-8 corrosion loops, a study was made to determine the effects of decarburization on selected mechanical properties. Sheet tensile specimens were decarburized to a carbon content of approximately 0.002 to 0.01% by exposure to NaK in a forced-flow type 316 stainless steel loop. As shown previously, there was general deterioration of mechanical properties. A second group of specimens was decarburized and tested to determine what effect prior heat treatment might have on the mechanical properties of decarburized standard Croloy 9M and decarburized modified Croloy 9M. Data reduction and analysis of these tests are in progress.

FORCED-FLOW CORROSION-LOOP EXPERIMENTS

W. R. Huntley	R. E. MacPherson
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General Status

The status of all corrosion loop experiments proposed and operated to date is given in Table 1 and the pertinent operating conditions for

Table 1. Status of All Proposed and Operated Forced-Convection Loops

Loop and Stand Nos.	Planned Operating Time (hr)	Maximum NaK Temperature (°F)	Oxygen Content of NaK (ppm)	Hydrogen Content of NaK	Variables To Be Studied	Comparison Loop	Time of Operation At Design Conditions (hr)	Status As of May 31, 1965
1-1	2000	1400	<30	0	Temperature Oxygen content	1A, 10 2	701 ^a	Complete
1A-1	2000	1400	<30	0	Hydrogen content Temperature	4 10	2003	Complete
2-2	2000	1400	~80	0	Oxygen content Hydrogen content Temperature	2 4 9	2004	Complete
3 ^b	2000	1400	~80	SNAP-8 ^c	Oxygen content Hydrogen content Temperature	1 3 7	0	Deferred ^b
4-4	2000	1400	<30	SNAP-8	Oxygen content Hydrogen content Temperature	4, 6 2 8	2033	Complete
5-5 6 ^b	6000 2000	1400 1400	SNAP-8 ^d SNAP-8	SNAP-8 SNAP-8	Oxygen content Hydrogen content Operating time	3, 6 1A, 1 6	5133 0	Complete Deferred ^b
7-3	2000	1300	~80	SNAP-8	Oxygen content Hydrogen content Temperature	3, 4 11, 12 3	2021	Complete
8-4	2000	1300	<30	SNAP-8	Oxygen content Hydrogen content Temperature	8 9 4	2053	Complete
9-2	2000	1300	~80	0	Oxygen content Hydrogen content Temperature	7 10 2	2000	Complete
10-1	2000	1300	<30	0	Oxygen content Hydrogen content Temperature	10 7 10	2000	Complete
11 ^b 12 ^b 13-3 14-4 15	2000 2000 2000 2000 2000	1400 1400 1450 1450 1450	SNAP-8 SNAP-8 SNAP-8 ~30 SNAP-8	SNAP-8 SNAP-8 SNAP-8 Low SNAP-8	Hydrogen content Hydrogen content Hydrogen content Hot spot Continuous cold trapping No Croloy 9M in system	8 6, 12 6, 11 6 13 14, 13	0 0 2000 2659 0	Deferred ^b Deferred ^b Complete Examination in progress Deferred

^aRun completed.

^bSee previous report ORNL-3730, p. 7, for reasons for deferral.

^cThe entry SNAP-8 indicates that the hydrogen content is that expected in the full-scale SNAP-8 system.

^dThe entry SNAP-8 indicates an oxygen level simulating that of the full-scale SNAP-8 system with initial oxygen removal from the NaK and no further oxygen trapping during operation.

each loop are described. Operation of the final loop in the corrosion program, loop 14-4, was completed, and the loop is currently being examined metallurgically. A summary report is being prepared.

Operation of Loop 14-4

Loop 14-4 was the second hot-spot loop to be operated. It was similar to previously operated loop 13-3, except that deuterium was injected into the NaK circuit in place of hydrogen and nearly continuous cold trapping was employed. These modifications in the test procedure were made in order to determine the differences in corrosion and mass transfer that might result from a controlled oxide level and to evaluate the effectiveness of cold trapping in reducing the hydrogen content of the SNAP-8 primary system. Deuterium was used to avoid the problem of extraneous hydrogen that occurred in previous tests.¹ Two cold traps were provided for the primary purpose of collecting hydrides and deuterides during the course of the test. In addition a larger cold trap was used primarily for removal of oxides from the system prior to operation of the deuteride traps. The deuteride traps also serve to maintain the oxide at a low level during the course of the test.

Extraneous Hydrogen Measurements on Loop 14-4

Extraneous hydrogen was observed with the thermal-conductivity cell of the hydrogen-monitoring system and by separate mass spectrometer analyses of gas samples taken from loop 14-4 during shakedown operations. One of the hydride traps was operated for 500 hr in an attempt to collect hydrogen from extraneous sources during a period in which no deuterium was being injected. The trap was then isolated from the loop by valving and kept isolated during the remainder of the test. Its contents are being analyzed chemically.

¹W. R. Huntley et al., Forced-Flow Corrosion-Loop Experiments, p. 10, SNAP-8 Corrosion Program Quart. Progr. Rept. Feb. 28, 1965, USAEC Report ORNL-3823, Oak Ridge National Laboratory.

Deuterium injection into the loop was started on March 19 at a rate of 0.3 scc/hr. This rate of injection was expected to produce a deuterium partial pressure within the loop similar to the equilibrium hydrogen partial pressure in a SNAP-8 primary system in space (2.4×10^{-5} atm). Deuterium outflow from the loop was monitored with a mass spectrometer, which was attached to a jacketed portion of the loop. Measurements showed the deuterium background from the annulus to be less than 10^{-8} scc/sec prior to D₂ injection. The D₂ outflow gradually increased when injection started and reached an equilibrium value of about 10^{-5} scc/sec after three days of operation. The time to attain equilibrium in loop 14-4 was much shorter than the 9- to 10-day periods required for two previous loops in which hydrogen was injected at a rate of 0.6 scc/hr. This considerable variation in equilibration time is not yet explainable.

The time to reach equilibrium was rechecked in loop 14-4 just prior to loop termination. For this test the loop was cold trapped at 100 to 115°F for 48 hr to remove deuterides from the NaK. Deuterium injection was then started and the buildup time was again observed. The loop reached equilibrium in approximately two days.

After the initial steady-state level of deuterium had been obtained in the loop (with no cold trapping), a series of gas samples was taken by passing argon sweep gas through one of the three annuli not attached to the on-line mass spectrometer. Gas samples were removed downstream of the sleeves and were analyzed with another mass spectrometer. These samples provided a means of determining deuterium levels in the loop NaK because the deuterium pressure in the slowly moving argon purge flow in the annulus tended to equilibrate with the deuterium pressure in the loop. This method of sampling was discussed in detail in a previous report.² The data from the sampling are presented in Table 2. The source of the hydrogen that increased the hydrogen level of the argon above the original value is undefined. The deuterium data, although scattered, indicated that the equilibrium pressure in the loop was approximately the same as

²E. L. Compere et al., SNAP-8 Chemistry Studies, p. 44, SNAP-8 Corrosion Program Quart. Progr. Rept. Aug. 31, 1964, USAEC Report ORNL-3730, Oak Ridge National Laboratory.

Table 2. Analysis of Gas Samples Removed from Loop 14
Detection Sleeves Prior to Cold Trapping

Argon purge flow: 10 cc/min
Original H₂ content of argon: 6 ppm

Date	Sample No.	Annulus No.	Sample Analysis (ppm)		
			H ₂ (a)	HD	D ₂ (a)
3-25-65	109	1	10	30	30
3-29-65	110	1	20	30	10
3-31-65	111	1	40	30	<10
3-31-65	112	1	40	40	10
3-31-65	113	4	20	20	30

^a1 ppm \cong 1×10^{-6} atm pressure.

the calculated hydrogen partial pressure of 2.4×10^{-5} atm expected in a SNAP-8 primary NaK system in space.³

The deuterium injection system in loop 14-4 and the accompanying mass spectrometer detection system, which can identify H₂, HD, and D₂, proved useful in observing the performance of cold traps in the loop. Extraneous hydrogen was observed throughout operation, but this was readily distinguishable from the deuterium outflow, which was of prime interest. Hydrogen outflow rates of about 10^{-5} scc/sec were observed when the mass spectrometer was attached to the detection annulus after approximately 1000 hr of loop operation. The amount of hydrogen flow generally decreased during the next month of operation to about 2×10^{-6} scc/sec. The hydrogen outflow remained at this general level throughout the final two months of operation. Numerous tests were made during the operating period which demonstrated that the flow of hydrogen decreased below 10^{-8} scc/sec when the loop was cooled to room temperature.

Control of Hydrogen Flow by Cold Trapping

A series of cold-trapping tests was made to determine the reduction of deuterium flow from the loop as a function of cold-trap temperature.

³Ibid., p. 36.

The loop was first allowed to reach equilibrium at design temperature with no cold trapping and with D₂ injection at a rate of about 0.3 scc/hr. Deuterium flow from the loop was monitored by the mass spectrometer leak detector. Prior to cold trapping, the D₂ flow from the jacketed portion of the loop was 2.5×10^{-5} scc/sec. Cold trapping with the large trap at a flow rate of 0.05 gpm and at 115 to 125°F reduced the D₂ outflow to about 10^{-8} scc/sec, or a reduction by a factor of 2500. On April 13, flow was directed from the large trap to the second small cold trap at a flow rate of 0.05 gpm and at a trap exit temperature of 260°F to more nearly simulate cold trapping that might be available on a SNAP-8 reactor in space. The loop reached a new equilibrium value in about 40 hr of operation, and the D₂ outflow at this time was about 4×10^{-6} scc/sec. This represents a reduction in D₂ outflow by a factor of 6 over the untrapped level of 2.5×10^{-5} scc/sec. Typical D₂ output data obtained with the mass spectrometer attached to the loop are shown in Fig. 1. Areas of the graph in which no data are presented generally represent times when data were being taken on masses 2 and 3 (H₂ and HD). The instrumentation was not designed to give simultaneous output readings for the three masses. The cause of the general rise in the D₂ outflow in the period extending from April 15 in Fig. 1 is not definitely known. However, post-run examination of the outer surface of the NaK-containing piping within the detection annulus showed a considerable oxide film, which is believed to have accumulated during early operation of the argon sweep gas system in this annulus. It is probable that this oxide film was being gradually reduced by the deuterium flow through the metal wall, and this is thought to account for the increasing D₂ flow rate at the annulus.

The D₂ partial pressure in the test loop prior to cold trapping was measured, as shown in Table 2, and found to be approximately the same as the estimated hydrogen partial pressure in a SNAP-8 primary system in space, that is, about 2.4×10^{-5} atm. Therefore, the reduction in D₂ outflow observed at the loop as a function of cold trap temperature is a reasonably good approximation of the reduction of hydrogen flow that might be accomplished in the SNAP-8 machine if attempts were made to reduce the amount of hydrogen permeating into the mercury boiler by cold trapping.

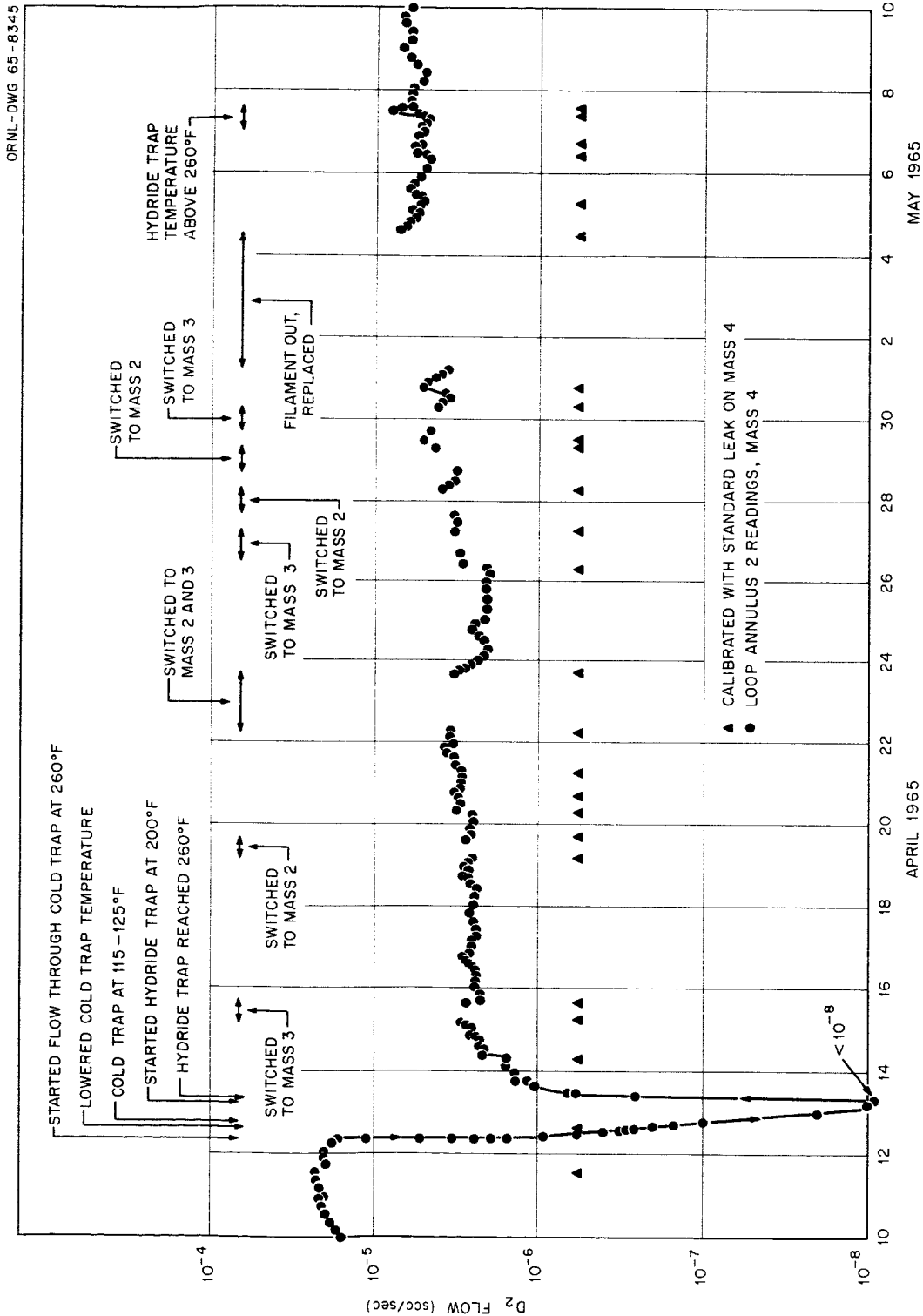


Fig. 1. Deuterium Flow from Annulus 2 of Loop 14-4. Measurements made with mass spectrometer attached to annulus around NaK piping.

The loop was operated for 676 hr with the second small cold trap in operation and with continuous deuterium injection at a rate of 0.3 scc/hr. The trap was isolated by valving at the end of the period on May 11 and is presently being chemically analyzed for O₂, D₂, HD, and H₂.

Operation of the loop was terminated on May 17 because of a NaK leak in the 1/4-in.-OD, 0.035-in.-wall Hastelloy N tubing at the point where the hot-spot flow returned to the loop surge tank. The failure was in either the weld or the heat-affected zone where the tubing passed through a reducer fitting in the surge tank wall. A total operating time of 2659 hr had accumulated at design temperature at the time of the NaK leak. The loop operating history is shown in Fig. 2, along with data from the oxide plugging indicator.

CORROSION-LOOP MATERIAL STUDIES

A. Taboada B. Fleischer

Decarburization of Croloy 9M

A test program was initiated to ascertain the general effect of decarburization on the mechanical properties of both standard and modified Croloy 9M. The plan for the second group of tests, outlined in Table 3, provides for determining whether the heat-treatment conditions prior to decarburization have a significant effect on the properties after decarburization.

The specimens were decarburized in a type 316 stainless steel-NaK loop at 1425°F for 400 hr as described previously.⁴ Control specimens were given the same exposure in helium. The tensile and strain-rate tests were performed, and data reduction and analysis are in progress. Creep-rupture tests were initiated.

⁴A. Taboada and B. Fleischer, Corrosion-Loop Material Studies, pp. 21-23, SNAP-8 Corrosion Program Quart. Progr. Rept. Nov. 30, 1964, USAEC Report ORNL-3784, Oak Ridge National Laboratory.

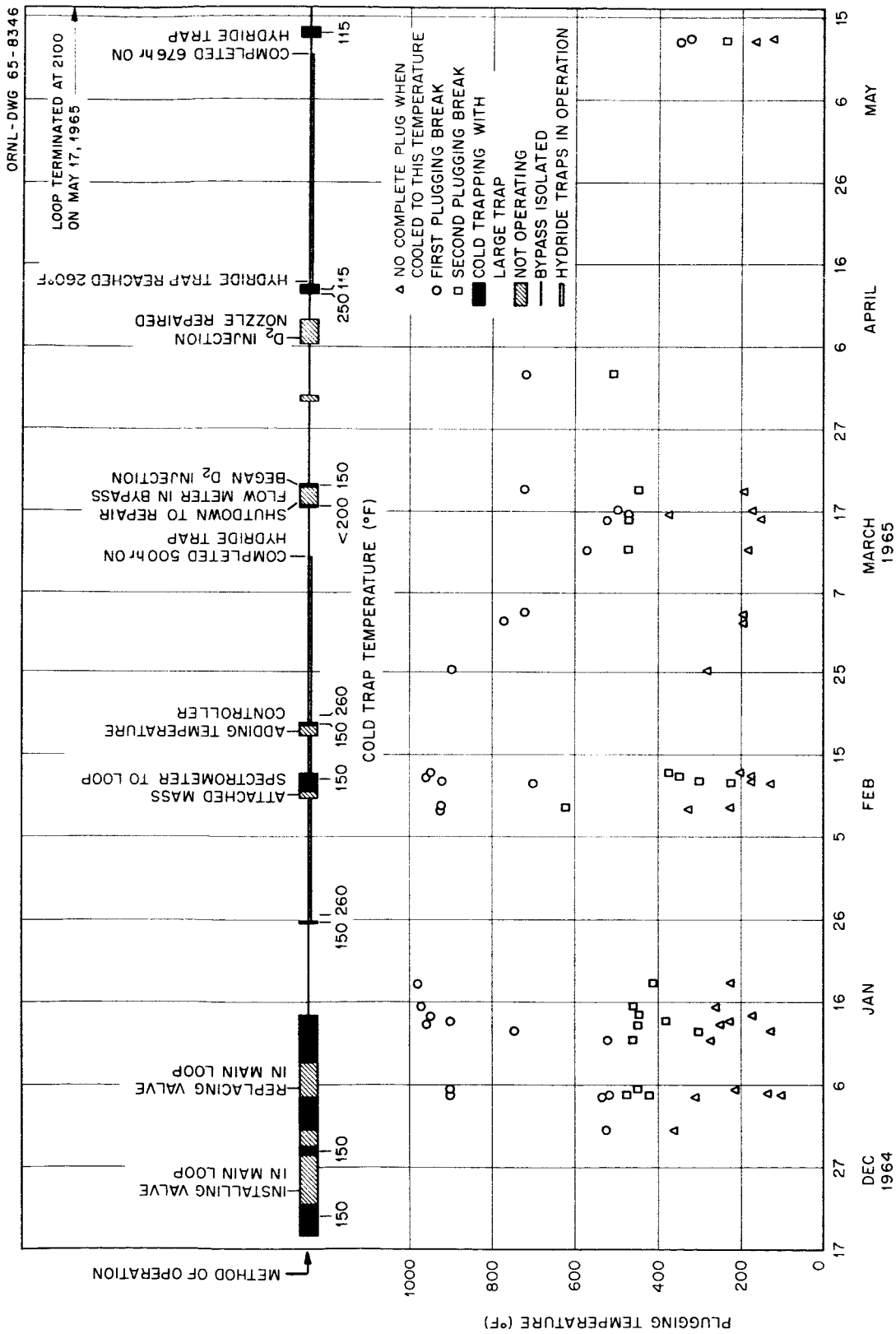


Fig. 2. Operating History of Loop 14-4.

Status of Analytical Examinations of Corrosion Loops

The status of the analytical examinations is reported in Table 4 for the loop tests that have been completed. In general, the examinations show that very little metal migration occurs in low-oxygen NaK at temperatures up to and including 1400°F; however, carbon migration is quite severe. Corrosion of Croloy 9M in low-oxygen NaK is undetectable and that of types 347 and 316 stainless steel is very low when compared with the corrosion of chromized Hastelloy N, which ranges from three to seven times as great. The oxygen content of the NaK is an important variable governing the corrosion rate of all the materials. At 1400°F, increasing the oxygen content of the NaK to approximately 80 ppm results in an increase in the corrosion rate of types 347 and 316 stainless steel by a factor of 3 to 4 over that at the low oxygen level. However, the effect of this level of oxygen on the corrosion rate of Hastelloy N is very small.

Table 4. Status of Loop Examinations

	Loop No. ^a									
	1-1	10-1	9-2	8-4	7-3	1A-1	2-2	4-4	5-5	13-3
Loop cutup and visual inspection	C	C	C	C	C	C	C	C	C	C
Posttest weighing of specimens	C	C	C	C	C	C	C	C	C	C
Carbon analysis of specimens	C	C	C	C	C	C	C	C	C	C
Carbon analysis of loop piping	C	C	C	C	C	C	C	C	C	C
Metallographic examinations	C	C	C	C	C	C	C	C	C	C
X-ray fluorescence analysis	C	C	C	C	C					
Electron microprobe analysis		C	C						C	

^aC indicates completed examinations.

Additions of H₂ to the NaK have not produced any discernible effects on the mass transfer rate of any of the materials. Furthermore, in evaluations made to date, the extent of carbon migration does not appear to be significantly affected by the level of hydrogen or oxygen in the NaK within the concentration ranges being studied.

Phase identification studies performed on types 347 and 316 stainless steel specimens exposed in the loops have indicated that the loop

exposures cause the development of carbide phases and sigma phase. Results also indicate that in type 347 stainless steel the sigma phase gradually decomposes as carburization proceeds across the specimen.

Examination of Loop 13-3

Corrosion measurements and examinations showed that very little corrosion occurred in hot spot loop 13-3, including the hot-spot area, as reported previously.⁵ Decarburization of the 0.122-in.-wall Croloy 9M piping was slight, being comparable to that found in loop 8-4.

Carbon profiles across the wall of the 0.122-in.-wall Croloy 9M piping revealed a low carbon ($\sim 0.002\%$ C) ferrite region that extended about 0.008 in. from the surface at the hot upstream end. Two feet downstream and at the same temperature the depth of decarburization was less than half as much. Between these two points, complete decarburization was found across the 0.020-in. Croloy 9M portion of the duplex type 316 stainless steel-Croloy 9M tubing all along its length. Carburization of the type 316 stainless steel at the interface was also noted. Metallographic examination and ultrasonic inspection of the bimetallic tubing showed the tubing to be sound throughout, including areas in the vicinity of butt welds and melt-through welds.

⁵A. Taboada and B. Fleischer, Corrosion-Loop Material Studies, p. 30, SNAP-8 Corrosion Program Quart. Progr. Rept. Feb. 28, 1965, USAEC Report ORNL-3823, Oak Ridge National Laboratory.

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