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SNAP-8 CORROSION PROGRAM

QUARTERLY PROGRESS REPORT

FOR PERIOD ENDING AUGUST 31, 1963

(Title Unclassified)

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SNAP-8 CORROSION PROGRAM
QUARTERLY PROGRESS REPORT

For Period Ending August 31, 1963

H. W. Savage
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Date Issued

DEC 19 1963

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INTRODUCTION

A corrosion program was initiated June 5, 1963 at the Oak Ridge National Laboratory in support of the SNAP-8 electrical-generating system. The primary objectives of the Oak Ridge National Laboratory SNAP-8 program are (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 into the power-conversion circuit.

The SNAP-8 system utilizes a compact NaK-cooled beryllium oxide-reflected reactor fueled with uranium-zirconium hydride. The containment material for the primary-circuit coolant is type 316 stainless steel, but, in addition, the NaK is exposed to chromized* Hastelloy N, to Hastelloy C, and to type 347 stainless steel in the reactor, and to Croloy 9M in the heat sink, which is a boiler for mercury. The SNAP-8 primary circuit materials and their uses are listed below:

<u>Material</u>	<u>Use</u>
Chromized and heat-treated Hastelloy N	Fuel cladding
Hastelloy C	Lower grid plate
Type 347 stainless steel	BeO cladding
Type 316 stainless steel	Reactor vessel and primary system piping
Croloy 9M and type 316 stain- less steel	Boiler material

Corrosion problems arising from use of mercury in the secondary SNAP-8 system may dictate replacement of Croloy 9M with an Nb-1% Zr alloy in areas having high corrosion potential. In this alternative design, SNAP-8 may utilize a boiler constructed of a type 316 stainless steel-columbium alloy composite, with type 316 stainless steel exposed to the

*Chromizing is the application of a chromium layer to the surface of a metal. The details of the process being used are proprietary information of the Chromizing Corporation.

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NaK. The scope of the present corrosion program is limited, however, to the initial choice of boiler tube material, Croloy 9M.

The reactor primary circuit operates with surface temperatures in the range of 1100 to 1425°F and is designed to supply 600 kw of thermal power to a boiling-mercury Rankine-cycle power-conversion system. The design life of the system is 10,000 hr of unattended operation.

The SNAP-8 reactor differs from conventional sodium- or NaK-cooled power reactors in three important respects: (1) fuel cladding temperatures are significantly higher (by about 200°F), (2) dissimilar alloys are employed in the primary circuit (see list above), and (3) hydrogen is continually discharged to the NaK coolant by dissociation of zirconium hydride in the fuel. The corrosion behavior of the SNAP-8 structural materials, excluding hydrogen effects, was examined by Atomics International under certain conditions¹ as part of the SNAP-2 Program. In the Atomics International experiments, specimens of the SNAP-8 core materials were suspended in the hot legs of thermal-convection loops circulating NaK, and the specimens showed negligible attack or interaction after 4000 hr of exposure at temperatures up to 1500°F. Earlier corrosion studies, at ORNL,² of Inconel and stainless steels exposed to alkali metal showed, however, that at temperatures above 1200°F, corrosion rates determined from thermal-convection loops were not as large as those encountered in forced-flow loops circulating alkali metal at high velocity. Both velocity and turbulence were important magnifying factors. The ORNL studies showed also that corrosion and mass transfer, while negligible at 1200°F, became significant for nickel-base alloys in which NaK was being circulated above 1300°F.

Corrosion is usually accelerated by the presence of oxygen in the NaK (which, except for an initial cleanup, may be uncontrolled in SNAP-8), and it might be augmented further by the presence of hydrogen. The continual introduction of hydrogen and its reaction with the NaK in SNAP-8

¹M. A. Perlow, "SNAP-2 Primary Coolant Development," USAEC Report NAA-SR-6439, Atomics International, July 15, 1961.

²ORNL Staff, "Summary Statement and Bibliography of Liquid-Metal Technology Developed at the Oak Ridge National Laboratory," USAEC Report ORNL CF-59-2-46, Oak Ridge National Laboratory, Feb. 16, 1959.

might also provide possible mechanisms for deterioration of flow in the NaK circuit as a result of hydride plugging in cold regions. In the primary pump, NaK at 1100°F is in direct (but essentially zero flow) communication with NaK at 300°F.

The transfer of hydrogen by diffusion through the boiler walls to the SNAP-8 secondary mercury circuit could affect performance of the secondary circuit. Firmer estimates are therefore needed on the rate at which hydrogen will diffuse into this power-conversion system.

The fuel elements are clad with chromized Hastelloy N, an alloy with high nickel content. Hastelloy N is normally less resistant than type 316 stainless steel to mass transfer in NaK above 1300°F, but the resistance of chromized and subsequently heat-treated Hastelloy N depends on the composition of the exposed surface and the manner in which the composition changes with depth. The extent to which decarburization of the Croloy 9M and carburization of the fuel elements and the type 316 stainless steel will occur is also unknown. Other imponderables include possible interactions between carbon, oxygen, and hydrogen and their effect on the mass-transfer behavior of the NaK system.

The ORNL SNAP-8 corrosion program is designed to study the significance of the problem areas. The SNAP-8 primary NaK system will be simulated as closely as possible with reduced-scale forced-flow loops operated at conditions which provide information on the effects of temperature and NaK purity on compatibility of materials in the SNAP-8 system. Loop operation will follow a statistically oriented program with variable loop parameters, including oxygen and hydrogen content of the NaK and the loop hot-leg temperature. Variations of these parameters will conform to a full replicate statistical pattern designed to analyze interdependent effects of these parameters at each of two levels. Additional loop tests will be included to investigate the effects of operating time and of methods for hydrogen elimination. Both the loops and contained specimens will be subjected to metallurgical and chemical examination. Concurrent with the loop program, experiments will be conducted to determine the relative effectiveness of gaseous hydrogen and dissolved hydride as sources of hydrogen diffusing from the primary system. Also a survey will be

made of existing data, and experiments will be conducted to determine the solubility of hydrogen in NaK as a function of temperature and hydrogen pressure.

SUMMARY

A program was initiated for investigating the compatibility of the structural materials and the NaK coolant in the SNAP-8 primary coolant loop and, also, to provide information on the behavior, control, and disposition of hydrogen, particularly with respect to its diffusion into the power-conversion system.

A loop program was planned which entailed operation of 13 reduced-scale forced-flow loops that closely simulate important features of the SNAP-8 primary system. The design of the loops, test stands, and all other appurtenant equipment was 95% completed. Two of the loops and the five test stands to be used in the program are under construction.

All materials for constructing the corrosion loops are being characterized and evaluated for conformance with quality specifications. An intense effort is being made to compare the chromizing result on Hastelloy N heater tubes with that on SNAP-8 fuel element cladding. Procedures were developed for the welding of Croloy 9M to austenitic stainless steel and Inconel.

An extensive literature survey is being conducted to evaluate the problem of determining the fate and condition of hydrogen in the SNAP-8 primary system. Sufficient data have been found to make preliminary estimates of the steady-state pressure and solubility of hydrogen in the SNAP-8 primary NaK coolant. From these estimates, it appears probable that the hydrogen will diffuse rapidly through the system walls and possibly 70% of it will enter the SNAP-8 secondary mercury system.

It was estimated that the steady-state hydrogen pressure in the SNAP-8 primary system will be a fraction of an atmosphere and that the hydrogen will be in dissolved form at NaK temperatures of 1100°F or higher. Experimental determinations of hydrogen solubility in NaK as a function of pressure and temperature are needed to verify the assumptions

and to check the accuracy of the estimates. Experiments will also be conducted to determine whether the presence of NaK affects the permeability constants for the passage of hydrogen from NaK through metals. Apparatus for carrying out these experiments was designed and is being constructed.

FORCED-FLOW CORROSION-LOOP EXPERIMENTS

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The corrosion loop program is planned for determining whether there are readily detectable conditions in the SNAP-8 primary system that contribute to serious deterioration of design performance. The proposed loops and the pertinent operating conditions of each are described in Table 1. Loops one through ten will provide data for the basic corrosion investigation, and loops 11, 12 and extra No. 1 will give information needed in studies of methods for controlling and removing hydrogen from the primary system.

For the basic study of the effects of the design variables on the mass-transfer behavior of the SNAP-8 primary system, eight loops (1, 2, 3, 4, 7, 8, 9, 10) will be operated for 2000 hr each. Four of these will operate without hydrogen addition to the NaK; the other four will operate with hydrogen injected into the NaK at a rate simulating the maximum anticipated SNAP-8 hydrogen-infusion rate. Two loops of each set of four will be operated at a constant oxygen level (<20 ppm), such as may be obtained by using cold or hot traps in the NaK circuit; the other two sets of two loops each will be operated at a constant high-oxygen level (~ 500 ppm), such as might result if the NaK in the SNAP-8 system were not passed through cold or hot traps during long-term operation. Of each set of two loops, one loop will be operated with a chromized Hastelloy N surface temperature which will result in a maximum NaK temperature of 1300°F , such as is expected to exist in the SNAP-8 reactor in regions where NaK velocity past the fuel element is high, and a NaK thermal gradient of 200°F ; the other loop will be operated with a maximum chromized Hastelloy N

Table 1. Program of Forced-Convection Loop Studies in Support of SNAP-8

Loop Designation	Operating Time (hr)	Maximum Temperature ^a (°F)	Oxygen Content of NaK (ppm)	Hydrogen Content of NaK	Variables To Be Studied	Comparison Loop
1	2000	1425	20	0	Temperature Oxygen content Hydrogen content	10 2 4
2	2000	1425	500	0	Temperature Oxygen content Hydrogen content	9 1 3
3	2000	1425	500	SNAP-8 ^h	Temperature Oxygen content Hydrogen content	7 4, 6 2
4	2000	1425	20	SNAP-8	Temperature Oxygen content Hydrogen content	8 3,6 1
5	6000	1425	SNAP-8 ^b		Operating time	6
6	2000	1425	SNAP-8	SNAP-8	Operating time Oxygen content Hydrogen content	5 3,4 11, 12, extra No. 1
7	2000	1300	500	SNAP-8	Temperature Oxygen content Hydrogen content	3 8 9
8	2000	1300	20	SNAP-8	Temperature Oxygen content Hydrogen content	4 7 10
9	2000	1300	500	0	Temperature Oxygen content Hydrogen content	2 10 7
10	2000	1300	20	0	Temperature Oxygen content Hydrogen content	10 9 8
11	2000	1425	SNAP-8	SNAP-8 with H ₂ window	Hydrogen content	6, 12, extra No. 1
12	2000	1425	SNAP-8	SNAP-8 with H ₂ getter	Hydrogen content	6, 11, extra No. 1
Extra No. 1	1500	1425	SNAP-8	SNAP-8 with H ₂ window and getter	Hydrogen content	6, 11, 12

^aIn the loops run at 1425°F, the maximum temperature is the maximum metal surface temperature; in the loops run at 1300°F, the maximum temperature is the NaK temperature.

^bThe entry SNAP-8 indicates that the oxygen or hydrogen content will be that expected in the full-scale SNAP-8 system.

surface temperature of 1425°F, to simulate the expected SNAP-8 fuel-cladding hot-spot condition, and a thermal gradient of approximately 300°F in the NaK. All loops will be operated with a minimum NaK temperature of 1100°F in the main circulating stream.

The above-described pattern for eight experiments is a full replicate statistical pattern, as shown in Table 2, for three variables (oxygen and hydrogen content and maximum fuel-cladding surface temperature) with each at two levels. Data from these experiments will be programmed for computer analyses and will be sufficient to determine the effect of each variable alone or the effects of interactions of combinations of the variables.

Table 2. Full Replicate Experimental Plan for SNAP-8 Corrosion Loop Studies of Three Variables

Maximum Temperature, ^a °F		Eight Loops Each Operated 2000 hr			
		1425	1300	1425	1300
Oxygen content of NaK	~20 ppm	Loop 1	Loop 9	Loop 4	Loop 8
	~500 ppm	Loop 2	Loop 10	Loop 3	Loop 7
Hydrogen condition		Without hydrogen		With hydrogen	
NaK temperature difference, °F		~300	200	~300	200

^aIn the loops run at 1425°F, the maximum temperature is the maximum metal surface temperature; in the loops run at 1300°F, the maximum temperature is the NaK temperature.

Many previous studies have shown that a marked increase in corrosion and mass transfer is to be expected as a result of increasing the oxygen content from 20 to approximately 500 ppm, and a definite but perhaps less severe increase is to be expected as a result of increasing the maximum surface temperature from 1300 to 1425°F. The effect of hydrogen is unknown.

In addition, the pair of loops numbered 5 and 6 are to be operated at conditions simulating the most severe hydrogen and temperature situations

expected and the oxide situation resulting from procedures and methods of operation planned for the SNAP-8 primary system. Loop 5 will be operated for 6000 hr and loop 6 for 2000 hr to study the effect of time on the corrosion and mass transfer behavior. The results from loop 6 will also be compared with those of all other loops also run for 2000 hr. In most alkali metal loops, corrosion rates are highest at the beginning of the time period and decrease as exposure lengthens.

All the corrosion test loops will include a dead-leg cold trap which may be expected to act like a diffusion cold trap exposed to 1100°F NaK to simulate the narrow annulus around the shaft of the SNAP-8 primary pump through which NaK at 1100°F in the SNAP-8 NaK primary circuit is in direct (but essentially zero flow) communication with the NaK at 300°F circulating within the SNAP-8 pump in an auxiliary closed circuit to cool windings and bearings.

The remaining three loops will be operated, as required, for studying hydrogen-distribution control and hydrogen removal, and they will include diffusion windows and chemical getters, both separately and in combination.

Corrosion-Loop Design

The corrosion loops were designed to be as nearly identical configurations as possible. The arrangement is shown isometrically in Fig. 1 and as a schematic flow diagram in Fig. 2. The main circuit of the loop is arranged approximately rectangularly in a vertical plane.

NaK is heated at the top of the loop as it passes through 70.2 in. of 3/4-in.-OD chromized Hastelloy N tubing. Heat (~13.4 kw) is supplied externally by clamshell radiant-wire heaters. The NaK then passes through 4.0 in. of type 347 stainless steel tubing and through the bottom part of an inert-gas-pressurized, type 316 stainless steel, cylindrical surge tank containing level indicators, NaK sampling ports, and (when required) a hydrogen-injection device. Thirty inches of 3/8-in.-OD type 316 stainless steel carries the flow at the maximum NaK temperature to 81.4 in. of 1-in.-OD Croloy 9M tubing; this tubing simulates the SNAP-8 mercury boiler, and about one-half of it is finned for cooling purposes. As the

NaK leaves the Croloy 9M heat exchanger, it passes over an insert of Hastelloy C, past the dead-leg cold trap simulating the SNAP-8 pump-shaft annulus, through 4 in. of 1/2-in.-OD type 347 stainless steel tubing, through 24 in. of 1/2-in.-OD type 316 stainless steel tubing, through the type 316 stainless steel cell of a standard General Electric G-3 electro-magnetic pump, through 96 in. of 3/8-in.-OD type 316 stainless steel tubing, past an electromagnetic flowmeter, and back into the heater section. A dump tank is connected to the bottom of the loop and isolated from it by a valve.

A bypass between the electromagnetic pump discharge and the inlet to the Hastelloy N heater provides three individually operable parallel paths with isolation valves. One path includes a hot trap containing zirconium foil, the second includes an air-cooled finned cold trap, and the third includes a precooler and a valve that can be used as a plugging indicator. These paths are used to determine and control the oxide concentration in the NaK in the main circulating loop. If oxygen addition is needed to maintain a level of approximately 500 ppm for the high oxide level runs, it will be added by a "tea bag" or other suitable technique through the NaK sampling port in the main-circuit surge tank.

NaK samples will be periodically taken from the high-temperature region of the loop through a ball valve and sampling-bucket apparatus located at the top of the surge tank. Corrosion specimens of Hastelloy N (chromized), type 316 stainless steel, Croloy 9M, type 347 stainless steel, and Hastelloy C are mounted throughout the main loops and will be checked metallographically and for weight changes at the end of each test run, at which time the main loop will also be cut apart and examined. The size, location, and other pertinent information regarding the specimens are given in Table 3.

Five test stands with associated instrumentation are being built to carry out the program of 13 experiments. At the end of a typical 2000-hr run the main loop piping will be removed for examination; however, the bypass oxide removal and control systems will be reused with subsequent loops installed in the same test stand.

Data on the SNAP-8 primary system volumes and surface areas to be simulated are presented in Table 4. These data reflect the most recent information from the reactor and systems designers, Atomics International and Aerojet General Corporation, respectively. The loop design was based on matching as nearly as practical the SNAP-8 NaK velocities, surface-to-volume ratios, and surface-area ratios of the various alloys in the primary circuit. The test loops were scaled down to about 1/45 the SNAP-8 size to allow the use of small components and power supplies. A summary of the selected pipe sizes and other design data for the final loop configuration is given in Table 5. Comparisons of parameters of the SNAP-8 system and of the final loop design are presented in Table 6.

Table 3. Corrosion Loop Materials and Dimensions

Specimen No.	Material	Shape	Size	Location	Comments
1-4	Chromized Hastelloy N	Thin-walled cylinder	0.560 in. OD, 0.010-in. wall, 1 in. long	Evenly spaced along heated Hastelloy N area	This material was supplied by Atomics International
5	Type 347 stainless steel	Thin-walled cylinder	0.682 in. OD, 0.020-in. wall, 1 in. long	Entrance to surge tank	
6	Type 316 stainless steel	Thin-walled cylinder	0.375 in. OD, 0.020-in. wall, 1 in. long	Mounted in surge tank	
7	Croloy 9M	Thin-walled cylinder	0.778 in. OD, 0.030-in. wall, 1 in. long	Inlet of Croloy 9M section	
8-11	Croloy 9M	Flat sheet	1 x 11/16 in., 0.030 in. thick	Evenly spaced in the finned cooler section	
12	Hastelloy C	Thick-walled cylinder	0.778 in. OD, 0.415 in. ID, 3 1/16 in. long	Exit of Croloy 9M section	This sample also represents the surface area of Hastelloy C needed to mock up the SNAP-8 grid plate
13	Type 316 stainless steel	Thin-walled cylinder	0.375 in. OD, 0.020-in. wall, 1 in. long	Downstream of electromagnetic pump	
14	Type 316 stainless steel	Thin-walled cylinder	9/32 in. OD, 0.010-in. wall, 1 in. long	Entrance to main heater section	
15, 16	Type 316 stainless steel	Flat sheet	1 x 11/16 in., 0.010 in. thick	Finned cooler section	Specimen 15 is located between specimens 9 and 10, and specimen 16 is located between specimens 10 and 11

Table 4. SNAP-8 Design Data

Surface	Material	Area (ft ²)	Percentage of Total Area	Temperature Distribution	
				Percentage of Area at 1100-1200°F	Percentage of Area at 1200-1300°F
Reactor core areas	Hastelloy N	45.2			
	Hastelloy C	1.35			
	Type 316 stainless steel	9.63			
	Type 347 stainless steel	5.31			
Other primary NaK system areas					
Piping from reactor to boiler (14 ft of 2-in.-OD, 0.049- in.-wall pipe)	Type 316 stainless steel	7.0			
Boiler tubes	Croloy 9M	72.3			
Boiler shell	Type 316 stainless steel	30.6			
Piping from boiler to reactor (17.3 ft of 2-in.-OD, 0.049- in.-wall pipe)	Type 316 stainless steel	8.65			
Parasitic load heater	Type 316 stainless steel	9.17			
Total areas, primary NaK system	Type 316 stainless steel	65.05	34.45	59	41
	Hastelloy N	45.2	23.8	50	50
	Hastelloy C	1.35	0.7	100	0
	Type 347 stainless steel	5.31	2.8	50	50
	Croloy 9M	72.3	38.25	50	50
Grand total area		189.21			

Table 5. Summary of Loop Design Data

Loop Section	Material	Pipe or Specimen Size	Inside Diameter (in.)	Length (in.)	Surface Area (in. ²)	Cross Sectional Area (ft ²)	NaK Volume (in. ³)	NaK Flow Velocity (ft/min)
Heated leg	Hastelloy N	3/4-in.-OD, 0.072-in.-wall tubing	0.606	70.2	134	0.0020	20.3	134
	Hastelloy N specimen ^a Type 347 stainless steel	1/2-in. sched-40 pipe	0.622	4.0	14 ^b 7.8	0.0021	1.2	127
Surge tank	Type 316 stainless steel	3-in. sched-40 pipe	3.068	4.45 ^c	58.4 ^c	0.0515	41.8	5.2
		4-in. sched-40 pipe	4.026					
Surge tank baffle and level-indicator tube	Type 316 stainless steel	1/8-in.-thick plate and 3/8-in.-OD tubing			8.2 ^c			
Specimen in surge tank	Type 316 stainless steel	3/8-in.-OD, 0.020-in.-wall tubing			2.4			
Hot leg from surge tank to heat exchanger	Type 316 stainless steel	3/8-in.-OD, 0.049-in.-wall tubing	0.277	30	26.1	0.00042	1.8	652
	Croloy 9M	1-in.-OD, 0.109-in.-wall tubing	0.782	81.4	200	0.00334	30.1	79.7
Heat exchanger	Croloy 9M specimen ^a	0.030-in.-thick sheet			5.5 ^b			
	Type 316 stainless steel specimens ^a	0.010-in.-thick sheet and 3/32-in. rod			6.9			
Cold leg (1100°F)	Hastelloy C insert ^d		0.415	3.06	4.0	0.00094	0.4	290
	Type 347 stainless steel	1/2-in. sched-40 pipe	0.622	4.0	7.8	0.0021	1.2	118
	Type 316 stainless steel	1/2-in.-OD, 0.065-in.-wall tubing	0.370	24	28.0	0.00075	2.6	344
	Type 316 stainless steel	3/8-in.-OD, 0.049-in.-wall tubing	0.277	96	83.7	0.00042	6.86	612
Totals					569.3		115.3	

^aMetal specimens listed are those that fit loosely inside piping and therefore present added area in contact with flowing NaK.

^bArea of four specimens.

^cBased on 4.45-in. depth of NaK in surge tank.

^dLocated at exit of Croloy 9M tubing.

Table 6. Comparison of SNAP-8 System and Test Loop Features

	SNAP-8	Test Loop
NaK velocity, ft/min		
In Croloy 9M	22.6 ^a	79.7
In type 316 stainless steel supply line	684 ^b	652
In type 316 stainless steel surge tank	22.6 ^c	5.2
In type 316 stainless steel return	662	612
In Hastelloy N	134	134
In type 347 stainless steel	134	127
In Hastelloy C	290 ^d	290
Ratio of Hastelloy N area to Croloy 9M area	0.625	0.643
Ratio of Hastelloy N area to NaK volume	15.8	15.4
Hastelloy N heat flux, w/in. ²	100	100 ^e
Reynolds number in Hastelloy N area	18,100	53,000
Ratio of Hastelloy N area to type 316 stainless steel area	0.695	0.693
NaK flow, lb/hr	36,000	720
Volume of NaK, ft ³	2.86	0.067

^aMaximum velocity in SNAP-8 boiler.

^bBased on 2-in.-OD, 0.049-in.-wall tubing in SNAP-8.

^cMaximum velocity in SNAP-8 boiler.

^dMaximum velocity in SNAP-8 grid plate.

^eFor loops operated at 1425°F.

The design of the loops and all appurtenant equipment is 95% complete. The flowsheet for loops without hydrogen injection is shown in Fig. 2. The first two loops being fabricated are of this design. Work is still in progress on the flowsheets and piping system drawings for the hydrogen-detection and -injection systems, which will be used on the third, fourth, and fifth loop assemblies and test stands.

A full-scale glass mockup of the surge tank was built to check turbulence when operated with water flowing at the design NaK flow rate of 2 gpm. Performance was satisfactory at all liquid levels 2 1/2 in. above the bottom of the tank, but to further assure a quiet liquid surface, a

small baffle was added just above and opposite the entrance pipe in the side of the vessel.

Hydrogen-Detection System

Hydrogen will be injected into the NaK through a Hastelloy N diffusion tube, which will be located in the loop surge tank near the bottom. The injection rate will be about $1 \text{ cm}^3 \text{ (STP)/hr}$ to mock up the predicted SNAP-8 hydrogen release rate. For loops operating at 1300°F , the diffusion tube will be 0.103 in. OD and $1/2$ in. long with a 0.020-in. wall. A driving pressure of 100 psi of purified hydrogen will be used. A diffusion tube wall thickness of 0.030 in. will be used on loops operating at 1425°F . The hydrogen flow will be measured periodically by isolating a small known volume of gas adjacent to the tube and observing the pressure drop. A bench test system was designed and is now being assembled to check out this device.

Four areas of the loop will be monitored for hydrogen loss. These areas represent 18% of the total main loop area and are located (1) in the type 316 stainless steel pipe at the exit of the surge tank, (2) in the Croloy 9M vertical section ahead of the heat exchanger, (3) in the Croloy 9M section at the exit of the heat exchanger, and (4) in the type 316 stainless steel tube ahead of the heated section. These areas were chosen as representative of the maximum and minimum temperature regions for the respective materials. Purified argon sweep gas will be passed through an annulus around the selected segments of the loop piping, and gas will be monitored for hydrogen content by Gow-Mac Instrument Company, model 9199, thermal-conductivity cells. A cell is presently being calibrated with known mixtures of gases. Full-scale output is obtained with argon containing 200 ppm of hydrogen. Argon flow will be maintained at all times when the loop is at temperature to prevent oxidation of the diffusion surfaces.

Corrosion-Loop Fabrication

Essentially all equipment and materials have been procured for the loops to be operated without hydrogen injection. Materials yet to be

procured for the hydrogen-injection loops are a thermal-conductivity cell, tubing and fittings, expansion bellows for the detection annuli, the hydrogen-injection system, and the flowmeters and flow recorders for the argon sweep gas.

Fabrication of the first loop is nearly completed. This loop is shown in Fig. 3 just prior to placing of the insulation. The corrosion-loop area and the present status of fabrication of loops 1 and 2 is shown in Fig. 4. The welding of all NaK containment pipe and tubing has been completed for the second loop.

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Fig. 3. SNAP-8 Corrosion Loop 1 Installed in Test Stand.

Fig. 4. Corrosion-Loop Area Showing Loops 1 and 2 Being Installed.

The major components for three additional loops have been fabricated and are ready for assembly in test stands 3, 4, and 5.

CORROSION-LOOP MATERIAL STUDIES

A. Taboada B. Fleischer
W. J. Leonard

Metallurgical studies related to the SNAP-8 corrosion-loop program include (1) establishing an optimum arrangement of the location of corrosion specimens to maximize data obtained on weight changes and mass transfer, (2) metallographic and analytical examination and evaluation of the loop material, including specimens, prior to loop assembly and on completion of each test, and (3) analyzing the effects of the metallurgical results when applied to the SNAP-8 system.

The locations and shapes of the test specimens are listed in Table 3. Chromized Hastelloy N tubular specimens in the heated section are being supplied by Atomics International from actual fuel-cladding material that was processed in the manner specified for the SNAP-8 fuel elements.

The Croloy 9M heat exchanger sections of the loops contain both Croloy 9M and type 316 stainless steel specimens, since the SNAP-8 boiler contains both materials. Both the heated and heat exchanger sections have specimens evenly spaced throughout in order to determine the effects of temperature gradients.

All materials used for constructing the loops and for specimens are being characterized and evaluated, and detailed records are being kept to ensure that the materials are equivalent to those currently intended for use in SNAP-8. In particular the chromized Hastelloy N heater tube is being compared with the SNAP-8 fuel-element cladding to determine whether increasing the tube thickness will affect the chromium-rich surface layer. A quality control and inspection program has been established for upgrading materials intended for loop construction from ASTM quality specifications to Oak Ridge National Laboratory reactor materials specifications (RMS series).

On completion of each test, the corrosion specimens from within the loop will be examined metallographically and analytically for corrosion, mass transfer, weight change, and changes in surface composition.

In addition to the test specimens, an adequate sampling of sections cut from the corrosion loop main circuit will also be examined completely. Auxiliary components used with the main corrosion loop, such as oxide traps and surge and dump tanks, will be visually examined for mass transfer and gross corrosion; if no obvious deleterious effects are evident, these parts will be reused in subsequent test loops.

The metallurgical investigations will include complete examination of materials for evidence of mass transfer of alloying elements as a result of thermal gradients, dissimilar metals, or the presence of oxygen and hydrogen in the system. Mass transfer could cause precipitation of solid particles which might plug small channels or possibly result in the formation of brittle intermetallics through diffusion of elements into the base metal. Another potentially serious consequence of alloy interaction is the displacement of carbon, the interstitial alloying element, which might possibly affect the mechanical properties of the alloys used. In the SNAP-8 system, the chromized Hastelloy N may be an excellent sink for carbon leached from the steels by NaK.

Metallurgical Evaluation of Chromized Hastelloy N

A metallurgical study is in progress to evaluate the structure and chemical makeup of the Hastelloy N tubing intended for use as SNAP-8 fuel-element cladding and also of tubing to be used in the heated sections of the corrosion loops. This study is intended to ensure that the material used for the corrosion loops is typical of the SNAP-8 cladding material it simulates.

Prior to its fabrication into fuel-elements for SNAP-8, Hastelloy N tubing is given a proprietary treatment by the Chromizing Corporation which places a thin, chromium-rich, layer on both surfaces. Further processing results in the tubes being heated to 1900°F in air for 45 min. A liquid honing operation with 240-mesh Al_2O_3 is used to remove oxide

from the surfaces. The metallurgical study will be made on material given this treatment. It will include specimens from each of the tubes used in the heated sections of the corrosion loops, samples of the SNAP-8 cladding material used as test specimens in the corrosion loops, and, if possible, a statistical sampling of fuel-element cladding material from Atomics International. The results of this work will be correlated with postrun corrosion data obtained from the forced-flow test loops.

Both electron microprobe analysis and metallographic studies were begun on available material, which included five specimens of 0.010-in.- and 0.015-in.-wall tubing received from Atomics International that were processed at different times and four specimens of 0.072-in.-wall, 3/4-in.-OD tubing intended for use in the corrosion-loop heated sections. Initial microprobe results on five specimens showed that the depth of chromium penetration was generally less than 0.001 in. and in at least one area on one 3/4-in.-OD tube, no chromium layer was measured.

Preliminary metallographic studies of the chromized tubing to be used in the corrosion-loop heated sections have shown a shallow layer of precipitate in some portions of the tubing that differs in depth on outer to inner surfaces, as shown in Fig. 5. This has also been seen in Atomics International specimens of fuel cladding. In most instances this precipitate extends to less than 0.001 in. in depth. Some sections in several of the thick-walled chromized Hastelloy N tubes contain a considerable amount of material which may be another precipitate that extends approximately 0.020 in. below the surface. There is also some indication that the location of this precipitate may be limited to end sections of the tubing. In one specimen, intergranular oxidation, shown in Fig. 6, was associated with these regions. In this case the precipitate was found to contain 60% molybdenum by microprobe analysis. Chemical and x-ray analyses are in progress in an attempt to positively identify the constituent. This second type of precipitate has not been seen in any specimen of fuel cladding.

Additional samples of chromized and heat-treated Hastelloy N tubing will be examined in an attempt to characterize the surface condition, and the results will be compared with the surface characteristics of

specimens supplied by Atomics International as typical of SNAP-8 fuel-element cladding.

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Fig. 5. Section Through Wall of 0.560-in.-OD, 0.010-in.-Wall Chromized Hastelloy N Tubing Showing Differences in Precipitate Layer at the Two Surfaces. Precipitate layer is more pronounced and deeper at outer surface (bottom of picture). Etchant: 1 part lactic acid, 2 parts HCl, 1 part HNO_3 .

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Photo Y52712

Fig. 6. Cross-Section at Inner Surface of 3/4-in.-OD, 0.072-in.-Wall Chromized Hastelloy N Tubing Showing Intergranular Oxidation Associated with Extensive Second-Phase Precipitation. Etchant: 1 part lactic acid, 2 parts HCl, 1 part HNO_3 .

Quality Control of Structural Materials

All materials used in loop construction were procured in accordance with applicable standard ASTM specifications. Although application of the materials in the corrosion loops justifies the use of the more restrictive ORNL Reactor Material Specifications (RMS series), the latter specifications cannot be met normally by existing warehouse inventories. Since the required special mill orders could not be obtained in a time consistent with the schedule for operating the loops, the material used was taken from existing warehouse inventories; however, it was inspected on site and upgraded to RMS standards by a sorting process. Certified chemical and physical analyses were required of all vendors to show compliance with the purchase order and applicable ASTM specifications, and these were supplemented by check analyses at ORNL. A list of the ASTM specifications which served as the final criteria for material acceptance is given in Table 7, and the results of some of the ORNL check analyses are presented in Table 8, along with the specification requirements and ladle analyses presented in the receiving reports. Additional data will be reported in the same form as they become available. It may be noted that the phosphorus content on the check analysis of 4-in. sched-40 type 316 stainless steel pipe is slightly above the specification requirement and is considerably higher than that reported for the ladle analysis. A recheck is being made. This small deviation has been considered to be acceptable, since the major effect of the high phosphorus content is to limit weldability, and no welding problems were encountered with this material during the fabrication of the corrosion loops. The welds were thoroughly inspected using dye-penetrant techniques and radiography. All inspection reports and chemical analyses will be kept on permanent file, and the appropriate file number will be recorded on each material.

Welding of Croloy 9M to Austenitic Stainless Steel

The welding of Croloy 9M tubing to both austenitic stainless steel (types 316 and 347) and Inconel tubing is required in the construction of the corrosion loops. Consequently, welding procedures were developed

Table 7. Corrosion-Loop Material Specifications

Material	Item	Purchase Specification	Inspection Procedure ^a	Acceptance Specification ^{a, b}
Type 316 stainless steel	Tubing	ASTM A-213	MET-NDT-3, -4	MET-RM-A202
	Pipe	ASTM A-376	MET-NDT-3, -4	MET-RM-A200
	Plate	ASTM A-240	MET-NDT-1, -4	MET-RM-A1
	Rod	ASTM A-276	MET-NDT-2, -4	MET-RM-A102
	Fittings	ASTM A-403	MET-NDT-4	MET-RM-A300
Type 347 stainless steel	Pipe	ASTM A-376	MET-NDT-3, -4	MET-RM-A200
	Rod	ASTM A-276	MET-NDT-2, -4	MET-RM-A102
Croloy 9M	Tubing	ASTM A-213-T-9	MET-NDT-3, -4	
Hastelloy N	Tubing	MET-RM-B167	MET-NDT-3, -4	MET-RM-B167
Hastelloy C	Rod	ASTM B-336-58T	MET-NDT-2, -4	MET-RM-B336

^aORNL specifications.

^bHydrostatic tests, required by MET-RM specifications, will normally not be made in the case of materials purchased to ASTM specifications.

Table 8. Results of Chemical Analyses of Corrosion-Loop Materials

	Alloying Constituents (%)										
	C	S	Si	P	Mn	Cr	Ni	Mo	Cu	Co	Nb + Ta
Type 316 stainless steel, 1 1/2-in. bar											
Specification ^a	0.08	0.030	1.00	0.045	2.00	16-18	10-14	2-3			
Ladle analysis	0.056	0.010	0.55	0.016	1.78	17.46	12.43	2.77	0.14	0.08	
Check analysis ^b	0.053	0.007	0.46	0.029	1.67	17.8	12.2	2.73			
Type 316 stainless steel, 3-in. sched-40 pipe											
Specification	0.08	0.030	0.75	0.030	2.00	16-18	11-14	2-3			
Ladle analysis	0.040	0.004	0.34	0.026	1.30	17.27	13.40	2.74			
Check analysis	0.046	0.007	0.66	0.023	1.70	16.7	13.0	2.24			
Type 316 stainless steel, 4-in. sched-40 pipe											
Specification	0.08	0.030	0.75	0.030	2.00	16-18	11-14	2-3			
Ladle analysis	0.056	0.016	0.44	0.012	1.68	17.44	13.21	2.31			
Check analysis	0.050	0.011	0.45	0.035	1.61	17.5	12.2	2.20			
Type 316 stainless steel, 1/4-plate											
Specification	0.08	0.030	1.00	0.045	2.00	16-18	10-14	2-3			
Ladle analysis	0.045	0.011	0.54	0.022	1.75	17.23	12.78	2.26	0.17		
Check analysis	0.042	0.008	0.43	0.016	1.53	16.9	12.6	2.15			
Type 347 stainless steel, 1/2-in. sched-40 pipe											
Specification	0.08	0.030	0.75	0.030	2.00	17-20	9-13				(c)
Ladle analysis	0.039	0.008	0.56	0.018	1.72	18.95	11.07				0.56
Check analysis	0.037	0.006	0.53	0.019	1.63	18.9	10.9				0.56

^aSpecification values for C, S, Si, P, and Mn are maximum allowable values.

^bUnless otherwise noted, all check analysis data are the results of one analysis.

^cThe ASTM specification calls for ten times the carbon content but less than 1%.

for these combinations of materials. Croloy 9M is a high-alloy steel and is quite similar in metallurgical behavior to straight chromium stainless steels. It is an air-hardening steel and is therefore susceptible to cracking as a result of the thermal cycles experienced during welding.

Type 347 stainless steel 3/4-in. sched-40 tubing and Croloy 9M tubing of the same size were used in the development of a welding procedure specification that is suitable for austenitic stainless steels and Inconel. The filler metal used in all cases was a high-nickel alloy containing chromium, iron, and columbium that was designated INCO-82T. The recommended welding procedure requires preheating to 500°F, maintenance of this temperature during welding, and not allowing the temperature to drop below 300°F on completion of the welding. The weldment is given an immediate postwelding heat treatment at 1350°F/hr; it is then air cooled to room temperature. Welds made by this procedure were found to be sound when checked by dye penetrant and radiographic techniques and by metallographic examination of specimens from the weldment.

Welds were also made using three other procedures with simpler heat treatments, since it was realized that the ideal procedure might not be practical under field-welding conditions. The second procedure deviated from the first in the postwelding heat treatment used. The sample was wrapped with asbestos when the temperature reached 1350°F and allowed to air cool to room temperature. The third procedure included a 500°F preheat that was maintained during welding. The sample was then wrapped in asbestos and allowed to air cool to room temperature. The fourth procedure used no preheat or postheat. Upon completion of welding, the sample was permitted to air cool to room temperature.

All weldments were found to be sound when evaluated by liquid penetrant and radiographic inspection techniques. Metallographic specimens were taken from each weldment that included the weld metal, heat-affected zone, and base metal. All portions of the weldments were defect free at the specimen locations. The heat-affected-zone width in the base metal was approximately 0.240 in. in samples prepared by the first three procedures and 0.175 in. in the sample prepared by the fourth procedure (no preheat).

The weld-metal hardness in all cases averaged 160 DPH (1-kg load). The Croloy 9M base metal hardness averaged 155 DPH. The heat-affected zone in weldments made by the standard procedure (first) had a maximum hardness of 265 DPH adjacent to the weld metal that decreased to 175 DPH adjacent to the unaffected Croloy 9M base metal. The second procedure resulted in a maximum hardness in the base metal adjacent to the weld metal of 312 DPH, which decreased to 245 DPH adjacent to the unaffected base metal. The third procedure resulted in a maximum hardness in the base metal adjacent to the weld metal of 415 DPH, which decreased to 280 DPH adjacent to the unaffected base metal. The hardness in the Croloy 9M base metal, using the fourth procedure, was the same as in the third.

Since it was demonstrated that cracking did not result in Croloy 9M 3/4-in. pipe weldments without postwelding heat treatment, the postwelding heat treatment operation was not used after welding of the corrosion-loop heat exchanger section. In this case, the undesirable hardness and associated loss in ductility of the weld heat-affected zone was eliminated during a hydrogen-firing operation at 1800°F that was specified to remove surface oxides from the metal surfaces. The hydrogen-firing operation was followed by slow cooling to 600°F. Two heat exchanger sections were built successfully in this manner.

SNAP-8 CHEMISTRY STUDIES

E. L. Compere	H. C. Savage
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The anticipated entry of hydrogen from uranium-zirconium hydride fuel elements into the NaK circulating in the SNAP-8 reactor primary coolant circuit introduces a number of questions concerned with the fate of the hydrogen and the effects to be anticipated from its presence. Preliminary analysis indicates that much of the hydrogen will be lost through the walls containing the primary coolant, including loss into the mercury boiler system.

A hydrogen steady state will be established quickly in the primary coolant system. In the absence of bubbles or precipitates, the hydrogen will be entirely in dissolved form in the NaK. The possibility of bubbles

should be eliminated by sufficient overpressure. Precipitation as the alkali-metal hydride will depend on the temperature and the concentration of dissolved hydrogen. Because of the low concentration of dissolved hydrogen, precipitation is not anticipated in NaK circulating at 1100 to 1300°F; however, it may occur in cold traps, in cold dead-legs, or in the cool NaK used internally in the SNAP-8 pump, since this NaK communicates with primary circuit NaK (by diffusion) through the NaK-filled narrow annulus around the SNAP-8 pump shaft.

The hydrogen of the primary system is expected to be in solution, probably in the hydride form, rather than in the gas phase as hydrogen molecules. This raises the question as to whether metal container permeability data obtained for gaseous hydrogen are valid for estimating the steady-state condition of the SNAP-8 system where the predominant form of hydrogen is expected to be dissolved hydride. Therefore, in order to establish the nature and limits of the problem of hydrogen in SNAP-8 NaK, it is necessary to determine phase equilibria in the hydrogen-sodium-potassium system over the relevant ranges of temperature, composition, and pressure.

Data are available on decomposition pressures of sodium, potassium, and other alkali-metal hydrides up to 1100°F for NaH and 800°F for KH. These are presumably equilibrium pressures for systems involving two non-gaseous phases: liquid metal saturated with hydride and a saturated solid solution of metal in the hydride. The solubility of sodium hydride in sodium at temperatures up to 830°F has been reported; however, because NaK (78 wt % K) contains 6.7 at. % potassium, the solubility of potassium hydride in NaK is likely to control hydride precipitation from NaK.

For the SNAP-8 system, it is important to determine the laws governing gaseous hydrogen solubility in NaK, since the dissolved hydrogen concentration will determine the lowest temperature that can exist in the system without hydride precipitation. Sievert's law, which states that the solubility is proportional to the square root of the pressure, has been used as a reasonable assumption by some investigators. However, since no direct experimental evidence has been found in the sodium or potassium systems to verify this assumption and arguments can be made in

support of other relationships, experimental work is being undertaken for establishing the actual relationship in relevant regions. Estimates of the probable condition of hydrogen in the SNAP-8 primary coolant system are being made initially in order to direct the experimentation.

Estimate of Steady-State Pressure Resulting
from Diffusion of Hydrogen

An estimate was made of the hydrogen steady-state pressure to be anticipated in the SNAP-8 primary coolant system providing the pressure depends only on simple diffusion loss through system walls. This established an upper limit for hydrogen pressure in the system. It was assumed that in the absence of precipitated hydride, the presence of NaK would not affect this steady-state pressure.

The diffusion of hydrogen through clean metal at the design temperatures, without considering the presence of NaK, may be expressed as follows:³

$$\frac{d(H_2)}{dt} = \frac{kA}{d} (P_1^{1/2} - P_2^{1/2}) ,$$

where $d(H_2)/dt$ is the hydrogen permeation rate [cm^3 (STP)/hr], A is the area (cm^2) of metal through which diffusion occurs, d is the metal thickness (in mm), P_1 is the interior hydrogen pressure (in atm), P_2 is the exterior hydrogen pressure (in atm), and k is the permeability of the metal to hydrogen at a given temperature expressed in the above units. Values of k are available for the metals of the SNAP-8 system.^{3,4,5}

³R. M. Barrer, Diffusion In and Through Solids, Cambridge University Press, 1951.

⁴D. W. Rudd and J. B. Vetrano, "Permeability of Metals and Enameled Metals to Hydrogen," USAEC Report NAA-SR-6109, Atomics International, Oct. 30, 1961.

⁵E. A. Steigerwald, "The Permeation of Hydrogen Through Constructional Materials," Thompson Ramo Wooldridge Technical Memo ER-4776, Nov. 30, 1961.

For the entire system, consisting of i regions,

$$\frac{d(H_2)}{dt} = \sum k_i \frac{A_i}{d_i} (P_{1i}^{1/2} - P_{2i}^{1/2}) .$$

As a first approximation, P_{2i} will be taken as zero in all regions, and P_{1i} will be taken as constant. As shown subsequently, a Sievert's solubility relationship, $S_i C_H = P_{1i}^{1/2}$, is a more accurate relationship, with C_H having the same value in all regions and S_i varying with the local NaK temperature, and it leads to more refined estimates.

Under these assumptions, it is possible to estimate approximately the steady-state hydrogen pressure in the primary coolant system by examining the areas and thickness of the various surfaces containing it through which hydrogen might escape. Although the dimensions may vary as the design is modified, the dimensions cited in Table 9 are presumed to be sufficiently close that the present approximation is meaningful. The steady-state pressure may be estimated based on an assumed flow rate of $1 \text{ cm}^3 \text{ (STP)/min}$ * [or $60 \text{ cm}^3 \text{ (STP)/hr}$] as follows:

$$\frac{d(H_2)}{dt} = k \sum \frac{A_i}{d_i} P_1^{1/2}$$

or

$$60 = 0.3 \times 1108 P_1^{1/2}$$

and

$$P_1 = \sim 4 \times 10^{-6} \text{ atm} .$$

This model assumes proportionality to the square root of pressure even at very low pressures. Barrer⁶ concludes that at very low pressures

*Other rates may be substituted, if appropriate. The pressure will vary with the square of the rate.

⁶R. M. Barrer, Diffusion In and Through Solids, Cambridge University Press, 1951, pp. 171 ff.

Table 9. Surface Area to Thickness Ratios for SNAP-8
Primary System Components

Region	Metal	Surface Area (ft ²)	Metal Thickness (in.)	A_i/d_i , Area-to- Thickness Ratio (cm ² /mm)
Reactor shell	Type 316 stainless steel	9.63	0.105	3,400
Piping	Type 347 stainless steel	5.31	0.155	1,300
	Type 316 stainless steel	15.7	0.065	9,100
Parasitic load heater	Type 316 stainless steel	9.17	0.25	1,400
Boiler shell	Type 316 stainless steel	30.6	0.065	11,800
Boiler tubes	Croloy 9M	72.3	0.035	77,800 ^a
			Total	110,800

^a77,800/110,800 = ~70%.

a "threshold" pressure for gas flow through metals appears to exist but that this is a result of phase-boundary processes and that the concentration gradient in the metal is thus diminished. For iron containing hydrogen at 702°C, an apparent threshold appeared to occur at about 0.005 atm.

Since any phase-boundary process leading to reduced but real concentration gradients would lead to reduced but real permeation rates, it follows that the steady-state pressure probably falls between a "threshold" value and a simple-model value such as that estimated above. The steady-state pressure for hydrogen in the SNAP-8 primary coolant system is thereby estimated as probably between 10^{-5} and 10^{-3} atm.

These estimates indicate that with clean oxide-free metals, the steady-state partial pressure of hydrogen in the primary coolant loop may be expected to be quite low, far below 1 atm. Furthermore, it is evident that a large proportion, possibly 70%, of the hydrogen can be

expected to diffuse through the boiler tubes into the secondary system. That the transfer of much of the hydrogen into the secondary system should be expected is indicated by noting that the paths of heat and hydrogen from the fuel element and thence to the secondary system are similar. Because the secondary system boiler areas are relatively large to receive this heat, they are also large to receive hydrogen. The buildup of hydrogen pressure in the boiler vapor, which could inhibit hydrogen diffusion into the boiler, will be greatly reduced by the tendency of the mercury vapor to sweep hydrogen to the condenser outlet, where it will accumulate and possibly seriously reduce the efficiency of the condenser.

Oxide films on the metals would increase the resistance to diffusion and the steady-state pressure. However, the oxides of iron, nickel, and molybdenum are indicated by thermodynamic considerations to be reduced by sodium. They may also be reduced by hydrogen if the partial pressure of water is kept quite low, as in the vacuum of space, or in the mercury boiler. Films of Cr_2O_3 and MnO are indicated to be stable to these reducing agents, but it is uncertain whether such films as are formed on system surfaces will inhibit diffusion.

It thus appears that unless the rate of diffusion is affected by the presence of the NaK and the existence of the hydrogen in NaK in the form of hydride ion, low steady-state pressures of hydrogen will exist in the NaK, and a major part of the hydrogen will diffuse into the mercury boiler.

Phase Equilibria in the Hydrogen-Sodium-Potassium System

Two regions of phase equilibria in the hydrogen-sodium-potassium system are important in considerations of the SNAP-8 primary coolant circuit. The diffusing gas will undoubtedly dissolve in the liquid metal to an extent limited by the pressure. At certain concentrations of dissolved hydride at given temperatures, a solid hydride-rich phase will be precipitated — an effect not desirable in the SNAP-8 system. The relationships governing the gas-solubility and the solid-solubility phenomena are consequently of interest. Furthermore, NaK is a mixture of two liquid metals, and some complexity in the phase equilibria may result from this.

A critical review of the chemistry of hydrogen-metal systems was recently given by Gibb.⁷ Earlier fairly complete bibliographies on metal hydrides prepared under the surveillance of Gibb are also available.^{8,9} These sources present a broad coverage of the literature. Based on a review and analysis of the literature, estimates are given below of the solubility of solid hydride in liquid alkali metal and the solubility of gaseous hydrogen in liquid alkali metal. The results of this consideration are also interpreted in terms of the SNAP-8 situation and the proposed experimental program.

Solubility of Solid Hydride in Liquid Metal

The solubility of solid hydride in liquid metal is significant both to indicate possible precipitation temperatures and to serve as a terminal composition coordinate of the pressure-concentration relationship at a given temperature. The hydride decomposition pressure considered later will be the corresponding pressure coordinate. These pressure and composition values will then be used to establish an extrapolation to estimate the concentration of dissolved hydrogen under the steady-state pressure estimated above.

Direct evidence as to the solubility of solid hydride in liquid alkali metal is given by Williams et al.¹⁰ for the Na-NaH system. These investigators found the solubility to increase exponentially with inverse absolute temperature, in the usual way. The solubility at 250°C was 0.005 wt % NaH, and at 445°C, it was 4.3 wt % NaH.

⁷T. R. P. Gibb, Jr., "Primary Solid Hydrides," pp. 315-509 in Progress in Inorganic Chemistry, Vol. III, F. A. Cotton (Ed.), Interscience Publishers, New York, 1962.

⁸"A Bibliography on the Hydrides of Metals and Metalloids," Third Supplement, Report NP-4716, Metal Hydrides Inc., Beverly, Mass., 1952.

⁹"Hydrides of Metals and Metalloids," USAEC Report TID-3902, prepared by Tufts University, Medford, Mass., 1960.

¹⁰D. D. Williams, J. A. Grand, and R. R. Miller, "The Solubility of Impurities in Metallic Sodium," NRL Memo Report No. 424, February 1955, and "The Solubility of Sodium Hydride in Sodium," J. Phys. Chem., 61: 379-380 (1957).

The solubility of alkali halide salts in their liquid metals has been reported by Bredig,¹¹ whose study is of interest because of the possibility of regarding the hydride ion as a member of the halide ion series. A noteworthy characteristic of these systems¹² is that while the solubility increases exponentially with inverse absolute temperature, at temperatures approaching the salt melting point a two-liquid-phase region is noted in most systems.

The Na-NaH data of Williams cited above¹⁰ appear to be compatible in slope with the alkali halide data provided a hydride melting point (necessarily under great hydrogen pressure) of about 600 to 700°C is assumed. Lithium hydride has been shown¹³ to exhibit a second liquid phase and also has solubility data fitting the alkali-alkali halide pattern¹² very well. From these considerations it appears likely that the solubility of NaH in sodium will increase exponentially with inverse absolute temperature to temperatures somewhat above 600°C; at this point a second liquid phase could appear.

No solubility data are available for potassium hydride in potassium.

Plugging-meter data obtained in engineering equipment have been reported by Atomics International.¹⁴ These data show a smaller temperature coefficient than that of the NaH-Na system. The solubility of hydride in NaK is indicated to be less than that in pure sodium at temperatures above about 750°F. However, the correlation between plugging-meter data and solubilities obtained in other ways has not been established in this

¹¹M. A. Bredig, "Mixtures of Metals with Molten Salts," USAEC Report ORNL-3391, Oak Ridge National Laboratory, Aug. 16, 1963.

¹²M. A. Bredig, H. R. Bronstein, and W. T. Smith, "Miscibility of Liquid Metals with Salts, II. The Potassium-Potassium Fluoride and Cesium-Cesium Halide Systems," J. Am. Chem. Soc., 77: 1454-1458 (1955).

¹³C. E. Messer, R. A. Seales, and J. Mellor, "Solid-Liquid Equilibrium in the Lithium-Lithium Hydride Systems, III. Period Covered: June 1, 1956 to June 1, 1957," USAEC Report NYO-8021, July 26, 1957.

¹⁴D. F. Atkins, letter to E. L. Compere, August 15, 1963 cites Reports NAA-SR-Memo 4018, NAA-SR-Memo 5472, and data he obtained at Atomics International.

system. It appears, in general, that hydride is likely to be quite soluble in NaK at SNAP-8 primary loop temperatures (1100 to 1300°F).

In order to judge whether precipitation conditions might exist in cooler regions of the circuit, such as the pump coolant auxiliary loop, the solubility temperature corresponding to the hydride dissolved in the NaK must be compared with the temperature of the region. The amount of dissolved hydride will vary with the hydrogen pressure, as described below.

Dissolution of Hydrogen Gas in Liquid Alkali Metal

No data have been found for the relationship between hydrogen pressure and dissolved hydrogen concentration in either sodium or potassium, with the exception of decomposition-pressure data on the hydrides themselves.

The solubility of hydrogen, deuterium, and tritium in molten lithium has been studied,¹⁵ and the relationships in this system are being used to establish the form of the pressure-composition relationship. At 700°C a pressure plateau exists in which the two phases, liquid lithium saturated with hydride and solid (or liquid) hydride containing metal in solution, are in equilibrium. At lower hydrogen pressures, solutions of hydride in lithium exist. The relationship $C = KP^n$ was followed up to the plateau pressure. A value of $n = 0.7$ has been obtained from the data, which is somewhat above the value of 0.5 that would indicate agreement with Sievert's law.

If this functional dependence is assumed for the NaK system, an order of magnitude estimate may be made of the amount of hydrogen which will be dissolved in NaK under SNAP-8 conditions. The value of K may be estimated by substituting the plateau decomposition pressure of the hydride and the solubility of solid hydride in liquid metal.

¹⁵F. K. Heumann and O. N. Salmon, "The Lithium Hydride, Deuteride, and Tritide Systems," USAEC Report KAPL-1667, Knolls Atomic Power Laboratory, Dec. 1, 1956.

The decomposition pressure of alkali hydrides has been given by Herold¹⁶ in the temperature range 288 to 414°C as follows:

$$\text{For NaH} \quad \log_{10} P_{\text{mm}} = 11.66 - 6100/T(^{\circ}\text{K}) .$$

$$\text{For KH} \quad \log_{10} P_{\text{mm}} = 11.69 - 6175/T(^{\circ}\text{K}) .$$

At 1300°F, the decomposition pressure of NaH is indicated to be 340 atm and that of KH to be 310 atm. Other investigators¹⁷⁻¹⁹ have reported equations which, considering the extrapolations involved, agree with pressures of these magnitudes and show little difference between the decomposition pressures of KH and NaH. It is presumed that decomposition pressures of hydride in NaK will also be similar.

The solubility of solid hydride in liquid alkali metal was discussed earlier. At 500 to 600°C, Banus et al.¹⁹ found a solubility of 10 to 30 at. % NaH in sodium. Heumann and Salmon¹⁵ found the solubility of LiH in lithium at 700°C to be 25 at. %.

For the purpose of estimating the concentration of dissolved hydride in SNAP-8 NaK, the equation $C = KP^n$ is used. Assuming $C = 0.3$, $P = 310$ atm, and $n = 0.7$, K may be estimated to have a value of 0.0054. Thereby, at an assumed SNAP-8 steady-state hydrogen pressure of 10^{-3} atm, C is estimated to be 4×10^{-5} mole fraction hydride in liquid metal. This corresponds to a hydrogen weight fraction of 1 to 2 ppm. If a Sievert's law value of n of 0.5 is used, then C is estimated to be 54×10^{-5} , corresponding to a hydrogen weight fraction of about 15 ppm.

A concentration of 4×10^{-5} mole fraction hydrogen in sodium would begin to precipitate sodium hydride at 470°F, according to the data of

¹⁶Albert Herold, "Contribution a l'Etude des Hydrures Alcalins," Annales de Chemie, 12(6): 536-581 (1951).

¹⁷F. G. Keyes, "The Dissociation Pressures of Sodium and Potassium Hydrides," J. Am. Chem. Soc., 34: 779-788 (1912).

¹⁸E. F. Sollers and J. L. Crenshaw, "The Dissociation Pressures of Potassium Deuteride and Potassium Hydride," J. Am. Chem. Soc., 59: 2019-2022 (1937).

¹⁹M. D. Banus, J. J. McSharry, and E. A. Sullivan, "The Sodium-Sodium Hydride Hydrogen System at 500-600°C," J. Am. Chem. Soc., 77: 2007-2010 (1955).

Williams.¹⁰ Similarly a concentration of 54×10^{-5} mole fraction hydrogen in sodium would precipitate sodium hydride at 590°F. However, solubility data in the NaK system are not adequate to permit good estimates in the NaH system. Experimental determination of such solubilities is desirable.

In conclusion, it appears evident that no precipitation of hydride is to be expected in the 1100 to 1300°F regions of the system. Experimental work appears to be necessary, however, in order to clarify the problem at lower temperatures.

Experimental Comparison of Diffusion Through Metal of Hydrogen from the Gas Phase and Hydrogen Dissolved in NaK

As discussed above, experiments will be done to determine whether the diffusion through metals of hydrogen dissolved in NaK differs from the diffusion of gaseous hydrogen. For this purpose, a diffusion cell* was designed to contain a small volume of NaK, as well as gaseous hydrogen, in the upstream region. The cell and diaphragm are machined from solid metal. The device is to be operated in a high-temperature furnace. The cell can be rotated so that the diaphragm can be covered with hydrogen-saturated NaK or be in contact with only gaseous hydrogen. The diffused hydrogen, accumulated from the downstream region in an assembly of three bulbs by a Toepler pump, will be measured by determining the pressure through a differential manometer. Provision for sampling the gas for mass spectrometry analysis is included in the system. Comparison of relative diffusion rates under the two conditions is expected to demonstrate whether the presence of the NaK affects the diffusion rate.

Most of the equipment for handling the hydrogen recovered from the diffusion cell is complete. An automatically actuated Toepler pump was assembled and tested, and the gas-measuring bulbs were calibrated. The assembly of the vacuum manifold, the mercury diffusion pump, and the rough vacuum pump was completed. Diffusion cells of Hastelloy N were assembled, and bar stock or forgings of type 316 stainless steel and of Croloy 9M

*Except for inlets for NaK and a shortened design to permit rotation in a furnace, the cells are similar to those described in refs. 2 and 3.

are being procured. Other diffusion cells will be fabricated of these materials.

Experimental Determination of the Solubility of Hydrogen
in NaK at Elevated Temperatures

An apparatus was designed in which hydrogen can be caused to dissolve in NaK, and the amount of dissolved hydrogen can be determined both by pressure measurements at temperature and by recovery and determination of the hydrogen. The NaK will be contained in a welded capsule of metal permeable to hydrogen at elevated temperature, possibly Armco iron or nickel. The capsule will be placed in a Vycor container which will be placed in a furnace and connected to a gas feed system for measuring the amount of hydrogen passed into the system. Dead space will be minimized.

The system will be closed off at selected equilibrium conditions and, after cooling, the hydrogen in the Vycor envelope will be recovered. The envelope and capsule will be transferred to the glove box, and the capsule will be punctured in an apparatus permitting recovery of the hydrogen in it. The capsule containing NaK and solid hydride will be submitted for analysis for hydrogen, oxygen, etc. The system will be calibrated and appropriate blank determinations will be made so that the amount of hydrogen dissolved in the NaK can be determined.

As indicated in discussions above, the steady-state hydrogen pressures in the SNAP-8 system are expected to be well below one atmosphere. Consequently the determination of hydrogen solubility will be conducted at pressures below one atmosphere and down to a few mm Hg. The measurements could be carried out at temperatures as low as 500°F. Such conditions should encounter hydride precipitation and permit its characterization. Meaningful extrapolation of precipitation conditions to even lower temperatures should be possible. Devices for the accomplishment of these experiments are in process of fabrication and assembly.

A system for the purification and supply of NaK for the above experiments is being fabricated. The NaK tank presently includes a cold trap and filters. A hot trap will be added later if indicated to be necessary. An inert gas blanket will be maintained on the NaK in the storage system

at all times. The system will allow complete drainage of the NaK. The NaK will be removed from the supply tank through a 20- μ sintered type 347 stainless steel filter and then purified by filtration at low temperature through a 5- μ "Purolator" filter of sintered type 347 stainless steel. Purified NaK from this system may be withdrawn through outlets fixed in a free-arm glove box filled with dry inert gas. Waste NaK will be removed under oil for disposal.

Various equipment can be loaded with NaK in the glove box, which is equipped with a vacuum port. The glove box is provided with a cable for a Heliarc electric welding torch which will be used for closing the iron capsules containing NaK. Inert gas in the box will be dried by recirculation through a liquid-nitrogen trap.

A hydrogen purifier is available that can produce 100 cm³ (STP)/min. at 10 psia. This will serve as a hydrogen source for both the diffusion studies and the NaK-hydrogen equilibrium studies. The gas will be purified by diffusing through a palladium-silver alloy at elevated temperatures.

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