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RESEARCH REPORTS

2630

ORNL-2630

Metallurgy and Ceramics

TID-4500 (14th ed.)

LIQUID METAL LOOPS IRRADIATED
IN THE ORNL GRAPHITE REACTOR AND THE LITR

W. W. Parkinson and O. Sisman

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Contract No. W-7405-eng-26

SOLID STATE DIVISION

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IN THE ORNL GRAPHITE REACTOR AND THE LITR

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JUN 1 1959

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W. W. Parkinson

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ABSTRACT

Liquid alkali metals were circulated in a series of structural alloy loops under reactor radiation at high temperature. The first of these loops was a lithium-stainless steel (type 316) system, and the remainder were sodium-Inconel.

The lithium-stainless steel loop operated at 1000°F with a flow of 2 fps for 160 hr, receiving a fast neutron dose of 3×10^{16} neutrons/cm². Three of the sodium loops operated at 1500°F in the in-pile section and 1100° at the pump cell, with flows of 1 to 1.5 fps; the duration of operation ranged from 101 to 235 hr to give fast neutron exposures of 1.8 to 4×10^{16} neutrons/cm². The fourth sodium loop incorporated a stress-corrosion specimen stressed at 1500 psi. This loop operated between 1070 and 1270°F for 159 hr at a flow of 1 fps through the specimen. The dose ranged up to 5×10^{18} thermal neutrons/cm² and 2×10^{18} fast neutrons/cm² at the in-pile end of the specimen.

In all loops, corrosion, if any, was less than 0.0005 in. No effect of radiation other than radioactivation was found. Mass transfer and metallurgical processes attributable to the operating temperatures were observed.

SUMMARY

The compatibility of molten alkali metals with structural alloys was investigated under reactor radiation at high temperatures. These studies were performed under dynamic conditions by circulating molten sodium or lithium through a "loop" of Inconel or stainless steel tubing inserted in a reactor. After operation and irradiation the tubing was sectioned for metallographic examination and comparison of irradiated, unirradiated, and as-received specimens.

The first loop was stainless steel, type 316, and circulated lithium at 2 fps and 1000°F. A corollary objective, along with the compatibility study, was the measurement of bremsstrahlung radiation from the 0.8-sec Li⁸ produced in the lithium stream. This loop operated in Hole 58N of the Graphite Reactor for 160 hr. The total thermal neutron exposure at the center of the lithium stream was 7×10^{16} neutrons/cm², and the exposure to neutrons of energy $E > 0.5$ Mev was 3×10^{16} neutrons/cm².

Measurement of bremsstrahlung radiation proved to be impractical because of the high background level at the mouth of the reactor hole. Postirradiation examination disclosed that the operating

temperatures had produced an increase in grain size and a decrease in hardness in the metal of the loop tubing. There was very little evidence of corrosion or of any effect of radiation on any of the processes occurring in the system (except for radioactivation, of course).

The remaining loops were sodium-Inconel systems, three of which were operated in the Graphite Reactor and the last, in the LITR. The three loops in the Graphite Reactor operated with the in-pile portion at 1500°F and the pump cell at 1100°. Flow was 1 to 1.5 fps. Operating periods exclusive of bench testing were 165, 101, and 235 hr, respectively, for the three loops. These periods gave neutron doses of 3×10^{17} thermal neutrons/cm² and 3×10^{16} fast neutrons/cm² for the first loop; 1.8×10^{17} and 1.8×10^{16} for the second; and 4×10^{17} thermal and 4×10^{16} fast for the third loop. In these loops, as in the lithium-stainless steel loop, there was little or no evidence of corrosion or of a radiation effect. Corrosive penetration was less than 0.0005 inch. Light deposits of mass-transferred metal were found, and in the first of the loops they were sufficient to obstruct flow at a constriction in the tubing. Carbide precipitation was observed in

the Inconel, but the precipitate was depleted to a depth of 0.005 in. from the sodium interface. In this depleted region marked grain growth took place in the loop, which was constructed of initially fine-grained Inconel.

Weight-change specimens of Inconel and stainless steels were included in one of the loops. Inconel in the hot zone lost about 2% in 235 hr, while the stainless steels (300 series) gained about this amount in both the hot and cold zones.

The LITR loop, which incorporated a stress-corrosion specimen, operated at 1070°F for 110 hr and at 1270° for 49 hr. The stress on the specimen was 1500 psi, and flow through it was 1 fps. The thermal neutron exposure at the in-pile end was 5×10^{18} neutrons/cm², and the fast neutron dose was about 2×10^{18} . No depletion of carbide precipitate was noted in the Inconel of this loop. In the stressed section of the specimen the precipitate had formed along crystallographic planes in a Widmanstaetten-type structure. Again corrosive penetration was less than 0.0005 in., but slight erosion was noted in the specimen opposite the sodium inlet tube.

1. INTRODUCTION

Reactors for motive power place a premium on high power density and therefore on efficient heat transfer media. Liquid metals possess superlative heat transfer properties, but their compatibility with structural materials at desirable temperatures is not adequately known. To solve this problem investigations of the corrosion of structural materials in alkali metals under both static and dynamic conditions have been performed in recent years at high temperatures.¹⁻³ The series of experiments reported here was carried out to investigate the effect of nuclear bombardment on the corrosion of high-temperature alloys in flowing sodium or lithium. These experiments were of an exploratory nature, and precise control of all the variables involved was not feasible in view of

the need for engineering data. Similar dynamic, irradiated systems have been studied at somewhat lower temperatures by Bruggeman⁴ and Haag and Kendall⁵ to estimate the transport of radioactive constituents of stainless steel.

The experiments described herein were carried out by pumping molten alkali metal through a closed system, or loop, of stainless steel or Inconel⁶ tubing inserted in a nuclear reactor. The experimental apparatus consisted essentially of the tubing loop, an electromagnetic pump, a flow-meter, and a surge tank for thermal expansion and convenience in filling. Heaters were required in order to attain the temperatures of interest, and electrical controls for these and for the pump were provided.

The usual alkali metal safety problems were aggravated by radiation and induced radioactivity. Personnel protection required radiation shielding and minimal volume of liquid metal outside the reactor. Leak and fire hazards necessitated an inert gas or vacuum jacket around the in-pile parts and an off-gas system, monitored for activity, around the external components of the loop. As protection for the reactor structure, water cooling was provided in the reactor hole liner or loop jacket, and over-temperature switches were included in the heater circuits.

2. GENERAL CONSTRUCTION FEATURES

The liquid metals under consideration were lithium and sodium. The first loop was filled with lithium, which required stainless steel (type 316) for its containment, and the later loops were sodium-Inconel systems. The first loop and the last sodium-Inconel loop, which operated in a stress-corrosion study in the LITR, were unique and are described individually in specific sections below.

The selection of a size for the loop tubing was a compromise between minimal volume for safety and adequate cross section for flows up to 4 fps

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¹L. R. Kelman, W. D. Wilkinson, and F. L. Yaggee, *Resistance of Materials to Attack by Liquid Metals*, ANL-4417 (July 1950).

²E. E. Hoffman and W. D. Manly, *Corrosion Resistance of Metals and Alloys to Sodium and Lithium*, ORNL-2271 (March 19, 1957).

³*Liquid-Metals Handbook*, 2d ed., R. N. Lyon (Editor-in-Chief), GPO, Washington, 1952; 3d ed., C. B. Jackson (Editor-in-Chief), 1955.

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⁴W. H. Bruggeman, KAPL-1244, p 99 (Dec. 9, 1954) (classified).

⁵F. G. Haag and W. W. Kendall, KAPL-1338 (May 16, 1955) (classified).

⁶Inconel is a proprietary alloy of the International Nickel Company having the composition: Ni, 72%; Cr, 14 to 17%; Fe, 6 to 10%; Mn, 1.0%; Cu, 0.5%; Si, 0.5%; C, 0.15% max; S, 0.015%.

with the available pumps. The tubing was 0.225 in. OD $\times$ 0.025 in. wall, except that the lithium-stainless steel loop was 0.385 in. OD $\times$ 0.0625 in. wall for the most part. The loops extended 18 to 19 ft into Hole 58N of the Graphite Reactor. Assembly was by inert arc welding with mass-spectrometer leak testing.

A typical loop system is shown schematically in Fig. 1. The general arrangement of a typical loop system in the reactor is shown in Fig. 2, with the pump and flowmeter magnet removed for clarity. The pump was electromagnetic to eliminate bearing and seal problems. It was a laboratory model (G-E model 9159849G3), alternating current, operating at 6 kva, with the pump cell modified for high-temperature use.⁷ The flow-

meter, also electromagnetic, was a straight section of tubing through the gap of a C-shaped permanent magnet (3000 or 10,000 gauss) with electrodes welded to the tubing normal to its axis and to the magnetic field.⁸ Both the tube section and the electrode wires were of the structural alloy of the loop tubing. The surge tank was a vertical cylinder 3 in. in diameter $\times$ 5 in. high in later loops and slightly larger in earlier systems. Through the top of the tank passed two tubes, one for maintaining an inert gas or a vacuum over the liquid metal, and the other a filling tube which was closed and sealed by silver solder after the loop had been filled. An insulated electrical probe also passed through the top of the tank to indicate the depth of the liquid metal.

⁷M. E. LaVerne, *An Improved AC Electromagnetic Pump Cell*, ORNL CF-52-12-153 (Dec. 7, 1952).

⁸R. M. Carroll, *A Simple Electromagnetic Flowmeter for Liquid Metals*, ORNL-1461 (March 4, 1953).

Fig. 1. Liquid Metal Loop System for the Graphite Reactor.

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Fig. 2. Loop in the Graphite Reactor. Flowmeter magnet and pump removed.

Specimens from most of the loop systems were restricted to transverse sections from the loop tubing for metallographic examination, since experience had shown that this type of examination was adequate. Sections were cut from the in-pile extremity of the loop for irradiated specimens and from the exterior part for unirradiated samples. In the later loops pieces were cut from the part within the outer region of the reactor shield for high-temperature unirradiated specimens.

The fourth Graphite Reactor loop and the LITR loop contained a bypass purification system to remove sodium oxide, which has been found to accelerate corrosion and mass transfer in liquid metal systems. The purification system was connected near the inlet and outlet of the pump and comprised a cooling-settling tank and a filter of sintered stainless steel. The solubility of the oxide in sodium varies strongly with temperature,⁹ so such a system can be quite effective in removing oxygen. After the bench runs this system was cut from the loop and the connecting tubes were crimped closed and sealed with hard solder.

The heating requirements were severe for the in-pile part of the loop, which operated at high temperature within a cooled jacket. This portion of the loop was heated by tubular, metal-sheathed resistance heaters (Calrod type) containing powdered oxide insulation. The heaters (20 kw, total) were attached to the loop tubing in the manner indicated in Fig. 2 by wrapping with stainless steel tape. The remainder of the liquid metal system was heated by tracing with Nichrome resistance wire insulated with ceramic beads, except for the lithium-stainless steel loop. This loop was traced throughout with metal-sheathed tubular heaters for isothermal operation. The exterior portion of the later loops was allowed to operate at lower temperatures than the in-pile part, both for protection of the pump and for observation of the effect of a temperature differential in the liquid metal circuit.

The Graphite Reactor hole into which the loops were inserted was lined with a 4-in.-square aluminum tube. Around the inner surface of the hole liner, water cooling lines were spiraled, and at its center was a 2-in. stainless steel tube. This tube served as a vacuum jacket

for the in-pile portion of the loop. The annulus between the vacuum jacket and the hole liner was filled with diatomaceous earth insulation. The external end of the vacuum jacket was closed by a copper-gasketed flange through which passed the loop tubing, heater ends, and thermocouple leads. The loop and heater ends were sealed to the flange by welding, while the thermocouple leads were conducted, through a tube, to Kovar seals in a sufficiently cool position.

Instrumentation and controls for the loop included bead-insulated Chromel-Alumel thermocouples for temperature measurements in the loop system and on the cooling water lines as well. Temperatures were recorded on a multipoint recording potentiometer. Additional thermocouples were connected to galvanometer-type indicators which contained over-temperature safety switches for the heaters. Control of the heaters and the electromagnetic pump was manual through autotransformers. Ionization chambers were located to measure the radioactivity of the liquid metal stream and of the off-gas line from the shield compartment. A conventional vacuum and inert gas system was connected to the jacket and to the surge tank.

Radiation shielding consisted of stacked lead bricks forming a compartment enclosing the external parts of the loop. Under this section was a stainless steel tray containing sodium carbonate as a fire extinguisher. A container of paraffin at the rear of the shielded compartment and sheets of borated plastic at the sides provided neutron shielding. The arrangement of apparatus at the pile face can be seen in Fig. 3, a photograph taken with a portion of the lead shielding removed.

3. CONSTRUCTION FEATURES OF THE LITHIUM-STAINLESS STEEL LOOP

The lithium-stainless steel loop, the first of the liquid metal loops, had dual objectives: the measurement of radiation arising from the stream of neutron-bombarded lithium and, additionally, the investigation of the effect of radiation on corrosion. Measurement of the radiation from the 0.8-sec Li^8 in the lithium stream required three calibrated ionization chambers spaced at known distances around the external parts of the loop. Measurement and control of the velocity of the liquid metal stream between almost zero and

⁹B. G. Voorhees and W. H. Bruggeman, *Interim Report on Cold Trap Investigation*, KAPL-612 (Oct. 1, 1951).

Fig. 3. Loop Parts at Reactor Face.

10 fps were also necessary. Figure 4 indicates the positions of the ionization chambers around the loop. To attain the desired linear velocities the external section of the loop was made of smaller tubing (0.223 in. OD $\times$ 0.0625 in. wall) than the in-pile part (0.385 in. OD $\times$ 0.0625 in. wall). As was mentioned earlier, this loop, which was designed for isothermal operation, had high-wattage Calrod-type heaters throughout, and the part outside the reactor was insulated with high-temperature pipe insulation.

4. CONSTRUCTION FEATURES OF THE LITR LOOP

The last loop of the series was designed to study stress corrosion and creep in a dynamic sodium-Inconel system under the higher flux of the LITR. Modifications of the Graphite Reactor loop design were required for the application

of a known stress and for the inclusion of shielding within the reactor hole. More elaborate temperature controls were necessary also for the creep investigation.

To provide a specimen of precise and uniform wall dimensions, a tubular piece (Fig. 5) with enlarged ends was machined from a billet of Inconel. Spiraled tubing to each end permitted circulation of sodium through the specimen piece and also isolated it mechanically from the loop tubing for stressing. For convenience in assembly, the loop tubing was joined to one end of the specimen by nickel-palladium braze, while the connection at the other end was welded. This brazing operation subjected the specimen and spiral tubing to hydrogen firing at 2380°F for 10 min. The load was applied to the specimen by clamps at each end. The clamp at the in-pile end was attached to the inner jacket around the

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Fig. 4. Location of Ionization Chambers in Lithium-Stainless Steel Loop.

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Fig. 5. Specimen for Stress-Corrosion Loop.

loop, and the clamp at the other end was loaded by a flexible cable through pulleys to a dead weight outside the reactor. The arrangement of parts around the specimen is pictured in Fig. 6. An S-shaped Bourdon tube with an electrical contact at the free end was calibrated at operating temperature to serve as an extensometer. This Bourdon tube is shown attached with its pressurizing line to one end of the specimen, while

the matching electrical contact is attached by adjustable screws to the other end.

The general layout of the loop and its jackets is shown in Fig. 7. There were two surge tanks in this loop, one in the inlet and one in the outlet tube near the specimen. These tanks were horizontal half cylinders with a hole in the center for a cylindrical, wire-wound ceramic heater, through which passed the loading cable.

Fig. 6. Specimen Section of Stress-Corrosion Loop.

From these tanks the loop tubing ran through spiral holes in a shield plug to the pump and flowmeter, which were outside the reactor shield. The pump and flowmeter were identical with those of the Graphite Reactor loops. A bypass purification system was used during the preliminary bench run in the same manner as described for the Graphite Reactor loops.

The heaters around the specimen and its coils of connecting tubing were of the wire-wound, ceramic clamshell type. The heater in the hole between the surge tanks has been described, and the rest of the loop system was traced with ceramic-beaded Nichrome resistance wire.

Jackets around the loop system provided an inert atmosphere, and also vacuum insulation and water cooling where these were needed. Appropriate jackets, tubing, and seals formed an inert-atmosphere enclosure which was continuous through the holes in the shield plug,

around all sodium-containing parts of the loop system. Surrounding 3 ft of the in-pile end (the specimen and surge-tank section) of the helium jacket was a vacuum jacket for thermal insulation and leak protection. The specimen loading rod passed through two bellows in the outer and inner walls of the vacuum jacket in an arrangement which prevented atmospheric pressure from affecting the loading system. The helium and vacuum jackets were of stainless steel and were attached to the shield plug to furnish mechanical support to the specimen-surge-tank portion of the loop.

The jacket and shield-plug assembly was inserted in a removable hole-liner in Hole HB-2 of the LITR. This hole-liner was of double-walled construction. The in-pile end for $2\frac{1}{2}$ ft comprised an aluminum water jacket for circulation of cooling water. The remainder of the hole-liner

Fig. 7. Liquid Metal Stress-Corrosion Loop for the LITR.

was steel, with the space between walls filled with concrete for shielding.

Instrumentation for this loop differed from the previous systems in having automatic temperature control for the irradiated section and safety circuits which would effectuate an emergency shutdown of the reactor. The clamshell heaters around the specimen and connecting tubing were controlled by a Leeds & Northrup DAT-type controller, which varied the duration of the heating period according to the demands of the system. Excessive temperatures, as indicated by thermocouples at several points on the loop and on the water jacket, not only would shut off the heaters and sound an alarm, but at slightly higher temperatures would give an emergency setback of reactor power. Jacket water flow and jacket vacuum were other quantities monitored to produce, respectively, a reactor setback and an alarm. The radiation level of the outlet line

from the helium jacket was measured by an ionization chamber which would produce an emergency shutdown of the reactor at a preset level. Figure 8 is a general view of the control panels.

The shield plug of this loop eliminated the need for neutron shielding outside the reactor. The radioactivity of the sodium stream required 8 in. of stacked lead brick surrounding the water-cooled stainless steel box around the pump and flowmeter (Fig. 8).

5. OPERATING HISTORY OF THE VARIOUS LOOPS

The operating conditions and histories of the loop experiments are summarized in Table 1. Following assembly, flushing with alcohol, leak testing, and attachment of thermocouples and heaters, the loop was filled with molten alkali metal from the charging apparatus shown in

Fig. 8. Control Panels and Shield for LITR Loop.

Table 1. Operating Conditions and Results of Irradiated Liquid Metals Loops

Loop No.	Liquid Metal	Loop Material	Bench Test		Irradiation Conditions								Metallography and Corrosion	Remarks
			Temperature (°F)	Duration (hr)	Temperature (°F)		Duration (hr)	Velocity (fps)	Flux (neutrons·cm ⁻² ·sec ⁻¹)		Exposure (neutrons·cm ⁻²)			
					External	In-Pile			Thermal	Fast	Thermal	Fast		
1	Lithium	316 SS	1000		1000	1000	160	~2	1.3×10^{11}	5×10^{10}	7×10^{16}	3×10^{16}	Penetration <0.5 mil; grain growth	No effect of irradiation
1	Sodium	Inconel	1000-1200	40	1100	1500	115	1 } 1 }	5×10^{11}	5×10^{10}	3×10^{17}	3×10^{16}	Intergranular penetration, <0.5 mil; unirradiated, marked carbide precipitation in grain boundary; irradiated, carbide precipitation in grains	Precipitation differences due to differences in temperature; precipitate reduced near sodium interface; mass transfer noted
					1000	1000	50							
2	Sodium	Inconel	1000-1200	40	1000	1000	6	1-1.5 } 1-1.5 }	5×10^{11}	5×10^{10}	2×10^{17}	2×10^{16}	Integranular penetration, <0.5 mil; unirradiated, precipitation in grains; irradiated, more precipitation in grains	Precipitate in irradiated part markedly reduced for 5 mils near sodium interface
					1100	1500	95							
3	Sodium	Inconel	1000	84	1100	1500	235	1	5×10^{11}	5×10^{10}	4×10^{17}	4×10^{16}	Intergranular penetration, ~0.5 mil; unirradiated and irradiated, precipitation in grains and grain growth	Precipitate depleted for 5 mils near sodium interface, all parts; all specimen plates gained ~2% except irradiated Inconel, which lost ~2%; mass transfer noted
4	Sodium	Inconel	900-1100	150	1000	1070	110	1 } 1 }	8×10^{12}	3×10^{12}	5×10^{18}	2×10^{18}	Intergranular penetration, <0.5 mil; irradiated, stressed specimen showed precipitate formed in grains along crystallographic planes	No depletion of precipitate by sodium; sodium leached palladium from nickel-palladium braze to produce leak
					1000	1280	49							

Fig. 9. The loop was first evacuated, the liquid metal was forced by gas pressure from the melt tank to the metering tank, and finally the desired volume was transferred from the metering tank to the surge tank of the loop.

After being filled, the loops were bench-tested in the laboratory by circulating the liquid metal at the desired velocities (1 to 2 fps) at about 1000°F continuously for a period of 40 to 100 hr. Following a successful bench test the loop was cooled carefully until the liquid metal had solidified.

The loop was then inserted in the hole-liner in the reactor hole. Melting of the alkali metal was done slowly, beginning at the surge tank to avoid tube rupture from expansion during melting. Operation of the heaters, pump, and ancillary apparatus was tested, and radiation shielding was erected. The loop was heated to operating temperature, and the reactor was started.

Fig. 9. Liquid Metal Charging Apparatus.

5.1. The Lithium-Stainless Steel Loop

Because lithium was known to leach nickel from stainless steel to a greater extent than other alkali metals, this loop was operated for one week at 1000°F. The radiation measurements, for which temperature was unimportant, were made during this period. During most of this time the velocity was about 2 fps in the in-pile section, but during measurement of the radiation the velocity was varied from almost zero to 10 fps for short periods. After this phase of operation the temperature was raised to 1500°F. After less than an hour the connections between heaters burned out, and circulation stopped. Total in-pile operating time was 160 hr at temperature, with 150 hr of this period under irradiation.

It was estimated that the thermal neutron flux in Hole 58N, empty, was 5×10^{11} neutrons·cm⁻²·sec⁻¹. The attenuation of the flux by the lithium was measured by the activation of a cobalt foil inserted in the center of a lithium-filled stainless steel tube of the same diameter as the loop and irradiated in the same position. The flux near the center of the loop tubing, as measured in this manner, was 1.3×10^{11} neutrons·cm⁻²·sec⁻¹, and the total thermal neutron exposure at this point was about 7×10^{16} neutrons/cm². The epithermal neutron flux was estimated at 5×10^{11} neutrons·cm⁻²·sec⁻¹, and the exposure was 3×10^{17} neutrons/cm², while the flux with an energy $E > 0.5$ Mev was one-tenth of this value.

The radiation measurements were made at various flow rates with ionization chambers which were calibrated with Co⁶⁰ sources for geometry and with lower-energy sources for energy response. The variation of the radiation intensity with velocity and with position around the loop was in agreement with a half life of 0.83 sec for the lithium. Iron and copper sheets of known thickness were inserted between loop tubing and chamber for a rough determination of the energy of the loop radiation. The results of these measurements are plotted for counter No. 2 in Fig. 10. Calculations showed that not all of the beta particles were stopped when sufficient absorber had been inserted to reduce the radiation to the background level. The greater sensitivity of the chamber to beta particles as compared with gamma photons made it impractical to attempt to reduce the

Fig. 10. Plot of Radiation vs Absorber Thickness for Lithium-Stainless Steel Loop.

background to a level that would permit measurements with the betas excluded. This difference in sensitivity also made it impossible to analyze the absorption curves into beta and bremsstrahlung components.

The radioactivity of the lithium stream did not increase perceptibly during the operating period, and the decay time of the radiation remained unchanged. This indicated that corrosion of the loop tubing was not extensive enough to introduce activated constituents into the liquid metal.

After operation had stopped and the liquid metal had been allowed to freeze, the loop was withdrawn from the reactor shield until the radioactive section was reached. The non-radioactive portion was cut off, and the radioactive section was withdrawn into a thick-walled steel pipe. Tube sections were cut from the loop for metallographic examination and for analysis of the alkali metal.

5.2. The First Sodium-Inconel Loop

The loop was filled, bench tested, and inserted in the reactor as described above. The loop was operated for 115 hr at a velocity of 1 fps; the temperature was 1500°F for the in-pile section and 1100° at the pump cell. Toward the end of this period flow stopped. Flow was restored by alternately evacuating and pressurizing the surge tank with the loop at operating temperature. Operation was continued for 50 hr with the in-pile section at 1000°F and the pump cell slightly cooler. Circulation stopped again and could not be restored.

The thermal neutron dose for this loop was about 3×10^{17} neutrons/cm². The epithermal neutron dose was approximately equal to this, while the exposure to neutrons of energy greater than 0.5 Mev was about 3×10^{16} neutrons/cm².

Removal of this loop differed from the handling of the lithium-stainless steel loop because of the 15-hr half life of the activated sodium. Two weeks' decay period was allowed before removal of the portion of the loop outside the active lattice of the reactor. The part which had been inside the lattice was permitted to decay for an additional two weeks in that part of the reactor hole within the reactor shield. This section of the loop was then withdrawn into a pipe and handled in the same manner as the earlier loop.

Dissolution of the sodium from the loop tubing revealed deposits of crystalline, metallic powder at a constriction caused by weld penetration at a butt joint near the surge tank. Analysis of the powder (both spectrographic and x-ray) showed the material to be mostly nickel, with less chromium and iron than Inconel. It was concluded that the obstruction consisted of mass-transferred crystals or possibly a metallic reduction product of weld scale. The usual tube sections were cut for metallographic examination.

5.3. The Second Sodium-Inconel Loop

During bench test operation of this loop a very small leak was discovered at the junction of the loop tubing with the pump cell. The leak was sealed successfully by welding, and bench testing was resumed. Upon completion of bench testing the loop was installed in the Graphite Reactor in the same manner as the previous loops.

This loop was operated for 6 hr at 1000°F, then for 95 hr with the in-pile part at 1500°. During the latter period the pump cell ran at about 1100°F. The flow velocity during the whole operation was about 1 to 1.5 fps. At the end of this period a small leak developed, again at the junction of the loop tubing and the pump cell. This leak was detected by the monitor on the shield off-gas line as an increase in radioactivity.

The thermal neutron exposure for this loop was 1.8×10^{17} neutrons/cm², and the epithermal dose was about the same. The dose for neutrons of $E > 0.5$ Mev was about 1.8×10^{16} neutrons/cm². This loop was removed from the reactor and sectioned for examination by the same procedure as the previous loop.

5.4. The Third Sodium-Inconel Loop

The third sodium-Inconel loop, the last loop in the Graphite Reactor, incorporated several modifications, among which was the bypass purification system described earlier. Other changes were short spiraled sections of loop tubing just inside the jacket closure flange, and specimen chambers consisting of 3-in. sections of 0.312-in.-OD $\times$ 0.025-in.-wall tubing. The spiraled sections relieved stresses due to the difference in temperature between the incoming and outgoing sodium streams. There were two specimen

chambers, one at the in-pile end and the other at the reactor shield face, both in the tube run leading from the reactor center. Specimens in the form of rectangular plates of Inconel and three 300-series stainless steels were cleaned, weighed, and inserted in each of the chambers prior to assembly of the loop.

The loop was filled in the same manner as the earlier loops. The bench test procedure was altered somewhat to maintain suitable conditions in the bypass purification system. Ten to twenty per cent of the flow (as indicated by a bypass flowmeter) was diverted through the purification system. The temperature at the outlet of the cooling-settling tank was controlled at 250 to 400°F by varying heater and pump power. After 84 hr of bench operation the loop was allowed to cool, the bypass system was removed, and the loop was inserted in the reactor.

Operating conditions were the usual temperatures, 1500°F in-pile and 1100° at the pump cell, and a flow of 1 fps. After 218 hr of reactor operation the heaters failed. The loop was removed from the reactor, and the heaters were replaced. The loop was again inserted in the reactor and was operated for 17 hr; at the end of this period a leak in the in-pile section into the vacuum jacket was detected.

At the usual neutron fluxes in Hole 58N, this loop received a thermal neutron dose of 4.2×10^{17} neutrons/cm² and an approximately equal exposure to epithermal neutrons. The exposure to neutrons of $E > 0.5$ Mev was about 4×10^{16} neutrons/cm².

The loop was removed as the previous loops had been. The leak into the vacuum jacket necessitated the removal of this jacket and the water-cooled hole-liner from the reactor. The loop tubing was sectioned for postirradiation examination and for removal of the specimen plates.

5.5. The Stress-Corrosion Loop in the LITR

The loop was charged with sodium by the same procedure as earlier loops. The loop was bench tested at 900 to 1100°F for about 150 hr. During bench operation the shells for the helium and vacuum jackets were clamped in place, but in order to permit inspection of the loop at the conclusion of the test they were not welded. After

bench operation the bypass purification system was removed, as was described for the previous loop. The helium and vacuum jackets were welded in place and leak tested. The loop, supported by its shield plug, was inserted in its hole liner in Hole HB-2 of the LITR.

The loop and reactor were started as usual with the flow at 1 fps and the stress on the specimen 1500 psi. The temperature of the specimen and the in-pile section was held down to 1070°F because concurrent tests of the nickel-palladium braze, with which the loop tubing was joined to one end of the specimen, showed the braze to be susceptible to sodium corrosion. Part of the sample heater failed after 16 hr of operation, but the temperature was maintained by increasing power to other heaters. The electrical contacts (tungsten) of the extensometer oxidized early in the operation to the extent that closure was uncertain. No significant measurements were made of the extension of the specimen.

After a total of 110 hr at 1070°F the temperature was increased to 1280°F. This temperature was maintained for 49 hr; at the end of this period circulation stopped. Examination of the loop after disassembly disclosed a leak at the brazed joint of the specimen; with destruction of the heater around the leak.

During operation of this loop the operating power of the LITR was 1500 kw. The neutron flux in HB-2 was estimated from the activation measurements described below in the section on postirradiation examination. The thermal neutron exposure ranged between 2 and 5×10^{18} neutrons/cm² over the specimen region. The fast neutron dose ($E > 0.6$ Mev) was about 2×10^{18} neutrons/cm² at the in-pile end of the specimen.

The radioactivity of the loop parts outside the reactor was allowed to decay for one week, and these parts were cut off and removed as before. The specimen section on its mounting plug was then withdrawn into a horizontal lead shield because of the activity of the specimen and its stainless steel jackets. After two months of decay the specimen section and its jackets were separated from the shield plug and were dissected by remote handling in a hot cell.

6. POSTIRRADIATION EXAMINATION OF THE LOOPS

6.1. The Lithium-Stainless Steel Loop

Metallographic examination of transverse sections of tubing from both the irradiated and unirradiated parts showed a definite increase in grain size as compared with an as-received control specimen. These specimens are shown in Figs. 11-13. This grain growth was caused by the heat treatment during operation and apparently was not a consequence of radiation or exposure to lithium. There was no intergranular penetration or other corrosive attack, although the surface roughness showed the possibility of light grain-boundary etching by the lithium. The operating temperatures had produced an annealing effect in the loop samples, indicated by the following hardness values:

Specimen	Vickers Hardness
Loop (irradiated)	137 $\pm$ 7
Loop (unirradiated)	132 $\pm$ 2
As-received control	169 $\pm$ 5

The radioactivity of the lithium metal was too low to measure with accuracy after decay of the short-lived isotopes; but the long-lived activity did provide at least an indication of removal of metal from the activated tube walls. If the following assumptions are made: long-lived activity was Co^{60} , cobalt concentration was 0.25% in the nickel of the steel, no Co^{60} was deposited in the cooler parts of the loop, and there was no preferential leaching of cobalt from the walls; then the dissolution of the tube wall was of the order of $1\frac{1}{2} \text{ mg}\cdot\text{cm}^{-2}\cdot\text{month}^{-1}$. This is consistent with the predictions of Epstein and Weber¹⁰ if the unirradiated solution rate of stainless steel in sodium is similar to that of iron ($\sim 0.8 \text{ mg}\cdot\text{cm}^{-2}\cdot\text{month}^{-1}$).

6.2. The First Sodium-Inconel Loop

Figures 14 and 15 show, respectively, an as-received control from the piece of tubing used to fabricate the exterior (out-of-pile) part of the

¹⁰L. F. Epstein and C. E. Weber, TID-70, p 36 (1951) (classified).

Fig. 11. Type 316 Stainless Steel. As-received control for lithium loop. Etchant: glyceria regia. 250X.

Fig. 12. Type 316 Stainless Steel. In lithium at 1000°F for 160 hr. Unirradiated. Etchant: glyceria regia. 250X.

Fig. 13. Type 316 Stainless Steel. In lithium at 1000°F for 160 hr. Irradiation, 3×10^{16} fast neutrons/cm². Etchant: glyceria regia. 250X.

Fig. 14. Inconel, Loop 1. As-received control for unirradiated specimen. Etchant: aqua regia. 250X.

Fig. 15. Inconel, Loop 1. In sodium at 1100°F for 205 hr. Unirradiated. Etchant: aqua regia. 250X.

loop, and a specimen from this section after operation of the loop. Figures 16 and 17 show the as-received control for the in-pile part of the loop, and a specimen from this irradiated section.

In this loop there was little change in grain size in the loop tubing. Again there was little or no evidence of corrosion; surface roughness and intergranular penetration (<0.0005 in.) appear in both the as-received and the loop specimens. In the out-of-pile specimen (Fig. 15) there is a marked intergranular precipitate, probably carbides. The irradiated specimen (Fig. 17) does not show such marked precipitation, and in this specimen the precipitate is intergranular. The operating temperatures of the loop are somewhat below the region where the usual quantities of carbon in Inconel are soluble. Consequently it is thought that the appearance of the precipitates in these specimens was caused by the temperature conditions. In all loop specimens the precipitate was noticeably reduced near the surface in contact with sodium.

To investigate the possibility that a difference in carbon content was responsible for the difference between the precipitates in the two loop specimens, the as-received controls were heat-treated. Each control was cut in half, and one half of each was heated to 1000°F for 120 hr, while the other piece of each control was heated to 1500° for the same period. Both specimens showed the same precipitation behavior at a given temperature, and therefore impurity content is eliminated as a cause of the difference between the loop specimens. On the other hand, the low-temperature heat treatment did not produce as marked a concentration of precipitate at the grain boundaries as the out-of-pile loop specimen showed. Furthermore, both control pieces heat-treated at 1500°F showed more precipitate than did the irradiated loop specimen.

6.3. The Second Sodium-Inconel Loop

From this loop, in addition to the usual in-pile and out-of-pile cuts, specimens were taken from the loop tubing within the reactor shield at a

Fig. 16. Inconel, Loop 1. As-received control for irradiated specimen. Etchant: aqua regia. 250X.

Fig. 17. Inconel, Loop 1. In sodium at 1500°F for 205 hr. Irradiation, 3×10^{16} fast neutrons/cm². Etchant: aqua regia. 250X.

point 6 ft from the reactor face. These provided specimens which were not irradiated but which were exposed to liquid metal at 1500°F. The as-received control for the out-of-pile piece is pictured in Fig. 18, and the out-of-pile specimen in Fig. 19. The as-received control for the high-temperature (1500°F) specimens is shown in Fig. 20, and the unirradiated and irradiated specimens are shown in Figs. 21 and 22, respectively.

Surface roughness was less in both the control and loop specimens than in the previous loop. As in the previous loop, there was no evidence of corrosion, and penetration was less than 0.0005 in. Again there was no change in the grain size in the loop specimens, and a precipitate appeared in the loop specimens. On the other hand, the intergranular precipitate was not as abundant as the intragranular, and the out-of-pile (1000°F) specimen showed less precipitate than the irradiated and unirradiated high-temperature pieces. As in the previous loop, the precipitate was depleted for a depth of 0.005 in. from the sodium interface.

6.4. The Third Sodium-Inconel Loop

Deposits of metallic, crystalline powder were found at the unirradiated sample chamber, similar to the obstructions responsible for the stoppage of the first sodium loop. As in the case of the earlier loop, the deposit was much richer in nickel than Inconel (analysis of deposit: 86% Ni, 12% Cr, 1% Fe). Radioactivity in the exterior part of the loop was very slight and was non-uniform. One small section showed greater specific activity than the mass-transferred crystals, possibly as a result of the transport of discrete particles from the irradiated section.

Metallographic specimens were examined from the same locations as in the earlier loop and also from the large tube forming the in-pile U-bend and sample chamber. As-received control samples for each piece from the loop were also examined. The heat treatment of the tubing for the loop prior to receipt had been such that the controls (Fig. 23, out-of-pile, and Fig. 24, high-temperature region) showed intragranular carbide precipitate

Fig. 18. Inconel, Loop 2. As-received control for unirradiated specimen. Etchant: aqua regia. 250X.

Fig. 19. Inconel, Loop 2. In sodium at 1100°F for 141 hr. Unirradiated. Etchant: aqua regia. 250X.

Fig. 20. Inconel, Loop 2. As-received control for high-temperature parts. Etchant: aqua regia. 250X.

Fig. 21. Inconel, Loop 2. In sodium at 1500°F for 141 hr. Unirradiated. Etchant: aqua regia. 250X.

Fig. 22. Inconel, Loop 2. In sodium at 1500°F for 141 hr. Irradiation, 2×10^{16} fast neutrons/cm². Etchant; aqua regia. 250X.

Fig. 23. Inconel, Loop 3. As-received control for unirradiated specimen. Etched electrolytically with oxalic acid. 200X.

Fig. 24. Inconel, Loop 3. As-received control for high-temperature parts. Etched electrolytically with oxalic acid. 200X.

and fine grain structure. This carbide precipitate had been leached out by the sodium to a depth of 0.006 to 0.008 in. The zone near the sodium interface which had been depleted in carbides underwent considerable grain growth, in both the unirradiated (Fig. 25) and the irradiated specimens (Fig. 26).

The unirradiated high-temperature specimen from within the reactor shield showed anomalous behavior, as is seen in Fig. 27. There was abundant precipitate, uniformly distributed, and a fine, ill-defined grain structure. This specimen was from the same length of tubing as the irradiated specimen and its as-received control. Additional specimens from this section were studied, and the zone of anomalous structure was found to extend about a foot. The evidence indicated that this section of loop tubing ran at a lower temperature than the rest of the loop, probably because the unirradiated sample chamber, which was contiguous, held the tubular heater away from the loop tubing.

As in the earlier loops there was an absence of evidence of corrosion. The inner surface of the control and the loop specimens was smooth and there was no void formation. Corrosive penetration was only very slight (~ 0.0005 in.), intergranular, and confined to the high-temperature specimens.

The in-pile U-bend was found to contain the leak which terminated operation, in the form of a large ragged crack. The as-received control (Fig. 28) for this piece showed the large grain structure and absence of precipitate characterizing the Inconel used in the earlier loops. Operation of the loop had produced the precipitation, seen in Fig. 29. The precipitation was absent from the 0.006- to 0.008-in. layer in contact with sodium, which had leached out the carbon as usual. There was little or no grain growth in the already large-grain metal. The absence of penetration and void formation at the inner surface indicated that corrosion was not responsible for the leak. Intergranular surface fissures near the

Fig. 25. Inconel, Loop 3. In sodium at 1200–1500°F for 319 hr. Unirradiated. Etched electrolytically with oxalic acid. 200X.

Fig. 26. Inconel, Loop 3. In sodium at 1500°F for 319 hr. Irradiation, 4×10^{16} fast neutrons/cm². Etched electrolytically with oxalic acid. 200X.

Fig. 27. Inconel, Loop 3. In sodium at 1200–1500°F for 319 hr. Unirradiated. Etched electrolytically with oxalic acid. 200X.

Fig. 28. Inconel, Loop 3. As-received control for terminal U-bend. Etched electrolytically with oxalic acid. 200X.

Fig. 29. Inconel, Loop 3. Terminal U-bend. In sodium at 1500°F for 319 hr. Irradiation, 4×10^{16} fast neutrons/cm². Etched electrolytically with oxalic acid. 200X.

leak (Fig. 30) suggested incipient fusion and overheating. Probably the failure of the heaters occurred by arcing from the central resistance wire to the tubular sheath (and perhaps to the loop tubing), with local overheating.

The inclusion of specimen chambers containing weighed plates of Inconel and 300-series stainless steels has been discussed in the earlier section on the construction of this loop. Both chambers were in the tubing run emerging from the center of the reactor, one at the in-pile end and the other at the outer face of the pile shield. Apparently, the unirradiated sample chamber operated at slightly lower temperatures than the irradiated specimens, because the mass-transferred deposits had collected there. Probably the tubular loop heater did not make good contact with the chamber. The specimen plates underwent the

following weight changes:

Alloy	Weight Change (%)	
	Unirradiated	Irradiated
Inconel	+0.9	-1.9
310 SS	+1.9	Not recovered
316 SS	+2.1	+2.6
347 SS	+1.8	+2.4

Apparently, the chemical potentials and solution rates of the various elements in the alloys are such that there is a tendency for some element to build up in the steels. This process occurred under isothermal conditions, in contrast to the mass-transfer process commonly observed in these systems.

Fig. 30. Inconel, Loop 3. Terminal U-bend. In sodium at 1500°F for 319 hr. Irradiation, 4×10^{16} fast neutrons/cm². Etched electrolytically with oxalic acid. 500X.

6.5. The Stress-Corrosion Loop in the LITR

Sections were examined from the specimen and loop tubing at the locations indicated in Fig. 31. The etched and unetched photomicrographs (Figs. 32 and 33) of the nickel-palladium brazed joint between tube and specimen show the porosity which accounted for the leak. The radioactivity analysis of the sodium, described below, confirmed the leaching of palladium from the braze during operation.

The brazing operation, which exposed the specimen region of the loop to 2380°F for 10 min, made the effect of the operating temperatures indefinite. There was no evidence of corrosive attack; there was no void formation, and intergranular penetration, if any, was less than 0.0005 in. A very slight depression appears to have been formed in the specimen by erosion at the point of impingement of the sodium stream (Fig. 34).

A section of the stressed tubular part of the specimen is shown in Figs. 35 and 36. There does not appear to be any depletion of carbide

Fig. 31. Locations of Samples Taken from Stress-Corrosion Loop (Loop 4).

precipitate either in this area or in the enlarged end of the specimen (shown in the previous figure). The precipitate in this stressed region of the specimen is unique in being located along preferred crystallographic planes in a Widmannstaetten-type structure. That stress

Fig. 32. Inconel, Loop 4. Nickel-palladium braze joint. In sodium at 1000–1280°F for 309 hr. Irradiation, 2×10^{18} fast neutrons/cm². Etchant: glyceria regia. 75X.

Fig. 33. Inconel, Loop 4. Nickel-palladium braze joint. In sodium at 1000–1280°F for 309 hr. Irradiation, 2×10^{18} fast neutrons/cm². Unetched. 75X.

Fig. 34. Inconel, Loop 4. Specimen inlet chamber. In sodium at 1000–1280°F for 309 hr. Irradiation, 2×10^{18} fast neutrons/cm². Etchant: glyceria regia. 75X.

Fig. 35. Inconel, Loop 4. Stressed section. In sodium at 1000–1280°F for 309 hr. Irradiation, 2×10^{18} fast neutrons/cm². Etchant: glyceria regia. 200X.

Fig. 36. Inconel, Loop 4. Stressed section (same area as in Fig. 35). In sodium at 1000–1280°F for 309 hr. Irradiation, 2×10^{18} fast neutrons/cm². Etchant: glyceria regia. 400X.

played a part in the development of the structure is indicated by the random distribution of precipitate in the unstressed end section and the connecting loop tubing (Fig. 37). The possibility exists that radiation-produced imperfections agglomerated under the stress at the operating temperature to nucleate the precipitation at crystalline planes.

The loop tubing in the irradiated region (Fig. 37) shows a large grain size, similar to that found in the early loops. The structure of this tubing underwent little change from its initial condition. The precipitate is not visibly depleted at the sodium interface, either because of the lower temperature of operation or because the different cooling rate within the water-cooled jacket allowed a different precipitate to form.

A spectrographic analysis of the sodium in the loop showed the presence (20–30 ppm, each) of palladium and silver from the braze. Measurement of the radiation from the sodium indicated that the short-lived activity was due to Pd¹⁰³ (half life, 17 days). A radiochemical analysis identified

Ag¹¹⁰ as the only other radioactive element present in appreciable quantity. These isotopes masked the radiation from any constituents of Inconel in the sodium and precluded any determination of the transport of radioactive elements of the Inconel itself.

Estimates of the flux incident on the loop were made by measurements of the activation of various components of the loop. Relative measurements of the specific activity of cuttings from the stainless steel loop jackets are plotted in Fig. 38. The thermal neutron flux had a different distribution from that observed by Trice¹¹ in measurements with the hole empty, and was smaller. On the other hand the fast neutron flux should not have been attenuated substantially by the thermal neutron absorbers in the stainless steel. With the LITR power at 1500 kw, the neutron flux of energy greater than 0.6 Mev was probably about 3×10^{12} neutrons·cm⁻²·sec⁻¹.

¹¹J. B. Trice, *Solid State Quar. Prog. Rep.* Aug. 10, 1952, ORNL-1359, p 10 (classified)

Fig. 37. Inconel, Loop 4. Loop tubing. In sodium at 1000–1280°F for 309 hr. Irradiation, 2×10^{18} fast neutrons/cm². Etchant: glyceria regia. 200X.

Fig. 38. Relative Activation of Loop Metal in LITR.

7. CONCLUSIONS

Radiation was not definitely observed to affect any of the processes taking place under the operating conditions of the loops. The mass transfer of a material high in nickel from the hot to the cold zones was observed generally in the loops. Nickel is extracted in mass transfer at a greater rate than the other constituents of stainless steel or Inconel. Therefore, in the loop having samples of several alloys, Inconel (highest in nickel) showed a greater weight loss in the hot zone than the other materials.

The Inconel loops which operated at 1500°F showed a depletion of carbide precipitates in the grain structure near the sodium interface. In these depleted regions there was an increase in grain size unless the metal initially had a very large-grain structure. The stress-corrosion loop indicated that carbide precipitates will be deposited at preferred locations along crystallographic planes of Inconel under the proper conditions of stress, temperature, and impurity content.

Suggestions for future research include investigation of the possibility that neutron bombardment assists in the nucleation of carbide precipitates along crystallographic planes in stressed specimens. Investigations more likely to yield affirmative results are measurements of the transfer of radioactive elements by mass transfer. Radiation-induced corrosion might also be studied profitably at fluxes two or three orders of magnitude higher than in these experiments.

ACKNOWLEDGMENTS

The metallographic examinations were carried out by R. J. Gray and T. K. Roche of the Metallurgy Division and by M. J. Feldman and A. E. Richt of the Solid State Division. The metallographic examinations constituted almost the entire analytical portion of these experiments, and the cooperation and assistance of those named are gratefully acknowledged. Appreciation is also due M. Murray and S. A. Reynolds of the Analytical Chemistry Division, who performed spectrographic and radiochemical analyses of the sodium from the LITR loop.

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