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CORROSION AND WEAR SURVEILLANCE IN
THE PLUTONIUM RECYCLE TEST REACTOR
THROUGH DECEMBER 1967

R. B. Richman
C. W. Pollock

July 1968

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THE PLUTONIUM RECYCLE TEST REACTOR
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By

R. B. Richman

Plutonium Recycle Test Reactor Section
Engineering Services Department

C. W. Pollock

Corrosion and Coolant Chemistry Section
Chemistry Department

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ABSTRACT

Continuing surveillance of corrosion and wear at the PRTR involves visual, X-ray, and ultrasonic inspections of selected parts of every fluid system, and measurements from electrical resistance probes and coupon samples in some of the systems. All PRTR systems are in good condition, except for certain portions of the secondary system, where repairs of conventional equipment have been required, mainly due to mechanical damage.



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INTRODUCTION

A corrosion surveillance program has been in use for all Plutonium Recycle Test Reactor (PRTR) systems since 1964. This program lists monitoring locations and specifies the type and frequency of measurement or inspection. Prior to 1964, specific corrosion phenomena were investigated to determine cause for failure and to find ways to prevent a recurrence of the specific problem. In 1964, a comprehensive coordinated program was begun to monitor potential problem areas to prevent or minimize corrosion damage. Occasional occurrence of corrosion damage at PRTR, plus frequent unhappy corrosion experience at other sites, had emphasized the desirability of having a regular surveillance program to prevent severe corrosion from going unnoticed.

The initial program successfully accomplished two major objectives:

- 1) To ascertain the corrosion characteristics of all systems where uncontrolled corrosion would compromise reactor safety, significantly interfere with continuity of operation, or result in costly repairs;
- 2) To establish surveillance procedures wherever variable corrosion conditions or the potential for serious damage requires continued surveillance to assure maximum reactor safety and efficiency.

Significant changes to be made in PRTR coolant chemistry for operation during the Batch Core Experiment prompted a review and updating of the existing program near the end of 1966. Three major changes in PRTR systems are reflected in the new program:

- 1) The use of pH 7 coolant in the Primary System instead of pH 10 with LiOH addition. This was done to prevent excessive Zircaloy oxidation and hydriding from concentration of caustic in fuel assembly crevices.
- 2) A variable concentration of boric acid (in the ppm range) in the moderator for nuclear reactivity control.
- 3) Replacement of the D_2O with H_2O in the reflector.^(1,2)

This report describes the present corrosion status of the various PRTR systems and includes surveillance information developed through December 1967.

SUMMARY

Since the beginning of the present surveillance program, PRTR systems have operated with minimum overall corrosion, although on two occasions mechanical and/or corrosion damage to heat exchanger components required several days reactor outage to complete repairs.

Measurements from coupons exposed in the Primary System and in the Moderator calandria show negligible uniform corrosion of stainless steel, Zircaloy, and/or aluminum in these systems (<0.004 mils/month). Continuous measurements from electrical resistance probes in the Moderator, Reflector, Top and Bottom Shield, and Core Blanket Helium Systems also show negligible corrosion for the aluminum or carbon steel probe elements (<0.005 mils/month). Minimum corrosive conditions have been maintained in the Secondary Coolant System (including the steam generator and the deaerator) by deaeration with steam and sodium sulfite treatment and by addition of sodium phosphate and a proprietary organic material for sludge conditioning. Inspection inside the steam generator indicated that the tube bundle and the shell were in good condition. Moderate corrosion rates (0.10 to 0.19 mils/month) were measured in the Biological and Thermal Shield Systems with continuous measurements from electrical resistance probes. In

the Fuel Element Rupture Test Facility (FERTF), a low concentration of dissolved O_2 was achieved by changing the water treatment from LiOH addition to NH_4OH addition and by conditioning system oxide surfaces with short-term hydrazine addition. Corrosion of silver bushings in the ammonia treated FERTF coolant is negligible.

Leakage of D_2O from two tubes in the primary treatment heat exchanger (HX-5) resulted in several days of reactor outage to identify and seal the leaking tubes. Although it was not possible to determine the cause of these leaks, wear from rubbing against a support baffle (similar to a previous failure in this heat exchanger) is considered the most probable cause.

Several days of reactor outage were required to replace damaged elbows on three steam generator vent lines. These elbows failed due to localized (trench) corrosion and high stress levels.

DISCUSSION

The surveillance program and corrosion status are described for each system of the reactor independently, since operating conditions and physical characteristics are markedly different for the different systems. Each PRTR system is well categorized, and in those relatively few cases where the systems are connected, the system-to-system interface is sharply defined.

PRIMARY

The PRTR Primary System operates at a nominal pressure of 1070 psi and a reactor outlet temperature of 265 °C (510 °F). A special concern regarding corrosion in this stainless steel system is the possibility of increased corrosion product formation under neutral pH operation. Subsequent release of these corrosion products and deposition as crud on the Zircaloy process tubes and fuel element cladding could result in reactor

flow decreases or interference with fuel element heat transfer. An extensive crud surveillance program is being performed at the PRTR and will be described separately. The conductivity and impurity levels (especially dissolved oxygen) in the primary coolant are maintained at minimum values (usually less than detectable amounts) to minimize corrosion of stainless steel and Zircaloy. A monitoring program for the Zircaloy process tubes has been in use since initial reactor startup. Metallurgical and mechanical evaluations have been conducted on four process tubes after reactor irradiation exposures ranging from 510 to 670 MWd (~ 2.0 to 2.7×10^{21} peak nvt, > 1 MeV). Most of the Zircaloy-2 process tubes in the PRTR have now seen seven years of service; maximum exposure exceeds 800 MWd (nearly 3×10^{21} nvt, > 1 MeV). Detailed results of the tube monitoring program are reported separately.⁽³⁾ Corrosion measurements are included in development studies and in the continuing evaluation of Zircaloy fuel-cladding performance in the PRTR. Results from these studies and evaluations are reported regularly by the appropriate research departments.⁽⁴⁾ Uniform and crevice corrosion of various materials are also measured in the PRTR Corrosion Loop, a special feature of the Primary System wherein samples are exposed to full system temperature and pressure. Samples removed from this loop showed that a 300 °F non-nuclear prefilming (using pump friction heat) following system decontamination was successful in reforming the desired thin adherent oxide surfaces on stainless steel and Zircaloy. This prefilming was done to minimize the rate of subsequent adsorption of radioactive material on the piping surfaces. Additional stainless steel samples from this Primary System Corrosion Loop showed initial rates of uniform corrosion of less than 0.003 mils/month.

The stainless steel Primary System is monitored for stress corrosion by regular visual inspections of vital reactor components. These inspections are supplemented by metallurgical,

X-ray, and eddy-current examinations of selected items. Regular inspections and examinations show no evidence of any stress corrosion damage in the PRTR Primary System. Oxygen and chloride concentrations are maintained as low as possible in the primary coolant to prevent stress corrosion damage. Total chloride is maintained at less than 0.1 ppm and is routinely monitored in the coolant. Oxygen is maintained at less than 0.14 ppm and is measured in the helium cover gas and in the coolant.

In-flux measurements during previous operation of the PRTR Primary System at pH 10 showed increased oxidation of Zircaloy-2 and Zircaloy-4 samples at high-flux positions.⁽⁴⁾ A second set of Zircaloy samples was installed in the reactor core in September 1967 to provide comparative oxidation data in the neutral pH environment. First examination of this assembly is scheduled for February 1968.

The recent defects of most concern in the PRTR Primary System were two leaking tubes in the primary treatment heat exchanger, HX-5. The rate of D₂O leakage from the Primary System was determined by measurements of tritium activity in the secondary coolant. The leaking tubes were located by pressure testing, then each leaking tube was capped. A tube failed in this heat exchanger in 1963 due to wear from rubbing against a support baffle, and it is probable that wear was the cause of the present leaks. Since access to either the inside or outside of these tubes within the tube bundle is extremely difficult, it has not been possible to determine the exact cause of the present leaks. Without this information precise prediction of the frequency or extent of future leaks is not possible. Accordingly, a close surveillance is being maintained by routine measurement of tritium activity to promptly detect any leakage into the secondary coolant. Excess cooling capacity is available in HX-5, but eventual replacement of this heat exchanger is planned.

A number of Primary System equipment faults that have occurred have required reactor outage for repair. In almost all of these instances, wear or mechanical damage has caused the problem, and corrosion has been of only minor significance:

- 1) Several seals on the primary pumps have been replaced to stop leakage. Each seal examined has shown evidence of wear on the sealing faces, although exact reasons for the wear have not been resolved. The only noticeable corrosion is a thick oxide coating on one portion of the seal assembly (not a sealing surface) identified by its magnetic properties as a carbon or low-alloy steel.
- 2) The inlet angle valve to process channel 1851 was replaced in late 1966 due to severe fretting wear of the retaining nut and valve stem. The fretting started after the retaining nut vibrated loose from the valve plug (the plug and the nut were both drilled for a locking pin, but no evidence could be found that the locking pin was ever in place).

No indications of stress cracking corrosion were found, but the Stellite surfaces of the angle valve showed characteristic dendritic attack resulting from exposure to alkaline permanganate during a decontamination. However, the penetration was only 0.0005 in. deeper than that measured on a valve plug removed in early 1963. The dendritic corrosion channels provided an environment for the start of high-pressure water erosion of the Stellite back-seat surface of this valve. Capping of leak collection lines from these valves and improved tightening of the packing should minimize further erosion of the back-seat surfaces and help to compensate for the reduced effectiveness of the back-seat. While the presence of 85 of these valves on the reactor requires occasional attention to maintain good overall D₂O leakage control, leakage from any individual valve is minor.

The potential contribution of ^{60}Co to the primary coolant from the Stellite surfaces of these angle valves has been calculated under the worst conditions of wear and corrosion to be 0.02 Ci/year. Activity measurements indicate very little increase in ^{60}Co inventory in the reactor since the start of the Batch Core Experiment.

- 3) Several reactor shutdowns were required during the latter part of 1967 to correct D_2O leaks from nozzle caps and gaskets. A typical leak would not be evident at reactor startup, but would develop suddenly after a few days of operation and increase rapidly as asbestos was washed out of the gasket. The majority of leaks resulted from inadequate seating of gaskets that were slightly out of tolerance. Scratches made on the gasket seating surface during previous gasket replacements caused the other cap leaks. Reworking of damaged seating surfaces and careful inspection of all caps and gaskets before installation has corrected this leak problem.
- 4) Replacement of trim was required in April and May 1967 in several Primary System valves (including primary pump bowl vent valves and ring header level control valve) to eliminate D_2O leaks. Apparent causes of the leaks were solid particles trapped in the valve or incorrect trim sizes, which caused initial leakage with subsequent erosion of the trim.
- 5) A stainless steel instrument line was replaced after it wore through from rubbing on an inlet jumper. The jumper was also replaced because its surface was worn to a depth of 18 mils. The worn jumper held hydrostatic pressure to 6000 psig, indicating that it still had a large factor of safety. Complete inspection of all jumpers and tubing revealed other locations with minor wear, and additional fastening was given these tubing lines to minimize vibration.

SECONDARY

Conventionally softened water is used to cool the secondary side of four heat exchangers: Top and Bottom Shield (HX-2), Reflector (HX-3), Moderator (HX-4), and Primary Treatment (HX-5), before it is deaerated for use as the feedwater to the PRTR Steam Generator (HX-1). During reactor operation, the secondary water leaves HX-5 at about 50 °C (120 °F). This water is deaerated with steam and sodium sulfite addition, and enters the Steam Generator at about 110 °C (228 °F). During shutdowns, secondary system water temperatures are 40 to 70 °F, and the Steam Generator is kept filled with water that has been treated with sodium sulfite and cobaltous nitrate catalyst to remove dissolved oxygen. Sodium phosphate and a sludge conditioner are added to the water in the Steam Generator to minimize scale formation on the heat transfer surfaces and to prevent inter-crystalline cracking. Surfaces in contact with the secondary coolant are carbon steel, except for the stainless steel tube bundles in heat exchangers HX-1, 3, 4 and 5.

Typical localized corrosion occurs in those parts of the system that contain some oxygen. Corrosion rates are very low wherever the water is flowing continuously, but relatively deep local attack can occur in side streams that have only occasional flow. Close monitoring of this corrosion is maintained by regular X-ray, ultrasonic, and visual examination of more than 20 specific locations in this system. Most of these sites have not shown appreciable corrosion for the past two to three years. However, three "deadleg" locations show some active corrosion and are scheduled for another X-ray examination during first quarter of 1968.

Considerable attention has been given during recent months to improvement, repair, and corrosion measurement in the boiler feedwater and steam portions of the Secondary Coolant System. Carbon steel pipe elbows on three steam generator vent lines were replaced when they began leaking. These short lines

serve as "internal" vents from the inlet collar to the shell of the steam generator, and receive intermittent vapor-liquid contact. The three elbows were of much thinner wall than the steam generator shell and connecting nipples. Localized corrosion with buildup of corrosion products in deep "trenches" combined with high cyclic stress levels to cause eventual failure of the elbows, after seven years of service. Annual X-rays are scheduled to monitor corrosion, if any, in the new elbows. During the system outage for this repair, all other lines connecting to HX-1 were X-rayed, and none showed evidence of any corrosion. Close visual inspection inside the steam generator and X-rays of the shell indicated that the tube bundle and the shell were in good condition, although a minor amount of localized corrosion of the shell had occurred, probably as a result of the long shutdown periods in 1966. Corrosion coupons inside the steam generator were examined and one short exposure coupon was replaced. The coupons were in good condition, and the appearance of the older coupons indicated that corrosion was occurring at a very slow rate. Measurements from the replaced carbon steel coupon showed that about four mils of metal were involved in the initial formation of the protective oxide layer on the surface.

A new feedwater and steam distribution cone was installed inside the deaerator in March 1967, and a thorough visual and X-ray inspection was made at that time. Results from pit impressions and the X-ray measurements indicated negligible increase in corrosion penetration since the previous inspection two years earlier. The new cone was inspected in October and was in excellent condition. The entire deaerator is scheduled for visual and X-ray inspection during first quarter, 1968.

A gradual buildup of charred organic material (residue from the proprietary material used for sludge-conditioning)

plugged the end of the line used to feed sodium phosphate and sludge-conditioner to the secondary water inside the Steam Generator (HX-1). When initial attempts to remove the plug from outside HX-1 were unsuccessful, the feedwater system was operated for one month with temporary addition of phosphate to the main boiler feedwater line. The plug was finally removed by hand-reaming from inside HX-1, and phosphate addition was restored to the normal feed location. Ultrasonic measurements of the boiler feedwater line at the temporary phosphate addition point indicated that no corrosion damage occurred during the relatively short time that phosphate was added at this point.

A comprehensive ultrasonic inspection was made on the PRTR blowdown flash tank in mid-1967 to determine the existing condition of the tank walls, especially near a patch-plate installed some years ago for erosion protection. These measurements showed that the tank was in good condition; the two heads averaged 0.337 in. thick, the side walls averaged 0.347 in., and the total thickness at the patch ranged from 0.690 to 0.710 in.

X-ray and ultrasonic measurements made at various locations on heat exchanger and water softener shells during the past year indicate that these locations are in good condition. Some piping locations showed active pitting corrosion and are scheduled for further X-ray during first quarter, 1968.

The flange gasket at the steam generator connection to the main steam export line was replaced in mid-1967 when a severe leak developed at this flange. Erosion was evident on the flange surfaces. Attempts to repair the damage with metal-epoxy materials were largely unsuccessful, due primarily to difficulty of access to the damaged surfaces (these are massive components nearly filling the available space, and can be moved apart by only a fraction of an inch). However, installation of the new gasket stopped the leak.

Fracture and partial disintegration of two bellows protectors was detected during a routine examination of the export steam line in 1966. Metallurgical examination of these 347 stainless steel sleeves showed that initial failure was due to fatigue from flexural stresses concentrated in an area near the weld that had been thinned by grinding. Additional failure occurred from corrosion fatigue with continued low-frequency vibration during periods of high steam flow. The sleeves were removed from the system, and the bellows are included in the program for continued corrosion surveillance.

MODERATOR

High purity heavy water (D_2O) at neutral pH, containing up to 25 ppm boron (as H_3BO_3) for nuclear reactivity control, is circulated at a maximum temperature of about 65 °C (150 °F) in the aluminum and stainless steel Moderator System. Uniform corrosion in this water is negligible for all exposed surfaces of this system, but the difficulty of access for repair of this vital system and the possibility of occasional contact with high pH or other water corrosive to aluminum makes it necessary to maintain close surveillance over the corrosion status of this system.

Continuous measurement of the rate of uniform corrosion of aluminum in this system is provided by an electrical resistance probe in the outlet header from the moderator pumps. Data from this probe show that the aluminum corrosion rate is presently less than 0.005 mils/month, and the total penetration from uniform corrosion has been less than 0.05 mils since the probe was installed in April 1965.

An important concern in the use of H_3BO_3 in the PRTR Moderator System is the effect on corrosion of the aluminum and stainless steel and possibility of excessive sorption of boron on moderator surfaces. Extensive laboratory, pilot-plant, and preliminary in-reactor testing indicated that no significant

corrosion problems should result from boron concentrations of less than 50 ppm in the moderator, provided water purity is maintained within limits and the mechanical shim rods remain out of the reactor.⁽⁵⁾ In-reactor measurements continue to be obtained from samples suspended directly in the calandria with a full range of exposure conditions: (1) continuously submerged, below weir level, (2) submerged during operation, but exposed to moist helium during reactor shutdown, and (3) continuously exposed to the moist helium atmosphere above the moderator liquid. Samples removed in June 1967 showed a corrosion penetration of less than 0.09 mils for exposure periods of 13 to 23 months. Average boron deposition was only 140 $\mu\text{g}/\text{dm}^2$ on submerged samples and about 600 $\mu\text{g}/\text{dm}^2$ on gas blanket samples. Preliminary measurements from samples removed in December showed a decrease in the amount of boron held by the aluminum surfaces, and corrosion penetrations comparable to the samples removed in June. A number of original and replacement samples are in place to provide continuing corrosion surveillance in the moderator calandria.

Calandria surfaces near shim openings C-1651 and C-1447 were visually examined through a borescope in June 1967, and appeared essentially the same as when examined in December 1965. No pits were seen on any surface. Visible surfaces of shroud tubes, calandria bottom plate, and shim well sides appeared smooth and in good condition. Some rust coating was visible in the top shield, as noted in previous borescope inspections, with some flaking occurring in opening C-1447. The bottoms of the shim wells were mostly covered with small debris, judged to be mainly rust and paint flakes, and resin beads.

A ball valve from the boron removal system, a shim recess drain line, and rupture discs from the "A" and "D" dump valves were replaced during October 1967 to correct D_2O leaks or improve equipment operation. The dump valve discs were replaced

to correct leakage around the edge. No corrosion was apparent on the discs, but the plastic liner was pinched and cut at the contact ridge around the circumference of the disc, and occasional copper-colored spots were visible on the monel disc surface. The shim recess drain line (1356) was leaking where its aluminum wall had worn through from vibration against a stainless steel primary system inlet jumper. Other lines were examined and appeared to be in satisfactory condition. The ball valve was examined to determine if corrosion or wear could be contributing to repeated difficulty in getting the valve open. The valve appeared to be in excellent condition; no corrosion or wear was evident.

REFLECTOR

The Reflector System was revised in 1966 to isolate it from the moderator and other D_2O cooled portions of the PRTR. Demineralized H_2O at neutral pH is now recirculated in this system at a maximum temperature of 57 °C (135 °F). These moderate operating conditions are not expected to cause appreciable corrosion of this aluminum and stainless steel system.

Continuous measurement of the rate of uniform corrosion of aluminum in the Reflector System is provided by an electrical resistance probe in the inlet line to the pumps. Data from this probe show that the aluminum corrosion rate is presently less than 0.001 mils/month, and the total penetration from uniform corrosion has been less than 0.001 in. (1 mil) since the probe was installed in April 1961.

SHIELDS

Top and Bottom Shields

Corrosion of the cast iron shot in the top and bottom shields and the carbon steel piping in this system is minimized by using demineralized H_2O as the coolant (maximum temperature

60 °C or 140 °F) with pH adjusted to 9.5 to 10.0. Ammonia has been used instead of lithium hydroxide for this pH adjustment since 1965 to avoid concentration of high pH residue on adjacent aluminum surfaces from a small leak from this Top and Bottom Shield System. Good control of corrosion and close surveillance are vital for this system because it would be virtually impossible to get to the shield leaks to repair them, and excessive leakage from this system would result in severe corrosion of the aluminum calandria and associated piping.

Continuous measurement of carbon steel corrosion in the Top and Bottom Shield System is provided by two electrical resistance probes: one in the inlet line to the reactor shield tanks and one in the outlet line. Data from both probes are in good agreement, showing very low corrosion rates (less than 0.001 mils/month) and showing a total uniform corrosion penetration of less than 0.06 mils since the two probes were installed in April 1965.

Although a significant corrosion rate is not indicated for the Top and Bottom Shield System, the water quality for this system has been repeatedly difficult to control. Difficulties in regeneration of the deoxygenating and mixed bed ion exchange resins in the makeup system have resulted in some poor quality makeup to the Top and Bottom Shield System, but the major problem is pH control in the main recirculating system. Frequent addition of NH_4OH is required to support the ammonia base cleanup resin in keeping the pH above 9.5. With some additions the ammonia is rapidly consumed by presently unexplained mechanisms, and only a minor portion can be detected in the system several hours after addition. With other additions the ammonia remains at relatively high levels in the system, but is apparently neutralized and is not effective in maintaining the system pH above 9.5. The large amounts of ammonia added, plus perhaps other materials in the system,

result in frequent occurrence of high conductivity and total solids concentrations, and occasional occurrence of high suspended solids. Relatively rapid depletion of cleanup ion exchange resins has required frequent replacement of these small units.* Black solids, mostly iron oxide, have been present in the top of each resin bed upon removal. Various tests and analyses are presently being performed in order to resolve this control problem.

Biological and Thermal Shields

Process water (filtered river water) is used in a carbon steel system to cool the concrete biological shield and the steel thermal shield. Maximum temperature of this water is 21 °C (70 °F). A relatively low rate of uniform corrosion is typical for this type of cooling. Surveillance is maintained on these systems to provide an accurate forecast of useful life, and to detect any localized corrosion that might develop in low-flow regions.

Continuous measurement of carbon steel corrosion in the Biological and Thermal Shield Cooling System is provided by three electrical resistance probes. One probe is located in the inlet line to the Biological Shield System, and separate probes are located in the reactor outlet line from each cooling system. Data from the two outlet probes both show rates of less than 0.10 mils/month. Total penetration has been 0.0028 in. (2.8 mils) for the thermal shield outlet and 0.0025 in. (2.5 mils) for the biological shield outlet since the probes were installed in November 1966. The probe in the biological shield inlet line shows a somewhat higher overall corrosion rate of 0.19 mils/month, with indications that the

* At the beginning of the Batch Core Experiment the large Top and Bottom Shield ion exchanger was repiped to the Moderator System for use in boron adjustment, and was replaced with a much smaller unit for cleanup of the Top and Bottom Shield coolant.

corrosion is not completely uniform. Estimated maximum penetration during the past year is 0.005 in. (5 mils).

X-ray examinations performed one year ago on selected locations on the thermal shield inlet line and on both the thermal and biological outlet lines showed that very little corrosion had occurred during the previous two years.

HELIUM SYSTEMS

The PRTR gas systems include (1) the primary pressurization system, in which stainless steel, carbon steel, and small amounts of other materials are in contact with either wet or dry helium at pressures up to 2200 psi and various temperatures up to 250 °C (480 °F); and (2) the core blanket, gas balance, and reflector sweep systems in which aluminum, stainless steel and carbon steel surfaces are in contact with wet or dry helium at pressures from atmospheric to slightly above and temperatures up to 60 °C (140 °F). Dependable operation of various gas blowers, control valves, and relief valves in the Helium Systems is vital to the safe and efficient operation of the reactor. Therefore, close corrosion surveillance is maintained for the portions of these systems that either regularly or occasionally contain water vapor or condensate.

An electrical resistance probe was installed in the reactor outlet line of the core blanket system in February 1967 to detect and monitor carbon steel corrosion that might result from moisture leaking out of the coolant systems. Data from this probe show that negligible corrosion has occurred since the probe was installed (less than 0.01 mils).

X-ray examinations of high-pressure helium lines and ultrasonic measurements of wall thickness of high-pressure and bulk storage tanks were made during the past year, and show that all of the measured locations are in good condition. Moderate accumulations of crud were reported in some of the

bottom lines on the storage tanks. This was confirmed by sampling to be iron oxide corrosion product resulting from the occasional presence of moisture in the tanks.

The following repairs have been necessary on several valves in the high pressure helium-steam environment at the top of the primary system pressurizer:

- 1) The flow control extension broke from the plug of a helium supply valve. Metallographic examination of the failed plug revealed a type of crack characteristic of vibration fatigue; stress corrosion cracking was not indicated.
- 2) The stem and seat of a heat exchanger vent valve required replacement when excessive leakage from this valve allowed D_2O to collect in the low pressure helium storage tanks. Several areas of both the seat and stem had suffered severe erosion damage characteristic of high velocity flow of vapor-liquid mixtures.
- 3) The safety relief valves on the primary pressurizer have required a number of adjustments and/or trim replacements to correct leakage. No corrosion or wear damage has been evident on parts of these safety relief valves examined to date.

Cracks several inches long have developed in the expansion bellows below "D" dump valve in the gas balance system on two occasions in the past 16 months. Each time, there was a considerable helium loss from the system. A crack also developed in the bellows on one of the top gas lines. The cracks have been successfully sealed by welding, and the other bellows have shown no evidence of damage.

PROCESS WATER

The process, emergency, and sanitary water supplies for the PRTR are chemically similar (treated river water or well water) and are transported in carbon steel systems at temperatures up to 60 °C (140 °F). Process water cools various

shields and step plugs (including the Biological and Thermal Shield System described above) and the fueling vehicle, provides the source of makeup for the Secondary, Top and Bottom Shield, and Reflector Systems, and supplies (with the Sanitary Water System) the conventional water requirements of the PRTR complex. A low rate of uniform corrosion is normally experienced in this type of water, but occasionally severe localized corrosion occurs in low-flow or "dead-leg" regions. Surveillance of process water locations provides data to help in forecasting the useful life and replacement frequency for important parts of the system.

X-ray examination of candidate locations for localized corrosion, and ultrasonic measurements of wall thickness of important heat exchanger components, all made during the past year, indicate that most of the measured locations are in good condition. However, a short branch line in "C" cell showed severe pitting corrosion occurring during the past two years. Parts of this line may need to be replaced within three to four years. Another X-ray of this location is scheduled for late 1968.

Replacement of a leaking flexible cooling line on low pressure helium compressor No. 1 provided an opportunity for close visual examination of this special type of process water line. This carbon steel line consists of a welded helical bellows with a protective braid on the outside. Localized corrosion was observed in the line, consistent with its long operating lifetime and operating conditions [about 2 ft/sec process water velocity at about 75 psig and 60 °C (140 °F)]. Failure occurred in the helix welds.

FUEL ELEMENT RUPTURE TEST FACILITY

An experimental facility is incorporated into the PRTR to determine the performance characteristics of new types of

fuel elements and to evaluate the failure response of purposely defected elements. Coolant for this Fuel Element Rupture Test Facility (FERTF) is demineralized H_2O at pH 9.5 to 11.0. Piping, valves, and heat exchanger components are primarily stainless steel, with Zircaloy-2 as the in-reactor test section and (ordinarily) the fuel element cladding. A noteworthy exception to the above materials is the relatively pure silver coating on the piston shaft bushings in the positive-displacement main system pumps. Other materials (e.g., Hastelloy-C, a Ni, Mo, Cr, Fe alloy) may occasionally be included with test fuel assemblies. The system is ordinarily operated at 1125 psig and 270 °C (570 °F), but in recent months the maximum temperature has been maintained at 65 °C (150 °F) while the effectiveness of certain emergency cooling supplies is being evaluated and improved. Approximately 95% of the recirculating coolant flow is depressurized, passed through cleanup ion exchange units, and returned to the system.

Maintenance of good quality coolant (especially low dissolved O_2 concentrations) and close corrosion surveillance are vital to assure that chloride stress corrosion possibilities are minimized, because this facility operates at high levels of thermal and vibratory stress.

An X-ray examination was made in September 1967 on four high-pressure and one low-pressure location considered most likely to suffer stress corrosion damage. No damage was evident. Another X-ray examination is scheduled for these locations just prior to the start of high-temperature operation.

The chemical used for pH adjustment in the FERTF was changed from LiOH to NH_4OH in December 1967 to provide positive control of dissolved O_2 concentrations. At the low system operating temperature of 65 °C (150 °F), $H_2 - O_2$ recombination was not sufficient in LiOH treated water to

maintain O_2 concentration below 5 ppm. Extensive single-pass operation required to maintain low O_2 concentration resulted in rapid depletion of the makeup ion exchange resins and accumulation of waste water. Initially, the addition of ammonia alone was not effective enough for the desired O_2 control, so hydrazine addition was started. This combination was effective, but the resulting H_2 produced could not be removed fast enough from the loop storage tank by the existing vent system. At the next opportunity, a continuous nitrogen purge system was added to the FERTF storage tank, ammonia base resin was put in place of the lithium base material in the cleanup ion exchange units, and hydrazine was added to the FERTF system for a short time during a reactor outage. Hydrazine was effective in the rapid removal of an apparent O_2 reservoir incorporated in highly oxidized corrosion product layers on the system surfaces. Subsequent operation has been excellent with only occasional addition of NH_4OH (and no hydrazine) needed to maintain O_2 concentrations less than 0.01 ppm, H_2 equal to 1% or less in the storage tank cover gas, and other loop constituents under good control. Possible corrosion and nuclear activation of silver from the main pump bushings was a prime concern at the start of ammonia addition to the FERTF. However, radiation measurements on the piping, and measurements of ^{110}Ag radioactivity and atomic absorption spectra from coolant samples show that corrosion and activation of the silver is negligible. Laboratory corrosion studies at 60 °C (140 °F) also showed that the silver is not damaged by exposure to the pH 10 ammonia solution. Laboratory tests also showed that copper alloy bellows and springs (as occasionally present in control instruments) were not affected by three months exposure to a pH 10 ammonia solution (about 10 ppm) at 25 °C (77 °F).

A special capability of the FERTF is the provision for chemical decontamination of the facility with direct measurement of the effectiveness and corrosion properties of

decontaminant mixtures with coupon samples exposed in a test section in the main piping. Since ordinary uniform corrosion of system materials is negligible, these samples are not intended to be a routine surveillance item. Accordingly, they are not examined or replaced except for special operations such as decontamination. The last measurements obtained from these samples were for a 20-month exposure period that included failure of a defected fuel element, a relatively long outage period, and the subsequent decontamination of the system. Data from stainless steel and inconel "X" samples showed a maximum total penetration of less than 0.06 mils for this period. Zircaloy corrosion was less than 0.005 mils.

The only significant deterioration to any parts of the FERTF system has come from wear or other physical damage, and not corrosion:

- 1) In September 1967, two loose pieces of metallic silver totalling 3/4 g were removed from the FERTF flow control valve and one tiny silver flake was removed from the fuel element. These pieces of silver appeared as scratched and badly wrinkled thin sheets, apparently torn from a bushing on one of the main FERTF pumps. The pieces showed no evidence of erosion, melting or corrosion damage. A damaged bushing, replaced several months ago, is the probable source of this silver. All of the bushings in both pumps were replaced in November. Some of the replaced bushings showed considerable wear on the silver surfaces, but none showed evidence of any loose or missing pieces of silver.
- 2) Inspection of FERTF piping in September revealed three locations where significant wear was occurring on branches of the main loop piping that were vibrating and rubbing together. Pieces of silicone rubber sheeting were fastened between the pipes to stop the wear. These locations are

inspected at regular intervals to assure that the wear protection remains in place, and that other wear points do not occur.

- 3) A sample of used FERTF ion exchange resin was removed from the ion exchange tank and examined to determine its residual capacity and identify the materials collected on it. This resin bed had been replaced after a valving error during single-pass operation resulted in washing of some resin into the loop piping. A considerable amount of fine suspended material was scattered through the resin sample. Emission spectrographic analysis of a separated sample of this fine material showed it to contain large quantities of iron and aluminum and moderate amounts of other metallic constituents, including silver. Major gamma emitting radioisotopes included ^{110}Ag , ^{106}Ru , and ^{144}Ce . Capacity measurements and hydraulic separation showed that most of the anion resin had apparently been washed out of the bed. The residual cation capacity (i.e., Li content) was about 86% of that specified for new resin. Regeneration of this fraction restored it to 95% of specified capacity for new cation resin, indicating that this cation fraction (although slightly depleted) was in good condition.

MISCELLANEOUS

Corrosion and wear surveillance is important for a number of PRTR components (chemical makeup tanks, ventilation equipment, etc.) that are not a definite part of any of the major cooling systems, but may play a key role in maintaining safe and efficient operation of a major PRTR system.

Ultrasonic measurements have been made on the tank used to supply brine (concentrated NaCl) for water softener regeneration. Relatively uniform corrosion of the side walls has occurred, reducing the 0.250 in. original wall thickness to 0.173 to 0.183 in. as measured at 5 levels on the tank wall. Annual

measurement of wall thickness is planned, but the tank is expected to give many more years of service.

Several copper lines and fittings failed on water chillers in the 309 Building during the first half of 1967, resulting in leakage of Freon refrigerant or glycol-water mixtures from the units. The failures were primarily fatigue cracks; the entire tubing assembly was free to vibrate excessively as a sort of tuning fork to the water chiller unit. A number of tubing runs were replaced with copper tubing having greater ductility, and the tubing assembly was fastened to the frame of the unit. No more failures have occurred.

Loosening of mechanical joints between the thermal barrier and various reactor moderator and gas lines caused increased loss of D₂O vapor from the access space below the reactor. These joints were sealed with a synthetic rubber (Room Temperature Vulcanizing) material, thus greatly improving recovery of the D₂O vapor.

REFERENCES

1. R. H. Purcell. Plutonium Recycle Test Reactor Experience, 1962-1966, BNWL-SA-557, Pacific Northwest Laboratory, Richland, Washington. April 15, 1966.
2. L. D. Turner et al. Unpublished Data on PRT Operational Planning, Pacific Northwest Laboratory, Richland, Washington. January 4, 1967.
3. L. D. Turner. Unpublished Data on In-Reactor Monitoring of Zircaloy-2 PRT Pressure Tubes, Pacific Northwest Laboratory, Richland, Washington. July 21, 1966.
4. A. B. Johnson, Jr. In-Reactor Corrosion of Zirconium Alloys, BNWL-SA-1611, Pacific Northwest Laboratory, Richland, Washington. January 1968.
5. G. R. Bloom and T. F. Demmitt. Corrosion and Boron Deposition in Boric Acid Solutions, BNWL-520, Pacific Northwest Laboratory, Richland, Washington. December 1967.



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