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YANKEE ATOMIC ELECTRIC COMPANY
RESEARCH AND DEVELOPMENT PROGRAM

**CORROSION PRODUCT TRANSPORT AND DEPOSITION
UNDER IONIZING RADIATION**

R & D SUBCONTRACT No. 1 under
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**WESTINGHOUSE ELECTRIC CORPORATION
ATOMIC POWER DEPARTMENT**

PITTSBURGH, 30

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Yankee Atomic Electric Company
Research And Development Program

CORROSION PRODUCT TRANSPORT AND DEPOSITION
UNDER IONIZING RADIATION

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I. ABSTRACT

This report covers a study of corrosion product transport and deposition on Zircaloy-2 and AISI 304 stainless steel in the presence and absence of ionizing radiation. Three 100 hour irradiation tests, using 2 Mev electrons from a Van de Graaff accelerator, and four 100 hour non-radiation tests were performed in 600°F pressurized water. Data from the seven runs and an additional exploratory run are presented. In addition, complete experimental procedures and descriptions of the apparatus are included.

PH control was obtained by using H and OH form ion exchange resins for pH 7 and Li and OH form ion exchange resins for pH 10.

The major conclusion to be drawn from the present work is that the deposition on Zircaloy-2 at pH 10 and 600°F is higher than on AISI 304 stainless steel at the same conditions.

II. INTRODUCTION

Bettis Plant workers in reactor technology found a gradually increasing coolant pressure drop across test sections undergoing prolonged pile irradiation at NRX. This was attributed to the deposition of migratory corrosion products depositing out from the moving coolant water on to the metallic surfaces in the highly irradiated field. W. T. Lindsay of the Bettis Plant and other have postulated that the mechanism of the deposition observed was ionic in nature, and that relatively short-lived free radicals were formed, which chemically recombined with the metallic fuel element test surface. Prolonged deposition of a tightly adherent, relatively porous, and therefore thermally insulating corrosion product could have disastrous results in terms of reduced heat transfer from cladding to coolant, resulting in increased fuel and cladding temperatures.

Fortunately, reactor experience so far gained has failed to justify these fears. Most of this reactor experience, however, has been on reactors operating at elevated pH with Zircaloy fuel cladding surfaces.

As a relatively inexpensive and convenient method of studying the phenomenon of crud deposition under ionizing radiation, Van de Graaff generators were adopted to beam high energy electrons into crud suspensions and solutions. Deposition onto metallic surfaces was then measured. The assumption is implicit in such experiments that the ionizing type of radiation produced by the electron beam is controlling, and that other portions of the radiation spectrum found in a reactor core are insignificant in their effect on crud deposition and transport.

Very early studies relating crud deposition and transport to Van de Graaff irradiation were conducted by Glick¹. His conclusions may be summarized as follows:

1. Deposition is a function of the pH at operating temperature*.
2. Deposition is increased by irradiation.
3. The deposition is produced by the absorption of ionizing radiation in the water, rather than by radiation damage effects on the irradiated surface.

Some later work conducted by McAllister² showed no detectable difference in the deposition of corrosion products from carbon steel and stainless steel crud sources.

Several unresolved questions arose as a result of these studies. An experimental program was initiated to carry on further work.^{3, 4, 5} The results, to date, of this study substantiate the effect of pH found in the earlier work. Also, they have shown that fresh crud behaves differently than aged crud or simulated crud (ferrous hydroxide or magnetite).

Most of the work cited above has been conducted at a water temperature of 450°F and concerns crud deposition on zirconium and Zircaloy-2 surfaces. The Yankee reactor will operate at water temperatures approaching 600°F, and with core and piping consisting almost entirely of AISI 304 stainless steel exposed to primary coolant water. For these reasons, the study reported here was initiated to investigate deposition on AISI 304 stainless steel at neutral and elevated pH (lithium controlled) in 600°F water as part of the Yankee research and development program.

* More deposition was observed at lower pH. Operating temperature 450 to 550°F.

III. CONCLUSIONS

Several tentative conclusions may be drawn on the basis of the present study:

1. The only deposits attributable to radiation induced deposition were found on the directly irradiated surfaces, rather than on the specimens.
2. No significant difference was noted in the amount of iron deposited under radiation at pH 7 and at pH 10 on AISI 304 stainless steel.
3. The deposition on Zircaloy-2 at pH 10 is greater than on AISI 304 stainless steel at pH 10.
4. The size of the crud particles appears to increase with pH.
5. Comparison of these results with previous experimenter's data shows considerable difference in the apparent crud release rates found at pH 7 and in the location of the crud deposits at either pH.

The major conclusion to be drawn from the present work is that there is a definite need for more work on the problem of crud deposition. In particular, the effect of temperature on crud deposition should be investigated. It would probably be of considerable help in future studies to use test specimens that do not contain elements in common with the crud.

IV. APPARATUS DESCRIPTION AND EXPERIMENTAL PROCEDURE

A. Apparatus Description

The apparatus used in the present system is similar to that previously reported^{3,4}. However, several modifications to the loop were made, based on the fact that insufficient crud was generated previously at elevated pH. The modifications were as follows:

- (1) The crud generator volume was increased to increase the crud source surface area.
- (2) The heat exchanger was modified to permit higher flow rates without exceeding the temperature limitation of the ion exchange columns. The experimental apparatus, as shown in Figures 1, 2, 3, and 4 consists essentially of three sections: a water treatment section, a pressurizer section, and a forced circulation section containing the sample and the sample chamber.

1. Water Treatment Section

The water treatment section consists of a preheating chamber, a transfer pump, a pressure rupture disc, and the associated piping. The preheating chamber is used to remove dissolved gases from the water, prior to transferring the water to the pressurizer section. The transfer pump is capable of delivering water into a system already pressurized to 2500 psi.

2. Pressurizer Section

The pressurizer section contains an autoclave used as a pressurizer, a pressure gage, a differential pressure cell, a pressure rupture disc, a hi-low pressure alarm, and the associated piping. The pressurizer is used to raise the water pressure to the required 2500 psi. The differential pressure cell is calibrated in inches of water and indicates the water level in the pressurizer. The hi-low pressure alarm is used to warn the operator if the pressure falls below 1875 psi or rises above 2750 psi.

The alarm system is connected to the Van de Graaff accelerator controls to automatically shut off the machine in case of high or low pressure. The alarm will also shut off the loop and pressurizer heaters and the canned rotor pump. The pressure in the pressurizer is maintained at the desired level by means of a thermocouple and a Brown Pyrovane Controller which controls the current to the pressurizer heaters.

SCHEMATIC ARRANGEMENT OF VAN DE GRAAF LOOP

FIGURE 1

CRUD DEPOSITION LOOP

(View from Front)

FIGURE 2

CRUD DEPOSITION LOOP

(View from Rear)

FIGURE 3

CRUD DEPOSITION LOOP

(View from Side)

FIGURE 4

B. Forced Circulation Section

The forced circulation section consists of a canned rotor pump, flowmeter, crud pot, sample chamber, heat exchanger, three demineralizers, and the associated valves, piping and heaters. The sample chamber is discussed later in this section. The bottom outlet of the pressurizer is connected to the suction side of the pump. The pump forces the water through the flowmeter, the heater section, the crud pot, the sample chamber, the heat exchanger, one or more of the three demineralizers, and back to the pump.

The sample chamber, Figure 5, consists of an upper and lower section. The upper section attaches to the Van de Graaff accelerator, during irradiation runs, and contains thirteen 5/64 inch diameter beam-defining holes arranged in three rows. The lower section is essentially a chamber with an inlet, an outlet and provisions for inserting the specimen holder. A stainless steel or Zircaloy-2 gasket (7-8 mils thick) separates the upper and lower section. The gasket serves as a high pressure radiation window. The radiation enters the lower section by passing through the beam-defining holes which permit 15 percent of the total beam to enter the lower section. The energy of the beam is reduced to approximately 1.8 Mev in penetrating the stainless steel or Zircaloy-2 gasket.

The temperature in the sample section is maintained at the desired level by controlling the temperature of the water leaving the crud pot. A thermocouple on the upstream side of the crud pot is connected to a Brown Pyrovane proportional controller which regulates the current to the crud pot.

Thermocouples are located at a number of points in the loop (Figure 1). The thermocouples that are not used for controllers are connected to a Leeds and Northrup multipoint recorder. The beam current (during irradiation) is also indicated by the recorder, providing a continuous record which is integrated to obtain the average beam current.

The specimen holder, Figure 6, is designed so that water flows over one side of a 5-inch long specimen and back over the other side. On each side the water is channeled through three narrow paths in series such that a path length of 12 inches per side is obtained. The specimen is placed in the holder under the Van de Graaff beam. Water from the loop enters the specimen holder after irradiation by the 1.8 Mev electron beam at a beam current of about 19 (15% of total) microamperes.

All the corrosion products released to the loop water by the crud pot pass over the surface of the specimen. The full stream is then cooled and purified. Solid particulate matter is removed by a HA Millipore filter mounted in the demineralizer head. The water then passes through a separately contained cation and anion resin which removes the soluble and colloidal corrosion product species. By analyzing for the iron on the filter, the ion-exchange resins, the specimen, and the high pressure window, the amount of corrosion product available to the specimens is inferred. The amount of corrosion product deposition on the heat exchanger and associated piping is unknown.

BEAM HOLE LAYOUT

SAMPLE CHAMBER

FIGURE 5

SPECIMEN HOLDER

FIGURE 6

C. Experimental Procedure

This section describes (1) the desired operating conditions, (2) the samples, (3) the preparation of the apparatus for the run, (4) the procedures for water analysis, (5) the measurement and control of operating conditions, and (6) the sampling procedures.

1. Desired Operating Conditions

The general operating conditions desired in all of the runs were as follows:

- (1) temperature - 610-620°F
- (2) pressure - 2500 psi
- (3) time of irradiation - 100 hours
- (4) beam current - 125 ± 2 microamperes
- (5) beam voltage - 2 Mev
- (6) flow - not less than 5 feet per second
- (7) water properties - oxygen less than 0.1 ppm, operating pH 6.7 to 7.3 or 9.8 to 10.5

2. Samples

The samples used in the present study were 10 - 11 mils (0.010 - 0.011 inch) thick. The AISI 304 stainless steel specimens were used in the as-rolled and acetone degreased conditions. The Zircaloy-2 specimens in the as-rolled condition were coated with a heavy corrosion product film and were, therefore, pickled prior to use. The pickling was done with a solution containing 48 percent nitric acid, 2 percent hydrofluoric acid and 50 percent water. The pickling was performed at room temperature followed by a two-hour rinse in running tap water. The tap water rinse was followed by demineralized water and acetone rinses.

The window between the upper and lower sample sections was of the same material as the test specimen.

3. Loop Start-Up Procedure

The test specimen and window were both weighed to the nearest 0.1 of a milligram before placing them in the apparatus. The carbon steel or stainless steel crud sources were weighed to the nearest 0.1 of a gram prior to loading the crud pot. Specimen and window gross weights ranged from 1.5 to 3.0 grams.

The apparatus was filled with oxygen-free water in the following manner. First the preheating (make-up) chamber was filled with distilled, demineralized water. The chamber was heated until 50 percent of the water was evacuated. The water from the pre-heating chamber was then transferred to the pressurizer. Several charges of water were transferred to the pressurizer in this manner. The pressure was then increased to 500 psi by heating the pressurizer; the differential pressure cell was purged and then the pressurizer was purged to 50 psi pressure. After the purging operation, the pressurizer was refilled with purified water. This purging operation was repeated each time the pressurizer had been exposed to atmospheric pressure.

The procedure for starting up the circulating loop is as follows:

- (1) Drain and evacuate loop.
- (2) Flash high temperature, high pressure water into loop from pressurizer with demineralizer closed off.
- (3) Slowly fill demineralizers.
- (4) Circulate water through loop and through all demineralizers.
- (5) Draw and analyze water samples. When oxygen is less than 0.1 ppm, close off all demineralizers except clean-up column.
- (6) Raise temperature to operating point. Establish equilibrium water conditions by running through clean-up ion exchanger.
- (7) Switch to fresh ion-exchanger at beginning of run.

4. Water Analysis Procedure

The oxygen analysis was run in the following manner. A 100 ml ground-glass stoppered (tapered stopper to prevent air bubble entrapment on filling and stoppering) bottle was twice rinsed with 20 ml portions of water, then slowly filled from the exit valve (shown in Figure 1). Fifty to one hundred ml of water were permitted to overflow to insure removal of all entrapped air.

One ml of alkaline potassium iodide (APHA) and one ml of manganous sulfate (APHA) were added to the contents of the bottle. The bottle was stoppered and shaken. Two ml of 18N sulphuric acid were added and the bottle was stoppered and shaken again. The solution was finally transferred to an evaporating dish and titrated to a water-white starch endpoint using 0.01 N sodium thiosulfate delivered from a 10 ml burette with a capillary tip. All of the reagents employed were received from Fisher Chemical Company and are standard for the determination of oxygen. By a similar titration procedure, a reagent blank was obtained and subtracted from the sample determination to give the corrected value. A sample calculation follows:

Analysis	0.15 ml
Blank	0.09 ml
	<hr/>
Actual value	0.06 ml

$$\text{ml} \times N = \text{me. of } O_2$$

$$\frac{\text{me. of } O_2}{4} = \text{millimoles of } O_2$$

$$\text{millimoles of } O_2 \times 22,400 \text{ ml (assuming STP)} = \text{ml of } O_2$$

$$\frac{\text{ml of } O_2}{\text{ml of water}} \times 1000 = \text{ml of } O_2/\text{liter of water}$$

$$\frac{0.06 \times 0.01 \times 22,400 \times 1000}{4 \times 1000 \times 53} = 0.06 \text{ ml } O_2/\text{liter}$$

In addition to the oxygen analysis, the pH of the water was determined by the use of a pH meter and the conductivity of the water was measured using a conductivity meter. The results are tabulated for the individual runs.

5. Measurement and Control of Operating Conditions

The following section describes the data taken during the run, the operating conditions during the run, and the methods used to control and record these conditions. During the run, a twelve-point recorder was used to record continuously the following data: pump coolant temperature, pump discharge water temperature, temperature at the center and the end of the heater section, temperature of water leaving the crud pot, sample inlet and outlet temperatures, heat exchanger outlet temperature, target current and three zero check points. The following data were recorded every two to three hours for irradiation runs and every 16 hours for non-irradiation runs: operator, total operating time, water pressure, water level, pressure controller reading, temperature controller reading, flow rate; and for Van de Graaff runs, machine on and off time, beam voltage, beam current, up-charge current and down charge current, column current, coil current, Van de Graaff vacuum meter, sample vacuum meter, and twelve readings from the multipoint recorder.

Typical values obtained during the run for all of these readings are as follows:

- (1) sample vacuum chamber, 200 - 1000 microns
- (2) water pressure, 2475 - 2525 psi
- (3) water level 4 to 10 inches with refill at about 4 inches

(4) recorder readings

- a. pump coolant - 0.7 to 0.8 millivolts = 64 - 68°F
- b. pump discharge - 3.0 to 4.0 millivolts = 165 - 208°F
- c. center of heater section - 7.0 to 8.0 millivolts = 341 - 386°F
- d. end of heater section - 10.5 to 11.8 millivolts = 497 - 555°F
- e. crud pot exit water - 12.8 to 13 millivolts = 597 - 606°F
- f. sample inlet water - 13.0 to 13.2 millivolts = 606 - 615°F
- g. sample outlet water - 13.0 to 13.2 millivolts = 606 - 615°F
- h. heat exchanger outlet water - 1.7 to 1.8 millivolts = 108 - 112°F
- i. zero - 0
- j. target current - 12.3 to 12.7 millivolts equivalent to 123 to 127 microamperes

- (5) pressure controller, 6.5 - 6.7 millivolts
- (6) temperature controller, 7.2 - 7.4 millivolts
- (7) flow rate, 9.0 - 9.7 gph equivalent to 7.5 to 8 feet per second in sample holder
- (8) beam voltage, 1.99 - 2.01 Mev
- (9) beam current (console) - 123 - 127 microamperes
- (10) coil current, 1.5 amperes
- (11) upcharge current, 150 - 160 microamperes
- (12) downcharge current - 0 microamperes
- (13) column current, 25 - 30 microamperes
- (14) leakage current, 15.5 - 19 microamperes
- (15) vacuum (Van de Graaff machine) - 0.5 - 30 millimicrons of mercury.

Servicing during the runs consisted mainly of (1) filling the mercury diffusion pump system with liquid nitrogen and dry ice, (2) filling the sample chamber pump trap with dry ice, (3) filling the standard cold junction for the recorder with crushed ice, and (4) adjusting the flow rate.

After the completion of the run, the same physical properties of the water were determined as before the run; oxygen content of water, pH, and conductivity. These values were in general unchanged or only slightly changed from pre-run values. The sample was then removed, permitted to air-dry, weighed, and percentage weight change determined.

6. Sampling Procedures

At the completion of a run the test demineralizer, test specimen, and the high pressure radiation window were removed from the apparatus.

- (1) Filter Treatment - The millipore filter and backing were digested in 250 ml of 3 N nitric acid. The solution was filtered and submitted for total iron analysis. If the residue from the filtration indicated the presence of iron, this was also submitted for analysis.

- (2) Cation Resin - The ion exchange columns contained 100 ml of cation resin separated from the anion resin by a Pyrex wool plug. The resins were removed from the column separately. The cation resin was slurried with 250 ml of 3 N nitric acid and quantitatively transferred to a glass column. The acid rinse was percolated slowly through the resin into a filtering funnel placed in a sample bottle and submitted for iron analysis. The column was allowed to go dry.
- (3) Anion Resin - The anion resin charge was 190 cc. The resin was slurried with 400 ml of 3 N nitric acid and transferred to a glass column. The acid was percolated through the resin as described previously. The solution was analyzed for contained iron.
- (4) High Pressure Radiation Windows - The windows from the radiation runs were cleaned in 3 N nitric acid and the cleaning solution was then assayed for iron. The cleaned radiation run windows and the uncleaned control run windows were further assayed as described below for test specimens.
- (5) Test Specimens - The specimens were electrolytically etched in dilute sulfuric acid. Control specimens were treated in the same manner as the test specimens. Specimen weight losses were determined and the etch solutions were assayed for total iron content. The iron removed from the test specimen, minus the iron removed from the control specimen, was reported as the descaled weight loss.

V. RESULTS

The experimental results are summarized in Table I. The windows from the irradiation runs showed a deposit that could definitely be attributed to radiation-induced crud deposition. The affected area was limited to that corresponding to the beam holes in the upper sample section. The deposit was somewhat built up and was markedly thicker than any deposit on the rest of the window. Similar deposits were not noted on windows from the control runs. The windows from the radiation runs were cleaned with 3 N nitric acid, removing the crud deposit. The amount of crud deposited was determined by chemical analysis of the cleaning solution. An unused window was treated in the same way. Analysis of the solution from this treatment showed that the acid did not attack the base metal. The windows from the control runs and the specimens from both control and radiation runs showed no deposits that could be attributed to crud deposition. The weight losses from the descaled specimens were, generally, about what would be expected from corrosion alone. This was true even for run CR-3. In this run the temperature control was unsatisfactory for a period of several hours and a steam phase existed in one section of the loop during this period. A number of the descaled weight losses were within the weighing error.

TABLE I

RESULTS OF VAN DE GRAAFF CRUD DEPOSITION EXPERIMENTS

Carbon Steel Crud Source

Run No.	CR-2 ^a	VG-1 ^a	CR-3 ^b	VG-2 ^b	CR-5 ^b	CR-4 ^b	VG-4 ^{b,d}
Test Material	304 ss	304 ss	304 ss	304 ss	304 ss	Zircaloy-2	Zircaloy-2
Average Temp. °F	612	613	614.5	613	611	604	607.5
Hourstat Temp.	111.5	111.0	100.9	103.3	99.8	101.5	104.7
Average Beam Current; amp ^c	-	125.1	-	124.6	-	-	124.8
Hours of Beam On	-	100.5	-	99.8	-	-	98.7
Beam Energy; Mev	-	2	-	2	-	-	2
Average Flow Rate in Sample Section; ft/sec	7.8	7.5	7.8	7.8	7.8	7.8	7.8
Crud Source Area; dm ²	498.6	508	544.5	529.9	537.4	588.8	503.2
Water Chemistry							
a. Dissolved Oxygen; ppm	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1
b. Max. pH	7.2	7.1	10.1	10.3	10.3	10.2	10.3
c. Max. Conductivity; micromhos	0.13	0.13	83	109	125	76	114
Analytical Results							
a. Iron on cation resin; mgs	9.07	3.02	2.59	2.79	0.000	4.45	0.19
b. Iron on anion resin; mgs	1.721	0.23	1.23	1.00	0.26	2.04	1.29
c. Iron on filter; mgs	1.01	4.24	16.33	11.56	74.25 ^e	19.58	19.09
d. Iron from window; mgs	none	0.10	none	0.08	none	none	0.84
Metallurgical Results							
a. Window weight before descaling; gms	1.4818	1.3451	1.4854	1.7493	1.6656	2.2974	2.2196
b. Specimen weight before descaling; gms	2.7862	2.7710	3.1042	3.0902	3.0898	2.6699	2.6420
c. Window descaled weight loss; mgs ^f	0.0	0.1 ^g	0.6 ⁱ	-0.2 ^g	1.8 ^j	0.2 ^g	-0.1 ^g
d. Specimen descaled weight loss; mg ^f	0.2 ^g	0.9 ^h	2.3 ⁱ	0.4 ^g	0.7 ^h	0.2 ^g	-0.1 ^g

TABLE I (cont'd)

Percentage Crud Deposited

a. Window ^k	none	1.3	none	0.5	none	none	3.9
b. Specimen ^k	none	none	none	none	none	none	none
c. Anion Resin	14.6	3.0	6.1	6.5	0.3	7.8	6.0
d. Cation Resin	76.9	39.9	12.9	18.1	100.0	17.1	0.9
e. Filter	8.5	55.8	81.0	74.9	99.7	75.1	89.2

-
- a. Operating conditions established by flowing water through H-OH resins.
 - b. Operating conditions established by flowing water through Li-OH resins.
 - c. 15% of total beam entered loop water.
 - d. Run VG-3ZIRC-CRUD-10 terminated after 25 hours due to rupture of high pressure window.
 - e. Results from filter in one of two test demineralizer columns were very high. Reason is not known but is not felt to be realistic; possibly iron contamination occurred during digestion of filter.
 - f. Weight loss of test sample minus weight loss of control sample treated in identical manner.
 - g. Weight change attributed to weighing error.
 - h. Weight loss is less than would be expected from corrosion alone.
 - i. Steam phase in sections of loop for several hours; explains high results. However weight losses are about those expected from corrosion alone.
 - j. Copper from upper sample section flange on back of window. Descaled weight loss probably result of loss of copper. No corrosion or crud deposit noticed on exposed surface of window.
 - k. Descaled weight loss neglected since they can be attributed to factors other than crud deposition.
 - l. Based on time at temperature, crud source area and 730 hours per month.

The release rates listed in Table I are based on the amount of iron found by analysis, the hours at temperature, the surface area of the crud source and a 730 hour month. It should be noted that this is not the total release rate since it was not feasible to determine the amount of iron deposited in the heat exchanger. It is assumed that the amount of iron deposited in the heat exchanger is constant for runs at a given pH.

In most of the runs it was necessary to use two demineralizers. The crud caught on the filter in the first demineralizer head would plug the filter thus making it difficult to maintain the desired flow rate and requiring diversion of the flow to the second demineralizer.

The estimated error in the analytical results is ± 20 percent. Consequently, there does not appear to be any significant difference in the amount (weight) of iron (crud) deposited on the window at the two pH values. If the amount of deposition is expressed as a percentage of the total iron found there is a difference. However, it is felt that there is not sufficient data to conclude that this difference is significant. There is, also, an apparent difference between the total iron found, and therefore the release rate, at the two pH values. Again there is not enough data to evaluate the significance of this difference. The difference in the amount of crud deposited on Zircaloy-2 and AISI 304 stainless steel at pH-10 is probably real.

One effect of pH that is probably real is an increase in the amount of iron found on the filter and a decrease in the iron found on the cation resin with increase in pH. This would indicate that the crud released to the system becomes more particulate as the pH increases. Similar results were found in the Bettis Plant experiments.

Photographs of windows and test specimens, prior to descaling, from a number of the runs are presented in Figures 7 through 13.

As pointed out above the only identifiable crud deposits found in the present study were on the windows from the radiation runs. In the Bettis Plant work the crud deposits were found on the test specimens from both control and radiation runs. Furthermore, the Bettis Plant workers have not found deposits on the windows of the type that were found in this work. It should be noted that Zircaloy-2 is the only material tested at the Bettis Plant in a loop similar to that used in these studies; even for Zircaloy-2 the operating conditions differed in the two studies, as shown in Table II. The Bettis Plant workers found that at pH 10 (Li controlled) 43.4 percent of the total iron found was deposited on the specimen in the presence of radiation and 18 percent in the absence of radiation. However, the total amount of iron found (averaged over five runs) was only 0.50 mg. The large experimental error at such low iron levels must be considered in evaluating these results. The Bettis Plant data at pH 7 shows an average of 14.6 (range 8.4 - 22.3) percent deposition with radiation and 2.3 (range 1.2 - 4.3) percent deposition without radiation on Zircaloy-2. The following calculation, using data from the present report (Table I) is of interest:

Water Inlet Side - Radiation Run at Bottom

Water Outlet Side - Radiation Run at Bottom

TEST SPECIMENS FROM RUNS VG-1 AND CR-2 PRIOR TO DESCALING
FIGURE 7

Radiation Runs - VG-1 pH 7, VG-2 pH 10

Control Run

Sides shown were exposed to loop water.
Control run window was not treated with nitric acid.

WINDOWS FROM RUNS VG-1, VG-2 and CR-5
AFTER NITRIC ACID DIGESTION AND PRIOR TO DESCALING

FIGURE 8

Radiation Run

Control Run

TEST SPECIMENS FROM RUNS VG-2 AND CR-5 PRIOR TO DESCALING
WATER INLET SIDE

FIGURE 9

Radiation Run

Control Run

TEST SPECIMENS FROM RUNS VG-2 AND CR-5 PRIOR TO DESCALING
WATER OUTLET SIDE

FIGURE 10

Radiation Run

Control Run

Sides shown were exposed to loop water.

Control run window was treated with nitric acid.

WINDOWS FROM RUNS VG-4 AND CR-4
AFTER NITRIC ACID DIGESTION AND PRIOR TO DESCALING

FIGURE 11

Radiation Run

Control Run

TEST SPECIMENS FROM RUNS VG-4 AND CR-4 PRIOR TO DESCALING
WATER INLET SIDE

FIGURE 12

Radiation Run

Control Run

TEST SPECIMENS FROM RUNS VG-4 AND CR-4 PRIOR TO DESCALING
WATER OUTLET SIDE

FIGURE 13

TABLE II

COMPARISON OF RADIATION AND NUCLEONICS LABORATORY
AND BETTIS PLANT LOOP OPERATING CONDITIONS

Site	Temperature, °F	Flow-Rate Sample Sect; fps	Operating Time; hrs.	Crud Source Surface ₂ Area; dm ²
RNL	600 - 615	7.5 - 7.8	100	500 - 590
Bettis	450	4.3	48 - 50 ^a	33.85
^a Some control runs were run as long as 98 hours.				

$$\frac{\% \text{ deposition on Zircaloy-2 at pH 10}}{\% \text{ deposition on AISI 304 stainless steel at pH 10}} \times \% \text{ deposition on AISI 304 stainless steel at pH 7}$$

$$\frac{3.9}{0.5} \times 1.3 = 10.14\% \text{ deposition expected on Zircaloy-2}$$

This extrapolation, based on the perhaps unwarranted assumption that the ratio is independent of pH, yields a predicted deposition on Zircaloy-2 in the range found in the Bettis Plant work. This agreement may well be accidental.

A comparison of other data from the two studies is given in Table III. The release rates at pH 7 are markedly different. The reason for this difference is not known, but it is felt to be primarily a temperature effect. The fact that crud deposited on the beam window (unobserved by previous experimenters) and failed to deposit on the samples (observed by previous experimenters) appears to indicate a greatly increased recombination rate of the free radicals formed during ionizing radiation as the water temperature is increased from 450°F to 600°F. Since the amount of crud reaching the filter and ion exchangers was greater at pH 10 than at pH 7, and the amount of crud deposited in the heat exchanger and associated piping is unknown, it may be that the more colloidal crud formed at pH 7 deposits more rapidly than the more particulate and filterable crud formed at pH 10. More work is needed before any definite conclusions can be drawn.

An exploratory run using AISI 410 stainless steel wool as a crud source was performed. Insufficient crud was released to the system to warrant further runs with this material.

TABLE III

COMPARISON OF RADIATION AND NUCLEONICS LABORATORY AND BETTIS PLANT DATA

pH and Site	Radiation	Total Iron Found; mg		Iron on Anion Resin; % ^a		Iron on Cation Resin; % ^a	
		Average	Range	Average	Range	Average	Range
pH 7 RNL	No	11.8 ^b		14.6 ^b		76.9 ^b	
pH 7 Bettis	No	8.85 ^c	8.27-9.45	3.3 ^c	1.2-3.2	70.0 ^c	52.1-96.6
pH 7 RNL	Yes	7.59 ^b		3.0 ^b		55.8 ^b	
pH 7 Bettis	Yes	12.42 ^c	7.99-20.12	1.6 ^c	0.1-2.6	56.6 ^c	48.1-72.8
pH 10 RNL	No	23.11 ^e	20.15-26.07	7.0 ^e	6.1-7.8	15.0 ^e	12.9-17.1
pH 10 Bettis	No	0.43 ^c	0.18-0.60	4.0 ^c	0.0-12.0	24.2 ^c	5.5-50
pH 10 RNL	Yes	18.42 ^f	15.43-21.41	6.3 ^f	6.0-6.5	18.5 ^f	8.8-18.1
pH 10 Bettis	Yes	0.59 ^g	0.35-0.83	5.0 ^g	0.0-10.0	4.9 ^g	1.2-8.6

pH and Site		Iron on Filter; % ^a		Iron on Specimen or Window; % ^a		Crud Release Rate; mg Fe/dm ² /mo	
		Average	Range	Average	Range	Average	Range
pH 7 RNL		8.5 ^b		none		0.12 ^b	
pH 7 Bettis		39.0 ^d	36.0-42.0	2.3 ^c	1.2-4.4	3.96 ^c	3.66-4.19
pH 7 RNL		39.9 ^b		1.3 ^b		0.1 ^b	
pH 7 Bettis		26.7 ^c	19.0-35.0	15.8 ^c	8.4-22.3	5.57	3.55-8.92
pH 10 RNL		78.1 ^e	75.1-81.0	none		0.30 ^e	0.27- .32
pH 10 Bettis		59.3 ^c	33.0-78.0	14.0 ^c	10.0-16.7	0.15 ^c	0.08- .23
pH 10 RNL		82.1 ^f	74.9-89.2	2.2 ^f	0.5-3.9	0.25 ^f	0.20- .30
pH 10 Bettis		46.5 ^g	33.0-60.0	43.4 ^g	31.4-55.4	0.25 ^g	0.14- .36

- Percentage of total iron found.
- One run only with AISI 304 stainless steel specimen and window.
- Average of three runs with Zircaloy-2 specimens.
- Average of two runs-filter from one run ruptured during run. Material on filter probably trapped on cation resin.
- Average of two runs - run CR-5 was not included - Zircaloy-2 or AISI 304 stainless steel specimens and windows.
- Average of two runs with Zircaloy-2 or AISI-304 stainless steel specimens and windows.
- Average of two runs with Zircaloy-2 specimens.

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