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**CORROSION AND BORON DEPOSITION
IN BORIC ACID SOLUTIONS**

G. R. BLOOM AND T. F. DEMMITT

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CORROSION AND BORON DEPOSITION IN BORIC ACID SOLUTIONS

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INTRODUCTION

The Plutonium Recycle Test Reactor (PRTR) has been modified to permit operation with a higher power density core. This change will expedite development of the technology associated with utilizing plutonium fuels in thermal power reactors. One of the critical requirements for this type of operation was to increase the shim capability for transient and long-term fuel burnup reactivity compensation. A chemical shim control system employing boric acid (H_3BO_3) as a neutron poison in the D_2O moderator offered substantial advantages over a mechanical control system for this application; this method of control will be used exclusively.

An extensive experimental program was conducted to assess the existence and significance of potential corrosion and boron deposition problems associated with using H_3BO_3 in the PRTR moderator system. The purpose of this report is to describe results of in-reactor, pilot plant, and laboratory tests designed to evaluate these potential problems.

SUMMARY

Materials representative of the PRTR moderator system have been exposed to boric acid solutions in pilot plant facilities and in the PRTR moderator system during the in-reactor chemical shim demonstration tests. Pilot plant tests of a PRTR dump valve, shim rod assembly, and process shroud-tube bellows assembly were performed. The pilot plant tests have demonstrated the potential for localized aluminum corrosion in the process shroud-tube bellows assembly. No other significant corrosion problems should result from using boric acid in the moderator system if (1) shim well refreshment

flow is guaranteed or the mechanical shim rods remain out of the reactor, (2) the boron concentration does not exceed 50 wppm B (weight parts per million boron), and (3) the moderator water purity is maintained within established control limits.*

Laboratory studies were performed to determine the amount of boron deposition that would occur on the moderator system surfaces. Boron deposition did not exceed three micrograms of boron per square centimeter of surface ($3 \mu\text{gB}/\text{cm}^2$) area during these laboratory tests at proposed PRTR operating conditions. The reactivity worth of these deposits (if 92% enriched $\text{H}_3^{10}\text{BO}_3$ were used) would be about 1.5 to 2.1 milli-k at full power with 18 to 20 wppm B in the PRTR moderator. This is well within the PRTR reactivity control capabilities.

In-reactor studies were performed to determine actual boron deposition and the effects on core reactivity during the in-reactor boron shim demonstration tests. Boron deposition measurements on specimens exposed in the PRTR calandria were less than $0.1 \mu\text{gB}/\text{cm}^2$. Reactor critical tests before and after in-reactor boron shim demonstration tests showed no measurable change in core reactivity.

DISCUSSION

The corrosion and boron deposition studies were conducted with "in-reactor," "pilot plant," and "laboratory" facilities and will be discussed under these headings.

IN-REACTOR TESTS

Experimental

In-reactor corrosion boron deposition and core-reactivity changes were measured during the boron shim demonstration tests to assess the effects of boric acid on the moderator system. The in-reactor corrosion tests were performed by

* Conductivity and pH control limits with boron present are:
Conductivity $< 3 \mu\text{mhos}/\text{cm}$ and pH of 6.0 to 7.5.

exposing specimens of the moderator system structural materials in the PRTR calandria. These specimens were charged prior to the in-reactor boron shim demonstration tests of July 1965 and were discharged periodically during the following 14 months. This exposure period for the specimens included the July 1965 in-reactor boron shim demonstration tests, the December 1965 calandria integrity test, and the September 1966 high power density core critical tests.

The in-reactor boron shim demonstration tests consisted of two tests. During the first, natural reagent grade boric acid (H_3BO_3) was injected into the moderator system as a concentrated solution to give a final concentration of 10.8 wppm B. This boric acid solution was recirculated for 12 hr, and the boron concentration was monitored to detect any decrease in dissolved boric acid as a result of deposition onto the oxide films present on the moderator system materials. The boric acid was then removed by ion exchange resins over a period of 55 hr. Eight days later, the boric acid was again injected into the moderator system to give a 11.1 wppm B solution, and the boric acid was removed over a period of two days during an actual cold startup of the reactor. Moderator critical-level measurements were made before and after the first boron shim test to measure any change in core reactivity and thus give an indication of the boron deposition during the test.

During the calandria integrity test in December 1965, a 20 wppm B solution of natural reagent grade boric acid in deionized light water was used as a chemical tracer for leak testing. The boron solution level in the calandria was maintained near the normal power operation level for three days and then drained from the reactor.

The PRTR critical tests of September 1966 involved the injection of 92% ^{10}B enriched reagent grade boric acid as a

chemical shim in the moderator system heavy water. The boron concentration was increased from about 2 wppm B to 20 wppm B over a one month period.

The material specimens consisted of aluminum, Inconel 600, 304 SS and Zircaloy-2 specimens, as well as aluminum to aluminum and aluminum to Inconel 600 couples, which were designed to provide crevice regions between the coupled specimens. Part of the aluminum specimens were prefilmed for two weeks in 80 °C deionized water prior to exposure in the reactor. The specimens were removed for examination after 5 days, 6 months, 10 months, and 14 months.

Specimen exposure to moderator water was dependent on the coupon holder position. During normal power operation of the PRTR, the moderator water level in the calandria is approximately 80 in. above the weir level, and is at the weir level during nonpower operation. Approximately 40% of the specimens placed in the calandria were below the weir level, and the remaining 60% were above the weir level. Those above the weir were in direct contact with the moderator water only during periods when the moderator level was raised, such as during power operation or critical tests. The coupons above the weir level were exposed to the highest neutron flux, and those below the weir level were exposed to lower flux levels.

Results

The specimens were dried, weighed, and examined under a microscope after discharge. Some of the specimens were defilmed in cleaning solutions and reweighed. The calculated uniform penetrations on the materials specimens as calculated from weight gain and weight loss data are given in Table I. There was no visible localized corrosion on any of these samples. Several 5052 aluminum coupons had tiny blisters in the oxide coating but no pitting or other obvious defects were visible under the blisters.

These results demonstrate that no accelerated corrosion occurred as the result of the various boric acid additions. This conclusion was confirmed by results obtained from a 6061 aluminum continuous monitor (probe) installed in the moderator piping, which underwent a corrosion penetration of 0.03 mils during this same period.

All evidence to date demonstrates that boron deposition onto the moderator material oxide films during in-reactor boron shim demonstration tests and the calandria integrity test were negligible. Aluminum specimens placed in the moderator system prior to the boron shim tests were discharged periodically over a 10-month period and analyzed for boron. The analysis was conducted by stripping the oxide layer off the specimen with 18% hydrofluoric acid, and then analyzing the strip solution for boron. All coupons analyzed had boron deposits of less than $0.10 \mu\text{gB}/\text{cm}^2$.

The continuous monitoring of the boron concentration during the first boron shim test showed no gross decrease of the boron concentration in the moderator system. A deposition of $1 \mu\text{gB}/\text{cm}^2$ would have given a 0.2 wppm B concentration decrease. The critical moderator level determinations before and after the first boric acid demonstration test detected no change in critical moderator level, and the moderator critical level was determined with a ± 0.1 in. uncertainty.

PILOT PLANT CORROSION TESTS

The primary objective of the pilot plant tests was to determine the corrosion behavior of PRTR components and materials in boric acid solutions. Pilot plant tests in the PRTR Shim Test Loop were conducted to evaluate potential localized corrosion problems at the Inconel-aluminum contact areas between the shim rods and shim recess wells, and at the aluminum-stainless steel joints at the flexible (bellows) seal between the process tube shrouds and the top of the calandria.

Pilot plant testing of a PRTR moderator dump valve was conducted to assure operation of the valve with the use of boric acid solutions in the moderator system.

Shim Rod Assembly Tests

Experimental Details

The shim rod assembly tests were primarily designed to evaluate potential corrosion problems to the 1/16-in. thick 6061 aluminum shim wells, since failure of these wells would cause loss of high purity heavy water from the moderator system. Test conditions of the five shim loop tests are given in Table II.

The PRTR Shim Test Loop consists of a storage tank, flow meter, and shim rod test section containing a PRTR shim rod. The loop is shown schematically in Figure 1. The shim rod test section simulates geometry and flow conditions to which the reactor shim rods are exposed. The inlet water to the shim rod test section entered about three feet above the bottom and exited near the top at a flow rate of 120 gal/hr. This relatively high flow rate simulated the high recirculation rate present in the reactor calandria.

A PRTR shim rod was placed in a vertical, glass test section with a force fitted 6061 aluminum sleeve during the first test and a 6061 aluminum pipe test section during the remaining four tests. An Inconel 600 shim on the shim rod was placed in direct contact with the aluminum well during each test to study galvanic and crevice effects.

There was a refreshment flow of 0.6 gal/hr to the bottom of the shim well during Test 3 and a refreshment flow of 2 gal/hr during Test 5. This flow was shut off during Tests 1, 2, and 4, and the bottom of the shim rod was tested in a stagnant well.

Results

Severe pitting up to 10 mils deep was found on the force fitted aluminum sleeve in the shim well at the end of the first shim loop test. Most of the pitting occurred at areas where the aluminum oxide film was damaged during the charging operation, but some pitting was observed on all areas of the interior surface except where the sleeve contacted the shim rod aluminum webbing.

It was determined that the aluminum pitting was caused by the presence of copper. Emission spectroscopy analysis of dissolved solids in the loop coolant samples taken during the test and the aluminum oxide mounds over pitted areas showed high concentrations of copper. The copper was attributed to the S-10 deoxygenator resin bed that was not followed by a mixed bed ion-exchange resin column. Laboratory corrosion tests confirmed that materials eluting from S-10 deoxygenator resin would cause accelerated localized corrosion in aluminum.

The 6061 aluminum sleeve and vertical glass test section were replaced with an etched 6061 aluminum pipe vertical test section during Tests 2 and 3. A boroscope examination of the shim well at the end of Test 2 revealed no visible pitting. This examination verified that contact was established between the aluminum shim well and the shim rod since a considerable number of long vertical scratch marks were visible in the well.

Destructive examination of the shim well test section at the end of the Test 3 revealed no significant general corrosion, but there were areas of minor localized corrosion and iron deposits where the shim rod webbing had scratched the shim well. A metallographic examination of the shim well test section showed a maximum aluminum penetration of 1 mil in the area where the shim well had been scratched during

charging operations of the shim rod. Photomicrographs of the shim well cross section are shown in Figure 2.

A new etched 6061 aluminum shim well was used during Test 4. Destructive examination of this shim well test section at the end of Test 4 showed localized aluminum corrosion where the shim rod assembly had contacted the shim well to form crevice regions and galvanic couples. There were 16 aluminum corrosion pits ranging between 1 and 5 mils deep when the well was examined with a microscope. A photograph of the most severe localized corrosion is shown in Figure 3 with a photomicrograph of the deepest pit.

A new 6061 aluminum shim well test section was installed for loop Test 5. Destructive examination of this test section at the conclusion of Test 5 showed no visible localized corrosion. A photograph of the shim well surface and a photomicrograph of a cross section through the well are given in Figure 4.

The results of shim loop Tests 2 and 3 demonstrated there would be no corrosion problems to the PRTR shim wells using either 50 wppm B in the moderator with 0.6 gal/hr refreshment to the well or 10 wppm B with no flow to the shim well.

Test 4 has demonstrated the potential for a serious corrosion problem to the shim wells with use of 50 wppm B solution in the moderator system and no refreshment flow to the bottom of the shim wells. The loop coolant was neither deoxygenated nor purified during shim loop Test 4. The corrosion product buildup in the loop coolant eventually increased the conductivity to 10 μ mhos/cm at the end of the test. These test conditions were definitely "worst case" conditions. The mechanical shim rods should not be placed in the reactor when boric acid solutions near 50 wppm B were used if refreshment flow to each shim well cannot be guaranteed. One method of

assuring flow to each shim well would be to install a flow meter on each of the 18 shim well refreshment lines.

During Test 5 the shim rod assembly and shim well were tested with a refreshment flow of 2 gal/hr to the bottom of the shim well. The loop coolant was continuously deoxygenated and deionized. Under these "ideal" operating conditions of refreshment flow and high water quality, no corrosion problems were observed in the shim well.

No serious corrosion was observed on the mechanical shim rod and its components during any of the pilot plant tests. A few very small pits were observed on the aluminum components of the shim rod after the first test. This pitting did not progress with additional exposure in the remaining four shim loop tests and is not considered serious.

Process Shroud-Tube Bellows Assembly Test

Experimental Details

A PRTR process shroud-tube bellows assembly was bolted to the top of the shim loop storage tank during shim loop Tests 2 through 5. The primary concern of this component was the 6063 aluminum process shroud tube which contacts the 321 (SS) bellows (through a thin titanium plated joint) forming a galvanic couple and crevices in the moist helium atmosphere present in the upper portion of the calandria. The technique used to join the aluminum and stainless steel actually involved first plating the stainless steel surface with a thin layer of titanium and then brazing the aluminum to the titanium. As a final step, the excess titanium was then removed. This joint is hereafter referred to as the shroud tube bellows joint.

Results

No significant corrosion was observed on the shroud tube after Test 2. The shroud tube bellows assembly was removed

from the shim loop storage tank and examined after 23 days exposure over a 10 wppm B solution. The only evidence of corrosion was an accumulation of aluminum corrosion product between the bottom ring of the bellows and the internal shroud tube section, and a small number of shallow pits on the shroud tube section where aluminum-aluminum crevices had been formed.

Examination of the same shroud tube after additional exposure during shim loop Tests 3 and 4 revealed localized corrosion but no significant general corrosion. The areas of localized corrosion were located where the 6063 aluminum shroud tube had contacted the 321 SS bellows forming a crevice region and galvanic couple. About 20 aluminum pits ranging from 1 to 5 mils deep were observed when the shroud tube was examined with a microscope. The shroud tube outer surface and the localized corrosion are shown in Figure 5. The aluminum pits observed at the end of Test 2 did not progress during Tests 3 or 4. One 5 mil pit actually formed a protective oxide layer with the continued exposure.

A new shroud tube section was installed before Test 5. After Test 5 was concluded, the section was examined and localized corrosion similar to that observed on the previous shroud tube was evident. A photograph of the shroud tube surface and a cross section of the pitted region are given in Figure 6. There was a total of about 40 aluminum pits observed in the aluminum shroud tube stainless bellows contact area when microscopic and metallographic examinations were made.

Both pilot plant tests demonstrated the potential for localized corrosion in the 6063 aluminum-321 SS contact areas of the bellows assembly under the pilot plant test conditions.

Special tests were conducted in 1964 and 1966 to evaluate gas transfer between the core blanket and gas balance

helium systems. Results of the tests indicated that transfer can indeed occur between these systems. The reason for this transfer is not definitely known, but it could involve leakage at the shroud tube bellows assembly.

The pilot plant bellows assembly test was conducted under worst-case conditions present in the reactor. During the pilot plant test the the bellows assembly was neither cooled nor heated, but was maintained at room temperature. The temperature of the shroud tube bellows assembly would be cooler during nonpower operation and warmer during power operation. Cooler operation would give more refreshment from condensate rinsing action. Higher temperature operation should keep the bellows assembly dry. Either condition would decrease the probability for localized corrosion.

Pilot Plant Materials Test

Experimental

Specimens representing the moderator system materials of construction were charged to the Shim Test Loop storage tank during the shim loop tests to assess the effects of boric acid solution on all moderator system materials.

Aluminum, stainless steel, carbon steel, Inconel 600, and Stellite 6 specimens were charged prior to each shim loop test and were discharged periodically. The specimen surfaces were examined under a microscope for localized corrosion after discharge. The oxide films formed during the test were chemically removed and the specimen weighed. Uniform penetrations were calculated from the weight difference between original weight before exposure and final weight after oxide film removal.

Results

Calculated uniform corrosion penetrations on specimens exposed during the first four Shim Loop Tests are given in

Table III. Pitted specimens discharged during the first two loop tests included two of the 6063 aluminum coupons that were coupled to 304 SS, one of the three 410 SS coupons, and all of the A-245 carbon steel and 416 SS coupons. The A-245 carbon steel and 416 SS coupons were very rusty, and the corrosion rates were high enough to limit their use in contact with boric acid solutions.

The 410 SS and 416 SS are common valve materials. Any valves installed in the moderator system containing these materials would be expected to lose about 1.5 mil per year when a 10 to 20 wppm B solution is present. The potential for localized corrosion in the process shroud tube bellows assembly was demonstrated by pitting of the aluminum to stainless steel couple. Uniform corrosion rates of the remaining alloys were acceptable.

Corrosion specimens charged prior to Test 3 were discharged periodically during the three months of shim loop Test 3 and the first month of shim loop Test 4. A 6061 aluminum specimen galvanically coupled to Inconel 600 had severe pitting in the crevice region after two months exposure. A photograph of the aluminum specimen surface is given in Figure 7. A metallographic photograph of the 20 mil deep penetration is given in the same figure.

The aluminum-Inconel couple simulated an Inconel 600 shim on a mechanical shim rod contacting a 6061 aluminum shim well. The pitting observed on the specimen couple was also observed on the pilot plant shim rod test. The water during shim loop test was purposely not refreshed, and the conductivity increased from a value of 1 to 10 $\mu\text{mhos/cm}$ as the corrosion product concentration increased. These were definitely worst-case conditions that might be present during off standard operating conditions such as shutting off the moderator cleanup ion exchangers or stopping refreshment flow to the shim wells.

A cast aluminum specimen developed minor pitting after two months exposure. A metallographic examination showed maximum penetrations of 1 mil after 55 days exposure. Additional cast aluminum coupons discharged at subsequent periods demonstrated that the pitting had terminated. Although the initial corrosion rate was high, the cast aluminum parts would be massive, and this amount of corrosion is not serious.

Additional aluminum-Inconel couples and aluminum-Teflon specimen assemblies with crevice areas were charged to the loop during shim loop Test 4. Aluminum pitting in the crevice area was found in each assembly after only two months exposure. Specimen examination results for these coupons are given in Table IV. This type of crevice corrosion was also observed on the shim well in loop Test 4 after five months exposure. The conductivity ranged between 3 and 10 μ mhos/cm during shim Test 4 with the boron concentration at 50 wppm B. The severe localized corrosion in the samples with crevices demonstrates that operation without refreshment or purification can be tolerated for only short periods. All the carbon steel coupons had minor localized pitting corrosion, and a maximum calculated uniform penetration of 2 mils in four months.

Specimens charged to the storage tank during Test 5 consisted of individual and galvanically coupled specimens with crevice areas. Calculated uniform penetrations and coupon pitting are given in Table V. The aluminum to aluminum, aluminum to Inconel 600, aluminum to aluminized Inconel 600, and aluminum to stainless steel couples had no localized corrosion. However, the stainless steel-aluminum couple exposed above the water level contained localized aluminum corrosion after four months testing. A photograph of the pitted aluminum specimen is given in Figure 8. The maximum aluminum penetration was 7.5 mils as shown in the

pit cross sections of Figure 8. The pitting of this aluminum specimen was similar to that of the aluminum shroud tube during the same loop test and is discussed in that section.

Calculated uniform corrosion penetrations and localized corrosion descriptions are tabulated in Table VI for the individual specimens exposed during Test 5. Only the cast aluminum and carbon steel specimens had localized corrosion. The pitting of the cast aluminum was very minor and did not progress after the first two months. The carbon steel specimens underwent severe pitting, particularly those specimens placed in the vapor space of the storage tank. Corrosion rates of the 6061 aluminum and 17-4 PH SS were acceptable.

The refreshment flow and high water quality maintained during shim loop Test 5 prevented the localized corrosion observed on coupled specimens immersed in loop coolant during the earlier loop tests. There was no localized corrosion observed on the pilot plant shim well during shim Test 5. Both test results demonstrate that water purity and refreshment must be maintained in the shim wells if the Inconel shims are driven into the shim wells.

The carbon steel specimens underwent severe localized corrosion in every shim loop test. Carbon steel should not be used in the moderator system except for very short periods.

Results of these material tests have demonstrated that present moderator structural materials will not undergo localized or accelerated corrosion if (1) shim well refreshment flow is guaranteed when the Inconel 600 shims are driven into the shim wells, (2) boron solutions with 50 wppm B or less are used in the reactor moderator system, and (3) the moderator water purity level is maintained within established control limits.

The present PRTR moderator water quality limits are:

pH	6.0 to 7.5
Conductivity	<3.0 μ mhos/cm
Chloride	<0.1 wppm
Fluoride	<0.2 wppm
Mercury	<0.01 wppm

No corrosive effects by oxygen concentrations below 1.6 wppm in the boric acid solutions were observed. However, upper permissible oxygen concentrations were not determined, and the effects of higher oxygen concentrations are not known. The oxygen concentrations in the PRTR moderator water is normally low because corrective action is taken if the helium cover gas (gas balance system) contains more than 1% nitrogen plus oxygen.

Pilot Plant Dump Valve Test

A PRTR dump valve was tested to assure operation of the 17-4 PH SS actuator spring during use of boric acid in the moderator system.

Experimental

The dump valve mock-up facility consisted of an actual PRTR dump valve with a small pipe reservoir bolted onto the bottom of the valve. The pipe reservoir under the valve was filled with boron solution and a positive helium pressure was maintained on the valve body. A photograph of the dump valve is given in Figure 9. The 17-4 PH SS actuator spring was part of the original PRTR equipment and was removed in May 1963. The spring was decontaminated prior to these tests. A carbon steel seat spring was used during the first dump valve test and part of the second test. This spring was replaced with a 316 SS seat spring, identical to those used in the reactor, during the remaining test periods.

The valve was tested in the moist helium atmosphere over the boric acid solution maintained at 60 °C. Periodically the valve springs were wetted with the boric acid solution and the valve actuated.

The dump valve was dismantled and fluorescent penetrant tested during and at the end of each test. Test operating conditions are given in Table VII.

Results

During exposure of one month over 10 wppm B solution, seven months over 50 to 100 wppm B solution, and five months over 160 to 230 wppm B solution, the valve operated normally. At the test conclusion, the valve was dismantled and the springs examined by the magnetic particle and fluorescent penetrant tests. These tests showed no defects in the springs other than small surface imperfections. Similar results were obtained by the fluorescent penetrant examinations conducted during the tests. There were areas of rust deposits on the 17-4 PH SS actuator spring but no areas of localized corrosion. Photographs of the springs after the completed test series are given in Figure 10.

The dump valve bellows stem nut constructed of 4140 carbon steel contained localized corrosion on the outer surface and a cross section of an outer thread are given in Figure 11. Cross sections of inside threads are given in Figure 12. Maximum pit depth is about 5 mils. This amount of corrosion would limit the service of the bellows stem nut. As the dump valves are periodically dismantled and inspected these nuts should be replaced. Materials such as 17-4 PH and 304 SS have been proven corrosion resistant to boric acid solution in the expected concentration ranges and would be candidate replacement materials. The final selection of a replacement material would have to also consider strength requirements of the bellows stem nut.

LABORATORY TESTS

Boron Deposition

Laboratory studies were performed to determine the amount of boron deposition that would occur on moderator system surfaces, to permit evaluation of the effect of these deposits on core reactivity. These tests were performed by exposing large surface area specimens in the form of 1/4 in. tubing to a relatively small volume of standardized boric acid solution in a closed glass test loop. The solution was circulated during these tests and boron deposition was followed by periodically withdrawing aliquots of the solution for boron analysis. Deposition values were then calculated, assuming that the boron depletion in the solution represented uniform deposition on the exposed metal surfaces.

The tests were performed at various solution boron concentrations and temperatures and with both 6063 aluminum and 304 SS tubing as deposition surfaces. Two initial aluminum surface conditions were studied: (1) defilmed specimens treated in sodium hydroxide to remove the surface oxide, and (2) filmed specimens exposed to 100 °C deionized water for ten days. Two initial stainless steel surface conditions were also investigated: (1) as-received after degreasing in acetone, and (2) filmed for ten days in 100 °C deionized water.

The results of the boron deposition tests are summarized in Table VIII. Note that deposition generally increased with temperature and boron concentration, and that results were quite similar for both aluminum and stainless steel. There was no measurable difference in deposition between the filmed and unfilmed specimens for either aluminum or stainless steel--apparently the films were not thick enough to cause a measurable effect. The values shown represent equilibrium conditions which were established within two days after the tests were started.

When the boron deposition tests were completed, the aluminum specimens from the 98 wppm B tests were rinsed in deionized water and then exposed in the glass loop to deionized water at 140 °F for four days to determine whether boron desorption would occur. Solution boron concentration measurements indicated that less than 1 $\mu\text{gB}/\text{cm}^2$ was released, and it was concluded that the deposits formed essentially irreversibly under these test conditions. Similar tests failed to indicate any boron desorption from the stainless steel specimens.

The effect of boron deposits on PRTR core reactivity was estimated based on the data presented in Table VIII. For an assumed maximum boron concentration of 40 wppm, and a moderator temperature of 140 °F, the maximum deposition should be about 3 $\mu\text{gB}/\text{cm}^2$. This corresponds to about 3.5 grams boron deposited on the shroud tube and calandria surfaces. If 90% enriched $\text{H}_3^{10}\text{BO}_3$ is used as the chemical shim additive, the deposited boron would have a reactivity effect of about 3.5 milli-k. This should decrease to 1.5 to 2.0 milli-k by the time the reactor achieves programmed full-power operation with a boron concentration of 18 to 20 wppm in the moderator. These values are well within the PRTR reactivity control capabilities and should present no significant reactor operation problems.

APPENDIX A

FIGURES

FIGURE 1. PRTR Shim Test Loop

*Test Conditions: 23 days in uncirculated 10 wppm B at 60 °C
50 days in circulated 50 wppm B at 60 °C*

FIGURE 2. *Shim Well Cross Sections
After Test II and III, 100 X*

Shim Well Surface at 6X

Shim Well Cross Section A-A at 100X

Test Conditions: 147 days in circulated 50 wppm at 50 °C

FIGURE 3. *Shim Well Examination after Test IV*

Shim Well Surface at 6X

Shim Well Cross Section A-A at 100X

*Test Conditions: 121 days in uncirculated
50 wppm B solution at 50 °C*

FIGURE 4. *Shim Well Examination after Test V*

Process Shroud Tube Surface at 6X

Process Shroud Tube Cross Section A-A at 100X

*Test Conditions: 23 days over 10 wppm B solution at 60 °C
 197 days over 50 wppm B solution at 60 °C*

FIGURE 5. *Process Shroud Tube after Tests II, III, and IV*

Process Shroud Tube Surface at 6X

Process Shroud Tube Cross Section A-A at 100X

Test Conditions: 121 days over 50 wppm B solution at 50 °C

FIGURE 6. *Process Shroud Tube After Test V*

6061 Aluminum Surface at 2X

6061 Aluminum Pit Cross Section A-A at 10X

Test Conditions: 90 days in 60 °C 50 wppm B solution

FIGURE 7. *6061 Aluminum-Inconel 600 Galvanic Couple with Crevice Area*

6061 Aluminum Specimen Surface at 5X

6061 Aluminum Pit Cross Sections Through A-A, 100X

*Test Conditions: 121 days suspended over 50 °C,
50 wppm B solution*

FIGURE 8. *6061 Aluminum-304 SS Galvanic
Couple with Contact Crevice*

FIGURE 9. Pilot Plant Dump Valve Facility

17-4 PH SS Activator Spring at 1/5X

Test Conditions:

*36 days over 10 wppm B solution
189 days over 50-100 wppm B solution
200 days over 160-230 wppm B solution*

316 SS Seat Spring at 1/5X

Test Conditions:

*25 days over 50-100 wppm B solution
200 days over 160-230 wppm B solution*

FIGURE 10. PRTR Dump Valve Springs

Outside Surface at 1-1/2X

Outside Thread at 50X

Test Conditions: 36 days over 10 wppm B solution
 189 days over 50 to 100 wppm B solution
 200 days over 160 to 230 wppm B solution

FIGURE 11. *PRTR Dump Valve Bellows Stem Nut*

Inside Thread at 50X

Inside Thread at 50X

Test Conditions: 36 days over 10 wppm B solution
 189 days over 50-100 wppm B solution
 200 days over 160-230 wppm B solution

FIGURE 12. PRTR Dump Valve Bellows Stem Nut Inside Threads

APPENDIX B

TABLES

TABLE I. *In-Reactor Corrosion Tests*

<u>Material Specimens Above Weir Level</u>	<u>Calculated Uniform Penetration, mils</u>	<u>Exposure, mo</u>
5050 A1	0.028	14
5052 A1	0.028	14
6061 A1	0.034	14
6061 A1 (prefilmed) (a)	0.040	6
6063 A1	0.035	14
<u>Material Specimens Below Weir Level</u>		
5050 A1 coupled to 6061 A1	0.032	14
5050 A1 coupled to 6063 A1	0.035	14
5052 A1	0.042	14
5052 A1 coupled to 6061 A1	0.034	14
5052 A1 coupled to 6063 A1	0.033	14
6061 A1 (prefilmed) (a)	0.043	6
6061 A1	0.046	14
6061 A1 coupled to 5050 A1	0.048	14
6061 A1 coupled to 5052 A1	0.050	14
6061 A1 coupled to Inconel 600	0.083	10
6063 A1 coupled to 5050 A1	0.040	14
6063 A1 coupled to 5052 A1	0.036	14
Inconel 600	<0.001	10
Inconel 600 coupled to 6061 A1	<0.001	10
Zircaloy-2	<0.001	10
304 SS	<0.001	10

(a) Prefilming involved exposure to 80 °C deionized water for 2 weeks.

TABLE II. *PRTR Shim Test Loop Operating Conditions and Water Quality*

Loop Operating Conditions	Test Number				
	I	II	III	IV	V
Test Duration, days	14	23	50	147	121
Reservoir Temperature, °C	60	60	60	60	50
Shim Well Temperature, °C	-	-	-	-	22-38
Loop Circulation to Middle of Shim Rod, gal/hr	120	120	120	120	120
Shim Well Circulation to Bottom of Shim Rod, gal/hr	0	0	0.6	0	0.9-2.4
Helium Pressure Above Reservoir, in. H ₂ O	1/2	1/2	1/2	1/2	1/2
Make-up Water Deionized	Yes	Yes	Yes	Yes	Yes
Make-up Water Deoxygenated	Yes	No	No	No	Yes
Loop Purification, hr	6	0	0	0	Continuous at 2 gal/hr
<u>Water Quality</u>					
Reservoir, pH	5.4-6.5	5.8-6.4	4.4-5.7	4.4-5.8	5.8-7.1
Shim Well, pH	-	-	4.4-5.7	-	5.8-7.1
Reservoir Boron Concentration, wppm B	25-50	9-11	37-54	38-50	52-82
Shim Well Boron Concentration, wppm B	-	-	37-54	-	50.80
Reservoir Conductivity, μmhos/cm	2.1-23.8	1.5-2.8	1.6-9.2	4.6-10.0	0.6-2.8
Shim Well Conductivity, μmhos/cm	-	-	1.6-9.2	-	0.8-1.2
Reservoir O ₂ , wppm O ₂	-	2.1-3.6	-	-	0.8-1.64
Shim Well O ₂ , wppm O ₂	-	-	-	-	0.2-0.72

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TABLE III. *Material Corrosion in 10, 20,
and 50 wppm Boron Solutions at 60 °C*

Material	Maximum Calculated Uniform Penetrations, mil			
	1 (a) Tests and 2 (b)	Pitting (d)	3 Tests and 4 (c)	Pitting (d)
Boron Concentration, wppm B	20 and 10		50	
A-245 Carbon Steel	0.85	(pitted)	2.01	(pitted)
Cast Aluminum	0.1	N	0.07	(pitted)
6061 Aluminum (coupled to Inconel)	-	-	0.05	(pitted)
6061 Aluminum	0.06	N	0.04	N
6063 Aluminum (coupled to 304 SS)	0.06	(pitted)	0.04	N
416 SS	0.12	(pitted)	0.04	N
5052 Aluminum	-	-	0.03	N
5050 Aluminum	-	-	0.02	N
Inconel-600 (coupled to 6061 Al)	-	-	<0.01	N
Inconel-600	<0.01	N	-	N
17-4 PH SS	<0.01	N	<0.01	N
304 SS	<0.01	N	<0.01	N
304-L SS	<0.0	N	<0.01	N
316 SS	<0.025	N	<0.01	N
410 SS	<0.01	(pitted)	<0.01	N
Stellite 6	<0.01	N	<0.01	N
Teflon	<0.01	N	<0.01	N

(a) 14 days in 20 wppm B

(b) 23 days in 10 wppm B

(c) 120 days in 50 wppm B

(d) N = no pitting

TABLE IV. Galvanic Couple-Crevice Tests in Unrefreshed
50 wppm Boron Solution at 60 °C

Specimen	Specimen Assembly Type	Exposure Period During Shim Loop Test IV					
		2 Months			3 Months		
		Weight Loss, mg/dm ²	Calculated Penetration, mils	Pitting ^(a)	Weight Loss, mg/cm ²	Calculated Penetration, mils	Pitting ^(a)
Inconel-600 6061 Aluminum	Crevice Couple	1.24	0.001	N	3.46	0.003	N
		17.3	0.046	>10 LC	21.0	0.056	2 LC
Inconel-600		1.73	0.001	N	1.24	0.001	>10 MC N
Inconel-600 6061 Aluminum	Crevice Couple	1.48	0.0013	N	0.98	0.0008	N
		20.5	0.054	>10 LC	16.3	0.043	3 LC
Inconel-600		1.73	0.0149	N	1.48	0.0013	N
6061 Aluminum	Crevice Couple	11.86	0.031	>10 LC	13.8	0.037	3 SC
		0.98	0.0008	N	1.24	0.0011	N
6061 Aluminum		9.14	0.024	2 LC	16.1	0.043	6 LC
6061 Aluminum	Crevice Couple	8.9	0.024	4 LC	14.8	0.039	>10 LC
		1.24	0.0011	N	1.24	0.0011	N
6061 Aluminum		12.11	0.032	4 LC	8.4	0.022	1 MC
6061 Aluminum	Crevice	7.91	0.021	N	8.4	0.022	N
Teflon		0.00	0.000	N	0.00	0.00	N
6061 Aluminum		7.91	0.021	2 LC	7.9	0.021	1 MC

(a) Description of Pitting

S - <1 mil deep
M - 1 to 4 mil deep
L - >4 mil deep
C - Crevice Region
N - None

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TABLE V. Galvanic Couple-Crevice Tests in Refreshed
50 wppm Boron Solution at 60 °C

Specimen	Type	Ratio	Phase	Exposure Period During Shim Loop Test V							
				1 Month		2 Months		3 Months		4 Months	
				Calculated Penetration, mils	Pitting ^(a)	Calculated Penetration, mils	Pitting ^(a)	Calculated Penetration, mils	Pitting ^(a)	Calculated Penetration, mils	Pitting ^(a)
6061 Al	Crevice	1	Liquid	0.05	N	0.05	N	0.08	N	0.09	N
6061 Al	Couple	1		0.05	N	0.05	N	0.07	N	0.08	N
6061 Al	Crevice	1	Liquid	0.03	N	-	-	0.07	N	0.08	N
Inconel-600	Couple	1		<0.001	N	-	-	<0.001	N	<0.001	N
Inconel-600	Crevice	1	Liquid	<0.001	N	<0.001	N	<0.001	N	<0.001	N
6061 Al	Couple	1		0.03	N	0.07	N	0.07	N	0.08	N
Inconel-600	Couple	1		<0.001	N	<0.001	N	<0.001	N	<0.001	N
6061 Al	Crevice	1	Liquid	0.03	N	0.05	N	0.06	N	0.07	N
Aluminized	Couple	1		-	N	-	N	-	N	-	N
Inconel-600											
6061 Al	Crevice	1	Liquid	0.04	N	-	-	-	-	0.08	N
304 SS	Couple	1		<0.001	N	-	-	-	-	<0.001	N
6061 Al	Crevice	1	Vapor	0.04	N	-	-	-	-	(b)	6 MC & 7 LC
304 SS	Couple	1		<0.001	N	-	-	-	-	<0.001	N
6063 Al	Crevice	1	Vapor	0.03	N	0.04	2 MC & 1 LC	0.05	2 MC	0.14	N
321 SS	Couple	1		-	-	-	-	-	-	-	-
6061 Al	Crevice	1	Liquid	0.04	N	0.05	N	0.08	N	0.09	N
Teflon	Couple	1		<0.001	N	<0.001	N	<0.001	N	<0.001	N

(a) Pit Depths and Description of Location

S - <1 mil deep
M - 1 to 4 mil deep
L - >4 mil deep
C - Crevice Area
N - None

(b) Coupon destructively examined

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TABLE VI. *Single Specimen Corrosion Test in Refreshed
50 wppm Boron Solution at 60 °C*

Specimen	Phase	Exposure Period During Shim Loop Test V							
		1 Month		2 Months		3 Months		4 Months	
		Calculated Penetration, mils	Pitting (a)	Calculated Penetration, mils	Pitting (a)	Calculated Penetration, mils	Pitting (a)	Calculated Penetration, mils	Pitting (a)
6061 Al	Liquid	0.005	N	0.01	N	0.01	N	0.01	N
A 245 C/S	Liquid	0.06	>10 L	0.06	>10 L	0.09	>10 L	0.19	>10 L
A 245 C/S	Vapor	0.43	>10 L	0.85	>10 L	1.34	>10 L	1.48	>10 L
17-4 PH SS	Liquid	0.003	N	-	-	-	-	0.014	N
17-4 PH SS	Vapor	0.003	N	0.004	N	-	-	0.003	N
Cast Al	Liquid	-	-	-	>10 S	-	-	-	>10 S

(a) *Pitting Description*
S - <1 mil deep
M - 1 to 4 mil deep
L - >4 mil deep
N - None

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TABLE VII. *PRTR Dump Valve Pilot Plant
Test Operating Conditions*

Test Conditions	Test Number		
	I	II	III
Test Duration, days	36	189	200
No. of Times Valve Actuated	22	82	85
No. of Times Springs Wetted With Boric Acid Solution	22	23	19
Reservoir Temperature, °C	57-63	57-63	58-71
Valve Body Temperature, °C	25	22-32	23-34
Valve Cover Gas He Pressure, in. H ₂ O	2	1/2-3	1/4-6
<u>Reservoir Water Quality</u>			
Boron Concentration, wppm	10	50-100	160-230
Conductivity, μmhos/cm	43.5	6.9-58.0	4.3-19.5
pH	6.4	6.2-6.8	5.9-6.1
O ₂ Concentration, wppm	-	-	0.84
<u>Examinations</u>			
Fluorescent Penetrant	2	2	1
Magnetic Particle	0	0	1

TABLE VIII. *Boron Deposition Information
from Laboratory Tests*

<u>Boron Deposition,</u> <u>$\mu\text{gB}/\text{cm}^2$</u>	<u>Boron</u> <u>Concentration,</u> <u>wppm</u>	<u>Temperature,</u> <u>$^{\circ}\text{F}$</u>	<u>Material</u>
3	98	74	6063 Al
2	52	74	6063 Al
2	36	74	6063 Al
1	10	74	6063 Al
4	98	140	6063 Al
2	10	140	6063 Al
3	102	76	304 SS
2	56	76	304 SS
2	12	76	304 SS
2	50	140	304 SS
1	10	140	304 SS

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