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DYNAMIC CORROSION STUDIES FOR THE HIGH FLUX ISOTOPE REACTOR

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1. ABSTRACT

The results of a testing program to examine the long-term corrosion behavior of materials of interest to the High Flux Isotope Reactor (HFIR) are presented, and their significance to the operation of the HFIR is discussed. The test environment was water, adjusted to a pH of 5.0 with nitric acid, at 100°C and at flow rates ranging from 0.5 fpm to 81 fps. Under all flow conditions, the aluminum alloys corroded at low rates, 0.2 mpy at the higher velocities and less than 0.1 mpy at the lowest velocities. Contacting aluminum with aluminum, beryllium, nickel, types 304, 416, 420, or 17-4PH stainless steel resulted in pitting of the aluminum in the contact areas only. While of appreciable depth, the pits were randomly spread and should not be of major consequence in the HFIR system. Beryllium, both by itself and in contact with other metals, was subject to light pitting attack. Pits did not exceed 13 mils in depth in tests that lasted in excess of one year. Within the velocity range of 13 to 81 fps, the average corrosion rate of beryllium was independent of velocity and constant at 2 mpy. One specimen of beryllium in contact with type 304 stainless steel developed evidence of stress-corrosion cracking. Commercial-grade nickel underwent shallow pitting attack and corroded at an average rate of 2 mpy. An electroless nickel deposit on 6061 aluminum corroded at the high rate of 19 mpy. Electrolyzed coatings (a proprietary process for coating materials with chromium) on stainless steel exhibited excellent corrosion resistance and good adherence to the stainless steel. Similar deposits flaked and peeled from aluminum surfaces. Hardened type 416 stainless steel was subject to blistering while type 420 stainless steel underwent stress-corrosion cracking. Delrin,

a polyformaldehyde plastic considered as a gasket material, suffered near-complete degradation after a short time in test. The results of this testing program and the experiences gained during operation of other water-cooled production and research reactors were fully utilized in the design of the HFIR. On this basis and assuming adequate control of the water chemistry, the HFIR should be free of major corrosion problems.

2. INTRODUCTION

A corrosion-test program has been conducted during the past several years to evaluate the corrosion resistance of a wide variety of materials considered for permanent or semipermanent installation in the HFIR. The program consisted of two phases, namely, static and dynamic types of testing. The static tests utilized conventional glass equipment and stainless steel autoclaves. Results of this phase of the program have been reported.[1] The dynamic tests, the subject of the present report, were carried out in a stainless steel pump loop.

One of the major objectives of the HFIR corrosion program was the procurement of long-term data on various materials. This report deals with such data for aluminum alloys, beryllium, stainless steels, and miscellaneous materials in pH 5.0 water at 100°C, over flow rates ranging from approximately 0.5 fpm to 81 fps.

3. EXPERIMENTAL

Corrosion tests were conducted in a type 347 stainless steel pump loop complete with parallel flow channels in which the specimens were exposed, a feed and letdown system, and bypass ion exchangers. Details of the facility and the means of temperature and flow controls have been described previously.[2]

The test medium was water adjusted to a pH of 5.0 with nitric acid at 100°C. During test a bypass stream was continuously passed through a cation exchange column in the hydrogen form. Any excess acid inadvertently added to the system was removed by temporarily passing the side stream through a separate mixed-bed ion exchanger. In this manner, the pH of the test solution was maintained within 0.1 pH unit of 5.0.

In the HFIR, some oxygen must be present in the coolant to prevent radiolytic decomposition of the small amount of nitric acid. In the tests, this condition was simulated by allowing the feed tank to be exposed to the air. With a feed and letdown rate of 3 liters per hour, a steady-state oxygen concentration of 4 to 5 ppm was maintained.

The test specimens were usually flat plates held in appropriate holders. The specimens were insulated from the holder and from each other with Teflon tape except in those cases where crevices or galvanic effects were investigated. In most of the latter cases, the assemblies were prepared by bolting flat plates of the materials together with a nut and bolt of the same material as one of the members of the particular couple.

Upon completion of the tests, specimens were defilmed to remove corrosion product. Aluminum and beryllium specimens were cleaned cathodically for 45 minutes in a 2 w/o CrO_3 - 5 w/o H_3PO_4 solution at 75°C. Stainless steels and other related materials were defilmed cathodically in inhibited 5 w/o H_2SO_4 solution for three minutes at 75°C. Both cleaning treatments resulted in negligible attack on the respective base metals.

Unless otherwise specified, all specimens were tested with either as-received or machined surfaces and were degreased in acetone and alcohol before exposure.

For 6061 aluminum specimens, the -T4 condition was obtained by heating for two hours at 413°C (775°F), furnace cooling to 260°C (500°F), air cooling to room temperature, and reheating for twenty minutes at 521°C (970°F) followed by air cooling to room temperature. The specimens were then pickled in warm 10 v/o HNO_3 - 3 v/o HF to remove oxide film. Material in the -T6 condition was received directly from the mill.

4. TEST RESULTS

4.1. Aluminum

Duplicate specimens of pickled 6061-T4 aluminum were exposed for 14,000 hours at flow rates between 2 and 3 fps. During test, both specimens showed slight weight increases. Defilmed corrosion rates were less than 0.1 mpy. All surfaces were free from pitting attack.

Duplicate specimens of 6061-T6 aluminum were exposed to each of four velocity ranges for 12,775 hours. The velocity ranges included 22 to 33, 33 to 51, 51 to 72, and 72 to 81 fps. Specimens exposed at flow rates up to 51 fps showed weight gains up to 0.2 mg/cm^2. Specimens exposed at 51 to 81 fps exhibited weight losses not in excess of 0.4 mg/cm^2. Final defilmed weight losses on all specimens were in excellent agreement with one another indicating no effect of velocity on corrosion. Final rates averaged 0.2 mpy and attack was uniform. The calculated film thickness at the end of the exposure was between 0.2 and 0.3 mil, assuming a density of 3.01 gm/cm^3 for the oxide,

$$\alpha \cdot Al_2O_3 \cdot H_2O \quad .$$

Similarly, no effect of velocity was found on the corrosion of X8001-F aluminum during an 11,775 hour period over the velocity range 22 to 51 fps. As-scrubbed weight losses at the end of the test varied between 0.2 and 0.3 mg/cm^2; defilmed weight losses were nearly the same for all specimens, averaging 2.6 mg/cm^2 (0.3 mpy). Attack was uniform. The calculated oxide thickness for the specimens was slightly over 0.3 mil.

The corrosion characteristics of two special cast aluminum alloys were determined at 2 to 3 fps. One alloy, designated Alloy 1 - Type B, had the following chemical composition: 5.8% Sn, 1.27% Cr, 0.96% Ni, 0.12% Si, and balance aluminum. The second alloy was identified as B-170 and contained: 5.0% Sn, 1.51% Cu, 0.84% Ni, 0.12% Si, 0.43% Fe, 2.5% Mg, 0.1% Ti, and balance aluminum. These alloys were considered as possible material for skids on the control plates. Since that time the decision was made to use roller bearings. Duplicate specimens of both alloys with abraded surfaces were exposed for 6390 hours. Alloy 1 specimens underwent substantial pitting with final pit depths measuring up to 37 mils. No corrosion rates were obtained since the standard defilming technique failed to remove the corrosion product. The B-170 specimens remained free from pitting. After defilming, the weight losses corresponded to a corrosion rate of 0.1 mpy.

Additional specimens of aluminum alloys were exposed in two other loops that were used to conduct experiments on the effect of heat flux on the corrosion of aluminum.[3-6] Eight specimens each of 1100-F, 6061-T6, and X8001-F aluminum were placed in the loop as "dummy" specimens to give an aluminum surface area-to-water volume ratio comparable to that in the HFIR. Whereas all the previously described aluminum corrosion data were obtained under constant conditions, the exposure conditions for these specimens changed substantially since test conditions for the heat flux - aluminum corrosion tests were varied. Generally the conditions were: (1) coolant pH from 4 (with nitric acid) to 7; (2) flow rate past specimens, 7.6 fps; (3) coolant temperature 90° to 250°C; and (4) total exposure time, 14,200 hours. A large majority of the tests were run at a pH between 5.0 and 6.0. One run of 382-hr duration was made at 250°C; otherwise, the maximum operating temperature was around 180°C. A summary of the corrosion information for the three alloys is give in Table I.

Table I. Corrosion of Aluminum Alloys by Water at 7.6 fps Over a Range of Water pH and Temperature

(Test Time: 14,200 hr)

Alloy Type	Average Defilmed Corrosion Rate* (mpy)	Observed Maximum Pit Depth (mils)
1100-F	0.16	6
6061-T6	0.19	7
X8001-F	0.14	7

*Average for eight specimens of each alloy type; deviation from average did not exceed 0.05 mpy.

There was no significant difference in corrosion rates among the three alloys; all corroded at rates of 0.1 to 0.2 mpy and all were subject to a mild pitting attack. There are no permanent components in the HFIR that will be exposed to conditions as severe as were these aluminum specimens. The only reason for including the results here is to show that even under more stringent conditions than will exist in the HFIR, 1100, 6061, and X8001 aluminum alloys had adequate corrosion resistance.

4.2. Beryllium

At least duplicate, and in some cases quadruplicate, specimens of reactor-grade, QMV beryllium were exposed at the following flow rates for the indicated times: 2 to 3 fps, 19,630 hr; 13 fps, 17,600 hr; 23 fps, 17,600 hr; 22 to 33 fps and 33 to 51 fps, 5,590 hr; 51 to 72 fps, 17,360 hr; and 72 to 81 fps, 17,360 hr. In all cases, the specimens developed only very thin, tarnish-like films which weighed only 0.1 to 0.2 mg/cm^2. All specimens were weighed several times during the exposures. At the lowest velocity (2 to 3 fps), the specimens corroded at a constant rate of 0.6 mpy. At all other velocities, the specimens corroded at the rate of 2.0 ± 0.1 mpy. Figure 1 shows a plot of weight loss versus time for specimens at the lowest and highest velocities tested. Very mild and infrequent pitting was first apparent on the specimens after 1000 to 3500 hours. With continued exposure, the pits deepened and the pitting frequency increased slightly. At the lowest velocity, the measured depths were about 8 mils after 17,600 hours. At the highest velocity after 17,360 hours, the maximum pit depth was essentially the same. Both the general corrosion rate of beryllium and its tendency to pit in water at a pH of 5 and at 100°C are such that the corrosion damage to the beryllium reflector in the HFIR should be inconsequential.

4.3. Other Materials

The behavior of commercial-purity nickel in pH 5.0 water at 100°C during a 7900-hr exposure period is shown in Fig. 2. For the first 2600-hr of the exposure the corrosion rate was 0.2 mpy. During the

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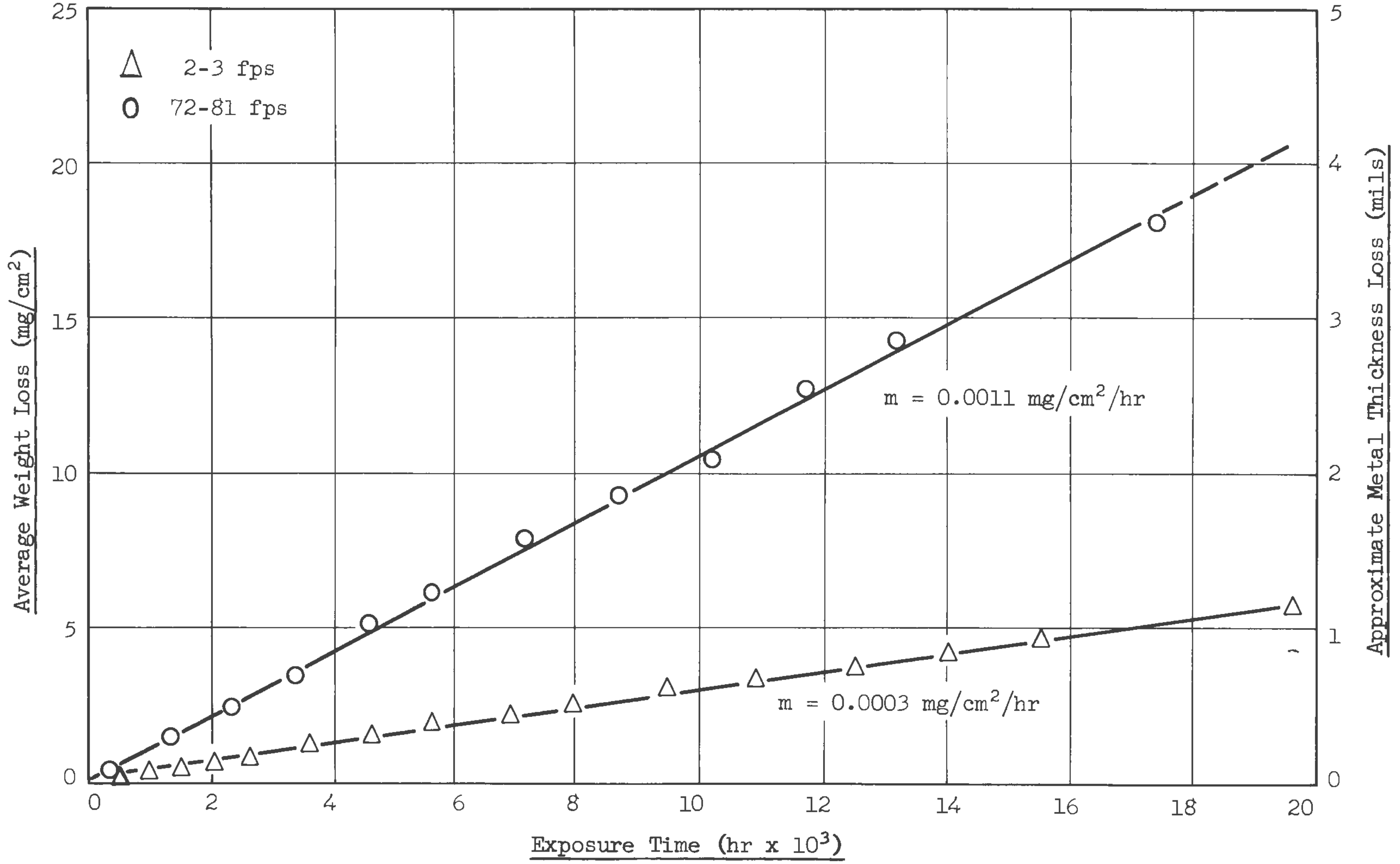

Fig. 1. Effect of Velocity on Corrosion of QMV Beryllium by pH 5.0 Water at 100°C.

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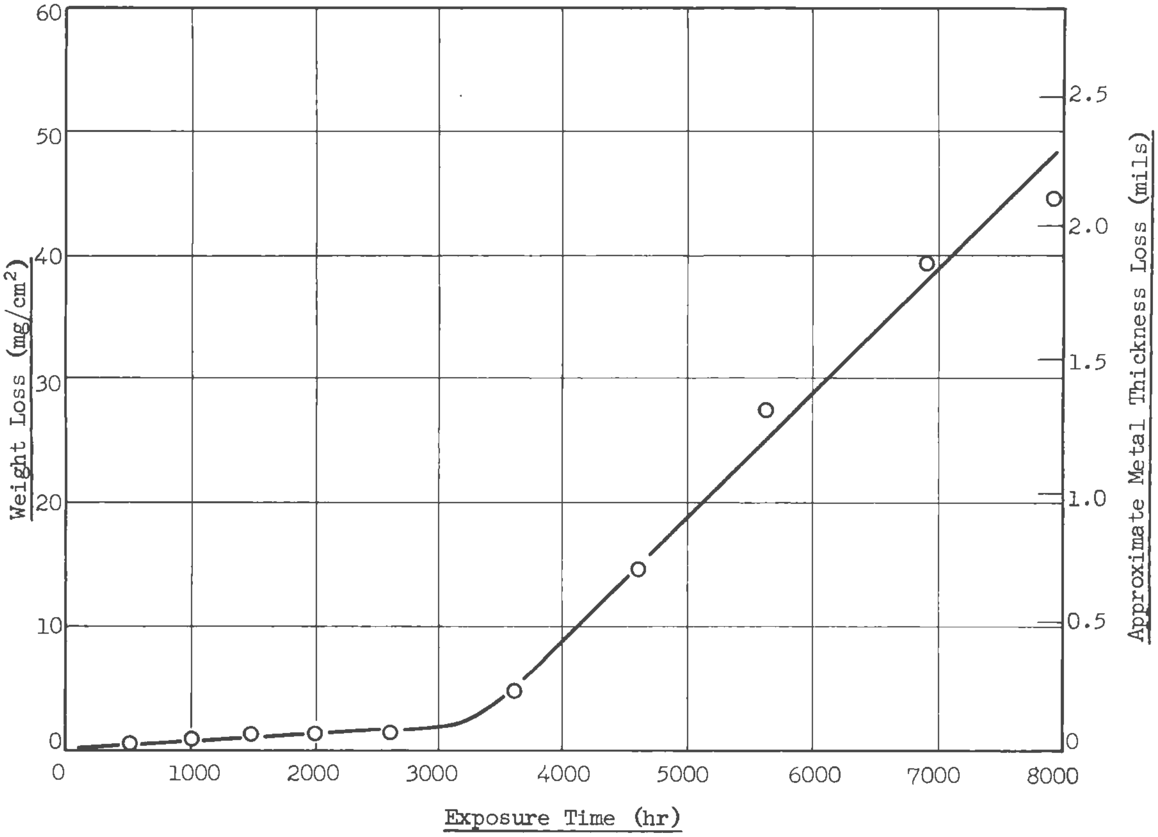

Fig. 2. Corrosion of Commercial-Purity Nickel in pH 5.0 Water at 100°C and 2-3 fps.

period between 2600 and 3100 hours, the corrosion rate increased to nearly 4 mpy and remained constant for the rest of the test. The average rate for the entire test was slightly greater than 2 mpy. Prolific shallow pitting took place during the first 1000 hours. By the end of the test, the depth of the attack had increased only slightly to 2 mils, but the frequency increased to such magnitude that it was impossible to measure the pit population per unit area.

One short test was conducted with metallic antimony. During a 505-hr exposure the specimen corroded at a rate of 213 mpy. This result showed that bare radioantimony could not be used as a neutron source in the beryllium reflector and pointed out the necessity of canning the antimony.

Other materials including the stainless steels were tested, but in most cases, these materials were coupled to aluminum or beryllium. These results will be discussed under crevice and galvanic corrosion tests (sections 4.4 and 4.5).

4.4. Flat-Plate Crevice and Galvanic Corrosion Tests

In this group of tests, flat plates of the materials under consideration were bolted together, usually with a nut and bolt of one of the materials under consideration. Since these specimens were exposed in various places in the loop, the flow rates past the specimens were not always the same. Although the flow rate past the specimens should be unimportant to the crevice areas, flow rate can influence the corrosion rate of the materials outside of the crevice. Table II summarizes the tests performed and the following paragraphs describe in more detail the results obtained.

An 11,970-hr exposure of two 6061-T4 aluminum plates bolted together (Test No. 1) resulted in pitting on the contact faces only. The plates were separated several times during the test for examination. At these intermediate examinations, the specimens were brushed to remove the loose corrosion products. However, some corrosion product always remained and obscured the true pit depths. After 2100 hours, frequent pits up to approximately 4 mils deep were observed. These gradually

Table II. The Corrosion of Materials During Crevice and Galvanic Corrosion Tests in pH 5.0 Water at 100°C

Test No.	Materials in Couple	Surface Area Ratio	Exposure Time, hr.	Maximum Pit Depth, mils	Average Corrosion Rate, mpy
1	6061-T4 — 6061-T4	-	11,970	8	<0.1
2	QMV Be — 6061-T4[a]	1.6 (Al:Be)	12,475	6 (Al) 6 (Be)	<0.1 (Al) 0.4 (Be)
3	QMV Be — 6061-T4[a,b]	1.0 (Al:Be)	17,602	3 (Al) 9 (Be)	0.2 (Al) 2.0 (Be)
4	304 SS — 6061-T4	0.5 (Al:SS)	12,475	45 (Al)	0.5 (Al)
5	304 SS — 6061-T4[b]	0.5 (Al:SS)	18,130	14 (Al)	0.1 (Al)
6	Ni — 6061-T4	2.2 (Al:Ni)	5,560	54 (Al) 2 (Ni)	0.4 (Al) 0.7 (Ni)
7	Ni — 6061-T4[c]	0.5 (Al:Ni + SS)	5,560	54 (Al) 2 (Ni)	0.8 (Al) 0.9 (Ni)
8	Ni — 6061-T4[a,d]	1 (Al:Ni)	1,500	N.D.	
9	17-4 PH SS — 6061-T4 — 304 SS	1.3 (Al:SS)	19,625	25 (Al)	0.1 (Al)
10	304 SS — 6061-T4 — 420 SS	1.1 (Al:SS)	12,475	10 (Al) 4 (420 SS)	<0.1 (Al) 0.2 (420 SS)
11	QMV Be — QMV Be	-	11,000	4	0.3
12	304 SS — QMV Be[a]	1.3 (Be:SS)	12,475	8 (Be)	0.6 (Be)
13	304 SS — QMV Be[a,b]	1.0 (Be:SS)	11,980	13 (Be)	2.2 (Be)

[a] Duplicate

[b] Couple not dismantled until end of test.

[c] Stainless steel nut and bolt.

[d] Joined with Al rivets.

deepened during the remainder of the exposure reaching a maximum depth of 8 mils (after defilming to remove corrosion product) at the end of the test.

Duplicate couple specimens of beryllium and 6061-T4 aluminum (Test No. 2) exposed for 12,475 hours showed pitting of the aluminum only on the contact surfaces. These specimens were disassembled, brushed, and examined several times during the test. Approximately 0.5-mil-deep pits were present on the beryllium surface contacting the aluminum after about 500 hours. Only after 10,900 hours were pits first observed on the aluminum at which time they were about 2 mils deep. Upon completion of the test and defilming the specimens, maximum pit depths between 4 and 6 mils were found on the contacting surfaces of both beryllium and aluminum. The pitting attack on the beryllium was unique in that the pits collected in fairly large clusters as opposed to a random type of distribution on aluminum. These specimens were exposed at a flow rate of 2 to 3 fps and showed average corrosion rates comparable to individual specimens exposed under the same conditions.

Similar specimens (Test No. 3) were exposed for 17,602 hours, except in this case, the specimens were separated for examination only at the end of the test. The aluminum specimens showed pits only on the contact areas and reached a maximum depth of only 3 mils, in contrast to the 6 mils observed during a shorter test where the specimens were examined frequently (Test No. 2). Also the pits were fewer in number than observed in Test No. 2. Scattered pits were present on all beryllium surfaces with no concentration on contacting areas. Maximum pit depths were 7 to 9 mils, the same as observed on single beryllium specimens exposed for the same time. One of the couple specimens was exposed at 13 fps and the other at 23 fps. The average corrosion rate of both materials was essentially the same as that observed for individual specimens exposed under the same conditions.

The coupling of 6061-T4 aluminum to type 304 stainless steel resulted in pitting on the contacting aluminum surfaces (Tests No. 4 and 5). One of the couple specimens exposed for 12,475 hours was frequently removed from test, dismantled, scrubbed, and examined. The other specimen was exposed for a total of 18,130 hours without dismantling. Pits up to 4 mils deep were observed on the aluminum contact surface of the dismantled couple after an initial 500-hr period; after 2000 hours, pits up to 33 mils were observed. At the end of the test the maximum measured pit depth was 45 mils. The couple dismantled only at the completion of the test showed maximum pit depths of 14 mils on the aluminum. The stainless steel specimens were unaffected by the exposure. Thus, even though the exposure time was 5600 hours longer, the pits were only a third as deep on the undisturbed specimen as on the one which was frequently examined. Also the average corrosion rate of the aluminum was greater on the latter specimen than on the former. These results indicate that the corrosion products formed in a tight crevice substantially reduce corrosion in that area.

At one time bare nickel was considered as a material for part of the complex HFIR control plates. Had nickel been used, it would have been necessary to join it to 6061 aluminum. For this reason, four couple specimens were included in the test environment. As shown in Table II (Tests 6 and 7), coupling nickel to aluminum produces very deep pits, up to 54 mils during a 5510-hr test, on the contacting aluminum surface. Use of either a stainless steel nut and bolt (Test 7) or an aluminum nut and bolt (Test 6) made no difference in the result.

In other tests, (Test No. 8) specimens of 6061-T4 aluminum were riveted to nickel specimens, using two different aluminum alloys as rivets. In one case, the rivets were a copper-containing alloy (designation unknown) and in the second the rivets were 5056-F aluminum alloy. During the 1500-hr exposure period, both types of rivets underwent substantial attack resulting in swelling and the accumulation of copious quantities of corrosion product. In a few instances the attack was sufficient to cause a separation of the rivet head from the main body. In view of the severe corrosion, no attempt was made to examine the contacting surfaces. Fortunately, the corrosion problem was eliminated by a decision to use a material other than nickel in the HFIR control plates.

A composite specimen made by bolting a plate of aluminum between plates of type 304 stainless steel and type 17-4PH stainless steel was exposed for 19,625 hours (Test No. 9). The 17-4PH steel was hardened to 33 Rockwell C by heating for four hours at 621°C (1150°F) and air cooling to room temperature. After descaling the aluminum specimen at the end of the test, a maximum pit depth of 25 mils was found. The intensity and frequency of pits was the same on both contact faces. Neither the 304 or the 17-4PH stainless steel was affected by the exposure.

A second composite specimen, prepared by bolting a 6061-T4 aluminum specimen between plates of 304 and 420 stainless steel, was exposed for 12,475 hours (Test No. 10). The 420 stainless steel was hardened to 43 Rockwell C by heating fifteen minutes at 1010°C (1850°F), water quenching, and reheating for $2\frac{1}{4}$ hours at 538°C (1000°F) followed by air cooling to room temperature. The specimen was dismantled frequently during the test. The first indication of pitting on the aluminum appeared after 1000 hours at which time pits slightly over 3 mils deep were observed. With increased exposure time, these pits deepened - becoming as deep as 10 mils at the end of the test. After a 3600-hr exposure, pits up to 1 mil deep were observed on the type 420 stainless steel. Also the 420 stainless steel specimen showed substantial staining and accumulation of rust-colored corrosion products. The pits were confined to the surface contacted by the aluminum. At the end of the test the 420 stainless steel specimen showed scattered pitting slightly over 4 mils in maximum depth. No pitting was found on other faces but two cracks were detected on opposite surfaces as shown in Fig. 3. The larger crack (Fig. 4) extended approximately 90 mils into the metal; the smaller crack was about one half this depth. Metallographic examination showed the cracks to be predominately intergranular (Fig. 5). The average attack rate on the 420 stainless steel was 0.2 mpy.

The coupling of beryllium to itself produced no increase in pitting in the crevice region (Test No. 11). Pits about 4 mils deep were found randomly distributed on all surfaces. The average corrosion rate of the beryllium was about half that observed on uncoupled specimens exposed under the same conditions.

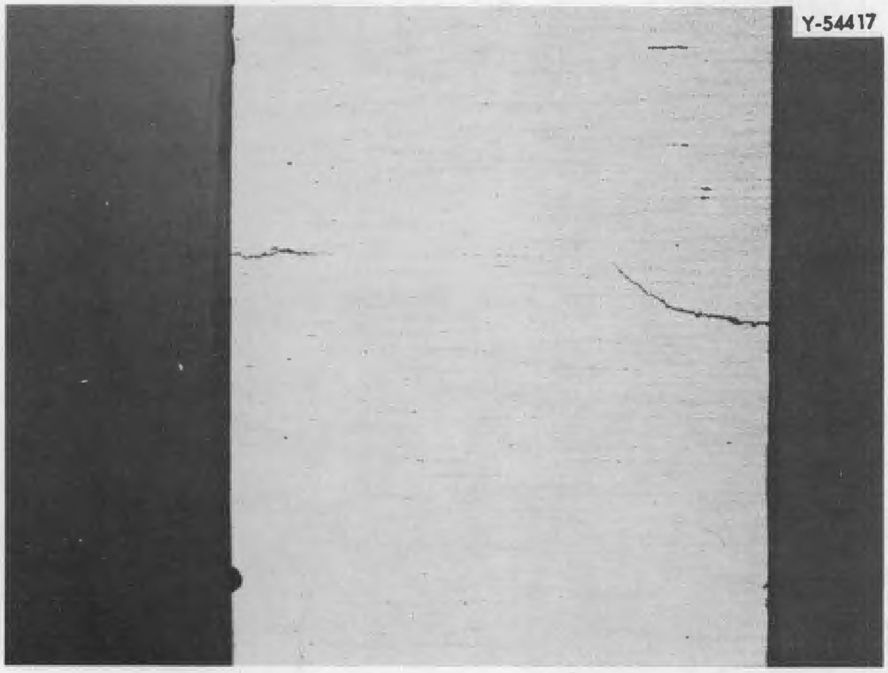

Fig. 3. Transverse View Showing Cracks in Hardened Type 420 Stainless Steel Specimen After 12,475 Hours in Boiling pH 5.0 Water. Etched in Glyceria. Magnification 9X.

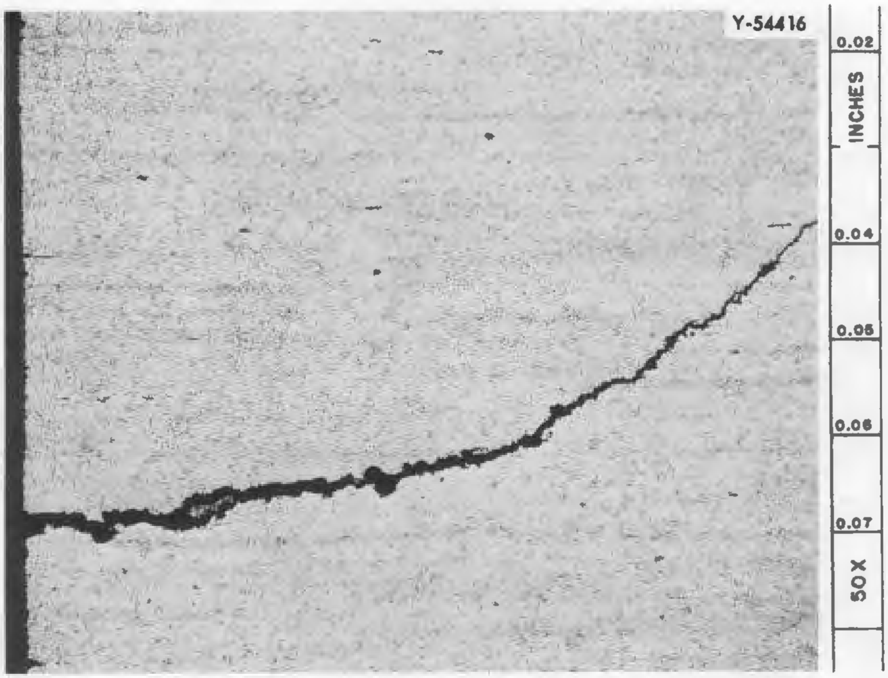

Fig. 4. Appearance of Larger Crack in Hardened Type 420 Stainless Steel Specimen. Etched in Glyceria. Magnification 50X.

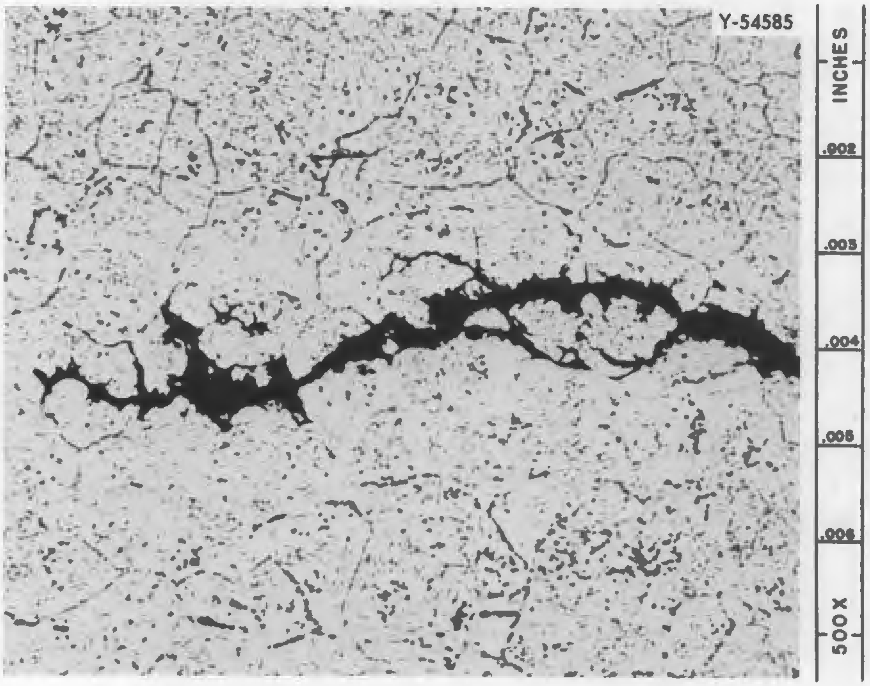

Fig. 5. Enlarged View Showing Predominant Intergranular Nature of Crack in Hardened Type 420 Stainless Steel Specimen. Electrolytic Oxalic Acid Etch. Magnification 500X.

The coupling of beryllium to type 304 stainless steel caused some increase in the pitting tendency of beryllium in the contact areas (Test No. 12 and 13). Comparison of tests 12 and 13 with 11 shows that for comparable times, pits were deeper when the beryllium was contacted with stainless steel than when contacted by itself. It should also be noted that the specimens not dismantled during test had deeper pits than those dismantled frequently. This behavior is opposite that observed when aluminum was coupled to stainless steel. The specimens in Test No. 12 were exposed at a flow rate of 2 to 3 fps whereas those in Test No. 13 were exposed at 13 and 23 fps. The average corrosion rate for the beryllium in the couples was the same as that for single specimens exposed under the same conditions.

During periodic examination of the beryllium - stainless couples, two cracks were found on one of the beryllium specimens after 10,440 hours of test. Prior to the beginning of the test and frequently up to this time all specimens were examined microscopically at 90X without evidence of cracks. The cracks originated from the edge of the specimen where identification numbers had been stamped. Figure 6 shows the general location of the cracks after the specimen has been separated from the stainless steel upon completion of the test. The four holes are sites where flat-headed stainless steel screws were used to bolt the two specimens together. It appeared that little, if any, extension of the cracks occurred during the balance of the test. Figure 7 shows a macrograph of one of the cracks extending into the flat surface of the specimen. Figures 8 and 9 show magnified views of the two cracks. The deeper crack (Fig. 8) penetrated 7 mils into the metal and the smaller one, 5 mils. A polarized light view disclosed that the cracks were transgranular (Fig. 10).

4.5. Multi-Material Crevice and Galvanic Corrosion Tests

Five multi-material assemblies, simulating some of the actual combinations of interest in the permanent components in the HFIR pressure vessel, were exposed to the test environment for various periods. The materials were sleeves and gaskets mounted on a bolt and held in place

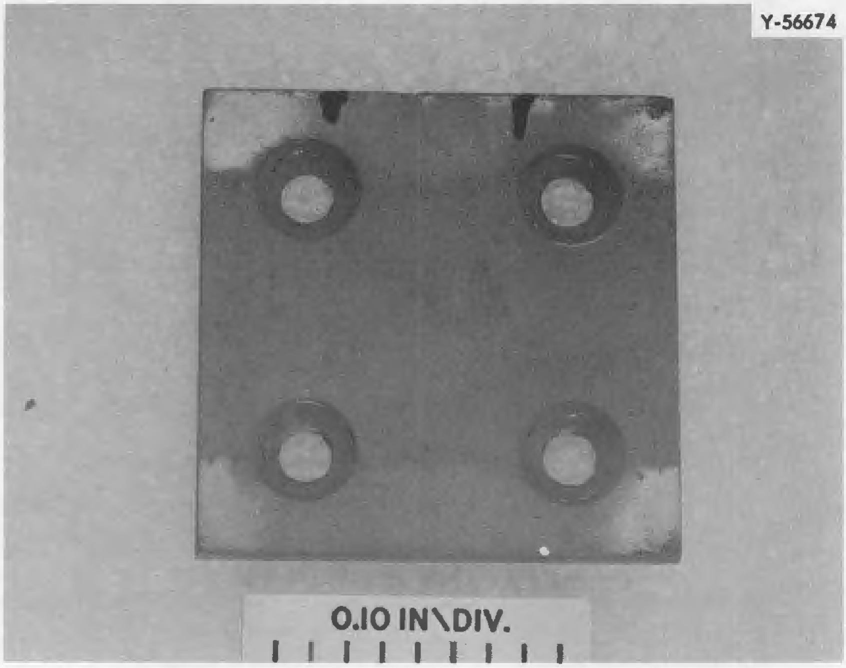

Fig. 6. Black-Marked Areas on Top of Specimen Show Approximate Location of Cracks on QMV Beryllium Coupled to Type 304 Stainless Steel. Total Exposure: 11,980 Hours at 100°C in pH 5.0 Water.

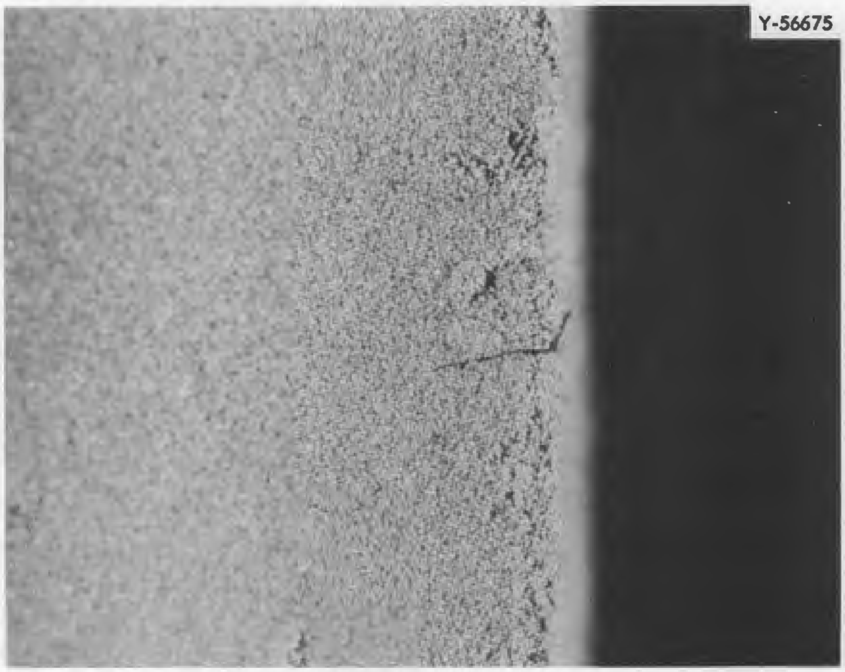

Fig. 7. View of One of Two Cracks in QMV Beryllium Specimen. Crack Originated on Edge and Extended Across Flat Surface. Defilmed. Magnification 10X.

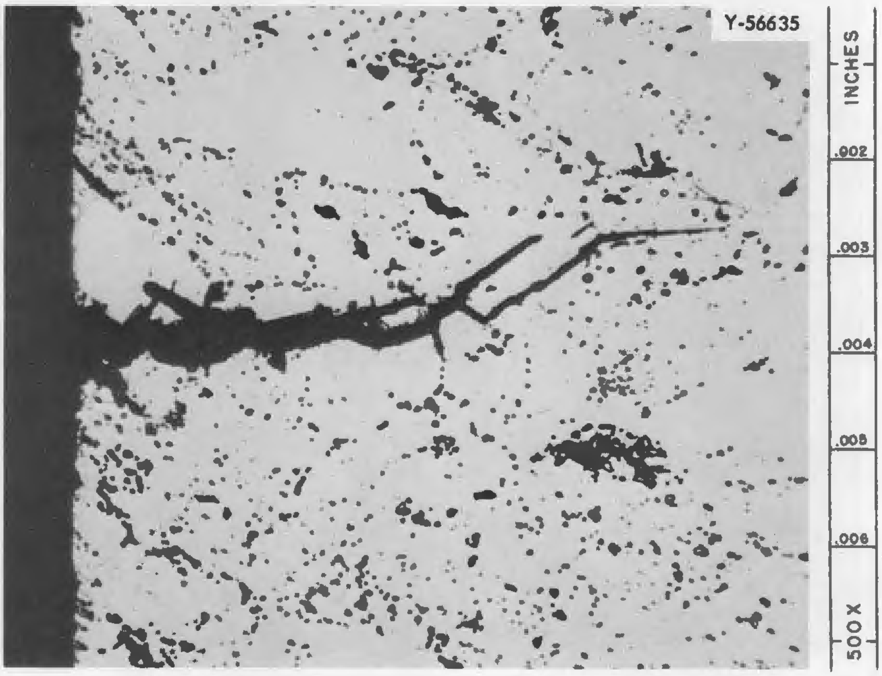

Fig. 8. One of Two Cracks Found in QMV Beryllium Specimen After Extended Exposure in pH 5.0 Water at 100°C. As-Polished. Magnification 500X.

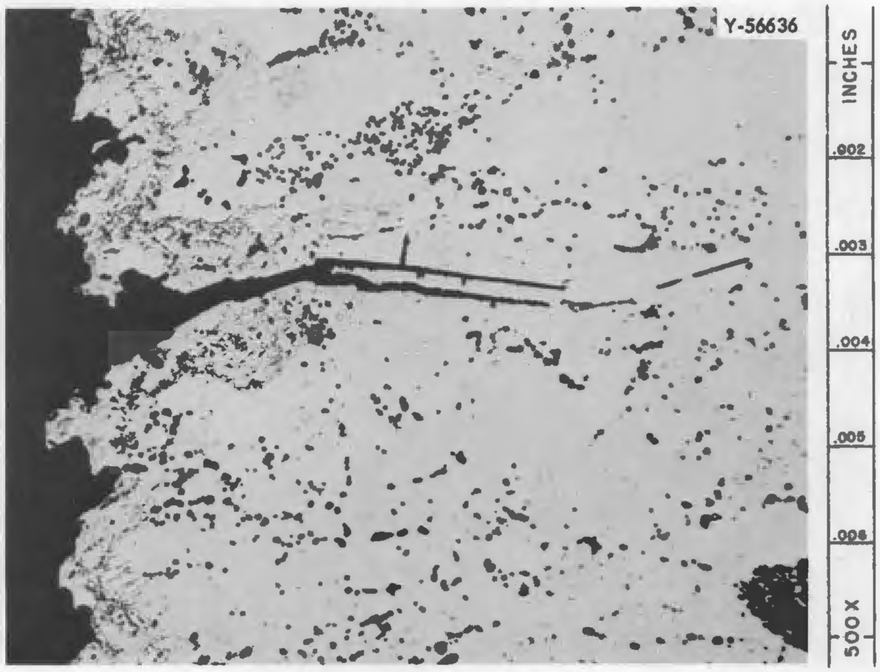

Fig. 9. Second of Two Cracks Found in QMV Beryllium Specimen After Extended Exposure in pH 5.0 Water at 100°C. As-Polished. Magnification 500X.

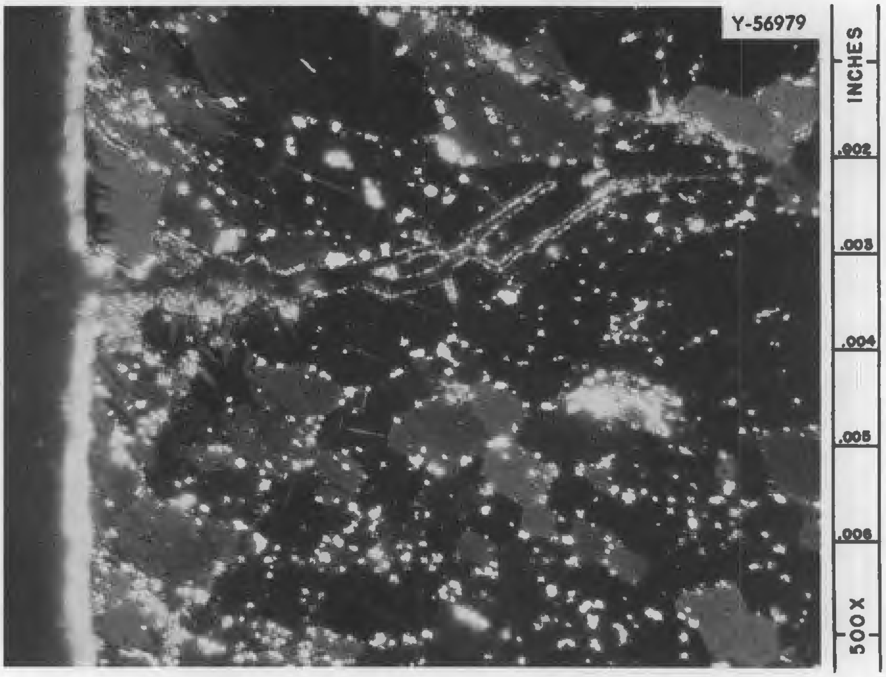

Fig. 10. Polarized Light View Showing Transgranular Nature of Crack in QMV Beryllium Specimen. As-Polished. Magnification 500X.

with a nut. Approximately 5.5 ft-lb of torque were used to tighten the nuts. The torque required to dismantle the assemblies was measured at the completion of each run to give some idea of the extent of galling. The assemblies were mounted in a bypass line of the loop where the flow rate past the specimens was about 0.5 fpm. All were insulated from the holders with Teflon spiders.

Galvanic Specimen No. 1 - Figure 11 presents a schematic diagram of galvanic specimen No. 1. Most of the components in the specimen were exposed for a total of 6385 hours during which period the assembly was dismantled six times for evaluation of corrosion behavior of the components. The highest torque required to loosen the assembly was 15 ft-lb at the end of the first run of 265 hours. Thereafter, the torque varied between 6 and 8 ft-lb.

The 1100 aluminum gasket (No. 4 in Fig. 11) was removed after 1775 hours and replaced with a 304 stainless steel gasket. At the same time, an additional 304 stainless steel gasket was placed between the 6061-T6 aluminum and 304 stainless steel sleeves (No. 2 and 3). The total exposure time for the two added gaskets was 4610 hours. At the time of removal, the 1100 aluminum gasket showed pits between 2 and 3 mils deep on contact surfaces.

During the course of the test, scattered pitting developed on most of the aluminum contact surfaces regardless of whether the contacting material was aluminum or stainless steel. Maximum pit depths were 7 to 8 mils at the end of the test. The average corrosion rate for aluminum was 0.1 mpy. Attack on stainless steel components was negligible.

Galvanic Specimen No. 2 - A second galvanic specimen was exposed for a total of 13,775 hours. A few of the components in the assembly were exposed for a slightly lesser period, 11,775 hours. Figure 12 shows a schematic view of the assembly.

The nickel-plated 6061-T6 aluminum sleeve was plated on all surfaces by an electroless process at the Oak Ridge Gaseous Diffusion Plant. The aluminum coating on the 304 stainless steels was applied by a commercial hot-dip process. One sleeve (No. 7 in Fig. 12) received a coating over

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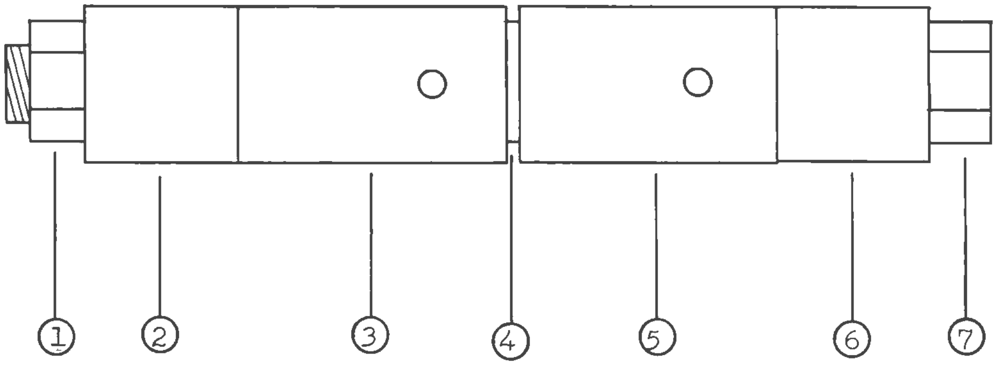

① 6061-T6 aluminum nut ($\frac{1}{2}$ in.)

② 6061-T6 aluminum sleeve

③ 304 stainless steel sleeve

④ 1100 aluminum gasket

⑤ 6060-T6 aluminum sleeve

⑥ 304 stainless steel sleeve

⑦ 6061-T6 aluminum bolt ($\frac{1}{2}$-in.-diam.)

Fig. 11. Schematic Diagram of Galvanic Specimen No. 1.

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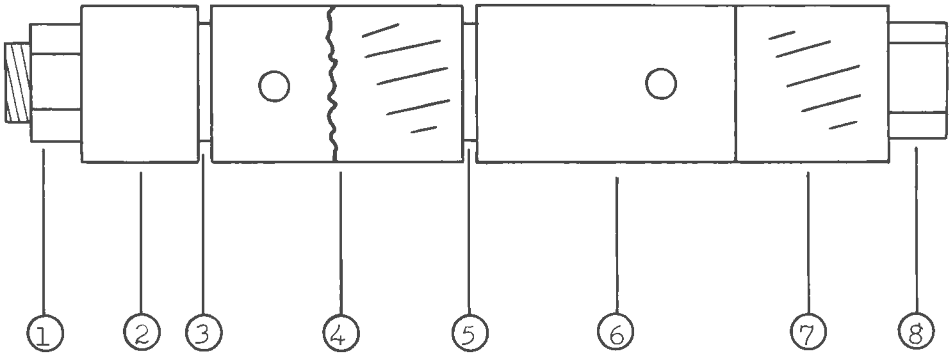

① 6061-T6 aluminum nut ($\frac{1}{2}$ in.)

② Electroless nickel coat on 6061-T6 aluminum sleeve

③ 1100 aluminum gasket

④ Aluminum coat on approximately half of 304 stainless steel sleeve

⑤ 1100 aluminum gasket

⑥ 6061-T6 aluminum sleeve

⑦ Aluminum-coated 304 stainless steel sleeve

⑧ 6061-T6 aluminum bolt ($\frac{1}{2}$-in.-diam)

Fig. 12. Schematic Diagram of Galvanic Specimen No. 2.

the entire surface. The second sleeve (No. 4) was coated over approximately one-half of the total surface. Prior to test, a small area on the coated portion was abraded through the coating and into the base metal to expose any intermetallic substrate layer which might be detrimental to corrosion resistance. No localized attack developed on the abraded area throughout the course of the test.

After the initial 2000-hr exposure, the two 1100 aluminum gaskets (No. 3 and 5 in Fig. 12) were removed and replaced with 304 stainless steel gaskets. In addition, a stainless steel gasket of type 304 was placed between the aluminum-coated stainless steel and the 6061-T6 aluminum sleeves (No. 6 and 7).

The assembly was dismantled for examination nine times during the 13,775-hr exposure. The torque required for loosening the components was measured at the end of each run. The highest torque encountered, 9.5 ft-lb, occurred after a 1520-hr run making a total of 3290 hr. Before and after this particular run, the torque required for dismantling did not exceed 6 ft-lb, an indication that galling was not a problem.

The electroless nickel coating underwent severe attack during the initial 770 hours. The observed weight loss was 38 mg/cm^2, equivalent to a corrosion rate of 19 mpy. At this time, the remaining nickel coating was stripped from the 6061-T6 aluminum sleeve by treatment with hot 15 v/o HNO_3 solution. The sleeve was then pickled in warm nitric-hydrofluoric acid mixture and exposed for the balance of the test in the pickled condition.

The 1100 aluminum gaskets that were removed after 2000 hours had pits up to 5 mils deep, independent of the material contacting them. At the end of the test the stainless steel gaskets showed no measurable corrosion other than some random staining on contact surfaces which were metallic and lustrous in appearance. The partially aluminum-coated stainless steel exhibited a weight loss of 0.7 mg/cm^2 (based on total surface) with scattered pits to 5 mils on the surface contacted by the 304 stainless steel gasket. The totally aluminum-coated stainless steel sleeve showed a weight loss of 1.8 mg/cm^2 with a few pits up to 8 mils deep on contact surfaces. The most severe pitting occurred on the 6061-T6 aluminum bolt; several pits to 20 mils were found. The corrosion rate for both the bolt and the 6061-T6 aluminum nut was 0.1 mpy for the 13,775-hr exposure. The aluminum sleeve from which the electroless nickel plate was removed by acid stripping after the initial 2000 hours corroded at a rate of slightly less than 0.1 mpy. Several pits reaching a maximum depth of 7 mils were found on the surface contacted by the stainless steel gasket. No pitting was found other than on contact surfaces of the aluminum members of the multimetal assembly.

Galvanic Specimen No. 3 - Figure 13 shows a schematic diagram of galvanic specimen No. 3. Included in the assembly were components of type 304 stainless steel and 6061-T6 aluminum that were "electrolized" by a proprietary process. The deposited coating contains approximately 97% Cr with the balance iron and silicon. The electrolized components included a 304 stainless steel nut (No. 1 in Fig. 13), threads only on the 304 stainless steel bolt (No. 8), a 6061-T6 aluminum sleeve (No. 5), and a second 6061-T6 aluminum sleeve with a groove 10 mils deep by $\frac{1}{8}$ in. wide machined through the electrolized layer.

After an initial run of 1000 hours, a torque of 5 ft-lb was sufficient to loosen the assembly, indicative of freedom from galling. The two 1100 aluminum gaskets showed a few scattered pits to 3 mils in depth on contact surfaces. One contact surface of one of the electrolized aluminum sleeves (No. 5) was badly flaked; the second electrolized sleeve (No. 3) showed less intense flaking on both contact surfaces. There was no evidence of flaking in the machined groove around the periphery of the latter sleeve. The electrolized layer on the stainless steel components was in excellent condition. Similarly, the bare 304 stainless steel sleeves showed no signs of corrosion damage.

Prior to the start of the second run, a $\frac{1}{8}$-in.-thick Delrin plastic gasket was placed between components No. 7 and 8 in Fig. 13 and the assembly was torqued to 5.5 ft/lb. The second run lasted 1520 hours. During this period the Delrin gasket underwent nearly complete degradation thereby releasing the torque on the assembly. The contact surfaces on

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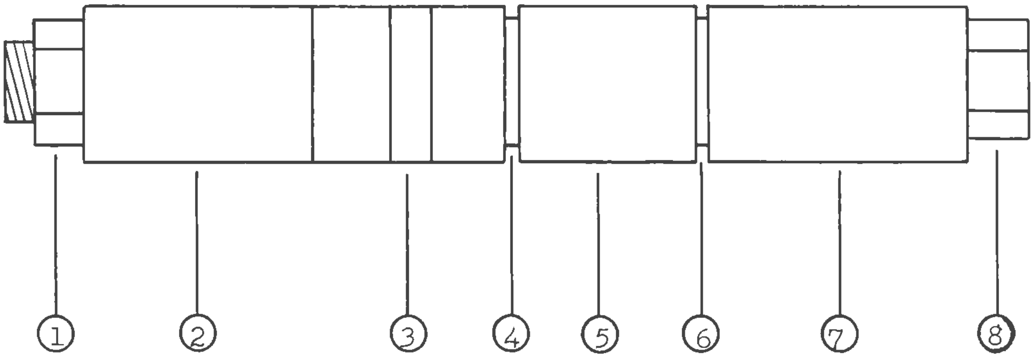

① Electrolized 304 stainless steel nut ($\frac{1}{2}$ in.)

② 304 stainless steel sleeve

③ Electrolized 6061-T6 aluminum sleeve

④ 1100 aluminum gasket

⑤ Electrolized 6061-T6 aluminum sleeve

⑥ 1100 aluminum gasket

⑦ 304 stainless steel sleeve

⑧ 304 stainless steel bolt with electrolized threads ($\frac{1}{2}$-in.-diam)

Fig. 13. Schematic Diagram of Galvanic Specimen No. 3.

both of the electrolized aluminum sleeves were badly flaked and peeled. Areas from which the electrolized layer had peeled showed pits to 11 mils in depth.

At the start of the third run, after a total of 2520 hours had been accumulated, all sleeves and gaskets were removed from the assembly, and the test was continued for an additional 9710 hours with just the stainless steel bolt (with electrolized threads only) and the electrolized stainless steel nut. During this period, the sample was dismantled four times. There was no evidence of galling or localized attack on the electrolized areas. Final defilmed corrosion rates for both the bolt and the nut were less than 0.1 mpy.

Galvanic Specimen No. 4 - Galvanic specimen No. 4 is shown schematically in Fig. 14. At the start of each run the test unit was tightened by snugging the threaded beryllium sleeve on the bolt assembly followed by snugging the electrolized aluminum nut against the beryllium sleeve. No torque measurements were made.

During the 12,755-hr exposure, seven dismantlings and examinations were made. At no time was there any indication of galling. After the first 1000 hours, an aluminum gasket was inserted between components No. 5 and 6 and between No. 6 and 7 in Fig. 14. These two gaskets received a total exposure of 11,755 hours.

All components were in excellent condition after the initial 1000 hours with the exception of the 6061-T6 aluminum sleeve (No. 3) which showed a few scattered pits on contact surfaces to a maximum of 2 mils in depth. No pitting was observed on any of the other components.

Several examinations later, after a total of 5615 hours, the electrolized aluminum sleeve (No. 5) showed severe flaking and blistering on contact surfaces. Areas from which the coating had peeled were badly pitted; a few pits were nearly 11 mils in depth. The electrolized aluminum nut (No. 2) remained in good condition with no signs of flaking. The 1100 aluminum gaskets showed pits, slightly in excess of 2 mils, on contact surfaces. The beryllium sleeves exhibited scattered pits up to 3 mils deep on all surfaces.

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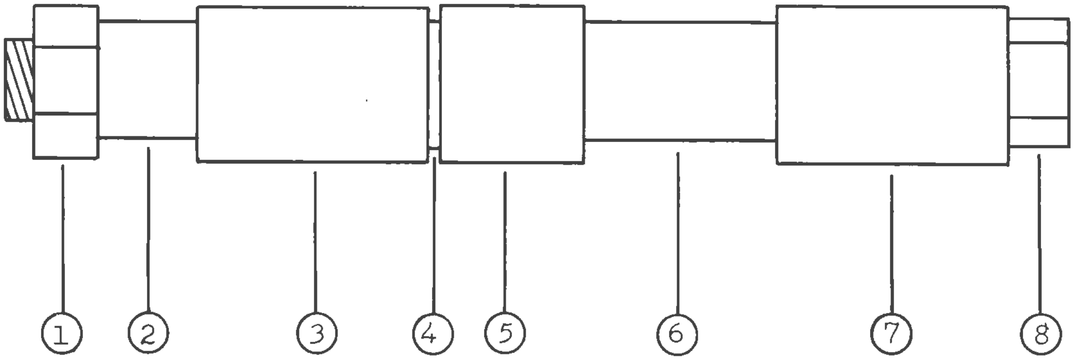

①	Electrolized 6061-T6 nut ($\frac{1}{2}$ in.)
②	Threaded QMV beryllium sleeve with two flat sides
③	6061-T6 aluminum sleeve with 8 rms finish
④	1100 aluminum gasket
⑤	Electrolized 6061-T6 aluminum sleeve
⑥	QMV beryllium sleeve
⑦	6061-T6 aluminum sleeve with 32 rms finish
⑧	6061-T6 aluminum bolt with electrolized threads ($\frac{1}{2}$-in.-diam)

Fig. 14. Schematic Diagram of Galvanic Specimen No. 4.

Final defilmed corrosion rates after 12,755 hours for the 6061-T6 aluminum sleeves with 8 and 32 rms finishes and for the aluminum gaskets were less than 0.1 mpy with the usual pitting on contact surfaces. The electrolized 6061-T6 aluminum sleeve and nut showed different behavior. As mentioned, the former underwent substantial flaking and peeling on contact surfaces only; pits to 16 mils in depth were measured on peeled areas. On the other hand, the electrolized nut remained in excellent condition, and there was no evidence of localized attack on the threads which contacted the electrolized threads of the 6061-T6 aluminum bolt. The two beryllium specimens corroded at the same rate, 0.3 mpy with scattered pits on all surfaces. No pits were found on the 6061-T6 aluminum bolt which corroded at a rate less than 0.1 mpy. The condition of the electrolized threads on the bolt was excellent.

Galvanic Specimen No. 5 - An assembly consisting of types 304 stainless steel and hardened 416 stainless steel (Fig. 15) was exposed for 11,775 hours in the pH 5.0 water at 100°C. The 416 stainless steel components were hardened to 43 Rockwell C by heating at 954°C (1750°F), oil quenching to room temperature, and tempering at 538°C (1000°F) for several hours.

As in previous specimens, the assembly was tightened to a torque of 5.5 ft-lb, at the start of each run. The assembly was dismantled for examination of components six times during the 11,775-hr exposure. No galling occurred as evidenced by the fact that the torque required for dismantling at the end of each run varied between 3 and 5 ft-lb.

With one exception, nothing of significance occurred on the components in the assembly during the entire exposure other than the formation of brown-colored stains on contact surfaces, particularly on the hardened 416 stainless steel members, and the development of several small blisters on the hardened 416 bolt during the final stages of the exposure. The location of the blisters was confined to the threaded portion of the bolt. Defilmed corrosion rates for all components were 0.1 mpy or less. There was no pitting.

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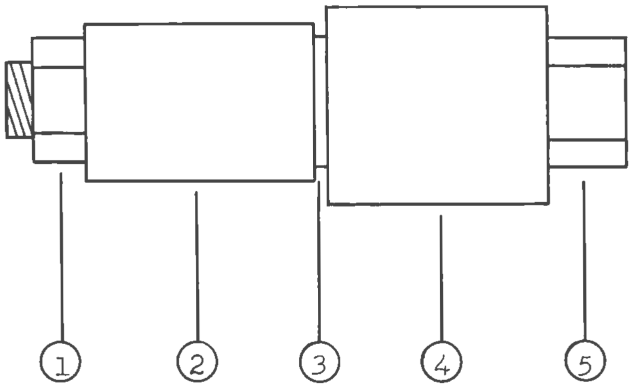


① 304 stainless steel nut ($\frac{1}{2}$ in.)

② Hardened 416 stainless steel sleeve

③ 304 stainless steel gasket

④ 304 stainless steel sleeve

⑤ Hardened 416 stainless steel bolt ($\frac{1}{2}$-in.-diam)

Fig. 15. Schematic Diagram of Galvanic Specimen No. 5.

Ball-Latch Test - A prototype HFIR ball-latch mechanism, contained in a 3-in-diam stainless steel autoclave and supplied with water from a bypass line from the main stream of the loop, was exposed for 11,700 hr at 100°C with frequent examinations during this period. The shock absorber piston shoulder was spring loaded against the ball latch with a 400 lb spring as it would be in the reactor.[7] Materials included in the mechanism were types 304, 416, and 17-4 PH stainless steel, Inconel, Stellite No. 3, and Stellite No. 1 overlayed on type 304 stainless steel.

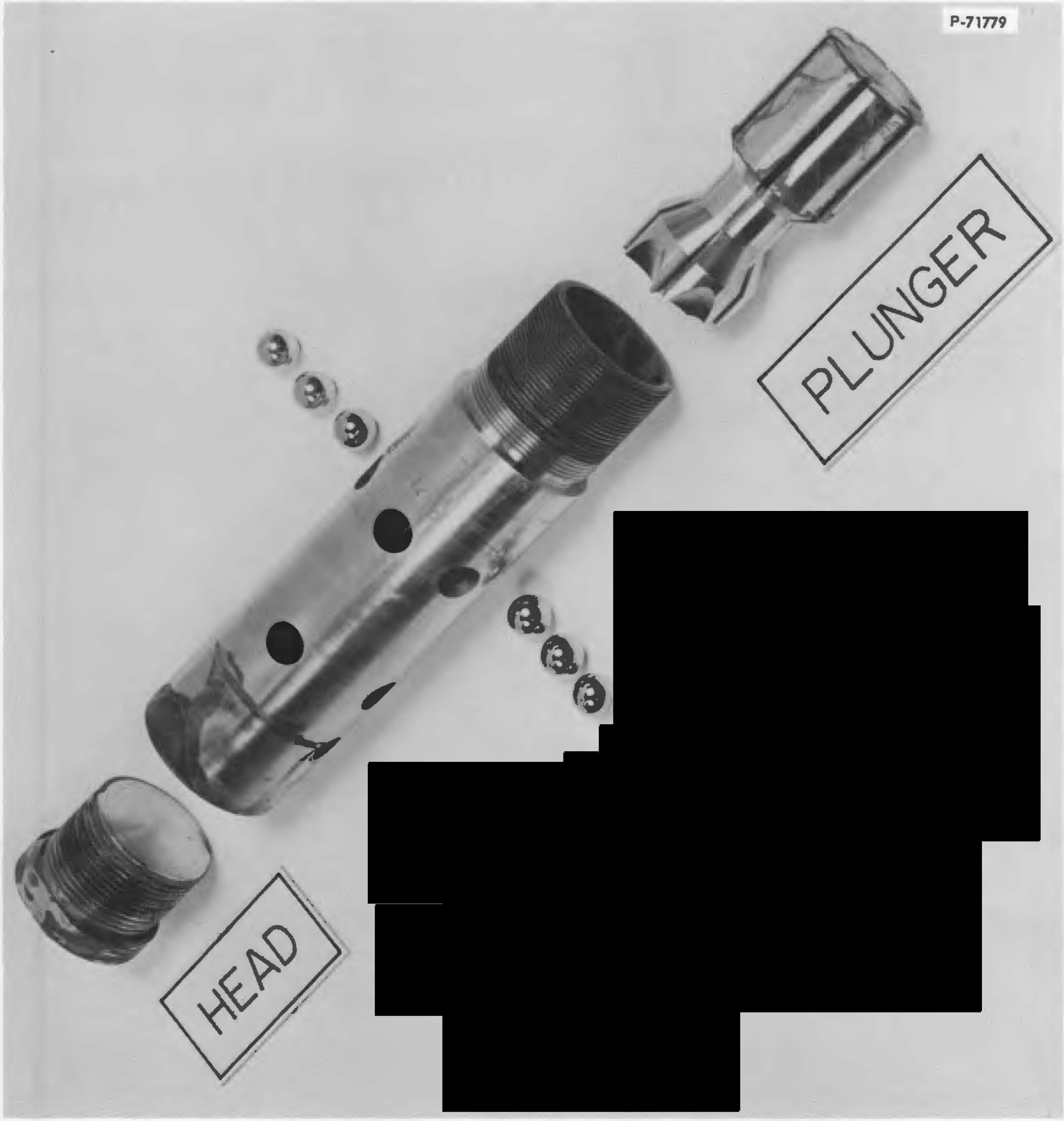
The final inspection disclosed the most significant effect to be on the 416 stainless steel ball cage. A hard, tenacious red-brown oxide appeared on the threads at both ends of the cage and on the inside diameter at an area where there was a close tolerance between the ball cage and the Stellite 1 overlayed 304 stainless steel plunger. The mating threads on the outside diameter of the head (both types 304 stainless steel) as well as the ball cage and the plunger also were stained. No staining was observed on the Stellite No. 3 balls nor on surfaces contacted by the balls. The post-test condition of the ball-latch mechanism is shown in Fig. 16.

Although the presence of substantial oxide on portions of the ball-latch mechanism (predominantly on the hardened 416 stainless parts) was undesirable, it was concluded that in view of the long and successful experience with a similar mechanism in the Oak Ridge Research Reactor, the oxide would not interfere with the operation of the assembly.

5. SUMMARY OF TEST RESULTS

The most significant results obtained from this testing program can be summarized as follows:

1. Type 6061 aluminum was shown to have excellent corrosion resistance to water adjusted to a pH of 5.0 with nitric acid at 100°C. In long-term tests, corrosion rates of 0.2 mpy or less were observed at flow rates as high as 81 fps. Similar corrosion behavior was exhibited by the X8001 alloy.



[redacted]

Fig. 16. Post-Test Condition of Ball-Latch Mechanism After 11,770 Hours in pH 5.0 Water at 100°C.

2. Aluminum was subject to pitting attack on surfaces contacted by itself, beryllium, nickel and types 304, 420 and 17-4PH stainless steel. Maximum pit depths, up to 54 mils in 5,560 hr, occurred when contacted by nickel. When contacted with type 304 stainless steel, random pits as deep as 45 mils occurred during 12,475-hr tests.

3. Beryllium, when exposed at flow rates of 2 to 3 fps, corroded at a constant rate of 0.6 mpy. At flow rates between 13 and 81 fps, the corrosion rate was independent of flow rate and constant at 2 mpy. Regardless of flow rate or contact with other materials, beryllium was subject to random pitting attack. Pits up to a maximum depth of 13 mils were present on specimens coupled to stainless steel and exposed for 11,980 hr. An apparent incident of stress corrosion cracking of beryllium was observed on one specimen in contact with type 304 stainless steel.

4. Both commercial-purity nickel and an electroless nickel plate showed poor resistance to the pH 5.0 environment. In the former case, the material developed numerous shallow pits, and in the latter case the material corroded at a rate of 19 mpy.

5. A proprietary coating ("electrolyzed" consisting of 97% Cr with balance iron and silicon) did not adhere well to 6061 aluminum surfaces. Similar coatings on type 304 stainless steel showed excellent behavior.

6. Hardened type 416 stainless steel underwent appreciable staining but corroded at a rate of less than 0.1 mpy. However, several small blisters developed on the threaded portion of a bolt. Hardened type 420 stainless steel was subject to stress-corrosion cracking.

7. Delrin, a polyformaldehyde plastic of potential interest as a gasket material, suffered near-complete degradation at the test temperature of 100°C.

8. A complete ball-latch mechanism was exposed for 11,770 hr in pH 5.0 water at 100°C. Materials included as part of the mechanism were types 304, 416, and 17-4PH stainless steel, Inconel, Stellite

No. 3, and Stellite No. 1 overlayed on type 304 stainless steel. Corrosion damage was insignificant except for staining of the hardened 416 stainless steel components.

6. DISCUSSION

The results of this investigation and those of a previous one[1] indicate that with proper control of the operating conditions and water chemistry, corrosion of the HFIR components should not be a significant problem. The corrosion behavior of the major materials of construction was excellent in water adjusted to a pH of 5.0 with nitric acid at 100°C. There are, however, a few points from the corrosion investigations as well as from other considerations that should be elaborated on, and these are discussed in the following paragraphs.

The surface of the HFIR pressure vessel exposed to the coolant is austenitic stainless steel. Many of the permanent or semipermanent components mounted in the pressure vessel are aluminum. It has been shown that in areas of contact between these materials appreciable pitting of the aluminum occurs. Although this situation is undesirable, the random nature of the pitting and the fact that the mating aluminum surfaces were designed with greater thicknesses than necessary indicate that corrosion of the aluminum in these regions should not be of major consequence. Furthermore, the present investigation showed that the attack rate significantly decreased as corrosion product formed in the crevice region. Since the contacting aluminum-stainless steel surfaces will seldom be disturbed in the reactor, and since the temperature in most of these regions will be much less than the 100°C used in the tests, it is highly probable that the attack on the aluminum in contact with stainless steel will be appreciably less than observed in the testing program.

The attack of beryllium in crevice regions is similar to that of aluminum, and it is concluded that this also is unlikely to present problems. Even in the absence of crevices, beryllium has a tendency to pit, but the rate of growth of the pits is such as to be of no concern to the HFIR operation. In tests that lasted between one and two years, maximum pit depths were only about 13 mils.

To the authors' knowledge, the cracks found in the one beryllium specimen coupled to stainless steel represent the only reported case of stress-corrosion cracking of beryllium in water. In the same environment three other specimens coupled to stainless steel were exposed under the same conditions without evidence of cracking. Also, many uncoupled specimens showed no signs of cracking after exposures in excess of two years. All had similar regions of stress concentration where identification numbers had been stamped on them. These observations and the successful use of unclad beryllium in the ORR and the MTR for many years lead to the conclusion that the development of significant stress-corrosion cracks in the beryllium reflector of the HFIR is highly improbable.

The general corrosion rate of the austenitic stainless steel in the primary system will be essentially nil, but the possibility of highly localized stress-corrosion cracking cannot be completely ignored. Two ions are notorious for producing cracks in austenitic stainless steel, viz chloride and hydroxide. Generally high concentrations of caustic and high temperatures are necessary to produce caustic cracking and in the HFIR these conditions will not exist. On the other hand only very low concentrations of chloride ions and relatively low temperatures can initiate stress-corrosion cracking in austenitic stainless steel. Oxygen or some other oxidizing agent must also be present. Generally the higher the chloride ion concentration and the higher the temperature, the greater the probability of cracking.

That stress-corrosion cracking can occur in reactor systems containing pure water at relatively low temperatures was demonstrated at the Savannah River Plant.[8] Two highly stressed outlet nozzles failed by stress-corrosion cracking after seven years of service even though the coolant was highly purified heavy water, containing less than 10 ppb chloride ions, at temperatures somewhat below 100°C. In these cases, the type 304 stainless steel had been highly sensitized and severely pickled during fabrication. General intergranular attack averaged 7.5 mils. Furthermore, over the years the surfaces had become covered with a thin layer of aluminum oxide (corrosion product from fuel element cladding) which contained several hundred parts per million of chloride ions. Apparently the hydrated aluminum oxide was an effective scavenger for the trace amounts of chloride in the coolant. It is possible that the aluminum oxide desorbed enough chloride directly on the surface of the steel as in the grain boundaries to produce actual concentrations of several parts per million.

Tests by Rideout[8] have shown that similar cracks can be produced in the laboratory at 90° to 100°C with chloride concentrations as low as 2 ppm. To produce cracks in this mild environment, it was necessary to sensitize the type 304 stainless steel and pickle it sufficiently to produce general intergranular corrosion to a depth of 8 to 15 mils prior to exposing the specimens to the chloride-containing solution.

It was decided that a meaningful stress-corrosion cracking investigation under HFIR conditions could not be conducted in the time allotted, but all possible precautions were taken to minimize the probability of cracking of the austenitic stainless steel in the system. During assembly great care was exercised in avoiding contact of the stainless steel with chloride-containing materials.

Pickling of sensitized areas such as heat-affected zones near welds was prohibited except in the case of the weld-clad pressure vessel. These surfaces were lightly pickled by the fabricator under carefully controlled conditions in the presence of an ORNL inspector. Those stainless steel parts which were to be located in the vicinity of the reactor core (exposed to the highest temperature) were made of a stabilized or low-carbon grade of stainless steel, either types 347 or 304L. All welds on these components were full-penetration welds rather than fillet welds where crevices could possibly lead to accumulation or concentration of chloride ions. In addition all parts in which high stresses were developed during fabrication were given appropriate stress-relief heat treatments to reduce residual stresses to low values. The oxide scales were removed from the components by vapor blasting rather than pickling, as a further precaution against the initiation of general intergranular attack.

The above design and fabrication considerations should significantly reduce the probability of stress-corrosion cracking in the HFIR. Another important factor that should make cracking less likely in the HFIR than in the Savannah River Reactors is the temperature of the coolant. The maximum temperature of the water in contact with the stainless steel containment system will be lower in the HFIR than in the Savannah River Reactors (165°F vs ~200°F). Since the activation energy for crack initiation and for crack propagation is high,[9] cracking, if it is to occur, should require a much longer time to start and if started should propagate at a much lower rate in the HFIR than in the Savannah River Reactors.

Stress-corrosion cracking of the heat exchanger tubes initiating from the cooling water side is possible but at present appears to be remote. Five cycles of concentration of the cooling-tower water could result in maximum chloride concentrations as high as 25 ppm. However, the low temperature of the tubes (165°F maximum) and the fact that the tower water will contain an inhibitor should reduce the probability of cracking to an extremely low value.

In the HFIR, most of the reactor components are mounted in the pressure vessel using studs and nuts. Some of the materials considered for this application were the 400 series stainless steels, 17-4 PH stainless steel and the austenitic stainless steels. The present investigation as well as observations made by others[10] showed that the corrosion resistance of the 400 series stainless steels in water is questionable; therefore, this class of materials was eliminated from consideration. Type 17-4 PH stainless steel in all heat-treated conditions has excellent general corrosion resistance to water at all temperatures, and in the age-hardened condition (H-900) it has excellent mechanical properties. Unfortunately, in this condition the alloy is highly susceptible to stress-corrosion cracking in all aqueous environments, including pure water. Overaging the alloy (H1100 condition) eliminates its susceptibility to cracking but also reduces its strength and hardness; but in this condition the alloy still has useful properties, is used in many operating reactors, and has found some use in the HFIR.

For most of the bolting applications in the HFIR, type 304 stainless steel has adequate strength, but galling in threaded joints is a well-recognized problem. An "electrolyzed" coating (97% Cr) on the threads can be used to eliminate the galling. Long-term tests showed that the corrosion of the deposited coating was negligible and that it had no tendency to peel from the stainless steel base. In view of the favorable test results, most of the bolts used in the HFIR pressure vessel are type 304 with an "electrolyzed" coating on the threaded surfaces.

It is impossible to guarantee the complete absence of significant corrosion problems during the life of any complex reactor. However, in the HFIR the results from the corrosion-test program and the experience of others were used as fully as possible in the choice of materials and in the design of the components. In view of the above considerations and with proper control of the coolant chemistry, it is the authors' opinion that the HFIR system, exclusive of the core, will be free from major corrosion problems for many years.

7. ACKNOWLEDGMENTS

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