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THE CORROSION OF ALUMINUM ALLOYS IN THE OAK RIDGE RESEARCH REACTOR

P. D. Neumann

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REACTOR CHEMISTRY DIVISION

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P. D. Neumann

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THE CORROSION OF ALUMINUM ALLOYS IN THE OAK RIDGE RESEARCH REACTOR

P. D. Neumann

ABSTRACT

A corrosion testing program designed to estimate the potential service life of aluminum alloys used in the construction of the Oak Ridge Research Reactor (ORR) cooling systems has been in progress for over two years. The five alloys (1100, 3003, 5052, 5154, and 6061) used to the greatest extent in the reactor have exhibited continously decreasing corrosion rates since the first 500-hr inspection. Samples exposed in the core-cooling loop have shown a decrease in corrosion rate from a 2.6 mpy maximum for one group during the first 500 hr to an over-all average of less than 0.1 mpy for another group after a full year in test, with the maximum metal loss less than 0.1 mils.

In the pool surrounding the reactor, and in a secondary cooling system, initial short-term rates of 1 to 2 mpy decreased to less than 0.05 mpy at the end of a full year in test and the maximum metal loss has been less than 0.2 mils.

Pitting does not appear to be a serious problem, even though a few isolated pits have been observed. These pits occur in crevices under mounting-washers on the corrosion specimens and occasionally on exposed areas where they are probably the result of inclusions or inhomogeneities in the alloy.

On the secondary side of the aluminum heat exchanger, standard water-treatment practices have proven adequate for scale and corrosion prevention. Examinations of the heat-exchanger tube bundle after 6 months and after 13 months of service showed only minor film formation and slight accumulations of fine, insoluble materials on the tubes. Fairly heavy accumulations were found in stagnant areas of the heat-exchanger shell.

These results indicate that with suitable water treatment the aluminum alloys used in the ORR may be expected to give satisfactory performance for many years. Based on the generalized corrosion rates alone, one might expect 40 to 50 years of service life. However, since occasional localized corrosion has been observed (rarely), minor repairs will almost certainly be required before that time.

INTRODUCTION

The Oak Ridge Research Reactor (ORR) is an MTR-type reactor fueled with enriched uranium in the form of aluminum-clad fuel elements. The fuel elements and the associated aluminum core assembly are enclosed in an aluminum alloy tank through which high-purity water is pumped. This water is then circulated through aluminum alloy finned-tube radiators which exhaust the reactor heat to the atmosphere. The entire core tank is submerged in a pool of high-purity water which serves as shielding. At 20 Mw total power, approximately 0.7 Mw of heat is generated in the pool. Heat is removed by pumping the pool water through an aluminum alloy heat exchanger which is served by a conventional redwood cooling tower.

Practically all materials in contact with the reactor cooling and shielding water are aluminum alloys. Four aluminum alloys were used in fabricating major components of the reactor. The core tank is constructed of 5052-F and 5154-F alloys, and the top cover is constructed of 6061-T6. Large-diameter pipelines (up to 36 in. OD) are of 5154-F, while small-diameter piping is of 6061-T6. The fuel elements are clad with 1100-H14 alloy. The heat exchanger that cools the pool water is a shell and U-tube type, with an all-welded 5154-F shell. The tubes are 3003 clad on the outside with 7072 alloy for increased corrosion resistance. The tube sheet is 6061-T6 for maximum strength. A few small pipes were fabricated from 6063-T6. All of these alloys except the 6063-T6 were included in the testing program carried out in the reactor. Table 1 presents the composition of these alloys, and Table 2 gives the temper designations.

Table 1. Composition of Aluminum Alloys[1]

Former designation:	2S	3S	52S	54S	61S
Aluminum Association designation:	1100	3003	5052	5154	6061
Al (min.)	99.00	Remainder	Remainder	Remainder	Remainder
Si	1.0*	0.6	0.45*	0.45*	0.40-0.80
Fe		0.7			0.7
Cu	0.20	0.2	0.10	0.10	0.15-0.40
Mn	0.05	1.0-1.5	0.10	0.10	0.15
Mg			2.2-2.8	3.1-3.9	0.8-1.2
Cr			0.15-0.35	0.15-0.35	0.15-0.35
Zn	0.10	0.1	0.10	0.20	0.25
Ti				0.20	0.15
Other	0.15	0.15	0.15	0.15	0.15

*Iron plus silicon.

The reactor began its first cycle on July 20, 1958, and since that time two memos concerning certain aspects of the corrosion program have been written.[2,3] The present report, however, summarizes all the data that have been obtained from specimens exposed in the reactor system from July 1958 to May 1960. The testing program in the reactor system is continuing.

Table 2. Temper Designations of Corrosion Coupons[1]

Designation		Treatment
Former	Aluminum Association	
2S 1/2H 3S 1/2H	1100-H14 3003-H14	Strain-hardened halfway between full anneal and full hardness.
52S 54S	5052-F 5154-F	As fabricated.
52S-0	5052-0	Annealed at 650°F, fully softened condition.
61S-T	6061-T6	Solution annealed at 970°F and quenched. Aged 6 to 10 hr at 350°F.

WATER TREATMENT PROGRAM

The water used to cool the core of the reactor is maintained at a high level of purity by continually diverting a part of the circulating stream through a demineralizer. The resistivity of the water varies between 0.75 and 1.5 x 10^6 ohm-cm; the pH of the water is usually between 5.5 and 6.5.

Under normal conditions of operation, the coolant water enters the reactor at 124°F and exits at 132°F. To maintain this temperature rise at 20 Mw reactor power, 16,000 gpm of water are pumped through the core. However, during the summer months the inlet temperature is higher than that listed above, and because it has been the practice to limit the coolant water to a maximum of 135°F, it has been necessary in the past to reduce the reactor power at these times. Supplemental[4] heat-removal equipment in the form of water-cooled heat exchangers has been installed in the reactor system, so that in the future total reactor power will not be limited by the atmospheric temperature.

The purity of the water in the pool surrounding the reactor tank is also maintained at a high level by use of deionizers. The average resistance of the water is 0.7 x 10^6 ohm-cm, and the pH ranges between 5.5 and 6.5. The average temperature of the pool water during reactor operation is 100°F. The pool water is circulated by convection currents due to heat from the reactor core and by a pump which circulates 900 gpm of water to a heat exchanger which transfers the heat to a cooling tower circuit. The temperature of the water entering the heat exchanger is 100 to 101°F; the exit temperature is 90°F.

Corrosion inhibition in the cooling-tower water is achieved with a proprietary mixture of sodium chromate and sodium polyphosphate glass. This material is fed to the system by passing the make-up water over balls of the chemical held in a bypass feeder. Because of the low solubility of the glass, the make-up water dissolves only a few parts per million of the chemical. The optimum concentration is maintained at 6.5 to 7.5 by the addition of sulfuric acid. Until early 1960 the acid was added periodically by an operator. At that time equipment to maintain the pH automatically was installed.

Two patented organic toxicants are used to control micro-organisms in the tower water. Once a week enough of an amine-type toxicant is added to the water to produce a concentration of 100 ppm. Once every three weeks a water-soluble unsaturated chlorophenate is fed to the system to produce a concentration of 300 ppm. The combined treatment has held biological growth in the cooling water to an insignificant amount.

EXPERIMENTAL

Test Specimens and Location

The coupons and holders used for corrosion testing in the reactor fuel lattice are shown in Fig. 1. They were designed to fit into the isotope irradiation assembly, shown in Fig. 2, in place of some of the isotope cans. The whole assembly occupied a spare fuel lattice position during operation of the reactor.

The two-piece coupon holders were 13/32 in. in diameter and 2 3/8 in. long. They were machined from 1/4-in. 6061-T6 aluminum plate and assembled with 5052-0 aluminum rivets. The guide rods shown on the bottom of the specimen holder were 5052-0 aluminum wire bent to form a cone which kept the specimen holder off the bottom of the isotope bucket and thereby facilitated the flow of cooling water through the holders. The cooling water flowed over the specimens at an estimated velocity of 8 to 12 fps.

Corrosion testing outside the core tank was conducted with specimens cut from 0.0625-in. sheet and machined to 1.00 in. x 2.50 in. These coupons were mounted on a stainless steel bar which contained several stainless steel studs. Teflon insulators were used to separate the aluminum coupons from the stainless steel. Teflon could be used because these holders were used only in regions of low-intensity radiation. Provisions were made so that several mounting bars could be connected in series to provide additional test coupons at each location. Stainless steel bars with 30 samples were installed in the body of a strainer in the 24-in. core cooling-water exit pipe downstream from the N^{14} decay tanks.

Two other such assemblies containing 40 specimens were also mounted in the center section of the pool where they were exposed to aerated demineralized water at approximately 100°F.

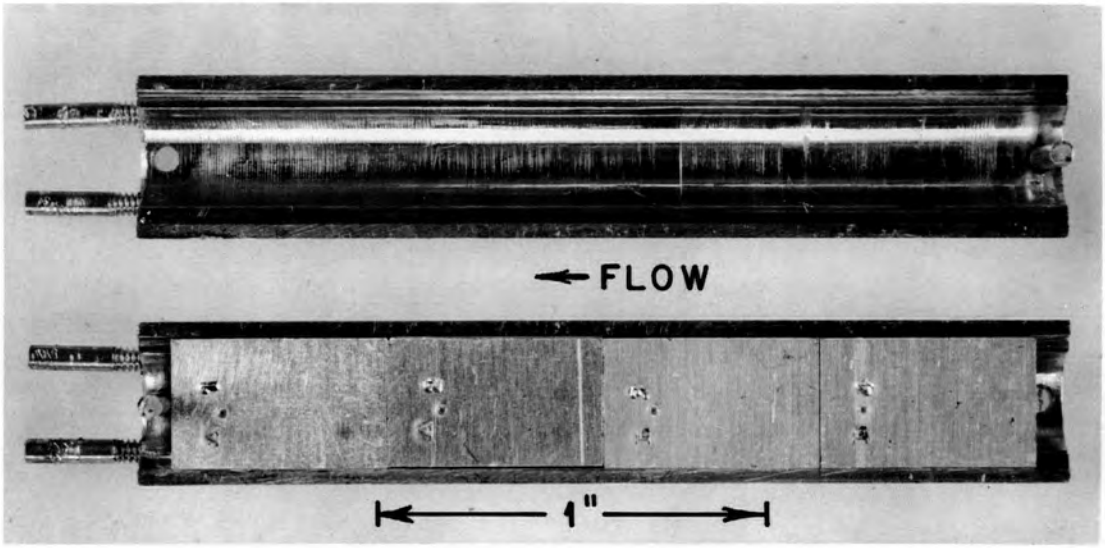
Two sets of these mounting bars with 40 specimens each were exposed to the cooling-tower water. One set was located in the exchanger-outlet pipe and exposed to cooling-tower water at approximately 90°F. The second set was located in the cooling-tower sump and was in contact with the same water after being cooled to approximately 80°F.

Preparation and Evaluation

After being machined, the specimens were marked with stamping dies and then pickled in a solution containing 58 ml of concentrated sulfuric acid and 30 g of chromium trioxide per liter of solution. The specimens were continuously agitated for 20 min in this solution at 70°F. They were then washed thoroughly in demineralized water, dried at 248°F, cooled, and weighed. After assembly, all parts were degreased in alcohol and acetone.

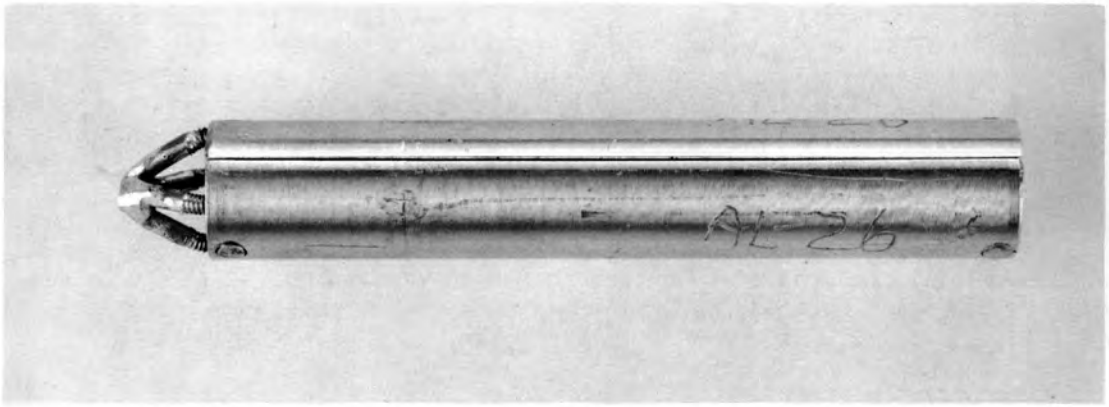
After testing, out-of-pile coupons were scrubbed with a soft brush and detergent; those coupons exposed in the core, however, were only rinsed in deionized water. All were then weighed and examined under a microscope. They were then given a defilming treatment of 45 min at 176°F in a solution containing 35 ml of concentrated phosphoric acid and 20 g of chromium trioxide per liter, washed in

Plate 3096-1



a. ORR Corrosion Test Coupons in Coupon Holder.

Plate 4355



b. Completely Assembled Corrosion Test Assembly.

Fig. 1. Corrosion Specimens and Holder
Exposed in ORR Core Assembly.

Photo 41741

Fig. 2. ORR Isotope Irradiation Assembly.
(Corrosion specimen holders were placed in the cylindrical buckets in place of some of the isotope cans.)

demineralized water, and dried at 248°F. Finally the specimens were reweighed and examined again under the microscope. Blank corrections were found to be negligible. Specimens showing localized corrosion were examined metallographically.

The coupons and holders exposed in the reactor core were sufficiently radioactive that they could not be examined directly. The first two groups of specimens removed were stored dry in lead carriers for about three months, at which time the activity had decayed to a level where the specimens could be examined without shielding. The third group of specimens was examined immediately after removal in one of the ORNL hot-cell facilities.

The samples from the strainer in the core-cooling loop were only slightly radioactive when removed from test. The maximum activity of any single specimen was 5 mr/hr on contact after a distilled-water rinse. No activity has been detected on the specimens exposed to the cooling-tower water.

Exposure Schedule

The ORR operates on a 4-week cycle in which three weeks of operations are followed by approximately one week of down-time. The first full-length operating cycle started July 20, 1958. Sixteen complete corrosion coupon holders, each containing four specimens, were exposed in the reactor core during this cycle. Eight holders were removed at the end of the first cycle and replaced with new ones. (The other eight holders have been in test since July 20, 1958.) During the 500 hr of reactor operation in the first cycle, the reactor accumulated

7582 Mw-hr of operation. The eight holders inserted in cycle 2 were removed and replaced after cycle 6, corresponding to a total time of 4032 hr in the reactor, of which 2318 hr were at power. Total power accumulation for this period was 45,563 Mw-hr. A third group of specimens was removed on January 24, 1960, after 8,712 hr in test. The reactor was at a significant power level for 6,630 hr and accumulated 120,218 Mw-hr during this period.

Corrosion coupons which were tested in the core coolant at the strainer were removed during scheduled reactor shutdowns as were those at the heat-exchanger outlet. Those from the pool and the cooling-tower sump could be inserted and removed at any convenient time because of their accessibility.

RESULTS

Core Specimens

Corrosion results for the five alloys tested in the reactor core are shown in Table 3. The weight losses and corrosion rates are averages for 6 to 10 specimens of the same alloy and include those which showed no weight losses.[3] Although the data show some scatter, the averages indicate that the corrosion rates increased with increasing alloying constituents (see Table 1). It is also evident that the rates decreased with time. The maximum weight loss shown by any specimen was 1.2 mg/cm^2.

Table 3. Corrosion of Five Aluminum Alloys in the ORR Core (values are averages for 6 to 10 specimens)

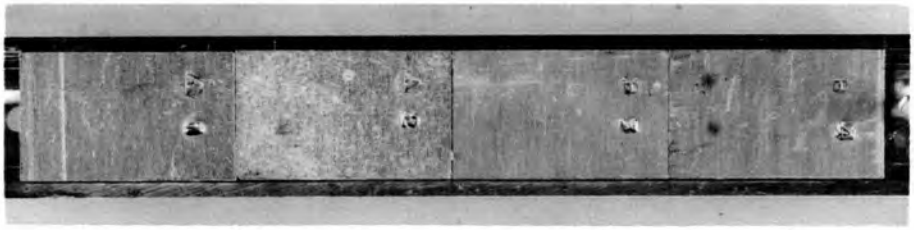
Exposure Time:	500 hr		4032 hr		8712 hr	
Accumulated Power:	7582 Mw-hr		45,563 Mw-hr		120,218 Mw-hr	
Alloy	mg/cm^2	mpy	mg/cm^2	mpy	mg/cm^2	mpy
1100	0.2	0.4	0.2	<0.1	WG*	-
3003	0.5	1.4	0.1	<0.1	WG*	-
5052	0.7	1.7	0.2	<0.1	WG*	-
5154	0.7	1.8	0.2	0.1	0.1	<0.1
6061	1.0	2.5	0.4	0.1	<0.1	<0.1

*Specimens could not be completely defilmed; therefore, they showed a net weight gain.

Many of the specimens which were in test for 8712 hr were found to exhibit weight gains after defilming in the solution described previously. The reason why the specimens could not be defilmed is not known. It has been shown by others[5] that films formed on aluminum at temperatures of 212°F and below are readily soluble in the defilming solution. Those specimens which could not be completely defilmed showed smooth surfaces with thin, semitransparent films and gave every indication that the corrosion rates were low.

Figures 3 through 5 are photographs of some test specimens taken before and after testing. The 500-hr test specimens were chosen for publication because they showed the most severe corrosion damage of any of the specimens exposed in this location. Some light staining of the surface and slight crevice corrosion where the coupons contacted the holders can be seen in the photographs. This crevice

Plate 3096-2



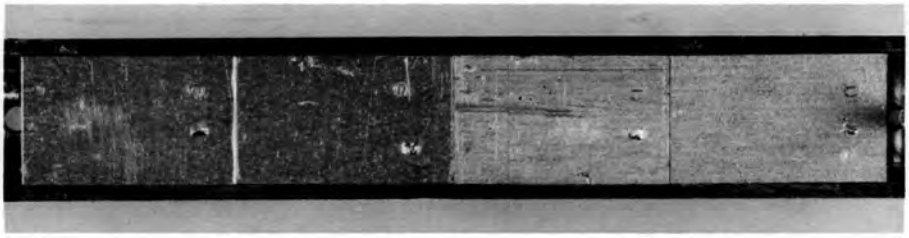
a. 1100 3003

Plate 4147-2

b. 1100 3003

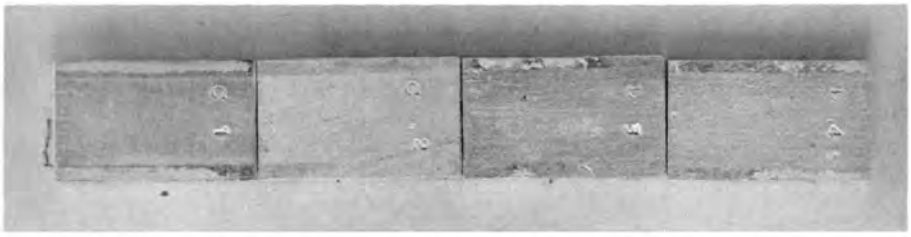
Fig. 3. The Appearance of 1100 and 3003 Aluminum Specimens Before (a) and After (b) a 500-hr Exposure in the Reactor.

Plate 3097-2



a. 5052 5154

Plate 4148-2



b. 5052 5154

Fig. 4. The Appearance of 5052 and 5154 Aluminum Specimens Before (a) and After (b) a 500-hr Exposure in the Reactor.

Plate 4000-2

a. 5052 6061

Plate 4149-2

b. 5052 6061

Fig. 5. The Appearance of 5052 and 6061 Aluminum Specimens Before (a) and After (b) a 500-hr Exposure in the Reactor.

corrosion was visible only as a darker stain on the coupon surface with roughening of the same magnitude as seen on the exposed surface. What appeared to be several deep pits were seen upon microscopic examination. Subsequent metallographic examination of these areas revealed that many of the apparent pits were, in reality, lightly attacked surfaces of mechanical defects as shown by the deformation in the surrounding microstructures. Such a defect is shown in Fig. 6. No evidence of intergranular corrosion was found in any of the coupons examined.

Figures 7 through 9 are photomicrographs of cross sections through typical surfaces of some specimens which were in test for 500 hr. No evidences of serious localized corrosion were found; neither were the average corrosion rates indicative of excessive generalized attack. The specimens exposed for 4032 and 8712 hr showed less corrosion damage in spite of the longer exposure times.

Strainer Specimens

The first coupons were started in test May 20, 1959. Two coupons of each alloy were removed from the strainer position after 720 hr of exposure. Two more coupons of each alloy were removed after 4700 and 8600 hours. Corrosion data for these coupons are shown in Table 4. The behavior of these coupons was very similar to that found in the core. The corrosion rates were low and decreased with

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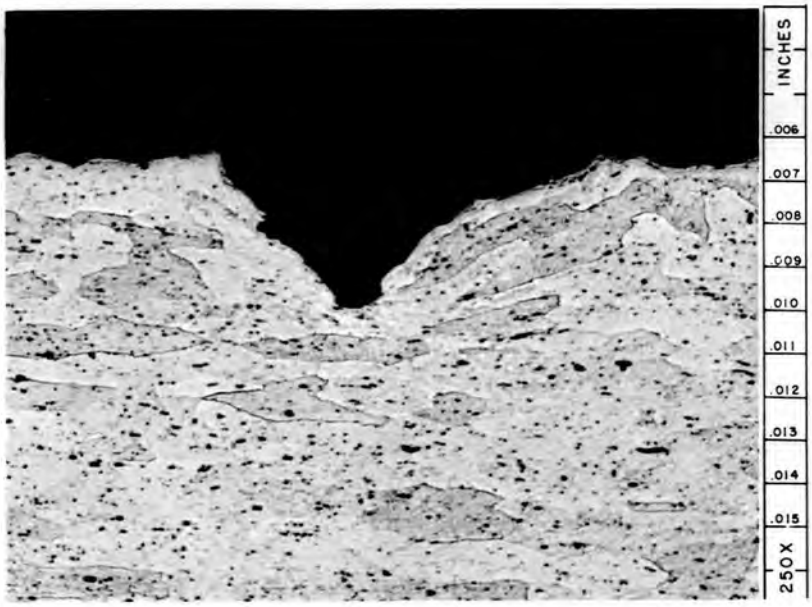


Fig. 6. Photomicrograph of Mechanical Defect in 1100 Alloy Showing Moderate to Light General Corrosion on the Surfaces. (Note the deformed microstructure around the defect.)

T-17019

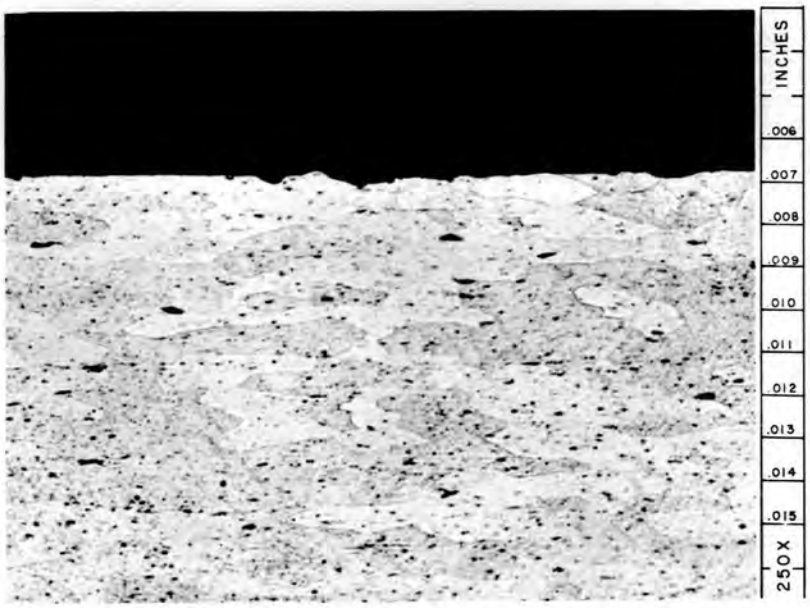


Fig. 7. Photomicrograph of Shallow Pits Found in 1100 Alloy Exposed for 500 hr in the ORR Core.

T-17023

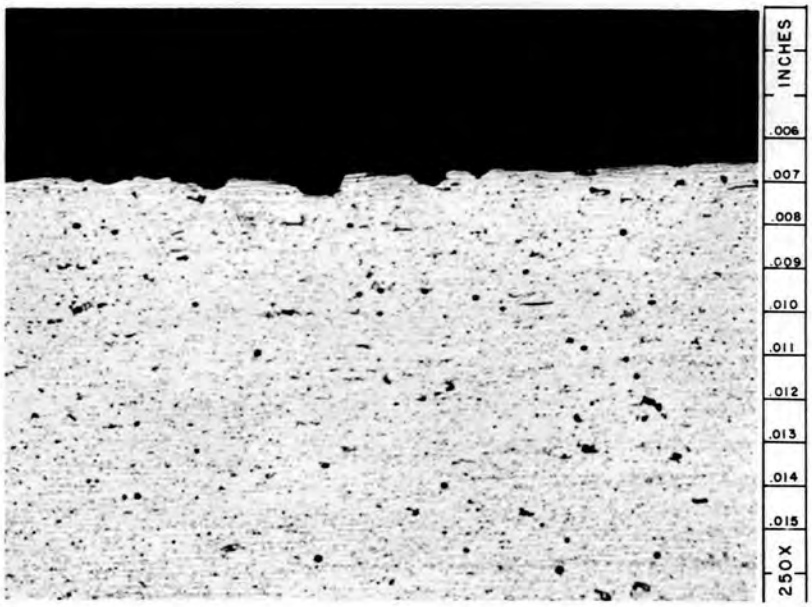


Fig. 8. Photomicrograph of 6061 Alloy Showing Shallow Pitting After 500 hr in ORR Core.

T-17022

Fig. 9. Photomicrograph of Shallow Pits Found in 5052 Alloy After 500 hr in ORR Core.

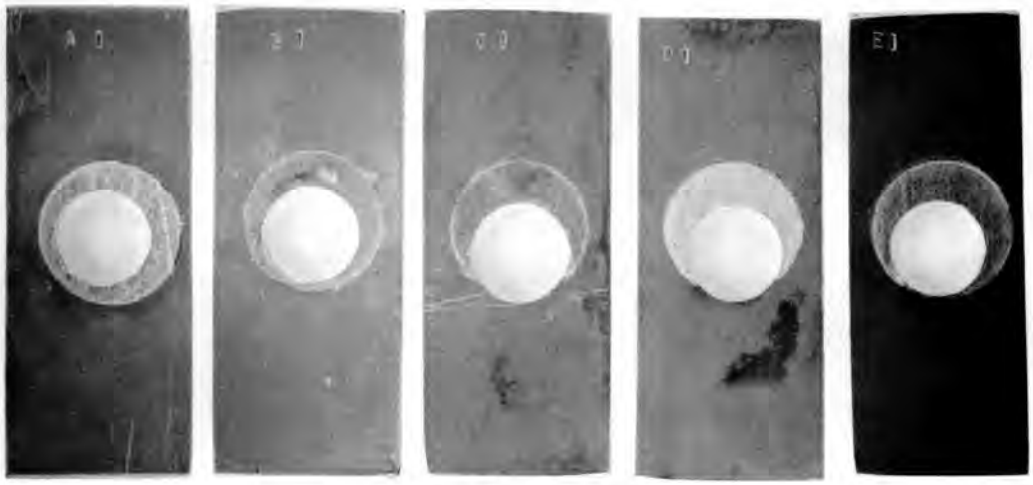
increasing exposure time. Slight staining of the surface was observed which was only partially removed by defilming. No evidence of pitting or localized corrosion was seen. Very light crevice corrosion was observed on a few coupons in the area between the coupon and the Teflon insulation.

Table 4. Corrosion Rates of Aluminum Alloys in ORR Core-Cooling Water (samples located at strainer in 24-in. exit line)

Alloy	Corrosion Rate					
	720 hr		4700 hr		8600 hr	
	(mg/cm^2)	(mpy)	(mg/cm^2)	(mpy)	(mg/cm^2)	(mpy)
1100	0.5	0.8	0.7	0.2	0.9	0.1
3003	0.5	0.8	0.7	0.2	0.9	0.1
5052	0.4	0.6	0.9	0.3	0.9	0.1
5154	0.4	0.6	0.8	0.2	0.9	0.1
6061	0.4	0.6	0.9	0.2	0.9	0.1

Figure 10 is a representative photograph showing typical coupons exposed in the strainer. The variations in shades of grey visible in the photographs resulted from differences in surface texture and reflectance. The dark areas are not stains but areas of low reflectance caused by surface roughening.

Plate 4418



1100 3003 5052 5154 6061

Fig. 10. The Appearance of Five Aluminum Alloys After 8600 hr in Strainer in ORR Core-Cooling System, Defilmed. (full size)

Pool Specimens

Although aluminum alloys tend to pit when immersed in static, oxygenated water, none of the coupons tested in the pool have exhibited any significant pitting for periods up to 8640 hr. Small spots of dark stain which suggest incipient pitting were observed, but metallographic examination of these areas failed to show more than a very slight attack (~0.1 mil). The weight-loss and corrosion-rate data are shown in Table 5. Figure 11 shows a group of specimens exposed in the pool for 8640 hr.

Table 5. Corrosion of Five Aluminum Alloys in the Center Section of the ORR Pool

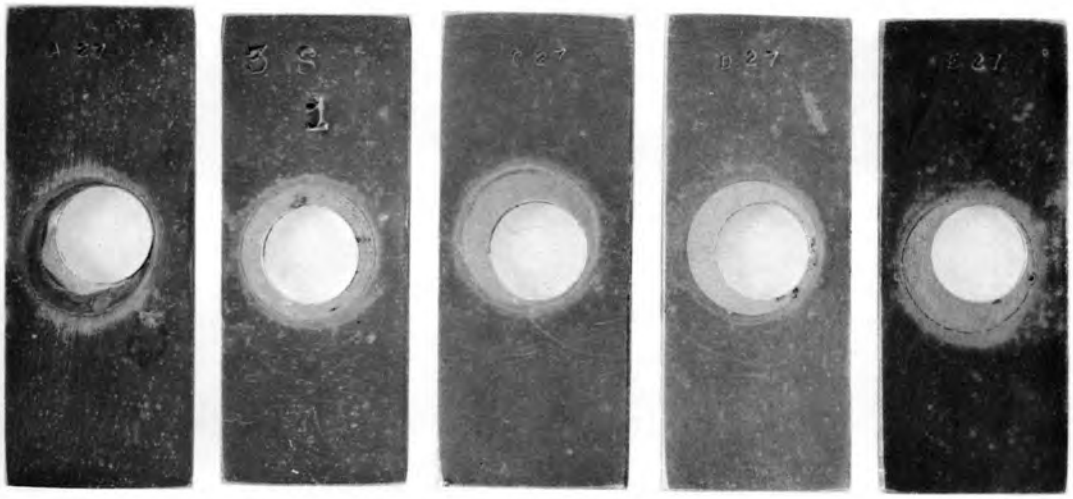
Alloy	672 hr		4656 hr		8640 hr	
	(mg/cm^2)	(mpy)	(mg/cm^2)	(mpy)	(mg/cm^2)	(mpy)
1100	<0.1	<0.1	0.1	<0.1	0.3	<0.1
3003	0.1	0.2	0.2	<0.1	0.2	<0.1
5052	<0.1	<0.1	0.1	<0.1	0.2	<0.1
5154	0.1	0.1	0.1	<0.1	0.2	<0.1
6061	<0.1	0.1	0.1	<0.1	0.2	<0.1

Secondary Side of Pool-Cooling Heat Exchanger

Table 6 lists the weight losses and calculated corrosion rates of the specimens exposed to the tower water, either at the outlet of the heat exchanger or in the cooling-tower sump. Since insertion of the samples in the heat-exchanger outlet required structural changes in the piping, no testing was done in that location until May, 1959. It is significant that the highest corrosion rates were found on the specimens in the cooling-tower sump prior to that time and that all subsequent tests showed lower and approximately the same corrosion rates regardless of the location. This is thought to be the result of better control of pH and chemical treatment achieved about this time. Figures 12 and 13 show the appearance of typical specimens after exposure in the cooling-tower water and after defilming.

Corrosion rates were low, and only a few pits were observed. Although the over-all appearance of all test specimens was satisfactory and the weight losses low, it must be pointed out that isolated examples of preferential attack were found by careful microscopic examinations. In the 92 specimens examined there were 6 to 10 defects which could be definitely classified as pits. A few instances of surface roughening were observed in small areas, and two cases of light, subsurface attack were discovered. The photomicrographs in Figs. 14 to 16 illustrate these types of attack. Because of the rarity of these occurrences, it is concluded that they were the result of metallurgical factors such as inclusions, inhomogenieties, or perhaps surface contamination in handling and preparing the alloys for testing, rather than interaction of the environment with the alloy of average composition. Figure 17 is a photomicrograph of an isolated pit found in one specimen after 720 hr in test.

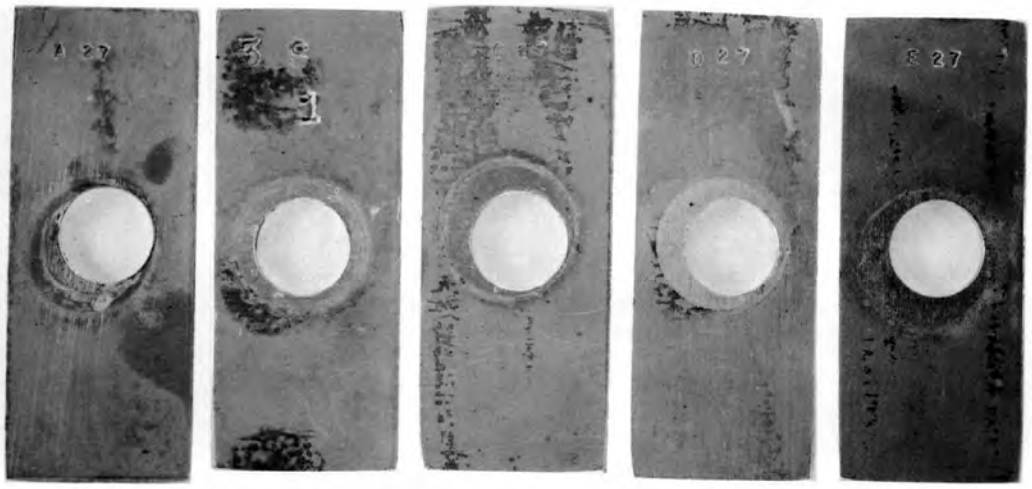
Plate 4417



1100 3003 5052 5154 6061

As Removed and Washed.

Plate 4421



1100 3003 5052 5154 6061

After Defilming.

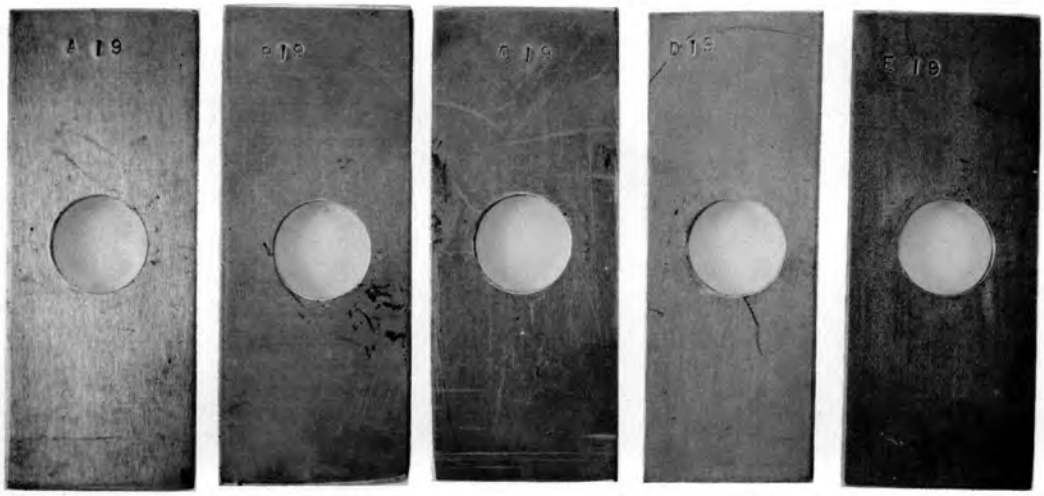
Fig. 11. The Appearance of Five Aluminum Alloys Exposed for 8640 hr in the Center Pool of the ORR (full size).

Table 6. Corrosion of Five Aluminum Alloys in the ORR Cooling-Tower Water

Time (hr)	Type 1100		Type 3003		Type 5052		Type 5154		Type 6061	
	(mg/cm^2)	(mpy)	(mg/cm^2)	(mpy)	(mg/cm^2)	(mpy)	(mg/cm^2)	(mpy)	(mg/cm^2)	(mpy)
					Cooling-Tower Sump					
190*	0.1	0.7	0.1	0.8	-	-	0.2	1.2	0.2	1.0
552**	0.0	0.0	<0.1	<0.1	<0.1	<0.1	0.0	0.0	0.0	0.0
2180*	1.1	0.6	0.5	0.3	-	-	1.2	0.7	0.5	0.3
4152*	1.1	0.3	0.1	WG	-	-	0.5	0.1	0.2	<0.1
4356**	<0.1	<0.1	WG	<0.1	0.0	0.0	<0.1	<0.1	WG	WG
5568*	1.8	0.4	0.8	0.2	-	-	2.0	0.5	1.5	0.3
8520**	<0.1	<0.1	<0.1	<1.1	0.0	0.0	<0.1	<0.1	WG	WG
					Heat-Exchanger Outlet					
720**	0.0	0.0	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	0.0
4707**	<0.1	<0.1	<0.1	0.0	<0.1	0.0	<0.1	<0.1	<0.1	<0.1
8664**	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1

* Run in 1958-59.
** Run in 1959-60.

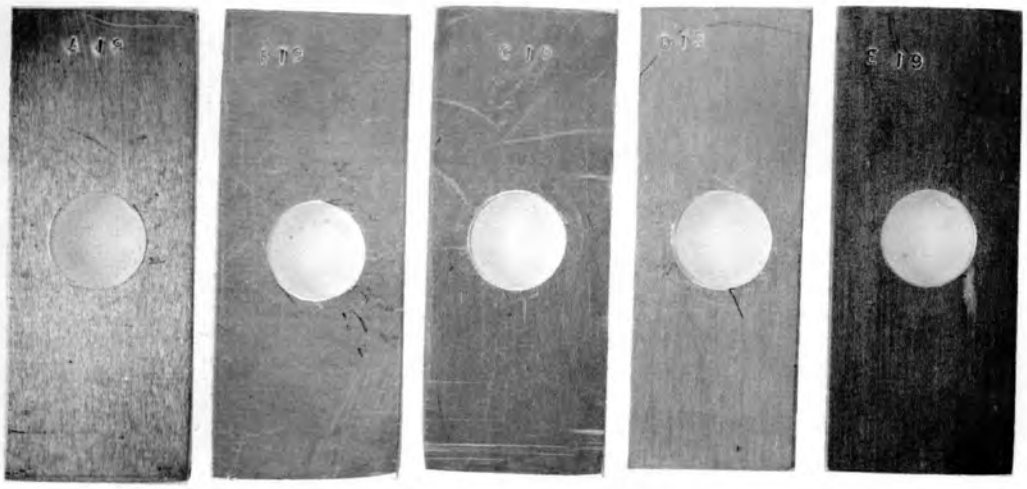
Plate 4416



1100 3003 5052 5154 6061

As Removed and Washed.

Plate 4420

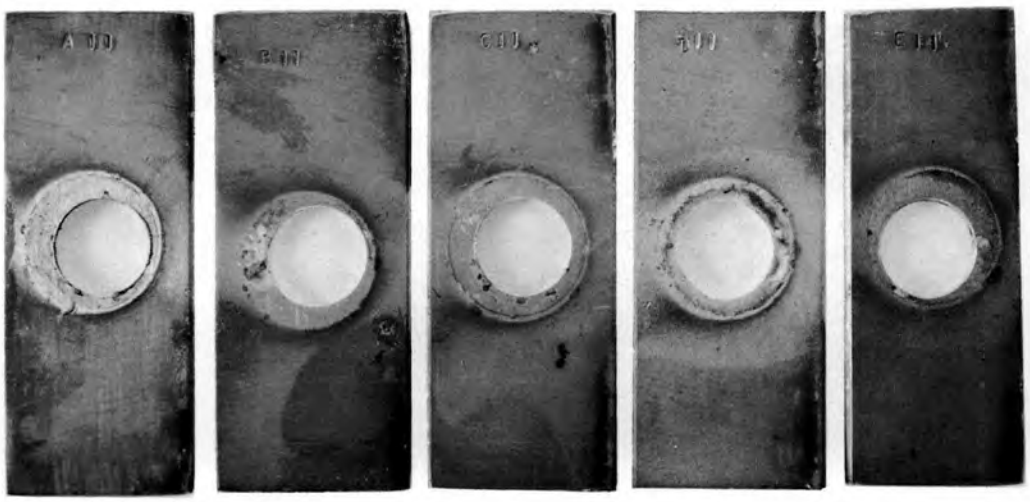


1100 3003 5052 5154 6061

After Defilming.

Fig. 12. Five Aluminum Alloys After 8520 hr Exposure in the Cooling-Tower Sump (full size).

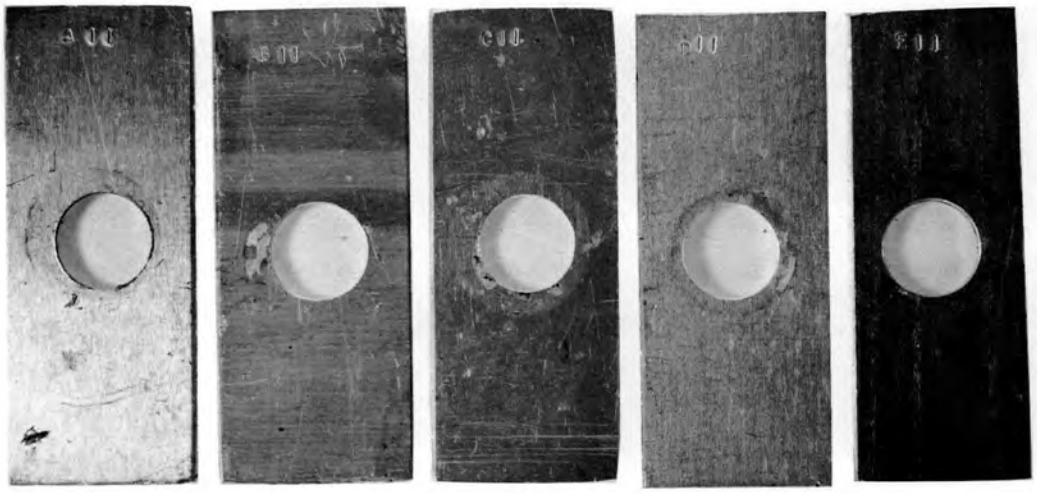
Plate 4415



1100 3003 5052 5154 6061

As Removed and Washed.

Plate 4419



1100 3003 5052 5154 6061

After Defilming.

Fig. 13. The Appearance of Five Aluminum Alloys After 8664 hr in Heat-Exchanger Outlet Pipe (full size).

T17043

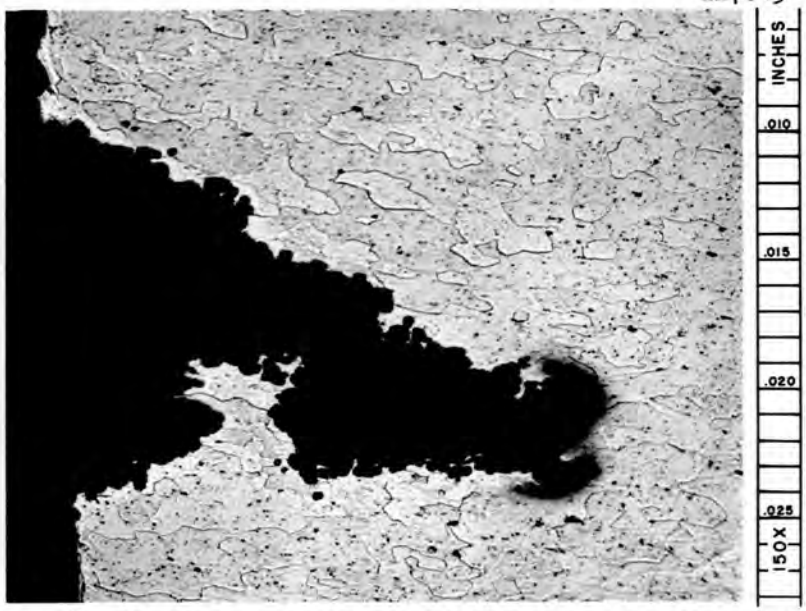


Fig. 14. Photomicrograph of a Pit Found on the Edge of One Specimen of 1100 Aluminum Alloy. Exposed in Cooling-Tower Sump for 5568 hr.
(Magnification 150X)

T17034

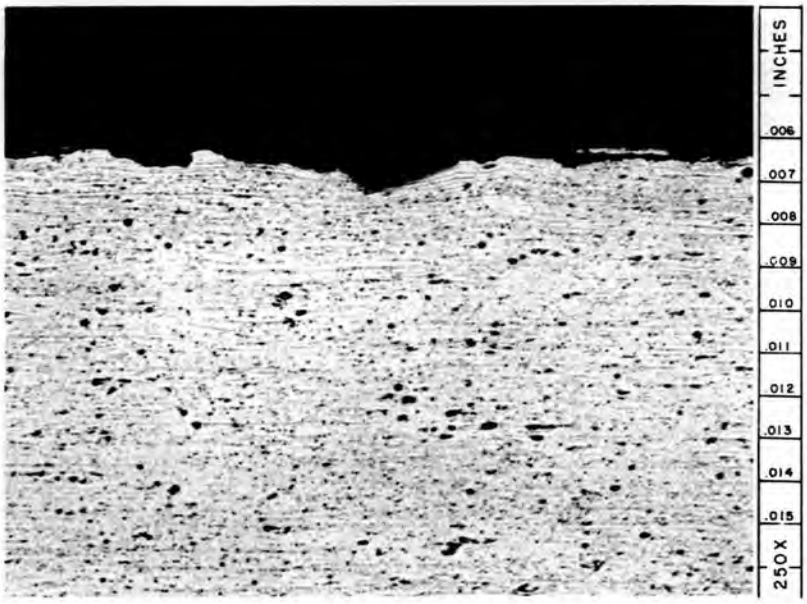


Fig. 15. Photomicrograph of Surface Roughening in 6061 Aluminum Alloy After 5568 hr in Cooling-Tower Sump. Some Mechanical Deformation is Also Visible.
(Magnification 250X)

T17830

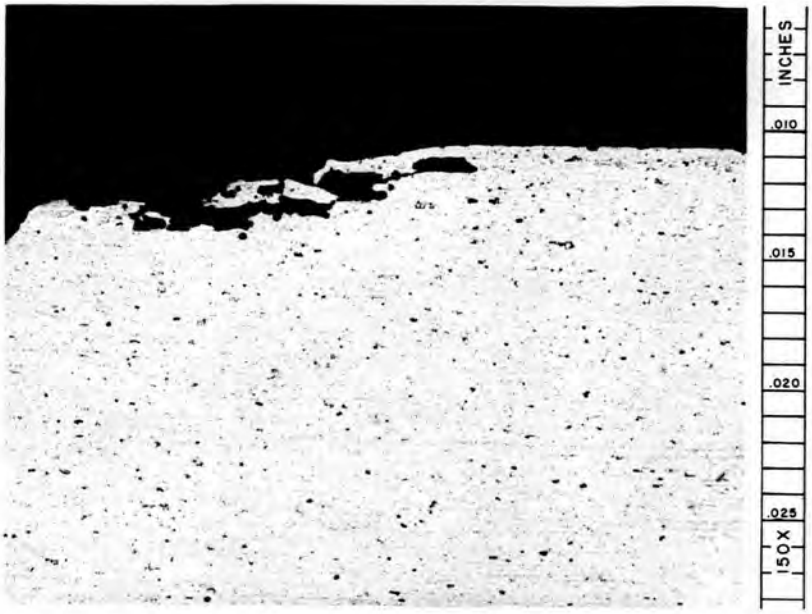


Fig. 16. Photomicrograph of Subsurface Attack in 6061 Aluminum Alloy Exposed for 4512 hr in the Cooling-Tower Sump (Magnification 150X).

T17899

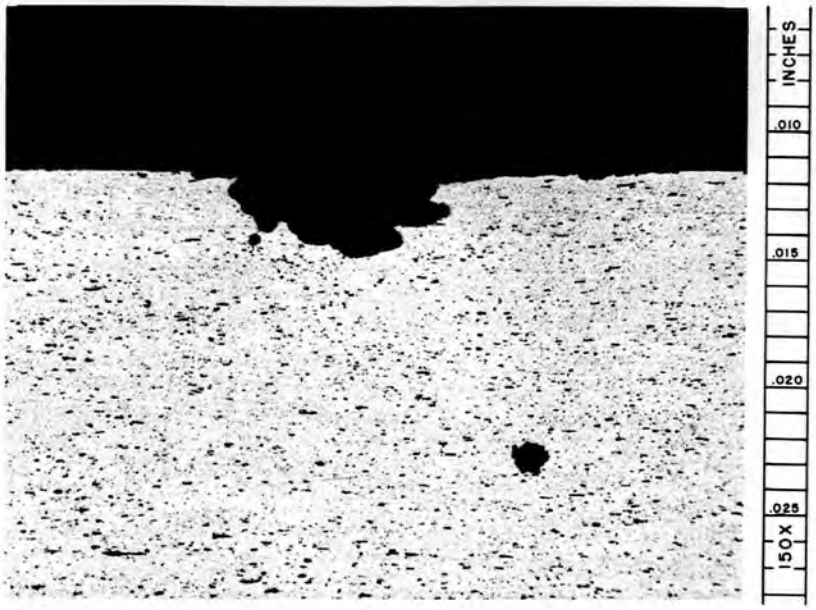


Fig. 17. Photomicrograph of a Pit Formed in 3003 Aluminum Alloy After 720 hr in the Secondary-Side Coolant Water at the Heat-Exchanger Outlet (Magnification 150X).

Inspection of the Pool Heat Exchanger

During the scheduled shutdown of the ORR during the week of November 3, 1958, the pool-cooling-water heat exchanger was disassembled and examined. At this time, water had been circulating in the secondary system for approximately six months. Very light, general corrosion was observed and only one deep pit could be seen after a thorough examination. There was, however, evidence of very minor localized attack over large areas of the tubes as shown in Fig. 18. This may be associated with the thin layer of deposit found over the entire tube bundle. Chemical analysis of this deposit showed the major constituents to be silicon, aluminum, iron, zinc, and calcium with traces of copper and lead. The analysis indicates that the system was operated for at least a part of the time with insufficient chemical treatment.

Photo 45513

Fig. 18. ORR Pool-Cooling Heat-Exchanger Tube Bundle After Six Months of Service.

Although this film would not, per se, be cause for great concern, the presence of small amounts of lead and copper in the film indicate that pitting may be expected after prolonged use unless the source of these metals is removed. None of the components of the secondary system is known to contain lead or copper; however, on one occasion several dozen short lengths of 16-gage copper wire were removed from the cooling-tower basin where they were evidently dropped by workmen replacing some of the insulation on the piping.

When the heat exchanger was examined again after 13 months of service, it was noticeably cleaner than on the previous occasion. The scale had all but disappeared, and only small quantities of solids were found in the shell.

CONCLUSIONS

Based on the generalized corrosion rates observed for all five aluminum alloys after long periods of testing, the resistance of these alloys to corrosion by the coolants in the ORR appears to be excellent. Assuming only a 5-mil corrosion allowance, a service life of 50 years would be predicted. It must be pointed out, however, that isolated defects in the alloys could result in a need for repair of particular components in much less time.

The corrosiveness of the coolants to the alloys of average composition can be controlled by proper attention to water treatment and system cleanliness; the failure of a component due to inclusions in the metal surface or inhomogeneities in the alloy cannot be economically controlled or predicted.

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