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DESIGN CONSIDERATIONS OF REACTOR CONTAINMENT SPRAY SYSTEMS – PART III. THE CORROSION OF MATERIALS IN SPRAY SOLUTIONS

J. C. Griess
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REACTOR DIVISION

DESIGN CONSIDERATIONS OF REACTOR CONTAINMENT SPRAY SYSTEMS – PART III. THE CORROSION OF MATERIALS IN SPRAY SOLUTIONS

J. C. Griess A. L. Bacarella

DECEMBER 1969

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FOREWORD

The Spray and Absorption Technology Program is coordinated by Oak Ridge National Laboratory for the AEC. The program involves research on all aspects of containment spray systems proposed for use as an engineered safety feature in pressurized water reactor containment buildings and investigations of certain aspects of the pool-pressure-suppression containment concept as applied to boiling water reactors.

The work reported here represents the final report on one of the program tasks, a study of the corrosive nature of proposed spray solutions. As stated in the abstract, "the corrosive nature of the solution must be such that the containment and emergency equipment must not be jeopardized, thereby propagating the accident." The information presented should serve as a guide for plant design as far as materials specification is concerned.

Work on protective coatings will be given in another report in this series to be issued soon.

Additional reports in this series include:

T. H. Row, L. F. Parsly, and H. E. Zittel, *Design Considerations of Reactor Containment Spray Systems – Part I,* USAEC Report ORNL-TM-2412, April 1969.

C. Stuart Patterson and Wm. T. Humphries, *Design Considerations of Reactor Containment Spray Systems – Part II. Removal of Iodine and Methyl Iodide from Air by Liquid Solutions,* USAEC Report ORNL-TM-2412, August 1969.

Thomas H. Row
Technical Coordinator
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CONTENTS

FOREWORD iii

ABSTRACT 1

INTRODUCTION 1

EXPERIMENTAL PROCEDURES 1

RESULTS 5

- Corrosion of Aluminum Alloys 5
- Corrosion of Ferrous Alloys 9
- Corrosion of Copper Alloys 11
- Corrosion of Galvanized Steel and Zinc 12
- Corrosion of Other Materials 13
- Concrete in Spray Solutions 14
- Solution Stability 15

DISCUSSION 15

SUMMARY 16

DESIGN CONSIDERATIONS OF REACTOR CONTAINMENT SPRAY SYSTEMS – PART III. THE CORROSION OF MATERIALS IN SPRAY SOLUTIONS

J. C. Griess A. L. Bacarella

ABSTRACT

The use of aqueous solutions containing various chemicals for nuclear reactivity control and iodine absorption has been proposed as a safeguard system in water-cooled nuclear reactors. In the event of a design basis accident such solutions would be sprayed into the containment building to reduce the steam pressure and to scavenge iodine from the atmosphere. For such a system to be practical it must not only accomplish the above two objectives but the corrosive nature of the solution must be such that the containment and emergency equipment must not be jeopardized, thereby propagating the accident. This program was undertaken to determine the corrosion rates of materials normally found in reactors and their containment buildings in spray solutions. Specimens of materials were exposed both in the spray and totally submerged, over the temperature range of 50 to 140°C. The results of these tests are presented in this report.

INTRODUCTION

A significant number of aqueous cooled nuclear power plants will use containment building spray systems for pressure reduction and fission product absorption in the event of a design basis accident. Since from a biological point of view iodine is the most hazardous fission product, materials added to the spray solutions are primarily for absorption and retention of iodine released to the containment building.

The sprays as currently envisioned will be alkaline borate solutions with or without the addition of sodium thiosulfate. It was the purpose of this investigation to determine the corrosion behavior of a variety of metals and alloys in sprays of these solutions and when totally immersed in them. The reason for conducting the corrosion study was to assure that the use of sprays would not further propagate the accident by violating the containment or cause degradation of necessary emergency equipment as a result of corrosive attack.

EXPERIMENTAL PROCEDURES

All of the tests were conducted in a test vessel made of 304 stainless steel. Figure 1 is a sketch of the apparatus showing the dimensions of the overall facility and the various components in the system.

The loop was designed to operate at pressures up to 150 psig and contained provisions for removing samples from the liquid and gas phases. A canned-rotor pump recirculated the test solution. Eight thermocouples were positioned at various places on the loop. One located immediately ahead of the spray nozzle was used to control the temperature of the spray solution. Two thermocouples were located in wells in the spray chamber; one terminated near the top and was in the spray region and the other extended to within 8 in. of the bottom and was always in the liquid phase. The solution that flowed from the spray chamber passed through a fine-mesh screen and was then cooled slightly to prevent possible cavitation of the pump. After leaving the pump it was heated (or cooled) to the desired temperature by a heater-cooler in the line to the nozzle. This line also contained a flowmeter to monitor the flow rate of solution passing through the nozzle.

A full-cone center-jet nozzle (Spray Engineering Company, Burlington, Massachusetts, nozzle 3C) with a flow rate of about 0.5 gpm was used in all tests. The orifice in the nozzle was 0.093 in.

Specimens were suspended either in the spray or totally submerged on a stainless steel rack which contained 108 Teflon-insulated hangers. The depth of liquid in the spray chamber was 24 in. in most cases, and thus the distance through which the spray fell was usually 28 in.

Figure 2 is a photograph of the test facility before the system was thermally insulated. The sample rack is shown at the left.

The specimens were usually rectangular coupons with a ¼-in. hole drilled near one end so that they could be

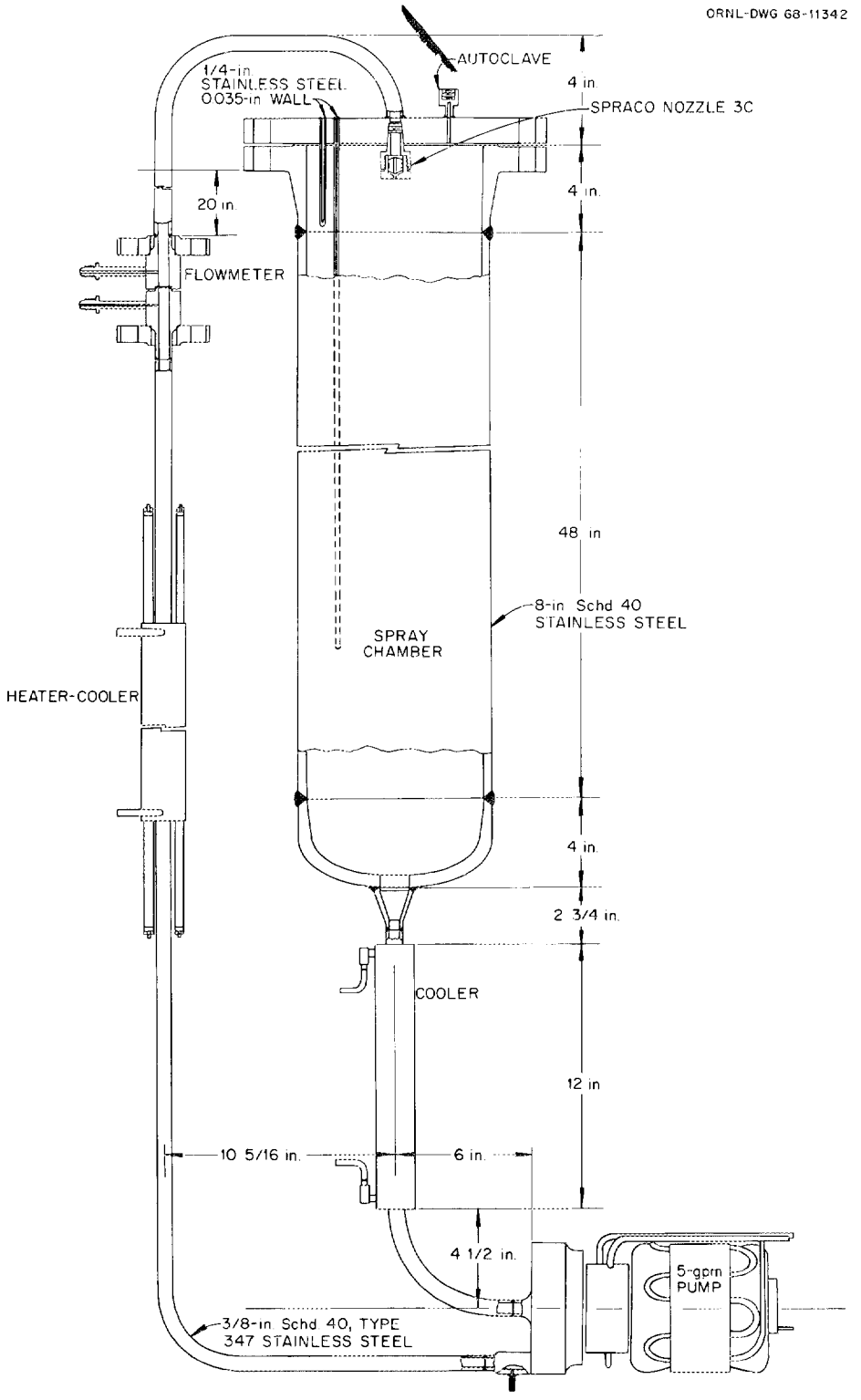

Fig. 1. Sketch of Test Facility.

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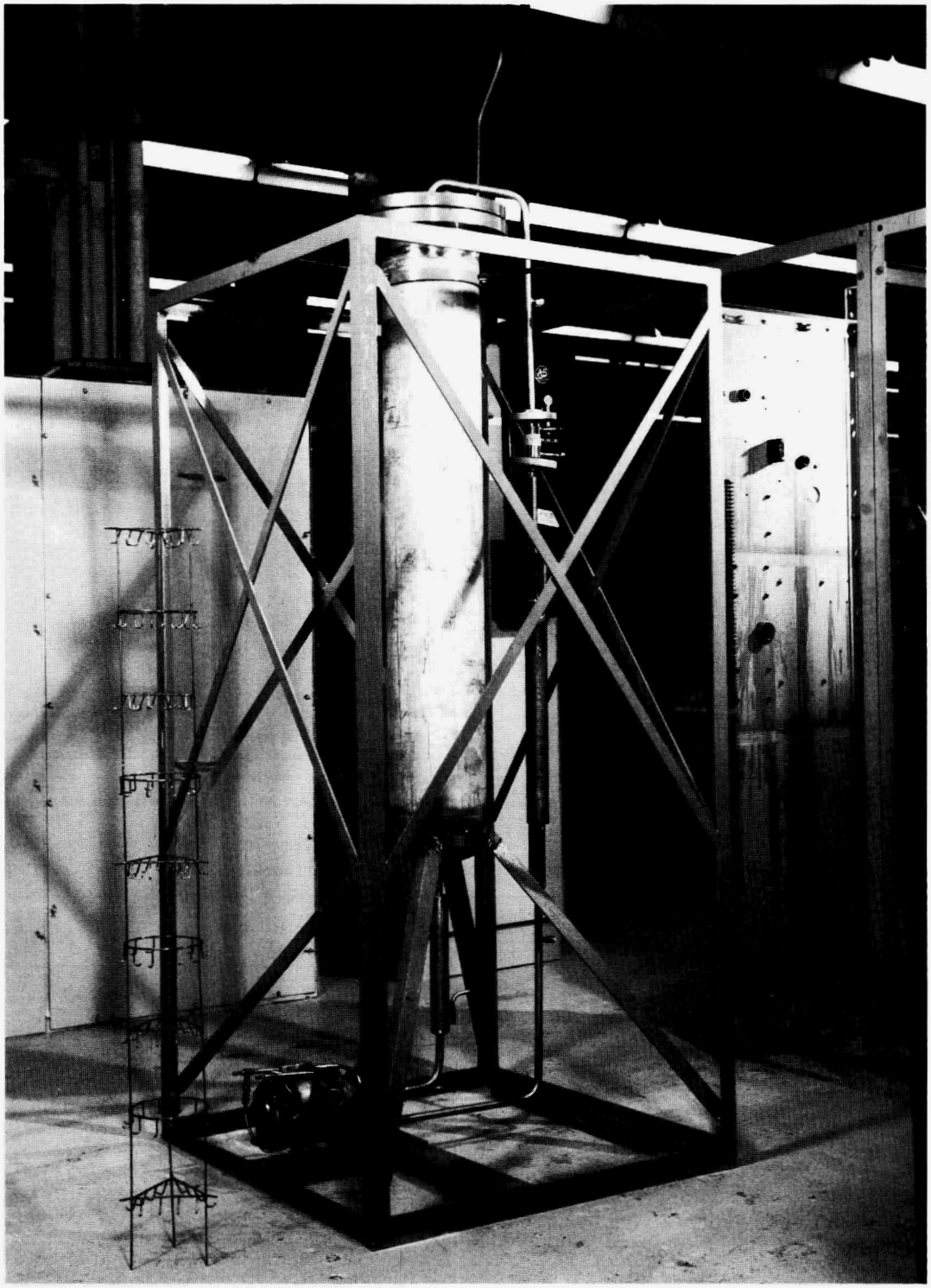

Fig. 2. Photograph of Test Facility. Specimen rack is shown on left.

hung on the rack. All were lightly abraded, degreased, and weighed before being placed in test. At the end of the test, the specimens were scrubbed with a soft brush, dried, and weighed. In those cases where there appeared to be significant amounts of corrosion products on the surfaces, the specimens were descaled and reweighed. Corrosion products were removed from the aluminum alloys in a solution containing 20 g CrO_3 and 60 g H_3PO_4 (85%) per liter. The other materials were cathodically descaled in inhibited 5% sulfuric acid solution. In both cases additional loss of metal was negligibly small.

In some tests U-bend specimens were used to determine the susceptibility of materials to stress corrosion cracking. These specimens were die-punched from thin sheets to $^5/_8$- by 3-in. strips with $^1/_4$-in.-diam holes at either end. The strips were formed into U-bend specimens in a simple bending machine, and the legs of the U were drawn parallel with tie bolts. Thus the specimens were both elastically and plastically deformed. The radius of curvature was $^5/_{16}$ in.

Only two different solutions were used in these tests. One contained 0.15 *m* NaOH and 0.28 *m* H_3BO_3 (3000 ppm B); the second contained the same concentrations of the two reagents and in addition 1 w/o $Na_2S_2O_3$ (0.064 *m*). The solutions were made using reagent-grade chemicals and deionized water. In all cases an atmosphere of air filled the free volume of the test facility at the time of closure.

Table 1 shows the conditions of the runs made in which at least some metal specimens were exposed. In runs 7 and 8 (not shown) only concrete test cylinders were exposed. Runs 11 and 12 were conducted primarily for the purpose of exposing protective coatings to simulated spray environments, and these tests will be the topic of a separate report in this series; however, in these two runs, specimens of welded stainless steel were also included.

Table 2 lists the alloys that were tested and their nominal compositions.

Table 1. Experimental Conditions of Tests

Run No.	Solution	Temperature (°C)	Time (hr)
1	AB[a]	100	168
2	ABT[b]	100	168
3	AB	140	24
4	ABT	140	24
5	AB	55	518
6	ABT	55	518
9	AB + 100 ppm Cl	140	24
		100	168
		55	504
10	ABT	140	14
		100	168
11	AB	149	0.08
		141	1.7
		107	22.3
		66	338
12	ABT	Same as run 11	
13	AB + 8 ppm I	Same as run 11	
14	AB + 8 ppm I	Same as run 11	

[a]AB = 0.15 *m* NaOH–0.28 *m* H_3BO_3.
[b]ABT = 0.15 *m* NaOH–0.28 *m* H_3BO_3–0.064 *m* $Na_2S_2O_3$.

Table 2. Nominal Compositions of Alloys Tested

Alloy	Weight Percent						
	Ni	Cr	Fe	Si	Cu	Mn	Other
304 stainless steel	9.5	19	Bal			2.0 max	0.08 C max
316 stainless steel	12	17	Bal			2.0 max	0.10 C max, 2.5 Mo
A108 steel			Bal			0.5	0.20 C, 0.05 S, 0.04 P
A210 steel			Bal	0.1		0.9	0.27 C, 0.05 S, 0.04 P
Inconel 600	76	16	7			0.2 max	0.04 C
Inconel 718	53	19	18			0.2	0.04 C, 5 Nb, 3 Mo, 0.8 Ti, 0.6 Al
Monel 400	66		2.5 max		Bal	2.0 max	0.30 C max
90-10 cupronickel	10		1.25		Bal	1.0 max	1.0 Zn max
70-30 cupronickel	30		0.5			1.0 max	1.0 Zn max
Admiralty brass			0.06 max		Bal		28 Zn, 1 Sn, 0.05 As
Zircaloy 2	0.05	0.10	0.15				1.4 Sn, bal Zr
1100 aluminum			(Si + Fe, 1.0 max)				99.0 Al min
3003 aluminum			0.7	0.6		1.3	Bal Al
5052 aluminum		0.25	(Si + Fe, 0.45 max)				2.5 Mg, bal Al
5154 aluminum		0.25	(Si + Fe, 0.45 max)				3.4 Mg, bal Al
5454 aluminum		0.15	(Si + Fe, 0.40 max)			0.8	2.7 Mg, bal Al
6061 aluminum		0.25	0.7	0.6	0.3	0.15 max	1 Mg, bal Al

RESULTS

Corrosion of Aluminum Alloys

The corrosion rates observed with six aluminum alloys in the standard alkaline borate solution at 55°C are shown in Table 3. Duplicate specimens of each alloy were exposed both in the spray and totally immersed in the solution. All specimens were weighed after cumulative exposure times of 44, 309, and 518 hr, after which they were descaled. The weights shown in the table for the 518-hr exposure represent weight losses after descaling, whereas at the other two time intervals the specimens contained an indeterminate weight of corrosion products. However, the weights of surface deposits after the total exposure varied only from 0.3 to 2.0 mg/cm^2, a small amount compared with the overall weight losses.

The data for most of the alloys indicate an essentially linear corrosion rate after some brief period of accelerated attack, as shown in Figs. 3 and 4, which are graphs of average weight loss vs time for spray- and solution-exposed specimens respectively. In the spray, corrosion rates ranged between 130 and 190 mils/year; totally submerged specimens corroded at rates of 45 to 78 mils/year. The 5154 alloy indicated a low corrosion rate for the first two periods, but during the last time interval the average rate was 49 mils/year. All alloys corroded more or less uniformly, and although the rates appeared to differ slightly from alloy to alloy, from the

Table 3. The Corrosion of Aluminum Alloys in 0.15 *m* NaOH–0.28 *m* H_3BO_3 at 55°C

Aluminum Alloy	Weight Loss (mg/cm^2)					
	Spray			Solution		
	44 hr	309 hr	518 hr	44 hr	309 hr	518 hr
1100	9.5	44.1	68.7	7.6	20.4	36.5
	9.2	42.9	69.0	13.2	27.0	43.9
3003	10.3	30.0	59.3	6.6	15.2	25.9
	8.9	35.5	59.9	8.7	21.1	35.2
5052	9.6	31.4	61.0	2.6	9.9	22.7
	9.0	31.0	59.7	3.6	14.8	28.5
5154	10.3	48.6	80.1	0.4	2.5	10.6
	10.2	46.9	81.7	0.4	2.4	10.6
5454	6.8	32.2	58.2	3.3	14.7	28.9
	9.3	32.0	56.0	0.6	3.6	12.0
6061	10.5	44.7	74.1	5.7	16.8	28.8
	8.8	36.2	61.2	2.4	16.0	30.5

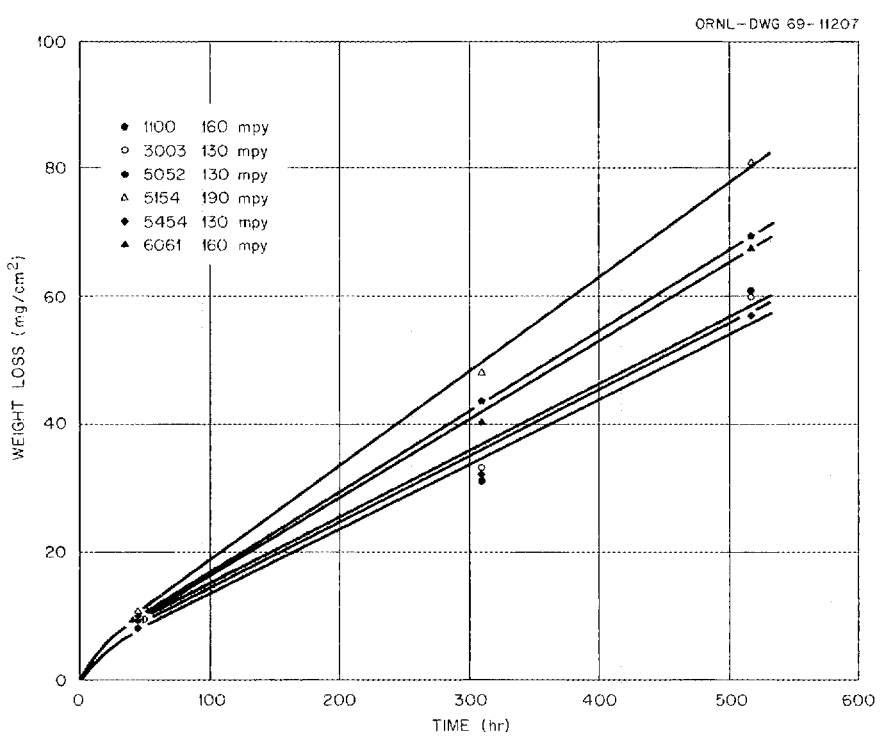

Fig. 3. The Corrosion of Aluminum Alloys in a Spray of a 0.15 *m* NaOH–0.28 *m* H_3BO_3 Solution at 55°C.

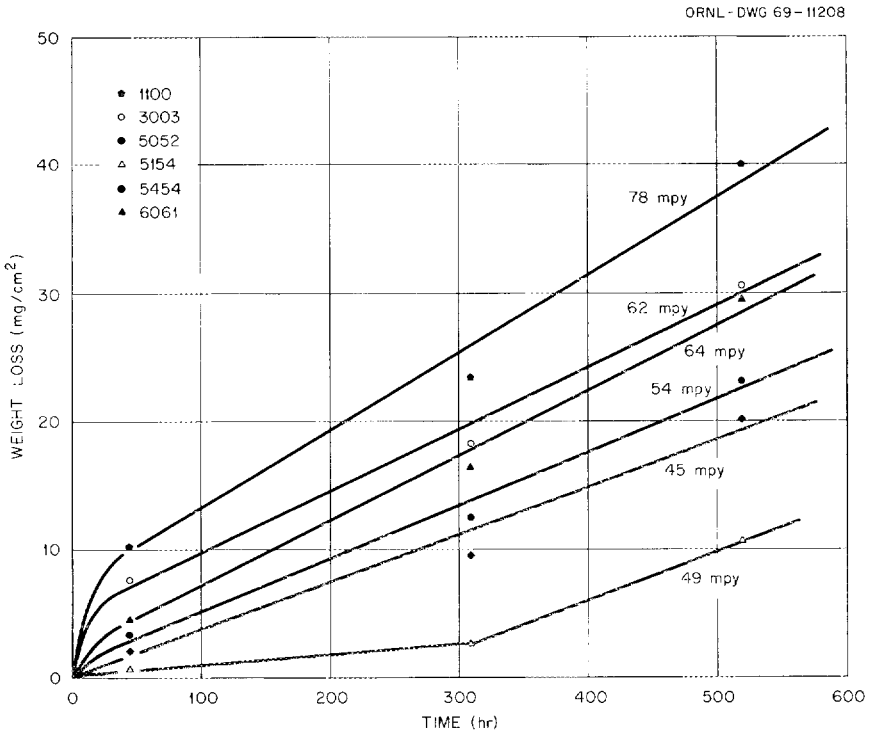

Fig. 4. The Corrosion of Aluminum Alloys Immersed in a 0.15 *m* NaOH–0.28 *m* H_3BO_3 Solution at 55°C.

corrosion standpoint there was little to recommend one alloy over another.

Four different aluminum alloys were exposed to the spray and totally submerged in the standard alkaline borate solution at 100°C. The test lasted for 168 hr, and the specimens were examined after cumulative exposure times of 24 and 72 hr. After 72 hr the extent of corrosion on the 1100, 3003, and 6061 specimens was so great that they were removed from the system; only the 5052 alloy specimens remained in test for the full 168-hr period. Table 4 shows the weight losses observed on all specimens. At the end of the test only the 5052 specimens were descaled, and the weight losses in the table for the 168-hr interval are descaled weight losses. All other alloys corroded so extensively that the weight of any corrosion product was insignificant.

The results indicate very high weight losses for all alloys except the 5052 alloy. Generally the weight losses were lower on the totally submerged specimens than on those in the spray, and the agreement between replicate specimens was generally better in the solution. Only uniform corrosion was observed on those specimens exposed in the solution. In the spray, attack was usually not uniform, with large areas of specimens sometimes heavily attacked while other areas were

Table 4. The Corrosion of Aluminum Alloys in 0.15 *m* NaOH–0.28 *m* H_3BO_3 at 100°C

Alloy	Weight Loss (mg/cm²)					
	Spray			Solution		
	24 hr	72 hr	168 hr	24 hr	72 hr	168 hr
1100	69.3	145		63.5	168	
	81.2	166		64.9	175	
	66.6	193				
	75.8	76				
3003	66.2	171		29.0	65.5	
	52.3	137		27.8	63.2	
	32.9	99				
	28.1	64				
5052	11.1	11.3	16.2	1.10	0.94	4.4
	7.3	7.1	19.8	1.10	0.95	4.6
	1.1	1.2	9.9			
	1.3	1.1	9.1			
6061	39.5	11.3		29.3	77.5	
	33.0	68		27.8	78.9	
	35.4	96				
	46.0	86				

relatively lightly attacked (see Fig. 6 as an example). For reference, a weight loss of 100 mg/cm^2 during a 72-hr period corresponds to an average corrosion rate of 1800 mils/year.

The one alloy that showed relatively good corrosion resistance in this environment was 5052 aluminum. Both in the solution and in the spray, the alloy developed a very uniform, black, smooth coating during the first exposure period, and this film appeared to minimize further corrosion. No form of localized attack was apparent on any of the specimens. The surfaces were as smooth and uniform as when the test started.

Because of the high corrosion rates observed with the other alloys at 100°C, only the 5052 alloy was exposed in the standard alkaline borate solution at 140°C. The test lasted for only 24 hr, and after removal of the corrosion products the following weight losses were observed on duplicate specimens exposed in the spray and in the solution: spray specimens: 16.7 and 25.0 mg/cm^2; solution specimens: 15.7 and 15.8 mg/cm^2.

In both locations, black, smooth corrosion product films covered the specimens. Although the observed weight losses correspond to high corrosion rates (average of 1100 mils/year in the spray and 840 mils/year in solution), the appearance of the specimens indicated that continuing rates would have been substantially lower. As at 100°C the attack was perfectly uniform.

Fewer but comparable data were obtained with some of the same aluminum alloys in the alkaline borate solution containing thiosulfate. In an initial test at 100°C, 1100, 3003, 5052, and 6061 aluminum specimens were exposed for 24 hr. In a subsequent test which lasted for 168 hr, only specimens of the 5052 and 6061 alloys were included in the sample array. The weight losses are shown in Table 5.

The 1100, 3003, and 6061 specimens corroded at very high rates, with the specimens in the spray generally showing greater attack than those in the solution. Attack was generally uniform in the solution but irregular in the spray region. Based on the 24-hr results, corrosion rates for the three alloys ranged from 4300 to 1200 mils/year in the spray and from 2300 to 1800 mils/year in the solution.

With the exception of one specimen exposed in the spray for 24 hr, the 5052 aluminum specimens were much more resistant than the other alloys. The single specimen underwent heavy attack on one side and did not develop a black film in that area. Films identical in appearance to those formed in the alkaline borate solution were present on all other specimens, and relatively little corrosion occurred. As indicated by the footnote in Table 5, those 5052 specimens exposed at 100°C for 168 hr had a prior exposure of 14 hr at 140°C. The weight losses shown in Table 5 probably occurred during film formation at the higher temperature, with very little attack occurring during the 168-hr period at 100°C. Figure 5 is a photograph of the 5052 aluminum specimens after film removal and shows the very uniform attack that was observed.

Table 5. The Corrosion of Aluminum Alloys in 0.15 *m* NaOH–0.28 *m* H_3BO_3–0.064 *m* $Na_2S_2O_3$ at 100°C

Alloy	Weight Loss (mg/cm^2)			
	Spray		Solution	
	24 hr	168 hr	24 hr	168 hr
1100	53.2		43.0	
	60.3		42.3	
	72.2			
	52.2			
3003	80.5		34.1	
	46.8		40.3	
	32.3			
	52.4			
5052	32.7	9.4[a]	2.56	4.5[a]
	2.6	6.1[a]	2.63	4.5[a]
	2.3	5.7[a]		4.7[a]
	3.0			
6061	73.2	217	36.3	124
	22.5	158	39.6	127
	28.9	219		126
	36.0			

[a]Specimens had prior exposure for 14 hr to the same solution at 140°C. Weight loss represents the total weight loss for both periods.

Specimens of 6061 aluminum were also exposed for 14 hr at 140°C, but the attack during this brief period was so great that new specimens were used for the 168-hr period at 100°C. Triplicate specimens exposed in the spray for 14 hr at 140°C showed weight losses of 148, 126, and 90 mg/cm^2; in solution the weight losses were 256, 268, and 267 mg/cm^2. These weight losses correspond to average corrosion rates of 30 mils penetration per day in the spray and 66 mils penetration per day in the solution. Photographs of these specimens are shown in Fig. 6. The irregular nature of the attack in the spray and the relatively uniform attack in solution is readily apparent.

Runs 13 and 14 were conducted to determine the effect of iodide ions on corrosion in general, and in the case of aluminum alloys to determine whether metallurgical condition or anodized coatings had an effect on corrosion. Furthermore, since the temperature was

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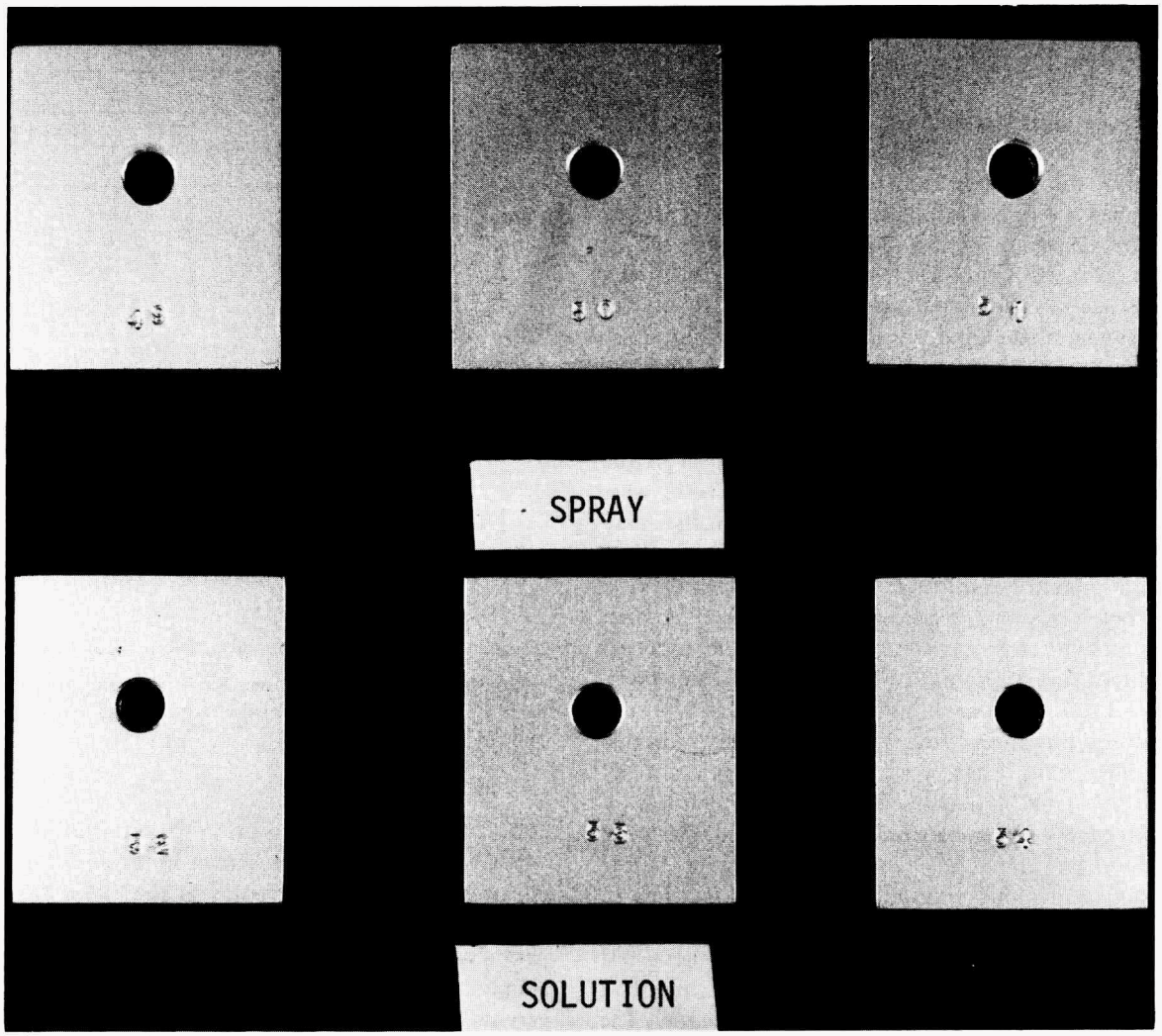

Fig. 5. 5052 Aluminum Specimens After 14 hr at 140°C and 168 hr at 100°C in a 0.15 *m* NaOH–0.28 *m* H_3BO_3–0.064 *m* $Na_2S_2O_3$ Solution. Specimens have been defilmed.

decreased during the run, the stability of the protective coating formed on 5052 aluminum at high temperature when subsequently exposed at low temperature could be evaluated. The results indicated that iodide had no discernible effect on the corrosion of aluminum. Corrosion damage to 1100, 6061, and 3003 aluminum was essentially what was estimated from data obtained at other temperatures. The data also clearly showed that the protective coating formed on 5052 alloy during exposure at temperatures of 149 to 107°C for 24 hr apparently remained intact at the lower temperature during the following 336 hr at 66°C; weight losses on all specimens during each run (Nos. 13 and 14) ranged between 3.8 and 7.6 mg/cm^2 both in the spray and in solution. For comparison, 6061 specimens showed weight losses ranging between 9 and 48 mg/cm^2 in the spray and between 69 and 108 mg/cm^2 totally submerged. The 1100 alloy was even more severely attacked; weight losses between 45 and 157 and between 210 and 242 mg/cm^2 were observed in the spray and solution respectively.

The 6061 aluminum alloy was exposed in a variety of different metallurgical conditions and forms during run 14. These included pipes, angle irons, anodized surfaces, and plates in the T6 and T0 condition both as received and after rolling. All of the above specimens corroded

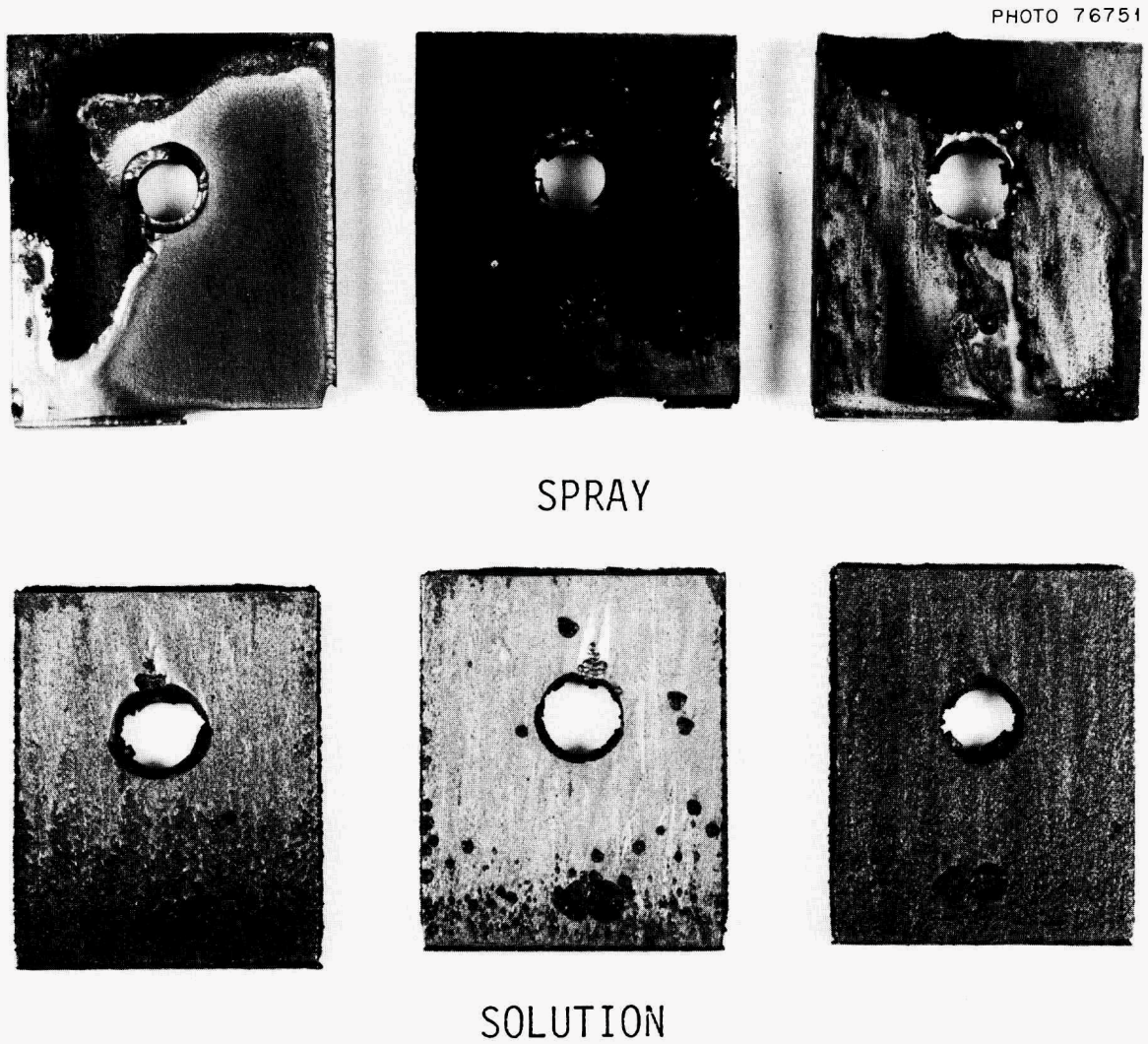

Fig. 6. 6061 Aluminum Specimens Exposed to 0.15 *m* NaOH–0.28 *m* H_3BO_3–0.064 *m* $Na_2S_2O_3$ Solution for 14 hr at 140°C.

to the same extent, and weight losses fell within the ranges listed above. Anodized specimens of 5052 lost about the same weight as did the abraded surfaces.

Corrosion of Ferrous Alloys

Specimens of A-108 and A-210 carbon steel were exposed in all tests shown in Table 1 except during runs 9, 11, and 12. In all runs with the standard alkaline borate solution, essentially no attack was noted on either alloy regardless of exposure conditions; an occasional small area of rust was observed on some of the specimens in the spray, but weight changes on all the specimens were within the weighing precision.

Attack was also minor in the thiosulfate-containing solutions, although occasionally light pitting was observed, and weight losses could be measured in most cases. From a corrosion point of view there was no difference between the two alloys. At 100°C (run 2) weight losses averaged 3.95 mg/cm^2 for eight specimens exposed to the spray and 2.69 mg/cm^2 for four specimens exposed in the solution. For the 168-hr run these weight losses correspond to average corrosion rates of 10 and 7 mils/year respectively. On all specimens isolated areas of rust were found, under which pits as deep as 5 mils were observed. On the other hand in run 10, where the specimens were at 140°C for 14 hr before exposure for 168 hr at 100°C,

weight losses for the two alloys averaged only 0.08 mg/cm^2, and pitting was not evident. In run 4 (24 hr at 140°C) attack was also absent, indicating that prior exposure at the high temperature tended to passivate the steel, so that subsequent exposure at lower temperatures produced less attack.

At 55°C (run 6) the specimens exposed in the spray underwent weight losses averaging 0.39 mg/cm^2 and those in the solution, 0.64 mg/cm^2. In both cases isolated shallow pits no more than 2 mils in depth were present on all specimens.

Specimens of types 304 and 316 stainless steel were exposed in all runs, and in no case was any attack observed. In addition during runs 11 and 12 welded specimens of type 304 stainless steel prepared by a qualified welder at the Commonwealth Edison Company's Zion Plant construction site were included in the test. These specimens were typical of the welds in the residual heat removal piping of that plant. Exposure in the two runs produced no evidence of any attack in the weld, in the heat-affected zone, or on the base plate.

Since it is well established that the austenitic stainless steels are susceptible to stress-corrosion cracking in the presence of chloride ions, it was of interest to determine the degree of such susceptibility in a chloride-contaminated spray solution. Accordingly, run 9 was made using the standard alkaline borate solution which contained 100 ppm Cl^-. This chloride ion concentration is ten times more than is expected in the course of a design basis accident. During this run, 52 U-bend-type specimens consisting of 40 type 304 and 12 type 316 stainless steel specimens were exposed. Half of the specimens of each alloy were sensitized [heated at 677°C (1250°F) for 1 hr and furnace cooled] and then pickled in an HNO_3-HF solution before being formed into U-bends. All of the sensitized specimens were given very heavy pickles, so that substantial intergranular attack was produced prior to exposure to the spray solution. It has been shown that sensitized and heavily pickled stainless steels are more susceptible to chloride-induced cracking than the corresponding annealed material,[1] and it was desired to have part of the specimens as susceptible as possible. All of the pickled 316 and half of the 304 stainless steel specimens had intergranular attack to a depth of about 5 mils; the other half of the sensitized 304 stainless steel specimens had intergranular penetrations as deep as 30 mils. All of the annealed materials were used as received from the mill, that is, hot rolled, annealed, and pickled (lightly). In addition, ten of the annealed type 304 stainless steel specimens were double U-bends formed by bending two strips at the same time. The purpose of the latter type specimen was to produce a crevice between the two highly stressed members.

At the completion of the test no cracks were visible under microscopic examination on any of the annealed specimens, including the double U-bend specimens. Metallographic sectioning and subsequent examination of several specimens also failed to locate a single crack.

Microscopic examination of the sensitized specimens indicated apparent cracks on several of the specimens in the region of maximum deformation. However, metallographic sections taken through the apparent cracks showed no evidence of cracking in the usual sense. Some grains were missing from the tensile surfaces, but this appeared to be due to corrosion of the grain-boundary material during the unusually heavy pickling and subsequent loosening of the grains during bending of the specimen. Grains were not missing from the inside surfaces of the U-bends (surfaces under compression), but the depth of intergranular attack was the same on both sides. Figures 7, 8, and 9 are photomicrographs of sections taken through regions of surfaces that appeared to contain cracks based on surface examination; typical stress corrosion cracks are not present, but the heavy intergranular attack is obvious. Thus no evidence of typical chloride-induced stress-corrosion cracking was found on any of the 52 specimens exposed in this test.

The test solution used in run 10 contained sodium thiosulfate, and the specimen array contained aluminum alloys as well as the other materials. As will be discussed later, aluminum reduced some of the thiosulfate to hydrogen sulfide, so that during this test the solution and the atmosphere contained an appreciable concentration of hydrogen sulfide. In this test three U-bend specimens of carbon steel and two of stainless steel were exposed both to the spray and in the solution. No evidence of stress-corrosion cracking or other localized attack was found on any of the specimens as a result of exposure in the presence of hydrogen sulfide.

Specimens of both carbon steels and both stainless steels were included in the sample array in run 13, in which the solution contained 10 ppm iodide. A few very shallow random pits well under a mil in maximum depth were noted on all specimens of all four alloys, and some small areas of light rust were found on the carbon steel specimens. However, the maximum weight loss observed on any specimen did not exceed 0.1 mg/cm^2, which is negligible.

[1] S. P. Rideout, DPSPU 62-30-26 (March 1963).

Fig. 7. Cross Section Through Tensile Surface of Sensitized and Pickled 316 Stainless Steel U-Bend Specimen Exposed for 24 hr at 140°C, 168 hr at 100°C, and 504 hr at 55°C to a 0.15 *m* NaOH–0.28 *m* H_3BO_3 Solution Containing 100 ppm Chloride.

Corrosion of Copper Alloys

Copper, 90-10 and 70-30 cupronickel, and Admiralty brass were exposed to both solutions in some of the runs. The behavior of the first three alloys in both test solutions at different temperatures is shown in Table 6. The weight losses were determined after removal of adherent corrosion products. Those specimens exposed in the alkaline borate solutions had only very thin films, whereas those tested in the thiosulfate solutions developed heavy black scales, the weight of which was roughly proportional to the amount of metal corroded.

The data in Table 6 clearly show that the thiosulfate-containing solution was much more corrosive to copper alloys than the alkaline borate solution; under the same conditions weight losses were about 100 times greater in the presence of thiosulfate. The addition of nickel to copper significantly increased the corrosion resistance of copper to both solutions. Thus in all cases copper was the least corrosion resistant and 70-30 cupronickel was the most resistant. In almost all cases, but particularly with copper, corrosion was much greater in the spray than in solution.

The density of the three alloys is practically the same (8.9 g/cm^3), and a weight loss of 10 mg/cm^2 in 168 hr (1 week) corresponds to a corrosion rate of 23 mils/year. It should be noted, however, that the corrosion rates were probably not constant, since in the thiosulfate solutions substantial films formed which probably retarded corrosion as the thickness increased.

Specimens of the above three alloys were coupled individually with both stainless and carbon steel during run 6. In all cases the weight losses of the coupled specimens exposed in the spray were within the spread of weight losses for the uncoupled specimens. Of the totally submerged coupled specimens only copper coupled either to stainless or carbon steel appeared to show an effect. The weight losses of the coupled copper specimens were about twice those for the uncoupled specimens. The cupronickel specimens, however, were

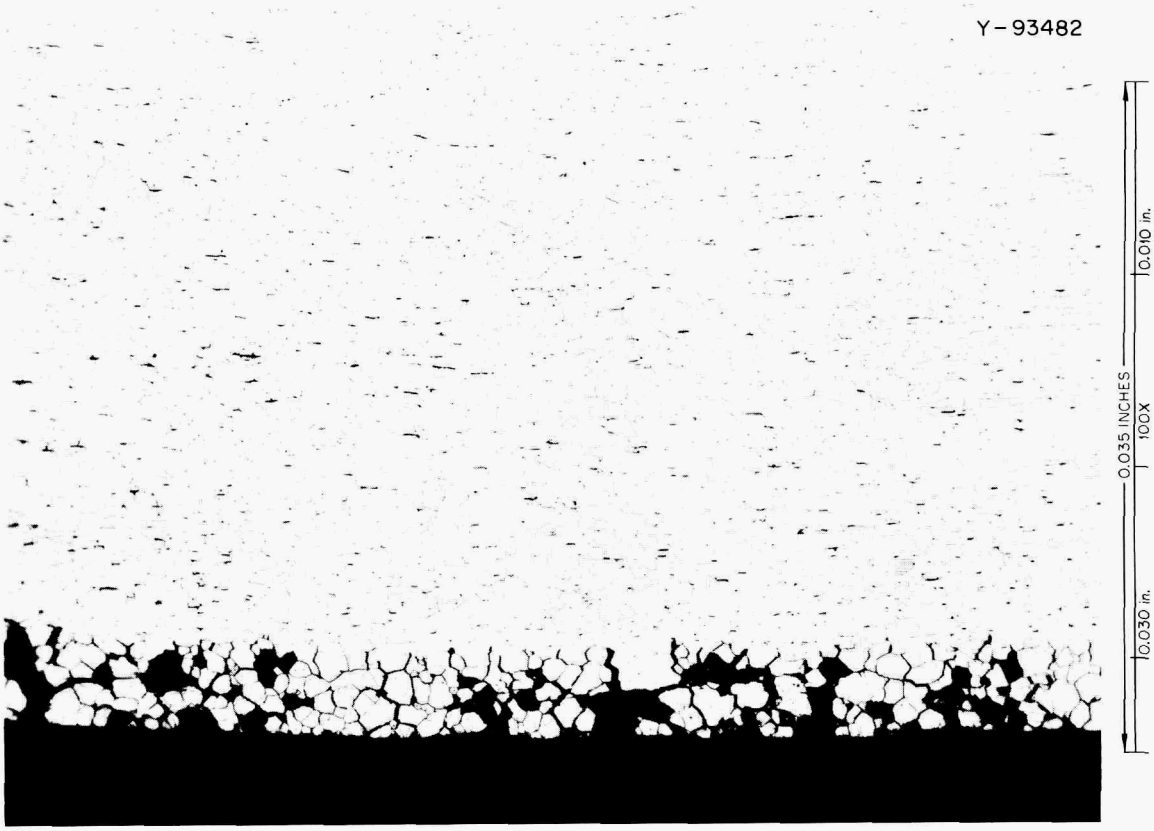

Fig. 8. Cross Section Through Tensile Surface of Sensitized and Pickled 316 Stainless Steel U-Bend Specimen Exposed for 24 hr at 140°C, 168 hr at 100°C, and 504 hr at 55°C to a 0.15 *m* NaOH–0.28 *m* H_3BO_3 Solution Containing 100 ppm Chloride.

unaffected by coupling with either steel. In all cases the stainless and carbon steel specimens were unaffected by coupling with copper or the cupronickels.

Specimens of the cupronickel alloys and copper were included in run 13 in which the solution contained 10 ppm iodide. The weight losses for the three alloys were about as expected based on the results shown in Table 6. There was no evidence of localized attack, and thus it can be concluded that the presence of iodide at the 10-ppm level had no effect on any of these alloys.

Admiralty brass specimens were exposed to both solutions only at 140°C (runs 3 and 4, Table 1). In the alkaline borate solution weight losses were 0.18 and 0.08 mg/cm^2 in the spray and solution, respectively; in the thiosulfate-containing solution the respective weight losses were 3.45 and 2.56 mg/cm^2. Based on only these two tests, the general corrosion resistance of Admiralty brass appears to be about the same as, or perhaps slightly better than, 90-10 cupronickel.

U-bend specimens of the three copper alloys and copper were exposed in runs 5 and 6. No evidence of cracking or stress-accelerated corrosion was found in either run.

Corrosion of Galvanized Steel and Zinc

Specimens of galvanized steel were exposed only in runs 9 and 14 and in part of run 6 (164 hr); pure zinc was included only in run 14.

Those specimens exposed for 164 hr at 55°C in an alkaline borate solution containing thiosulfate lost 2.8 mg/cm^2 in the spray and 0.84 mg/cm^2 submerged in the solution. In both cases some zinc was present on all specimens at the end of the test, but on other random areas zinc was completely missing. On the surfaces around the ungalvanized sheared edges the zinc was most heavily attacked.

Specimens exposed to the alkaline borate solution over the temperature range of 140 to 50°C (run 9)

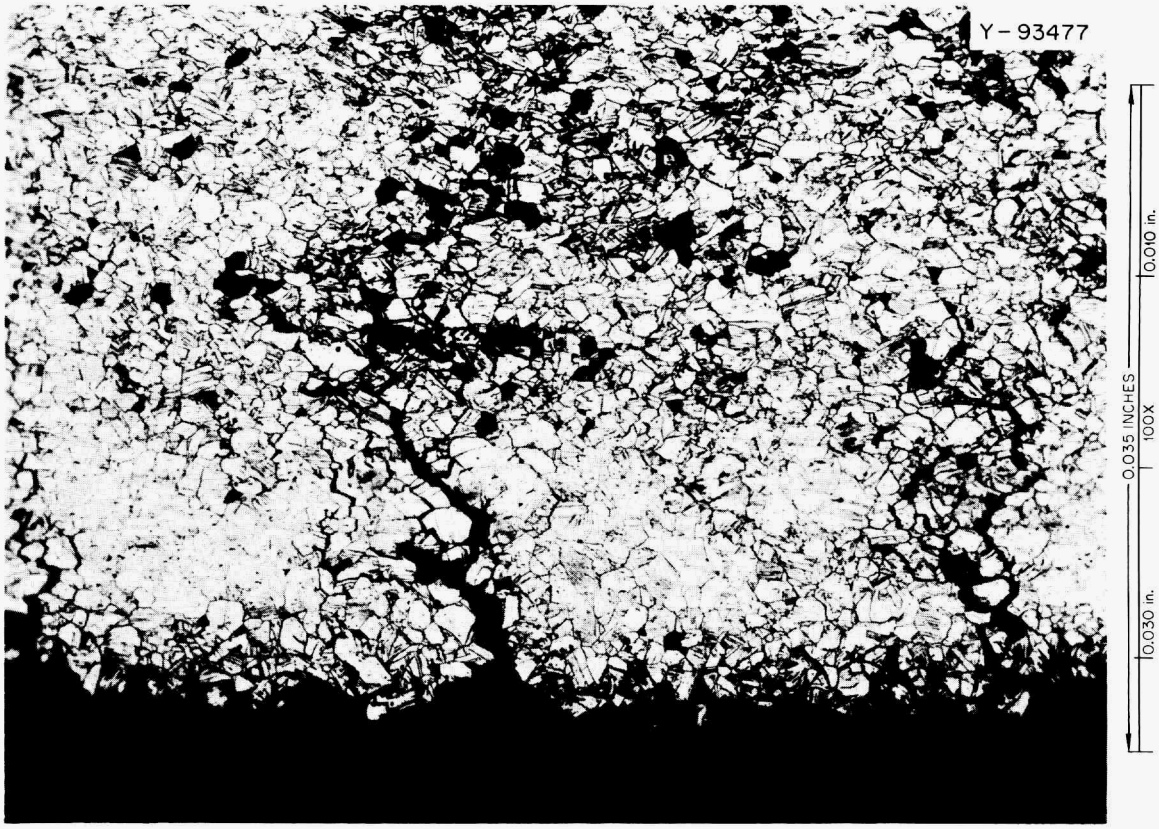

Fig. 9. Cross Section Through Tensile Surface of Sensitized and Very Heavily Pickled 304 Stainless Steel U-Bend Specimen Exposed for 24 hr at 140°C, 168 hr at 100°C, and 504 hr at 55°C to a 0.15 *m* NaOH–0.28 *m* H_3BO_3 Solution Containing 100 ppm Chloride.

developed many small blisters in the remaining zinc and showed slight weight gains. The material in the blisters was zinc oxide. As in the previous case zinc was largely missing from the surfaces adjacent to the sheared edges.

During run 14 in which the temperatures and duration of the test were generally lower than in run 9, the galvanized surfaces underwent relatively little attack. The surfaces had a thin, white, unidentified deposit, and numerous small blisters were also present. In contrast to run 14, no solid corrosion products appeared to be in the blisters. Weight losses of all specimens did not exceed 0.3 mg/cm^2. Similar specimens of pure zinc underwent only minor attack; the surfaces were covered with a thin, milky film, and weight losses in all cases were less than 0.4 mg/cm^2.

The above results indicate that in the event a spray system is used in a reactor containment building, corrosion damage to galvanized surfaces should not be serious. Furthermore, even if all the zinc corroded, the integrity of the system would not be violated, because corrosion of ungalvanized steel is essentially nil in the alkaline spray solutions.

Corrosion of Other Materials

Specimens of Inconel 600 and 718, Monel 400, and Zircaloy 2 were exposed in most of the runs shown in Table 1. Under all conditions these four alloys were essentially unaffected; occasionally very small weight changes were noted, but these were usually within the weighing precision.

During runs 11 and 12, specimens of 35 different protective coatings supplied by 11 companies were exposed. A wide variety of behavior was observed for the different coatings, but there appeared to be little difference in behavior between the solution with thiosulfate and that without thiosulfate. The complete results of these two tests will be the subject of a separate report in this series.

Table 6. The Corrosion of Copper Alloys in Simulated Spray Solutions

Test Conditions	Weight Loss (mg/cm^2)					
	Copper		90-10 Cupronickel		70-30 Cupronickel	
	Spray	Solution	Spray	Solution	Spray	Solution
AB,[a] 55°C, 518 hr	1.35	0.28	0.09	0.05	0.03	0.03
	0.99	0.42	0.07	0.03	0.03	0.01
	0.67		0.06		0.02	
	1.11		0.09		0.03	
ABT,[b] 55°C, 518 hr	121	5.3	0.45	0.23	0.24	0.10
	121	6.6	0.37	0.29	0.11	0.10
	114		0.32		0.10	
	107		0.40		0.12	
AB, 100°C, 168 hr	0.85	0.28	0.01	0.02	<0.01	<0.01
	1.30	0.16	0.04	0.08	<0.01	<0.01
	0.43		0.02		<0.01	
	0.36		0.01		<0.01	
ABT, 100°C, 168 hr	102	4.14	0.70	0.43	0.09	0.51
	89	5.32	0.77	0.51	0.09	0.18
	96		0.70		0.09	
	78		1.40		0.07	
AB, 140°, 24 hr	0.90	0.15	0.21	0.33	0.06	<0.01
	0.42	0.08	0.07	0.26	0.02	<0.01
	0.21		0.05		0.01	
	0.26		<0.01		0.01	
ABT, 140°C, 24 hr	111	1.10	11.6	0.89	0.13	0.15
	56	1.11	11.6	0.95	0.07	0.16
	69		12.0		0.08	
	69		12.8		0.07	

[a]AB = 0.15 *m* NaOH–0.28 *m* H_3BO_3 solution.
[b]ABT = 0.15 *m* NaOH–0.28 *m* H_3BO_3–0.064 *m* $Na_2S_2O_3$ solution.

Concrete in Spray Solutions

To determine the effect of the alkaline spray solutions on concrete, eight concrete cylinders obtained from the Browns Ferry Nuclear Plant were immersed in the spray solutions. These specimens were 6 in. in diameter by 12 in. long and were poured from actual construction mixes at the reactor site. Additional cylinders were also poured at the same time to provide an adequate number of control cylinders.

Four of the cylinders were exposed to a solution containing 0.15 *m* NaOH and 0.28 *m* H_3BO_3, and four were exposed to the same solution containing in addition 0.064 *m* $Na_2S_2O_3$. In both cases the specimens were exposed continuously for 1 day at 140°C, 7 days at 100°C, and 21 days at 50°C. As a result of the exposures none of the specimens underwent any visible changes, and the cylinders were returned to Browns Ferry Plant for strength determinations.

The crush strengths of control cylinders were determined 7 and 28 days after preparation, and a third control was crushed at the same time as the specimens exposed to the spray solutions. The strengths reported for all the cylinders are shown in Table 7.

The number of the mix refers to the design strength of the concrete and the maximum size of the aggregate. Thus 301.5 refers to a design strength of 3000 psi and a maximum aggregate size of 1.5 in. Similarly, 430.75 indicates a design strength of 4300 psi and a maximum aggregate size of 0.75 in. The results show that with the three sets of specimens from the No. 301.5 mixes only small or no losses in strength were observed. In fact, losses as large as those observed would have been expected just from the thermal cycle to which the concrete was exposed. The large loss of strength in the case of mix No. 430.75 is unexplained. However, in all cases the strength of the concrete after exposure exceeded the design strength, indicating that in the event of a design basis accident the concrete in the containment building should retain its structural integrity.

Table 7. Strength Data on Concrete Cylinders Prepared at Browns Ferry Nuclear Plant

Date of Mix – Nov. 7, 1968 Mix No. 301.5		Date of Mix – Nov. 5, 1968 Mix No. 301.5	
Date of Test	Strength (psi)	Date of Test	Strength (psi)
Nov 14	2230	Nov 12	2140
Dec 15	4190	Dec 3	3810
Mar 26	6630	Mar 26	6295
Mar 26	6685[a]	Mar 26	5660[a]
Mar 26	7020[a]	Mar 26	5730[a]
Date of Mix – Nov. 6, 1968 Mix No. 301.5		**Date of Mix – Nov. 4, 1968 Mix No. 430.75**	
Date of Test	Strength (psi)	Date of Test	Strength (psi)
Nov 13	2070	Nov 11	3400
Dec 4	3930	Dec 2	5410
Mar 26	6370	Mar 26	8170
Mar 26	5570[b]	Mar 26	4970[b]
Mar 26	5750[b]	Mar 26	4940[b]

[a]Exposed to 0.15 *m* NaOH–0.28 *m* H_3BO_3–0.064 *m* $Na_2S_2O_3$.

[b]Exposed to 0.15 *m* NaOH–0.28 *m* H_3BO_3.

At the end of the tests the solutions contained about 10 ppm chloride (leached from the concrete) and about 10% less boron than at the start. In these tests the ratio of the volume of solution to the plane surface area of the concrete was very low, 2.5 ml/cm^2 (0.6 gal/ft^2). Because of this fact the loss of boron and the buildup of chloride ions were probably substantially greater than would be expected in an actual reactor system subjected to a spray solution.

Solution Stability

The composition of the alkaline borate solution remained constant in all tests regardless of the total amount of metal that corroded (mostly aluminum) except that, as mentioned previously, some boron was lost when the concrete cylinders were in the system. The pH of the solution also remained constant at 9.3 to 9.4.

The thiosulfate containing solutions were not completely thermally stable, and some decreases in thiosulfate concentration were always noted. For example, during a 96-hr test at 100°C 16% of the thiosulfate was lost; essentially the same amount was lost during a 24-hr test at 140°C. These decomposition rates are very similar to those obtained by Zittel[2] in all-glass equipment and indicate that the presence of metal surfaces does not accelerate the decomposition reaction.

With aluminum in the system some of the thiosulfate was reduced to hydrogen sulfide. The rate of reduction is a function of the temperature and the aluminum surface area available for reaction. However, even in those cases where half of the thiosulfate had been reduced the pH of the solution remained at 9.3 ± 0.1.

As noted in Table 6, copper corroded at an appreciable rate in the presence of thiosulfate, and a gelatinous blue-green precipitate was always present in the solution. In one test where aluminum and copper specimens were exposed to a thiosulfate-containing solution at the same time, the copper corrosion product suspended in solution was no longer gelatinous but consisted of hard granular particles which plugged the small (0.093 in.) spray nozzle orifice in the system. Apparently the normally gelatinous corrosion product of unknown composition reacted with the hydrogen sulfide produced by the corrosion of aluminum to form copper sulfide. Even in this case, however, the pH remained constant and no boron was lost from solution.

DISCUSSION

With the exception of the aluminum alloys in both solutions and copper and the copper alloys in the thiosulfate-containing solution, corrosion damage to materials in the reactor system or in the containment building should be insignificant in the event a spray system is used. While it is true that galvanized surfaces corrode in both alkaline spray solutions, the corrosion rate of the zinc is much less than for most aluminum alloys. Because the corrosion rate of the substrate steel is very low, even complete dissolution of the zinc from the galvanized surface would not alter the integrity of the containment system.

The corrosion rate of copper and the copper alloys is low enough in the alkaline borate solution to be of no practical concern. However, with thiosulfate in the solution the corrosion rate of pure copper is appreciable, particularly in the spray region. Since the use of pure copper in a reactor system is limited to electrical conductors, it should be possible to enclose those that must be used in the event of a design basis accident in

[2]H. E. Zittel, pp. 77–81 in *ORNL Nuclear Safety Research and Development Program Bimonthly Report for March–April 1968,* ORNL-TM-2230.

sealed metallic conduit. Perhaps certain waterproof electrical insulating materials would be sufficiently resistant to the spray solutions so that conduit would not be necessary. However, in this program the resistance of such materials to spray solutions was not investigated. Although measurable corrosion rates were observed with the cupronickel alloys and Admiralty brass, the corrosion rates were low enough that corrosion damage should be small even in the thiosulfate solutions.

As indicated by the data presented in Tables 3, 4, and 5, most aluminum alloys corrode at very high rates. Of the alloys investigated only 5052 aluminum seemed to form a protective coating at 100 and 140°C, and once formed this coating seemed to greatly minimize subsequent corrosion. However, when exposed directly at 55°C, the alloy did not form a film and corroded at a constant rate comparable with the other alloys. Why the 5052 alloy behaves differently from the other alloys is not known, but it is clear that if aluminum is to be used in a containment system the 5052 alloy is a better choice from the corrosion standpoint than any other alloy tested.

The corrosion of aluminum can be considered as the sum of the anodic and cathodic half reactions. In its simplest form the anodic reaction can be represented by

$$Al \rightarrow Al^{3+} + 3e^- .$$

Either or both of the following cathodic processes can occur:

$$O_2 + 2H^+ + 4e^- \rightarrow 2OH^- ,$$

$$2H_2O + 2e^- \rightarrow H_2 + 2OH^- .$$

The first cathodic reaction probably accounts for the fact that at low temperature aluminum corrodes faster in the spray than when totally submerged. With the continuously moving thin film of solution on the specimens in the spray, oxygen is more readily available to aluminum surfaces than it is to comparable surfaces totally submerged in the semistatic solution. On the other hand, at higher temperatures there is more of a tendency to form corrosion product films in the spray than in solution, so rates are lower. If this speculation is correct, then the added corrosion observed in the spray at low temperatures does not produce more hydrogen but only consumes oxygen.

Excluding the results obtained with the 5052 alloy, the data in Tables 3, 4, and 5 show that the corrosion rate is greater the higher the temperature and that the corrosion rate was about the same either with or without thiosulfate present. To correlate the effect of temperature, an Arrhenius-type plot of the data, Fig. 10, was constructed using only the data obtained from specimens totally submerged in solution. The two points at 1/°K equal to 0.00305 (55°C) were the highest (1100 Al) and lowest (5454 Al) rates shown in Fig. 4. At 100°C (1/°K = 0.00268) the points represent the average corrosion rates for the alloys with the highest and lowest rates both with and without thiosulfate present (calculated from the data in Tables 4 and 3 respectively). At 140°C (1/°K = 0.00242) the average corrosion rate obtained with 6061 aluminum is plotted. The line as drawn corresponds to an activation energy of 19 kcal/mole.

Although the data for the corrosion of aluminum are quite scattered, the treatment of the data in the above manner gives at least a rational basis for estimating the corrosion rate of aluminum alloys (except 5052 Al) and the related hydrogen generation rate. A corrosion rate of 1000 mils/year corresponds to a hydrogen generation rate of 0.9 standard liter/hr per square foot of aluminum surface area, assuming that the cathodic process involves only the reduction of water. From a knowledge of the surface area of aluminum and the volume of the containment building it should be possible to calculate the approximate concentration of hydrogen originating from corrosion of aluminum surfaces. Then considering the hydrogen production rates from both corrosion and radiolytic decomposition of the spray solution, it should be possible to determine when or whether the gas in the containment vessel will reach an explosive composition.

SUMMARY

The results obtained from the exposure of various materials in a recirculating spray loop either to a 0.15 *m* NaOH–0.28 *m* H_3BO_3 solution or to the same solution containing in addition 0.064 *m* $Na_2S_2O_3$ can be summarized as follows:

1. Aluminum alloys were subject to severe corrosion in either solution. The one exception was the 5052 alloy, which at 100 to 140°C formed a protective coating of corrosion products and nearly stopped corroding. At 55°C such a coating did not form, and the 5052 alloy corroded at about the same rate as the other alloys. However, a protective coating formed at the high temperature remained intact for at least two weeks when exposed at 55°C.

2. The approximate temperature dependence of the corrosion rate of aluminum alloys was established; and

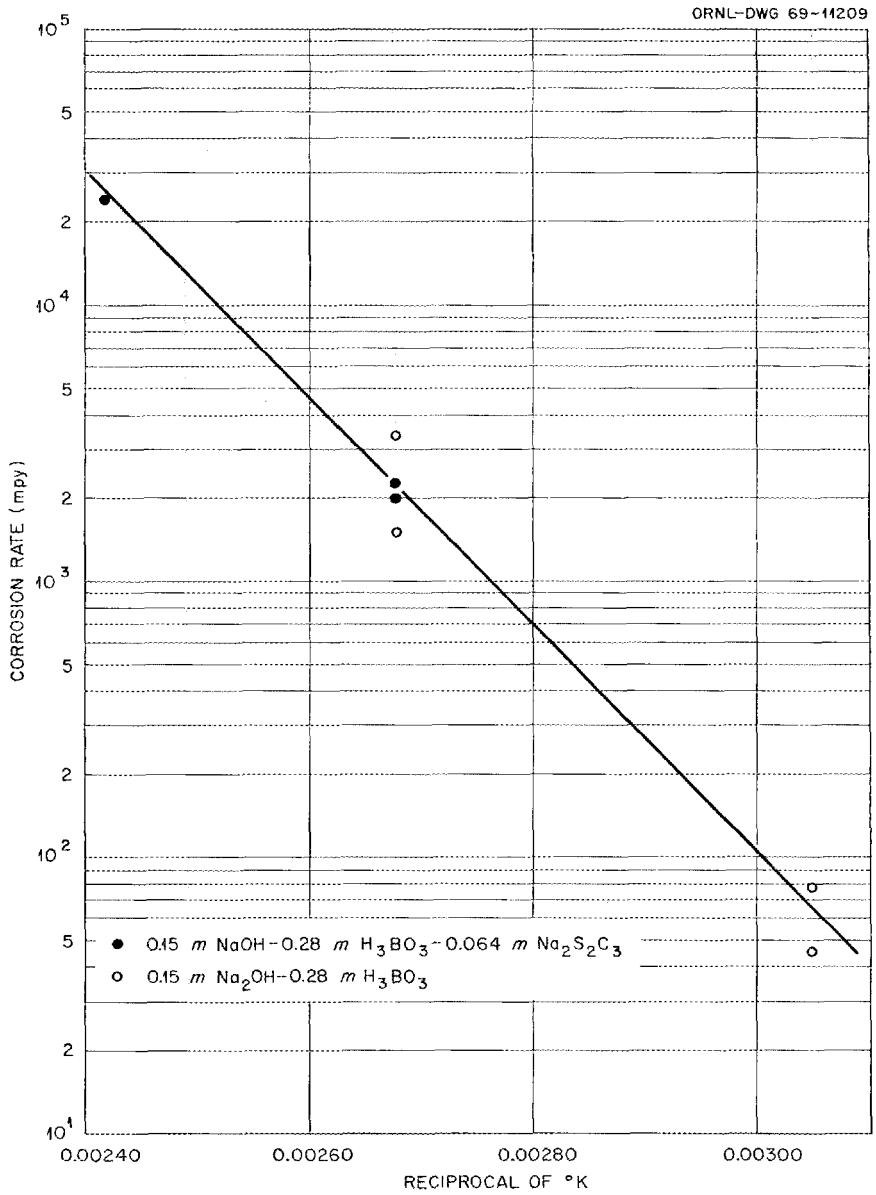

Fig. 10. The Temperature Dependence of the Corrosion Rate of Aluminum Alloys.

assuming that aluminum corrodes exclusively by reduction of water, this relationship can be used as a basis for estimating the buildup of hydrogen in the containment building as a result of corrosion of aluminum.

3. Pure copper corroded at an appreciable rate in the thiosulfate-containing solution, indicating that electrical conductors that must be used in the event of a design basis accident should be enclosed in conduit or protected with waterproof electrical insulators that resist attack by spray solutions. The alloying of nickel with copper significantly increased the resistance of copper to attack by the solutions with thiosulfate. Thus attack on cupronickel alloys was relatively low, and their use in reactor systems should present no major problems. In the alkaline borate solution without thiosulfate, copper and its alloys corroded at low rates.

4. The carbon steels and stainless steels were generally resistant to both solutions under all conditions,

although light pitting was occasionally noted on the carbon steels in the thiosulfate-containing solution. The stainless steel specimens were essentially inert in either solution even when the stainless steel was in a highly stressed condition and the solution contained 100 ppm chloride.

5. Galvanized surfaces were subject to attack in either solution. No quantitative measure of corrosion rate was made, but the rate of attack was much lower than for aluminum alloys. Since the steel substrate is resistant to attack, total removal of zinc from the surface would not compromise the integrity of the system.

6. Inconel 600 and 718, Zircaloy 2, and Monel 400 were almost completely inert in both spray solutions.

7. Concrete test cylinders exposed to both solutions for 29 days over the temperature range of 140 to 50°C remained unchanged in appearance and, except for one mix, suffered little or no loss of crush strength. Even in the latter case the strength after exposure exceeded the design strength, indicating that exposure of structural concrete to either solution would not endanger its load-carrying capacity.

8. The alkaline borate solution remained stable under all conditions except that a small amount of the boron was absorbed by the concrete specimens. Thiosulfate solutions were not completely thermally stable, although the rate of decomposition was such that sufficient thiosulfate would be present to react with all of the iodine in a system even after prolonged circulation of the solution. Aluminum in the system reduced some thiosulfate to hydrogen sulfide, which appeared to have little effect on the corrosion behavior of the materials tested. However, normally gelatinous copper corrosion products were converted to hard granular sulfides with hydrogen sulfide in the system. In all cases the pH of both solutions remained at 9.3 ± 0.1.

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