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Corrosion of Materials in Absorption Heating and Refrigeration Fluids

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CORROSION OF MATERIALS IN ABSORPTION HEATING AND REFRIGERATION FLUIDS

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ABSTRACT

The corrosion of metals and alloys in absorption refrigeration fluids has received little attention except for the behavior of steel and copper alloys in aqueous lithium bromide solutions. This report presents results of short-term corrosion tests on several materials in lithium bromide solutions as well as in other fluids of potential interest in advanced absorption machines. All materials tested had extremely high resistance to organic fluids, but in the aqueous systems some of the materials underwent localized attack. Type 304 stainless steel had acceptable corrosion resistance in most environments, but it underwent stress corrosion cracking in oxygen- or chromate-containing lithium bromide and even in the unstressed condition was subject to intergranular attack in hot concentrated caustic solutions.

INTRODUCTION

Absorption refrigeration and heating systems in use today are based on the lithium bromide—water and the water—ammonia cycles.[1] Advanced absorption technology is being developed in many countries, with the efforts primarily focused on new cycles[2] and new fluids[3] in the hope of improving the efficiency of such machines. The Energy Division of Oak Ridge National Laboratory (ORNL) is one of the organizations conducting research in advanced absorption cycles and has plans for a test facility where a wide range of new fluids and new heat exchangers can be tested. In addition, theoretical solutions to the transport equations will be investigated.

Corrosion data are almost nonexistent for materials in the fluids considered in the advanced absorption machines. Even with the commonly used lithium bromide—water system, published data seem to be limited to the behavior of steel and copper alloys that are used in commercial machines. The present investigation is an effort to partially fill that

void and to help select a single material for the ORNL test facility that can handle as many different fluids as possible without experiencing excessive corrosion. All cycles operate over a range of temperatures, but our tests were conducted only at the highest practical temperature for the particular system. Furthermore, most of our tests lasted only one week (168 h); lower average corrosion rates may have been reported in some cases had the tests lasted longer, because corrosion rates are generally higher initially and decrease as a corrosion-product film forms. Thus, the combination of high temperature and short test times probably produced higher average corrosion rates than would be experienced in an actual machine, excluding velocity and thermal cycling effects, if any. For our application, a corrosion rate as high as 125 μm/y (5 mils/y) was deemed satisfactory, although in actual advanced machines corrosion rates no greater than 50 μm/y (2 mils/y) probably would be sought, depending on expected life, warranty requirements, and type of refrigerant-absorbent combination.

EXPERIMENTAL CONDITIONS

Most of the tests were conducted in stainless steel autoclaves that had Teflon liners. The internal volume of the autoclaves was 235 mL, and the volume of test fluid was 150 to 170 mL in each test. A stainless steel or nickel rod attached to the head of the autoclave had cross wires attached for hanging specimens. Glass or Teflon was used to electrically insulate the specimens from the holder. Specimens of several different alloys were exposed in the same autoclave, and in some tests stressed specimens were also included. The test specimens were thin strips measuring about 13 by 25 mm (~6.5 cm^2) with a hole drilled near one end. All materials were used "as received" (rolled and annealed). The specimens were washed in a detergent, rinsed in deionized water and acetone, dried, and weighed. After the test the specimens were washed in deionized water and acetone, dried, and reweighed. In most cases the amount of corrosion products on the specimen was negligibly small. Corrosion rates were calculated from weight losses assuming uniform attack.

Stressed specimens, in the configuration of C-rings or U-bends, were included in some tests.[4] The C-rings were made from 1-cm lengths of 13-mm-diam tubing. Holes were drilled 180° apart, and a section of the tube wall 90° from the holes was removed. A small stainless steel bolt and nut, insulated with Teflon, were used to compress the tubing and place the outer surface across the opening in tension. The U-bends were made from thin strips measuring about 10 by 50 mm bent over a 9.4-mm-diam bar so that the legs were parallel. Wire or bolts of the same composition prevented the legs from springing apart. With both types of specimens, the material was plastically deformed in the region of maximum stress. The stressed specimens were not weighed.

A valve was welded to the head of each autoclave so that gas could be added to or removed from the autoclave. In most tests the closed autoclave with the test solution and specimens in it was placed in liquid nitrogen; after freezing, the air was pumped out. The enclosed solution was then thawed and refrozen, and the gas was again pumped from the autoclave. The autoclaves were heated for the desired time in electric furnaces.

A few tests were conducted in heavy-walled Pyrex glass tubes. In those tests a single unstressed strip specimen and the solution were placed in a tube closed at one end. The solution was frozen and the open end was fused shut under vacuum. The specimens, somewhat smaller than those used in the autoclave tests, had surface areas of 3 to 5 cm^2. The volume of solution was 12 to 20 mL.

The solutions were made from reagent-grade chemicals and distilled water. Most of the solutions were made by weight or volumetric measurements of liquids and solids, but, for Refrigerant 22, a known volume of gas at atmospheric pressure and room temperature was transferred to the test vessel by cooling the vessel in liquid nitrogen. Concentrations are reported as weight percent.

RESULTS

PRELIMINARY TESTS

The first two tests, made to check the equipment, were conducted with aqueous solutions of 5% ammonia at 180°C and with 70% lithium bromide at 150°C. Both tests lasted for one week, and air occupied the free space in the autoclaves. In the ammonia solution, pure nickel, Monel 400, and 304 stainless steel corroded at average rates of 39, 92, and <2 μm/y, respectively. In the lithium bromide solution, nickel, Monel 400, 70-30 and 90-10 cupronickels, and types 304 and 430 stainless steel corroded at average rates of 0, 4.7, 70, 300, 2.6, and 5.3 μm/y, respectively. Microscopic examination failed to show any evidence of localized attack on the specimens in either solution. The corrosion rates observed in these tests may be higher than would have been observed in the absence of air (oxygen), but the tests demonstrated that the test equipment performed as expected.

LITHIUM BROMIDE—AMMONIA—WATER SYSTEM

A series of week-long tests was conducted in a solution containing 54% LiBr, 10% NH_3, and 36% H_2O at 200°C. The specimens tested and the corrosion rates observed are shown in Table 1.

In Test 1 with air in the autoclave all rates were reasonably low except those for carbon steel. The values for carbon steel and stainless steel shown in the table are somewhat low because both had black films that were not removed before weighing. All other specimens had very thin tarnish-like films that were removed by wiping with tissue. There was no evidence of localized attack on any specimen. After the test the solution contained a finely divided black precipitate that slowly settled from solution. The precipitate was magnetic and probably was magnetite.

Test 2 was intended to duplicate the previous experiment except for the absence of air. However, at the start of the test, the temperature controller malfunctioned and the temperature was above 265°C for 4 to 8 h. For the rest of the test the temperature was maintained at 200 ± 2°C. All specimens except the 304 stainless steel (SS) specimen had a bluish cast,

which was removed on washing. The stainless steel specimen had a shiny black film, and the solution contained a small amount of a black precipitate. There was no evidence of localized attack on any specimen.

Table 1. Corrosion rates of various materials in a solution containing 54% LiBr, 10% NH_3, and 36% H_2O at 200°C

Material	Corrosion rate (μm/y)			
	Test 1[a]	Test 2[b]	Test 3	Test 4[c]
Nickel	100	350	150	280
Monel 400		280	390	
70-30 cupronickel	32	140	46	8.2
90-10 cupronickel	30	120	64	68
304 stainless steel	50	41	430	3.9
Carbon steel	320			
304 stainless steel[d]			0	

[a]Air filled the free space of the autoclave; air was removed in all other tests.

[b]During the first few hours, the temperature was >265°C.

[c]Solution contained 500 ppm $CrO_4^{=}$ added as K_2CrO_4 at the start of the test.

[d]Sample preexposed during Test 2.

Because of the temperature excursion in the previous test, the test was repeated with the temperature controlled at 200°C. In addition to the usual array of new specimens, the 304 SS specimen that had the black coating from the previous run was reexposed. The results are shown as Test 3 in Table 1. Nickel and the 90-10 and 70-30 cupronickels had etched appearances, and the 70-30 cupronickel had many shallow pits (all less than 25 μm deep). Monel 400 also had an etched appearance, but pitting was totally absent. The new 304 SS specimen had developed a thin tenacious film on most of its surface, but there were regions where the film was not complete. Apparently the absence of a film allowed more corrosion than was observed in the previous run. On the other hand, the 304 SS specimen that had the black film from the previous run remained unchanged. As in the previous test, the solution contained a finely divided black suspension at the end of the test.

Test 4 was conducted under the same conditions as Test 3 except that the $LiBr-NH_3-H_2O$ solution contained 500 ppm $CrO_4^{=}$ (added as K_2CrO_4). In addition to the specimens shown in Table 1, C-rings of 304 SS and carbon steel were exposed in the solution and in the vapor above the solution. At the end of Test 4 the solution was colorless, indicating complete reduction and precipitation of the $CrO_4^{=}$ ions. A loosely adherent red precipitate coated all specimens as well as the bottom of the Teflon liner. Nickel and 90-10 and 70-30 cupronickel specimens had etched appearances, and a few random shallow pits were noted on the 70-30 cupronickel specimen. The 304 SS specimen and C-ring exposed in the solution had dark adherent films, and the specimen lost very little weight. The 304 SS C-ring exposed in the vapor developed only a tarnish-like film; neither stainless steel C-ring showed any evidence of stress corrosion cracks. Both carbon steel C-rings developed relatively heavy scales, and the one exposed in solution developed a single crack (in the most highly stressed region) that extended nearly across the width of the specimen. Figure 1 shows the entire crack; Fig. 2 shows the tip of the crack at higher magnification and illustrates the intercrystalline nature of the crack.

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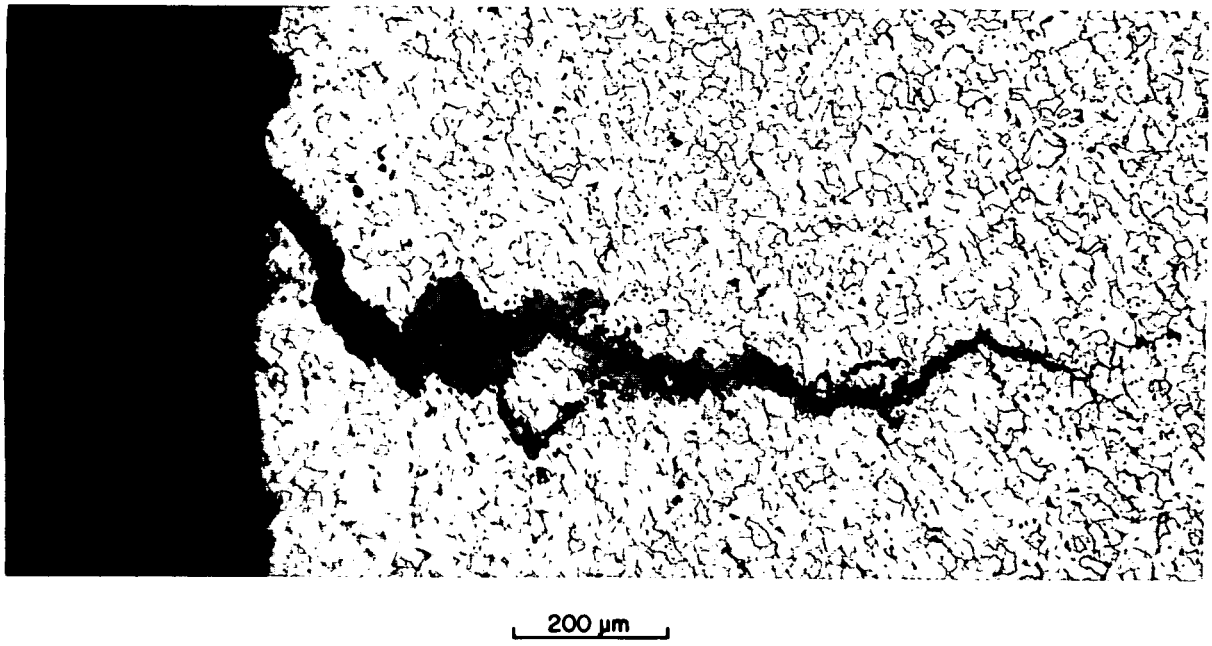

Fig. 1. Cross section of a crack that developed in the stressed region of a carbon steel C-ring on exposure to an aqueous solution containing 54% LiBr, 10% NH_3, and 500 ppm $CrO_4^{=}$ at 200°C.

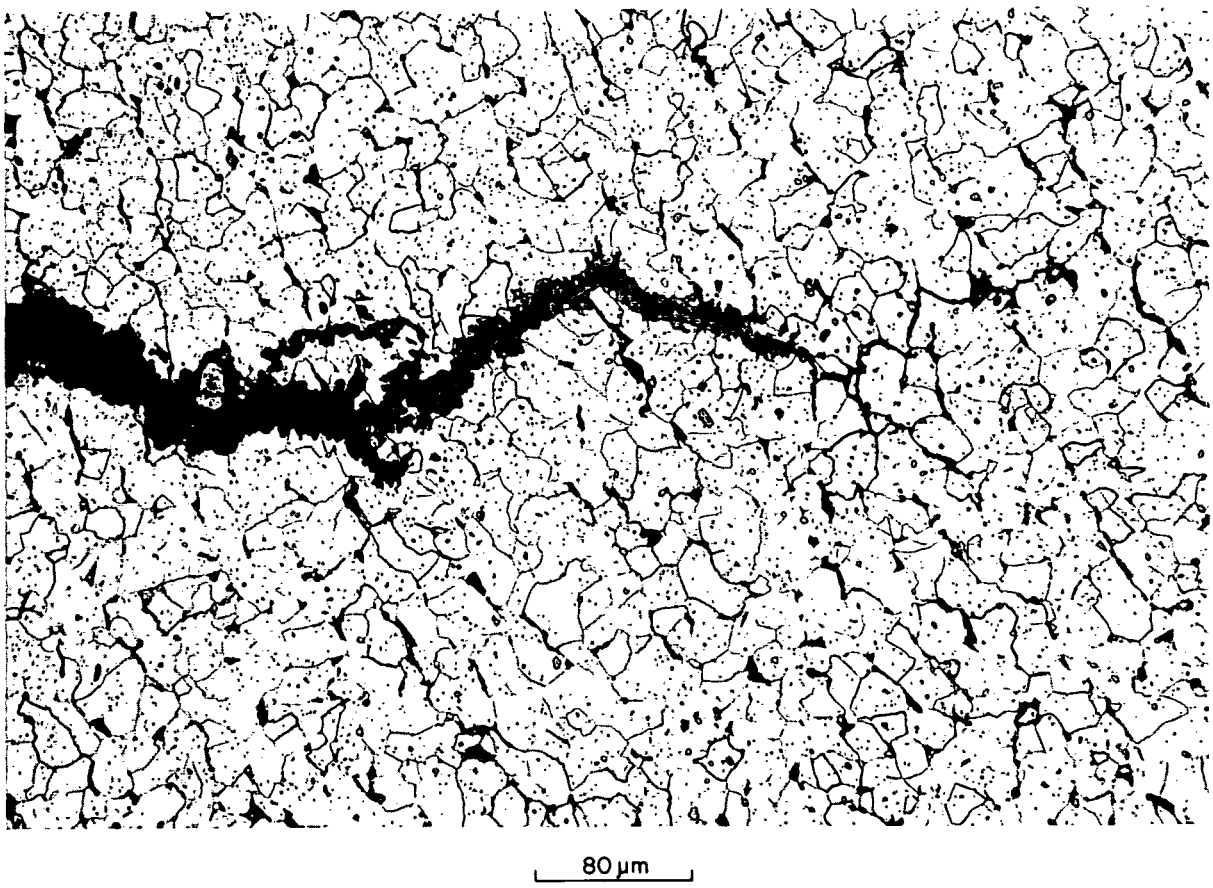

Fig. 2. Enlarged view of the tip of the crack shown in Fig. 1.

The previous data indicated that in the $LiBr-NH_3-H_2O$ solution stainless steel corroded for some period, during which a protective film formed, and then practically ceased corroding. Furthermore, the data for ferrous alloys suggested that the initial extent of attack was less the greater the total surface area exposed. Therefore, we conducted tests in which only stainless steel specimens were exposed to a deaerated solution of 54% LiBr, 10% NH_3, and 36% H_2O. The materials tested were types 304, 304L, and 316L SS. Some specimens of the latter two alloys had a weld bead across the specimen. The weld bead on the 304L SS specimen was 308L SS, whereas the weld bead on the 316L SS specimen was 316L SS.

The weight losses observed for the specimens during the first week of test are shown in Table 2. All specimens had developed glossy black films, and the solution contained a relatively large amount of a black magnetic solid. These same specimens were placed in a fresh oxygen-free solution of the same composition and heated for an additional week at 200°C. After the second week there was no change in specimen appearance, and weight changes were insignificant (Table 2). The solution was free of solid particles. The same specimens were returned to the same solution, air was removed, and the autoclave was heated at 200°C for an additional 16 days. At the end of this time, the specimens and the solution remained unchanged, and, as shown in Table 2, the weights of the specimens were nearly the same as after the first week. The results from this series of exposures clearly show that some stainless steel must corrode to form a protective coating, after which the attack rate is very low.

Table 2. Corrosion of stainless steel specimens in a deaerated solution containing 54% LiBr, 10% NH_3, and 36% H_2O at 200°C

Type of stainless steel[a]	Cumulative weight loss (mg/cm^2)		
	7 days	14 days	30 days
304	0.93	0.94	0.91
304L	0.45	0.55	0.49
304LW	0.71	0.65	0.75
316L	1.71	1.70	1.67
316LW	1.43	1.42	1.43
316LW	0.85	0.90	0.87

[a]W indicates that the specimen had a weld bead across it.

LITHIUM BROMIDE—METHYLAMINE—WATER SYSTEM

A single week-long test was conducted in a deaerated solution containing 36% LiBr, 10% CH_3NH_2 (methylamine), and 54% H_2O at 160°C. The specimens exposed and the calculated corrosion rates are shown in Table 3. Only the 316L and 304L SS specimens developed dark films around the edges; all others remained unchanged in appearance. The solution also remained unchanged, but a distinct odor of ammonia (as well as of methylamine) indicated some hydrolysis of methylamine.

Table 3. Average corrosion rates of materials exposed to a solution containing 36% LiBr, 10% CH_3NH_2, and 54% H_2O at 160°C for one week

Material	Corrosion rate (μm/y)
Nickel	15
Monel 400	4.6
70-30 cupronickel	5.2
90-10 cupronickel	10
304L stainless steel	5.9
316L stainless steel	<2

SODIUM HYDROXIDE—WATER SYSTEM

A single test was conducted in a 55% NaOH solution at 150°C for one week in the presence of air. The materials tested were nickel, Monel 400, 90-10 and 70-30 cupronickel, and type 304 SS. Except for the stainless steel all materials retained their original appearance, and weight changes per specimen (surface area 5—7 cm^2) were only ±0.3 mg, well within our weighing error. The stainless steel specimen had a film on part of its surface and had undergone substantial intergranular attack. Some grain dropping had occurred, and scraping the surface removed many additional grains. Because stainless steel (and mild steel also) is not a suitable material for use in hot concentrated caustic solutions,[5,6] no attempt was made to determine the depth of the intergranular penetration.

A single test was run in deaerated 75% NaOH solution at 175°C for one week. Specimens of nickel, Monel 400, and 90-10 and 70-30 cupronickel showed total weight changes (surface area 5—7 cm^2) ranging from -0.2 mg to +0.5 mg per specimen. There were no changes in the appearance of the specimens.

ORGANIC FLUIDS

Several week-long corrosion tests were conducted in organic fluids. Table 4 shows the mixtures of organic fluids used and the temperature of the tests. Some were conducted in autoclaves and others in Pyrex tubes. Single specimens of types 430 and 304 SS, Monel 400, nickel, 90-10 and 70-30 cupronickel, and 1100 aluminum were exposed in each environment.

Table 4. Composition and temperature of organic solutions used in corrosion tests

Composition of test medium	Temperature (°C)
95% tetraglyme,[a] 4% TFE[b]	110
95% tetraglyme, 5% TFE	130
90% tetraglyme, 10% Refrigerant 22[c]	130
93% NMP,[d] 7% TFE	110

[a]Tetraethylene glycol dimethyl ether.

[b]Trifluoroethanol.

[c]Chlorodifluoromethane.

[d]1-methyl-2-pyrrolidinone.

None of the specimens showed any visible change as a result of the exposure. The largest weight loss on any specimen (surface area 3–5 cm^2) was 0.5 mg, which was practically within our weighing error. Thus, all of the materials were essentially inert in all of the organic fluids under our conditions of test. In agreement with our results, Eiseman[7] also noted extremely low corrosion rates for copper and aluminum in tetraglyme–Refrigerant 22 at temperatures varying from 121 to 177°C (250–350°F). He also observed that Refrigerant 22 was very stable under these conditions. Comparable data were not found for the other two systems.

The tetraglyme-TFE solutions turned light brown during the tests, both at 110°C and at 130°C. The NMP-TFE solution also darkened slightly and acquired an amine-like odor. Tests with the former solutions in Pyrex tubes showed that the change in color occurred within one day at 130°C, and no further change occurred on continued exposure. Three sealed Pyrex tubes containing the latter solution were heated in an oven for times ranging from 7 to 23 days at 130°C. After one day the color was the same as after 23 days, and infrared spectra were the same regardless of exposure time. Thus, our data indicate that neither solution decomposed significantly up to 130°C. The color changes were probably due to trace impurities in the reagents. The tetraglyme–Refrigerant 22 solution showed no changes in appearance after one week at 130°C.

STRESS CORROSION CRACKING TESTS IN LiBr-H_2O SOLUTION

Because the austenitic stainless steels are highly susceptible to stress corrosion cracking in chloride solutions, it was important to determine whether those steels are also susceptible to cracking in concentrated lithium bromide solutions. Initially two tests were conducted with a 68% LiBr solution at 160°C, each test containing 20 U-bend specimens of type 304 SS. In one test air was removed from the autoclave, and in the other it was not. Both tests lasted 3 weeks without interruption. In the autoclave that contained air every specimen had at least one crack that caused the specimen to fail; in the absence of air not a single specimen developed a crack. Interestingly, 18 of the failed specimens contained only a single crack; only two cracks were found in each of the other two specimens. Usually when cracking occurs, a single crack causes failure, but additional smaller cracks are found. Our results indicate that once the first crack nucleated, the crack growth rate was so great that the tensile stresses were relieved before other cracks could start.

In both tests the specimens remained free of visible films and appeared to have undergone only limited general attack. The solution from the autoclave without oxygen had a very slight blue-green color that slowly formed a flocculent reddish precipitate on exposure to air. In the presence of air, there was a fine reddish preprecipitate in the bottom of the liner.

Lithium chromate or lithium hydroxide is frequently used in lithium bromide solutions to inhibit corrosion of carbon steels.[8] Two additional tests were run to determine if those inhibitors had any effect on stress corrosion cracking of stainless steel. In one test the 68% LiBr solution contained 1000 ppm chromium as Li_2CrO_4. In the other, enough solid lithium hydroxide was added to make the 68% LiBr solution 0.1M (0.16%) in LiOH. Ten U-bend specimens of type 304 SS were exposed in each environment, and air occupied the free space in the autoclaves. Both autoclaves were heated at 160°C for 3 weeks.

All specimens exposed in the chromate-containing solution failed during the test. Most specimens contained more than one crack. Each crack was marked by a reddish-brown deposit, whereas the rest of the

surface had a slightly bluish film. There was a small amount of black precipitate in the bottom of the liner, but the solution had the same yellow color as at the start, indicating that most of the chromate ions were not reduced.

On the other hand, none of the specimens exposed in the solution containing lithium hydroxide developed a crack. The specimens had a loosely adherent brownish film, most of which could be removed by wiping with tissue. The solution contained a similarly colored precipitate that slowly settled.

These results clearly show that chromate ions did not inhibit stress corrosion cracking of stainless steel in lithium bromide solutions, but making the solution alkaline with lithium hydroxide completely stopped stress corrosion cracking of stainless steel under our test conditions.

After these tests were completed, the crystalline lithium bromide used to make the solutions was analyzed for the presence of chloride. Spark source mass spectroscopy indicated 55 ± 15 ppm chloride. This concentration in the dry salt corresponds to about 37 ppm chlorine in the solution, which in the absence of lithium bromide is sufficient to produce cracking in stressed austenitic stainless steel under our conditions of tests. Thus, there remains the possibility that the cracking we observed was due to the presence of trace amounts of chloride in the lithium bromide.

DISCUSSION AND CONCLUSIONS

The preliminary results obtained in this study show that most of the fluids we examined, with the exception of the organic solutions, were corrosive to at least one of the materials tested. It is also possible that additional corrosion may be encountered at longer testing periods. However, inhibitors, which could reduce corrosion in some cases, were not evaluated except in lithium bromide solutions.

Overall, the austenitic stainless steels appear to be able to withstand more of the test solutions than any other material that we examined. Of course, stainless steel is not suitable for containing

hot caustic solutions, and concentrated lithium bromide solutions containing either molecular oxygen or chromate ions readily crack highly stressed stainless steel; however, the latter attack can be prevented by removing the oxygen or chromate from the system or by adding lithium hydroxide to the solution.

It appears that stainless steel is not susceptible to cracking in a lithium bromide solution containing ammonia in the absence of air, but only a single test was run. In the same test a mild-steel stressed specimen developed an intergranular crack. Whether cracking will occur in either alloy in the presence of oxygen remains to be demonstrated. In the ammoniacal lithium bromide solution, stainless steel corroded at an appreciable rate initially while a protective coating formed, following which the corrosion rate was negligibly small. When methylamine was substituted for ammonia in the above solution, stainless steel developed a dark film, but its susceptibility to cracking was not examined.

On the basis of our limited results, an austenitic stainless steel seems to be the best choice for a material from which to construct a test facility for study of a wide variety of fluids. However, before such a facility is built, long corrosion tests are needed to confirm that stainless steel is as resistant a material as the preliminary tests indicate.

Although it was not unambiguously established, our data indicated that a concentrated lithium bromide solution is an aggressive cracking agent for highly stressed stainless steel. Our solutions contained low levels of chloride ions (~37 ppm), which in the absence of lithium bromide could produce cracking in stainless steel; however, chloride-induced cracking is usually inhibited by chromate ions[9] and, because cracking occurred in the presence of chromate, chloride ions did not appear to be responsible for the observed cracking. Because bromide ions do not usually induce cracking in austenitic stainless steels, an investigation into the cracking phenomenon in lithium bromide solutions should be informative.

The containment of concentrated sodium hydroxide solutions at high temperatures usually requires nickel or a high-nickel alloy.[10] Those materials are expensive compared with carbon steel, which is used in existing absorption machines. Our results show that both 90-10 and 70-30 cupronickel have high resistance to hot caustic solutions. They are less expensive than nickel and, in addition, have higher thermal conductivities. Although further testing is required, those alloys may be good choices for an absorption machine based on the sodium hydroxide—water system.

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