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Effects of Chloride and Caustic on the Cracking Behavior of Several Materials Under Alternate Wet and Dry Steam Conditions

J. C. Griess
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P.O. Box 62, Oak Ridge, Tennessee 37830
Price: Printed Copy $4.00 ; Microfiche $2.25

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ORNL-TM-4995
UC-79b, -h, -k
(Liquid Metal Fast
Breeder Reactors)

Contract No. W-7405-eng-26

METALS AND CERAMICS DIVISION

EFFECTS OF CHLORIDE AND CAUSTIC ON THE CRACKING BEHAVIOR OF SEVERAL MATERIALS UNDER ALTERNATE WET AND DRY STEAM CONDITIONS

J. C. Griess, J. P. Hammond, and W. A. Maxwell

SEPTEMBER 1975

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J. C. Griess, J. P. Hammond, and W. A. Maxwell[1]

ABSTRACT

The susceptibility of a number of alloys to stress-corrosion cracking in steam to which either chloride or caustic has been added was determined. U-bend specimens were alternately exposed to superheated steam and to saturated steam containing appreciable moisture. Since the solubility of sodium hydroxide in superheated steam is extremely low under the test conditions, caustic was added only during the saturated steam portion of the test cycle, and the maximum concentration was 100 ppm sodium hydroxide. All alloys tested in this environment — including low-alloy steels, types 410, 310, and 304 stainless steel, and high-nickel alloys — completely resisted cracking. In chloride environments either sodium or calcium chloride was added during both the superheated and saturated part of the cycle (maximum concentration, 10 ppm chloride). The oxygen concentration was 8 ppm. Both Hastelloy N and E-Brite 26-1 were very susceptible to cracking. Incoloy 800 weldments in the as-welded condition cracked in this test, while Incoloy 800 base metal and postweld heat-treated specimens did not. Inconel 601 and 2 1/4 Cr-1 Mo steel resisted cracking, but a single crack developed in one specimen each of Inconel 600, Inconel 625, and Hastelloy X.

INTRODUCTION

An important consideration in the selection of steam generator materials is the susceptibility of candidate materials to localized corrosion in impure steam. For example, in sodium-cooled reactors contamination of the boiler water and steam with sodium hydroxide is possible, and a knowledge of the effects produced by the caustic on materials in the system is required to assess corrosion damage that could occur under such faulted or off-specification conditions. Chloride ions are universal contaminants, and the effect of their presence in the system must also be considered. Both caustic and chloride ions are widely recognized as stress-corrosion cracking agents for certain alloys, and numerous investigations on this subject are reported. However, the effects of these two corrodents on

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[1]Southern Nuclear Department, Nuclear Utilities Service Corporation, Clearwater, Florida 33515.

various materials at temperatures intermediate between those of the evaporator and superheater outlets, where alternate wetting and drying of surfaces by boiler water entrained in the steam might occur, have not been widely investigated.

The present report discusses the effects of chloride ions and caustic on the cracking behavior of potential steam generator materials under alternately wet and dry steam conditions. Since we did not want to have solid chlorides deposit on our specimens during the superheated part of the test cycle, we determined the solubility of sodium and calcium chloride — the two salts used in these tests — under our test conditions, and these results are also presented.

EXPERIMENTAL PROCEDURES

All the testing described in this report was conducted in a "once-through" loop made of Inconel 625. This alloy is reportedly very resistant to stress-corrosion cracking in either caustic or chloride environments.[2] Figure 1 shows the loop with the heaters in place but without the thermal insulation. The vertical flanged pipe (upper left) connects to a steam source discussed below. The flow rate of the steam in the tests was 100 lb/hr (45 kg/hr), which corresponds to a velocity of about 5 ft/sec (1.5 m/sec) past the test specimens. Contaminants were added to the incoming steam through a flanged connection (upper left side of loop containment shield). The steam was condensed after exit from the loop and the concentration of contaminants was determined in the condensate. The loop has two chambers or autoclaves in which test specimens can be placed (flanged openings on either side of the shield). The inside diameter of the chambers is 1.9 in. (48 mm) and each is 14 ft (4.3 m) long.

The loop is located at the Bartow Plant of the Florida Power Company, and steam for the tests is taken directly from the main steam line to one of the turbines. At this point, the temperature and pressure of the steam in the line are 538°C (1000°F) and 1800 psi (12.4 MPa), respectively, and these values are adjusted to the desired levels before the steam contacts the test specimens. The loop is operated by the Southern Nuclear Department of the Nuclear Utilities Services Corporation for the Oak Ridge National Laboratory. This loop was first put into service in November 1972.

The test specimens were U-bends with a 1/2-in. (13-mm) bend radius and were formed from strips 2 7/8 by 1/2 by 1/16 in. (73 by 1.27 by 1.6 mm). Specimens designated as "ground" were finished on a belt grinder using 100-mesh abrasive. When welded specimens were tested, the 1/16-in.-thick (1.6-mm) strips of the above size were cut from 1/2-in.-thick (13-mm) plates that had multipass welds across their centers. The welding procedure is described in another paper.[3] The welded test specimens had the

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[2] *Huntington Alloys Inconel Alloy 625,* revised, International Nickel Company, Inc., 1970.

[3] J. P. Hammond, P. Patriarca, G. M. Slaughter, and W. A. Maxwell, "Corrosion of Incoloy 800 and Nickel Base Alloy Weldments in Steam," *Weld. J. (Miami)* 52(6): 268-s–280-s (June 1973).

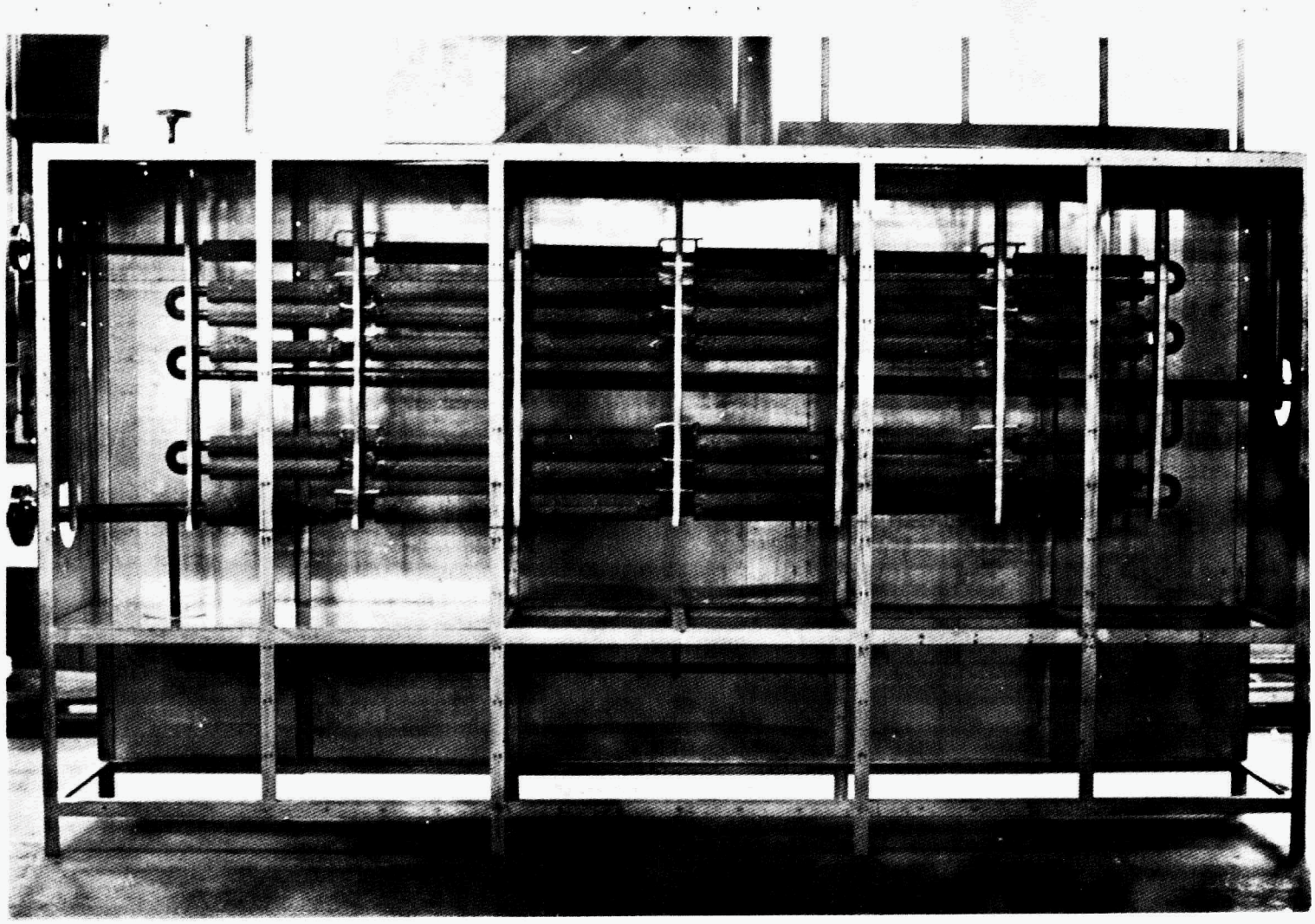

Fig. 1. Inconel 625 Loop with Heaters in Place, but without Insulation. Nuclear Utilities Services Photograph.

welds at the apex of the U's. The U-bends were mounted in holders as shown in Fig. 2. The 1/2-in. bend radius provided an extension of about 6.2% in the outer test surface. The specimens were arranged in the loop so that the bottom of the U specimens was about 1/16 in. off the bottom of the autoclaves. In this position the most highly strained part of the specimen was exposed to water during the saturation phase of the test. Other details of the testing procedure have been described previously.[4]

Once a week the specimens were removed from the loop and examined under 10× magnification for evidence of cracks. When a crack was found, its appearance was noted, and marked on a sketch, and the specimen was returned to the loop unless complete failure had occurred. At the end of the test, all remaining specimens were examined with 30× magnification. Clear-cut cases of cracks were recorded, and questionable ones were examined metallographically.

RESULTS

Tests in Caustic Environments

After the Inconel 625 Loop had undergone shakedown tests to establish design conditions and to check the injection and safety systems, the solubility of sodium hydroxide in superheated steam was determined under conditions of interest. Increasing amounts of a sodium hydroxide solution were injected into the incoming steam while the loop temperature and pressure were held constant at 427°C (800°F) and 900 psi (5.2 MPa), respectively. We determined the concentration of caustic at which the electrical conductivity of the condensate from the effluent ceased to increase. Conductivity did not increase measurably with injection rate, indicating the solubility of sodium hydroxide in steam at 427°C (800°F) and 900 psi (6.2 MPa) is very low. In solubility determinations such as the one described here, the added solute that did not dissolve in the steam presumably deposited on the loop walls.

Since these corrosion tests were designed to determine the resistance of a number of alloys to stress-corrosion cracking under alternating wet-dry conditions, the temperature of the loop was cycled between a superheat temperature and the saturation temperature while the steam pressure was maintained constant. For the test with caustic the pressure was 900 psi (6.2 MPa) and the temperature was alternated between 371 and 282°C (700 and 540°F) on a 48-hr cycle. Sodium hydroxide was injected only during the saturated part of the cycle (540°F).

In preliminary corrosion tests the concentration of sodium hydroxide injected into the loop was progressively increased from 2 to 4 to 10 ppm over a ten-week period, and a 12-week run was made with 100 ppm NaOH. Only during the time when 4 ppm NaOH was added was oxygen (4 ppm)

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[4]J. P. Hammond, P. Patriarca, G. M. Slaughter, and W. A. Maxwell, *Comparative Results on Chloride Stress Corrosion Cracking of Steam Generator Materials in Cyclic Steam Environments*, ORNL-5031 (June 1975).

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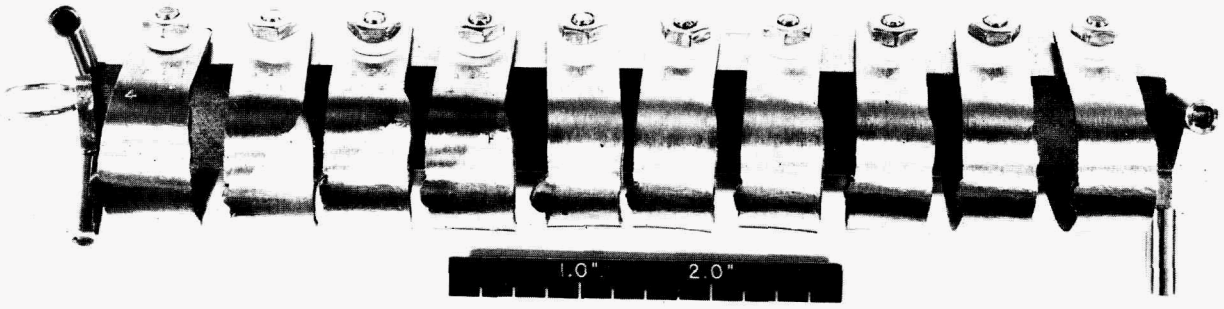

Fig. 2. U-Bend Specimens Mounted on Holder.

introduced into the system; at all other times oxygen was held below the limit of detection, (less than 0.007 ppm). During these tests, welded U-bend specimens of 2 1/4 Cr-1 Mo steel, type 410 stainless steel, Incoloy 800, Inconel 625, and Hastelloy X and nonwelded U-bends of 2 1/4 Cr-1 Mo, type 304 stainless steel, Incoloy 800, Inconel 601, and Inconel 625 were in the loop. No evidence of cracking or other forms of localized attack was found in any of the specimens.

An additional run, designated Test A, was made in the same manner except 50 ppm NaOH was injected during the saturation portion of the cycle. In addition to the alloys exposed in the earlier runs, U-bends of 9 Cr-1 Mo steel, sensitized type 304 stainless steel, type 304 stainless steel welded with type 308 filler metal, welded and unwelded Inconel 600, types 310 and 410 stainless steel, Hastelloy alloys C, G, N, and S, and E-Brite (electron-beam-melted 26 Cr-1 Mo) were included. Specimens of these materials were tesed in triplicate. This run lasted 18 weeks, and during the entire test none of the specimens showed any evidence of cracking. Thus, in all these cyclic tests with relatively low concentrations of caustic not a single crack was found in any of the materials tested.

Tests in Chloride Environments

Only one extended cyclic test was made with chloride in the system, and during this test sodium chloride was employed as the contaminant for the earlier part and calcium chloride for the later part. The pressure and oxygen concentration were held constant at 1625 psi (11.2 MPa) and 8 ppm, respectively. The temperature was cycled on a 48-hr period between 385 and 318°C (725 and 605°F). During the superheat part of the cycle the chloride concentration (as NaCl) was maintained at 4.2 ppm for the first 3146 hr of test and 2.7 ppm (as $CaCl_2$) for the rest of the test (2960 hr). The chloride concentration during the saturated phase of the cyclic test was increased to 10 ppm regardless of the salt that was used.

The chloride concentrations in the superheated steam represent the solubility limits of the two salts, as determined in our test loop. These solubility measurements were made with the loop temperature and pressure maintained constant. Solutions with known concentrations of either calcium or sodium chloride were added to the superheated steam entering the loop. As the volume of solution added to the loop increased, the concentration of chloride in the effluent condensate increased until the solubility limit of the chloride in the steam was reached. Further increases in the addition rate resulted in deposition of solid chlorides on the loop walls and no increase in the chloride concentration in the condensate. The above procedure was repeated decreasing instead of increasing the chloride feed rate, and the same results were obtained. Between successive runs the loop temperature was lowered to the saturation temperature to wash deposited chlorides from the loop. Our results are shown in Table 1. The two values obtained with sodium chloride at 482°C (900°F) were obtained a month apart and demonstrate the reproducibility of the data. The results are in reasonable agreement with published results[5] for sodium chloride.

Table 1. The Solubility of Sodium and Calcium Chlorides in Superheated Steam at 1625 psig (11.2 MPa)

Chloride	Temperature		Concentration, ppm	
	(°C)	(°F)	As Chloride	As Salt
NaCl	385	725	4.2	6.9
$CaCl_2$	385	725	2.7	4.2
NaCl	482	900	2.0	3.3
NaCl	482	900	2.0	3.3
$CaCl_2$	482	900	1.2	1.9

The test with chloride, designated 2A, was started November 1, 1973, and was terminated on September 24, 1974. During the period between March 15 and May 10, 1974, the specimens were removed from the loop, and the loop was used for another purpose. In addition to the specimens that were placed in test at the start, a second group was added to the loop on June 14, 1974. The specimens initially installed in the loop were made from three alloys, Incoloy 800, Hastelloy N, and E-Brite, each with a variety of different surface treatments. These treatments, the number of specimens exposed, the number that cracked, and the time for the first appearance of cracks are shown in Table 2. None of the Incoloy 800 specimens cracked, but extensive cracking of the other two alloys was noted. Most of the notations under Surface Conditions are self-explanatory. However, with each alloy the last three conditions need explanation.

[5]M. A. Styrkovitch et al., *Teploenergetika* 4:3 (1957); 10: 50 (1963).

Table 2. U-Bend Specimens Exposed in Test 2A of the Inconel 625 Loop for 6106 hr

Alloy	Surface Condition	Number of Specimens Cracked	Time for First Crack (weeks)
Incoloy 800	Ground	0 of 3	
	Ground, annealed	0 of 3	
	Electropolished	0 of 3	
	Half ground, half electropolished	0 of 3	
	Line electropolished, surface ground	0 of 3	
	Line ground, surface electropolished	0 of 3	
	As above plus annealed	0 of 3	
Hastelloy N	Ground	0 of 3	
	Ground, annealed	3 of 3	6–8
	Electropolished	3 of 3	5–9
	Half ground, half electropolished	0 of 3	
	Line electropolished, surface ground	2 of 3	8–12
	Line ground, surface electropolished	3 of 3	11–25
	As above plus annealed	3 of 3	3
E-Brite (26 Cr-1 Mo)	Ground	2 of 3	14
	Ground, annealed	2 of 3	13–14
	Electropolished	3 of 3	14–19
	Half ground, half electropolished	3 of 3	11–15
	Line electropolished, surface ground	3 of 3	5–11
	Line ground, surface electropolished	2 of 3	4–12
	As above plus annealed	3 of 3	11–25

In each case a line 1/16 in. (1.6 mm) wide was made across the test strip so that when the U was formed the line was at the apex. The designation "line electropolished, surface ground," indicates that the specimen was first ground and all but the very narrow strip was covered with masking tape so that the line was formed by electropolishing. The notation "line ground surface electropolished," indicates an identical treatment except that the area to become the line was masked before electropolishing. Thus, the line was above the plane of the rest of the surface. The same notation as the latter followed by "annealed" means that following the electropolish the strip was annealed before forming the U.

Table 3 shows the specimens that were exposed during the latter part of Test 2A and the results that were obtained. At the end of the test one holder of U-bends that had been installed on June 14, 1974 was stuck and could not be removed. After 2828 hr of exposure in the next test, Test B, the holder was freed and it was removed at that time. The environmental conditions were the same in Test B as in 2A, and consequently Table 3 also shows the results obtained from these specimens.

As noted in Table 3 only the two E-Brite, the three as-welded Incoloy 800, one as-welded Inconel 600, one ground Hastelloy X, and one ground Inconel 625 specimens developed cracks during the latter part of the test. Only two of the three cracks in the welded Incoloy 800 and the two in E-Brite were obvious during the test. All the other cracks were found only at the end of the test by metallography.

Table 3. U-Bend Specimens Exposed for Only the Last 2169 hr of Test 2A (Except as Noted) in the Inconel 625 Loop

Alloy	Surface Condition	Number of Specimens Cracked	Time for First Cracks (weeks)
Incoloy 800	As received[a]	0 of 3	
	Ground	0 of 3	
	Welded with Inco 82, annealed[b]	0 of 3	
	Welded with Inco 82, ground	3 of 3	6–13
Inconel 600	Welded with Inco 82, annealed[b]	0 of 3	
	Welded with Inco 82, ground	1 of 1	13
Inconel 601	Ground[c]	0 of 2	
Inconel 625	As received[c]	0 of 3	
	Ground[c]	1 of 3	30
Hastelloy X	As received	0 of 3	
	Ground	1 of 3	13
410 Stainless Steel	Welded with same, annealed[d]	0 of 2	
E-Brite (26 Cr-1 Mo)	Ground	2 of 2	1–2
2 1/4 Cr-1 Mo	Welded with same, ground	0 of 3	
	Welded with same, annealed[e]	0 of 3	
	Ground, annealed	0 of 3	
	Ground	0 of 3	
2 1/4 Cr-1 Mo-Nb	Ground, annealed	0 of 2	

[a] Two of three specimens exposed for a total time of 4997 hr.

[b] 982°C (1800°F), 10 min, rapid cool.

[c] All specimens exposed for 4997 hr.

[d] 760°C (1400°F), 10 min, rapid cool.

[e] 732°C (1350°F), 10 min, rapid cool.

Figure 3 shows part of a crack formed in a welded Incoloy 800 U-bend. The crack progressed intergranularly through the large-grained heat-affected zone.

The only indication of a crack in Hastelloy X was found under 30× magnification, and its presence was confirmed by metallographic examination. Figure 4 shows that the crack was intergranular.

Fig. 3. Crack Formed in a Welded Incoloy 800 U-Bend Exposed During the Last 2169 hr of Test 2A in the Inconel 625 Loop. 100×.

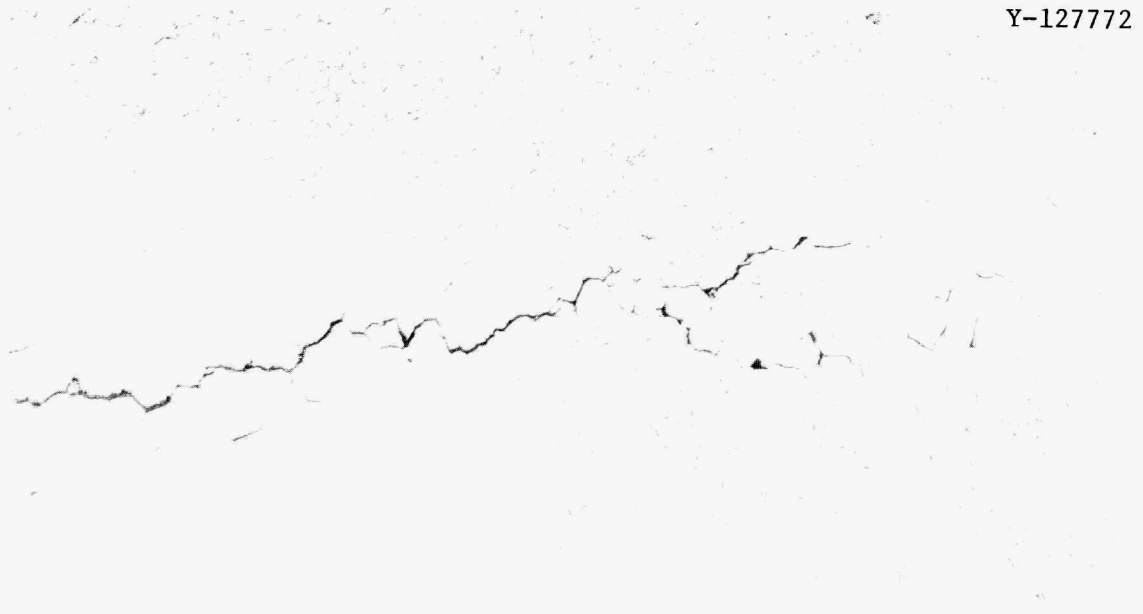

Fig. 4. Crack Formed in a Hastelloy X U-Bend During the Last 2169 hr of Test 2A in the Inconel 625 Loop. 100×.

The one crack in the welded Inconel 600 specimen was not apparent until the end of the test. Figure 5 shows the location of the crack with reference to the weld and a photomicrograph of the crack. The crack is intergranular. In contrast to the crack in the welded Incoloy 800 specimen, the crack appeared to be in the unaffected base metal and not in the heat-affected zone.

Figure 6(a) is a photomicrograph of the as-polished surface of the Inconel 625 U-bend showing the one crack found during the later part of the test. Note the small cracks emanating from the side of the main crack and the presence of corrosion products in the crack. Figure 6(b) is a further enlargement after etching and shows the intergranular nature of the crack.

DISCUSSION

The materials used in the tests described here were subjected to both elastic and plastic deformation. In fact, the strain in the U-bend specimens amounted to about 6%. Furthermore, the relaxation of stresses during these tests was small. Based on springback of the U's the maximum relaxation was estimated to be only 10 to 15% in the chloride tests and even less in the caustic tests. Thus these experiments provided a severe test of the susceptibility of these materials to stress-corrosion cracking.

Repeatedly wetting a surface with a dilute solution and subsequently drying it can produce locally high concentrations of nonvolatile substances on the drying surface. By this means very dilute solutions, which may of themselves have no adverse effect on materials, can be concentrated and produce serious localized attack. The tests described in this report were conducted under such conditions.

In the caustic tests the solubility of sodium hydroxide in superheated steam was so low that measurable concentrations could be added only when water was present in the stean (saturated). Although the maximum concentration added to the steam was only 100 ppm, it was expected that as the temperature was increased from the saturation part of the cycle, droplets of the dilute solution would become concentrated enough to produce localized attack, specifically stress-corrosion cracking, on most of if not all alloys. The fact that no cracking was noted in any alloy is encouraging from the standpoint of small sodium leakages into the steam systems of sodium-cooled reactors.

The results obtained in the chloride tests presented in Table 2 are basically in agreement with previous data. Thus, nonwelded Incoloy 800 did not crack. But as-welded specimens did crack (Table 3). Although none of the unwelded specimens of Incoloy 800 or those that were welded and annealed, cracked, our results must not be interpreted as indicative of immunity of this alloy to cracking. Hammond[6,7] showed that similar specimens in both the ground and ground and annealed states developed cracks

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[6]J. P. Hammond, "General Corrosion," *Fuels and Materials Development Program Quart. Progr. Rep. Sept. 30, 1971*, ORNL-TM-3550, pp. 134–42.

[7]Ibid. *Dec. 31, 1971*, ORNL-TM-3703, pp. 142–50.

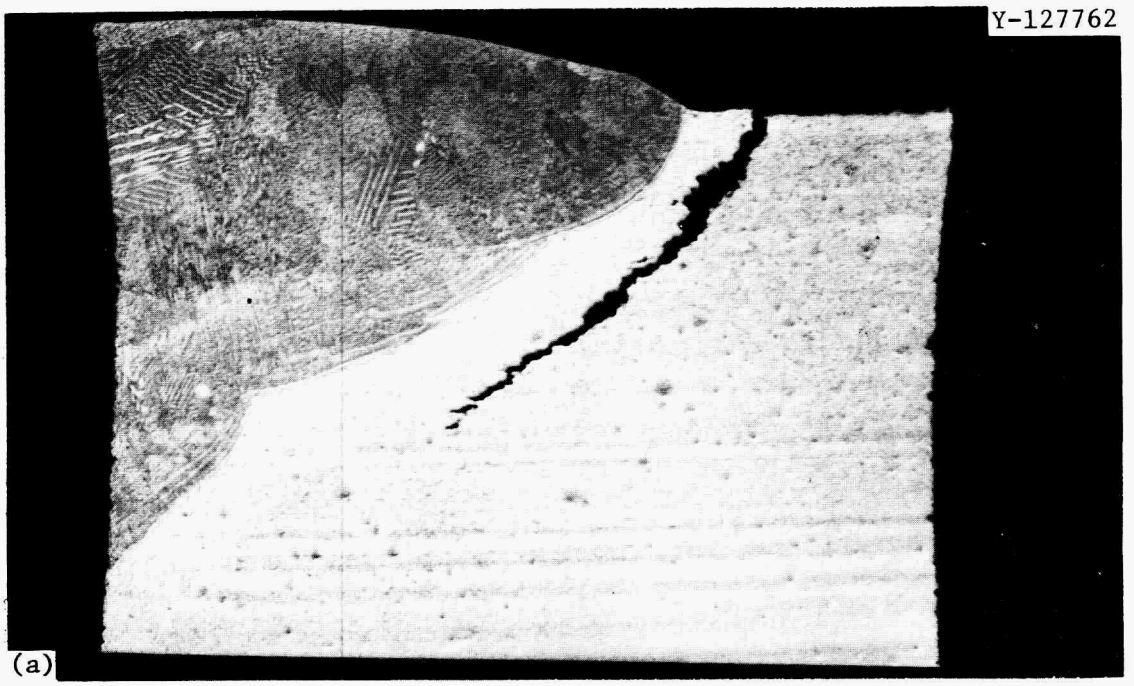

Fig. 5. Crack Formed in a Welded Inconel 600 U-Bend During the Last 2169 hr of Test 2A in the Inconel 625 Loop. (a) 12.5×. (b) Etched. 100×.

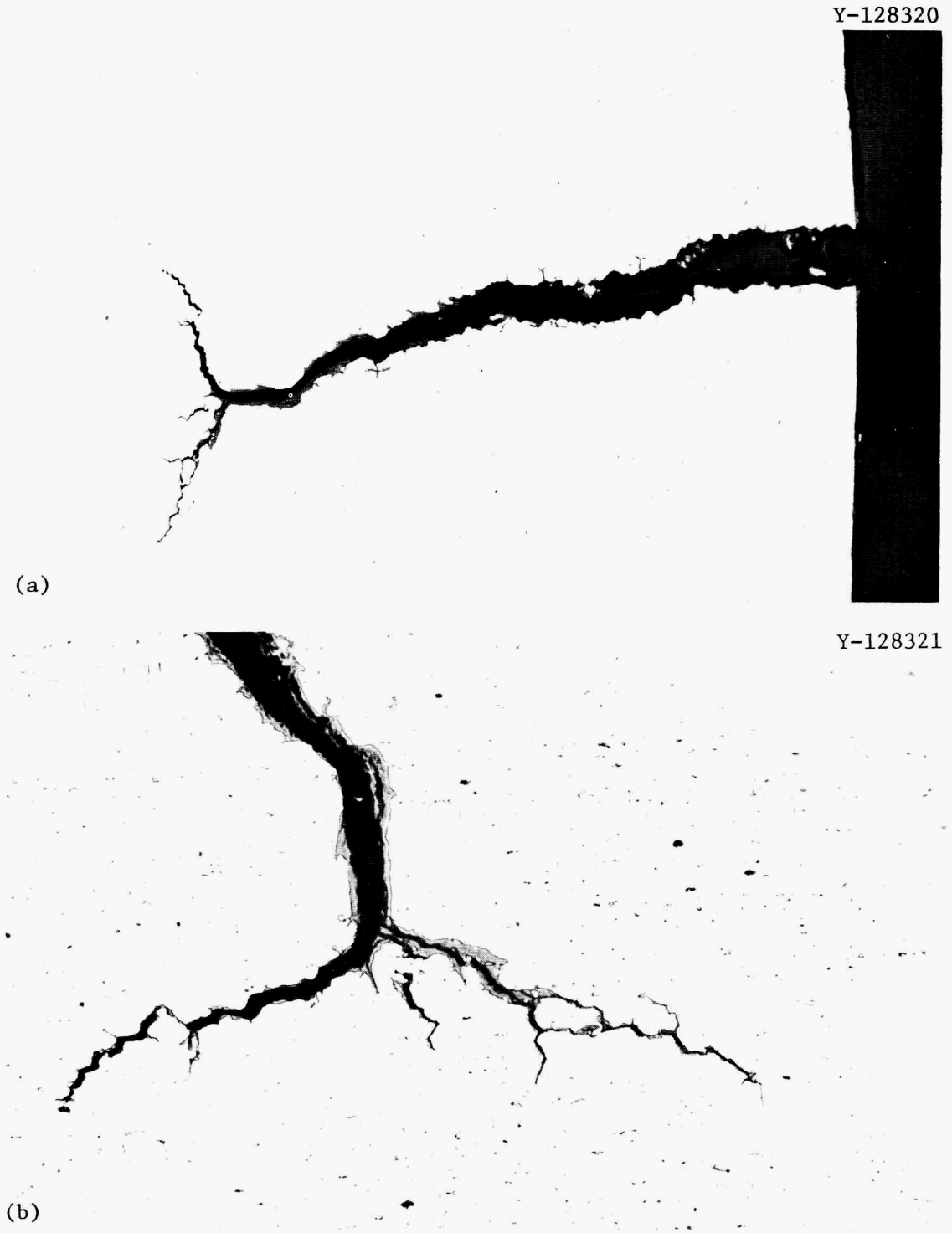

Fig. 6. The One Crack Found in an Inconel 625 U-Bend Specimen Exposed for 2169 hr in Test 2A and 2828 hr in Test B. (a) As polished, 100×. (b) End of the crack after etching, 250×.

in cyclic tests with 10 ppm chloride and 20 ppm O_2. In the same environment he also showed that some specimens welded with Inconel 82 filler metal and exposed both as welded and as postweld annealed cracked. However, in agreement with our results, welded specimens that had been annealed were less susceptible to cracking than those that were tested as welded.

Also, E-Brite had been shown to be very susceptible to chloride-induced cracking in high-temperature steam[8,9] and the present tests indicate that this susceptibility is essentially independent of surface condition. At much lower temperatures this alloy has a very high degree of resistance to both pitting and cracking in chloride environments, but in our tests its resistance to cracking was extremely low.

The very high incidence of cracking in Hastelloy N was expected in view of previous experience.[6,7] Why cracking was not observed in any of the three ground specimens or in the three half ground and half electropolished specimens is not understood. In the above referenced tests, specimens with totally ground surfaces cracked, and in the present test the totally electropolished specimens failed.

Both Inconel 601 and Inconel 625 are highly resistant to chloride-induced cracking. A large number of tests with these two alloys has been completed in other parts of the test program (not included in this report), and the only crack found in either alloy was the small crack shown in Fig. 6. Furthermore, no failures have been found in the Inconel 625 loop, which has been in use with chloride-containing steam for about one year. Continued operation of this loop in chloride environments is planned; but, when decommissioned, it will be thoroughly examined for evidence of corrosion damage.

As expected, 2 1/4 Cr-1 Mo steel showed no tendency to crack in the oxygenated chloride environment. However, its resistance to general corrosion was poor. The U-bend specimens were covered with heavy scale, much of which was easily removed by gentle tapping of the specimen. It should be pointed out, however, that the conditions used in this test were not typical of those that could exist for any extended time in a reactor steam system, but rather were chosen to determine the degree of resistance of a number of alloys to chloride-induced cracking. The high general or uniform corrosion experienced by 2 1/4 Cr-1 Mo steel in these tests, therefore, in no way compromises its use in the superheater and evaporator of the steam generator in the Clinch River Breeder Reactor. The resistance of this alloy to cracking in the presence of chloride as well as its demonstrated resistance to cracking in caustic environments, both in our tests and those of Indig,[10] means that failure of the steam

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[8]J. P. Hammond and G. M. Slaughter, "Corrosion of Advanced Steam Generator Alloys," *Metals and Ceramics Div. Annu. Progr. Rep. June 30, 1972*, ORNL-4820, pp. 42–46.

[9]J. P. Hammond and W. A. Maxwell, "Florida Loop Program on General and Stress Corrosion Cracking of Steam Generator Alloys," *Fuels and Materials Development Program Quart. Progr. Rep. Sept. 30, 1972*, ORNL-TM-4055, pp. 212–2.12.

[10]M. E. Indig, *Stress Corrosion Studies of LMFBR Steam Generator Materials*, GEAP-12536 (August 1974).

system in the Clinch River Breeder Reactor by a stress-corrosion cracking mode is extremely remote.

As indicated earlier, the stress levels in the specimens used in these tests were very high (6.2% total strain with little thermal relaxation of stresses). When cracking occurred, it usually originated in plastically deformed surfaces. Other tests in chloride environments with many of the same alloys are now being conducted with a specimen design for which a constant stress exists in the test section. The stresses being investigated are in the range of design stresses, and fewer cracks are to be expected. The results of those tests will be reported later.

ACKNOWLEDGMENTS

The authors wish to express their appreciation to the personnel of the Bartow Plant of the Florida Power Corporation for their support and cooperation in conducting these experiments and to Scott Theiss of the NUS Corporation for examining the specimens during the course of the tests. The metallographic examinations for this report were prepared by C. E. Zachary of ORNL. The report was edited by Sigfred Peterson and typed and prepared for reproduction by Regina Collins.

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