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STRESS CORROSION CRACKING OF Fe-Ni-Cr-Mo, Ni-Cr-Mo AND Ti ALLOYS IN 90°C ACIDIC BRINE

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ABSTRACT

Susceptibility to stress corrosion cracking (SCC) of candidate materials for the inner container of the multi-barrier nuclear waste package was evaluated by using wedge-loaded precracked double-cantilever-beam (DCB) specimens in deaerated acidic brine (pH ≈ 2.70) at 90°C. Materials tested include Alloys 825, G-30, C-4, 625 and C-22; and Ti Grade-12. Duplicate specimen of each material was loaded at different initial stress intensity factor (K_I) values ranging between 23 and 46 ksi$\sqrt{in}$. Both metallography and compliance method were used to determine the final crack length. The final stress intensity for SCC (K_{ISCC}) was computed from the measured final wedge load and the average crack length. The results indicate that, in general, the final crack length measured by metallography and compliance was very close to each other, thus, providing very similar K_{ISCC} values. While tests are still ongoing, the preliminary results suggest that, compared to other five alloys tested, Alloy 825 may exhibit the maximum tendency to SCC.

Keywords: Stress corrosion cracking, double-cantilever-beam specimen, nickel-rich and nickel-base alloys, titanium alloy, stress intensity factor.

INTRODUCTION

The high-level nuclear waste package design, currently being considered to contain the nation's spent nuclear fuel and vitrified defense nuclear waste at the potential underground Yucca Mountain repository, will consist of two layers of metal barriers. The outer cylindrical barrier, made of corrosion-allowance material, will be thicker than the inner barrier which will be fabricated from corrosion-resistant material. The thick corrosion-allowance outer barrier is being designed to undergo degradation at a very low rate resulting from the potential repository environment, while providing galvanic protection to the thinner corrosion-resistant inner barrier. The precise method of fabricating these waste packages has not yet been finalized. Irrespective of the fabrication technique, some form of welding of the container metallic materials may be involved in producing cylindrical packages of large diameters, which may generate enough residual stresses causing the waste package materials to become susceptible to SCC as they come in contact with the repository environment. This paper presents the results of SCC tests of candidate inner container metallic materials exposed to an aqueous environment relevant to the potential underground repository.

MATERIALS AND EXPERIMENTAL PROCEDURE

Materials tested include iron-nickel-chromium-molybdenum (Fe-Ni-Cr-Mo) Alloys 825, G-3 and G-30; Ni-Cr-Mo Alloys C-4, C-22 and 625; and a titanium-base alloy Ti Grade-12. Their chemical compositions are given in Table 1. The rectangular DCB specimens (4 inch long, 1 inch wide, and 0.375 inch thick) with one end slotted for wedge-loading, and V-shaped side grooves extending from the slot to the opposite end were fabricated from mill-annealed plate materials by a qualified vendor. Additional thermal treatments were not given to these specimens prior to their exposure to the test environment. The DCB specimens were machined in such a way that the crack plane was perpendicular to the short transverse direction, and that crack propagation would occur in the longitudinal rolling direction. The test specimens were fatigue-precracked under load-control (load ratio of 0.10) at a frequency of 20 Hz using an Instron Servohydraulic testing machine having a 55 kip load-cell. A clip gauge was attached to the specimen to determine the precrack length from the compliance measured during the fatigue cycle. The precracked DCB specimen was then loaded by inserting a double taper wedge (Figure 1) made of a similar material into the specimen slot using the same Instron testing machine. The wedge thickness ranged between 0 110 and 0.115 inch to provide specimen arm-displacement of about 0.016 to 0.020 inch.

A total of eight specimens per candidate alloy were tested. Duplicate specimens of each alloy were totally immersed in a vertical position into the test solution contained in a glass vessel which was heated to the desired test temperature by means of a hot water bath. The environment used was a 90°C deaerated acidic brine (pH≈2.70) containing 5 weight percent NaCl The selection of the acidic brine as test environment was based on the results of a recent electrochemical localized corrosion study[1] which revealed the most severe pitting and crevice corrosion tendency of susceptible container materials in a similar environment The pH of the test solution was measured before, during, and after each test. Tests were performed for periods ranging between one and eight months. At the conclusion of each test, the specimens were visually examined followed by an optical microscopic evaluation of cracking along the side grooves. The final or equilibrium wedge load was then measured by applying a separating force to the specimen arms in the Instron testing machine. The final crack length was measured by both compliance and metallographic techniques. For metallographic measurement, the test specimen was pulled apart and the crack length was measured on the broken faces Values for K_I and K_{ISCC} were obtained using the following equation[2]

$$K = \frac{Pa\,(2\sqrt{3} + 2.38\,h/a)\,(B/B_n)^{1/\sqrt{3}}}{Bh^{3/2}}$$

where: P = Wedge load (before or after exposure), measured in the loading plane
a = Initial or final crack length, measured from the load line
h = Height of each arm
B = Specimen thickness
B_n = Web thickness

At the time of writing of this paper the 8-month tests are ongoing for all six alloys. Therefore, results of testing conducted for periods ranging between 1 and 5 months will be presented in the next section.

RESULTS AND DISCUSSION

Results indicate that for Alloy 825, which had shown the maximum susceptibility to localized corrosion in a previous study[3], the stress intensity factor value was significantly reduced upon exposure to the test solution, as shown in Table 2. Data indicate that, compared to the K_I value, the final stress intensity (K_{ISCC}) was at least 8 to 21 ksi$\sqrt{in}$ lower after testing for one, two, and four months. The average crack growth during these test periods was 0.036 inch, 0.065 inch, and 0.046 inch, respectively. Compared to Alloy 825, the average crack growth for Alloy G-30 was 0.011 inch following exposure for one month. The crack extension in this alloy was approximately 0.031 inch for both two and four month exposure period, suggesting that the crack-growth might have stopped after two months. The reduction in stress intensity factor ($\Delta K = K_I - K_{ISCC}$) value for this material upon completion of testing was much lower (approximately 2 to 7 ksi$\sqrt{in}$) compared to that for Alloy 825.

A comparison of crack-growth behavior of Alloys C-4 and C-22 indicates that the later alloy did not show any crack extension after one-month testing even though the applied K_I value for both materials was within a very narrow range (36 - 40 ksi$\sqrt{in}$). The extent of crack growth in both alloys was very similar (0.036 - 0.037 inch) when tested for two months. Data also indicate that Alloy C-4 showed an average crack extension of 0.036 inch after four months of testing, suggesting that no appreciable crack growth occurred between two and four months of testing. However, for Alloy C-22, the average crack growth was almost doubled (0.036 inch versus 0.065 inch) as the test duration was increased from two to five months.

As to the crack growth behavior of Alloy 625, no measurable crack extension was observed in this material after testing for two months. An average crack extension of 0.042 inch was observed in this alloy when tested for three months. For Ti Grade-12 only the four-month testing has been completed, showing an average crack growth of 0.033 inch which is very similar to that for Alloys G-30 and C-4 tested for almost an identical duration. ΔK value for Alloy 625, and Ti Grade-12 was very low, ranging between 2 and 5 ksi$\sqrt{in}$. The relationship between average crack-growth and exposure time for all six alloys is illustrated in Figure 2.

A comparison of pH of the test solution measured before, during, and after SCC testing indicate that for Alloy 825 the pH was shifted to more neutral values (up to 6.55) with increased exposure time. At the completion of testing, the test solution showed some light orange-colored corrosion product at the bottom of the cell, which was not analyzed. It is possible that the higher pH values may be the result of dissolution of elements such as iron, copper and nickel. An examination of Alloy 825 DCB specimen revealed significant crevice corrosion at the slotted end where the wedge was inserted (Figure 1). A shift in pH from 2.66 to 5.51 was also observed with Alloy C-4 after four months of testing. The amount of corrosion product in this case was very negligible. No attempt has yet been made to evaluate the morphology of cracking observed in materials tested so far. Future effort will be made to study the mode of cracking (intergranular versus transgranular) in selected materials.

SUMMARY AND CONCLUSIONS

Wedge-loaded fatigue-precracked DCB specimens were used to study the SCC behavior of Alloys 825, G-30, C-4, C-22 and 625; and Ti Grade-12 in an acidic brine at 90°C. The initial and the final stress intensity factor (K_I and K_{ISCC}) values were compared. The average crack growth was evaluated as a function of test duration. The significant conclusions drawn from this study are the following:

- Maximum reduction in stress intensity (ΔK) due to SCC was observed with Alloy 825 during comparable testing periods. The crack extension following two-month and four-month tests was also maximum with Alloy 825, indicating its worst SCC resistance compared to other alloys tested. In addition, this material showed a tendency to crevice corrosion at the slotted end of the test specimen.
- ΔK for Alloy 625 and Ti Grade-12 was within a very narrow range of 2 to 5 ksi$\sqrt{in}$. Although Alloy 625 did not show any crack extension in the two-month test, substantial crack growth occurred during the three-month test. Moderate crack extension was observed with Ti Grade-12 after four months of testing, the magnitude being very similar to those experienced by Alloys G-30 and C-4.
- Alloy C-22 did not exhibit any crack extension during one-month test. But cracking occurred in both two-month and four-month tests, showing enhanced crack growth during the longer test duration.
- The pH became more neutral during the testing of Alloy 825, possibly due to the dissolution of metallic elements.

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Table 1

Chemical Composition of Materials Tested (wt%)

Material	Heat #	C	Mn	P	S	Si	Ni	Cr	Mo	Fe	Ti	Al	Cu	Others
Alloy 825	L393	0.01	0.32	0.017	0.002	0.18	Bal	23.12	3.28	27.04	1.00	0.10	1.69	—
Alloy G-30	E084	0 01	0.60	0.011	<0 002	<0.05	Bal	29.30	5.00	14.80	—	—	1.60	Nb: 0 70 Co: 0.47 W: 2.70
Alloy C-4	K991	0 004	0 16	0 006	0.006	0.02	Bal	15.68	15.49	0.25	0 21	—	—	—
Alloy C-22	L380	0 004	0.15	0.010	0.007	0.027	Bal	21.40	13 41	4.90	—	—	—	W: 3 10 Co. 1.50 V 0.12
Ti Gr-12	D145	0.015	—	—	—	—	0.72	—	0.29	0 10	Bal	—	—	O 0.12 N. 0.008 H: 0.008
Alloy 625	K221	0.01	0.08	0.007	<0 001	0.05	Bal	21.90	9.02	4.41	0.25	0.21	—	Nb+Ta:3.4

Table 2

Results of SCC Tests

Material	K_I (ksi√in)	K_{ISCC} (ksi√in)	ΔK (ksi√in)	Avg. Crack Growth (inch)	Test Duration (hours)
Alloy 825	35.57, 38.43	27.26, 18.45	8.31, 19.98	0.036	738
	25.84, 39.68	13.41, 18.43	12.43, 21.25	0.065	1484
	38.48, 35.30	25.43, 21.66	13.05, 13.64	0.046	2898
Alloy G-30	41.07, 40.19	37.79, 38.35	3.28, 1.84	0.011	811.50
	36.56, 37.35	33.77, 33.53	2.79, 3.82	0.031	1415.50
	38.04, 37.95	31.10, 31.42	6.94, 6.53	0.031	2879.50
Alloy C-4	38.14, 42.48	30.33, 41.21	7.81, 1.27	0.036	716
	36.72, 42.28	29.31, 26.23	7.41, 16.05	0.037	1458
	43.89, 37.72	38.70, 32.77	5.19, 4.95	0.036	2880
Alloy C-22	38.78, 32.44	28.73, 28.78	10.05, 3.66	None	698
	35.12, 36.33	27.96, 30.94	7.16, 5.39	0.036	1414.50
	36.59, 36. 35	29.58, 32.39	7.01, 3.96	0.065	3598
Alloy 625	40.01, 37.20	37.84, 35.97	2.17, 1.23	None	1467.50
	45.02, 38.11	41.20, 33.00	3.82, 5.11	0.042	2180.50
Ti Grade-12	23.82, 23.49	19.51, 20.67	4.31, 2.82	0.033	2927.50

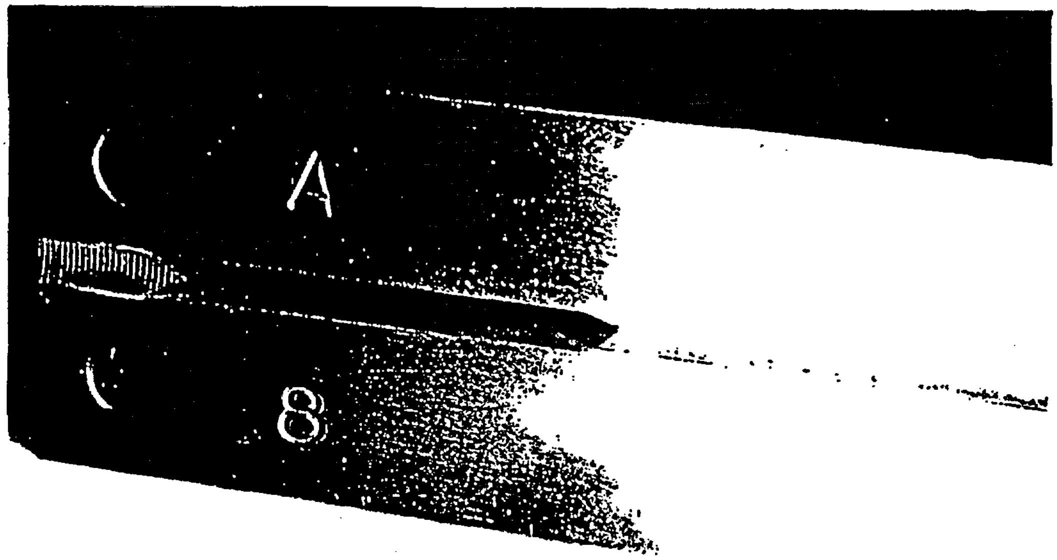
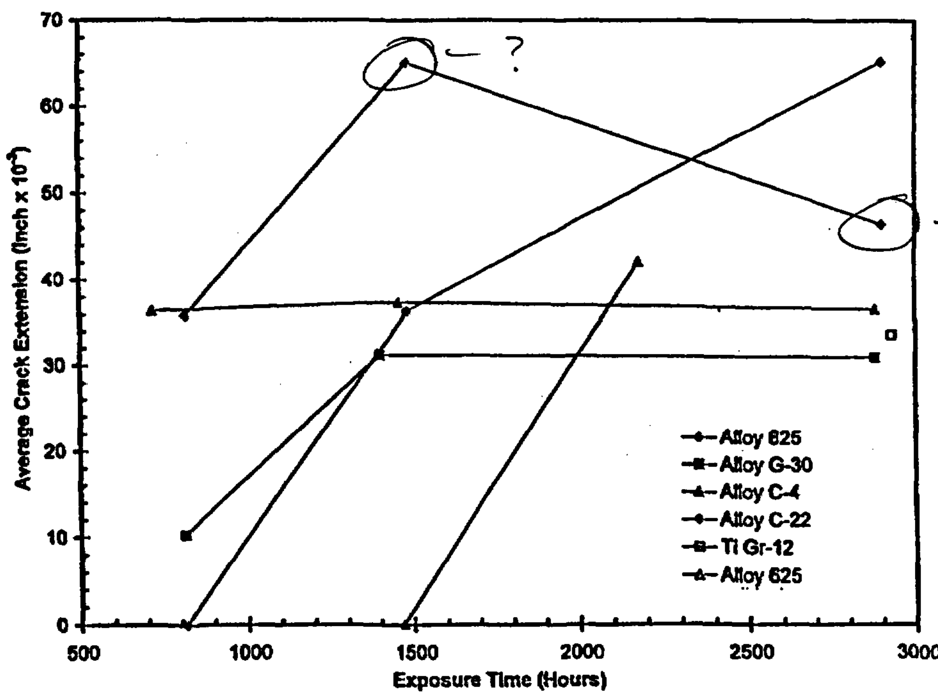


Figure 1 Wedge-Loaded DCB Specimen



? how can you have less cracking in longer duration test ?!

Figure 2 Average Crack Extension vs Exposure Time

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