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A.K. Roy, D.C. Freeman, B.Y. Lum, M.K. Spragge

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Lawrence
Livermore
National
Laboratory

SCC EVALUATION OF CANDIDATE CONTAINER ALLOYS BY DCB METHOD

Ajit K. Roy
Framatome Cogema Fuels
c/o LLNL (On Assignment)
7000 East Ave, M/S L-631
Livermore, CA 94550

Dennis C. Freeman
Beverly Y. Lum
Maura K. Spragge
Lawrence Livermore National Laboratory
7000 East Ave, M/S L-342
Livermore, CA 94550

ABSTRACT

The susceptibility to stress corrosion cracking (SCC) of two candidate container materials for the multi-barrier nuclear waste package was evaluated by using wedge-loaded and precracked double-cantilever-beam (DCB) specimens in a deaerated 90^{o}C acidic brine (pH ≈ 2.7). Materials tested include Alloy C-22 (UNS N06022) and Ti Grade-12 (UNS R53400). Duplicate samples of each material were loaded at different initial stress-intensity factor (K_I) values that ranged from 18 and 47 MPa√m. The DCB specimens were exposed to the test solution for one year. Both compliance and metallographic methods were used to determine the final crack length. The final stress intensity (K_f) for SCC was computed from the measured final wedge load and the average crack length. The results indicate that Alloy C-22 did not experience any cracking susceptibility at K_I values between 18 and 33 MPa√m. However, measurable cracking was observed with this alloy at a K_I value above 44 MPa√m. As to the SCC behavior of Ti Gr-12, cracks were observed at all K_I levels between 23 and 47 MPa√m. In general, Ti Gr-12 showed a higher susceptibility to cracking than Alloy C-22 in terms of the crack extension and the crack growth rate at comparable K_I values. Fractographic evaluation of broken DCB specimens by scanning electron microscopy (SEM) revealed three distinct regions showing the characteristics of fatigue precrack, SCC, and fast fracture.

Keywords: Stress corrosion cracking, double-cantilever-beam technique, nickel-base and titanium alloys, stress intensity factor, scanning electron microscopy.

INTRODUCTION

During the current suitability evaluation, the waste package design for spent nuclear fuel and defense high-level waste has focused on all-metallic, multi-barrier concepts to accommodate geologic disposal in the potential repository at Yucca Mountain, near Las Vegas, Nevada. This design incorporates a thin outer corrosion-resistant metal barrier over a thicker inner container made of a less corrosion-resistant alloy. The precise method of fabricating the waste packages is yet to be finalized. Regardless of the fabrication technique, however, some form of welding of the container's metallic materials will likely be involved in producing cylindrical packages of large diameters. The welding process could generate enough residual stresses to cause the waste package materials to become susceptible to SCC as they are placed in the potential repository environment. This paper presents the results of SCC tests of two candidate container alloys exposed to an aqueous environment using fracture-mechanics-based DCB method. The simulated conditions used in these tests are similar to some of the conditions expected in the potential repository.

MATERIALS AND EXPERIMENTAL PROCEDURE

Materials tested include nickel-chromium-molybdenum (Ni-Cr-Mo) Alloy C-22 and a titanium (Ti)-base alloy Ti Gr-12. Their chemical compositions are given in Table 1. The rectangular DCB specimens (10.16-cm long, 2.54-cm wide and 0.953-cm 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. No additional thermal treatments were given to these specimens prior to their being exposed to the test environment. The DCB specimens were machined so that the crack plane was perpendicular to the short transverse direction, thus ensuring that crack propagation would occur in the longitudinal rolling direction.

The DCB specimens were fatigue-precracked under load-control (load ratio of 0.10) at a frequency of 20 Hz using an Instron Servohydraulic testing machine with 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. To avoid exceeding the desired K_I value, the peak K value during precracking was maintained at 70% of the desired K_I value of the specimen.[1] 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. Wedges of three different thicknesses per material were used to load these DCB specimens to three K_I levels. Duplicate specimens of each alloy were tested at each K_I level. However, for Ti Gr-12, the results of a single specimen at each K_I level were analyzed.

The wedge-loaded specimens were completely immersed vertically in the test solution contained in a glass vessel and heated by a water bath. The environment used was a 90°C, continuously deaerated, acidic brine (pH ≈ 2.7) containing 5 weight percent sodium chloride (NaCl). Acidification was done by adding 0.055 milliliter of concentrated (95-98%) sulfuric acid per liter of salt solution. The acidified, concentrated salt solution was used to represent an extreme case in which microbiologically-influenced corrosion can occur as a result of microbial activity involving certain man-made materials (diesel fuels, organics, and sulfur-containing compounds) and water. These materials may be introduced into the potential repository during construction and operation, and may not be removed or be inadvertently left behind when operations cease. The acidic pH can also simulate some of the effects of radiolysis. The environment to be used in future SCC tests will be modified to reflect the saturated alkaline water chemistry.

Tests were performed for one year. At the conclusion of each test, the specimens were cleaned in acetone and deionized water. 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_f were computed using an equation derived by Heady.[2]

Fractographic evaluation of the broken DCB specimens was performed by SEM to characterize the morphology of fatigue precrack, environment-assisted failure, and fast fracture resulting from the separation of the two arms of the DCB specimens.

RESULTS AND DISCUSSION

The results, shown in Table 2, indicate that Alloy C-22 showed no measurable crack extension after being exposed to the test solution for one year and loaded at K_I values ranging from 18 to 33 MPa√m. However, the average K_f value was reduced by approximately 2.50 to 3.13 MPa√m corresponding to these K_I levels. As to the cracking susceptibility of this alloy at higher K_I levels (43 to 45 MPa√m), an average crack growth of 0.1714 mm was observed. The average crack growth rate (CGR) corresponding to this crack extension is approximately 3.26×10^{-7} mm/min, which is an order magnitude lower than the CGR for the same alloy tested in a similar environment for eight months.[3] However, it must be noted that the DCB technique of SCC evaluation constitutes a constant-crack-opening displacement phenomenon in which the load drops as the crack grows. The wedge-load measured in this K_I range during this current investigation, when the test was complete, was approximately 320 N lower than the initial wedge-load. The results[3] of a previous study performed at comparable K_I levels showed that due to this drop in wedge-load following a critical exposure period (4 to 5 months), the final crack could not grow any further, thus arresting the growth. It is possible that a similar crack-arrest phenomenon might have occurred in Alloy C-22 after some exposure period, which is difficult to determine in the present study. The lower CGR value obtained in the current investigation is the result of reduced crack extension and longer exposure time (one year versus one, two, four and eight months).

As to the crack growth behavior of Ti Gr-12, crack extensions of 0.1168, 0.1762 and 0.9034 mm were observed following 1-year exposure at three different K_I levels, as shown in Table 2. It is interesting to note that Ti Gr-12 exhibited SCC in a similar environment when loaded at K_I values ranging between 22 and 27 MPa√m.[3] The current results indicate that the crack growth was enhanced as the K_I value was gradually increased. However, the crack extensions in the present study are lower than those observed in the previous tests at comparable K_I levels.[3] In view of these lower crack extensions and the longer test duration, the corresponding CGR values are also lower. It should, however, be mentioned that the CGR corresponding to the highest applied stress intensity level (46.84 MPa√m) is very similar to that observed in the previous study at lower K_I level.[3]

Figure 2 shows an optical micrograph of a broken Alloy C-22 DCB specimen loaded to a K_I value of approximately 45 MPa√m. The extent of cracking resulting from the synergistic effect of the wedge-load and the acidic brine is illustrated in this figure, differentiating it from that of the fatigue-precrack initiated at the tip of the notch. The morphology of environment-induced cracking in both Alloy C-22 and Ti Gr-12 was further analyzed by SEM. Fractographic evaluation of test specimens of both alloys by SEM revealed three distinct regions showing the characteristics of fatigue failure, SCC, and tensile-overload fracture. The SEM micrograph of a broken Ti Gr-12 DCB specimen loaded at a K_I value of approximately 47 MPa√m is illustrated in Figure 3. Precracking of the specimen in air at the notched area by cyclic loading is characterized by striations. The fracture area immediately following the precracked region is possibly the result of transgranular brittle or quasi-cleavage failure that occurred in the wedge-loaded specimen due to its exposure to the 90°C acidic salt solution. Similar types of failure have been observed[3] in a previous study, and have also been reported elsewhere[4] for Ni-base and Ti alloys in a hydrogen-containing environment as well as in methanol. The tensile-overload or fast fracture of the specimen upon completion of testing and removal of wedge is characterized by dimpled rupture indicating ductile failure.

A comparison of the pH of the test environment before, during, and after testing indicates that for both alloys the pH changed very little and ranged between 2.56 and 2.86. Further, the amount of corrosion product was negligible.

SUMMARY AND CONCLUSIONS

Wedge-loaded and fatigue-precracked DCB specimens of Alloy C-22 and Ti Gr-12 were tested to evaluate their susceptibility to SCC in a 90°C acidic brine. The initial and the final stress intensity factors (K_I and K_f) were compared. The crack growth rates were determined based on the measured crack extension and the test duration. SEM was used to analyze the morphology of fracture in different regions of the broken DCB specimen. The significant conclusions drawn from this investigation are summarized below.

- Alloy C-22 exhibited cracking at the highest applied K_I level (43-45 MPa√m), confirming the observation made[3] in a previous study. But the extent of cracking and the CGR determined in the present investigation were much lower. No cracking was observed at lower K_I levels (18-33 MPa√m) tested.

- Ti Gr-12 showed cracking at all three K_I levels tested. The extent of cracking was enhanced at higher K_I levels, as expected. However, the crack growths were lower than those observed[3] in a previous study.

- Precrack due to cyclic loading at the notched area was characterized by striations. SCC may be the result of a transgranular brittle or quasi-cleavage failure. Dimpled rupture, a characteristic of ductile overload failure, was also observed in the SEM fractographs.

- The overall results suggest that both Alloy C-22 and Ti Gr-12 may become susceptible to SCC under the environmental condition tested in this program. However, this cracking susceptibility may be mitigated by relieving the residual stresses generated during the fabrication of the waste packages.

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TABLE 1
CHEMICAL COMPOSITION OF MATERIALS TESTED (WT%)

Material / Lot No.	C	Mn	P	S	Si	Ni	Cr	Mo	Fe	Ti	Al	Cu	Other
Alloy C-22 / M924	0.003	0.260	0.011	0.002	0.023	Bal	21.570	13.390	5.170	---	---	---	W: 2.900 Co: 0.370 V: 0.160
Ti Gr-12 / M896	0.006	---	---	---	---	0.650	---	0.280	0.100	Bal	---	---	N: 0.006 H: 0.0017 O: 0.160

TABLE 2
SCC TEST RESULTS

Material / Lot No.	K_I (MPa√m)	K_f (MPa√m)	Crack Extension (mm)	Duration (h)	CGR (mm/min)
Alloy C-22 / M924	18.899, 18.847 *	16.584, 16.169 *	None	8757.50	None
	32.632, 32.197 *	30.083, 28.486 *	None	Same	None
	43.275, 44.638 *	38.684, 40.430 *	0.1714 +	Same	3.26×10^{-7}
Ti Gr-12 / M896	23.610	23.046	0.1168	8853.50	2.2×10^{-7}
	36.841	33.510	0.1762	Same	3.32×10^{-7}
	46.838	36.919	0.9034	Same	1.7×10^{-6}

* Measured on duplicate specimens
+ Average value

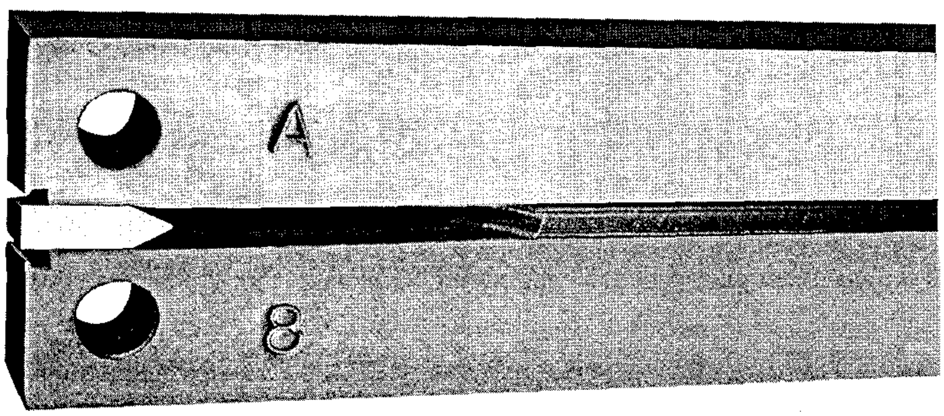

FIGURE 1 - Wedge-Loaded DCB Specimen

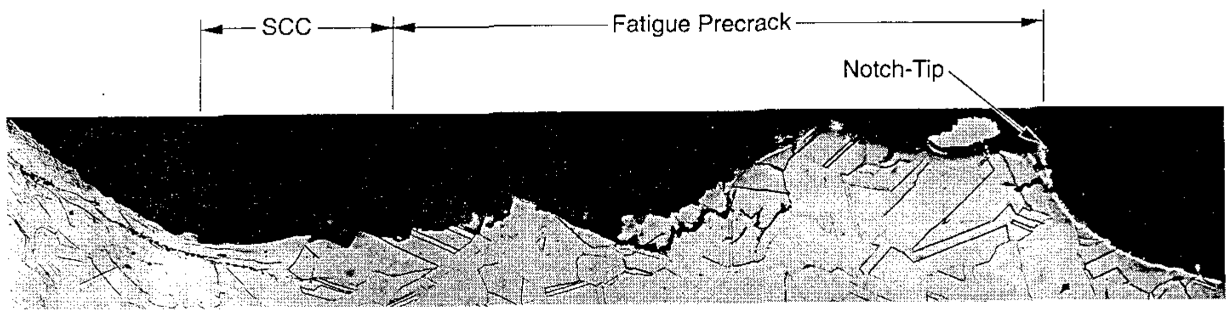

FIGURE 2 - Optical Micrograph for Alloy C-22 DCB Specimen ($K_I \approx$ 45 MPa$\sqrt{m}$). Polished and Etched. 100X

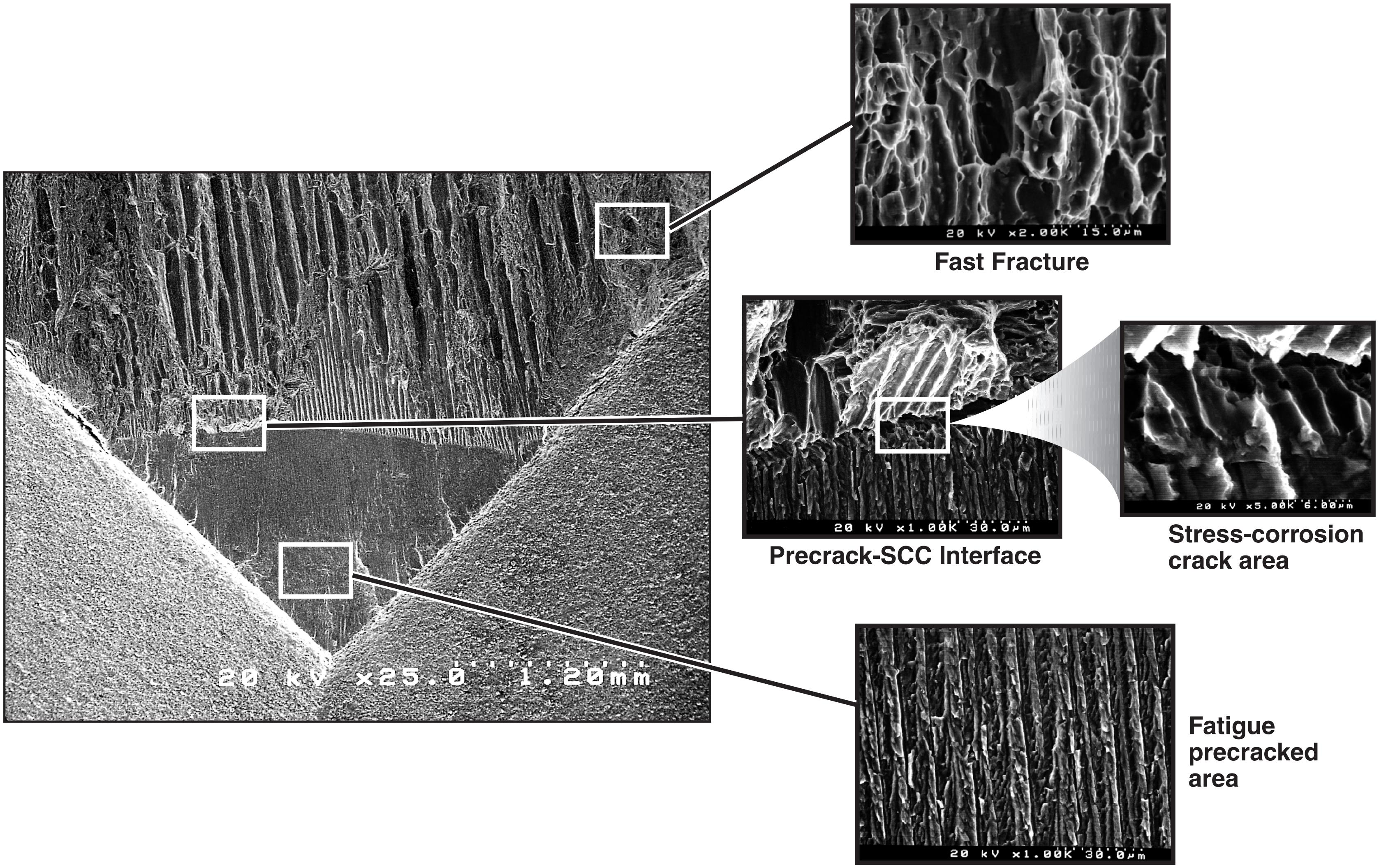

FIGURE 3 - SEM Micrograph of Ti Gr-12 DCB Specimen ($K_I \simeq$ 47 MPa÷m).