

Corrosion Fatigue of High-Strength Aircraft Structural Alloys

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Results of corrosion fatigue characterization of AA7075-T6 and AF1410 steel under different simulated marine environments and loading conditions are presented. In comparison with baseline tests conducted in laboratory air, corrosion fatigue experiments performed at 1-Hz frequency in the presence of 1% NaCl environment indicated a substantial reduction in fatigue lifetime in the case of AA7075-T6, whereas AF1410 corrosion fatigue life was found to be statistically unaffected at 1-Hz frequency in the presence of 1% and 3.5% NaCl. However, a reduction of frequency to 0.5 Hz significantly reduced the lifetime of AF1410 steel. On the other hand, Cd-plated AF1410 tested to study fatigue characteristics in a hydrogen-rich metal surface environment yielded minimal change in the lifetime. Atomic force microscope analysis was performed to discern features in fracture surface morphology leading to changes in lifetime of AF1410 and AA7075-T6 alloys.

I. Introduction

ENVIRONMENTAL effects (corrosion and hydrogen) can pose a serious threat to mission readiness and reliability inasmuch as the large inventory of the aging U.S. Navy fleet (>4600 aircraft) can spend several months in the midst of highly corrosive marine environment. Aircraft components operating under extremely high flight loads, often approaching design limits, are exposed to a multitude of corrosive agents, such as sea water, chlorides, and a variety of other exhaust gases that are practically impossible to keep out of structural joints and surface imperfections. It is commonly known that the synergistic combination of mechanical fatigue stresses and environmental agents acting together is more detrimental than that of either one acting separately.¹ Quantitative fatigue life predictions are often not possible because of the numerous interacting factors that influence environmental fatigue behavior and the resulting lack of significant data. Sources of complexity are various, for example, the bulk and crack-tip environmental conditions are seldom the same quantitatively and that the kinetics of the (electrochemical) reactions occurring at the crack tip can be immensely accelerated under a given stress, pH level, salt content, and rate of loading frequency in some high strength alloys, whereas the same conditions may not have any significant effect on other types of alloys.^{2–7} To complicate matters further, there is serious lack of experimental techniques that can yield quantifiable electrochemical information, such as corrosion current density, hydrogen generation and absorption, and microcracking, etc., at the tip of the growing crack.^{1,8} Therefore, understanding the interaction among the effects of a material's microstructure, chemical composition of the environment, and thermal and mechanical loading conditions, especially at the crack tip, becomes an enormous task. This leads to significant uncertainty in fatigue lifetime prediction, and therefore, no existing model can adequately address the U.S. Navy's concerns about aircraft structural integrity.^{1,8} Despite these difficulties, developing a fair understanding of the effects of corrosion to obtain reasonably good data that can be applied to aging aircraft is an important element in more accurately estimating aircraft's fatigue life.

High strength aluminum AA7075-T6 (strength ~537 MPa and toughness ~27 MPa · m^{1/2}) has been widely used in aircraft structural longeron and frame elements. Therefore, it is important to improve understanding of various agents contributing to corrosion fatigue characteristics of this material.⁹ AF1410 steel, on the other hand, with ultra high strength (~1.8 GPa) and toughness (~130 MPa · m^{1/2}) is a very attractive aircraft structural material (currently used mostly in arresting shanks of some U.S. Navy F-18s) with almost no literature available related to its corrosion fatigue crack growth (CFCG) properties in aggressive marine environments.¹⁰ Therefore, results from a corrosion fatigue study of two promising aircraft structural alloys, namely, AA7075-T6 and AF1410, conducted in the presence of simulated marine environment, and of hydrogen-assisted fatigue in AF1410 steel are presented and discussed.

II. Experimental Setup

A. Test Parameters

It has been suggested that in the presence of chloride ions the pH of crack tip can be acidic even when the bulk solution is almost neutral.^{7,11} This occurs due to the hydrolysis of corroding species, such as aluminum and steel. Because the pH of the naval (service) environment can vary anywhere between 3 and 5, mild 1% and 3.5% NaCl of pH ~2.5 (simulating crack-tip pH) electrolyte was selected for fatigue testing. AA7075-T6 was tested in 1% NaCl solution at a frequency of 1 Hz, and AF1410 was tested in 1% NaCl and 3.5% NaCl at 1 and 0.5 Hz frequencies at a constant R (=0.4). Additionally, because diffusion of hydrogen in steel is a very slow process ($D < 10^{-9}$ cm²/s), aircraft parts in service do not, in general, experience a hydrogen flux through the thickness. Significant hydrogen concentration, therefore, remains in the vicinity of the metal surface, where it manifests localized embrittlement that leads to subsequent crack initiation and growth under stress. Therefore, as a first step to understand the role of atomic hydrogen on fatigue, bright 1-mil Cd-plated AF1410 specimens were tested at 1- and 0.5-Hz frequency. The process of Cd electroplating deposited an estimated concentration of atomic hydrogen of 11, 2, and 300 ppm on the surface of plated samples, in steel only and in cadmium plate only, respectively.¹²

Four specimens each were tested under a given loading and environmental condition. All testing was conducted at room temperature on compact tension specimens of dimensions $5 \times 5 \times 0.64$ cm³ and $5 \times 4 \times 0.32$ cm³ AA7075-T6 and AF1410, respectively. Preliminary fatigue testing was performed in laboratory air at 1 Hz to establish a baseline. All specimens were precracked according to the American Society for Testing and Materials ASTM E399-95 standard. Quasi-static tests performed at 8.4 N/s yielded average ultimate static strength of 17 and 39 kN for AA7075-T6 and AF1410, respectively. Fatigue testing was performed at crack

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opening displacement (COD) values that corresponded to initial $k = 5.6 \text{ kN} \cdot \text{m}^{1/2}$ for AA7075-T6 and $15.2 \text{ kN} \cdot \text{m}^{1/2}$ for AF1410.

B. Load Decrementing Testing

Fatigue crack nucleation generally occurs from plastic straining in localized regions. As a result, strain-controlled tests can better characterize the fatigue behavior of a material than load-controlled tests, especially in the notched specimens where significant localized plastic deformation is frequently present. Load-decrementing testing (LDT) is a strain-controlled fatigue mechanism in which the COD (maximum and minimum) values are kept constant while the load drops in response to lowering stress intensity at the tip of growing crack. LDT is an excellent technique in conducting comparative fatigue crack growth studies, and it has several advantages over the load-controlled fatigue testing, such as multiple but complete lifetimes can be obtained from a single specimen, testing time is greatly reduced, and greater control can be exercised in obtaining stable crack growth (corresponding to region 2 in the conventional load-control testing). The LDT technique was, therefore, currently employed to study fatigue characteristics. After sufficient preliminary testing and based on the constraints imposed by the initial k values used, two complete lifetimes were obtained from each specimen.

C. Crack Length Measurement

Special care was exercised in measuring crack length, which is prone to error, depending on the method used. Crack length was, therefore, calculated using compliance technique based on Eq. (1),¹³ and stress analysis based on Eq. (3)¹⁴ and via visual inspection. Compliance-based crack length is given as

$$a = W \left[1.0010 - 4.6695U_X + 18.46U_X^2 - 236.82U_X^3 + 1214.90U_X^4 - 2143.60U_X^5 \right] \quad (1)$$

where a is the crack length, W is the depth of the specimen, and

$$U_X = 1 / \left[(tEV_X/P)^{0.5} + 1 \right] \quad (2)$$

where t , E , V_X , and P are the specimen thickness, Young's modulus, COD, and load, respectively. Based on stress analysis, crack length was obtained from the real and positive root of the following equation:

$$35.6(1 - a/W)^3 + [V_X Et/P - 52.91](1 - a/W)^2 + 30.13(1 - a/W) - 19.75 = 0 \quad (3)$$

Crack lengths from these three techniques were virtually the same, as shown in Fig. 1 for typical AA7075-T6 and AF1410 specimens.

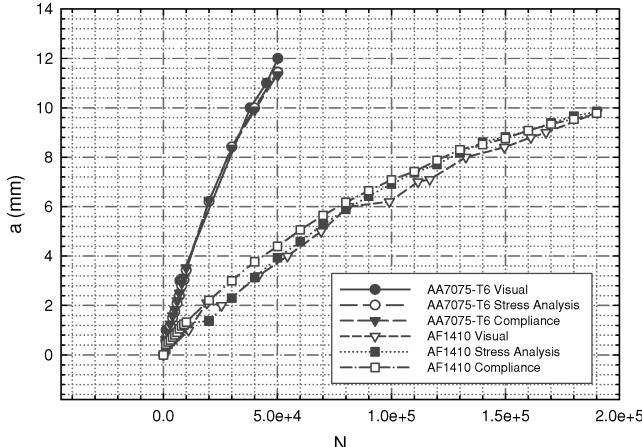


Fig. 1 Crack length obtained from compliance, stress analysis, and visual inspection.

D. Atomic Force Microscope Analysis

Because microscopic observation is a requisite to understand the mechanisms of fatigue processes in materials, the atomic force microscope (AFM) was utilized to study fracture surface morphology. AFM has been widely used in the study of microcrack nucleation and growth, striation shapes, and slip bands and to extract general failure features of a variety of materials.^{15,16} While interpreting current AFM images, caution must be exercised because the system software automatically sets the maximum depth (z range) based on the highest point on the given surface morphology under observation. Therefore, two images taken on adjacent sites of the sample may look visually incomparable because the depth perception would be different. Similarly, the z range (depth) of the fracture surface of two otherwise identical specimens inspected at the same x and y coordinates can look different as dictated by the AFM operating software.

III. Experimental Results

A. AA7075-T6

Initial tests conducted in the presence of electrolyte indicated that the second lifetime obtained from the same specimen was consistently lower than the first lifetime, as shown for a typical specimen in Fig. 2. It was an interesting observation because it suggested that the salt-laden electrolyte was being diffused into the corrosion process zone ahead of crack tip even at a relatively high test frequency of 1 Hz. The implications were that the electrochemical processes were not limited to the crack tip and that the crack may be propagating into a region already altered by the corrosion process. Therefore, as a material fatigues, the corrosion process zone can be significant in CFCG analysis and modeling. However, to obtain identical initial conditions for each test cycle, specimens were precracked intermittently in laboratory air to slightly over the estimated corrosion process zone size according to ASTM E399-95 standard from one life cycle to the next in subsequent tests. Figure 2 shows that by doing thus, better correlation and less spread in the data (Fig. 3) was obtained in the lifetimes of each test cycle.

Test results indicated that the presence of a mildly aggressive marine environment, even at subcritical fatigue stresses, can significantly raise CFCG rates and impose its detrimental effects on AA7075-T6 aircraft structural components. When error bars are used for clarity, average results given in Fig. 3 show up to four times increase in the CFCG for all specimens tested in the presence of electrolyte as compared to those tested in laboratory air. The results matched well with published literature.^{9,17}

B. AF1410 Steel

When AF1410 specimens were subjected to 1% and 3.5% NaCl solution at 1-Hz frequency, statistically insignificant change in the CFCG rate was observed as compared to the tests conducted in laboratory air. However, reducing the frequency to 0.5 Hz significantly increased the CFCG at both 1% and 3.5% NaCl electrolyte,

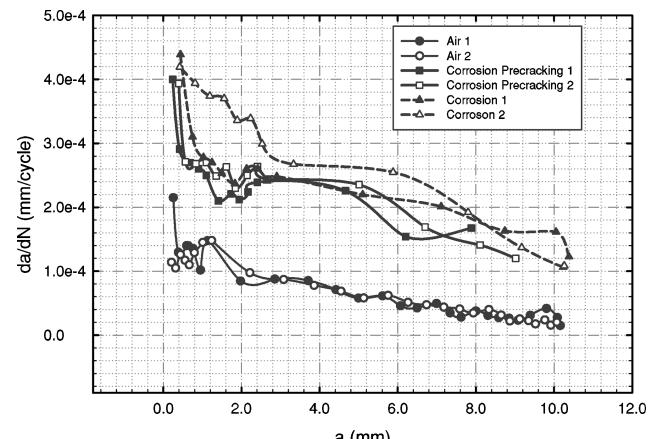


Fig. 2 Typical results of CFCG lifetime for AA7075-T6 specimens.

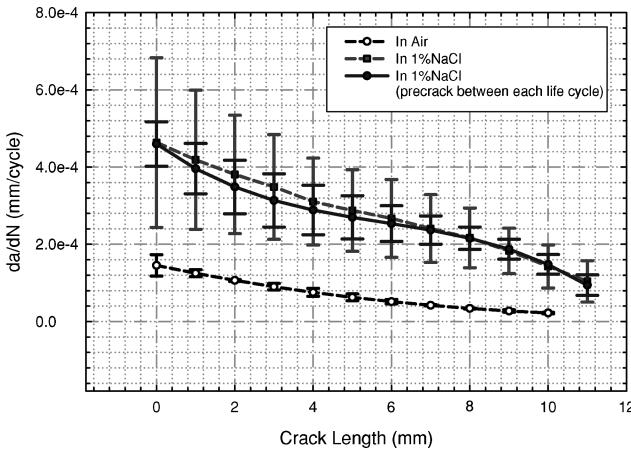


Fig. 3 Average results of CFCG for AA7075-T6 specimens.

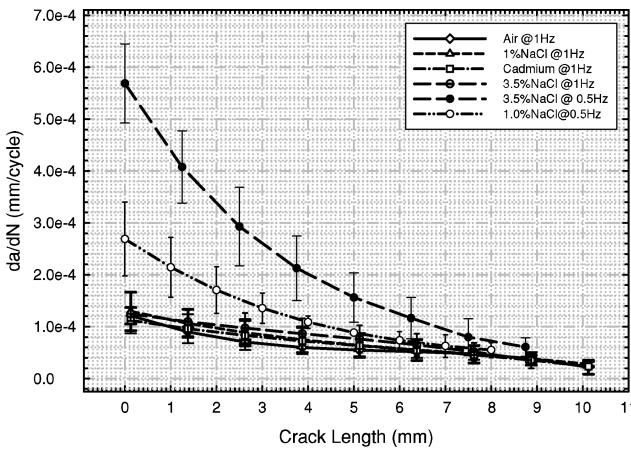


Fig. 4 Average results of CFCG for AF1410 specimens.

as shown in Fig. 4 (with error bar graph for clarity). It is obvious that the reduction in lifetime at 0.5-Hz frequency is greater in 3.5% NaCl solution compared to 1% NaCl. These observations were not in complete agreement with the sole literature.¹⁰

To understand the role of atomic hydrogen concentration near the metal surface, 1-mil thickness bright Cd-plated AF1410 specimens were tested. Figure 4 indicates virtually no change in the crack growth rate (CGR) as compared to the tests conducted in laboratory air. The expectation was that the hydrogen from the Cd plate (exceeding 300 ppm) would penetrate the metal surface at an accelerating rate under fatigue stresses and migrate toward the crack tip.^{6,12} However, there is no evidence to suggest that such hydrogen ingress into the metal surface took place in significant amounts. The inconsequential effect on the lifetime by Cd plating is due to the minute Cd film thickness (~ 0.0254 mm) when it is compared to the sample thickness, ~ 4 mm. Also, H diffusivity into the steel for a thickness of 0.30 mm was low: $D = 1.53 \times 10^{-8} \text{ cm}^2/\text{s}$; thus, obviously, the diffusivity for the thicker Cd-plated specimen should be lower. In general, as the sample thickness increases, the diffusivity decreases and the H solubility rises. A larger thickness favors generating potential trapping sites for hydrogen, which reduce the hydrogen diffusivity into the material.

IV. Discussion

The observed increase in CFCG of AA7075-T6 and AF1410 can be explained in terms of various electrochemical/micromechanical processes simultaneously taking place at the crack tip, such as, metal dissolution (MD), hydrogen embrittlement (HE), crack-tip microcrazing, frequency of loading and ionic concentration, etc.^{1,6-10,17}

AF1410 results shown in Fig. 4 indicated a significant change in the CFCG rate as the NaCl concentration increased but only at a rel-

atively low frequency of 0.5 Hz. However, the literature on AF1410 and AA7075-T6 provides a substantial reduction in lifetime at up to 10-Hz frequency and as the percentage of NaCl is increased.^{9,10} Rate of loading frequency can have profound effect on the crack-tip chemistry; however, its effect on CGR is not quite clear because a complex interaction of various factors are involved in the latter.⁷ For example, higher frequencies can cause an increase in the extent of charging, whereas reducing the frequency can cause a decrease in the dissolution rate at the crack tip. However, reducing frequency also causes crack-tip pH to decrease as a result of increasing dissolution time.

The process of MD plays a major role in the observed enhancement of CFCG (as indicated in Fig. 4) and depends highly on the NaCl concentration in the aqueous solution. Crack propagation by MD involves ionic diffusion (of aggressive anions such as Cl^-) down the crack length, the rupture of the protective oxide film, followed by dissolution of the bare metal surface (especially under stress) due to localized metal loss. Hence, an increase in the percentage (amount) of NaCl leads to higher concentration of chloride ions with antecedent deleterious effects. Based on electrochemical considerations, presence of Fe^{2+} , OH^- , H^+ , Cl^- , etc., implies that there will be reactions preferences based on relative anodic/cathodic tendencies. For instance, $\text{Fe}^{2+} + 2\text{e}^- = \text{Fe}$ will be anodic with respect to $\text{Fe}^{3+} + \text{e}^- = \text{Fe}^{2+}$, etc. Therefore, the presence of chloride ions will lead to the development of multiple and/or parallel sources of enhanced CFCG due to the multivalence nature of Fe. The possibility of forming varying transient and stable compounds with Fe suggests that increased Cl^- concentration will ultimately translate to accelerated CFCG rate.

Effect of NaCl concentration on CFCG can also be deduced from Faraday's law given as (see Ref. 1)

$$\frac{dx}{dt_{\text{MD}}} = \frac{Mi_a}{z\beta F\rho t^*} [1 - \exp(\beta t^*)] \quad (4)$$

where M is the atomic weight, i_a is the current density, ρ is the density of the metal, F is the Faraday's constant, z is the number of electrons involved in the overall oxidation, β is the passivation rate parameter, and t^* is the periodicity of oxide rupture events. Because Cl^- diffuses preferentially toward the crack tip, a higher concentration of NaCl will enhance crack-tip current density i_a and subsequently promote MD ($\text{Fe} = \text{Fe}^{2+} + 2\text{e}^-$), and, hence, enhanced CFCG.

Furthermore, when a material is under stress, the generation and accompanying nonuniformity distribution of dislocation densities, dislocation networks, stacking faults, etc., cause certain regions of a homogeneous material to be anodic (such as the crack tip, which is smallest in area and highest in stress) with respect to certain other locations. This means that with stress fields leading to nonuniformity of electron distribution there is possibility of MD with just H_2O . However, when NaCl concentration increases in the aqueous solution, Cl^- will exacerbate the anodic/cathodic reaction and preferentially be attracted to the crack tip through combined diffusion and electrolysis. This process will weaken Fe (or materials atomic) bonds and serve to enhance local stress intensity and, thus, CFCG rate.

Therefore, MD as a function of loading frequency, pH and NaCl content, etc., plays a significant role in the observed experimental results. However, there is considerable controversy associated with known MD-based models primarily because the current flowing at the crack tip can not be isolated from processes occurring in the area adjoining it and it is nearly impossible to quantify i_a accurately at the tip of the growing crack, which must be known for CFCG calculations in Eq. (4). As a result, accurate quantification of lifetime remains a challenge.^{1,8}

Changes in lifetime observed in Figs. 3 and 4 can also be explained in terms of hydrogen effect. In the presence of aqueous solution, environmental HE results from generation of hydrogen on the metal surface as a partial reaction in the electrochemical (corrosion) process. Dissolving NaCl in H_2O causes the H^+ and OH^- ions to be "freed" more than otherwise and causes the dissociation of Na^+ and

Cl^- ions such that overall conduction activity is increased. Whereas Cl^- activity takes place in the MD process, H^+ simultaneously gets dissolved as well in the material, thereby weakening the material through reduction in bond strength. H^+ is much smaller than Cl^- and, therefore, diffuses faster than Cl^- . Some of it forms HCl with Cl^- ($\text{H}^+ + \text{Cl}^- = \text{HCl}$), which causes further electrolysis. One of the likely sources of e^- (electrons needed) for this reaction comes from the electronic charge cloud asymmetry due to crack-tip stress fields. The H^+ and OH^- produced subsequently react and/or get absorbed into the metal surface to form a brittle oxide film or a hydride phase or capture and move with the vacancies.^{1,18,19} Environmental hydrogen diffusion generally tends to regions of high triaxial tensile stress, such as, the crack tip, where the material undergoes dilation. This process is particularly conducive with lower loading frequencies as the diffusion time increases. Brittle crack growth occurs as the film is ruptured and/or crack grows into brittle hydride phase by plastic strain. Combination of stress and electrolytic medium, such as aqueous NaCl , increases chances of H generation, increased mobility of Cl^- , weakening of atomic bonds (faster at the crack tip), and overall increase in the CFCG.

Hydrogen-based models have to account for the crack-tip kinetics of generation of H atoms and their absorption, the transport and distribution of H atoms in the lattice and microstructural trap sites, and knowledge of transient currents. Once again, it is a daunting task experimentally to quantify these variables at the tip of the growing crack. As a result, HE models, except in certain specific cases, also match poorly with the actual service lifetime of a metallic component.^{1,17-20}

To discern crack growth features and understand the role of MD and HE, the surface morphology of cracked specimens was studied using the AFM technique. The fracture surface morphology under HE and/or due to MD is well established.^{1,15,16,21} AFM analysis of cracked AA7075-T6 specimen surfaces, presented in Fig. 5, indicated MD to be the predominant corrosion process leading to lifetime reduction in specimens exposed to electrolyte. In all of these AFM images, the direction of crack growth is northeast/southwest, that is, along a diagonal at 45 deg with respect to x and y axis.

The specimens tested in laboratory air showed a typical rough surface (with roughness average $R_a = 1.02 \mu\text{m}$ taken at 9 mm from the crack tip as shown in Fig. 5) caused by grain pullout and intragranular failure, whereas the specimens tested in the presence of electrolyte exhibited intergranular failure reflected by smoother cracked surface (with $R_a = 0.905 \mu\text{m}$) along with pits and crevices, a commonly observed feature of MD-induced corroded surface, as

shown in Fig. 5 for a typical specimen. As can be seen for the Al 7075 T6 alloy, although roughness average was smaller for specimens exposed to saline solution (1% NaCl) compared to the specimen tested in air, this behavior could be associated to the presence of the MD that covers partially the features in the fracture surface. Therefore, deposits of corrosion residues hinder a conclusive interpretation of the fracture surface. In Fig. 5, the specimen tested in air shows the presence of mixed fracture mode, that is, brittle and ductile. An indicator of brittle fracture mode is the presence of ripples. The ductile behavior is revealed by the typical grooves and the presence of microvoids or dimples. In general, this aluminum alloy (Al-7075-T6) tended to behave more in a brittle manner than in a ductile mode. With the 1% NaCl solution, the river pattern lines were generally coarser due to the presence of the MD. The fatigue features formed in that saline solution had a brittle appearance. The reduction in the fatigue life of the specimens tested in the 1% NaCl solution was around one-half of the lifetime of the specimen tested in air. This was also connected to the fracture appearance (more brittle). It is conjectured that the mechanism responsible for the lifetime decrease is the presence of the acid solution that induces the embrittlement by hydrogen evolution. The embrittlement effect is enhanced by the cyclic mechanical rupture (pumping effect of the solution into the crack tip under cyclic loading) of the passive film and the subsequent lack of metal repassivation due to chloride ions.

Figure 6 shows AFM images of a typical AF1410 specimen at 30- μm resolution of fracture surface morphology obtained at distances of 2 and 5 mm from the precrack under various testing conditions. The reported z range of the images corresponds to the maximum height measured at the mapped region on each individual image. Crack growth direction is parallel to x axis from left to right. The analysis indicated an increase in fracture surface roughness as the frequency or NaCl concentration increased. The largest roughness values were observed at 2 mm (larger values of da/dN , mm/cycle) from the precrack tip on all specimens. Therefore, at larger crack lengths, the roughness average tends to decrease as the stress intensity decreases.²¹ AFM analysis of Cd-plated specimens (Fig. 6) showed fracture surface features that were devoid of any evidence of hydride formation or brittle failure. Crack propagation in 1% and 3.5% NaCl exhibited crack-arrest marks on the fracture surface that resemble brittle fatigue striations, which are common in materials under aqueous environments such as the used saline solution (acidic environments).^{21,22} At 2 mm from the precrack tip, a mixed fracture mode (brittle and ductile) was observed. Generally these features occur during beginning of stage 2 fatigue (under typical

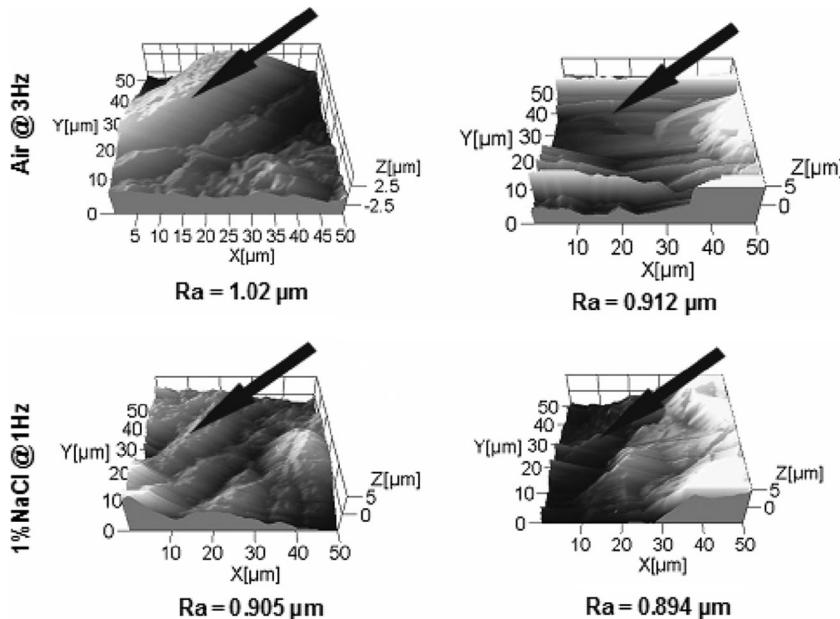


Fig. 5 AFM images of AA7075-T6 fractured surfaces obtained from specimens tested 9-mm distance from precrack tip (left) and 13-mm distance from precrack tip (right) under various conditions: arrow indicates direction of crack growth (northeast to southwest).

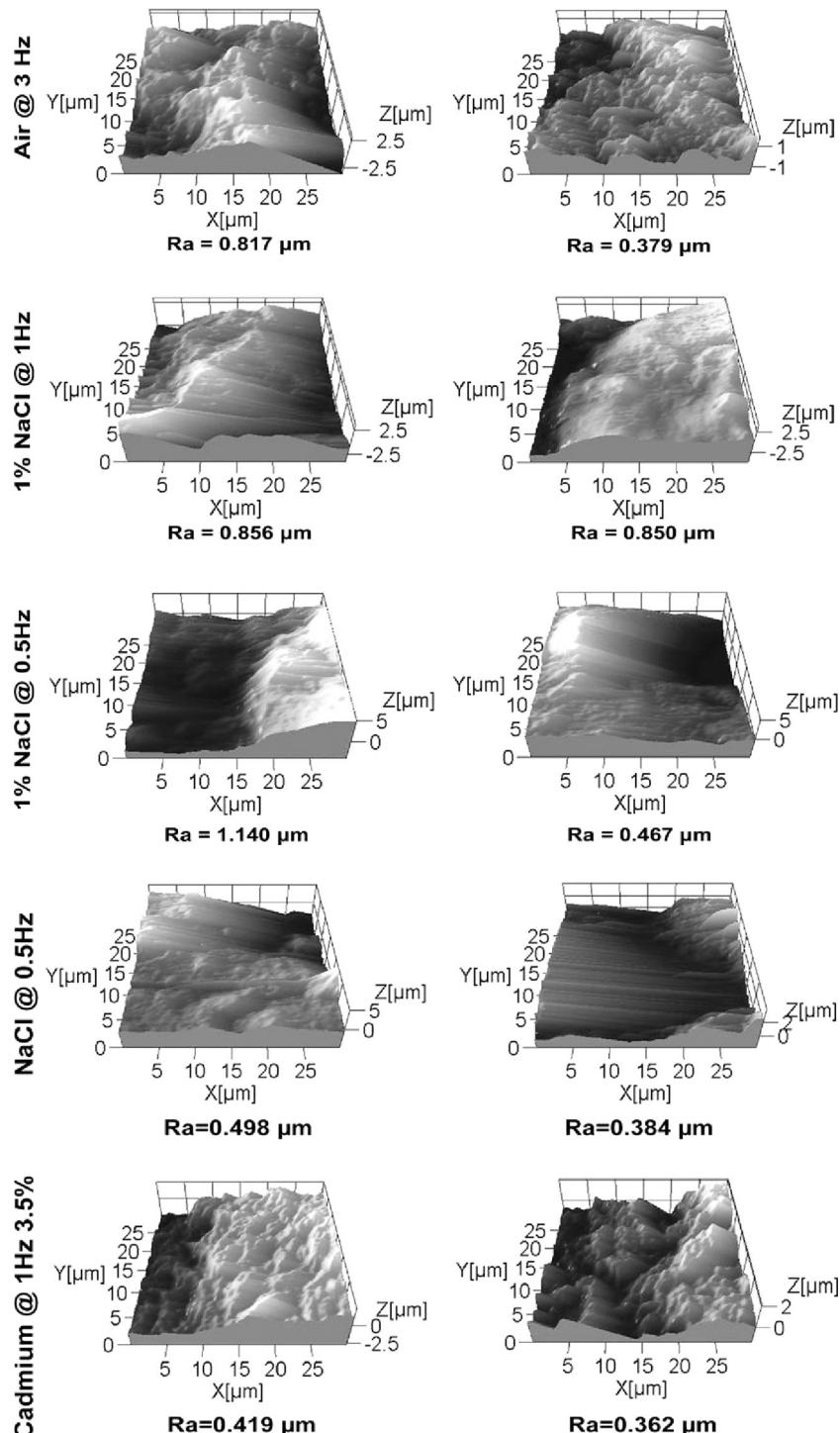


Fig. 6 AFM images of AF 1410 fractured surfaces obtained from specimens tested 2-mm distance from precrack tip (left) and 6-mm distance from precrack tip (right) under various conditions.

load-control conditions). Further analysis must be performed to determine the striation height and width and the correlation to the failure mode developed under saline environments. In theory, each striation should have been the result of one load cycle and marks the position of the fatigue crack front at the time the striation was formed. Also these striations should have been accompanied by the presence of slip band or shear bands. Shear bands can also be observed in Figs. 5 and 6, but striations were not as apparent. Because the opening and blunting process of a crack tip has to be operated to form a striation, as reported in the literature,²¹ the correlation between the striation width and height are directly proportional to crack growth rate (da/dN , mm/cycle).

AFM evidence collected in the current case reveals features related to the presence of HE, such as straight lines (striations and shear bands). Note that the AF1410 steel does not have alloy elements in the Group VB of the periodic table, which are the ones susceptible to form hydrides. Therefore, the brittle nature of the fracture cannot be attributed to the formation of hydrides. Remember that there is not substantial hydrogen flux through the metal in the presence of the electrolyte and that the hydrogen produced permeates a very short distance (perhaps $< 10^{-3}$ cm) into the metal; therefore, its features are very susceptible to MD-induced open-circuit potential. Therefore, controlled bulk charging experiments generating known values of hydrogen are needed to characterize properly

CFCG behavior of high-strength steels. This effort is currently underway.

V. Conclusions

Corrosion fatigue testing of AA7075-T6 and AF1410 steel was performed under a simulated marine environment. Results indicate an enhancement of CFCG in AA7075-T6 in the presence of 1% NaCl electrolyte, whereas AF1410 indicated an increase in CFCG at 1% and 3.5% NaCl only as frequency of loading was decreased. Lifetime in AF1410 also reduced as a function of increasing NaCl concentration at low testing frequencies (0.5 Hz). Furthermore, AF1410 specimens indicated no change in the CFCG as a result of Cd plating. AFM analysis was used to discern fracture morphology features. From an electrochemical perspective, both MD and HE were deemed likely sources of enhanced CFCG as a function of increasing NaCl concentration and decreasing frequency of loading. AFM analysis also pointed to (brittle and ductile) mixed-mode failure with dominant brittle features, thus, effects of aqueous hydrogen.

Acknowledgments

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