DETERMINATION OF THE CORROSIVE CONDITIONS PRESENT WITHIN AIRCRAFT LAP-SPLICE JOINTS

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ABSTRACT

The complexity of airframe structure lends itself to damage resulting from crevice corrosion. Fuselage lap-splice joints are a particularly important structural detail in this regard because of the difficulty associated with detection and measurement of corrosion in these occluded regions. The objective of this work is to develop a laboratory corrosion test protocol to identify the chemistry to which lap joints are exposed and to develop a model of the corrosion within the joints.

A protocol for collecting and identifying the chemistry of airframe crevice corrosion has been developed. Capillary electrophoresis (CE) is used to identify the ionic species contained in corrosion product samples removed from fuselage lap splice joints. CE analysis has been performed on over sixty corrosion product samples removed from both civilian and military aircraft. Over twenty different ions have been detected. Measurements of pH of wetted corroded surfaces indicated an alkaline occluded solution. After determining the species present and their relative concentrations, the resultant solution was reproduced in bulk and electrochemical tests were performed to determine the corrosion rate. Electrochemical analyses of the behavior of AA2024-T3 in these solutions gave corrosion rates of up to 250 microns per year (10 mpy). Additional tests have determined the relative importance of each of the detected ions in model solutions used for future predictive tests. The statistically significant ions have been used to create a second generation solution.

Laboratory studies have also included exposure tests involving artificial lap joints exposed to various simulated bulk and crevice environments. The extent and morphology of the attack in artificial lap joints has been compared to studies of corroded samples from actual aircraft. Other effects, such as temperature and potential, as well as the impact of the environment on fatigue crack growth have also been studied.

1. INTRODUCTION

As the fleet of both commercial and military aircraft ages, damage caused by corrosion becomes a more pressing concern. The KC-135 is one aircraft for which life extension is an important issue. Built in the mid-1950s, these aircraft are now expected to continue flying until the year 2040. In 1990, Groner[1] estimated that corrosion maintenance was costing $185,000 per aircraft per year. With a fleet of over six hundred KC-135s still flying, this reaches over $110 million per year in repair costs. However, this is not

only a high cost issue, but also leads to a decrease in aircraft readiness because of the length of time aircraft spend in depots for repair and a possible loss of safety if the damage becomes severe.

Lap-splice joints are the regions of overlap on the fuselage of an aircraft where two pieces of aircraft skin are riveted together. This arrangement forms an occluded region in which detection of corrosion is especially difficult. Although much work is underway to improve non-destructive inspection, more information about the corrosion process is necessary to aid in setting reasonable inspection intervals which are currently based on calendar time. The current mentality when dealing with aircraft corrosion is "find it - fix it". This idea means that any time corrosion is found, it is either ground out or, if too severe, the panel of skin is removed or reinforced. Grinding out corrosion leads to concern about the decrease of skin thickness and resulting loss of strength. Although Scheuring[2] has shown that failure due to fatigue does not decrease if the corrosion is polished off of a sample, if the corrosion is not detected, Scheuring determined that the skins would fail in one-quarter of the expected number of cycles.

Most research addressing strength concerns is performed in 0.6 M sodium chloride solution. This research attempts to determine if this is a relevant environment for the testing of lap splice joints. In order to investigate the actual chemical environment present within these joints, corrosion product samples were dissolved and analyzed with capillary electrophoresis (CE). CE is a technique used extensively by the biological sciences and analytical chemists. There are several advantages to this technique. It can be used to analyze very small (< 30 μL) solution samples and can detect ions in concentrations as small as a few ppb. Depending on the electrolyte and run parameters chosen, CE can detect anions or cations. It is capable of both qualitative and quantitative analysis.

Development of a more relevant model environment aided in the determination of boundary conditions for future lifetime prediction models. Performing electrochemical and mechanical tests in the more relevant environment improved predictive ability. This work determined the chemical environment, the pH, the electrochemical environment, and the corrosion morphology found within aircraft lap-splice joints.

2. EXPERIMENTAL TECHNIQUES

2.1 SAMPLING METHODS

Two primary methods were used to obtain samples from aircraft for solution analysis. The scraping method was the field sampling method. When aircraft were undergoing repairs at an Air Logistics Center (ALC) or commercial aircraft refurbisher, small amounts (< 0.5 g) of corrosion product were removed from recently opened lap-splice joints. The product was removed by gently scraping it off using a new rubber policeman on a glass rod and it was collected in a clean Petri dish. It was important throughout this process that contamination was avoided because of the sensitive nature of the analysis technique. Upon arrival at the University of Virginia, the product was soaked in two milliliters of high purity water for twenty-four hours. The solution was then removed, filtered to remove any insoluble particles, and transferred to vials for ion analysis.

The second sampling method was a laboratory procedure which will be referred to as the microsampling method. Unopened aircraft doublers were sent to the University of Virginia where they were opened and exposed to a high humidity environment for twenty-four hours. Small amounts of solution (less than 5 μL) were removed using a microsyringe. These solutions were then diluted with 30 to 50 microliters of high purity water. This method allowed a better estimation of the dilution of the sample and easier control of contamination, but unopened aircraft skins are difficult to obtain.

2.2 ION ANALYSIS

Both sampling methods produced solutions that were analyzed with a Waters Quanta 4000 capillary electrophoresis system connected to a computer with Millennium 2.10 software. Capillaries were sixty centimeter long tubes of fused silica with an inner diameter of seventy-five micrometers and an effective length (from sample to detector) of fifty-two centimeters. Each capillary was prepared by purging for two minutes with 0.1 M KOH, followed by ten minutes with high purity water, and finally fifteen minutes with electrolyte. Four electrolytes were used to analyze each solution. For cations, UVCat1 and UVCat2 (Waters Corp. Milford, MA) were used and for anions, chromate and phosphate electrolytes were used.[3] Calibration curves with an R^2 value of 0.99 or greater were required for analysis using commercial standards from Analytical Products Group, Inc. (Belpre, OH). Sample injection was performed hydrostatically for thirty seconds at a ten centimeter elevation.

After analysis of sixty samples, a first generation solution was developed. This solution contained all of the ions detected by CE at their average concentrations. The solution was developed so that no additional ions were added except for H^+ and OH^- which were added as needed to maintain electroneutrality. The solution contained eighteen salts for a total of twenty-three ions. Through electrochemical testing, the ions which affected corrosion current density, corrosion potential, and corrosion morphology were determined. A final solution composition was thus determined. Only these ions were added to the final solution in the highest concentrations measured by CE. The highest concentration was used to observe the greatest effect possible in the electrochemical testing and was less than twenty millimolar for any ion.

2.3 ELECTROCHEMICAL TESTS

Electrochemical testing was used to determine any difference in the behavior of AA2024-T3 in CE-derived solutions versus sodium chloride. All tests were performed with a PAR Versastat (EG&G) connected to a computer with PAR M352 Softcorr II software. Testing was performed a flat cell with a one square centimeter working electrode of bare aluminum alloy 2024-T3 with 32 mil thickness. The coupons were wet polished by hand to a 600 grit finish with silicon carbide paper. Potentiodynamic tests were used to compare the open circuit potentials, pitting potentials, and passive behavior of 2024 in the first generation solution and 0.6 M sodium chloride.

A factorial design was used to develop two sets of experiments that could determine the statistically significant variables in control of important corrosion parameters. The first set of experiments studied six variables: chloride, nitrite, sulfate, bicarbonate, the pH, and "others". The "others" are all of the other anions detected by capillary electrophoresis added as a group. When a species was added to the solution (as determined by the factorial design), it was added in the highest concentration measured in the lap-joints by CE. The pH was either 3.5 or 9.0 with 9.0 chosen because it is the average ambient pH of our bulk analogue solutions and 3.5 chosen because it is the pH found in occluded regions on aluminum alloys with a boldly exposed cathode. Cyclic polarization scans were generated using a platinum counter electrode and a saturated calomel reference electrode. After testing, the samples were examined with optical microscopy to determine the presence of pits, the relative size and density of pits, and any changes in surface roughness. Changes in surface roughness were defined as the inability to see the scratches caused by the initial polishing. The results were analyzed using the statistical analysis program SPSS ver. 8.0 (Statistical Product and Service Solutions, Chicago, IL) to determine which species caused a statistically significant change (greater than 99% confidence level) in the parameters of interest. The second factorial investigated the ions that were in the "others" category in the first set of experiments. The variables were: fluoride, nitrate and phosphate, organics (formate, acetate, propionate, and butyrate), and the pH.

2.4 EXPOSURE TESTING

Model lap joints were constructed for tests to determine the corrosion morphology produced by exposure of 2024 to various solutions. The model lap joints were constructed from two sheets of AA2024-T3 cut to pieces one inch by three inches. The pieces were held together by two aluminum pop rivets. Studies were conducted for three months at three different temperatures in four solutions. The temperatures were room, 35, and 45 degrees Celsius. The solutions were the first generation solution, DI water, 0.6 M sodium chloride, and 1 mM hydrochloric acid and 4 mM $Cu(NO_3)_2$. Model joints were placed in solution so that the solution could wick into the joint by capillary action. Periodically, water was added to maintain a constant water level. After three months the bulk and occluded pH values were measured. The corrosion rate was calculated from weight loss data and corrosion morphology was examined with cross-sectional metallography. The model joints were cross-sections, mounted in epoxy, and polished to a one micron finish with diamond spray compounds. The samples were then etched with Keller's etch for 45 seconds before examination with an optical microscope.

Model joints were also used to determine the potential within a joint. Any area directly exposed to bulk solution was painted with XP-2000 Peelable Stop-off Laquer (Tolbar Products, Hope, AR) to force all electrochemical activity to occur within the joint. The same solutions were used as for the exposure tests. The tip of a saturated calomel reference electrode was contacted with solution at a marked spot and the potential was measured by contacting one lead of a voltmeter to the reference and the other to the model joint. This potential was measured weekly for three months.

2.5 EXPERIMENTAL pH

Coupons of 2024 were placed in four solutions and the pH was measured weekly for six months. The solutions used were DI water, 0.6 M sodium chloride, the first generation solution, and the first generation solution diluted one to ten. One hundred milliliters of each solution were used. Another experiment to show that a range of pH values could be measured using our techniques involved exposure of 1 mM HCl solution to a sheet of 2024. The pH of this solution was measured daily until no solution remained on the aluminum surface.

2.6 FATIGUE TESTING

A series of corrosion fatigue tests were conducted to study the effects of crevice environment on the growth of fatigue cracks in aluminum alloy 2024-T3 sheet. Constant amplitude fatigue crack growth tests were performed using the eccentrically-loaded single edge crack tension specimen (ESE(T) – formally the EC(T) specimen)[4]. The back-face strain compliance method was used to continuously monitor crack length and to verify closure free (intrinsic) crack growth. The constant cyclic stress intensity factor (ΔK) experiments were conducted using a computer controlled servohydraulic test machine that continuously adjusted the load to maintain the programmed ΔK at a constant stress ratio (R). The mid-half of the ESE(T) specimen (fatigue crack region) was encapsulated by a 1.5 liter O-ring sealed plastic chamber that contained the crevice solution at room temperature. Specimen electrochemical potential was potentiostatically controlled using a platinum counter and Ag/AgCl reference electrodes. A reservoir containing 20 liters of solution exposed to air was continuously circulated through the test chamber at a rate of 30 ml/min.

3. RESULTS AND DISCUSSION

3.1 ION ANALYSIS

In the sixty corrosion product samples analyzed to date, twenty-three ions have been detected. The result from analysis of a capillary electrophoresis sample is an electropherogram. The peaks represent the changes in UV absorbance that occur as the bands of each ion pass by the UV detector. Example electropherograms showing the common ions detected are shown in Figure 1. The commonly found anions were chloride, sulfate, nitrate, and bicarbonate. Aluminum, potassium, sodium, and magnesium were the commonly found cations. Common ions are those that are present in at least seventy-five percent of the sample solutions. It is important to note that the solution present within lap-splice joints is not simply sodium chloride, but is actually rather complicated. It is also not as concentrated as the standard 0.6 M sodium chloride solution. The sum of all of the highest ion concentrations is only 80 mM.
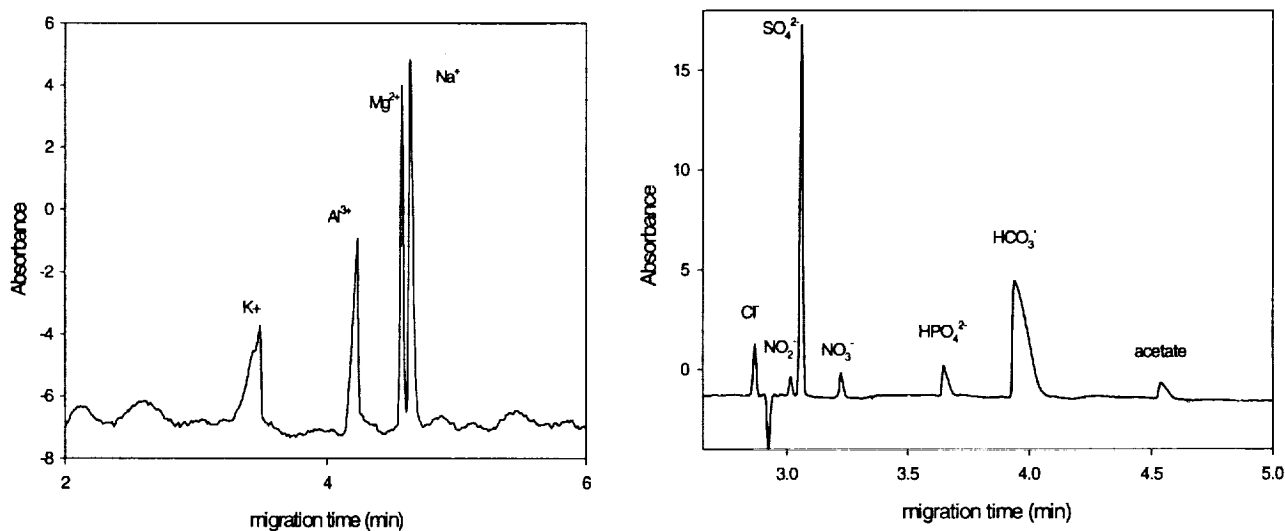

Figure 1: Example electropherograms showing common cations and anions

The ions present in the samples varied greatly even between samples from a single aircraft. Figure 2 shows two charts that illustrate the variability of the samples. The top chart shows the percentage of samples in which each anion is found. The range of concentrations also varies between samples. The lower chart shows the number of samples that contained a concentration of chloride in each range. Like ocean water, there is not a single solution that will have the exact ions and concentrations as every sample taken. Instead, our goal has been to develop a model solution that recreates the corrosion morphology and important electrochemical parameters that exist within actual lap-splice joints. A solution derived from the CE results better reproduced the corrosion environment found within these joints and so electrochemical and mechanical tests performed in this solution will better examine the conditions to which lap-splice joints are exposed.
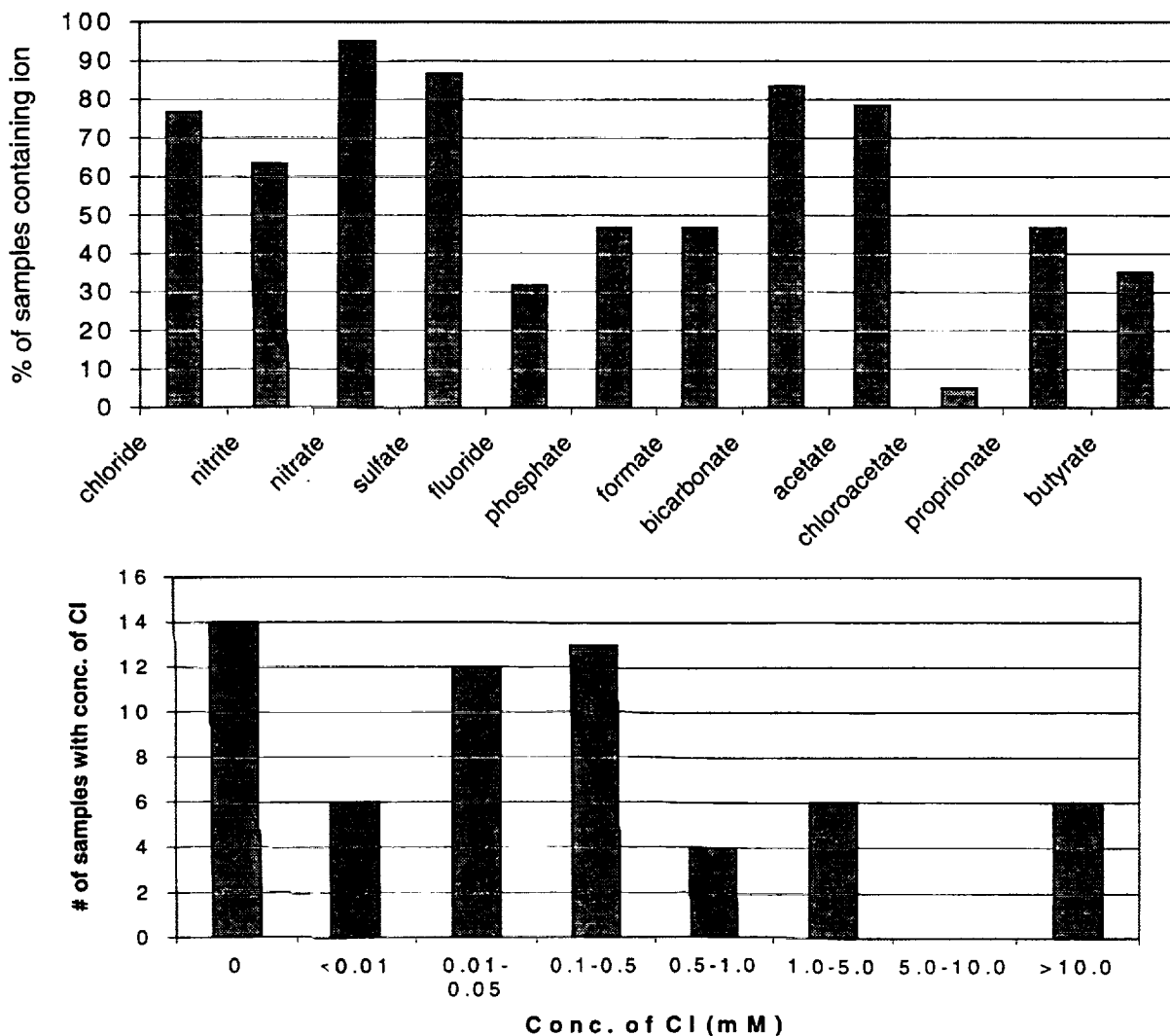
Figure 2: Variability of anions and concentrations detected by capillary electrophoresis

3.2 ELECTROCHEMICAL TESTING

The initial testing demonstrated that the electrochemical behavior of 2024-T3 in the first generation solution was very different from that in sodium chloride. As shown in Figure 3, the sample tested in 0.6 M sodium chloride did not passivate, but was immediately pitting while the sample run in the first generation solution has about an 600 mV passive region. This passive region give the sample in the first generation solution a higher pitting potential (about -0.2 V_{SCE}) than in the sodium chloride where the sample actively pits at all potentials above the open circuit (-0.6 V_{SCE}).

The results of the two factorial designs from the cyclic polarization scan data are shown in Table 1. The arrows indicate the direction in which the parameter was changed with the increase of that variable. The variable listed first for each parameter was the one that was found to be the most significant. All of the investigated pitting parameters (presence, size, and density) depended solely on the chloride concentration, but chloride was not a dominant factor in all of the studied parameters. This reinforces the idea that testing in sodium chloride solution is insufficient. This information allowed for the

determination of the recommended solution composition containing only the significant ions (20 mM chloride, 4 mM nitrite, 4 mM bicarbonate, and 2 mM fluoride at pH 9.0). The factorial also demonstrated that even in dilute solutions (<80 mM = total ionic concentration) the impact of individual ions on the corrosion rate, potential, and morphology could be observed.
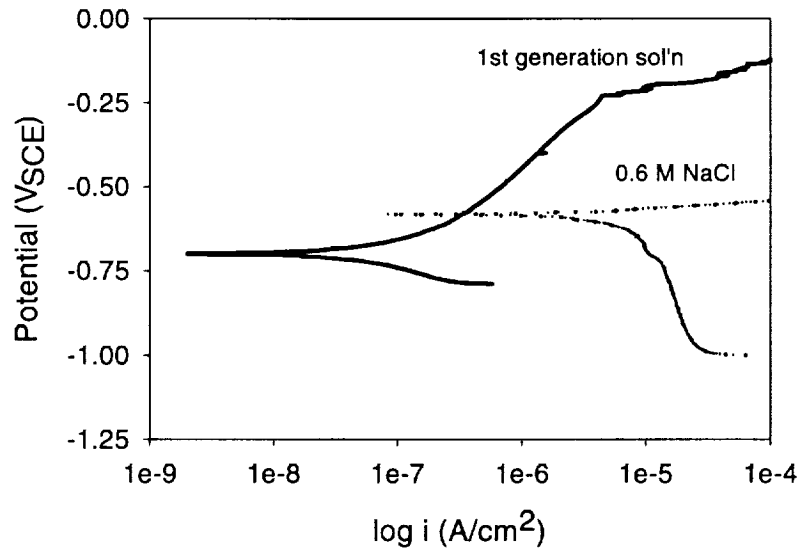

Figure 3. Comparison of electrochemical behavior of AA2024 in 0.6 M NaCl and the 1st generation solution

TABLE 1. Results from the factorial design analyses

Parameter	significant variables
i_{corr}	pH↓ , F↑ , NO$_2$↑
E_{corr}	Cl↓, HCO$_3$↓, NO$_2$↑
pitting during polarization scan	Cl↑
increased surface roughness	HCO$_3$↑, pH↑

3.3 EXPOSURE TESTING

Exposure of model joints to the test solutions allowed for comparisons of the resulting corrosion morphology and calculated corrosion rates. Along with examination of corroded model lap joints, doubler samples were studied. Cross-sectional metallography determined that the most common corrosion morphology in the doubler samples is general corrosion. In about half of the doublers studied, the aluminum clad layer, a thin layer of pure aluminum used for corrosion prevention, was still visible. In one of the eleven doubler samples examined, pitting corrosion was found. Also on this doubler intergranular corrosion was evident near the spot welds. The morphology of models exposed to sodium chloride solution was well-defined pitting. Within models exposed to the first generation solution, uniform corrosion similar to that observed in the doubler samples was observed.

Both the bulk and occluded pH were measured at the end of the temperature study. The bulk pH was measured with a pH meter and glass electrode while the occluded pH was measured with pH paper. As can be seen in Table 2, the occluded pH in all solutions approached a value of 8.0. This agrees with pH measurements taken directly of wetted doublers that were corroded in the field and with the pH of our CE derived solutions which are also mildly alkaline. From the weight loss data, the corrosion rates were calculated. These rates are shown in Table 2 and indicate that corrosion rate does not increase significantly with temperature. Additional research is underway to investigate the dependence.

TABLE 2. Results from exposure test at three temperatures

solution	temperature	bulk pH	occluded pH	μm/yr (mpy)
DI water	room	7.2	8.0	177.0 (7.1)
1st generation	room	8.5	7.7	227.0 (9.1)
NaCl	room	7.3	8.0	256.3 (10.2)
HCl+Cu	room	2.5	7.7	286.7 (11.5)
DI water	35 C	7.5	7.7	217.2 (8.7)
1st generation	35 C	8.5	7.7	217.2 (8.7)
NaCl	35 C	7.6	8.0	294.3 (11.8)
DI water	45 C	7.2	8.0	196.6 (7.8)
1st generation	45 C	8.7	8.0	220.5 (8.8)

The potential within model lap joints was measured over thirty days. As shown in Figure 5, the occluded potential reached a value above the open circuit potential of AA2024 when boldly exposed to the same solution for five hours. The open circuit potentials of AA 2024-T3 in DI water is -0.4 Volts, in CE-derived solutions is -1.0 Volts, and in sodium chloride solution is -0.7 V_{SCE}. The occluded potentials of model joints that had wicked in these solutions were all above -0.3 Volts. The potentials have been used in fatigue crack growth experiments to simulate more complete environmental conditions.
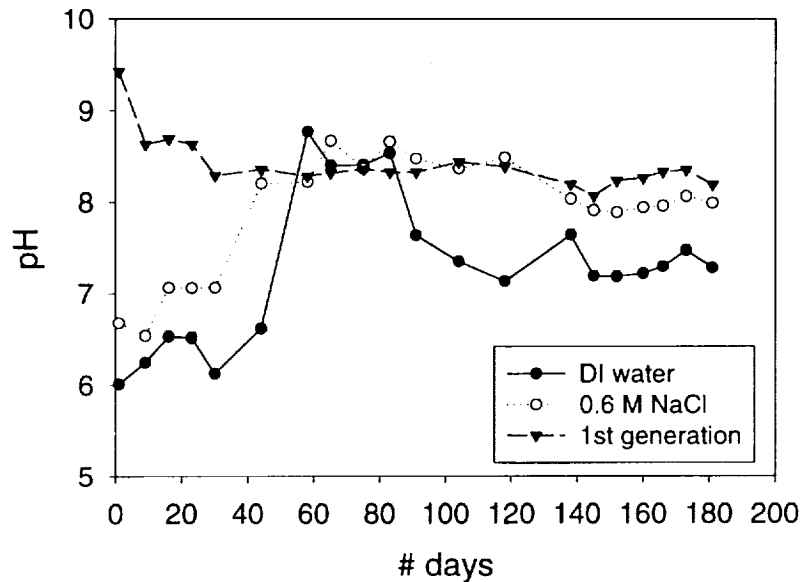

Figure 5. Occluded potentials of model joints over time

3.4 EVOLUTION OF pH

As shown in the exposure testing, the occluded pH of model joints approaches 8.0 after a three month exposure. The pH of similar solutions exposed to a piece of 2024 with ambient aeration, also approaches a mildly alkaline pH value. Figure 6 shows the pH evolution of these solutions over a 180 day exposure. All four solutions reached a pH of between 7.5 and 9.0. It is important to note that in both this set of experiments and the temperature study that not only did the lower pH solutions rise to a higher pH, but also the higher pH solutions also decreased to this mildly alkaline pH value.
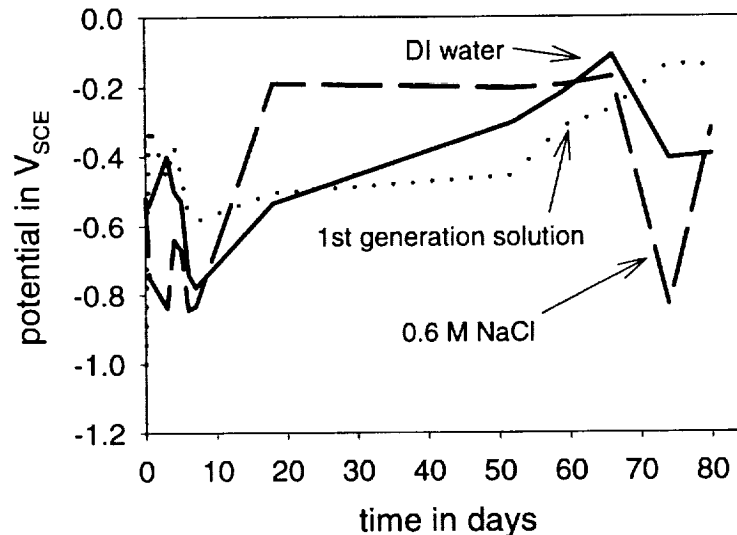

Figure 6. pH values of solutions exposed to AA2024 over time

When 0.5 mL of one millimolar hydrochloric acid was placed on a sheet of 2024-T3 and the pH was measured over time, the pH also increased with time as shown in Figure 7. This shows that a large range of pH can be measured with pH paper and again confirms the tendency of solutions to increase in pH when exposed to this alloy.
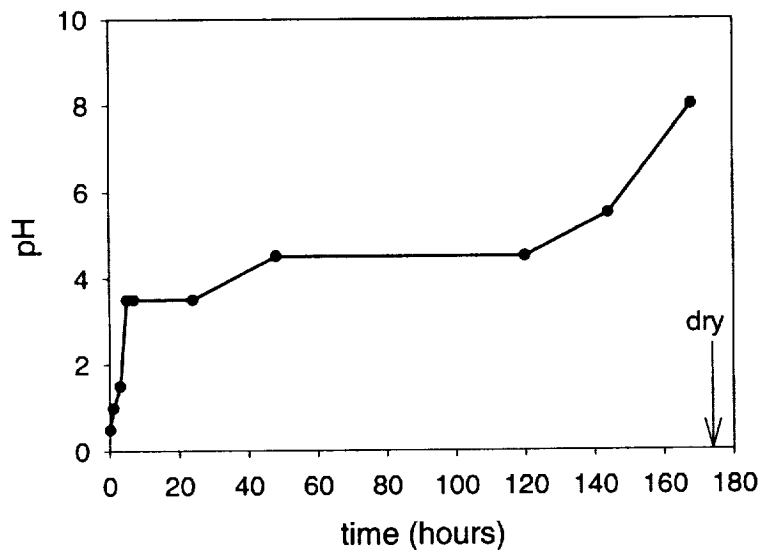

Figure 7. Values of pH over time of HCl solution on AA2024

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3.5 FATIGUE TESTING

The objective of the first series of corrosion fatigue tests was to determine the cumulative affects of the compound groups listed in Table 3 on the fatigue crack growth (FCG) characteristics of alloy 2024. Eleven constant ΔK tests (5.5 MPa$\sqrt{m}$, R=0.5, f=2Hz) were performed in lab air followed by distilled water and nine solutions containing the additions listed in Table 3. A stress ratio of 0.5 was selected to limit the effects of crack-tip closure and thereby produce intrinsic environmental FCG rates. To study the effect of each compound group, changes in crack growth rate were monitored as each of the nine sets of compounds was added to the reservoir of distilled water (combined with the previously added compounds). The series of tests were designed to expose the crack to more benign environments (compounds) initially followed by increasingly more aggressive compounds. The test results shown in Table 3 reveal no difference in the intrinsic fatigue crack growth rate characteristics of alloy 2024 for air, distilled water, and eight of the nine compounds over a pH range of 5.5 to 10.5. A small decrease in FCG rate was observed after adding compound group 2 to the solution.

TABLE 3. TEST SERIES #1 ENVIRONMENTS

Group #	Compounds Added	pH	FCG rate (mm/cycle) x10^5
Air	-----------------	------	2.4
H_2O	-----------------	5.5	2.5
1	$Al(OH)_3$, $Ca(OH)_2$, KOH, NaOH	10.5	2.5
2	$SrCO_3$, $CuSO_4$, $MnCl_3\cdot4H_2O$, $ZnC_2O_2H_3$, $MgCO_3\cdot3H_2O$	10.1	1.5
3	$Al_2F_6\cdot H_2O$, NaF, $NaC_3H_7CO_2$, $NaC_2H_5CO_2$	10.1	2.4
4	$AlCl_3\cdot6H_2O$	9.8	2.6
5	KNO_3	9.7	2.8
6	$NaNO_2$	9.4	2.4
7	K_2SO_4	9.4	2.2
8	$K_2H_2PO_4$	7.8	2.3
9	$NaHCO_3$	8.1	2.2

The second series of constant ΔK tests determined the affect of electrochemical potential on the FCG rate in the crevice solution containing all the compound groups listed in Table 3. Thirteen fatigue crack growth tests were conducted; test #1 was performed in laboratory air and the remaining twelve crack growth tests were conducted in the crevice solution at different constant electrochemical potentials. The tests were conducted at potentials both cathodic to and anodic to the corrosion potential of -575 mV$_{SCE}$. Figure 8 is a summary of results for the constant ΔK test; here, a change the slope of the crack length versus load cycles plot represents a change in FCG rate. Figure 8a shows that similar intrinsic FCG rates were obtained for laboratory air and crevice environments at cathodic potentials of -1015 mV$_{SCE}$, -815 mV$_{SCE}$, -615 mV$_{SCE}$ and an anodic potential of -415 mV$_{SCE}$. Figure 8b is an enlargement of Figure 8a showing the results of tests 6 - 13. As the potential was changed from -415 mV (test #6) to -215 mV (test #7), the FCG rate initially increased and then rapidly decreased. The rapid decease in FCG rate (region A in Figure 8b) corresponded to the observation of copious amounts of corrosion product along the outside surface of the fatigue crack and a corresponding increase in the measured level of crack closure. This behavior was reproduced at regions B and C in Figure 8b where the fatigue crack stopped growing when exposed to the same highly corrosive anodic (-215 mV) environment. Presumably, the initial increase in intrinsic (closure free) FCG resulted from the additional chemical crack-tip driving force produced by an increase in crack-tip corrosion at an anodic potential of -215 mV$_{SCE}$. The retardation in FCG rate at -215 mV$_{SCE}$ was caused by the rapid buildup of corrosion products that

produced crack closure and a corresponding decrease in the mechanical crack-tip driving force. Figure 8b also shows that loading frequency influences the observed corrosion effects, possibly suggesting complex time dependent behavior.
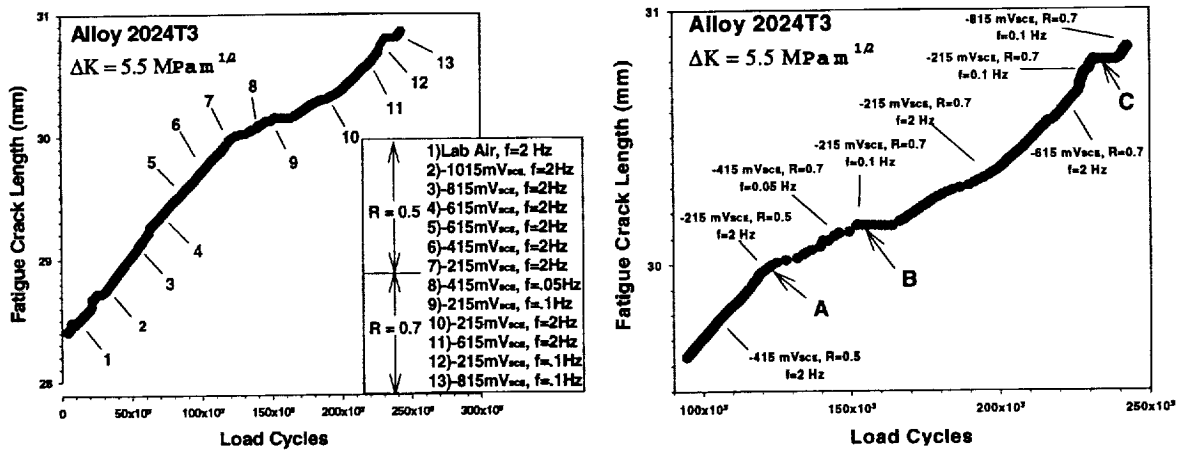

Figure 8. A summary of constant ΔK test results showing the effect of electrochemical potential on the fatigue crack growth characteristics of alloy 2024 exposed to a laboratory simulated crevice corrosion environment.

CONCLUSIONS

By collecting samples directly from field-exposed aircraft, the environment to which the interior of a lap-splice joint is exposed has been investigated. Analyses of the solutions formed by dissolving corrosion product sample have determined that the occluded solutions found within these aircraft joints are complicated, dilute, and mildly alkaline. They are complicated as over twenty different ions have been detected in the sample solutions. The total concentration of all ions is less than 80 mM which is significantly more dilute than 0.6 M sodium chloride that is typically used in such testing. By exposure testing and direct measurement of the surface of a corroded aircraft skin, the pH of the solution was found to be mildly alkaline. A bulk analogue solution with these characteristics has been developed using the information obtained by analyzing solutions derived from scraping samples with capillary electophoresis and used for electrochemical and exposure testing. Based on statistical analyses of the effects of each ion on corrosion current density, potential, and morphology, the first generation solution was simplified to a solution containing 20 mM chloride, 4 mM nitrite, 4 mM bicarbonate, and 2 mM fluoride at pH 9.0. Model lap joints were used to determine that the occluded potential within a lap joint is greater than the open circuit potential of 2024 exposed to the same solution. Potentiodynamic scans performed on AA2024 using CE-derived solution and 0.6 M sodium chloride had determined that the electrochemical behavior of 2024 is significantly different in the two solutions. There is also an important difference in the corrosion morphology of model lap joints exposed to the two solutions for three months. General corrosion occurs when model lap joints are exposed to CE-derived solutions, whereas pitting occurs when the model joints are exposed to 0.6 M NaCl. This difference is important because the observed corrosion morphology in skins obtained from actual aircraft is general corrosion. In fatigue testing, the observation that the occluded potential is above the open circuit potential of boldly exposed surfaces has proven to be important in producing corrosion product and leading to crack closure. All of these observations indicate that the CE-derived solutions are more relevant to the corrosion environment found within actual aircraft lap-splice joints and should be used for future electrochemical and mechanical testing.

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ACKNOWLEDGMENTS

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