Corrosion Fatigue Pit-to-Crack Test Methodology

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Abstract: The objective of this work was to develop a test methodology for corrosion fatigue that allows researchers to investigate the effects of environmental parameters on crack growth rates of cracks nucleating from corrosion pits. Small changes in crack growth rates while the crack is small can have large effects on the life of structural components due to the exponential nature of fatigue crack growth. The effect of changing environments on the crack growth rate is important to the corrosion fatigue community as chromate containing primers and protective coatings are thought to help protect components during this mechanically small crack growth regime by chemically altering the local environment around the crack tip. The chromate protection offered in the early stages of crack growth can become very significant to part life as the effect of the chromate is potentially lessened as the crack becomes larger. This is due to the environment having less of an influence on crack growth as the mechanical crack driving force overcomes the environmental effect as crack length increases. The current push for reduction in chromate containing coatings due to health and environmental concerns, could have an unintended consequence that component lives will suffer due to the removal of the chromate's corrosion protection. The test methodology developed herein, was designed to address the potential reduction in life by providing a test platform where the effects of varying environmental factors on the crack growth of small cracks could be fully investigated.

BACKGROUND

Corrosion pits in aircraft structure have long been known for their role in crack nucleation[1]. For any given crack that nucleates from a corrosion pit, the majority of the crack growth life is spent nucleating and growing in the mechanically small crack length scale. In this case, mechanically small is considered to be on the order of the largest grain dimension. These small cracks from pits are of no consequence to fleet managers using the USAF damage tolerant approach because a 1.27 mm rogue flaw is assumed to be present in the component. For USAF durability analysis, the initial crack size is assumed to be 0.25 – 0.50 mm. The US Navy safe-life management approach is based on crack nucleation to a 0.25 mm crack. Thus, this test method is relevant to both USAF durability and US Navy safe-life analyses if based on linear elastic fracture mechanics (LEFM). Furthermore, any change in the part coating system or service environment that may be more aggressive or beneficial may not be accounted for in the component life. To be conservative, the safe-life methodology uses a harsher environment to calculate the time required to propagate a crack to 0.25 mm. Research into chromate coatings[2] have shown a reduction in crack growth rates at long crack lengths; however, influences in the crack growth rates in the small crack range can be even more significant. The exponential nature of crack growth can magnify any small change in either crack nucleation time, or crack growth rate, resulting in large changes to the total life of the component. It is currently not fully understood what level of protection chromate provides during early crack growth, nor is there a standardized test methodology for research to produce quality, comparable data. The push for less toxic materials is changing the chemistry of some primers and full consequences of the changes in terms of corrosion fatigue performance should be understood by designers and maintainers prior to implementation. This test protocol is designed to give researchers a tool which can provide data to evaluate these changes in crack growth allowing better prediction and modelling techniques for cracks nucleating from corrosion pits.

TEST METHODS

Direct Current Potential Drop (DCPD)

DCPD crack growth monitoring was used for its accuracy and durability [3]. DCPD probes can be placed in positions not possible or practical with conventional measurment techniques. The most basic theory of operation for the DCPD method of monitoring crack length, is that the electrical resistance of a body changes when a crack grows through it. A constant current is passed through the specimen such that when two probes are placed across the cracked body a change in the resulting voltage can be measured due to the change in resistance. The DCPD system requires specialized hardware, and the attachment of voltage sensing lead wires to the specimen. The engineering challenge from the latter requirement becomes evident when dealing with small defects such as corrosion pits. The stress concentration due to the wire attachment can be greater than that of the corrosion pit causing fatigue nucleation at the lead attachments. Additionally, lead wire placement and spacing have precision and accuracy effects on the measured voltage. A protocol was developed solely devoted to producing a robust lead wire attachment to the specimen through micro-spot welding of copper or platinum wire, and is discussed in the Spot Welding section below.

Specimen Design and Validation

An open hole specimen was developed to serve as the standard platform on which the corrosion fatigue test methodology is built. The design of the specimen was envisioned to be easily fabricated with minimal specialty tooling or fixturing required. The specimen hole diameter and gage thickness are common in aircraft construction and the narrow width was chosen to have the stress intensity factor (*K*) equal at the crack tip along the hole bore and plate surface. In addition, this geometry was chosen for its openness to environment, and flexibility for future geometries such as filled holes. The long-term vision is to develop this methodology into a standardized test procedure that can be shared among research laboratories. Open hole specimens were fabricated from 50.8 mm thick plate aluminium alloy 7075-T651. The aluminum plate used throughout the test program was produced in a manner which replicated legacy aluminium alloys by keeping historically relevant material compositions [4]. Particular focus was placed on the iron content of the aluminium, as crack nucleation has been observed from the iron bearing constituent particles [5]. This material was used throughout the pit-to-crack program because of the characterization work performed during the aforementioned program. The material was characterized for fatigue performance, allowing for comparisons to be made between the open hole data generated from this work, and the previously generated characterization data. All data generated and compared herein has been procured from the same lot of material to eliminate the lot-to-lot material variances. All open hole specimens were taken from a thick plate of aluminum, as such, care was taken to remove and fabricate specimens with their centerline located at the T/6 thickness from the aluminum plate to minimize through-thickness effects. Figure 1 shows an overview of the open hole specimen used throughout this test method.

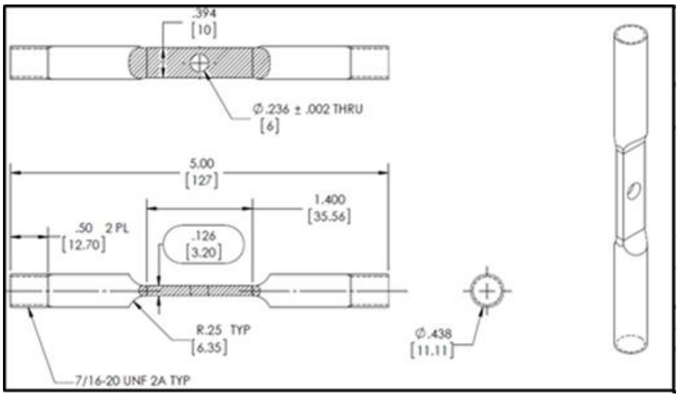

Figure 1. Open hole specimen geometry.

Laboratory Pit Creation

Since fatigue cracks often nucleate form corrosion pits and the transition from corrosion pit to fatigue crack is significant in the time to fail a component, the open hole specimen has a laboratory created pit as the crack nucleating feature. The laboratory generated pit was placed at the corner of the open hole specimen to promote crack nucleation from the pit. These laboratory generated pits are formed from an electro-chemical process which produces a rough surface morphology that is similar to naturally occurring pits observed on in-service parts [6]. The rough surface morphology is desired because the typical crack nucleating features tend to be micro-pits or jut-in features that form at the interface between the bulk material and pit surface. These small surface features produce a large cyclic plastic strain that promotes crack nucleation. This test method used these laboratory generated pits exclusively to nucleate small fatigue cracks. A detailed description of the pitting process is discussed in reference [7].

Spot Welding Methods

The use of DCPD as the crack measurement method requires that conductive leads be welded to the specimen. The application and positioning of the DCPD leads can have an effect on the measured DCPD voltage quality and by extension the crack length data. A second protocol was developed to guide the user through the procedure of attaching the DCPD leads, such that an even, and accurately positioned lead spacing will produce quality DCPD voltage data. In general, the closer the DCPD leads are attached to the nucleating defect, and the closer the two leads are to one another, will produce the most precise data. Spot welding variables such as wire spacing, damage site sizes, DCPD wire material selection, and reference probe usage are discussed. A detailed description of the welding process is described in reference [7].

Crack length calculation using DCPD and marker band data

In order to correlate normalized DCPD voltages to crack lengths, a relationship had to be developed by linking both the DCPD voltages and crack lengths to cycle count [8]. DCPD voltages are recorded through onboard data acquisition, while crack length data was collected from the specimen fracture surface. To observe the crack evolution as a function of time (cycle count) a marker load spectrum was applied during specimen fatigue loading. The marker spectrum consisted of a series of loads applied in a manner that leaves distinct bands across the facture surface that are correlated back to cycle count post-test. A schematic outlining the applied loading used throughout fatigue testing is shown in **Error! Reference source not found.**.

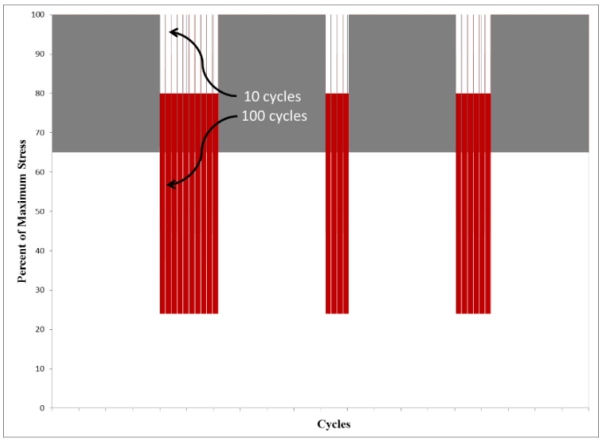

Figure 2. Schematic showing marker spectrum.

Post-test fracture surface reconstruction was carried out via scanning electron microscope (SEM). During the post-test reconstruction of the fracture surface each marker band is mapped out to provide the location of the crack front in relation to the nucleating point along the corrosion pit. An example marker band map is shown in Figure 3.

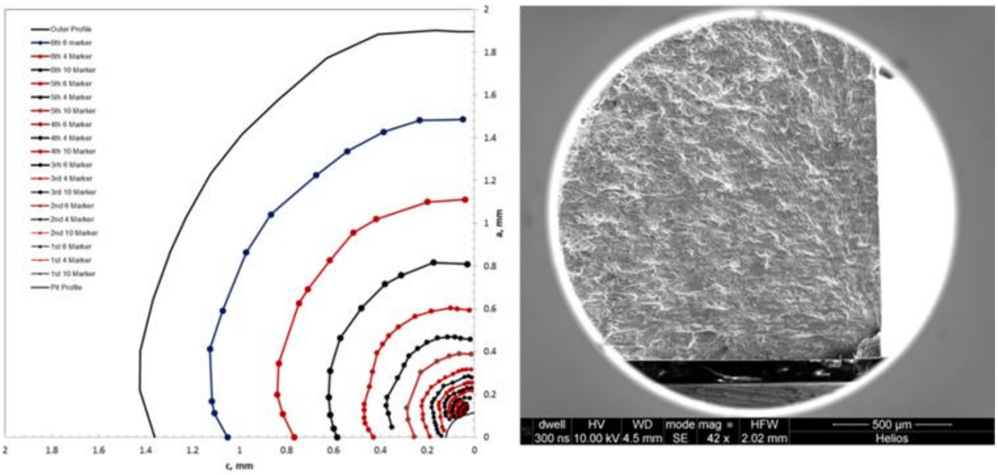

Figure 3. Example marker band mapping.

The distance from the nucleation point along the length and thickness of the specimen are recoded as the crack length in the *a* (thickness) and *c* (length) directions. Loading conditions for all tests were conducted at a maximum remote stress (S_{max}) of 125 MPa with a stress ratio (R) equal to 0.65 at a frequency of 10 Hz. The fracture surface mapping coupled with known cycle counts enabled reconstruction of the specimen crack front. Once the reconstructed crack front progression is known, the estimation of key points of interest, such as nucleation time and periphery crack formation can be found. Periphery crack formation occurs when a continuous crack front intersects both surfaces (hole bore and specimen surface) of the ligament, and is particularly important for damage modeling as this is the earliest time that crack growth predictions can be made using existing *K* solutions. Using the normalized DCPD voltage and plotting against the marker band measured crack lengths, a calibration curve relating the two can be created. Figure 4 shows the calibration curve for the open hole specimens.

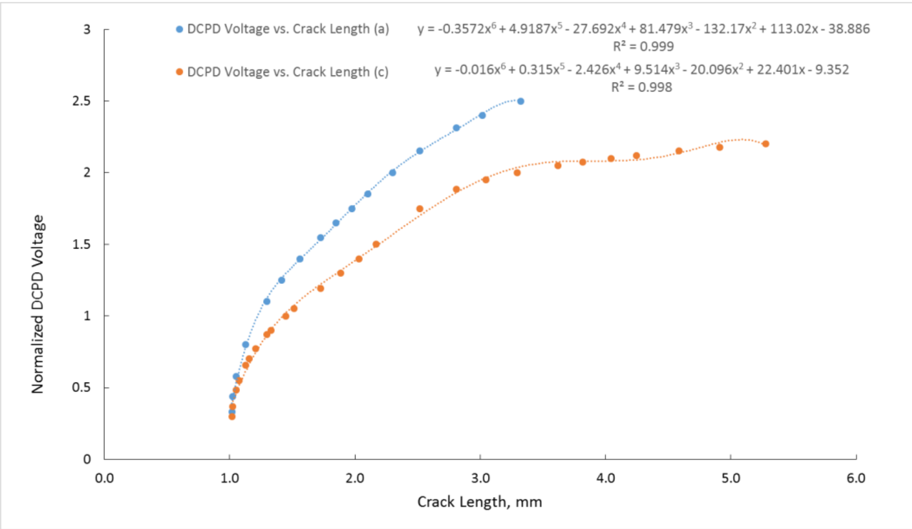

Figure 4. DCPD calibration curve for the open hole specimen.

Fitting a 6th order polynomial for each of the two crack directions provides equations that relate each of the two crack directions to a single normalized DCPD voltage. By calculating each of the two crack directions separately, both the *a* and *c* direction crack lengths are correlated independently removing a dependence on relating *a/c* ratios. Creating the crack length calibration with specimens in laboratory air is required as subsequent tests in corrosive environments can leave deposits, or attack the fracture surface during exposure obscuring the marker bands.

Full Immersion Testing

Validation of the test method required that a corrosive environment be introduced to prove the test method can generate the required data to detect marked changes in crack growth. The environment chosen for the full immersion testing was a 0.06M solution of NaCl. The salt water solution is known to accelerate crack growth significantly [9]. The open hole fatigue specimens tested in immersion were identical in geometry to those tested in laboratory air. The loading conditions between the laboratory air and full immersion testing were also kept constant. The test frequency was slowed to 1 Hz in immersion from 10 Hz in laboratory air. The reduction in test frequency allows more time for the environment to affect the exposed crack tip. If the test frequency is too fast, the environmental effect is minimal producing results closer to those in air. This test frequency also affects the potential efficiency of future testing when added corrosion inhibitor will be added to the environment. In order to keep the frequency consistent between test conditions it was required to be fixed at this time and account for both test conditions. The 1 Hz test frequency is ultimately a trade-off between test duration and time of exposure to environment. Figure 5 shows an open hole specimen mounted inside of the full immersion test cell. Note, there is relief around the top of the specimen as it passes through the upper end cap of the test cell. This relief is provided to ensure that no load transfer was allowed to bypass the specimen through the test cell.

Figure 5. Open hole specimen in full immersion test cell.

Crack acceleration due to environment is not the only valid test result. The test method must also measure decreases in crack growth rates in the presence of crack growth inhibition. Verification of the method's ability to measure crack growth rate inhibition was conducted by measuring the decrease in crack growth rates of open hole specimens tested in 0.06M NaCl solution with 0.03M NaMoO4 inhibitor added to the bulk solution. One litre of the bulk solution was circulated by peristaltic pump throughout the test duration, with no additional aeration of the solution. The test conditions for specimen geometry, applied load, test frequency, and DCPD settings remained identical to the full immersion testing.

Atmospheric Testing

Expansion of the test method into more atmospherically relevant environments required a slightly different approach than what was required for the full immersion tests. Rather than simply submerging the specimen in a salt solution to increase crack growth rates, salt was applied directly to the specimen surface. Applying a thin layer of salt to the specimen surface better replicates how salt ingresses into cavities in real world scenarios. The thin salt layer once applied to the specimen is then rehydrated at a constant relative humidity controlled by a water/glycerol solution. The action of salt deposition and rehydration in the test cell is a close approximation to real world outdoor exposures where changes in relative humidity can cause the wetting and drying of exposed surfaces resulting in hydration and drying of atmospherically deposited salt. To deposit the salt on the specimen surface sodium chloride was dissolved in methanol at a rate of 140 mg. of NaCl per 50 mL. of methanol. The mixture was then dispensed by pipet onto the specimen surface at a dosing rate of 400 $\mu g/cm^2$. The 400 μg salt load was chosen to provide an accelerated test (higher chloride concentration) with respect to measured atmospheric deposition rates of salt near coastal areas [10]. Creating an even coat of salt across the specimen surface requires that the specimen be heated by a heat gun to approximately 55 °C. At this temperature the specimen surface is hot enough that when a drop of the methanol and salt solution hits the specimen the methanol evaporates immediately leaving behind the salt in an even coat where the droplet made contact with the specimen surface. Droplets can then be spread about the surface in an even fashion. The end result of this deposition being an evenly coated specimen surface with approximately 400 $\mu g/cm^2$. The surface may require reheating to keep the temperature in this range. Heating the surface much beyond 60 °C temperature would bring the methanol beyond its Leidenfrost point. At this point the methanol stays in its droplet form with a thin layer of air insulating it from the specimen surface keeping the salt from being deposited on the surface. This condition also frequently leads to the droplet falling off the specimen. Care needs to be taken to avoid excessive heat as this can change the aluminum temper, however, even at the highest temperatures of 70-80 °C, there is little effect on the temper of the aluminum.

TEST RESULTS

Baseline

Using the methods and specimen discussed earlier, baseline testing was completed in laboratory air at room temperature. The testing was performed in order to validate the specimen design by comparing crack growth rate data between the characterized plate of 7075-T651 and data generated using the open hole specimen. This characterized data would serve as the benchmark for all subsequent data generated from the pit-to-crack development work. Baseline testing was performed at a peak stress of 125 MPa and *R* equal to 0.65. The variable amplitude marker spectrum was used throughout all tests. Figure 6 below shows a comparison of crack growth rates between the open hole pit-to-crack test specimen and the previously characterized 7075-T651 plate data.

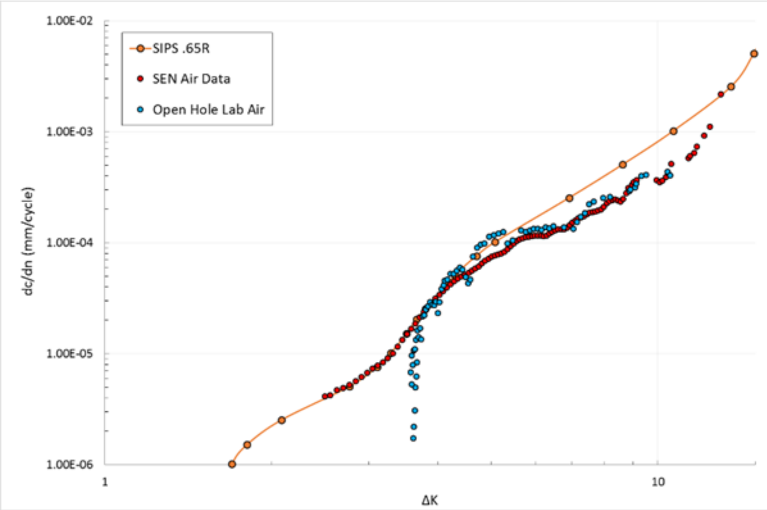

Figure 6. Crack growth rate comparison between characterized 7075-T651, open hole specimen, SEN specimen.

Baseline testing showed that a good comparison between the published 7075-T651 data and the open hole data can be made. There are some observable differences between the two data sets. One difference appears to be the threshold around *ΔK* of 3.5MPa√m for the open hole data. This apparent threshold is mostly due to the very small crack growth at the start of the fatigue test coupled with the variable amplitude loading. These two factors make it difficult to discern crack growth from noise at small crack growth. A *K* controlled test is being considered to possibly generate the near-threshold rate data. The open hole crack growth data matches the legacy crack growth data very well between *ΔK* of 3.75MPa√m to 5.5MPa√m. After 5.5MPa√m the rate dips below the previous fatigue crack growth rate data. Verification of the dip in crack growth rates was performed by analyzing another set of crack growth rate data from a second geometry. The second geometry was a single edge notch specimen (SEN) fabricated from the same lot of 7075-T651 plate at the T/6 plate thickness. The only difference between the SEN specimen and the open hole specimen is geometry. The SEN crack growth data follows the same trend beyond *ΔK* of 5.5MPa√m. The SEN data dipping below the previously published data suggests well known inter-laboratory variability [11] between the previously generated and current data. Additional noise in the crack growth data was generated by the variable amplitude loading, as the crack growth rate during the 80% marker band cycle still uses the *K* data from the 100% load cycles. Therefore under the 80% marker cycles an apparent decrease in crack growth is observed. The general trend in crack growth rate still provides a comparison between conditions as the data is consistent across all environments. Future data reduction will address this error by recalculating *K* for the 80% marker cycles.

Full Immersion in Salt Water

The baseline testing showed the specimen and DCPD crack growth calibration worked well for air testing, however the interesting data produced by the pit-to-crack test method are the tests conducted in various environments. While not relevant to operational aircraft, full immersion testing is commonly performed to investigate corrosion fatigue behavior. It is well known that immersion in a chloride solution will accelerate crack growth [9]. The sensitivity required of this test method is such that verification of increased crack growth rates in aggressive environments needed to be confirmed. Verification of such was performed with the open hole specimen immersed in a 0.06M NaCl solution. Loading was kept consistent with air data by continued use of the marker spectrum with a 125MPa maximum remote stress and 0.65 *R*. The test frequency was slowed to 1 Hz for full immersion testing in salt water. Figure 7 shows a comparison of crack growth rates between the open hole specimen in laboratory air and full immersion in saltwater.

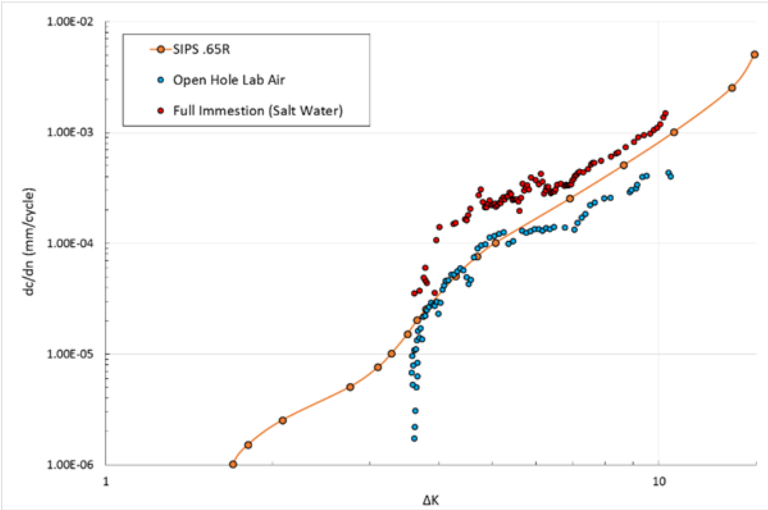

Figure 7. Fatigue crack growth rate data comparing laboratory air and full immersion in salt water.

The result of the full immersion verification tests in 0.06M salt water solution was an upward shift in the fatigue crack growth rate throughout the stress intensity range. The increase in crack growth rate due to the salt solution was the expected result and one that validates the ability of the test method to detect the acceleration of cracking due to an aggressive environment.

Full Immersion in Salt Water with added inhibitor

With the baseline testing having validated the specimen and DCPD crack growth calibration, and the full immersion testing in salt water providing the expected increase in crack growth rate, the next method that required validation was to show measurable inhibition. A previous study by Warner [12], had already shown that with relatively low concentrations of molybdate, and at low test frequencies (about 1 Hz), small amounts of crack growth inhibition were observed. To further validate the test method the open hole specimen was fatigue tested fully immersed in a 0.06M NaCl solution with 0.03M $NaMoO4$ added. Loading was kept consistent with previous testing, using the 125MPa maximum remote stress, with the variable amplitude marker spectrum keeping R equal to 0.65. Figure 8 shows a comparison between the open hole specimen in lab air data and full immersion in saltwater with added inhibitor data.

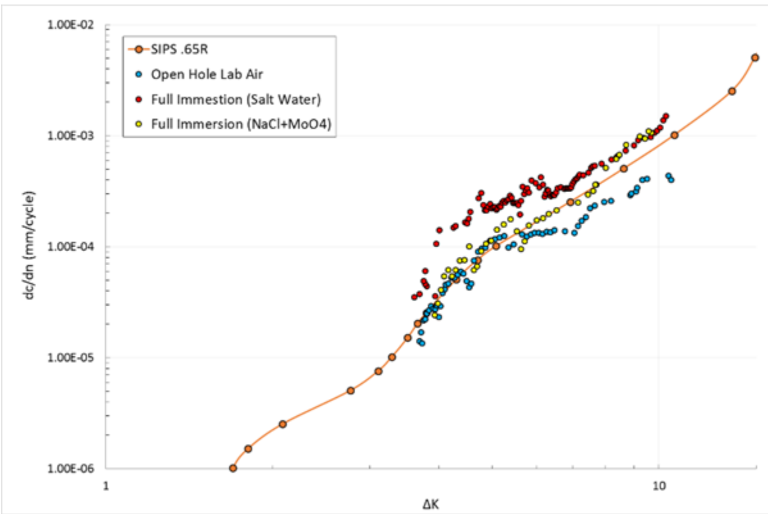

Figure 8. Comparison between laboratory air, full immersion in salt water, full immersion in salt water with added molybdate.

Adding the inhibitor to the salt solution was found to inhibit crack growth at lower *ΔK* values when compared to the full immersion in salt water specimen. The effect of inhibitor can be seen from a *ΔK* of approximately 4MPa√m until 5.75MPa√m, where the crack growth rate is similar to the specimen tested in air. Beyond *ΔK* of 5.75MPa√m the crack growth rate increases until it matches the full immersion data. The decrease in crack growth rate was successfully measured by the test method validating the inhibition measurement capability.

Atmospherically relevant salt film testing

The thin salt specimens were prepared for testing by depositing the salt by the hand deposition method described earlier. The specimen was then installed inside a test cell. A mixture of glycerol and water was added to bring the inside of the test cell to a constant relative humidity. The glycerol and water mixture was not allowed to make contact with the specimen. Figure 9 shows a specimen installed in the test cell and affixed to the test frame.

Figure 9. Open hole specimen in test cell.

Atmospheric corrosion testing was conducted at 80% relative humidity with a test frequency of 1 Hz. The marker load spectrum with a peak stress of 125MPa was applied with *R* equal to 0.65 for consistency between all test environments. Figure 10 compares the fatigue crack growth rate curves between the laboratory air, salt water full immersion, and thin salt film specimens.

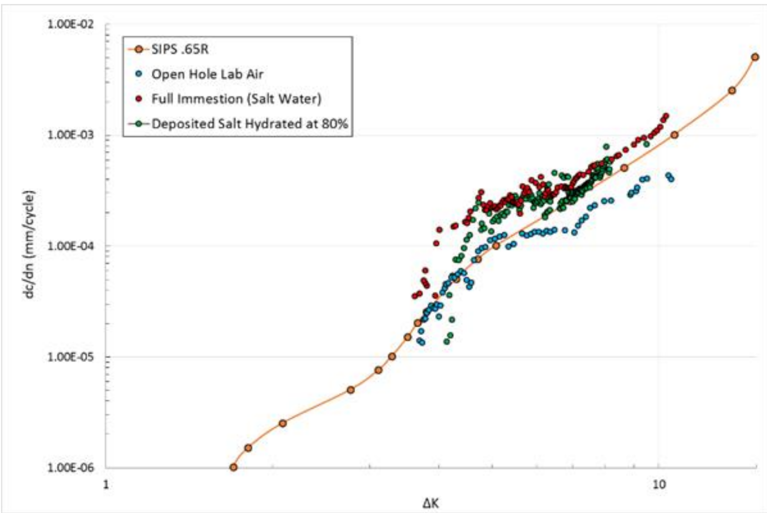

Figure 10. Comparison of fatigue crack growth rates between laboratory air and hydrated thin salt film test conditions.

The hydrated thin salt specimens exhibited an increase in crack growth rate slightly below the full immersion specimens. Comparing the two sets of data the open hole specimens tested in the 80% relative humidity environment with a thin salt layer applied to the specimen surface showed an upward shift in fatigue crack growth rate for a given ΔK versus the laboratory air specimens. The increase in crack growth rate appears to happen across the entire range of ΔK values.

CONCLUSIONS

This program successfully developed test methodologies for measuring the effects of environment on the crack growth rates of cracks nucleating from a small corrosion pit. The open hole specimen geometry developed for this test method was validated by creating a crack length calibration curve using the fracture surface data generated during laboratory air testing. The resulting crack growth rates compared favorably to the previously published 7075-T651 fatigue crack growth data. The calibration curves generated from the laboratory air data were then applied to DCPD crack length measurements and crack growth rate data was collected for additional environments relevant to corrosion fatigue research. Open hole specimens tested under full immersion in a salt water solution produced an increase in crack growth rate when compared to laboratory air. The increase in crack growth rate was observed throughout the entire crack growth curve, as expected in a known aggressive test environment. Similarly, when 0.03M sodium molybdate was added to the sodium chloride solution the test method successfully captured the inhibition of crack growth at lower values of ΔK replicating observations made by other researchers. The reduced crack growth resulted in capturing the expected decrease in fatigue crack growth rate. The inhibition effect was pronounced enough that the decrease in crack growth rate increased fatigue life beyond the laboratory air data. Test sensitivity is good enough that trends in initiation data are beginning to form. From the test data it was observed that inhibited crack growth behaved like a hybrid of the air and full immersion data. For small crack sizes at low ΔK crack growth was below the air data (inhibited region), however once the ΔK reaches approximately $6 MPa\sqrt{m}$ the crack growth rate begins increasing until matching the saltwater environment crack growth rate throughout the remainder of the test. Atmospherically relevant thin salt films were successfully applied to the open hole specimen surface through means of hand deposition. The resulting thin salt films were rehydrated with a water and glycerol solution to maintain constant relative humidity inside the test cell. The resulting increase in fatigue crack growth rate was captured by the test method and was found to be similar to full immersion rates when hydrated at 80% relative humidity.

FUTURE WORK

Future work will expand the current pit-to-crack test methodology into more atmospherically relevant environments. Current tests focused on corrosion fatigue in either benign (laboratory air) or overly aggressive (full immersion) environments. While needed for research either as baseline or comparisons to established tests, these tests do not have much relevance when compared to the actual environments in which aircraft operate. By expanding the test method to include chromate and non-chromate primers, ultra violet radiation, ozone, temperature and variable relative humidities, more relevant corrosion fatigue test data can be generated. Finally, by coupling flight loading with the simultaneous environmental effects the test method can provide data more accurately representative of flight and ground conditions, allowing for more accurate crack modeling predictions in all corrosion fatigue situations.

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