INCORRECT MATERIALS SELECTION AS A CAUSE OF ENVIRONMENTALLY ASSISTED CRACKING

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Selecting materials for structures and components involves consideration of numerous factors, including material properties, e.g. strength-to-weight ratio, resistance to fatigue, fracture, and various types of corrosion, as well as fabricability, cost, and availability. Choosing materials with ideal combinations of properties is usually not possible so that compromises are required, and failures often occur when due consideration is not given to (i) the effects of environment under the full range of operating conditions, and (ii) the effects of heat treatment on environmentally assisted cracking. Some examples of the above issues are described and discussed so that similar failures can be avoided in the future. Stress corrosion cracking of aluminium alloys is discussed in particular.

CRACK NUCLEATION, GROWTH & ARREST UNDER SUBCRITICAL CRACK GROWTH

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Crack nucleation (and growth) can be characterized under static load (or cyclic loads) in the presence of an environment. Chemically assisted cracking is occurs when stresses to nucleate (or propagate cracks) are much lower fracture of a material in an inert (like in vacuum) environment where a material is free from the damaging chemical environment. Similarly, embrittlement occurs if chemically active elements are dispersed internally as in internal hydrogen, metalloids and other embrittling elements. The materials where such embrittlement phenomena occurs are also divergent: pure metals, alloys, and ceramics or glasses. Since materials are used in applications involve various chemical environments under load, the importance of understanding the role of environment in material performance need not be stressed. In fact, there are many analyses in the past highlighting the divergent behaviors in each of the systems and environments emphasizing the specialties specific to a given material/environment system. In addition, many efforts have been made in the past to arrive at some unifying principles governing the embrittlement phenomena. An inescapable conclusion reached on this topic by many is that the behavior is very "complex". Hence, recognizing the complexity of material/environment behavior, we focus our attention, mainly on metallic systems, in extracting some similarities to arrive at some generic principles involved. The ultimate goal of this effort is to arrive at some self-consistent scheme for incorporating "chemical effects" into a life prediction model for components in service.

A PERSPECTIVE ON ENVIRONMENTALLY-INDUCED CRACKING

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The premature failure of engineering alloys in service is often associated with exposure to unintended environmental chemistry. High strength carbon steels and stainless steel will fail prematurely if exposed under tensile stress to absorbed atomic hydrogen. This is known as hydrogen embrittlement. Similarly, when aluminum alloys are exposed to liquid mercury, they are likely to crack prematurely if tensile stresses are present This is an example of liquid metal embrittlement. Silicate glass cracks in the presence of water, a phenomenon known as static fatigue of glass. Plastics fail prematurely in the presence of organic solvents. Service environments that contain soluble chlorides may lead to premature transgranular cracking of austenitic stainless steels. Interestingly, the same environments will not cause such failure in ferritic stainless steels. Likewise, caustic solutions are known to promote the premature failure of carbon steels and austenitic stainless steels.

All of the above phenomena are described as environmentally-induced embrittlement or environmentally-induced cracking, EIC. These phenomena have been known for a very long time. Materials engineers are fully able to make materials selection decisions for the construction of engineering systems that see service in virtually any environment. What is also not known with certainty at this stage is the mechanism by which these examples of environmentally-induced embrittlement occur. There are multiple schools of thought regarding the mechanisms of each phenomenon mentioned above. As such, it is not uncommon to find such phenomena described broadly as stress corrosion cracking even though it is clear that corrosion is not a general prerequisite to such phenomena. It should be noted that in many cases of EIC, the alloy is virtually corrosion-free over most of its surface, including the fracture surface. As such, chemical or electrochemical dissolution has minimal if any effect on the fracture process.

There has been an enormous amount of effort directed toward identifying the mechanism or mechanisms of environmentally-induced cracking, but there is wide disagreement and debate on this subject. Most involve in some fashion either the adsorption of specific embrittling species and subsequent lowering of the surface energy for fracture or localized anodic electrochemical processes such as dissolution or film formation. Our goal in this presentation is to assess the current state of knowledge of EIC in terms of the above phenomena, to identify what is known in a mechanistic sense and what remains to be understood in terms of the path forward toward a more complete mechanistic understanding.

MICROMECHANISMS OF FRACTURE PROPAGATION IN GLASSY POLYMERS

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Key Words: creep fracture, glassy polymers, atomic force microscopy, modeling toughness

While most glassy polymers are nominally brittle at macroscopic scales, they are known to exhibit plastic deformation in indentation, scratching, and microcutting when the loaded region is sufficiently small. The same applies to the micrometer size process zone at the tip of a propagating crack. While the presence and approximate size of this microscale plastic zone is well described by the Dugdale model, the prediction of the toughness of these materials is not possible without accounting for the details of the local large strain field and the work hardening behaviour of these polymers, which can be inferred from their response to compressive tests. Strain localization mechanisms such as crazing or shear banding should also be taken into account to properly model toughness. Finally, viscoplastic creep plays a major role in determining the dependence of the toughness on crack propagation velocity, as well as the important difference between the initiation and propagation toughness, which is responsible for the occurrence of a characteristic stick-slip propagation under some loading conditions.

We present here the important insights that can be obtained from an in-situ experimental investigation of the strain fields in the micrometric neighbourhood of a propagating crack. Atomic Force Microscopy combined with Digital Image Correlation reveals to be a very rich technique for our aim, although it limits our observations to the external surface of the sample and to very slow crack propagation (below nm/s). As an example, Figure 1 reports the very different look of fractures propagating at similar slow velocities in PMMA and epoxy resin.
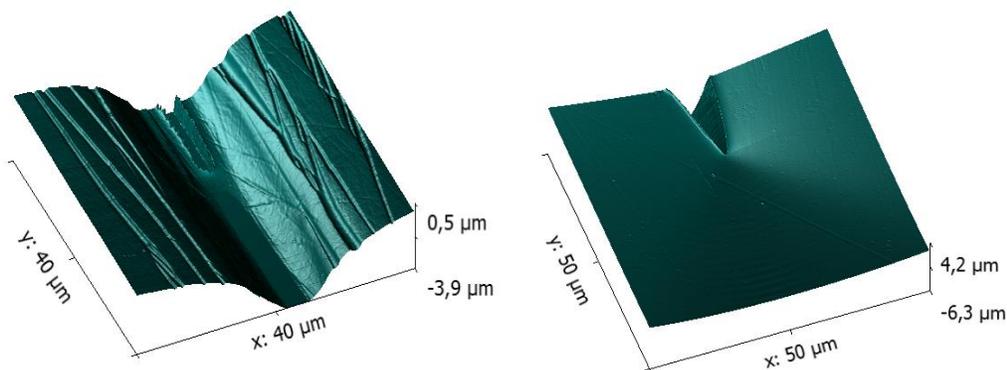

Figure 1: How the crack tip looks at micro scale in PMMA (left) and epoxy resin (right). AFM topographic images of the external sample surface with a vertically striking crack slowly propagating in the y direction (NB: unitary aspect ratio).

The consequences of these observations on understanding the toughness of glassy polymers and the influence of the environment will be discussed, as well as their implication on the fracture behaviour of these polymers in very confined conditions such as when used as a matrix of a long fiber composite material.

ENVIRONMENTALLY ASSISTED FATIGUE OF SUPERELASTIC NiTi

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Key Words: NiTi, Shape memory alloy, Tensile fatigue, Hydrogen, Electrochemistry

Superelastic NiTi implants transforming cyclically in body fluids suffer from fatigue failures which are extremely difficult to predict. This clearly points out towards environmental effects promoting surface dominated fatigue degradation. The specialty of phase transforming NiTi shape memory alloy is that either the parent austenite or the product martensite phase exist at the excessively deforming metal/liquid interface covered by the thin TiO_2 surface. In order to explore the environmental effects at such mechanically active metal/liquid interface, we have developed dedicated electrochemical apparatus and methods combining electrochemical cell, mechanical tester and thermal chamber. We are able to follow and/or control the mechanically triggered periodical breakdown/passivation process on the metal/liquid interface occurring during cyclic tensile tests on NiTi wires and springs in fluids. In this way we are able to analyze the effect of surface finishing treatments on fatigue performance and/or control it electrochemically. In this talk, we will introduce two in-situ electrochemical methods especially open circuit potential and potentiostatic polarization applied during fatigue testing. We will focus on the problem of non-stationary thermodynamic equilibrium established at the mechanochemically loaded wire surface. Kinetics of the surface reactions encountered during this type of environmental fatigue testing will be revealed. SIMS depth profile analysis and chemical imaging of the surfaces of fatigued wires was employed to prove the assumed electrochemical activity upon cycling, particularly to the hydrogen absorption and growth of passive oxide layer within cracks. Microcracks forming on the surface of fatigued wires were observed by 3D SEM/FIB sectioning method. Based on the results, mechanisms of environmental fatigue degradation of NiTi implants deforming cyclically in body fluids will be proposed.

COMPETITION OF STRESS CORROSION CRACK BRANCHES OBSERVED IN-SITU USING TIME-LAPSE 3D X-RAY SYNCHROTRON COMPUTED TOMOGRAPHY

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Key Words: Stress corrosion cracking, in situ x-ray synchrotron computed tomography

The progress of a stress corrosion crack in a sensitized AA7075 alloy was studied by in-situ x-ray synchrotron computed tomography. A load was applied to a pre-cracked specimen inside an environmental cell containing moist air and the propagation of the stress corrosion crack was observed. Measurements from the 3D image of the crack have already been shown to provide better quantification compared to observations of the crack from the outer surface. In this paper we study in detail the progress of the stress corrosion crack as it propagates through the material. We reveal how the formation of metal ligaments occurs and the competition of the 'main' crack and its branches. We have visualized these features to show the complexity of the local variation in crack morphology in a way that brings new insight into the interaction of the stress corrosion crack with the microstructure of the material.

PREDICTING FATIGUE CRACK INITIATION IN METALS USING DISLOCATION DYNAMICS SIMULATIONS

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The presented work aims at deepening the understanding of the initiation of fatigue cracks in metals. The work is based on an argument of Mura and Nakasone [1] where an energy criterion is used to predict the initiation of a fatigue crack from a slip band. The discussion is based on the evolution of dislocation networks, as these are the prime cause for permanent deformation in metals. Using high performance computing, 3D dislocation dynamics simulations [2] are performed over several cycles to study the growth of the dislocation arrangement. Then the evolution of energy in the system is determined, including all relevant terms such as: energy of the elastic field of the dislocations and their interaction, core energies, dissipation, energy stored in the continuum and external work. A hypothetical crack is placed in the region of the largest dislocation density and it is checked, if the energies stored in part of the dislocation network should be exchanged with the surface energy of a crack to lower the overall energy state of the system. The size of the crack is based on the number of dislocations that were previously formed in the network, as the motion of these dislocations towards the hypothetically formed free surface would form the actual crack. The presented work requires only minimal input in form of elastic constants and dislocation mobilities (for example from molecular dynamics). Results are presented for different materials (Cu and Mg), grain sizes and loading rates.

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ROLE OF DEFECT INTERACTIONS DURING HYDROGEN EMBRITTLEMENT IN IRON: A MULTISCALE PERSPECTIVE

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Hydrogen embrittlement (HE) is a phenomenon that affects both the physical and chemical properties of several intrinsically ductile metals. Consequently, understanding the mechanisms behind HE has been of particular interest in both experimental and modeling research. Discrepancies between experimental observations and modeling results have led to various proposals for HE mechanisms. Therefore, in this work we systematically examined the effect of hydrogen on two fundamental HE mechanisms in iron, namely, adsorption induced dislocation emission (AIDE) and hydrogen-enhanced decohesion (HED). In this work, we used density functional theory, atomistic simulations, and continuum Rice-Thompson criterion to systematically investigate: a) the incipient event ahead of a crack tip in single crystals subjected to mode-I loading conditions; b) the cohesive strength of grain boundaries; and c) the energy barrier for a slip transmission across the grain boundary. We observed that the presence of hydrogen 1) reduces the stress intensity factor required for both the dislocation nucleation and the cleavage response for different crack orientations, and 2) increases the energy barrier for slip transmission. Most importantly it was found that the AIDE and HED mechanisms are acting together to cause HE.

SCC CHARACTERIZATION OF AL-LI-CU-X ALLOYS

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In the past several decades many Al-Li-X alloys have been studied to understand the fracture properties due to its superior specific strength and modulus over the currently dominated 2024 & 7075 type aircraft structural alloys. It is also observed that their stress corrosion resistance is also good in the peak aged condition depending on the (Li/Cu) ratio. To understand its behavior with respect to its heat treatment, a systematic collection of data is required from the published literature.

We have collected such systematic data for three alloys from the early 1980's on Al-3Li, Al-2Li-3Cu and Al-1.5Li-4.5Cu, all with 0.12Zr to keep the grain structure predominantly un-recrystalized. Such alloys were tested in the S-T direction in 3.5%NaCl solution to obtain the plateau velocities (da/dt) and threshold KIscc. It is observed that in all the alloys, (da/dt) decreased with aging heat treatment time, the decrease increased with (Li/Cu) ratio. This decrease is interpreted in terms of the degree of anodic dissolution of the grain boundary precipitates. The results are compared with the SCC behavior of 7050 and 2024 alloys. Additional data where reversion aging treatment was given shows that grain boundary precipitates play an important role in the SCC behavior. The overall trend in the SCC behavior is discussed in light of the current understanding of the aluminum alloy behavior in terms of anodic dissolution and the role of hydrogen.

THE EFFECT OF COMPOSITION, TEMPER, AND CRACK ORIENTATION ON THE STRESS CORROSION CRACKING BEHAVIOR OF Al-Mg ALLOYS

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Key Words: Stress corrosion cracking, aluminum, temper, composition, fracture mechanics

Non-heat-treatable Al-Mg alloys are used in modern marine applications, however these alloys are susceptible to intergranular corrosion (IGC) and intergranular stress corrosion cracking (IGSCC) following the precipitation of an active β phase (Al3Mg2) along the grain boundaries. The precipitation of β (sensitization) can occur during in-service thermal exposure of marine components (at temperatures as low as 50°C); the degree of sensitization (DoS) is typically characterized by nitric acid mass loss testing (NAMLT). Recent efforts quantitatively established the IGSCC susceptibility of 5083-H131 in NaCl via fracture mechanics-based slow-rising displacement testing where crack extension is monitored via high fidelity direct current potential (dcPD) techniques [1,2].   The effect of DoS and applied potential on the threshold stress intensity for SCC (KISCC) and the stage II crack growth rates (da/dtII) were established and interpreted in the context of varying crack tip conditions influencing a H environment assisted cracking (HEAC) mechanism [1,2]. This research extends this methodology to quantitatively evaluate the influence of different engineering relevant material compositions (5083 and 5456) and tempers (H131, H116, and solution heat treated and quenched) on the KISCC and da/dtII; such data are interpreted in the context of proposed HEAC damage mechanism. Furthermore, the effect of continuity and tortuosity of the susceptible grain boundary path is evaluated by investigating the IGSCC behavior for different crack orientations (SL and TL) and degrees of grain size isotropy.
Testing for each material was conducted at two DoS (22 and 40) values. Consistent with the slight variation in composition, the 5083 was shown to be only slightly more susceptible than 5456 at a constant temper. However, for a constant composition the temper demonstrated a more pronounced influence with H131 being the most susceptible followed by H116 then SHTQ. These trends are analyzed based on (1) strength differences between the tempers, (2) Mg content changes, and (3) inaccuracies of DoS in capturing the true degree of and morphology of β precipitation on the grain boundaries. A strong influence of grain orientation is observed where SL oriented cracks are significantly more susceptible than TL samples. Fracture surface analysis of the TL samples show significant degree of fissures parallel to the loading axis. The beneficial effect of this delamination toughening on the HEAC mechanism is rationalized through decohesion-based micro-mechanical models where the loss of constraint decreases the local hydrostatic stress, thus (1) lowers the local H concentration in the process zone and (2) reduces the local tensile force that drives grain boundary decohesion.

The focus of this research effort on systematically quantifying the SCC behavior (KISCC and da/dtII) of engineering relevant Al-Mg alloy system and interpreting these data in the context of the microstructure and prominent failure mechanisms is directly aligned with the stated goals of the conference.

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EFFECT OF GRAIN BOUNDARY MICROSTRUCTURAL FEATURES ON THE FRACTURE BEHAVIOR OF AL-LI ALLOYS

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Commercial Al-Li-Cu alloys are highly desirable candidate for aircraft components because of their lower density, higher stiffness and high strength as compared to conventional Al alloys. These alloys, however, have low fracture toughness and exhibit brittle intergranular fracture. It is, therefore, important to understand the fracture behavior to design better Al-Li alloys for future aircraft systems. The critical issue is to understand the nature of grain boundary phases and their interfacial characteristics, as these characteristics dictate the fracture toughness of Al-Li alloys, and also to determine the localized dissolution characteristics of grain boundary intermetallic phases at nanoscale. Using transmission electron microscopy (TEM) and density functional theory (DFT) simulations, we recently demonstrated for Al-Cu-Li alloys the failure is likely to occur at the grain boundary T1/matrix interfaces, as these interfaces have low decohesion energies. It was reported that the toughness of the third generation Al-Li alloys has been significantly improved by reducing Li content and by adding small amounts of Ag and Mg. There are unresolved questions as to how Ag and Mg modify the interfacial characteristics of grain boundary/matrix interfaces. We will discuss the microstructure and interfacial characteristics of grain boundary precipitates/matrix interfaces, and correlate with the observed fracture behavior of third generation Al-Li alloys.

THE EFFECT OF CORROSION MORPHOLOGY ON THE FATIGUE INITIATION AND SMALL CRACK GROWTH BEHAVIOR OF AA7050-T7451

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Key Words: Corrosion-fatigue, initiation, small crack growth, aluminum, aerospace

Complex airframe structures often require the use of stainless steel fasteners to assemble/join aluminum substructures. A galvanic couple is created when surface coatings/sealants are breached enabling ingress of an electrolyte; this leads to corrosion damage at these inherently high stress joints. Recent US Air Force studies have demonstrated that corrosion nucleated fatigue damage represents roughly 80% of airframe fatigue damage initiation sites [1]. Despite the critical importance of this failure mode the interaction of the mechanical and electrochemical interactions for a realistic galvanic couple configuration are poorly understood. This talk will report on a collaborative effort that aims to quantify the local galvanic environments, quantitatively characterize the corrosion morphology associated with such environments, and evaluate how such morphologies influence the fatigue behavior of a modern aerospace aluminum alloy. The primary focus will be on quantitatively evaluating the macro-features, micro-features, and microstructural interactions that govern the crack formation behavior and how the relative influence of each varies with different corrosion morphologies produced using electrochemical conditions pertinent to an in-service galvanic couple.

Leveraging collaborator inputs from experimental and computation analysis of the electrochemistry of a representative galvanic couple, three corrosion morphologies are considered: discrete pitting (small and large scale), a broadly corroded surface with surface recession and intergranular corrosion (IGC). Each damage morphology is induced on the SL surface of the AA7050-T7451 fatigue samples. Optical microscopy, white light interferometry, and x-ray computed tomography (XCT) are used to characterize the features of the corroded specimens. XCT is also used to identify the location of underlying constituent particles. Corroded specimens are fatigue loaded (σmax of 200 MPa, R of 0.5 and f of 20 Hz) along the L-direction in a high humidity (RH>90%) that is maintained inside a plexiglass chamber. A programmed fatigue loading sequence is used mark the crack front intermittently on the fracture surface of the specimens; these fatigue-sequence induced marker bands are analyzed using the scanning electron microscope to quantify crack formation location and life ((N_i) to ≈10 µm) and crack growth rates (da/dN). Once fractography is complete the fracture surface is polished (roughly 15 µm deep) and electron back-scatter diffraction analysis is performed to enable characterization of the microstructure proximate to the crack formation site and how it intersects the growing small crack.

Overall fatigue life results show a substantial and similar reduction in fatigue life due to each of the corrosion morphologies; markerband analysis demonstrates that this strong reduction is primarily due to a vast decrease in the crack formation life. Similar small crack growth rates are observed proximate to each of the corrosion features. Analysis of the macro-features of the corrosion morphology show no clear trend between crack formation sites and the damage depth, width, 3D volume, density, or proximity to surrounding damage. Furthermore the similarity in the crack formation life between different morphologies suggests that the micro-features associated with each damage type results in a similarly deleterious local condition for crack formation. The proximity of local constituent particles and the local grain orientations are evaluated to determine if there is commonality between the crack formation location and a consistent microstructure feature(s).

The results and conclusion of this effort will quantitatively characterize the crack formation behavior of a relevant aerospace Al alloy in realistic conditions and leverage this data to further the mechanistic understanding of the factors governing the corrosion to fatigue crack transition. This understanding it critical to inform engineering scale prognosis strategies and provide guidance on the critical criteria for designing corrosion mitigation strategies in the context fatigue damage.

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MICROSTRUCTURAL AND ENVIRONMENTAL EFFECTS ON STRESS CORROSION AND CORROSION FATIGUE OF 7075 ALUMINUM ALLOY

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The design and development of high performance structural materials requires a thorough understanding of the relationship between environment, mechanical stresses, microstructure, and properties. The corrosion and fatigue behavior of aluminum alloys is greatly influenced by environment and precipitate structure. A comprehensive, mechanistic understanding of the role of environment on cyclic fatigue of Al alloys is needed. The relationship between environmental and mechanical effects is not well understood. The driving force at the crack tip is clearly a combination of chemical and mechanical processes operating together. A synergy between these processes is also present.

In this talk, the role of moisture on stress corrosion and corrosion-fatigue of 7075 Al alloy will be presented. Rolled 7075 Al alloy was heat-treated to underaged, peak-aged, and overaged conditions. To investigate the effects of corrosion and fatigue on peak-aged 7075 aluminum alloy, corroded samples were tested via *in situ* x-Ray tomography. The samples were mechanically polished, then soaked in covered 3.5 wt.% NaCl for fifteen days to allow for significant corrosion to occur. Then, they were fatigue tested in situ in 3.5 wt.% NaCl using synchrotron x-ray tomography to analyze the fatigue crack initiation and growth characteristics. Hydrogen bubbles were observed between the sample and the fluid upon crack initiation, indicating chemical changes in the sample during in situ corrosion fatigue. The effect of oxide layers forming during corrosion and 2nd phase inclusions, on fatigue initiation and propagation, will be discussed. The microstructure and morphology of the fracture surfaces were examined by scanning electron microscopy (SEM) and correlated with the crack growth behavior. The crack initiation, growth, and damage were also quantified by sophisticated three dimensional (3D) *in situ* x-ray synchrotron tomography technique. This technique provided interesting insights into the onset of crack initiation and growth

MECHANICAL & CHEMICAL DRIVING FORCE AFFECTING CRACK NUCLEATION

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Load history has been known to affect fracture and stress corrosion behavior. The degree to which it affects depends on the severity of the load history. It is known that shop peening can retard the SCC lives markedly in steels. Similarly, prestraining can reduce the KIscc and plateau velocity in high strength steels.

These types of experiments are difficult to quantify their effects on the SCC behavior. One can analyze the prestarining effects in a better way by analyzing the effects of single overloads followed by constant applied load to study the behavior. Such experiments can be done by observing the 'incubation time' for a crack to initiate in a fatigue precracked sample, at various constant applied loads in a chemical environment. Such experiments have been conducted on 7075 aluminum alloy and a 4130 steel. It is observed that results are similar in behavior. The data indicates the overall behavior can be analysed by suggesting that the total stress at the crack tip is related to the contributions from chemistry of the environment and an additional factor from "internal stress" Hence we can describe the overall data in terms of:

$$K_{Iscc} = K_{applied} + K_{internal\ stress} + K_{environment}$$

Such trends in the behavior, has been observed in prestrained steel alloys prior to environmental exposure. The general behavior suggests that the internal stress affects the threshold K_{Iscc} more than the plateau velocity. The general SCC behavior is affected by both chemistry and internal stress under external static or cyclic loads.

RATE-CONTROLLING PROCESSES DURING ENVIRONMENT-SENSITIVE CRACK PROPAGATION IN ALUMINUM

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Key Words: Stress corrosion cracking, crack branching,

Recent experimental findings are challenging today's conventional view on the rate-controlling processes during environment-sensitive crack growth in aluminum alloys when exposed to moist air and aqueous environments. X-ray computed tomography has revealed the detailed crack morphology of several stress corrosion cracks in 7000 series alloys and this has shown that the complexity of the mesoscale is incredibly important to understand the links between the gross morphology of the crack and the crack front/tip. We will show the large local variation that exists in the crack morphology. At the same time we will show how average measurements of crack velocity and crack opening displacement remain surprisingly uniform across the width of the crack. Discussion will follow in an effort to quantify the effect of Keffective compared to Kapplied and to provide a rationalization of these findings with respect to previously published theories.

SOLUTION CONDUCTIVITY DEPENDENT CRACK SIZE EFFECT IN STRESS CORROSION CRACKING AND CORROSION FATIGUE

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The chemical crack size effect on environmentally assisted crack growth was first demonstrated experimentally by Gangloff [1] and supported on a more robust theoretical framework by Turnbull et al. [2,3]. It is probably better dubbed the electrochemical crack size effect since the potential drop in the crack was a critical factor in determining the solution chemistry and the sensitivity to crack size. In recent experimental studies [4] we have focused on the growth rate of small and long stress corrosion and corrosion fatigue cracks in 12Cr steam turbine blade steels in low conductivity water containing 35 ppm Cl- (simulating upset steam condensate chemistry). A large effect of crack size on growth rate was observed for the same mechanical driving force. However, the crack-size effect disappeared in lower conductivity solution, 300 ppb Cl- and 300 ppb SO42- (corresponding to normal steam condensate chemistry). Furthermore, corrosion fatigue long crack growth rates were the same in aerated and in deaerated solutions for the two environments but stress corrosion cracks arrested in deaerated solution. An explanation for these varied results will be presented based on the concept of the solution conductivity dependent crack size effect and its impact on potential drop and the crack-tip potential. To underpin this conceptual idea and to explore further the scale of this effect for varied crack size and solution conductivity combinations, modelling of crack electrochemistry is being undertaken and the preliminary results will be discussed.

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ENVIRONMENTAL ASSISTED CRACKING OF PIPELINE STEELS IN CO2 CONTAINING ENVIRONMENT

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Buried pipelines are susceptible to Environmental Assisted Cracking (EAC) in three different conditions: Stress Corrosion Cracking in the presence of Carbonate-Bicarbonate (CB-SCC), Near Neutral Stress Corrosion Cracking (NN-SCC) and Hydrogen Embrittlement (HE). Both CB-SCC and NN-SCC involve in their mechanism the presence of CO_2 and/or its dissociated species dissolved in moisture under the coating, while the generic term of HE refers to the phenomena of brittle fracture taking place for entry of atomic hydrogen inside the metal lattice, owing to the applied cathodic protection.

Historically the CB-SCC was the first established form of stress corrosion on pipelines in the 60s, while Trans Canada Pipeline published in 1985-86 the first case of NN-SCC. The initiation conditions and the mechanism of propagation of CB-SCC were well established by means of the works of Parkins in 70ths. Many studies were carried out on NN-SCC, but actually there is not an exhaustive understanding of the problem.

This paper summarizes the results obtained by authors on pipeline steels in NN-SCC promoting environments. Constant load, constant deformation, slow strain rate, slow bending and corrosion fatigue tests were executed on different grades of pipeline steels. The obtained results pointed out the effect of continuously plastic deformation to observe the propagation of NN-SCC cracks. NN-SCC cracks are preferentially nucleated from localized attacks. Inside the pits, the decreasing of pH enhances the hydrogen ions reduction. The results of slow strain rate tests are in agreement with a hydrogen embrittlement mechanism for the NN-SCC cracks propagation.

Electrochemical tests, potentiodynamic and cyclic voltammetry were also carried out in order to analyze the effect of temperature, pH, CO_2 and bicarbonate concentration on the pitting initiation.
A pre-corrosion procedure, using a solution with high concentration of bicarbonate ions saturated with CO_2 and a great number of voltammetry cycles was developed to obtain localized attacks on the specimen surface, similar than those observed in the failure analysis of the cracked pipelines. The SSR and slow bending tests carried out on these pre-corroded specimens evidenced presence of cracks with the same morphology of NN-SCC.

Finally some corrosion-fatigue tests carried out on linear elastic fracture mechanics specimens (Single Edge Notch three four point Bending specimens) evidenced the increasing of fatigue propagation crack growth in NN-SCC environment.

In the range of traditional and innovative pipeline steels, the mechanical properties (ultimate tensile strength and yield strength) seem do not influence their NN-SCC resistance.

MATERIAL WEAKENING DUE TO CORROSION IN HARDENED BEARING STEELS

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Key Words: Corrosion, hydrogen, rolling contact fatigue

In rolling and ball bearings, materials are designed to withstand extremely high loads for long operation times. Most bearing steels are hardened steels with high carbon content that typically can function up to the Very High Cycle Fatigue regime (>1010 load cycles). The loads under which this occurs are given by the fatigue limit, which is a parameter that does depend on the strength of the material, but is commonly seen as a fixed parameter Therefore, material weakening is playing an important role when components fail as a result of rolling contact fatigue loading. In this paper, the effects of corrosion and hydrogen generation on the material weakening of hardened ball bearing steels are described. The results of corrosion exposure tests, fatigue tests with corrosion and tests with increased hydrogen levels in steel on mechanical properties will be presented. The role of hydrogen in bearing steel will be discussed on basis of the atomistic processes that play a role in rolling contact fatigue.

ELECTROCHEMICAL STRESS INTENSITY APPROACH TO MODELING GALVANIC COUPLING AND LOCALIZED DAMAGE INITIATION IN STRUCTURES

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Keywords: Aircraft Structures; Corrosion; Fatigue; Stress Corrosion Cracking

Traditionally, airframe structures are designed for immediate mechanical performance and loads-only structural response; the lifetime of aircraft structures is predicted on these analyses and environmental degradation of properties over the life cycle and during operations is often an afterthought. Although the maintenance of aircraft structures is primarily determined by material degradation, galvanic management of airframe designs and corrosion resistant material selection has never been done systematically. From end of life tear-down inspections, we know that, predominantly, structural failures are initiated from corrosion features, especially those accelerated by dissimilar material coupling. In its most simplistic form, this environmental exposure, "loading", creates corrosion features, such as pitting, that produce crack initiation morphologies, cracks nucleate from these features and then grow under the combined influence of mechanical stress and corrosion, eventually leading to structural failure. There is clearly a strong correlation between corrosion and structural damage, which we think of as corrosion fatigue and stress corrosion cracking. We propose that it is possible to treat "electrochemical stress" mathematically in a similar way to mechanical stress, with numerically equivalent approaches.   Using such a model, the combined influence of electrochemistry and stress can, in principle, be treated as the sum of these two stresses, allowing us to develop models to predict the risk of environmentally assisted fatigue and stress corrosion cracking damage.

ONR's Sea-Based Aviation program is developing computational approaches to corrosion activity prediction, crack initiation, and crack growth, with the ultimate aim of predicting service life in terms of the combination of mechanical and chemical stress. This approach is intended to be the basis for design of durable aircraft structures, using design principles that will take into account both stress and corrosion in the design phase, rather than designing for stress and then maintaining for corrosion.

RELATIONSHIP BETWEEN ELECTROCHEMICAL REACTION PROCESSES AND ENVIRONMENT-ASSISTED CRACK GROWTH UNDER STATIC AND DYNAMIC ATMOSPHERIC CONDITIONS

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Key words: Atmospheric corrosion, thin film electrolyte, occluded cell

Environment-assisted cracking (EAC) of aluminum alloys in corrosive atmospheres is an important maintenance and safety issue for aerospace structures. EAC initiation and propagation are influenced by the interaction of load, environment, and alloy properties. For atmospheric corrosion, environmental conditions are dynamic; where temperature, relative humidity, and surface contaminants interact to control thin film electrolyte properties. Recent studies have determined that stage II stress corrosion crack (SCC) propagation, under atmospheric conditions is strongly dependent on humidity. SCC propagation in AA5083 and AA7075 alloys is a maximum during drying processes at intermediate humidity levels at or below the deliquescence relative humidity (DRH) for the applied corrosive salt. An improved understanding of the dependence of cracking on atmospheric conditions is important to testing material performance, establishing durable designs, and managing structural integrity.

Electrochemical testing is typically performed in immersion cells where the bulk electrolyte volume is large enough to assume solution chemistry is relatively constant throughout a test. These bulk electrolyte assumptions are not satisfied for atmospheric corrosion processes (Figure 1). Salt concentrations on surfaces are dependent on relative humidity and may be different for wetting and drying cycles as affected by the DRH and efflorescence relative humidity (ERH) of surface contaminants and corrosion products. In the case of localized corrosion and EAC mechanisms, separation of the anode and cathode may occur due to variation of chemical composition, oxygen availability, and pH differences between the crack tip, crack mouth, and boldly exposed surfaces. Because the electrolyte volume-to-surface area ratio for atmospheric corrosion is much lower than in bulk immersion conditions, reaction processes can alter the solution chemistry even under static environmental conditions. For aerospace structures and components, the area, location, and reaction kinetics continuously change during atmospheric corrosion due to natural diurnal cycling or operating conditions.
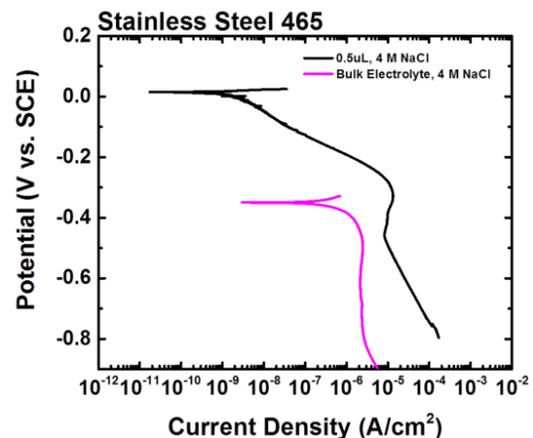

Figure 1. Comparison of cathodic polarization curves on stainless steel in bulk 4 M NaCl and in a 0.5 μL droplet.

An improved understanding of the processes that are significant to EAC of aluminum alloys has been pursued by conducting electrochemical tests to quantify the interaction of crack tips, crack mouth, and boldly exposed surfaces under thin film electrolyte conditions. These tests are being performed with zero resistance ammeter (ZRA) techniques that couple occluded cells to boldly exposed samples with controlled surface area density of salt. The variation and distribution of currents under statically controlled and dynamic environmental conditions are compared to stress corrosion cracking tests under similar conditions. The stress corrosion crack (SCC) velocity is measured using mixed mode (mode I and II) loading of aluminum alloy four-point bend samples to produce long, S-L, stage II cracks ($\approx$20 mm) in controlled atmospheric test chambers. The velocity is calculated using sample compliance and continuous displacement measurements. By performing atmospheric electrochemical tests in conjunction with crack velocity experiments, the significance of static and dynamic environmental parameters and electrochemical processes are related to instantaneous crack velocities in important engineering alloys.

EFFECT OF SENSITIZATION ON THE STRESS CORROSION CRACKING OF AA5083

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Stress corrosion cracking of AA5083 was evaluated using both slow strain rate testing, there was a strong effect of sensitization with aging time. Increasing aging time leads to a sharp decrease in strain to failure, as well as ultimate tensile strength (UTS). In the fully sensitized condition (175°C/240h) there was a significant effect of chloride concentration, with increasing chloride concentration leading to a sharp decrease in strain to failure and UTS. However, in the as received condition there was no significant effect of chloride concentration on the slow strain rate behavior. The difference in behavior is likely associated with continuous precipitation of β phase along the boundaries. Rising displacement tests were performed as a function of sensitization time and the Kth measured decreased sharply with aging time reaching ~4MPa√m at 240h. The effect of chloride concentration and applied potential in the sensitized condition (175C/240h) was evaluated and the Kth decreased with increasing chloride concentration. At applied potentials below the breakdown potential of β there was an increase in the Kth but the values of Kth were not as those observed in the as received condition. The observations made suggest that the stress corrosion cracking of AA5083 was sensitive to the applied potential and chloride concentration which in turn control the crack tip conditions. The observed data were analyzed in terms of a crack tip strain rate model to quantify the behavior of AA5083.

PRE-EXPOSURE EMBRITTLEMENT OF SENSITIZED ALUMIMIUM-MAGNESIUM ALLOY, 5083-H131

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Environment-sensitive fracture of aluminum-magnesium alloys containing above ~3 wt% magnesium historically has been considered under anodic-dissolution control. Information from more recent studies, however, suggests a hydrogen-related process is also often involved. Further evidence supporting the involvement of a hydrogen-related process of during Intergranular stress corrosion cracking (IGSCC) will be presented using information gleaned from smooth and pre-cracked test specimens, previously sensitized over a range of temperatures in both 'dry' and 'wet' conditions and then subjected to rising-load testing in a range of environments. A detailed evaluation of the IGSCC using X-ray computed tomography to provide 3-D images and ultra-high-resolution electron microscopy to characterize selected regions within intergranular stress corrosion cracks enables mechanistic insights.

INVESTIGATION OF ELECTROCHEMICALLY-INDUCED REPASSIVATION OF Al 7075-T6 AND Al 2024-T3 AS A FUNCTION OF APPLIED LOAD AND GALVANIC CORROSION

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Key Words: Electrochemistry, Repassivation, Al alloys, Stress Corrosion, Galvanic corrosion

The repassivation behavior of Al alloys 7075-T6 and 2024-T3 was investigated by means of pitting scan (PS) technique (Fig. 1) (Trueba, Trasatti, 2015). The effect of mechanical load and galvanic coupling was estimated by considering principally the electrochemical characteristics of the reverse curve, namely the pit transition potential (Eptp), the associated current density (iptp) and the steepness. The load levels explored were mostly below the elastic limit, using four point bent-beam (4PBB) stress-corrosion test specimens (ASTM G39-99). CRES 304 or Ti6Al4V alloy were physically joined with Al sheet for simulating stress-induced galvanic corrosion. Different experimental variables (e.g. irev, [Cl-], pH, scan rate, etc.) were also considered. The study was complemented with corrosion morphology analysis.

Among the electrochemical characteristics, the steepness varied more importantly with the applied stress ($\square$), though prevalently in a complex manner. That is, for some conditions, the steepness -$\square$ plots showed a "zig-zag" like shape, as illustrated in Figure 2 for Al 7075-T6. The results suggest that the steepness, associated with the film rupture - dissolution events after the repassivation onset, is sensitive to stress gradients induced by corrosion for a given load level (Hall, 2009). The relation of such trends with the rate of formation of poorly protective passive film at Eptp is suggested also from the analysis of iptp – $\square$ plots. The electrochemical behavior under conditions promoting stress-assisted galvanic corrosion was determined by the galvanic corrosion behavior of a given galvanic couple.
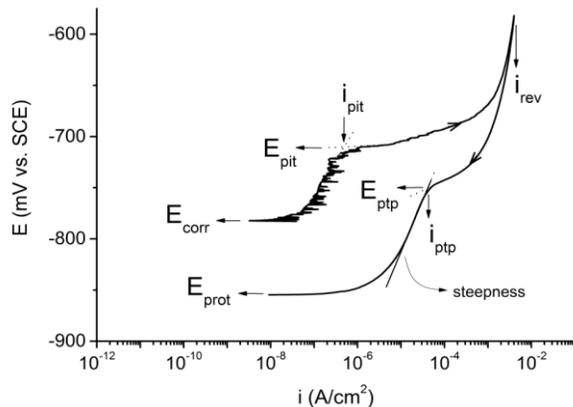

Figure 1: Pitting scan and characteristic electrochemical parameters: Epit and ipit – potential and current density of pitting onset; Eptp and iptp – potential and current density of the repassivation onset; steepness – potential decrease with current below the inflection; Eprot – protection potential; irev – current density limit of the forward scan;.
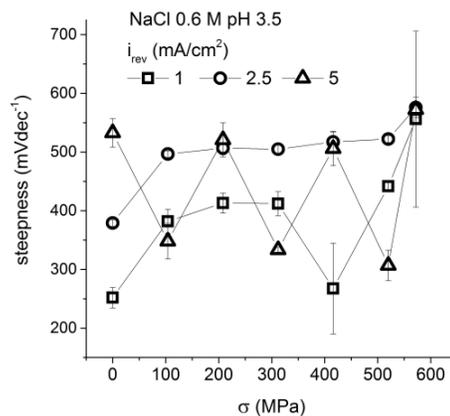

Figure 2: Variation of the steepness with the applied stress for different irev for Al 7075-T6 in 0.6 M NaCl, pH 3.5

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UNDERSTANDING SMALL CRACK EFFECTS ON FAILURE AND THRESHOLD DIAGRAMS

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It is well recognized that crack length has significant effects on both the monotonic failure stress and cyclic threshold stress conditions. Existing experimental data indicates that LEFM predicts correctly the failure and threshold stresses only for relatively long cracks. For small/short cracks, the LEFM overestimates both the failure and threshold stresses. Usually, an argument has been made that LEFM is violated in terms of a crack length with respect to the crack-tip plastic zone that results in overestimating the failure stress for small cracks. On the other hand, the Kitagawa diagram has been used to experimentally illustrate the connection between the observed threshold stresses for very small cracks and predictions from LEFM analysis.

To understand the failure stress diagram for small/short cracks, we introduce a plasticity correction in calculation of the effective critical stress intensity factor (KC eff). Earlier analysis showed that Irwin's correction of KC eff underestimates whereas the conventional Dugdale - Barenblatt yield-strip model overestimates the effects of the crack-tip plasticity on the failure stress for relatively small/short cracks. The rationale of a plasticity correction utilized in the failure stress diagram is extended to understand the Kitagawa diagram in terms of the observed threshold stress versus a crack size. This plasticity correction also explains the associated physics behind experimental observations in contrast to El Haddad's phenomenological curve fitting approach. Both type of diagrams demonstrate an analogy which governs interrelation between stress, crack/defect size and fracture mechanics analysis. Using the existing data from the literature, the proposed analysis of small cracks effects is discussed with relevance to monotonic and cyclic loading conditions, under inert and corrosion environments.

A NUMERICAL MODEL TO ASSESS THE ROLE OF CRACK-TIP HYDROSTATIC STRESS AND PLASTIC DEFORMATION IN ENVIRONMENTAL ASSISTED FATIGUE CRACKING

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Key Words: Hydrostatic stress, Hydrogen Embrittlement, Environmentally Assisted Cracking, Fatigue, Finite Element Mode

To better understand the mechanics of environmentally assisted cracking, and particularly hydrogen embrittlement, a correct description of the hydrostatic stress field is indispensable. The concentration of hydrogen in the proximity of the crack tip is indeed dependent of the hydrostatic stress effect on the microstructural lattice of the material. The overall parameters of the hydrostatic stress, including peak value, its location, gradient and distribution size are fundamental to assess the effect on hydrogen distribution near the crack tip. H concentration show indeed an exponential dependence on hydrostatic stress, so that even a moderate increase in the applied stress is traduced in a dramatic rise of hydrogen concentration. In order to investigate the mechanism beneath hydrogen embrittlement, a proper modelling of the hydrostatic stress is mandatory. Moreover, strain hardening might significantly interfere with the actual stress distribution, thus becoming a relevant parameter. The stress and strain field is also deeply affected by the presence of sharp notches. In the present work, a detailed Finite Element model, based on actual fatigue testing of notched Ti-6Al-4V specimens is proposed, providing a deeply informative tool to assess the hydrostatic stress and the plastic strain in the proximity of the crack tip. The data from the model are compared to available results and experiences in literature.
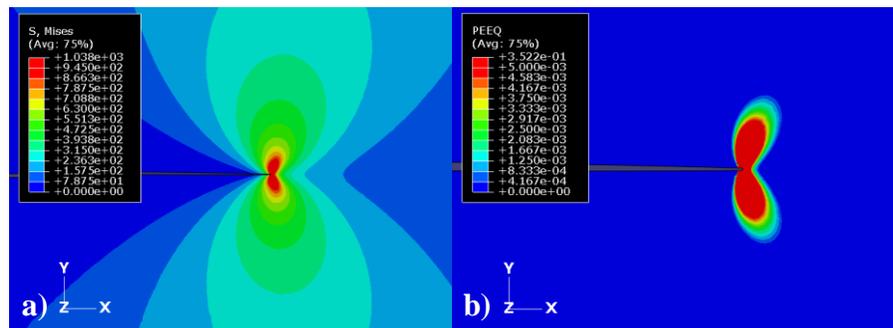

Figure 1 – FE simulation results: (a) von Mises Stress and (b) Equivalent Plastic Strain at the crack tip.
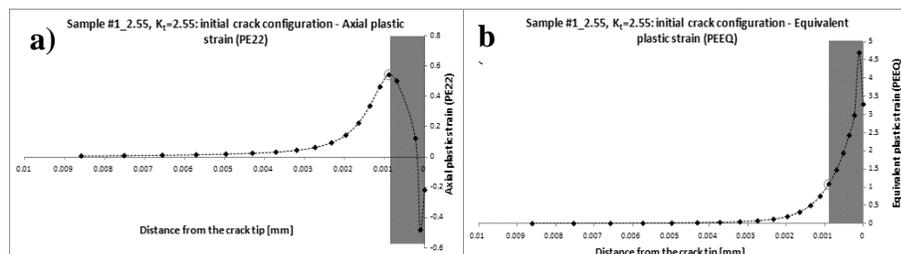

Fig. 2: Axial strain in the proximity of the crack tip for Kt = 2.55: (a) axial plastic strain and (b) equivalent plastic strain.

EXAMINATION AND PREDICTION OF CORROSION FATIGUE DAMAGE AND INHIBITION

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Key Words: Corrosion fatigue, fatigue crack growth inhibition, corrosion nucleated fatigue crack growth

The United States Department of Defense (DoD) estimated that the annual cost of corrosion, the unintended material degradation due to the environment, to weapon systems and infrastructure in 2010 exceeded $21 billion, and that the number was likely to continue to rise. Unfortunately, as the warfighters demand more from their systems, corrosion prevention and control is frequently traded during the acquisition cycle for weapon system performance. As a result, the DoD remains entrenched in a find-and-fix corrosion management philosophy which is expensive and unsustainable. Better standardized fatigue laboratory procedures are need to help the DoD develop (1) a fundamental understanding of corrosion damage, (2) material performance data relevant to corrosion damage, (3) prediction methodologies to help mitigate the effects of corrosion nucleated fatigue damage and (4) to develop an understanding of how corrosion preventative coatings can slow fatigue crack propagation. All of these research area lead to developing better damage prediction methods which can account for the effect of environment on fatigue crack propagation.

Aircraft structural tear-down analysis provides substantial evidence of the importance of corrosion in nucleating fatigue cracks, as such the ability to protect against such damage would be of great benefit to the DoD. While chromate and other inhibitors have been shown to slow fatigue crack growth rates when added to a bulk solution of a fully immersed sample, research has not been completed showing how these inhibitors effect crack nucleation from corrosion damage. In research funded by the Office of Naval Research (ONR) and the Office of the Secretary of Defense's Office of Corrosion Policy and Oversight (OSD-CPO) work is being completed to develop a better understanding how to better predict and prevent environmental effects on fatigue crack propagation.

While chromate has been used successfully for many years on United States Air Force (USAF) and Navy (USN) aircraft to prevent corrosion damage, the environmental and personnel risks associated with chromate have caused the DoD to pursue non-chromate containing corrosion prevention systems. To fully quantify chromate replacement coatings an understanding of the effects that chromate has on corrosion fatigue must be fully documented and understood. Some researchers have shown that high levels of inhibitors (chromate; molybdate) added to full immersion corrosion fatigue tests on aluminum alloys slow the fatigue crack growth rate substantially. The limitation of this research was that the amount of inhibitor present in the environment was not related to leach rates of chromate from polymeric coatings. For these inhibitors to slow fatigue crack propagation the inhibitors must become mobile from the polymer coating matrix. Based on this mechanism of corrosion inhibitor release, the examination of atmospheric corrosion fatigue, hydrated salt layers which better represent operating environments compared to full immersion testing, becomes important to help quantify how inhibitors affect fatigue damage in service.

Another focus area of the ONR funded research is in quantifying the corrosion damage to fatigue crack transition. A standardized specimen and testing protocol to evaluate the relative influence of material, environment, inhibitors, and loading spectrum on the pit-to-crack transition was developed. The methodology uses a narrow plate specimen with a centrally located hole with a preferential pit (diameter approximately 150 μm) placed at the corner of the hole; current work is being completed on legacy aluminum alloy AA7075-T651. The plate thickness and hole diameter are consistent with commercial and military airframe applications. Current DoD engineering approaches to manage corrosion-fatigue, are incomplete in terms of the effect of service environment. Poor component life prediction results in an increased flight line inspection and maintenance burden along with premature component retirement. The overarching objective of the research is to improve and transition the results on the effect of environmentally assisted fatigue in high performance metallic alloys (crack growth rate data) to the DoD research and depot maintenance activities by integrating all data into the AFGROW fracture toughness and fatigue crack prediction software allowing for the inclusion of corrosion damage and environment effects on fatigue crack life predictions.

UNIGROW-UNICORR FRAMEWORK - PROSPECTS FOR CORROSION MODELING AND SENSOR DEVELOPMENT

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In this brief, UniGrow-UniCorr framework for fatigue crack initiation, growth, and corrosion are discussed along with perspectives on modeling and sensor development for detecting on-set of corrosion damage. The UniGrow crack growth program is first described with brief discussion of its formulation, implementation, and validation results. Various features that are currently present in UniGrow software and planned extensions are also discussed. The current status and development plans for UniCorr to address stress-free and stress-assisted environment assisted cracking are then described.

Salient aspects of corrosion modeling are briefly described along with a description of a continuum damage mechanics model to study pit-to-crack transition life in aluminum alloys. Current development efforts that are underway to advance model prediction based on multi-physics study of corrosion fatigue damage mechanism by taking into account the microstructural features are described. In this context, coupled modeling of micro-plasticity and micro galvanic process is discussed to address the synergistic effect between stress and corrosion. The effects of intermetallic particles on pit initiation and growth mechanisms as well as stress effect on corrosion kinetics are described.

Requirements and challenges in appropriate sensor development especially for those intended as leave-in-place devices and for detecting on-set of corrosion crack initiation are outlined.