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14. ABSTRACT

The objective of this research is to quantitatively establish the governing crack tip mechanics conditions and damage mechanisms pertinent to environmental crack propagation. The central goals are: (1) to develop accurate predictions of crack tip stresses and plastic strains for incorporation into micromechanical descriptions of crack growth, (2) to validate crack tip mechanics models by high resolution experiments, and (3) to resolve physical characteristics of fatigue crack tip hydrogen damage.

Environment has a dominant and deleterious effect on fatigue crack propagation in airframe and engine components. Despite this known fact, a fundamental understanding of environment-enhanced fatigue remains elusive. We have recently examined in detail the characteristics of plasticity and crack path during environmental fatigue cracking in airframe aluminum alloys using state-of-the-art diffraction-based tools, nanoindentation and continuum mechanics modeling. For aerospace aluminum alloys, environmental fatigue crack advance most likely involves the interaction of environment-produced H, crack tip plastic strain accumulation, and local normal stress [1]. In particular, we focus explored the possibility that strain gradients elevate crack tip stresses to a level where significant H accumulation would be expected. The interaction of this H with the level of accumulated plasticity, and the crystallographic characteristics of the resulting damage were probed experimentally. The understanding developed within this program will advance the management of fatigue durability of airframe components; by providing guidance to future alloy development, chemical-environment control schemes, and fracture mechanics-based performance prognosis.

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**CONTROLLING ENVIRONMENTAL FATIGUE IN AEROSPACE AL ALLOYS
BY MULTISCALE CRACK TIP MEASUREMENTS AND MODELING
F49620-03-1-0155**

FINAL REPORT

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Executive Summary

Environment has a dominant and deleterious effect on fatigue crack propagation in airframe and engine components. Despite this known fact, a fundamental understanding of environment-enhanced fatigue remains elusive. We have recently examined in detail the characteristics of plasticity and crack path during environmental fatigue cracking in airframe aluminum alloys using state-of-the-art diffraction-based tools, nanoindentation and continuum mechanics modeling. For aerospace aluminum alloys, environmental fatigue crack advance most likely involves the interaction of environment-produced H, crack tip plastic strain accumulation, and local normal stress [1]. In particular, we focus explored the possibility that strain gradients elevate crack tip stresses to a level where significant H accumulation would be expected. The interaction of this H with the level of accumulated plasticity, and the crystallographic characteristics of the resulting damage were probed experimentally. The understanding developed within this program will advance the management of fatigue durability of airframe components; by providing guidance to future alloy development, chemical-environment control schemes, and fracture mechanics-based performance prognosis. This final report is organized as follows: Acknowledgements of the support received from the AFOSR and elsewhere, Research Objectives, Major Conclusions of each of the 3 Research Foci and Cited References, Educational Impact (Human Resource Development), and a List of Research Publications. The journal publications are attached to this document for your perusal.

Acknowledgment/Disclaimer

This work was sponsored (in part) by the Air Force Office of Scientific Research, USAF, under grant/contract number F49620-03-1-0155. The views and conclusions contained herein are those of the authors and should not be interpreted as necessarily representing the official policies or endorsements, either expressed or implied, of the Air Force Office of Scientific Research or the U.S. Government. The goals of this program were: facilitated by matching funds from the University of Virginia and leveraged against a related project sponsored by the National Institute for Aerospace Associates, Inc. (NIAA) and materials and support provided by the Aluminum Company of America (ALCOA).

Research Objective

The objective of this research is to quantitatively establish the governing crack tip mechanics conditions and damage mechanisms pertinent to environmental crack propagation. The central goals are: (1) to develop accurate predictions of crack tip stresses and plastic strains for incorporation into micromechanical descriptions of crack growth, (2) to validate crack tip mechanics models by high resolution experiments, and (3) to resolve physical characteristics of fatigue crack tip hydrogen damage.

1. Crack Tip Mechanics

The stress required to trigger decohesion, or even significant bulk H trapping that could ultimately lower the required decohesion stress, is higher than that predicted to occur at crack tips via conventional plasticity analysis, such as the Hutchinson-Rice-Rosengren (HRR) asymptotic and the so-called J_2 blunt crack solutions. The central question then arises: what mechanism is responsible for elevated crack tip stresses that are large enough to trigger decohesion associated with lattice planes or microstructure interfaces? Previous models focused on specific dislocation arrangements, such as a total absence of dislocations at the crack tip [2], super-dislocations [3], and recently dislocation dynamics simulations [e.g., 4]. However, it has been observed that materials can exhibit higher flow stresses in the presence of strong gradients in plastic strain which are accommodated by geometrically necessary dislocations. The present study examines the applicability of phenomenological strain gradient plasticity (SGP) models [5], implemented within a finite element framework, to the issue of H enhanced cracking. While pertinent to fatigue in aluminum alloys, this work is important to the whole of environmental cracking.

We developed a single parameter SGP-based finite element code and performed a parametric study to determine scaling relations between material constitutive parameters and loading conditions where the phenomenological approach is most likely to be appropriate [6], i.e., where the scale of the gradient affected zone, r^* , is large enough to justify the inherent homogenization requirements of the phenomenological approach and where this zone is of appreciable size with respect to the overall plastic zone size, r_p . An incremental flow theory implementation was selected, in which residual tolerances enforcing equilibrium and plastic consistency are strictly enforced. This enables future simulations of cyclic plasticity. A key conclusion of this initial study was that SGP effects are not relevant to many structural alloys (i.e. those with high yield strength and low toughness), because the physical length-scale over which stresses are different from HRR is too small to allow plausible application of continuum theories. Nevertheless, SGP-assisted fracture is likely relevant to softer materials with greater strain hardening. While this conclusion may seem obvious in light of previous results (which illustrated SGP effects are most pronounced for soft materials), the present calculations quantitatively assess the relevance of SGP to a specific material in terms of the affected length-scales, stress-levels, and crack-tip-openings.

In addition to the above theoretical research, nanoindentation was used to assess the characteristic length scale parameter l^* of AA2024 as a function of aging condition [8] using the method of Nix and Gao [9]. It was determined that l^* of AA2024 varies

between 50 and 200 nm, depending on aging condition; 100 nm is a good estimate for the technologically important T351 temper. By incorporating this finding into the single-parameter SGP model, it was shown that even at this very small distance from the crack tip (~ 100 nm), where continuum modeling is on tenuous footing, the predicted SGP stress elevation may be insufficient to explain the level of H accumulation required to explain environmentally assisted cracking. However, there were still outstanding issues (cyclic plasticity, blunting, and multiple length scale models) to be explored before dismissing the possibility that SGP models have a role to play in crack tip micromechanics and damage modeling.

The role of multiple material length-scale parameters in modulating crack tip stress fields and crack opening displacements has now been quantified [8] for a wide range of macroscopic hardening parameters and applied stress intensity factors. The calculations support the following general conclusions (a complete discussion can be found in [ref. 8]):

- Multiple length-scale theories that incorporate the full complement of gradient terms (corresponding to invariants of the strain gradient tensor) predict significantly larger SGP zones near crack tips than single length-scale theories, if it is assumed that $l_1 = l_2 = l_3$. There is an order of magnitude difference in the size of the elevated stress zone (relative to conventional plasticity) under such conditions.
- The material length-scale parameter associated with the first invariant of the strain gradient tensor [5], l_1 , has a dominant effect on crack tip behavior, as the extent and severity of SGP effects are most sensitive to this parameter. However, significant increases in l_2 can increase SGP-effects. The isolated variation of the third parameter, l_3 , has a moderate effect that does not produce changes as large as the variation of l_1 and l_2 .
- Strain gradient effects persist to distances larger than the length-scale parameter l_1 when $K_I / \sigma_Y \sqrt{l_1} > \sim 300$ for the case with $l_1 = l_2 = l_3$, and $K_I / \sigma_Y \sqrt{l_1} > \sim 15$ for the case with $l_2 = l_3 = 10l_1$. The generalizations are applicable to a wide range of strain hardening exponents; in contrast, the magnitude of stress elevation depends strongly on hardening exponent.
- The much larger extent and severity of SGP stress elevation from the multiple length-scale theory indicates a critical need to calibrate SGP models with indentation experiments, to determine whether or not previously inferred experimental length scales are indicative of l_1 . The present calculations illustrate that l_1 dominates mode I crack behavior in a manner consistent with indentation behavior, suggesting that SGP parameters determined via indentation will be predictive for mode I behavior.
- If existing experimental length-scales (typically in the range of 10 nm to 1 μ m for moderate to high strength materials) are indicative of l_1 , then SGP hardening is

relevant to both unstable and subcritical crack propagation. For unstable cracking, SGP enhanced stresses are predicted by a continuum model, but the governing distance may be less than a physical fracture process zone.

- Crack tip stress enhancement from SGP, predicted by multiple length scale models, occurs over sufficient distance and magnitude to substantially impact hydrogen localization and the resulting hydrogen assisted crack propagation in alloys including a wide range of strength and loading conditions.

It is important to note that our analyses neglected an important physical length-scale, namely the distance over which crack blunting effects are important. It may turn out that even when SGP persists to a physically meaningful length-scale, the size of the SGP affected zone is still smaller than the length-scale over which traction distributions are altered by the presence of crack tip blunting. The interplay between finite deformation effects, material properties and SGP is an outstanding issue. On a related note, the implication of the present results is that SGP has a small role to play in multiscale models which attempt to bridge behaviors over multiple scales. It is unlikely that SGP effects will ever traverse more than a micron, implying that effective multiscale models may simply involve transitions directly from HRR fields to discrete dislocation or molecular dynamics simulations.

2. Crack Tip Plasticity Characterization

One means of validating the application of a given plasticity theory to fatigue crack growth is to perform targeted characterization of the plasticity associated with actual cracks. The crystallographic nature of plastic deformation makes it particularly amenable to diffraction-based characterization approaches. Electron backscattered diffraction (EBSD) has sufficient resolution (< 200 nm) and, unlike TEM-based methods, EBSD boasts the potential to perform statistically significant investigations of crack wake plasticity [10] by examining a large sample area. The EBSD technique was used during the present AFOSR program to assess the environmental effect on crack wake plasticity at various stress intensity ranges (ΔK) for the Al-Cu-Mg alloy AA2024 [11]

- Band contrast maps reveal the plastically deformed region in the vicinity of crack wake and, furthermore, may be used to measure the plastic zone size.
- Crack closure is observed due to the tortuous cracking induced during vacuum cracking. Evidence of crack closure helps to explain the lower than expected cyclic plastic zone size for low R ratio conditions due lower effective ΔK values.
- Deformation bands, apparently aligned with $\{111\}$ planes, were observed in the wake of the crack produced in a humid environment, but not for a vacuum environment. This is consistent with the hydrogen enhanced localized plasticity (HELP) model for environmental degradation due to hydrogen, however, further experimentation will be required to clarify this conclusion since it is known that the crack path departs from $\{111\}$ slip band cracking upon exposure to humidity.
- Plastic zone size measurements over a range of ΔK are consistent with the widely accepted quadratic relationship suggested by conventional fracture mechanics.

Further, fatigue cracking in vacuum results in a larger plastic zone size as compared to fatigue in a humid environment.

- The plastic zone size varies grain-to-grain in both wet air and vacuum environments. Ease of deformation and dislocation arrangements in crack wake depend on orientation of the grain that lead to differences in extent of crack wake plasticity. Crystal plasticity modeling is needed to fully interpret these results

Micro-Laue diffraction at the Advanced Photon Source (APS) x-ray synchrotron was also used to characterize the plastic damage in the vicinity of crack wakes. Micro-Laue diffraction is the basis of a three-dimensional x-ray microscope, which can provide information similar to the SEM-EBSD approach discussed above, but in 3D. Our major contribution in this area, with help from collaborators at the Oak Ridge National Laboratory, has been to develop a new Laue diffraction indexing procedure that is amenable to heavily dislocated and/or multiphase materials¹. Ultimately, the spatial resolution required to characterize crack wake plasticity about fatigue cracks in the near threshold regime has favored characterization by EBSD. However, advances in the Micro-Laue diffraction technology promise to make the spatial resolution comparable and there are dramatic advantages when it comes to angular resolution, which would be useful for characterizing plastic damage, and the 3D capability itself, which may allow one to characterize material below the surface, which is often damaged by surface characterization techniques.

3. Crack Path Characterization

Determining the dominant mechanism of H-decohesion provides a means of corroborating the physics base for modeling fatigue crack growth, since the critical inputs to the failure criterion (i.e., normal stress, H content, and intrinsic feature strength) will differ for different failure modes. Transgranular fatigue crack paths may be broadly classified: i) crystallographic; ii) interfacial, iii) striated, per cycle, and (iv) damage accumulation followed by advance in N cycles. Early studies suggested that fatigue cracking in Al alloys is crystallographic, based on etch pit or combined stereological and Laue x-ray diffraction analyses [1]. However, quantitative studies performed during this Air Force program have demonstrated that environmental fatigue cracking in precipitate strengthened aluminum alloys appears crystallographic, but is not dominated by separation of low-index planes [12,13]. Cracking is clearly sensitive to grain orientation, evidenced by the change in facet-like features at grain boundaries, but the precise selection criteria have not been identified. More specific observations follow.

Within 2xxx series aluminum alloys {peak aged Al-Li Cu (C47A) and underaged Al-Cu-Mg (AA2024)} [12], fatigue cracking in ultrahigh vacuum (the absence of environment) yields a very torturous fracture surface.

¹ A paper documenting the new method is in preparation for the Journal of Applied Crystallography.

- Crystallographic facets form nearly parallel to up to 4 variants of $\{111\}$ for two aluminum alloys that deform by highly localized planar slip bands; over a wide range of applied ΔK for C47A with shearable precipitates and at near threshold ΔK levels for AA2024 with solute clusters and perhaps fine-shearable precipitates.
- High resolved shear stress coupled with available $\{111\}$ planes are required to form the facets in age-hardened Al-alloys according to the positive correlation between facet normals, resolved 3-dimensional elastic shear stresses, and local $\{111\}$ distribution about the crack tip.
- Over 70% of crack surface facet orientations are near- $\{111\}$ planes, but only a low percentage of facet orientations are precisely parallel to $\{111\}$, with better than 90% certainty, suggesting that vacuum crack extension involves multiple deformation band segments formed in the crack tip process zone and intraband cracking on the submicron scale.

Within aggressive environments, the fracture surfaces of the 2xxx series alloys lose their tortuosity. In fact, water vapor and NaCl solution environments affect a similar dramatic change in crack morphology and crystallography, and deformation band cracking with near- $\{111\}$ facets is never observed, which calls into question any mechanism for increased da/dN based on H-enhanced slip localization.

- High resolved-normal stresses are required to form environmental fatigue crack facets according to the positive correlation between facet normals and resolved 3 dimensional elastic tensile stresses about the crack tip.
- Grain boundaries affect changes in transgranular fatigue crack surface facet orientation and morphology, suggesting a crystallographic basis for damage. However, the data do not indicate a dominant low-index orientation of environmental fatigue crack facets.
- Two facet morphologies (broad-flat and repeating-step) are produced for each alloy stressed in moist environments, are defined by and coexist in adjacent as well as the same grains, and exhibit a wide range of orientations between $\{001\}$ and $\{101\}$.
- Repetitive-stepped facets typically do contain areas parallel to $\{100\}/\{110\}$ on the $1\ \mu\text{m}$ scale, combined with surface curvature, consistent with a mechanism for discontinuous fatigue crack growth involving H-enhanced $\{100\}/\{110\}$ cleavage and crack tip plasticity.
- Broad-flat faceted regions are generally parallel to a variety of high index planes (generally inclined more than 15° from $\{100\}/\{110\}$, although there are exceptions), consistent with a mechanism for cracking that combines very high crack tip tensile stresses, intensely trapped H at complex dislocation/vacancy structure, and possibly near-isotropic cleavage fracture strength; within $\sim 1\ \mu\text{m}$ of the crack tip.

In order to determine the effect of exposure levels on the mechanisms of environmental fatigue, targeted experiments were performed on samples fatigue cracked in different water vapor pressures and frequencies [14], prior to examination [15].

- Fatigue crack surface morphology depends on the level of environmental exposure, indicating multiple transitions in damage mechanism, crack growth rate, and facet orientation that are similar for each alloy.
- Moderate to high levels of environmental exposure eliminate $\{111\}$ facets, characteristic of slip band cracking in high vacuum and suggesting that environmental hydrogen does not enhance this slip-based damage process.
- Complex facet crystallography, typical of fatigue crack growth at relatively high environmental exposure, is explained by evolution of several facet orientations with increasing water vapor pressure/frequency ratio above a high vacuum reference.
- A fraction of high index facets is produced by very low water vapor pressure, due to either a hard surface film effect on slip or low-concentration H embrittlement. Further increases in exposure introduce a significant fraction of low index fatigue facets; $\{100\}$ for Al-Cu-Mg and $\{100\}$ plus $\{110\}$ for Al-Cu-Li. Additional high index cracking is produced at very high exposures.

Most recently, a FIB/TEM study demonstrated the capability to view structure within 500 nm of the crack wake.

- Subgrains are observed within ~ 200 nm of the fatigue crack wake for Al-Cu-Mg stressed in high pressure water vapor, perhaps contributing to fatigue damage and facet orientations; very high density defect structure or amorphous regions are not present.

In addition to the above detailed analyses of cracking in 2xxx series alloys, the SEM-based EBSD/Stereology technique was also successfully used to determine the moist air-environmental fatigue crack facet crystallography for slightly over-aged Al-Zn-Cu-Mg-X (X = Zr or Mn) 7xxx series alloys stressed in the low growth rate regime.

- Transgranular crack facet characteristics are similar for the Mn and Zr variants, independent of grain size/recrystallization, and conform with a substantial published data base for Al-Cu-Mg/Li.
- Cracks form in the more highly stressed spatial orientations about the crack tip and change character at grain boundaries, indicating a role of the grain crystallographic orientation.
- Two morphologies of facets exhibit a complex mixture of high index orientations, some low index $\{100\}$ and $\{110\}$ facets, and only rare occurrence of cracks near $\{111\}$ planes.
- The similar facet characteristics for Al-Zn and Al-Cu alloy classes suggest a common environment sensitive damage mechanism that is not exclusive to simple slip or cleavage processes that lead to the low index plane cracking.
- Repetitively stepped facets with surface curvature may involve H-enhanced cleavage along $\{100\}$ or $\{110\}$ planes distorted by plasticity. Broad-flat facets speculatively result from either isotropic lattice strength that is H degraded, or tensile stress-based cracking through structure highly defected by cyclic plasticity with trapped H.

The similarity in the observations between 2xxx and 7xxx suggests that there are universal mechanisms responsible for environmental degradation of fatigue crack growth resistance in aluminum alloys.

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EDUCATIONAL IMPACT OF THIS PROGRAM

Yunjo Ro, MS Thesis, "Fatigue Crack Surface Crystallography of Precipitation Hardened Aluminum Alloys," Department of Materials Science and Engineering, University of Virginia, 2004. (50% support)

PhD in Materials Sci. and Eng., University of Virginia, in progress. (50% support) Mr. Ro has established the interaction of environmental exposure and precipitation hardening microstructure for two modern aluminum alloys, Al-Cu-Mg and Al-Cu-Li. In addition to providing new insight regarding the interaction of various mass transport and reaction rate limitations of da/dN , this work demonstrates the important regimes for microstructural dependence (Fig. 4) that is a foundation element for the present proposal. Further, he has performed proof of principle experiments using the focused ion beam to prepare TEM foils from the crack wake to discern the effect of environment on reaction layer formation and local plasticity.

Uday Komaragiri, PhD in Civil Eng., "Crack Tip Length Scales and Stress Elevation from Phenomenological Strain Gradient Plasticity," University of Virginia, 2007 (100% support).

Mr. Komaragiri developed a finite element code for performing elasto-plastic simulations incorporating strain gradient plasticity effects in an incremental flow theory approach. Simulation work to date has emphasized monotonic loading of a crack, but the code is sufficiently robust to include cyclic loading. This work has achieved three important accomplishments: (1) The finite element implementation of gradient plasticity is fully functional, showing potentially important stress elevation ahead of the crack tip, (2) The relationship is established between the size of this stress elevated zone, relative to the standard HRR solution for crack tip stresses, and the parameters of applied K , strain hardening exponent, and material dependent length scale (l^*), which is related to microstructure, and finally, (3) the full three-parameter version of the phenomenological strain gradient plasticity model has been implemented and the relative impacts of the various microstructural length scale parameters have been determined.

Vipul Gupta, PhD in Materials Sci. and Eng., University of Virginia, in progress. (75% support)

Mr. Gupta has been working to interpret micro-Laue diffraction data obtained at the Advanced Photon Source (APS), Argonne National Laboratory, from cracked samples of alloy 2024. The results have demonstrated a capacity to study cracking phenomena by microscopically probing the material crystal and defect structure in three dimensions. However, the present resolution of the technique may be insufficient to answer critical questions germane to the present project. The emphasis of the research has shifted to developing a new approach to Laue pattern indexing based on digital image correlation, as opposed to traditional methods of examining angles between diffraction peaks. This new approach has a number of benefits, including being able to index patterns in the presence of heavy plasticity, second phases and grain boundaries. He has further explored electron back-scattered diffraction (EBSD) as higher resolution alternative, albeit 2-dimensionally limited and his first journal publication is submitted for review.

ARCHIVAL JOURNAL PAPERS RESULTING FROM THIS PROGRAM

1. Vipul K. Gupta and Sean R. Agnew, "Measuring the Effect of Environment on Fatigue Crack-Wake Plasticity in Aluminum Alloy 2024 Using Electron Backscattered Diffraction," submitted to Materials Science and Engineering A (2007).
2. Uday Komaragiri, Sean R. Agnew, Richard P. Gangloff and Matthew R. Begley, "The Role of Individual Material Length-Scales in Strain Gradient Plasticity Predictions of Crack Tip Behavior", International Journal of Fracture, in review (2007).
3. Uday Komaragiri, Sean R. Agnew, Richard P. Gangloff and Matthew R. Begley, "Crack Tip Length Scales and Stress Elevation from Phenomenological Strain Gradient Plasticity", Journal of Mechanics and Physics of Solids, in review (2007).
4. Yunjo Ro, Sean R. Agnew and Richard. P. Gangloff, "Environmental Fatigue Crack Surface Crystallography for Al-Zn-Cu-Mg-Mn/Zr" Metallurgical and Materials Transactions, A, in review (2007).
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PROCEEDINGS PAPERS RESULTING FROM THIS PROGRAM

10. Y.J. Ro, S.R. Agnew, and R.P. Gangloff, "Environmental Exposure Dependence of Low Growth Rate Fatigue Crack Damage in Al-Cu-Li/Mg Alloys", in Proceedings, Conference on Very High Cycle Fatigue, J. Allison, J.W. Jones, J.M. Larsen and R.O. Ritchie, eds., TMS-AIME, Warrendale, PA, in press (2007).
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