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Sarah E Galyon Dorman *SAFE, Inc,* sgd@saf-engineering.com

Justin W Rausch *SAFE, Inc* 

Saravanan Arunachalam *SAFE, Inc* 

Scott A Fawaz SAFE, Inc

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## Examination and Prediction of Corrosion Fatigue Damage and Inhibition



Sarah E.Galyon Dorman Justin W. Rausch Saravanan Arunachalam Scott A. Fawaz SAFE Inc.



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- Current environmentally assisted fatigue testing methodologies do not account for nucleation from corrosion damage
- The DoD is moving to ban the use of chromates as a protection system for DoD assets<sup>1</sup>
- The protection chromate provides to fatigue crack growth needs to be fully characterized so replacement coatings can be properly assessed and there is no unexpected loss of corrosion fatigue protection as chromate is phased out
- Current coating qualification methods do not account for mechanical damage

<sup>1</sup>Undersecretary of Defense, John, J. Young, Jr., (2009) "Limiting the Use of Hexavalent Chromium," Memorandum for Secretaries of the Military Departments. © 2016 SAFE Inc Proprietary



## **Objective**



- To address the issue of corrosion damage as it relates to crack nucleation and propagation of relevance to the DoD a large test development program has been undertaken to examine the effects of
  - **1. Corrosion inhibitors on corrosion fatigue damage**
  - 2. Crack nucleation and propagation from a corrosion pit to a fatigue crack
  - 3. Help influence methods for coating and inhibitor qualifications





- The United States Air Force Academy's (USAF) Center for Aircraft Structural Life Extension (CAStLE) has completed several aircraft structural teardowns
  - KC-135, C-130, C-5, T-37, T-38
- The findings from the teardowns were analyzed to breakdown the types and cause of damage on several aircraft<sup>2</sup>
- This analysis found that 78% of corrosion pits noted in the teardowns resulted in fatigue damage<sup>2</sup>
- It was also noted that many of the fatigue cracks had signs of corrosion oxides on the surface suggesting that environment is entering the cracks at some point during service<sup>2</sup>

<sup>2</sup> Shoales, G.A., Walters, M.R., Fawaz, S.A. (2009) "Compilation of Damage Findings from Multiple Recent Teardown Analysis Programs," Proceedings of the 2009 International Conference on Aeronautical Fatigue (ICAF) Symosium, Rotterdam, The Netherlands. pp. 187-208 © 2016 SAFE Inc Proprietary



### Environmental and Mechanical Interactions



SCC and Fatigue combined on fracture surface



SEM micrograph shows three regions

### Fatigue fracture with intergranular corrosion





Fatigue crack with corrosion debris on surface

Intergranular corrosion noted on the shelf edges when sample tilted; main surface only shows fatigue





Corrosion Fatigue Inhibition by Chromate SAFE



<sup>3</sup> Gasem, Z. and Gangloff, R.P., (2001) "Rate-Limiting Processes in Environmental Fatigue Crack Propagation in 7000-series Aluminum Alloys", in *Chemisty and Electrochemistry of Corrosion and Stress Corrosion Cracking*, p. 501-521. R.H. Jones, Editor. TMS-AIME: Warrendale, PA. © 2016 SAFE Inc Proprietary



#### Corrosion Fatigue Inhibition with Low Concentration Molybdate





<sup>5</sup> Warner, J.S. (2010) "The Inhibition of Environmental Fatigue Crack Propagation in Age-Hardenable Aluminum Alloys." PhD Dissertation, University of Virginia, Charlottesville, VA. <sup>6</sup> Wanhill RJH (2001) Flight simulation fatigue crack growth guidelines, NRL-TP-2001-545. NRL, Amsterdam, The Netherlands. © 2016 SAFE Inc Proprietary



- Can polymer coatings release enough inhibitor to protect against environmental damage?
- What amount of inhibitor is expected to leach from the coating?
  - All corrosion fatigue work to date used highly soluble inhibitor salts to minimize precipitate-phase crack closure

Inhibitor Pigment	Solubility (g/100 mL)
Na₂CrO₄	87.6 (7.55 M)
Na <sub>2</sub> MoO <sub>4</sub>	65.0 (3.25 M)

 Coatings typically use low-solubility salts to prevent polymer matrix blistering

<b>Inhibitor Pigment</b>	Solubility (g/100 mL)	
SrCrO <sub>4</sub>	0.096 (4.7 mM)	MIL-SPEC Class 1
BaCrO <sub>4</sub>	0.00026 (0.01mM)	MIL-SPEC Class 2
CaMoO <sub>4</sub>	0.0011 (0.05 mM)	MolyWhite 501

<sup>7</sup> Haynes, W.M. Ed, (2011) The Handbook of Chemistry and Physics, 92<sup>nd</sup> Ed.

<sup>8</sup> Sinko, J. (2001) Progress in Organic Coatings, vol. 42. pp.267-282 © 2016 SAFE Inc Proprietary



## Inhibition Test Development

 For the area in the wing known to collect liquid, analysis of the coated surface area-to-volume on fluid ratio was calculated using a 3D model of the aircraft



<sup>9</sup> Hammond, Matthew, James Greer, Scott Fawaz, Börje Andersson, Robert Rainsberger, Monica Poelking (2010), "Detailed Three-Dimensional Modeling of the C-130 Center Wing Box for Damage Tolerance Analyses", Proceedings of the 2010 Aircraft Airworthiness & Sustainment Conference, Austin, TX



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NOTCH (

### **Inhibition Test Development**



Bulk Solution



Atmospheric Thin Film NaCl deposited by printing

Single Edge Notch (SEN) Specimen

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<sup>10</sup> Chen, Z.Y., Cui, F. Kelly, R.G. (2007) "An Analytical Model for Calculating Current Delivery Capacity of Thin-film Cathode and the Stability of Localized Corrosion under Atmospheric Environments," ECS Proceedings 3(21) pp443-457. © 2016 SAFE Inc Proprietary





- The maximum concentration of inhibitor expected in solution is limited by solubility:
  - 4.7 mM CrO<sub>4</sub><sup>=</sup> from SrCrO<sub>4</sub> coating
  - 0.05 mM MoO<sub>4</sub> = from CaMoO<sub>4</sub> coating
- Bulk solution (500 mL) fatigue testing of a COATED SPECIMEN produces low inhibitor concentration
  - Concentrations < 0.05mM CrO<sub>4</sub><sup>=</sup> from SrCrO<sub>4</sub>
  - Concentrations < 0.002mM MoO<sub>4</sub><sup>=</sup> from CaMoO<sub>4</sub>



#### **Effect of Chromate**







#### **Effect of Molybdate**









- A salt layer added to mimic representative corrosion conditions-loading was 400µg/cm<sup>2</sup>
- Using glycerin solutions to hold steady RH (80%)







- A salt layer added to mimic representative corrosion conditions-loading was 400µg/cm<sup>2</sup>
- Using glycerin solutions to hold steady RH (80%)





### **Atmospheric Corrosion Fatigue**









- Width: 10 mm
- Thickness: 3.2 mm
- Central hole: 6 mm
- Corrosion pit: 0.15 mm diameter











- Crack growth monitored by dcPD method and unique marker loading spectrum
  - Marker loading spectra: 10-4-6, Baseline loading at stress ratio (R) = 0.65













- Obtained the crack growth data from marker bands and identified crack nucleating features using a scanning electron microscope
- Calculated the crack growth rates and life to crack nucleation and periphery crack formation
- Determined the normalized voltage signal limits to detect crack nucleation and periphery crack formation irrespective of pit dimensions, stress conditions

Stress,	Specimen	Pit Dim		
$\sigma_{max}$ , MPa	ID	a, mm	c, mm	a/C
70	S70-1	0.0984	0.100	0.987
	S70-2	0.084	0.117	0.718
	S70-3	0.121	0.091	1.330
	S124-1	0.116	0.096	1.208
124	S124-2	0.111	0.112	0.991
	S124-3	0.207	0.136	1.552
180	S180-1	0.113	0.134	0.843
	S180-2	0.084	0.109	0.771
	S180-3	0.113	0.123	0.919







- Crack nucleation typically at *a*-tip of the pit from a jut-in or micro pit
- For the σ<sub>max</sub> =70 MPa case 90% of the life is spent on growth of a mechanically small crack
- For the σ<sub>max</sub> =180 MPa crack nucleation occurs at less than 3% of the total life for all cases
- Crack nucleation occurs before the normalized dcPD signal (V/V<sub>o</sub>) is 1.00036
- The full periphery crack is formed by the time V/V<sub>o</sub> is greater than 1.02 © 2016 SAFE Inc Proprietary





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- The measured and calculated crack lengths were compared using the marker bands from the fracture surface
- The fatigue crack initiation site was also determine and the corrosion pit roughness observed







#### **Corrosion Damage to Fatigue Test Development-Stress Ratio**



		Pit Dimensions Periphery Crack Dimensions						
Max Remote Stress ( σ)	Stress Ratio (R)	a (mm)	c (mm)	a (mm)	c (mm)	Cycles to Periphery Crack	Total Cycles (1.5mm)	Nucleating Feature
123.7	0.65	0.080	0.087	0.290	0.218	21,475	60,399	Micro-pit
124.2	0.65	0.113	0.122	0.284	0.181	27,600	50,523	Micro-pit
124.2	0.65	0.207	0.136	0.333	0.212	13,475	36,833	Micro-pit
127.5	0.10	0.117	0.108	0.230	0.181	2,545	9,504	Micro-pit
128.4	0.10	0.118	0.108	1.018	0.824	8,180	12,447	Micro-pit

 R= 0.1 developed fully formed fatigue crack and grew to 1.5 mm faster than the R=0.65 tests

 The fatigue crack nucleating feature did not change based on stress ratio





- Crack growth rates were calculated from the measured crack dimensions using the incremental polynomial method using the Fawaz/Andersson solutions in AFGROW
- The ΔK values in the a and c-directions range from 1.2 to 12 MPa√m for all conditions tested
- The da/dN and dc/dN results were comparable with the published SIPS data for this AA7075-T651 plate being used for the study





# Corrosion Damage to Fatigue Test









### Summary



- Low solubility inhibitors (chromate & molybdate) have been shown to slow fatigue crack growth rates, but only below a ∆K=5 MPa√m at frequencies of 0.2 Hz
- New test methodologies for examining inhibitor effects have been designed using aircraft geometries and an understanding of atmospheric corrosion processes
- A standardized test method for studying the corrosion pit to fatigue crack transition using dcPD has been developed and validated
- The combination of these two test methods is moving forward to continue to expand the understanding of fatigue crack inhibition
- Current coating qualification methods fail to consider mechanical loading; test methodology to address this limitation has been designed and validated

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- Use the pit to crack transition fatigue methodology with coated samples under more complex environments
- Add other environmental factors including UVlight, ozone and temperature effects
- Integrate the crack growth rate data inhibitor and environment into AFGROW for better modeling and prediction capability
- Publish updated testing protocols related to the pit to crack transition sample







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