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The effect of corrosion inhibitors on environmental fatigue crack growth in Al–Zn–Mg–Cu $\stackrel{_{\diamond}}{\sim}$



^a Center for Aircraft Structural Life Extension, 2354 Fairchild Drive, Suite 2J2, USAF Academy, CO 80840, United States
^b United States Air Force Academy, Department of Biology, 2355 Faculty Drive, Suite 2P389, USAF Academy, CO 80840, United States

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1. Introduction

ABSTRACT

Researchers have shown that high levels of chromate added to corrosion fatigue tests on 7xxx series aluminum alloys slows the fatigue crack growth rate (Gasem and Gangloff, 2001; Lui et al., 2003). Corrosion fatigue testing was completed with polymer coating leaching rate relevant inhibitor concentrations to determine the effect on fatigue damage. These results were compared to inhibition by the bacteria *Ralstonia pickettii*. For the test conditions, the bacteria showed better inhibition than the ionic inhibitors. More research is needed to quantify the mechanical load range and atmospheric corrosion conditions over which ionic inhibition will occur with the low levels of inhibitors available from coatings. © 2014 Elsevier Ltd. All rights reserved.

As the USAF and DoD extend the life of current weapon systems, corrosion damage becomes a larger concern. At the same time the DoD has moved to phase out the use of chromates as a corrosion inhibitor due to the environmental and personnel risks [1]. Chromates are used in a variety of corrosion prevention coatings including conversion coatings and primers. Corrosion damage is of concern to the aircraft sustainment community for many structural integrity reasons such as reduction in static strength and stability allowables, including situations in which corrosion damage may act as a stress raiser and initiate fatigue cracks [4]. While it has been documented that high levels of chromate and a chromate replacement inhibitor, molybdate, added to a bulk solution can inhibit fatigue crack propagation, it has not been shown that these inhibitors leaching from a coating can do the same [2,3,5,6]. However, if chromate does provide protection that slows corrosion fatigue crack propagation, current systems have unaccounted for this corrosion fatigue protection mechanism. If that protection is not documented, then the protection will be lost in replacement chromate-free systems. At the same time, chromate is considered the benchmark for all other corrosion inhibitors to meet, so an understanding of the protection current chromate coatings provides to corrosion fatigue is needed. New and novel coating options are also being pursued as the DoD moves away from chromate in an effort to find more environmentally friendly options.

* Corresponding author.

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E-mail address: sgd@saf-engineering.com (S.E. Galyon Dorman).

Nomenclature	
ΔK dcPD EDS f ICP-MS R SEN UV-vis	stress intensity range $(K_{max}-K_{min})$ direct current potential drop electron dispersive spectroscopy frequency inductively coupled plasma mass spectrometry stress ratio (K_{min}/K_{max}) single edge notch ultraviolet-visible spectroscopy

For current commercial coatings the loading amounts of inhibitors can vary, but are designed such that the coating is not depleted of inhibitor during a maintenance cycle, typically 6–8 years [7–9]. Organic coatings with inhibitors are understood to readily absorb water or other liquid environments through inherent defects in the coating resulting in interconnected pores which allows inhibitor pigments within the coating to dissolve and dissociate in electrolyte within the coating itself [8–11]. Typically pigments with low water solubility are selected for use in coatings so that (a) osmotic pressure-induced blisters are avoided, and (b) the inhibitor remains in the coating over long times rather than rapidly dissolving out when first in contact with corrosive environments [8,10]. Because of the low solubility, only low concentrations of inhibitors would be expected in the surrounding solution before precipitation of solids occurs. For the previously completed bulk solution corrosion fatigue testing (sample fully immersed), Na₂CrO₄ (solubility in water: 87.6 g/100 mL) and Na₂MoO₄ (65.0 g/100 mL) were used as inhibitors; these salts have a much higher water solubility than coating pigments (SrCrO₄: 0.096 g/100 mL, CaMoO₄: 0.0011 g/100 mL) [8,10,12]. However the high solubility salts have advantages in studying corrosion fatigue inhibitor in that the high solubility prevents precipitates that could cause crack closure, allowing for a better understanding of the crack tip passivation mechanism [2].

As new corrosion protection mechanisms are of interest to the DoD, work is underway to quantify and understand the possible corrosion fatigue protection afforded by microbial effects. Historically researchers have observed that microbial induced corrosion was detrimental to the metal and accelerated the corrosion process. More recent research has examined the ability of a microbe to protect aluminum against corrosive effects [13]. Given that bacteria are diverse living organisms with many reactions and processes available depending on the genus and species, it is likely that the reason for corrosion protection or acceleration can be material, bacteria and/or environment dependent.

While there is some research looking at the effect of bacteria on pitting and general corrosion very little work has been completed looking at the effect of bacteria on corrosion fatigue inhibition [13–15]. The Center for Aircraft Structural Life Extension (CAStLE) at the United States Air Force Academy (USAFA) has discovered that a bacteria *Ralstonia pickettii* (generally thought to be of limited occupational concern for otherwise healthy individuals) slows the fatigue crack growth rates in 7xxx series aluminum alloys [16].

2. Experimental procedure

For a proper understanding of the protection afforded by epoxy primers appropriate corrosion fatigue test geometries needed to be determined to control the amount of chemical inhibitor present. The results of the chemical inhibitor fatigue testing would then be used to help quantify *R. pickettii*'s inhibition ability.

2.1. Chemical inhibitors

To determine the effect chemical inhibitors (chromate/molybdate) have on corrosion fatigue damage in DoD-relevant materials, a single edge notch (SEN) specimen, shown in Fig. 1, was used for fatigue testing. The samples were made from a peak-aged, legacy age-hardenable Al–Zn–Mg–Cu aluminum alloy and temper (7075-T651). The SEN sample was loaded into a computer-controlled servohydraulic test frame and crack growth was measured using a direct current potential drop (dcPD) system. The test load was controlled to provide a constant ΔK of 6 MPa \sqrt{m} with a stress ratio (R) of 0.65. The loading frequency (f) was 0.02–20 Hz in a bulk solution 0.06 M NaCl environment with inhibitor added to the bulk solution.

To determine the relevant chromate/molybdate amounts for fatigue testing, leaching studies were completed on four different epoxy coatings containing different loading amounts of $SrCrO_4$ (either 12 or 17 wt%) developed by Luna Innovations [17]. Leaching studies were completed by exposing the free-standing films in 100 mL of DI water for two and four days. The leached liquid samples were then examined using Ultraviolet–Visible (UV–Vis) spectroscopy using standards serially diluted from a 1000 ppm CrO_4^{2-} purchased standard solution to determine the amount of CrO_4^{2-} present. The leaching data from the free films was extrapolated from standard solutions by the WinUV software on the Varian Cary Series spectroscope [18].

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