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SUMMARY
1. PURPOSE: To provide security and policy review on the document(s) at Tab 1 prior to release to the public.

2. BACKGROUND:
- Title (1): The Effect of Corrosion Inhibitors on Environmental Fatigue Crack Growth in Al-Zn-Mg-Cu
- Author(s): Sarah E Galyon Dorman et al, Center for Aircraft Structural Life Extension


Thesis Dissertation Book Other:

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- Description: Special Issue (SI) of Engineering Fracture Mechanics: Corrosion Fatigue


- Recommended distribution statement: Distribution A. Approved for public release, distribution unlimited.

3. DISCUSSION: This research was performed under the sponsorship of the DoD Office for Corrosion Policy and Oversight under the auspices of their Technical Corrosion Consortium.

4. RECOMMENDATION: Department Head or designee review as subject matter expert. DFER review for policy and security and provide public release clearance.

JAMES M. GREER, JR., PhD, PE
Technical Director, CASIL

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The Effect of Corrosion Inhibitors on Environmental Fatigue Crack Growth in Al-Zn-Mg-Cu

Sarah E. Galyon Dorman1, a Benjamin C. Hoff1, Daniel H. Henning1, Sarah E. Collins1

1 Center for Aircraft Structural Life Extension, 2354 Fairchild Drive, Suite 2J2, USAF Academy, CO 80840, United States

sgd@saengineering.com

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Keywords: Corrosion fatigue inhibition, chromate, molybdate, inhibitor leaching, AA7075

Abstract. Corrosion fatigue is an area of concern for the United States Air Force (USAF) and other Department of Defense (DoD) organizations. Often DoD corrosion prevention systems include chromate containing coatings, typically in the form of chromate conversion coatings and polymer primers. Chromate has been used successfully for many years within the DoD to prevent corrosion damage. However the environmental and personnel risks associated with chromate coatings have caused the USAF to pursue non-chromate containing corrosion prevention coatings [1]. To fully quantify chromate replacement coatings, an understanding of the effects that chromate has on corrosion fatigue crack growth rates must be fully characterized. Some researchers have shown that high levels of chromate added to 0.6 M NaCl full immersion corrosion fatigue tests on 7xxx series aluminum alloys slows the fatigue crack growth rate substantially [2,3]. The limitation of that research was that the amount of chromate present in the test solution environment was not correlated to expected leak rates of chromate from polymeric coatings and a high solubility salt was used.

The majority of DoD assets are protected from corrosion by polymer coatings loaded with corrosion inhibitors. For these coatings to slow fatigue crack propagation the corrosion inhibitors must become mobile as a consequence of hydration of the polymer coating matrix. Based on this mechanism of corrosion inhibitor release, the examination of atmospheric corrosion fatigue becomes important to help understand how inhibitors work in real world situations with hydrated salt layers rather than only fully immersed solutions. At the same time the DoD has become interested in new, novel corrosion protection mechanisms that could show promise as chromate replacing coatings.

Introduction

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.

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 to 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), Na$_2$CrO$_4$ (solubility in water: 87.6 g/100mL) and Na$_2$MoO$_4$ (65.0g/100mL) were used as inhibitors; these salts have a much higher water solubility than coating pigments (SrCrO$_4$: 0.096g/100mL, CaMoO$_4$: 0.0011g/100mL) [8,10,12]. However the high solubility salts have advantages in studying corrosion fatigue inhibition 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 (CASiLE) 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].

**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.

**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 Figure 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 $\Delta K$ of 6 MPa$\sqrt{m}$ with a stress ratio ($R$) of 0.65. The loading frequency ($f$) was 0.2 - 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 weight %) 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].

**Biological Inhibition.** In 2011, stress life corrosion fatigue tests were being completed at varying stress levels and stress ratios ($R$) to examine the effect of a modern alloy and temper (AA7475-T7351) on the initiation of a crack from corrosion damage (pit) compared to a legacy alloy and temper (AA7075-T651). Bacteria grew on some of the samples increasing the fatigue life five to six times depending on the stress ratio.

![Sample geometry and volumes used for Concentration Calculations](image)

Figure 1: Sample geometry and volumes used for Concentration Calculations

After the stress life testing was completed, the bacterial effect was examined using the dcPD method to measure the effect on the crack growth rate. All fatigue crack growth rate testing was completed using the SEN, shown in Figure 1. Like the chemical inhibitor comparison, fatigue testing was completed at a constant $\Delta K$ (6 MPa$\sqrt{m}$) over a range of frequencies (0.02-20 Hz) and at a stress ratio ($R$) of either 0.10 or 0.65. The test parameters were selected to compare the crack growth rates to published inhibited corrosion fatigue crack growth rates using chromate and molybdate [2,5,6]. The corrosive environment was 0.06 M NaCl. The bacteria were allowed to grow naturally or intentionally added to solution to quantify the effect. To add bacteria to solution, colonies of *R. picketti* were grown on R2A agar and single colonies were added to the 0.06 M NaCl solution. Figure 2 shows the growth of the bacteria on the 7075-T651 aluminum sample. *R. picketti* is known to build biofilms and the growth is thought to be a biofilm [16,19-21]. Figure 2b gives a schematic of biofilm development [19].

**Results**

**Chemical Inhibitor Leaching.** All of the prior corrosion fatigue work with aluminum alloys was completed using inhibitors added to a bulk NaCl solution or deliquesced onto the surface of a
sample rather than the migration of inhibitors from polymer matrix coatings [2,3,5,6]. The inhibitors used for most of these fatigue studies were high rather than low solubility inhibitors, the latter of which are typically used in military aviation corrosion preventive coatings [2,3,5,6]. This difference may be of importance as the leaching of inhibitors is purposely controlled in organic coatings via salt solubility in order to make the life of the coating acceptable. That said, inhibitor leaching from a coating, primer or corrosion prevention compound (CPC), would be the source of any inhibitor that could slow fatigue crack propagation. All of these variables make understanding the leaching rate of known coatings and inhibitors critical to designing appropriate fatigue test criteria. An understanding of the chromate leaching rate from coatings and how chloride concentration and pH could change the amount of chromate present in a solution was needed. To answer these questions literature reviews of chromate leaching rates and inhibitor pigment solubility effects were performed, along with in-house leaching studies.

Figure 2: (a) Bacterial growth ("fuzz" at bottom of samples) on the 7xxx series aluminum alloy in 0.06M NaCl solution. The white on the dcPD wires is a protective coating. The fuzz continues up the entire length of the sample. (b) Biofilm development [19].

Figure 1 shows the single edge notch (SEN) sample and three different test configurations. The Bulk Testing label refers to a standard test cell filled with 500 mL of solution with a SEN sample primed on all four sides (Coating Surface Area: 7.92 cm²) which mimics areas of standing water. The Restricted Volume refers to a very small cell containing approximately 0.5 mL of solution covering a 2 cm high portion of the flat on the SEN sample (Coating Surface Area: 5.28 cm²) which mimic areas such as lap joints and other occluded regions of aircraft. The Coating Surface to Volume ratio for the reduced volume cell was determined using aircraft components from the USAF. Thin Film refers to salt deliquesced to form a 100 µm thickness film (8x10⁻⁵ L) onto the four flat surfaces of the SEN sample (Coating Surface Area: 7.92 cm²). The Thin Film environment is used to mimic how atmospheric corrosion is understood to occur on aircraft structure rather than a large pool of liquid around the sample [22-24].

The leach rates from the model films were compared to that expected from published CrO₄²⁻ leach rates [8-10]. To make the comparison, the published leaching data were converted to molarity using the geometries and volumes of the Luna film leaching experiments and the testing geometries reviewed above. The Luna films were able to leach about 6-20 times more chromate into solution than the commercial epoxy primer. This result suggests that the Luna film is likely more porous
than the commercial primer as the loaded salt was the same (SrCrO₄). A 12-17% loading amount is on the low end for chromate, so that is unlikely to be the cause of the leaching differences [7,8].

A summary of the published chromate leaching data follows. The overall leaching rates varied by about 16 times between the high and low leaching coatings [8-10]. The loading amount of chromate for all of the coatings can vary, however, as the porosity of the film can also greatly affect the leaching rates. Therefore it is unclear what causes the variation in leaching amounts [7,8]. There is no significant difference in leaching rates with the variation of low (below 0.1 M NaCl) chloride content. However higher chloride content (0.85 M) makes a large difference, nearly doubling the amount of CrO₄²⁻ leached from the coating. This result suggests there could be a threshold of chloride content over which the chloride is detrimental to the polymer matrix allowing for more inhibitor release [7,8]. The published data also suggests that the change in pH does not cause large changes in the amount of chromate leached. There is some suggestion that more acidic environments cause more leaching for that coating, but the increase in the amount of chromate leached is within the overall scatter of chromate leaching from epoxy coatings [8-10].

Table I: Expected leaching results for SrCrO₄ primer films converted to the test three geometries. All concentrations are in terms of mol/L (M) CrO₄²⁻.

<table>
<thead>
<tr>
<th>Film Number</th>
<th>Expected Concentration (M) Bulk Solution (500mL) 4 Days</th>
<th>Expected Concentration (M) Restricted Volume (0.5 mL) 4 Days</th>
<th>Expected Concentration (M) Thin Film (0.08 mL) 4 Days</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>3.21 x 10⁻¹</td>
<td>0.021</td>
<td>0.201</td>
</tr>
<tr>
<td>2</td>
<td>2.41 x 10⁻¹</td>
<td>0.016</td>
<td>0.151</td>
</tr>
<tr>
<td>3</td>
<td>4.28 x 10⁻¹</td>
<td>0.029</td>
<td>0.268</td>
</tr>
<tr>
<td>4</td>
<td>2.95 x 10⁻¹</td>
<td>0.020</td>
<td>0.184</td>
</tr>
</tbody>
</table>

*above the SrCrO₄ solubility limit of 0.0047 M CrO₄²⁻ [12]

Similar leaching studies were completed on molybdate (a chromate replacement) primers. The University of Southern Mississippi (USM) developed three CaMoO₄ containing primers [25]. Each primer had a different loading amount of MoO₄²⁻ (20.8%, 28.7%, 36.9% weight percent). USM coated and scribed AA 7075 panels (Surface Area: 6.45 cm²) prior to completing leaching experiments in 100 mL DI water. The leaching results appear in Table II along with the concentrations expected for the three test conditions. Table II shows that the higher the loading amount of MoO₄²⁻, the lower the leaching amount. This result is unexpected, and the mechanism behind this result is currently not understood. It was originally thought the solubility limit between the CaMoO₄ and solution was being exceeded, but that is not the case as the leaching values are always below the solubility limit of 0.05 mM [12]. Also, as the inductively coupled plasma mass spectrometry (ICP-MS) is capable of detecting 1 part per billion, it is unlikely the molybdate leaching from the coating was being incorrectly measured. The explanation for these results remains to be determined.

Table II: Leaching results for the CaMoO₄ coatings developed by USM [25].

<table>
<thead>
<tr>
<th>Loading Amount</th>
<th>Leaching Concentration (100mL) 4 Days</th>
<th>Expected Concentration (M) Bulk Solution (500mL) 4 Days</th>
<th>Expected Concentration (M) Restricted Volume (0.5 mL) 4 Days</th>
<th>Expected Concentration (M) Thin Film (0.08 mL) 4 Days</th>
</tr>
</thead>
<tbody>
<tr>
<td>20.8% Moly White</td>
<td>1.8 x 10⁻⁶</td>
<td>4.3 x 10⁻⁷</td>
<td>2.9 x 10⁻⁷</td>
<td>0.003</td>
</tr>
<tr>
<td>28.7% Moly White</td>
<td>4.1 x 10⁻⁷</td>
<td>1.0 x 10⁻⁷</td>
<td>6.7 x 10⁻⁷</td>
<td>6.0 x 10⁻⁷</td>
</tr>
<tr>
<td>36.9% Moly White</td>
<td>4.7 x 10⁻⁷</td>
<td>1.1 x 10⁻⁷</td>
<td>7.7 x 10⁻⁷</td>
<td>7.0 x 10⁻⁷</td>
</tr>
</tbody>
</table>

*above solubility CaMoO₄ limit of 0.055 mM [12]
Chemical Inhibitor Fatigue Testing. Based on the leaching results from the literature, the maximum amount of SrCrO₄ in solution would be 4.7 mM (the solubility limit), however for the bulk solution (500 mL) only 0.05 mM would be expected based on the highest leaching rate for the chromate coatings in literature [8-10,12]. For CaMoO₄ the maximum concentration of inhibitor in solution would be 0.05 mM and for the bulk solution test only 0.002 mM would be expected from the leaching tests [12]. However, it was noted for the Reduced Volume and Thin Film cases the solubility limit for both SrCrO₄ and CaMoO₄ could be reached causing precipitation of solids in and about the crack which could cause crack closure.

Fatigue testing was completed using inhibitor pigments that would be found in commercial molybdate coatings, calcium molybdate (CaMoO₄) at a constant ΔK=6 MPa√m, R=0.65 in a bulk (500 mL) 0.06 M NaCl solution. The results for this test are shown in Figure 3. The starting concentration of CaMoO₄ was 0.002 mM (gray star) which is below the solubility limit; the concentration was then increased to 0.01 mM CaMoO₄ (white star). For these concentrations the solubility limit would be expected to have been exceeded in the crack. The crack growth rates for the two concentrations of CaMoO₄ are in line with the pure 0.06 M NaCl showing no inhibition with the low concentrations of calcium molybdate added to a bulk solution and no inhibition due to crack closure. All other data in Figure 3 are from other published work [5,6].

![Figure 3: Crack growth rate data for low concentrations of low solubility corrosion fatigue inhibitor CaMoO₄ [5,6].](image)

Bacterial Inhibition Fatigue Testing. Figure 4 shows the comparison of high and low amounts of *R. pickettii* when added to a 0.6 M NaCl solution to chromate (Na₂CrO₄) at f = 1 Hz [6]. The amount of bacteria was quantified visually, based on the amount of “fuzz” seen. For crack lengths of 2-4 mm both the high and low bacteria concentrations slow the crack growth rates to that of high amounts of inhibiting pigments [2]. For the 6-6.5 mm data the inhibition appears to be less effective particularly for the low bacteria case. However it should be noted that the presence of the bacteria can cause the dCPD system to under-measure the crack length (increased conductivity of the sample) which means for a constant ΔK test, where the load is being constantly adjusted, an overload is applied. Based on post-test analysis of the crack length, the 6-6.5 mm crack length data is likely at a ΔK between 10-14 MPa√m, meaning that inhibition is still occurring even though the figure suggests it is not.

Figure 4 shows the comparison between *R. pickettii* and molybdate [5,6]. The tests were run as frequency scans, starting at either the high (20 Hz) or low (0.02 Hz) frequency and then progressing
in the order of the arrows. Each frequency scan would take on the order of two weeks to complete. The results showed that if a test is started before the bacteria has had time to become visible, see Figure 5, then inhibition of the crack growth rate does not occur, however once the fuzz is visible then inhibition can occur even up to frequencies of 20 Hz. It should also be noted that the reduced flushing of the crack tip caused by the increased stress ratio does not increase the ability of the bacteria to inhibit corrosion fatigue when compared to the testing completed in comparison with chromate shown in Figure 4 [26].

Discussion

Low solubility corrosion inhibition pigments (CaMoO₄) have not shown the ability to reduce fatigue crack growth rates in bulk 0.06 M NaCl solutions whether through passive film formation or crack closure. It is currently unknown if moving toward slower frequencies or lower ΔK values would allow for inhibition at these low molybdate concentrations. The effect of chromate on small-scale fatigue damage can be characterized for the different primer types. The analysis of surface area-to-volume for leaching suggests that a thin salt film may be more applicable than full immersion testing for aircraft applications, particularly lap joints.

*R. pickettii* consistently shows inhibition on the order of high levels of known corrosion inhibitors as shown in Figures 4 and 5 and better than levels of molybdate that would be expected from a coating, Figure 3 [2,5,6,8-11]. The reason for the bacterial inhibition is currently unknown, however there are three leading theories being investigated: (1) *R. pickettii* is sequestering metal (possibly copper) into its cell wall which is stabilizing the passive film at the crack tip [15,21,26], (2) the bacteria is producing oxygen which is causing an oxide layer to develop and protect the crack tip, (3) the extracellular polymeric substance from biofilm the bacteria produces allows for protection of the crack through an inhibitive species or coating [29]. *R. pickettii* has been shown to uptake certain metals into its cell wall to protect itself from hazardous environments [27, 28]. Based on aluminum corrosion fatigue inhibition research and *R. pickettii* metal uptake literature, copper and silicon were loaded into R2A agar and *R. pickettii* colonies grown on the agar were analyzed by scanning electron microscopy and electron dispersive spectroscopy (EDS) [5, 27, 28]. The results did not show evidence of metal uptake. The ability of bacteria to help repair the oxide layer is supported by the literature and could possibly be occurring in this case [13]. When the aluminum samples with bacterial growth were analyzed using EDS, the areas with biofilm have large amounts of oxygen present, suggesting a heavy oxide layer.

![Figure 4: Comparison of the effect of *R. pickettii* on fatigue crack growth rates on AA7075-T651 to a known corrosion fatigue inhibitor chromate at ΔK=6 MPa√m; R=0.1, f=1 Hz in 0.6 M NaCl [2].](image)
Figure 5: Comparison of *R. pickettii* inhibition to a known corrosion fatigue inhibitor, molybdate, at a constant $\Delta K$ of 6 MPa/m, $R$ of 0.65 over frequencies from 0.02 to 20 Hz [5,6]. The arrows denote the order of testing.

During the corrosion fatigue testing it has been shown that *R. pickettii* can desalinate the sodium chloride test solution. The salinity is expected to drop by approximately half in 48 hours. For aluminum alloys the presence of chloride can be detrimental to corrosion fatigue but is not a primary driving force as the presence of water vapor is sufficient to raise crack growth rates over that of dry air or inert environments [30]. This suggests that the desalination of the test solution is not the primary cause of the corrosion fatigue inhibition.

Work is underway to analyze the structure of the biofilm. The early research has looked at how the biofilm develops under varying conditions in an effort to understand why the biofilm sometimes appears to be produced faster and thicker than at other times [31]. Figure 6 shows the development of the *R. pickettii* biofilm using a live/dead stain with confocal microscopy under varying temperatures, pH, and salinities and with various alloys. The amount of growth and living bacteria is rated from no growth/living (-) to heavy biofilm/all living (+++++). It appears that a neutral to slightly basic pH (7-8) allows for string-like bacteria formations (5a). Higher temperatures (30 and 37 °C) (5b) and low salt content (0.06 to 0.6 M NaCl) (5c), or neutral to slightly basic pH (7-8) cause thick biofilm formation. Formation on 4130 steel is scattered but not stringy (5d). Interestingly these are conditions very close to what exist in the corrosion fatigue test cell.

Over the course of this project, research has also been conducted into how difficult *R. pickettii* is to kill. This is particularly important because if bacteria were to be developed into a natural protective coating for any sort of military or commercial application it needs to be able to withstand a variety of environmental conditions. To date the bacteria has proven to be extremely resistant to most forms of disinfection including, heat, freezing, bleach solutions, heavy metal exposure, hydrogen peroxide (3% and 6%) and UV light exposure. The ability of these bacteria to resist disinfection suggests that the processes of living bacteria could be placed into a coating to make a non-toxic, environmentally friendly coating with corrosion fatigue protection better than that of a chromate or molybdate coating.
Figure 6: Development of a R. picketti biofilm under varying conditions; pictures and charts [31].

Conclusions

Based on the testing completed using low level, epoxy coating leaching relevant concentrations, CaMoO₄ was unable to inhibit corrosion fatigue in 0.06 M NaCl at a ΔK=6 MPavm, R=0.65 at a frequency of 0.2 Hz. It is unclear if lowering the frequency or ΔK would allow for inhibition to occur. R. picketti, is able to slow fatigue crack growth rates in 0.06M NaCl over a wide range of frequencies (0.05-20Hz) at ΔK=6MPavm, R=0.65 depending on time of bacteria exposure. It is currently unclear the mechanism the bacteria is using to protect against corrosion fatigue damage.

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