Development of Improved Accelerated Corrosion Qualification Test Methodology for Aerospace Materials

18-20 Nov 2014

Chad N. Hunter
AFRL Corrosion IPT (AFRL/RXSS)
Air Force Research Laboratory Materials and Manufacturing Directorate
**Report Documentation Page**

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Standard Form 298 (Rev. 8-98)  
Prescribed by ANSI Std Z39-18
## Acknowledgements

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- **AFRL Corrosion IPT**
Outline

• Motivation for effort/background
• Aircraft organic coating failure mechanisms
• State of the art corrosion testing and characterization of organic coatings- deficiencies
• AFRL efforts to address gaps:
  • AFRL SERDP project
  • SBIRs
  • AFRL in-house program, “Structural Component Corrosion Simulation”
• Conclusions
Motivation/Background

• Weapon system corrosion performance requirements:
  – New acquisition–design, intended environment and expected service life taken into account
  – Legacy systems– field/depot maintenance, material substitution/replacement

• Driven by several synergistic factors including:
  – Environmental regulations and high corrosion costs (DoD-wide),
  – Requirement to account for corrosion in management of structures (MIL-STD-1530C, Aircraft Structural Integrity Program, Air Force-specific but approaches could apply to other services)
  – Improved performance

• Current accelerated laboratory methodology inadequate to predict performance with relevant degradation modes

• Long-term outdoor exposure is current best practice for performance prediction, but takes 1 year+ and doesn’t mimic service conditions precisely
Background- Air Force Requirements

• MIL-STD-1530C (Aircraft Structural Integrity Program), Section 5.1.5 requires the establishment of a Corrosion Prevention and Control Program
  • 5.1.5.1 Corrosion Prevention and Control Plan
  • 5.1.5.2 Evaluation of Corrosion Susceptibility (accounting for base metals, coatings, sealants, service environments & maintenance practices, etc.)
• “Materials and processes, finishes, coatings, and films which have been proven in service or by comparative testing in the laboratory shall be selected to prevent corrosion…”
• There is currently no way to reliably meet the above criteria for emerging environmentally-compliant coatings M&P!
Coating Degradation Mechanisms

1. Mechanical defect (pitting)
2. Coating delamination (crevice corrosion)
3. Fastener/structure interface (galvanic/crevice corrosion)
4. Coating delamination (crevice corrosion/pitting)
5. Coating cracking
6. Air pocket (crevice corrosion/pitting)
7. Water-induced disbond (crevice corrosion/pitting)
8. Typical morphology of organic coating around fastener
Corrosion Testing and Characterization of Organic Coatings - Deficiencies

Laboratory salt fog (ASTM B117, 5% NaCl spray at 35°C) 2000 hrs

Outdoor Exposure After 3+ Years At Daytona, FL (Failure <1 year)
AFRL SERDP Project

• AFRL project proposed against 2009 Strategic Environmental Research and Development (SERDP) Statement of Need “Dynamic Accelerated Corrosion Test Protocol”

• Bare and coated metal samples exposed:
  – At 8 outdoor test sites
  – Laboratory, ASTM B117 salt fog
  – Laboratory, ASTM B117 salt fog with UVA irradiation and ozone gas

• Cumulative damage model for predicting atmospheric corrosion rates of 1010 steel was developed using inputs from weather data:
  – Temperature,
  – Relative humidity (%RH)
  – Atmospheric contaminants (chloride, SO₂, and ozone) levels
AFRL SERDP Project - Results

- AgCl film develops on Ag coupons exposed in modified B117 lab test with UV/ozone and outdoors, much higher than what occurs in ASTM B117

**Figure 53.** AgCl film thickness measurements on pure silver coupons as a function of exposure condition (UV/ozone) over 1000 hours in the modified exposure chamber and the B117 test chamber.

**Figure 20.** AgCl film thickness on pure silver coupons as a function of time over a two year period at all exposure sites.

Coulometric reduction method
AFRL SERDP Project - Results

Cr system

Mg rich system
Figure 60. Side-by-side chamber exposure comparison of the three coating systems on AA2024-T3 panels at (top) 400 hours and (bottom) 2000 hours exposure in the modified UV/ozone and B117 chambers, respectively. Panel coating designation code: A1A: magnesium rich coating system; A1C: rare earth conversion coat (RECC) system; A1H: full chromate coating system.
AFRL SERDP Project – Test Results

- Corrosion of coated panels in outdoor environments: strong correlation to elevated T and % RH
- Cumulative amount of time coated panel is exposed to damaging environments was dominant factor in corrosion severity
- Degradation of polyurethane topcoat observed (FTIR analysis)
- UV and ozone under constant salt fog on coated panels in laboratory was much more damaging than 2 years field exposure
- Promising results; further development of laboratory apparatus and improved methods needed
Cumulative Damage Model for Prediction of Atmospheric Corrosion

- There are 3 principal boundary conditions
  - The corrosion rate equals zero when:
    - Relative humidity drops to a threshold value, RH_{TH}
      - 60% RH for iron and steel
    - Temperature drops to freezing or below
    - Contaminant level falls to zero
- A piecewise function is used to implement the temperature and RH boundary conditions

\[
K_i = \begin{cases} 
  f(T, RH, Cl, SO_2, O_3), & \text{RH > RH}_{TH} \text{ and } T > T_f \\
  0, & \text{RH \leq RH}_{TH} \text{ or } T \leq T_f 
\end{cases}
\]

Form based on Eyring equation describing the variance of the rate of a chemical reaction with temperature

\[
K_i = \exp\left(\frac{\Delta H}{kT}\right)
\]

Material Reactivity (kinetics)

Chloride Reaction

Sulfur Dioxide Reaction

Ozone Reaction

David Rose, University of Dayton PhD Dissertation, 2014
AFRL SERDP Project - Cumulative Damage Model Results

AISI 1010 Steel Hourly Corrosion Rate Predictions for Kennedy Space Center, FL (midnight 12-13-05 to midnight 12-14-05)

AISI 1010 Steel Cumulative Corrosion Rate Predictions for Kennedy Space Center, FL (midnight 12-13-05 to midnight 12-14-05)

Comparison of AISI 1010 Steel Corrosion Test Points and Associated Predictions
Calibration/Validation Sites

- Data from four different locations with diverse conditions was used to initially calibrate candidate models... later reduced to three sites
- Candidates were validated by applying them to independent proxy data for locations not used for calibration
- Final model was validated using data from seven different sites in four different climate zones
R² value of 0.86 is higher than any published atmospheric corrosion rate prediction model intended for application at locations with diverse environmental conditions.
2014.1 SBIR topic AF141-164, “Programmable Accelerated Environmental Test System for Aerospace Materials”

Combined environmental effects:
- Salt fog (NaCl, CaCl₂, etc.)
- Gas exposure (ozone, CO₂, etc.)
- Artificial sunlight-UV weathering
- Temperature and humidity cycling
- Dynamic mechanical loading

Four contracts awarded; final reports due ~Feb 2015:
- Systems and Materials Research Corporation
- Luna Innovations
- SAFE Engineering
- Mainstream Engineering

Goal: commercialization of apparatus, test method development, inclusion in MIL Specifications (e.g. MIL-PRF-32239, “COATING SYSTEM, ADVANCED PERFORMANCE, FOR AEROSPACE APPLICATIONS”)
Programmable Accelerated Environmental Test System for Aerospace Materials

Expected Advantages:
• Improved correlation between test results and service performance
• Failure modes similar to those observed in service
• Accelerated test times compared to outdoor exposure
• Ability to simulate environmental conditions for specific operational and test locations (e.g. Hickam AFB, Daytona Beach)
• Programmable and fully automated

Existing test standards can be modified and tailored to specific applications
• Example: ASTM D7869 – (Xenon Arc UV + water spray)

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<th>Step Number</th>
<th>Step Minutes</th>
<th>Function</th>
<th>Irradiance Set Point(^a) at 340 nm W/(m(^2)-nm)</th>
<th>Black Panel Temperature Set Point(^a)</th>
<th>Chamber Air Temperature Set Point(^a)</th>
<th>Relative Humidity Set Point(^a)</th>
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<td>—</td>
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<td>95 %</td>
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<td>0.40</td>
<td>—</td>
<td>42°C</td>
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<tr>
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<td>50°C</td>
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<td>50 %</td>
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<td>Repeat subcycle steps 6 to 9 (shown in bold) an additional 3 times (for a total or 24 h – 1 cycle).</td>
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\(^a\) Process variables can be modified as required.
Corrosion and Coating Evaluation (CorRES) System

**Standard Test Panel**, maintains continuity with existing MIL SPEC and other standardized specification requirements

**Mass Loss Specimen**

**Wireless Interface**, receive and transmit corrosion sensor data

**Accelerated laboratory test chamber or outdoor exposure environment**

**Multi-Sensor Panel**, measures coating effects on free corrosion and galvanic corrosion along with coating barrier properties,

**Instrumented Test Rack**, sealed enclosure containing wireless instrumentation

**Corrosion Fatigue Module**, fracture mechanics sample for mechanical testing (fatigue)

**Base Station**, test configuration, control and data management with automated data processing to visualize and assess damage

POC: David Ellicks, AFRL/AFCPCO
AFRL Structural Component Corrosion Simulation (SCCS)

- Driven by ASIP requirements for fleet corrosion management, especially with emerging environmentally compliant materials and processes

- Specimen will have representative materials and geometries

- Test will combine stress with simulated aircraft environment that includes T, RH%, wet/dry cycles, UV, and background gases (ozone, CO₂, etc.)

- Deliverable will be JTP that prescribes:
  - Specimen design and construction materials
  - Finish system – organic coatings, sealants, CPCs, etc.
  - Laboratory exposures to simulated environments
  - Non-destructive inspection during testing
  - Teardown and analysis protocol
Baseline study: representative large airframe legacy aircraft materials selected; “worst case” condition

- Bare 7075-T6 skin, stiffener, splice plate
- Cd-plated steel fasteners
- Dry-installed fasteners; no fay surface sealants or CPCs
  - Chromated and non-chromated coating systems

- Specimens subjected to alternating ASTM B117 salt fog, UV (500 hrs, UVB), axial cyclic loading with temp. cycling -65°F to 85°F
- All relevant control groups (64 total specimens)
- NDI during testing with complete teardown analysis

Fatigue loading:

- $R = 0.05$, $f = 5$ Hz
- 11.7 ksi peak stress for 5,000 cycles per block
- 2 full temperature cycles per block (-65°F to 85°F)
- Purpose of loading is to stress the coating to initiate corrosion
AFRL Structural Component Corrosion Simulation (SCCS)

Mismatched Fastener

Counter Sink & Fastener Match

Lens Z20:X100

0.02 inch
Non-Destructive Inspection

Pulsed Thermography

X-Ray Computed Tomography

Computed Radiography

SCCS Specimen
Reference Standard

Specimen Location
Tube Target
37.5 inches
Corrosion modeling

• Commercially available software (e.g. GalvanicMaster) uses finite element models and electrochemical data input and can provide prediction of initial corrosion rates for a metallic structure assembly, given a set of assumptions

• AFRL 2014.1 Phase I SBIR “Galvanic Corrosion Prediction of Aircraft Structures” to expand capability to aluminum structure/composite joints with fasteners

• Eventual goal is to allow for dynamic prediction and include coatings, sealants, corrosion preventative compounds, etc.
Conclusions

- Air Force need/requirement for rapid (< 1 month) evaluation of aircraft corrosion protection schemes to comply with MIL-STD-1530C and hazardous material elimination demands
- No methods or test apparatus exist that can simulate service conditions/accurate degradation mechanisms in the laboratory!
- Multiple AFRL projects/programs addressing this gap
- Desired end state: accurate forecast of service performance of corrosion protection scheme via improved test protocols (informed by corrosion/coatings science and computational models).
Backup
• Part of DoD Corrosion Forum
  – DoD Corrosion Policy and Oversight Office (under OUSD AT&L)
  – Meet 3-4 times annually
  – Tri-service participation

• Goal is to create a product: **White Paper Summary**
  that includes:
  • Define the current state-of-the-art of subject
  • Identify gaps and needs, and recommend next steps
  • Grand vision – consider the level of technical maturity or complexity of the product necessary to solve “the” DoD problem

• Five year plan detailing an investment strategy
Cumulative corrosion damage models (developed using computer simulations) consider actual variable environmental conditions...
- Approach is analogous to random amplitude fatigue modeling

Optimized model resulting from PhD research program focused on AISI 1010 Steel
- Non-optimized models have also been created for copper and 2024, 6061, and 7075 aluminum alloys
  - Annual cumulative predictions (for all materials) were made for over 110 C-5 deployment locations world-wide

Ongoing internship program sponsored by the DoD HPC program is using a supercomputer to further optimize all models

Cumulative predictions not limited to single locations
- The same approach could be used to estimate environmental attack on aircraft that fly between bases
  - Would need dates and times on the ground to account for diurnal and seasonal temperature changes and related changes to humidity levels
Cumulative Damage Model for Prediction of Atmospheric Corrosion

\[
K_i = \exp \left( \frac{\Delta H}{kT} \right) A_{CL} T^{\alpha_{CL}} f_{CL}(T, RH) f(T, Cl) + A_{SO2} T^{\alpha_{SO2}} f_{SO2}(T, RH) f(T, SO_2) + A_{O3} T^{\alpha_{O3}} f_{O3}(T, RH) f(T, O_3)
\]

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<th>Description</th>
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<td>Hourly corrosion rate</td>
<td>(\mu g/cm^2)</td>
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<td>Scaling factor for the chloride reaction</td>
<td>(\mu g/cm^2)</td>
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<tr>
<td>(A_{SO2})</td>
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<td>(\mu g/cm^2)</td>
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<tr>
<td>(A_{O3})</td>
<td>Scaling factor for the ozone reaction</td>
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<td>Temperature adjustment exponent used for the chloride reaction</td>
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<tr>
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<td>Temperature adjustment exponent used for the SO(_2) reaction</td>
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</tr>
<tr>
<td>(T)</td>
<td>Temperature</td>
<td>Kelvin (K)</td>
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<td>(\Delta H)</td>
<td>Activation energy for the single activation energy formulation</td>
<td>eV/K</td>
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<tr>
<td>(K)</td>
<td>Boltzmann constant ((=8.6173 \times 10^{-5} \text{ eV/K}))</td>
<td>eV/K</td>
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<td>Temperature-Relative Humidity shape function for the chloride reaction.</td>
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<td>(f_{SO2}(T,RH))</td>
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<td>(f_{O3}(T,RH))</td>
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<tr>
<td>(f_{CL}(T,Cl))</td>
<td>Temperature-Contaminant shape function for the chloride reaction. Calibrated using chloride deposition measurements (mass per unit volume of rainwater(^*))</td>
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<td>(f_{O3}(T,O_3))</td>
<td>Temperature-Contaminant shape function for the ozone reaction. Calibrated using hourly gaseous measurements (ppm) measured by automated air pollution monitoring systems.</td>
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DoD Corrosion “Gap” Analysis

• Air Force materials and processes subject matter experts met Sep 2011 to discuss corrosion

• Identified gaps/needs:
  • Ability to translate top-level service life (hours and years in service) and sustainment requirements into selection of materials, finish systems, etc. that withstands competing pressures during design
  • Well defined and agreed-to accelerated test methods and accept/reject criteria for corrosion evaluation for a range of environments and service life requirements
  • DoD-wide evaluation & recommendation/approval for cross-cutting material substitutions, process changes, such as:
    – Material substitutions: chromated primer, chromic acid anodize, chrome plating, cadmium plating
    – Process changes: paint removal (chemical, plastic media, laser, etc.)
Pulsed Thermography—Uses pulsed thermal excitation and infrared camera to image the coated surface. Detects corrosion formation at coating to substrate interface.
Computed Radiography—X-ray 2D digital imaging to identify inter-layer material loss and provide rough estimates of thickness loss and area.

Flat bottom holes to calibrate thickness loss.
Computed Tomography (CT) - High resolution 3D imaging of material loss. Anticipated to provide accurate measure of thickness loss within individual layers of the SCCS specimens without disassembly.
AFRL X-Ray Computed Tomography

• North Star Imaging (NSI) X50 CT System (Enclosed cabinet)
  – Ability to move detector closer or further away from tube/stage
  – Tube is Fixed
  – Scanning envelope for stage: X (up/down), Y (left/right), Tilt (+20 / -10°; 20 to detector / 10 to source), and Rotate (Continuous 360°)

• FeinFocus FXE 225.48 Micro-focus X-ray Tube
  – 225 kV
  – 3 mA

• Perkin Elmer XRD 0822 AO Digital X-Ray Flat Panel Detector
  – Field of View: 8” x 8”

• Stage
  – Diameter: 8 inches
  – Load Limit: 25 lbs.

• System Resolution
  – ~ 1 μm voxel size (depends on distance to x-ray tube, size of part)
  – ~ 4 μm voxel size (best resolution we have achieved on a component)
Electrochemical Setup

- Working Electrode
  - Mounted Sample

- Counter Electrode
  - Platinum

- Reference Electrode
  - SCE .241V vs. SHE