Accelerated Corrosion Test Method Development

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Outline

- Summary of SERDP WP-1673 findings
- First attempt at an accelerated test
- Summary of ESTCP WP18-5087

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Summary of SERDP WP-1673 Findings

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Problem Statement

- Hexavalent chromium (Cr^{6+}) is particularly effective in reducing corrosion, and is currently found in many DoD assets.
- However, hexavalent chromium is a known carcinogen and environmental toxin, and it is facing increased regulation.
- The need to identify and qualify alternatives to formulations based on hexavalent chromium (Cr^{6+}) – e.g., conversion coatings, primers, etc. – necessitates the ability to replicate, in an accelerated manner, the damage that materials and coating systems experience in operational environments.
- Outdoor exposure testing is the most effective at replicating field damage, but it is time-consuming and not widely accessible.
- Although several accelerated corrosion tests (ACT) are available for DoD use, none of them replicate damage observed in field environments (e.g., ASTM B117, ASTM G85, ASTM 810, etc.).
- In addition, current ACT is well known for chamber inconsistencies, which are more pronounced during cyclic tests.

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Objective

- Create an improved accelerated corrosion test method that can
  - Accurately replicate corrosion damage modes
  - Accurately rank material performance under atmospheric conditions
  - Control severity
  - Reduce development time for new corrosion mitigation technologies

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Acknowledgements

- Funding was provided by SERDP Weapon Systems and Platforms Program (Robing Nissan, PM) under project WP-1673
- Teamed with Government Labs, University, and Industry

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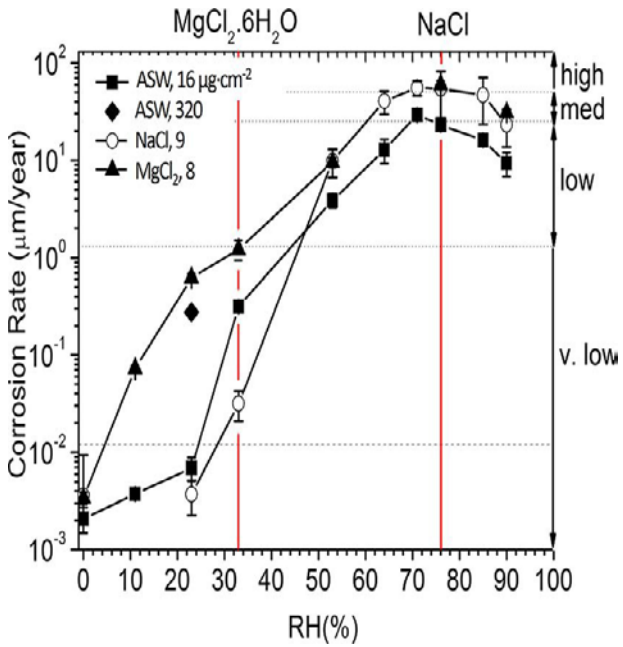
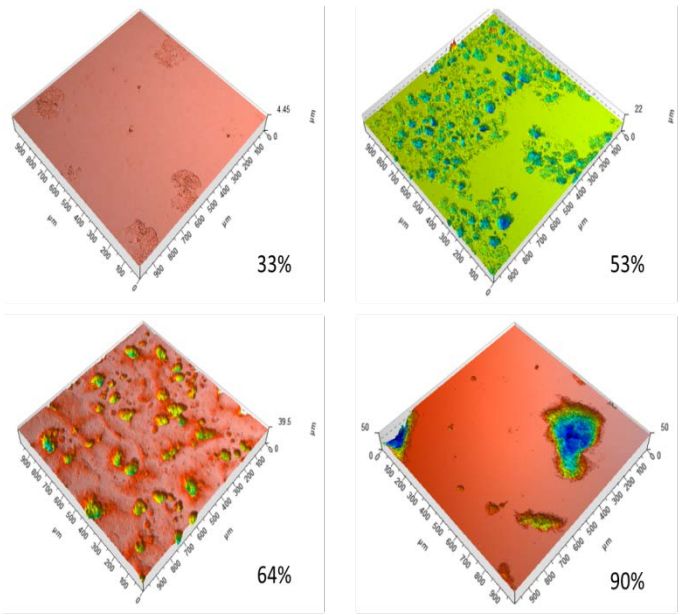
Corrosion below DRH





- Corrosion of steel occurs at reasonable rates down to 50% RH
- Corrosion morphology is strongly affected by RH

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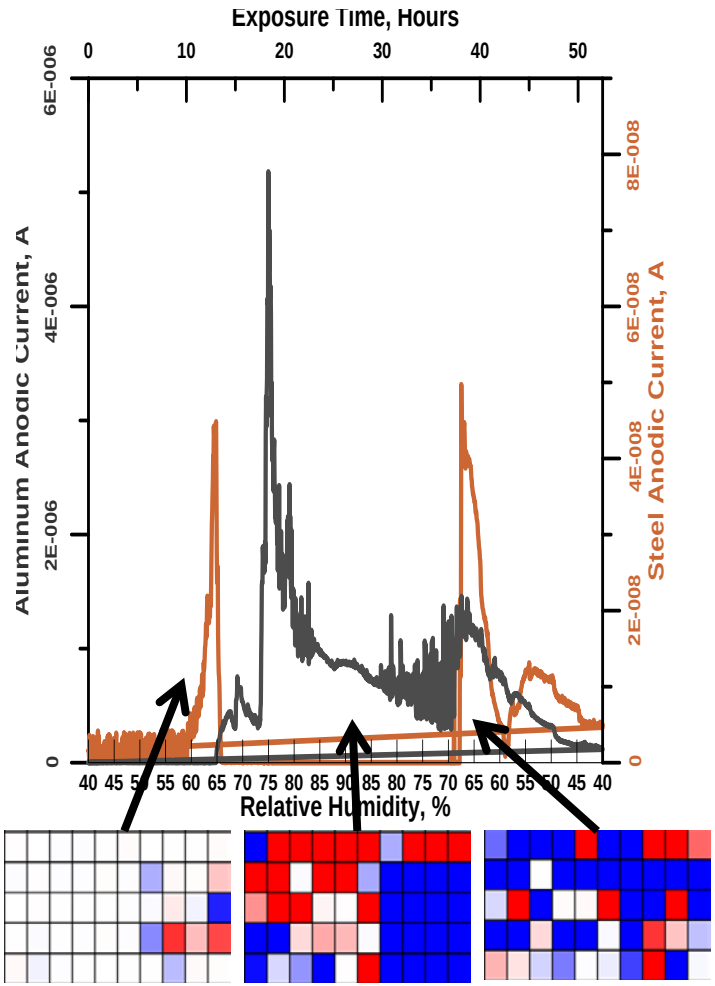
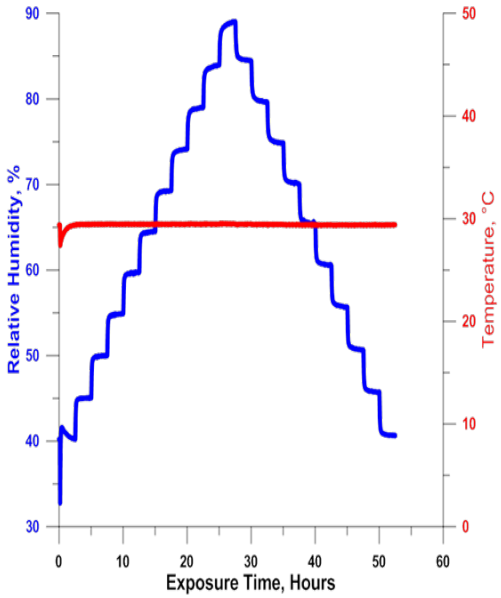
RH Affects Galvanic Coupling

- Corrosion of steel occurs below DRH
- Galvanic coupling primarily above the DRH
  - Continuous water layer forms
  - Al 2024 corrosion initiates



60% RH 90% RH 65% RH



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RH Affects Surface Morphology

- Degradation mechanisms can be triggered by the right combination of humidity cycling:

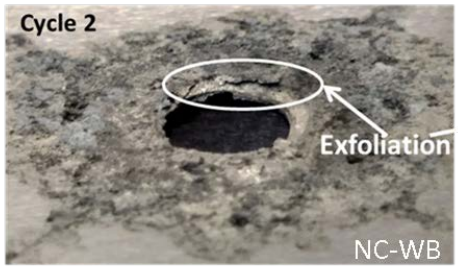
- Accelerated Test Cycle Conditions, T = 49° C
- TOW (RH> 76%) = 67% of total exposure time
- Salt deposition: 0.6M NaCl, pH = 3, salt dip = 15 minutes

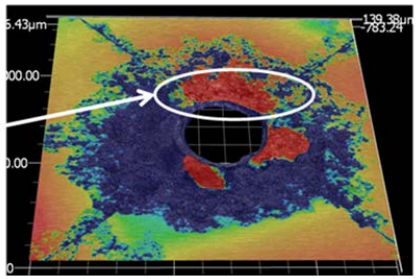
	Cycle 1	Cycle 2	Cycle 3
Max RH	90%	90%	90%
Min RH	40%	65%	40%
Max RH Time (hr)	2	2	8
Min RH Time (hr)	1	1	4
Salt Dip Frequency (per wk)	1	1	1

Effect of degree of drying

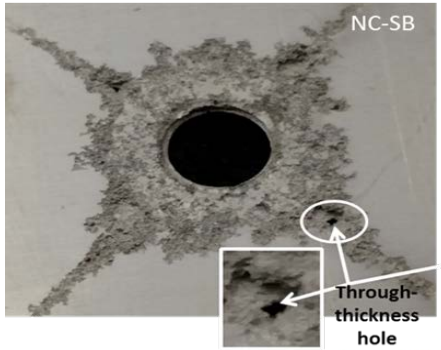
Effect of frequency of cycles and high RH dwell time

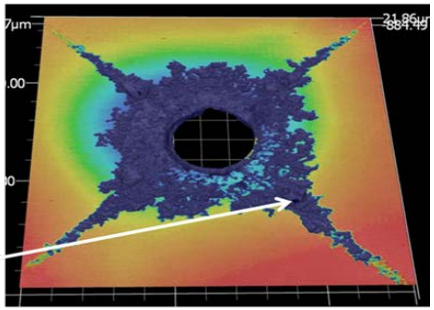
- **Cycle 1:** very shallow damage restricted to the uppermost surface layer.
- **Cycle 2:** Exfoliation, extensive coating delamination and material volume loss.





- **Cycle 3:** Deep corrosion trenches along fastener and significant pitting.





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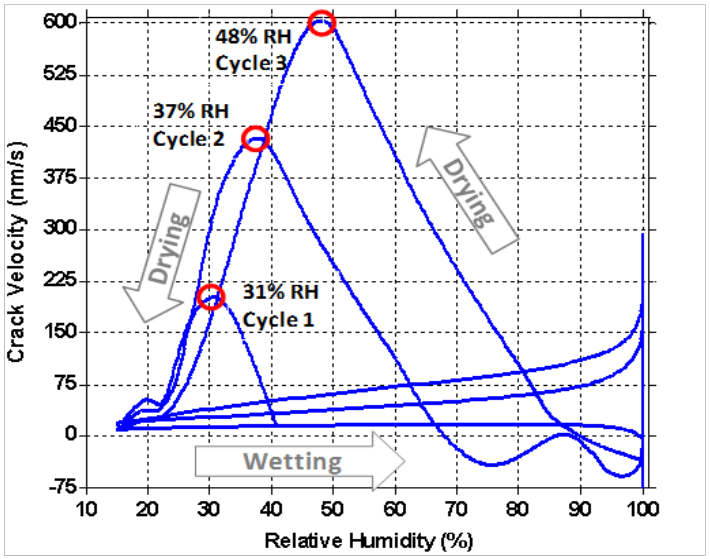
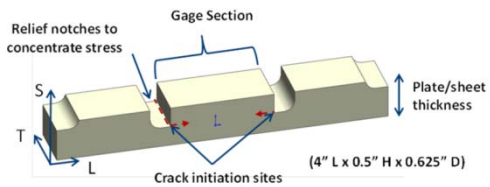
RH Affects SCC

- Chemistry and RH strongly affect degree of corrosion within a fastener hole
- Cyclic RH strongly affects SCC



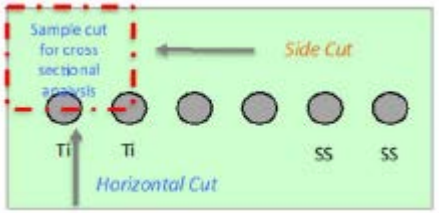
Sensitized AA5083 in modified ASTM G85-A5.



Pt. Judith
7075-T6
Al/SS
12 months

G85-A5
7075-T6
Al/SS
1,000 hrs

Experiment 1
7075-T6
Al/SS
1,000 hrs



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How Can We Improve Standardized Testing

- Main parameters affecting corrosion:
  - RH – defines periods when **active** electrolyte exists
  - RH Duty Cycle – determines attack **morphology**
  - Electrolyte chemistry and deposition rate – determines **degree** of attack
- Based on laboratory testing and comparison with outdoor exposure, to replicate costal damage
  - MUST includes cycles
  - RH must be > DRH over 50% of the time
  - The test must spend > 25% of the time 50% < RH < DRH
  - pH < 5
  - Dial up aggressiveness by combination of time RH > DRH and solution chemistry
- Traditional cyclic tests do not include specific control of RH

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First Attempt at an Accelerated Test

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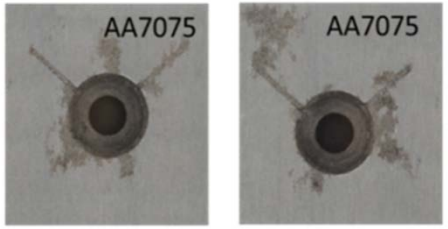
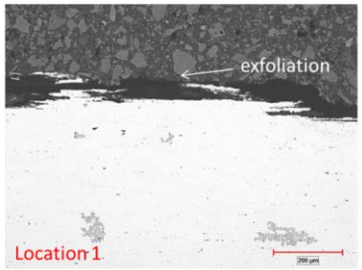
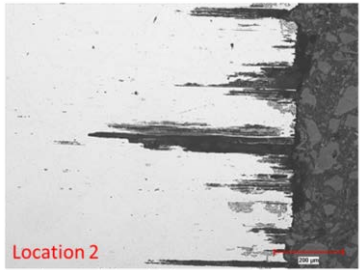
1,000 hour Exposure to NE#1

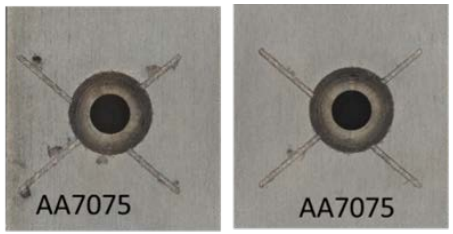
Solvent Borne System

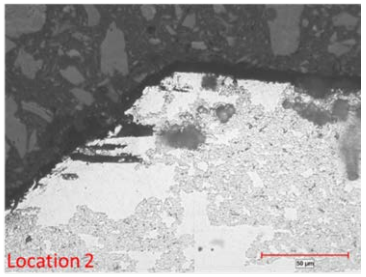




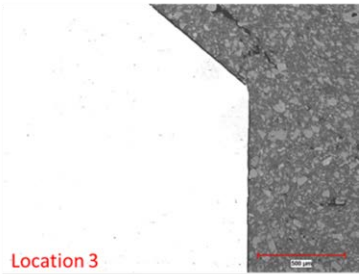
Pt. Judith







NE#1



Surface Attack

Fastener Hole Attack

Proposed Chemistries*

Reagent	g/L (Reagent) NE#1	g/L (Reagent) NE#2
NaCl	24.53	22.26
$MgCl_2.6H_2O$	11.10	11.10
Na_2SO_4	4.00	4.00
$NaNO_3$		3.27
HCl (1N)	(1 ml) (pH=3)	(1 ml) (pH=3)

*Normalized to ionic strength.

	Description
Step 1	15 minute spray
Step 2	45 minute hold at 80% RH
Step 3	25 minute ramp to 40% RH
Step 4	35 minute hold at 40% RH

	Cumulative SurfaceDamage (μm^3)		
PRIMER	NE#1	DAYTONA	Pt.J
SB	1.20×10^7	1.20×10^9	4.75×10^9
WB	1.71×10^8	1.85×10^9	1.45×10^9

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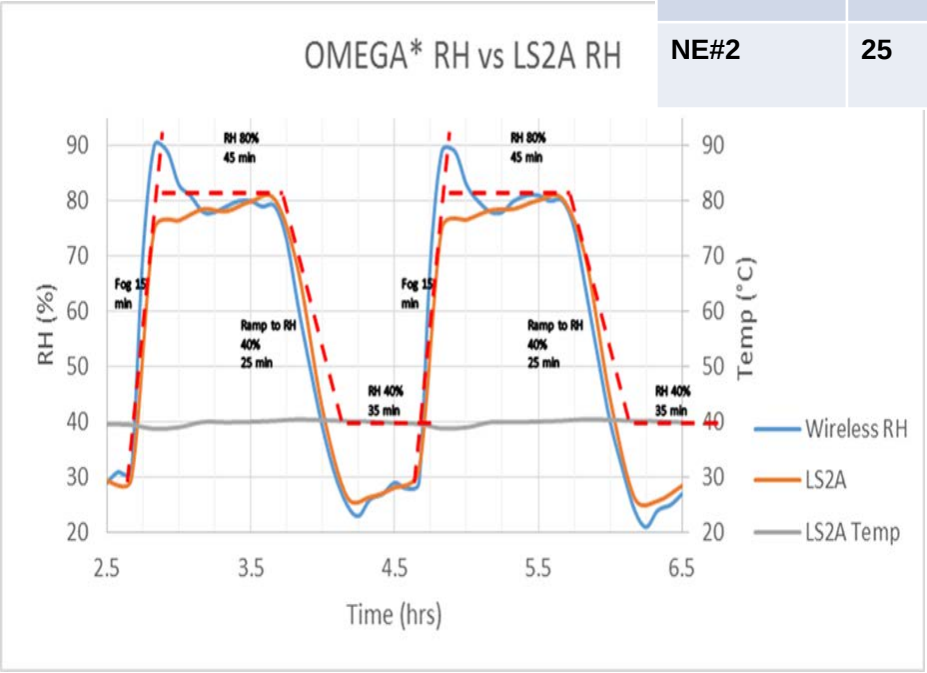
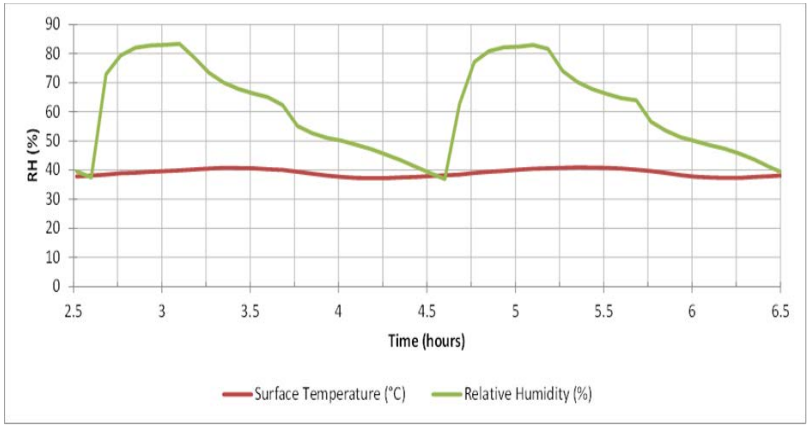
What Went Wrong?

- Damage in NE#1 matched what is observed after 3 years in LAX (low TOW + high salt load)
- Time above DRH was < 50% so despite aggressive electrolyte, not wet enough (no corrosion observed in NE#2)
- **Note significantly different chamber response!!!**

	RH > DRH	50% < RH < DRH	RH < 50%	Comment
NE#1	45	13	42	Mod sea salt exposure, 15 min spray, RH control, 12 cycle/day, pH = 3
NE#2	25	43	32	Mod sea salt with nitrate, 30 min fog, wet bottom RH, 12 cycles/day, pH = 3





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Summary of ESTCP WP18-5087

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Validation and Demonstration of Relative Humidity Controlled Accelerated Corrosion Test

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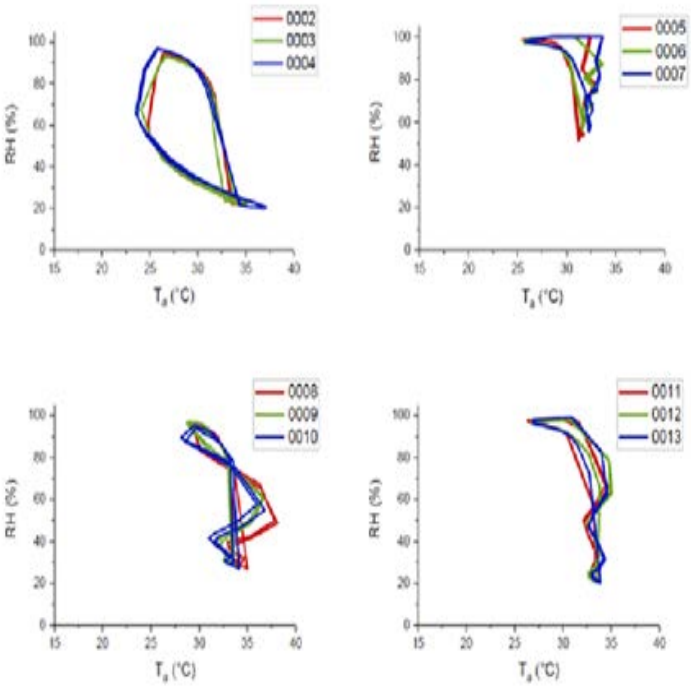
What's Next?

- The innovative aspect of this work is the control of cyclic variation of relative humidity and the periodic salt deposition of salt solution within each cycle
- Traditional DoD cyclic tests do not include this degree of control, limiting their usefulness and introducing a high degree of variation in test results
- A proposed ESTCP effort is designed to develop existing corrosion tests sufficiently to allow its widespread use in testing labs across the DoD, original equipment manufacturers, DoD contractors, and other testing labs.
  - **Objective 1**: Mature the accelerated corrosion test protocol developed during the SERDP WP-1673, WP-1674, and SERDP WP-2521 efforts*.
  - **Objective 2**: Characterize protocol variability by means of round robin testing with participation from DoD laboratories, contractors, and private industry.
  - **Objective 3**: Establish a national standard to be used throughout the acquisition life cycle for testing and qualification of corrosion prevention schemes.



Luna LS2A sensor monitoring ASTM G8-A5 chamber RH and Temperature in 4 laboratories (funding from AFCPO))

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Related Efforts

The following efforts not only reflect the expertise within the proposal team to successfully carry out this effort, as indicated, but they also provide support that will be directly leveraged with the proposed ESTCP effort **(>$12M**).

- SERDP WP-1673 "Development of a Dynamic Accelerated Corrosion Test Method"
- SEDRP WP-1674 "Dynamic Multivariate Accelerated Corrosion Test Protocol"
- SERDP WP-2521 "Standardized Test Methodologies for Low Observable Coating Durability"
- ONR "Development of Test Methodologies for Degradation and Corrosion Models".
- ESTCP WP-201710 "Corrosion Monitoring System to Reduce Environmental Burden and Corrosion Maintenance Costs"
- AFMC/LCMC FA8604-16-C-7009 "Support for AFLCMC Corrosion Sensor Implementation into CBM+"
- SwRI Internal Research "Analysis of Corrosion Damage on Samples Exposed to Accelerated and Outdoor Corrosion Environments"
- Boeing Internal Application Development (IAD), Fast Lean Environmental Cell Project
- Q-Lab Internal Research and Development: "Development of a Salt Fog Chamber Capable of Controlling RH"
- Q-Lab Internal Research and Development: "Repeatability and Reliability of Mass Loss Coupons"
- Q-Lab Internal Research and Development: "Independent Verification of C/RH Control System Environmental Parameters"

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Task 1: Optimization of Cycle Period

- Although the test protocol provides good agreement for several milder exposure locations, previous results indicate that small shifts in cycle duration and solution chemistry may have large effects on total damage. Therefore, a test protocol to simulate more aggressive environments will be developed.
- **Two chemistries** and **three RH cycles** will be tested. The cycles will be defined by NAVAIR, SwRI, Boeing, ARL, and AFRL. Testing during for this task will be performed be NAVAIR and SwRI.
- Samples designed during SERDP WP-1673 will be used in this program. Samples include a lap-joint and fasteners to mimic non-ideal in-service equipment configurations.
- Image analysis techniques will be used to quantify and compare surface damage, volume loss, and damage within fastener holes
- Goals:
  - Distinguish coating performance within 1,000 hours
  - Define conditions for a severe and moderate environment

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Task 2: Round Robin Testing

- This task constitutes the centerpiece of the program. The **objective** of this task is to **determine** the amount of **variation** that should be expected from this **test method**.
- Eight testing laboratories will participate (NAVAIR, SwRI, Boeing, ARL, AMCOM, AFRL, AFLCMC, Q-Lab).
  - Required **minimum** number of labs for ASTM E691 inter-laboratory studies for round robin validity.
  - At the completion of testing, **repeatability and reproducibility** estimates will be obtained following ASTM E691 to determine test precision.
- Specimen material and coating systems will include common coating systems used on military **aircraft** and tested in Task 1 (MIL-PRF-85582 Class N, MIL-PRF-23377 Class C and Class N) and ground assets.
- Specimens for **ground-based equipment** will include a steel and aluminum panels and will use MIL-DTL-53022 Type IV, and MIL-PRF-32550 Form A coatings
- **This round robin task is crucial for the development of the specification** since it will provide the inter- and intra-laboratory statistics that will support the standard.
- Goal: No outlier laboratories

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Task 3: Standard Development and Acceptance

- The **objective** of this task is to **formalize** the test procedure into a NACE International laboratory accelerated corrosion **test standard**.
- A society standard offers several advantages over a MIL-STD:
  - OEMs can participate in development and drafting of the standard, thus reducing resistance to acceptance.
  - Society standards require revisions every three to five years. This means the standard can be updated as new technical developments are made.
  - If DoD components wanted a MIL-STD version, it is easier to convert a pre-vetted standard.
- The approach is to create a NACE Task Group (TG) within TEG 189x "Atmospheric Corrosion"
- Victor Rodriguez-Santiago (PI, NAVAIR), Sean Fowler (Q-Lab), James Dante (SwRI, PI from WP-1673) and Kris Williams (Boeing, Key personnel on WP-2521) will serve on the TG as representatives from this ESTCP effort.
- Specification layout
  - The front end of the specification will define the overall testing approach.
  - Specific environmental test cycles will be included within the annex section of the specification.
  - Because of the flexible nature of the specification, additional annexes can be created to include other types of environments

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Expected DoD Benefit

- The benefit of creating a more representative accelerated corrosion test standard is two-fold:
  1. It facilitates **ease of implementation** of corrosion prevention technologies. Currently, prospective technologies are developed much faster than they can be tested and qualified. Prospective coatings are not implemented—even on a trial basis—until years of outdoor exposure data are generated.
  2. It provides a **better understanding of risk.** Currently, the field of corrosion engineering does not have a quantitative way of assessing risk. Current testing methodologies do not replicate damage seen in outdoor exposure tests or damage observed during aircraft maintenance. Thus assessing the long term performance of corrosion prevention schemes is not possible.
- This test method aims to provide representative predictions of corrosion within 1000 hours, reducing initial screening time and the need for lengthy field exposures prior to trial applications.

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Technology Transition

- The methodology developed in this effort will transition in the form of a new ACT test standard through NACE. Support in NACE for the standard has already been procured, with the relevant technical committees, STG 60 ("Corrosion Mechanisms"), STG 41 ("Electric Utility Generation, Transmission, and Distribution"), and STG 2 ("Coatings and Linings"). The specification will be developed under TEG 189x.
- NAVAIR will serve as the adoption agency for the NACE standard, enabling its use by the other military services and agencies throughout the DoD. By virtue of being a society standard, it will be immediately available to commercial companies and OEMs.
- NAVAIR is the preparing activity for numerous coatings and corrosion specifications and qualifies products to them, thus transition to a new test method will be facilitated via its addition to existing military standards (e.g., MIL-STD-7179, MIL-STD-1568).
- Technology transition into the Air Force will be accomplished through validation and inclusion of the newly developed test method into MIL-PRF-32239A, *Performance Specification, Coating System, Advanced Performance, for Aerospace Applications* (2014). AFRL/RXSS has engineering authority for this specification and is part of the round robin testing group.
- ARL maintains the MIL-DTL-53072, which controls the CARC coatings required on all Army equipment.

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