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Accelerated Corrosion and Ageing Studies

April 25, 2019

SERDP DOD • EPA • DOE | ESTCP

Welcome and Introductions

Jennifer Nyman, Ph.D., P.E.
Webinar Facilitator

SERDP DOD • EPA • DOE | ESTCP

Webinar Agenda

- **Webinar Logistics** (5 minutes)
  Dr. Jennifer Nyman, Geosyntec Consultants
- **Overview of SERDP and ESTCP** (5 minutes)
  Dr. Kurt Preston, SERDP and ESTCP
- **Cyclic Corrosion and Failure Mechanisms** (25 minutes + Q&A)
  Mr. James Dante, Southwest Research Institute
- **Development and Validation of a Cyclic Humidity Corrosion Test** (25 minutes + Q&A)
  Dr. Victor Rodriguez-Santiago, Naval Air Systems Command
- **Final Q&A session**

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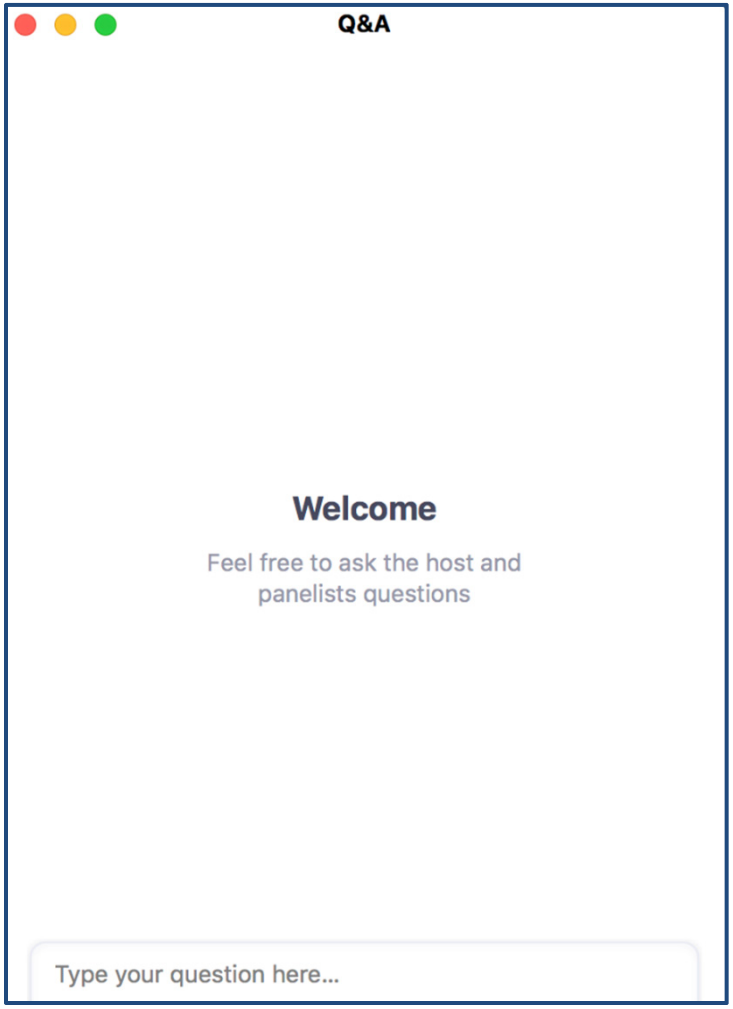

SERDP and ESTCP Overview

Kurt Preston, Ph.D.
Resource Conservation and Resiliency Program Manager

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SERDP

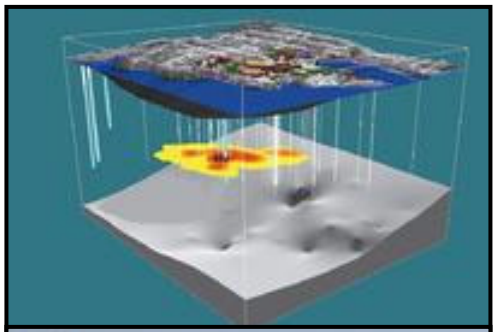
- Strategic Environmental Research and Development Program
- Established by Congress in FY 1991
  - DoD, DOE and EPA partnership
- SERDP is a requirements driven program which identifies high-priority environmental science and technology investment opportunities that address DoD requirements
  - Advanced technology development to address near term needs
  - Fundamental research to impact real world environmental management

ESTCP

- Environmental Security Technology Certification Program
- Demonstrate innovative cost-effective environmental and energy technologies
  - Capitalize on past investments
  - Transition technology out of the lab
- Promote implementation
  - Facilitate regulatory acceptance

Program Areas

1. Environmental Restoration
2. Installation Energy and Water
3. Munitions Response
4. Resource Conservation and Resiliency
5. Weapons Systems and Platforms



Weapons Systems and Platforms

- Major focus areas
  - Surface engineering and structural materials
  - Energetic materials and munitions
  - Noise and emissions
  - Waste reduction and treatment in DoD operations
  - Lead free electronics

SERDP and ESTCP Webinar Series

Date	Topic
May 09, 2019	Roles of Soil Microbial Communities in Ecosystem Restoration
May 23, 2019	Treatment Options for the Emerging Contaminants 1,2,3-Trichloropropane and 1,2-Dibromoethane
June 6, 2019	Developing and Demonstrating Non-Toxic Paints for Corrosion Protection
June 20, 2019	Infrastructure Adaptation Strategies to Mitigate Climate Change and Uncertainty
July 11, 2019	Vapor Intrusion: Modeling Tools and Cost Effective Mitigation
July 25, 2019	Building Energy and Water Efficiency Solutions

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SERDP • ESTCP
SYMPOSIUM

A three-day symposium showcasing the latest technologies that enhance DoD's mission through improved environmental and energy performance

December 3-5, 2019

Washington Marriott Wardman Park

Registration open!

Cyclic Corrosion and Failure Mechanisms

James F. Dante
Southwest Research Institute

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Agenda

- Background
- Technical objective
- Technical approach
- Results
- Conclusions
- Benefits to DoD

Background: Anti-Corrosive Coatings

- Hexavalent chromium (Cr^{6+})
  - Particularly effective in reducing corrosion
  - Known carcinogen, environmental toxin
  - Increasingly regulated
- Need to identify alternatives to Cr^{6+} based formulations

Background: Corrosion Testing

- Outdoor exposure testing
  - Most effective at replicating corrosion observed in the field
  - Time-consuming and not widely accessible
- Accelerated corrosion tests (ACT)
  - Several ACT are available for DoD use[1]
  - None fully replicate corrosion observed for multifunctional coating systems

[1] ASTM B117, ASTM G85, ASTM 810, etc.

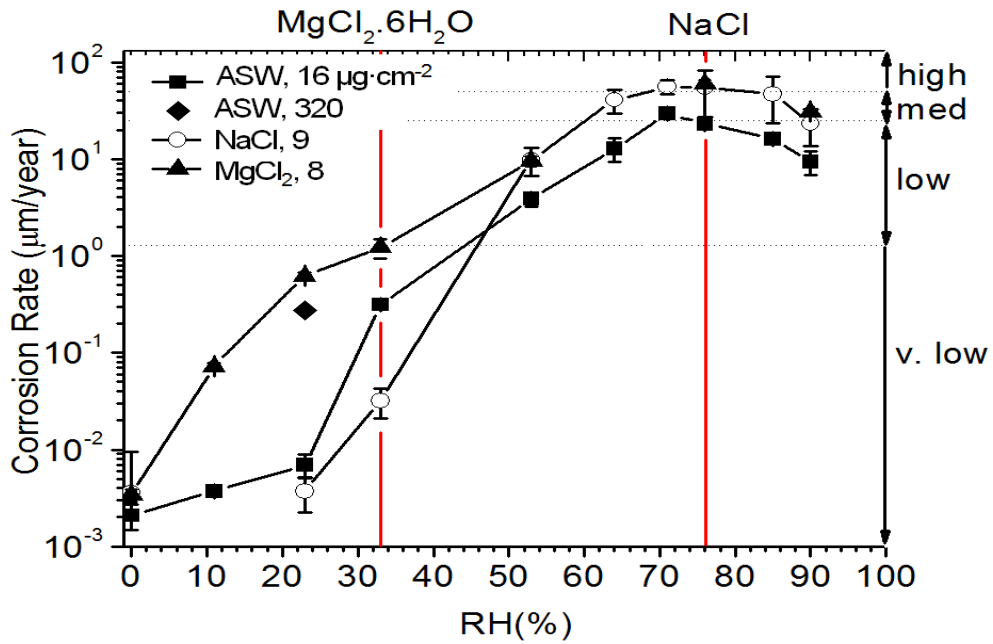
Technical Objective

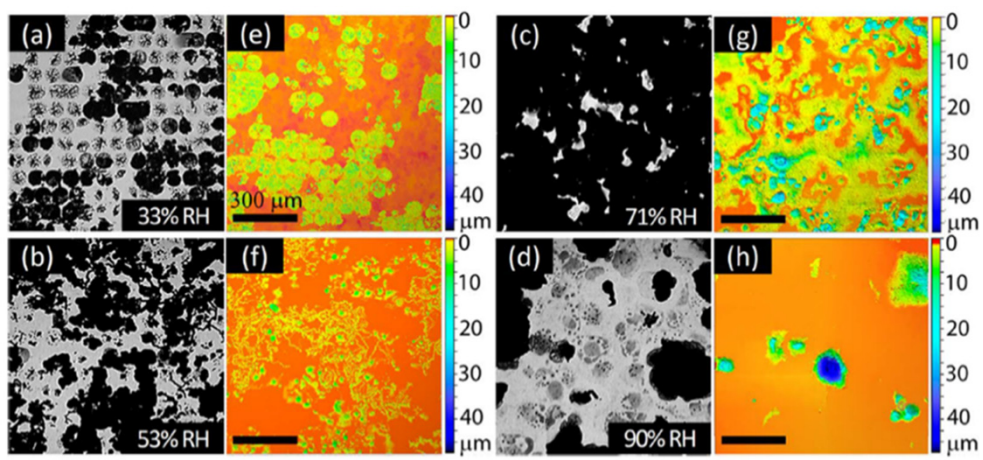
- Create an improved accelerated corrosion test method
  - Accurately replicate corrosion damage modes
  - Accurately rank material performance under atmospheric conditions
  - Control severity
  - Reduce development time for new corrosion mitigation technologies

Technical Approach

- Integrate representative sample designs into accelerated corrosion testing
- Determine the effect of critical environmental and mechanical parameters on degradation modes of system components
- Characterize and compare the development of corrosive environments for “real world” and current accelerated corrosion tests

Corrosion Rate of Steel vs. RH





Corrosion Rate of Steel vs. RH

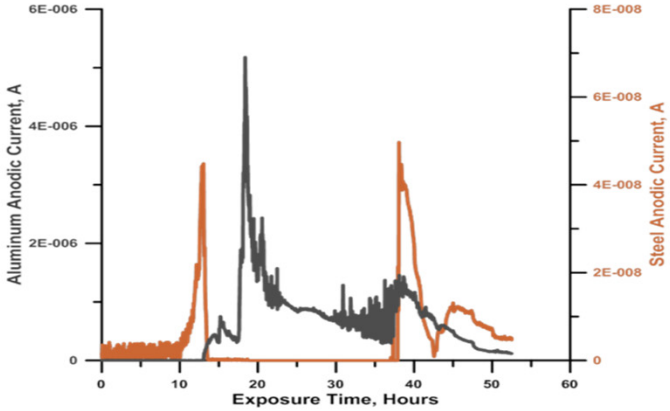
- ASW and $MgCl_2$ provide considerable conductance to <2% RH for up to 24 hours
- Sustained corrosion detectable
  - 11% RH for $MgCl_2$
  - 23% for AS
  - DRHs of contaminant salts do not serve as threshold for wet-dry or significant corrosion
- Large disparity in attack between 33% and 53% RH for NaCl and ASW (Critical RH): linked to ERH and the presence of other salts
- Above DRH, continuous electrolyte forms
  - Cathode moves away from single drop edge
  - Large cathodic drives fewer but deeper pits

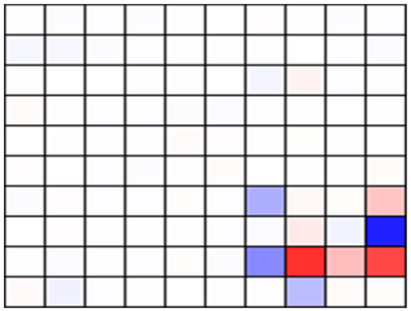
Notes: RH – relative humidity; ASW – artificial sea water; DRH – deliquescence relative humidity; ERH - efflorescence relative humidity

Uninhibited Galvanic Electrode

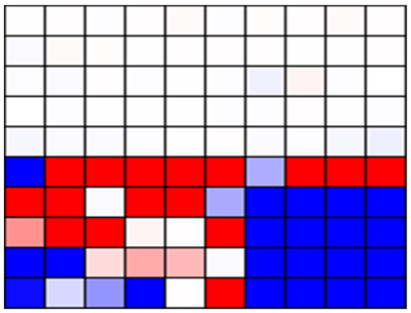
ASTM B117
Constant fog

ASTM G85-A5
Cyclic wet/dry

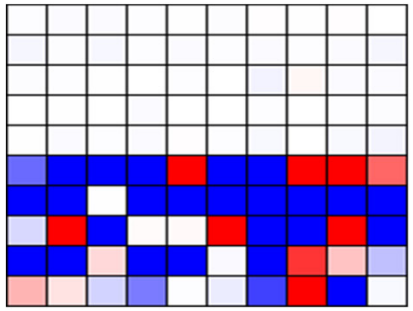




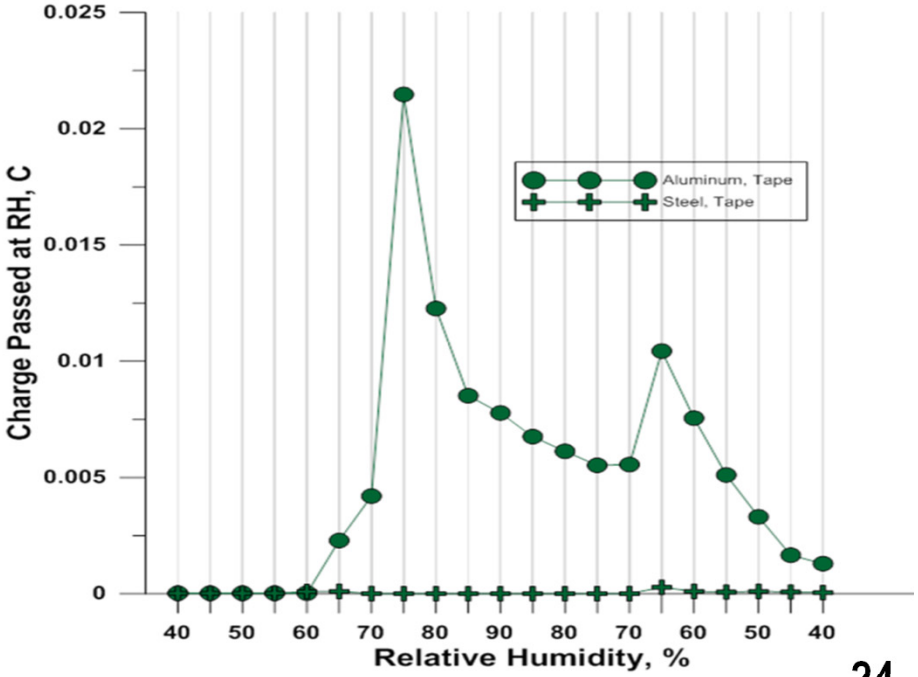
60% RH



90% RH



65% RH



Uninhibited Galvanic Electrode

- Mimicking aircraft corrosion is not easy
  - Steel is anodic ONLY when anodic aluminum current activity is low during wetting and drying transitions
  - Above DRH, steel becomes a cathode and aluminum an anode
- Suggests RH governs galvanic coupling
- Most charge is passed during transition between wet and dry

Surface Morphology of Coated Aluminum

Affected by RH

- Degradation mechanisms triggered by the right combination of humidity cycling
  - Accelerated Test Cycle Conditions, Temp = 49°C
  - TOW (RH> 76%) = 67% of total exposure time
  - Salt deposition: 0.6M NaCl, pH = 3, salt dip = 15 minutes once per week

	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

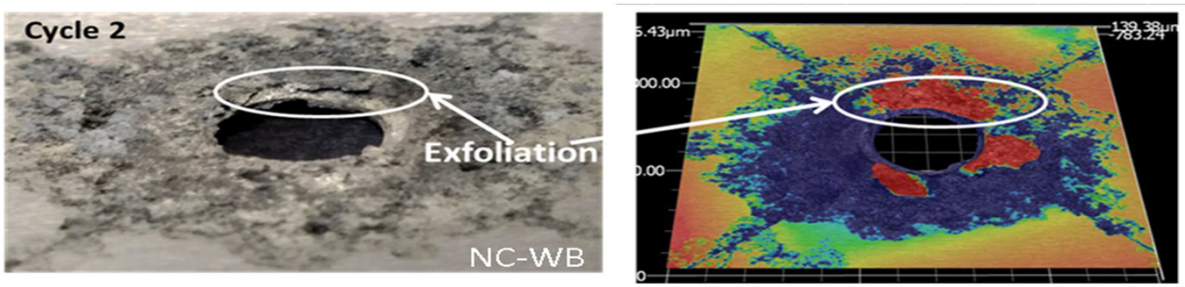
Effect of frequency of cycles and high RH dwell time

Notes: TOW – time of wetness

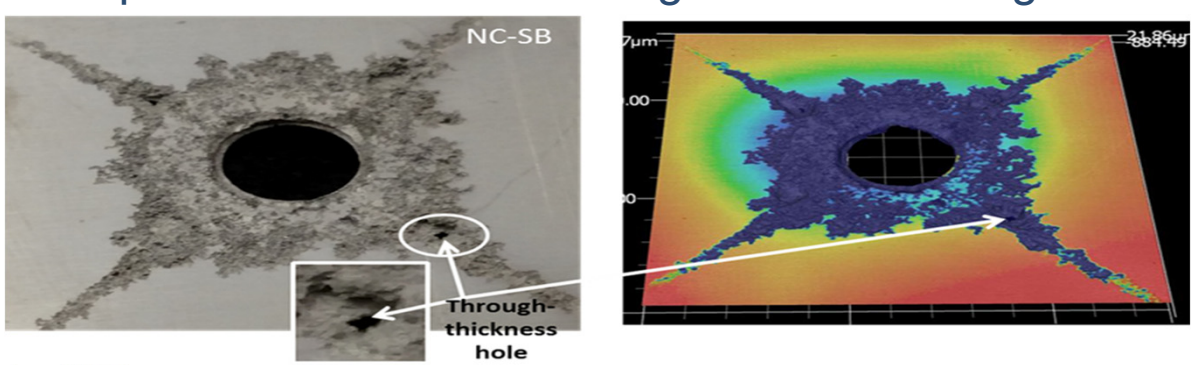
Surface Morphology of Coated Aluminum

Affected by RH

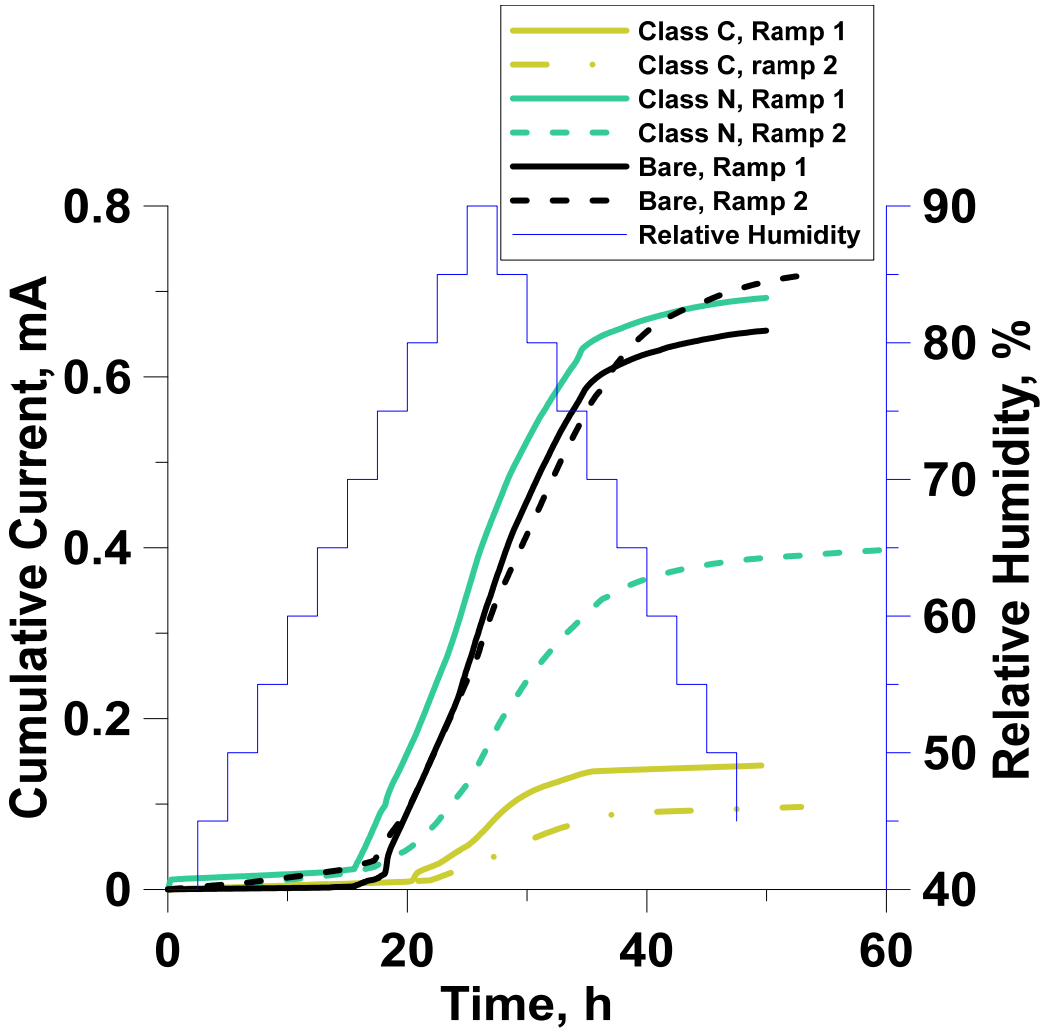
- Degradation mechanisms can be triggered by the right combination of humidity cycling
  - Cycle 1: very shallow damage restricted to the uppermost surface layer with significant coating delamination
  - Cycle 2: Exfoliation, extensive coating delamination and material volume loss



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



RH Effect on Inhibitor Release



RH Effect on Inhibitor Release

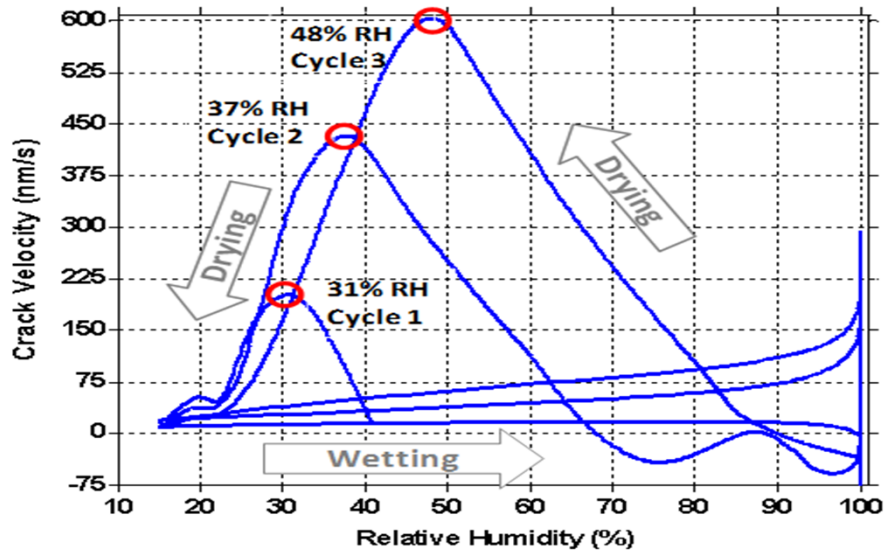
- Cumulative currents
  - Lower for the second ramp than for the first ramp in the presence of a primer
  - Remain relatively unchanged for bare MEA
- Initial wetting of the surface during the high RH periods of the first ramp
  - Initiates inhibitor release
  - Protects the surfaces during subsequent wetting events

	Bare	Class C	Class N
Charge Passed 1st Cycle (C)	0.078	0.017	0.083
Charge Passed 2nd Cycle (C)	0.085	0.011	0.047
Percent of charge passed in 2nd Cycle vs. 1st	109%	66%	56%

Notes: MEA – multi-electrode array

RH Affects IGC and SCC of Aluminum

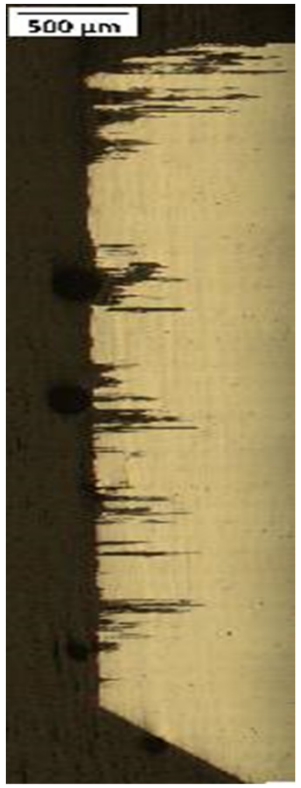
- Cyclic RH strongly affects SCC
  - CGR increase during drying
- Chemistry and RH strongly affect degree of corrosion within a fastener hole



Sensitized AA5083 in modified ASTM G85-A5.

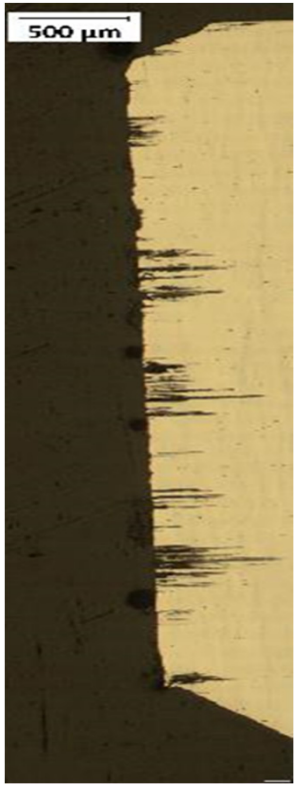
Notes: IGC – intergranular corrosion; SCC – stress corrosion cracking; CGR – crack growth rate

RH Affects IGC and SCC of Aluminum



Pt. Judith
7075-T6
Al/SS
12 months

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



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

Notes: Al/SS – aluminum/stainless steel couple

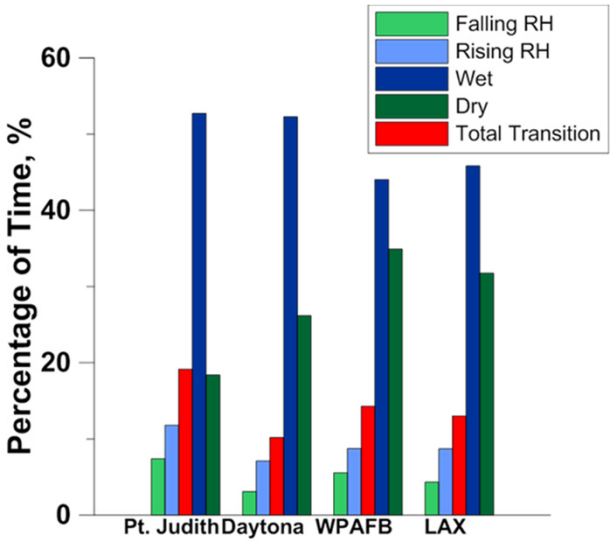
RH Breakdown

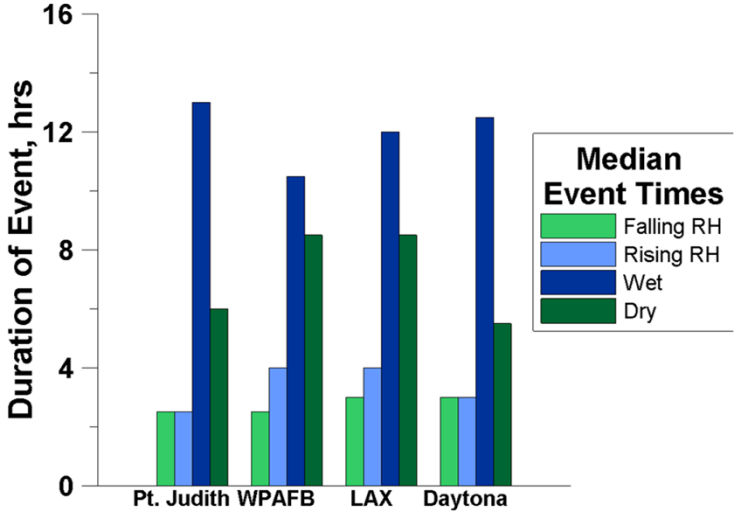
	RH > DRH	50% < RH < DRH	RH < 50%	Comment
Point Judith* (3 years)	53	29	18	Sea salt exposure, saturated
Daytona* (3 years)	52	23	25	Sea salt exposure, saturated
WPAFB* (3 years)	45	20	35	Very low chloride
LAX* (3 years)	46	21	33	high chloride, industrial
ASTM G85-A5* (2000 hrs)	39.5	26.7	34	Dilute NaCl fog, pH = 5, 12 cycles/day
GM9540P (2000 hrs)	56.3	18.3	25.4	High NaCl brief spray, pH=5, 1 cycle/day, diluting fog
ASTM B117 (2000 hrs)	100	0	0	High NaCl, no cycles
Adhesion Testing (Cycle 1) (1400 hrs)	66	0	34	High NaCl, pH=3, dip applied 1/week, 8 cycles/day, high temp, RH controlled
Adhesion Testing (Cycle 2) 1400 hrs)	66	34	0	High NaCl, pH=3, dip applied 1/week, 8 cycles/day, high temp, RH controlled
Adhesion Testing (Cycle 3) (1400 hrs)	66	0	34	High NaCl, pH=3, dip applied 1/week, 2 cycles/day, high temp, RH controlled
UVA Trial 1 (1000 hrs)	33	34	33	High NaCl fog, pH=3, 16 cycles/day, high temp (49° C), modified ASTM G85-A2
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

Mimicking Coating Delamination

Criteria for Accelerated Method

- Must have cyclic RH to mimic observed corrosion failure modes
- RH > DRH for > 50% of the time in natural aggressive environments
- Coating delamination is a strong function of the time of wetness within a given cycle

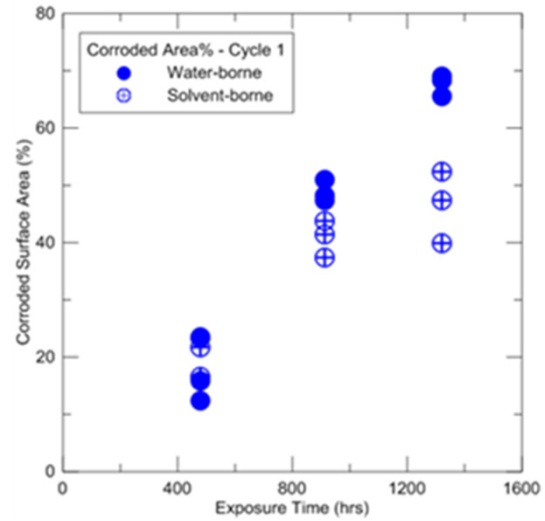


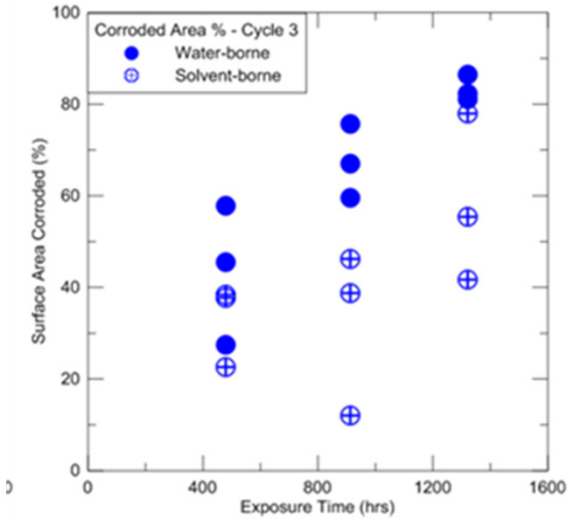


Mimicking Coating Delamination

Criteria for Accelerated Method

- Trade off between solution aggressiveness and percent of time wet and absolute time wet in each cycle
  - Less aggressive solutions need longer absolute wet time
- Increase aggressiveness of delamination
  - pH ≤ 5
  - Increase salt deposition
  - RH > DRH for longer periods of time

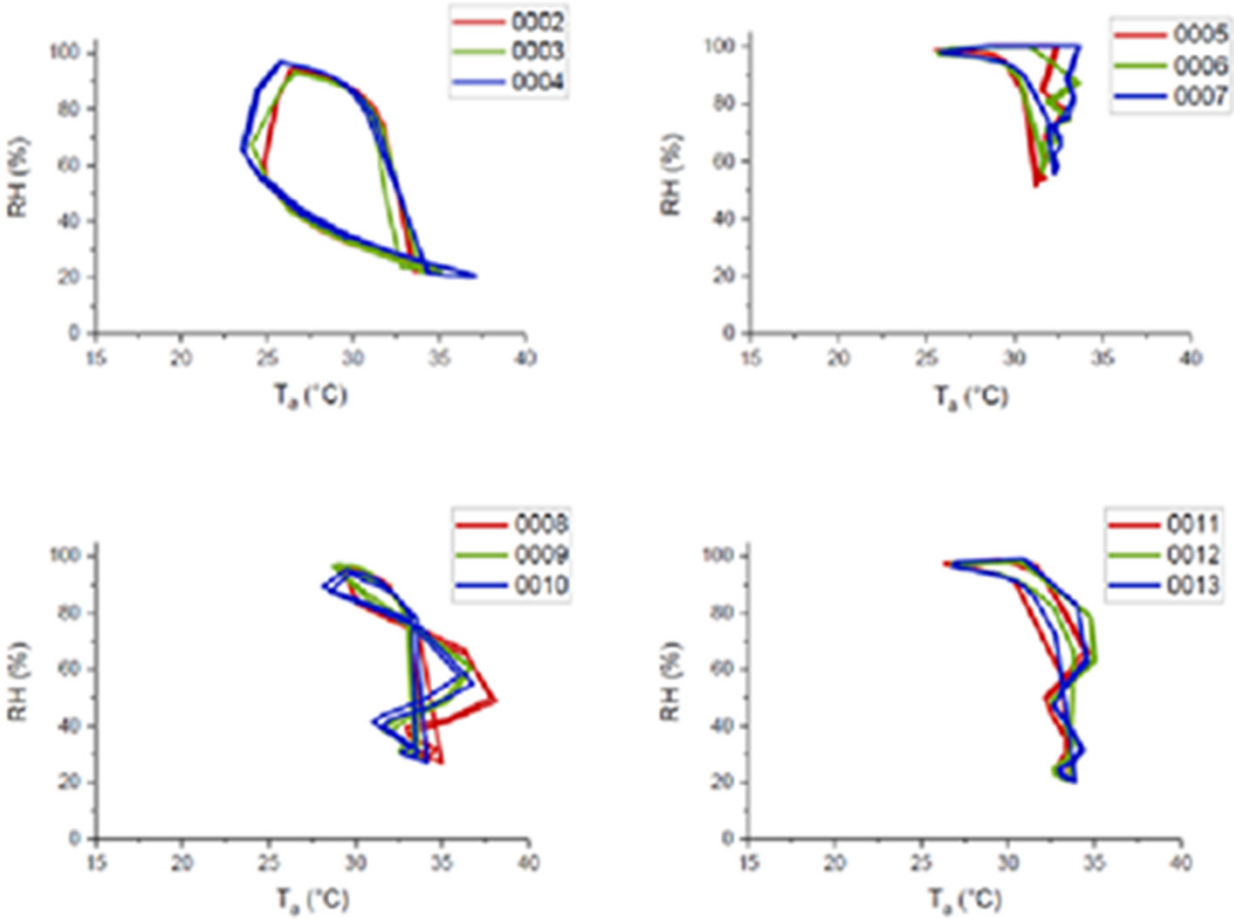




Conclusions

- RH is the single most relevant factor in atmospheric corrosion
- Strict control of wetting and drying cycles are important for simulating observed corrosion failure modes
- Atmospheric chemistry is another major factor governing corrosion rates and failure modes and must be balanced correctly with RH cycles to accurately simulate corrosion

What's Next?



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

Benefits to DoD

- Develop improved standard test protocol for accelerated corrosion testing
- More accurately predict corrosion in operational environments
- Reduce rejection of promising new environmentally friendly technologies
- Reduce use of carcinogenic materials

Acknowledgements

- Project team included government labs, universities and industry

For additional information, please visit

https://www.serdp-estcp.org/Program-Areas/Weapons-Systems-and-Platforms/Waste-Reduction-and-Treatment-in-DoD-Operations/WP-1673

Speaker Contact Information

james.dante@swri.org; 210-522-5458

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Q&A Session 1

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Development and Validation of a Cyclic Humidity Corrosion Test

Dr. Victor Rodriguez-Santiago
Naval Air Systems Command

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Agenda

- Benefits to DoD
- Background
- Dynamic monitoring
- Technical approach
- Conclusions

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What do we know?

- Problems with traditional accelerated corrosion tests (ACT)
  - Fail to replicate representative corrosion severity and morphology
    - Ex: ASTM B117, ASTM G85, MIL-STD-810, GMW 14872
  - Have chamber inconsistencies
    - Can be more pronounced during cyclic tests
  - Do not control relative humidity, a crucial factor in replicating damage

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What can we do?

- Understand individual and combined environmental factors and solution chemistry on the mechanisms and severity of corrosion
- Understand environment dynamic changes to quantify environmental severity

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Dynamic Monitoring

Critical to Understand Corrosion Evolution

Environmental exposure sites

- Closest correlation between corrosion degradation and damage experienced in-service
- Time-consuming
- Not widely accessible
- Provides cumulative data only
- *No dynamic monitoring*

Outdoor Exposure of Samples by W.H. Abbott at Battelle Columbus Operations[1]

[1] *W.H. Abbott, "A Decade of Corrosion Monitoring in the World's Military Operating Environments: A Summary of Results," DoD CorrDefense, 2008*

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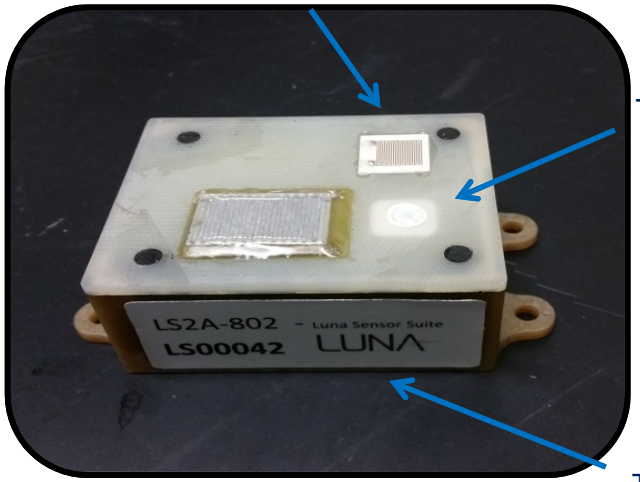
Dynamic Monitoring Devices

Environmental Conditions and Corrosion Severity

- Capture temperature, relative humidity, and *solution resistance*
- No information of the sample's surface condition is necessary
  - i.e. salt concentration, surface contaminants, etc.
- Environmental data and statistical analysis can be used to develop a better accelerated test

Gold interdigitated electrode



Air Temperature Relative Humidity

Surface Temperature

Multi-sensor device measuring temperature, relative humidity, and solution resistance across gold interdigitated electrode

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Environmental Severity Classification

- Traditional environmental severity classification methods fail to reliably classify corrosion severity
- Classification of the corrosion severity of atmospheres on the basis of three key factors (ISO 9223:20122)
  - TOW (time of wetness)
    - Temperature > 0 °C
    - Relative humidity > 80%
  - Deposition rate of chlorides
  - Sulfur dioxide concentration
- Five corrosivity classes: C1 to C5

[2] *ISO 9223, "Corrosion of Metals and Alloys – Corrosivity of Environments – Classification, Determination and Estimation" (Geneva, Switzerland: ISO, 2012)*

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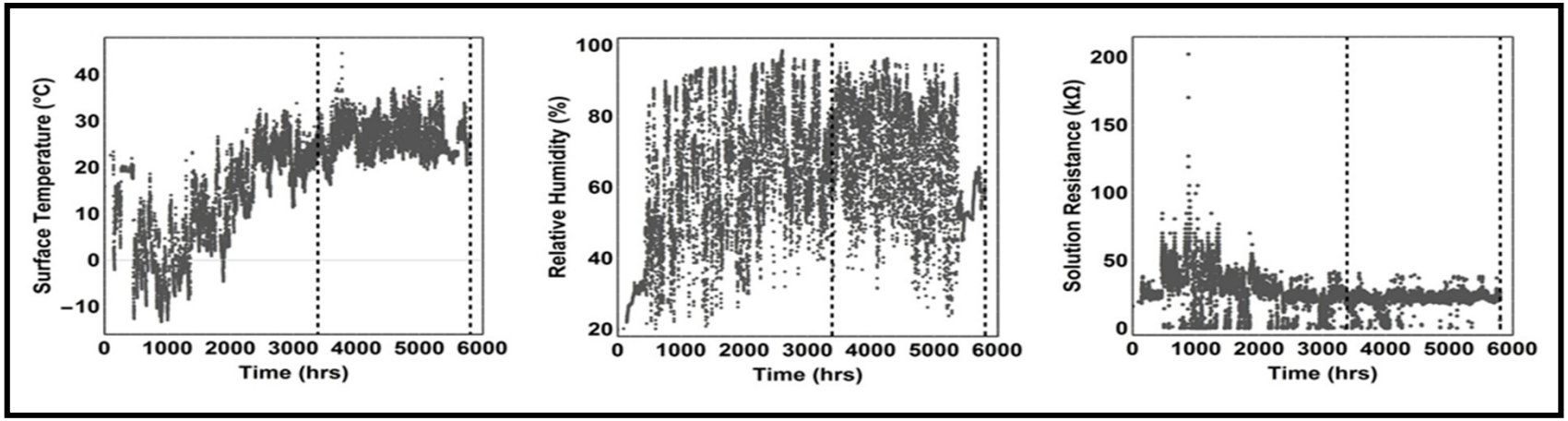
Dynamic Monitoring

Correlate Environment and Corrosion Severity



- Variables of interest are plotted as a function of time
  - Daily oscillation of surface temperature and relative humidity
  - Seasonal changes in average temperature
  - Solution resistance is measured across a broad range of values
  - Dashed lines represent data retrieval and therefore mass loss measurements

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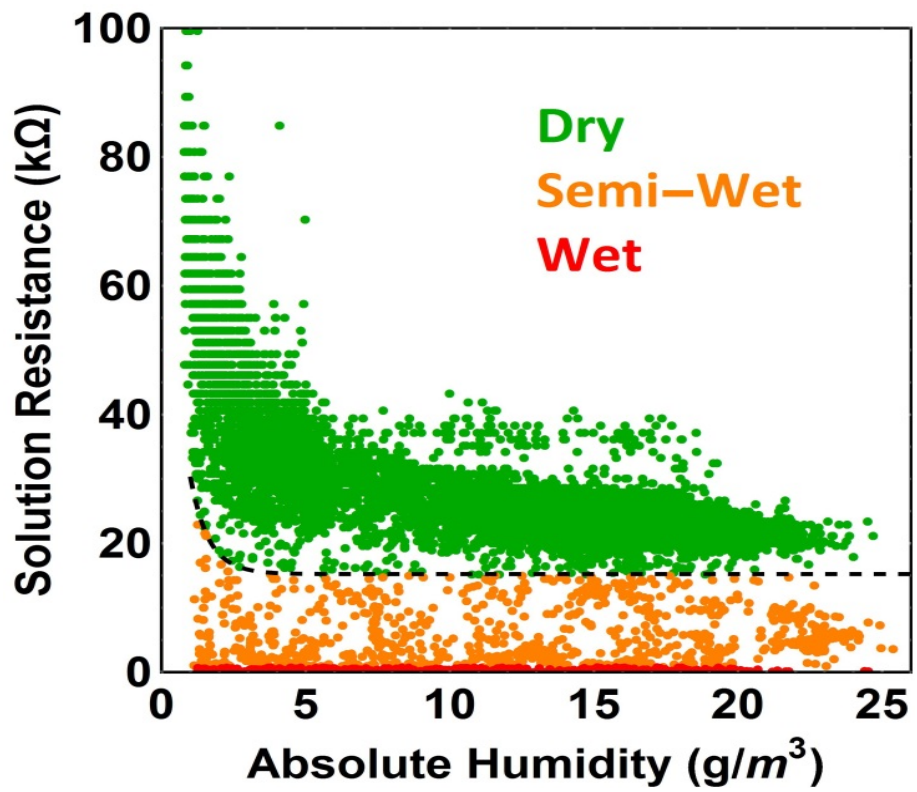
Dynamic Monitoring

Statistical Methodologies

- Data collected on-board USS Wasp over 9 months[3]
- Data < 819 Ω = "wet"
- Data between 819 Ω and "dry" = "semi-wet"
- From these data, number of wetness and semi-wetness events can be determined, as well as their average duration



[3] *Cosima Boswell-Koller, Victor Rodriguez-Santiago, Statistical Analysis of Environmental Parameters: Correlations between Time of Wetness and Corrosion Severity, CORROSION*

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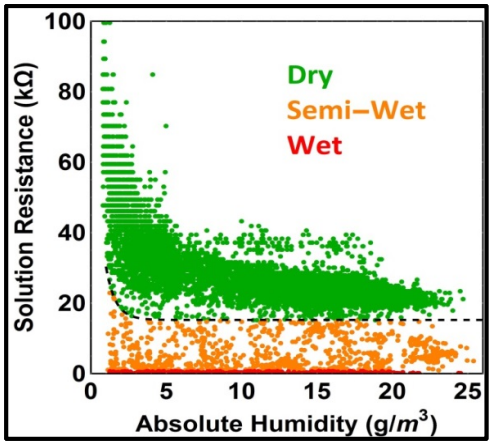
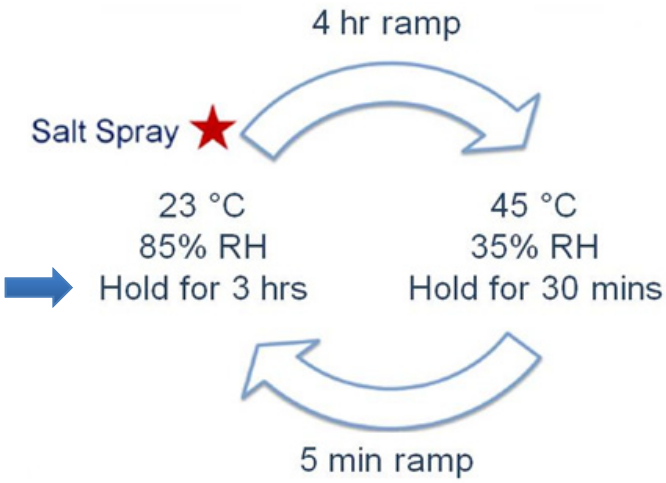
Dynamic Monitoring

Develop Effective Accelerated Corrosion Methods





	Actual (g)	Predicted (g)	Comparison to Environmental Exposure
Al 7075	0.0044 g	0.0038 g	4/9 mass loss in 1/30 time
1020 CS	0.1457 g	0.15 g	1/4 mass loss in 1/30 time

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Technical Approach

- Specific variables allow for tunable environmental corrosion severity
- Main parameters affecting corrosion

Parameter	Role
Relative Humidity (RH)	Defines periods when active electrolyte exists
RH duty cycle	Determines attack morphology
Electrolyte chemistry and deposition rate	Determines degree of attack

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Technical Approach

- Innovation
  - Control of cyclic variation of relative humidity and the periodic salt deposition of salt solution within each cycle
- Traditional cyclic tests
  - Do not include this degree of control
  - Introduces a high degree of variation in results
  - Limited usefulness
- Recent gains in understanding role of humidity cycling control only within the last 5 years

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Task 1: Optimization of Cycle Period

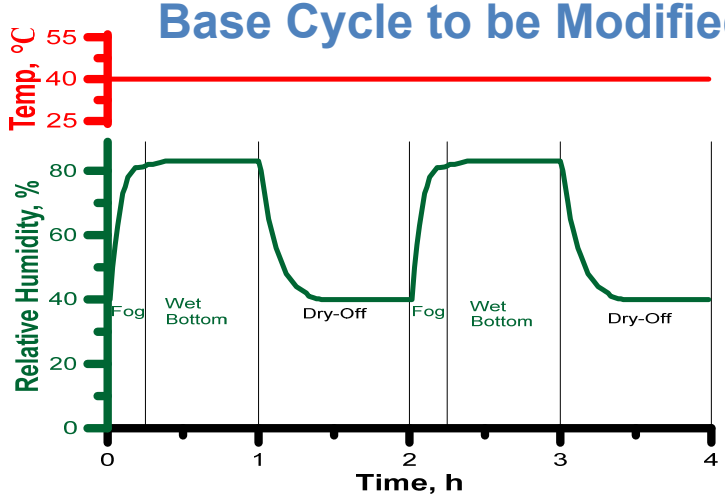
- To be tested
  - 2 solution chemistries
  - 3 RH cycles

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
$NaNO_3$	0.00	3.27
HCl (1N)	(1 ml)	(1 ml)

*Normalized to ionic strength

Base Cycle to be Modified



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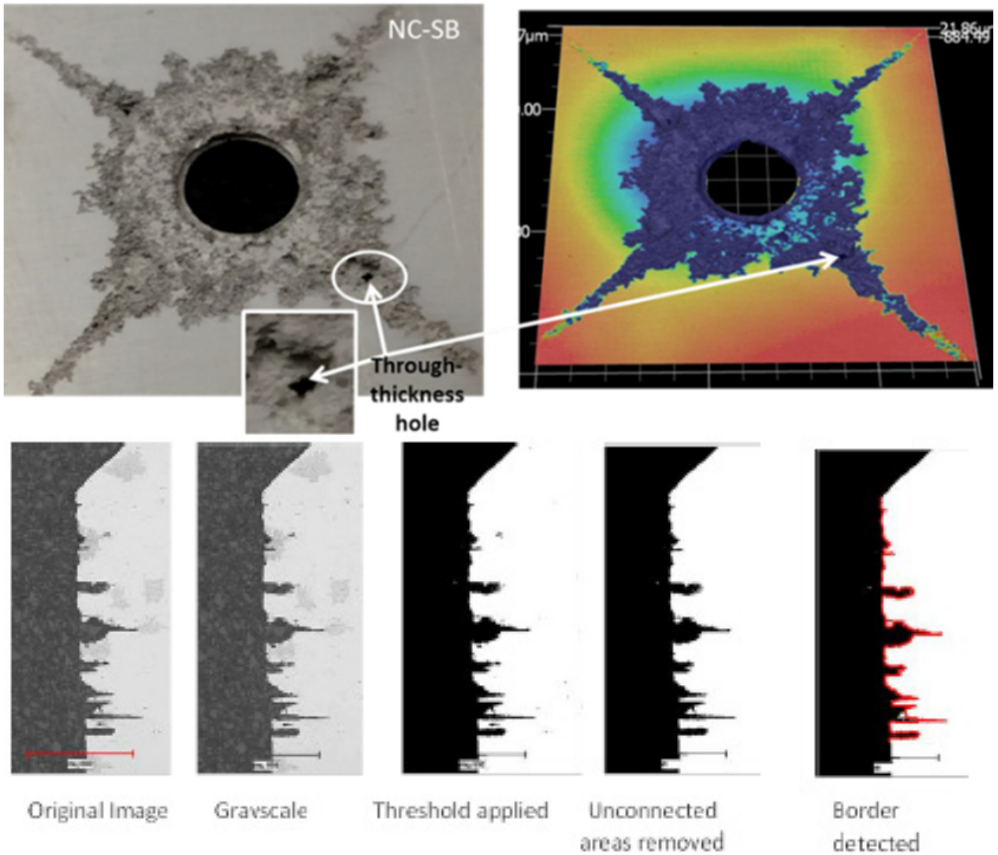
Task 1: Optimization of Cycle Period

- Generate 3D optical images
- Rank coating performance and environmental severity by image analysis



Go/No-Go decision will be made at the end of this task

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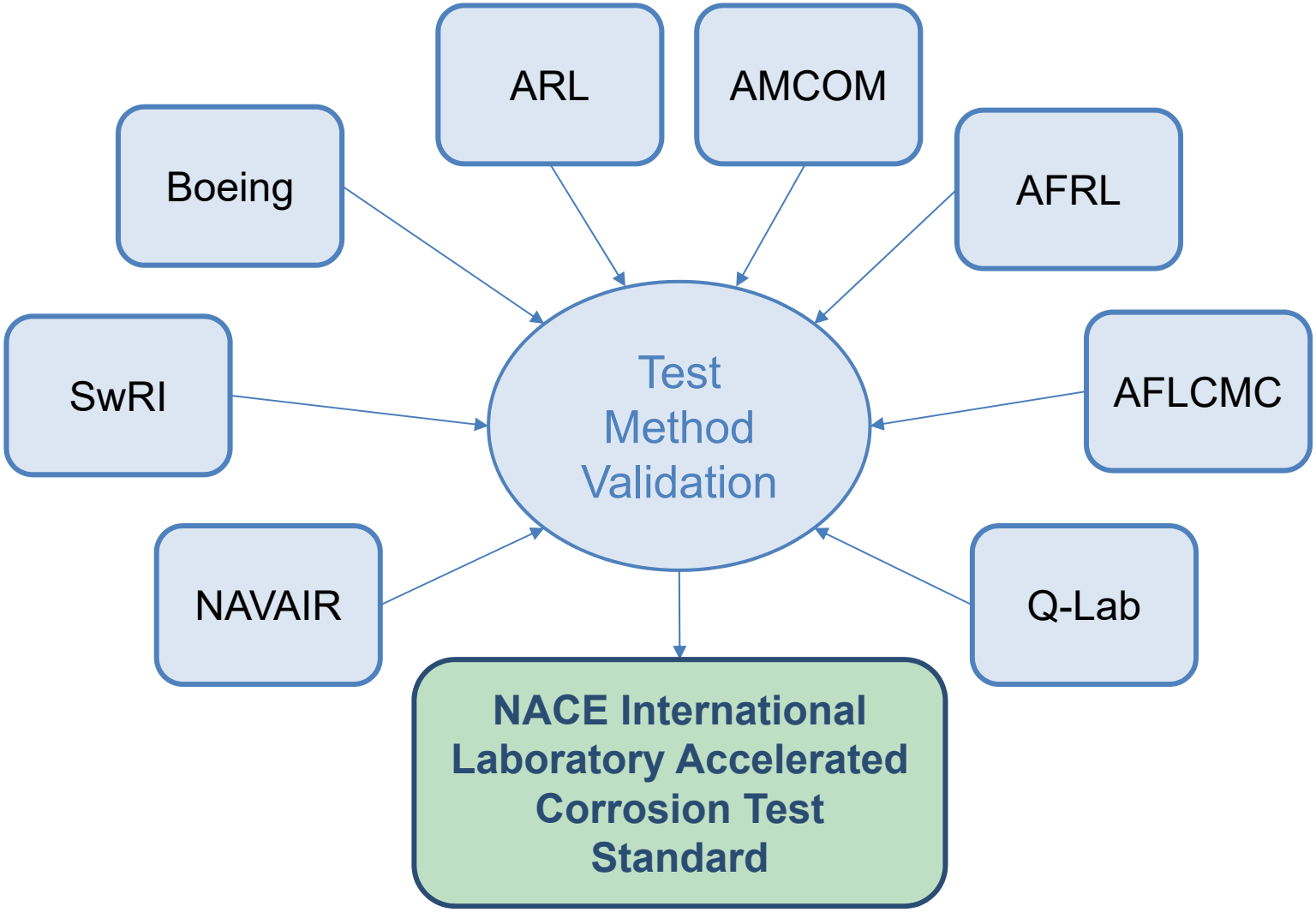
Task 2: Round Robin Testing



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Task 3: Standard Development and Acceptance

- Objective: Formalize the test procedure
  - NACE International laboratory standard
- Advantages of a society standard over a MIL-STD
  - OEMs can participate in development and drafting of the standard
    - Reduces resistance to acceptance
  - Society standards require revisions every 3-5 years
    - Standard can be updated as technical developments are made
  - Easier to convert a pre-vetted standard if DoD wants a MIL-STD version

Notes: NACE – National Association of Corrosion Engineers; MIL-STD – US Military Standard; OEM – original equipment manufacturers

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Task 3: Standard Development and Acceptance

- Create a NACE Task Group (TG) within STG 41 (Electric Utility Generation, Transmission, and Distribution) to develop the specification
- Front end of specification will define overall testing approach
- Specific environmental test cycles included in annex section of the specification
- Additional annexes can be created to include other types of environments

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Conclusions

- Dynamic environmental monitoring devices and data analysis methodologies used to correlate environmental conditions and corrosion severity
- Dynamic environmental monitoring used to develop effective accelerated corrosion test methodologies which replicate corrosion severity of outdoor field exposures
- Combination of effects observed from varying solution chemistries, time of wetness, and drying:wetting time ratios may allow for full tunable corrosion severity

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Conclusions (Cont'd)

- Corrosion severity and mechanisms will be quantified using 3-D image analysis.
- Develop a more representative and repetitive accelerated corrosion test standard through inter-laboratory collaboration

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Benefits to DoD

- Identify corrosion prevention technologies
- Understand risk
- Reduce testing costs
- Introduce better technologies faster

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References

- W.H. Abbott, “A Decade of Corrosion Monitoring in the World’s Military Operating Environments: A Summary of Results,” DoD CorrDefense, 2008
- ISO 9223, “Corrosion of Metals and Alloys – Corrosivity of Environments – Classification, Determination and Estimation” (Geneva, Switzerland: ISO, 2012)
- Cosima Boswell-Koller, Victor Rodriguez-Santiago, Statistical Analysis of Environmental Parameters: Correlations between Time of Wetness and Corrosion Severity, CORROSION
- James Dante, Accelerated Dynamic Corrosion Test Method Development, SERDP Project WP-1673

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For additional information, please visit

https://www.serdp-estcp.org/Program-Areas/Weapons-Systems-and-Platforms/WP18-5087

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Q&A Session 2

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The next webinar is on May 9, 2019

Roles of Soil Microbial Communities in Ecosystem Restoration

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Survey Reminder

Please take a moment to complete the survey that will pop up on your screen when the webinar ends

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