Solvent Replacement for Super Corr-A Corrosion Preventive Compound (CPC)

2011 Air Force Corrosion Conference

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Overview

- Project Team
- Background & Objectives
- Technical Approach
- Test Matrix
- Laboratory and Field Testing – Results
- Conclusions
- Recommendations

Project Team

- Primary Stakeholder – F-16 SPO, 388th Fighter Wing
- COTR – Paul Hoth 501 ACSS/GFLB
- Program Manager – John Stropki
- Task Leader – Jim Tankersley
- Support Staff
  - Bill Abbott (Consultant)
  - Annie Lane (Research Scientist)
  - Jill Gregory (Researcher)
- Subcontractor Support
  - Lektro-Tech, Inc., Tampa, FL (Ron Knight and Robert Kay)
    - Assistance w/ solvent down-selection and formulation
  - SMI, Inc., Miami, FL
    - Perform first article testing on new formulations

Background

- The Super Corr-A corrosion preventative compound (CPC) is qualified as a MIL-L-87177A, Type I, Grade B material for electrical connector applications
  - The Super Corr-A lubricant has had two solvent-related formulation modifications since 1994 (CFC-113 and HCFC-141B)
  - Super Corr-A has met or exceeded performance requirements in extensive evaluations by Hill AFB
- The current Super Corr-A formulation contains an HCFC AK225T solvent
  - Considered Class II Ozone Depleting Substances (ODS)
  - Banned in the European Union (EU) and Canada on 1 January 2009
- All maintenance and manufacturing operations in the EU requiring use of MIL-L-87177A are currently shutdown with no alternative replacement identified
- Unless a replacement solvent can be implemented, use of these ODSs will also be prohibited in the United States beginning in 2015

Objective & Approach

Objective:

Identify a more environmentally friendly and COTS alternative to the HCFC AK225T solvent currently in the Super Corr-A lubricant.

Program Approach:

- Research US and EU compliant solvents with chemistry compatible with Super Corr-A CPC
- Define material and performance requirements based on previous assessments of lubricants
- Conduct laboratory and field testing for comparative evaluation of the lubricant performance containing the alternative solvents
- As required, update MIL-L-87177A specification and associated process order

Test Matrix

- Test plan includes nine CPC formulations and one control
  1. Existing Super Corr-A formulation with AK225T solvent
  2. Previous Super Corr-A formulation with 141B solvent
  3-6. Super Corr-A formulated with 4 solvent candidates
     a. DuPont Vertrel® SDG w/ current concentration of CPCs
     b. DuPont Vertrel® SDG w/ higher concentration of CPCs
     c. Kyzen Cybersolv® 141R w/ higher concentration of CPCs
     d. Kyzen Cybersolv® 141R w/ current concentration of CPCs
  7. ILFC 1006 CON-TAC
  8. Zip-Chem D-5026NS
  9. Zip-Chem D-5026NS with alternative propellant (Noxit-86)

MIL-L-87177A Assessments

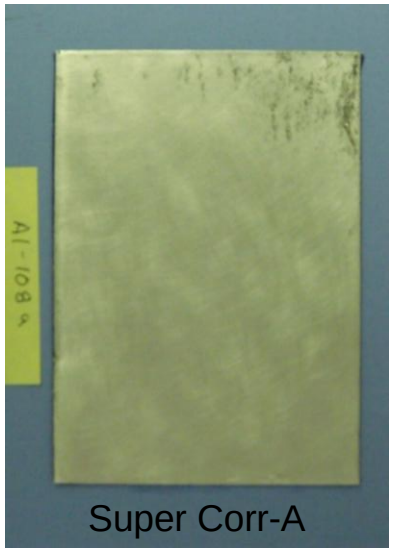
- SMI Laboratories conducted first article testing specified in MIL-SPEC to validate performance characteristic requirements of experimental lubricant formulations
- **Results:** New and old formulations of Super Corr-A do not meet first article requirements of MIL-L-87177A
  - Original formulations were never tested
  - Both formulations perform appropriately for intended application
- **Recommendation:** Update first article requirements and revise MIL-SPEC
  - Stakeholders include; Hill AFB, DLA-Richmond, AFRL/CTIO, and AFCPCO

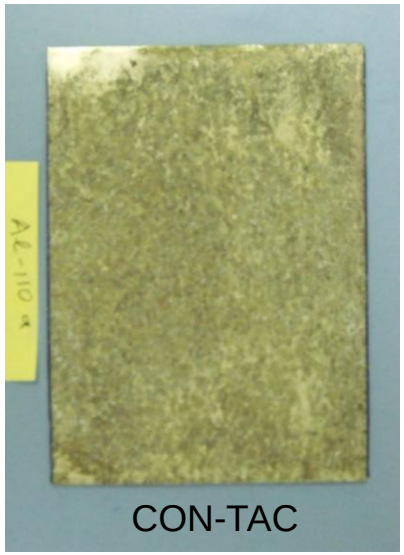
First Article Testing Results

Requirement	Test Method Specification	Limit	Result
Dryness	MIL-SPEC 4.6.1	0.0100 gram (max)	Failed
Flash Point	ASTM D1310	243°C/470°F (min)	Inconclusive
Dielectric Breakdown	ASTM D877	24,000 volts (min)	Failed
Lubricity	ASTM D226	1.20 mm wear scar diameter (max)	Failed
Residue Solubility	MIL-SPEC 4.6.3	No visible residue	Failed
Leakage	MIL-SPEC 4.6.4	No leakage or distortion	Passed
Content	MIL-SPEC 4.6.5	16 ounces (min)	Failed (container content 12 oz.)
Performance of pressurized containers	MIL-SPEC 4.6.6	Uniform spray, panel adherence, no sagging	Passed
Oxidation Stability	ASTM D942	<5 pounds/100 hours	Failed
Grade B Corrosion	ASTM B117	No corrosion after 168 hours	Passed
Sprayability	MIL-SPEC 4.6.9	Sprayable w/ no clogs	Passed

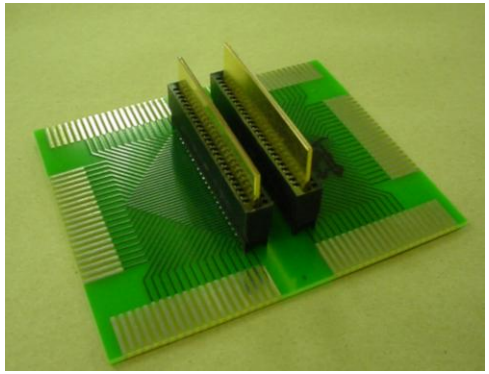
Battelle Laboratory Results

- Grade B Corrosion Testing
  - Alternative Super Corr-A formulations showed improved corrosion resistance in salt fog exposure testing
  - Most extensive pitting damage noted with the control and CON-TAC
  - "Streaked" pitting noted on Noxit-86, D5026NS; may have been caused by formation and collection of water droplets along top edge





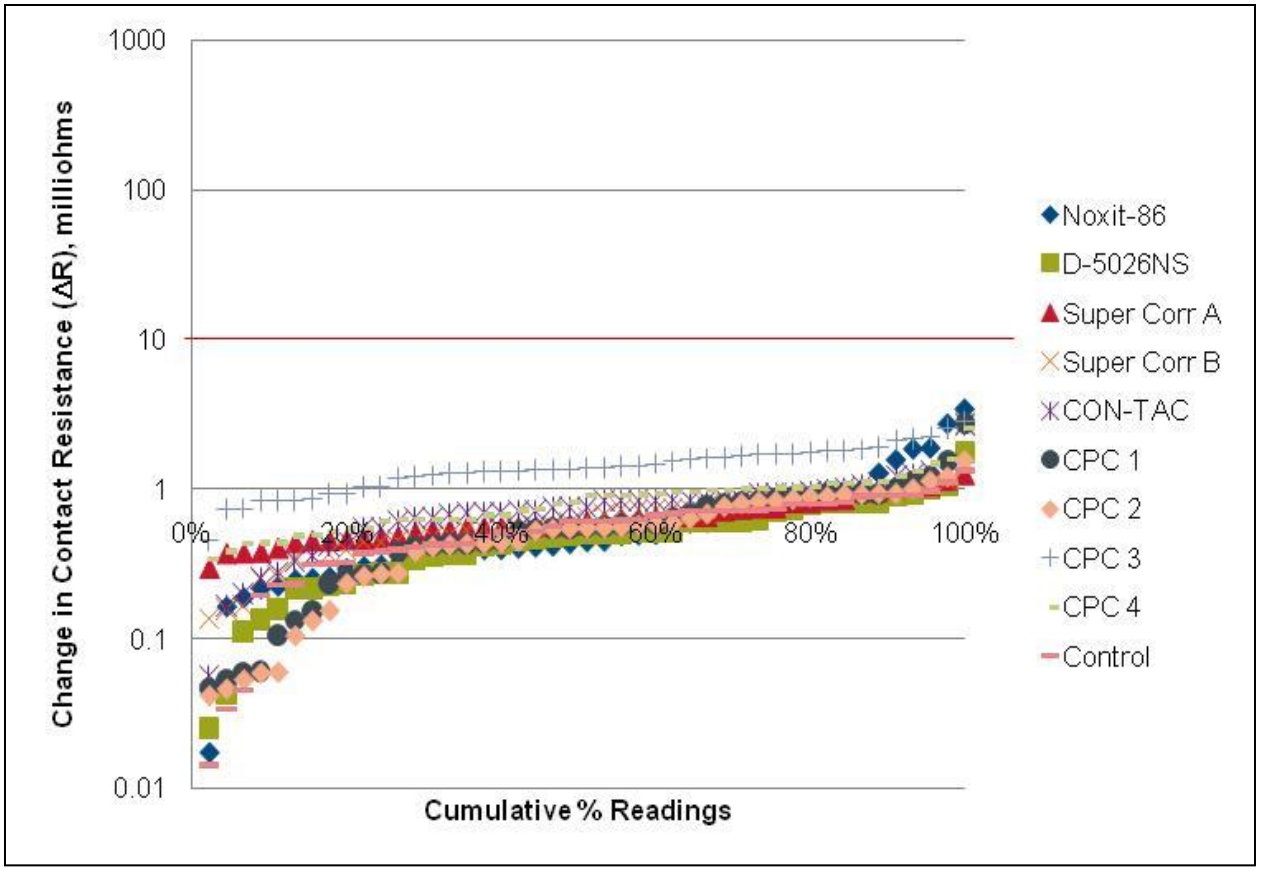
Battelle Laboratory Results - Connector Card Testing



Conditions:
- 1000 hours
- 80° C (176° F)

Requirements:
- ΔR < 10 milliohms

Results:
- All passed



Change in Contact Resistance Resulting from Thermal Aging Exposure Testing of Coated Electrical Connectors

Battelle Laboratory Results – Low Temperature Testing

Conditions:

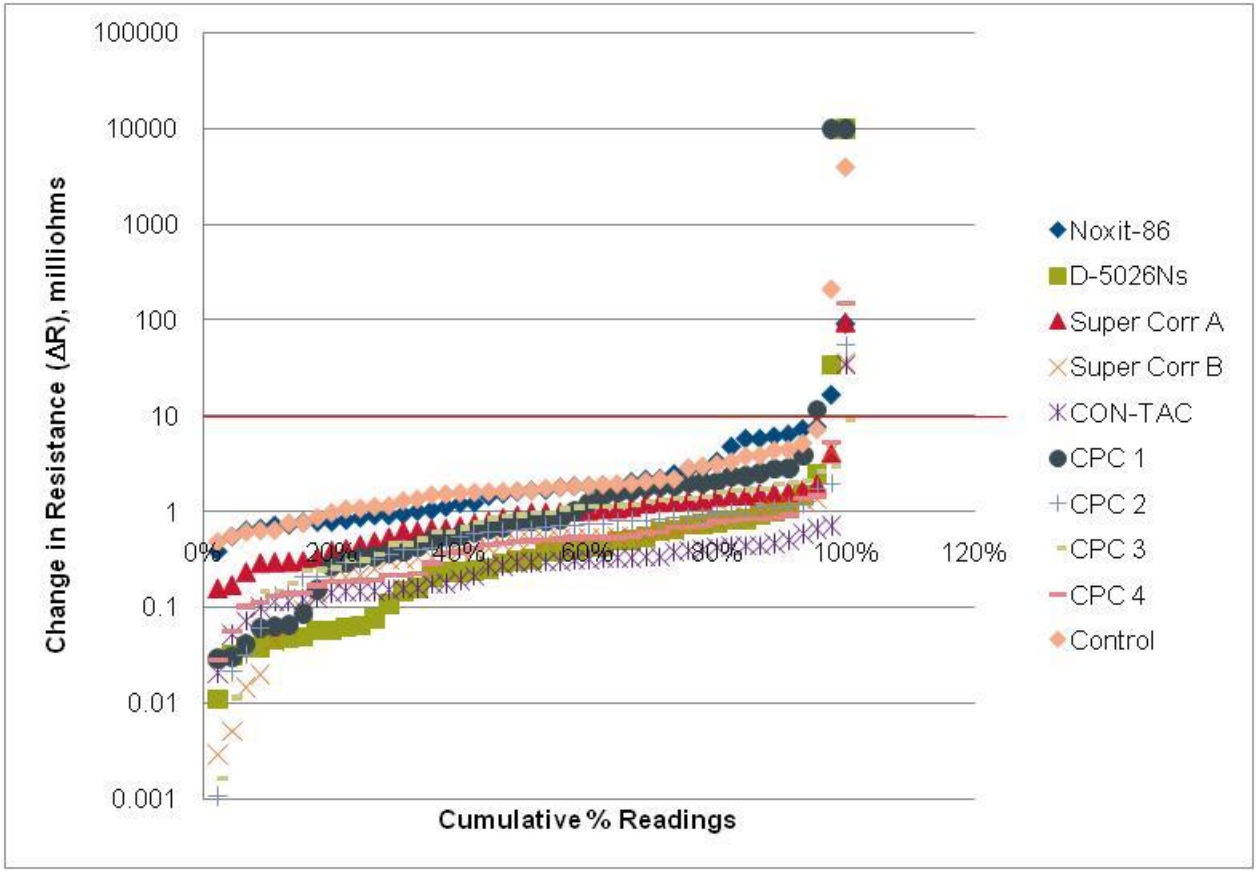
- Cycling at 25º , 5º , -15º , -35º , -55º , -15º , 5º , 25º
- 15 minutes @ each temperature

Requirements:

- ΔR < 10 milliohms

Results:

- Only CPC No. 1 failed



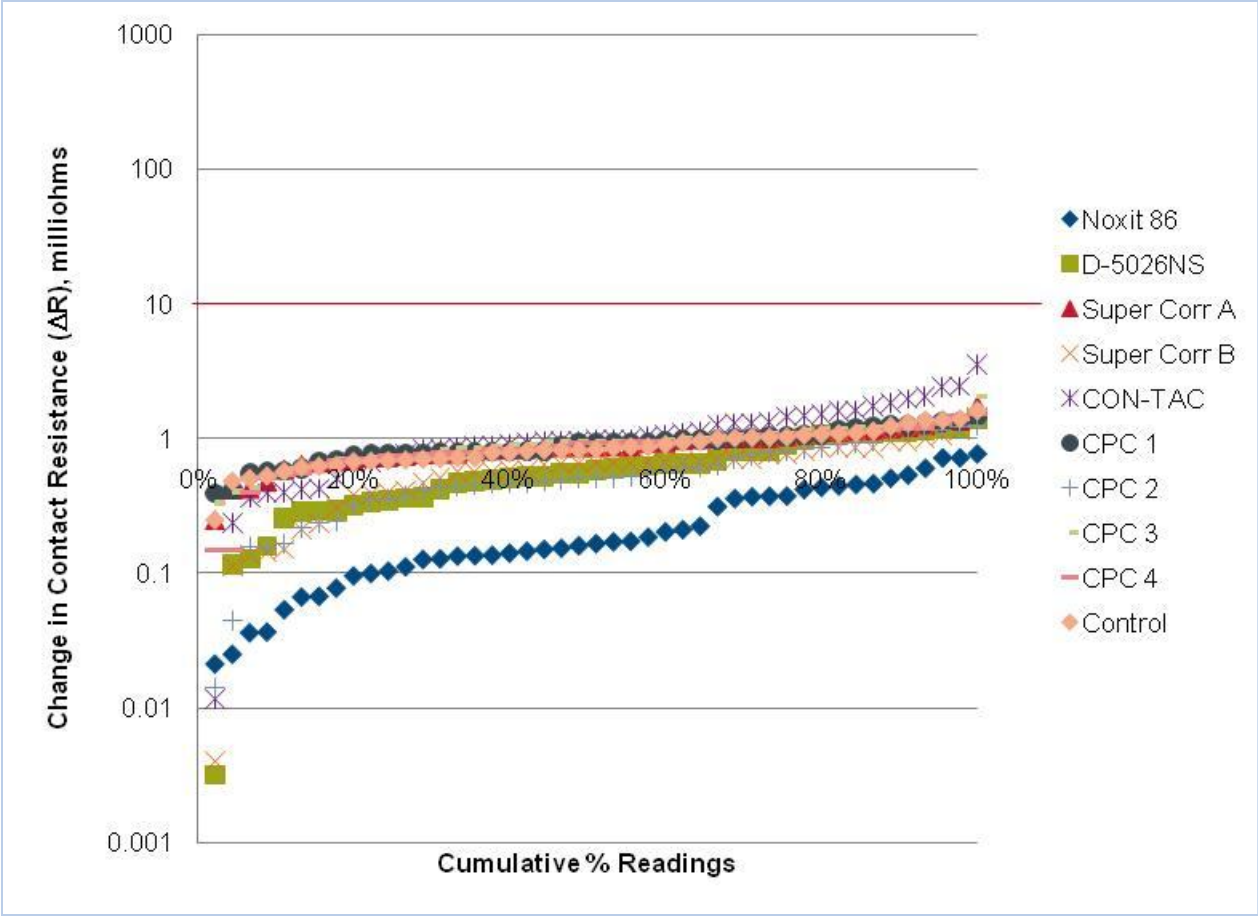
Change in Contact Resistance Resulting from Low Temperature Cycling of CPC Coated Electrical Connectors

Battelle Laboratory Results – Disturbance Cycle Testing

Conditions:
- 100 demate/remate cycles

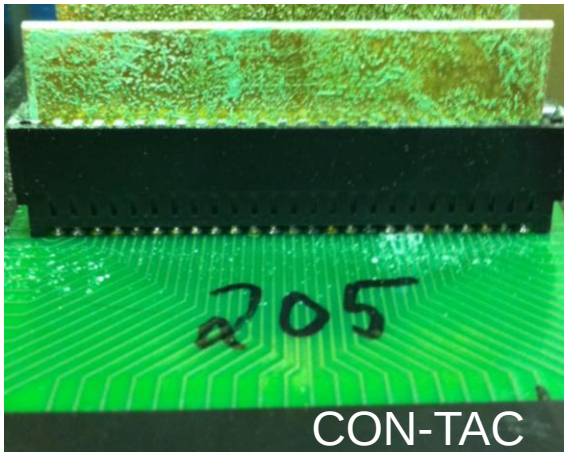
Requirements:
- ΔR < 10 milliohms

Results:
- All passed



Change in Contact Resistance Resulting from 100 Disturbance Cycles Completed on Coated Coupons attached to Connector Card

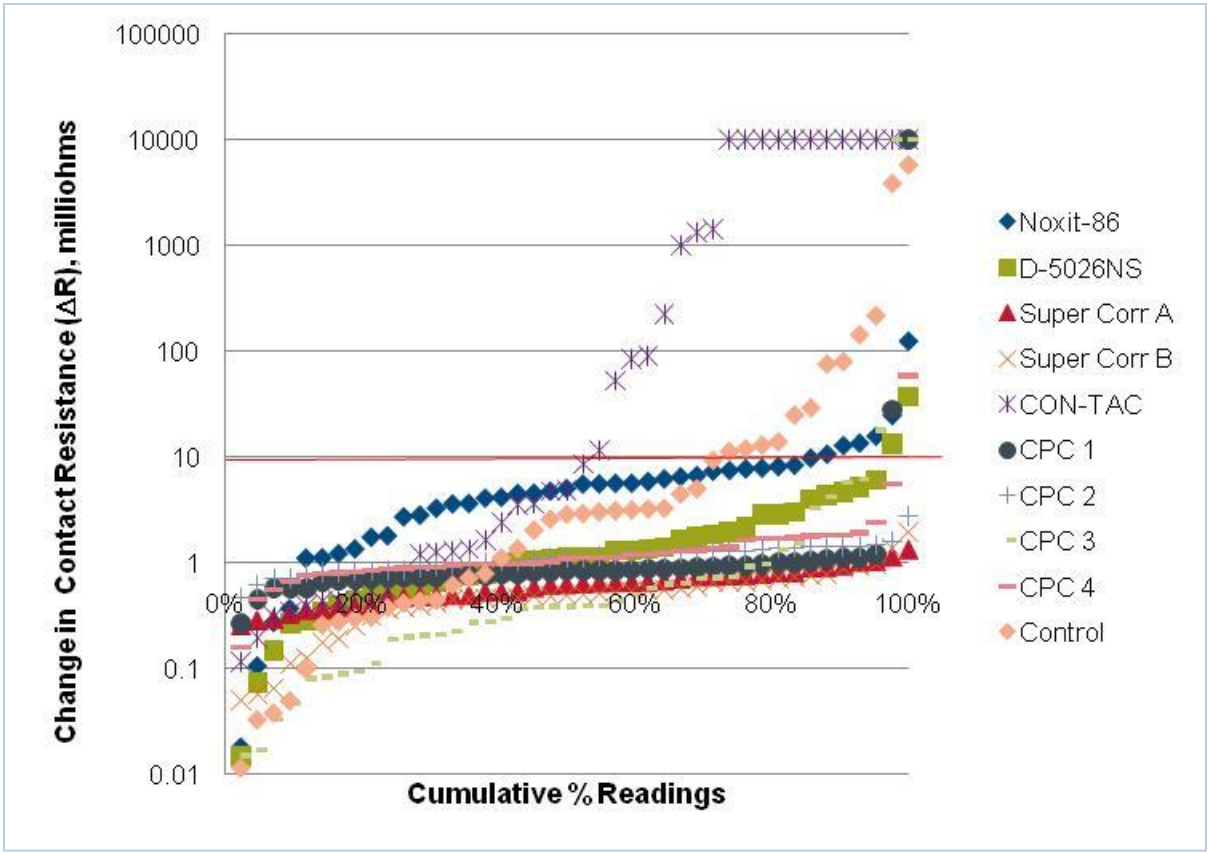
Battelle Laboratory Results – Class II Flowing Mixed Gas Testing



Conditions:
- 10 day exposure

Requirements:
- ΔR < 10 milliohms

Results:
- CPCs No. 1 & 3, CON-TAC, and Noxit-86 failed



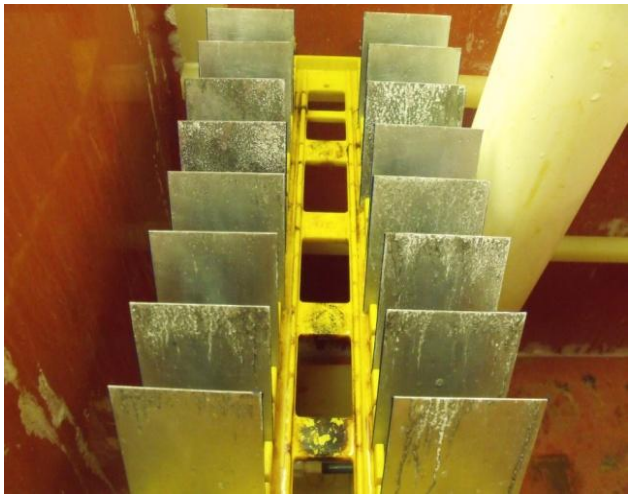
Change in Contact Resistance After Exposure of Coated Coupons to Class II Flowing Mixed Gas Test

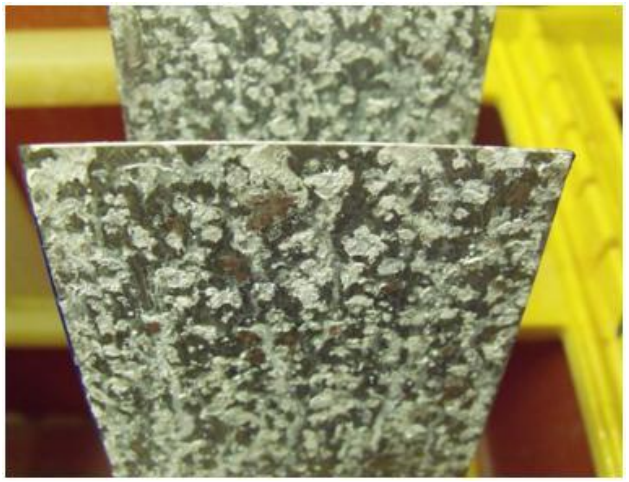
Battelle Laboratory Results – Grade B Corrosion Testing

CPC	Panel 1	Panel 2	Panel 3	Average Score (Max: 5)
Control	5	5	5	**5.0**
CPC No. 1	2	2	2	2.0
CPC No. 2	1	2	1	1.3
CPC No. 3	1	1	1	**1.0**
CPC No. 4	1	1	2	1.3
Super Corr A	3	2	1	2.0
Super Corr B	1	1	2	1.3
CON-TAC	5	5	4	4.7
Noxit-86	3	2	3	2.7
D-5026NS	3	2	3	2.7

Salt Fog CPC Ratings Calculated from Pit Density Evaluation Referenced in ASTM G46-94 and ASTM D610-08

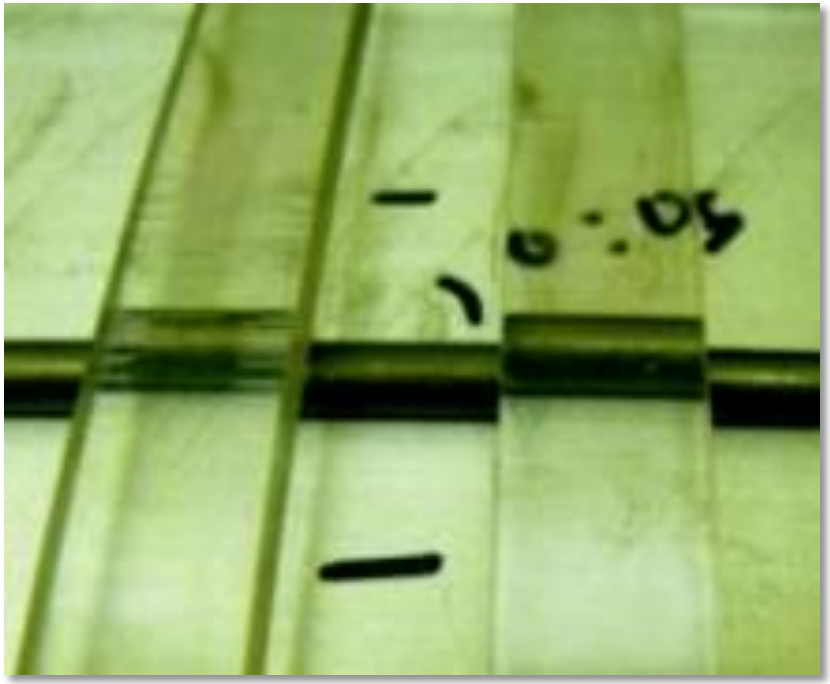
Photographs Documenting Placement of Coated Panels in ASTM B117 Salt Fog Cabinet and Corrosion Pitting Noted on Coupons Coated with CON-TAC CPC





Battelle Laboratory Results – Polycarbonate Compatibility (canopies)

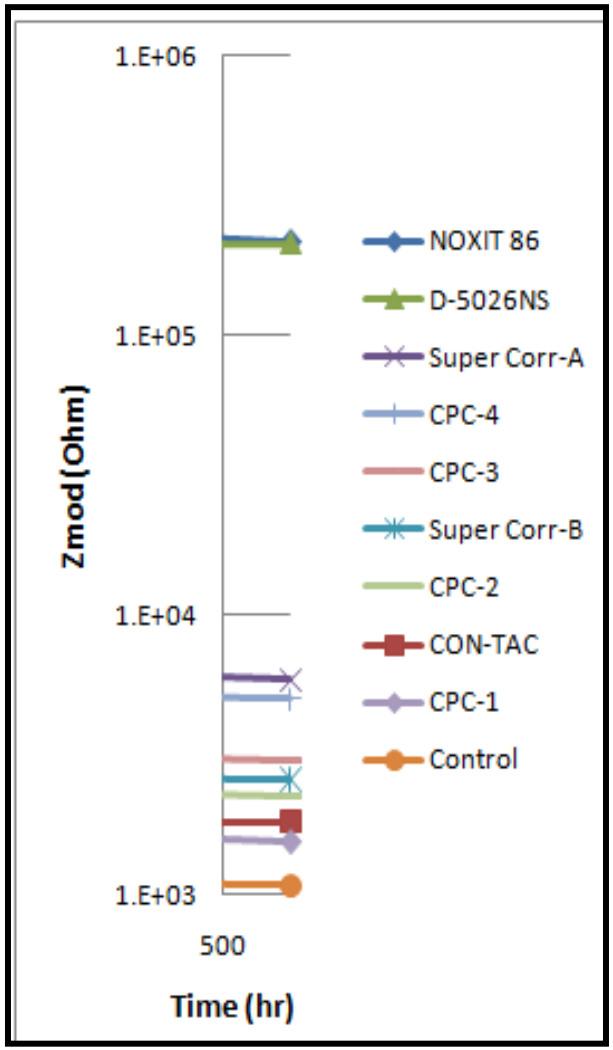
Consistent with previous testing, crazing noted with CON-TAC, AK225T (slight), 141-B (dramatic)



Polycarbonate Stressed Coupons:
CON-TAC (left), Control (right)

UC Laboratory Testing Results

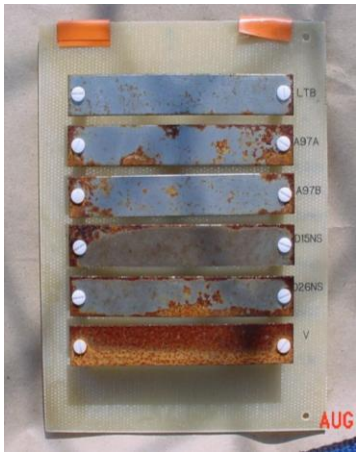
Ranking of EIS Data	
Noxit86	1
D-5026NS	2
Super Corr-A	3
CPC-4	4
CPC-3	5
Super Corr-B	6
CPC-2	7
CON-TAC	8
CPC-1	9
Control (uncoated)	10



Battelle Field Testing

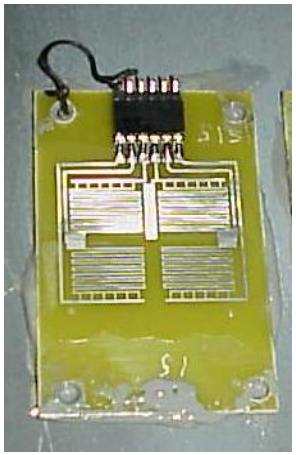
Test Type	Tests	Test Reference	Sample Size	Time Periods	Replicates	Sample Material
Field Exposure Testing	Connector Field Testing	Abbott 1996 report	10 CPCs	3 (1 mo, 3 mo, 6 mo)	50 (pin count)	Test connectors with gold-plated bars (2 to a PC board)
	Corrosion Coupons	Abbott 1996 report	10 CPCs	3 (1 mo, 3 mo, 6 mo)	1	Rack with 5 steel coupons
	Lap Splice Testing	Rice 2004 report	10 CPCs	3 (1 mo, 3 mo, 6 mo)	1	Lap splice fixture with steel coupon fastened to 2024 T3 Al coupon
	Steel Sensors	Recent Abbott work	10 CPCs	Measurements in place at 1 mo, 3 mo, 6 mo	1	Steel sensors

Corrosion Coupons



Lap Splice Fixtures

Steel Sensor



BUSINESS SENSITIVE

Battelle Field Testing Results - Corrosion Testing Summary

CPC	Average Weight Loss (g)	Average Corrosion Rate (mpy)
D-5026NS	0.14590	2.22
CPC 2	0.21215	3.23
CPC 4	0.21465	3.27
Noxit 86	0.23494	3.58
CPC 1	0.32854	5.01
CPC 3	0.33280	5.07
Super Corr-A	0.33346	5.08
Super Corr-B	0.35096	5.35
CON-TAC	0.43267	6.59
Control	0.51872	7.91

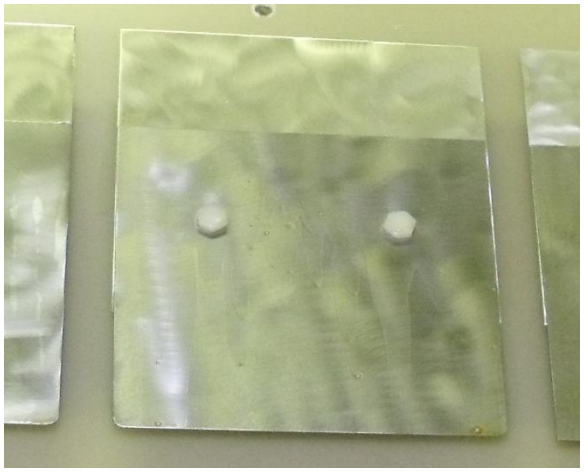
***Average for each CPC over the 4 month period with the three location sets combined**

CPC Lubricant Ranking of Coated Corrosion Coupons Based on Weight Loss

Battelle Field Testing Results - Summary

- The worst corrosion resistance was measured for the control or uncoated coupon sets,
- The best corrosion resistance was measured for the coupon sets coated with the D-5026N lubricant,
- The corrosion resistance of the CPC-2 lubricant was only slightly lower than the performance measured for the D-5026N material,
- The corrosion related performance of the coupons coated with the Noxit-86, CPC-3, CPC-4, Super Corr-A and Super Corr-B was identical.

Battelle Field Testing Results – Lap Splice Testing



Area of CPC Application Along Upper Edge of Lap Splice Coupons

Lap Splice Coupon Sets Mounted on Chain Link Fence at FMRF

Battelle Field Testing Results – Lap Splice Testing Summary

	West Jefferson			FMRF			
CPC	**1 mo.**	**3 mo.**	**4 mo.**	**1 mo.**	**3 mo.**	**4 mo.**	**Total (Max: 60)**
Control	1	0	1	0	0	0	**2**
CPC No. 1	3	2	2	1	0	1	9
CPC No. 2	3	3	1	2	1	0	10
CPC No. 3	2	5	9	2	2	0	20
CPC No. 4	3	3	3	3	2	0	14
Super Corr A	3	2	1	1	0	0	7
Super Corr B	3	0	2	2	0	0	7
CON-TAC	3	0	2	1	0	0	6
Noxit-86	10	5	10	10	3	3	41
D-5026NS	10	9	8	9	4	4	**44**

Ranking Scores for CPC Coated Lap Splice Coupons (ref. ASTM D610-08)

Battelle Field Testing Results – Steel Sensors at FMRF and West Jefferson

- Horizontally mounted sensors had increased corrosion
- Visual corrosion on controls, CON-TAC, and D5026NS variants
- CPC No. 2 consistently showed the least change in resistance

Horizontal

Vertical

Conclusions

- No tested lubricants met all first article testing requirements
- DuPont Vertrel SDG and Kyzen Cybersolv C141R performed well
- Independent testing conducted by SMI Laboratories confirm solvent alternatives are not corrosive or embrittling to high strength aerospace alloys
- Performance of formulations blended with compliant solvents and higher concentrations of proprietary CPC was equal to, or greater than lubricants approved per MIL-L-87177A and MIL-PRF-81309F
- Demonstrated superior performance of the D-5026NS, CPC No. 3 and CPC No. 4 lubricants
- Compliant solvent alternatives can replace the 225T solvent in the current Super Corr-A formulation without compromising the performance of the lubricant

Recommendations

- Work closely with representatives at Hill AFB, DLA, AFRL, and AFCPCO to revise or update the chemical, physical and performance requirements currently referenced in the MIL-L-87177A specification
- A preliminary set of deletions, modifications or additions include:
  - Update flash point requirement based on lubricant chemistry
  - Update or delete the dielectric breakdown requirement based on lubricant chemistry and intended use applications
  - Assess and update oxidation stability requirements
  - Input compatibility requirement with MIL-PRF-32033 and MIL-PRF-81309F lubricants
  - Input Electronics Lubricant Effectiveness tests referenced in MIL-PRF-81309F
    - Initial contact resistance (fixed and disturbed)
    - Low temperature exposures
    - Thermal aging
    - Durability cycling
    - Corrosive gas exposures
    - Compatibility with electrical insulating compounds

Questions & Discussion

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Back-up Slides

UC Laboratory Testing

- Testing by University of Cincinnati (UC) supplemented Battelle's CPC performance testing

Test Type	Tests	Test Reference	Sample Size	Time Periods	Replicates	Sample Material
UC Laboratory Testing	Grade B Corrosion	MIL-L-877177A ASTM B117 - 168 hrs salt fog exposure	9 CPCs	1 (168 hrs)	3	2024 T3 Al coupons
	DC Polarization Resistance	ASTM G59, ASTM G96, ASTM G102	9 CPCs	1 (record resistance for each sample)	2	2024 T3 Al coupons
	Electrochemical Impedance Spectroscopy	Battelle April 2005 study	9 CPCs	7 (at 8, 24, 48, 96, 168, 336, and 504 hrs)	2	2024 T3 Al coupons

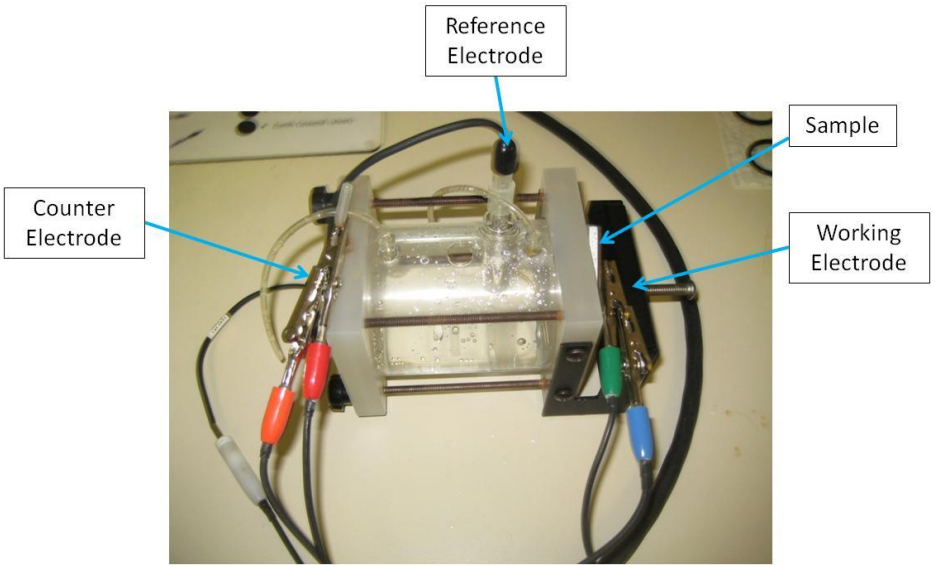
UC Laboratory Testing Results

- ASTM B117 Neutral Salt Spray Corrosion Testing Results
  - 168 hour exposure period
  - Extensive corrosion pitting observed on control coupons
  - Good corrosion resistance for all CPCs tested
  - Visual scoring ranked CPCs:
    - CON-TAC (best)
    - D-5026NS, CPCs No. 2, 3, 4
    - Noxit-86, SC-A, SC-B, and CPC No. 1 (worst)

UC samples in salt fog chamber at ECOSIL

UC Laboratory Testing Results

- Polarization Resistance
  - Electrochemical technique that assesses corrosion rates using direct applied current



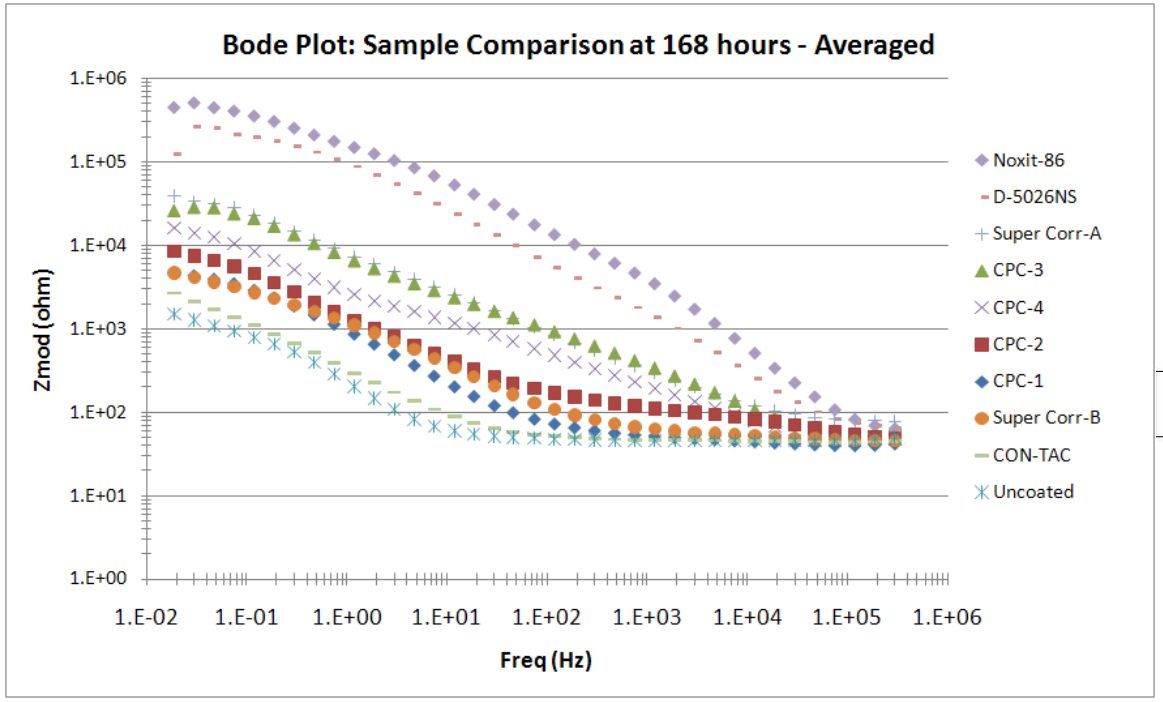
Polarization Resistance (R_p) Results

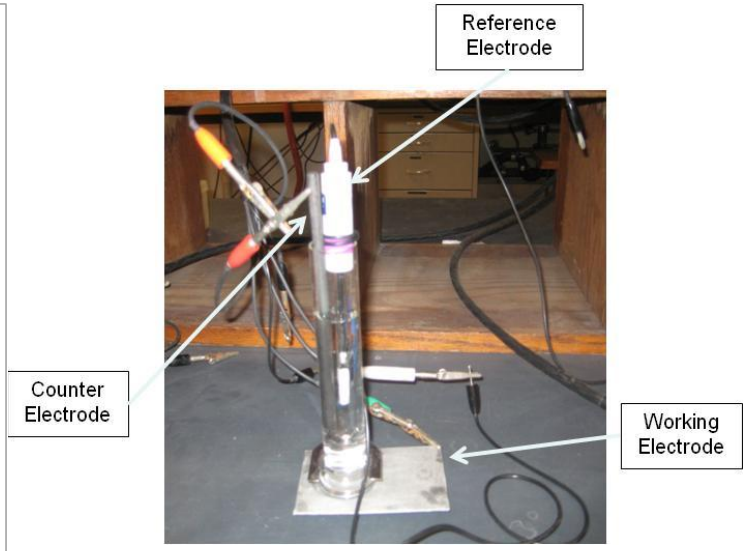
Sample	OCP (mV)	Polarization Resistance, R_p (kOhm)	Ranked by Highest Corrosion Resistance
CPC-4	-476.3	22530	1
CPC-3	-404.7	4487	2
Super Corr-A	-414.8	2465	3
D-5026NS	-381	782.2	4
Super Corr-B	-394.9	388.8	5
CPC-1	-385.8	337.5	6
NOXIT86	-385.9	295.3	7
CON-TAC	-374.5	145.7	8
Control - uncoated	-383.2	7.7	9
CPC-2	N/A	N/A	N/A

1 = highest corrosion resistance

UC Laboratory Testing Results

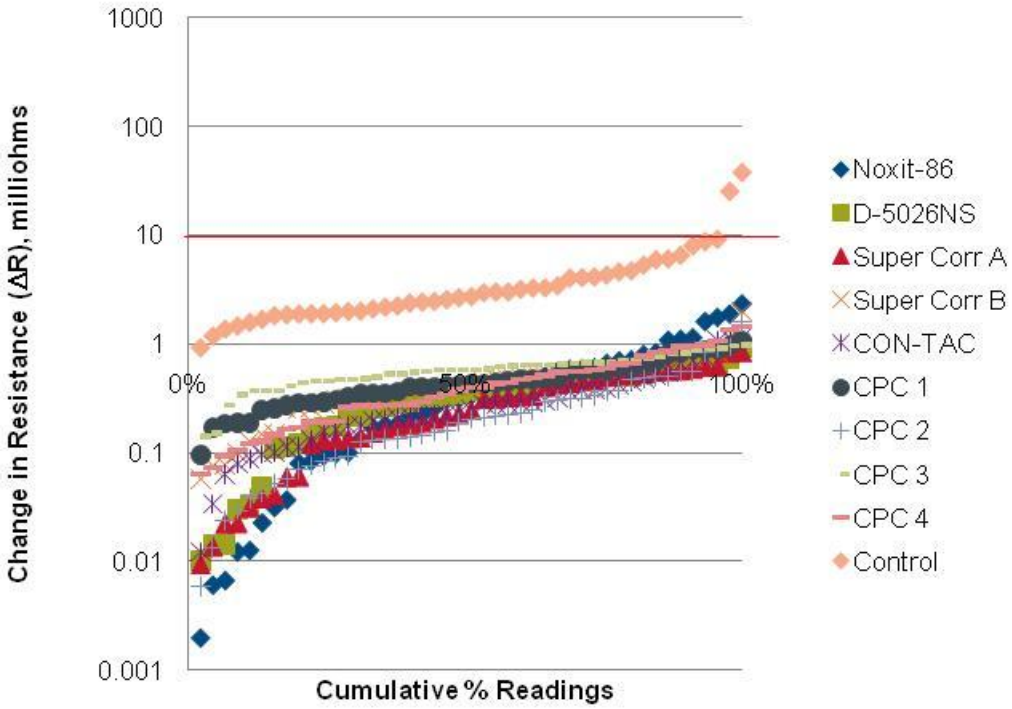
- Electrochemical Impedance Spectroscopy (EIS)
  - Estimates corrosion rate
  - Rapid evaluation of thin coatings
  - Results plotted in a **Bode** plot (Impedance vs. Frequency)





Battelle Field Testing Results

- Connector field testing – West Jefferson, OH
  - All CPC lubricants passed the five month exposure with a change in the initial contact resistance of <10 mΩ

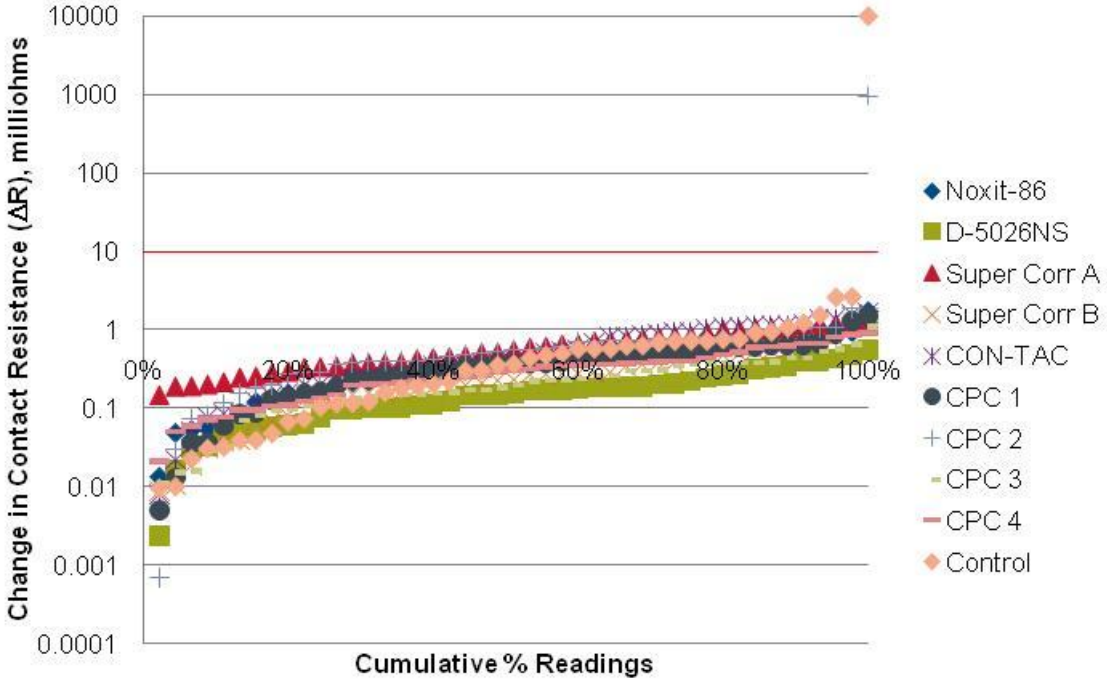


Change in Contact Resistance After 5 Months Field Exposure at West Jefferson Test Location

Battelle Field Testing Results

- Connector field testing – FMRF Daytona Beach, FL
  - All passed the five month exposure with a change in the initial contact resistance of <10 mΩ

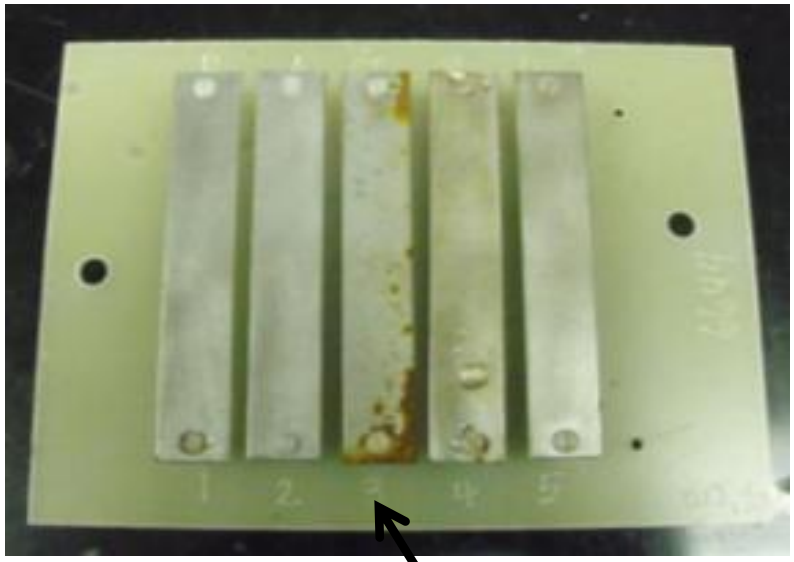
FMRF Connector Field Assembly



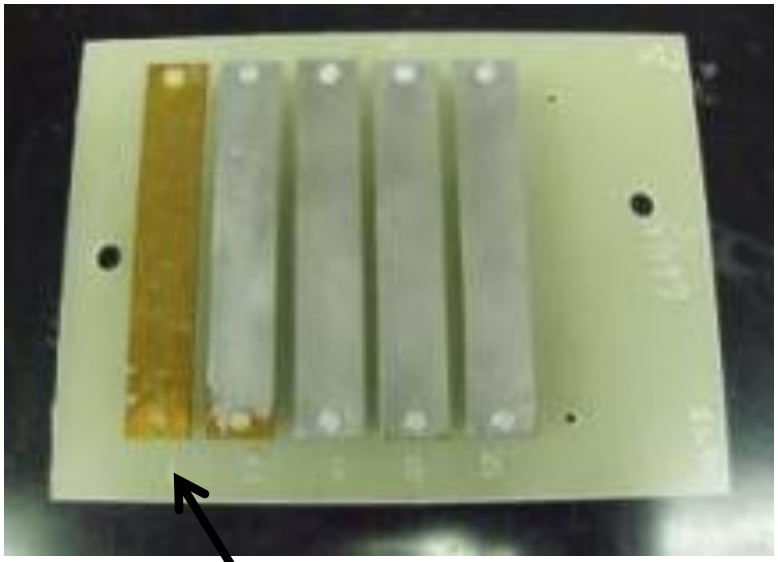
Change in Contact Resistance After 5 Months Field Exposure at FMRF Test Location

Battelle Field Testing Results – Corrosion Coupons – West Jefferson, OH

- All lubricants showed improved corrosion resistance over the control
- CON-TAC showed the least corrosion resistance
- CPCs No. 3 and 4 performance comparable to SC-A and SC-B

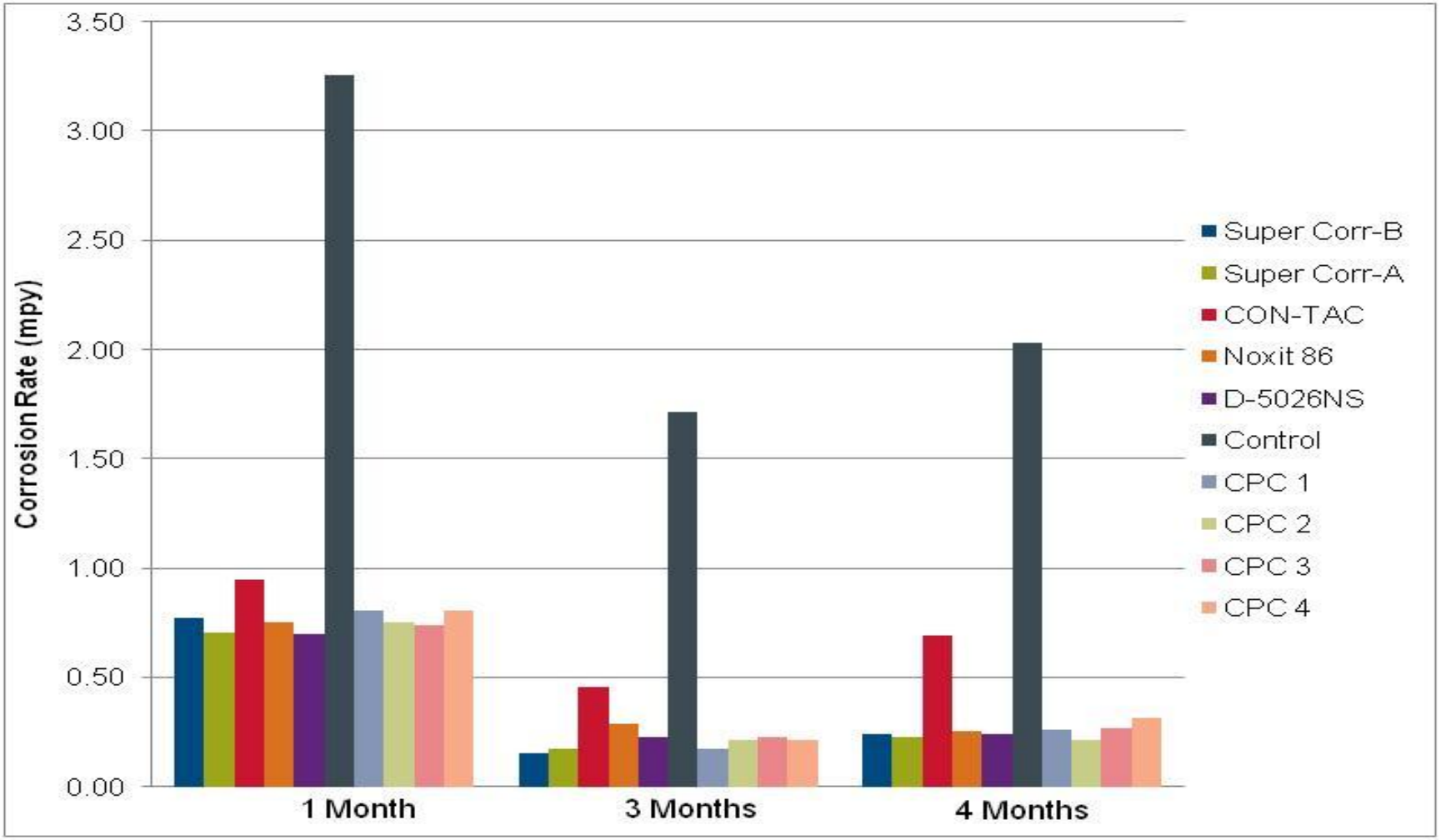


CON-TAC



Control

Battelle Field Testing Results – Corrosion Coupons – West Jefferson, OH



Corrosion Rates Calculated for Corrosion Coupons Exposed Vertically at West Jefferson Test Location

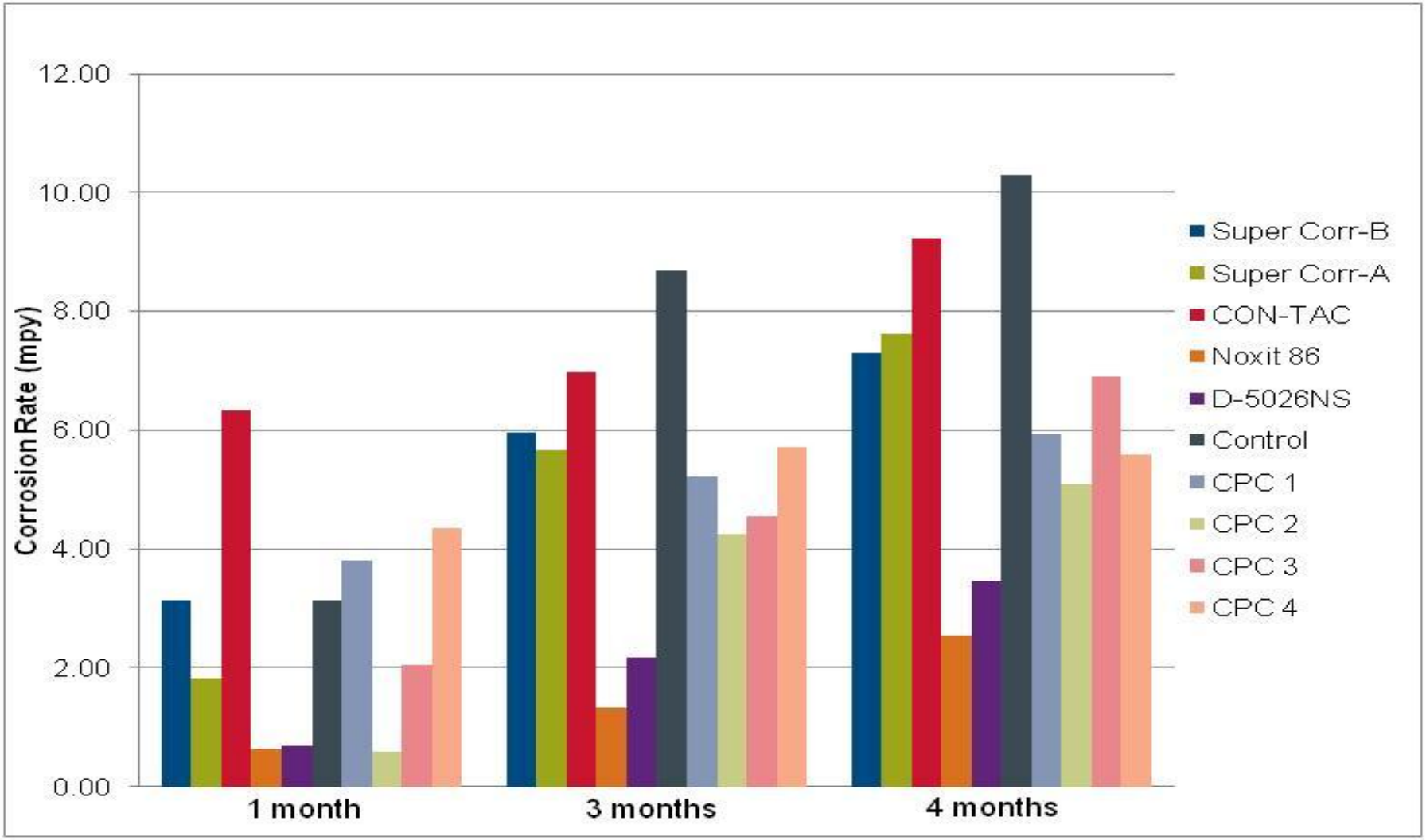
Battelle Field Testing Results - Corrosion Coupons @ FMRF Location

- Similar CPC performance was observed at FMRF with increased overall corrosion on all coupons due to the harsher environmental conditions
- CON-TAC showed the greatest overall corrosion following the control

CPC Coated Corrosion Coupon Sets Mounted at FMRF Test Location:

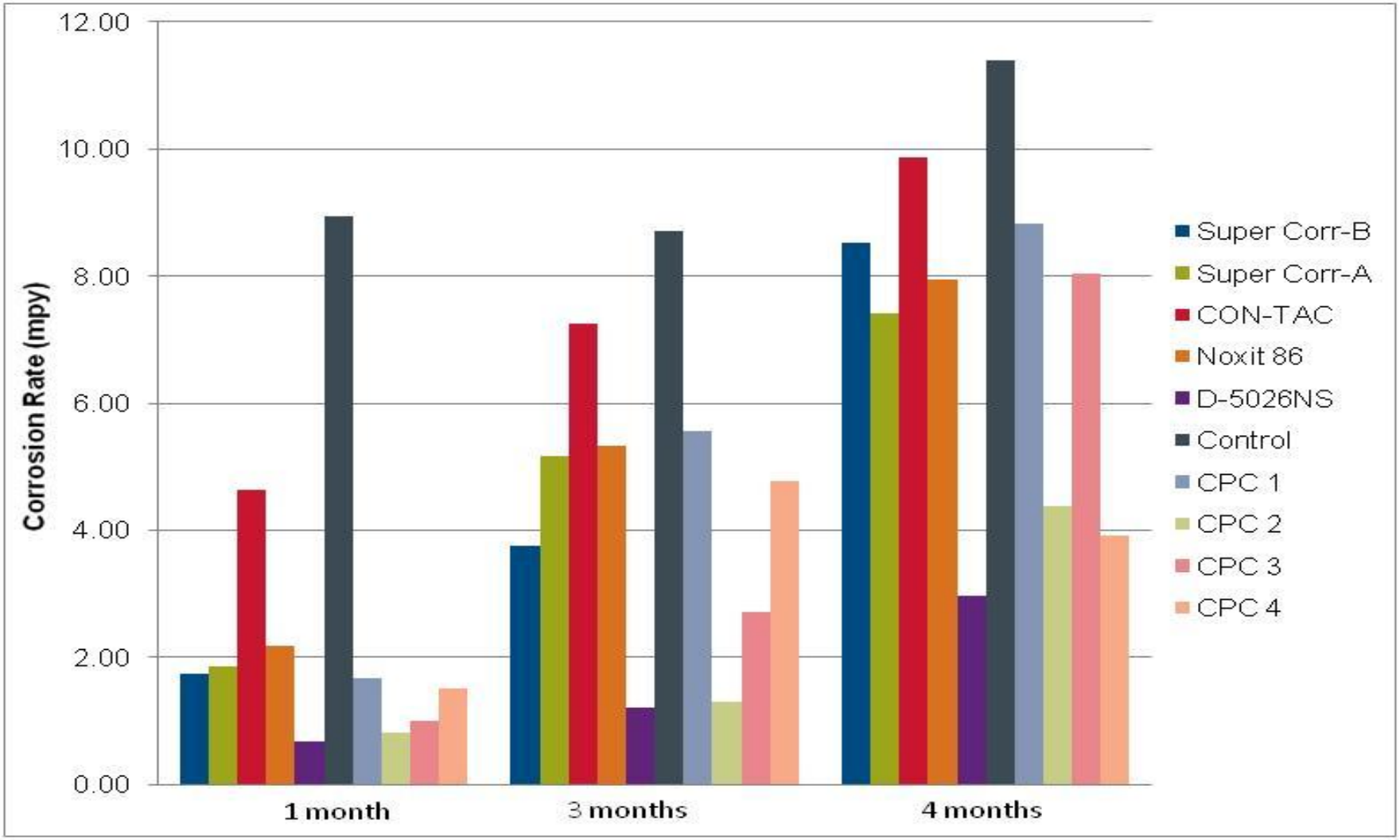
Horizontal Mount (left)
Vertical Mount (right)

Battelle Field Testing Results – Corrosion Coupons @ FMRF Location



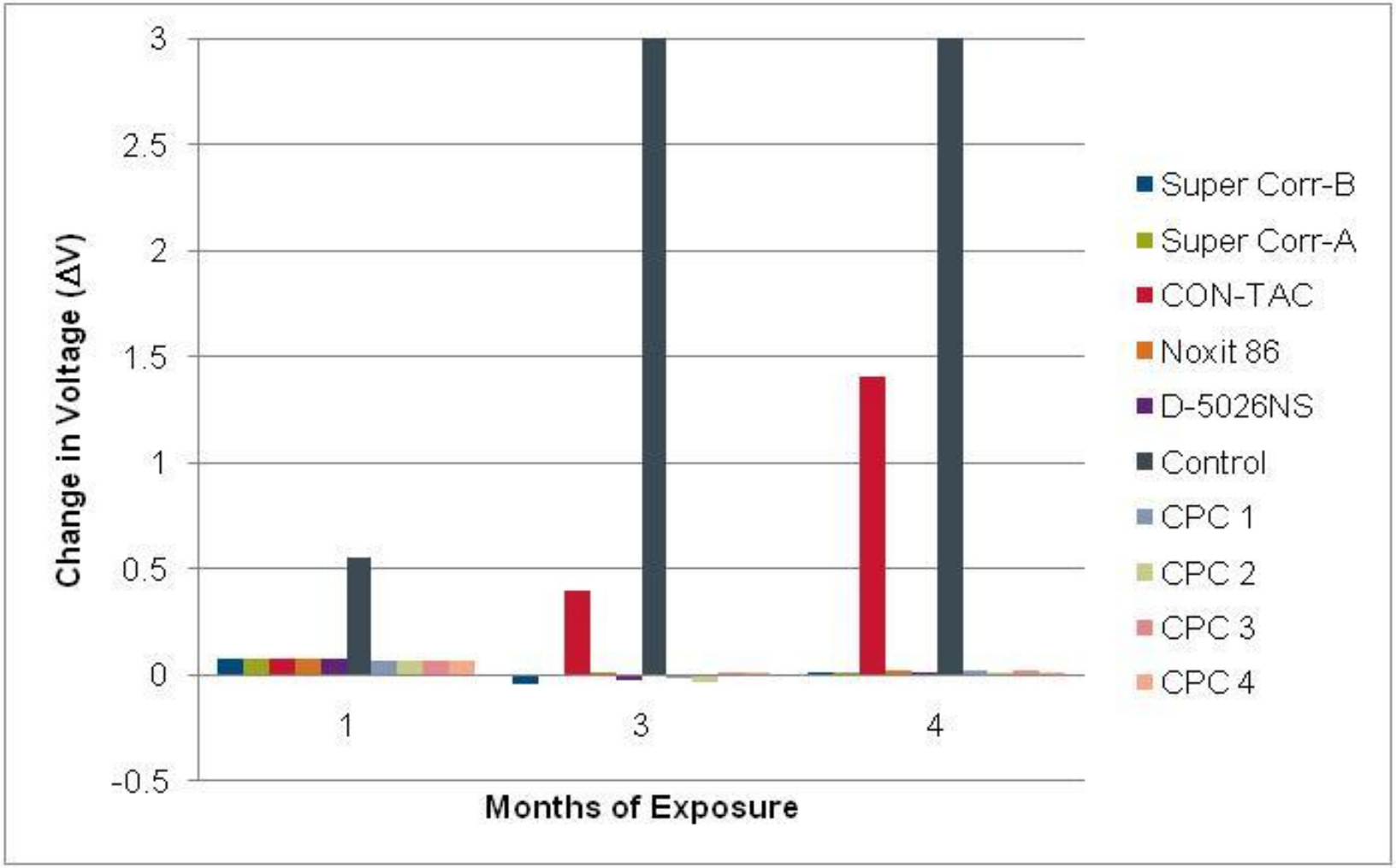
Corrosion Rates Calculated for Corrosion Coupons Exposed Horizontally at FMRF Test Location

Battelle Field Testing Results – Corrosion Coupons @ FMRF Location



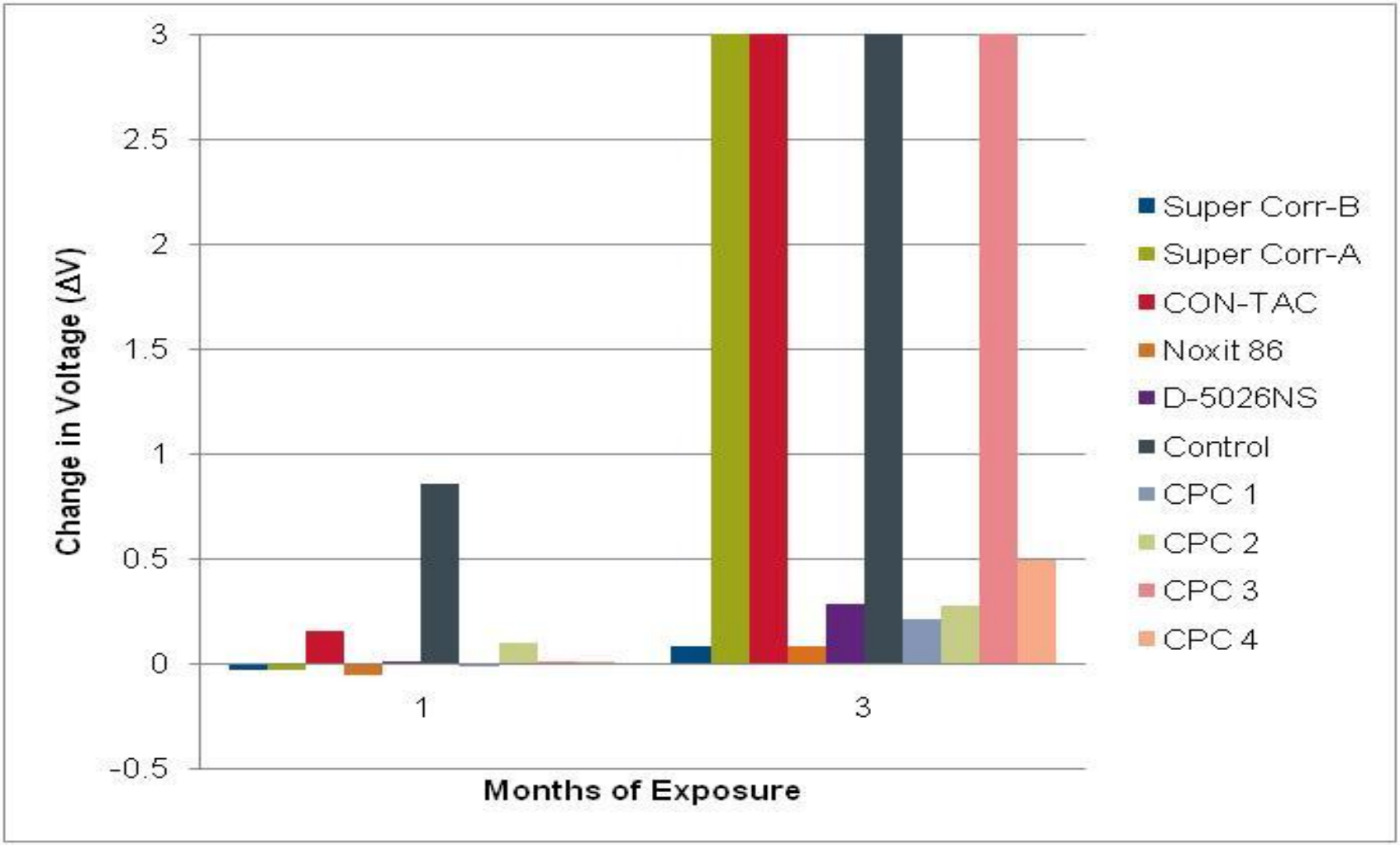
Corrosion Rates Calculated for Corrosion Coupons Exposed Vertically at FMRF Test Location

Battelle Field Testing Results – Steel Sensors at FMRF and West Jefferson



Steel Sensors Exposed Vertically at West Jefferson Test Location

Battelle Field Testing Results – Steel Sensors at FMRF and West Jefferson



Steel Sensors Exposed Vertically at FMRF Test Location