National Aeronautics and Space Administration

Environmentally Friendly Corrosion Preventative Compounds

Final Report

Submitted to: William B. Simmonds

Ground Systems Development and Operations

Principal Investigator: Dr. Luz Marina Calle

Project Team: Dr. Eliza Montgomery, Dr. Mark Kolody, Jerry Curran, Teddy Back, Angela Balles

November 30, 2012

Engineering Technology Directorate

Materials Science Division

Materials Engineering Branch

Corrosion Technology Laboratory

NASA Kennedy Space Center

Table of Contents

Figures

Tables

EXECUTIVE SUMMARY

The objective of the Ground Systems Development and Operations Program Environmentally Friendly Corrosion Protective Coatings and Corrosion Preventive Compounds (CPCs) project is to identify, test, and develop qualification criteria for the use of environmentally friendly corrosion protective coatings and CPCs for flight hardware and ground support equipment. This document is the Final Report for Phase I evaluations, which included physical property, corrosion resistance, and NASA spaceport environment compatibility testing and analysis of fifteen CPC types. The CPCs consisted of ten different oily film CPCs and five different wax or grease CPC types. Physical property testing encompassed measuring various properties of the bulk CPCs, while corrosion resistance testing directly measured the ability of each CPC material to protect various metals against corrosion. The NASA spaceport environment compatibility testing included common tests required by NASA-STD-6001, "Flammability, Odor, Offgassing, and Compatibility Requirements and Test Procedures for Materials in Environments that Support Combustion". At the end of Phase I, CPC materials were down-selected for inclusion in the next test phases.

This final report includes all data and analysis of results obtained by following the experimental test plan that was developed as part of the project. Highlights of the results are summarized by test criteria type.

Physical Testing:

No critical problems were discovered during the sprayability, removability, or wire compatibility testing.

Results for viscosity, CPC wettability, CPC hydrophobicity, and functional penetration were reported, although no pass or fail criteria were established based on these results. These results will be used when determining appropriate end-use applications in the upcoming test phases.

Atmospheric Corrosion:

CPCs did offer a significant amount of corrosion protection when considering the aggressive long-term six month atmospheric testing performed at KSC's Beachside Atmospheric Corrosion Test Site. All of the CPC types performed similar to or better than the control on carbon steel, but behaved differently on the stainless steel and aluminum alloys. No CPC performed the best in all corrosion evaluations; therefore, the CPCs will be best ranked by end-use application.

NASA Spaceport Environment Compatibility:

All of the CPC types met the NASA flammability requirements. All but two of the CPC types met all of the hypergolic fluids compatibility requirements. The liquid oxygen compatibility requirement was determined to be impractical, as currently no CPC-type materials are foreseen to be in contact with the pressure vessels. No critical incompatibility issues were discovered through the NASA spaceport environment compatibility testing.

1 INTRODUCTION

The objective of the Ground Systems Development and Operations Program Environmentally Friendly Corrosion Protective Coatings and Corrosion Preventive Compounds (CPCs) project is to identify, test, and develop qualification criteria for the use of environmentally friendly corrosion protective coatings and CPCs for flight hardware and ground support equipment.

Typically, when a bare metal surface could or should not be coated with a permanent coating (paint or sacrificial coating), a temporary coating, CPC, is used to protect the exposed surface from corrosion. CPCs commonly contain corrosion inhibitors suspended in a mixture of solvents and a base oil or grease. The base oil acts as a carrier fluid for the inhibitors and also as a protective barrier to environmental elements. The solvent acts as a base oil and inhibitor dispersant and is intended to evaporate after application. CPCs can be soft or hard films, and can be primarily composed of a petroleum, hydrocarbon, or fluoropolymer material depending on their end user requirements. Although CPCs provide corrosion protection, there are a number of environmental and safety issues associated with their use:

- Base oils are not environmentally benign
- Solvents can be high in volatile organic compounds (VOCs) and toxic
- Corrosion inhibitors can be toxic
- Worker safety issues

This report contains the critical requirements and tests necessary to evaluate environmentally friendly CPCs as effective corrosion control. These tests were derived from engineering, performance, and operational impact (supportability) requirements defined by a consensus of NASA participants.

It was decided at the beginning of the project that the most efficient way to manage the report of the background research, testing plans, and corresponding results was to create a single document that would be completed by adding information as it became available. To minimize duplication of effort, the final report will serve as a reference for future CPC users at NASA, the Department of Defense (DoD), other government organizations, and commercial users.

2 BACKGROUND

CPCs typically fall within the categories of water displacing to non-water displacing soft films and water displacing to non-water displacing hard films. The exact composition of many CPCs remains unknown due to their proprietary nature. Information available in the Materials Safety Data Sheets (MSDSs) reveal that they may include some of the following elements:

- an oil, grease or resin based film former
- a volatile, low surface tension carrier solvent
- a nonvolatile hydrophobic additive
- various corrosion inhibitors or surface active agents

Water displacing CPCs spread across the surface of the metal parts, into tiny holes, cracks, and crevices where they displace moisture and leave a film behind to act as a protective barrier. Non-water displacing CPCs dry to a soft waxy, greasy, or somewhat thicker film and provide a barrier film to most corrosive environments.

Water displacing CPCs are useful in providing supplementary protection for paint systems that have deteriorated or become damaged in service. They are applied as fluids by wiping, brushing, spraying, or dipping, and are usually immiscible with water and displace water from surfaces and crevices. The evaporation of solvents leaves either thin soft films, semi-hard films, or hard resin films that provide varying degrees of corrosion protection.

CPCs have been used at NASA since at least the 1980's[1], though earlier use is likely. CPCs are used to protect the aft skirts of Solid Rocket Boosters (SRB's)[2], as general lubrication and can provide corrosion protection, as in the case of the well-known product WD-40®. They have been used on the orbiters as temporary films to control corrosion[3] and are currently used as lubricants and corrosion barriers on connectors for the International Space Station's (ISS) and International Low Docking System (ILDS). Beyond NASA's use, the DoD is a primary user of CPCs. CPCs are used by all of the DoD services primarily in transport vehicle and munitions applications. Historically, CPCs have been comprised of petroleum base oils with corrosion inhibitor additives. Recently, more environmentally friendly base oil options that claim to provide the same corrosion protection as the petroleum-based counterparts, most notably using canola and soy-based oils, have been developed. This test plan aims to identify those CPCs that provide corrosion protection for NASA's use and are considered non-toxic to the natural environment.

---

[1] Simmons, J.R., *NASA CR-161431: Study of Etchants for Corrosion-Resistant Metals, Space Shuttle External Tank*, Martin-Marietta Aerospace prepared for NASA – George C. Marshall Space Flight Center, 1980.

[2] Novak, H.L., Hall, P.B., *Environmentally Compatible Vapor-Phase Corrosion Inhibitor for Space Shuttle Hardware*, 5th Conference on Aerospace Materials, Processes, and Environmental Technology, 2003.

[3] The Boeing Company, *Use of Corrosion Preventive Compounds on Space Shuttle Orbiter*, Specification MF0004-135, 2006.

2.1 CPC Applications

2.1.1 CPCs for Ground Support Equipment at Kennedy Space Center

The Corrosion Control and Treatment Manual, TM-584C[4], highlights multiple applications where CPCs are to be used to control corrosion of materials in facilities, systems, and equipment at KSC. The manual cites for CPC use in the protection of exposed bearing surfaces, tubular structural steel, electrical connectors, steel cabling, piano-type hinges, adjustable parts, and bare metal piston surfaces using corrosion inhibiting lubricants in the form of oil and greases. The manual cites several military specifications to refer to many of the NASA approved CPC types. The specifications are listed as: MIL-PRF-16173E[5] (NAVSEA), grades, 2, 3, and 4, MIL-DTL-23549D[6] (NAVAIR), MIL-PRF-81322G[7] (NAVAIR) MIL-PRF-46000D[8] (Army), MIL-PRF-46010D[9] (Army), and MIL-PRF-46002D (Army).[10]

CPCs are used for temporary corrosion protection on both bare metal and coated, often damaged, surfaces on ground support equipment, including but not limited to the Mobile Launcher Platform (MLP), the fixed service structures (FSS) at the Launch Pads, and the Crawler-Transporters. One major use is on the Thrust Vector Control (TVC) frames that structurally support components of the TVC system that is located in the aft skirt of the Solid Rocket Boosters (SRBs).[2] TVC frames are exposed to the seacoast environment after refurbishment, seawater immersion after splashdown, and during tow-back to Cape Canaveral Air Force Station (CCAFS)-Hangar AF refurbishment facilities. During refurbishment operations, it was found that numerous TVC frames were experiencing internal corrosion and coating failures, both from salt air and seawater intrusions. Inspectors using borescopes would visually examine the internal cavities of the complicated aluminum alloy welded tubular structure. It was very difficult for inspectors to examine cavity corners and tubing intersections and particularly, to determine the extent of the corrosion and coating anomalies. Physical access to TVC frame internal cavities for corrosion removal and coating repair was virtually impossible, and an improved method, using a CPC for preventing initiation of new corrosion and mitigating and/or stopping existing corrosion growth, has been used ever since.[2]

---

[4] NASA, *TM-584C, Corrosion Control and Treatment Manual*, November 1, 1994.

[5] DoD, *MIL-PRF-16173E(SH), Performance Specification Corrosion Preventive Compound, Solvent Cutback, Cold-application*, September 7, 2006.

[6] DoD, *MIL-DTL-23549D, Detail Specification, Grease, General Purpose*, May 10, 2002.

[7] DoD, *MIL-PRF-81322G, Grease, Aircraft, General Purpose, Wide Temperature Range*, January 24, 2005.

[8] DoD, *MIL-L-46000 Lubricant, Semi-Fluid (Automatic Weapons)*, February 25, 1987.

[9] DoD, *MIL-L-46010 Lubricant, Solid Film, Heat Cured, Corrosion Inhibiting*, August 6, 2008.

[10] DoD, *MIL-P-46002 Preservative Oil, Contact and Volatile Corrosion-Inhibited,* January 20, 2010.

The current NASA Engineering Structures Division cited multiple uses of CPCs. CPCs are commonly used specifically for corrosion prevention on the Vehicle Assembly Building (VAB) Vertical Door lower limit switch springs.[11] CPCs are used elsewhere, but as lubrication and corrosion protection in tandem. Some applications include wire rope, moving parts, and electrical connections, on cranes and general structures.

CPCs are used on the ISS for the iLIDS, which is a government furnished connector design made for anyone to dock components to the ISS. The iLIDS components consist of mixed metals, such as Aluminum alloys (2219, 2024, 7075), Stainless steels, titanium (for hook assembly), and 440C and 52100 high alloy steel (for bushing and bearing materials), and Aluminum-bronze (for bushings and pins). There are issues with faying surfaces and galvanic couples that are corrected using CPCs.[12] One problem noted was that, although the more corrosion resistant alloy 440C is specified, the less corrosion resistant alloy 52100 is often used due to alloy availability issues. Should this problem continue, an increased use in temporary CPC coatings will result.[13]

In the past, CPCs were used on the Space Shuttle orbiters to cover paint nicks between repairs.[14] Because the CPCs must survive the launch environment, Low Earth Orbit, and other flight cycle environments, thickened grease materials were used. They greases were often fluorinated vacuum greases with corrosion inhibitor additives.[15] When the Space Shuttles were flying, the frequency of corrosion issues on the orbiters regularly exceeded 400 cases annually.[16] Typically, locations where CPCs were used on the orbiters were the rudder speed brake, vertical tail, elevons, wing leading edge, ET door cavity, and body flap.[14] The longest time a CPC protected the substrate was four mission cycles.[15]

One application that has been identified as a possible future use for CPCs at NASA is to temporarily cover space flight hardware that consists of bare metal components, prior to launch. Current material specifications require that all manufacturers' coatings (usually CPCs) be removed prior to use. When space flight hardware awaiting launch is exposed to KSC's atmospheric conditions, corrosion occurs on the surface. A temporary CPC coating that could be removed prior to launch can be considered as an ideal solution to this problem.[17]

---

[11] Van Den Dreissche, J. NASA Kennedy Space Center, email correspondence to E. L. Montgomery, December 16, 2011.

[12] Shindo, D., NASA Johnson Space Center, Personal interview, September 20, 2011.

[13] Dube, M. NASA Goddard Space Flight Center, Personal interview, October 11, 2011.

[14] Patterson, J.D., Corrosion Inhibiting Grease Study, Boeing Lab Report No. M&P-3-1868, August 24, 2007.

[15] Hale, S., Identification of the Effectiveness of Current Coatings and Corrosion Preventive Compounds Used on the Space Shuttle Orbiter, Report No. SETS FPR23100.8, September 9, 2005.

[16] Hale, S., Corrosion Preventive Compounds Lifetime Testing, United Space Alliance, April 19, 2007.

[17] Dellacorte, C. NASA Glenn Research Center, Personal Interview, October 18, 2011.

2.1.2 CPC Applications throughout NASA, DoD and the Aerospace Industry

CPCs are used at NASA and extensively throughout the DoD in aircraft, ship, transport vehicle, and armored vehicle applications, as well as on many types of ground support structures and munitions. From a materials perspective, metal substrates are used in the majority of vehicles and structures, therefore the opportunities for corrosion problems abound. It is estimated that the cost of corrosion to the DoD is estimated between $10 billion and $20 billion dollars annually.[18] Although NASA has not conducted a formal cost of corrosion study, it can be inferred that given the highly corrosive conditions at KSC and the even more severe corrosion conditions of the launch environment, that the cost of corrosion at NASA is also significant.

2.1.2.1 <u>Aircraft Applications</u>

Aircraft face some of the most common corrosion problems encountered throughout the DoD and in the general aerospace industry. The constant cycling of wetting and drying due to condensation that occurs during take-off and landing is a root cause of much of the corrosion problems. Because of the shape of aircraft, there are many crevices and occluded areas built into the design that become traps for moisture. Aircraft have many components that are bare metal, as the substrates are almost always aluminum-based alloys. The lack of a protective layer, other than the natural oxide film, makes the substrate more prone to corrosion. Services, including the U.S. Air Force, Marines, Army, and NAVAIR, all face the same types of problems with their aircraft regardless of type. The most common areas of corrosion where CPCs are used include beams, joints, fastener areas, electrical wiring components,[19] inner and outer mold lines, cargo floor end fittings, fuselage belly skins, wheel well aft bulkheads, mainframes, stringers,[20] landing gear, flapwells,[21] lap joints, beneath the floorboards in the bilge areas,[22] the lavatory and galley, wing interior sections, doors and hatches, skin panel faying surface.[23] The F-18 has had some of the most severe corrosion problems thus far because dissimilar metals and a lack of drain holes for moisture build-up were flaws inherent to the design. CPCs have been used to control this type of corrosion, ever since the problems first surfaced.[22]

---

[18] CorrDefense, Why DoD Must Protect its Assets, DoD Office of Corrosion Policy and Oversight website: https://www.corrdefense.org/CorrDefense%20WebPage%20Content/WhyDoDMustProtectItsAssets.aspx.

[19] Jones, S. C-130 CPC Application and Evaluation Program, 2003 Air Force Corrosion Conference, 2003

[20] McTish, D., Jones, S., C-5 Corrosion Prevention Compound Application Program, 2005 Air Force Corrosion Conference, March 14-17, 2005.

[21] Abbott, W. A Decade of Corrosion Monitoring in the World's Military Operating Environments, A Summary of Results, 2008.

[22] Shah, S.R., Shoales, G.A., Fawaz, S.A., Lap Joint Integrity and Corrosion Preventive Compound Evaluation Using Electrochemical Impedance Spectroscopy.

[23] Arafat, E., High Performance Corrosion Preventive Compound for Internal Aircraft and Other Weapon System Applications, ESTCP Project WP 0615, Final Report, November 15, 2010.

2.1.2.2 Marine Applications

Much of the vehicles used in marine environments, especially those deployed at sea, use paints and cathodic protection to manage corrosion because they need more permanent solutions to block the direct metal contact with the seawater. One common vehicle that routinely uses CPCs for corrosion control is the Expeditionary Fighting Vehicle (EFV). This vehicle is an amphibian type that sees both seawater, freshwater, and many cycles of drying. The seal frame, armor panel frame, threaded inserts, fasteners, and the environmental seal areas are the most common places that CPCs are used.[24]

2.1.2.3 Ground Operations Applications

Ground operations face multiple corrosion problems with fixed structures, transport vehicles, and armored vehicles. The Army and Marines have the common problems with corrosion on the ground. CPCs are heavily used for electrical hardware, fuel cell rooms,[25] occluded sites (hinges, fasteners, under lap seams),[26] and hydraulic lifts.[27] Both transport and armored vehicle types, including high mobility multipurpose wheeled vehicles (HMMWVs), trucks, medium tactical vehicle replacements (MTVRs), and internally transportable vehicles (ITVs) heavily use CPCs as a last layer of corrosion defense over their vehicle paint.[24]

2.1.2.4 Launch Applications

Non-NASA launch vehicles and structures also face critical corrosion issues. In 2008, Space Exploration Technologies faced a failure of their Falcon 1 launch due to a corroded aluminum bolt.[28] Depending on their location with respect to seawater, launch structures will face differing degrees of corrosion; however, corrosion will most commonly exist on the fixed structures (fasteners, exposed metal, and all areas (similar to those identified in the NASA Corrosion Control and Treatment Manual), the rocket interior and exterior, the fuel cell areas, and the mixer assembly areas.[29]

[24] Arafat, E., Demonstration/Validation of High Performance Corrosion Preventive Compound for Interior Aircraft Applications, SERDP/ESTCP Workshop, Tempe AZ, February 26-28, 2008.

[25] Army Aviation, 2005 Air Force corrosion Conference, March 14-17, 2005.

[26] Price, K., Dante, J. CPC Performance in Occluded Sites, 2005 Tri-Service Corrosion Conference.

[27] Ferris, D., Darter, K., Hays, R. US Marine Corps Corrosion Programs, 2004 Air Force Corrosion Conference, March 9, 2004.

[28] Berger, B., Falcon 1 Failure Traced to a Busted Nut, Space.com, July 19 2006. http://www.space.com/2643-falcon-1-failure-traced-busted-nut.html.

[29] Ellicks, D., Bloyer, J., Alternative Coatings for Missile Launch Support, 2004 Air Force Corrosion Conference.

2.2 CPC Technologies

2.2.1 Current CPC Technologies

Since the beginning of their use, CPCs have primarily been comprised of petroleum-based carrier oils, corrosion inhibitors, surfactants, and solvents.[30,31,32] The use of petroleum gives CPCs an unlimited shelf life, because the oils slowly oxidize over time. In general, these petroleum-based products require personal protection equipment during use and are harmful to the natural environment if spilled.[33] Some CPCs are made using lanolin-based carrier oil[34] or a high grade machine oil.[35] The CPC manufacturers have begun to lower the solvent content in their CPCs so that they have low Volatile Organic Components (VOCs).[36] This effort is primarily due to public demand to make the CPCs less harmful to the environment.

2.2.2 Environmentally Friendly CPCs: State of the Art

New CPC products are being made with canola, soy, and other vegetable-based carrier oils.[37,38] These products are also made so that they are solvent free, thus they contain no VOCs. The advantage to these products is that they are non-toxic and are easy to dispose of. They are made with no carcinogenic compounds or hazardous materials. They are also a renewable resource which will decrease our dependence on foreign oil and help federal agencies meet their sustainability goals under Executive Orders (EO) 13514 Federal Leadership in Environmental, Energy, and Economic Performance and EO 13423 Strengthening Federal Environmental, Energy, and Transportation Management. There are questions as to the durability of these new plant-based CPCs, as the carrier oils are more likely to degrade at a faster rate than their petroleum-based counterparts. Because CPCs are meant, in most cases, to be used as a temporary line of defense from corrosion, many CPC manufacturers claim that their products perform the same as or better than petroleum-based products as a temporary protection in the normal use time.

---

[30] Gui, F., Novel Corrosion Schemes for the Aerospace Industry, Corrosion Control in the Aerospace Industry, Benavides, S. editor, Woodhead Publishing Limited, Cambridge, England, 2009, p249.

[31] Corrosion Technologies Corporation, Corrosion X MSDS, 2011.

[32] Cortec Corporation, VpCI-368 MSDS, 2011.

[33] PMS Products, Inc. Boeshield T-9 MSDS, 2011.

[34] Eureka Chemical Company, http://www.fluid-film.com/environment/index.html.

[35] Akin, K.D., Greases and Their Role in Corrosion Control in the Aerospace Industry, Benavides, S. editor, Woodhead Publishing Limited, Cambridge, England, 2009, p267.

[36] NAVAIR, Office of Research and Technology Applications, NAVGUARD, Navy Case #95904 and Nacy Case #97473, 2006.

[37] Cortec Corporation, EcoLine 3220 MSDS, 2011.

[38] Renewalbe Lubricants, Inc., Bio-Medium Preservative Liquid MSDS, 2008.

3 ENGINEERING, PERFORMANCE, AND TESTING REQUIREMENTS

A group led by NASA and consisting of technical representatives from NASA centers discussed engineering, performance, and testing requirements for environmentally friendly CPCs. The group defined critical tests with procedures, methodologies, and acceptance criteria to qualify alternatives against these technical requirements.

Once the test plan criteria were approved, testing was performed in a manner that optimized the use of each test panel. For example, where practical, more than one type of test was performed on the coated test panels. The number and types of tests performed on a given panel will be determined by the destructive nature of the tests in question.

This project compared the performance of environmentally friendly CPCs candidates on various metal substrates used for flight hardware and ground support equipment. The tests described in this test plan are summarized in Tables 1 – 4 which include acceptance criteria and the reference specifications, if any, used to conduct the tests. A more thorough discussion of the testing is provided later in this report.

Table 1. Physical Property Testing

Test	*Test Specimen*	*Acceptance Criteria*	*Test Methodology References*
Application Characteristics	Judged when long-term atmospheric exposure samples are prepared.	Based on Applicator Evaluation: Smooth coat, with acceptable appearance. Ability to cover substrate properly. Sprayable after 20 hours at 40 degrees F.	MIL-PRF-81309F, ASTM D 4414, SSPC PA-2
Viscosity	Liquid Sample, 3 per CPC	record value	ASTM D445
Contact Angle, Wettability of CPC	Liquid Sample, 10 per CPC	record value	ASTM D7334
Contact Angle, Hydrophobicity of CPC on Substrate	2”x2”x0.125”, 10 per CPC	record value	ASTM D7334

Functional Penetration	2, 4” x 6” x 0.125”, Al 7075-T6 coupons sandwiched, treated in accordance with MIL-PRF-23377	No panel faying surface area to be less than 80 percent wetted in 24 hours. Average of two panels to be 85 percent or better, wetted in 24 hours.	MIL-PRF-81309F
Wire Compatibility	24 inches of wire conforming to MIL-W-81381/11, MIL-W-81044, MILW-5086, and MIL-W-81822/6, 4 per wire, 3 per CPC	No cracking or degradation of insulation following prolonged exposure	MIL-PRF-81309F
Removability	4”x6”x0.125” Al 7075 Coupon, 1 per CPC	Completely removable with Mineral Spirits	MIL-PRF-81309F

Table 2. Accelerated Corrosion Testing

Test	*Test Specimen*	*Acceptance Criteria*	*Test Methodology References*
Cyclic Salt Fog	3”x6”x0.125” Coupon, 3 per CPC	Performs better than untreated. Performs similar to control CPC.	ASTM D5894

Table 3. Atmospheric Corrosion Testing

Test	*Test Specimen*	*Acceptance Criteria*	*Test Methodology References*
Long-term Beachside Atmospheric Exposure	4”x6”x0.125” Coupon, 7 per alloy, 6 per CPC	Performs better than untreated. Performs similar to control CPC.	ASTM D610, ASTM G1, ASTM G33, ASTM G 44, ASTM G46, ASTM G50
Sandwich Corrosion	4” x 6” x 0.125”, Coupons sandwiched, 4 per alloy, 3 per CPC	Performs better than untreated. Performs similar to control CPC.	ASTM F1110, ASTM G 50
Crevice Corrosion via Fasteners	Same panel as the sandwich corrosion panel, 316 SS washers as the crevice inducer, 4 per alloy, 3 per CPC	Performs better than untreated. Performs similar to control CPC.	ASTM G78, ASTM G 50
Galvanic Corrosion via Fasteners	Same panel as the sandwich corrosion panel, 316 SS washers as the galvanic corrosion inducer, 4 per alloy, 3 per CPC	Performs better than untreated. Performs similar to control CPC.	ASTM G104, ASTM G 50
Wire on Bolt Atmospheric Galvanic Corrosion	1100 aluminum anode wire wrapped around cathode rods of nylon, 1010 mild steel, and CA110 copper	Performs better than untreated. Performs similar to control CPC.	ASTM G116, ASTM G 50
Stress Corrosion Cracking	0.75”x5”x0.60” Bent Coupon, 7 per alloy, 6 per CPC	Performs better than untreated. Performs similar to control CPC.	ASTM G47, ASTM G 50

Table 4. Compatibility with NASA Environments

Test	*Test Specimen*	*Acceptance Criteria*	*Test Methodology References*
LOx Compatibility	4"x6"x0.125" SS Coupon, cut into 0.75" diameter samples, 20 per alloy	Twenty samples must not react when impacted at 72 foot-pounds [ft-lbs or 98 Joules (J)]. If one sample out of 20 reacts, 40 additional samples must be tested without any reactions.	ASTM D 2512; NASA-STD-6001
Hypergol Compatibility	4" x 4" aluminum foil coupon, 1 per CPC	Slight to Moderate Reactivity Observed: When test data based on visual observations with the unaided eye reveal reactivity (but no ignition) and/or any changes in the visual	KSC MTB-175; NASA-STD-6001
Flammability	12" x 2.5", 1 alloy, 5 per CPC	No test specimen of the five standard-sized specimens burns >6 inches. No test specimen propagates a flame by the transfer of burning debris.	NASA-STD-6001, ISO 14624-1:2003

4 SELECTED ALLOYS AND CPCs

For each test requiring panels, a minimum of five (5) coupons were prepared. Those with the best coating (as determined by the technician) were used in accordance with the number of coupons specified in the Test Methodology. Unless otherwise required by a specific test, all coupons were prepared as follows:

Metal coupons WERE prepared in accordance with NACE-STD-RP0281 [*Method for Conducting Coating (Paint) Panel Evaluation Testing in Atmospheric Exposures*].

Each CPC system was applied according to the instructions provided by the manufacturer. Coating systems will were applied by spraying, or, in the case of advanced film technology, by hand to the dry film thickness recommended by the coating manufacturer. Application was conducted at a minimum temperature of 75 ± 5 degrees Fahrenheit (°F) and 50% ± 10% relative humidity (RH), unless otherwise specified.

Test Specimens

Table 5 contains a listing of substrate types that were used for testing.

Table 5. Test Specimen Codes and Substrate Descriptions

Test Coupon Code	*Substrate Description*
1010 CS	Carbon Steel: Low-carbon, cold-rolled steel complying with SAE 1008/1010 specifications.
304 SS	Stainless Steel: Austenitic Cr-Ni stainless steel complying with ASTM A240/A240M specifications.
2024-T3 Bare	Aluminum: Aluminum-copper (2xxx series) alloy complying with ASTM B209.
2219-T87	Aluminum: an age-hardenable copper containing alloy of aluminum complying with ASTM B209.
7075-T6	Aluminum: Al-Zn-Mg-Cu high strength alloy with the addition of chromium complying with QQ-A-250/12 specifications.
1100-O	Aluminum wire: Un-alloyed 99% pure aluminum wire complying with ASTM B221.
CLiMAT	1100 Aluminum wire on nylon, 1010 steel, and CA110 copper bolts.

5 CPCs OF INTEREST AND TYPES

CPCs have been used at NASA for several years. These CPCs have had varying degrees of protection based upon the alloy of interest and environmental conditions.

For the purpose of this report and project, CPCs were down-selected based upon the CPCs ability to protect ground support equipment. Of great interest is the desire to test and compare environmentally friendly CPCs and compare their performance to traditional (petroleum based) CPCs. For purposes of this project, environmentally friendly refers to CPCs that have low VOCs (less than 100g/L), are non-HAPs, and are non-toxic and non-carcinogenic.

A literature and vendor survey was conducted to down-select possible CPCs for use on ground support equipment at KSC. Although multiple CPC products are used at KSC, only one control was chosen, and the remaining CPC types were included for comparison purposes. The new CPC candidates are designated as such. Note the NAVGURARD I, CPC 3, was never received by the vendor; however, a new environmentally-friendly CPC was identified and added to the candidate list, CPC 15. Those CPCs chosen for testing are as follows:

Table 6. Corrosion Preventative Compounds Reviewed in this Study

	CPC Type	Sample Number	Product Name	Primary Composition
Soft Film	Oily film	1	Corrosion X Aviation (Control)	Petroleum distillates
	Oily film	2	WD-40 (for comparison)	Petroleum distillates
	Oily film	4	NAVGUARD II (for comparison)	Petroleum distillates
	Oily film	5	MX4 (for comparison)	High grade machine oil
	Oily film	6	EcoLine 3690 (candidate)	Canola oil
	Oily film	7	Zerust Axxanol 46-BIO (candidate)	Soy/Canola oil
	Oily film	8	Bio-Medium Preservative Lubricant (candidate)	Soy and canola oil
	Oily film	9	Fluid Film (candidate)	Lanolin
	Oily film	15	WRL (candidate)	High grade oil, biodegradable
Grease or Wax Film	Wax	10	VpCI 368 (Control)	Petroleum distillates
	Wax	11	Ardrox AV-30 (for comparison)	Petroleum distillates
	Wax	12	Nox-Rust 3100 (for comparison)	Petroleum distillates
	Wax	13	Bio-Acid Fume Rust Preventative Fluids (candidate)	Soy and canola oil based
	Grease	14	EcoLine Heavy Duty Grease (candidate)	Soybean oil and clay thickener

6 TEST DESCRIPTIONS

Test requirements are further defined in this section to include the test description, rationale, and test methodology. The Test Methodology lists the major parameters, test coupon descriptions, number of test coupons, number of coupons per coating system, number of control coupons and acceptance (pass/fail) criteria. Any Unique Equipment or Instrumentation requirements and Data Analysis and Reporting Criteria are also included. The latest revision of each specification or standard shall be used unless otherwise stated.

6.1 Physical Property Testing

6.1.1 Application Characteristics

<u>Test description</u>

This procedure was used to determine how easily a CPC system may be applied at room temperature and cooler temperatures. The film thickness was determined.

A set of test coupons was prepared noting the appropriate coating application processes and equipment. The coating was applied to panels, which consisted of cardboard pieces of known and uniform dimensions, under ambient conditions at 75 ± 5 °F and 50 ± 10% RH. A second set of panels was prepared after conditioning the CPC container for 20 hours at 40°F. The self-pressurized container was removed from the cold chamber and the contents were sprayed. The product shall be able to readily wet the surfaces of test coupons in order to pass the test. The applications characteristics were additionally judged as they were applied to metal panels in preparation for long-term beachside atmospheric exposure. A failure was denoted if froth, bubbling, or excessive runoff was present.

<u>Film Thickness</u>:

The Wet Film Thickness (WFT) was measured in accordance with ASTM D4414 (*Standard Method for Measurement of Wet-film Thickness by Notch Gages*). Note that the film did not dry completely; therefore three different WFT measurements were made instead. The WFT was measured immediately after the application with the panels in a flat orientation, 24 hours after application with the panel orientation flat, and 48 hours after the initial application and the panels held at a 60° angle (the same angle that the panel is held during salt fog chamber testing).

<u>Rationale</u>

This screening test was conducted to identify and eliminate those candidate CPCs that were difficult to properly apply under normal maintenance operation conditions.

Methodology Table

Table 7. Test Methodology for Application Characteristics

Parameters	Coating Manufacturer instructions; Application temperature, 75 ± 5 °F and 50 ± 10% RH Both room temperature application and 40 degree F temperatures
Coupons Per CPC	Three (3)
Trials Per CPC	One (1)
Control Coupons Required For Testing	Not Applicable – Each CPC will be judged to pass or fail based upon their own merit.
Acceptance Criteria	Shall not exhibit froth, bubbling, or excessive runoff and shall readily wet the surfaces of test panels. Measure WFT.

Unique Equipment or Instrumentation

Notched Wet Film Gauge

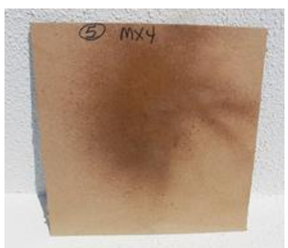
Data Analysis and Reporting

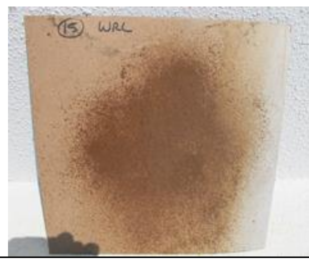
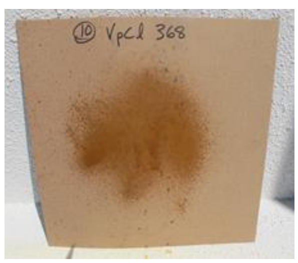
The CPCs were sprayed onto uniform pieces of cardboard to capture the spray pattern after two pumps of the spray nozzle. The results were photographed, shown in Table 8, for both ambient and cold spray conditions.

Under ambient spray conditions, the following CPCs sprayed evenly: Corrosion X, WD-40, MX4, EcoLine 3690, Zerust Axxanol, WRL, VpCI 368, and Nox-Rust. Bio-Medium Preservative Lubricant, Ardrox AV-30, and Bio-Acid Fume Rust Preventative Fluids sprayed as a stream. All of the CPCs performed worse for sprayability under the cold spray conditions, as they all sprayed as a thicker stream with little to no misting capabilities. Fluid Film and EcoLine Heavy Duty Grease were not sprayable and had to be applied with a brush.

Table 8. CPC Application Results for Sprayability

CPC Type	Coverage, Ambient Conditions	Coverage, Cold Conditions	Comments from Metal Panel Application

Corrosion X (Control)			Sprayed evenly, poor wettability
WD-40 (comparison)			Sprayed as an even mist
NAVGUARD II (comparison)			Thick spray (aerosol), poor wettability.
MX4 (comparison)			Sprayed unevenly, poor wetting.
EcoLine 3690 (candidate)			Sprayed unevenly, poor wetting.
Zerust Axxanol 46-BIO (candidate)			Sprayed as a thick mist, good wettability.

Bio-Medium Preservative Lubricant (candidate)			Sprayed as a stream, no misting.
Fluid Film (candidate)	n/a	n/a	No sprayability. Applied with brush.
WRL (candidate)			Sprayed as an even mist.
VpCI 368 (Control)			Sprayed as an even mist.
Ardrox AV-30 (comparison)			Sprayed as a stream, no misting.
Nox-Rust 3100 (comparison)			Sprayed as an even mist.

Bio-Acid Fume Rust Preventative Fluids (candidate)			Sprayed as a stream, no misting.
EcoLine Heavy Duty Grease (candidate)	n/a	n/a	No sprayability. Applied with brush.

The wet film thickness was measured immediately after CPC application (panels lay flat in a horizontal position), after 24 hours of curing in a flat (horizontal) position, and after 48 hours at a 60° angle. The corresponding results for wet film thickness are shown in Table 9.

Table 9. Wet Film Thickness Results

	CPC Type	WFT applied (flat), mils	WFT after 24 hours (flat), mils	WFT after 48 hours (60° angle), mils	Application Method (spray or brush)
Soft Film	Corrosion X (Control)	20	6	<1	Spray
	WD-40 (for comparison)	5	1	<1	Spray
	NAVGUARD II (for comparison)	2	2	<1	Spray
	MX4 (for comparison)	6	3.5	<1	Spray
	EcoLine 3690 (candidate)	7	7	<1	Spray
	Zerust Axxanol 46-BIO (candidate)	9	7	<1	Spray
	Bio-Medium Preservative Lubricant (candidate)	6	3	<1	Spray
	Fluid Film (candidate)	10	10	10	Brush
	WRL (candidate)	12	12	12	Spray
Grease or Wax Film	VpCI 368 (Control)	10	5	3	Brush
	Ardrox AV-30 (for comparison)	6	3	2	Brush
	Nox-Rust 3100 (for comparison)	6	5	5	Brush
	Bio-Acid Fume Rust Preventative Fluids (candidate)	7	3.5	<1	Spray
	EcoLine Heavy Duty Grease (candidate)	14	12	12	Brush

For the CPCs sprayed on the metal panels, some CPCs exhibited poor wettability. These details are noted in Table 8, and a photograph corresponding to poor wettability is shown in Figure 1.

Figure 1. Example of poor wettability of a CPC on a metal surface.

6.1.2 Viscosity

<u>Test description</u>

Viscosity (*n*) is a measure of the resistance of a fluid which is being deformed by either shear stress or tensile stress. The concept is better understood by contemplating the thickness (or internal friction) exhibited by a liquid. Water in general can be thought of as being a relatively thin liquid that flows easily (low value of *n*). Maple syrup on the other hand is thicker or more viscous and does not flow as readily (high value of *n*).

ASTM D445, Standard Test Method for Kinematic Viscosity of Transparent and Opaque Liquids, specifies a procedure for the determination of kinematic viscosity, n, by measuring the time for a volume of liquid that flows through a calibrated glass capillary viscometer. For this report, the rate of flow was measured at 40°C and 100°C.

A photograph of the test apparatus that was used to provide the data is shown in Figure 2. Initially, the viscometer is charged with the liquid (CPC) of interest and immersed in a heated water bath. After the apparatus is brought to temperature, the rubber stopper on the top of the tube is removed to allow the CPC to flow through the viscometer under the force of gravity. The time required for the liquid to flow through the viscometer is recorded, and based upon the calibration constant provided by the manufacturer, the kinematic viscosity is calculated via the following equation.

$$n = C*t$$

n = Kinematic Viscosity (mm^2/s)
C = Calibration Constant of the Viscometer (mm^2/s^2)
t = time to flow through viscometer (s)

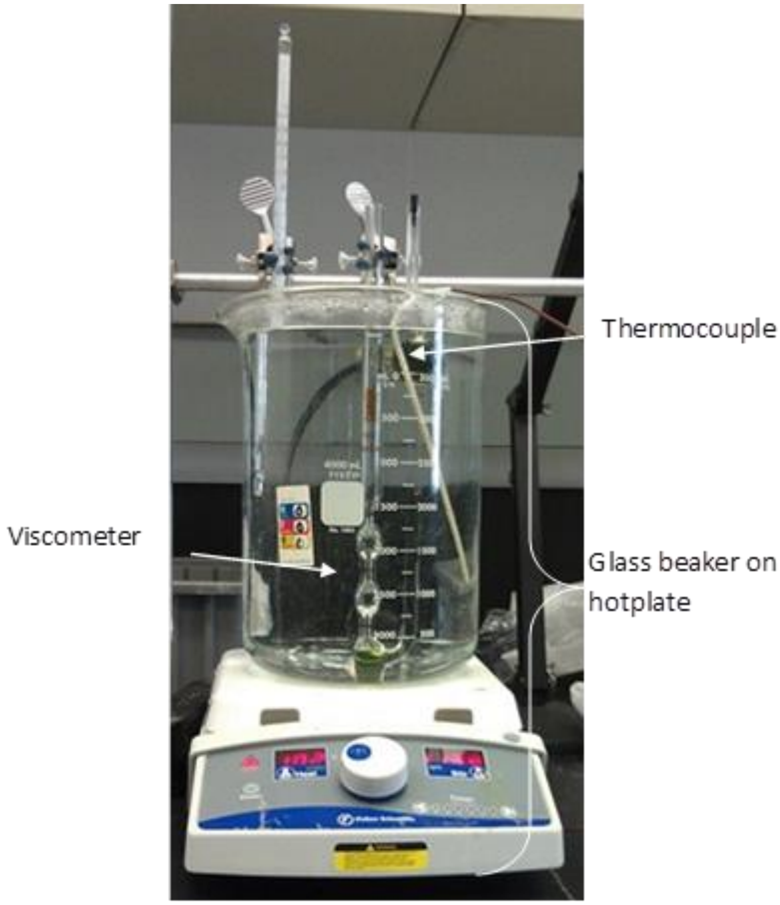


Figure 2. Experimental apparatus for viscosity measurement

Rationale

CPCs are used to protect surfaces, often in crevices which are not easily accessible for corrosion control maintenance. The viscosity of a CPC is an important characteristic since it is inversely proportional to its spreading rate, or the rate in which a liquid wicks into an occluded site.[39,40,41]

Methodology Table

---

[39] J. C. Berg. Wettability. (New York, NY: Marcel Dekker, 1993).

[40] M. Schrader, G. Loeb, Modem Approaches to Wettability~Theory and Application, (New York, NY: Plenum Press,1992).

[41] Kendra T. Price* and James F. Dante, “CPC Performance in Occluded Sites” Mechanical & Materials Engineering Department Southwest Research Institute

Table 10. Test Methodology - Viscosity

Parameters	Perform measurements in accordance with ASTM D445.
Coupons Per CPC	Not Applicable
Trials Per CPC	Two (2)
Control Coupons Required For Testing	Not Applicable
Acceptance Criteria	Obtain Engineering Value

Unique Equipment or Instrumentation

Cannon-Fenske opaque glass capillary viscometers were used to measure the viscosity of the CPCs at both 40°C and 100°C temperatures.

Data Analysis and Reporting

Both Fluid Film and EcoLine Heavy Duty Grease were not conducive to the viscometers, as they were too thick to flow into the glass capillary tubes. NAVGUARD II could not be measured at 100°C because its constituents were too volatile at the high temperature. The average kinematic viscosity values for each CPC were calculated at each temperature (40°C and 100°C) using two determinations. The results for the kinematic viscosity of each CPC at 40°C and 100°C are shown in Table 11, the higher the number, the more viscous or thicker the sample.

Table 11. Kinematic Viscosity

CPC Type	Average Kinematic Viscosity, 40°C (cSt or mm^2/s)	Standard Deviation Between Trials	Average Kinematic Viscosity, 100°C (cSt or mm^2/s)	Standard Deviation Between Trials
Corrosion X (Control)	35.18	2.478	6.26	0
WD-40 (comparison)	2.98	0.050	1.31	0.019
NAVGUARD II (comparison)	34.43	0.964	**	**
MX4 (comparison)	17.79	0.125	3.82	0.021
EcoLine 3690 (candidate)	31.69	0.043	7.01	0.054
Zerust Axxanol 46-BIO (candidate)	37.74	0.211	8.30	0.028
Bio-Medium Preservative	92.43		17.78	

CPC Type	Average Kinematic Viscosity, 40°C (cSt or mm^2/s)	Standard Deviation Between Trials	Average Kinematic Viscosity, 100°C (cSt or mm^2/s)	Standard Deviation Between Trials
Lubricant (candidate)		0.652		0.121
Fluid Film (candidate)	*	*	*	*
VpCI 368 (Control)	77.68	3.070	5.57	0.038
Ardrox AV-30 (comparison)	104.99	3.180	9.77	0.243
Nox-Rust 310 (comparison)	37.16	0.952	4.90	0.040
Bio-Acid Fume Rust Preventative Fluids (candidate)	40.13	0.802	9.91	0.030
EcoLine Heavy Duty Grease (candidate)	*	*	*	*
WRL Control	29.02	0.336	4.90	0.028

*CPC too viscous to measure at 40°C. ** CPC too volatile to measure at 100°C

6.1.3 Contact Angle/Surface Wettability of CPC

<u>Test description</u>
ASTM D7334, Standard Practice for Surface Wettability of Coatings, Substrates and Pigments by Advancing Contact Angle Measurement, was used to measure the wettability of the CPC on an aluminum surface in ambient conditions. A droplet of CPC was placed, via a syringe, onto a clean aluminum substrate, Type A 3003 H14 with a smooth mill finish, and the corresponding angle of the droplet was measured immediately. A separate sterile 100 µl syringe and needle were used for each fluid. The contact angle instrument used was an AST Products Optima XE, utilizing a precision motor controlled attachment for the syringes allowing precise accurate deposition of known amounts of fluid. For this study, 3 µl in volume of fluid was deposited onto the aluminum surface. Due to the spreading of the hydrophilic fluids, any larger droplet size would have spread out of the visual angle of the camera. The method of deposition was to allow the droplet to form on the end of the syringe, and raise the platform containing the aluminum coupon to meet the droplet. The platform was lowered with the deposited droplet. The image of the droplet was captured within 3 seconds of the droplet deposition. Figure 3 is a pictorial description of how the contact angle was measured.

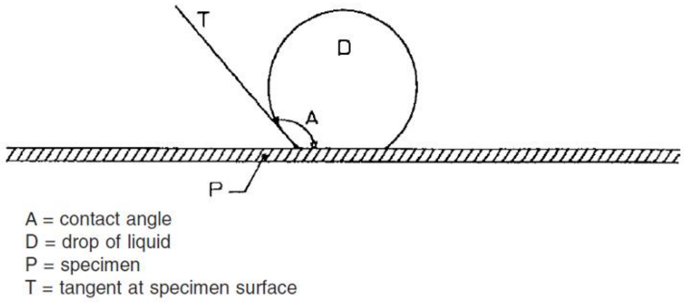


Figure 3. Pictorial description of measuring contact angle from ASTM D7334.

Rationale

CPCs are used to protect surfaces, often in crevices not easily accessible for corrosion control maintenance. Contact angle of a CPC is important because it is directly proportionate to its wetting rate or the rate in which a liquid wicks into an occluded site.[38,39,40]

Methodology Table

Table 12. Test Methodology for Contact Angle/Surface Wettability of CPC

Parameters	Perform measurements in accordance with ASTM D7334.
Coupons Per CPC	1
Trials Per CPC	Twelve (12) drops per CPC
Control Coupons Required For Testing	N/A
Acceptance Criteria	Obtain Engineering Value

Unique Equipment or Instrumentation

A goniometer by use of the Sessile Drop Method.

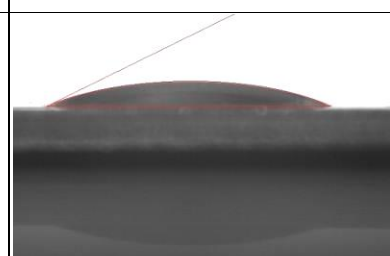
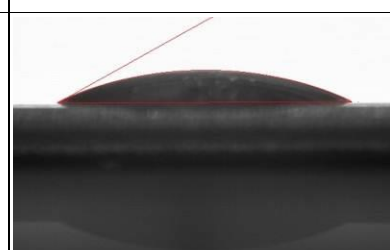
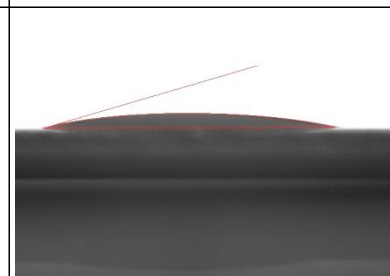
Data Analysis and Reporting

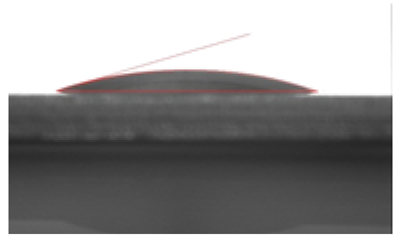
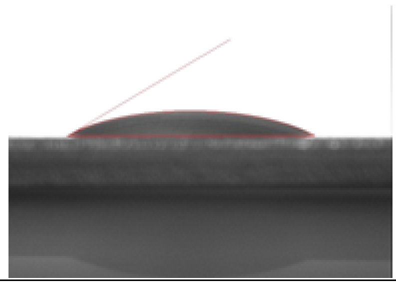
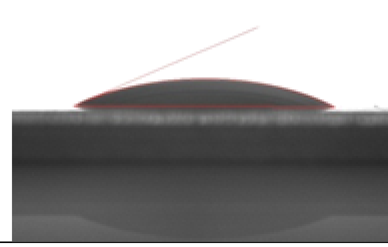
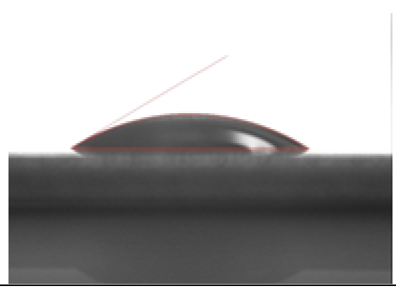
Prior to the actual analysis on the aluminum coupons, the system was calibrated using di-ionized water on cleaned polytetrafluoroethylene (PTFE). Ten drops were deposited and the mean contact angle was measured at 102º. This is consistent with values obtained from the literature for PTFE in the laboratory ambient conditions.

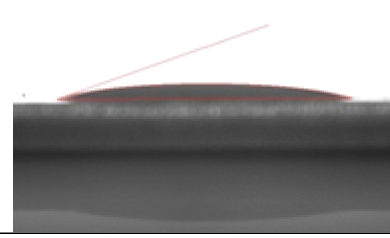
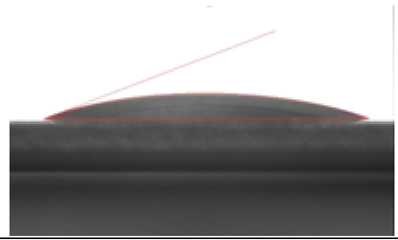
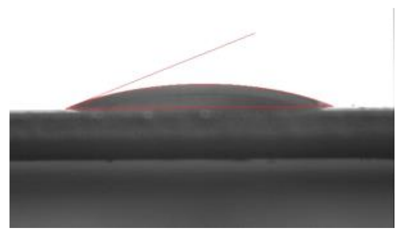
Twelve droplets in total were deposited for each fluid on the aluminum coupons. The angle on each edge of the droplet where it met the substrate was taken, as shown in Figure 3 for the left hand side of the droplet, and the average of the two angles was recorded. From the data, the two outliers were discarded and the mean of the remaining ten was taken. The results are presented in Table 13. For the WD-40, the fluid spread as soon as it was deposited on the surface, not allowing any contact angle to be determined. This fluid can be considered super-hydrophilic, which by definition is any fluid with a contact angle of less than 10º. Fluid Film and EcoLine Heavy Duty Grease were too viscous to be used in the syringe and so no data was obtained.

From the data in Table 13, it was observed that all fluids tested can be considered hydrophilic, that is by definition is any fluid with a contact angle of less than 90º. However, what is not shown in this data, but was observed in the experiments, was that for some of the fluids, they continued to spread until they had completely wetted the surface with no detectable contact angle after the image was captured. In these cases, the wetting is a factor of time. This factor is a nature of the wetting mechanism and surface topography. The aluminum coupons were relatively smooth, and from the same batch so any differences in surface roughness between the coupons can be considered negligible. Typically clean metal surfaces covered with just a native oxide layer tend to have a high energy. It is well known that low energy liquids spread rapidly on high energy surfaces, so the rapid spreading of some of the fluids after deposition is due to the differences in the surface tension components (dispersive vs. polar, hydrogen bonding, acid-base contributions) of the different fluids.

Table 13. Contact Angle of CPC Liquids on Aluminum

CPC Type	Contact Angle (°) mean	Std Dev	
Corrosion X (Control)	23.19	1.72	
WD-40 (comparison)	< 5 wetted easily	n/a	
NAVGUARD II (comparison)	25.05	1.36	
MX4 (comparison)	15.77	1.98	

CPC Type	Contact Angle (°) mean	Std Dev	
EcoLine 3690 (candidate)	21.49	2.37	
Zerust Axxanol 46-BIO (candidate)	25.34	1.90	
Bio-Medium Preservative Lubricant (candidate)	26.66	1.08	
Fluid Film (candidate)	Too viscous	n/a	
VpCI 368 (Control)	23.32	4.21	
Ardrox AV-30 (comparison)	32.43	1.17	

CPC Type	Contact Angle (°) mean	Std Dev	
Nox-Rust 310 (comparison)	17.72	1.76	
Bio-Acid Fume Rust Preventative Fluids (candidate)	21.26	1.20	
EcoLine Heavy Duty Grease (candidate)	Too viscous	n/a	
WRL Control	20.58	1.64	

6.1.4 Contact Angle/Hydrophobicity of CPC-treated Substrates

<u>Test description</u>
ASTM D7334, Standard Practice for Surface Wettability of Coatings, Substrates and Pigments by Advancing Contact Angle Measurement, was used to measure the hydrophobicity of the CPC-coated aluminum substrate. In this case, the angle of contact was measured when a drop of water was applied to a CPC-treated surface.   The testing was completed on aluminum Type A 3003 H14 coupons that were coated with CPCs and allowed to cure for 72 hours. The contact angle instrument used was an AST Products Optima XE, utilizing a precision motor controlled attachment for the syringes allowing precise accurate deposition of known amounts of fluid (Figure 4). For this study, 3 µl in volume of deionized (DI) water was deposited onto the coupons. Due to the spreading of the hydrophilic fluids, any larger droplet size would have spread out of the visual angle of the camera. The method of deposition was to allow the droplet to form on the end of the syringe, and raise the platform containing the aluminum coupon to meet the droplet. The platform was lowered with the deposited droplet. The image of the droplet was captured within 3 seconds of the droplet deposition.

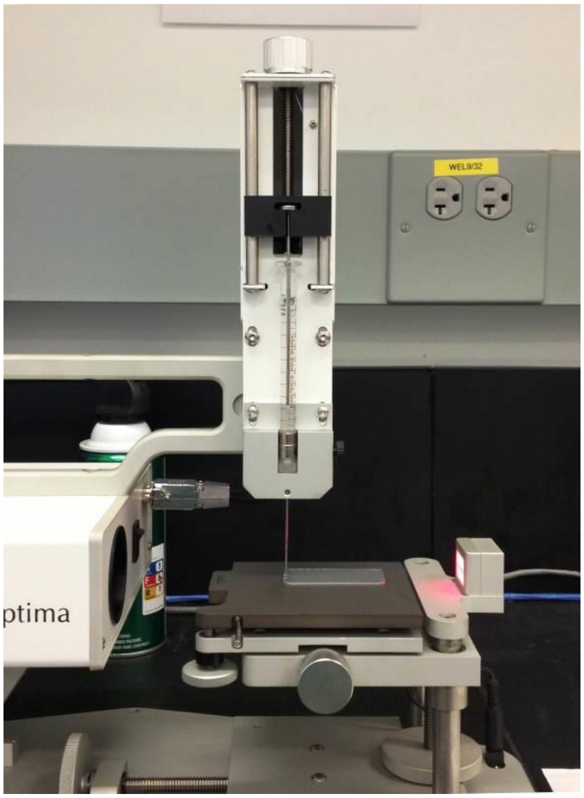


Figure 4. Sessile drop contact angle apparatus

Rationale

CPCs are used to protect surfaces not boldly exposed and often in crevices not easily accessible for corrosion control maintenance. The contact angle of a CPC is important because it will determine the degree of hydrophobicity of the CPC film as cured on the substrate surface.

Methodology Table

Table 14. Test Methodology for Contact Angle/Hydrophobicity of CPC-treated Substrates

Parameters	Perform measurements in accordance with ASTM D7334.
Coupons Per CPC	One (1)
Trials Per CPC	Ten (10) after initial curing
Control Coupons Required For Testing	N/A
Acceptance Criteria	Obtain Engineering Value

Unique Equipment or Instrumentation

A goniometer by use of the Sessile Drop Method.

Data Analysis and Reporting

Prior to the actual analysis on the aluminum coupons, the system was calibrated using DI water on cleaned polytetrafluoroethylene (PTFE). Ten drops were deposited and the mean contact angle was measured at 102º. This is consistent with values obtained from the literature for PTFE in the laboratory ambient conditions.

Twelve droplets of deionized water in total were deposited for CPC coated coupons. The angle on each edge of the droplet where it met the substrate was taken, as shown in Figure 5 for the left hand side of the droplet, and the average of the two angles was recorded. From the data, the two outliers were discarded and the mean of the remaining ten was taken.

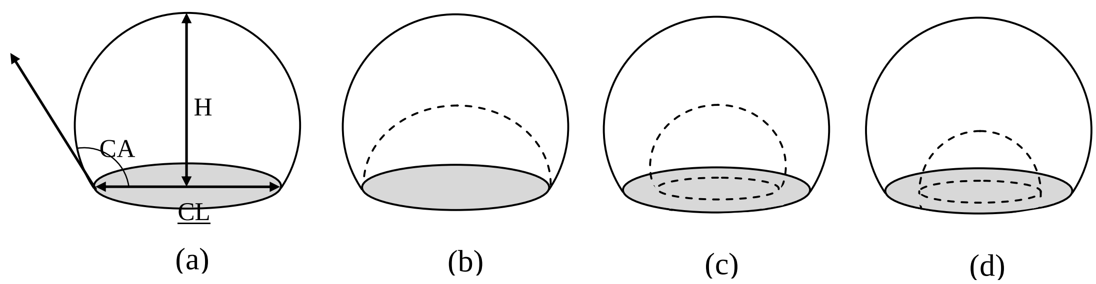


Figure 5. The three modes of evaporation for water droplets placed on hydrophobic/philic substrates, (a) droplet as placed, (b) Constant Contact Line (CCL), (c) Constant Contact Angle (CCA), and (d), Mixed Mode (MM).

The results are shown in Figure 6. From this data it was observed that there was a relatively small standard deviation (SD) for each sample, indicating a relatively smooth coating for each surface. For all coatings, they showed varying degrees of hydrophilicity, with the Zerust Axxanol 46-BIO having the most spreading and therefore being the most hydrophilic. The WRL showed a contact angle of 86.49° that is on the borderline between hydrophilic and hydrophobic, with 90° being the boundary. The waxes, VpCI 368, Ardrox AV-30, and Nox Rust 3100, all had very high contact angles above 100°. As observed in the previous analysis, although the contact angle was measured within a few seconds of the DI water being deposited onto the surface, it was observed after the measurements that the DI water had continued spreading to various degrees for each coating sample. Therefore an additional analysis was preformed, in which the contact angle for each coating was measured as a function of time after deposition.

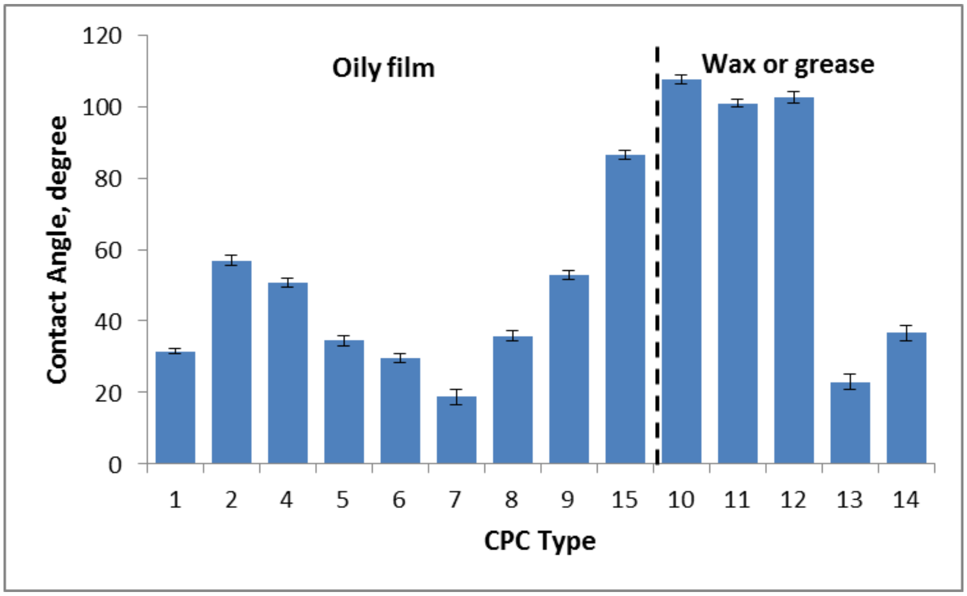


Figure 6. Surface hydrophobicity of the CPC-coated panels.

In this analysis, the contact angle was measured after 30s, 1, 2, 3, 5, 10, 15, 20, 25, and 30 minutes. The results are presented in Figure 7 for the oils and Figure 8 for the waxes and greases. From this, it can be observed that for some coatings there is a significant drop in the contact angle indicating quick wetting within the first couple of minutes (Zerust Axxanol-46 and Bio-Medium Preservative Lubricant), while others slowly spread. For the Corrosion X and Navguard Type II, the contact angle actually increased slightly at 30 mins, however, at this point the droplet was considerably smaller due to evaporation and so this was the maximum time monitored. For the waxes, the surface coatings remained hydrophobic initially, but the water began to spread as a function of time until the droplet actually began to evaporate. The greases, Bio-Acid Fume Rust Preventative and EcoLine Heavy Duty Grease, did not maintain a stable hydrophobic surface as a function of time.

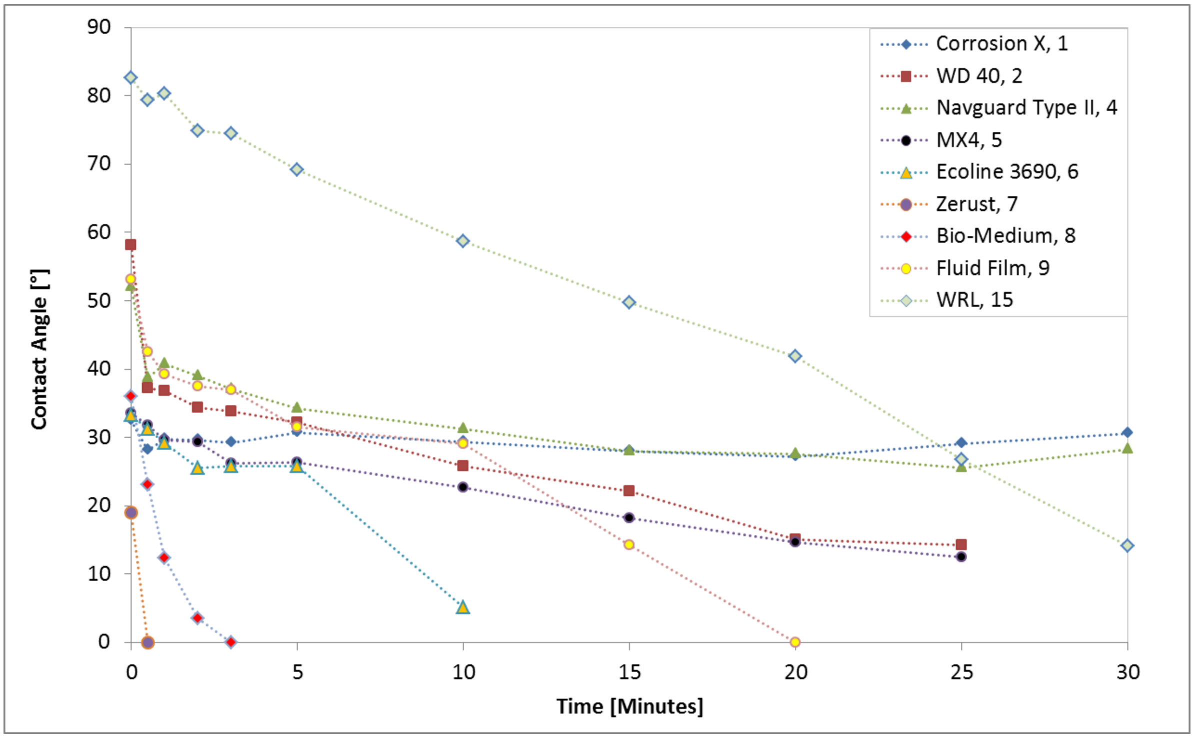


Figure 7. Hydrophobicity of the oily film CPC-coated panels as a function of time.

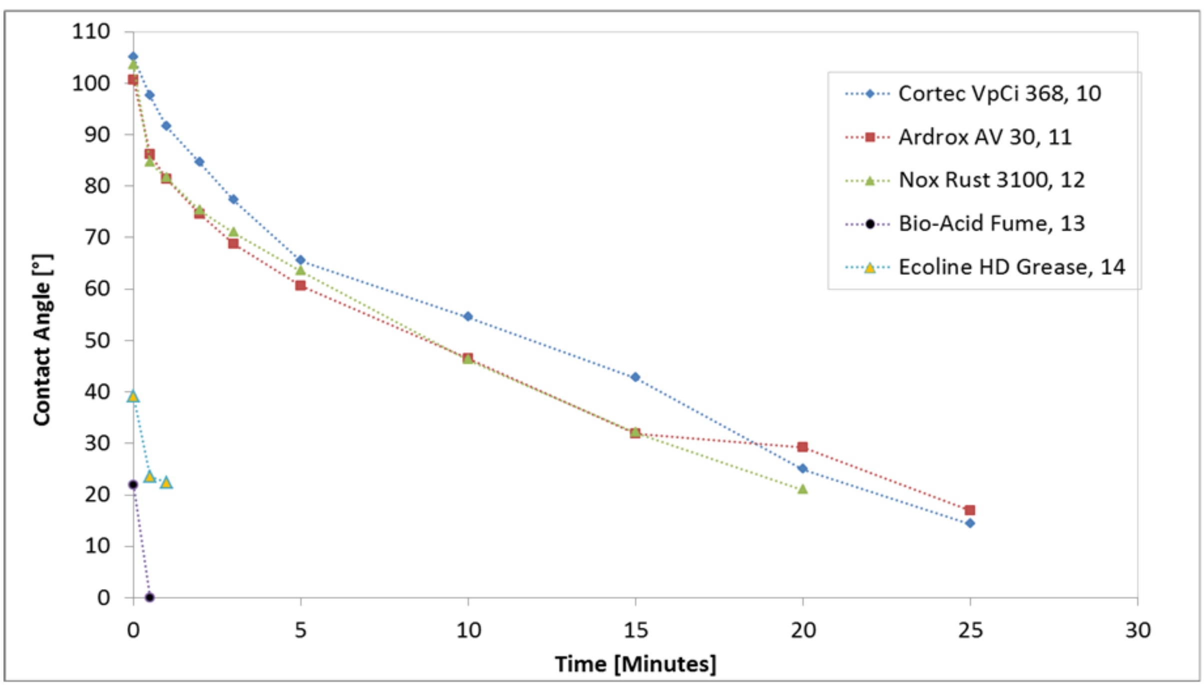


Figure 8. Hydrophobicity of the wax and grease CPC-coated panels as a function of time.

Of all the oily CPC coatings, WRL (CPC 15) had the highest initial contact angle, followed by a nearly linear drop off of contact angle. For evaporation of a water droplet on a surface, the three dimensions of interest are the contact angle (CA), the contact line (CL), and the height of the droplet (H), as shown in Figure 5 (a). There are three modes of water droplet evaporation, as shown in Figure 5. These are the Constant Contact Line (CCL) mode, the Constant Contact Angle (CCA) mode and the Mixed Mode (MM). The presence of a specific mode of evaporation on the solid surface is directly associated with the surface geometry and surface chemistry of the sample in addition to the type of associated wetting regime. Theoretically, for smooth solid substrates, the water droplets should retain the initial CA during the entire evaporation process. Experimentally it has been reported that the CCL mode is the dominant characteristic of the water droplet evaporation process over smooth hydrophilic surfaces, while the CCA mode is dominant for smooth hydrophobic surfaces. A hydrophobic surface is considered less "sticky", and so the CL will reduce as the water droplet evaporates. For a hydrophilic surface the "stickiness" keeps the CL constant, which results in a reduction in the CA during evaporation. Water droplet evaporation on rough surfaces undergoes various modes with different time durations due to changes in the wetting regime. For rough surfaces, the two wetting regimes are known as the Wenzel state and the Cassie state.
The wetting property of rough surfaces in terms of apparent contact angle (ACA) was first described by Wenzel. When the increase in surface area at the interface of liquid/solid due to surface roughness is incorporated, the Young model becomes the following:

$$\cos\Theta^* = r\cos\Theta \quad \cos\theta^* = r\cos\theta$$

(1)

where Θ^* and Θ are the Wenzel ACA and Young CA on the rough and corresponding smooth surface, respectively, and r is the surface roughness, which is the ratio of real area to apparent area with values always greater than one. According to the Wenzel model, which describes the homogenous wetting regime, the surface roughness magnifies the wetting properties of the surface. Hydrophobic surfaces tend to seem more hydrophobic and hydrophilic surfaces tend to seem more hydrophilic. When there is a large degree of surface roughness, which allows air to be trapped at the interface between the grooves, the composite wetting regime of liquid/air/solid will be promoted. Cassie-Baxter further modified the Wenzel model to consider the composite state of both solid fraction and air fraction ($f_a + f_s = 1$) at the interface with water droplets.

More generally, the ACA predicted simply by the Cassie model is the combination of ACAs for different surfaces related to their fraction in contact with a liquid. In the case of a homogenous solid material and air at the interface, while air possesses negligible surface tension having a Young CA of 180^0 with water, the Cassie model can be simplified as in the following:

$$\cos\Theta^* = r_s f_s \cos\Theta - f_a \qquad \cos\theta^* = r_s f_s \cos\theta - f_a \tag{2}$$

The Cassie model is a more general model that can be used to predict entire wetting regimes from low extreme to high extreme, whereas the Wenzel model can predict only moderate homogenous wetting regimes between the two extremes. From the Cassie model, it can be noticed that a reduction in the solid fraction and an increase in the air fraction would enhance the water repellency of a surface regardless of whether the surface is hydrophobic or hydrophilic.

In the study of wetting of surfaces, it is vital to be able to predict the switching point or the borderline between the two states, beyond which the composite state of air and solid might be adopted by the texture, shown in Figure 9. The composite state can be maintained by designing surface geometries that favor water bridging over their tips with air pockets trapped in between the geometries at the interface of solid/liquid. The critical contact angle, which is the function of both surface roughness and solid contact area, should be considered. It can be deduced by equating the equation (1) (Wenzel model) and equation (2) (Cassie model) for Θ^{*} θ^{*} as follows:

$$\cos\Theta_c=(f_s-1)/(r-f_s)\cos\theta_c = \frac{(f_s-1)}{(r-f_s)}$$

(3)

For $(\Theta^{*} > \Theta_c)(\theta^{*} > \theta_c)$ the Cassie state and for $(\Theta^{*} < \Theta_c)(\theta^{*} > \theta_c)$ $(\theta^{*} < \theta_c)$ the Wenzel state, would be favorable by the surface.

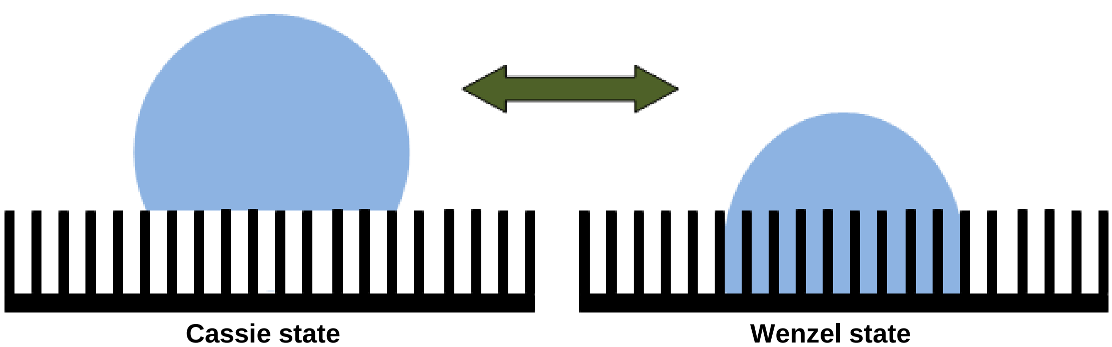


Figure 9. The transition from Cassie to Wenzel state and vice versa.

The CA, CL, and H for the DI water droplets on the WRL coating were monitored and are plotted as normalized functions Figure 10 and shown in Figure 11, and. The CA and H show a linear decrease with time, but the CL remained constant for 15 minutes, where a drop-off was observed. This behavior indicated a hydrophilic surface, even though an initial high contact angle was measured, due to the lack of wetting. After 20 minutes, evaporation had now shrunk the droplet such that the drop in the CL and does not necessarily mean a transition in the regime from a Cassie state to the Wenzel state.

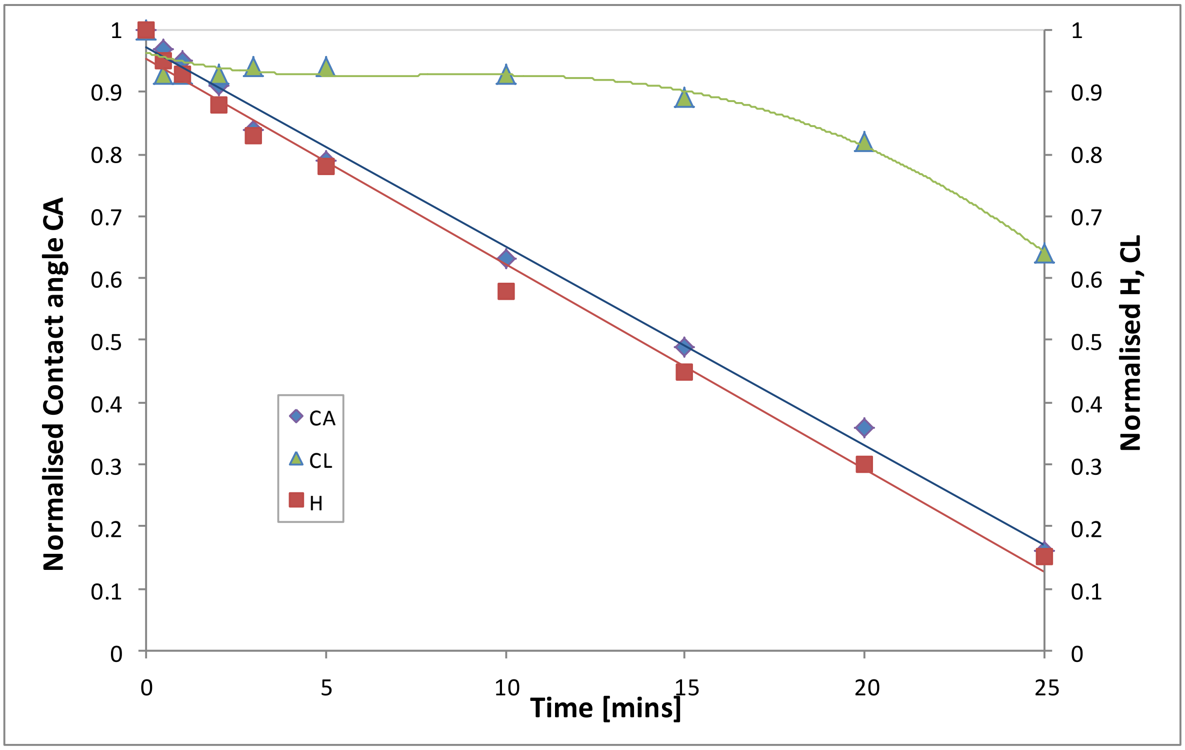


Figure 10. Progression of contact angle as a function of time for CPC type WRL

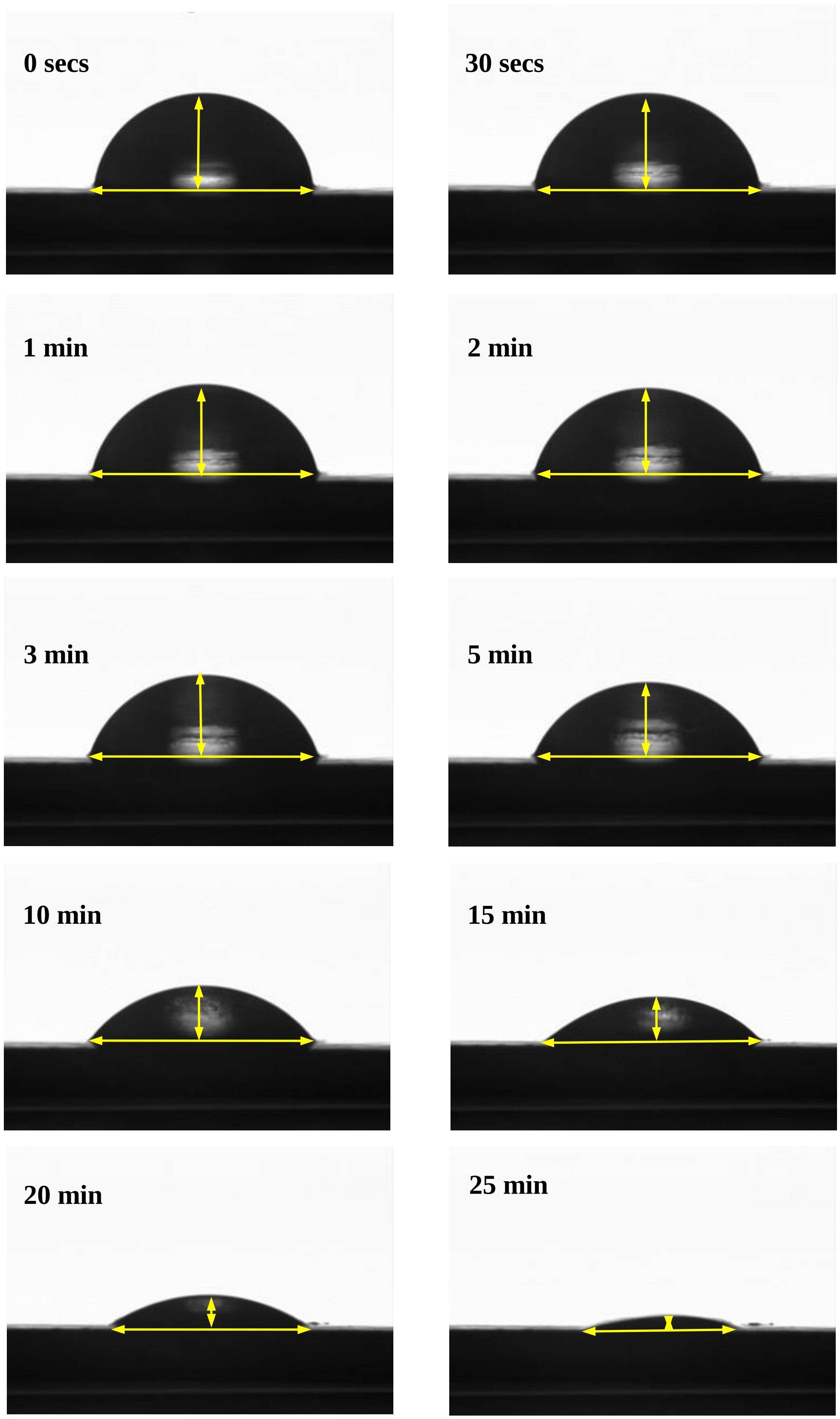


Figure 11. Deionized water on WRL as a function of time

6.1.5 Functional Penetration

Test description

This test was performed to provide visual evidence of a CPC's ability to penetrate a crevice or faying surface. MIL-PRF-81309F, Performance Specification: Corrosion Preventive Compounds, Water Displacing, Ultra-Thin Film, Section 4.6.13, was used to measure the functional penetration of the CPC. The test method used lap-joint specimens, shown in Figure 12, with two strips of vacuum bag sealing tape on each side of one lap joint. This configuration created a barrier so that the CPC could not travel beyond the edge of the sample panel. The panels were elevated on one end creating a 10 degree slope. A 1 ml by volume amount of CPC was poured on the surface and allowed to seep in the crevice over a 24 hour period of time at room temperature. The sandwich panels were separated and the area of penetration was calculated using a grid system. The grid was created from a transparency, where measurements were made in ¼" x ¼" sections across the crevice area. The number of squares covered by the CPC was divided by the total number of squares to get a percent penetration.

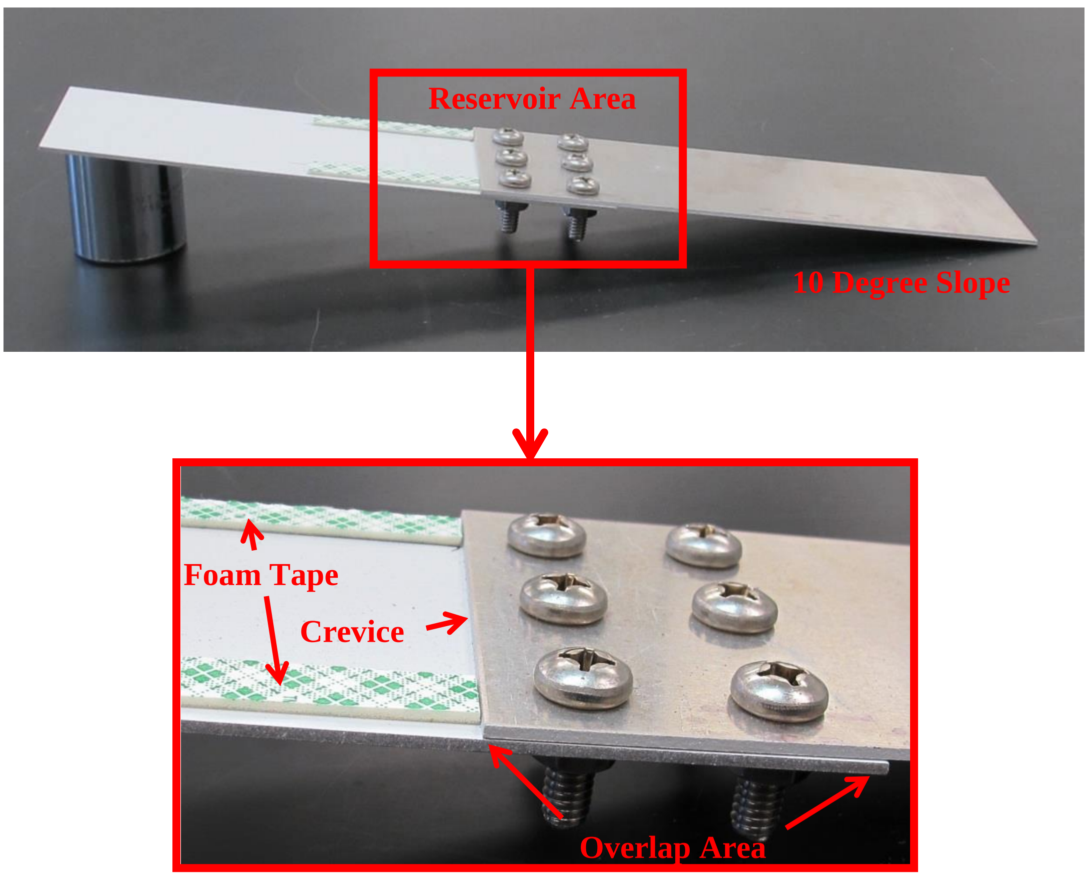


Figure 12. Test Configuration for Functional Penetration.

Rationale

CPCs are used to protect surfaces not boldly exposed and often in crevices not easily accessible for corrosion control maintenance. This test provided data used to correlate the theoretical calculated wetting rate found in previous physical property tests.

Methodology Table

Table 15. Test Methodology for Functional Penetration Test

Parameters	Perform measurements in accordance with MIL-PRF-81309F, Section 4.6.13.
Coupons Per CPC	Two (2) sandwich coupons, Al 7075-T6 only
Trials Per CPC	One (1)
Control Coupons Required For Testing	N/A
Acceptance Criteria	No panel faying surface area to be less than 80 percent wetted in 24 hours. Average of two panels to be 85 percent or better, wetted in 24 hours.

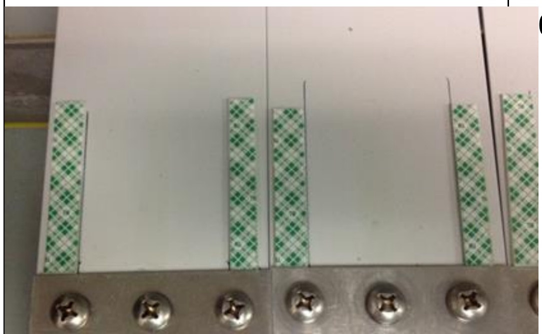
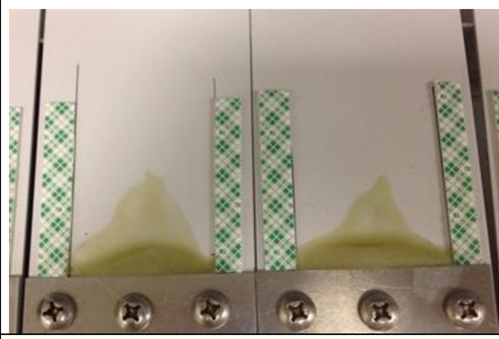
Unique Equipment or Instrumentation

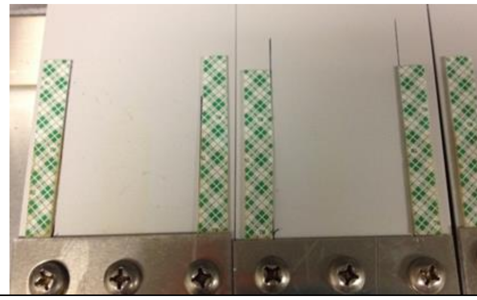
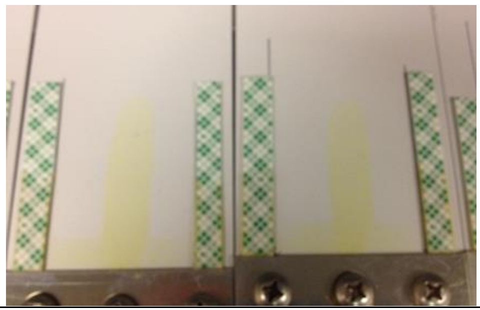
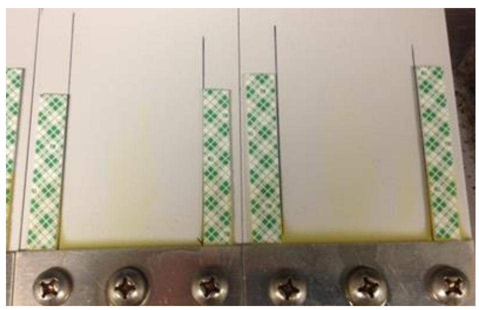
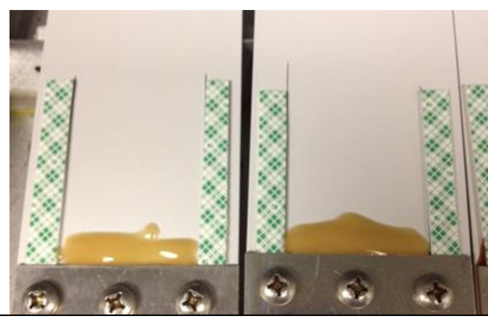
None

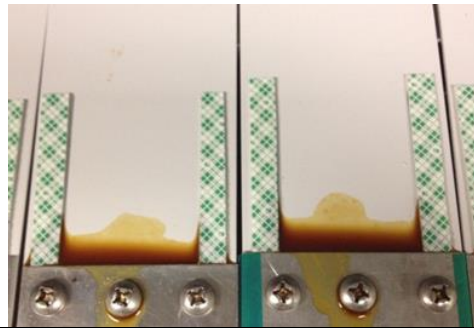
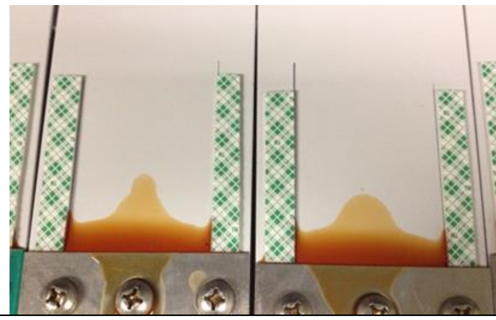
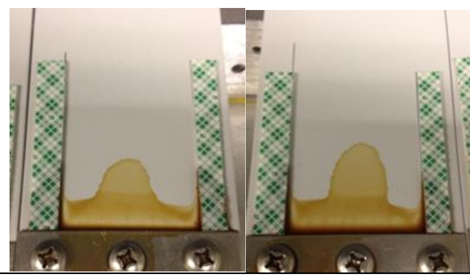
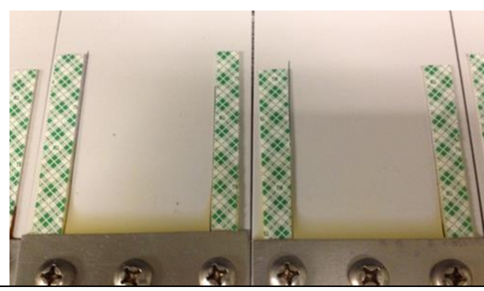
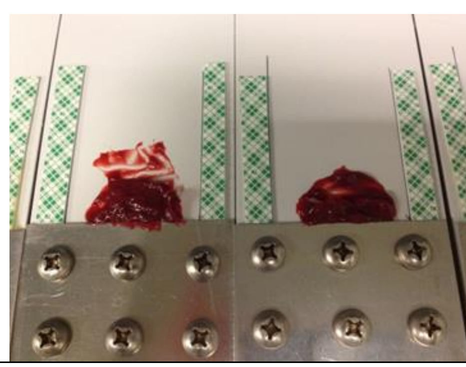
Data Analysis and Reporting

Table 15 shows photographs of the initial CPC deposition along the crevice, as well as the reported percent penetration of each CPC into the crevice after a 24-hour period. Interestingly, even the seemingly thicker and more static CPCs did penetrate into the crevice. For example, the thickest CPC, EcoLine Heavy Duty Grease, eventually penetrated enough to result in 30 percent penetration across the crevice area. VpCI 368 penetrated at 28 percent. The remaining CPCs penetrated at or near 100 percent.

Table 16. Functional Penetration Test Results

CPC Type	Photographs of Initial Penetration	Percent Penetration
Corrosion X (Control)		00
WD-40 (for comparison)		00
NAVGUARD II (for comparison)		100
MX4 (for comparison)		100

EcoLine 3690 (candidate)		100
Zerust Axxanol 46-BIO (candidate)		100
Bio-Medium Preservative Lubricant (candidate)		100
Fluid Film (candidate)		100
WRL (candidate)		100

VpCI 368 (Control)		28
Ardrox AV-30 (for comparison)		100
Nox-Rust 3100 (for comparison)		79
Bio-Acid Fume Rust Preventative Fluids (candidate)		96
EcoLine Heavy Duty Grease (candidate)		30

6.1.6 Wire Compatibility

Test description

This test was designed to test a CPC's compatibility with different types of wire insulation. Two wire types that were determined to be most relevant to future use at NASA were PTFE and ETFE insulated wires. These wires are currently designed for use on Orion-based flight hardware. The types were specifically MIL-DTL-22859/87 and MIL-DTL-22759/16. Three wires, measured 18 inches each, were immersed in a CPC for 14 days. The wires were cleaned with deionized water and allowed to completely dry. Afterward, the wires were wrapped around a 0.125" mandrel to determine if the CPCs degraded the insulating material of the wire to induce cracking. Each wire was then soaked in 5 percent by weight sodium chloride solution for four hours, and then subjected to a one-minute dielectric test of 2500 volts using a Keithley 248 High Voltage Supply. A resistance measurement of 500 Ohms or higher indicates failure, and any values less than 500 Ohms indicates degradation. An overload indicates no damage.

Rationale

CPCs are widely used in and around areas that contain electrical and data wiring. It is important to know if the products will deteriorate wire insulation and breakdown their dielectric properties.

Methodology Table

Table 17. Test Methodology for Wire Insulation Compatibility

Parameters	Perform test in accordance with MIL-PRF-81309F, Section 4.6.4.
Coupons Per CPC	Three (3) coils of wire
Trials Per CPC	One
Control Coupons Required For Testing	One
Acceptance Criteria	No cracking or degradation of insulation following prolonged exposure; No dielectric leakage.

Unique Equipment or Instrumentation

High voltage source.

Data Analysis and Reporting

The CPCs were found to cause no cracking or degradation to the wire types, MIL-DTL-22859/87 and MIL-DTL-22759/16, chosen for this study. Table 18 shows the results.

Table 18. Wire Insulation Compatibility Results

CPC Type	CPC	Cracking	Resistance
	No CPC or immersion	None	*OL/no defects
	Deionized Water	None	*OL/no defects
Soft Film	Corrosion X (Control)	None	*OL/no defects
	WD-40 (for comparison)	None	*OL/no defects
	NAVGUARD II (for comparison)	None	*OL/no defects
	MX4 (for comparison)	None	*OL/no defects
	EcoLine 3690 (candidate)	None	*OL/no defects
	Zerust Axxanol 46-BIO (candidate)	None	*OL/no defects
	Bio-Medium Preservative Lubricant (candidate)	None	*OL/no defects
	Fluid Film (candidate)	None	*OL/no defects
	WRL (candidate)	None	*OL/no defects
Grease or Wax Film	VpCI 368 (Control)	None	*OL/no defects
	Ardrox AV-30 (for comparison)	None	*OL/no defects
	Nox-Rust 3100 (for comparison)	None	*OL/no defects
	Bio-Acid Fume Rust Preventative Fluids (candidate)	None	*OL/no defects
	EcoLine Heavy Duty Grease (candidate)	None	*OL/no defects

*Over Load (OL) equates to no defects.

6.1.7 Removability

Test description

This test determined the ability of a CPC to be easily removed by hand, using typically used solvents. This test was conducted to identify and eliminate those candidate CPCs that are difficult to properly remove under normal maintenance operation conditions. The CPCs were evaluated for removability using mineral spirits. A mineral spirit-soaked lint-free cloth was wiped across each CPC-coated panel surface for four continuous passes. The excess mineral spirits were wiped of the panel using clean lint-free clothes.

Rationale

Knowing that a CPC can be easily removed is an important criteria because in order to perform maintenance duties or to gain access to areas, a CPC will have to be removed.

Methodology Table

Table 19. Test Methodology for Removability

Parameters	Perform test in accordance with MIL-PRF-81309F, Section 4.6.18.
Coupons Per CPC	One (1), One(1) alloy system only
Trials Per CPC	One (1)
Control Coupons Required For Testing	N/A
Acceptance Criteria	Completely removable with mineral spirits

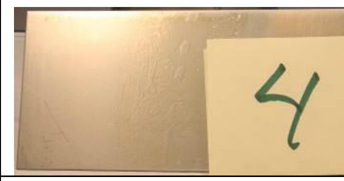
Unique Equipment or Instrumentation

Lint free cloth

Data Analysis and Reporting

All of the CPC types were easily removed; however, VpCI 368, Nox-Rust 3100, and EcoLine Heavy Duty Grease required twice as much effort, but were still considered easily removed. For all but three CPC types, Corrosion X, MX3, and Fluid Film,, a thin film residue remained on the surface after removal. Nox-Rust 3100 had a slightly tacky film residue that remained after the initial film removal. Table 18 shows the CPC removability results.

Table 20. CPC Removability Results

CPC Type	Initial Condition of Film	Final Condition of Bare Metal	Photo Record
Corrosion X Aviation (Control)	Wet and transparent	All removed	
WD-40 (for comparison)	Wet and transparent	Thin film remained	
NAVGUARD II (for comparison)	Tacky and transparent	Thin film remained	
MX3 (for comparison)	Wet and transparent	All removed	
EcoLine 3690 (candidate)	Wet and transparent	Very thin film remained	
Zerust Axxanol 46-BIO (candidate)	Very tacky (almost waxy) and transparent	Very thin film remained	
Bio-Medium Preservative Lubricant (candidate)	Tacky and transparent	Very thin film remained	
Fluid Film (candidate)	Wet and opaque (thick and tan)	All removed	

CPC Type	Initial Condition of Film	Final Condition of Bare Metal	Photo Record
VpCI 368 (Control)	Waxy and brown	Thin film remained	
Ardrox AV-30 (for comparison)	Waxy and brown	Thin film remained	
Nox-Rust 3100 (for comparison)	Waxy with light tack and brown	Thin, tacky film remained	
Bio-Acid Fume Rust Preventative Fluids (candidate)	Very tacky (almost waxy) and transparent	Very thin film remained	
EcoLine Heavy Duty Grease (candidate)	Tacky and opaque (red)	Thin film remained	
WRL (candidate)	Wet and transparent	Thin film remained	

6.2 Accelerated Corrosion Testing

6.2.1 UV Weathering/Cyclic Salt Fog

<u>Test Description</u>

Structures must withstand daily outdoor exposure to sunlight and wet/dry cycles. This procedure documented CPC resistance to accelerated outdoor weather exposure conditions. This test series consisted of cyclic corrosion and UV exposure using alternating periods of exposure in two different cabinets, first a fluorescent UV/condensation cabinet for one week (168 hours), followed by a cycling salt fog/dry cabinet for one week (168 hours), in accordance with ASTM D5894, Standard Practice for Cyclic Salt Fog/UV Exposure of Painted Metal, (Alternating Exposures in a Fog/Dry Cabinet and a UV/Condensation Cabinet). This test was run for six week-long cycles, totaling 1008 hours.

The UV portion of the test consisted of placing the CPC coated panels in a QUV chamber set to run a fluorescent UV/condensation cycle of 4-hours UV with an irradiance of 0.89 $W/(m^2 \cdot nm)$/340 nm at 60°C and 4-h condensation at 50°C, using UVA-340 lamps.

The salt fog portion of the test consisted of placing the test panels in an Autotechnology CCT chamber programmed to run a cycle of 1-h fog at ambient temperature and 1-h dry-off at 35°C. The fog electrolyte was a relatively dilute solution, with 0.05 % sodium chloride and 0.35 % ammonium sulfate.

The panels used for this test were three (3) inches by five (inches) to accommodate the UV weathering chamber.

Rationale

The cyclic corrosion method can be used to accelerate some factors of the atmospheric corrosion conditions.

Test Methodology

Table 21. Test Methodology for Cyclic Corrosion Resistance Test

Parameters	Perform test in accordance with ASTM D5894.
Coupons Per CPC/alloy	Two (2)
Trials Per Coupon	One (1)
Control Coupons Required For Testing	One (1) untreated per alloy.
Acceptance Criteria	Panel condition (per ASTM G1 or ASTM G 46) of candidate CPC rated better than untreated. Performs similar or better to control CPC.

Unique Equipment or Instrumentation
Programmable salt spray (fog) chamber, programmable UV chamber.

Data Analysis and Reporting

After the panels were removed from the cyclic corrosion chamber, they were allowed to dry completely. The CPCs were removed from the panels using a solvent soak and wipe. The panels were cleaned using ASTM G1, Chemical Cleaning Procedures. For the aluminum alloys, an additional ultrasonic cleaning step was taken to loosen corrosion around the pits for more effective pit identification. The carbon steel panels were evaluated for corrosion rate using the mass loss method, where corrosion rate (CR) is calculated using the following equation:

$$CR = (K \times W)/(A \times T \times D)$$

Where:

K = a constant (in this case 8.76×10^4 for mm/y)

T = time of exposure in hours

A = area in cm^2

W = mass loss in grams

D = density in g/cm^3

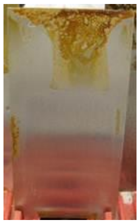
The corrosion rate data, in mm/y, from the carbon steel control coupon shows that the cyclic environment was about 3.6 times more aggressive than the beachside atmospheric exposure environment. The carbon steel corrosion results with and without the CPC coating are shown in Figure 13. The corrosion protection results are generally consistent with the atmospheric corrosion results; however, the general ranking of best to worst performing CPC is different. The data will be compared further in the Conclusions section of this report. For the cyclic results, some of the CPC types, 2(WD-40), 5(MX4), and 15(WRL), did not perform similar to or as well as the control CPC. The best performing CPCs were wax or grease based, and the best oil-based CPCs were two of the environmentally-friendly CPCs, 8(Bio-Medium Rust Preventative) and 9(Fluid Film). An overwhelming factor that affected the performance of the different CPCs is exposure and resistance to UV degradation. The CPC-coated panels were initially exposed to one week's worth of UV light. For many of the CPC types, the films had heavily degraded after the UV exposure. Thus, the coating was greatly compromised before it was exposed to the salt fog chamber. A table showing the UV-exposed vs. the unexposed areas, Table 22, reveals the extreme effect that UV-only exposure had on the CPCs. Week 1 pictures in Table 22 are the UV-only results, while week 6 pictures are after exposure to both UV and the salt fog chamber for alternating weeks. This pictorial comparison was included for reference so that the effects of UV degradation can be visually compared to the corrosion results.

The aluminum alloys, 7075 and 2219, were evaluated for their pitting corrosion behavior. The pit density and pit size were recorded, per ASTM G46. The criteria are shown in Figure 14 for both pit density and size. Pit density calculations were made to determine the number of pits per area viewed at 40X under a microscope, so that the same pit density per ASTM

G46 was maintained but the pits could be more effectively identified. Figure 15 and Figure 16 show the pit density and pit size results for all of the CPCs. The uncoated results for the 7075 and 2219 panels showed that the corrosion susceptibility was equal for both alloys. The CPC types performed differently for each alloy type, though overall the pit density and pit size results indicated sparse pitting at a rating of 2 for the worst case. Many of the CPCs on the 2219 substrate did not result in any pitting due to the fact that the alloy was a clad version instead of the non-clad version used for atmospheric corrosion testing.

Table 22. Cyclic Corrosion Testing Comparison of UV Effects

					
Week 1	Week 6	Week 1	Week 6	Week 1	Week 6
Blank (control)		Corrosion X (comparison) - 1		WD-40 (comparison) - 2	
					
Week 1	Week 6	Week 1	Week 6	Week 1	Week 6
NAVGUARD II (comparison) - 4		MX4 (comparison) - 5		EcoLine 3690 (candidate) - 6	
					
Week 1	Week 6	Week 1	Week 6	Week 1	Week 6
Zerust Axxanol (candidate) - 7		Bio-Medium Pres. (candidate) - 8		Fluid Film (candidate) - 9	
					
Week 1	Week 6	Week 1	Week 6	Week 1	Week 6
WRL (candidate) - 15		VpCI 368 (control) - 10		Ardrox AV-30 (comparison)	

				- 11	
					
Week 1	Week 6	Week 1	Week 6	Week 1	Week 6
NoxRust 3100 (comparison) - 12		Bio-Acid Prev. (candidate) - 13		EcoLine Grease (candidate) - 14	

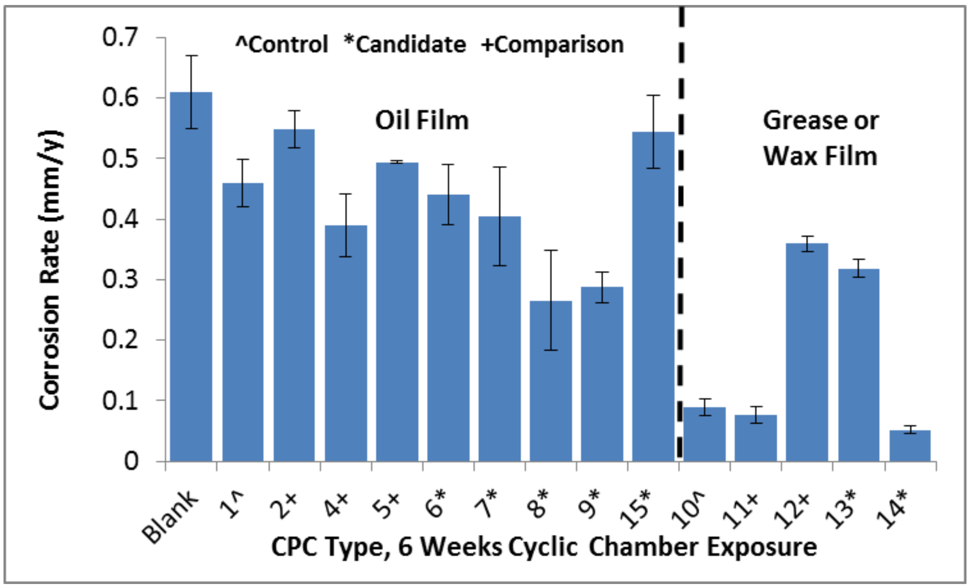


Figure 13. Corrosion rate results of CPC-coated carbon steel panels exposed to the accelerated cyclic chamber for six weeks.

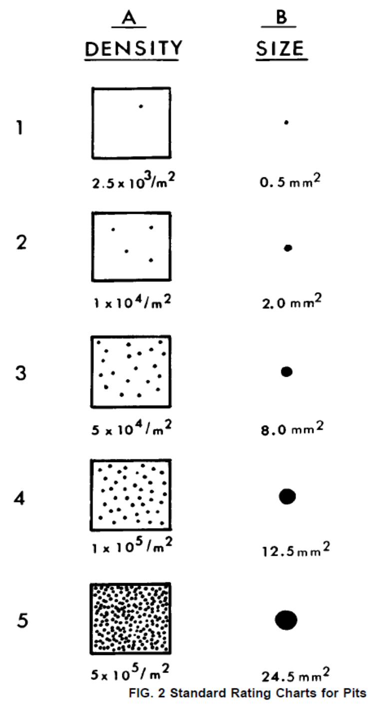


Figure 14. Pitting Corrosion Criteria per ASTM G46

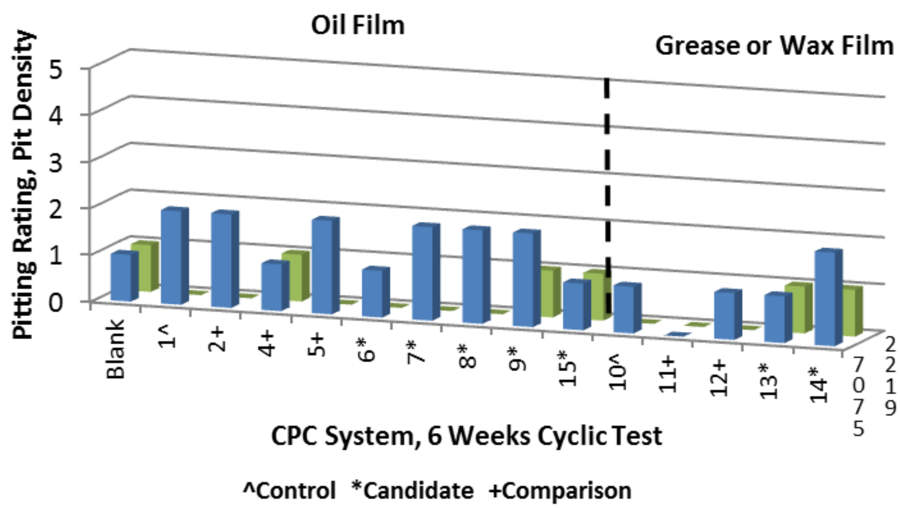


Figure 15. Pit density results of CPC-coated aluminum alloy panels (7075 and 2219 – clad) exposed to the accelerated cyclic chamber for six weeks.

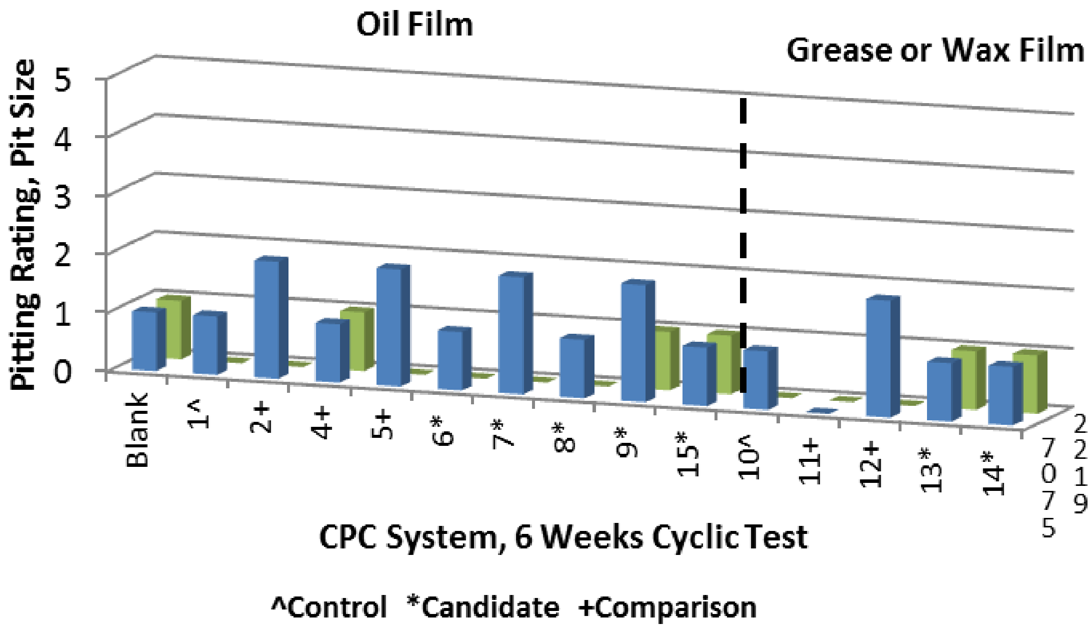


Figure 16. Pit size results of CPC-coated aluminum alloy panels (7075 and 2219 – clad) exposed to the accelerated cyclic chamber for six weeks.

6.3 Atmospheric Corrosion Testing

Racks were created for atmospheric corrosion testing to include panels for the following tests: Long-term Beachside Atmospheric Exposure (7.3.1), Sandwich Corrosion (7.3.2), Crevice Corrosion (7.3.3), Galvanic Corrosion with Fasteners (7.3.4), Wire on Bolt Atmospheric Galvanic Corrosion (7.3.5), and Stress Corrosion Cracking (7.3.6). An example of the rack set-up for the panels is included in Figure 17. After the CPCs were applied to all of the panels on each rack, the racks were held horizontally for 2 hours to allow for curing. After the curing time, the racks were oriented at a 30° angle to the horizon and directly facing the Atlantic Ocean. Photographs of each CPC type after initial exposure are documented below.

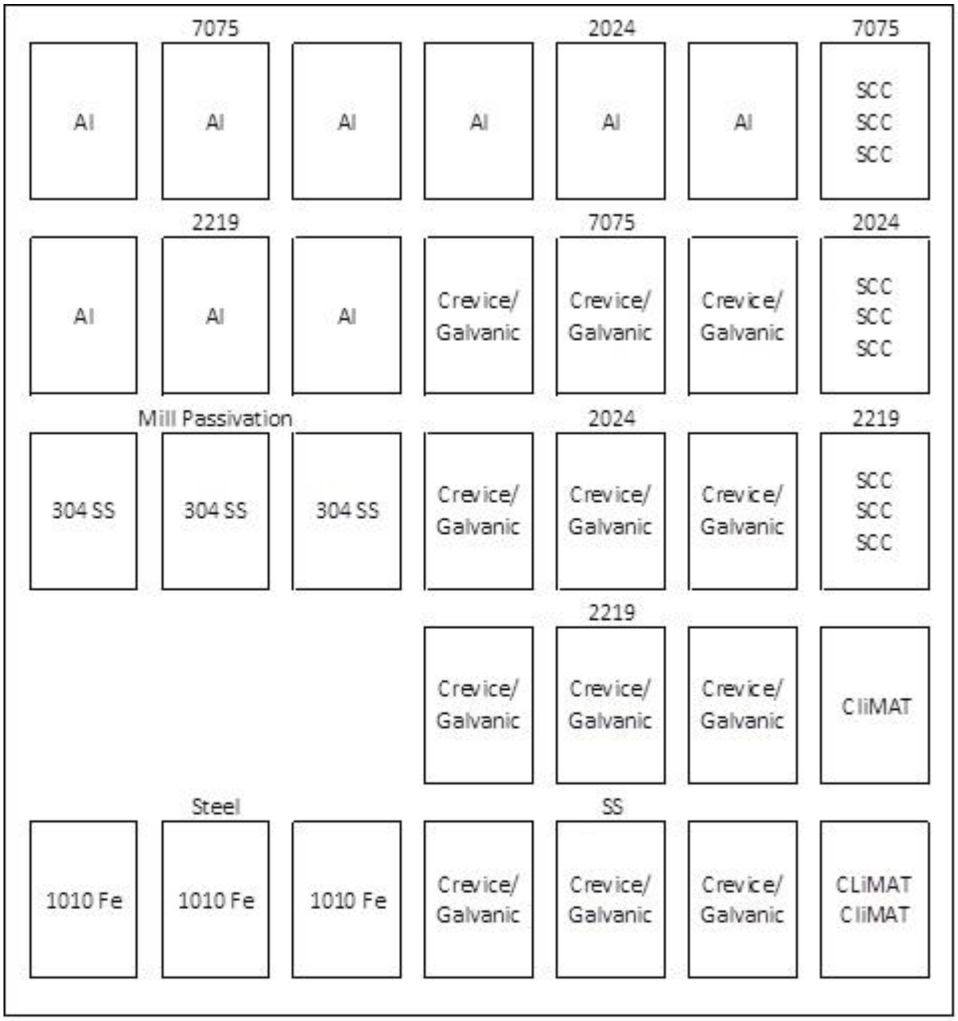


Figure 17. Test panel rack layout for each CPC type.

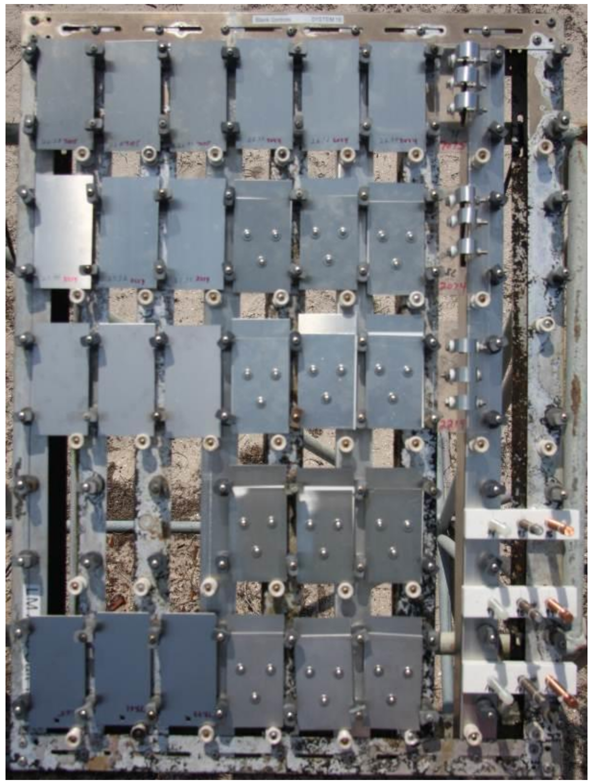


Figure 18. Blank Control

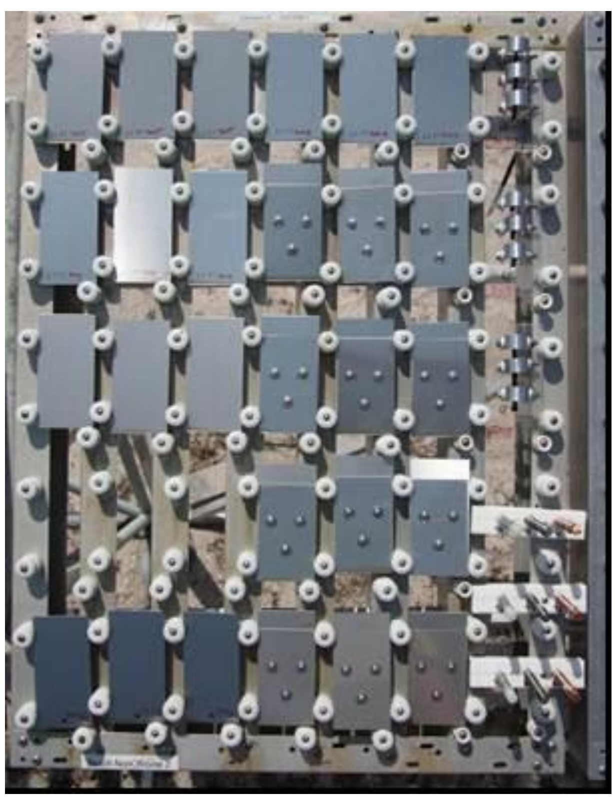


Figure 19. Corrosion X

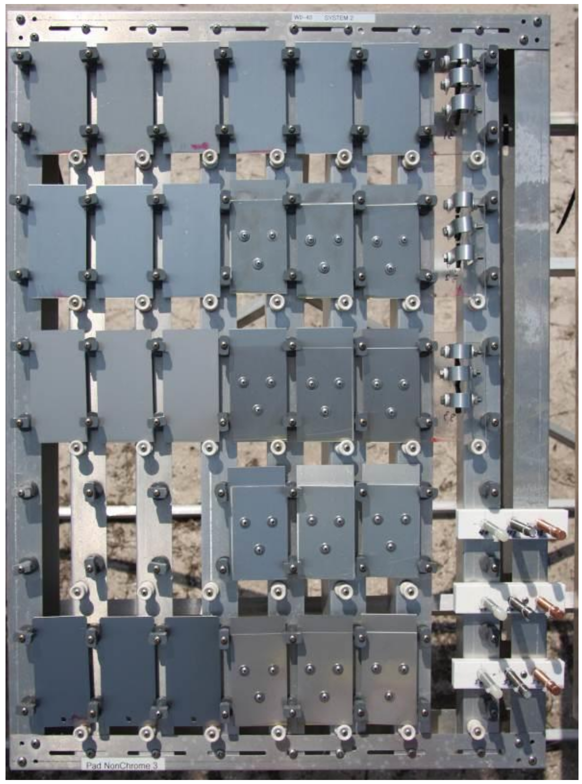


Figure 20. WD-40

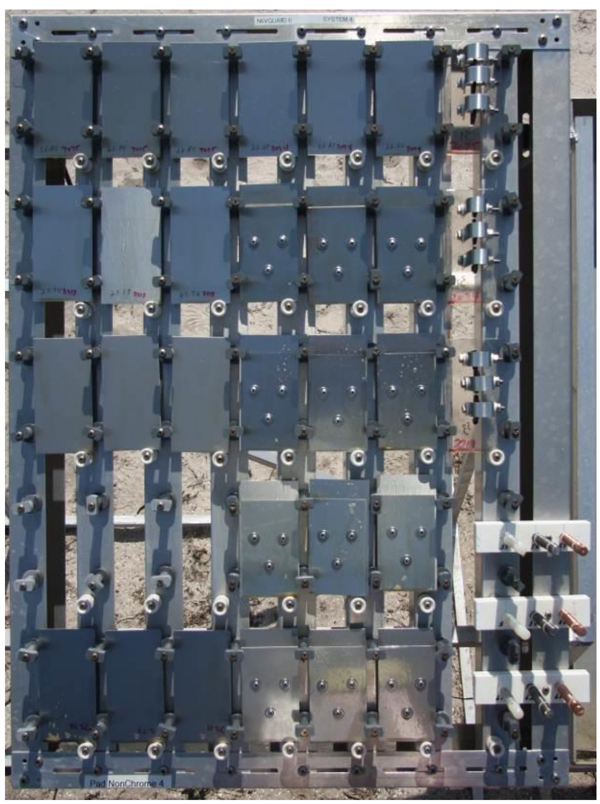


Figure 21. NAVGUARD II

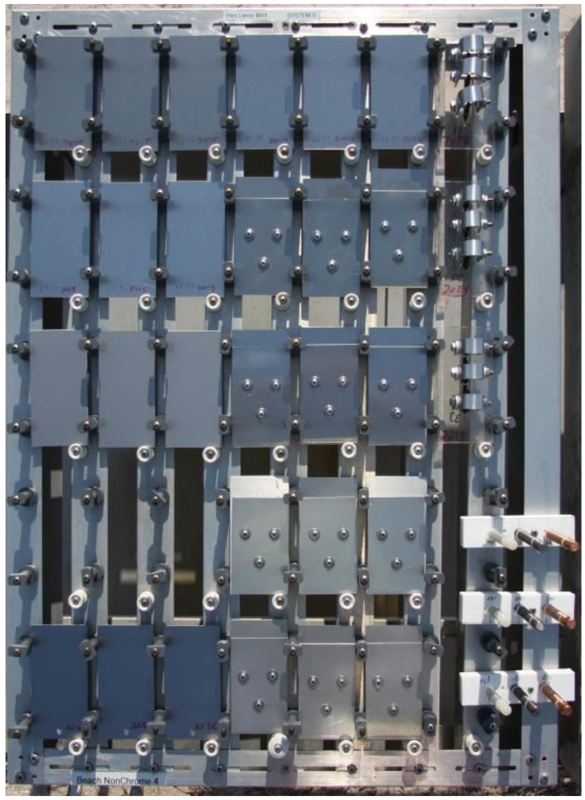


Figure 22. MX4

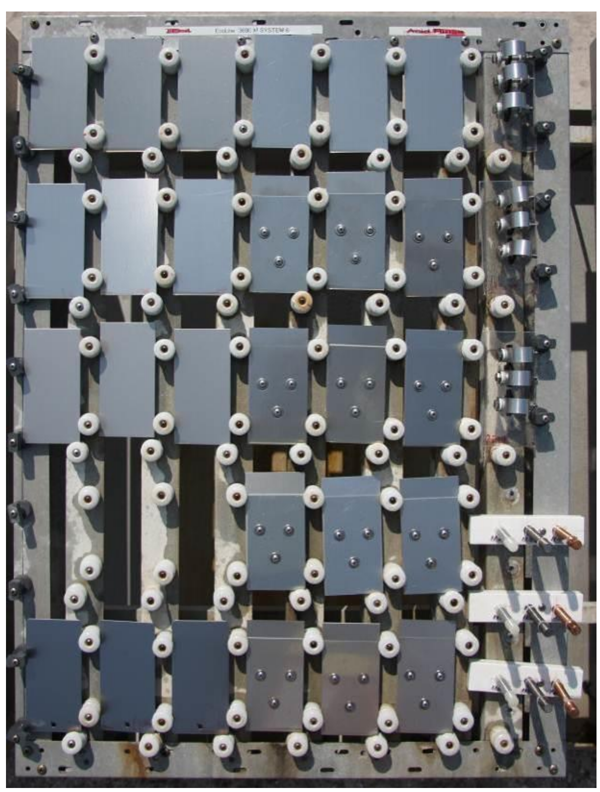


Figure 23. EcoLine 3690

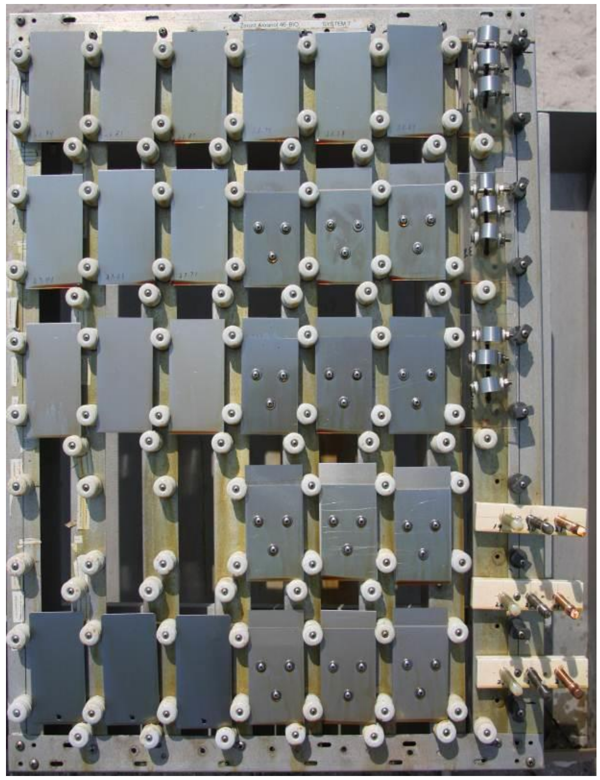


Figure 24. Zerust Axxanol 46-Bio

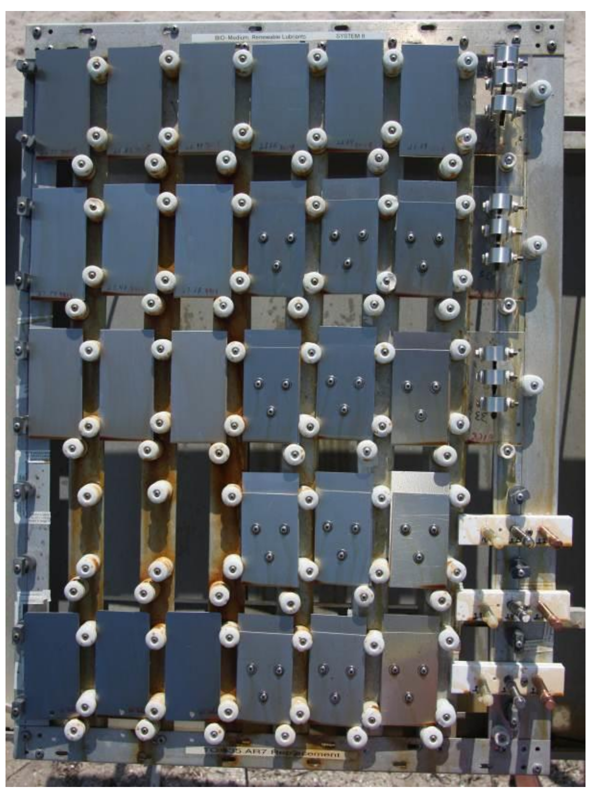


Figure 25. Bio-Medium Preservative Lubricant

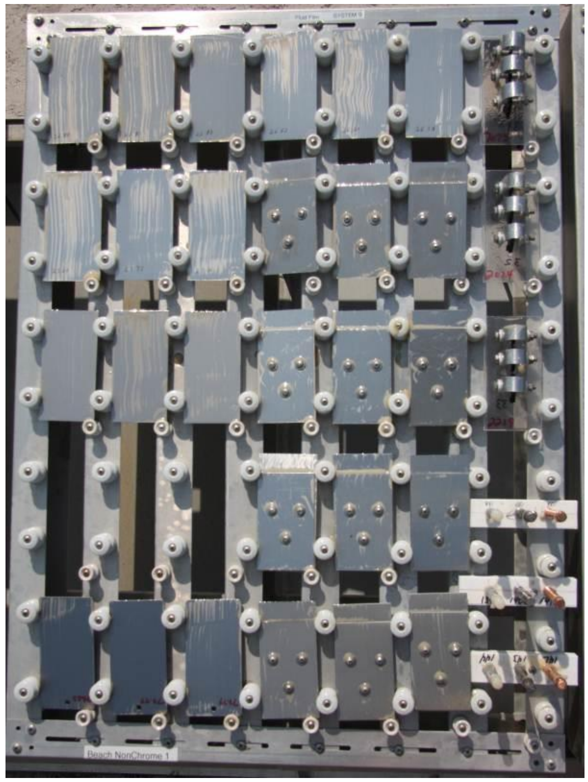


Figure 26. Fluid Film

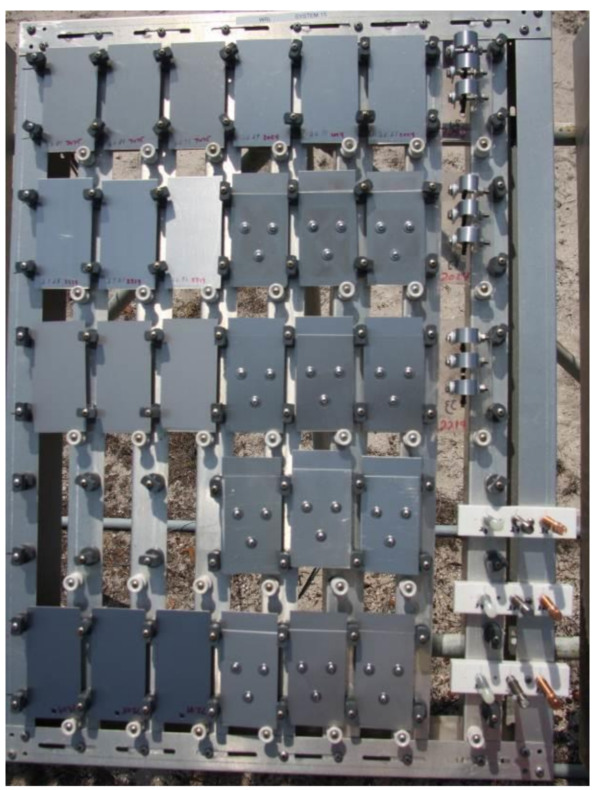


Figure 27. WRL

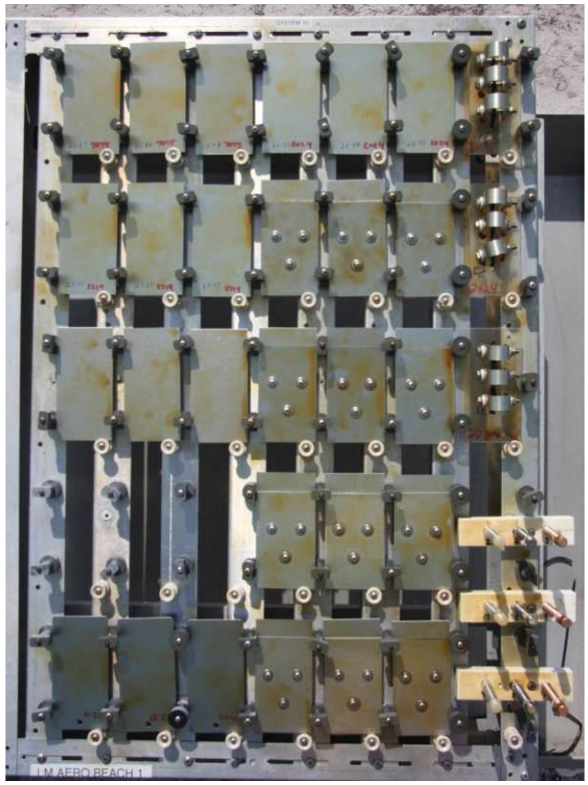


Figure 28. VpCI 368

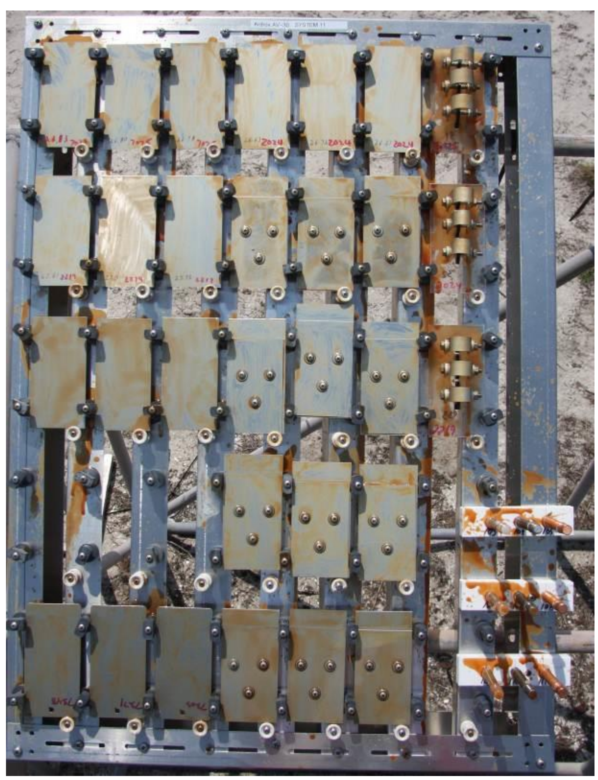


Figure 29. Ardrox AV-30

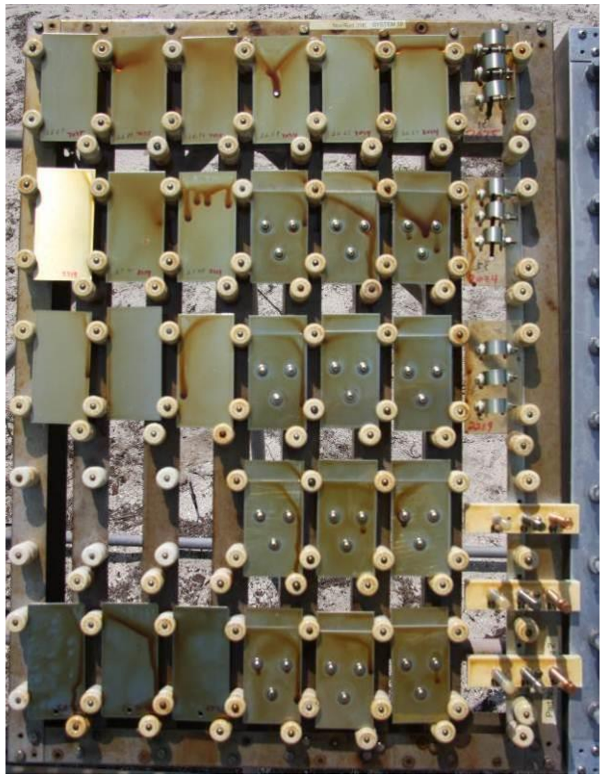


Figure 30. Nox-Rust 3100

Figure 31. Bio-Acid Fume Rust Preventative

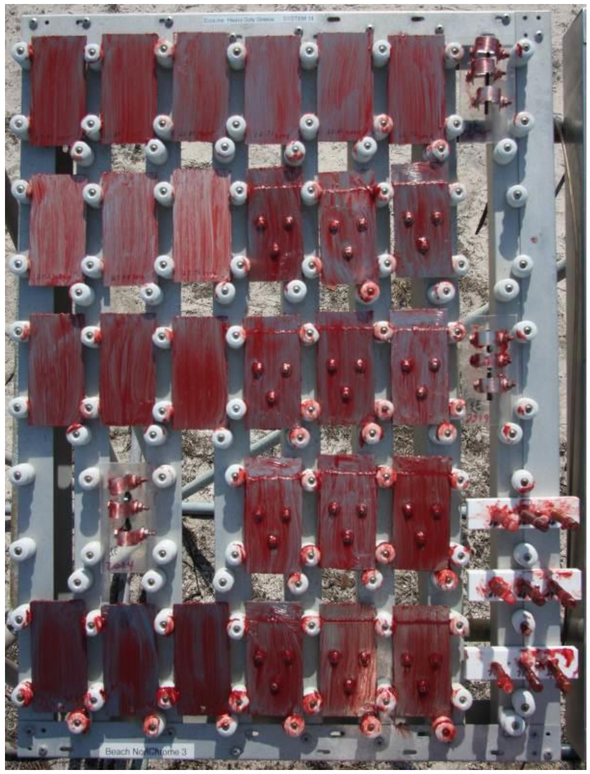


Figure 32. EcoLine Heavy Duty Grease

6.3.1 Long-term Beachside Atmospheric Exposure

<u>Test description</u>

This test evaluated the performance of the test CPC coatings after a 6-month outdoor exposure in a marine environment. The test panels were installed on April 10, 2012 at the KSC Beachside Atmospheric Corrosion Test Site on racks that are 150 feet from the ocean high tide line, Figure 33. The test panels were removed on September 27, 2012.

The test coupons were rated per ASTM G1, ASTM D610, and ASTM G46, depending on the substrate. ASTM G1 is used for cleaning procedures and is used for determining corrosion rates or mass loss. ASTM D610 uses the numerical grade scale in ASTM D 610, Scale and Description of Rust Grades, where 0 indicates 100% surface rusting and 10 indicating less than 0.01% surface rusting.

Figure 33. Initial exposure of the CPC-coated panels at the KSC Beachside Atmospheric Test Site

Rationale

This test documents the actual exposure of the coatings to the natural environment at KSC: ultraviolet radiation and the different cycles of salt spray exposure. NASA requires this test for validation of alternative coating systems and will provide similar data on the performance of CPCs.

Test Methodology

Table 23. Test methodology for long-term beachside atmospheric exposure

Parameters	150 feet from the ocean high tide at NASA Beachside Atmospheric Corrosion Test Site, ASTM G50
Coupons Per CPC/alloy	Three (3)
Trials Per Coupon	One (1)
Control Coupons Required For Testing	Three (3)
Acceptance Criteria	Panel condition (per ASTM D 610 or ASTM G 46) of candidate CPC rated equal to or better than untreated. Performs similar to control CPC.

Unique Equipment or Instrumentation

Outdoor test rack located 150 feet from ocean high tide line.

Data Analysis and Reporting

All of the CPC-coated panels were cleaned using Mineral Spirits to remove most of the CPC residue.

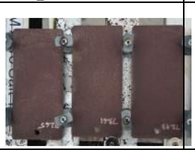
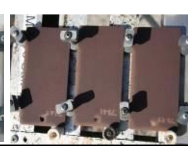
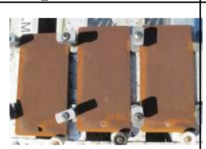
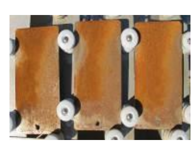
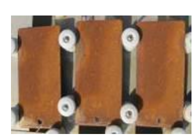
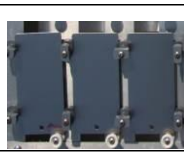
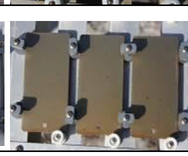
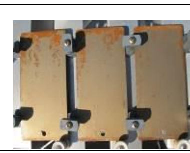
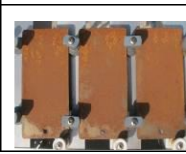
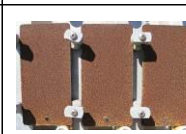
Iron Alloys

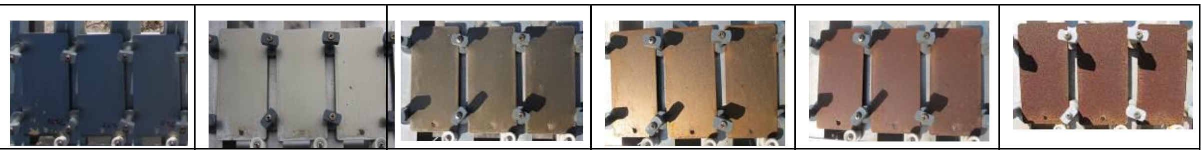
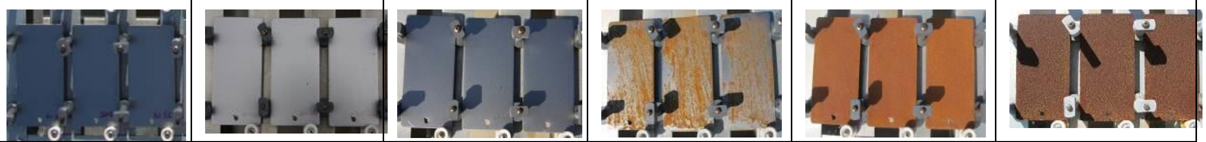
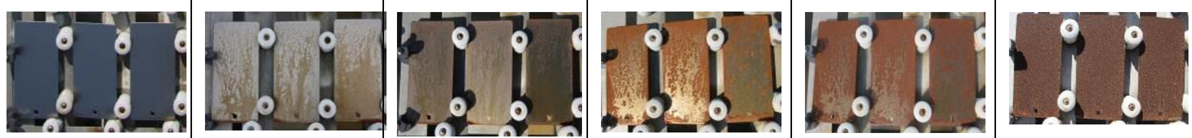
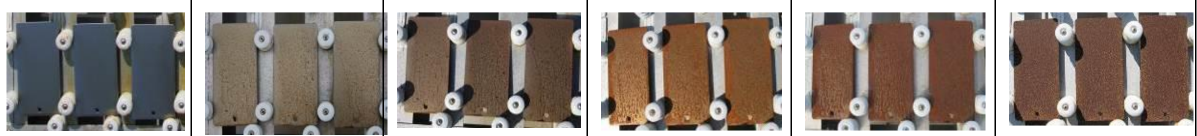
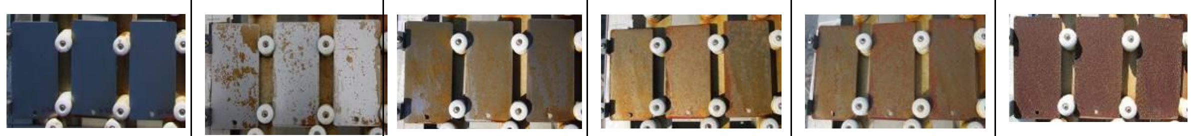
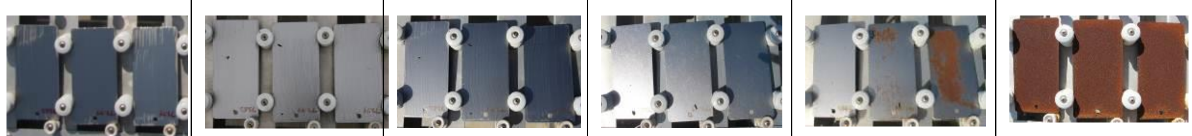
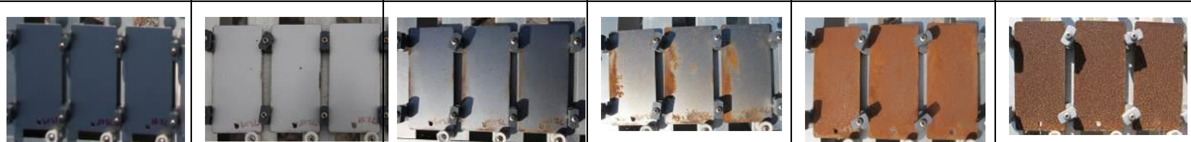
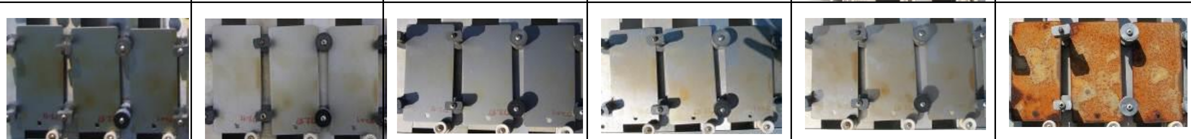
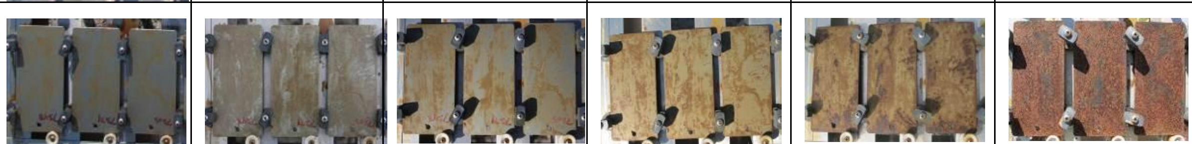
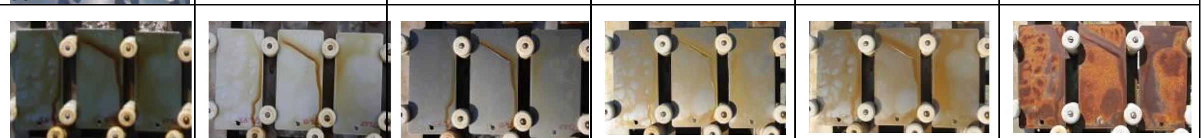
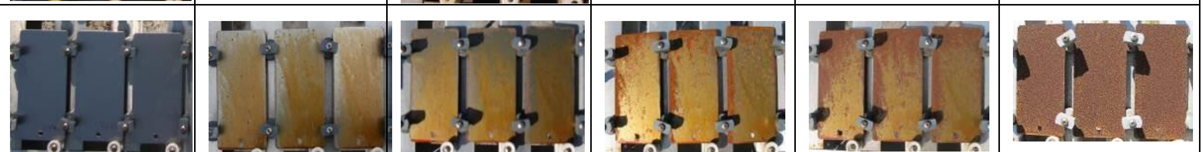
Carbon Steel:

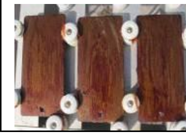
The carbon steel panels were cleaned of corrosion products using ASTM G1, Chemical Cleaning Procedures, where the mass loss was converted to corrosion rate as stated in Equation 1. Visual atmospheric exposure results for the CPCs on carbon steel and shown in Table 21. After twenty-two days of atmospheric exposure, Fluid Film, VpCI 368, Nox Rust 3100, Ardrox AV-30, and Bio-Acid Fume Rust Preventative Fluids, were performing the best. Only VpCI 368 and Nox Rust 3100 exhibited no corrosion products. All of the CPCs were performing better than the control. After six months all of the CPCs performed better than the uncoated panel and better than or similar to the control CPC, Corrosion X. CPC types VpCI368, Nox Rust 3100, and EcoLine Heavy Duty Grease had surface areas where no corrosion formed.

The corrosion rates, in mm/y, are shown in Figure 34 for the carbon steel panels after the six month atmospheric corrosion exposure. The corrosion rate correlates to the amount of mass loss that occurred as a function of time. The corrosion rates correlate with the visual results in Table 24, where the same CPCs that visually had less corrosion also had the lowest corrosion rates. Considering the aggressive KSC beachside environment and the long exposure time, all of the CPCs had a satisfactory degree of corrosion protection.

Table 24. CPC-coated Carbon Steel Panel from Initial Exposure through 6 Months

CPC Type	0 days exposure	3 days exposure	6 days exposure	13 days exposure	22 days exposure	6 months exposure
Blank						
Corrosion X (Control) 1						
WD-40 (comparison) 2						

NAVGUARD II (comparison) 4	
MX4 (comparison) 5	
EcoLine 3690 (candidate) 6	
Zerust Axxanol 46-BIO (candidate) 7	
Bio-Medium Preservative Lubricant (candidate) 8	
Fluid Film (candidate) 9	
WRL (candidate) 15	
VpCI 368 (Control) 10	
Ardrox AV-30 (comparison) 11	
Nox-Rust 3100 (comparison) 12	
Bio-Acid Fume Rust Preventative Fluids (candidate) 13	

EcoLine Heavy Duty Grease (candidate) 14						

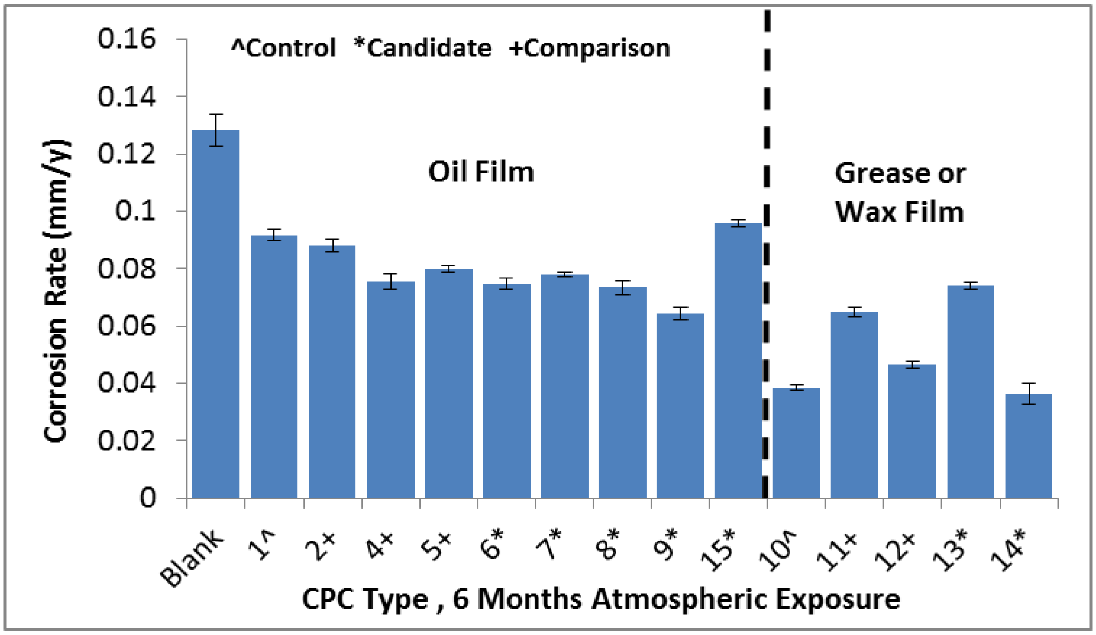


Figure 34. Corrosion rate results of CPC-coated carbon steel panels exposed to KSC's Beachside Atmospheric Corrosion Test Site for six months.

<u>Stainless Steel:</u>
The 304 stainless steel panels were rated for staining as a function of exposure time using ASTM D610. The rust grade percent was determined using visual inspection and quantified using the ASTM D610 grading system. This system grades the panels using the following guide for percent rust:

10: Less than or equal to 0.01 percent
9: Greater than 0.01 percent and up to 0.03
8: Greater than 0.03 percent and up to 0.1
7: Greater than 0.1 percent and up to 0.3
6: Greater than 0.3 percent and up to 1.0
5: Greater than 1.0 percent and up to 3.
4: Greater than 3.0 percent and up to 10.0
3: Greater than 10.0 percent and up to 16.0
2: Greater than 16.0 percent and up to 33.0
1: Greater than 33.0 percent and up to 50.0
0: Greater than 50 percent

The rust percent results are shown in Figure 35, where the wax films generally performed much better than the oil films in this corrosion environment. Candidate 9, Fluid Film, outperformed all other oil film types and three of the five wax films. The environmentally friendly wax and grease, 13(Bio-Acid Fume Rust Preventive Fluid) and 14 (EcoLine Heavy Duty Grease), performed much lower than the petroleum-based waxes, 10, 11, and 12.

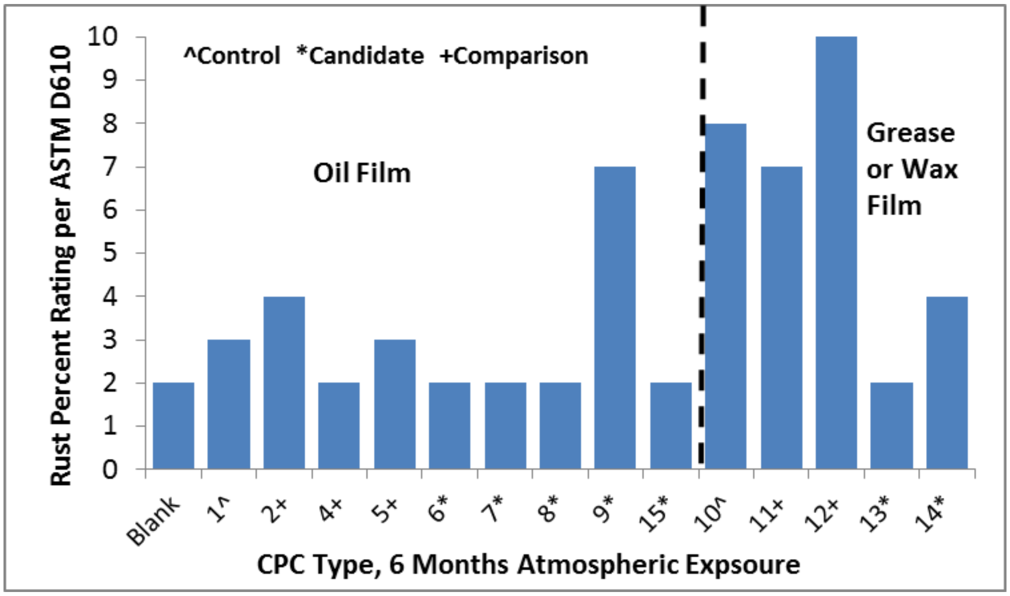


Figure 35. Corrosion ratings of CPC-coated stainless steel panels exposed to KSC's Beachside Atmospheric Corrosion Test Site for six months.

Aluminum Alloys

The aluminum alloys, 7075, 2024, and 2219, were evaluated for their pitting corrosion behavior. The pit density and pit size were recorded, per ASTM G46 and with the same modifications as for the Cyclic Corrosion Testing (7.2.1). The criteria were shown in Figure 14 for both pit density and size. The measurements were made using a microscope at 10X.

The different alloys had varying degrees of susceptibility to corrosion; therefore, the results are discussed by alloy type.

<u>7075:</u>

For the CPC films, most of the CPC types protected against pitting, as the pit density was 1 for all but the following CPCs: 4(NAVGUARD II), 7(Zerust), 11(Ardrox AV 30), and 14 (EcoLine Heavy Duty Grease). The pit size was nominal at a rating of 1 for all of the CPCs types and the blank panels. CPC type 10(VpCI368) exhibited no pitting and was considered to have provided excellent protection to the 7075 substrate.

<u>2024:</u>

Aluminum alloy 2024 is more susceptible to corrosion; however, the results were not entirely consistent for ranking purposes to the 7075 results. In this case, seven different CPC types performed the same as the untreated panel: 4(NAVGUARD II), 6(EcoLine 3690), 8(Bio-Medium Rust Inhibitor), 9(Fluid Film), 15(WRL), 12(Nox Rust 3100), and 14(EcoLine Heavy Duty Grease). The pit size was nominal at a rating of 1 for all but one of the CPCs types and the blank panels. WRL performed worse than the untreated or control-treated 2024 panels. CPC type 10(VpCI368) exhibited no pitting and was considered to have provided excellent protection to the 7075 substrate.

2219:

Aluminum alloy 2219 was the most susceptible to pitting corrosion of the alloys tested. Only two CPC types, 10 (VpCI368) and 12(Nox Rust 3100), exhibited a lower pit density than the untreated panels. CPC 10 had not pitting and was considered excellent in corrosion protection of aluminum considering the aggressive length of time that the temporary coating was exposed to the beachside environment. An example of pitting on 2219 is shown in Figure 36 for NAVGUARD II and VpCI368, along with the uncoated panel.

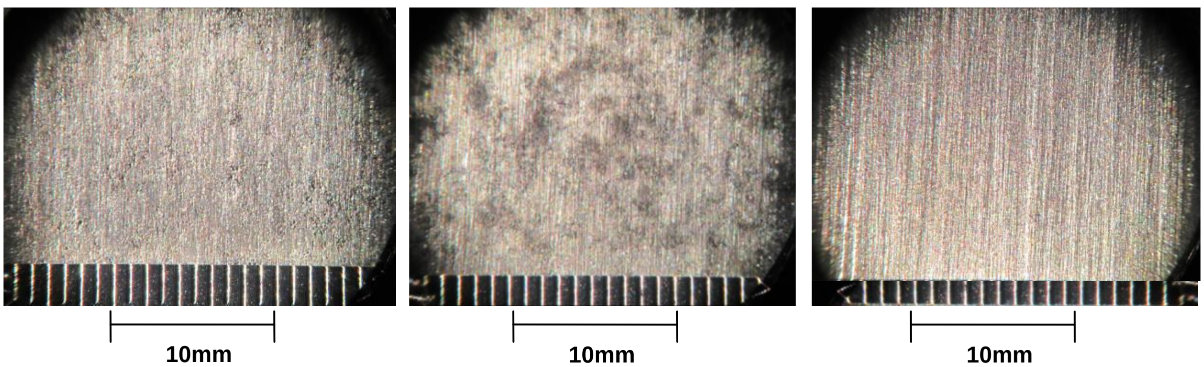


Figure 36. Examples of pitting results for long-term atmospheric exposure of CPC-coated aluminum alloy 2219: Blank (left), CPC 4 (center), and CPC 10 (right).

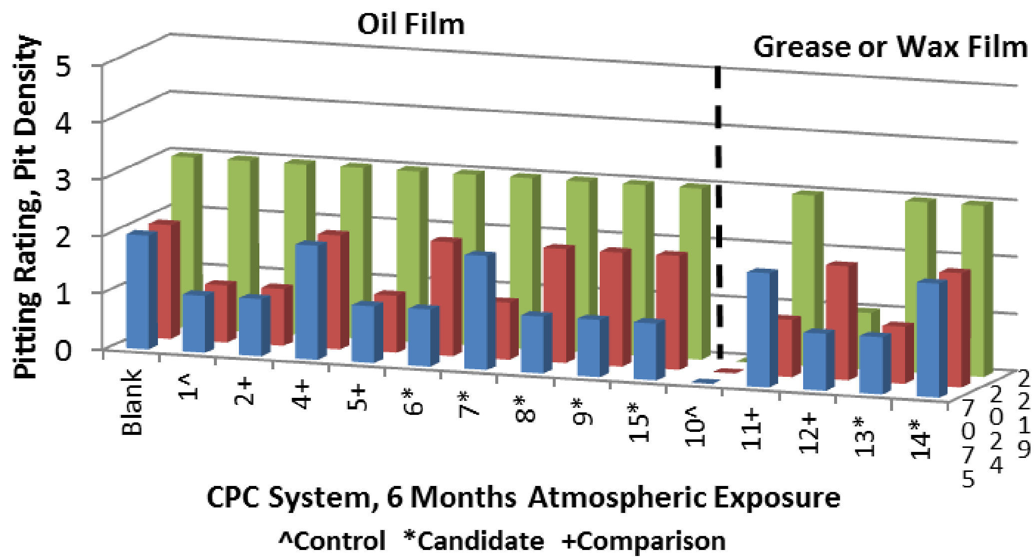


Figure 37. Pit density results of CPC-coated aluminum alloy panels exposed to KSC's Beachside Atmospheric Corrosion Test Site for six months.

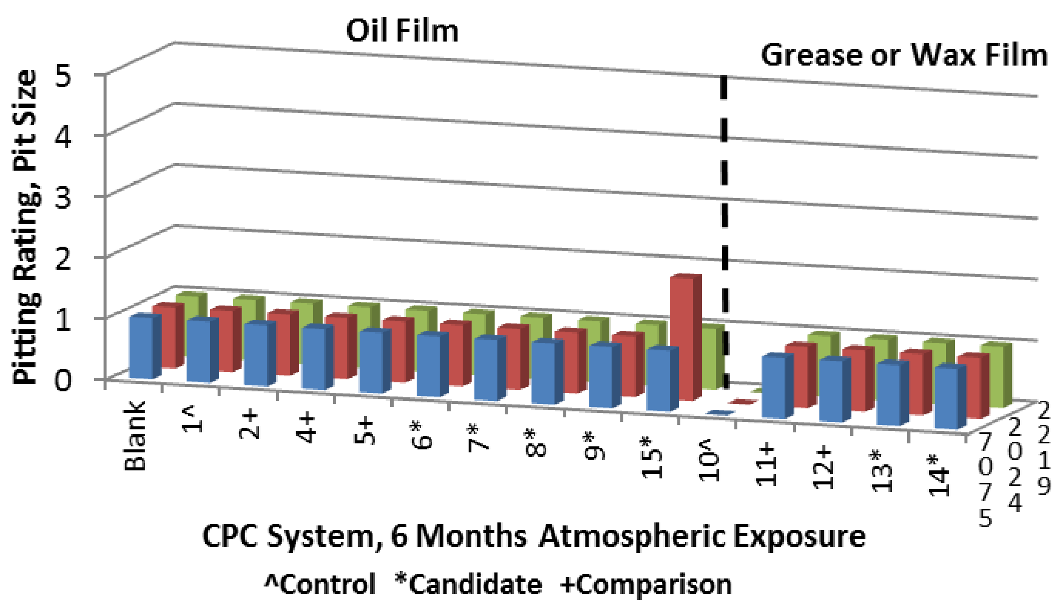


Figure 38. Pit size results of CPC-coated aluminum alloy panels exposed to KSC's Beachside Atmospheric Corrosion Test Site for six months.

6.3.2 Sandwich Corrosion

Test description

This test method, ASTM F1110, Standard Test Method for Sandwich Corrosion Test, was used to determine the suitability of a CPC to limit or prevent, as opposed to induce, corrosion in a sandwiched configuration. A CPC-soaked piece of filter paper was sandwiched between two panels of the same metal type. The panels were then fastened together using a washer, nut, and bolt configuration, shown in Figure 39. The panels were exposed at the KSC Beachside Atmospheric Corrosion Test Site on racks that are 150 feet from the ocean high tide line for 6 months.

Figure 39. Sandwich corrosion panels that were also used for crevice and galvanic corrosion (at fasteners).

Rationale

CPCs are used specifically in sandwich configurations that are difficult to otherwise coat. It is important that the CPCs do not cause corrosion.

Methodology Table

Table 25. Test Methodology for Crevice Corrosion Test

Parameters	Reference ASTM F1110, ASTM G50
Coupons Per CPC/alloy	Three (3)
Trials Per Coupon	One (1)
Control Coupons Required For Testing	One (1) untreated per alloy

Acceptance Criteria	Performs better than untreated. Performs similar to control CPC.

Unique Equipment or Instrumentation

Outdoor test rack located 150 feet from ocean high tide line.

Data Analysis and Reporting

The sandwich panels were evaluated using ASTM F1110. This method uses a scale to quantify the appearance of the area under the filter paper. The following rating system was used:

- 0: No visible corrosion and no discoloration present
- 1: Very slight corrosion or very slight discoloration, and/or up to 5% of area corroded
- 2: Discoloration and/or up to 10% of area corroded
- 3: Discoloration and/or up to 25% of area corroded
- 4: Discoloration and/or more than 25% of area corroded, and/or pitting present

The sandwich panel test results for the aluminum alloys are shown in Figure 40, where CPCs 4, 5, 8, 9, 10, 12, and 14 performed the best overall, and CPCs 2, 6, 7, and 15 performed the worst and nearly as bad as the untreated sandwich panels. The black areas on the panels, shown in Table 26, are the remains of mold that grew in the sandwich area during the exposure period. Mold was noted in Table 26 for each panel/CPC type. CPCs 8 and 9 were the only CPCs that showed no signs of mold on any of the aluminum alloy types.

The sandwich panel test results for stainless steel are shown in Figure 41, where the majority of CPC types showed no signs of corrosion and had a rating of 0. CPC types 7, 10, and 13 had a rating of 1 or higher for at least one of the triplicate coupons. Photographs of the panels after exposure are shown in Table 26. No mold was observed on the stainless steel panels.

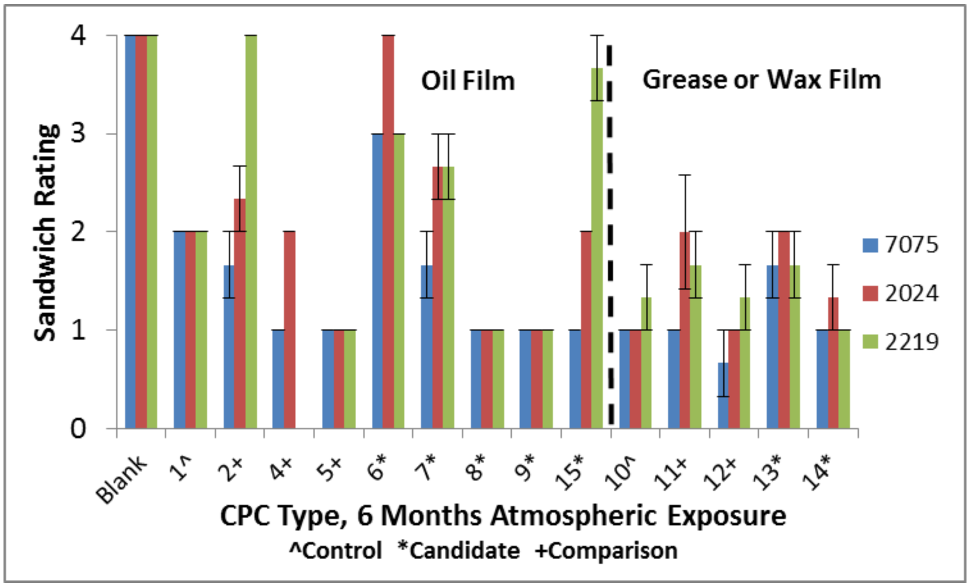
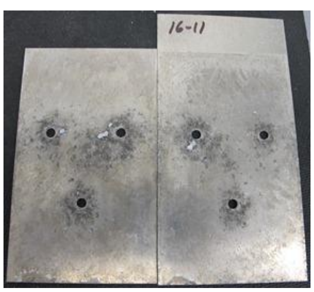
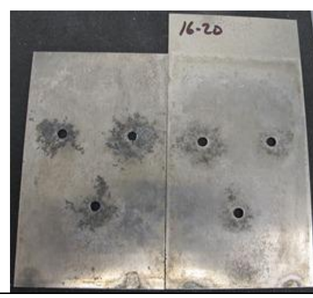
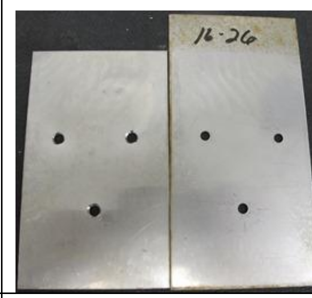
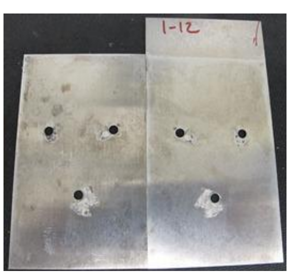
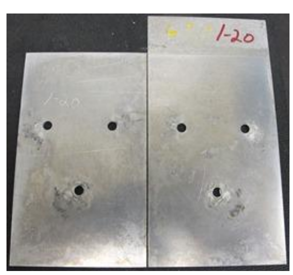
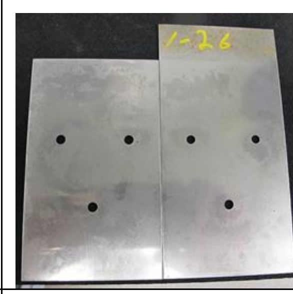
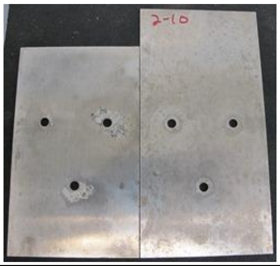
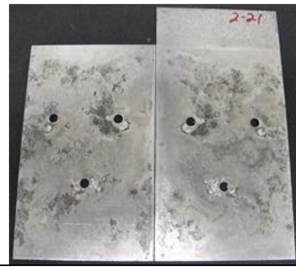
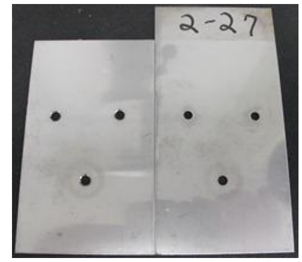
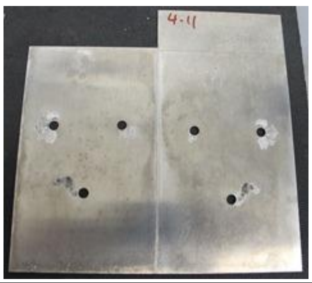
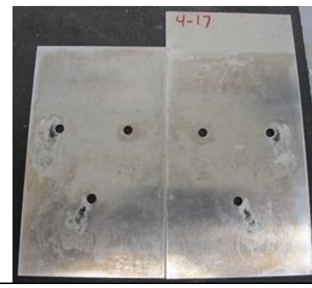
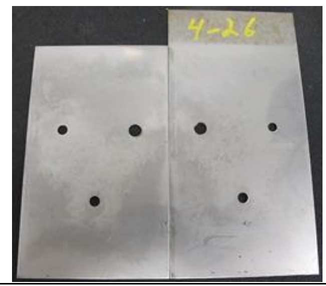
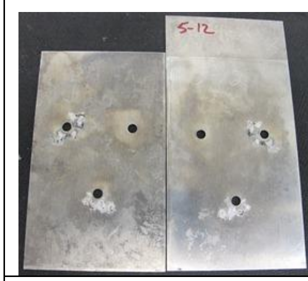
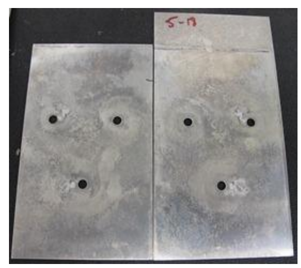
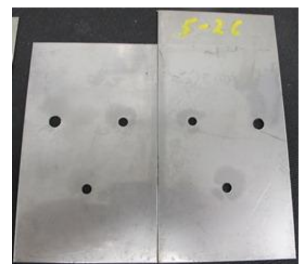
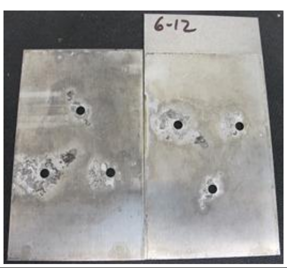
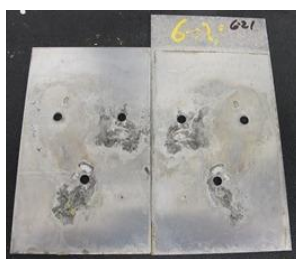
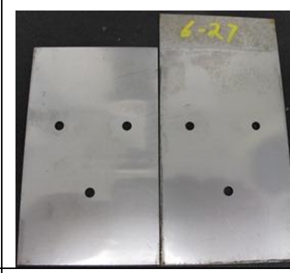
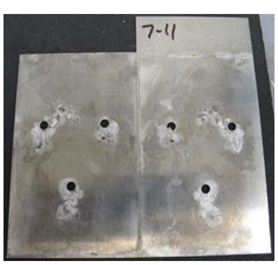
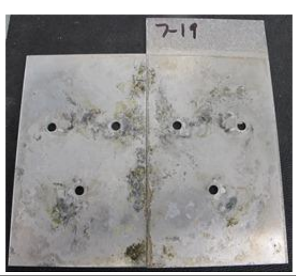
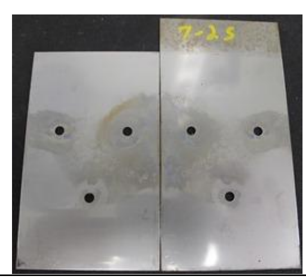
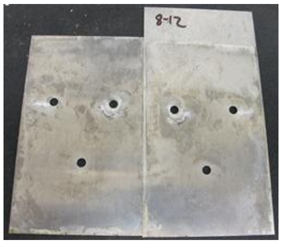
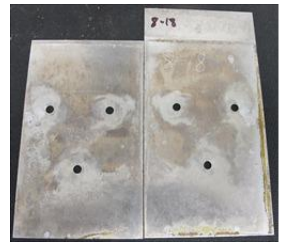
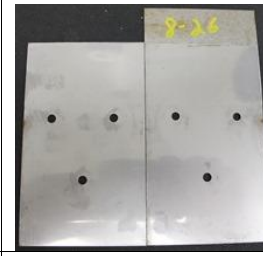
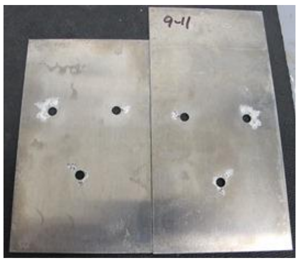
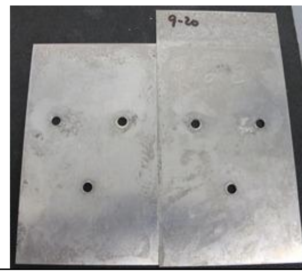
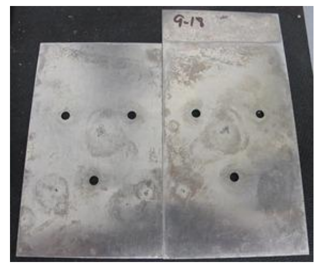
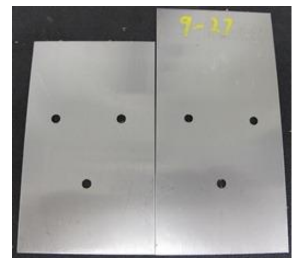
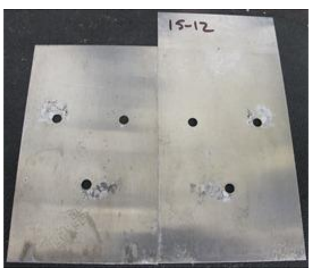
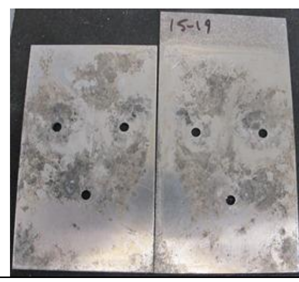
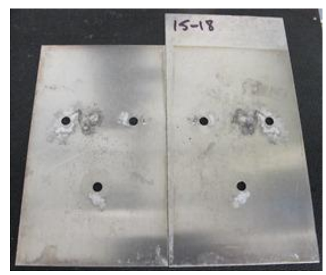
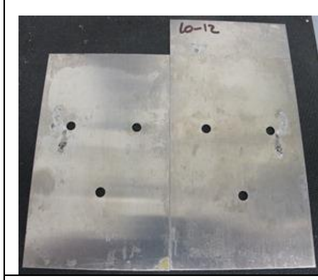
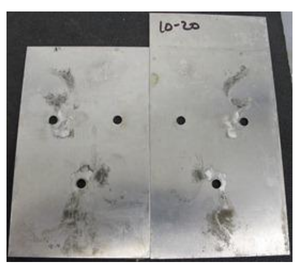
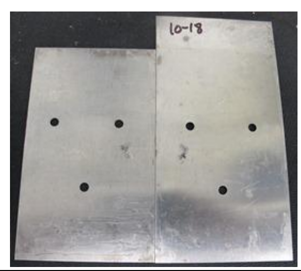
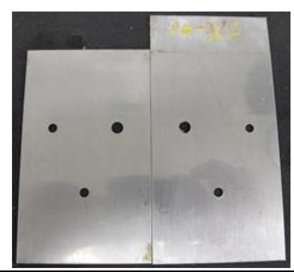
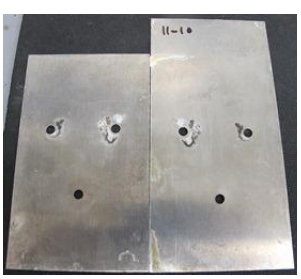
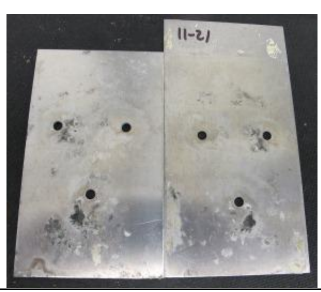
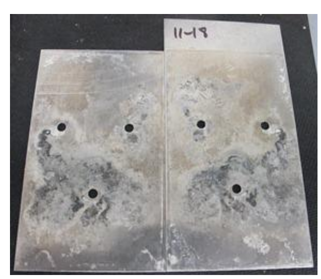


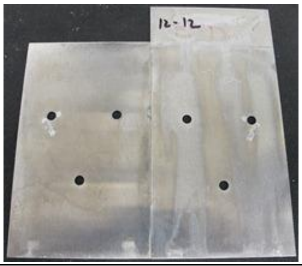
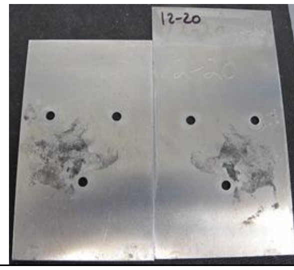
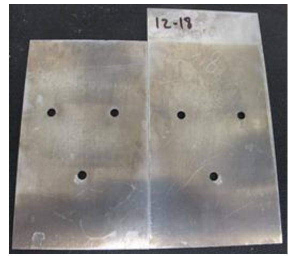
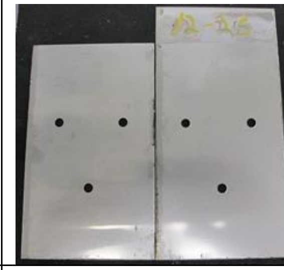
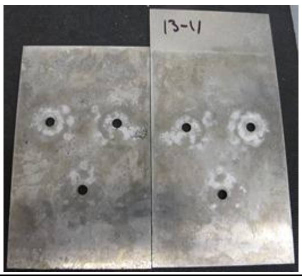
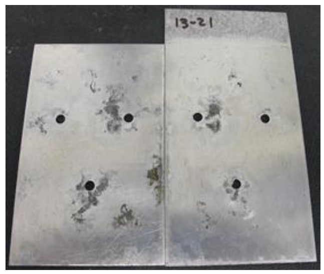
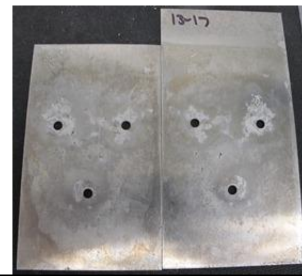
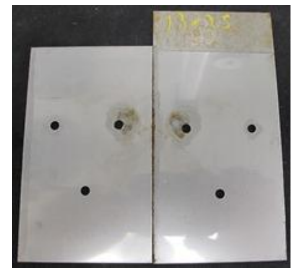
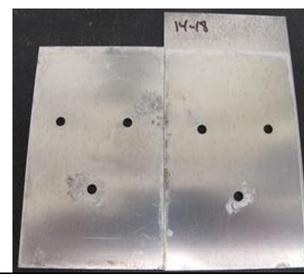
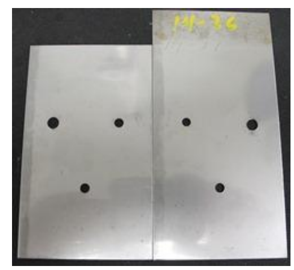
Figure 40. Sandwich corrosion results for aluminum alloys exposed to the KSC Beachside Atmospheric Corrosion Test Site for Six Months.

Table 26. Photographs of Sandwich Corrosion Results

CPC Type	AA7075	AA2219	AA2024	304 SS
Blank				
	Mold	Mold	Mold	No Mold
Corrosion X (Control) 1				
	No Mold	Mold	Mold	No Mold

WD-40 (comparison) 2				
	No Mold	Mold	Mold	No Mold
NAVGUARD II (comparison) 4				
	Mold	Mold	Mold	No Mold
MX4 (comparison) 5				
	Mold	Mold	Mold	No Mold
EcoLine 3690 (candidate) 6				
	Mold	Mold	Mold	No Mold
Zerust Axxanol 46-BIO (candidate) 7				
	Mold	Mold	Mold	No Mold

Bio-Medium Preservative Lubricant (candidate) 8				
	No Mold	No Mold	No Mold	No Mold
Fluid Film (candidate) 9				
	No Mold	No Mold	No Mold	No Mold
WRL (candidate) 15				
	Mold	Mold	Mold	No Mold
VpCI 368 (Control) 10				
	Mold	Mold	Mold	No Mold
Ardrox AV-30 (comparison) 11				
	Mold	Mold	Mold	No Mold

Nox-Rust 3100 (comparison) 12				
	No Mold	Mold	No Mold	No Mold
Bio-Acid Fume Rust Preventative Fluids (candidate) 13				
	No Mold	Mold	No Mold	No Mold
EcoLine Heavy Duty Grease (candidate) 14				
	No Mold	No Mold	Mold	No Mold

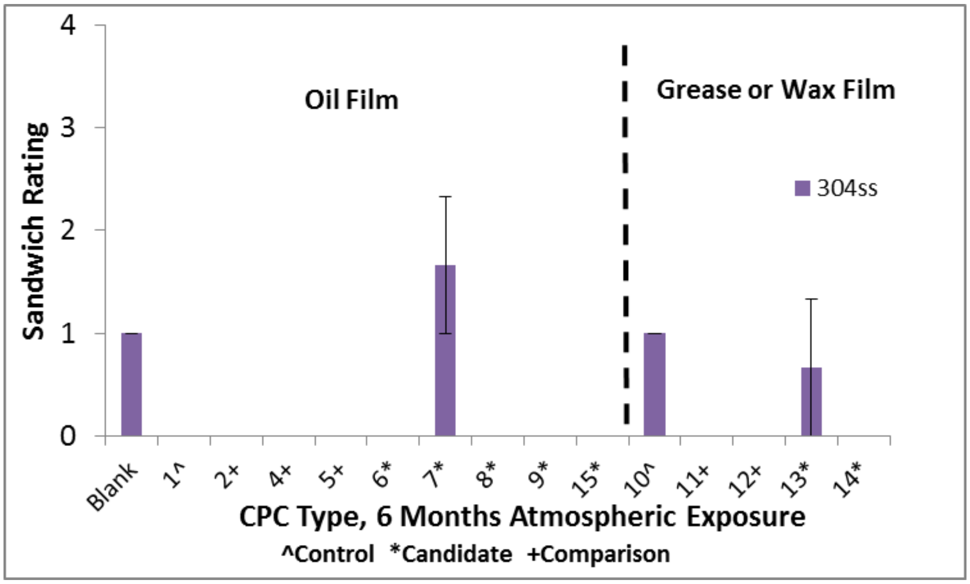


Figure 41. Sandwich corrosion results for 304 SS exposed to the KSC Beachside Atmospheric Corrosion Test Site for Six Months.

6.3.3 Crevice Corrosion

<u>Test description</u>

A modified version of ASTM G78, Standard Guide for Crevice Corrosion Testing of Iron-Base and Nickel-Base Stainless Alloys in Seawater and Other Chloride-Containing Aqueous Environments, was designed to determine the suitability of a CPC to limit or prevent, as opposed to induce, crevice corrosion. Fasteners with crevice forming stainless steel washers were attached to a flat panel prior to application of the CPCs to induce crevice corrosion around a washer in the atmospheric corrosion environment. The panels were exposed at the KSC Beachside Atmospheric Corrosion Test Site on racks that are 150 feet from the ocean high tide line for 6 months. In this case, the fastener assemblies were attached to the lower portion of the sandwich corrosion coupons, shown in Figure 42.

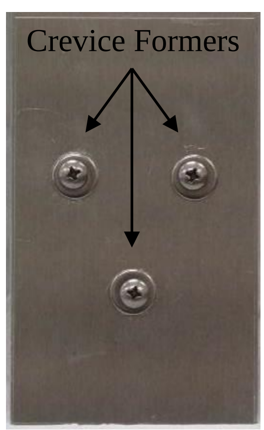


Figure 42. Crevice Corrosion Panel

Rationale

CPCs are used specifically in creviced areas that are difficult to otherwise coat. It is important that the CPCs do not cause crevice corrosion.

Methodology Table

Table 27. Test Methodology for Crevice Corrosion Test

Parameters	Reference ASTM G78 , ASTM G50
Coupons Per CPC/alloy	Three (3)
Trials Per Coupon	One (1)
Control Coupons Required For Testing	One (1) untreated per alloy
Acceptance Criteria	Performs better than untreated. Performs similar to control CPC.

Unique Equipment or Instrumentation

Outdoor test rack located 150 feet from ocean high tide line.

Data Analysis and Reporting

Most of the CPC-coated panels showed some sort of crevice corrosion, whether shown as small pits along the fastener ring edge or deep crevices induced across the entire fastener surface. A photograph of examples of the crevice corrosion types is shown in Figure 43.

The aluminum alloy panels induced crevice corrosion primarily along the fastener edge, but also across the fastener surface. Because the washer in direct contact to the aluminum alloy panels were 316SS, a dissimilar metal, galvanic corrosion was also induced and noted separately from the crevice corrosion. Crevice corrosion was induced for all but one CPC/alloy systems, Corrosion X on AA2219. Any form of crevice corrosion is considered a failure because it is assumed that even a small crevice will eventually form into a larger crevice. Bio-Medium Preservative Lubricant, VpCI368, and Nox-Rust 3100 had the least amount of crevice corrosion. Results for WRL were nearly as severe as the uncoated panels. Figure 44 shows the percent crevice corrosion results, reported as the percent of total coverage around the fastener, for the aluminum alloys.

The stainless steel panels only induced crevice corrosion, as the fastener and panel metal type were not considered to be dissimilar metals (no galvanic corrosion would be induced as well). All of the CPCs did a fairly good job at preventing extensive crevice corrosion; however, most of the CPCs performed worse than the control CPC. The degree of crevice corrosion, reported as the percent of total coverage around the fastener, is shown in Figure 45.

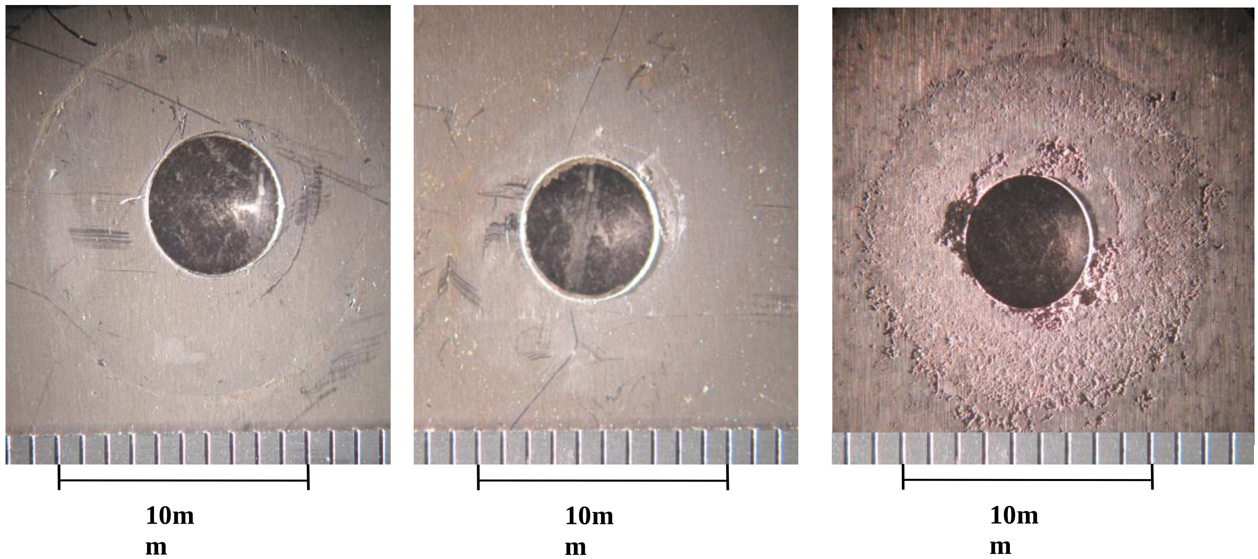


Figure 43. Examples of crevice corrosion for atmospheric exposure of CPC-coated stainless steel (left - blank and center- CPC 6), and crevice and galvanic corrosion of aluminum alloy 7075 (right -CPC 7).

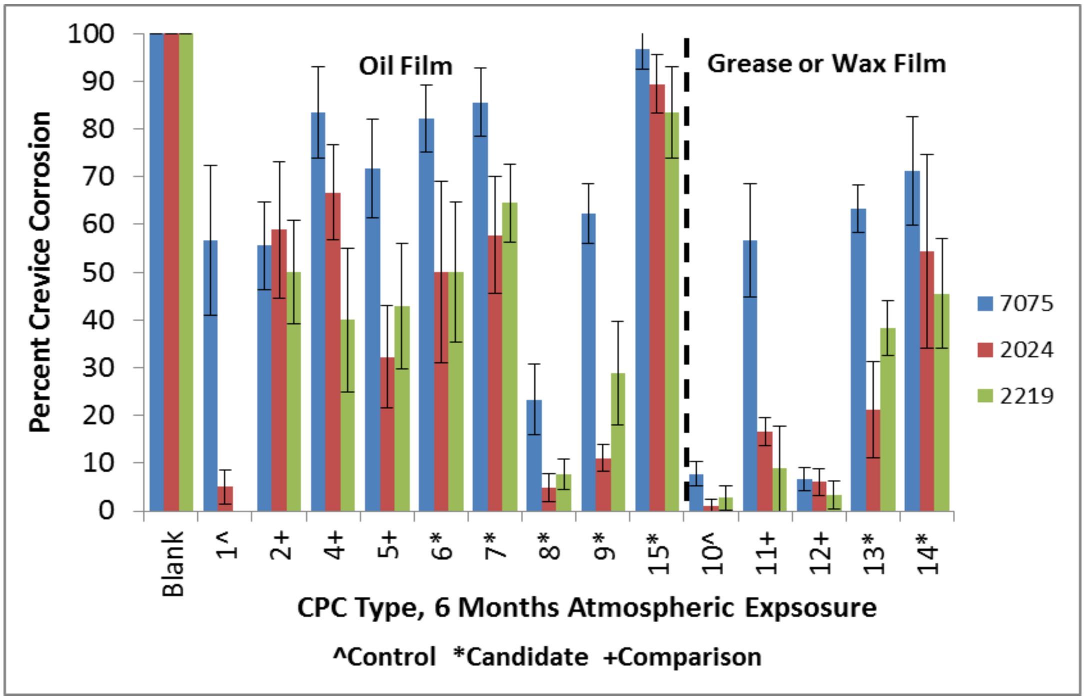


Figure 44. Percent crevice corrosion via fasteners results of CPC-coated aluminum alloy panels exposed to KSC's Beachside Atmospheric Corrosion Test Site for six months.

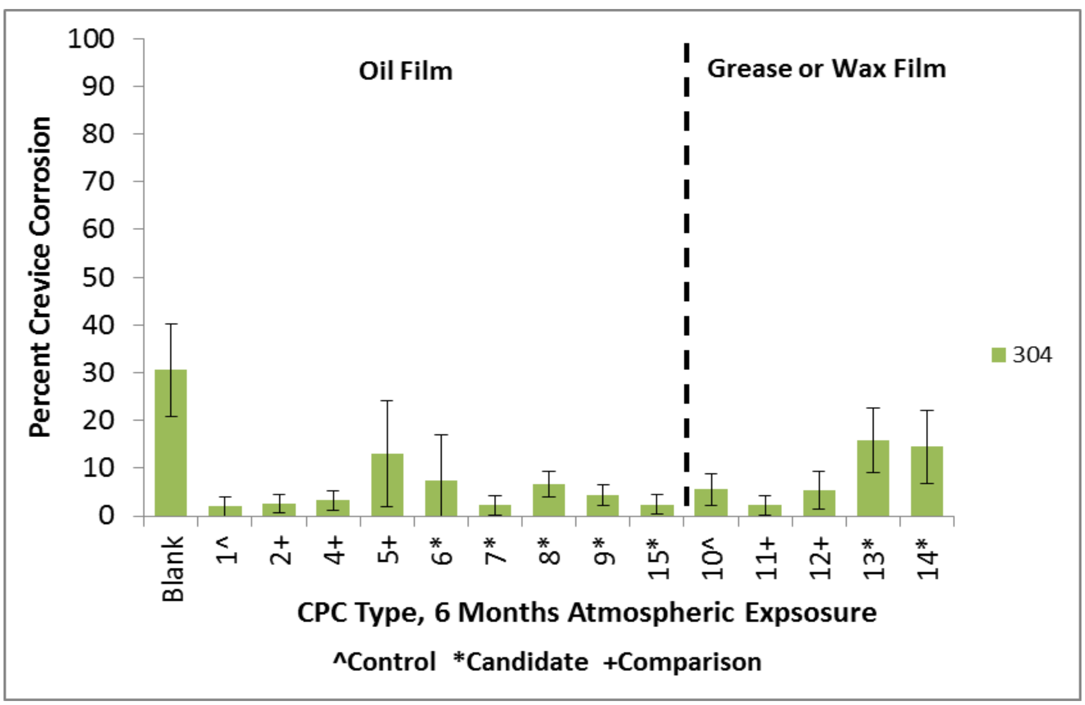


Figure 45. Percent crevice corrosion via fasteners results of CPC-coated 304 stainless steel panels exposed to KSC's Beachside Atmospheric Corrosion Test Site for six months.

6.3.4 Galvanic Corrosion via Fasteners

Test description

This test method, a modified version of ASTM G104, "*Standard Test Method for Assessing Galvanic Corrosion Caused by the Atmosphere*", was used to determine the relative amount and characteristics of galvanic corrosion, where two dissimilar metals were in intimate electrical contact while being exposed to a corrosive environment. The test method uses a nut and bolt assembly to create the galvanic conditions using a washer that is a dissimilar metal. The panels were exposed at the KSC Beachside Atmospheric Corrosion Test Site on racks that were 150 feet from the ocean high tide line. In this case, the nut and bolt assemblies were attached to the lower portion of the sandwich corrosion coupons, shown in Figure 42.

Rationale

CPCs are often used to protect against galvanic corrosion, especially on structures where nut and bolt configurations are used heavily. This test was used to determine a CPCs ability to inhibit or induce galvanic corrosion. CPCs should not cause galvanic corrosion and may be beneficial in inhibiting the corrosion.

Methodology Table

Table 28. Test Methodology for Galvanic Corrosion via Fasteners

Parameters	Reference ASTM G104, ASTM G50
Coupons Per CPC/alloy	Three (3), will use the same coupons as for crevice corrosion. Three (3) bolts per panel.
Trials Per Coupon	One (1)
Control Coupons Required For Testing	One (1) untreated per alloy.
Acceptance Criteria	Performs better than untreated. Performs similar to control CPC.

Unique Equipment or Instrumentation

Outdoor test rack located 150 feet from ocean high tide line.

Data Analysis and Reporting

All of the CPC-coated panels induced a degree of galvanic corrosion except for VpCI 368 on AA7075. Overall any galvanic corrosion is considered a failure, as it is assumed that initial corrosion will eventually proceed as a stronger corrosion cell under the washer configuration.

Bio-Medium Preservative Lubricant, VpCI 368, and Nox-Rust 3100, Ardrox AV-30, and Bio-Acid Fume Rust Preventative were most successful in controlling galvanic corrosion when compared to the other CPC types. Results for WRL were nearly as severe as the uncoated panels. Figure 46 shows the percent galvanic corrosion results, reported as the percent of total coverage around the fastener, for the aluminum alloys.

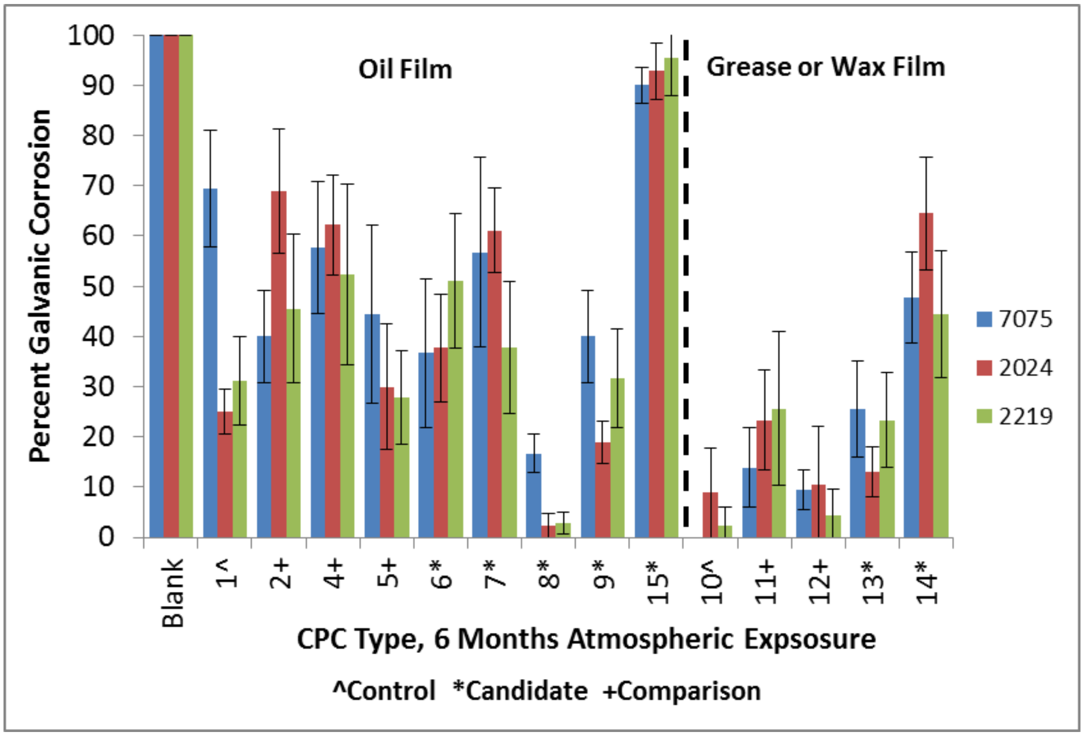


Figure 46. Percent galvanic corrosion via fasteners results of CPC-coated aluminum alloy panels exposed to KSC's Beachside Atmospheric Corrosion Test Site for six months.

6.3.5 Galvanic Corrosion via CLIMAT Wire on Bolt Assemblies

Test description

ASTM G116, Galvanic Corrosion via the CLIMAT (CLassify Industrial and Marine ATmospheres), Wire on Bolt, is a test that creates an interaction between two materials of different galvanic potentials. This interaction is formed by wrapping a wire of an anodic material around the threads of a bolt or threaded rod of a cathodic material which produces a galvanic cell. The anodic wire preferentially corrodes as a result of the galvanic interaction with the cathodic bolt. Reference specimens of the anode wire on a threaded, nonconductive, non-porous rod (nylon) are used to separate general and crevice corrosion effects from galvanic corrosion effects produced by the galvanic cells. Exposing the galvanic cell in a corrosive atmospheric environment for a set duration of time allows for a comparison of the effectiveness of the CPCs to protect the materials from the effects of galvanic corrosion in an atmospheric environment.

CLIMAT assemblies were constructed by measuring the mass of a known quantity of aluminum wire and wrapping it around a non-conductive and cathodic material. The non-conductive material was nylon, which was chosen to elucidate general and crevice corrosion from galvanic corrosion because it does not initiate a galvanic cell. The mass of the aluminum wire was measured and subsequently wrapped around copper and iron material (bolt). The finished assembly is shown in Figure 47.

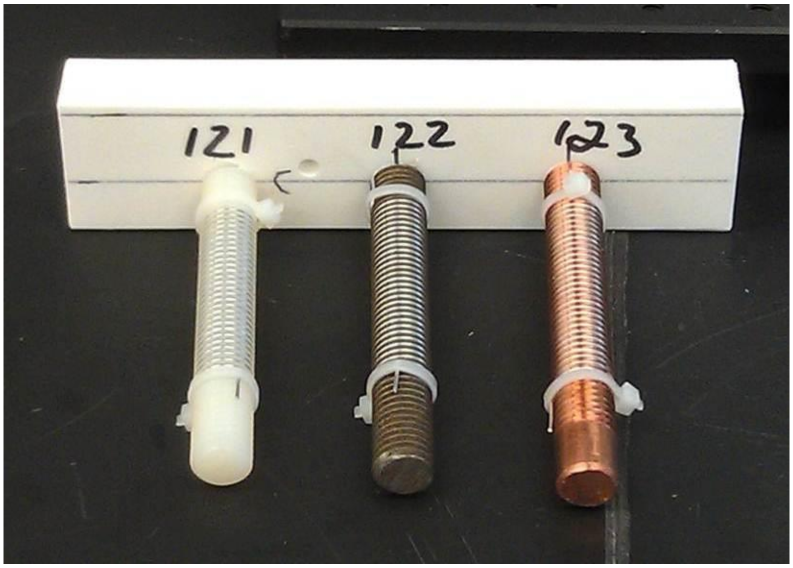


Figure 47. CLIMAT Assembly

CPCs were applied to the appropriate CLIMAT assemblies as required, and the assemblies were mounted to test fixtures at the KSC Beachside Atmospheric Corrosion Test Site, on racks that are 150 feet from the ocean high tide line. These assemblies were inspected weekly. After the one month exposure, the aluminum wire was removed from the bolts and cleaned of corrosion products according to ASTM G1. The aluminum wire was weighed and a mass loss was calculated from the pre-exposure masses.

Rationale

CPCs are often used to protect against galvanic corrosion, especially on structures that utilize dissimilar metals. CPCs reduce corrosion to galvanic assemblies through processes which include, but are not limited to, protective redox reactions, barrier properties and water displacing characteristics. By measuring the mass loss of the wire on the CLIMAT assemblies, it is possible to measure the effectiveness in which the CPC protects the dissimilar (galvanic) materials. CPCs should not cause galvanic corrosion and may be beneficial in inhibiting the corrosion. To investigate accelerated corrosion resulting from the application of CPCs, a non-treated assembly was used as a control. Consequently, this test will be used to determine a CPCs ability to inhibit or induce galvanic corrosion.

Methodology Table

Table 29. Test Methodology for Galvanic Corrosion via Wire and Bolt

Parameters	Reference ASTM G116, ASTM G50, ASTM G1
Coupons Per CPC/alloy	Three (3) per cathode type, 1100 series aluminum anode wire wrapped around rods of nylon, 1010 mild steel, and CA110 copper
Trials Per Coupon	One (1)
Control Coupons Required For Testing	One (1) untreated per cathode type.
Acceptance Criteria	Performs better than untreated control. Performs similar to control CPC.

Unique Equipment or Instrumentation

Outdoor test racks and stands located 150 feet from the high tide line of the Atlantic Ocean.

Data Analysis and Reporting

The mass loss of the aluminum wire as a function of CPC protected CLIMAT assembly is shown in Figure 48. Analysis of the data in Figure 48 clearly shows that Corrosion X, Bio-Medium Preservative Lubricant, Fluid Film, and Bio-Acid Rust Preventative provided the most effective protection against corrosion for both the aluminum-iron and aluminum-copper galvanic couples. Although other CPC types were successful against the iron-based galvanic corrosion, a CPC will be considered successful if the more aggressive copper galvanic corrosion is also protected. None of the CPCs accelerated corrosion since they all exhibited mass losses lower than the control, designated as Blank.

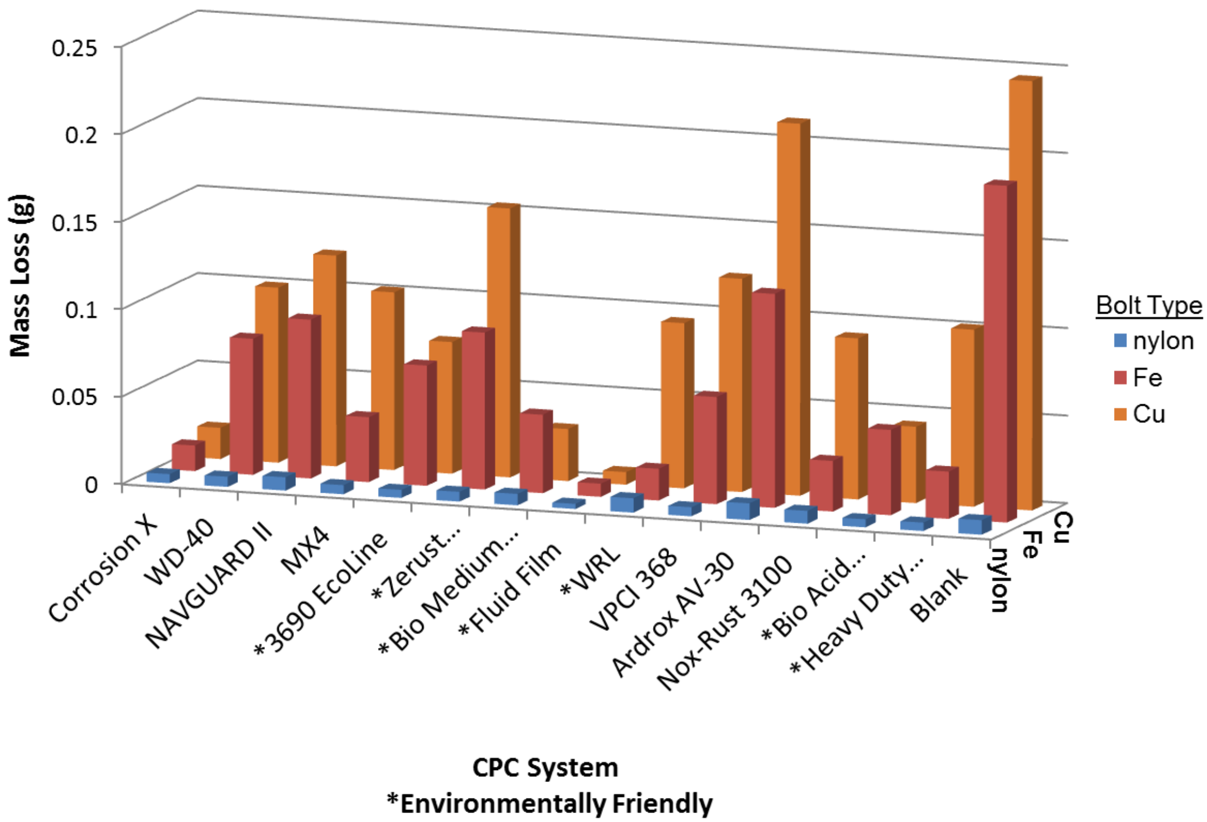


Figure 48. Mass Loss of Aluminum Wire on Threaded Bolts after 6 Months Exposure at the KSC Beachside Atmospheric Corrosion Test Site.

6.3.6 Stress Corrosion Cracking

Test description

This test method covers a uniform accelerated procedure for characterizing the resistance to stress-corrosion cracking (SCC) of high-strength aluminum alloy wrought products, particularly when stressed in the short transverse grain direction. The assemblies, shown in Figure 49 were exposed at the KSC Beachside Atmospheric Corrosion Test Site, on racks that are 150 feet from the ocean high tide line, for a six month period.

Figure 49. Picture of a C-ring clamp for exposure at the KSC Beachside Atmospheric Corrosion Test Site.

Rationale

This test was used to determine a CPCs ability to inhibit or induce stress corrosion cracking on 2000 and 7000 series aluminum alloys products. CPCs should not cause stress corrosion cracking and may be beneficial in inhibiting the corrosion.

Methodology Table

Table 30. Test Methodology for Stress Corrosion Cracking

Parameters	Reference ASTM G47, ASTM G50
Coupons Per CPC/alloy	Three (3), aluminum alloys only
Trials Per Coupon	One (1)
Control Coupons Required For Testing	One (1) untreated per alloy.
Acceptance Criteria	Performs better than untreated. Performs similar to control CPC.

Unique Equipment or Instrumentation

C-ring clamps for atmospheric exposure. Outdoor test rack located 150 feet from ocean high tide line.

Data Analysis and Reporting

No samples, including the uncoated samples, showed any degree of stress-induced cracking after six months of beachside atmospheric exposure.

6.4 Compatibility with NASA Environments

6.4.1 Liquid Oxygen (LOX)

Test description

The purpose of this test was to determine if materials in liquid oxygen (LOX) environments react when mechanically impacted. A reaction from mechanical impact can be determined by an audible report, an electronically or visually detected flash, or obvious charring of the sample, sample cup, or striker pin.

This test was to be performed in accordance with NASA-STD-6001, Flammability, Odor, Offgassing, and Compatibility Requirements and Test Procedures for Materials in Environments that Support Combustion, specifically, Test Method 13A, Mechanical Impact for Materials in Ambient Pressure LOX. The test system would be identical to that described in ASTM D 2512 [Compatibility of Materials with Liquid Oxygen (Impact Sensitivity Threshold and Pass-Fail Techniques)].

Rationale

This test is specified in NASA-STD-6001 and was initially identified as a testing requirement. Materials intended for use in space vehicles, specified test facilities, and specified GSE must meet the requirements of this document.

Test Methodology

Table 31. Test Methodology for LOX Compatibility Test

Parameters	Per NASA-STD-6001; The thickness of the sample must be the worst-case thickness. Test conditions (pressure and temperature) are the ambient pressure of the test facility and the boiling point of LOX at that pressure.
Coupons Per CPC	Twenty (20)
Trials Per Coupon	One (1)
Control Coupons Required For Testing	None
Acceptance Criteria	Twenty samples must not react when impacted at 72 ft-lbs (98 J). If one sample out of 20 reacts, 40 additional samples must be tested without any reactions.

Unique Equipment and Instrumentation

ABMA-Type Impact Tester

Data Analysis and Reporting

The test criteria was reviewed by NASA Engineering, and it was determined that the LOX compatibility testing is not practical for CPC testing.[42] Currently no hydrocarbon materials are compatible and they were considered to surely fail. Unless the CPC materials are going to be used within the pressure vessels, the testing was deemed not necessary. CPC use at KSC will be avoided in the same way that hydrocarbon materials are used currently. Only fluoropolymer CPC types are used with LOX currently.

6.4.2 Hypergol Compatibility

Test description

This procedure evaluated the effects on coatings from casual exposure to hypergolic fluids [nitrogen tetroxide (N_2O_4), hydrazine (N_2H_4), and monomethylhydrazine (MMH)]. This procedure provided the method to determine if a fluid could react exothermally or spontaneously ignite on contact with a material.

This test was performed in accordance with NASA KSC MTB-175-88, Procedure for Casual Exposure of Materials to Hypergolic Fluids, Test Method 7.1, Reactivity Test Method. The CPC coatings were applied in a thickness equivalent to normal use on aluminum foil measuring four (4) inches by four (4) inches. The CPCs were tested in an uncured state.

The testing procedure consisted of the following steps:

1. Apply 4 drops of CPC to be tested to aluminum weigh boat (triplicate per CPC)
   a. For thicker CPCs, an equivalent volume (~1cm^2) was applied to the bottom of the aluminum weigh boat)
2. Determine pre-exposure temperature of CPC sample (allow to stabilize for 30 seconds)
3. Expose each sample to hypergolic fuel or oxidizer simulant, 4 drops (1:1 ratio by volume)
4. Monitor temperature and observe samples for signs of gross incompatibilities for the duration of the test. Notate the maximum temperature reached by the sample at any point during the test.

Rationale

This test is specified in NASA-STD-6001 and was identified as a testing requirement. Materials intended for use in space vehicles, specified test facilities, and specified ground support equipment (GSE) must meet the requirements of this document.

---

[42] Ward, J., NASA John F. Kennedy Space Center, Personal Interview, October 18, 2012.

Test Methodology

Table 32. Test Methodology for Hypergol Compatibility

Parameters	Per NASA KSC MTB-175-88: N_2O_4, N_2H_4, and MMH
Coupons Per CPC	Three (3) four (4) inch x four (4) inch aluminum foil coupon
Trials Per Coupon	Three (3)
Control Coupons Required For Testing	None
Acceptance Criteria	Slight to Moderate Reactivity Observed: When test data based on visual observations with the unaided eye reveal reactivity (but no ignition) and/or any changes in the visual characteristics, bulk characteristics, and/or surface characteristics of the test sample.

Unique Equipment and Instrumentation

None

Data Analysis and Reporting

Testing was conducted on the different CPC types for both hypergol fuel (hydrazine and monomethylhydrazine) and simulated oxidizer (using concentrated nitric acid) for compatibility purposes. As this work was for preliminary down-select purposes only, drop-testing for screening purposes was conducted on each of the various CPCs to look for signs of gross incompatibilities as defined in with NASA KSC MTB-175-88. This includes, but is not limited to, smoking, bubbling, solubility, charring, and/or color changes. Modifications were made to the procedure in NASA KSC MTB-175-88 as the CPCs were tested wet (uncured). Samples were tested for duration of 10 minutes. Temperature was monitored throughout the testing process using a Fluke Ti30 Thermal Imager and the pre-exposure and maximum temperature reading were recorded from each set of samples. The temperature was recorded for only a single sample, as the thermal imager takes a temperature reading from only a single point, however all 3 samples were in the field of view for the imager so each sample could be monitored for any temperature spikes visually.

Almost all of the samples tested exhibited at least a small temperature increase, as was expected. The results are listed in Table 33,

Table 34 and Table 35. For the simulated oxidizer results, two CPCs, EcoLine 3690 and Zerust Axxanol 46-BIO, exhibited some smoking and color change when exposed to HNO_3. EcoLine 3690 was grossly incompatible with the oxidizer simulant, and exhibited bubbling, smoking, and vigorous reaction when the nitric acid was added. Varying degrees of color change only was noted for six different CPCs: Corrosion X, WD-40, NAVGUARD II, EcoLine 3690, Bio-Medium Preservative Lubricant, Nox-Rust 3100, and Bio-Acid Fume

Rust Preventative Fluids. This color change was not considered detrimental. The remaining six CPCs had no reaction to HNO_3.

For the hydrazine-exposed samples, a few exhibited color changes (Corrosion X, NAVGUARD II, Bio-Acid Fume Rust Preventative, EcoLine Heavy Duty Grease, and WRL). VpCI 368 also had a small amount of bubble formation (not continuous). None of the hydrazine-exposed samples exhibited gross material incompatibility. Fluid Film and EcoLine Heavy Duty Grease registered the highest ΔT(°F) (8.3 °F and 7.5 °F, respectively) upon exposure to hydrazine.

For the samples exposed to monomethylhydrazine, the majority of the samples had a small amount of bubble formation (MX4, EcoLine 3690, Zerust Axonol 46-Bio, Bio-Medium Preservative Lubricant, Fluid Film, Ardrox AV-30, Nox-Rust 3100, and Bio-Acid Fume Rust Preventative). NAVGUARD II exhibited a color change, and MX4 and VpCI 368 showed temperature decrease upon exposure to monomethylhydrazine. None of the monomethylhydrazine-exposed samples exhibited gross material incompatibility.

Table 33.Results from simulated oxidizer testing of CPCs (using HNO_3)

CPC Type	T_i (°F)	T_f (°F)	ΔT(°F)	Observations
Blank	71.2	70.5	-0.7	None. Temperature decreased due to evaporation
Corrosion X Aviation (Control)	71.2	73.2	2.0	Color change; clear → yellow
WD-40 (for comparison)	72.0	74.2	2.2	Slight color change; clear → opaque
NAVGUARD II (for comparison)	71.4	73.4	2.0	Color change; green → yellow
MX3 (for comparison)	71.0	72.5	1.5	None
EcoLine 3690 (candidate)	72.3	75.4	3.1	Smoking, bubbling. Color change; red → orange
Zerust Axxanol 46-BIO (candidate)	71.3	74.0	2.7	Small amount of smoking. Color change; yellow → brown
Bio-Medium Preservative Lubricant (candidate)	71.8	75.4	3.6	Color change; pale yellow → dark yellow or orange
Fluid Film (candidate)	72.1	74.7	2.6	None
VpCI 368 (Control)	71.7	74.3	2.6	None
Ardrox AV-30 (for comparison)	70.4	73.7	3.3	None
Nox-Rust 3100 (for comparison)	72.3	75.3	3.0	Slight color change; brown → darker brown

Bio-Acid Fume Rust Preventative Fluids (candidate)	72.5	75.4	2.9	Slight color change; yellow → darker yellow
EcoLine Heavy Duty Grease (candidate)	71.5	75.6	4.1	None
WRL (candidate)	71.5	73.3	1.8	None

Table 34. Results from hydrazine testing of CPCs

Sample ID	T_i (°F)	T_f (°F)	ΔT(°F)	Observations
Blank	72.2	74.3	2.1	Small amount of condensation.
Corrosion X Aviation (Control)	69.0	72.0	3.0	Color change; white spots.
WD-40 (for comparison)	69.5	73.1	3.6	None.
NAVGUARD II (for comparison)	71.0	73.8	2.8	Slight discoloration
MX3 (for comparison)	68.0	69.2	1.2	Immiscible.
EcoLine 3690 (candidate)	69.0	74.1	5.1	None.
Zerust Axxanol 46-BIO (candidate)	67.5	73.2	5.7	None.
Bio-Medium Preservative Lubricant (candidate)	69.0	73.3	4.3	None.
Fluid Film (candidate)	68.8	77.1	8.3	None.
VpCI 368 (Control)	69.8	73.8	4.0	Small amount of bubbles formed
Ardrox AV-30 (for comparison)	70.0	75.2	5.2	None.
Nox-Rust 3100 (for comparison)	69.8	75.0	5.2	Immiscible
Bio-Acid Fume Rust Preventative Fluids (candidate)	70.3	74.1	3.8	Immiscible, slight darkening at interface
EcoLine Heavy Duty Grease (candidate)	68.0	75.5	7.5	Slight discoloration at interface

WRL (candidate)	71.5	75.1	3.6	Color change; clear → white

Table 35. Results from monomethylhydrazine testing of CPCs

Sample ID	T_i (°F)	T_f (°F)	ΔT(°F)	Observations
Blank	70.6	67	-3.6	Small amount of bubbles formed
Corrosion X Aviation (Control)	68.9	70.6	1.7	Immiscible
WD-40 (for comparison)	68.0	70.1	2.1	None
NAVGUARD II (for comparison)	68.4	71.3	2.9	Color change; Green → orange
MX3 (for comparison)	68.0	69.2	1.2	Small amount of bubbles formed; temp reached a low of 66.5
EcoLine 3690 (candidate)	67.8	69.7	1.9	Small amount of bubbles formed
Zerust Axxanol 46-BIO (candidate)	68.5	69.7	1.2	Small amount of bubbles formed
Bio-Medium Preservative Lubricant (candidate)	68.0	69.5	1.5	Small amount of bubbles formed
Fluid Film (candidate)	71.3	72.6	1.3	Minute amount of bubbles formed at interface
VpCI 368 (Control)	71.2	70.3	-0.9	Immiscible
Ardrox AV-30 (for comparison)	69.8	71.1	1.3	Small amount of bubbles formed
Nox-Rust 3100 (for comparison)	69.5	72.0	2.5	Immiscible; small amount of bubbles formed
Bio-Acid Fume Rust Preventative Fluids (candidate)	70.0	71.4	1.4	Immiscible; small amount of bubbles formed
EcoLine Heavy Duty Grease (candidate)	69.5	70.8	1.3	None
WRL (candidate)	69.4	70.5	1.1	Immiscible; small amount of bubbles formed

6.4.3 Upward Flame Propagation

Test description

The purpose of this test was to determine if a material, when exposed to a standard ignition source, will self-extinguish and not transfer burning debris, which can ignite adjacent materials. The specimens were ignited at the bottom by an ignition system and allowed to burn until each self-extinguished.

This test was performed in accordance with NASA-STD-6001, which is defined in ISO mtb-1:2003, Space Systems - Safety and Compatibility of Materials - Part 1: Determination of Forward Flammability of Materials shall be followed for this test, with the following exceptions, clarifications, and additions as stated in NASA-STD-6001.

For this test, the CPCs were applied to AA6051 panels 24 hours prior to the testing. The flammability cabinet is shown in Figure 50, where the flame source's average burn time was certified as 25.009sec and the average flame temperature was 2018.6°F.

Figure 50. Upward Flammability Cabinet Hardware.

Rationale

This test is specified in NASA-STD-6001 and was identified as a testing requirement. Materials intended for use in space vehicles, specified test facilities, and specified ground support equipment (GSE) must meet the requirements of this document.

Test Methodology

Table 36. Test Methodology for Upward Flame Propagation

Parameters	Per NASA-STD-6001; The test method defined in ISO 14624-1:2003 with exceptions.
Coupons Per CPC	Three (3) at 12" x 2.5"
Trials Per Coupon	One (1)
Control Coupons Required For Testing	None
Acceptance Criteria	No test specimen of the five standard-sized specimens burns >6 inches. No test specimen propagates a flame by the transfer of burning debris.

Unique Equipment and Instrumentation

Flame propagation hood.

Data Analysis

The CPC coating had to meet the test acceptance criteria and be considered self-extinguishing, which was governed by meeting both of the following conditions: No test specimen of the five standard-sized specimens burned greater than 6 inches, and no test specimen propagates a flame by the transfer of burning debris. All of the CPC-coated panels passed the Upward Flammability testing. No residue or other visual indications of CPC coating degradation was apparent for any of the CPC types.

7 CONCLUSIONS

Physical Testing:

No critical problems were discovered during the sprayability, removability, or wire compatibility testing. In general, the cold CPCs sprayed more poorly than the CPCs at ambient temperature. The methods for applying the CPCs, either spraying, rolling, or painting, are all practical means for future end use.

Results for viscosity, CPC wettability, CPC hydrophobicity, and functional penetration were reported, although no pass or fail criteria were established based on these results. These results will be used when determining appropriate end-use applications in the upcoming test phases.

Accelerated Chamber and Atmospheric Corrosion Testing:

The accelerated testing included separate UV-only and salt fog-only cycles in a test chamber. The initial UV-only cycle seemed to significantly degrade many of the CPC types. Bio-Medium Preservative Lubricant, Fluid Film, EcoLine Heavy Duty Grease (all environmentally-friendly), and VpCI 368 and Ardrox AV-30 (not environmentally-friendly) were least affected by the UV, while WD-40, MX4, Nox Rust 3100 (not environmentally-friendly) and WRL (environmentally-friendly) were most negativity affected by UV.

CPCs did offer a significant amount of corrosion protection even in the aggressive longer-term six month atmospheric testing performed at KSC’s Beachside Atmospheric Corrosion Test Site. All of the CPC types performed similar to or better than the control on carbon steel. For carbon steel, the CPCs that offered the highest degree of protection from corrosion were Fluid Film and EcoLine Heavy Duty Grease (both environmentally-friendly) and VpCI 368 and Nox Rust 3100 (not environmentally-friendly). A second tier of successful performing CPC types consisted of EcoLine 3690, Zerust Axxanol 46-BIO, Bio-Medium Preservative Lubricant, Bio-Acid Fume Rust Preventative (all environmentally-friendly) and Ardrox AV-30 (not environmentally-friendly).

The CPC types provided different degrees of corrosion protection on the stainless steel and aluminum alloys than on the carbon steel, with different CPCs even affecting the aluminum alloy types in a different manner. Only one CPC type, VpCI 368, protected all aluminum alloys from pitting corrosion. CPC types VpCI 368, Nox Rust 3100, and Bio-Medium Preservative Lubricant all performed significantly better than the other CPC types in the prevention of crevice and galvanic corrosion.

The aggressive wire-on-bolt atmospheric galvanic corrosion testing showed that Corrosion X, Bio-Medium Preservative Lubricant, Fluid Film, and Nox-Rust 3100 provided the most effective protection from galvanic corrosion.

No CPC types induced stress corrosion cracking.

NASA Spaceport Environment Compatibility:

All of the CPC types met the NASA flammability requirements. All but two of the CPC types, EcoLine 3690 and Zerust Axxanol 46-BIO, met all of the hypergolic fluids compatibility requirements. The liquid oxygen compatibility requirement was determined to be impractical, as currently no CPC-type materials are foreseen to be in contact with the pressure vessels. No critical incompatibility issues were discovered through the NASA spaceport environment compatibility testing.

8 ACKNOWLEDGEMENTS

The technical support of the following individuals is gratefully acknowledged: Dr. Tracy Gibson, ESC Polymer Science Laboratory, for his help with wire compatibility testing; Dr. Bobby Devor and Brint Bauer, ESC Applied Chemistry Laboratory, for their help with hypergol compatibility testing: Dr. Steve Trigwell, ESC Surface Systems, for his help with contact angle measurements: Dean Lewis and Brian Cheshire, NASA Materials Testing, for their help with upward flame propagation testing; and Virginia Ward, NASA Materials and Processes, for her help with liquid oxygen compatibility testing.

Reference Documents

The documents in Table 37 were referenced in the development and execution of this Test Plan.

Table 37. Test and Evaluation Reference Listing

Reference Document	*Title*	*Test*
ASTM D 445	Standard Test Method for Kinematic Viscosity of Transparent and Opaque Liquids (and Calculation of Dynamic Viscosity)	Viscosity
ASTM D 610	Evaluating Degree of Rusting on Painted Steel Surfaces	Long-term Beachside Atmospheric Exposure, Alternating Seawater Spray Testing
ASTM D 2512	Compatibility of Materials with Liquid Oxygen (Impact Sensitivity Threshold and Pass-Fail Techniques)	LOX Compatibility
ASTM D 4414	Standard Practice for Measurement of Wet Film Thickness by Notch Gages	Application Characteristics
ASTM D 5894	Standard Practice for Cyclic Salt Fog/UV Exposure of Painted Metal, (Alternating Exposures in a Fog/Dry Cabinet and a UV/Condensation Cabinet)	Cyclic Salt Fog
ASTM D 7334	Standard Practice for Surface Wettability of Coatings, Substrates and Pigments by Advancing Contact Angle Measurement	Wettability of CPC, Hydrophobicity of CPC on Substrate
ASTM F 1110	Standard Test Method for Sandwich Corrosion Test	Crevice Corrosion
ASTM G 1	Standard Practice for Preparing, Cleaning, and Evaluating Corrosion Test Specimens	Long-term Beachside Atmospheric Exposure, Alternating Seawater Spray Testing

Reference Document	*Title*	*Test*
ASTM G 33	Standard Practice for Recording Data from Atmospheric Corrosion Tests of Metallic-Coated Steel Specimens	Long-term Beachside Atmospheric Exposure
ASTM G 44	Standard Practice for Evaluating Stress Corrosion Cracking Resistance of Metals and Alloys by Alternate Immersion in 3.5 % Sodium Chloride Solution	Alternating Seawater Spray Testing
ASTM G 46	Standard Guide for Examination and Evaluation of Pitting Corrosion	Long-term Beachside Atmospheric Exposure, Alternating Seawater Spray Testing
ASTM G 47	Standard Test Method for Determining Susceptibility to Stress-Corrosion Cracking of 2XXX and 7XXX Aluminum Alloy Products	Stress Corrosion Cracking
ASTM G 50	Standard Practice for Conducting Atmospheric Corrosion Tests on Metals	Long-term Beachside Atmospheric Exposure, Alternating Seawater Spray Testing, Crevice Corrosion, Galvanic Corrosion via Fasteners, Wire on Bolt Atmospheric Galvanic Corrosion,
ASTM G 78	Standard Guide for Crevice Corrosion Testing of Iron-Base and Nickel-Base Stainless Alloys in Seawater and Other Chloride-Containing Aqueous Environments	Crevice Corrosion
ASTM G104	Standard Test Method for Assessing Galvanic Corrosion Caused by the Atmosphere	Galvanic Corrosion via Fasteners
ASTM G 116	Standard Practice for Conducting Wire-on-Bolt Test for Atmospheric Galvanic Corrosion	Galvanic Corrosion, Wire on Bolt

Reference Document	*Title*	*Test*
ISO 14624-1:2003	Space systems - Safety and compatibility of materials - Part 1: Determination of upward flammability of materials	Flammability
KSC Report MTB-175-88	Procedure For Casual Exposure Of Materials To Hypergolic Fluids	Hypergol Compatibility
MIL-PRF-6001F	Performance Specification Corrosion Preventive Compounds, Water Displacing, Ultra-thin Film	Application Characteristics, Functional Penetration, Wire Compatibility, Removability
NASA-STD-6001	Flammability, Odor, Offgassing, and Compatibility Requirements and Test Procedures for Materials in Environments that Support Combustion	LOX Compatibility, Hypergol Compatibility, Flammability