ARL-TR-8905 ● Feb 2020

Evaluation of Commercial-off-the-Shelf Chrome-Free Pretreatments for Aluminum Substrates

by Daniel Pope, Thomas Considine, Fred Lafferman, and Christopher Miller

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REPORT DOCUMENTATION PAGE						*Form Approved* *OMB No. 0704-0188*

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1. REPORT DATE *(DD-MM-YYYY)*	**2. REPORT TYPE**	**3. DATES COVERED (From - To)**
February 2020	Technical Report	3 March 2014 – 9 March 2018

4. TITLE AND SUBTITLE	**5a. CONTRACT NUMBER**
Evaluation of Commercial-off-the-Shelf Chrome-Free Pretreatments for Aluminum Substrates	
	5b. GRANT NUMBER
	5c. PROGRAM ELEMENT NUMBER
6. AUTHOR(S)	**5d. PROJECT NUMBER**
Daniel Pope, Thomas Considine, Fred Lafferman, and Christopher Miller	W911NF1820271
	5e. TASK NUMBER
	5f. WORK UNIT NUMBER
7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES)	**8. PERFORMING ORGANIZATION REPORT NUMBER**
CCDC Army Research Laboratory* ATTN: FCDD-RLW-MC Aberdeen Proving Ground, MD 21005-5066	ARL-TR-8905
9. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS(ES)	**10. SPONSOR/MONITOR'S ACRONYM(S)**
	11. SPONSOR/MONITOR'S REPORT NUMBER(S)

12. DISTRIBUTION/AVAILABILITY STATEMENT

Approved for public release; distribution is unlimited.

* The work outlined in this report was performed while the Weapons and Materials Research Directorate (WMRD) was a directorate within the US Army Research Laboratory. As of the date of report publication, WMRD is now reporting directly to the US Army Combat Capabilities Development Command Army Research Laboratory (ARL).

13. SUPPLEMENTARY NOTES

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14. ABSTRACT

The objective of this work was to determine what products were commercially available that were chrome-free and could be used in place of hexavalent or trivalent chromium conversion coatings for both aviation and ground equipment as part of the Chemical Agent Resistant Coating (CARC) system. This work also looked at alternative ways to test chrome-free conversion coatings since the methods in MIL-DTL-81706 are specifically for chrome-containing systems. The results of this testing conducted at the US Army Research Laboratory provided critical data that showed there are chrome-free commercially available products that can perform as part of the CARC system, and that the use of a galvanic panel with titanium and stainless steel fasteners can quickly delineate good-performing coatings systems in a lab setting. Outdoor corrosion testing at Cape Canaveral Air Force Station showed similar results as lab testing with the galvanic panels.

15. SUBJECT TERMS

conversion coating, zirconium, galvanic, adhesion, corrosion, stainless steel, titanium, pretreatment, coating system, deoxidizer, cleaner, aluminum

16. SECURITY CLASSIFICATION OF:			**17. LIMITATION OF ABSTRACT**	**18. NUMBER OF PAGES**	**19a. NAME OF RESPONSIBLE PERSON**
					Daniel Pope
a. REPORT	**b. ABSTRACT**	**c. THIS PAGE**			**19b. TELEPHONE NUMBER (Include area code)**
Unclassified	Unclassified	Unclassified	UU	51	(410) 306-1688

Standard Form 298 (Rev. 8/98)
Prescribed by ANSI Std. Z39.18

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1. Introduction

The Chemical Agent Resistant Coating (CARC) system application and inspection specification, MIL-DTL-53072,[1] requires that metal surfaces on tactical assets be treated to improve adhesion and corrosion resistance prior to coating with an epoxy primer and a camouflage topcoat. However, there are many problems associated with currently fielded legacy pretreatment technologies. Problems inherent with the chromate conversion coatings begin with the potential exposure to hazardous materials of paint, pretreatment, blast media applicators during the application and removal processes, and the associated Occupational Safety and Health Administration (OSHA) impacts. Additionally, continually increasing costs associated with disposal of wastes contaminated with hexavalent chromium (Cr^{6+}) and the Resource Conservation and Recovery Act impacts add to the issues experienced when fielding legacy pretreatments.

A major issue is the potential for near-term obsolescence, as these materials are facing increasing regulatory scrutiny, with several recently revised specifications already eliminating their use. Verification of suitable alternatives will be continue to be crucial as the Cr^{6+}-containing products are prohibited. Numerous international Cr^{6+} regulations affect the use of coatings containing these compounds and thus already impact maintenance and repair in overseas posts. The US Army needs to act proactively to be ready when additional regulations outright ban these products.

The primary objective of this program is to demonstrate and validate novel Cr^{6+}-free pretreatment technologies in relevant Department of Defense (DOD) environments. There is a need to implement innovative and cost-effective replacement technologies to address the multiple health, safety, and compliance issues associated with the legacy systems while maintaining military readiness for national defense. In addition, the new technology must have the following attributes: 1) compatibility with original equipment manufacturer/depot infrastructure, 2) corrosion performance equivalent to (or better than) current Cr^6+- and phosphate-based pretreatments, 3) broad compatibility with the current suite of military coatings, and 4) compatibility with all substrates used by DOD. Although the primary application will be on aluminum (Al) substrates, these technologies will also be evaluated for performance on additional substrates, such as ferrous alloys, mixed alloys, and composites. The technologies to be demonstrated and validated through asset field exposure will encompass zirconium oxide/fluoro-chemistry, rare-earth chemistry, and silane/sol-gel chemistry. These chemistries will be modified to meet the application criterion with respect to spray and immersion for aircraft, immersion for components of ground-support equipment, and assets incorporating multiple substrates, including ferrous and Al alloys.

The following are the regulatory drivers that serves as justification for testing, validation, and demonstration of non- Cr^{6+} pretreatment coatings:

1) OSHA Regulation 1910.1026. *Regulatory Requirements and Allowable Exposures to Cr^{+6}*, dated 2013.[2]

2) Deputy Under Secretary of Defense (Acquisition, Technology and Logistics). *Memorandum for Minimizing the Use of Hexavalent Chromium (Cr^{+6})*, dated 8 April 2009.[3]

3) Deputy Under Secretary of Defense Directive-Type Memorandum 12-003. *Control and Management of Surface Accumulations from Lead, Hexavalent Chromium, and Cadmium Operations*, dated 18 April 2012.[4]

4) Defense Pricing and Contracting (DPC). *Defense Federal Acquisition Regulation Supplement and Procedures, Guidance, and Information*, dated 23 January 2006[5]:

   > Prohibition (223.7302): As provided in Policy 223.73, no DoD contract may include a specification or standard that results in a deliverable containing hexavalent chromium or the use of hexavalent chromium during sustainment phases of any aviation system. This prohibition is in addition to any imposed by the Clean Air Act and applies to all DoD contracts awarded after April 8, 2009, regardless of the place of performance.
   >
   > Exceptions (223.7303): The prohibition in 223.7302 does not apply if the use of hexavalent chromium in a specification or standard is ***specifically authorized at a level no lower than a general or flag officer or a member of the Senior Executive Service from the Program Executive Office or equivalent level***, in coordination with the component Corrosion Control and Prevention Executive. The prohibition in 223.7302 does not apply to legacy systems and their related parts, subsystems, and components that already contain hexavalent chromium. However, alternatives to hexavalent chromium should be considered during aviation system modifications, new procurements of legacy systems, or maintenance procedure updates.

5) Registration, evaluation, authorization, and restriction of chemicals[3] will potentially impact maintenance and repair in the near future as many of the chemicals used (precursor and final products) reach their "sunset" dates. These dates will eliminate any future production as well as severely limit or totally restrict Army ability to transport, store, use, and dispose of these chemicals in the European Union without specific authorization/exemption.

6) Memorandum from John Young, Under Secretary of Defense for Acquisition, Technology and Logistics to Secretaries of Military Departments: *Minimizing the Use of Hexavalent Chromium*, dated 8 April 2008.[6]

7) US Army Aviation and Missile Command. *Reduction of Toxic Materials in Army Surface Finishing Processes: Environmental Requirement and Technology Assessment*, dated 1 December 2010.[7]

8) US Army. *Army Environmental Requirements and Technology Assessment Requirement PP-2-02-04: Toxic Metal Reduction in Surface Finishing of Army Weapon Systems*, specifically the requirement for an alternative to Alodine (conversion coating), dated 13 December 2007.[8]

This program for Cr^{6+}-free conversion coatings will eliminate the use of both Cr^{6+} and trivalent chromium (Cr^{3+}) in the pretreatment of Al substrates prior to the application of the CARC coating system. This includes limiting the waste stream generated from the application and the removal of existing legacy pretreatments from depot and corrosion repair location operations. The alternative chrome-free pretreatments will be validated for corrosion resistance against the baseline chromate-containing pretreatments through laboratory evaluations, outdoor validations, and a demonstration program on representative assets.

The purpose of this effort is to test chrome-free surface treatments, as part of the coating stack-ups commonly seen on Army aviation assets, on galvanic panels that include Al, titanium (Ti), and stainless steel (SS). The chrome-free treatments will be compared with currently fielded legacy materials approved to MIL-DTL-81706 standard,[9] including Cr^{6+} and Cr^{3+} products. The best-performing chrome-free surface treatments will be selected to be demonstrated at Corpus Christi Army Depot (CCAD) in Texas and the Theater Aviation Sustainment Maintenance Group in Groton, Connecticut.

2. Technical Approach

Three technologies were laboratory tested and evaluated. The best-performing products were demonstrated as an alternative to chromate-containing conversion coatings at CCAD. All products being tested are mature technologies currently being manufactured and supplied to either industrial or military users, but not yet approved for Army application. The following is a brief description of each technology:

Zirconium Pretreatments: Provide a high-quality, continuous, zirconium-based pretreatment on multiple types of ferrous, zinc, and Al substrates by immersing the metal into a dilute solution of fluorozirconic acid (FZA) at ambient temperature for 30–120 s. The rheology will be modified for spray application to allow for the ability to hang onto vertical surfaces, which is a requirement for aviation assets. The dilute, aqueous FZA pretreatment bath has a pH of 4.5 and does not contain any volatile organic compounds. During the treatment process, the substrate is etched slightly, which results in a pH increase at the substrate/solution interface. This change in pH results in the precipitation and subsequent bonding of zirconium oxide and additives to the surface of the substrate. The chemistry does not contain any regulated heavy metals such as chromium or nickel. Other environmental benefits relative to existing zinc phosphate treatments include 1) significant reduction (>90%) in the amount of sludge byproducts produced, 2) reduced energy consumption since the process can operate at ambient temperatures and fewer stages required, and 3) reduced material usage since the coating thicknesses are only 20–50 nm. This technology is now being used within the automobile industry as a replacement for both zinc phosphate and Cr^{6+} by immersion technology.

Rare Earth Conversion Coating (RECC): Cerium (Ce) compounds have proved to be effective corrosion inhibitors on Al alloys and, with modification, on ferrous substrates. Ce-based conversion coatings are capable of inhibiting the corrosion of Al alloys and meet the requirements established for conversion coatings in MIL-DTL-81706.[9] Research through Strategic Environmental Research and Development Program WP-1618[10] showed that Ce-based conversion coatings not only protected the underlying substrate directly beneath the coating, but also inhibited corrosion beneath the coating by depositing Al oxide in the crevices of the Al. The RECC has been approved by the Air Force for F-15 aircraft by both spray and immersion application. This technology is also being used within the heavy equipment industry as a pretreatment for ferrous and Al substrates.

Metal Oxide/Silane Pretreatment: This spray-in-place technology specific for Al normally does not require rinse-off. These technologies have increased the adhesion properties of subsequent coatings and produce excellent corrosion resistance as part of a full CARC stack-up system. Application is primarily by spray or immersion. This technology, used as adhesion promoters, has been approved by the Air Force and Boeing Industries.

The commercial-off-the-shelf (COTS) products were applied to bare test coupons as per manufacturer recommendations. This includes using manufacturer recommendations for cleaning the panel prior to applying the surface treatment. Once applied, the treated coupons were allowed to dry and or cure in accordance with the manufacture's recommendations prior to receiving a primer in accordance with MIL-PRF-23377[11] or MIL-PRF-85582.[12]

3. Experimental Procedure

3.1 Products

Table 1 list the products that were selected for this testing. The products being tested include zirconium products, silanes, and rare-earth chemistries. The controls selected are approved products on the Qualified Products Database for MIL-DTL-81706.[9] The Type I material was Henkel's Bonderite (formerly Alodine) 1200 and is a Cr^{6+}-based conversion coating. The Type II material used was the SurTec 650 Cr^{3+}-based conversion coating.

Table 1 Surface treatments tested

Manufacture	Material	Chemistry
Control	81706 Type I	Cr^{6+} conversion coating
Control	81706 Type II	Cr^{3+} conversion coating
Henkel	5700	Zirconium pretreatment
MacDermid	Iridite NCP	Zirconium pretreatment
PPG	X-Bond 3000	Zirconium pretreatment
3M	AC 131	Silane pretreatment
Deft	DFM IV/DFS-II	RECC
Deft	1043/3024	RECC
Deft	1060/3007	RECC
Pantheon	PreKote	Silane pretreatment

3.2 Coatings

The surface treatments in the test were meant to be part of a coating stack-up. To simulate what would be seen on an Army aviation asset, one of three primers and one topcoat were used. Table 2 lists the coatings that were used in this testing. Figure 1 shows a diagram of the galvanic panel, specifying which areas that were coated with only primer and areas that were coated with primer and topcoat.

Table 2 List of primers and topcoats used in testing

Specification	Details
MIL-PRF-23377 Class C	Primer: strontium chromate, high solids, solvent-based epoxy
MIL-PRF-23377 Class N	Primer: Cr^{6+}-free, high solids, solvent-based epoxy
MIL-PRF-85582 Class N	Primer: Cr^{6+}-free, high solids, water-reducible epoxy
MIL-DTL-53039[12] Type IX	Topcoat: solvent-based urethane

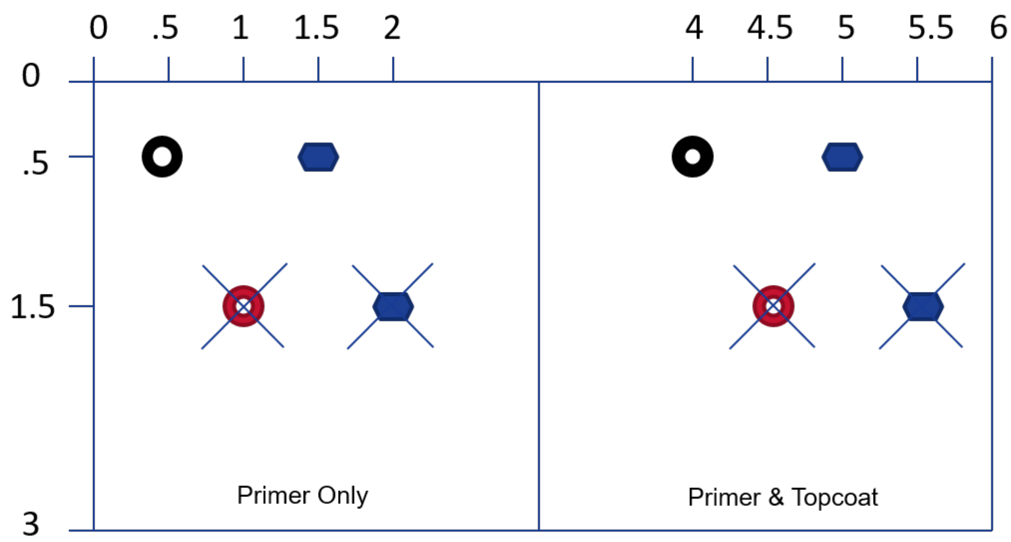

Fig. 1 Stainless steel fasteners. Black circles represent holes, drilled for additional Ti, not used as part of this test.

3.3 Test Coupons

The test panel selected was presented by the US Naval Air Systems Command (NAVAIR) at ASETSDefense 2012.[13] The setup presented incorporated Ti and SS fasteners. This creates galvanic incompatibility between Al and the fasteners, thus creating more stress on the pretreatment and coating system. These failures better match the failures seen in the field that originate from fasteners, rivets, or faying surfaces. Legacy pretreatments were not tested for this type of stress.

All products were tested over 2024-T3 Al panels purchased from Q-Lab. For adhesion testing, a 4- × 6- × 0.063-inch panel was used. The surface had a mill finish. There was no chemical treatment on the panel prior the application of the test products. For the galvanic test, large sheets of 1/4-inch-thick 2024-T3 Al were cut into 3- × 6-inch coupons. The panels were drilled eight times using a #11 drill bit. Figure 1 shows the location of the holes in the panel. After the holes were drilled, all holes and edges of the panel were deburred. Once the panels were fully coated, the holes 1.5 inch from the top received an "X" scribe through the coatings, with the center of the X as the center of the bore hole. The length of each scribe was approximately 1 inch. Then fasteners were dry installed into the panel in positions noted in Fig. 1. The torque values for each type of fastener are found in Table 3. A washer of the same material as the bolt was used on the front of the panel to ensure consistent contact with the panel. All of the fasteners were torqued from the back of the panel to prevent any unnecessary damage to the coating.

Table 3 Fasteners used in testing

Fastener material	Size	Torque	Washer circumference
316 SS	10-32 × 3/4-inch hex machine screw	100 ± 5 inch oz	34.5 mm
6AL-4V Ti	10-32 × 3/4-inch socket cap screw	100 ± 5 inch oz	34.5 mm

3.4 Cross-Hatch Adhesion and Tape Test

This test was done in accordance with ASTM D3359 Method B,[15] as stated in the specifications for MIL-DTL-53039E (3.6.7).[13] Using a cutting-wheel-style tool with six blades, a lattice pattern was cut through the coating down to the substrate with the parallel scribes 2 mm apart. After the lattice had been cut, the testing area was gently brushed to remove loose particles, and a certified, nonexpired pressure-sensitive tape was placed over the lattice. The tape was rubbed with even pressure using a pencil eraser to ensure complete adhesion. After 90 ± 30 s, the tape was removed by pulling the tape straight back at as close as possible to 180° in a single motion. The test area was inspected and given a rating based upon comparison with the paint removal classifications illustrated in the test method.

3.5 Corrosion Testing

Cyclic corrosion testing was completed in accordance with GMW14872.[16] Testing occurred in a calibrated Autotechnology Cyclic Test Chamber, Model NC90 (Fig. 2), for a duration of 21 cycles. Fog deposition rates were recorded daily by chamber operators, and temperature/humidity data are recorded internally by the chamber. A premixed, certified salt solution (NaCl 0.9%, $CaCl_2$ 0.1%, and $NaHCO_3$ 0.075%) was used during spray cycles, and the humidity cycles used laboratory-supplied deionized water, conforming to ASTM D1193[17] Type IV. Chamber operation was validated using standard mass-loss coupons. The galvanic panels were oriented at a 15° incline. At the end of 21 cycles, all panels were pulled from the chamber. Inspections were made at the end of the exposure. A measuring optical magnifier was used examine the panels. Each panel was rated on the primer-only side and the primer with topcoat side. First, areas away from the fasteners and scribes were rated for blisters in field (BIF) in accordance with Table 4. Each fastener received two ratings for corrosion. The first represented the maximum growth of blisters radially from the washer interface. These ratings were based upon the rating scale from ASTM D1654[18] Procedure A and presented in the column labeled "Blister Rating". When blisters were present, the length of contact with the washer was measured and presented in the column "AMT (mm)". This measurement approaches a maximum of 30 mm. Finally, the maximum creep from scribe was captured in accordance with ASTM D1654 Procedure B.

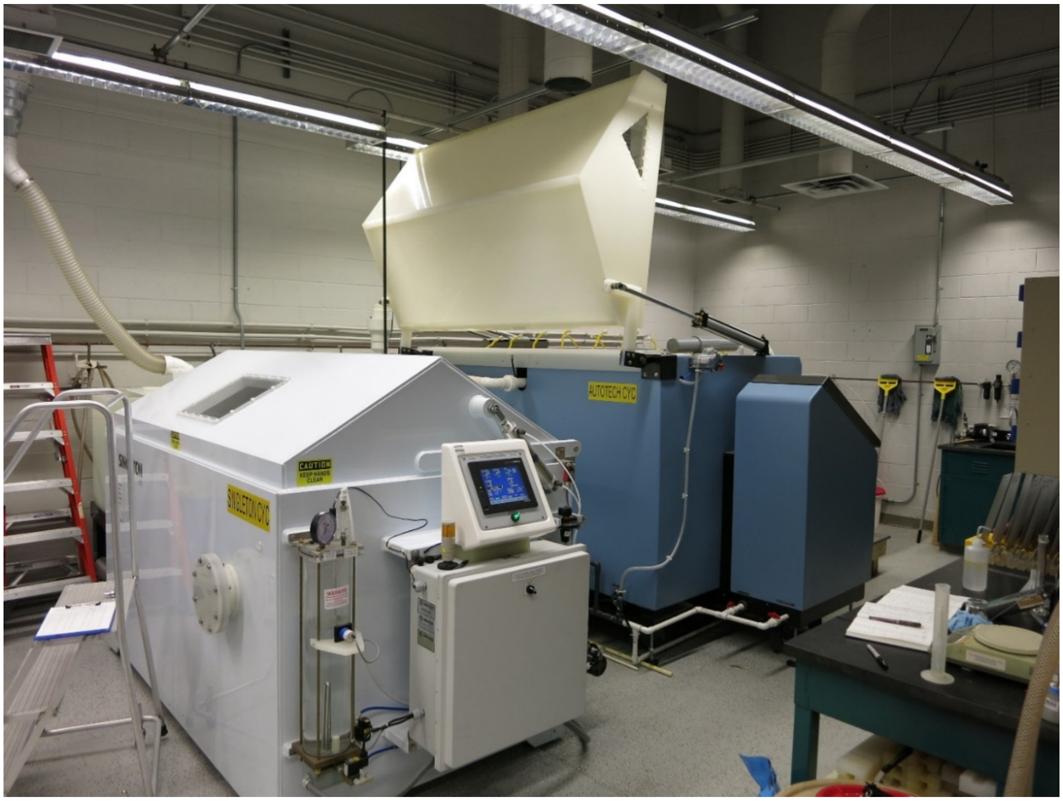

Fig. 2 Autotechnology cyclic test chamber, Model NC90

Table 4 Rating for unscribed areas with color coding: green (acceptable), yellow (borderline, fail), and red (failure)

Area failed (%)	Rating number
No failures	10
>0 to 1	9
2 to 3	8
4 to 6	7
7 to 10	6
11 to 20	5
21 to 30	4
31 to 40	3
41 to 55	2
56 to 75	1
> 75	0

3.6 Outdoor Exposure Testing at Cape Canaveral Air Force Station (CCAFS)/US Army Tank-automotive and Armaments Command (TACOM)

The best-performing pretreatments from the GMW 14872[16] test were selected to be tested outdoors. These samples were shipped from the US Army Combat Capabilities Development Command Army Research Laboratory (ARL) at Aberdeen Proving Ground, Maryland, to Florida for outdoor testing at the ARL Outdoor Exposure at CCAFS/TACOM. Cape Canaveral is considered one of the most corrosive environments in the continental United States. The corrosion rate observed by ASTM International is 5.17 mil per year (mpy) on standard steel mass-loss coupons at 55 m inland. For this reason, ARL selected this outdoor exposure facility for much of its outdoor testing. The ARL corrosion racks are set at approximately 170 and 220 m inland and parallel to the ocean, facing southeast (Fig. 3). The average corrosion rate in mil per year observed by ARL since 2011 on standard mass loss coupons is 5.4 mpy at 170 m inland. The test panels were scribed in accordance with ASTM D1654[18] and held in place on wood or composite racks with nylon standoffs and SS fasteners (Fig. 4). The coupons are inspected at each individual fastener and evaluated quarterly in accordance with ASTM D1654 for both corrosion creep from the scribe when a scribe was present and blistering.

Fig. 3 Satellite image of CCAFS/TACOM outdoor exposure site in relation to ocean

Fig. 4 Racks at CCAFS/TACOM outdoor exposure site with galvanic panels on the top three rows

4. Results

4.1 Application

One of the most important facets of this program was to determine if the recommended application process can be transitioned into the Army repair depots. Currently at CCAD, MIL-PRF-81706[9] is mopped onto the airframes due to restrictions on spraying Cr^{6+} materials at the facility. Ideally, a drop-in process would involve spray or immersion chemical cleaning and deoxidizer to prep the surface for the pretreatment. The ideal pretreatment would be spray or immersion application depending on the use, size, and geometry of the item. For the test matrix, the manufacturer's recommended procedures were used to prep and pretreat the panel for all of the pretreatments. The controls were applied at NAVAIR in Patuxent River, Maryland, and thus their internal process was used to clean and pretreat the panels. This process includes the panels being immersed in a bath consisting of 10% sodium hydroxide (NaOH) after cleaning but prior to the deoxidizer. Using NaOH chemically mills the panel and allows the oxidation on the panel to be easily removed in the deoxidizer. This extra step creates a pristine surface prior to pretreatment. This step did not happen with any of the products tested in this matrix besides the control panels.

Details of each products application process can be found in Table 5. All of the possible ways of applying each pretreatment are listed. The method used for this testing is marked in bold in the table. It was recommended for the panel testing that all vendors used a solvent wipe prior to using a deoxidizer on the panels. For most of the products, the required processes tentatively appear to meet the needs of CCAD. A few steps were flagged as possible roadblocks for future transition to the field. These steps are highlighted in yellow in Table 5. The AC-131 and PreKote recommended a mechanical deoxidization. This was a concern for helicopter skins because it could be a time-consuming process when scaled-up for use at the depot and could cause loss in metal mass. PreKote was recommended to be scrubbed into the substrate with a green Scotch-Brite heavy duty scour pad. This would be very time-consuming for a full airframe, especially when performed in conjunction with the aforementioned mechanical deoxidation step. Therefore, a hand-scrub process would not be feasible to transition to CCAD.

Table 5 Application details for each pretreatment

Manufacturer	Material	Cleaned	Deoxidizer	Pretreatment application	Application location	Primer/topcoat
Control	81706 Type I	Immersion	Immersion chemical	Mop/spray/immersion	NAVAIR	NAVAIR
Control	81706 Type II	Immersion	Immersion chemical	Spray/immersion	NAVAIR	NAVAIR
Henkel	5700	Solvent wipe	Spray chemical	Spray/immersion	Henkel	ARL
MacDermid	MacDermid Iridite NCP	Solvent wipe	Spray chemical	Spray/immersion	ARL	ARL
PPG	PPG X-Bond 3000	Solvent wipe	Spray chemical	Spray	PPG	PPG
3M	3M AC-131	Solvent wipe	**Mechanical 150-grit sander**	Spray	ARL	ARL
Deft	DFM IV/DFS-II Series	Solvent wipe	Immersion chemical	Immersion	Deft	Deft
Deft	Deft 1043/3024	Solvent wipe	Spray chemical	Spray	Deft	Deft
Deft	Deft 1060/3007	Solvent wipe	Spray chemical	Spray	Deft	Deft
Pantheon	PreKote	Solvent wipe	**Scotch-Brite in solution**	**Scrub in**	ARL	ARL

In most cases, the panels were pretreated, primed, and top-coated in the same location. This ensured there were no issues with contamination and pretreatments going unprimed for extended periods of time. This was not the case for the Henkel products. Henkel prepared panels at their facility in Madison, Michigan, and shipped them to ARL to receive primer and topcoat. This presented greater opportunity for these panels to be contaminated. It also meant that these panels stayed in a pretreatment-only state for a longer time than all of the other sets, creating a worst-case, out-of-spec scenario.

4.2 Adhesion

Adhesion results are presented in Table 6. Passing for these materials is considered a rating of 4B (<5% paint loss) or better. Overall results showed less than 5% paint loss in the paint adhesion test completed on most panels. For the most part, there was not a difference in adhesion with the primers. The nonchrome primers adhered as well to the pretreatments as the chrome-containing primer. One panel in the set pretreated with the Henkel 5700 and primed with MIL-PRF-85582[12] Class N had a rating of 3B. The other two samples in that set had ratings of 4B. Because of the higher ratings with that stack-up and the consistent ratings with the other primers, this was not considered to be a failure for that system but rather a possible issue with the prep of that particular panel.

Table 6 Adhesion results

Material	Mil-PRF-23377 Class C			MIl-PRF-23377 Class N			MIl-PRF-85582 Class N		
	Result 1	Result 2	Result 3	Result 1	Result 2	Result 3	Result 1	Result 2	Result 3
81706 Type I	5B	5B	4B	5B	4B	4B	5B	5B	5B
81706 Type II	4B	5B	5B	5B	5B	5B	5B	4B	4B
Henkel 5700	5B	5B	5B	5B	5B	4B	**3B**	4B	4B
MacDermid Iridite NCP	4B	4B	5B	4B	4B	5B	5B	5B	4B
PPG X-bond 3000	4B	4B	5B	5B	5B	4B	4B	5B	5B
3M AC 131	5B	5B	5B	5B	5B	5B	5B	5B	5B
Deft DFM IV/DFS-II	5B	4B	4B	4B	4B	5B	4B	5B	4B
Deft 1043/3024	5B	4B	4B	4B	4B	5B	5B	4B	4B
Deft 1060/3007	4B	5B	5B	5B	5B	4B	5B	5B	4B
Pantheon PreKote	5B	5B	5B	5B	5B	5B	5B	5B	5B

4.3 Corrosion

Corrosion results for the galvanic panels can be found in Tables 7–15. The images of these panels can be found in Figs. 3–10. In the tables, results that were determined to be acceptable were shaded green. Results that were borderline but not quite acceptable were shaded yellow. Areas that were considered failures were shaded red. For the amount of corrosion present around the fasteners, there were many results that were unshaded. It was determined that more testing and rating need to be conducted in this fashion to determine what is and is not acceptable. At the same time, results of no corrosion present around the fastener is acceptable and was shaded green. Results with 15 mm of corrosion (approximate 50% of the circumference of the washer) was deemed to be unacceptable and shaded red. For creep ratings in Tables 13–15, anything that was rated as a 7 (<2 mm of creep) or greater as per ASTM D1654[18] was shaded green. Creep ratings that were classified as 5 or 6 (2–5 mm of creep) were shaded yellow for borderline failure. Creep ratings that were classified as 4 or lower (>5 mm of creep) were shaded red for failures.

Table 7 21-cycle galvanic panel results: MIL-PFR-23377[11] Class C primer-only area

Pretreatment	Panel	Primer						
		BIF	Ti bolt, scribed		SS bolt, scribed		SS bolt, no scribe	
			Blister rating	Amt (mm)	Blister rating	Amt (mm)	Blister rating	Amt (mm)
81706 Type I	1	10	10	…	7	2.5	10	…
81706 Type I	2	10	10	…	10	…	10	…
81706 Type I	3	10	10	…	10	…	9	…
81706 Type II	1	10	10	…	10	…	6	2
81706 Type II	2	10	10	…	8	1.5	10	--
81706 Type II	3	10	10	…	10	…	8	6
Henkel 5700	1	10	10	…	7	…	7	10
Henkel 5700	2	10	10	…	8	2	7	10
Henkel 5700	3	10	10	…	8	6	6	7
MacDermid Iridite	1	10	10	…	9	1	8	2.5
MacDermid Iridite	2	10	10	…	7	3.5	7	1.5
MacDermid Iridite	3	10	7	1	7	12	6	5
PPG Xbond 3000	1	10	8	1	7	9	10	…
PPG Xbond 3000	2	10	8	1	6	14	3	22
PPG Xbond 3000	3	10	7	3.5	7	12	10	…
3M AC131	1	10	10	…	9	6	7	13
3M AC131	2	10	10	…	7	3	8	3
3M AC131	3	10	10	…	8	7	7	5
Deft DFM IV/DFS	1	10	7	1	6	8	10	…
Deft DFM IV/DFS	2	10	7	3	5	11	4	3
Deft DFM IV/DFS	3	10	10	…	5	19	9	1
Deft 1043/3024	1	10	6	4	6	12	10	…
Deft 1043/3024	2	10	8	1	6	12	8	6
Deft 1043/3024	3	10	7	3	6	5	7	13
Deft 1060/3007	1	10	10	…	8	3.5	7	9
Deft 1060/3007	2	10	9	1	7	4.5	10	…
Deft 1060/3007	3	10	10	…	8	1	10	…
Pantheon PreKote	1	10	10	…	8	9	6	4
Pantheon PreKote	2	10	10	…	8	4	10	…
Pantheon PreKote	3	10	10	…	6	7	10	…

Table 8 21-cycle galvanic panel results: MIL-PFR-23377[11] Class C primer with topcoat area

Pretreatment	Panel	Primer with topcoat						
			Ti bolt, scribed		SS bolt, scribed		SS bolt, no scribe	
		BIF	Blister rating	Amt (mm)	Blister rating	Amt (mm)	Blister rating	Amt (mm)
81706 Type I	1	10	10	…	7	3.5	10	…
81706 Type I	2	10	10	…	10	…	10	…
81706 Type I	3	10	10	…	10	…	10	…
81706 Type II	1	10	10	…	10	…	10	…
81706 Type II	2	10	9	1.5	7	6.5	10	…
81706 Type II	3	10	10	…	10	…	10	…
Henkel 5700	1	10	10	…	7	4	7	24
Henkel 5700	2	10	10	…	7	6	7	13
Henkel 5700	3	10	7	2	6	4	7	11
MacDermid Iridite	1	10	10	…	8	5	10	…
MacDermid Iridite	2	10	9	0.5	8	4	10	…
MacDermid Iridite	3	10	10	…	6	9	10	…
PPG Xbond 3000	1	10	10	…	7	3.5	10	…
PPG Xbond 3000	2	10	8	5	7	6	10	…
PPG Xbond 3000	3	10	8	6	10	--	7	5
3M AC131	1	10	9	1.5	7	12	8	15
3M AC131	2	10	10	…	7	6	10	…
3M AC131	3	10	10	…	5	10	7	22
Deft DFM IV/DFS	1	10	9	10	7	12	10	…
Deft DFM IV/DFS	2	10	8	3	7	11	10	…
Deft DFM IV/DFS	3	10	9	1	8	10	7	3
Deft 1043/3024	1	10	8	3	7	10	8	4
Deft 1043/3024	2	10	10	…	7	13	10	…
Deft 1043/3024	3	10	7	7	6	18	10	…
Deft 1060/3007	1	10	9	1.5	8	2.5	10	…
Deft 1060/3007	2	10	7	5	7	4	7	6
Deft 1060/3007	3	10	9	1	8	5	10	…
Pantheon PreKote	1	10	10	…	7	11	10	…
Pantheon PreKote	2	10	10	…	8	2	10	…
Pantheon PreKote	3	10	8	2	7	19	10	…

Table 9 21-cycle galvanic panel results: MIL-PFR-23377[11] Class N primer-only area

Pretreatment	Panel	Primer						
		BIF	Ti bolt, scribed		SS bolt, scribed		SS bolt, no scribe	
			Blister rating	Amt (mm)	Blister rating	Amt (mm)	Blister rating	Amt (mm)
81706 Type I	1	10	10	…	7	7	3	8
81706 Type I	2	10	10	…	9	1	9	1
81706 Type I	3	10	8	1	8	0.5	10	…
81706 Type II	1	10	7	2.5	8	1.5	7	1.5
81706 Type II	2	10	8	3	8	3	10	…
81706 Type II	3	10	8	3	7	5	10	…
Henkel 5700	1	10	7	1	7	3	5	16
Henkel 5700	2	10	7	8	6	7	4	16
Henkel 5700	3	10	7	6	7	7	8	1
MacDermid Iridite	1	10	7	5.5	5	9	7	3.5
MacDermid Iridite	2	10	7	2.5	6	6.5	5	8.5
MacDermid Iridite	3	10	8	2.5	7	6	8	2.5
PPG Xbond 3000	1	10	6	8	7	3	9	0.5
PPG Xbond 3000	2	10	7	14	7	6	10	…
PPG Xbond 3000	3	10	7	1.5	8	1	10	…
3M AC131	1	10	10	…	5	11	7	8
3M AC131	2	10	7	4	6	23	2	26
3M AC131	3	10	5	6	5	15	4	18
Deft DFM IV/DFS	1	10	7	2	4	18	4	27
Deft DFM IV/DFS	2	10	7	8	5	13	3	24
Deft DFM IV/DFS	3	10	7	6	6	13	5	19
Deft 1043/3024	1	10	6	9	4	25	4	21
Deft 1043/3024	2	10	7	4	5	27	3	23
Deft 1043/3024	3	10	7	24	6	21	5	14
Deft 1060/3007	1	10	8	1.5	5	22	4	15
Deft 1060/3007	2	10	5	17	5	27	5	18
Deft 1060/3007	3	10	7	4	6	14	6	23
Pantheon PreKote	1	10	8	2	5	16	5	13
Pantheon PreKote	2	10	7	8	7	16	7	6
Pantheon PreKote	3	10	8	2	7	16	6	10

Table 10 21-cycle galvanic panel results: MIL-PFR-23377[11] Class N primer and topcoat area

Pretreatment	Panel	Primer with topcoat						
			Ti bolt, scribed		SS bolt, scribed		SS bolt, no scribe	
		BIF	Blister rating	Amt (mm)	Blister rating	Amt (mm)	Blister rating	Amt (mm)
81706 Type I	1	10	10	…	10	…	10	…
81706 Type I	2	10	7	2	9	8	10	…
81706 Type I	3	10	8	2	10	…	7	12
81706 Type II	1	10	8	1.5	7	16	10	…
81706 Type II	2	10	8	6	8	1.5	9	1.5
81706 Type II	3	10	10	…	10	…	10	…
Henkel 5700	1	10	7	3	7	4	7	8
Henkel 5700	2	10	7	5	7	5	10	…
Henkel 5700	3	10	8	3	8	3	7	9
MacDermid Iridite	1	10	6	8	7	5	8	3
MacDermid Iridite	2	10	7	2	7	10.5	8	1.5
MacDermid Iridite	3	10	8	6	7	6	10	…
PPG Xbond 3000	1	10	6	7	8	5	7	1.5
PPG Xbond 3000	2	10	5	9	6	6	10	…
PPG Xbond 3000	3	10	8	1	8	3	6	2
3M AC131	1	10	9	1	7	10	10	…
3M AC131	2	10	7	3	5	15	5	22
3M AC131	3	10	7	4	7	8	7	3
Deft DFM IV/DFS	1	10	8	1	7	4	10	…
Deft DFM IV/DFS	2	10	9	1	5	10	10	…
Deft DFM IV/DFS	3	10	9	1	7	6	5	10
Deft 1043/3024	1	10	5	16	6	12	5	5
Deft 1043/3024	2	10	6	7	5	14	5	12
Deft 1043/3024	3	10	7	24	6	26	8	6
Deft 1060/3007	1	10	6	13	7	9	6	5
Deft 1060/3007	2	10	7	8	6	16	8	2
Deft 1060/3007	3	10	6	10	8	7	6	5
Pantheon PreKote	1	10	7	3	6	13	8	3
Pantheon PreKote	2	10	7	11	7	10	6	8
Pantheon PreKote	3	10	10	…	7	10	10	…

Table 11 21-cycle galvanic panel results: MIL-PFR-85582[12] Class N primer-only area

Pretreatment	Panel	Primer with topcoat: BIF	Ti bolt, scribed: Blister rating	Ti bolt, scribed: Amt (mm)	SS bolt, scribed: Blister rating	SS bolt, scribed: Amt (mm)	SS bolt, no scribe: Blister rating	SS bolt, no scribe: Amt (mm)
81706 Type I	1	10	8	6	7	12	5	7
81706 Type I	2	10	10	…	8	1.5	10	…
81706 Type I	3	10	10	…	6	7	6	
81706 Type II	1	10	9	0.5	5	8	5	16
81706 Type II	2	10	8	1.5	7	8	10	…
81706 Type II	3	10	10	…	6	7	10	…
Henkel 5700	1	6	10	…	7	8	5	14
Henkel 5700	2	10	8	3	7	7	6	11
Henkel 5700	3	10	8	5	7	5	7	7
MacDermid Iridite	1	10	7	1.5	5	13	4	5
MacDermid Iridite	2	10	8	1	4	27	5	5
MacDermid Iridite	3	10	8	1	6	13	6	13
PPG Xbond 3000	1	10	8	2	5	18	10	…
PPG Xbond 3000	2	10	7	10	5	18	5	13
PPG Xbond 3000	3	10	5	8	5	13	3	8
3M AC131	1	10	6	7	6	18	3	14
3M AC131	2	10	8	1	5	16	5	26
3M AC131	3	10	7	2	6	27	4	24
Deft DFM IV/ DFS-II	1	10	6	8	5	18	10	…
Deft DFM IV/ DFS-II	2	10	7	4	6	19	4	23
Deft DFM IV/ DFS-II	3	10	8	1.5	6	8	5	8
Deft 1043/3024	1	10	5	14	4	25	4	30
Deft 1043/3024	2	10	7	9	4	32	3	36
Deft 1043/3024	3	10	7	10	6	26	4	26
Deft 1060/3007	1	10	8	2	5	23	5	16
Deft 1060/3007	2	10	7	1.5	6	27	5	22
Deft 1060/3007	3	10	7	3	5	24	5	26
Pantheon PreKote	1	10	9	2	6	16	3	7
Pantheon PreKote	2	10	7	3	7	17	6	7.5
Pantheon PreKote	3	10	9	1	7	15	4	5

Table 12 21-cycle galvanic panel results: MIL-PFR-85582[12] Class N primer and topcoat area

Pretreatment	Panel	Primer with topcoat						
		BIF	Ti bolt, scribed		SS bolt, scribed		SS bolt, no scribe	
			Blister rating	Amt (mm)	Blister rating	Amt (mm)	Blister rating	Amt (mm)
81706 Type I	1	10	10	…	10	…	9	0.5
81706 Type I	2	10	9	1	7	7	10	…
81706 Type I	3	10	9	1	8	0.5	10	--
81706 Type II	1	10	9	1	7	5	7	2
81706 Type II	2	10	8	0.5	7	2.5	10	…
81706 Type II	3	10	10	…	10	…	10	…
Henkel 5700	1	10	7	3	7	10	8	11
Henkel 5700	2	10	7	3	6	8	8	1
Henkel 5700	3	10	7	2	7	5	7	6
MacDermid Iridite	1	10	9	1	6	6	8	1.5
MacDermid Iridite	2	10	7	4.5	6	6.5	5	7
MacDermid Iridite	3	10	7	--	5	7	10	--
PPG Xbond 3000	1	10	8	2	8	5	10	--
PPG Xbond 3000	2	10	7	7	6	13	8	7
PPG Xbond 3000	3	10	8	1.5	7	6	6	8
3M AC131	1	10	7	6	6	6	7	15
3M AC131	2	10	7	13	5	12	10	--
3M AC131	3	10	6	7	5	21	10	--
Deft DFM IV/DFS-II	1	10	6	10	5	21	10	--
Deft DFM IV/DFS-II	2	10	7	4	7	11	7	2.5
Deft DFM IV/DFS-II	3	10	7	6	5	14	10	--
Deft 1043/3024	1	10	7	9	6	12	7	4
Deft 1043/3024	2	10	7	13	6	18	6	4
Deft 1043/3024	3	10	8	4	8	7	8	4
Deft 1060/3007	1	10	8	6	7	13	7	6
Deft 1060/3007	2	10	7	3	7	16	8	5
Deft 1060/3007	3	10	7	9	6	16	10	--
Pantheon PreKote	1	10	9	2	6	12	10	--
Pantheon PreKote	2	10	8	5	6	11	10	--
Pantheon PreKote	3	10	9	1	7	8	10	--

Table 13 Max scribe creep for samples with MIL-PRF-23377[11] Class C primer

Pretreatment	Panel	Primer only	Primer with topcoat
		Scribe creep rating	Scribe creep rating
81706 Type I	1	9	8
81706 Type I	2	9	10
81706 Type I	3	10	10
81706 Type II	1	8	9
81706 Type II	2	9	9
81706 Type II	3	9	6
Henkel 5700	1	9	7
Henkel 5700	2	9	7
Henkel 5700	3	7	8
MacDermid Iridite	1	10	9
MacDermid Iridite	2	9	9
MacDermid Iridite	3	7	7
PPG Xbond 3000	1	8	8
PPG Xbond 3000	2	7	8
PPG Xbond 3000	3	8	9
3M AC131	1	7	7
3M AC131	2	9	7
3M AC131	3	8	8
Deft DFM IV/DFS-II	1	6	8
Deft DFM IV/DFS-II	2	6	6
Deft DFM IV/DFS-II	3	5	8
Deft 1043/3024	1	7	9
Deft 1043/3024	2	5	6
Deft 1043/3024	3	6	7
Deft 1060/3007	1	9	8
Deft 1060/3007	2	9	8
Deft 1060/3007	3	9	8
Pantheon PreKote	1	8	7
Pantheon PreKote	2	8	8
Pantheon PreKote	3	6	6

Table 14 Max scribe creep for samples with MIL-PRF-23377[11] Class N primer

Pretreatment	Panel	Primer only	Primer with topcoat
		Scribe creep rating	Scribe creep rating
81706 Type I	1	7	7
81706 Type I	2	9	7
81706 Type I	3	7	8
81706 Type II	1	9	8
81706 Type II	2	8	7
81706 Type II	3	8	8
Henkel 5700	1	8	5
Henkel 5700	2	8	7
Henkel 5700	3	7	7
MacDermid Iridite	1	7	7
MacDermid Iridite	2	7	7
MacDermid Iridite	3	7	7
PPG Xbond 3000	1	7	6
PPG Xbond 3000	2	7	5
PPG Xbond 3000	3	7	8
3M AC131	1	7	7
3M AC131	2	5	5
3M AC131	3	6	7
Deft DFM IV/ DFS-II	1	5	7
Deft DFM IV/ DFS-II	2	6	7
Deft DFM IV/ DFS-II	3	5	6
Deft 1043/3024	1	4	6
Deft 1043/3024	2	3	5
Deft 1043/3024	3	4	5
Deft 1060/3007	1	7	6
Deft 1060/3007	2	8	8
Deft 1060/3007	3	7	7
Pantheon PreKote	1	7	6
Pantheon PreKote	2	5	7
Pantheon PreKote	3	6	7

Table 15 **Max scribe creep for samples with MIL-PRF-85582[12] Class N primer**

Pretreatment	Panel	Primer only	Primer with topcoat
		Scribe creep rating	Scribe creep rating
81706 Type I	1	6	7
81706 Type I	2	9	9
81706 Type I	3	7	7
81706 Type II	1	9	6
81706 Type II	2	8	9
81706 Type II	3	7	8
Henkel 5700	1	7	7
Henkel 5700	2	7	7
Henkel 5700	3	7	6
MacDermid Iridite	1	7	6
MacDermid Iridite	2	9	6
MacDermid Iridite	3	7	5
PPG Xbond 3000	1	5	6
PPG Xbond 3000	2	7	7
PPG Xbond 3000	3	6	7
3M AC131	1	7	7
3M AC131	2	7	7
3M AC131	3	6	6
Deft DFM IV/DFS-II	1	7	7
Deft DFM IV/DFS-II	2	7	7
Deft DFM IV/DFS-II	3	7	7
Deft 1043/3024	1	5	7
Deft 1043/3024	2	5	6
Deft 1043/3024	3	7	7
Deft 1060/3007	1	8	9
Deft 1060/3007	2	6	7
Deft 1060/3007	3	7	7
Pantheon PreKote	1	7	6
Pantheon PreKote	2	7	7
Pantheon PreKote	3	7	7

Results for BIF can be found in the column labeled "BIF" in Tables 7–12. Overall, areas that did not have direct contact with the dry-installed fastener performed well. Only one area on one panel had a rating less than 10. This was the primer-only area of a panel that received Henkel's 5700 pretreatment and MIL-PRF-85582[12] Class N. On this panel, the rating was the result of one isolated blister that did not appear to have initiated from a fastener.

Corrosion results for blisters from the area of the Ti fastener can be found in Tables 7–12 in the column labeled "Ti Bolt, scribed". Overall, for the sets coated with the MIL-PRF-23377[11] Class C primer, there was only one panel that had blisters ratings of a 6 or worse. That was a panel that was pretreated with the Deft 1043/3024. On panels coated with MIL-PRF- 23377 Class N and MIL-PRF-85582[12] Class N, there were a few more panels that had maximum blisters ratings of a 6 or worse. More concerning, there are select panels that has areas totaling more than 50% (15 mm or higher in the "AMT [mm]" column) of the washer with some form of corrosion present. This means that the system is failing in not just one spot, but all around the fastener.

The control pretreatment images of the galvanic panels after 21 cycles of GMW14872[16] can be found in Fig. 5 (MIL-DTL-81706[9] Type I) and Fig. 6 (MIL-DTL-81706 Type II). In each image, the top three panels are primed with MIL-PRF-23377[11] Class C, the middle three panels are primed with MIL-PRF-23377 Class N, and the bottom three panels are primed with MIL-PRF-85582[12] Class N. A large blister originated from the unscribed SS fastener on one of the MIL-DTL-81706 Type I with MIL-DTL-23377 panels. There were many smaller blisters around the SS fasteners on both control pretreatments with the MIL-PRF-85582 Class N.

Fig. 5 MIL-DTL-81706 Type I: top three primed with MIL-PRF-23377 Class C, middle three primed with MIL-PRF-23377 Class N, and bottom three primed with MIL-PRF-85582

Fig. 6 MIL-DTL-81706 Type II: top three primed with MIL-PRF-23377 Class C, middle three primed with MIL-PRF-23377 Class N, and bottom three primed with MIL-PRF-85582

Henkel's 5700 galvanic panels after 21 cycles of GMW14872 are shown in Fig. 7. The top three panels are primed with MIL-PRF-23377 Class C, the middle three panels are primed with MIL-PRF-23377 Class N, and the bottom three panels are primed with MIL-PRF-85582 Class N. There were some issues with corrosion around the unscribed SS fasteners on the MIL-PRF-23377 Class N panels where the topcoat was not present. On the MIL-PRF-85582 Class N panels, there was one random blister that was not associated with a fastener. On that same set of panels, there was some corrosion coming from around the unscribed SS fastener. In this area, the ratings were similar to the controls.

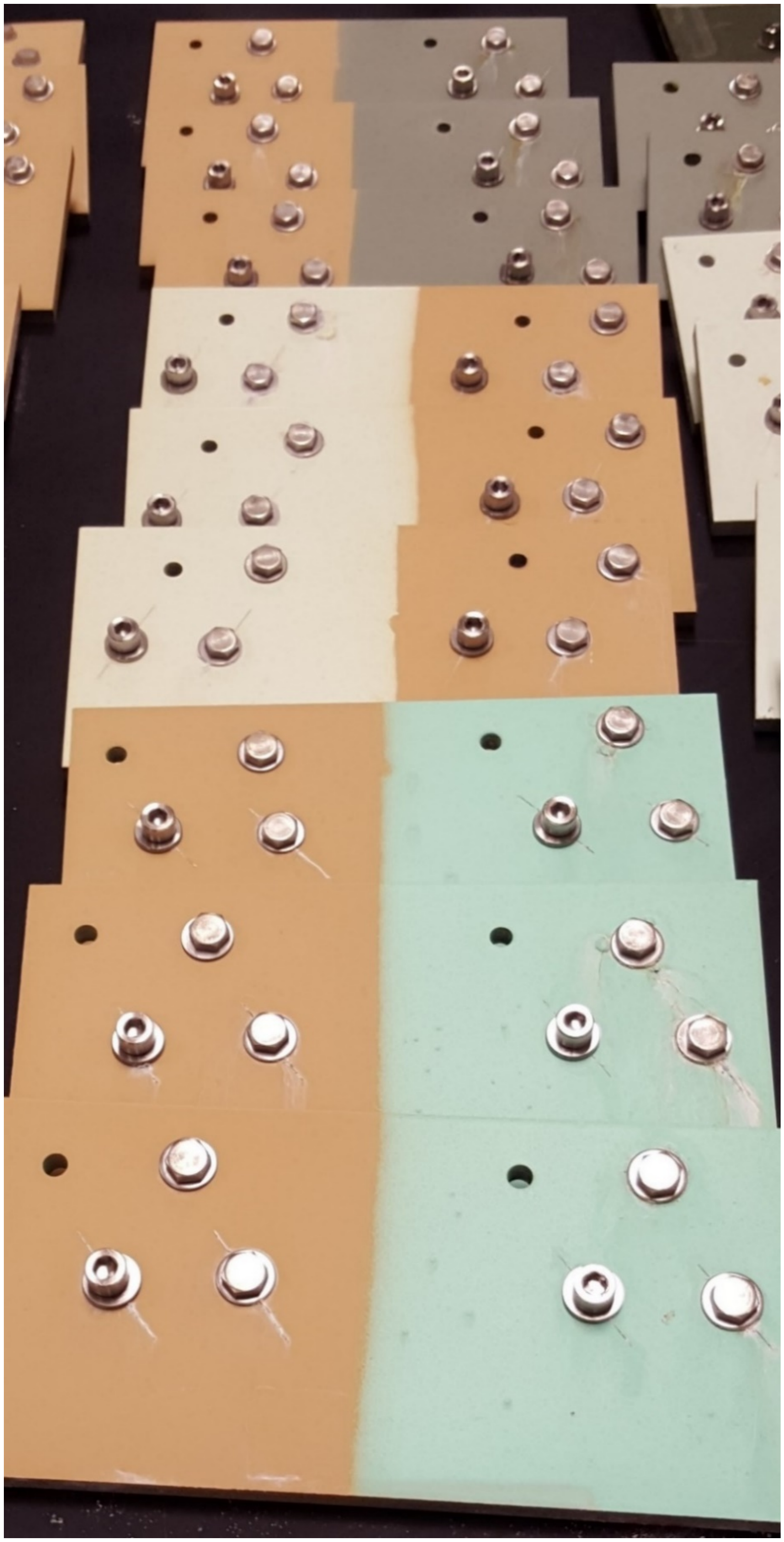

Fig. 7 Henkel 5700: top three primed with MIL-PRF-23377 Class C, middle three primed with MIL-PRF-23377 Class N, and bottom three primed with MIL-PRF-85582

MacDermid's Iridite NCP galvanic panels after 21 cycles of GMW14872 are shown in Fig. 8. The top three panels are primed with MIL-PRF-23377 Class C, the middle three panels are primed with MIL-PRF-23377 Class N, and the bottom three panels are primed with MIL-PRF-85582 Class N. There was some issues with blisters near the fastener and scribes when the MIL-PRF-85582 Class N primer was used. There was also some corrosion present when the MIL-PRF-23377 Class N primer was used. In most areas, ratings were similar to the controls, but the MIL-85582 Class N panels did not perform quite as well as the controls in the cyclic test.

Fig. 8 MacDermid Iridite NCP: top three primed with MIL-PRF-23377 Class C, middle three primed with MIL-PRF-23377 Class N, and bottom three primed with MIL-PRF-85582

PPG's X-Bond 3000 galvanic panels after 21 cycles of GMW14872 are shown in Fig. 9. The top three panels are primed with MIL-PRF-23377 Class C, the middle three panels are primed with MIL-PRF-23377 Class N, and the bottom three panels are primed with MIL-PRF-85582 Class N. Similar to the MacDermid product, there was some corrosion present on the MIL-PRF-85582 Class N panels in areas with no topcoat. There was also a little bit more creep from the scribe present on a MIL-PRF-23377 Class N panel preset near the Ti fastener. Other than those areas, the results for the X-Bond 3000 were similar to the controls.

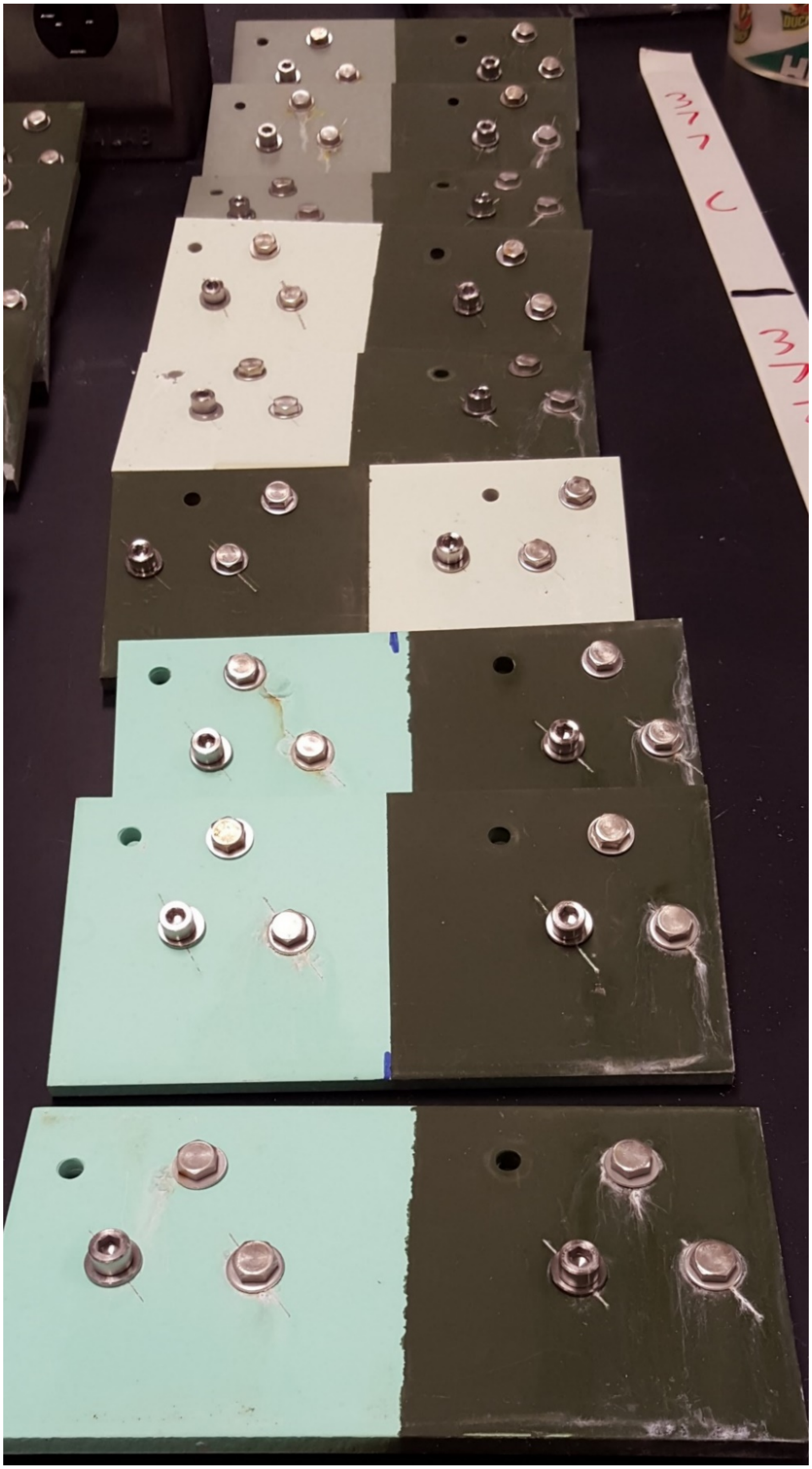

Fig. 9 PPG X-bond 3000: top three primed with MIL-PRF-23377 Class C, middle three primed with MIL-PRF-23377 Class N, and bottom three primed with MIL-PRF-85582

3M's AC-131 galvanic panels after 21 cycles of GMW14872 are shown in Fig. 10. The top three panels are primed with MIL-PRF-23377 Class C, the middle three panels are primed with MIL-PRF-23377 Class N, and the bottom three panels are primed with MIL-PRF-85582 Class N. Overall, this pretreatment had a lot of failures around SS fasteners when Class N primers were used. This is evident with the large blisters visible on the MIL-PRF-23377 Class N panels in Fig. 10. Overall, this pretreatment did not perform as well as the controls.

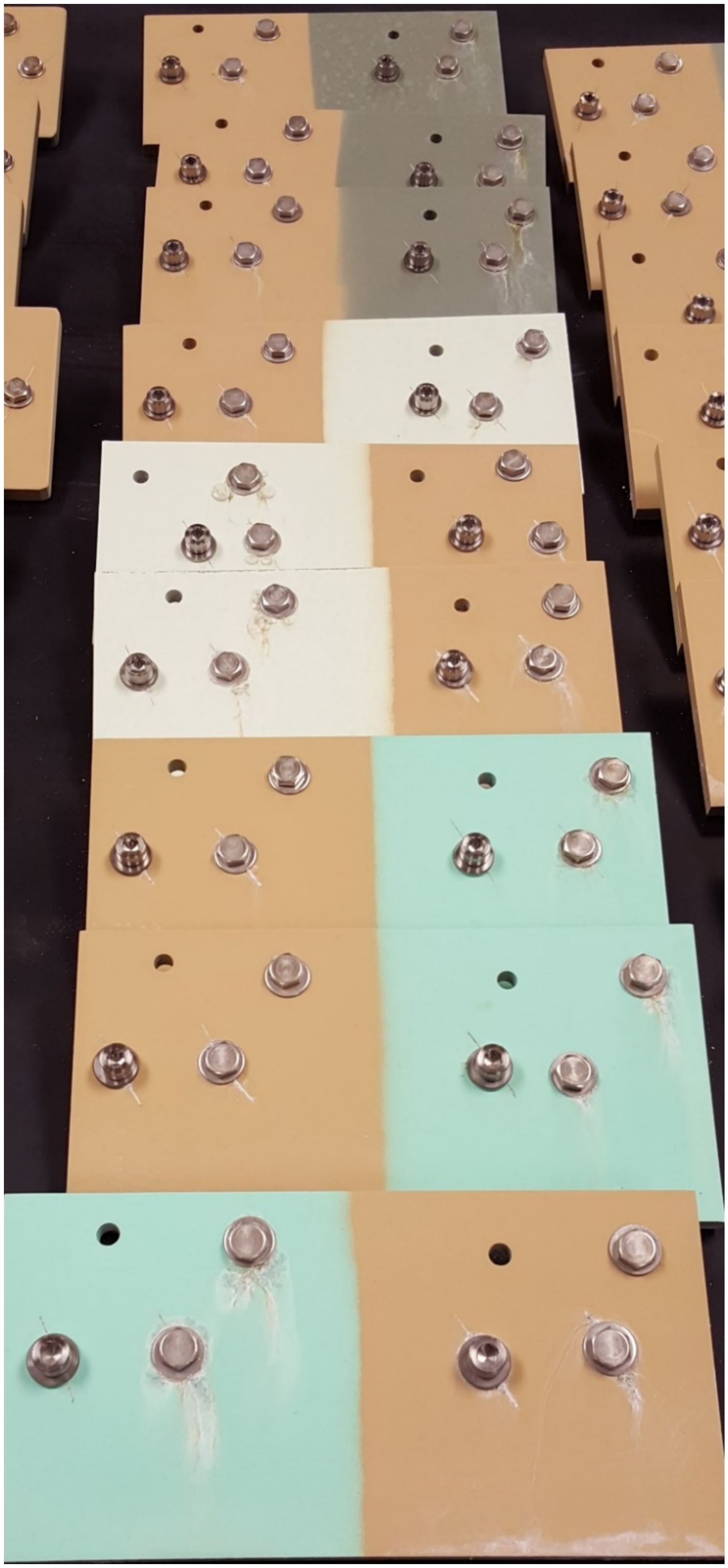

Fig. 10 3M AC131: top three primed with MIL-PRF-23377 Class C, middle three primed with MIL-PRF-23377 Class N, and bottom three primed with MIL-PRF-85582

Deft's three systems (DFM IV/DFS-II, 1043/3024, and 1060/3007) can be found in Fig. 11. The top three panels of each column are primed with MIL-PRF-23377 Class C, the middle three panels are primed with MIL-PRF-23377 Class N, and the bottom three panels are primed with MIL-PRF-85582 Class N. Overall, there were a lot of corrosion issues with all three pretreatments when the panels were primed with the Class N primers. It was very common for the SS fasteners found on the panels with these pretreatments and Class N primers to have coverage of corrosion around the washer in excess of 50%. Even on the panels with the MIL-DTL-23377 Class C primer there was more creep from the scribe of the SS fasteners than witnessed with the controls. Overall, these three pretreatments did not perform as well as the controls.

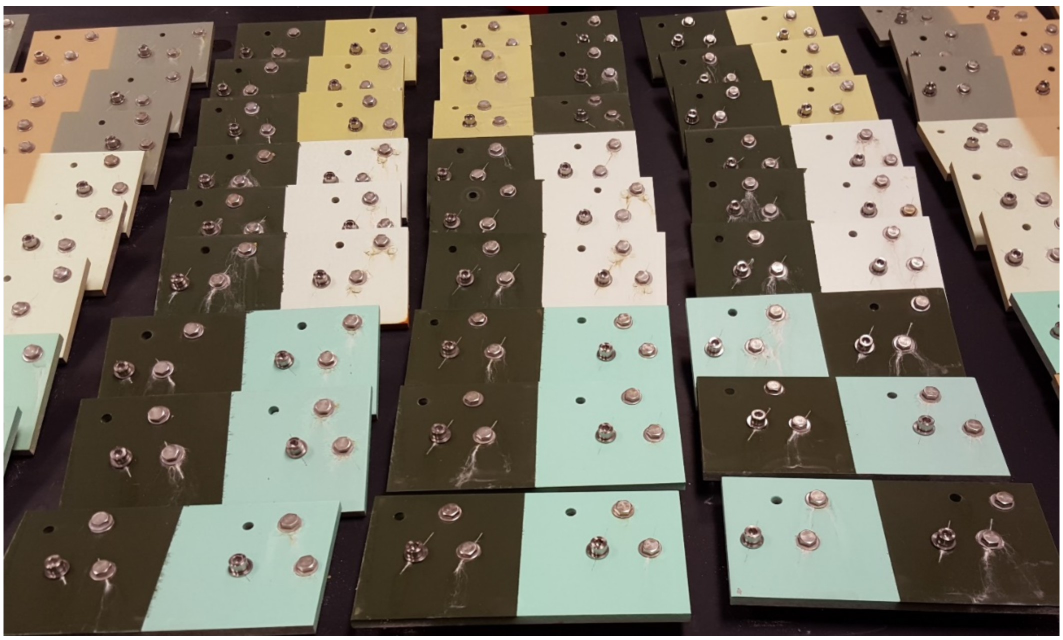

Fig. 11 Deft 1043(L), DFS (C), and 1060(R): top three primed with MIL-PRF-23377 Class C, middle three primed with MIL-PRF-23377 Class N, and bottom three primed with MIL-PRF-85582

Pantheon's PreKote galvanic panels after 21 cycles of GMW14872 are shown in Fig. 12. The top three panels are primed with MIL-PRF-23377 Class C, the middle three panels are primed with MIL-PRF-23377 Class N, and the bottom three panels are primed with MIL-PRF-85582 Class N. The panels with the MIL-PRF-23377 Class N primer-only showed a lot of corrosion product around the SS fasteners. The three SS fasteners with scribes had 46% (16 mm) of corrosion present around each washer. A lot of larger blisters were witnessed around the unscribed SS fasteners under the MIL-PRF-85582 Class N primer. Overall, in areas with a topcoat, the PreKote performed similar to what was seen with the controls. In areas of primer-only, the PreKote did not perform as well as the controls.

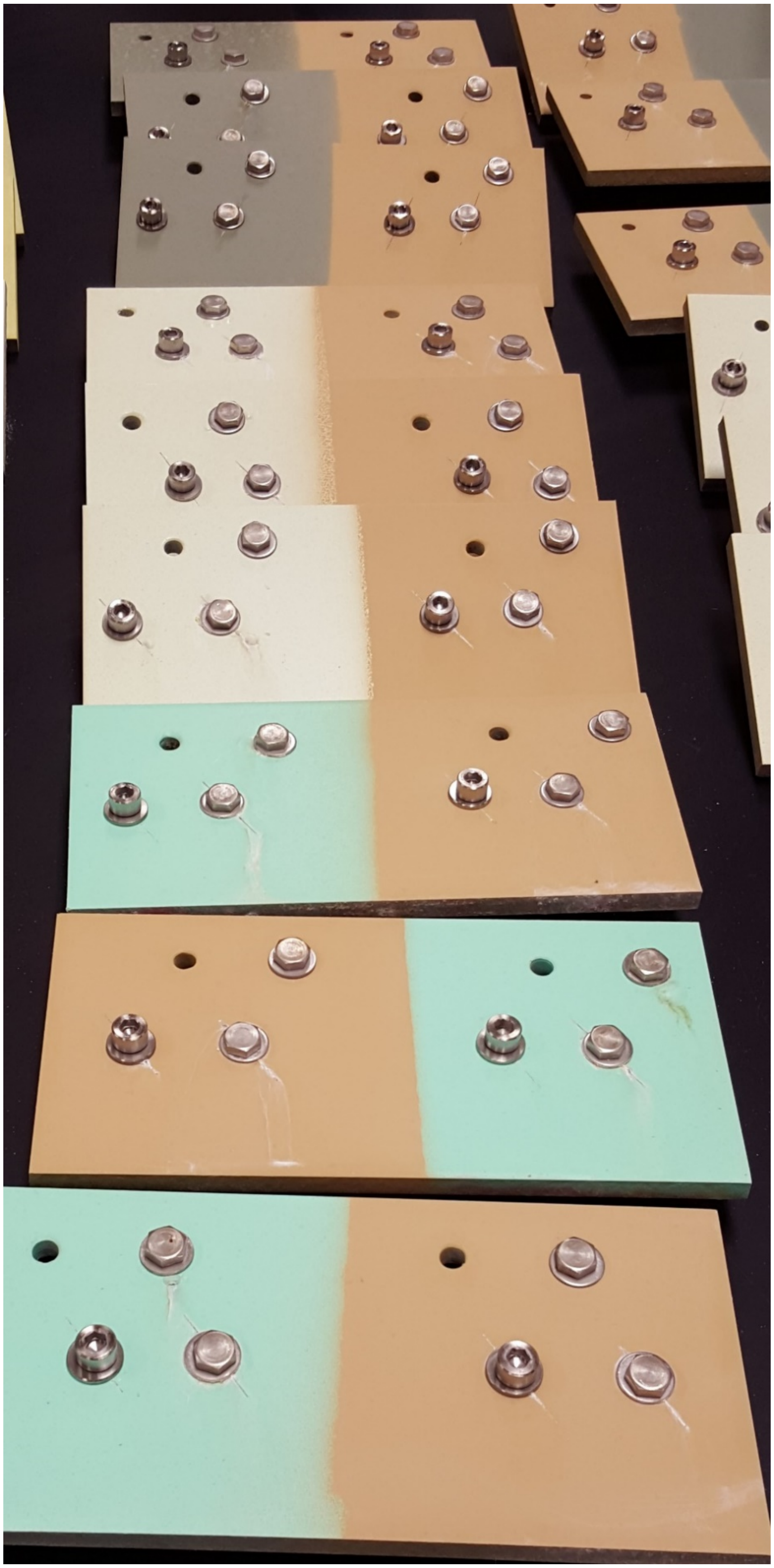

Fig. 12   Pantheon PreKote

Based on the results from GMW14872 cyclic corrosion testing, the Henkel 5700, MacDermid Iridite NCP, and PPG X-Bond 3000 were selected for outdoor weathering at CCAFS. These panels were installed on the site in March 2016. During the time they were exposed, the site observed approximately 3.44 mpy in mass loss.

The results from the outdoor weathering can be found in Tables 16–18. If rust was observed, the ratings included the letter "R". If delamination or blisters were observed, the rating included the letters "D/B". All scribed areas received a rating in accordance with ASTM 1654-08[18] Method A for the creep from scribe rating.

Table 16 15-month outdoor weathering galvanic panel results using MIL-PFR-23377 Class C primer

Pretreatment	Primer/topcoat	Fastener	6 months	9 months	12 months	15 months
MIL-DTL-81706 Type I	MIL-PRF-2377 C no topcoat	SS, no scribe	R	R	R	R
		Ti, scribe		D/B - 9	D/B - 9	D/B - 9
		SS, scribe	D/B	D/B - 9	D/B - 9	D/B - 9
	MIL-PRF-2377 C with topcoat	SS, no scribe	R	R	R	R
		Ti, scribe		D/B - 9	D/B - 9	D/B - 9
		SS, scribe	R	R - 9	R - 9	R - 9
MIL-DTL-81706 Type II	MIL-PRF-2377 C no topcoat	SS, no scribe	R	R	R	R
		Ti, scribe		D/B - 9	D/B - 9	D/B - 9
		SS, scribe	R	R - 9	R - 9	R - 9
	MIL-PRF-2377 C with topcoat	SS, no scribe	R	R	R	R
		Ti, scribe		D/B - 9	D/B - 9	D/B - 9
		SS, scribe	R	R - 9	R - 9	R - 9
PPG X-Bond 3000	MIL-PRF-2377 C no topcoat	SS, no scribe	R	R	R	R
		Ti, scribe		D/B - 9	D/B - 9	D/B - 9
		SS, scribe	D/B	D/B - 8	D/B - 8	D/B - 8
	MIL-PRF-2377 C with topcoat	SS, no scribe	R	R	R	R
		Ti, scribe		D/B - 9	D/B - 9	D/B - 9
		SS, scribe	R	R - 9	R - 9	R - 9
MacDermid Iridite NCP	MIL-PRF-2377 C no topcoat	SS, no scribe	R	R	R	R
		Ti, scribe		D/B - 9	D/B - 9	D/B - 9
		SS, scribe		D/B - 9	D/B - 9	D/B - 9
	MIL-PRF-2377 C with topcoat	SS, no scribe	R	R	R	R
		Ti, scribe		D/B - 9	D/B - 9	D/B - 9
		SS, scribe	R	R - 9	R - 9	R - 9
Henkel 5700	MIL-PRF-2377 C No Topcoat	SS, no scribe	R	R	R	R
		Ti, scribe		D/B - 9	D/B - 9	D/B - 9
		SS, scribe		D/B - 9	D/B - 9	D/B - 9
	MIL-PRF-2377 C with topcoat	SS, no scribe	R	R	R	R
		Ti, scribe		D/B - 9	D/B - 9	D/B - 9
		SS, scribe		D/B - 9	D/B - 9	D/B - 9

Table 17 15-month outdoor weathering galvanic panel results using MIL-PFR-23377 Class N primer

Pretreatment	Primer/topcoat	Fastener	6 months	9 months	12 months	15 months
MIL-DTL-81706 Type I	MIL-PRF-2377 N no topcoat	SS, no scribe	R	R	R	R
		Ti, scribe	D/B	D/B - 7	D/B - 7	D/B - 7
		SS, scribe		R - 9	R - 9	R - 9
	MIL-PRF-2377 N with topcoat	SS, no scribe	R	R	R	R
		Ti, scribe	D/B	D/B - 7	D/B - 7	D/B - 7
		SS, scribe	R	R - 9	R - 9	R - 9
MIL-DTL-81706 Type II	MIL-PRF-2377 N no topcoat	SS, no scribe	R	R	R	R
		Ti, scribe	D/B	D/B - 7	D/B - 7	D/B - 7
		SS, scribe	D/B	R - 7	R - 7	R - 7
	MIL-PRF-2377 N with topcoat	SS, no scribe	R	R	R	R
		Ti, scribe		D/B - 7	D/B - 7	D/B - 7
		SS, scribe	R	R - 7	R - 7	R - 7
PPG X-bond 3000	MIL-PRF-2377 N no topcoat	SS, no scribe	R	R	R	R
		Ti, scribe		D/B - 9	D/B - 9	D/B - 9
		SS, scribe	D/B	D/B - 9	D/B - 9	D/B - 9
	MIL-PRF-2377 N with topcoat	SS, no scribe	R	R	R	R
		Ti, scribe		D/B - 9	D/B - 9	D/B - 9
		SS, scribe	D/B	R - 8	R - 8	R - 8
MacDermid Iridite NCP	MIL-PRF-2377 N no topcoat	SS, no scribe	R	R	R	R
		Ti, scribe		D/B - 9	D/B - 9	D/B - 9
		SS, scribe	D/B	D/B - 6	D/B - 6	D/B - 6
	MIL-PRF-2377 N with topcoat	SS, no scribe	R	R	R	R
		Ti, scribe		D/B - 9	D/B - 9	D/B - 9
		SS, scribe	R	R - 9	R - 9	R - 9
Henkel 5700	MIL-PRF-2377 N no topcoat	SS, no scribe	R	R	R	R
		Ti, scribe		D/B - 9	D/B - 9	D/B - 9
		SS, scribe	R	R - 9	R - 9	R - 9
	MIL-PRF-2377 N with topcoat	SS, no scribe	R	R	R	R
		Ti, scribe		D/B - 9	D/B - 9	D/B - 9
		SS, scribe	D/B	R - 8	R - 8	R - 8

Table 18 15-month outdoor weathering galvanic panel results using MIL-PFR-85582 Class N primer

Pretreatment	Primer/topcoat	Fastener	6 months	9 months	12 months	15 months
MIL-DTL-81706 Type I	MIL-PRF-85582 N no topcoat	SS, no scribe	R	R	R	R
		Ti, scribe		D/B - 8	D/B - 8	D/B - 8
		SS, scribe	D/B	R - 8	R - 8	R - 8
	MIL-PRF-85582 N with topcoat	SS, no scribe	R	R	R	R
		Ti, scribe	D/B	D/B - 9	D/B - 9	D/B - 9
		SS, scribe	D/B	R - 7	R - 7	R - 7
MIL-DTL-81706 Type II	MIL-PRF-85582 N no topcoat	SS, no scribe	R	R	R	R
		Ti, scribe		D/B - 9	D/B - 9	D/B - 9
		SS, scribe	D/B	R - 8	R - 8	R - 8
	MIL-PRF-85582 N with topcoat	SS, no scribe	R	R	R	R
		Ti, scribe		D/B - 9	D/B - 9	D/B - 9
		SS, scribe	D/B	R - 7	R - 7	R - 7
PPG X-bond 3000	MIL-PRF-85582 N no topcoat	SS, no scribe	R	R	R	R
		Ti, scribe		D/B - 9	D/B - 9	D/B - 9
		SS, scribe	D/B	D/B - 8	D/B - 8	D/B - 8
	MIL-PRF-85582 N with topcoat	SS, no scribe	R	R	R	R
		Ti, scribe		D/B - 7	D/B - 7	D/B - 7
		SS, scribe	D/B	R - 9	R - 9	R - 9
MacDermid Iridite NCP	MIL-PRF-85582 N no topcoat	SS, no scribe	R	R	R	R
		Ti, scribe		D/B - 9	D/B - 9	D/B - 9
		SS, scribe	R	R - 8	R - 8	R - 8
	MIL-PRF-85582 N with topcoat	SS, no scribe		R	R	R
		Ti, scribe		D/B - 9	D/B - 9	D/B - 9
		SS, scribe		D/B - 9	D/B - 9	D/B - 9
Henkel 5700	MIL-PRF-85582 N no topcoat	SS, no scribe		R	R	R
		Ti, scribe	D/B	D/B - 7	D/B - 7	D/B - 7
		SS, scribe	D/B	R - 6	R - 6	R - 6
	MIL-PRF-85582 N with topcoat	SS, no scribe		R	R	R
		Ti, scribe		D/B - 9	D/B - 9	D/B - 9
		SS, scribe	D/B	D/B - 7	D/B - 7	D/B - 7

Overall, almost all samples had a scribe creep rating of 7 (2 mm) or better. The samples for the MacDermid product under MIL-PRF-23377 Class N with no topcoat and the Henkel product under MIL-PRF-85582 Class N with no topcoat received a rating of 6 at the scribes around the SS fastener. Overall, all three products provided consistent performance to what was seen with the control MIL-DTL-81706 Type I and Type II.

5. Conclusion and Discussion

Generally, the Henkel 5700, MacDermid Iridite NCP, and PPG X-Bond 3000 performed the best of the nonchrome COTS pretreatments tested. These products showed performance that was similar to currently approved Cr^{6+} and Cr^{3+} pretreatments as part of a stack-up coating system that would be fielded on an Army aviation asset. Additionally, these systems are able to be applied in a fashion that would be considered a drop-in replacement to what is currently being used at CCAD.

Application of the pretreatments is very important to implementing a nonchrome pretreatment. In discussions with repair facilities, it was established that a drop-in process that limited the cost of facility upgrades and did not increase the time to process parts and/or airframes would be easier to implement. As such, it was important that we had products that could be sprayed in the wash bays and immersed as part of the processes in the plating shop. Adding a step such as hand sanding did not have a chance of being fully implemented as part of the repair process.

The outdoor weathering results showed the nonchrome pretreatments performing on par with Cr^{3+} pretreatments and Cr^{6+} pretreatments under all primers used. More outdoor testing with the full stack-up, including topcoat, will need to be completed to fully capture expected performance.

The galvanic panel proved to be a good panel set up for helping to determine coating stack-up performance. As expected, the SS fastener was more aggressive than the Ti fastener. The galvanic potential created a harsh test for the overall coating system in the different corrosive lab and natural environments. While corrosion did emanate from scribes under fasteners, blistering and general corrosion originating from the washer seemed to be a better determining factor for performance. Systems that did not perform as well had more blistering. The degree of blistering was not captured as well with outdoor testing as it was with the lab corrosion testing.

6. Path Forward

Further testing will need to be completed to fully capture the potential of the nonchrome pretreatments in a lab setting. Because the control panels had the extra chemical milling step in the process, it created a more pristine and reactive surface for both the Cr^{3+} and Cr^{6+} pretreatment systems to bond with the Al substrate. It is important to capture how this step may affect the performance of the nonchrome pretreatment to create a true side-by-side comparison.

Further outdoor testing will need to be completed to ensure the nonchrome pretreatments will perform in harsh environments. While limited outdoor testing was completed as part of this project, it would be preferred to have results from multiple sites. Outdoor testing also needs to be completed on actual parts to ensure protection on complex geometries. This work is currently ongoing.

Prior to implementing this technology, multiple demonstrations will have to be completed. First, a demonstration of application on parts will need to be conducted at a repair facility. This demonstration with show if these products can be applied in the facilities as they are currently set up. This demonstration will also provide information on processing times and if the new pretreatments will perform when applied in the repair facilities. Other demonstrations will need to be completed on flying assets. At first, smaller, easily removable parts will be used to keep the risk of failure low. Finally, this will need to be demonstrated on the outer mold lines of a full helicopter.

These materials would have to be written into specifications and production documents to be transitioned to the field for use. The additional laboratory and demonstration work will assist in developing the requirements that need to be included in the specification. At this point, the most appropriate specification for these type of materials would be MIL-DTL-81706. Then process specifications would have to be updated to call out the new nonchrome pretreatments.

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List of Symbols, Abbreviations, and Acronyms

BIF	blisters in field
CARC	Chemical Agent Resistant Coating
CCAD	Corpus Christi Army Depot
CCAFS	Cape Canaveral Air Force Station
Ce	cerium
Cr^{3+}	trivalent chromium
Cr^{6+}	hexavalent chromium
COTS	commercial off the shelf
DOD	US Department of Defense
FZA	fluorozirconic acid
NaCl	sodium chloride
NaOH	sodium hydroxide
NAVAIR	US Naval Air Systems Command
OSHA	Occupational and Health Administration
RECC	rare earth conversion coating
SS	stainless steel
Ti	titanium

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