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Environmentally Friendly Zirconium Oxide Pretreatment

by Fred Lafferman, Jeff Stalker, and Daniel Pope

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Environmentally Friendly Zirconium Oxide Pretreatment

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14. ABSTRACT Laboratory corrosion testing has shown zirconium pretreatment to be an environmentally friendly replacement for zinc phosphate treatment on steel and for hexavalent and trivalent chromium on aluminum. To document the performance of zirconium pretreatment in production, demonstrations were conducted at Letterkenny Army Depot in Chambersburg, Pennsylvania, and US Marine Corps Logistics Base Albany in Albany, Georgia. The demonstrations consisted of cleaning parts and panels and treating them with an X-Bond 4000 zirconium treatment followed by painting them with the currently used primer and topcoat. Parts and panels were also prepared using the existent depot technologies for cold rolled steel (zinc phosphate), aluminum (chrome 3+ and chrome 6+), and high hard armor (clean-only). Parts and panels were tested in laboratory-accelerated corrosion chambers, installed on vehicles at US Marine Corps Camp Lejeune, or placed on outdoor exposure at seacoast test facilities. After testing or exposure per the appropriate specification, parts and panels were inspected. Results showed that the zirconium pretreatment under solventborne, waterborne, and powder primers performed better than trivalent chromium and nearly equal to hexavalent chromium on aluminum. On steel, X-Bond 4000 ranged from slightly worse to slightly better than zinc phosphate; it met the performance requirements of TT-C-490. On high hard armor, X-Bond showed improved corrosion inhibition versus grit-blasted only.					
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Executive Summary: Environmentally Friendly Zirconium Oxide Pretreatment, ESTCP Project WP-201318

Introduction

PPG Industries, Inc.'s (PPG's) zirconium-based pretreatment technology provides the initial layer of corrosion protection and enhances adhesion to both steel and aluminum substrates. This reduces pretreatment line complexity and costs over incumbent materials. The replacement offers several other advantages over present pretreatments, including the elimination of toxic metals such as nickel used in zinc phosphate and chromium. The zirconium pretreatments developed by PPG have been in commercial use on automotive and industrial paint lines for over 15 years and represent a mature technology for transition to military use.

The focus of this demonstration was to validate use of zirconium-based pretreatments as an alternative to those currently approved for use on military vehicles. For steel substrates, the incumbent pretreatment technology is zinc phosphate, as described in TT-C-490 Type I, including di- and tri-cation versions. Zinc phosphate provides a crystal structure to which organic coatings such as paints and powder coatings can adhere. A chrome sealer or rinse provides improved corrosion resistance but increases waste disposal costs and introduces additional EH&S concerns. Chromium-based pretreatments are more commonly employed for aluminum substrates; these include hexavalent (MIL-DTL-5541 Type I) and trivalent (MIL-DTL -5541 Type II) chromium products.

This demonstration project built on the findings from WP-1676 and demonstrated the performance capabilities of zirconium pretreatment for protection of reconditioned material at US Army and US Marine Corps depots. X-Bond 4000, a commercial pretreatment, was selected for this demonstration and is commonly used with a variety of metal substrates and industrial finishes. WP-1676 provided the initial evaluation of zirconium immersion pretreatments intended for Original Equipment Manufacturer (OEM) application over CRS and aluminum with water-borne and solvent-borne Mil-Spec primers. Results in corrosion performance were similar to the chrome (VI) and zinc phosphate pretreatment controls. In conjunction with the US Army Combat Capabilities Development Command Army Research Laboratory (ARL), modified versions of the commercial zirconium pretreatment showed corrosion resistance equal to or better than zinc phosphate in both spray-applied and immersion-applied processes. Accordingly, the work showed that the current commercial zirconium pretreatment technology, with some modifications, is suited for use in DOD OEM processes. Visits to Marine Corps

Logistics Base Albany, Albany, Georgia, and Letterkenny Army Depot, Chambersburg, Pennsylvania, suggested that development of an immersion zirconium pretreatment process would be of utility for DOD repair depots as well.

Objectives

The goal of this project was to process and field test coupons or small components that demonstrated the corrosion resistance capabilities of a MIL-DTL-53072 finish, replacing the typical pretreating step (whether ferrous/zinc-phosphate or non-ferrous/chrome-functional) with a zirconium oxide pretreatment film. The environmental benefits of a zirconium-based pretreatment, such as PPG's X-Bond 4000, in place of existing pretreatments include elimination of nickel and hexavalent chromium compounds, decreased amount of sludge generated from operation of the pretreatment baths, reduced water and energy consumption, and a smaller footprint for pretreatment operations.

Performance validation for all parts tested focused on the corrosion resistance, water and chemical resistance, and adhesion performance as required by pretreatment, primer, and full CARC system specifications as shown in Table ES-1.

Table ES-1: Demonstration part performance expectations

Target HazMat	Current process	Applications	Current specifications	Affected programs	Candidate parts and substrates
Nickel-containing zinc phosphate sludge	Zinc Phosphate	Steel substrates	TT-C-490 MIL-DTL-53022 MIL-DTL-53030 MIL-DTL-53072	Depot repair operations using zinc phosphate	All-blasted steel Cold rolled steel Hot rolled steel Armor steel
Hexavalent Chromium (Cr6) from seal coat application					
Hexavalent Chromium (Cr6)	Aluminum conversion coating	Aluminum substrates	MIL-DTL-5541 MIL-DTL-53022 MIL-DTL-53030 MIL-DTL-53072	Depot repair operations for aluminum substrates	All-blasted AA2024-T3 AA6061-T6

Technology Description

X-Bond 4000 is a hexafluorozirconic acid (FZA) based pretreatment developed and commercialized by PPG. First commercialized in 2006, X-Bond 4000 is a general use conversion coating for both steel and aluminum. It relies on the ability of FZA to etch the surface of the substrate, creating a localized pH imbalance and resulting in precipitation of zirconium oxide onto the surface. The zirconium oxide deposits, which adhere tightly to the underlying steel or aluminum, provide both physical texture and opportunities for noncovalent chemical interactions. These significantly improve adhesion and corrosion resistance relative to a clean, but untreated, surface. In addition, the zirconium oxide film is much thinner than zinc phosphate (Fig. ES-1), inherently consuming less raw material and reducing replenishment costs.

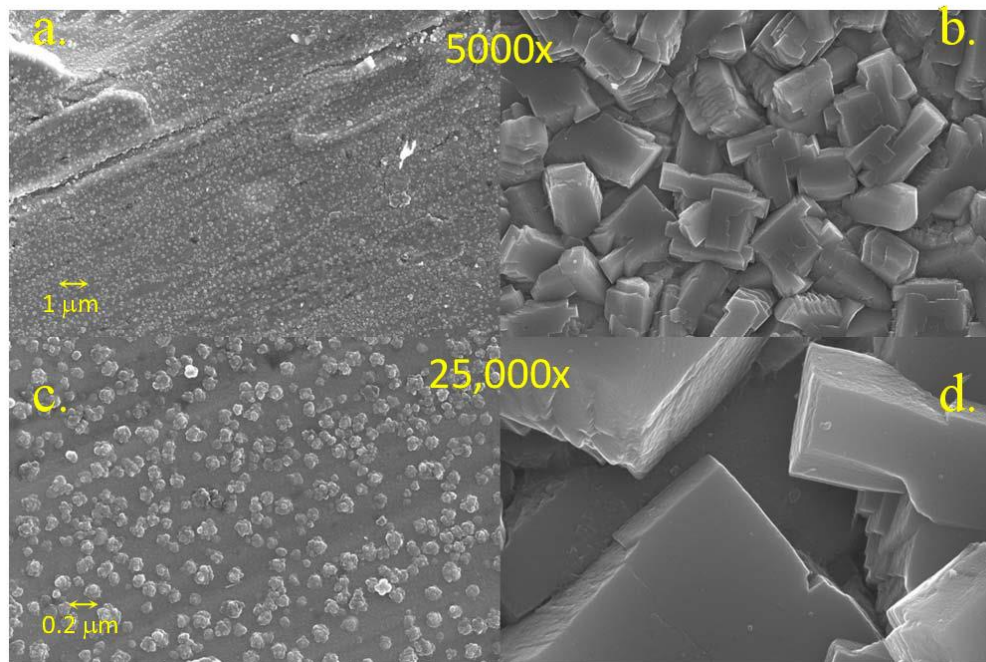


Fig. ES-1 SEM images of zirconium and zinc phosphate pretreatments

- (a) Zirconium-based pretreatment SEM image at 5,000× magnification
- (b) Zinc phosphate SEM image at 5,000× magnification
- (c) Zirconium-based pretreatment, 25,000×
- (d) Zinc phosphate, 25,000×

In existing paint lines, zirconium pretreatment technologies are drop-in replacements for the existent technology. Tanks set up to handle either zinc phosphate or Cr^{6+} conversion coatings are already capable of handling zirconium

chemistries. A schematic of the process for X-Bond 4000 in the demonstration conducted at Letterkenny Army Depot (LEAD) is shown in Fig. ES-2.

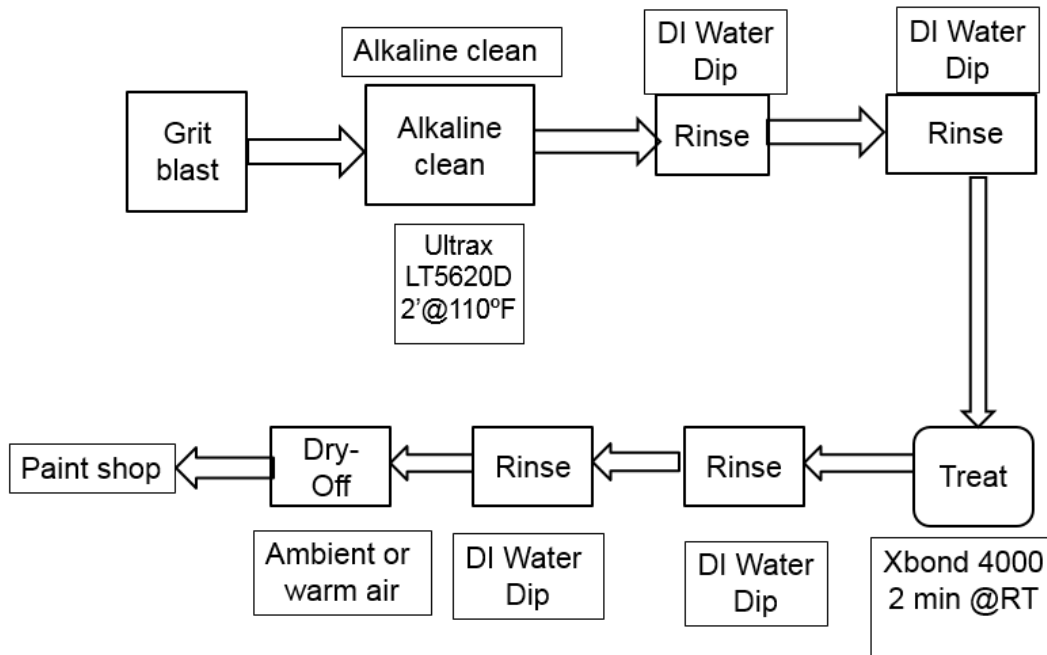


Fig. ES-2 Schematic of X-Bond 4000 process for the LEAD demonstration

The use of zirconium treatment technology offers several advantages over current technologies:

- Zirconium treatments can be used at room temperature, reducing the energy costs of maintaining heated tanks.
- The relatively thin films of the zirconium pretreatments compared to zinc phosphate pretreatments consume significantly less raw material.
- Zinc phosphate baths cannot accommodate both ferrous and non-ferrous substrates in the same bath. In contrast, zirconium treatments can handle both steel and aluminum in the same bath without the need for close monitoring of the bath composition. This provides greater scheduling and processing flexibility to the depots and reduces the footprint of the painting process.
- The operating pH of zinc phosphate processes can generate hydrogen gas when treating steel, which leads to embrittlement. The pH of zirconium treatment is high enough (4.0–5.0) that it does not generate hydrogen gas and therefore can be used to treat high hard armor steel.

- Zinc phosphate generates substantial sludge during operation. Throughout the crystallization process, the acids in a zinc phosphate bath chemically etch soluble steel from the substrate surface. The resulting pH gradient leads to both formation of zinc phosphate on the substrate and precipitation of insoluble iron phosphate in solution. The waste iron phosphate solids settle out of the tank, at which time they are filtered, pumped, or shoveled out to waste containers. Not only does waste sludge represent lost efficiency, but it can also include nickel, causing a hazardous waste designation and dramatic increases in the cost of disposal. Zirconium treatments do not generate hazardous sludge. Depending on local regulations, the baths are sent to drain after pH adjustment.
- With the designation of hexavalent chrome as a health and environmental toxin, zirconium pretreatments offer the ability to eliminate these concerns from the painting operations.

There are some limitations of zirconium technology.

- The thinness of the deposited films makes quantification more difficult. Conventional weigh/strip/weigh methods are not sufficiently sensitive.
- Performance of zirconium pretreatment on smooth, mild steel substrates is equal to or slightly worse than zinc phosphate, depending on primer technology.

The performance objectives, with acceptance criteria for the demonstrated technology, were evaluated in accordance with the tests described in the Joint Test Protocol. This demonstration involved both steel and aluminum substrates. The baseline reference pretreatment technologies are zinc phosphate, for steel processed at LEAD and Marine Depot Maintenance Command Production Plant, Albany (MDMC-PPA), hexavalent chromium, for aluminum processed at LEAD, and trivalent chrome passivation (TCP), for aluminum processed at MDMC-PPA.

Results from the demonstration testing showed that X-Bond 4000 met or exceeded the performance requirements of TT-C-490. In accelerated laboratory corrosion testing on steel substrates, X-Bond exhibited corrosion performance ranging from slightly worse than to equal to zinc phosphate. X-Bond showed improved corrosion performance versus blasting-only (direct to metal application), which is the current commercial practice on HHA steel, since zinc phosphate would cause embrittlement. On aluminum substrates, X-Bond met the performance requirements of MIL-DTL-5541 for conversion coatings, and it performed better than chrome III in laboratory testing. Its performance was equal to that of chrome VI. The same results were observed on outdoor exposure panels at the CCAFS

corrosion test site and at Camp Lejeune. ASTM B117 results illustrate the general trends observed throughout the program. Figure ES-3 shows performance of the zirconium pretreatment on blasted steel or 2024 T3 aluminum panels after 336 or 1000 h, respectively. In each case the zirconium pretreatment performed equivalent or better than controls.

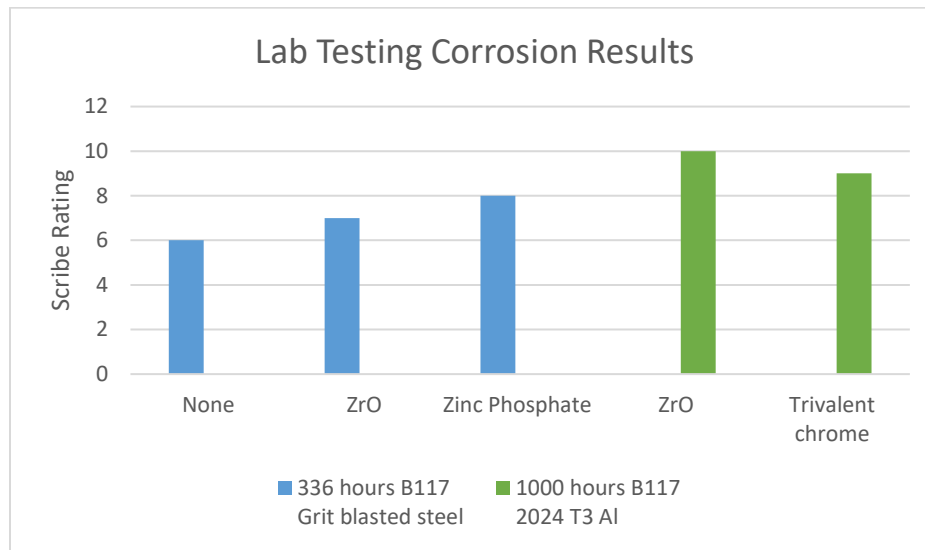


Fig. ES-3 Accelerated corrosion test results over steel and aluminum

Results of neutral salt spray testing in accordance with ASTM B117 are representative of overall performance of the zirconium-based pretreatment throughout this project. X-Bond performed better than blasted-only and equal to or slightly worse than zinc phosphate on CRS, but it generally met the specification requirements of a minimum rating of six for steel. On aluminum substrates, X-Bond performed better than chrome III and equal to chrome VI, meeting the minimum rating of eight for aluminum.

Cost Assessment

The demonstrations conducted at LEAD and MDMC-PPA utilized X-Bond 4000, a zirconium-based pretreatment from PPG, to pretreat both steel and aluminum parts. Separate pretreatments for steel and aluminum substrates are currently being used at both locations. LEAD uses Oakite Cryscoat OC zinc phosphate for steel and ChromiCoat L25 for aluminum, while MDMC-PPA uses Oakite Cryscoat CM for steel and Metalast TCP-HF for aluminum. The performance assessments conducted on the parts and panels from the demonstrations indicated that X-Bond 4000 pretreatment conforms to the requirements of TT-C-490 and MIL-DTL-5541, thus savings may be achieved and productivity enhanced by replacing separate

pretreatment approaches with a single product that can treat both ferrous and non-ferrous substrates.

Additional cost savings are achieved through a reduction of hazardous waste handling costs. The replacement of current pretreatments for aluminum would remove a significant source of chrome from the depots, hexavalent chrome at LEAD, and trivalent chrome at MDMC-PPA. A 2016 report to SERDP titled “Cadmium and Hexavalent Chromium Alternatives Implementation Plan for Letterkenny Army Depot, Contract # W9128F12-D-0041”, indicates that using a non-chrome pretreatment, such as X-Bond 4000, would eliminate 412 pounds of chrome or 40% of the total chrome usage annually from operations at LEAD. Elimination of the zinc phosphate processes would remove nickel-containing sludge, another serious hazardous waste present in the current processes.

The combination of effective corrosion performance and significant reduction in environmental impact can be achieved with an overall cost reduction in the operation of the pretreatment process. The cost analysis was based on data obtained from LEAD and resulted in a return-on-investment (ROI) calculation. Methods developed by the Office of the Secretary of Defense (OSD) Corrosion Policy Office (CPO) for funded projects were employed to assess ROI. This analysis compares baseline and new system costs, considers projected improvements in performance of the new system, and utilizes a 7% discount rate to determine the present-value (PV) of the project savings over a period of 30 years after implementation. The positive savings is then shown as a ratio to the project costs (investment), which provides the ROI for the project.

Utilizing the information provided by LEAD and the estimated costs for the zirconium pretreatment, the following were used for developing the ROI for this depot:

- Fixed/one-time costs
 - Baseline: \$0
 - Zirconium: \$117,500
- Annual operating costs
 - Baseline: \$194,995.61
 - Zirconium: \$107,183.82
- Performance benefits
 - Baseline: \$0
 - Zirconium: \$0

Excluding the one-time capital cost for a DI water system (\$117,500), the implementation of the zirconium pretreatment is estimated to have an annual

savings of \$87,811.79 at LEAD. This results in minimum estimated ROI of 2:1. The ROI is increased to 4:1 if the MDMC depot also converts operations to a zirconium-based pretreatment. Each subsequent depot adopting the technology further improves the ROI such that an ROI of 8:1 is possible with at least four depots adopting the technology.

Implementation Issues

The switch from zinc phosphate to zirconium pretreatment is a simple transition. Metal surfaces must be cleaned of oil and corrosion before application of the zirconium pretreatment and subsequent paint layers. This is accomplished by existing grit-blasting practices. Commercially available alkaline cleaners are also used to remove oil and dirt. The zirconium can be applied using the same tanks and spray systems already in place for chromium and zinc phosphate; however, thorough cleaning and rinsing of the tanks prior to zirconium treatment is required.

Process control is accomplished by monitoring pH, zirconium content, and iron content. Automated process controls can be used where high throughput necessitates frequent adjustment. The introduction of zirconium presents minimal risk, as the proposed zirconium products are already being manufactured and supplied in commercial quantities. Proper control of the zirconium process will ensure consistent quality. When using zirconium pretreatments to coat multiple substrates in the same process, compatibility in the X-Bond process should be checked. The control required is well within the capability of personnel currently applying zinc phosphate or chrome-based treatments.

1. Introduction

1.1 Background

Finishing systems for military vehicles include topcoats, which provide resistance to sunlight-driven polymer degradation; primers, which provide adhesion and corrosion resistance; and metallic substrates, which enhance adhesion and corrosion resistance. The focus of this demonstration was on metallic substrate pretreatments that are currently approved for use on military vehicles. For steel substrates, the existent pretreatment technology is zinc phosphate, as described in TT-C-490 Type I, including di- and tri-cation versions. Zinc phosphate provides a crystal structure to which organic coatings such as paints and powder coatings can adhere. To enhance corrosion protection, a sealer or rinse is often recommended. In most cases, the sealer or rinse products contain hexavalent chromium. For aluminum substrates, chromium-based pretreatments are more commonly employed; these include hexavalent (MIL-DTL-5541 Type I) and trivalent (MIL-DTL-5541 Type II) chromium products. PPG Industries, Inc. (PPG) has developed a pretreatment technology based on zirconium chemistry that is set up such that steel and aluminum substrates can be pretreated on the same line. This replacement offers several other advantages over present pretreatments, including the elimination of toxic metals such as nickel used in zinc phosphate and chromium. The zirconium pretreatments developed by PPG have been in commercial use on automotive and industrial paint lines for over 10 years and represent a mature technology for transition to military use.

Additional background information can be found in the final report for Strategic Environmental Research and Development Program (SERDP) Project WP-1676, in which several varieties of zirconium oxide pretreatments were developed for use with MIL-DTL-53022 Type II primers (MIL-DTL-53022 2012). This demonstration project built on the learning from WP-1676 and demonstrated the performance capabilities of zirconium pretreatment for protection of reconditioned material at US Army and US Marine Corps (USMC) depots. X-Bond 4000, a commercial pretreatment used with a variety of metal substrates and industrial finishes, was demonstrated.

1.2 Objective of the Demonstration

The goal of this project was to process and field test coupons or small components that demonstrated the corrosion resistance capabilities of a MIL-DTL-53072 (2013) finish, replacing the typical pretreating step (whether ferrous/zinc-phosphate or non-ferrous/chrome-functional) with a zirconium oxide pretreatment film. The

environmental benefits realized from the use of zirconium-based pretreatment, such as PPG's X-Bond 4000, in place of existing pretreatments include the following: elimination of nickel and hexavalent chromium compounds, decreased amount of sludge generated from operation of the pretreatment baths, reduced water and energy consumption, and a smaller footprint for pretreatment operations.

Performance validation for all parts tested was focused on the corrosion resistance, water and chemical resistance, and adhesion performance as required by pretreatment, primer, and full Chemical Agent Resistant Coating (CARC) system specifications as shown in Table 1.

Table 1 Demonstration part performance expectations

Target HazMat	Current process	Applications	Current specifications	Affected programs	Candidate parts and substrates
Nickel-containing zinc phosphate sludge	Zinc phosphate	Steel substrates	TT-C-490 MIL-DTL-53022 MIL-DTL-53030 MIL-DTL-53072	Depot repair operations using zinc phosphate	All-blasted Cold-rolled steel Hot-rolled steel Armor steel
Hexavalent Chromium (Cr6) from seal coat application					
Hexavalent Chromium (Cr6)	Aluminum conversion coating	Aluminum substrates	MIL-DTL-5541 MIL-DTL-53022 MIL-DTL-53030 MIL-DTL-53072	Depot repair operations for aluminum substrates	All-blasted AA2024-T3 AA6061-T6

1.3 Regulatory Drivers

The Occupational Safety and Health Administration (OSHA) Final Rules, effective May 30, 2006, Federal Register #71:10099-10385 (OSHA 2006) states in part that OSHA has amended the standard limiting occupational exposure to hexavalent chromium (Cr^{6+}). The final rule establishes an 8-h time-weighted average (TWA) exposure limit of 5 micrograms of Cr^{6+} per cubic meter of air ($5 \mu\text{g}/\text{m}^3$). This is a considerable reduction from the previous Permissible Exposure Limits (PELs) of 1 milligram per 10 cubic meters of air ($1 \text{ mg}/10 \text{ m}^3$ or $100 \mu\text{g}/\text{m}^3$) reported as CrO_3 , which is equivalent to a limit of $52 \mu\text{g}/\text{m}^3$ of Cr^{6+} . The final rule also contains ancillary provisions for worker protection, such as requirements for exposure determination, preferred exposure control methods, respiratory protection, protective clothing and equipment, hygiene areas and practices, medical

surveillance, and recordkeeping. The PEL established by this rule reduces the significant risk posed to workers by occupational exposure to Cr^{6+} to the maximum extent that is technologically and economically feasible.

In April 2009, a memo from the Office of the Secretary of Defense was released outlining a new policy for reducing the use of Cr^{6+} for US Department of Defense (DOD) applications. The memo specifically directs the military to do the following:

- 1) Approve the use of alternatives where they can perform adequately for the intended application and operating environment.
- 2) Update relevant technical documents and specifications to authorize the use of qualified alternatives.
- 3) Require Program Executive Office or equivalent in coordination with the Military Department's Corrosion Control and Prevention Executive to certify that there is no acceptable alternative to the use of Cr^{6+} on a new system.

Effectively, the memo directs DOD Military Departments to restrict the use of Cr^{6+} unless no cost-effective alternative with satisfactory performance has been identified.

In addition to eliminating Cr^{6+} on aluminum, this project sought to replace zinc phosphate on steel. One of the inevitable byproducts of a zinc phosphate line is sludge. Sludge is created when a quantity of the substrate being pretreated is dissolved into the general pretreatment bath solution. When floating freely, substrate ions readily react with the phosphates in solution and form insoluble materials free in suspension. An elevated concentration of suspended, insoluble phosphate particles interferes with proper deposition of the desired pretreatment coating, so these byproducts must be removed from the bath as they are generated. The sludge is largely made up of insoluble iron phosphate but also can contain phosphates of zinc, manganese, and nickel. Recently, increased attention has been given to more stringent regulations for waste disposal of nickel. Furthermore, any automotive-related zinc phosphating line that processes any amount of aluminum content classifies the associated sludge as "F019" waste by Code of Federal Regulations Title 40, part 261.31. This requires more expensive disposal in specially designed landfills capable of preventing the leaching of aluminum-laden wastes into the water table.

2. Demonstration Technology

2.1 Technology Description: X-Bond 4000

X-Bond 4000 is a hexafluorozirconic acid-functional pretreatment developed and commercialized by PPG. First commercialized in 2006, X-Bond 4000 was formulated for general use as a conversion coating for steel and aluminum. It relies on the ability of fluorozirconic acid (FZA) to etch the surface of the substrate, creating a localized pH imbalance and resulting in precipitation of zirconium oxide onto the surface. The zirconium oxide deposits, which adhere tightly to the underlying steel or aluminum, provide both physical texture and opportunities for noncovalent chemical interactions. These significantly improve adhesion and corrosion resistance relative to a clean, but untreated, surface. In addition, the zirconium oxide film is much thinner than zinc phosphate (Fig. 1), inherently consuming less raw material and reducing replenishment costs.

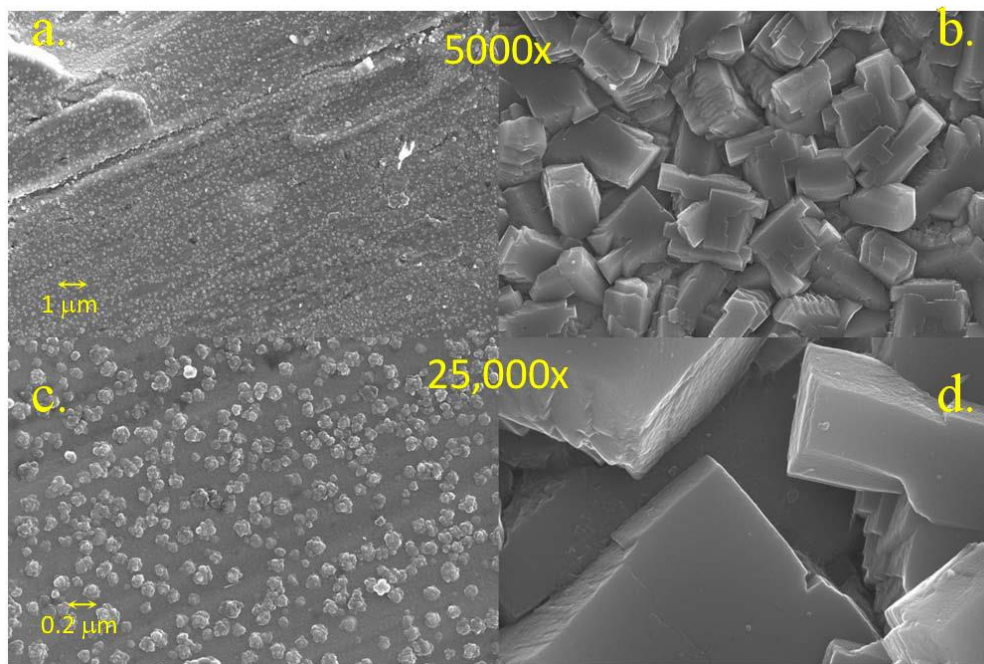


Fig. 1 Scanning electron microscope (SEM) images of zirconium and zinc phosphate pretreatments: a) zirconium-based pretreatment SEM image at 5,000× magnification, b) zinc phosphate SEM image at 5,000× magnification, c) zirconium-based pretreatment at 25,000× magnification, and d) zinc phosphate at 25,000× magnification

In existing paint lines, zirconium pretreatment technologies are generally used as drop-in replacements for the existent technology. Tanks set up to handle either zinc phosphate or Cr^{6+} conversion coatings are already capable of handling zirconium chemistries. A schematic of the process for X-Bond 4000 in the demonstration conducted at LEAD is shown in Fig. 2.

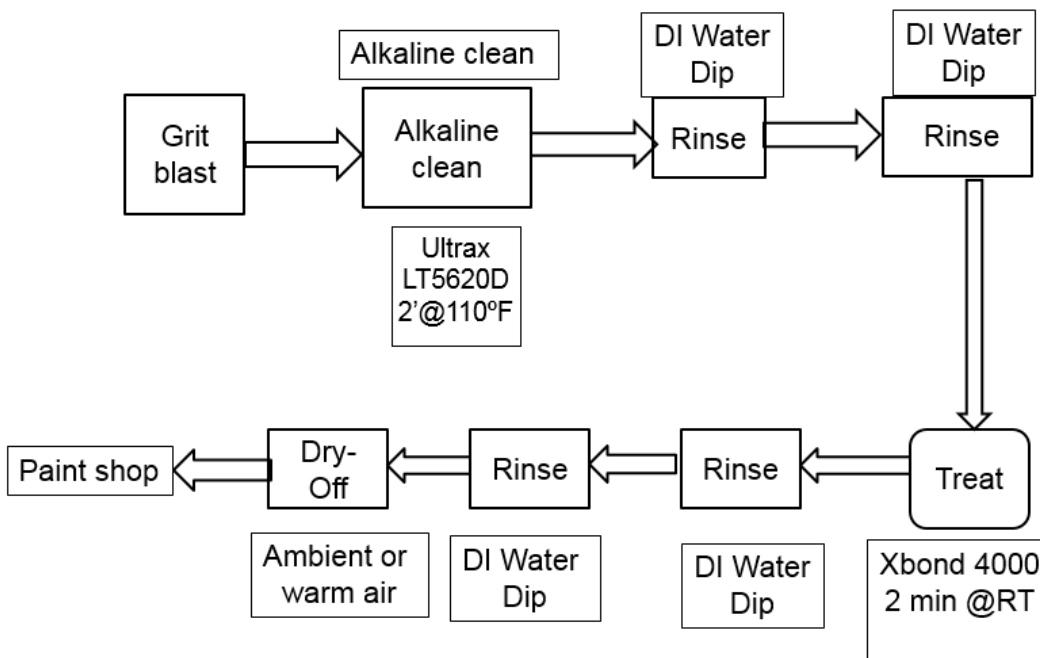


Fig. 2 Schematic of X-Bond 4000 process for the LEAD demonstration

2.2 Technology Development

The earliest development of zirconium-based pretreatment technologies dates back to Betz Laboratories in the 1980s. In 1990, the Man-Gill Chemical Company acquired rights to the Betz-developed use of zirconium as a conversion coating. Man-Gill continued development of the technology until their 1997 acquisition by PPG, at which point the first commercial zirconium product was in the process of being launched. The new PPG/Man-Gill organization commercialized zirconium technology in 1997 for use on aluminum beverage cans.

Following this success, the concept of zirconium oxide conversion coatings on steel and aluminum was taken up by the PPG pretreatment technology organization and went through a series of development phases for the general industrial and automotive markets.

Initial observations from competitive products and PPG pilot trials demonstrated a need to reduce the generation of iron oxide in zirconium systems used on mild steel, both in the process bath and on parts waiting to be painted. After development of a

now-patented solution to the iron oxide problem, PPG, led by the product development team at their Euclid, Ohio, laboratory and production facility, commercialized X-Bond 4000 in 2006. X-Bond was quickly adopted for use under electrocoat, powder, and liquid coatings.

The extremely thin nature of zirconium oxide films relative to zinc phosphate results in an associated reduction in electrical resistivity of the treated substrate. When used under electrodeposition products, this decreased resistivity has serious negative implications for the throwpower capabilities of established coating lines. After additional codevelopment between pretreatment and electrocoat technologists, in part to address the throwpower concern, the PPG Automotive Substrate Protection group commercialized Zircobond 4200 on an Original Equipment Manufacturer (OEM) body line in 2008. The technology there has been running successfully ever since, has been introduced to several additional lines, and has continued to gain ground as a zinc phosphate replacement for automotive OEM body painting (electrocoating) lines.

Recent investigations of zirconium pretreatment technology have not been limited to the general industrial and automotive markets. Under multiple preceding DOD projects, zirconium conversion coatings have been investigated for military use over a broad range of substrates and under a wide variety of primers. These contracts include SERDP WP-1676 and TARDEC Advanced Corrosion W56HZV-09-C-0601. Substrates used include cold rolled steel, hot rolled steel, high hard armor (HHA) steel, galvanized steel, and several alloys of aluminum. Coatings investigated include electrodeposition, polyurea anti-chip thick films, powder, and zinc-rich and liquid solvent and waterborne primers.

It was the intent of this program to prove that zirconium pretreatment technologies are capable of delivering the performance expected from depot painting lines while reducing cost of operation, cost of waste handling, and the presence of undesirable transition and heavy metals, like nickel and chromium.

2.3 Advantages and Limitations of the Technology

The use of zirconium treatment technology offers several advantages over current technologies:

- Zirconium treatments can be used at room temperature, reducing the energy costs of maintaining heated tanks.
- The relatively thin films of the zirconium pretreatments compared to zinc phosphate pretreatments consume significantly less raw material.

- Zinc phosphate baths cannot accommodate both ferrous and non-ferrous substrates in the same bath. In contrast, zirconium treatments can handle both steel and aluminum in the same bath without the need for close monitoring of the bath composition. This provides greater scheduling and processing flexibility to the depots and reduces the footprint of the painting process.
- The operating pH of zinc phosphate processes can generate hydrogen gas when treating steel, which leads to embrittlement. The pH of zirconium treatment is high enough (4.0–5.0) that it does not generate hydrogen gas and therefore can be used to treat HHA steel.
- Zinc phosphate generates substantial sludge during operation. Throughout the crystallization process, the acids in a zinc phosphate bath chemically etch soluble steel from the substrate surface. A pH gradient is created, leading to both formation of zinc phosphate on the substrate and precipitation of insoluble iron phosphate in solution. The waste iron phosphate solids are periodically allowed to settle out of the tank, at which time they are filtered, pumped, or shoveled out to waste containers. Not only does waste sludge represent lost efficiency, but it can also include nickel, causing a hazardous waste designation and dramatic increases in the cost of disposal. Zirconium treatments do not generate hazardous sludge. Depending on local regulations, the baths can usually be sent to drain after pH adjustment.
- With the designation of hexavalent chrome as a health and environmental toxin, zirconium pretreatments offer the ability to eliminate these concerns from the painting operations.

There are some limitations of zirconium technology:

- The thinness of the deposited films makes quantification more difficult. Conventional weigh/strip/weigh methods are not sufficiently sensitive.
- Performance of zirconium pretreatment on smooth, mild steel substrates is equal to or slightly worse than zinc phosphate, depending on primer technology.

3. Performance Objectives

The performance objectives, with acceptance criteria for the demonstrated technology, were evaluated in accordance with the tests described in the Joint Test Protocol and summarized in Tables 2 and 3. This demonstration involved both steel and aluminum substrates. The baseline reference pretreatment technologies are zinc phosphate, for steel processed at LEAD and MDMC-PPA, hexavalent chromium, for aluminum processed at LEAD, and trivalent chrome passivation (TCP), for aluminum processed at MDMC-PPA.

The major requirements for testing were chosen from the appropriate specifications covering pretreatments of steel and aluminum. The specifications were TT-C-490 (1961), *Chemical Conversion Coatings and Pretreatments for Metallic Substrates (Base for Organic Coatings)*, for ferrous substrates and MIL-DTL-5541F (2009), *Chemical Conversion Coatings on Aluminum and Aluminum Alloys*, for aluminum substrates.

During the demonstration at MDMC-PPA, all of the panels primed with MIL-DTL-53030 were inadvertently topcoated with CARC topcoat MIL-DTL-64159, so primed-only panels were not available for testing. Testing was carried out on the fully finished panels. Phase II is extended testing, consisting of environmental testing or in-service testing. This testing calls for using substrate coated with both primer and topcoat.

In summary and as outlined in Tables 2 and 3, the X-Bond pretreatment satisfied the performance requirements of the demonstration. X-Bond met the corrosion, adhesion, flexibility, and chemical resistance requirements for TT-C-490 Type IV inorganic conversion coating on steel for MIL-DTL-5541 Type II on aluminum. Exposure for 12 months in outdoor marine environments and on vehicles confirmed that the performance of X-Bond pretreatment is equivalent to control pretreatments.

Table 2 Summary of engineering, performance, and testing requirements

Phase	Engineering requirement	Test	JTP section	Acceptance criteria	References	Results
I	Corrosion resistance	Neutral salt fog – flat panels	3.1	336–1,000–2,000–3,000 h, minimal scribe corrosion, no loss of adhesion, and no blisters, ratings of ≥ 6 for ferrous and ≥ 8 for aluminum	ASTM B117, ASTM D1654, ASTM D714	Pass (See Sections 6.2, 6.3)
I	Corrosion resistance	Cyclic corrosion – flat panels	3.2	40–60–120 cycles, minimal scribe corrosion, no loss of adhesion, and max of 5 scattered blisters, ratings of ≥ 7 for ferrous and ≥ 8 for aluminum	GMW 14872, ASTM D1654, ASTM D714, ASTM G1	Pass (See Sections 6.2, 6.3)
I	Adhesion	Dry tape adhesion (cross-cut)	3.3	Equivalent or improved performance compared to pretreatment and primer controls, 4B rating minimum.	ASTM D3359 Method B	Pass
I	Flexibility	Mandrel bend	3.5	No cracking or delamination from substrate or intercoat adhesion loss, as compared to baseline system.	ASTM D522 Method B	Pass
I	Water immersion	Pencil hardness after immersion	3.7	≤ 2 Pencil hardness difference from an unexposed film; no blistering or delamination of un-scribed coating area.	ASTM D3363, ASTM D1308	Pass
I	Hydrocarbon immersion (JP-8)	Pencil hardness after immersion	3.8	≤ 2 Pencil hardness difference from an unexposed film; no blistering or delamination of un-scribed coating area	ASTM D3363	Pass
I	Hydrogen embrittlement	Mechanical hydrogen embrittlement	3.9	No fracturing after 200 h at 75% of nominal tensile strength	ASTM F519-12	Pass

Table 3 Extended performance and testing requirements

Phase	Engineering requirement	Test	JTP section	Acceptance criteria	References	Results
II	Corrosion	Marine environment outdoor exposure	3.10	Three years of exposure: Rated every 3 months for first year and 6 months thereafter. Specimen will be rated as less or more creepage from scribe than current corrosion protection system	Approved test site standard practice: ASTM G50 ASTM D1654 ASTM D714	See Section 6
II	Corrosion and adhesion	Field exposure, on-vehicle	3.11	Exposure time of two years on vehicle platform or panels attached to platform. Equivalent or improved performance compared to baseline pretreatment controls	ASTM D1654 ASTM D714	See Section 6

3.1 ASTM B117 Neutral Salt Spray: Flat Panels

ASTM B117 calls for parts and panels to be continuously sprayed with 5% NaCl fog at 95 °F over a specified time period after which they are inspected for coating adhesion loss/blistering on the flat surface and along a line scored across the face of the panel or part (ASTM B117 2007). *Evaluation of Painted or Coated Specimens Subjected to Corrosive Environments* (ASTM D1654 2008) describes the procedure for preparing the scribed panels. All governing military coating specs require an X-scribe on panels. The X-scribed lines cut through the paint and treatment to the base metal. Due to the small surface areas available on some parts, a single line scribe was used. After the required exposure, corrosion performance is rated using ASTM D1654 Procedure A. The duration of the test depends on the substrate and primer being used. Procedure A is used to assign a numerical rating based on the degree of blistering along the scribed lines. The primer specifications call for minimum average scribe-creep values. These values also provide a means of comparing the relative corrosion resistance of the test specimen against a system of known performance. No reliable correlation between salt spray performance and field performance has been fully established.

X-Bond 4000 passed the requirement of a minimum average scribe creep rating greater than or equal to eight and was equal to hexavalent chrome and better than trivalent chrome on aluminum parts and panels. Steel panels and parts treated with

X-Bond 4000 also passed the scribe creep minimum average rating of six and received ratings equal to or slightly worse than zinc phosphate. On HHA, X-Bond 4000 performed much better than blasted-only and equal to or slightly worse than zinc phosphate, while still meeting the specification requirement.

3.2 Cyclic Corrosion: Flat Panels

This test describes a field-correlated, laboratory corrosion method for determining cosmetic corrosion performance. The method uses cyclic conditions (immersion in salt solution, temperature, and humidity) to mimic outdoor conditions and accelerate the corrosion process. Panel preparation is identical to that used in ASTM B117, described previously. The panels are subjected to the cyclic environment for the number of cycles prescribed in the primer specifications. After exposure, the test specimens are rated for scribe creep, again using ASTM D1654 Procedure A.

Results of the cyclic corrosion testing showed X-Bond 4000 passed the minimum rating of six. One set of HHA panels from Letterkenny with primer-only received a rating of five but still was better than the blasted-only set. Zinc phosphate passed with an average rating of seven.

3.3 Dry Tape Adhesion: Cross Cut

This method describes the procedure for establishing adequacy of intercoat and surface adhesion of an organic coating in ambient conditions by applying a pressure-sensitive tape over a scribed area. In order for a coating system to perform its protective and decorative functions, it must adhere to the targeted surface. This test provides a semi-quantitative evaluation of adhesion. According to ASTM D3359 Method B, a knife is used to cut a grid pattern through the coating layers down to the metal (ASTM D3359 2009). A standard adhesive tape is used to pull any loosened layers from the substrate. Numerical ratings are assigned based on comparison to a visual standard. 3M Scotch 898 Filament tape was used for all adhesion testing.

3.4 Flexibility: Mandrel Bend

This test evaluates resistance to cracking (flexibility) of organic coatings applied to pretreated, sheet metal substrates. Testing was carried out using ASTM D522 Method B (ASTM D522 2008). The coated panels/coupons were bent over a 0.25-inch mandrel. The samples were then examined for cracks or delamination of the coating over the area of the bend. No cracking or flaking is permitted.

3.5 Water Resistance

This test evaluates the resistance of the coating system to adhesion loss, color change, and gloss change after immersion in water for one week at room temperature. According to MIL-DTL-53072, the coating will show no difference in appearance or adhesion after immersion when compared to a non-immersed sample (MIL-DTL-53072 2013).

3.6 Pencil Hardness after Water Immersion

This test method (ASTM D3363 2011) details a procedure for rapid, qualitative determination of the film hardness of an organic coating on a substrate using drawing or pencil leads of known hardness. As detailed in MIL-DTL-53022 and MIL-DTL-53030 primer specifications, finished panels are immersed in deionized (DI) water for one week at room temperature. After removing the panel and drying for 24 h, the pencil hardness is measured and compared to an area that was not immersed. No difference greater than two-pencil hardness is permitted. This test is useful in identifying coating systems that are likely to absorb water, leading to physical damage in high moisture environments and possibly corrosion.

Results of the testing showed all samples passing.

3.7 Pencil Hardness after Hydrocarbon Immersion

This test is similar to the water immersion test. Instead of DI water, the painted panels are immersed in JP-8 (Jet Propellant 8) for one week at room temperature. After removal, the panels are allowed to dry. After 2 h air drying, the immersed coating is tested for pencil hardness, discoloration, and adhesion using ASTM D3363. No softening greater than two-pencil hardness difference is permitted.

As with water immersion, all samples passed JP-8 immersion.

3.8 Hydrogen Embrittlement of Steel after X-Bond Treatment and Fully Finishing ASTM F519

While the demonstration plan called for testing hydrogen embrittlement using E399-97, military specification TT-C-490 calls for testing using ASTM F519. Consequently, hydrogen embrittlement testing was carried out using ASTM F519 (*Standard Test Method for Mechanical Hydrogen Embrittlement Evaluation of Plating/Coating Processes and Service Environments*). This method calls for treating and/or finishing notched bars of AISI E4340 steel. These bars are manufactured to a nominal notch tensile strength. After finishing, the bars are placed under a load of 75% of their nominal notch tensile strength for 200 h.

If there is no sign of failure (i.e., cracking or change of dimensions), the finish is considered non-hydrogen embrittling.

For the demonstration, notched bars were obtained from Assured Testing Services of Ridgway, Pennsylvania. The notched bars were treated/finished in the same manner as the panels and parts at LEAD. Three bars were treated only with zirconium and three were finished with zirconium treatment and MIL-DTL-53022 primer. The notched bars were returned to Assured Testing Services for tensile testing. It was reported that all samples passed tensile testing. Therefore, the X-Bond 4000 treatment process does not cause hydrogen embrittlement. A copy of the test report from Assured Testing Services is given in Appendix B.

3.9 Marine Environment Outdoor Exposure

This test method evaluates the ability of a coating system to prevent substrate corrosion. Outdoor exposure testing was conducted at the Cape Canaveral Air Force Station (CCAFS) corrosion test site. This site is located 100 yards from and faces the Atlantic Ocean. Test coupons were installed on either wooden or metal racks using plastic insulator stand-offs with stainless steel fasteners. The rack angle of the coupons is 30° from horizontal.

Coupons were evaluated periodically for surface corrosion (blisters in field) and creep from scribe, as per ASTM D1654, at six-month intervals for one year. At each inspection interval, the surfaces of the coupons were visually examined. Corrosive salts or oxides from the scribes were ignored in these measurements. The inspection was more concerned with perforation of the film than with staining. Photos were taken of test coupons at each inspection interval. Coupons were left on exposure for an additional 12 months. Results confirmed laboratory that X-Bond 4000 zirconium pretreatment was equivalent to or better than control pretreatments.

3.10 Field Exposure, On-Vehicle

This test method describes a basic procedure for conducting on-vehicle testing of candidates. This may be performed by selective replacement or refinishing of a representative substrate/component on a vehicle incorporating the candidate or by the use of test specimens incorporating the candidate attached to the military ground vehicle. The ability of the coating system to maintain adhesion to the substrate after exposure to field conditions and to prevent corrosion was analyzed. Components were evaluated during periodic inspections by visual comparison with the base vehicle or control samples attached to the vehicle. *Standard Method for Evaluating the Degree of Rusting on Painted Steel Surfaces* (SSPC-VIS-2 2008), was used for evaluating component substrates and control samples. The success criteria for field

testing was performance greater than or equal to the baseline. Results confirmed laboratory testing that X-Bond 4000 zirconium pretreatment was equivalent to or better than control pretreatments.

4. Site/Platform Descriptions

4.1 Test Platforms/Facilities

ARL chose facilities at which the demonstrations would take place, based on the need to eliminate chrome and phosphate, the types of substrates being processed, and the production schedule. Before deciding on the exact locations within the designated test facilities, an inspection tour was conducted at the facilities.

4.1.1 Letterkenny Army Depot (LEAD)

LEAD has the ability to completely disassemble, replace, repair, refinish, and reassemble a number of ground vehicle assets that include High Mobility Multi-Purpose Wheeled Vehicles (HMMWVs), High Mobility Artillery Rocket Systems (HMARs), Ground Mobility Vehicles (GMVs), Mine-Resistant Ambush Protected Vehicles (MRAPs), Biological Integrated Detection Systems (BIDS), Avengers (Army air defense), Patriot and Hawk (Defense) missile systems, and material handling equipment.

4.1.2 MDMC-PPA

MDMC-PPA has the mission to service all kinds of assault vehicles, combat vehicles, ordnance systems, small arms, automotive vehicles, and engineering equipment. Like LEAD, it can completely disassemble, replace, repair, refinish, and reassemble a number of ground vehicle and defense systems used by the USMC.

The service parts demonstrated at MDMC-PPA include steel-lift, hook retainer D-rings located on the hoods of HMMWVs (Fig. 3) as well as test coupons designed for mounting on exposure racks fixed to the lower hitch/frame areas on HMMWVs and MTRVs (Fig. 4). These were actual service vehicles stationed at Camp Lejeune for ease of tracking and inspection.

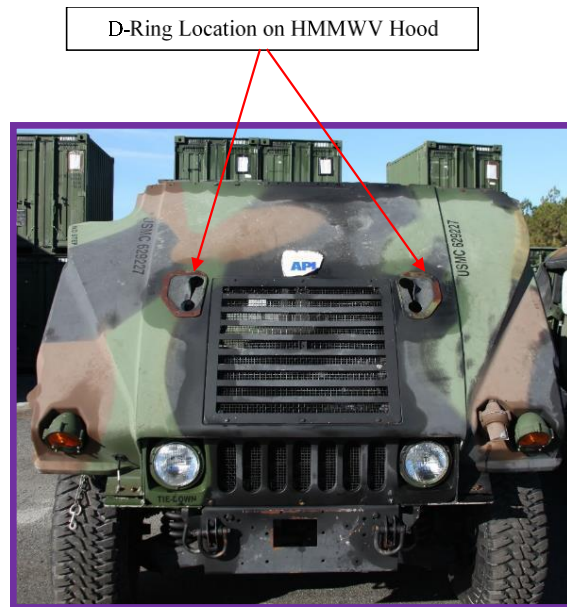


Fig. 3 Location of steel D-rings on HMMWVs at Camp Lejeune

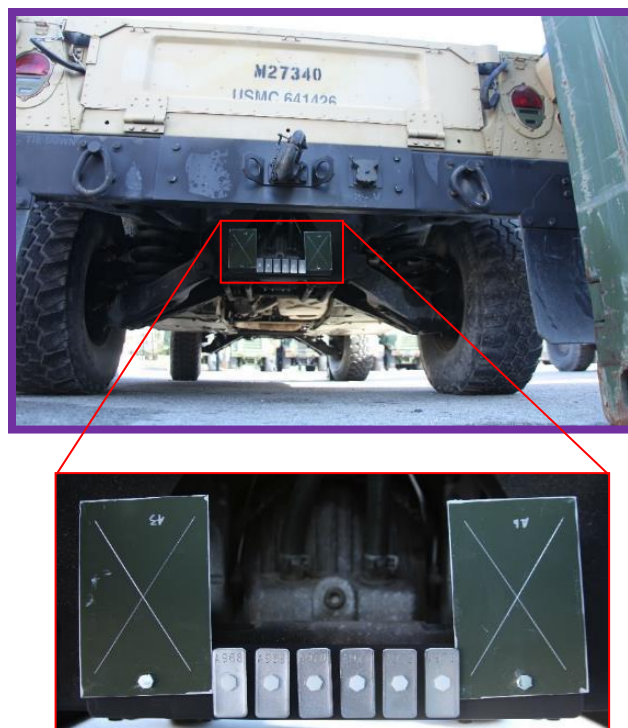


Fig. 4 Location of test panel racks on HMMWVs at Camp Lejeune

Parts from LEAD to be put into service on vehicles were intended to include a mixture of substrates, both ferrous and non-ferrous. However, none were available at LEAD at the time of the demonstration. Demonstration parts and coupons from LEAD were evaluated in natural and accelerated corrosion testing by ARL, the primary investigating authority for this project, with support from secondary investigators.

4.2 Current Operations

4.2.1 LEAD

Building 370 houses a small pretreatment line equipped with 500-gal process tanks. All of the tanks were occupied by actively used chemicals. Building 350 houses a larger pretreatment line using 3,000-gal tanks that were also filled with actively operating chemicals. To limit downtime and simplify this half of the demonstration, it was decided that portable tanks would be used for the zirconium demonstration at LEAD in Building 350 where floor space was available near the paint line. This allowed for the use of lined, 55-gal drums as temporary process, avoiding the need to drain, clean, and refill any of the active system tanks.

4.2.2 MDMC-PPA

The stripper building at MDMC-PPA houses several empty 1,500-gal process tanks with low production activity in that area. A specific part was chosen for processing—the rear, ladder-cover access panel from the MTRV. At the time of the demonstration, there were not sufficient quantities of the access panel to complete the demonstration testing. Instead, CRS D-ring lift hook surrounds on HMMWVs were chosen. These parts are easily installed, inspected, and replaced. The vehicles with the D-rings were used to mount panel racks loaded with demonstration test panels, bare corrosion coupons for reference, and coated fasteners being co-demonstrated under ESTCP Project No. WP-201315.

Both LEAD and MDMC-PPA are current users of zinc phosphate for steel and hexavalent chromium for aluminum. Both sites presented excellent opportunities to demonstrate a multi-metal capable pretreatment technology that has the potential to streamline operations, reduce cost, and eliminate toxic materials, all without sacrificing appreciable corrosion protection.

The current process at both LEAD and MDMC-PPA begins with preparing parts by either direct abrasive blasting or chemical stripping followed by a finishing abrasive blast. Alternatives to abrasive blasting (e.g., DA orbital sanding) may be used provided they do not adversely affect coating weight, salt spray, or hydrogen

embrittlement properties. Once the parts are properly cleaned and prepared, they are phosphated and allowed to air dry for several hours, most often overnight. The parts are then primed and painted. If the unpainted parts will be stored long term, a corrosion-preventive compound or preservative lubricating oil is applied. Figure 5 shows a typical flow diagram of the zinc phosphating lines currently employed at LEAD and MDMC-PPA.

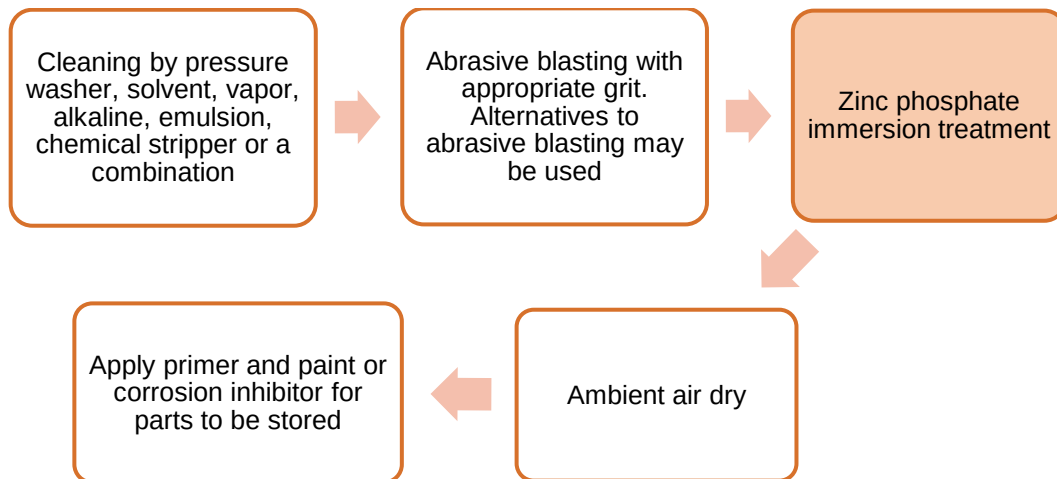


Fig. 5 Flow diagram of a typical phosphate line as currently employed at LEAD/MDMC-PPA

Treatment of aluminum substrates at both facilities is essentially the same, except for the use of an immersion chrome conversion coating in place of the zinc phosphate. LEAD uses a hexavalent chrome pretreatment, while MDMC-PPA uses a trivalent chrome pretreatment.

HHA steel is the exception to the diagram shown in Fig. 5. Because of the possibility of hydrogen embrittlement caused by immersion into the acidic phosphate bath, HHA steel generally goes directly from cleaning/blasting to painting. On this substrate, zinc-rich epoxy primers are often used under the standard epoxy primer to improve performance in the absence of pretreatment.

Since zirconium immersion baths generally run higher in pH than zinc phosphate baths and substantially less hydrogen is evolved during the nano-scale zirconium oxide deposition, it is believed that zirconium pretreatment will be much less likely to embrittle HHA steel. It is the intention of this demonstration plan to include HHA for embrittlement testing.

4.3 Site-Related Permits and Regulations

Additional site-related permits or regulations are not anticipated for the demonstration to be conducted at either LEAD or MDMC-PPA. The facilities have the capability to process and apply pretreatments, including hexavalent chrome pretreatments, and hold the necessary documentation to perform the demonstrated chemical pretreatments and dispose of any waste as necessary.

4.4 Demonstration Execution

As previously stated, the demonstrations were carried out using 55-gal drums. The steps for applying the zirconium pretreatment were as follows:

- 1) Alkaline cleaning
- 2) City water rinse
- 3) DI water rinse
- 4) X-Bond 4000 bath
- 5) City water rinse
- 6) DI water rinse
- 7) Air dry

Figure 6 shows the setup of the X-Bond cleaning and treatment steps at LEAD with the lined steel drums containing the cleaner, rinse, and treatment stages.



Fig. 6 Setup of the X-Bond cleaning and treatment process at LEAD

Details of the process were as follows:

- 1) Alkaline cleaning was carried out using PPG's Ultrax LT5620D low temperature, immersion cleaner. The cleaner was heated to 100 °F using a drum heater and agitated with an air mixer, fitted with a four foot, pitched-blade agitator (Fig. 7). Parts and panels were hung from steel bars using metal hooks. Panels had 0.25-inch holes predrilled to accommodate the hooks (Fig. 8).



Fig. 7 Cleaner bath setup for demonstrations at LEAD



Fig. 8 Panels in cleaner tank at LEAD demonstration

- 2) The first rinse was carried out using the facility's tap water.
- 3) The second rinse used DI water, supplied by PPG, delivered in a 500-gal tote tank.

- 4) X-Bond immersion was carried out in a lined drum with agitation. The X-Bond bath was prepared according to the supplied data sheet as a 2% volume solution using DI water. The pH of the bath was adjusted to the working range of 4.8–5.2 using Chemfil buffer. Pretreatment was carried out for 2 min.
- 5) After treatment the panels were rinsed for 30 s in city water.
- 6) The final rinse was 30 s in DI water.
- 7) Parts and panels were hung on rolling racks to air dry (Fig. 9).



Fig. 9 Panels drying on portable rack after zirconium treatment at LEAD

Observations: The demonstrations were carried out over several days. The baths maintained stable pH over that time. At MDMC-PPA, a drum liner in the X-Bond bath leaked causing the iron content to rise overnight to an unacceptable level, so a new bath was prepared on the second day. The parts and panels processed through the pretreatment as expected. After drying, they had the characteristic gold-blue color. No flash rusting was observed on the steel in spite of high ambient temperature and humidity, especially at MDMC-PPA. Some panel sets that were originally primer only received a topcoat at MDMC-PPA. It was determined to test them as originally planned because the affected sets were meant to be direct comparisons of pretreatments. Information could still be gathered from those panel sets.

5. Test Design

5.1 Design of Demonstration

After the inspection and documentation of the current finishing processes at the depots, a test matrix for each location was developed. This was done to ensure that the use of zirconium was evaluated in the most significant coating systems at each location. The demonstration matrix is laid out in Tables 4 and 5.

Table 4 Design matrix for LEAD demonstration of X-Bond 4000

Code	Substrate	Treatment	Primer	Topcoat
C2	CRS	None	MIL-DTL-53022	None
C3	CRS	None	MIL-DTL-53022	MIL-DTL-53039
C5	CRS	X-Bond	MIL-DTL-53022	None
C6	CRS	X-Bond	MIL-DTL-53022	MIL-DTL-53039
C8	CRS	Zinc phosphate	MIL-DTL-53022	None
C9	CRS	Zinc phosphate	MIL-DTL-53022	MIL-DTL-53039
A2	Aluminum	X-bond	MIL-DTL-53022	None
A3	Aluminum	X-bond	MIL-DTL-53022	MIL-DTL-53039
A5	Aluminum	Cr ⁶⁺	MIL-DTL-53022	None
A6	Aluminum	Cr ⁶⁺	MIL-DTL-53022	MIL-DTL-53039
H2	HHA	None	MIL-DTL-53022	None
H3	HHA	None	MIL-DTL-53022	MIL-DTL-53039
		None	Zinc-rich+	
H4	HHA		MIL-DTL-53022	MIL-DTL-53039
H6	HHA	X-bond	MIL-DTL-53022	None
H7	HHA	X-bond	MIL-DTL-53022	MIL-DTL-53039
		X-bond	Zinc-rich+	
H8	HHA		MIL-DTL-53022	MIL-DTL-53039
			Zinc-rich+	
H10	HHA	Zinc phosphate	MIL-DTL-53022	None
H11	HHA	Zinc phosphate	MIL-DTL-53022	MIL-DTL-53039
			Zinc-rich+	
H12	HHA	Zinc phosphate	MIL-DTL-53022	MIL-DTL-53039

Table 5 Design matrix for MDMC-PPA demonstration of X-Bond 4000

Code	Substrate	Treatment	Primer	Topcoat
S2	CRS	None	MIL-DTL-53030	None
S3	CRS	None	Zinc-rich+ MIL-DTL-53030	None
S4	CRS	None	MIL-DTL-53030	MIL-DTL-64159
S5	CRS	None	Zinc-rich+MIL-DTL-53030	MIL-DTL-64159
S7	CRS	X-Bond	MIL-DTL-53030	None
S8	CRS	X-Bond	Zinc-rich+MIL-DTL-53030	None
S9	CRS	X-Bond	MIL-DTL-53030	MIL-DTL-64159
S10	CRS	X-Bond	Zinc-rich+MIL-DTL-53030	MIL-DTL-64159
S11	CRS	X-Bond	MIL-DTL-53022	MIL-DTL-64159
S12	CRS	X-Bond	MIL-PRF-32348	MIL-DTL-64159
S14	CRS	Zinc phosphate	MIL-DTL-53030	None
S15	CRS	Zinc phosphate	Zinc-rich+MIL-DTL-53030	None
S16	CRS	Zinc phosphate	MIL-DTL-53030	MIL-DTL-64159
S17	CRS	Zinc phosphate	Zinc-rich+MIL-DTL-53030	MIL-DTL-64159
S18	CRS	Zinc phosphate	MIL-PRF-32348	MIL-DTL-64159
S19	CRS	Zinc phosphate	MIL-DTL-53022	MIL-DTL-53039
S20	D-Rings	X-bond	MIL-DTL-53030	MIL-FTL-64159
S21	D-Rings	Zinc phosphate	MIL-DTL-53030	MIL-FTL-64159
M2	Aluminum	X-bond	MIL-DTL-53030	None
M3	Aluminum	X-bond	MIL-DTL-53030	MIL-DTL-64159
M4	Aluminum	X-bond	MIL-PRF-32348	MIL-DTL-64159
M5	Aluminum	X-bond	MIL-DTL-53022	MIL-DTL-53039
M7	Aluminum	Cr ³⁺	MIL-DTL-53030	MIL-DTL-64159
M8	Aluminum	Cr ³⁺	MIL-DTL-53030	MIL-DTL-64159
M9	Aluminum	Cr ³⁺	MIL-PRF-32348	MIL-DTL-64159
M10	Aluminum	Cr ³⁺	MIL-DTL-53022	MIL-DTL-53039
M11	Al Bracket	X-bond	MIL-DTL-53030	MIL-DTL-64159
M11	Al Bracket	Cr ³⁺	MIL-DTL-53030	MIL-DTL-64159
A2	HHA	None	MIL-DTL-53030	None
A3	HHA	None	MIL-DTL-53030	MIL-DTL-64159
A4	HHA	None	Zinc-rich+MIL-DTL-53030	MIL-DTL-64159
A6	HHA	X-bond	MIL-DTL-53030	None
A7	HHA	X-bond	MIL-DTL-53030	MIL-DTL-64159
A8	HHA	X-bond	Zinc-rich+MIL-DTL-53030	MIL-DTL-64159

5.2 Laboratory Testing

5.2.1 ASTM B117 Standard Practice for Operating Salt Spray (Fog) Apparatus

The fog chamber for this test was run in accordance with ASTM B117 (Table 6).

This test calls for placing painted parts or panels in a chamber in which a neutral 5% salt solution fog is continuously injected into a cabinet maintained at a constant temperature. Five separate test specimens were used for performance testing. Test panels were cut to 102- × 152-mm (4- × 6-inch) test size. An “X” incision was scribed through the coating to the substrate. The resultant incision was approximately 0.2 mm in width. Bare edges of the coupons were coated with PPG Multiprime 97-680 air-dry, epoxy primer to prevent corrosion products from contaminating the chamber. Parts were placed in the chamber as received. Where possible a scribed line was made on the part. The test specimens were placed in the chambers on racks at an angle of 30° from vertical with the scribed surface facing upwards. The 5% salt solution was prepared as specified in ASTM B117 in the pH range of 6.5–7.2. Exposure time was mandated by the primer and pretreatment specifications. At the conclusion of the exposure period, the test specimens were removed and cleaned by gently flushing with running tap water. Samples were scraped side-to-side with a scalpel at a 30° contact angle. Test specimens were evaluated for corrosion resistance and creepage in accordance with the latest version of ASTM D1654, *Standard Test Method for Evaluation of Painted or Coated Specimens Subjected to Corrosive Environments*. Samples were rated for corrosion or loss of coating extending back from the scribe mark; unscribed areas were evaluated for corrosion spots, blisters, and any other types of failure. The ASTM D1654 rating system was used to rate scribed areas, and the ASTM D714 rating system was used for unscribed areas (ASTM D714 2009). Samples were photographed after rating.

Rationale

The neutral salt-fog test on scribed, painted substrates is a key accelerated test to compare the overall corrosion performance against currently used systems.

Equipment and Instrumentation

- **Neutral Salt Spray (Fog) Chamber.** This equipment consists of a heated fog chamber, a salt solution reservoir, a supply of conditioned (oil- and contaminant-free) compressed air, atomizing nozzles, and specimen supports.

- **Imaging System.** A means of visually recording corrosion effects on all tested specimens, such as a digital camera or scanner/software system.
- **Scribe Tool.** An ANSI B94.50, style E scribe.
- **Straightedge.** Any straightedge of sufficient length to guide the scribing tool in a straight line across the specimen surface.
- **Air Source.** A source of clean, dry, compressed air capable of delivering at least 10 cfm at 80 psi.
- **Air Gun and Guard.** An air dusting gun and nozzle combination to meet the specification in ASTM D1654. A guard to protect the operator, such as a sandblasting cabinet.
- **Scale.** A ruler with 1-mm (0.04-inch) divisions.
- **Putty Knife.** Blunt-edged, 38 mm (1.5 inch) wide.

Table 6 ASTM B117 salt spray test methodology

Parameters	• Test coupons at a 15° angle • Temperature of exposed salt spray zone = 35 ± 1.7 °C (95 ± 3 °F) • Every 80-cm² horizontal area, two collectors gather 1.0-2.0 ml fog/hr. • 5% salt solution (5 ± 1 parts by weight of NaCl in 95 parts of water). • pH = 6.5–7.2 when atomized at 35°C (95°F) • Duration of testing varies by substrate, pretreatment, and primer
Number and type of specimens per candidate alternative	Five of each variable were tested. Values are reported as the average of all five samples.
Trials per specimen	1
Experimental control specimens	Zinc phosphate pretreatment according to TT-C-490 for ferrous panels and conversion coating conforming to MIL-DTL-5541 for aluminum panels.
Acceptance criteria	Scribe rating of ≥ 6 for ferrous substrates and ≥ 8 for aluminum. Performance equal to or better than control zinc phosphate or conversion coating over similar substrates and under similar primers.

Data Analysis

Ratings, as described in Table 7, were based on condition of scribe, amount and size of undercutting, and number and size of face blisters. The rating system used to quantify results was frequency of blisters measured according to ASTM D714.

Table 7 ASTM D1654-08 corrosion rating system

TABLE 1 Rating of Failure at Scribe (Procedure A)		
Representative Mean Creepage From Scribe		
Millimetres	Inches (Approximate)	Rating Number
Zero	0	10
Over 0 to 0.5	0 to 1/64	9
Over 0.5 to 1.0	1/64 to 1/32	8
Over 1.0 to 2.0	1/32 to 1/16	7
Over 2.0 to 3.0	1/16 to 1/8	6
Over 3.0 to 5.0	1/8 to 3/16	5
Over 5.0 to 7.0	3/16 to 1/4	4
Over 7.0 to 10.0	1/4 to 3/8	3
Over 10.0 to 13.0	3/8 to 1/2	2
Over 13.0 to 16.0	1/2 to 5/8	1
Over 16.0 to more	5/8 to more	0

5.2.2 GMW 14872 Cyclic Corrosion Laboratory Test

This cyclic test is intended to produce corrosion results in the laboratory that reproduce outdoor environments. This is accomplished by cycling the environmental chamber in which the samples are placed through steps consisting of salt spray, high temperature humidity, and high temperature dry-off as described in Figure 10.

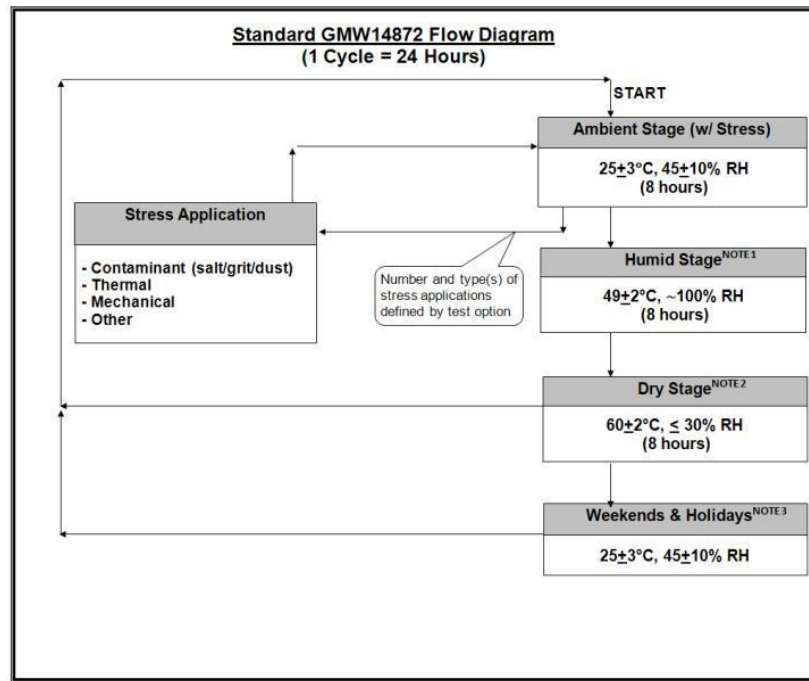


Fig. 10 GMW 14872 cycle process diagram

The cyclic fog chamber for this test was run in accordance with the Accelerated Corrosion Test (GMW 14872 2010), utilizing the “underbody” mode of the test cabinet operation and using four salt solution applications per 8-h ambient cycle (Table 8).

Table 8 GMW 14872 cyclic corrosion test methodology

Parameters	• Test coupons at 15° angle • Up to 120 cycles • 1 cycle: 24 h • Fog humidity: per GM 4465P at 49 ± 2 °C. • Salt solution: 0.9% sodium chloride, 0.1% calcium chloride, 0.25% sodium bicarbonate; pH of 6 to 9. • Drying environment: 60 ± 2 °C, < 30% relative humidity (RH); ambient environment: 25 ± 2 °C, 40-50% RH
Experimental control specimens	Zinc phosphate pretreatment according to TT-C-490 for ferrous panels and conversion coating conforming to MIL-DTL-5541 for aluminum panels.
Acceptance criteria	Scribe rating of ≥ 7 for ferrous substrates and ≥ 8 for aluminum. Performance equal to or better than control zinc phosphate or conversion coating over similar substrates and under similar primers.

Steel panels and components were used for test specimens. Five test specimens of each variable were selected for the test exposure duration. Test panels were cut to 102- × 152-mm (4- × 6-inch) test size. An “X” incision was scribed through the coating all the way to the substrate. Bare edges of the coupons were coated with PPG Multiprime 97-680 air-dry epoxy primer to prevent corrosion products from contaminating the chamber. The scribed test panels were placed in the chamber, leaning at an angle of approximately 45° from vertical with the scribed surface facing upwards. Salt solutions were prepared per GMW 14872 and pH was measured prior to the start of the test and on a weekly basis thereafter. Duration of exposure time was determined by the primer, substrate, and pretreatment specifications. The number of test cycles was specified in the primer specifications and in the TT-C-490 specification. Because both demonstration locations used MIL-DTL-53022 Type II primer, which has no cyclic corrosion performance requirement, cyclic corrosion testing was carried out to 20 cycles, at which point comparisons to the zinc phosphate could be made and performance compared. After exposure, the test specimens were thoroughly rinsed with DI water, while gently

scraping with a scalpel across the X-scribe to remove any loose coating or corrosion products. Scribe creepback values were determined using ASTM D1654. The unscribed areas were also evaluated for corrosion spots, blisters, and any other types of failure. Each test specimen was photographically documented.

Rationale

The cyclic, accelerated corrosion test on scribed, painted substrates is key to determining overall corrosion inhibition compared to controls. Testing at various cycles provides the information necessary to compare the performance of the zirconium oxide pretreatment to the baseline pretreatments.

Equipment and Instrumentation

- **Programmable Cyclic Environmental Test Chamber.** A chamber capable of controlling temperature and humidity, as well as spraying panels with salt fog in order to obtain and maintain the required environmental conditions as specified in GMW 14872.
- **Imaging System.** A means of visually recording corrosion effects on all tested specimens, such as a digital camera or scanner/software system.
- **Scribe Tool.** An ANSI B94.50, style E scribe.
- **Scale.** A ruler or caliper capable of measuring to within 1-mm (0.04-inch) divisions
- **Straightedge.** Any straightedge of sufficient length to guide the scribing tool in a straight line across the specimen surface.
- **Scalpel or Spatula.** Object with flat metal edge used to scrape paint from area of scribe.

Data Analysis

All information was reported as required in ASTM D714 and ASTM D1654, including the photographs from the imaging system.

5.2.3 ASTM D522 Mandrel Bend Flexibility Test

Test Description

This test, which is described in Table 9, evaluates the resistance to cracking (flexibility) of attached organic coatings on substrates of pretreated sheet metal. Flexibility is tested in accordance with ASTM D522 Method B. Coated specimens are bent over a 0.25-inch mandrel as seen in Figure 11. The coatings are examined for cracks or delamination over the area of the bend.

Rationale

This test method determines the resistance to cracking (flexibility) of attached organic coatings on metal substrates.

Table 9 Flexibility: mandrel bend test methodology

Parameters	• Cured primer test specimens • 1-inch (25.6-mm) mandrel
Experimental control specimens	Zinc phosphate pretreatment according to TT-C-490 for ferrous panels and conversion coating conforming to MIL-DTL-5541 for aluminum panels.
Acceptance criteria	Test specimen shall exhibit no cracking or delamination when tested.

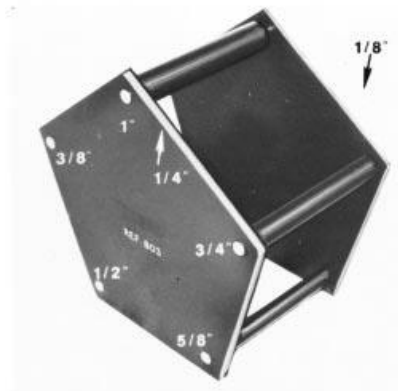


Fig. 11 Cylindrical mandrel test apparatus

Data Analysis

Cracking or delamination of coating on pretreated substrate of coating system was reported.

5.2.4 Water Resistance: Tape Adhesion

Test Description

This test method, as described in Table 10, outlines a procedure for evaluating the intercoat and surface adhesion of an organic coating that has been immersed in water by applying pressure-sensitive tape over a scribed area of coating. The test also measures the coating's ability to resist penetration by water. This test was performed in accordance with Method 6301 of *Paint, Varnish, Lacquer and Related Materials: Methods of Inspection Sampling and Testing* (FED-STD-141 1986) and was rated according to ASTM D3359 (2009). Per MIL-DTL-53022E (2012) and

MIL-DTL-53030C (2012), a film of primer, tested after a 168-h immersion in DI water at room temperature, shall show no wrinkling or blistering immediately after removal of the panel from the water. The primer shall be no more than slightly affected when examined 2 h after removal. After 24 h air drying, the portion of the panel that was immersed shall be the same with regard to hardness, adhesion, color, and gloss as compared to the portion that was not immersed.

Each test panel was also subjected to a crosshatch tape test. A crosshatch tool making a grid pattern that consists of 11 cuts 1 mm apart was used. All cuts went through the coating and into the substrate. Immediately after crosshatching, a piece of tape was placed over the incision parallel to the bottom edge of the panel, ensuring that the tape completely covered the grid formed by the crosshatch. The tape was rapidly pulled at approximately a 180° angle. The incision area was examined for peel away and rated using ASTM D3359.

Rationale

This test was identified as a performance requirement for the baseline pretreatment specifications TT-C-490 and MIL-DTL-5541 when applying specified primers. Loss of paint adhesion is the primary failure mode on ferrous and aluminum substrates.

Table 10 Water resistance: tape adhesion after water immersion

Parameters	7-day cured primed panels; 24-h DI water immersion at room temperature.
Experimental control specimens	Zinc phosphate pretreatment according to TT-C-490 for ferrous panels and conversion coating conforming to MIL-DTL-5541 for aluminum panels.
Acceptance criteria	No peel away; at least 4A per ASTM D 3359; no blistering of unscribed coating area.

Data Analysis

All information, including any delamination or blistering in areas away from the scribes, was reported per the latest version of ASTM D3359 Method A.

5.2.5 Water Immersion: Pencil Hardness after Immersion

Test Description

This test method, as described in Table 11, covers a procedure for rapid, inexpensive determination of the film hardness of an organic coating on a substrate

in terms of drawing leads or pencil leads of known hardness. This test is performed and rated in accordance to ASTM D3363 (2011) and MIL-DTL-53022E (2012).

Table 11 Pencil hardness after water immersion test methodology

Parameters	The coated metal substrate is allowed to cure/react for 7 days; the panel is then immersed 50% for 168 h in DI water at room temperature.
Number and type of specimens per candidate alternative	Primed-only panels
Trials per specimen	1
Experimental control specimens	Zinc phosphate pretreatment according to TT-C-490 for ferrous panels and conversion coating conforming to MIL-DTL-5541 for aluminum panels.
Acceptance criteria	<2 Pencil hardness difference from an unexposed film; no blistering of unscribed coating area.

A coated panel consisting of pretreatment and epoxy primer is placed on a firm horizontal surface. The pencil is held firmly against the film at a 45° angle (pointed away from the operator) and pushed away from the operator in a 6.5-mm (1/4-inch) stroke. The process starts with the hardest pencil and continues down the scale of hardness to either of two end points: the pencil that will not cut into or gouge the film (pencil hardness) or the pencil that will not scratch the film (scratch hardness).

Per MIL-DTL-53022E Section 3.5.7, a film of primer, immersed in distilled water per ASTM D1308 for 168 h or 7 days, shall show no wrinkling or blistering immediately after removal from the water (MIL-DTL-53022E 2012). The primer shall be no more than slightly affected when examined 2 h after removal. After 24 h air drying, the portion of the panel that was immersed shall be the same with regard to hardness, adhesion, color, and gloss as compared to the portion that was not immersed. Film softening shall not exceed a two-pencil hardness difference (ASTM D3363) from an unexposed film with identical cure history prior to water exposure.

Rationale

This test was identified as a performance requirement for designated epoxy primers when applied over the required conversion pretreatment coatings. Loss of paint adhesion is the primary failure mode on aluminum and steel.

Equipment and Instrumentation

- Set of calibrated drawing leads meeting the following scale of hardness:

6B-5B-4B-3B-2B-B-HB-F-H-2H-3H-4H-5H-6H

Softer

Harder

- Mechanical lead holder (drawing leads only)
- Mechanical sharpener, draftsman-type (wood pencils only)
- Abrasive paper, grit No. 400
- Distilled water and container

Data Analysis

The results were rated according to ASTM D3363 (*Standard Test Method for Film Hardness by Pencil Test*). Included with the results were instances of discoloration, loss of gloss, delamination, wrinkling, and blistering.

5.2.6 Pencil Hardness after JP-8 Immersion

Test Description

This test method, as described in Table 12, covers a procedure for rapid, inexpensive determination of the film hardness of an organic coating on a substrate in terms of drawing leads or pencil leads of known hardness. This test is performed and rated in accordance to ASTM D3363 and MIL-DTL-53022.

A coated panel consisting of pretreatment and epoxy primer is placed on a firm horizontal surface. The pencil is held firmly against the film at a 45° angle (pointed away from the operator) and pushed away from the operator in a 6.5-mm (1/4-inch) stroke. The process starts with the hardest pencil and continues down the scale of hardness to either of two end points: the pencil that will not cut into or gouge the film (pencil hardness) or the pencil that will not scratch the film (scratch hardness).

A film of primer, partially immersed in JP-8 for 168 h or 7 days, shall show no blistering or wrinkling and no more than a slight yellow to beige color change on the submerged area of the panel. Upon removal from the fluid, slight softening is acceptable. After 2 h air drying, the panel that was immersed shall be almost indistinguishable with regard to hardness, adhesion, color, and gloss from a panel prepared at the same time but not immersed. Film softening shall not exceed a two-pencil hardness difference (ASTM D3363) from an unexposed film with identical cure history prior to hydrocarbon fluid exposure.

Rationale

This test was identified as a performance requirement for designated epoxy primers when applied over the required conversion pretreatment coatings. Loss of paint adhesion is the primary failure mode on aluminum and steel. The ability of a coating to maintain its properties after exposure to fuel is an important property for effectiveness in the field.

Table 12 Pencil hardness after JP-8 immersion test methodology

Parameters	The coated metal substrate is allowed to cure/react for 7 days; 50% of the panel is then immersed for 168 h in JP8 at room temperature.
Experimental control specimens	Zinc phosphate pretreatment according to TT-C-490 for ferrous panels and conversion coating conforming to MIL-DTL-5541 for aluminum panels.
Acceptance criteria	<2 Pencil hardness difference from an unexposed film no blistering of unscribed coating area.

Equipment and Instrumentation

- Set of calibrated drawing leads meeting the following scale of hardness:

6B-5B-4B-3B-2B-B-HB-F-H-2H-3H-4H-5H-6H

Softer

Harder

- Mechanical lead holder (drawing leads only)
- Mechanical sharpener, draftsman-type (wood pencils only)
- Abrasive paper, grit No. 400
- JP-8 and container

Data Analysis

The results were rated according to ASTM D3363 (*Standard Test Method for Film Hardness by Pencil Test*). Included with the results were instances of discoloration, loss of gloss, delamination, wrinkling, and blistering.

5.2.7 Hydrogen Embrittlement

Test Description

This test method provides a means of detecting possible hydrogen embrittlement of steel parts during manufacture by verifying strict controls, such as surface preparation, pretreatments, and plating/coating, during production operations. In this case, the test for chemical conversion coatings and pretreatments is intended to predict the effect of a coating or pretreatment on the ballistic properties of armor steel substrates. The method used, as described in Table 13, was ASTM F519-13 (*Mechanical Hydrogen Embrittlement Evaluation of Plating/Coating Processes and Service Environments*). Testing is carried out using a notched rod of steel that has been heat treated to a specified hardness. After treating/painting, the rod is placed under a sustained load of 75% of its notch tensile strength for 200 h and the time to fracture and the amount of elongation is determined. If no fracturing occurs after 200 h, the coating is considered nonembrittling.

For this project, hydrogen embrittlement samples were provided and testing was carried out by Assured Testing Services of Ridgway, Pennsylvania.

Table 13 Hydrogen embrittlement ASTM F519-13

Parameters	Notched round tension metal bars of Type 1a.1 made from AISI E4340 steel with a known notch tensile strength.
Experimental control specimens	Samples of notched tension bars, treated with X-Bond 4000 zirconium treatment and a set of bars treated with zirconium and primed with MIL-DTL-53022 primer.
Acceptance criteria	No rupture or cracking of the bars after 200 h under a continuous load of 75% of tensile strength.

5.3 Field Testing and Exposure

5.3.1 Marine Environment Outdoor Exposure

Test Description

This test method, as described in Table 14, evaluates a coating system's ability to prevent substrate corrosion under environmental conditions. Outdoor exposure testing of panels was conducted at NASA's Kennedy Space Center, while parts were tested at CCAFS corrosion test site per Federal Specification TT-C-490. The CCAFS site is located 100 yards from and faces the Atlantic Ocean. Test coupons were installed on Trex racks using plastic insulator stand-offs with stainless steel

fasteners. The rack angle of the coupons was 30° from horizontal. Figure 12 shows an example of the type of exposure racks that were used. The Kennedy Space Center site is located 100 ft from the water's edge, and is reported to be four to five times more corrosive than the CCAFS site.

Table 14 Test methodology for outdoor exposure

Parameters	• Test coupons at a 30° angle. • Temperature of exposed coupons varies with outdoor conditions. • 24 months (interim ratings at 6, 12, and 18 months).
Experimental control specimens	Zinc phosphate pretreatment according to TT-C-490 for ferrous panels and conversion coating conforming to MIL-DTL-5541 for aluminum panels, primed with MIL-DTL-53022 and topcoated with MIL-DTL-64159.
Acceptance criteria	Equal to or better than experimental control specimens, but having a minimum rating as described in Section 3.1 for acceptance criteria.



Fig. 12 CCAFS exposure rack

The decision to split the panel and part exposures between the two sites was based on the fact that previous data generated under the SERDP project was from the

Kennedy Space Center, but exposure at CCAFS is required for product qualification.

At least five specimens of each variable were exposed. They consisted of manufactured parts that simulate current production material and manufacturing processes, parts incorporating the candidate zirconium oxide pretreatment, and five specimens incorporating the current corrosion protection system.

An “X” incision was cut through the coating to the substrate so that the smaller angle of the “X” was 30° to 45°, as described in ASTM D3359. The scribe crossed the entire length of the panel but ended at 1 inch from the edges. The edges and back of the panel were coated with the same coating system as the one being tested. Test samples were stamped or engraved with an identifying marking under the coating.

Coupons for surface corrosion (blisters in field) and creep from scribe, as per ASTM D1654, were evaluated at six-month intervals for two years. This report contains data taken after one year, but panels and parts will remain on exposure for up to at least two years. At each inspection interval the coupons were examined using a measuring magnifier device. Corrosive salts or oxides from the scribes running down the surface of the coupon were ignored in these measurements. The inspections were focused on perforation of the film not staining. Photos were taken of test coupons at each inspection interval. Photos were not available to PPG at the time of the report. Upon completion of testing, coupons were transported to ARL for scanning. After being scanned, each panel was scraped as described in TT-C-490, rerated using ASTM D1654, and scanned a final time.

Rationale

The 24-month outdoor exposure of scribed, painted substrates is a key accelerated test to determine overall corrosion inhibition and coating adhesion as compared to controls.

Equipment and Instrumentation

- **Standard Racks.** See Section 5 of ASTM G50 (2010).
- **Scribe Tool.** Carbide tip scribe as per ASTM D1654
- **Straightedge.** Any straightedge of sufficient length to guide the scribing tool in a straight line.

Data Analysis

The final corrosion test results and six-month visual observations were rated, according to ASTM D1654.

5.3.2 Field Exposure, On-Vehicle (ASTM D1654)

Test Description

The test method parameters described in Table 15 are a basic procedure for conducting on-vehicle testing of candidates. This was performed by selective replacement or refinishing of an appropriate representative substrate/component on a vehicle incorporating the candidate and by the use of test specimens incorporating the candidate attached to the military ground vehicle. This method describes a procedure for monitoring a coating system's performance when applied to specific target areas of an operational platform as compared to the baseline system. The ability to maintain adhesion to the substrate after exposure to field conditions and to prevent corrosion were analyzed.

The process was conducted to replace or refinish a part on the vehicle in accordance with the suggested finishing parameters and the controls established by the CARC applications specification MIL-DTL-53072 (2013). Test panels were prepared during the demonstrations at LEAD and MDMC-PPA in accordance with above ASTM G50 for static field testing and evaluated using ASTM D1654. The components chosen for on-vehicle field exposure were CRS D-ring lift hook surrounds, used on the hoods of HMMWVs, and aluminum battery hold-down brackets, installed under the passenger seats of HMMWVs. Other samples of these components were exposed at CCAFS. In addition to the parts, panels prepared using both the zirconium pretreatment and the presently used production treatments were installed on HMMWVs and MTVRs using racks prepared for the demo. The rack/panels were installed on the rear frames of the vehicles. Each vehicle had both the test pretreatment as well as the control treatments. The parts and panels were evaluated at one year and again at two years. After 12 months' exposure, sets of the test panels and parts were removed from the vehicles and returned to PPG for evaluation. The aluminum hold-down brackets were not available for the 12-month evaluation, so those results are not included in the 12-month report. The test vehicles remained on-site at Camp Lejeune for approximately 18 months, at which time they were deployed onto ocean-going vessels for maneuvers. During this time, the vehicles were periodically washed with sea water. *Standard Method for Evaluating the Degree of Rusting on Painted Steel Surfaces* (SSPC-VIS-2 2008), was used for evaluating component substrates and control samples. The success criteria for field testing was performance greater than or equal to the base vehicle (baseline) or control sample.

Table 15 Test methodology for in-service exposure

Parameters	Ferrous and/or mixed substrate components.
Number and type of specimens per candidate alternative	Number of platforms required is determined by cognizant authority.
Trials per specimen	1
Experimental control specimens	GSE components coated with standard coating system.
Acceptance criteria	≥ 2 years operational service or other user-defined interval, with a minimum of comparable performance to similar platform coated with standard system.

Rationale

Pretreatment coatings must perform successfully in the field before they are transitioned to the end users via revisions of technical manuals, military specifications, and qualified product databases.

Equipment and Instrumentation

- **Military Ground Vehicle.** A vehicle used for standard deployment.

Data Analysis

After a predetermined exposure time agreed upon by the stakeholders, the affected vehicles/parts were evaluated for coating adhesion, color, and corrosion resistance in accordance with SSPC-VIS-2 (2008), MIL-DTL-53072 (2013), and ASTM D1654.

6. Performance Assessment

6.1 General Performance Assessment

Scribe creep analysis was carried out using ASTM D1654. Details of the measurement process are found in Appendix B.

Results from the demonstration testing showed that X-Bond 4000 met or exceeded the performance requirements of TT-C-490.

In accelerated laboratory corrosion testing on steel substrates, X-Bond exhibited corrosion performance ranging from slightly worse than to equal to zinc phosphate. X-Bond showed improved corrosion performance versus blasting-only (direct to metal application), which is the current commercial practice on HHA steel, since

zinc phosphate would cause embrittlement. On aluminum substrates, X-Bond met the performance requirements of MIL-DTL-5541 for conversion coatings, and it performed better than chrome 3+ in laboratory testing. Its performance was equal to that of chrome 6+. The same results were observed on outdoor exposure panels at the CCAFS corrosion test site and at Camp Lejeune.

6.2 Primer-Only Testing

Primer-only testing was included in the demonstrations because the primer specifications must be met to satisfy the requirements of TT-C-490 and MIL-DTL-5541. As previously mentioned in Section 4.4, some of the panels intended for primer-only testing at MDMC-PPA were inadvertently topcoated, so there were no primer-only panels. All of the primer-only panels planned for LEAD were produced.

6.2.1 Adhesion Testing ASTM D3359

In order for a coating system to provide its protective and decorative properties, it must adhere to the targeted surface. This test provides a semi-quantitative evaluation of adhesion. Poor initial dry adhesion can be an early indicator of inadequate performance of a coating system in providing corrosion protection. ASTM D3359 Method B calls for cutting a grid pattern with a knife through the coatings to the metal and using a standard adhesive tape to pull any loosened layers from the substrate. Numerical ratings are given based on a comparison to visual standards. The specification calls for an adhesion rating of 4B or higher on the D3359 scale. CRS samples coated with zinc-rich/MIL-DTL-53030 primer on a zinc phosphate pretreatment, prepared at MDMC-PPA, failed the adhesion test (S15). Additionally, the sample of HHA with MIL-DTL-53022 primer on a zinc phosphate pretreatment failed the adhesion test (H10). All samples treated with zirconium pretreatment passed the dry adhesion test. All primer-only samples met the performance requirements of no initial adhesion loss between layers or to the metal substrate. The zinc-rich primer over grit-blasted HHA steel showed a loss of adhesion, with a rating of 3B. The same primer over grit-blasted and zirconium-treated HHA passed with a 4B rating. Results are shown in Table 16.

Table 16 Adhesion testing of primer-only demonstration panels from LEAD

Code	Substrate	Treatment	Primer	Location	Adhesion rating	Status
C2	CRS	Blasted-only	MIL-DTL-53022	LEAD	4B	Pass
C8	CRS	Blasted+ Zinc Phosphate	MIL-DTL-53022	LEAD	4B	Pass
C5	CRS	Blasted+ X-Bond	MIL-DTL-53022	LEAD	4B	Pass
S3	CRS	Blasted-only	Zinc-Rich+ MIL-DTL-53030	MDMC-PPA	4B	Pass
S15	CRS	Blasted+ Zinc Phosphate	Zinc-Rich+ MIL-DTL-53030	MDMC-PPA	3B	Fail
S8	CRS	Blasted+ X-Bond	Zinc-Rich+ MIL-DTL-53030	MDMC-PPA	4B	Pass
A5	Aluminum	Chrome 6+	MIL-DTL-53022	LEAD	5B	Pass
A2	Aluminum	X-Bond	MIL-DTL-53022	LEAD	4B	Pass
H2	HHA	Blasted-only	MIL-DTL-53022	LEAD	4B	Pass
H10	HHA	Blasted+ Zinc Phosphate	MIL-DTL-53022	LEAD	3B	Fail
H6	HHA	Blasted+ X-Bond	MIL-DTL-53022	LEAD	4B	Pass

Flexibility was assessed with a mandrel bend test, in which painted substrates were wrapped around a cylindrical mandrel, 0.25 inch in diameter, and evaluated for cracking and adhesion loss along the bend. According to ASTM D522, the sample must show no cracking or flaking. All samples passed except for CRS samples with the zinc-rich/MIL-DTL-53030 primer combination on zinc phosphate pretreatment (S15). HHA was not tested. The results for the flexibility testing are shown in Table 17.

Table 17 Flexibility testing of primer-only demonstration panels from LEAD and MDMC-PPA

Code	Substrate	Treatment	Primer	Location	Flexibility	Status
C8	CRS	Blasted+ Zinc Phosphate	MIL-DTL-53022	LEAD	No cracking	Pass
C5	CRS	Blasted+ X-Bond	MIL-DTL-53022	LEAD	No cracking	Pass
C2	CRS	Blasted-only	MIL-DTL-53022	LEAD	No cracking	Pass
S3	CRS	Blasted-only	Zinc-Rich+ MIL-DTL-53030	MDMC-PPA	No cracking	Pass
S8	CRS	Blasted+ X-Bond	Zinc-Rich+ MIL-DTL-53030	MDMC-PPA	No cracking	Pass
S15	CRS	Blasted+ Zinc Phosphate	Zinc-Rich+ MIL-DTL-53030	MDMC-PPA	Slight cracking	Fail
A5	Aluminum	Chrome 6+	MIL-DTL-53022	LEAD	No cracking	Pass
A2	Aluminum	X-Bond	MIL-DTL-53022	LEAD	No cracking	Pass

6.2.2 Water Resistance: Adhesion after Water Immersion

In this test, panels were soaked in water for 7 days at room temperature, after which they were removed and evaluated for wrinkling, blistering, and discoloration. There should be no wrinkling, blistering, or change in gloss or color observed immediately after removal of the panel from the water. Tape adhesion per ASTM D3359 was also evaluated. All samples passed these tests. The zinc-rich/MIL-DTL-53030 primer combination on CRS with zinc phosphate pretreatment (Sample S15) and the MIL-DTL-53022 on HHA treated with zinc phosphate (Sample H10) showed no drop-off of adhesion, although the adhesion was below specifications initially. Results are in Table 18.

Table 18 Water resistance: pencil hardness after water immersion testing of primer-only demonstration panels with MIL-DTL-53022 from LEAD and MDMC-PPA

Code	Substrate	Treatment	Primer	Location	Adhesion rating/softening	Status
C8	CRS	Blasted+ Zinc Phosphate	MIL-DTL-53022	LEAD	4B/None	Pass
C5	CRS	Blasted+ X-Bond	MIL-DTL-53022	LEAD	4B/None	Pass
C2	CRS	Blasted-only	MIL-DTL-53022	LEAD	4B/None	Pass
S3	CRS	Blasted-only	Zinc-Rich+ MIL-DTL-53030	MDMC-PPA	4B/None	Pass
S8	CRS	Blasted+ X-Bond	Zinc-Rich+ MIL-DTL-53030	MDMC-PPA	4B/None	Pass
S15	CRS	Blasted+ Zinc Phosphate	Zinc-Rich+ MIL-DTL-53030	MDMC-PPA	4B/None	Pass
A5	Aluminum	Chrome 6+	MIL-DTL-53022	LEAD	5B/None	Pass
A2	Aluminum	X-Bond	MIL-DTL-53022	LEAD	4B/None	Pass
H2	HHA	Blasted-only	MIL-DTL-53022	LEAD	4B/None	Pass
H6	HHA	Blasted+ X-Bond	MIL-DTL-53022	LEAD	4B/None	Pass
H10	HHA	Blasted+ Zinc Phosphate	MIL-DTL-53022	LEAD	4B/None	Pass

6.2.3 Water Resistance: Pencil Hardness after Water Immersion

In this test, panels were soaked in water for 7 days at room temperature, after which they were removed and evaluated for wrinkling, blistering, and discoloration. There should be no wrinkling, blistering, change in gloss or color observed immediately after removal of the panel from the water. No more than a two-pencil softening is permitted. All samples passed the testing. Results are in Table 18.

6.2.4 Hydrocarbon Resistance: Pencil Hardness after JP8 Immersion

This test measures the resistance of the paint system to hydrocarbons, such as JP8. Panels were immersed in JP8 for one week, after which the pencil hardness was measured according to ASTM D3363. Softening by the JP8 indicates that the fuel is being absorbed into the coating and suggests poor adhesion and corrosion performance. Again, the only failures noted were on panels with the zinc phosphate pretreatment (S15 and H10). The results are shown in Table 19.

Table 19 Hydrocarbon resistance: pencil hardness after JP8 immersion testing of primer-only demonstration panels from LEAD and MDMC-PPA

Code	Substrate	Treatment	Primer	Location	Adhesion rating/softening	Status
C8	CRS	Blasted+ Zinc Phosphate	MIL-DTL-53022	LEAD	4B/None	Pass
C5	CRS	Blasted+ X-Bond	MIL-DTL-53022	LEAD	4B/None	Pass
C2	CRS	Blasted-only	MIL-DTL-53022	LEAD	4B/None	Pass
S3	CRS	Blasted-only	Zinc-Rich+ MIL-DTL-53030	MDMC-PPA	4B/None	Pass
S8	CRS	Blasted+ X-Bond	Zinc-Rich+ MIL-DTL-53030	MDMC-PPA	4B/None	Pass
S15	CRS	Blasted+ Zinc Phosphate	Zinc-Rich+ MIL-DTL-53030	MDMC-PPA	3B/None	Fail
A5	Aluminum	Chrome 6+	MIL-DTL-53022	LEAD	4B/None	Pass
A2	Aluminum	X-Bond	MIL-DTL-53022	LEAD	4B/None	Pass
H2	HHA	Blasted-only	MIL-DTL-53022	LEAD	4B/None	Pass
H6	HHA	Blasted+ X-Bond	MIL-DTL-53022	LEAD	4B/None	Pass
H10	HHA	Blasted+ Zinc Phosphate	MIL-DTL-53022	LEAD	3B/None	Fail

6.2.5 Hydrogen Embrittlement of Steel-Treated-Only and Primed-Only after X-Bond Treatment

For the demonstration, notched bars were obtained from Assured Testing Services of Ridgway, Pennsylvania. The notched bars were treated and finished in the same manner as the panels and parts at LEAD. Three bars were treated only with zirconium, and three bars were finished with zirconium treatment MIL-DTL-53022 primer. The notched bars were returned to Assured Testing Services for ASTM F519 hydrogen embrittlement testing. Assured Testing Services reported that all samples passed the hydrogen embrittlement testing. Therefore, the X-Bond 4000 treatment process does not cause hydrogen embrittlement. A copy of the test report from Assured Testing Services is found in Appendix C.

6.2.6 Neutral Salt Spray Corrosion Test ASTM B117

In this test and as shown in Figs. 13 and 14, the zirconium-treated panels showed more red rust along the scribes than zinc phosphate on CRS and HHA. Corrosion ratings for zirconium met the minimum rating of six. On aluminum panels, the performance of X-Bond was equal to that of chrome 6+ and met the minimum

performance requirement of a scribe creep rating greater than or equal to eight. Primer-only panels of X-Bond versus chrome 3+ were not available due to the designated panels being inadvertently top-coated at MDMC-PPA. Results from salt spray testing are shown in Tables 20 and 21. The results on aluminum substrates are shown in Table 22 and Figure 15.

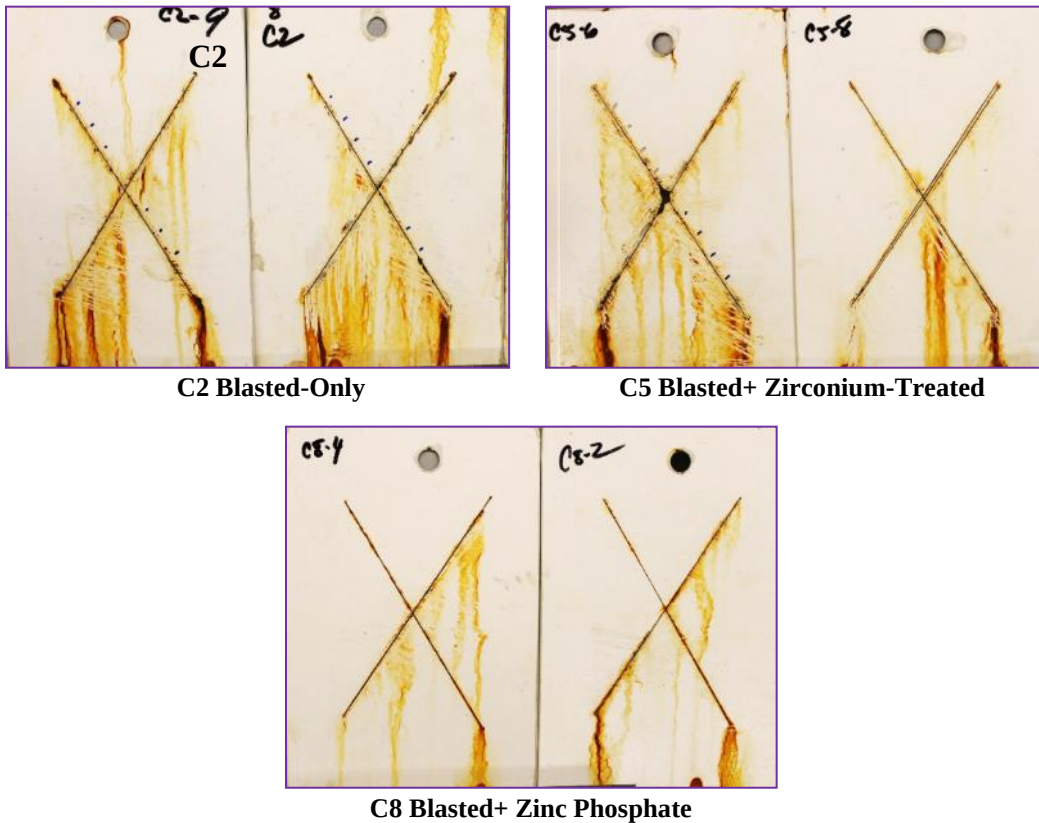


Fig. 13 ASTM B117 salt spray corrosion of CRS treated with zirconium vs. blasted-only versus zinc phosphate under MIL-DTL-53022 primer at LEAD

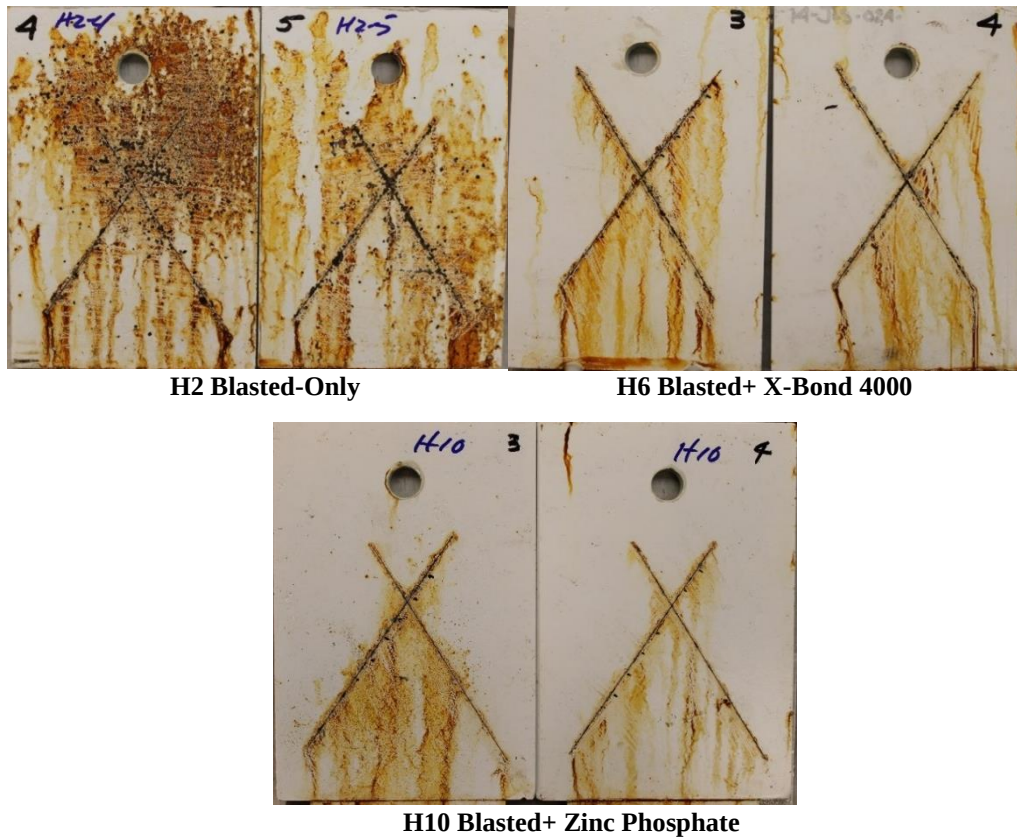


Fig. 14 336-h neutral salt spray testing of HHA steel demonstration panels blasted-only vs. blasted+ X-Bond 4000 vs. blasted+ zinc phosphate under MIL-DTL-53022 primer at LEAD

Table 20 ASTM B117 neutral salt spray testing of CRS demonstration panels with MIL-DTL-53022 primer-only from LEAD

Code	Substrate	Treatment	Primer	Location	Duration	Scribe creep	Status
C2	CRS	Blasted-only	MIL-DTL-53022	LEAD	336 h	6	Pass
C5	CRS	Blasted+ X-Bond	MIL-DTL-53022	LEAD	336 h	7	Pass
C8	CRS	Blasted+ Zinc Phosphate	MIL-DTL-53022	LEAD	336 h	8	Pass

Table 21 ASTM B117 neutral salt spray testing of HHA steel demonstration panels from LEAD

Code	Substrate	Treatment	Primer	Location	Duration	Scribe creep	Status
H2	HHA	Blasted-only	MIL-DTL-53022	LEAD	336 h	3	Fail
H6	HHA	Blasted+ X-Bond	MIL-DTL-53022	LEAD	336 h	7	Pass
H10	HHA	Blasted+ Zinc Phosphate	MIL-DTL-53022	LEAD	336 h	9	Pass

Table 22 ASTM B117 neutral salt spray testing of aluminum demonstration panels chrome 6+ vs. X-Bond 4000 under MIL-DTL-53022 primer at LEAD

Code	Substrate	Treatment	Primer	Location	Duration	Scribe creep	Status
A2	Aluminum	X-Bond 4000	MIL-DTL-53022	LEAD	1000 h	9	Pass
A5	Aluminum	Chrome 6+	MIL-DTL-53022	LEAD	1000 h	9	Pass

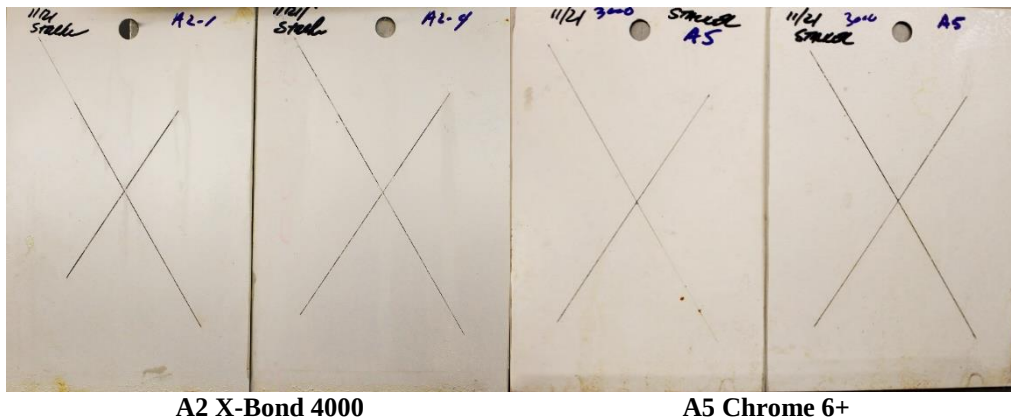


Fig. 15 ASTM B117 neutral salt spray testing of aluminum demonstration panels chrome 6+ vs. X-Bond 4000 under MIL-DTL-53022 primer and MIL-DTL-53039 topcoat at LEAD

6.2.7 Cyclic Corrosion Test GMW 14872

The performance requirement is a minimum scribe creep rating of six on CRS per TT-C-490 and eight on aluminum as determined by ASTM D1654. TT-C-490 calls for 30 cycles of GMW 14872 to be run for all MIL-DTL-53022 primers. However, MIL-DTL-53022 specification has no cyclic corrosion performance requirement

for Type II products, which are used at both LEAD and MDMC-PPA. Therefore, 20 cycles were run after which comparison of the pretreatments was made to establish relative performance.

The results in Tables 23 and 24 show that X-Bond performed better than blasted-only CRS and HHA. Although the performance of zirconium was slightly worse than zinc phosphate, it still passed 20 cycles of testing. Figures 16 and 17 show the comparisons on the different substrates and pretreatments.

Table 23 Cyclic corrosion testing of MIL-DTL-53022 Type II primer-only CRS demonstration panels from LEAD

Code	Substrate	Treatment	Primer	Location	Duration	Scribe creep	Status
C2	CRS	Blasted-only	MIL-DTL-53022	LEAD	20 cycles	5	Pass
C5	CRS	Blasted+ X-Bond	MIL-DTL-53022	LEAD	20 cycles	6	Pass
C8	CRS	Blasted+ Zinc Phosphate	MIL-DTL-53022	LEAD	20 cycles	7	Pass

Table 24 GMW 14872 cyclic corrosion testing of HHA panels blasted-only vs. X-Bond 4000 vs. zinc phosphate demonstration panels under MIL-DTL-53022 primer at LEAD

Code	Substrate	Treatment	Primer	Location	Duration	Scribe creep	Status
H2	HHA	None	MIL-DTL-53022	LEAD	20 cycles	3	N/A
H6	HHA	Blasted+ X-Bond 4000	MIL-DTL-53022	LEAD	20 cycles	5	Fail
H10	HHA	Blasted+ Zinc Phosphate	MIL-DTL-53022	LEAD	20 cycles	7	Pass

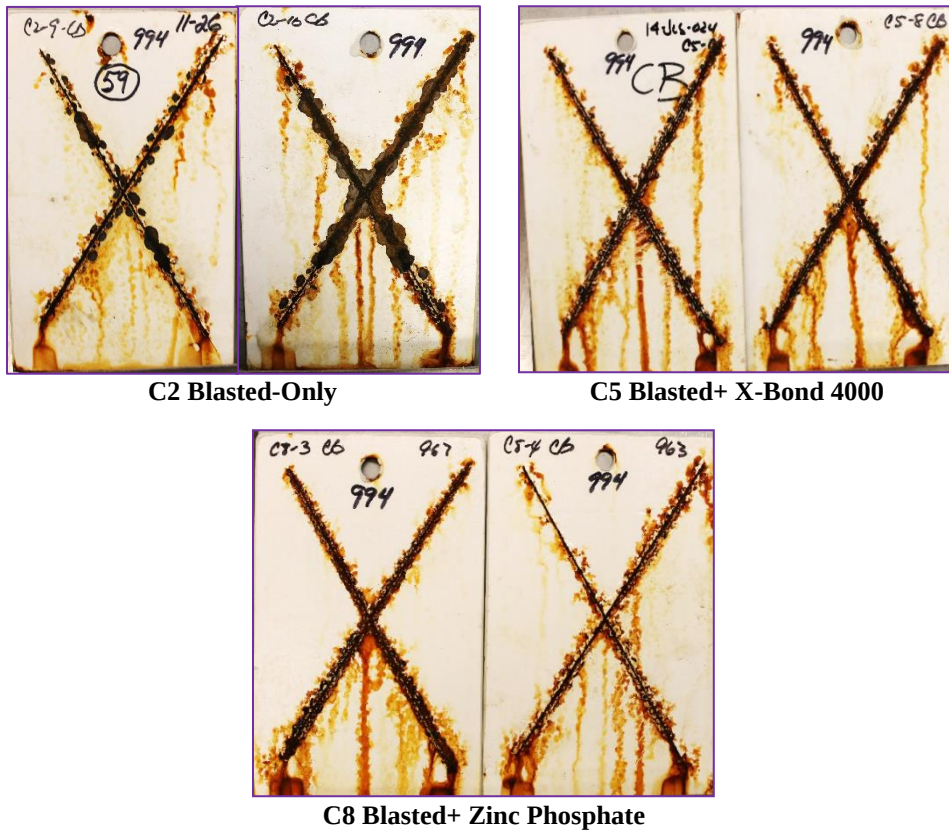


Fig. 16 GMW 14872 testing of CRS demonstration panels blasted-only vu.s blasted+ X-Bond 4000 vs. blasted+ zinc phosphate under MIL-DTL-53022 primer at LEAD

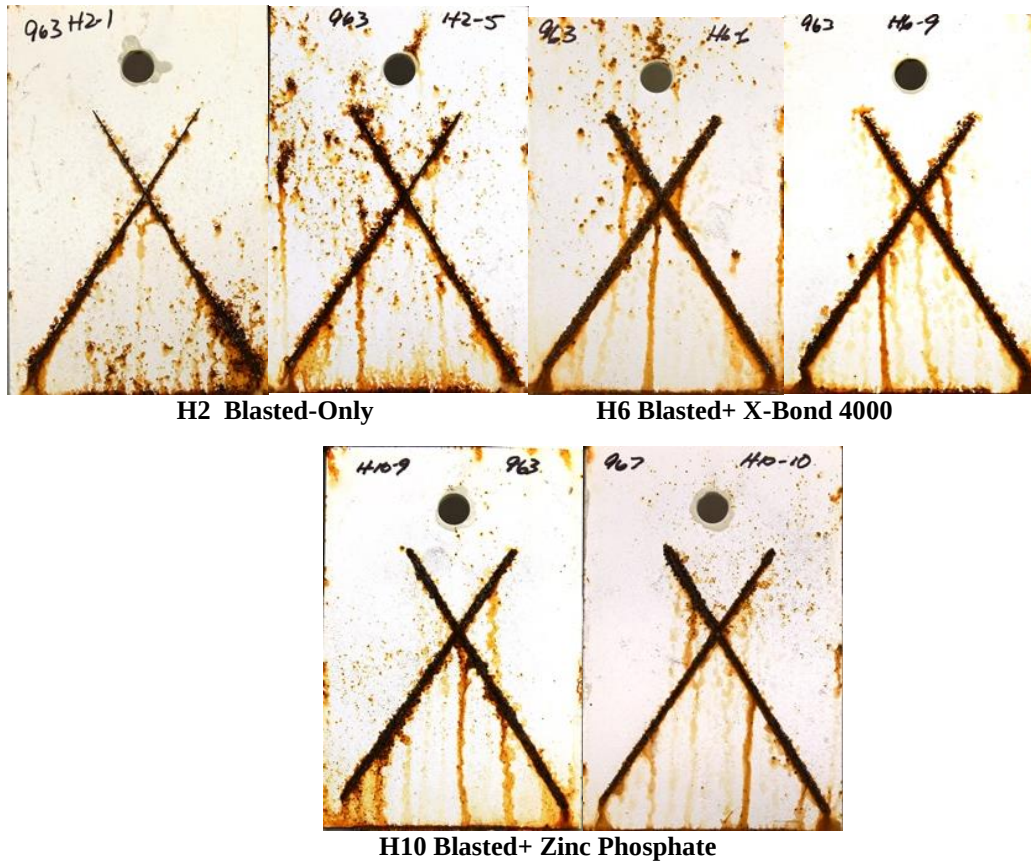


Fig. 17 GMW 14872 cyclic corrosion testing of HHA panels blasted-only vs. blasted+ X-Bond 4000 vs. blasted zinc phosphate demonstration panels under MIL-DTL-53022 primer at LEAD

6.3 Performance of Fully Finished Panels

6.3.1 Adhesion Testing of Fully Finished Demonstration Panels from LEAD and MDMC-PPA

In order for a coating system to perform its protective and decorative functions, it must adhere to the targeted surface. ASTM D3359 Method B calls for cutting a grid pattern with a knife through the coatings to the metal and using a standard adhesive tape to pull any loosened layers from the substrate. Numerical ratings are given based on a comparison to visual standards. The requirement per TT-C-490 is a rating of 4B or better (5B = 0% loss and 0B = >65% loss) and per MIL-DTL-5541 is a 4A or better (trace peeling or removal along incisions or at intersections). Results show X-Bond 4000 to pass adhesion testing under all of the primer substrate combinations. Blasted-only HHA that was coated with zinc-rich primer and MIL-DTL-53022 (Sample H4) failed, as did HHA with zinc phosphate and MIL-DTL-53022 (H11). CRS treated with zinc phosphate and coated with zinc-rich primer and MIL-DTL-53030 (S17) also failed the adhesion test. The zirconium-treated sample under the zinc-rich and MIL-DTL-53030 primers showed no adhesion loss. The results of the adhesion testing are shown in Table 25.

Table 25 ASTM D3359 adhesion testing of fully finished demonstration panels from LEAD and MDMC-PPA

Code	Substrate	Treatment	Primer	Topcoat	Location	Adhesion rating	Status
C3	CRS	Blasted-only	MIL-DTL-53022	MIL-DTL-53039	LEAD	4B	Pass
		Blasted+					
C6	CRS	X-Bond	MIL-DTL-53022	MIL-DTL-53039	LEAD	4B	Pass
		Blasted+					
C9	CRS	Zinc Phosphate	MIL-DTL-53022	MIL-DTL-53039	LEAD	4B	Pass
S4	CRS	Blasted-only	MIL-DTL-53030	MIL-DTL-64159	MDMC-PPA	4B	Pass
		Blasted+					
S9	CRS	X-Bond	MIL-DTL-53030	MIL-DTL-64159	MDMC-PPA	4B	Pass
		Blasted+					
S16	CRS	Zinc Phosphate	MIL-DTL-53030	MIL-DTL-64159	MDMC-PPA	4B	Pass
		Blasted+					
S19	CRS	Zinc Phosphate	MIL-DTL-53022	MIL-DTL-53039	MDMC-PPA	4B	Pass
		Blasted+					
S11	CRS	X-Bond	MIL-DTL-53022	MIL-DTL-53039	MDMC-PPA	4B	Pass
			Zinc-Rich+ MIL-				
S5	CRS	Blasted-only	DTL-53030	MIL-DTL-64159	MDMC-PPA	4B	Pass

Table 25 ASTM D3359 adhesion testing of fully finished demonstration panels from LEAD and MDMC-PPA (continued)

Code	Substrate	Treatment	Primer	Topcoat	Location	Adhesion rating	Status
S10	CRS	Blasted+ X-Bond	Zinc-Rich+ MIL-DTL-53030	MIL-DTL-64159	MDMC-PPA	4B	Pass
S17	CRS	Blasted+ Zinc Phosphate	Zinc-Rich+ MIL-DTL-53030	MIL-DTL-64159	MDMC-PPA	3B	Fail
S18	CRS	Blasted+ Zinc Phosphate	MIL-DTL-32348	MIL-DTL-64159	MDMC-PPA	4B	Pass
S12	CRS	Blasted+ X-Bond	MIL-DTL-32348	MIL-DTL-64159	MDMC-PPA	5B	Pass
A6	Aluminum	Chrome 6+	MIL-DTL-53022	MIL-DTL-53039	LEAD	4B	Pass
A3	Aluminum	X-Bond	MIL-DTL-53022	MIL-DTL-53039	LEAD	4B	Pass
M8	Aluminum	Metalast TCP Chrome 3+	MIL-DTL-53030	MIL-DTL-64159	MDMC-PPA	4B	Pass
M3	Aluminum	X-Bond	MIL-DTL-53030	MIL-DTL-64159	MDMC-PPA	4B	Pass
M10	Aluminum	Metalast TCP Chrome 3+	MIL-DTL-53022	MIL-DTL-53039	MDMC-PPA	4B	Pass
M5	Aluminum	Blasted+ X-Bond	MIL-DTL-53022	MIL-DTL-53039	MDMC-PPA	5B	Pass
H3	HHA	Blasted-only	MIL-DTL-53022	MIL-DTL-53039	LEAD	4B	Pass
H7	HHA	Blasted+ X-Bond	MIL-DTL-53022	MIL-DTL-53039	LEAD	4B	Pass
H11	HHA	Blasted+ Zinc Phosphate	MIL-DTL-53022	MIL-DTL-53039	LEAD	3B	Fail
H4	HHA	Blasted-only	Zinc-Rich+ MIL-DTL-53022	MIL-DTL-53039	LEAD	3B	Fail
H8	HHA	Blasted+ X-Bond	Zinc-Rich+ MIL-DTL-53022	MIL-DTL-53039	LEAD	4B	Pass

6.3.2 Flexibility Testing of Fully Finished Demonstration Panels from LEAD and MDMC-PPA

Flexibility was assessed with a mandrel bend test, in which painted substrates were wrapped around a cylindrical mandrel, 0.25 inch in diameter, and evaluated for cracking and adhesion loss along the bend. According to ASTM D522, the sample must show no cracking or flaking. As shown in Table 26, the zirconium-treated samples passed the test, while the CRS under zinc phosphate pretreatment and zinc-rich primer did not.

Table 26 ASTM D522 flexibility testing of fully finished demonstration panels from LEAD and MDMC-PPA

Code	Substrate	Treatment	Primer	Topcoat	Location	Flexibility	Status
C3	CRS	Blasted-only	MIL-DTL-53022	MIL-DTL-53039	LEAD	No cracking/no adhesion loss	Pass
C6	CRS	Blasted+ X-Bond	MIL-DTL-53022	MIL-DTL-53039	LEAD	No cracking/no adhesion loss	Pass
C9	CRS	Blasted+ Zinc Phosphate	MIL-DTL-53022	MIL-DTL-53039	LEAD	No cracking/no adhesion loss	Pass
S4	CRS	Blasted-only	MIL-DTL-53030	MIL-DTL-64159	MDMC-PPA	No cracking/no adhesion loss	Pass
S9	CRS	Blasted+ X-Bond	MIL-DTL-53030	MIL-DTL-64159	MDMC-PPA	No cracking/no adhesion loss	Pass
S16	CRS	Blasted/Zinc Phosphate	MIL-DTL-53030	MIL-DTL-64159	MDMC-PPA	No crack/no adhesion loss	Pass
S19	CRS	Blasted+ Zinc Phosphate	MIL-DTL-53022	MIL-DTL-53039	MDMC-PPA	No cracking/no adhesion loss	Pass
S11	CRS	Blasted+ X-Bond	MIL-DTL-53022	MIL-DTL-53039	MDMC-PPA	No cracking/no adhesion loss	Pass
S5	CRS	Blasted-only	Zinc-Rich+ MIL-DTL-53030	MIL-DTL-64159	MDMC-PPA	No cracking/no adhesion loss	Pass
S10	CRS	Blasted/ X-Bond	Zinc-Rich+ MIL-DTL-53030	MIL-DTL-64159	MDMC-PPA	No cracking/no adhesion loss	Pass
S17	CRS	Blasted/Zinc Phosphate	Zinc-Rich+ MIL-DTL-53030	MIL-DTL-64159	MDMC-PPA	No cracking/no adhesion loss	Fail
S18	CRS	Blasted+ Zinc Phosphate	MIL-DTL-32348	MIL-DTL-64159	MDMC-PPA	No cracking/no adhesion loss	Pass
S12	CRS	Blasted+ X-Bond	MIL-DTL-32348	MIL-DTL-64159	MDMC-PPA	No cracking/no adhesion loss	Pass
A6	Aluminum	Chrome 6+	MIL-DTL-53022	MIL-DTL-53039	LEAD	No cracking/no adhesion loss	Pass
A3	Aluminum	X-Bond	MIL-DTL-53022	MIL-DTL-53039	LEAD	No cracking/no adhesion loss	Pass
M8	Aluminum	Metalast TCP Chrome 3+	MIL-DTL-53030	MIL-DTL-64159	MDMC-PPA	No cracking/no adhesion loss	Pass
M3	Aluminum	X-Bond	MIL-DTL-53030	MIL-DTL-64159	MDMC-PPA	No cracking/no adhesion loss	Pass

Table 26 ASTM D522 flexibility testing of fully finished demonstration panels from LEAD and MDMC-PPA (continued)

Code	Substrate	Treatment	Primer	Topcoat	Location	Flexibility	Status
M10	Aluminum	Metalast TCP Chrome 3+	MIL-DTL-53022	MIL-DTL-53039	MDMC-PPA	No cracking/no adhesion loss	Pass
M5	Aluminum	Blasted+ X- Bond	MIL-DTL-53022	MIL-DTL-53039	MDMC-PPA	No cracking/no adhesion loss	Pass

6.3.3 Water Resistance Testing of Fully Finished Demonstration Panels from LEAD and MDMC-PPA

In this test, panels were soaked in water for 7 days at room temperature, after which they were removed and evaluated for wrinkling, blistering, and discoloration. Tape adhesion per ASTM D3359 was also evaluated. All samples passed this test as shown in Table 27.

Table 27 Water resistance testing of fully finished demonstration panels from LEAD and MDMC-PPA

Code	Substrate	Treatment	Primer	Topcoat	Location	Adhesion	Status
C3	CRS	Blasted-only	MIL-DTL-53022	MIL-DTL-53039	LEAD	OK	Pass
C6	CRS	Blasted+ X-Bond	MIL-DTL-53022	MIL-DTL-53039	LEAD	OK	Pass
C9	CRS	Blasted+ Zinc Phosphate	MIL-DTL-53022	MIL-DTL-53039	LEAD	OK	Pass
S4	CRS	Blasted-only	MIL-DTL-53030	MIL-DTL-64159	MDMC-PPA	OK	Pass
S9	CRS	Blasted+ X-Bond	MIL-DTL-53030	MIL-DTL-64159	MDMC-PPA	OK	Pass
S16	CRS	Blasted+ Zinc Phosphate	MIL-DTL-53030	MIL-DTL-64159	MDMC-PPA	OK	Pass
S19	CRS	Blasted+ Zinc Phosphate	MIL-DTL-53022	MIL-DTL-53039	MDMC-PPA	OK	Pass
S11	CRS	Blasted+ X-Bond	MIL-DTL-53022	MIL-DTL-53039	MDMC-PPA	OK	Pass
S5	CRS	Blasted-only	Zinc-Rich+ MIL- DTL-53030	MIL-DTL-64159	MDMC-PPA	OK	Pass
S10	CRS	Blasted+ X-Bond	Zinc-Rich+ MIL- DTL-53030	MIL-DTL-64159	MDMC-PPA	OK	Pass
S17	CRS	Blasted+ Zinc Phosphate	Zinc-Rich+ MIL- DTL-53030	MIL-DTL-64159	MDMC-PPA	OK	Pass

Table 27 Water resistance testing of fully finished demonstration panels from LEAD and MDMC-PPA (continued)

Code	Substrate	Treatment	Primer	Topcoat	Location	Adhesion	Status
S18	CRS	Blasted+ Zinc Phosphate	MIL-DTL-32348	MIL-DTL-64159	MDMC-PPA	OK	Pass
S12	CRS	Blasted+ X-Bond	MIL-DTL-32348	MIL-DTL-64159	MDMC-PPA	OK	Pass
A6	Aluminum	Chrome 6+	MIL-DTL-53022	MIL-DTL-53039	LEAD	OK	Pass
A3	Aluminum	X-Bond	MIL-DTL-53022	MIL-DTL-53039	LEAD	OK	Pass
M8	Aluminum	Metalast TCP Chrome 3+	MIL-DTL-53030	MIL-DTL-64159	MDMC-PPA	OK	Pass
M3	Aluminum	X-Bond	MIL-DTL-53030	MIL-DTL-64159	MDMC-PPA	OK	Pass
M10	Aluminum	Metalast TCP Chrome 3+	MIL-DTL-53022	MIL-DTL-53039	MDMC-PPA	OK	Pass
M5	Aluminum	Blasted+ X-Bond	MIL-DTL-53022	MIL-DTL-53039	MDMC-PPA	OK	Pass
H3	HHA	Blasted-only	MIL-DTL-53022	MIL-DTL-53039	LEAD	OK	Pass
H7	HHA	Blasted+ X-Bond	MIL-DTL-53022	MIL-DTL-53039	LEAD	OK	Pass
H11	HHA	Blasted+ Zinc Phosphate	MIL-DTL-53022	MIL-DTL-53039	LEAD	OK	Pass
H4	HHA	Blasted-only	Zinc-Rich+ MIL-DTL-53022	MIL-DTL-53039	LEAD	OK	Pass
H8	HHA	Blasted+ X-Bond	Zinc-Rich+ MIL-DTL-53022	MIL-DTL-53039	LEAD	OK	Pass

6.3.4 Water Immersion Test

This test measures the effect of water immersion on the integrity of painted surfaces. Panels were immersed in water for 7 days at room temperature. After removal and drying for 2 h, the paint films were examined for appearance defects. Pencil hardness was measured 24 h after removal. According to the primer and topcoat specifications, a loss of pencil hardness greater than two pencils is considered a failure. Testing was carried out at ARL and was reported verbally. All samples, as reported in Table 28, passed this test.

Table 28 Water immersion testing of fully finished demonstration panels from LEAD and MDMC-PPA

Code	Substrate	Treatment	Primer	Topcoat	Location	Status
C3	CRS	Blasted-only	MIL-DTL-53022	MIL-DTL-53039	LEAD	Pass
		Blasted+				
C6	CRS	X-Bond	MIL-DTL-53022	MIL-DTL-53039	LEAD	Pass
		Blasted+				
C9	CRS	Zinc Phosphate	MIL-DTL-53022	MIL-DTL-53039	LEAD	Pass
S4	CRS	Blasted-only	MIL-DTL-53030	MIL-DTL-64159	MDMC-PPA	Pass
		Blasted+				
S9	CRS	X-Bond	MIL-DTL-53030	MIL-DTL-64159	MDMC-PPA	Pass
		Blasted+				
S16	CRS	Zinc Phosphate	MIL-DTL-53030	MIL-DTL-64159	MDMC-PPA	Pass
		Blasted+				
S19	CRS	Zinc Phosphate	MIL-DTL-53022	MIL-DTL-53039	MDMC-PPA	Pass
		Blasted+				
S11	CRS	X-Bond	MIL-DTL-53022	MIL-DTL-53039	MDMC-PPA	Pass
S5	CRS	Blasted-only	Zinc-Rich+ MIL-DTL-53030	MIL-DTL-64159	MDMC-PPA	Pass
		Blasted+				
S10	CRS	X-Bond	Zinc-Rich+ MIL-DTL-53030	MIL-DTL-64159	MDMC-PPA	Pass
		Blasted+				
S17	CRS	Zinc Phosphate	Zinc-Rich+ MIL-DTL-53030	MIL-DTL-64159	MDMC-PPA	Pass
S18	CRS	Blasted+ Zinc Phosphate	MIL-DTL-32348	MIL-DTL-64159	MDMC-PPA	Pass
		Blasted+				
S12	CRS	X-Bond	MIL-DTL-32348	MIL-DTL-64159	MDMC-PPA	Pass
A6	Aluminum	Chrome 6+	MIL-DTL-53022	MIL-DTL-53039	LEAD	Pass
A3	Aluminum	X-Bond	MIL-DTL-53022	MIL-DTL-53039	LEAD	Pass
M8	Aluminum	Metalast TCP Chrome 3+	MIL-DTL-53030	MIL-DTL-64159	MDMC-PPA	Pass
M3	Aluminum	X-Bond	MIL-DTL-53030	MIL-DTL-64159	MDMC-PPA	Pass
M10	Aluminum	Metalast TCP Chrome 3+	MIL-DTL-53022	MIL-DTL-53039	MDMC-PPA	Pass
M5	Aluminum	Blasted+ X-Bond	MIL-DTL-53022	MIL-DTL-53039	MDMC-PPA	Pass
H3	HHA	Blasted-only	MIL-DTL-53022	MIL-DTL-53039	LEAD	Pass

Table 28 Water immersion testing of fully finished demonstration panels from LEAD and MDMC-PPA (continued)

Code	Substrate	Treatment	Primer	Topcoat	Location	Status
H7	HHA	Blasted+ X-Bond	MIL-DTL-53022	MIL-DTL-53039	LEAD	Pass
H11	HHA	Blasted+ Zinc Phosphate	MIL-DTL-53022	MIL-DTL-53039	LEAD	Pass
H4	HHA	Blasted-only	Zinc-Rich+ MIL-DTL-53022	MIL-DTL-53039	LEAD	Pass
H8	HHA	Blasted+ X-Bond	Zinc-Rich+ MIL-DTL-53022	MIL-DTL-53039	LEAD	Pass

6.3.5 Hydrocarbon Resistance (JP8) Testing of Fully Finished Demonstration Panels from LEAD and MDMC-PPA

As shown in Table 29, all samples passed this test except the zinc-rich/MIL-DTL-53030 primer combination on zinc phosphate-treated CRS (S17), the zinc-rich/MIL-DTL-53022 primer combination on blasted HHA with X-Bond (H4 & H8), and the MIL-DTL-53022 primer on zinc phosphate-treated HHA (H11).

Table 29 Hydrocarbon resistance (JP8) testing of fully finished demonstration panels from LEAD and MDMC-PPA

Code	Substrate	Treatment	Primer	Topcoat	Location	Adhesion/ softening	Status
C3	CRS	Blasted-only	MIL-DTL-53022	MIL-DTL-53039	LEAD	4B/OK	Pass
C6	CRS	Blasted+ X-Bond	MIL-DTL-53022	MIL-DTL-53039	LEAD	4B/OK	Pass
C9	CRS	Blasted+ Zinc Phosphate	MIL-DTL-53022	MIL-DTL-53039	LEAD	4B/OK	Pass
S4	CRS	Blasted-only	MIL-DTL-53030	MIL-DTL-64159	MDMC-PPA	4B/OK	Pass
S9	CRS	Blasted+ X-Bond	MIL-DTL-53030	MIL-DTL-64159	MDMC-PPA	4B/OK	Pass
S16	CRS	Blasted+ Zinc Phosphate	MIL-DTL-53030	MIL-DTL-64159	MDMC-PPA	4B/OK	Pass
S19	CRS	Blasted+ Zinc Phosphate	MIL-DTL-53022	MIL-DTL-53039	MDMC-PPA	4B/OK	Pass
S11	CRS	Blasted+ X-Bond	MIL-DTL-53022	MIL-DTL-53039	MDMC-PPA	4B/OK	Pass

Table 29 Hydrocarbon resistance (JP8) testing of fully finished demonstration panels from LEAD and MDMC-PPA (continued)

Code	Substrate	Treatment	Primer	Topcoat	Location	Adhesion/ softening	Status
S5	CRS	Blasted-only	Zinc-Rich+ MIL- DTL-53030	MIL-DTL-64159	MDMC-PPA	4B/OK	Pass
S10	CRS	Blasted+ X-Bond	Zinc-Rich+ MIL- DTL-53030	MIL-DTL-64159	MDMC-PPA	4B/OK	Pass
S17	CRS	Blasted+ Zinc Phosphate	Zinc-Rich+ MIL- DTL-53030	MIL-DTL-64159	MDMC-PPA	3B/OK	Fail
S18	CRS	Blasted+ Zinc Phosphate	MIL-DTL-32348	MIL-DTL-64159	MDMC-PPA	4B/OK	Pass
S12	CRS	Blasted+ X-Bond	MIL-DTL-32348	MIL-DTL-64159	MDMC-PPA	5B/OK	Pass
A6	Aluminum	Chrome 6+	MIL-DTL-53022	MIL-DTL-53039	LEAD	4B/OK	Pass
A3	Aluminum	X-Bond	MIL-DTL-53022	MIL-DTL-53039	LEAD	4B/OK	Pass
M8	Aluminum	Metalast TCP Chrome 3+	MIL-DTL-53030	MIL-DTL-64159	MDMC-PPA	4B/OK	Pass
M3	Aluminum	X-Bond	MIL-DTL-53030	MIL-DTL-64159	MDMC-PPA	4B/OK	Pass
M10	Aluminum	Metalast TCP Chrome 3+	MIL-DTL-53022	MIL-DTL-53039	MDMC-PPA	4B/OK	Pass
M5	Aluminum	Blasted+ X- Bond	MIL-DTL-53022	MIL-DTL-53039	MDMC-PPA	5B/OK	Pass
H3	HHA	Blasted-only	MIL-DTL-53022	MIL-DTL-53039	LEAD	4B/OK	Pass
H7	HHA	Blasted+ X- Bond	MIL-DTL-53022	MIL-DTL-53039	LEAD	4B/OK	Pass
H11	HHA	Blasted+ Zinc Phosphate	MIL-DTL-53022	MIL-DTL-53039	LEAD	3B/OK	Fail
H4	HHA	Blasted-only	Zinc-Rich+ MIL- DTL-53022	MIL-DTL-53039	LEAD	3B/OK	Fail
H8	HHA	Blasted+ X- Bond	Zinc-Rich+ MIL- DTL-53022	MIL-DTL-53039	LEAD	3B/OK	Fail

6.3.6 ASTM B117 Neutral Salt Spray Corrosion Testing of Fully Finished Demonstration Panels from LEAD and MDMC-PPA

Results, as seen in Tables 30–36, showed that X-Bond performed better than blasted-only and equal to or slightly worse than zinc phosphate on CRS, but it generally met the specification requirements of a minimum rating of six for steel. On aluminum substrates, X-Bond performed better than chrome 3+ and equal to chrome 6+, meeting the minimum rating of eight for aluminum. X-Bond met the

requirements of the specifications. Figures 18–24 show the performance of the various pretreatments on the different substrates with different stack-up systems from the demonstrations.

Table 30 ASTM B117 salt spray testing of fully finished CRS demonstration panels treated with X-Bond 4000 vs. blasted-only vs. zinc phosphate coated with MIL-DTL-53022 primer and MIL-DTL-53039 topcoat at LEAD

Code	Substrate	Treatment	Primer	Topcoat	Location	Duration	Scribe creep	Status
C3	CRS	Blasted-only	MIL-DTL-53022	MIL-DTL-53039	LEAD	336 h	6	Pass
C6	CRS	Blasted+ X-Bond	MIL-DTL-53022	MIL-DTL-53039	LEAD	336 h	7	Pass/ Sl worse than Zinc Phosphate
C9	CRS	Blasted+ Zinc Phosphate	MIL-DTL-53022	MIL-DTL-53039	LEAD	336 h	8	Pass

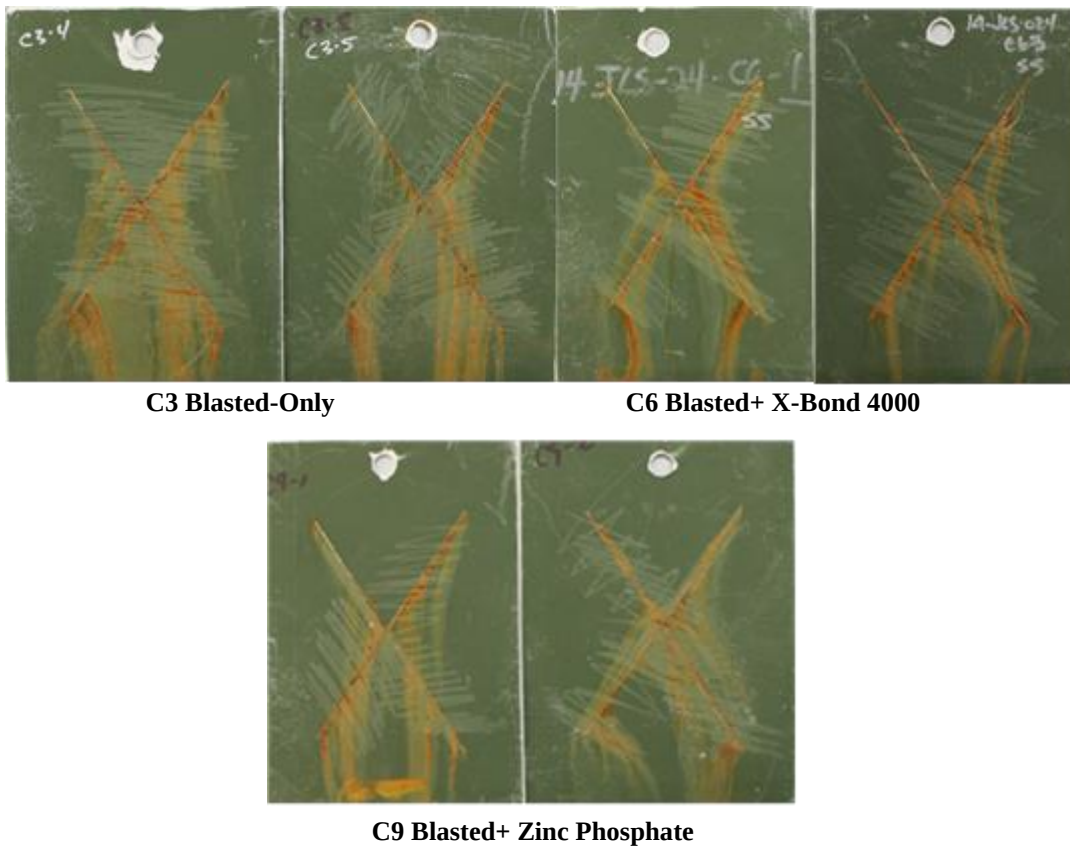


Fig. 18 TM B117 neutral salt spray testing of CRS demonstration panels treated with X-Bond 4000 vs. blasted-only vs. zinc phosphate under MIL-DTL-53022 primer and MIL-DTL-53039 topcoat at LEAD

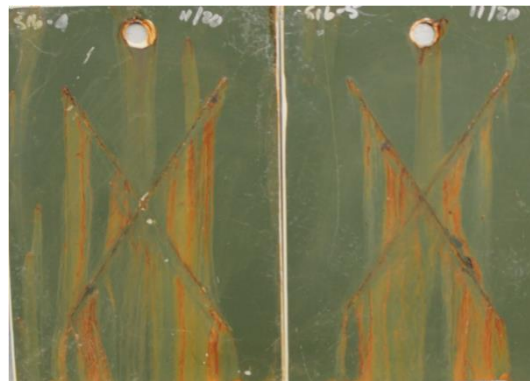
Table 31 ASTM B117 neutral salt spray testing of CRS demonstration panels treated with X-Bond 4000 vs. blasted-only vs. zinc phosphate under MIL-DTL-53030 primer and MIL-DTL-64159 topcoat at MDMC-PPA

Code	Substrate	Treatment	Primer	Topcoat	Location	Duration	Scribe creep	Status
S4	CRS	Blasted-only	MIL-DTL-53030	MIL-DTL-64159	MDMC-PPA	1000 h	5	Fail
S9	CRS	Blasted+ X-Bond	MIL-DTL-53030	MIL-DTL-64159	MDMC-PPA	1000 h	5	Fail/Worse than zinc phosphate
S16	CRS	Blasted+ Zinc Phosphate	MIL-DTL-53030	MIL-DTL-64159	MDMC-PPA	1000 h	8	Pass



S4 Blasted-Only

S9 Blasted+ X-Bond



S16 Blasted+ Zinc Phosphate

Fig. 19 1000-h ASTM B117 neutral salt spray testing of CRS demonstration panels treated with X-Bond 4000 vs. blasted-only vs. zinc phosphate under MIL-DTL-53030 primer and MIL-DTL-64159 topcoat at MDMC-PPA

Table 32 ASTM B117 neutral salt spray testing of CRS demonstration panels treated with X-Bond 4000 vs. zinc phosphate under MIL-DTL-53022 primer and MIL-DTL-53039 topcoat at MDMC-PPA

Code	Substrate	Treatment	Primer	Topcoat	Location	Duration	Scribe creep	Status
S11	CRS	Blasted+ X-Bond	MIL-DTL-53022	MIL-DTL-53039	MDMC-PPA	336 h	7	Pass/Equal to zinc phosphate
S19	CRS	Blasted+ Zinc Phosphate	MIL-DTL-53022	MIL-DTL-53039	MDMC-PPA	336 h	7	Pass

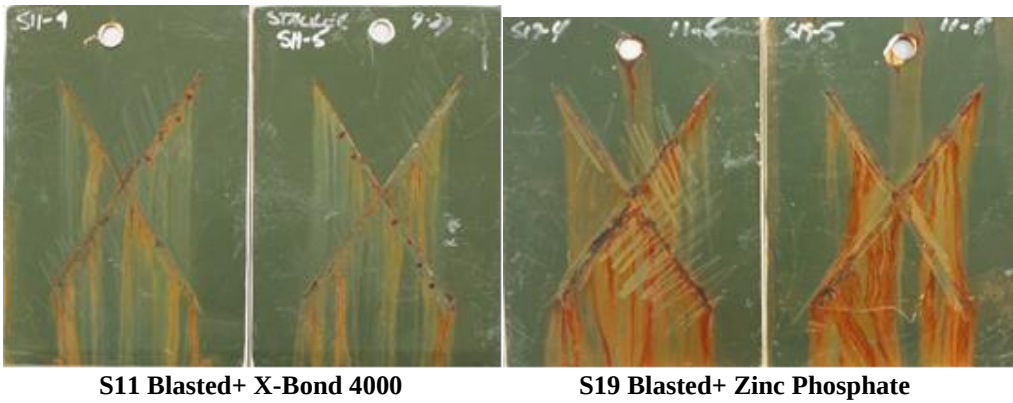
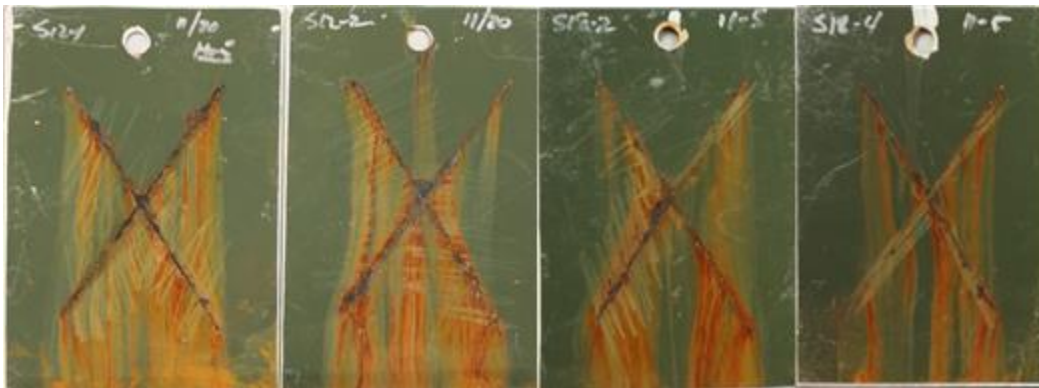


Fig. 20 ASTM B117 neutral salt spray testing for 336 h of CRS demonstration panels treated with X-Bond 4000 vs. zinc phosphate under MIL-DTL-53022 primer and MIL-DTL-53039 topcoat at MDMC-PPA

Table 33 ASTM B117 salt spray corrosion of CRS treated with X-Bond 4000 vs. blasted-only under MIL-PRF-32348 powder primer and MIL-DTL-64159 topcoat at MDMC-PPA

Code	Substrate	Treatment	Primer	Topcoat	Location	Duration	Scribe creep	Status
S12	CRS	Blasted+ X-Bond	MIL-PRF-32348	MIL-DTL-64159	MDMC-PPA	1000 h	7	Pass/Equal to zinc phosphate
S18	CRS	Blasted+ Zinc Phosphate	MIL-PRF-32348	MIL-DTL-64159	MDMC-PPA	1000 h	7	Pass



S12 Blasted+ X-Bond 4000

S18 Blasted+ Zinc Phosphate

Fig. 21 Salt spray corrosion for 1000 h of CRS treated with X-Bond 4000 vs. zinc phosphate under MIL-PRF-32348 powder primer and MIL-DTL-64159 topcoat at MDMC-PPA

Table 34 ASTM B117 neutral salt spray testing of CRS demonstration panels blasted-only vs. blasted+ X-Bond 4000 vs. blasted+ zinc phosphate under zinc-rich primer, MIL-DTL-53030 primer, and MIL-DTL-64159 topcoat at MDMC-PPA

Code	Substrate	Treatment	Primer	Topcoat	Location	Duration	Scribe creep	Status
S5	CRS	Blasted-only	Zinc-Rich + MIL-DTL-53030	MIL-DTL-64159	MDMC-PPA	1000 h	9	Pass
S10	CRS	Blasted+ X-Bond	Zinc-Rich + MIL-DTL-53022	MIL-DTL-64159	MDMC-PPA	1000 h	9	Pass/Equal to zinc phosphate
S17	CRS	Blasted+ Zinc Phosphate	Zinc-Rich + MIL-DTL-53022	MIL-DTL-64159	MDMC-PPA	1000 h	9	Pass

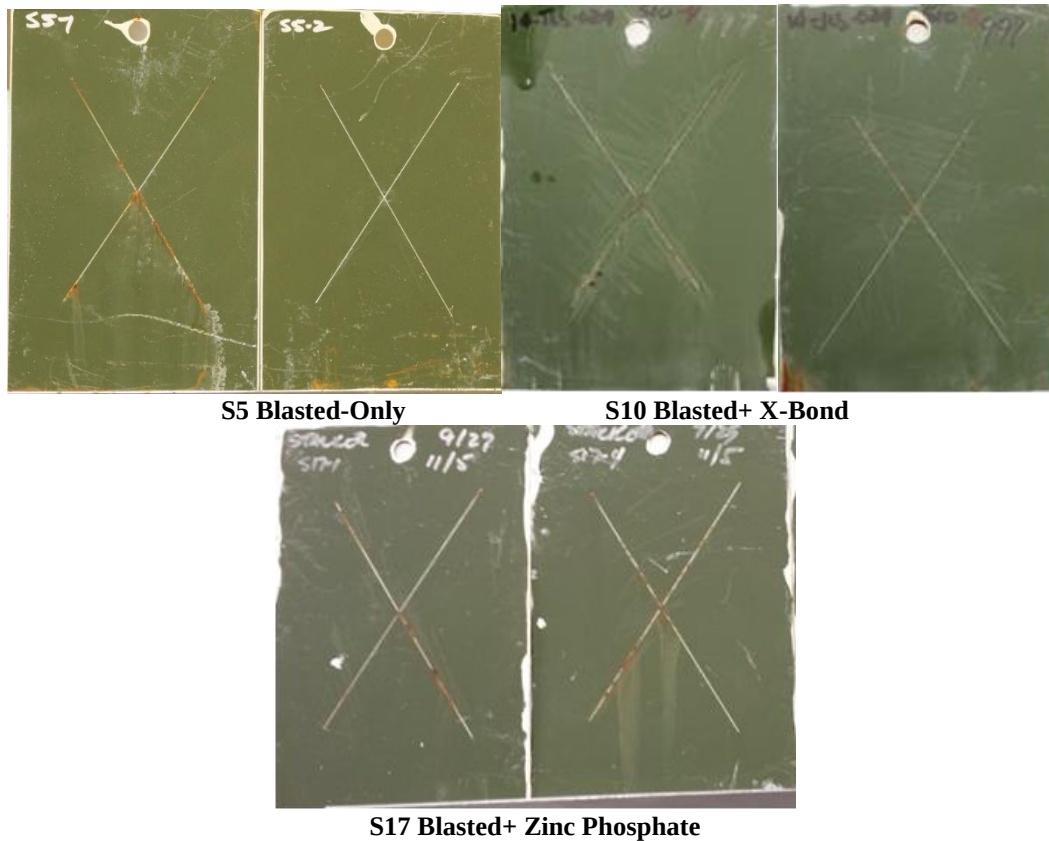


Fig. 22 ASTM B117 h neutral salt spray testing of CRS demonstration panels blasted-only vs. blasted+ X-Bond 4000 vs. blasted+ zinc phosphate under zinc-rich primer, MIL-DTL-53030 primer, and MIL-DTL-64159 topcoat at MDMC-PPA

Table 35 ASTM B117 salt spray corrosion of CRS brackets treated with X-Bond 4000 vs. zinc phosphate under MIL-DTL-53022 primer and MIL-DTL-53039 topcoat at LEAD

Code	Substrate	Treatment	Primer	Topcoat	Location	Duration	Scribe creep	Status
C11	CRS	Blasted+ X-Bond	MIL-DTL-53022	MIL-DTL-53039	LEAD	336 h	9	Pass/Equal to zinc phosphate
C12	CRS	Blasted+ Zinc Phosphate	MIL-DTL-53022	MIL-DTL-53039	LEAD	336 h	9	Pass

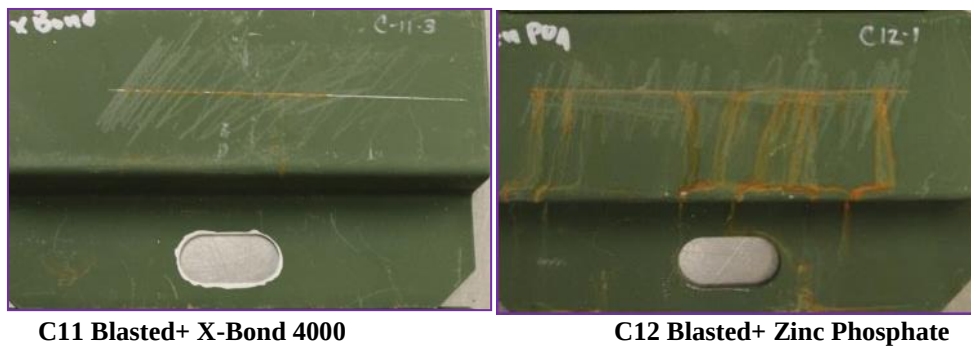


Fig. 23 ASTM B117 salt spray corrosion of CRS brackets treated with X-Bond 4000 vs. zinc phosphate under MIL-DTL-53022 primer and MIL-DTL-53039 topcoat at LEAD

Table 36 ASTM B117 salt spray corrosion of CRS D-ring lift hook surrounds treated with X-Bond 4000 vs. zinc phosphate under MIL-DTL-53030 primer and MIL-DTL-64159 topcoat at MDMC-PPA

Code	Substrate	Treatment	Primer	Topcoat	Location	Duration	Scribe creep	Status
S20	CRS	Blasted+ Zinc Phosphate	MIL-DTL-53030	MIL-DTL-64159	MDMC-PPA	1000 h	8	Pass
S21	CRS	Blasted+ X-Bond	MIL-DTL-53030	MIL-DTL-64159	MDMC-PPA	1000 h	8	Pass/Equal to zinc phosphate

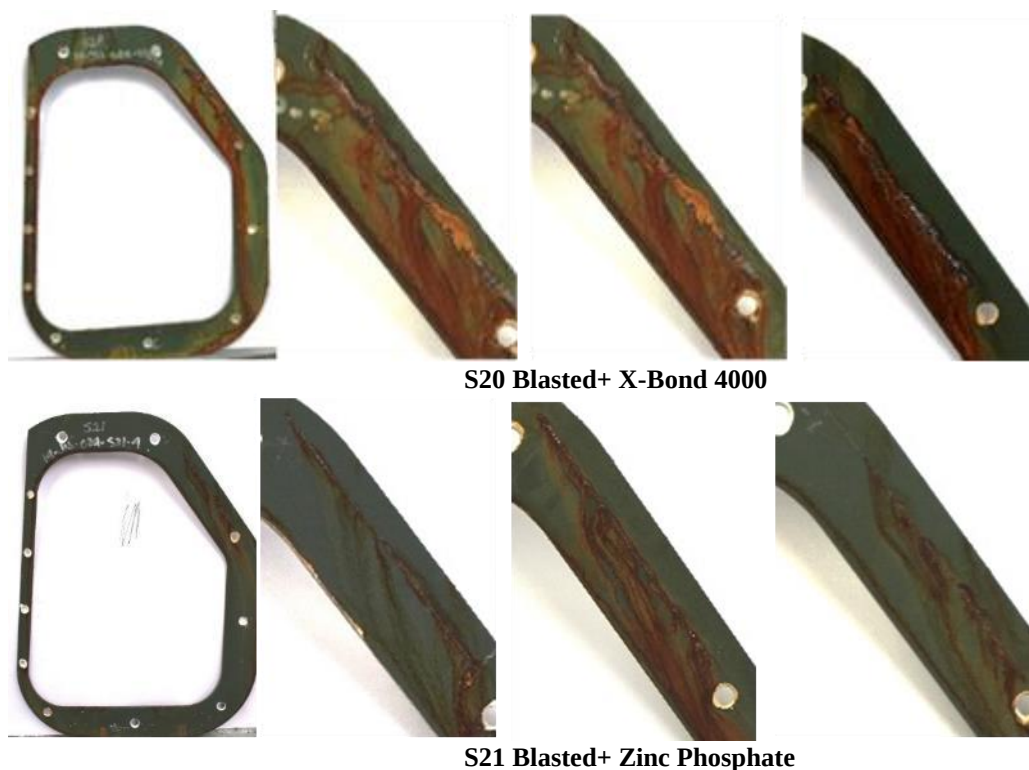


Fig. 24 Salt spray corrosion of CRS D-ring lift hook surrounds treated with X-Bond 4000 vs. zinc phosphate under MIL-DTL-53030 primer and MIL-DTL-64159 topcoat at MDMC-PPA

6.3.7 Profilometry Analysis of D-Ring Salt Spray Corrosion

In order to obtain additional measurements of the salt spray scribe corrosion of D-rings treated with X-Bond versus zinc phosphate, the exposed D-rings were submitted to the analytical chemistry department at the PPG Coatings Innovation Center for profilometry measurements.

One representative sample was chosen from both the X-Bond treated D-rings and the zinc phosphate treated D-rings. After stripping the primer and topcoat layers with DuraPrep Prep 200 Architectural Coating Remover, three profile measurements were made across the scribes at random points on each D-ring. Measurements were made of scribe width and scribe depth. Results show greater erosion of steel in the X-Bond-treated sample than the zinc-phosphate-treated sample as seen in Table 37. This was seen in the loss of both width and depth. No requirement is specified in the military specifications for the amount of metal erosion. This greater scribe volume loss may account for the larger amount of red rust along the scribe, even though the scribe creep of the zirconium is equal to that of zinc phosphate. The scribe width measurements correspond to a 9–10 rating, which is within the specification of a rating greater than six. Tables 38–42 show

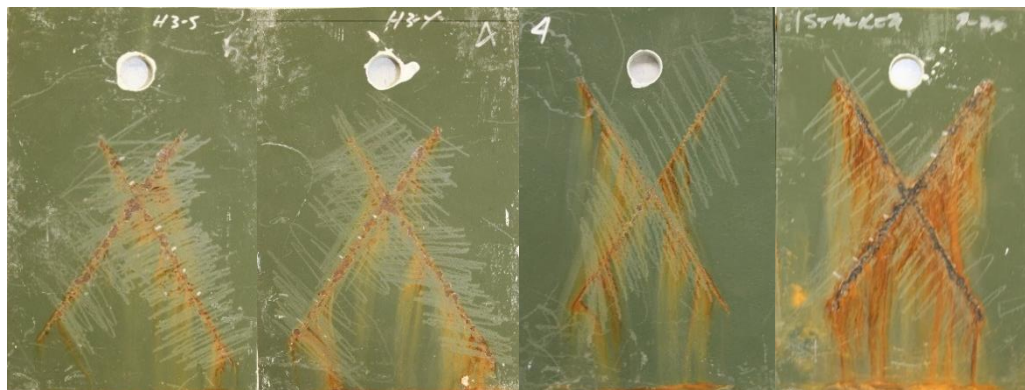
the corrosion results on HHA and aluminum panels from the demonstrations at LEAD and MDMC-PPA and the comparisons to the baseline systems. Figures 25–30 are the panels from those demonstrations for comparisons.

Table 37 Scribe profile of CRS D-rings treated with X-Bond vs. zinc phosphate after 336 h of ASTM B117 salt spray

Sample	Spot	Scribe width (μm)	Scribe depth (μm)
S20-X-Bond	1	210	50
	2	230	43
	3	420	77
Average		290	580
S21-Zinc Phosphate	1	85	6
	2	240	25
	3	213	24
Average		180	18

Table 38 ASTM B117 salt spray testing of HHA steel demonstration panels blasted-only vs. blasted+ X-Bond 4000 under MIL-DTL-53022 primer and MIL-DTL-53039 topcoat at LEAD

Code	Substrate	Treatment	Primer	Topcoat	Location	Duration	Scribe creep	Status
H3	HHA	Blasted-only	MIL-DTL-53022	MIL-DTL53039	LEAD	336 h	7	Pass
H7	HHA	Blasted+ X-Bond	MIL-DTL-53022	MIL-DTL53039	LEAD	336 h	7	Pass /Worse than zinc phosphate
H11	HHA	Blasted+ Zinc Phosphate	MIL-DTL-53022	MIL-DTL53039	LEAD	336 h	9	Pass
A3	HHA	Blasted-only	MIL-DTL-53030	MIL-DTL64159	MDMC-PPA	1000 h	3	Fail
A7	HHA	Blasted+ X-Bond	MIL-DTL-53030	MIL-DTL64159	MDMC-PPA	1000 h	6	Fail/Worse than zinc phosphate



H3 Blasted-Only

H7 Blasted+ X-Bond



H11 Blasted+ Zinc Phosphate

Fig. 25 Neutral salt spray testing for 336 h of HHA steel demonstration panels blasted-only vs. blasted+ X-Bond 4000 vs. blasted+ zinc phosphate under MIL-DTL-53022 primer and MIL-DTL-53039 topcoat at LEAD



A3 Blasted-Only

A7 Blasted+ X-Bond

Fig. 26 Neutral salt spray testing for 1000 h of HHA steel demonstration panels blasted-only vs. blasted+ X-Bond 4000 under MIL-DTL-53030 primer and MIL-DTL-64159 topcoat at MDMC-PPA

Table 39 ASTM B117 salt spray testing of HHA steel demonstration panels blasted-only vs blasted and treated with X-Bond 4000 under zinc-rich primer, MIL-DTL-53022 primer, and MIL-DTL-53039 topcoat at LEAD

Code	Substrate	Treatment	Primer	Topcoat	Location	Duration	Scribe creep	Status
H4	HHA	Blasted-only	Zinc-Rich + MIL-DTL-53022	MIL-DTL-53039	LEAD	1000 h	9	Pass
H8	HHA	Blasted+ X-Bond	MIL-DTL-53022	MIL-DTL-53039	LEAD	1000 h	9	Pass/ Blasted-only



H4 Blasted-Only

H8 Blasted+ X-Bond

Fig. 27 Neutral salt spray testing for 1000 h of HHA steel demonstration panels blasted-only vs. blasted+ X-Bond 4000 under zinc-rich primer, MIL-DTL-53022 primer, and MIL-DTL-53039 topcoat at LEAD

Table 40 ASTM B117 salt spray testing of aluminum demonstration chrome 6+ vs. X-Bond 4000 under MIL-DTL-53022 primer and MIL-DTL-53039 topcoat at LEAD

Code	Substrate	Treatment	Primer	Topcoat	Location	Duration	Scribe creep	Status
A6	2024T3 Aluminum	Chrome 6+	MIL-DTL-53022	MIL-DTL53039	LEAD	1000 h	10	Pass
A3	2024T3 Aluminum	X-Bond	MIL-DTL-53022	MIL-DTL53039	LEAD	1000 h	9	Pass/Equal to Cr ⁶⁺

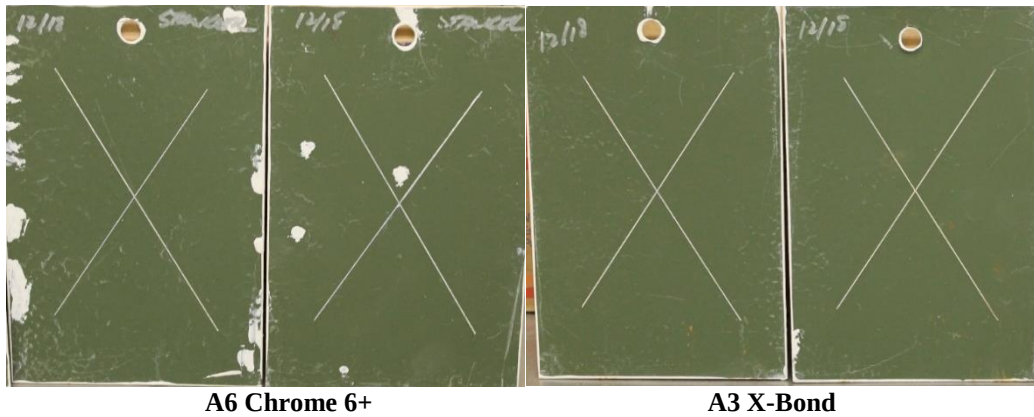


Fig. 28 ASTM B117 salt spray testing of aluminum demonstration panels Cr6+ vs. X-Bond 4000 under MIL-DTL-53022 primer and MIL-DTL-53039 topcoat at LEAD

Table 41 ASTM B117 salt spray testing of aluminum demonstration panels Cr³⁺ vs. X-Bond 4000 under MIL-DTL-53030 primer and MIL-DTL-64159 topcoat at MDMC-PPA

Code	Substrate	Treatment	Primer	Topcoat	Location	Duration	Scribe creep	Status
M8	2024T3 Aluminum	Chrome 3+	MIL-DTL-53030	MIL-DTL64159	MDMC-PPA	1000 h	9	Pass / Equal to Cr ³⁺
M3	2024T3 Aluminum	X-Bond	MIL-DTL-53030	MIL-DTL64159	MDMC-PPA	1000 h	9	Pass

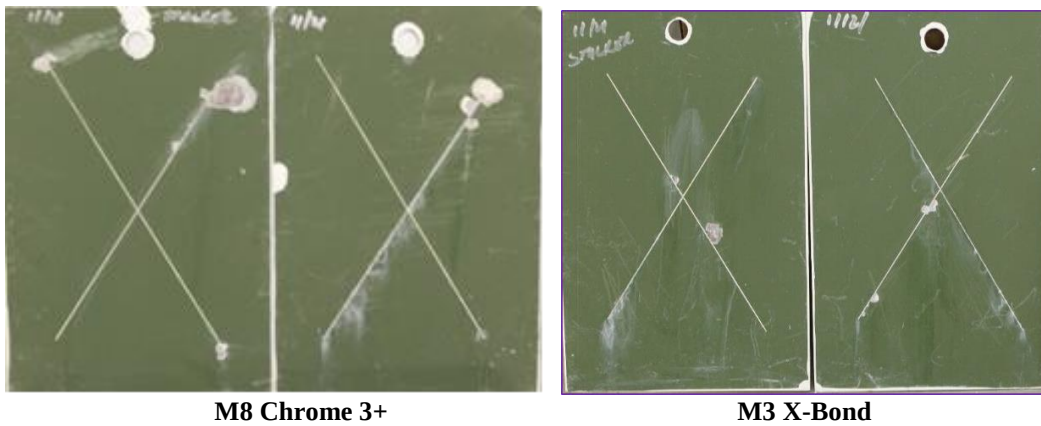


Fig. 29 1000-h neutral salt spray testing of aluminum demonstration panels Cr3+ vs. X-Bond 4000 under MIL-DTL-53030 primer and MIL-DTL-64159 topcoat at MDMC-PPA

Table 42 ASTM B117 neutral salt spray testing of aluminum demonstration panels Cr³⁺ vs. X-Bond 4000 under MIL-PRF-32348 powder primer and MIL-DTL-64159 topcoat at MDMC-PPA

Code	Substrate	Treatment	Primer	Topcoat	Location	Duration	Scribe creep	Status
M9	2024T3 Aluminum	Chrome 3+	MIL-PRF-32348	MIL-DTL-64159	MDMC-PPA	3000 h	7	Fail
M4	2024T3 Aluminum	X-Bond	MIL-PRF-32348	MIL-DTL-64159	MDMC-PPA	3000 h	9	Pass/ Better than Cr ³⁺

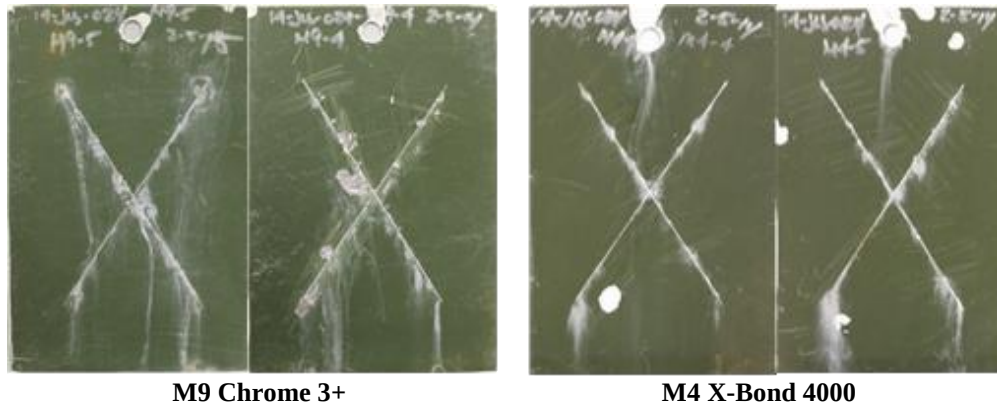


Fig. 30 3000-h neutral salt spray testing of aluminum demonstration panels Cr³⁺ vs. X-Bond 4000 under MIL-PRF-32348 powder primer and MIL-DTL-64159 topcoat at MDMC-PPA

6.3.8 Cyclic Corrosion Test GMW 14872 of Fully Finished Panels

Cyclic corrosion testing was carried out using test method GMW 14872. This method calls for cycles consisting of a salt spray step, a warm/humid step, and a hot drying step. Each combination of primer and substrate requires a specific number of cycles per TT-C-490 and the primer specifications. After completing the required cycles, panels were examined for corrosion along a scribed line and rated for extent of corrosion per ASTM D1654. Results for each substrate were tabulated separately and shown in Tables 43–52. Pictures of the panels from this testing are shown in Figures 31–40. The X-Bond 4000 zirconium pretreatment improved the corrosion performance of blasted-only panels and was equal to zinc phosphate pretreatment on HHA steel and CRS panels under all primers. On aluminum, X-Bond was equal to both chrome 3+ and chrome 6+ under all primers.

Table 43 MW 14872 cyclic corrosion testing of fully finished CRS demonstration panels treated with X-Bond 4000 vs. blasted-only vs. zinc phosphate treatments at LEAD

Code	Substrate	Treatment	Primer	Topcoat	Location	Duration	Scribe creep	Status
C3	CRS	Blasted-only	MIL-DTL-53022	MIL-DTL-53039	LEAD	20 cycles	6	Pass
C6	CRS	Blasted+ X-Bond	MIL-DTL-53022	MIL-DTL-53039	LEAD	20 cycles	7	Pass/Sl worse than zinc phosphate
C9	CRS	Blasted+ Zinc Phosphate	MIL-DTL-53022	MIL-DTL-53039	LEAD	20 cycles	8	Pass

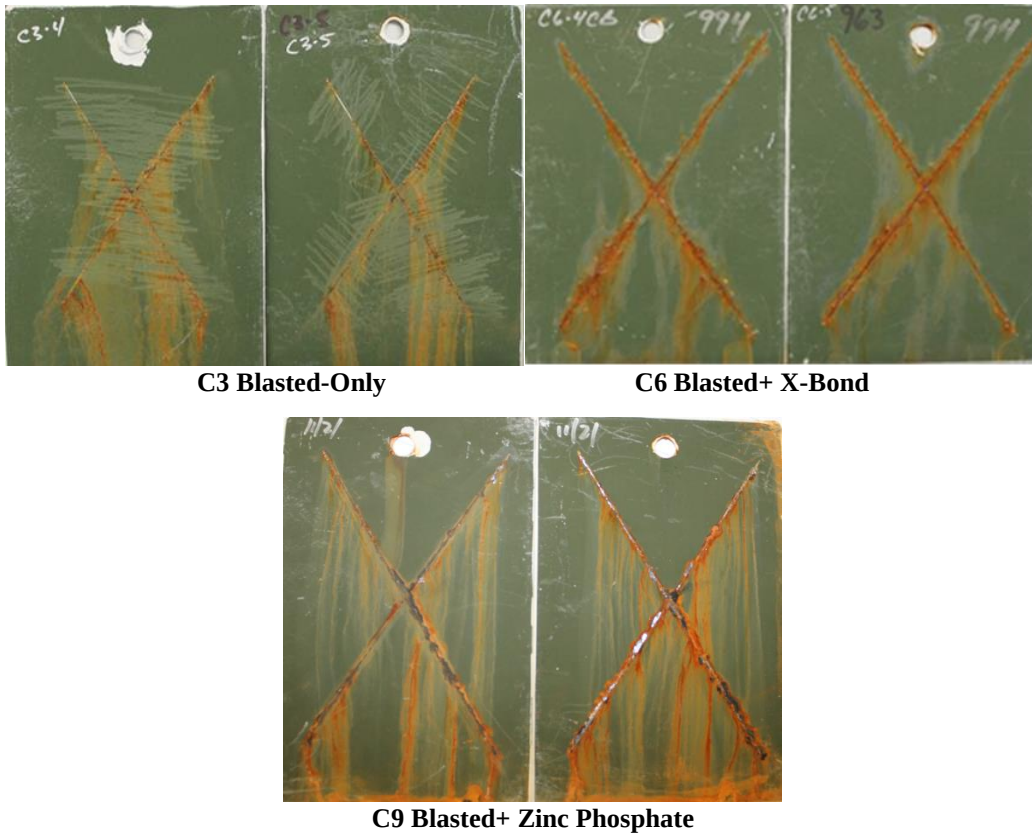


Fig. 31 GMW 14872 cyclic corrosion testing of CRS demonstration panels X-Bond 4000 versus blasted-only vs. zinc phosphate under MIL-DTL-53022 primer and MIL-DTL-53039 topcoat at LEAD

Table 44 GMW 14872 cyclic corrosion testing of CRS demonstration panels X-Bond 4000 vs. zinc phosphate under MIL-DTL-53022 primer and MIL-DTL-53039 topcoat at MDMC-PPA

Code	Substrate	Treatment	Primer	Topcoat	Location	Duration	Scribe creep	Status
S11	CRS	Blasted+ X-Bond	MIL-DTL-53022	MIL-DTL-53039	MDMC-PPA	20 cycles	7	Pass/Equal to zinc phosphate
S19	CRS	Blasted+ Zinc Phosphate	MIL-DTL-53022	MIL-DTL-53039	MDMC-PPA	20 cycles	7	Pass

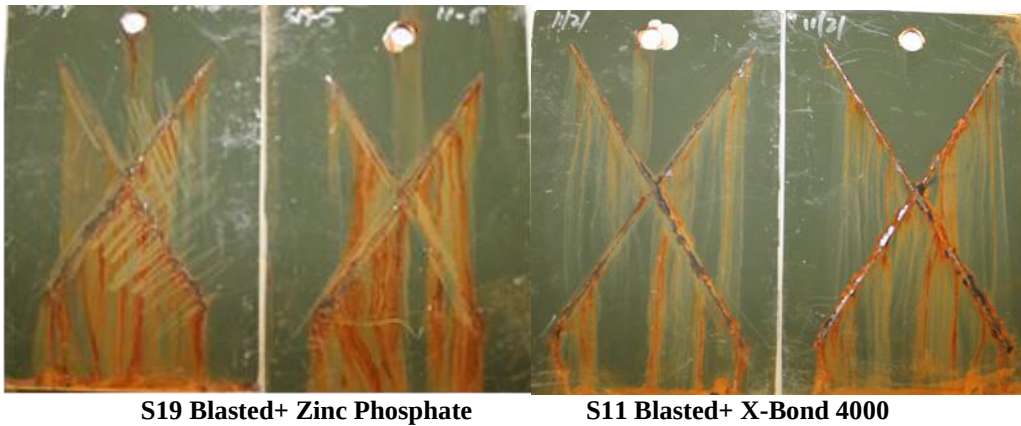


Fig. 32 GMW 14872 cyclic corrosion testing of CRS demonstration panels X-Bond 4000 vs. zinc phosphate under MIL-DTL-53022 primer and MIL-DTL-53039 topcoat at MDMC-PPA

Table 45 Thirty cycles GMW 14872 cyclic corrosion testing of CRS demonstration panels X-Bond 4000 vs. blasted-only vs. zinc phosphate under MIL-DTL-53030 primer and MIL-DTL-64159 topcoat at MDMC-PPA

Code	Substrate	Treatment	Primer	Topcoat	Location	Duration	Scribe creep	Status
S4	CRS	Blasted-only	MIL-DTL-53030	MIL-DTL-64159	MDMC-PPA	20 cycles	5	Fail
S9	CRS	Blasted+ X-Bond	MIL-DTL-53030	MIL-DTL-64159	MDMC-PPA	20 cycles	8	Pass/Equal to zinc phosphate
S16	CRS	Blasted+ Zinc Phosphate	MIL-DTL-53030	MIL-DTL-64159	MDMC-PPA	20 cycles	8	Pass

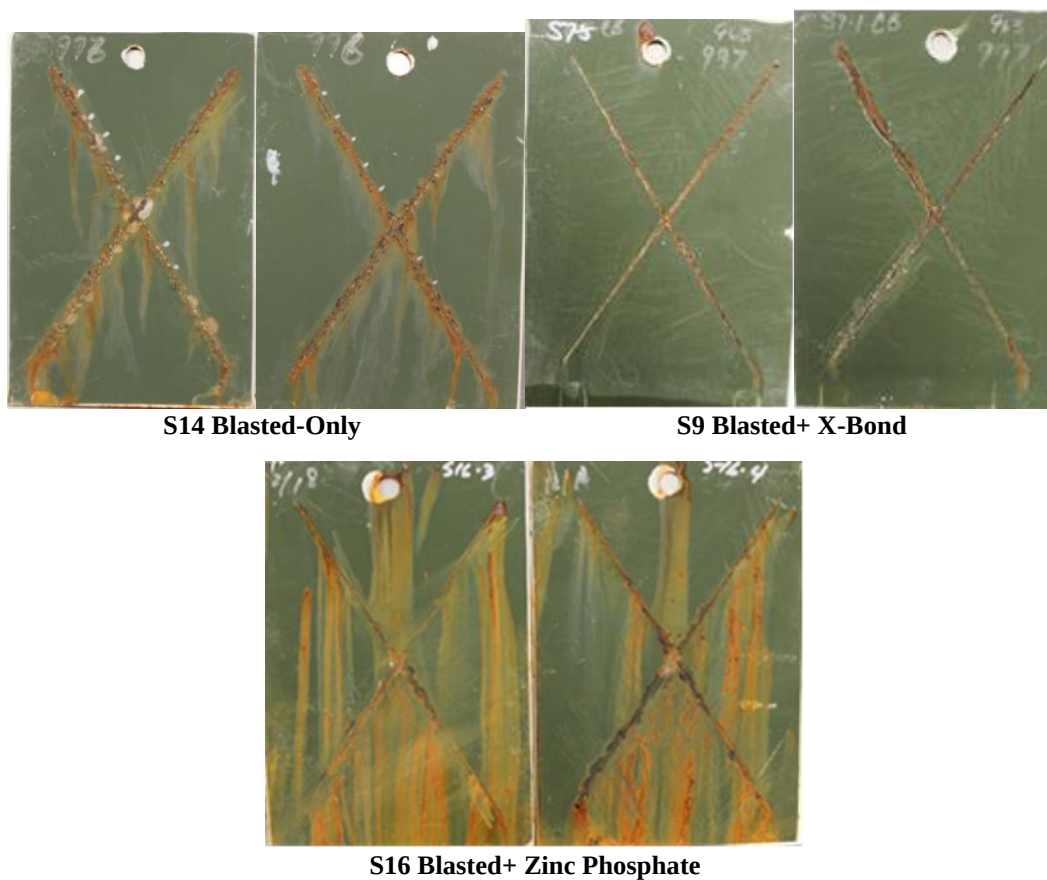


Fig. 33 GMW 14872 cyclic corrosion testing of CRS demonstration panels X-Bond 4000 vs. blasted-only vs. zinc phosphate under MIL-DTL-53030 primer and MIL-DTL-64159 topcoat at MDMC-PPA

Table 46 GMW 14872 cyclic corrosion testing of CRS demonstration panels X-Bond 4000 vs. zinc phosphate under MIL-PRF-32348 powder primer and MIL-DTL-64159 topcoat at MDMC-PPA

Code	Substrate	Treatment	Primer	Topcoat	Location	Duration	Scribe creep	Status
S18	CRS	Blasted+ Zinc Phosphate	MIL-PRF-32348	MIL-DTL-64159	MDMC-PPA	60 cycles	7	Pass
S12	CRS	Blasted+ X-Bond	MIL-PRF-32348	MIL-DTL-64159	MDMC-PPA	60 cycles	7	Pass/ Equal to zinc phosphate

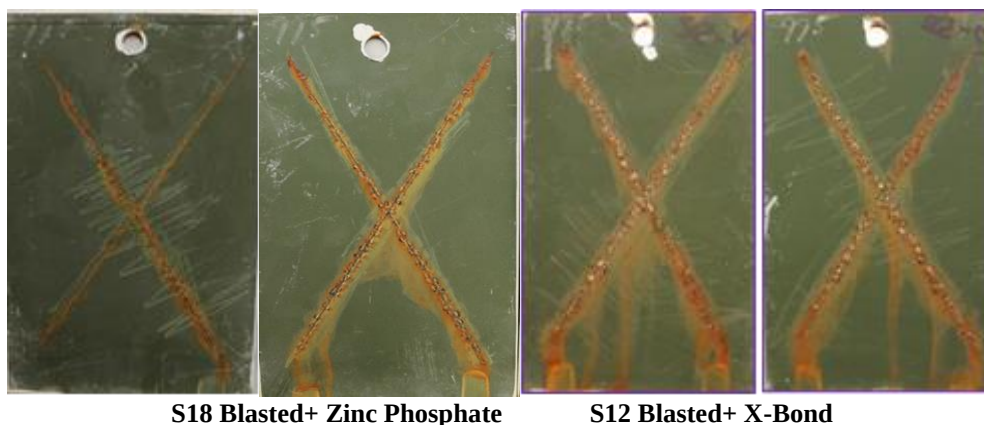


Fig. 34 Sixty cycles GMW 14872 cyclic corrosion testing of CRS demonstration panels X-Bond 4000 vs. zinc phosphate under MIL-PRF-32348 powder primer and MIL-DTL-64159 topcoat at MDMC-PPA

Table 47 GMW 14872 cyclic corrosion testing of CRS demonstration panels X-Bond 4000 vs. blasted-only versus zinc phosphate under MIL-PRF-32348 powder primer and MIL-DTL-64159 topcoat at MDMC-PPA

Code	Substrate	Treatment	Primer	Topcoat	Location	Duration	Scribe creep	Status
S5	CRS	Blasted-only	Zinc-Rich+ MIL-DTL-53030	MIL-DTL-64159	MDMC-PPA	120 cycles	9	Pass
S10	CRS	Blasted+ X-Bond	Zinc-Rich+ MIL-DTL-53030	MIL-DTL-64159	MDMC-PPA	120 cycles	10	Pass/ Equal to zinc phosphate
S17	CRS	Blasted+ Zinc Phosphate	Zinc-Rich+ MIL-DTL-53030	MIL-DTL-64159	MDMC-PPA	120 cycles	10	Pass

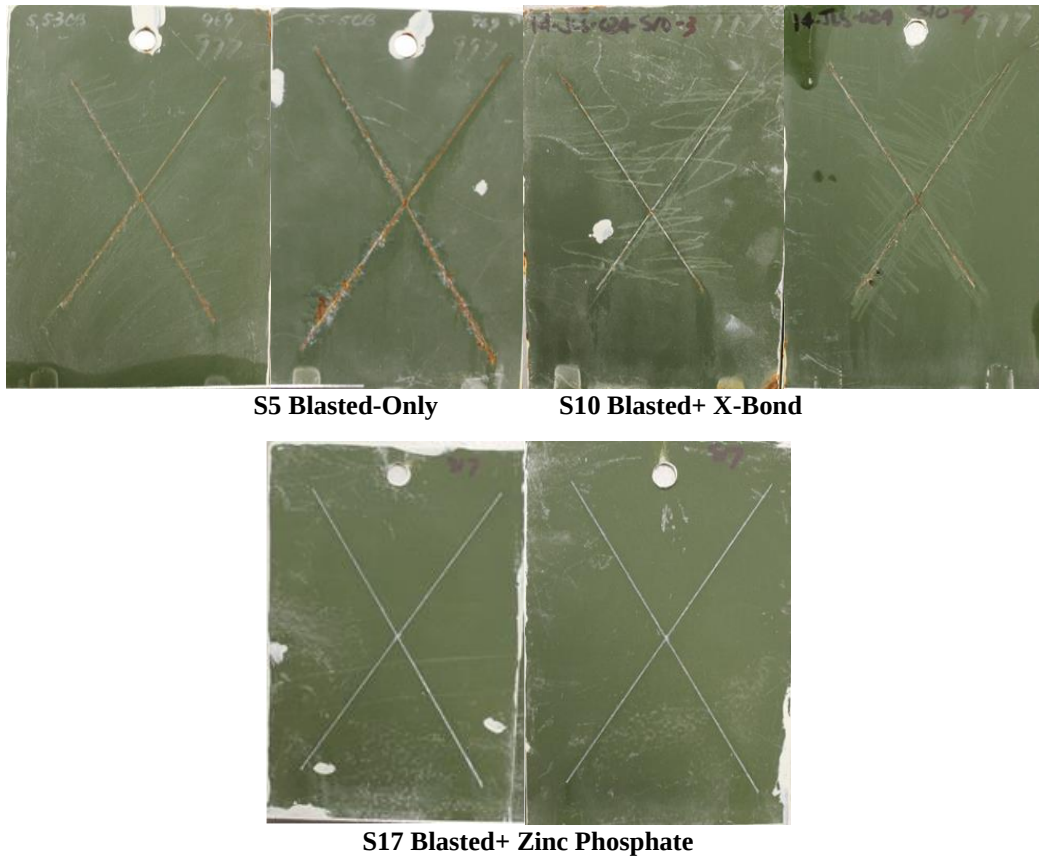


Fig. 35 GMW 124872 cyclic corrosion testing of CRS demonstration panels X-Bond 4000 versus blasted-only vs. zinc phosphate under zinc-rich and MIL-DTL-53030 primers and MIL-DTL-64159 topcoat at MDMC-PPA

Table 48 GMW 14872 cyclic corrosion testing of HHA demonstration panels blasted-only vs. blasted+ X-Bond 4000 under zinc-rich and MIL-DTL-53022 primers and MIL-DTL-53039 topcoat at LEAD

Code	Substrate	Treatment	Primer	Topcoat	Location	Test Duration	Scribe creep	Status
H4	HHA	Blasted-only	MIL-DTL-53022	MIL-DTL-53039	LEAD	20 cycles	6	Pass
H8	HHA	Blasted+ X-Bond	MIL-DTL-53022	MIL-DTL-53039	LEAD	20 cycles	7	Pass/ Better than blasted-only

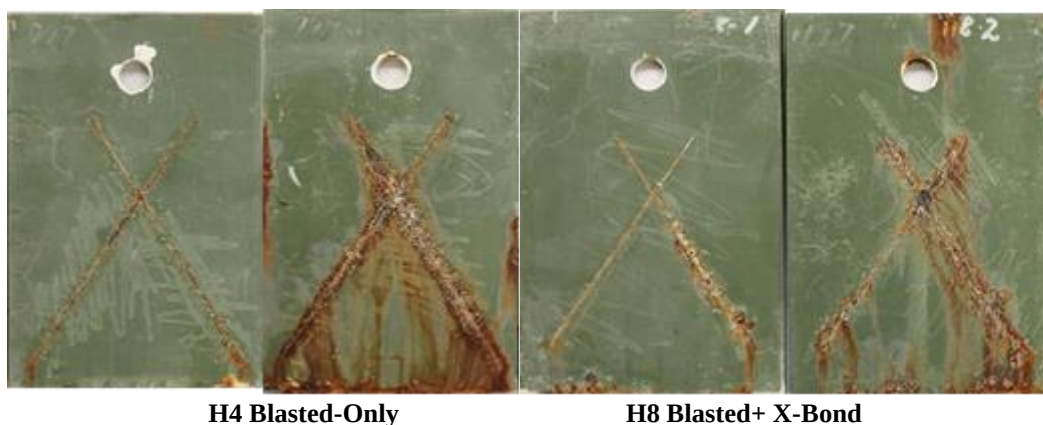


Fig. 36 GMW 14872 cyclic corrosion testing of HHA steel blasted-only vs. blasted+ X-Bond 4000 under zinc-rich and MIL-DTL-53022 primers and MIL-DTL-53039 topcoat at LEAD

Table 49 GMW 14872 cyclic corrosion testing of aluminum demonstration panels chrome 6+ vs. X-Bond 4000 under MIL-DTL-53022 primer and MIL-DTL-53039 topcoat at LEAD

Code	Substrate	Treatment	Primer	Topcoat	Location	Duration	Scribe creep	Status
A6	2024T3 Aluminum	Chrome 6+	MIL-DTL-53022	MIL-DTL-53039	LEAD	40 cycles	10	Pass
A3	2024T3 Aluminum	X-Bond	MIL-DTL-53022	MIL-DTL-53039	LEAD	40 cycles	10	Pass / Equal to Cr ⁶⁺

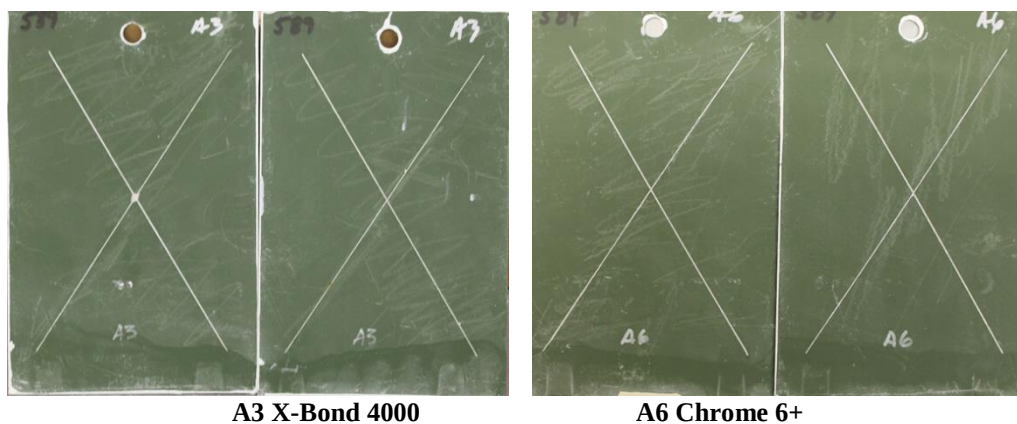


Fig. 37 Forty cycles GMW 14872 cyclic corrosion testing of AA6061 aluminum treated with X-Bond 4000 vs. chrome 3+ under MIL-DTL-53022 primer and MIL-DTL-53039 topcoat at LEAD

Table 50 GMW 14872 cyclic corrosion testing of aluminum demonstration panels chrome 3+ vs. X-Bond 4000 under MIL-DTL-53022 primer and MIL-DTL-53039 topcoat at MDMC-PPA

Code	Substrate	Treatment	Primer	Topcoat	Location	Duration	Scribe creep	Status
M5	2024T3 Aluminum	X-Bond	MIL-DTL-53022	MIL-DTL-53039	MDMC-PPA	20 cycles	10	Pass
M10	2024T3 Aluminum	Chrome 3+	MIL-DTL-53022	MIL-DTL-53039	MDMC-PPA	20 cycles	10	Pass/ equal Equal to Cr ³⁺

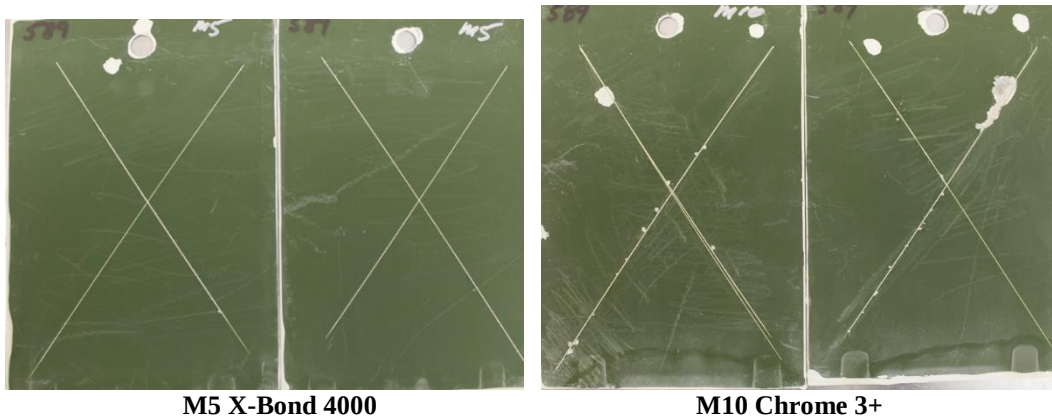


Fig. 38 Forty cycles GMW 14872 cyclic corrosion testing of AA6061 aluminum treated with X-Bond 4000 vs. chrome 3+ under MIL-DTL-53022 primer and MIL-DTL-53039 topcoat at MDMC-PPA

Table 51 GMW 14872 cyclic corrosion testing of aluminum demonstration panels chrome 3+ vs. X-Bond 4000 under MIL-DTL-53030 primer and MIL-DTL-53039 topcoat at MDMC-PPA

Code	Substrate	Treatment	Primer	Topcoat	Location	Duration	Scribe creep	Status
M8	2024T3 Aluminum	Chrome 3+	MIL-DTL-53030	MIL-DTL-64159	MDMC-PPA	30 cycles	9.5	Pass
M3	2024T3 Aluminum	X-Bond	MIL-DTL-53030	MIL-DTL-64159	MDMC-PPA	30 cycles	10	Pass/ Better than Cr ³⁺

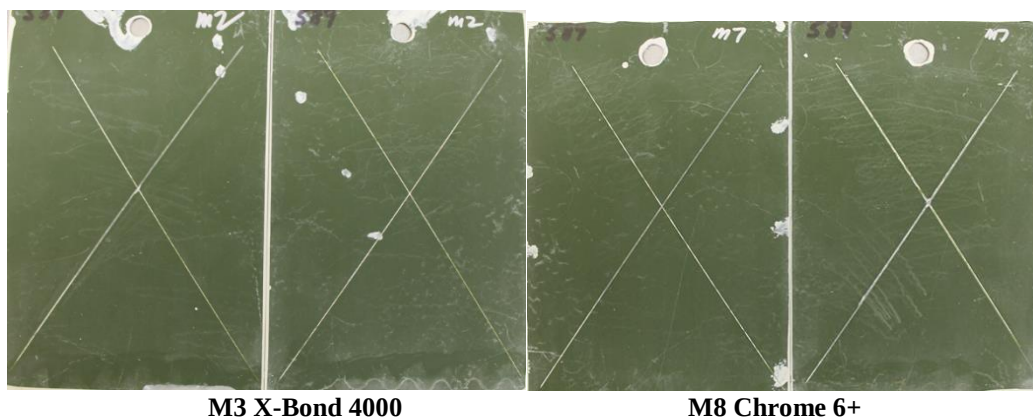


Fig. 39 GMW 14872 cyclic corrosion testing of AA6061 aluminum treated with X-Bond 4000 vs. chrome 3+ under MIL-DTL-53030 primer and MIL-DTL-64159 topcoat at MDMC-PPA

Table 52 GMW 14872 cyclic corrosion testing of aluminum demonstration panels chrome 3+ vs. X-Bond 4000 under MIL-PRF-32348 powder primer and MIL-DTL-64159 topcoat at MDMC-PPA

Code	Substrate	Treatment	Primer	Topcoat	Location	Duration	Scribe creep	Status
M9	2024T3 Aluminum	Chrome 3+	MIL-PRF-32348	MIL-DTL-64159	MDMC-PPA	45 cycles	10	Pass
M4	2024T3 Aluminum	X-Bond	MIL-PRF-32348	MIL-DTL-64159	MDMC-PPA	45 cycles	10	Pass/ Equal to Cr ³⁺

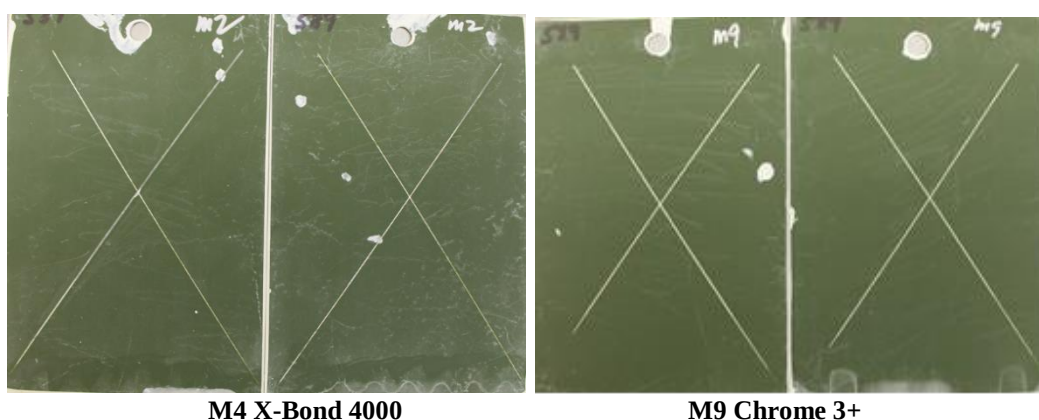


Fig. 40 Forty-five cycles GMW 14872 cyclic corrosion testing of AA6061 aluminum treated with X-Bond 4000 vs. chrome 6+ under MIL-PRF-32348 powder primer and MIL-DTL-64159 topcoat at MDMC-PPA

6.3.9 Marine Environment Outdoor Exposure ASTM G50

This test evaluates the ability of a coating system to prevent substrate corrosion in an outdoor marine environment. Exposure testing was conducted at the CCAFS corrosion test site, located 100 yards from and facing the Atlantic Ocean. Test coupons were installed on nonmetallic racks using plastic insulator stand-offs with stainless steel fasteners. The rack angle of the coupons was 30° from horizontal. Panels were evaluated every 3 months for scribe creep.

6.3.10 Twelve Months' Exposure of Demonstration Panels at Kennedy Space Center

After two years of exposure, the corrosion requirement for painted steel is a minimum rating of six. For aluminum, a minimum rating of eight is required. After 12 months of exposure, X-Bond performance on aluminum was equal to chrome 6+ performance and better than chrome 3+ performance. The aluminum panels coated with MIL-DTL-53022 over both X-Bond 4000 and over chrome 3+ at MDMC-PPA failed to meet the minimum rating of 6 after 12 months. Chrome 3+ on aluminum under MIL-DTL-53030 primer also failed to meet the minimum after 12 months, but X-Bond 4000 on aluminum under the same primer passed. X-Bond 4000 performance was equal to chrome 3+ under MIL-PRF-32348 powder primer. On CRS, X-Bond ranged from slightly worse than to better than zinc phosphate. It performed slightly worse than zinc phosphate under MIL-DTL-53022 primer at LEAD and equal to zinc phosphate under the same primer at MDMC-PPA. X-Bond performed the same as zinc phosphate both when used under the zinc-rich primer/MIL-DTL-53022 combination and when used under MIL-DTL-53030. On HHA steel, X-Bond was better than zinc phosphate under MIL-DTL-53022 at MDMC-PPA and worse under the same primer at LEAD. Under zinc-rich primer, X-Bond was a better treatment than blasted-only. Photographs were not available from CCAFS. Results were tabulated according to primer, substrate, and location in Table 53.

Table 53 Average scribe creep ratings of aluminum, steel, and HHA panels after 12 months' exposure at Kennedy Space Center beach-front exposure site from June 2015 to June 2016

Code	Location	Substrate	Treatment	Coating	Average scribe creep (per ASTM D1654)	Status
A3	LEAD	Aluminum	X-Bond	MIL-DTL-53022+ MIL-DTL-53039	10	Pass/ Equal to Cr ⁶⁺
A6	LEAD	Aluminum	Chrome 6+	MIL-DTL-53022+ MIL-DTL-53039	10	Pass
C3	LEAD	CRS	Blasted-only	MIL-DTL-53022+ MIL-DTL-53039	7	Pass
C6	LEAD	CRS	Blasted+ X-Bond	MIL-DTL-53022+ MIL-DTL-53039	7	Pass/ Sl worse than zinc phosphate
C9	LEAD	CRS	Blasted+ Zinc Phosphate	MIL-DTL-53022+ MIL-DTL-53040	8	Pass
H3	LEAD	HHA	Blasted-only	MIL-DTL-53022+ MIL-DTL-53039	-	No Data
H7	LEAD	HHA	X-Bond	MIL-DTL-53022+ MIL-DTL-53039	8	Pass/ Better than zinc phosphate
H11	LEAD	HHA	Zinc Phosphate	MIL-DTL-53022 MIL-DTL-53039	7	Pass
H4	LEAD	HHA	Blasted-only	Zinc-Rich+ MIL-DTL-53022+ MIL-DTL-53039	9	Pass
H8	LEAD	HHA	X-Bond	Zinc-Rich+ MIL-DTL-53022+ MIL-DTL-53039	10	Pass/ Better than blasted-only
M4	MDMC-PPA	Aluminum	X-Bond	MIL-PRF-32348+ MIL-DTL-64159	10	Pass/ Equal to Cr ³⁺
M9	MDMC-PPA	Aluminum	Chrome 3+	MIL-DTL-32348+ MIL-DTL-64159	10	Pass
M5	MDMC-PPA	Aluminum	X-Bond	MIL-DTL-53022+ MIL-DTL-64159	4	Fail/ Better than Cr ³⁺
M10	MDMC-PPA	Aluminum	Chrome 3+	MIL-DTL-53022+ MIL-DTL-64159	3	Fail
M3	MDMC-PPA	Aluminum	X-Bond	MIL-DTL-53030+ MIL-DTL-64159	8	Pass/ Better than Cr ³⁺
M8	MDMC-PPA	Aluminum	Chrome 3+	MIL-DTL-53030+ MIL-DTL-64159	5	Fail
S2/4	MDMC-PPA	CRS	Blasted-only	MIL-DTL-53030+ MIL-DTL-64159	9	Pass

Table 53 Average scribe creep ratings of aluminum, steel, and HHA panels after 12 months' exposure at Kennedy Space Center beach-front exposure site from June 2015 to June 2016 (continued)

Code	Location	Substrate	Treatment	Coating	Average scribe creep (per ASTM D1654)	Status
S9	MDMC-PPA	CRS	Blasted+ X-Bond	MIL-DTL-53030+ MIL-DTL-64159	9	Pass/Better than zinc phosphate
S12	MDMC-PPA	CRS	Blasted+ X-Bond	MIL-PRF-32348+ MIL-DTL-64159	9	Pass/Equal to zinc phosphate
S18	MDMC-PPA	CRS	Blasted+ Zinc Phosphate	MIL-PRF-32348+ MIL-DTL-64159	9	Pass
S11	MDMC-PPA	CRS	Blasted+ X-Bond	MIL-DTL-53022+ MIL-DTL-64159	10	Pass/Equal to zinc phosphate
S19	MDMC-PPA	CRS	Blasted+ Zinc Phosphate	MIL-DTL-53022+ MIL-DTL-64159	10	Pass
S5	MDMC-PPA	CRS	Blasted-only	Zinc-Rich+ MIL-DTL-53030+ MIL-DTL-64159	10	Pass
S10	MDMC-PPA	CRS	Blasted+ X-Bond	Zinc-Rich+ MIL-DTL-53030+ MIL-DTL-64159	10	Pass/Equal to zinc phosphate
S17	MDMC-PPA	CRS	Blasted+ Zinc Phosphate	Zinc-Rich+ MIL-DTL-53030+ MIL-DTL-64159	10	Pass
A4	MDMC-PPA	HHA	X-Bond	MIL-DTL-53030+ MIL-DTL-64159	10	Pass

6.3.11 Twenty-Four Months' Exposure of Demonstration Parts at CCAFS

Parts that were painted during the demonstrations at LEAD and miscellaneous aluminum parts and CRS cable hold-down brackets were exposed at the CCAFS static outdoor corrosion site. Parts from MDMC-PPA included aluminum battery hold-down brackets for HMMWVs, as seen in Figs. 41 and 42, and CRS D-ring lift hook surrounds for HMMWVs, as seen in Fig. 43. Results on these parts duplicated results found on the panels.

At LEAD, X-Bond 4000 was equal to chrome 6+ on aluminum, with no corrosion observed after 24 months.

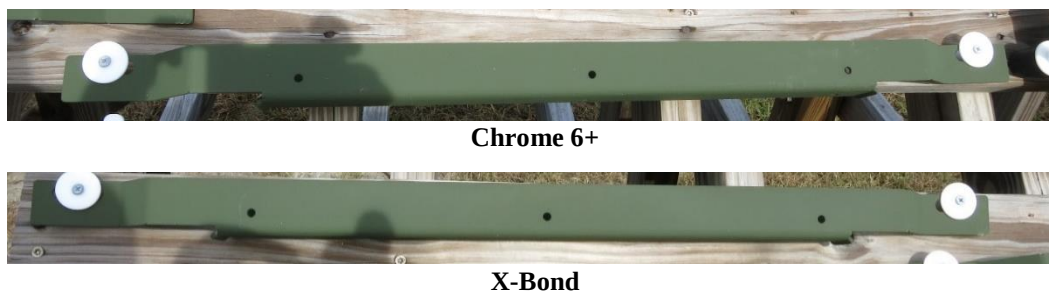


Fig. 41 CCAFS 24 months' exposure from December 2014 to December 2016 of aluminum brackets treated with chrome 3+ versus X-Bond 4000 coated with MIL-DTL-53022 primer and MIL-DTL-53039 topcoat at LEAD

At MDMC-PPA, aluminum parts treated with X-Bond 4000 and coated with MIL-DTL-53030 primer and MIL-DTL-64159 topcoat showed equal performance to chrome 3+ for face corrosion. No face blistering was observed with either pretreatment.



Fig. 42 CCAFS 24 months' exposure from December 2014 to December 2016 of aluminum HMMWV battery hold-down brackets treated with chrome 3+ vs. X-Bond 4000 coated with MIL-DTL-53030 primer and MIL-DTL-64159 topcoat

CRS D-ring lift hook surrounds treated with X-Bond 4000 and coated with MIL-DTL-53030 primer and MIL-DTL-64159 topcoat at MDMC-PPA showed slightly more corrosion than the zinc phosphate-treated parts. Red rust corrosion products were more evident along the edges of the D-rings treated with X-Bond than those treated with zinc phosphate.



Fig. 43 CCAFS 24 months' exposure from December 2014 to December 2016. CRS lift hook D-ring surrounds treated with X-Bond vs. zinc-phosphate and painted with MIL-DTL-53030 primer and MIL-DTL-64159 topcoat at MDMC-PPA.

6.3.12 Twelve Months' On-Vehicle Field Exposure at USMC Camp Lejeune

Parts and panels were mounted on vehicles at USMC Camp Lejeune. The vehicles included seven HMMWVs and five MTVRs. Panels were mounted on the vehicles by means of racks located on the lower hitch/frame areas at the rear of the vehicles (see Fig. 4). Panels were scribed per ASTM D1654 before mounting in order to measure scribe creep. In addition to the demonstration panels, steel weight loss panels were mounted to the vehicles to measure the corrosivity of the test environment. After 12 months in the field, some of these preweighed samples were removed, stripped of corrosion products via hydrochloric acid immersion, and reweighed. It was determined that the vehicles used for the demonstration would not immediately be placed into active service and tracked for use, but were designated to do so by June 2016. Therefore, a few representative samples were removed to evaluate the corrosion caused by sitting in lots at Camp Lejeune. The mass-loss coupons removed with the parts and panels and evaluated for corrosion products showed that the environment met the corrosion rate requirements of TT-C-490. These results were tabulated in Tables 54–58 and represented in Figs. 44–48. They illustrate that the environment to which the vehicles were subjected was not as corrosive as a typical seacoast environment. On-vehicle exposure continued to the 24-month specification requirement.

Table 54 Twelve months' on-vehicle exposure from December 2014 to December 2015 at Camp Lejeune of CRS blasted-only vs. blasted+ X-Bond vs. zinc phosphate under MIL-DTL-53022 primer and MIL-DTL-53039 topcoat from LEAD

Code	Location	Substrate	Pretreatment	Coating	Average scribe creep (scrapped) (mm)	Maximum scribe creep (mm)	Face corrosion	Status
C3	LEAD	CRS	Blasted-only	53022+ 53039	0.1	0.2	None	Pass
C6	LEAD	CRS	Blasted+ X-Bond	53022+ 53039	0.5	0.5	None	Pass/ Worse than zinc phosphate
C9	LEAD	CRS	Blasted+ Zinc Phosphate	53022+ 53039	0.1	0.1	None	Pass

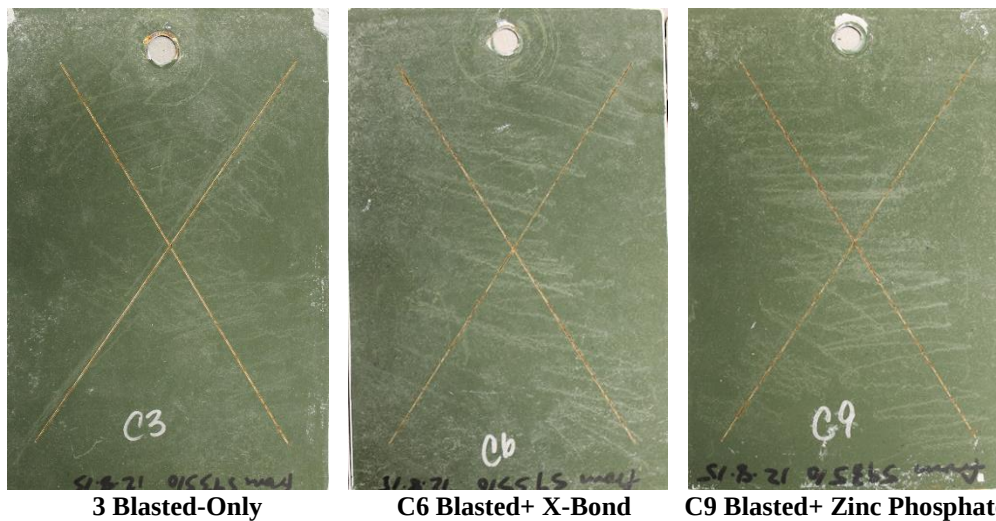


Fig. 44 Twelve months' on-vehicle exposure from December 2014 to December 2015 at Camp Lejeune of CRS blasted-only vs. blasted+ X-Bond vs. blasted+ zinc phosphate under MIL-DTL-53022 primer and MIL-DTL-53039 topcoat from LEAD

Table 55 Twelve months' on-vehicle exposure from December 2014 to December 2015 at Camp Lejeune of CRS blasted-only vs. blasted+ X-Bond vs. zinc phosphate under MIL-DTL-53030 primer and MIL-DTL-64159 topcoat from MDMC-PPA

Code	Location	Substrate	Pretreatment	Coating	Average scribe creep (scraped) (mm)	Maximum scribe creep (mm)	Face corrosion	Status
S4	MDMC-PPA	CRS	Blasted-only	53030+ 64159	0.1	0.6	None	Pass
S9	MDMC-PPA	CRS	Blasted+ X-Bond	53030+ 64159	0.1	0.1	None	Pass/ Better than zinc phosphate
S16	MDMC-PPA	CRS	Blasted+ Zinc Phosphate	53030+ 64159	0.4	0.5	None	Pass

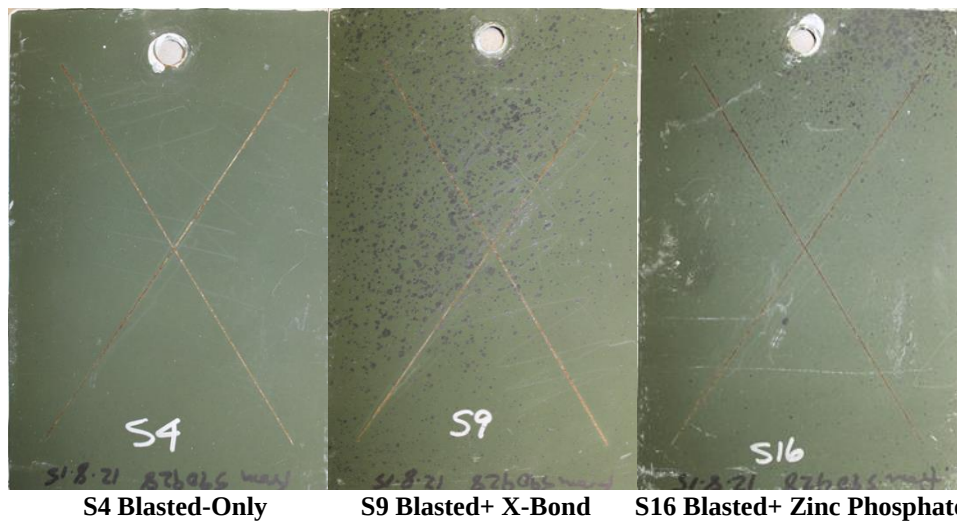


Fig. 45 Twelve months' on-vehicle exposure from December 2014 to December 2015 at Camp Lejeune of CRS blasted-only vs. blasted+ X-Bond vs. blasted+Zinc zinc phosphate under MIL-DTL-53030 primer and MIL-DTL-64159 topcoat from MDMC-PPA

Table 56 Twelve months' on-vehicle exposure from December 2014 to December 2015 at Camp Lejeune of aluminum treated with chrome 6+ vs. X-Bond under MIL-DTL- 53022 primer and MIL-DTL-53039 topcoat from LEAD

Code	Location	Substrate	Pretreatment	Coating	Average scribe creep (scraped) (mm)	Maximum scribe creep (mm)	Face corrosion	Status
A3	LEAD	AA2024T3 Aluminum	Chrome 6+	53022+ 53039	0	0.6	None	Pass
A6	LEAD	AA2024T3 Aluminum	X-Bond	53022+ 53039	0	0	None	Pass/ Better than Cr ⁶⁺

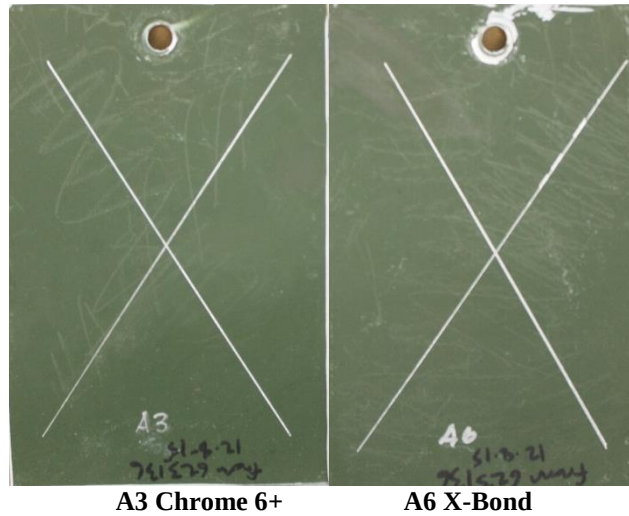


Fig. 46 Twelve months' on-vehicle exposure from December 2014 to December 2015 at Camp Lejeune of aluminum treated with X-Bond vs. chrome 6+ under MIL-DTL-53022 primer and MIL-DTL-53039 topcoat from LEAD

Table 57 Twelve months' on-vehicle exposure from December 2014 to December 2015 at Camp Lejeune of aluminum with X-Bond vs. chrome 3+ under MIL-DTL-53030 primer and MIL-DTL-64159 topcoat from MDMC-PPA

Code	Location	Substrate	Pretreatment	Coating	Average scribe creep (scraped) (mm)	Maximum scribe creep (mm)	Face corrosion	Status
M3	MDMC-PPA	2024T3 Aluminum	X-Bond	53030+ 64159	0	0	None	Pass/ Better than Cr ³⁺
M8	MDMC-PPA	2024T3 Aluminum	Chrome 3+	53030+ 64159	0.65	5.1	None	Pass

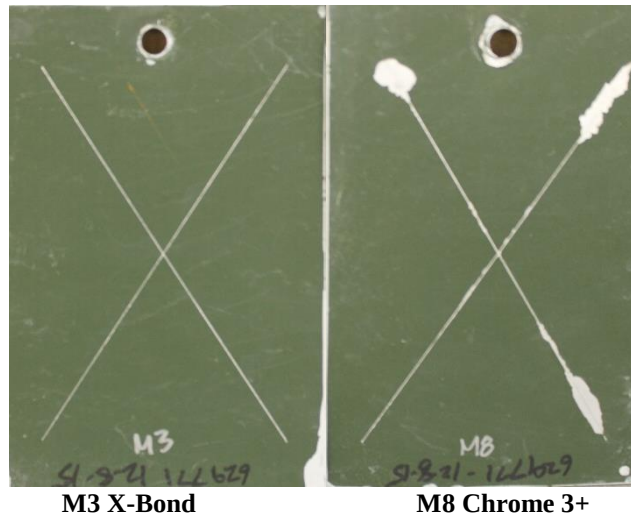


Fig. 47 Twelve months' on-vehicle exposure from December 2014 to December 2015 at Camp Lejeune of aluminum X-Bond vs. chrome 3+ under MIL-DTL-53030 primer and MIL-DTL-64159 topcoat from MDMC-PPA

Table 58 12 months' on-vehicle exposure from December 2014 to December 2015 at Camp Lejeune of aluminum with X-Bond vs. zinc phosphate under MIL-DTL-53030 primer and MIL-DTL-64159 topcoat from MDMC-PPA

Code	Location	Substrate	Pretreatment	Coating	Average scribe creep (scraped) (mm)	Maximum scribe creep (mm)	Face corrosion
S21	MDMC-PPA	CRS D-Ring Surround	Blasted+ Zinc Phosphate	53030+ 64159	0.1	0.3	None
S20	MDMC-PPA	CRS D-Ring Surround	Blasted+ X-Bond	53030+ 64159	0.1	0.5	None/ Equal to zinc phosphate

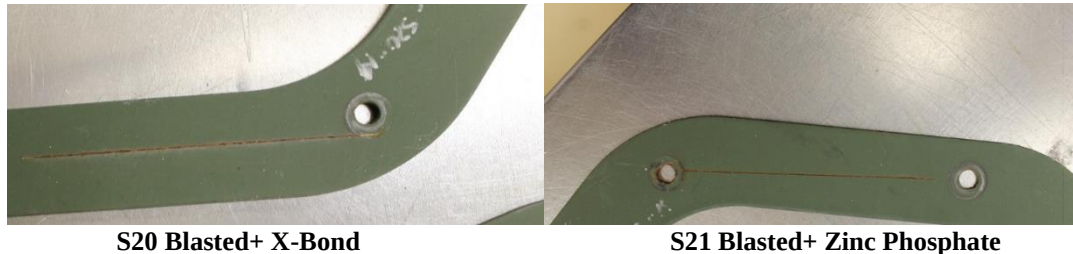


Fig. 48 Twelve months' on-vehicle exposure from December 2014 to December 2015 at Camp Lejeune of CRS D-ring lift hook surrounds blasted+ X-Bond vs. blasted+ zinc phosphate under MIL-DTL-53022 primer and MIL-DTL-53039 topcoat from LEAD

6.3.13 Corrosion Rate at Camp Lejeune

A process check was conducted to verify that the atmospheric conditions at Camp Lejeune met the requirement for corrosivity outlined in TT-C-490. CRS mass-loss coupons were mounted on the vehicles along with test panels. The coupons were removed at the same time as the panels and returned to PPG. The mass-loss coupons were cleaned of rust using a dilute hydrochloric acid solution. They were weighed, and the mass of lost material was determined by subtracting the stripped weight from the original weight. The corrosion rate was calculated using Equation 1.

$$\text{Corrosion Rate (CR)} = \frac{\text{Weight loss (g)} * K}{\text{Alloy Density (g/cm}^3) * \text{Exposed Area (A)} * \text{Exposure Time (hr)}} \quad (1)$$

The constant K is 22,300. The density of carbon steel is 7.85 gm/cm³. The exposed area is 4 in². The exposure time is one year. The results showed in Table 59 that the corrosion environment of the vehicle-mounted coupons was less corrosive than the

minimum of 1.5 mil per year (mpy), as specified in TT-C-490 for a seacoast environment.

Table 59 Corrosion rate from mass-loss coupons on inspected vehicles at Camp Lejeune after 12 months from December 2014 to December 2015

Vehicle	Serial number	Coupon1 weight loss (Gm/m ²)	Coupon2 weight loss (Gm/m ²)	Coupon3 weight loss (Gm/m ²)	Coupon4 weight loss (Gm/m ²)	Coupon5 weight loss (Gm/m ²)	Average weight loss (Gm/m ²)	Corrosion rate (mpy)
HMMWV	629771	0.218	0.359	0.218	0.195	0.225	0.243	0.472812
HMMWV	623136	0.200	0.272	0.200	0.284	N/A	0.239	0.465029
MTVR	593510	0.232	0.235	.233	N/A	N/A	0.234	0.455301
MTVR	590928	0.214	0.216	0.215	N/A	N/A	0.215	0.422223

7. Cost Assessment

The demonstrations conducted at LEAD and MDMC-PPA utilized X-Bond 4000 to pretreat both steel and aluminum parts. At both locations, separate pretreatments for steel and aluminum substrates are currently being used: LEAD uses Oakite Cryscoat OC zinc phosphate for steel and ChromiCoat L25 for aluminum, while MDMC-PPA uses Oakite Cryscoat CM for steel and Metalast TCP-HF for aluminum. The performance assessments conducted on the parts and panels from the demonstrations indicated that X-Bond 4000 pretreatment conforms to the requirements of TT-C-490 and MIL-DTL-5541, as detailed in Section 6.

The environmental benefits have also been detailed in Sections 1.2 and 2.3. First and foremost among these is that the replacement of current pretreatments for aluminum would remove a significant source of chrome from the depots, hexavalent chrome at LEAD and trivalent chrome at MDMC-PPA. A 2016 report to SERDP, titled *Cadmium and Hexavalent Chromium Alternatives Implementation Plan for Letterkenny Army Depot, Contract # W9128F12-D-0041*, indicates that using a non-chrome pretreatment, as X-Bond 4000, would eliminate 412 lb of chrome or 40% of the total chrome usage annually from operations at LEAD. Elimination of the zinc phosphate processes would remove nickel-containing sludge, another serious hazardous waste present in the current processes.

The combination of effective corrosion performance and significant reduction in environmental impact can be achieved with an overall cost reduction in the operation of the pretreatment process. This section presents the thought process for the cost assessment of utilizing a zirconium-based (zirconium) pretreatment at

LEAD. This utilizes the cost analysis information provided by LEAD in November 2017 and identifies needed information for completing the cost analysis.

8. Cost Analysis

The cost analysis performed is a return-on-investment (ROI) calculation, which is based on the methods used for Office of the Secretary of Defense (OSD) Corrosion Policy Office (CPO) funded projects. This analysis compares baseline and new system costs, considers projected improvements in performance of the new system, and utilizes a 7% discount rate to determine the present-value (PV) of the project savings over a period of 30 years after implementation. The positive savings is then shown as a ratio of the project costs (investment), which provides the ROI for the project.*

8.1 Capital Investments/Fixed One-time Costs

These costs include the upfront investment needed to implement the alternative pretreatment system at a facility. As the zirconium pretreatment is meant to be a drop-in replacement for the existing hexavalent, trivalent, and zinc-phosphate processes, upfront costs are expected to be minimal.

At LEAD, the only upfront cost listed was the addition of a new water system to produce DI water to support the zirconium pretreatment process. This was estimated as being the following:

- \$117,500
 - Equipment/material costs: \$81,500
 - Labor costs: \$36,000

The one-time upfront costs for adding the DI system of \$117,500 will be included in the cost analysis.

8.2 Operating Costs

Operating costs are the costs needed to operate the system. These include costs for chemicals, cleaning/hazmat disposal, utilities, and labor/operator costs.

For this project, there is no expected effect on the labor/operator costs. As a drop-in replacement, the zirconium pretreatment is expected to utilize the same

* An installation that wishes to implement this process would have a different ROI, as their investment would be the ratio of future savings (in present dollars) to the initial capital costs of equipment or training needed to implement the new process; they do not include the project costs.

equipment, personnel, and nominally the same process times as the existing hexavalent chromium and zinc-phosphate pretreatments. Therefore, the net change to labor costs is expected to be \$0 and will not be included in this analysis. Each of the other costs is discussed herein.

8.3 Annual Chemical Costs

LEAD utilized their pretreatment processes performed in Building 350 as an example for this cost analysis. Table 60 shows their estimated annual costs for these chemicals; this will be included in the baseline annual costs.

Table 60 LEAD estimated annual chemical costs current pretreatment

Chemical	Annual usage (drums)	Unit drum cost	Annual cost
Oakite 31	18	\$2,349.60	\$42,292.80
Deoxidizer LNC	5	\$1,492.70	\$7,463.50
Chromicoat L25	3	\$2,440.35	\$7,321.05
Chromicoat T3	1	\$3,138.85	\$3,138.85
Cryscosat OC	1	\$2,210.76	\$2,210.76
Oakite FH3	1	\$602.24	\$602.24
TOTAL ANNUAL COSTS			\$63,029.20

Implementation of the zirconium process at LEAD for both aluminum and steel substrates would allow for elimination of the hexavalent chromium (Chromicoat L25 and Chromicoat T3), zinc-phosphate (Cryscosat OC), and acid rinse for zinc-phosphate (Oakite FH3). These would be replaced by the zirconium pretreatment. Table 61 shows the estimated annual chemical costs for implementation of the zirconium pretreatment at LEAD in Building 350.

Table 61 LEAD estimated annual chemical costs zirconium pretreatment

Chemical	Annual usage (drums)	Unit drum cost	Annual cost
Oakite 31	18	\$2,349.60	\$42,292.80
Deoxidizer LNC	5	\$1,492.70	\$7,463.50
Zirconium [†]	1	\$3,138.85	\$3,138.85
TOTAL ANNUAL COSTS			\$ 52,895.15

[†] This is estimated for the pretreatment of aluminum (X drums) and steel (Y drums) parts.

On an annual basis, the cost of chemicals for implementing zirconium is estimated to be lower than the current pretreatment processes. This results in an annual savings of \$10,134.05. This will be included in the annual operating costs.

8.4 Annual Cleaning Costs

Utilizing the information provided by LEAD for Building 350 processes, the estimated annual cleaning costs are shown in Table 62.

Table 62 LEAD estimated annual cleaning costs current pretreatment

Chemical	Annual tank cleanings	Unit cost	Annual cost
Oakite 31	2	\$12,646.29	\$25,292.57
Deoxidizer LNC	1	\$10,984.99	\$10,984.99
Chromicoat L25	1	\$11,122.18	\$11,122.18
Cryscoat OC	1	\$3,131.11	\$3,131.11
Oakite FH3	4	\$3,488.89	\$13,955.56
TOTAL ANNUAL COSTS			\$64,486.41

The use of the zirconium pretreatment has an immediate savings on the cleaning costs of nearly \$14,000 a year, as this process eliminates the need for the Oakite FH3 acid rinse step for the pretreatment of steel substrates. The consolidation of the Chromicoat L25 (hexavalent chromium) and Cryscoat OC (zinc-phosphate) processes into a single line is expected to require only a single annual cleaning of the tank. Table 63 shows the estimated annual cleaning costs for the zirconium pretreatment process

Table 63 LEAD estimated annual cleaning costs zirconium pretreatment

Chemical	Annual tank cleanings	Unit cost	Annual cost
Oakite 31	2	\$12,646.29	\$25,292.57
Deoxidizer LNC	1	\$10,984.99	\$10,984.99
Zirconium	1	\$3,131.11	\$3,131.11
TOTAL ANNUAL COSTS			\$ 39,408.67

On an annual basis, the cost of cleaning associated with implementing zirconium is estimated to be lower than the current pretreatment processes. This results in an annual savings of \$25,077.74. This will be included in the annual operating costs.

8.5 Annual Utility Costs

LEAD provided an estimated cost for heat required for their current and zirconium pretreatment operations. Their current process, which uses steam to provide heat, is expected to require 756,500 BTU an hour heat load, which results in a cost of \$16.87 an hour. The zirconium process is expected to require 166,800 BTU an hour heat load, which results in a cost of \$3.72 an hour. The difference in hourly costs results in a savings of \$13.15 an hour (78%).

LEAD was not able to provide a specific number of operating hours for their pretreatment operations. Therefore, an assumption was made that at maximum capacity, the pretreatment operations would run on two 8-h shifts per day, 5 days per week (excluding federal holidays), and that heat would be applied continuously during the work-shift, regardless if active pretreatment operations were being conducted. This was expected to apply to both the current and zirconium pretreatment operations; therefore the estimated cost savings was calculated as follows:

- \$52,600 annual savings in heating costs
 - 250 work days per year $\times$ 16 operational hours = 4,000 operating hours per year
 - Baseline hourly cost of \$16.87 $\times$ 4,000 operating hours per year = \$67,480 per year
 - Zirconium hourly cost of \$3.72 $\times$ 4,000 operating hours per year = \$14,880 per year
 - Savings of \$67,480 – 14,880 = \$52,600 per year

The annual savings of \$52,600 will be included in the annual operating costs.

8.6 Estimated Performance Benefits

The performance of the zirconium pretreatment showed that for aluminum and steel substrates there is either an equivalent, or slightly improved performance as compared to other pretreatments. Without a marked improvement over the current system, it is unlikely that the use of the zirconium pretreatment will result in a change in the frequency (positive or negative) of the repainting of assets. Therefore, there is expected to be no impact on repainting costs associated with using this pretreatment process.

8.7 ROI Analysis

Utilizing the information provided by LEAD and the estimated costs for the zirconium pretreatment, the following are being used for developing the ROI for this:

- Fixed/one-time costs
 - Baseline: \$0
 - Zirconium: \$117,500
- Annual operating costs
 - Baseline: \$194,995.61
 - Zirconium: \$107,183.82
- Performance benefits
 - Baseline: \$0
 - Zirconium: \$0

The fixed/one-time costs are added to Year 1 of the ROI table, with the annual costs included for all future years. The cost of the project is used as the investment made on evaluating zirconium for use in a depot setting. Table 64 shows the results of this ROI analysis, if the process was only implemented at LEAD. Assuming similar implementation and operational costs at MDMC PPA, a commensurately higher ROI is estimated (Table 65).

Table 64 Estimated ROI for implementation at LEAD only

Return on Investment Calculation							
				Investment Required			500,000
				Return on Investment Ratio	1.96	Percent	196%
				Net Present Value of Costs and Benefits/Savings	1,439,827	2,419,642	979,815
A	B	C	D	E	F	G	H
Future Year	Baseline Costs	Baseline Benefits/Savings	New System Costs	New System Benefits/Savings	Present Value of Costs	Present Value of Savings	Total Present Value
1	194,996		224,684		209,989	182,243	-27,747
2	194,996		107,184		93,614	170,309	76,695
3	194,996		107,184		87,494	159,175	71,681
4	194,996		107,184		81,771	148,762	66,992
5	194,996		107,184		76,422	139,032	62,610
6	194,996		107,184		71,417	129,926	58,509
7	194,996		107,184		66,743	121,424	54,680
8	194,996		107,184		62,381	113,487	51,106
9	194,996		107,184		58,297	106,058	47,761
10	194,996		107,184		54,482	99,116	44,635
11	194,996		107,184		50,923	92,642	41,719
12	194,996		107,184		47,590	86,578	38,988
13	194,996		107,184		44,481	80,923	36,442
14	194,996		107,184		41,566	75,619	34,053
15	194,996		107,184		38,843	70,666	31,823
16	194,996		107,184		36,303	66,045	29,742
17	194,996		107,184		33,934	61,736	27,801
18	194,996		107,184		31,716	57,699	25,984
19	194,996		107,184		29,636	53,916	24,280
20	194,996		107,184		27,696	50,387	22,691
21	194,996		107,184		25,885	47,091	21,207
22	194,996		107,184		24,191	44,011	19,819
23	194,996		107,184		22,605	41,125	18,520
24	194,996		107,184		21,126	38,434	17,308
25	194,996		107,184		19,743	35,918	16,175
26	194,996		107,184		18,457	33,578	15,121
27	194,996		107,184		17,246	31,375	14,129
28	194,996		107,184		16,120	29,327	13,207
29	194,996		107,184		15,070	27,416	12,346
30	194,996		107,184		14,084	25,622	11,538

Table 65 Estimated ROI for implementation at LEAD and MDMC PPA only

Return on Investment Calculation							
				Investment Required			500,000
				Return on Investment Ratio	3.92	Percent	392%
				Net Present Value of Costs and Benefits/Savings	2,879,655	4,839,284	1,959,629
A	B	C	D	E	F	G	H
Future Year	Baseline Costs	Baseline Benefits/Savings	New System Costs	New System Benefits/Savings	Present Value of Costs	Present Value of Savings	Total Present Value
1	389,991		449,368		419,979	364,486	-55,493
2	389,991		214,368		187,229	340,618	153,390
3	389,991		214,368		174,988	318,350	143,362
4	389,991		214,368		163,541	297,524	133,983
5	389,991		214,368		152,844	278,064	125,220
6	389,991		214,368		142,833	259,851	117,018
7	389,991		214,368		133,487	242,848	109,361
8	389,991		214,368		124,762	226,975	102,213
9	389,991		214,368		116,595	212,116	95,522
10	389,991		214,368		108,963	198,233	89,269
11	389,991		214,368		101,846	185,285	83,439
12	389,991		214,368		95,179	173,156	77,977
13	389,991		214,368		88,963	161,846	72,884
14	389,991		214,368		83,132	151,239	68,107
15	389,991		214,368		77,687	141,333	63,646
16	389,991		214,368		72,606	132,090	59,484
17	389,991		214,368		67,869	123,471	55,602
18	389,991		214,368		63,431	115,398	51,967
19	389,991		214,368		59,273	107,833	48,560
20	389,991		214,368		55,393	100,774	45,381
21	389,991		214,368		51,770	94,183	42,413
22	389,991		214,368		48,383	88,021	39,638
23	389,991		214,368		45,210	82,249	37,039
24	389,991		214,368		42,252	76,867	34,615
25	389,991		214,368		39,487	71,836	32,350
26	389,991		214,368		36,914	67,156	30,242
27	389,991		214,368		34,492	62,750	28,258
28	389,991		214,368		32,241	58,655	26,414
29	389,991		214,368		30,140	54,833	24,693
30	389,991		214,368		28,168	51,245	23,077

Excluding the one-time capital cost for a DI water system (\$117,500), the implementation of the zirconium pretreatment was estimated to have an annual savings of \$87,811.79. As shown in Table 64, this is expected to have a minimum estimated ROI of 2:1 if the project is only implemented at LEAD. Table 65 shows that by expanding to MDMC PPA (and assuming similar implementation costs), the ROI is increased to 4:1. Therefore, implementation at additional locations (e.g., Red River Army Depot, Anniston Army Depot, etc.) are expected to result in a higher ROI.

Conservatively, the ROI (over a 30-year period) is estimated at 2:1, but could be as high as 8:1 if implemented at four depots.

9. Implementation Issues and Conclusion

The results of the demonstrations to date show that zirconium pretreatment X-Bond 4000 meets the performance criteria of a TT-C-490 Type IV Inorganic Pretreatment. Outdoor exposure testing will continue until 18 months of exposure for parts and panels is reached at Camp Lejeune on vehicles and at Cape Canaveral on parts. This technology has been approved by the US Army and included in the Qualified Products List for TT-C-490 in Fiscal Year 2018.

Since the initiation of this program, changes to MIL-DTL-5541 are being pursued by the US Naval Air Systems Command (NAVAIR), the custodian of this specification. The specification at present calls for salt spray corrosion testing of treated-only (i.e., unpainted) aluminum surfaces. Currently, there are no chrome-free conversion coatings that meet this requirement. As a result, NAVAIR plans to drop the treated-only corrosion requirement; investigations are being conducted into the addition of a galvanic corrosion requirement as a replacement. This requirement would call for testing an assembly prepared by treating and priming a 3- × 6-inch plate of 2024T3 aluminum. The plate will have eight predrilled holes and will be primed with a chrome-free, aerospace primer. One half of the plate will be coated with a CARC topcoat. After scribing across the holes, stainless steel and titanium fasteners will be installed in the holes, and the assembly will be placed into GMW 14872 cyclic corrosion testing for 40 cycles. After exposure, the fasteners will be removed and the plate examined for corrosion, especially around the fasteners. No criteria for acceptable performance have been established.

Testing of X-Bond 4000 according to this potential requirement shows corrosion around the stainless steel fastener that is worse than with chrome 6+ and approximately the same as chrome 3+. The use of an acidic deoxidizing step, developed after the demonstrations, has shown improved galvanic corrosion results. Therefore, any aluminum treatment that needs to meet MIL-DTL-5541 will require a deoxidizer step.

As stated in Section 7, the switch from zinc phosphate to zirconium pretreatment is a simple transition. Metal surfaces must be cleaned of oil and corrosion before application of the zirconium pretreatment and subsequent paint layers. This is accomplished by existing grit-blasting practices. Commercially available alkaline cleaners are also used to remove oil and dirt. The zirconium can be applied using the same tanks and spray systems already in place for chromium and zinc phosphate; however, thorough cleaning and rinsing of the tanks prior to zirconium treatment is required. The treatment suppliers can provide guidance for this process.

Process control is accomplished by monitoring pH, zirconium content, and iron content. Automated process controls can be used where high throughput necessitates frequent adjustment.

The introduction of zirconium presents minimal risk, as the proposed zirconium products are already being manufactured and supplied in commercial quantities. Proper control of the zirconium process will ensure consistent quality. When using zirconium pretreatments to coat multiple substrates in the same process, compatibility in the X-Bond process should be checked. The control required is well within the capability of personnel currently applying zinc phosphate or chrome-based treatments.

10. References

- ASTM B117. Standard practice for operating salt spray (fog) apparatus. West Conshohocken (PA): ASTM International; 2007.
- ASTM D522. Standard test methods for mandrel bend test of attached organic coatings. West Conshohocken (PA): ASTM International; 2008.
- ASTM D714. Standard test method for evaluating degree of blistering of paints. West Conshohocken (PA): ASTM International; 2009.
- ASTM D1654. Standard test method for evaluation of painted or coated specimens subjected to corrosive environments. West Conshohocken (PA): ASTM International; 2008.
- ASTM D3359. Standard test methods for measuring adhesion by tape test. West Conshohocken (PA): ASTM International; 2009.
- ASTM D3363. Standard test method for film hardness by pencil test. West Conshohocken (PA): ASTM International; 2011.
- ASTM G1. Standard practice for preparing, cleaning, and evaluation corrosion test specimens. West Conshohocken (PA): ASTM International; 1999.
- ASTM G50. Standard practice for conducting atmospheric corrosion tests on metals. West Conshohocken (PA): ASTM International; 2010.
- ASTM F1624. Standard test method for measurement of hydrogen embrittlement threshold in steel by the incremental step loading technique. West Conshohocken (PA): ASTM International; 1995.
- FED-STD-141. Paint, varnish, lacquer and related materials; methods of inspection sampling and testing; 1986.
- Silvernail N. Environmentally friendly zirconium oxide pretreatment, project WP-1676. Alexandria (VA): Environmental Security Technology Certification Program (ESTCP); 2013.
- GMW 14872. Cyclic corrosion laboratory test. Warren (MI): General Motors International; 2010.
- MIL-DTL-53022. Primer, epoxy coating, corrosion inhibiting, lead and chromate free. Falls Church (VA): Defense Quality and Standardization Office; 2012.
- MIL-DTL-53030C. Primer Coating, Epoxy, Water Base, Lead and Chromate Free. Falls Church (VA): Defense Quality and Standardization Office; 2012.

- MIL-DTL-53072. Chemical agent resistant coating (CARC) system application procedures and quality control. Falls Church (VA): Defense Quality and Standardization Office; 2013.
- MIL-DTL-5541. Chemical conversion coatings on aluminum and aluminum alloys. Falls Church (VA): Defense Quality and Standardization Office; 2009.
- Occupational Safety and Health Administration (OSHA) Final Rules. Washington (DC): Occupational Safety and Health Administration; 2006 May 30. Federal Register No.: 71:10099-10385.
- SSPC-VIS-2. Standard method of evaluating degree of rusting on painted steel surfaces. Pittsburgh (PA): Society for Protective Coatings; 2000.
- TT-C-490. Chemical conversion coatings and pretreatments for metallic substrates (base for organic coatings); 1961.

Appendix A. Participating Organizations and Representatives

Table A-1 Participating organizations and representatives

Organization	Representative	Type
CCDC Army Research Laboratory (ARL)	Fred Lafferman (410) 306-1520	Government
Army Aviation and Missile Command (AMCOM)	Mark Feathers (256) 842-7355	Government
Army Tank and Automotive Command (TACOM)	Daniel Nymberg (586) 282-7445	Government
Naval Surface Warfare Center - Carderock Division (NSWCCD)	Andrew Sheetz (301) 227-5037	Government
Letterkenny Army Depot (LEAD)	Dennis Reed (717) 267-8376	Government
Albany Marine Corps Logistics Base	Steve Allen (229) 639-5310	Government
CCDC Army Research Laboratory (ARL)	Daniel Pope (410) 306-1688	Government
CCDC Army Research Laboratory (ARL)	John Escarsega (410) 306-0693	Government
PPG Industries	Larry Fitzgerald (412) 492-5396	Contractor
PPG Industries	Jonathan Love (412) 492-5206	Contractor
PPG Industries	Jeffrey Stalker (412) 492-5206	Contractor

Appendix B. Method for Measuring Scribe Creep after Corrosion Testing

Panels and parts tested for scribe corrosion were tested according to *Evaluation of Painted or Coated Specimens Subjected to Corrosive Environments* (ASTM D1654 2008). Prior to exposure, an “X” scribe was cut across the face of the panel using a carbide-tipped pencil scribe, per the test method. The scribe cut through the coatings to the substrate, leaving bare metal exposed. The average width of the scribes was measured as 0.2 mm. Bare edges of the panels were protected by coating them with PPG Industry’s 97-680 White Multiprime Air-dry Primer.

After preparing the panels, they were placed in the prescribed test environment. Following exposure, the panels were rinsed with deionized water to remove salt and dirt. Panels in salt spray or cyclic corrosion were scraped across the face of the panel and across the scribe with a scalpel under running tap water to remove any loose coating.

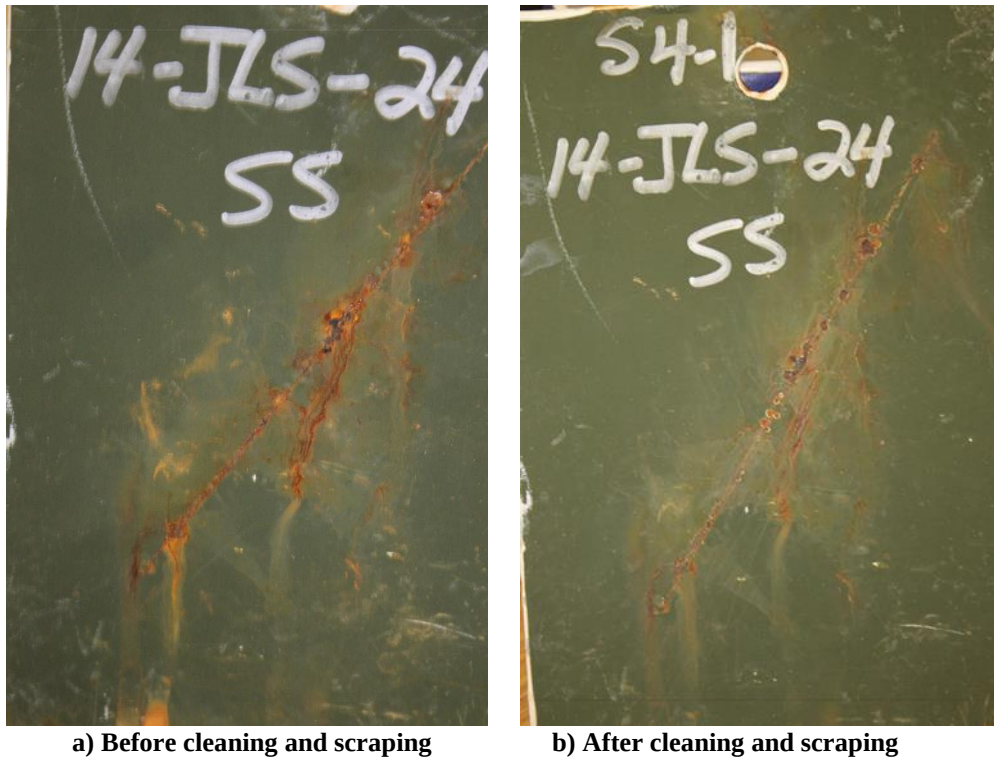
The panels with the X-scribe were evaluated for scribe corrosion by taking six measurements of creep using digital calipers. The photographs in Fig. A-1 show a single scribed panel before and after cleaning and scraping and the orientation of the jaws of the calipers during measurement. (An X-scribed panel was not available to show before and after, so a single-scribed panel was photographed.)

The scribe creep, C, was then calculated using the following equation:

$$C = \frac{w_c - w}{2}$$

where w_c = measured width of the corrosion zone and w = the width of the original scribe.

The scribe creep value for a particular panel was the average of six random readings. The maximum scribe creep was measured using the calipers at the point of the scribe lines that showed the greatest amount of adhesion loss (see Fig. B–1).



a) Before cleaning and scraping

b) After cleaning and scraping



Fig. B-1 Measuring scribe creep with calipers

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**Appendix C. Report on Results of Hydrogen Embrittlement
Testing of Notched Bars Treated and Treated/Primed at
Letterkenny Army Depot**

This appendix appears in its original form, without editorial change.

TEST REPORT -13299**Assured Testing Services**

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Test Report#: 13299 Entry Date: Tuesday, August 19, 2014 Contact: Jeff Stalker PO#: CC J

Stalker

Embrittlement Testing IAW ASTM F519.

V notch 1a.1 bars were loaded at 75% nominal notch tensile strength.

Notch Tensile Strength - 8888 pounds.

Part Test Information

PART NUMBER	LOT NUMBER	S#	SAMPLE DESCRIPTION					
TEST SPECIFICATION NAME		TEST PROCEDURE NAME			HOURS	CYCLES		
TEST RESULTS		INDATE	OUTDATE	TESTCNT	RETCNT	REQ	COMP	REQ
1a.1 Notch Bars Set #1								
Unspecified		Hydrogen Embrittlement ASTM F519-1 experimental m						
25-Aug-14 Results: 3 of 3 PASS with no fracture		02-Sep-14	3	0	200	200		
ritting if none of the plated/coated specimen								
1a.1 Notch Bars Set #2		Treated and painted						
Unspecified		Hydrogen Embrittlement ASTM F519-13						
25-Aug-14 Results: 3 of 3 PASS with no fracture.		02-Sep-14	3	0	200	200		
Requirements: A plating/coating process shall be considered non-embrittling if none of the plated/coated specimens fracture within 200 hours after loading.								

Michele Singer

Revision# 1.10 Date 12/16/10

End Of Report

Quality Assurance Administrator

The results stated above relate only to the specific items tested. Information and statements in this report are derived from material, information, and/or specifications furnished by the client and exclude any expressed or implied warranties as to the fitness of material tested or analyzed for any particular purpose or use. This report is confidential property of our client and may not be used for advertising purposes. This report shall not be reproduced except in full, without written approval of this

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List of Symbols, Abbreviations, and Acronyms

AISI	American Iron and Steel Institute
AMCOM	Army Aviation and Missile Command
ANSI	American National Standards Institute
ARL	Army Research Laboratory
ASTM	American Society for Testing and Materials
BP	best performance
BTU	British Thermal Unit
CARC	Chemical Agent Resistant Coating
CCAFS	Cape Canaveral Air Force Station
CCDC	US Army Combat Capabilities Development Command
CPAC	Corrosion Prevention and Control
CRS	cold-rolled steel
DI	deionized
DOD	Department of Defense
EAC	environmentally assisted cracking
ESOH	Environment, Safety and Occupational Health
ESTCP	Environmental Security Technology Certification Program
FZA	fluorozirconic acid
GMW	General Motors Worldwide
GSE	Ground Support Equipment
HAP	Hazardous Air Pollutant
HAZMAT	Hazardous Material
HHA	High Hard Armor
HSLA	High-Strength Low-Alloy
HMMWV	High Mobility Multi-Purpose Wheeled Vehicle
IP	improved performance

JTP	Joint Test Protocol
JTR	Joint Test Report
LEAD	Letterkenny Army Depot
MDMC-PPA	Marine Depot Maintenance Command – Production Plant Albany
MMC	Metal-Matrix Composite
MTVR	Medium Tactical Vehicle Replacement
MP	minimum performance
NAVAIR	US Naval Air Systems Command
NSS	Neutral Salt Spray
OEM	Original Equipment Manufacturer
OSHA	Occupational Safety and Health Administration
PPG	PPG Industries, Inc.
SEM	scanning electron microscope
SERDP	Strategic Environmental Research and Development Program
SSPC	Society for Protective Coatings
TACOM	US Army Tank-Automotive and Armaments Command
TARDEC	US Army Tank Automotive Research, Development and Engineering Center
TRL	Technology Readiness Level
TWA	time-weighted average
USMC	US Marine Corps
VOC	Volatile Organic Compound

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