ESTCP
Cost and Performance Report

(WP-200906)

Non-Chromate, ZVOC Coatings for Steel Substrates on Army and Navy Aircraft and Ground Vehicles

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ESTCP

ENVIRONMENTAL SECURITY
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COST & PERFORMANCE REPORT
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ACRONYMS AND ABBREVIATIONS

ANAD	Anniston Army Depot
AP	Armor Piercing
ARL	Army Research Laboratory
ASTM	American Society for Testing and Materials International
CARC	Chemical Agent Resistant Coating
CCPE	Corrosion Control and Prevention Executive
CHPPM	Center for Health Promotion and Preventive Medicine
CPAC	corrosion prevention and control
DI	deionized
DM	depot maintenance
DoD	U.S. Department of Defense
DPW	Department of Public Works
DRCF	Depot Repair Cycle Float
EAC	environmentally assisted cracking
ESTCP	Environmental Security Technology Certification Program
FY	fiscal year
GDLS	General Dynamics Land Systems
HAP	hazardous air pollutant
HATE	Hydraulic Adhesion Test Equipment
HHA	High Hard Armor
IAW	In Accordance With
IR	infrared
JBLM	Joint Base Lewis-McChord
JPO	Joint Program Office
JTP	Joint Test Protocol
L-T	longitudinal-transverse
MRAP	Mine Resistant Ambush Protected
NAVAIR	Naval Air Warfare Center
OEM	original equipment manufacturer
OSHA	Occupational Safety and Health Administration
PADDS	Procurement Automated Data and Document System
PDM	Programmed Depot Maintenance

PEL	permissible exposure limit
PEO	Program Executive Office
PEP	power entry panel
PM	?? pg vii
PMO	Program Managers Office
POC	point of contact
PPG	Pittsburgh Plate Glass Industries
PSI	pounds per square inch
QPD	Qualified Products Database
RH	Relative Humidity
RTU	ready to use
SAE	Society of Automotive Engineers
SBCT	Stryker Brigade Combat Team
SCC	Stress Corrosion Cracking
SERDP	Strategic Environmental Research and Development Program
SP	Surface Profile
SPC	Society for Protective Coatings
SSPC	Steel Structures Painting Council
TACOM	Tactical Army Command
TCP	Trivalent Chrome Pretreatment
TDS	Technical datasheet
TWA	time-weighted average
USMC	U.S. Marine Corps
UV	ultraviolet
VIN	Vehicle Identification Number
VOC	volatile organic compound

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EXECUTIVE SUMMARY

OBJECTIVES OF THE DEMONSTRATION

The objective of this demonstration is to determine the viability of non-chromate pretreatments for High Hard Armor (HHA) steel in order to improve the long term corrosion resistance of the low-volatile organic compound (VOC) chemical agent resistant coating (CARC) system to reduce lifecycle costs for these weapon systems. Stryker and Mine Resistant Ambush Protected Armored Vehicle (MRAP) contracts are prohibited from using hex-chrome and have been coated without any corrosion inhibitive pretreatment or conversion coating. The products demonstrated satisfy the hexavalent chrome prohibition for both vehicles while minimizing environmental impact.

TECHNOLOGY DESCRIPTION

These technologies are spray-applied pretreatments meant to be used on properly prepared steel substrates. These technologies include trivalent chromium (SurTec 650) and two non-chromium pretreatments (Chemetall Oxsilan 9810/2, and Pittsburgh Plate Glass Industries [PPG] Zircobond 4200). Trivalent chrome pretreatments have been used successfully on aluminum substrates for many years. Oxsilan 9810/2 is a phosphorus free liquid, silane-based product that is intended to enhance the performance of organic coatings. As the film dries, neighboring hydroxyl groups react with each other to form a dense, interpenetrating, crosslinked network that is chemically bound to the metal surface. Zircobond 4200 is a zirconate based pretreatment developed as a replacement for zinc phosphate. All of these pretreatments are commercially available.

DEMONSTRATION RESULTS

Three pretreatment candidates (Oxsilan 9810/2, SurTec 650, and Zircobond 4200) were laboratory validated on HHA with two different organic coating systems (MIL-DTL-53022/MIL-DTL-53039, MIL-DTL-53022/MIL-DTL-64159), and demonstrated on a variety of platforms and components. Flash rust inhibition was assessed using a modified version of ASTM International (ASTM) D 1735. Flash rust inhibitors, such as the Cheminhib 420, are used on both Stryker and MRAP to prevent corrosion prior to painting. Two of the alternatives, Oxsilan 9810/2 and SurTec 650, protected the HHA from flash rust significantly better than baseline 1% Cheminhib 420 through 48 hours in the humidity chamber.

All three of the pretreatment candidates met the adhesion requirements. The SurTec 650 dry tape adhesion ASTM-3359 ratings were slightly lower than the Cheminhib baseline, but still outperformed the standard, DoD-P-15328 washprimer. All three of the alternatives provided adhesion as good as, or better, than the baseline in wet tape adhesion tests, again outperforming the washprimer. Chip resistance was assessed using Society of Automotive Engineers (SAE) J400. Of the alternatives, only the SurTec 650 did not meet the minimum performance objective of a 5B rating with MIL-DTL-53022/MIL-DTL-64159, however, it exceeded the minimum with a 5A rating with MIL-DTL-53022/MIL-DTL-53039. Of note is that the DoD-P-15328 chromated washprimer did not meet the performance objectives with either coating system for chip resistance.

In the accelerated corrosion tests (ASTM B117, GM 9540P), all three alternatives significantly outperformed the baseline Cheminhib 420 and passed the all performance objectives. Perhaps more unexpected was that the alternatives were the only pretreatments tested that passed the performance objectives, and outperformed both the baseline Cheminhib and the DoD-P-15328 chromated washprimer.

The performance objective for static outdoor exposure tests are 25% less creepage from the scribe than the Cheminhib 420 baseline. This will not be evaluated until after 3 years of outdoor exposure has been completed. However, inspections were conducted at 2 years exposure and the trend appears to be correlating with the accelerated corrosion results.

It was important to determine if any of the proposed pretreatments would have a detrimental effect on the HHA resistance to environmentally assisted cracking. When the empirical data for K1EAC is compared with the control and that found in the literature, it is clear that none of the alternatives had any influence on the MIL-DTL-46100 resistance to environmentally assisted cracking.

There were two demonstrations carried out on HHA platforms: the first was on Strykers at Anniston Army Depot (ANAD); the second on MRAP at Camp Lejeune; and third a follow up demonstration on a set of MRAP doors at Aberdeen. All three alternatives were used on Stryker while only the Oxsilan 9810/2 was selected for demonstration on MRAPs at Camp Lejeune because it contained no chromium in the chemistry. The three Strykers had three hatches each pretreated with an alternative pretreatment. Of the three Strykers, two were accessible for field inspection. All of the hatches on both vehicles looked in good condition with no noticeable paint delamination or significant corrosion after 2 years and 7 months in service when compared to the base vehicle.

The MRAP treated with Oxsilan unexpectedly turned reddish-brown in color during application. Subsequent testing indicated that parameter adjustments for scale-up from laboratory sized and smaller hatch sized parts to a full sized MRAP were underestimated, resulting in inadequate flow of the applied Oxsilan 9810/2. We also believe that because the MRAP was abrasive blasted 3 days prior, the 72 hours the bare surface was exposed to the environment led to some surface contamination that likely affected the reaction of the Oxsilan with the steel surface. A subsequent demonstration was arranged using two rear MRAP doors. In this limited demonstration, the Oxsilan was successfully applied using adequate flow rate within 2-4 hours of abrasive blasting. The doors were primed and painted and have been in static outdoor exposure for 13 months with no noticeable corrosion damage. These demonstrations revealed that when properly applied to HHA, the SurTec 650, Oxsilan 9810/2, and Zircobond 4200 provide very good adhesion for the subsequent primer and topcoat. These pretreatments also performed better in all of the laboratory corrosion tests and 2 years in outdoor exposure than the baseline product Cheminhib 420. The full scale testing illustrates that the surface condition and application rate of the pretreatment must be diligently controlled similar to any other pretreatment process including the legacy phosphate conversion coating for steel, TT-C-490 Type I.

IMPLEMENTATION

The synergy of this project and the revision of Federal Specification TT-C-490 has provided a pathway for the implementation of these and other new pretreatment technologies. All three pretreatments evaluated in WP200906 met the minimum requirements of TT-C-490, Revision F and were assigned a qualified products database (QPD) number making them available to any Original Equipment Manufacturer (OEM), or Depot, for use on abrasive blasted steel. This is especially useful on contracts issued that must be free of hexavalent chromium.

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1.0 INTRODUCTION

1.1 BACKGROUND

The high hard armor steels (HHA) that are used on Strykers, Mine Resistant Ambush Protected (MRAP) armored vehicles, and a wide range of other systems provide good protection against armor piercing (AP) threats. However, these steels corrode rapidly without good corrosion protective coatings. HHA is also susceptible to structural damage from environmentally assisted cracking (EAC) whenever residual stresses are present, especially when inferior plate cutting and welding procedures are used. Recently, significant corrosion has been observed on newly fabricated, unfielded MRAP vehicles. While some may dismiss this rust as merely cosmetic corrosion, the reality is that such corrosion on military ground vehicles increases the infrared (IR) signal from the vehicle that the topcoat camouflage is designed to inhibit, making the vehicle more vulnerable to detection by the enemy.

Moreover, corrosion abatement costs the Department of Defense (DoD) $22.5B annually, with more than 25% of depot maintenance (DM) costs attributed to corrosion of Army ground vehicles.[1] Many of the coatings and pretreatments that the Army currently uses to mitigate corrosion contain toxic heavy metals, volatile organic compounds (VOC), and hazardous air pollutants (HAP). For the wash primer pretreatment process alone, the Army uses an annual average of 400,000 gallons of the DoD-P-15328 wash primer that generates 2.4 million pounds of VOC; 852,000 pounds of HAPs; and 24,000 pounds of hexavalent chrome. Although effective at mitigating corrosion for many years, products such as DoD-P-15328 have been targeted for elimination because they are risks to human health and the environment. A coating exception/waiver was granted to the Stryker original equipment manufacturer (OEM) to allow the omission of a pretreatment/conversion coating step. Permission was also extended to MRAP OEMs to omit pretreatments on that platform allowing the primer to be directly applied to the high hard steel substrate prior to topcoating using only a flash rust inhibitor the buy time between surface prep and painting. The original reasons that justified skipping this pretreatment/conversion coating step were: 1) hexavalent chromium based pretreatments such as DoD-P-15328[2] wash primer were (and are) typically prohibited from use on new ground systems; and 2) viable alternatives, while promising in laboratory studies, had still not been demonstrated on fielded HHA based systems such as Stryker.[3] Significant progress has been made during the execution of Strategic Environmental Research and Development Program (SERDP) Project WP-1521, "Non-Chromate, Non-VOC Coating Systems for DoD Applications." The project, which was completed in fiscal year (FY) 2008, investigated a number of promising pretreatment technologies.

1.2 OBJECTIVE OF THE DEMONSTRATION

The objective of this demonstration is to determine the viability of non-chromate pretreatments for HHA steel in order to improve the long term corrosion resistance of the low-VOC Chemical Agent Resistant Coating (CARC) system to reduce lifecycle costs for these weapon systems. Stryker and MRAP vehicle contracts are prohibited from using hex-chrome and are currently coated without any corrosion inhibitive pretreatment or conversion coating. The products demonstrated here satisfy the hexavalent chrome prohibition for both vehicles while minimizing environmental impact and promoting worker safety. This demonstration was designed to

generate the data necessary for the authorization and implementation decisions by appropriate authorities within the DoD.

1.3 REGULATORY DRIVERS

The Occupational Safety and Health Administration (OSHA) Final Rules effective May 30, 2006, Federal Register #71:10099-10385 states in part that OSHA has amended the standard limiting occupational exposure to hexavalent chromium (Cr^{6+}). OSHA determined that at the current permissible exposure limit (PEL) for Cr^{6+} workers face a significant risk to hexavalent chrome related heath disorders. The evidence in the record for this rulemaking indicates that workers exposed to Cr^{6+} are at an increased risk of developing lung cancer. The record also indicates that occupational exposure to Cr^{6+} may result in asthma and damage to the nasal epithelia and skin. The final rule establishes an 8-hour time-weighted average (TWA) exposure limit of 5 micrograms of Cr^{6+} per cubic meter of air (5 $\mu g/m^3$). This is a considerable reduction from the previous PEL of 1 milligram per 10 cubic meters of air (1 mg/10 m^3, or 100 $\mu g/m^3$) reported as CrO_3, which is equivalent to a limit of 52 $\mu g/m^3$ as Cr^{6+}. The final rule also contains extensive ancillary provisions for worker protection. These requirements for exposure determination and preferred exposure control methods include a compliance alternative for smaller companies with limited manufacturing for which the new PEL is cost prohibitive and infeasible. Respiratory protection, protective clothing and equipment, hygiene areas and practices, medical surveillance, record-keeping, and start-up dates that include four years for the implementation of engineering controls to meet the PEL are necessary. The PEL established by this rule purportedly 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 of 2009, a memo from The Under Secretary of Defense, and signed by Mr. John Young was released outlining a new policy for reducing the use of Cr^{6+} for DoD applications. The memo specifically directs the military to approve the use of alternatives to Cr^{6+} where they can perform adequately for the intended application and operating environment, update relevant technical documents and specifications to authorize the use of qualified alternatives, and require the Program Executive Office (PEO) or equivalent, in coordination with the Military Department's Corrosion Control and Prevention Executive (CCPE), 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 is identified.

2.0 DEMONSTRATION TECHNOLOGY

2.1 TECHNOLOGY DESCRIPTION

The proposed alternative pretreatments can, in many cases, be used in place of chromated zinc phosphate and DoD-P-15328 wash primer. The technologies being demonstrated were spray applied pretreatments for steel substrates. The technologies being investigated include trivalent chromium and two non-chromium coatings that are commercially available: Surtec 650, Chemetall Oxsilan 9810/2, and Pittsburgh Plate Glass Industries (PPG) Zircobond 4200. Below is a brief description of each of the technologies.

2.1.1 Trivalent Chrome Pretreatment (TCP)

Trivalent Chrome Pretreatment (TCP) was developed by Naval Air Warfare Center (NAVAIR) in an effort to replace chromated sealers, post-treatments, and conversion coatings on aluminum. TCP is based on a fluorozirconate complex with a trivalent chromium salt. TCP contains significantly less total chromium than the current Cr^{6+} conversion coatings and has no Cr^{6+}.

SERDP project WP-1521[4] established that TCP forms a predominately zirconium oxide/fluoride, chromium oxide conversion coating on the aluminum alloy surface. Previous work has been conducted on hexavalent chromium films, suggesting a film backbone consisting of polymerized trivalent chromium hydroxide species with a loosely hydrogen-bonded active chromate inhibitor species. Chromate films tend to be very thin over precipitates and inter-metallics, only releasing the inhibitor species after the film has broken down and substrate metal is exposed. The mechanisms have been published elsewhere.[5] Simply stated, chromate is mobile in solution and migrates to exposed areas on the substrate surface, absorbs on the active corrosion sites and is reduced to form a monolayer of CrIII species. Electrochemical evidence suggests that the TCP forms a much more uniform film thickness across these inter-metallic sites with improved barrier coating properties than hex chrome. Additionally, a denser zirconium oxide and localized corrosion inhibition through the ability of the trivalent chromium species slows the reaction kinetics of metal-attacking anions, such as chloride. The product manufactured by SurTec International is a green, clear-turbid liquid with a density of 1.00-1.01 grams per milliliter (g/ml) and an approximate pH of 3.8. Project WP-1521 showed that SurTec 650 demonstrated benefits as a flash rust inhibitor as well as an adhesion promoter. Below are the manufacturer's instructions for applying SurTec 650:

1. Pressure wash all parts to remove dirt and grime;
2. Abrasive blast to 1.5 mils Surface Profile using Al oxide (or equivalent) 54-60 grit;
3. Spray clean with mild/neutral cleaner containing slight rust inhibitor (Surtec 011 or 101);
4. Rinse clean with deionized (DI) Water;
5. Spray with SurTec 650 ready to use (RTU) keeping surface area moist for 5-6 minutes;
6. Rinse with DI water and blow dry; and
7. Apply CARC system after complete dry.

2.1.2 Oxsilan 9810/2

Chemetall's Oxsilan 9810/2 is a silane product modified with metallic acids. A simple silane molecule consists of a silicon atom combined with an organic molecule. For paint pretreatment, however, more complex silanes described as "organofunctional" such as the Oxsilan 9810/2 are used. Through proper selection of the organic constituents used in the silane molecule an organofunctional silane molecule is created that reacts and forms bonds with metal hydroxides on the substrate and organic groups on paint resins. These organofunctional silanes are then reacted with water introduced during the pretreatment supplier's manufacturing process. They form what are called "polycondensates." This complex retains the paint and metal-bonding properties of the silane, but in an easy-to-use form. The polycondensate is the innocuous chemical form in which silane products are usually made commercially available to metal finishers.

In use, as the silane film dries on the pretreated substrate, neighboring hydroxyl groups on the silane molecule react with each other to form a dense cross-linked network. Finally, in order to further enhance performance, non-regulated group IV-B metals, such as zirconium, are used to selectively and preferentially bond to the metal substrate, providing improved corrosion resistance compared to a silane-only process. In effect, a dual coating is formed in one step: an inorganic coating comprised of zirconium and other unregulated metals, as well as organofunctional silane coating. Oxsilan 9810/2, is a phosphorus free liquid, free of any regulated heavy metals, with a pH of 4-6, formulated for use on multiple metals including steel, aluminum, and zinc. It is applied at ambient temperature by either spray or immersion.[6] Manufacturer's instruction for applying Oxsilan 9810/2:

1. Pressure wash all parts to remove dirt and grime;
2. Abrasive blast to 1.5 Surface Profile in accordance with (IAW) Society for Protective Coatings (SSPC) SP 10;
3. Blow-down dust;
4. Apply Oxsilan 9810/2 solution (IAW Chemetall Technical datasheet [TDS]) @ 70 - 80 EF for 60 - 90 seconds contact time;
5. Rinse with clean with DI water and blow dry; and
6. Apply CARC system after complete dry.

2.1.3 Zircobond 4200

PPG has developed Zircobond 4200 pretreatment, an alternative pretreatment based on zirconium chemistry and a proprietary blend of additives. Zircobond 4200 pretreatment reduces sludge by-product from the pretreatment process by at least 80 percent compared to zinc-phosphate-based products and it can be used as a drop in replacement in existing pretreatment lines. The Zircobond 4200 system is formulated to provide corrosion resistance for steel, galvanized steel and aluminum substrates. It is a clear light-blue liquid with a specific gravity of 1.104 and has a diluted working pH of between 4.0 and 5.0. Manufacturer's instructions for applying Zircobond 4200:

1. Pressure wash all parts to remove dirt and grime
2. Abrasive blast to 1.5 mils Surface Profile using Al oxide (or equivalent) 54-60 grit
3. Blow off dust
4. Chemkleen254LF (2% by volume) 60 second spray at 125 EF
5. Ambient DI water rinse
6. Zicrobond4200 (3% by volume) 120 second spray at 80 EF
7. DI Rinse
8. Forced Air Dry
9. Apply CARC system after complete dry.

2.2 ADVANTAGES AND LIMITATIONS OF THE TECHNOLOGY

Because of Stress Corrosion Cracking (SCC) concerns perceived with HHA when using some pretreatments such as zinc phosphate and wash primer, some platforms have been painted without the benefits of a pretreatment. Only a flash rust inhibitor is currently used on Stryker and MRAP and overall, the application processes of the alternative technologies are very similar to the current process. The alternatives demonstrated are hexavalent chrome-free processes that have no HAP's and are low VOC. The addition of a true chemical pretreatment/conversion coating provides a complete CARC system as defined in MIL-DTL-53072 for armor steel platforms and adds another layer of corrosion protection while improving coating adhesion. Flash rust capability would not be compromised as the alternatives provide this protection. Since the application process is similar with all of the alternatives, they would be considered drop-in replacements with no environmentally assisted cracking concerns. All of the alternatives are relatively low cost and do not include a large capital investment.

Some limitations were discovered during the demonstrations. We learned that the Oxsilan is sensitive to some surface conditions and application rates. These parameters must be diligently controlled during the process similar to any other spray pretreatment process. Although the Zircobond provided adequate corrosion protection, it left a blotchy finish on the substrate that made it difficult for the user to determine if it was properly applied.

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3.0 PERFORMANCE OBJECTIVES

The performance objectives with success criteria for the demonstrated technologies were evaluated in accordance with the tests delineated in the Joint Test Protocol (JTP) provided in Appendix A of the Final Report. The performance objectives are summarized in Table 1. The primary material used in the construction of these platforms is MIL-DTL-46100 (HHA) steel. Performance objectives were derived using HHA as the base metal. The existing product currently used on Stryker (Cheminhib 420) and similar material on MRAP is considered the baseline process.

Table 1. Performance objectives for alternative pretreatments.

Performance Objective	Data Requirements	Success Criteria	Results		
			SurTec 650	Oxsilan 9810/2	Zircobond 4200
Quantitative Performance Objectives					
Humidity Testing	Comparative test for flash rust inhibition	No flash rust after 24 hours of exposure to ambient temperature and 90% relative humidity	Met	Met	Not Met
Adhesion Test	ASTM International - 4541 Pull-off Adhesion	Minimum average 30 events rating of 1200 pounds per square inch (PSI) on 1.5 mil profile surface	Met	Met	Met
	ASTM- D3359 Dry Adhesion	Adhesion rating (steel) > 4B; adhesion rating	Met	Met	Met
	ASTM- D3359 Wet Adhesion	Scribed area rating (steel) ≥ 3A after 24 hours at ambient;	Met	Met	Met
Chip Resistance	SAE-J400	After one cycle, chip rating NLT 5B for steel	Met	Met	Met
Accelerated corrosion	ASTM-B117 Salt Fog	After 500 hours of exposure: steel substrate rating ≥6 scribed	Met	Met	Met
	GM-9540P ASTM D 1654	After 60 cycles: steel substrate rating ≥ 4	Met	Met	Met
Outdoor Exposure	Tropical climate Cape Canaveral ASTM D 1654 ASTM G50	Exposure for 3 years: specimen has a minimum of 25% less creepage from scribe than current corrosion protection system	N/A	N/A	N/A
Hydrogen Embrittlement	ASTM E 399-97	No detrimental effect to K1c of substrate. High Hard K1c @ 48-51Rc shall maintain $K_{1EAC} \geq 19$ (ksi√in)	Met	Met	Met
Toxicity Clearance	Toxicity clearances and full disclosure from CHPPM	Approved by processing facility	Met	Met	Met
Processing time	TT-C-490	Equivalent or less than existing process	Met	Met	Met
Field Testing	TT-C-490	Equivalent or less than existing process	Met	Met	Met
Qualitative Performance Objectives					
Ease of use	Feedback from field technician on usability of technology	Minimal operator training required	Met	Met	Met

There were three demonstrations conducted using the Oxsilan 9810/2. The performance objectives in Table 1 represent the overall view considering the lab results as well as all of the demonstrations. The project team believes for Ease-of-Use was based on the manufacturers recommended application instructions and the limited input from the applicators during the demonstrations. The objective for Oxsilan was considered “Not Met” during the MRAP demonstration because the Oxsilan 9810/2 process appeared to be (visually) more sensitive to flow rates and substrate surface conditions than applying a simple flash rust inhibitor such as the baseline Cheminhib 420. This was our view of this isolated event using the equipment available at the time. However, it was shown that surface contamination played a role, and with adequate flow of the Oxsilan 9810/2 on a substrate properly prepared according to TT-C-490F, the process is considered no more complicated than the baseline. This was shown in the subsequent MRAP Doors demo of which the objective here was considered “Met.” Overall (Table 1) it is believed that when the manufacturer’s instructions and TT-C-490F are followed, no additional training is needed, which meets the objective for ease of use. Additionally, the results that occurred with an improperly applied Oxsilan 9810/2 at Camp LeJeune would help serve as a quality indicator for the pretreatment.

4.0 SITES/PLATFORM DESCRIPTION

4.1 TEST PLATFORMS/FACILITIES

There are two parts to this demonstration of pretreatments for HHA: 1) Stryker parts and 2) MRAP. The first was carried out at Anniston Army Depot (ANAD) during an ongoing RESET of Stryker Depot Repair Cycle Float (DRCF)-3 vehicles. The Stryker Brigade Combat Team (SBCT) agreed to allow the Army Research Laboratory (ARL) to demonstrate the pretreatments on the hatches of three Stryker vehicles (power entry panel [PEP] hatch, front access hatch, and side egress hatch). The RESET of these vehicles was set to end on or about October 15, 2010.

The ANAD site was selected because it was the location performing the RESET on a major combat vehicle constructed of high hard steel, and the Program Managers Office (PMO) SBCT and ARL have a written Memorandum of Agreement for environmental compliance, enhanced materials, advanced coatings, and improved processes at OEM and depot facilities. All of the work was performed on site at ANAD. The parts (hatches) were removed from each vehicle by the Stryker RESET team and tagged in order to stay mated with their specific vehicles. Each hatch was abrasive blasted, pretreated, primed and CARC topcoated then reinstalled. The pictures below in Figure 1 are of one of the actual vehicles used for the demonstration.

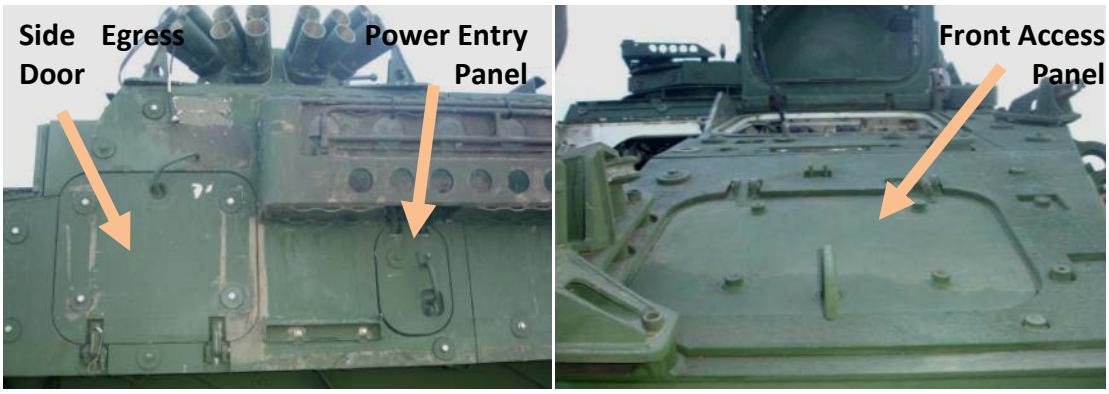


Figure 1. One of the Stryker vehicles and hatches used in the demonstration.

The second part of the demonstration of pretreatments for HHA steel was carried out at Camp Lejeune, North Carolina on MRAPs returning from theater. Camp Lejeune was selected for this demonstration for three reasons: 1) MRAPs would be returning from theater at approximately the same timeframe the demonstrations would begin 2) ARL received early support from the U.S. Marine Corps (USMC), Corrosion Prevention and Control (CPAC) Program Support to MRAP II Acquisition and from the MRAP JPO 3) Camp Lejeune has all of the capabilities necessary to process the vehicles as required for the pretreatments.

Two MRAP vehicles were selected from a lot of 33 returning from theater. Every effort was taken to ensure the vehicles were as similar as practical to remove as many variables as possible. Once identified, the entire exterior of each vehicle was completely abrasive blasted down to near-white metal prior to pretreatment and paint. A follow-on demonstration was conducted at Aberdeen Proving Ground using a set of MRAP rear doors in order to validate the lessons-learned from the Camp Lejeune demonstration.

4.2 PRESENT OPERATIONS

As mentioned earlier, a true CARC system as defined in MIL-DTL-53072. Figures 2 and 3 are flow diagrams for the painting process for Stryker and MRAP respectively. Note that there are interim steps in both cases that involve the application of a flash rust inhibitor that temporarily suppresses corrosion and, not meant to assist in the long term corrosion protection or adhesion of the CARC system.

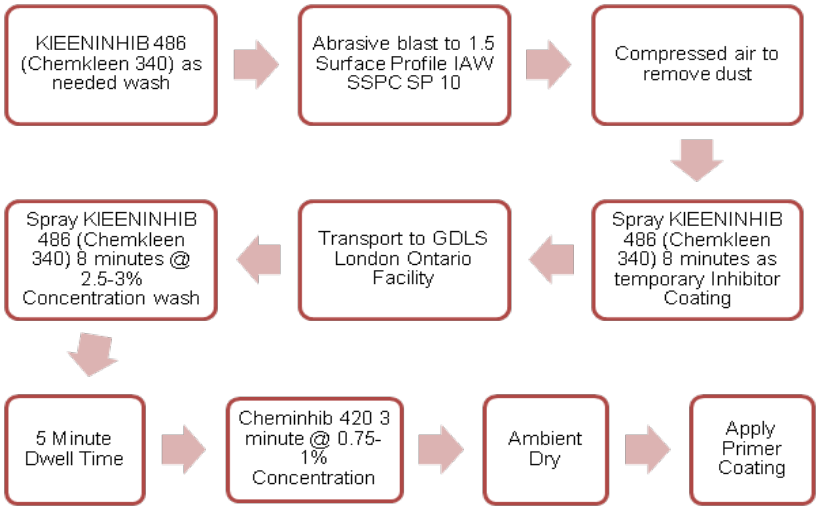


Figure 2. Typical flow diagram of the current painting process for Stryker vehicles.

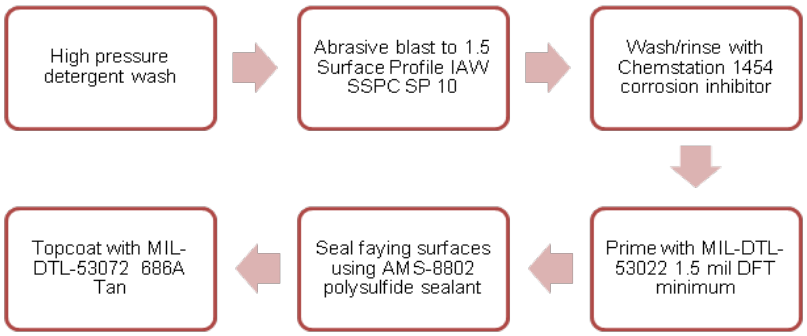


Figure 3. Typical flow diagram of the current painting process for MRAP vehicles.

The demonstrated technology is intended to replace the temporary flash rust inhibitor step in the process with a more effective pretreatment, and thus will not require additional steps to the current process. In fact, in some cases, it is expected to save time overall and the added benefit of providing enhanced corrosion protection and better adhesion of the CARC system.

4.3 SITE-RELATED PERMITS AND REGULATIONS

Additional site related permits or regulations were not required for the demonstration to be conducted at ANAD and Camp Lejeune. These 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 if necessary.

5.0 TEST DESIGN

5.1 CONCEPTUAL EXPERIMENTAL DESIGN

The laboratory testing is described in more detail in the Final Report.[7] The three pretreatments: SurTec 650 TCP, Chemetall Oxsilan 9810/2 (Silane), and PPG Zircobond 4200 (ZrOx) were laboratory validated and field tested according to the JTP. In addition, field testing on Stryker and MRAP components was conducted.

The experiments were conducted using 4 inch x 6 inch x 3/16 inch MIL-DTL-46100 steel test panels. All of the test panels were abrasive blasted to a 1.5 mil surface finish and the pretreatments applied by the vendors in order to eliminate inconsistencies in the processes. All primer and topcoats were applied by ARL. The coatings used were MIL-DTL-53022 Type II primer, solvent borne MIL-DTL-53039 Type III topcoat, and water borne MIL-DTL-64159 Type II topcoat all manufactured by Hentzen.

Flash rust inhibition was assessed using a modified version of ASTM D 1735. Pretreatment candidates were applied to freshly abrasive blasted HHA test panels and left unpainted in a chamber with continuous static environment of 100°F chamber temperature and at 90% relative humidity (RH). Samples were periodically removed for evaluation using 3M pressure sensitive tape to capture any existing surface corrosion every 24 hours.

Dry tape adhesion tests were conducted at room temperature as defined in ASTM D 3924. Using a sharp cutting tool, 6 parallel cuts @2mm spacing through the paint film to the metal substrate were made. A second series of cuts at 90 degrees to the initial set were then made. A piece of 3M 396 tape was used to further clean off the area by lightly touching it to the grid lines to remove any detritus that would interfere with the full application of the test tape. The center of the tape was placed over the grid and the area of the grid smoothed into place. The tape was removed, pulling (not jerked) back upon itself (parallel to the test surface) at as close to an angle of 180° as possible. Following the tape pull off, each grid was rated using the classification in ASTM D 3359.

Wet tape adhesion test was performed in accordance to Method 6301 of FED-STD-141[8] and rated per ASTM D 3359. The samples were immersed in distilled water for 24 and 96 hours at room temperature and 120EF, respectively. Two parallel lines were scribed approximately one inch apart with an “X” scribed between the two parallel lines. A piece of tape was placed over the scribes and removed. The areas around the scribes were inspected for peel-away/delamination, rated, and photo-documented.

Pull-off Adhesion was assessed using ASTM D 4541. An Elcometer Model 108 Hydraulic Adhesion Test Equipment (HATE) was used to obtain the pull-off adhesion strength in PSI. A loading fixture commonly referred to as a “dolly” was secured to the coating using Instabond™ S-100 cyanoacrylate adhesive. After allowing the adhesive to cure for 24 hours at 25 ºC at 50% RH, a gradually increasing load was applied to the dolly until a plug of coating was detached. The failure tension in PSI was recorded and the failure mode and location within the coating system was recorded.

The panels were then subjected to chip resistance testing IAW Society of Automotive Engineers (SAE) J400 at ambient temperature using a Q-Lab Gravelometer. The panels were held in a 45° angle specimen holder and air pressure was used to propel gravel at the sample. Tape was then applied to the entire tested surface in order to remove any loose fragments of the coating. The tested panel was then compared to standard SAE transparencies to determine a chipping rating.

Two accelerated corrosion test tests were conducted to assess the corrosion resistance of the pretreatments: ASTM B117 salt fog, and GM9540P cyclic corrosion. All B117 test coupons completed 1000 hours of exposure while GM9540P samples completed 80 cycles. The samples were periodically inspected and evaluated accordance with ASTM D1654 for both corrosion creep from the scribe as well as blistering in the field. Once panels completed the full duration, they were scraped and re evaluated for a final rating.

Outdoor Exposure coupons were X-scribed as described in ASTM D1654. These panels were mounted to racks at Cape Canaveral parallel to and approximately 100 yards inland from the ocean. The coupons are being inspected and evaluated biannually in June and December in accordance with ASTM D1654 for both corrosion creep from the scribe as well as blistering in the field. Weather data was collected utilizing a data-logging weather station and downloaded annually.

Stress Corrosion Cracking Evaluation:
The resistance to environmentally assisted cracking was assessed using the rising step load method for determination of K1EAC using CV2 Charpy specimens machined longitudinal-transverse (L-T) orientation according to IAW ASTM E 399-97.

5.2 STRYKER COMPONENT DEMONSTRATION

The demonstration on Stryker vehicles was initiated on September 28, 2010. All hatches were first pressure washed to remove dirt, grease and grime prior to abrasive blasting then abrasive blasted to a surface profile of 1.5 mil in accordance with Steel Structures Painting Council (SPC) standards. Visual cleanliness was determined using SSPC VIS1, Standard for Abrasive Blasting. A water break test was performed to determine the presence of any contaminants prior to pretreatment.

Each candidate pretreatment was applied to hatches of each platform according to the manufacturer's required procedure described in Section 2.0. Once pretreated, all of the hatches were stored overnight for 19 hours in ambient shop conditions (60%-70% RH) to duplicate actual coating process lines and to evaluate flash rust inhibition. According to Section 3.5.4 of TT-C-490[9], the organic coating shall be applied to thoroughly dried surfaces within 24 hours after pretreatment. All hatches were primed within 23 hours of pretreatment and topcoated the following morning. After the hatches were painted, they were reinstalled on their respective vehicles. Table 2 lists the actual vehicle identifications and the pretreatments used for each hatch on the vehicles.

Table 2. Pretreatments used to treat specific components.

Component	Stryker Demonstration Vehicles Identification		
	MEV-76	MGS-25	ICV-382
PEP Hatch	SurTec 650 (TCP)	PPG Zircobond 4200	Chemetall Oxsilan
Front Access Hatch	PPG Zircobond 4200	Chemetall Oxsilan	SurTec 650 (TCP)
Side Egress Hatch	Chemetall Oxsilan	SurTec 650 (TCP)	PPG Zircobond 4200

Once hatches were reinstalled, ARL tracked vehicle locations for subsequent inspections. The vehicles were designated for Joint Base Lewis-McChord (JBLM) Fort Lewis Washington and the point of contact (POC) was: Catherine Doherty, catherine.doherty@us.army.mil, office: 586-282-2157, DSN: 782-2157, BB: 586-770-8721.

The metric for evaluating the hatches during inspection was a visual comparison with the base vehicle using the Society for Protective Coatings SSPC-VIS 2 "Standard Method for Evaluating the Degree of Rusting on Painted Steel Surfaces." The success criterion for the fielded hatches is performance greater than or equal to the base vehicle (baseline). SSPC-VIS 2 quantified the degree of rusting on painted steel surfaces with a zero to ten scale based on percentage of visible rust present on the surface. Visible rust includes rust blisters and undercutting of the coating.

5.3 MRAP AND MRAP COMPONENT DEMONSTRATION

The MRAP demonstrations were coordinated through the MRAP PMO and the USMC, CPAC Program Support to MRAP II Acquisition. Camp Lejeune is a major repair facility for the Marine Corps. CWO5 Mark Schmidt and Mr. Daniel Cooper CWO-5 USMC (ret) Senior Logistics Support Coordinator for the II MEF LNO office provided two trucks (MRAPs) for the demonstration. Once each vehicle was completely processed and fielded, Mr. Cooper would provide the destination of each demonstration vehicle to ARL. The following is his contact information: Daniel Cooper, daniel.cooper@usmc.mil, Office (910) 451-8151 (DSN 751), BB/Cell (910) 581-8644.

Oxsilan 9810/2 was preferred because it contained no chromium component and would be easier to get approved by Camp Lejeune. Every attempt was made to select two variants that were as similar as possible to minimize variability. The demonstration team arrived at Camp Lejeune on the morning of June 20, 2011. Upon arrival, MRAP #1 (USMC Vehicle Identification Number [VIN] 634590) was fully abrasive blasted using 60-grit garnet blast media on the previous Friday, June 17, 2011. MRAP #2 (USMC VIN 633359) was approximately 80% abrasive blasted and would not be finished until the following day. Application commenced on MRAP #1 at approximately 1300 hours in a covered outdoor environment outside the blast booth. Environmental conditions were sunny and clear with a temperature of 85 EF and 55% RH at the beginning of the application. A full account of the weather conditions from June 17 to June 20, 2011, are shown below in Table 3.[10]

Table 3. Conditions during demonstration at Camp Lejeune.

	Vehicle Abrasive Blasted Friday, June 17, 2011	Saturday, June 18, 2011	Sunday, June 19, 2011	Day of Demonstration Monday, June 20, 2011
Mean Temperature	82 °F	82 °F	82 °F	84 °F
Maximum Temperature	91 °F	93 °F	95 °F	93 °F
Minimum Temperature	73 °F	72 °F	70 °F	75 °F
Dew Point	71 °F	71 °F	70 °F	70 °F
Average Humidity	75	74	71	73
Maximum Humidity	94	94	93	100
Minimum Humidity	42	39	41	36
Precipitation	0.00 in	0.16 in	0.00 in	0.16 in
Sea Level Pressure	29.96 in	29.94 in	29.87 in	29.85 in
Wind Speed	7 mph (SW)	7 mph (SSW)	8 mph (WSW)	8 mph (WNW)
Maximum Wind Speed	17 mph	28 mph	20 mph	21 mph
Maximum Gust Speed	21 mph	34 mph	29 mph	26 mph
Visibility	9 miles	8 miles	9 miles	8 miles
Events	T-storm	Rain , T-storm	---	Rain , T-storm

The application procedure for Oxsilan 9810/2 recommended by Gary Nelson, Product Manager, Chemetall, New Jersey is shown in Section 2. Two 1.1 gallons per minute (gpm) capacity spray pumps were used for the application and rinsing. In addition, a person with a stop-watch was designated to monitor the required time intervals. DI water was used in all steps of the process. Notes were taken throughout the process. A representative from the manufacturer was there to monitor the processing of the vehicles and guide ARL and Camp Lejeune through the pretreatment process.

Supplemental MRAP Demonstration:

During the week of August 20, 2012, the supplemental demonstration was initiated on two HHA MRAP rear armor steel doors. The two HHA steel doors were abrasive blasted to near-white metal. Oxsilan 9810/2 RTU was applied to both sides of one door with a portable sprayer recommended by Chemetall. The sprayer was a Yamada NDP-15 BPT pump with an 80E nozzle with a 4.0 gpm rating. At 10 PSI estimated nozzle pressure, the flow calculates to about 2 gpm. The door was rinsed with DI water using the sprayer and then force dried with compressed air. The door was allowed to air dry an additional 1.5 hours to ensure no moisture remained. Door temperature, air temperature and dew point were measured to make sure there was a wide enough spread to ensure drying and that no condensation would form when painting. The door that was not coated with Oxsilan 9810/2, it was coated with DoD-P-15328 washprimer according to standard procedure. Both doors were then primed with a MIL-DTL-53022 primer and MIL-DTL-53039 CARC topcoat. When fully dry, the coated doors were placed on a rack at an angle of ~30º outdoors in the back lot of the ARL Rodman Building at Aberdeen Proving Ground and subjected to environmental exposure testing.

6.0 PERFORMANCE ASSESSMENT

Some of the initial testing is described in Section 2.0 Demonstration Technology. However, to fully evaluate the steel pretreatments on armor steel, initial screening tests were performed to gage the relative performance of the alternatives versus the baseline Cheminhib 420 and control. Because of the very small window of opportunity for access to Stryker vehicles during the Reset of the former 1/25 SBCT vehicles (DRCF-3 vehicles), a full battery of tests could not be completed prior to initiating the demonstration. Screening tests were performed on the candidate pretreatments. Table 4 lists the success criteria that were used in screening the candidate pretreatments demonstrated on Stryker. Much of the laboratory testing was completed prior to initiating the demonstration on MRAP at Camp Lejeune NC. These results are presented later in this section.

Table 4. Screening requirements for demonstrations on Stryker.

Test	Acceptance Criteria	Test Method References	Result
Adhesion (Pull-Off)	Meets or exceeds adhesion strength of DoD-P-15328 on similarly prepared abrasive blasted surface of 1.5 mil profile or 1200 PSI	ASTM-4541 Pull-off Adhesion	Met
Corrosion Resistance	336 hours of exposure: Steel substrate rating ≥ 7 scribed	ASTM B117, ASTM D1654	Met
Toxicity Clearance	Toxicity Clearances and site approval	None	Met

For the MRAP and Supplemental MRAP doors demonstrations, much of the laboratory tests outlined in the Performance Objectives in Table 1 were completed. Only the outdoor exposure tests are ongoing. At the time of the writing of this report, all samples have reached two of the three years needed to assess performance. This section will discuss the laboratory test results along with the assessment of the demonstrations.

6.1 LABORATORY RESULTS

Flash rust inhibitors such as the Cheminhib 420 are used on both Stryker and MRAP to prevent corrosion prior to painting. Oxsilan 9810/2 and the SurTec 650 provided better flash rust inhibition than the baseline 1% Cheminhib 420 through both 24 and 48 hours in the humidity chamber. The Zircobond did not meet this requirement because it was not clear if the blotchy coloring was indicative of the pretreatment or corrosion. In any case, this type of discoloration would make it difficult to assess the quality of the coating to the user.

The adhesion of the primer and topcoat to the substrate as enhanced by the pretreatments is an important consideration. Table 5 is a summary of all indicators of adhesion strength provided by the pretreatment/coating combinations. The success criterion for pull-off adhesion was set at 1200 PSI which represents the average pull-off strength achieved for DoD-P-15328 wash primer on low carbon steel with a milled finish (63-125µ inch). This is considered to be ample pull off strength for an organic coating. All of the pretreatments met the pull off strength criterion. For dry tape adhesion, the success criterion is 4B. All of the alternatives met the 4B rating with the exception of SurTec 650 with the MIL-DTL-53022/ MIL-DTL-64159 paint system. Note that of

the baselines and control, only one, Cheminhib 420 with MIL-DTL-53022/ MIL-DTL-64159 met the dry tape rating requirement. All pretreatments and baseline had better dry adhesion ratings than the control (abrasive blasted-no pretreatment) indicating that the chemical pretreatments enhanced the adhesion of the organic coatings.

Table 5. Average results for coating adhesion and chip resistance.

Pretreatments	MIL-DTL-53022/MIL-DTL-53039				MIL-DTL-53022/MIL-DTL-64159			
	Pull Off Adhesion (PSI)	Dry Tape Adhesion Rating	Wet Tape Adhesion Rating	Chip Resistance Rating	Pull Off Adhesion (PSI)	Dry Tape Adhesion Rating	Wet Tape Adhesion Rating	Chip Resistance Rating
Abrasive Blast Only	1549	2.7	4	6 A/B	2080	3.5	3	5 B/A
Cheminhib 420	1626	3.5	4	5 B/A	1854	4.7	4	5 B
DOD-P-15328	1573	3.7	4	4 B	1420	3.8	0	4 B
SurTec 650	1546	4.0	5	5 A	1961	3.8	5	4 B/A
Oxsilan 9810/2	1651	4.3	5	5 B	1883	5.0	5	5 B
Zircobond 4200	1636	4.7	4	5 B/A	1820	4.3	4	5 B

Wet adhesion tests were carried out according to ADTM-3359 method A scribing technique with this caveat: The specification does not prescribe water, temperature; or duration. The success criterion for the pretreatments was derived using NAVAIR requirements. The wet tape adhesion test results are also shown in Table 5. The success criterion is minimum rating of three after 24 hours immersed in ambient DI water. All of the samples tested met the minimum rating of three with the exception of DoD-P-15328 wash primer with MIL-DTL-53022/MIL-DTL-64159 coating system. Under these conditions, the SurTec 650 and the Oxsilan 9810/2 performed better than the baseline and control achieving a 5 rating.

Another indication of adhesion is the ability of the coating system to resist chipping. This is particularly important for military ground vehicles that navigate in rough terrain. The success criterion for chip resistance is a 5B. The DoD-P-15328 did not pass with either coating system. Of the alternatives, the SurTec 650 was only able to achieve a 4B/A rating, meaning the size of chips are acceptable, but the four ratings suggests that it was slightly more susceptible to chipping. SurTec 650 performance was similar to the wash primer with the MIL-DTL-53022/MIL-DTL-64159 system. All other alternatives met or exceeded the success criterion.

Accelerated Corrosion:

Only test panels coated with CARC system (MIL-DTL-53022/MIL-DTL-53039) were tested in ASTM B117. The primary mode of failure measured was creepage from the scribe. The ratings of five replicates of each pretreatment were averaged and presented in Figure 4. Beyond 500 hours of exposure, the alternatives displayed less creepage from the scribe than the baseline and control, and meeting the success criteria of a $\geq$ 6 scribed. After all samples had completed 1000 hours, they were scraped before the final measurements were made. All three of the alternatives were markedly better than the control, baseline Cheminhib 420, and chromate wash primer DoD-P-15328. In fact, all were very close to meeting the 6.0 rating even after 1000 hours. As previously mentioned, the Cheminhib 420 is the baseline the alternatives are being evaluated against. It proved to be the worst of all the test panels measured in ASTM B117 salt fog.

Representative panels from each set of replicates are shown in Figure 5. Ignoring the rust bleeding from the scribe, one can see that there is more pitting and creepback on the Cheminhib 420 and DoD-P-15328 panels.

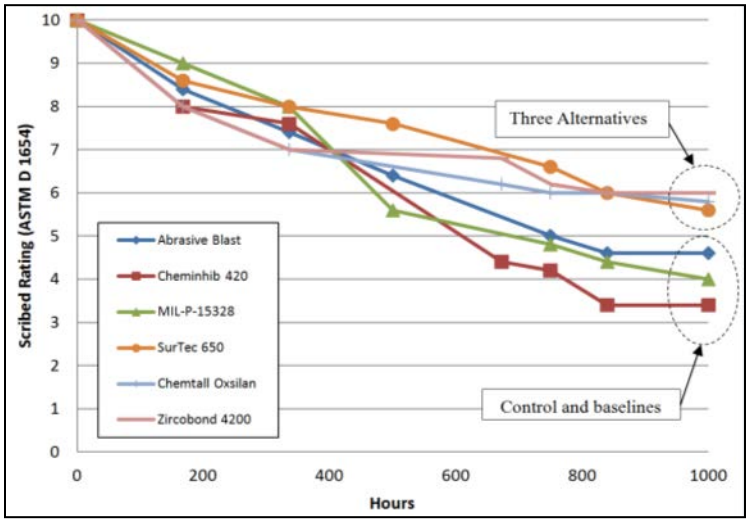


Figure 4. ASTM D 1654 rating for abrasive blasted HHA panels through 1000 hours of ASTM B117 salt fog exposure.

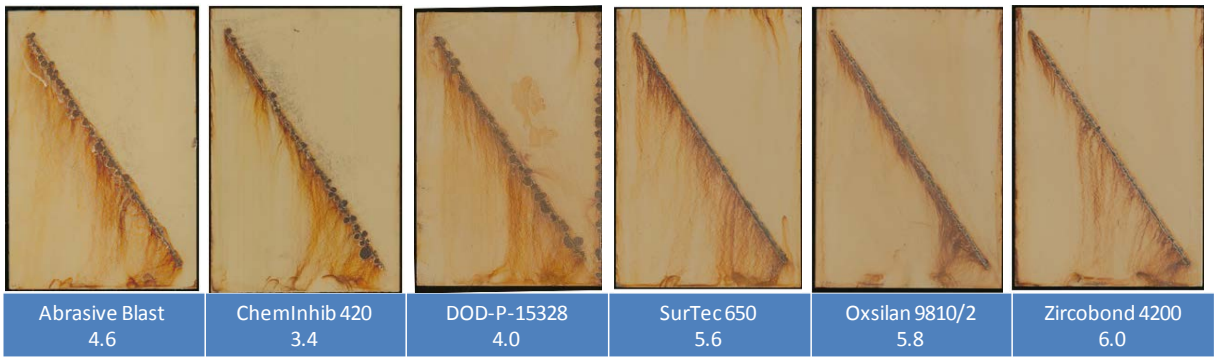


Figure 5. Abrasive blasted HHA with MIL-DTL-53022/MIL-DTL-53039 scraped after 1000 hrs B117 exposure.

Sets of panels with two CARC coating systems, MIL-DTL-53022 / MIL-DTL-53039 and MIL-DTL-53022 / MIL-DTL-64159 were tested in GM 9540P cyclic corrosion. Replicates of three were used for the GM 9540P tests for each pretreatment and baseline. Table 6 lists the ratings averaged over the three replicates. The success criterion is an average ASTM 1654 rating of ≥ 4 for "X" scribed panels at 60 cycles. The test was carried out to 80 cycles before scraping the panels for a final rating. Similar to the ASTM B117 results, only the alternative pretreatments were able to meet the required ≥ 4. The only exception was the Zircobond 4200 coating with DTL-53022 / MIL-DTL-64159 where one of the three panels in the set rated a three dropping the average rating below four. Even with an average of 3.7, the Zircobond performed better than any baseline with either coating system. When the test reach 80 cycles and the panels were scraped and measured, all of the controls (abrasive blast only), and nearly all baselines and control test panels rated a zero or near zero. Photographs of representative panels are in Figures 6 and 7. The

three alternatives clearly are better at resisting creepage from the scribe; however there is some secondary blistering seen in areas away from the scribe on the SurTec 650. Overall, there was no real difference between pretreatments coated with MIL-DTL-53022 / MIL-DTL-53039 or MIL-DTL-53022 / MIL-DTL-64159. The similar performance of the two coating systems is likely because MIL-DTL-53022 was used for the primer in both cases.

Table 6. GM 9540P results for HHA pretreated and coated with two CARC systems.

Pretreatment	Average for 60 Cycles		Average for 80 Cycles	
	MIL-DTL-53039	MIL-DTL-64159	MIL-DTL-53039	MIL-DTL-64159
Abrasive Blast Only	1.3	2.0	0.0	0.0
PPG Cheminhib 420	1.0	0.3	0.0	0.0
DOD-P-15328	2.3	2.7	0.3	1.0
SurTec 650 (TCP)	4.3	4.7	2.3	3.3
Chemetall Oxsilan 9810/2	4.3	4.0	2.7	2.7
PPG Zircobond 4200	5.3	3.7	3.3	2.0

Figure 6. Average ratings of HHA test panels scraped after 80 cycles of GM 9540P with MIL-DTL-53022/MIL-DTL-53039 CARC.

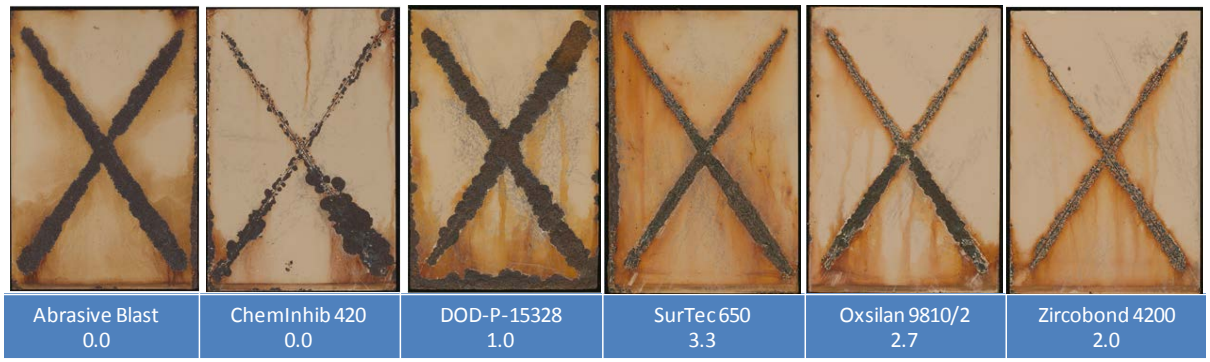


Figure 7. Average ratings of HHA test panels scraped after 80 cycles of GM 9540P with MIL-DTL-53022/MIL-DTL-64159 CARC.

Outdoor Exposure at Cape Canaveral:

The success criteria of 25% less creepage from the scribe than the Cheminhib 420 baseline will not be fully assessed until after 3 years of outdoor exposure has been completed. When 3 years

of exposure is achieved, a final rating will be determined when the test panels are scraped and measured. The most recent inspections were conducted at 2 years and those results for both coating systems are presented in Figure 8. The data presented here represent the average ASTM-1654 ratings vs time of exposure. All three of the alternatives; SurTec 650, Oxsilan 9810/2 and Zircobond 4200 are outperforming the baselines with MIL-DTL-53022/MIL-DTL-53039. For the MIL-DTL-53022/MIL-DTL-64159 dataset, the grouping is tighter with Oxsilan 9810/2 and SurTec 650 rating above 7.0. These ratings are likely higher than the MIL-DTL-53039 dataset in outdoor exposure because the waterborne CARC topcoat (MIL-DTL-64159) has been proven to have better ultraviolet (UV) resistance in previous testing.

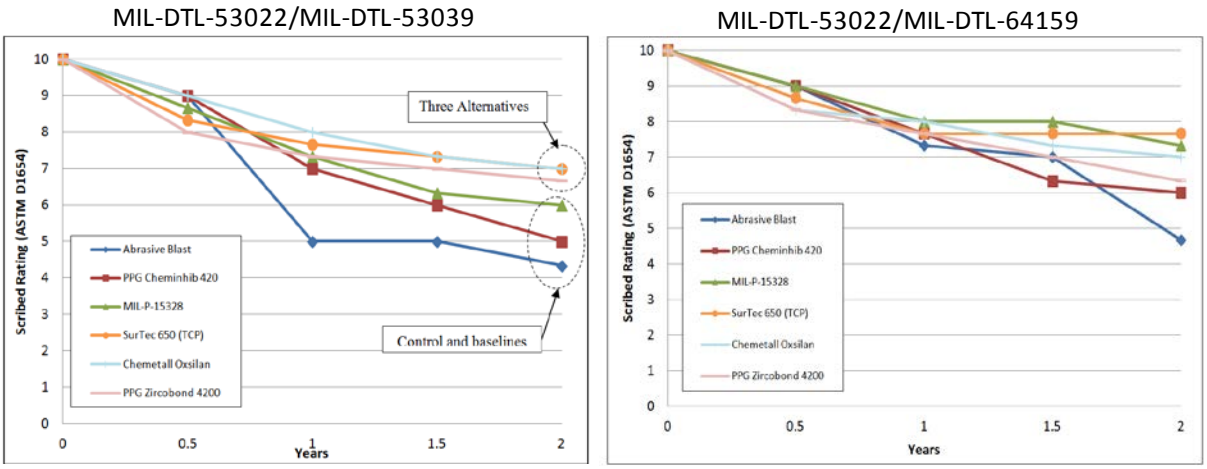


Figure 8. ASTM D 1654 ratings for coated HHA panels after 2 years outdoor exposure.

Visual inspections of the outdoor exposure panels indicate that each of the alternatives is performing better than the Cheminhib baseline. All appear to enhance the corrosion performance of the coating system versus abrasive blasting alone. To date, this suggests that a direct-to-metal process alone may not be a sufficient method for preparing HHA for paint.

Finally, it is important to determine if any of the proposed pretreatments would have a detrimental effect on the HHA resistance to environmentally assisted cracking. The K1EAC results were measured using the rising step load method. When the empirical data for K1EAC is compared with that found in the literature, it is clear that none of the alternatives had any influence on the MIL-DTL-46100 resistance to environmentally assisted cracking.[11, 12]

6.2 STRYKER DEMONSTRATION RESULTS

Figure 9 shows three hatches 19 hours after the application of the pretreatments. Only the Zircobond showed noticeable discoloration of the steel. The blotchy color change initially appeared as a pinkish rose color almost immediately after application and turned yellowish as it dried as seen in the picture below. It was not clear whether it was flash rust or an expected result from the reaction of the Zircobond and the steel. The Oxsilan 9810/2 and SurTec 650 showed no significant discoloration. Only slight darkening was observed with these two pretreatments. The lack of color or some type of indicator, however, makes it a challenge to detect if proper coverage was achieved.

Oxsilan 9810/2	SurTec 650	Zircobond 4200

Figure 9. Front access hatches after approx 19 hours ambient indoor exposure.

The Stryker hatch table (Table 2) in section 5 shows the vehicle identifications and how each of the hatches was pretreated. Two of the three Strykers (MGS-25 and ICV-382) were photographed and the results examined in April 2013 (after 2 years, 7 months in service). The third (MEV-76) was unavailable for inspection, so its condition is unknown at this time. The photographs below show the condition of the hatches from each vehicle that was inspected. It is important to note that these hatches are mated with bolt-on composite armor that is fastened on top of the HHA. Some wearing of the topcoat that occurs from vehicle vibration is typical.

The primary and secondary performance criteria evaluated in a production setting during the demonstration are presented in Table 7. In product evaluations with laboratory tests, almost without exception, all of the alternatives exceeded the performance of the baseline Cheminhib 420. Implementation of any of the three alternatives will maintain a Cr^{6+} free platform as none contain Cr^{6+}. The waste disposal metric was considered met because the application process is similar to the baseline and identical waste procedure, if any would apply. In each case, disposal would not necessitate the type of reporting required for the disposal of hexavalent chrome. Comparing the alternatives in identical processes conditions as the baseline can be accomplished by examining each process described in Section 2.0 with the baselines in Figures 2 and 3. The processes are not significantly different. Both baseline and alternative processes can be accomplished in about the same amount of time.

The secondary performance criteria are also described in Table 7. All of the alternatives tested can be used in a similar fashion as the baseline Cheminhib 420. No significant training would be required because the baseline and alternative application processes are similar. Provided the proper equipment is employed, only the manufacturer's instructions and a representative on site to guide the initial implementation would be necessary. This is not unique to the implementation of any drop-in-replacement. Storage and recordkeeping for any of the alternatives will depend on how it is purchased. They are mixed with DI water at a concentration ranging from 3-5% by volume. Therefore, the alternatives can be purchased and stored as a concentrate in smaller quantities, which will reduce the logistics and storage burden and the need to store and track partially used open containers. All are also water clean-up, which again, is similar to the baseline.

Table 7. Validation methods and performance metrics for demonstration on Stryker.

Performance Criteria	Expected Performance Metric (Pre-Demonstration)	Performance Evaluation Method	Actual Performance (Post-Demonstration)		
Primary Performance Criteria			**SurTec 650**	**Oxsilan 9810/2**	**Zircobond 4200**
Product Testing	Performance of alternatives will meet or exceed the current process used on Stryker during manufacturing as defined in the JTP in Appendix A of the Final Report	Laboratory analysis and field testing	Met	Met	Met
Hazardous Materials	Maintains a Cr^{6+} free platform	Assessment of product constituents and previous studies	Met	Met	Met
Hazardous Waste	Meets or exceeds current process used in Stryker manufacturing	Operating experience and assessments	Met	Met	Met
Factors Affecting Technology Performance	Comparison of alternatives in identical operating conditions	Operating Experience	Met	Met	Met
Secondary Performance Criteria			**SurTec 650**	**Oxsilan 9810/2**	**Zircobond 4200**
Ease of Use	Man hours and training shall be equivalent to current process used in Stryker and MRAP manufacturing	Operating experience	Met	Met	Met
Maintenance	Requirements for record keeping for storage, and clean up shall be equivalent to current process	Compare records	Met	Met	Met
Scale up capability	Identify additional equipment, if any, necessary to scale up process for full vehicle treatment	Operating experience and investigation	N/A	N/A	N/A

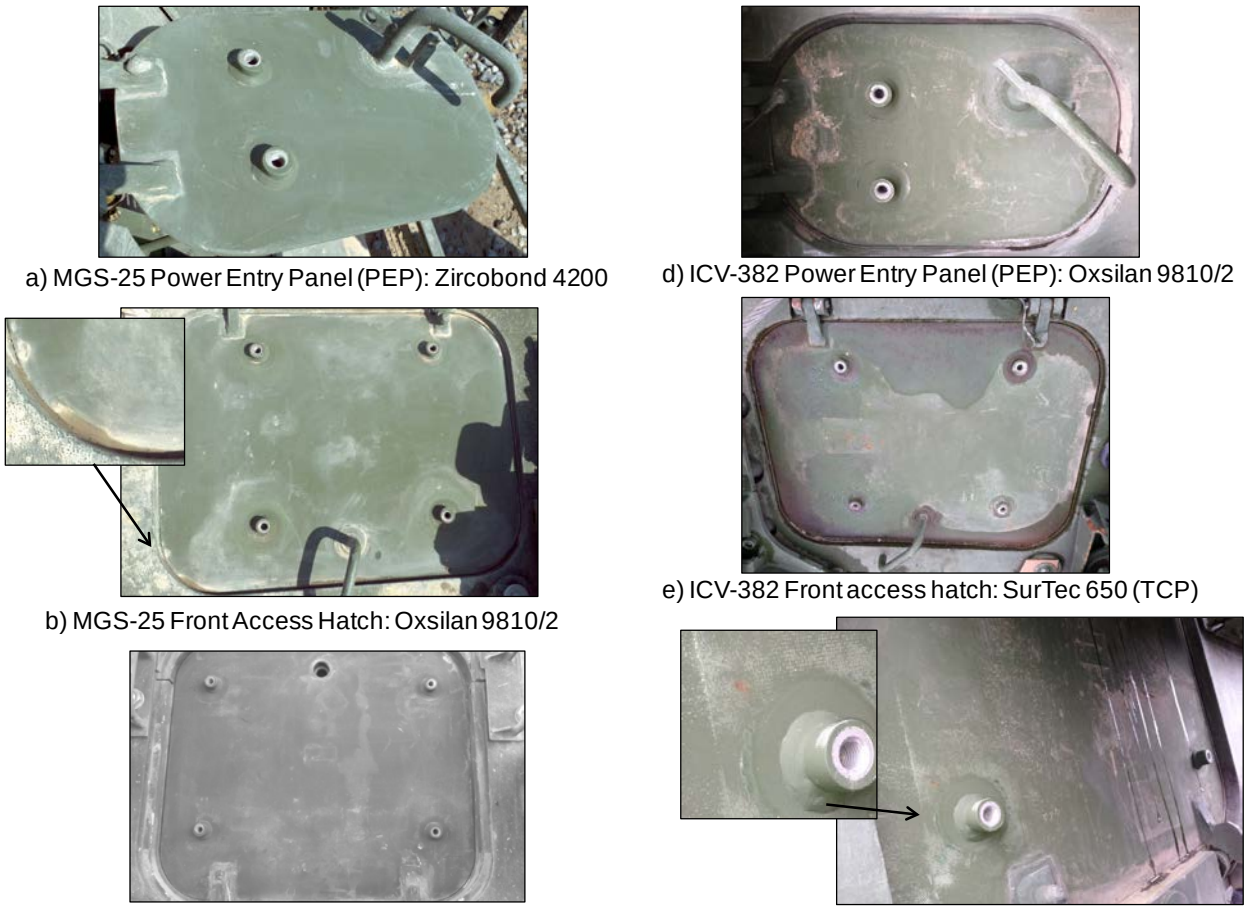
The issues that have been identified with scale up are discussed further in the next section on the MRAP demonstration. The Stryker parts were relatively small and scale up requirements could not fully be assessed. However, the primary concern in this demonstration was with the Zircobond 4200. The blotchy surface finish (Figure 9) will be a challenge for quality assurance engineers to determine if the resulting film is acceptable. SurTec 650 and Oxsilan 9810/2 had little to no color change on Stryker. The challenge here is to ensure that a large complex structure is completely and adequately wet for the required duration. It is believed that this is a challenge that can be overcome with proper equipment that can produce adequate flow of the product and capture and recycle the run-off.

The Strykers from the demonstration at ANAD were sent to JBLM. ARL made numerous inquiries and requests through Ms. Catherine Doherty (section 5-2) to locate and allow access to the vehicles for inspection without success. Eventually, ARL was able to coordinate through Ms.

Terry L. Austin, Pollution Prevention Program Manager, Installation Sustainability Coordinator, DPW-Env Div, JBLM. Ms. Austin, was a valuable resource and provided ARL with the following Stryker POC information:

> *MGS-0025:*
> POC: Mr. Douglas Saunders, Stryker Fielding Office, JBLM Logistics Support. Vehicle is deployed with 1-38 in Afghanistan. GDLS will photograph at the first opportunity.
>
> *ICV-0382 and MEV-0076:*
> POC: MSG Sanders, 7ID; 7ID has all SBCT at JBLM. SFC Jackson will locate vehicles and photograph treated parts.

ARL contacted each of the POCs and gained their cooperation. MEV-0076 and ICV-0382 were still located at JBLM, however MEV-0076 was unavailable for inspection or photographing at the time of the inquiry. Even though MGS-0025 was in Afghanistan, contractors were able to locate and provide ARL with the photographs necessary. Because of travel restrictions ARL was unable to physically inspect the hatches at JBLM, therefore the photographs were provided by the POCs listed above. The photographs of the treated hatches on MGS-0025 and ICV-0382 are shown in Figure 10. The photographs in Figure 10 indicate that, overall very little corrosion exists on the hatches after 31 months in service. Even with magnification of the high resolution photos, only the front access hatch shows some indication of wear that may have been caused by rubbing of the composite armor. In an email SFC Jackson-Smith provided an eye witness assessment on the condition of the hatches on ICV-0382. He wrote: “I didn't see any paint peeling on any of the hatches. There were a few small red rust areas on the side egress hatch up towards the top that appeared to me to be just light surface rust.” Although one of the hatches, Figure 10e appears to have some red rust near the edges, it is thought that the darker areas around the edge of that hatch may be dirt or clay that darkened when wet. The right side of that hatch, (dry side) is a light brown/tan color, not red. It’s possible the darker brown/red is just mud that collected under the composite armor outer. All of the hatches on both vehicles have some type of dirt/sand that collected under the composite armor. The only corrosion damage recognizable from the photographs is the spot in 6-7f, the side egress hatch mentioned by SFC Jackson-Smith. This can be rated using SSPC-VIS-2 as “rust grade 9-S (spot) less than 0.03 percent. At this time, the hatches are considered comparable to the base vehicle.



a) MGS-25 Power Entry Panel (PEP): Zircobond 4200

b) MGS-25 Front Access Hatch: Oxsilan 9810/2

c) MGS-25 Side Egress Hatch: SurTec 650 (TCP)

d) ICV-382 Power Entry Panel (PEP): Oxsilan 9810/2

e) ICV-382 Front access hatch: SurTec 650 (TCP)

f) ICV-382 Side Egress Hatch: Zircobond 4200

Figure 10. Photographs of hatches from Stryker vehicles MGS-25 and ICV-382 located at JBLM after 31 months in service.[13]

6.3 MRAP FULL SCALE DEMONSTRATION

Two electric pumps (depicted in the demonstration plan[14]) were used to apply the Oxsilan 9810/2 and DI rinse water. The flow rate of each of the pumps was measured to be 1.1 gpm. The process called for wetting the MRAP with the Oxsilan 9810/2, allowing it to dwell for 60-90 seconds, and rinsing with clean DI water. The process was carried out as close as practical with two applicators and one person blow-drying with compressed air. However, during the application of the pretreatment, the color of MRAP #1 began to change to a reddish hue within one minute. As the application progressed into the rinse and dry phase, a darker reddish-brown color appeared which looked similar to flash rusting on the steel surface. We estimated that about 90% of the vehicle was covered with this reddish-brown product. After some of the areas on the vehicle were fully dry, pull-off tape tests were conducted to determine the stability of the reddish-brown surface finish. Tape adhesion was very tight, with little or no reddish-brown product pulled off with the tape. In fact, in some cases, adhesion on the backing adhesive was pulled off of the test tape, indicating improved adhesion.

Figure 11 shows MRAP #1 after the application of the Oxsilan 9810/2. These results were completely unexpected and bear no resemblance to the surface finish that was achieved on the earlier demonstration on Stryker hatches treated Oxsilan 9810/2 at ANAD (see Figure 9).

Figure 11. MRAP #1 after application of with Oxsilan 9810/2, June 20, 2011.

During a meeting and conference call with Chemetall America's Product Manager on the evening of June 20, 2011, several possibilities for the unexpected results were discussed: 1) improper solution chemistry, 2) inadequate application rate [not enough flow], 3) surface contamination because MRAP #1 was not abrasive blasted within the required 24 hours prior to pretreatment, but rather 72 hours prior to treatment; and/ or blast media was contaminated.

A sample of the Oxsilan 9810/2 was taken from the drums and sent to the Chemetall laboratories for analysis. Chemetall laboratory determined that the solution chemistry was within their acceptable range. As a result, further tests by ARL were conducted to attempt to duplicate the earlier desired results from tests using the Oxsilan taken from the drums at Camp Lejeune, as well as to replicate the (undesirable) results obtained at Camp Lejeune.

HHA test panels were abrasive blasted with 60-grit aluminum oxide to SSPC-SP 5 to provide a clean surface prior to spray applying Oxsilan 9810/2. Beyond the initial abrasive blasted finish, the test panels were prepared to mimic different scenarios: Best case (maximum flow for 90 seconds), worst case (minimum flow for 30 seconds), and using contaminated substrates. The role of contaminants on the surface of the HHA prior to treatment with Oxsilan was examined two ways: 1) Test panels were sprayed with 3.5% sodium chloride (NaCl) solution and allowed to dry prior to applying Oxsilan 9810/2, and 2) freshly abrasive blasted panels were pre-exposed to a covered outdoor environment for 72 hours prior to applying the Oxsilan 9810/2 to mimic events at Camp Lejeune.

A significant change in the color was evident in the panels with surface contaminants. Figure 12 shows a comparison of a freshly blasted HHA panel (left), with panels that were treated with Oxsilan 9810/2 following 72 hours in an outside environment. The center panel was treated using minimal flow for 30 seconds, and right panel bathed for 90 seconds. The appearance of the center panel is very similar to what was seen on the MRAP during the demonstration at Camp Lejeune (Figure 11). Unique to these test panels vs. all the others was the spotting and streaking of the panel that was only allowed 30 seconds of dwell for the Oxsilan 9810/2; again, similar to

the MRAP. The surface is clearly contaminated, and it appears that the contamination has had an effect on the consistency and ability of the Oxsilan 9810/2 to react with the steel substrate. Although there was also a color change with the panel treated for 90 seconds, it did not resemble the MRAP results.

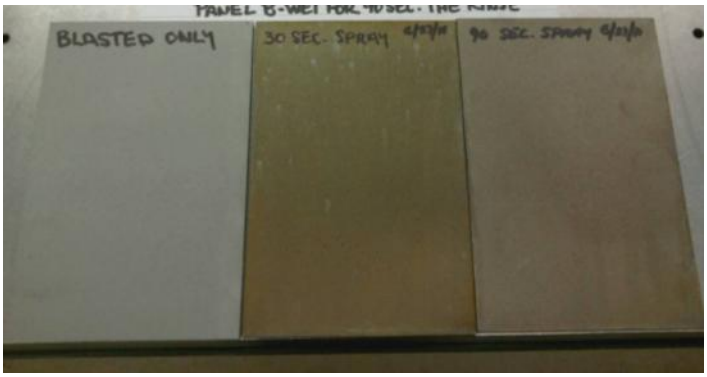


Figure 12. Comparison of HHA test panels that were weathered then pretreated with Oxsilan 9810/2.

These results indicate that a combination of events contributed to the undesirable appearance of the pretreated MRAP. The scale-up from laboratory sized and hatch sized parts to a full sized MRAP, was underestimated, resulting in inadequate flow of the applied Oxsilan 9810/2. We also believe that the 72 hours the bare surface of the vehicle was exposed to the environment led to some surface contamination which likely affected the reaction of the Oxsilan 9810/2 with the steel surface. We cannot rule out the possibility that the grit used for abrasive blasting the vehicles may have contained chlorides or other salts that would have also served to contaminate the steel surface. The laboratory tests and the previous demonstration of Strykers indicate that the Oxsilan 9810/2 must be applied to a freshly cleaned, abrasive blasted surface as soon as practical. Preferably within 2-4 hours of abrasive blasting. The flow rate used for the Oxsilan 9810/2 must be sufficient enough to keep the vehicle wet throughout the treatment. Rinsing with clean water should be done using adequate flow rate to thoroughly remove the un-reacted Oxsilan 9810/2. We are confident that the desired results can be achieved by following these recommendations.[15]

To prove our hypothesis, ARL worked closely with the MRAP-PMO to secure another demonstration of a limited scope. The MRAP-PMO agreed to provide one set of two rear doors from an MRAP variant for pretreatment using Oxsilan 9810/2. During this demonstration, we were very cognizant of the mistakes made at Camp Lejeune. Therefore, two improvements were made over the process used at Camp Lejeune: 1) the freshly abrasive blasted doors were pretreated with Oxsilan 9810/2 within 2 hours of blasting, and 2) adequate flow of the pretreatment solution was achieved by using a Yamada NDP-15 BPT pump with a 4.0 gpm rating. The Oxsilan treated surfaces of the door looked similar to the results seen at ANAD during the Stryker hatch demonstration. As can be seen in Figure 13, they appeared light grayish blue. No reddish/brown discoloration occurred anywhere on the door. The application of the Oxsilan was carried out similar to the Stryker hatches at ANAD. The surfaces were treated with Oxsilan less than 2 hours after abrasive blasting using a pump sprayer with adequate flow to keep the surfaces wet throughout the pretreatment process. This is added evidence that the

undesirable results from the MRAP demonstration at Camp Lejeune was a result of surface contamination, and/or inadequate flow rate of the Oxsilan 9810/2.

Figure 13. MRAP rear doors.
(left: freshly abrasive blasted, center: during application of Oxsilan 9810/2, right: drying)

The other of the two doors was pretreated with the DoD-P-15328 washprimer and both doors were then primed with MIL-DTL-53022, and topcoated with MIL-DTL-53039. An "X" was scribed near the bottom of each door and both doors were subjected to outdoor exposure at the Aberdeen Proving Ground (see Figure 14).

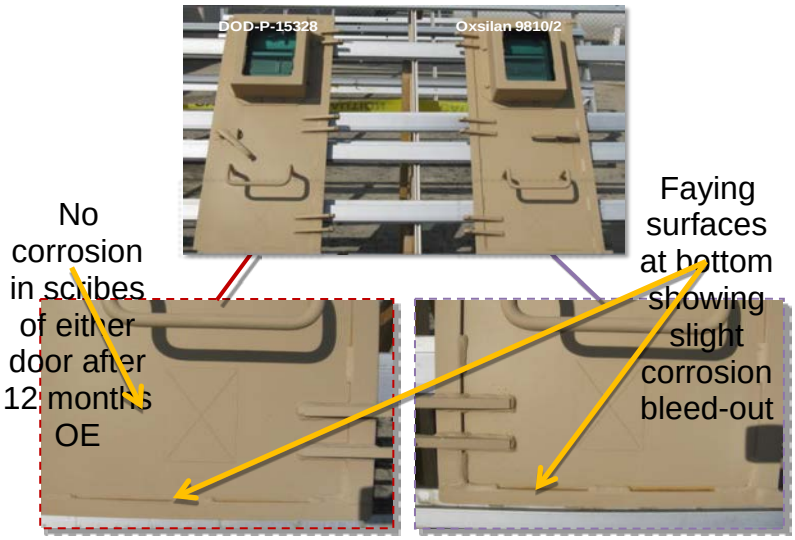


Figure 14. MRAP rear doors after 1 year in outdoor exposure at APG.
(Left: DoD-P-15328; on right: Oxsilan 9810/2)

After 12 months of exposure, there is no noticeable difference between the Oxsilan 9810/2 treated door and the door pretreated with standard washprimer. Only slight corrosion "bleed out" exists in the faying surfaces at the bottom of each door. The performance after 1 year is considered comparable thus far. The doors will remain in outdoor exposure at least until the scribed areas are rated a failure. Only the Oxsilan 9810/2 was assessed on MRAP and MRAP components. Table 8 presents the results of the demonstration assessments. For the same reasons given in Section 6.2, Demonstration on Stryker, Oxsilan 9810/2 met all of the primary

performance criteria when tested on the MRAP doors. The demonstration on the full MRAP was not carried out to the point where field testing was conducted and therefore the field performance was not assessed. The application of the product on the large MRAP platform was a challenge and may be more complicated to apply than the baseline Cheminhib 420. However, the superior performance validated in laboratory tests and on smaller components would be worth the added effort. The ease-of-use criterion for full vehicle application was considered not met because the Oxsilan 9810/2 process appears to be more sensitive to flow rates and substrate surface conditions than applying a simple flash rust inhibitor such as the baseline Cheminhib 420. Although it's been shown that surface contamination played a role, we also know that adequate flow rate of the Oxsilan 9810/2 is essential. We believe that optimum efficiency can be achieved by using a high volume halo-type sprayer with recirculation system for applying Oxsilan 9810/2. However, application can be performed using multiple high volume (4 gpm) sprayers in a catch basin for installations that perform rework of Armor vehicles.

Table 8. Validation methods and expected performance metrics for demonstrating Oxsilan 9810/2 on MRAP and MRAP doors.

Performance Criteria	Expected Performance Metric (Pre-Demonstration)	Performance Evaluation Method	Actual Performance (Post-Demonstration)	
Primary Performance Criteria			**Vehicle**	**Doors**
Product Testing	The performance of the alternative technology will meet or exceed the current process employed on MRAP during manufacturing as defined in the JTP in Appendix A.	Laboratory analysis and field testing	N/A	Met
Hazardous Materials	Maintains a hex-chrome free platform	Assessment of product constituents and previous studies	Met	Met
Hazardous Waste	Meets or exceeds current process used in MRAP manufacturing	Operating experience and assessments	Met	Met
Factors Affecting Technology Performance	Comparison of alternatives in identical operating conditions	Operating Experience	Not Met	Met
Secondary Performance Criteria			**Vehicle**	**Doors**
Ease of Use	Man hours and training shall be equivalent to current process used in MRAP manufacturing	Operating experience	Not Met	Met
Maintenance	Requirements for record keeping for storage, and clean up shall be equivalent to current process	Compare records	Met	Met
Scale up capability*	Identify additional equipment, if any, necessary to scale up process for full vehicle treatment.	Operating experience and investigation	Met	Met

* Although the Oxsilan was not applied successfully to a full MRAP, the subsequent investigation identified modifications and equipment necessary for scale up and were validated by the demonstration on MRAP doors.

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7.0 COST ASSESSMENT

This project is unique in that it has three technology areas being demonstrated and would have been expensive and time consuming to conduct a comprehensive cost assessment on each. An attempt was made to conduct the cost analysis during the MRAP demonstration at Camp Lejeune, but events occurring during that demonstration prevented us from making a reasonable cost and performance assessment. A cost assessment was performed for this project as it related to MRAP, but it is believed that the assumptions made apply to vehicles of similar size and material.

The work time required to disassemble, prepare and paint an MRAP is approximately 96 hours per vehicle at a cost of $13,440. The total paint used is estimated to be 4.9 gallons of MIL-DTL-0053022 primer at cost of $56.00/gallon and five gallons of MIL-DTL-53039 topcoat at a cost of $50.52/gallon resulting in a total cost of paint of $527 per vehicle. The total cost for repainting an MRAP is calculated at $13,967.00. The preparation steps and associated costs such as labor will all remain as stated above to implement any of the pretreatments. A modest additional cost per vehicle will be added as a result of the pretreatment step. Although, as mentioned earlier, a flash rust inhibitor step exists in the current process and therefore this assumption is considered conservative.

A conservative estimated surface area for an MRAP vehicle is 600 square feet. For the Oxsilan 9810/2 pretreatment, the cost depends on the type of system used for application and how the product is purchased. It can be spray applied with the runoff collected and disposed (spray-to-drain), or by conventional recirculating spray system. Oxsilan 9810/2 can be purchased either as a concentrate or as RTU premixed drums. The most cost effective approach is using a conventional recirculating spray system. In this case, the product cost is reduced to approximately $6.00 per vehicle when the Oxsilan is purchased as a concentrate and $12.00 per vehicle when the Oxsilan is purchased RTU. The most costly scenario is when Oxsilan 9810/2 is applied spray-to-drain. In this example, the cost per vehicle increases dramatically. Using the concentrate, the cost is $250.00-$400.00 per vehicle, and $1600.00-$2300.00 per vehicle for RTU. The pretreatment discussed here would increase the total cost of repainting the vehicle ($13.967.00) from 0.5% (recycling spray system) to 16% (spray-to-drain) depending on the pretreatment application process used.

The current coating system used for MRAPs has shown obvious deficiencies and as such each MRAP will likely need to be completely repainted on an average of every three years if the current processes remain in place. If improved coating systems that include a pretreatment are fully utilized from this demonstration, it is expected that the repaint interval will increase by a factor of two over the current baseline. This assumes that a third of the fleet will be repainted every year to maintain a consistent processing cycle. By implementing the new pretreatment system, the repaint cycle will likely double, thereby reducing the annual recoating costs by 50%. This reduction means that beginning after year four, only 1/6th of the MRAP fleet will need to be repainted, at a cost of $14,692/vehicle.

While number of Programmed Depot Maintenance (PDM) would not change, there would be a reduction in unscheduled maintenance events due to coating failure, and the extent of the repairs

performed during a RESET. The better condition and more well adhered the coating is at the time of RESET, the less labor intensive preparation for re-paint if needed. A 50% reduction is fair and reflects the reduction in overall labor costs for repairs due to coating failure and corrosion.

8.0 IMPLEMENTATION ISSUES

The implementation of non-hexavalent chrome steel pretreatments will be expedited by the recent publication of the reconstructed Federal specification TT-C-490F. This specification has been the overarching document referenced in dozens of military coating specifications and tens of thousands of military drawings for the cleaning and pretreatment of (only) ferrous substrates prior to the application of organic finishes such as CARC.

ARL recognized the synergy that existed between the TT-C-490 and the Environmental Security Technology Certification Program (ESTCP) funded project WP-200906 to examine alternative steel pretreatments. Several of the candidates evaluated were found to at least achieve the performance requirements and in some cases exceed the performance of existing hexavalent chrome pretreatments. The JTP and success criteria developed under WP-200906 became a basis for the performance specification. With this improved testing regimen, ARL can transition pretreatment materials that meet the established performance criteria through the Qualified Product Database. Details of TT-C-490 Revision F can be seen in the specification in Appendix C. The revisions enable many improvements to multiple alloy finishing operations within industry and the DoD. TT-C-490F and the associated QPD provides new commercially available technologies a pathway for implementation, encourages innovation and promotes low-energy and green technologies.

The revised document is being adopted by entire DoD and beyond (i.e., industry) for surface finishing of alloys – TACOM has adopted the language and principles of Objective Quality Evidence in the new TT-C-490F specification and has begun placing it in their Procurement Automated Data and Document System (PADDS clause) for pretreatments and CARC on all new contract requirements that requires all DoD and DoD contractors to follow the doctrine of the newly revised TT-C-490F specification.

The QPD has been populated by two of the products evaluated in WP-200906: SurTec 650 and Oxsilan 9810/2. These two spray applied pretreatments have been approved for abrasive blasted steel substrates. Currently there are two more WP-200906 candidates in the QPD approval pipeline: SurTec 580 and Zircobond 4200. The SurTec 580 is a non-hexavalent sealer for zinc phosphate and the Zircobond 4200 is another spray applied pretreatment for steel. Both have met or exceeded the success criteria and are in the application process for inclusion on the QPD.

While the demonstrations are very helpful in adjusting parameter for scale up, there is no demonstration requirement for approval to the QPD. However, the approval comes with qualifiers to guide the user. The Oxsilan9810/2 for example, has been shown to be an affective pretreatment for abrasive blasted steel when used in a consciously applied process in accordance with TT-C-490F and the manufacturer's technical data sheets and procedures. TT-C-490F also addresses the cleaning and preparation of the substrate prior to application of the pretreatment. There were three demonstrations for Oxsilan 9810/2, and combined, they served to validate the qualifiers and requirements in the QPD. All pretreatments listed on the QPD (including legacy pretreatments) include qualifiers associated with their processes.

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APPENDIX A

POINTS OF CONTACT

Point of Contact	Organization	Phone E-Mail	Role In Project
John Kelley	U.S. Army Research Lab	410-306-0837 jkelley@arl.army.mil	Lead PI
Thomas Braswell	U.S. Army Research Lab	410-306-0935 Thomas.e.braswell@us.army.mil	Co-PI
William Nickerson	NAVAIR	703-501-6226 William.nickerson@navy.mil	Co-PI
Luwam Hagos	NAVAIR	301-342-8159 Luwam.hagos@navy.mil	Co-performer
Amy Fowler	NAVAIR	301-342-0986 Amy.fowler1@navy.mil	Co-performer

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ESTCP Office
4800 Mark Center Drive
Suite 17D08
Alexandria, VA 22350-3605

(571) 372-6565 (Phone)

E-mail: estcp@estcp.org
www.serdp-estcp.org