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Preliminary Assessment of a Chemical Pretreatment on Titanium

by John Kelley, Thomas Considine, Thomas Braswell, Tyler Sexton, and David Orsini

NOTICES

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14. ABSTRACT
The need to lighten next-generation vehicles will likely require the use of titanium (Ti) alloys to meet weight requirements. Under the regulation AR 750-1, all Army-based ground equipment is required to have a full Chemical Agent Resistant Coating (CARC) system. The typical CARC system consists of a pretreatment in direct contact with the substrate properly applied in accordance with TT-C-490 prior to applying the epoxy primer and polyurethane topcoat. The current practice for painting Ti involves abrasive blast and applying the CARC system directly to metal. Moreover, applying the CARC system to these multimetal assets can be complicated, since it may not be practical to coat the components and parts separately. This limited-scope investigation examines Bonderite M-NT 7400, a widely used, TT-C-490 qualified product database-listed pretreatment approved for steel and aluminum substrates. This work tests Bonderite M-NT 7400 on Ti using adhesion and accelerated corrosion test methods. The results show Ti surface prepared without a pretreatment and those with the Bonderite M-NT 7400: both provided a good initial and post-ASTM B117-exposed coating adhesion. This suggests that Ti surfaces can be effectively pretreated with adjacent non-Ti surfaces during the same repair/reset operations without masking.

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1. Introduction

The use of titanium (Ti) alloys in Army ground vehicles has been increasing in recent years and is expected to continue to grow in the future. Previous Stryker variants contained mixed-metal assemblies, which include Ti alloys. The next-generation tank will likely require the use of Ti alloys to meet weight requirements. Under the regulation AR 750-1,[1] all Army-based ground equipment is required to have a full Chemical Agent Resistant Coating (CARC) system. The CARC system is defined in MIL-DTL-53072.[2] The typical CARC system comprises 1) a conversion coating or pretreatment in direct contact with the substrate properly applied in accordance with (IAW) TT-C-490,[3] followed by 2) an epoxy primer IAW MIL-DTL-53022[4] or MIL-DTL-53030[5], and 3) the polyurethane-based topcoat IAW MIL-DTL-53039[6] or MIL-DTL-64159.[7] Completed assemblies, whole platforms, and vehicles instead require a spray-applied pretreatment IAW TT-C-490 prior to applying the epoxy primer and polyurethane topcoat. However, the current practice for painting Ti alloys on Army vehicles is to abrasive blast and apply the CARC system direct-to-metal (DTM). Moreover, applying the CARC system to these multimetal assets can be complicated, since it may not be practical to coat the components and parts separately.

Because of the growing interest in Ti alloys for armored systems, and the complexities of properly coating mixed metal assemblies, a need exists for common pretreatment methods effective on mixed-metal assemblies that include Ti alloys. Steel and aluminum used on these vehicles typically require TT-C-490 qualified chemical pretreatments. This limited-scope investigation examines a commonly used TT-C-490 qualified product database (QPD)-listed chemical pretreatment on a Ti alloy. The pretreatment Bonderite M-NT 7400, approved for steel and aluminum substrates, was evaluated on Ti using adhesion and accelerated corrosion test methods. All Ti substrates were abrasive blasted to clean off the native oxide prior to coating**.** Although a pretreatment effective on Ti alloys in lieu of abrasive blasting would have added benefits including manufacturing cost savings, it was beyond the scope of this preliminary assessment.

2. Experimental

2.1 Samples Preparation

All samples were abrasive blasted to a 1-mil finish using aluminum oxide grit. One set was pretreated using a TT-C-490 Type IV approved pretreatment, Bonderite M-NT 7400, and subsequently coated with MIL-DTL-53022 Type IV epoxy primer.

A second set of panels was coated DTM with MIL-DTL-53022 Type IV epoxy primer. All panels were allowed to fully cure over a 2-week period in ambient laboratory conditions.

2.2 Dry Tape Adhesion

Tests were conducted at room temperature as defined in ASTM D3359[8] Method B. An area of the panel free of blemishes was selected. Using a sharp cutting tool, six parallel cuts at 2-mm spacing through the paint film to the metal substrate were made. A second set of six cuts were made perpendicular to the initial set (Fig. 1). Both cuts were made ensuring that they were sufficiently long enough to make a complete set of 6 × 6 grid lines. Three data points per coupon were collected. The grid lines were then brushed lightly to remove any detached flakes or ribbons of coating. A complete lap of tape was removed from the roll and discarded prior to removing the length of tape used for the test. A length of tape was removed at a steady (that is, not jerked) rate and cut about 75 mm (3 inches) long. The center of the tape was placed over the grid and the area of the grid smoothed into place by a finger. To ensure good contact with the film, the tape was rubbed firmly with the eraser on the end of a pencil. The tape was then removed by grasping the free end and rapidly pulling back upon itself at as close to an angle of 180° to the surface of the panel as possible. Following the tape pull-off, each grid was rated using the classification in ASTM D3359 Method B, as shown in Fig. 1. To further ensure accuracy, a second rating was made by another US Army Combat Capabilities Development Command Army Research Laboratory researcher. The two ratings were averaged for the final rating.

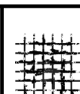
Surface of cross-cut area from which flaking has occured. (Example for 6 parallel cuts).	None					Greater than 65%
Classification	5	4	3	2	1	0

Fig. 1 ASTM D3359 method B: crosshatch adhesion ratings

2.3 Pull-off Adhesion

Pull-off adhesion measurements were performed IAW ASTM D4541.[9] A loading fixture commonly referred to as a "dolly" is adhered normal to the coating surface using cyanoacrylate adhesive. After allowing the adhesive to cure for 24 h in laboratory ambient conditions, the attached dolly is segregated from the rest of the coating by cutting into the coating around the dolly with a similarly sized hole saw. The dolly was then inserted into the hydraulic pull-test apparatus. The load applied by the apparatus was gradually increased and monitored on the gauge until the dolly was detached. The failure value (in pounds per square inch [psi]) and the failure mode were characterized and recorded.

2.4 ASTM B117 Neutral Salt Fog Testing

Two sets of Ti coupons were tested in neutral salt fog using an Auto Technology Company standard salt fog chamber (Fig. 2).[10] The panels were held in polypropylene trays tilted at an angle no more than 15° from the vertical with the primary surface facing upward. One of the panel sets was pretreated with Bonderite M-NT 7400 and coated with MIL-DTL-53022 Type IV primer, while the other set was coated with Type IV primer applied DTM (no pretreatment). Both sets were X-scribed prior to being placed in the chamber IAW DEVCOM Army Research Laboratory procedure.[11] Neutral salt fog conditions are 95 °F with saturated humidity and an atomized fog of a certified 5% NaCl solution. Daily fog deposit volumes, pH, and other records are available upon request. Test panels were evaluated IAW ASTM D1654.[12] Boths sets of panels were exposed for a period of 2160 h. Upon completion of testing, the panels were removed from the chamber, rinsed in deionized water, air dried, and examined for any coating delamination. Adhesion tests were also performed on these postexposed test panels.

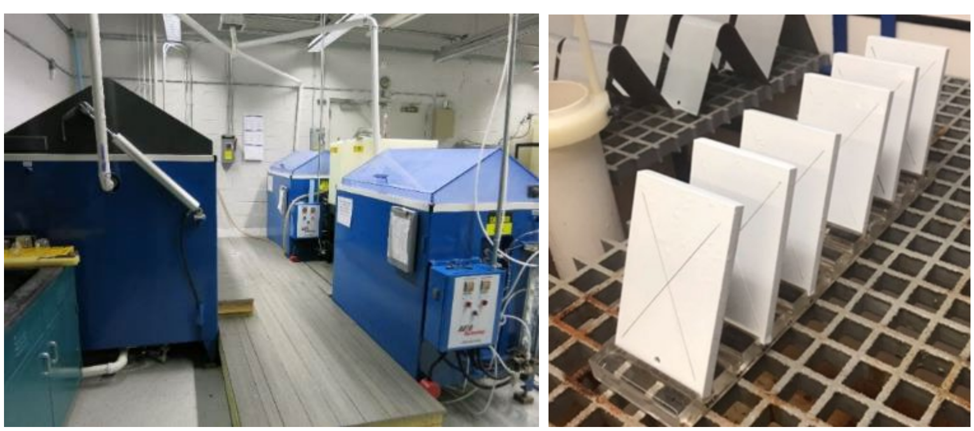


Fig. 2 Salt fog chambers (left) and coated and scribed Ti panels in chambers (right)

3. Results and Discussion

Images were taken weekly of all panels over the course of the 2160 h of ASTM B117 exposure. Both DTM and Bonderite M-NT 7400 panels performed well. No delamination or pitting was observed on any of the panels over the duration of testing. As illustrated in Fig. 3, all coatings appeared intact along scribes and even near edges where coating failure is common.

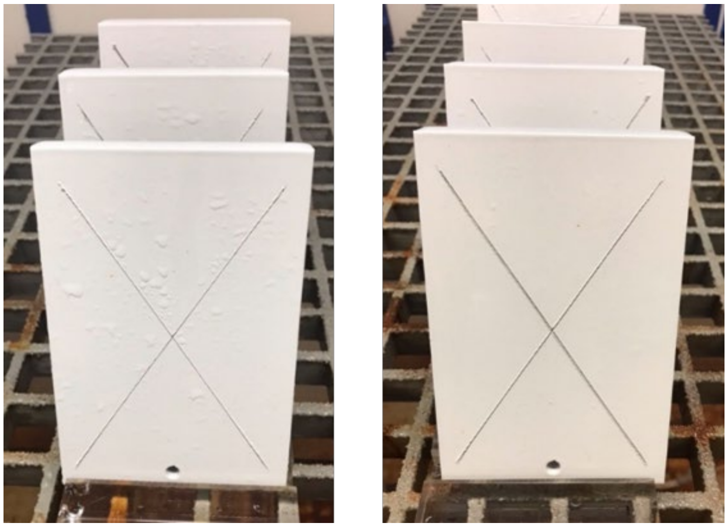


Fig. 3 Coated and scribed Ti panels in ASTM B117. The baseline DTM (left) and the Bonderite M-NT 7400 pretreated panels (right).

To assess the initial adhesion strength, an identical set of DTM and Bonderite M-NT 7400 pretreated and coated panels was tested for both dry tape adhesion and pull-off adhesion. The results in Table 1 show that both the abrasive blasted only and Bonderite M-NT 7400 surface conditions provided a good base for coating application. Both surface conditions achieved a 5B rating (see rating chart in Fig. 1). The pull-off adhesion strength was very good for both surface conditions. DTM provided slightly better (6% higher) initial pull-off adhesion than the Bonderite M-NT 7400. Figures 4 and 5 show the crosshatch results. Note no chipping or peeling within the crosshatch section justifying the 5B rating.

Table 1 Coating adhesion of panels that were not exposed to ASTM B117

Surface treatment	Dry Tape adhesion ASTM D3359	Pull-off adhesion ASTM D4541 (psi)
DTM	5B	2451
Bonderite M-NT 7400	5B	2296

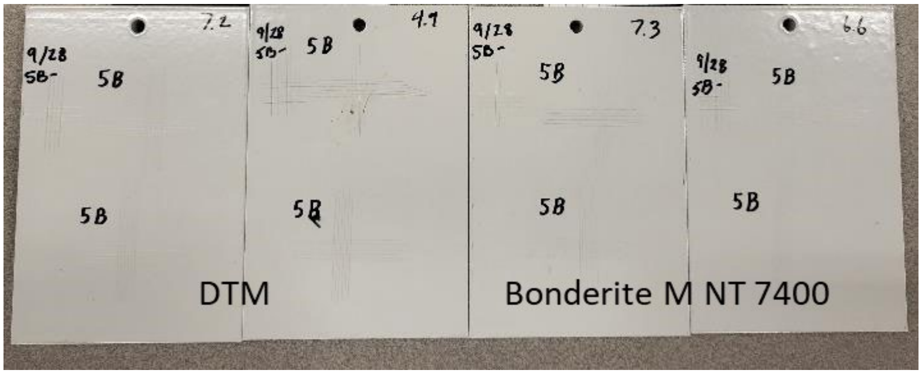


Fig. 4 Dry crosshatch adhesion tests per ASTM 3359

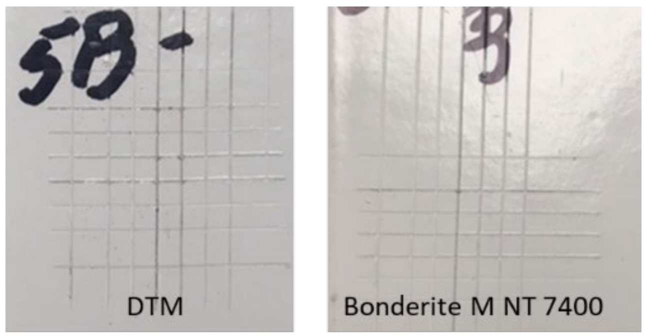


Fig. 5 Magnified (2×) view of crosshatch adhesion results from unexposed panels to illustrate adhesion rating

To determine the residual adhesion of the coatings following exposure, the adhesion tests were repeated on the panels exposed through 2160 h of ASTM B117 and allowed to dry for 7 days before adhesion tests were performed. Postexposure tape-adhesion ratings were slightly lower at 4B versus the initial 5B. This was the case for both surface preparation methods. The magnified view of the crosshatch in Fig. 6 shows some lifting along the scribe intersection likely due to moisture uptake during exposure. The captured moisture will cause the coating to expand slightly, creating stress in the coating. The stress is relieved by the scribe, allowing the coating to lift slightly. Nevertheless, pull-off results of the postexposure panels (Table 2) remained relatively unchanged from the initial pull-off results. Interestingly, the average adhesion strength of the postexposed panels was slightly higher than the initial pull-off results in Table 1. In some cases, individual adhesion strength exceeded the 3000-psi limit of the pull-off adhesion apparatus.

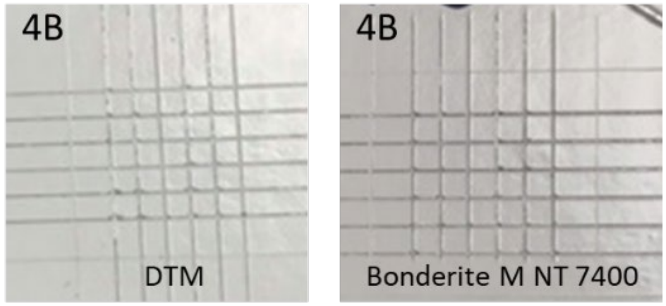


Fig. 6 Magnified (2×) view of crosshatch adhesion results following 2160 h of ASTM B117 exposure

Table 2 Coating adhesion of panels following 2160 h of ASTM B117 exposure with cyanoacrelate

Surface treatment	Dry tape adhesion ASTM D3359	Pull-off adhesion ASTM D4541 (psi)
DTM	4B	2560
Bonderite M-NT 7400	4B	2322

This is seen in Figs. 7 and 8 with both the DTM and Bonderite M-NT 7400 prepared panels. The areas of the panel marked as 3000 psi still have the dolly adhered to the coating. In all cases, the mode of failure was a failure of the adhesive. The adhesion requirements as per MIL-PRF-53072[13] are listed as 1200 psi, and these results indicate that the coating performed well beyond the specification parameters. However, because the consistent mode of failure was the adhesive, a second round of pull-offs was performed using a two-part epoxy adhesive known to have a much greater tensile strength. In this case, almost every pull exceeded the 3000-psi limit of the hydraulic tester again. Those few dollies that did break free from the surface of the panel showed mostly failure of the adhesive and 10%–20% cohesive failure of the primer. Given the intense stresses well outside the adhesion requirements, none of these failure modes are considered abnormal or cause for concern.

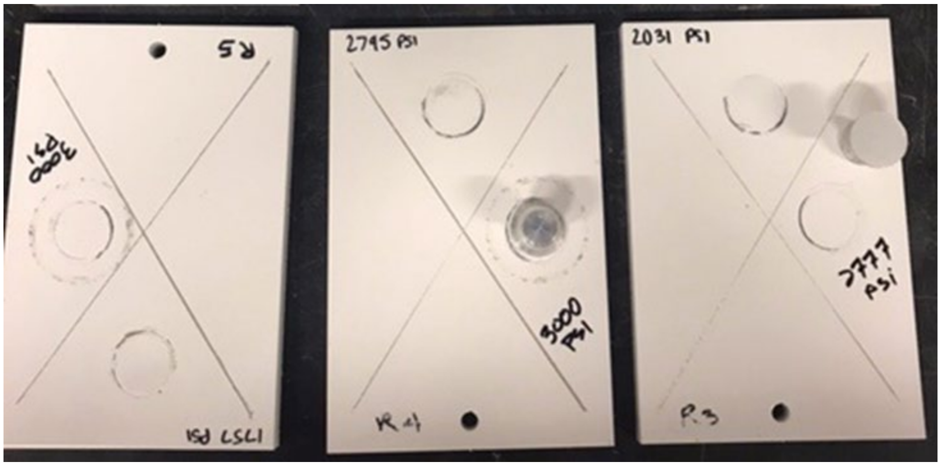


Fig. 7 Pull-off adhesion of postexposed DTM MIL-DTL-53022 coated Ti test panels

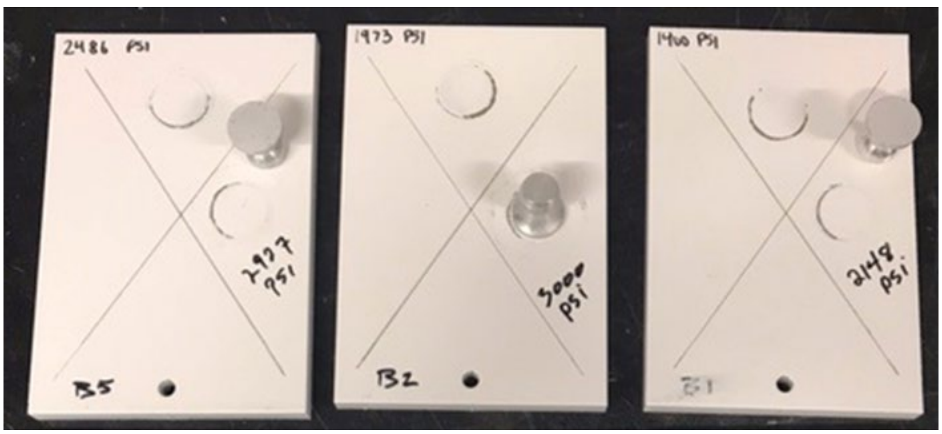


Fig. 8 Pull-off adhesion of postexposed Bonderite M-NT 7400 pretreated and MIL-DTL-53022 coated Ti test panels

4. Conclusions and Recommendations

Ti alloys are extremely corrosion resistant and therefore do not typically require additional corrosion protection beyond adequate surface preparation for subsequent coating. The purpose of this limited-scope investigation was to determine if a chemical pretreatment designed for steel and aluminum, and qualified to TT-C-490, would have any detrimental effects on the adhesion of the organic coating system to Ti substrate.

The results show that the Ti surface prepared without a chemical pretreatment, and those with the Bonderite M-NT 7400, both provided good initial adhesion of the organic coating. This held true for all test panels through 2160 h of ASTM B117 exposure. This suggests that Ti surfaces can be effectively pretreated along with adjacent non-Ti surfaces (e.g., steel and aluminum) during the same repair/reset operations using chemical pretreatments approved for multimetal assemblies. No masking of the dissimilar metals would be necessary during pretreatment.

It is recommended that a more comprehensive evaluation be performed IAW TT-C-490 using QPD-listed pretreatments to gain full qualification on Ti alloys, especially those that are part of multimetal assemblies where galvanic corrosion may be of concern. Various surface profiles, primers, and exposure conditions should also be investigated. Additionally, understanding the effectiveness of pretreatments on Ti with varying stages of surface oxidation will help determine pretreatment boundaries for pretreating Ti, and ultimately aid in revising TT-C-490.

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List of Symbols, Abbreviations, and Acronyms

ARL	Army Research Laboratory
ASTM	American Society for Testing and Materials
CARC	Chemical Agent Resistant Coating
DEVCOM	US Army Combat Capabilities Development Command
DTM	direct-to-metal
IAW	in accordance with
NaCl	sodium chloride
psi	pounds per square inch
QPD	qualified product database
Ti	Ti

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