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ARMY RESEARCH LABORATORY

Long-Term Accelerated Corrosion and Adhesion Assessment of CARC Prepared Aluminum Alloy 5059-H131 Using Three Different Surface Preparation Methods

by Brian E. Placzankis and Amy L. Hilgeman

ARL-TR-4547 August 2008

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Army Research Laboratory
Aberdeen Proving Ground, MD 21005-5069

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Brian E. Placzankis
Weapons and Materials Research Directorate, ARL

Amy L. Hilgeman
U.S. Naval Air Warfare Center

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14. ABSTRACT
Aluminum alloy 5059-H131 panels rolled to 0.25-in thickness were prepared using chemical agent resistant coating (CARC) for evaluation under three different pretreatment conditions. The pretreatment conditions were abrasive blasted; a nonchromate pretreatment, Alodine 5200; or a commercial trivalent chromate pretreatment, Metalast TCP-HF. The primer used was MIL-DTL-0053022C and the topcoat was MIL-DTL-53039. Corrosion resistance was evaluated under GM 9540P and ASTM B 117 neutral salt fog (NSF) methods for durations well beyond what is typical for most substrates and coating systems. The extended durations were one full calendar year for NSF and 400 cycles for GM 9540P. Adhesion was assessed under dry conditions using ASTM D 4541 pull-off and under wet conditions using ASTM D 3359A. Prior to coating, the conversion coated surfaces were compared for uniformity and color vs. identically prepared AA5083 samples to determine whether or not the alloy differences will warrant modifications to current pretreatment processes.

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1. Introduction

Current U.S. Army and Marine Corps vehicle deployments require improved survivability and lightweight armor designs to maintain mission performance. Historically, AA5083-H131 has been used in armor systems such as the M113, the M109, and the U.S. Marine Corps (USMC) amphibious assault vehicle in accordance with the MIL-DTL-46027J (*1*) for its combination of desirable traits such as lighter weight, ease of manufacturing via welds, excellent performance against fragmentation-based threats, and excellent corrosion resistance. As threat levels have increased, more recently designed aluminum armor-based systems such as the M2 Bradley and the USMC expeditionary fighting vehicle have migrated to higher strength Al alloys such as AA7039 (*2*), AA2219 (*3*), and AA2519 (*4*). These higher strength alloys provide better ballistic protection vs. armor piercing threats and can also derive lighter weight hull designs from their increased yield and tensile strengths. Unfortunately, corrosion resistance for these alloys significantly decreases vs. AA5083-H131, whether by stress corrosion cracking in AA7039 or from pitting and exfoliation in AA2519. This decrease in corrosion resistance has had profound effects on maintenance, the application of coatings, as well as environmental implications from the need to compensate for corrosion deficiencies through the use of hexavalent chromium-based protection schemes.

Aluminum alloy 5083-H131 has many desirable traits, but is lacking higher strength, thus imparting decreased survivability vs. armor-piercing (AP) threats. An alternate aluminum alloy that delivered the positive characteristics of AA5083-H131 with increased mechanical properties and improved performance against AP threats would represent an ideal choice for consideration as an aluminum armor material for production and repair of new and existing systems.

One possible alloy that could fulfill this role is AA5059-H131, an alloy that is produced by Aleris International Inc.[*] in Koblenz, Germany. As with AA5083, AA5059 is a magnesium-based, non-heat-treatable aluminum alloy that is strengthened via mechanical strain hardening. As a result of the strain hardening process, the 5000 series alloys receive the “H” designation rather than the “T” designation that is typical for heat-treatable Al alloys. The 5059 alloy contains greater amounts of Mg than 5083 as well as some additions of Zn and Zr for grain refinement. Tables 1 and 2 compare the compositional and mechanical properties of AA5083, AA5059, and other military specification aluminum armors. Marine grade tempers of AA5059 such as H116 and H321 have been commercially available and in use for many years on yachts, ferries, and catamarans. However, limited information was known regarding the hard H131 temper that would be applicable for armor plate.

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[*] Aleris International, Inc., 25825 Science Park Drive, Suite 400, Beachwood, OH 44122-7392.

Table 1. Chemical composition requirements for qualified military specification aluminum armor alloys (%).

Element	5083	5456	5059	7039	2219	2519
Silicon	0.40 max	0.25 max	0.50 max	0.30 max	0.20 max	0.25 max
Iron	0.40 max	0.40 max	0.50 max	0.40 max	0.30 max	0.30 max
Copper	0.10 max	0.10 max	0.40 max	0.10 max	5.8–6.8	5.3–6.4
Manganese	0.4–1.0	0.5–1.0	0.60–1.2	0.10–0.40	0.20–0.40	0.10–0.50
Magnesium	4.0–4.9	4.7–5.5	5.0–6.0	2.3–3.3	0.02 max	0.05–0.40
Chromium	0.05–0.25	0.05–0.20	0.30 max	0.15–0.25	—	—
Zinc	0.25 max	0.25 max	0.40–1.5	3.5–4.5	0.10 max	0.10 max
Titanium	0.15 max	0.20 max	0.20 max	0.10 max	0.02–0.10	0.02–0.10
Zirconium	—	—	0.05–0.25	—	0.10–0.25	0.10–0.25
Vanadium	—	—	—	—	0.05–0.15	0.05–0.15
Others (each)	0.05 max	0.05 max	0.05 max	0.05 max	0.05 max	0.05 max
Others (max)	0.15 max	0.15 max	0.15 max	0.15 max	0.15 max	0.15 max
Aluminum	Remainder	Remainder	Remainder	Remainder	Remainder	Remainder

Table 2. Minimum mechanical requirements for military specification aluminum armor alloys.

Property	5083	5456	5059	7039	2219	2519
Yield stress (ksi) (0.2% offset min)	35	35	44	51	46	58
Ultimate stress (ksi)	45	45	57	60	62	68
Percent elongation	8	8	8	9	7	7

A Foreign Comparative Test Proposal was submitted in 2004 to the Office of the Secretary of Defense to examine and verify AA5059-H131 for possible use as an armor repair material for battle damaged or cracked armor plate sections on the M2 Bradley hull. Although the project was approved, it was initially unfunded for FY05 but eventually funded for FY06. The project goals were to verify the material performance in ballistics (*5*), blast resistance, weldability, corrosion due to sensitization, general corrosion and chemical agent resistant coating (CARC) compatibility, and to update or create a military specification to include the alloy if proven successful.

To date, all of the evaluations have been highly successful except for decreased resistance to sensitization. A recent study by Field and Wong of NAVSEA (*6*) found the AA5059 to be more susceptible to sensitization than AA5083. However, AA5059 was no more susceptible than AA5456, an armor alloy that in addition to AA5083 is fully compliant under the MIL-DTL-46027 armor specification. As a result of easily meeting or exceeding the balance of the remaining project goals, AA5059 was included in a revised edition of the armor military specification, MIL-DTL-46027K (*7*), effective 31 July 2007.

2. Experimental Procedure

The purpose of this study was to verify the performance and compatibility of the AA5059 alloy when coated with variations of CARC coating systems most likely to be used at the original equipment manufacturer production facilities and to determine what differences or unforeseen issues may exist when 5059 is used. The focus areas for these evaluations included ASTM B 117-90 (*8*) neutral salt fog (NSF) corrosion, GM 9540P (*9*) cyclic corrosion, ASTM D 4541 (*10*) pull-off adhesion, and ASTM D 3359A (*11*) wet adhesion. Test panels of AA5059-H131 measuring 4 × 6 in were machined from 0.25-in-thick plate. Based upon feedback from Project Manager Heavy Brigade Combat Team (*12*) (PM-HBCT) and BAE Systems (*13*), a coatings matrix based upon likely production line scenarios was devised. The matrix consisted of three different surface preparations: abrasive blast, Alodine 5200,[*] and Metalast TCP-HF[†] with one primer/topcoat combination. The Metalast pretreatment is a variant of the U.S. Naval Air System Command (NAVAIR) developed trivalent chromate pretreatment (TCP) formulation and fully complies with both MIL-DTL-5541F (*14*) and MIL-DTL-81706B (*15*) as a type II nonhexchrome variation. It is listed on the MIL-DTL-81706B Qualified Products List (*16*) (QPL). The abrasive blast media was 85-grit almandite garnet applied at 110 psi. Prior to applying the conversion coating, test panels were cleaned using a nonsilicated, nonchromated, mildly alkaline aluminum cleaner and deoxidized using a nonchromated ferrous sulfate/nitric acid-based desmutter. Metalast TCP-HF (50%) and Alodine 5200 were spray applied for 10 min at ambient conditions. All test panels were given a final deionized water rinse following completion of conversion coating application. The process flow diagram used for Alodine 5200 and Metalast TCP-HF is illustrated in figure 1. The primer and topcoat combination used was manufactured by Hentzen[‡] and consisted of the solvent-based MIL-DTL-0053022C (*17*) primer and a 1-lb/gal volatile organic compound (VOC) 686 tan pigmented topcoat formulation compliant with the new class of type II low VOC and polymeric bead flattened topcoats described under MIL-DTL-53039B (*18*).

After 2 weeks of cure time, test panels for NSF (ASTM B 117) and cyclic accelerated corrosion (GM 9540P) were scribed with an "X" using a carbide-tipped, hardened steel scribe and placed into their respective chambers. A Harshaw Model 22 test chamber was used for NSF testing, and an Attotech Model CCT-NC-20 was used for cyclic testing. The NSF operating parameters were in accordance with ASTM B 117, 95 ºF with saturated humidity and atomized fog of 5% NaCl solution. The cyclic accelerated corrosion test was in accordance with GM 9540P, consisting of 18 separate stages that included the following: saltwater spray, humidity, drying, ambient, and heated drying. The environmental conditions and duration of each stage for one complete cycle

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[*] Alodine 5200 is a registered trademark of KGaA (Headquarters), Henkelstrasse 67, 40589 Düsseldorf, Germany.

[†] Metalast TCP-HF is a registered trademark of Metalast International, Inc., 2241 Park Place, Suite C, Minden, NV 89423.

[‡] Hentzen Coatings, Inc., 6937 W. Mill Road, Milwaukee, WI 53218.

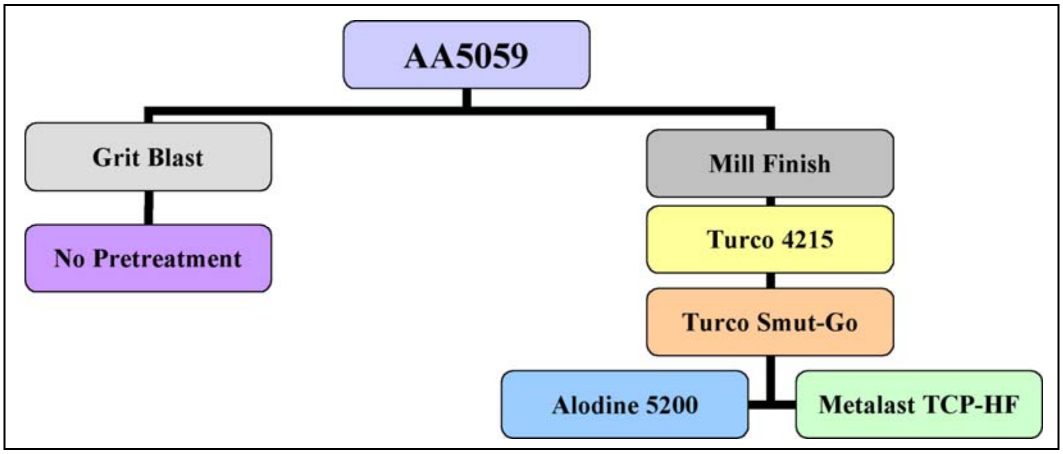

Figure 1. AA5059 test panel conversion coating processing flowchart.

are provided in table 3. The standard 0.9% NaCl, 0.1% $CaCl_2$, 0.25% $NaHCO_3$ test solution was used. In addition, the cyclic chamber was calibrated with standard steel mass loss calibration coupons, as described in the GM 9540P test specification, and an uncoated 4 × 6 in 5059-H131 test panel was added as a control to assess the overall corrosion resistance of the unprotected alloy.

Table 3. GM 9540P cyclic corrosion test details.

Interval	Description	Time (min)	Temperature (±3 °C)
1	Ramp to salt mist	15	25
2	Salt mist cycle	1	25
3	Dry cycle	15	30
4	Ramp to salt mist	70	25
5	Salt mist cycle	1	25
6	Dry cycle	15	30
7	Ramp to salt mist	70	25
8	Salt mist cycle	1	25
9	Dry cycle	15	30
10	Ramp to salt mist	70	25
11	Salt mist cycle	1	25
12	Dry cycle	15	30
13	Ramp to humidity	15	49
14	Humidity cycle	480	49
15	Ramp to dry	15	60
16	Dry cycle	480	60
17	Ramp to ambient	15	25
18	Ambient cycle	480	25

In order to quantify the corrosion, all panels were numerically rated for scribe corrosion creepback damage at scheduled intervals in accordance with method ASTM D 1654 (*19*). The NSF panels were rated weekly for the first 3 weeks (504 hr) and then every 3 weeks henceforth until 4032 hr. The longer term NSF rating intervals after 4032 hr were every 2016 hr, concluding with a final assessment at 8760 hr (1 year). The cyclic panels were rated after 10 cycles, 20 cycles, and then every 20 cycles thereafter until 200 cycles. As with salt fog, extended ratings used longer cycle intervals until the exposure conclusion at 400 cycles. To facilitate easier viewing, color codes were assigned based upon ranges of ASTM D 1654 ratings.

Table 4 depicts the ASTM D 1654 rating parameters and also defines the colors and their respective rating ranges. Upon measurements of scribe creepback ratings of 8 or less, accompanying images were obtained via 600-dpi digital flatbed scans and then subsequently rescanned at every remaining inspection interval until the conclusion of the experiment.

Table 4. Evaluation and color coding of scribed coated specimens subjected to corrosive environments (ASTM D 1654).

Rating of Failure at Scribe (Procedure A)		
Representative Mean Creepage From Scribe		**Rating No.**
(mm)	**(in)**	
0	0	10
Over 0 to 0.5	0 to 1/64	9
Over 0.5 to 1	1/64 to 1/32	8
Over 1 to 2	1/32 to 1/16	7
Over 2 to 3	1/16 to 1/8	6
Over 3 to 5	1/8 to 3/16	5
Over 5 to 7	3/16 to 1/4	4
Over 7 to 10	1/4 to 3/8	3
Over 10 to 13	3/8 to 1/2	2
Over 13 to 16	1/2 to 5/8	1
Over 16 to more	5/8 to more	0

Wet adhesion was performed in accordance with Method 6301 of Federal Test Method Standard 141 (*20*) and rated in accordance with ASTM D 3359A. Previous studies (*21*) on similar systems had indicated good performance from the conversion coatings in this study. Therefore, a more severe form of the wet adhesion procedure was chosen. Two replicates of each coating system were immersed in deionized water undisturbed for 1 week at 150 °F. After 1 week, the panels each were removed from the bath, patted dry with a lint-free wipe, then scribed and tested for wet tape adhesion. The rating system for ASTM D 3359A is described in table 5.

Table 5. Wet adhesion rating – method ASTM D 3359A.

Rating	**Description of Coating After Tape Removal**
	Method A – Wet Adhesion
5[a]	No peeling or removal.
4	Trace peeling or removal along scribes.
3	Jagged removal along scribes up to 1/16 in (1.6 mm) on either side.
2	Jagged removal along most of the scribes up to 1/8 in (3.2 mm) on either side.
1	Removal from most of the area between the scribes under the tape.
0	Removal beyond the area of the scribes.

[a] Passes military performance criteria.

Pull-off adhesion measurements assessing the performance of the coating system, surface preparation, and/or pretreatments were performed in accordance with ASTM D 4541. An Elcometer Model 108 Hydraulic Adhesion Tester was used for this procedure. In addition to being a more quantitative test method, pull-off adhesion was also less prone to inevitable human influences in testing such as variations in pressure applied during scribing as well as interpretation and perception of results. For the pull-off adhesion test, a loading fixture commonly referred to as a “dolly” was secured normal to the coating surface using an adhesive. The first adhesives used were several different brands of standard cyanoacrylate in a variety of viscosities. After allowing the adhesive to cure for 24 hr at 25 ºC and 65% relative humidity (table 6), the attached dolly was inserted into the test apparatus. The load applied by the apparatus was gradually increased and monitored on the gauge until a plug of coating was detached. Though it is not common practice to vary adhesives, the multitude of cyanoacrylate formulations were used due to repeated bonding difficulties to the steel surface of the test dollies. Due to these unforeseen adhesion problems with the standard cyanoacrylates, an alphacyanoacrylate ester formulation[*] was chosen and successfully substituted. Coating failure tensions (in psi) were then successfully recorded with the accompanying failure modes characterized. The pull-off test apparatus and dolly configuration are illustrated in figure 2. For pull-off data to be valid, the specimen substrate must be sufficiently thick to ensure that the coaxial load applied during the removal stage does not distort the substrate material and cause a bulging or “trampoline effect.” If a thin specimen is used, the resultant bulge causes the coating to radially peel away outwards from the center instead of being uniformly pulled away in pure tension. Thus, using a thinner substrate results in significantly lower and erroneous readings than for identically prepared specimens at greater substrate thickness. At 0.25 in, all of the 5059-H131 armor panels evaluated had adequate thickness for valid pull-off test results. In order to capture a statistically meaningful numerical assessment of coating adhesion, a minimum of 30 pull-off data points each were collected for each of the three coating systems.

Table 6. Laboratory conditions for pull-off adhesion ASTM D 4541.

Adhesive Type Used	**Alphacyanoacrylate Ester**
Cure time (hr)	24
Temperature (°C)	25
Percent relative humidity	65
Substrate material	AA85059-H131
Substrate thickness (in)	0.25
Pretreatment types	Abrasive blast Alodine 5200 Metalast TCP-HF
Primer used	MIL-DTL-0053022
Topcoat used	MIL-DTL-53039B (type II)
Total coating thickness (mil)	Abrasive blast ~3.1 Conversion coated ~2.5

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[*] Permabond Adhesive, Grade 910, General Purpose, Metal Bonding, Permabond Industrial Adhesives, 20C World’s Fair Drive, Somerset, NJ 08873.

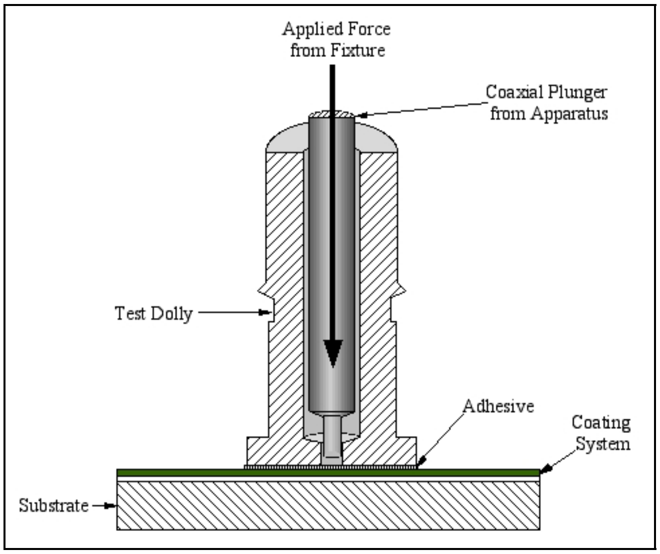


Figure 2. Pull-off hydraulic adhesion test (ASTM D 4541).

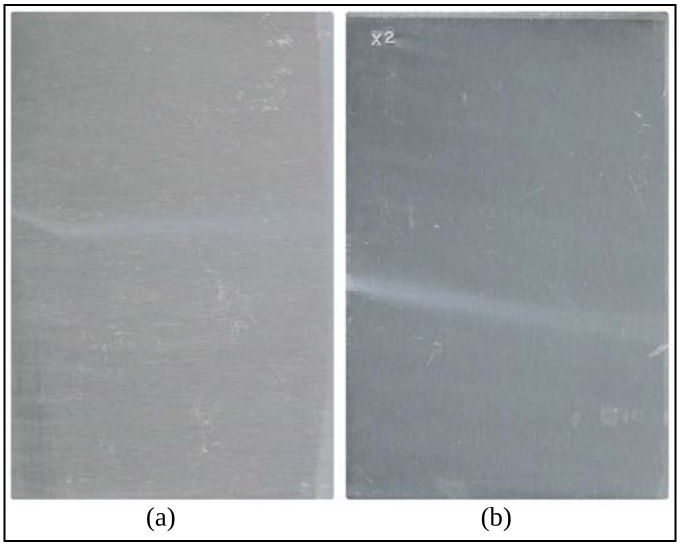
3. Results

3.1 Processing and Coating Application

An additional goal of this study was to assess the AA5059 alloy from a processing standpoint, with the ultimate goal of production in mind. From a processing standpoint, the abrasive blast and conversion coatings process steps were identical to AA5083. For the Metalast TCP-HF, there was even the added bonus of a more distinct color change to the substrate produced by the conversion coating. The Metalast TCP-HF formed a significantly darker blue-gray coating on AA5059 vs. AA5083, as seen in figure 3. From a quality control standpoint, this obvious color change was advantageous for production lines and improved assurance of complete conversion coating coverage prior to applying the primer.

3.2 Accelerated Corrosion

As in previous studies with similar coatings on AA5083 (*22*, *23*), the AA5059 panels exposed to NSF and cyclic corrosion testing exhibited excellent performance under accelerated corrosion conditions. After 8760 hr of NSF exposure, the only apparent creepback damage occurred on the abrasive blast prepared panels. After 400 cycles of GM 9540P cyclic testing, there was no visible creepback damage whatsoever across all coated replicates and only very slight damage to the bare control in the form of a few small pits seen in figure 4. As evident in table 7, the bulk of the damage to the abrasive blast prepared NSF panels appeared within the first week via rapid nucleation and growth of blisters along the scribe. These blisters then progressed at a much slower rate after the initial 1-week observation. Scans depicting the relative progress of blisters



(a) (b)

Figure 3. Metalast TCP-HF color change on (a) AA5083 and (b) AA5059.

for the same panels at 168, 4032, and 8760 hr are shown in figure 5. The conversion coated panels had no coating creepback whatsoever under the full durations of both NSF and cyclic exposures. The “9” ratings that appeared after 2016 hr were solely the result of inevitable discoloration and dulling from oxidation of the bare 5059 substrate exposed within the scribe. Interestingly, while the abrasive blast prepared panels performed significantly worse than the conversion coated counterparts under NSF, they performed just as well as the conversion coated panels under cyclic testing with ratings of 9 across all five replicates at 400 cycles, as listed in table 8. Final scans for all five replicates of all surface pretreatments at 1 year NSF and 400 cycles of GM 9540P are shown in figures 6–11.

3.3 Adhesion

As mentioned in the procedure, a much more rigorous form of wet adhesion testing was conducted. After a full week of immersion in deionized water at 150 ºF, there was no coating removal produced for any of the panels when the tape was removed. All of the panels were rated at 5, a perfect rating and fully compliant with the performance level set for military applications. The wet adhesion data are listed in table 9, and representative scans of the successful results are presented in figure 12.

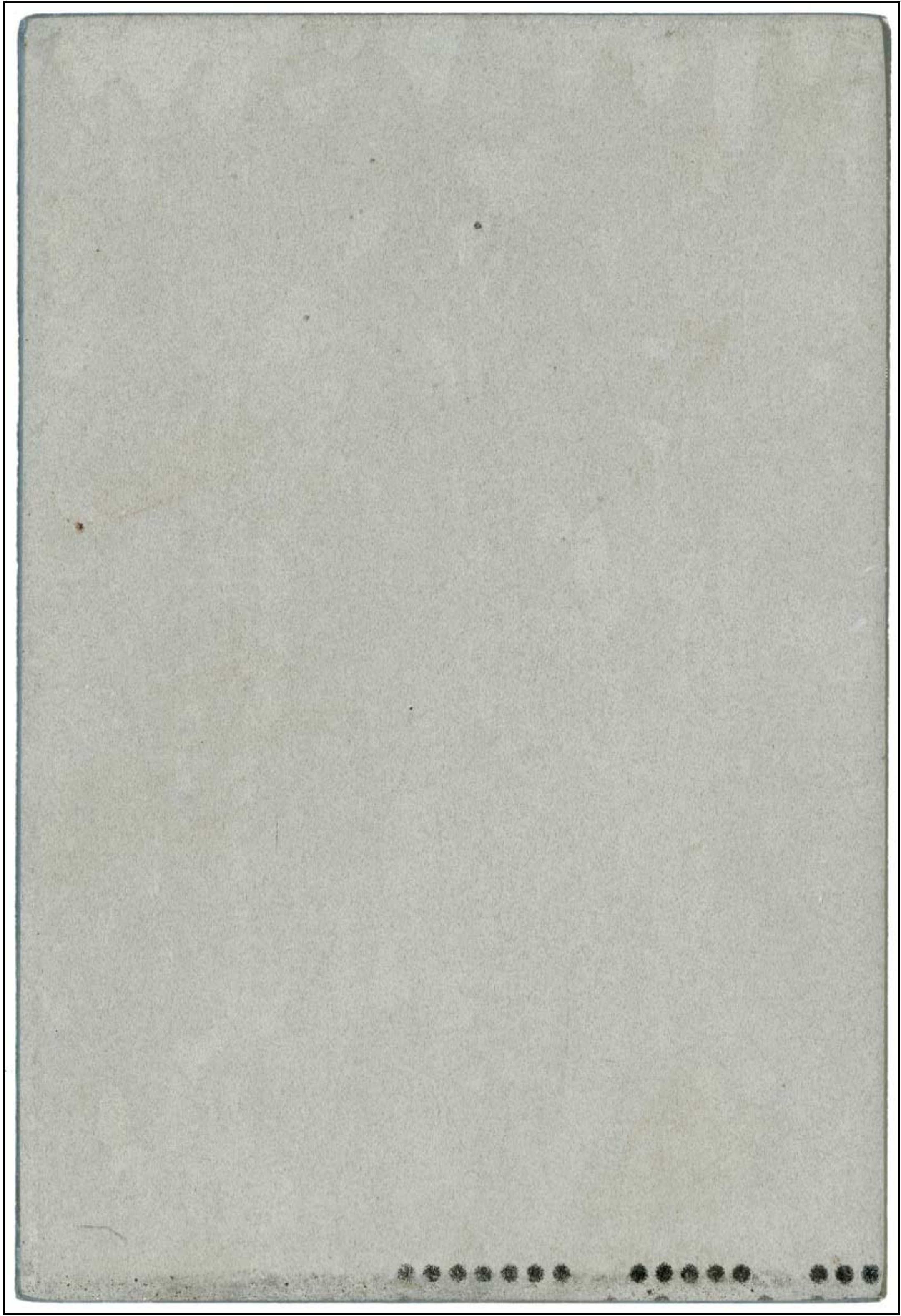


Figure 4. Uncoated AA5059-H131 control after 400 cycles of GM 9540P with dull gray oxidation and a few minor pits (1.33×).

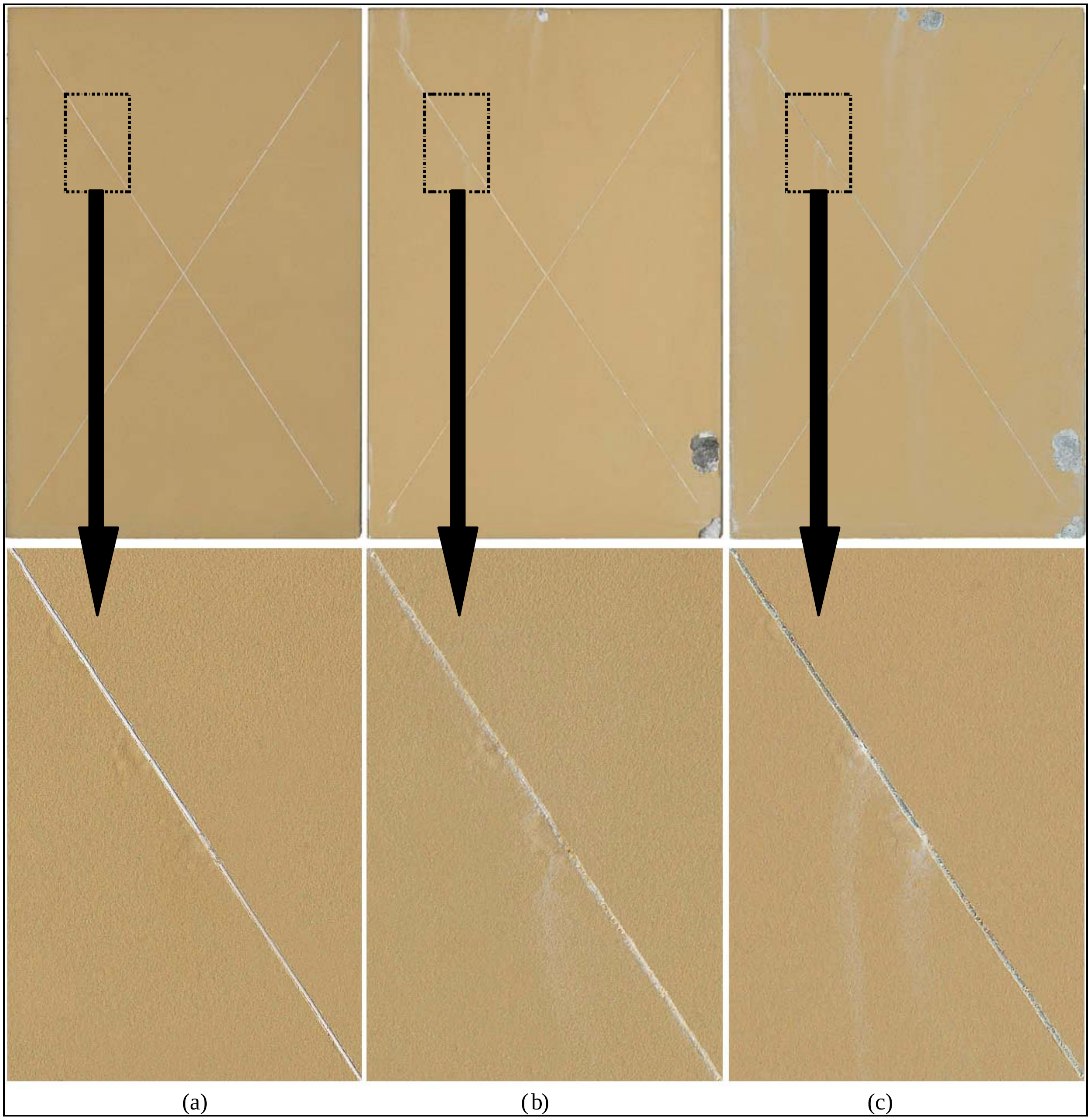
Table 7. ASTM D 1654 creepback ratings for ASTM B 117 NSF.

Panel Designation	Pretreatment	Surface Finish	ASTMB 117 Hours												
			24	168	336	504	1512	2016	2520	3024	3528	4032	6048	8064	8760
A1	Alodine 5200	Mill finish	10	10	10	10	10	10	9	9	9	9	9	9	9
A2	Alodine 5200	Mill finish	10	10	10	10	10	10	9	9	9	9	9	9	9
A3	Alodine 5200	Mill finish	10	10	10	10	10	10	9	9	9	9	9	9	9
A4	Alodine 5200	Mill finish	10	10	10	10	10	10	9	9	9	9	9	9	9
A5	Alodine 5200	Mill finish	10	10	10	10	10	10	9	9	9	9	9	9	9
T1	Metalast TCP-HF	Mill finish	10	10	10	10	10	10	9	9	9	9	9	9	9
T2	Metalast TCP-HF	Mill finish	10	10	10	10	10	10	9	9	9	9	9	9	9
T3	Metalast TCP-HF	Mill finish	10	10	10	10	10	10	9	9	9	9	9	9	9
T4	Metalast TCP-HF	Mill finish	10	10	10	10	10	10	9	9	9	9	9	9	9
T5	Metalast TCP-HF	Mill finish	10	10	10	10	10	10	9	9	9	9	9	9	9
GB1	None - DTM	Abrasive blasted	10	6	6	6	6	5	5	5	5	5	5	5	5
GB2	None - DTM	Abrasive blasted	10	7	7	6	6	6	6	6	6	6	6	6	6
GB3	None - DTM	Abrasive blasted	10	6	6	6	6	6	6	6	6	6	6	6	6
GB4	None - DTM	Abrasive blasted	10	7	7	7	7	7	7	7	7	7	7	7	7
GB5	None - DTM	Abrasive blasted	10	6	6	6	6	6	5	5	5	5	5	5	5

Note: DTM = direct to metal.

The pull-off adhesion evaluations proved more complex than with previous efforts. In addition to evaluating the new 5059-H131 substrate, the MIL-DTL-53039, type II topcoat used was also previously untested. As mentioned in the procedure, it became necessary to use an alphacyanoacrylate ester-based formulation rather than the common commercial off-the-shelf cyanoacrylate formulas more typically used for pull-off adhesion testing. The use of this alternate adhesive was necessitated due to insufficient bonding strength between the metallic surface of the test dolly itself and the cured adhesive. For every measurement attempt, the dolly would separate at the metal-adhesive interface with a very low reading, well under 1000 psi. Several different brands and viscosities of cyanoacrylate were evaluated, in addition to grinding fresh surfaces onto the dollies, in an attempt to improve the mechanical bond strength. In many years of performing this procedure and taking tens of thousands of individual measurements, this behavior had never previously been observed. In order to promote better adhesion to metallic surfaces, alphacyanoacrylate ester, a formulation promoted for its performance when bonded to metals, was selected as a substitute for standard cyanoacrylate and was found to perform satisfactorily.

Thus, when the pull-off measurements were successfully taken, the results also proved interesting regarding the new MIL-DTL-53039 formulation. In every measurement obtained for the panel sets, the failure locations were not at the substrate but instead were located within the primer and topcoat layers. For the conversion coated panels, the pull-off results were all characterized as cohesive within the topcoat layer. This meant that the cohesive strength of the primer layer, the adhesive strength of the topcoat to the primer layer, as well as the adhesive strength of the primer to the conversion coated substrate surface, were all in excess of the



(a) (b) (c)

Figure 5. Blistering comparison between abrasive blast prepared panel with lower half of images magnified 5.5× relative to top at (a) 168 hr, (b) 4032 hr, and (c) 8760 hr (1 year) of neutral salt fog exposure.

cohesive strength of the topcoat. The actual average pull-off values measured between the Alodine 5200 prepared panels and the Metalast TCP-HF panels, 2012 psi and 1974 psi respectively, differed only slightly. This was to be expected as the primer and topcoat layers were identically prepared and failed in identical modes. For the abrasive blast prepared panels, the pull-off values measured were consistently lower averaging 1694 psi. Correspondingly, the

Table 8. ASTM D 1654 creepback ratings for GM 9540P cyclic corrosion.

Panel Designation	Pretreatment	Surface Finish	GM 9540P Cycles													
			10	20	40	60	80	100	120	140	160	180	200	320	340	400
A1	Alodine 5200	Mill finish	10	10	10	10	10	10	9	9	9	9	9	9	9	9
A2	Alodine 5200	Mill finish	10	10	10	10	10	10	9	9	9	9	9	9	9	9
A3	Alodine 5200	Mill finish	10	10	10	10	10	10	9	9	9	9	9	9	9	9
A4	Alodine 5200	Mill finish	10	10	10	10	10	10	9	9	9	9	9	9	9	9
A5	Alodine 5200	Mill finish	10	10	10	10	10	10	9	9	9	9	9	9	9	9
T1	Metalast TCP-HF	Mill finish	10	10	10	10	10	10	9	9	9	9	9	9	9	9
T2	Metalast TCP-HF	Mill finish	10	10	10	10	10	10	9	9	9	9	9	9	9	9
T3	Metalast TCP-HF	Mill finish	10	10	10	10	10	10	9	9	9	9	9	9	9	9
T4	Metalast TCP-HF	Mill finish	10	10	10	10	10	10	9	9	9	9	9	9	9	9
T5	Metalast TCP-HF	Mill finish	10	10	10	10	10	10	9	9	9	9	9	9	9	9
GB1	None - DTM	Abrasive blasted	10	10	10	10	10	10	9	9	9	9	9	9	9	9
GB2	None - DTM	Abrasive blasted	10	10	10	10	10	10	9	9	9	9	9	9	9	9
GB3	None - DTM	Abrasive blasted	10	10	10	10	10	10	9	9	9	9	9	9	9	9
GB4	None - DTM	Abrasive blasted	10	10	10	10	10	10	9	9	9	9	9	9	9	9
GB5	None - DTM	Abrasive blasted	10	10	10	10	10	10	9	9	9	9	9	9	9	9

Note: DTM = direct to metal.

failure mode differed vs. the conversion coated panels and was characterized as adhesive at the topcoat-primer interface. This meant that the weakest link in the coating system occurred between the topcoat and the primer, thus the pull-off tensions were correspondingly lower as would be expected vs. the conversion coated panels that failed cohesively. While all failure tensions measured individually were high enough to justify a good coating, it also meant that it was not possible to ascertain any differences between the different coating types at the primer-substrate interface. While no exact measurement at the primer-substrate layer was possible, it can be stated that the performance was in excess of the adhesive strength of the topcoat to the primer for the blast prepared panels and in excess of the cohesive strength of the topcoat for the conversion-coated panels and, therefore, satisfactory. All pull-off adhesion data with accompanying representative pictures of the pull-off failure modes are listed in tables 10–12. Correspondingly, the pull-off data are plotted as histograms in figures 13–15.

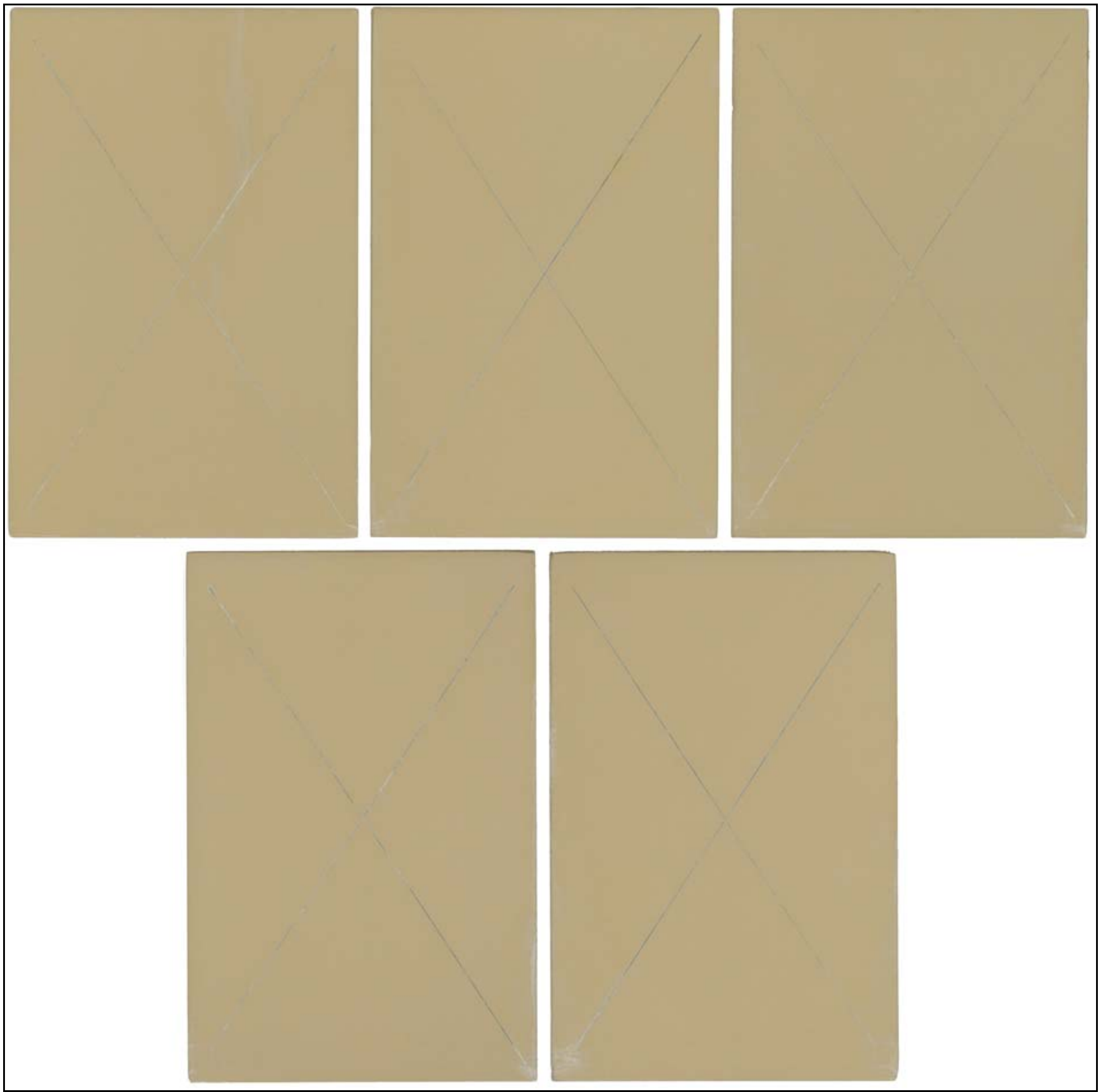


Figure 6. All five replicates of Alodine 5200 after 8760 hr (1 year) NSF exposure, no creepback or blistering.

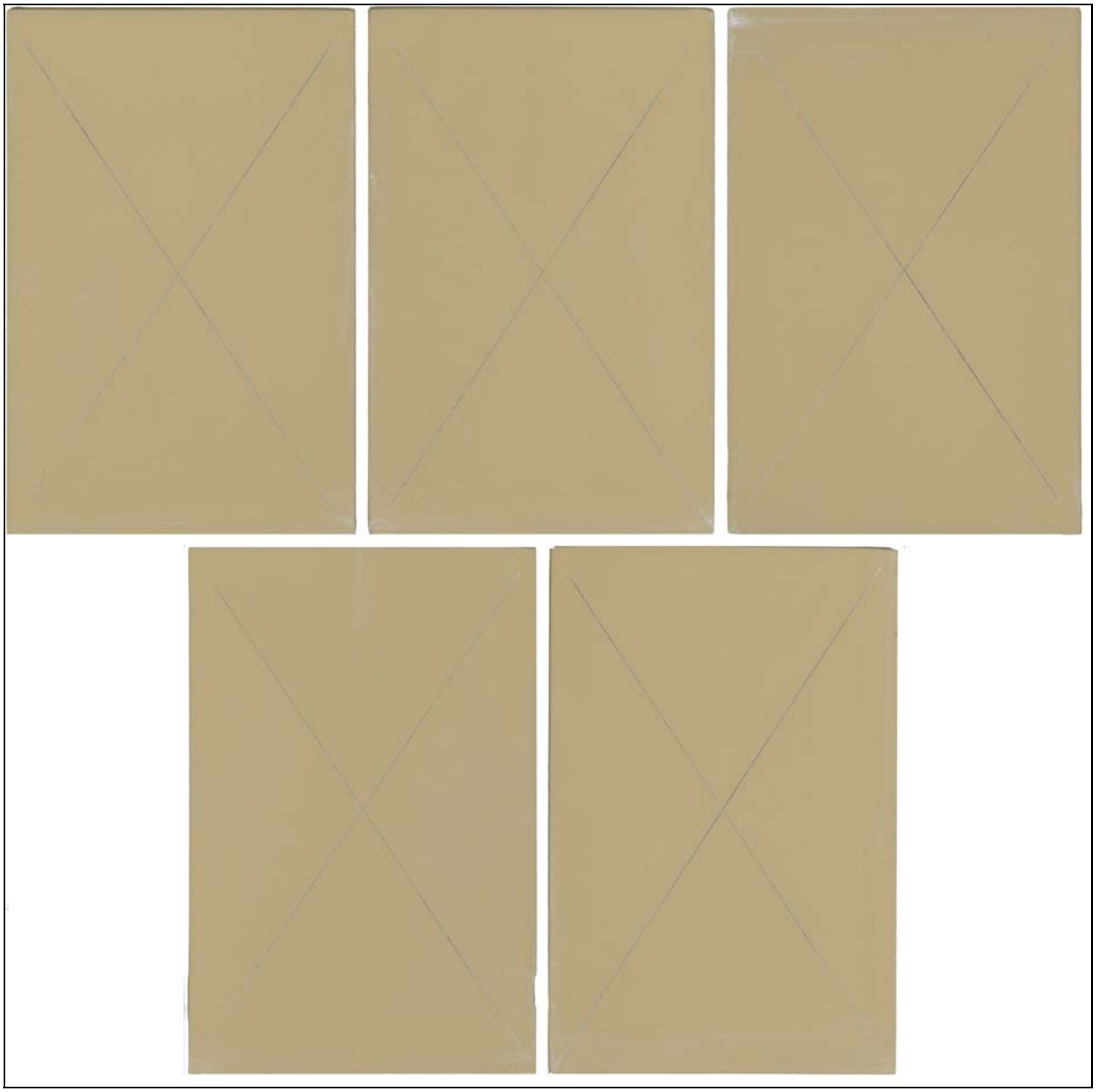


Figure 7. All five replicates of Metalast TCP-HF after 8760 hr (1 year) NSF exposure, no creepback or blistering.

Figure 8. All five replicates prepared by abrasive blast after 8760 hr (1 year) NSF exposure, blisters along scribe and attack at panel edges.

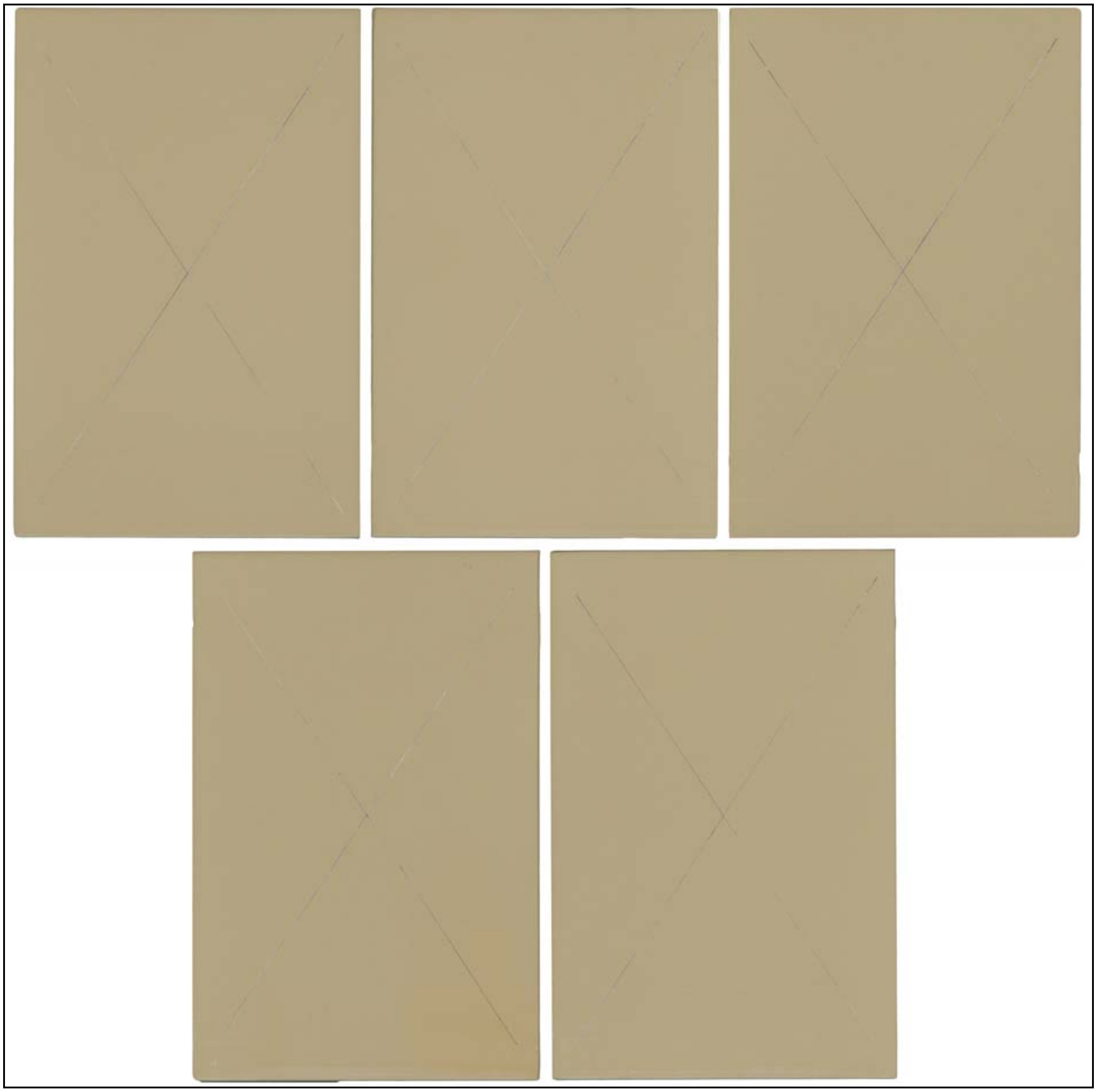


Figure 9. All five replicates of Alodine 5200 after 400 cycles of GM 9540P, no creepback or blistering.

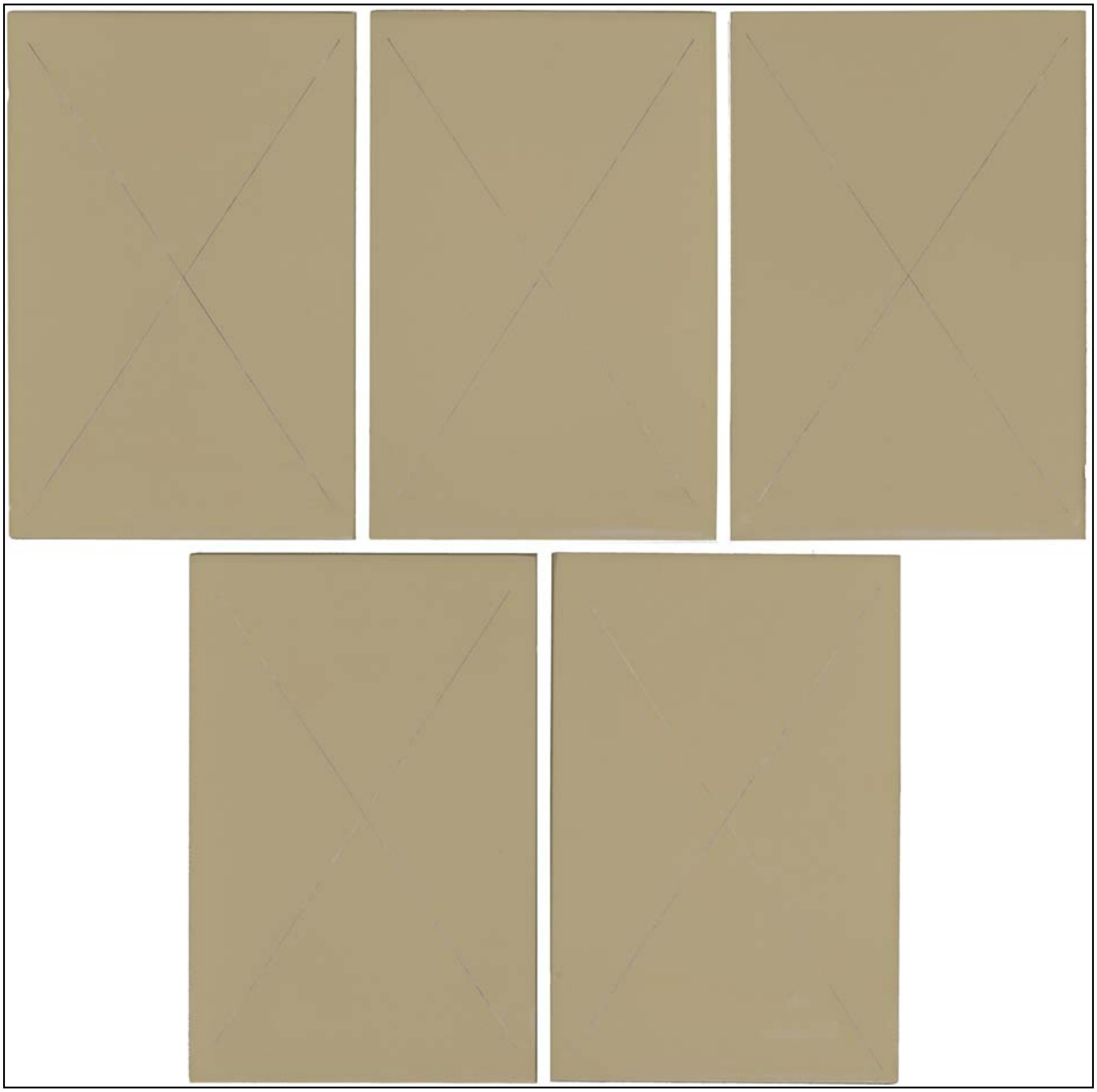


Figure 10. All five replicates of Metalast TCP-HF after 400 cycles of GM 9540P, no creepback or blistering.

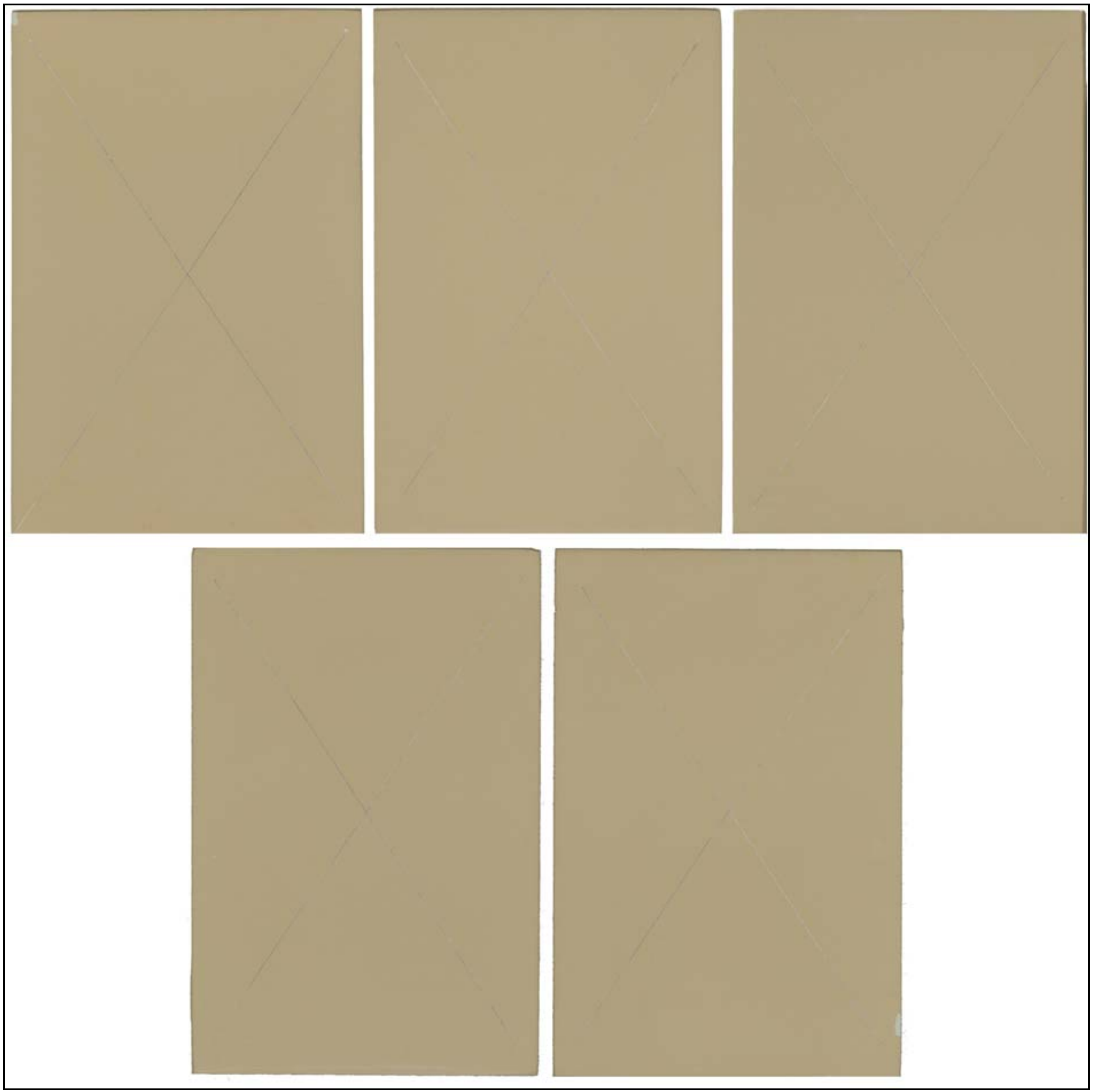


Figure 11. All five replicates prepared by abrasive blast after 400 cycles of GM 9540P, no creepback or blistering.

Table 9. Extended wet adhesion results for coated AA5059 panels at 150 ºF.

Panel Designation	Measurement	Surface Type/Pretreatment	Rating
A19	1	Mill finish/Alodine5200	5A
A19	2	Mill finish/Alodine5200	5A
A20	1	Mill finish/Alodine5200	5A
A20	2	Mill finish/Alodine5200	5A
T19	1	Mill finish/Metalast TCP-HF	5A
T19	2	Mill finish/Metalast TCP-HF	5A
T20	1	Mill finish/Metalast TCP-HF	5A
T20	2	Mill finish/Metalast TCP-HF	5A
GB19	1	Abrasive blasted/none	5A
GB19	2	Abrasive blasted/none	5A
GB20	1	Abrasive blasted/none	5A
GB20	2	Abrasive blasted/none	5A

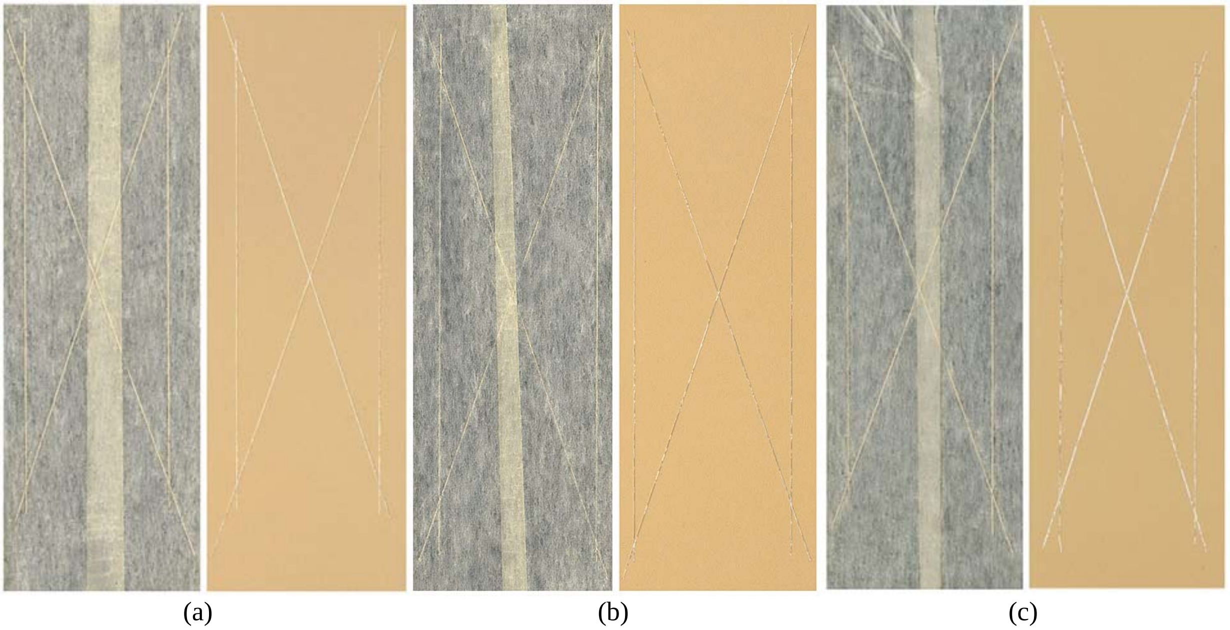


Figure 12. One-week, 150 ºF, immersed wet adhesion panels representative scans showing coating surface incisions and corresponding test tape back sides, all rated at 5A per ASTM D 3359A.
(a) Alodine 5200, (b) Metalast TCP-HF, and (c) grit blast.

Table 10. Pull-off results for Alodine 5200 conversion coated AA5059 panels.

Pretreatment	**Adhesion (psi)**	**Failure/Mode**
Alodine 5200	2000	Topcoat/cohesive
Alodine 5200	1880	Topcoat/cohesive
Alodine 5200	2380	Topcoat/cohesive
Alodine 5200	2380	Topcoat/cohesive
Alodine 5200	2110	Topcoat/cohesive
Alodine 5200	2070	Topcoat/cohesive
Alodine 5200	2020	Topcoat/cohesive
Alodine 5200	2240	Topcoat/cohesive
Alodine 5200	2120	Topcoat/cohesive
Alodine 5200	1800	Topcoat/cohesive
Alodine 5200	1900	Topcoat/cohesive
Alodine 5200	1890	Topcoat/cohesive
Alodine 5200	1800	Topcoat/cohesive
Alodine 5200	2390	Topcoat/cohesive
Alodine 5200	2240	Topcoat/cohesive
Alodine 5200	1790	Topcoat/cohesive
Alodine 5200	2090	Topcoat/cohesive
Alodine 5200	1750	Topcoat/cohesive
Alodine 5200	1640	Topcoat/cohesive
Alodine 5200	1820	Topcoat/cohesive
Alodine 5200	1820	Topcoat/cohesive
Alodine 5200	1800	Topcoat/cohesive
Alodine 5200	1900	Topcoat/cohesive
Alodine 5200	2480	Topcoat/cohesive
Alodine 5200	2690	Topcoat/cohesive
Alodine 5200	1900	Topcoat/cohesive
Alodine 5200	1900	Topcoat/cohesive
Alodine 5200	2230	Topcoat/cohesive
Alodine 5200	1970	Topcoat/cohesive
Alodine 5200	1800	Topcoat/cohesive
Alodine 5200	1810	Topcoat/cohesive
Alodine 5200	1910	Topcoat/cohesive
Alodine 5200	1880	Topcoat/cohesive
Average	2012.12	
Std. dev.	246.07	
Geometric mean	1998.41	
Median	1900.00	
95% confidence	83.95	
Maximum	2690.00	
Minimum	1640.00	

Table 11. Pull-off results for Metalast TCP-HF conversion coated AA5059 panels.

Pretreatment	Adhesion (psi)	Failure/Mode
Metalast TCP-HF	1970	Topcoat/cohesive
Metalast TCP-HF	1860	Topcoat/cohesive
Metalast TCP-HF	1960	Topcoat/cohesive
Metalast TCP-HF	2170	Topcoat/cohesive
Metalast TCP-HF	1920	Topcoat/cohesive
Metalast TCP-HF	1980	Topcoat/cohesive
Metalast TCP-HF	2120	Topcoat/cohesive
Metalast TCP-HF	1860	Topcoat/cohesive
Metalast TCP-HF	1710	Topcoat/cohesive
Metalast TCP-HF	1750	Topcoat/cohesive
Metalast TCP-HF	2240	Topcoat/cohesive
Metalast TCP-HF	1770	Topcoat/cohesive
Metalast TCP-HF	1940	Topcoat/cohesive
Metalast TCP-HF	2140	Topcoat/cohesive
Metalast TCP-HF	2010	Topcoat/cohesive
Metalast TCP-HF	2310	Topcoat/cohesive
Metalast TCP-HF	1890	Topcoat/cohesive
Metalast TCP-HF	2090	Topcoat/cohesive
Metalast TCP-HF	1930	Topcoat/cohesive
Metalast TCP-HF	1800	Topcoat/cohesive
Metalast TCP-HF	2200	Topcoat/cohesive
Metalast TCP-HF	1920	Topcoat/cohesive
Metalast TCP-HF	2120	Topcoat/cohesive
Metalast TCP-HF	2030	Topcoat/cohesive
Metalast TCP-HF	2000	Topcoat/cohesive
Metalast TCP-HF	2080	Topcoat/cohesive
Metalast TCP-HF	1910	Topcoat/cohesive
Metalast TCP-HF	1980	Topcoat/cohesive
Metalast TCP-HF	1700	Topcoat/cohesive
Metalast TCP-HF	1880	Topcoat/cohesive
Average	1974.67	
Std. dev.	154.82	
Geometric mean	1968.82	
Median	1965.00	
95% confidence	55.40	
Maximum	2310.00	
Minimum	1700.00	

Table 12. Pull-off results for abrasive blast prepared AA5059 panels.

Preparation	**Adhesion (psi)**	**Failure/Mode**
Abrasive blast	2020	Topcoat/adhesive
Abrasive blast	1800	Topcoat/adhesive
Abrasive blast	1790	Topcoat/adhesive
Abrasive blast	1870	Topcoat/adhesive
Abrasive blast	1800	Topcoat/adhesive
Abrasive blast	1600	Topcoat/adhesive
Abrasive blast	1780	Topcoat/adhesive
Abrasive blast	1630	Topcoat/adhesive
Abrasive blast	1390	Topcoat/adhesive
Abrasive blast	1520	Topcoat/adhesive
Abrasive blast	2000	Topcoat/adhesive
Abrasive blast	1650	Topcoat/adhesive
Abrasive blast	1450	Topcoat/adhesive
Abrasive blast	1800	Topcoat/adhesive
Abrasive blast	1730	Topcoat/adhesive
Abrasive blast	1520	Topcoat/adhesive
Abrasive blast	1420	Topcoat/adhesive
Abrasive blast	1510	Topcoat/adhesive
Abrasive blast	1630	Topcoat/adhesive
Abrasive blast	1250	Topcoat/adhesive
Abrasive blast	1870	Topcoat/adhesive
Abrasive blast	1990	Topcoat/adhesive
Abrasive blast	1860	Topcoat/adhesive
Abrasive blast	1720	Topcoat/adhesive
Abrasive blast	2000	Topcoat/adhesive
Abrasive blast	1790	Topcoat/adhesive
Abrasive blast	1800	Topcoat/adhesive
Abrasive blast	1860	Topcoat/adhesive
Abrasive blast	1610	Topcoat/adhesive
Abrasive blast	1590	Topcoat/adhesive
Abrasive blast	1540	Topcoat/adhesive
Abrasive blast	1420	Topcoat/adhesive
Average	1694.06	
Std. dev.	198.80	
Geometric mean	1682.43	
Median	1725.00	
95% confidence	68.88	
Maximum	2020.00	
Minimum	1250.00	

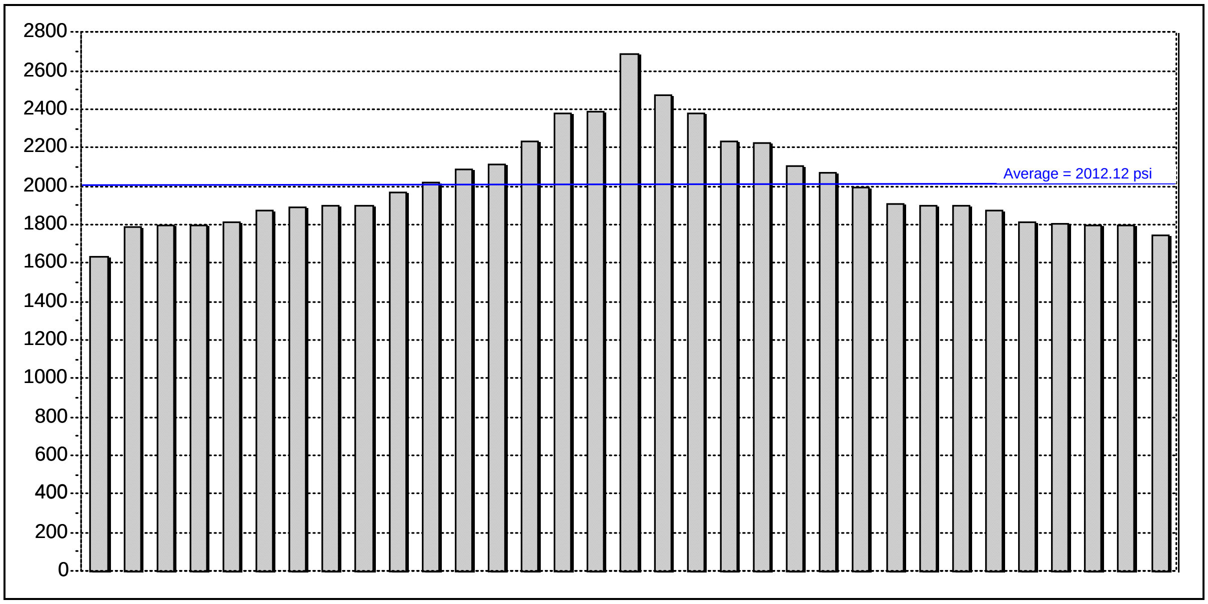


Figure 13. Pull-off data distribution for Alodine 5200 conversion coated AA5059.

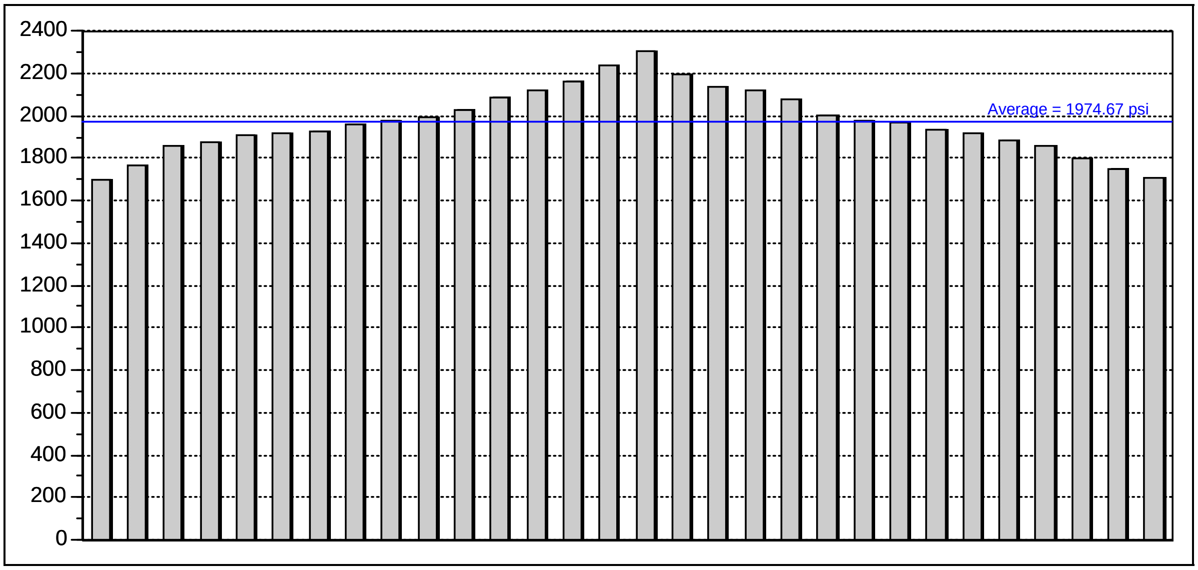


Figure 14. Pull-off data distribution for Metalast TCP-HF conversion coated AA5059.

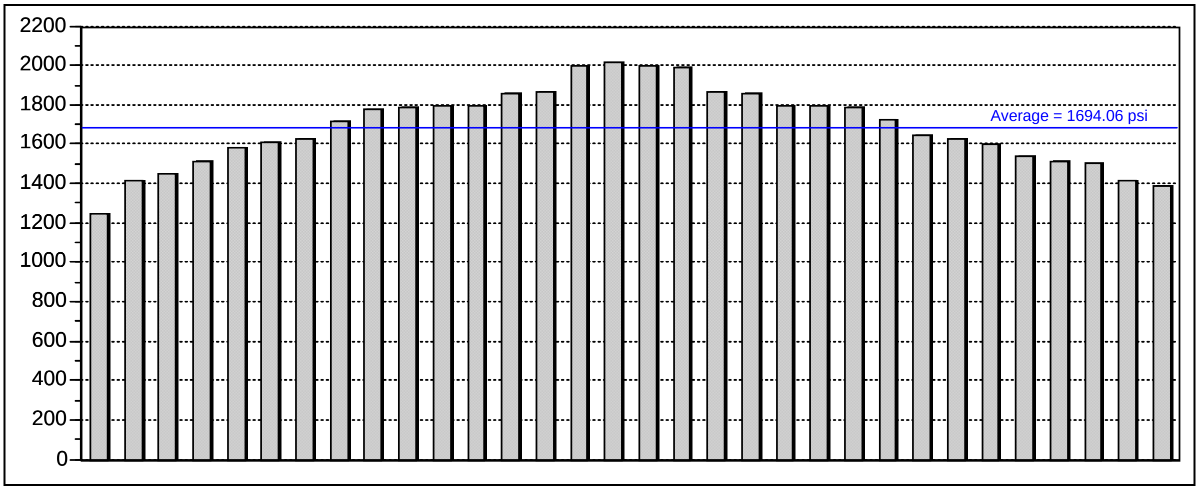


Figure 15. Pull-off data distribution for abrasive blast prepared AA5059.

4. Discussion

The goal of this study was to evaluate the compatibility of the AA5059-H131 substrate with CARC coating systems that are relevant to current and future weapon systems. The excellent corrosion resistance inherent to the AA5059 alloy necessitated the longer exposure durations vs. the shorter intervals normally relevant to the more corrosion prone alloys among the 2000 and 7000 series aluminums. Even after the much greater extended exposure durations, the AA5059 showed little or no damage from corrosion except for the abrasive blast prepared samples under NSF that showed relatively mild blistering along the scribe and some corrosion attack at the test panel edges. Either Alodine 5200 or the Metalast TCP-HF would qualify as an excellent spray-based hexavalent chromium-free pretreatment step on any repair or production line. The Metalast TCP-HF is fully compliant as a type II, hexavalent chromate-free conversion coating under MIL-DTL-5541. The previously referenced studies (*27*, *28*) that also examined less corrosion-resistant Al alloys such as 2024-T3 and 7075-T6 determined the performance of NAVAIR-based TCP variants to be greatest among any of the hexavalent chromate-free conversion coatings.

If a situation arises where absolutely no chromium containing compounds are permitted, then the Alodine 5200 will function very well, but only as part of a complete coating system. Note that unlike Metalast TCP-HF and other accepted TCP-based products, the Alodine 5200 process does not meet the MIL-DTL-5541 specification due to its poor bare corrosion resistance. The aforementioned studies demonstrated that Alodine 5200, while indeed a very good conversion coating, only works well as part of a complete coating system, as was the case in this study. Trivalent chromium pretreatments such as Metalast TCP-HF must provide additional bare corrosion resistance performance in order to qualify under MIL-DTL-5541 and

MIL-DTL-81706. It is important to distinguish this difference for the purpose of highlighting possible performance differences if a coating system were to be damaged or omitted.

In the specific case of AA5059 and other 5000 series alloys where bare corrosion resistance is excellent, the impact from the absence of a primer and topcoat would likely be minimal and the temptation to even omit the pretreatment may exist. In this particular study, the only corrosion damage observed was on the abrasive blasted panels with no conversion coating. In addition, the corresponding pull-off adhesion values were lowest on the abrasive blast prepared set. Keeping the experimental results in mind, based upon its compliance with the conversion coating military specifications and its ability to sustain performance under bare conditions, one would logically conclude that the best overall system to use would be TCP based. It can be mentioned that while the abrasive blast method certainly works, it is certainly not optimum and not recommended, other than perhaps for field repairs where conversion coatings are not always available or practical.

A secondary goal of this study was to obtain more data on the new class of low VOC single component topcoats specified under MIL-DTL-53039B. In all of the corrosion and adhesion evaluations, no detrimental effects were observed. The only real anomalous behavior observed was the significant decrease in bonding performance of standard cyanoacrylate adhesives to the steel pull-off dollies when bonded to the new coating. Ultimately, it was rationalized that some component unique to the new MIL-DTL-53039B, type II topcoat formulation with the polymeric beads interacted with the adhesive and thus degraded the bonding strength of standard cyanoacrylate formulas to the steel dolly surfaces. Whether or not this interaction is specific to all MIL-DTL-53039, type II coatings, MIL-DTL-53039, type II coatings containing polymeric beads for flattening, or MIL-DTL-53039, type II coatings from Hentzen has not been determined.

5. Conclusions

1. AA5059-H131 did not display any negative compatibility problems with any of the CARC coating components, processes, or the complete coating systems.
2. The performance for all three coating systems on the AA5059 was exemplary under extended accelerated corrosion durations of 8760 hr in NSF and 400 cycles in GM 9540P cyclic.
3. Based upon excellent performance, the Metalast TCP-HF formulation evaluated in this study as well as any other NAVAIR-based TCP variant listed on the MIL-DTL-81706 QPL would be recommended as the pretreatment of choice based upon its qualification

with the conversion coating MIL-DTL-5541 and MIL-DTL-81706 and its ability to sustain performance under bare conditions.

4. The nonchromate pretreatment Alodine 5200 exhibited excellent performance as part of a complete coating system and would be ideal for situations in which any chromium containing compounds, hexavalent or otherwise, are completely banned.
5. All coating systems exhibited superior extended 150 ºF wet adhesion performance in ASTM D 3359A, with perfect ratings of 5.
6. Adhesion problems with cyanoacrylate bonding to the pull-off test dollies were likely caused by absorption of a component or additive within the MIL-DTL-53039B, type II topcoat and subsequent interaction and degradation of the adhesive.

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