



Performance Assessment of Two Different Aviation CARC Coating Systems on Steel When Cadmium Plating Is Eliminated

**by Brian E. Placzankis, Chris E. Miller, Scott M. Grendahl, Michael J. Kane,
and Kirit J. Bhansali**

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14. ABSTRACT Corrosion-test specimens of AISI 4130 and 4340 steel with and without cadmium plating were prepared for evaluation using two different aviation versions of the chemical agent resistant coating system. Both coating systems used MIL-PRF-23377 high solids primer and MIL-DTL-64159 type II waterborne topcoat. One system incorporated the established MIL-PRF-23377 primer formula with hexavalent chromium (class C), while the other utilized an improved nonchromate (class N) MIL-PRF-23377 formulation that recently passed the specification performance criteria. The 4130 steel specimens were used to assess performance for general corrosion, throwing power, and crevice corrosion all under GM 9540P accelerated cyclic-corrosion conditions. In addition, thick 4130 steel panels were prepared using a variety of surface preparation conditions to assess pull-off coating adhesion, in accordance with ASTM-D-4541. AISI 4340 material was fabricated in accordance with ASTM-F-519. Type 1d C-rings were utilized to assess hydrogen embrittlement tolerance differences between the various coating systems under GM 9540P conditions. (Note: the complete reference information for all standards and specifications mentioned here can be found in the report.)					
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1. Introduction

For many decades, cadmium (Cd) electroplating has been an effective means of protecting low alloy and high-strength ferrous substrates via its relatively mild anodic sacrificial contribution (compared to other sacrificial coatings, such as zinc). Unfortunately, in addition to its success as a corrosion inhibitor, Cd plating and its accompanying hexavalent chromium-based posttreatments are carcinogenic sources to humans and animals via occupational and environmental exposures (1). Though some eliminations have been possible for lower-strength steel applications in less corrosive environments and on ground-based systems (2), Cd plating remains the coating of choice for high-strength steels used in flight-critical applications for military aviation. Because of recent improvements to primers (notably, chromate-free formulations covered under the military specification MIL-PRF-23377 [3]) the feasibility of eliminating Cd plating from the coating system, leaving only the epoxy primer and chemical agent resistance coating (CARC) topcoat, was revisited. In addition, because of more stringent environmental and safety regulations, the availability of job shops offering Cd plating has significantly decreased over the years. It is becoming increasingly difficult to Cd plate or obtain acceptable quality Cd-plating for a variety of critical safety items, especially those items manufactured by sub-tier vendors that rely small plating job shops. In many instances, Cd plating does not meet the minimum thickness requirements per the specification; some of the coating thicknesses are as low as 10% of the specified requirement. Cd-plating availability will continue to decline in the foreseeable future, and it may no longer be feasible to Cd plate some critical safety items. The data generated in this effort will help to assess the corrosion performance risk of omitting Cd from the overall coating system and will provide insight into the effect of Cd thicknesses less than the specified requirement.

2. Experimental Procedure

The study emphasized five key performance attributes: general corrosion, crevice corrosion, throwing power, coating adhesion, and hydrogen embrittlement. The experimental matrix listing the substrates, test conditions, plating, and coating parameters is presented in table 1. While the physical specimen parameters varied from group to group, all coating preparation for the specimens followed standard methods. Procedures and specimen traits unique to each major test area are addressed separately.

For all groups (except hydrogen embrittlement), the substrates used were AISI 4130 (77-81 HRB). The embrittlement specimens used AISI 4340 high-strength steel (51–53 HRC). The substrates were prepared with and without electroplated Cd. The Cd-plated specimens were prepared in accordance with SAE AMS QQ-P-416 (4), type II (with supplementary chromate

Table 1. Coating systems.

Designation	Plating	Primer
1	Cadmium	MIL-PRF-23377, class C
2	None	MIL-PRF-23377, class C
3	Cadmium	MIL-PRF-23377, class N
4	None	MIL-PRF-23377, class N

Note: all panels were topcoated using MIL-DTL-64159 (5).

treatment), class II (0.00032 in, min). The primer coatings varied between two MIL-PRF-23377 classes, chromated and nonchromated. All of the primers used were supplied by Hentzen, with the exception of the chromated hydrogen-embrittlement C-ring specimens that used Sherwin Williams because of supply constraints. The primer coats were all applied at 1–1.5-mils dry film thickness. All topcoated specimens were applied at 2–2.5-mils dry film thickness with MIL-DTL-64159 383 Green waterborne topcoat. The coated specimens were all allowed 1 week to cure under standard laboratory conditions (25 °C), followed by 1 week of elevated temperature curing at 65 °C. The coating systems were assigned an abbreviated numbering system to enable easy specimen identification.

Table 1 presents the identification scheme used throughout this study. With the exception of the adhesion specimens, all groups utilized GM 9540P (6) accelerated cyclic corrosion to produce the corrosive conditions. The GM 9540P exposure was conducted in a cyclic-corrosion chamber from Atotech (shown in figure 1). The GM 9540P test consisted of 18 separate stages, including saltwater spray, high humidity exposure, drying, an ambient dwell, and elevated temperature heated drying. The environmental conditions and duration of each stage for one complete GM 9540P cycle are provided in table 2. A standard GM 9540P test solution consisting by weight of 0.9% NaCl, 0.1% CaCl₂, and 0.25% NaHCO₃ was used. In addition, the cyclic chamber was calibrated with standard steel mass-loss calibration coupons, as described in the GM 9540P test specification.

For general corrosion, test panels of AISI 4130 measuring 4 × 6 × 0.06 in were utilized. Half of the panels were electroplated with Cd, while the remaining panels were left with the as-received mill finish. Prior to priming, all of the panels were solvent-wiped with acetone to remove any residual particulates and/or machine oils. The panels were halved again with one group receiving MIL-PRF-23377C (chromated, greenish-yellow pigment) and the balance receiving MIL-PRF-23377N (nonchromated, beige) for the Cd-plated and as-received groups, respectively. All of the panels were then topcoated with MIL-DTL-64159, type II, CARC, 383 Green waterborne topcoat. After curing, the panels were X scribed and exposed under GM 9540P for 80 cycles. In accordance with ASTM D 1654-79A (7), scribe creepback measurements were conducted at 10, 20, 40, 60, and 80 cycles.



Figure 1. Test chamber configuration used for GM 9540P cyclic corrosion.

Table 2. 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

To more easily view the inevitable large quantities of raw data from this test matrix, color codes were assigned based upon ranges of ASTM D 1654 ratings. Table 3 depicts the ASTM D 1654 rating parameters and also defines the colors and their respective rating ranges. For panels where coating corrosion blisters occurred away from the scribe, a diagonal crosshatched pattern was

Table 3. Evaluation of scribed, coated specimens subjected to corrosive environments (ATSM D 1654).

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

added to the data cells. An asterisk was added after the ratings at the first appearance of red rust. When creepback ratings exceeded 16 mm (meriting a 0 rating under ASTM D 1654), early exposure terminations were made for panels. Images that compared the general corrosion between coating systems were obtained via 600 dpi digital flatbed scans at 80 cycles or at the cycle of termination. Table 4 lists the general-corrosion experimental parameters.

Table 4. Experimental matrix for general corrosion under GM 9540P.

Designation	Plating	Primer
1G	Cadmium	MIL-PRF-23377, Class C
2G	None	MIL-PRF-23377, Class C
3G	Cadmium	MIL-PRF-23377, Class N
4G	None	MIL-PRF-23377, Class N

Note: all panels were topcoated using MIL-DTL-64159 and had mill finish surface profiles.

Crevice corrosion was evaluated using sandwich-type specimen configurations. The sandwich configurations used panels and coating configurations identical to the general corrosion, but with three panels comprising one complete specimen sandwich. The sandwich specimens were arranged with the panels' test faces directly contacting each other. Two outer panels with one side fully prepared faced a third inner panel with both sides fully prepared. The outside faces of the two outside panels were only primed (not topcoated), which facilitated easy identification by using different pigments for the chromated and nonchromated primer formulations. The sandwich crevice-corrosion method examined damaged and undamaged conditions

simultaneously, with two replicates for each coating system and removal interval for damaged panels and one replicate for undamaged panels. For the damaged condition, all of the inward facing sides were X scribed with offsets between the inner and outer contacting faces. The undamaged panels were not altered. All of the sandwich assemblies were clamped wet in GM 9540P test solution under a 50-lb load using spring tension adjustable clamps (figure 2). Figure 3 depicts the arrangement and clamping of the three crevice test panels in sandwich-style crevice-corrosion assemblies. The sandwich assemblies were then placed in GM 9540P with set removal intervals. For the damaged/scribed assemblies, two assemblies were removed at 10, 20, 30, 40, and 50 cycle intervals and rated for scribe creepback in accordance with ASTM D 1654. For the undamaged condition, one assembly was removed at 20, 40, 60, 80, and 100 cycles and rated for blister damage using ASTM D 1654. Figure 4 depicts an overview of the entire crevice-corrosion experiment. As in general corrosion, images comparing the crevice-corrosion severity between coating systems were obtained via digital flatbed scanning. Table 5 provides an overview listing of crevice-corrosion experimental parameters.



Figure 2. Adjustable clamp used for sandwich assemblies in crevice corrosion.

The ability of a coating to inhibit areas of exposed metallic substrate adjacent to, but not under, the coating from the onset of corrosion (also known as throwing power) was evaluated with the same panel and coating configurations used in general- and crevice-corrosion testing.

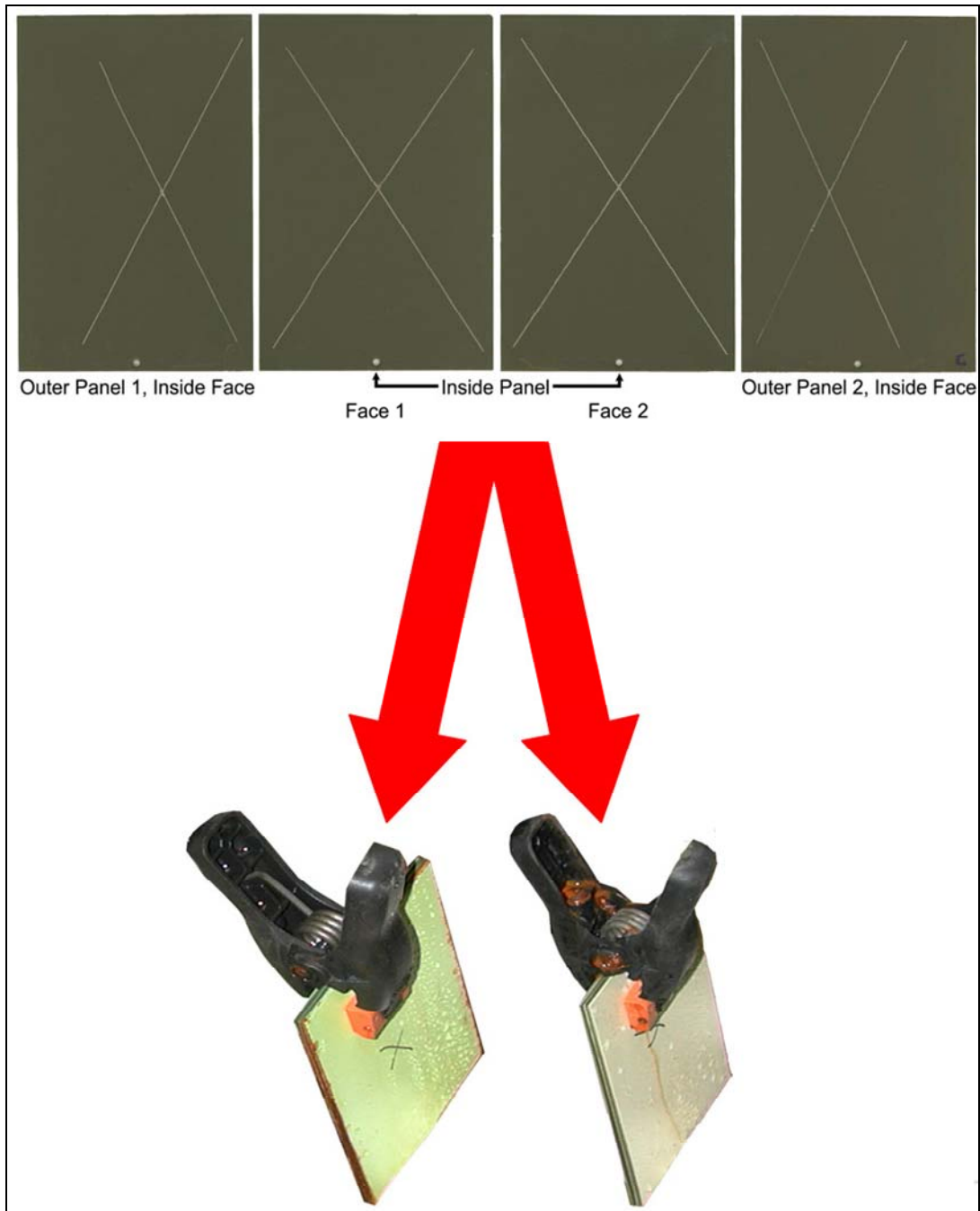


Figure 3. Crevice-corrosion sandwich-assembly configuration showing scribe layouts and chromated and nonchromated assemblies.

Throwing-power test panels, with and without Cd plating, were prepared by masking the centers with single strips of vertically arranged tape in a range of widths (0.0625, 0.125, 0.250, 0.5, 1, and 2 in as shown in figure 5). With the masking tape in place, five replicates of each panel were sprayed in the standard manner. In addition, five replicates for each thickness and primer were prepared without MIL-DTL-64159 topcoat. After the standard cure, the panels were exposed

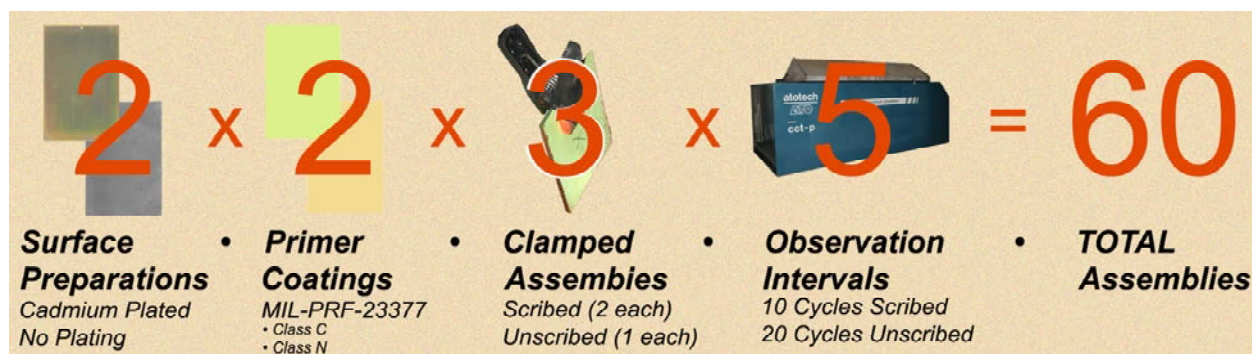


Figure 4. Total matrix overview for crevice corrosion.

Table 5. Experimental parameters and general matrix for crevice corrosion.

Panel No.	Condition	Replicates /Removal Interval	GM 9540P Cycles /Removal Interval	Plating	Primer
1C	Scribed	2	10	Cadmium	MIL-PRF-23377, class C
1C	Unscribed	1	20	Cadmium	MIL-PRF-23377, class C
2C	Scribed	2	10	None	MIL-PRF-23377, class C
2C	Unscribed	1	20	None	MIL-PRF-23377, class C
3C	Scribed	2	10	Cadmium	MIL-PRF-23377, class N
3C	Unscribed	1	20	Cadmium	MIL-PRF-23377, class N
4C	Scribed	2	10	None	MIL-PRF-23377, class N
4C	Unscribed	1	20	None	MIL-PRF-23377, class N

Note: all panels were topcoated using MIL-DTL-64159 and had mill finish surface profiles.

under GM 9540P and monitored for the first appearance of red ferrous rust. When a rusted panel was detected, the panel was removed and the cycles were recorded. Digital images of the panels were scanned in the same manner described for general corrosion.

Changes in coating adhesion in response to changes in plating, primer, surface preparation, and preservation were assessed using pull-off adhesion, in accordance with ASTM D 4541 (8). Multiple sets of three replicate panels (4 × 6 × 0.12 in) were prepared under a variety of surface-preparation conditions. For the Cd-free unplated panels, the conditions were as-received (mill finish) and abrasive-blasted. The abrasive-blast category was further subdivided by set time intervals between blasting and the application of the primers. The panels were blasted with 60-grit aluminum oxide media to an SSPC-10 grade surface and cleaned under lab air at 120 psi to remove remaining residuals from the blasting operation. The abrasive-blasted panels were then left exposed in open lab-air conditions (25 °C at 30% relative humidity for time intervals of 15 and 30 min and 1, 2, and 4 hr). Abrasive-blasting steels in high-humidity environments (and even relatively dry indoor ambient air) commonly leads to flash rusting and degradation of prepared surfaces' capabilities to provide optimum adhesion of applied coatings. In order to minimize blasted-surface degradations in open-air facilities, long-term storage methods, such as nitrogen packaging, can be used. In order to assess the effect of dwell time before coating, additional sets for each of the timed intervals were abrasive-blasted as before.

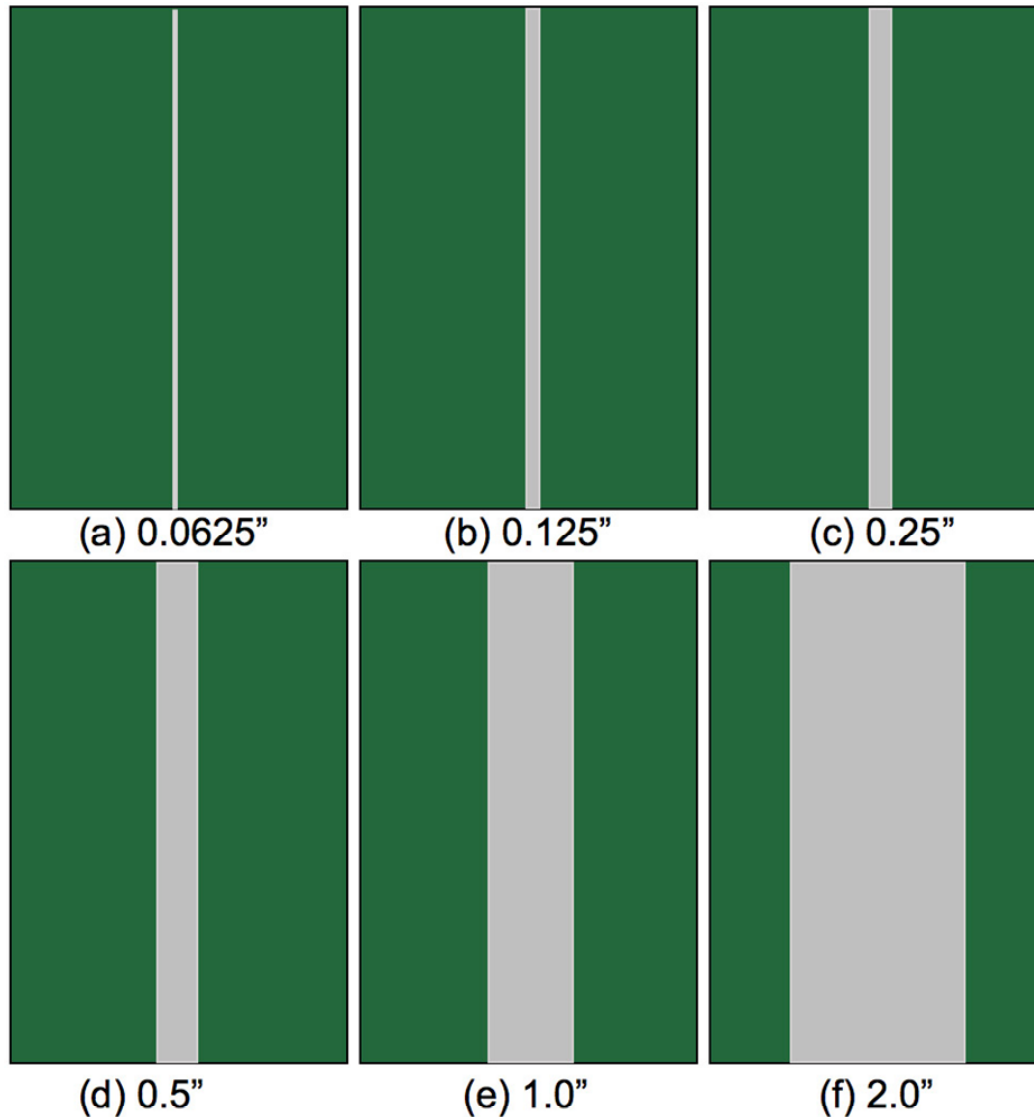


Figure 5. Throwing-power-panel experimental configuration showing widths of primer- and topcoat-free areas.

However, they were immediately vacuum-evacuated and backfilled with laboratory pure nitrogen gas in poly-nylon bags (Jetplate) using a tabletop vacuum impulse sealer (Packaging Aids Corporation, series 88) shown in figure 6. These packaged sets were allowed to dwell for time intervals corresponding to the open-air panels and were primed simultaneously with the open-air sets using the standard coating systems from this study. Additional sets of Cd-plated panels and unplated steel with unmodified mill finish were solvent-wiped with acetone, primed, and topcoated. No other surface treatments were applied. An hydraulic adhesion tester (Elcometer, model 108) was used for this procedure. In addition to being a more quantitative test method, pull-off adhesion is also less prone to human subjectivity, i.e., variations in pressure applied during scribing and interpretation and perception of results. For the pull-off adhesion test, a loading fixture commonly referred to as a dolly is secured normal to the coating surface



Figure 6. Tabletop vacuum impulse sealer with nitrogen backfill (Packaging Aids Corporation, series 88).

with an adhesive (INSTAbond[®] S-100, lot no. FF-236 cyanoacrylate). After allowing the adhesive to cure for 24 hr at 40 °C in 65% relative humidity conditions (table 6), the attached dolly was inserted into the test apparatus.

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

Adhesive Type	Cyanoacrylate
Cure Time	24 hr
Temperature	40 °C
Relative Humidity	~65%
Substrate Material	AISI 4130 steel
Substrate Thickness	0.12 in
Substrate Surface	Cd-plated SSPC-10-blasted Mill finish
Pretreatment Types	Chromate rinse (Cd)
Primer Types	MIL-PRF-23377, class C MIL-PRF-23377, class N
Topcoat	MIL-DTL-64149
Coating Thickness	4 mils (maximum)

* INSTAbond is a trademark of Accrabond, Inc.

The load applied by the apparatus was gradually increased and monitored on the gauge until a plug of coating was detached. The failure tension in pounds per square inch and the failure mode and location within the coating system were recorded. The pull-off test apparatus and dolly configuration are illustrated in figure 7. 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.” When a thin specimen is used, the resultant bulge causes the coating to radially peel away from the center instead of being uniformly pulled away in pure tension and, thus, results in significantly lower readings for identically prepared specimens at greater substrate thickness. At 0.12 in, the panels were adequately thick for valid pull-off test results. For each set of three replicates, a minimum of 42 pull-off readings were collected. Only clear adhesive or cohesive pull-off tension values within the coating system or at the substrate were reported. Measurements for samples with cohesive or adhesive failure of the cyanoacrylate adhesive above the topmost coating surface were rejected. A complete overview and description of the adhesion matrix, including panel designation, is provided in table 7.

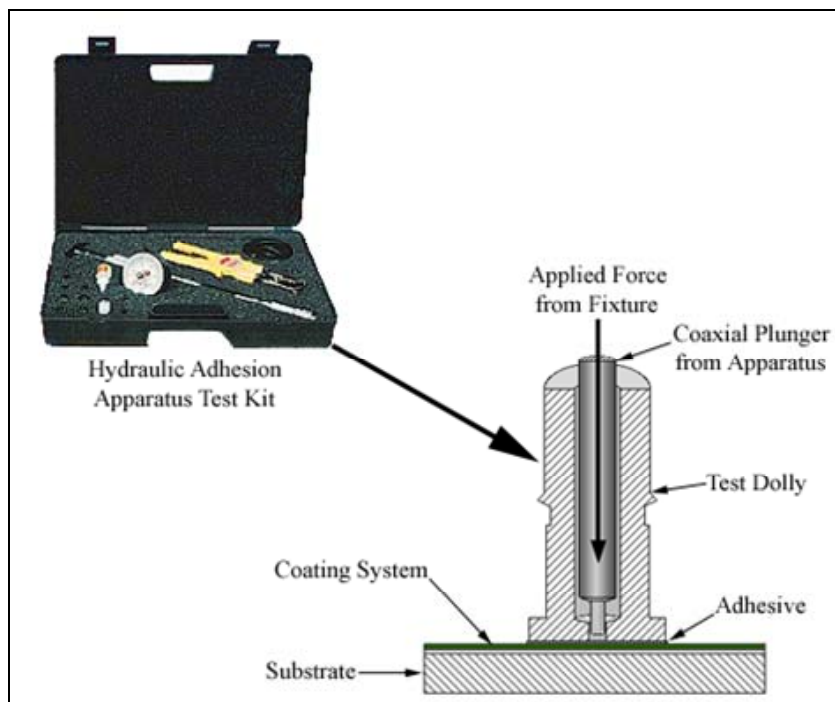


Figure 7. Pull-off hydraulic adhesion test (ASTM D 4541).

Evaluating in-service hydrogen re-embrittlement/stress corrosion cracking of high-strength steel under Cd-plated and nonplated conditions with different primers used type 1d C-ring specimens, all prepared from the same heat as AISI 4340, in accordance with ASTM F 519 (9). Deflection measurements at fractures on 10 untreated C-rings were generated using vernier calipers. These 10 measurements were then averaged to determine the 100% notch fracture load. Sensitivity calibration tests for bright and dull Cd-plated C-rings (three each) were conducted in accordance

Table 7. Experimental matrix for pull-off adhesion (ASTM D 4541).

Panel Designation	Plating System	Surface Profile	Post Blast Dwell Time (min)	Primer Coating
1	Cadmium	Mill with plating	NA	MIL-PRF-23377, class C
2A	None	Abrasive blasted	15	MIL-PRF-23377, class C
2AS	None	Abrasive blasted	15	MIL-PRF-23377, class C
2B	None	Abrasive blasted	30	MIL-PRF-23377, class C
2BS	None	Abrasive blasted	30	MIL-PRF-23377, class C
2C	None	Abrasive blasted	60	MIL-PRF-23377, class C
2CS	None	Abrasive blasted	60	MIL-PRF-23377, class C
2D	None	Abrasive blasted	120	MIL-PRF-23377, class C
2DS	None	Abrasive blasted	120	MIL-PRF-23377, class C
2E	None	Abrasive blasted	240	MIL-PRF-23377, class C
2ES	None	Abrasive blasted	240	MIL-PRF-23377, class C
2M	None	Mill finish	NA	MIL-PRF-23377, class C
3	Cadmium	Mill with plating	NA	MIL-PRF-23377, class N
4A	None	Abrasive blasted	15	MIL-PRF-23377, class N
4AS	None	Abrasive blasted	15	MIL-PRF-23377, class N
4B	None	Abrasive blasted	30	MIL-PRF-23377, class N
4BS	None	Abrasive blasted	30	MIL-PRF-23377, class N
4C	None	Abrasive blasted	60	MIL-PRF-23377, class N
4CS	None	Abrasive blasted	60	MIL-PRF-23377, class N
4D	None	Abrasive blasted	120	MIL-PRF-23377, class N
4DS	None	Abrasive blasted	120	MIL-PRF-23377, class N
4E	None	Abrasive blasted	240	MIL-PRF-23377, class N
4ES	None	Abrasive blasted	240	MIL-PRF-23377, class N
4M	None	Mill finish	NA	MIL-PRF-23377, class N

Notes: all panels were topcoated using MIL-DTL-64159.

NA = not applicable.

with ASTM F 519 and utilizing the average fracture strength previously determined. Tables 8 and 9 list the calibration parameters for the C-ring specimens. The C-ring loads, initially proposed for 65% of notch-bend fracture, were ultimately downscaled to 40% after premature failures (<200 hr) of the SAE AMS QQ-P-416 Cd-plated C-rings under open air at loads of 65% and 50%. The experimental load was determined after a SAE AMS QQ-P-416 prepared C-ring withstood over 200 hr at 40% load in open air. The remaining 40 C-rings were divided into four groups based upon the coating configurations and procedures described previously. Half of these were left coated and the other half were “damaged” by removing the coating from the notched portion. In order to avoid metal-to-metal contact between the loading fasteners and C-ring substrate, the regions surrounding the holes where the tensioning fasteners rubbed against the C-ring surface during loading operations were thoroughly masked. Immediately after loading the C-rings at 40%, the remaining coating-free surfaces adjacent to the fastener-loading regions were brush-coated with primers respective to the prepared set. The C-rings were then placed into

Table 8. Determination of ultimate tensile strength (UTS).

Specimen No.	Beginning Width (in)	Final Width at Fracture (in)	Deflection (Δ) (in)
1	1.959	1.825	0.1340
2	1.956	1.826	0.1300
3	1.957	1.824	0.1330
4	1.958	1.828	0.1300
5	1.957	1.823	0.1340
6	1.957	1.822	0.1350
7	1.957	1.827	0.1300
8	1.956	1.828	0.1280
9	1.953	1.819	0.1340
10	1.957	1.822	0.1350

Calculation notes: Average deflection = 0.1323 in.

Are all deflections within 0.005 in of average? Yes.

Deflection at 65% UTS = 0.086 in.

Deflection at 75% UTS = 0.099 in.

Table 9. Sensitivity and experimental load calibration for Cd-plated, type 1d C-rings.

Specimen No.	Beginning Width (in)	Loaded Width (in)	UTS (%)	Displacement at Load (in)	Time Until Failure (hr)
Bright Cd 1	1.962	1.863	75	0.099	<1
Bright Cd 2	1.962	1.863	75	0.099	<1
Bright Cd 3	1.962	1.863	75	0.099	<1
Dull Cd 1	1.962	1.863	75	0.099	>200
Dull Cd 2	1.962	1.863	75	0.099	>200
Dull Cd 3	1.963	1.864	75	0.099	>200
Plain 1	1.966	1.867	75	0.099	Did not fail
Plain 2	1.968	1.869	75	0.099	Did not fail
Plain 3	1.968	1.869	75	0.099	Did not fail
SAE AMS QQ-P-416, 1	1.968	1.882	65	0.086	<6
SAE AMS QQ-P-416, 2	1.970	1.884	65	0.086	<6
SAE AMS QQ-P-416, 3	1.971	1.885	65	0.086	<6
SAE AMS QQ-P-416, 4	1.964	1.898	50	0.066	<24
SAE AMS QQ-P-416, 5	1.970	1.917	40	0.053	>200 ^a

^a Used as basis for loading C-ring test matrix.

GM 9540P cyclic corrosion with the notched toward the chamber spray nozzles to ensure an unobstructed angle of the GM 9540P spray solution (figure 8). When exposure commenced, the C-rings were monitored for failure. When specimen failure was detected, the C-ring was removed and the GM 9540 cycle was recorded.

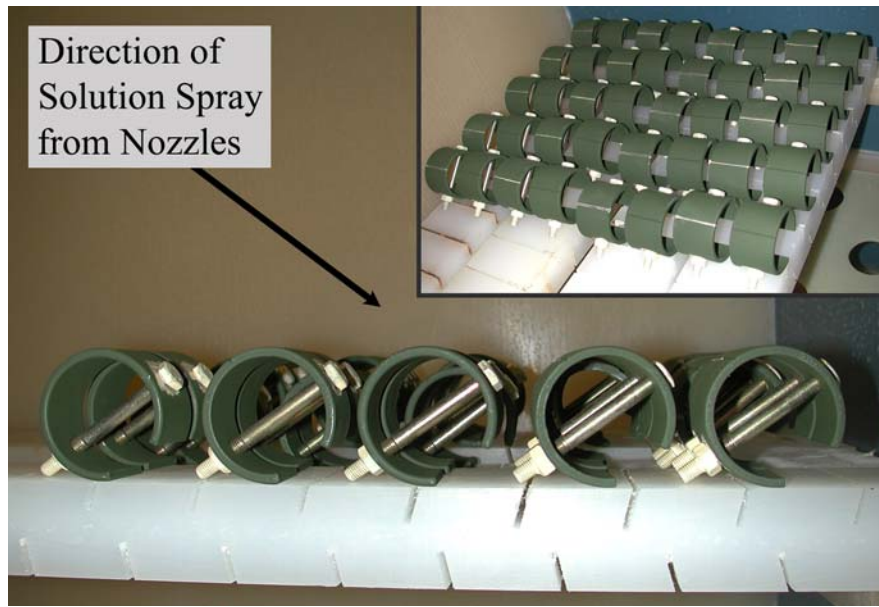


Figure 8. Damaged and undamaged 1d C-ring specimens arrayed in test chamber for GM 9540P exposure.

3. Results

3.1 General Corrosion

As expected, the general-corrosion panels exhibited major variations in performance that depended on the coating system used. The 1G system, consisting of Cd plating with the MIL-PRF-23377 class C primer, performed the best for scribed 4130 panels. This particular system showed no damage beyond the initial scribe damage until 40 GM 9540P cycles and showed absolutely no red rust up to the 30-cycle observation. Three of the five panels went the full 80-cycle duration without total failure from scribe creepback. The next best performance was from 3G, comprised of Cd plating with MIL-PRF-23377 class N primer. The 3G system was, with one exception, worse than the 1G system for every replicate; however, 3G had much better performance than the 2G and 4G cadmium-free systems. The 2G and 4G performances were comparable—there was no apparent benefit from 2G’s use of the chromated primer formulation. Figure 9 presents results typical for the different scribed coating systems with nonchromated specimens terminated well before 80 cycles from severe corrosion damage. All of the coating systems performed well in the unscribed configuration, went the full 80 cycles of GM 9540P with perfect 10 ratings, and exhibited no blistering or rust. It should be noted that although edge areas of the test panels were not rated, there was significantly more damage from rusting and edge creepback for the Cd-free coating systems (figure 10). Tables 10 and 11 give complete ASTM D 3359 ratings and observations of first red rust (designated with an asterisk) for all general-corrosion specimens (refer to table 3 for rating guide).

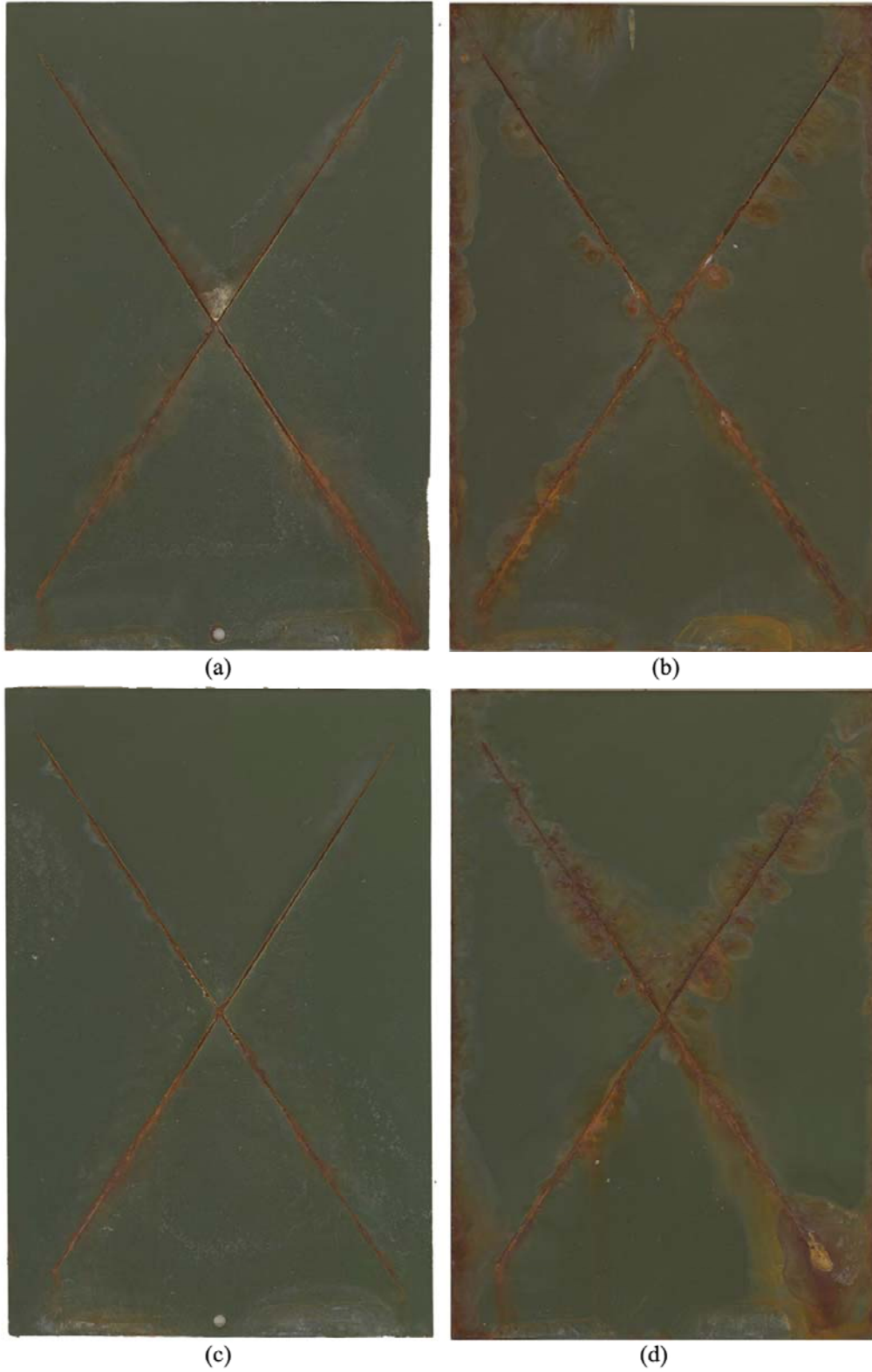


Figure 9. GM 9540P results for scribed general corrosion showing (a) 80 cycles with coating system 1, (b) 50 cycles with coating system 2, (c) 80 cycles with coating system 3, and (d) 50 cycles with coating system 4.

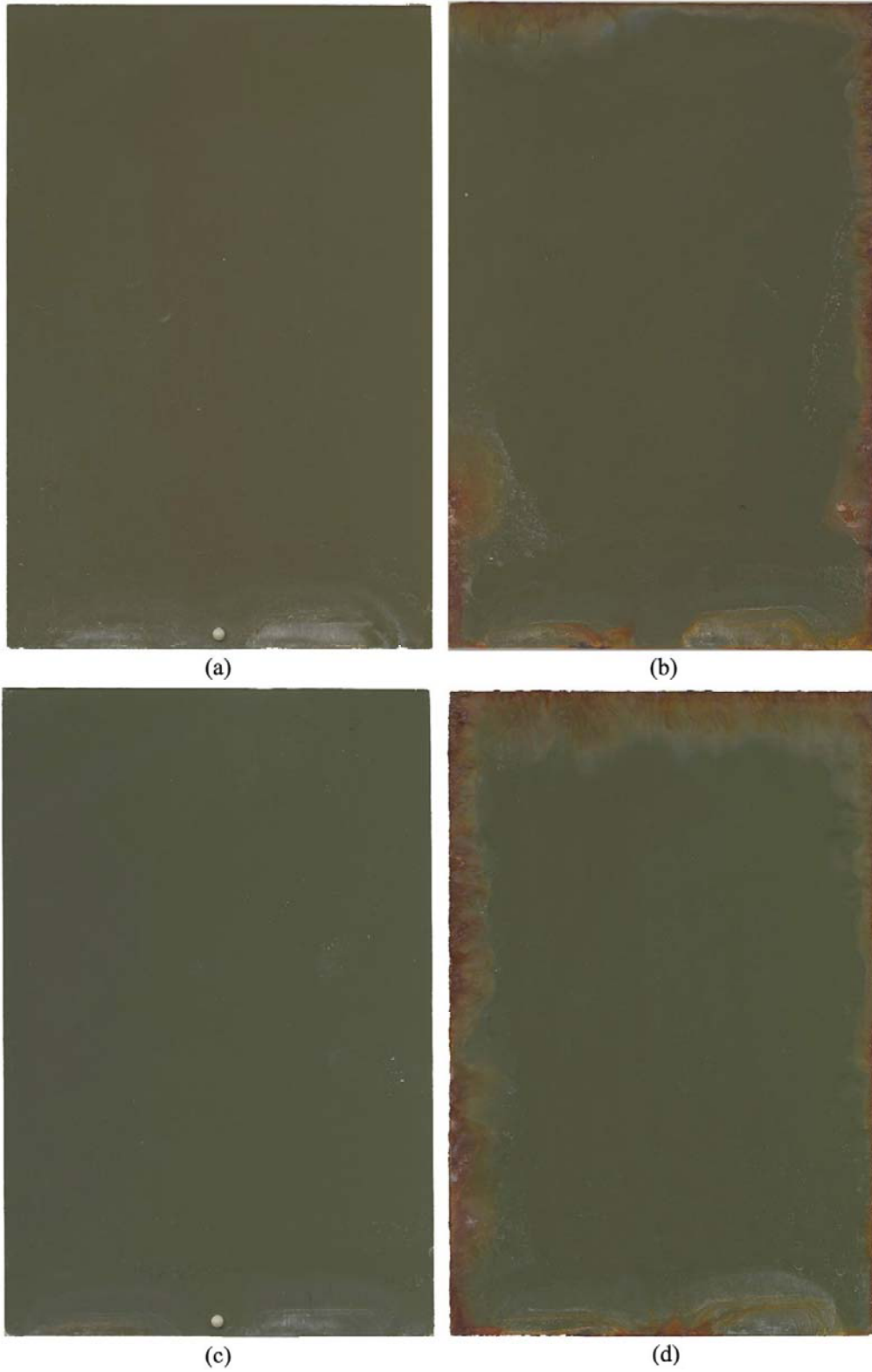


Figure 10. The 80-cycle GM 9540P results for unscribed general corrosion showing relative corrosion attack amounts at the edges for coating systems (a) 1, (b) 2, (c) 3, and (d) 4.

Table 10. ASTM D 1654 ratings for scribed general-corrosion panels in GM 9540P.

Panel #	Initial Scribe	10 Cycles	20 Cycles	30 Cycles	40 Cycles	50 Cycles	60 Cycles	70 Cycles	80 Cycles
1G	8	8	8	8*	8	7	5	4	2
1G	8	8	8	8	6	6	6	6	6*
1G	8	8	8	8	8*	6	3	2	0
1G	8	8	8	8	8*	5	3	3	0
1G	8	8	8	8	8	8*	8	4	3
2G	8	6*	5	4	3	2	1	0	
2G	9	6*	4	2	1	1	0		
2G	8	7*	5	4	2	0			
2G	8	7*	5	4	3	0			
2G	7	6*	5	3	2	0			
3G	9	8	8	8*	7	4	3	0	
3G	8	8	8	8	8	8	8*	8	8
3G	9	8*	6	4	3	3	1	0	
3G	9	9	9*	9	9	9	5	2	2
3G	8	8	8	8*	8	5	4	3	0
4G	9	6*	5	3	3	1	1	0	
4G	9	6*	4	2	1	0			
4G	9	6*	3	2	0				
4G	9	7*	4	3	2	1	1	0	
4G	8	6*	5	4	3	1	0		

Table 11. ASTM D 1654 ratings for unscribed general-corrosion panels in GM 9540P.

Panel #	10 Cycles	20 Cycles	30 Cycles	40 Cycles	50 Cycles	60 Cycles	70 Cycles	80 Cycles
1G	10	10	10	10	10	10	10	10
1G	10	10	10	10	10	10	10	10
1G	10	10	10	10	10	10	10	10
1G	10	10	10	10	10	10	10	10
1G	10	10	10	10	10	10	10	10
2G	10	10	10	10	10	10	10	10
2G	10	10	10	10	10	10	10	10
2G	10	10	10	10	10	10	10	10
2G	10	10	10	10	10	10	10	10
2G	10	10	10	10	10	10	10	10
3G	10	10	10	10	10	10	10	10
3G	10	10	10	10	10	10	10	10
3G	10	10	10	10	10	10	10	10
3G	10	10	10	10	10	10	10	10
3G	10	10	10	10	10	10	10	10
4G	10	10	10	10	10	10	10	10
4G	10	10	10	10	10	10	10	10
4G	10	10	10	10	10	10	10	10
4G	10	10	10	10	10	10	10	10
4G	10	10	10	10	10	10	10	10

3.2 Crevice Corrosion

The coating system's performances in crevice corrosion were similar to those observed in general corrosion. Tables 12–15 show the ratings for the scribed and unscribed contacting faces in the crevice assemblies at their respective removal intervals. Differences between all four coating systems in the scribed sandwich assemblies were visible by the first observation at 10 GM 9540P cycles. Figure 11 shows the crevice-corrosion differences for center coupon faces sampled at 10 cycles. Cd with chromated primer showed no corrosion damage, Cd with nonchromated primer showed rust staining in the scribe, unplated steel with chromated primer showed significant rust and scribe creepback, and unplated steel with chromate-free primer showed the most severe corrosion damage. As figure 12 shows, coating systems 1 and 3 (with Cd) still remained largely unaffected at 50 cycles across all replicates and looked significantly better than any of the Cd-free assemblies, even when compared to those removed at 10 cycles. Digital scans of unscribed Cd-free crevice assemblies (figure 13) show that, similar to the general-corrosion panels, much of the immediately obvious corrosion had originated at the panel edges. However, closely examining the center-lying regions away from the edges revealed significant blistering on the Cd-free sets. Among the unscribed sets, it was the chromated-primed, Cd-free panels that unexpectedly exhibited the worst blistering, even reaching first rust earlier on average. The blistering consisted of small blisters widely distributed over large areas, hence, the lower ASTM D 1654 ratings.

Table 12. The 10–30-cycle ASTM D 1654 ratings for scribed crevice-corrosion panels in GM 9540P.

Panel #	10 Cycles				20 Cycles				30 Cycles			
	Outer 1	Center 1	Center 2	Outer 2	Outer 1	Center 1	Center 2	Outer 2	Outer 1	Center 1	Center 2	Outer 2
1C	8	8	8	8	8*	8*	8	8	8	8	9*	8*
1C	8	8	8	8	8	9	8*	8	8	8*	8*	8
2C	5*	5*	5*	6*	4*	5*	4*	4*	4*	2*	4*	5*
2C	7*	6*	5*	6*	4*	4*	4*	5*	4*	4*	3*	3*
3C	9*	8	9*	9	9*	8*	9*	9*	6*	9*	9*	9*
3C	8*	8*	8*	7*	9*	9*	9*	9*	9*	9	9*	8*
4C	5*	6*	5*	4*	3*	3*	5*	3*	3*	3*	3*	3*
4C	4*	5*	6*	5*	4*	4*	4*	5*	3*	3*	4*	5*

Table 13. The 40–50-cycle ASTM D 1654 ratings for scribed crevice-corrosion panels in GM 9540P.

Panel #	40 Cycles				50 Cycles			
	Outer 1	Center 1	Center 2	Outer 2	Outer 1	Center 1	Center 2	Outer 2
1C	8*	8*	8*	8*	7	9	8	8
1C	8*	8*	8*	8*	8*	8*	8*	8*
2C	3*	1*	3*	4*	3*	2*	2*	3*
2C	3*	3*	4*	4*	3*	2*	3*	4*
3C	9*	9*	9*	8*	9*	9*	9*	7*
3C	7*	9*	9*	9*	7*	6*	8*	9
4C	3*	2*	3*	3*	5*	2*	4*	2*
4C	3*	3*	3*	2*	1*	2*	3*	1*

Table 14. The 20–60-cycle ASTM D 1654 ratings for unscribed crevice-corrosion panels in GM 9540P.

Panel #	20 Cycles				40 Cycles				60 Cycles			
	Outer 1	Center 1	Center 2	Outer 2	Outer 1	Center 1	Center 2	Outer 2	Outer 1	Center 1	Center 2	Outer 2
1C	10	10	10	9*	10	10	10	10	10	10	10	5
2C	3*	9*	9*	1	2*	4*	1*	1*	1	10	10	2
3C	10	10	10	10	10	10	10	10	9	10	10	10
4C	9*	10	8*	10	1*	10	10	9*	2*	9	3*	8

Table 15. The 80–100-cycle ASTM D 1654 ratings for unscribed crevice-corrosion panels in GM 9540P.

Panel #	80 Cycles				100 Cycles			
	Outer 1	Center 1	Center 2	Outer 2	Outer 1	Center 1	Center 2	Outer 2
1C	10	8	9	9	9	10	7	4
2C	0*	4	5	2	2	6	7	1
3C	7	10	10	9	10	10	10	10
4C	8	5	3	1	4	10	8	3

Figure 14 shows blistering on a coating system 2, crevice-panel face (3× magnification) after 40-cycle GM 9540P exposure. For the unscribed Cd-plated systems 1 and 3, performance was comparable at all observation cycles with no apparent advantage to system 1, which contained the chromated primer.

3.3 Throwing Power

The performance pattern established for general- and crevice-corrosion methods remained true for throwing power. Most strikingly apparent was the enormous difference in Cd-plated and unplated panel performance, irrespective of primer system, topcoat, or differences in the width of the uncoated regions. All 120 of the unplated throwing-power panels failed for red rust in less than one GM 9540P cycle and were immediately terminated. Among the chromated vs. nonchromated primer formulations and the topcoated vs. no topcoat sets across all replicates, absolutely no subtle differences were detected on the unplated panels. Figure 15 shows a sample across the entire spectrum of unplated panels with early red-rust failures (all at less than one GM 9540P cycle). Coating adhesion on the edges of the masked areas initially remained intact for all of the throwing-power panels. There were no signs of coating creepback from delamination on any of the panels, up to 120 cycles. Beyond 120 panels, the coating systems began to embrittle and lift along the masked regions and crack and peel at the panel edges. In contrast to the unplated steel, the Cd-plated throwing power panels fared significantly better and, in some cases, by more than two orders of magnitude vs. the unplated specimens. The worst of the Cd-plated throwing-power panels lasted eight cycles before rust appeared, and the best ran to an impressive 395 cycles with no rust appearing. Significant variations were noted among the panel sets. Differences of well over one order of magnitude occurred between identically prepared replicate sets of the plated specimens. Although the cycles to red rust varied widely among the Cd-plated throwing-power specimens, the corrosion progression of the Cd coating

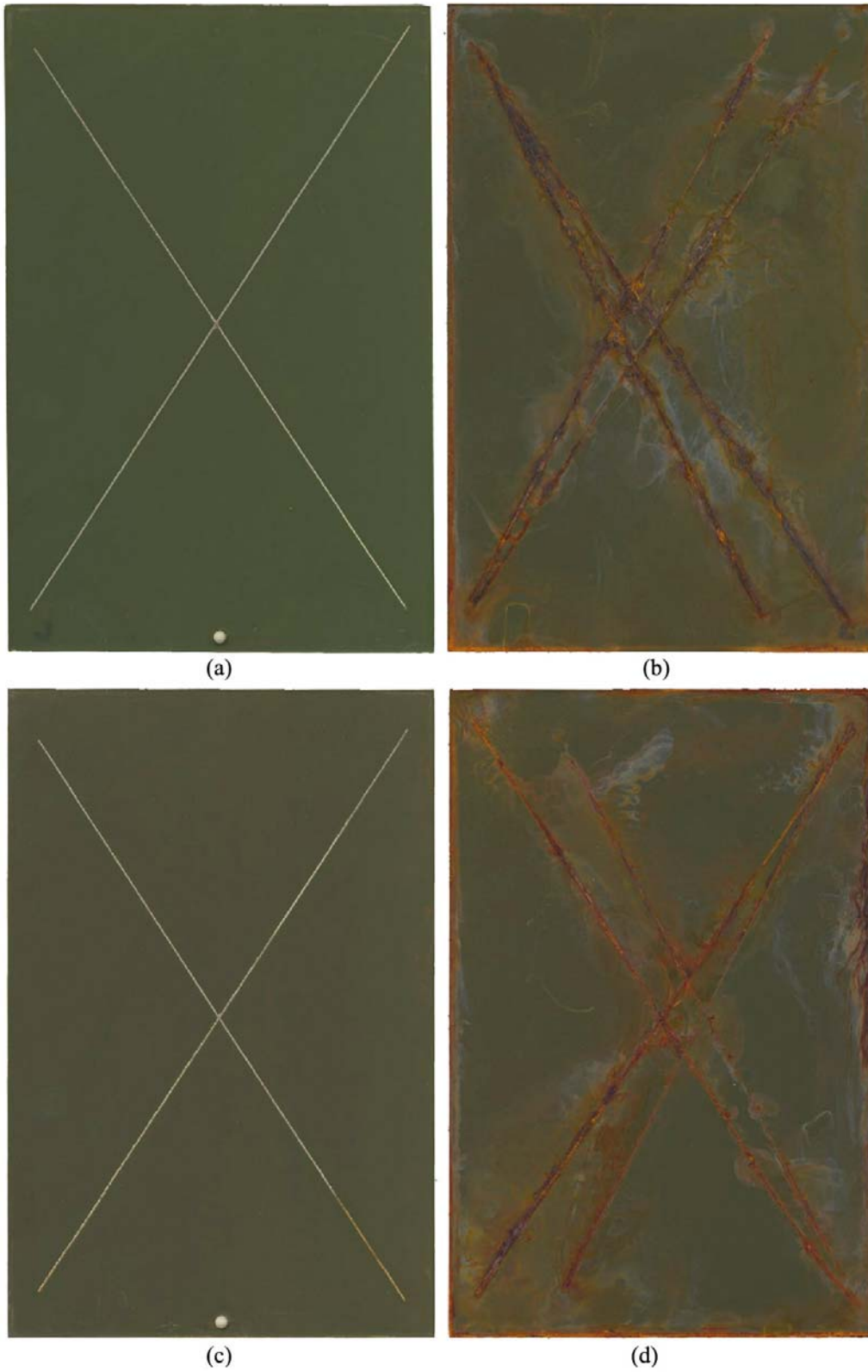


Figure 11. The 10-cycle GM 9540P results for scribed crevice corrosion showing relative corrosion severities for coating systems (a) 1, (b) 2, (c) 3, and (d) 4.

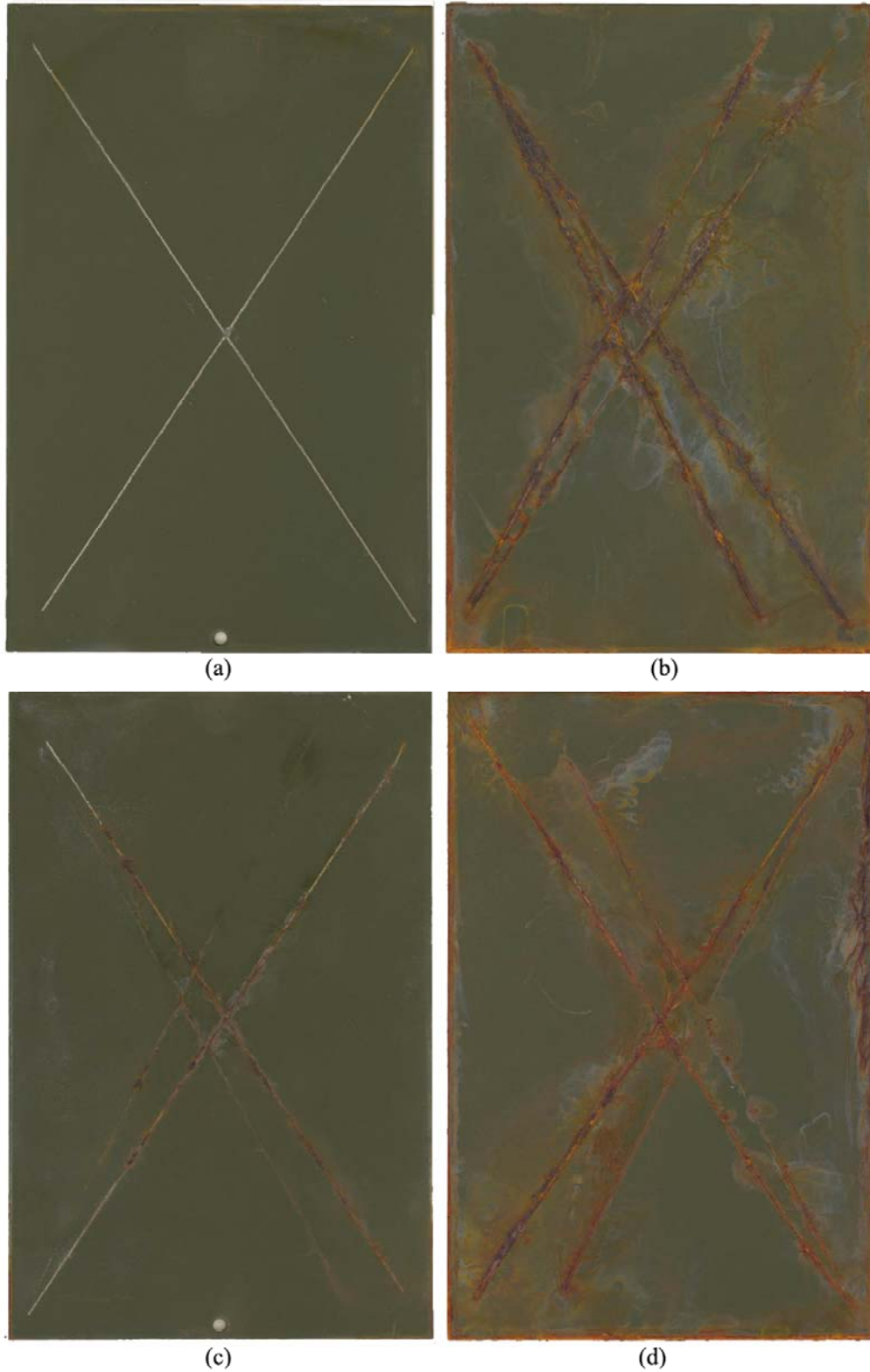


Figure 12. GM 9540P scribed crevice-corrosion damage for (a) coating system 1 at 50 cycles, (b) coating system 2 at 10 cycles, (c) coating system 3 at 50 cycles, and (d) coating system 4 at 10 cycles.

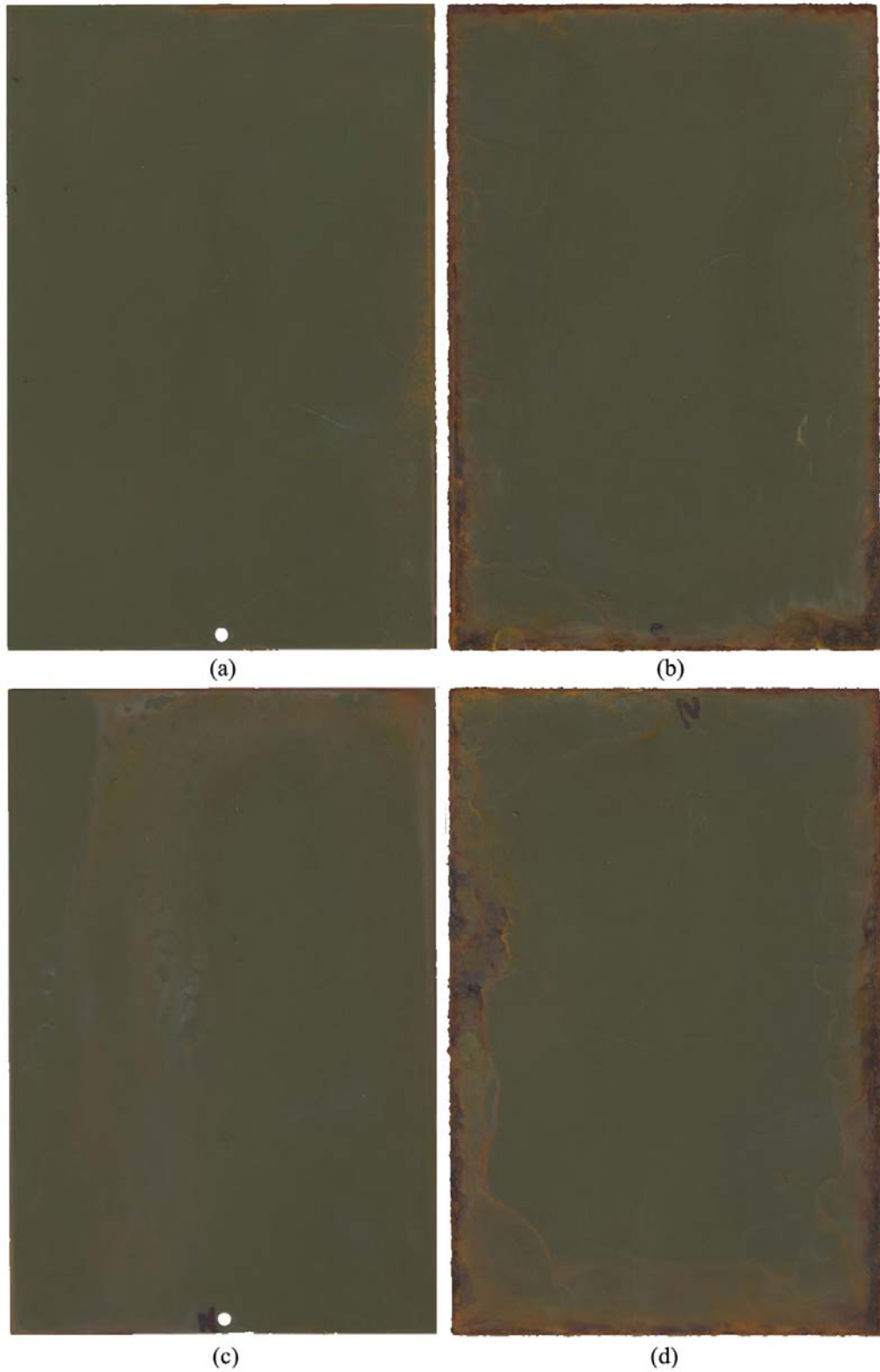


Figure 13. The 60-cycle GM 9540P results for unscribed crevice corrosion showing relative corrosion at the edges for coating systems (a) 1, (b) 2, (c) 3, and (d) 4.

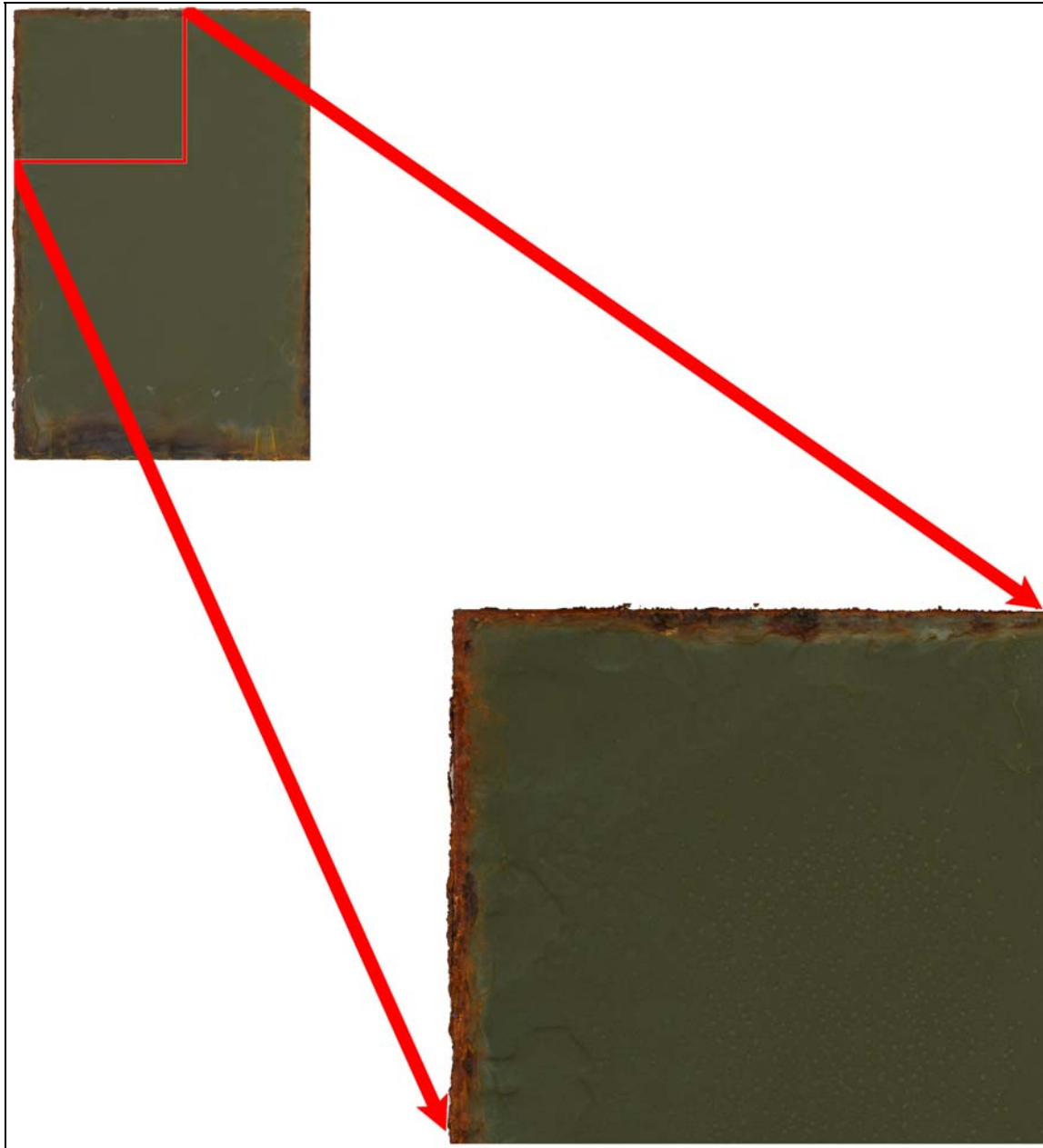


Figure 14. Unscrubbed crevice corrosion showing coating blistering (3× magnification) for coating system 2 after a 40-cycle GM 9540P exposure.

followed a basic sequence of steps. Initially, white Cd corrosion products and blotching formed, followed by dark gray to black blotches of oxidized Cd, followed by growing exposed areas of gray unrusted steel or chromate-depleted Cd, and, finally, red rust. Figure 16 shows the five steps in the Cd-plating corrosion progression. Tables 16 and 17 show the complete GM 9540P red-rust data for Cd-plated throwing-power specimens (all masked widths), with and without topcoats.

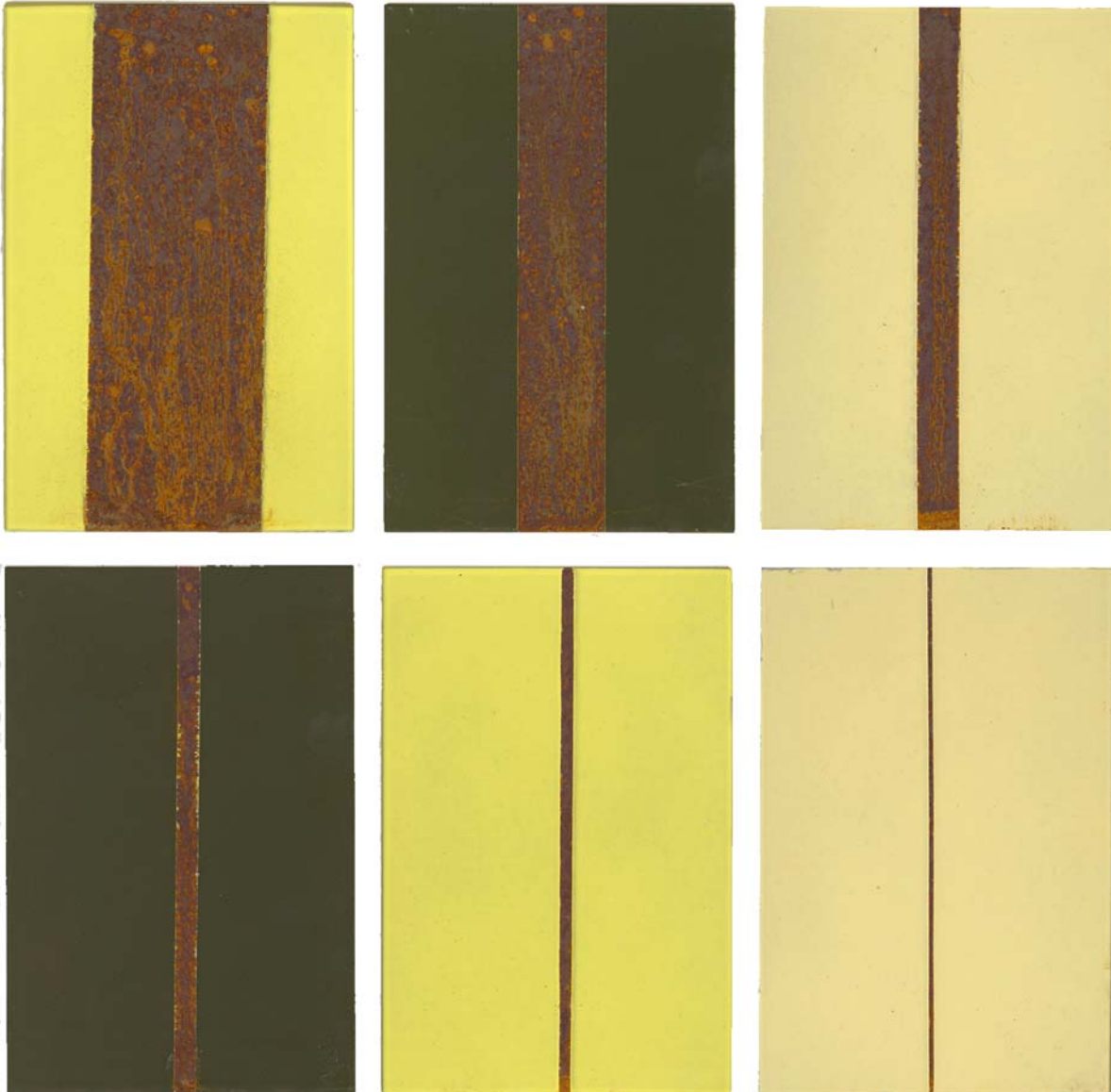


Figure 15. Cd-free throwing-power test panels terminated at less than one GM 9540P cycle.

In an attempt to detect trends among the Cd-plated throwing-power sets, bar graphs (figures 17 and 18) were plotted using the three median values for the five replicates of each set. Examining the data reveals some interesting trends. When chromated primer is topcoated, much of the primer layer's benefit is negated, as seen when comparing the primed and topcoated 2-in bare-stripped chromated-primer panels against those with primer only. Among the topcoated panels, there seemed to be no clear difference between chromated and nonchromated panels. The chromate-primer topcoated panels performed better at 0.0625-, 0.25-, and 0.5-in uncoated stripe widths, while the nonchromate-primed panels performed better at 0.125-, 1-, and 2-in

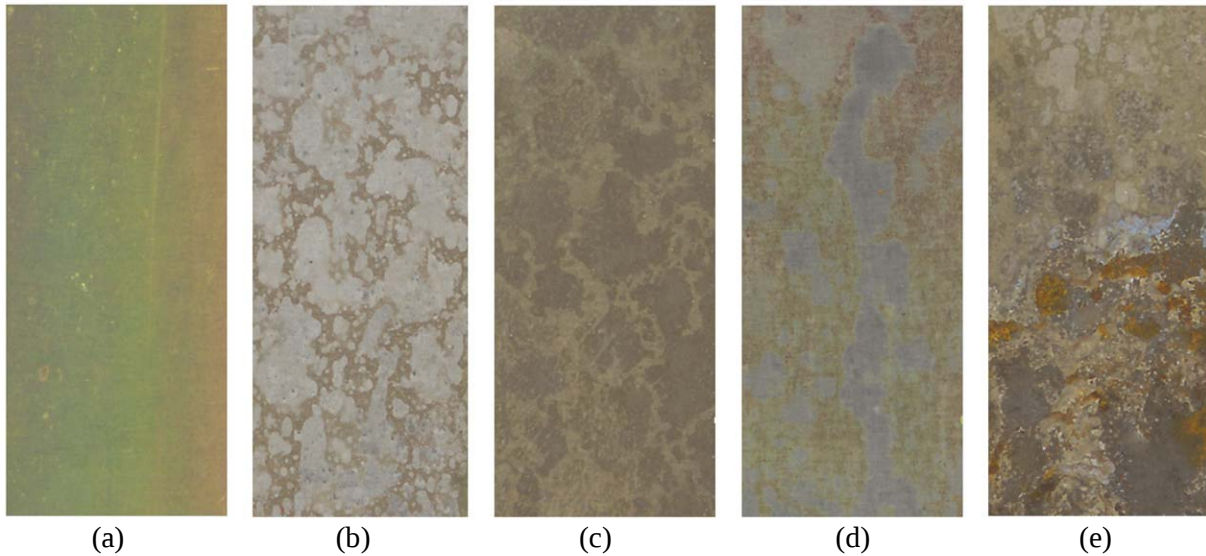


Figure 16. Magnification (5×) of the Cd-plating corrosion progression: (a) initial, (b) white Cd corrosion products and blotching, (c) dark gray to black blotches of oxidized Cd, (d) exposed areas of gray unruled steel or chromate-depleted Cd plating, and (e) rusting of steel substrate.

stripe widths. For primer-only throwing-power panels, the chromated-primer panels were clearly superior, performing better in five of six bare-stripe widths. The exception occurred at the 0.125-in bare-stripe width: one chromated-primer panel did significantly worse at just 37 cycles, while two other panels from the same set performed significantly better at 364 and 395 cycles (compared to the more tightly grouped nonchromated primer data between 129 and 220 cycles).

Interestingly, the 0.125- and 2-in chromated-primer systems performed better than or equal to their topcoated counterparts within the same uncoated-stripe widths.

3.4 Pull-Off Adhesion

Adhesion results differed more as a function of primer formulation than within surface-preparation methods. While collecting adhesion measurements, over 1000 data points were entered. Listing all would require several pages of tables; however, the basic statistics for each of the coating systems and conditions are included in tables 18–22. For the Cd-free steels, the nonchromated MIL-PRF-23377 class N showed little to no variation among the various ambient exposure times between abrasive blasting and coating application. All of the pull-off failure modes for the nonchromated primer were cohesive—meaning the adhesion to the substrate, although unknown, was consistently above the cohesive strength of the primer itself, even on the smooth-profiled, Cd-plated and unplated, solvent-wiped, mill-finish, steel panels. For the abrasive-blasted panels, the chromated MIL-PRF-23377 generally had higher pull-off tension values, compared to the nonchromated primer, but it was less consistent with its pull-off

Table 16. Complete GM 9540P data for throwing-power panels without topcoat.

Masked Area Width (in)	Coating System 1 (cycles)					Coating System 2 (cycles)					Coating System 3 (cycles)					Coating System 4 (cycles)				
0.0625	29	32	91	138	177	1	1	1	1	1	18	48	48	63	91	1	1	1	1	1
0.125	37	139	143	364	395	1	1	1	1	1	129	129	136	158	220	1	1	1	1	1
0.25	21	32	103	136	218	1	1	1	1	1	8	24	67	138	333	1	1	1	1	1
0.5	29	44	53	59	143	1	1	1	1	1	23	32	44	61	74	1	1	1	1	1
1	8	8	44	59	129	1	1	1	1	1	8	8	8	14	79	1	1	1	1	1
2	33	71	163	174	192	1	1	1	1	1	8	8	23	44	59	1	1	1	1	1

Table 17. Complete GM 9540P data for throwing-power panels with full-coating systems.

Masked Area Width (in)	Coating System 1 (cycles)					Coating System 2 (cycles)					Coating System 3 (cycles)					Coating System 4 (cycles)				
0.0625	37	101	123	198	334	1	1	1	1	1	8	18	24	129	136	1	1	1	1	1
0.125	44	48	100	200	259	1	1	1	1	1	129	129	136	185	395	1	1	1	1	1
0.25	29	48	100	246	252	1	1	1	1	1	8	18	56	97	198	1	1	1	1	1
0.5	18	44	101	152	198	1	1	1	1	1	44	44	56	97	107	1	1	1	1	1
1	8	8	18	23	44	1	1	1	1	1	21	44	56	105	200	1	1	1	1	1
2	8	8	8	29	225	1	1	1	1	1	29	44	53	56	200	1	1	1	1	1

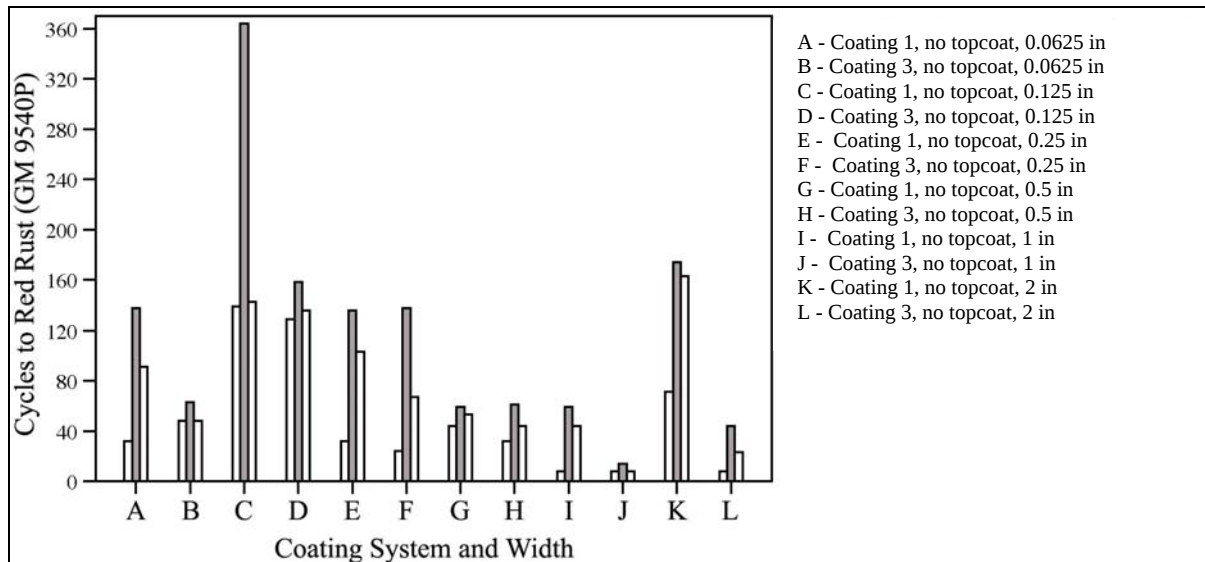


Figure 17. Coating system and throwing-power masked-area width (no topcoat) vs. GM 9540P cycles to red rust.

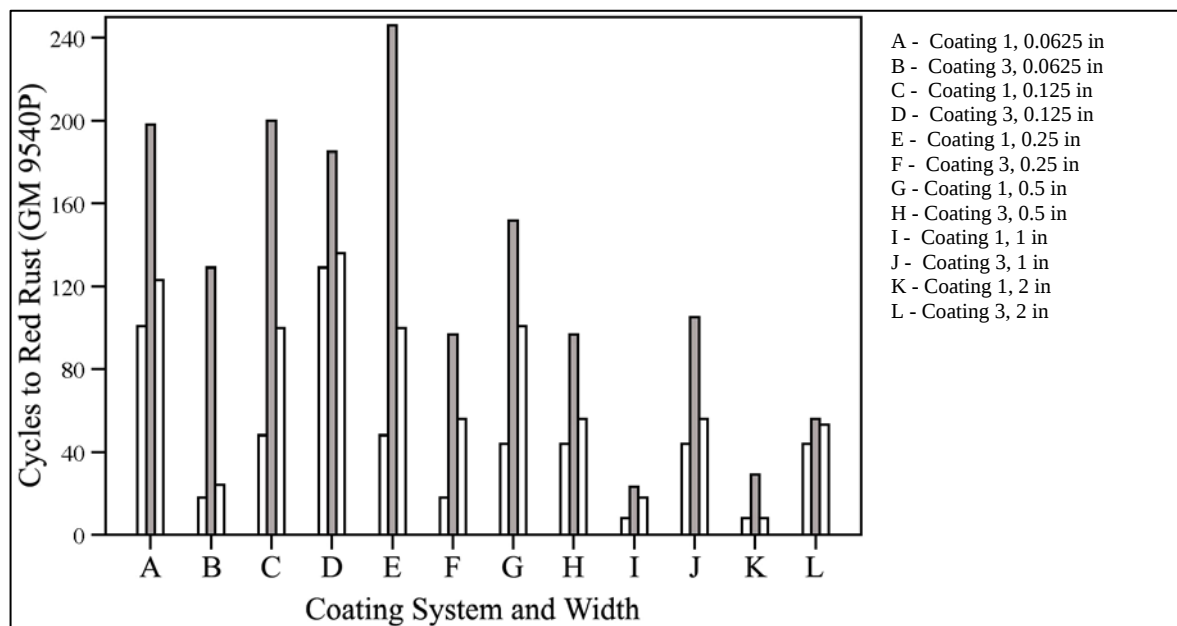


Figure 18. Coating system and throwing-power masked-area width vs. GM 9540P cycles to red rust.

failure modes. While the vast majority of the pull-off failures were cohesive within the primer layer, there were occasional instances of adhesive failure between the topcoat and primer and between the primer and the substrate. Introducing nitrogen-sealed packaging produced little or no difference. Adhesion averages between unsealed and sealed counterparts were mixed. Average adhesion values for open-air or nitrogen-sealed exposure times showed a mainly upward trend (plotted in figure 19). For the smoother surface, Cd-plated and unplated,

Table 18. Pull-off adhesion data for smooth-profiled panels.

Panel	1	3	2M	4M
Exposure	No dwell	No dwell	No dwell	No dwell
Average	1594.77	1849.39	1783.18	1829.38
Standard Deviation	462.45	183.33	421.3	128.8
Geometric Mean	1531.8	1839.41	1734.78	1824.94
Median	1475	1860	1705	1810
95% Confidence	136.64	51.33	124.48	36.44
Maximum	2530	2300	2550	2090
Minimum	900	1100	95	1600

Table 19. Pull-off adhesion data for abrasive-blasted coating, system 2 panels with open-air exposures.

Panel	2A	2B	2C	2D	2W
Exposure (min)	15	30	60	120	40
Average	2276.38	2323.95	2373.95	2362.61	2440.2
Standard Deviation	353.81	244.09	325.44	318.05	320.18
Geometric Mean	2252.05	2313.45	2352.78	2342.32	2419.69
Median	2140	22.95	2380	2315	2430
95% Confidence	101.15	73.82	97.27	91.91	89.65
Maximum	3320	2840	3530	3380	3110
Minimum	1770	1870	1680	1770	1800

Table 20. Pull-off adhesion data for abrasive-blasted, coating system 2 panels with nitrogen-sealed exposures.

Panel	2AS	2BS	2CS	2DS	2ES
Exposure (min)	15	30	60	120	40
Average	2367.73	2164.13	2372.56	2383.49	2469.05
Standard Deviation	312.74	334.01	304.21	301.06	305.02
Geometric Mean	2347.92	2139.96	2353.73	2365.25	2451.37
Median	2335	2110	2360	2330	2420
95% Confidence	92.41	96.52	90.93	89.98	92.25
Maximum	3180	2920	3080	3120	3160
Minimum	1700	1630	1890	1850	2000

Table 21. Pull-off adhesion data for abrasive-blasted, coating system 4 panels with open-air exposures.

Panel	4A	4B	4C	4D	4E
Exposure (min)	15	30	60	120	40
Average	1864.90	1977.84	1921.04	1958.37	1992.71
Standard Deviation	233.45	130.83	139.10	101.89	133.51
Geometric Mean	1852.60	1973.67	1916.12	1955.78	1988.27
Median	1810	1980	1895	1950	2000
95% Confidence	64.07	35.91	39.35	28.53	37.77
Maximum	2880	2250	2210	2180	2200
Minimum	1520	1780	1630	1790	1700

Table 22. Pull-off adhesion data for abrasive-blasted, coating system 4 panels with nitrogen-sealed exposures.

Panel	4AS	4BS	4CS	4DS	4ES
Exposure (min)	15	30	60	120	40
Average	1856.27	1918.98	1940.82	1957.87	1963.54
Standard Deviation	167.15	142.95	137.26	154.45	140.17
Geometric Mean	1849.15	1913.64	1936.06	1951.92	1958.69
Median	1810	1920	1940	1950	1970
95% Confidence	45.87	40.02	38.43	44.16	39.65
Maximum	2310	2180	2230	2290	2360
Minimum	1510	1560	1620	1610	1620

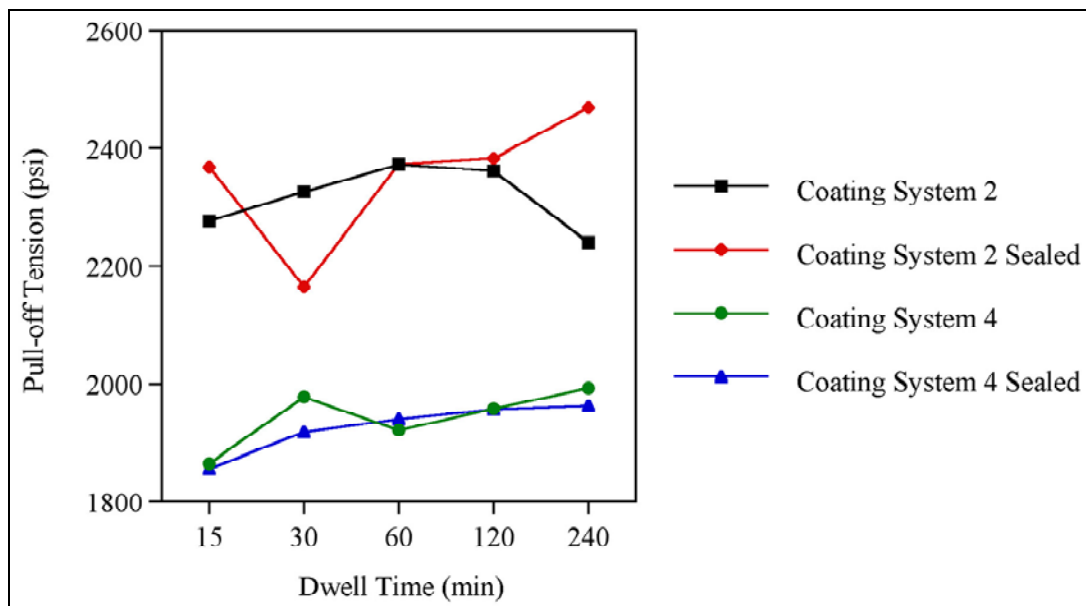


Figure 19. Average pull-off tensions vs. dwell time for abrasive-blasted Cd-free systems.

solvent-wiped, mill-finish panels, the chromated-primer pull-off failures were adhesive at the primer-substrate interface, with average tension values lower than the nonchromate primer panels, which all failed cohesively. This was the case in all instances, with two exceptions: coating system 2 and mill finish. Figure 20 highlights the differences in pull-off failure modes on the smooth-profiled, Cd-plated and mill-finish steel surfaces, where the substrate can clearly be seen on the chromated primer pull-off panels.

3.5 Hydrogen Embrittlement

Despite the 40% lower notch-bend fracture load, the type 1d C-rings displayed significant differences among the plating, primer, and damage to the coating at the C-ring notch. Table 23 lists the complete GM 9540P cycles to fracture for the C-rings. Figure 21 plots the coating system damage GM 9540P cycles to failure using the three median values from the five replicates for each of the configurations. The Cd-plated C-rings with chromated primer

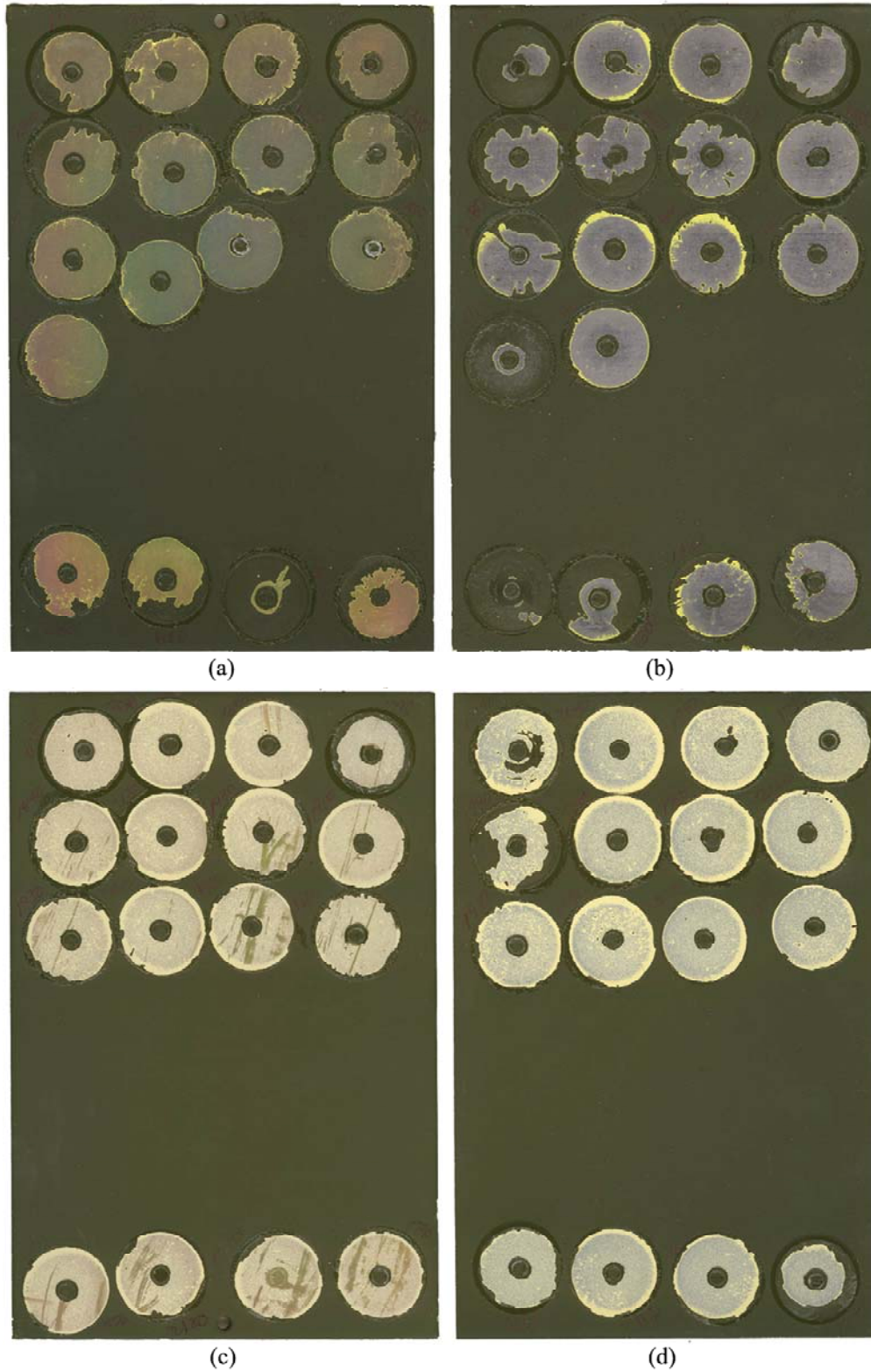


Figure 20. Pull-off failure modes for coating systems (a) 1, (b) 2 on unblasted mill-finish steel, (c) 3, and (d) 4 on unblasted mill-finish steel.

Table 23. Complete GM 9540P cycles to fracture for damaged and undamaged type 1d C-rings.

Designation	Coating System Description	GM 9540P Cycles to Fracture (Replicates 1–5)				
1	Cd plating with MIL-PRF-23377C	1	1	15	1	1
1D	Cd plating with MIL-PRF-23377C, damaged	1	4	6	1	1
2	Unplated with MIL-PRF-23377C	80	80	80	80	80
2D	Unplated with MIL-PRF-23377N, damaged	7	9	48	9	8
3	Cd plating with MIL-PRF-23377C	4	15	26	8	4
3D	Cd plating with MIL-PRF-23377C, damaged	1	1	5	1	1
4	Unplated with MIL-PRF-23377N	4	54	71	64	48
4D	Unplated with MIL-PRF-23377N, damaged	1	3	3	2	1

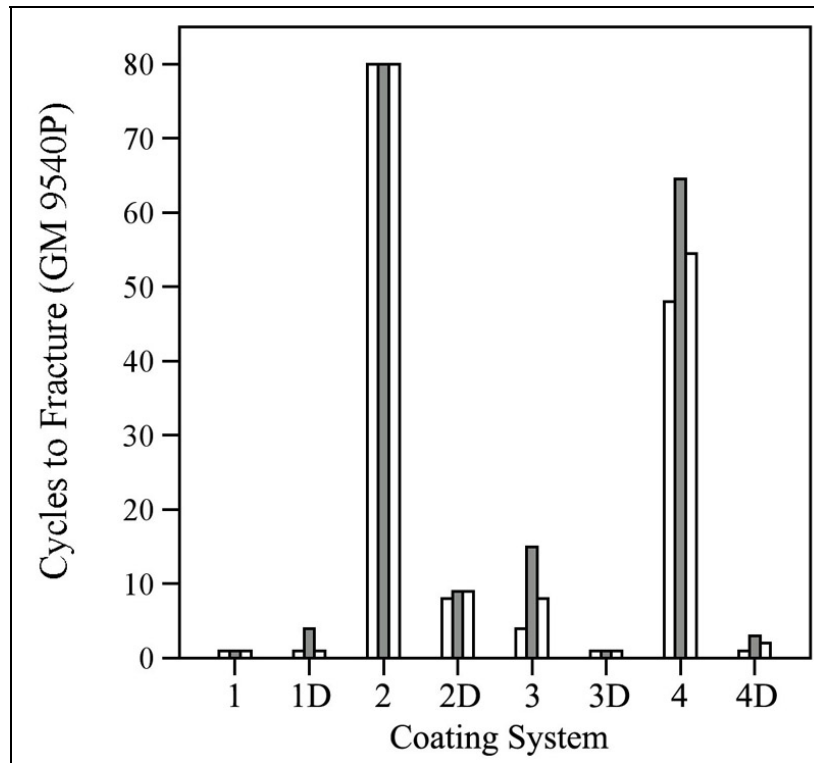


Figure 21. Coating-system damage vs. GM 9540P cycles to C-ring fracture.

(damaged and undamaged conditions) performed the worst. Interestingly, the undamaged Cd-plated C-rings with nonchromated primer lasted several more cycles to fracture than their chromated counterparts. The unplated system with chromated primer (in damaged and undamaged conditions) performed the best overall, followed closely by the unplated system with nonchromated primer.

4. Discussion

The purpose of this study was to ascertain current coating systems' abilities to fulfill their functions without Cd plating. For current aviation systems, eliminating Cd may be possible but perhaps only in nonstructural applications, such as low-strength, bolt-on components that can be readily serviced. Even if such noncritical components are identified, a sacrificial coating other than Cd is still recommended.

The general, crevice-corrosion, and throwing-power exposures under GM 9540P defined the extent of the essential role that electroplated Cd plays in protecting aviation materiel when damage or defects to the coating system are present. One immediately obvious difference among the coating systems (with and without Cd) was seen before a test was even completed. In crevice corrosion, before they were even disassembled, the sandwich-type assemblies showed major differences in corrosion damage. Figure 22 displays the major differences between coating systems 1–4, with Cd-based systems 1 and 3 clearly superior to the noncadmium-based systems. The contrast among the throwing-power panels was especially dramatic—not a single Cd-free panel withstood one GM 9540P cycle without severe damage from rust. At their absolute worst, the Cd panels withstood eight cycles, with many lasting significantly beyond 120 cycles. As for the large performance disparities among the Cd-plated panels, it was initially thought that the wider-striped throwing-power panels (especially the 2 in) corroded sooner solely because of localized crevice-corrosion conditions introduced by contact with the holding rack. This contact area (seen in figure 23) had tight tolerances. For regions located higher up on the panels and for narrower-striped panels that only contacted the holding racks in located areas, further corrosion progression of the Cd plating and chromate depletion originated from additional capillary wetting of corrosive solution. Interestingly, the corrosion problem introduced by the rack configuration provided some information on the primer coating's contribution to throwing power. The 2-in masked area of chromated MIL-PRF-23377 class C panels without topcoat outlasted all other panels for that group, including its identical counterparts with topcoat. It is likely the chromate from the large exposed areas of the chromated primer may have contributed some additional inhibition (from transport through the wet film) during the wet stages of GM 9540P, such as spray and high-humidity cycles. This additional chromate supplied from the primer was not freely available on the topcoated panels, and, thus, corrosion failure from red rust occurred much sooner for those panels.

While the holding-rack crevice may have indeed accounted for the earlier failures on wider-masked gaps, the wide scatter in the data for the five throwing-panel replicates remained puzzling. In order to diagnose the discrepancies, samples of the worst-performance 8-cycle panels and the best-performance 395-cycle panels were sectioned, mounted, and polished



Figure 22. Clamped, unscribed crevice assemblies at 80 cycles of GM 9540P showing relative damage among coating systems 1–4 (from left to right).



Figure 23. Nucleation of corrosion to lower areas of panels from specimen racks used in GM 9540P.

for scanning electron microscope (SEM) examination. During sectioning, care was taken to use interior portions of the coated regions on the sample panels. This minimized exposure to edge areas of the bare regions or the actual panel edges. The remaining portions of the samples were also immersed in methylene-chloride-based chemical stripper to remove the CARC paint layers, which facilitated in visually examining the substrate and inorganic coating layers. The chemical stripper revealed substrate areas completely devoid of Cd plating with less corresponding chromate seal. Figures 24 and 25 show the major difference in Cd-plating coverage and the differences in the chromate sealer integrity. This wide distribution of data, even among identically prepared panels indicates wide variations in the quality of Cd plating—from partial substrate coverage with voids to complete coverage with accompanying variations in plating thickness. Examining these panels under SEM revealed Cd-plating layers ranging from 2 to 3 μm , eight-cycle sections measuring 2 and 2.5 μm , and 395-cycle specimens measuring 2 and 3 μm . The minimal thickness variation between the early-failure samples and the

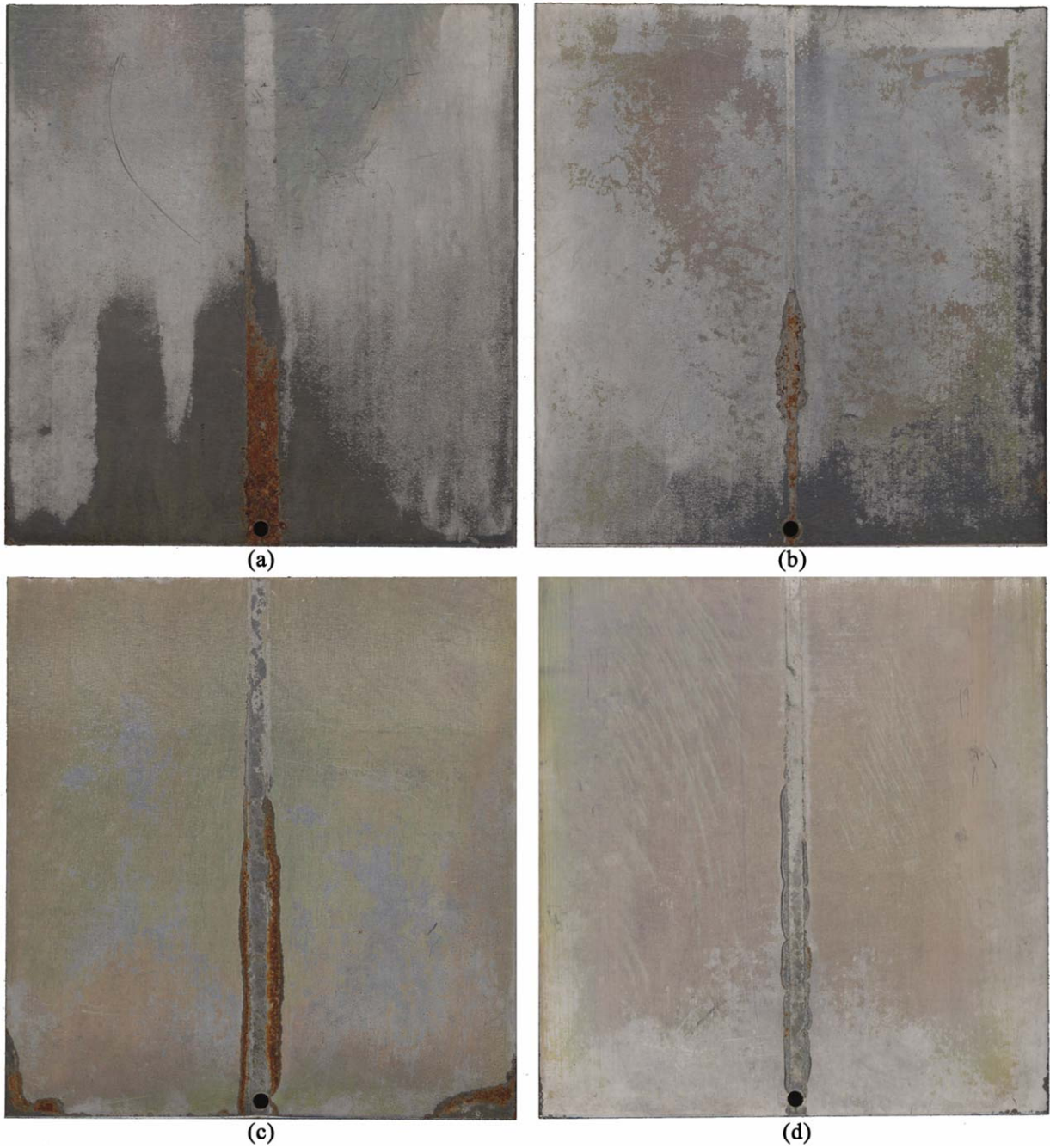


Figure 24. Throwing-power test panels chemically stripped to reveal variations in Cd-plating uniformity: (a and b) panels failing earliest (eight GM 9540P cycles) with obvious Cd-depleted regions and (c and d) panels lasting longest (395 GM9540P cycles) with complete Cd coverage and better uniformity of the chromate layer (magnified 0.75 $\times$).



Figure 25. Magnified (1.5 $\times$) view of an eight-cycle, failed throwing-power test panel (light grey streaks show Cd distribution, bare dark grey steel substrate are areas with no Cd, and the rusted region corresponds to the original throwing-power masked area [Cd free]).

best-performance samples indicates that plating thicknesses were adequate when they were present. Problems occurred on panels where the Cd did not plate. The sectioned panel portions used for measuring thickness were obviously representative of the intact portions of the affected panels. The Cd-free areas seen on the eight-cycle test panels were likely from deficiencies in plating-bath circulation, surface preparation, or cleaning processes. Before the Cd-plated panels were coated, significant differences in color and mottling patterns were noticed. Figure 26 illustrates some typical variations in uniformity of appearance from panel to panel. The earliest failures (with 20 cycles or less) were most likely from gross defects in the Cd-plating quality. For panels that lasted beyond 200 cycles, longevity was likely increasingly due to organic-coating degradation with edge lifting that exposed fresh Cd-plated surface area and corresponding chromate. For those remaining panels (figures 27 and 28), supplemental chromate nourishment emanating from the backside of the lifting or peeling chromated primer and from the Cd plating itself may have aided in significantly extending the no-red-rust performance of the panels. Moreover, exposed Cd from regions outside of the evaluation area (such as the edges and panel backs, where the coatings embrittled, cracked, and lifted from the additive damage of constant GM 9540P cycling) may have also contributed additional late-stage throwing power to prevent the nucleation of red rust. Aside from the infantile failures under 20 cycles, the degree

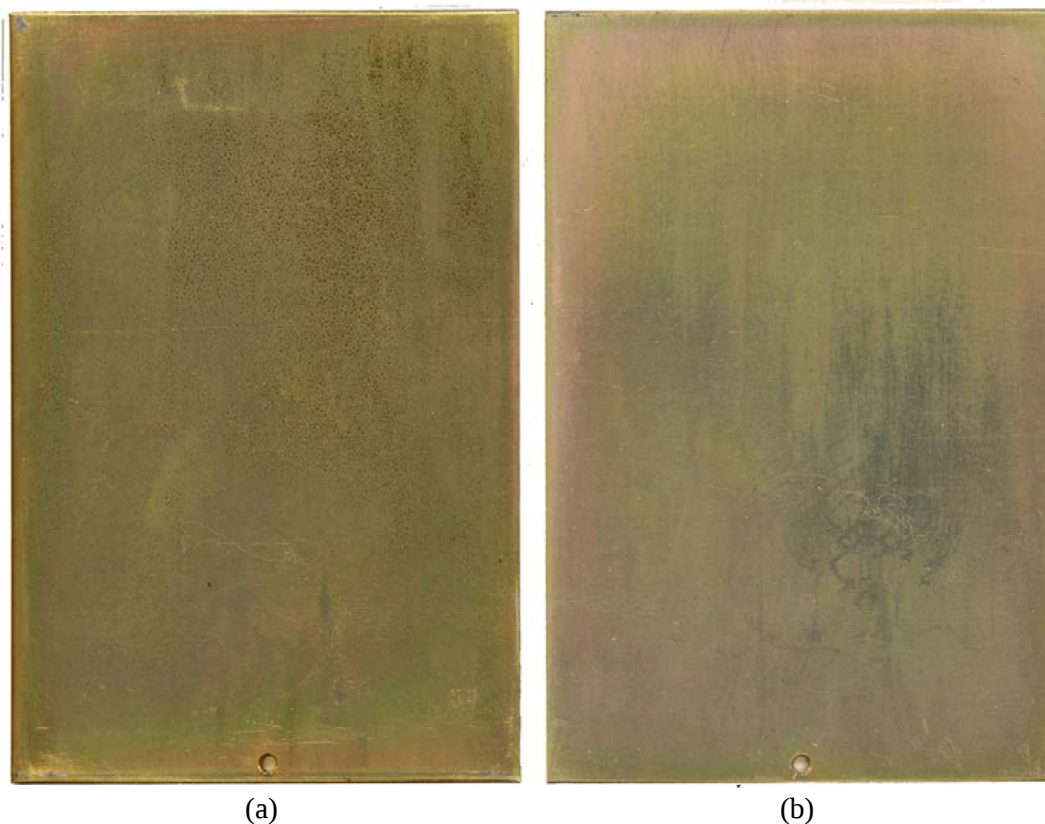


Figure 26. Variations among cadmium plated test panels showing (a) mottling and (b) uniform conditions.

to which differences in Cd plating altered corrosion or adhesion performance was likely overshadowed by the extra corrosive environment introduced locally by the specimen rack, especially in the panels with the wider-masked areas. It is likely that the contribution from Cd-plating appearance in the over-20-cycle panels was much smaller, because the narrower masked panels (under 0.5 in) lasted significantly longer overall than their wider masked counterparts. For any future throwing-power evaluations, it is recommended that the masked-off bare regions stop 1 in short of the test panel's bottom edge.

Evaluating adhesion among the unplated systems with varying dwell times after abrasive blasting revealed (from the lack of any differences among the dwell times and packaging) that the ambient conditions of the laboratory environment were not harsh enough to show any differences. Aviation depots, such as Corpus Christi Army Depot, have much greater relative-humidity values and possibly even some chlorides. The most notable observation was the differences in adhesion behavior between the chromated and nonchromated formulations of the MIL-PRF-23377 primer. Qualitatively, the chromated formulation appeared to be a stiffer, higher tensile-strength coating, evidenced by the higher pull-off tension value on the abrasive-blasted panels where a strong mechanical bond was possible. However, when pull-off was

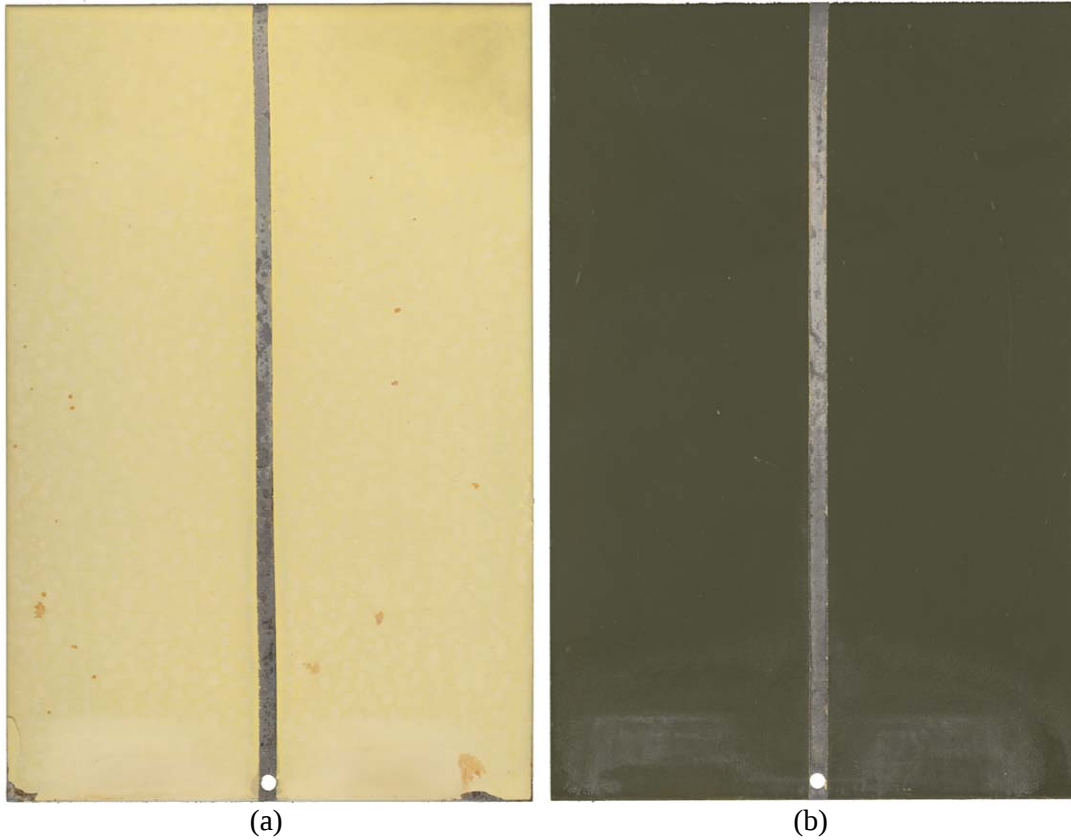


Figure 27. Coatings lifting and peeling on Cd-plated, 0.125-in masked panels after 395 cycles of GM 9540P with (a) MIL-PRF-23377C and (b) MIL-PRF-23377N with MIL-DTL-64159.

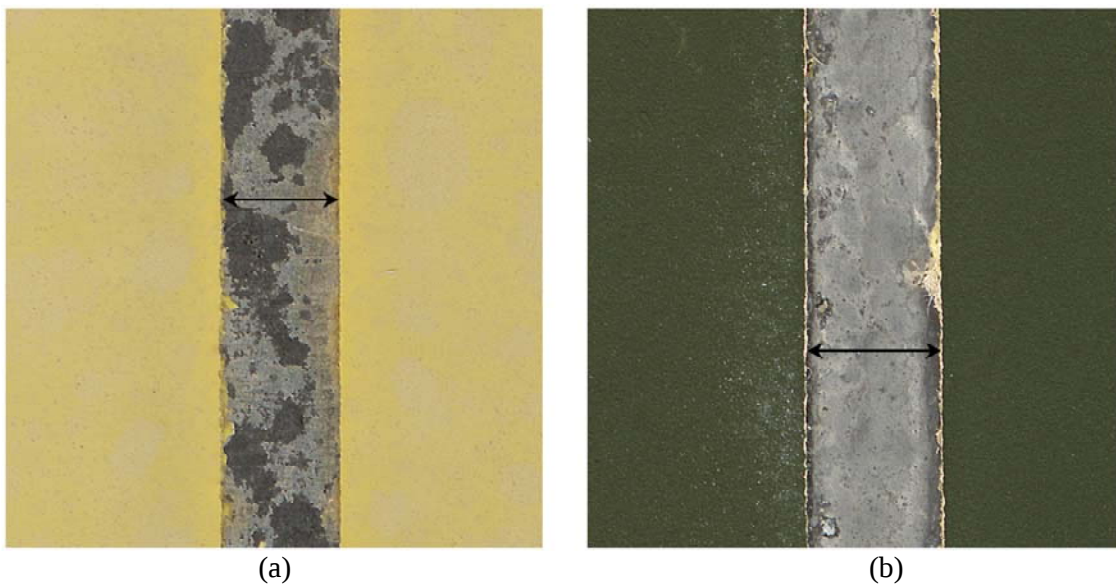
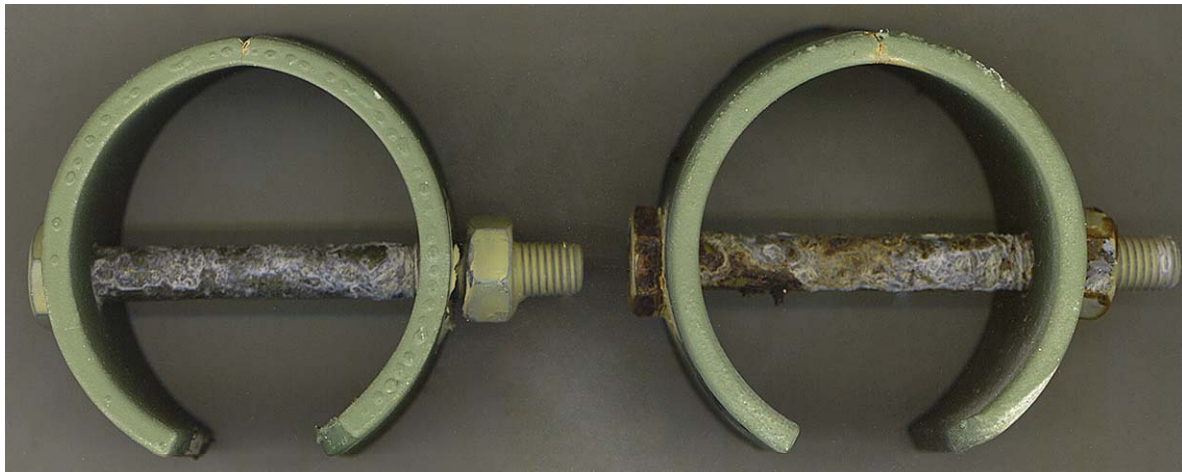


Figure 28. Magnified images (6×) of Cd-plated, 0.125-in masked panels after 395 cycles of GM 9540P with (a) MIL-PRF-23377C and (b) MIL-PRF-23377N with MIL-DTL-64159 (arrows indicate areas where coating is lifting).

evaluated for smooth-profiled systems (coating systems 1 and 3 with Cd plating or the mill-finished steel panels), it was the nonchromated primer that had the higher pull-off tension value with cohesive failure modes (compared to the lower tension values and adhesive failures for the chromated primer). This suggests a lower tensile strength but greater overall adhesion to the substrate.

Additional data produced in the C-ring testing suggests improved flexibility in the nonchromated (vs. the chromated) primers. Referring once again to figure 21, undamaged coating system 3 (containing nonchromated primer) lasted significantly longer than coating system 1 (containing chromated primer), yet, when damaged, it performed only as well or slightly worse than coating system 1. These differences suggest that the chromated MIL-PRF-23377 class C primer coating on undamaged system 1 was more brittle and cracked at or near the notch during the initial 40% loading, while the nonchromated primer was more pliable and sustained the flexibility to stretch without cracking. Therefore, it resisted the intrusion of corrosive solution and subsequently endured more cycles before fracturing. Only the undamaged Cd-free coating systems 2 and 4 demonstrated an advantage to class C primers—all five replicates of coating system 4 had fractured by 71 cycles, while the coating system 2 C-rings endured GM 9540P exposures beyond 80 cycles. All but one of the undamaged coating system 4 C-ring assemblies exhibited, just prior to fracture, rust staining on the edges or sides adjacent to the notch. The undamaged coating system 4 C-ring was the exception; though free of rust staining, it exhibited coating blistering in close proximity to the notch and fractured earliest (after just four GM 9540P cycles). High-resolution scans of the coating system 4 C-ring side and edge damage are provided in figure 29. In sharp contrast, all of the undamaged coating system 2 C-rings remained free of rust staining or blistering on the sides and edges adjacent to the notch through 80 cycles, indicating good corrosion inhibition by the class C primer.

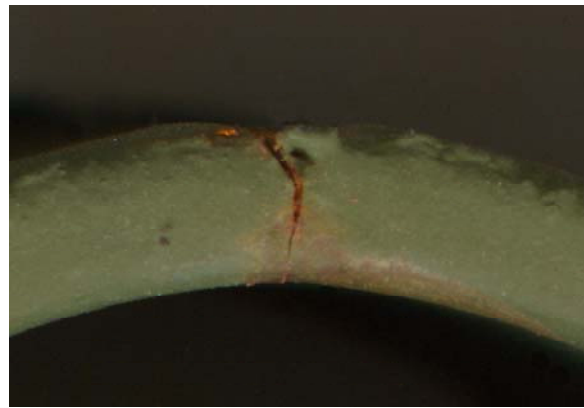
The C-rings performed differently than expected. It was apparent that the Cd-plating process itself imparted a great deal of hydrogen to the specimens. As previously stated, the original plated C-ring specimens did not meet the in-air, 200-hr, sustained-load requirement at 65% notch fracture strength (NFS). This was indicative of a poor plating process and/or an inadequate hydrogen bake relief cycle (either through time, temperature, or dwell time between plating before baking). In the interest of time, the loading protocol of the specimens was lowered to the point at which they would meet the 200-hr, sustained-load requirement. This level proved to be 40% of the NFS. However, this did not negate the fact that the specimens contained a significant amount of hydrogen and hydrogen damage. It was observed in the data that all the Cd-plated specimens failed significantly before the rest of the matrix. Undamaged Cd-coated specimens should have been among the best performers in the matrix (they should have been the most resistant to the addition of hydrogen from the normal corrosion process). In their condition, they were at the brink of failure going into the test. The small amount of additional hydrogen they could absorb before catastrophic failure during the normal -corrosion process proved to be far



(a)



(b)



(c)

Figure 29. Corrosion ingress for undamaged coating system 4 C-rings (a, left) near-notch side blistering at 4 cycles GM 9540P, (a, right) near-notch rust staining of edge at 48 GM 9540P cycles, (b) side blistering (7 $\times$ magnification), and (c) rust staining near edge (7 $\times$ magnification).

less than that which was absorbed by the hydrogen-free specimens undergoing more severe corrosion. This was just the opposite of what was expected but, in hindsight, is understandable since the Cd-plated specimens were significantly damaged. There comes a time after the plating process when it becomes difficult or impossible to relieve all of the hydrogen out of the specimens. Thus, within the Cd-plating specification SAE AMS QQ-P-416 there is a maximum requirement for a 4-hr dwell after plating, before hydrogen relief.

Despite these vast differences, Cd plating, even at its worst, is still significantly better than no plating and implies that, for the purposes of general-corrosion, a carelessly applied electroplated-Cd coating is still quite effective within a general-corrosion application. It must be stressed that the purpose of electroplated Cd is not just limited to general corrosion of low-strength alloys. For higher-strength steels at hardness levels of 50 HRC and above used in flight-critical components, the Cd plating must not only prevent general corrosive attack but also must do it in a manner that limits the kinetics of consumption of the sacrificial Cd. These conditions must be

met in order to limit the amount of hydrogen available to diffuse into the steel and lead to possible failure. Any failure to mitigate retained hydrogen from the plating operations can lead to catastrophic failures and, thus, heightened scrutiny must be given to post-plate rinsing, baking, and chromate sealing to prevent in situ hydrogen from electroplating operations from remaining within the substrate. Many process controls are built in as a precautionary part of the electroplating operation and serve to increase the quality and robustness of all forms of Cd plating.

It is apparent that unless flight-critical aviation performance standards are lowered, a sacrificial coating is necessary for future systems and components to perform at the level that is currently enjoyed. Finding a sacrificial coating system that can assume the role of electroplated Cd may prove to be very difficult. Many different Cd-alternative efforts are concurrently underway within the U.S. Department of Defense but until such time as a worthy substitute is found, there is no current workaround, even with current improvements in primer technologies. In the near future, reductions in hexavalent chromate may be possible via the primer component, through carefully considered use of MIL-PRF-23377 class N qualified primers.

5. Conclusions

- Electroplated Cd cannot be eliminated without detrimentally affecting corrosion resistance.
- Substituting MIL-PRF-23377 class C chromated primer with MIL-PRF-23377 class N qualified nonchromate primers may be possible when Cd plating is retained, as was observed in general- and crevice-corrosion conditions.
- Throwing power is overwhelmingly a function of a sacrificial coating like Cd, evidenced when all 120 panels without Cd failed before the end of the first corrosion cycle. No differences or trends could be established for any of the 120 panels without Cd plating, whether or not a chromate or nonchromate primer was used.
- The presence of topcoat hindered the corrosion performance of chromate-inhibited epoxy primer during the evaluation of throwing power. Chromate-inhibited epoxy primer may be beneficial for a sacrificial Cd coating's throwing-power effectiveness but only when exposed without a topcoat (or, perhaps, in certain situations where large portions of the topcoat is significantly damaged or degraded).
- Wide variations in throwing-power performance (ranging from 8 to 395 cycles of GM 9540P among the Cd-plated specimens) indicate throwing-power performance is a combination of Cd-plating thickness and uniformity and width of the organic coating-system gap.

- Early throwing-power failures of 20 cycles or less were likely caused by defective Cd plating.
- Cd plating, even when done poorly, is still much better than no Cd plating (eight cycles vs. less than one cycle of GM 9540P in throwing power).
- For throwing-power panels that lasted beyond 200 GM 9540P cycles, lifting and peeling of the organic coating system extended the cycles to red rust for the panels by exposing fresh areas of Cd.
- For smooth-profiled surfaces, nonchromated MIL-PRF-23377 class N adheres better than chromated MIL-PRF-23377 class C.
- Nonchromated MIL-PRF-23377 class N has better flexibility than chromated MIL-PRF-23377 class C.
- Abrasive blasting is recommended to maximize MIL-PRC-23377 class C coating adhesion to steels in low-risk applications where CD plating is not used.
- Applying MIL-PRF-23377 primers to abrasive-blasted steel surfaces within 4 hr of the blast step is acceptable in depot situations, when below 50% relative humidity is maintained and the environment remains free of particulate debris.

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