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Subtask 024: Testing Cadmium Alternatives for High Strength Steel: Phases II and III Testing

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Testing Cadmium Alternatives for High Strength Steel Phase II Joint Test Report

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Submitted by: CTC Concurrent Technologies Corporation

Mr. Thomas Naguy, AFRL
Program Manager
thomas.naguy@wpafb.af.mil

Mr. Warren Assink, AFMC
Quality Assurance Personnel
warren.assink@wpafb.af.mil

Ms. Beth Campbell, AFRL
Program Controller
elizabeth.campbell@wpafb.af.mil

Ms. Tamera Froehlich
Contract Manager
froehlit@ctc.com

Mr. Phillip O'Saben
Contract Administrator
osabenp@ctc.com

Mr. Clayton Drees
Subtask Manager
dreesc@ctc.com

TABLE OF CONTENTS

LIST OF TABLES

LIST OF FIGURES

LIST OF APPENDICES

EXECUTIVE SUMMARY

Cadmium (Cd) electroplating is widely used by the United States Air Force (USAF) and Department of Defense (DoD) to coat various metal substrates in weapons systems due to Cd's exceptional performance characteristics, low cost of application, and versatility of use. However, there are significant environmental, health, and safety issues associated with its use. Specifically, Cd is known to be a carcinogen, a toxic heavy metal, and, when used in electroplating, has an associated hazard related to the cyanide chemicals in the plating bath. Ion vapor deposited aluminum (IVD-Al) is one suitable Cd replacement for many applications, but it does not provide the lubricity of Cd, nor does it always provide sufficient corrosion protection due to coating porosity. Therefore, other alternatives need to be identified and validated as a replacement for Cd for these applications. In order to evaluate other potential alternatives to Cd electroplating, a Joint Services effort was initiated to develop the "High-Strength Steel Joint Test Protocol (JTP) for Validation of Alternatives to Low Hydrogen Embrittlement Cadmium for High-Strength Steel Landing Gear and Component Applications." The purpose of the JTP was to design and outline a single suite of performance requirements and test methods that could be used to fully assess the fundamental capabilities of alternative Cd plating processes in accordance with DoD-wide requirements and acceptance criteria. The effort was divided into three phases of testing. This test report discusses the results of the Phase II evaluations.

Phase II testing was conducted for both primary coatings and repair coatings. Primary coatings evaluated in this effort included sputtered aluminum (Al), electroplated Al, and Low Hydrogen Embrittlement (LHE) Zn-Ni (IZ-C17 process), which were compared against control coatings LHE Cd and IVD-Al. Testing was performed by Westmoreland Research Testing Lab (WMTR), Army Research Laboratory (Aberdeen Proving Ground, MD), Concurrent Technologies Corporation (Johnstown, PA) and Naval Air Systems Command (NAVAIR) Patuxent River, MD. Repair coatings that were evaluated included brush plated Zn-Ni, brush Sn-Zn, and spray/brush-applied SermeTel 249/273. The control coating for repair was brush Cd. In general, results showed that the Al based coatings performed well in bend adhesion, paint adhesion, and chemical strippability, which included embrittlement testing and bend adhesion both prior to and after coating rework by the vendor. These coatings also performed well in lubricity testing. Sputtered Al showed the greatest overall bend adhesion when considering the other two substrates in this study (titanium and corrosion resistant steel). LHE Zn-Ni showed inconsistent elevated temperature wet tape adhesion characteristics with MIL-PRF-85582 Class C1 primer, but met the JTP requirement of one day ambient immersion. Electroplated Al coating performed best in embrittlement and re-embrittlement quality control testing as previously observed in Phase I. Sulfur dioxide salt fog tests results indicated that sputtered and plated Al coatings both outperformed IVD-Al in bare and painted conditions. LHE Zn-Ni performed similar to Cd in the painted condition, whereas in the bare condition it performed better than Cd, although not as good as the Al coatings.

ACRONYMS, ABBREVIATIONS, AND NOMENCLATURE

°C	Degrees Celsius
°F	Degrees Fahrenheit
$\mu g/m^3$	Microgram(s) per cubic meter
AFMC	Air Force Materiel Command
AFRL	Air Force Research Laboratory
Al	Aluminum
Al-Ni-Br	Aluminum-Nickel-Bronze
ARL	Army Research Laboratory
Avg	Average
Cd	Cadmium
CD	Compact Disc
CTC	Concurrent Technologies Corporation
CuBe	Copper beryllium
DoD	Department of Defense
ESTCP	Environmental Security Technology Certification Program
g	Gram(s)
HAFB	Hill Air Force Base
HE	Hydrogen Embrittlement
HRE	Hydrogen Re-Embrittlement
HSS	High Strength Steel
IAW	In Accordance With
in-lb	Inch pound(s)
ISL	Incremental Step Load
IVD-Al	Ion Vapor Deposited Aluminum
JCAT	Joint Cadmium Alternatives Team
JTP	Joint Test Protocol
JTR	Joint Test Report
ksi	Kilopound(s) per square inch
kW	Kilowatt(s)
LHE	Low Hydrogen Embrittlement
mil	0.001 inch
MIL-STD	Military Standard
NA	Not Applicable
NaCl	Sodium chloride
NAVAIR	Naval Air Systems Command
NFS	Notch Fracture Strength
Ni	Nickel
NSS	Neutral Salt Spray
OEM	Original Equipment Manufacturer
OO-ALC	Ogden Air Logistics Center
OSHA	Occupational Safety and Health Administration
PEL	Permissible Exposure Limit
SCC	Stress Corrosion Cracking
SEM	Scanning Electron Microscopy
SLT	Sustained Load Test
SM	Subtask Manager
Sn-Zn	Tin-zinc
SO_2	Sulfur dioxide
Ti-6Al-4V	Titanium-6%aluminum-4%vanadium

USAF	United States Air Force
UTS	Ultimate Tensile Strength
WMTR	Westmoreland Mechanical Testing and Research
wt%	Weight percentage
Zn	Zinc
Zn-Ni	Zinc-nickel

1.0 INTRODUCTION

1.1 Background

Cadmium (Cd) electroplating is widely used by the United States Air Force (USAF) and Department of Defense (DoD) to coat various metal substrates in weapons systems due to Cd's exceptional performance characteristics, such as sacrificial corrosion protection, lubricity, galling prevention, and useful torque-tension properties. Additionally, Cd electroplating is a relatively simple and cost-effective process to operate and maintain. Cd is also used as a protective (sacrificial) metal coating under painted surfaces. Unfortunately, Cd is a toxic heavy metal and a carcinogen. Cd plating is easily removed during depainting operations, resulting in costly disposal of large volumes of waste and concerns with Cd dust generation (as is the case with mechanical removal). Furthermore, when used in electroplating, Cd has an associated hazard related to the cyanide chemicals in the plating bath. Therefore, despite Cd's performance characteristics, low processing cost, and versatility, the environmental, health, and safety issues associated with its use are significant, and various current and forthcoming regulations have been imposed on its use and disposal. For example, the Occupational Safety and Health Administration (OSHA) has imposed a permissible exposure limit (PEL) to Cd dust[1], leading to increased compliance costs. In response, the DoD has initiated efforts to search for alternative coatings and coating processes to Cd plating.

Ion vapor deposited aluminum (IVD-Al) is one suitable Cd replacement for many applications, but it does not provide the lubricity of Cd, nor does it always provide sufficient corrosion protection due to coating porosity. Additional post processing steps are often required, such as labor-intensive glass bead peening, which further densifies the coating to improve corrosion protection and adhesion to the substrate material. Even though aluminum (Al) is not considered a detrimental material, IVD-Al is a dimensionally-limited process. At the present time, it cannot treat components that have deep recesses or blind holes, as are common to many landing gear components. Additionally, some components, such as C-5 main landing gear, are too large to be accommodated by the IVD-Al chambers. These limitations demonstrate that IVD-Al does not completely eliminate the use of Cd.

1.2 Joint Test Protocol Development

A number of alternatives have been proposed to replace Cd electroplating and IVD-Al. However, performance testing is needed to verify whether the alternative(s) can impart the required characteristics for weapons systems applications. To address this need, the Air Force Research Laboratory (AFRL) contracted Concurrent Technologies Corporation (*CTC*), in cooperation with The Boeing Company (Boeing), to develop the "High-Strength Steel Joint Test Protocol (JTP) for Validation of Alternatives to Low Hydrogen Embrittlement Cadmium for High-Strength Steel Landing Gear and Component Applications." The purpose of the JTP was to design and outline a single suite of performance requirements and test methods that could be used to fully assess the fundamental capabilities of alternative Cd plating processes in accordance with DoD-wide requirements and acceptance criteria. To support JTP development and ensure accuracy and effectiveness, *CTC* and Boeing worked with the Joint Services (Air Force,

[1]The OSHA PEL established for cadmium dust is five micrograms per cubic meter of air (5 $\mu g/m^3$), calculated as an eight-hour, time-weighted, average exposure.

Army, and Navy) and original equipment manufacturers (OEMs) to determine the necessary test information (i.e., common and Service-specific needs). The JTP provided a means of confirming vendor performance claims, allowed for Joint Service analyses, and outlined the requirements for coating developers to qualify new materials and processes to replace Cd.

Before the JTP was developed to its present state, an initial test protocol was prepared to delineate and describe the performance requirements for coatings that are applied to high strength structural alloy steel [>200 Kilopound(s) per square inch (ksi)] landing gear components, as processed by Hill Air Force Base (HAFB)/Ogden Air Logistics Center (OO-ALC). This initial test protocol also was a collaborative effort between *CTC* and Boeing. Specifically, *CTC* assisted Boeing in the establishment of the team, which included representatives from Boeing-St. Louis, Boeing-Mesa, Air Force Materiel Command (AFMC), AFRL, HAFB/OO-ALC, and *CTC*.

Boeing designed an outline for the initial test protocol based on the performance requirements listed within SAE AMS-QQ-P-416 and Military Standard (MIL-STD)-870B. These items were combined with input that had been gathered from the team and direct feedback from HAFB/OO-ALC personnel that focused on additional requirements not specifically called out within the specifications and current Cd plating practices. Once completed, the outline was presented to the HAFB/OO-ALC landing gear experts. Upon its presentation, the initial test protocol outline was reviewed, and it was determined that a distinction needed to be made between performance requirements and additional testing (i.e., testing based on commercial practice or inputs from individuals experienced in dealing with high-strength steel applications).

Per AFRL direction, the test protocol was expanded to cover the Joint services, and therefore, required input from the Army and Navy, in addition to, OEMs. To properly acknowledge the joint focus of the test protocol, the document was officially renamed as the JTP. Further, a formal team consisting of representatives from the Joint Cadmium Alternatives Team (JCAT) as well as new representatives from all of the DoD services, the OEM community, and *CTC* was formed. This reformed team kept the name JCAT, and is generally managed by Navy personnel from Naval Air Systems Command (NAVAIR), Patuxent River.

1.3 Phase I Testing

In addition to the JTP, the USAF and *CTC* developed the Environmental Security Technology Certification Program (ESTCP) "High Strength Steel (HSS) Cadmium Alternative Test Plan". This test plan, located in Appendix A of the attached Compact Disc (CD), organized the required testing into sequential phases, and described the logistics, roles, and responsibilities that were involved with the execution of the JTP. Phase I test activities were completed under the supervision of NAVAIR for both primary and repair coatings identified as potential replacements for Cd and IVD-Al.

Phase I testing consisted of hydrogen embrittlement (HE), hydrogen re-embrittlement (HRE), and stress-corrosion cracking (SCC) analysis of the selected coatings to ensure that potential replacement processes had no detrimental effect on the steel substrates. Likewise, bend adhesion testing was performed for each process to determine whether the deposited coating was capable of adequately adhering to the substrate materials.

Data generated during Phase I testing[2] was reviewed with the JCAT. The team down-selected the coatings and processes for testing and evaluation in Phase II. An electroplated Al coating outperformed all other primary coatings, including Cd, in Phase I evaluations, while the tin-zinc (Sn-Zn) primary coating and an acidic zinc-nickel (Zn-Ni) coating were dropped from the study due to poor performance. Results for the repair coatings were mixed, with brush plated Sn-Zn performing the best, though there was considerable interest in the other repair coatings, and all three were continued to Phase II testing.

1.4 Phase II Joint Test Report Overview

This Joint Test Report (JTR) includes the data interpretation and test results for the testing conducted in support of the JTP for both primary and repair coatings identified as potential replacements for low hydrogen embrittlement (LHE)-Cd and IVD-Al. Likewise, deviations to the test methods outlined within the JTP, a discussion of the acceptance criteria governing each test, and an evaluation of the performance of each coating specific to the individual tests and the overall JTP are included in this JTR. All Phase II testing and reporting was coordinated by *CTC* and was conducted through a collaborative effort involving *CTC*, NAVAIR, the Army Research Laboratory (ARL), and Westmoreland Mechanical Testing and Research (WMTR) personnel and facilities, with guidance and support from the USAF Subtask Manager (SM). Details related to the descriptions, rationale, and methodologies for each of the tests conducted within Phase II of this effort are located within the JTP. Section 2 of this JTR contains information specific to the coatings evaluated during Phase II along with the testing conducted and the location where each test was conducted. Section 3 of this JTR contains all of the results and discussions of tests conducted during Phase II. All raw data, results, and photographs obtained during this effort are located in Appendix B, on the attached CD.

2.0 TECHNICAL APPROACH

2.1 Coating Information

Based on the results from Phase I testing, three alternative coatings were selected to undergo a suite of performance tests for further evaluation of the ability of these coatings to meet the requirements of a Cd replacement process. The primary coatings selected were LHE Zn-Ni (Dipsol IZ-C17), electroplated Al (AlumiPlate), and sputtered Al. LHE-Cd and IVD-Al coated panels and components were selected as baselines for comparison during the evaluation of the Phase II data.

While identifying a primary coating capable of replacing Cd and IVD-Al is one goal of the current project, identifying a coating capable of replacing brush plated Cd for touch up and/or repair applications is essential for the total systems approach to the replacement of Cd. The selected repair coatings were a brush plated Zn-Ni, a brush plated Sn-Zn, and a sprayed Al-ceramic (SermeTel). Brush plated LHE Cd was utilized as the baseline repair coating. While repair coatings are typically used to deposit a protective layer on areas where the primary coating has been damaged or compromised, Phase II

[2] Beck, Erin N., "Joint Test Report for Execution of Phase I of High Strength Steel Joint Test Protocol for Validation of Alternatives to Low Hydrogen Embrittlement Cadmium for High Strength Steel Landing Gear and Component Application – of July 2003", Naval Air Warfare Center Aircraft Division Technical Report NAWCADPAX/TR-2006/164, 10 January 2007.

testing focused on evaluating the performance of repair coatings that have been deposited on bare substrates, in accordance with the JTP.

Table 1 lists the alternatives selected by the JCAT that were evaluated during Phase II. In addition, the table lists the basic coating information, which includes the name of the vendor or DoD facility that applied the coating(s), the specification followed or product utilized for application, target thickness, and other relevant information. It is important to note that all coatings were applied according to the vendor recommended specifications if a military standard did not exist. All specimens received a subsequent hexavalent chromium-based conversion coating seal. All vendors/coaters were asked to target a 0.5 thousandths of an inch (mil) coating thickness. Coating thickness was measured on 4 inch x 6 inch steel flat panels using an Elcometer 456 Coating Thickness Gauge with a ferrous F1 probe. In addition, detailed panel processing information is located in Appendix A on the attached CD.

Table 1. Cadmium Alternatives Subjected to Phase II Testing

Coating	Coater	Coating Process Specification or Product	Post-Plate Hydrogen Relief Bake	Targeted Coating Thickness	Other
LHE Cd	HAFB	MIL-STD-870	Yes	0.6 mil	
IVD-Al	HAFB	MIL-DTL-83488 Class 2	No	minimum 0.5 mil	Unpeened
IVD-Al	Cametoid Technologies or Navy Fleet Readiness Center Southwest	MIL-DTL-83488 Class 2	No	minimum 0.5 mil	Unpeened
Magnetron Sputtered Al	Marshall Laboratories	Marshall Lab Process	No	0.5 mil	Conversion coating applied at *CTC* (panels) and NAVAIR Patuxent River (1a.1 bars)
Electroplated Al	AlumiPlate, Incorporated	MIL-DTL-83488 Class 2	No	0.9 mil	
Zinc-Nickel (Dipsol LHE)	Dipsol of America	Boeing/Dipsol Procedure IZ-C17	Yes	0.3-0.5 mil	84-88% Zinc (Zn) 13-17% Nickel (Ni)
Brush Cd	Boeing St. Louis	SIFCO 2023	No	0.5 mil	
Brush Zinc-Nickel	Boeing St. Louis	SIFCO 4018	No	0.5 mil	
Brush Tin-Zinc	Boeing St. Louis	LDC 5030	No	0.5 mil	
SermeTel 249/273	Boeing St. Louis	Sermetech Engineering Bulletin 249	No	0.5 mil	Al and Zn

2.2 Testing Approach

Per JCAT decision and AFRL direction, *CTC* coordinated all of the testing activities associated with Phase II of the JTP. The AFRL, NAVAIR, ARL, and the ESTCP provided test support. Because the goal of the JCAT is to actively involve DoD facilities with the execution of the JTP, the primary contacts for processing and testing are the DoD facilities.

As previously mentioned, the testing contained within the JTP was outlined as a three-phased approach. Phase I, which has been completed and is not a part of this JTR, consisted of HE and HRE testing, including the In-Service Hydrogen Re-Embrittlement/Stress Corrosion Cracking C-Ring Test (an Army recommended test). The results from Phase I were compiled and reviewed by the JCAT, and a core team determined which alternatives were to be evaluated in Phase II. Phase II consisted of the majority of the JTP tests, with the addition of the Navy requested sulfur dioxide (SO_2) salt fog testing. The Phase II results compiled within this JTR will be reviewed by the JCAT. Finally, Phase III work planned to conduct fatigue testing, which is the most expensive test outlined in the JTP. However, a decision was made by the AFRL to cancel Phase III evaluation activities under Subtask 024.

Execution of Phase II activities detailed in this JTR was achieved through a collaborative effort. Substrate materials for coating were purchased by NAVAIR and *CTC*. *CTC* was responsible for coordinating sample coating activities at the various vendor and DoD locations. Sample analyses were performed by NAVAIR, ARL, *CTC*, and the *CTC* subcontractor, WMTR. Test results and analyses were forwarded to *CTC* for compilation into this JTR at the completion of testing. Also, as part of the collaborative effort, *CTC* subcontracted Boeing to ensure that Boeing's plating expertise and involvement were maintained throughout JTP execution.

Table 2 identifies the testing facility for each performance test conducted during Phase II. It is important to note that quality assurance tests were included to verify that the plating processes were comparable in Phases I and II.

Table 2. JTP Phase II Testing Facility

Test Category	Test	Testing Facility
General Properties (primary coatings)	Appearance	*CTC*
	Throwing power and alloy composition uniformity	*CTC*
	Stripability	NAVAIR
	Galvanic potential	Not tested
Adhesion (primary coatings)	Bend adhesion	NAVAIR
	Paint adhesion	NAVAIR
Corrosion (primary coatings)	Unscribed neutral salt spray (NSS) (bare)	ARL
	Scribed NSS (bare)	ARL
	Galvanic corrosion resistance	ARL
	Fluid corrosion resistance	ARL
	Scribed w/ primer & topcoat	NAVAIR (paint)/ARL (test)
	SO_2 Salt Fog *	NAVAIR

Table 2. JTP Phase II Testing Facility (Continued)

Test Category	Test	Testing Facility
Lubricity (primary coatings)	Run-on/break-away torque	WMTR
	Torque-tension	WMTR
Reparability (repair coatings)	Appearance	*CTC*
	Bend adhesion	ARL
	Thickness	*CTC*
	Paint adhesion	Not tested
	Unscribed corrosion resistance	ARL
	Scribed corrosion resistance	ARL
Quality Assurance (primary and repair coatings)	Hydrogen embrittlement – notched bar	NAVAIR

* Testing agreed upon by the JCAT.

3.0 TEST RESULTS AND DISCUSSION

This section outlines the test methods utilized for analysis from the JTP. Any deviations to the test methods are listed in each section, along with the test results, and a discussion of the results.

3.1 General Properties

3.1.1 Appearance

Test Description

Specimens were prepared with the appropriate coating systems. Once returned from the respective vendors, the coatings were evaluated by observing color, texture, and uniformity of appearance by unaided visual inspection. Any coating defects that were observed were recorded and reported. The coating was compared to the Cd plating specifications, MIL-STD-870B and Fed-Std-QQ-P-416F (*Plating, Cadmium, [Electrodeposited]*, issued October 1, 1991), which is now AMSQQP416 (*Plating, Cadmium, [Electrodeposited]*, issued September 2, 2009), where the coating was to be smooth, adherent, uniform in appearance, and free from blisters, pits, nodules, burning and other defects.

Test Methodology

Parameters	Unaided visual inspection
Type/Number of Specimens	Three specimens, 4130 steel
Experimental Control Specimens	None
Acceptance Criteria	Coating must be continuous, smooth, adherent, uniform in appearance, free from blisters, pits, nodules, burning, contaminants, excessive powder, and other apparent defects.
Reference Document	MIL-STD-870B, AMSQQP416

<u>Test Results</u>

In general, the appearance of all primary coatings was determined to be acceptable, and all candidate coatings, as well as baseline Cd and IVD-Al coatings, were given a "pass" rating for appearance. Results documented from the visual examination of the primary coatings are presented in Table 3.

Table 3. Appearance of Primary Coatings

Coating	Appearance Results
LHE Cd (Baseline) - HAFB	Coating is continuous but not uniform, showing some edge effect; coating is smooth, adherent, and free from blisters, pits, excessive powder, and contamination
IVD-Al (Baseline) - HAFB	Coating is continuous, uniform, smooth, adherent, and free from blisters, pits, excessive powder, and contamination
IVD-Al (Baseline) - Commercial Vendor	Coating is continuous, uniform, smooth, adherent, and free from blisters, pits, excessive powder, and contamination
LHE Zn-Ni - Commercial Vendor	Coating is continuous but not uniform, also containing a few spots of possible contamination; otherwise, the coating is smooth, adherent, and free from pits, blisters, and excessive powder
Electroplated Al - Commercial Vendor	Coating is continuous, uniform, smooth, adherent, and free from blisters, pits, excessive powder, and contamination
Sputtered Al - Commercial Vendor	Coating is continuous, uniform, smooth, adherent, and free from blisters, pits, excessive powder, and contamination

3.1.2 Throwing Power and Alloy Composition Uniformity

The ability of a process to coat complex shapes including the inside of blind holes while maintaining a consistent composition is critical to successful application on complex components. This test used a fixture to create a cavity on a flat substrate surface. After coating, the substrate was removed and composition of the coating inside the cavity was determined. Composition of the coating was determined by scanning electron microscopy (SEM) methods. Thickness testing was also planned as part of throwing power testing; however, thickness testing was not completed during this effort.

<u>Test Description</u>

Fixture: Fixtures were constructed to the dimensions shown in Figure 1. The fixtures were made from Teflon, with the exception of the fixtures for electroplated Al. These fixtures were made by the vendor, to the same dimensions, to ensure that the fixture would not react with the electroplating bath. In both cases, the fixtures were constructed from a section of pipe with one capped end. Slots were cut into the interior to allow a 3 inch x 5 inch test panel to slide snuggly into the pipe. A threaded open cap closed off the fixture.

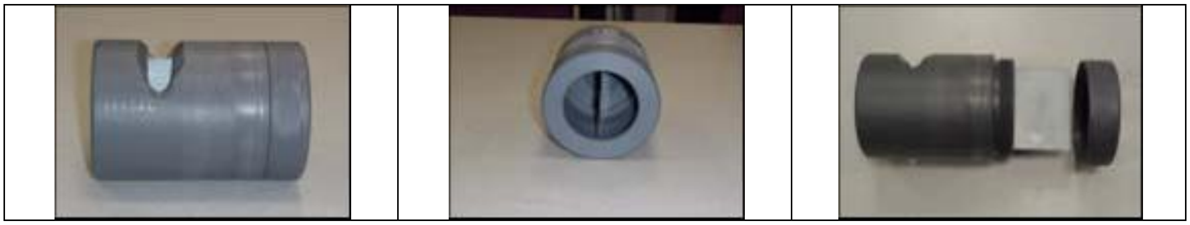


Figure 1. Fixture for Throwing Power and Alloy Composition Test

Test panels were placed in the fixtures with caps and any electrical connections installed. Coatings were applied by the respective manufacturer, with the fixture aligned at different orientations, where possible.

Coating Composition: SEM was utilized to determine the composition of the coatings. Standards were utilized to calibrate the equipment. Oxygen and iron were measured for all samples in addition to the major alloy components for the coating systems. Composition readings were taken at 0.5 inch increments over the length of the panel (5 inches), at the center of the width (3 inches), resulting in nine readings.

<u>Rationale</u>

The composition and thickness of coatings is critical to their performance. Consistent composition is required of all coatings. Understanding how far coatings will "throw" into holes and cavities will determine the applicability of a process to potential hardware.

<u>Test Methodology</u>

Parameters	Measured on at least three (3) locations on each test specimen surface, separated by 1 inch. Measured spots shall not overlap. The total measured area shall be greater than 1 square inch.
Type/Number of Specimens	Three specimens, 4130 steel with fixtures at different orientations to the coating "chamber" geometry.
Experimental Control Specimens	LHE-Cd (MIL-STD-870B or equivalent). Three (3) LS1 with fixtures at different orientations to the coating "chamber" geometry.
Acceptance Criteria	Composition stays within the process specification requirements.
Reference Document	MIL-STD-870B, AMSQQP416

<u>Test Results</u>

Coating composition test results are listed in Tables 4 through 12. The first three tables list the results for LHE-Cd, the second set of three tables (7 – 9) contains the results for electroplated Al and the third set of three tables (10 – 12) lists the results for LHE Zn-Ni. Throwing power samples could not be prepared for IVD-Al and sputtered Al, due to the nature of their coating processes. Each table contains weight percent values of the elements in the coatings, with Reading #1 corresponding to the end of the sample closest to the open end of the fixture. Note that the composition measurements taken at each location were normalized to total 100 weight percent (wt %).

Table 4. LHE-Cadmium Panel Composition Results – Orientation #1

Reading #	Wt % Oxygen	Wt% Cadmium	Wt% Chromium
1	37.6%	46.2%	16.2%
2	34.6%	50.4%	15.0%
3	33.5%	51.5%	15.0%
4	32.3%	52.9%	14.8%
5	35.0%	51.1%	13.9%
6	33.6%	52.8%	13.7%
7	33.6%	53.1%	13.3%
8	33.7%	52.9%	13.4%
9	33.7%	51.2%	15.1%

Table 5. LHE-Cadmium Panel Composition Results – Orientation #2

Reading #	Wt % Oxygen	Wt% Cadmium	Wt% Chromium
1	28.7%	60.5%	10.8%
2	28.0%	62.1%	9.9%
3	24.5%	65.4%	10.1%
4	26.1%	62.9%	11.0%
5	27.2%	61.1%	11.7%
6	27.9%	59.9%	12.2%
7	29.0%	58.6%	12.5%
8	29.2%	58.4%	12.5%
9	30.6%	56.8%	12.7%

Table 6. LHE-Cadmium Panel Composition Results – Orientation #3

Reading #	Wt % Oxygen	Wt% Cadmium	Wt% Chromium	Wt% Iron
1	26.7%	63.4%	9.8%	0%
2	25.5%	64.1%	10.4%	0%
3	28.5%	61.0%	10.5%	0%
4	29.4%	59.5%	11.1%	0%
5	28.0%	56.5%	12.2%	3.3%
6	28.0%	59.1%	12.0%	1.0%
7	28.4%	56.6%	13.5%	1.5%
8	30.9%	55.1%	13.1%	1.0%
9	32.5%	53.3%	13.2%	1.0%

The orientations of the fixtures in the Cd plating bath were not provided by HAFB, so each table above is simply labeled as Orientation #1, #2, and #3. The ratios of oxygen to Cd to chromium in the first orientation showed higher concentrations of oxygen and chromium when compared to the other two orientations. Also, the concentration of Cd varied from approximately 46% to 53%, which then dropped to 51% at the opposite edge of the panel due to potential insulation from the fixture. This same effect of edge insulation was also visible in the results for the second orientation. The third orientation showed the largest variance in Cd concentration over the panel, with the side of the panel that had the lower Cd concentration also displaying detectable iron concentration. This would indicate incomplete coverage of the coating over the steel substrate.

Table 7. Electroplated Al Panel Composition Results – Open End in Horizontal Position

Reading #	Wt% Oxygen	Wt% Aluminum	Wt% Chromium	Wt% Iron	Wt% Nickel
1	11.8%	77.6%	5.2%	2.5%	3.0%
2	10.8%	78.7%	4.7%	2.4%	3.5%
3	10.6%	74.9%	4.8%	2.9%	6.8%
4	9.1%	72.5%	4.4%	3.2%	10.8%
5	11.7%	74.9%	5.0%	3.2%	5.2%
6	12.3%	76.8%	5.2%	3.0%	2.9%
7	12.0%	76.6%	5.6%	3.2%	2.6%
8	11.8%	76.6%	5.7%	3.0%	2.9%
9	10.8%	77.4%	4.3%	3.0%	4.5%

Table 8. Electroplated Al Panel Composition Results – Open End Facing Downward

Reading #	Wt% Oxygen	Wt% Aluminum	Wt% Chromium	Wt% Iron	Wt% Nickel
1	13.3%	76.9%	4.7%	2.6%	2.5%
2	11.5%	78.7%	4.4%	2.7%	2.6%
3	10.2%	80.0%	3.7%	2.8%	3.3%
4	9.0%	79.8%	3.8%	3.2%	4.2%
5	8.7%	81.8%	3.1%	3.3%	3.2%
6	11.1%	80.0%	3.8%	3.1%	2.0%
7	9.3%	80.4%	3.9%	3.0%	3.5%
8	10.1%	78.8%	3.9%	2.8%	4.4%
9	10.8%	77.2%	2.9%	2.8%	6.3%

Table 9. Electroplated Al Panel Composition Results – Open End Facing Upward

Reading #	Wt% Oxygen	Wt% Aluminum	Wt% Chromium	Wt% Iron	Wt% Nickel
1	9.9%	80.5%	3.9%	3.0%	2.7%
2	11.0%	79.8%	3.9%	2.9%	2.4%
3	9.4%	79.7%	3.5%	2.7%	4.8%
4	8.2%	76.8%	2.8%	3.0%	9.2%
5	8.5%	74.6%	3.5%	3.8%	9.7%
6	8.3%	78.6%	3.0%	4.4%	5.7%
7	9.4%	77.3%	3.2%	3.2%	6.9%
8	7.9%	70.9%	2.7%	5.8%	12.7%
9	3.6%	54.4%	0.9%	4.1%	37.0%

The results for the electroplated Al panels varied based upon the orientation of the fixture. When the fixture was placed in the bath in the horizontal position, the concentration of Al decreased by up to 5% in the center of the panel, while the concentration of nickel increased. The concentrations of oxygen, chromium, and iron remained fairly consistent. For the second panel, which was oriented in the vertical position with the open end facing downward, the concentration of the elements across the panel was nearly uniform. However, there was an increase in Al concentration up to 5% at the center of the panel, an inverse of the results of the first panel. The third panel, having the open end facing upward, had a dramatic loss in Al concentration beginning at reading number 8 and extending to the end of the panel, losing almost 25% Al over this span. In addition, the nickel concentration increased by over 30% in this same measurement range. The vendor stated that they deposited the Al over a nickel strike bond layer, showing that the Al coating did not "throw" over the length of the panel, getting much thinner and potentially porous over the last 1.0 – 1.5 inches of the panel.

Table 10. LHE Zn-Ni Panel Composition Results – Open End Facing Node (Upward)

Reading #	Wt% Oxygen	Wt% Iron	Wt% Nickel	Wt% Zinc
1	1.6%	0.3%	15.9%	82.2%
2	1.5%	0.3%	16.1%	82.1%
3	1.9%	0.5%	15.7%	81.9%
4	1.9%	1.5%	15.6%	81.0%
5	2.1%	1.7%	15.3%	80.8%
6	1.9%	2.0%	14.1%	82.0%
7	2.0%	2.8%	14.3%	80.8%
8	2.0%	2.8%	14.0%	81.2%
9	1.5%	3.2%	13.6%	81.7%

Table 11. LHE Zn-Ni Panel Composition Results – Open End Away from Node (Downward)

Reading #	Wt% Oxygen	Wt% Iron	Wt% Nickel	Wt% Zinc
1	1.4%	0.2%	15.9%	82.5%
2	1.7%	0.3%	15.9%	82.1%
3	1.8%	0.3%	15.2%	82.7%
4	1.8%	0.8%	15.4%	82.1%
5	1.7%	1.0%	15.8%	81.6%
6	1.8%	1.3%	14.7%	82.1%
7	1.4%	1.5%	14.9%	82.2%
8	1.7%	1.7%	14.5%	82.1%
9	2.2%	2.2%	14.0%	81.6%

Table 12. LHE Zn-Ni Panel Composition Results – Open End in Horizontal Position

Reading #	Wt% Oxygen	Wt% Iron	Wt% Nickel	Wt% Zinc
1	1.7%	5.6%	11.2%	81.6%
2	2.0%	2.6%	11.9%	83.5%
3	2.2%	1.8%	13.3%	82.7%
4	2.4%	1.5%	14.3%	81.9%
5	2.2%	1.2%	15.1%	81.5%
6	2.3%	0.8%	15.4%	81.5%
7	2.1%	0.8%	15.0%	82.1%
8	2.4%	0.8%	15.2%	81.6%
9	2.6%	1.4%	15.5%	80.5%

For the LHE Zn-Ni panels, the concentration of zinc remained uniform, regardless of orientation. However, the change in the nickel concentration varied from 2 – 4%, depending on orientation. In the horizontal position, the nickel concentration increased across the panel, where the samples in the vertical positions exhibited decreases in nickel concentration.

3.1.3 Stripability of Coatings

Test Description

This test evaluated the ability to remove and replace the candidate coating from high strength substrates as would be required during rework procedures. As such, the candidate coatings were chemically stripped as recommended by the manufacturer or per accepted practice (e.g., caustic bath for Al removal). Bath chemistry information for the stripping solutions is as follows.

For the Al coatings (IVD-Al, sputtered Al, and electroplated Al), a caustic stripping bath was prepared utilizing the product, Specialty 835, procured from U.S. Specialty Color Corporation from Monroe, North Carolina. This product, a dry, sodium hydroxide chemistry, was mixed at 4 – 6 ounces/gallon of water. The test specimens were immersed at 130 degrees Fahrenheit (°F) ± 5°F.

A stripping process was provided by The Boeing Corporation for stripping the LHE Zn-Ni coating, utilizing ammonium nitrate. The targeted bath makeup was 16 ounces/gallon. Then, the pH of the bath was adjusted to 8.8 – 8.9 with dropwise additions of 10% sodium hydroxide solution. The strip time ranged from 45 minutes to over one hour at near room temperature conditions.

For each coating evaluated, eight notched tensile bars (ASTM F519, Type 1a.1) were used. Four of these were stripped and tested according to Section 3.6.1 of the JTP (Hydrogen Embrittlement) without performing an embrittlement relief bake prior to testing. Results presented in Table 14 reflect a maximum one hour delay between stripping and loading the specimen into the testing machine. Four additional bars were chemically stripped, shipped back to the coating vendor for coating reapplication, and subsequently tested for HE at NAVAIR, Patuxent River. During shipment, the bars were protected using the corrosion protective compound specified in ASTM F519 (MIL-PRF-16173, Type 2). Three bend adhesion panels were also re-coated by the vendor after stripping and retested per Section 3.2.1 (Bend Adhesion).

Rationale

This test is necessary to ensure that candidate coatings can be removed, replaced, and still meet the requirements for acceptable adhesion to the substrate and that the stripping method will not cause HE.

Test Methodology

Parameters	Specimens stripped of candidate coating, recoated, and tested for adhesion per Section 3.2.1 - Bend Adhesion and HE per Section 3.6.1 – Hydrogen Embrittlement
Type/Number of Specimens	Three 4130 steel bend specimens (1 inch x4 inches) and eight HE bars
Experimental Control Specimens	None
Acceptance Criteria	Candidate coating should be removed in two (2) hours or less using appropriate removal method. Substrate surface after coating removal must meet requirements of MIL-S-5002D prior to refinishing. Embrittlement by the stripping process is undesirable but acceptable. Re-applied coating must meet the acceptance criteria of Section 3.2.1 - Bend Adhesion and 3.6.1 HE
Reference Document	MIL-STD-870B, AMSQQP416

Test Results

Time Required for Stripping: Electroplated Al-coated bars, as conversion coated from the vendor, stripped in 10 – 15 minutes in the 130ºF solution. Sputtered Al bars were received from the coater without a conversion coating, and strip times were much shorter (2 – 3 minutes) in the same caustic bath. The LHE Zn-Ni coating strip times averaged more than an hour for what appeared visually to be full coating removal. The shortest strip time was approximately 45 minutes. When a new bath was prepared to strip the longer bend specimens (1 inch x 4 inches), pH was not adjusted far enough into the alkaline range and a lower strip rate occurred (2+ hours). This may have affected the results for bend recoat adhesion which was much lower than typical as-plated results.

Table 13 lists a summary of the results of the bend adhesion and HE testing for the alternative coatings. Testing of the baseline samples was not required for this testing method. In addition, sputtered Al test pieces were not returned from the vendor. Table 14 lists the HE results for the stripped and recoated specimens, followed by Table 15 with the bend adhesion results.

Table 13. Summary of Stripability Test Results

Coating	Change in Hydrogen Embrittlement	Change in Bend Adhesion
LHE Cd (Baseline) – Hill AFB	Not required	Not required
IVD-Al (Baseline) – Hill AFB	Not required	Not required
IVD-Al (Baseline) – Commercial vendor	Not required	Not required
LHE Zinc-Nickel	Pass – average of 88.5% fracture strength for 200 hours (3 of 4 specimens)	Fail – coating failure in 1-2 bend cycles
Electroplated Al	Pass – average of 93.6% fracture strength for 200 hours (4 of 4 specimens)	Pass – no coating failure before substrate rupture (12 cycles)
Sputtered Al	No data	No data

Table 14. HE Test Results for Chemically Stripped and Recoated Specimens

Coating	Sample #	Stripped Fracture Strength	Time to Failure*	Recoated Fracture Strength	Time to Failure*	Pass/Fail (Stripped/ Recoated)
Cd-plated	1	89.7 %	203	Not tested	Not tested	Pass
	2	89.3 %	203			
	3	88.4 %	203			
	4	90.1 %	203			
	Average (Avg.)	89.4 %				
LHE Zn-Ni	1	98.6 %	205	93.6 %	204	Pass/Pass
	2	99.4 %	205	75.0 %	13	
	3	98.0 %	205	94.3 %	204	
	4	95.2 %	204	91.1%	204	
	Avg.	97.8 %		88.5 %		
Electroplated Al	1	98.6 %	205	93.5 %	203	Pass/Pass
	2	100.0 %	205	94.2 %	203	
	3	95.6 %	205	92.0 %	203	
	4	93.6 %	204	94.5%	203	
	Avg.	97.0 %		93.6 %		
Sputtered Al	1	95.9 %	205	Not returned	Not returned	Pass
	2	98.7 %	205			
	3	95.5 %	204			
	4	97.1 %	205			
	Avg.	96.8 %				

* Test was ended after 200 hours, if failure does not occur.

Table 15. Bend Adhesion Results after Recoating

Coating	Sample #	Bend Cycles to Coating Failure	Bend Cycles to Substrate Fracture	Pass/Fail
LHE Zn-Ni	1	Not Applicable (NA)	1-2	Fail*
	2	NA	1-2	
	3	NA	1-2	
Electroplated Al	1	12	NA	Pass
	2	10	NA	
	3	12	NA	
Sputtered Al	1			Not returned
	2			
	3			

* Strip bath alkalinity was not properly adjusted for these specimens, which may have caused residual plating to interfere with the reworked plating adhesion (although grit blasting should have mitigated or eliminated this possibility).

The sputtered and electroplated Al coatings, as well as the LHE Zn-Ni coating, were able to be removed chemically from the high strength steel bars and still permit average fracture strengths of approximately 97% of the baseline notched fracture strength (NFS) for the lot of bars (without any baking step). Cd plated bars passed at an average strength of 89.4% NFS after stripping, which is slightly lower than the as-plated values determined in Phases I and II of this study (91.8% NFS and 93.7% NFS, respectively). Testing of the baseline coatings was not required for this testing method.

Four of the eight bars were stripped and sent back to the coating suppliers for re-coating. Reworked bars were not received back for the sputtered Al coating. The specimens re-coated with electroplated Al passed with average fracture strength of 93.6% for 4 bars. Of the specimens re-coated with LHE Zn-Ni, three performed well with an average of 93.0% NFS, while the fourth failed in the threads at 13 hours (75% NFS). Both coatings tested earned ‘Pass’ ratings according to the acceptance criteria.

As stated previously, the longer strip time required for the bend adhesion samples coated with LHE Zn-Ni may have affected the recoat adhesion properties of the coating, resulting in failures in bend adhesion.

3.1.4 Electrochemical Galvanic Potential

The electrochemical galvanic potential was not conducted under this effort.

3.2 Adhesion

3.2.1 Bend Adhesion

This test evaluated the ability of a coating to adhere to the substrate.

Test Description

The coatings were applied to the test specimens as recommended by the manufacturer. The specimens were clamped in a vise and then the projecting end was bent back and forth until rupture of the basis metal and/or coating occurred, in accordance with (IAW) ASTM B571-91 (*Standard Test Methods for Adhesion of Metallic Coatings*, issued February 22, 1991). The edges of the ruptured coatings were examined at four times magnification for peeling or flaking of the coating from the substrate.

Rationale

The acceptance criteria for this adhesion test match the requirements specified in MIL-STD-870B and AMS QQ-P-416 (*Plating, Cadmium, [Electrodeposited]*, issued July 2000). This test is necessary to qualify candidate coatings for acceptable adhesion to the substrate.

Test Methodology

Parameters	Specimen is bent back and forth through 180° until the coating and/or substrate ruptures
Type/Number of Specimens	3 specimens of each of the following substrate materials: 4130 steel, 17-4 PH stainless steel, 6Al-4V Titanium
Experimental Control Specimens	None required
Acceptance Criteria	No separation (flaking, peeling, or blistering) of the coating from the basis metal or from any under-plating at the rupture edge. Cracking is acceptable in the bend area if the coating cannot be peeled back with a sharp instrument.
Reference Document	MIL-STD-870B, AMSQQP416

Test Results

Table 16 lists the results of bend adhesion testing. Alternative coatings failed strictly on the Titanium-6%aluminum-4%vanadium (Ti-6Al-4V) substrate material. Following the table, Figures 2 and 3 contain example photographs and photographs of the failure modes.

Table 16. Bend Adhesion Test Results

Coating	Bend Adhesion Results		
	4130 steel substrate	**17-4 PH stainless substrate**	**Ti-6-4 substrate**
LHE Cd (Baseline) – Hill AFB	No data	Pass; 3 cycles to substrate fracture	Pass; no fracture
IVD-Al(Baseline) – Hill AFB	No data	No data	No data
LHE Zinc-Nickel	Pass – cracking of coating up to 3/8 inch; 16-18 cycles to substrate fracture	Pass – no cracking or defect; 3 cycles to substrate fracture	Fail – during 1st bend cycle; spalling beyond 3/8 inch

Table 16. Bend Adhesion Test Results (Continued)

Coating	Bend Adhesion Results		
	4130 steel substrate	**17-4 PH stainless substrate**	**Ti-6-4 substrate**
Electroplated Al	Pass – cracking of coating up to 1/8 inch; 16-18 cycles to substrate fracture	Pass - no cracking or defect; 3-4 cycles to substrate fracture	Fail – edge buckling to ½ inch; 6 cycles to substrate fracture
Sputtered Al	Pass – no cracking or defect; 13-14 cycles to substrate fracture	Pass - no cracking or defect; 3 cycles to substrate fracture	Pass - no cracking or defect; 3-6 cycles to substrate fracture

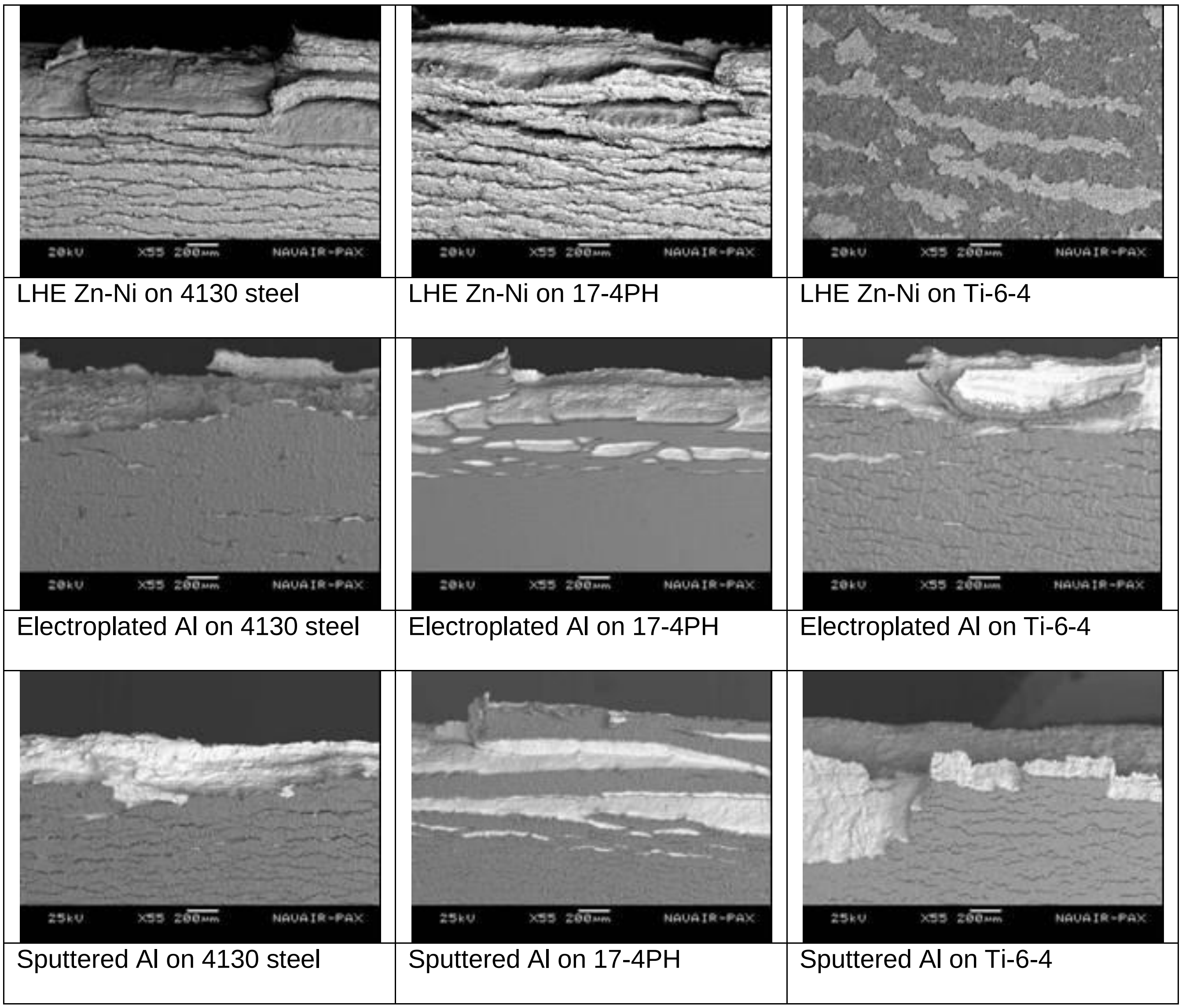
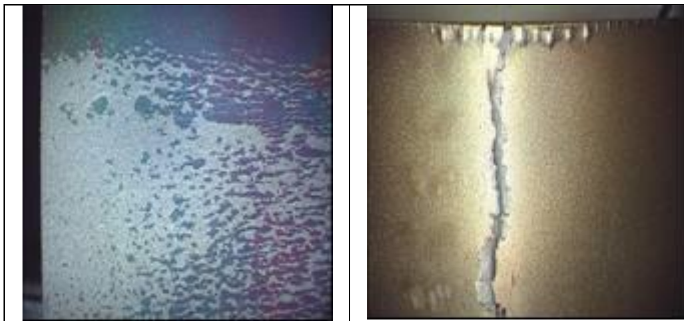


Figure 2. Low Magnification SEM Images of Bend Samples (55X)



Zinc-Nickel on Ti-6-4	Electroplated Al on Ti-6Al-4V

Figure 3. Photos of Failure Modes of Alternative Coatings (no magnification)

It is evident from Table 16 that the coatings tested generally displayed good adhesion to each substrate, though two failures were noted on titanium substrates. The plated and sputtered Al coatings performed well on most substrates as may be observed in the low magnification SEM images shown in Figure 2. Though cracks appeared in the Al coatings near the fracture surface due to the severe deformation, they were not able to be lifted with a sharp blade. On Ti-6Al-4V, however, the electroplated Al coating buckled and lifted along the sides of the bend sample up to 0.5 inch away from the fracture plane (Figure 3). These areas could be removed with a blade. According to the manufacturer, the electroplated Al had been deposited over a nickel strike bond layer. The sputtered Al coating performed well on titanium. The LHE Zn-Ni plating performed well on 17-4 PH stainless steel and 4130 steel, although the plating flaked off the Ti-6Al-4V substrate before one full 90 degree bend of the substrate. The coating dusted off in 0.02 – 0.04 inch flakes, and exhibited spalling beyond 0.375 inch from the fracture plane. It should be noted that titanium is a difficult substrate for activation and plating, and that the failures experienced on these substrates might be attributable to pretreatment rather than coating properties.

3.2.2 Wet Tape Paint Adhesion

This test assessed the general adequacy of paint adhesion to flat surfaces coated with the candidate coating. The test was conducted by applying and removing pressure-sensitive adhesive tape over scratches made through the paint.

<u>Test Description</u>

The wet tape adhesion test was performed IAW ASTM D3359 Method B (*Standard Test Methods for Measuring Adhesion by Tape Test*). Coatings were applied to the test specimens as recommended by the manufacturer. Waterborne epoxy primer, conforming to MIL-PRF-85582 Type I, Class C1 (*Primer Coatings: Epoxy, Waterborne*), was applied to one set of specimens and allowed to dry in air for 14 days prior to testing. A non-chromated waterborne epoxy primer, reference MIL-PRF-85582 Type I Class N (e.g., PRC Desoto/Spraylat EWAE118 A/B Type II, Class N or Akzo Nobel 10PW22-2/ECW119) was applied to a second set of panels. This primer was also dried in air for 14 days prior to testing. A solvent borne primer conforming to MIL-PRF-23377 Type I, Class C (*Primer Coatings: Epoxy, High Solids)* was applied to a third set of panels and dried in air for 14 days prior to testing. For each primer type, sets of three candidate and three control specimens were immersed in distilled water at each of three conditions: 24

hours at 23 degrees Celsius (°C), 96 hours at 49°C, and for 168 hours at 65°C. At the conclusion of exposure, the specimens were removed and wiped dry with a soft cloth. Within one minute of removal from the water, the coating was scribed through to the basis metal in a grid pattern IAW ASTM D3359 Method B. A one-inch wide strip of masking tape was immediately applied (average adhesion of 75-80 ounces/inch) and centered on the grid. The tape was pressed with a pencil eraser, or other appropriate tool, until the tape was firmly adhered to the coating. The tape was removed with one quick motion and the grid was examined for coating adhesion.

Rationale

This test, conducted at 23°C, is necessary to qualify candidate coatings for use on substrates that may be painted. Testing conducted at 49°C and 65°C is for informational purposes only and does not include acceptance criteria.

Test Methodology

Parameters	Immerse separate specimens in distilled water for 24 hours and 186 hours at 23°C, 96 hours at 49°C, and 168 hours at 65°C, respectively.
Type/Number of Specimens	Three 4130 specimens per primer per time/temperature combination
Experimental Control Specimens	Three 4130 specimens per primer per time/temperature combination
Acceptance Criteria	Adhesion not less than 4B as determined using the criteria in ASTM D3359 for specimens immersed for 24 hours at 23°C.
Reference Document	MIL-STD-870B, AMSQQP416

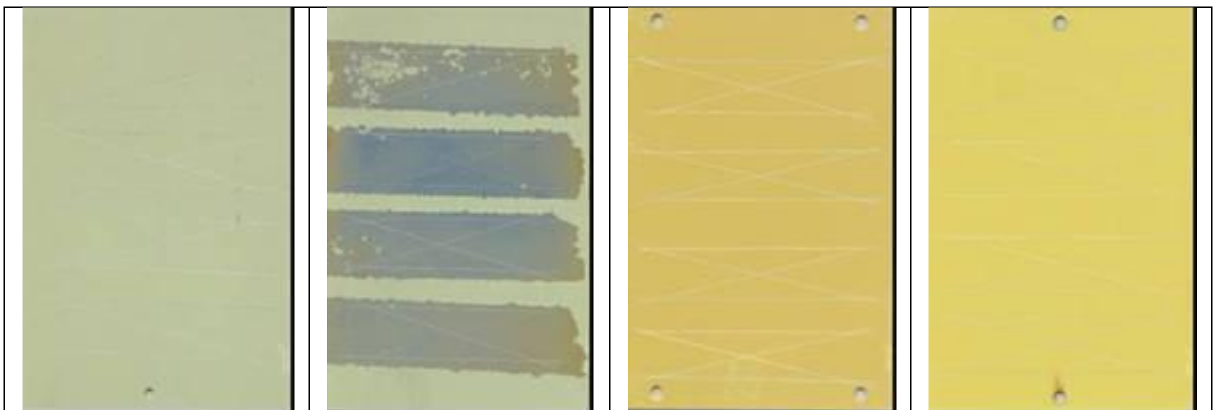
Test Results

Table 17 lists the results of the tape adhesion test for each coating system, at each time/temperature combination. The specific tape used for this testing was 3M #250 with a nominal adhesion value of 75-80 ounces/inch. Each coating was tested on triplicate 4 inch x 6 inch steel panels for each duration test, using four adhesion test sites per panel, yielding a total of twelve values which were averaged to provide the data for each entry below. Average primer thickness values were as follows: 23377 Class C2 (2.0 mil), 85582 Class C1 (0.9 mil), 85582 Class N (1.2 mil). Figure 4 contains representative photos of the tape adhesion panels.

Table 17. Averaged Wet Tape Adhesion Results

Primer application →	MIL-PRF-23377, Class C2			MIL-PRF-85582, Class C1			MIL-PRF-85582, Class N		
Test Duration →	1 day	4 days	7 days	1 day	4 days	7 days	1 day	4 days	7 days
Coating	Test Results (average of 12 measurements)								
Cd-plated (control)			5B[1]			5B[1]			5B[1]
IVD-Al (Hill AFB)[2]	5B	5B	5B	5B	5B	5B	5B	5B	5B
LHE Zinc-Nickel[3]	4.8B	4.4B	4B	5B	1.3B	0.83B	5B	5B	5B
Electroplated Al	5B	5B	5B	5B	5B	5B	5B	5B	5B
Sputtered Al	5B	5B	5B	5B	5B	4.7B	5B	5B	5B

[1] Cd plated panels were inadvertently topcoated with MIL-PRF-85285 (PPG CA8211).
[2] Primer pinholes were more prevalent with IVD panels, especially with MIL-PRF-85582 C1.
[3] A solvent wipe prior to painting may have improved results, although compatibility with 85582-N primer was excellent.



IVD-Al with MIL-PRF-85582 C1, after 4 days	LHE Zn-Ni with MIL-PRF-85582 C1, after 4 days	Electroplated Al with MIL-PRF-23377, after 7 days	Sputtered Al with MIL-PRF-23377, after 4 days

Figure 4. Representative Photos of Wet Tape Adhesion Panels

All inorganic coatings passed the JTP criterion for paint adhesion with ratings no less than 4.8 in the one day ambient temperature test. Additionally, all of the chromate post-treated Al coatings performed very well up to the seven day highest temperature test with typical ratings of 5.0. The LHE Zn-Ni coating exhibited slightly lower average paint adhesion values with MIL-PRF-23377 primer in the elevated temperature tests (4.0-4.4 ratings), but performed very well with the non-chromate primer MIL-PRF-85582 Class N (5.0 values). Poor adhesion was observed on the Zn-Ni with MIL-PRF-85582 Class C1, with primer blistering and substantial coating removal by the tape. All of the panels painted and tested in this study were treated the same prior to painting, as they were unwrapped from the coater's packaging the day prior to primer application. Typically, the panels were individually wrapped and sealed in Ziploc bags, thus the delay observed prior to painting was similar for each coating.

3.3 Corrosion

3.3.1 Unscribed Salt Spray (Fog) Corrosion Resistance

This test evaluated the ability of a candidate coating to prevent corrosion of coated substrates exposed to salt spray.

Test Description

The coated test specimens were placed in a salt spray chamber operated IAW ASTM B117-94 (*Standard Practice for Operating Salt Spray [Fog] Testing Apparatus*, approved February 15, 1994). The specimens were examined in the test chamber weekly and appearance was recorded. In addition, the first appearance and progress of white and black corrosion products were recorded. The specimens were removed from the salt spray chamber when examination revealed the formation of red rust from the substrate material. Appearance of the coatings was also rated according to the scale listed in ASTM D1654 *Standard Test Method for Evaluation of Painted and Coated Specimens Subjected to Corrosive Environments* (shown as Table 18).

Table 18. ASTM D1654 Rating Scale for Corrosion Resistance Specimens

Rating Number	Unscribed Rating	Scribe Rating, Distance from Scribe	
	% Area Failed	Millimeters	Inches
10	No Failure	0	0
9	0 to 1	0 to 0.5	0 to 1/64
8	2 to 3	> 0.5 to 1.0	1/64 to 1/32
7	4 to 6	> 1.0 to 2.0	1/32 to 1/16
6	7 to 10	> 2.0 to 3.0	1/16 to 1/8
5	11 to 20	> 3.0 to 5.0	1/8 to 3/16
4	21 to 30	> 5.0 to 7.0	3/16 to ¼
3	31 to 40	> 7.0 to 10.0	¼ to 3/8
2	41 to 55	> 10.0 to 13.0	3/8 to ½
1	56 to 75	> 13.0 to 16.0	½ to 5/8
0	> 75	> 16.0	5/8 +

Rationale

This test is necessary to qualify candidate coatings for use on substrates that are not resistant to corrosion.

Test Methodology

Parameters	5% sodium chloride (NaCl) solution sprayed at 35°C until coating failure. See ASTM B117. Angle panels at 6° off normal.
Type/Number of Specimens	Three 4130 specimens
Experimental Control Specimens	Three 4130 specimens
Acceptance Criteria	Minimum of 3,000 hours exposure before appearance of red rust or comparable to LHE Cd. Record observations of first appearance and progression of white and black corrosion products.
Reference Document	MIL-STD-870B, AMSQQP416

Test Results

Table 19 lists the results of the salt spray exposure of unscribed panels for each of the primary coatings. The table includes the time when red rust first appeared, as well as a description of the coating and rating at the end of the test, for each panel tested. Photos of each of the coatings at the conclusion of the test are located in Figure 5, after the table.

Table 19. Corrosion Resistance Results – Unscribed Panels

Coating	Panel #	First Appearance of Corrosion/Observation	Time of Termination/Observation	Rating at Test Termination
Cd-plated	1	No rust	3000 hours/No damage	9
	2	No rust	3000 hours/No damage	9
	3	No rust	3000 hours/No damage	9
IVD-Al (baseline – Hill AFB)	1	1500 hours/ Significant rusting	1500 hours/ Excessive rusting	0
	2	2000 hours/Sacrificial coating breakdown – pinhole rust	3000 hours/ Significant rusting	0
	3	72 hours/ Significant rusting	168 hours/ Excessive rusting	0
LHE Zinc-Nickel	1	No rust	3000 hours/ Sacrificial coating breakdown/ no rust	9
	2	No rust	3000 hours/ Sacrificial coating breakdown/ no rust	9
	3	No rust	3000 hours/ Sacrificial coating breakdown/ no rust	9

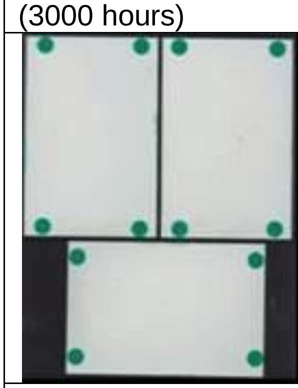
Table 19. Corrosion Resistance Results – Unscribed Panels (Continued)

Coating	Panel #	First Appearance of Corrosion/Observation	Time of Termination/Observation	Rating at Test Termination
Electroplated Al	1	1500 hours/ Chromate depletion and pin holes	3000 hours/ Chromate depletion and pin holes	7
	2	1500 hours/ Chromate depletion and pin holes	3000 hours/ Chromate depletion and pin holes	7
	3	1500 hours/ Chromate depletion and pin holes	3000 hours/ Chromate depletion and pin holes	7
Sputtered Al	1	500 hours/ Excessive rust	500 hours/ Excessive rust	0
	2	500 hours/ Excessive rust	500 hours/ Excessive rust	0
	3	500 hours/ Excessive rust	500 hours/ Excessive rust	0

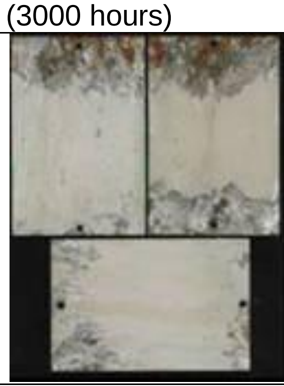
Cd plated
(3000 hours)

IVD-Al (Panel 2)
(3000 hours)

LHE ZN-Ni
(3000 hours)



Electroplated Al
(3000 hours)



Sputtered Al
(500 hours)

Figure 5. Photos of Unscribed Corrosion Resistance Panels After Exposure

The results listed in the table show that the Cd-plated panels met the specification of showing no rust after 3000 hours salt spray exposure. LHE Zn-Ni also performed well, with breakdown of the sacrificial coating noted after 3000 hours exposure, but no rust formation. The electroplated Al coating began exhibiting depletion of the chromate conversion coating at 1500 hours exposure, with some pinhole formation. The appearance of the panels at 3000 hours was the same, indicating that the pinholes did not progress in size during the second half of the test to result in additional red rust.

Two of three IVD-Al panels and the sputtered Al panels developed red rust and were pulled from testing prior to the completion of 3000 hours exposure. The first appearance of corrosion on the IVD-Al panels varied greatly, from 72 hours to 2000 hours. The sputtered Al panels were very consistent, developing excessive rust within 500 hours, terminating the test. However, it is important to note that the specification for IVD-Al coatings, SAE AMS 2427, *Aluminum Coating, Ion Vapor Deposition*, indicates that the acceptance criteria for IVD-Al corrosion resistance is 504 hours. If applied to this testing, two of the three IVD-Al samples would meet this acceptance criteria.

3.3.2 Scribed Salt Spray (Fog) Corrosion Resistance

This test evaluated the ability of a candidate coating to prevent corrosion of coated and scribed substrates exposed to salt spray.

Test Description

The 4 inches x 6 inches coated test specimens were scribed from corner-to-corner forming an "X" pattern. The test specimens were then placed in a salt spray chamber operated IAW ASTM B117-94 (*Standard Practice for Operating Salt Spray [Fog] Testing Apparatus*, approved February 15, 1994). The test specimens were evaluated weekly and appearance was recorded, which included the first appearance and progress of white and black corrosion products. The specimens were removed from the salt spray chamber when red rust was determined during the weekly evaluations. In addition, panels were rated in accordance with ASTM D1654, as listed in Table 18, above.

Rationale

This test is necessary to qualify candidate coatings for use on substrates that are not resistant to corrosion.

Test Methodology

Parameters	5% NaCl solution sprayed at 35°C until coating failure. See ASTM B117. Angle panels at 6° off normal.
Type/Number of Specimens	Three 4130 specimens
Experimental Control Specimens	Three 4130 specimens
Acceptance Criteria	Minimum of 1,000 hours exposure before appearance of red rust or comparable to LHE Cd. Record observations of first appearance and progression of white and black corrosion products.
Reference Document	MIL-STD-870B, AMSQQP416

Test Results

Table 20 lists the test results for the scribed corrosion resistance test panels. Again, the time of the first appearance of corrosion is listed, as well as time of test termination and final rating. Photos are listed in Figure 6, below the table.

Table 20. Corrosion Resistance Results – Scribed Panels

Coating	Panel #	First Appearance of Corrosion/Observation	Time of Termination/Observation	Rating at Test Termination
Cd-plated	1	No rust	3000 hours/No damage	9
	2	No rust	3000 hours/No damage	9
	3	No rust	3000 hours/No damage	9
IVD-Al(baseline – Hill AFB)	1	72 hours/ Excessive rusting	72 hours/ Excessive rusting	0
	2	72 hours/ Significant rusting	168 hours/ Excessive rusting	0
	3	500 hours/ One rust spot on scribe	1000 hours/ Excessive rusting	0
LHE Zinc-Nickel	1	No rust	3000 hours/ Sacrificial coating breakdown/ no rust	9
	2	No rust	3000 hours/ Sacrificial coating breakdown/ no rust	9
	3	No rust	3000 hours/ Sacrificial coating breakdown/ no rust	9
Electroplated Al	1	500 hours/ Chromate depletion and pin holes	3000 hours/ Sacrificial corrosion & rust in scribe	0
	2	1000 hours/ Chromate depletion and rust in scribe	2000 hours/ Sacrificial corrosion & significant rust	0
	3	1000 hours/ Chromate depletion and rust in scribe	2000 hours/ Sacrificial corrosion & significant rust	0
Sputtered Al	1	500 hours/ Excessive rust	500 hours/ Excessive rust	0
	2	500 hours/ Excessive rust	500 hours/ Excessive rust	0
	3	500 hours/ Excessive rust	500 hours/ Excessive rust	0

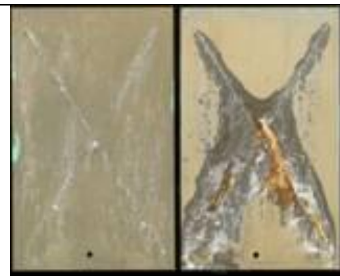
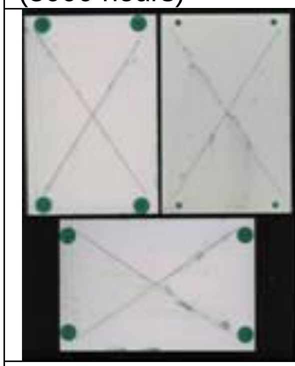
		
Cd plated (3000 hours)	IVD-Al (72 hours)	LHE ZN-Ni (3000 hours)
		
Electroplated Al (2000 hours)	Sputtered Al (500 hours)	

Figure 6. Photos of Scribed Corrosion Resistance Panels after Exposure

The results obtained for the scribed panels were very similar to the results for the unscribed panels. The Cd-plated panels did not exhibit any damage from exposure, as expected. The LHE Zn-Ni received the next highest rating, with some sacrificial coating breakdown but no rust. The electroplated Al followed, but two of three panels had to be pulled prior to 3000 hours due to significant rust. Once again, the IVD-Al and sputtered Al panels had the most significant corrosion present, and testing was terminated at 1000 hours or less.

3.3.3 Galvanic Corrosion Resistance

Providing galvanic compatibility between the dissimilar metals used on high strength steel components and assemblies, such as landing gear, is a critical function of candidate coatings.

<u>Test Description</u>

Baseline and alternative coatings were applied to test washers that were fabricated from 1/8 inch sheet stock of the following materials: 4130 steel, 17-4PH stainless steel, copper beryllium (CuBe), and Aluminum-Nickel-Bronze alloy (Al-Ni-Br). The dimensions of the washers were approximately one inch in diameter with a 0.27 inch hole for fastening the washer to the test block. The sketches of the complete fixture are available in the JTP. Basic components are as follows:

1. Fastener: ¼-28 X 7/8 Hex Cap Screw or equivalent, Stainless Steel
2. Nylon Insert: ¼ Nylon Insert. Length equal to test block + washer thickness
3. Test Block: 0.125–0.250 inches thick, from 2024 Al and 7075 Al, coated with MIL- PRF-85582 Type I, Class N
4. Test Washer (4 substrates with alternative and baseline coatings)
5. Anodized Washer: Anodized Aluminum (NAS1149D0463K) – 2 – secure test washer to test block.

In order to ensure that the assembly was securely held together, a torque wrench was used to tighten the nut to a reading of 70-80 inch-pound (in-lb). The electrical resistivity between the bare area on the test block and the scribed test washer was then measured with an ohmmeter, where the electrical resistivity in all cases was less than one milliohm. A photo of the assembled test fixtures is located in Figure 7.

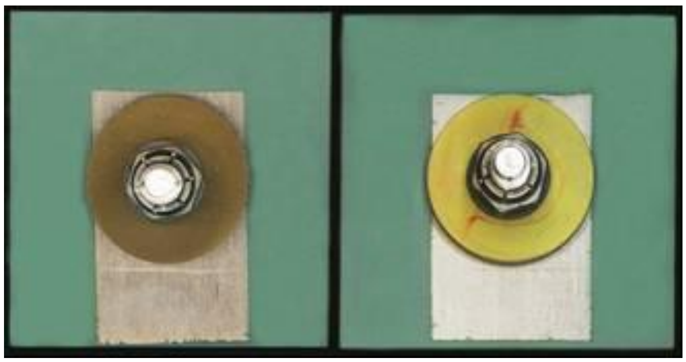


Figure 7. Representative Photos of Test Fixture Assembly

<u>Exposure and Measurement</u>

The test fixtures were then placed in a salt spray chamber and tested IAW ASTM B117-94 (*Standard Practice for Operating Salt Spray [Fog] Testing Apparatus*, approved February 15, 1994) for 168 hours. Duplicate assemblies were placed in a cyclic corrosion chamber and tested IAW GM9540P (*General Motors Engineering Standards, Accelerated Corrosion Test*, issued June 1997) for 336 hours. The assemblies were removed from the corrosion chambers and rinsed to remove the excess salt. The rinsed test assemblies then dried for 3 - 5 hours in air. One ohmmeter probe was placed in the scribe on the washer and the other probe was placed on the test block. As necessary to assure good probe connections, corrosion was removed from the test block and/or washer using sandpaper. The electrical resistivity was then measured and recorded and any corrosion products around the washer were noted. The change in resistivity was then calculated from before to after exposure.

<u>Rationale</u>

This test is necessary to ensure candidate coatings provide adequate galvanic corrosion protection to dissimilar metal systems, especially on Al substrates.

Test Methodology

Parameters	Salt Spray for 168 hours (ASTM B117) and cyclic corrosion for 336 hours (ASTM G85 Annex 5). Angle panels at 6° off normal.
Type/Number of Specimens	Three (3) test assemblies for each candidate coated washer substrate to be installed in each test block material. (48 total; four (4) washer substrates, three (3) washers for each test block, two (2) test block substrates, two (2) exposure conditions) Note: All candidate and control assemblies may be assembled onto one large test block.
Experimental Control Specimens	Three (3) test assemblies with Cd coated washers of each substrate. Three (3) test assemblies with bare washers of each substrate.
Acceptance Criteria	Alternative meets or exceeds Cd in appearance, corrosion resistance, and electrical conductivity (or remains nonconductive for nonconductive coatings).
Reference Document	MIL-STD-870B, AMSQQP416

Test Results

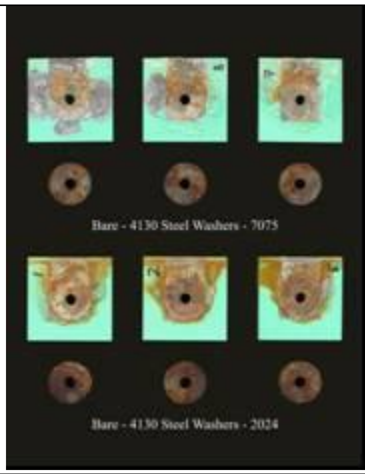
Table 21 lists the change in electrical resistivity for each coating and each substrate material, for 2024 Al and 7075 Al alloy test blocks, exposed to ASTM B117 salt fog for 168 hours. Figure 8 contains photos of the salt spray-exposed assemblies. Table 22 lists the same information for the samples exposed to cyclic corrosion for 336 hours, followed by Figure 9, with photos of the specimens.

Table 21. Change in Resistivity for Salt Spray-Exposed Fixtures

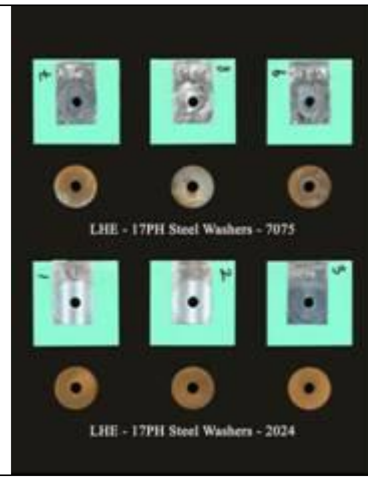
Coating	Test Block Substrate	Change in Resistivity (milliohms) 3 Fixtures Each			
		4130 Washer	17-4PH Washer	CuBe Washer	AlNiBr Washer
Bare (no coating)	2024 Al	62.3, 114, 87.7	66.5, 6.9, 84.6	13.4, 78.5, 1.9	0, 0, 0
	7075 Al	165, 0, 39.8	38.7, 84.3, 687	0.7, 18.5, 18.6	11.8, 0, 0
Cd-plated	2024 Al	0, 0, 0	0.6, 0.4, 0.3	0, 0, 0	0, 0, 0
	7075 Al	0, 0, 0	0.2, 0.3, 0.2	0, 0, 0	0, 0, 0
IVD-Al	2024 Al	0, 0, 0	0, 0, 0.1	0, 0, 0	Infinity, 0, 0
	7075 Al	0, 0	0, 0, broke	0, 0, 0	0, 0
LHE Zn-Ni	2024 Al	0.4, 0.2, 0.4	0.9, 0.4, 0.1	5.1, 1.1, 0	Broke, 0.3, 0.1
	7075 Al	0.5, 0.4, 0.5	11600, 0.6, 0.6	5.6, 2.3, 1.7	1.1, 0.5, 0.6

Table 21. Change in Resistivity for Salt Spray-Exposed Fixtures (Continued)

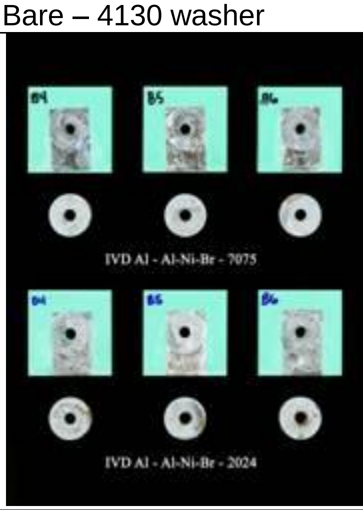
Coating	Test Block Substrate	Change in Resistivity (milliohms) 3 Fixtures Each			
		4130 Washer	17-4PH Washer	CuBe Washer	AlNiBr Washer
Electro-plated Al	2024 Al	0.3, 0.2, 0	0, 0, 0	0, 0, 0	0.4, 0.5, 0.5
	7075 Al	0.3, 0.7, 3.6	1.0, 0.8, 0	0, 0, 0	1.0, 0.5, 0.7
Sputtered Al	2024 Al	0.3, 0, 0	0, 0, 0.2	0.1, 0, 0.1	0, 0.2, 0
	7075 Al	0, 0, 0	0, 0, 0	0, 0.5, 0	0, 0.4, 0.6



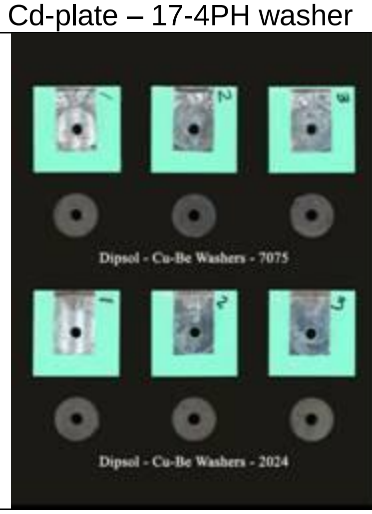
Bare – 4130 washer



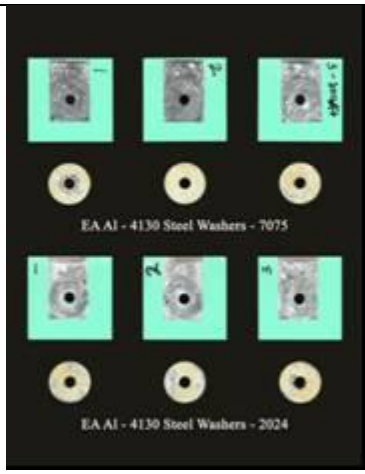
Cd-plate – 17-4PH washer



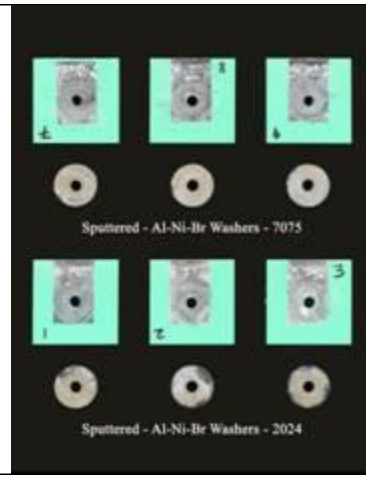
IVD-Al – AlNiBr washer



LHE Zn-Ni – CuBe washer



Electroplated Al – 4130 washer

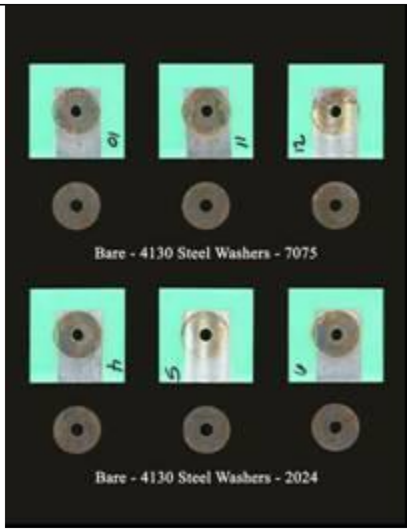


Sputtered Al – AlNiBr washer

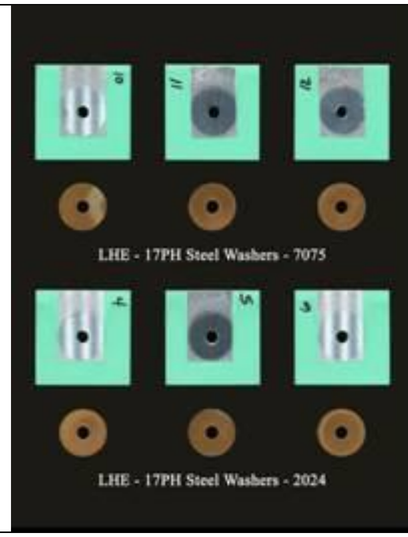
Figure 8. Representative Photos of Salt Spray-Exposed Fixtures

Table 22. Change in Resistivity of Cyclic Corrosion Fixtures

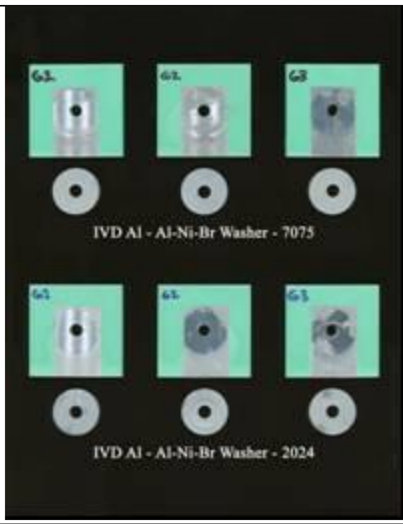
Coating	Test Block Substrate	Change in Resistivity (milliohms) 3 Fixtures Each			
		4130 Washer	17-4PH Washer	CuBe Washer	AlNiBr Washer
Bare (no coating)	2024 Al	80000, 0, 550000	5.6, 0.8, 1.4	0, 0, 0	0, 0, 0
	7075 Al	660000, 1.0, 0.8	15300, 0, 0	0, 0, 0	0, 0, 0
Cd-plated	2024 Al	0, 0, 0	0.6, 0.3, 0.2	0, 0, 0	0, 0.8, 0.6
	7075 Al	0, 0, 0	0.6, 0, 0.8	0, 0, 0	0, 0, 0
IVD-Al	2024 Al	0, 0, 0	0, 0, 0	0, 0, 0	0, 0, 0
	7075 Al	0, 0, 0	0, 0, 0	0, 0, 0	0, 0, 0
LHE Zn-Ni	2024 Al	0, 0, 0	0, 0, 0	0, 0, 0	0, 0, broke
	7075 Al	0, 0, 0.1	0, 0, 0	0, 0, broke	0, 0, 0
Electro-plated Al	2024 Al	0, 0, 0	0, 0, 0	0, 0, 0	0, 0, 0
	7075 Al	0, 0, 0	0, 0, 0	0, 0, 0	0, 0, 0
Sputtered Al	2024 Al	0, 0, 0	0, 0, 0	0, 0, 0.1	0, 0, 0
	7075 Al	0, 0, 0	0, 0, 0	0, 0, 0	0, 0, 0



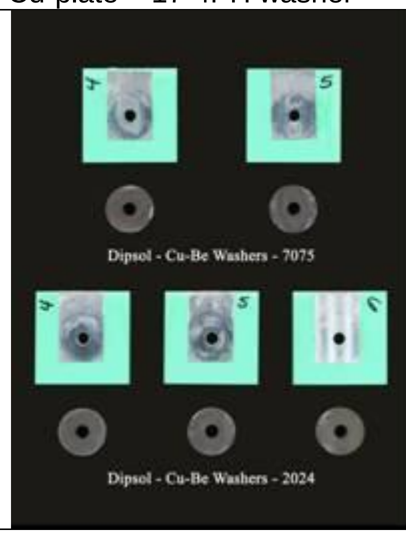
Bare – 4130 washer



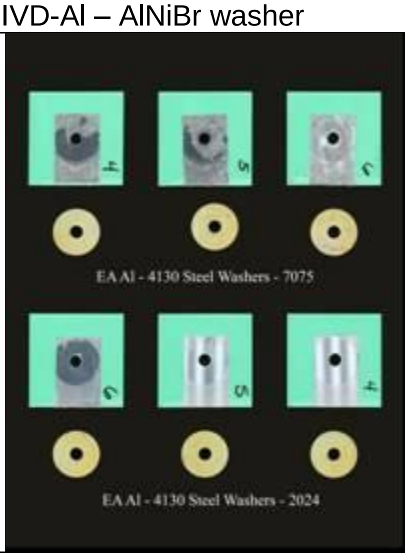
Cd-plate – 17-4PH washer



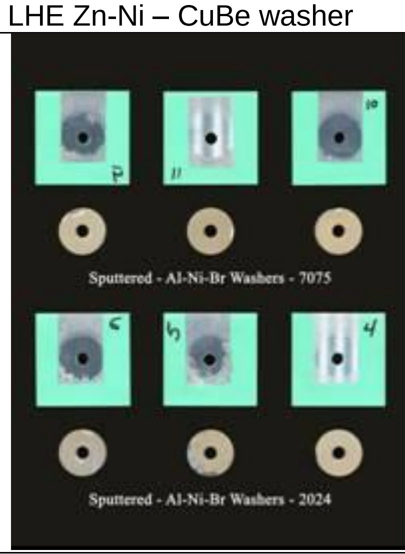
IVD-Al – AlNiBr washer



LHE Zn-Ni – CuBe washer



Electroplated Al – 4130 washer



Sputtered Al – AlNiBr washer

Figure 9. Representative Photos of Cyclic Corrosion-Exposed Fixtures

Both the test results and photos indicate that the fixtures performed better in cyclic corrosion than salt spray. In addition, as expected, the bare test washer exhibited the most corrosion and had the largest changes in conductivity. The Cd-plated test washers only showed a conductivity change for the 17-4PH stainless steel substrate in both the salt spray and cyclic corrosion tests. The alternative coating that had the most changes in conductivity upon salt spray exposure was LHE Zn-Ni. This was followed by electroplated Al and sputtered Al, which both had small changes in conductivity for almost every test washer/test block alloy combination. The exception was electroplated Al on CuBe, which had no change in conductivity. All alternatives exhibited little to no change in conductivity for assemblies exposed to cyclic corrosion.

3.3.4 Fluid Corrosion Resistance

<u>Test Description</u>

Three 1 inch x 2 inches x 0.032 inch 4130 steel test specimens were cut and identified for each test fluid. Three test specimens were coated with each alternative coating, including any proposed conversion coatings to be used, to a minimum thickness of 0.3 mils. In addition, three test specimens were coated with Cd, meeting the requirements of MIL-STD-870B. The test specimens were cleaned and dried and then stored for a minimum of 16 hours in a desiccator over a suitable desiccant. Following desiccation, each specimen was then weighed to the nearest milligram (0.001 grams). Figure 10 shows a representative panel for each coating prior to immersion.

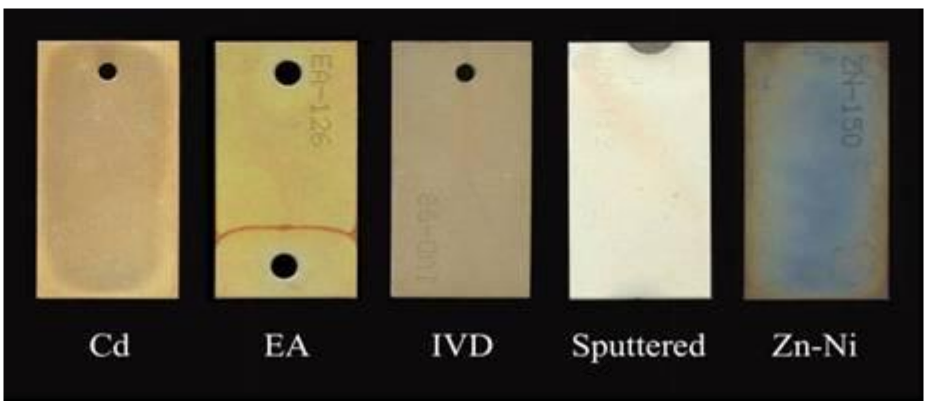


Figure 10. Representative Photo of Coated Panels Prior to Test

The specimens were tested IAW ASTM F483 (*Total Immersion Corrosion Test for Aircraft Maintenance Chemicals*), except that the specimens were immersed for seven days. After immersion, the specimens were removed from the test fluid, cleaned with a suitable solvent, and dried in a desiccator for a minimum of 16 hours. After desiccation, the specimens were reweighed to the nearest milligram. The appearance of the specimens was then rated using the criteria described in ASTM F1110 (*Sandwich Corrosion Test*), which is described in Table 23.

Table 23. ASTM F110 Corrosion Severity Rating System

Rating	Description
0	No visible corrosion (none)
1	Very slight corrosion or discoloration (up to 5% of the surface area corroded)
2	Slight corrosion (5 – 10%)
3	Moderate corrosion (10 – 25%)
4	Extensive corrosion or pitting (> 25%)

The following test fluids were utilized for the immersions:

- Reagent water (ASTM D1193)
- Three parts by volume propylene glycol: one part distilled water
- Synthetic sea water (ASTM D1141)
- Aircraft Deicing/Anti-Icing Fluid (SAE AMS 1424 / 1435)
- Runway deicing fluid (SAE AMS 1435)
- Cleaning compound, parts washer (MIL-C-29602)
- Cleaning compound, aerospace equipment (MIL-PRF-87937 Type I, Type II)
- Paint remover (MIL-R-81294)
- Paint remover (TT-R-2918 Type I [Turco 6813E])
- Paint remover (MIL-PRF-87978 Type I or equivalent)
- Paint remover – peroxide based
- Wheel well cleaning compound (MIL-PRF-85570 Type V)
- Water saturated MIL-PRF-8757 lubricant
- Water saturated MIL-PRF-5606 lubricant.

Rationale

This test is necessary to qualify candidate coatings for use on substrates that may be exposed to fluids, which could promote corrosion.

Test Methodology

Parameters	Immersion in specified fluid at 100°F ±2°F (unless otherwise specified) for seven days, desiccation for 16 hours.
Type/Number of Specimens	Three 4130 specimens, 1 inch x 2 inches x 0.032 inch, per candidate for each test fluid.
Experimental Control Specimens	Three 4130 specimens, 1 inch x 2 inches x 0.032 inch, Cd plated, for each test fluid.
Acceptance Criteria	No coating degradation greater than that of Cd plated control specimens as determined by weight loss and appearance.
Reference Document	MIL-STD-870B, AMSQQP416

Test Results

The following tables and figures list the average weight loss and appearance rating for each coating system exposed to each fluid. Appearance ratings for each coating are based upon comparison to the control specimens for that coating (see Figure 10). The discussion following the figures and Table 24 describes how the alternative coatings performed when compared to the acceptance criteria, which states that the alternatives should perform as well as the Cd plated samples as determined by weight loss and appearance. Figures 11 to 15 contain photos of the Cd-plated panels and each alternative after exposure to the test fluids. Table 24 contains the weight loss and appearance results.

Figure 11. Cd-Plated Panels after Fluid Immersion

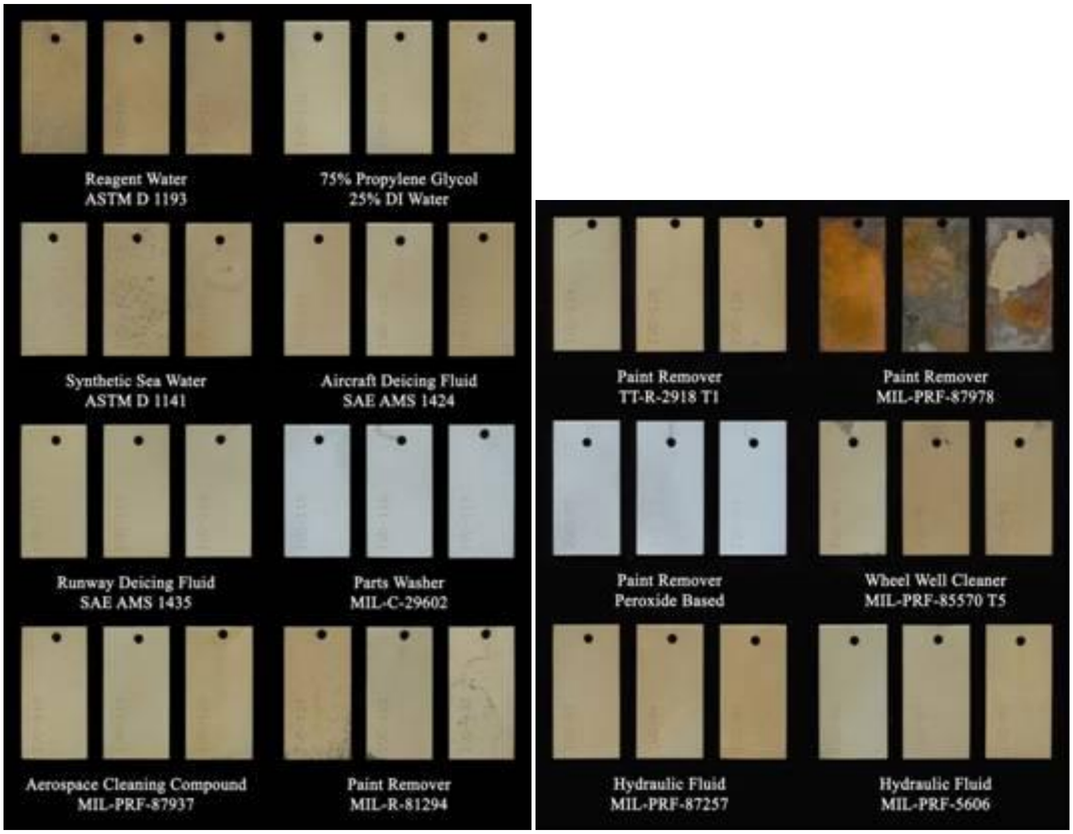


Figure 12. IVD-Aluminum Panels after Fluid Immersion

Figure 13. LHE Zn-Ni Panels after Immersion

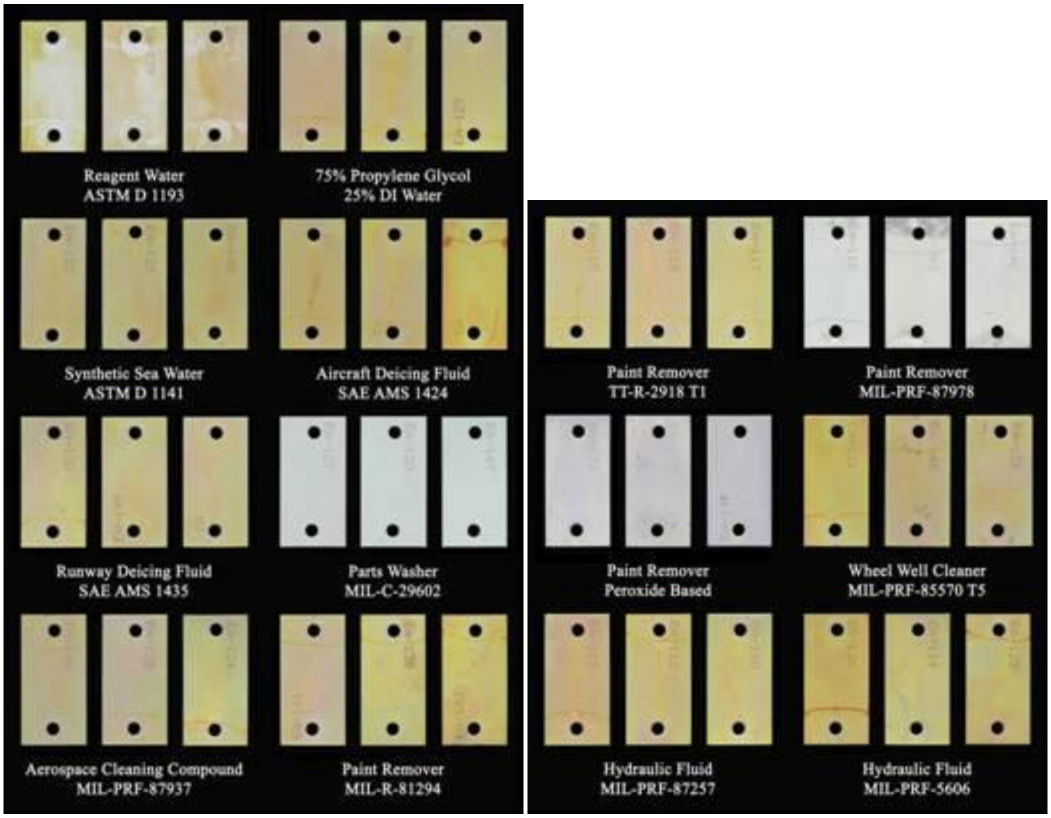


Figure 14. Electroplated Al Panels after Immersion

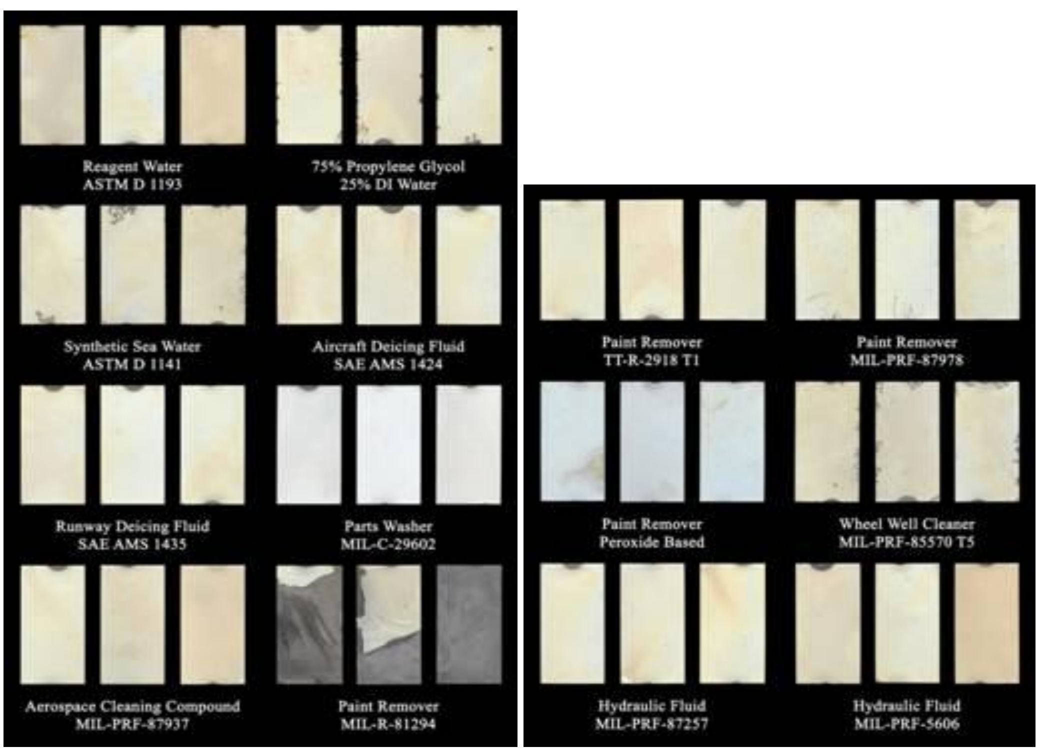


Figure 15. Sputtered Al Panels after Immersion

Table 24. Fluid Corrosion Resistance Test Results for Cd-plated Panels

Test Fluid	Cd-plated		IVD-Al		LHE Zn-Ni		Electroplated Al		Sputtered Al	
	Avg. Δ Mass	Appear. Rating	Avg. Δ Mass	Appear. Rating	Avg. Δ Mass	Appear. Rating	Avg. Δ Mass	Appear. Rating	Avg. Δ Mass	Appear. Rating
Reagent water (ASTM D1193)	0.0015 grams (g)	2	0.0014 g	0	0.0001 g	0	0.0001 g	1	0.0097 g	0
3:1 Propylene glycol to water	0.0008 g	0	0.0006 g	0	0.0000 g	1	0.0003 g	0	0.0008 g	1
Synthetic sea water (ASTM D1141)	0.0033 g	1	0.0016 g	2	0.0001 g	0	0.0002 g	0	0.0008 g	1
Aircraft Deicing/Anti-icing Fluid (SAE AMS 1425/1435)	0.0025 g	0	0.0001 g	0	0.0002 g	0	0.0003 g	0	0.1366 g	0
Runway Deicing Fluid (SAE AMS 1435)	0.0003 g	1	0.0004 g	0	0.0003 g	1	0.0002 g	0	0.0022 g	0
Cleaning Cpd, Parts Washer (MIL-C-29602)	0.0010 g	2	0.0013 g	2	0.0003 g	1	0.0001 g	0	0.0007 g	1
Cleaning Compound, Aerospace Equipment (MIL-PRF-87937 TI/TII)	0.0042 g	2	0.0005 g	0	0.0449 g	0	0.0001 g	0	0.0014 g	0
Paint Remover (MIL-R-81294)	0.0234 g	1	0.0004 g	1	0.0032 g	0	0.0003 g	0	0.2724 g	4
Paint Remover (TT-R-2918 TI)	0.0029 g	2	0.0005 g	0	0.0470 g	3	0.0001 g	0	0.0006 g	0
Paint Remover (MIL-PRF-87978 TI)	0.5235 g	4	0.3125 g	4	0.5920 g	4	0.0010 g	2	0.0000 g	2
Paint Remover – peroxide based	0.1869 g	4	0.0007 g	2	0.0570 g	4	0.0009 g	2	0.0007 g	2
Wheel well cleaning cmpd. (MIL-PRF-85570 TV)	0.0015 g	1	0.0002 g	1	0.0415 g	0	0.0004 g	0	0.0011 g	1
Water Saturated MIL-PRF-87257	0.0010 g	0	0.0002 g	0	0.0001 g	0	0.0004 g	0	0.0155 g	0
Water Saturated MIL-PRF-5606	0.0004 g	0	0.0004 g	0	0.0001 g	1	0.0001 g	0	0.0003 g	0

For the baseline Cd-plated specimens, the group of fluids that had caused the largest change in mass was the paint removers. In addition, when checking the appearance of the samples, the paint removers caused the most corrosion/removal of the coating. One other observation for the Cd-plated panels was that the parts washer cleaning compound and the reagent water appeared to remove the conversion coat. Of the alternative coating systems, the electroplated Al seemed to be least affected by the fluids. Slight changes in weight were measured for the 87978 paint remover and peroxide-based paint remover, with visual examinations also showing some effect along with potential removal of the conversion coat. The parts washer cleaning compound also appeared to remove the conversion coat from the electroplated Al.

The alternative coating that was most affected by the fluid immersion was the LHE Zn-Ni. It performed comparably to the Cd-plated specimens, with the paint removers having the most effect on weight and appearance. Mass was also affected by the aerospace equipment cleaning compound and the wheel well cleaning compound. For the sputtered Al coating, the 81294 paint remover caused the coating to flake off of the panels. Changes in weight resulted from immersion in water-saturated 87257 lubricant, reagent water, and deicing fluid. Overall, it appears that the alternative coatings were comparable to the fluid immersion results of the Cd-plated and IVD-Al baseline panels.

3.3.5 Corrosion Resistance of Scribed Painted Coatings

This test assessed the corrosion resistance of painted candidate coatings. This test best replicated the overall coating system that is used on landing gear and other painted components.

<u>Test Description</u>

Test panels were coated per manufacturer's recommendations. Then, one set of specimens was also coated with a waterborne epoxy primer, conforming to MIL-PRF-85582 Type I, Class C1 (*Primer Coatings: Epoxy, Waterborne*), and allowed to dry in air for 14 days prior to testing. A non-chromated waterborne epoxy primer, reference MIL-PRF-85582 Type I Class N (e.g., PRC Desoto/Spraylat EWAE118 A/B Type II, Class N or Akzo Nobel 10PW22-2/ECW119) was applied to the second set of specimens and dried in air for 14 days prior to testing. A solvent borne primer conforming to MIL-PRF-23377 Type I, Class C (*Primer Coatings: Epoxy, High Solids*) was applied to the third set of specimens and dried in air for 14 days prior to testing.

After paint cure, each specimen was machined from corner-to-corner, forming an "X" pattern, using a 0.030 - 0.060 inch wheel cutter with a "V" cut down to a depth of plating thickness plus 0.010 ± 0.001 inch. The test specimens were then placed in a salt spray chamber operated IAW ASTM B117-94 (*Standard Practice for Operating Salt Spray [Fog] Testing Apparatus*, approved February 15, 1994). The panels were examined weekly and the appearance of the test panels was recorded, to include the first appearance and progress of white and black corrosion products. The specimens were removed from the salt spray chamber after 3,000 hours exposure or when the examination revealed red rust.

Rationale

This test is for comparison purposes. Performance of the candidate coatings should be equal to or better than Cd.

Test Methodology

Parameters	5% NaCl solution sprayed at 35°C until coating failure. See ASTM B117. Angle panels at 6° off normal.
Type/Number of Specimens	Three 4130 specimens for each primer and topcoat
Experimental Control Specimens	Three 4130 specimens for each primer and topcoat
Acceptance Criteria	Performance equal to or better than LHE-Cd or no red rust after 3,000 hours exposure. Record observations of first appearance and progression of white and black corrosion products.
Reference Document	MIL-STD-870B, AMSQQP416

Test Results

Table 25 lists the corrosion resistance results for the painted and scribed panels. Representative photos are listed in Figure 16. As listed in the Panel # column, the following primers were applied to the panels, followed by MIL-PRF-85285 topcoat:

P1 = Deft MIL-PRF-23377, Class C2
P2 = Deft MIL-PRF-85582, Class C1
P3 = Deft MIL-PRF-85582, Class N

Table 25. Corrosion Resistance Results – Painted and Scribed Panels

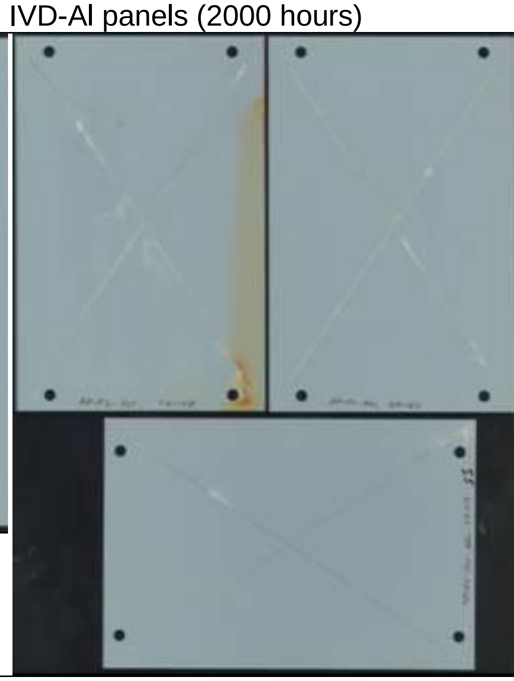
Coating	Panel #	First Appearance of Corrosion/Observation	Time of Termination/Observation	Rating at Test Termination
Cd-plated	1 – P3	336 hours/ scribe rust	3000 hours/ scribe rust	9
	2 – P3	336 hours/ scribe rust	3000 hours/ scribe rust	9
	3 – P3	336 hours/ scribe rust	3000 hours/ scribe rust	9
	4 – P2	168 hours/ scribe rust	3000 hours/ scribe rust	9
	5 – P2	2000 hours/ scribe rust	3000 hours/ scribe rust	9
IVD-Al(baseline – Hill AFB)	1 – P1	336 hours/some creepage from scribe	3000 hours/ 5 – 7 mm creepage from scribe	4
	2 – P2	168 hours/pinhole rust	2000 hours/ Excessive rusting	0
	3 – P2	168 hours/5 – 7 mm creepage from scribe	2500 hours/excessive pinhole rusting	0
	4 – P3	168 hours/pinhole rust with 3 – 5 mm creepage from scribe	1000 hours/excessive pinhole rusting	0

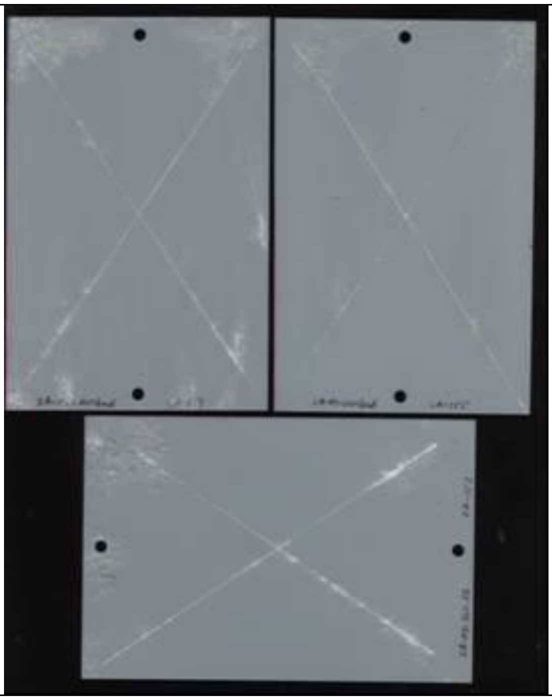
Table 25. Corrosion Resistance Results – Painted and Scribed Panels (Continued)

Coating	Panel #	First Appearance of Corrosion/Observation	Time of Termination/Observation	Rating at Test Termination
IVD-Al(baseline – Hill AFB)	5 – P3	168 hours/some creepage from scribe	2500hours/excessive scribe rust	0
LHE Zinc-Nickel	1 – P1	No rust	3000 hours/ Sacrificial coating breakdown/ no rust	9
	2 – P1	No rust	3000 hours/ Sacrificial coating breakdown/ no rust	9
	3 – P2	336 hours/pinhole rust	3000 hours/pinhole rust, 5 – 7 mm creepage	4
	4 – P2	2500 hours/field blisters	3000 hours/field blisters	9
	5 – P3	2500 hours/pinhole rust with 2-3 mm creepage	3000 hours/pinhole rust with 3-5 mm creepage	5
Electro-plated Al	1 – P2	168 hours/slight creepage	3000 hours/scribe rust with field blisters	0
	2 – P2	168 hours/2-3 mm creepage	3000 hours/7-10 mm creepage	3
	3 – P2	168 hours/slight creepage	3000 hours/pinhole rust with 13-16 mm creepage	1
	4 – P3	168 hours/slight creepage	3000 hours/7-10 mm creepage	3
	5 – P1	336 hours/2-3 mm creepage	3000 hours/3-5 mm creepage	5
Sputtered Al	1 – P3	336 hours/2-3 mm creepage	1500 hours/ Excessive rust	0
	2 – P2	168 hours/slight creepage	1500 hours/ Excessive rust	0
	3 – P1	168 hours/5-7 mm creepage	1500 hours/ Excessive rust	0
	4 – P2	168 hours/2-3 mm creepage	1500 hours/ Excessive rust	0
	5 – P3	168 hours/slight creepage	1500 hours/ Excessive rust	0

The corrosion resistance results for the painted panels were similar to the results for the unpainted panels. Again, the Cd-plated panels performed the best, with only rust in the scribed area after 3000 hours of exposure, resulting in a “9” rating. As with the unpainted panels, LHE ZN-Ni followed with three panels having a “9” rating after 3000 hours exposure. The other 2 panels were rated at 4 and 5 after 3000 hours due to pinhole rust and some creepage from the scribe.

The electroplated Al panels were the next to follow in rank of performance. These panels were all exposed for 3000 hours, with final ratings of 0, 1, 3, and 5, due to various levels of creepage of corrosion from the scribe. The sputtered Al panels were all pulled from testing after 1500 hours due to excessive rust formation. The first signs of corrosion developed after 168 hours, as creepage from the scribe. Finally, the IVD-Al baseline had four specimens with “0” ratings that were pulled from testing from 1000 to 2500 hours. One IVD-Al specimen, the only IVD-Al panel coated with 23377 primer, remained exposed for 3000 hours and received a "4" rating, based on the length of creepage from the scribe. Otherwise, it does not appear that any particular primer performed better or provided more corrosion resistance.

	
Cd-plated panels (3000 hours)	IVD-Al panels (2000 hours)
	
LHE Zn-Ni (3000 hours)	Electroplated Al (3000 hours)



Sputtered Al panels (1500 hours)	

Figure 16. Representative Painted and Scribed Corrosion Resistance Panels

3.3.6 Navy Added Corrosion Testing Requirements

3.3.6.1 Unscribed Cyclic SO_2 Salt Spray (Fog) Corrosion Resistance

This test evaluated the ability of a candidate coating to prevent corrosion of coated substrates exposed to cyclic SO_2 salt spray.

Test Description

The coated test specimens were placed in a salt spray chamber operated IAW ASTM G85 Annex 4 (*Modified Salt Spray [Fog] Testing, Cyclic SO_2 Salt Spray Test)*. The panels were examined daily for the first week and then weekly to record performance. The first appearance and progress of white and black corrosion products was noted and the specimens were removed from the salt spray chamber when examination revealed red rust.

Rationale

This test is necessary to qualify candidate coatings for use in environments with significant exposures to SO_2 gas.

Test Methodology

Parameters	Exposure to 5% NaCl solution and SO_2 gas IAW ASTM G85 A4 until coating failure. Coupons racked at 15-degree angle.
Type/Number of Specimens	Three 4130 specimens
Experimental Control Specimens	Three 4130 specimens
Acceptance Criteria	Performance equal to or better than LHE-Cd. Record observations of first appearance and progression of white and black corrosion products.
Reference Document	MIL-STD-870B, AMSQQP416

Test Results

The graph in Figure 17 shows how the ratings by visual examination of the panels in the chamber changed over time. From the graph, it is very clear that all coatings performed at least two times better than Cd, with the Al coatings also performing better than LHE Zn-Ni. Figure 18 contains representative photos of each of the coatings at the given exposure times.

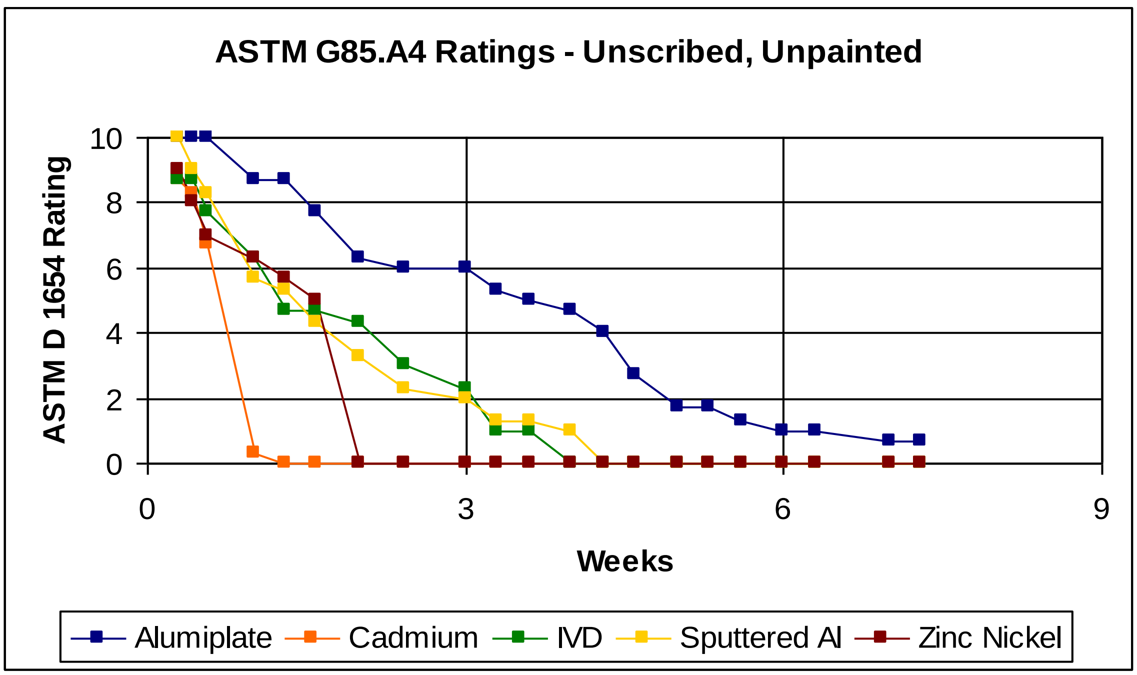
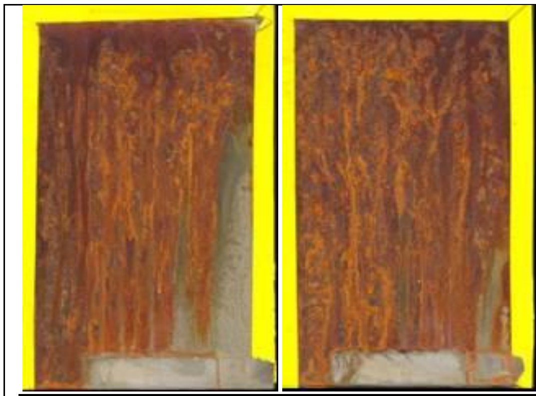


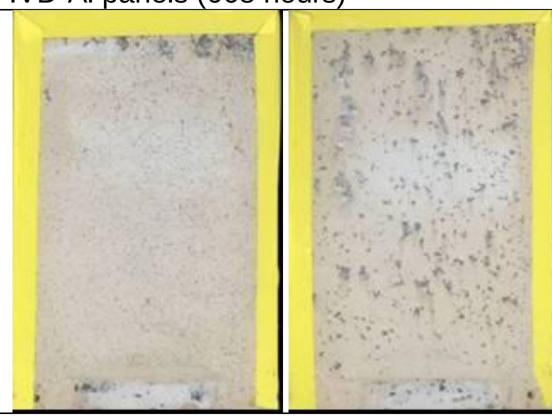
Figure 17. Graph of Ratings for SO_2 Corrosion Resistance – Unscribed Panels



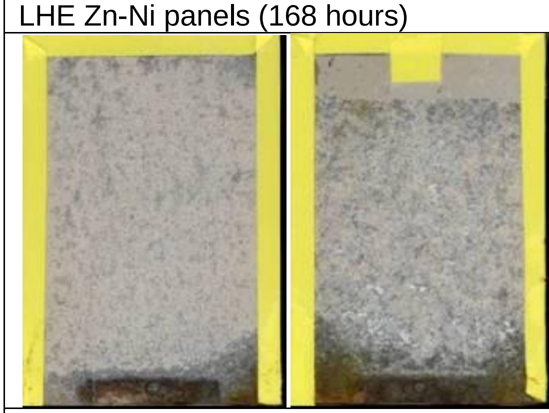
Cd-plated panels (96 hours)

IVD-Al panels (668 hours)

LHE Zn-Ni panels (168 hours)



Electroplated Al (668 hours)



Sputtered Al (668 hours)

Figure 18. Representative Photos of SO_2 Salt Fog-Exposed Panels

In the acidified salt fog environment (ASTM G85, Annex 4), the Al coatings generally performed best. In the unscribed condition, Cd failed significantly in four days (96 hour). The LHE Zn-Ni coating performed better than Cd in this study; however, some red rust was apparent after one week. After two weeks, the Zn-Ni looked similar to Cd after one week (i.e. mostly corroded), and hence performed about twice as well as Cd in this environment. IVD-Al exhibited red rust on one of three panels prior to the 3 week (500 hour) inspection, and is shown at the 4 week (668 hour) inspection in Figure 18. For this part of the testing (SO_2 salt fog), the IVD-Al panels had been provided by Navy Fleet Readiness Center Southwest. The electroplated Al coating had the best appearance of the alternatives when evaluated after 668 hours, where two of three panels were significantly better than either of the other Al coatings tested. The sputtered Al coating was fairly similar to IVD-Al in performance after excluding the bottom portions of the panels, which failed prematurely due to inferior protection of the back surface (Note: IVD-Al and Alumiplate coated panels had Al coatings applied on both sides of the panels, while the line-of-sight sputtered coating was applied to only the front surface. Therefore, premature coating failure initiated from the bottom of the sputtered Al panels).

3.3.6.2 Scribed Cyclic SO_2 Salt Spray (Fog) Corrosion Resistance

This test evaluated the ability of a candidate coating to prevent corrosion of coated and scribed substrates exposed to cyclic SO_2 salt spray.

Test Description

After coating, each specimen was scribed from corner-to-corner to form an "X" pattern. The test specimens were placed in a salt spray chamber operated IAW ASTM G85 Annex 4 (*Modified Salt Spray [Fog] Testing, Cyclic SO_2 Salt Spray Test)*. The panels were examined in the test chamber daily for the first week and then weekly to record performance. The first appearance and progress of white and black corrosion products was recorded and the specimens were removed from the salt spray chamber when examination revealed red rust.

Rationale

This test is necessary to qualify candidate coatings for use in environments with significant exposures to SO_2 gas.
Test Methodology

Parameters	Exposure to 5% NaCl solution and SO_2 gas IAW ASTM G85 A4 until coating failure. Coupons racked at 15-degree angle.
Type/Number of Specimens	Three 4130 specimens
Experimental Control Specimens	Three 4130 specimens
Acceptance Criteria	Performance equal to or better than LHE-Cd. Record observations of first appearance and progression of white and black corrosion products.
Reference Document	MIL-STD-870B, AMSQQP416

Test Results

Figure 19 contains a graph of the results of SO_2 corrosion resistance for the scribed panels, which are very similar to the results seen for the unscribed panels, with all alternative coatings performing better than the Cd-plated panels. Figure 20 contains representative photos of the panels at the exposure times listed in the figure.

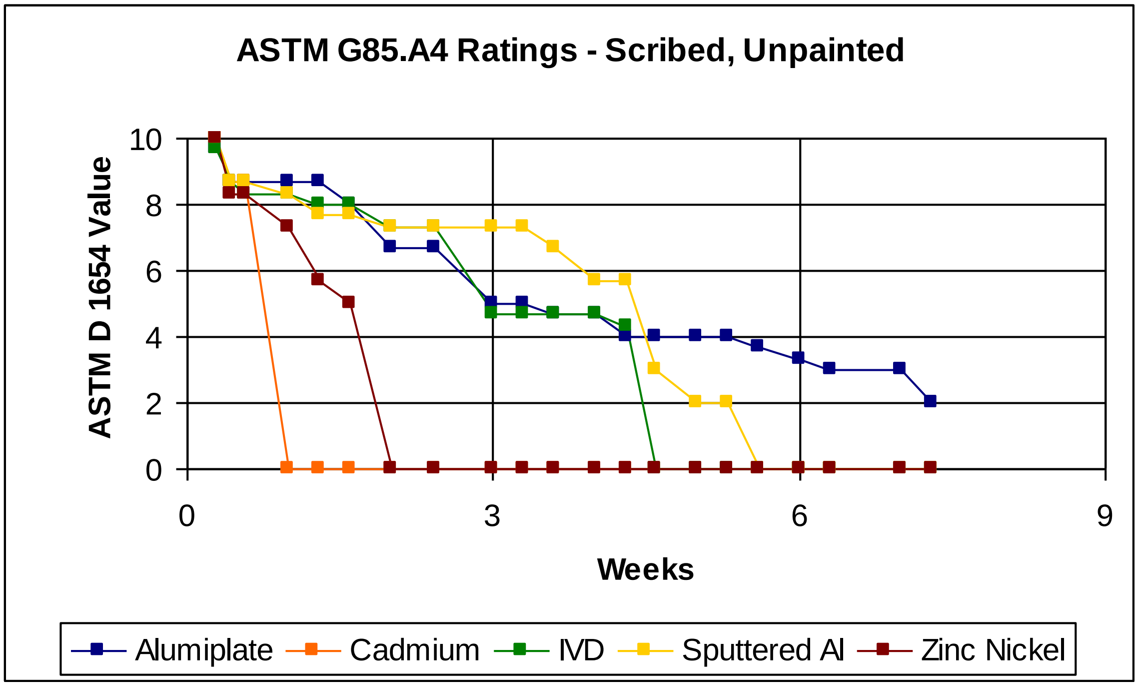
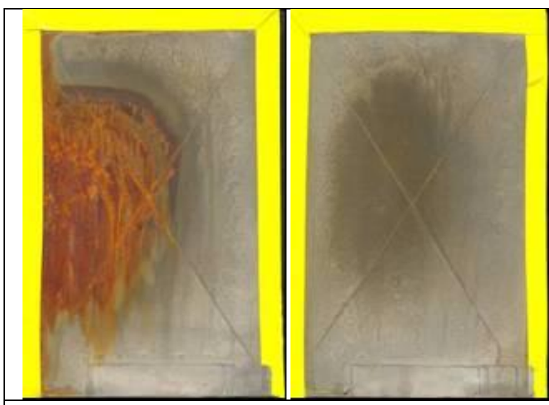
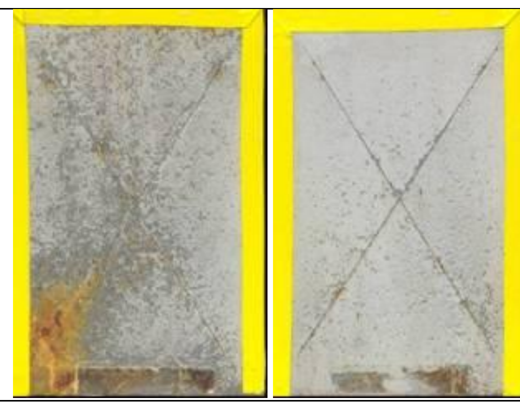
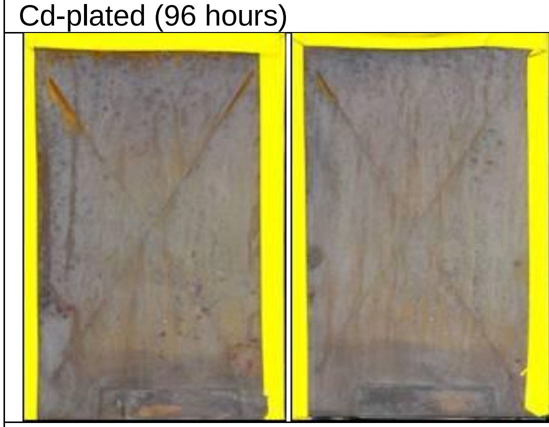
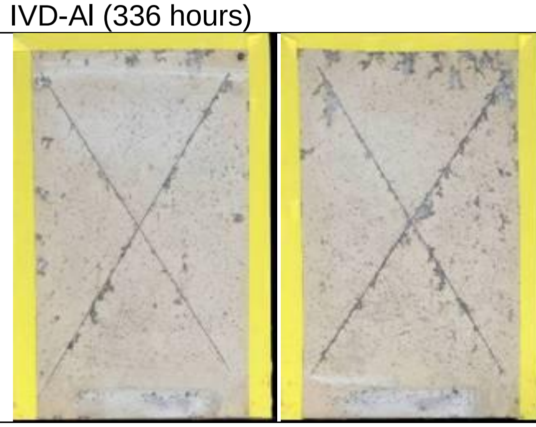


Figure 19. Graph of Ratings for SO_2 Corrosion Resistance – Scribed Panels

	
Cd-plated (96 hours)	IVD-Al (336 hours)
	
LHE Zn-Ni (168 hours)	Electroplated Al (668 hours)

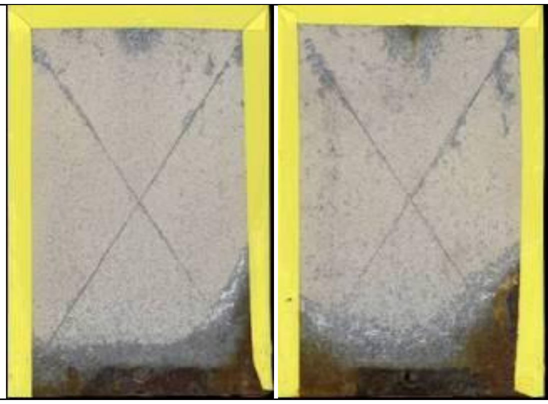
	
Sputtered Al (668 hours)	

Figure 20. Representative Photos of SO_2 Salt Fog-Exposed Scribed Panels

Two out of three Cd plated panels exhibited red rust by 96 hours. At 168 hours, the LHE Zn-Ni panels also exhibited red rust, which was most noticeable in the top portions of the scribes. After two weeks the LHE Zn-Ni had failed and was similar in appearance to the Cd panels after one week. After two weeks (336 hours) the IVD-Al panels contained some red rust in the scribes as well as in the field area of one panel. The electroplated Al and sputtered Al coatings both resisted red rust in the scribes at 668 hours. This test was concluded at approximately seven weeks, as shown in the graph in Figure 19. From this graph, it is evident that the Al coatings were the best performers.

3.3.6.3 Cyclic SO_2 Salt Spray (Fog) Corrosion Resistance of Scribed Painted Coatings

This test assessed the corrosion resistance of painted candidate coatings. This test best replicated the overall coating system that is used on painted HSS components.

<u>Test Description</u>

Cd alternative coatings were primed with each of three different epoxy primers (1) MIL-PRF-23377 Class C2 (strontium chromate inhibited); (2) MIL-PRF-23377 Class N (non-chromate inhibited); and (3) MIL-PRF-85582 Class N (non-chromate inhibited). A standard polyurethane topcoat conforming to MIL-PRF-85285 was applied and then the panels were cured for two weeks. The panels were then manually scribed using a carbide tipped scribe tool and exposed to the acidic salt fog environment (IAW ASTM G85 Annex 4: *Modified Salt Spray [Fog] Testing Cyclic SO_2 Salt Spray Test*). Panels were periodically examined to rate performance, noting the appearance and progress of white and/or black corrosion product. Specimens were then removed from test after the appearance of red rust.

<u>Rationale</u>

This test is for comparison purposes. Performance of the candidate coatings should be equal to or better than Cd.

Test Methodology

Parameters	Exposure to 5% NaCl solution and SO_2 gas IAW ASTM G85 A4 until coating failure. Coupons racked at 15-degree angle.
Type/Number of Specimens	Three 4130 specimens for each primer and topcoat
Experimental Control Specimens	Three 4130 specimens for each primer and topcoat
Acceptance Criteria	Performance equal to or better than LHE-Cd. Record observations of first appearance and progression of white and black corrosion products.
Reference Document	MIL-STD-870B, AMSQQP416

Test Results

Figures 21, 22, and 23 contain graphs of the SO_2 corrosion resistance results for each primer tested. Overall, the best protection from red rust at the scribes was provided by the Al coatings, as would be expected from the bare coating results presented earlier. However, even though there was little to no red rust observed throughout the duration of the test, significant field blistering and adhesion issues were noted as the test progressed. Figure 24 contains representative photos of the test panels.

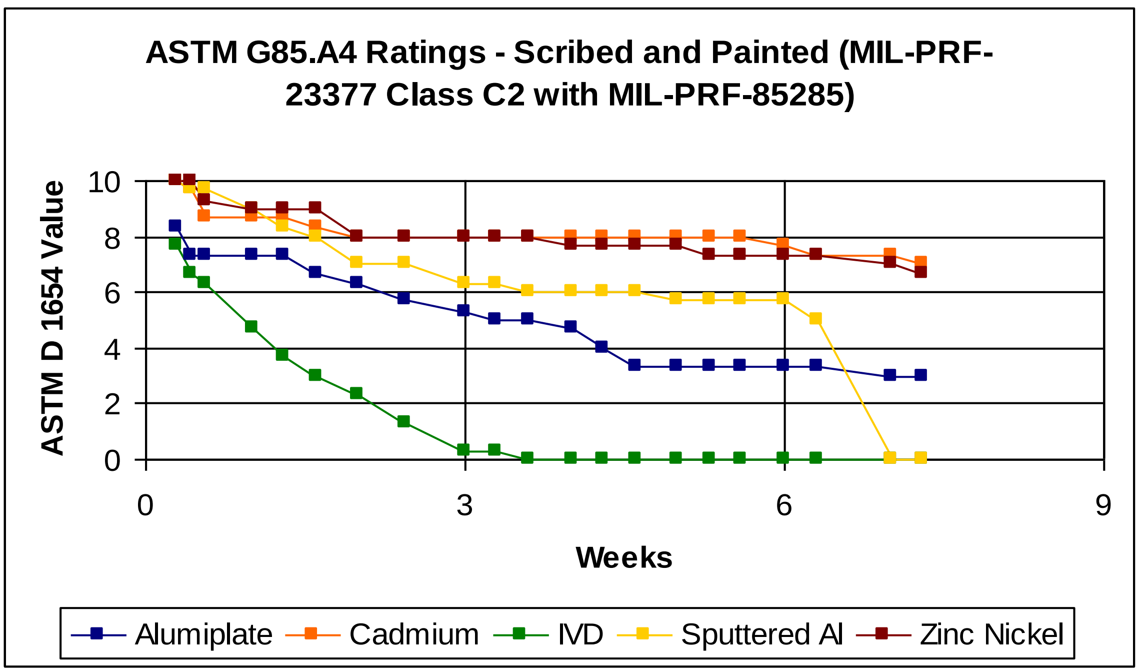


Figure 21. Graph of Ratings for SO_2 Corrosion Resistance – Panels Painted with MIL-PRF-23377 Class C2

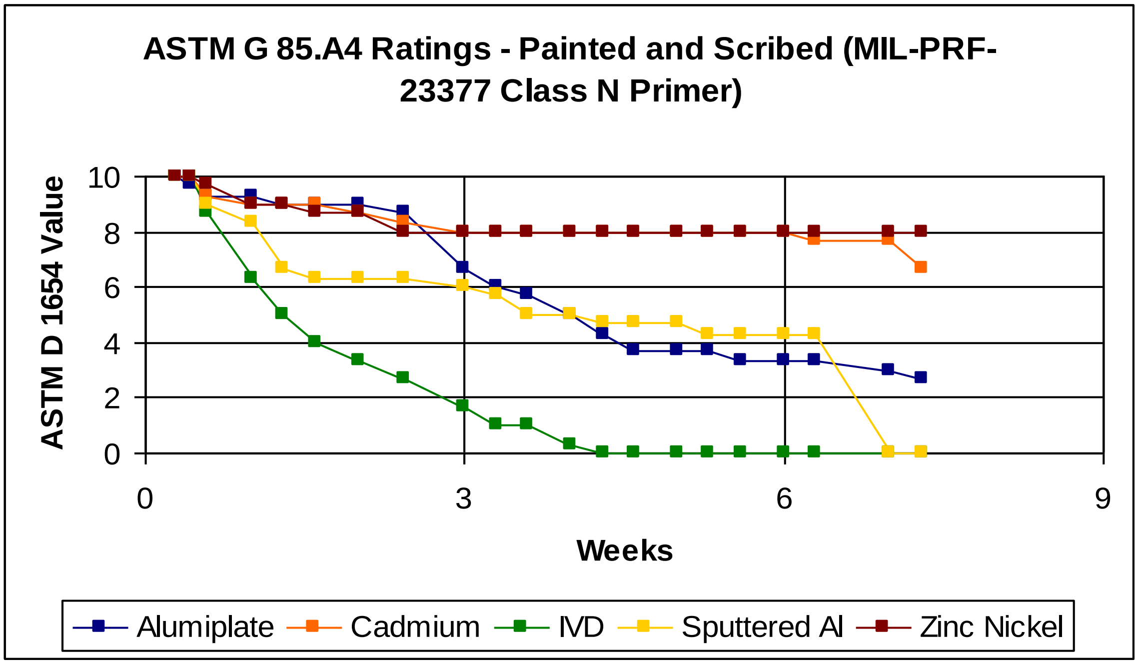


Figure 22. Graph of Ratings for SO_2 Corrosion Resistance – Panels Painted with MIL-PRF-23377 Class N

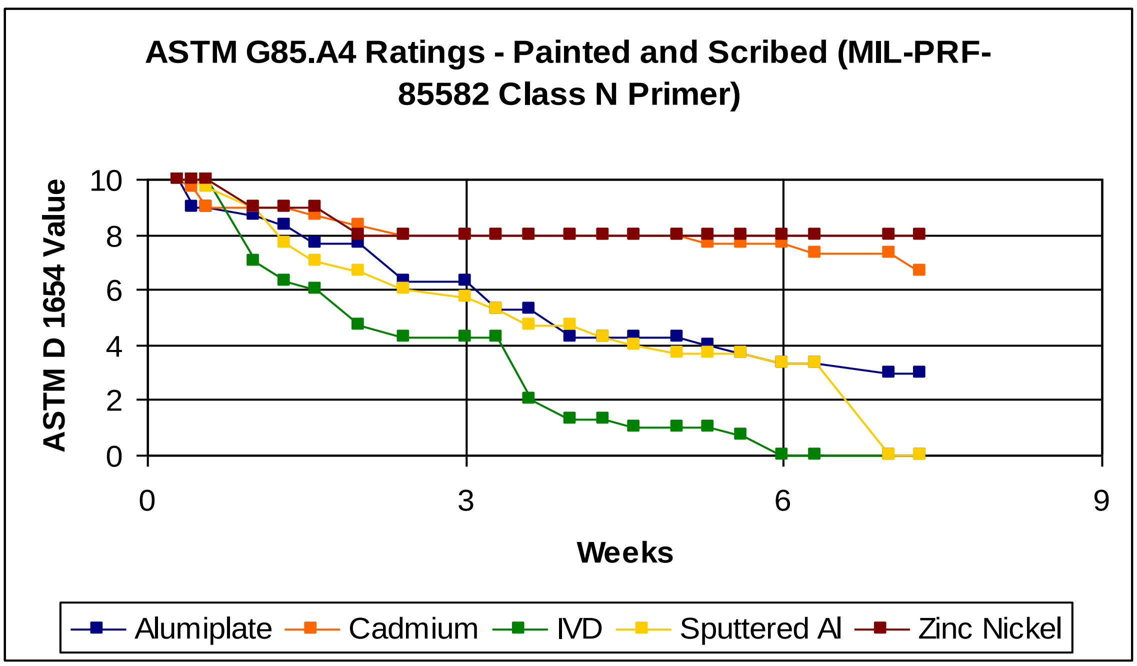
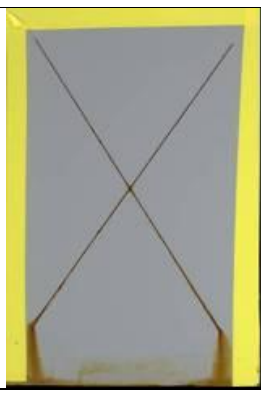
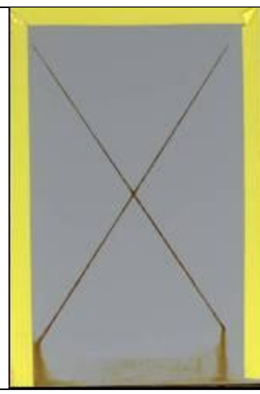
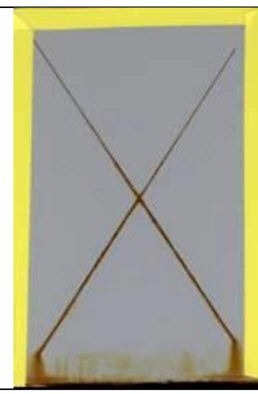


Figure 23. Graph of Ratings for SO_2 Corrosion Resistance – Panels Painted with MIL-PRF-85582 Class N

Both Cd and LHE Zn-Ni performed very similarly with each primer/paint system, whereby each experienced red rust formation early in the test which filled the scribe, although there was no blistering of the paint system adjacent to the scribe or in the field areas (see Figure 24).

As seen in Figures 21, 22, and 23, the IVD-Al panels consistently received the lowest ratings. For the electroplated Al painted with the MIL-PRF-23377-C2 primer, there was a small amount of blistering at the scribe. With the 23377-N primer, larger blisters were evident along the scribe, while the 85582-N primer also showed significant blistering along the scribes. The sputtered Al panels exhibited blistering failures initiating from the bottom edge of the panels, which were not protected on the backsides or bottom edges as well as the other Al coatings. This is because both IVD and electroplating processes are capable of coating backsides/edges, whereas sputtering is a line-of-sight process. Nevertheless, it may be observed that the 85582-N primer provided the least resistance to blistering of the three primers in this study. Graphically, the ratings for each primer/paint system are fairly consistent relative to performance.

Results of this accelerated corrosion test are influenced by the quality of the conversion coating applied by the coating vendors. In the case of the sputtered Al, the panels arrived at NAVAIR, Patuxent River for test in the non-conversion coated condition since the vendor did not have this capability. A fresh chromate conversion coating was applied the day prior to the primer application, so time-to-paint was not a factor for the observed paint adhesion failure on Sputtered Al. All panels were shipped by the vendors in a fairly tightly wrapped condition, and they were unpacked the day prior to primer application to ensure the surfaces remained clean.

		
Cd plating with MIL-PRF-23377 C2 (1168 hours)	Cd plating with MIL-PRF-23377 N (1168 hours)	Cd plating with MIL-PRF-85582 N (1168 hours)
		
IVD-Al with MIL-PRF-23377 C2 (500 hours)	IVD-Al with MIL-PRF-23377 N (668 hours)	IVD-Al with MIL-PRF-85582 N (840 hours)

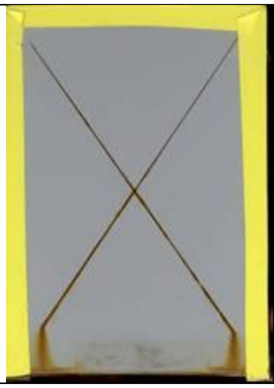
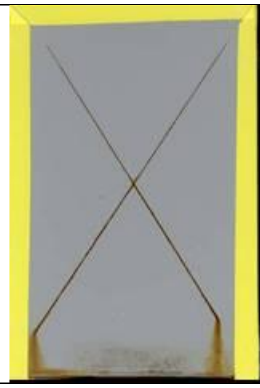
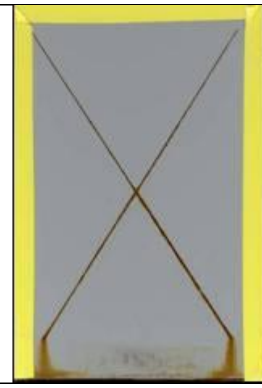
		
LHE Zn-Ni with MIL-PRF-23377 C2 (1168 hours)	LHE Zn-Ni with MIL-PRF-23377 N (1168 hours)	LHE Zn-Ni with MIL-PRF-85582 N (1168 hours)
		
Electroplated Al with MIL-PRF-23377 C2 (1168 hours)	Electroplated Al with MIL-PRF-23377 N (1168 hours)	Electroplated Al with MIL-PRF-85582 N (1168 hours)
		
Sputtered Al with MIL-PRF-23377 C2 (1168 hours)	Sputtered Al with MIL-PRF-23377 N (1168 hours)	Sputtered Al with MIL-PRF-85582 N (1168 hours)

Figure 24. Representative Photos of SO_2 Salt Fog-Exposed Painted Scribed Panels

3.4 Lubricity

3.4.1 Run-on and Breakaway Torque

This test measures the maximum torque value during the assembly of a nut on a bolt, and the torque required to initiate removal of a threaded part (breakaway torque). If the maximum locking torque is too high, the preload is low, shortening the fatigue life. If the minimum breakaway torque is too low, the nut may vibrate off during use.

Test Description

Fasteners were received from the vendor with a Cd plate. This coating was stripped at *CTC* using ammonium nitrate, then the fasteners were sent to the coating vendors for application of the candidate coatings. To conduct the test, the nut was first lubricated with SAE AMS 2518 (*Thread Compound, Anti-Seize*, *Graphite-Petrolatum*, revised July 2001). The nut was installed and removed once at room temperature. A drawing of the test fixture is located in Figure 25.

Next, the maximum locking torque was measured after two (2) complete turns (720° rotation) from the point where the top of the nut is flush with the end of the bolt. The maximum locking torque was the highest reading obtained during the third full turn (360° rotation).

The breakaway torque was then measured during removal of the nut from the clamped up threaded part. The nut was removed from the test bolt between each cycle. In addition, any loosened particles were blown off with compressed air, if necessary, before continuing. The locking and breakaway torque was measured for 15 lock/breakaway cycles and at completion of the testing, the nut and bolt were examined for thread damage at ten (10) times magnification.

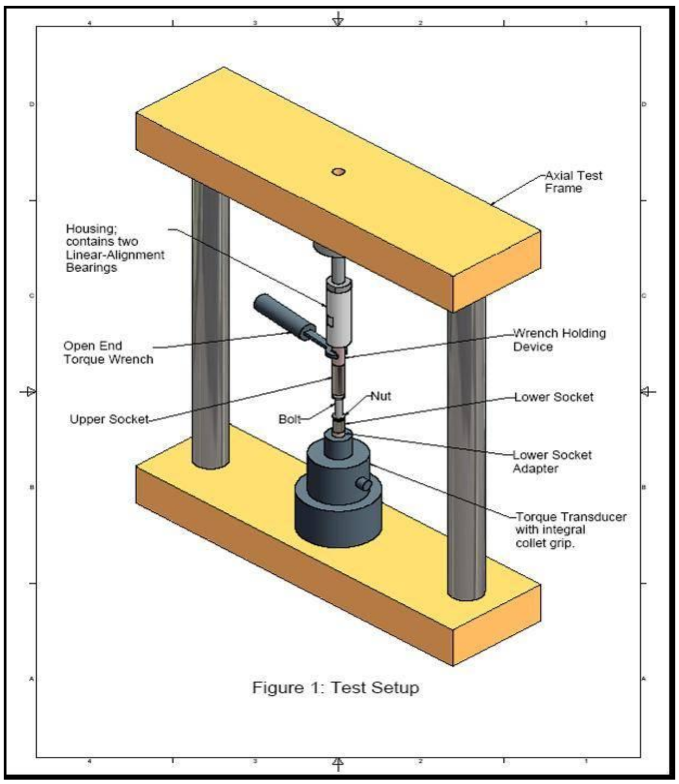


Figure 25. Maximum Locking Torque and Breakaway Torque Test Setup

Rationale

This test is necessary to qualify candidate coatings for use on threaded parts, in order to determine the maximum locking torque and minimum breakaway torque of threaded parts. The acceptance criteria are in conformance with NASM25027.

Test Methodology

Parameters	Using an adequate torque wrench, the locking and breakaway torque of fasteners was measured for 15 lock/breakaway cycles. Fasteners were also examined for damage.
Type/Number of Specimens	Test Bolts/Nuts: NASM21250-06032/ NAS1804-6 (alloy steel, candidate coated), Five specimens Test Bolts/Nuts: NASM21250-10032/ NAS1804-10 (alloy steel, candidate coated), Five specimens
Experimental Control Specimens	Test Bolts/Nuts: NASM21250-06032/ NAS1804-6 (alloy steel, Cd plated), Five specimens Test Bolts/Nuts: NASM21250-10032/ NAS1804-10 (alloy steel, Cd plated), Five specimens
Acceptance Criteria	During installation, the maximum Locking Torque shall not exceed 80 in-lb for -06032 and 300 in-lb for -10032. During removal, the minimum breakaway torque shall not be less than 9.5 in-lb for -06032 and 32 in-lb for -10032. After 15 cycles locking torque test, nut and bolt threads shall remain in serviceable condition: no thread peel, missing segments, cracks, galling, or splits when examined at 10 times magnification; thread peel, missing segments, cracks, galling, or splits are unacceptable.
Reference Document	MIL-STD-870B, AMSQQP416

Test Results

Figures 26 through 29 contain graphs of the maximum locking torque and breakaway torque for each size fastener, with the alternative coating systems and Cd baseline. Note that the acceptance criterion for the 3/8-inch fastener is a locking torque of less than 80 in-lb and a minimum breakaway torque of 9.5 in-lb. The criterion for the 5/8-inch fastener is a locking torque of less than 300 in-lb and a breakaway torque of more than 32 in-lb. In addition to the graph, all data is available in Appendix B of this document.

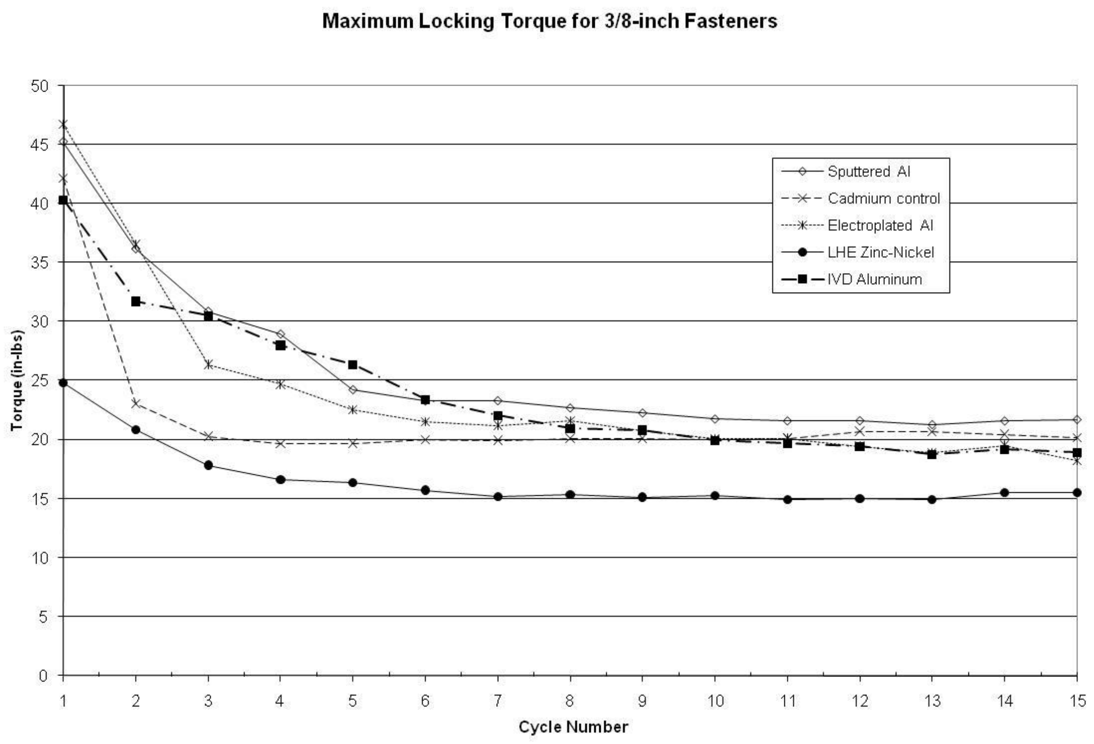


Figure 26. Graph of Maximum Locking Torque for the 3/8-inch Fasteners

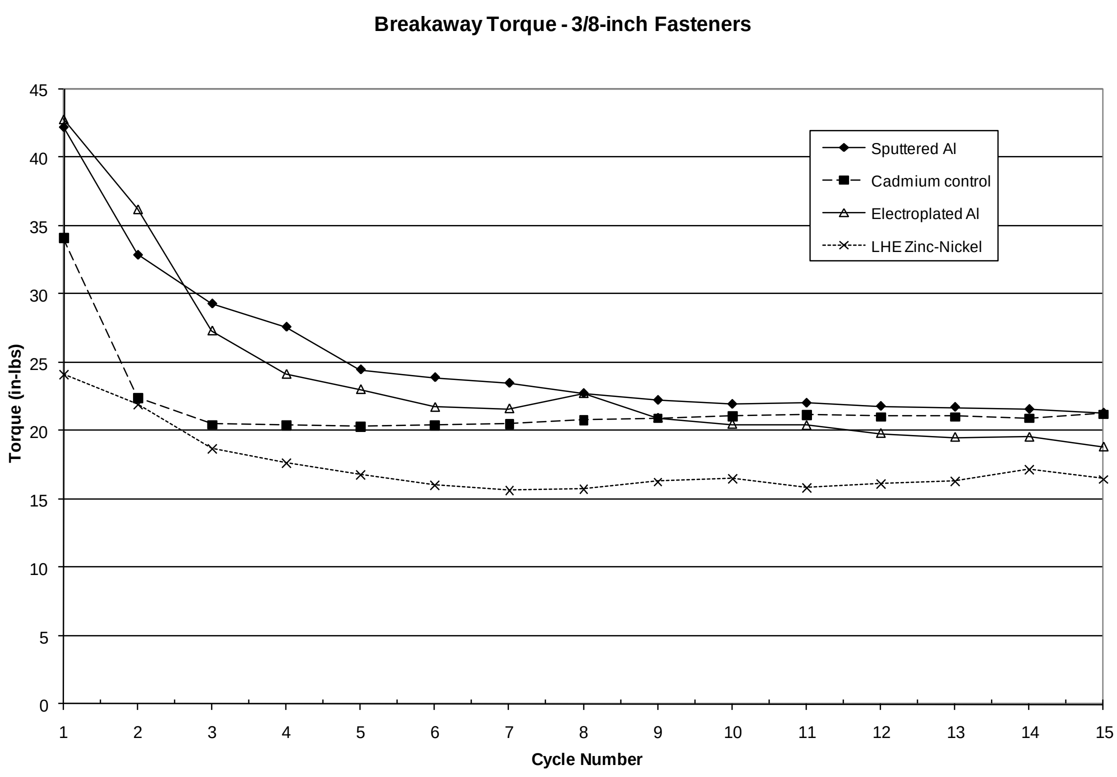


Figure 27. Graph of Breakaway Torque for 3/8-inch Fasteners

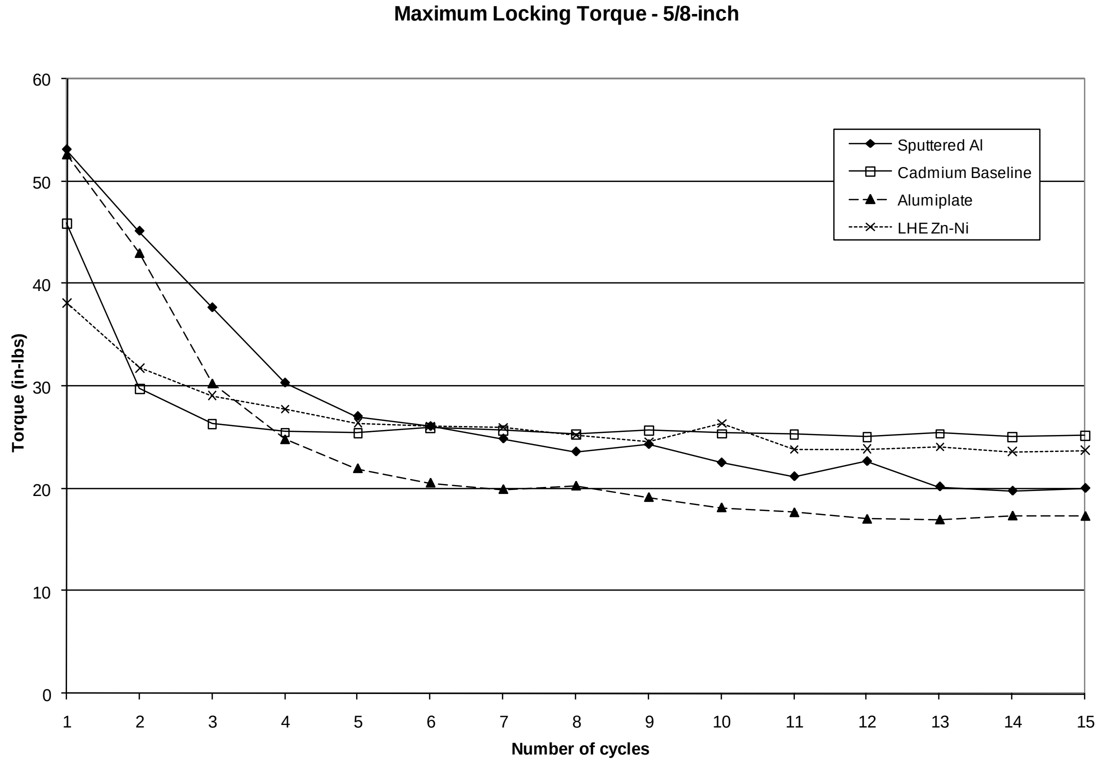


Figure 28. Graph of Maximum Locking Torque for 5/8-inch Fasteners

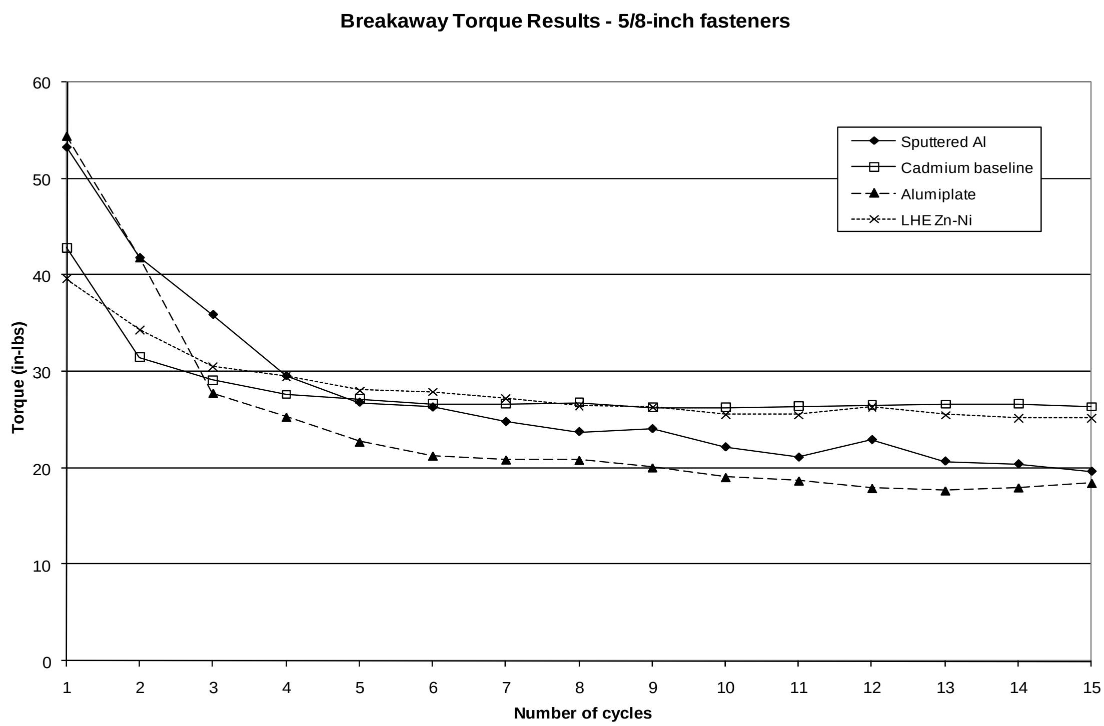


Figure 29. Graph of Breakaway Torque for 5/8-inch Fasteners

Figures 26 and 27 represent the testing performed on the 3/8-inch fasteners. All coatings met both the maximum locking and breakaway torque acceptance criteria. The locking torque results stabilized for all coatings after approximately the fifth cycle with the LHE Zn-Ni having the lowest locking torque values and the sputtered Al coating had the highest. Also, the shapes of the curves and trends for locking torque closely matched the curves for breakaway torque, with the only difference being that the breakaway torque values were lower than the locking torque values, which was expected.

For the 5/8-inch fasteners, the Cd baseline and the LHE Zn-Ni had the highest maximum torque readings, once the readings stabilized at cycle 6. All results met the acceptance criteria of less than 300 in-lb. The breakaway torque readings, however, did not meet the acceptance criteria of greater than 32 in-lb. All coatings were less than 32 in-lb of torque after the second cycle, with the Cd baseline and LHE ZN-Ni having consistent results between 25 and 30 in-lb of torque.

3.4.2 Torque Tension

This test measures the torque-tension during installation of threaded parts to achieve specified clamp-up force. Torque-tension is measured with SAE AMS 2518 (*Thread Compound, Anti-Seize*, *Graphite-Petrolatum*, revised July 2001).

Test Description

The coating was applied as recommended by the manufacturer, to the same thickness class as the original coatings. A representative test fixture to measure torque-tension is shown in Figure 30. The test nut, bolt, and washers were tested with SAE AMS 2518 (*Thread Compound, Anti-Seize*, *Graphite-Petrolatum*, revised July 2001).

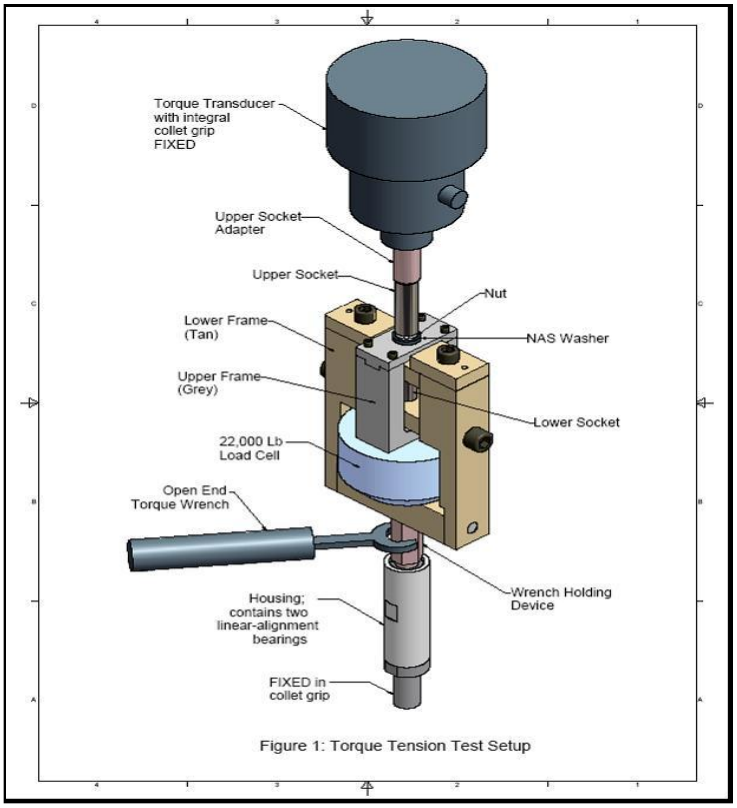


Figure 30. Representative Torque Tension Test Fixture

The nut was assembled onto the bolt so that a minimum of one complete thread extended beyond the top of the nut. The torque-tension was measured using the recommended torque transducer and force washer. The torque and induced load was recorded for the range of 30% to 60% of the ultimate tensile strength (UTS) of the bolt (3,435-6,870 lb for -06032 bolts, 10,239-20,478 lb for -10032 bolts).

Each assembly was tested for a total of five cycles. A test cycle included wrenching nut onto bolt until the desired preload was achieved, then completely removing nut. Any loosened particles were blown off using 60 psi compressed air, if necessary, between cycles. The mean and standard deviation of the test result were recorded and results were plotted.

Rationale

This test is a screening test necessary for comparing the torque-tension values of candidate coated threaded parts to Cd coated threaded parts.

Test Methodology

Parameters	Room temperature (68–78°F), installation torque range of 50–75 in-lb
Type/Number of Specimens	Test Bolts/Nuts: NASM21250-06032/ NAS1804-6 (alloy steel, candidate coated, 3/8 inch), five (5) specimens Test Bolts/Nuts: NASM21250-10032/ NAS1804-10 (alloy steel, candidate coated, 5/8 inch), five (5) specimens Test Washers: NASM14155-6 or NASM14155-10 (alloy steel, candidate coated) two (2) per test specimen
Experimental Control Specimens	Test Bolts/Nuts: NASM21250-06032/ NAS1804-6 (alloy steel, Cd coated, 3/8 inch), five (5) specimens Test Bolts/Nuts: NASM21250-10032/ NAS1804-10 (alloy steel, Cd coated, 5/8 inch), five (5) specimens Test Washers: NASM14155-6 or NASM14155-10 (alloy steel, Cd coated) two (2) per test specimen
Acceptance Criteria	Torque-tension for candidate material is within the range for Cd plated threaded parts. Threaded part does not yield or fracture, threads do not strip.
Reference Document	MIL-STD-870B, AMSQQP416

Data Analysis

Record values during preload and plot the resulting load versus torque. Data should be linear in the elastic range. Compare to Cd control plots. Values for candidate should be within the torque range given for Cd.

Test Results

Representative graphs are listed as Figures 31 through 34 for the torque tension, load versus torque curves for the 3/8-inch fasteners. Each graph contains data for 5 cycles of loading and then completely removing the nut. Visual observation provided by WMTR was that there was no loose/peeling coating or stripping of the threads on the bolts during testing.

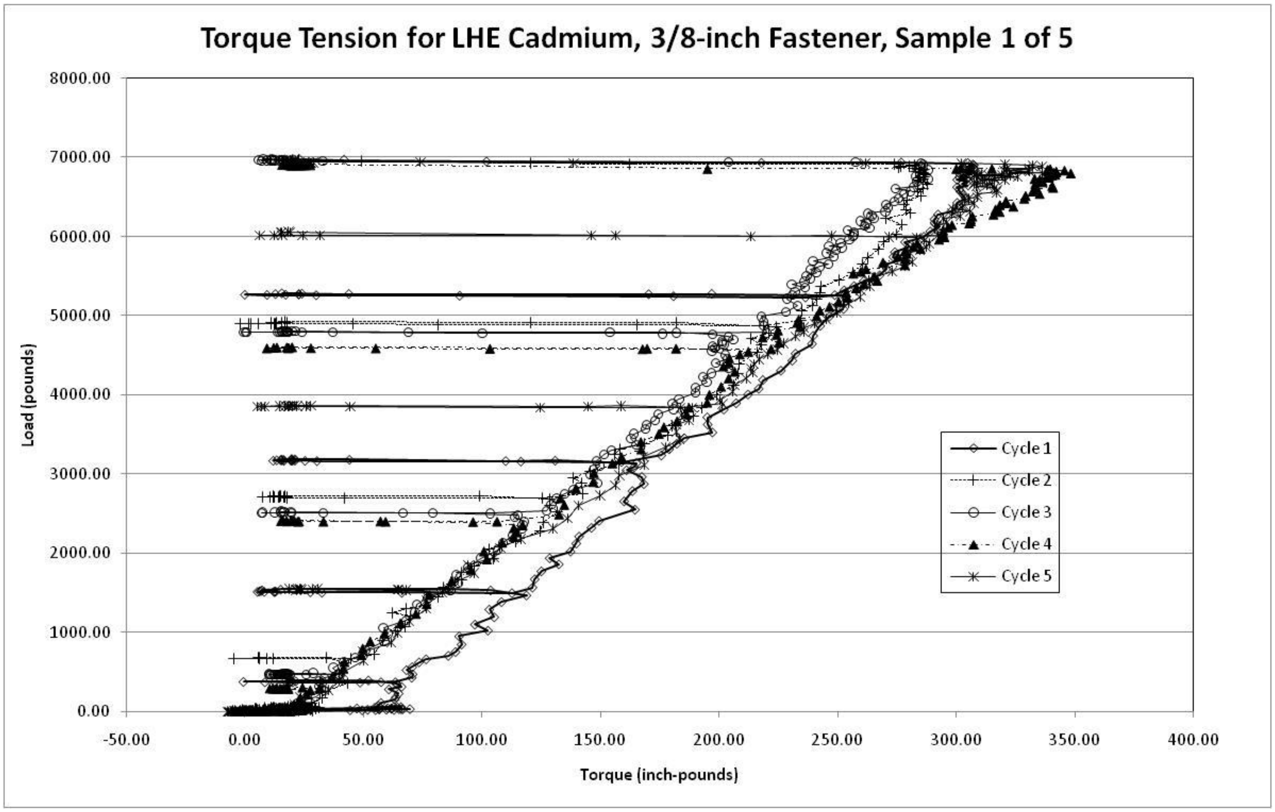


Figure 31. Torque Tension Results for LHE Cd, 3/8-inch Fastener

The load versus torque curves for each of the five cycles for Cd appears to be relatively linear throughout the range. In addition, there was little change in the torque values amongst the cycles, indicating that the coating remained intact and continued to provide adequate lubricity throughout the five cycle test.

The IVD-Al samples were tested separately, at a later date, than the rest of the samples. The load versus torque curves prepared by WMTR are listed in Appendix B. To summarize, the curves for the IVD-Al samples showed that, for 4 of 5 samples, the slope of the curves became increasingly steeper, with the maximum achievable torque decreasing per cycle. The maximum torque for Cycle 1 ranged from 1000-1100 in-lbs, while the Cycle 5 results were generally 600-700 in-lbs. This indicates a loss of lubricity, but there was no visible loss of coating. Figure 32 displays the results for the Zn-Ni coated 3/8-inch fasteners.

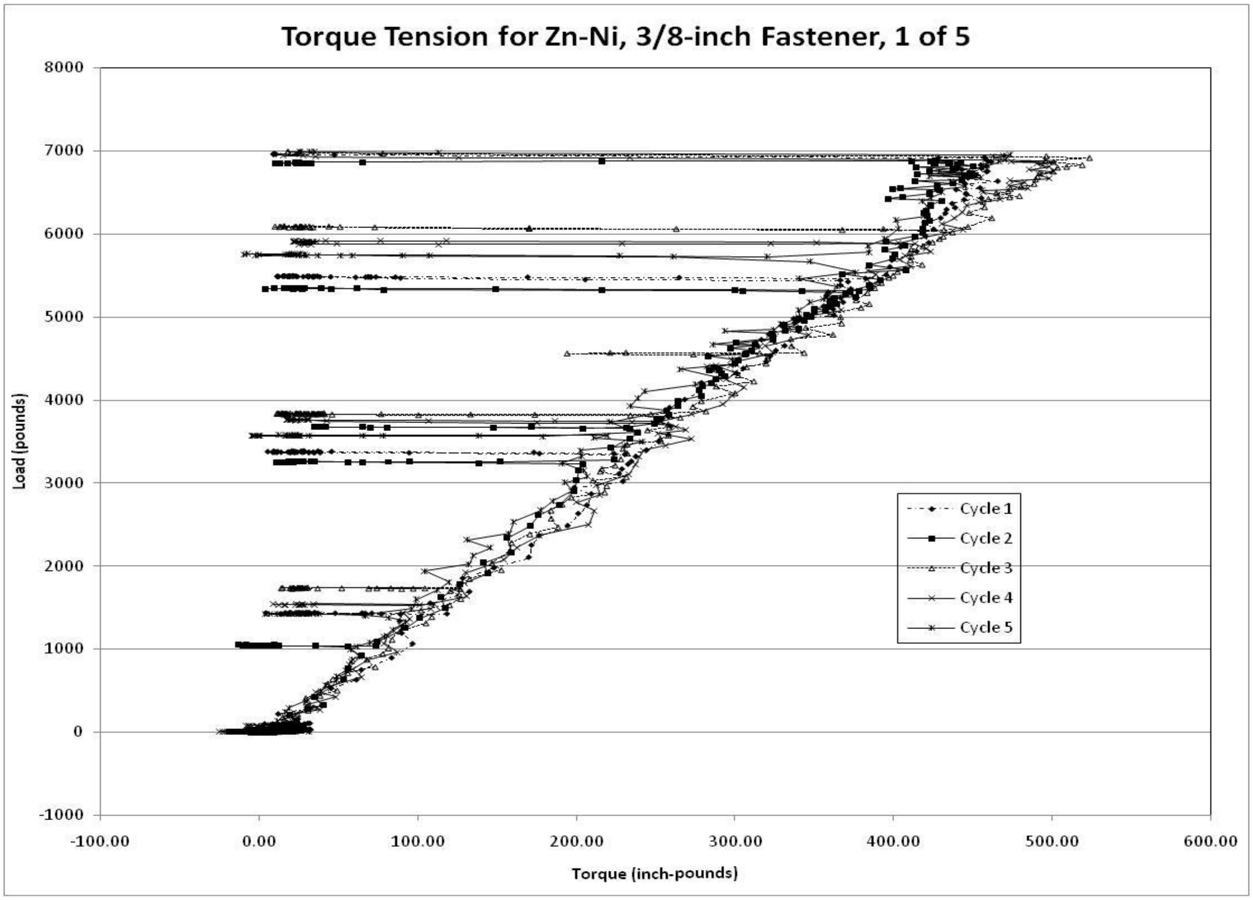


Figure 32. Torque Tension Results for Zn-Ni Coated 3/8-inch Fasteners

The Zn-Ni results are similar to LHE Cd, with little variance in torque values from Cycle 1 to Cycle 5. The maximum torque values were slightly higher than Cd, ranging from approximately 400 - 500 in-lbs, compared to 275 – 350 in-lbs for Cd. Figure 33 contains the curves for electroplated Al on 3/8-inch fasteners.

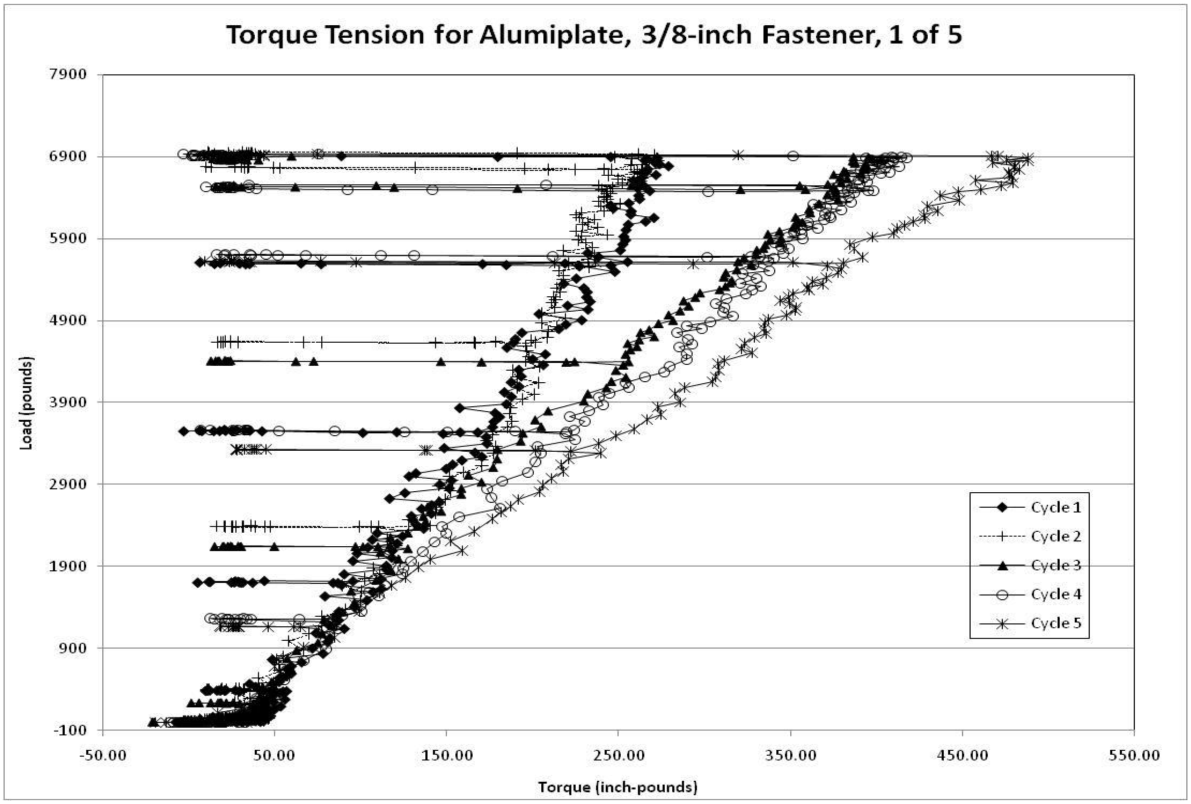


Figure 33. Torque Tension Results for 3/8-inch Fasteners Coated with Electroplated Al

For the electroplated Al samples, the torque values increased with the additional cycles, indicating that the electroplated Al actually increases in lubricity through wear. Again, all curve are relatively linear during the loading cycle. Figure 34 shows the results for sputtered Al-coated 3/8-inch fasteners.

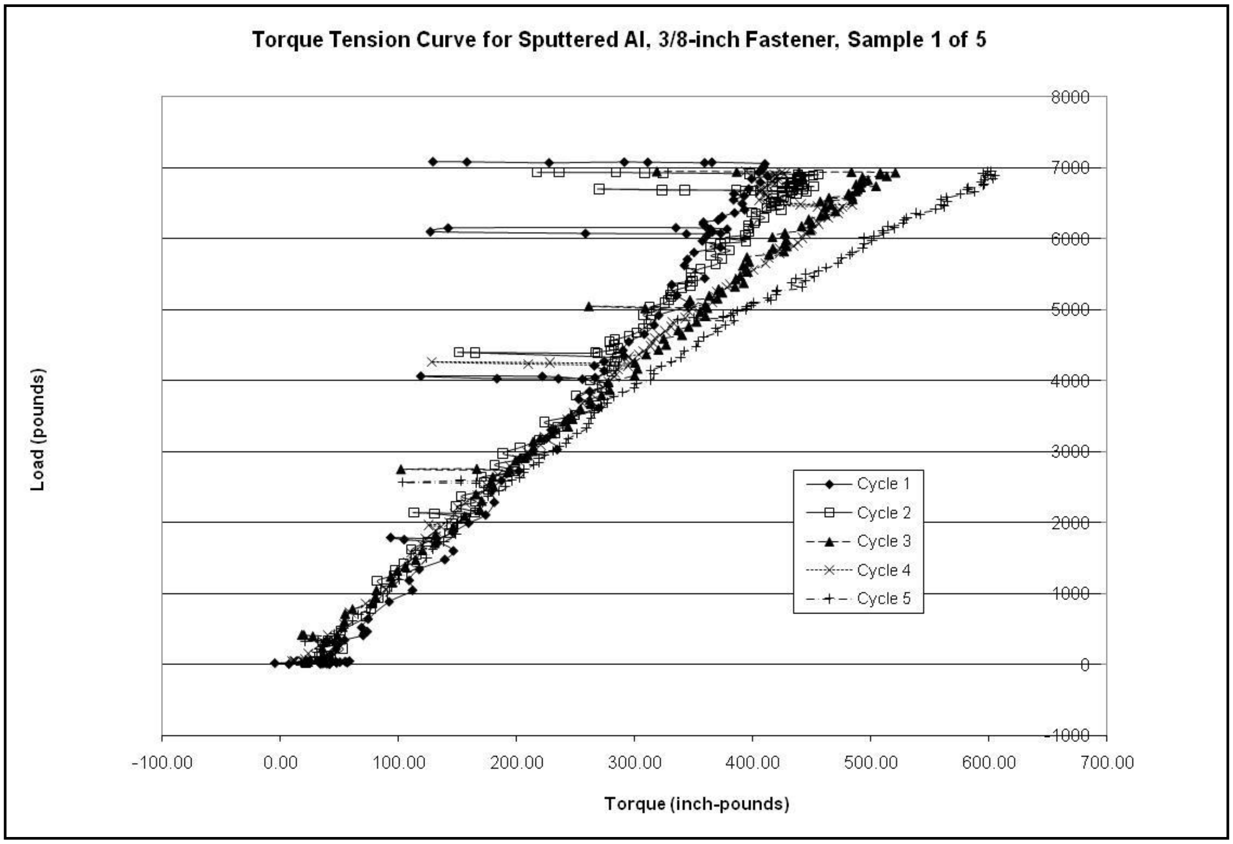


Figure 34. Torque Tension Results for 3/8-inch Fasteners Coated with Sputtered Al

The curves for sputtered Al show the same trend as the electroplated Al, with torque values increasing during loading from Cycle 1 to Cycle 5. The maximum torque values achieved for sputtered Al are slightly higher than electroplated Al, ranging from 350 – 600 in-lbs, as opposed to 200 – 475 in-lbs for electroplated Al. The next series of graphs (Figures 35 – 38) represent the results achieved for the 5/8-inch fasteners. The results for LHE Cd are listed in Figure 35.

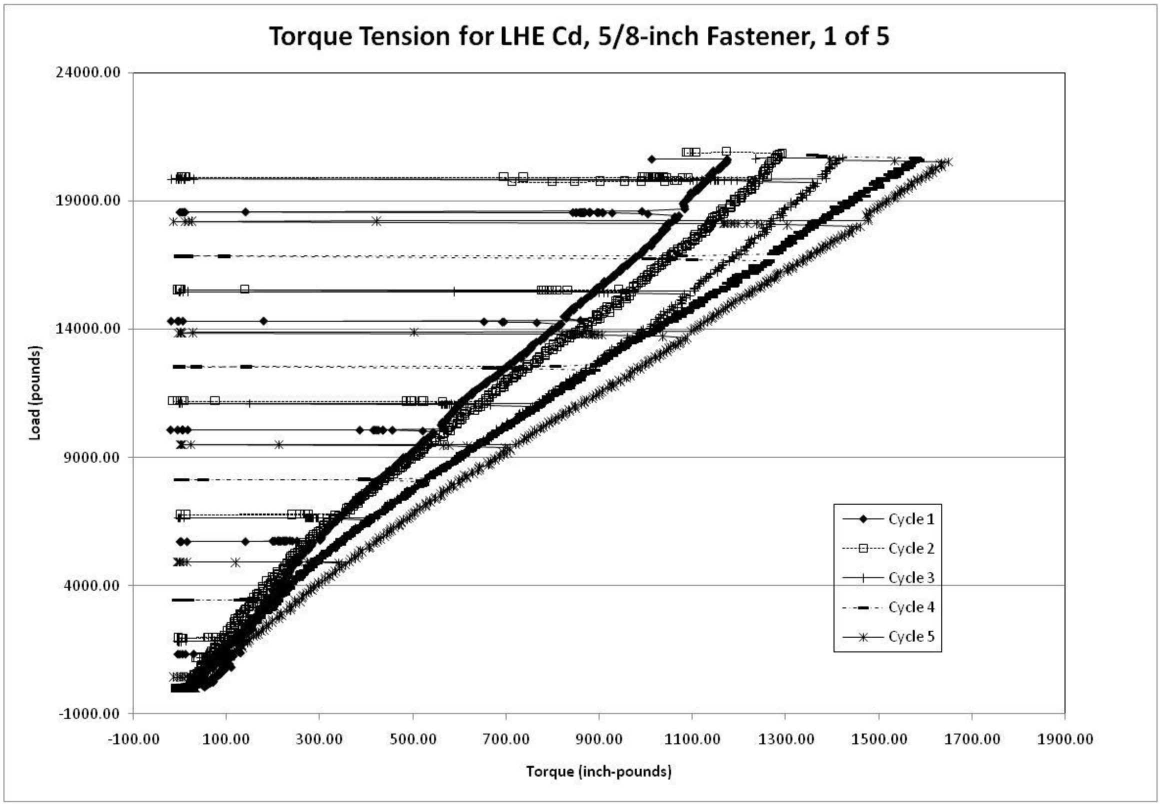


Figure 35. Torque Tension Results for LHE Cd-coated 5/8-inch Fastener

The results for the 5/8-inch fastener coated with Cd show a self-lubricating trend, where the load versus torque curves increase over the cycles. Again, as with the 3/8-inch fasteners, the 5/8-inch fasteners coated with IVD-Al were tested at a later date, and WMTR generated the curves, located in Appendix B. The trend was the same as the 3/8-inch fasteners, with a decrease in torque versus load from Cycle 1 to Cycle 5. Figure 36 contains a representative graph for the Zn-Ni coated fasteners.

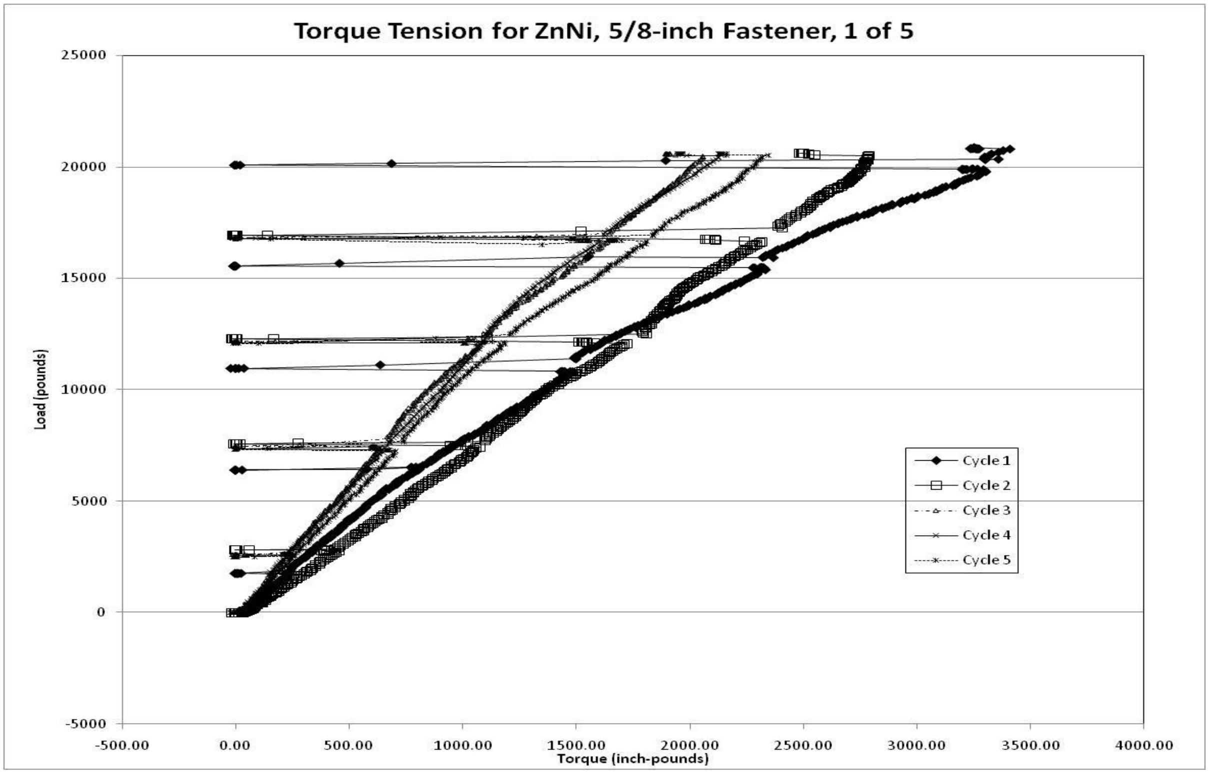


Figure 36. Torque Tension Results for 5/8-inch Fastener Coated with ZnNi

Again, the results for the 5/8-inch fastener have a similar trend as the 3/8-inch fastener, with the torque versus load decreasing for each cycle. Figure 37 shows a graph of a 5/8-inch fastener coated with electroplated Al.

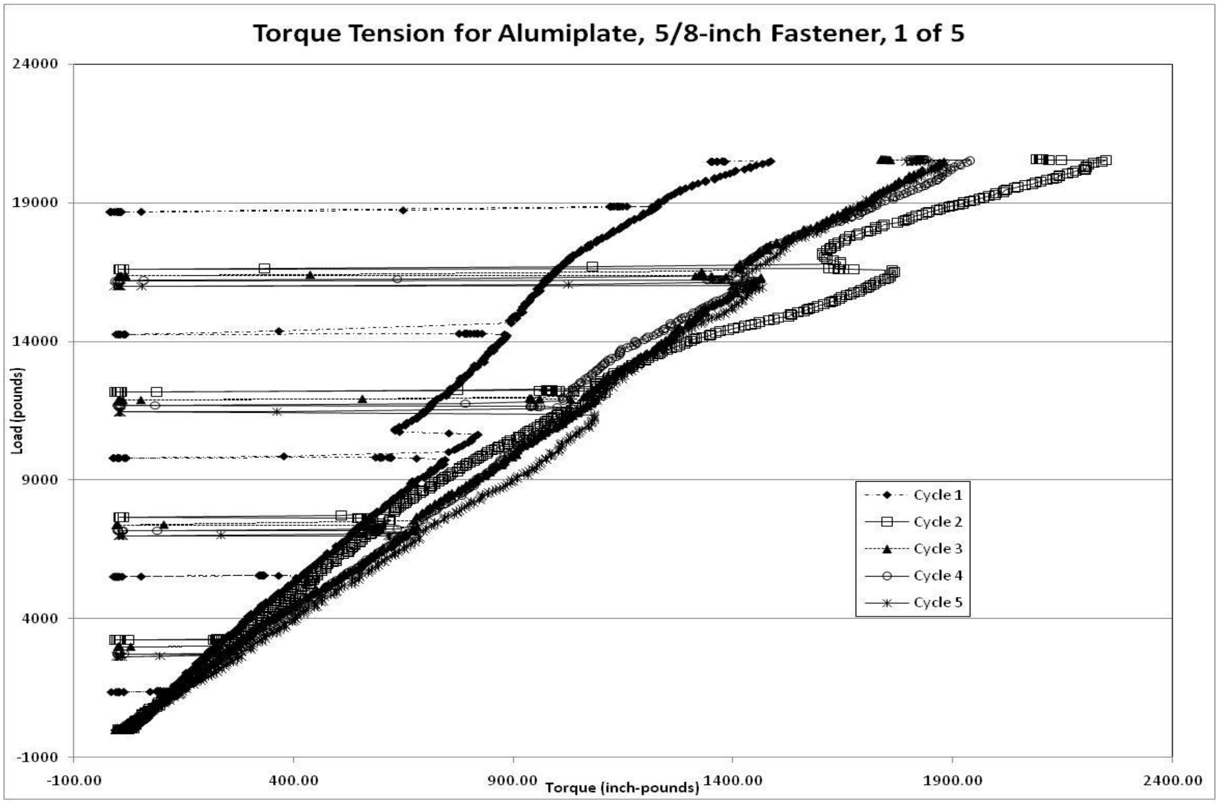


Figure 37. Torque Tension Results for 5/8-inch Fastener Coated with Electroplated Al

This curve does not have a clear trend, showing a lower load versus torque curve for Cycle 1, then the highest torque readings for Cycle 2, with the last three cycles having curves between the first two. In addition, the curves are not as linear as the 3/8-inch fasteners. Finally, Figure 38 contains a graph of the sputtered Al torque tension results.

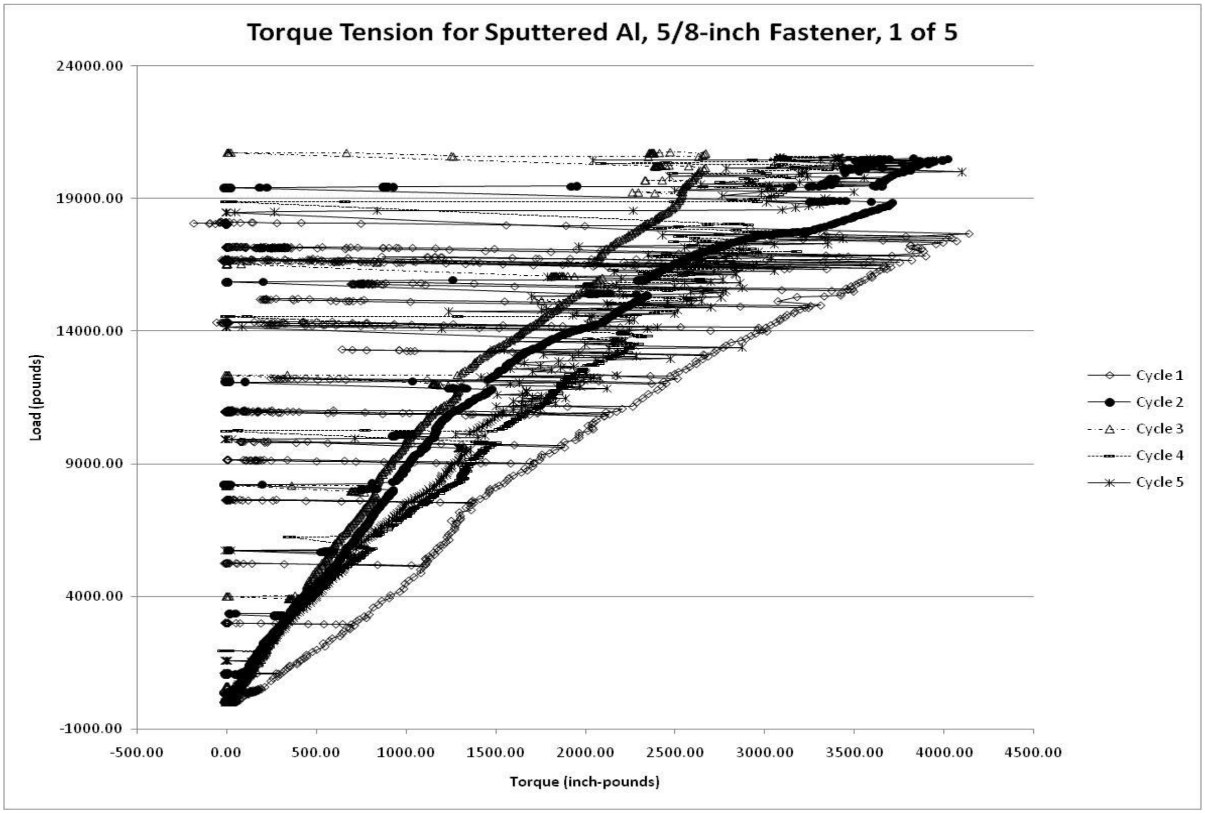


Figure 38. Torque Tension Results for 5/8-inch Fastener Coated with Sputtered Al

The results for the 5/8-inch fasteners for sputtered Al show many more drop points on the curves than the previous samples. Also, the trend is not clearly defined, similar to the electroplated Al results. For this particular fastener, there appeared to be a great deal of scatter to the Cycle 4 and Cycle 5 curves, when the torque values exceeded 1500 – 2000 in-lbs. There does appear to be more of an effect on the coating when conducting torque tension testing on the 5/8-inch fasteners as opposed to the 3/8-inch fasteners.

Overall, the torque tension results for the 3/8-inch fasteners show a trend, with LHE Zn-Ni performing similar to LHE Cd, where the load versus torque curves showed decreasing values over time. The Al coatings had the reverse trend, where, the load versus torque curves had higher torque values for each subsequent cycle. This same trend was not present for the 5/8-inch fasteners. The scatter in torque values between cycles was much greater for each coating. LHE Cd performed the same for the 5/8 and 3/8-inch fasteners, with decreasing torque values, while LHE Zn-Ni had the same trend for Cycles 1 and 2, then Cycle 3 had lower values than 4 and 5. The load versus torque curves for the Al coatings were not as linear as the 3/8-inch fasteners, and the torque values for each cycle varied, not showing a trend relating number of cycles with effect on the coating.

3.4.3 Torque Tension for Corrosion Exposed Fasteners

At the beginning of this testing program, a test facility could not be located by *CTC* to perform this testing (a number of no-bid responses were received). More recently, *CTC* has developed the capability in-house to conduct this testing. *CTC* has the coated fasteners to conduct the testing, if desired by the JCAT, as part of a future (Phase III) effort.

3.5 Hydrogen Embrittlement and Re-Embrittlement

HE of metals is a critical performance characteristic for high-strength steels. The term "hydrogen embrittlement" refers to hydrogen dissolved in the metal and characterized by delayed brittle failure of components under stress. The hydrogen is introduced into the component during application of coatings (electroplated coatings in particular). This hydrogen can be removed by baking the component in an oven soon after coating application. The permeability of the coating to hydrogen determines the success of the bake out operation. The process by which hydrogen is introduced into a component as a result of interaction of the component or its coating with the operating environment is often referred to as "hydrogen re-embrittlement" although other terms are also used (e.g., environmentally induced cracking).

There are several methods for testing the state of "embrittlement" and "re-embrittlement" with no clear consensus as to the best method, particularly in the case of "re-embrittlement". The method used here is the standard sustained load test (SLT) as described in ASTM F519 for HE and ASTM F1624 Incremental Step Loading Technique for HRE. It is recommended that processes be evaluated for HE first. Those that can produce acceptable hardware during manufacturing can then be tested for the effects of the operating environment to cause "re-embrittlement".

3.5.1 Hydrogen Embrittlement – Reproducibility

This test was performed IAW ASTM F519 (*Standard Test Method for Mechanical H-E Evaluation of Plating Processes and Service Environments E (1998)*, issued May 10, 1997). HE testing was conducted in Phase II as a quality assurance procedure to ensure reproducibility with Phase I results. In this test ASTM F519 Type 1a.1, 4340 high strength steel notched round bars were loaded in tension (in air) for an extended period of time to determine whether the coating process was embrittling. Each coated bar was loaded for 200 hours at 75% of the NFS established for uncoated bars of the same lot. The tensile load was subsequently increased stepwise by 5% per hour until fracture. Four replicates of each coating were tested in Phase II, except for LHE Zn-Ni. The acceptance criteria for this test were the NFS of bare and coated bars be within 10 ksi of the average reported by the manufacturer for bare bars, and four of four bars sustain 75% NFS for 200 hours without fracture; OR only one of four bars fracture in less than 200 hours and the remaining three sustain at least one hour at 90%. The constant rate tensile pull test, used to determine whether the coated test bars fracture within 10 ksi of bare bars as reported by the manufacturer, has generally not been conducted industry-wide in the past and was not conducted in this effort.

<u>Rationale</u>

It is known that the application of some metallic coatings to high-strength steels such as SAE 4340 at the strength levels commonly used for landing gear can induce HE. The JTP participants agreed that this test is necessary to qualify candidate coatings. ASTM F519 has been the aerospace industry standard for testing for HE since its original release in 1977. Since that time, the USAF, the Boeing Company and the aerospace industry have typically used the Type 1a.1 specimen and SLT method. As a result there is a significant historical database for the Type 1a.1/SLT combination.

Test Methodology

Parameters	ASTM E8, bare and coated (Baseline only) ASTM F519: Load to 75% NFS for 200 hours. Incremental Step Load (ISL) 5% NFS steps with one (1) hour dwell to fracture.
Type/Number of Specimens	Four (4) each HS1 IAW ASTM F519 Type 1a.1 for candidate, control and baseline
Experimental Control Specimens	IAW MIL-STD-870B Class 1 Type II (thickness at 0.0005 inches to 0.0008 inches with a supplementary chromate treatment). Baseline: Bare, un-coated Type 1a.1 bar Coated ASTM E8 tensile bar
Acceptance Criteria	NFS of bare and coated specimens within 10 ksi of average reported by manufacturer for bare • Four of four specimens sustain 75% NFS for 200 hours SLT without fracture. or • Only one of four specimens fracture in less than 200 hours and the remaining three sustain at least one (1) hour at 90% NFS.
Reference Document	MIL-STD-870B, AMSQQP416

Test Results

Figure 39 contains a photo of the test equipment utilized at NAVAIR, Patuxent River to conduct the HE testing. Table 27 lists the results of the HE test, which include the fracture strength and time to failure. LHE Zn-Ni was not tested.

Figure 39. Hydrogen Embrittlement Test Equipment

Table 26. Hydrogen Embrittlement/Reproducibility Test Results

Coating	Replicate	Fracture Strength (%)	Average Fracture Strength (%)	Time to Failure (hours)	Pass/Fail
Cd-plated	1	93.6%	92.7%	204	Pass
	2	93.7%		204	
	3	90.4%		204	
	4	93.1%		204	
Electroplated Al	1	92.6%	97.3%	203	Pass
	2	100.0%		204	
	3	97.3%		205	
	4	99.1%		205	
Sputtered Al	1	95.2%	97.1%	203	Pass
	2	95.1%		203	
	3	99.0%		205	
	4	99.1%		205	

The sputtered Al coating was tested more extensively in Phase II since the fracture strength values measured in Phase I were lower than expected. It was suspected that the 4340 steel temper had been adversely affected by process heating. To limit the maximum temperature experienced by the round bars, Marshall Labs altered their sputtering parameters (power was reduced from ~8 kilowatt (kW) to 1.6 kW while time was increased from 2.5 hours to 8 hours per Appendix A). As expected, the Phase II HE results in air were much more favorable: 97.1% average strength as compared to only 83.1% in Phase I. All other coatings passed this test according to the JTP criterion outlined above.

3.5.2 Hydrogen Re-Embrittlement – Reproducibility

<u>Test Description</u>

This test was used for screening purposes only and followed the procedures described in ASTM F1624 (*Standard Test Method for Measurement of Hydrogen Embrittlement Threshold in Steel by the Incremental Step Loading Technique*). The same 1a.1 notched test specimens were utilized for this test. Specimens were tested using an enhanced ISL procedure, which had a longer step load of 45% loading for 150 hours, then stepping 5% per hour to failure (Phase I was 24 hours loading at 45%). The test fluids were introduced immediately prior to application of the load.

The following test fluids were used:

- 1 mega ohm reagent water IAW ASTM D1193 Type 2
- Three parts by volume propylene glycol: one part distilled water
- Synthetic sea water IAW ASTM D1141

Qualification Criteria: The candidate coating was considered acceptable if the average load and time to fracture for the candidate was greater than or equal to the control (LHE-Cd) in 1 mega ohm reagent water.

Rationale

It is known that the application of some metallic coatings to high-strength steels such as SAE 4340 at the strength levels commonly used for landing gear can induce HE. In addition, it is known that, due to the sacrificial nature of some metallic coatings, these alloys may become embrittled during exposure to certain substances which can act as electrolytes. The JTP participants agreed that testing of specific substances would over burden the JTP and that each individual user group prior to implementation should perform such testing. Furthermore, the JTP participants agreed that this test would provide a satisfactory comparison with LHE-Cd and indication of the susceptibility of the candidate to re-embrittlement.

Test Methodology

Parameters	ASTM F519 Type 1a.1, 1c or 1e • Load to 45% NFS and hold for 24 hours while wetted with the test fluid. • ISL 5% NFS steps with one (1) hour dwell to fracture while wetted with the test fluid. Test Fluids • 1 mega ohm reagent water IAW ASTM D 1193 Type 2 • Three (3) parts by volume propylene glycol: one part distilled water • Synthetic sea water IAW ASTM D 1141
Type/Number of Specimens	Four (4) each HS1 IAW ASTM F519 Type 1a.1 for candidate, control and test fluid
Experimental Control Specimens	LHE-Cd plated per Fed-Std-MIL-STD-870B Class 1 Type II (thickness at 0.0005 inches to 0.0008 inches with a supplementary chromate treatment).
Acceptance Criteria	Average load and time to fracture greater than or equal to LHE-Cd when tested in 1 mega ohm reagent water.
Reference Document	MIL-STD-870B, AMSQQP416

Test Results

Figure 40 contains a photo of the equipment utilized by NAVAIR PATUXENT River to test for hydrogen re-embrittlement. Figure 40 is followed by Table 27, which lists the results of the testing.

Figure 40. Hydrogen Re-Embrittlement Test Equipment

Table 27. Hydrogen Re-Embrittlement Test Results

Test Phase →			Phase II Results			Phase I Results (1)		
Coating	Test Fluid	Rep.	Fracture Strength (%)	Average Fracture Strength (%)	Time to Failure (hours)	Fracture Strength (%)	Average Fracture Strength (%)	Time to Failure (hours)
LHE Zn-Ni	ASTM D1141 Sea Water	1	60.3%	57.7%	153	45.0%	57.6%	0.7
		2	45.0%		13	65.1%		27.4
		3	80.3% (2)		157	60.0%		26.1
		4	45.0%		< 1	60.2%		26.5
	ASTM D1193 Reagent Water	1	90.5%	93.0%	157	90.3%	70.2%	32.0
		2	95.4%		160	60.3%		26.1
		3	--		--	80.0%		30.1
		4	--		--	50.2%		24.1
Electroplated Al	ASTM D1141 Sea Water	1	99.8%	99.0%	160	Note (4)	93.9%	
		2	98.2%		160 (3)			
	3:1 propylene glycol	1	98.8%	95.8%	160		95.1%	
		2	92.7%		159			
	ASTM D1193 Reagent Water	1	93.2%	95.7%	159		95.0%	
		2	98.2%		157			
Sputtered Al	ASTM D1141 Sea Water	1	45.0%	45.2%	0.1	45.2%	49.0%	0.1
		2	45.2%		0.1	45.1%		0.1
		3	45.0%		0.1	50.0%		24.1
		4	45.4%		0.9	55.5%		25.7
	3:1 propylene glycol	1	90.2%	85.3%	158.9	65.0%	77.4%	27.1
		2	80.5%		136.0	84.4%		31.0
		3	90.0%		158.8	80.0%		30.0
		4	80.4%		156.6	80.1%		30.0

Table 27. Hydrogen Re-Embrittlement Test Results (Continued)

Test Phase →			Phase II Results			Phase I Results (1)		
Coating	Test Fluid	Rep.	Fracture Strength (%)	Average Fracture Strength (%)	Time to Failure (hours)	Fracture Strength (%)	Average Fracture Strength (%)	Time to Failure (hours)
	ASTM D1193 Reagent Water	1	45.1%	48.9%	42.4	45.2%	47.7%	6.3
		2	50.2%		151	50.2%		24.5
		3	45.0%		81.8	50.0%		24.2
		4	55.4%		151.4	45.3%		1.5

Notes: (1) The test profile in Phase I was shorter in duration than Phase II (24 hour vs. 150 hour initial hold at 45%); (2) Sample loaded to 75% NFS in air for 180h, then re-started at 45% in sea water environment; (3) Machine experienced a power failure; the sample remained under load and the test was restarted at ~96 h; (4) Phase I averages provided for Alumiplate represent the average of four specimens for each fluid (all individual specimens were >90%).

Selected HRE tests were performed in Phase II to supplement the existing Phase I data. Some coatings were tested less extensively than others, depending on the number of additional test specimens that were available for this test. Sputtered Al was fully re-tested for re-embrittlement characteristics in Phase II due to the process change which was required to avoid overheating of the ¼ inch diameter round bar specimens (ASTM F519, Type 1a.1). This process change will not be required for full size components, so the data was generated for comparative purposes. While results had improved for HE testing as expected (see previous section), results for HRE were fairly similar to those obtained in Phase I. Average fracture strength for sputtered Al was slightly higher in propylene glycol in Phase II (85.3% vs. 77.4%), slightly lower in synthetic seawater (45.2% vs. 49%), and about the same in reagent water (48.9% vs. 47.7%). In reagent water, two sputtered Al bars failed before the end of the 45% hold in each test phase. In synthetic seawater, the Phase II specimens all fractured within 1 hour at the 45% static load, while two of four specimens in Phase I had also failed within 1 hour, and the other two failed at slightly higher values (50.0% and 55.5% NFS). This test variability seems to represent normal statistical variation.

Consistent with Phase I, re-embrittlement test results for Alumiplate were best in Phase II, with fracture values well into the 90% range for each test fluid environment. In Phase II, LHE Zn-Ni performed better in reagent water (93% vs. 70.2% in Phase I) and about the same in synthetic seawater (~58% average). In the seawater test, two specimens failed prior to 24 hours in Phase II, whereas only one did in Phase I. Tests were not performed in the propylene glycol for LHE Zn-Ni. One interesting note is that the best performing specimen in the seawater test had been mistakenly loaded in air to 75% NFS (180 hours), then re-started at 45% in seawater to complete that dataset. This specimen failed at 80.3% NFS which was the highest of the 8 round bars tested in Phases I/II.

3.6 Reparability

This test evaluates the reparability of the candidate Cd-free coatings with non-Cd repair methods. The test also evaluates the use of Cd-free coatings as the repair coating for damaged Cd-plated hardware. This test is applicable for evaluating candidate repair coatings where the repair technique is done by brush plating.

The initial qualification of the brush plate solutions required verification of the integrity of the repair coating applied on standard specimens. Repair coatings that met the initial qualification were to go on to final qualification. Final qualification required verifying the compatibility of the repair coatings with the substrate and the surrounding plating. The final qualification portion of this procedure was not completed.

Test Description

For initial qualification, the candidate repair coating or Cd repair coating was applied to the bare test specimens. The repair coating was either applied by hand or by automated brush-plating equipment for increased consistency across specimens. If applied by hand, only experienced brush-plating operators should have been utilized. After the test specimens had been coated, and conversion coated if required, initial qualification tests began.

For final qualification, the candidate coating or Cd coating was to be applied to the test specimens. Then a bare area was to be generated on each specimen by manually abrading or machining and abrasive blasting the coating down to the substrate. Bare areas on the specimens would then be repaired by brush plating with the candidate repair material on candidate coated and Cd-coated test specimens and with Cd repair material on Cd-coated control specimens by experienced operators. The success of the repair of any damaged metal coating on a part is heavily dependent upon the proficiency of the operator performing the repair. The repair plating may be automated for increased consistency across specimens. After the repair coating had been applied to the test specimens, and conversion coated if required, final qualification testing was to have been conducted.

Rationale

Techniques must be available to repair scratches, gouges, worn areas and voids in the coating to return the hardware to the original design configuration and meet all acceptance criteria of this test plan. Brush plate repair of metal coatings has been successfully used to restore mis-machined parts, scratches, gouges and worn plating or bare spots on parts to drawing requirements.

Test Methodology

Initial Qualification –

Parameters	Apply brush plate repair by experienced operator onto standardized test specimens.
Type/Number of Specimens	Candidate repair coating on bare 4130 steel and 4340 steel test specimens. Appearance: 4 inches x 6 inches 4130 specimens - 1 ea* Adhesion: 1 inch x 4 inches 4130 specimens - 3 each Thickness: 1 inch x 4 inches 4130 specimens - 1 each Unscribed Corrosion Resistance: 4 inches x 6 inches 4130 specimens - 3 each* Scribed Corrosion Resistance: 4 inches x 6 inches 4130 specimens - 3 each* Hydrogen Embrittlement (No Bake): ASTM F519, Type 1a.1, 4340 - 4 each
Experimental Control Specimens	LHE-Cd plated per Fed-Std-MIL-STD-870B
Acceptance Criteria	Repair performance meets or exceeds performance of experimental control specimens.
Reference Document	MIL-STD-870B, AMSQQP416

Note: * = Conversion Coated

Final Qualification – NOT PERFORMED

Parameters	Manually scribe and abrade coating to substrate. Brush plate repair by experienced operator.
Type/Number of Specimens	Candidate repair coating on bare 4130 steel and Cd-coated 4130 steel (2 sets total). Appearance: 4 inches x 6 inches 4130 specimens - 1 ea* Adhesion: 1 inch x 4 inches 4130 specimens - 3 each Thickness: 1 inch x 4 inches 4130 specimens - 1 each Unscribed Corrosion Resistance: 4 inches x 6 inches 4130 specimens - 3 each* Scribed Corrosion Resistance: 4 inches x 6 inches 4130 specimens - 3 each* Paint Adhesion: 4 inches x 6 inches specimens (PRIMER + TOPCOAT) - 3 each, 3 each*
Experimental Control Specimens	LHE-Cd plated per Fed-Std-MIL-STD-870B
Acceptance Criteria	Repair performance meets or exceeds performance of experimental control specimens.
Reference Document	MIL-STD-870B, AMSQQP416

3.6.1 Appearance

Repair coatings were evaluated for appearance and held to the same requirements as the primary coatings. Brush plated Cd applied by Boeing was selected as the baseline coating for comparison. Two of the three candidate repair coatings, as well as the baseline coating, were given a pass rating. The brush plated Sn-Zn coating was given a “fail” rating, due in part to the observation of a dark brown area through the center of the panel. The observations from the appearance evaluation of the repair coatings are located in Table 28.

Table 28. Appearance of Repair Coatings

Coating	Appearance Results
Brush Plated Cd (Baseline) - Boeing	Coating is continuous but not uniform, showing swirls from processing; coating is smooth, adherent, and free from blisters, pits, excessive powder, and contamination
Brush Plated Zn-Ni - Boeing	Coating is not continuous or uniform; coating is adherent, but rough, with excessive powder and possible rust spots
Brush Plated Sn-Zn - Boeing	Coating is continuous but not uniform, with a dark brown area through the center of the panel; the coating is smooth and adherent, but has excessive powder
Sprayed Al-Ceramic (SermeTel) – Boeing	Coating is continuous and uniform, smooth, adherent, and free from pits, blisters, excessive powder, and contamination

3.6.2 Bend Adhesion

Bend adhesion testing was also conducted in the same manner as the primary coatings, by clamping the specimen into a vice and bending the free end back and forth (one cycle) until failure of the coating or substrate occurs. The results of bend adhesion testing of the repair coatings are listed in Table 29. In addition, Figure 41 contains representative photos of the test specimens after testing.

Table 29. Bend Adhesion Results for Repair Coatings

Coating	Replicate	Cycles to Fracture	Comments	Pass/Fail
Brush Plated Cd	1	2-3	Significant coating adhesion loss after 1.5-3 cycles	Fail
	2	2		
	3	2-3		
Brush Plated Zn-Ni	1	13	Coating cracks at 8-9 cycles	Pass
	2	11		
	3	12		
Brush Plated Sn-Zn	1	10		Pass
	2	17		
	3	14		
Al-Ceramic (SermeTel)	1	2	Coating failure across full width of 2 specimens	Fail
	2	2-3		
	3	3		

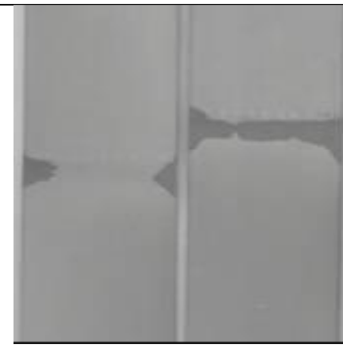
			
Brush Plated Cd	Brush Plated Zn-Ni	Brush Plated Sn-Zn	Al-Ceramic (SermeTel)

Figure 41. Representative Photos of Bend Adhesion Results for Repair Coatings

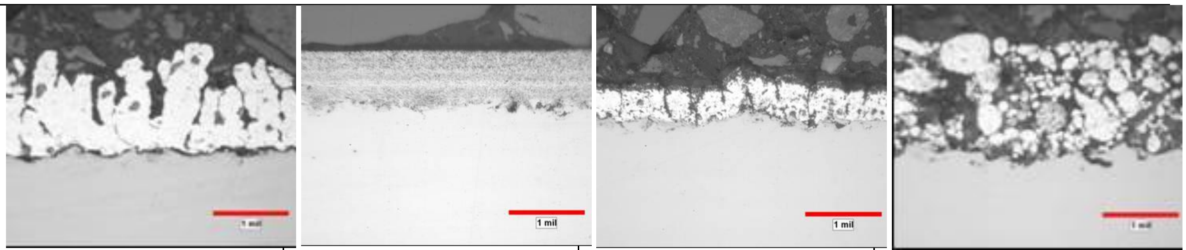
Bend adhesion test results for the repair coatings were similar to those obtained in Phase I (again only tested on low alloy 4130 steel). Brush plated Cd exhibited significant adhesion loss after 1.5 bend cycles (second tensile cycle for the coating). Application of a sharp blade easily removed most of the coating. Brush Sn-Zn performed well on all specimens. One representative panel is shown in Figure 41. The Brush Zn-Ni panels performed well on all specimens, with only slight coating removal at the edges of the specimens. The bulk of the area did not have visually apparent cracks developing until 8-9 bend cycles, and the deposit was adherent when challenged with a blade after substrate failure. The SermeTel coating exhibited adhesion failure after 2-3 cycles. Light brushing with a fingernail removed the coating across the full width of one specimen out of three, while the other two exhibited removal closer to the edges (pictured in Figure 41).

3.6.3 Thickness

Coating thickness was measured by cross-sectioning, mounting, and polishing sections of a 1 inch x 4 inches test panel. The thickness was measured at five locations, at a magnification of 1000x. Table 30 contains the results of the thickness measurements with representative photos located in Figure 42.

Table 30. Coating Thickness Results for Repair Coatings

Coating	Thickness Measurements					
	Reading 1	Reading 2	Reading 3	Reading 4	Reading 5	Average
Brush Plated Cd	1.54 mil	1.57 mil	1.28 mil	1.14 mil	1.19 mil	1.34 mil
Brush Plated Zn-Ni	1.16 mil	0.93 mil	0.78 mil	0.79 mil	0.77 mil	0.89 mil
Brush Plated Sn-Zn	0.48 mil	0.53 mil	0.57 mil	0.51 mil	0.41 mil	0.50 mil
SermeTel	1.47 mil	1.37 mil	1.51 mil	1.45 mil	1.28 mil	1.42 mil



Brush Plated Cd	Brush Plated Zn-Ni	Brush Plated Sn-Zn	SermeTel

Figure 42. Cross-sectional Images of Coating for Thickness Measurement, 500X

If plating to the specification, the Sn-Zn coating was within the 0.3 – 0.5 mil thickness requirement. All other coatings exceeded the specification.

3.6.4 Unscribed Corrosion Resistance

Table 31 lists the results of the unscribed corrosion resistance test. Panels were prepared and exposed in the same manner as the primary coatings. Test duration was 3000 hours. Photos of the panels at the conclusion of testing are located in Figure 43.

Table 31. Unscribed Corrosion Resistance Results for Repair Coatings

Coating	Panel #	First Appearance of Corrosion/Observation	Time of Termination/Observation	Rating at Test Termination
Brush Plated Cd	1	No rust	3000 hours/ No rust	10
	2	No rust	3000 hours/ No rust	10
	3	No rust	3000 hours/ No rust	10
Brush Plated Zn-Ni	1	No rust	3000 hours/ Sacrificial coating breakdown/ no rust	9
	2	No rust	3000 hours/ Sacrificial coating breakdown/ no rust	9
	3	No rust	3000 hours/ Sacrificial coating breakdown/ no rust	9
Brush Plated Sn-Zn	1	72 hours/Chromate depletion	3000 hours/ Chromate depletion and pin holes	8
	2	72 hours/Chromate depletion	3000 hours/ Chromate depletion and pin holes	8
	3	72 hours/Chromate depletion	3000 hours/ Chromate depletion and pin holes	8

Table 31. Unscribed Corrosion Resistance Results for Repair Coatings (Continued)

Coating	Panel #	First Appearance of Corrosion/Observation	Time of Termination/Observation	Rating at Test Termination
Al-Ceramic (SermeTel)	1	24 hours/ Pinhole rust spots through coating	500 hours/ Excessive rust	0
	2	24 hours/ Pinhole rust spots through coating	500 hours/ Excessive rust	0
	3	24 hours/ Pinhole rust spots through coating	500 hours/ Excessive rust	0

Brush Plated Cd (3000 hours)	Brush Plated Zn-Ni (3000 hours)	Brush Plated Sn-Zn (500 hours)	Al-Ceramic (SermeTel) (500 hours)

Figure 43. Photos of Unscribed Corrosion Resistance Panels for Repair Coatings

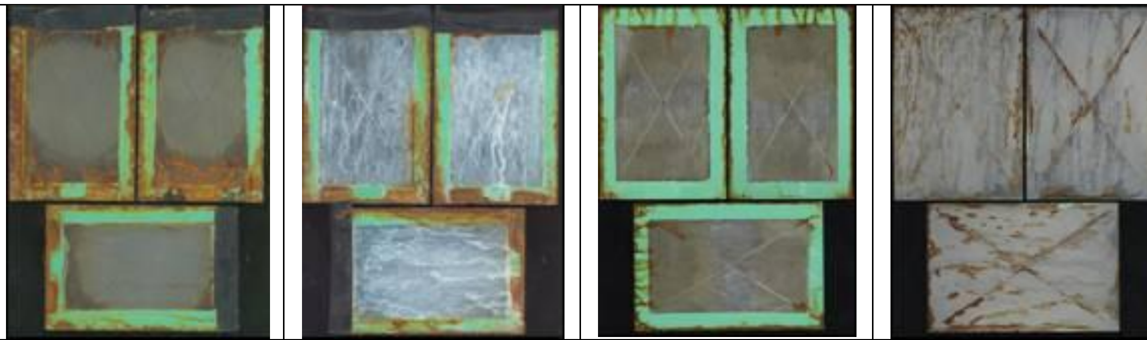
The brush plated metallic coatings had results similar to the primary coatings, with little to no rust present and breakdown of the sacrificial coating during the test. The spray-applied SermeTel coating performed similar to sputtered Al and some of the IVD-Al panels, with early rust formation causing test termination at approximately 500 hours.

3.6.5 Scribed Corrosion Resistance

Table 32 lists the results of the scribed corrosion resistance evaluation of the repair coatings. These test panels were prepared and exposed in the same manner as the panels tested with primary coatings. In addition, Figure 44 contains photos of the panels after testing.

Table 32. Scribed Corrosion Resistance Test Results for Repair Coatings

Coating	Panel #	First Appearance of Corrosion/Observation	Time of Termination/Observation	Rating at Test Termination
Brush Plated Cd	1	No rust	3000 hours/ No rust	9
	2	No rust	3000 hours/ No rust	9
	3	No rust	3000 hours/ No rust	9
Brush Plated Zn-Ni	1	No rust	3000 hours/ Sacrificial coating breakdown/ no rust	9
	2	No rust	3000 hours/ Sacrificial coating breakdown/ no rust	7
	3	No rust	3000 hours/ Sacrificial coating breakdown/ no rust	7
Brush Plated Sn-Zn	1	168 hours/ Rust in scribe	500 hours/ Excessive rust	9 at scribe
	2	168 hours/ Rust in scribe	500 hours/ Excessive rust	9 at scribe
	3	168 hours/ Rust in scribe	500 hours/ Excessive rust	8 at scribe
Al-Ceramic (SermeTel)	1	24 hours/ Pinhole rust spots through coating	500 hours/ Excessive rust	0
	2	24 hours/ Pinhole rust spots through coating	500 hours/ Excessive rust	0
	3	24 hours/ Pinhole rust spots through coating	500 hours/ Excessive rust	0



Brush Plated Cd (3000 hours)	Brush Plated Zn-Ni (3000 hours)	Brush Plated Sn-Zn (500 hours)	Al-Ceramic (SermeTel) (500 hours)

Figure 44. Photographs of Scribed Corrosion Resistance Panels for Repair Coatings

The results for the scribed panels are very similar to the results for the unscribed panels, with the exception of the Sn-Zn panels. These panels developed rust in the scribe and were pulled from testing due to excessive rust at 500 hours.

3.6.6 Hydrogen Embrittlement – Reproducibility

HE testing was conducted once again as a quality assurance test for comparison with Phase I results. Testing was conducted in the same manner as the primary coatings.

Table 33. Hydrogen Embrittlement/Reproducibility Test Results for the Repair Coatings

Coating	Replicate	Fracture Strength (%)	Average Fracture Strength	Time to Failure (hours)	Pass/Fail
Brush plated Cd	1	95.1%	96.3%	203	Pass
	2	95.0%		203	
	3	97.5%		204	
	4	97.6%		204	
Brush plated Zn-Ni	1	92.5%	93.8%	203	Pass
	2	93.0%		203	
	3	94.6%		203	
	4	94.9%		203	
Brush Plated Sn-Zn	1	100.0%	98.1%	205	Pass
	2	96.1%		205	
	3	97.1%		205	
	4	99.3%		205	
Al-Ceramic (Sermetel)	1	97.6%	96.0%	204	Pass
	2	94.9%		203	
	3	95.1%		204	
	4	96.4%		204	

For the repair coatings, brush Zn-Ni performed better than in Phase I where one bar had failed at a strength <90%. All four bars tested in Phase II passed with average fracture strength of 93.8%. The brush Sn-Zn coating had the highest average strength at 98.1%, while the brush Cd and SermeTel 249 coatings were 96.3% and 96.0%, respectively.

4.0 CONCLUSIONS

The conclusions for the results from each of the testing methods are listed in this section, divided between primary coatings and repair coatings. The conclusions list how the coatings performed in each test, as well as which coatings met the acceptance criteria established for each testing method.

4.1 Primary Coatings

This section describes the results for the primary coatings.

Throwing Power: One of the three Cd samples showed thinning of the coating, with iron detected by the SEM during the scans for changes in concentration across the panels. The orientation of the Cd panels in the bath during the test was not provided. For the electroplated Al samples that were horizontal and vertical, with open end facing downward, the concentrations of the main constituents in the remained fairly uniform. However, the sample oriented in the vertical position, with the open end of the fixture facing upward, had a dramatic loss in Al concentration beginning at reading number 8 and extending to the end of the panel, losing almost 25% Al over this span. In addition, the nickel concentration increased by over 30% in this same measurement range. The vendor stated that they deposited the Al over a nickel strike bond layer, showing that the Al coating did not “throw” over the length of the panel, getting much thinner and potentially porous over the last 1.0 – 1.5 inches of the panel. For the LHE Zn-Ni panels, the concentration of zinc remained uniform, regardless of orientation.

Stripability: The sputtered and electroplated Al coatings, as well as the LHE Zn-Ni coating, were able to be removed chemically from the high strength steel bars and still permit average fracture strengths of about 97% of the baseline (without any baking step). Cd plated bars passed at an average strength of 89.4% NFS after stripping.

Samples of the alternatives were then sent back to the coating suppliers for re-coating. Reworked bars were not received back for the sputtered Al coating. The specimens re-coated with electroplated Al passed with average fracture strength of 93.6% for 4 bars. Of the specimens re-coated with LHE Zn-Ni, three performed well with an average of 93.0% NFS, while the fourth failed in the threads at 13 hours (75% NFS). Both coatings tested earn ‘Pass’ ratings according to the acceptance criteria for HE, but the LHE Zn-Ni samples failed bend adhesion, which was likely due to the longer strip time used for the LHE Zn-Ni samples.

Bend Adhesion: When evaluated on three different substrate types (alloy steel, stainless steel, and titanium), bend adhesion for each coating was adequate on all substrates except Ti-6Al-4V which showed passing results only with sputtered Al. IVD-Al was not tested on titanium in this effort.

Wet Tape Paint Adhesion: Paint adhesion was excellent for each coating at the 1 day immersion test (ambient temperature). In the elevated temperature wet tape adhesion exposures, LHE Zn-Ni displayed some inconsistent results with 85582-C1 primer. Two test sites on one panel had a high adhesion rating, while the other ten test sites (spread over three different test panels) showed very poor adhesion, so the average result was quite low.

Unscribed Salt Spray Corrosion Resistance: The results listed in the table show that the Cd-plated panels met the specification of showing no rust after 3000 hours salt spray exposure. LHE Zn-Ni also performed well, with breakdown of the sacrificial coating noted after 3000 hours exposure, but no rust formation. The electroplated Al coating began having depletion of the chromate conversion coating at 1500 hours exposure, with some pinhole formation. The appearance of the panels at 3000 hours was the same, indicating that the pinholes did not progress in size during the second half of the test to result in additional red rust.

The two coating systems that developed red rust and were pulled from testing prior to the completion of 3000 hours exposure were two of three IVD-Al panels and the sputtered Al panels. The first appearance of corrosion on the IVD-Al panels varied greatly, from 72 hours to 2000 hours. The sputtered Al panels were very consistent, developing excessive rust within 500 hours, terminating the test. One note, the specification for IVD-Al coatings, SAE AMS 2427, *Aluminum Coating, Ion Vapor Deposition*, indicates that the acceptance criteria for IVD Al c-rrosion resistance is 504 hours. Applied here, two of the three IVD-Al samples would meet this acceptance criterion.

Scribed Salt Spray Corrosion Resistance: The results for the scribed panels are very similar to the results for the unscribed panels. The Cd-plated panels did not exhibit any damage from exposure, as expected. The LHE Zn-Ni had the next highest rating, with some sacrificial coating breakdown but no rust. The electroplated Al followed, but two of three panels had to be pulled prior to 3000 hours due to significant rust. Once again, the IVD-Al and sputtered Al panels had the most significant corrosion present and testing was terminated at 1000 hours or less.

Galvanic Corrosion Resistance: Both the test results and photos indicate that the fixtures performed better in cyclic corrosion than salt spray. In addition, as expected, the bare test washer corroded that most and had the largest changes in conductivity. The Cd-plated test washers only showed a conductivity change for the 17-4PH stainless steel substrate in both the salt spray and cyclic corrosion tests. The alternative coating that had the most changes in conductivity upon salt spray exposure was LHE Zn-Ni. This was followed by electroplated Al and sputtered Al, which both had small changes in conductivity for almost every test washer/test block alloy combination. The exception was electroplated Al on CuBe, which had no change in conductivity. All alternatives had basically no change in conductivity for assemblies exposed to cyclic corrosion.

Fluid Corrosion Resistance: For the baseline Cd-plated specimens, the group of fluids that had caused the largest change in mass was the paint removers. In addition, when checking the appearance of the samples, the paint removers caused the most corrosion/removal of the coating. One other observation for the Cd-plated panels was that the parts washer cleaning compound and the reagent water appeared to remove the conversion coat. Of the alternative coating systems, the electroplated Al appeared to be least affected by the fluids. Slight changes in weight were measured for the 87978 paint remover and peroxide-based paint remover, with visual exams also showing some affect and potentially the removal of the conversion coat. The parts washer cleaning compound also appeared to remove the conversion coat from the electroplated Al.

The alternative coating that was most affected by the fluid immersion was the LHE Zn-Ni. It performed comparably to the Cd-plated specimens, with the paint removers having the most affect on weight and appearance. Mass was also affected by the aerospace equipment cleaning compound and the wheel well cleaning compound. For the sputtered Al coating, the 81294 paint remover caused the coating to flake off of the panels. Changes in weight resulted from immersion in water-saturated 87257 lubricant, reagent water, and deicing fluid. Overall, it appears that the alternative coatings were comparable to the fluid immersion results of the Cd-plated and IVD-Al baseline panels.

<u>Scribed and Painted Corrosion Resistance</u>: The corrosion resistance results for the painted panels were similar to the results for the unpainted panels. Again the Cd-plated panels performed the best, with only rust in the scribed area after 3000 hours of exposure, resulting in a "9" rating. As with the unpainted panels, LHE ZN-Ni followed with three panels having a "9" rating after 3000 hours exposure. The other 2 panels were rated at 4 and 5 after 3000 hours due to pinhole rust and some creepage from the scribe.

The electroplated Al panels were the next to follow in rank of performance. These panels were all exposed for 3000 hours, with final ratings of 0, 1, 3, and 5, due to various levels of creepage of corrosion from the scribe. The sputtered Al panels were all pulled from testing after 1500 hours due to excessive rust formation. The first signs of corrosion developed after 168 hours, as creepage from the scribe. Finally, the IVD-Al baseline had four specimens with "0" ratings that were pulled from testing within 1000 to 2500 hours. One IVD-Al specimen, the only IVD-Al panel coated with 23377 primer, did remain exposed for 3000 hours and received a "4" rating, based on the length of creepage from the scribe. Otherwise, it does not appear that any particular primer performed better or provided more corrosion resistance.

<u>Acidic (SO_2) Salt Spray Corrosion Resistance</u>: Acidic salt fog test results indicated that all coatings were at least 2X better than Cd, and that both Cd and LHE Zn-Ni developed red rust much more quickly than the Al coatings. Of the Al coatings, IVD-Al had the most corrosion across the board, while Alumiplate performed best overall. Results for sputtered Al were less than definitive due to inadequate protection of backside and bottom surfaces of the test panels which resulted in significant paint system blistering. Nevertheless, sputtered Al outperformed IVD-Al in this test, likely due to its less porous coating structure.

<u>Run-on and Breakaway Torque</u>: All of the coatings on the 3/8-inch fasteners met both the maximum locking and breakaway torque acceptance criteria. The locking torque results stabilized for all coatings after approximately the fifth cycle with the LHE Zn-Ni having the lowest locking torque values and the sputtered Al coating had the highest. Also, the shapes of the curves and trends for locking torque closely matched the curves for breakaway torque, with the only difference being that the breakaway torque values are lower than the locking torque values, which is expected.

For the 5/8-inch fasteners, the Cd baseline and the LHE Zn-Ni had the highest maximum torque readings, once the readings stabilized at cycle 6. All results met the acceptance criteria of less than 300 in-lb. The breakaway torque readings, however, did not meet the acceptance criteria of greater than 32 in-lb. All coatings were less than 32 in-lb of torque after the second cycle, with the Cd baseline and LHE ZN-Ni having consistent results between 25 and 30 in-lb of torque.

Torque Tension: Overall, the torque tension results for the 3/8-inch fasteners show a trend, with LHE Zn-Ni performing similar to LHE Cd, where the load versus torque curves showed decreasing values over time. The Al coatings had the reverse trend, where, the load versus torque curves had higher torque values for each subsequent cycle. This same trend was not present for the 5/8-inch fasteners. The scatter in torque values between cycles was much greater for each coating. LHE Cd performed the same for the 5/8 and 3/8-inch fasteners, with decreasing torque values, while LHE Zn-Ni had the same trend for Cycles 1 and 2, then Cycle 3 had lower values than 4 and 5. The load versus torque curves for the Al coatings were not as linear as the 3/8-inch fasteners, and the torque values for each cycle varied, not showing a trend relating number of cycles with effect on the coating.

Hydrogen Embrittlement: Hydrogen embrittlement test results were satisfactory for each primary coating tested in this effort in the following conditions: (1) as-plated, (2) chemically stripped, and (3) re-plated. After chemical strip, only LHE Zn-Ni did not pass bend adhesion after re-plating the same specimen. Small amounts of plating residues were detected on some stripped specimens by energy dispersive X-ray analysis. A Boeing representative indicated that a dilute hydrochloric acid strip may be required for more complete chemical removal of the LHE Zn-Ni coating, in lieu of the pH-adjusted ammonium nitrate solution which had been recommended during this evaluation.

Results from the evaluation of the primary coatings indicated that the electroplated Al)\ coating performed very well in most tests, though it was not capable of meeting corrosion resistance requirements. The LHE Zn-Ni coating evaluated during this effort was capable of meeting most corrosion requirements, but failed a number of other tests, indicating that it was not the single suitable replacement for Cd. Finally, the sputtered Al coating exhibited the worst performance of the alternatives tested.

4.2 Repair Coatings

Brush repair coatings were subjected to only a limited number of tests in Phase II, as they were to be tested more fully in Phase III (currently not planned or funded through JCAT). Appearance, coating thickness, bend adhesion, corrosion resistance, and HE quality control testing were performed under this effort.

Appearance: Repair coatings were evaluated for appearance and held to the same requirements as the primary coatings. Brush plated Cd applied by Boeing was selected as the baseline coating for comparison. Two of the three candidate repair coatings, as well as the baseline coating, were given a pass rating. The brush plated Sn-Zn coating was given a “fail” rating, due in part to the observation of a dark brown area through the center of the panel.

Coating Thickness: If plating to the specification, the Sn-Zn coating is within the 0.3 – 0.5 mil thickness requirement. All other coatings exceeded the specification, based on the average of five measurements and five locations along the cross-section.

Bend Adhesion: Bend adhesion test results for the repair coatings were similar to those obtained in Phase I (only tested on 4130 steel). Brush plated Cd exhibited significant adhesion loss after 1.5 bend cycles (2^{nd} tensile cycle for the coating). Application of a sharp blade easily removed most of the coating. Brush Sn-Zn performed well on all specimens, as did the brush Zn-Ni panels, with only slight coating removal at the edges of the specimens. The bulk of the area did not have visually apparent cracks developing until 8-9 bend cycles, and the deposit was adherent when challenged with a blade after substrate failure. The SermeTel coating exhibited adhesion failure after 2-3 cycles. Light brushing with a fingernail removed the coating across the full width of one specimen out of three, while the other two exhibited removal closer to the edges.

Unscribed Corrosion Resistance: The brush plated metallic coatings had results similar to the primary coatings, with little to no rust present and breakdown of the sacrificial coating during the test. The spray-applied SermeTel coating performed similar to sputtered Al and some of the IVD-Al panels, with early rust formation causing test termination at about 500 hours.

Scribed Corrosion Resistance: The results for the scribed panels are very similar to the results for the unscribed panels, with the exception of the Sn-Zn panels. These panels developed rust in the scribe and were pulled from testing due to excessive rust at 500 hours.

Hydrogen Embrittlement: Brush Sn-Zn outperformed brush Zn-Ni (98.1% vs. 93.8% NFS, respectively), as was also the case in Phase I. The brush Cd and SermeTel 249 coatings had NFS values of 96.3% and 96.0%, respectively.

Similar to the primary coating, mixed results were obtained for the repair coatings. Brush plated Zn-Ni performed best overall, although it received a failing rating for coating appearance due to excessive surface roughness. Brush plated Sn-Zn was ranked second, with failing results noted in corrosion tests. The SermeTel sprayed Al ceramic coating performed worst of the repair coatings, failing both corrosion and adhesion tests. The results indicate that while brush plated Zn-Ni performed best, additional testing and evaluation is required to identify and confirm a suitable replacement for brush plated Cd.

APPENDIX A:
High Strength Steel (HSS) Cadmium Alternative Test Plan

(Available on CD)

APPENDIX B:
Raw Test Data, Results, and Photographs

(Available on CD)