Laboratory Comparison of Polysiloxane and Vinyl Coatings and Polysiloxane Field Inspection

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Bureau of Reclamation and Army Corp of Engineers are investigating materials to provide a replacement for vinyl coatings. Previous research showed epoxy-polysiloxanes as a potential replacement. This research provided three phases of polysiloxane evaluation: (1) lab testing of five manufacturers' products with candidate primers for corrosion performance, (2) lab testing of return-to-service time, i.e., a cure study to determine when water immersion service could begin, and (3) condition assessment of a field application at Tennessee Valley Authority's Fontana Dam, NC. Laboratory results showed that two of the commercial polysiloxane systems are a suitable equivalent to vinyl for immersion service, atmospheric, and cyclic environments that do not see high energy impact, erosion, or abrasion forces. Vinyl is still the preferred the coating in impacted immersion environments.

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Acronyms and Abbreviations

DI	Deionized water
DTH	Dry to handle
DTM	Direct to metal
EIS	Electrochemical impedance spectroscopy
HAR	Dilute Harrison solution
Mil Spec	Military Specification
OCP	Open circuit potential
PDS	Product data sheet
PRO	Prohesion
PSI	Pounds per square inch
Reclamation	Bureau of Reclamation
SCE	Saturated calomel electrode
S&T	Science and Technology
TVA	Tennessee Valley Authority
USACE	United States Army Corps of Engineers
UV	Ultraviolet

Measurements

RH	Relative humidity
°F	Degrees Fahrenheit
Hz	Hertz
in.	Inch

Contents

Page

Executive Summary .... **ix**
1. Introduction **1**
2. Laboratory Experiments **2**
2.1 Performance Evaluation of Commercially Available Epoxy Polysiloxane Coatings 2
2.1.1 Surface Preparation and Coating Application 2
2.1.2 Electrochemical Impedance Spectroscopy Testing 2
2.1.3 Coating Performance Evaluation 2
2.2 Reproducibility Testing and Multi-Coat System Study 7
2.3 Cure to Service Study 8
3. Results and Discussion **8**
3.1 Coating Performance Evaluation 8
3.2 Reproducibility Study and Multi-Coat Study 23
3.2.1 Reproducibility Study 23
3.2.2 Two, Three, or Four Coat System 27
3.2.3 Standard versus Fast Dry Formulation 31
3.3 Cure Study 32
4. Field Evaluation of Epoxy Polysiloxane **34**
Conclusion **37**
Recommendations **38**
Future Work **38**
References **39**
Appendix A – Coatings Rating Criteria **40**
Appendix B – EIS Data Summary for Coating Performance Evaluation **41**
Appendix C – Summary of Test Results Coating Performance Evaluation ... **43**
Appendix D – EIS Summary for Reproducibility Study **46**
Appendix E – Summary of Test Results for Reproducibility Study **47**
Appendix F – EIS Summary for Cure Study **49**
Appendix G – Adhesion Results for Cure Study **50**

Executive Summary

The historical coatings and linings used on Bureau of Reclamation (Reclamation) infrastructure provided a minimum of 50-year service lifetime. Modern coatings have been shown to provide reduced service lives. The United States Army Corps of Engineers (USACE) and Reclamation researchers have studied historical coatings, including vinyl resin coatings, to better understand what properties provided long-term corrosion resistance. Within the past five years, researchers identified several epoxy polysiloxane coatings that have similar barrier properties to these historical coatings and could potentially provide a similar service life. However, manufacturers designed epoxy polysiloxane coatings for ultraviolet (UV) exposure in atmospheric service environments as an alternate system to polyurethane coatings which contain isocyanates. Therefore, the suitability and performance of epoxy polysiloxanes for water immersion service required laboratory and field validation.

The present study performed three phases of laboratory investigation to further understand potential applications at Reclamation. The investigation involved laboratory testing five manufacturers' products with candidate primers to evaluate each system's corrosion performance. The second investigation was a reproducibility study that looked if the same results can be achieved with different batch numbers and applicators for the same coating system. The reproducibility study also looked at the difference between using one, two, and three coats of polysiloxane paired with a zinc rich primer. The third investigation was lab testing to evaluate return-to-service time, i.e., a cure study to determine when water immersion service could begin. A subsequent field application investigation was a condition assessment of a structure coated with epoxy polysiloxane at the Tennessee Valley Authority's Fontana Dam, NC.

This research investigated five different coating manufacturers' epoxy polysiloxanes and recommended primer systems for a total of 32 coating systems. The laboratory investigated coating systems were compared to USACE vinyl System 4 and System 5-E-Z and epoxy Mil Spec 24441 via exposure of coated metal coupons to five accelerated weathering cyclic tests for 5040 hours and long-term water immersion. The epoxy polysiloxanes systems used epoxy primers, zinc phosphate inhibitive primers, zinc rich primers, or direct to metal (DTM), i.e., no primer. The performance analysis included electrochemical impedance spectroscopy (EIS), adhesion testing, cathodic disbondment, direct impact testing, slurry erosion testing, and Taber abrasion.

Results from laboratory testing showed when epoxy primers are utilized, polysiloxane 1 from Manufacturer 1 and polysiloxane 2 and 3 from Manufacturer 2 both have a polysiloxane that performed well in water immersion service. Manufacturers do not include cure time required for immersion service for atmospheric service products; a cure study was conducted to determine how different cure times impact the coatings performance. The cure study showed that the polysiloxanes tested increase in impedance once placed in water immersion. This could suggest they finish curing in water and provide the same barrier properties as the polysiloxanes cured in air. Only one of the two recommended manufacturers' polysiloxanes was used for the cure study and it is recommended that this product be allowed to cure for one week before exposure to immersion service. Cure

studies for additional products would need to be conducted to determine recommended cure time before being placed in immersion service.

Polysiloxane 1 from Manufacturer 1 and polysiloxane 2 and 3 from Manufacturer 2provided excellent corrosion protection through all accelerated weathering. However, for direct impact, abrasion resistance, and erosion resistance properties were not comparable to vinyl coatings but were comparable to Mil Spec epoxy 24441. Manufacturer 1 and 2 coatings eroded 1.5 times as fast as vinyl within the 96-hour test which would be unacceptable in areas that are subjected to erosion from sediment. The epoxy polysiloxanes had better cathodic disbondment resistance than the vinyl systems, indicating good compatibility with cathodic protection.

The Tennessee Valley Authority (TVA) applied an epoxy polysiloxane to four radial gates structures at Fontana Dam in 2015 and 2016. In the fall of 2019, Reclamation researchers inspected and collected quantitative EIS data on the gates. The gates were in excellent condition with adequate barrier properties.

Recommendations (see main report body for comprehensive list)

- Vinyl coatings are still the recommended coating for impacted immersion service.
- Polysiloxane 1 from Manufacturer 1 and Polysiloxanes 2 and 3 from Manufacturer 2 with the manufacturer recommended epoxy primer are suitable for use on Reclamation structures in immersion service and fluctuating immersion service if there is no risk of impact damage, erosion, or abrasion.
- Field applications of polysiloxanes should be monitored for corrosion protection and durability performance including long-term data collection.
- Partnership opportunities should be explored with manufacturers and other coatings experts to improve overall polysiloxane durability.

1. Introduction

In 2016, Reclamation and the USACE entered a collaborative effort to better understand how vinyl coatings provided long-term corrosion protection and to look for alternative coatings that were equivalents to the USACE vinyl systems 4 and 5-E-Z. In the initial studies, Science and Technology (S&T) project 8835 (Finding a Green Alternative to Vinyl Coatings), two different epoxy polysiloxanes from two different manufacturers were investigated in conjunction with the manufacturer's recommended epoxy primers. The findings showed one of the epoxy polysiloxanes had excellent corrosion resistant barrier properties but didn't have the same undercutting resistance in cyclic testing as the USACE vinyl systems. However, the undercutting resistance was improved when a zinc rich primer or different epoxy primer was used. This initial study provided the motivation to further study epoxy polysiloxanes.

The present research project evaluated the performance of all epoxy polysiloxane coatings commercially available between 2016-2021, utilizing different primer systems and DTM applications for a total of 32 different systems. Coating manufacturers market epoxy polysiloxanes for atmospheric exposure and do not list water immersion service on the product data sheets. Previous Reclamation research found some provide excellent barrier properties for water immersion service. Due to these results, all coatings systems for this research were evaluated for their corrosion protection in water immersion. This is to determine polysiloxanes from different manufacturers have satisfactory barrier properties in water immersion. Results from previous research projects also have Reclamation researchers recommending polysiloxane coatings for immersion and cyclic immersion environments which currently all outside the manufacturers' designation. The manufacturers' product datasheets do not report a return to service time; therefore, this research also included a cure to service study which evaluates when the coating has cured to the point it can be exposed to the service environment without damage occurring to the coating.

In addition, manufacturers are recommending that a two-coat system of zinc rich primer and single coat polysiloxane could be utilized to save costs in the number of coats required. This study evaluated whether a two-coat system could provide adequate corrosion protection or if multiple coats of polysiloxane are required. A three-coat system (one coat of zinc rich primer and two coats of epoxy polysiloxane) and four coat system (one coat of zinc rich primer and three coats of epoxy polysiloxane) were applied in a side-by-side comparison in laboratory testing for corrosion protection, in addition to the manufacturer recommended two coat system. The study looked at how stresses from multiple-coat systems would affect the coating system. By increasing the number of coats for a system, the internal stress from cross-linking can increase. These stresses can cause the coating to micro-crack or become brittle and more susceptible to damage.

The research also evaluated the field performance of the epoxy polysiloxane coating system in water immersion service. Tennessee Valley Authority (TVA) recoated four radial gates at Fontana Dam, NC, in 2015 and 2016 with an epoxy primer and polysiloxane topcoat. Reclamation requested to inspect the gates to see how the polysiloxane coating system had performed after 4 to 5 years of field exposure. A field scale up application of polysiloxane compared to vinyl was included on the original scope of this project. However, due to COVID-19, this portion of the scope was removed from the project and the data from the inspection at Fontana Dam was the only field data collected.

2. Laboratory Experiments

2.1 Performance Evaluation of Commercially Available Epoxy Polysiloxane Coatings

2.1.1 Surface Preparation and Coating Application

Surface preparation for all panels consisted of removing oil and contaminants by detergent cleaning following SSPC-SP1. Once panels were cleaned, they were abrasive blast cleaned to SSPC-SP 5/NACE 1 with an angular profile of 3.5 mils. Coatings were applied in accordance with coating manufacturers' instructions. The epoxy polysiloxane coatings were applied with conventional spray equipment. The polysiloxane systems required 40 percent relative humidity (RH) and were placed in a containment with a humidifier to obtain 60 percent RH during cure inside the spray booth. After coatings were cured dry to handle (DTH), they were moved to an environmentally controlled room held at 70 degrees Fahrenheit (°F) with 70 percent RH and remained there until fully cured. Each coating system had a set of twenty-four 3-inch (in.) wide by 6-in. long by 0.125-in. thick steel coupons, two 4-in. diameter by 0.125-in. thick round disks, two 11-in. diameter by 0.125-in. thick round disks, and one 3-in. diameter by 1-foot-long section of pipe. All specimens were made using A36 carbon steel.

2.1.2 Electrochemical Impedance Spectroscopy Testing

Electrochemical impedance spectroscopy (EIS) was performed with a Gamry Instruments FAS2 Femtostat, with dedicated EIS300 software. All measurements had a 10-millivolt sinusoidal perturbation at the open circuit potential, a frequency range of 10^5 to 10^{-2} Hertz (Hz), and ten data points per decade. The EIS test cell was consistent with a three-electrode set-up, a saturated calomel electrode (SCE), platinum mesh electrode, and the steel substrate were connected to the instrument as the reference, counter, and working electrode, respectively. The EIS testing surface area, as defined by the test cell, is 23 square centimeters. No corrections were made to the raw data for surface area. EIS was performed periodically throughout a 30-week exposure. If coatings performed well during the 30-week evaluation, long-term testing in water immersion service commenced to monitor the barrier properties over time.

2.1.3 Coating Performance Evaluation

Reclamation's standard testing protocol was used (listed below) and the rating criteria can be found in Appendix A:

- HAR - Immersion in a dilute Harrison solution (HAR), 0.5 grams sodium chloride and 3.5 grams ammonium sulfate per liter of deionized (DI) water (ASTM D870 Modified)
- DI - Immersion in DI water (ASTM D870)
- FOG - Prohesion testing in a salt fog test cabinet (ASTM G85 Annex A4)

- QUV - Condensation and UV cyclic test (ASTM D4587)
- PRO - Prohesion cyclic testing (ASTM D5894)
- BOR - Modified Prohesion test
- Pull-off Adhesion testing (ASTM D4541)
- Cathodic disbondment (ASTM G8)
- Direct impact testing (ASTM D2794)
- Erosion resistance (USBR-5071-2015)
- Knife adhesion (ASTM D6677)

HAR and DI immersion panels were suspended from hooks through pre-cut holes in an immersion tank. The tank depth was sufficient to fully submerse the panels. To ensure homogeneity, the solutions were mixed before adding to the tank. Each tank was connected to a filtration system that circulated the solutions through the tank. The tanks were cleaned, and solutions replaced once a year.

The BOR test is a modified Prohesion test, which is intended to simulate the effects of a fluctuating immersion environment/splash zone. Panels were rotated weekly in the following order: QUV-FOG-HAR-FOG.

All test panels were exposed for approximately 30 weeks (5040 hours) in accordance with industrial standard practices. Panel evaluation proceeded according to ASTM D1654, Standard Test Method for Evaluation of Painted or Coated Specimens Subjected to Corrosive Environments (rust creep), and ASTM D714, Standard Test Method for Evaluating Degree of Blistering of Paints. Two panels for HAR and DI immersion, BOR, PRO, FOG, and QUV tests were scribed down the center, on one side, with a Dremel® tool. The scribes were approximately 1 millimeter in width and 3 inches in length to expose the steel substrate. Post-exposure analysis of the scribe determines how well the coating could arrest corrosion and resist undercutting if a coated structure was damaged to the substrate.

To test each system's compatibility with cathodic protection systems, 3-inch diameter pipes were coated with each system. A cathodic protection system was then simulated in accordance with ASTM G8, Standard Test Methods for Cathodic Disbonding of Pipeline Coatings, for a test duration of 120 days.

The slurry erosion test is a Reclamation standard following the USBR-5071-2015 testing procedure. Coated 11-inch diameter, 0.125-in. thick steel discs were used. The test duration was 96 hours per specimen. Duplicate specimens were run. The average weight loss was compared between test specimens and a control.

The coating systems in this report are given a product code: First, manufacturers are numbered 1-5; then coating type: E = epoxy, I = inhibitive epoxy, Z = zinc rich epoxy, and PS = polysiloxane; the final number indicates if there were multiple coating types evaluated. For example, product code 1-E1-PS1, means manufacturer number 1, epoxy number 1 as the primer, and polysiloxane number 1 single coat. If multiple coats of a coating were applied the number of coats followed by "C" was included in the product code. Systems that only used one product were classified as DTM. For example, product code 2-PS1DTM-3C, is Manufacturer 2 polysiloxane applied DTM as a three-coat system. If the product code includes an "R," this represents a coating system that had a second set of test specimens produced for the reproducibility study.

All systems were compared to USACE vinyl System 4 and vinyl System 5-E-Z, along with Military Specification (Mil Spec) 24441 epoxy, which is a solvent borne epoxy. These coatings were selected since vinyl is still the preferred choice for impacted water immersion coatings, and solvent borne epoxies are still widely used in the industry. Table 1 is a summary of all coating systems tested during study.

Table 1: All coating systems for performance evaluation study. A total of eight polysiloxane were tested. A total of 32 coating systems were tested for this study. For coating system that utilized a primer, the recommended primer system from the same manufacturer were used.

Coating Manufacturer	Report Code	Primer	Intermediate Coat(s)	Topcoat
	Vinyl System 4	Vinyl	3 coats vinyl	Vinyl
	Vinyl System 5-E-Z	1 coat zinc rich vinyl	4 coats vinyl	Vinyl
	Epoxy Mil Spec 24441	Solvent borne epoxy	Solvent borne epoxy	Solvent borne epoxy
1	1-E1-PS1	Epoxy primer 1	Epoxy primer 1	Polysiloxane 1
1	1-E2-PS1	Epoxy primer 2	Epoxy 2 reinforced with glass flake	Polysiloxane 1
1	1-E3-PS1	Epoxy primer 3	Epoxy primer 3	Polysiloxane 1
1	1-I1-PS1	Inhibitive epoxy primer	n/a	Polysiloxane 1
1	1-PS1DTM-2C	Polysiloxane 1	n/a	Polysiloxane 1
1	1-PS1DTM-3C	Polysiloxane 1	Polysiloxane 1	Polysiloxane 1
1	1-Z1-PS1-1C	Zinc rich primer 1	n/a	Polysiloxane 1
1	1-Z1-PS1-2C	Zinc rich primer 1	Polysiloxane 1	Polysiloxane 1
1	1-Z1-PS1-3C	Zinc rich primer 1	2 coats polysiloxane 1	Polysiloxane 1
1	1-PS2DTM -3C	Polysiloxane 2	Polysiloxane 2	Polysiloxane 2
1	1-E3-PS2	Epoxy primer 3	Epoxy primer 3	Polysiloxane 2
2	2-E1-PS1	Epoxy primer 1	Epoxy primer 1	Polysiloxane 1
2	2-E2-PS1	Epoxy primer 2	Epoxy primer 2	Polysiloxane 1
2	2-Z1-E3-PS1	Zinc rich primer 1	Epoxy 3	Polysiloxane 1
2	2-PS1DTM -3C	Polysiloxane 1	Polysiloxane 1	Polysiloxane 1
2	2-E2-PS2	Epoxy primer 2	Epoxy primer 2	Polysiloxane 2
2	2-I1-PS2	Inhibitive primer 1	n/a	Polysiloxane 2
2	2-PS2DTM-3C	Polysiloxane 2	Polysiloxane 2	Polysiloxane 2
2	2-Z1-PS2- 1C	Zinc rich primer 1	n/a	Polysiloxane 2
2	2-Z1-PS2- 2C	Zinc rich primer 1	Polysiloxane 2	Polysiloxane 2
2	2-Z1-PS2- 3C	Zinc rich primer 1	2 coats polysiloxane 2	Polysiloxane 2
2	2-Z1-PS3- 1C	Zinc rich primer 1	n/a	Polysiloxane 3
2	2-Z1-PS3- 2C	Zinc rich primer 1	Polysiloxane 3	Polysiloxane 3
2	2-Z1-PS3- 3C	Zinc rich primer 1	2 coats polysiloxane 3	Polysiloxane 3

Coating Manufacturer	Report Code	Primer	Intermediate Coat(s)	Topcoat
3	3-PS1DTM-3C	Polysiloxane 1	Polysiloxane 1	Polysiloxane 1
	3-E1-PS1	Epoxy primer 1	Epoxy primer 1	Polysiloxane 1
4	4-PS1DTM - 3C	Polysiloxane 1	Polysiloxane 1	Polysiloxane 1
	4-Z1-PS1	Zinc rich primer	n/a	Polysiloxane 1
	4-Z1-E1-PS1	Zinc rich primer	Epoxy 1	Polysiloxane 1
	4-E1-PS1	Epoxy primer 1	Epoxy 1	Polysiloxane 1
5	5-PS1DTM-2C	Polysiloxane 1	n/a	Polysiloxane 1
	5-PS1DTM-3C	Polysiloxane 1	Polysiloxane 1	Polysiloxane 1
	5-Z1-PS1	Zinc rich primer 1	n/a	Polysiloxane 1
	5-Z2-PS1	Zinc rich primer 2	n/a	Polysiloxane 1
	5-I1-PS1	Inhibitive primer 1	n/a	Polysiloxane 1
	5-E1-PS1	Epoxy primer 1	Epoxy 1	Polysiloxane 1

2.2 Reproducibility Testing and Multi-Coat System Study

Coating systems test specimens for the reproducibility portion of testing were prepared using coatings from different batch numbers and applied by different applicators. This was to verify reproducibility of the results in laboratory corrosion protection testing. A total of four applicators were used to apply the different coating systems. For system 1-Z1-PS1-2C-R, the zinc primer was sanded due to applicator error. The zinc coating was applied too thick and was sanded down to achieve the correct DFT. Sanding occurred the same day as the polysiloxane application. Table 2 summarizes all coating systems evaluated for the reproducibility study.

Table 2: Coating systems evaluated for reproducibility study.

Coating Manufacturer	Report Code	Primer	Intermediate Coat(s)	Topcoat
1	1-Z1-PS1-2C-R	Zinc rich primer	Polysiloxane 1	Polysiloxane 1
	1-PS1DTM-3C-R	Polysiloxane 1	Polysiloxane 1	Polysiloxane 1

The multicoat portion of testing used two products, each from a different manufacturer. Several coating manufacturers recommend a two-coat system, for corrosion protection, comprised of a zinc rich epoxy and a single coat of polysiloxane. This study investigated whether equivalent or better performance is obtained by increasing the number of polysiloxane coats in the system. Each system consisted of one coat of zinc rich epoxy primer followed by one, two, or three coats of polysiloxane. Manufacturer 1 also included a two- and three-coat system of the polysiloxane applied DTM. The panels for Manufacturer 2 were coated using their standard polysiloxane formulation, report code PS2. Two manufacturers developed a fast-dry version which cures at lower humidity levels down to 15 percent RH and lower temperatures down to 20 °F. These products address applications issues in arid and colder climates by allowing the product to cure at lower temperatures and percent RH. Coating systems were applied using Manufacturer 2's fast dry formulation and given the report code PS3. All coating systems for the reproducibility study and multi-coat study cured following the same cure procedure outlined in section 2.1.1. Table 3 summarizes all coating systems used for the two-, three-, and four-coat system study. This test was conducted for products from Manufacturers 1 and 2.

Table 3: Coating systems evaluated for one, two, and three coats of polysiloxane.

Coating Manufacturer	Report Code	Primer	Intermediate Coat(s)	Topcoat
1	1-Z1-PS1-1C	Zinc rich primer	n/a	Polysiloxane 1
	1-Z1-PS1-3C	Zinc rich primer	2 coats polysiloxane 1	Polysiloxane 1
	1-PS1DTM-2C	Polysiloxane 1	n/a	Polysiloxane 1
2	2-Z1-PS2-1C	Zinc rich primer	n/a	Polysiloxane 2
	2-Z1-PS2-2C	Zinc rich primer	Polysiloxane 2	Polysiloxane 2
	2-Z1-PS2-3C	Zinc rich primer	2 coats polysiloxane 2	Polysiloxane 2
	2-Z1-PS3-1C	Zinc rich primer	n/a	Polysiloxane 3
	2-Z1-PS3-2C	Zinc rich primer	Polysiloxane 3	Polysiloxane 3
	2-Z1-PS3-3C	Zinc rich primer	2 coats polysiloxane 3	Polysiloxane 3

2.3 Cure to Service Study

Coating manufacturers do not publish a cure time for immersion service on the product datasheets since they only recommend polysiloxanes for atmospheric exposure. Panels for the cure to service study were prepared separately from panels for the coating's performance evaluation study in Section 2.1. The surface preparation followed the procedure described in Section 2.1.1. Six panels for each cure duration were prepared for a total of 30 panels for the study. Once the final coat was applied, the panels cured to DTH before being transferred to a 70 percent RH, 70 °F room. The coatings cured for prescribed durations in the environmentally controlled room prior to being placed in a HAR immersion tank. Table 4 shows the cure durations for each test.

Table 4: Cure schedule for immersion and adhesion panels. Six panels for each cure duration were produced.

Coating System	Test	Cure Duration (At 70% RH 70°F)				
		0 hrs (DTH)	72 Hrs	1 Week	2 Weeks	8 Weeks
2-PS1-DTM2C	EIS	X	X	X	X	X
	Adhesion	X	X	X	X	X

EIS was used to evaluate the performance of each cure duration and determine the impact cure has on corrosion protection in immersion service. EIS tests were conducted daily for the first two weeks after being placed in immersion, weekly for the next eight weeks, and monthly for the remainder of the study. The cure study also included direct pull-off adhesion testing to see if cure duration effects the coatings' adhesion strengths. Adhesion testing was performed in accordance with ASTM D4541, Standard Test Method for Pull-Off Strength of Coatings Using Portable Adhesion Testers.

3. Results and Discussion

3.1 Coating Performance Evaluation

A summary of the EIS test results, at selected test dates, for all commercially available epoxy polysiloxane coatings evaluated can be found in Appendix B. The coatings' barrier properties were determined from the overall impedance magnitude and the phase angle; both are measured at 0.01 Hz. The phase angles indicate resistive behavior (pure resistor is 0 degrees), capacitive behavior (pure capacitor is -90 degrees), or a combination, which is the case for most coatings. Resistive behavior is generally indicative of corrosion reactions at the substrate. For this report the phase angle was divided into three categories to aid in the interpretation of the data. Coatings are labeled as having more resistive behavior when the phase angle is 0 to -30 degrees, capacitive behavior from -60 to -90 degrees, and the term "mixed" when the coating has both capacitive and resistive behavior between -30 to -60 degrees.

Coatings with higher impedance magnitude and capacitive behavior may tend to indicate good long-term performance in the field. The EIS test results showed that most of the polysiloxane coating systems evaluated had "excellent" barrier properties. These coating systems had an impedance magnitude greater than 1 x 10^9 ohms and had less than one order of magnitude impedance reduction over the course of the seven-month testing period. For the coating systems that passed this Reclamation testing criterion, the EIS panels were kept in immersion testing and will be evaluated for long-term EIS data collection which is ongoing. Most of the polysiloxane systems demonstrated capacitive or mixed, i.e., intermediate to resistive and capacitive behaviors. Further, the use of different primers and DTM application for the polysiloxanes did not appear to influence the coating system's barrier properties, i.e., the measure impedance magnitude. This suggests that the impedance of the polysiloxane exceeds that of the other layers of the coating system.

Five of the coating systems were considered to have "good" barrier properties, which is the second highest rating given. Those systems included the control Mil Spec 24441 and all four of Manufacturer 2's systems that were top-coated with PS1. These systems had a final impedance magnitude between 1 x 10^8 and 1 x 10^9 ohms with less than two orders of magnitude reduction over the testing period. Coating systems 1-PS2DTM-3C and 4-Z1-PS1 had "poor" barrier properties having an impedance magnitude around 1 x 10^7 ohms or less. 1-PS2DTM-3C blistered during the first month of water immersion testing.

The EIS results showed that most of the polysiloxanes tested have barrier properties that are equivalent or better than the vinyl systems over the same seven-month testing period. Figure 1 shows the EIS Bode plots for vinyl System 4, Figure 2 shows the EIS Bode plots for vinyl System 5-E-Z, and Figure 3 shows the EIS Bode plots for polysiloxane system 1-E1-PS1. The polysiloxane system 1-E1-PS1 had the best combination of high performance and a long evaluation period and still displays excellent barrier properties after five years of immersion testing. Most polysiloxane systems exposed in HAR and DI immersion testing for three years or less had similar EIS results to the Bode plot of system 1-E1-PS1 shown in Figure 3.

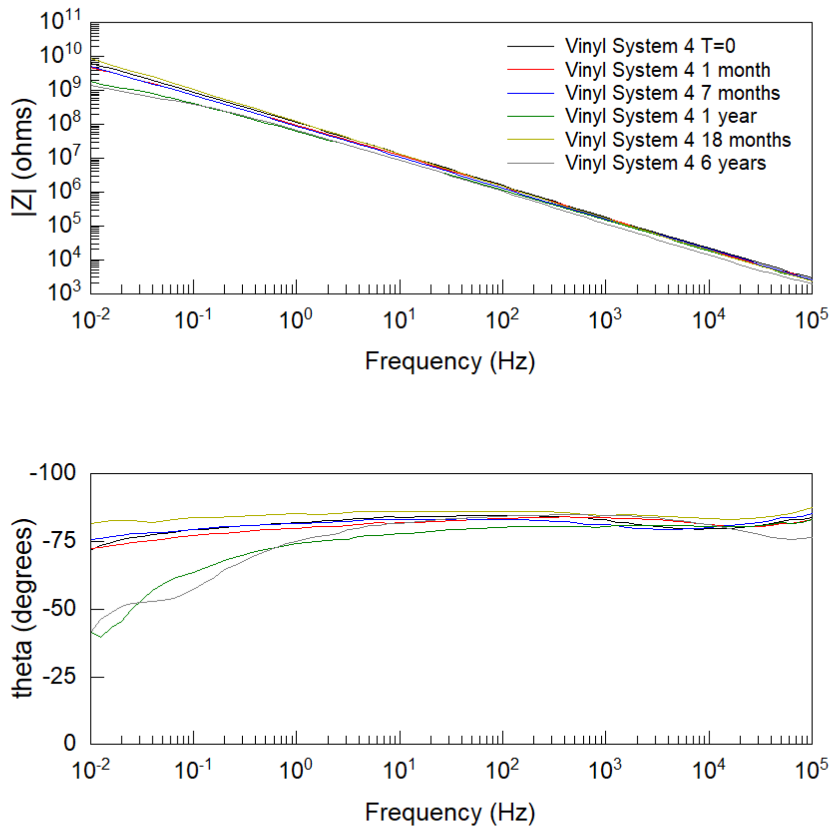

Figure 1. EIS data presented as Bode plot for vinyl system 4, exposure time is 6 years. The impedance magnitude is stable and stays above 1 x 10^9 ohms, and phase angle stays below -45 degrees.

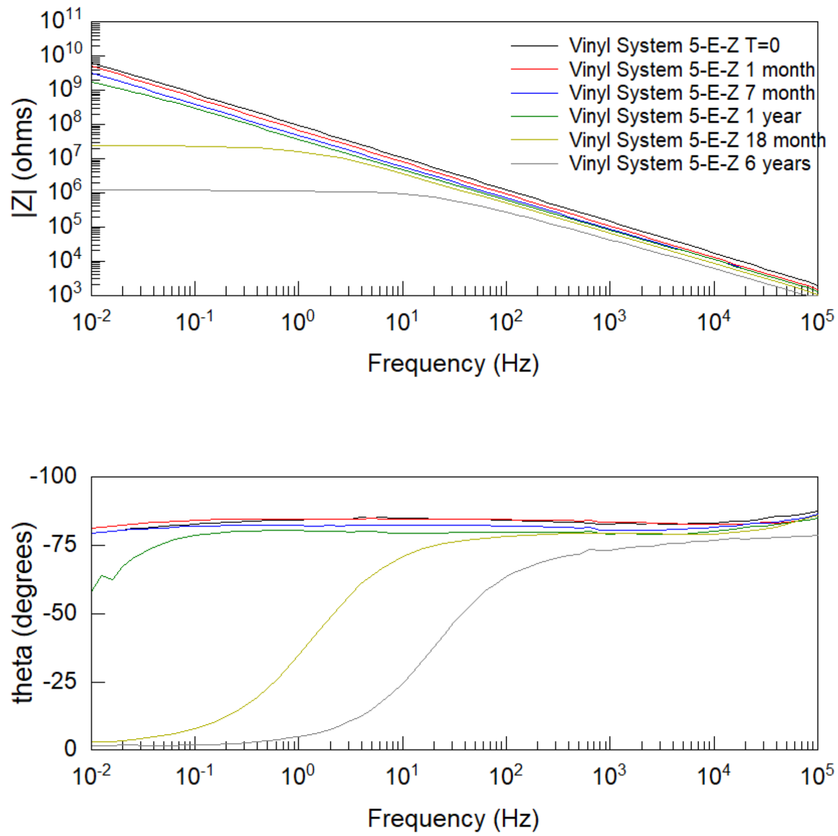

Figure 2. EIS data presented as Bode plot for vinyl system 5-E-Z, exposure time is 6 years. The impedance magnitude and phase angle decreased during immersion testing.

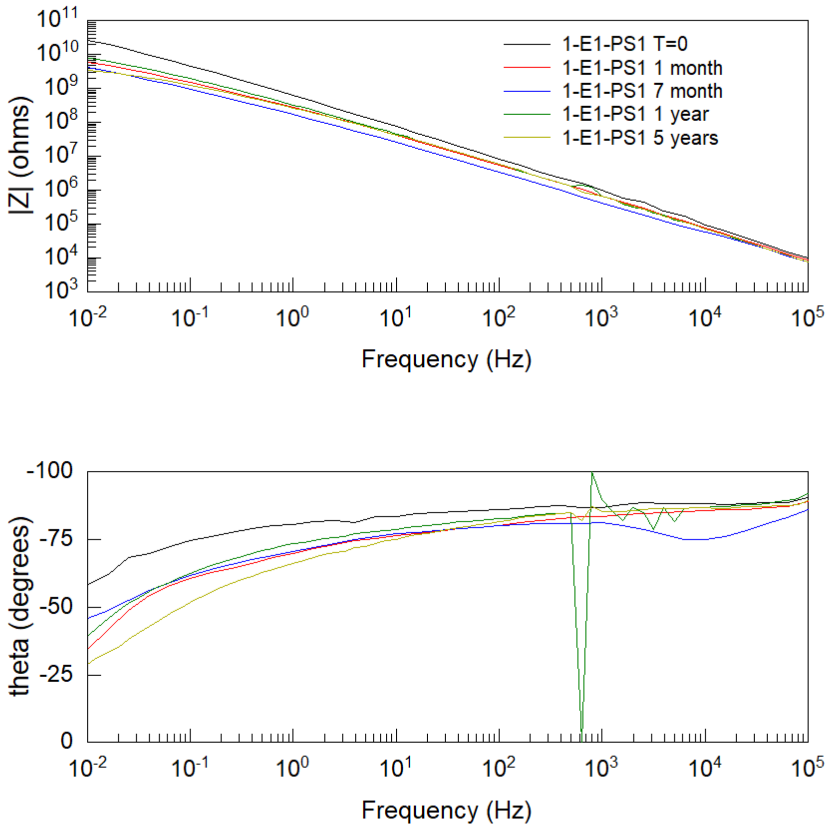

Figure 3. Bode plot for polysiloxane system 1-E1-PS1, exposure time is 5 years.

System 5-E-Z shows capacitive behavior for the eighteen months, but then becomes resistive. Resistive behavior usually indicates that the coating is becoming more conductive due to ions in the electrolyte penetrating the coating and participating in charge transfer, i.e., corrosion reactions at the substrate. However, for the System 5-E-Z it could also be due to zinc metal reactions on the conductive pigments in the primer. Resistive properties can be seen in the Mil Spec 24441 epoxy, a commonly used epoxy, and polysiloxane systems 2-E1-PS1, 2-E2-PS1, 2-Z1-E3-PS1, and 2-PS1DTM-3C. Figure 4 show the EIS Bode for Mil Spec 24441 epoxy, and Figure 5 show the EIS Bode for polysiloxane system 2-E1-PS1. These plots look very similar to each other with resistive behaviors present at the beginning of the exposure. The data also suggests that the polysiloxane has more stable properties than this epoxy, particularly after the initial exposure measurement.

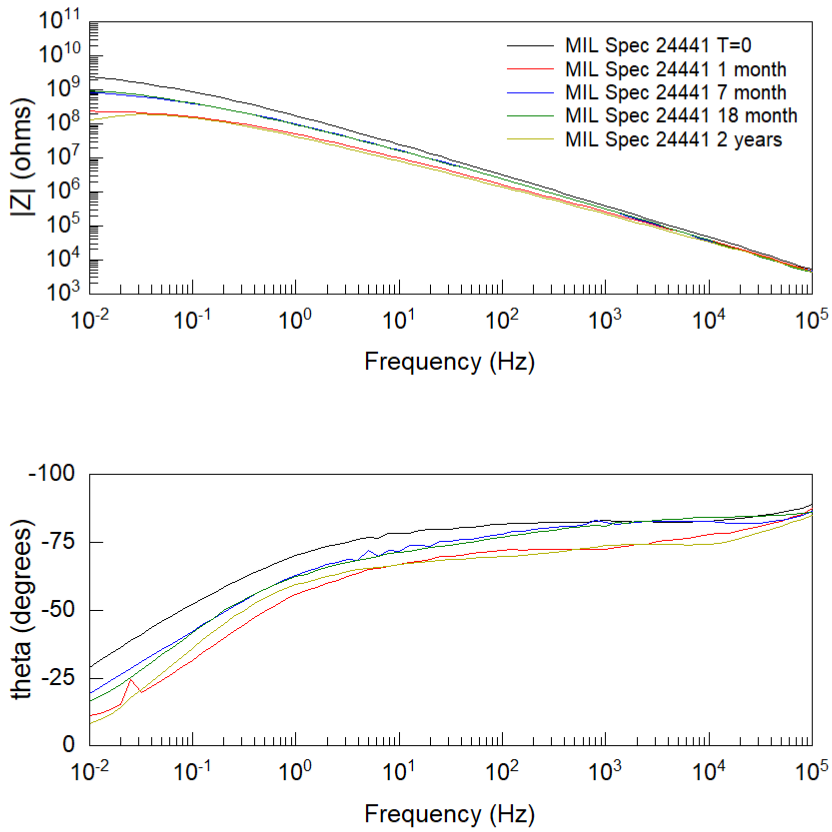

Figure 4. Bode plot for Mil Spec 24441 epoxy, exposure time is 2 years.

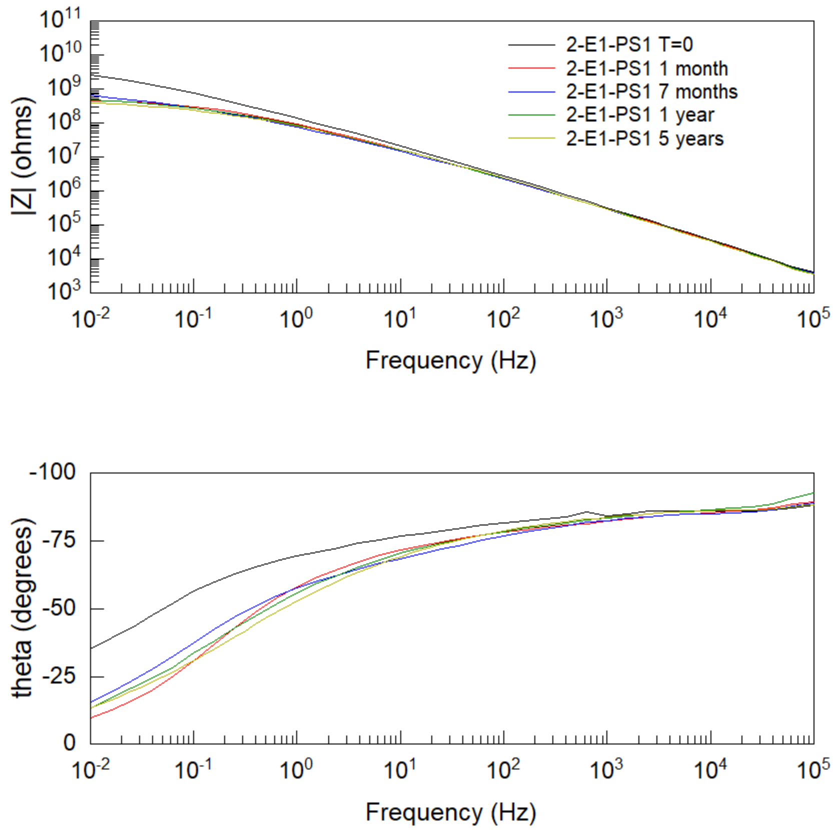

Figure 5. Bode plot for polysiloxane system 2-E1-PS1, exposure time is 5 years.

Figure 6 provides the impedance magnitude at 0.01 Hz for entire period of testing for coating systems vinyl 5-E-Z, vinyl 4, Mil Spec 24441, 1-E1-PS1, and 2-E1-PS1. Both vinyl systems show a decrease in the impedance magnitude with a steep drop in impedance after 1.5 years in immersion testing. Vinyl system 5-E-Z also has some impedance magnitude measurements around 1×10^7 ohms at 0.01 Hz. One hypothesis behind this is the zinc pigments, which act as a sacrificial anode in the coating, are conductive and as electrolytes enter the coating over time the impedance magnitude drops, and the phase angle approaches zero as a result.

Mil Spec 24441 epoxy and both polysiloxane systems have an initial slight decrease in impedance but remain stable afterwards. Polysiloxane system 1-E1-PS1 maintains the highest impedance value out of these five coating systems. Mil Spec 24441 and 2-E1-PS1 have the lowest initial impedance values of the five coatings but both are stable after several years of immersion testing. System 2-E1-PS1 has the lowest impedance with a magnitude of 1×10^8 ohms which is consistent for all four coating systems that had 2-PS1 as the topcoat. This may be caused by greater porosity or damage within the coating matrix or at the steel substrate lowering the impedance magnitude.

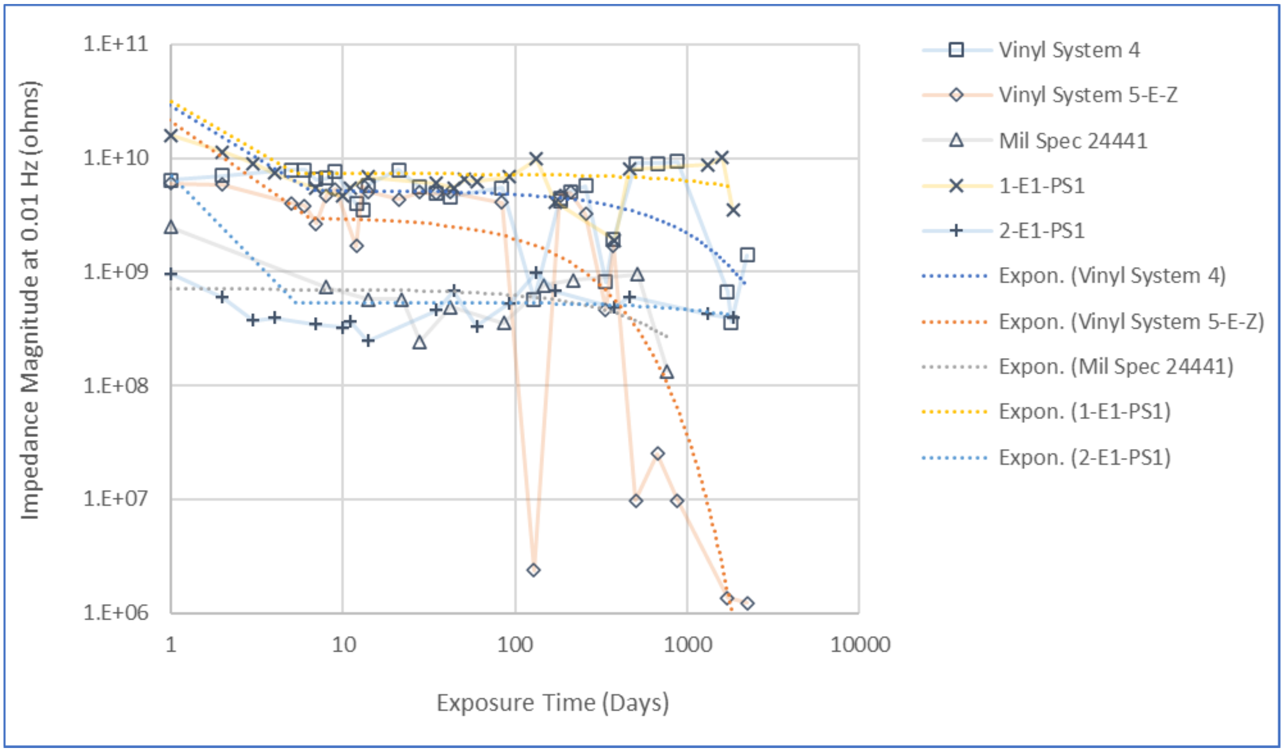

Figure 6. EIS plot of low frequency impedance at 0.01Hz vs. exposure time. An exponential trend line was fitted for each coating system. Notice Vinyl System 5-E-Z significantly drops in EIS magnitude after 1.5 years (about 500 days) in immersion.

Vinyl system 5-E-Z is the best performing coating system used by USACE and obtains a long service life. In direct comparison between the polysiloxane systems with zinc rich primers, 1-Z1-PS1 and 2-Z1-PS2, to vinyl System 5-E-Z, the polysiloxane systems still have capacitive properties at 1.5 years, whereas the vinyl has become resistive. Long term testing should continue to determine when these polysiloxane systems become resistive and could provide an indication of service life.

Close visual inspection of immersion panels for both vinyl System 4 and vinyl System 5-E-Z indicated that defects formed on the surface after six years in immersion testing. Vinyl System 5-E-Z had formed a textured surface, as shown in Figure 7. This could be caused by the formation of zinc oxide which is less dense than the zinc pigments in the coatings. As the galvanic reaction takes place, the zinc is converted into zinc oxide, expanding, causing the textured surface of the coating. At this time, the cause of the textured surface is not determined. A few large diameter blisters had formed on two of the vinyl System 4 panels shown in Figure 8. These panels can be compared to two polysiloxane systems, 2-E1-PS1 and 1-E1-PS1, which have been in immersion testing for five years. Both had mild discoloration from staining and system 2-E1-PS1 had one blister form at the bottom of the panel as shown in Figure 9. System 1-E1-PS1 did not have visible defects.

Figure 7: Vinyl System 5-E-Z after six years of immersion testing. Textured surface is possibly due to zinc pigments reacting to form zinc oxide and zinc hydroxide.

Figure 8. Vinyl System 4 after six years on immersion testing. Large blisters have formed around edges and small blisters formed near EIS test cell.

Figure 9. System 1-E1-PS1 (Left) and system 2-E1-PS1 (Right) after five years immersion testing. Notice one small blister on the bottom of the panel of system 2-E1-PS1.

At the beginning of the study, no manufacturers had water immersion service included on their product data sheets and currently do not include as a suitable service environment. Results from laboratory testing show most of the evaluated polysiloxane coatings systems provided excellent corrosion protection and undercutting resistance in water immersion. Eight polysiloxane systems formed blisters in both HAR and DI immersion testing. The blisters formed at different stages during testing with the earliest being after one month of testing and the latest being after five years of testing. Five of the six Manufacturer 5 systems blistered in water immersion service and it's unknown how or why blisters formed, see Figure 10. These polysiloxane systems had impedance magnitudes of 1×10^{10} and phase angles between -50 and -65 degrees and should have shown good corrosion protection. There are many possible reasons blisters formed, but identifying the cause was not part of the scope of work. These systems failed to meet the desired performance criteria and were removed from testing.

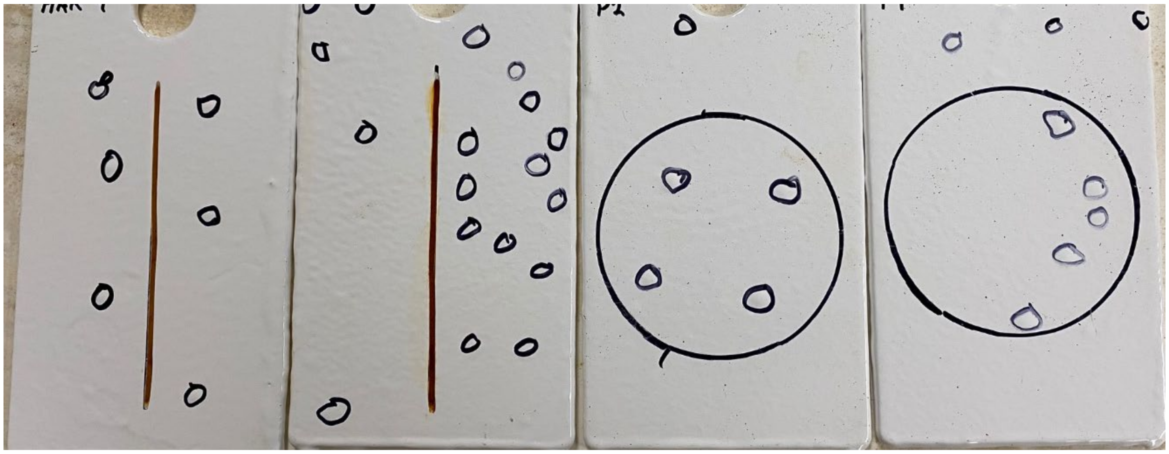

Figure 10. Polysiloxane system 5-Z1-PS1 after 1 month in HAR water immersion. The black circles show the blister locations.

The cyclic test results are found in Appendix C. The primer system used had a large influence on the undercutting resistance of the coating system, as to be expected. Figure 11 shows that polysiloxanes paired with a zinc rich primer improved the undercutting resistance in cyclic testing and perform equal to or better than vinyl System 5-E-Z. Figure 12 through Figure 15 show photos of BOR and PRO cyclic testing for vinyl System 5-E-Z and system 2-Z1-PS2 in side-by-side testing. The inhibitive primers reduce the undercutting slightly but did not meet the performance of the zinc rich primers. The undercutting resistance performance of the DTM systems depended on the manufacturer or number of coats in the system. Some of the DTM performed better than epoxy while other DTM polysiloxane systems performed worse. All the polysiloxanes had excellent gloss and color retention in the QUV cyclic weather test, while there was a slight change in gloss and color for the vinyl Systems 4 and 5-E-Z.

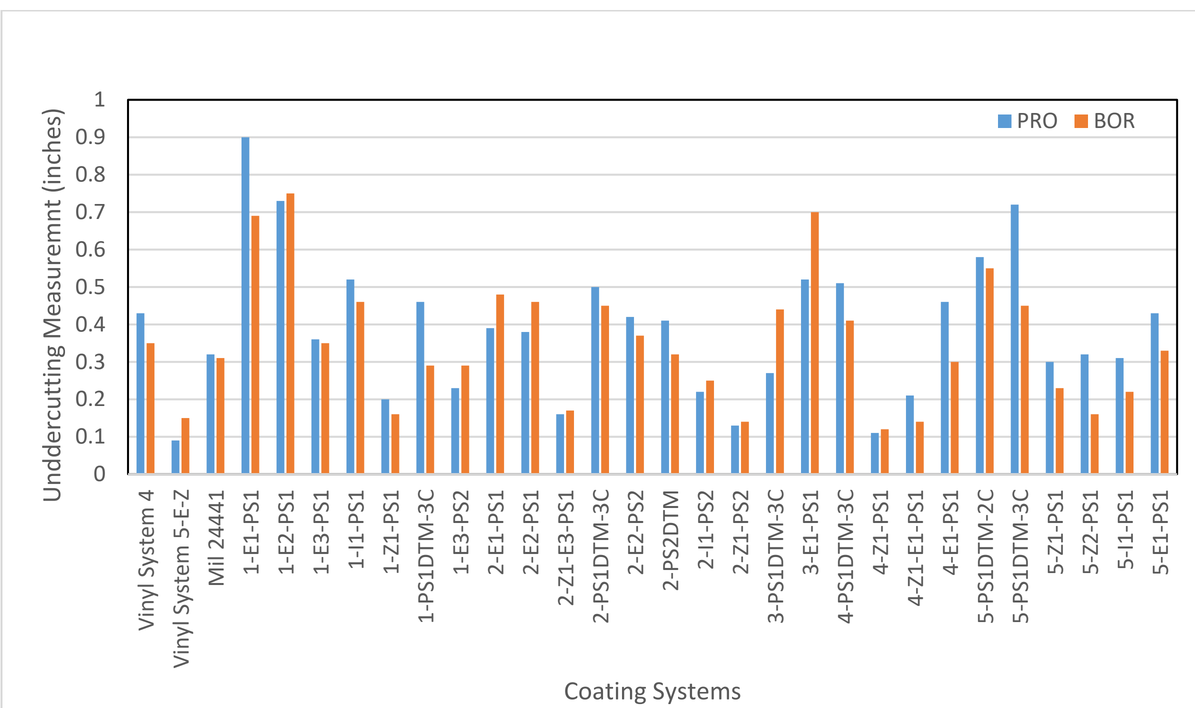

Figure 11: Undercutting results for all polysiloxanes and controls in cyclic testing.

Figure 12. PRO cyclic test of vinyl system 5-E-Z; (left) before scraping (right) after scraping away coating.

Figure 13. BOR cyclic test of vinyl system 5-E-Z; (left) before scraping (right) after scraping away coating.

Figure 14. BOR cyclic test of 2-Z1-PS2; (left) before scraping (right) after scraping away coating.

Figure 15. PRO cyclic test of 2-Z1-PS2; (left) before scraping (right) after scraping away coating.

Most of the polysiloxanes performed better than vinyl in cathodic disbondment testing, and several zinc rich primer systems performed well with no blistering, as seen in Figure 16. Systems 1-PS2DTM-3C, 5-Z1-PS1, and 5-Z2-PS1 were not tested due to blisters forming during the early stages of immersion testing. Systems 4-PS1DTM-3C, 4-Z1-E1-PS1, 4-E1-PS1, and 5-PS1DTM-2C formed blisters and cracked during the disbondment test resulting in a complete failure.

Notably, coating systems 1-Z1-PS1 and 2-Z1-PS2 did not blister. This is the first time in recent Reclamation testing history that a zinc primer system in immersion service did not blister randomly over the entire pipe when paired with a cathodic protection system. In conjunction with a zinc rich primer, resistive coatings allow a small amount of current to flow through the coating at the weakest point and react with the zinc to form blisters. The polysiloxane topcoat barrier properties minimize the ions entering the coating reducing the current through the coating to the substrate forcing the current flow through the defect. However, the cathodic disbondment test duration was only four months which might not be long enough to assess compatibility with cathodic protection in the long term.

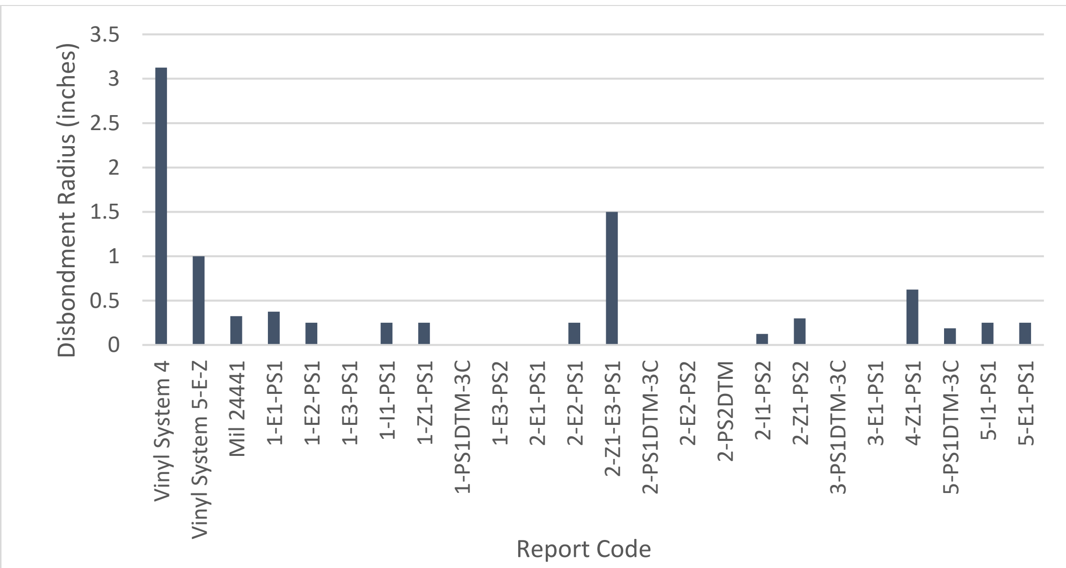

Figure 16. Cathodic disbondment result showing that most polysiloxanes perform similar to the Mil 24441 control and better than the vinyl systems. Three systems were not tested due to blistering in water immersion prior to the cathodic disbondment test and were withdrawn, and four systems had blisters or cracking.

Figure 17 provides the abrasion and erosion resistance results. The abrasion results indicated that most polysiloxanes performed similar to or better than the experimental controls. The weight loss for polysiloxanes ranged from 90 mg to 182 mg, compared to an average of 164 mg for vinyls and 177 mg for Mil Spec 24441. Products from Manufacturers 2 and 5 performed better than the controls. Product PS1 from Manufacturer 1 performed better than the controls, while PS2 did not meet the performance of the controls. Products from Manufacturers 3 and 4 performed similar to the controls.

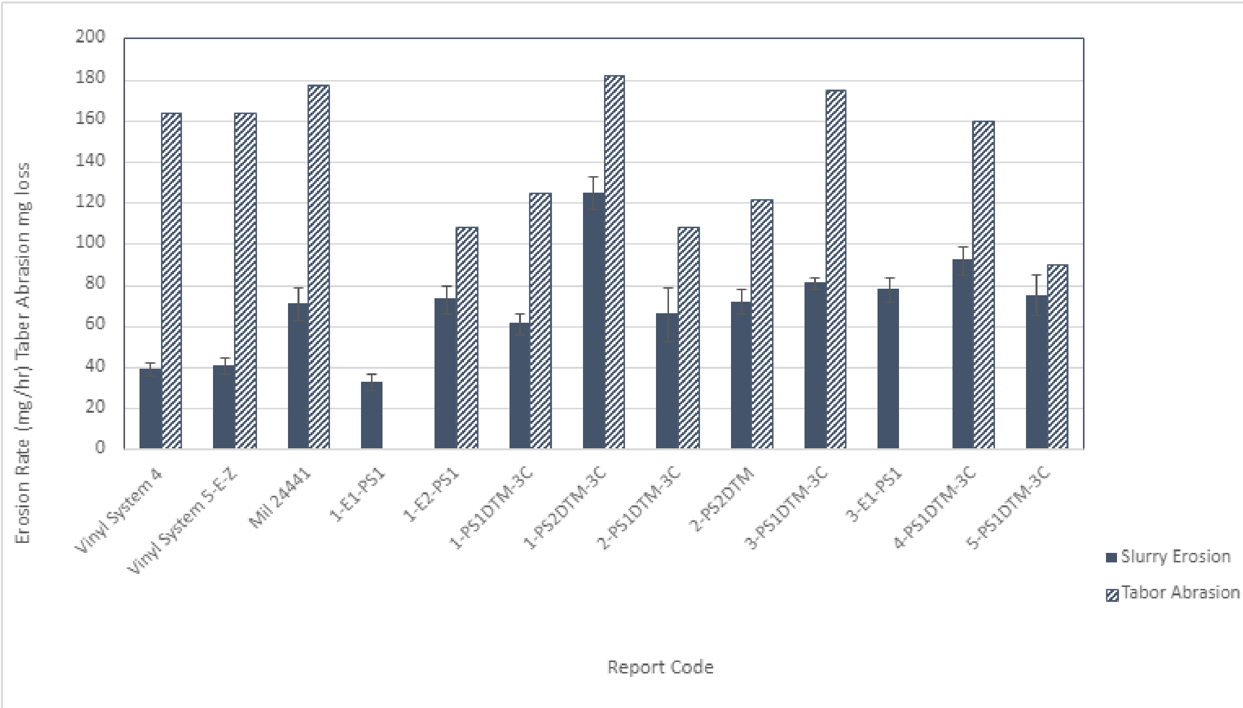

Figure 17. Erosion and abrasion test results. Error bars are included for the slurry erosion results.

The erosion results indicated that polysiloxanes do not meet the performance of the vinyl system experimental controls. The vinyl systems had an average erosion rate of 0.040 g/hr and Mil Spec 24441 had an erosion rate of 0.071 g/hr. Polysiloxane system PS1 from Manufacturer 1 performed slightly better than the Mil Spec 24441, with an erosion rate of approximately 0.06 to 0.07 g/hr. The first experiment for PS1, 1-E1-PS1, was 0.033 g/hr, but the results could not be reproduced in subsequent experiments. Manufacturer 2 erosion rates were 0.066 g/hr for PS1 and 0.072 g/hr for PS2. Manufacturer 5 systems performed similar to Mil Spec 24441, with an average erosion rate of 0.075 g/hr. Manufacturers 3 and 4 erosion results did not meet the performance of Mil Spec 24441, nor did PS2 from Manufacturer 1.

Most impact resistance results did not meet the performance of the vinyl System 5-E-Z, which is 100 inch-lbs, and the results varied greatly across all polysiloxane manufacturers as shown in Figure 18. The zinc-primer containing system for Manufacturer 1 had the highest impact resistance at 160 inch-lbs, and systems from Manufacturers 4 and 5 were very brittle, with impact resistance values ranging from 10 to 20 inch-lbs. The vinyl System 4 and Mil Spec 24441 also had very low impact resistance values in the same range of 10 to 20 inch-lbs. The desired impact resistance is greater than 100 inch-lbs, ideally 160 inch-lbs or above.

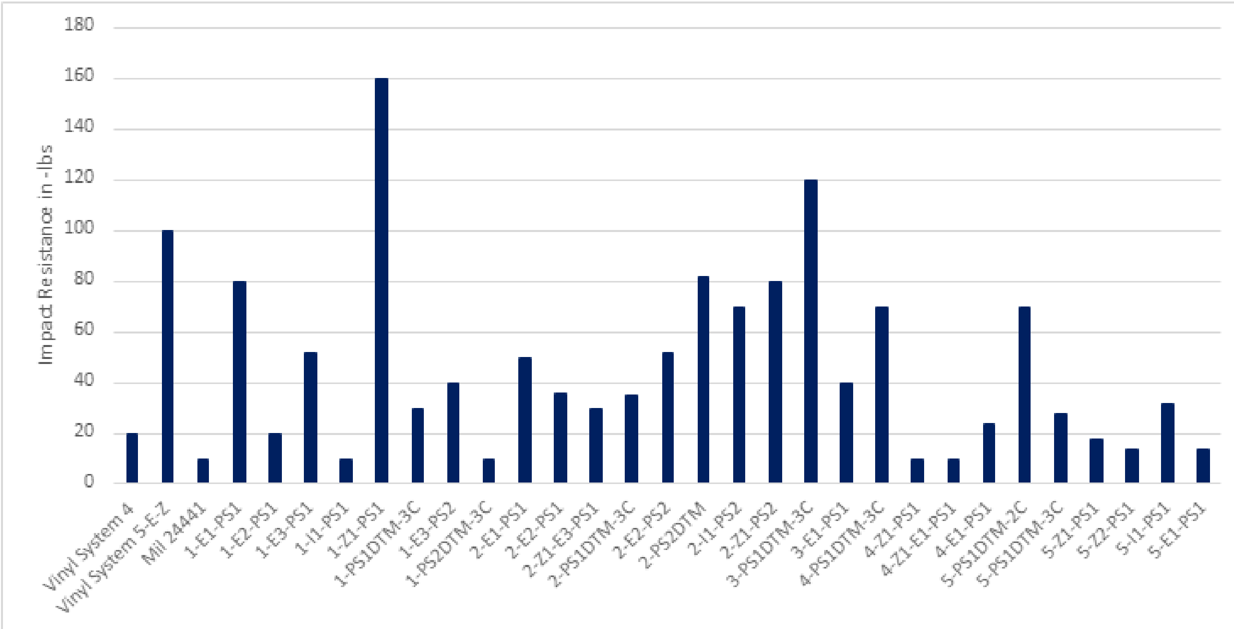

Figure 18: Direct impact results.

The adhesion properties of the polysiloxane coatings had variability in overall strength and in change from dry to wet adhesion. The average dry adhesion strength ranged between 1,000 pounds per square inch (psi) and 1,500 psi which is equivalent to the control coatings. Most of the system with a zinc rich primer had poor results for the adhesion pull-off with the average for those systems falling between 500 psi and 1,000 psi. The zinc rich primer for Manufacturer 1 was the exception and had excellent results for the adhesion pull-off with an average greater than 1500 psi. Systems with an epoxy primer or which were applied DTM had fair to good adhesion strength with the average adhesion pull-off value for the systems being greater than 1,000 psi. The polysiloxanes did appear to have a slight decrease in wet adhesion strength, but still obtained a fair to good performance. The exceptions to this were systems 1-E3-PS1, 2-PS2DTM, 2-I1-PS2, 4-E1-PS1, and four of the Manufacturer 5 coating systems which had an increase in adhesion strength from dry to wet adhesion. Wet adhesion was not tested on coating systems 2-E1-PS1, 4-Z1-PS1, 4-Z1-E1-PS1, 5-Z1-PS1, and 5-Z2-PS1 due to their failure in water immersion testing after the formation of blisters or other defects.

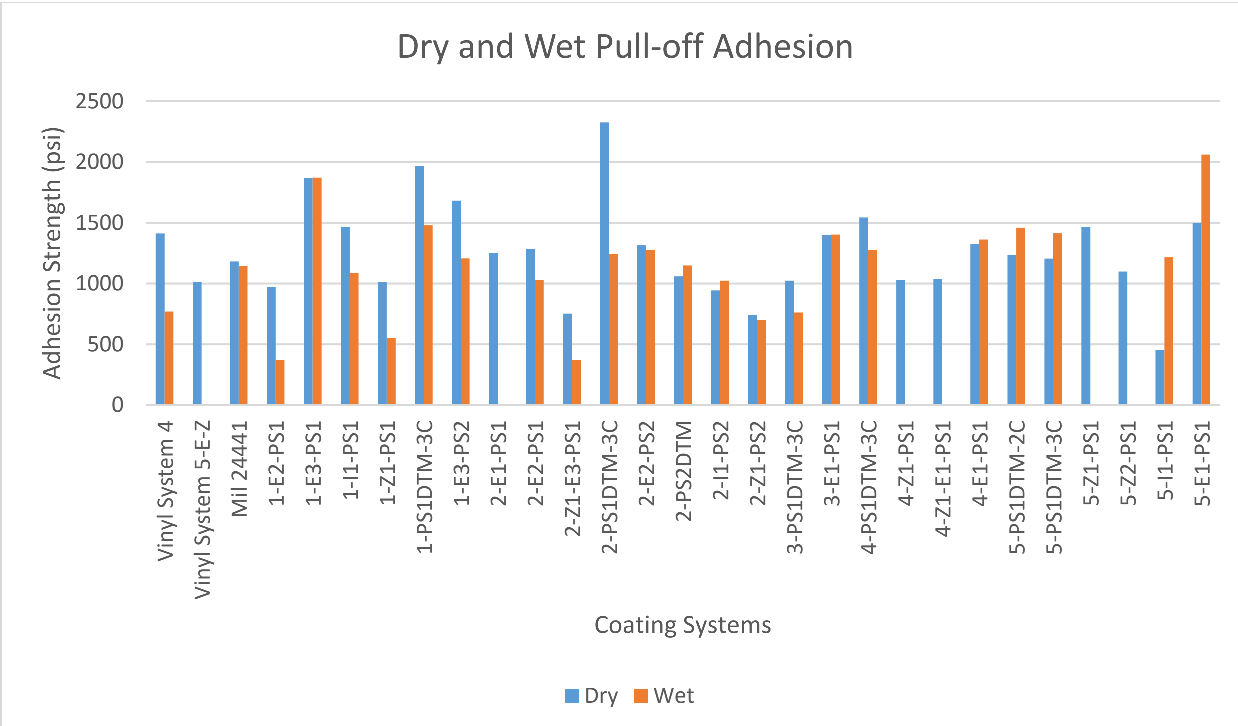

Figure 19. Average pull-off strength (psi) for dry and wet adhesion test results.

The epoxy primer and DTM systems had excellent knife adhesion. These systems received a rating score between 8 and 10 per the rating criteria in ASTM D6677. The systems with a zinc rich primer received a score between 4 and 6, which was the same as vinyl coatings. Overall, the polysiloxanes performed equivalent to or better than the vinyl systems and Mil Spec 24441 epoxy coating in knife adhesion.

3.2 Reproducibility Study and Multi-Coat Study

3.2.1 Reproducibility Study

The primary goal of the reproducibility study was to verify the performance of the polysiloxanes by using different batch numbers and having a different coating applicator. Manufacturers 1's products were selected for the reproducibility testing utilizing the DTM application and zinc rich primers. The EIS data in Appendix D shows minimal change in the barrier properties of the reproducible systems also shown in Figure 20. While the EIS data was consistent, other properties evaluated in the reproducibility study were not consistent.

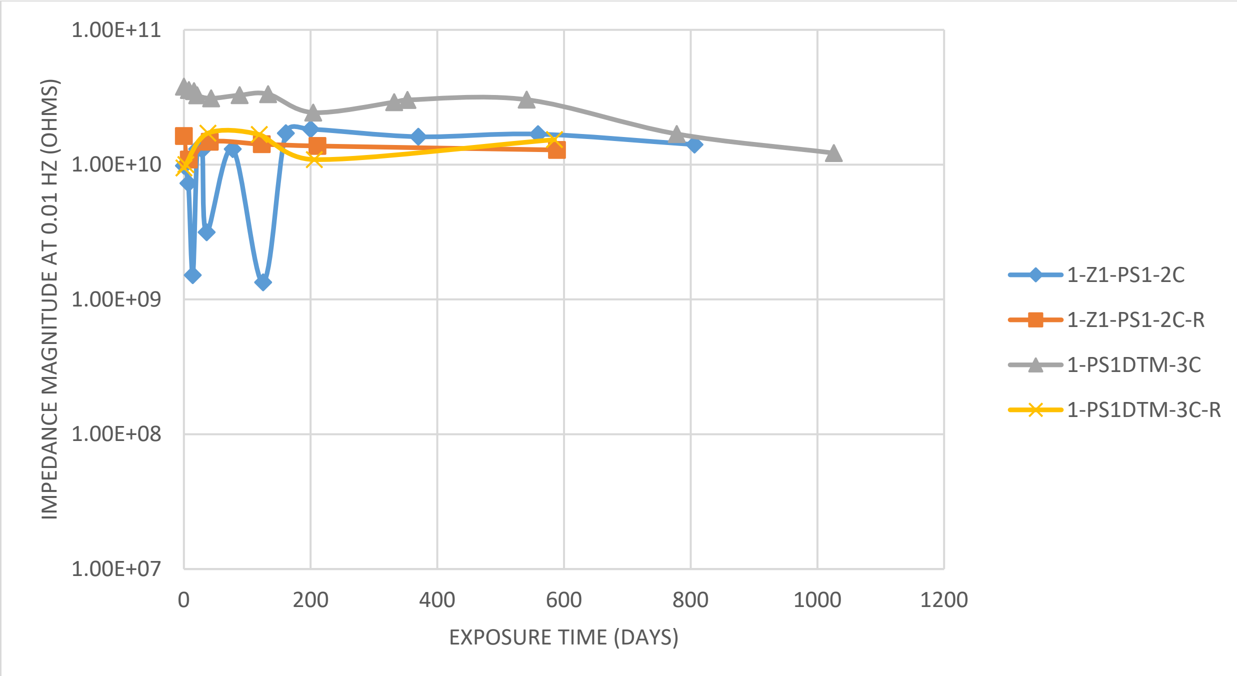

Figure 20: EIS impedance at 0.01 Hz over time for reproducibility study.

Appendix E contains the results for all tests for the reproducibility study. There was no difference in undercutting resistance for water immersion for either coating system, and all panels had no undercutting. The original application of 1-Z1-PS1 provided excellent undercutting resistance in cyclic testing. However, the repeat application showed the undercutting resistance to be lower than expected as shown in Figure 21. The zinc rich primer was sanded on the panels for only the reproducibility study. Sanding and application of the polysiloxane topcoat occurred on the same day. This procedure didn't allow the entrapped solvent to escape, and as a result it is hypothesized that this had a negative impact on undercutting causing worse undercutting performance for the reproducibility study than the original application.

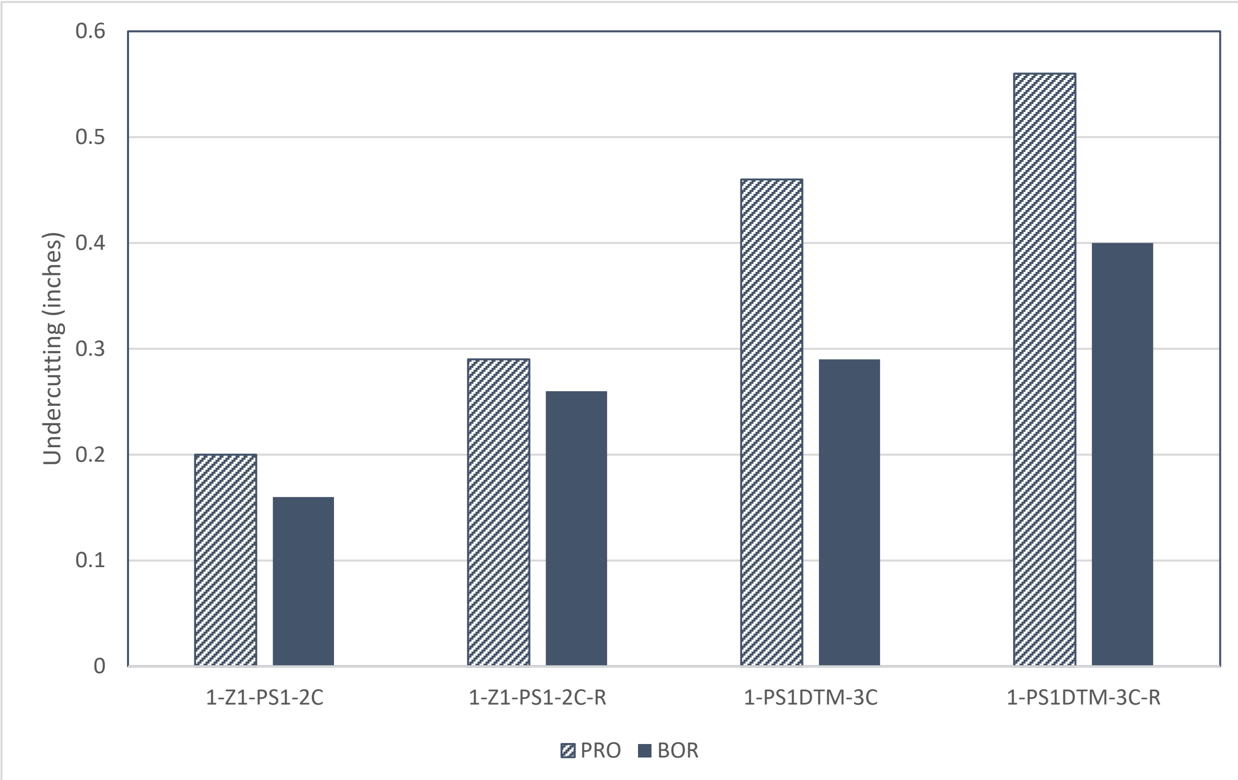

Figure 21: Cyclic test results for PRO and BOR test reproducibility study. Undercutting resistance decreased for both coating systems and both tests.

Some of the physical properties for both coating systems evaluated in the reproducibility study also differed from original testing. Direct impact results were similar between the initial testing and reproducibility results. System 1-Z1-PS1 had a difference of 80 inch-lbs. Coating system 1-PS1DTM-3C had a difference of 15 inch-lbs. The impact resistance of the coating does not appear to be easily reproducible. This test demonstrates that utilizing a zinc rich primer improved the impact resistance significantly. Figure 22 shows the variability in impact resistance.

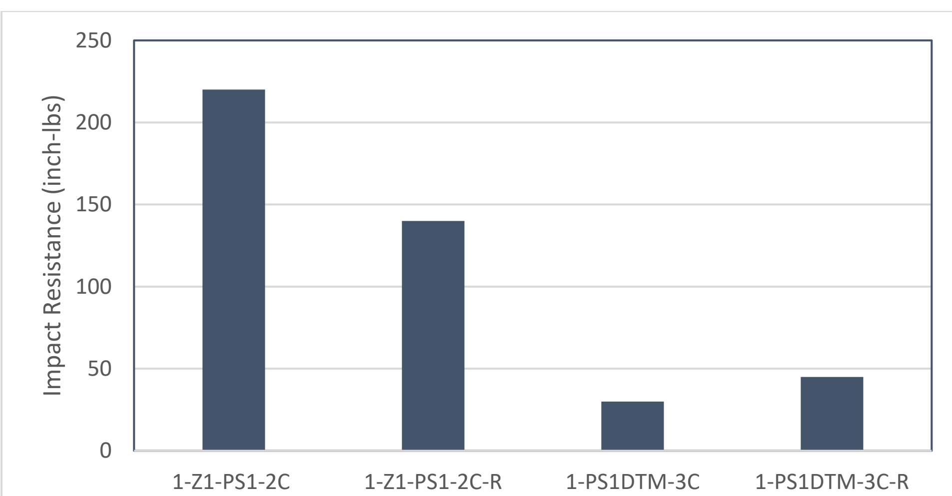

Figure 22. Impact resistance of the reproducibility study.

The dry and wet adhesion results were higher in the repeat study than the original application. The zinc primer had lower cohesive strength than the polysiloxane, and no adhesion failures occurred during testing. There were adhesion issues with the original application of 1-Z1-PS1-2C between the primer and the topcoat with the failure mode as inter-coat adhesion. This was originally thought to have been a compatibility issue, but after reviewing the data, the dry film thickness of the primer was within the manufacturer's recommendation, and sanding was not required. During the application of the reproducibility panels the zinc rich primer was sanded to achieved recommended DFT. In the fall of 2019, a contractor informed Reclamation staff that they sand all the zinc rich primers before applying an intermediate or topcoat to obtain adequate adhesion, and if sanding is not done, then it results in poor adhesion. The results of repeat application 1-Z1-PS1-2C-R, support that sanding between a zinc rich primer and intermediate or topcoat of polysiloxane provided better adhesion between the two coatings. Undercutting resistance for the reproducibility study was worse for the panels that had the zinc rich primer sanded. However, it was hypothesized that this was a result of not allowing proper time for entrapped solvent to off-gas and not caused by the sanding. From these studies, it was found that it is important not to exceed the manufacturer's thickness and sanding should be required between the zinc rich primer and the topcoats. If the thickness is exceeded, the manufacturer should be consulted to know how long to wait to topcoat to remove as much entrapped solvent out of the primer. These are important lessons learned for future applications.

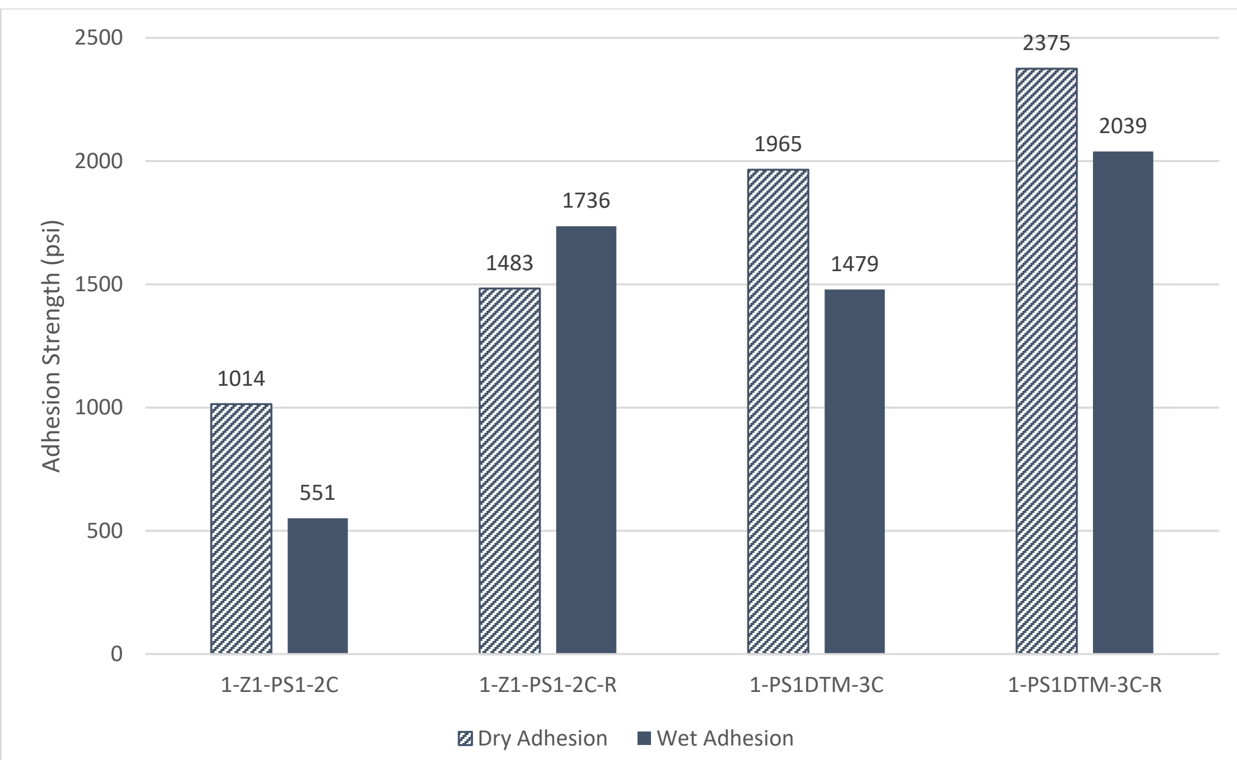

Figure 23: Dry and wet pull-off adhesion results for reproducibility study. Adhesion pull-off values increased for both systems.

3.2.2 Two, Three, or Four Coat System

This study used products from Manufacturers 1 and 2 to evaluate the impact of the number of polysiloxane coats on corrosion protection and other properties of the coating system. Coating systems consisted of a zinc rich primer with either one, two, or three coats of polysiloxane for both manufacturer products. Figure 24 and Figure 25 show the EIS impedance results at 0.01 Hz for all coating systems. The EIS results show that the magnitude increases as the thickness of the polysiloxane increases, which is to be expected. The phase angle slightly decreases during the first seven months of exposure. Since polysiloxanes require moisture to cure, it is assumed that the amino silane doesn't react entirely before subsequent coats are applied resulting in slight increase in impedance once placed in immersion. Currently this data set has 18 months of exposure and is still exhibiting capacitive behavior with a high impedance magnitude for all two, three, and four-coat systems evaluated from both manufacturers. These polysiloxane systems are currently outperforming vinyl System 5-E-Z when we compare the same exposure times for corrosion resistance.

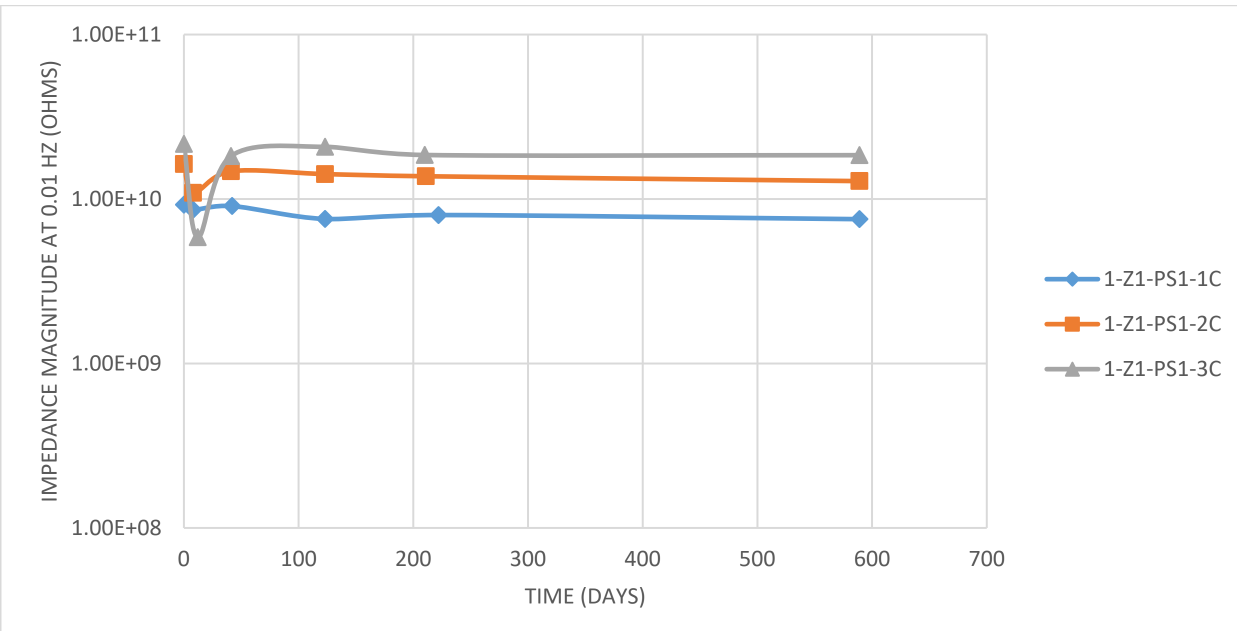

Figure 24: EIS results at 0.01 Hz for 1-Z1-PS1 coating systems. As the number of coats increases so does the impedance.

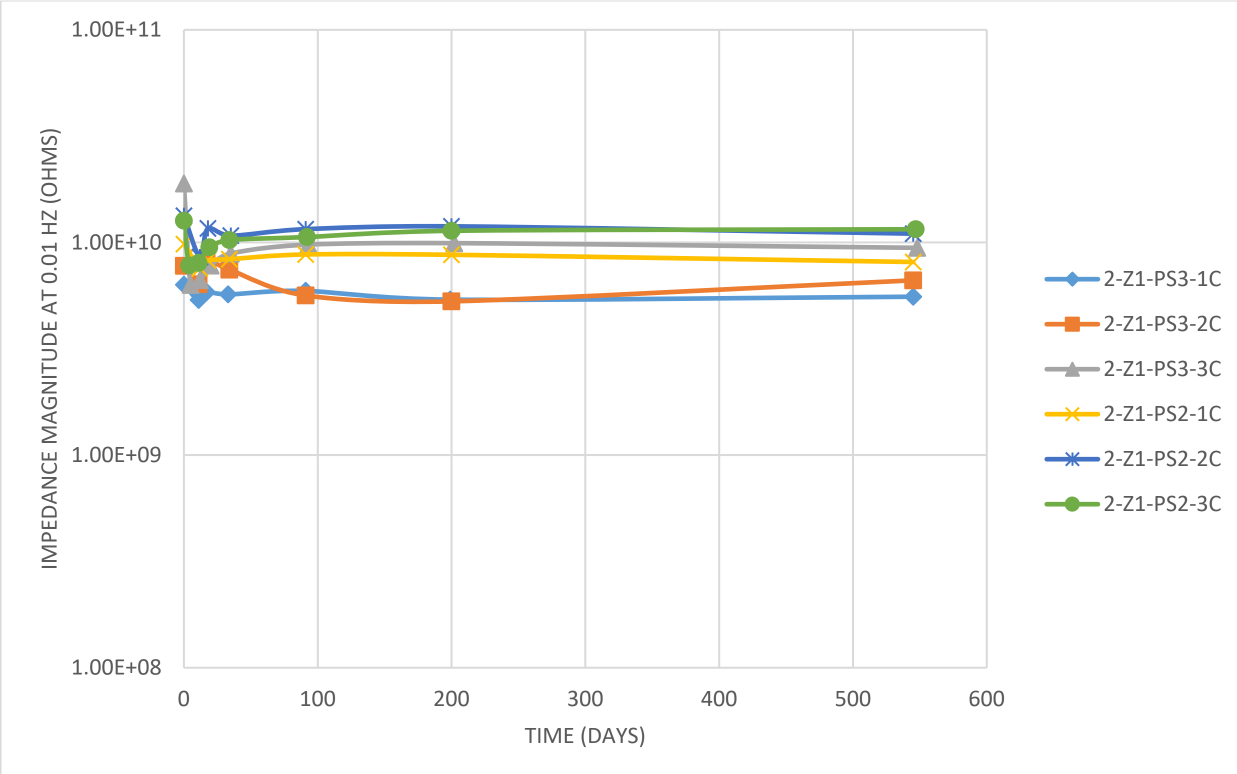

Figure 25: EIS results at 0.01 Hz for 2-Z1-PS2 coating systems. As the number of coats increases the overall impedance of the system increases.

Appendix E provides the other test results for the two, three, and four coat systems. Figure 26 shows the undercutting results. Manufacturer 1 provided mixed results regarding the relationship between the number of coats and undercutting resistance. In PRO testing the undercutting increased as the number of coats increased, whereas in BOR cyclic testing the undercutting decreased as the number of coats increased. It was also observed that the undercutting resistance of Manufacturer 1's systems for this portion of the study had an overall worse performance for undercutting resistance. The zinc rich primer exceeded the recommended DFT. This likely resulted in solvent entrapment and other stresses reducing the undercutting resistance of the coating. This information combined with the results from the reproducibility study suggest the best solution is to blast and remove the zinc coating and repeat the application.

For Manufacturer 2, the undercutting increased as the number of coats increased for all six systems. This is likely attributed to the polysiloxane cure level. It is likely the intermediate coats for the three-coat and four-coat systems were unable to fully cure and did not provide the same protections as the single coat system which was able to cure fully.

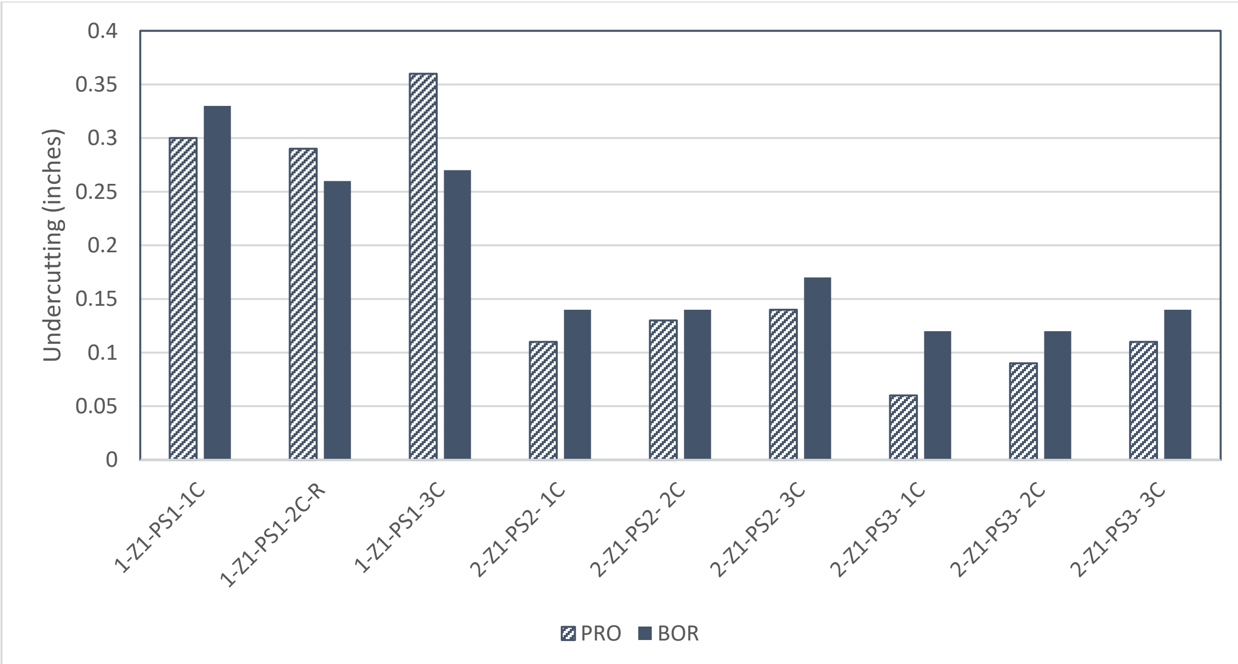

Figure 26: Cyclic test results showing the PRO and BOR undercutting resistance for two, three, and four coat systems.

The direct impact resistance decreases as the coating thickness increases from the number of coats. Polysiloxanes are already a considered brittle and low resistance to direct impact. Manufacturer 1 systems had higher impact resistance than Manufacturer 2 as shown in Figure 27. The two-coat system provided the best impact resistance for Manufacturer 1 products, even though it is only a 6-10 mil thick coating system while the two and three coat systems had equal performance for Manufacturer 2. It appears that there may be increased stress building within the coating as the thickness increases, causing it to become more brittle. This is likely caused by an increase in internal stresses from higher crosslinking.

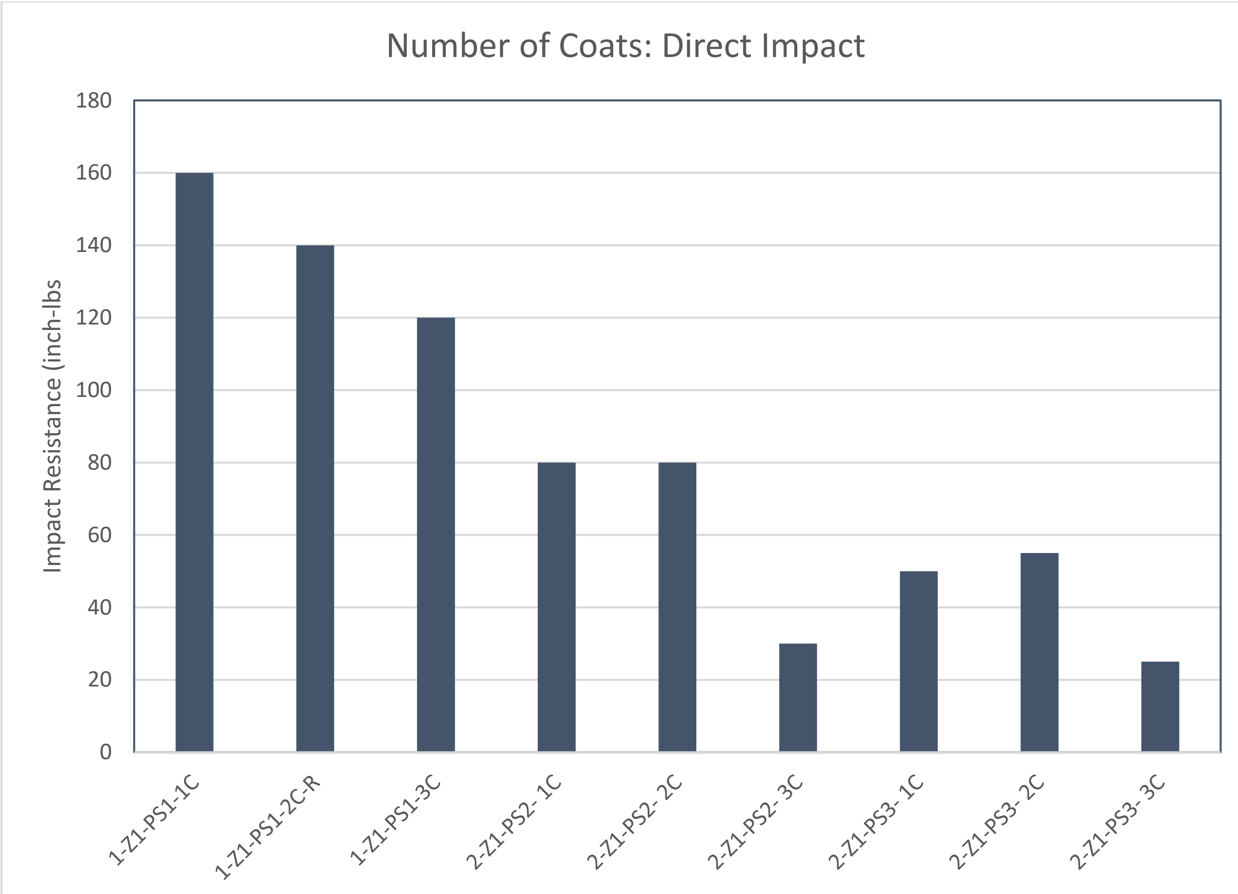

Figure 27: Direct impact results. Manufacturer 1 had higher impact resistance than Manufacturer 2. As the number of coats increased the impact resistance decreased.

The pull-off adhesion testing results varied widely for Manufacturer 1 and were consistent for Manufacturer 2. Failure mechanism was primarily cohesion failure indicating the internal coating strength was less than the adhesion strength of the coating to the steel substrate. For Manufacturer 1, as the number of coats increased for the dry adhesion pull-off strength decreased while the wet-adhesion strength increased. It is undetermined why Manufacturer 1 systems had such variability. Neither dry nor wet adhesion strength for Manufacturer 2 coating systems were impacted by the number of coats. All systems were approximately within 100 psi of each other.

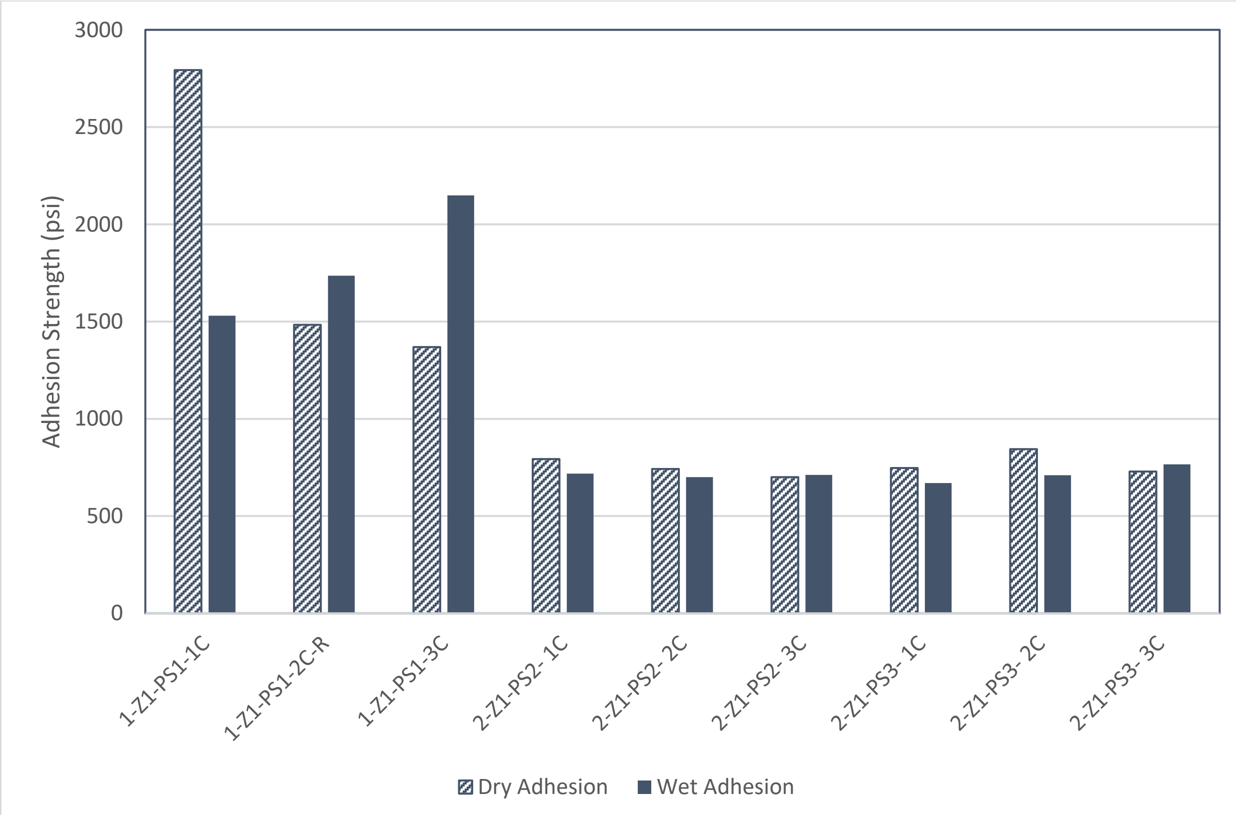

Figure 28: Average dry and wet pull-off adhesion results for two, three, and four coat system study.

3.2.3 Standard versus Fast Dry Formulation

This study also compared the fast dry formulation, PS3, versus standard formulation, PS2, for Manufacturer 2's polysiloxane. The fast dry formulation is designed to be able to cure at lower temperatures and percent RH. The test results found that there was little performance difference between the standard product and fast dry product. Both polysiloxane PS2 and PS3 provided similar barrier properties indicated by EIS with an impedance magnitude around 1×10^{10} ohms at 0.01 Hz as shown in Figure 25. Both formulations provided excellent results in immersion testing with no undercutting occurring. The only noticeable difference was observed in cyclic testing and impact resistance. The fast-dry formulation had better performance against undercutting in both cyclic tests compared to the standard formulation as seen in Figure 26. The likely reason for this is the fast-dry formulations are designed to cure at lower humidity and temperatures. The fast-dry product was able to reach a more uniform and complete cure in the arid environment of Colorado where they were applied. The fast dry formulation had lower impact resistance due to being more brittle from having a higher crosslink density caused by a more complete and the faster cure time would likely increase the internal stresses of the coating.

3.3 Cure Study

To assess the impact of cure time on barrier properties, EIS was performed on polysiloxane system 2-PS2DTM-2C after the following cure durations: DTH, 72-hour, 1 week, 2 weeks, and 8 weeks. EIS measurements are shown as the impedance magnitude and phase angles of the coatings at 0.01 Hz to monitor the total impedance for the system as well as the capacitive and resistive behavior changes. According to manufacturer data sheets, full cure for atmospheric service is seven days at 77 °F, 50 percent RH for both the standard and fast dry formulations before being placed into service. Appendix F provides a summary of the EIS results for the various cure durations.

Figure 29 and Figure 30 show the impedance magnitude and phase angle, respectively, at 0.01 Hz for all cure durations over the testing period. All cure durations showed an initial drop in impedance during the first few days after being placed in immersion testing, but then began to increase in impedance values. After three weeks in immersion testing, all cure durations had an impedance value above 5.0 x 10^9 ohms and displayed capacitive behavior. After nine months in water immersion testing, all cure durations had stabilized and had the same impedance value of 1.2 x 10^{10} ohms and similar phase angles of approximately -60 degrees. This shows the coatings will reach the same properties even when placed into immersion service as soon as the system is DTH. These results suggest the coating will provide excellent corrosion protection, even at this short cure duration. Additional evaluations could help to determine if the cure duration affects the long-term corrosion protection.

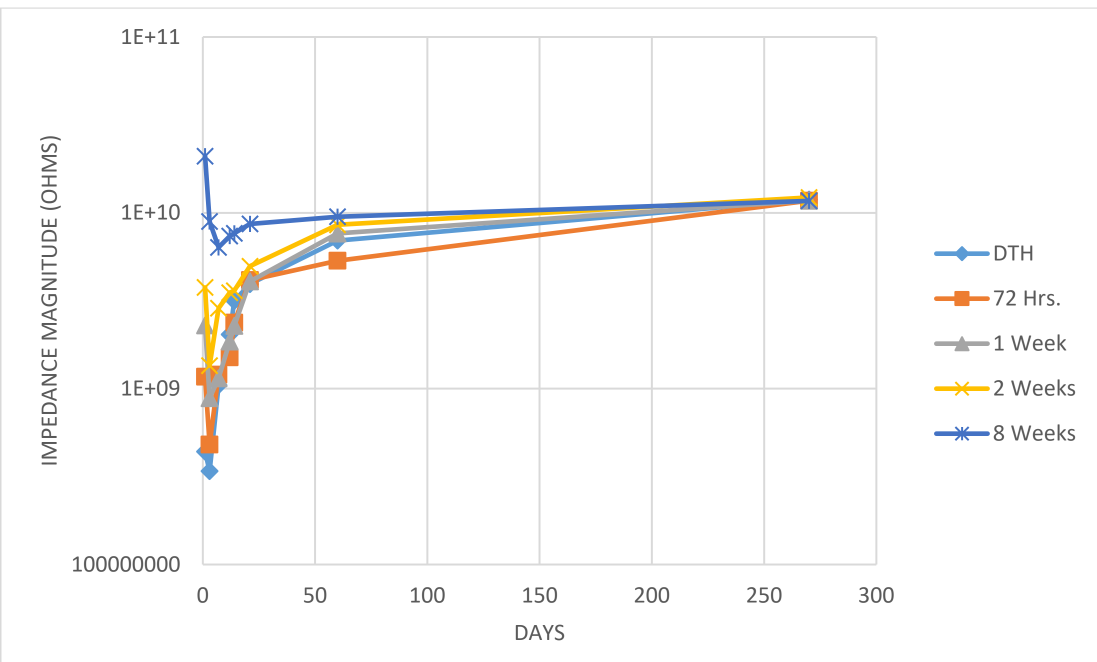

Figure 29: Impedance magnitude at low frequency over the duration of testing. The cure durations have different starting impedance values but all level out to the same impedance value at approximately 250 days.

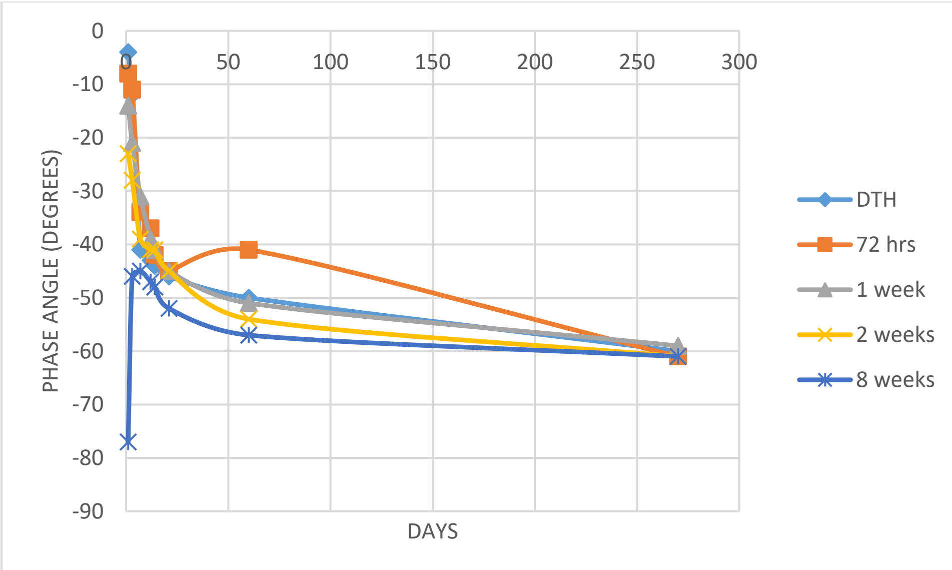

Figure 30: Phase angle of all cure durations during the evaluation period. The initial phase angles are different for all cure durations, but all converge to approximately -60 degrees after 250 days showing pseudo capacitive behavior.

The cure study also investigated the effect cure duration has on adhesion strength; panels of each cure duration were placed in the immersion tank and tested after 9 months from initial application. ASTM D4541 pull-off adhesion was used to determine the average adhesion, from six data points, and results are summarized in Table 5 which are categorized as wet adhesion. The average pull-off adhesion for each cure duration exceeded 1000 psi and there was no significant change between the cure durations. The failure mechanisms also remained the same for the different cure schedules, with the main type of failure being approximately 55 percent adhesion, followed by cohesion failure at approximately 35 percent, and glue failure at 10 percent of the dolly area. The results indicated that cure time for the epoxy polysiloxanes evaluated in this study has little to no impact on adhesion strength.

Table 5: Direct pull-off wet adhesion results for cure study.

Cure Duration	**Adhesion strength** (psi)	**Standard Deviation**	**Type of Failure**		
			Adhesion	Cohesion	Glue
DTH	1218	219	59	30	11
72 Hrs	1161	101	58	32	10
1 Week	1165	202	55	35	10
2 Weeks	1387	220	52	36	12
8 Weeks	1246	246	53	35	12

EIS and adhesion strength test results indicate that there are no significant impacts from cure duration on barrier properties or adhesion. For best results, polysiloxane coatings should cure for eight weeks. However, EIS data shows that the coatings continue to cure while in immersion service and can still provide excellent corrosion protection and adhesion testing shows the coating can obtain adequate adhesion strength before the full cure time is reached. Because of this, return to service after one week of curing will provide adequate corrosion protection and coating adhesion to the substrate.

4. Field Evaluation of Epoxy Polysiloxane

In November 2019, Reclamation researchers inspected the three-to-four–year-old epoxy polysiloxane coating on TVA's Fontana Dam, NC, spillway radial gates. Between 2015 and 2016, all four radial gates were coated with a three-coat system of an epoxy primer, epoxy with glass flake intermediate coat, and an epoxy polysiloxane topcoat. The coating system utilized by TVA was duplicated for laboratory testing and is system 1-E2-PS1 in this report. During the field inspection, Reclamation staff performed a visual inspection of the gates, shown in Figure 31, and field EIS testing.

The visual inspection of the gates showed that the coating was in overall good condition. All impedance magnitude values recorded were above 1 x 10^9 ohms at 0.1 Hz, indicating that the three-part coating system is providing excellent corrosion protection in the areas tested. However, microcracking was observed in areas where the coatings were applied too thick, and drips, runs, and sags were also visible as shown in Figure 32 through Figure 34. Microcracking was also observed on the seal clamp bars. These flaws were likely from coating application errors such as solvent entrapment from applying the coating too thick. As the solvent evaporates in a cured coating, increased internal stresses can cause microcracking. Additionally, areas of corrosion caused by abrasion from the wire ropes were also noted. Details can be found in Reclamation Technical Memorandum 8540-2020-01. The gates at Fontana Dam show that in the short term polysiloxanes have a successful performance in a field service environment. Routine inspections to monitor the coating's performance should be conducted and will provide useful information on the coating's service life.

Figure 31: Upstream side of the four radial gates at Fontana Dam.

Figure 32: Large drip from application. Pitted area of steel with no defects to the coating or corrosion observed.

Figure 33: Sag of coating cause by excess buildup of coating during application. Cracking and corrosion have occurred.

Figure 34: Micro cracking in coating, caused by excess film build up, on seal-clamp bars. Coating on bolts has become damaged and corrosion is present

Conclusion

USACE and Reclamation have severe environments that require higher durability coatings. Coatings in these environments need to be able to withstand impact from debris and erosion from sediment in flowing water while providing corrosion protection. Polysiloxanes were shown in a previous study to have the potential to replace the vinyl coatings systems typically used in these environments. The current study presents comprehensive laboratory evaluations of commercially available polysiloxanes as compared to vinyl control systems. Experiments evaluated reproducibility, the effect of the number of coats applied, the effect of a fast dry formulation, and the effect of cure duration prior to placing in water immersion. An inspection was also performed on an in-service polysiloxane coating system after three to four years in fluctuating immersion and atmospheric exposure on radial gates. Findings and takeaways from this research are listed below.

- Polysiloxane 1 from Manufacturers 1 and polysiloxane 2 and 3 from Manufacturer 2 provided "excellent" corrosion protection in cyclic environments and water immersion. The undercutting resistance can be improved using zinc rich epoxy primer systems.
- Most polysiloxanes evaluated provided equivalent or better Taber abrasion resistance but worse erosion resistance than the vinyl systems.
- Only two polysiloxane systems had equivalent or better impact resistance than vinyl System 5-E-Z.
- Current polysiloxane formulations do not have the same durability as vinyl and require improved abrasion, erosion, and impact resistance to be considered an equivalent product.
- Current polysiloxane formulations meet the same levels of durability as the epoxy Mil Spec. 24441 formulation but provide better barrier properties.
- Polysiloxanes paired with zinc rich epoxy primers significantly reduced the undercutting to provide equivalent corrosion protection to the vinyl System 5-E-Z which contains a zinc rich vinyl primer.
- One coat of zinc rich primer and one coat of polysiloxane for Manufacturer 1 and 2 coating systems provided "excellent" corrosion protection compared to one coat of zinc rich primer with two or three polysiloxane coat systems.
- There is no difference in performance between the standard formulation and fast dry formulation for Manufacturer 2's polysiloxane.
- Manufacturer 1 polysiloxane coatings placed in water immersion exposure after curing to DTH provide equivalent EIS and adhesion data to coatings cured for up to 8 weeks. Therefore, this short cure duration does not appear to have a detrimental impact on resulting properties.
- Field evaluation of a polysiloxane coating system applied on the gates at TVA's Fontana Dam, NC, exhibited successful performance after three to four years of exposure to field conditions. A few areas of the gates showed drips and micro-cracking, both appear to be due to applying coatings too thick.

Recommendations

While some properties, namely those related to durability, need improvement, laboratory screening found that epoxy polysiloxanes provide excellent corrosion protection in certain immersion service environments. Long-term laboratory testing and field analysis will need to be demonstrated before polysiloxanes can see widespread application. However, based on the laboratory test result, the recommendations for use of polysiloxane coatings within Reclamation are given below:

- Vinyl coatings are still the recommended coating for impacted immersion service.
- Polysiloxanes with a zinc rich primer are recommended for atmospheric service in accordance with manufacturer recommendations.
- Polysiloxane 1 from Manufacturer 1 and polysiloxanes 2 and 3 from Manufacturer 2 with the manufacturer recommended epoxy primer are suitable for use on Reclamation structures in immersion service and fluctuating immersion service if there is no risk of impact damage, erosion, or abrasion. This will limit their widespread use until durability properties can be improved. Polysiloxanes from Manufacturers 3, 4, and 5 are not recommended for this service condition.
- In locations with relative humidity consistently below 50 percent, such as the arid western United States, fast-dry products are recommended.
- Due to the poor edge retention of polysiloxanes, direct-to-metal application of the tested products is not recommended, until manufacturer's address the edge retention issue direct-to-metal applications should not be done.
- A stripe coat should be applied before each coat of polysiloxane for all service conditions.
- A cathodic protection system should be used in conjunction with polysiloxanes in immersion service where feasible.
- Field applications of polysiloxanes should be monitored for corrosion protection and durability performance including long-term data collection.

Future Work

- Epoxy primer/polysiloxane topcoat systems should be scaled-up to field structures for immersion service and annually inspected for long-term corrosion performance.
- Zinc rich epoxy primers are typically not recommended for immersion and fluctuating immersion service. However, this study indicated "excellent" laboratory corrosion protection performance for zinc rich epoxy primers with polysiloxane topcoat. Further laboratory research and field scale-up are needed to validate this performance.
- The effect of sanding a zinc rich primer before applying the polysiloxane should be studied to understand the impact on adhesion and coating compatibility.
- Direct-to-metal application should be reinvestigated if products with improved edge retention become available.
- Partnership opportunities should be explored with manufacturers and other coatings experts to improve overall polysiloxane durability.

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Appendix A – Coatings Rating Criteria

Immersion Testing (Dilute Harrison, Deionized):
Excellent: No visual defects
Good: No blistering, minor rust creep up to ⅛”
Fair: No blistering, moderate rust creep up to ¼”
Poor: Blistering, delamination or rust creep over ¼”

Cyclic Weathering Testing (BOR, Prohesion, FOG):
Excellent: No blistering, minor rust creep up to ⅛”
Good: No blistering, minor-moderate rust creep up to ¼”
Fair: No blistering, moderate rust creep up to ½”
Poor: Blistering, delamination or rust creep > ½”

UV and Condensation Cabinet Cyclic Weathering Testing (QUV)
Excellent: No visual defects
Good: No blistering, no rust creep, minor color change
Fair: No blistering, moderate color/gloss change, chalking, or undercut up to ⅛”
Poor: Any of the following: blistering, delamination, rust creep > ⅛”

EIS (immersion):
Excellent: After 5000 hrs - Minimal degradation < 1 order of magnitude @ 0.01 Hz and > 10^9 ohms
Good: Minimal degradation after 5000 hrs ≤ 2 order of magnitude @ 0.01 Hz and ≥ 10^8 ohms
Fair: Moderate degradation after 5000 hrs ≤ 3 orders of magnitude @ 0.01 Hz and ≥ 10^7 ohms
Poor: Signification degradation after 5000 hrs > 3 orders of magnitude @ 0.01 Hz

Adhesion (initial, dry):
Excellent: ≥ 2,500 psi
Good: ≥ 1,500 psi
Fair: ≥ 1,000 psi
Poor: < 1,000 psi

Wet Adhesion:
Excellent: ≥ 2,000 psi
Good: ≥ 1,000 psi
Fair: ≥ 500 psi
Poor: < 500 psi

Tabor Abrasion (ASTM D4060):
Excellent: < 30 mg loss
Good: < 40 mg loss
Fair: < 100 mg loss
Poor: > 100 mg

Erosion (USBR-5071-2015):
Excellent: < 30 mg/hr average loss
Good: < 50 mg/hr average loss
Fair: < 100 mg/hr average loss
Poor: > 100 mg/hr average loss

Impact:
Excellent: ≥ 160 in-lbs
Good: ≥ 100 in-lbs
Fair: ≥ 50 in-lbs
Poor: < 50 in-lbs

Cathodic Disbondment (ASTM G8)
Excellent: Disbondment radius < ¼”
Good: Disbondment radius < ½”
Fair: Disbondment radius < 1”
Poor: Disbondment radius > 1”

Knife Adhesion Testing (ASTM D6677)
Excellent: ASTM Rating 8.5-10 - Coatings is extremely difficult to remove. Chips up to 0.8 mm by 0.8 mm.
Good: ASTM Rating 6-8 - Coating is difficult or at least somewhat difficult to remove. Chips up to 6.3 mm by 6.3 mm.
Fair: ASTM Rating 3.5-5.5 - Coating chips in excess of 6.3 mm by 6.3 mm, can be remove with light pressure from a knife blade.
Poor: ASTM Rating 0-3 - Coating peels with fingers once started with a knife blade

Appendix B – EIS Data Summary for Coating Performance Evaluation

EIS Data summary for all tested systems. Data includes initial, 1 month, 7 months, 1 years, 1.5 years, and most recent data point collection.

	Initial @ 0.01Hz				1 month @ 0.01Hz				7 months @ 0.01Hz				1 year @ 0.01Hz				1.5 years @ 0.01Hz				X years @ 0.01Hz				X=	Notes
Report Code	|Z|	Phase	Coatings Behavior	OCP	|Z|	Phase	Coatings Behavior	OCP	|Z|	Phase	Coatings Behavior	OCP	|Z|	Phase	Coatings Behavior	OCP	|Z|	Phase	Coatings Behavior	OCP	|Z|	Phase	Coatings Behavior	OCP		
Vinyl System 4	2.7E+09	-76	cap	0.45	4.9E+09	-72	cap	-0.11	5.1E+09	-76	cap	0.042	1.9E+09	-42	both 2nd	-0.179	8.9E+09	-82	cap	0.426	1.4E+09	-41	mixed	-0.125	6 years	Blisters formed, noticed at 6 years
Vinyl System 5EZ	6.5E+09	-79	cap	0.305	5.1E+09	-81	cap	0.231	3.2E+09	-79	cap	-0.306	1.7E+09	-58	cap	0.111	2.5E+07	-3	resist	-0.774	1.2E+06	-1	resist	-0.35	6 years	Surface looks textured
Mil 24441	2.5E+09	-29	resist	0.136	4.2E+08	-11	resist	0.058	8.5E+08	-19	resist	-0.123	N/A	N/A	N/A	N/A	9.5E+08	-16	resist	-0.168	1.3E+08	-8	resist/ ind	-0.147	2 years	
1-E1-PS1	2.7E+10	-58	mixed	0.067	6.0E+09	-34	mixed	0.014	4.1E+09	-46	mixed	-0.051	8.0E+09	-39	mixed	-0.066	N/A	N/A	N/A	N/A	3.5E+09	-29	mixed	-0.025	5 years	1 yr is actually 15 months
1-E2-PS1	6.1E+09	-42	mixed	-0.031	6.7E+09	-48	mixed	-0.015	9.3E+09	-45	mixed	-0.025	6.6E+09	-36	mixed	-0.058	9.9E+09	-45	mixed	0.002	6.0E+09	-35	mixed	0.007	2 years	
1-E3-PS1	4.4E+10	-73	cap	-1.605	2.4E+10	-64	cap	-0.449	2.1E+10	-67	cap	0.598	1.6E+10	-67	cap	-0.038	1.2E+10	-76	cap	-0.75	1.0E+10	-69	cap	-0.183	3 years	
1-I1-PS1	9.0E+09	-72	cap	0.295	7.0E+09	-73	cap	0.609	1.1E+10	-73	cap	-1.04	N/A	N/A	N/A	N/A	1.1E+10	-70	cap	0.193	8.8E+09	-65	cap	0.26	2 years	
1-Z1-PS1-2C	9.8E+09	-47	mixed	0.027	1.3E+10	-58	mixed	0.34	1.8E+10	-71	cap	0.167	1.6E+10	-66	cap	-0.09	1.7E+10	-68	cap	0.047	1.4E+10	-61	cap	0.124	2.25 years	
1-PS1DTM-3C	3.8E+10	-83	cap	-0.868	3.1E+10	-84	cap	-0.462	2.4E+10	-51	mixed	0.042	3.0E+10	-81	cap	0.68	3.0E+10	-80	cap	-0.276	1.2E+10	-32	mixed	-0.342	3 years	
1-E3-PS2	2.6E+10	-72	cap	-0.09	1.6E+10	-60	cap	-0.568	1.0E+10	-59	mixed	0.195	6.6E+09	-56	mixed	-0.076	5.2E+09	-57	mixed	-0.347	4.0E+09	-55	mixed	-0.111	3 years	
1-PS2DTM -3C	1.8E+07	-2	resist	0.014	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	3 weeks	Blisters formed immediately
2-E1-PS1	2.7E+09	-35	mixed	-0.096	4.6E+08	-10	resist	-0.062	6.8E+08	-16	resist	-0.137	4.8E+08	-13	resist	-0.204	N/A	N/A	N/A	N/A	8.7E+08	-16	resist	0.058	5 years	1 small blister after 5 years
2-E2-PS1	3.1E+09	-31	mixed	-0.112	6.0E+08	-10	resist	-0.186	6.3E+08	-10	resist	-0.154	4.5E+08	-10	resist	-0.209	5.6E+08	-10	resist	-0.179	3.4E+08	-13	resist	-0.139	3 years	
2-Z1-E3-PS1	5.2E+08	-8	resist	-0.926	7.0E+08	-12	resist	-0.941	7.2E+08	-15	resist	-0.868	4.2E+08	-11	resist	-0.777	N/A	N/A	N/A	N/A	4.3E+08	-13	resist	-0.543	2.25 years	
2-PS1DTM -3C	2.9E+08	-6	resist	0.003	1.4E+08	-6	resist	-0.06	2.2E+08	-5	resist	-0.047	2.7E+08	-6	resist	-0.121	2.4E+08	-8	resist/ tail	-0.12	2.2E+08	-10	resist/ 2nd	-0.083	3 years	
2-E2-PS2	1.2E+10	-59	mixed	-0.21	5.4E+09	-64	cap	-0.112	5.1E+09	-65	cap	0.148	4.6E+09	-63	cap	-0.006	3.0E+09	-59	mixed	-0.027	3.9E+09	-55	mixed	-0.045	3 years	
2-PS2DTM-3C	3.1E+10	-80	cap	-0.966	2.3E+10	-72	cap	-0.66	1.8E+10	-69	cap	0.27	1.8E+10	-63	cap	0.045	2.0E+10	-67	cap	-0.209	1.6E+10	-61	cap	-0.121	3 years	
2-I1-PS2	4.0E+09	-58	mixed	0.098	4.8E+09	-70	cap	0.26	4.5E+09	-64	cap	0.066	3.8E+09	-55	mixed	-0.176	4.5E+09	-61	cap	-0.169	3.7E+09	-52	mixed	-0.121	2.25 years	
2-Z1-PS2- 1C	9.8E+09	-70	cap	0.207	8.3E+09	-71	cap	0.169	8.8E+09	-73	cap	0.045	N/A	N/A	N/A	N/A	8.1E+09	-72	cap	-0.011	N/A	N/A	N/A	N/A	1.5 years	
2-Z1-PS2- 2C	1.3E+10	-68	cap	0.624	1.1E+10	-69	cap	-0.064	1.2E+10	-72	cap	0.01	N/A	N/A	N/A	N/A	1.1E+10	-71	cap	0.168	N/A	N/A	N/A	N/A	1.5 years	
2-Z1-PS2- 3C	1.3E+10	-67	cap	-0.31	1.0E+10	-67	cap	-0.132	1.1E+10	-71	cap	0.433	N/A	N/A	N/A	N/A	1.2E+10	-69	cap	-0.008	N/A	N/A	N/A	N/A	1.5 years	
2-Z1-PS3- 1C	6.3E+09	-64	cap	-0.622	5.7E+09	-68	cap	0.035	5.4E+09	-66	cap	0.002	N/A	N/A	N/A	N/A	5.6E+09	-62	cap	-0.184	N/A	N/A	N/A	N/A	1.5 years	
2-Z1-PS3- 2C	7.8E+09	-64	cap	0.18	7.4E+09	-67	cap	-0.266	5.3E+09	-63	cap	-0.172	N/A	N/A	N/A	N/A	6.6E+09	-62	cap	-0.254	N/A	N/A	N/A	N/A	1.5 years	

	Initial @ 0.01Hz				1 month @ 0.01Hz				7 months @ 0.01Hz				1 year @ 0.01Hz				1.5 years @ 0.01Hz				X years @ 0.01Hz				X=	Notes
Report Code	|Z|	Phase	Coatings Behavior	OCP	|Z|	Phase	Coatings Behavior	OCP	|Z|	Phase	Coatings Behavior	OCP	|Z|	Phase	Coatings Behavior	OCP	|Z|	Phase	Coatings Behavior	OCP	|Z|	Phase	Coatings Behavior	OCP		
2-Z1-PS3- 3C	1.9E+10	-82	cap	-0.893	8.9E+09	-62	cap	-0.032	9.9E+09	-64	cap	-0.018	N/A	N/A	N/A	N/A	9.4E+09	-60	cap	-0.223	N/A	N/A	N/A	N/A	1.5 years	
3-PS1DTM-3C	3.6E+09	-43	mixed	-0.118	3.9E+09	-47	mixed	0.066	4.3E+09	-41	mixed	-0.169	1.9E+09	-33	mixed	0.023	3.2E+09	-36	mixed	-0.05	1.9E+09	-30	mixed	0.105	2.25 years	
3-E1-PS1	2.5E+10	-54	mixed	-0.02	1.5E+10	-55	mixed	0.128	1.1E+10	-48	mixed	0.083	7.0E+09	-39	mixed	-0.034	7.4E+09	-40	mixed	-0.014	5.8E+09	-35	mixed	-0.065	2.25 years	
4-PS1DTM - 3C	8.0E+09	-54	mixed	0.15	8.1E+09	-52	mixed	0.115	6.7E+09	-46	mixed	0.007	8.0E+09	-49	mixed	0.136	6.7E+09	-46	mixed	0.036	N/A	N/A	N/A	N/A	1.5 years	
4-Z1-PS1	6.5E+09	-57	mixed	-0.443	4.4E+09	-48	mixed	-0.323	6.4E+07	-6	resist	-0.655	1.1E+07	-5	resist	-0.611	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	1 year	
4-Z1-E1-PS1	7.1E+09	-52	mixed	-0.132	3.4E+09	-37	mixed	-0.474	1.8E+09	-26	resist	-0.575	2.3E+09	-29	resist	-0.471	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	1 year	
4-E1-PS1	1.4E+10	-55	mixed	0.019	5.7E+09	-46	mixed	-0.042	4.2E+09	-39	mixed	-0.069	5.7E+09	-46	mixed	-0.039	4.3E+09	-40	mixed	-0.027	N/A	N/A	N/A	N/A	1.5 years	
5-PS1DTM-2C	1.6E+10	-64	cap	1.035	1.2E+10	-65	cap	0.164	1.0E+10	-61	cap	-0.076	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	7 months	Blisters formed at 6 months
5-PS1DTM-3C	2.1E+10	-53	mixed	0.495	8.0E+09	-50	mixed	0.016	1.4E+10	-56	mixed	-0.004	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	7 months	Blisters formed at 6 months
5-Z1-PS1	2.2E+10	-62	cap	-0.269	1.0E+10	-55	mixed	-0.276	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	1 month	Blisters formed at 1 months
5-Z2-PS1	1.8E+10	-65	cap	0.03	1.0E+10	-58	mixed	-0.254	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	1 month	Blisters formed at 1 months
5-I1-PS1	1.6E+10	-71	cap	-0.118	1.1E+10	-65	cap	0.35	1.2E+10	-65	cap	0.122	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	4 months	Blisters formed at 4 months
5-E1-PS1	2.4E+10	-73	cap	0.516	1.3E+10	-65	cap	0.082	7.1E+09	-40	mixed	0.054	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	7 months	
5-PS1DTM-3C	2.1E+10	-53	mixed	0.495	8.0E+09	-50	mixed	0.016	1.4E+10	-56	mixed	-0.004	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	7 months	Blisters formed at 6 months
5-Z1-PS1	2.2E+10	-62	cap	-0.269	1.0E+10	-55	mixed	-0.276	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	1 month	Blisters formed at 1 months
5-Z2-PS1	1.8E+10	-65	cap	0.03	1.0E+10	-58	mixed	-0.254	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	1 month	Blisters formed at 1 months
5-I1-PS1	1.6E+10	-71	cap	-0.118	1.1E+10	-65	cap	0.35	1.2E+10	-65	cap	0.122	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	4 months	Blisters formed at 4 months
5-E1-PS1	2.4E+10	-73	cap	0.516	1.3E+10	-65	cap	0.082	7.1E+09	-40	mixed	0.054	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	7 months	

Appendix C – Summary of Test Results Coating Performance Evaluation

Summary of test results for all tested systems. Data for slurry erosion and Tabor abrasion was collected only on one coating system for coating systems with the same polysiloxane topcoat.

Excellent
Good
Fair
Poor

	Cathodic Disbondment	Slurry Erosion	Tabor Abrasion	Impact	Pull-off Adhesion	Pull-off Adhesion (Wet)	Knife Adhesion (wet)	HAR Immersion	DI Immersion	PRO	BOR	QUV	EIS
Report Code	Radius (inches)	Stabilized weight loss rate (g/hr)	Total wight loss (mg)	Threshold with no cracking or holidays (inch-lbs)	Stress (psi)	Stress (psi)	ASTM Rating	Undercutting (inches)					
Vinyl System 4	3.125	0.039 +/- 0.003	164	20	1412 +/- 125 g/coh	769 +/- 56 g	4	0.11	0.11	0.43	0.35	0	Excellent
Vinyl System 5-E-Z	1	0.041 +/- 0.004	164	100	1012 +/- 226 g or g/coh	No data	4	0	0	0.09	0.15	0	Excellent
Mil 24441	0.325	0.071+/- .008	177	10	1181 +/- 195 coh	1144+/-241	10	0	0	0.32	0.31		Good
1-E1-PS1	0.375	0.033 +/- 0.004	125	80	1299 +/- 123 50/50 coh/adh	No data	8	0.03	0.11	0.9	0.69	0	Excellent
1-E2-PS1	0.25	0.073 +/- 0.007	108	20	970 +/- 120 coh in int	371+/-15	4	0	0	0.73	0.75	Fine microcracking at 4000 hrs	Excellent
1-E3-PS1	0	0.061 +/- 0.014	125	52	1867 +/- 168 coh	1871 +/- 8 Coh	10	0	0	0.36	0.35	0	Excellent
1-I1-PS1	0.25	N/A	N/A	10	1466 +/- 108 coh primer	1087+/-168	10	0	0	0.52	0.46	0	Excellent

	Cathodic Disbondment	Slurry Erosion	Tabor Abrasion	Impact	Pull-off Adhesion	Pull-off Adhesion (Wet)	Knife Adhesion (wet)	HAR Immersion	DI Immersion	PRO	BOR	QUV	EIS
Report Code	Radius (inches)	Stabilized weight loss rate (g/hr)	Total wight loss (mg)	Threshold with no cracking or holidays (inch-lbs)	Stress (psi)	Stress (psi)	ASTM Rating	Undercutting (inches)					
1-Z1-PS1	0.25	N/A	N/A	220	1014 +/- 140 glue/intercoat int/top	551+/-131	0	0	0	0.2	0.16	0	Excellent
1-PS1DTM-3C	0	N/A	125	30	1965 +/- 344 g/coh	1479 +/- 143 coh/ adh	10	0	0	0.46	0.29	0	Excellent
1-E3-PS2	0	N/A	N/A	40	1681 +/- 420 coh	1207 +/- 233 50/50 coh	10	0	0	0.23	0.29	0	Excellent
1-PS2DTM-3C	No Data	0.125 +/- 0.008	182	10	No data	No data	No data	Blisters	Blisters	Blisters/ cracks to substrate	Blisters/ cracks to substrate	Blisters/ cracks to substrate	Poor
2-E1-PS1	0	N/A	N/A	50	1250 +/- 164 coh	No Data	6	0	0.09	0.39	0.48	0	Good
2-E2-PS1	0.25	N/A	N/A	36	1285 +/-120 coh	1028 +/- 251 Coh	10	0	0	0.38	0.46	0	Good
2-Z1-E3-PS1	1.5	N/A	N/A	30	752 +/- 103	371 +/- 150	4	0	0	0.16	0.17	0	Good
2-PS1DTM-3C	0	0.066 +/- 0.013	108	35	2325 +/- 461 coh	1243 +/- 79 Coh/ Adh	10	0	0	0.5	0.45	0	Good
2-E2-PS2	0	N/A	N/A	52	1314 +/-116 coh	1275 +/-168 Coh	10	0	0.02	0.42	0.37	0	Excellent
2-PS2DTM	0	0.072 +/- 0.006	122	82	1059 +/-256 coh	1149 +/- 176 coh/ adh	10	0	0	0.41	0.32	0	Excellent
2-I1-PS2	0.125	N/A	N/A	70	943 +/- 90 coh in primer	1024+/-82	10	0	0	0.22	0.25	0	Excellent
2-Z1-PS2	0.3	N/A	N/A	80	742 +/- 141	700 +/- 122 adh	10	0	0	0.13	0.14	0	Excellent

	Cathodic Disbondment	Slurry Erosion	Tabor Abrasion	Impact	Pull-off Adhesion	Pull-off Adhesion (Wet)	Knife Adhesion (wet)	HAR Immersion	DI Immersion	PRO	BOR	QUV	EIS
Report Code	**Radius (inches)**	**Stabilized weight loss rate (g/hr)**	**Total wight loss (mg)**	**Threshold with no cracking or holidays (inch-lbs)**	**Stress (psi)**	**Stress (psi)**	**ASTM Rating**	**Undercutting (inches)**					
3-PS1DTM-3C	0	0.081 +/- 0.003	175	120	1024 +/- 191 coh	762+/-90	8	0.05	0.09	0.27	0.44	0	Excellent
3-E1-PS1	0	N/A	N/A	40	1401 +/- 120 coh	1403+/-186	8	0	0.09	0.52	0.7	0	Excellent
4-PS1DTM-3C	cracks at splash zone	.091 +/- 0.003	160	70	1543 +/- 150 coh	1277 +/- 272	10	0	0	0.51	0.41	0	Excellent
4-Z1-PS1	.625 & blisters	N/A	N/A	10	1028 +/-109 coh primer	No data	No data	Blistered	Blistered	0.11	0.12	0	Poor
4-Z1-E1-PS1	Blisters	N/A	N/A	10	1036 +/- 163 coh primer	No data	No data	Blistered	Blistered	0.21	0.14	0	Excellent
4-E1-PS1	Blister between topcoat and intermediate	N/A	N/A	24	1324 +/- 269 coh int	1362 +/- 236	10	0	0	0.46	0.3	0	Excellent
5-PS1DTM-2C	Blistered all over pipe	N/A	N/A	70	1237 +/-204	1459 +/- 89 adh, coh, g	No data	Blistered at 6 months	Blistered at 6 months	0.58	0.55	0	Excellent
5-PS1DTM-3C	0.187	0.075 +/- 0.01	90	28	1205 +/- 143	1413 +/- 283 ahd, coh, g	No data	Blistered at 6 months	Blistered at 6 months	0.72	0.45	0	Excellent
5-Z1-PS1	No Data	N/A	N/A	18	1463 +/- 175	No data	No data	Blistered at 1 month	Blistered at 1 month	0.3	0.23	0	Excellent
5-Z2-PS1	No Data	N/A	N/A	14	1099 +/-154	No data	No data	Blistered at 1 month	Blistered at 1 month	0.32	0.16	0	Excellent
5-I1-PS1	0.25	N/A	N/A	32	452 +/- 520	1215 +/- 228 adh primer to intermediate	No data	Blistered at 3 months	Blistered at 3 months	0.31	0.22	0	Excellent
5-E1-PS1	0.25	N/A	N/A	14	1498 +/- 465	2060 +/- 120 coh, g	10	0	0	0.43	0.33	0	Excellent

Appendix D – EIS Summary for Reproducibility Study

EIS results for reproducibility study. Data points include initial value, 1 month, 7 months, 1 year, 1.5 years, and most recent data point. Both vinyl systems and Mil Spec 24441 epoxy are listed as references.

	Initial @ 0.01Hz				1 month @ 0.01Hz				7 months @ 0.01Hz				1 year @ 0.01Hz				1.5 years @ 0.01Hz				X years @ 0.01Hz				X=	Notes
Report Code	|Z|	Phase	Coating Behavior	OCP	|Z|	Phase	Coating Behavior	OCP	|Z|	Phase	Coating Behavior	OCP	|Z|	Phase	Coating Behavior	OCP	|Z|	Phase	Coating Behavior	OCP	|Z|	Phase	Coating Behavior	OCP		
Vinyl System 4	2.7E+09	-76	cap	0.45	4.9E+09	-72	cap	-0.11	5.1E+09	-76	cap	0.042	1.9E+09	-42	mixed	-0.179	8.9E+09	-82	cap	0.426	1.4E+09	-41	mixed	-0.125	6 years	Blisters formed, noticed at 6 years
Vinyl System 5EZ	6.5E+09	-79	cap	0.305	5.1E+09	-81	cap	0.231	3.2E+09	-79	cap	-0.306	1.7E+09	-58	cap	0.111	2.5E+07	-3	resist	-0.774	1.2E+06	-1	resist	-0.35	6 years	Surface looks textured
Mil 24441	2.5E+09	-29	resist	0.136	4.2E+08	-11	resist	0.058	8.5E+08	-19	resist	-0.123	N/A	N/A	N/A	N/A	9.5E+08	-16	resist	-0.168	1.3E+08	-8	resist/ ind	-0.147	2 years	
1-Z1-PS1-2C	9.8E+09	-47	mixed	0.027	1.3E+10	-58	mixed	0.34	1.8E+10	-71	cap	0.167	1.6E+10	-66	cap	-0.09	1.7E+10	-68	cap	0.047	1.4E+10	-61	cap	0.124	2.25 years	
1-PS1DTM-3C	3.8E+10	-83	cap	-0.868	3.1E+10	-84	cap	-0.462	2.4E+10	-51	mixed	0.042	3.0E+10	-81	cap	0.68	3.0E+10	-80	cap	-0.276	1.2E+10	-32	mixed	-0.342	3 years	
1-Z1-PS1-1C	9.2E+09	-78	cap	-0.157	9.1E+09	-80	cap	0.341	8.0E+09	-73	cap	0.295	N/A	N/A	N/A	N/A	7.5E+09	-71	cap	-0.078	N/A	N/A	N/A	N/A	1.5 years	
1-Z1-PS1-2C-R	1.6E+10	-77	cap	0.136	1.5E+10	-77	cap	-0.373	1.4E+10	-73	cap	-0.091	N/A	N/A	N/A	N/A	1.3E+10	-71	cap	-0.022	N/A	N/A	N/A	N/A	1.5 years	
1-Z1-PS1-3C	2.2E+10	-74	cap	0.2	1.8E+10	-71	cap	0.355	1.8E+10	-73	cap	0.239	N/A	N/A	N/A	N/A	1.8E+10	-72	cap	0.193	N/A	N/A	N/A	N/A	1.5 years	
1-PS1DTM-2C	1.1E+10	-63	cap	0.08	1.6E+10	-80	cap	-5.38	1.4E+10	-75	cap	0.95	N/A	N/A	N/A	N/A	1.3E+10	-72	cap	0.154	N/A	N/A	N/A	N/A	1.5 years	
1-PS1DTM-3C-R	1.0E+10	-56	mixed	0.011	1.7E+10	-74	cap	0.397	1.5E+10	-72	cap	0.282	N/A	N/A	N/A	N/A	1.5E+10	-71	cap	0.046	N/A	N/A	N/A	N/A	1.5 years	
2-Z1-PS2- 1C	9.8E+09	-70	cap	0.207	8.3E+09	-71	cap	0.169	8.8E+09	-73	cap	0.045	N/A	N/A	N/A	N/A	8.1E+09	-72	cap	-0.011	N/A	N/A	N/A	N/A	1.5 years	
2-Z1-PS2- 2C	1.3E+10	-68	cap	0.624	1.1E+10	-69	cap	-0.064	1.2E+10	-72	cap	0.01	N/A	N/A	N/A	N/A	1.1E+10	-71	cap	0.168	N/A	N/A	N/A	N/A	1.5 years	
2-Z1-PS2- 3C	1.3E+10	-67	cap	-0.31	1.0E+10	-67	cap	-0.132	1.1E+10	-71	cap	0.433	N/A	N/A	N/A	N/A	1.2E+10	-69	cap	-0.008	N/A	N/A	N/A	N/A	1.5 years	
2-Z1-PS3- 1C	6.3E+09	-64	cap	-0.622	5.7E+09	-68	cap	0.035	5.4E+09	-66	cap	0.002	N/A	N/A	N/A	N/A	5.6E+09	-62	cap	-0.184	N/A	N/A	N/A	N/A	1.5 years	
2-Z1-PS3- 2C	7.8E+09	-64	cap	0.18	7.4E+09	-67	cap	-0.266	5.3E+09	-63	cap	-0.172	N/A	N/A	N/A	N/A	6.6E+09	-62	cap	-0.254	N/A	N/A	N/A	N/A	1.5 years	
2-Z1-PS3- 3C	1.9E+10	-82	cap	-0.893	8.9E+09	-62	cap	-0.032	9.9E+09	-64	cap	-0.018	N/A	N/A	N/A	N/A	9.4E+09	-60	cap	-0.223	N/A	N/A	N/A	N/A	1.5 years	

Appendix E – Summary of Test Results for Reproducibility Study

Summary of results for reproducibility study and multiple polysiloxane coats. Both vinyl systems and Mil Spec 24441 epoxy are listed as references from the prior study and were not included in side-by-side testing with the polysiloxane systems shown.

Excellent
Good
Fair
Poor

	Cathodic Disbondment	Impact	Pull-off Adhesion	Pull-off Adhesion (Wet)	Knife Adhesion (wet)	HAR Immersion	DI Immersion	PRO	BOR	QUV	EIS	Notes
Report Code	Radius (inches)	Threshold with no cracking or holidays (inch-lbs)	Stress (psi)	Stress (psi)	ASTM Rating (0-10)	undercutting (in)						
Vinyl System 4	3.125	20	1412 +/- 125 g/coh	769 +/- 56 g	4	0.11	0.11	0.43	0.35	0	Excellent	
Vinyl System 5-E-Z	1	100	1012 +/- 226 g or g/coh	No data	4	0	0	0.09	0.15	0	Excellent	
Mil 24441	0.325	10	1181 +/- 195 coh	1144+/- 241	10	0	0	0.32	0.31		Good	
1-Z1-PS1-1C-R	Blistered randomly	160	2793 Coh	1530 +/- 438	10	0	0	0.3	0.33	0	Excellent	Cathodic Disbondment blistered randomly
1-Z1-PS1-2C-R	Blistered	140	1483 intercoat	1736 +/- 356	10	0	0	0.29	0.26	0	Excellent	Cathodic Disbondment blistered randomly
1-Z1-PS1-3C-R	0.25	120	1369 intercoat	2149 +/- 638	10	0	0	0.36	0.27	0	Excellent	

	Cathodic Disbondment	Impact	Pull-off Adhesion	Pull-off Adhesion (Wet)	Knife Adhesion (wet)	HAR Immersion	DI Immersion	PRO	BOR	QUV	EIS	Notes
Report Code	Radius (inches)	Threshold with no cracking or holidays (inch-lbs)	Stress (psi)	Stress (psi)	ASTM Rating (0-10)	undercutting (in)						
1-PS1DTM-2C-R	0	140	2157 coh	2273 +/- 394	10	0	0	0.5	0.41	0	Excellent	
1-PS1DTM-3C-R	0	45	2375 coh	2039 +/- 710	10	0	0	0.56	0.4	0	Excellent	
2-Z1-PS2-1C-R	No Data	80	793 +/- 91 coh	718 +/- 71 adh	10	0	0	0.11	0.14	0	Excellent	
2-Z1-PS2-2C-R	0.3	80	742 +/- 141 Coh	700 +/- 122 adh	10	0	0	0.13	0.14	0	Excellent	
2-Z1-PS2-3C-R	0.65	30	700 coh	712 +/- 71 adh	10	0	0	0.14	0.17	0	Excellent	
2-Z1-PS3-1C-R	No Data	50	747 +/- 119 Coh	670 +/- 59	10	0	0	0.06	0.12	0	Excellent	
2-Z1-PS3-2C-R	0.4	55	844 +/- 78 Coh	710 +/- 119 adh	10	0	0	0.09	0.12	0	Excellent	
2-Z1-PS3-3C-R	0.4	25	729 +/- 152 coh	766 +/- 16 adh	10	0	0	0.11	0.14	0	Excellent	

Appendix F – EIS Summary for Cure Study

EIS results for cure study at 0.01 Hz.

Days in Immersion	DTH			72 Hrs			1 Week			2 Weeks			8 Weeks		
	Avg. Z	Avg Theta	Coating Behavior	Avg. Z	Avg Theta	Coating Behavior	Avg. Z	Avg Theta	Coating Behavior	Avg. Z	Avg Theta	Coating Behavior	Avg. Z	Avg Theta	Coating Behavior
Day 1	4.39E+08	-4	Resistive	1.17E+09	-8	Resistive	2.29E+09	-14	Resistive	3.77E+09	-23	Resistive	2.1E+10	-77	Capacitive
Day 3	3.4E+08	-12	Resistive	4.81E+08	-11	Resistive	8.84E+08	-21	Resistive	1.35E+09	-28	Resistive	8.93E+09	-46	Mixed
Week 1	1.05E+09	-41	Mixed	1.2E+09	-34	Mixed	1.15E+09	-31	Mixed	2.87E+09	-39	Mixed	6.37E+09	-45	Mixed
Day12	2.03E+09	-43	Mixed	1.51E+09	-37	Mixed	1.86E+09	-39	Mixed	3.52E+09	-41	Mixed	7.37E+09	-47	Mixed
Week 2	3.16E+09	-44	Mixed	2.38E+09	-42	Mixed	2.28E+09	-41	Mixed	3.61E+09	-41	Mixed	7.65E+09	-48	Mixed
Week 3	3.94E+09	-46	Mixed	4.15E+09	-45	Mixed	4.08E+09	-45	Mixed	4.97E+09	-45	Mixed	8.65E+09	-52	Mixed
Month 2	6.96E+09	-50	Mixed	5.34E+09	-41	Mixed	7.64E+09	-51	Mixed	8.55E+09	-54	Mixed	9.50E+09	-57	Mixed
Month 9	1.19E+10	-60	Capacitive	1.17E+10	-61	Capacitive	1.17E+10	-59	Mixed	1.22E+10	-61	Capacitive	1.17E+10	-61	Capacitive

Appendix G – Adhesion Results for Cure Study

Panel	**PSI** (lbs-in)						**PSI Avg.** (in/lbs)	**Std. Dev.**	**Failure Mode**		
	1	**2**	**3**	**4**	**5**	**6**			**Adhesion**	**Cohesion**	**Glue**
DTH 1 (D)	1194	1449	960	1509	975	1476	1261	253	55	31	14
DTH 1 (W)	1187	1307	1275	1376	989	915	1175	184	63	28	9
72 hrs (D)	743	1001	819	832	1002	841	873	105	70	22	8
72 hrs (W)	1436	1469	1501	1506	1261	1516	1448	96	46	43	11
1 Week (D)	1555	1118	1392	1306	1107	1210	1281	173	47	40	14
1 Week (W)	1407	865	820	913	1061	1227	1049	230	64	28	8
2 Week (D)	1494	1803	1072	1618	1351	1407	1458	249	48	46	7
2 Week (W)	1474	1376	1113	1502	1042	1391	1316	192	57	27	17
8 Week (D)	1527	883	946	975	1591	1095	1170	310	52	36	11
8 Week (W)	1331	1196	1121	1621	1433	1231	1322	182	53	34	13