

# RECLAMATION

*Managing Water in the West*

## Evaluating Corrosion Protection Methods for Riveted and Bolted Connections

Research and Development Office  
Science and Technology Program  
Final Report ST-2019-1717-01  
Technical Memorandum No. 8540-2019-34





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**Evaluating Corrosion Protection Methods for  
Riveted and Bolted Connections**

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# Acronyms and Abbreviations

5-E-Z	U.S. Army Corps of Engineers vinyl system 5-E-Z
adh.	adhesion
BOR	Cyclic immersion – atmospheric exposure
CERL	Construction Engineering Research Laboratory
cm <sup>2</sup>	centimeter squared
coh.	cohesion
CP	cathodic protection
DI	deionized water immersion
DTM	direct-to-metal
EIS	Electrochemical impedance spectroscopy
EPA	Environmental Protection Agency
ERDC	Engineer Research and Development Center
F	Fahrenheit
FED STD	Federal standard
FOG	salt fog
g	glue
g/coh	glue and cohesive
g/hr	grams per hour
g/l	grams per liter
HAR	dilute Harrison solution water immersion
hrs	hours
Hz	hertz
inch-lbs	inch-pounds
int.	intermediate coat
mg	milligram
mV	millivolt
N/A	not applicable
NACE	National Association of Corrosion Engineers
PA	paint application
PRO	Prohesion
psi	pounds per square inch

QUV	Q-Panel UV exposure
Reclamation	Bureau of Reclamation
SCE	saturated calomel electrode
SHDE	superhydrophobic diatomaceous earth
SSPC	Society for Protective Coatings
U.S.	United States
USACE	U.S. Army Corps of Engineers
USBR	Bureau of Reclamation
UV	ultraviolet light
VOC	volatile organic compounds
Z	impedance magnitude

# Executive Summary

The majority of the Bureau of Reclamation's (Reclamation) infrastructure built prior to 1950 has riveted construction. Riveted construction is one of the most challenging design features to protect because it is a non-continuous surface and is susceptible to crevice corrosion. Loads are typically transmitted through riveted or bolted connections, and any resulting flexing or vibration cause the newer generation coatings to crack, including zinc rich primers, epoxies, and polyurethanes. This is a new problem that was not observed with the original vinyl, lead-based paint, or coal tar enamel. SSPC-PA Guide 11 provides guidance on proper coating application techniques for these challenging areas [1]. More flexible materials are required to protect these connections.

Presently, the exterior penstocks at Flatiron Powerplant near Loveland, Colorado are coated with a zinc rich primer, epoxy intermediate coat, and polyurethane topcoat (a 3-coat system). The research was intended to identify flexible coatings or materials to be used on spot repairs in field testing on the exterior of the penstocks. The flexible materials were a calcium sulfide alkyd, an anodic alkyd, and a polyurethane caulk and were intended for atmospheric service. The flexible materials were applied to bare metal, but also overlapped onto the 3-coat system.

Within 6 months of exposure the 3-coat system continued to crack, lifting the flexible coating off the surface on overlapped areas. The riveted collars probably require to be fully recoated or at least scribing the 3-coat system around the riveted collar to mitigate the cracking of the 3-coat system.

For infrastructure subject to immersion service, finding flexible coatings that do not crack around rivets is significantly more challenging. Three different sets of eight coating systems for a total of twenty-four systems were investigated in the laboratory: flexible coatings, superhydrophobic coatings, and polysiloxanes.

For the laboratory portion of the study, flexible coatings were investigated first. This set had three of the eight coatings fail by blistering or wrinkling during testing. The barrier properties were evaluated periodically using electrochemical impedance spectroscopy (EIS) and were compared to the U.S. Corps of Engineers (USACE) vinyl systems 4 and 5-E-Z. Four coatings had a combination of resistive and capacitive behavior. Three coatings that failed were completely resistive. One of the eight coatings was completely capacitive, similar to the vinyl systems, but had poor wet knife adhesion and was easily peeled from the substrate.

The second set of coatings investigated included a superhydrophobic additive (superhydrophobic diatomaceous earth, SHDE) to increase the water repellency and barrier properties. Four different systems of coatings were used with and without the SHDE additive:

1. water borne acrylic
2. 3-coat epoxy
3. zinc rich epoxy/ epoxy intermediate/ polyurethane topcoat
4. 2-coats epoxy/ polyurethane topcoat

The superhydrophobic systems incorporated the SHDE into the topcoat. The SHDE improved the undercutting resistance in cyclic weathering, however, there was no change for water immersion exposure. The SHDE also negatively affected gloss, impact resistance, and adhesion.

In addition, this study identified potential issues with using cathodic protection (CP) with the 2-coats epoxy/ polyurethane topcoat system. Large blisters formed during the cathodic disbondment test on surfaces closest to the anodes. The 2-coat epoxy/ polyurethane system was not previously tested by Reclamation in the laboratory, but has been used on Reclamation infrastructure for fluctuating water exposure since the 1990's.

The third set investigated polysiloxane systems both with epoxy primers and direct-to-metal. Two of the tested systems were found to provide excellent barrier protection and were completely capacitive. These polysiloxanes also provide excellent barrier protection even when they were applied to epoxy primers. The corrosion protection was found to be similar to that of the USACE vinyl systems 4 and 5-E-Z. These systems performed worse than vinyls in erosion and impact testing and did not resist undercutting as much as the 5-E-Z.

The next steps are to evaluate the polysiloxanes with zinc rich primers and inhibitive primers. A field trial will need to be conducted to verify laboratory testing. Finally, the data has been shared with the two manufacturers and requests have been made to improve the erosion and impact resistance of the polysiloxane coatings.

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## Introduction

Maintaining aged infrastructure can be challenging, especially when structures were designed with difficult-to-coat features, such as riveted construction, back-to-back plates, or skip welds. According to NACE SP0178 - Design, Fabrication, and Surface Finish Practices for Tanks and Vessels to be Lined for Immersion Service, these construction methods are not recommended for immersion service [2]. However, Reclamation structures do contain these features in immersion service, and the existing structures must be periodically recoated. Modern coating systems are typically highly crosslinked polymers such as epoxies and polyurethanes, which makes them more susceptible to cracking due to higher internal stress. The standard practice detailed in SSPC-PA Guide 11 is to brush apply stripe coats to work the coating into crevices prior to spray application [1]. In the case where the gap is too large for a coating to bridge, 100 percent solids epoxy mastic fillers or caulks are used to fill the void. This procedure was followed at several Reclamation facilities using newer coating systems but cracking still occurred around rivets and back-to-back plates. Potential causes of this damage are stress due to coating shrinkage and joint movement. The legacy coating systems, lead-based paints, solution vinyl resins, and coal tar enamel coating systems, were able to protect crevices for 40-50 years without cracking.

In 2010, the exterior surfaces of the Flatiron Powerplant Penstocks were coated with a zinc rich epoxy/ epoxy intermediate/ polyurethane topcoat 3-coat system. The coating has experienced cracking at the riveted connections and collars. The cracking appears to be occurring due to the vibration of the pipe as water is flowing through the interior. The vibration concentrates at the riveted collars due to the steel thickness in those areas. As a result, the highly crosslinked, brittle coating cracks and chips. Figures 1 and 2 show the condition representative of every riveted connection on these penstocks after 7 years in service.

This appears to be a new problem, unforeseen by coating specialists and requires to be further investigated. The purpose of this study is to test and identify coatings that are resistant to cracking on riveted structures. To this end, the study was bifurcated into two approaches. The first approach was to evaluate flexible coating systems in the field on riveted sections of the penstocks at Flatiron Powerplant.

The second part of this study was to evaluate coating systems with flexible and/or superhydrophobic properties that were designed for water immersion service. It was decided to screen these coatings through laboratory testing, prior to trying a field demonstration on riveted construction.



**Figure 1. Flatiron penstock riveted connection, coating failure at collar due to vibration**



Figure 2. Flatiron penstock riveted connection, closeup of coating failure at collar

## Field Experimental Procedure

In late 2017, test patches consisting of a polyurethane caulk, a calcium silicate alkyd, and an anodic alkyd were applied to the penstocks Flatirons Powerplant Penstocks at the locations with damaged exterior coating. These systems were selected because they are known to be flexible materials. Atmospheric service conditions were selected for field trials first because rural atmospheric exposures are not as harsh as other service conditions. Also, the inspections and monitoring are easier than interior surface.

The surface preparation consisted of hand-tool cleaning to remove all loose material, hand sanding, and solvent cleaning to remove dust and loose corrosion products. Figure 3 shows the extent of surface preparation. Power tool cleaning was not performed due to the potential that sparking could set fire to the dry brush on the hillside. Repairing the full circumference of the joint required extensive surface preparation, so spot repairs were conducted instead.

The polyurethane caulk was applied at the transition between the concrete anchor block and penstock. The anodic alkyd was applied at the first two sets of rivets upstream from block 14. A typical spot repair is shown in Figure 4. The calcium sulfonate alkyd was applied at the fourth set of rivets upstream from block 14.



**Figure 3. Appearance of steel collar/ pipe interface after surface preparation.**



Figure 4. Anodic alkyd spot repairs at the steel collar interface.

## Field Test Results and Discussion

After 6 months of exposure, an inspection of the field trials was conducted. The majority of the spot repairs were found to have failed, and the original coating (a brittle epoxy) under the flexible coating had cracked and flaked as shown in Figures 5, 6, and 7. Areas that were previously undamaged had also cracked and spalled, indicating the vibration problem continued to spread, and the entire joint requires recoating. Spot repairs are not recommended since the damage progression continues around the full circumference. The 3-coat system should have been completely removed or cut through to bare metal for the full circumference of the joint. Figure 8 shows one area that was successful.



Figure 5. Anodic alkyd spot repairs after 6 months exposure, some areas had cracked coating.



**Figure 6. Anodic alkyd spot repair after 6 months the epoxy coating cracked and flaked the spot repair.**



**Figure 7. Adjacent to the anodic alkyd spot repair (lower area) another area has cracked and spalled (upper area).**



**Figure 8. A successful anodic alkyd spot repair after 6 months.**

## Laboratory Testing

Historically, vinyl systems have been the best flexible coating systems for water immersion service. In the 1990's the Environmental Protection Agency (EPA) reduced the allowable limit on volatile organic compounds (VOC's) in coating systems thus significantly limiting the use of the solution vinyl coatings that contain over 700 g/l VOC. As part of the new regulations, the EPA created a coatings category called Impacted Immersion Coatings which allows vinyl to be used under certain circumstances [3]. While vinyl systems are still available, they are limited in applications. In recent years, Reclamation collaborated with the USACE to investigate the underlying reasons for the vinyl systems' long service lives. Another goal is to identify "greener" coatings that could potentially be an alternative to vinyl.

Two primary strategies are envisioned to mitigate corrosion at rivets. The obvious solution is to use something more flexible in order to allow the movements to be accommodated without coating damage. Another approach would be to use a hydrophobic coating to prevent water ingress to the steel substrate in the event that a crack develops in the coating. This mechanism would promote the formation of water droplets instead of alloying the water to wet out the surface and flow into the crack.

The laboratory study investigated three sets of coating systems for water immersion service. The first set of systems were flexible coatings that manufacturers recommended for immersion service. The second set investigated a superhydrophobic additive to determine if the additive could increase corrosion protection. The third set investigated a variety of polysiloxanes with epoxy primers as well as direct-to-metal. Some of the polysiloxane passed a 1/8" mandrel according to manufacturers' product data sheet before cracking, indicating good flexibility. Therefore, to obtain flexibility throughout the coating system, direct-to-metal applications were added to the scope. All three sets were directly compared to USACE vinyl formulations, listed in the Tables.

### Flexible Coatings

The first set of coating systems investigated were designed for immersion service but still had flexibility. These materials use higher molecular weight polymers in order to achieve their flexibility. Table 1 describes the flexible coating systems tested. A wide variety of products were selected for testing.

**Table 1. Flexible Coatings Set Naming Codes and Description of Product**

Designation	Description
USACE System 4	Vinyl formulation V766E, similar to USBR VR3 coating system, 5 coats
USACE System 5-E-Z	Zinc rich vinyl primer with 4 coats V-766E, 5 coats
Flexible coating 1	Polyurea with adhesion promotion additive, single coat
Flexible coating 2	Epoxy siloxane, flexible and abrasion resistant, 2 coats
Flexible coating 3	Epoxy fluoro ceramic, flexible and abrasion resistant, 2 coats
Flexible coating 4	Polysulfide epoxy, flexible and abrasion resistance, 2 coats
Flexible coating 5	Polysulfide epoxy, high elongation, flexible and abrasion resistance, 2 coats
Flexible coating 6	Navy polysulfide epoxy, flexible and abrasion resistance, 2 coats
Flexible coating 7	Zinc rich moisture cured urethane with polyurethane elastomer, 2 coats
Flexible coating 8	Flexible epoxy, designed for riveted construction

## Superhydrophobic Coatings

One way that a surface can be characterized is through its hydrophobicity or hydrophilicity. A surface is considered hydrophilic if the contact angle between a drop of water and the substrate is less than 90 degrees. A surface is hydrophobic if the contact angle is greater than 90 degrees. A superhydrophobic surface has a contact angle of greater than 150 degrees. The majority of superhydrophobic materials rely on micro- and nano-textured surfaces. These surfaces trap air, which prevents the water from wetting out the surfaces, resulting in high contact angles. The theory is that the hydrophobic properties could improve barrier protection, thus increase the coating service lifetime of the coating system. There are many different procedures and methods to create materials that have superhydrophobic properties [5]. A superhydrophobic additive, (superhydrophobic diatomaceous earth, SHDE) was incorporated into coatings in an attempt to increase the water repellency of the system. This study investigated the effect of SHDE in a variety of coating systems listed in Table 2. Superhydrophobic coatings were selected for evaluation to determine whether superhydrophobic properties can effectively prevent water from entering a break in the coating.

One aspect to consider is coating degradation. Coatings weather and degrade within their environment and are damaged by mechanical impact or erosion. The superhydrophobic additive is on the surface interface and it is necessary to determine whether degradation results in a loss of hydrophobic properties as the coating weathers.

## Superhydrophobic Materials and Application

### Superhydrophobic Additive:

The superhydrophobic additive utilized was a chemically modified diatomaceous earth. The diatomaceous earth was pretreated with a hydrophobic silane, which resulted in its surface becoming superhydrophobic [5]. Diatomaceous earth is common and naturally occurring composed of siliceous sedimentary rock. It is highly porous with air voids, while exhibiting micro and nano textured outer surfaces. The hydrophobic silane chemical treatment, along with the micro and nano texture, is what gives it the superhydrophobic properties [5]. The most common use of diatomaceous earth in coatings is as a flattening agent. For the purposes of this paper, the additive is referred to as SHDE.

### Coatings and Incorporating SHDE:

Coating systems were applied in accordance with the coating manufacturers' instructions, except for the topcoats, which incorporated the SHDE. The primers and intermediate coats were commercially available products. Each system had an equivalent control that did not contain SHDE.

The SHDE cannot be added directly to the coating, because the coating would encapsulate the air voids of the diatomaceous earth, impacting the superhydrophobic properties. First, a slurry was made using a fluorinated solvent, FC-40 Fluoroinert, and the SHDE, to completely wet out and fill the air voids of the SHDE [5]. The FC-40 is inert. FC-40 can be thought of as a solvent that protects the SHDE micro and nanopores from being completely engulfed by the paint systems during the application process. The protective solvent (FC-40) evaporates during the paint curing process, and results in a macro-porous paint structure with the macro-pores covered with nanoporous SHDE. A total of 15 percent by weight of SHDE was incorporated into each system's topcoat.

Table 2. Superhydrophobic coating system description

Designation	Description
USACE System 4	Vinyl formulation V766E, similar to USBR VR3 coating system, 5 coats
USACE System 5-E-Z	Zinc rich vinyl primer with 4 coats V-766E, 5 coats
System 1A	Waterborne Acrylic
System 1B	Waterborne Acrylic with SHDE
System 2A	3 coats epoxy
System 2B	3 coats epoxy w/ SHDE
System 3A	Zinc rich epoxy/ epoxy int./ polyurethane topcoat
System 3B	Zinc rich epoxy/ epoxy int./ polyurethane topcoat w/ SHDE
System 4A	Epoxy primer/ epoxy int./ polyurethane topcoat
System 4B	Epoxy primer/ epoxy int./ polyurethane topcoat w/ SHDE

## Polysiloxane Coatings

Polysiloxane coatings are known for their excellent ultraviolet (UV) light stability and color and gloss retention in atmospheric exposure. Coating manufacturers claim a 20-30 year service life for atmospheric exposure with minimal loss of gloss or color change. In 2017, Reclamation evaluated polysiloxane topcoats with abrasion resistant epoxy primers [6]. The results showed increased barrier properties, but the undercutting resistance was not as good as vinyl [7] [8] [9] [10]. For this study, different epoxy primers and polysiloxane direct-to-metal (DTM) were investigated. A few polysiloxanes pass a 1/8-inch mandrel bend test (as reported in the product data sheet) indicating they have good flexibility.

**Table 3. Polysiloxane coating system description**

Designation	Description
USACE System 4	Vinyl formulation V766E, similar to USBR VR3 coating system, 5 coats
USACE System 5-E-Z	Zinc rich vinyl primer with 4 coats V-766E, 5 coats
Polysiloxane 1 DTM	Manufacturer 1 Original Polysiloxane direct-to-metal
Polysiloxane 2 DTM	Manufacturer 2 New Polysiloxane direct-to-metal
Polysiloxane 3 DTM	Manufacturer 2 Original Polysiloxane direct-to-metal
Polysiloxane 4 DTM	Manufacturer 1 New Polysiloxane direct-to-metal
Epoxy A/ Polysiloxane 1	Manufacturer 1 Original Polysiloxane with epoxy primer
Epoxy B/ Polysiloxane 2	Manufacturer 2 New Polysiloxane with epoxy primer
Epoxy B/ Polysiloxane 3	Manufacturer 2 Original Polysiloxane epoxy primer
Epoxy A/ Polysiloxane 4	Manufacturer 1 New Polysiloxane epoxy primer

## Laboratory Experimental Procedures

### Surface Preparation and Coating Application

The surface preparation 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 manufacturer's instructions. The polysiloxane systems required 40 percent relative humidity and were placed in a containment with a humidifier to obtain 60 percent relative humidity during cure inside the spray booth.

## 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-mV 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 centimeters squared ( $\text{cm}^2$ ). No corrections were made to the raw data for surface area. EIS was performed periodically throughout the exposure conditions over a 30-week time frame.

## Coating Performance Evaluation

It is difficult to simulate the dynamic conditions that riveted equipment is subject to in the field and under load. Hence no attempts were made to do so in this study. Instead, Reclamation's standard testing protocol was used as follows:

- HAR - Immersion in a dilute Harrison solution (DHS, ASTM D870 Modified)
- DI - Immersion in a deionized solution (ASTM D870)
- FOG - Prohesion testing in a salt fog test cabinet (ASTM G85 Annex A4)
- QUV - Accelerated weathering 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)

The BOR test is a modified Prohesion test, which is intended to simulate the effects of a fluctuating immersion environment/ splash zone. Panels are rotated every week in the following order: QUV-FOG-HAR-FOG. Some panels were scribed down the center with a Dremel® tool. The scribes, which were approximately 1 millimeter in width and 3 inches in length, exposed the bare substrate.

All tests were exposed for approximately 30 weeks (5040 hours) in accordance with industrial standard practices. Panel evaluation proceeded according to ASTM D1654 rust creep and ASTM D714 degree of blistering.

To test each system's compatibility with CP systems, 3-inch pipes were coated with each system. A cathodic protection system was then simulated in accordance with ASTM G8 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, 1/8-inch thick steel discs were used. The test duration was 96 hours per sample. Duplicate samples were run. The average weight loss was compared between samples and a control.

## Laboratory Results and Discussion

The coating benchmark has excellent barrier properties showing capacitive behavior, i.e. the Bode plot curve should have a continuous 45-degree slope without any horizontal bending. The phase angle should remain near -90 degrees at all frequencies. The USACE system 4 and 5-E-Z both exhibits these properties [7] [8]. Figure 9 shows the Bode plot of USACE system 4 in deionized water after 7 months exposure. Notice there is minimal degradation and the 45-degree line is continuous, indicating the coating is capacitive with no corrosion under the coating.

The USACE system 5-E-Z provides the best corrosion protection and undercutting resistance because the system contains a zinc rich primer. In addition, system 5-E-Z has good impact resistance and slurry erosion resistance [7] [8]. The adhesion failed by glue failure, therefore the pull off adhesion is unknown since glue failures indicate that the coating adhesion is stronger than the glue's tolerance. The coating was easy to peel by knife adhesion.

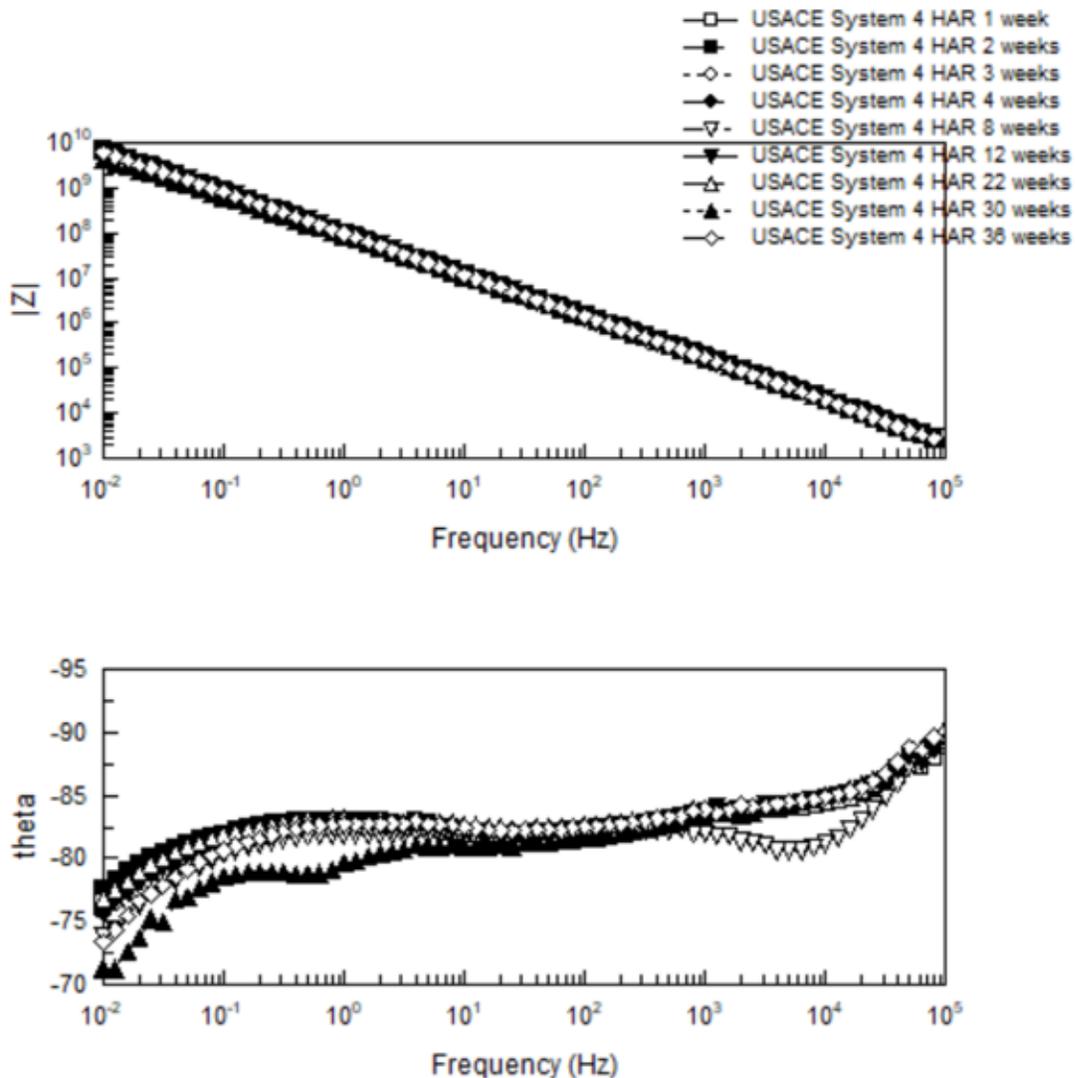


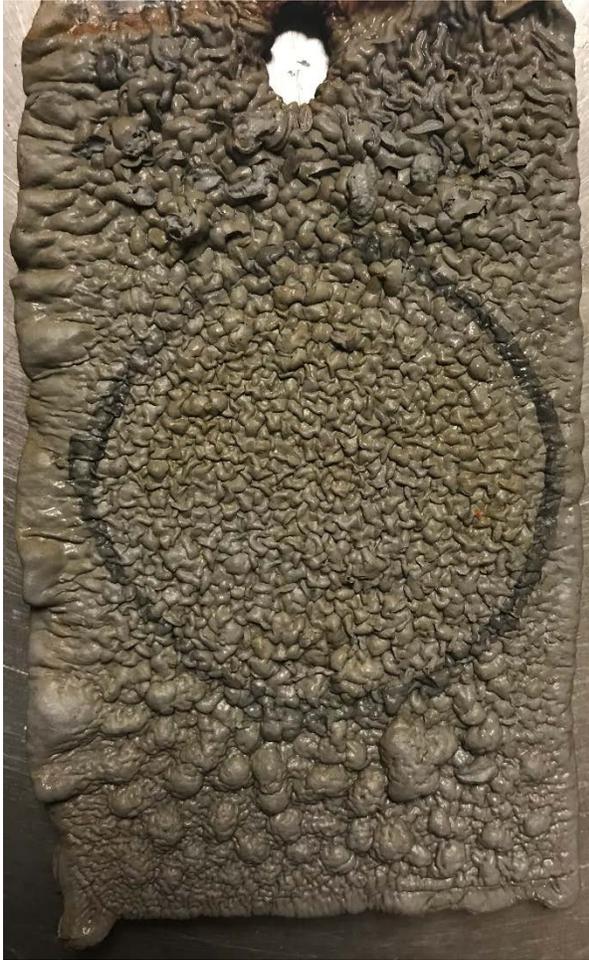
Figure 9. Bode plot of USACE System 4 in HAR water after 7 months. Capacitive behavior, phase angle is above -70 degrees.

## Flexible Coatings

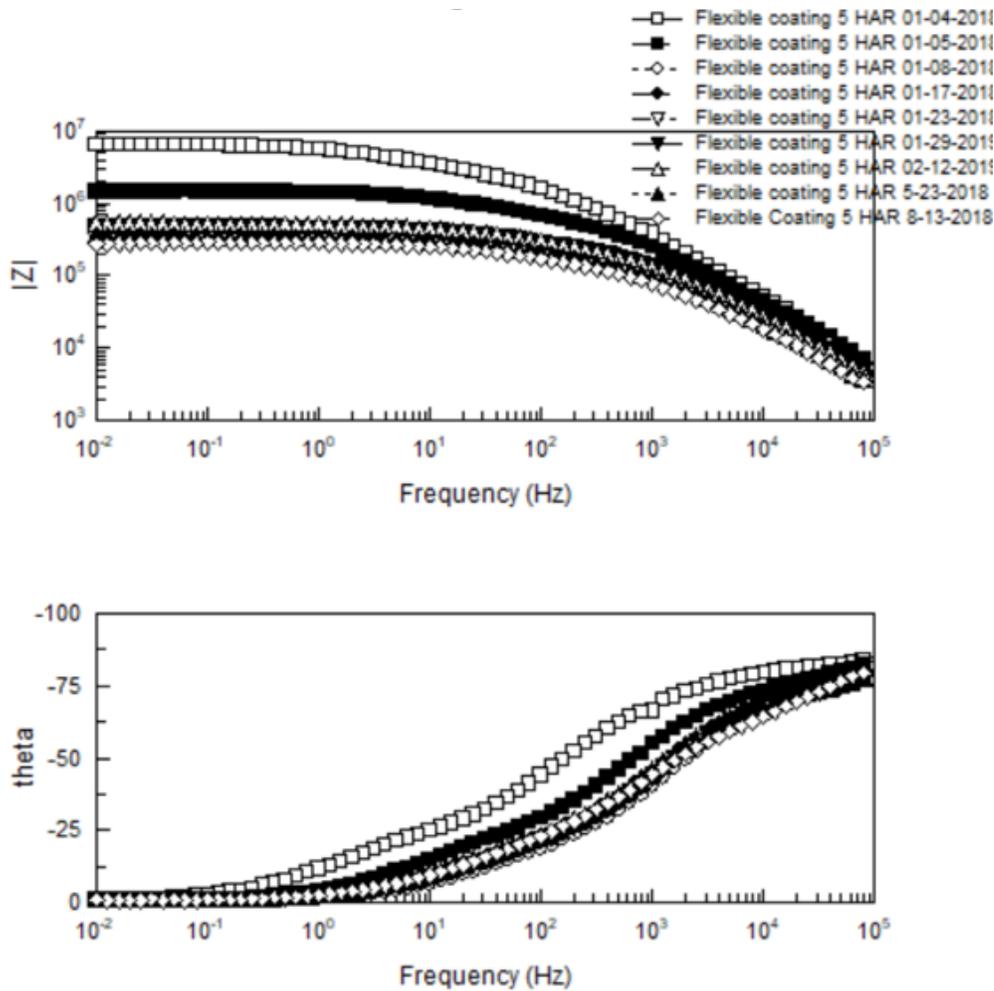
Appendix Table 1 summarizes the results of the flexible systems study [11] [12] [13] [14] [15] [16] [17] [18]. The data collected include the extent of undercutting, barrier properties using EIS, cathodic disbondment, erosion and abrasion resistance, impact, and dry, wet, and knife adhesion. Key findings will be discussed, but due to the volume of data not all of the results will not be discussed; therefore, the tables should be reviewed for comparison to the USACE vinyl systems. None of the flexible coating systems tested were found to be equivalent to vinyl. This does not mean there isn't a flexible coating suitable out there, but it wasn't in the current set of systems.

## Evaluating Corrosion Protection at Riveted and Bolted Connections

Flexible coating systems 2, 3, and 5 completely failed by either blistering or wrinkling as seen in Figure 10 [13] [15] [16]. The EIS data showed high permeability of electrolyte through these coating systems [19] [20] [21]. Figure 11 shows the EIS Bode plots of flexible coating 5, (an example of a coating system that failed) with high permeability because of the low impedance magnitude,  $|Z|$ , value and the phase angle (lower graph) goes to zero degrees [13]. The plots also show resistive behavior with the curve becoming horizontal at high frequencies, indicating the presence of corrosion at the steel-coating interface.



**Figure 10. Failure of coating system 5, severely wrinkled in water immersion service.**



**Figure 11. EIS data of flexible coating 5 in HAR immersion. Bode (top) plot, shows resistive behavior and phase angle (bottom) shows high permeability properties as the phase goes to zero.**

Flexible coating 6 was developed by the Navy to be a rapidly curing product to coat steel pilings during low tide. The polysulfide epoxy has good flexibility. Figure 12 shows the EIS results of the system which was found to have high permeability with a combination of capacitive and resistive behavior indicating there is a small amount of corrosion at the interface [18]. Many coatings have similar EIS plots. Reclamation uses coatings that have the lowest resistive behavior as possible. Flexible coating 6 has higher resistive behavior than desired and should not be used on Reclamation structures for corrosion protection.

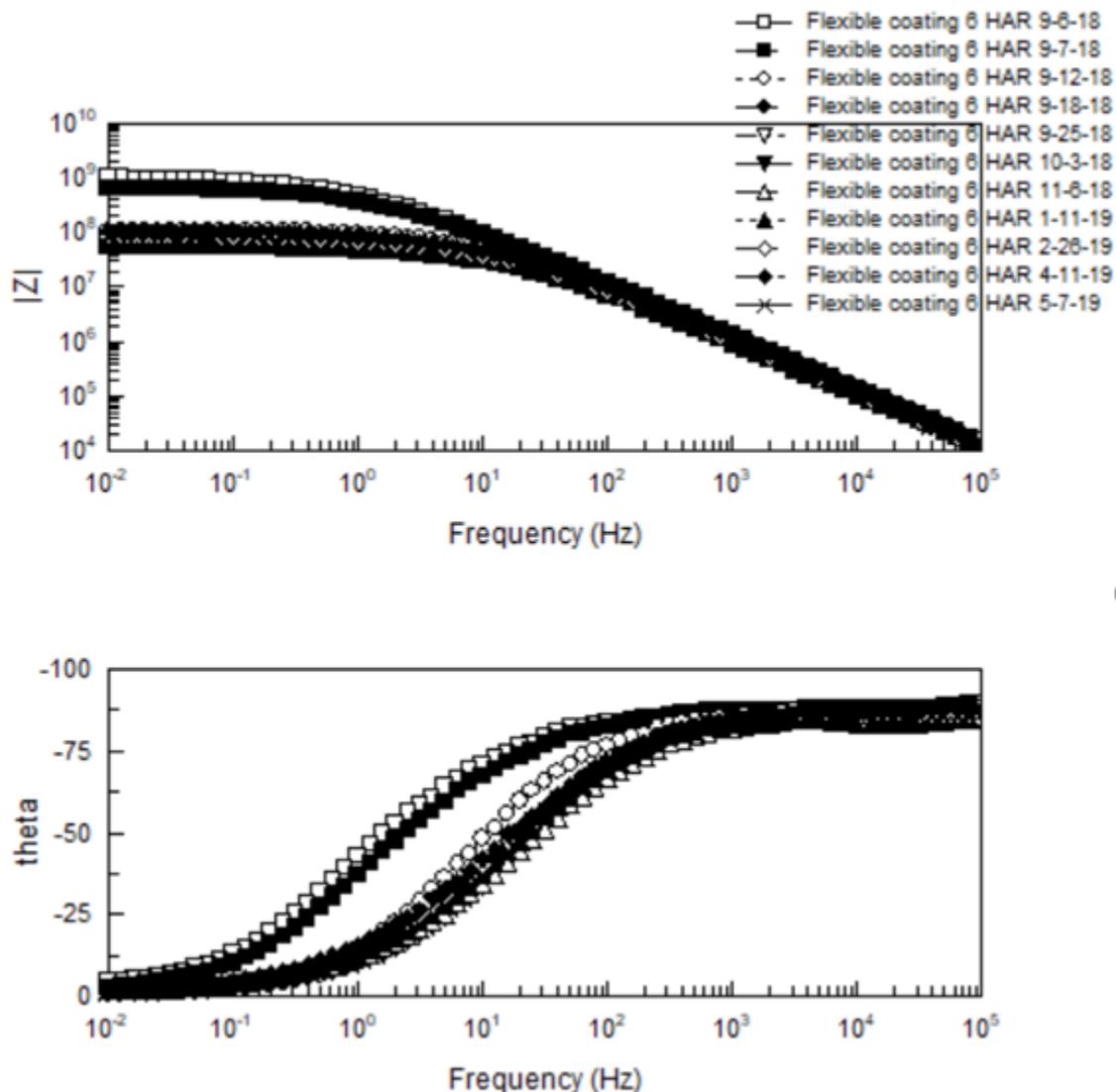


Figure 12. Bode plot of Flexible coating 6 in HAR immersion after 7 months. Capacitive and resistive behavior, but phase angle goes to zero indicating corrosion at interface.

Flexible coating 7 was the only coating in the entire set that provided capacitive properties in EIS [10]. However, the wet adhesion between the topcoat and the zinc rich primer was low and the topcoat could be easily peeled from the primer. This coating system would be highly susceptible to delamination if it were to be used in flowing water applications as seen in Figure 13.



**Figure 13. Flexible coating 7 wet adhesion disbondment between the zinc rich primer and the topcoat.**

## Superhydrophobic Coatings

The superhydrophobic additive provided slight undercutting improvement over the controls when subject to cyclic weathering. The full dataset is found in Appendix Table 2 [22]. EIS for all samples showed a combination of resistive and capacitive behavior. The SDHE additive reduced mechanical and physical properties such as adhesion, impact, and gloss compared to the controls [22]. The SHDE additive had no effect on corrosion undercutting in immersion service but slightly increased the systems' porosity and resistive behavior [22].

The cathodic disbondment testing showed that the only systems to be compatible with cathodic protection were Systems 2A and 2B [22]. This study revealed that large blisters formed on surfaces closest to the anodes, not at the intentional defect, for Systems 4A and 4B shown in Figure 14 [22]. Systems 4A and 4B had the same result with large blisters forming on the side closest to the anode [22]. Reclamation has specified this system, two coats of epoxy with a polyurethane topcoat, for the splash zone of gate structures. The polyurethane was used to provide UV protection. This is concerning because many Reclamation structures coated with this system use CP to enhance corrosion protection on riveted construction. There is potential that the CP system may result in blistering of the coating.



**Figure 14. Cathodic disbondment of two coats of epoxy with polyurethane topcoat with and without SHDE.**

### Polysiloxane Coatings

The polysiloxane set provided the most promising results for corrosion protection during laboratory testing. Some of the polysiloxane systems were found to provide excellent barrier properties like the legacy coatings such as coal tar enamel and vinyl resin. The results are compared to USACE vinyl systems 4 and 5-E-Z in Appendix Table 3 for further review [23] [24] [25] [26] [27] [28] [29]. The EIS results showed capacitive behavior for five systems: polysiloxane 1 DTM, polysiloxane 2 DTM, epoxy A/polysiloxane 1, epoxy A/ polysiloxane 4, and epoxy B/ polysiloxane 2 [23] [24] [26] [27] [29]. Figure 15 and Figure 16 show the EIS Bode plots for polysiloxane 1 and polysiloxane 2 respectively; both have capacitive properties with values above  $10^{10}$  at 0.01 Hz. These systems exhibit EIS performance similar to USACE system 4 shown in Figure 9. Since similar results had not previously been observed for epoxy systems alone, polysiloxanes 1 and 2 provide the capacitive properties for the abrasion resistant epoxy primer/ polysiloxane topcoat systems. Polysiloxane 3 DTM and epoxy B/ polysiloxane 3 had a combination of resistive and capacitive behavior [25] [28]. Figure 17 shows the resistive and capacitive behavior of the polysiloxane 3 DTM in the EIS Bode plot. Polysiloxane 4 DTM blistered and micro-cracked within the first few weeks of testing and was withdrawn from testing.

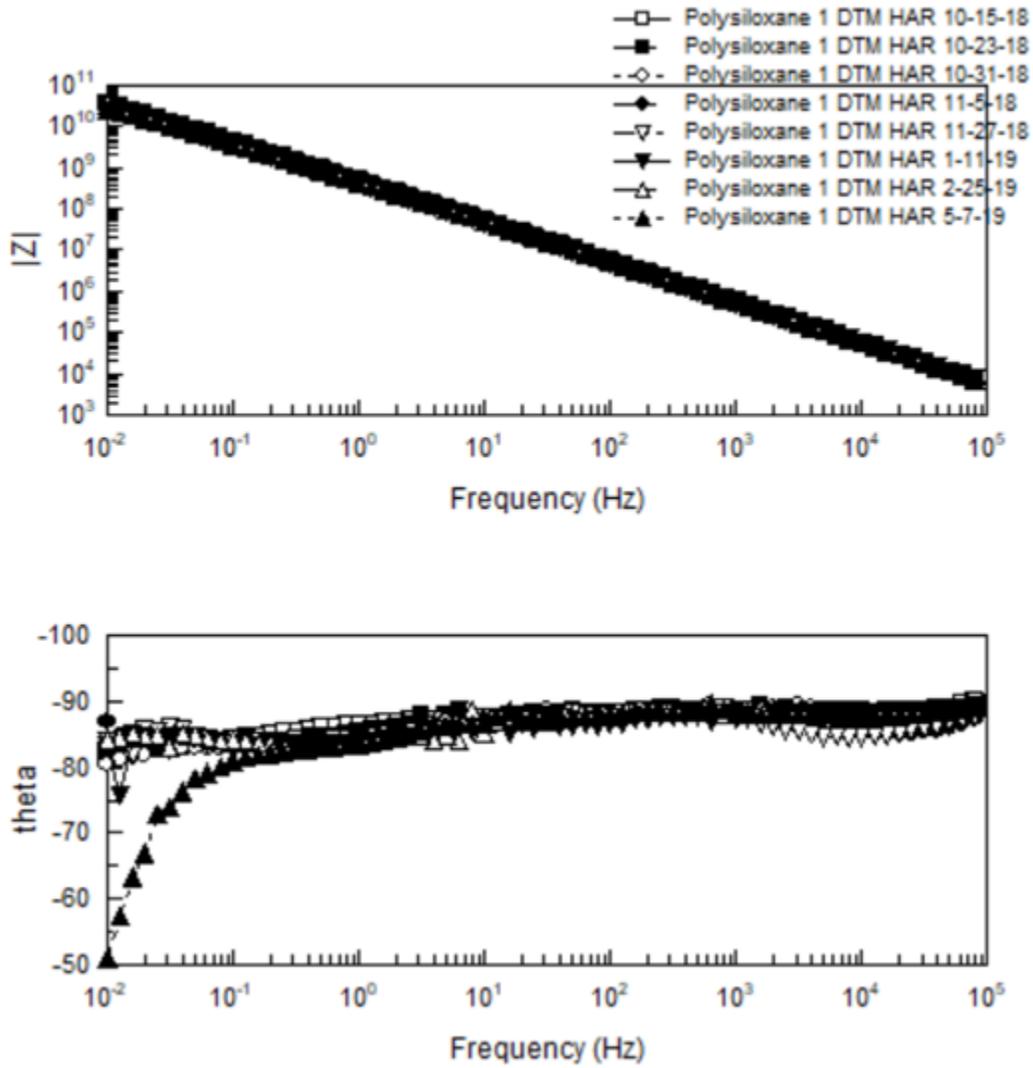


Figure 15. EIS Bode Plot of Polysiloxane 1 DTM after 7 months exposure in HAR immersion

Evaluating Corrosion Protection at Riveted and Bolted Connections

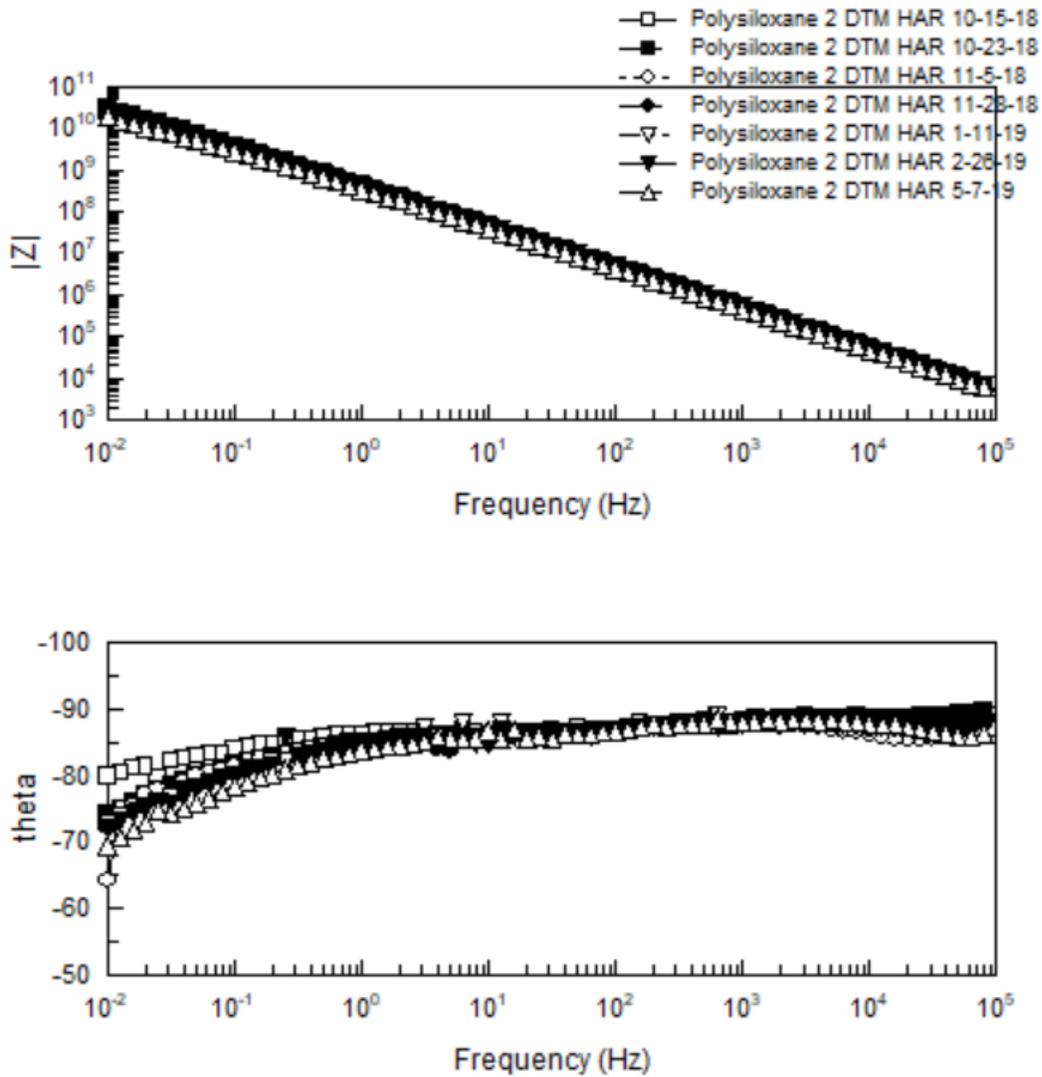


Figure 16. EIS Bode plot of Polysiloxane 2 DTM after 7 months exposure in HAR immersion

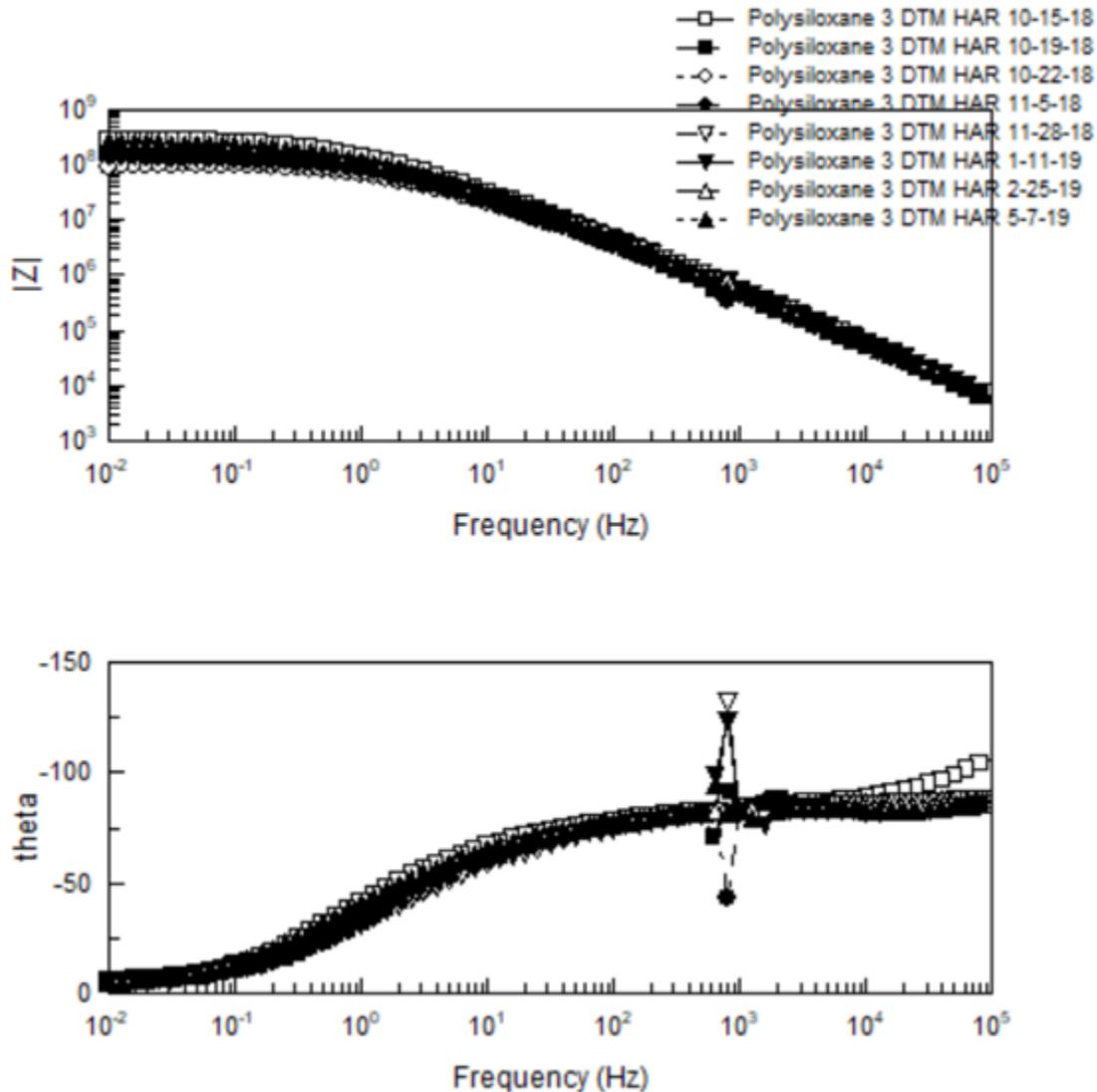


Figure 17. EIS Bode plot of polysiloxane 3 DTM after 7 months exposure in HAR immersion

The systems that closely match the properties to the USACE vinyl systems 4 and 5-E-Z are polysiloxane 1 DTM, polysiloxane 2 DTM, epoxy A/ polysiloxane 1, and epoxy B/ polysiloxane 2. The USACE vinyl system 5-E-Z has better undercutting resistance because it contains a zinc rich primer, whereas none of the other systems contain zinc. The  $|Z|$  for the four polysiloxane systems were slightly higher than the vinyl systems. When subjected to the cathodic disbondment test, the polysiloxane systems prevented disbondment, which was a significant improvement compared to vinyl. Based on the test data, these systems could provide long term corrosion protection with or without CP. One aspect that could reduce the service life is the durability of the coating system. The polysiloxane systems did not have as good of erosion resistance or impact resistance as USACE vinyl system 5-E-Z. Figure 18 shows the erosion pattern of polysiloxane 2 after a 96-hour test the topcoat has eroded through to the intermediate

coat [24]. The longevity of these systems will be highly dependent upon the erosion rate, impact damage from debris, and ability to withstand cracking.



Figure 18. Erosion of Polysiloxane 2 DTM after 96hrs of slurry erosion

## Conclusions

- The field trials for atmospheric exposure showed that the spot repair method was not adequate and full circumference repairs are the only way to repair the riveted collars.
- The laboratory testing showed the majority of the systems were not satisfactory for Reclamation use on riveted construction.
- The superhydrophobic study identified potential problems with incompatibility with CP and the epoxy/ polyurethane topcoat system.
- The polysiloxane study had promising results for a few systems. Some of the polysiloxane systems have barrier properties similar to the USACE vinyl systems 4 and 5-E-Z.

## Next Steps

Some of polysiloxane systems tested could provide long term corrosion protection, based on preliminary findings of this laboratory study. One of the primary motivations of this work was to find a coating system that was durable enough to withstand cracking on the coatings of structures that have non-continuous surfaces. This work presents results on flat panels. The next steps for the polysiloxane coatings should be conducted on complex geometries.

The following recommendations are made:

- Investigate zinc rich epoxy/ two coats polysiloxanes and epoxy primers/ two coats polysiloxanes for improved undercutting resistance.
- Accelerated laboratory testing of polysiloxane on complex geometries.
- Field trials of the promising polysiloxane systems and monitoring of long-term performance using field EIS and visual evaluation methods.
- Field trials on riveted construction to determine if the polysiloxanes can withstand movement and cracking.
- Collaborate with coating manufacturers to improve coating system formulations.

## References

- [1] *SSPC PA Guide 11*, Pittsburgh: SSPC: The Society for Protective Coatings, 2008.
- [2] *NACE SP 0178 Design, Fabrication, and Surface Finish Practices for Tank and Vessels to be Lined for Immersion Service*, Houston: NACE International, 2007.
- [3] Environmental Protection Agency, "40 CFR Part 59 subpart D, Volatile Organic Compounds(VOC), Content Limits for Architectural Coatings," Government Printing Office, Washington D.C., 1999.
- [4] K.-y. Law, "Definitions for Hydrophilicity, Hydrophobicity, and Superhydrophobicity: Getting the Basics Right," *Journal of Physical Chemistry*, vol. 5, no. 4, pp. 686-688, 2014.
- [5] S. H. T. A. J. Simpson, "Superhydrophobic Materials and Coatings: A Review," *Rep Prog Phys*, vol. 78, pp. 1-14, 2015.
- [6] D. Tordonato, "Finding a Green Alternative to Vinyl Resin Coatings - Final Report," Bureau of Reclamation, Denver, 2017.
- [7] Bureau of Reclamation, "Coatings Test Report 8540-2017-031," Materials and Corrosion Laboratory, Denver, CO, 2017.
- [8] Bureau of Reclamation, "Coatings Test Report 8540-2017-032," Materials and Corrosion Laboratory, Denver, CO, 2017.
- [9] Bureau of Reclamation, "Coatings Test Report 8540-2017-033," Materials and Corrosion Laboratory, Denver, CO, 2017.
- [10] Bureau of Reclamation, "Coatings Test Report 8540-2017-034," Materials and Corrosion Laboratory, Denver, CO, 2017.
- [11] C. Gulsvig, "8540-2018-07," Bureau of Reclamation, Denver, 2018.
- [12] C. Gulsvig, "8540-2018-08," Bureau of Reclamation, Denver, 2018.
- [13] C. Gulsvig, "8540-2018-43," Bureau of Reclamation, Denver, 2018.
- [14] C. Gulsvig, "8540-2018-44," Bureau of Reclamation, Denver, 2018.
- [15] C. Gulsvig, "8540-2018-45," Bureau of Reclamation, Denver, 2018.

- [16] C. Gulsvig, "8540-2018-46," Bureau of Reclamation, Denver, 2018.
- [17] C. Gulsvig, "8540-2018-47," Bureau of Reclamation, Denver, 2018.
- [18] A. Skaja, "8540-2019-21," Bureau of Reclamation, Denver, 2019.
- [19] B. Hinderliter, S. C. D. Tallman, Q. Su and G. Bierwagen, "Interpretations of EIS Data from Accelerated Exposure of Coated Metals Based on Modeling of Coating Physical Properties," *Electrochimica Acta*, vol. 51, pp. 4505-4515, 2006.
- [20] D. Loveday, P. Peterson and B. Rodgers, "Evaluation of Organic Coatings with Electrochemical Impedance Spectroscopy. Part 2: Application of EIS to Coatings," *JCT Coatings Tech*, pp. 88-92, 2004.
- [21] F. Mansfeld and C. Tsai, "Determination of Coating Deterioration with EIS. Part I. Basic Relationships," *Corrosion*, vol. 47, pp. 726-737, 1991.
- [22] S. P. C. G. Allen Skaja, "Myth Busters: Superhydrophobic Additives for Coatings," in *Coatings +*, Orlando, 2019.
- [23] A. Skaja, "8540-2019-17," Bureau of Reclamation, Denver, 2019.
- [24] A. Skaja, "8540-2019-18," Bureau of Reclamation, Denver, 2019.
- [25] A. Skaja, "8540-2019-19," Bureau of Reclamation, Denver, 2019.
- [26] A. Skaja, "8540-2019-22," Bureau of Reclamation, Denver, 2019.
- [27] A. Skaja, "8540-2019-23," Bureau of Reclamation, Denver, 2019.
- [28] A. Skaja, "8540-2019-24," Bureau of Reclamation, Denver, 2019.
- [29] A. Skaja, "8540-2019-25," Bureau of Reclamation, Denver, 2019.
- [30] D. Tordonato, A. Skaja and B. Merten, "Past versus present – Comparing laboratory performance of Vinyl Resin Coatings with Modern Epoxy-Polysiloxane Coating Systems," in *SSPC's Greencoat Conference*, Tampa, FL, 2017.
- [31] D. Tordonato, B. Merten, A. Skaja and A. Beitleman, "Secrets to Success - Examining the History and Chemistry behind the Performance of Vinyl Resin Coatings," in *SSPC's Greencoat*, San Antonio, TX, 2016.



## Data Sets that Support the Final Report

- Share Drive folder name and path where data are stored:  
T:\Jobs\DO\\_NonFeature\Science and Technology\2016-PRG-Coating Riveted Construction
- Point of Contact: Allen Skaja, askaja@usbr.gov, 303-445-2396
- Folder includes all data, photographs, reports, and presentations associated with this project.
- Keywords: Flexible Coatings, Corrosion Protection, Caulks, polysiloxanes superhydrophobic, riveted and bolted construction
- Approximate total size of all files: 900 MB

## Appendix A – Summary Tables

Appendix Table 1. Summary results of flexible coatings set after 5000 hours of exposure

	HAR Immersion	DI Immersion	PRO	BOR	QUV	EIS HAR	EIS DI	Cathodic Disbondment	Slurry Erosion	Tabor Abrasion	Impact	Pull-off Adhesion	Pull-off Adhesion (Wet)	Knife Adhesion (Wet)
	undercutting (in) Max	undercutting (in) Max	undercutting (in) Ave	undercutting (in) Ave	undercutting (in) Ave	(High - Low Impedance value at 0.01Hz), (Ohms)	(High - Low Impedance value at 0.01Hz), (Ohms)	Radius (inches)	Stabilized weight loss rate (g/hr)	Total weight loss, mg	No cracking or holidays (inch-lbs)	Stress (psi)	Stress (psi)	ASTM Rating (0-10)
USACE System 4	0.11	0.11	0.43	0.35	0	7.8E9 - 2.7E9 Capacitive	8.5E9 - 4.5E9 Capacitive	3.125	0.039 +/- 0.003	164	20	1412 +/- 125 glue /Coh.	769 +/- 56 glue	4
USACE System 5-E-Z	0	0	0.09	0.15	0	6.5E9 - 4.1E9 Capacitive	7.5E9 - 2.2E9 Capacitive	1	0.041 +/- 0.004	164	100	1012 +/- 226 glue or glue /Coh.	No data	4
Flexible coating 1	0.04	0.14	0.4	0.35	0 Discoloration	1.3E10 - 1.2E9 Resistive	2.5E9 - 1.4E9 Resistive	N/A	N/A	N/A	232	898 +/- 148 glue	1075 +/- 225 glue	0
Flexible coating 2	Blistered	Blistered	0.65	0.53	0 Discoloration	6.0E8 - 1.4E8 Resistive	1.0E9 - 1.9E8 Resistive	N/A	N/A	N/A	38	1322 +/- 446	278 +/- 240	2
Flexible coating 3	Blistered	Blistered	0.56	0.38	0 Discoloration	8.4E8 - 2.0E5 Resistive	1.4E8 - 3.3E7 Resistive	N/A	N/A	N/A	34	2080 +/- 185	583 +/- 221 Adh.	2
Flexible coating 4	0	0	0.55	0.47	0 Discoloration	2.8E9 - 5.1E7 Resistive	3.5E8 - 9.9E7 Resistive	N/A	N/A	N/A	112	2990	1202 +/- 223	0
Flexible coating 5	Wrinkling	Wrinkling	0.21 pinpoint	0.21 pinpoint	0.1 pinpoint	6.5E6 - 2.7E5 Resistive	1.6E6 - 3.4E5 Resistive	N/A	N/A	N/A	25	454	100	0
Flexible coating 6	0.07	0.03	0.47	0.25	0 Discoloration	1.0E9 - 5.8E7 Resistive	9.7E8 - 6.7E7 Resistive	0.625	0.071 +/- 0.011	0.119	88	2409 +/- 120	1341 +/- 214	10
Flexible coating 7	0 peel	0 peel	0.17 peel	0.34 peel	0 Discoloration	8.7E10 - 3.8E10 Capacitive	7.1E10 - 2.1E10 Capacitive	0.5	0.066 +/- 0.017	0.061	80	1614	1065	0
Flexible coating 8	0.16	0.16	0.29	0.33	0 Discoloration	1.7E10 - 3.9E8 Resistive	1.0E9 - 1.6E8 Resistive	0.95	0.045 +/- 0.005	0.095	40	1714	871	10

Appendix Table 2. Summary results of superhydrophobic additive study after 5000 hours of exposure

	HAR Immersion	DI Immersion	PRO	BOR	QUV	EIS HAR	EIS DI	Cathodic Disbondment	Impact	Pull-off Adhesion
	undercutting (in) Max	undercutting (in) Max	undercutting (in) Ave	undercutting (in) Ave	undercutting (in) Ave	(High - Low Impedance value at 0.01Hz), (Ohms)	(High - Low Impedance value at 0.01Hz), (Ohms)	Radius (inches)	No cracking or holidays (inch-lbs)	Stress (psi)
USACE System 4	0.11	0.11	0.43	0.35	0	7.8E9 - 2.7E9 Capacitive	8.5E9 - 4.5E9 Capacitive	3.125	20	1412 +/- 125 glue /Coh.
USACE System 5-E-Z	0	0	0.09	0.15	0	6.5E9 - 4.1E9 Capacitive	7.5E9 - 2.2E9 Capacitive	1	100	1012 +/- 226 glue /Coh.
System 1A	0.05" blisters	0.01" blisters	0.37"+/- .12	0.42" +/- .08	0.00"	1.0E5 - 1.0E4 Resistive	1.1E5 - 1.7E7 Resistive	Complete failure within 1 week	35	1390 +/- 434
System 1B	0.05" blisters	0.01" blisters	0.33"+/- .13	0.43" +/- .08	0.00"	2.8E3 - 9.3E2 Resistive	6.5E5 - 8.6E4 Resistive	Complete failure within 1 week	50	565 +/- 36
System 2A	0.01"	0.04"	0.63"+/- .06	0.60"+/- .18	0.00" discolored	6.1E9 - 7.2E8 Resistive	4.6E9 - 7.2E8 Resistive	0.25" radius	30	1617 +/- 196
System 2B	0.03"	0.06"	0.26"+/- .01	0.31"+/- .03	0.00" discolored	1.3E10 - 4.8E8 Resistive	7.8E9 - 2.4E6 Resistive	0.25" radius	50	941 +/- 103
System 3A	0.00"	0.00"	0.10"+/- .01	0.07"+/- .01	0.00"	7.7E8 - 2.7E8 Resistive	8.3E8 - 2.6E8 Resistive	Random blisters throughout	45	934 +/- 102
System 3B	0.02"	0.01"	0.06"+/- .01	0.11"+/- .01	0.00"	8.6E8 - 1.6E7 Resistive	4.9E8 - 1.7E7 Resistive	Random blisters throughout	40	644 +/- 165
System 4A	0.01"	0.04"	0.67"+/- .27	0.34"+/- .10	0.00"	1.4E9 - 1.7E8 Resistive	4.8E8 - 5.1E7 Resistive	Large 3" diameter blister closest to anode away from defect	45	1707 +/- 187
System 4B	0.00"	0.06"	0.23"+/- .05	0.46"+/- .10	0.00"	1.2E9 - 1.4E8 Resistive	1.5E9 - 8.9E7 Resistive	Large 3" diameter blister closest to anode away from defect	40	574 +/- 91

Appendix Table 3. Summary results of polysiloxane data set after 5000 hours of exposure

	HAR Immersion	DI Immersion	PRO	BOR	QUV	EIS HAR	EIS DI	Cathodic Disbondment	Slurry Erosion	Tabor Abrasion	Impact	Pull-off Adhesion	Pull-off Adhesion (Wet)	Knife Adhesion (Wet)
	undercutting (in) Max	undercutting (in) Max	undercutting (in) Ave	undercutting (in) Ave	undercutting (in) Ave	(High - Low Impedance value at 0.01Hz), (Ohms)	(High - Low Impedance value at 0.01Hz), (Ohms)	Radius (inches)	Stabilized weight loss rate (g/hr)	Total weight loss, mg	No cracking or holidays (inch-lbs)	Stress (psi)	Stress (psi)	ASTM Rating (0-10)
USACE System 4	0.11	0.11	0.43	0.35	0	7.8E9 - 2.7E9 Capacitive	8.5E9 - 4.5E9 Capacitive	3.125	0.039 +/- 0.003	164	20	1412 +/- 125 glue /Coh.	769 +/- 56 glue	4
USACE System 5-E-Z	0	0	0.09	0.15	0	6.5E9 - 4.1E9 Capacitive	7.5E9 - 2.2E9 Capacitive	1	0.041 +/- 0.004	164	100	1012 +/- 226 glue or glue/Coh.	No data	4
Polysiloxane 1 DTM	0	0	0.46	0.29	0	3.8E10 - 2.4E10 Capacitive	3.9E10 - 1.1E10 Capacitive	0	0.061 +/- 0.014	125	30	1965 +/- 344 glue/Coh.	1479 +/- 143 Coh./ Adh.	10
Polysiloxane 2 DTM	0	0	0.41	0.32	0	3.2E10 - 1.8E10 Capacitive	2.6E10 - 1.9E10 Capacitive	0	0.072 +/- 0.006	122	82	1059 +/- 256 Coh.	1149 +/- 176 Coh./ Adh.	10
Polysiloxane 3 DTM	0	0.07	0.5	0.45	0	3.0E8 - 9.1E7 Resistive	3.6E8 - 1.4E8 Resistive	0	0.066 +/- 0.013	108	35	2325 +/- 461 Coh.	1243 +/- 79 Coh./ Adh.	10
Polysiloxane 4 DTM	Blisters	Blisters	Blisters/ cracks to substrate	Blisters/ cracks to substrate	Blisters/ cracks to substrate	Withdrawn from testing	Withdrawn from testing	No data	No data	182	10	No data	No data	No data
Epoxy A/ Polysiloxane 1	0.12	0.03	0.36	0.35	0	4.4E10 - 2.0E10 Capacitive	3.0E10 - 1.3E10 Capacitive	0	0.061 +/- 0.014	125	52	1867 +/- 168 Coh.	1871 +/- 8 Coh.	10
Epoxy B/ Polysiloxane 2	0	0.09	0.42	0.37	0	1.2E10 - 4.8E9 Capacitive	1.1E10 - 6.8E9 Capacitive	0	0.072 +/- 0.006	122	52	1314 +/- 116 Coh.	1275 +/- 168 Coh.	10
Epoxy B/ Polysiloxane 3	0	0.09	0.38	0.46	0	3.1E9 - 6.0E8 Resistive	2.4E9 - 6.2E8 Resistive	0.25	0.066 +/- 0.013	108	36	1285 +/- 120 Coh.	1028 +/- 251 Coh.	10
Epoxy A/ Polysiloxane 4	0	0	0.23	0.29	0	2.6E10 - 1.0E10 Capacitive	2.2E10 - 1.0E10 Capacitive	0	0.125 +/- 0.008	182	40	1681 +/- 420 Coh.	1207 +/- 233 50/50 Coh.	10

