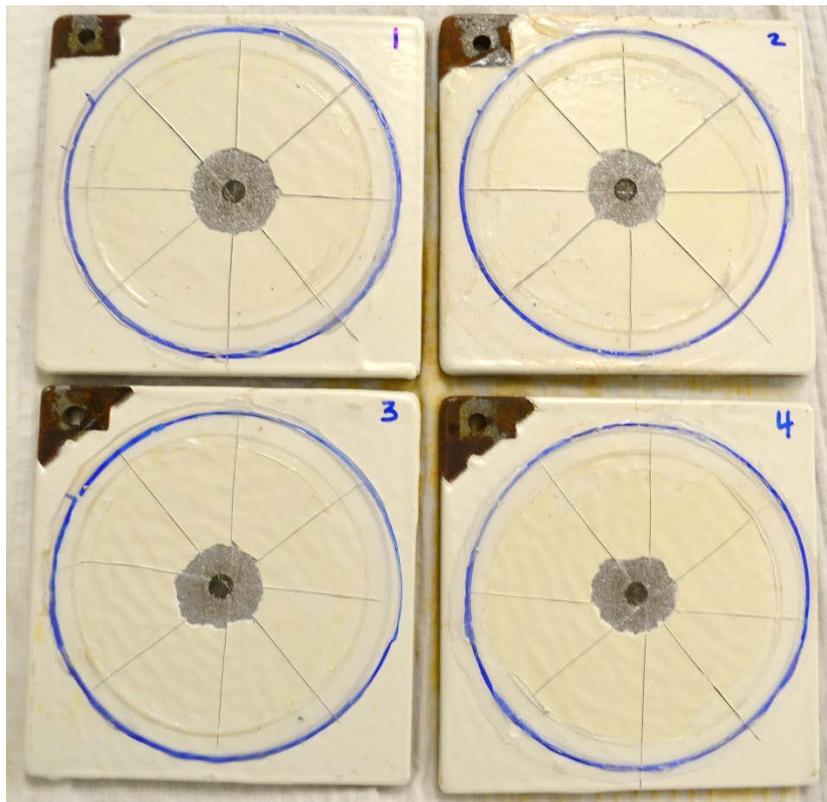


RECLAMATION

Managing Water in the West

Comparison and Evaluation of Cathodic Disbondment Standards

Research and Development Office
Science and Technology Program
Final Report ST-2019-8100-01
Technical Memorandum No. 8540-2019-29



Mission Statements

The U.S. Department of the Interior protects America's natural resources and heritage, honors our cultures and tribal communities, and supplies the energy to power our future.

The Department of the Interior protects and manages the Nation's natural resources and cultural heritage; provides scientific and other information about those resources; and honors its trust responsibilities or special commitments to American Indians, Alaska Natives, and affiliated island communities

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Science and Technology Program**

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Comparison and Evaluation of Cathodic Disbondment Standards

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Acronyms and Abbreviations

CD	Cathodic disbondment
CP	Cathodic protection
G8	ASTM G8, Standard Test Methods for Cathodic Disbonding of Pipeline Coatings
in	Inches
mV	Millivolts
OD	Outer diameter
TM0115	NACE TM0115, Cathodic Disbondment Test for Coated Steel Structures under Cathodic Protection

Executive Summary

Corrosion mitigation systems are necessary to prevent corrosion on metallic structures and improve their service life. The most effective system is a combination of protective coatings and cathodic protection (CP). However, when using the two methods in conjunction, care must be taken to avoid damage to the coating caused by the cathodic current. Cathodic disbondment (CD) testing is used to evaluate the robustness of coatings under CP. However, there are over 20 CD testing standards available worldwide which questions the appropriateness of comparing results across different test methods.¹ To address this concern, NACE formed a technical exchange group—TEG 349X—to examine the differences between the test methods, and ultimately to develop a more universal method: NACE TM0115.² The new method provides a simplified, accelerated test with high-temperature testing options, and includes all parameters found by TEG 349X to be significant to CD results.

In 2018, researchers at the Bureau of Reclamation compared the new NACE TM0115 test method to ASTM G8, their current practice for CD testing.³ The two test methods were compared for results and ease of implementation. This study showed that CD values were consistent between the methods, but that specimen geometry produced a varying effect on CD results.³ This contradicted the findings of TEG 349X, which showed CD values to be independent of specimen geometry, and prompted further study. A subsequent study investigated the impact of specimen geometry on CD by comparing five specimen geometries evaluated via the NACE TM0115 standard. Researchers also considered the parameters of polarized and applied potential because the 2018 study's findings showed that even at a constant applied potential, their polarized potential varied. Due to the comparative nature of the CD test, all test cells must be subject to the same testing conditions, so any variation in environment between cells can significantly impact results.

The results of the subsequent study showed that specimen geometry had a varying impact on CD results depending on the coating system. The epoxy-coated specimens had no variation in size or reproducibility of disbonded area, whereas the vinyl-coated specimens had larger disbonded areas and decreased reproducibility as specimen curvature increased.³ This suggests that comparing results across CD tests that use different specimen geometries may not be appropriate. Supporting the findings of the previous study, it was again found that polarized potential was consistently 300 millivolts (mV) less negative than applied potential, although there was no correlation between polarized potential and disbondment values.³

Both studies were presented at NACE International CORROSION conferences and published in the conference proceedings. A truncated version of the first paper was also published in the Materials Performance magazine. This report summarizes the findings and provides the published manuscripts as appendices.

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Introduction

Corrosion mitigation systems are necessary to prevent corrosion on metallic structures and improve their service life. The most effective system is a combination of protective coatings and cathodic protection (CP). The CP current requirement to adequately protect a structure can be dramatically reduced by applying a coating to the exposed metal. However, to achieve the most efficient corrosion protection system, the protective coating and cathodic protection system must complement each other, i.e. the CP cannot cause damage to the coating. If the distance between the anodes and structure is too small, or the driving voltage is too high, the CP system may cause cathodic damage to the coating.⁴ Cathodic disbondment (CD) is a phenomenon whereby organic coatings lose adhesion at the steel interface due to the effects of over-polarization by a cathodic protection system.⁵ CD has been called the “most important degradation mechanism for organic coatings on submerged steel.”²

To compare the ability of different coatings to withstand cathodic polarization, CD testing can be done in a laboratory setting. However, there are over 20 CD testing standards available worldwide, each with different experimental set-ups and parameters.¹ This can make cross-comparison of results difficult between laboratories that use different test methods. In 2015, NACE published a new CD test method, NACE TM0115, Cathodic Disbondment Test for Coated Steel Structures under Cathodic Protection (TM0115). The goal of TM0115 was to produce a simpler, more universal test method. Researchers at the Bureau of Reclamation compared the new test method for results and ease of implementation against their current practice of using ASTM G8, Standard Test Methods for Cathodic Disbonding of Pipeline Coatings (G8). This testing was performed in 2017, and results from this research led to further work using TM0115 to explore the impacts of specimen geometry and applied and polarized potential on CD test results.

Laboratory Testing

Researchers conducted two research studies investigating the new NACE TM0115 test method—“Comparison of Cathodic Disbondment Test Methods for Water Infrastructure Coatings” in 2017 and “Impact of Specimen Geometry on Cathodic Disbondment Testing for Protective Coatings” in 2018.^{3,6} The papers were presented at NACE International CORROSION conference in 2018 and 2019 and published in the proceedings as NACE C2018-10914 and NACE C2019-13367, respectively. The first paper was also selected for a highlight in Materials Performance magazine.⁷

Comparison of CD Test Methods

To compare TM0115 and G8 for ease of implementation and CD results, CD testing was performed on 34 specimens in 16 test vessels. Testing was conducted with half of the vessels following G8 and the other half following TM0115.^{2,8} Three specimen geometries were used: flat panels (4 in x 6 in x 0.125 in), 2-in tubes (2-in outer diameter [OD], 18 in long), and 4-in tubes (4-in OD, 18 in long). Half of the specimens from each geometry type were coated with a commercial industrial maintenance epoxy and the other half with a zinc-rich vinyl primer and

vinyl topcoats. CD testing was conducted for 30 days for G8 specimens and 28 days for TM0115 specimens, as specified by each test method.

The details of the experimental setup, results and discussion, and conclusions can be found in conference article NACE C2018-10914, included as Appendix A.⁶

Impact of Specimen Geometry on CD Testing

Based on the unexpected results from the “Comparison of CD Test Methods” study, further work was performed using only TM0115 with 80 specimens in 32 test vessels. This research investigated the effects of specimen geometry and applied and polarized potential on CD results. Testing was conducted with five specimen geometries: flat panels (4 in x 6 in x 0.125 in), square panels for the attached cell method (4 in x 4 in x 0.25 in), 2-in tubes (2-in OD, 12 in long), 3-in tubes (3-in OD, 12 in long) and 4-in tubes (4-in OD, 12 in long). Conforming with the previous study, half of the specimens from each geometry were coated with a commercial industrial maintenance epoxy and the other half with a zinc-rich vinyl primer and vinyl topcoats.

The details of the experimental setup, results and discussion, and conclusions can be found in conference article NACE C2019-13367, included as Appendix B.³

Conclusions

Brief conclusions from each study are included in this report. For a more thorough explanation of conclusions and recommendations, refer to each paper included in the appendices.

Comparison of CD Test Methods

- TM0115 and G8 did not yield a significant difference in CD for the coatings evaluated.
- Both showed variation of CD for a given coating system by specimen geometry, which necessitates further investigation into the causes of these observations.
- Both produced polarized potentials 300-400 mV less negative than applied potentials.
- TM0115 has improved ease of implementation, with several advantages over G8.
- Several recommended improvements to TM0115 include definition of polarization terms, clarification of electrolyte maintenance, and more detailed test set-up schematics.

Impact of Specimen Geometry on CD Testing

- Specimen geometry did not influence CD or reproducibility for the epoxy-coated system.
- The vinyl-coated system had increased CD and decreased reproducibility with increasing specimen curvature.
- The polarized potential was typically 300 mV less negative than the applied potential.
- Comparison of the polarized potential to the disbonded area showed no correlation.
- Data cross-comparison may not be appropriate when different geometries are used for CD testing.

References

1. B.T.A. Chang, et al., "Critical Evaluation of International Cathodic Disbondment Test Methods," presented at 17th International Corrosion Congress, Las Vegas, NV, <http://www.chartercoating.com/library.php> (accessed December 2018).
2. NACE TM0115 (2015), "Cathodic Disbondment Test for Coated Steel Structures under Cathodic Protection" (Houston, TX: NACE International).
3. G. Weber, et al., "Impact of Specimen Geometry on Cathodic Disbondment Testing for Protective Coatings," CORROSION 2019, paper no. 13367 (Nashville, TN: NACE International, 2019).
4. A. W. Peabody, "Control of Pipeline Corrosion," (Houston, TX: NACE International, 2001).
5. F. Mahdavi, et al., "Techniques for Testing and Monitoring the Cathodic Disbondment of Organic Coatings: An Overview of Major Obstacles and Innovations," Progress in Organic Coatings 105, (2017): p. 163.
6. G. Weber, et al., "Comparison of Cathodic Disbondment Test Methods for Water Infrastructure Coatings," CORROSION 2018, paper no. 10914 (Houston, TX: NACE International, 2018).
7. G. Weber, B. J. Merten, and J. D. Torrey, "Comparison of Cathodic Disbondment Test Methods for Water Infrastructure Coatings," CORROSION 2018, Pipeline Coatings" (West Conshohocken, PA: ASTM International).

Appendix A – NACE C2018-10914

Comparison of Cathodic Disbondment Test Methods for Water Infrastructure Coatings

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ABSTRACT

Protective coatings are used ubiquitously as the primary means of corrosion defense for buried or immersed metallic structures. Cathodic protection is often used in conjunction with coatings as a secondary means of corrosion protection, however it can also have detrimental effects on the coating itself if not properly applied. Cathodic disbondment testing is used to measure a coating's susceptibility to loss of adhesion to the substrate due to cathodic polarization.

NACE recently published a new cathodic disbondment test method, TM0115-2015, Cathodic Disbondment Test for Coated Steel Structures under Cathodic Protection. This research compares and contrasts NACE TM0115 to ASTM G8, Standard Test Methods for Cathodic Disbonding of Pipeline Coatings. The paper highlights the strengths and challenges of each test method as applied to coatings used in water infrastructure corrosion control.

Key words: cathodic disbondment, protective coatings, cathodic protection

INTRODUCTION

In its mission to manage water resources in the western United States, the Bureau of Reclamation utilizes billions of dollars' worth of infrastructure, much of which is steel, both in burial and immersion service. To prevent corrosion on this steel infrastructure, a combination of protective coatings and cathodic protection (CP) are implemented, offering a greater level of protection to a steel structure than either technique on its own.¹

When CP is used as a secondary protection method, the protective coating must be able to withstand cathodic polarized potentials, often in the range of -0.85 ± 0.20 volts vs copper-copper sulfate reference electrode (V_{CSE}). Polarizing the steel infrastructure surface converts available sites to cathodes by electrochemical reaction, which is primarily the reduction of oxygen or dissociation of water, Equations (1) and (2). Organic coatings may undergo cathodic disbondment (CD), or loss of adhesion, at the

steel interface as a result of the reduction reaction and the corresponding reaction products. Mahdavi demonstrated that Equation (1) is the dominant reaction for applied potentials between the open circuit potential and -1.10 volts vs saturated silver-silver chloride reference electrode (V_{SSC}), whereas Equation (2) dominates at potentials more negative than -1.10 V_{SSC} and greatly accelerates CD of the coating.² Note that Mahdavi and other authors commonly report applied potential rather than the polarized potential.



The CD mechanism for a protective coating is an effect of its susceptibility to high pH, hydrogen gas evolution, and other possible damaging factors.³⁻⁵ Because of the largely chemical nature of cathodic polarization, it is the adhesive bond chemistry, rather than physical adhesion, that provides the greater contribution to coating CD resistance.⁶ It has been written that CD is the “most important degradation mechanism for organic coatings on submerged steel.”⁷

Terminology

Cathodic protection testing literature varies in its use of key terms. For the purposes of this paper the following terminology is used:

Corrosion Potential- the potential of a corroding surface in an electrolyte measured under open-circuit conditions relative to a reference electrode (also known as electrochemical corrosion potential, free corrosion potential, open-circuit potential).⁸

Cathodic Polarization- a forced active (negative) shift in electrode potential.⁸

Polarized potential- the potential across the structure/electrolyte interface that is the sum of the corrosion potential and the cathodic polarization (also known as instant-off potential).⁸

Applied potential- the voltage applied to a structure by an external power supply, i.e., the sum of the corrosion potential, the cathodic polarization, and the voltage drops other than that at the structure/electrolyte interface.

Cathodic Disbondment Testing for Protective Coatings

To determine the service performance of different coatings under CP, and to ensure that disbondment will not occur on structures in the field, a large variety of CD laboratory tests have been developed. Currently there are at least twenty-two test standards for CD testing, with no single test standard being universally accepted.

Many authors have explored the impact of physical and chemical factors on CD for organic coatings as well as the effect of CD testing parameters.^{2,9-13} Several experimental factors of CD testing are shown to have an impact on the validity and repeatability of laboratory results. An early study by Rodriguez et al. showed that increased alkalinity, higher applied potential, and cyclic wetting and drying were all factors that contributed to greater cathodic disbondment.¹⁴ Cameron found that anode configuration, test temperature, heating method, and frequency of replacing electrolyte were the most critical factors.¹⁵ Holub et al. compared twenty-two test standards and found that test duration, current control, coating film thickness, and frequency of replacing electrolyte, which impacted hypochlorite concentration, yielded the greatest impact on results.¹⁶ Each of these studies emphasized that the

large variation in experimental parameters among the different test standards impacts and limits the ability to compare studies performed following different CD test standards.

To address the differences in experimental parameters between CD test standards, NACE International⁽¹⁾ Technical Exchange Group TEG 349X was created to perform a comprehensive evaluation of international CD test methods. This evaluation looked at the differences in test parameters of 8 international standards and how each difference impacted the CD testing results. Based on the significance of each factor on the results of the experiment, the group developed a basis for a new, universally-applicable CD test standard. The findings of the group showed that the most significant factors were dissolved oxygen content, electrolyte alkalinity, applied potential, specimen surface profile and coating film thickness, hypochlorite formation, and test temperature.¹⁷ Based on this list, the group developed a table of recommendations for test conditions that could be applied in a laboratory environment to best replicate field conditions.

In 2012, NACE Technical Committee TG 470 was formed to create a universal CD testing standard, and using the findings of TEG 349X, the group released NACE TM0115 in 2015.¹⁸ This new standard provides guidelines for a CD test method with an accelerated test procedure that is applicable for all service temperature ranges and specimen types. The standard also takes into account the most significant test factors on cathodic disbondment to create a universal set of test parameters that will be most accurate to conditions experienced by coatings in the field.

CD testing for coating performance is commonly run following the ASTM International⁽²⁾ G8 or G95 standard, the ASTM G95 test method being used when an attached cell method, rather than immersion, is desired.^{19,20} The present study provides a side-by-side examination of NACE TM0115 and ASTM G8 test methods.^{18,19} Table 1 (below) provides the experimental parameters of these standards; it includes ASTM G95 parameters to provide comparison with the NACE standard which includes test methods for both attached cell and immersion CD testing. However, the attached cell methods were not studied.²⁰

While obtaining comparable results between the two tests is important, the study also placed emphasis on interpretability and clarity of the standard and ease of implementation.

EXPERIMENTAL PROCEDURE

NACE TM0115 and ASTM G8 Standard Test Methods, hereafter referred to as NACE and ASTM test methods, respectively, are applied to the evaluation of two coating system types and three specimen geometries. Test vessels were set up following both standards. An initial round of testing was performed to determine the best test methodology, and a secondary round of testing applied this information to compare the results of each test standard. Observations were made on similarities and differences between each test method with regard to setup, ability to maintain the experimental parameters, and results. All specimens were monitored at least twice weekly for electrolyte pH and temperature, applied current, applied potential, and polarized potential.

Sample Preparation

NACE TM0115 standard allows the use of flat panel specimens in an immersion bath, unlike ASTM G8; therefore, both flat panel and tube-shaped steel specimens were included in this investigation. Flat panels had dimensions of 4 in (10.2 cm) x 6 in (15.2 cm) x 0.125 in (0.3 cm); tube-shaped specimens had a length of 18 in (45.7 cm) and an outer diameter (OD) of 2 in (5.1 cm) or 4 in (10.2 cm).

⁽¹⁾ NACE International, 15835 Park Ten Place, Houston, Texas 77084.

⁽²⁾ ASTM International, 100 Barr Harbor Dr., West Conshohocken, PA 19428.

**Table 1
Comparison of Test Parameters**

Test Parameter	NACE TM0115 ¹⁸	ASTM G8 ¹⁹	ASTM G95 ²⁰
Test Method	Salt Bath and Attached Cell	Salt Bath	Attached Cell
Specimen Geometry	Flat panel, curved panel, or tube	Tube	Flat panel, curved panel, ring, or in-service pipe
Specimen Size	Attached Cell: 4 in x 4 in x 0.25 in min.; Salt Bath: tube 2 in OD x 4 in length min.; flat panel 4 in x 6 in x 0.125 in	Any size to allow 36 in ² min. to be immersed; 1 ft ² preferable	Large enough to accommodate a 4 in diameter tube centered over the holiday
Number of Replicates	3 (min.)	Not specified	Not specified
Test Vessel	Attached Cell: plastic or glass tube 3-4 in diameter and 4 in height; Salt Bath: nonreactive, nonconductive vessel of suitable size for specimen(s)	Nonconductive vessel of suitable size for specimen	Transparent plastic or glass 4 in diameter tube and 5 in height
Specimen Configuration	Attached Cell: no instruction Salt Bath: Min. 1 in between anode/specimen, anode/wall, and specimen/specimen; equal spacing between anode and multiple specimens in same bath	Min. 1 in between specimen/bottom; min. 1.5 in separation between specimen and anode, wall, and other specimens	Edge of fritted disc is 1 in above holiday and 0.5 in offset from holiday
Holiday Geometry	0.25 in diameter x 0.02 in deep hole drilled with flat bit	1 or 3 holidays; hole diameter not less than three times the coating thickness, min. 0.25 in drilled with cone point bit	0.125 in diameter hole drilled with cone point bit
Holiday Orientation	Attached Cell: centered in cell Salt Bath: centered, facing anode	Middle of immersed length, facing away from anode	Centered in cell
Electrolyte Type and Concentration	Deionized/distilled water with 3 mass % NaCl	Potable tap water with 1 mass % each NaCl, Na ₂ SO ₄ , and Na ₂ CO ₃ ; maintain daily	Deionized/distilled water with 3 mass % NaCl; maintain daily
Type of Anode	Platinum, mixed metal oxide (MMO), or other appropriate anode which does not corrode during test period	Magnesium anode, impressed current anode	Platinum wire 0.02 in diameter
Anode Isolation	Glass tube with glass wool plug	None	Immersion tube with fritted disk
Applied Potential	-1.38 ± 0.02 V _{SSC} (-1.50 V _{CSE}); adjust daily	-1.45 to -1.55 V _{CSE}	-3 V _{CSE} ; adjust min. twice weekly
Duration	28 days or as specified	30 days or as specified	90 days
Temperature	Room temperature (RT) or elevated	RT (21–25°C)	RT (21–25°C)
Reference Electrode	Ag/AgCl (saturated KCl)	Cu/CuSO ₄ or calomel	Cu/CuSO ₄ or calomel
Measurements and Frequency	pH: initial and weekly; Cathodic current: min. twice weekly; Disbondment exam: final	Applied potential, polarized potential, cathodic current: initial, final (optional) Disbondment exam: final	pH: initial; Disbondment exam: final

Two different coating systems were included to provide a more thorough comparison. For each of the three sample geometries, half of the specimens were coated with a commercial industrial maintenance epoxy and half were coated with zinc-rich vinyl primer followed by vinyl topcoats (United States Army Corps of Engineers System 5-E-Z).

All specimens were degreased, solvent cleaned, and abrasive blasted to obtain a white metal finish.²¹ The resulting surface profile was 3.9 ± 1.6 mils for the epoxy (2–3 mils manufacturer’s recommendation) and 3.3 ± 1.0 mils for the zinc-rich vinyl coating (1.5–2.5 mils manufacturer’s recommendation). After coating, dry film thickness (DFT) measurements were recorded and found to be within manufacturer recommendations (Table 2). The recommended total DFT for the epoxy is 15–30 mils. The recommended DFT for the vinyl is a minimum of 7 mils.

Table 2
Coating Dry Film Thickness Values

Coating System	Specimen Geometry (Replicates)	DFT (mils)	Standard Deviation (mils)
Epoxy	flat panels (8)	21.3	3.6
	2 in tubes (5)	15.4	1.6
	4 in tubes (4)	20.0	2.9
Vinyl	flat panels (8)	10.9	1.4
	2 in tubes (5)	9.0	1.5
	4 in tubes (4)	7.6	1.0

To prepare the specimens for the CD test, a rotary tool was used to grind a small area of coating above the immersion line down to bare steel. A drill was used to produce a hole through the specimen, and a bolt was used to fasten a metallic structure-to-power supply connection at this location. Each specimen received one 0.25-in (0.6 cm) diameter holiday drilled with a flat head end mill bit, as specified by the NACE test method. While the ASTM test method specifies a cone-shaped bit, it has been shown that holiday shape does not affect the results of the test.¹⁷

Test Set-Up

To provide an initial evaluation of the test methods, eight flat panel specimens and nine tube-shaped specimens were tested in a total of eight test vessels by the salt bath method. Three of the test vessels were designed in accordance with the ASTM G8 test method and held either two 2-in tube specimens or one 4-in tube specimen each. Five of the test vessels were designed in accordance with the NACE TM0115 test method and held either four flat panels, three 2-in tube specimens, or one 4-in tube specimen each. The test vessels were plastic five-gallon buckets, and a custom-milled clear acrylic plastic sheet was placed on top of each bucket to maintain the position of the specimens and anode, provide a location for the reference electrode when measuring the applied and polarized potentials, and prevent electrolyte evaporation. During testing it was determined that the reference electrode location in the test vessel did not have a measurable effect on the potential readings.

In the second round of testing, the same combinations of coating and geometry were tested. For the test vessels containing tube-shaped specimens, the same test set-up was used as in the first round of testing with plastic five-gallon buckets and custom-milled acrylic sheets. For test vessels containing flat panels, an insulated aluminum frame was created to hold the specimens in place rather than using the custom-milled acrylic sheet. The aluminum frame was found to be more effective at suspending the flat panels and maintaining their location in the test vessel. See Figures 1-3 for test vessel set-ups and schematics for the large and small OD tube-shaped specimens and flat panel specimens.

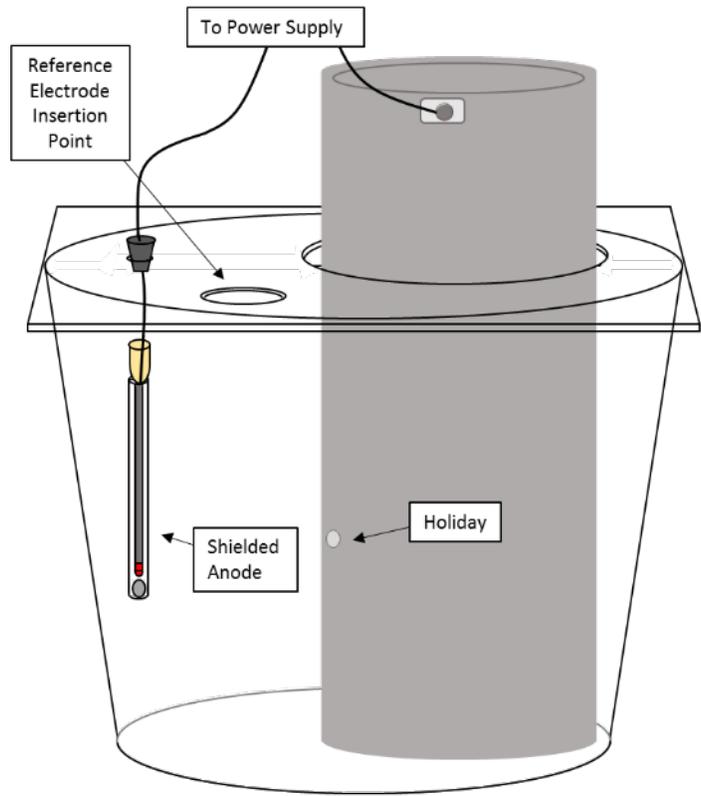


Figure 1. Photograph and schematic of experimental set-up for 4-in diameter tube per NACE TM0115.

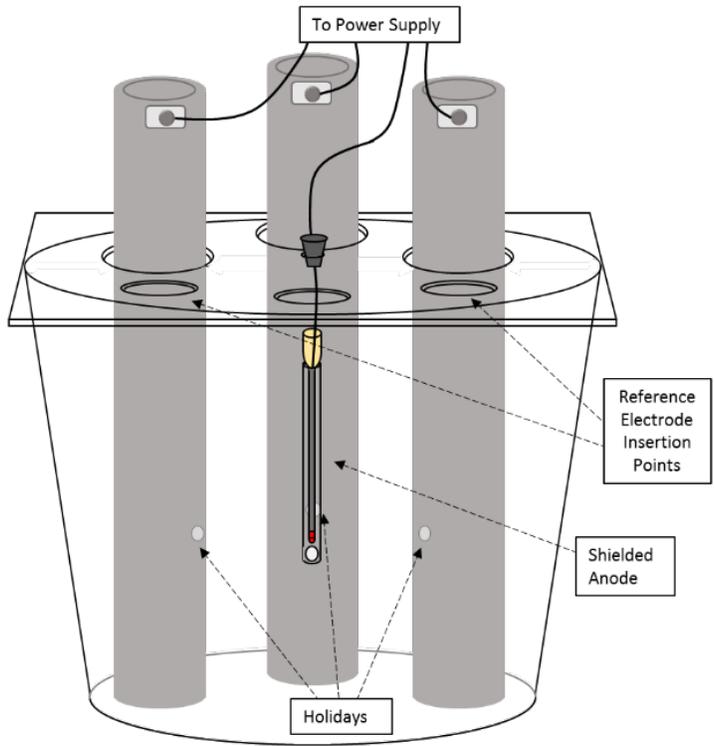


Figure 2. Photograph and schematic of experimental set-up for 2-in diameter tubes per NACE TM0115.

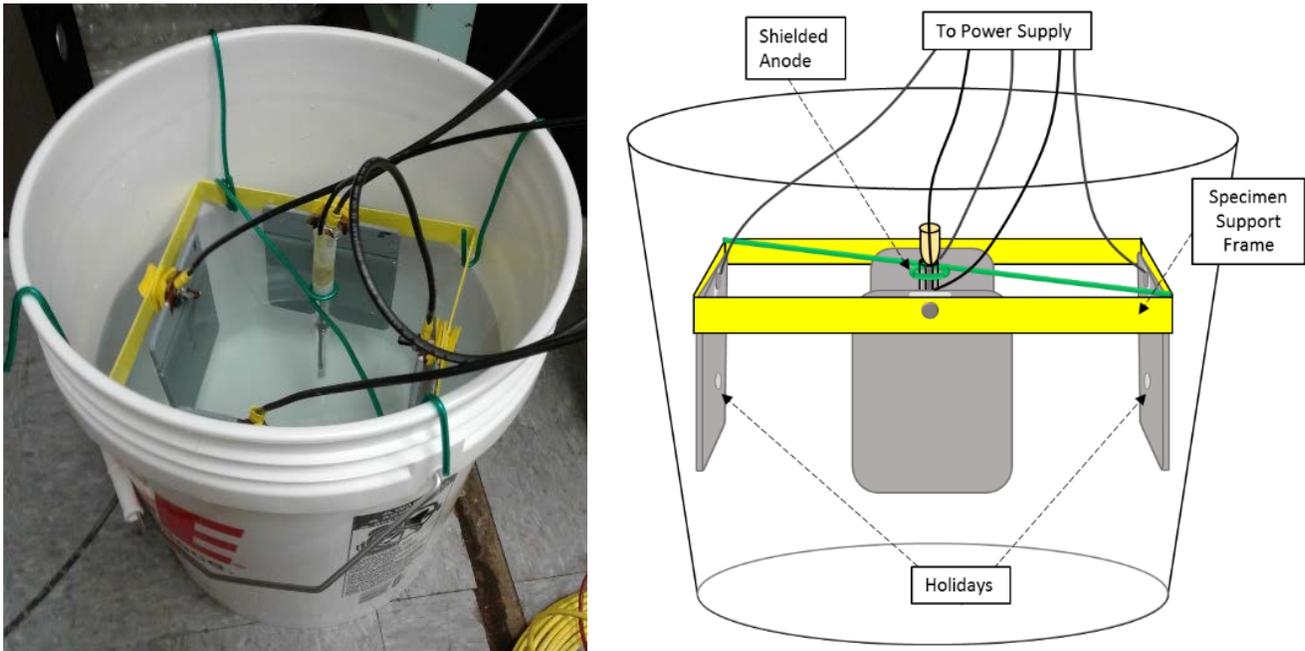


Figure 3. Photograph and schematic of experimental set-up for flat panels per NACE TM0115.

Platinum clad anodes, 0.125-in (0.3 cm) diameter, with a copper core were used. As required by the standard, all of the anodes in NACE TM0115 test vessels were placed in an anode isolation system consisting of a glass tube with a glass wool plug at the end. The top of the glass tube was left open to allow chlorine gas to escape, and the anode assembly was suspended from the acrylic sheet by a rubber stopper. Anodes in the ASTM G8 experiments were left bare.

In the first round of testing, each test vessel was filled with a salt solution as dictated by the respective standard to a volume that placed the holiday at a depth halfway between the bottom of the bucket and the surface of the solution. This yielded an immersed area of 144 in² for the 4-in pipes and 43 in² for the panels and 2-in pipes. The NACE test method does not specify an immersed area, just a specimen geometry, but the ASTM test method requires a minimum immersed area of 23,227 mm² (36 in²) with a preferred immersed area of 92,900 mm² (144 in²). The NACE TM 0115 test vessels were filled with a solution of deionized (DI) water and 3% sodium chloride by mass, and the ASTM G8 test vessels were filled with DI water and 1% by mass of the following: sodium chloride, sodium sulfate, and sodium carbonate. The electrolyte level in the test vessels was maintained by adding DI water. In the second round of testing, the electrolyte was replaced three times during the test.

The test specimens were connected to the power supply through 0.1-ohm shunts. This is a deviation from both standards, which call for 1.0-ohm precision resistors to measure cathodic current.

Procedure

The CD test were conducted for 28 days and 30 days for NACE TM0115 and ASTM G8, respectively. Over the duration of each test period, and for comparison purposes, all test parameters were monitored at least twice weekly. This included: applied potential, polarized potential, applied current and the electrolyte temperature and pH. After recording all measurements from the as-found specimen, each specimen would be adjusted to the mandated applied potential (-1.38 V_{SSC}/-1.5 V_{CSE}). At the conclusion of the testing period, the average CD radius at each holiday was measured and calculated as described in the NACE test method for all specimens for comparison purposes.

RESULTS AND DISCUSSION

The test results use a naming convention to distinguish specimens. The first letter designates whether the specimen underwent NACE or ASTM standard testing, given as “N” or “A,” respectively. The second letter provides the coating system, given as “V” for vinyl and “E” for epoxy. The final value provides the specimen geometry, with options of “P,” “2,” and “4” for panel, 2-in tube, and 4-in tube, respectively. As an example, the vinyl 4-in tube evaluated by the ASTM test method is “AV4.”

First Round Test Results

The data from the initial round of testing was discarded due to the magnitude of variations in the testing parameters. Rather, the first round allowed for an optimization of the test set-up and critique of the ease-of-use.

Accelerated Corrosion of Anode

Both standards require a non-consumable anode which does not corrode in the electrolyte solution, and the initial results used available platinum wire anodes with copper cores that were cut to size. The electrolyte end of the anode was not sealed, and the anodes were attached to copper cable using crimp-on butt splices. Corrosion resulted at both locations during CD testing, requiring mitigation.

Factory-sealed insulated anodes required special manufacturing. Instead, several corrosion protection techniques were tried to mitigate the copper wire corrosion, including a thin coating of two-part liquid epoxy, a thin coating of epoxy putty, and a polyethylene end cap with liquid epoxy. Both epoxy materials disbonded from the anode within several days of immersion; the plastic cap proved a more robust method of end-capping the anode. The test results showed no significant difference during anode core corrosion when compared to the test period in which the anode was end-capped.

The corrosion at the crimp connection between the anode and the copper wire caused the anode to lose connection to the power supply, resulting in no current being delivered to the system. Potting the connection in a two-part epoxy yielded a suitable solution to this challenge.

Second Round Test Results

Cathodic Disbondment

The results of the second round of testing include evaluation of the cathodic disbondment area after test completion. Figure 4 shows panel specimens upon removal from testing (at top) and the subsequent disbondment evaluation results by the NACE test method approach (at bottom). The example in Figure 4 shows a larger disbondment radius for the vinyl (bottom, left) than for the epoxy (bottom, right). The cable connection area and the specimen water immersion line also appear at the top of each specimen.

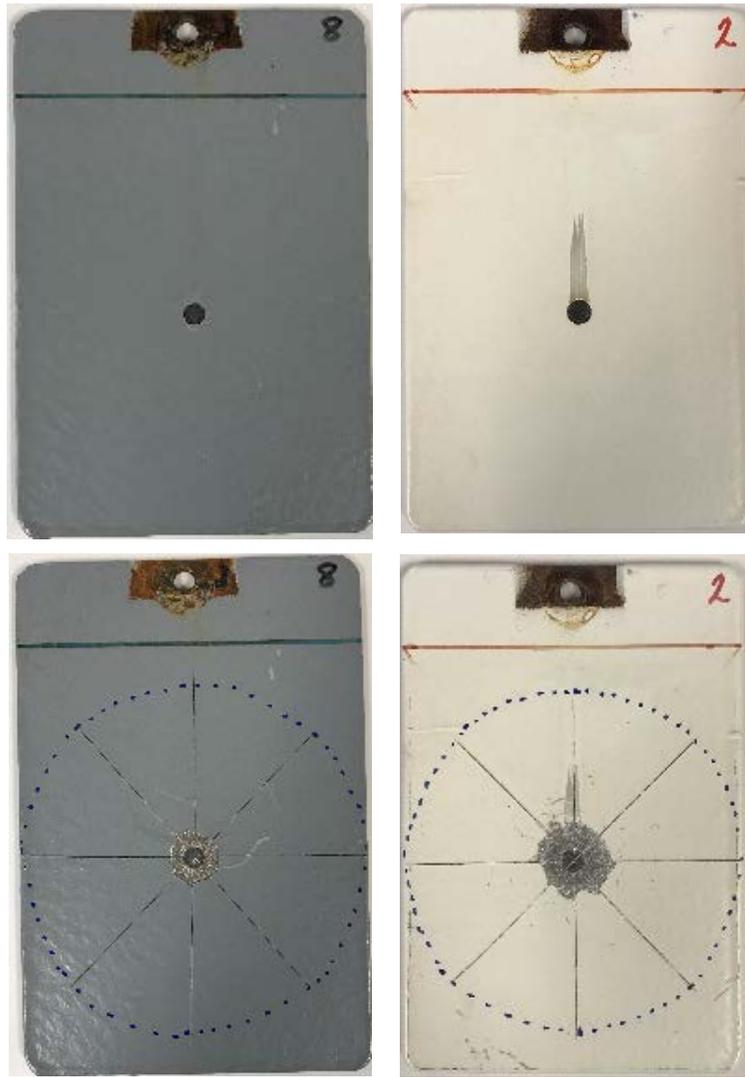


Figure 4. Cathodic disbondment results before (top) and after (bottom) application of NACE-specified method for vinyl (left) and epoxy (right) panel specimens.

Figure 5(a) (below) provides the results of the CD evaluation for all specimens. The results do not yield a significant difference in measured disbondment for NACE versus ASTM test methods for the coating system type and specimen geometry evaluated. However, the specimen geometry produces different outcomes for a given coating system type—vinyl disbondment was nearly one order of magnitude greater for the 4-in tube specimens than for the panels. Therefore, the data values produced by the cathodic disbondment test are useful only for comparison and ranking of materials evaluated by an identical procedure. It should be noted that the standard deviation for all 4-in tube results is calculated from multiple measurements on a single specimen. For other geometries, the standard deviation is the average value for all measurements taken on all specimens of that geometry.

pH and DFT

A comparison of the average measured pH in each test vessel over the duration of the experiment showed a deviation in the two test methods, as shown in Figure 5(b). The NACE test vessels are distinctly higher pH than the ASTM test vessels. Specifically, the resulting hydroxyl ion concentration is 1–2 orders of magnitude greater for the NACE test method. A likely reason for the pH difference is the formation of hypochlorite according to the following reaction:^{22,23}

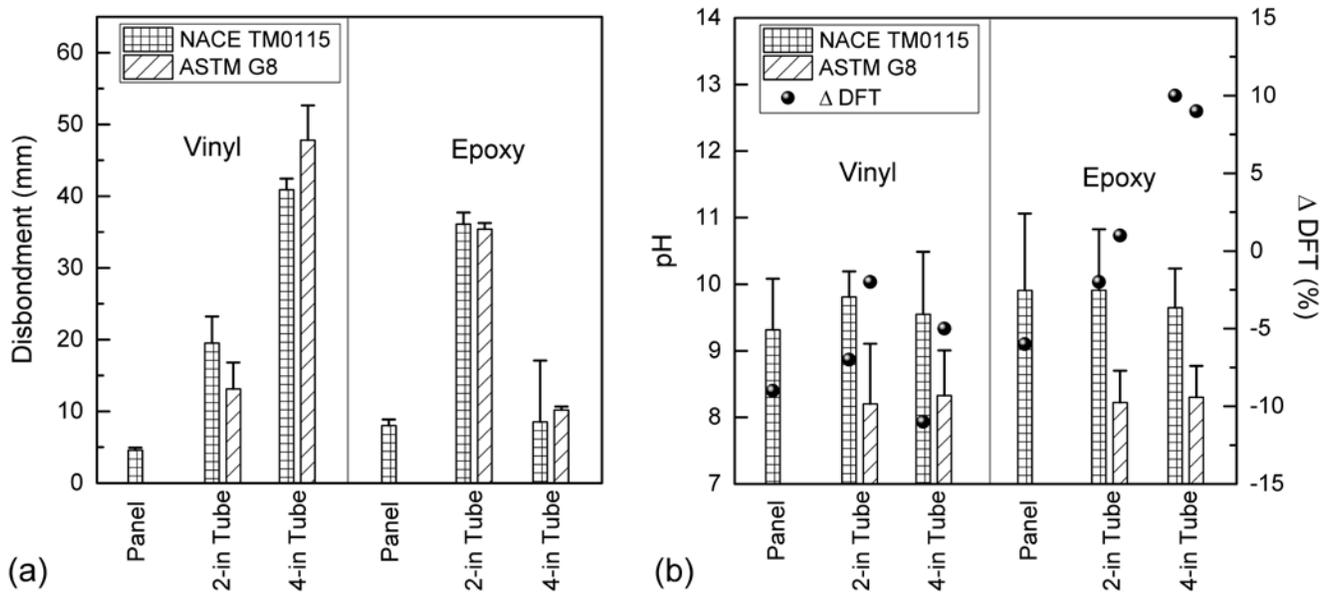


Figure 5. Results of (a) cathodic disbondment evaluation and (b) test vessel average pH and coating DFT change.

The ASTM test method does not specify anode isolation, and the hypochlorite reaction is much more likely to occur than in the NACE test vessels where the anodes are isolated and chlorine gas formed at the anode is channeled away from solution by the glass tube. In the case of the non-isolated anodes, the chlorine gas can go into solution and react with the hydroxyl ions formed at the anode. This reaction consumes hydroxyl ions and effectively lowers the measured pH.

This effect is not seen in the field due to the large separation distance between the anode and cathode. However, hypochlorite can degrade the coating and produce artificially poor results in lab tests, but Figure 5(a) and (b) does not reveal a correlation between the test vessel pH and the resulting degree of disbondment.

The change in coating DFT over the course of the experiment, measured before test initiation and after completion, is also shown in Figure 5(b). The NACE specimens experienced approximately 5% greater reduction in DFT compared to the ASTM specimens—the epoxy-coated 4-in tube is an exception. The higher pH in the NACE test vessels is a possible contributor to this observation. However, additional testing may be necessary to confirm the findings.

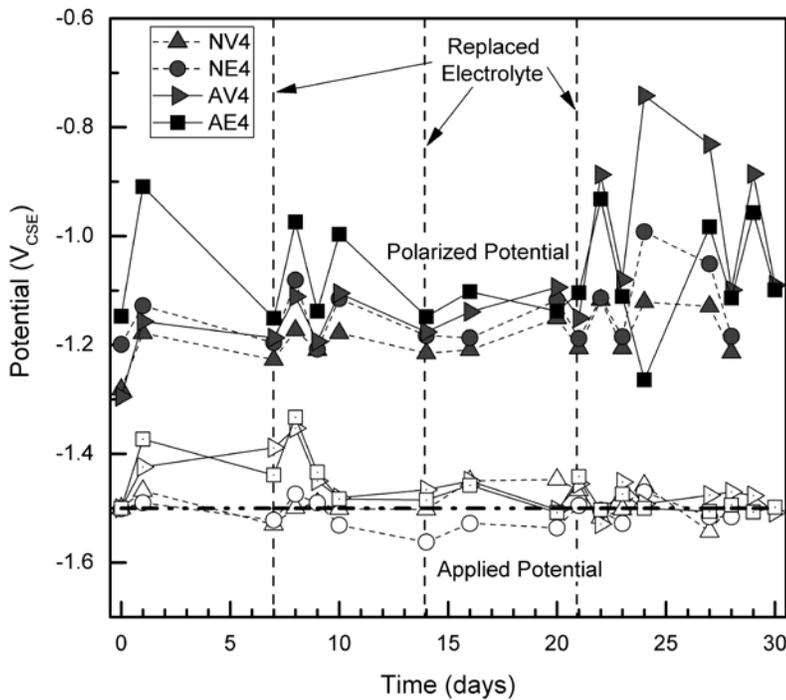


Table 3
Average Applied Potential and Polarized Potential for 4-in Tube Specimens

Specimen	Applied Potential (V _{CSE})	Polarized Potential (V _{CSE})
NV4	-1.49±0.03	-1.19±0.04
NE4	-1.51±0.02	-1.14±0.06
AV4	-1.46±0.04	-1.07±0.15
AE4	-1.47±0.05	-1.07±0.10

Figure 6. Measured (left) and average (right) potentials for 4-in diameter tubes over test duration.

Applied and Polarized Potential

Figure 6 provides the measured values for applied potential and polarized potential on the 4-in tube specimens. The open symbols are the applied potential, and the solid symbols are the polarized potential. All potential values were adjusted to V_{CSE} for direct comparison. A horizontal dotted line is given at $-1.5 V_{CSE}$ to illustrate the specified applied potential. The plot also includes three vertical lines to indicate the replenishment of electrolyte solution, which required interruption of the power supply, draining of the test vessel, and then refilling of the test vessel.

The applied potential is within the specified range for the majority of the test duration, although both ASTM specimens averaged on the positive end of the range. The ASTM specimens also averaged a more positive polarized potential, with greater variability over the test duration. The exchange of electrolyte produces a slight change in the positive direction for the measured applied potential. The polarized potential observations complement the applied potential trends for Days 1, 8, and 15. Near the end of the test (Day 22), the replacement of electrolyte shows the same decrease in polarized potential, but the applied potential is unchanged.

The polarized potential data begins to show differentiation between the test specimens in the final 10 days of testing. However, the applied potentials are steady and within the specified potential range. The two ASTM specimens show the greatest impact with a polarized potential more positive than $-1.0 V_{CSE}$ for most measurements. A more positive polarized potential, as seen for AV4 after Day 22 in Figure 6, may indicate an increase in exposed steel area, or CD. The 4-in tube specimens for the vinyl coating, including AV4, experienced a greater CD value than the epoxy coating for this geometry (see Figure 5(a) above), supporting the increased CD hypothesis. However, AE4 shows similar polarized potential values but has a contradicting, low CD value.

Test Method Analysis and Discussion

While the reported results of the CD tests are important, the main objective of this study is to compare and contrast the testing methods themselves, with emphasis on clarity, ease of implementation, and applicability. As with most experimental set-ups, both test methods demonstrate a learning curve for initial implementation.

Ease of Use

The NACE test method is generally straightforward and easy to follow; the standard has succeeded in streamlining the test parameters and methods. The prescribed configuration allowed for several specimens to be included in a single 5-gallon bucket; for example four 2-in diameter tubes could be arranged in the test vessel compared to only two for the ASTM test method. The smaller footprint allows for more specimens and yields a more robust data set per available test space. The NACE test method also allows for flat panel specimens to be used in the immersion test. The preparation of flat panels is easier to handle for laboratory-scale abrasive blasting and coating equipment, and requires less space for handling and storage. It should be noted, however, that the corners and edges of flat panel specimens are prone to defects that could skew CD test results. The specified pre-screening to detect defects or holidays should identify defects that exist prior to the initiation of testing. Application of additional high impedance coating to all edges could reduce or eliminate the development of edge defects during testing.

Another advantage of the NACE test method is the electrolyte: a sodium chloride solution versus a triple salt solution for the ASTM test method. It has previously been shown that the rate of CD is independent of anion type.⁷ With the effect on disbondment being negligible, a simple sodium chloride solution is easier to prepare and maintain than the triple salt solution specified by ASTM.

High Through-put Test Set-up

Both standards could benefit from more detail in how to run a multi-specimen immersion set-up. The NACE test method should ideally include a dimensioned schematic for each of the test set-up options, including the flat panel and multi-specimen salt bath immersion method. The diagram should include minimum separation distance and location of shunts and rheostats in the circuit. The ASTM test method provides a schematic of a multi-specimen test, but has little written direction for the modification.

Electrolyte Maintenance

Neither standard provides clarity on how to maintain the electrolyte for the test duration, despite the fact that many sources cite the oxygen concentration in the electrolyte as a critical parameter, with higher oxygen concentrations resulting in higher rates of CD.^{7,17} The ASTM test method specifies daily maintenance of the electrolyte depth by addition of potable water, which has the effect of increasing ion concentration as the test proceeds. The NACE test method mentions maintaining the electrolyte volume only for the attached cell method.

During both the first and second rounds of testing for the present study, the electrolyte in some test vessels experienced discoloration. The typical color changes were clear to yellow or brown, and clear to dark grey or black. These color changes are likely due to formation of corrosion products or salt precipitants, and neither the test method, specimen geometry, nor coating type appeared to influence whether or not the color of the solution would change. Attempts to prevent the electrolyte color change were unsuccessful. For the second round of testing, the electrolyte was filled daily and changed weekly. This served to temporarily return the solution to a clear color and allow for easier visual monitoring of the holidays and anode in each test vessel, as well as lower the pH towards neutral, but these effects only lasted at maximum 48 hours before the electrolyte resumed its previously cloudy

state and more basic pH. The interruption in current due to the electrolyte exchange also caused a marked fluctuation in both the applied and polarized potentials. This issue warrants more study in order to find a balance between limiting alternative degradation mechanisms due to corrosion or reaction products in the electrolyte (e.g. hypochlorite degradation of the coating) and maintaining a consistent polarization on the sample.

Anode Isolation

The isolation of the anode is a significant difference between the two standards. Anode isolation should prevent the formation of hypochlorite from the reaction of chlorine gas and hydroxide ions, as previously discussed. This occurs because of the proximity of the anode to the cathode in the laboratory test methods, and can be detrimental to some coatings.^{17,22} The NACE test method requires the anode to be isolated unless otherwise specified. The isolation scheme is not prescribed, but it suggests a glass tube with a glass wool plug. The anode isolation also seemed to provide a secondary benefit of reducing the sensitivity of the polarized potential to changes in the applied potential. Without the glass tube, small changes to applied potential from the power supply in the ASTM set-up often resulted in large fluctuations in the measured polarized potential.

Polarized Potential Measurement

The terms “applied potential” and “polarized potential” require intentional definition and use. The NACE test method appears to interchangeably employ both “applied potential” and “cathodic potential” to mean the negative potential measured between the specimen and a reference cell. The NACE test method also does not require measurement of the polarized potential.

As presented in Table 3, the polarized potential was on average 300-400 mV_{CSE} more positive than the applied potential. This must be noted when assuming a uniform acceleration of cathodic disbondment rate between test vessels and coating types. As this is a comparative test method, it relies on all coating systems being subjected to similar accelerated conditions. This data suggests that, despite a uniform applied potential, the ohmic drop in the test cells was enough to significantly affect the polarization of the specimen. It is worth exploring this further and either minimizing the ohmic drop within the test cell or adjusting the method to specify a polarized potential.

CONCLUSIONS

- The NACE TM0115 and ASTM G8 test methods did not yield a significant difference in measured disbondment for materials evaluated in this study.
- Both test methods resulted in variation of the measured disbondment values for a given coating system by specimen geometry. This emphasized the need to use cathodic disbondment test methods for ranking purposes only, but begs for further investigation into the causes of these observations.
- Both test methods produced polarized potentials 300-400 mV_{CSE} more positive than the applied potentials. The NACE test method averaged a more negative and less variable polarized potential over the test duration.
- The ASTM test method resulted in a test vessel pH that is 1–2 units lower than the NACE test method. The cause of the lower pH may be attributed to the non-isolated anode specified for this standard.
- NACE TM0115 is a streamlined standard for cathodic disbondment that has advantages over ASTM G8 in allowing a variety of specimen geometries, requiring multiple test specimens per data set, using a single analyte sodium chloride solution, and requiring isolation of the anode.
- Recommended improvements to NACE TM0115 are 1) definition of the terms used for polarization, 2) clarification of electrolyte maintenance procedures, and 3) more detailed schematics of the test set-ups.

REFERENCES

1. C.G. Munger, L.D. Vincent, *Corrosion Prevention by Protective Coatings*, 2nd ed. (Houston, TX: NACE, 1999).
2. F. Mahdavi, M. Forsyth, M.Y.J. Tan, "Understanding the Effects of Applied Cathodic Protection Potential and Environmental Conditions on the Rate of Cathodic Disbondment of Coatings by Means of Local Electrochemical Measurements on a Multi-Electrode Array," *Progress in Organic Coatings* 103, Supplement C (2017): p. 83.
3. H. Leidheiser, W. Wang, L. Igetoft, "The Mechanism for the Cathodic Delamination of Organic Coatings from a Metal Surface," *Progress in Organic Coatings* 11, 1 (1983): p. 19.
4. H. Leidheiser, Jr., P.D. Deck, "Chemistry of the Metal-Polymer Interfacial Region," *Science* 241, 4870 (1988): p. 1176.
5. A. Leng, H. Streckel, K. Hofmann, M. Stratmann, "The Delamination of Polymeric Coatings from Steel Part 3: Effect of the Oxygen Partial Pressure on the Delamination Reaction and Current Distribution at the Metal/Polymer Interface," *Corrosion Science* 41, 3 (1998): p. 599.
6. M. Kendig, D.J. Mills, "An Historical Perspective on the Corrosion Protection by Paints," *Progress in Organic Coatings* 102, Part A, (2017): p. 53.
7. O.O. Knudsen, A. Forsgren, *Corrosion Control through Organic Coatings*, (Boca Raton, FL: CRC Press, 2017).
8. *Corr-Wiki Glossary*, <https://www.nace.org/StarterApps/Wiki/Wiki.aspx?wiki=141>, (Houston, TX: NACE International, accessed November 2017).
9. P.A. Sørensen, K. Dam-Johansen, C.E. Weinell, S. Kiil, "Cathodic Delamination of Seawater-Immersed Anticorrosive Coatings: Mapping of Parameters Affecting the Rate," *Progress in Organic Coatings* 68, 4 (2010): p. 283.
10. H. Bi, J. Sykes, "An Investigation of Cathodic Oxygen Reduction beneath an Intact Organic Coating on Mild Steel and Its Relevance to Cathodic Disbonding," *Progress in Organic Coatings* 87, Supplement C (2015): p. 83.
11. F. Mahdavi, M. Forsyth, M.Y.J. Tan, "Techniques for Testing and Monitoring the Cathodic Disbondment of Organic Coatings: An Overview of Major Obstacles and Innovations," *Progress in Organic Coatings* 105, (2017): p. 163.
12. H. Bi, J. Sykes, "Cathodic Delamination of Unpigmented and Pigmented Epoxy Coatings from Mild Steel," *Progress in Organic Coatings* 90, Supplement C (2016): p. 114.
13. S. Shreepathi, "Physicochemical Parameters Influencing the Testing of Cathodic Delamination Resistance of High Build Pigmented Epoxy Coating," *Progress in Organic Coatings* 90, Supplement C (2016): p. 438.
14. R.E. Rodriguez, B.L. Trautman, J.H. Payer, "Influencing Factors in Cathodic Disbondment of Fusion Bonded Epoxy Coatings," CORROSION 2000, paper no. 00166 (Houston, TX: NACE, 2000).
15. K. Cameron, D. Wong, J. Holub, "Practical Analysis of Cathodic Disbondment Test Methods," CORROSION 2005, paper no. 05029 (Houston, TX: NACE, 2005).

16. J. Holub, D. Wong, M. Tan, "Analysis of Cdt Methods and Factors Affecting Cathodic Disbondment," CORROSION 2007, paper no. 07022 (Houston, TX: NACE, 2007).
17. B.T.A. Chang, O.O. Knudsen, D. Wong, J. Holub, A. Al-Borno, J.A. Kehr, "Critical Evaluation of International Cathodic Disbondment Test Methods," presented at 17th International Corrosion Congress, Las Vegas, NV, <http://www.chartercoating.com/library.php> (accessed September 2017).
18. NACE TM0115 (2015), "Cathodic Disbondment Test for Coated Steel Structures under Cathodic Protection" (Houston, TX: NACE).
19. ASTM G8 (2010), "Cathodic Disbonding of Pipeline Coatings" (West Conshohocken, PA: ASTM International).
20. ASTM G95 (2013), "Cathodic Disbondment Test of Pipeline Coatings (Attached Cell Method)" (West Conshohocken, PA: ASTM International).
21. SSPC SP 5/NACE No. 1 (2006), "White Metal Blast Cleaning" (Houston, TX: NACE and SSPC: The Society for Protective Coatings).
22. A. Al-Borno, M. Brown, S. Rao, "The Effect of Anode Isolation Methods on Coating Cathodic Disbondment," CORROSION 2008, paper no. 08007 (Houston, TX: NACE, 2008).
23. O.O. Knudsen, J.I. Skar, "Cathodic Disbonding of Epoxy Coatings - Effect of Test Parameters," CORROSION 2008, paper no. 08005 (Houston, TX: NACE, 2008).

Appendix B – NACE C2019-13367

Impact of Specimen Geometry on Cathodic Disbondment Testing for Protective Coatings

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ABSTRACT

Corrosion protection is necessary to preserve the expected service lifetime of metallic structures. The most effective method of corrosion protection for buried or immersion service is a combination of protective coatings and cathodic protection. However, when applied improperly, cathodic protection may result in damage to the protective coating. Cathodic disbondment testing evaluates a coating's resistance to disbondment in the presence of cathodic protection. Researchers previously compared cathodic disbondment test methods NACE⁽¹⁾ TM0115 and ASTM⁽²⁾ G8, showing that cathodic disbondment varied based on specimen geometry, contrary to previous findings that claimed independence. Researchers designed a subsequent NACE TM0115 experiment using two coating systems and five specimen geometries to further elucidate the results of the prior study. One test set-up evaluated 2-in, 3-in, and 4-in diameter tubes as well as 4-in by 6-in flat panels in immersion, while a second applied the attached cell method. The results provide insight for how to create a more robust test method that aids in the selection of coatings systems for use in conjunction with cathodic protection.

Key words: cathodic disbondment, protective coatings, cathodic protection

INTRODUCTION

Through proper corrosion protection techniques, the lifetime of metallic structures should meet or exceed their original design life. Therefore, good corrosion protection is necessary to keep rehabilitation and replacement costs at a minimum from an annualized cost perspective.¹ The most effective corrosion protection technique for buried and immersion service is to combine a protective coating with a properly designed and well-maintained cathodic protection system. However, if a coating has a low resistance to the chemical environment created by cathodic protection, or if the cathodic protection is applied improperly, coating damage via cathodic disbondment (CD) may be accelerated at the steel-coating interface.

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⁽²⁾ ASTM International (ASTM), 100 Barr Harbor Dr., West Conshohocken, PA 19428

More than twenty CD laboratory testing standards are available to evaluate coating resistance to CD.^{2,3} The results of these test standards are often used to determine which coatings are best suited for use with cathodic protection in the field. A recent study provided a side-by-side comparison of CD results and ease of implementation for two CD test standards: NACE TM0115 and ASTM G8.^{4,5} The results for measured CD were consistent between standards; however, the specimen geometry produced a varying effect on these CD values. This contradicted previous findings by NACE TEG 349X—the technical exchange group created to evaluate international CD test methods during the development of NACE TM0115. The group found CD results to be independent of specimen curvature, leading to the inclusion of flat and cylindrical specimens in NACE TM0115. This paper further investigates the impact of specimen geometry on CD, comparing five specimen geometries evaluated via the NACE TM0115 standard.

The previous study also found that polarized potentials, the potential across the structure/electrolyte interface, ranged from 300 to 400 mV more positive than applied potentials from the external power supply over the duration of the test.⁴ Both test standards mandate a constantly held applied potential for specimens, which does not account for the ohmic drop in the electrolyte. However, in practice, the polarized potential determines the chemical environment produced at the substrate and, in theory, should be a better determinant of coating resistance to CD.

Variation in a specimen's polarized potential, even with a constant applied potential, results in specimens having different levels of polarization during the test period. Because the CD test is comparative, all specimens must be subject to the same test conditions to produce meaningful results. This paper evaluates the impact of polarized potential versus applied potential on CD results, specifically regarding each specimen geometry.

EXPERIMENTAL PROCEDURE

Sample Preparation

NACE TM0115 Standard Test Method was applied to a test matrix of two coating systems and five specimen geometries. Mild steel tubes of three selected diameters were used as tube-shaped specimens and mild steel plates were used as flat panel specimens. The tube-shaped specimens had a length of 12 in (300 mm) and a nominal outer diameter of 2 in (50 mm), 3 in (80 mm), or 4 in (100 mm). Flat panel specimens tested in immersion had dimensions of 4 in (100 mm) x 6 in (150 mm) x 0.125 in (3 mm). Flat panels tested using the attached cell method had dimensions of 4 in (100 mm) x 4 in (100 mm) x 0.25 in (6 mm).

Specimens received either a zinc-rich vinyl primer followed by vinyl topcoats (United States Army Corps of Engineers System 5-E-Z) or a commercial industrial maintenance epoxy. To prepare for coating, specimens were degreased, solvent cleaned, and abrasive blasted to an SSPC-SP5 white metal finish.⁶ The resulting surface profile averaged 3.4 mils (86 μm) for all specimens; the manufacturer recommendations are 2–3 mils (51–76 μm) for the epoxy and 1.5–2.5 mils (38–64 μm) for the zinc-rich vinyl coating. The abrasive blasting set-up used contributed to poor compliance with the manufacturer recommendations.

Wet film thickness measurements were taken during the coating process to ensure that manufacturer recommendations were being met. Dry film thickness (DFT) measurements were recorded after the film had dried or cured. Five DFT measurements were taken for each replicate; Table 1 reports the average and standard deviation of all measurements on all eight replicates for each geometry. The recommended total DFT for the epoxy coating system is 15–30 mils (380–760 μm), and the recommended DFT for the zinc-rich vinyl coating system is a minimum of 7 mils (180 μm).

Table 1
Coating Dry Film Thickness Values

Coating System	Specimen Geometry (Replicates)	DFT in mils (μm)	Standard Deviation in mils (μm)
Epoxy	Attached cell flat panels (8)	22.8 (579)	3.85 (97.8)
	Immersion flat panels (8)	20.9 (531)	4.44 (113)
	2-in tubes (8)	19.9 (505)	3.66 (93.0)
	3-in tubes (8)	20.3 (516)	3.03 (77.0)
	4-in tubes (8)	22.8 (579)	2.69 (68.3)
Vinyl	Attached cell flat panels (8)	9.4 (239)	0.88 (22.4)
	Immersion flat panels (8)	10.0 (254)	1.01 (25.7)
	2-in tubes (8)	8.7 (221)	1.17 (29.7)
	3-in tubes (8)	9.0 (229)	1.14 (29.0)
	4-in tubes (8)	10.7 (272)	1.89 (48.0)

After 14 days, all specimens received holiday detection testing in accordance with NACE SP0188-2006 at 90 V.⁷ No specimens failed this evaluation, and therefore, all could be used for testing.

A small area of each specimen was ground to bare steel above the immersion line using a rotary tool. A drill was used to produce a hole through this bare steel surface and a bolt fastened as the structure-to-power supply connection.

Each specimen also received a single 0.25-in (6-mm) diameter holiday drilled with a flat head end mill bit. This holiday was drilled at the center of the attached cell flat panel specimens, and in the center of the immersed area of the immersion flat panel specimens. The holiday location of the tube-shaped specimens is at a height halfway between the top and the bottom of the immersed section.

Experiment

The CD testing proceeded in two rounds, each with a duration of 28 days per NACE TM0115. The test utilized eight immersion test vessels and eight attached cell vessels, for a total of 16 vessels per round. To attain a similar multi-specimen set-up for all test vessels, all immersion vessels contained four specimens, each of the same coating system. Specimen spacing and orientation were maintained during the test period to ensure that all test vessels adhered to the minimum spacing requirements of the test methodology. This was achieved using custom-milled acrylic sheets suspended with insulated wire hooks for tube-shaped specimens and using an insulated aluminum frame above the water line for flat panel specimens.

The 2-in tube-shaped specimens and immersed flat panels were tested in 5-gallon (19-L) vessels (Figures 1(a) and 1(b)). The 3-in and 4-in tube-shaped specimens were tested in 32-gallon (120-L) vessels (Figure 1(c)). Attached cell vessels were constructed using clear, 3-in (80-mm) inner diameter polyvinyl chloride plastic (PVC) tubes adhered to each specimen with a silicone adhesive (Figure 1(d)).

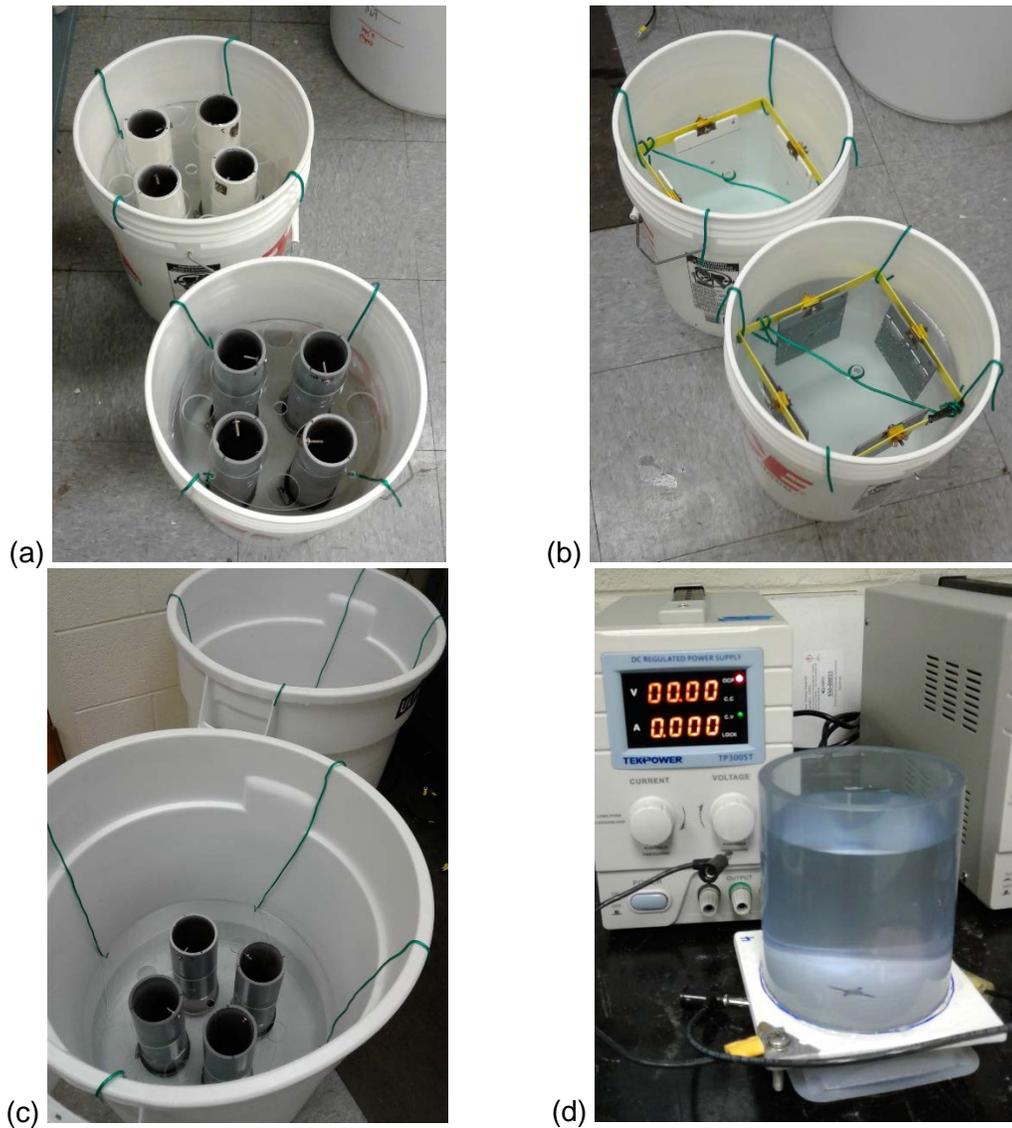


Figure 1. Multi-specimen test set-up for (a) 2-in tube, (b) flat panel, (c) 3-in and 4-in tube, and (d) attached cell specimens.

The vessels received an electrolyte solution of deionized water and 3% sodium chloride by mass. The electrolyte was filled to a prescribed volume in each vessel that provided a consistent immersed surface area for all immersed specimen geometries. For attached cell vessels, electrolyte volume was maintained at a fixed volume greater than 300 mL to meet the requirements of the test method. Deionized water was used to top-off each test vessel as needed to maintain the correct volume throughout the test duration.

The investigation utilized a 0.125-in (3-mm) diameter, platinum-clad copper core anode to be consistent with the test set-up from the recent study.⁴ All anodes were isolated in a glass tube that was plugged with glass wool at the bottom and left open at the top to allow the escape of chlorine gas produced during the test.⁸ The test specimens were connected to the power supply using 0.1-ohm shunts, consistent with the previous study but deviating from NACE TM0115 which requires 1.0-ohm precision resistors for measuring cathodic current.

The test used an applied potential of -1.38 ± 0.02 V vs silver-silver chloride (saturated KCl) reference electrode (V_{SSC}) throughout the experiment. Test monitoring occurred no less than four days each week

with data recorded for electrolyte pH and temperature, applied voltage, applied current, applied potential, and polarized potential (via instant-off technique). Following data collection on day 28 of the testing, specimens were removed from the test vessels, rinsed, dried, and disbonded area was measured and recorded per NACE TM0115.

RESULTS

Cathodic Disbondment

Measurement of disbonded area occurred at the completion of each round of testing; the results combine both rounds of testing into one dataset for evaluation. Figure 2 provides the average disbonded area and standard deviation for each specimen geometry and coating system. The epoxy-coated specimens produced a consistent disbonded area for each geometry; the attached cell was the lowest average at 10.1 mm and the 3-in tube was the highest at 12.3 mm. The epoxy specimens also had consistent reproducibility for each geometry with an average standard deviation of 0.7 to 2.2 mm.

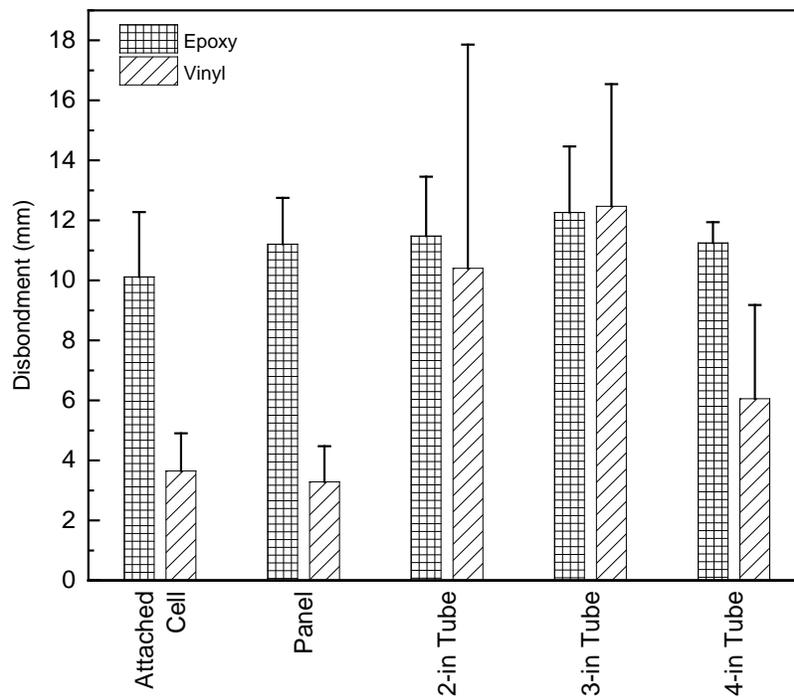


Figure 2. Disbonded area for each specimen geometry and coating system.

The vinyl-coated specimens ranged from an average disbondment of 3.3 mm (attached cell) to 12.5 mm (3-in tube). The attached cell and panel had the lowest disbonded areas and correspondingly low standard deviations. The 4-in tube had intermediate results with an average disbonded area of 6.1 mm and a standard deviation of 3.1 mm. The 2-in tube had the poorest reproducibility with disparate disbonded area results ranging from 2 mm to 21 mm. The 3-in tube results were marginally more consistent with an average of 12.5 mm and results ranging from 3 mm to 16 mm.

Figure 3 shows the specimen geometries with the smallest and largest disbonded area for each coating system in the first round of testing. The attached cell geometry produced the smallest disbonded area for each coating type and geometry combination and a tube specimen produced the largest.

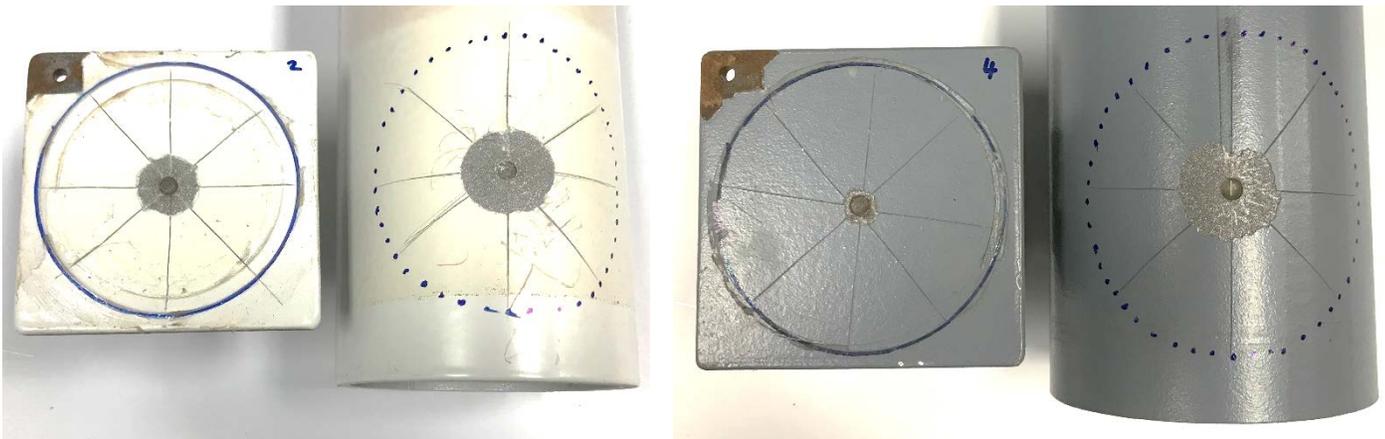


Figure 3. Comparison of the specimen geometries with the largest and smallest disbondment for (left) epoxy coating on attached cell panel and 4-in tube and (right) vinyl coating on attached cell panel and 3-in tube. For all specimens, the circles outline a 4-in (100-mm) diameter region.

Potential

The applied potential for each specimen geometry was adjusted daily to $-1.38 \pm 0.02 V_{SSC}$, as specified by the NACE test method. Figure 4 illustrates resulting applied and polarized potential raw data for two epoxy-coated and two vinyl-coated specimens. The specimen identifier designates testing round (R1 or R2), coating system (E for epoxy or V for vinyl), geometry (AC for attached cell or 3P for 3-in tube), and specimen number.

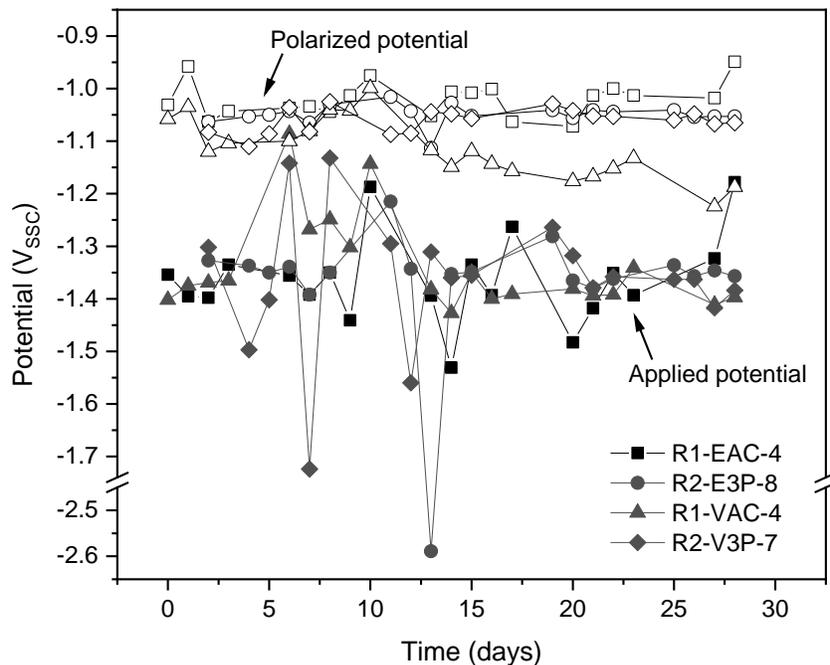


Figure 4. Comparison of applied and polarized potential measurements over the duration of the test for specimens with the highest and lowest potential values from each coating system.

The polarized potential in Figure 4 is approximately 300 mV less negative than the applied potential. However, the specified $-1.38 V_{SSC}$ applied potential is approximately met, albeit with notable variability.

Both “R2” specimens shown experienced spikes in the applied potential, although no corresponding spike in the polarized potential occurred. The polarized potential has greater stability than the applied potential, but the potential can drift, such as for R1-VAC-4.

Figure 5 shows the average applied and polarized potentials for each dataset and includes a dashed line at the $-1.38 V_{SSC}$ target applied potential. The average applied potentials (lower bound of each bar) were up to 100 mV less negative ($-1.28 V_{SSC}$) than required for the epoxy-coated specimens, except for the immersed flat panel which met the required $-1.38 V_{SSC}$. The vinyl-coated specimens had a consistent average applied potential of approximately 50 mV less negative ($-1.33 V_{SSC}$) than required. Furthermore, the vinyl-coated specimen standard deviations are less than the epoxy-coated specimens.

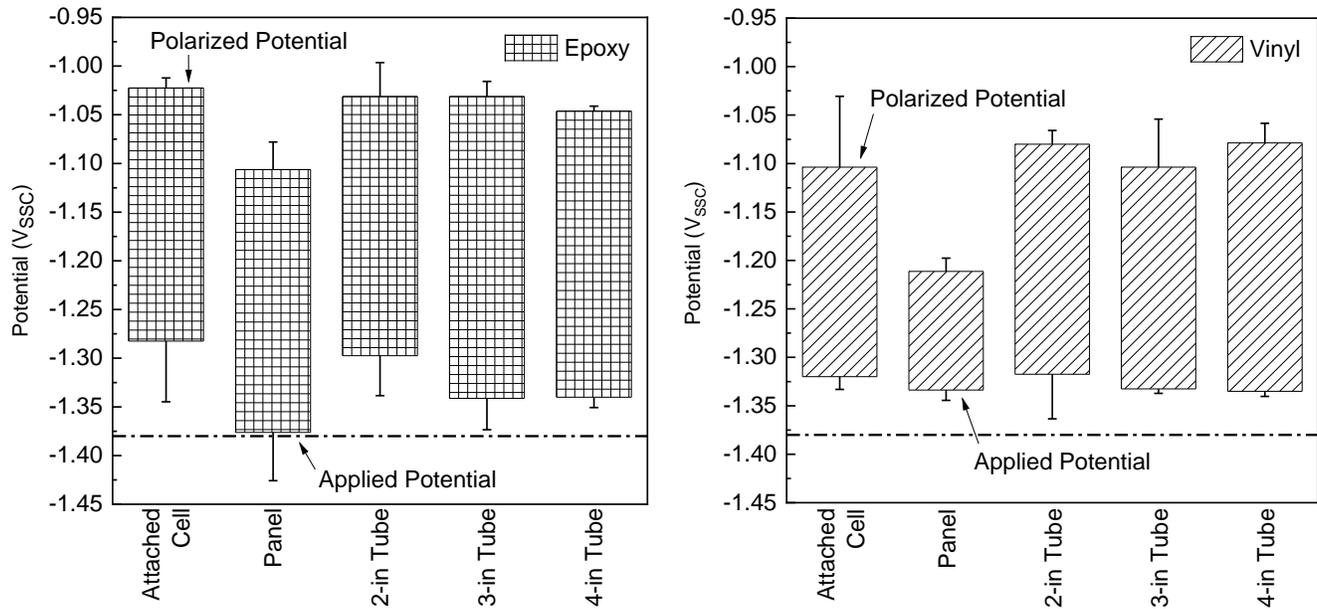


Figure 5. Comparison of average applied and polarized potential measurements for each specimen geometry for (left) epoxy and (right) vinyl.

The average polarized potential (Figure 5, upper bound of each bar) for the epoxy-coated specimens was approximately $-1.03 V_{SSC}$ with the flat panel again being an outlier at $-1.11 V_{SSC}$. The vinyl-coated specimens had an average polarized potential near $-1.10 V_{SSC}$, except for the flat panel at $-1.22 V_{SSC}$. Overall, the experiment produced approximately 50 mV more polarization for the vinyl-coated specimens than the epoxy-coated specimens. Both coating types had a range of standard deviations associated with the average polarized potential, demonstrating variability in the polarization.

Table 2 presents average applied and polarized potential compared to disbonded area for selected attached cell and 3-in tube replicates. The attached cell specimens consistently measured the lowest average disbonded area, and the 3-in tube specimens measured the highest average disbonded area. The table presents the lowest and highest disbonded area results of these respective geometries for each testing round.

Table 2
Average Applied Potential and Polarized Potential Attached Cell and 3-in Tube Specimens

Specimen	Applied Potential (V _{ssc})	Polarized Potential (V _{ssc})	Cathodic Disbondment (mm)
R1-EAC-4	-1.32±0.21	-1.03±0.04	7.45±0.59
R2-EAC-9	-1.30±0.17	-1.03±0.02	10.90±0.81
R1-E3P-4	-1.33±0.25	-1.03±0.03	10.65±0.67
R2-E3P-8	-1.40±0.28	-1.03±0.10	13.90±0.28
R1-VAC-4	-1.30±0.21	-1.02±0.45	2.50±0.11
R2-VAC-9	-1.31±0.23	-1.06±0.03	3.93±0.59
R1-V3P-4	-1.34±0.23	-1.15±0.06	14.60±0.41
R2-V3P-7	-1.33±0.21	-1.06±0.02	16.00±0.57

The experiment's average potential values were compared to the resulting disbonded area for all 40 specimens via calculation of the correlation coefficients to determine if a relationship exists. Analysis of the applied potential versus the disbonded area resulted in a correlation coefficient of 0.23 for epoxy and 0.01 for vinyl. The epoxy presents a possible weak relationship, suggesting that the disbonded area increases as the applied potential increases. The polarized potential versus disbonded area resulted in a correlation coefficient of 0.16 for epoxy and -0.34 for vinyl. The epoxy result is low enough to suggest no relationship. However, the vinyl result suggests a moderate negative relationship—the disbonded area decreases as polarized potential increases. The cause of this negative relationship is not readily understood and could be explored further.

pH

Average pH was consistent across all geometries and coating systems with an average standard deviation of 0.3 (see Figure 6). The epoxy-coated specimens resulted in a slightly higher pH than the vinyl-coated specimens for the attached cell specimens; the other specimen geometries have high standard deviations. The attached cell had the lowest volume of electrolyte, while the 3-in tube and 4-in tube had the highest vessel volume. The pH values show a possible inverse relationship with vessel volume, in which less dilution of the electrochemical reaction products at the cathode occurs, notably hydroxyl ions. The measured pH values were consistent with previous research.⁴

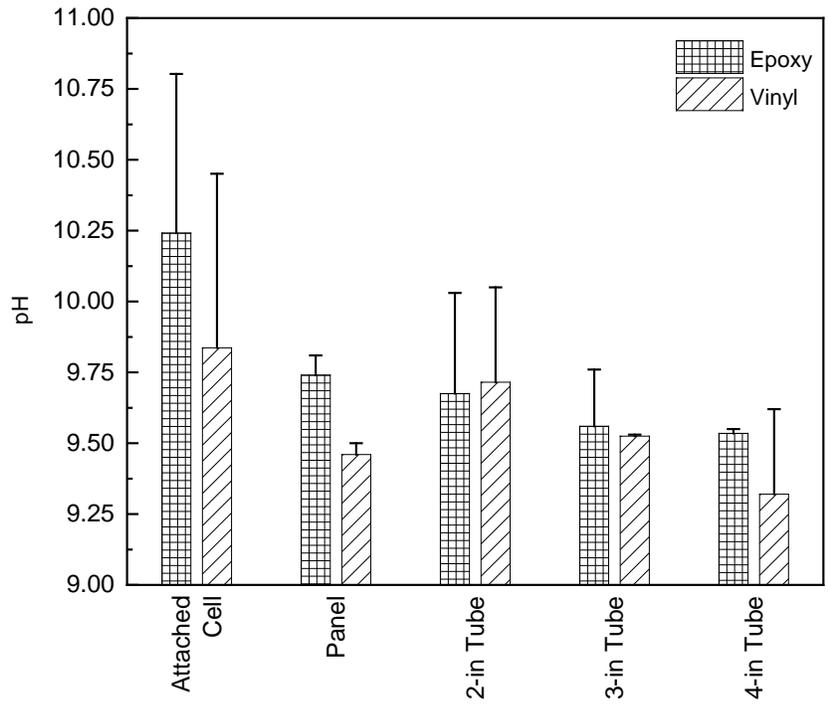


Figure 6. Average and standard deviation of pH measurements for each specimen geometry and coating system.

DFT

Figure 7 gives the average change in DFT for each specimen geometry and coating type from before the testing began to after test conclusion. The average change in DFT for all specimens was negative (a reduction in DFT) except for the epoxy-coated attached cell and epoxy-coated flat panel specimens which increased by 4.6% and 10.8%, respectively. The 2-in tube specimens for both coating systems had the greatest average decrease in DFT at 12.2% for the epoxy coating and 9.2% for the vinyl coating. The vinyl-coated 4-in tube had the smallest average decrease of the vinyl-coated geometries. All specimens produced large standard deviations.

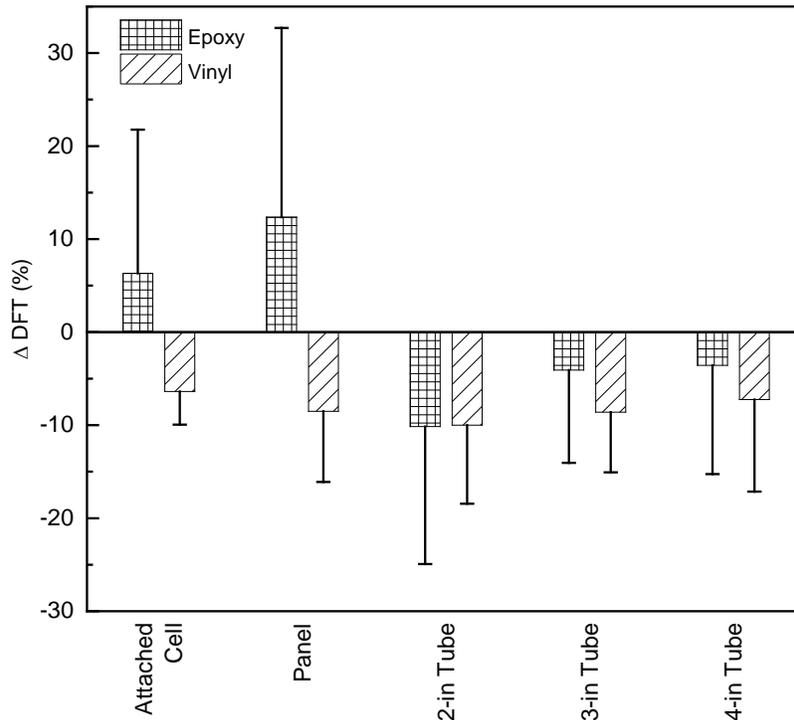


Figure 7. Average change in coating DFT change during the test for each specimen geometry and coating system.

DISCUSSION

This study evaluated two different coatings systems to provide a more thorough study of the impacts of specimen geometry on cathodic disbondment test results. These two coating systems are the same as those used in a previous test method comparison study presented at NACE CORROSION 2018 to allow for comparison of results.⁴

Effect of Geometry

The results showed that the specimen geometry can have a significant effect on the degree of cathodic disbondment in certain coating systems, but this effect is varied by coating type. Geometry had little effect on the results for the epoxy-coated specimens, but significant impact on the results for the vinyl-coated specimens. The vinyl-coated 2-in tube and 3-in tube produced the largest disbonded area and both had high standard deviations, indicating poor test reproducibility for those geometries. The flat panel and attached cell produced the smallest disbonded area. The 4-in tube had intermediate results. The difference between the smallest and largest average disbonded areas is an increase of 280%.

It should be noted that the vinyl coating system included a zinc-rich primer. Zinc is a more electronegative metal than steel; it will act to polarize the steel at defects in the coating. This could affect the polarized potential at the coating defect during the cathodic disbondment test and play a role in the high variability of the results for the vinyl-coated specimens, however further investigation would be needed to determine the mechanisms at play. The effect of poor test reproducibility increased as pipe curvature increased.

In addition to variability based on coating system, the attached cell method, one of the most widely-used geometries, generated less disbonded area compared to the other specimen geometries. The effect was marginal for the epoxy-coated system and pronounced for the vinyl-coated system. NACE TM0115 does not specify a testing geometry in its method, however these results show that data comparison across

different specimen geometries may not be valid. Care should be taken when reporting and interpreting data to note the specimen geometry, and a robust test method within a lab should consider consistency in specimen geometry important when selecting coatings systems for use in conjunction with cathodic protection. Future research could evaluate other coating systems for the effects observed and whether other factors, such as cathodic shielding, inclusion of metallic pigments, or different applied current densities, impact NACE TM0115 outcomes.

Applied vs Polarized Potential

The average applied potentials were approximately 100 mV less negative than required for the epoxy-coated specimens and approximately 50 mV less negative for the vinyl-coated specimens. The epoxy-coated flat panel was an exception and met the required $-1.38 V_{SSC}$. The cause for the difficulty in maintaining the specified applied potential is unknown. The metallic zinc pigments within the vinyl-coated specimens may be providing a stabilizing, mixed potential effect or impacting the conduction of the cathodic protection current.

The average polarized potential for the experiment was approximately $-1.02 V_{SSC}$ for the epoxy-coated specimens and $-1.10 V_{SSC}$ for the vinyl-coated specimens. The immersed flat panel was an exception and provided a significantly more negative polarized potential for both coating systems; the average was 100 mV more negative for the vinyl-coated flat panel. Although the vinyl-coated specimens received greater polarization during the experiment, the variability in polarization was similar for both.

As previously observed, the polarized potential was consistently 300mV less negative than the applied potential.⁴ While the applied potential showed several unexplained spikes in the data, the polarized potentials remained consistent. Even for outlier specimens (Figure 4), no significant trend can be elucidated between the polarized potential and the measured disbonded area. In fact, the specimen set with the highest polarization, the vinyl-coated flat panel, produced the lowest average disbonded area; this is counterintuitive as one would think higher polarization would lead to more disbondment. Although the polarized potential had minimal correlation to disbonded area in this study, it would likely be a better value to benchmark in a standard for consistency purposes than the applied potential.

While NACE TM0115 is a test method for “determining comparative resistance to cathodic disbondment of protective coating systems,” the protective coating systems subjected to this test may be intended for use in conjunction with cathodic protection systems in service. Common practice for buried and immersed structures is a maximum polarized potential of approximately $-1.1 V_{SSC}$ in routine cases to reduce cathodic disbondment. An area for future study would be to maintain the maximum recommended polarized potential and compare coatings types to determine how they would behave under cathodic protection.

CONCLUSIONS

- The experiment evaluated two coating systems via NACE TM0115 using five specimen geometries: attached cell, immersed flat panel, and tubes with a nominal outer diameter of 2 in (50 mm), 3 in (80 mm), or 4 in (100 mm).
- Specimen geometry did not influence the quantity and reproducibility of disbonded area for the epoxy-coated system.
- The vinyl-coated system had increased disbonded area and decreased experiment reproducibility as the degree of specimen curvature increased, i.e., comparing the flat surface of the attached cell and panel to each tube. The investigation did not reveal the reason for the difference.
- The results suggest that data cross-comparison may not be appropriate when different geometries are used for testing.
- The polarized potential was consistently 300mV less negative than the applied potential. Comparison of the polarized potential to the disbonded area showed no correlation.

REFERENCES

- 1 B.J. Merten, et al., "Developing a Life-Cycle Cost Analysis Framework to Evaluate the Cost-Effectiveness of Hydroelectric Penstock Corrosion Control Strategies," CORROSION 2017, paper no. 9099 (Houston, TX: NACE, 2017).
- 2 B.T.A. Chang, et al., "Critical Evaluation of International Cathodic Disbondment Test Methods," presented at 17th International Corrosion Congress, Las Vegas, NV, <http://www.chartercoating.com/library.php> (accessed December 2018).
- 3 F. Mahdavi, et al., "Techniques for Testing and Monitoring the Cathodic Disbondment of Organic Coatings: An Overview of Major Obstacles and Innovations," Progress in Organic Coatings 105, (2017): p. 163.
- 4 G. Weber, et al., "Comparison of Cathodic Disbondment Test Methods for Water Infrastructure Coatings," CORROSION 2018, paper no. 10914 (Houston, TX: NACE, 2018).
- 5 NACE TM0115 " (2015), "Cathodic Disbondment Test for Coated Steel Structures under Cathodic Protection"" (Houston, TX: NACE).
- 6 SSPC SP 5/NACE No. 1 (2006), "White Metal Blast Cleaning" (Houston, TX: NACE and SSPC: The Society for Protective Coatings).
- 7 NACE SP0188 (2006), "Discontinuity (Holiday) Testing of New Protective Coatings on Conductive Substrates" (Houston, TX: NACE).
- 8 A. Al-Borno, et al., "The Effect of Anode Isolation Methods on Coating Cathodic Disbondment," CORROSION 2008, paper no. 08007 (Houston, TX: NACE, 2008).

Data Sets that Support the Final Report

If there are any data sets with your research, please note:

- \\bor\do\Team\ENGRLAB\MERL\Corrosion\Cathodic Disbondment Testing
- T:\Jobs\DO_NonFeature\Science and Technology\2018-PRG-Impact of Specimen Geometry and Polarized Potential on Cathodic Disbondment Testing for Protective Coatings
- Folders include all associated data, photographs, reports, and presentation
- Keywords: cathodic disbondment, protective coatings, cathodic protection
- Point of contact: Grace Weber, gweber@usbr.gov, 303-445-2327
- Approximate total size of all files: 1.48 GB, 510 MB