

High Temperature Cathodic Disbondment Testing for Pipeline Coatings

Shiwei William Guan
Bredero Shaw
101 Thomson Road, #17-01 United Square
Singapore 307591
Email: sguan@brederoshaw.com

Alan Kehr
Alan Kehr Anti-Corrosion, LLC (AKAC)
2303 RR 620 S, Suite 135, #235 Lakeway
TX 78734, U.S.A.
Email: alankehr@gmail.com

ABSTRACT

Cathodic Disbondment (CD) testing has historically been performed on protective coatings to assess coating delamination resistance when exposed to cathodic polarization. Unfortunately, there is no broadly accepted high temperature CD testing standard in the pipeline industry. There are disparate reference standards, testing durations, reference electrode types, electrolyte temperatures and topping up/replacing frequency, testing potentials, sample testing temperature, means of assessment, and non-standard test procedures that characterize the varying practices currently used in the industry. This paper has three segments: (1) Reviews factors that affect the high temperature CD test behavior of fusion bonded epoxy (FBE) based pipeline coatings – some are critical. (2) Reports the results of a global survey on actual CD test practices used by laboratories – the survey shows a wider variety of practices. (3) A test program reports the results of tests from a pipeline project, illustrating the effects of specific test methods and test variables on results and reproducibility. The review, the survey, and the test results highlight the need to develop a definitive high temperature CD test procedure that is stable, understood, easy to run, and provides reproducible results.

Key words: Cathodic disbondment, high temperature, pipeline coating, Fusion bonded epoxy, Test method

INTRODUCTION

Cathodic Disbondment (CD) testing has historically been performed on protective coatings in order to assess the resistance to coating delamination from exposure to cathodic polarization. As explorations & production for oil and gas reservoirs go deeper and fluid temperatures get higher, there are new demands for high temperature CD performance of a pipeline coating system and therefore on suitable high temperature CD test methods. Unfortunately, a common standard CD test method well accepted by the industry does not exist and well-accepted procedures for evaluating CD behavior of a pipeline coating for higher operating temperatures do not exist at this time.

Adding to the complexity is the fact that pipe coating materials/products for high temperature applications, such as high temperature FBE powders, are relatively new to the industry. For example, the great majority of conventional FBE powders in the market developed prior to the year of 2000 have a glass transition temperature (T_g) around 100°C. Major pipeline owners specify the maximum operating temperature for conventional standalone FBE pipeline coatings as 60°C. The commonly recommended maximum operating temperature is 85°C and 110°C for a standard three-layer polyethylene (3LPE) system and for a standard three-layer polypropylene (3LPP) system, respectively. Only recently have coating manufacturers developed high T_g FBE coatings, which are also recommended as a primer for multilayered PP (polypropylene) systems for pipelines operated at temperatures >110°C. The pipeline industry is currently discussing relevant standards and testing techniques to qualify high temperature FBE and polypropylene anti-corrosion and/or thermal insulation coating products for operating temperatures of 150°C or higher. The procedures are to verify the performance of these coating products over the design life of the pipeline and for use as a quality control (QC) tool during coating production. However, these high-T_g FBE coating materials often do not have a long track record of field performance. Also, there is insufficient public data available to determine the best high temperature CD testing procedures or the best way to interpret the high temperature CD testing results and suitable pass/fail criteria.

This paper reviews various critical factors that affect the high temperature CD behavior of a FBE based pipeline coating. Results of an industry global survey are reported, revealing the varying practices currently used in the industry for CD testing which can be characterized by broadly disparate reference standards, testing durations, reference electrode types and uses, electrolyte temperatures and topping up/replacing frequency, testing potentials, sample testing temperature, and means of assessment. Results of a real offshore pipeline project are presented, illustrating the effect of some of these critical factors on the CD testing results of a high temperature pipe coating as a result of adopting two different standard test methods. The data highlights the need for the industry to develop a definitive high temperature CD testing procedure that is stable, understood, easy to run, and provides reproducible results.

REVIEW: FACTORS AFFECTING HIGH TEMPERATURE CATHODIC DISBONDMENT

Factors affecting CD behavior of a coating have been extensively studied and reported, reflecting in many critical reviews conducted by NACE International (NACE)^① Technology Exchange Group TEG349X on existing international standard CD test methods.¹⁻³ These reported factors include: hypochlorite formation (anode isolation), coating dry film thickness (DFT), size and shape of the artificial holiday, testing temperature and electrolyte temperature, oxygen concentration in the electrolyte, applied potential, test duration, reference electrode and calibration, pretreatment of the substrate and surface profile.

Unfortunately, despite a good understanding of the above, there is no broadly accepted CD test standard yet established in the pipeline industry, nor for high temperature applications. In an attempt to address this issue, NACE Task Group (TG) 470 is preparing a draft NACE Standard “Cathodic Disbondment Test for Coated Steel Structures Under Cathodic Protection” and International Standards Organization (ISO)^② is in the process of developing a standard test method for cathodic disbondment of coatings >95°C. The drafted documents are the culmination of a tremendous amount of excellent work by the task groups. However, attention to the following points is needed:

- Most of the existing standard CD test methods were originally designed for or started for onshore pipeline applications with service/testing temperatures ≤95°C. Limited CD data is available in public domain for service/testing temperatures of higher than 95°C for pipeline coatings. Attempts that

^① NACE International (NACE), 1440 South Creek Drive Houston, Texas, USA 77084-4906

^② International Organization for Standardization (ISO), 1 ch. de la Voie-Creuse, CP 56, CH-1211 Geneva 20, Switzerland

have been made to further develop existing standard CD test methods and specifications to cover higher temperature systems have generated certain practices and concepts that are no longer valid. Whether existing standards or their modifications might or might not be suitable for the needs of subsea/deepwater pipeline applications needs to be addressed where new, unfamiliar, higher temperature and often much thicker coating systems are in use.

- Even if common standard CD test methods are used, broadly disparate test settings and practices that characterize the pipeline industry are likely to result in inconsistent and non-reproducible CD data – see Table 2. This means that the industry still has extensive work ahead to understand the true mechanism of coating cathodic disbondment. This information is vital to devising detailed methods that insure CD test settings and practices that are consistent and uniform.
- Many existing standard CD test methods may have worked perfectly for small runs in a laboratory setting on lab prepared samples. Due to increasing total length of pipeline projects in today's pipeline industry, a large quantity of QC samples and tests is often required by a pipeline project. That creates the need to develop a definitive and workable CD test method/procedure that is stable, understood, easy to run (with the correct equipment and experience), and provides reproducible results. An example is testing CD at temperatures over 100°C in a high temperature - high pressure autoclave presented by Al-Borno⁴. CD testing in autoclaves involves heating the electrolyte to the same temperature as the sample plates, which is unrealistic when compared to the conditions encountered in the field. The use of a high temperature/high pressure autoclave also brings the complexity of the apparatus and the test, and the difficulty of running CD testing for massive numbers of production samples – different apparatus is required for each sample. These are rather research and development techniques for studying materials than useable QC methods.
- With limited CD data available for higher operating temperature pipeline projects and high temperature coatings, developing procedures/criteria that satisfy the coating applicator's capabilities and the engineering requirements for the pipeline project's owner requires careful considerations. New CD pass/fail criteria for quality controls might need to be established rather than adopting the same from the previous/existing specifications designed for lower operating temperatures.

Some critical factors affecting the high temperature CD behavior of today's FBE based pipeline coatings are discussed as follows:

Hypochlorite effect and anode isolation:

It is generally agreed by the industry that a chemical attack to the coating during CD testing can be caused by formation of hypochlorite or chlorate(I) anions (ClO^-), causing coating deterioration/delamination quite different from the delamination due to cathodic electrochemical reaction (cathodic disbondment).² The rate of hypochlorite attack to the coating is proportional to its concentration and to the electrolyte temperature. The concentration of ClO^- increases with increasing current flow to the test cell. The hypochlorite effect is more significant at higher testing temperatures/electrolyte temperatures. The phenomenon does not occur in the field because the anode and cathode are far apart and do not produce hypochlorite.

It appears that both NACE TG470 and the ISO working group require anode isolation in their newly drafted standard CD test methods as the only means to prevent the anolyte chlorine gases from migrating to the cathodic sites to form hypochlorite. However, the majority of historical CD testing data obtained by the industry over the past several decades was done with no anode isolation. Recent research by Al-Borno⁵ found that the use of anode isolation caused the pH value of CD test environment to be significantly higher than that with no anode isolation even as early as the first 24 hours, but disbondment was also reduced. This obviously contradicted the pH effect revealed from

earlier studies by Rodriguez,⁶ which suggested highly alkaline solutions penetrate further into the crevice formed by a disbonded FBE coating than neutral solutions. This added penetration produces more disbondment by affecting the coating-to-substrate interaction and displacing the coating. Al-Borno⁵ suggested that the greater disbondment with no anode isolation was an indication that the hypochlorite effect was more significant in increasing disbondment than the pH effect. However, it is important to point out that Al-Borno only reported results of CD tests conducted for 72 hours and 28 days. As it takes time to build up hypochlorite, it is possible that the hypochlorite effect might become more significant in increasing disbondment than the pH effect, only when the testing duration is long enough (72 hours and 28 days or longer, depending on electrolyte temperatures) to generate sufficient hypochlorite. For shorter term tests such as 24 or 48 hours, the pH effect appears to be more significant than the hypochlorite effect in determining the disbondment.

The implementation of “anode isolation” in the newly drafted standard CD test methods may result in significantly different disbondment results compared to currently used tests. Introduction of this variable in the test procedure requires at least an evaluation of the resulting data before setting CD acceptance criteria. The industry needs to build a new data pool for the acceptance criteria with anode isolation. J. Holub² suggests altering the procedure to the use of anode isolation, frequent electrolyte refreshment, and proper selection of the electrolyte temperature for a CD test can be effective in avoiding coating delamination due to chemical attack. Again, those changes mean that historical data may not be useful for establishing acceptance values prior to developing a sufficiently large data base.

Specimen geometry and preparation before and after CD test:

Although it was found that CD specimen geometry, either flat, curved steel panel or tube, had no impact on CD results,³ it is recommended for practical reasons to request tube (full ring) specimen only for pipe size <16 inch (406 mm) Ø. Cut panels or quarter ring/half shell should be considered only for specimen from pipe size >20 inch (508 mm) Ø. Also for practical reasons, the pipe ring specimen should be at least 30 cm in length with test area >15 cm from the cut ends.

Conventional onshore FBE/three layer polyolefin (3LPO) pipeline coatings are often only 0.5 to 3 mm thick or less, requiring no special specimen preparation or assessment for CD testing. For onshore horizontal directional drill (HDD) applications or subsea thermal insulation, however, the pipeline coating used can be much thicker: some subsea insulation coatings are hundreds of millimeters thick. A CD test on an unprepared specimen with such a thick coating layer often ends up with meaningless results of very little or zero disbondment. Sometimes making the radial cuts and lifting the disbonded coating with a knife is not possible (Figure 1). Consider specific specimen preparation on the thick coating, e.g. trimming down of the outer layer of a thick insulation coating to 3 mm or less before CD testing rather than testing the thick layer. This facilitates easy assessment of disbonded coating at the end of the test. Some standards/specifications (such as NF A 49-711⁸) assess the CD after heating the 3LPO coating in a furnace to soften the adhesive layer and detaching the top layer (Figure 2). Such a test procedure can be dangerous, since excessive heating to soften the adhesive has the potential of damaging the FBE during the removal process.



Figure 1: A thick 3LPP sample after CD

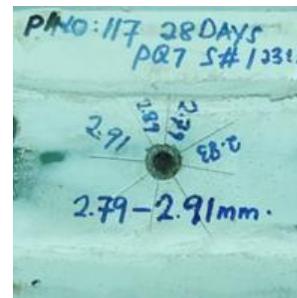


Figure 2: Removing outer layer after CD

FBE Film thickness

FBE film thickness plays an important role in cathodic disbondment resistance: a thicker coating shows less disbondment. This is of practical importance because some specifications request a cathodic disbondment test on the primer alone. FBE, as a primer for three layer coating, has a normal thickness significantly lower than as a standalone coating. Application temperature and quenching effects are often significantly different between multilayer polyolefin coatings and standalone FBE. These factors can result in significantly different results between an FBE layer applied as a primer without the top coat compared to an FBE standalone coating. The acceptance criteria need to be different.

Steel types:

The effect of steel type on performance testing of an FBE based pipeline coating has rarely been studied, since old and existing pipelines usually are low carbon mild steel. The pipeline industry now uses different types of steel such as high strength steel (X80 and up to X120), duplex and stainless steel pipes, and corrosion-resistant alloy (CRA) (mechanically or metallurgical lined) clad pipes. Unfortunately, these special types of steel tend to behave quite differently during the application process of a FBE based coating system, compared with their regular low carbon mild steel grades. For example, grit blasting different types of steel results in different anchor patterns. The anchor pattern can affect CD results.

Another factor is their temperature behavior during the pipe preheating process prior to coating application. Table 1 illustrates a pipe preheating temperature profile prior to FBE application of a submerged arc welded (SAW) duplex steel pipe (408.4mm Ø x 25.0mm WT) after going through induction coils at a line speed of 13.4 fpm. The surface temperatures on the pipe body and along/near the weld-seam area were significantly different with a change in temperature (ΔT) of up to 40°C. The large difference in surface temperatures and less predictable heat profile/pattern along these pipes can have an impact on the performance properties (such as CD and hot water soak) of an FBE based coating on different sections of the same pipe. The root-cause of the problem is that SAW duplex steel pipe has different ferritic-austenitic structures and ferrite contents in the weld seam and on the pipe body. These do not respond uniformly to the electro-magnetic induction heating of the coating process.

Table 1 Heating profiles of a SAW duplex steel pipe during FBE coating process

Location		Surface Temperature, T (°C)
Lead End	On Body	242°C < T < 246°C
	Weld-seam	210°C < T < 225°C
Middle	On Body	241°C < T < 246°C
	Weld-seam	184°C < T < 210°C
Trail End	On Body	239°C < T < 246°C
	Weld-seam	184°C < T < 210°C

Holiday depth:

A typical specification states that an artificial holiday of 6 mm in size, unless specified/agreed otherwise, shall be drilled with a flat head end mill bit and penetrated less than 0.5 mm into the steel. Figures 3 and 4 illustrate the 28 day at -1.5V and 65°C CD test results of two production qualification trial (PQT) FBE samples from the same production conditions. All test parameters were the same except holiday depths. The impact of the holiday depth variations on the CD results was evident only after testing for 28 days. No differences in CD results were found with samples with different depths

after being tested for 48 hours at -3.5V and 65°C. Compared with Figure 3 (average CD=8.28 mm), darker and more scattered rusty marks are seen on the larger disbonded surface of the sample in Figure 4 (average CD=11.94 mm). This suggests that the increased disbonding may be due to an increase in hypochlorite production because more current flows through the system with a deeper holiday. The increase in current flows might also result in much faster OH⁻ build up and thus higher rate of disbondment.



Figure 3: A FBE sample after CD testing for 28 days at -1.5V and 65°C (holiday depth = 0.1 mm)

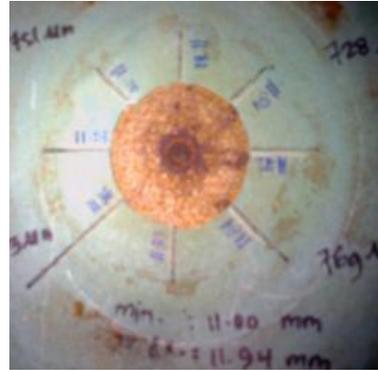


Figure 4: A FBE sample after CD testing for 28 days at -1.5V and 65°C (holiday depth = 1.1 mm)

Test temperature and electrolyte temperature:

Test temperature has the most significant effect on CD results in short-term tests, such as those used as part of in-plant quality control program. It also has a significant effect in long-term CD tests. Temperatures measured by a dial thermometer on the test panel surface (by most CD standards and specifications), or by an immersion thermometer in the electrolyte (by some standards such as NF A 49-711) are different.

Many industry standards, e.g., CSA[®] Z245.20⁹, ASTM[®] G42¹⁰, G95¹¹, and DIN[®] EN-10289¹², specify a test temperature from room temperature up to the maximum operating temperature, but not above 90 °C or 95 °C. The tests were often conducted without electrolyte cooling or electrolyte temperature control, and do not distinguish between using an oven to maintain temperature – where the electrolyte is the same temperature as the test panel – and a hot plate, where the steel temperature is different from the electrolyte temperature. For an offshore pipeline subsea installation carrying hot fluid, this may be an issue: the hot internal fluid and the cold external seawater result in a temperature gradient through the coated steel. A CD test conducted without cooling the electrolyte to the expected seawater temperature does not simulate the offshore/subsea operating condition. On the other hand, high temperature is often used to accelerate the property degradation process, thus reducing the time required for predictive models of the performance of a pipeline coating. The question is the understanding of the temperature effect with hot electrolyte. The effects are not understood well enough to establish a universal acceptance criterion.

In designing a CD qualification test for a pipeline coating for a specific project application, the electrolyte temperature must be controlled. One idea is to ‘simulate’ the actual/specific external service condition/temperature, e.g., buried/non-buried, onshore or offshore environment. For offshore pipelines, sea temperature varies significantly depending on geographic location and water depth. If the test

⁹ Canadian Standards Association (CSA), 5060 Spectrum Way, Mississauga, ON, Canada L4W 5N6

¹⁰ ASTM International (ASTM), 100 Barr Harbor Drive, PO Box C700, West Conshohocken, PA, 19428-2959 USA

¹¹ Deutsches Institut für Normung (DIN), Am DIN-Platz, Burggrafenstraße 6, 10787 Berlin, Germany

temperature is high, e.g., ≥ 95 °C, the electrolyte temperature should be maintained at an acceptable fixed temperature with an immersion cooling coil. Current standards do not address the details of the process of maintaining electrolyte temperature or for the prevention of electrolyte evaporation. Lack of detailed instructions lead to non-standard test set up and inconsistencies in test outcome. A starting point for subsea pipelines/structures is to cool the electrolyte to 30°C as per the NF A 49-711. This takes advantage of historical data.

Electrolyte volume and oxygen concentration:

Electrolyte evaporation is one of the biggest challenges for high temperature CD testing because it results in a change of volume and increase in the concentration of the ionic components of the electrolyte. Some standards (such as CSA Z245.20) require topping up the electrolyte by the frequent addition of distilled water and replacing the solution every 7 days. Other standards do not have such requirements. For CD testing at 90°C or higher, electrolyte topping up every few hours is often needed. Alternatively, a condenser or continuous feed of electrolyte from a bulk supply may be used. Depending on the design temperature of electrolyte test solution, the variation of electrolyte topping up frequency has a significant impact on CD results. A common test lab practice is to use a rubber cap to seal the CD test cell or use a closed CD cell in order to prevent the electrolyte from evaporation. This practice results in low oxygen concentration or the absence of oxygen during the CD testing, which significantly affects the CD results, particularly during long term CD tests (28 days or longer). Knudsen⁷ pointed out that little CD disbonding occurs in the absence of oxygen.

Reference electrode

One of the common mistakes in CD test methods and practices is using a wrong reference electrode. Requirements for calibration or the type of reference electrode are often not defined. The proper selection a reference electrode for CD testing is critical, because different electrodes suit different electrolyte temperatures. A saturated calomel electrode (SCE) is based on the compositions of $\text{Hg}/\text{Hg}_2\text{Cl}_2$ in saturated KCl. SCE has the disadvantage that it cannot be used above 50°C due to instability of the Hg_2Cl_2 . It also has a significantly higher linear reference potential to temperature coefficient compared with the much lower coefficient of a saturated Ag/AgCl reference electrode. The temperature coefficient is large enough to produce a significant error in potential measurements if they are left uncompensated. As a result, a SCE reference electrode can only be used for CD testing at ambient temperature and is not suitable and should not be recommended for high temperature CD testing (such as 65°C, 95°C, or higher). For CD testing at high temperatures, Silver/Silver Chloride (Ag/AgCl in saturated KCl) should be used. If an Ag/AgCl electrode is used as a reference electrode, one should ensure it is a saturated one. In comparison to the SCE, the Ag/AgCl electrode has the advantage of being useful at higher temperatures. On the other hand, the Ag/AgCl electrode is more prone to reacting with solutions to form insoluble silver complexes that may plug the salt bridge between the electrode and the solution. A double junction Ag/AgCl electrode is recommended.

Both SCE and Ag/AgCl reference electrodes are wet electrodes, where the element is immersed in an electrolyte with a known salt concentration, as opposed to a Cu/CuSO_4 reference electrode, where the metal is in a solution containing dissolved ions of that metal. This electrolyte should be renewed periodically. In use, the electrolyte slowly leaks into the environment through the porous plug. If a reverse flow occurs, the element becomes contaminated. Since wet electrodes require periodic electrolyte replenishment, they are not suitable for permanent installation for long term CD testing. Therefore, regular change or maintenance of the reference electrode, including cleaning and topping up of the electrolyte is recommended. As a result, whether the reference electrode is permanently immersed during the testing, whether the filling hole is open during measuring, whether the reference electrode is regularly checked or calibrated, all of which affect the accuracy of electric potential measured by the reference electrode and thus the CD results.

Other factors that often do not receive attention:

Other factors include: whether a properly sized anode is used with sufficient exposed area, whether the radial cuts are made within 1 hour or longer after cooling of the tested sample, how the radial cuts are made and what cutting tools are used, whether the disbonding radius assessment is made along the cut lines or mid-segments, and so on. Figure 5 illustrates a high temperature FBE coating which became 'brittle' and prone to damage from the cutting process; especially at the tip of the crossing between the two cuts. Measurements along the radial cut lines or mid-segments give different results.



Figure 5: A high temperature FBE sample after CD testing

SURVEY: A GLOBAL INDUSTRY SURVEY ON CD TESTING

A survey was recently conducted on CD testing practices used by the industry, and the detailed questionnaires and responses are given in Table 2. Questionnaires were sent out to global industry shareholders including coating suppliers, coating applicators, and independent testing laboratories. 58 responses from around the world were received. The purpose of this testing survey was to identify just how vastly different a standard CD test method/procedure is interpreted by testing laboratories (whether they are a large accredited laboratory or a small QC lab). The survey highlights the need for greater detail and explanation in written test procedures to align all the laboratories so that the data pool on products and plant performance is comparable. Many of the items in the survey have minor impact on high temperature CD results, but a few are important, e.g., choice and calibration of reference electrode, the topping up or replacement of the electrolyte, and the CD measurements along the cut line or to mid-segment. A future R&D study to get a complete and accurate picture of the CD mechanism will be of value, but the focus of this survey was on production testing and a sampling of test differences from one testing laboratory to another.

Table 2 A global industry survey on CD testing

Question	Response and percentage rating%
What are the commonly used reference standards by you?	(in order of popularity) CSA Z245.20/21 NACE RP0394 ¹³ GBE/CW6 Part 1 ¹⁴ NF A 49-711 ISO 21809-2 ¹⁵ ASTM G95 Other company or project specifications
Do you have a reference electrode permanently immersed in the test cell for the duration of the test?	Yes (23%); No (77%)
Do you change out / replace the electrodes at a regular frequency for long term testing?	Yes (35%); No (65%)
What is the frequency of replacement of reference electrode during testing?	Daily inspection* (2%); None/6 months or more/if it is broken (98%) * Daily cleaning and visual inspection. Add electrode solution as require. We will change the electrode if voltage irregularities are observed; i.e. out of range
What is the base element of your reference electrodes?	Mercury (83%); Silver (42%); Copper (9%)
Do you have the Fill Hole in the electrode cap open or closed when being used for testing?	Open (25%); Closed (75%)
Do you top up the electrode solution or flush out and replace after use?	Don't top up (31%); Top up (53%); Top up and flush out (16%)
How do you determine when the Electrode should be discarded and replaced?	Calibration measures (26%); If broken or supplier's life time (74%)
For test temperatures, do you set the temperature of the steel to the required value or the test cell electrolyte?	Steel only (46%); Electrolyte or both (54%)
During testing do you monitor the test cell electrolyte temperature?	Yes (67%); No (33%)
During testing, do you top up the test cell electrolyte? If yes, what solution do you use?	Yes with distilled/deionized water (67%); Yes with 3% NaCl (18%); Yes with tap water (1%); No (14%)
Do you completely change the electrolyte periodically for long term tests?	Yes (63%); No (37%)
Do you monitor the pH of the test cell electrolyte during testing?	Yes (17%); No (83%)
Do you adjust the test cell electrolyte pH by adding a pH buffer solution?	Yes (13%); No (87%)
After the test duration for high temperature tests, how do you cool the test panels to ambient?	Water quench after a few minutes (9%); Cool to ambient in air conditioned room or fan assisted cooling (91%)
When do you make the radial cuts – immediately upon cell disassembly, or when the panel is cool?	Immediately (27%); Once the panel has cooled to ambient (73%)
What tool do you use to make the radial cuts?	Retractable Blade Knife/Utility Knife (100%)

For testing on thick 3LPE/PP coating samples, how do you remove the top coat to gain access to the FBE layer for final assessment?	Heating the panel to soften the adhesive and peel off the top layer (71%); None or other methods (29%)
How many times do you attempt to lift the edge of disbonded coating using a flicking action with the tool tip?	Once (17%); 2-3 times (54%); More (29%)
When measuring the disbondment, do you measure along the cut line or to mid-segment?	Mid-segment (44%); Farthest disbondment (12%); Cut line (44%)

TEST PROGRAM

A parallel test program was set up for a real offshore pipeline project. The client required conducting the CD tests at 110°C on the same PQT pipe samples for 48 hours (-3.5 V SCE), 7 days (-1.5 V SCE) and 28 days (-1.5 V SCE) in accordance with two different standard test methods:

- Group #1 – CSA Z245.20 standard: Without electrolyte cooling, but the electrolyte was topped up as required to maintain the minimum level and holiday size = 3.2 mm. The electrolyte bulk temperature was measured and recorded.
- Group #2 – NF-A 49-711 standard: The electrolyte temperature was maintained at 30±5°C, and the electrolyte was topped up as required to maintain the minimum level. Holiday size = 6.0 mm

Testing results obtained as per the two standard test methods were to be compared, in order to determine the effect of test methods and testing parameters on the CD behavior of a high temperature FBE primer coating as well as to establish the final test method and criterion for production quality control tests of the pipeline coating for the offshore project.

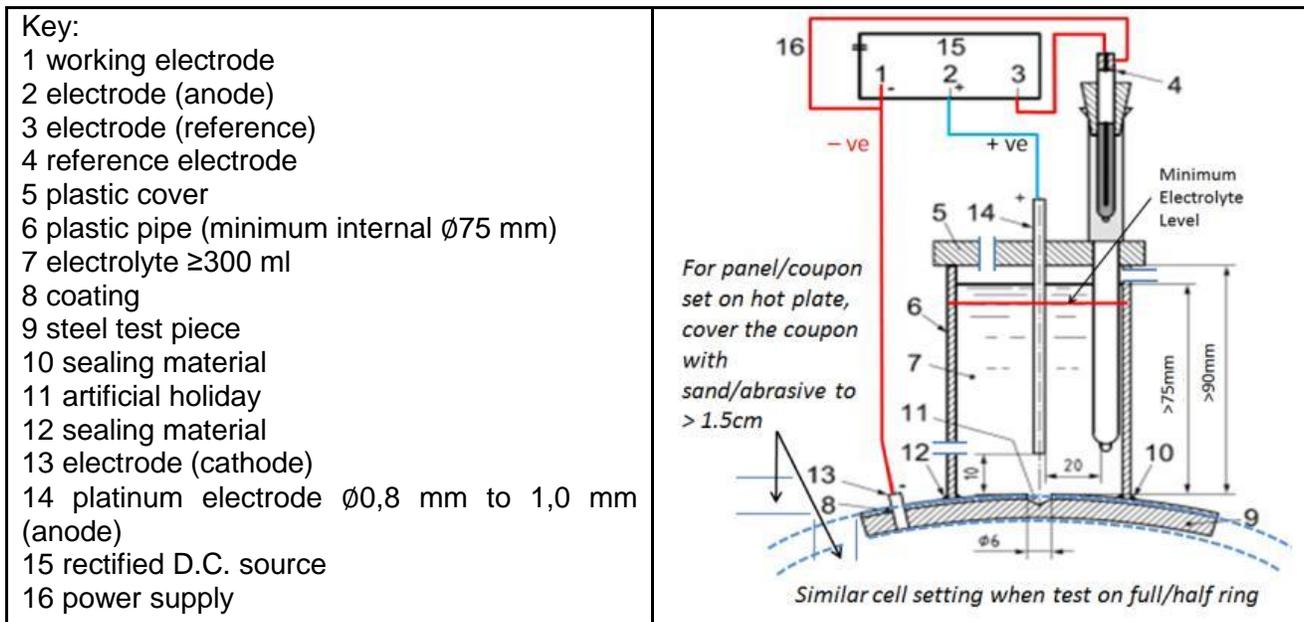


Figure 6: A CD Test Cell

Details of the test set up (shown in Figure 6) were as follows:

Two groups of 24 specimens taken from two adjacent pipes from the same PQT run were tested. For each test duration, 8 specimens were used. The test specimens were cut from the CRA clad pipes coated with a high temperature FBE coating with a thickness of 320-350 μ m. The FBE was to be used

as a primer for 3LPP and multi-layer PP (MLPP) coating systems of an offshore pipeline project and therefore the thickness was lower than normally specified for a standalone FBE. The test coupon was placed in a metal box on a hot plate. The plate was covered with sand/abrasive and the box filled to approximately 1.5 cm above the coating surface. To prepare the panel for the test cell, an artificial holiday of either 3.2mm or 6.0 mm was drilled at the center of the coupon no more than 0.5 mm into the steel. A reference holiday for temperature monitoring was drilled on the same axis several centimeters away. The test cell, 75 mm in diameter, was then attached, centered over the CD holiday. The cell was marked to show the level for 300 ml of electrolyte.

The heat was applied gradually starting from 20°C below the desired test temperature. The temperature control was set to achieve a stabilized temperature between the holiday, the underside of the panel, and the reference holiday. The temperature-reference holiday was insulated during the test to prevent heat loss or faulty readings. Upon achieving a stabilized test temperature of 110°C ±2°C at all the three reference points, the temperature probe was removed and the test cell was filled with at least 300 ml of a 3% NaCl electrolyte solution. For Group #2 NF-A 49-711 tests, a cooling flux was installed inside the test cell to maintain the electrolyte temperature.

The electric cell was then produced by connecting the test specimen to the negative terminal of a source of direct current and by connecting an anode of platinum wire to the positive terminal. The anode was inserted to 10 mm from the bottom. A SCE reference electrode was inserted, 20 mm from the anode. The current flow in the cell was measured and monitored. The CD testing potential of the coated steel specimen was polarized to the specified voltage ± 10 mV with respect to the reference electrode. This potential was measured and adjusted every 4 hours. The electrolyte level was also monitored every 4 hours topped up with distilled water preheated to the test temperature to a level above the 300 ml mark. Upon test completion, the test cell was dismantled and the test plate was allowed to cool to room temperature. The cathodic disbonding of the tested specimen was evaluated within 1 hour of removal from the heat. The disbonding radius from the edge of the original holiday was measured along the radial cut lines, and the average of the measured values was derived from 8 readings taken from one specimen.

RESULTS AND DISCUSSIONS

The two sets of 24 samples were obtained from two adjacent pipes of the same plant and PQT run, therefore coated through an identical and controlled process. The results of Group #1 based on CSA Z245.20 CD testing are displayed in Figure 7. Without electrolyte cooling, the electrolyte temperatures during the tests were found to be stable at 94°-96°C. As shown in Figure 7, CD results of the 48 hour testing were very stable and consistent from one sample to another. Some variations of the CD results were observed from the 7 day testing, but significantly scattered results were obtained from the 28 day testing. During the 28 day testing, very frequent (3-4 times a day) topping up of electrolyte with distilled water was required to maintain the 300 ml minimum electrolyte level. The daily topping up frequency was so high that a decision was made to change the electrolyte replacement from every 7 days to every 4 days.

In addition to the wide scattered CD results, some blisters and non-CD type of disbondment were found on a few 28 day CD samples. Dark color and uneven steel surface were observed on these blister/unintentional disbondment areas. Observations during the 28 day CD testing suggested that those unintentional holidays and associated disbondment were started from the formation of blisters of the coating film away from the original artificial holiday. The radius of these unintentional disbondment/blistering areas varied from 5.00 mm to 13.02 mm (Figure 8). Very little or no unintentional disbondment occurred on samples with minimal coating thickness reduction (Figure 9).

FBE coating thicknesses before and after the 28 day CD test were measured. It was evident that the formation of the unintentional disbondment/blisters was directly related to the reduction of coating thickness of the tested samples. More unintentional disbondment/blistering occurred with those samples in the areas with significant DFT reduction (up to 20%). This significant FBE coating thickness reduction, as well as the dark color and uneven surface associated with the blistering areas, suggested there was a chemical attack on the FBE in these CD test samples. The DFT values measured before the 28 day CD testing were 320-350 μm . In many cases, the reduced coating thickness after the 28 day CDT fell well below 300 μm . It is reasonable to conclude that the inconsistent CD test results between samples, the large amount of disbondment, as well as the formation of unintentional holidays or blistering observed from the CD tests, were a result of the synergistic effect of those influences described by J. Holub, D. Wong, and M. Tan²:

- “Delamination and chemical attack”, “Coating thickness influence” due to the significantly reduced coating thickness as a result of chemical attack, and “Temperature influence” with the hot electrolyte due to the fact that higher water vapour pressure increases water vapour permeation through the coating and porosity of the coating grows as a result of chemical attack and thermal expansion.

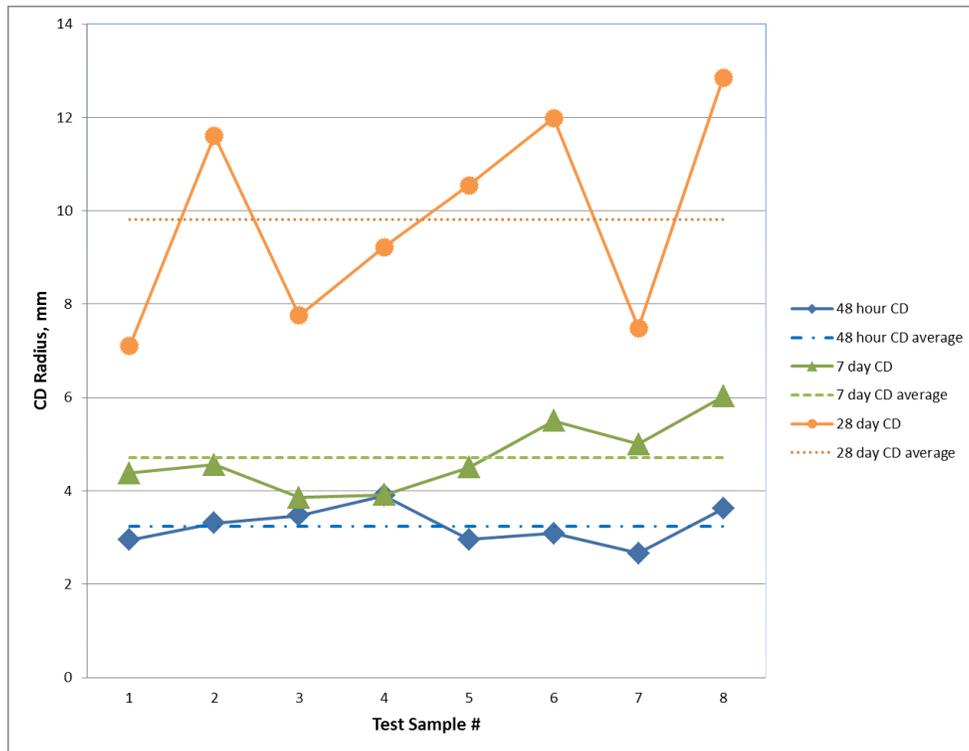


Figure 7: CD results of 48 hours (-3.5 V SCE), 7 days (-1.5 V SCE) and 28 days (-1.5 V SCE) as per CSA Z245.20 (electrolyte temperature of 94°C – 96°C)

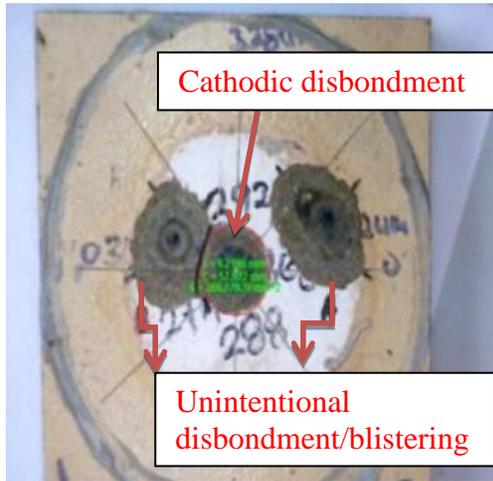


Figure 8: Unintentional disbondment on FBE coating in areas where there was thickness reduction of ~20%

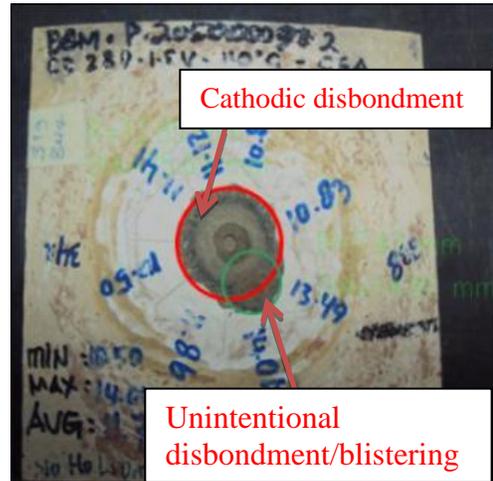


Figure 9: Very little or no unintentional disbondment occurred on samples with insignificant coating thickness reduction

During this 28 day CD test, pH readings were taken each time the electrolyte was replaced. Increases of pH values show that the testing environment became more alkaline. It is difficult to relate the pH readings with hypochlorite building up. In addition to the chemical attack, the near to boiling electrolyte (94°-96°C) as per this Group #1 CD test method creates unstable testing environment/conditions: The near-to-boiling electrolyte could create bubbles at the holiday which impedes the flow of the impressed current. As the electrolyte constantly evaporates in these CD test cells, replacing the electrolyte or topping up distilled water to level up the solution was frequently needed (at least 3-4 times per day). Variation in the amount or timing of the distilled water additions may add to test variability.

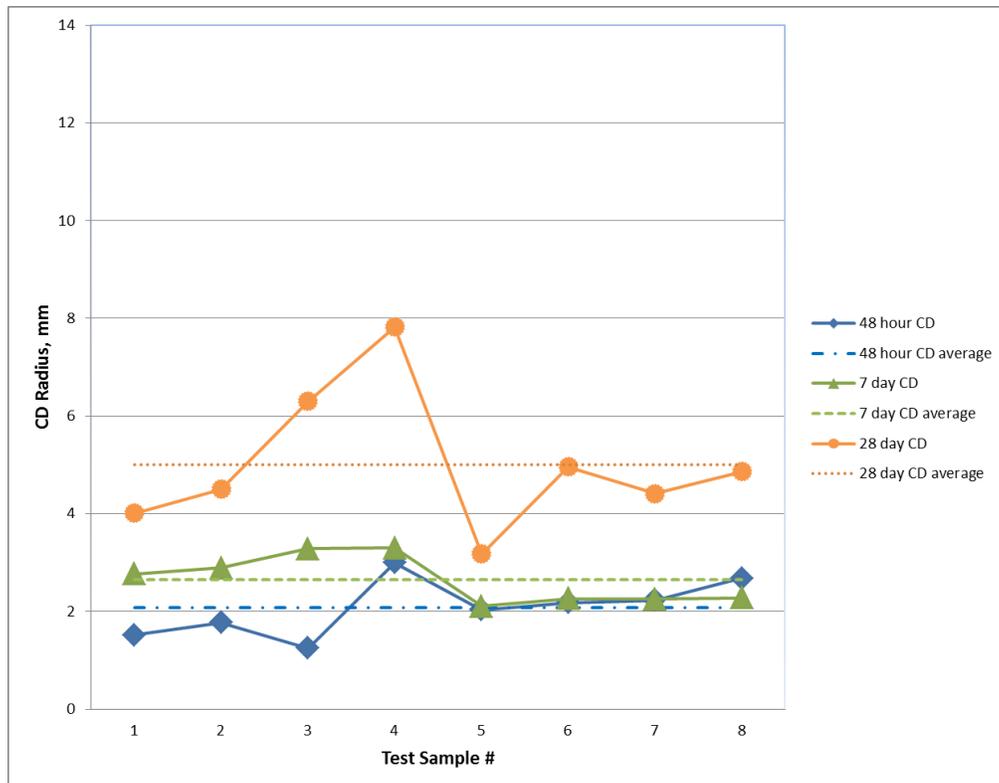


Figure 10: CD results of 48 hours (-3.5 V SCE), 7 days (-1.5 V SCE) and 28 days (-1.5 V SCE) as per NF-A 49-711 with the electrolyte temperature maintained at 30°C±5°C

The results of Group #2 NF-A 49-711 CD testing are displayed in Figure 10. The French standard test procedure requires the electrolyte temperature maintained at a nominal 30°C. The CD results of Group #2 exemplified low scatter between samples and between pipes for all test durations. There was a smaller standard deviation of 1.44 vs. 2.23 of Group #1 for 28 day CD. No unintentional disbondment/blisters were found on the 28 day CD samples of the Group #2 tests. No significant coating thickness reduction was observed on the samples before and after all tests. The consistency of 48 hour, 7 day, 28 day CDT results obtained with this testing method with previous historical data suggests that the coating has been applied within established parameters.

As the industry still searches for, and is trying to develop a definitive, well-accepted standard CD test procedure for evaluating high-temperature coatings and pipelines, a very basic question must be asked: what is the purpose of the test? If the answer is that it is used as a research tool to understand test parameters or as a selection device during the development of new materials or in comparing existing tools, then many proposed test methods, apparatus, and procedures such as those described in the referenced papers or discussed in this paper may be of value. In that case, new or unproven tests can be useful to tease out performance characteristics hitherto not seen or understood. It may also be useful to look at several established products or even new ones and rate how they perform.

However, for production testing, the particular materials and the process of application are known and understood. There is a track record for both and the goal is to assure ourselves that all steps have been taken as required. In that case, the industry needs a test procedure that is stable, understood, easy to run (with the correct equipment and experience), and provides reproducible results. It should have a track record that allows comparison with earlier runs in the same or different plants. For those purposes, the procedure must provide a direction for controlling critical factors which can cause variability in CD test results.

Five of the indicated factors appear to be applicable in the Group #1 CSA Z245.20 and Group #2 NF A 49-711 tests in this study: test duration, film thickness, test temperature, hypochlorite formation/coating attack, and reference electrode.

TEST DURATION: Adding non-standard test procedures in a project specification from the specific consideration of CD test duration may cause issues or confusions on interpretations of test results, as there will be no historical data for comparison. It should be noted that the CD tests for 48 hours at -3.5V in this study is not a common industry practice but a special project request. The normal industry practice is that the production QC tester would either test 24 hours at -3.5V or 48 hours at -1.5V. The production QC CD test procedure of using 24 hours, -3.5 V at 65°C started originally as a CSA standard requirement. The main purpose of the 24 hour CD test is to shorten the response time to catch process problems. It does not make sense to extend it to 48 hours where the -1.5 V test is adequate. A few minutes delay in test termination does not affect the results, but a variation of a few hours does have an effect¹. The results of 48 hour CD tests at -3.5V during both groups of tests with either a hot or a cooled electrolyte were within one mm of each other and comparable with the historic data of 24 hour CD tests at -3.5V on FBE coating at much greater thicknesses. This suggests that a relatively good quality application of the coating was used for this study.

FILM THICKNESS: As discussed earlier in this paper on critical factors affecting CD behavior, film thickness plays an important role. There is a direct correlation of thickness with reduced cathodic disbondment. In the CD tests of this study, the coating thickness ranged between 320 to 350 µm. Typical standalone FBE specifications are in the range of 400 to 550 µm. For high temperature pipelines, the trend is toward the 500 to 600 µm range. In the early days of FBE pipeline coatings, many specifications called for a coating of a nominal 200 µm thickness and the 90 day ASTM G8¹⁶ test often resulted in extraneous disbondment. Normally, the use of greater thicknesses improves the result.

This study shows that the coating thickness of an originally thin film could be further reduced at hot electrolyte temperatures, resulting in extraneous non CD type of disbondment.

TEST TEMPERATURE: A rule of thumb is that higher test temperatures result in larger cathodic disbondment. In practice that 'rule' is confounded by the fact that higher oxygen concentrations in the electrolyte also results in a higher rate of disbondment and, as the electrolyte temperature approaches the boiling point of water, the oxygen concentration drops. Electrolyte temperature also plays a significant role in the rate of hypochlorite formation – where higher temperature results in a higher hypochlorite concentration and a greater film-thickness loss.

HYPOCHLORITE: As there is little historical data from production testing with an isolated anode, non-anode isolation was used for the CD test of this offshore project and the hypochlorite effect was expected. At the anode – which in the field is remote from the pipeline – chloride ions are formed. These chloride ions migrate to and react with hydroxyls at the cathode in the CD cell to form hypochlorite (ClO^-). Hypochlorite is a strong oxidizing agent that attacks the FBE coating. Hypochlorite formation near the FBE is strictly an artificial laboratory phenomenon, but significantly affects the long term CD test results. It causes a reduction in the coating thickness, increasing the rate of disbondment and the likelihood of extraneous disbondments. Utilizing a high-temperature electrolyte is not simulating the actual condition of offshore/subsea pipeline service; it causes an artificial environment that creates an aggressive attack that weakens and thins the coating. In combination with an already thin FBE coating, it results in greater disbondment and an increased propensity to extraneous areas of disbondment. The 48 hour and 7 day CD test results in this study suggest that the impact of the hypochlorite effect might not be significant for short-term production QC test of high temperature FBE based products, as it takes time for hypochlorite to build up and chemical attack to cause coating thickness reduction. However, for long term (28 days or longer) CD tests, it is recommended to adopt a test protocol that maintains the electrolyte temperature in the range of that expected at the pipeline installation. It should also require specified electrolyte topping up/replacement frequencies and the minimum electrolyte level. Defining these variables will improve reproducibility of test results.

REFERENCE ELECTRODE: Both CSA Z245.20 and NF A 49-711 standards require the use of a SCE reference electrode for CD tests. As discussed earlier, SCE has the disadvantage of Hg_2Cl_2 instability and high linear reference potential - temperature coefficient at temperatures of 50°C and above. Although required by CSA Z245.20, SCE was not suitable for Group #1 CSA Z245.20 tests in this study with the electrolyte temperatures measured at 94°C - 96°C . This might also be a contributing factor for the less stable 28 day CD results of this group. On the other hand, the use of SCE should not be an issue for Group #2 NF A 49-711 tests as the electrolyte temperatures were maintained at $30^\circ\text{C}\pm 5^\circ\text{C}$.

SUMMARY

Most of the existing standard CD test methods were originally designed for or started with onshore pipeline applications for service/testing temperatures $\leq 95^\circ\text{C}$. Limited CD data has been seen for service/testing temperatures of higher than 95°C for pipeline coatings, particularly for offshore/subsea pipeline application. Attempts to further develop existing standard CD test methods and specifications to cover higher temperature systems often retain practices that are no longer valid. Some specifications call for procedures that do not have a history and no comparative data to guide acceptance criterion. As such, there is much work needed to understand critical factors affecting cathodic disbonding and to develop a standard CD test method suitable for production qualification and quality control tests.

In the first phase of evaluating standard practices, a literature survey provided a basis for further study of critical factors believed to affect CD test results, particularly at high temperatures.

Next, to determine what is in actual use today, a global survey conducted on CD test practices showed that there are broadly disparate CD test settings with the existing standard test procedures (testing durations, reference electrode types and uses, electrolyte temperatures and topping up/replacing frequency, testing potentials, sample testing temperature, and means of assessment). There are also non-standard test procedures requested in many specifications. This study of actual practices highlights the need for greater detail and explanation in written test procedures or standards.

Finally, to illustrate the effects of test variations on results, parallel laboratory tests conducted on the same plant PQT samples of an offshore/subsea pipeline project showed the effect of using two different standard test methods (one as per CSA Z245.20 and the other as per NF-A 49-711) to meet the requirements of a specification. The CD tests in this study were short term (48 hours and 7 days) and long term (28 days) on a high temperature FBE coating used as a primer for a 3LPP coating system. The results were significantly different.

The three studies highlight a need for the industry to develop a definitive high temperature CD test procedure that is stable, understood, easy to run (with the correct equipment and experience), and provides reproducible results. For long term (e.g., 28 days or longer) CD tests, the industry needs a test protocol that maintains the electrolyte temperature, preferably in the range of that expected at the pipeline installation, and specifies the electrolyte topping up/replacement frequencies and the minimum electrolyte level. Clearly written specification of the important variables will result in more consistent and reproducible CD test results. The draft NACE Standard “Cathodic Disbondment Test for Coated Steel Structures Under Cathodic Protection” prepared by NACE Task Group TG470 is on the right track to address the above needs, however, some critical items discussed in this paper are not covered in the current draft document. Some examples for TG470 group’s consideration are:

- Maximum depth requirement for artificial holidays (e.g. penetration less than 0.5 mm into the steel is needed for more consistent long term and high temperature CDT results among different samples);
- Minimum coating thickness requirement for CD test samples (e.g. CSA Z245.20 requires 350 ± 50 μm for FBE, and also a need to address the thinning effect due to chemical attack of hypochlorite at high temperatures);
- Maximum coating thickness and sample trimming requirement for very thick coating system (e.g. for high temperature coatings and insulations);
- Electrolyte cooling temperature for high temperature CD tests (e.g. unstable CD results with electrolyte temperature of 95°C vs. stable results at 30°C for offshore pipeline project investigated in this study)
- Electrolyte addition and replacement frequency for hot electrolyte (e.g. more frequent refreshment/replacement than weekly may be required for test temperature above 65°C)

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