High-Temperature Cathodic Disbondment Testing: Review and Survey—Part 1

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Cathodic disbondment 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 cathodic disbondment testing standard in the pipeline industry. Factors that affect the high-temperature cathodic disbondment test behavior of fusion-bonded epoxy-based coatings are reported, as well as the results of a global survey on laboratory test practices. Part 2 of this article, to be published in March 2015 MP, will discuss a program that tested the cathodic disbondment of an actual pipe coating.

As explorations and production for oil and gas reservoirs go deeper and fluid temperatures get hotter, there are new demands to fill the gap for test methods to evaluate high-temperature cathodic disbondment performance of pipeline coating systems. Pipe coating materials/products for high-temperature applications, such as high-temperature fusion-bonded epoxy (FBE) powders, are relatively new to the industry. The great majority of conventional FBE powders developed prior to 2000 have a glass transition temperature (Tg) of ~100 °C, and the usual maximum operating temperature specified for conventional standalone FBE is 60 °C. The commonly recommended maximum operating temperatures for standard three-layer polyethylene (3LPE) systems and standard three-layer polypropylene (3LPP) systems are 85 and 110 °C, respectively. Only recently have coating manufacturers developed high-Tg FBE coatings that are also recommended as a primer for multilayered PP systems at temperatures >110 °C. The pipeline industry is currently discussing relevant standards and testing techniques to qualify high-temperature FBE and PP coating products for operating temperatures of 150 °C and higher.

Review: Factors Affecting High-Temperature Cathodic Disbondment

Factors affecting cathodic disbondment behavior of a coating have been extensively studied and reported, and these factors are considered in many reviews by NACE International Technology Exchange Group (TEG) 349X and Task Group (TG) 470 of existing international standard cathodic disbondment test methods.[1-3] Some critical factors, which are more specific to high-temperature cathodic disbondment behavior, include the following.

Hypochlorite Effect and Anode Isolation

Chemical attack of the coating during cathodic disbondment testing can be caused by the formation of hypochlorite or chlorate(I) anions (ClO^-), resulting in coating deterioration/delamination that is quite different from cathodic disbondment.[2] The hypochlorite effect is more significant at higher temperatures. This phenomenon does not occur in the field because the anode and cathode are far apart and do not produce hypochlorite.

An anode isolation scheme can prevent anolyte chlorine gases from migrating to the cathodic sites to form hypochlorite during testing. Historically, the majority of cathodic disbondment testing data have been obtained without anode isolation. Recent research by Al-Borno[4] found that the use of anode isolation causes the pH value of the cathodic disbondment test environment to be significantly higher as early as the first 24 h, but disbondment is also reduced. This contradicts the pH effect revealed in earlier studies by Rodriguez,[5] which suggest that highly alkaline solutions penetrate further than neutral solutions into the crevice formed by a disbonded FBE coating.

This added penetration produces more disbondment by affecting the coating-to-substrate interaction and displacing the coating. Al-Borno[6] also suggests that greater disbondment without anode isolation is an indication that the hypochlorite effect is more significant than the pH effect in increasing disbondment; however, this work only reports results of cathodic disbondment tests conducted for 72 h and 28 days. It is possible that an extended test duration will allow the build-up of hypochlorite to become more significant than the pH effect that initially dominates.

The implementation of anode isolation may result in significantly different disbondment results compared to existing tests. Introduction of this variable requires an evaluation of the resulting data before setting cathodic disbondment acceptance criteria. J. Holub[2] suggests the use of anode isolation, frequent electrolyte refreshment, and proper selection of the electrolyte temperature to avoid coating delamination due to chemical attack. These modifications may invalidate most historical data when establishing a sufficiently large database.

FIGURE 1 A thick 3LPP sample after cathodic disbondment testing for 48 h at –1.5 V and 95 °C.

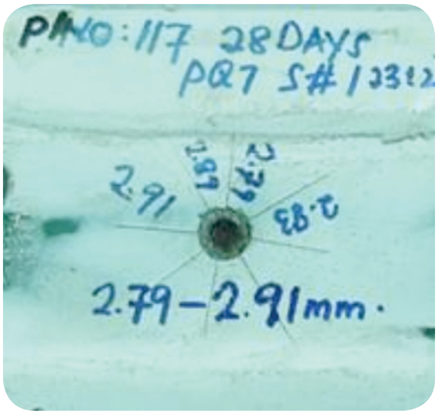

FIGURE 2 Removing the outer layer after cathodic disbondment testing could damage the coating.

TABLE 1. HEATING PROFILES OF A SUBMERGED ARC-WELDED DUPLEX STEEL PIPE DURING THE 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

Specimen Geometry and Preparation Before and After Cathodic Disbondment Test

Although it was found that cathodic disbondment test specimen geometry, whether flat, a curved steel panel, or a tube, had no impact on cathodic disbondment test results,[3] it is practical to use only full ring tube specimens for pipe sizes <16 in (406 mm) in diameter. Cut panels or quarter ring/half shell tubes should only be considered for specimens from pipes that are >20 in (508 mm) in diameter. Pipe ring specimens should be at least 12-in (305-mm) long with the test area >6 in (152 mm) from the cut ends.

Conventional onshore FBE/three layer polyolefin (3LPO) pipeline coatings are often 0.5- to 3-mm thick, and do not require special specimen preparation. For onshore horizontal directional drilling (HDD) or offshore insulation applications, the pipeline coating can be hundreds of millimeters thick. Cathodic disbondment tests on such thick coatings often yield meaningless results with little or no disbondment. Sometimes making the radial cuts and lifting the disbonded coating with a knife is not possible (Figure 1). One solution is to machine thick, rigid coatings to a thickness no greater than 3 mm before cathodic disbondment testing. Some standards/specifications (such as NFA 49-711[7]) assess the cathodic disbondment after heating the 3LPO coating in a furnace to soften the adhesive layer, and detaching the top layer (Figure 2). Excessive heating, however, has the potential of damaging the FBE during the removal process.

Fusion-Bonded Epoxy Film Thickness

FBE film thickness plays an important role in cathodic disbondment resistance: a thicker coating typically shows less disbondment. This is of practical importance because some specifications request a cathodic disbondment test on the primer

FIGURE 3 A FBE sample after cathodic disbondment testing for 28 days at –1.5 V and 65 °C (holiday depth = 0.1 mm).

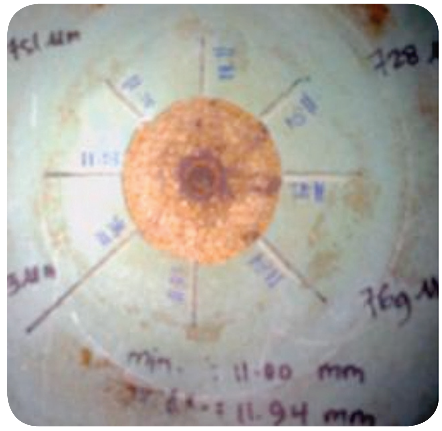

FIGURE 4 A FBE sample after cathodic disbondment testing for 28 days at –1.5 V and 65 °C (holiday depth = 1.1 mm).

alone. FBE as a primer for three-layer coatings has a thickness that is normally significantly lower than the thickness of FBE 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. 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 because existing pipelines usually are low-carbon mild steel. The pipeline industry now uses different types of steel, such as high-strength steel (X80 to X120), duplex and stainless steel, and corrosion-resistant alloys that are mechanically or metallurgically clad to pipes. These special types of steel tend to behave quite differently during the FBE application process. For example, grit blasting different types of steel results in different anchor patterns, which can affect cathodic disbondment results.

Table 1 illustrates variations in a pipe preheating temperature profile of a ~16-in (408-mm) diameter submerged arc-welded duplex steel pipe after going through induction coils at a line speed of 13.4 ft/min (4 m/min) and prior to FBE application. The surface temperatures on the pipe body and along/near the weld seam area were significantly different, with a change in temperature of up to 40 °C. The root cause is that the weld seam and the pipe body of a submerged arc-welded duplex steel pipe have different ferritic-austenitic structures and ferrite contents, which do not respond uniformly to the electro-magnetic induction heating during the coating process.

Holiday Depth

To set up a typical cathodic disbondment test, an artificial holiday, 6 mm in size, shall be drilled with a flat head end mill bit and penetrate <0.5 mm into the steel. Figures 3 and 4 illustrate the results of a 28-day cathodic disbondment test at –1.5 V and 65 °C for two FBE production qualification trial samples with the same production conditions. Only the holiday depths were different. No differences in cathodic disbondment results were found after 48 h at –1.5 V and 65 °C. Compared with Figure 3 (with a holiday depth of 0.1 mm and average cathodic disbondment of 8.28 mm), Figure 4 (with a holiday depth of 1.1 mm and average cathodic disbondment of 11.94 mm) shows darker and more scattered rust marks on the larger disbonded surface. The disbonding increase might be due to an increase in hypochlorite production because more current flows through a system with a deeper holiday. The increase in current flow might also result in much faster OH^- build-up and a higher rate of disbondment.

Test Temperature and Electrolyte Temperature

Industry standards, such as CSA Z245.20,[8] ASTM G42,[9] ASTM G95,[10] and DIN EN-10289,[11] specify a test temperature ranging from room temperature up to the maximum operating temperature, but not above 95 °C. Tests are often conducted without electrolyte cooling or temperature control and do not distinguish between using an oven to maintain temperature—where the electrolyte is the same temperature as the steel test panel—or a hot plate, which allows the steel test panel temperature and the electrolyte temperature to be different. For an offshore subsea pipeline 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 cathodic disbondment test conducted without cooling the electrolyte to the expected seawater temperature does not simulate the offshore/subsea operating conditions. On the other hand, a high temperature is often used to accelerate the degradation process and reduce the time required for a model to predict the performance of a pipeline coating. The challenge is the incomplete understanding of the temperature effect with a hot electrolyte.

In designing a cathodic disbondment qualification test for a coating in a specific project application, the electrolyte temperature must be controlled. One idea is to simulate the actual/specific external service temperature. For offshore pipelines, the sea temperature varies significantly depending on geographic location and water depth. Existing standards do not address the details of maintaining electrolyte temperature or the prevention of electrolyte evaporation. This results in a non-standard test set-up and inconsistencies in test outcomes. Historical data suggest a starting point for subsea structures with the electrolyte at 30 °C per NFA 49-711.

Electrolyte Volume and Oxygen Concentration

Electrolyte evaporation is one of the biggest challenges for high-temperature ca-

TABLE 2. A GLOBAL INDUSTRY SURVEY ON CATHODIC DISBONDMENT TESTING(A)

Question	Response and Percentage Rating
What reference standards do you commonly use?	(In order of popularity) CSA Z245.20/21 NACE SP0394[13] GBE/CW6 Part 1[14] NFA 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 a reference electrode during testing?	Daily inspection* (2%); none/six months or more if it is broken (98%) *Daily cleaning and visual inspection. Add electrode solution as required. 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?	Heat 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%); two to three 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%)

(A)Results are based on 58 responses from global coatings industry shareholders.

thodic disbondment testing because it results in a change of volume and an increase in the concentration of the ionic components of the electrolyte. Some standards (e.g., CSA Z245.20) require topping up the electrolyte by frequently adding distilled water and replacing the solution every seven days. Other standards do not have such requirements. For cathodic disbondment testing at 90 °C or higher, topping up the electrolyte every few hours is often needed. Alternatively, a condenser or continuous feed of electrolyte from a bulk supply may be used. A rubber cap that seals the cathodic disbondment test cell or a closed cathodic disbondment cell can prevent electrolyte evaporation. Unfortunately, this practice results in low oxygen concentration during cathodic disbondment testing, which significantly affects the cathodic disbondment results, particularly during long-term cathodic disbondment tests (28 days or longer). Knudsen[12] pointed out that little cathodic disbonding occurs in the absence of oxygen.

Reference Electrode

Common mistakes in cathodic disbondment test methods and practices result from using the wrong reference electrode and undefined requirements for calibration/maintenance of the reference electrode. Different electrodes suit different electrolyte temperatures. A saturated calomel electrode (SCE) is based on the composition mercury/mercury(I) (Hg/Hg_2Cl_2) in saturated potassium chloride (KCl). A SCE 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 saturated silver/silver chloride (Ag/AgCl) reference electrode. The temperature coefficient difference is large enough to produce a significant error in potential measurements unless compensation is made. As a result, at high temperatures, a saturated Ag/AgCl reference electrode should be used.

Both SCE and Ag/AgCl reference electrodes are wet electrodes, requiring periodic electrolyte replenishment. They are not suitable for permanent installation for long-term cathodic disbondment testing. The Ag/AgCl electrode is also more prone to reacting with solutions to form insoluble silver complexes that can plug the salt bridge between the electrode and the solution; so regular change or maintenance of the reference electrode, as well as cleaning and topping up of the electrolyte, is recommended. Other considerations include whether or not the reference electrode is permanently immersed during the testing, the filling hole is open during measuring, and the reference electrode is regularly checked or calibrated.

Other Factors

Other factors that often do not receive attention include whether the anode surface area is large enough to provide sufficient current flow to the cathodic, whether the radial cuts are made within 1 h or longer after the test sample cools, how the radial cuts are made and the cutting tools are used, whether the disbonding radius assessment is made along the cut lines or mid-segments, etc. Some high-temperature FBE coatings tend to be brittle and are prone to damage from the cutting process, especially at the tip of the crossing between the two cuts. Measurements along the radial cut lines and mid-segments give different results.

A Global Industry Survey on Cathodic Disbondment Testing

Table 2 summarizes 58 responses to a questionnaire sent to global industry shareholders, including coating suppliers, coating applicators, and independent testing laboratories. These responses demonstrate the wide variations in interpreting existing standards and the need for a clearly detailed test method that can be uniformly applied.

Closing Remarks

Many critical factors affect cathodic disbondment test results, particularly at high temperatures. The global survey of cathodic disbondment test practices shows there are broadly disparate test settings within the existing standard test procedures (i.e., test durations, reference electrode types and uses, electrolyte temperatures and topping up/replacment frequency, testing potentials, sample testing temperatures, and means of assessment). The study highlights the need for greater detail and explanation in written test procedures or standards.

Attempts to further develop existing standard cathodic disbondment 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 or comparative data to guide acceptance criterion. As such, much work needs to be done to understand critical factors affecting cathodic disbonding and develop a standard cathodic disbondment test method suitable for production qualification and quality control tests.

Part 2 of this article, to be published in March 2015 *MP*, will discuss a program that tested the cathodic disbondment of an actual pipe coating.

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