Considerations associated with the testing of external pipeline coatings


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CONSIDERATIONS ASSOCIATED WITH THE TESTING OF EXTERNAL PIPELINE COATINGS

D. Abreu¹, M. Y.J. Tan¹, A. Bryson³, A. Fletcher⁴, G. Cope⁵, G. Pearson⁶

¹Deakin University, Geelong, ³APA Group, Melbourne, ⁴Pentair Water Solutions, Melbourne, ⁵Geoff Cope & Associates Pty Ltd, Melbourne, ⁶Pipeline industry consultant, Melbourne

SUMMARY: Reliable testing methodologies for the assessment of protective coatings are critical for ensuring the integrity and durability of pipeline coatings (such as field joint coatings) and the mitigation of pipeline corrosion. Currently the failure of joint coatings is one of the major concerns in corrosion protection of pipelines, although they represent only approximately 5% of the coated area in a pipeline system. This paper presents an overview of major testing methodologies currently used in the pipeline industry for the selection, testing, and life prediction of coatings, in particular field joint coatings. Particular focus is on the discussion of difficulties and limitations in testing methods for assessing pipeline coating cracking, cathodic disbondment and loss of adhesion. It is shown that there are limitations in current methodologies in evaluating the coating flexibility - a key parameter for avoiding coatings cracking during hydrostatic testing, cyclic pressure operation and field bending. Methodologies for assessing the effect of holidays in coatings on the cathodic disbondment of pipeline coating under excessively negative cathodic protection (CP) voltages also require improvement. Furthermore, methods for understanding the effects of coating wet adhesion on pipeline coating, cracking and disbondment also need attention. Some preliminary results for addressing some of these issues are also presented in this paper.

Keywords: Protective coating, testing methods, cathodic disbondment, flexibility, adhesion.

1. INTRODUCTION

Steel pipelines have been used for a long time as a feasible and reliable system for oil and gas transportation as well as for water transmission. This paper focusses on oil and gas (Energy) pipelines. Since the 1950s the Australian oil and natural gas pipeline industry has grown to meet the country’s needs. Currently there are more than 33,000 kilometres of pipelines transporting oil, gas, ethane and slurry in Australia (according to The Australia Pipeline Industry Association Ltd information of 11/06/2010). Since the late 1990s the Australian pipeline industry and the Federal Government has increased investments in research related to the pipeline industry development [1]. The establishment of the Energy Pipeline Cooperative Research Centre (EPCRC) is an example of the government and industry investment in pipeline research and development. External pipeline coating cracking and disbondment is one of the EPCRC’s research focused areas. To protect steel pipelines, external protective coatings are used in conjunction with an adequate level of impressed-current cathodic protection (CP). The coatings act as the primary physical and dielectric barrier for protection against corrosion, with the CP designed to protect the pipe against corrosion when parts of the coating are inadequately applied, damaged or degraded [2-4]. For instance when a holiday occurs in the coating, the CP works to protect the holiday area. A possible complication is the disbondment of the coating from a holiday area, creating a crevice. Such crevices may become a ‘shielded area’ on the pipe surface that is not sufficiently protected by the CP [5].

To build a long length pipeline, a large number of factory coated pipes are used and joined on the field. The pipe’s coating is normally applied in pipe production plants, however the area surrounding the joints is uncoated and a girth welding operation for joining the pipes is performed in the field. Once the pipes are welded and joined, the exposed joint area surface is prepared to receive a coating, which commonly is identified as the field joint coating (FJC). The FJC needs special attention due to difficulties in its application and uncontrollable environmental conditions [6-8]. Currently the
failure of joint coatings is one of the major concerns in corrosion protection of pipelines, although they represent only approximately 5% of the coated area in a pipeline system. Consequently, reliable tests to assess the coating’s performance and clearly identify potential failure modes are mandatory for pre-qualification of pipeline coatings in order to ensure a suitable pipeline lifetime.

1.1 Coating evaluation tests
By 1958 a ten years program to evaluate coatings was completed using field and laboratory experiments [9]. The results obtained showed that burial tests in the field needed a long time and the variability of results was high, leading to inconclusive observations. The laboratory tests undertaken evaluated one specific property per type of test. The table below presents a list of the coatings’ performance characteristics studied under laboratory controlled conditions:

Table 1. List of performance characteristics of coatings studied by Allen study of 1958 [9]

<table>
<thead>
<tr>
<th>Characteristic</th>
</tr>
</thead>
<tbody>
<tr>
<td>Electrical resistance</td>
</tr>
<tr>
<td>Rate and amount of water absorption</td>
</tr>
<tr>
<td>Resistance to deformation under pressure</td>
</tr>
<tr>
<td>Resistance to cracking and spalling under impact</td>
</tr>
<tr>
<td>Resistance to cracking in bending</td>
</tr>
<tr>
<td>Adhesion to pipe metal</td>
</tr>
<tr>
<td>Deterioration to soil environment</td>
</tr>
<tr>
<td>Deterioration in petroleum oils</td>
</tr>
<tr>
<td>Effects of cathodic protection</td>
</tr>
</tbody>
</table>

From 1964 to 1977 standardized tests were developed and reviewed aiming to evaluate pipe coatings [10]. In 1978 [11], thirteen test methods from the American Society for Testing and Materials (ASTM) had been developed specifically for pipeline coating testing:

Table 2. Initial 13 ASTM test methods for pipeline coating evaluation [11]

<table>
<thead>
<tr>
<th>Standard</th>
<th>Standard title</th>
</tr>
</thead>
<tbody>
<tr>
<td>ASTM G6</td>
<td>Abrasion Resistance of Pipeline Coatings</td>
</tr>
<tr>
<td>ASTM G8</td>
<td>Cathodic Disbondment of Pipeline Coatings</td>
</tr>
<tr>
<td>ASTM G9</td>
<td>Water Penetration into Pipeline Coatings</td>
</tr>
<tr>
<td>ASTM G10</td>
<td>Bendability of Pipeline Coatings</td>
</tr>
<tr>
<td>ASTM G11</td>
<td>Effects of Outdoor Weathering on Pipeline Coatings</td>
</tr>
<tr>
<td>ASTM G13</td>
<td>Limestone Drop Test for Pipeline Coatings (Withdrawn in 2003)</td>
</tr>
<tr>
<td>ASTM G14</td>
<td>Falling Weight Test for Pipeline Coatings</td>
</tr>
<tr>
<td>ASTM G17</td>
<td>Penetration Resistance of Pipeline Coatings</td>
</tr>
<tr>
<td>ASTM G18</td>
<td>Test for Joints, Fittings, and Patches in Coated Pipelines</td>
</tr>
<tr>
<td>ASTM G19</td>
<td>Disbondment of Pipeline Coatings by Direct Soil Burial (Withdrawn in 2010)</td>
</tr>
<tr>
<td>ASTM G20</td>
<td>Chemical Resistance of Pipeline Coatings</td>
</tr>
<tr>
<td>ASTM G42</td>
<td>Cathodic Disbonding of Pipeline Coatings Subjected to Elevated or Cyclic Temperatures</td>
</tr>
<tr>
<td>ASTM G55</td>
<td>Evaluating Pipeline Coating Patch Materials</td>
</tr>
</tbody>
</table>

Additional ASTM pipeline coating standards have been approved since 1978 as follows:
- ASTM G62 (07) Holiday detection of pipeline coatings;
- ASTM G70 (07) Ring bendability of pipeline coatings;
- ASTM G80 (07) Specific cathodic disbondment testing of pipeline coatings;
- ASTM G95 (07) Cathodic disbonding test of pipeline coatings (attached cell method).

Two of these initial Standards were subsequently withdrawn, and all the remaining Standards have been revised. Other standards were developed and are periodically revised in order to provide better evaluation of the pipeline coating properties and to accommodate the fact that coatings have changed and new materials have been introduced over the decades. Local standards were also created for the fulfillment of local market needs such as the standard test methods generated by Standards Australia and Standards New Zealand. A list of the most important standard test methods related to the pipeline industry and focused on the Australian market is shown below in Table 3.

Additionally, one recent concern for liquid applied coatings is the volatile organic compounds (VOC) and other coating constituents that could have an impact on human health and on the environment [12]. Considering the continuous development of the Australian pipeline industry and the objective to reach a longer pipeline lifetime, it is necessary to continuously revise and develop testing methods in order to better represent field conditions. Simulating field conditions in a laboratory is a very complex and challenging task and different field condition might need different evaluation tests [13].

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Good application properties, toughness (impact resistance and indentation damage resistance), water and chemical resistance, and good adhesion to the substrate are key properties of a good protective coating for buried pipes [14, 15]. The interface between the steel (substrate) and the coating is the most important location for failure initiation [10]. Three main coating properties are related to this interface: dry/wet adhesion, cathodic disbondment, and bendability or flexibility [10]. The EPCRC is working on projects in order to verify the suitability of laboratory testing methods for pipeline coating performance evaluation. As a result, some standard test methods are under evaluation for possible future improvements and better understanding of the relevance of the test results, to actual field performance, their limitations and applicability.

Table 3. Some standard test methods with high impact on the Australian pipeline industry and related to pipeline coating properties evaluation

<table>
<thead>
<tr>
<th>Standard</th>
<th>Standard title</th>
</tr>
</thead>
<tbody>
<tr>
<td>AS/NZS 1518:2002</td>
<td>External extruded high-density polyethylene coating system for pipes</td>
</tr>
<tr>
<td>AS/NZS 3862:2002</td>
<td>External fusion-bonded epoxy coating for steel pipes</td>
</tr>
<tr>
<td>AS 1580.408 series</td>
<td>Paints and related materials – Methods of test (parts 2, 4 e 5)</td>
</tr>
<tr>
<td>AS 2331 series</td>
<td>Tests for coatings (parts 0 to 4)</td>
</tr>
<tr>
<td>AS 3894 series</td>
<td>Site testing for protective coatings (parts 0 to 13)</td>
</tr>
<tr>
<td>AS 4321-2001</td>
<td>Fusion-bonded medium-density polyethylene coating and lining for pipes and fittings</td>
</tr>
<tr>
<td>AS 4352-2005</td>
<td>Tests for coating resistance to cathodic disbonding</td>
</tr>
<tr>
<td>AS 4822-2008</td>
<td>External field joint coatings for steel pipelines</td>
</tr>
</tbody>
</table>

1.2 Coating Flexibility

To confirm the pressure bearing capability, and to identify the presence of any leaks on a pipeline, a hydrostatic test may be undertaken [16, 17]. The hydrostatic test is performed by subjecting the pipeline segment to a higher stress / strain level than what is normally experienced during service. During testing of a pipeline the coatings are also subjected to these higher limits. The coatings applied to both the main pipe body (applied in the factory) and the FJC’s (applied in the field) both need to be able to withstand the strains associated with hydrostatic testing as well as any other loadings that may occur. Pipeline coatings need an adequate level of flexibility in order to remain intact after such tests. Additionally other factors such as local inhomogeneities, strain concentrations at welds and higher than nominal applied coating thicknesses can lead to higher internal stresses in the coating and could be a mechanism for coating cracks and loss of adhesion [18].

1.3 Cathodic Disbondment

On a coated pipeline system using CP a potential is applied in order to protect the pipe steel against corrosion. Changes to the soil and coating resistivity during service may require frequent CP tuning. At coating holidays a cathodic reaction occurs in the presence of moderate applied potential (CP). The oxygen reduction reaction is dominant and is described on the equation below (1):

\[ 2H_2O + O_2 + 4e^- = 4OH^- \]  (1)

The hydrogen evolution reaction occurs at “higher” (more negative) applied CP potentials as shown (2):

\[ 2H_2O + 2e^- = H_2 + 2OH^- \]  (2)

The increase of OH- ions might have an influence on the loss of adhesion between the coating and the steel through the pH increase [19]. This alkalinization can be a main factor affecting cathodic disbondment by hydrolysis of the interface coating-steel or the coating de-polymerization [20]. If the H atoms enter on the metal surface, corrosion on the pipeline steel may also occur through hydrogen induced cracking (HIC), sulphide stress cracking (SSC) or the development of high pH stress corrosion cracking (SCC) [21]. There is also a possibility that the evolution of H gas might contribute to the coating disbondment [22]. Moreover, a recent study presented results showing that the cathodic delamination has a high dependency on the coating permeability of corrosive species and the number of interaction points between the coating and the substrate [23].

1.4 Adhesion

Adhesion is the sum of the interaction forces between the coating and the substrate (steel) and cohesion is the sum of the interaction forces within the coating itself. The loss of adhesion can be from the coating to the substrate or to another coating such as between the plant coating and the FJC or between coating layers. The loss of cohesion can be a horizontal...
rupture (lateral splitting) or a vertical rupture (vertical splitting, cracking, checking, alligatoring, etc) [24]. Figure 1 represents a three layer coating system and the forms in which adhesion loss and cohesion loss may occur. The standard AS4822-2008 [25] Section 5 defines testing methods to evaluate the adhesion of Petrolatum and Polymeric Tape Coatings for FJC repair, these tests include testing the adhesion between tape layers, testing the adhesion from the tape to the steel (substrate) and from the tape to the plant coating for instance.

![Diagram of coating system with failure modes](image)

Figure 1 Adhesive and cohesive failure forms: (a) loss of adhesion to the substrate; (b) loss of adhesion to another type of coating; (c) horizontal loss of cohesion on one of the coatings; (d) vertical loss of cohesion of one or more coatings.

Coatings can absorb moisture thereby changing their electrical resistance and increasing the transport of ions to the interface coating-steel. This can cause loss of adhesion and/or blistering between coatings or within the coating [26]. Water and salts can penetrate the coating. This is exacerbated by any impurities on the metal substrate that can lead to osmotic blistering and therefore loss of adhesion on the coating-substrate interface. The formation of blistering can cause corrosion underneath the blister if underneath the blister a suitable environment develops [27]. CP can also contribute to ions transport to the coating-substrate interface contributing to cathodic disbondment surrounding the holiday creating crevices or even contributing to blistering. Whilst that is uncommon it is possible with more permeable coating types as the FBE coatings [26].

2. CONSIDERATIONS ABOUT THE TESTING METHODS

Following on from the discussion in Section 1 a range of testing was undertaken related to coating flexibility, cathodic disbondment and adhesion. The considerations here mentioned are derived from performing the flexibility, cathodic disbondment and adhesion tests. All tests were based on Australian and commercially well-known testing methods. All samples used were prepared following commercial practices and using API 5L X-65 grade steel plates coated with epoxy coatings, as are commonly used in the pipeline industry. The steel plates were coated with a range of different coating types and thicknesses.

2.1 Bendability/Flexibility of FJC based on AS/NZS3862:2002 Appendix L

The main purpose of flexibility tests was to simulate field bending that might occur on pipeline field assembly operations [28]. Additionally it is important where high strains can occur during field hydrostatic testing. The first effort to generate a standard test method for laboratory simulation of field bending conditions resulted in the issuing of ASTM G10 [29], which was originally approved in 1969, is currently active and was lastly reapproved in 2010. This standard describes a specific radius variable mandrel test system – well defined and explained on the document – to perform bending on a pipe of 2.5 m in length and 33.4 mm in diameter over a range of different varying diameters, allowing determination of the diameter at which cracks occur.

ASTM G70 [30], firstly published in 1981, uses a section of a real field pipe such as a ring of 50 mm in length, removing the coatings on opposite sides of the pipe, resulting in an uncoated section of 60° on both sides, and pressing the ring laterally as shown on Figure 2. The compression is related to the pipe diameter and the presence of cracks is monitored during the ovalisation process, stopping the process when cracks are observed.
Figure 2 ASTM G70 – 07 test method for horizontal bending/ovalization aiming to evaluate coating flexibility.

The European standard test method BS EN 10290:2002 [31] Appendix K of 2002 describes a different test method based on a 3 point bend test using mandrels, to bend a coated metal plate of 50 mm x 300 mm x 6 mm in size. After the bending process no cracks shall be identified using a high voltage holiday detector. To calculate the mandrel diameter a formula is used (3), where D is the mandrel diameter in millimetres, t is the coupon thickness in millimetres and S is a factor dependent of the testing temperature and the piping component type.

\[
D = \frac{t(1-S)}{S} \quad (3)
\]

Additionally it is possible to perform the test using a real pipe section of 55 mm x 355 mm in length (along the longitudinal axis of the pipe) where the mandrel diameter is calculated using the formula (4), \(D_0\) is the original specified pipe diameter in millimetres, t is the nominal pipe wall thickness in millimetres and S is a factor as previously defined.

\[
D_1 = \left( \frac{1}{\frac{S}{t} + \frac{1}{D_0 - t}} \right) - t \quad (4)
\]

This standard specifies the mandrel diameter depending on whether it’s a pipe or a field joint coating and specifies testing at 23°C and or 0°C. The standard is for polyurethane coatings and requires the pipe coatings to withstand 3.0% strain at 23°C and 2.0% strain at 0°C. A set deflection rate of 25 mm/min is specified. Interestingly the standard also recognises the problem of “peaking” of samples during 3 point bending, whereby a gap develops in the central region that is in contact with the mandrel/former. In this region the coating may be bent to a lower diameter (higher strain) than the adjacent areas (if not held tightly against the mandrel). The standard requires that areas of “peaking” above a specified amount be discarded from the area to be holiday tested.

The NACE International has published a standard, RP0394-2002 [32], which also has a flexibility test. It specifies both a 3 point and a 4 point bending test. The standard defines two possible samples, a laboratory coated specimen with 25 mm wide x 200 mm long x 6 mm thick or a pipe section cut as Figure 3 shows, with 25 mm wide x 200 mm long (along the pipe longitudinal axis) x pipe wall thickness.

Figure 3 NACE standard RP0394-2002 pipe specimen schematic figure.

To calculate the mandrel diameter to be used, this standard presents an equation (5) where R is the mandrel radius, t is the effective strap thickness as shown in Figure 3 and S is the strain (deflection) in degrees per pipe diameter (°/PD). There is no comment on the phenomenon of “peaking” that occurs in 3 point bending, and no comment on how the 4 point bending
can give accurate results given that “peaking” will also occur, and that constant strain in a 4 point bend test only occurs between the central two mandrels when the steel is in the elastic range (i.e. up to approx. 0.5 % strain).

\[ R = \frac{(57.3) (t)}{S} - \frac{t}{2} \quad (5) \]

Similar to the European standard testing method, the Australian standard AS/NZS 3862:2002 [33] Appendix L specifies a 3 point bend test with a specific formula for calculating the mandrel diameter over which a coated coupon shall be bent. The coupons dimensions are defined as 25 mm x 200 mm x 10 mm or 25 mm x 200 mm x pipe wall thickness, with the 200 mm dimension oriented as parallel to the pipe axis, i.e. longitudinally. For this test the coupons should be cooled to 0°C and held at that temperature for 1h before the bending starts. The inspection for cracks or disbonding is visual and the bending process should occur in 10s to 30s. There is no comment on the phenomenon of “peaking” that occurs in 3 point bending. This standard requires that for pipes with diameters larger than 273 mm, the coating shall withstand a deflection of 3.75°/pipe diameter and for smaller diameter pipes a deflection of the 1.5°/pipe diameter. In calculating the mandrel diameter the following (6) formula shall be used:

\[ \beta = \frac{360}{\pi (2R + C)} \quad (6) \]

where \( \beta \) is the angle of deflection in degrees per pipe diameter length, \( C \) is the coupon thickness in millimetres and \( R \) is the mandrel radius in millimetres.

The relationship between strain and deflection in degrees per pipe diameter (a term used for field pipe bending) is given in Equation (7). A deflection of 3.75° per pipe diameter is equal to a strain of 3.3%.

\[ \text{Deflection (Degrees per pipe diameter)} = (\% \ \text{strain}) \times 3.6/\pi \quad (7) \]

It was decided to build equipment for 3 point bending tests. A set of mandrels of different diameters were built and coupons of 25 mm x 200 mm x 5 mm coated on different thickness were tested. Aiming to evaluate the difficulties associated with this test, some important observations can be made. Firstly, there is a difficulty associated with this type of bending system, where “peaking” was observed, even on larger diameter mandrels, Figure 4. The problem of this “peaking” is that these regions experienced a much higher level of strain, resulting in cracking occurring prematurely due to the high strain concentration on the centre of the coupon. This effect was confirmed on strain gauges measurements and simple computational analysis, Figure 5. The Standard BS EN10290:2002 [31] identifies this problem and give the orientation to disregard the results on the central region if a “peaking” is presented. This problem has been recognised in the coatings industry and ways to eliminate the problem have been developed. Following the advice of an industry partner, clamps were used to hold the coupon tightly against the mandrel during testing. This prevented any peaking and resulted in much more consistent results and a much more uniform application of strain to the coating under test, as shown on Figure 6. Another issue associated with this test is cracks that might occur during over-bending. When the side of the inner mandrel comes in contact with the sample, increasing sharply the strain on that region it can become wedged between the inner and outer mandrels, leading to not representative cracks. The solution in this case is to avoid this over-bending by stopping the bending process before it can occur.

Figure 4 “Peaking” occurred when a mandrel is pressed on a specimen of 5 mm of steel coated with FBE.
Figure 5 Higher strain on the coupon centre caused localized plastic forming and “peaking”: (a) Strain gauges measurement; (b) Simple computational simulation.

Recent work based on the evaluation of the AS/NZ 3862:2002 [33] showed that the results of some different coating type/thickness composition on the flexibility behaviour [34]. This work is continuing at Deakin University with the EPCRC supporting it.

2.2 Cathodic Disbondment Resistance (CDR) test using AS4352-2005

The first standard test designed to evaluate the coating resistance to cathodic disbondment dates from the 1960s. As burial tests can take a long time to provide any result, laboratory tests were presented as a solution to simulate the field condition with repeatability and able to evaluate the coating properties. The standard test method ASTM G8-96 [35] was initially published in 1969 and its last revision date was 2010. Another related standard test method initially published in 1975, ASTM G42-11 [36], was designed to evaluate the coatings resistance to cathodic disbondment at elevated temperatures. The cathodic disbondment test was considered as the most reliable test to evaluate the coatings performance [37]. In 1988 a study conducted tests using these two standards and showed that the test results presented in a high variation between samples and that the high temperature condition did not necessarily represent an accelerated testing method [15]. However, the authors’ opinion is that the CDR tests should be done at the temperature at which the coating will be subjected to during field operation.

Other relevant standards are ISO 21809-3:2008(E) [38] for FJC and AS 4352-2005 [39] for CDR testing. Special attention is given to this Australian standard which was initially published in 1995. Although the three standards mentioned have similar CDR tests, ASTM G8 [35] and ASTM G42 [36] uses as the electrolyte 1% of weight as an equal composition of sodium chloride, sodium sulphate and sodium carbonate while ISO 21809-3:2008 [38] and AS 4352-2005 [39] use 3% of sodium chloride. Another main difference is the electrolytic cell (test cell), where the European standard and the American standard are designed using a reference electrode aiming to control the cathodic potential applied, whilst the Australian standard focuses on the impressed current applied. ASTM G8 uses a magnesium anode to provide the cathodic protection whilst ASTM G42, the Australian and the European standards use platinum electrodes and an impressed current to provide the protection. The Australian standard AS 4352-2005 [39] uses a fixed impressed current for the CDR test and not a fixed voltage and the current must be adjusted (if needed) during the 28 days of testing in order to keep the same current flow to the steel.

Using the standard AS 4352-2005 [39] some tests were undertaken in order to evaluate the cathodic disbondment of a range of FBE coatings. To measure the disbondment area the formula below is used (8):
where \( A \) is the disbondment area and \( R \) is the average of eight evenly spaced radius measurements of the disbondment verified beyond the edge of the drilled initial defect.

The test results showed that the CDR test present variations between different tests done on the same coating applied on a specific steel substrate and there are no noticeable differences between most of coating types. Six coating types were used testing 4 samples for each type. One coating type presented no disbonding in one sample while the other three samples presented disbondment. Another coating type showed a lower disbondment in 2 samples and a higher disbondment in other 2 samples. Two samples presented a higher disbondment with at least one sample having a disbondment rate slightly higher than 100 square millimetres.

It is important to observe that all samples were ranked as grade A according to the disbondment rate defined on the AS 4352-2005 [39], the best grade possible, with the disbondment area far from what would be classified as grade B. The grade B would require a disbondment higher than 500 square millimetres though the samples tested presented a much lower disbondment. The standard could be more restrictive in order to evaluate better this type of FBE field joint coatings.

One issue with this standard is that the assessment can be subjective to some extent. Firstly in identifying the disbondment area and secondly in measuring the disbonded radius. Using a sharp rigid knife the disbondment shall be identified by removing the coating which requires less force than that required to remove the non-disbonded coating. It is not a big issue considering that to remove the non-disbonded coating a mark on the metal plate will be left but this operation requires some attentive observations to avoid false disbondment identification. To measure the disbondment radius, eight evenly spaced radius measurement must be done but it is not defined the initial orientation of the measurement. As Figure 7 shows, the orientation on the sample cut direction (rectangular) or on the largest disbondment direction might result in different disbondment area. The measurements of some samples were done using both orientations and a difference of almost 10% were found in one sample with differences in all samples, some for higher value but other for a lower disbondment area. The measurement was done using the sample orientation direction as shown in Figure 7. However differences of 10% would usually be considered as being within the normal range of variability for this test. Furthermore the extent of disbondment can often be seen in the colour change of the steel surface over the disbonded coating area once the disbonded coating has been removed.

![Image](image_url)

Figure 7 The disbondment radius measurement may vary the result.

### 2.3 Adhesion tests using ASTM D4541-09

Adhesion tests usually measure the extraction forces to remove the coating from its substrate, which combines multiple factors including the adhesion, and in our tests there was some correlation between good adhesion and high flexibility. However, results from adhesion tests are not considered as unequivocal criterion to evaluate coating quality [10]. One challenge about adhesion tests is to comprehend the results meaningfully, since most tests relate only to the pull-off force measured. There are different standard testing methods aiming to evaluation coating adhesion but they are not necessarily equivalent and may vary also from differences in equipment used to perform the tests. Another work presented at the NACE Corrosion Conference showed that all adhesion tests performed on different types of pipeline coatings using the standard test method ASTM 4541-09 [40] resulted in loss of adhesion between the coating and the glue used to set the extraction dolly, and not between the coating and the steel [28]. The coating types used on this research were 3-layer polyethylene (3LPE), powder coated multicomponent, fusion-bonded epoxy (FBE), dual powder FBE, epoxy polymer concrete and urethane type coating. The extraction force could reach 13.8MPa (2000 psi) stress on the dollies. Loss of adhesion occurred
on the dolly-glue interface on all samples and not on the coating-steel interface, though at an extremely high stress [28]. Different types of adhesion tests were developed for different coating types and the commonly tests used are pull-off, shear and peel.

This work evaluated results of adhesion tests performed based on the standard test method ASTM D4541-09 [40]. The pull-off force for coating-steel adhesion measurement can be measured using a specific system as described in Annex A4 of the standard. An automatic pull-off adhesion tester was used for this work. Table 5 shows the results obtained. The dollies used had a diameter of 20 mm and the adhesion strength was calculated from the force measured. The same procedure was used on samples after 24h hot water immersion, as described on the standard test method AS/NZS 3862:2002 [33] Appendix B Table B3, resulting in another group of samples for the test. The glue used for attaching the dollies to the coatings was the one proposed by the equipment manufacturer and the dollies were 20 mm in diameter.

Figure 8 (a) presents a loss of adhesion after the pull off adhesion test while Figure 8 (b) presents a loss of cohesion.

Table 5. Adhesion test results to ASTM 4541-09.

<table>
<thead>
<tr>
<th>Coating Type/thickness (µm)</th>
<th>Preconditioning</th>
<th>Extraction Force (MPa)</th>
<th>Visual Adhesion loss</th>
</tr>
</thead>
<tbody>
<tr>
<td>Type A / 600</td>
<td>None</td>
<td>10.7</td>
<td>Total loss of adhesion</td>
</tr>
<tr>
<td>Type A / 600</td>
<td>Hot wet immersion</td>
<td>6.4</td>
<td>Small loss of cohesion</td>
</tr>
<tr>
<td>Type B / 1000</td>
<td>None</td>
<td>13.0</td>
<td>Partial loss of cohesion</td>
</tr>
<tr>
<td>Type B / 1000</td>
<td>Hot wet immersion</td>
<td>9.5</td>
<td>Small loss of cohesion</td>
</tr>
<tr>
<td>Type C / 500</td>
<td>None</td>
<td>9.4</td>
<td>Adhesion loss on the glue</td>
</tr>
<tr>
<td>Type C / 500</td>
<td>Hot wet immersion</td>
<td>7.5</td>
<td>Adhesion loss on the glue</td>
</tr>
<tr>
<td>Type D / 800</td>
<td>None</td>
<td>10.0</td>
<td>Adhesion loss on the glue</td>
</tr>
<tr>
<td>Type D / 800</td>
<td>Hot wet immersion</td>
<td>10.2</td>
<td>Partial loss of cohesion</td>
</tr>
<tr>
<td>Type E / 800</td>
<td>None</td>
<td>11.0</td>
<td>Adhesion loss on the glue</td>
</tr>
<tr>
<td>Type E / 800</td>
<td>Hot wet immersion</td>
<td>2.7</td>
<td>Adhesion loss on the glue</td>
</tr>
<tr>
<td>Type F / 700</td>
<td>None</td>
<td>14.4</td>
<td>Adhesion loss on the glue</td>
</tr>
<tr>
<td>Type F / 700</td>
<td>Hot wet immersion</td>
<td>7.6</td>
<td>Adhesion loss on the glue</td>
</tr>
</tbody>
</table>

(a) (b)

Figure 8 Adhesion test result: (a) Loss of adhesion; (b) Loss of cohesion.

It can be observed that the adhesion strength was lower for the samples submitted to hot wet immersion, except for the Type D coating which had a similar extraction force. The sample Type A that was not submitted to preconditioning was the unique sample where there was a real loss of adhesion on the interface coating-substrate. All other changes occurred as a loss of cohesion either within the coating or associated with the glue. For the samples Type A and Type B, the preconditioned ones presented lower loss of adhesion but detached on lower forces. One possible conclusion is that the preconditioning caused a loss of cohesion on the coatings. However, for all other types, the loss of adhesion occurred at the glue / dolly / coating boundaries indicating that the glue was inadequate for these high levels of stress. Two other types of glue were used for this test and the adhesion between the glue and the coating were even lower.

3. CONCLUSIONS

The results presented show that there is much more to study on the evaluation of pipeline coatings performance.

The standards aiming to evaluate coating flexibility were similarly designed but presenting differences on the methods. The BS EN 10290:2002 [38] includes the information about a possible gap between the mandrel and the coupon when bending. This effect was easily observed on the tests performed using AS/NZS 3862:2002 [33], where clamps were applied to solve the issue. The test can certainly bring good information about the coating flexibility but must be carefully done in order to
avoid or solve such issues and therefore an improvements on the information included in the standards should be considered.

For the CDR tests, the standard AS 4352-2005 [39] was used and the results showed some variation between samples. This effect might be due to variations on the coating thickness and on the coating behaviour itself. Considering that previous work using the ASTM G8-96 [35] and ASTM G42-11 [36] found high variation between samples, it can be considered that the variation is a result of the chemical/mechanical process itself. On the other hand, as all the various FBE coatings tested were evaluated as grade A, the rating do not discern between coatings. All coating type/thicknesses provided good resistance to cathodic disbondment but a more refined grade system could bring more useful information about the best types. Additionally, one coating type/thickness presented no disbonding.

The adhesion test performed showed that there is an effect on the cohesion when the samples are submitted to a hot wet immersion to absorb water and there is a variation in the glue adhesion performance.

4. ACKNOWLEDGMENTS

This work was funded by the Energy Pipelines CRC, supported through the Australian Government’s Cooperative Research Centres Program. The funding and in-kind support from the Australian Pipeline Industry Association Research and Standards Committee (APIA RSC) is gratefully acknowledge.

5. REFERENCES


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6. AUTHOR DETAILS

D Abreu is the research engineer coordinating the National Facility for Pipeline Coating Assessment (NFFPCA) at Deakin University. He has 11 years experience on the research and development of engineering products and reliability tests. He is collaborating with the corrosion group at Deakin University for pipeline coating testing and research supported by the Energy Pipeline Cooperative Research Centre (EPCRC) projects.

Dr M Tan’s principal teaching and research interests are in corrosion science and engineering and their applications for enhancing the reliability and durability of civil and industrial infrastructures. He contributed to electrochemical methods for corrosion testing, monitoring and prediction and corrosion inhibitor and anti-corrosion coating research. He is the author of over 130 publications and a book entitled ‘Heterogeneous Electrode Processes and Localized Corrosion’ (2012 John Wiley & Sons). He is the project leader of some EPCRC projects.
A Bryson is the Integrity Manager, East Coast Grid, of APA Group, Australia’s largest natural gas infrastructure business. He has over 30 years experience in corrosion protection of oil and gas pipelines. He is the lead industry advisor on the cathodic protection and coatings research projects of the EPCRC and a member of various standards committees.

A Fletcher is the Principal Materials Technologist for Pentair. He has over 30 years experience in coatings application, research, testing and specification on water, oil and gas industry pipelines. He is a member of the APIA RSC and is a Lead Industry Advisor to the EPCRC.

G Cope is a consultant with Geoff Cope & Associates Pty Ltd. He has over 40 years experience in pipeline cathodic protection and coatings, and has been engaged in various research and standards activities throughout his career. He is a member of the APIA Research & Standards Committee and an Industry Advisor to the EPCRC.

Grant Pearson has been formally retired since 2005 although conducts occasional commercial consulting work on materials science, chemical and metallurgical concerns and is presently also a student at Monash University (Materials Engineering). He has been variously an Experimental Officer at Institute of Materials Research, Metallurgy Dept. University of Melbourne, the Laboratory Manager of Rocla Concrete Pipes and also Laboratory Manager at Mount Carbine Wolfram in Northern Queensland. Most recently he was Senior Scientist (metallurgist, analyst) at the Scientific Services Laboratory (Gas Technology Services) of the Gas and Fuel Corporation Of Victoria until its disaggregation early in the 2000’s. He is an Industry Advisor to the EPCRC.