

# FEELING THE HEAT

ROBERT BUCHANAN, CANUSA-CPS, CANADA, PRESENTS A DETAILED ANALYSIS OF COATINGS DESIGNED FOR HIGH OPERATING TEMPERATURE PIPELINES AND THE TEST METHODS USED TO EVALUATE THEM.

High performance corrosion protection coatings for pipelines are constantly being required to perform on increasingly high operating temperature pipelines. In addition to high operating temperatures, these pipeline coatings must withstand tough construction conditions, since many of the major oil and gas discoveries are in remote fields located in developing countries.

Recent advancements in epoxy and multi-layer polyolefin coating materials have created coating systems that are pushing traditional temperature ceilings and are tough enough to survive the construction phase.

However, in the absence of industry wide consensus standards and because there are only a few owner specifications for high operating temperature pipeline coatings, each manufacturer or supplier of coating products for high temperature pipelines will make claims based on a variety of test parameters. Additionally, the requirement for high temperature pipeline coatings is relatively new and little history of performance data is available.

Because of this, a NACE task group was formed to

investigate the requirements for high operating temperature pipeline coatings and it has a huge challenge ahead of it. At the recent BHR conference on pipeline coatings<sup>1</sup>, several papers were presented but none could definitively state what was important to long term performance.

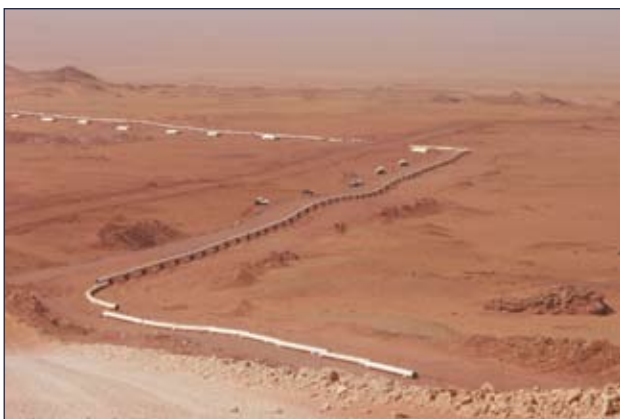
## PIPELINE COATINGS

The development of pipeline coatings over the past 65 years followed the natural progression of technology development required as a result of environmental, economic or performance pressures. The technologies in the mid 1900s were generally well suited for pipelines operating up to 50 °C (122 °F). Until recently, maximum operating temperatures were in the 80 °C (176 °F) range and the coatings, again, met that need well. Pipelines today are required to operate at temperatures greater than 100 °C (212 °F) and fast approaching 150 °C (302 °F). At these temperatures, the traditional methods of determining fitness for purpose are quite different than previously used.

Figure 1. High temperature pipeline construction.



Technology/product	Max operating temp.	Prime properties
FBE (standard)	65 °C	T <sub>g</sub> (DSC) Water absorption
FBE (high temp)	120 – 140 °C	T <sub>g</sub> (DSC) Water absorption
Dual layer FBE	130 °C	T <sub>g</sub> (DSC) Water absorption
Liquid epoxy (100% Solids)	150 °C	Cathodic Disbondment (CD)
3 layer polyethylene	90 °C <sup>3</sup>	Shear indentation, CD
3 layer polypropylene	130 °C	Adhesion, indentation, CD



**Figure 2. High temperature pipeline construction in a remote environment.**



**Figure 3. High temperature CD apparatus. Photo courtesy of ShawCor Ltd.**

Additionally, as with mainline coatings, the field joint coating must perform in-service at a level that meets the requirement of the line. Although the joint coating does not need to withstand the rigours of transportation to the right-of-way, it does need to maintain integrity for the life cycle of the line. The challenge with field joints is threefold:

- It must be able to be installed under field conditions in many different climates and under virtually any weather condition, as opposed to the in-plant conditions of applying the coating.<sup>2</sup>
- It is the last construction process before the pipeline is lowered into the ditch or let off of the back of a lay barge. This means that the contractor is generally rushed to do this operation.
- The contractor often uses their own labour for the operation or, alternatively, subcontracts the operation to a service company. The latter is potentially more expensive and is not therefore always the preferred option.

## PERFORMANCE PARAMETERS

Typical industry wide performance standards and pipeline owner specifications include a number of performance measures with test methods specified. Many of the test methods are run at ambient temperature, at a specified maximum temperature or at “pipeline operating” temperature, i.e. T<sub>max</sub>. However, limitations in equipment capacity or limitations in the test parameters may result in the testing being carried out at the maximum capability of the equipment or in a “modified” regime that may not represent in-service conditions.

Additionally, manufacturers of coatings and/or raw materials for coatings will make claims of performance based on material properties, ambient test methods and one or two modified test methods. The question remains, what testing is required to determine the in-service high temperature performance of a coating?

Table 1 is a summary of coating technologies with respective typical maximum pipeline operating temperatures listed and the traditional prime driver for the rated operating temperature identified.

Table 2 is a summary of a number of material properties and potential test methods with parameters and relevance noted based on a review of the draft ISO<sup>4</sup> standard and existing ASTM<sup>5</sup>, French<sup>6</sup>, Canadian<sup>7</sup> and European<sup>8,9</sup> coatings standards and test methods plus various pipeline owner specifications<sup>10,11</sup>.

This article describes a number of coating technologies and this inevitably invites classic “apples & oranges” comparisons. The coating technology must be considered with respect to what is important for a specific pipeline project. As an example, epoxy materials may be more suitable for dry non-aggressive soil conditions, whereas for wet or particularly aggressive soils, multi-layer coatings should be considered. Onshore versus offshore; the location of the pipeline; and construction conditions; would also have an impact on the choice of coating system.

The decision needs to be made by informed specifiers and may require consultation with specialists or with the manufacturers of the coating products.<sup>12</sup>

## TEST METHODS

Focusing on some key attributes and commonly accepted test methods, the following is a discussion about the merits and parameters that are important for select test methods.

## ADHESION

For multi-layer coatings a peel adhesion test is common. Although it does not represent conditions that a coating may experience in-service, it is a good indication of how well a coating has been applied by determining how well it is bonded to the substrate. This in turn represents the quality and tenacity of a coating, especially if the test is carried out at T<sub>max</sub>. Various test methods may be specified, with the main differences being angle of pull and speed of pull. Commonly the angle is 90° to the pipe surface with the speed varying from 10 - 300 mm/min. Higher speeds will generally yield higher values. A hanging weight test is also sometimes specified.

For epoxy coatings, a peel test is not practical but a pull-off adhesion test can be performed as an alternative. For thin systems such as epoxies, this test is more important as a quality control measure in determining

if the coating was applied correctly. However, experienced lab technicians and notations within international standards<sup>13,14</sup> caution that this seemingly simple test can yield widely diverse results depending on the apparatus and methodology used.

A third test that can be performed for either type of coating is a hot water immersion followed by a crosscut adhesion test.<sup>15</sup> Although it will not provide a numerical value measured on an instrument, it is a good indicator of a coating's performance after subjecting it to water immersion or elevated temperatures. For example, liquid based coatings will initially show reasonable adhesion to an abraded polyolefin surface, but after hot water immersion, the coating will readily peel off.<sup>16</sup> Secondly, when an applied epoxy is heated above its  $T_g$  (see Glass Transition notes below) its hardness is reduced and it may readily peel off of a steel substrate.

### CATHODIC DISBONDMENT (CD)

A CD test measures a coating's resistance to disbondment at a holiday in the coating and simulates an operating pipeline subjected to cathodic protection. Pipeline engineers strive for lowest possible levels of disbondment at damages in the coating and tend to put a lot of stock in this test.

The challenge is that the test has many variables and there are a number of test methods and methodologies available. Additionally, various specs may stipulate parameters for some of the variables while others do not. A CD number of 10 mm radius, for example, may be good in one test but not in another. There have been many technical papers written on this subject and here follows a brief discussion of key issues.

One of the realities of a CD test is that the electrolyte used to promote disbondment is water based and, as such, it boils at approximately 100 °C and evaporates readily at elevated temperatures. Some methods, such as high pressure autoclaves set to achieve an electrolyte temperature of 150 °C, simply do not replicate in-service conditions. Therefore, by specifying a CD test at  $T_{max}$ , a 120 °C test is virtually impossible without setting a test regime that represents how cathodic protection affects an in-service

pipeline. Various schools of thought include setting the relative temperatures of the electrolyte and test specimen at various levels such as in Table 3.

As mentioned, several papers<sup>17,18</sup> have been written on the subject and the general conclusion is as noted above. Therefore, a reasonable assumption would be to run ambient temperature CD tests according to regime A and elevated temperature tests according to regime B. The detail then involves how to consistently maintain temperatures and replenish and recycle the electrolyte so that it does not become overly concentrated and corrosive in itself.

As a final thought on elevated temperature CD testing, some specifications put more or less weight on this. Results are available that demonstrate that a CD test carried out above 95 °C is not practical, nor representative of in-service conditions and that CD testing at lower temperatures, i.e., 65 °C, can actually be more severe.<sup>18</sup>

### HARDNESS AND INDENTATION TESTING

These are measures of a coating's toughness and resistance to damage during construction and while in-service, especially where aggressive soils and backfill materials are present. Most coatings are thermoplastic

**Table 3. CD test methodologies.**

Test regime	Electrolyte	Specimen	Expected result
A (ASTM G8)	@ test temperature ( $\leq 95$ °C)	@ test temperature ( $\leq 95$ °C)	Representative of ambient conditions
B (ShawCor)*	@ test temperature (95 °C max.)	@ test temperature	Representative of high temperature conditions
C (NF A 49-711)	@ ambient temperature	@ test temperature	Representative of offshore conditions
D (Dow Chemical)	@ test temperature	@ ambient temperature	Not applicable to pipelines

\* similar to CSA Z245.20

**Table 2. Example requirements**

Material property	Test method	Typical requirement	Relevance
Impact resistance at ambient & low temp.	ASTM G14 / CSA DIN 30 670	Epoxy - 1.5J Polyolefin - 3-5J/mm	Durability
Shore D hardness at ambient	ASTM D2240	Epoxy - 90 Polyolefin <sup>A</sup> - 50 - 60	Durability/qualitative
Indentation resistance at $T_{max}$	ASTM G17 NF A 49-711	0.4 mm	Durability
Adhesion (peel) at ambient & $T_{max}$ (Polyolefins) <sup>A</sup>	ASTM D1000 EN 12068 / DIN 30 672	20-40N/cm 5N/cm	Qualitative/durability
Adhesion (pull off) (Epoxy) <sup>B</sup>	ASTM D4541	Liquid >14MPa FBE > 21MPa	Qualitative/durability
Lap shear strength at ambient & $T_{max}$	ASTM D1002 EN 12068 / DIN 30 672	17 N/cm <sup>2</sup> 5 N/cm <sup>2</sup>	In-service performance
Cathodic Disbondment at ambient & $T_{max}$	ASTM G8 (28 days) CSA Z245.20 (28 days)	<10 mm radius <15 mm radius	In-service performance
Thermal ageing (Polyolefin Backing)	EN 12068 / DIN 30 670	500h @ 150 °C >70% of original	Service life
Vicat SP (adhesive)	ASTM D1525	80 - 140 °C <sup>A</sup>	In-service performance
Melting point	ASTM D3418	110 - 165 °C <sup>A</sup>	In-service performance
Glass Transition ( $T_g$ ) <sup>C</sup> (mid points)	ASTM D3418 (DSC) ASTM D 5026 (DMA)	141 °C and 166 °C 118 °C and 136 °C	In-service performance

**A. Range for polyethylene and polypropylene.**  
**B. FBE and 2 component 100% solids liquid epoxy.**  
**C. For high operating temperature FBE, two grades.**

in nature, even those considered as thermosets, since both polyolefin and epoxy coatings will soften as they heat up. Therefore an ambient temperature hardness measure or an indentation test will provide a baseline, but a test at  $T_{max}$  will provide an insight into how the coating will perform during the operational life of the pipeline.

## GLASS TRANSITION ( $T_g$ )

This is a measure for epoxy coatings either used as standalone corrosion coatings or as primers in a 3 layer polyolefin coating. It is quite complex and can be used to determine how to evaluate a coating under other tests as temperatures approach and surpass the material's  $T_g$ .

The  $T_g$  can also be an indicator of the maximum service temperature of a coating and a rule of thumb was that  $T_{max}$  should be 10 - 20 °C below the specific product's  $T_g$ . However, epoxy coatings have been more recently rated higher than their  $T_g$  considering other factors such as: modulus at temperatures beyond the  $T_g$ , and the capacity of the coating to resist diffusion of corrosion species such as moisture, oxygen, chlorides, etc. For example, in a 3 layer coating the epoxy is protected from damage and resists diffusion by the adhesive and polyolefin and thus will perform at higher temperatures. Likewise, the theory for dual layer FBE systems is that the top epoxy layer protects the underlying epoxy and the same assumption applies.

The exception to the rule seems to be 2 component, 100% solids liquid epoxy products that some manufacturers indicate can be used on pipelines operating up to 150 °C. This is seemingly based on CD testing, but little or no long term performance data is available and more work is required to prove these systems.

## SOFTENING POINT AND MELTING POINT

For lower operating temperature coatings, the adhesives are relatively simple with the softening point measured using the ASTM E28 "ring & ball" method. For higher operating temperature coatings such as 3 layer polyethylene and 3 layer polypropylene, the adhesives can be tested to determine their Vicat softening point and melting points. These become measures of where the adhesives lose strength, which contributes to the determination of  $T_{max}$ .

## ARTIFICIAL AGEING

Heat ageing is an effective evaluation for coatings with a common requirement to retain certain levels of performance or measure levels of degradation after exposure to elevated temperature, moisture and time. For epoxy systems, some of the key issues are thermal stability, hygro-thermal stability and diffusion characteristics, whereas for multi-layer polyolefin systems, retention of properties through Arrhenius ageing studies may be more suited.

## MAXIMUM OPERATING TEMPERATURE ( $T_{max}$ )

How do these evaluation methods contribute to the determination of  $T_{max}$ ? Many owner specifications that were developed with maximum operating temperatures of 80 °C (176 °F) in mind may still be valid for higher temperatures. However, the test methodology must be factored in along with an understanding of the characteristics of the technology chosen.

Thermal ageing and relative ageing studies are good indicators of long term performance, but correlation between hours of ageing and years of service life are difficult to make considering that exposure and operational variables will be different for every pipeline.

Physical and mechanical performance properties evaluated at  $T_{max}$  can ensure that the integrity of the coating will be maintained.

Cathodic disbondment testing is certainly a factor, but must not be the only parameter in determining a coating's long term performance expectations.



Figure 4. Pipeline construction in aggressive conditions.



Figure 5. Aggressive construction conditions.



Figure 6. Terrain creates construction challenges.

Determination of  $T_{max}$  then comes from informed evaluation of the construction of the system and a variety of test results.

## TECHNOLOGY DEVELOPMENTS

The common and more recent technologies for high operating temperature pipelines are 3 layer polyethylene; 3 layer polypropylene; dual layer fusion bonded epoxy; and liquid 2 component, 100% solids epoxies. Upon reviewing the various manufacturers' published literature, much of the data supplied does not clearly support the operating temperatures at which these coatings are being asked to perform. Some manufacturers, however, tend to take a conservative approach and provide information to pipeline specifiers who are asked to make their own decisions based on testing according to their own specifications.

Ultimately, the mainline coating must resist damage from handling, transportation, laying and in-service conditions at  $T_{max}$ . The field joint coating must also be compatible with the plant applied coating and resist damage from in-service conditions.

## FIELD JOINTS

Considering the field joint coating system, it is important to specify systems that are fully compatible with the mainline coating.

For 3 layer polyethylene, cross linked polyethylene heat shrinkable sleeves have been the common field joint choice for a variety of reasons. For multi-layer polypropylene, there are several field joint options on the market including flame spray, multi-layer tape and polypropylene heat shrinkable sleeves that need to be evaluated to exceed the performance specifications for the mainline coating system.

For epoxy systems such as liquid epoxy and dual layer FBE, field joints using like technologies that can be effectively applied in the field are critical. High temperature heat shrinkable sleeves are also available that are proven to conform to owner specifications.

## CONCLUSION

This article is intended as an information piece with the goal to raise awareness of an industry-wide need to standardise how coatings are evaluated for high temperature service. The hope is that pipeline owners and specifiers take into account some of the information as they develop their specifications.

Of note, NACE recently formed a task group to develop a NACE document relative to Elevated Temperature Pipeline Coatings, which is currently under development. There is also a NACE task group looking at CD test methodologies as a result of the issues noted earlier.

Pipeline specifiers and/or owner groups have developed specifications over the years and are generally comfortable that the requirements that they have laid out adequately represent their respective

needs. These specs do not need to change significantly for high temperature service pipelines, but the methodology of how the testing and evaluation is performed does need to be considered. ●●●

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