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Evaluation of Anticorrosion Coatings For High Temperature Service

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ABSTRACT

The continued development of the oil sands in northern Alberta Canada has led to increased use of thermal recovery methods such as Steam Assisted Gravity Drainage (SAG-D) for extraction of the bitumen from deep reserves. The transportation of this heated bitumen to the processing facilities generally requires the use of insulated pipes capable of withstanding internal service temperatures as high as 150°C. Based on existing Canadian regulations, it has been necessary to use anticorrosion coatings under the thermal insulation on buried pipelines intended for petrochemical service. Traditionally, anticorrosion coatings such as cold applied tapes, 2 or 3 layer polyethylene systems, and also epoxy coatings have been available for insulated pipelines operating at temperatures of up to 110°C. The introduction of new heat resistant insulating foams capable of withstanding much higher operating temperatures of at least 150°C has led to the need to also identify compatible anticorrosion coatings. One such material that has been successfully qualified for use with high temperature insulation systems is an epoxy-based coating. In order to evaluate this coating, a variety of methods were used which included; elevated temperature cathodic disbondment testing, thermal gravimetric analysis (TGA), dynamic mechanical analysis (DMA), accelerated heat ageing, and also standard coatings tests specified by the Canadian standard CSA Z245.20. The results of this investigation support the recommendation that the selected coating can withstand operating temperatures of at least 150°C for the expected 30-year life in a typical insulated pipeline.

KEYWORDS

Arrhenius, Cathodic Disbondment, CCOT, FBE, Glass Transition, Insulation, Polyurethane Foam, Thermal Ageing

INTRODUCTION

Ongoing development of the oil sands in northern Alberta Canada has led to increased use of thermal recovery methods such as Steam Assisted Gravity Drainage (SAG-D) for extraction of the bitumen from deep reserves. This heavy crude oil has a high viscosity at standard temperatures and requires considerable heat to allow it to flow. Thus, the transportation of the bitumen to the processing facilities often requires the use of buried insulated pipes capable of withstanding internal service temperatures as high as 150^{o}C. In order to reduce the likelihood of corrosion and potential rupture of the pipeline, it is typically necessary to apply anticorrosion coatings to the external surface of the steel carrier pipe for use underneath the thermal insulation. Traditionally, anticorrosion coatings such as cold applied tapes, 2 or 3 layer polyethylene systems, and also epoxy coatings have been used for this purpose on insulated pipelines operating at conventional temperatures of typically less than 110^{o}C. The recent development of heat resistant insulating polyurethane foams capable of withstanding higher temperatures of at least 150^{o}C has created the need for qualifying compatible anticorrosion coatings that can also remain stable for the expected service life of the pipeline that is typically specified as up to 30 years. This paper discusses the various methodologies that were used to evaluate and qualify one such coating for the use in the new insulation systems.

COMPONENTS OF THE INSULATION COATING SYSTEM

The coating materials typically used on insulated pipes for buried service include the following (see Figure 1):

1. Anticorrosion coating.
2. Polyurethane foam thermal insulation layer, and
3. Polyethylene protective topcoat / outer jacket layer.

Anticorrosion Coating

The use of anticorrosion coatings applied to the external diameter of the steel carrier pipe is now commonly performed for most factory applied pipeline insulation systems produced in North America. In the past, foam insulation materials had often been applied directly to bare steel pipes, but due to the potential corrosion and rupture of the pipelines as a result of water ingress through damaged outer jacket or field joint coatings, such practices were mostly abandoned, particularly with the introduction of effective coatings such as cold applied tapes and fusion bonded epoxy (FBE) coatings which provided good protection and which could easily be applied in the factory. However, even the more heat resistant coatings like FBE were often only rated for use at temperatures of less than 110^{o}C, as there were concerns about the long-term performance of these materials at higher temperatures. This was particularly the case for those used above their glass transition (Tg) temperature. In general terms, the value for Tg plays a key role for influencing the performance of a polymeric material in a number of ways, as listed here[1]:

Below Tg
- Molecular motion is restricted.
- Films are hard and glassy, tending to break rather than deform under stress and have high internal stress.
- Films have reduced impact resistance and flexibility, and poor formability.
- Chemical resistance is maximized.

Above Tg
- Molecular motion is greatly increased.
- Films are soft, rubbery and relaxed and deform under stress.
- Films have good impact resistance, flexibility and formability.
- Permeability increases.

It is typically desirable to have the Tg value of the anticorrosion layer to be equal too or greater than the operating temperature of the pipeline in order to ensure that the coating remains mechanically stable at high temperatures and also retains its maximum corrosion resistance. In the coatings industry there had often been a reluctance to exceed the Tg value of the coatings due to concerns of a failure and so this remained a limiting factor in the development of higher temperature insulation systems. In addition, qualification testing of these coatings rarely employed any life prediction methodologies that could be used to clearly demonstrate their effectiveness for long periods at high temperatures. Instead, the evaluations were often done for relatively short durations such as 28-days with testing temperatures being limited by the boiling point of water used for the corrosion resistance tests.

In recent years there had been work performed by several coating manufacturers to develop FBE coatings capable of operating at the much higher temperatures being required for many of the insulated pipelines used for offshore/marine service. Much of this research was focused on using the FBE solely as a primer that would allow the specialized insulation / water barrier materials such as solid or foamed polypropylene to be bonded to the steel pipe. Some of the resulting insulation coating systems produced using this concept were subsequently rated for operating temperatures as high as 155^{o}C[2]. Unfortunately, the relatively poor low temperature flexibility and decreased corrosion resistance compared to conventional stand-alone FBE coatings made some of these new coatings less attractive for use on insulated pipes for onshore buried service in Canada. This was due to these pipelines typically being installed in the winter at ambient temperatures of -30^{o}C or lower, and also because it was generally required to demonstrate that the FBE was effective as a corrosion barrier on its own, in accordance with the CSA Z245.20 fusion bonded epoxy standard.

New developments in the formulation of the FBE coatings has resulted in materials with higher Tg's that not only have improved flexibility at low temperatures, but also better resistance to direct exposure to water or other corrosive conditions. Demonstrating that these coatings could maintain adequate performance for use on insulated pipelines operating for at least 30 years at temperatures as high as 150^{o}C remained a challenge and thus a number of different methodologies were investigated for qualifying the FBE coating. These included:

1. Evaluation of the glass transition temperature (Tg) by differential scanning calorimetry (DSC) and dynamic mechanical analysis (DMA).
2. Testing for decomposition kinetics by thermogravimetric analysis (TGA)
3. Accelerated heat ageing of anticorrosion coated insulated pipes in conjunction with shear strength testing.
4. Cathodic disbondment and hot water adhesion testing of thermally aged epoxy coatings.

PROCEDURES

In order to conduct this study, test pieces and other material specimens of the FBE coating were produced in the following configurations:

- Steel pipes coated in a factory with the FBE coating only.
- Factory coated insulated pipes consisting of; FBE anticorrosion coating, polyurethane foam insulation, and extruded polyethylene outer jacket.
- Steel panels coated in the laboratory with the FBE material.
- Free-film specimens of the FBE coating.

FBE and Insulation Coated Pipes

These were produced in a commercial coating factory by first applying the FBE coating onto abrasive blasted and preheated 114.3mm (4.5") OD steel pipes at a nominal thickness of 400μm (15mils). The pipes were subsequently over-coated with approximately 50mm of heat resistant spray-applied polyurethane foam insulation and then extrusion coated with a 5mm thick high-density polyethylene jacket to provide mechanical and environmental protection. All coating application processes were performed using standard production techniques.

Laboratory Coated Panels

Mild steel panels were washed with detergent to remove oil and grease and then abrasive blasted using G25 steel grit. The panels were then preheated in a laboratory oven prior to application of the epoxy powder coating to a nominal thickness of 400μm using electrostatic powder spray equipment. Each coated panel was then returned to the oven for several minutes to complete the curing reaction before being finally quenched in water.

Free Film Material Samples

Specimens of the bulk epoxy coating not attached to a substrate were prepared by applying the FBE onto a preheated non-stick coated steel pan and then curing and quenching the material in a manner similar to that used for the laboratory coated panels. Once quenched, the free film sheet could easily be removed from the pan.

GLASS TRANSITION TEMPERATURE

For the initial selection of the anticorrosion coating, it was determined that the material should ideally posses a Tg value equal to, or greater than the proposed maximum 150^{o}C operating temperature for a typical insulated hot bitumen pipeline. Although the Tg values of FBE coatings are most often measured using differential scanning calorimeter (DSC) instruments, this is an indirect correlation established from thermal events such as heat flow rather than being measured directly as a physical property. Thus, to more accurately determine this value, a dynamic mechanical analysis (DMA) instrument was used. This device applied an oscillating strain to the specimen while it was exposed to temperature ramping conditions and then measured the physical response of the material in relation to the temperature. A 25mm long x 12.5mm wide x 0.8mm thick test specimen was prepared from a flat sheet of free-film material. A ramping rate of 2^{o}C/minute was used from 25^{o}C to a final temperature of 200^{o}C and the sample was tested in the torsion rectangular position in an air atmosphere at a frequency of 1 rad/s. The Tg value was determined from the peak tan-delta (tan δ) value, which is the ratio of the loss modulus to the storage modulus for the material.

For comparison purposes and also for testing the FBE from the pipes subjected to thermal ageing, Tg determinations were also made using a DSC instrument. Unlike the DMA that typically requires a free-film sample of the material, the DSC only needs a small amount (<10mg) of shavings or chips of the coating for the analysis which makes it better suited as a QC tool since the coating can be tested in the as-applied condition from the pipes instead of as a separately prepared sample. This testing was based on standard procedures outlined in section 12.7 of CSA Z245.20.

Results and Discussion

It was determined by the DMA testing that the Tg temperature of the epoxy was 150.09^{o}C (Figure 2). This result was approximately 7^{o}C higher than the Tg of 143.05^{o}C obtained by the DSC instrument method from chips removed from a coated pipe (Figure 3).

THERMAL LIFE PREDICTION BY TGA

Testing of the epoxy coating using a thermogravimetric analysis (TGA) instrument was conducted to provide an initial assessment of the material's stability at high temperatures. Unlike larger scale methods such as the accelerated heat ageing of coated pipes which could take many months to complete, TGA testing for the purpose of 'life prediction' could be conducted in as short a period as one or two days. It should be noted that this analysis is based on making certain assumptions about the material; in particular the significance of certain amount of weight loss on the performance of the coating; and further assuming that all of the chemical reactions would be of the first order variety. Thus, it can be a useful tool for providing additional information for screening or comparing materials, but likely should not be used as the sole means of predicting the actual in-service performance of a material.

The TGA instrument consists of a highly sensitive weighing balance situated within a temperature-controlled chamber. Small specimens of the FBE prepared as shavings from free-film samples were placed onto the weighing balance pans and heated at various ramping rates while under precisely controlled flowing atmospheres of either air or high purity nitrogen gas. The instrument automatically recorded the weight losses of the specimens versus the temperature as it was ramped.

The activation energy of the epoxy was calculated by conducting a series of experiments under different thermal conditions. Temperature ramping rates of 1, 2, 4, and 8^{o}C/minute were used for testing in both air and pure nitrogen environments and the weight losses occurring during the heating cycle were plotted on a graph based on a 5% weight loss at a 90% conversion level. The activation energy was calculated per the procedures outlined in ASTM E 1641.[3] An Arrhenius plot was then constructed from on the logarithm of the heating rate versus the reciprocal of the absolute temperature for the chosen conversion level, and the expected life of the epoxy was determined by plotting the logarithm of lifetime in years versus the reciprocal of the absolute temperature in kelvin.

Results and Discussion

The TGA testing in nitrogen indicated that the epoxy should be able to withstand exposure temperatures upwards of 140^{o}C for over 30 years, with temperatures of even as high as 150^{o}C still giving an expected life of over 27 years (See Table 1). Testing in air gave a considerably lower calculated lifetime, however based on examination of the epoxy coatings thermally aged under insulation on the pipes used for the CCOT testing, it is believed that the conditions of nitrogen exposure would be most comparable to the relatively oxygen free environment created when the coating is overlaid with a bonded insulating foam, that is typically blown with a hydrocarbon gas.

QUALIFICATION OF UNAGED FBE COATING

Qualification to CSA Z245.20-06

In 2006, a new edition of Canadian Standards Association document, CSA Z245.20 'External Fusion Bond Epoxy Coatings for Steel Pipe' was issued which included within its scope FBE pipeline coatings for higher service temperature. These types of coatings are defined in the standard as "single-layer FBE with a glass transition temperature greater than 110^{o}C"[4]. Therefore, prior to conducting the CCOT ageing, the coating was first qualified in the new (unaged) condition in accordance to this standard to ensure it met these base requirements. Such testing is required by to comply with section 9.2.7 of CSA Z662 'Oil and Gas Pipeline Systems' which is used as the main reference document for the material and installation requirements for all such pipelines in Canada.

The qualification testing of the FBE coating was conducted on both laboratory coated plates as well as factory-coated pipes and based on the requirements outlined in Table 2 of the CSA Z245.20 standard. The coating thickness was a nominal 400μm (15mils).

Results and Discussion

The results of the qualification testing are shown in Table 2. The FBE coating was found to meet all of the requirements of the CSA Z245.20 standard for system 1B.

High Temperature Cathodic Disbondment Testing

Cathodic disbondment (CD) testing of the FBE at 150°C was used to evaluate the resistance of the coating to disbonding under temperature conditions similar to those being considered for operation of the high temperature pipelines. Although for most buried-service insulated pipes the external surface of the steel carrier pipe is unlikely to come into contact with large amounts of water, cathodic protection is still used in many cases as a contingency.

Test panels consisting of a nominal 400μm thick layer of the FBE coating applied to flat steel were used for this study. A 3.2mm diameter drill was used to expose the steel substrate at the center of each panel simulating a perforation of the coating (also known as a 'holiday'). Small sections of plastic pipe with a 75mm ID were then centered over the holiday and bonded to the panels using a silicone adhesive. These cups were then filled with approximately 300ml of a 3% sodium chloride salt solution and the specimens were placed onto a test apparatus that heated the underside of the test piece with a temperature controlled oil bath. A non-metallic lid with an attached platinum wire electrode was placed onto each of the cups with the electrode immersed in the salt solution. The electrodes as well as a positive polarity connection attached to each test piece were connected to a voltage controlled direct current power supply and a calomel reference electrode was used to set the potential to the specified value.

The electrolyte for each specimen was continuously recirculated from a common larger temperature controlled reservoir maintained at 95°C throughout the testing period, and an automatic make up system was used to maintain the correct salt concentration. The panels were tested for durations of 28 and 56 days using a potential of –1.5V with the steel temperature of the coated panels being maintained at 150°C.

Results and Discussion

After the 28 days of testing at 150°C, the average disbondment radius values for the three specimens ranged from 5-7mm, while for panels exposed for 56 days they ranged from 10-15mm (Table 3). There is no direct reference to this particular equipment setup or testing temperature within the CSA Z245.20 standard, however the results for both exposure periods were well within the maximum 20mm disbondment radius permitted for 28-day testing at 95°C.

CALCULATED CONTINUOUS OPERATING TEMPERATURE (CCOT) OF INSULATION SYSTEM

Accelerated heat ageing of insulated pipes was conducted based on the procedures outlined in the European District Heating Insulated Pipe standard EN 253[5]. This document specifies a method referred to as Calculated Continuous Operating Temperature (CCOT) which is used for predicting the long term performance of the insulation coating based on shorter term testing conducted using internal pipe temperatures much higher than the actual intended service conditions. This procedure relies on using the Arrhenius equation for calculating the activation energy of a material by testing for chemical reactions occurring in relation to temperature. The activation energy is defined as the minimum amount of energy required to initiate a chemical change in a material, and determining this value allows one to predict the effects that heat and time will have on materials like the insulation or FBE coating. The Arrhenius equation can be expressed as:

$$K = Ae^{-E/RT} \qquad (1)$$

Where,

K= Rate constant
A = Pre exponential rate factor
e = Natural exponent
E = Activation energy
R= Gas constant
T= Temperature in K

This relationship may in turn be used to allow for predicting the time to failure as follows:[6]

$$Tf = Ae^{[\Delta H/KT]} \qquad (2)$$

Where,

Tf = Time to failure
A = Scaling / pre exponential rate factor
e = Natural exponent
ΔH = Activation energy
k = Boltzmann's gas constant of 8.617 x 10^{-5} eV/^{o}K (1.380,658 x 10^{-23} J/^{o}K)
T = Temperature in K.

The use of this principle for predicting the performance of materials at a particular temperature is also applicable to many other different testing methodologies and products.

Determining the thermal life of the insulated pipes was based on selecting a critical property, in this case the shear strength of the bond between the insulation to the FBE coated pipes, and testing for this value on pipes exposed to several different ageing temperatures which accelerated the thermally induced break down of this property over time. By using the much higher ageing temperatures, this permitted to greatly reduce the total testing time required. Based on similar previous work, the ageing temperatures selected for this study were: 180^{o}, 185^{o}, 188^{o} and 193^{o}C. Four insulated pipes, each approximately 6m long, and all produced under similar conditions in a pipe coating factory were used, with one pipe being tested per ageing temperature.

After the initial inspection and testing of the new (unaged) properties of the coating system, the pipes were each assembled into individually controlled flow loops that used pressurised hot water to raise the internal temperature of the pipes to the specified values. The pipes were each maintained at the required ageing temperature for varying periods of time at which point the internal water temperature was then temporarily lowered to 140^{o}C and a shear adhesion test was conducted on the coating system. This was performed by cutting into the entire coating to isolate a 100mm (4") wide ring section of the insulation exposed down to the surface of the pipe, then attaching a mechanical clamp to the test section and applying a tangential force using a controlled loading system equipped with load sensors. The force required to shear or break the bond of the insulation coating to the epoxy coated pipe was recorded and the shear stress calculated as the peak load divided by the surface area of the test section adjacent to the pipe surface. Once each test was completed, the pipe was reheated back to the original set point and the ageing continued until the next required testing interval.

The life for each accelerated ageing temperature was determined based on the time at which the floating mean shear strength value of the insulation coating first crossed the minimum specified acceptance value of 0.13Mpa (18.9psi) listed in the EN 253 standard. A plot was then constructed of the natural logarithm of the ageing time required to reach the acceptance value versus the reciprocal of the ageing temperature in units of K. By extrapolating from the ageing data points plotted on this graph, a prediction of the maximum continuous operating temperature was obtained based on the commonly required service life of 30 years. Because of this relationship, using a lower value for the service life such as 25 years would allow for a significant increase in the allowable operating temperature of the coating system, while using a higher operating temperature (or allowances for occasional temperature spikes) would in turn reduce its predicted life.

Results and Discussion

Testing of the insulated pipes determined that the coating system should be able to withstand 150^{o}C for 30 years of service life based on maintaining the minimum shear strength of 0.13Mpa (18.9 Psi) as shown in Figure 2. It should be noted that the foam insulation has considerably lower shear strength than the epoxy coating and so all failures typically occurred within the foam itself, as it would fracture cohesively leaving residual material bonded to the epoxy coating.

EVALUATION OF ACCELERATED AGED FBE COATING

Since the CCOT testing conducted on the insulation system required that the pipes remain intact for the entire ageing period, it was not possible to perform testing for corrosion resistance on these same samples at periodic intervals. Instead, it was decided to complete all the CCOT ageing and then section the pipes for the purpose of evaluating the coating in accordance to the procedures for cathodic disbondment and hot water soak (HWS) adhesion outlined in tables 2 and 4 of CSA Z245.20. Tests for flexibility and impact resistance etc were deemed unnecessary as these were of primary importance for installation of the new pipes rather than for in-service performance. Although the testing listed in the CSA standard is only required for newly produced and unaged coatings, it was decided that still meeting the requirements for corrosion resistance after the ageing period would demonstrate that the FBE had retained its effectiveness in protecting the pipe against any possible corrosion.

Specimens were cut from each of the four ageing temperature pipes and the bonded foam insulation was removed from the surface of the FBE coating as much as possible by carefully scraping with a sharp tool, avoiding damage to the coating. The coating specimens were then tested for:

- Cathodic disbondment (CD) resistance at 65^{o}C, -3.5V for 24 Hours,
- Hot water soak (HWS) adhesion for 24 hours at 75^{o}C,
- Glass transition (Tg) temperature.

The Tg values of the coating for each ageing condition were measured using a differential scanning calorimeter (DSC) instrument, as even though it might not posses the accuracy of the DMA, it was better suited for testing the small coating chips removed from the pipe sections and could thus still give a relative comparison for any changes in the Tg value.

Results and Discussion

See Table 3. From the testing of the aged panels it was determined that all of the specimens met the CD and HWS requirements of both Tables 2 and 4 for new condition coatings (system 1B) according to the CSA Z245.20 standard. The average disbondment radii for the CD testing ranged from 2.5-3.5mm, while the HWS results had ratings of 1. Both tests met the maximum CSA acceptance criteria of 6.5mm disbondment radius for the CD testing and the maximum number 3 rating allowed for the HWS evaluation (Figure 6).

The glass transition temperature of the coating measured by the DSC method was determined to increase from an unaged value of approximately 143^{o}C, to nearly 155^{o}C after the CCOT ageing.

CONCLUSIONS

Glass Transition (Tg) Temperature

The evaluation for the Tg of the unaged epoxy using a DMA instrument showed that the material possessed a Tg of approximately 150^{o}C based on the peak of the tan delta plot. This result was approximately 7°C higher than for what was obtained by the less sensitive DSC method, however differences in sample preparation and thermal history could play a role in affecting the results. Based on the DMA testing, the coating could be rated for use to at least 150^{o}C, however excursions beyond this value do not appear to be detrimental based on the CCOT ageing study.

Thermal Life Prediction by TGA

The life prediction determinations based on the TGA analysis indicated that the epoxy coating could withstand greater than 30 years at 140^{o}C and over 27 years at close to 150^{o}C. It should be noted that the weight loss criteria of 5% used for the life prediction calculations was selected from previous materials testing parameters, and so using a different value for weight loss could significantly affect the predicted life obtained by this test. The correlation from TGA testing to actual operating conditions has not yet been determined for this material.

Qualification of Unaged FBE Coating

The initial qualification testing of the new condition FBE in accordance to CSA Z245.20 showed that the coating met the requirements of this standard for system 1B. This coating therefore meets the requirements for stand-alone service which is likely more severe compared to the relatively protected conditions when used under foam insulation where there will be much less chance of exposure to water and other corrosive conditions.

High Temperature Cathodic Disbondment Testing

Cathodic disbondment testing with a substrate temperature of 150^{o}C for 28 and 56 days indicated that the coating had good performance for these severe test conditions. Even after 56 days of testing, the average disbondment radii of 10, 14, and 15mm on each of the three specimens were well within the maximum allowed value of 20mm for the 28 day testing at 95^{o}C and –1.5V based on CSA Z245.20 (see Table 4).

Calculated Continuous Operating Temperature of Insulation System

From the shear strength testing of the four pipes subjected to accelerated heat ageing, it was determined that the insulation system comprised of the FBE coating and polyurethane foam could be rated for an operating temperature of at least 150^{o}C for 30 years of service. Even though the heat ageing was conducted at temperatures well above the glass transition temperature of the FBE, there did not appear to be any detrimental effects on this coating as the shear failures occurred within the foam material itself and not in the FBE. Subsequent performance testing of the FBE supported this conclusion.

Evaluation of Accelerated Aged FBE Coating

Testing the FBE coating from the CCOT aged pipes for 24-hour cathodic disbondment and hot water adhesion in accordance with the CSA Z245.20 standard demonstrated that the coating retained very good resistance to these conditions even after being aged well in excess of the maximum life for the foam insulation. Both the CD and HWS results for the epoxy were within the maximum acceptance criteria rating of 6.5mm for the CD testing and a maximum rating of 3 for the HWS evaluation according to the CSA Z245 standard.

SUMMARY

An FBE coating material with a glass transition temperature of 150^{o}C that complies with the requirements of CSA Z2455.20 for system 1B has been identified and qualified for a continuous service life of at least 30 years at 150^{o}C when used under predominately dry service conditions as part of an insulation coating system. Additional cathodic disbondment and hot water adhesion testing of the heat aged FBE coating has further demonstrated that the material can retain its effectiveness as a corrosion barrier even after prolonged exposure to elevated temperature conditions in excess of the rated life of the insulation foam.

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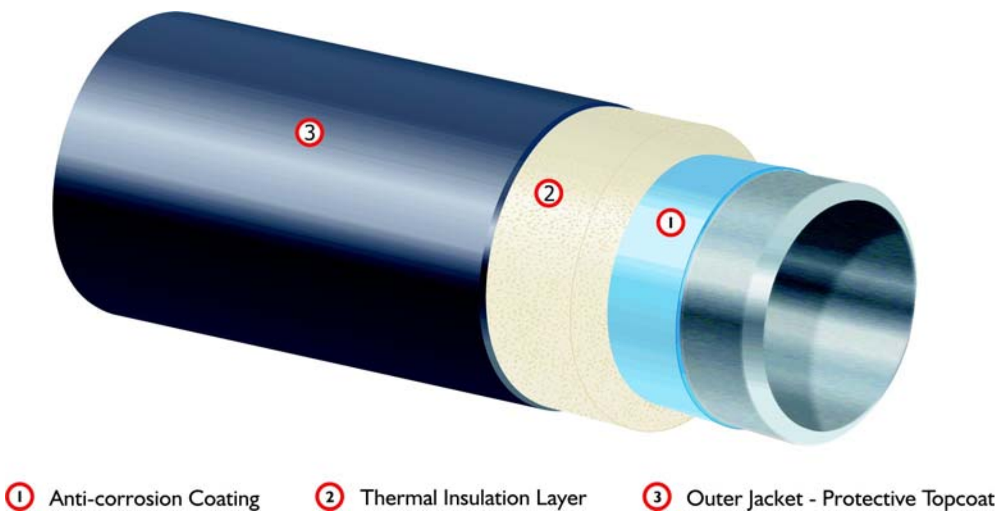


FIGURE 1 - Components of an Insulated Pipe

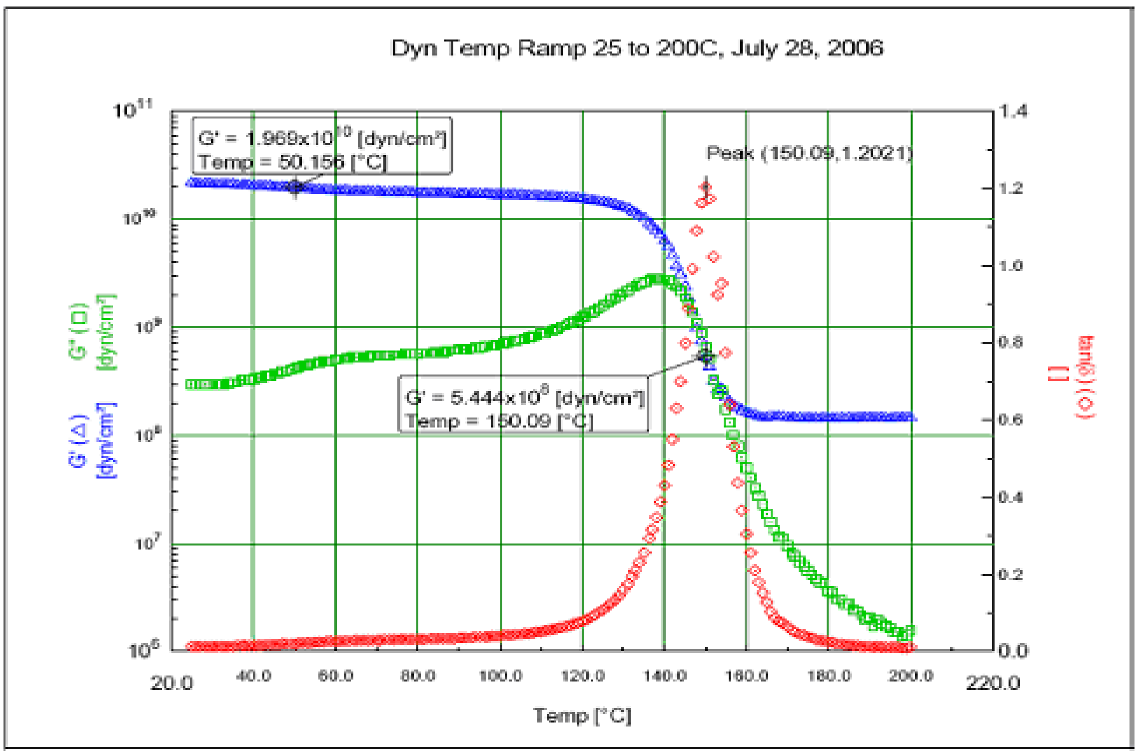


FIGURE 2 - DMA Test Results for Epoxy Coating

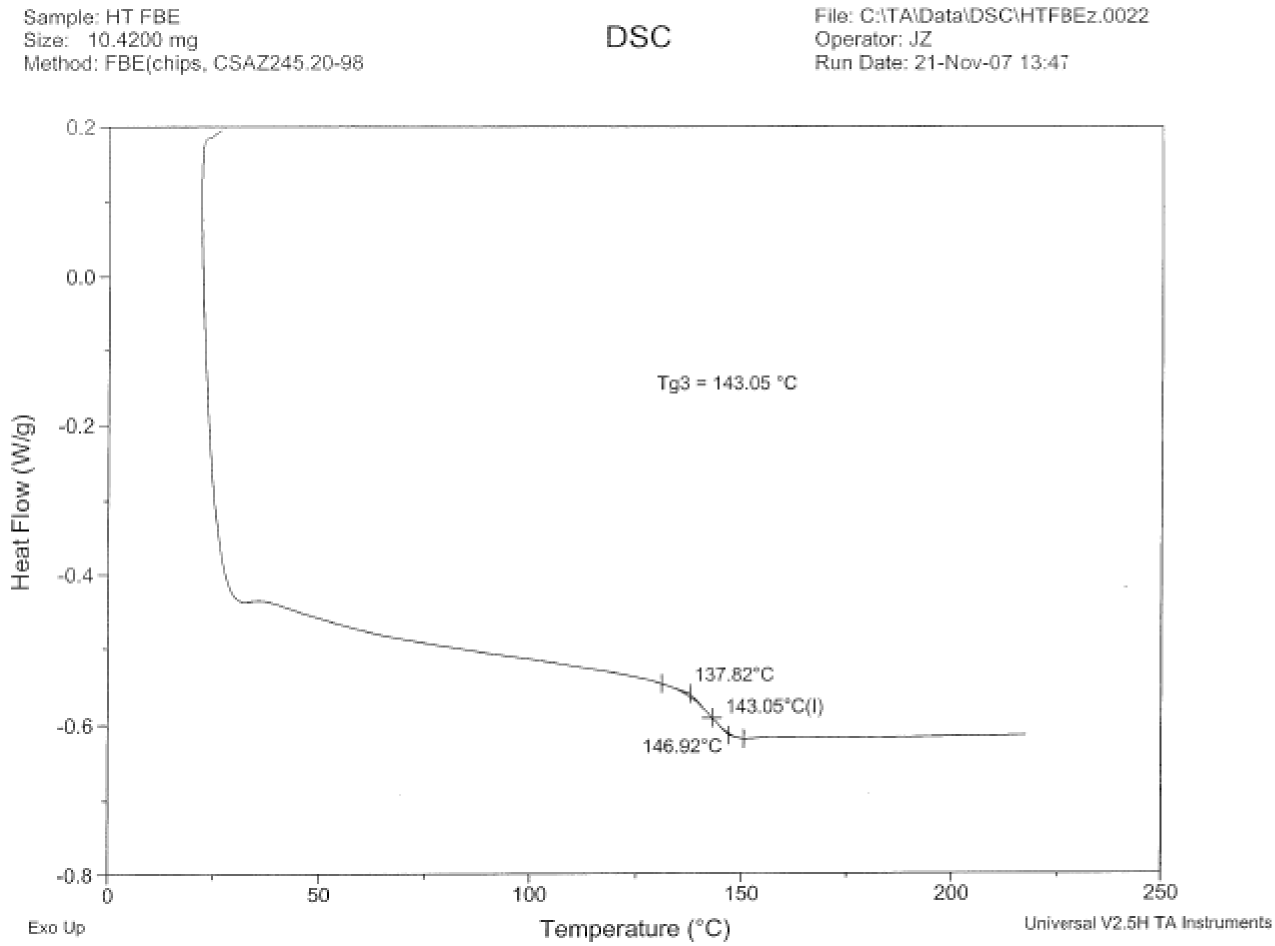


FIGURE 3 - DSC Scan for Glass Transition Temperature of FBE

TABLE 1
POLYMER LIFETIME FOR FBE MATERIAL BY TGA

Percent Conversion	Thermal Life at 5% Wt Loss (Years)					
	130°C		140°C		150°C	
	Nitrogen	Air	Nitrogen	Air	Nitrogen	Air
90% Conversion	240.8	26.2	79.0	10.2	27.3	4.1

TABLE 2
TYPICAL QUALIFICATION RESULTS FOR UNAGED FBE COATING TO CSA Z245.20-06

Test	Acceptance Criteria		
	System 1B	Results	Test Method
Thermal Characteristics	Meets Manufacturer's Specification	Passes	Clause 12.7
24h Cathodic Disbondment at 65°C	6.5mm Maximum Radius	2	Clause 12.8
28d Cathodic Disbondment at 22°C	8.5mm Maximum Radius	3 to 5	Clause 12.8
28d Cathodic Disbondment at 95°C	20mm Maximum Radius	9 to 11	Clause 12.8
Cross-Section Porosity	Rating of 1-4	2-3	Clause 12.10
Interface Porosity	Rating of 1-4	2-3	Clause 12.10
2.0° Flexibility at –30°C	No Cracking	No cracking at 2.20°/PPD	Clause 12.11
1.5 J Impact Resistance at –30°C	No Holidays	No holidays at 3.0 & 4.0 J	Clause 12.12
1.5° Strained Coating, 28d Cathodic Disbondment at 22°C	No Cracking	No cracking at 2.20°/PPD	Clause 12.13
24h Adhesion at 75°C	Rating of 1-3	1	Clause 12.14
28d Adhesion at 75°C	Rating of 1-3	1	Clause 12.14
Cure – Percent Conversion	95%	99.74%	Clause 12.7

TABLE 3
ELEVATED TEMPERATURE CATHODIC DISBONDMENT TESTING OF FBE

Test	Average Disbondment Radius (mm)		
	Panel-A	Panel-B	Panel-C
28 days CDT: -1.5V at 150°C Disb. radius, mm	6	5	7
56 days CDT: -1.5V at 150°C Disb. Radius, mm	14	15	10
Coating Thickness, μm	356-406	381- 406	381-406

FIGURE 4 - Insulated Pipe Accelerated Heat-Ageing (CCOT) Assembly

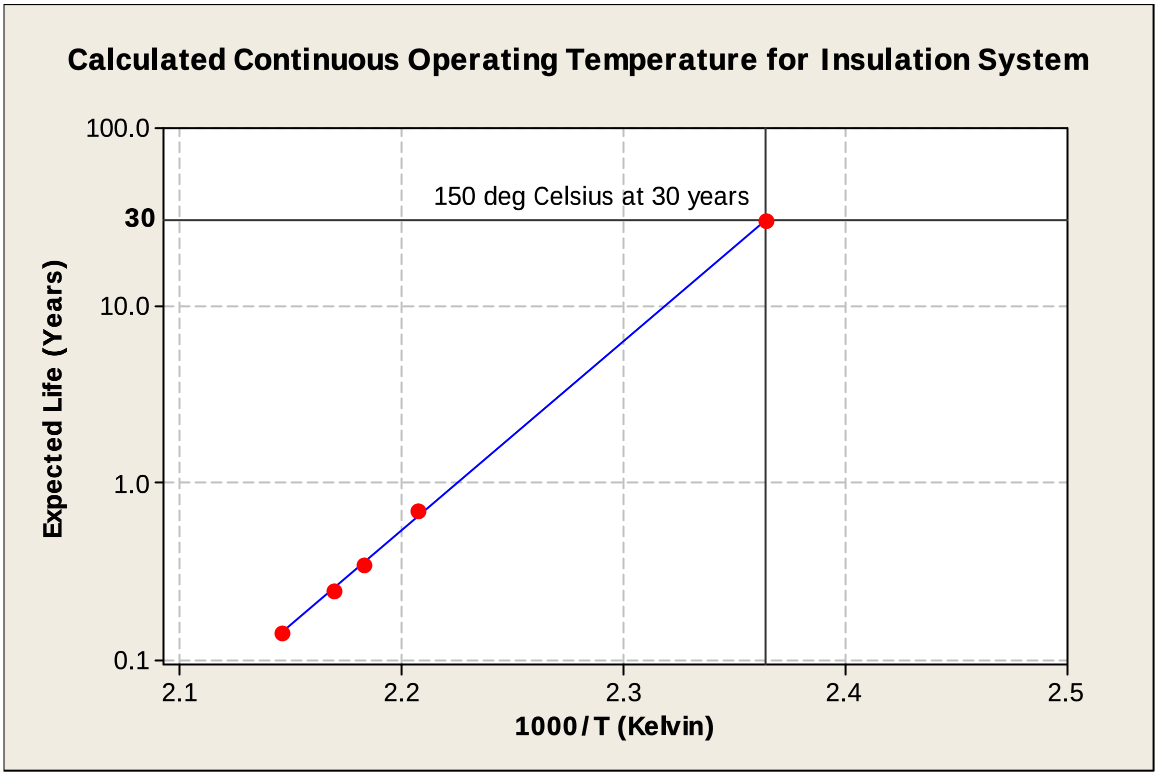
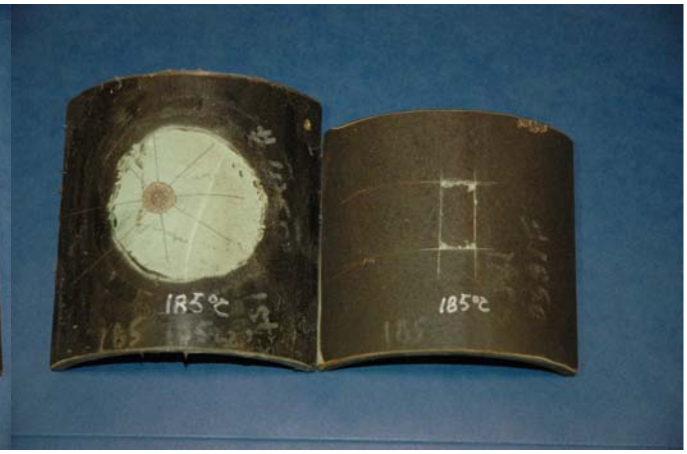


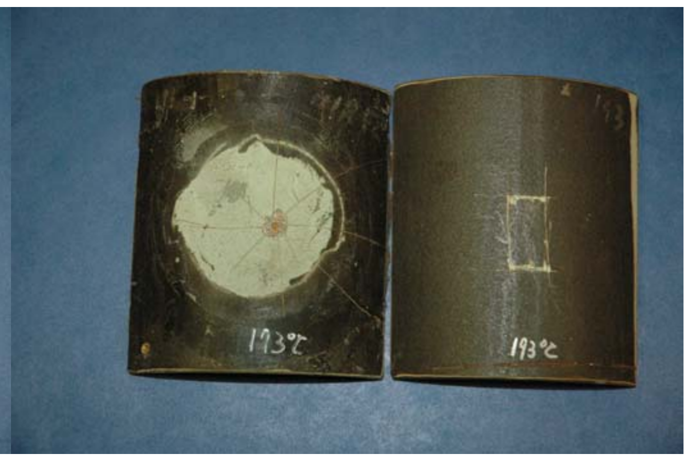
FIGURE 5 - Calculated Thermal Life (CCOT) For Insulation System

TABLE 4
RESULTS FOR PERFORMANCE TESTING OF CCOT AGED FBE COATINGS

Item	**Exposure Temperature**				
	22	**180**	**185**	**188**	**193**
Total Ageing Time (days)	Unaged	264	150	101	63
CD at 65°C, 24hrs, -3.5V Disbondment radius, mm	2.13	2.50	3.50	3.00	3.50
Hot Water Adhesion CSA Rating	1	1	1	1	1
Glass Transition, Tg3 (°C)	143.05	154.4	154.5	154.5	153.9



A B



C D

FIGURE 6 – CATHODIC DISBONDMENT AND HOT WATER SOAK ADHESION TESTING OF FBE FROM CCOT AGED PIPES.

Legend:

A: CCOT Aged 264 days at 180°C: 24-hrs CD at 65°C, -3.5V. HWS, 24-hrs at 75°C.
B: CCOT Aged 150 days at 185°C: 24-hrs CD at 65°C, -3.5V. HWS, 24-hrs at 75°C.
C: CCOT Aged 101 days at 188°C: 24-hrs CD at 65°C, -3.5V. HWS, 24-hrs at 75°C.
D: CCOT Aged 63 days at 193°C: 24-hrs CD at 65°C, -3.5V. HWS, 24-hrs at 75°C.