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UNIVERSITY OF CALGARY

Environmental Stress Corrosion Cracking Resistance of High Density Polyethylene Pipes in Alkali Surfactant Polymer Floods

By

Colin Dooley, P.Eng

A THESIS

SUBMITTED TO THE FACULTY OF GRADUATE STUDIES IN PARTIAL FULFILLMENT OF THE REQUIREENTS FOR THE DEGREE OF MASTER OF ENGINEERING

Department of Mechanical and Manufacturing Engineering

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UNIVERSITY OF CALGARY FACULTY OF GRADUATE STUDIES

The undersigned certify that they have read, and recommend to the Faculty of Graduate Studies for Acceptance, a thesis entitled "Determination of Stress Corrosion Cracking Resistance of High Density Polyethylene Pipes in Alkali Surfactant Polymer Floods" submitted by Colin Dooley in partial fulfillment of the requirements of the degree of Master of Engineering.

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ABSTRACT

Enhanced oil recovery (EOR) techniques such as alkali surfactant polymer (ASP) flooding will be required to meet future energy requirements. Large pipeline networks are required for the transportation of ASP fluids. To ensure the safe and reliable operation of these pipelines, high density polyethylene (HDPE) materials are frequently used due to its corrosion resistance from oil-field fluids. Surfactant chemicals and other wetting agents are known to cause environmental stress corrosion cracking (SCC) of polyethylene plastics. This thesis studies the resistance of HDPE pipeline materials to environmental stress corrosion cracking due to ASP chemicals.

This research taken an industrial focus, as a result, test methods were selected that closely represented actual pipeline conditions. Full scale HDPE pipe samples were pressurized with a solution of alkali and surfactant chemicals commonly used for ASP floods. Following conditioning, test specimens were prepared from the pipe samples and subjected to tensile testing to investigate for evidence of stress corrosion cracking. The results showed that conditioned specimens had no significant degradation of the mechanical properties compared to unconditioned samples.

Based on the test results, it is concluded that there is no evidence of environmental SCC of the HDPE pipe materials due to pressurized exposure to ASP fluids. Additional testing utilizing more severe accelerated test methods are recommended to confirm the long-term resistance of HDPE materials in ASP floods. Nevertheless, this work supports the empirical industry experience that current HDPE pipeline materials are resistance to ASP chemicals. These research results can be incorporated into current pipeline design and operating procedures to ensure the safe and reliable operation of ASP systems.

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LIST OF SYMBOLS, ABBREVIATIONS AND NOMENCLATURE

ABBREVIATON DEFINITION

ASP	Alkali surfactant polymer
ASTM	ASTM International
EOR	Enhanced oil recovery
IFT	Inter facial tension
HDPE	High density polyethylene
HDS	Hydrostatic design stress
LDPE	Low density polyethylene
MOP	Maximum Operating Pressure
КОН	Potassium hydroxide
MRS	Minimum required strength
NaOH	Sodium hydroxide
NPS	Nominal Pipe Size
PE	Polyethylene
SCC	Stress corrosion cracking
SDR	Standard dimension ratio
SMYS	Specified minimum yield strength
%wt	Weight percentage
YS	Yield Strength

1.0 BACKGROUND INFORMATION

1.1 Alkali Surfactant Polymer Flooding

Alkali surfactant polymer (ASP) flooding is an enhanced oil recovery (EOR) process that is used to increase the amount of crude oil that can be extracted from conventional oil reservoirs. Depending upon the reservoir characteristics and applicable EOR process, an additional 10% - 30% of the in-situ oil can be recovered compared to primary or secondary recovery methods [EPRI, 1999].

In the ASP process, low concentrations of an alkali chemical (e.g. NaOH, KOH) and surfactant are injected into the reservoir. The surfactant is used to achieve ultra-low interfacial tension between the trapped oil and the formation water. The ultra-low interfacial tension allows the alkali in the injection fluid to deeply penetrate the formation and react with the acidic components in the crude oil to form additional in-situ surfactants. Polymer is sometimes added to increase the viscosity of the injection fluid to minimize channeling and provide mobility control.

1.2 Use of HDPE in Oil and Gas Pipelines

Due to its corrosion resistance, high density polyethylene (HDPE) is commonly used in upstream pipelines (Figure 1-1). HDPE is used in in three forms:

- Free standing HDPE pipelines
- Steel pipelines fitted with tight-fit HDPE liners
- Spoolable composite pipelines with internal HDPE liner



Figure 1-1: Free-Standing HDPE, Tight-Fit Liner and Composite Pipe

1.3 HDPE Cracking in ASP Service

Due to unrelated issues, a tight-fit HDPE liner was removed by an operator from a steel ASP injection pipeline. Samples were sent to an independent third party consultant for analysis. Microscopic examination from one of the samples revealed the presence of a number of very tight cracks along the internal liner surface, as seen in Figure 1-2. The consultant could not ascertain the cause of the cracking.



Figure 1-2 Cracks on HDPE Liner (Magnification X100)

Historically, the pipeline operator had not observed this type of cracking in pipelines that transported fluids other than ASP. If the observed cracking in the HDPE was due to ASP fluids, it would lead to premature failure of the pipelines. Cracking in a free-standing HDPE pipeline would concentrate the circumferential hoop stress at the crack tip leading to failure once the crack grew to critical size. Through-wall cracking of internal liners in tight-fit steel pipelines and spoolable composite would lead to premature pipeline failures by allowing the ASP fluid to contact the pressure containing steel pipe or reinforcing fibreglass layers.

For pipelines with tight-fit HDPE liners, the carrier pipes are constructed from low alloy carbon steel. ASP injection fluid is typically transported at $20^{\circ}C - 60^{\circ}C$ with a pH of from 12.5 - 14. Under these operating conditions, the steel carrier pipe would be susceptible to caustic corrosion (API RP579, 2011), which would lead to premature failure of the pipeline.

Spoolable composite pipelines are constructed with reinforcing layers of varying materials from fibreglass to steel. Through-wall cracking of HDPE liners in composites with steel reinforcing layers would lead to caustic corrosion of the steel reinforcing layer. Through-wall cracking of HDPE liners in composites with fibre reinforcing layers may result in the saturation of fibres. The strength of some spoolable composite pipe systems is severely diminished if the fibreglass layers are exposed to moisture.

The small number of ASP systems in North America and limited experience with HDPE materials in ASP service suggests that a single finding of HDPE cracking should be examined closely to better understand potential SCC mechanisms. If the presence of ASP fluids induces cracking in HDPE materials it will result in premature pipeline failures. Safe and reliable pipeline networks are critical to the effective use of EOR methods such as ASP flooding. A better understanding of HDPE compatibility with ASP fluids is required by industry for the successful design, operation, and integrity management of pipeline systems.

1.4 Objectives

The primary objective of this research was to determine the environmental stress corrosion cracking resistance of HDPE pipe in ASP service. Due to the industrial application of the research, the test methods had to closely resemble actual pipeline operating conditions and render results that were directly relatable to current operating pipeline systems.

1.5 Content of Thesis

The thesis contains the following main sections:

- Literature Review
- Operator Qualification of HDPE in ASP Service
- Experimental
- Results
- Conclusions
- Future Work

The literature review examines the current state of knowledge regarding the use of and potential damage mechanisms of HDPE materials in a combined environment of alkali and surfactant. The literature review showed that surfactant chemicals are known to cause environmental stress corrosion cracking in HDPE. However, no research was found that tested the cracking resistance of HDPE pipe in ASP Service.

The next section presents non-public testing results completed by a pipe manufacturer for a Western Canadian oil and gas company that was constructing as ASP system. The test results showed no degradation of the HDPE material in ASP fluids. However, the test specimens were not subject to any tensile stress during testing. As a result, the testing performed did not adequately test the resistance of the material to environmental stress corrosion cracking.

The experimental section presents the test methods, materials, conditions and equipment utilized to evaluate the stress corrosion cracking resistance of HDPE in ASP service. Testing was conducted utilizing full scale pipe samples under conditions that were representative of actual pipeline operating conditions. Following conditioning, the samples were subjected to mechanical testing utilizing a tensile tester to investigate for the presence of SCC.

Finally, the results, conclusions and recommendations for future work are outlined in the remaining sections.

2.0 LITERATURE REVIEW

2.1 Polyethylene

Polymers are large molecule formed by the polymerization (i.e. the chemical linking) or repeating of small molecular units. Polyethylene is generally formed by the polymerization of ethylene (Figure 2-1). Ethylene can also be copolymerized with small amount of other monomers such as butane, propylene, hexane, and octane, which result in small modifications in chemical structure which are reflected in differences in material properties such as density, ductility, hardness, etc.



Figure 2-1: Diagram of Polymerization of Ethylene to Polyethylene

2.1.1 Branching

The amount of side branching determines the density of the polyethylene molecule. The more side branches, the lower the density. Controlled branching results in improved performance where certain types of stresses are involved. Figure 2-2 depicts the various molecular structures associated with the four main classifications of polyethylene:

- PE homopolymer
- PE copolymer
- High Pressure LDPE
- Linear LDPE



Figure 2-2: Chain Branching Structure of Polyethylene [PPI 2007].

2.1.2 Crystalline Structure

The relationship between branching and density can be explained in terms of crystalline versus non-crystalline or amorphous regions. Portions of the polymer chain in certain regions align themselves in closely packed and very well ordered arrangements of polyhedral-shaped, microscopic crystals called spherulites. Other portions of the polymer chain lie in amorphous regions that have no definite molecular arrangement. Since polyethylene contains both crystalline and amorphous regions, it is called a semi-crystalline material. High density polyethylene grades can contain up to 90% crystalline regions compared to 40% for low density polyethylene.

2.1.3 Density

Polyethylene pipe in North America is generally classified by its density in accordance with ASTM D3350 (Table 2-1). In Europe, polyethylene pipe is generally classified by performance, generally tensile strength. This classification is beginning to emerge in North America; however, classification by density is still the most common.

Type Density		Class	
I	l 0.910 – 0.925 Low		
II	0.926 - 0.940	Medium	
111	0.941 – 0.959	High	
IV ≥ 0.960 High		High or Homopolymer	

Table 2-1: Density Classifications for Polyethylene

High density polyethylene is chemically the closet in structure to pure polyethylene. It consists primarily of unbranched molecules with very few flaws to impair its linearity. With a low level of flaws to hinder organization, the resins achieve a high degree of crystallinity resulting in a high density relative to other types of polyethylene.

Low density polyethylene contains substantial concentrations of branches that hinder the crystallization process, resulting in relatively low densities. The branches primarily consist of ethyl and butyl group with some long chain branches. Medium density polyethylene contains a level of crystallization and branching between high density and low density polyethylene.

2.1.4 Molecular Weight Distribution

The size of a polymer molecule is represented by is molecular weight. Molecular weights exert a great influence on the processability and the final physical and mechanical properties of the polymer. During the production of polyethylene, not all molecules grow to the same length. As a result, the molecular weight is a distribution and often expressed as an average value.

The distribution of different sized molecules in a polyethylene polymer typically follows the bell shaped normal distribution curve described by the Gaussian probability theory. Polymers can also have bimodal shaped distribution curves which depict a blend of two different polymer populations, each with its particular average and distribution. Resins having a bimodal molecular weight distribution contain both very short and very long molecules, resulting in excellent resin physical properties while maintaining good processability. Figure 2-3 shows the difference in thee distributions.



Figure 2-3: Polyethylene Molecular Weight Distributions [PI 2007]

The latest generation of North American HDPE pipe materials (e.g. PE4710) are typically produced from bimodal resins. These materials are characterized by improved resistance to slow crack growth, higher pressure ratings and improved chemical resistance.

Molecular weight distribution (MWD) is dependent upon the type of process used to manufacture the particular polyethylene resin. The effects of density and molecular weight distribution on physical properties are summarized in Table 2-2.

Property		As Density Increases	As MWD Broadens
	Yield Strength	Increases	Varies
	Stiffness	Increases	Decreases
	Impact Strength	Decreases	Decreases
L	ow Temperature Brittleness	Increases	Decreases
	Hardness	Increases	Varies
	Stress Crack Resistance	Decreases	Increases
	Chemical Resistance	Increases	Varies
	Shrinkage	Decreases	Increases

Table 2-2: Effects of Changes in Density and Molecular Weight Distribution

2.1.5 Tensile Strength

A traditional means for determining the strength of materials has been the tensile test, by which the stress / strain behaviour of the material of interest is evaluated under a constant strain rate. Because of its viscoelastic nature, polyethylene does exhibit a true elastic region. As illustrated in Figure 2-4, polyethylene exhibits a yield point in the tensile test. Increasing the strain further results in irreversible yielding of the material until failure is achieved at the ultimate strength of the material. Also, as is illustrated by Figure 2-4 the stress strain curve is significantly affected by the rate of straining.



Figure 2-4: Example Stress vs. Strain Curves for Polyethylene [PI 2007]

2.1.6 Chemical Resistance

The chemical resistance of solid materials is often defined as the ability of the material to resist damage or degradation by chemical reactivity or solvent action. Generally, polyethylene is widely recognized for its unique chemical resistance to a wide variety of chemicals.

A comprehensive chemical resistance chart for polyethylene has been published by the Plastics Pipe Institute (PPI) in the *Handbook of Polyethylene Pipe* (PPI 2009). It is important to note that these chemical resistance tables are only a guideline. Chemical resistance data, including those provided in the handbook, are generally developed on the basis of laboratory tests involving the evaluation of tensile coupons in the presence of a single chemical at atmospheric pressure. As such, they do not assess the effect produced by exposure to various combinations of chemicals listed. Additionally, these chemical resistance tables do not take into consideration the effect of stress loading, magnitude or duration.

A preliminary measure of the potential effects of chemicals on the properties of polyethylene is by means of a "soak" or "chemical immersion" test in which the polyethylene is not subjected to any stresses. In this type of laboratory test, strips of PE material are soaked for different periods of time – generally, not longer than a month – in the medium of interest, which is maintained at a specified temperature. After certain soaking periods, changes are noted in appearance, dimensions, weight gain or loss, and in strength properties – generally, tensile strength at yield or elongation at break.

The chemical resistance of HDPE to ASP fluids is generally understood to be:

 Aqueous solutions of salts, acids, bases – Because polyethylene is virtually immune to electrolytic attack these solutions have no adverse effect. Nearly all manufacturers list the chemical resistance of HDPE to strong salts, acids, and bases such as sodium hydroxide as excellent.

- Surface active agents (e.g. surfactants) –Surface active agents are known to cause environmental stress cracking of polyethylene.
- Aqueous Polymer Mixture The aqueous polymer mixture utilized in ASP systems consist of a small quantity of solid polymer dispersed in water. In this sense, the mixture is not a liquid chemical, but a dilute solid in water. The dispersed polymer particles would be extremely large compared to the individual polymer chains in the HDPE material. Overall, the aqueous polymer solution would have no effect on the polyethylene materials.

Nearly all chemical compatibility of HDPE is conducted with single chemicals under atmospheric pressures. Limited testing has been conducted on the combined or synergistic effects of chemicals. No literature was found discussing the compatibility of HDPE in an aqueous alkaline environment with surfactants, with or without pressure.

2.2 Polyethylene and Pipelines

Since its discovery in 1933, polyethylene has grown to become of the world's most widely used and recognized thermoplastic material (AMC, 2005). Polyethylene's use as a piping material first occurred in the mid 1950's. The early success of polyethylene pipes lead to its widespread use in the natural gas distribution industry. It is estimated that nearly 95% of new gas distribution pipe installations in North America that are 12" in diameter or smaller are polyethylene piping [PPI, 2011]. The use of polyethylene materials in the upstream oil and gas industry has been increasing recently with the advent of spoolable composite pipe and tight-fit liners, as well as stronger grades of HDPE for higher pressure ratings in free-standing HDPE pipelines.

Some of the specific benefits of using polyethylene in pipelines are:

- Life Cycle Cost Savings The life cyclic cost of polyethylene pipe can be significantly less than steel pipe. The extremely smooth inside surface results in exceptional flow characteristics that reduce total system operating costs.
- Corrosion and Chemical Resistance Polyethylene pipe does not rot, corrode or support biological growth. It has excellent chemical resistance in a wide range of environments.
- Flexibility and Construction Advantages Polyethylene pipe can be field bent to a radius of about 30 times the normal pipe diameter or less depending o wall thickness. This allows the use of spoolable pipe resulting in less field joining of 'stick' pipe.

2.2.1 HDPE Pipe Grades

The three most common grades of HDPE currently used in North America are PE3608 and PE4710. PE100+ grades have been used in Europe for several years and are beginning to have a presence in the North American market. Direct comparison of PE3608/4710 and PE100+ is difficult because PE3608/4710 materials are generally tested to American ASTM standards, whereas PE100+ is tested to international ISO standards. A selection of the physical properties for PE3608 and PE4710 materials is shown in Table 2-3.

Table 2-3: Material	Physical	Properties for	Common	HDPE	Grades

Physical Property	Test Method	PE3608	PE4710
Tensile Strength at Yield	ASTM D638	20.6 MPa	25 MPa
Tensile Elongation	ASTM D638	>700%	>700%
HDS for water at 60°C	PPI TR-3	5.51 MPa	6.89 MPa
SCG Resistance, PENT	ASTM F1473	>100 hrs	> 2500 hrs

The primary difference between PE100+ grades and traditional HDPE grades is the creep rupture strength, stress crack resistance and resistance to rapid crack propagation (PE100+ Association, 2012). North American ASTM standards for HDPE are at a lower level compared to European or ISO HDPE standards. The PE100+ testing requirements are shown in Table 2-4.

Physical Property	Test Method	CEN/ISO Requirement	PE100+ Requirement	
Creep Rupture Strength	Pressure test at 20oC and 12.4 MPa	≥ 100 hrs	≥ 200 hrs	
Stress Crack Resistance	Pipe notch test at 80°C and 0.92 MPa	≥ 165 hrs	≥ 500 hrs	
Resistance to Rapid Crack Propagation	S4 Test a 0°C	$PC \ge \frac{MOP}{2.4} - \frac{13}{18}$	≥ 1.0 MPa	

Table 2-4: PE 100+ Requirements Compared to European Standards

2.2.2 Pressure Rating of HDPE Pipe

Polyethylene is different than other common pipeline materials because its strength under load depends on the magnitude and the duration of the load. In North America, Hydrostatic Design Stress (HDS) ratings for long-term internal pressure service are determined in accordance with ASTM D1598 and PPI TR-3 standards. Outside of North America and for PE100+ grades, the Minimum Required Strength (MRS) is determined in accordance with ISO 12162.

Table 2-5: HDS and MRS for HDPE Pipe at 20°C

HDPE Grade	HDS (MPa)	MRS (MPa)
PE3608	5.8	
PE4710	7.1	
PE100+		10

Industrial pressure rating for HDPE pipe is calculated based on a combination of PE grade, diameter, wall thickness, temperature and internal fluid. The following equation is used to determine the long-term internal pressure rating:

$$PR = \frac{2 \cdot HDS \cdot f_E \cdot f_T}{(SDR - 1)}$$

Where:

- *PR* Pressure Rating
- HDS Hydrostatic design stress (MRS may be substituted)
- *f_E* Environment Design Factor
- *f*_T Operating Temperature Multiplier
- SDR Standard Dimension Ratio

For a pressurized fluid inside the pipe or for a chemically significant environment outside the pipe, an environmental factor (f_E) is applied to reduce the pressure rating of the HDPE Pipe (Table 2-6).

Table	2-6:	HDPE	Pipe	Environment	Factor,	f _F

f _E	Fluid and Environmental Comments
1.00	Internal liquids, gases and external soils or liquids that are chemically benign to polyethylene such as water, sewage, brine solutions, glycol, alcohols, dry natural gas, landfill gas, nitrogen, air, oxygen, carbon dioxide, and hydrogen sulfide
0.80	Buried distribution, transmission or gathering systems for Canadian Federal and Provincial regulated fuel gases such as natural gas, LP gas, propane, butane, landfill gas
0.64	Buried distribution, gathering or transmission systems for US Federal and State regulated dry fuel gases such as natural gas, LP gas, propane, butane, and landfill gas
0.50	Multi-phase fluids, wet natural gas, and liquids containing >2% permeating or solvating liquids in the pipe or surrounding soil such as hydrocarbons (gasoline, fuel oil, kerosene, crude oil, diesel fuel)

For distribution and transmission of liquids such as water or water-born slurries, surge pressure allowances are applied above the pipe pressure rating. Occasional pressure surges typically result from instantaneous liquid velocity changes from component failure. Recurring pressure surges typically result from cyclical events such as pump or system control operation or regularly occurring system draws. Pressure surge allowance is provided for occasional or recurring pressure surges using the following equations:

$$P_{OS} = 1.00 \times PR$$
$$P_{RS} = 0.50 \times PR$$

Where:

 P_{OS} = Surge pressure allowance for occasional surge P_{RS} = Surge pressure allowance for recurring surge

2.3 Chemicals Used in ASP Floods

2.3.1 Alkali

Several different alkaline agents have been used in ASP flooding including sodium hydroxide, sodium orthosilicate, sodium carbonate, ammonium hydroxide and ammonium carbonate (Gogarty, W.B., 1983). The first three have been the most widely considered because of their widespread availability, relatively low cost and effective performance in ASP laboratory studies.

Addition of alkali chemicals results in an increase in pH because of the dissociation in the aqueous phase. For example, sodium hydroxide (NaOH) disassociates to yield [OH⁻] as follows:

$$NaOH \rightarrow Na^+ + OH^-$$

Equilibrium dissociation of water is given by:

$$K = \frac{[OH^-][H^+]}{[H_2O]}$$

Where brackets indicate molar concentration and an increase in [OH⁻] causes a decrease in [H⁺]. Since the concentration of water is essentially constant pH is defined as:

$$pH = -\log_{10}[H^+]$$

Therefore, if the concentration of [OH-] increases, the [H+] decreases and the pH increases resulting in a more basic environment.

Sodium carbonate disassociates into carbonate as:

$$Na_2CO_3 \rightarrow 2Na^+ + CO_3^{2-}$$

Followed by the hydrolysis reaction:

$$CO_3^{2-} + H_2O \rightarrow HCO_3^{-} + OH^{-}$$

The dissociation of sodium silicate compounds is complete, involving formation of oligomeric species. Consequently, cannot be represented by a single chemical equation. Figure 2-5 compares several commonly used alkaline materials (Mayer. et al, 1983).



Figure 2-5: pH Comparison of Common Alkaline Chemicals [Mayer 83]

2.3.2 Surfactants

Surface active agents, or surfactants, are chemical substances that adsorb on or concentrate at a surface or fluid/fluid interface when present at low concentrations in a system [Rosen, 1978]. They alter the interfacial properties significantly; in particular, they decrease the interfacial surface tension, or IFT. Surfactants usually consist of a hydrocarbon portion (nonpolar) and a polar or ionic portion.

The nonpolar or tail end of a surfactant is hydrophobic. Conversely, the polar or head end interacts strongly with water molecules, undergoing solvation. This part of the surfactant is hydrophilic. Basically, it is the balance between the hydrophilic and hydrophobic parts of a surfactant that gives it the characteristics associated with a surface active agent.

Surfactants may be classified according to the ionic nature of the head group as anion, cationic, non-ionic and zwitterionic [Ottewill, 1984]. Anionic surfactants have been the most widely used in EOR processes because they have good surfactant properties, are relatively stable, exhibit relatively low adsorption on reservoir rock, and can be manufactured economically. The most common surfactants used in ASP flooding are sulfonated hydrocarbons. The molecular structure of a common anionic sulfonated surfactant used in ASP systems is shown in Figure 2-6.



Figure 2-6: Molecular Structure of Sodium Dodecyl Sulfate Surfactant

2.3.3 Polymers

Low concentrations of polymers are sometimes added to the injection water to reduce channelling and improve sweep efficiencies in ASP flood. Under formation or process conditions the injected polymer is inert and is used solely as a viscosity thickening agent. The polymer is chemically inert and does not react chemically with the formation fluids, injected alkali, surfactant, or HDPE in the pipeline gathering system.

2.4 Stress Corrosion Cracking (SCC)

Stress corrosion cracking is the cracking of a material produced by the combined action of corrosion and tensile stress (residual or applied). Three conditions must be present simultaneously to produce SCC; critical environment, susceptible material and tensile stress (Figure 2-7).



Figure 2-7: Three Conditions Required for SCC

The critical environment is often specific to the material and may not have an effect on other material types. For example, hot aqueous chloride solutions readily crack stainless steels, but do not have the same effect on carbon steels,

aluminum, or other nonferrous alloys. For polyethylene materials, surface active agents such as surfactants have been known to induce stress corrosion cracking. It is important to note that cracking of polyethylene under the effect of wetting agents starts under stresses far below the allowable proof stress of the material.

2.5 Pipeline Performance in Alberta

At the end of 2005 there were over 377,000 kilometres of energy related pipelines in Alberta (ERCB, 2007). During the period from 1990 to 2005, there were 12,191 pipeline failures in Alberta. Internal corrosion is the prevalent cause of pipeline failures during this period representing 57.7% of all releases (Figure 2-8).



Figure 2-8: Pipeline Failures by Cause in Alberta from 1990-2005 [ERCB 05]

2.5.1 Pipeline Failure Frequency

The average frequency of pipeline incidents in Alberta declined steadily from 1990 through 2005 (see Figure 2-9). The ERCB is continuing to look for ways to improve pipeline performance with emphasis on new corrosion-resistant composite and polymeric pipelines. The vast majority of these new pipeline materials utilized HDPE as an internal liner or bladder. The ERCB expects growth in the installation of non-metallic pipelines in the future (ERCB, 2007).



Figure 2-9: Average Frequency of Alberta Pipelines, 1990–2005 [ERCB 05]

2.5.2 Performance of Non-Metallic Pipelines in Alberta

In 2007, an analysis of pipeline failure statistics was performed and presented at a pipeline symposium held in Banff, Alberta [CAPP, 2009]. The analysis showed a relatively high incident rate with reinforced composite pipelines, with fibreglass pipelines having particular high failure rate (Figure 2-10).



Figure 2-10: Pipeline Failure Frequency for Alberta, 2007 [CAPP 07]

Figure 2-11 provides a summary of reinforced composite pipeline failures in Alberta by cause from 2002 to 2007. The data indicates that the most common and reoccurring causes of failures include:

- Damage from construction or installation
- Corrosion of associated steel pipe risers and fittings
- Damage by others (third party damage)
- Mechanical failures of valves or fittings



Figure 2-11: Reinforced Composite Failures by Cause [CAPP 07]

2.5.3 Factors in Non-Metallic Pipeline Failures

Based on experience in the pipeline industry in Western Canada, it can be inferred that the high failure rate associated with reinforced composite pipelines and other non-metallic pipe systems can be largely attributed to the lack of industry experience in North America in designing, constructing and operating pipelines that are not constructed from steel.

Steel pipelines have been constructed since the late 1800's and early 1900's. As a result, there is a large depth of knowledge and experience related to the design, construction and operation of steel pipelines. The codes and regulations governing steel pipelines are mature representing the collection of knowledge gained from more than a century of operating hundreds of thousands of kilometers of steel pipelines. In contrast, non-metallic pipelines containing HDPE materials (e.g. liners, spoolable composites) have only been used by the oil and gas industry for the past 10 - 15 years¹. Many of the failures associated with non-metallic pipelines are believed to be related to "growing pains" often associated with the adoption of a new materials or technologies. The lessons learned by industry from these failures will be incorporated into the next generation of non-metallic pipeline codes and regulations to prevent similar occurrences. As the industry matures, it is expected that the failure rate associated with non-metallic pipelines will decrease over time.

However, the high failure rate associated with non-metallic pipelines coupled with the relative lack of industry experience and knowledge (compared to the steel pipeline industry), highlights the need to increase the collective knowledge and understanding of the complete life cycle of non-metallic pipelines. Moreover, with the increased utilization of new enhanced oil recovery methods, non-metallic pipelines systems will be required to operate in unfamiliar conditions with new operating parameters and production fluids that must also be understood.

¹ Free-standing HDPE pipelines have been used by the low pressure natural gas distribution industry since the 1970's. However, due to their very low operating pressures, the low pressure systems do not face the same challenges as seen in the upstream oil and gas industry. Many of the non-metallic pipeline failures experienced by upstream oil and gas pipelines do not occur in the natural gas distribution industry due to their low operating pressures and small pipe diameters.

3.0 OPERATOR QUALIFICATION OF HDPE IN ASP SERVICE

To qualify the suitability of HDPE for use in ASP pipelines, the pipeline operator requested that the pipe manufacturer conduct testing of the HDPE pipe material in ASP chemicals (Section 3.1). Following operation of the ASP pipeline system, a section of a reinforced composite pipeline was removed and analyzed to determine if it had degraded as a result of the ASP service (Section 3.2).

3.1 Industry Testing of HDPE in ASP Fluid

Six (6) dumbbell specimens of HDPE were immersed in a glass bottle containing ASP Fluid supplied by the Company. The bottle was sealed to avoid any evaporation and kept in a controlled environment at 60°C for three months.

All samples were weighed once a week for the first month using a high-accuracy balance with an accuracy of 0.01 grams. After weighing, all samples were immediately immersed back into the fluid. No significant weight gain or loss was observed during the three month exposure period (see Figure 3-1). In the first week approximately 1%wt loss was noticed. The reason for this weight loss is unknown. In the three (3) month period, all specimens gains around 0.4%wt. This amount of weight gain is considered minimal in comparison to weight gain from other fluids common to the oil and gas industry such as crude oil or aromatics.



Figure 3-1: Weight Change over Time for HDPE at 60°C in ASP Fluid [Ref]

The mechanical properties of the immersed specimens were tested at 60°C and compared with the properties of the unexposed material. Mechanical properties decreased slightly following exposure (see Figure 3-2). The decrease in mechanical properties is consistent with immersion tests conducted with water at 60°C and is not attributed to the presence of ASP fluid.



Figure 3-2: Mechanical Properties of HDPE Samples Tested at 60°C [Ref]

3.2 Analysis of Pipe Sample in ASP Service

A section of pipe transporting oil effluent and diluted ASP fluid was cut-out and analyzed. The pipeline was operating at 1.0 MPa, 10° C and a pH of 11.7. Undiluted ASP fluid typically has a pH ranging from 12 - 14. The pipeline had been in service for approximately four years.

Tensile testing on HDPE specimens removed from the pipeline was conducted in accordance with ASTM D638. The yield strength and elongation at break for these specimens were marginally lower than typical values reported on the resin manufacturer data sheets for this material (see Table 3-1). However, these pipe samples are manufactured from an extrusion process. Polyethylene manufactured from extrusion processes are known to have slightly lower physical properties than resin samples made from compression molded processes, as used by resin manufactures for the purpose of publishing data sheets.

Moreover, the results are comparable to other pipe cut-outs previously conducted in non-ASP service conditions. Hence, hence it is believed that the decrease in tensile strength is caused by a combination of aqueous immersion and manufacturing method, not from the presence of ASP chemicals.

Description	Yield Stress (MPa)	Elongation at Break (%)	Elastic Modulus (MPa)
Average Specimen Values	20.2	797	572
Resin Manufacturer Datasheet	22.4	850	760 - 860

 Table 3-1: HDPE Tensile Properties from Pipeline Cut-Out

The decrease in tensile elastic modulus was greater compared to the drop in yield strength and elongation at break (see Table 3-1). However, this result compares favourably with the drop in tensile modulus that is considered acceptable in other service conditions such as those with high aromatic hydrocarbon contents. Thus, the HDPE is considered to be in good condition and well able to perform a non-structural role of fluid bladder.

4.0 EXPERIMENTAL

A review of the chemical resistance of HDPE shows that surface active agents such as surfactants have been known to cause environmentally stress corrosion cracking (SCC) of polyethylene. However, the qualification testing performed by the pipe manufacturer did not apply any tensile stress to the test specimens. Without the application of tensile stress, one of the three necessary conditions for stress corrosion cracking was not present. Hence, the qualification tests did not evaluate the stress corrosion cracking resistance of the HDPE to the ASP chemicals. The aim of this research is to test the HDPE materials in an overall environment that susceptible to stress corrosion cracking. Therefore, the HDPE will be tested in ASP chemicals while applying a tensile stress.

4.1 Overview of Research Experiment

The purpose of these experiments is to determine the resistance of common pipeline HDPE grades to environmental stress corrosion cracking in an aqueous alkaline environment containing surfactant commonly used in ASP floods. Different HDPE pipe samples were filled with an alkali and surfactant mixture representative of ASP flood conditions. To apply a circumferential tensile stress, the samples were pressurized to the maximum pressure rating of the respective pipe and set in a controlled temperature environment. After the pressurized immersion testing the mechanical properties of the HDPE were evaluated using a tensile tester. The testing results were compared to native HDPE pipe samples to determine the effect of the ASP chemicals in a stressed state.

4.2 Materials

4.2.1 HDPE Pipe Samples

Tests were conducted on three HDPE pipe grades (see Table 4-1). All pipe materials used in this experiment were donated and subject to availability from the pipe manufacturers.

Grade	Diameter	SDR
PE3608	NPS 4	17
PE4710	NPS 4	17
PE100+	NPS 3	11

Table 4-1: HDPE Pipe Grades in Experiment

4.2.2 Chemicals

Tests were conducted in distilled water with the addition of an alkali and surfactant commonly used in Western Canadian ASP floods (see Table 4-2). Petrostep S-1 is the trade name for an anionic surfactant with the chemical description C_{16-17} – 7PO – SO₄. Note, the structure does not indicate the degree to which the surfactant branches.

Table 4-2: Chemical Type and Concentrations in Experiment

Description	Chemical	Concentration
Alkali	Sodium hydroxide (NaOH)	1.5%
Surfactant	Stepan Petrostep S-1	0.5%

The polymer component the ASP flood was not added to the test mixture for a combination of reasons. First and foremost, as polymer is an inert solid material that is dispersed in the solution, it should have no chemical effect on the HDPE. Second the polymer solution is difficult to produce, requiring several grinding and blending steps. Based on the production difficulties and lack of mechanism that could damage the HDPE it was not included.

4.3 Test Conditions

4.3.1 Temperature

The maximum operating temperature of most HDPE pipe is 60° C, with most ASP flood systems operating between 15° C – 40° C. In this experiment, HDPE pipe samples were testing at 25° C (room temperature) and 75° C, with the 75° C test being more severe than actual ASP system conditions.

4.3.2 Pressure

The experiment was conducted at the applicable pressure rating for the HDPE pipe sample with no de-rating for environment or surging (i.e. $f_E = 1$, $PR = P_{RS}$). Due to the discrepancy between standards for the PE3608/4710 and PE100+ grades, the hydrostatic design stress (HDS) of PE4710 was utilized for PE100+ because of safety considerations. Although this likely resulted in a lower stress in the PE100+ pipe sample, the stress is still higher then would be allowed in ASP service due to environmental de-rating factor (f_E) as required by Canadian pipeline regulations and codes. Test Pressures for the applicable pipe samples are shown in Table 4-3.

HDPE	Pressure (kPa)		
Grade	25°C	75°C	
PE3608	670	240	
PE4710	845	420	
PE100+	1340	675	

Table 4-3: Test Pressure Based on HDPE Pipe Pressure Rating

4.4 Experimental Procedure

The HDPE pipe samples were filled with the ASP solution, mounted in a pressure containing aluminum jig assembly and pressurized to the respective test pressure (see Figure 4-1). The 75°C samples were placed in a controlled temperature oven (25°C were left at room temperature). All the samples were 31

left pressurized for a period of three months. The samples were routinely checked to ensure the samples remaining pressurized.



Figure 4-1: Pressurized HDPE Pipe Samples

During immersion testing, the pressure retraining seals failed on two of the three HDPE pipe samples that were being tested at 75° C (Table 4-4). As a result, these samples had to be discarded. Additional attempts to improve the seal at 75° C were unsuccessful.

HDPE Result		Comments
PE3608	3608 Good Test Test sample remained pressurized for 3 mc	
PE4710	Seal Failure	Seal failed resulting in loss of pressure
PE100+ Seal Failure		Seal failed resulting in loss of pressure

Table 4-4: Summary of Failed 75°C Test Samples

4.5 Tensile Testing

Tensile testing of the HDPE pipe samples was completed using a BOSE 10 Kip SmartTest Servopneumatic Fatigue Test System in accordance with ASTM D638-03 "Standard Test Method for Tensile Properties of Plastics".

Test specimens were cut-out of the HDPE pipe samples and prepared with the dimensions shown in Figure 4-2. In accordance with ASTM D638-03, speed of testing was 500 mm/min +/- 10%. Tests were conducted at room temperature (approximately 25° C).



Figure 4-2: Test Specimen Dimensions

5.0 RESULTS

5.1 Stress Strain Curves

The stress-strain curves measured for un-soaked HDPE pipe samples are shown in Figure 5-1 through Figure 5-3. Stress-strain curves for HDPE pipe samples soaked in ASP solution are shown in Figure 5-4 through Figure 5-7. Note, tensile testing results for the 4710 and PE100+ samples that were immersed at 75° C are not available due to seal failures.



5.1.1 Un-Soaked HDPE Pipe Samples

Figure 5-1: Stress-Strain Curve for Un-Soaked 3608



Figure 5-2:Stress-Strain Curve for Un-Soaked 4710



Figure 5-3: Stress-Strain Curve for Un-Soaked PE100+





Figure 5-4: Stress-Strain Curve for 3608 Conditioned in ASP at 25°C



Figure 5-5: Stress-Strain Curve for 4710 Conditioned in ASP at 25°C



Figure 5-6: Stress-Strain Curve for PE100+ Conditioned in ASP at 25°C



Figure 5-7: Stress-Strain Curve for 3608 Conditioned in ASP at 75°C

5.2 Ultimate Tensile Strength

5.2.1 Results

The ultimate tensile strength for each combination of pipe grade and test condition was calculated by taking the average value from the samples tested (see Table 5-1 and Table 5-2).

HDPE	Tensile Str		
Grade	Un-Soaked	Cond. 25°C	Difference
PE3608	24.7	24.6	-0.4%
PE4710	25.1	25.7	+2.3%
PE100+	26.6	25.7	-3.3%

Table 5-1: Tensile Strength Comparison of Samples Conditioned at 25°C

Table 5-2: Tensile Strength Comparison of Samples Conditioned at 75°C

HDPE	Tensile Str	Difference		
Grade	Un-Soaked	Cond. 75°C	Difference	
PE3608	24.7	26.1	+5.7%	
PE4710	25.1	n/a ¹		
PE100+	26.6	n/a ¹		

Notes

 Conditioning of PE4710 and PE100+ samples at 75°C was unsuccessful due to failure of end seals.

5.2.2 Analysis and Discussion

Although the small sample sizes do not permit statistical analysis of the data, meaningful conclusions can be obtained from a qualitative analysis of the data. If cracking was experienced on the conditioned samples, a significant decrease in the tensile strength would be expected. The magnitude of the decrease would depend on the crack dimensions (i.e. length and depth) and level of crack interaction (i.e. spacing between cracks). With no cracking present, the tensile

strengths of the conditioned samples should be similar, but may decrease slightly (<1 - 2%) due to absorption and/or creep from being pressurized.

Review of the tensile measurements showed that the PE4710 conditioned at 25°C and the PE3608 conditioned at 75°C showed an increase in tensile strength of +2.3% and +5.7% respectively. The increase in the measured tensile strength is attributed to the variability of the test measurements and is not significant. Tensile measurements of a given pipe grade and test condition routinely varied by 1.5 MPa (approximately +/- 3% of the yield strengths). It is believed that the variability is caused by a combination of sample preparation, clamping in tensile tester and inherent tensile variability in pipe material introduced during the extrusion process (the wall thickness at the bottom of the pipe during the extrusion process may be marginally thicker due to gravitational influences).

The PE3608 and PE100+ conditioned at 25°C showed a slight decrease in tensile strength of -0.3% and -3.3% respectively. Given the similar magnitude of change, the slight decrease in tensile strength is attributed to variability of the test measurements and not the presence of cracking. A substantial decrease in tensile strength would be expected if cracking was experienced on the conditioned samples. As a result, it is concluded that the tensile measurements present no evidence of environmental stress corrosion cracking (SCC) from immersion in the ASP fluid.

5.3 Elongation at Break

5.3.1 Results

The elongation at break for each combination of pipe grade and test condition was calculated by taking the average value from the samples tested (see Table 5-3 and Table 5-4).

HDPE	Elongation a	Difference	
Grade	Un-Soaked	Cond. 25°C	Difference
PE3608	65%	57%	-8.0%
PE4710	47%	51%	+4.0%
PE100+	57%	48%	-9.0%

Table 5-3: Elongation at Break Comparison of Samples Conditioned at 25°C

Table 5-4: Elongation at Break Comparison of Samples Conditioned at 75°C

HDPE	Elongation a	Difference	
Grade	Un-Soaked	Cond. 75°C	Difference
PE3608	65%	45%	-20%
PE4710	47%	n/a ¹	
PE100+	57%	n/a ¹	

Notes

(2) Conditioning of PE4710 and PE100+ samples at 75°C was unsuccessful due to failure of end seals.

5.3.2 Analysis and Discussion

The immersion of HDPE normally causes a discernible decrease in the elongation due to the absorption of the fluid. The elongation decreases with increasing temperature and increasing hydrocarbon solvency of the liquid (e.g. aromatic solvents such as benzene or toluene). Under normal operating conditions, the decrease in elongation does not affect the integrity of the pipelines in the oil and gas industry as HDPE is very flexible compared to other non-metallic pipe materials.

Environmental stress corrosion cracking (SCC) of HDPE causes the material to become brittle resulting in very large decreases in elongation. Testing of HDPE coating with stress corrosion cracking (SCC) shows that the elongation at break decreased by nearly an order of magnitude.

Review of the samples conditioned at 25°C showed that the elongation at break decreased on the order of 5% – 10% for PE100+ and PE3608.The decrease is consistent with the author's experience from industry and the immersion testing in Section 3. As expected, the PE3608 conditioned at 75°C showed a larger decrease in elongation due to the higher immersion temperature. The elongation at break of PE4710 conditioned at 25°C increased by 4%. This is attributed to experimental error resulting from the variability of the testing methods and material. Overall, the decreases in elongation at break are considered normal for the immersion conditions. It is concluded that the elongation at break measurements show no evidence of environmental stress corrosion cracking of the HDPE material from the ASP fluid.

6.0 CONCLUSIONS

The aim of this project was to study to environmental stress corrosion cracking (SCC) resistance of high density polyethylene in alkali surfactant polymer (ASP) service conditions. Due to the industrial nature of this project, the experimental test methods closely represented pipeline operating conditions such that the experimental results would be directly applicable to industry. The mechanical testing showed no evidence of environmental stress corrosion cracking of the HDPE pipe materials from the ASP fluid. As a result of this research, oil and gas operators will be able to maximize the corrosion resistance benefits associated with HDPE pipe materials to improve the reliability and performance of the large pipeline networks required for ASP flood systems.

Pressurized immersion testing of full scale HDPE pipe samples was conducted in ASP solution representative of actual pipeline operating conditions. Due to the accelerated nature of the testing (3 month soak periods), the specific test parameters were selected to be more severe than actual operating conditions. The concentration of the surfactant utilized in this experiment is two to three times more concentrated than ASP systems in Western Canada. Similarly, the test pressures were 1.25 - 1.50 times higher than those allowed by North American pipeline regulations.

Tensile testing was performed on the conditioned and unconditioned HDPE pipe samples to investigate for the presence of stress corrosion cracking. The mechanical properties of the respective samples were compared to determine the presence of any cracking.

Measurement of the yield strength showed no significant difference between the unconditioned and conditioned samples. Any variability in the test results was attributed to experimental error. The conditioned samples showed a decrease in elongation at break compared to the unconditioned samples. The elongation of samples conditioned at 25° C decreased by 5% - 10%, while the elongation of the sample conditioned at 75° C decreased by 20%.

These decreases in elongation at break are considered normal for immersion testing and are attributed to absorption of the fluid, not SCC cracking of the HDPE material. Environmental stress corrosion cracking of the HDPE should cause it to become significantly more brittle resulting in much larger decreases in elongation at break than which were observed.

Overall, the experimental results showed no evidence of environmental stress corrosion cracking of the HDPE pipe materials resulting from pressurized immersion in ASP fluids. These results are directly applicable to the oil and gas industry and the information can be incorporated into the design and operation of safe and reliable pipeline systems for ASP flood systems.

7.0 FUTURE WORK

Due to the industrial nature of this research, the experimental results had to be directly applicable to industry. As a result, testing was performed on full scale pipe samples that closely resembled actual pipeline operating conditions. However, full scale pipe sample testing is expensive and time consuming and not practical for testing small changes to ASP chemicals or process conditions.

As a result, it is recommended that HDPE pipe materials be tested utilizing accelerated standard test methods modified to incorporate ASP fluids. These test methods utilize artificial conditions to increase the severity of the test environment. These styles of test methods can generally be performed quicker and with less resources then full scale test methods. The main disadvantage to these styles of test methods is they are generally not directly relatable to real world conditions. However, with the full scale pipe sample testing already completed the results from the accelerated standardized testing could be cross referenced to the full scale testing make the results more directly applicable.

7.1 Testing Proposal

It is proposed that testing of two (2) standard test methods modified to incorporate ASP fluids be conducted to help evaluate the environmental stress corrosion cracking resistance of HDPE materials. The test methods are:

- Bent Strip Test (ASTM D1693)
- Full Notch Creep Test (ISO 16770)

The duration of the Bent Strip Test (ASTM D1693) can be impractically long for newer HDPE grades with higher cracking resistance such as PE100+. Despite this limitation, it is still the most common method for evaluating the environmental stress corrosion resistance of HDPE pipe materials in North America.

To ensure adequate evaluation of the newer HDPE pipe grades, it is proposed that ISO 166770 Full-Notch Creep Test (FNCT) be conducted concurrently with 44

the ASTM D1693 Bent Strip Testing. The North American pipeline industry is more familiar and comfortable with the Bent Strip Test (despite it's limitations). Conducting the tests concurrently will help industry understand and adopt the results more effectively. Combining the results from this research with accelerated standard testing in more severe environments should provide the oil and gas industry with sufficient information to feel comfortable with the resistance of HDPE pipe materials to environmental stress corrosion cracking from ASP flood systems.

7.2 Bent Strip Test (ASTM D1693)

This test was the dominant QA/QC test for polyethylene materials in the 1960s and 1970s and is still commonly used in evaluating North American HDPE resin grades. The test involves cutting 10 rectangular samples notched longitudinally on the surface. The specimens are then bent into an 180° arc and placed in a metal specimen holder. The entire assembly is immersed in a specified solution (see Figure 7-1).



Figure 7-1: Bent Strip Test Sample, Holder and Test Assembly

The specimens are examined after certain periods of time and the percentage of the failed specimens is recorded. The test duration varies from 24 hours to 1000 hours depending on the HDPE material. The bent strip test is simple and easy to perform. However, significant stress relaxation occurs during the test, and rate of stress relaxation is difficult to quantify. Although the test is still commonly used, it has been found to be insufficient to distinguish the SCR property of current HDPE materials.

7.3 Full-Notch Creep Test (ISO 16770)

The full-notch creep test (FNCT) was developed by Nishio et al. (1982), and is the preferred test method in Europe due to its shorter failure time compared to the PENT test. The FNCT specimen is a square section of 10x10mm bar with four coplanar notches 1.5mm, made by a razor. The test is performed in a liquid environment at 80°C under a single stress level. The test is suitable to evaluate the latest pipe resins, such as PE100+, because it takes a few hundred hours to fail the specimens, compared to a few thousand hours for traditional test methods.

7.4 Future Testing of Other ASP Surfactants

Although the results show no evidence of environmental stress corrosion cracking from the ASP fluids, one series of immersion testing is insufficient to fully understand the environmental stress corrosion cracking resistance of HDPE pipe in ASP service. As ASP systems change and evolve, it will be important to understand the cracking resistance of HDPE under more severe and diverse process conditions.

If the prior accelerated standardized testing is performed under similar conditions to the full scale pipe testing the results will be more relatable to the actual performance of the material in ASP flood systems. With the ability to relate accelerated testing to actual performance, the chemicals and conditions can be readily varied to test any combination of conditions that may result as ASP flood systems become more common in North America.

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