

Stress-Cracking Resistance of Bi-modal PE-RT HDPE Geomembrane

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ABSTRACT

Stress crack resistance (SCR) of high-density polyethylene (HDPE) geomembranes, with densities higher than 0.94 g/cc, has been widely researched and documented. Over the years, PE resin technology has evolved and has helped to improve many long-term performance properties, including SCR. In this study, resistance to stress-cracking of bi-modal polyethylene raised temperature (PE-RT) HDPE was assessed and compared to HDPE and LLDPE geomembranes over a load range from 28 to 60% of yield stress. The higher tensile resistance of this specific bi-modal resin resulted in higher resistance in the ductile failure mode than standard HDPE, but moreover, its stress-cracking resistance behavior was found without any clear transition of the failure envelope from a ductile to a brittle behavior.

This observation suggests a longer projected lifespan of this unique bi-modal PE compared to existing geomembranes developed using uni-modal resins. Previous studies on this material have analyzed tensile, antioxidant depletion, and resistance to brine and chlorine at elevated temperatures. In all cases, an improvement in durability was demonstrated. This study on stress cracking provides a comprehensive understanding of all known key mechanisms contributing to bi-modal PE geomembrane durability.

INTRODUCTION

This paper describes the stress crack testing that was performed on a geomembrane made with a bi-modal HDPE resin. It is well documented that stress cracking in geomembranes is caused by tensile stresses lower than their short-term mechanical strength. Stress cracking is essentially a brittle cracking phenomenon that occurs at a constant stress lower than the short-term yield strength of the material (Peggs, 2003).

Most stress crack resistance testing on bi-modal resins has been performed on pipe grade resins that are reported to have high resistance to the primary exposures that lead to failure in polyethylene gas piping systems. Notable material properties that are used as performance indicators of pipe robustness are 1. slow crack growth (SCG) resistance and 2. rapid crack growth (RCG) resistance. Resistance to defects and external damage is another property tested by pipe manufacturers to evaluate the robustness of bi-modal resins for high performance pipe applications. These tests "as is" can't be extended to geomembranes primarily due to product type and the end use applications.

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In 2014, the authors started a research project to see if any of these new bi-modal pipe resins had the potential to be used as a geomembrane. While the pipe industry testing looked very good, there was no correlation that would show how a geomembrane would work with the same resin. This is the fifth research paper on the development of bi-modal resins for geomembrane applications.

NCTL TESTING

Notched Constant Tensile Load test is a standard test to evaluate stress cracking under a constant tensile load condition in an accelerated environmental condition. In this test, dumbbell shaped specimens are notched to 20% of their thickness and are subjected to stress between 20-50% of the geomembrane's tensile yield strength as measured using ASTM D6693. The specimens are then placed in a solution containing surface-active agent and tested at 50°C. These accelerated test conditions cause the material to develop crack growth and fail. A plot of the percent yield stress versus the average failure time of the three tests at each load is shown in Figure 1 below (GRI GM10). The test requires at least 3 points to be in the ductile region of the curve, and at least 3 points shall be in the brittle region of the curve. The transition between ductile and brittle failure indicates the stress crack resistance of the material.

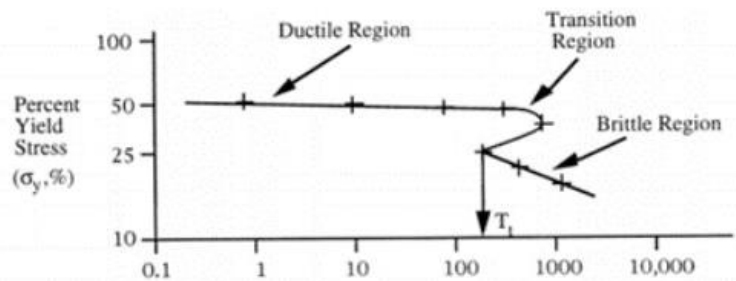


Figure 1. Transition from ductile to brittle failure, adapted from Hsuan, 1995.

BI-MODAL POLYETHYLENE TECHNOLOGY

In general, the molecular weight distribution of the polymer determines the polymer properties (plasticpipe.org). The bottom axis shows the molecular weight (the length of each chain). The vertical axis shows the weight fraction. These graphs usually create a classic bell curve (normal distribution in statistics). A uni-modal distribution, on the left of Figure 2, shows a single peak distribution.

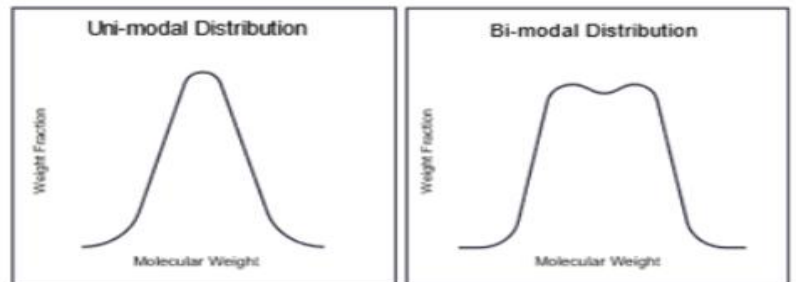


Figure 2. Uni-modal and bi-modal distribution.

The resin manufacturer can control the reactor conditions and change the catalysts to control the kind of polymer that is made. A very narrow distribution will be strong, stiff, and difficult to push through plastic processing equipment (all other properties being equal). A very wide distribution (graph is shorter and wider at the base) will normally be softer and more pliable, and easier to process. Current HDPE geomembrane resins are made with unimodal resins.

Over the years, stress cracking issues have led resin manufacturers to reduce the density of their resins and widen the molecular weight distribution. The result is that current HDPE geomembranes are made with medium density (MDPE) resins. While this has reduced the risk of stress cracking, it has resulted in the reduction of chemical resistance. When considering ESCR as a failure mechanism, in the traditional unimodal sense, the lower the density generally led to better performance (less stress cracking risk). This results from a higher amount of comonomer incorporation into the polymer backbone. Unfortunately, the higher the amount of comonomer, the lower the density; thus, lower chemical resistance.

Newer resins have been developed using a technique that creates a bi-modal molecular weight distribution. In Figure 2, a bi-modal distribution shows two distinct peaks on the graph. Bi-modal resins are generally made utilizing dual reactors. The first reactor has the feedstock, catalyst and comonomer injected into it, where a polymer is formed with a tremendous amount of comonomer.

That material is then transferred to a second reactor operating in a comonomer-starved condition where the polymer continues to build the homopolymer backbone. The result is a more robust product with a very low risk of stress cracking.

The research into bi-modal PERT HDPE geomembrane has included high temperature tensile strength testing (Beaumier et al 2016), high service temperature testing (Mills and Beaumier, 2017), chlorine resistance testing (Rangel et al 2017), and brine resistance testing, (Mills et al 2019).

MATERIAL AND METHODS

The main material investigated in this evaluation was a bi-modal PERT geomembrane. Since this was the first time a bi-modal PERT geomembrane resin was evaluated for stress cracking performance, the authors felt it was necessary to compare its performance with other polyethylene types commonly used for the manufacturing of geomembranes. The following geomembranes were part of this study:

- GM13 complaint HDPE
- GRI GM 17 complaint LLDPE
- Bi-modal PERT HDPE

Table 1 provides a summary of the physical properties of the material tested in this study.

Table 1. Material Properties of geomembranes tested.

Material	Density ¹ , g/cm ³ (nominal range)	Thickness, mm (mils)	Tensile strength ² , psi (yield)
HDPE	0.940 – 0.955	1.5 (60)	1754 / 1753
LLDPE	0.910 – 0.939	1.5 (60)	2676 / 2667
PE-RT HDPE (bi-modal)	0.940 – 0.955	1.5 (60)	3378 / 3564

Notes to Table 1:

1. GRI-GM13 and GRI-GM17 does not establish density range but a cut-off value for LLDPE, 0.939 g/cm³ and under, or HDPE, 0.940 g/cm³ and over.
2. Tensile strength is reported respectively in machine and cross machine directions.

TEST METHODOLOGY

The resistance to stress cracking was determined in accordance to ASTM D5397, including a complete evaluation of test loads, test loads were selected between 28-68% of the measured stress at yield which was determined as per ASTM D6693. Table 2 summarizes the test loads used for this study. Samples were retrieved at different intervals and evaluated for stress crack resistance. The last sample tested was exposed to 10,000 hours before being tested for SCR. Images by scanning electron microscopy (SEM) on the surface was carried out to determine brittle or ductile failure.

Table 2. Test loads for NCTL testing

Load	LLDPE		HDPE		PE-RT HDPE		PE-RT HDPE	
	(tested in CMD)		(tested in CMD)		(tested in CMD)		(tested in MD)	
	psi	% y.s	Psi	% y.s	psi	% y.s	psi	% y.s
#1	526	30	747	28	1069	30	1013	30
#2	614	35	800	30	1247	35	1182	35
#3	701	40	853	32	1426	40	1351	40
#4	789	45	933	35	1604	45	1520	45
#5	877	50	1067	40	1782	50	1689	50
#6	964	55	1200	45	1960	55	1858	55
#7	1052	60	1334	50	2138	60	2027	60
#8	1139	65	1467	55	2317	65	2196	65

RESULTS AND DISCUSSION

NCTL Tests

Figure 3 describes the relation between the load in percent of the measured yield stress to failure time. This type of analysis induces a bias from the initial resistance to tensile stress. On an absolute basis, Figure 4 presents the resistance to stress crack propagation in terms of tensile stress, in psi.

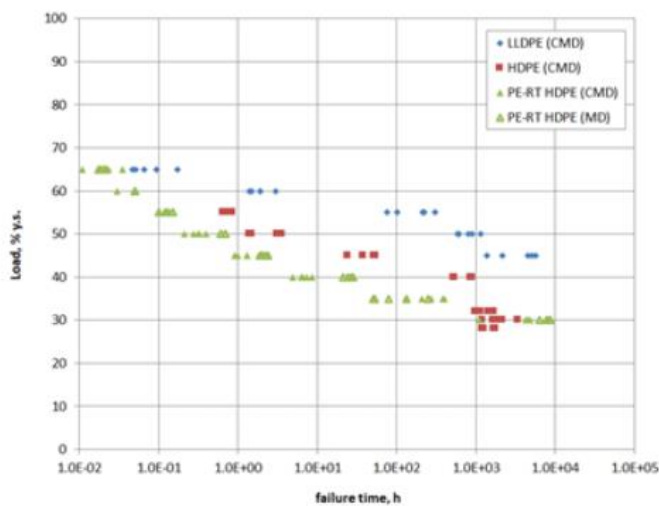


Figure 3. Comparison of geomembrane types to stress crack propagation, in terms of percent yield stress.

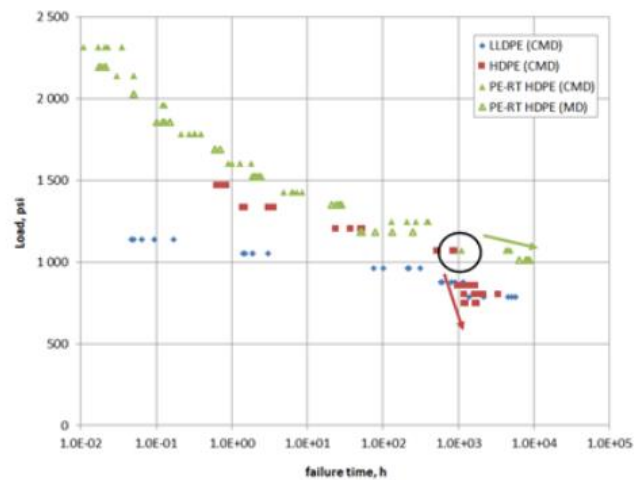


Figure 4. Comparison of stress crack propagation of geomembranes under tensile load.

This graph shows a more realistic picture of the resistance of long-term tensile stress and stress-crack propagation. The LLDPE shows lower stress resistance because its resistance to a ductile failure mode remains lower than the stress crack propagation of HDPE.

In addition to its greatest resistance to failure in the ductile region, bi-modal PE-RT HDPE shows a completely different trend of its failure time to tensile load after reaching its ductile-brittle transition when compared to standard grade HDPE, shown in the circle in Figure 4.

The mode of failure was confirmed from images under electronic microscopy. An apparent textured surface is observed when brittle failure occurs by stress-crack propagation. A wavy surface likely correlates to ductile failures. In this study, no failure by stress-cracking was observed with LLDPE. The ductile to brittle transition was evaluated at 35% of HDPE yield stress. For PE-RT HDPE it was evaluated at 30% of its yield stress. Figure 5 presents the surface indicating failure at the ductile-brittle transition for each material.

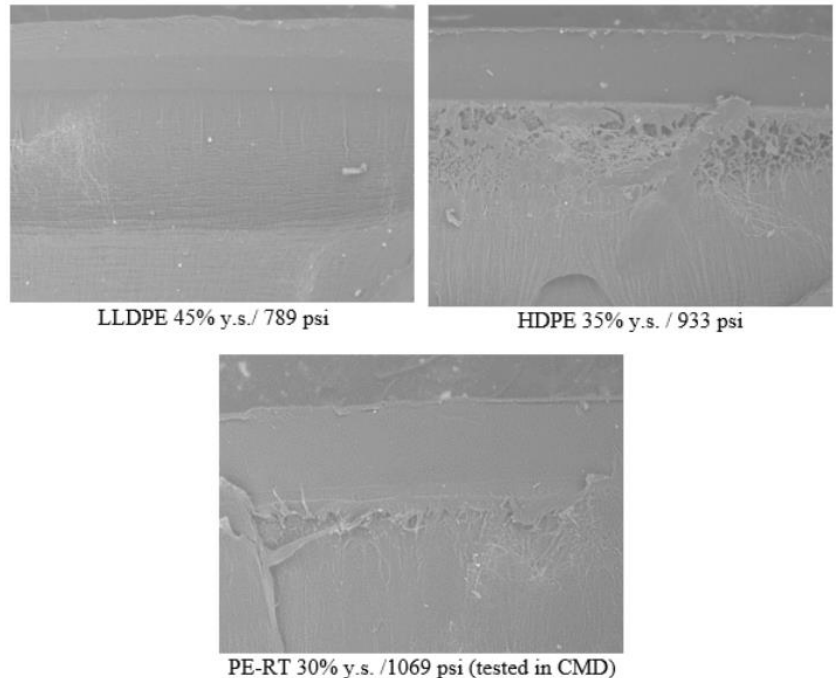


Figure 5. Images of rupture faces by scanning electron microscopy.

DISCUSSION

The behavior of PE-RT HDPE to ASTM D5397 describes three possible NCTL response curves, as shown in Figure 6. These are the bi-linear or knee curve, the overshoot or nose curve, and the tri-linear or step curve.

Figure 6. Evaluation of ductile-brittle failure adapted from ASTM D5397.

While variability in testing data makes it difficult to determine which of these curves may apply, the more important goal is to identify the transition point between a brittle failure and a ductile failure. In all the curves shown, it is the transition in the slope of the line in which we are most interested.

Identifying the transition point in our data (highlighted in Figure 1) is complicated by the variability of the data. Fitting curves to the data, it is still not immediately clear enough to draw a conclusion from trends on the location of the ductile-brittle transition. However, the transition point may be assessed from a microscopic view of the specimen rupture faces, which may provide evidence of ductile or brittle failures, shown in Figure 3. The use of microscopy allowed us to find the transition times for each material, this transition can then be looked upon on the graph to see if the data on the graph are close to that transition time. For bi-modal PE-RT HDPE, this transition was showing an inflection opposite to the ones proposed by ASTM D5397.

CONCLUSION

The assessment of stress-crack resistance of PE-RT bi-modal HDPE was done using ASTM D5397, and compared to reference materials commonly used for geomembranes. Whereas no stress-cracking failures were observed with LLDPE, both HDPE grades have shown a transition from ductile to brittle behavior under accelerated long-term tensile stresses. The bi-modal PE-RT HDPE grade has shown longer time to ductile-brittle transition, and failure by stress-cracking than the reference HDPE. The findings of this study are in accordance with previous studies on the aging of bi-modal polyethylene grades for its usage in the geomembrane applications, including high temperature tensile resistance, antioxidant depletion, and resistance to brine and chlorine at elevated temperatures. In all cases, an improvement in durability was demonstrated with bi-modal HDPE.

ACKNOWLEDGEMENT

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