

Stress Cracking Behaviour of a High Density Polyethylene Geomembrane Seam Created in Sub-Zero Conditions.

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ABSTRACT

High-density polyethylene (HDPE) geomembranes are used extensively in barrier systems for landfills and mining applications. Shipped as rolls these geomembranes require in-situ seaming to create a continuous barrier against liquid and gas migration. The most commonly employed seaming techniques for HDPE geomembranes include dual track wedge welding and extrusion welding, both of which subject overlapped sections of geomembrane sheet to high temperatures and molten polymer during seaming. Reports have indicated cracking failures at the heat-affected zone (HAZ) for both dual track wedge welds and extrusions welds created during sub-zero temperatures, prompting interest into the examination of a geomembrane's susceptibility to stress cracking following cold weather seaming. Stress crack resistance (SCR) tests of one HDPE seam, in both notched and un-notched configurations, and sheet specimens are reported. The craze formation and failures times of a seam created at a sheet temperature of -27°C for one welding speed, pressure, and welding temperature combination, as well as rapidly cooled extrudate and seam squeeze-out crystallinity tests are examined. Notched SCR results indicate little difference between -27°C seam and sheet values while un-notched seams exhibited a significantly shorter failure time (2895 ± 64hrs) than un-notched sheet, which have yet to fail after ~8000hrs. Crystallinity results suggest that material embrittlement did not occur within the seam. Furthermore, rapid cooling, specifically in terms of material crystallinity, may actually be beneficial in hindering crystalline region growth in HDPE GMBs.

1. INTRODUCTION

High-density polyethylene (HDPE) geomembranes (GMBs) are generally shipped to site in ~6.8-8 m wide rolls that range significantly in length depending on requested material thickness. Often the facility requiring a hydraulic barrier is too large to install a prefabricated GMB and consequently, in-situ seaming must be performed. Rolls are deployed on a prepared subgrade, often a compacted clay or soil layer poor in coarser material and, in the case of HDPE, seamed together using thermo-fusion methods. This technique works by passing a hot metal wedge between two overlapped sections of deployed GMB panels. The welding machine propels itself down the length of the seam automatically applying heat and pressure creating one continuous weld. Wedge temperature, machine speed and the seaming pressure are all parameters controlled by the equipment operator. Typically, wedge temperatures fall between 400-460°C, and machine speeds between 1-3m/min (Müller, 2007; Scheirs, 2009; Zhang et al., 2017). These parameters are changed to suit the current environmental and sun exposure conditions, as improper calibration of the equipment is known to increase the likelihood of faulty seams. Currently there is little published work focusing on the performance of HDPE geomembrane seams, however, several recent published papers have examined particular aspect of their performance. One recent study conducted by Rowe and Shoaib (2017) found that the material directly adjacent to the weld, referred to as the heat-affected zone (HAZ), may age more rapidly than the sheet. This poses a potentially significant problem as the dual track seams can reach length of ~1500m/ha in a typical landfill. Zhang et al. (2017) reported irregular HAZ thermograms and Std-OIT potentially falling between 5%-95% of virgin sheet values in seam squeeze-out, suggesting non-uniform antioxidant loss during welding and sample heterogeneity. In another paper, by Kavazanjian et al (2017), digital image correlation (DIC) was used in an experimental evaluation of HDPE seam strain concentration resulting from seam rotation when loaded in tension. It was concluded that the average stains calculated via DIC fell reasonably close to the predicted strains calculated by Giroud et al (1995). However, the maximum strains adjacent to the seams were found to be significantly greater, as much as 1.4 to 2.0 times those predicted by Giroud et al. (1995). With evidence of accelerated chemical degradation and a means through which to concentrate strain at the HAZ, seams serve as critical location in regards to performance of HDPE GMB barriers.

HDPE GMB seams, both wedge and extrusion welds, serve as a potential zone for stress cracking failures (Thomas and Woods-DeSchepper, 1993; Peggs, 1997; Scheirs 2009; Peggs et al., 2014). This is due in part to seam rotation concentrating strains along the HAZ, but also hypothesized to be influenced by a sudden change in crystallinity between the weld and sheet material (Peggs and Carlson, 1990). Cracks are thought to be initiated by the formation of a craze along the bottoms sheet's HAZ and propagate perpendicular to the applied stress. In this case, seam rotation would exacerbate the situation by increasing the resulting strains at the HAZ's craze, further accelerating cracking. Peggs and Carlson (1990) examined the stress crack resistance (SCR) performance of both dual track wedge welds and extrusion fillet seams using notched constant tensile load (NCTL) SCR tests (in accordance with GRI GM5(c)). Dual track and extrusions fillet seams exhibited as much as a 46% and 56% decrease in SCR when compared to sheet. In another study (Halse et al., 1989), HDPE sheet was found to resist stress cracking better than welds, where 1% of sheet material and



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38% of seamed material exhibited cracking. The type of crack growth captured in these tests is referred to as slow crack growth (SCG). This failure mechanism is hypothesize to work as a slow disentanglement or breaking of interlamellar tie molecules within the polymers amorphous phase (Lustiger and Markham, 1983; Lustiger and Rosenberg, 1989). Another mechanism, referred to as rapid crack propagation (RCP), produces a "shattering" like appearance in the GMB and is characterized by crack propagation rates of over 300m/s (Hsuan, 2000). Although this exact failure mechanism is not as well understood for HDPE GMBs as it is for HDPE pipe, it is hypothesize to occur as a result of preliminary SCG achieving a critical geometry and growth rate, ultimately leading to RCP (Peggs et al., 2014). This phenomenon has been recently reported to occur in sub-zero conditions initiating at the location of both extrusion and dual track wedge welds (Peggs et al., 2014). This paper intends to gain insight into the effect seaming in sub-zero conditions may have on the cracking potential of HDPE seams. Seam SCG and seam crystallinity, both considered factors influencing RCP, will be examined for a dual track wedge weld created in sub-zero conditions. The results of which are intended to help gain insight into the driving factors influencing RCP in exposed GMBs.

2. MATERIALS AND METHODS

2.1 Geomembrane Properties and Seaming

The relevant properties of the 1.5mm HDPE geomembrane examined can be found in Table 1. Sub-zero seam samples were welded in an environmental chamber set to -27°C. Samples were conditioned to ambient temperature 24 hours prior to welding within the environmental chamber, allowing sheet temperatures to reach equilibrium. GMB temperature was measured using an infrared thermometer reporting values to an accuracy of +/- 1°C. Two 30cm x 200cm samples were cut from a 1.5mm GMB and welded by an experienced technician using a Demtech Pro-Wedge series wedge welder. Wedge temperature and speed settings were set to 460°C and 1.8m/min, respectively. In order to obtain seam samples more representative of the welding parameters examined, SCR and crystallinity specimens were taken towards the end of the 2m long seam.

Table 1: Relevant properties of the HDPE GMB examined.

Property in Question	Method	Unit	Value
Nominal thickness	ASTM D 5199	mm	1.5
Manufacturing date			2011
Manufacturing method			Flat die
Standard oxidative induction time (Std-OIT)	ASTM D 3895	min	185 ± 2.1
High-pressure oxidative induction time (HP-OIT)	ASTM D 5885	min	1321 ± 12
High-load melt index	ASTM D 1238	g/10 min	21.5 ± 0.2
Sress crack resistance (SCR)	ASTM D 5397	hours	1078 ± 83
Yield strength used for SCR	ASTM D 6693	kN/m	29.3

2.2 Seam Stress Crack Resistance Testing

Sheet and seam SCR testing was performed in accordance with ASTM D5397 and GRI GM5(c), respectively. All testing procedures utilized the same applied load and testing conditions, where samples are hung in tension while submersed in a 10% Igepal CO-630 solution held at 50°C. Figure 1 illustrates the two configurations of HDPE seam SCR tests examined. Where the notched configuration positions a 20% nominal thickness notch on the outside face opposite the bottom sheet HAZ, and the un-notched configuration forgoes the use of a notch and instead adjusts the applied force to suit sample dimensions. Both notched and un-notched samples were subjected to a load equal to 30% the material yield strength for their given cross-sectional area. Calculation of the applied force for testing is as follows:

applied force =
$$(A)(\sigma_y)(w)(t_L)(1/MA)$$

(1)

Where: *A* is the percentage of yield stress examined, σ_y is the room temperature yield stress, *w* is the neck width of the test specimen, t_L is the ligament thickness of the test specimen, and *MA* is the mechanical advantage of the test apparatus. All variables of equation 1 were considered the same for notched and un-notched samples, except t_L , which was set to 80% and 100% the nominal thickness for each case, respectively. Notching seamed samples was found to be particularly difficult as the opposing faces of the seam SCR samples are often slightly off parallel, in turn making it difficult to notch at an angle perpendicular to the outside face of the sheet material. Furthermore, GRI GM(c) does not specify a tolerance for the position of the notch relative to the seam-sheet interface. To the best of the operator's ability, notches were positioned no more than 0.2mm away from the edge of the seam on the sheet material. Furthermore, all notches created were located beneath the squeeze-out of the seam.



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Figure 1: Configuration of seam SCR specimens examined. Where the notched variant contains a notch equal to 20% the materials nominal thickness opposing the seams bottom sheet HAZ.

2.3 Crystallinity Examination

Polymer crystallinity, or the percentage of crystal lamellae in the polymer structure, is a common index parameter used in charactering HDPE GMBs. The degree of crystallinity for the geomembrane samples examined was tested in accordance with ASTM E793 using a TA instruments Q-2000 series differential scanning calorimeter (DSC). Sections of geomembrane, cut with a punch, were weighed and encapsulated in aluminum pans before loaded into the DSC. Calculation of crystallinity was performed as follows:

$$X_{c}\% = \frac{\Delta H}{\Delta H_{100}} * 100$$
 (2)

Where: X_c % is the degree of crystallinity in %, Δ H is the enthalpy of fusion (or crystallization) found through integrating the area of the melting curve (or crystallization curve) between heat flow baselines of the DSC thermogram, and Δ H₁₀₀ is the enthalpy of fusion of 100% crystalline polyethylene, assumed to be 290 J/g (Wunderlich and Cormier, 1967; Choudhury et al. 1989). 5 replicate samples were taken for each of the seam squeeze-out samples examined, followed by 10 replicate samples for both sheet and extrudate crystallinity values.

3. PRELIMINARY RESULTS

3.1 Notched and Un-notched Stress Crack Resistance

Notched seam SCR values were similar to that of notched sheet values (Table 2). Sheet samples failed with an average failure time of 1078hrs while seamed samples failed with an average SCR time of 1018hrs, both representing an average of 5 samples. The range of values observed for seamed samples were much greater, with a standard deviation of 289hrs, suggesting a potential cause of greater variability. This is likely attributed to the difficulty in notching seamed samples. Unlike sheet samples, which have two parallel faces providing ease in notching, seam samples tend to have two outer faces that are slightly off parallel, making notching more difficult as one face of the sample must rest on the notching mount. Furthermore, the difficulty in consistently positioning the notch below the seam-sheet interface may have also contributed to sample variability. Despite a slightly lower SCR value for the notched seam compared to the notched sheet, means are still within one standard deviation of each other, suggesting an effectively similar mean for both notched sheet and notched seam.

Table 2. Stress crack resistance for notched and un-notched seams and sheet.

Sample Type	Stress Crack Resistance (hours)
Notched Sheet	1078 ± 83
Notched Seam ¹	1018 ± 289
Un-notched Sheet	> 8000 hrs
Un-notched Seam	¹ 2895 ± 64

¹Seam created using the following welding parameters: Sheet temperature at time of welding, -27°C; wedge temperature, 460°C; welding speed, 1.8m/min.



Un-notched seam failure times were found to be \sim 3 times greater that of notched seams, with an SCR value of 2895 ± 64hrs. However, this value represents both craze formation along the HAZ as well as crack propagation time across the GMBs thickness. Interestingly, un-notched sheet samples have yet to fail after 8000hrs of testing, suggesting a seam failure time less than or equal to \sim 35% that of the un-notched sheet values, or in other words, a sheet failure time \sim 2.75 times greater than that of the seam. All un-notched seam samples exhibited failure in the material directly adjacent to the seam, were craze formation started from the location of the seam-sheet interface and SCG propagated outwards towards the outside face of the sheet material opposite the HAZ (Figure 2).



Figure 2: Microscope images highlighting the failure locations and surfaces of both notched and un-notched seam samples. Note the blurry portion of images b) and d), where the extent of ductile detachment made it difficult to focus on both brittle and ductile regions.

Figure 2 highlights the failure locations and resulting brittle detachment areas of both notched and un-notched seams samples. Where images a) and c) illustrate the relative position and direction of crack propagation for notched and un-notched samples, respectively, and images b) and c) provide a close up of the brittle detachment regions. Both notched and un-notched samples exhibited brittle detachment leading to a reduction in cross sectional area and an eventual plastic failure. This is consistent with what was observed in the literature (Peggs and Carlson, 1990; Halse et al., 1989; Halse et al., 1990). Interestingly, in the notched specimen, crack propagation initiated close to the notch itself and extended towards the HAZ. As a result, the HAZ area of the notched sample was included within the ductile detachment region of the sample. This suggests that potentially any material degradation within the HAZ would likely not affect the rate of crack propagation across the sample, as the potentially degraded material would only influence the rate of ductile failure, which is comparably shorter in duration. Conversely, all un-notched seam SCR samples exhibited crack propagation like that shown in Figure 2 c) and d), where craze initiation and crack propagation all start at the HAZ and extend outwards. This suggests that the failure time for un-notched samples may better reflect the effect seaming has on stress crack resistance, as the potentially degraded HAZ is included within the brittle region. Clearly there is a significant difference in the failure time between un-



notched sheet and un-notched seams, a difference which was not effectively shown in notched samples. Further work should be conducted to see under what conditions one might expect notched samples to exhibit a difference in failure time like that of un-notched seams and sheet samples. Although given the relative difficulty in consistently notching seam samples, it seems that un-notched seam SCR tests serve as more reliable testing metric for comparing sheet and seam SCR.

3.2 Extrudate and Squeeze-out Crystallinity

The degree of crystallinity has been suggested to influence the materials susceptibility to SCR, where the higher the proportion of crystal lamellae the greater the likelihood of SCR susceptibility (Scheirs, 2009; Peggs et al., 2014). Crystallinity results were examined to identify if the degree of crystallinity, within the squeeze-out bead of a seam, increased relative to the sheet material. The squeeze-out bead was examined as it is expected to have experienced a greater degree of melting compared to the HAZ, suggesting any crystallization which may result from heating and subsequent cooling would be exaggerated relative to the HAZ. A second seam, of the same parent GMB, was added to examine the effect of a prolonged cooling period on the squeeze-out bead crystallinity. This seam, labeled "Overheat Seam", was welded in the field during a sunny day when sheet temperatures were recorded to be as high as 65°C. The seam was later left to cool slowly for 1 hour in the midday sun and brought back to 21°C storage in the evening. Wedge temperature and speed settings were set to 400°C and 1.7m/min, respectively.

Similar to the "Sub-zero" and "Overheat" seams, an exaggerated case was examined. Molten extrudate from melt index testing equipment, using the same parent GMB as the seams, was both rapidly cooled and oven cooled. ~30ml of GMB chunks were melted in a piston style chamber at 190°C for 240 seconds before being extruded out a small die. In the rapidly cooled case molten polymer exiting the melt index machine was immediately cooled with liquid nitrogen down to -50°C. In the oven cooled case, set to examine the effect of a prolonged cooling duration, molten polymer was removed from the melt index machine and placed immediately in an oven set to 70°C for 3 hours in an attempt to simulate daytime sun exposure temperatures. The extrudate was then moved to a 40 degree oven for 2 hours, and then lastly was left at room temperature overnight. Samples were removed the next day for crystallinity testing. Figure 3 highlights the results of both the extrudate and weld squeeze-out crystallinity tests relative to the sheet material.

Seam squeeze-out crystallinity values calculated were similar to that of the virgin sheet value of $51.0 \pm 1.9\%$. Both the subzero (49.4 ± 1.4%) and overheat squeeze-out ($52.5 \pm 2.1\%$) crystallinity values fall within one standard deviation away from the virgin sheet mean, suggesting seaming, for these particular welds, did not significantly alter the material crystallinity. Interestingly, the oven cooled extrudate sample ($56.0 \pm 1.6\%$) exhibited a significantly greater crystallinity value when compared to the virgin sheet. This is attributed to prolonged cooling allowing greater time for crystal lamellae formation within the polymer (Abdelaal et al., 2015). On the other hand, the rapidly cooled extrudate specimen ($50.8 \pm 1.3\%$) exhibited a mean value almost identical to that of the sheet with a slightly lower standard deviation.





Figure 3: Crystallinity (%) results for the cases examined. Diagonal line texture indicates melt index extrudate samples, while dotted texture represent seam squeeze-out crystallinity.



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These data suggest, for the materials and welding parameters examined, seams created in sub-zero conditions are less likely to experience an increase in material crystallinity than seams welded at warmer temperatures, although more data is need to definitively state the magnitude of this relationship in seams. The exaggerated melt index samples indicate the effect prolonged cooling may have on the molten polymer during welding. However, this method and its thermal history do not match that of the seamed samples. As such, these tests were meant to illustrate the general relationship between polymer melting and cooling rate on crystallinity, not the exact magnitude one should expect in the field.

4. CONCLUSIONS

This paper has described a preliminary examination into the SCR performance of HDPE geomembrane seams welded in sub-zero conditions. Recorded SCR failure times for both notched and un-notched welds, as well as an examination into the crystallinity of a seams squeeze-out have led to the following preliminary conclusions:

- That un-notched seam SCR testing appears to provide a more suitable means for assessing HAZ stress cracking potential. This is due to the incorporation of craze formation time and inclusion of the HAZ into the brittle detachment region. Neither of which was demonstrated in notched seam SCR specimens.
- Un-notched seam SCR specimens can exhibit failure times significantly lower than that of un-notched sheet.
- For the materials and welding parameters examined, no significant difference was observed between sheet and seam squeeze-out crystallinity post-welding.
- Some evidence from oven cooled melt index extrudate suggests that higher material crystallinity may be more likely for prolonged cooled samples than rapidly cooled ones.

In regards to RCP, these data suggest that seaming at sub-zero temperatures likely has a limited effect on altering the crystallinity of dual track wedge welds. It was also shown that the HAZ may serve as a crack initiation point, at which SCG leads to cracking failure adjacent to the seam. Considering these preliminary conclusions, RCP failures are likely more influenced by thermal contraction stresses, strain concentrations, and residual stresses within the weld zone due to rapid seam cooling then an increase in material crystallinity. Future work should be conducted on identifying an appropriate RCP testing metric for HDPE geomembrane and further examination into the behavior of extrusion welds created in similar environments.

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