# *EuroGeo4 Paper number 267* FACTORS THAT AFFECT THE STRESS CRACKING RESISTANCE OF HDPE GEOMEMBRANE LINERS: PART 1 – WHAT WE KNOW AND MIGHT EXPECT

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**Abstract:** The stress cracking resistance (SCR) is the most important mechanical durability parameter of HDPE geomembranes. While all HDPE geomembranes made from different HDPE resins with different co-monomers have almost identical uniaxial tensile strengths and elongations, tear strengths, and puncture resistances, their stress cracking resistances can vary by a factor of 100 or more. Since SCR is a function of the microstructure of the geomembrane it is also a function of the method of preparation of the specimen, and perhaps even the thickness of the geomembrane. In this paper a projection is made of those factors that influence the results of SCR measurements and how they may be assessed. A future paper will describe the actual test data obtained by implementation of the research program proposed in this paper.

Keywords: stress crack, HDPE, geomembrane

# **INTRODUCTION**

Stress cracking (SC) is a fundamental performance characteristic of high density polyethylene (HDPE). It occurs in all HDPE geomembranes to different degrees. It is a function of the semi-crystalline microstructure of HDPE and the co-monomer that is used to tie the crystalline regions together. It is manifested as a quasi-brittle fracture under a constant load below the yield or break stress of the material – similar to a non-cyclic or monotonic fatigue stress. Thus the material we know that yields and elongates like chewing gum to over 700% of its gauge length during an index tensile test can slowly grow a brittle crack (Figure 1) or can shatter like glass (Figure 2) at low stresses. The former is termed Slow Crack Growth (SCG) and is the subject of this paper. The latter is Rapid Crack Propagation (RCP).



Figure 1. Typical SCG cracks on extrusion die marks at subgrade stone protrusion



Figure 2. RCP cracking

RCP can happen as the result of an impact load or, as usually happens, as the result of rapidly dropping temperature as an exposed liner contracts and becomes tensioned. Peggs et al. (1991) hypothesized that a SCG crack of critical size growing at a critical rate is required for RCP to occur. RCP has been measured in HDPE pipes at speeds exceeding 300 m/s, easily passing through butt fusion welds. Geomembrane areas as large as 2 ha have suffered RCP "shattering". Fortunately, there have been no known instances of RCP in HDPE geomembranes for 10 years or more. Resin formulations have become much more resistant to SC.

The characteristic straightness of stress cracks is shown in Figure 3 and the characteristic smoothness of the fracture face is shown in Figure 4. Figure 4 also shows the stop/start nature of crack propagation radiating from the initiation site.



Figure 3. Straightness of stress crack through thickness of geomembrane



Figure 4. Stress crack fracture face.

The ASTM standard to assess stress cracking resistance (SCR) is D5397. It uses a small dogbone specimen with a width of 3.2 mm. On one of the 3.2 mm wide faces a transverse razor blade notch is made with a depth equal to 20% of the thickness of the specimen/geomembrane thickness. Specimens are loaded to different percentages of the yield stress and the time to break is monitored, generating a curve as shown in Figure 5 for five different HDPE geomembranes.

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Figure 5. Stress rupture curves for 5 HDPE geomembranes (Hsuan et al. 1992)

At the higher stresses, above the knee of the curve, the breaks are ductile, but below the knee they are quasi-brittle; brittle on a macro scale, ductile on a micro-scale. In fact, as the stress in the unbroken ligament rises above the knee in the curve the initial brittle crack becomes a final ductile break as shown in Figure 6.



Figure 6. SC specimen fracture face. N-notch, B-brittle break, D-final ductile break

A simple conformance test is performed with five specimens at 30% of the measured yield stress of the material (based on the cross sectional area of the ligament at the root of the notch) to give an assured brittle break below the knee of the curve. In the test the specimen temperature is raised to  $50^{\circ}$ C to accelerate the break time, and the test is performed in an Igepal CO630 surfactant solution to further accelerate the break time. In the Geosynthetic Research Institute (GRI) GM 13 specification for HDPE geomembranes, the initial break time in the single point SC tests of >200 h was increased to >300 h in May 2003, and is now often quoted as >400 h by some geomembrane manufacturers.

In a recent HDPE capping project requiring a 1000 year lifetime, the SCR was specified at 1500 h. No geomembrane manufacturer would guarantee to meet this specification, even though HDPE resins are now exceeding SCR's of 10,000 h. The resin selected for the project had, in fact, been tested to more than 12,000 h without break. The closest a resin manufacturer would informally assure a geomembrane was >900 h. The tested geomembrane had SCRs exceeding 1500 h. Another has measured at >2000h.

In comparison to the test conditions, note that exposed liners in the field can easily reach 80°C under summer sunshine. The acceleration of SC in a chemical environment is known as environmental SC (ESC). Typical chemicals that cause ESC are chlorinated solvents, oxidizing acids, and aromatic hydrocarbons. It should be noted that chemical resistance charts typically do not list compatibility with materials under stress. For materials such as HDPE that are

susceptible to ESC this can be a major omission. For instance HDPE is generally shown resistant to sulphuric acid except at high concentrations and at over 60°C, yet SC of HDPE has been observed at much lower concentrations and temperatures (Peggs 2008).

The apparent differences between resin and geomembrane led to consideration of possible differences within geomembranes and how they might lead to different test results. The following discussions have led to the proposal for a testing program which will be presented within this paper. A second paper will describe the test program performed and its findings and compare them with the projections made in this paper.

The microstructure of the material within the test specimen and at the root of the notch must play a major part in these considerations therefore requiring some understanding of the influence of microstructure on the mechanism of stress cracking. Lustiger (1983) presented the most commonly adopted model for stress cracking as shown in Figure 7. The organized regions of HDPE (about 55% by volume) are separated by amorphous structural regions in which the ends of long molecules (cilia) and loose loops hanging from one crystalline region are entangled in adjacent crystallites. These are termed "tie molecules" whose lengths and distribution are a function of the co-monomer used to improve the performance of the basic HDPE resin. The crystallites are themselves organized radially in spherical groups called spherulites.



Figure 7. Structure of HDPE and model for stress cracking (Lustiger, 1983)

In a relatively fast conventional uniaxial tensile test the tie molecules remain embedded in the crystalline regions tending to pull the crystalline regions apart thereby generating a ductile break with long fibrils of some substance. At the lower constant stresses however the fibrils slowly disentangle thereby generating a relatively smooth interface (the stress crack) between crystalline regions. Thus, stress cracking, as shown in Figure 8, occurs between and diametrically through the crystalline spherulites.



Figure 8. Stress crack through HDPE spherulitic microstructure (Lustiger 1983)

Environmental stress cracking occurs when a specific chemical enters the amorphous regions and lubricates the tie molecules enabling them to be more easily extracted from the adjacent crystalline regions. And when oxidation

occurs, primarily in the amorphous region after the antioxidants have been consumed, chain scission (cutting) of the tie molecules occurs and stress cracking occurs more easily. Therefore, the amount of crystallinity, the sizes of the crystals and spherulites, the lengths, number and frequency of tie molecules, the orientation of the microstructure, and the performance of the antioxidant additives, all contribute to SCR performance.

When geomembranes are tested the specimens are stamped out of the geomembrane itself, so their thickness is the same as the geomembrane. However, the notch depth and the thickness of the remaining ligament vary with geomembrane thickness as shown in Table 1.

Thickness (mm)	Notch depth (mm)	Ligament thickness (mm)
1.0	0.2	0.8
1.5	0.3	1.2
2.0	0.4	1.6
2.5	0.5	2.0

Table 1. Geomembrane SC specimen details

The intent of the notch is to develop plane strain conditions at the root of the notch, the success of which is a function of the width to thickness ratio of the remaining ligament. Thinner specimens will be more successful at achieving this than thicker ones. The stress intensity factor responsible for the initiation and propagation of the crack (notch) is a function of the ratios of notch depth/width and notch width/thickness of ligament both of which will vary with specimen thickness. Therefore, crack growth rates in the same material will vary with specimen/geomembrane thickness.

Tests on resin, thin (<1 mm) geomembranes, and thicker liners, such a 5 mm thick studded concrete protection liners, are made on specimens made by melting fragments of the material and forming a plaque typically 1.0 or 1.5 mm in thickness, according to ASTM D4703, and at a cooling rate of 5, 10, 15, or  $60^{\circ}$ C/min. Clearly, the faster the cooling rate, the lower the crystallinity of the material and the steeper the microstructural gradients from the surface to the interior of the plaque. It is also apparent that the in-plane orientation of the microstructure in a plaque will be much less than that in an extruded geomembrane.

Cooling rate differences may also be a factor on each surface of a round die blown film geomembrane. As the extruded bubble rises circa 23 m from the horizontal die up the tower, the inside surface will likely be cooling at a lower rate than the outside surface thereby generating different microstructural gradients at each surface. These gradients are significant in that they will generate different microstructures at the roots of the notches in the SCR test specimens.

The practical consequence of these differences in the specimens is that a geomembrane that meets specifications in one thickness may not meet specifications in another thickness.

A testing program to evaluate the extent of these differences and to better understand SC is proposed as follows:

- Work with a geomembrane made from resin with an SCR less than 5,000 h.
- Use resin test plaques fast-cooled at 60°C/min and slow-cooled at 5°C/min to compare the effects of cooling rates. The slow cooled specimens will have a higher crystallinity than the fast cooled specimens so should have a lower SCR.
- Test 1.5, 2.0, and 3 mm thick geomembranes made from the same resin with notches on the outside surface of the bubble. Due to the low thermal conductivity of HDPE the microstructural gradients on the surface of the geomembranes will be similar, essentially independent of thickness, so the microstructures at the roots of the increasingly deep notches will differ. It has been noted that the thicker materials typically have lower SCRs.
- Repeat 3 above with notches on the inside surface of the bubble.
- Repeat 3 above but with extruder parameters the same for all thicknesses. This will generate more controlled baseline specimens than 3 above and will facilitate an understanding of variability in conventional production material for which extruder parameters are adjusted for the different geomembrane thicknesses
- Make plaques from 1 mm and 3 mm thick geomembrane to assess the effects of orientation change and to determine if melting and slow cooling normalize SCR. It is not known how orientation and microstructure will combine to affect the ultimate SCR.

When textured materials are tested the ASTM D5397 single point test is usually performed on specimens from the smooth edge strip. However, it is important not to rely on nominal thickness of randomly textured sheets but to make actual measurements of edge strip thickness since on randomly textured materials the core thickness is usually about 10% higher than the nominal thickness. It is also important to measure strip yield stress in order to determine the correct applied load. In one case (Thomas 2008) an unusual stress cracking result was obtained. Examination of the fracture did not show the conventional knife cut, the radiating quasi-brittle fracture, and the final ductile break, as shown in Figure 6, but rather showed two quasi-brittle fracture regions separated by a ductile region (Figure 9). The cause of this morphology is not known. The strip material was made into a plaque and retested. The conventional break morphology was observed and the SC break time increased.

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Figure 9. Double break morphology, Notch (N), Brittle Break (B), Ductile Break (D)

In another recent case the primary author was seeking an SCR of >1500 h on smooth 2.0 mm thick HDPE. Samples from two rolls achieved this specification, but on a third roll (between the other two) four of the five specimens had consistent break times of 689, 678, 668, and 709 h, but the fifth specimen broke at 1297 h. The first four all had break morphologies as shown in Figure 6, but the last had the double brittle fracture morphology as shown in Figure 10. Since this specimen had the longer break time it is appears that the crack at the root of the notch propagated as usual, but then it transitioned to a ductile break for some reason before returning to a brittle break and final ductile break. The cause of this behavior is yet to be determined.



Figure 10. Double break morphology

Another factor that can impact the results of a long term test is the requirement in ASTM D5397 to refresh the Igepal solution every two weeks (294 h). This means that a conventional GRI GM 13 SCR test requiring an SCR >300 h can be performed without changing baths. However, a 1500 h test period may require four bath changes – times during which temperature and load changes may be introduced. These are also factors that can affect the final break times.

In practical terms the days of significant fundamental stress cracking failures in exposed HDPE geomembranes are behind us. However, we must now be wary, as service lifetimes approach 30 years, of another round of stress cracking failures as HDPE geomembranes lose their antioxidants and start to oxidize. Koerner et al. (2008) has recently presented the estimated service lives of exposed geomembranes (in Texas, WA) as shown in Table 2. HDPE exceeds only 28 years. While service life will unquestionably be a function of resin used, AO additive formulation, service

stresses, liner temperatures, quality of welding, etc., when AO packages are depleted, failure will occur by stress cracking. So that if lives are as short as 28 years, we may soon expect to see more SC failures.

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Туре	Specification	Predicted lifetime in Texas, USA
HDPE-1	GRI GM 13	>28 yrs (incubation ongoing)
LLDPE-1	GRI GM 17	>28 yrs (incubation ongoing)
EPDM-1	GRI GM 21	>20 yrs (incubation ongoing)
PE-R-1	GRI GM 22	$\sim$ 17 yrs (reached half-life)
fPP-2	GRI GM 18 (temp. susp.)	>27 yrs (incubation ongoing)
fPP-3	GRI GM 18 (temp. susp.)	>17 yrs (incubation ongoing)

 Table 2. Projected exposed geomembrane lifetimes (Koerner et al. 2008)

## CONCLUSIONS

There are many factors that can influence the SCR of an HDPE geomembrane. Many are not well established or simply have not been investigated. A testing program is proposed that will generate some of the unknown data. Testing will be performed and results will be presented in a follow-up paper.

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