New technique for predictive geomembrane stress crack performance: Commercial application

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ABSTRACT:Stress crack resistance of HDPE geomembranes has always been a major consideration for geomembrane raw material suppliers, geomembrane manufacturers and end users. In the past, Environmental Stress Crack Resistance (ESCR), often described a “bent strip test”, characterized by ASTM D1693 and other test protocols was common. As resin quality and geomembrane performance levels improved over time and with technical advances this test became less useful as longer and longer test durations (2000 hours or more) were needed to test to the failure of the materials. In response to this issue the geosynthetic industry developed a new testing protocol, Notched Constant Tensile Load (NCTL). NCTL, characterized by ASTM D5397 and other testing protocols which were created tested resins of that era within test durations of a few hundred hours. NCTL is now globally accepted as both a measure of HDPE geomembrane quality and predictive of lifespan. However, in the recent decade, resin and geomembrane performance has continued to again improve and NCTL testing durations of 1000 h or more currently present the recurring issue of a test duration that is too long for technical, quality, and business considerations. New testing protocols examining the stress strain behavior of geomembrane have been found in recent evaluations to offer values that are predictive of stress crack performance. While these new testing protocols have been utilized with some success in the pipe industry, these methods are applied here to geomembranes of a fully commercial scale with results that represent an advancement in the technology of evaluating geomembranes.

Keywords:geomembrane, stress crack, NCTL, ESCR, strain hardening

1 INTRODUCTION

Polyethylene has been the primary material of choice for geosynthetic barriers for the past several decades. There are a multitude of considerations in the materials selection, but the primary basis for the selection and the success of polyethylene geomembrane is chemical resistance. Polyethylene is unaffected by a broad range of chemicals, pH ranges and environments. This has fit very well with the fact that the chemical content of a landfill, dump, waste pile or other waste disposal collections is nearly always somewhat unknown and variable. People throw a great variety of things in the trash.

Polyethylene has this broad based chemical resistance as a result of its chemical structure. Polyethylene is a semi-crystalline material with zones of both rigid crystalline components and amorphous, more flexible zones. This mixture of structures occurs within the total polymer matrix and the quantities of the respective components vary as a result of many factors including, but not limited to polymerization technique and environment, catalyst type and structure, co-monomer(s) and polymerization and processing conditions. A consequence of this crystallinity manifests itself in the most common failure mode of the material, stress crack failure (Carey, 1950 and McTigue, 1959). This failure is the sudden and occasionally catastrophic cracking of the material resulting in cracks that may extend for meters or more and branch out to create other multiple cracks (Popelar et al. 1998). In the most damaging manifestation, cracks can measure meters or more in lengthand in shape, resemble the silhouette of a tree, trunk to multiple branches etc. Thus, the measurement of the tendency of a material to resist stress cracking has been, over many decades, a way to compare materials and select grades better suited for geomembrane barrier applications. The first of these methods was Environmental Stress Crack resistance (ESCR) as embodied by ISO 22088-1 and ASTM 1693-13. The more recent methods NCTLareembodied by ASTM D5397 and EN 14576. The aforementioned tests are specific for geomembrane; it is noteworthy that there is a host of other methodologies designed for pipe and other applications (ISO 16770). This paper highlights the use of a new test and evaluation method for evaluating stress crack behavior of HDPE geomembranes on a commercial scale – tensile strain hardening.

2 HISTORICAL TIMELINE

Regardless of the test, ESCR in the 1980s, NCTL in the late 1990s and forward, the primary limitation of stress crack resistance evaluation has always been time. Standards were developed that exposed the materials to a specific combination of temperature, physical stresses and a more aggressive chemical environment. These conditions varied but were always selected with a reasonable goal of comparing the materials in existence at the time of the tests creation. Materials were then evaluated under these sets of conditions until failure – the stress crack failure of the materials. Table one presents the duration requirements common to different time periods

Table 1. Specification requirements timeline.

Test type	Specificationrequirement	Date / reference
ESCR* (ASTM D1693)	500 h	1983 NSF 54 requirement
	1000 h	Mid 1980s Manufacture data sheet values
	1500 h	1993 NSF 54 requirement
	2000 h	Early 1990s Manufacture data sheet values
	10000 h	1995 Manufacture data sheet values
NCTL (ASTM D5397)	200 h	1997 original publication GRI-GM-13
	300 h	2003 change to GRI-GM-13
	500 h	2014 change to GRI-GM-13
	1000 h	2017 Manufacture data sheet values

* note testing conditions (temperature and chemical exposure) varied

The difficulties in material evaluation and materials comparison have arisen over time as the polyethylene resins and polyethylene geomembranes improved in quality and stress crack resistance capability.

The duration of the test is the factor that has spurred the changes in testing protocol. As the materials have improved, the testing durations, under a given set of conditions, have increased. This increase continues until the duration of the test becomes too long and more of a hindrance to commercial manufacturing operations rather than a tool to identify material concerns prior to geomembrane manufacture.

3 COMMERCIAL AND FINANCIAL IMPLICATIONS

The duration and response time of a stress crack resistance test has significant commercial and financial impact.

Ideally, one would test the material to completion prior to utilizing a given lot of the material in question to make geomembrane. One function of these tests is to screen out “bad” materials, or more practically, identify when an error has occurred and the materials will not comply with quality requirements and expectations. However this decision, to test the material to completion prior to using it, has significant commercial and financial ramifications.

Assume that a 1000-hour testing time is the standard. This was a past practice for ESCR testing and is currently the standard for some high-performance grades of geomembrane when tested using NCTL. For a single round die production line running at 900 kg/hour a commitment to test stress crack resistance to completion, prior to any material use is, in fact, a commitment to invest in a raw material inventory in excess of USD$2,500,000 at the prices in effect at the time of this writing. This figure does not include the capital cost of silage to store the materials prior to use, the cost of laboratory equipment to test significant quantities of materials and further, assumes that the testing will always be timely, complete and achieve a positive result. If one assumes the occasional material performance problem – (logical, or why test in the first place) and the need to have at least a week to 10 days of approved and available raw materials in the case of such problem, a more realistic estimate is in excess of USD$3,000,000 per round die line. A “typical” flat cast line would run at roughly double this production rate and thus, double the raw material inventory cost. Again, not including the infrastructure required to operate in this fashion.

4 NCTL VARIABILITY ISSUES

The above commercial considerations would be a concern even if the NCTL test was readily reproducible, repeatable and did not vary significantly from laboratory to laboratory. Unfortunately, that is not the case and NCTL variability further hinders a “clean and efficient” supply chain. Within the Geosynthetic Accreditation Institute – Laboratory Accreditation Program (GAI-LAP) the uncertainty of the NCTL test is listed as 21% (Koerner, 2016). This figure is based on and measures the variability of multiple laboratories testing a single common specimen source. Thus, the comparison of different materials, or different lots of the same material would be expected to increase the uncertainty, and in fact, significantly does so.

While the purpose of this paper is not to address the specifics of the NCTL test, it is appropriate here to list a number of factors that have, over time, contributed to variability of results in the testing:

- Sample homogeneity: a relatively small portion of the material is being testing.
- Sample preparation: testing of textured material usually requires additional sample preparation steps.
- Notching: cutting blades must be conditioned properly prior to use and have limited lifespan.
- Notch placement: for multilayer materials, this may be a consideration.
- Notch depth: a critical factor with wide extra-laboratory variability in measurement technique.
- Chemical exposure: variability in concentration, age and effects of the surfactant used.
- Environment (test) consistency: variability in solution, mandated changes in solution during tests.

Clearly the NCTL test, while the effective measure of performance used by the global industry, carries with it some variability, potential for error and associated high costs (both in the operation of the test and the financial commitments estimated above)(Bobsein, 1998). As the quality of polyethylene has improved, and test durations lengthen, these issues become more critical.

5 STRAIN HARDENING

Within the parameters described above, it is advantageous to develop a method for a more rapid evaluation of stress crack behavior. It is not the intent of this paper to discuss the strain hardening modulus testing protocol in detail. The protocol is reviewed in the documents referenced herein and currently under development in the European Committee for Standardization (CEN)Technical Committee (TC)189 (prEN 17096:2017) and other entities are researching the function and utility of the testing (Zanzinger et al. 2015). The core of this paper is the reporting of an evaluation of commercial materials via strain hardening, however, a brief overview may be in order for some readers as well as the illustration in Fig. 1.

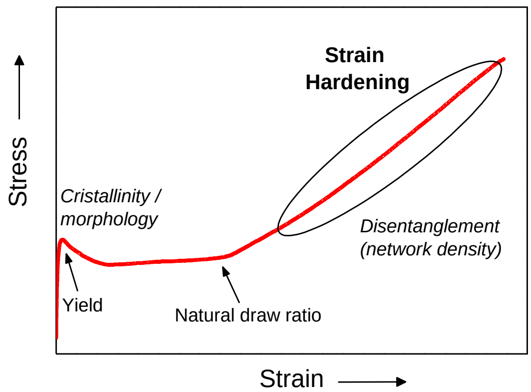

Figure 1. Strainhardening behavior – taken from Havermans-van Beek et al. (2010).

In short, strain hardening is the outcome of multiple tensile tests conducted on the material at a single, or a range of temperatures. The technical definition currently under development is: “Strain hardening modulus, slope of the Neo-Hookean constitutive model between a true strain of 8 and up to the point of maximum stress but not above 12, in megapascal (MPa)". In simpler language, the slope of the stress strain curve in the area of significant deformation, but prior to breakage. And in perhaps an oversimplification, how the materials respond in this range of the stress strain curve.

The proposition that has been supported by work completed in the pipe industry is that materials with higher strain hardening moduli will have better stress crack resistance than materials with lower strain hardening moduli. That is supported, for geomembranes, in the values below.

6 COMMERCIAL SAMPLE EVALUATION

A total of twelve materials were tested for strain hardening moduli. Nine of these materials were samples of commercial round die geomembrane production; three tested samples were produced on a laboratory scale flat cast line. All of the materials utilized the same base resin, a modern hexane co-polymer polyethylene. The materials were all similarly stabilized and in the 11 samples where the materials were colored (either black, or black-white coextrusions). The masterbatches used were the same.

The geomembrane materials were manufactured over a two-year period, utilizing different processing lines. While these materials all used the "same" feedstock materials – resin and masterbatch grades and formulations, the raw materials were produced over a wider time period and in differing campaigns and in some cases, at different production plants. The laboratory materials were processed consecutively beginning with sample "P" a natural resin, sample "Q" was this same resin with the addition of carbon black masterbatch and sample "R" contained an additional polymer blend. One can very reasonably expect greater variance with the production materials and a relative "best case" for the laboratory samples where many of the variables have been eliminated.

Table 2. Materials summary and strain hardening moduli data.

Sample identification	Strain hardening modulus [MPa]	Sample source	Resin lot no.	Thickness [mm] - color	Sample density (as tested complete) [grams/cm³]	Sample density (as tested black only) [grams/cm³]	NCTL values ranges
A	29.07	Full scale production	XXX717	2.0 - B/W	0.954	0.947	Low
B	29.47		XXX567	2.0 - B/W	0.953	0.946	Medium
C	31.51		YYY291	1.5 - Black	0.948	n/a	High
D	31.37		YYY216	1.5 - Black	0.945	n/a	High
E	29.09	Full scale production	XXX716	2.0 - B/W	0.952	0.946	Medium
F	33.61		YYY291	1.5 - Black	0.945	n/a	High
H	28.22		XXX753	2.0 - B/W	0.954	0.945	Medium
K	30.20	Full scale production	XXX716	2.0 - B/W	0.951	0.948	Medium
L	29.69		XXX717	2.0 - B/W	0.950	0.949	Low
P	34.24	Laboratory scale	XXX792	1.2 - Clear	0.933	n/a	High
Q	31.67		XXX792	1.1 - Black	0.944	n/a	High
R	28.86		XXX792	1.2 - Black	0.944	n/a	Medium

Likely the least satisfying column to most readers will be the listing of NCTL performance. For a variety of reasons, these materials were tested for NCTL properties multiple times, by multiple laboratories, over a 9-12 month period with significant disagreement regarding the respective values. For this reason, as well as the reasons stated above regarding variability of NCTL testing, we have chosen not to report specific hour values for NCTL. A "Low" NCTL value is in the range of 300-500 h, a "Medium" NCTL value 500-700 h and a "High" NCTL value from 700-1000+ h (700 to >1000 hours with no failure).

7 DISCUSSION OF RESULTS

In general, the strain hardening moduli accurately predicted and mirrored the NCTL behavior of the materials. In each of the three test groupings (as indicated by separation in Tab. 2) the material with the lowest strain hardening moduli had the lowest NCTL value. In each set of test groupings the respective ranking of NCTL and strain hardening moduli was identical. The laboratory scale evaluation, with the expected lower variability, was the most definitive in the ranking and predictive demonstration of NCTL performance.

Comparing the entire data set, there is a clear relationship between strain hardening moduli and NCTL performance as indicated in Fig. 2.The strength of the data and the predictive nature of strain hardening moduli are clear and apparent.

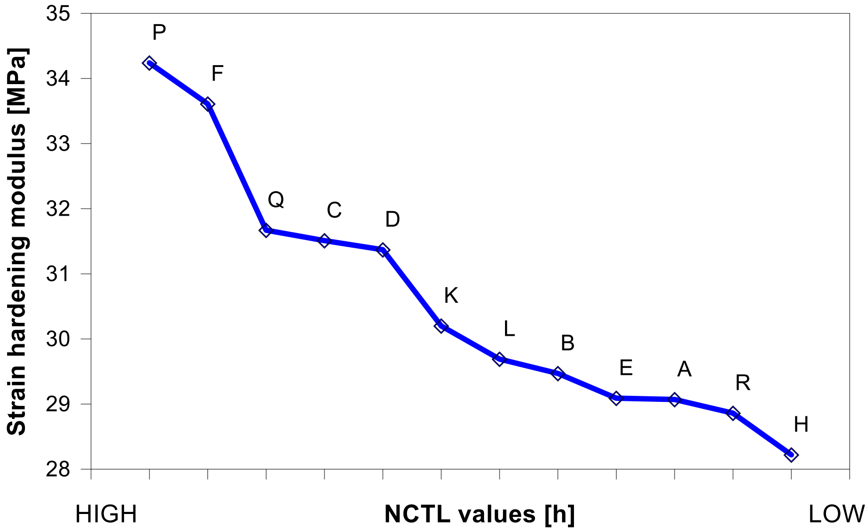

Figure 2. Strainhardening / NCTL comparison.

However, the authors do not feel that a clear quantitative and mathematical relationship has been established between strain hardening moduli and stress crack performance. This is due, in part to the variability of the materials tested, with multiple resin lots, multiple production equipment and multiple technicians and laboratories contributing to the NCTL evaluations. In defense of the endorsement of strain hardening moduli as a predictive tool, this data source is nearly a worst-case scenario. The only expected large impact complication could be the addition of multiple resin grades/sources.

8 CONCLUSION

Data and history is presented on the evaluation of multiple commercial geomembrane products via strain hardening moduli. A relationship clearly exists between strain hardening moduli and NCTL performance for commercial geomembrane materials. As the usage of strain hardening evaluation continues to expand and additional materials are evaluated, it is a very reasonable expectation that a definitive relationship will be established between strain hardening moduli and stress crack resistance and that eventually, strain hardening moduli may become the principle criteria for resin evaluation and quality control.

ACKNOWLEDGEMENTS

The authors would like to thank GSE Environmental for supplying the data and funding this testing. GSE has made significant contributions to the geosynthetics industry over the past several decades – this paper is another.

REFERENCES

ASTM D1693 - 13, Standard Test Method for Environmental Stress-Cracking of Ethylene Plastics.
ASTM D5397 - 07 (Reapproved 2012), Standard Test Method for Evaluation of Stress Crack Resistance of Polyolefin Geomembranes Using Notched Constant Tensile Load Test.

ASTM D6992, Test method for accelerated tensile creep and creep-rupture of geosynthetic materials based on time-temperature superposition using the stepped isothermal method.
Bobsein, R.L. (1998). Factors Influencing SP-NCTL Test Results, Proceedings of the 12th GRI Conference, The Geosynthetic Institute, Folsom, PA, USA.
Carey, R.H. (1950). Stress-Cracking of Polyethylene, ASTM Bulletin, No. 167, July 1950, 56 p.
EN 14576, Geosynthetics - Test method for determining the resistance of polymeric geosynthetic barriers to environmental stress cracking.
Havermans - van Beek, D.J.M., Delieck, R., McCarthy, M., Kloth, R. and Kurelec, L. (2010). An elegant and fast method to predict the slow crack growth behavior of high density polyethylene pipe materials. Proceedings of the Plastics Pipes XV Conference, Vancouver, Canada.
ISO 16770, Plastics - Determination of environmental stress cracking (ESC) of polyethylene – Full notch creep test (FNCT).
ISO 22088-1:2006(en) Plastics — Determination of resistance to environmental stress cracking (ESC) — Part 1: General guidance.
Koerner, G. (2016). Geosynthetic Institute’s Laboratory Accreditation Program: a Twenty year Review. Proceedings of GeoAmericas 2016, Miami, Florida, USA.
Lustiger,A., Corneliussen, R.D. and Kantz, R. (1978). Environmental stress crack growth in low density polyethylene. Materials Science and Engineering, pp 117-123.
McTigue, F.H. (1959). Environmental Stress-Cracking Resistance of High Density Polyethylene. Plastic Technology, Vol. 5, February 1959, pp 35-39.
Personal correspondence of the principal author with Gary Kolbasuk, October 2017.
Popelar, C.H., Kuhlman, C.J. and Peggs, I.D. (1998). A fracture mechanics assessment for the long term durability of HDPE geomembranes. Sixth International Conference on Geosynthetics, pp 365-369.
prEN 17096:2017, Geosynthetics - Test method for the determination of the strain hardening modulus of HDPE geosynthetic barriers.
Zanzinger, H., Gerets, B., Wenzel, M., Hausmann, S. and Engelsing, K. (2015): Determination of the Stress Cracking Resistance of HDPE Geomembranes by Using Accelerated Test Methods. Geosynthetics 2015, Portland, Oregon, USA, 15-18 February 2015, pp 938-944.