Strain hardening behavior of HDPE geomembranes

Helmut Zanzinger[1], *Kurt* Engelsing[1], *Britta* Gerets[1]

[1]SKZ – German Plastics Center, Friedrich-Bergius-Ring 22, 97076 Würzburg, Germany

Abstract. The strain hardening behavior is a good measure for the stress cracking resistance of HDPE geomembranes. Influences of the testing parameters temperature and cross head speed on the strain hardening modulus in material testing of PE were quantified. For tests of HDPE geomembranes at 80 °C and 10 mm/min a good correlation with SP-NCTL tests was demonstrated.

1 Introduction

In polyethylene (PE) three main failure mechanisms can be observed: First ductile failure due to locally exceeding the yield stress, second failure basing on slow crack growth and third brittle failure evoked by thermo-oxidative degradation. While in PE products ductile failure and degradation during service can be prevented by a proper design and an adequate stabilization, slow crack growth is correlated to the molecular structure [1]. For long-term applications, e.g., geomembranes, nowadays materials on the market promise service lives up to 100 years [2]. For this reason, one challenge is to quantitatively characterize the slow crack growth behavior of HDPE geomembranes. The strain hardening behavior in uniaxial tensile tests has been shown to correlate with the materials resistance against slow crack growth respectively fibril formation and failure during crazing [3]-[5]. For HDPE geomembranes the test method is described in EN 17096:2018. A deep understanding of the strain hardening behavior in such tests is crucial. Therefore, the influence of temperature and deformation speed was thoroughly investigated for different PE types, providing a basis for a discussion of the advantages and disadvantages of the various test parameters.

2 Materials and Methods

The investigations regarding the test parameter influences were performed on commercial PE grades of different stress cracking resistance (material A < material B < material C < material D). Sample preparation was done via compression molding of 1 mm sheets on a P/ 300, Dr. Collin GmbH, Ebersberg, Germany, and subsequent annealing in a heating chamber FED 240, Binder GmbH, Tutlingen, Germany, as described in ISO 18488:2015. Subsequently, dog bone specimens were punched with a modified EN ISO 527-2:2012 type 5B geometry. This very small geometry was developed to allow for strain hardening tests on specimens directly prepared from e.g. geomembranes [6] and became part of

EN 17096:2018. Using a cross head speed of 15 mm/min at a testing temperature of 80 °C leads to the same results as the testing according to ISO 18488:2015 [6]. All uniaxial tensile (strain hardening) tests were done on a universal testing machine Z010, Zwick/Roell GmbH & Co. KG, Ulm, Germany, equipped with an OptiXtens optical extensometer and a temperature chamber. Thermography measurements were conducted with a VarioCam® high solution, InfraTec GmbH, Dresden, Germany, camera and the related software IRBIS® remote 3.0 was used to analyze the thermographic images.

Evaluation of the measured stress-strain behavior was done analogous to the approach described in ISO 18488:2015 using a Neo-Hooke model and deriving the strain hardening modulus $\langle G_p \rangle$ via differentiation as it is defined as the slope in the strain hardening region. If test conditions differ from 80 °C and / or 15 mm/min, using constant limits for the evaluated draw ratio λ range (ISO 18488:2015: $\lambda = 8\ldots12$) fail [6]. In Fig. **1** the evaluation procedure used is illustrated: Starting with the technical stress-strain-curve (Fig. **1**, left side) the lower limit at the natural draw ratio λ_{NDR} is determined as the intersection point of the linear fits for the cold drawing and strain hardening regions. Considerations to use a factor (of e.g., 1.3) in combination with λ_{NDR} as the lower limit are part of current research. The draw ratio at maximum true stress $\lambda_{\sigma,max}$ is used as the upper limit in evaluating the true stress-draw ratio-curve (Fig. **1**, right side).

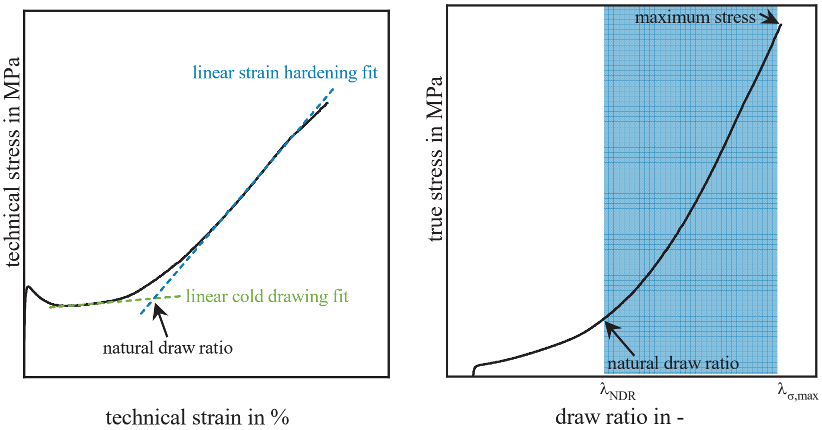

Fig. 1. Technical stress-strain-diagram and true stress-draw ratio-diagram of material C tested at a temperature of 80 °C and cross head speed of 15 mm/min. [13]

All strain hardening moduli on HDPE geomembranes were tested according to EN 17096:2018, which specifies a test speed of 10 mm/min.

For the ageing of a 2 mm thick smooth HDPE geomembrane oxidation tests acc. to ISO 13438:2004, in high-pressure autoclaves were used, which allow synchronous ageing due to oxidation as well as leaching in an aqueous medium [12]. The test specimens were exposed to pure oxygen (O_2) in a 0.01 mol/l NaHCO3 solution at pH10 at a temperature of 80 °C under three different oxygen pressures of 51 bar, 26 bar and 6 bar respectively for periods of approx. 60 days, 55 days, 130 days.

3 Results and Discussion

Fig. **2** shows the strain hardening moduli of the four materials tested at a testing temperature range of 23 °C to 80 °C with a constant cross head speed of 15 mm/min. For all materials linear correlations between strain hardening modulus and testing temperature were found. The slopes are not the same, so enlargements of the testing temperature evoke a steeper decrease in the resulting strain hardening modulus for materials with lower stress cracking resistance than for those with a higher. This means the discrimination of materials with different stress cracking resistances becomes better at higher temperatures. Nonetheless, all slopes are quite similar as all materials under investigation were PE grades.

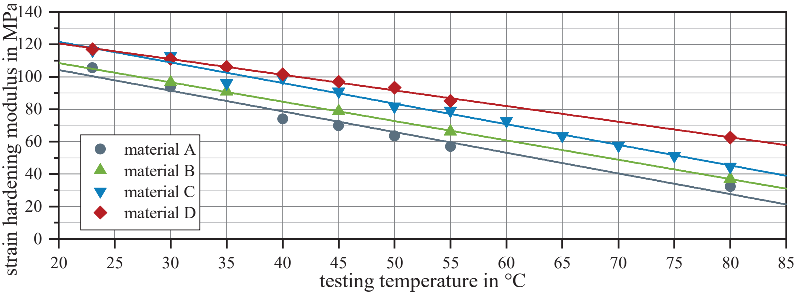

Fig. 2. Influence of the testing temperature on the strain hardening modulus for different PE materials tested at a cross-head speed of 15 mm/min. [13]

To test the influence of the cross-head speed a range of 1 mm/min to 100 mm/min was tested at 23 °C. As can be seen from Fig. **3** all materials show linear correlations between strain hardening modulus and the logarithm of the cross-head speed. Again, the slopes are very similar, but enlargements of the cross-head speed lead to a larger increase in the resulting strain hardening modulus for materials with lower stress cracking resistance than for those with a higher. This means the discrimination of materials with different stress cracking resistances becomes worse at higher cross head speeds.

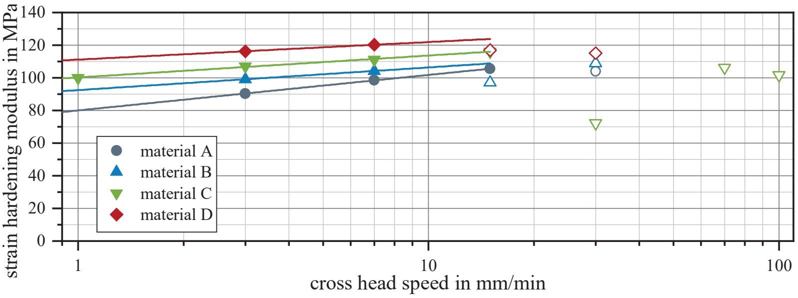

Fig. 3. Influence of the cross-head speed on the strain hardening modulus for different PE materials tested at a temperature of 23 °C. [13]

Observing linear correlations between the strain hardening modulus and testing temperature as well as logarithm of cross head speed, both influences can be directly transformed in each other: An increase in testing temperature of ΔT = 10 °C corresponds to a decrease in cross head speed $\Delta v_{\mathrm{cross\ head}}$ of about one decade. [13]

Furthermore, Fig. **3** prevails that the linear correlations only hold up to cross head speeds of about 7 mm/min. Taking a closer look to the technical stress-strain curves of such measurements (shown for material C in Fig. **4**, left side), with higher testing speeds the transition from the cold drawing to the strain hardening region becomes less smooth. As the thermographic images (Fig. **4**, right side) clearly reveal, the phenomenon comes from an intrinsic heating of the specimen (more than 10 °C at $v_{\mathrm{cross\ head}}$ = 70 mm/min).

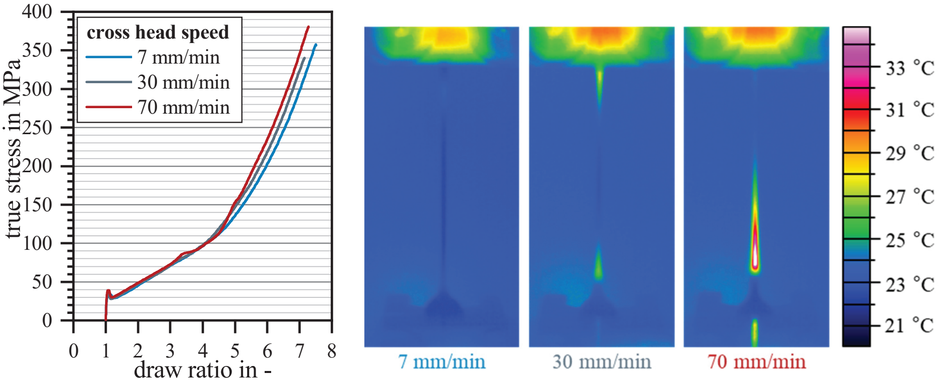

Fig. 4. Influence of the cross-head speed on the stress-strain behavior and intrinsic heating of material C under uniaxial tensile load tested at a temperature of 23 °C. [13]

To check the potential effect of material coloring, material B was tested in an uncolored and black version at cross-head speeds of 7 mm/min and 70 mm/min at a temperature of 23 °C (Fig. **5**). For the slower cross-head speed of 7 mm/min no effect was detected, while both specimens showed severe intrinsic heating for testing with 70 mm/min cross-head speed. The deviations between the intrinsic heating detected for the uncolored and black material are negligible compared to the influence of the cross-head speed. [13]

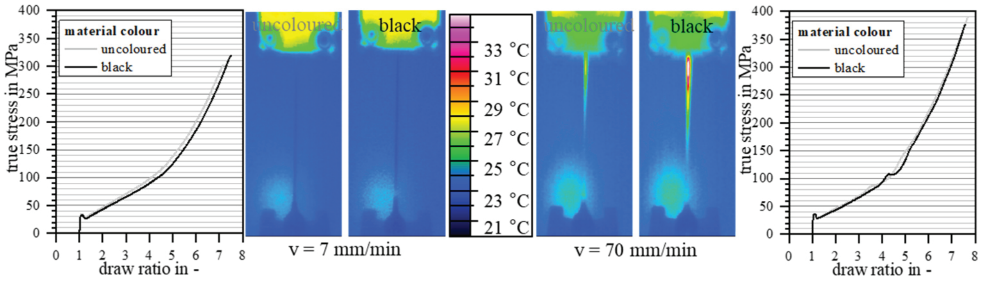

Fig. 5. Influence of coloring on the stress-strain behavior and intrinsic heating of material B under uniaxial tensile load tested at a temperature of 23 °C and cross-head speeds of 7 mm/min respectively 70 mm/min. [13]

On that basis, assuming the physical effects observed in material testing should be also preserved in component testing, specimen directly taken from HDPE geomembranes were tested at 80 °C and a test speed of 10 mm/min. Comparing the results to SP-NCTL test results, required in many specifications, good correlations were found between the strain hardening modulus according to EN 17096:2018 and the SP-NCTL test according to EN 14576:2005 [7]-[12]. When comparing the strain hardening modules with the SP-NCTL failure times, it must be considered that the SHT modules were all carried out in the same laboratory. The SP-NCTL results from [7], on the other hand, come from 3 different laboratories and the SP-NCTL results from 12 further commercially available materials [11] were often tested in several laboratories over a 9-12 months period with significant disagreement regarding the respective values. For this reason, as well as the reasons regarding variability of NCTL testing [11], it was chosen not to report specific hour values for NCTL in Fig. **7**. Based on the results of Figure 6 the failure time of 500 h in SP-NCTL tests would correspond with a strain hardening modulus of around 30 MPa.

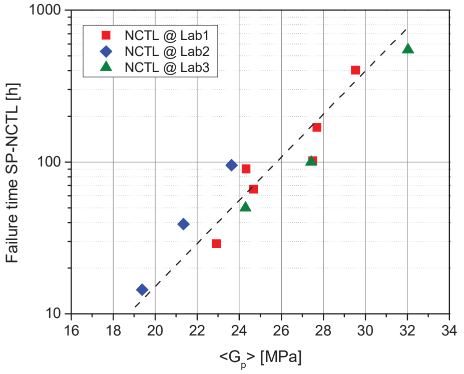

Fig. 6. Strain hardening modulus <Gp> versus SP-NCTL failure times for different HDPE geomembranes [7]

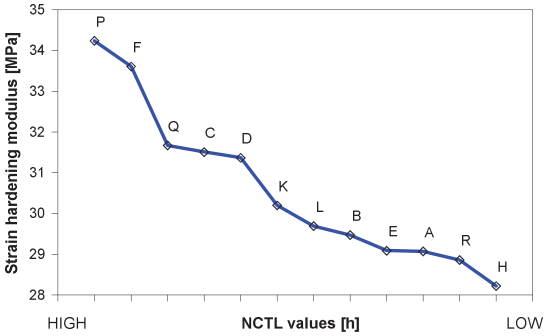

Fig. 7. Strain hardening / NCTL comparison of 12 commercially available materials [11]

In the case of the investigation of a 2 mm thick smooth HDPE geomembrane aged in high-pressure autoclaves following method C of EN ISO 13438:2004, both the failure times in the SP-NCTL test and the strain hardening moduli were investigated on the same material in the same laboratory. The correlations found between strain hardening modulus and failure time in the SP-NCTL test are very good.

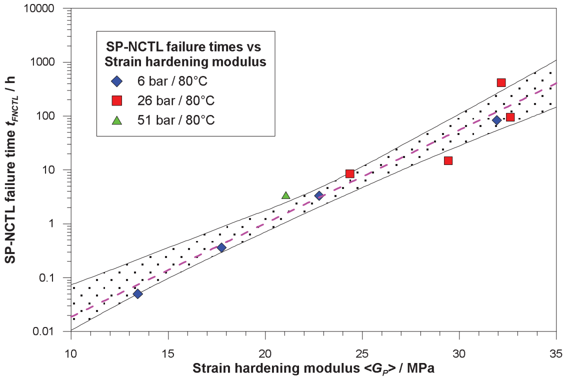

Fig. 8. SP-NCTL failure times acc. to EN 14576 vs. strain hardening modulus acc. to EN 17096 on aged HDPE geomembrane specimens [12]

4 Conclusions

For molecularly different PE grades the influences of testing temperature and cross-head speed on the strain hardening behavior were thoroughly investigated. In both cases linear correlations were found under the precondition of an adequate determination of the lower and upper limit in strain hardening modulus calculation referring to the natural draw ratio. Through thermography measurements deviations in the linear correlation of strain hardening modulus and logarithmic cross-head speed at a temperature of 23 °C were proven to result from an unwanted intrinsic material heating, whereas the influence of material coloring is of minor importance. In summary, the results show that strain hardening tests could be performed at different test conditions. But it should be considered, when choosing test parameters, that the discrimination of materials with different stress cracking resistances becomes better at higher temperatures and worse at higher cross-head speeds. Additionally, an effect in test practice is a less smooth transition from the cold drawing to the strain hardening region with higher testing speeds.

For HDPE geomembranes tested in strain hardening tests at 80 °C with test speeds of 10 mm/min acc. to EN 17096:2018 good correlations with SP-NCTL were shown. This gives evidence of the presented strain hardening test setup (acc. to EN 17096:2018) having the potential to provide the same material ranking regarding stress crack resistance as SP-NCTL tests acc. to EN 14576:2005 resp. ASTM D5397-20 Appendix X1.

Acknowledgement

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References

1. G. Strobl, *The Physics of Polymers*, Springer-Verlag: Berlin, Heidelberg (2007)
2. W. Müller, *HDPE Geomembranes in Geotechnics*, Springer-Verlag, Heidelberg (2007)
3. H.H. Kausch, R. Gensler, Ch. Grein, C.J.G. Plummer, P. Scaramuzzino, *Journal of Macromolecular Science Part B, **38***, 803-815 (1999)
4. L. Kurelec, M. Teeuwen, H. Schoffeleers, R. Deblieck, *Polymer, **46***, 6369-6379 (2005)
5. R.A.C. Deblieck, D.J.M. van Beek, K. Remerie, I.M. Ward, *Polymer, **52***, 2979-2990 (2011)
6. B. Gerets, *Schnelltest-Methode zur Charakterisierung der Spannungsrissbeständigkeit von Polyethylen*, Schlussbericht IGF-Nr. 18324 N, SKZ – German Plastics Center: Würzburg, (in German) (2016)
7. K. Engelsing, H. Zanzinger, *A new accelerated test method for stress crack resistance of HDPE geomembranes,* in Segundo Congreso Panamericano de Geosintéticos, GEOAMERICAS 2012, Lima, Peru, 8p (2012)
8. H. Zanzinger, *Evaluation of the stress cracking resistance of exposed geomembranes*. 6th International Conference Geosynthetics Middle East, Abu Dhabi, UAE, SKZ-ConSem GmbH, pp 165-174 (2013)
9. H. Zanzinger, I.D. Peggs, *Stress cracking resistance of HDPE geomembranes with very high densities,* in GeoAfrica 2013 Conference, Accra, Ghana (2013)

10. H. Zanzinger, B. Gerets, M. Wenzel, S. Hausmann, K. Engelsing, *Determination of the Stress Cracking Resistance of HDPE Geomembranes by Using Accelerated Test Methods*, in Geosynthetics 2015, Portland, Oregon, USA, pp 938-944 (2015)
11. R. Ramsey, E. Zimmel, H. Zanzinger, *New technique for predictive geomembrane stress crack performance: commercial application*, in 11th International Conference on Geosynthetics, Seoul, Korea (2018)
12. H. Zanzinger, *Evaluation of the Durability of PE-HD Geosynthetic Barriers Using High-Pressure Autoclave Tests,* Int. J. of Geosynth. and Ground Eng. **6**, 40 (2020)
13. B. Gerets, K. Engelsing, *Strain hardening behaviour of polyethylene: Influence of temperature and time*, in Book of Abstracts, DYFP 2018 conference, Kerkrade, Netherlands (2018)