Accelerated Slow Crack Growth Resistance Tests For Characterization of High-Performance Bimodal HDPE Pipe Materials

Univation Technologies /
The Dow Chemical Company

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Topics

- Introduction
- Accelerated tests for SCGR
- Bimodal Polymer Structure
- Results of accelerated tests
- Conclusions

Univation Technologies –
Global Licensor Leader of UNIPOL™ PE Technology

171

Licensed reactor lines in **28** countries

51

Millions tonnes annually global licensed capacity

28

Operating lines ≥400 kTA capacities – proven large capacity leader

47

Reactor lines in design or under construction

Univation Technologies –

World's Leading PE Technology

BROADEST PRODUCT CAPABILITY

- Unparalleled single-reactor product flexibility
- Diversified PE portfolio
- Full density HD / LLD product coverage
- Differentiated products
- Large-volume applications

WORLD'S LEADING PE TECHNOLOGY

- Streamlined design
- Largest proven single-line capacity: 650 kTA
- Lowest CAPEX: 95 – 120 million USD *lower* investment than multi-reactor / multi-loop processes
- Maximizes return on your overall investment
- Enables capturing new opportunities

SERVICES FOR LONG-TERM SUCCESS

- Comprehensive project support
  - Engineering
  - Construction
  - Commissioning
  - Start-up
- Exceptional technology transfer
- PREMIER™ Products and Services
  - Takes performance to the next level

COMMITTED TO INNOVATION

- UNIPOL™ PE Technology transformed the industry – continues today
- Recognized PE industry leader
- Track-record of innovation
- Full-range R&D programs
  - Products
  - Process
  - Catalysts
  - Process control

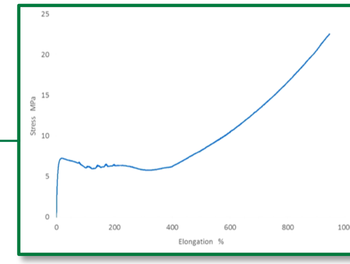
Source for capex data: NexantThinking™ Polyolefins Technology Analysis Report 2018

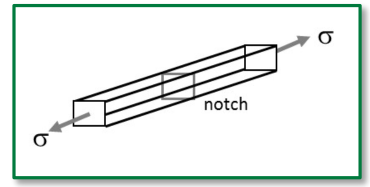
High-Performance HDPE Pipe Materials

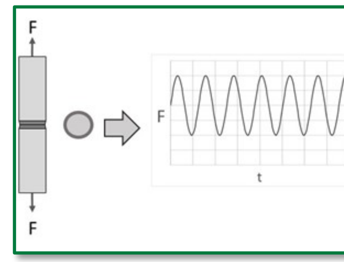
- PE100 → PE100 RC: NPT (ISO 13479) > 1 year
- PE4710: PENT (ASTM F1473) >> 10,000 hours
- Material suppliers have a need for faster evaluation of SCGR of pipe materials for new product development
- Batch release to ensure production quality

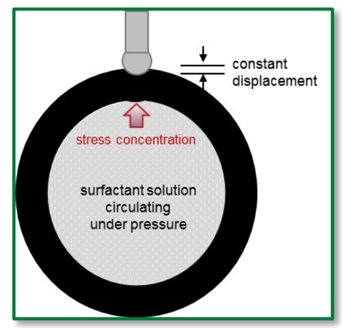
Accelerated Tests

- New methods are allowing characterization of SCGR in times ranging from 1 day to weeks
- Strain hardening modulus (SHM), ISO 18488: data from tensile testing
- Accelerated full notch creep test (aFNCT), ISO 16770:
- Cracked round bar (CRB), ISO 18489: Fatigue crack growth (FCG) predicts creep crack growth(CCG).
- Accelerated point load (PLT+): Pipe with point loading accelerated with surfactant solution.





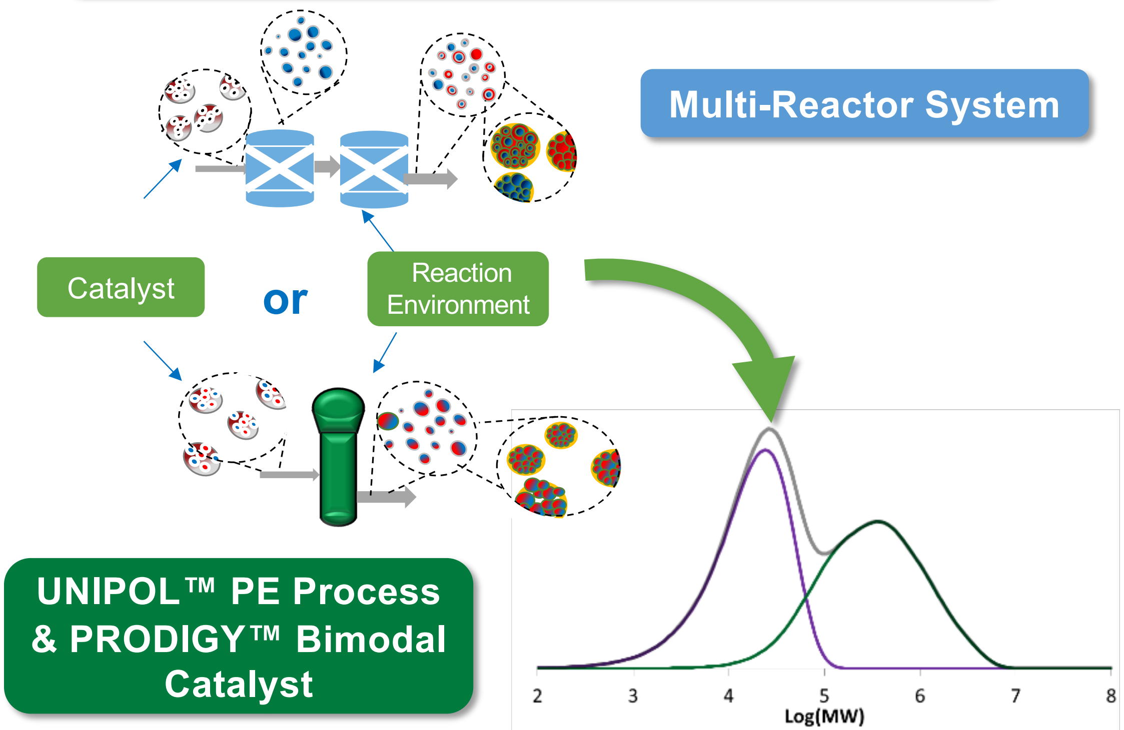




Bimodal HDPE

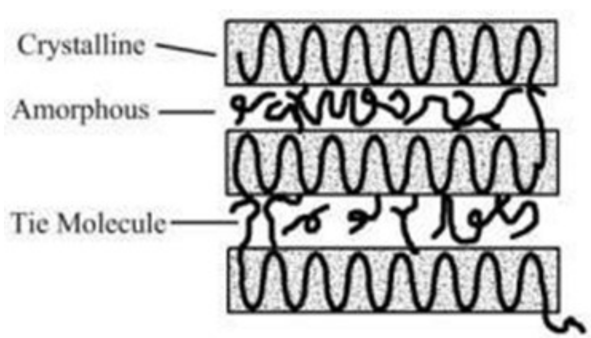
- How do we make bimodal HDPE?
- Reactor configurations – 1, 2, 3
- Catalyst type
- Reaction environment – T, gas composition
- Post-reactor treatment – additives, compounding, etc.

Many factors determine the resulting bimodal polymer

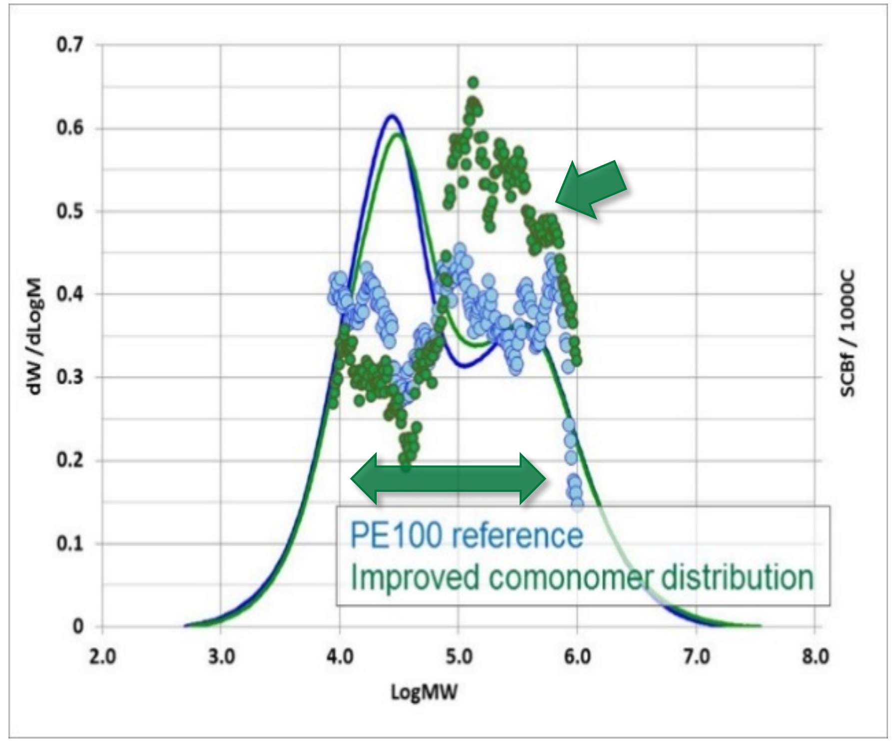


Polymer Structure Effects on SCGR

- Tie molecule structure for SCGR performance
- Polymer variables to affect tie molecules
- Length of chains – MW, MWD
- Comonomer – Type, amount and placement



Semi-crystalline structure



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Effects of Improved Comonomer Distribution

- The materials were characterized by SHM, aFNCT, and PLT+ (Hessel)
- The samples with improved comonomer distribution exhibits higher performance in each of the tests – SHM, aFNCT, CRB, and PLT
- Accelerated version of PLT was used for the pipe test
- SHM is useful for batch release.

Description	SDH Modulus	aFNCT @ 5 MPa hrs	CRB cycles at Ds=12.5 MPa	PLT+ hrs (accelerated version)
PE100 Control	58	249	1.50 x 10^6	429
Improved Comonomer distribution	61	473	1.57 x 10^6	841

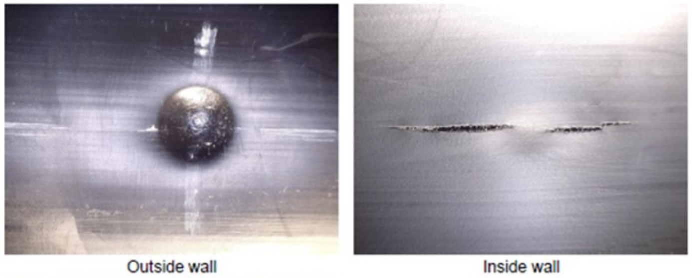
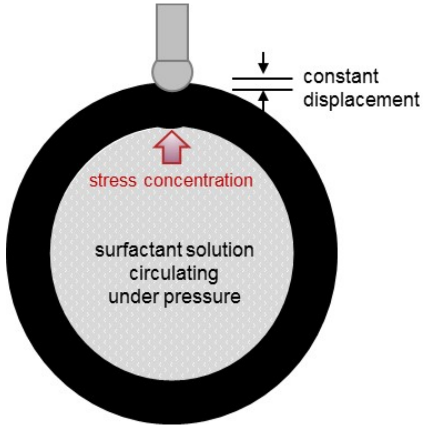


Figure 1: Point loaded location of sample B5



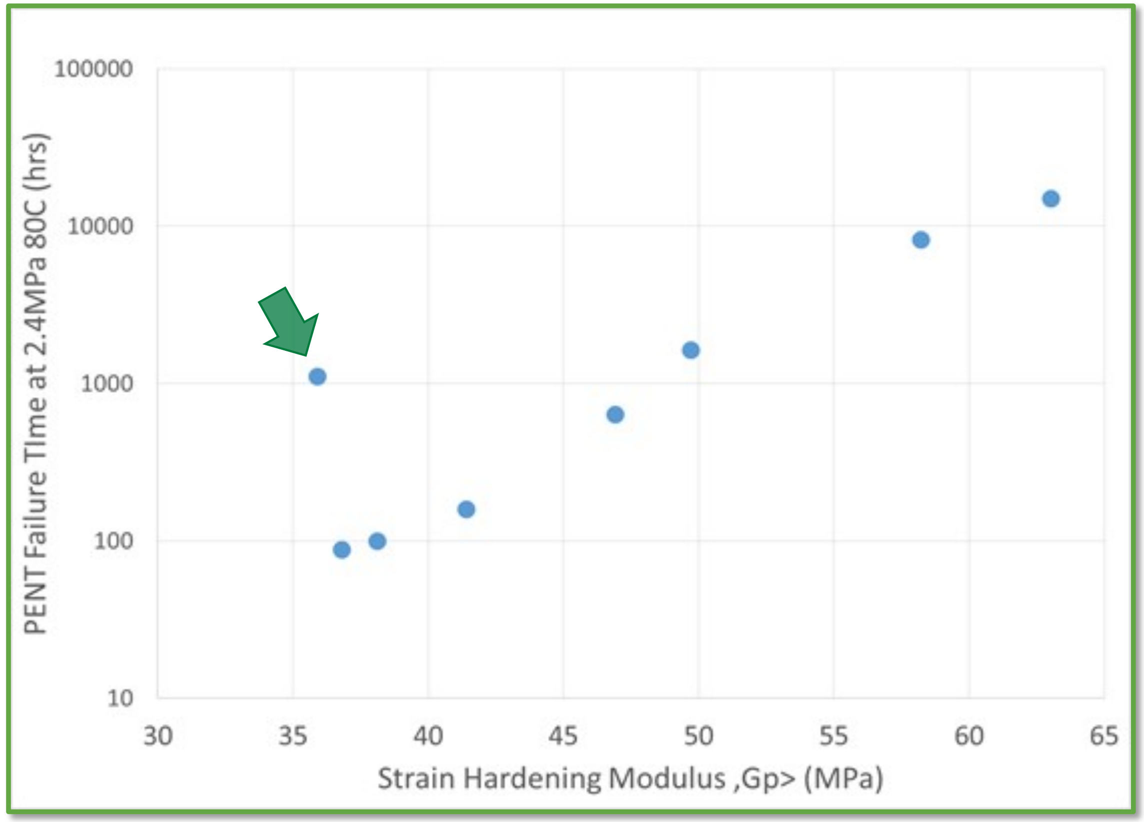
Design of Experiment

- A DOE was conducted to study effects of bimodal PE design variables – HMW density and HLMI, split (% HMW)
- The density and HLMI of the HMW component are strong drivers of SCGR
- Relatively narrow range of density
- The SCGR performance was measured by PENT and SHM

Sample #	Overall Product Density	HMW density	HMW I21	Split ratio (%)	Strain Hardening modulus (Mpa)	PENT Failure time at 2.4MPa 80C (hrs)
Sample 1	0.949	- -	-	-	63	15000
Sample 2	0.952	++	+	+	37	88
Sample 3	0.952	+	+	+	41	160
Sample 4	0.952	0	0	+	47	640
Sample 5	0.952	0	0	-	36	1120
Sample 6	0.951	0	0	-	50	1630
Sample 7	0.951	- -	-	-	58	8200
Sample 8	0.949	- -	-	0		17000
Sample 9	0.954	++	-	0	38	100

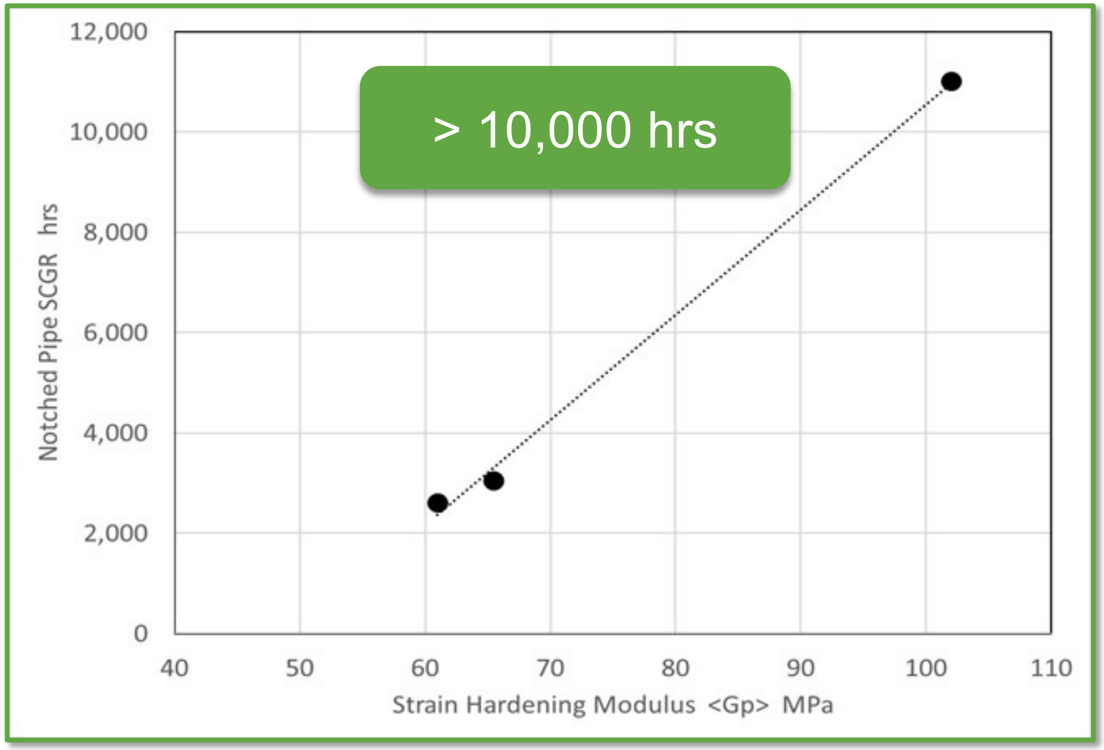
PENT vs. SHM

- The PENT-SHM correlation generally looks good.
- One possible outlier. This will be investigated further.



NPT vs. SHM

- The NPT performance is required for ISO PE100 water and gas pipe
- Higher-performing PE100 RC materials exhibit failure times of > 1 year.
- SHM exhibits a good correlation with NPT



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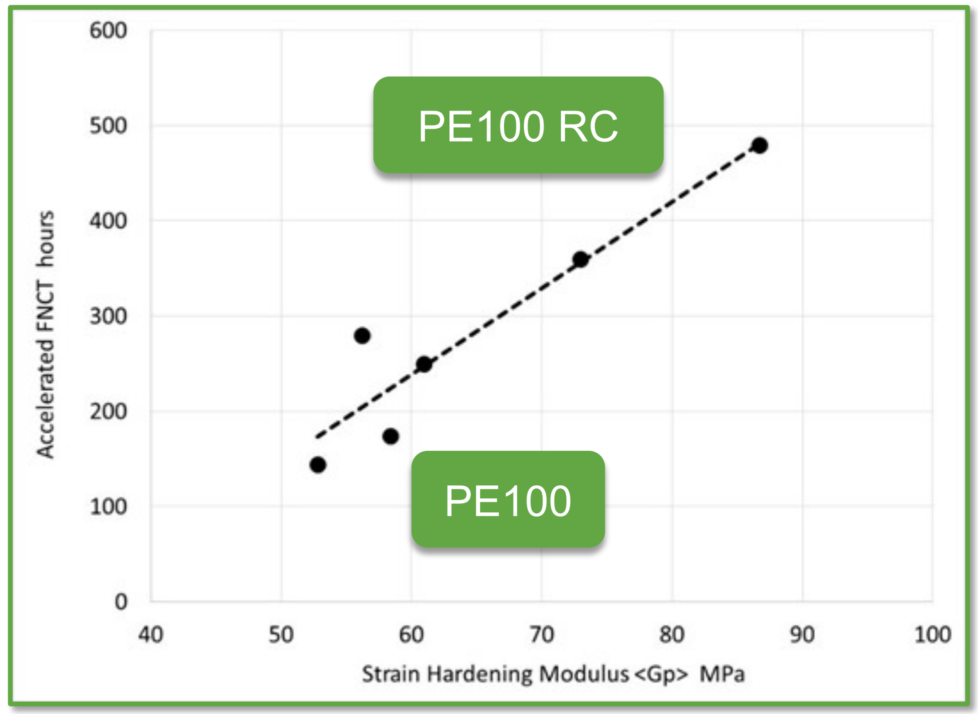
aFNCT vs. SHM

- aFNCT is used to characterize PE100 RC materials. Failure times are relatively long for batch release testing
- Materials used include C_6 and C_4 copolymers and different polymerization processes
- aFNCT conditions: 2% Dehyton, 90°C, 5.0 MPa
- Good agreement between SHM and aFNCT



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Conclusions

- New generations of high-performance PE100 and PE4710 type materials present challenges for testing developmental products and for production QA testing
- SCGR has been improved by lowering the density of the HMW component and optimizing the distribution of comonomer across the MWD. SCGR of these materials by traditional tests exceeds 1 year
- The SHM showed good correlation with NPT, aFNCT and PENT
- SHM, with the shortest time required to achieve results, is especially well-suited for batch release testing

Thank You!

Questions?

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