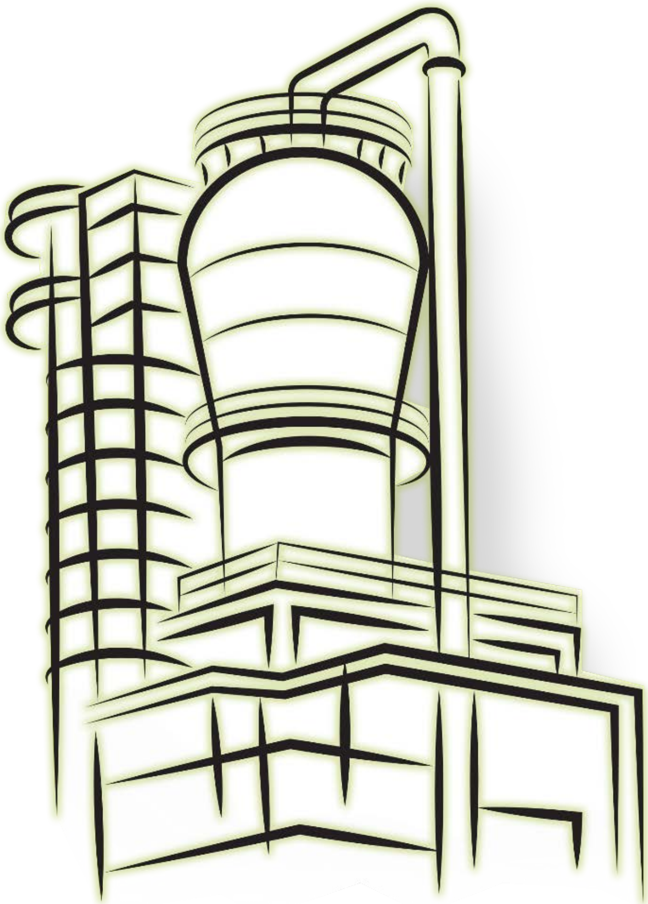
TECHNOLOGY TO **LEAD**
SERVICE TO **SUCCEED**
INNOVATION TO **GROW**



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TECHNOLOGIES

Advances in HDPE Technology for Large Diameter PE100 Pipe Applications

2017 SPE International Polyolefins Conference
Houston, TX
February 26 – March 1, 2017

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Topics

- PE100 Pipe Applications
- PRODIGY™ HD Advanced Metallocene Bimodal Technology: Resin, Process, and Catalyst
- Polymer Properties
- Extrusion Processability
- Large Diameter Pipe Case Studies
- PE100 Pipe Standards
- Electrofusion Test Program

PE Pipe Applications

- Advantages relative to other materials
  - Balance of stiffness, toughness
  - Flexible – easy to install
  - Corrosion resistance
- Innovations in PE material technology are allowing PE100 pipes to be used in a wider range of applications
- PE piping systems are increasingly specified for large diameter projects.
  - Water supply
  - Mining slurries
  - Coal seam gas
- PE100 Pipe Industry Trends
  - Higher strength
  - Large/thick pipes

Photos: Courtesy Qenos, Ltd

PRODIGY™ HD Advanced Metallocene Bimodal Technology –
Inherently Superior Bimodal Technology

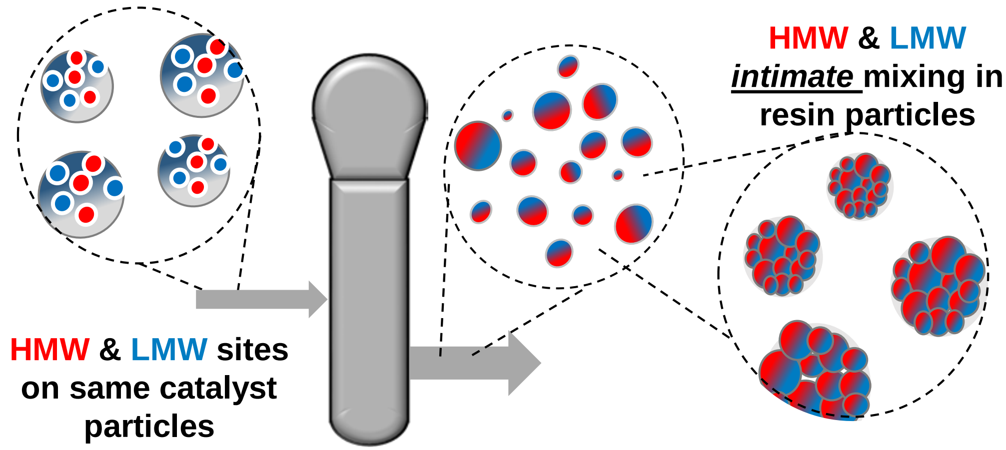
Inherently superior properties vs Ziegler-Natta bimodal

- Superior PE100 pipe
- Low gel film due to intimate mixing of HMW & LMW domains
- Optimized balance between high ESCR, high top load & tune-able melt strength for blow molding
- Properties through design of catalyst chemistry

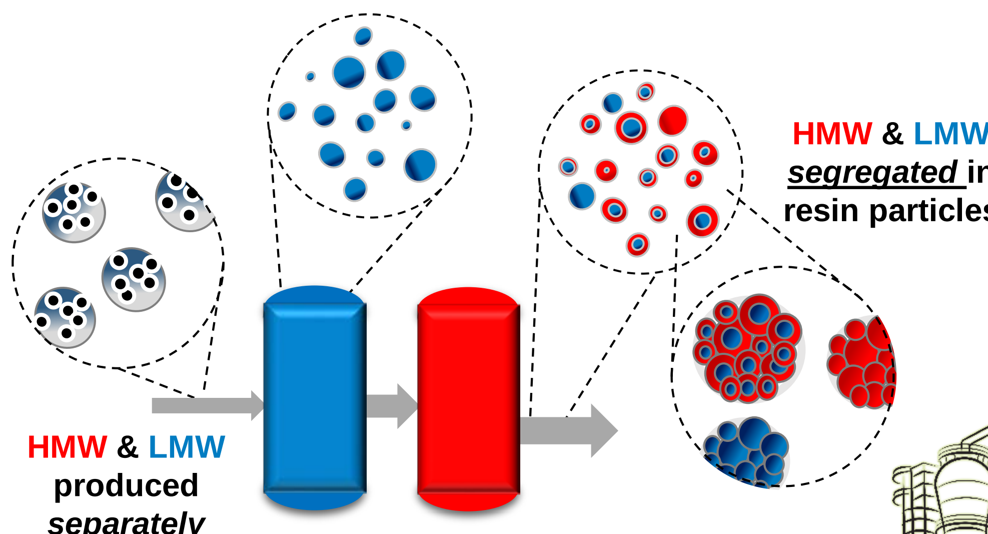
Intimate mixing within resin particles

- Z-N-based technology produces different resin in each reactor then must attempt to mix in compounder

UNIPOL™ PE Process & PRODIGY™ Bimodal Catalysts



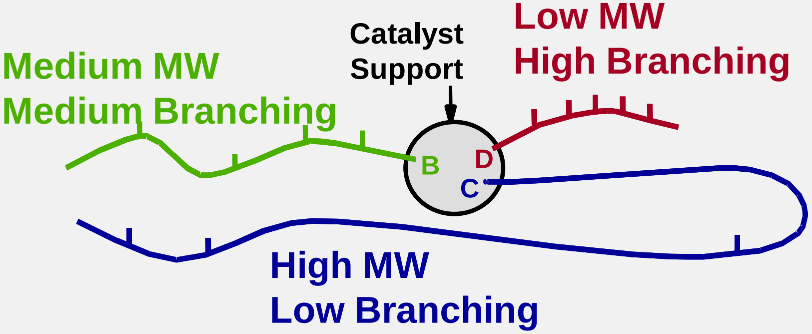
Conventional Staged Dual / Multi-Reactor Process



PRODIGY™ HD Advanced Metallocene Bimodal Technology –
Engineered Properties for Superior Performance

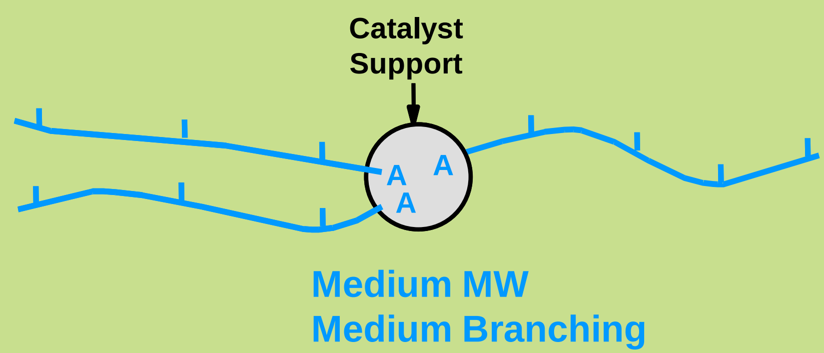
Ziegler-Natta and Chromium Catalysts

- Different types of reaction sites on each catalyst particle
- Broad range of molecular weights and comonomer distributions



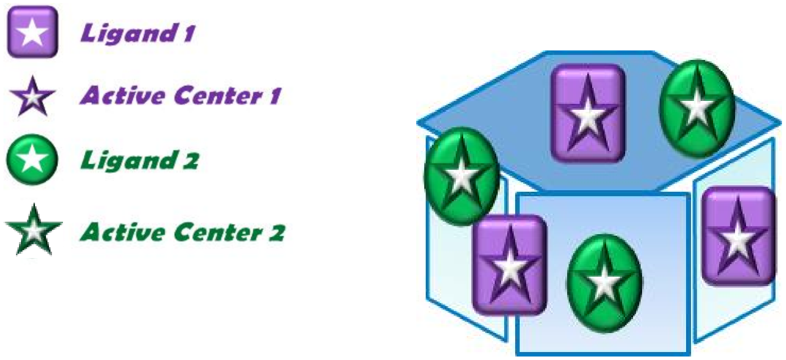
Metallocene Catalysts

- Single, consistent reaction sites
- Uniform molecular weights and comonomer distribution
- Precision-tailored polymer properties for advantaged performance



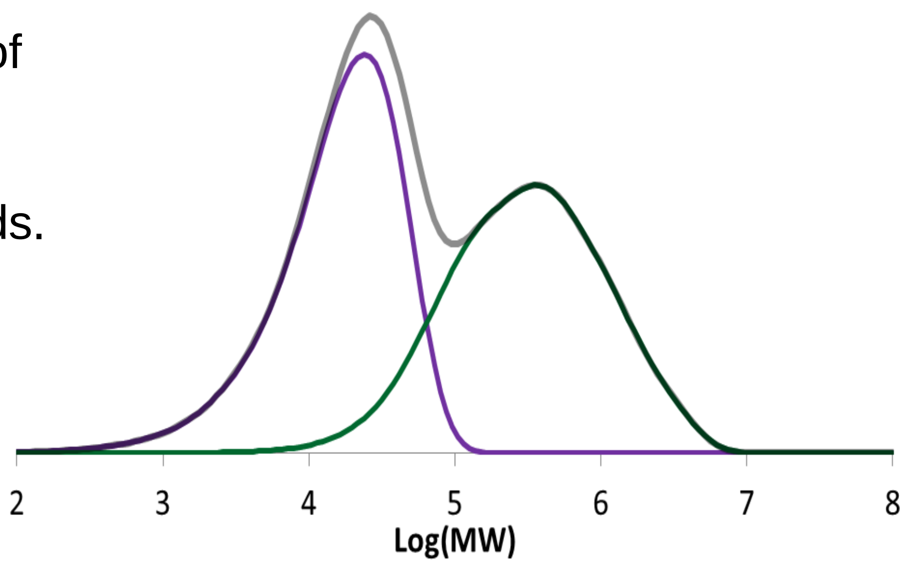
Metallocene Technology – expanding the reach of PE applications

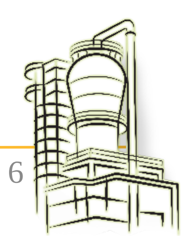
PRODIGY™ HD Advanced Metallocene Bimodal Technology – Unique Bifunctional Polymerization Catalyst



- Two very different active sites on the same support.
- Ligands are selected to produce active sites with
  - Different hydrogen responses
  - Different comonomer responses

- Polymer characteristics are influenced by the environment of the active catalyst centers.
- Active sites are surrounded by organic molecules, called ligands.
- The nature of the polymer is determined by the ligand.

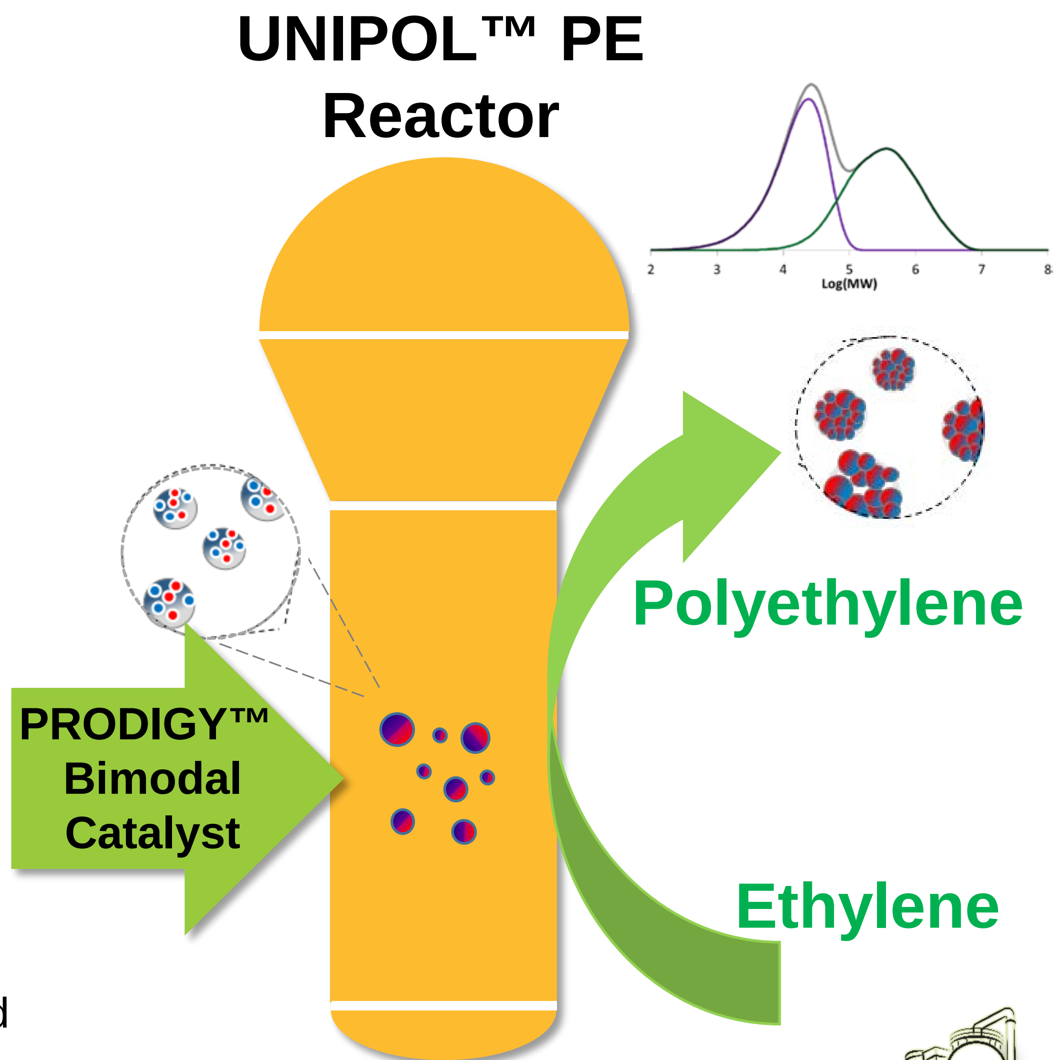




PRODIGY™ HD Advanced Metallocene Bimodal Technology – Enables Single-Reactor bimodal HDPE Production

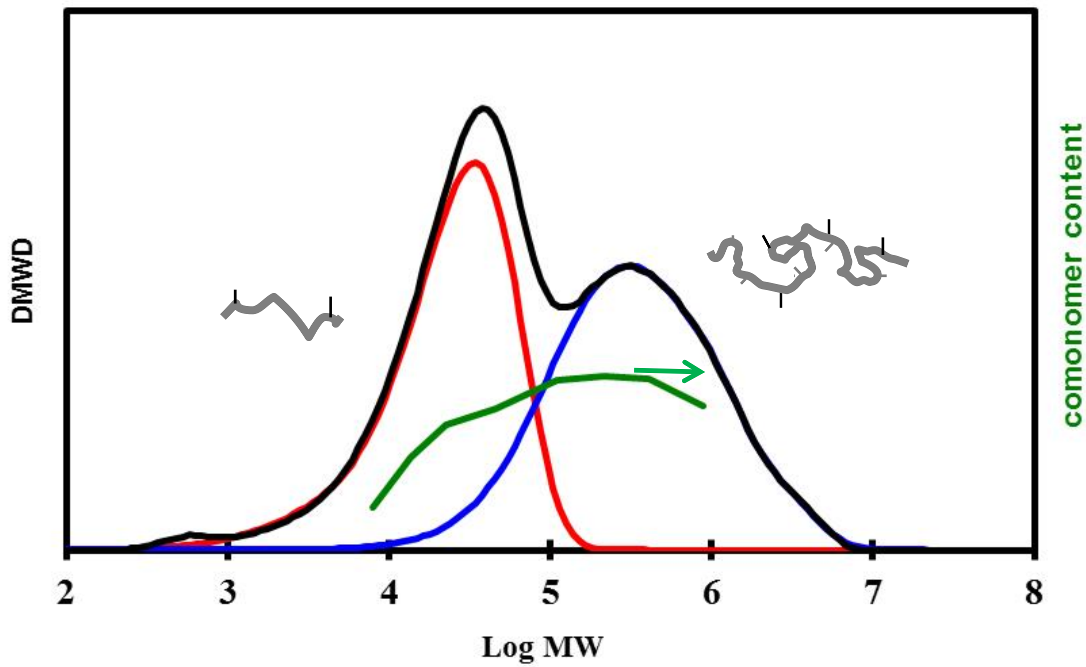
Polymer Property Control

- Active sites and ligands for each of 2 catalyst components determines relative:
  - Mw
  - MWD
  - Relative comonomer incorporation
- Density and Melt Index Control
  - Reactor temperature
  - Gas composition, H_2, hexene
- The HMW and LMW components are intimately mixed on particles leaving the reactor
  - Good homogeneity
  - No special compounding needed



PRODIGY™ HD Advanced Metallocene Bimodal Technology – Bimodal Polymer Structure

- HMW Polymer
  - High comonomer
  - Tie Molecules
  - Toughness, SCGR
- LMW Polymer
  - Low comonomer
  - High crystallinity
    - Stiffness, pressure rating
- Broad MWD for processability

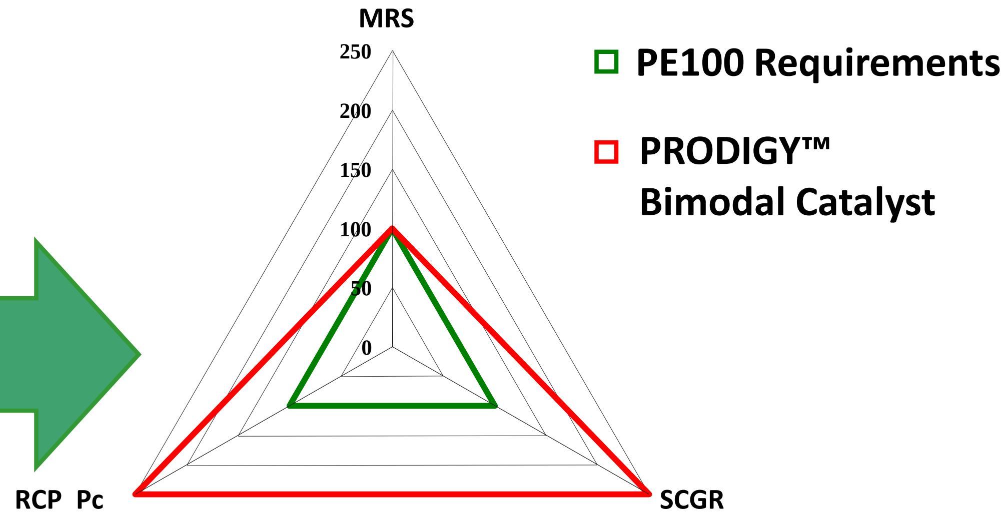


PRODIGY™ HD Advanced Metallocene Bimodal Technology –
Broad HDPE Resin Product Capability

Bimodal Film

Bimodal Blow Molding

Bimodal Pipe



- Long-term pressure rating – MRS10
- Resistance to slow crack growth
- Resistance to rapid crack propagation (RCP)

ISO PE100 Pipe Standards

- The single-reactor bimodal product was designed with component Mw, MWD, split and comonomer to optimize the mechanical properties and processing. The melt index fell outside of the range specified in 2007 versions of gas and water pipe standards.
- Revisions to ISO 4437 and ISO 4427 were made in 2014-2015 to accommodate PE100 materials with lower MFR_5.
  - Footnote to Table 1. For materials with $0.15 < MFR_5 < 0.20$ …. “attention is drawn to the fusion compatibility”
  - Section 6.3 in ISO 4437 states that, when electrofusion is to be used, “appropriate testing should be carried out to verify the fusion capability of such pipes”.
  - Fusion testing was conducted

Characteristic	Requirement [a]	Test parameters		Test method
		Parameter	Value	
Melt mass-flow rate (MFR) for PE 80 and 100	0,20 to 1,40 g/10 min [g] Maximum deviation of ± 20 % of the nominated value	Load	5 kg	ISO 1133-1
		Test temperature	190 °C	
		Time	10 min	
		Number of test pieces [b]	According to ISO 1133-1	

[g] Materials of nominated value 0,15 ≤ MFR < 0,20 may be introduced, in such case attention is drawn to the fusion compatibility subclause 4.5. The lowest MFR value resulting from the maximum lower deviation of the nominated value to be not less than 0,15.

PRODIGY™ HD Advanced Metallocene Bimodal – Meets PE100 Certification Requirements

- HDF145B produced by Qenos in Australia
  - Black Compound
  - Certifying lab: Exova
- International Standards
  - ISO 4427 – water pipe
  - ISO 4437 – gas pipe
  - EN12201 – water pipe (European)
  - EN1555 – gas pipe (European)
- Resin Properties
  - MI/density
  - Carbon black content/dispersion
  - Volatiles
- Pipe Properties
  - MRS10 – long-term pressure
  - SCGR
  - RCP
  - Resistance to gas constituents
  - Butt Fusion

TYPE TESTING

Type testing (TT) according to EN 1555-1:2010/ISO 4437-1:2014 and EN 12201-1:2011/ISO 4427-1:2007 of the black PE 100 pipe compound HDF145B from Qenos Pty Ltd produced with Univation PRODIGY™ Catalyst Technology

ABSTRACT

The aim of this project was to perform type testing (TT) of the PE pipe compound HDF145B according to EN 1555-1:2010/ISO 4437-1:2014 and EN 12201-1:2011/ISO 4427-1:2007. The ISO 9080 was performed by Exova and reported in Exova P-10/114, while the other tests have been carried out by Qenos Pty Ltd and Kiwa.

Tested characteristics according to EN 1555-1/ISO 4437-1 and EN 12201-1/ISO 4427-1

CHARACTERISTIC	METHOD	UNIT	REQUIREMENT	RESULT	PASS/FAIL
Compound density	ISO 1183-1:2004 (A)	[kg/m³]	≥930	961	Pass
Carbon black content	ISO 6964:1986	[wt %]	2.0-2.5	2.2	Pass
Carbon black dispersion	ISO 18553:2002	-	≤ Grade 3	1.5	Pass
			A1, A2, A3, B	A2, A3	Pass
Melt mass flow rate (5kg/190°C)	ISO 1133-1:2011	[g/10 min]	0.2-1.4	0.19	Pass
Oxidation induction time (200 °C)	ISO 11357-6:2001 EN 728:1997	[min]	≥20	91	Pass
Volatile content	EN 12099:1997	[mg/kg]	≤350	233	Pass
Tensile strength for butt-fusion	ISO 13953:2001	-	Ductile	Ductile	Pass
RCP-S4 (250 mm SDR 11)	ISO 13477:2008	[bar]	10*	≥10	Pass
Resistance to slow crack growth	ISO 13479:2009	[h]	500	1 152	Pass
Gas condensate resistance	ISO 1167:2006	[h]	≥20	≥20	Pass
MRS Classification	ISO 12162:2009	[MPa]	10	10	Pass

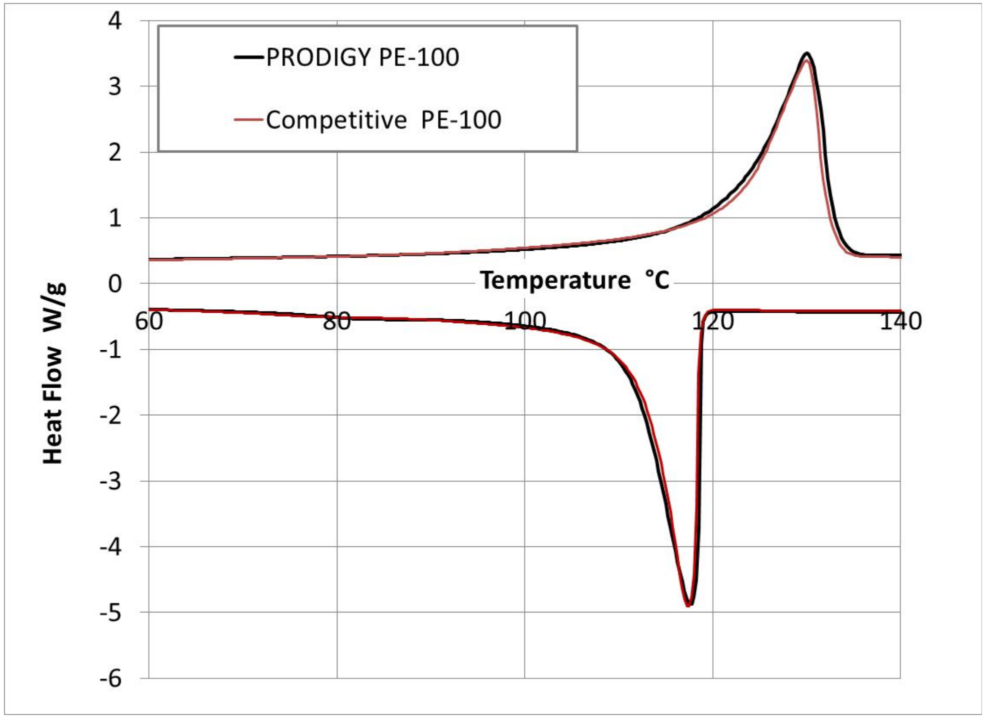
* *The requirement in EN 1555-1:2010 is 10 bar and in ISO 4437-1:2014 it is 3.4 bar (using a design coefficient, C, of 2).*

The PE pipe compound HDF145B with a MRS of 10 MPa shows conformity with the type testing requirements for a PE 100 pipe compound according to EN 1555:2010/ISO 4437:2014 and EN 12201:2011/ISO 4427:2007.

PRODIGY™ HD Advanced Metallocene Bimodal – Thermal Properties

- The crystallinity is comparable to that of typical PE100s
  - DSC melting and crystallization behavior are comparable
  - Melting and cooling behavior will be comparable in fusion processes

DSC Melting and Crystallization

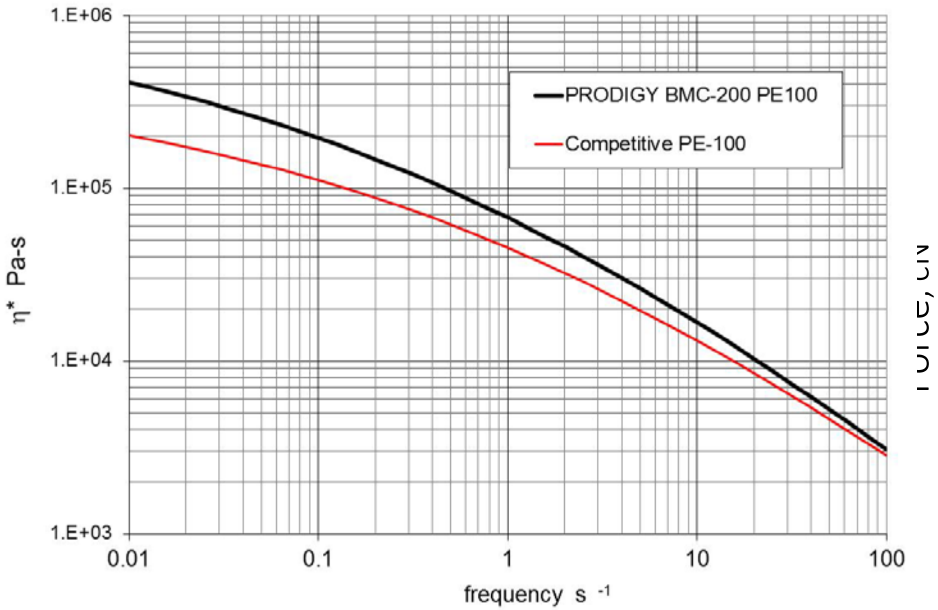


PRODIGY™ HD Advanced Metallocene Bimodal – Rheology

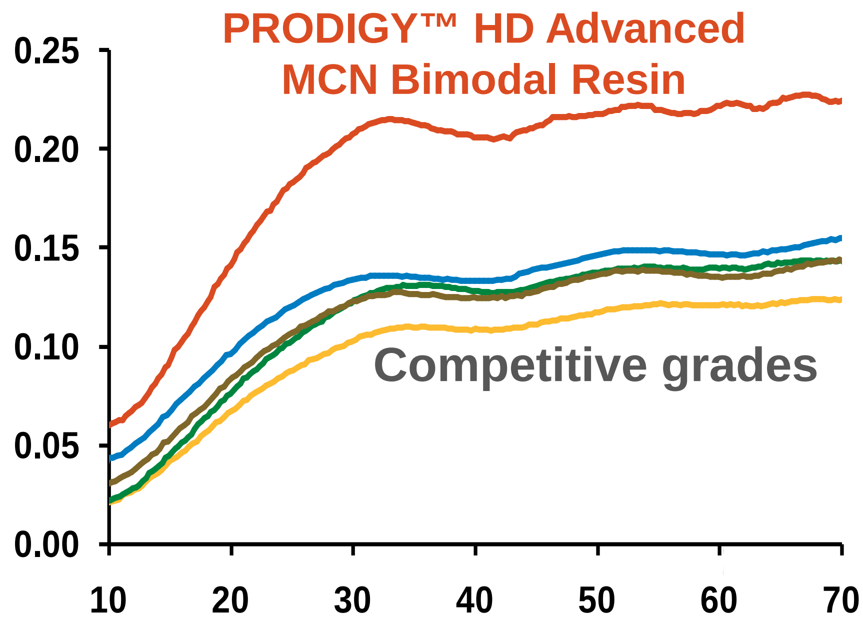
- The MFR_5 is 0.15 – 0.18 compared to the typical MFR_5 of 0.25 – 0.30 for other PE100s

- The broad MWD results in a high degree of shear thinning
  - High viscosity at low shear
  - Similar viscosity at high shear
- Rheotens melt strength is very high

Viscosity at 190°C

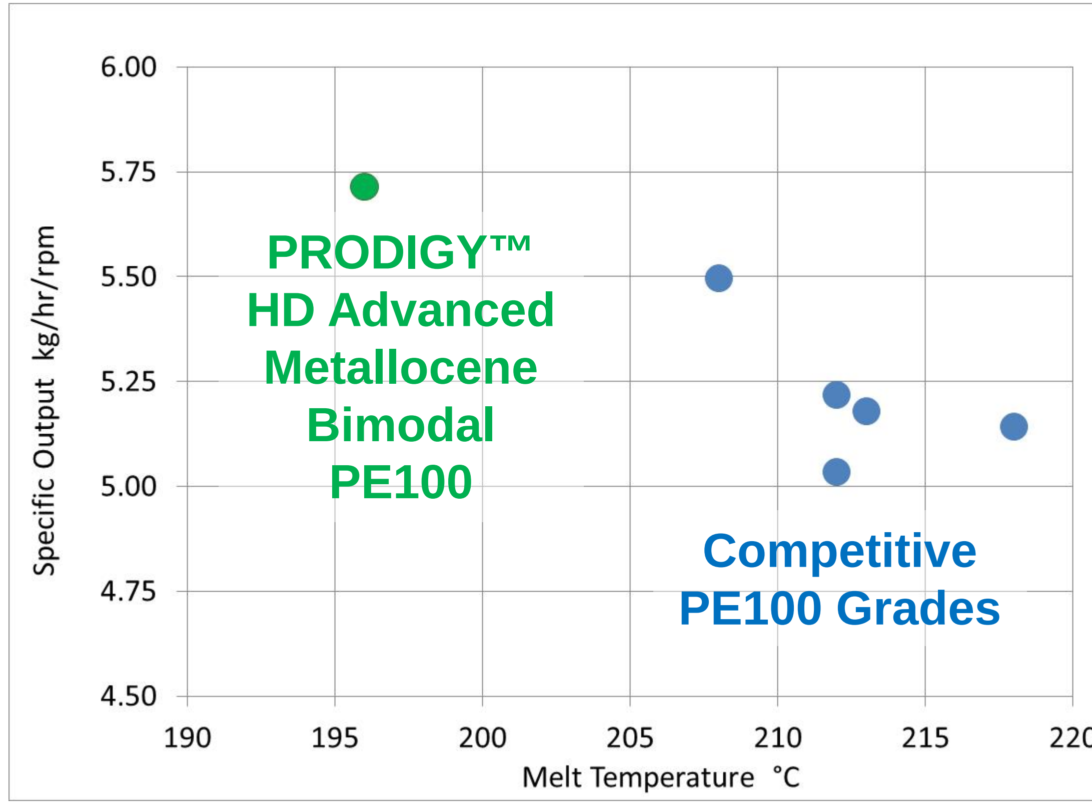


Melt Strength at 190°C

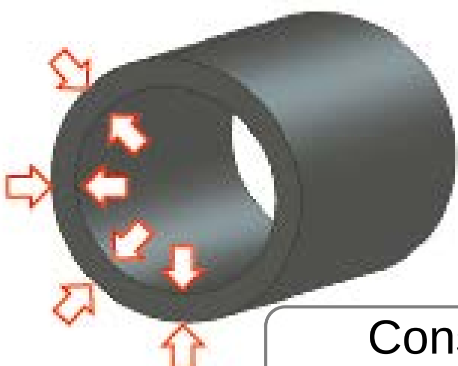


PRODIGY™ HD Advanced Metallocene Bimodal – Extrusion Processability

- At constant conditions, output rate and line speed, the lower MFR_5 resin has exhibited:
  - Lower melt temperature
  - Higher specific output, kg/hr/rpm
- Benefits for pipe production
  - Less cooling required → increased production capacity
  - High viscosity/low sag results in consistent wall thickness distribution



Extrusion of 250 mm SDR11 pipes
90 mm grooved-barrel extruder



Consistent Wall Thickness Distribution

PRODIGY™ HD Advanced Metallocene Bimodal –
Delivers Consistent Pipe Wall Thickness

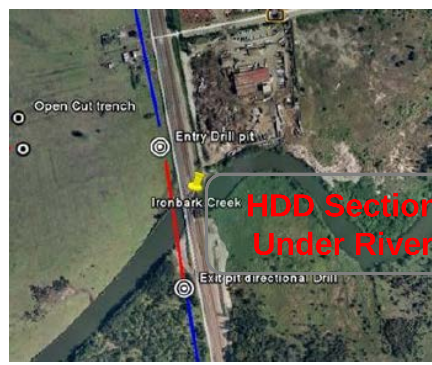
- Excellent pipe dimensional stability
- Raw material savings with lower target wall thickness
- More efficient pipe installation due to pipe matching during welding
- Reduction in need for routing of pipe walls before welding

1,200 mm diameter, 100 mm thick water pipe with excellent thickness consistency

Case Study 1: Water Pipe Installation

PRODIGY™ HD Advanced Metallocene Bimodal Resin for PE100 pipe gives exceptional performance in large pipe projects requiring high melt strength.

- In 2011, a section of a water distribution pipeline in Australia needed replacement
  - 900 mm diameter steel
  - Corroded, required maintenance
- PE100 chosen for the new pipeline
  - 1,000 mm PN16 pipe
  - Qenos Alkadyne HDF145B (with PRODIGY™ BMC Catalyst)
  - Corrosion resistance
  - Flexibility – ease of installation
- Sensitive environment - wetlands



- P. Micic, T. Campbell, Challenging installation of large diameter HDPE pipe by directional drilling provides a comprehensive water management solution and an improved environmental outcome, Plastic Pipes Symposium XVI Conference Proceedings, September 24-26, 2012

Case Study 1:
Water Pipe Installation cont'd

PRODIGY™ HD Advanced Metallocene Bimodal Resin for PE100 pipe gives exceptional performance in large pipe projects requiring high melt strength.

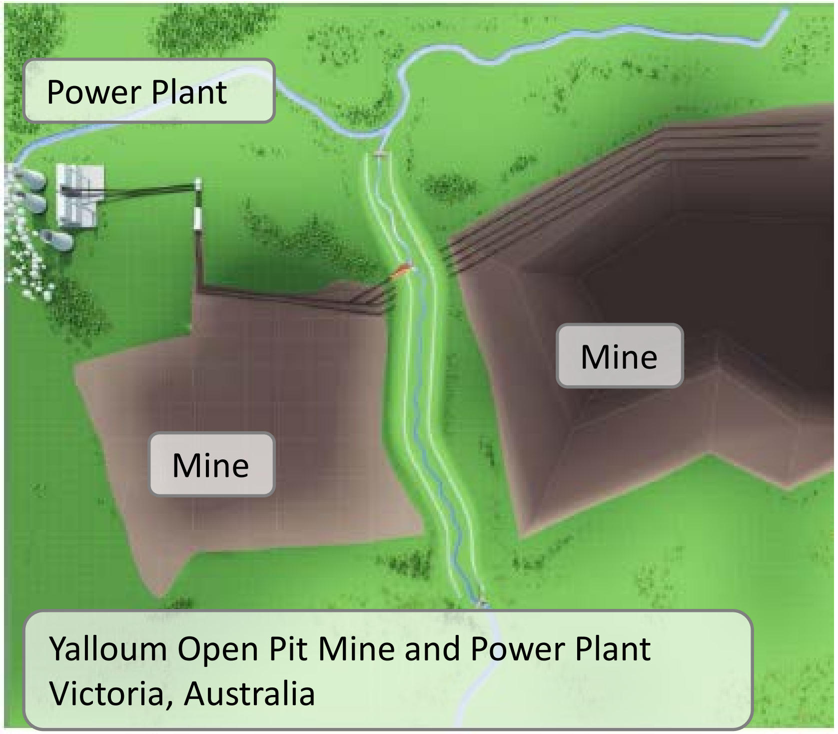
- Horizontal directional drilling (HDD)
  - 300 meters of HDD PE100 replaced a section of steel pipe on a bridge
  - Section of pipe was pulled under the riverbed
  - Less environmental impact

Photos: Courtesy Qenos, Ltd

Case Study 2: River Flow Diversion

PRODIGY™ HD Advanced Metallocene Bimodal Resin for PE100 pipe gives excellent processability for fast delivery in emergency situation.

- In 2012, an open pit mine in Victoria, Australia was flooded
  - Historical rainfall
  - Levee was breached
  - Power plant is directly sourced from this mine



- P. Micic, G. Beckton, 1600mm PE Pipe Installation Saves Power Supply in Victoria, Australia, Plastic Pipes Symposium XVII Con ference Proceedings, September 22-24, 2014

Case Study 2: River Flow Diversion cont'd

PRODIGY™ HD Advanced Metallocene Bimodal Resin for PE100 pipe gives excellent processability for fast delivery in emergency situation.

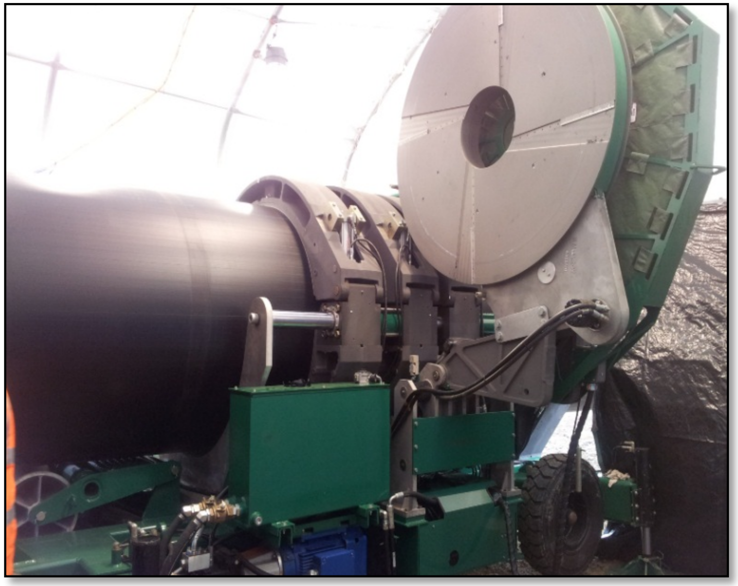
- Emergency response – 2 phases:
  - Dewatering the mine
  - Diversion of river
- PE100 pipes were chosen as the preferred method to move the water
  - Critical timeline
    - Large PE100 pipes could be extruded in Australia
  - Project completed in about 2 months
- Qenos HDF145B was used
  - Pressure rating, SCGR
  - High melt strength
  - Extrusion output

Photos: Courtesy Qenos, Ltd

Case Study 2: River Flow Diversion cont'd

PRODIGY™ HD Advanced Metallocene Bimodal Resin for PE100 pipe gives excellent processability for fast delivery in emergency situation.

- 1,600mm diameter, 80mm wall thickness pipe butt welded in the field
- Installation time critical
- Tight wall thickness and ovality tolerance of HDF145B pipe alleviated need for routing and doubled weld rate



Butt Welding in the Field

Photos: Courtesy Qenos, Ltd

Case Study 2: River Flow Diversion cont'd

PRODIGY™ HD Advanced Metallocene Bimodal Resin for PE100 pipe gives excellent processability for fast delivery in emergency situation.

- For dewatering the mine, 500 mm and 630 mm diameter pipes were used
- River diversion, 1,600 mm diameter pipes were used

Photos: Courtesy Qenos, Ltd

Electrofusion Test Program Large Diameter Pipes

Electrofusion was successfully demonstrated for joining large pipes produced with PRODIGY™ HD Advanced Metallocene Bimodal Resin for PE100 pipe and off-the-shelf fittings.

- Electrofusion study conducted to demonstrate compatibility of PRODIGY™ HD Advanced Metallocene Bimodal Resin derived pipes with other commercial materials.
  - MFR_5 of “off-the-shelf” fittings is about 0.30
  - MFR_5 of BMC-200 pipe is about 0.15 – 0.18
- Large pipes are most challenging case, up to 800 mm diameter
  - Thick
  - Potential for larger gaps between fitting and pipe
- Saddle and coupler joints formed
- A range of weld conditions used
  - Weld temperature (ambient): -10°C, +45°C
  - Controller weld energy – low, high
  - Maximum gap machined
- EF Joint Test conditions
  - Hydrostatic pressure testing according to EN 12201-3
  - Decohesion and strip bend testing according to ISO 21751
- All joint testing passed

An EF Saddle Fitting

Photos: Courtesy Qenos, Ltd

Electrofusion Test Program
Large Diameter Pipes

110 mm SDR17 water pipe and tapping saddle EF assembly ready for product testing

630 mm SDR17 gas pipe and EF coupler being lowered into hydrostatic bath

Ductile decohesion specimen

Photos: Courtesy Qenos, Ltd

Conclusions

- HDPE PE100 materials are an excellent choice for large diameter pipe applications.
- The PRODIGY™ HD Advanced Metallocene Bimodal Technology and the UNIPOL™ PE Process were used to produce a bimodal HDPE PE100 pipe with an excellent balance of mechanical properties.
- The PRODIGY™ HD Advanced Metallocene Bimodal Resin for PE100 pipe applications exhibited some unique rheology resulting in exceptional melt strength, which was advantageous for producing large diameter / thick-wall pipe.
- Electrofusion was successfully demonstrated for joining large pipes produced with PRODIGY™ HD Advanced Metallocene Bimodal Resin for PE100 pipe and off-the-shelf fittings.
- Qenos HDF145B bimodal HDPE pipe resin, produced with the PRODIGY™ HD Advanced Metallocene Bimodal Technology, was used successfully in very demanding large pipe applications.

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