Les silicones aux interfaces des composites produits par mise en œuvre réactive.

Silicones at interfaces of composites produced by reactive compounding

Dow Corning Europe s.a.

Surface & Interface Solutions Center (SISC)

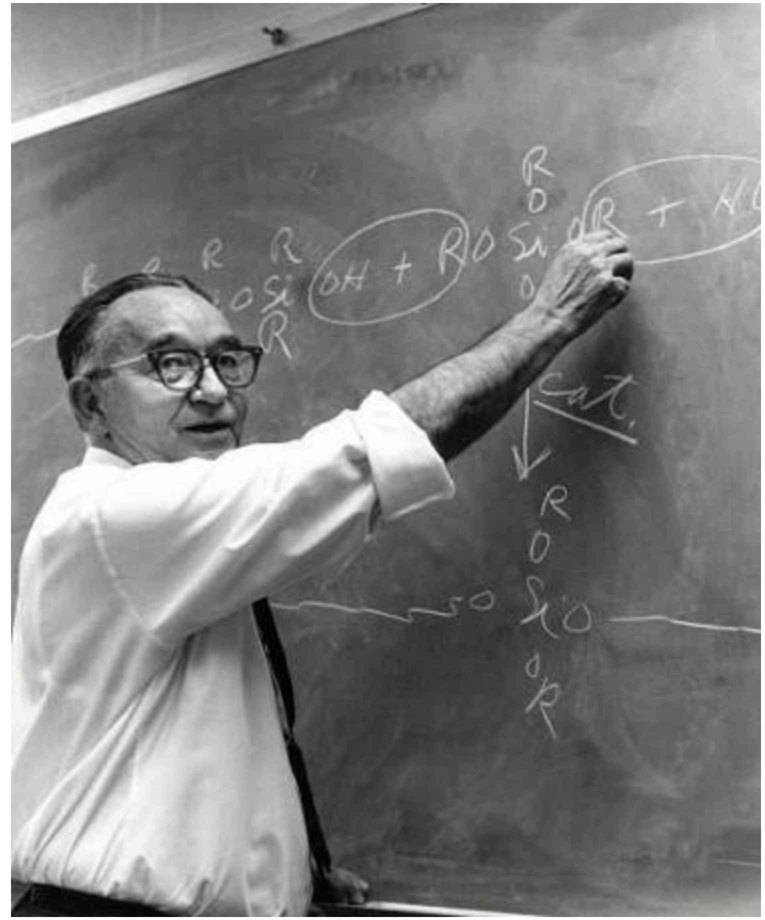
François de Buyl, Damien Deheunynck, Vincent Rerat

DOW CORNING

We help you invent the future™

Dow Corning…

- **A joint venture of The Dow Chemical Company and Corning Incorporated**
- **Organized to explore the potential of the silicon atom in 1943**



Dr. Franklin J. Hyde

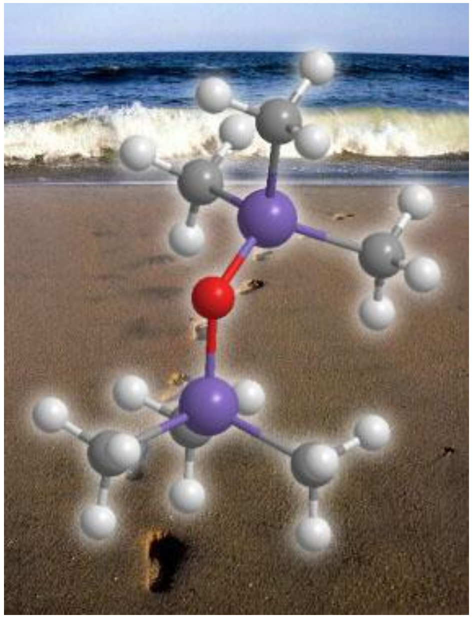
Fascinating Silicone™

Synthetic polymers

Silicon and oxygen

Carbon

Hydrogen



The missing link

Combining the characteristics of plastics and glass

Plastic and Composites

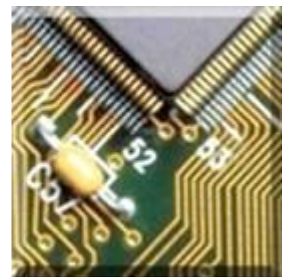
Applications For Virtually Every Industry

Automotive
Beauty & Personal Care
Chemical Manufacturing
Compound Semiconductor
Construction
Electronics
Food & Beverage
Healthcare
Household & Cleaning
Imaging
Paints and Inks
Power and Utility
Pressure Sensitive
Rubber Fabrication
Textile, Leather & Nonwovens







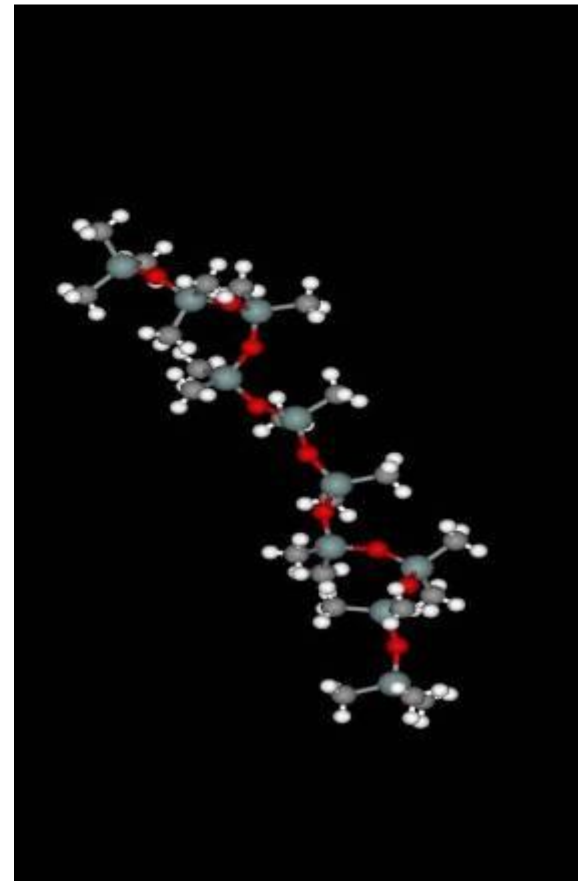





Almost endless applicability and more to come!

Dow Corning innovating around the world

- More than 7,000 products and related services
- Approximately 25,000 customers
- Over $5 billion in sales in 2008, globally dispersed with more than 60% outside the US
- Approx. 10,000 employees worldwide
- A global leader in silicones and high purity silicon
- Strong channels to market – distributors, web, commercial organization



Dow Corning – One Company, Many Big Opportunities

DOW CORNING

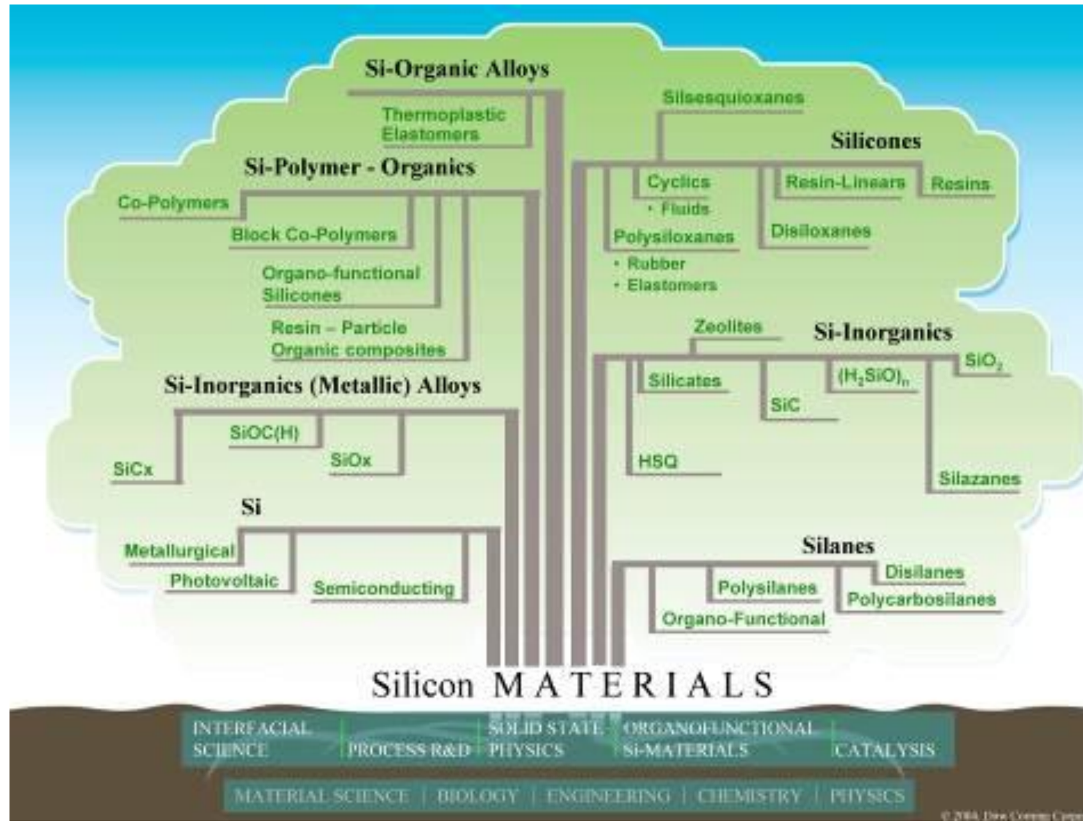
Dow Corning Corporation

HSC HEMLOCK SEMICONDUCTOR	XIAMETER from DOW CORNING	DOW CORNING *We help you invent the future.*™
Focus is on Polysilicon for Semiconductor and Solar Industries	Focus is on Products Standard Silicone Products *Relentless pursuit of efficiency*	Focus is on Markets Specialty Products & Services *Relentless pursuit of innovation*

6

Innovation is at the core of what we do

- 15-20% of our products and services are < five years old
- 4-5% of sales spent on R&D
- More than 4,000 active patents
- Beyond products -> solutions
- Approximately 50% of R&D spent on sustainability-related projects



Delivering Sustainability Benefits Through Product Innovation

Energy savings,
CO_2 reductions,
Water savings,
Natural resources preservation,
Waste reduction…

LEDs: Energy + $C0_2$ savings

Paper: Water savings

Lubrication: Energy savings

Textiles: Water savings

Construction: Energy efficiency

Tires: Fuel + $C0_2$ savings

Solar: Significant $C0_2$ savings

Agriculture: Water savings

Dow Corning Locations Worldwide

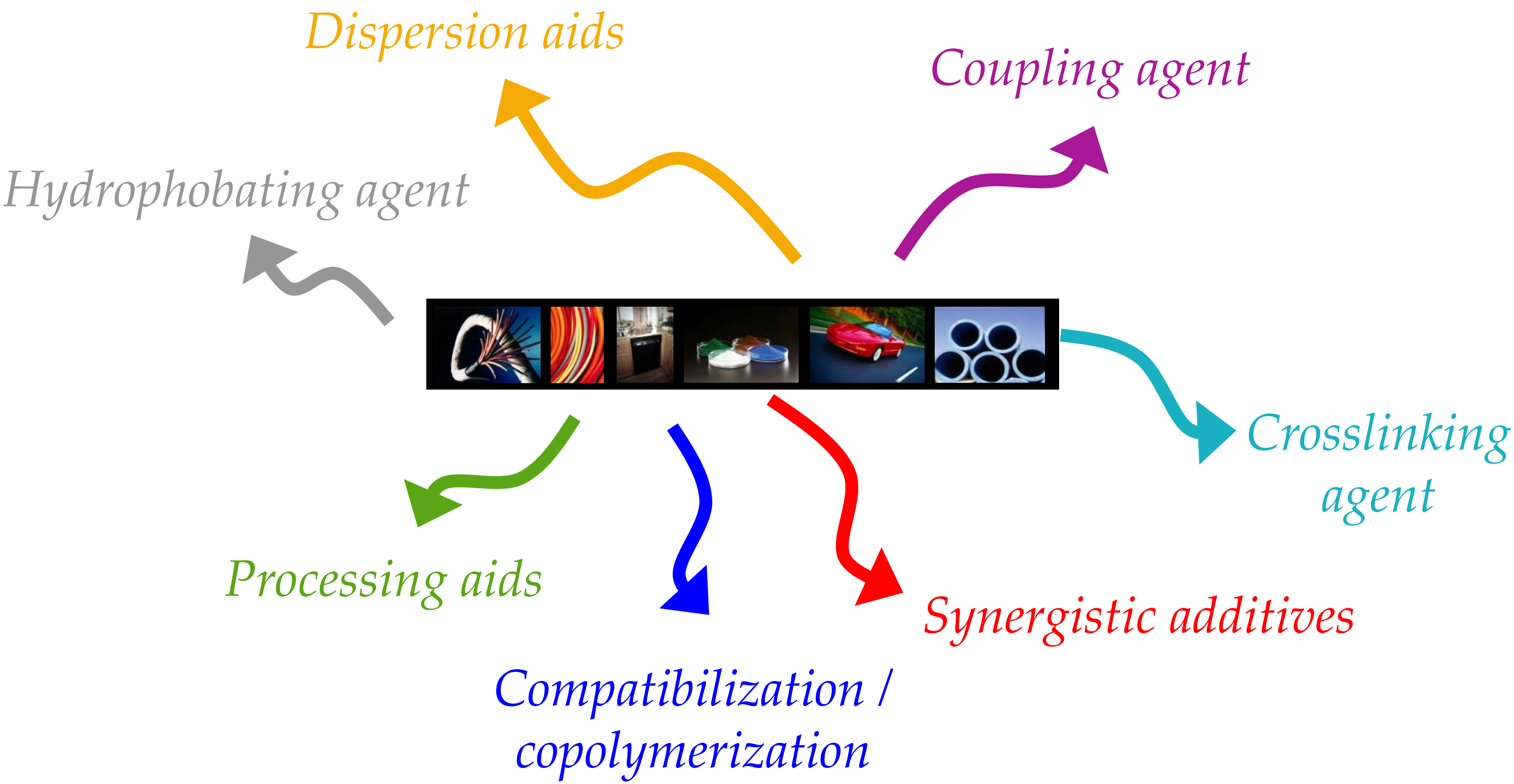
Surface & Interface Solutions Center (SISC)

Lab development capabilities established in Seneffe, Belgium, for:

- **Plastics & Composites Applications cell**
- **Rubbers Application cells**

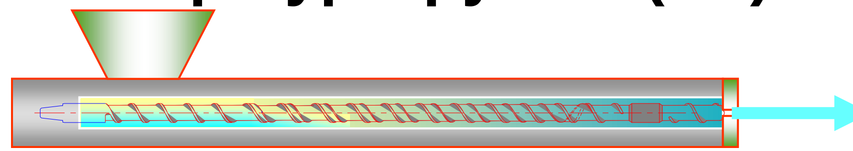
Global Team with worldwide networking

“Si” in Plastics & Composites



Case Studies

Melt extrusion reaction of silane to polypropylene (PP)

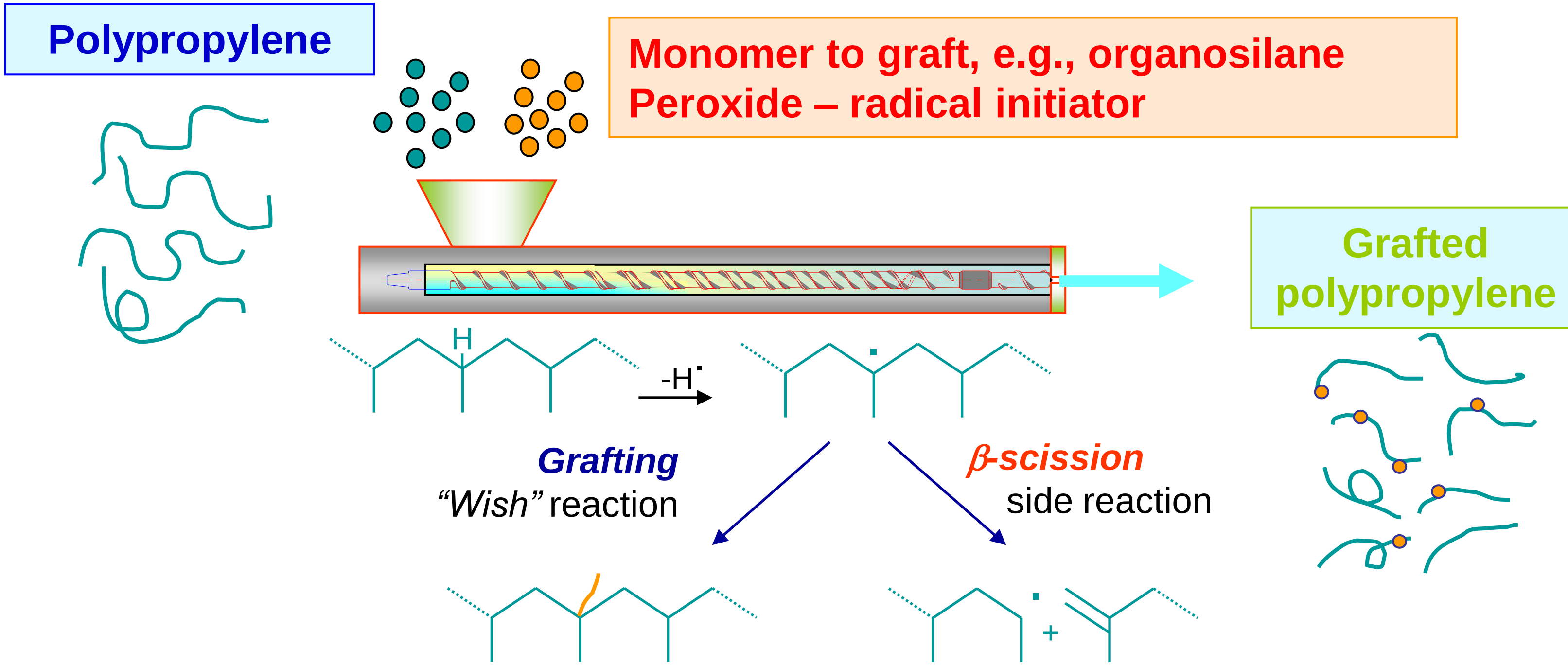


1. Water Uptake in Wood-Flours-PP Composites

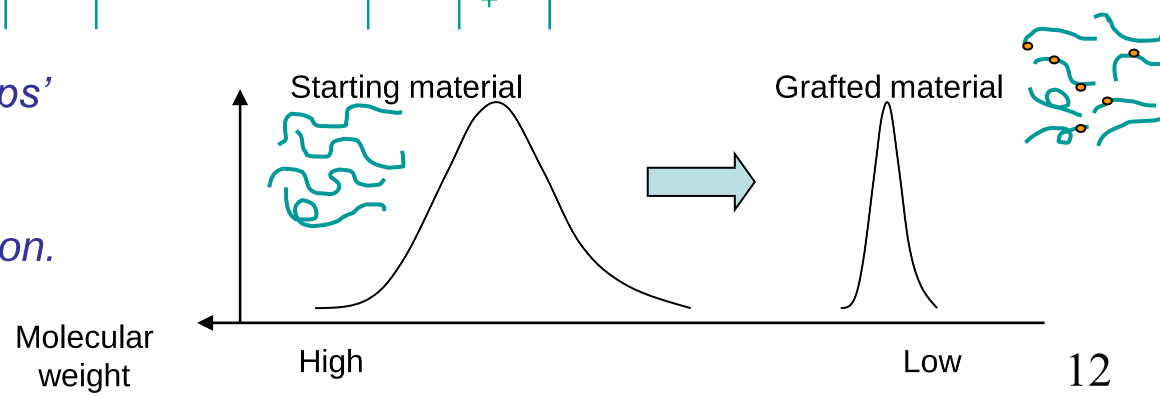
2. Heat Deflection Temperature in Glass-Fibers-PP Composites

Melt Extrusion Reactive Processing

Plastic and Composites

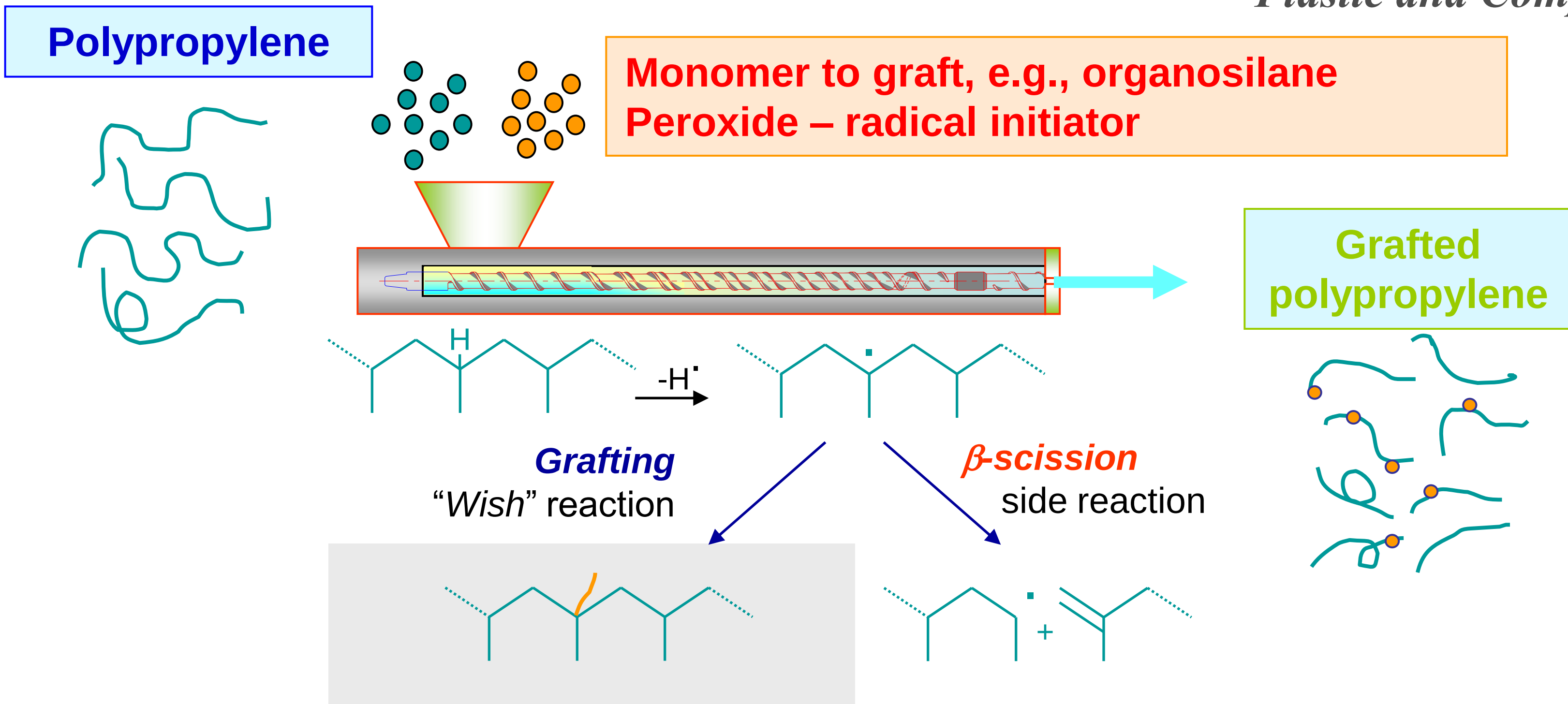


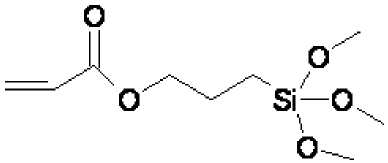
The speed at which the polymer 'unzips' typically occurs faster than any other desired reaction, leading to base polypropylene performance degradation.



Melt Extrusion Reactive Processing

Plastic and Composites



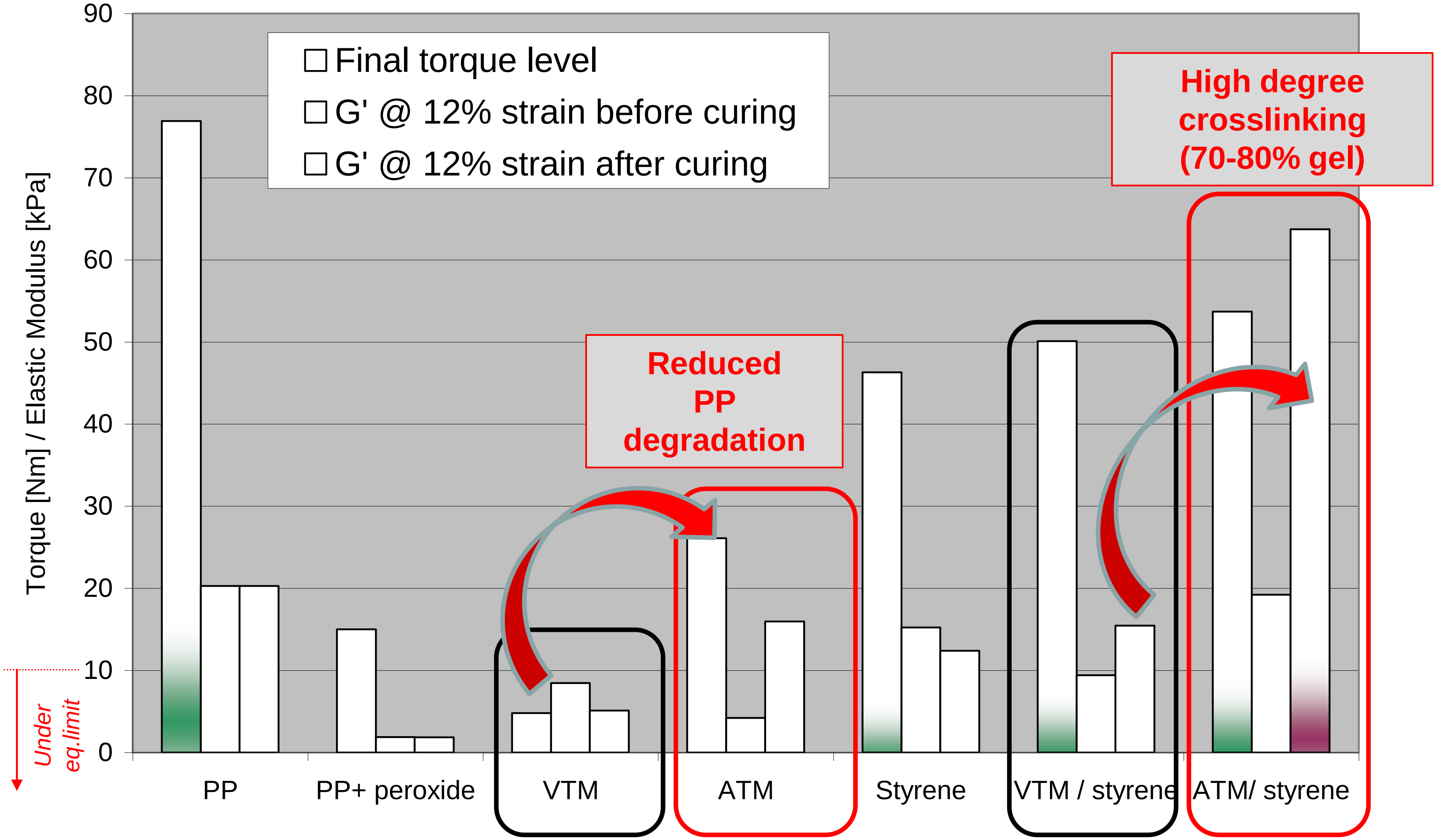


Acryloxy-trimethoxysilane (ATM)

Breakthrough discovery in favor to grafting of ATM (WO2010/000478-9) against prior works with, e.g, vinyltrimethoxysilane.

Functionalized PP

Plastic and Composites

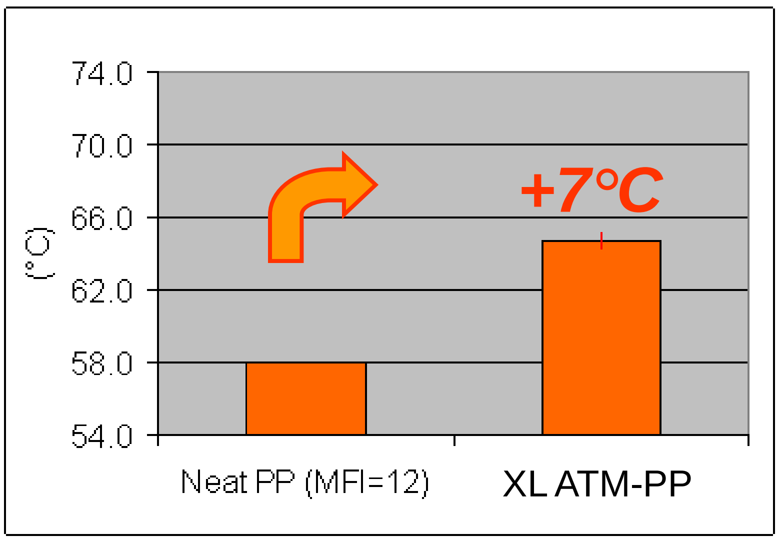


Crosslinking PP

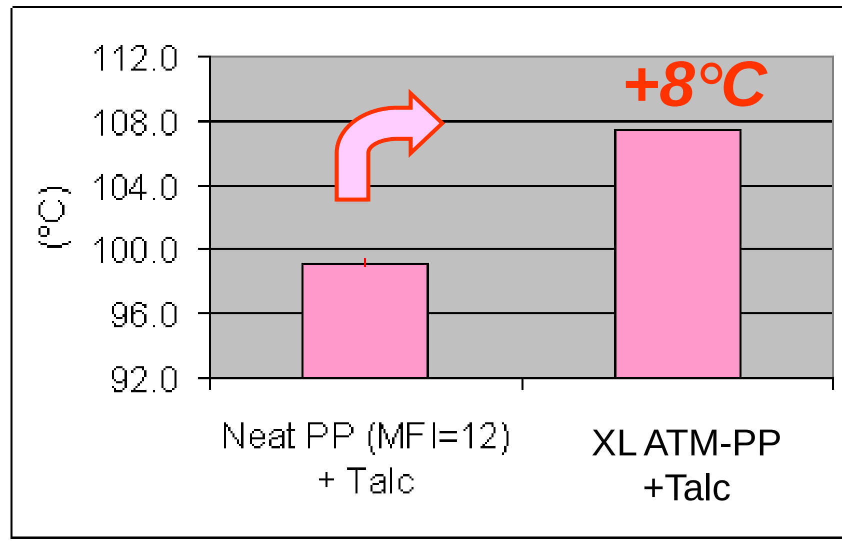
Plastic and Composites

Heat deflection temperature - ISO 75-1 Meth. A (1.8 MPa)

Polypropylene



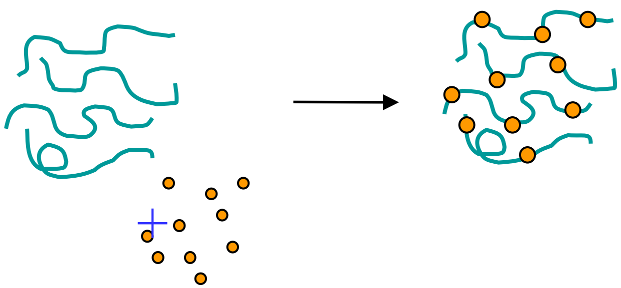
30 wt% Talc filled Polypropylene



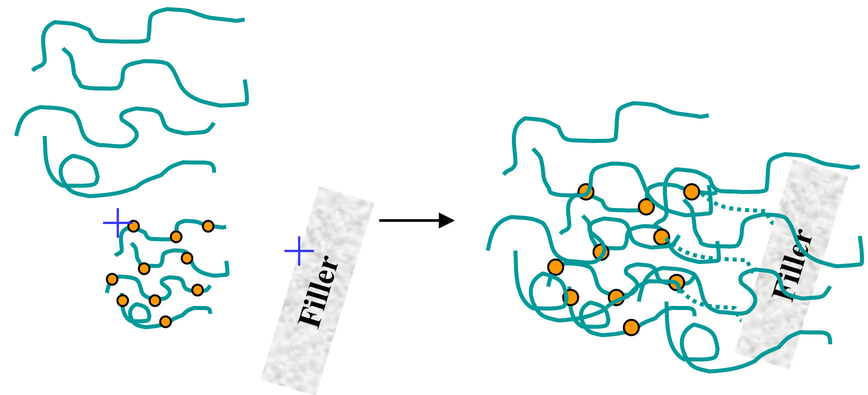
Melt Extrusion Reactive Processing in Presence of Fillers

Plastic and Composites

Pre-grafting

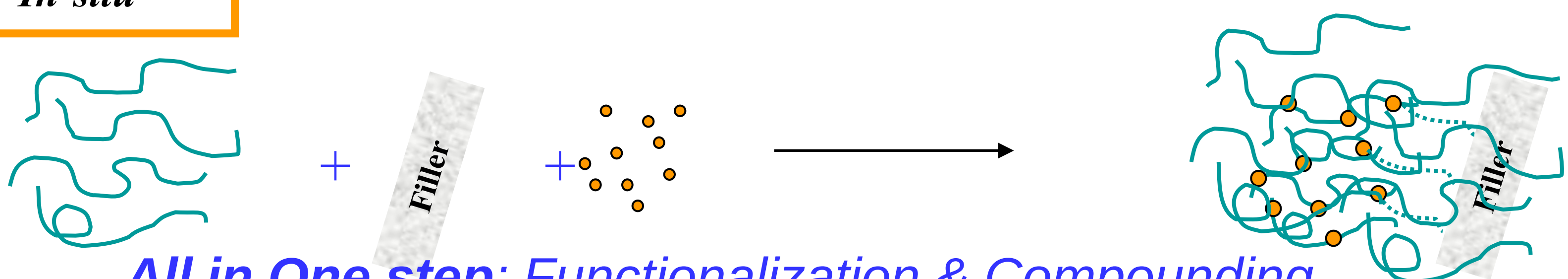


First step: Functionalization



Second step: Compounding

In-situ

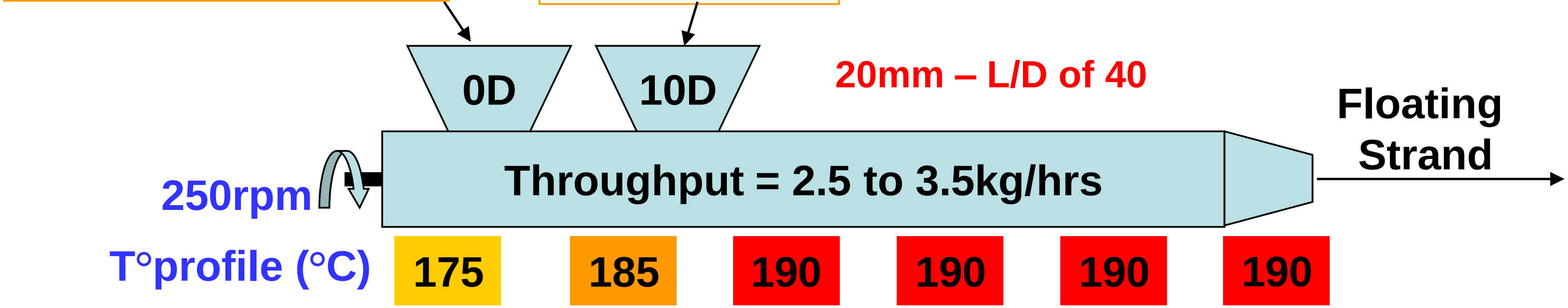


All in One step: Functionalization & Compounding

Wood-Flours-PP

30w% pine tries wood flours

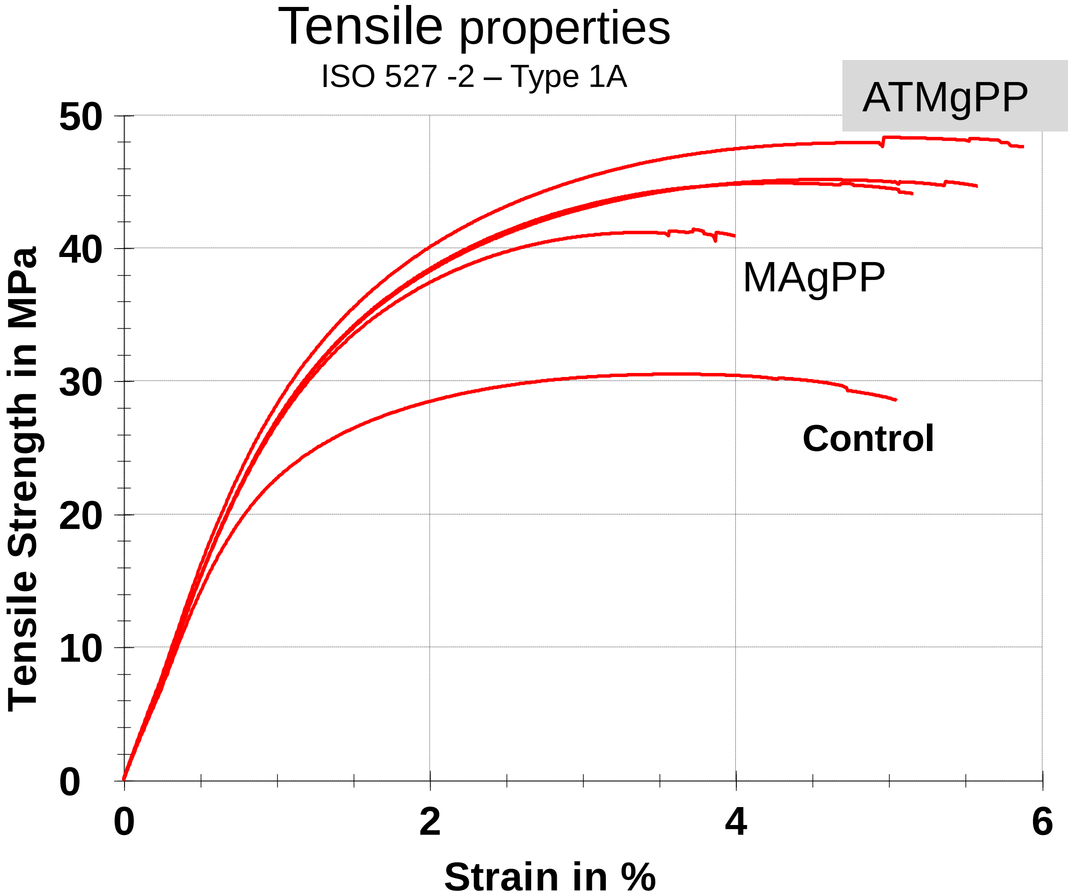
~200-500µm sizes



Residence time ~ 2 – 2.5min

Wood-Flours-PP

Plastic and Composites



- Increased Tensile Modulus > 3000MPa
- Increased Impact Strength > 23kJ/m2
- Long-term aging stability (underwater, heat)

Neat PPh resin: **E_{mod}=1350 Mpa; Tensile Strength max.= 32 Mpa; Strain max. = 10%**

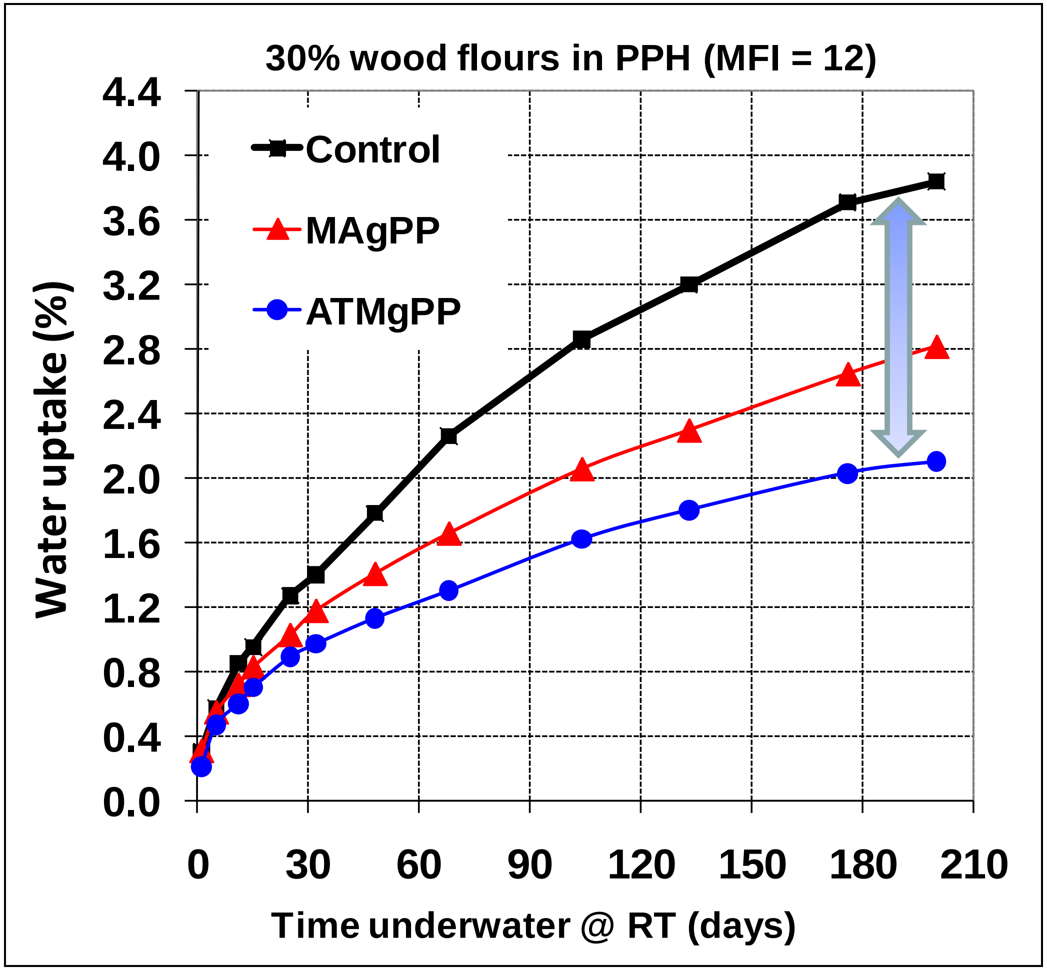
Wood-Flours-PP

Plastic and Composites

- ATMgPP coupling provides long-term stability upon aging, e.g., > 1,000hrs underwater/90°C and air/150°C, without any sign of deterioration.

Wood-Flours-PP

Plastic and Composites



- Significantly reduced water uptake upon time with ATMgPP coupling, i.e., 2w% / 6 months only.
- Diffusion of water significantly slowed down thanks to hydrophobicity of silane coupling between WF and PP resin.





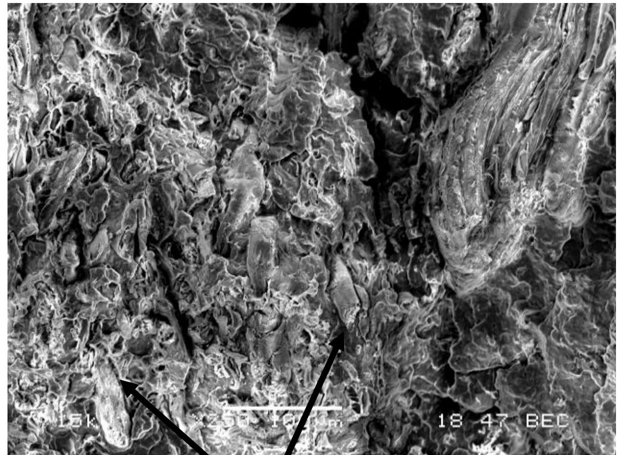
Wood-Flours-PP

Plastic and Composites

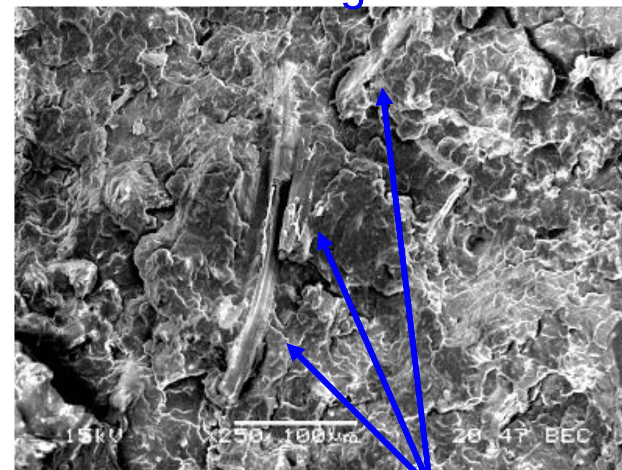
Scanning Electron Micrographs

Control

MAgPP



ATMgPP



No coupling

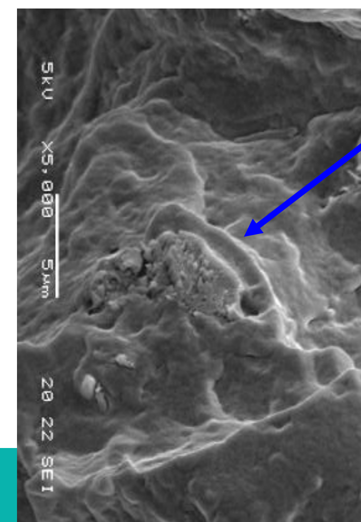
Wood Fiber delaminating from PP resin

Heterogeneous structure

Partial coupling

Partial coupling

Fibers delaminating from PP resin



Coupling

Wood Fiber *adhesion* with PP resin

Homogeneous structure

Glass-Fibers-PP

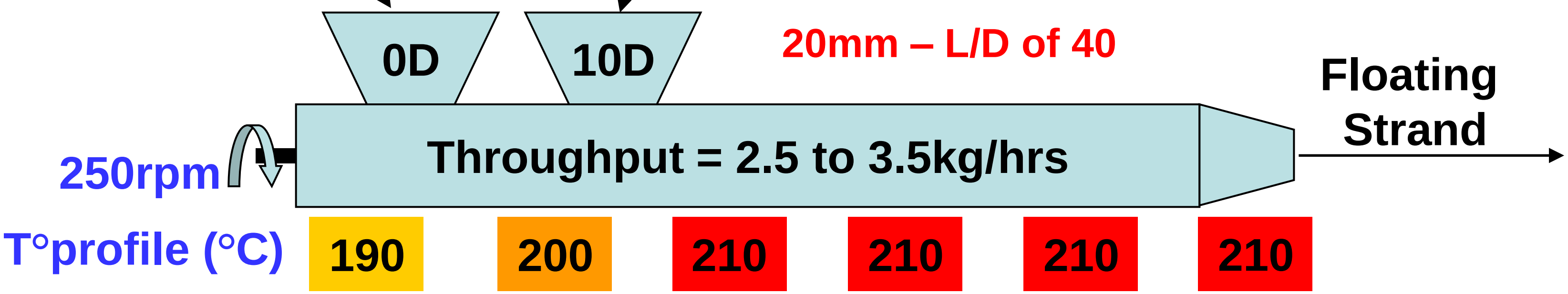
Plastic and Composites

Neat PP (MFI=12)+
[ATM:coagent:Peroxide]
+AOx MB

Glass-Fibers

30w% chopped strand

4mm length, 13µm diameter

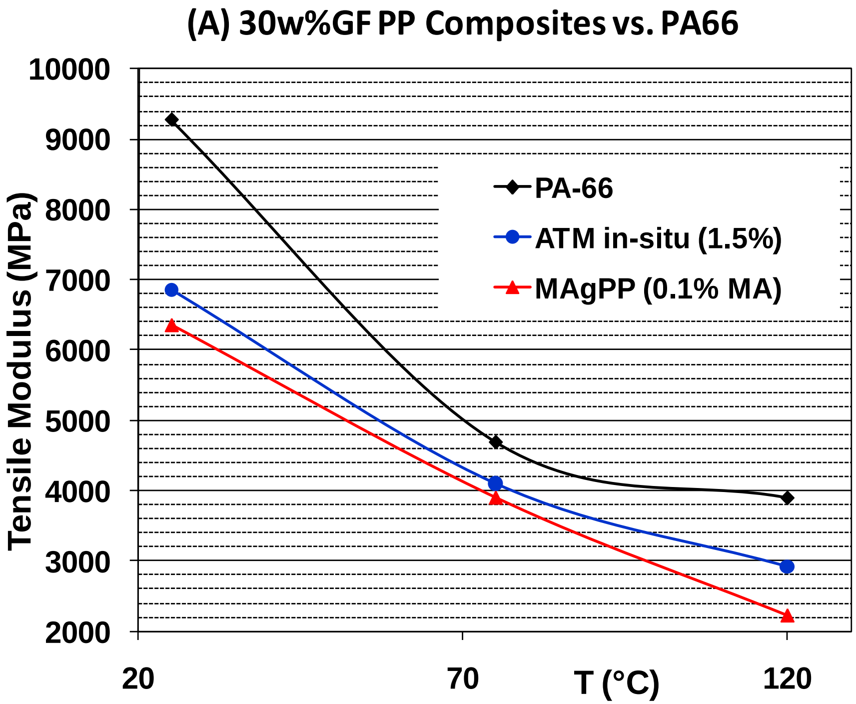


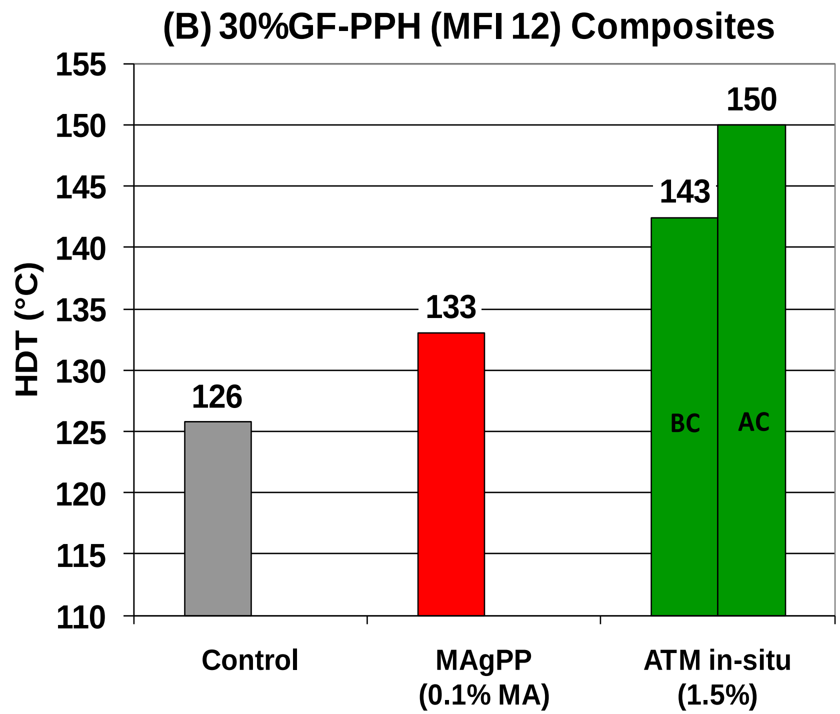
Residence time ~ 2 – 2.5min

Glass-Fibers-PP

Plastic and Composites

Tensile and Heat Deflection Temperature on ISO 527 -2 – Type 1A



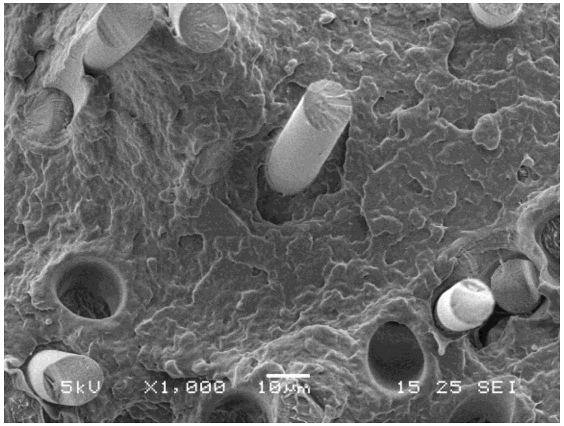


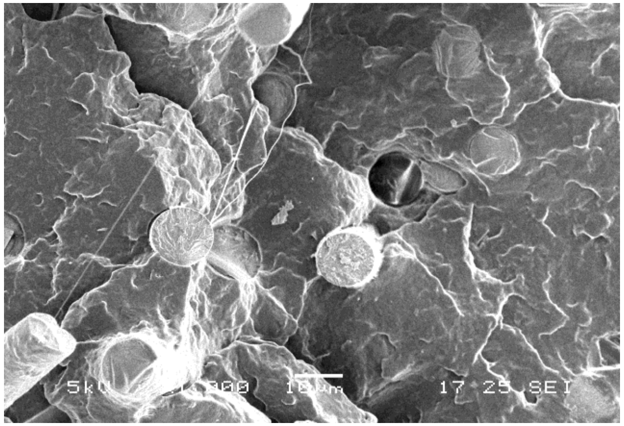
Benefits against MAgPP coupling:

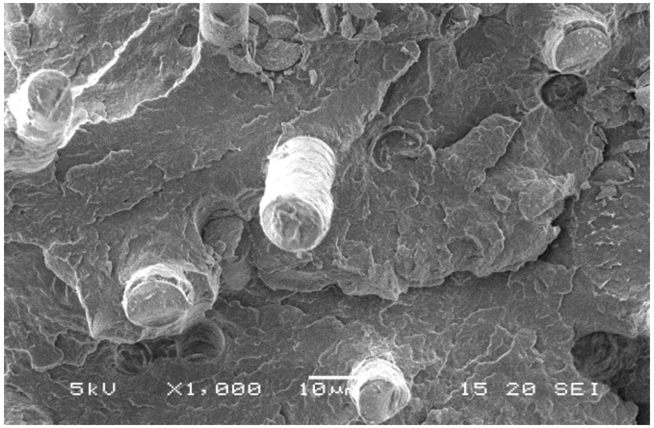
- High Temperature Tensile Modulus: +30% at 120°C
- HDT *(internal method on Metravib®DMA50)*: +10 (BC) to +17°C (AC)

Glass-Fibers-PP

Plastic and Composites







MAgPP(0.1% MA)	ATM in-situ (1.5%)	PA-66

Intimate coupling with GF and crosslinking to PP resin shows enhanced composites properties … approaching GF-PA66.

Conclusions & Perspectives

Plastic and Composites

- Like Sioplas® process discovered forty years ago on polyethylene resins, melt extrusion reaction of α,β-unsaturated carboxylic functional-silanes in presence of free radical initiator enables modification and crosslinking of PP resins and composites;
- Broad perspectives for engineering up PP composites were demonstrated, such as, e.g.,
  - underwater aging resistance in WF-PP
  - heat resistance in GF-PP;
- Dow Corning SISC is leading R&D projects
  - helping partners in Automotive, Appliances and Construction business
  - exploiting the tremendous opportunities offered by “Si” chemistry.

Thank You !...

Discover more at www.dowcorning.com/plasticandcomposite

Surface & Interface Solutions Center - SISC