IFPA

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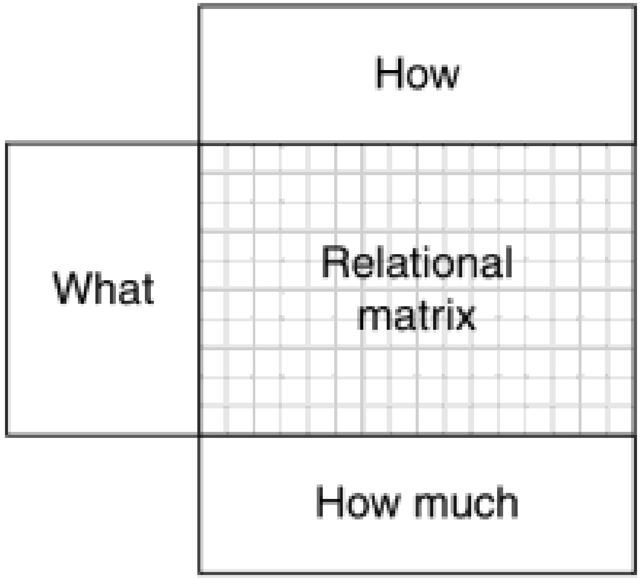
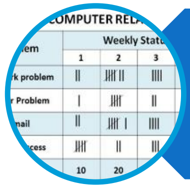
How do you improve the impact of your organization

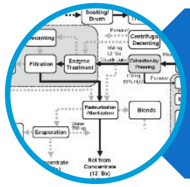


Sampling

Surveying



Check Sheets



Problem Concentration Diagrams

What can be added to your business

Filler Masterbatches

Impact modifier

Processing Lubrication

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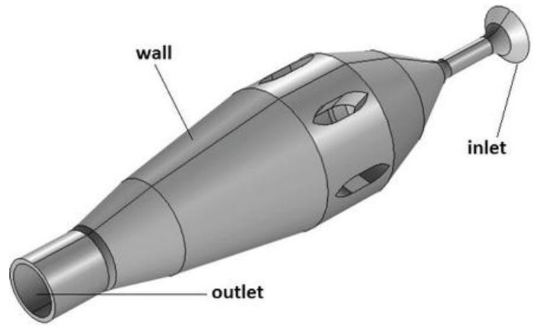
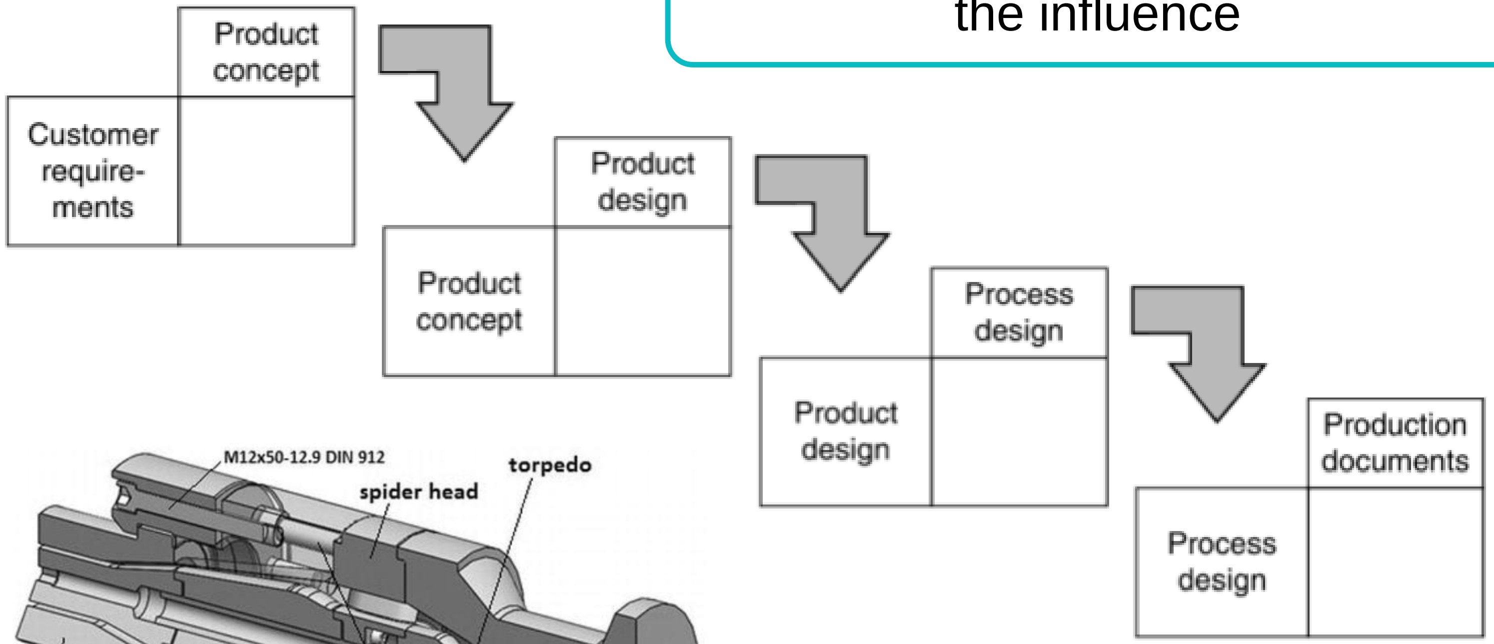
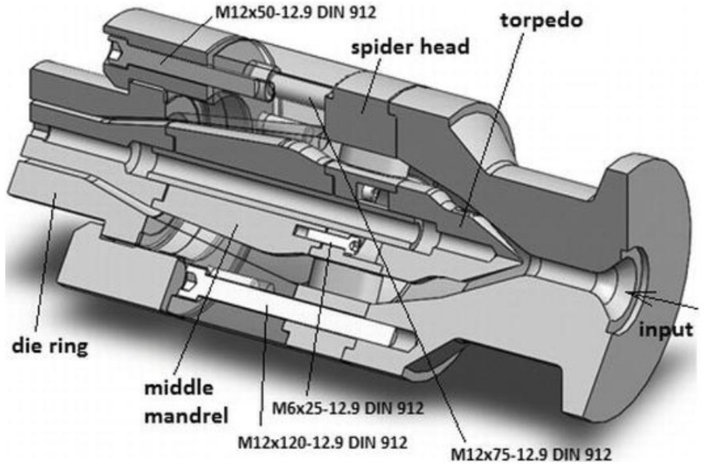


What gets influenced

Where is the influence impacting the most

How can I control and understand the influence





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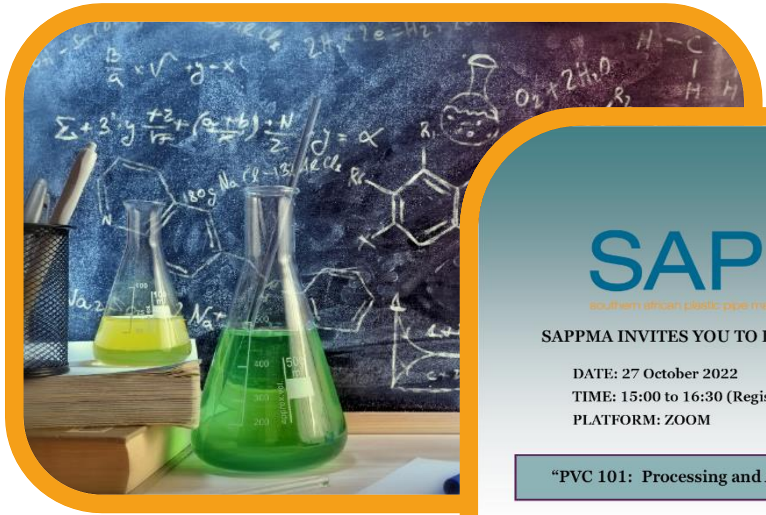
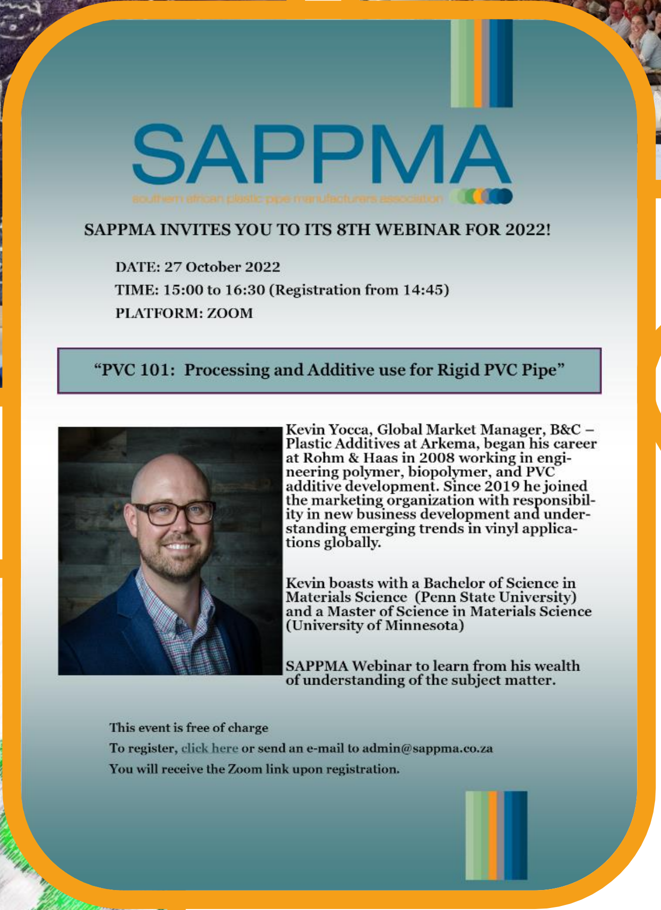
SAPPMA Webinar IX 2022

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SAPPMA INVITES YOU TO ITS 8TH WEBINAR FOR 2022!

DATE: 27 October 2022
TIME: 15:00 to 16:30 (Registration from 14:45)
PLATFORM: ZOOM

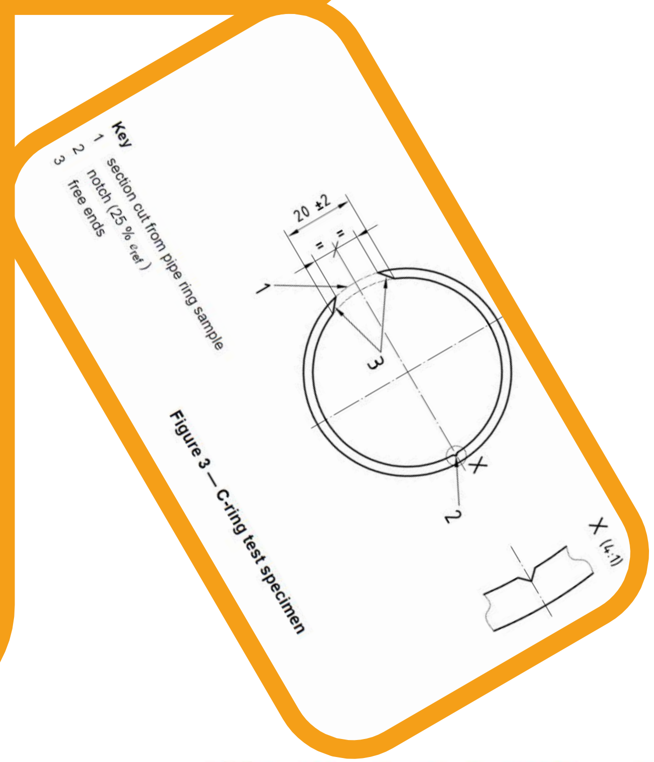
"PVC 101: Processing and Additive use for Rigid PVC Pipe"

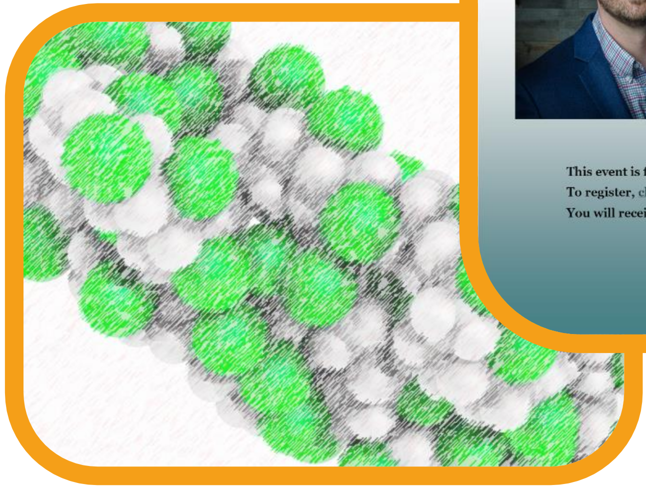
Kevin Yocca, Global Market Manager, B&C – Plastic Additives at Arkema, began his career at Rohm & Haas in 2008 working in engineering polymer, biopolymer, and PVC additive development. Since 2019 he joined the marketing organization with responsibility in new business development and understanding emerging trends in vinyl applications globally.

Kevin boasts with a Bachelor of Science in Materials Science (Penn State University) and a Master of Science in Materials Science (University of Minnesota)

SAPPMA Webinar to learn from his wealth of understanding of the subject matter.

This event is free of charge
To register, click here or send an e-mail to admin@sappma.co.za
You will receive the Zoom link upon registration.





Presenter

SAPPMA Webinar IX

27 October 2022

ARKEMA

Kevin Yocca

ARKEMA

PVC 101: Processing and Additive use for Rigid PVC Pipe

Kevin Yocca

SAPPMA
southern african plastic pipe manufacturers association

SAPPMA Webinar Series – October 27, 2022

Content overview

→ Introduction to PVC Gelation and Processing

- What is PVC gelation – why does it matter?
- The (famous) torque rheometer curve

→ PVC Formulations and Blending

- Rigid PVC formulations
- PVC lubrication theory
- Blending order and why it matters

→ Acrylic impact modifiers and rigid PVC formulations

- Core-shell acrylic impact modifiers
- Mechanical performance vs. Processing

→ Acrylic process aids and rigid PVC formulations

- Mineral content incorporation
- Cellular PVC and melt strength

Arkema Plastic Additives

→ Clearstrength® MBS impact modifiers

- Core-shell modifiers based on METHYL METACRYLATE / BUTADIENE / STYRENE
- Excellent cold temperature impact strength and transparency
- *Applications:* PVC film & sheet, CPVC pipes and fittings, engineering polymers and thermosets

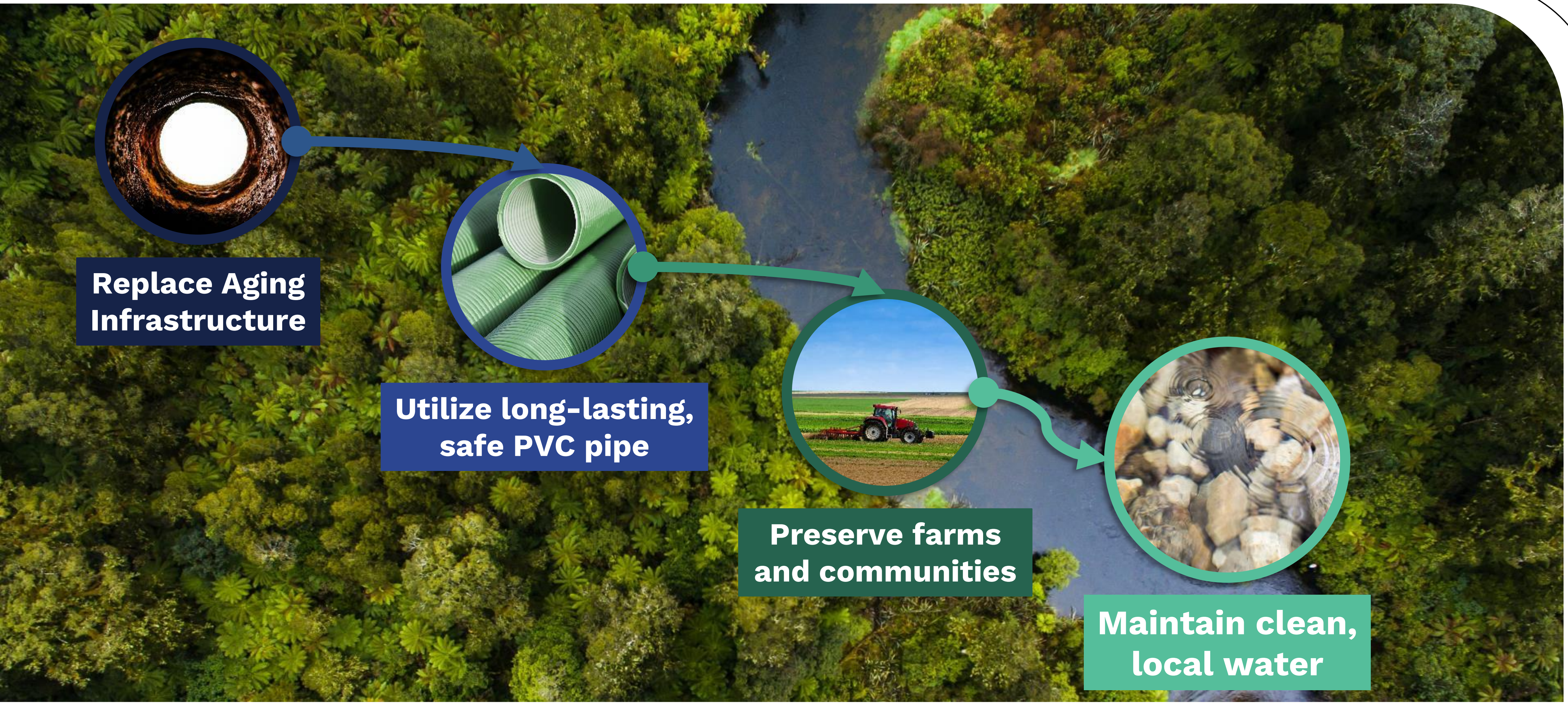
→ Durastrength® acrylic impact modifiers

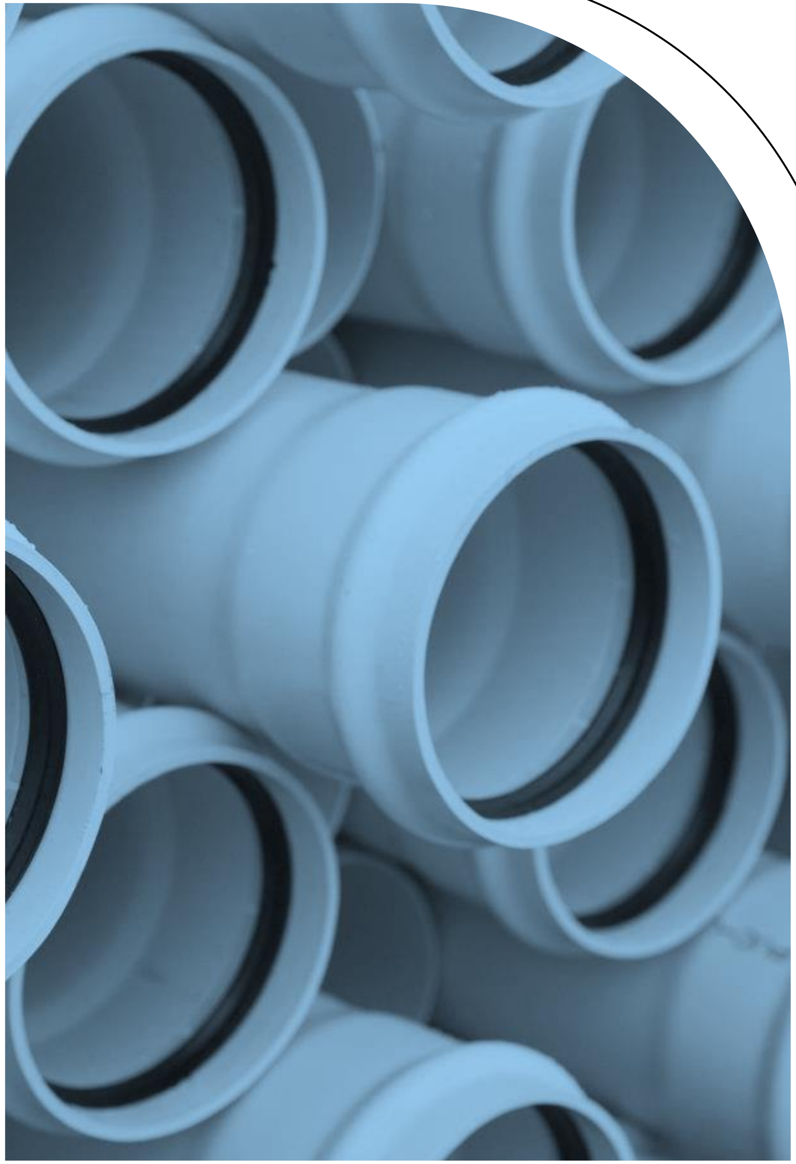
- Core-shell modifiers based on acrylic chemistry
- Optimized balance of impact performance and weatherability
- *Applications:* PVC window profiles, pipe and fittings, vinyl fence and siding, roofing membranes

→ Plastistrength® acrylic process aids

- High molecular weight acrylic co-polymers
- Enhances processing, PVC fusion promotion, and rheology / melt strength
- *Applications:* vinyl resilient flooring, PVC foam, film & sheet, pipe and profiles, vinyl fence and siding, bio-based polymers, and engineering polymers

Rigid PVC Pipe: modern infrastructure improving local communities





Introduction to PVC Gelation and Processing

PVC resins for rigid (and flexible) applications

→ 3 main PVC resin product processes:

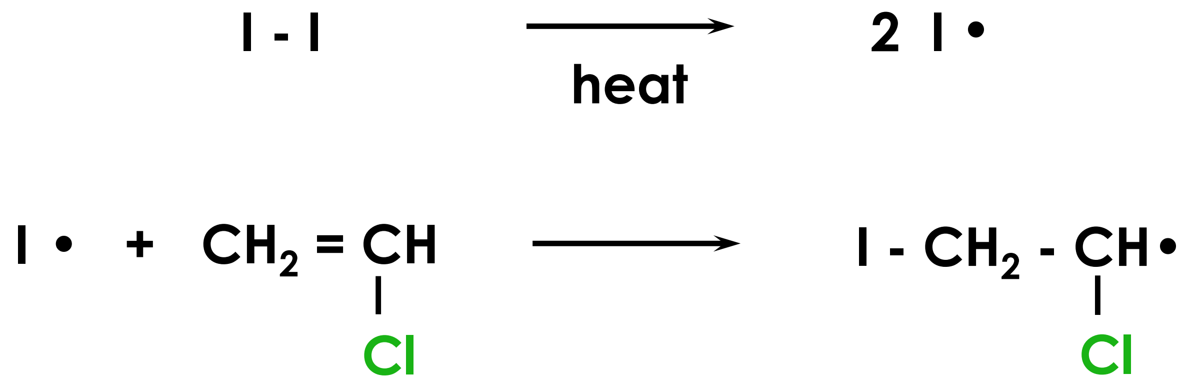
- Suspension: majority
- Mass: PVC synthesized in its own monomer
- Emulsion: plastisols, dispersion resin, some flexibles

→ Suspension PVC process (K-value or IV (intrinsic viscosity) characterizes MW):

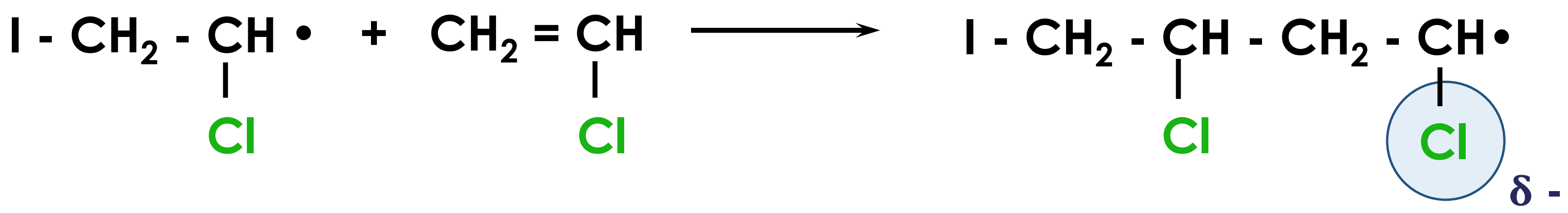
- VCM polymerized in water where PVC grains are maintained in suspension with dispersion agents (PVC, surfactants, emulsifiers, etc.)
- Initiator in monomer phase
- PVA and suspending additives remain in the final product
- Skin around PVC grains
- Remaining residual additives have some influence on the applicative performance:
  - Heat stability (+)
  - Color (+)
  - Haze (-)
  - Water blush (-)

Free radical mechanism: and why PVC is a dark art

Initiation



Propagation

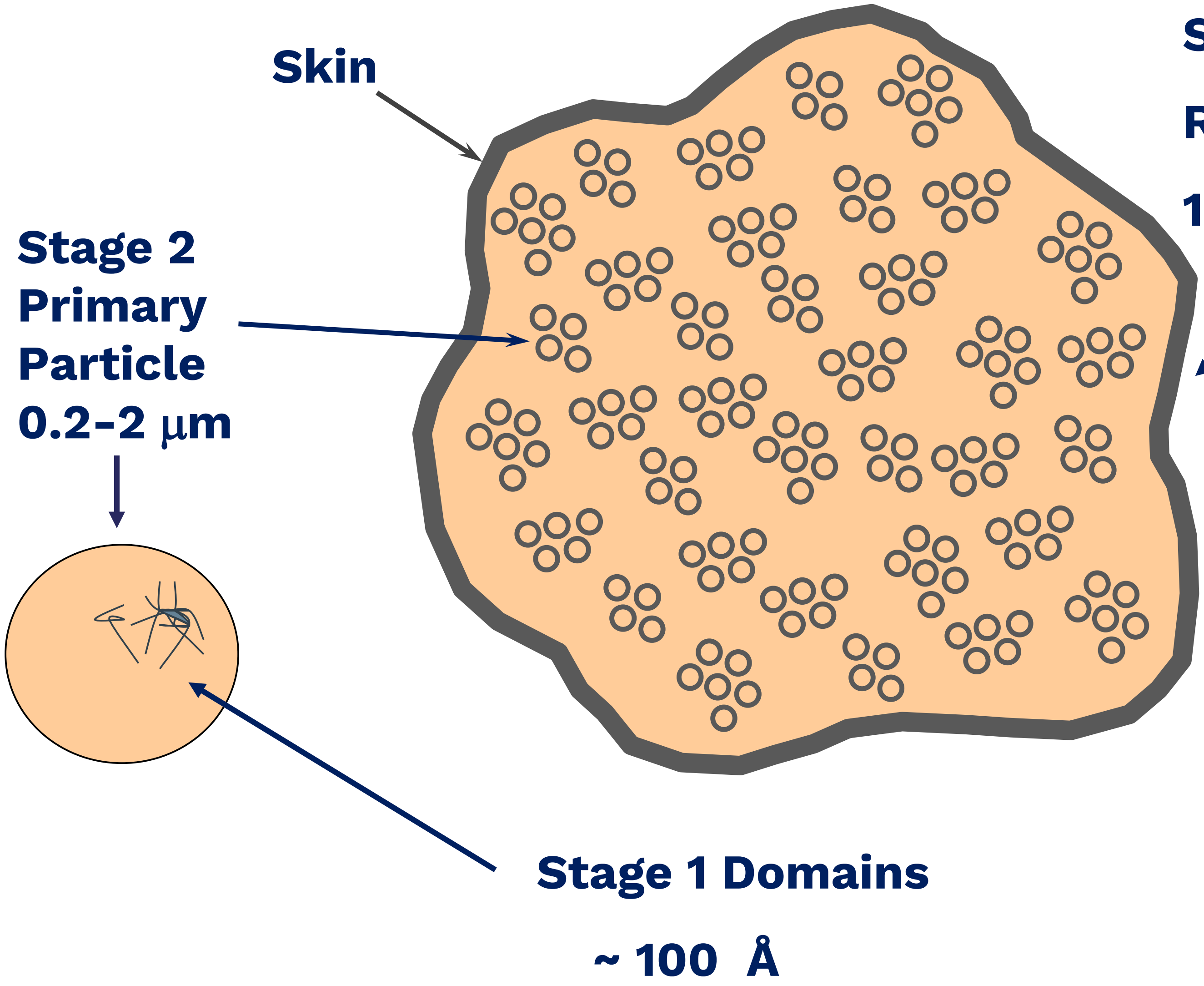


Why a PVC Particle?

PVC is the only polymer that is processed into end products in the same physical form that it was produced in the reactors. All other major polymers are compounded and sold as pellets. The formation and morphology of the PVC particles in the reactor is the key to processing and its many uses.

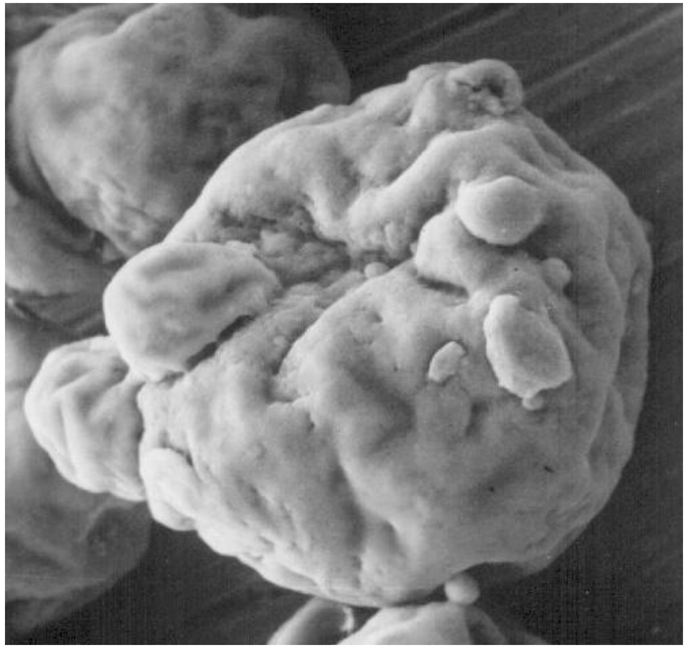
- Bob Paradis (Formosa)

PVC particle morphology



→ Zooming out to look at a PVC particle:

- Wrinkled surface (popcorn structure) due to change in density from monomer to polymer (> 1 g/cc to 1.4 g/cc)
- Resin particle size 100 - 180μm



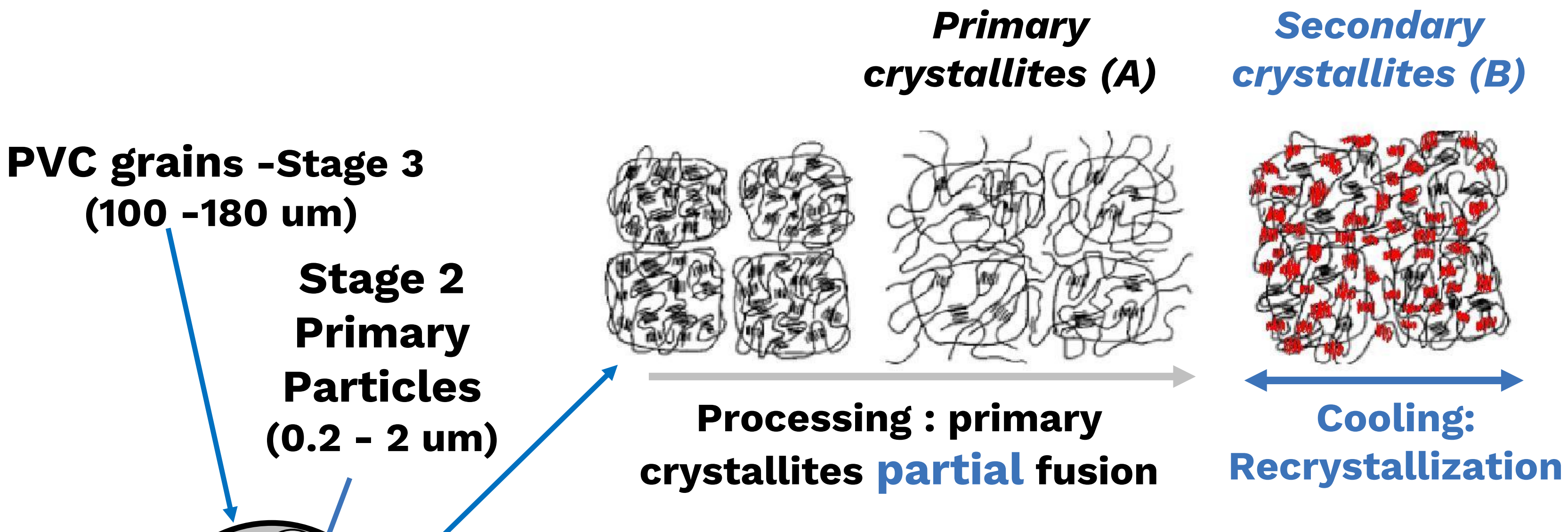
PVC Gelation – the Summers Model

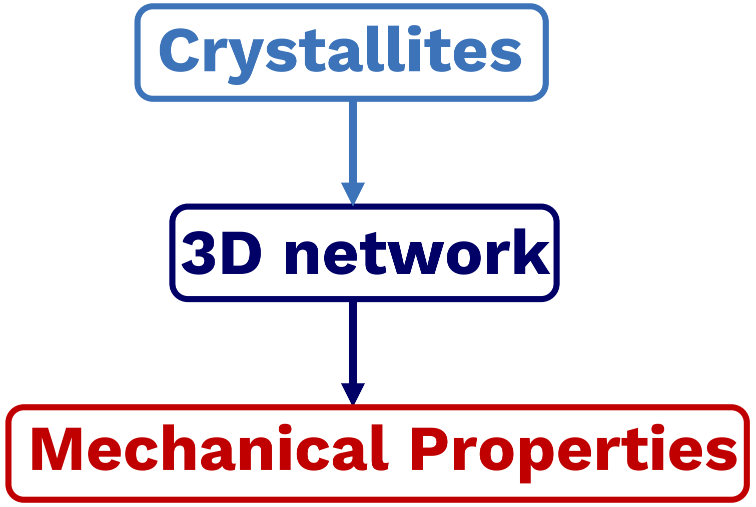
→ Gelation:

- The act of breaking down all the Stage 3 particles (grains)
- Melting the Stage 2 particles (primary particles) and forcing them together
- The degree that one melts the Stage 1 domains and reorders them is the key to the final physical properties of the PVC compound

→ Gelation of 65 – 80% is considered optimum for opaque formulations:

- Considerable molecular interdiffusion between the Stage 1 and 2 particles
- Interfaces in parts still exist resulting in better impact





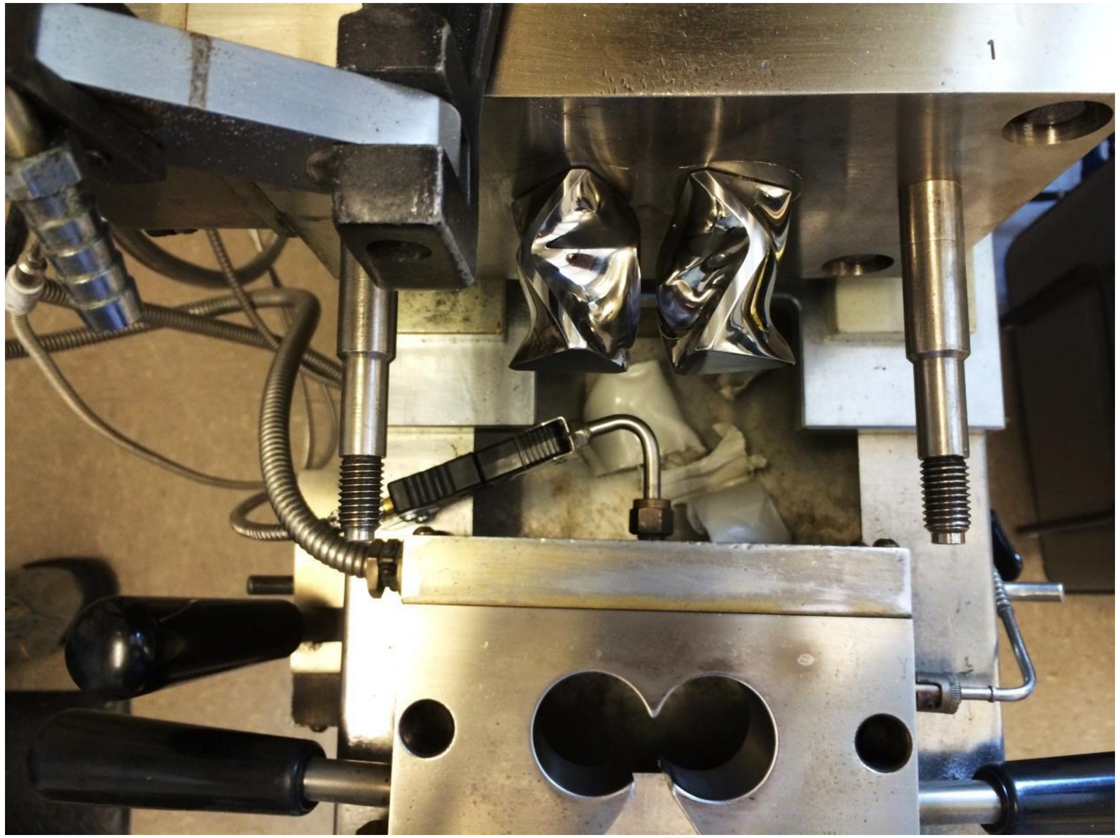
Summers, J.W., Rabinovitch, E.B., Booth, P.C. Measurement of PVC fusion (gelation). Journal of Vinyl Technology, March 1986, pp 2-6.

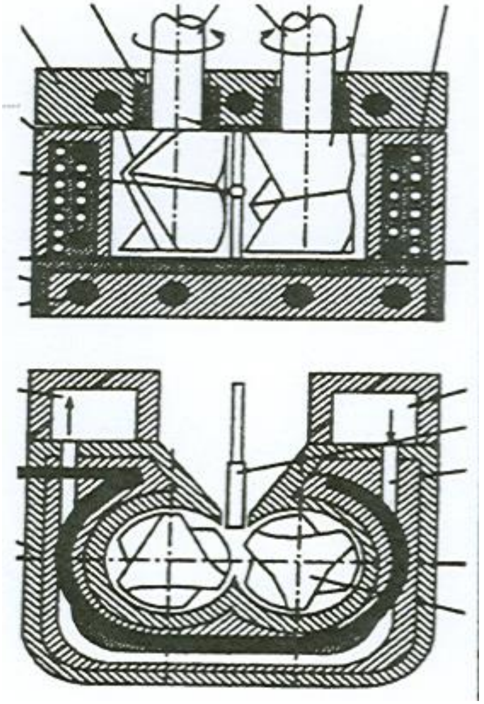
Torque Rheometer (Plasticorder®) Testing Instrument

→ Torque rheometry (ASTM D2538)

→ Primary analysis tool for PVC compound processing characteristics

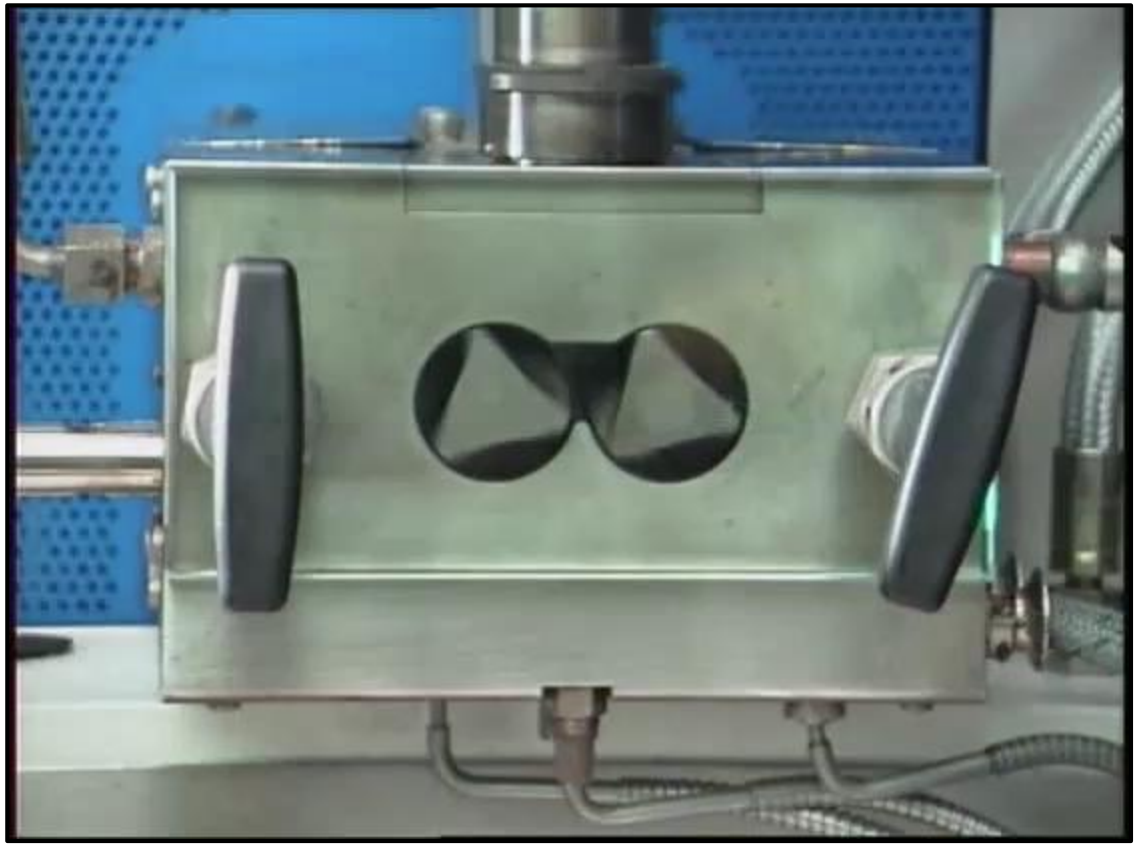
- Fusion time
  - Appearance at vent
  - Temperature specific
  - Approximates powder to melt transition
- Fusion Torque
  - Related to extrusion amperage
- Equilibrium torque
  - Continuous melt
  - Approximates melt viscosity in die
- Stability time
  - Time to degradation
- Color chip analysis
  - Important for stabilization

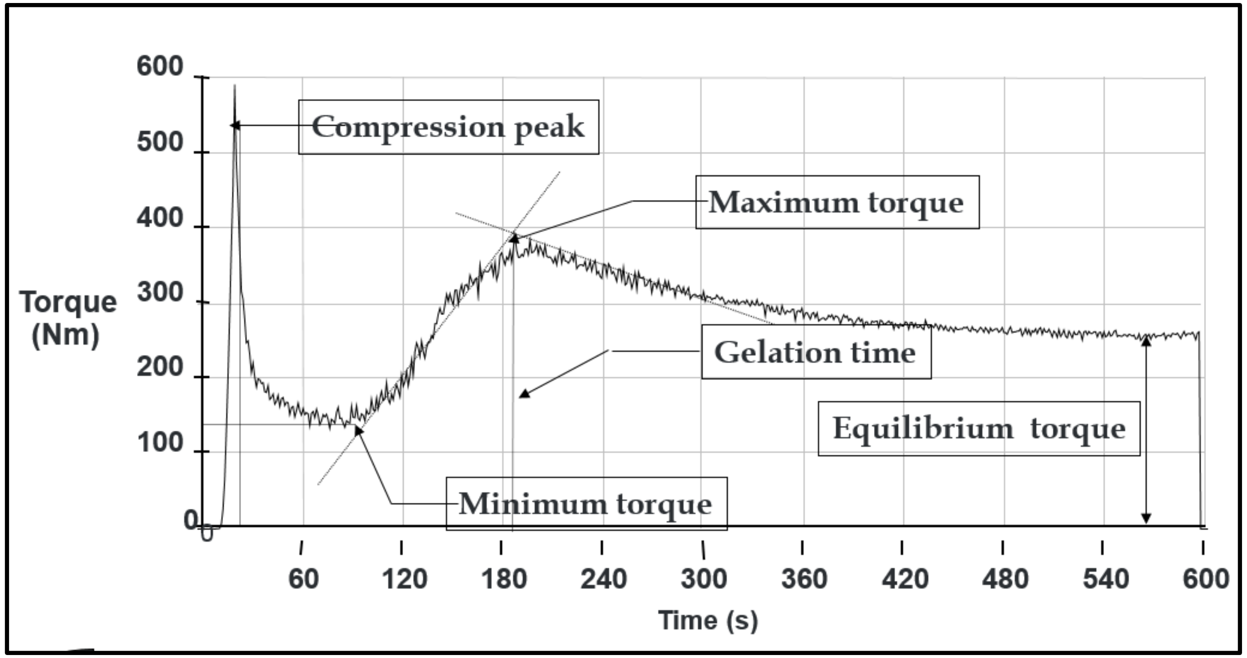




"The Brabender Bowl"

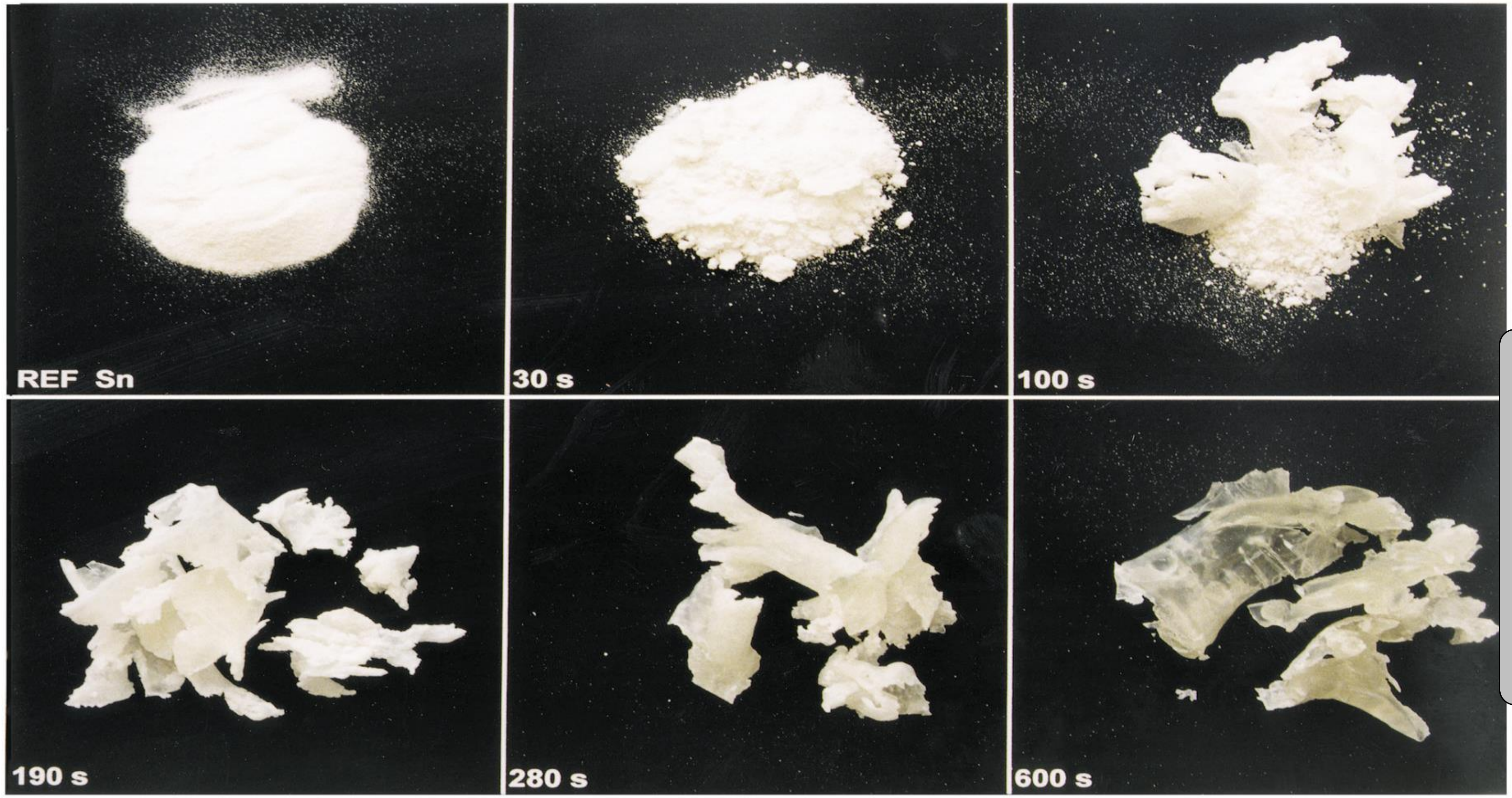
Torque rheometer – A.K.A. the “Brabender curve”



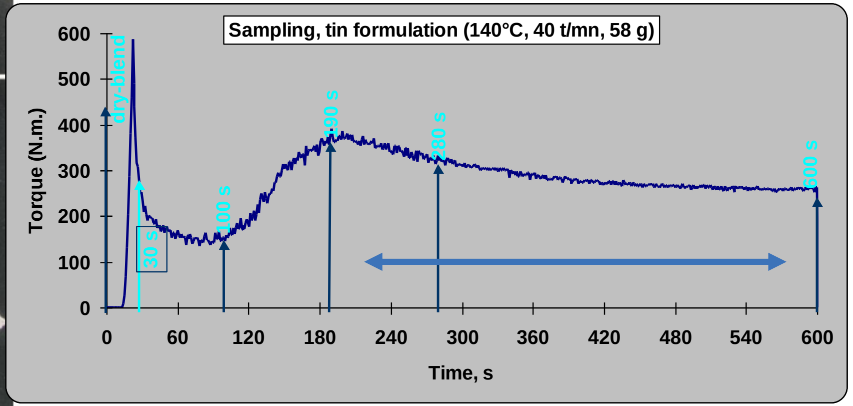


Torque Rheometer Test with Haake Internal Mixer (https://www.youtube.com/watch?v=zlCP3D3tnD8)

What is happening during the processing macroscopically?



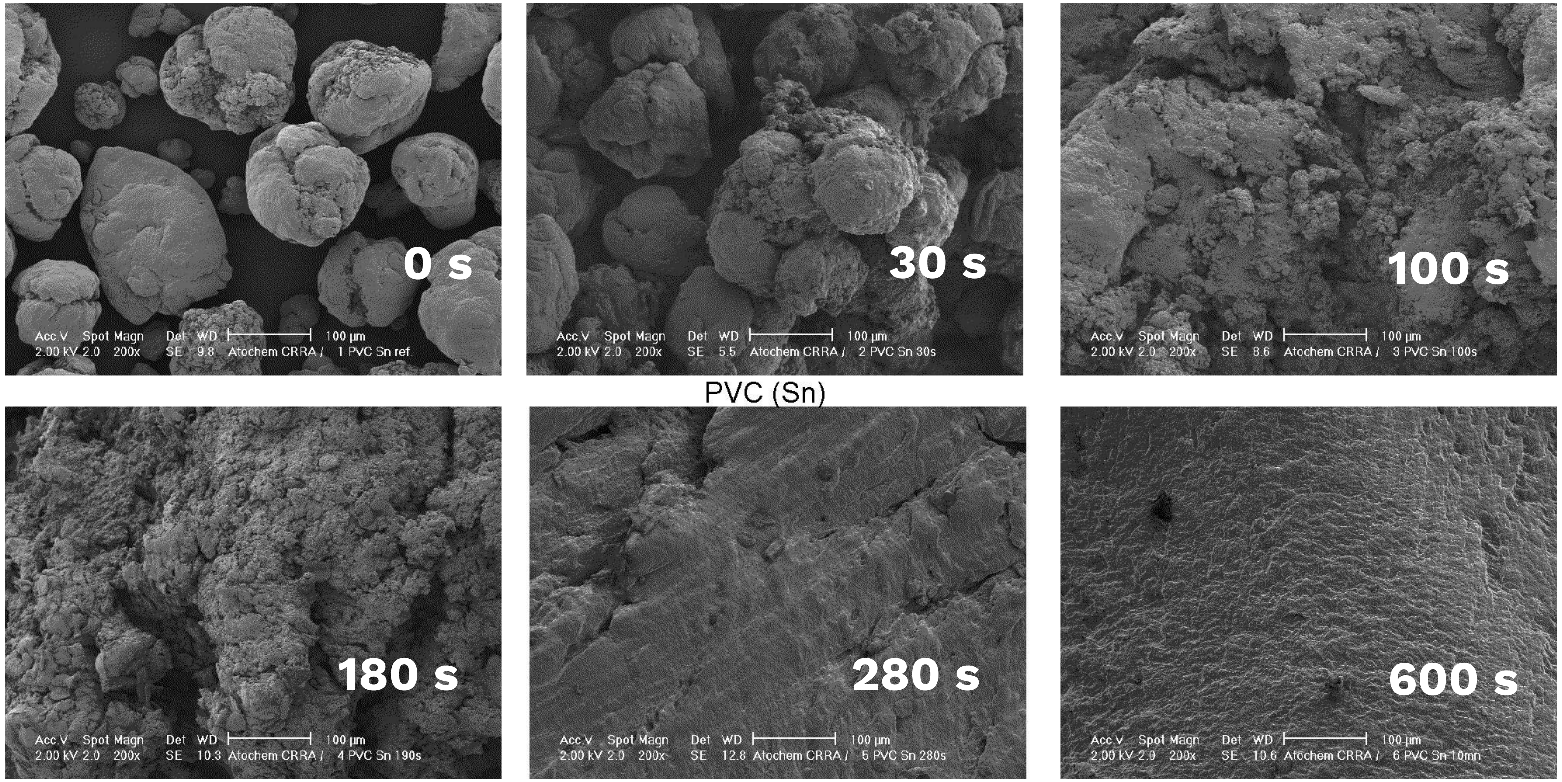
Note how appearance changes from powder, to a mixture of powder and melt to a fused mass



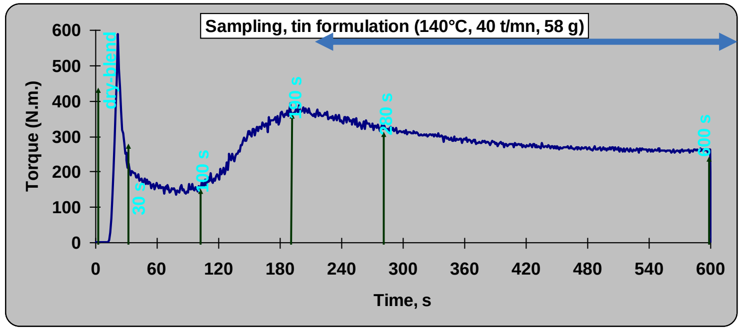
What is happening during the processing microscopically?

→ Gelation via torque rheometer:

- Breakdown of PVC grains to a continuous melt easier to imagine as it relates to a Brabender test
- Skin of PVC powder grain opens up releasing primary particles (PVC flow units)
- Eventually a continuous melt is developed with the desired rheology based on formulation

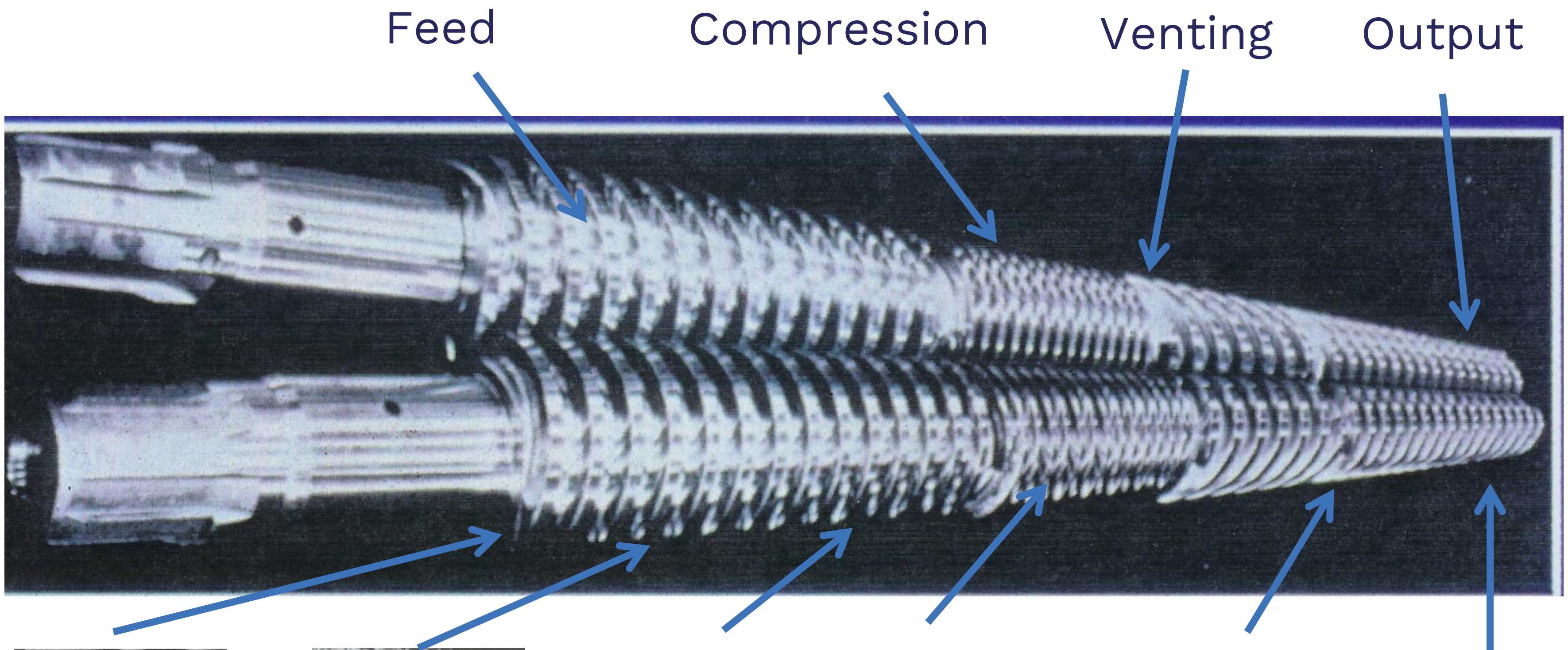


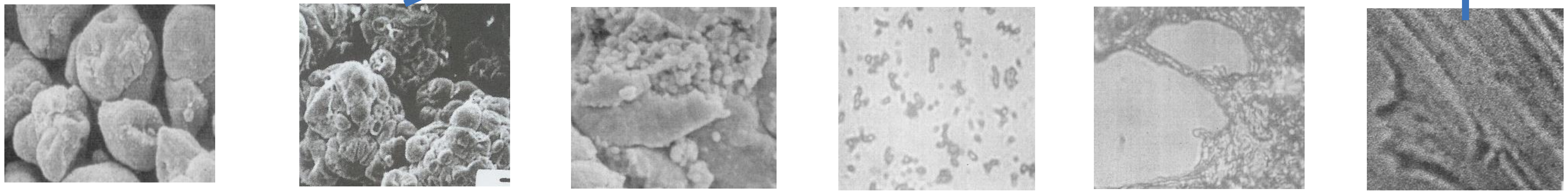
Bar = 100 µm



Creation of a macromolecular network is key. Low levels of gelation will lead to poor mechanical properties.

Gelation – correlations to the extruder*





Grains	Compaction 80°C - 120°C	Skin tear 140°C	Primary Particles 160°C	Gelation 185°C	Continuous Melt 190°C – 200°C

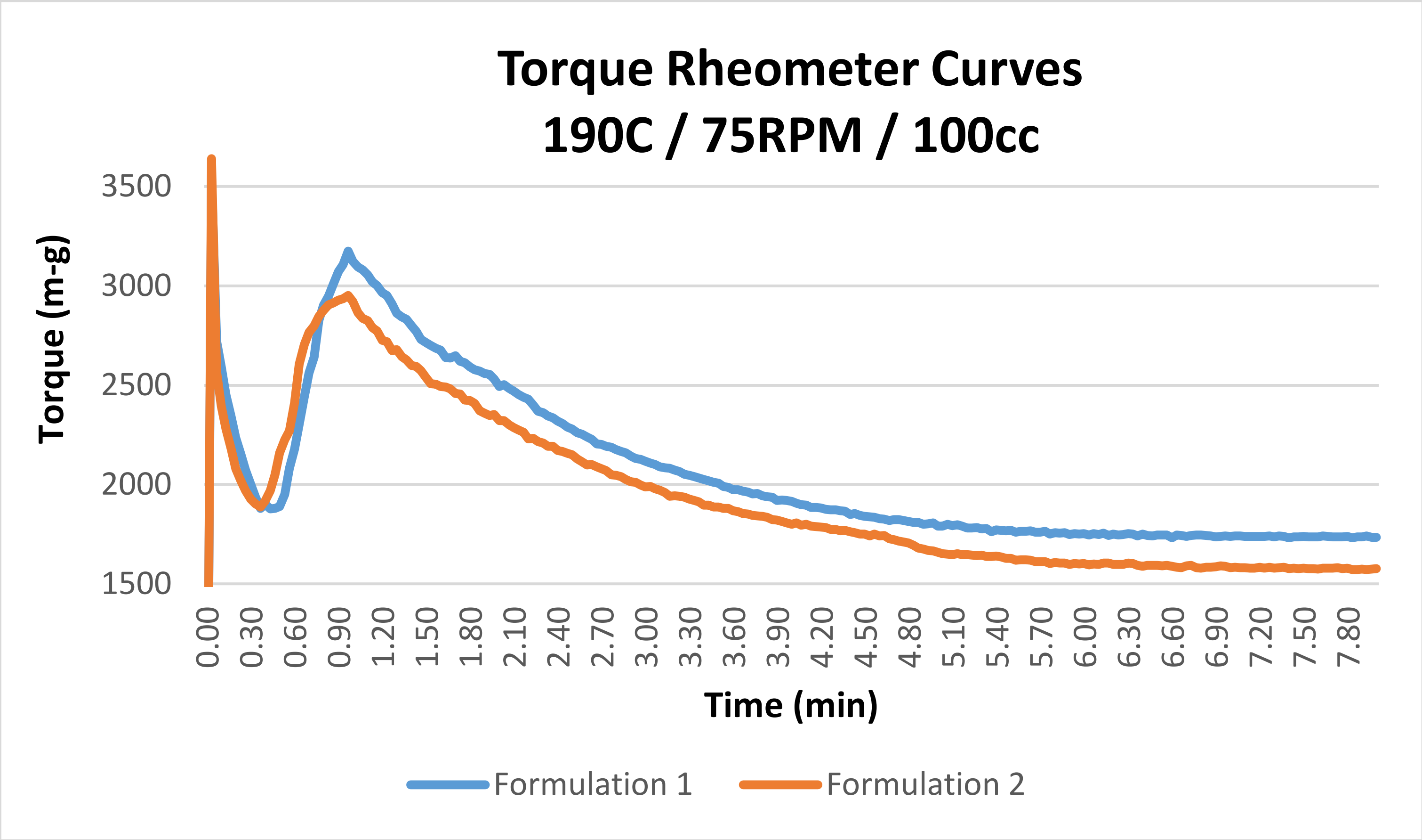
Formulate for the equipment used for processing

Choose components necessary for property and asthetic requirements

Think output rates and tooling for the final PVC article

*used with permission of Ella Rabinovitch, P3 Consultants

Performance can be predictive of gelation



Formulation 1 was control, a new formulation (Formulation 2) was tried and adjusted to give similar fusion time but the change resulted in lower viscosities

Impact Resistance of Formulation 1
68 in-lbs (Mean Failure Energy)

Impact Resistance of Formulation 2
58 in-lbs (Mean Failure Energy)

Specification is 60 in-lbs

Lower torques result in less work put into the melt, and could lower gelation levels. **% GELATION CAN BE APPROXIMATED BY WORK** put into the compound by integrating the area under the rheometer curves. Rheometer software can do this.

PVC Formulations and Blending

PVC Formulation – rigid PVC basics

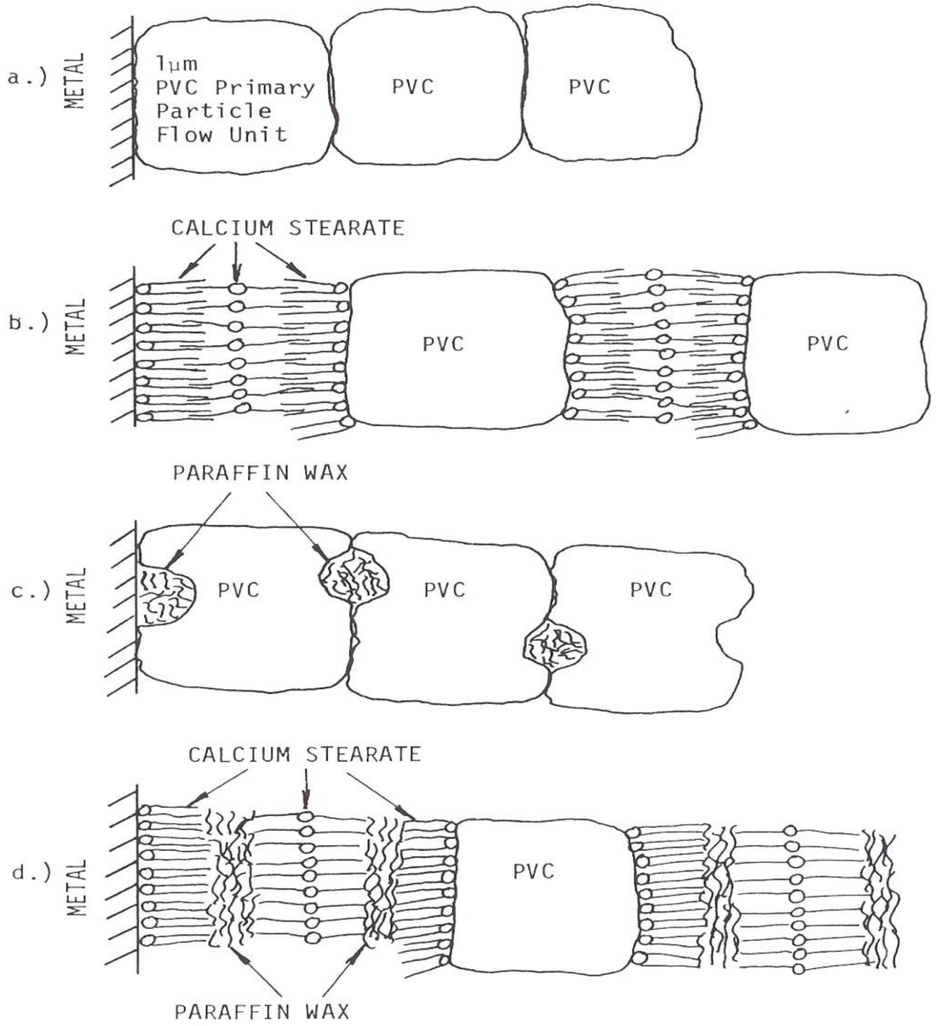
Function	Ingredient	Role Within Formulation
Base Resin	PVC Resin	Basic properties
Heat Stability	Organotin Heat Stabilizer	Thermal stability, PVC resin can be easily degraded by exposure to high temperatures
Internal Lubricant	*Calcium Stearate*	*Helps to process PVC by promoting PVC particle breakdown*
External Lubricant	*Paraffin Wax*	*Helps regulate extrusion process*
External Lubricant	*PE / OPE Wax*	*Helps regulate extrusion process / metal release*
Fusion Promotion Melt Strength	Process Aid (PLASTISTRENGTH®)	Helps molten PVC compound maintain integrity during processing / promote fusion as needed
Impact Resistance	Impact Modifier (DURASTRENGTH®)	Provides PVC articles with improved toughness
Filler	Calcium Carbonate (0.7 µm)	Used for cost reduction in PVC processes. May help, or hurt certain physical properties
Pigment / UV Protection	Titanium Dioxide	Provides protection from UV light
Color	Pigments / Colorants	As Needed

Lubrication Theory - PVC

Combination of Calcium Stearate (CaSt) and Paraffin Wax are the most common throughout the industry

These are among the fundamental building used to control gelation in rigid PVC formulations.

The Summers model for lubrication explains how these work together during processing

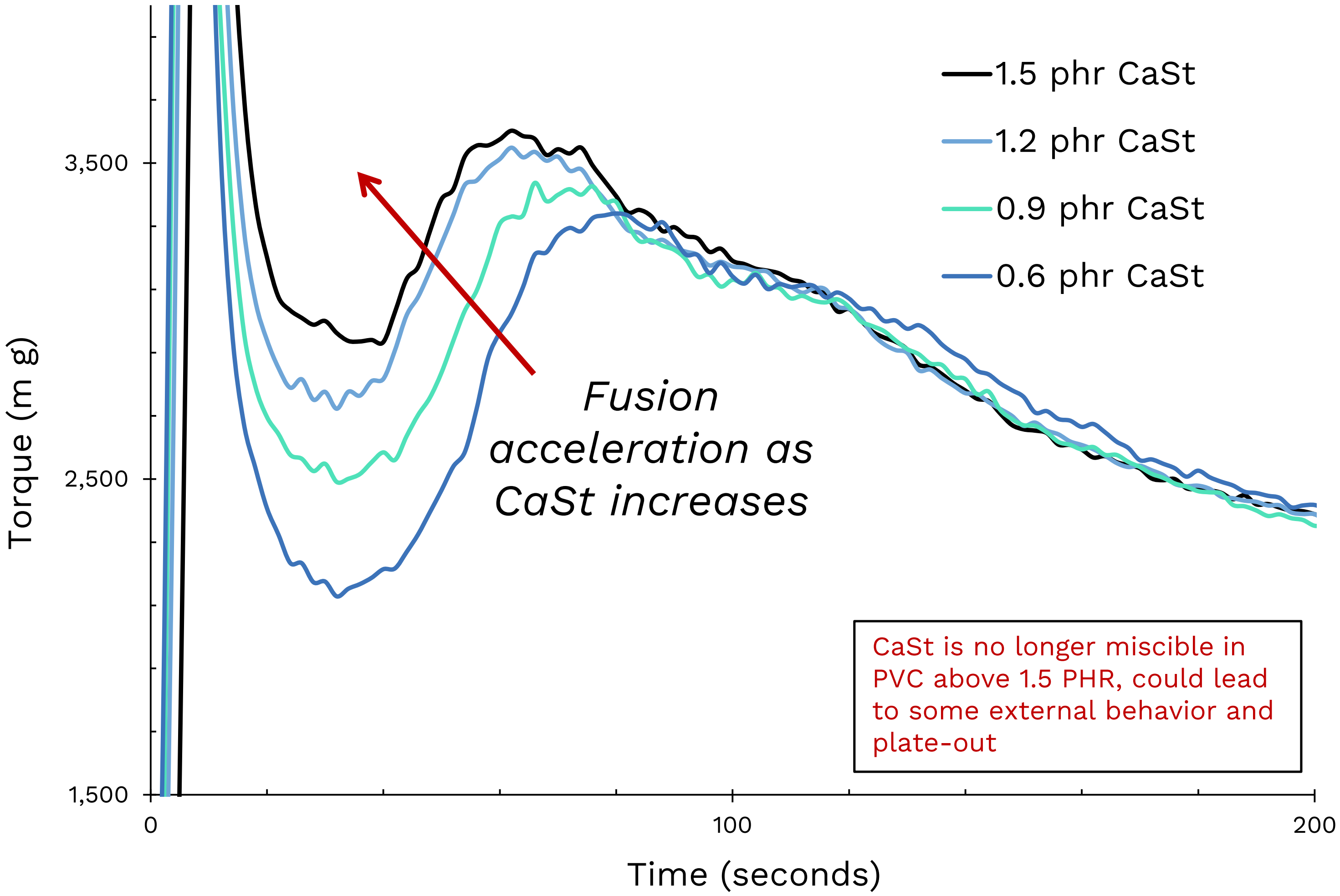


Summers, J.W., Proceedings SPE ANTEC 2006, p2882

Effect of calcium stearate level on rheology

Generic PVC Substrate Formulation

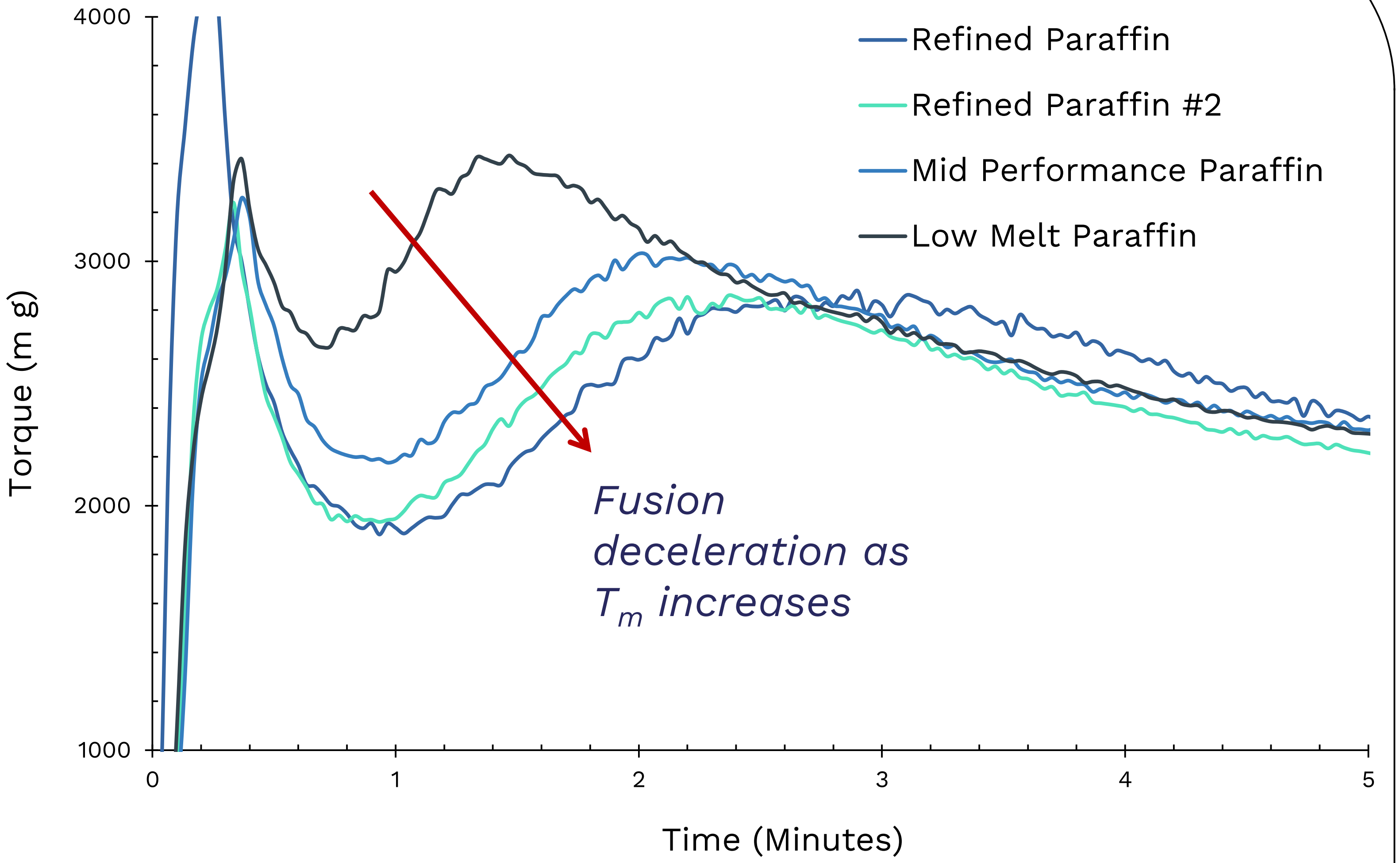
Component	phr
PVC Resin (K67)	100
Organotin Stab (8% RE Sn)	1.0
Calcium stearate	***Varies***
Paraffin wax	0.7
Durastrength® 200	2.5
Plastistrength® 530	0.5
$CaCO_3$ (0.7 µm, treated)	18
Titanium dioxide	1.0



Effect of paraffin wax melting point on rheology

Generic PVC Substrate Formulation

Component	phr
PVC Resin (K67)	100
Organotin Stab (8% RE Sn)	1.0
Calcium stearate	0.8
Paraffin wax (vary T_m)	***0.7***
Durastrength® 200	2.5
Plastistrength® 530	0.5
$CaCO_3$ (0.7 µm, treated)	18
Titanium dioxide	1.0



Blending Order – it matters!

→ Blend in order listed at defined temperatures, deviations could have consequences

→ Start with PVC resin and Stabilizers

- Allows for stabilizer diffusion into PVC particle

→ Add lubricants next to facilitate melting and ‘coating’ of PVC grains (60°C – 65°C)

- Never put lubricants in prior to organotin stabilizer

→ Add impact modifiers and process aids (< 75°C)

→ Add fillers (calcium carbonate) (< 90°C)

→ Add pigments (titanium dioxide) (> 95°C)

- Blend time is minimized in mixer to reduce wear

Mixers are dumped between 100°C and 120°C depending on geography (moisture removal). Note that extended times at these temperatures can initiate the degradation process.

Processing effects due to blend order changes

Ingredients	Too early? Impact on the compound? Effect at the extrusion?	Too late? Impact on the compound? Effect at the extrusion?
Stabilizer	n/a	Degradation of PVC resin may occur (>120°C) resulting in lower stability time. Poor dispersion
Solid Paraffin Wax	Could interfere with stabilizer migration into PVC particles	Could preferentially coat fillers and additives rather than resin. Poor dispersion.
Calcium Stearate	Could interfere with stabilizer migration into PVC particles. If CaSt added before stabilizer, it can lead to agglomeration of stearate leading to conveying issues.	Could preferentially coat fillers and additives rather than resin. Poor dispersion.
Paraffin / PE Wax	Could interfere with stabilizer migration into PVC particles	Could preferentially coat fillers and additives rather than resin. Poor dispersion.
Titanium dioxide	Premature equipment wear. Waxes could preferentially coat the TiO_2 lessening their effect on the PVC resin.	Poor dispersion.
Calcium carbonate / filler	Waxes could preferentially coat the $CaCO_3$ lessening their effect on the PVC resin / could lead to conveying issues to extrusion lines	Poor compound homogeneity at higher filler levels.
Pigment	Agglomeration of pigment particles	Poor mixing.
Process Aid	Could be preferentially coated by lubricants	Poor dispersion.
Impact Modifier	Could be preferentially coated by lubricants. Rubber agglomeration resulting in loss of impact properties and of conveying issues.	Poor dispersion.

Formulations: rigid PVC pipe – low shear, non-weatherable PVC

- → Accounts for largest PVC resin consumption (by far)
- → Formulating largely governed by regulatory entities (e.g. HSB TR-2 range formulations)
- → Pipe extrusion can be lower shear vs. window profile or other rigid PVC processing
- → Tensile and burst physical properties most critical (less impact needed)
- → *impact modifier used in foam core pipe (FCP skin)
- → **process aid used in FCP core for cellular PVC (> 0.90 g/cc density)

Component	PHR
PVC Resin (0.91 IV or K-65)	100.0
Sn Stabilizer (< 12% Sn reverse ester stab.)	0.3 – 1.0
Calcium stearate	0.4 – 1.5
Paraffin wax (165°F MP)	0.6 – 1.5
Oxidized PE (low MW common)	0 – 0.5
Durastrength® 200, 320, 350	***0 – 3.0***
*****Plastistrength® 379, 580***	***0 – 3.0***
*****Plastistrength® 770***	***0 – 2.0***
Calcium Carbonate (3.0 μm) – can vary	0 – 5.0
Titanium dioxide (chalk / non-chalk)	0.5 – 3.0
Color / pigments	0 – 1.0, as needed
total	***~ 108***

Formulations: rigid PVC conduit / corrugated pipe

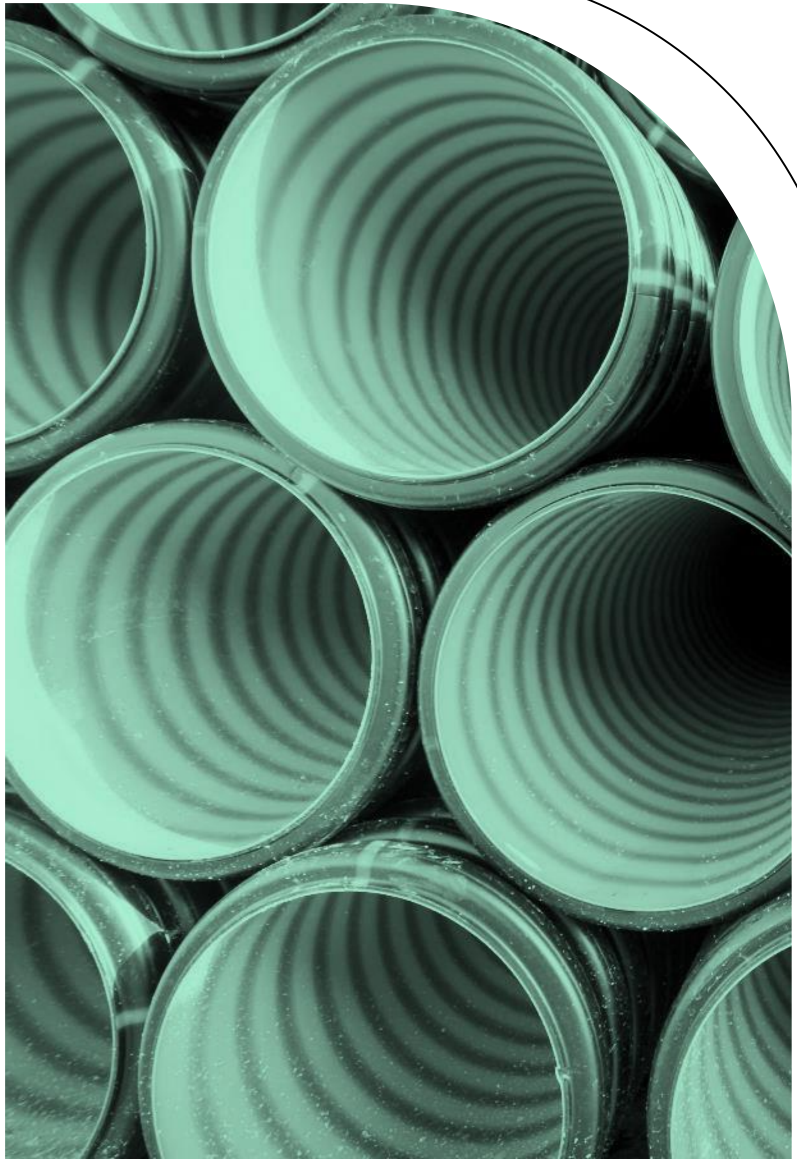
→ Conduit typically for electrical applications:

- Often governed by regulatory bodies (e.g. UL certification and others)
- Typically formulated with elevated levels of calcium carbonate (can be > 50 phr)
- Requires impact modifier for crush testing / mechanical integrity

→ Corrugated pipe can be single or dual wall:

- Formulations are not significantly different vs. conduit, however are often made on much larger diameters
- Impact performance critical for wall integrity
- Processing can be complicated – process aids can help with larger diameter fusion
- Calcium carbonate levels typically at least 20 phr

Component	PHR
PVC Resin (K-67)	100
Stabilizer (10% Sn)	1.2
Paraffin Wax (165°F)	1.3
Calcium Stearate	0.7
Oxidized PE Wax	0.2
Durastrength® 367 or Chlorinated PE	4
Plastistrength® 562	0 – 0.7
Calcium Carbonate (1µm)	20
Titanium dioxide	0.5
total	***~ 128***

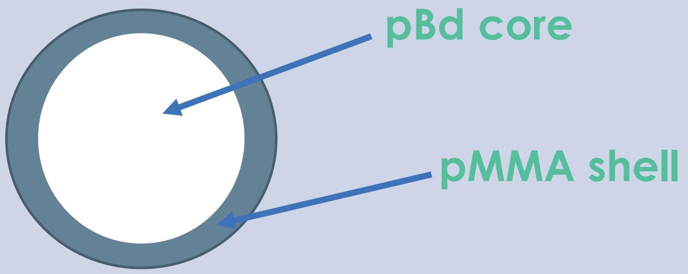


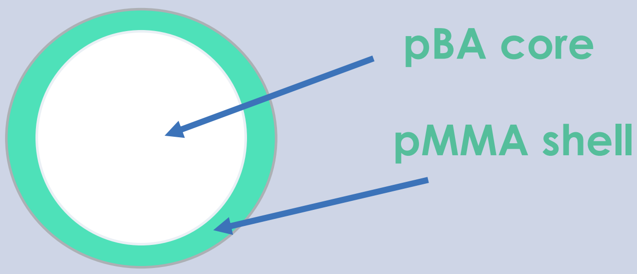
Acrylic impact modifiers and rigid PVC formulations

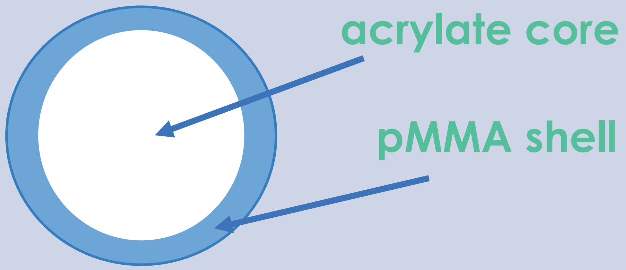
DURASTRENGTH®

Impact modifier core / shell technologies

Methacrylate – Butadiene – Styrene (MBS)	Acrylic Impact Modifiers (AIM)	
	Traditional	Specialty





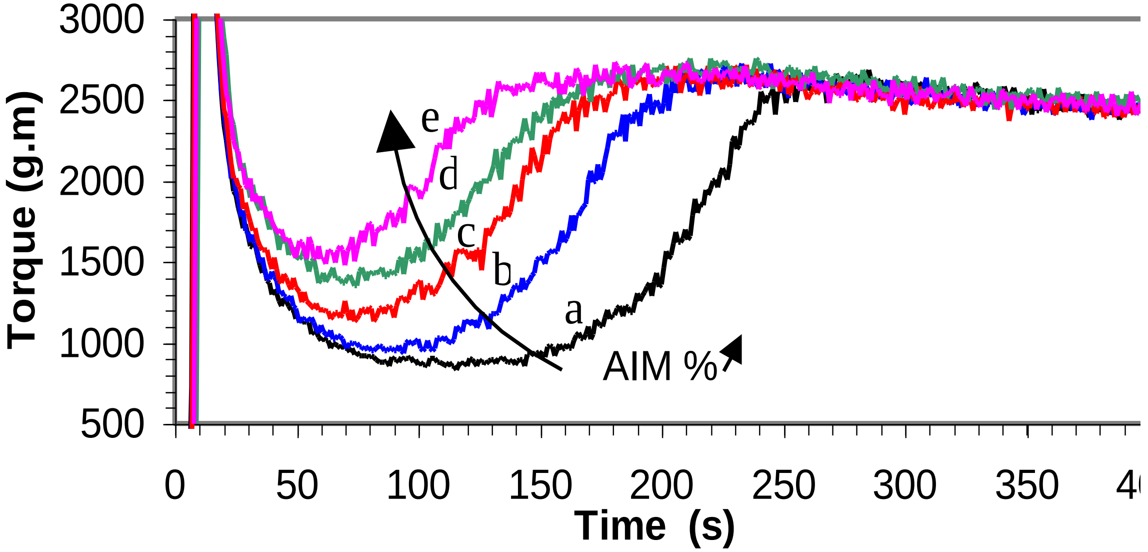


MBS	AIM – Traditional	AIM – Specialty
• Excellent low temperature impact • Acceptable thermal stability • **Refractive index similar to PVC** → **Good transparency** • **Non-weatherable**	• Very good low temperature impact • Excellent thermal stability • **Lower refractive index than PVC** → **Higher haze** • **Excellent weatherability**	• Good low temperature impact • Excellent thermal stability • **Refractive index similar to PVC** → **Good transparency** • **Good weatherability**
CLEARSTRENGTH®	DURASTRENGTH®	

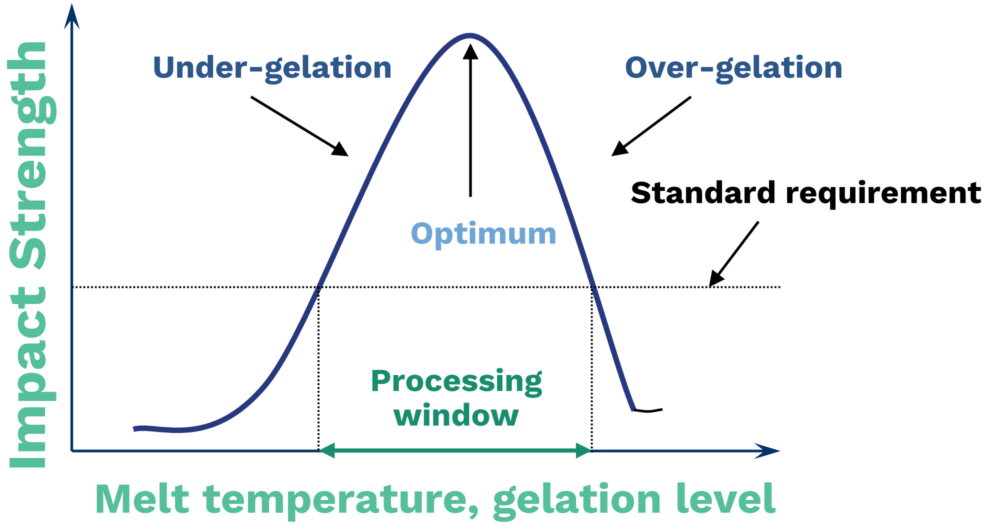
Impact modifier influence: processing and rheology

→ PVC mechanical properties and melt rheology are correlated:

- Transition from a brittle to ductile PVC material is highly dependent on impact modifier content
- Increasing modifier levels generally improves fusion and mechanical properties
- Cost-efficiency gains possible due to improved fusion at higher filler loading levels while maintaining mechanical properties

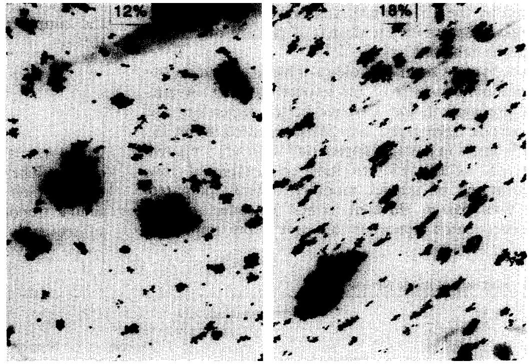


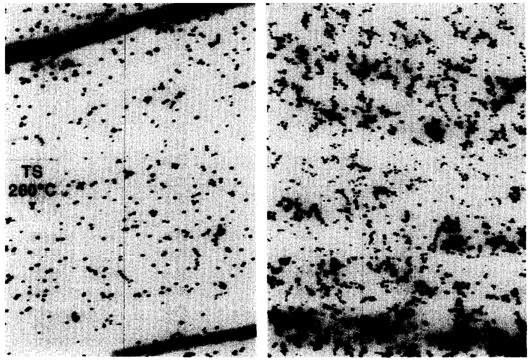
(a) = 4.5 phr; (b) = 5.5 phr; (c) = 6.5 phr;
(d) = 7.5 phr; (e) = 8.5 phr

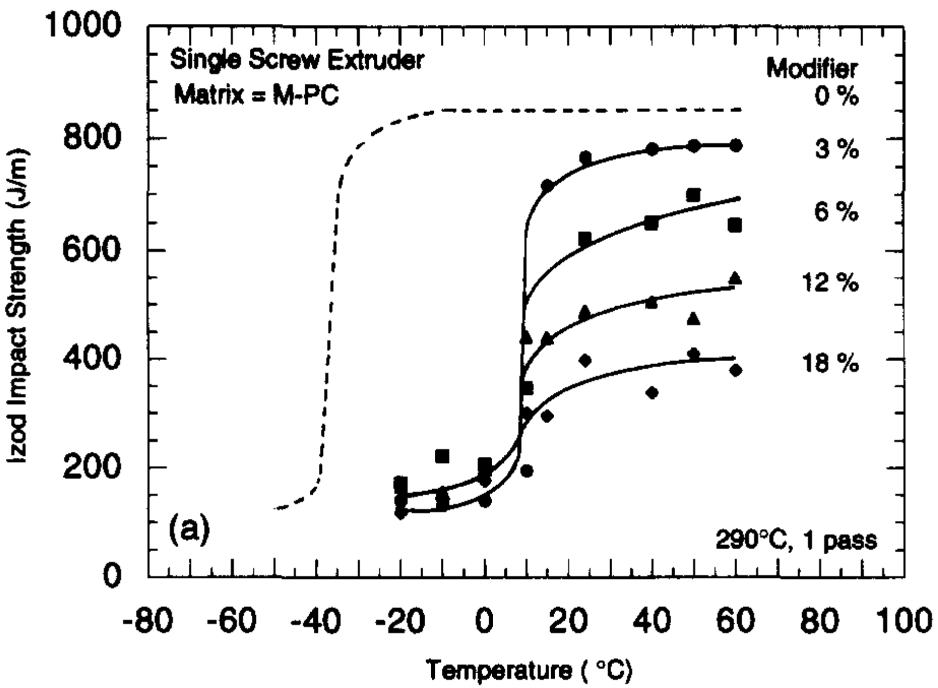


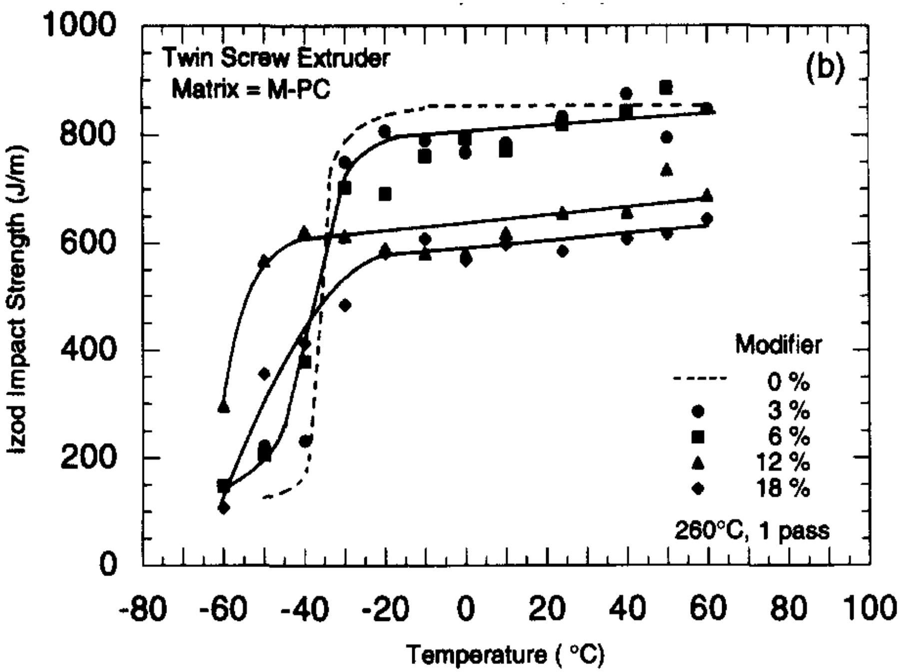
Processing: dispersion of core-shell particles

Y. Kayano et al., Polymer 37 (1996) 4505-4515.







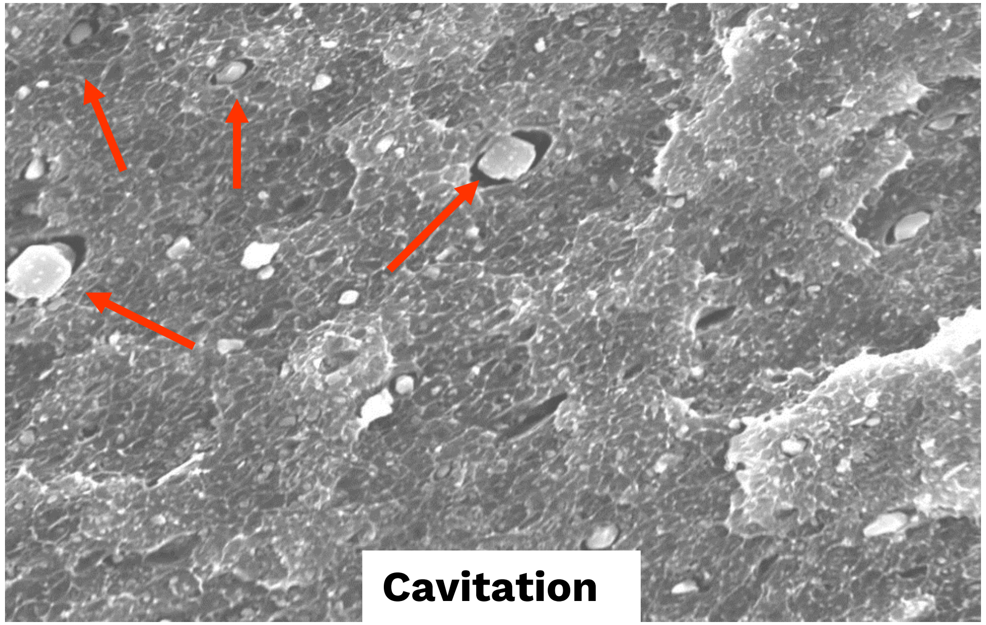


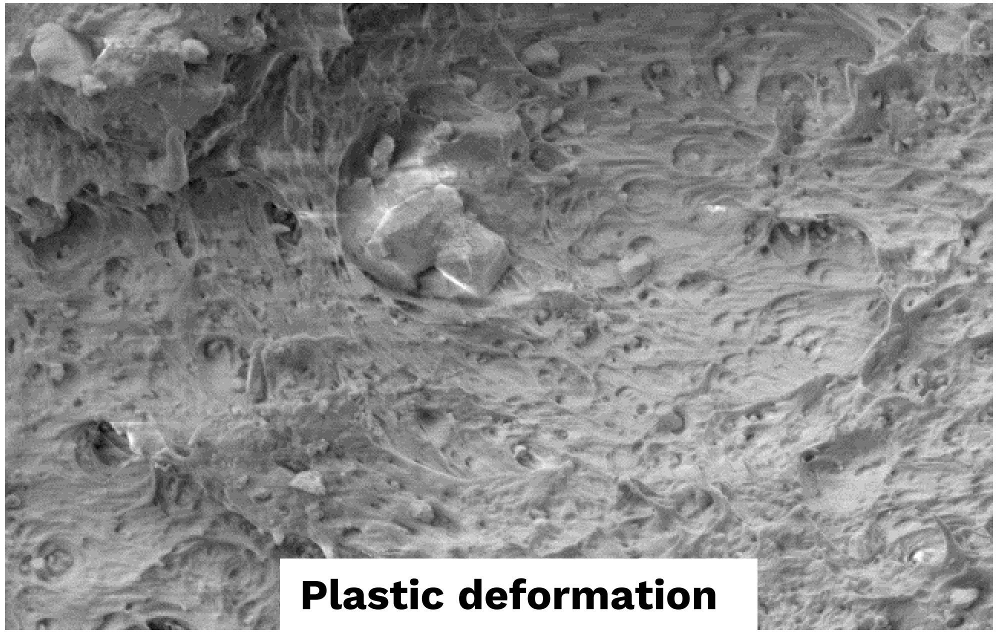
- → Processing plays a key role in achieving success with impact modifiers
- → An impact modifier is only as good as the process that disperses it

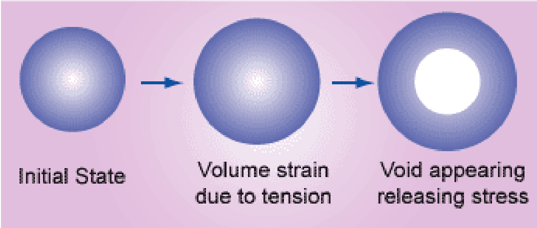
Impact modifier – inorganic filler synergies

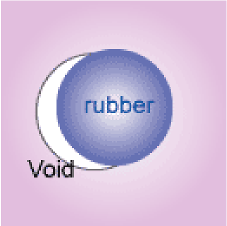
→ Example of mineral / AIM synergy via stress concentrators

SEM Morphology investigations after impact testing (ductile break)









- ***Dewetting of inorganic/PVC interfaces leads to cavitation promoting shear yielding***
- ***Larger energy dissipation***
- ***Impact modifiers AND filler particles can participate to aid the matrix***

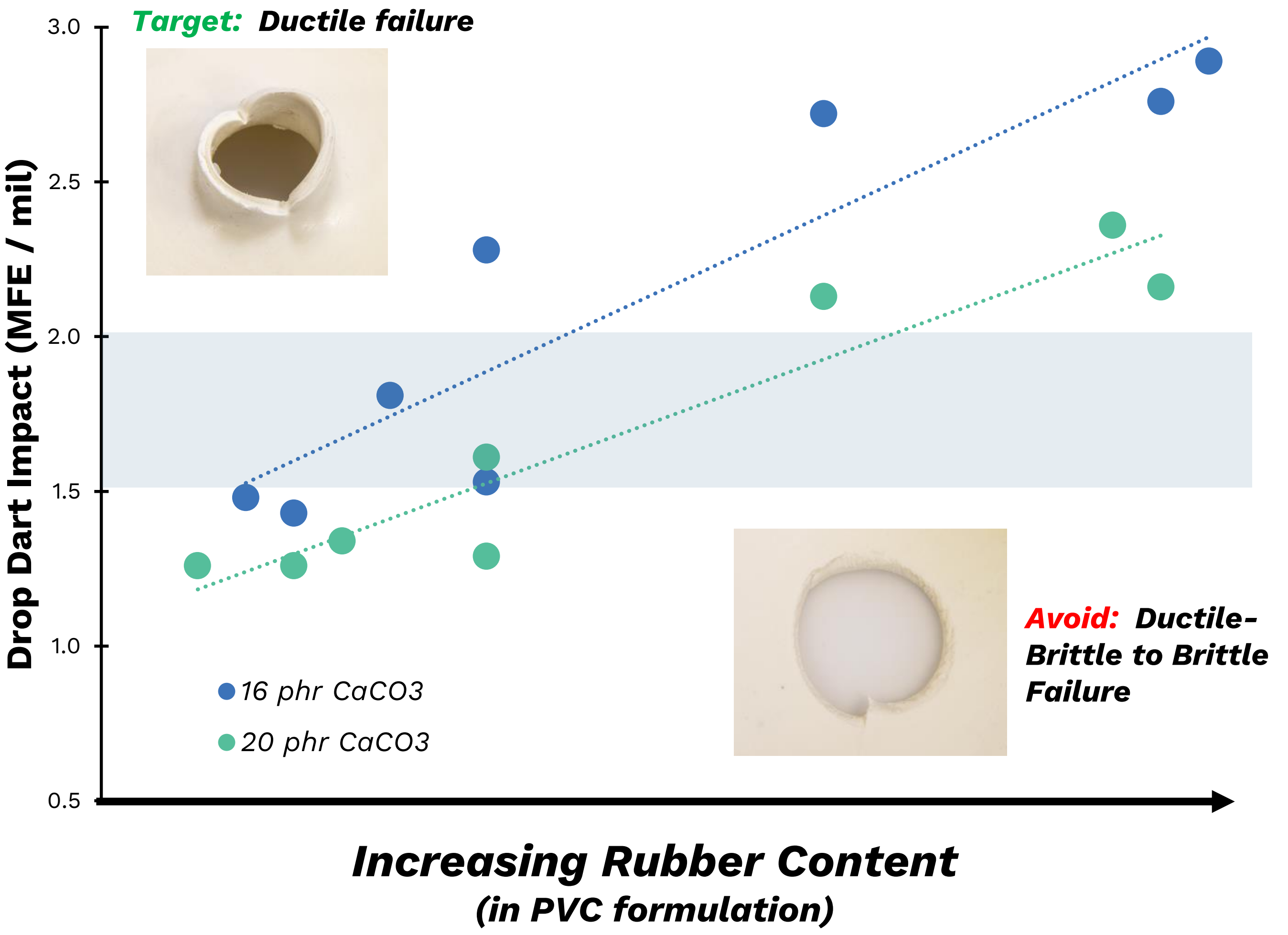
Catastrophic impact / falling weight evaluations

→ Core-shell impact modifiers protect against catastrophic failure

- Engineer formulations for ductile failure
- Prevent brittle "blow-outs" and cracking
- Withstand building material shipping, handling and installation
- Enhance extreme temperature performance

→ Formulation perspective:

- Increasing filler content typically reduces overall impact strength
- Monitoring overall rubber content in formulations can balance failure mode
- Acrylic content can compensate for processing differences leading to gelation optimization

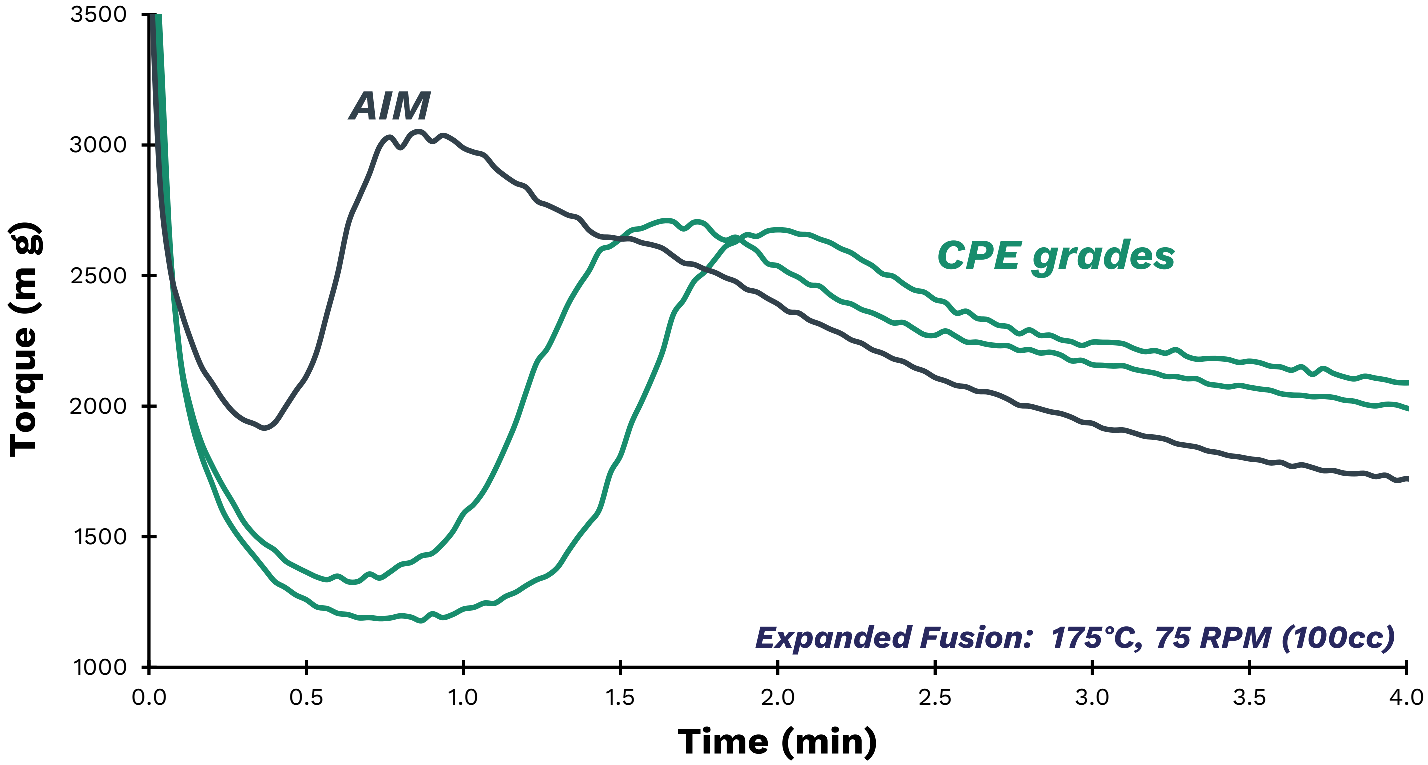


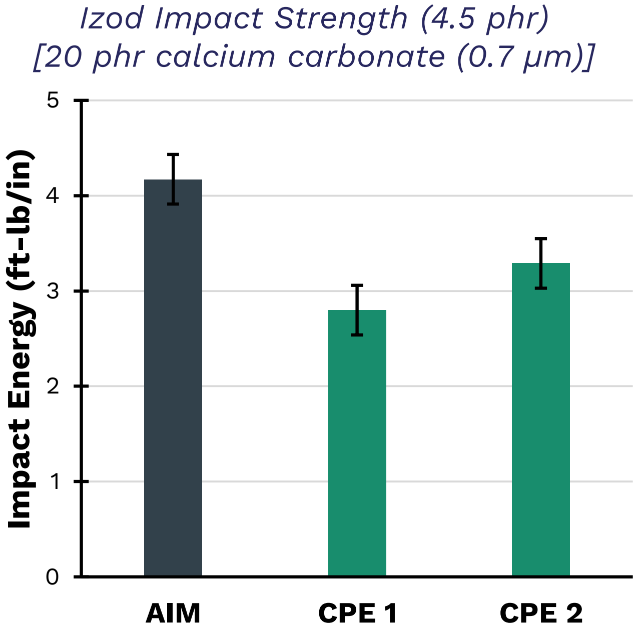
internal test results

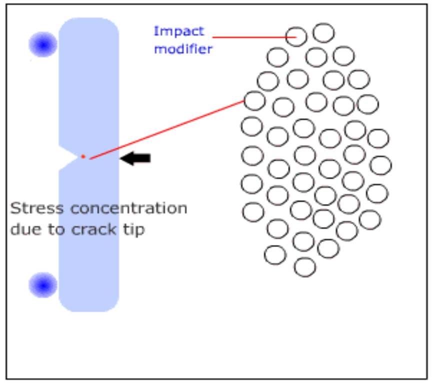
Notch sensitivity and impact strength

→ Core-shell impact modifiers protect against catastrophic failure

- Acrylic impact modifiers provide impact performance and also acrylic fusion
- Added mechanical integrity to tongue and groove assemblies and notch sensitive / exposed building material parts







internal test results

Acrylic process aids and rigid PVC formulations

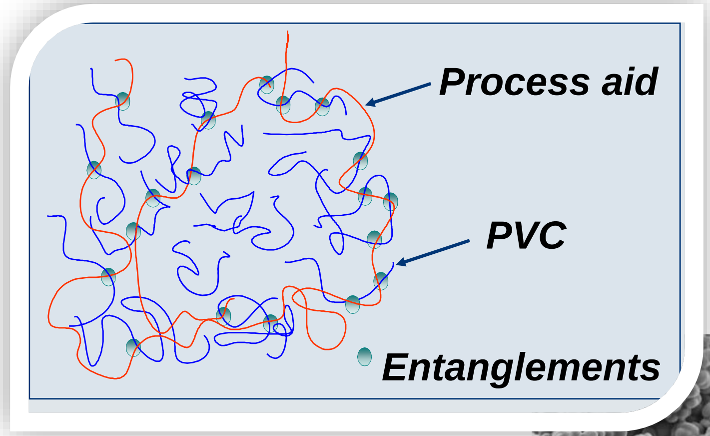
Acrylic process aids and PVC rheology

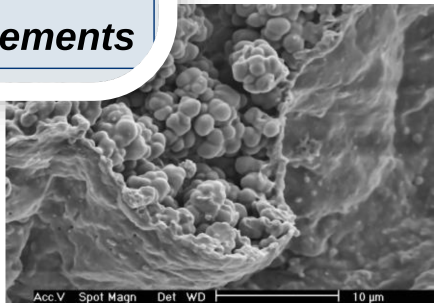
→ Process aids enhance fusion: force and shear transfer between primary PVC particles!

- Typically based on acrylic emulsion chemistry
- Can use a variety of acrylic and specialty monomers
- Molecular weights < 1 MM – 10 MM+ g/mol vs. PVC molecules < 100k g/mol
- Excellent compatibility with PVC matrix (high miscibility!)

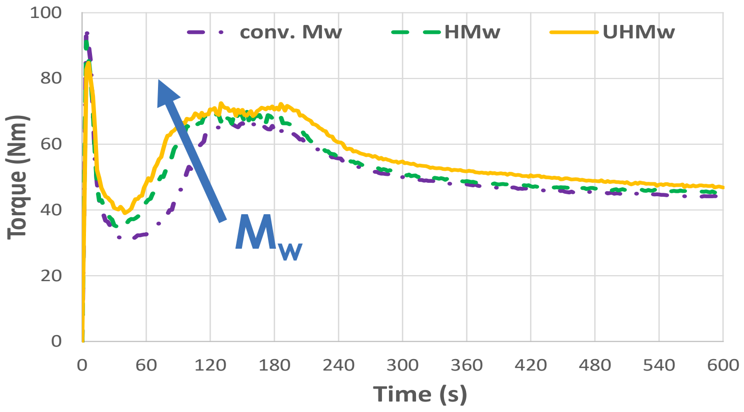
→ Process aid composition and rheology control

- Process aid Tg can manipulate fusion promotion
- Acrylic emulsion polymerization allows for a broad range of compositional and structural changes to the co-polymers
- Molecular weight effects can complicate Tg effects
  - Torque variation in equipment during fusion
  - Processing differences such as flow, dimensional stability

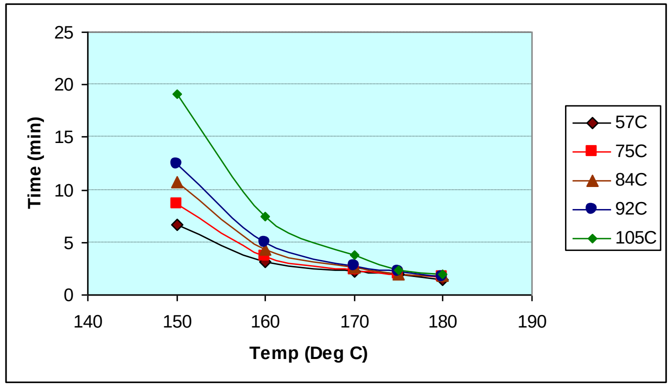




M_w effect



Tg effect



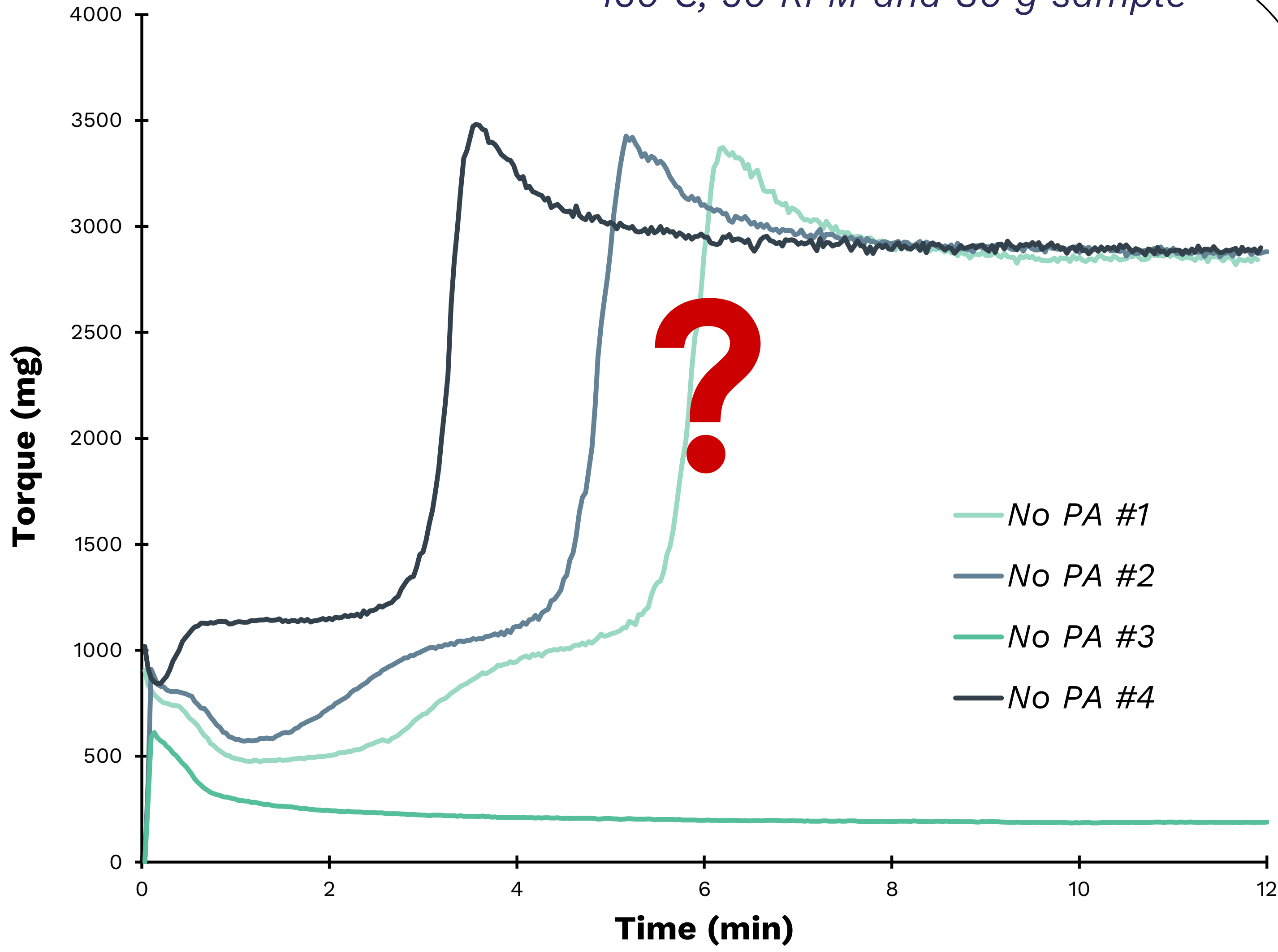
Consistent processing paramount with elevated filler content

→ Challenging production with higher filler incorporation continues:

- **At ultra-high filler loading levels**, luxury vinyl tile backlayer is ~ 65% $CaCO_3$
- **Filler necessary** for product performance with consumers, but difficult to incorporate into a continuous melt

250 phr $CaCO_3$ in formulation

130°C, 50 RPM and 80 g sample

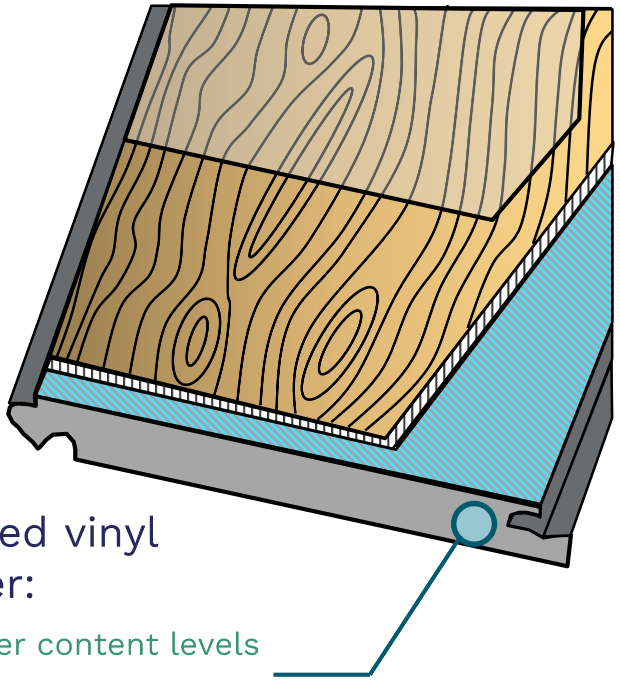


internal test results

Acrylic process aid in highly filled formulations

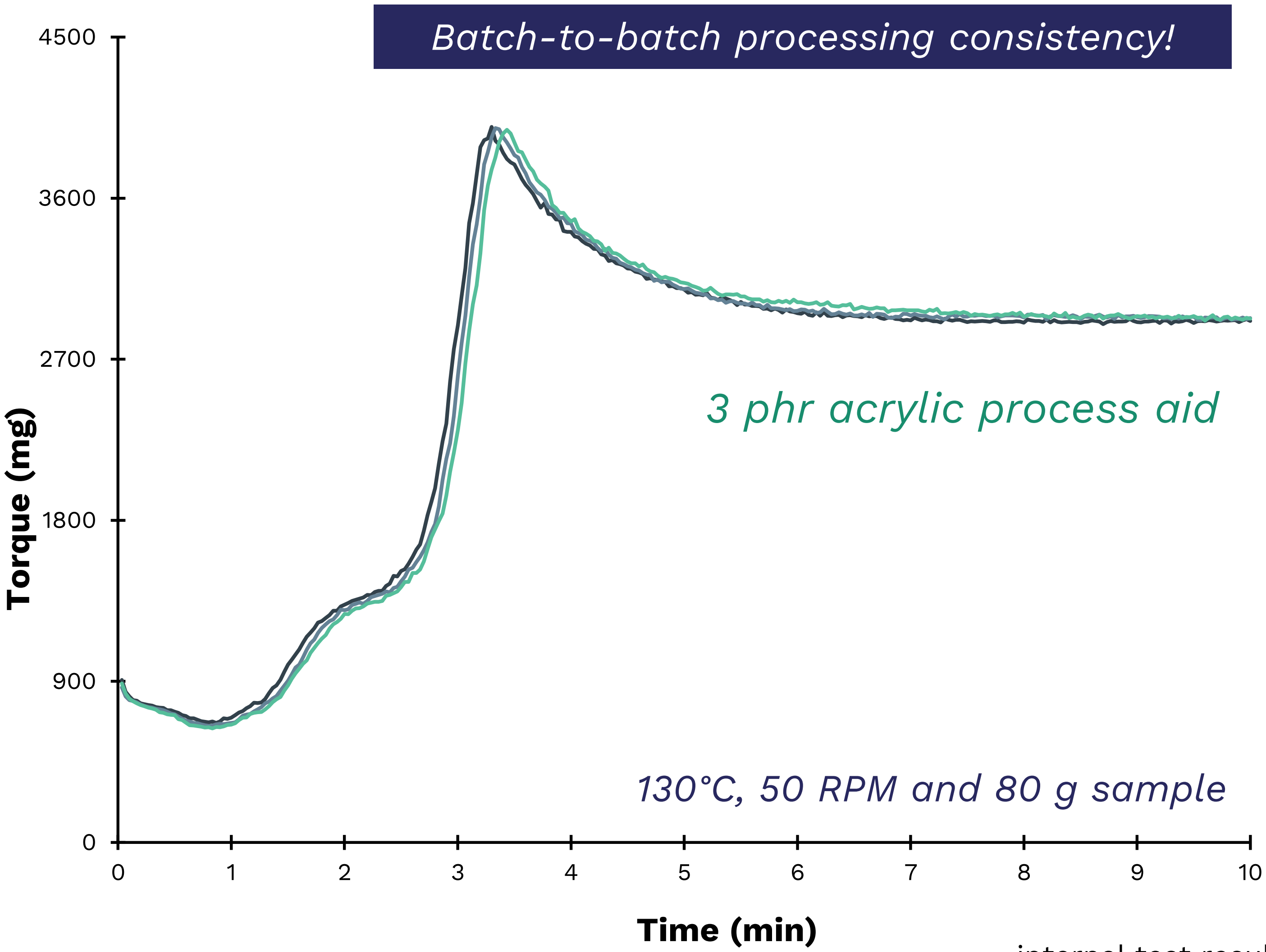
→ Excels in highly filled PVC formulations:

- Process aid provides processing consistency and promotes fusion



→ Reinforced vinyl backlayer:

- Extreme filler content levels
- Wide D50 range of calcium carbonate and filler used (µm)
- Wide range of blending and processing equipment



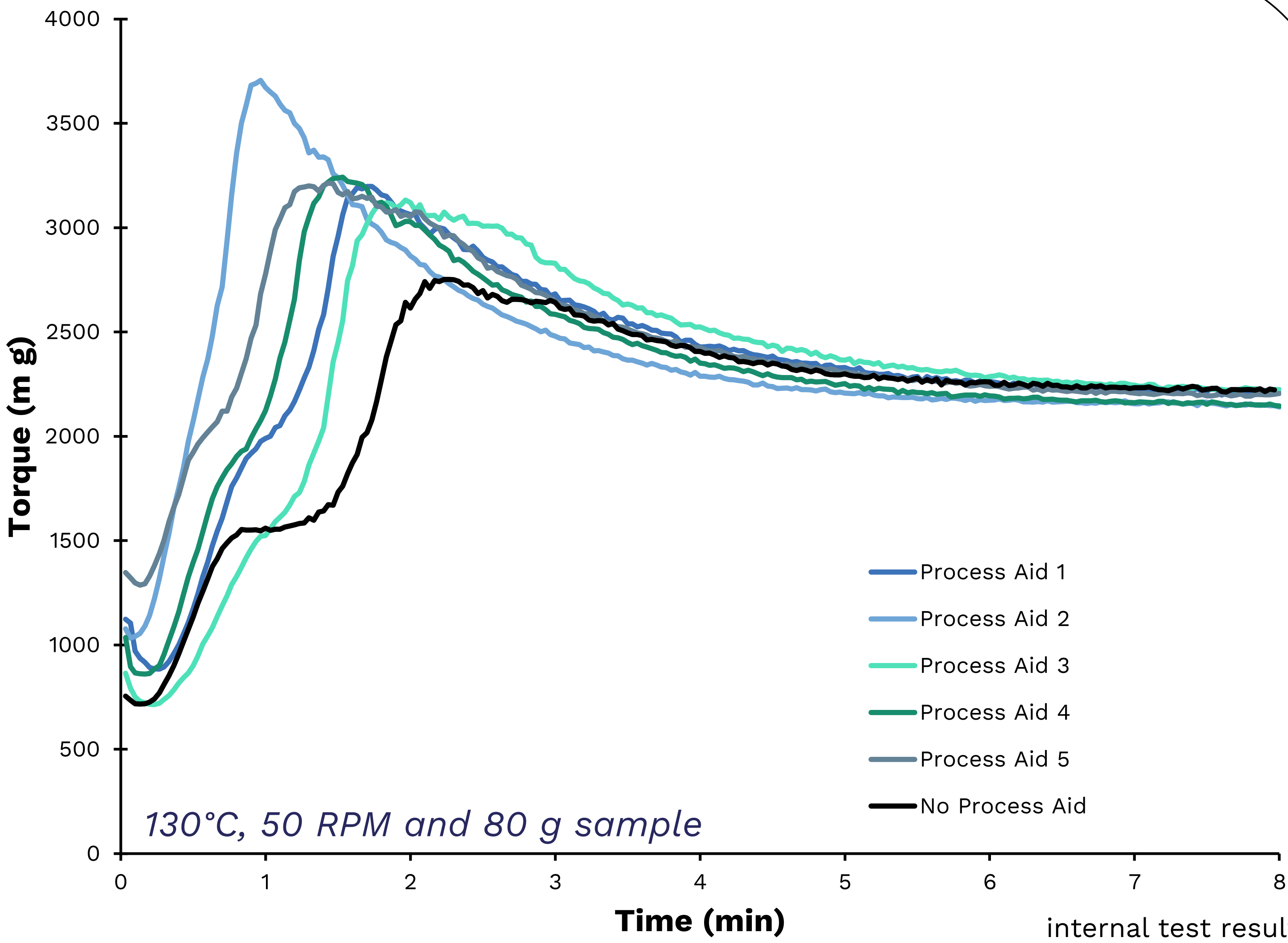
internal test results

Acrylic process aid influence on rheology

→ **Filler incorporation by process aid selection:**

- Torque and fusion time can be modified by product selection
- Process aid chemistry can determine effectiveness

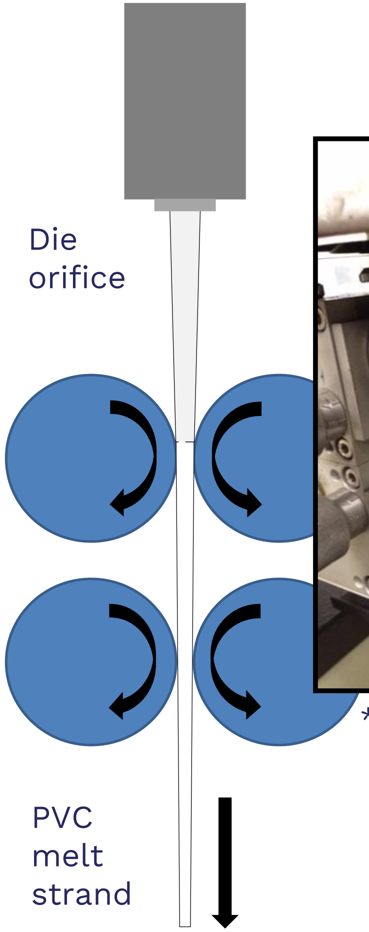
150 phr $CaCO_3$ in formulation

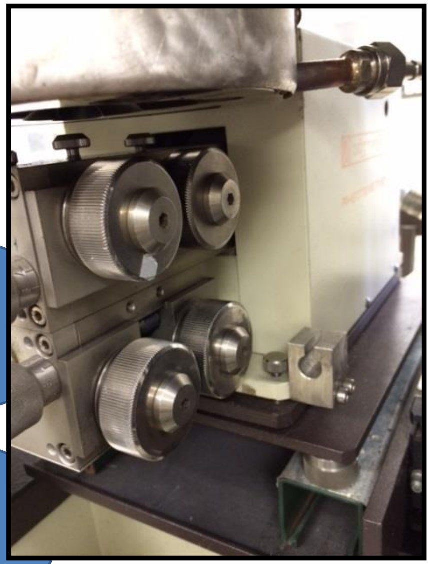


internal test results

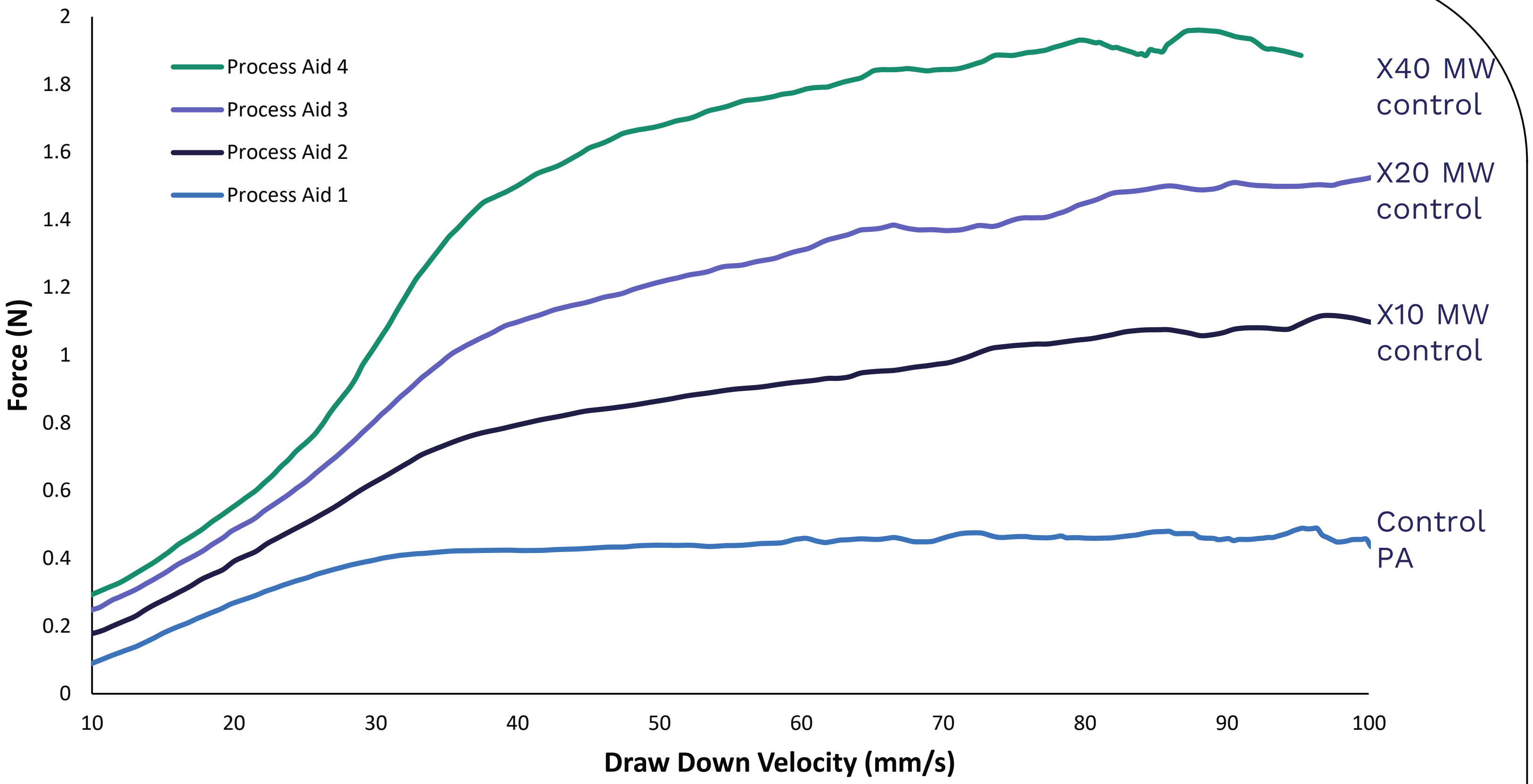
Melt strength guides process aid selection for foam applications

Rheotens: Melt Strength Evaluation





* Goettfert® Rheotens 71.97



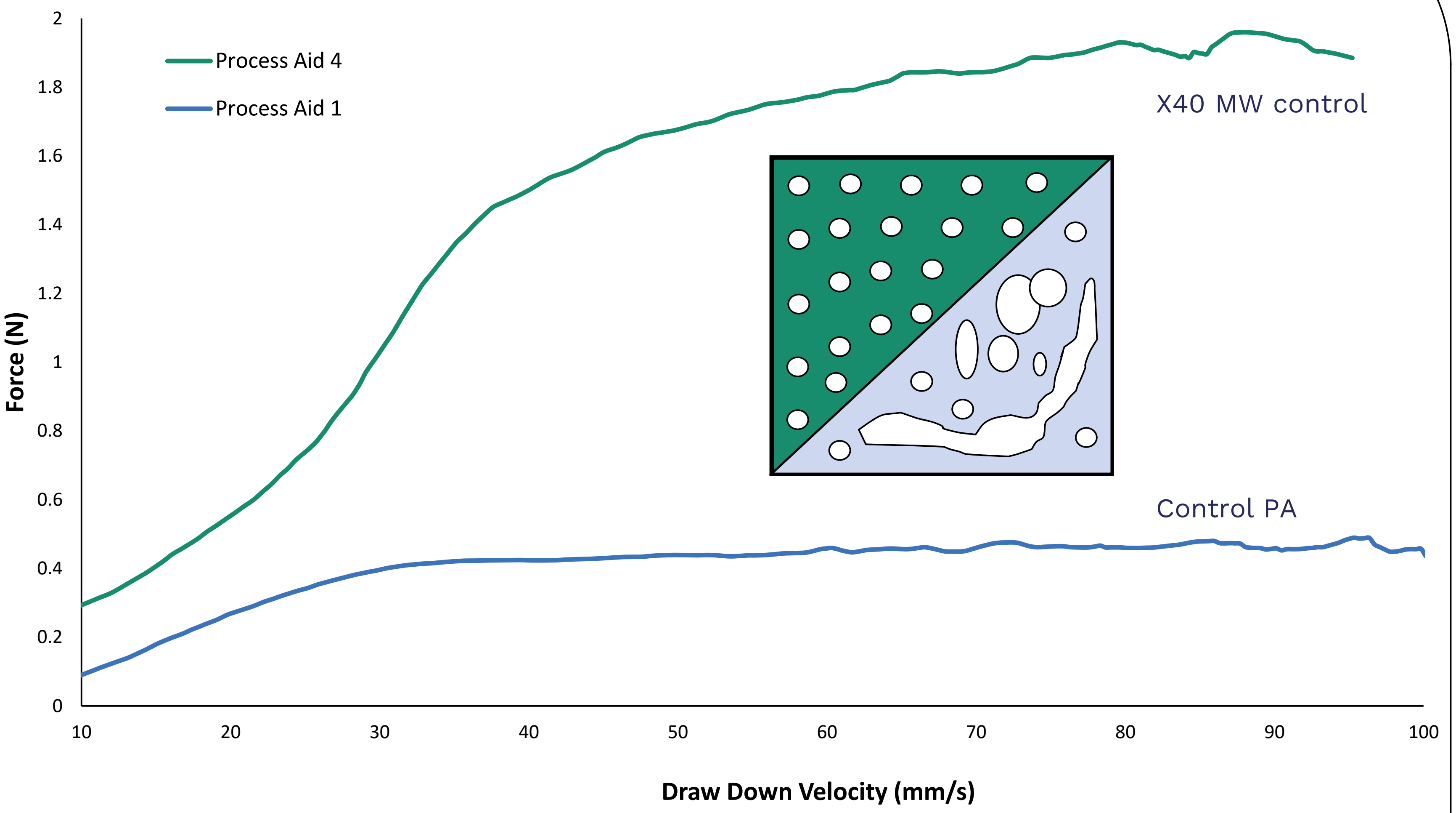
→ Process aid melt strength evolution based on molecular weight:

- Force versus velocity: drawing down the strand out of the die (load cell)
- Remember: the curves represent PVC compound melt strength (PVC K-value = 58)
- The differences in melt strength are caused by process aid selection

Melt strength guides process aid selection for foam applications

→ Cell structure based on available melt strength:

- Melt strength largely determined by acrylic process aid selection
- Capturing and surviving gas evolution most important during cellular PVC processing
- If MW chosen is too low, large voids will appear in the foam structure throughout the PVC article

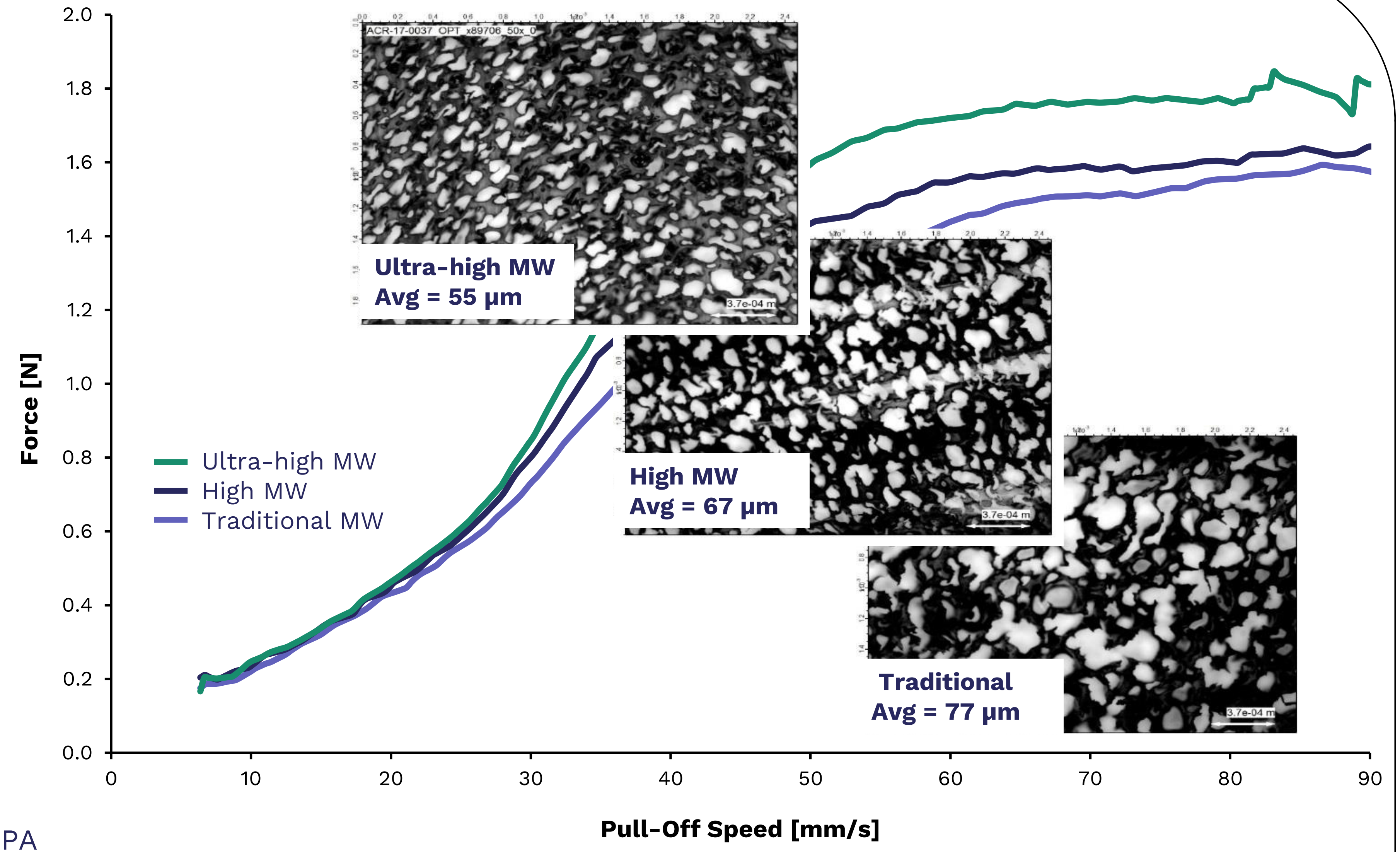


Acrylic process aids for foam PVC and density reduction

PVC melt strength and cell structure uniformity ↑ as M_w ↑

→ Molecular weight for cellular applications is critical:

- Generally, increasing molecular weight is desirable for improved efficiency and final performance
- Drawbacks of ultra-high MW technology are possible with diminished melt flow and high extrusion torque
- Process aid selection based on understanding processing needs is essential



*cellular PVC formulations w/ 5 phr PA

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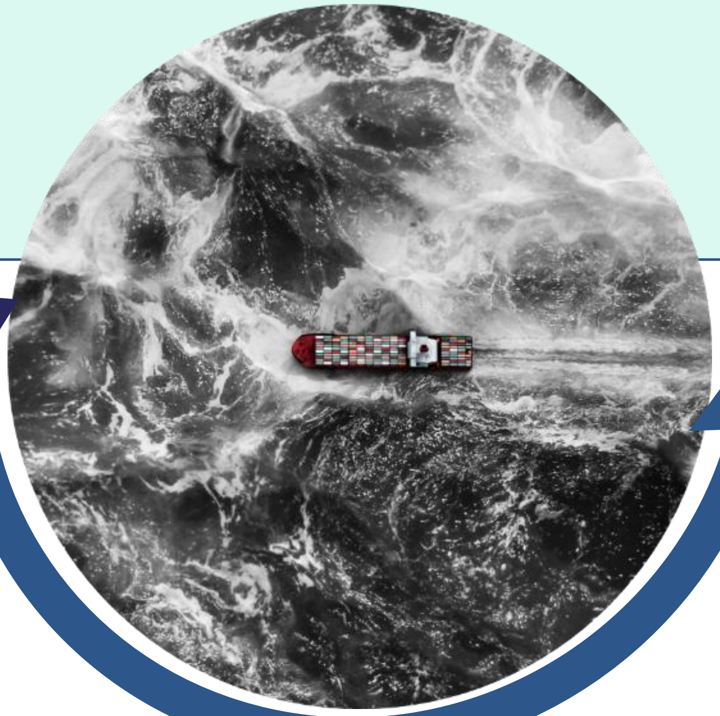
Warehousing
Regional warehousing of acrylic additives

Responsive Service
Local sales and customer service



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Questions and Answers

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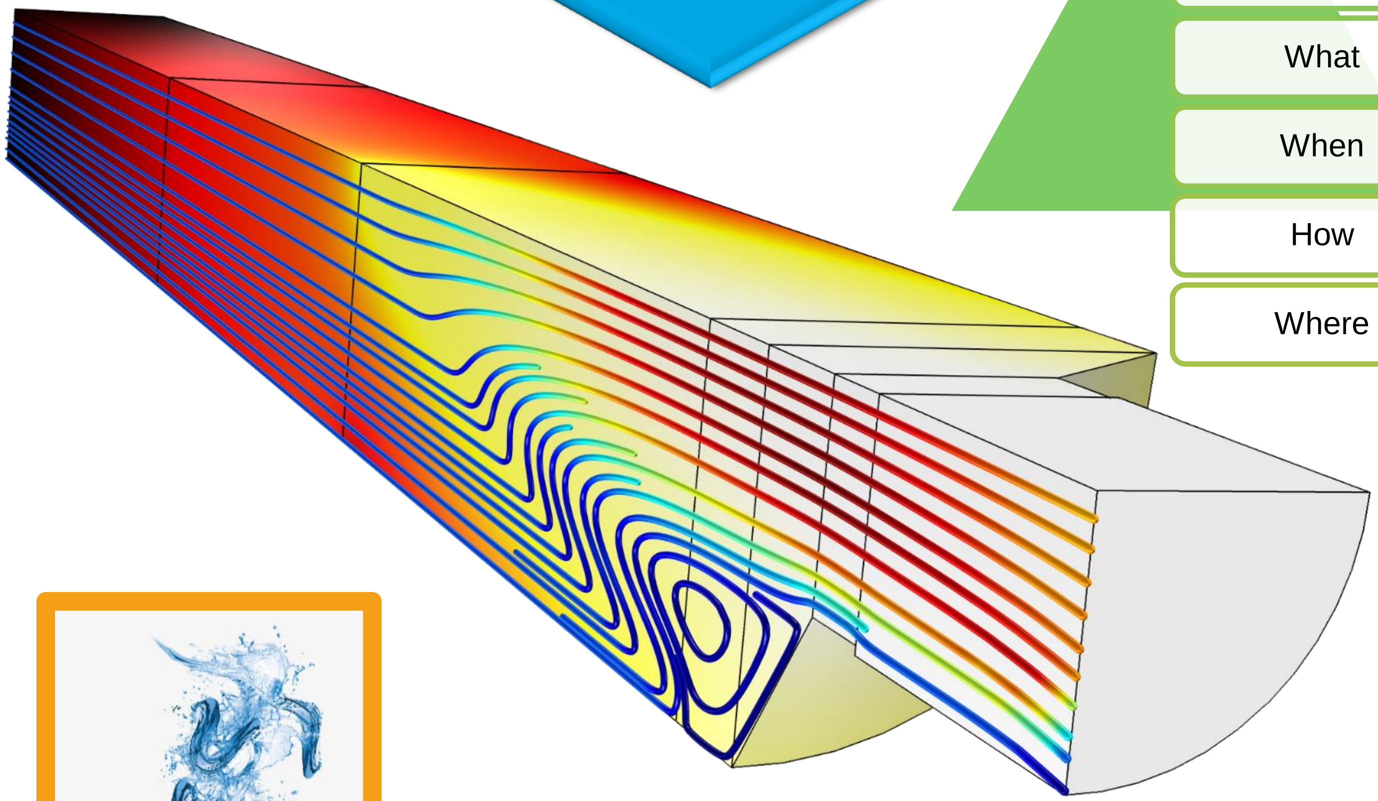
Allow us to show you the way

- Go out of your way
- Make an impact
- Who
- What
- When
- How
- Where



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Questions and Answers

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