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Microstructure - Rheology Relationship

Study of Mineral Reinforced Nylon 6

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Introduction

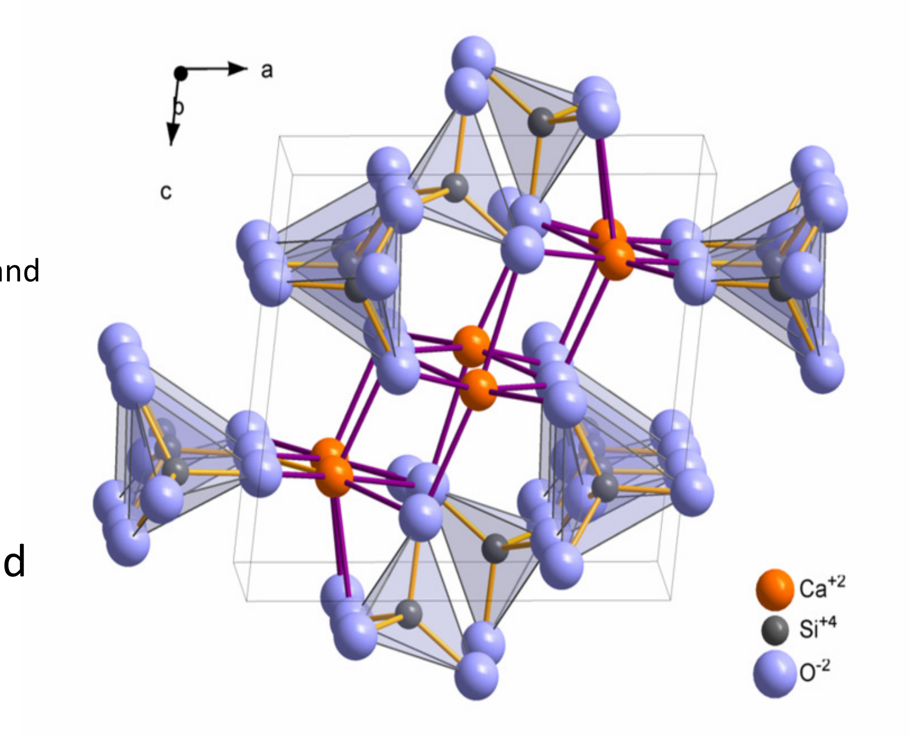
- NYLON-6 is widely used in engineering fields
  - Easy process ability
  - good Thermal Stability
  - Semi Crystaline Plastic; good mechanical characteristics.
  - low heat distortion temperature
  - High water absorption
- Wollastonite
  - High Purity,
  - high brightness,
  - acicularity,
  - low oil and moisture absorption,
  - Availability-cost
- Wollastonite Reinforced Nylon
  - Balance of physical properties and processability
  - Improve Mechanical strength and stiffness
  - Increase Heat Distortion Temperature
  - Nice surface finish and reflectivity
  - Reduce Warpage and better dimensional stability
  - Natural smoke and fire suppressant

Wollastonite

- It is the only naturally occurring, nonmetallic, white, needle-like mineral
- Aspect Ratio range of 8-20.
  - Lower aspect ratio compare to GF but it's thinner and has higher surface area

- **Advantage:** chemical purity, thermal stability, low water absorptivity, white colour, and small health hazard
- **Disadvantages**: its relatively high price and high hardness in comparison to other particulate fillers that may damage the processing equipment

- It is widely used with Polyamide 6 & 66, Polypropylene (PP), Polycorbonate (PC), Polyurethane (PU), PEEK, Polystyrene (PS), Thermoplastic elastomers (TPE) etc.

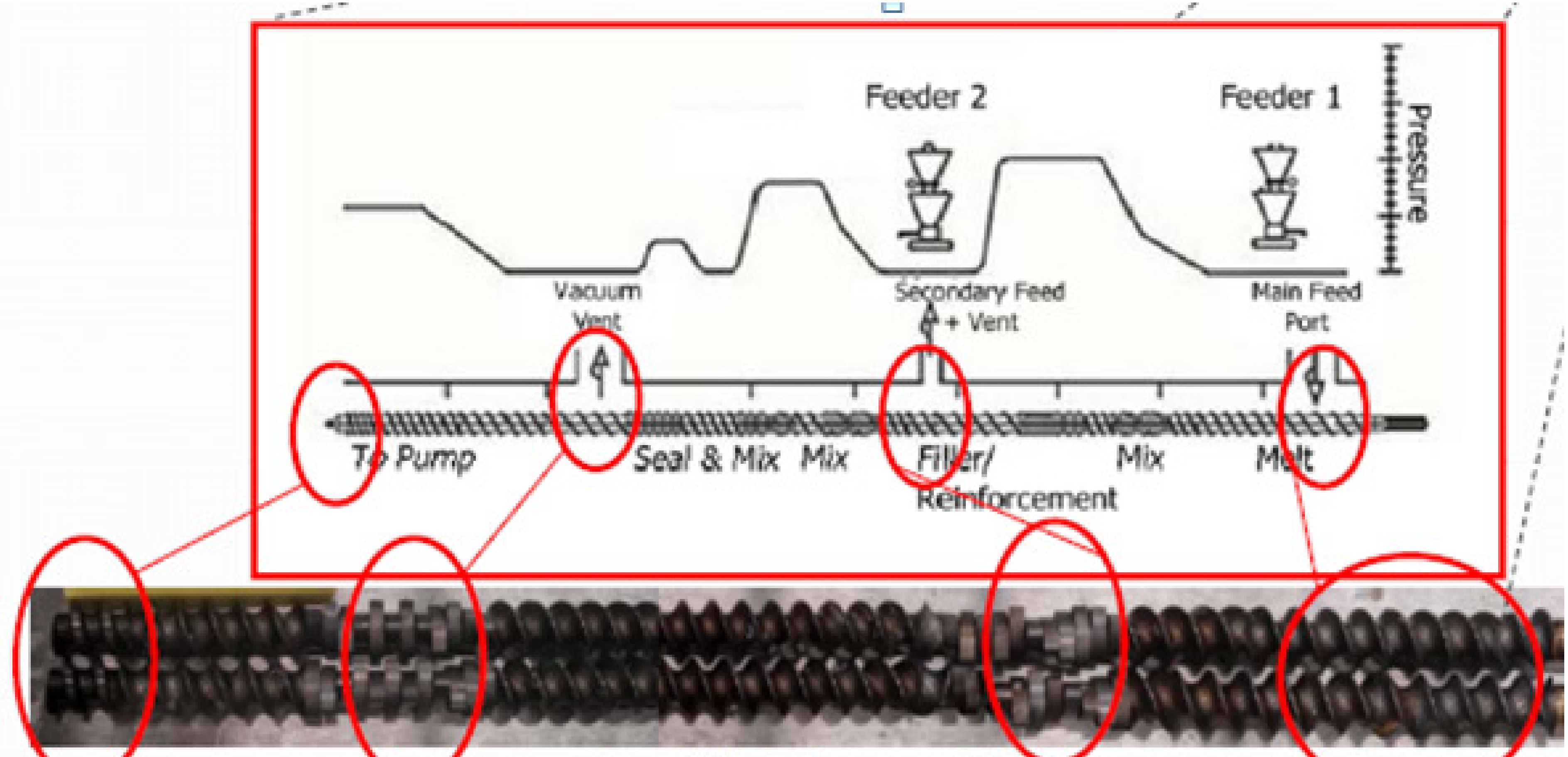


Calcium Meta Silicate - $CaSiO3$
specific gravity of 2.9,
Mohs hardness of 4.5
Refractive index between 1.63 and 1.67.

Compounding

- Nylon-6/Wollastonite composites were prepared by corotating twin screw extruder
- challenges
  – Mix Filler and matrix uniformly
  – Broke filler agglomerate
  – Minimum Filler breakage
    - Controlled by screw profile

Different Zones

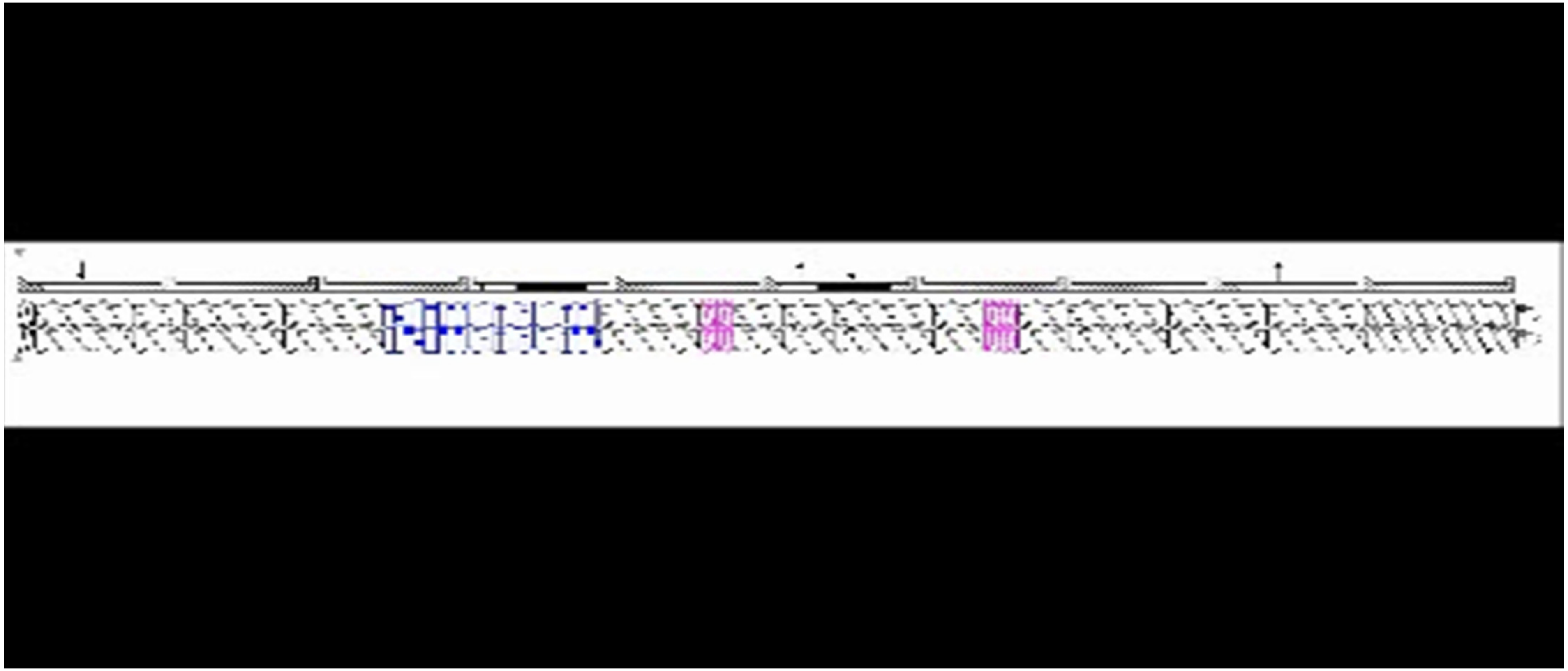


Co- rotating twin screws 400mm

Engineering Screw Configuration

- Compromise between mixing level and Wollastonite retained Aspect ratio
- Use Shear and Elongation fields
- Control shear Stress
  - Adjust pitch of kneading blocks
  - Reverse elements to build to pressure
  - Screw Speed
    - Control shear rate
    - Pressure build up in die
    - Pseduplastic behaviour

Screw Design Software



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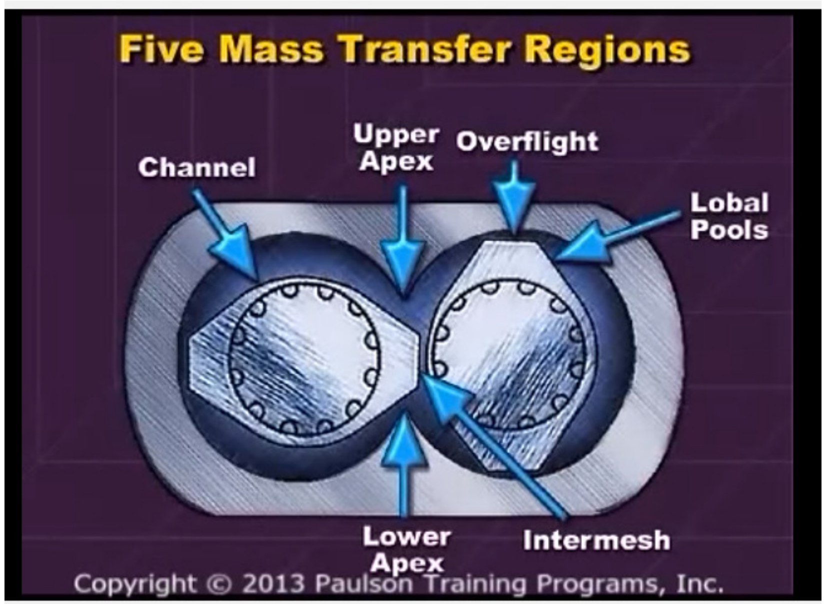
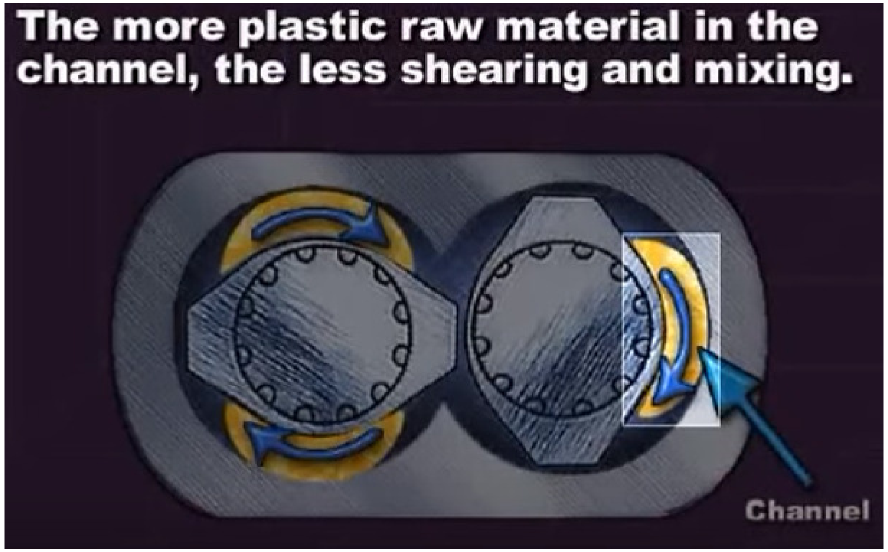
Starve feeding

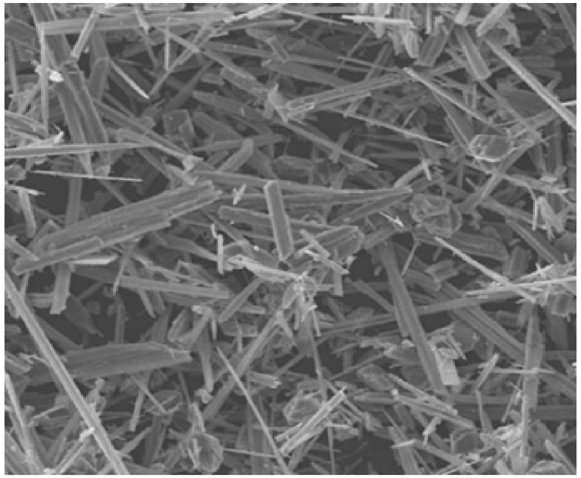
Control the percentage of material in high shear zones;





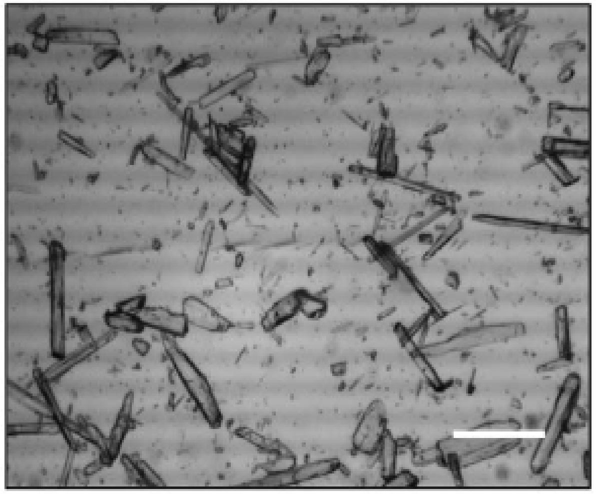
Microscopic Image

Wollastonite Before Compounding

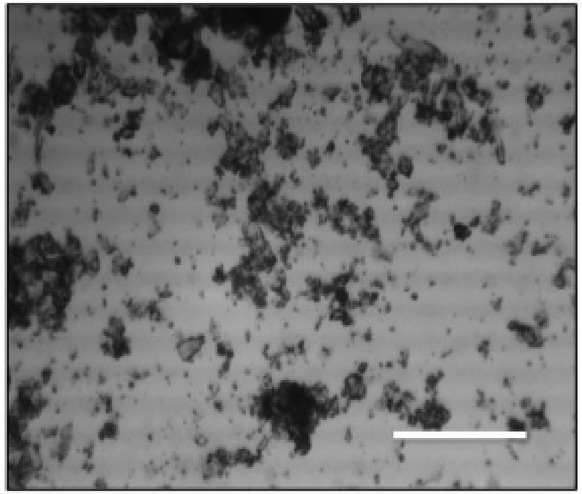


250X

Wollastonite After Mild Compounding



Wollastonite After Strong Compounding



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Theoretical Aspects

The mechanical properties of a reinforced thermoplastic are controlled by

- Filler and matrix individual properties
- Filler Volume fraction
- Filler orientation
- Filler- Matrix interface strength
- Filler Aspect Ratio

$$E_{Composite} = k\,\eta_l\,\eta_o\,V_{filler}\,E_{filler} + V_{matrix}\,E_{matrix}$$

E_c = Young's modulus of the composite

E_f = Young's modulus of the fibre

E_m = Young's modulus of the matrix

V_f = fibre volume fraction

V_m = matrix volume fraction $(1-V_f-V_v)$

V_v = void volume fraction

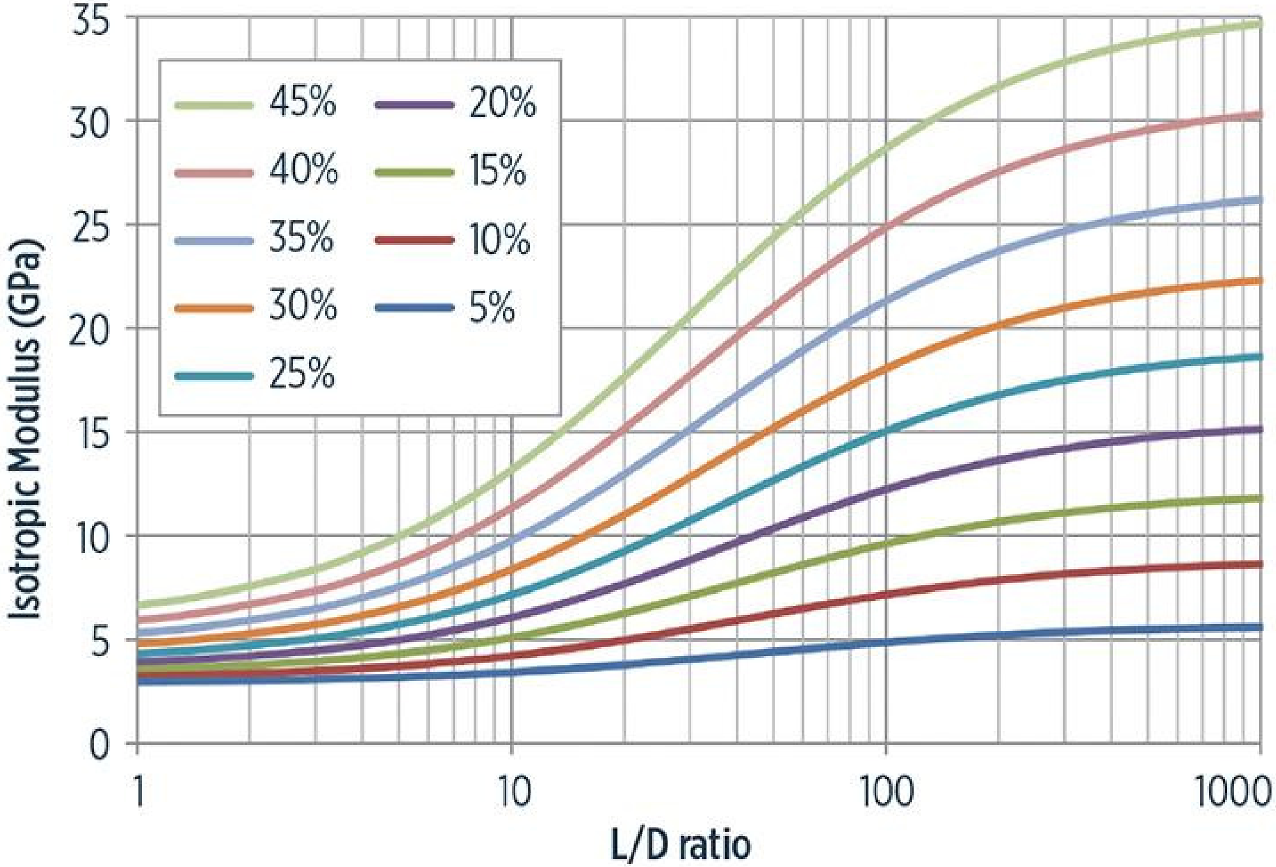
κ = fibre area and Diameter correction factor

η_l = fibre length distribution factor

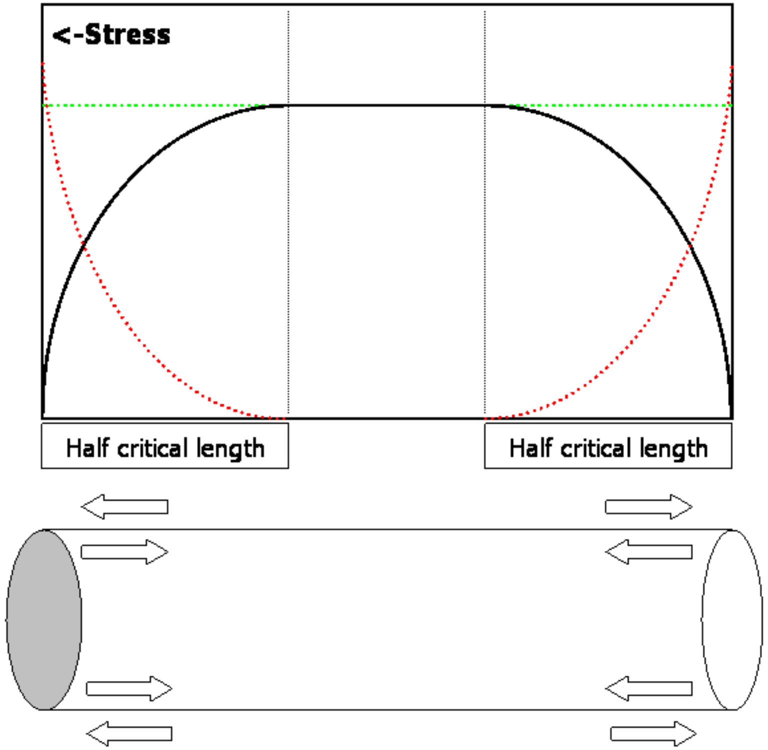
η_o = fibre orientation distribution factor

Fig. 1

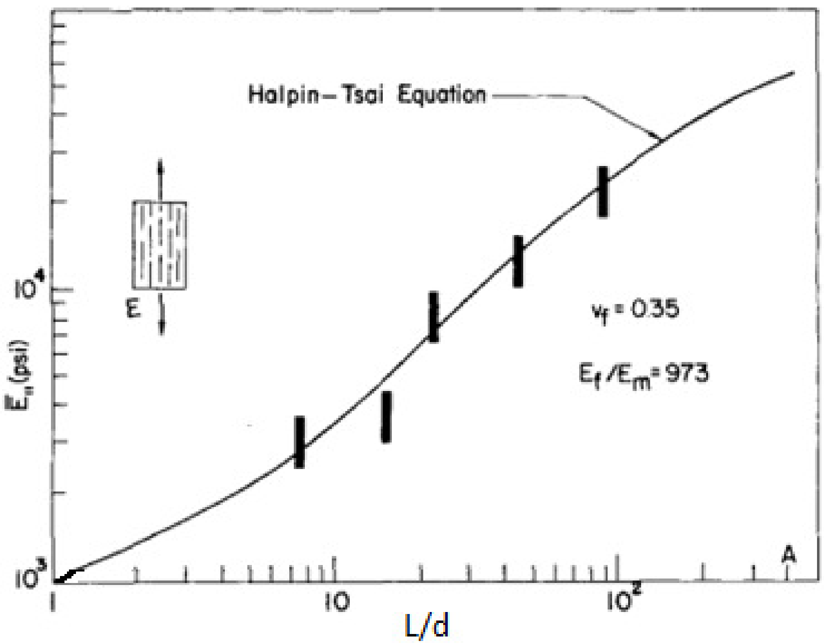
Isotropic modulus vs. fiber aspect ratios and various fiber weight percentage. Note that the figure shows *fiber weight percent* in the compound to be an important factor in achieving higher modulus.



Aspect Ratio



- The optimum performance of a fibre-like reinforming filler cannot be achieved below aspect ratio of 100:1



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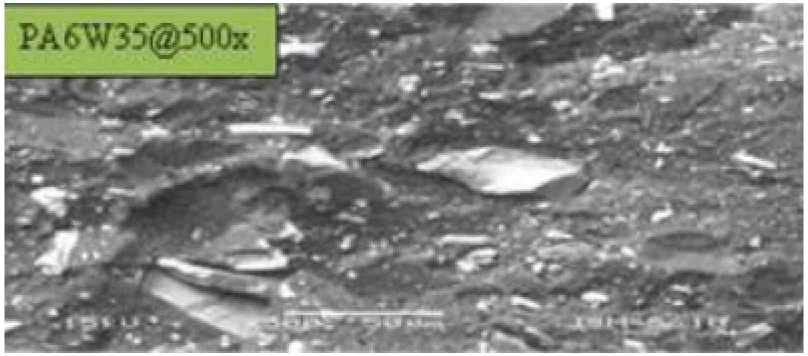

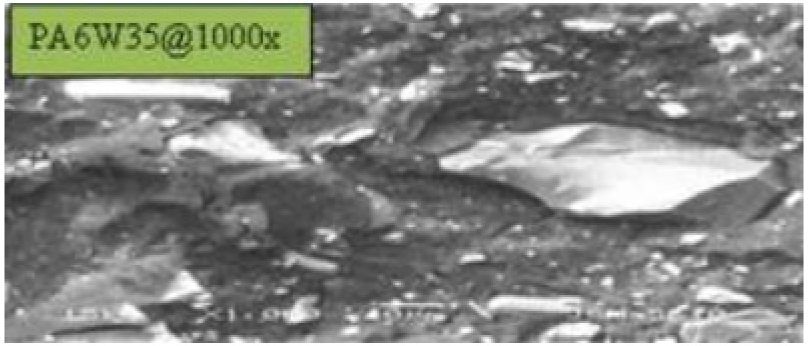

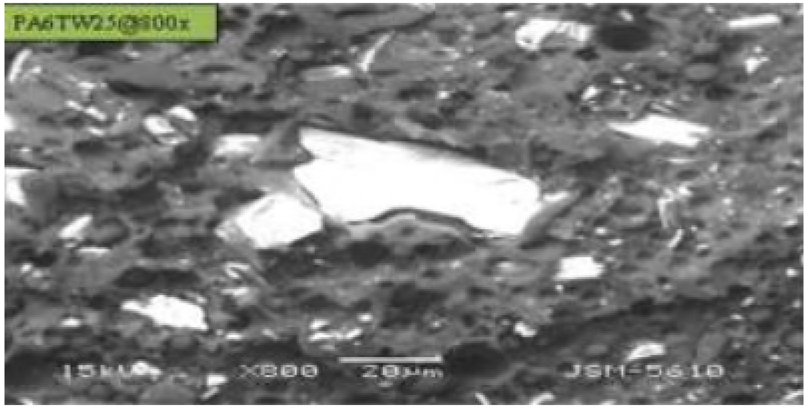

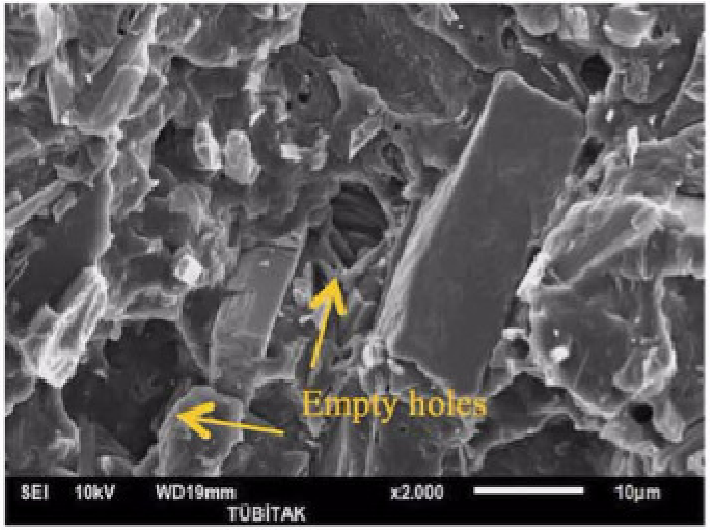

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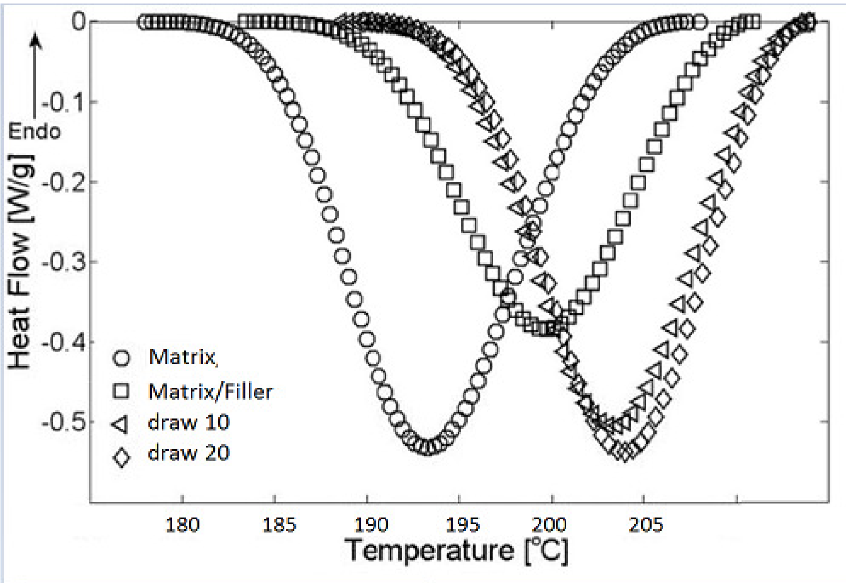
Microstructure

- In most synthetic materials, the spatial orientation of the discontinuous Filler is between a truly random 3D arrangement and a random 2D arrangement
- The Melt flow breaks the 3D network and align needle like Wollastonite ;
  - Filler orientation
  - affect the final crystallization and melt behaviour
- Studies show that rheology can be used as a powerful tool for studying composite microstructure

Induced crystallization

- Enhanced nucleation caused by flow
- Orientation o f polymer
- Wollastonite act as a start point for crystalization
- High aspect ratio fillers in a shear field can intensify adjacent polymer chains alignment and increase crystalization
- Alongational flow

Change in Crystallinity



- Higher Tc, nucleating effect
- Higher crystalinity % (22 compared to 18%)
- Higher melting point : Thicker Lamelaes

Rheological properties of the Composite

- Adding Fillers increased shear viscousity
- The effect of Wollastonite is more than glass Fibre (larger surface area)
- More shear thinning behaviour
- Elongation resistance will increase
- More elastic behaviour
- Fibre orientation control flow rate

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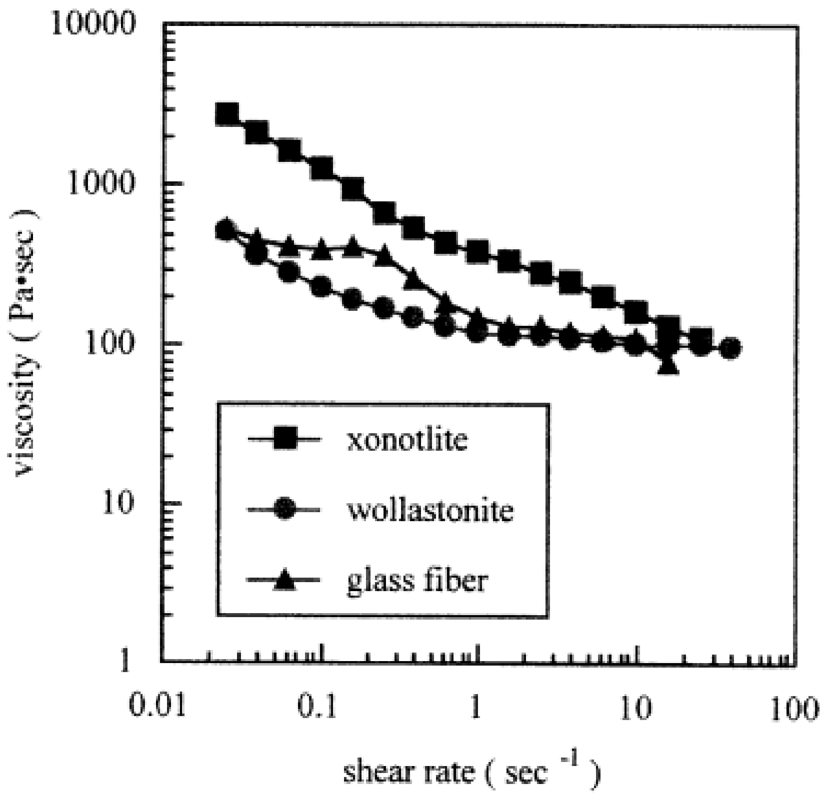
Dynamic Melt Behaviour

- Rheological behaviour of polymer melt reinforced by Microfibrous mineral fillers is quite different from Glass/aramid fibre filled composites.
- At low shear: GF composites act as Newtonian fluid. Wollastonite filled composites show yield.
- yield stress is inversely proportional to particle diameter



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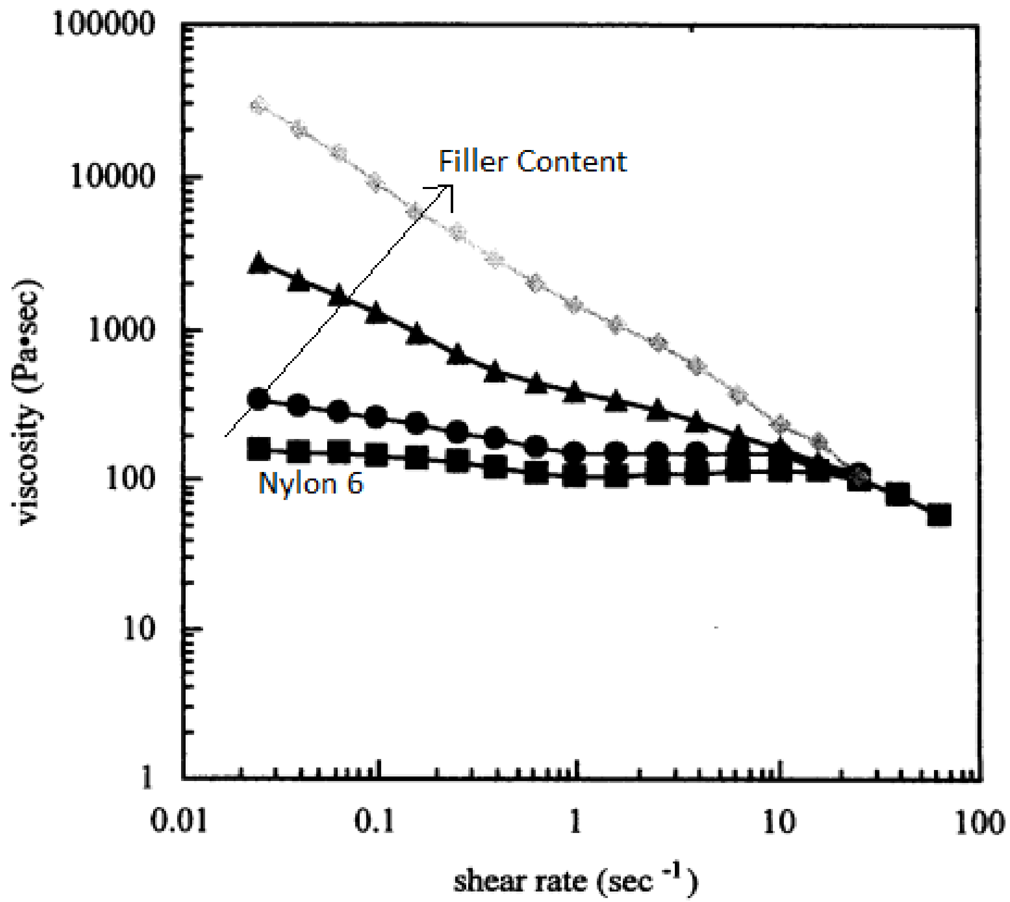
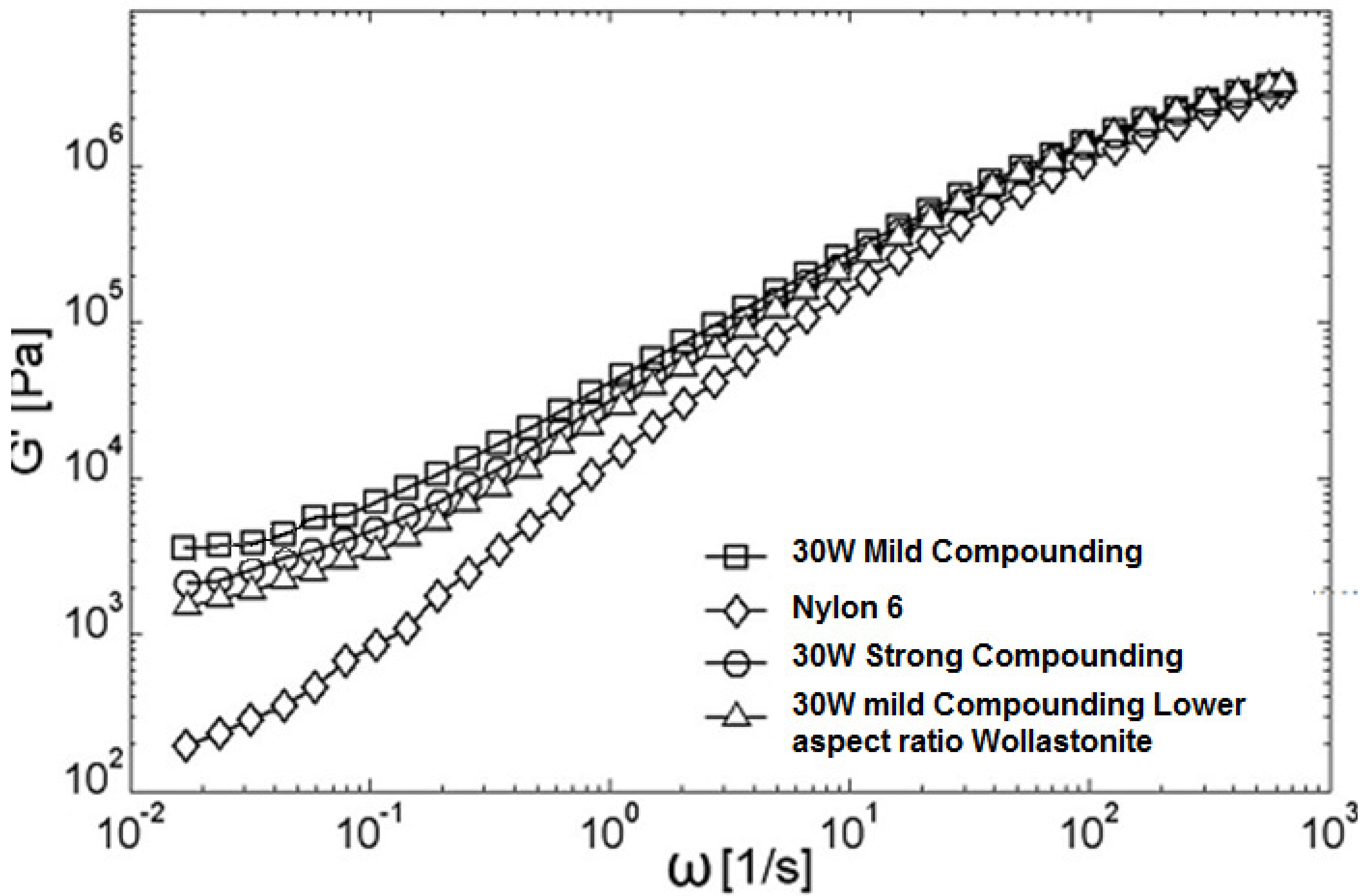
Shear Rate dependency



Storage Modulus



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Mechanical Properties

Sample	Flex Modulus, Mpa	Tensile Strength, MPa	Calculated L/d	Microstructure
30% Wollastonite, Mild Mixing	7500	100	17	3D network, Filler orientation, Induced Crystalinity
25% Wollastonite, Strong Mixing	4000	70	5.5	Random dispersion, no solid like behaviour
Matrix (Nylon 6)	2200	75	NA	Semicrystaline

HDT

	Matrix	15%	25%
Normal Mixing	164	179	185
Mild Screw Profile	164	185	190

- Increased Stiffness of the Composite
- Higher Crystallinity

conclusion

- Composite stiffness and strength is directly proportional to the Filler survived length after mixing
- To achieve desired Mechanical Properties, a certain microstructure is needed and it can achieve by controling elongational and shear fields in the extruder.
- Rheology is a strong tool to study Microstructure and so the final properties of a composite

Thank you For your Attention

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