CNC-based Glass Fiber Sizing for Composite Reinforcement

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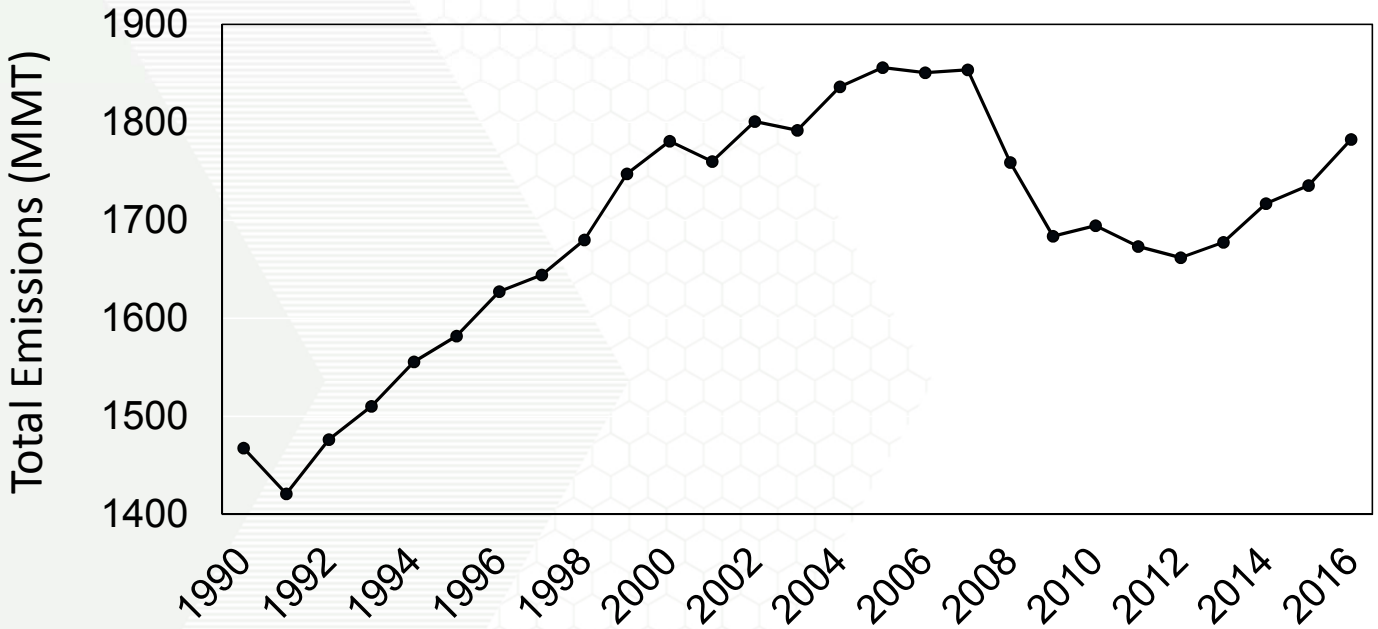
TAPPI

Motivation

AUTOMOTIVE EMISSIONS PROBLEM



Transportation accounts for the most greenhouse gas emissions of any economic sector in the United States – and the trend is worsening

Inventory of U.S. Greenhouse Gas Emissions and Sinks: 1990-2017, E.P. Agency. 2019.

LIGHTWEIGHT MATERIALS FOR FUEL ECONOMY

Heavy

Challenge

Prohibitive

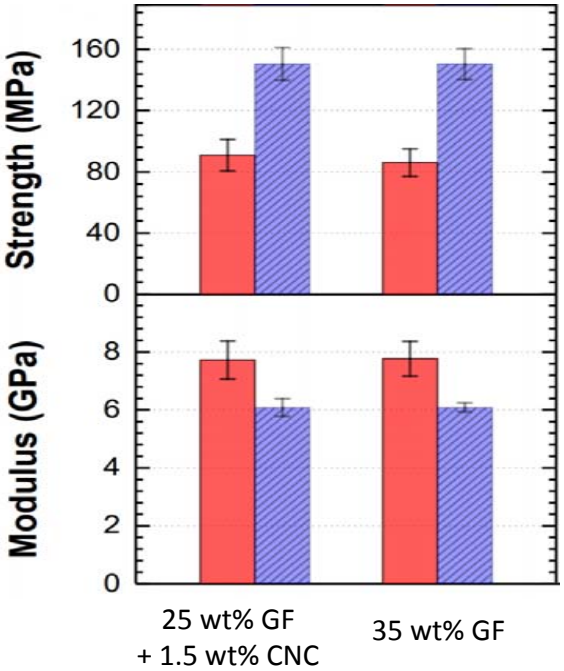
Mat	Density (g/cm³)	Comparison to steel			
		Strength/density	Modulus/density	Cost	Mass reduction potential
Mild steel	7.87	1	1	1	0%
High-strength steel	7.87	1.86	1	0.9–1.2	10%
steel	7.87	3	1	0.8–1.5	10%–28%
th steel	7.87	7	1	1.0–2.0	15%–30%
Ceramics	3.9	0.7	3.05	1.5–3.0	10%–30%
Sheet molding compound	1.1–1.9	4.39	1.16	0.5–1.5	20%–30%
Glass fiber composites	1.4–2.4	4.74	5.75	0.9–1.5	25%–35%
Plastics	0.9–1.5	0.82	0.08	0.7–3.0	20%–50%
Aluminum	2.7	3.95	1.02	1.3–2.0	30%–60%
Titanium	4.51	4.73	0.98	1.5–10	4
Metal matrix composites	1.9–2.7	5.41	35.28	1.5–3.0	50%–65%
Magnesium	1.74	3.66	1.02	1.5–2.5	0%–70%
Carbon fiber composites	1.0–1.6	20.9	5.41	1.5–5.0	50%–70%

U.S. Department of Energy: The Quadrennial Technology Review. p.290 (2015)

WEIGHT REDUCTION WITH NANOFILLERS

CNC have been shown to be effective nanofillers in composites

- Improves tensile/flexural properties in fiberglass composites
- Allows for removal of glass fibers, **lowering composite weight**



Asadi et al. *Composites: Part A* 88 (2016)

SCALABILITY CONCERNS

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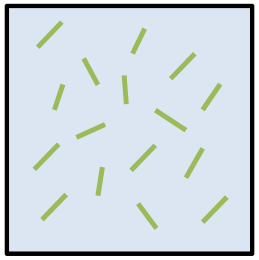
Difficult to mix with polymers

- CNC hydrophilicity limits compatibility
- Worse properties, harder to scale



CNC in H_2O

CNC in Polymer

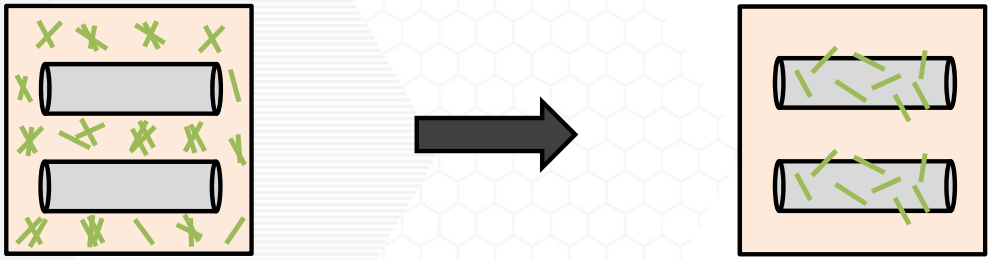
Must develop alternate approach

- Coating fibers has shown potential

	Fiber (0.17 wt%)	Epoxy (1 wt%)
Tensile modulus	+10%	+25%
Tensile strength	+10%	+30%
Flexural modulus	+40%	+44%
Flexural strength	+42%	+33%

Asadi et al. *eXPRESS Polymer Letters* 10 (2016)

GOAL: DEVELOP OPTIMIZED CNC COATINGS



Disperse CNC in epoxy

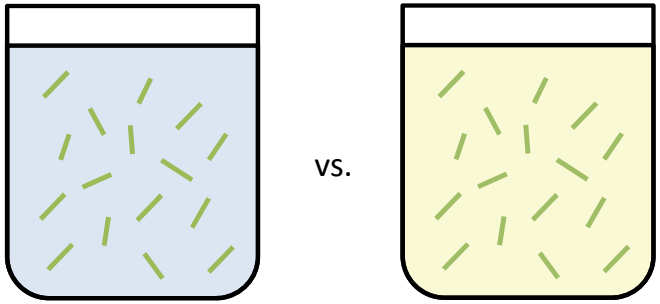
Add CNC to glass fibers

Approach

CNC COATING VARIABLES

Optimize interface via 3 parameters:

- *CNC dispersant (water, commercial sizing, triblock copolymer emulsion)*

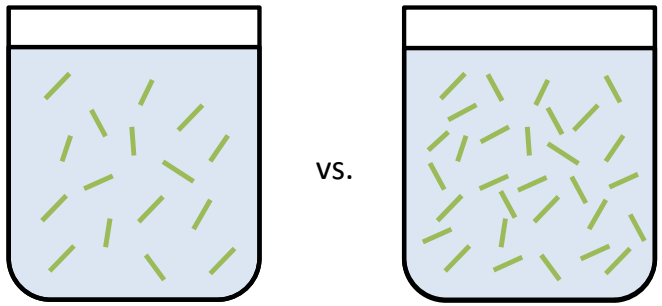


Sizing Solutions Containing CNC

CNC COATING VARIABLES

Optimize interface via 3 parameters:

- CNC dispersant
- *CNC concentration (1 wt% vs. 2 wt%)*

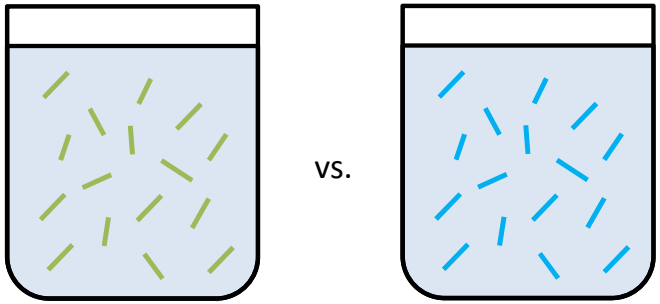


Sizing Solutions Containing CNC

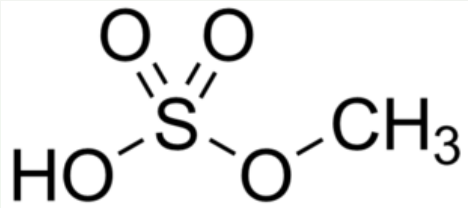
CNC COATING VARIABLES

Optimize interface via 3 parameters:

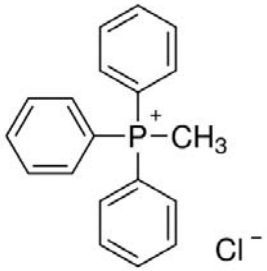
- CNC dispersant
- CNC concentration
- *CNC functionality (CNC-S vs. CNC-Ph)*



Sizing Solutions Containing CNC



CNC-S (11.9 wt% suspension from FPL)

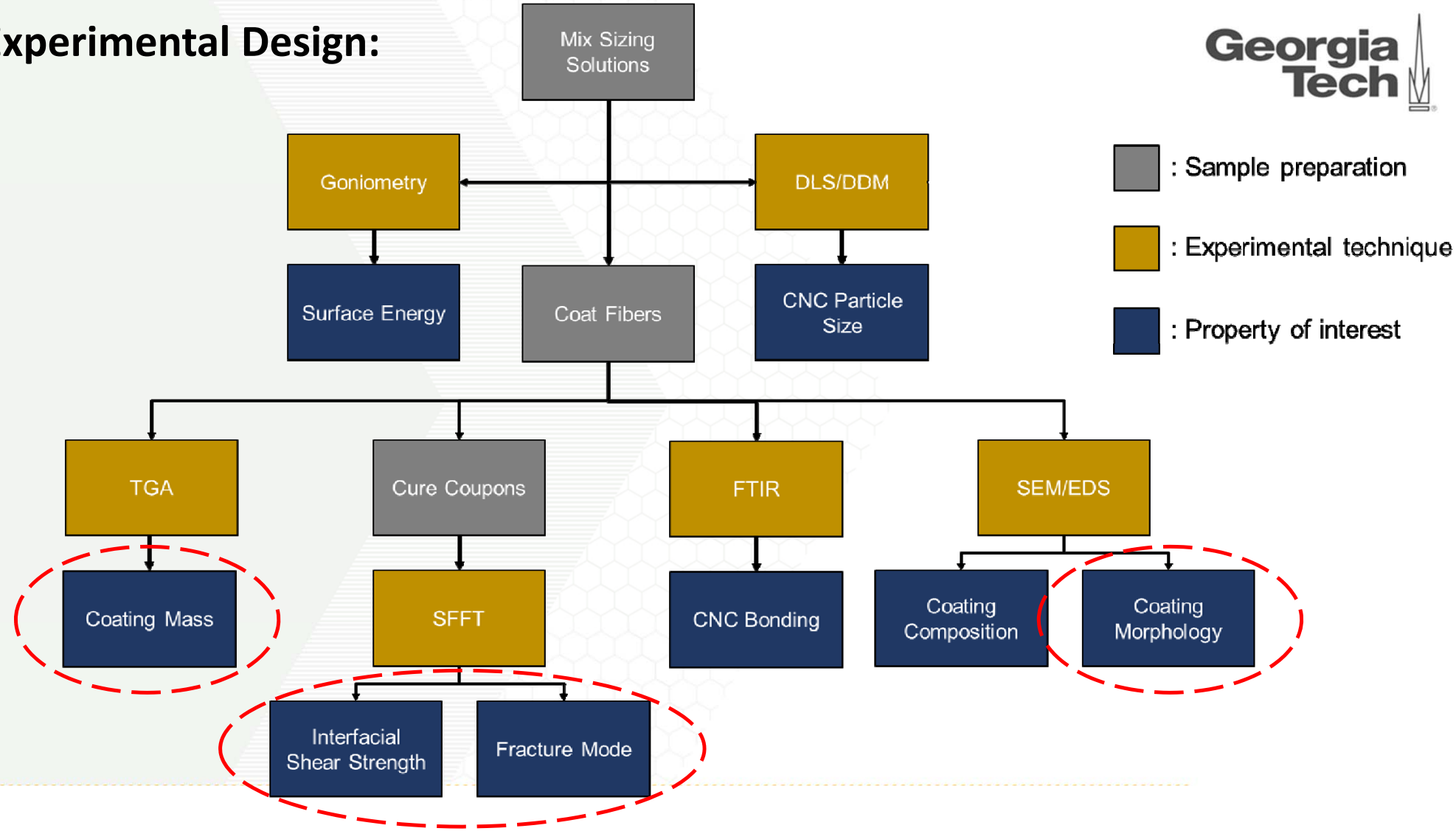


CNC-Ph (ion exchanged, 4% from Prof. D.M. Fox)

THREE-QUESTION APPROACH

1. Are CNC present on the fiber surface?
2. Do they improve composite properties?
3. If so, how?

Experimental Design:



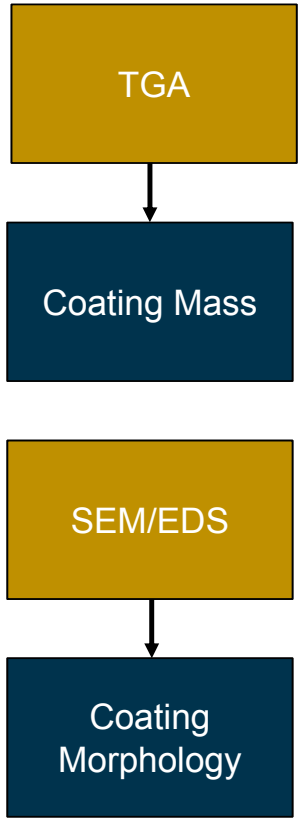
"ARE CNC PRESENT ON THE FIBER SURFACE?"

Thermogravimetric analysis (TGA)

- Thermally remove adsorbed coating
- Higher mass loss may indicate superior adhesion

Scanning electron microscopy (SEM)

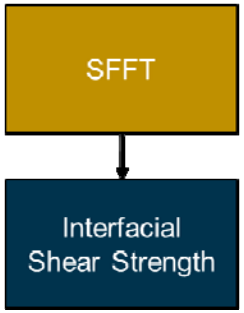
- Analysis on coated fibers before curing
- Resolve fine features (uniformity, porosity, etc.)

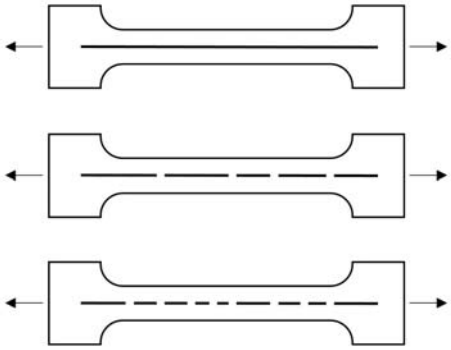


"DO THEY IMPROVE COMPOSITE PROPERTIES?"

Single fiber fragmentation test (SFFT)

- Coupons with single fibers subjected to tensile load
- Applied load is transferred from matrix to fiber
- Better fiber/matrix adhesion → more fractures
- Number of fractures related to IFSS



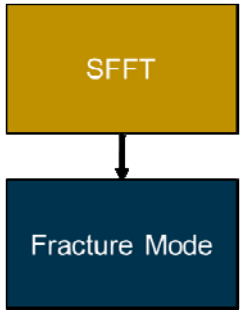


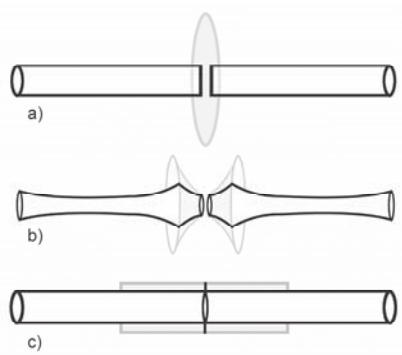
Fiber fractures with load

"IF SO, HOW?"

Optical microscopy performed after SFFT

- Allows for determination of fracture count
- Also allows for analysis of fracture geometry
- Fracture shape, birefringence, and debonding patterns are qualitatively related to adhesion

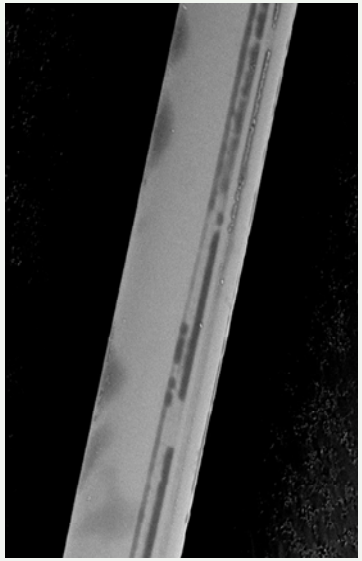




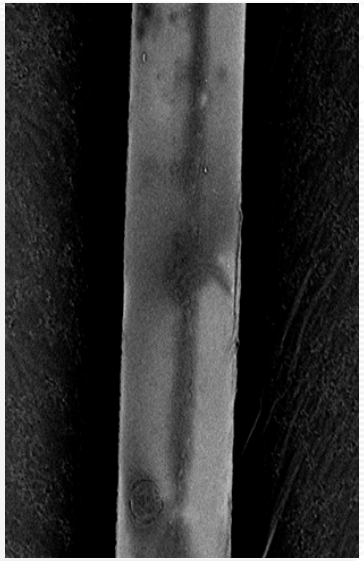
Types of fracture observed

Results

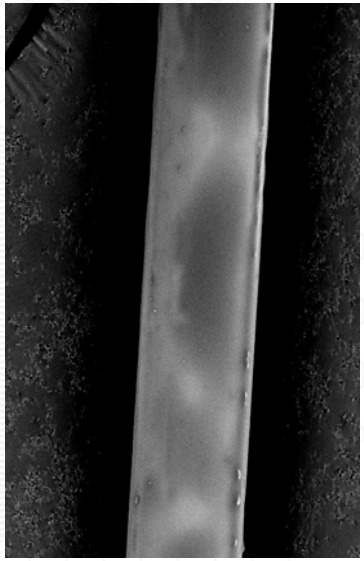
PRESENCE OF CNC – COATING MORPHOLOGY



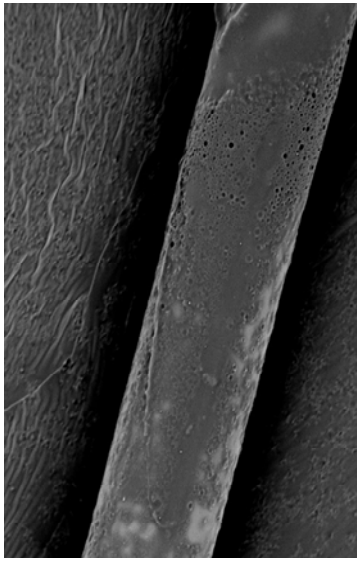
Control (no CNC)



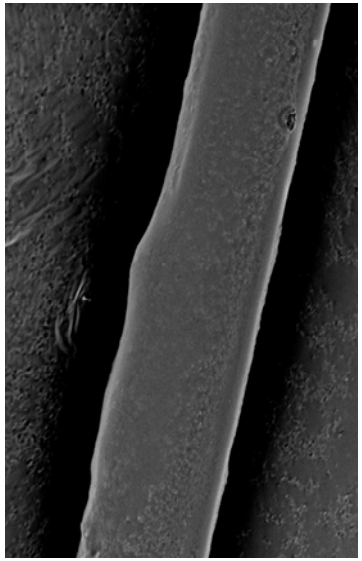
CNC-S in water



CNC-Ph in water



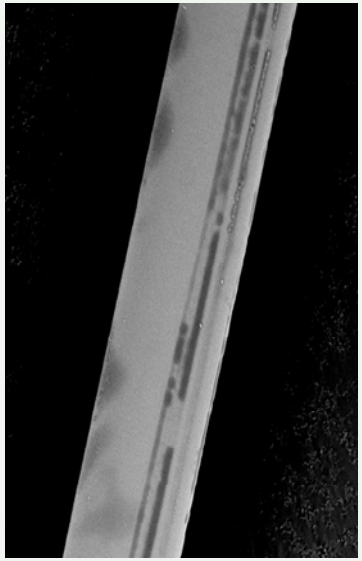
1 wt% CNC-S in commercial sizing



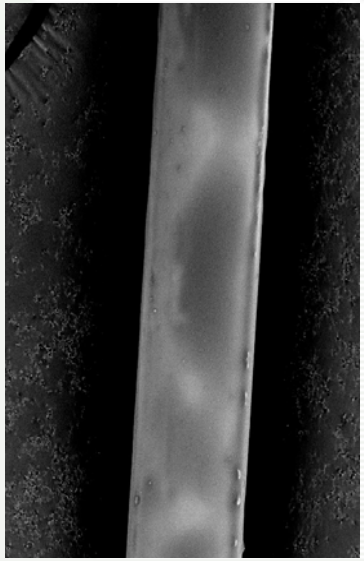
2 wt% CNC-S in commercial sizing

Fibers are 10 µm in diameter

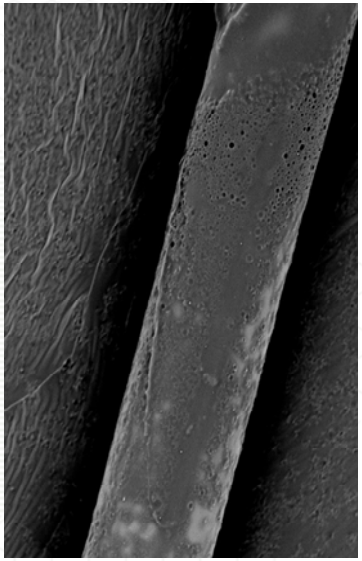
PRESENCE OF CNC – COATING ADSORPTION



Control (no CNC)



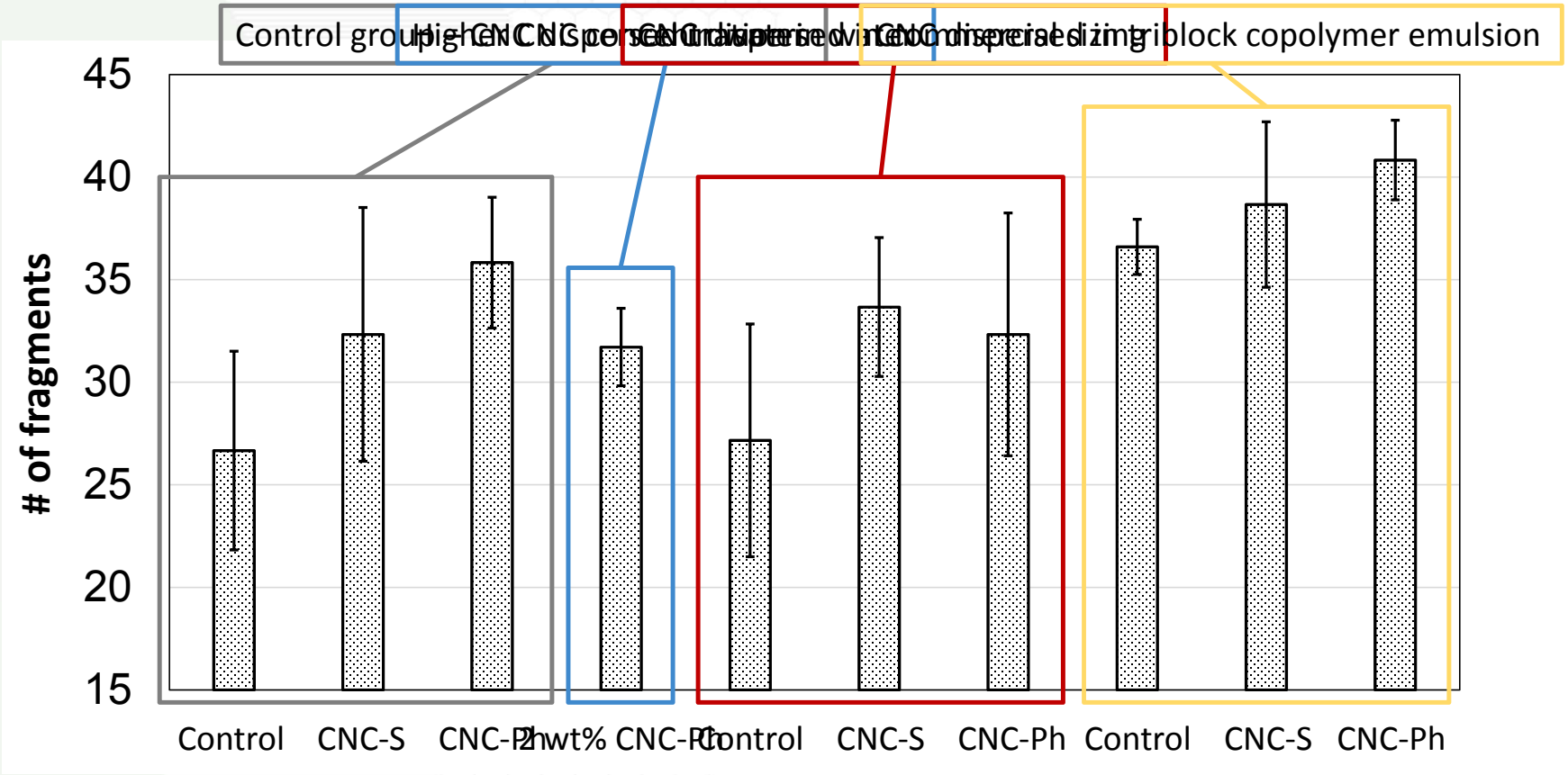
CNC-Ph in water



1 wt% CNC-S in commercial sizing

Fiber Type	Mass Loss (%)
As-received	0.75
CNC-Ph	2.15
Commercial Sizing + CNC	7.44

EFFECT OF CNC – SFFT FRAGMENT COUNT



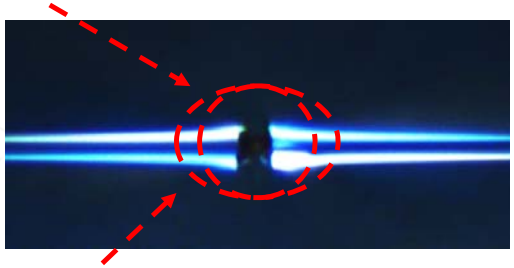
RESULTS – IFSS

Debonding + birefringence = weak interface

Fiber fracture

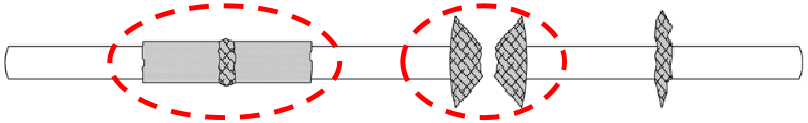
Double cone = moderately strong interface

Fiber



CNC-S

CNC-Ph



Possible fracture geometries

Concluding Remarks

CONCLUSIONS

1. CNC improve interfacial adhesion in fiberglass
2. Only 1 wt% in solution is needed to achieve improvement
3. CNC functionality may play important role in improvement mechanism
4. Polymeric dispersants produce more homogenous coatings without inhibiting effect of CNC

ACKNOWLEDGMENTS

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- P^3Nano
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- **Renewable Bioproducts Institute at Georgia Tech**

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- Prof. Douglas M. Fox (CNC-Ph)
- Michelman (commercial sizing)
- Prof. Satish Kumar
- Prof. Jon Colton

Thank you

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