A Comparison of All-Glass and Hybrid AGM

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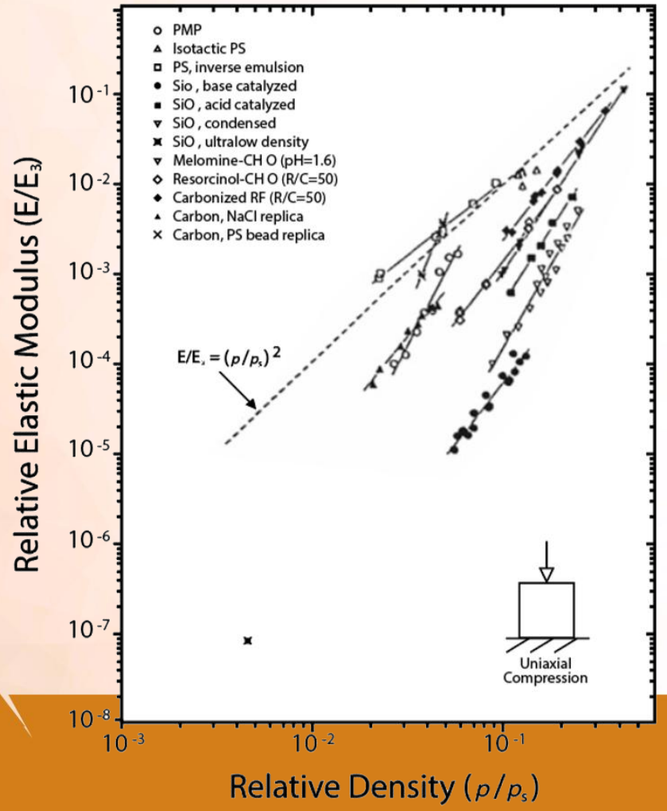
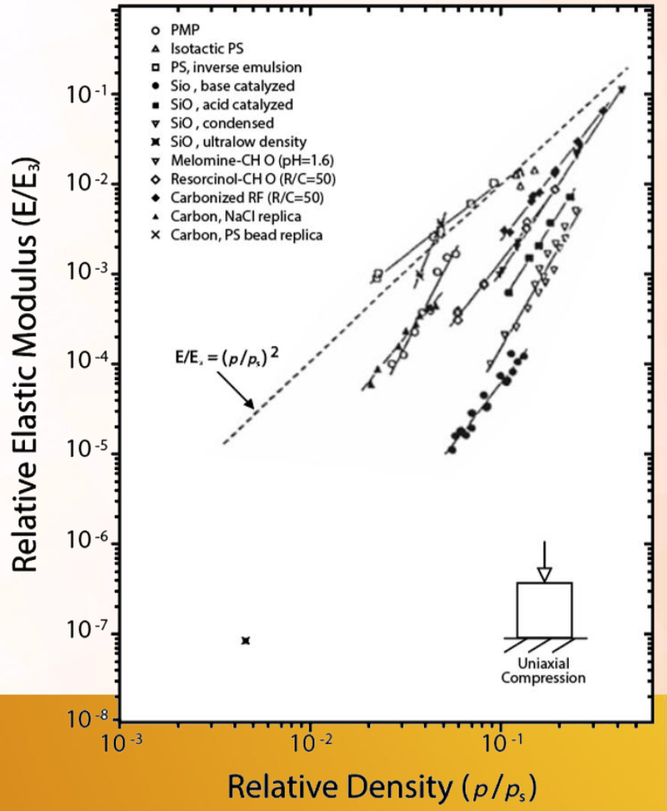
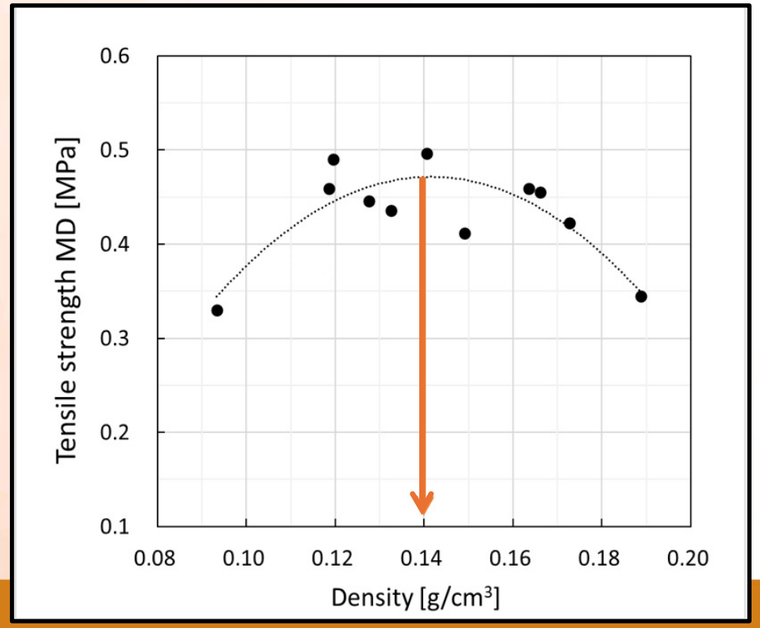
M. Otsubo, M. Onishi, C. La, E. Hostetler, C. Rogers, and R.W. Pekala

Porous Materials – Power Law Dependence of Mechanical Properties on Relative Density

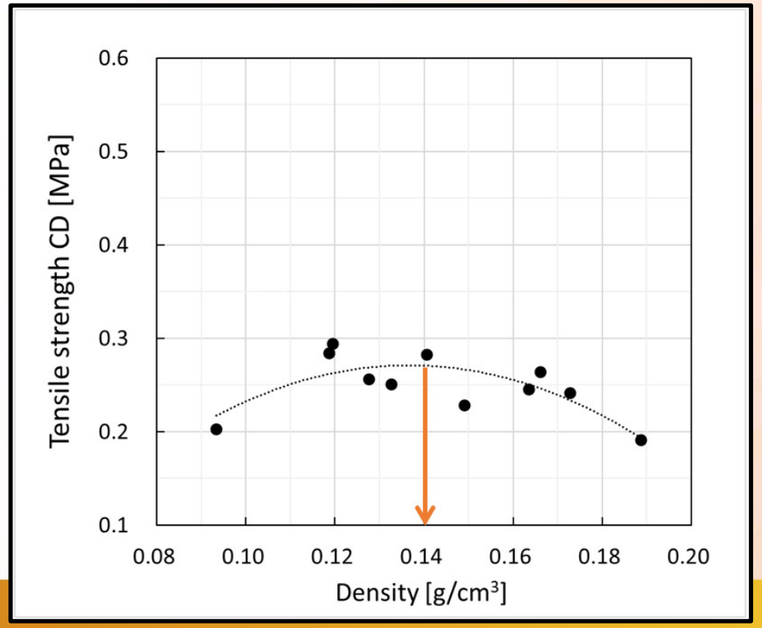




All Glass AGM Exhibits Maximum in Tensile Strength



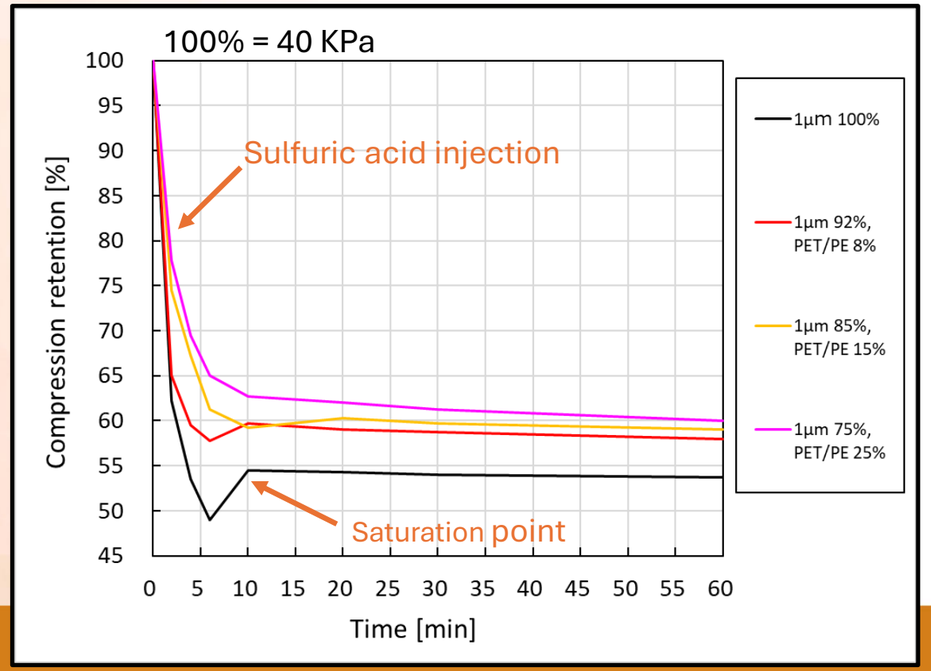
Single micro-glass fiber with a diameter of 1μm was used to make AGM with the same basis weight, but different densities (adjusted by wet press).



AGM Hybrids Produced on Pilot Line

Item		Unit	All glass AGM	Hybrid AGM						
Formu-lation	Glass fiber : 1μm	wt%	100	92	85	80	75	70	60	50
	PET/PE core-sheath fiber : 12μm		-	8	15	20	25	30	40	50
Basis weight		g/m^2	200	200						
Thickness, 10kPa		mm	1.33	1.33						
Density, 10kPa		g/cm^3	0.15	0.15						

Sulfuric Acid Injection and Compression Retention



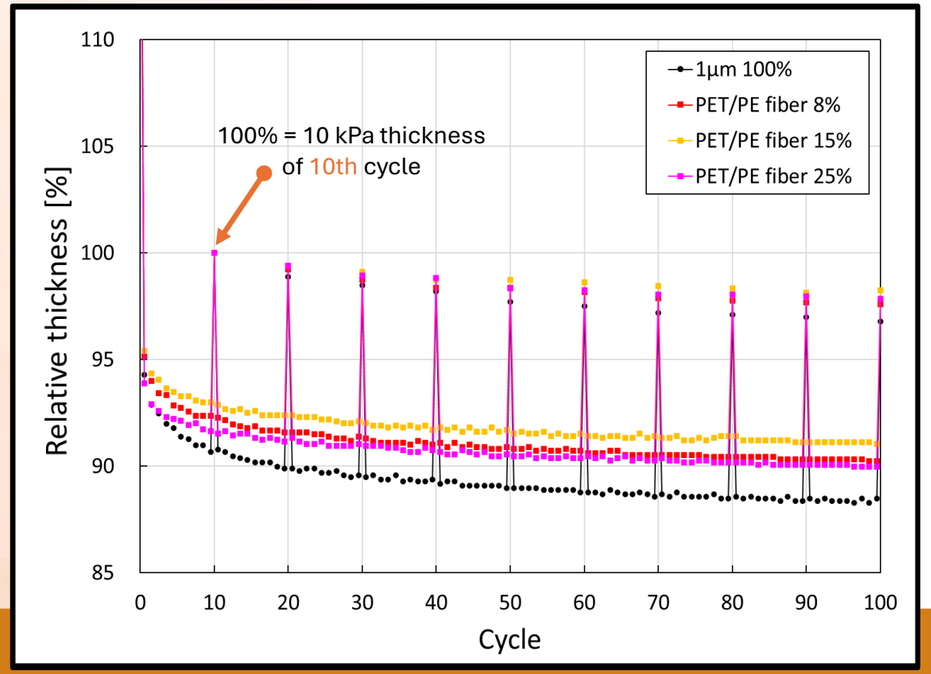
After compression to 40 kPa, the sulfuric acid is injected

Hybrid AGM samples maintain higher pressure

A slight decrease in pressure is still observed after the saturation point has been reached

After 60 minutes, the following mass loadings were determined
- 5.70 g/g for 1 µm 100%
- 5.81 g/g for PET/PE 8%
- 6.08 g/g for PET/PE 15%
- 5.68 g/g for PET/PE 25%.

Compression-Recovery Behavior in Wet State

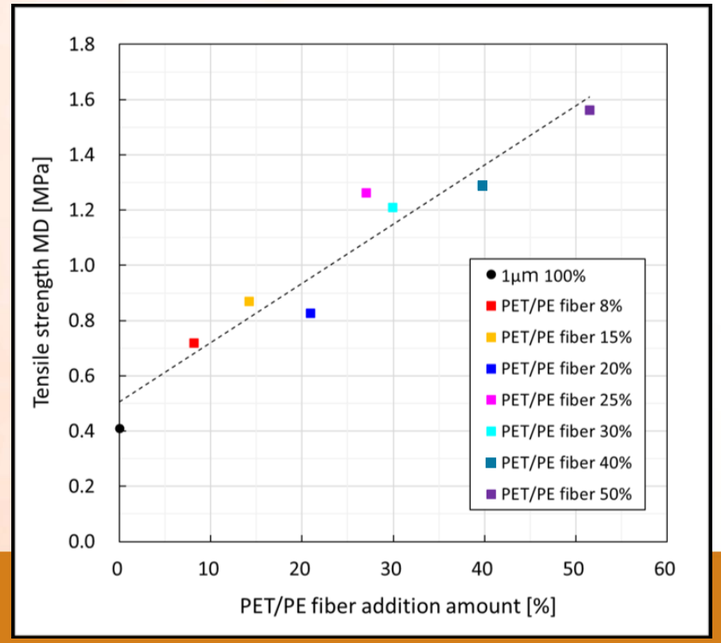


10 → 50 → 10 kPa ; 100 cycles

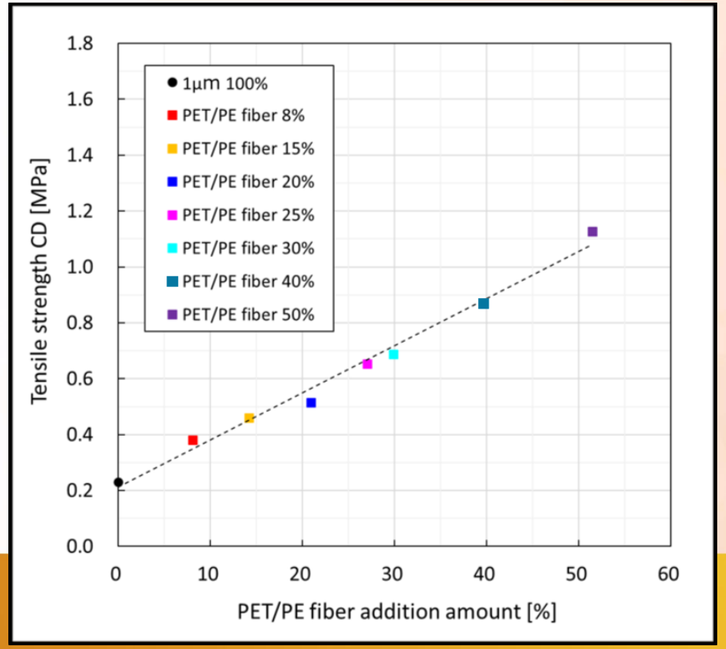
Hybrid AGM samples are significantly stiffer and exhibit less hysteresis (i.e., thickness reduction) as cycled at room temperature

Tensile Strength Increases with Organic Fiber Content

Machine Direction

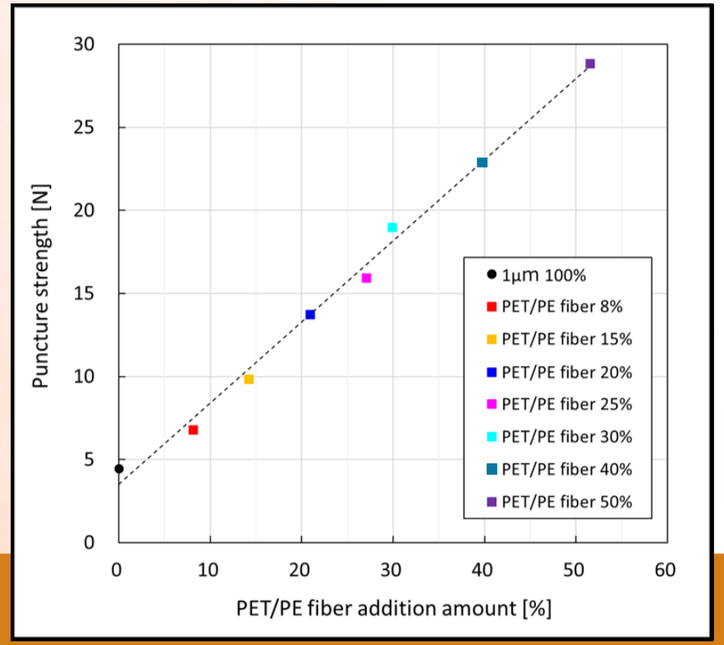


Cross Direction

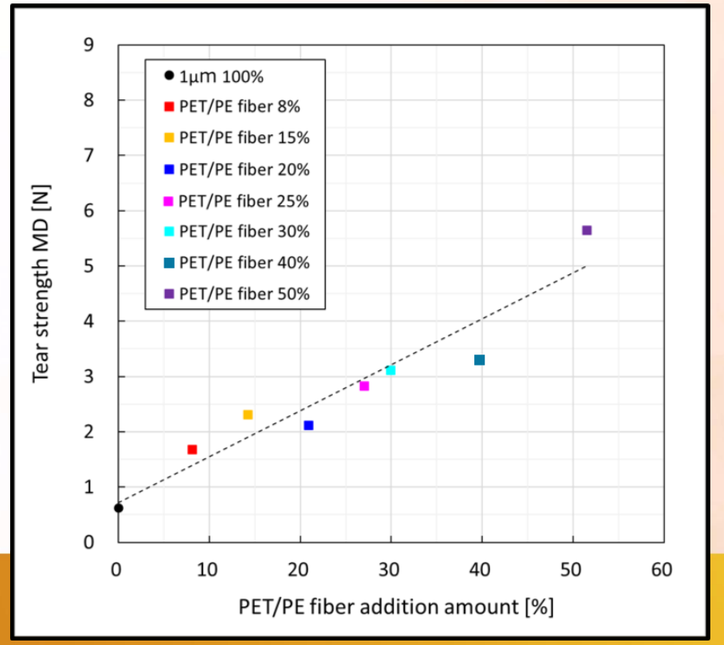


Puncture and Tear Strength Increase with Organic Fiber Content

Puncture Strength

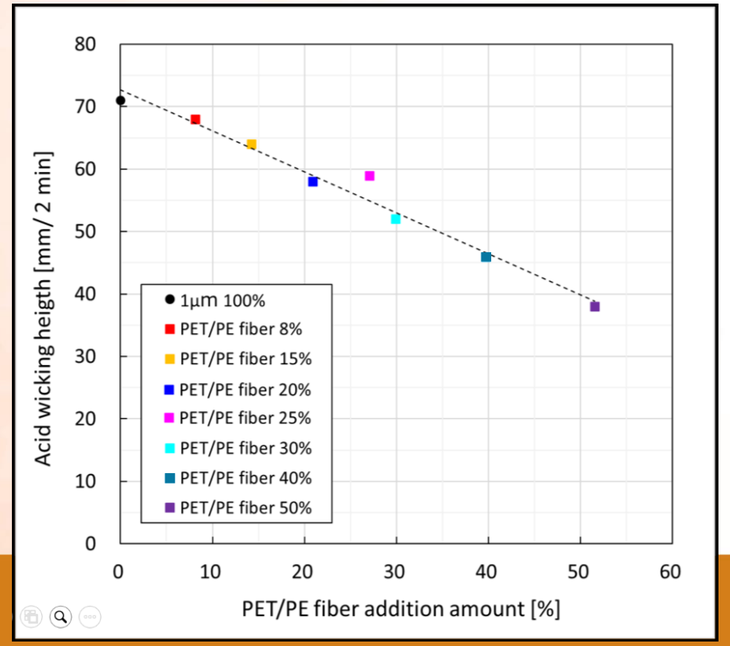


Machine Direction Tear Strength

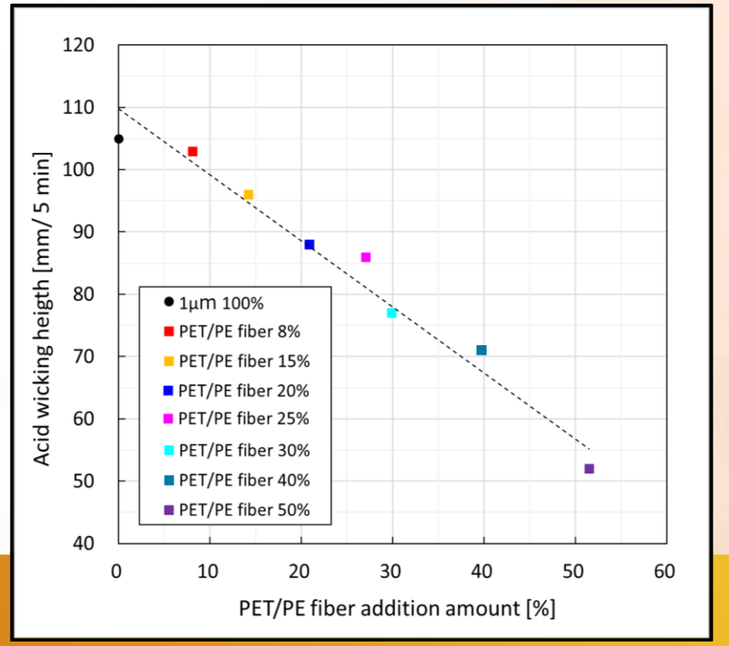


Organic Fiber Content Impacts Acid Wicking Rate

2 minute

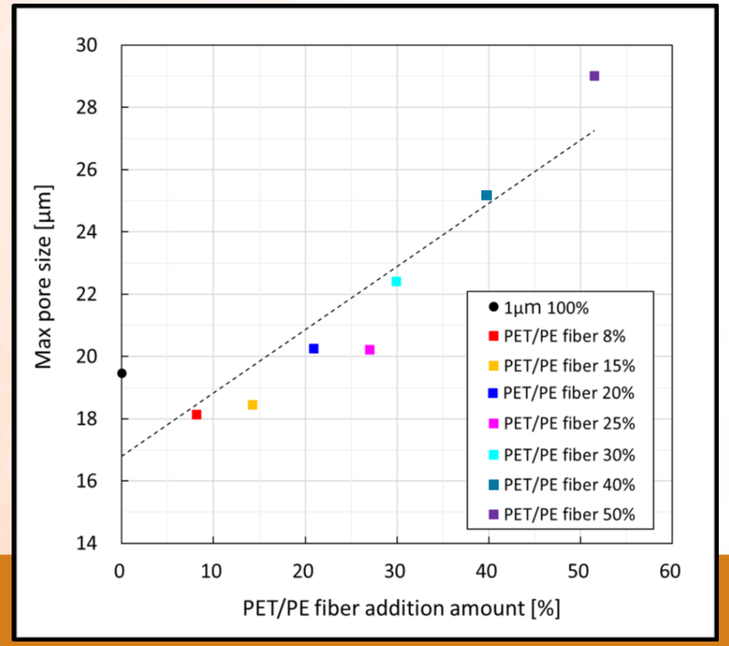


5 minute

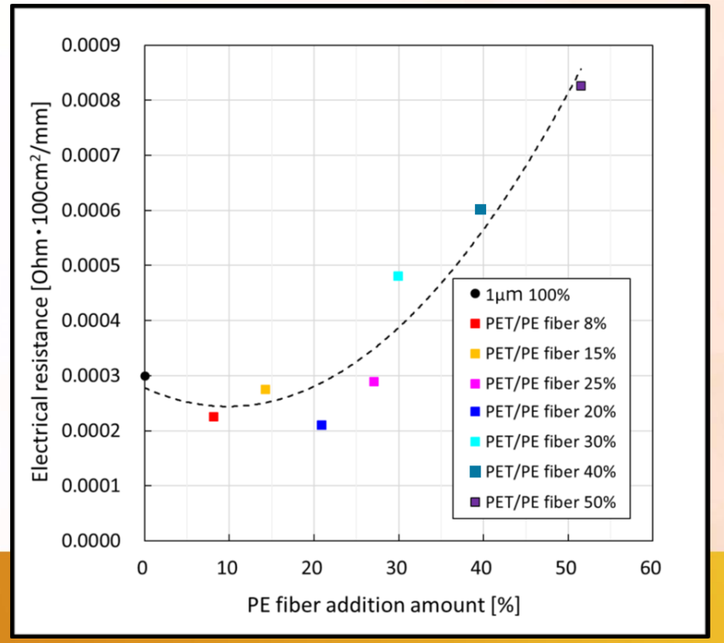


Organic Fiber Content Impacts Pore Size and Ionic Resistance

Bubble Point Max Pore Size

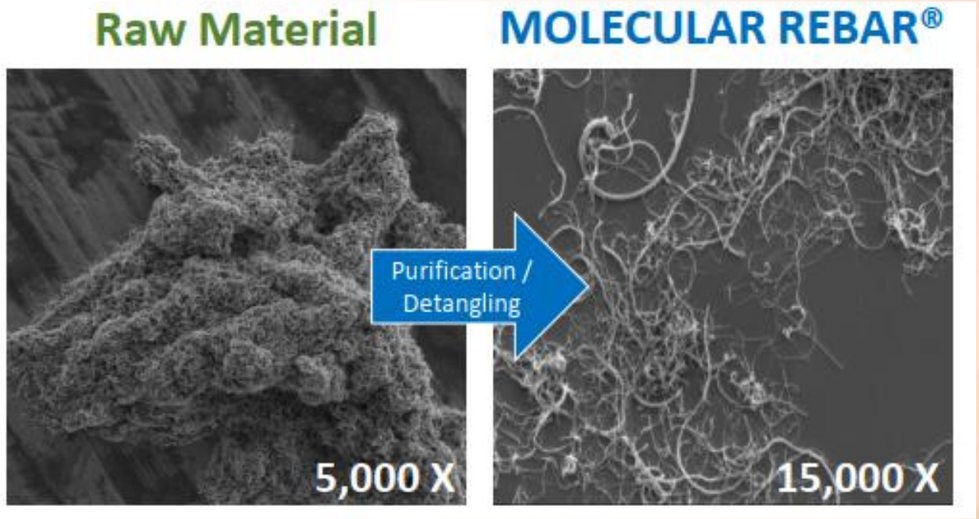


Ionic Resistance



Can Organic Fiber Diameter Impact Performance ?

Material	CNT
Product Name	MOLECULAR REBAR® Aqueous Dispersion
Part Number	Pb1330
Manufacturer	Black Diamond Structures
CNT Solid Content	3.06%
Total Solid Content	5.36%
Diameter of CNT	15 – 20 nm
Length of CNT	800 – 850 nm

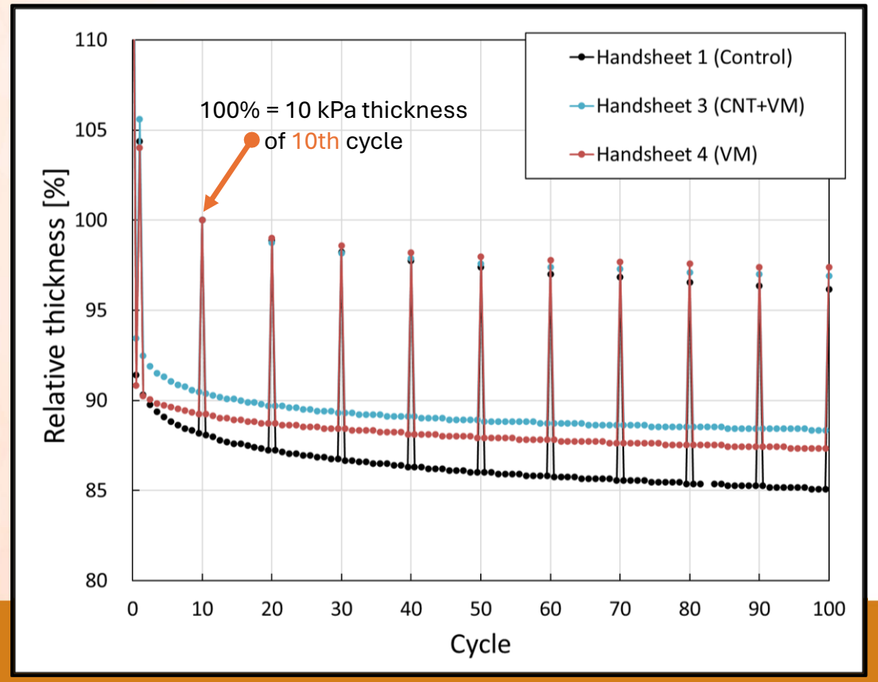


Handsheet Formulations

	Material	Solid Content (%)	Handsheet 1 (Control)	Handsheet 2 (CNT)	Handsheet 3 (CNT+VM)	Handsheet 4 (VM)
Input	Glass fiber	-	11.5 g	11.7 g	11.5 g	11.5 g
	CNT Dispersion	3.06%	-	6.0 g	6.0 g	-
	Viscosity Modifier (VM)	0.5%	-	-	25 g	25 g
Results	Thickness (mm)		1.28	1.31	1.38	1.09
	Grammage (g/m2)		191	194	216	204
	Adhesion Amount (%)		-	0.3%	3.0%	0.2%
	Loss on Ignition (%)		0.5%	-	3.5%	0.8%

A viscosity modifier (VM) was added to the fiber dispersion to prevent CNT removal from the bulk structure during drainage under vacuum

Compression-Recovery in Wet State

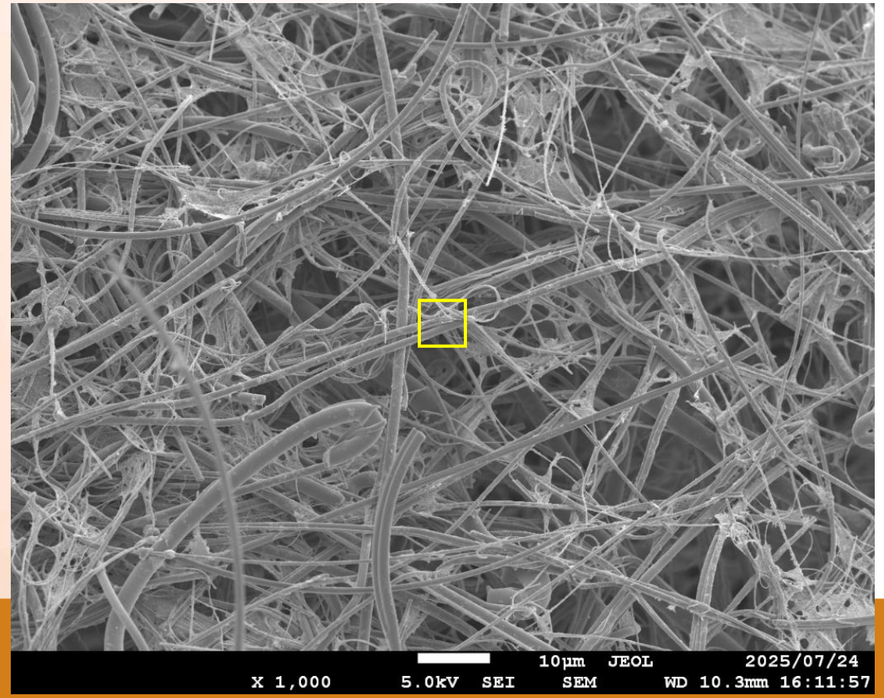


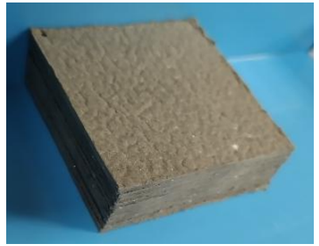
10 → 50 → 10 kPa ; 100 cycles

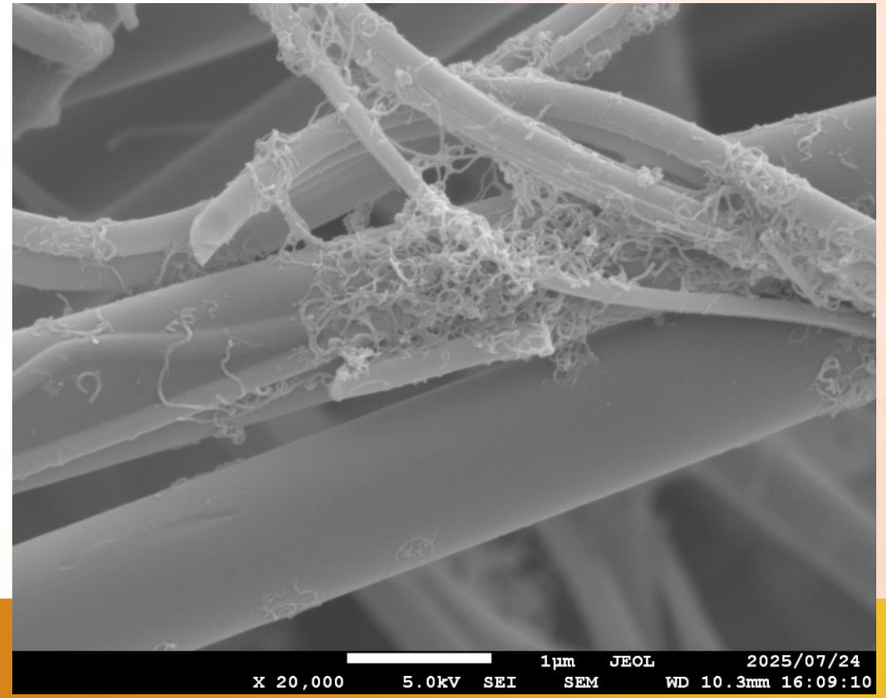
The VM-containing samples exhibit less hysteresis

	Handsheet 1 (Control)	Handsheet 3 (CNT+VM)	Handsheet 4 (VM)
Tensile Strength (MPa)	0.15	0.22	0.03
Elongation (%)	3.1	0.8	2.5

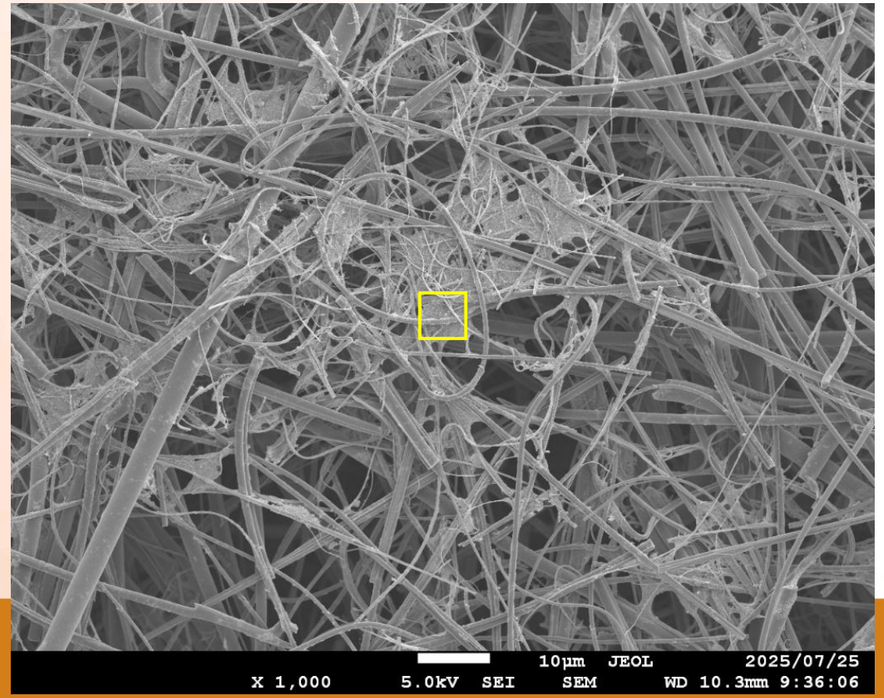
Surface SEM --- Handsheet 3 (CNT + VM)

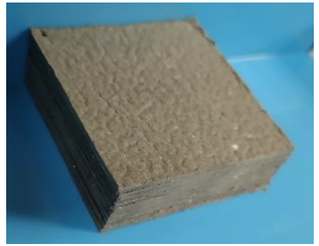


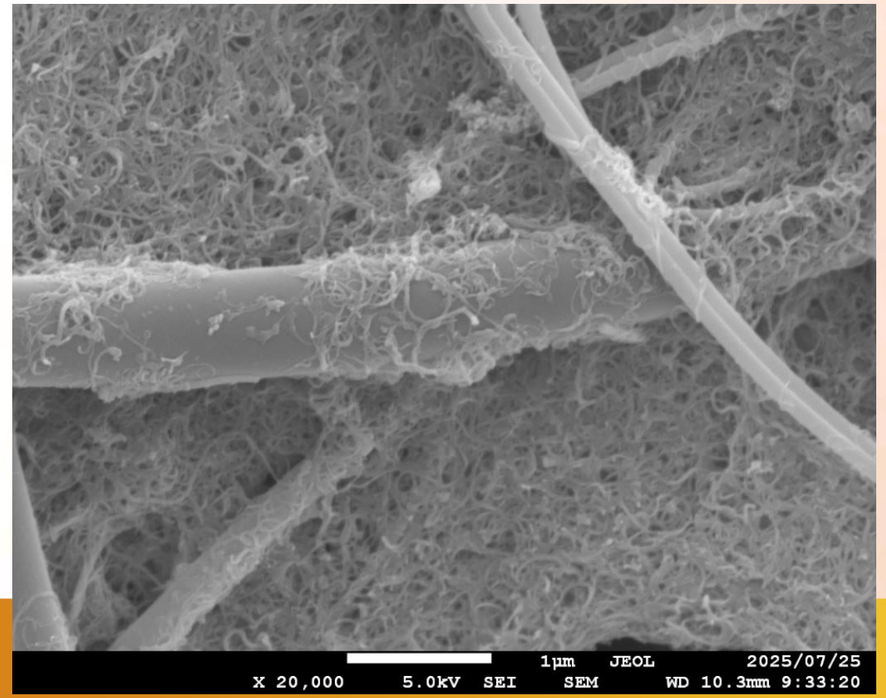




Fracture SEM --- Handsheet 3 (CNT + VM)







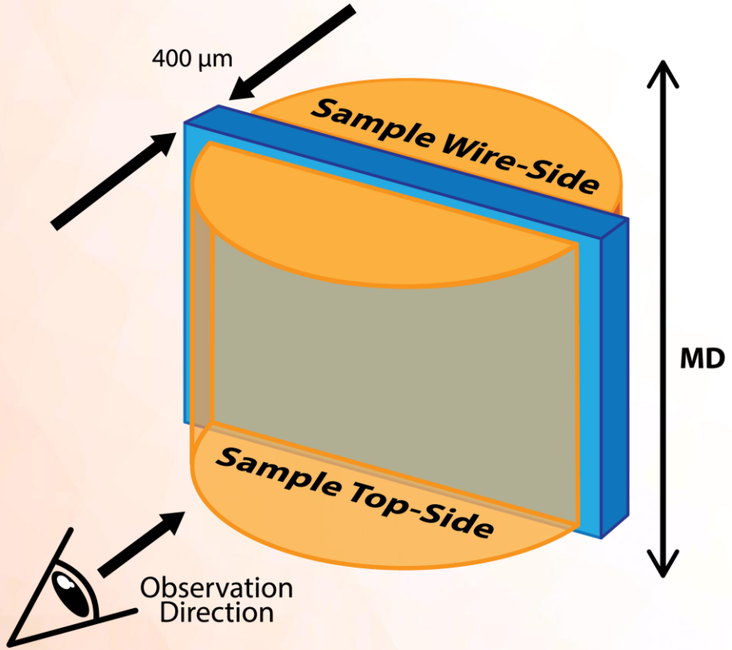
X-Ray Tomography --- Experimental Conditions

- Equipment — X-ray CT nano3DX (Rigaku)
- Location — Shizuoka Industrial Research Institute
- X-ray
  - Target — Cu
  - Voltage — 40 kV
  - Power — 1200W@40kV
- # of pics — 400 pics
- Condition — Wide and Narrow

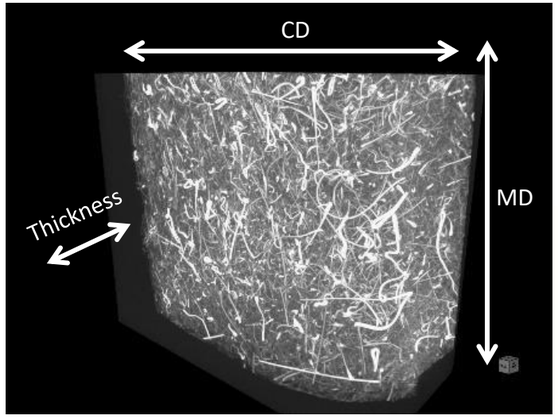
Lens Condition	Field of view	Voxel size
Wide view	3.6mm x 2.8mm	4.32 micron
Narrow view	0.9mm x 0.7mm	1.08 micron

Typical Sample Size
W x L x T = 2 mm x 7 mm x 1.4 mm

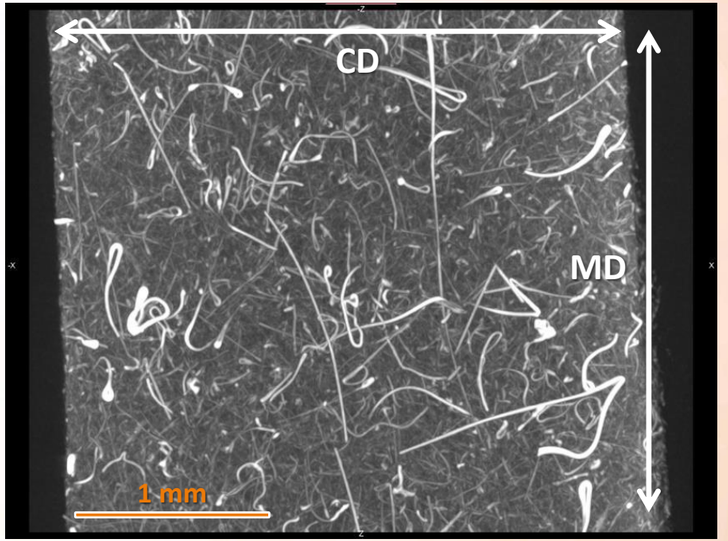
Analysis area – Front view



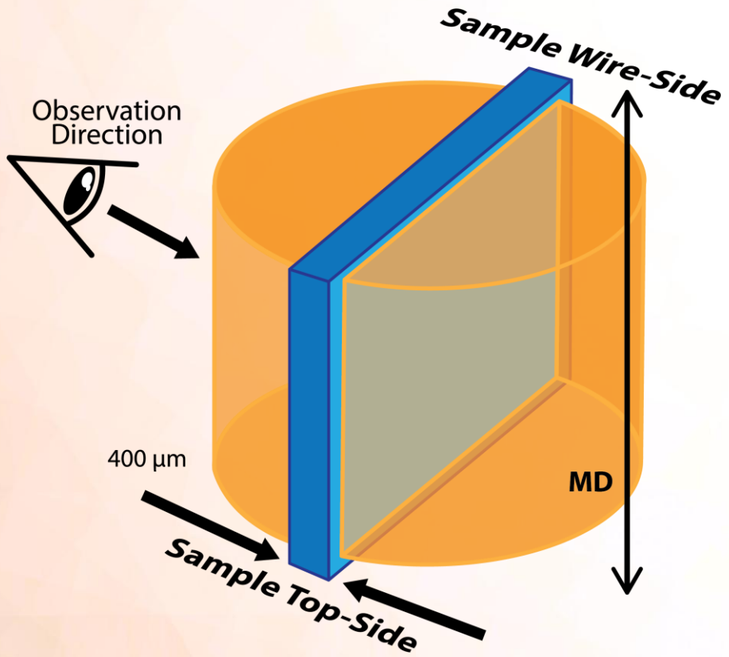
3D view



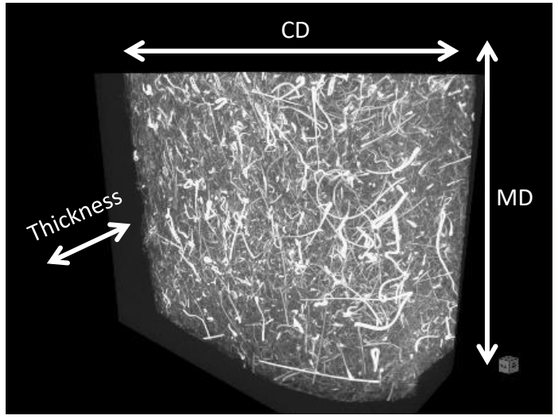
Front View



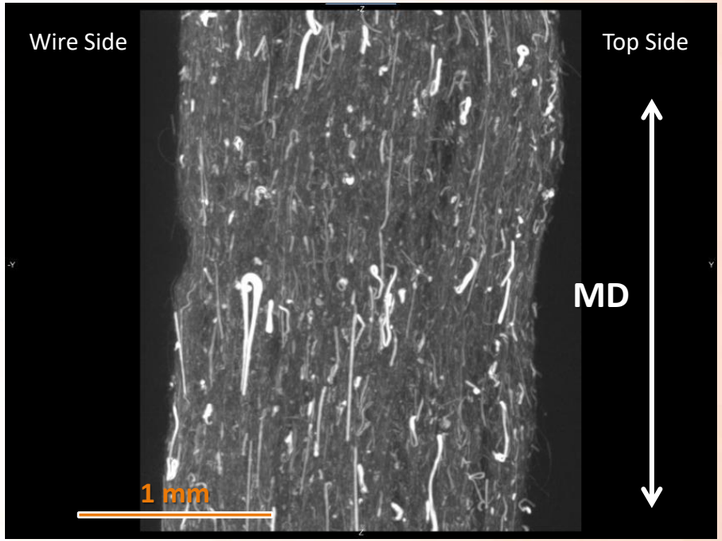
Analysis area – Side view



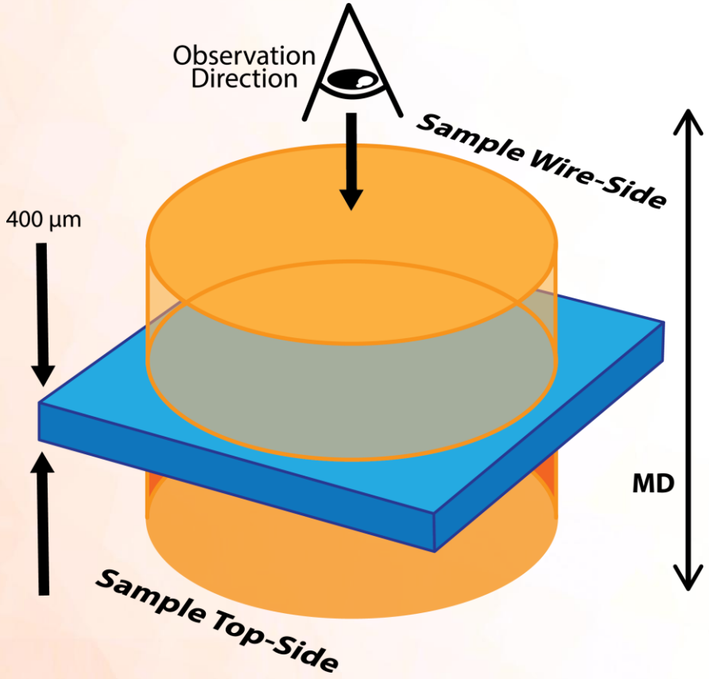
3D view



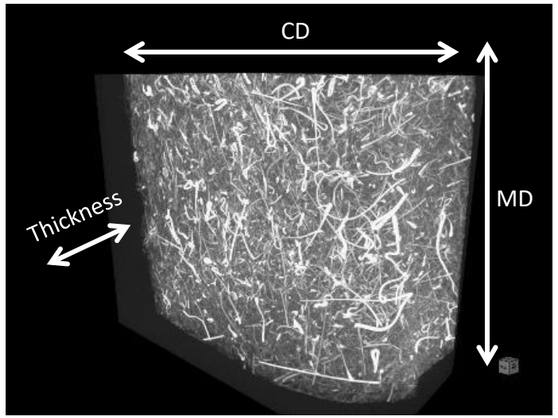
Side View



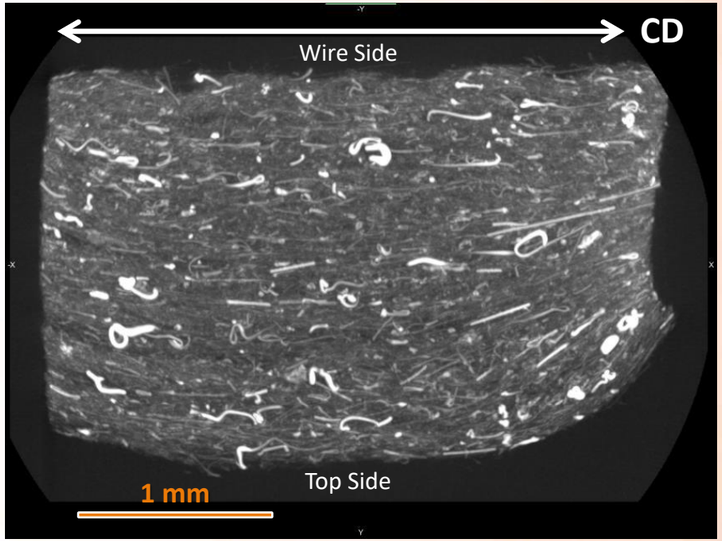
Analysis area – Top view



3D view

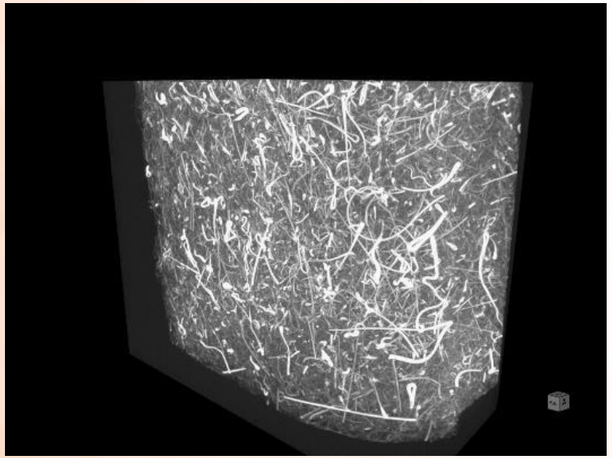


Top View



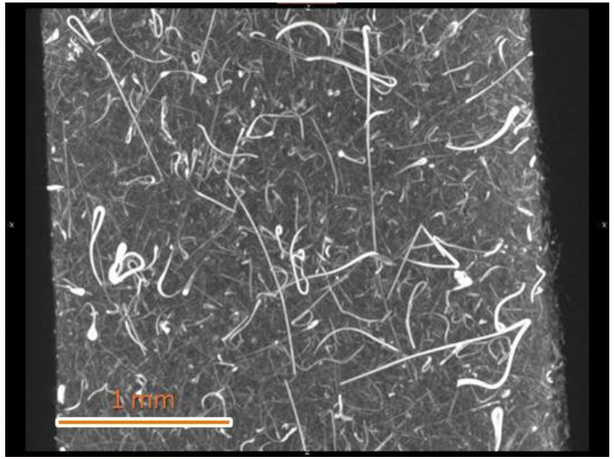
BMS-5 (ENTEK Asia; all glass fiber)

3D View

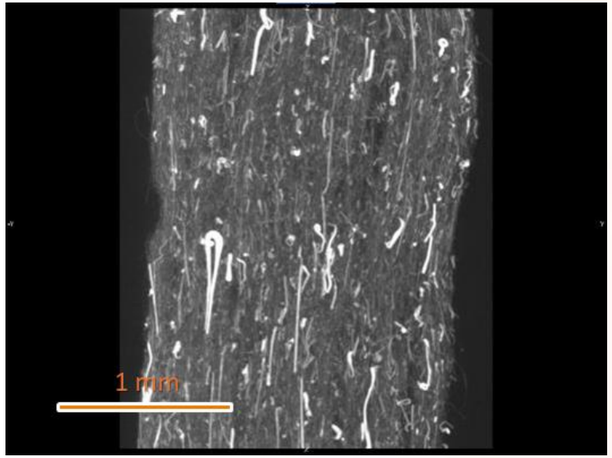


400 um slab view

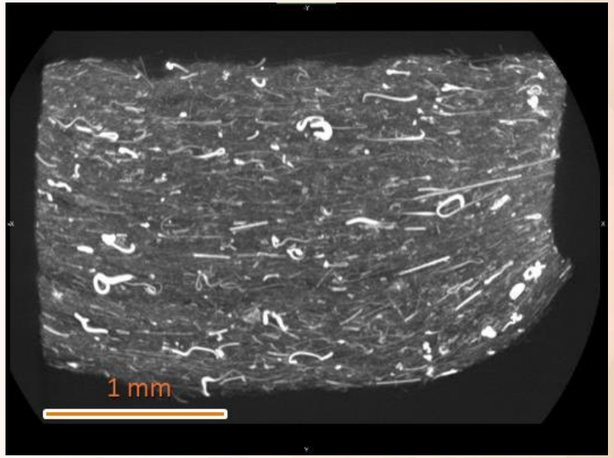
Front View



Side View

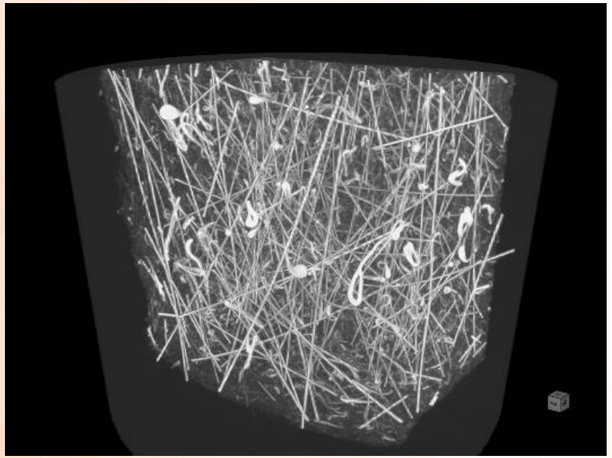


Top View



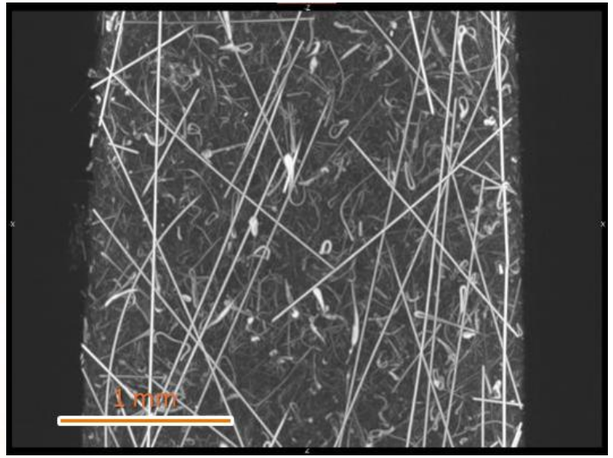
Competitor A

3D View

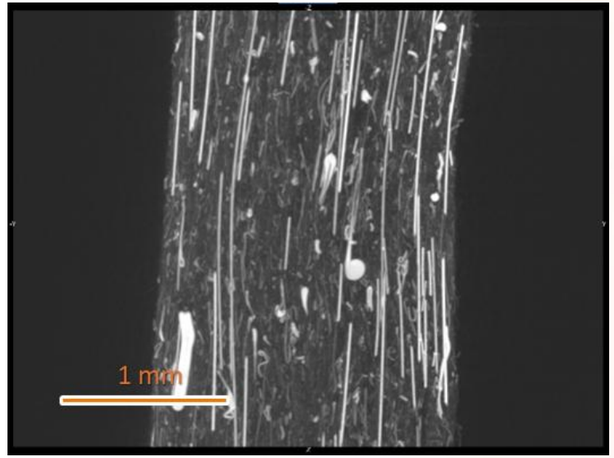


400 um slab view

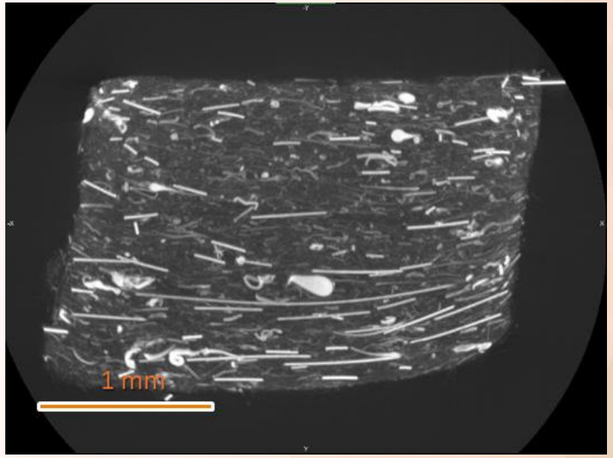
Front View



Side View

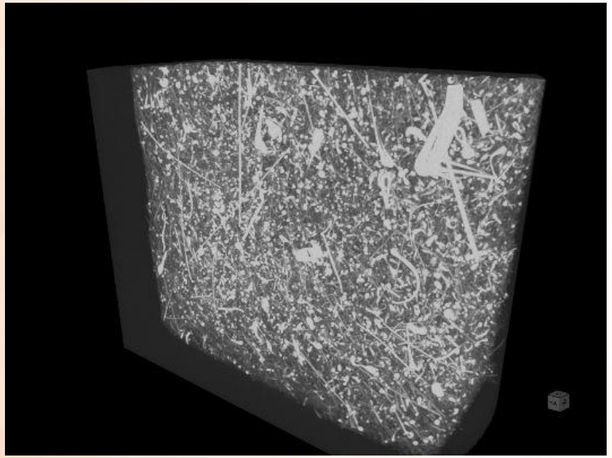


Top View

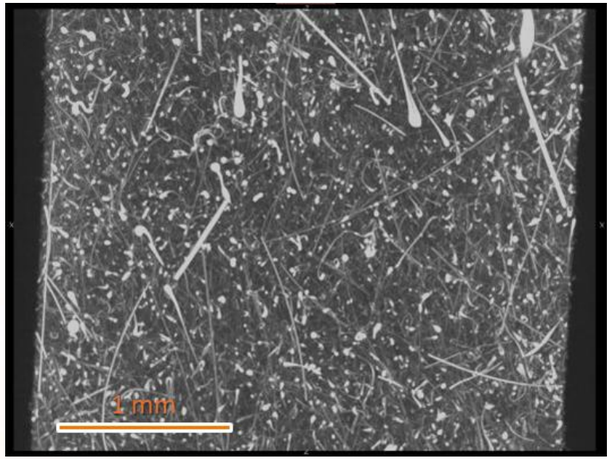


Competitor B

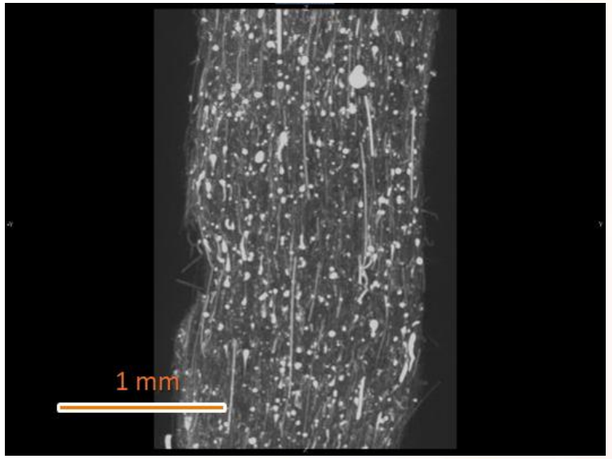
3D View



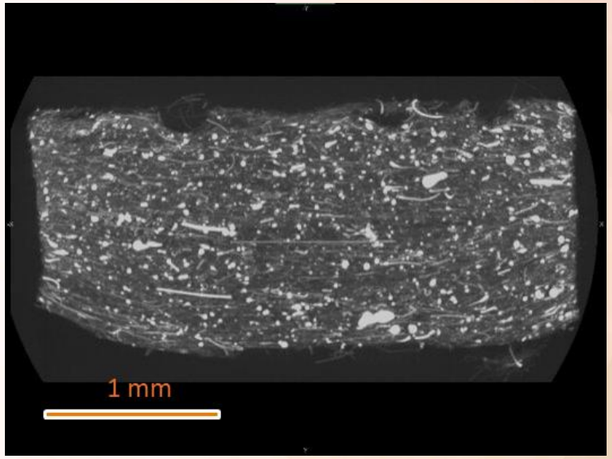
Front View



Side View

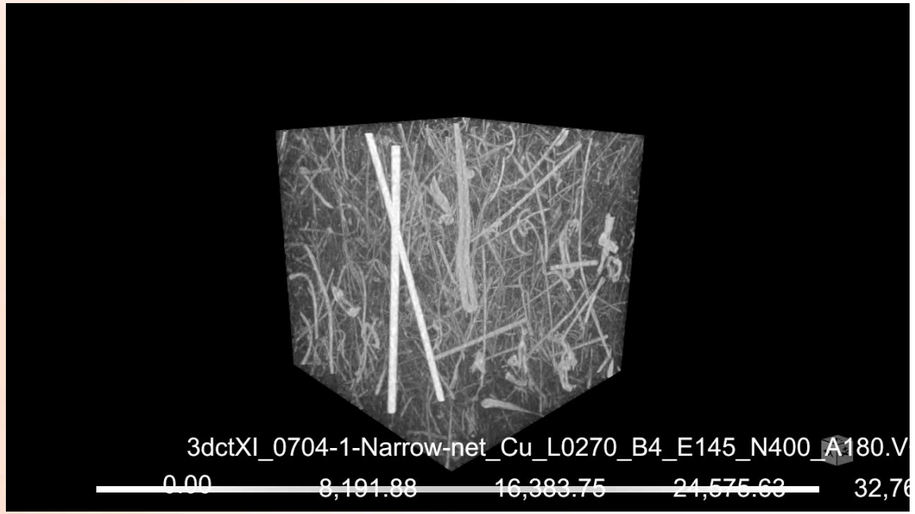


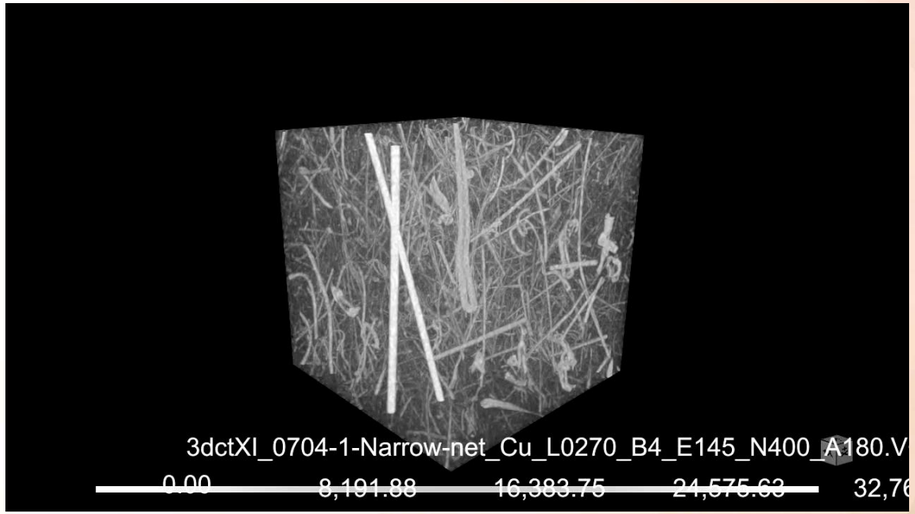
Top View



400 um slab view

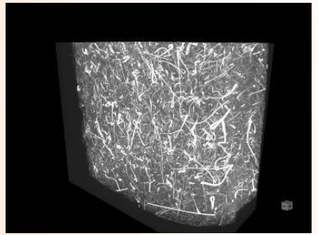
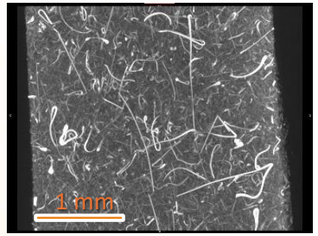
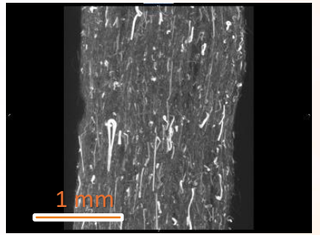
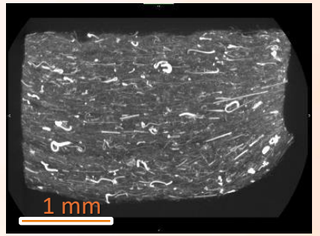
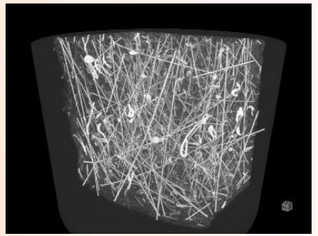
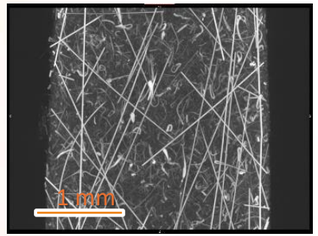
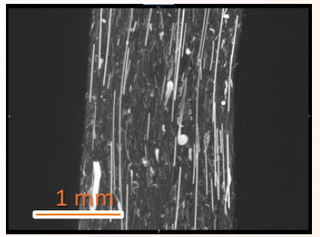
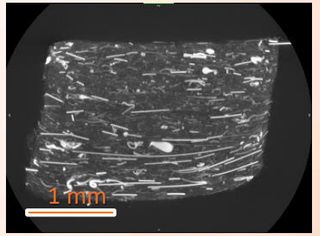
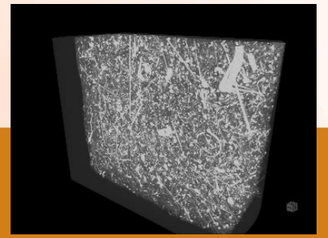
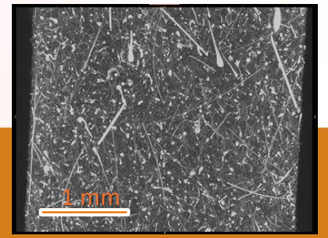
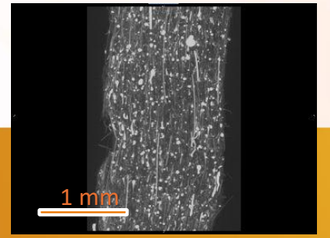
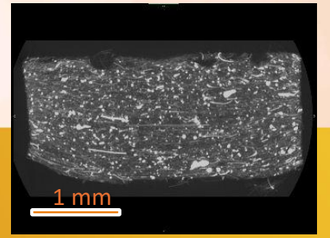
3 Dimensional Video





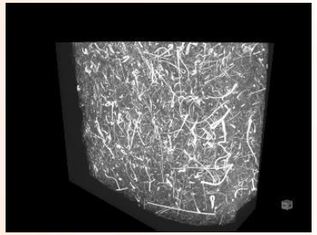
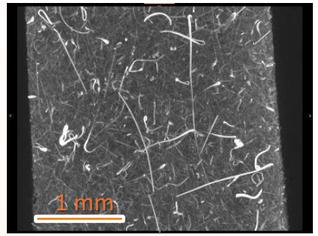
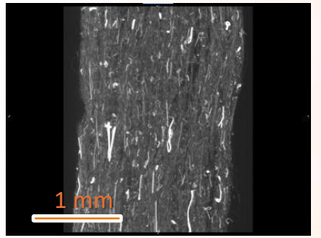
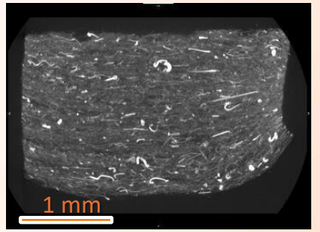
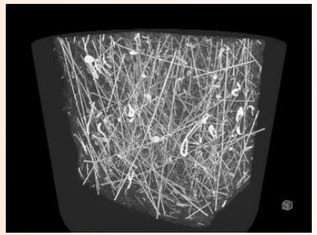
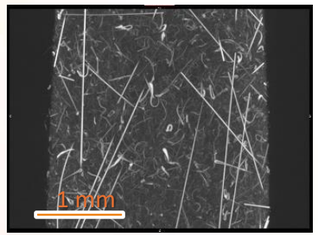
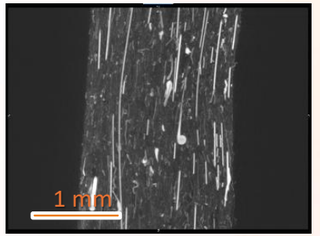
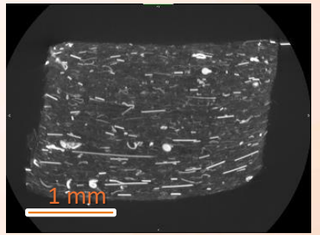
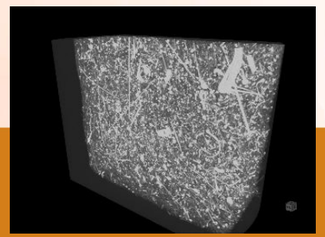
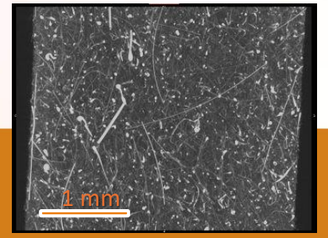
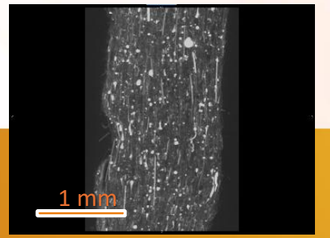
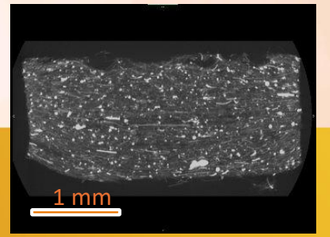
Wide view comparison

400um slab thickness

3D View	Front View		Side View	Top View
		BMS-5		
		Competitor A		
		Competitor B		

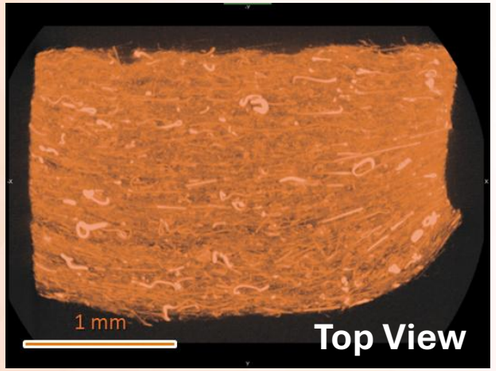
Wide view comparison

200um slab thickness

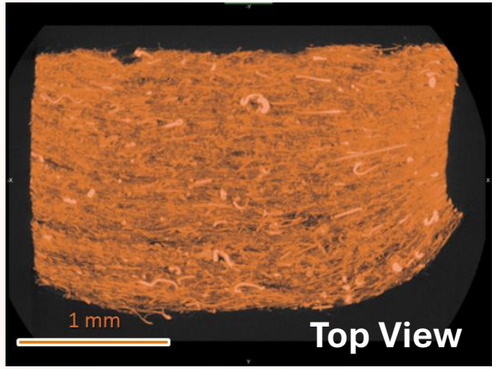
3D View	Front View		Side View	Top View
		BMS-5	BMS-5 	
		Competitor A	Competitor A 	
		Competitor B	Competitor B 	

Pores Are Easier to Visualize at Thinner Slab Thickness

400um slab

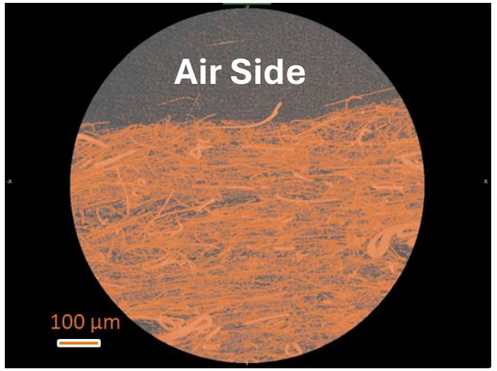


200um slab

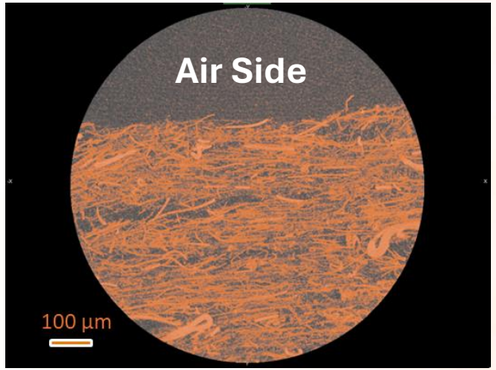


BMS-5 Wide Views

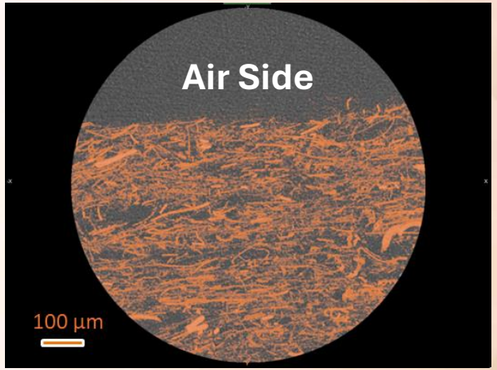
200um slab



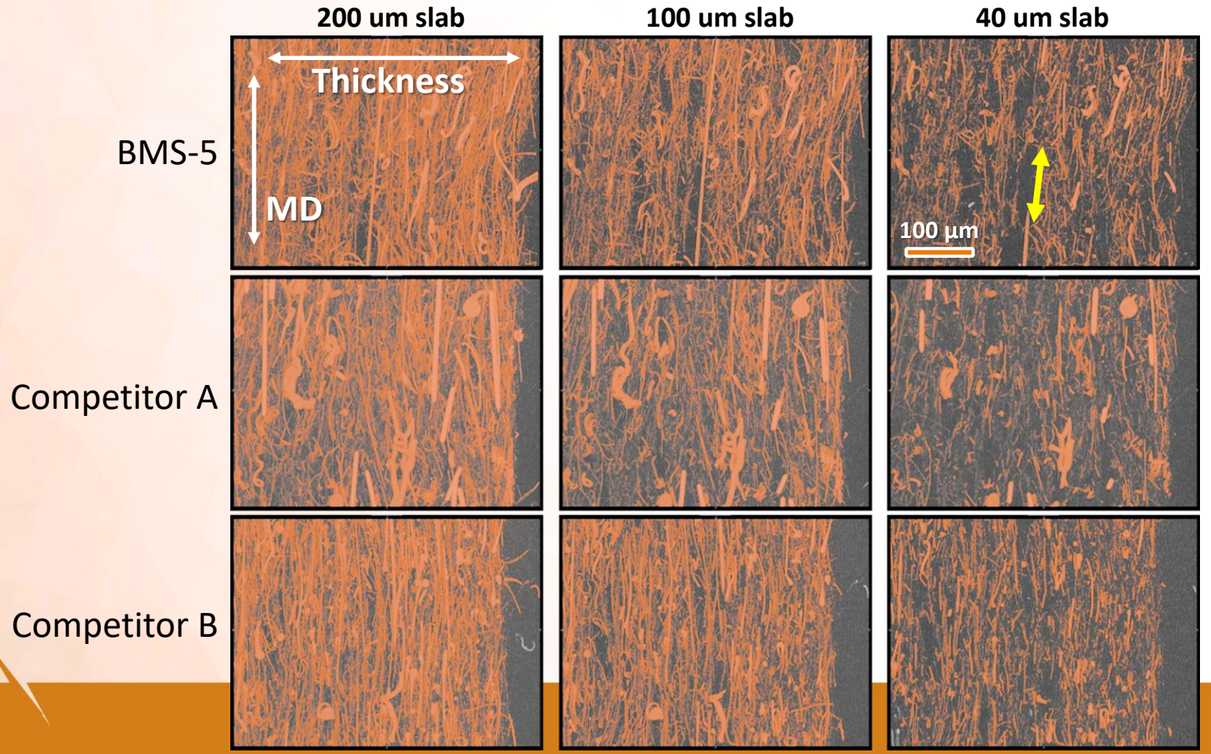
100um slab



40um slab



BMS-5 Narrow Views

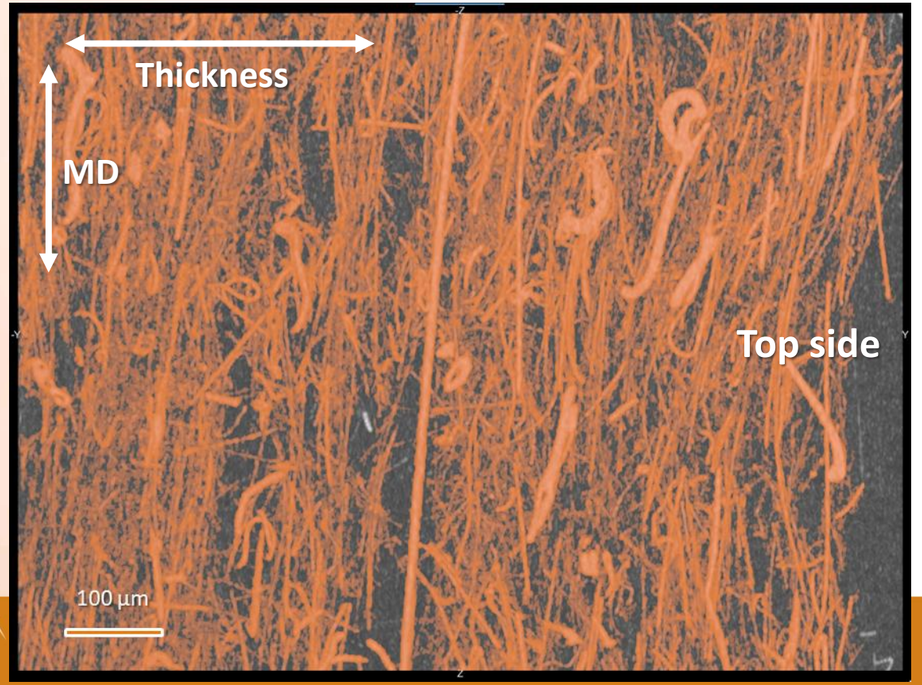


AGM Image Comparison at Thinner Slab Thickness

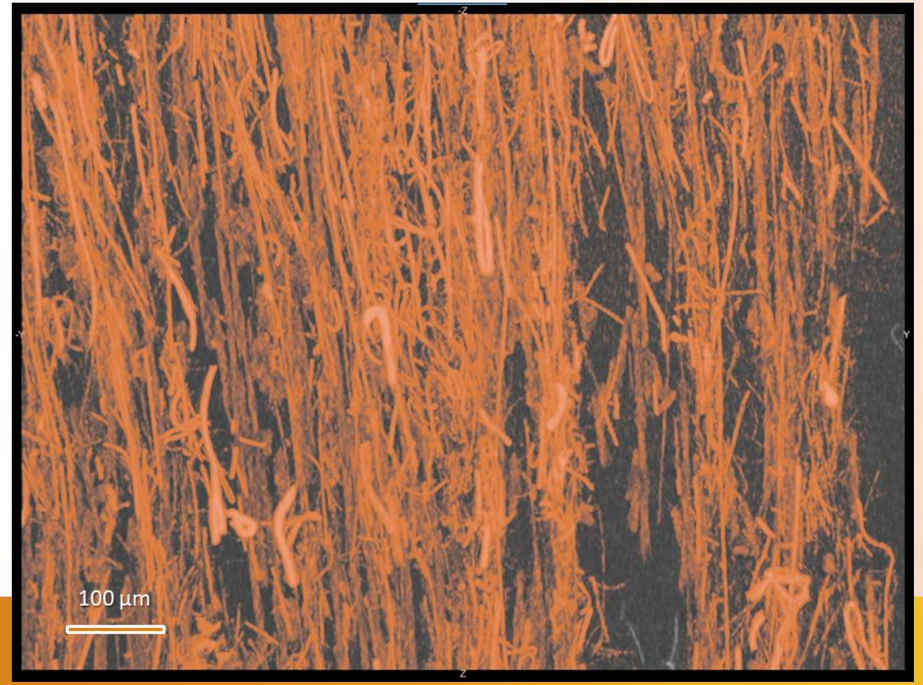
All Glass vs Hybrid AGM

100 um slab view

All glass AGM



Hybrid AGM



Summary

- AGM does not follow the power-law dependence of mechanical properties on relative density that is observed in other porous materials
- AGM exhibits inelastic behavior and a high degree of hysteresis in “wet” compression-recovery experiments
- The tensile, puncture, and tear strength of hybrid AGM increased with organic PET/PE fiber content
- A good balance of improved mechanical properties, fast wicking rate, and low ionic resistance was achieved with PET/PE fiber loading up to 15 weight %.
- Carbon nanotubes were successfully incorporated into AGM but did not dramatically improve key properties
- X-ray tomography shows promise as a non-destructive tool for investigating the fiber and pore structure of AGM.

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