

# Reinforced Composite Material Research

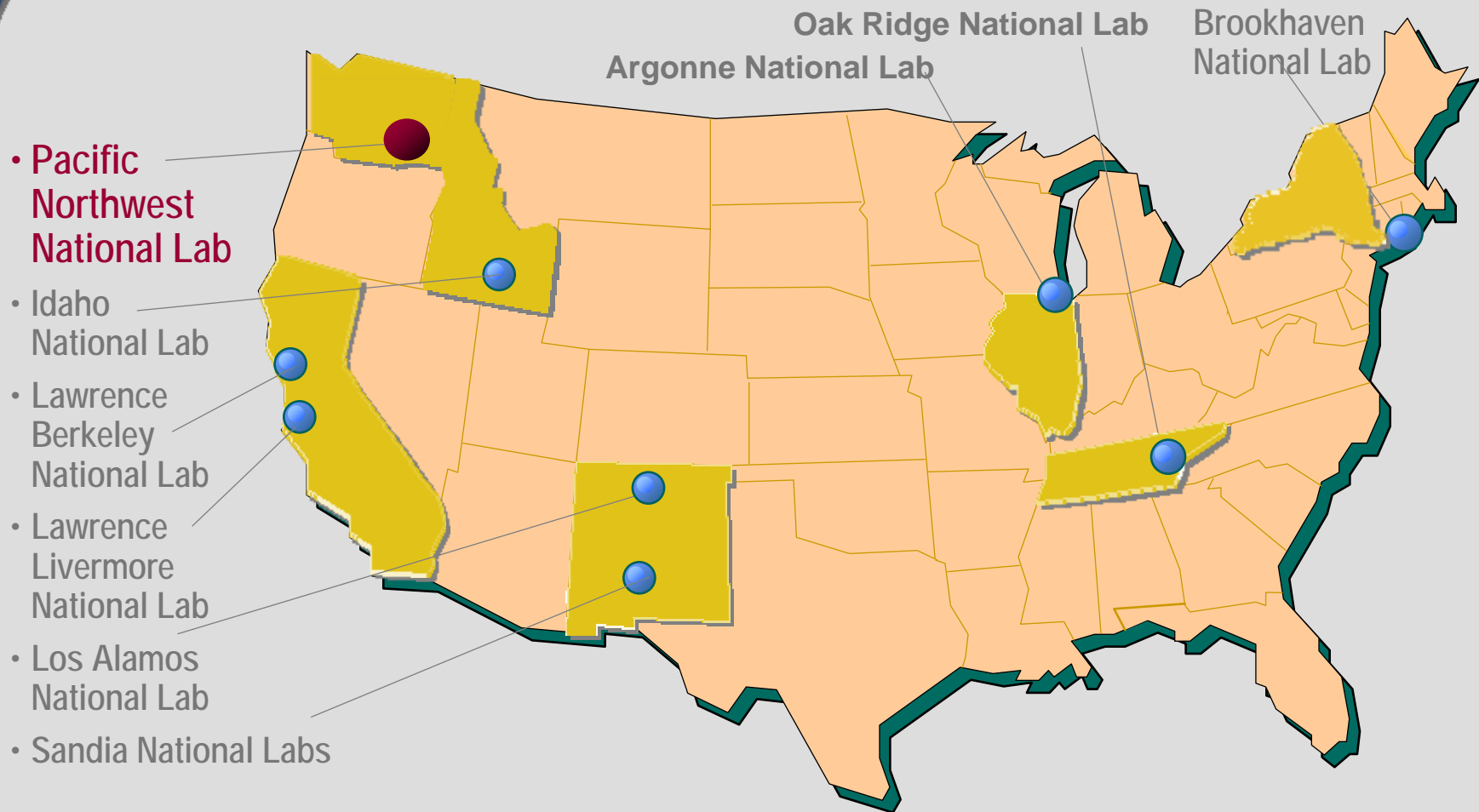
## Energy & Environment Directorate

Jim Holbery, Kevin Simmons, Nghiep Nguyen,  
Ken Johnson, Siva Pilli, Dan Howe, Naveen Karri,  
Leo Fifield, Cheng Huang



Pacific Northwest  
National Laboratory  
Operated by Battelle for the  
U.S. Department of Energy

# Washington is one of only 7 states to host a multi-program DOE national lab



# PNNL Material Research

## Materials R&D personnel at PNNL

- Approximately 300 full-time, 150 PhDs
- Polymer Composites - 8 full-time researchers, 3 PhD

## Composite Processing Lab

- RTM, Inj. Mold. – 40 ton Arburg, compression presses, extrusion (twin and single screw), Haake 65-150cc rheometer, powder coater, etc.

## Polymer/Ceramic Chemistry Labs

- Reactors, casting, colloidal science, furnaces, etc.
- CombiCat Lab: Three 96-well (~2 mL each) robotic systems for synthesis – solution makeup, solids loading, GC, HPLC integrated

## Analytical Capabilities

- DSC, DMA, TMA, TGA, DTA, DSC, SEM-ESEM, FIB-TEM, FTIR-ATR, NMR, GC-MS, AFM, STM, XPS-SIMS, Nanoindenter, etc.



# 1. Engineering Property Prediction – Long Fiber Thermoplastics

TEAM: PNNL, ORNL, U-Illinois, Moldflow Corp., Ford/GM/DC (\$4M/4 Years)

## ► Develop Long Fiber Injection Molded Fiber Orientation Models

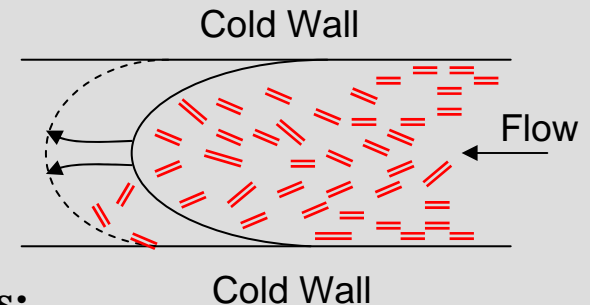
- Fiber length range 3~10 mm
- Geometrical restrictions on fiber motion
- Interaction between fibers and fiber domains: fibers organized in domains and locally aligned
- Wall effect may dominate the orientation behavior

## ► Extend current models

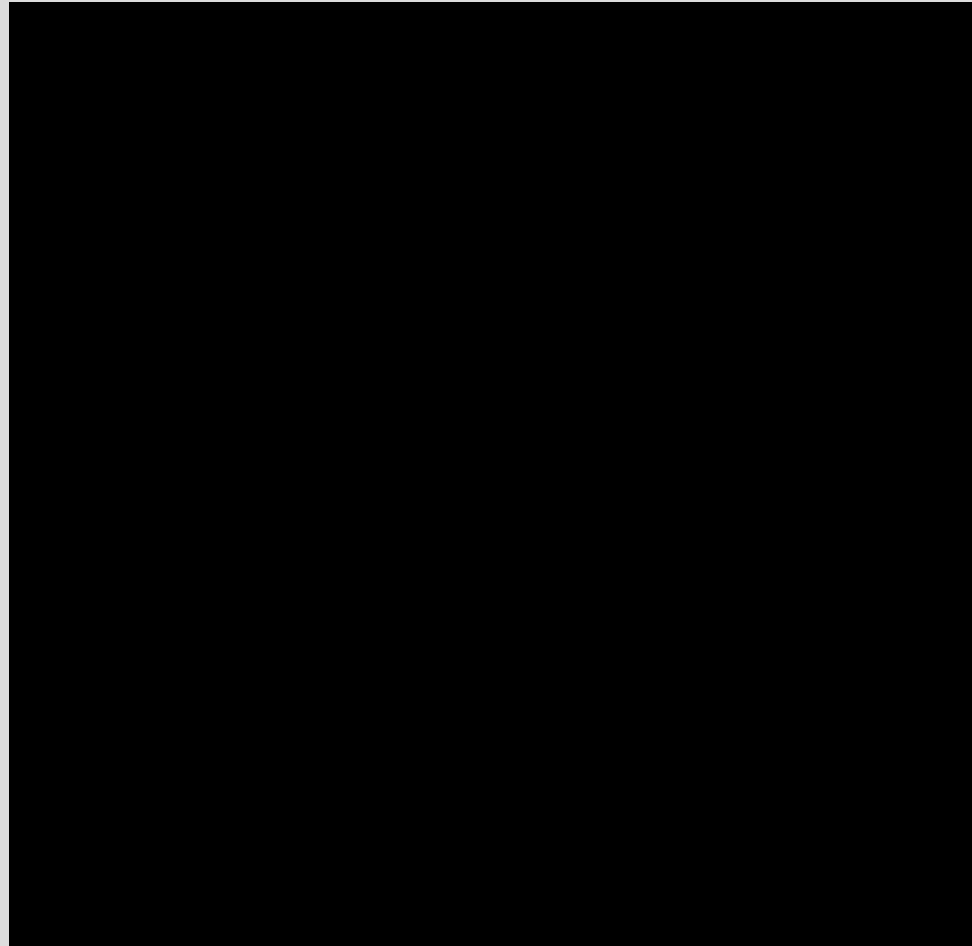
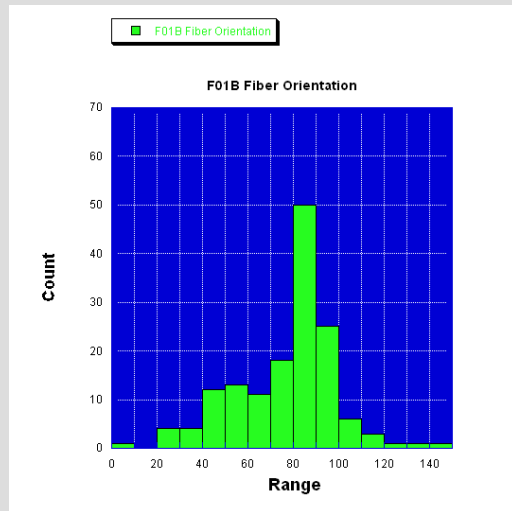
- Explore decoupled fiber orientation & flow kinematics:
  - Express the fiber interaction coefficient  $C_I$  in Advani-Tucker or Folgar-Tucker model as a function of **the fiber aspect ratio and volume fraction**

$$C_I = C_I(\text{Fiber volume fraction, aspect ratio})$$

- Develop a coupled approach
- Account for effects of fibers on flow kinematics
  - Determine the effect of processing conditions and fiber characteristics on the composite



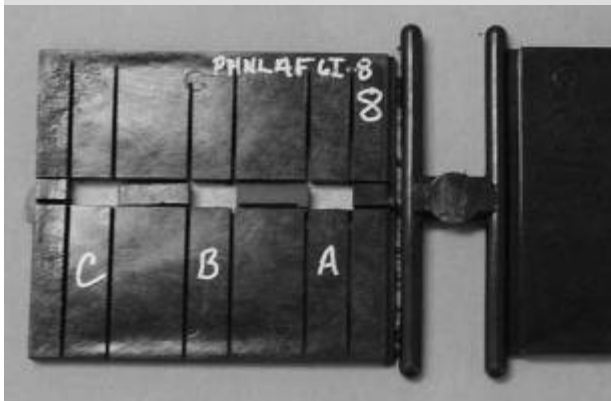
# XCT of Ni-coated CF – Polypropylene



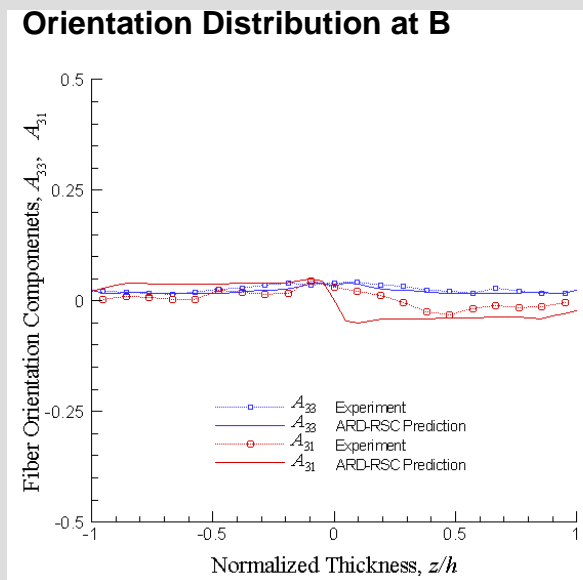
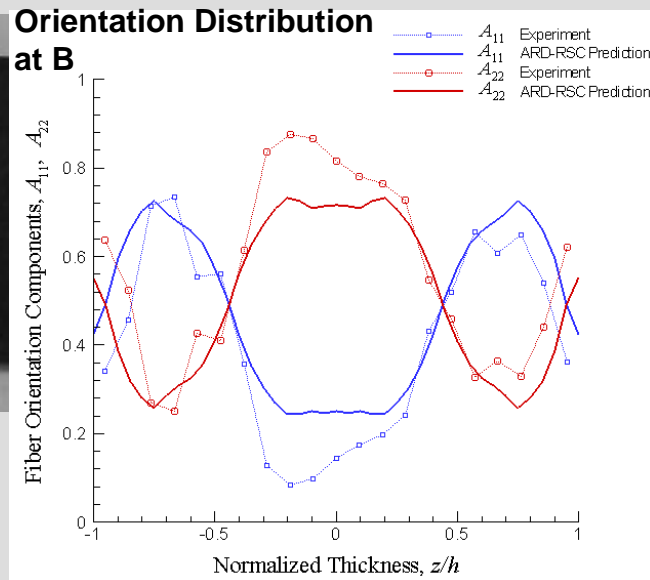
Injection molded ISO plaque, 31% fiber volume, ISO Plaque

# Process Modeling to Property Prediction of Injection-Molded Long-Fiber Thermoplastics

- ▶ A new fiber orientation model was developed to incorporate an *anisotropic rotary diffusion* (ARD) term to accurately model fiber-fiber interaction in long-fiber thermoplastics (2007):
  - The constant  $C_1$  in the Folgar-Tucker model is replaced by an anisotropic rotary diffusion tensorial term.
  - The new model is being implemented in Moldflow for process-linked-structural analysis



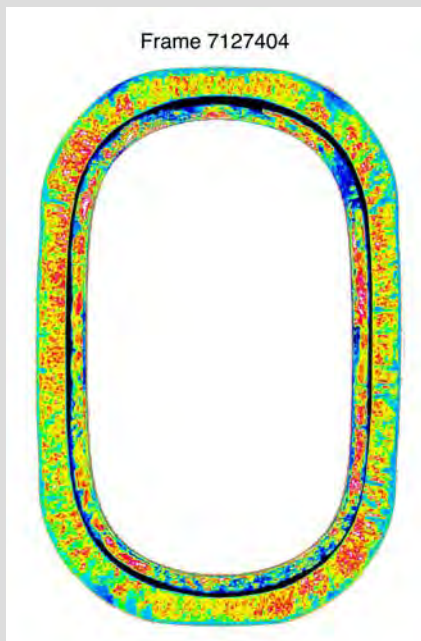
IM long-glass-fiber/PP ISO-plaque: 3 regions denoted as A, B, and C were measured fiber orientation



Effects of Fiber Length and Orientation Distributions on the Elastic Properties of Long-Fiber Injection-Molded Thermoplastics: Part I – Property Prediction, Submitted to *Composites Science & Technology*, B. N. Nguyen, V. Kunc, B. Frame, J. H. Phelps, C. L. Tucker III, S. K. Bapanapalli, J. D. Holbery, M. T. Smith

## 2. Analysis of Compression Molded Carbon Fiber/Epoxy Composites

1. Forming analysis on HexMC CF compression molded hardware.
2. Develop NDE techniques specific to chopped CF material.
3. Develop property prediction algorithms specific to chopped fiber CF.
4. Develop accelerated moisture uptake test for CF composites.



# Real Time Ultrasonic Inspection System (RTUIS)

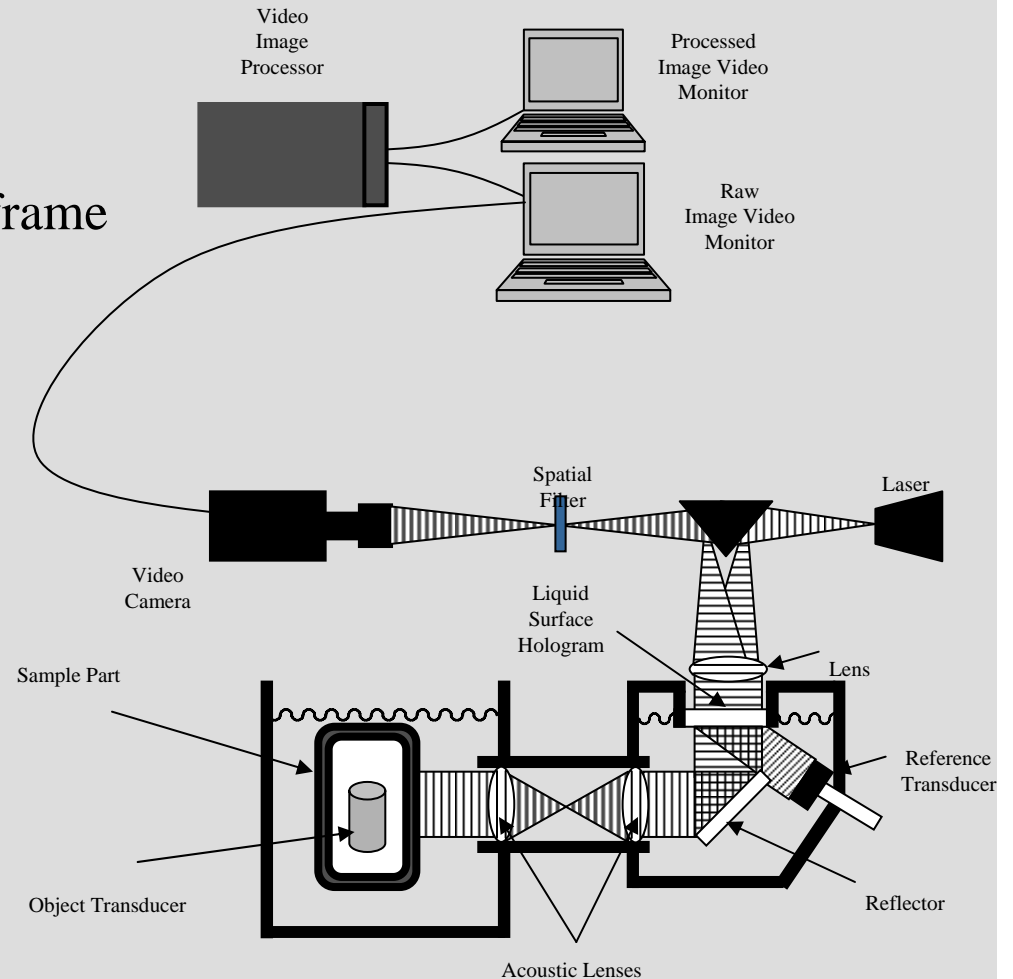
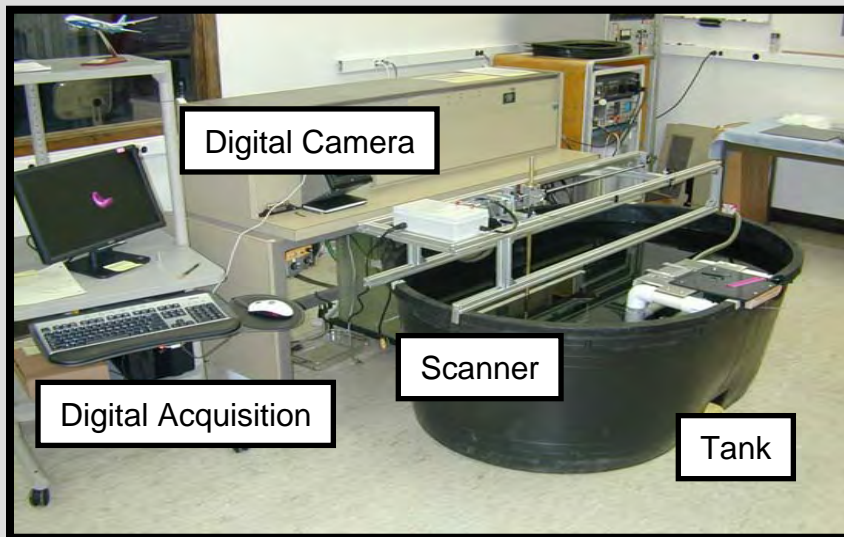
Liquid surface ultrasonic detector that produces an instantaneous two-dimensional image

## 1. Speed

- Instantaneous Image
- 2.5" x 4.0" FOV, 6 $\mu$ sec
- 60 frames/sec; 5625 pixels/frame
- 20 m<sup>2</sup>/hour

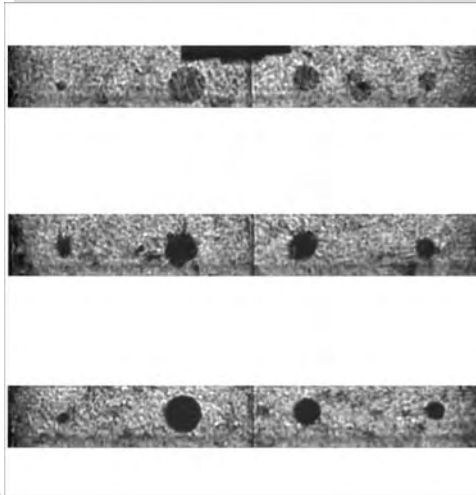
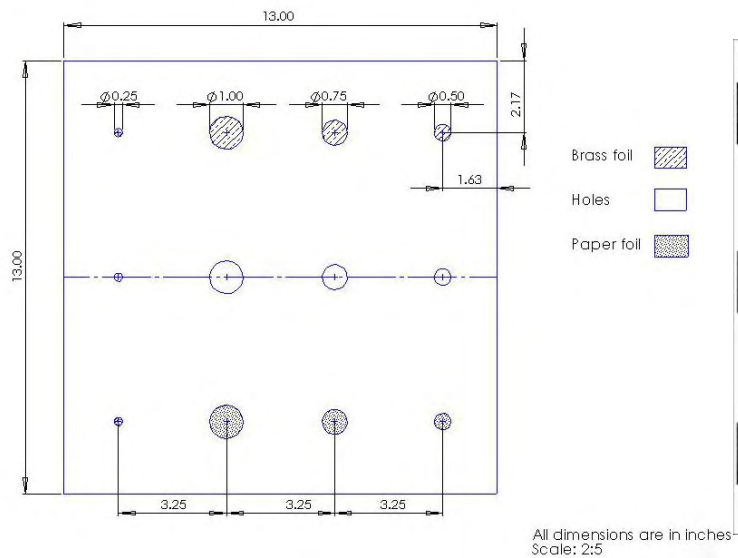
## 2. Resolution

- 3 wave lengths
- 1mm at 5MHz

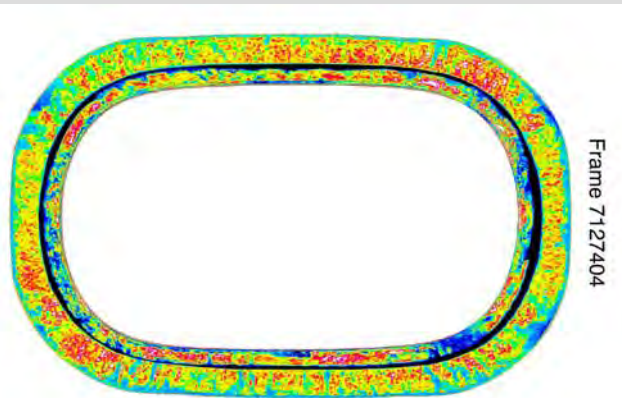
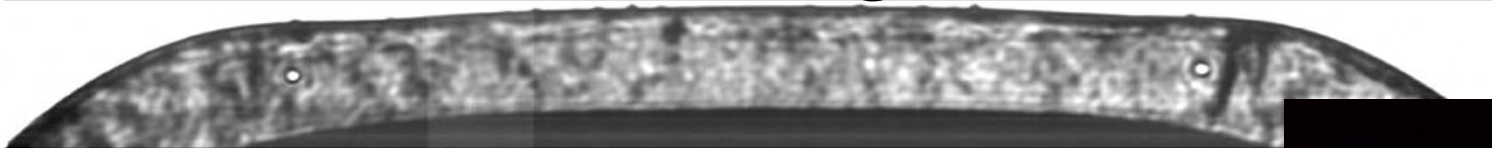




# Plate with Known Defects/Inclusions



# Boeing-Hexcel Part



Battelle



# 3. Natural Fiber Composites

DOE/Automotive Composites Consortium Funding  
\$2.5M, 3 years, start date June 1, 2007

- Enablers for large-scale automotive insertion:
  - Fiber preparation – from the farm to manufacture
  - Moisture absorption - thermal degradation during processing
  - Fiber preform – tailoring, hybrid architectures & molding (SMC)



Flax, hemp, sisal, wool and other natural fibers are used to make **50** Mercedes-Benz E-Class components.

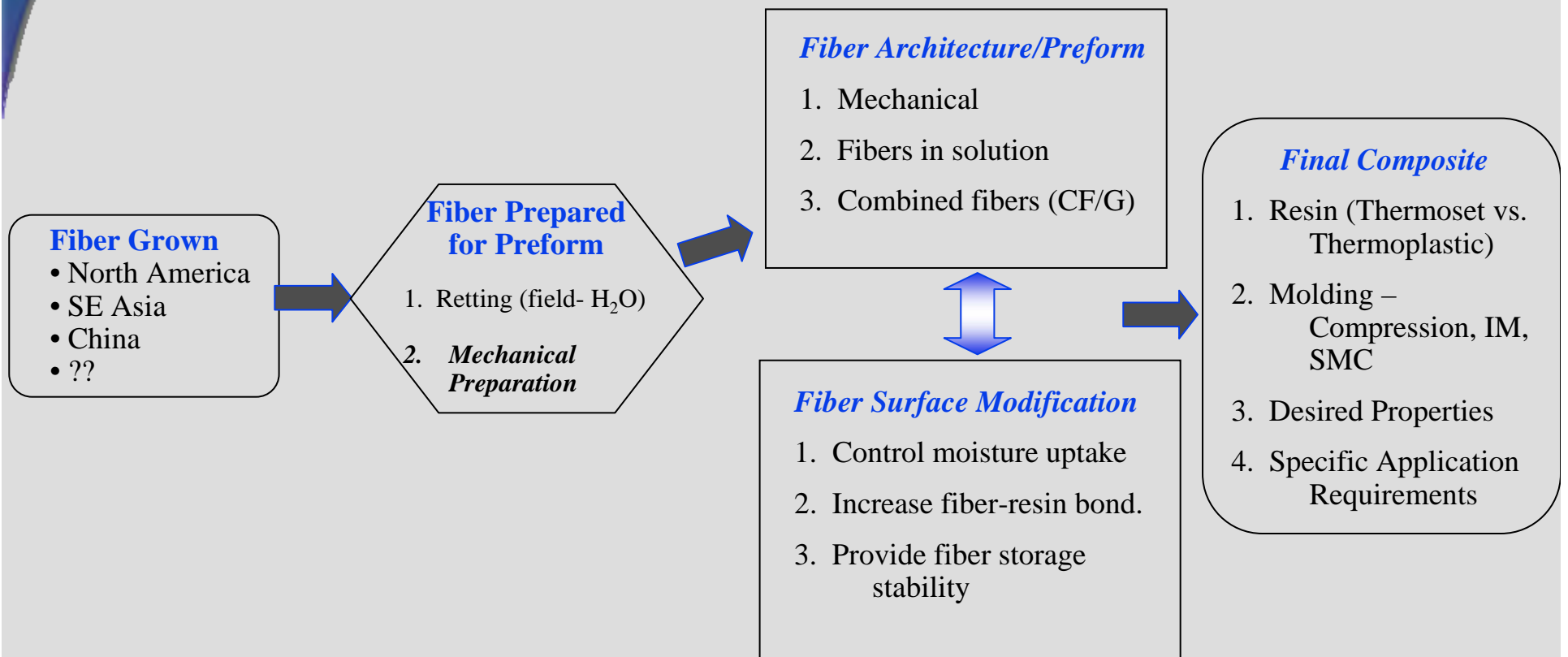
Mercedes-Benz S-Class has 42.7 kg of natural fiber components: Door & pillar inners, head liner, rear cargo shelf & trunk components, thermal insulation

# Typical Natural Fiber Properties

Property	E-Glass	Hemp <sup>2,3</sup>	Flax <sup>3</sup>	Ramie	Kenaf <sup>4</sup>	Coir <sup>3</sup>	Sisal <sup>3</sup>	Jute <sup>3</sup>	Wood Fiber <sup>1</sup>
Cost (\$/lb)	~ 1.10	0.30	0.33	?	0.24	0.20	0.36	0.20	??
Density (g/cc)	2.62	1.47	1.4	1.5	1.45	1.25	1.33	1.46	0.6 – 1.1
Tensile Strength (MPa)	<b>3400</b>	<b>550-900</b>	<b>800-1500</b>	500	<b>930</b>	220	600-700	400-800	900-1500
Specific Strength (s/ρ)	<b>1275</b>	<b>~475</b>	<b>~800</b>	333	<b>641</b>	176	488	410	??
Elastic Modulus (GPa)	73	70	60-80	44	53	6	38	10-30	10 - 80
Specific Modulus (E/ρ)	<b>28</b>	<b>~47</b>	<b>~26-46</b>	29	<b>36</b>	5	29	7-21	??
Elongation at Failure (%)	4.8	1.6	2.7-3.2	3.6-3.8	1.6	15-40	3-7	1.5	??
Moisture Absorption (%) <sup>4</sup>	-	<b>6-12</b>	<b>8-12</b>	8-17	10-12	8	10-22	12-14	12-14

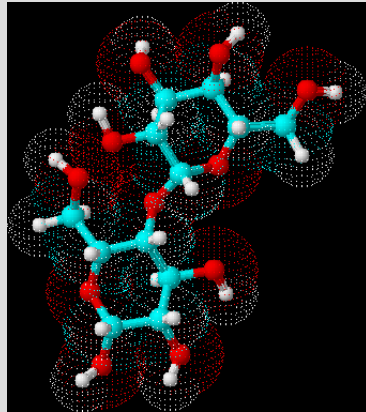
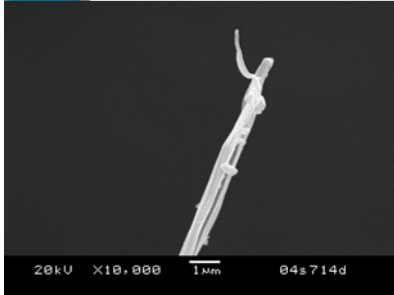
1. Peltola, P., "Green Composites", Chapter 5, CRC Press 2004.
2. Hempline, Inc., Ontario, Canada.
3. Eichhorn, S.J., et al., J. of Mat. Sci. (2001) 36, pp. 2107-2131.
4. Natural Fibers, Biopolymers, and Biocomposites, Mohanty et al., CRC Press, p. 41, 2005.

# Natural Fiber Composite Roadmap



*PNNL Current Research*

# Natural Fiber Surface Modification

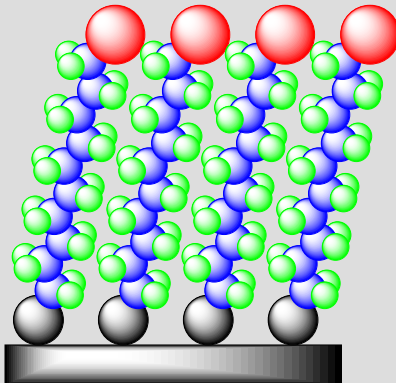
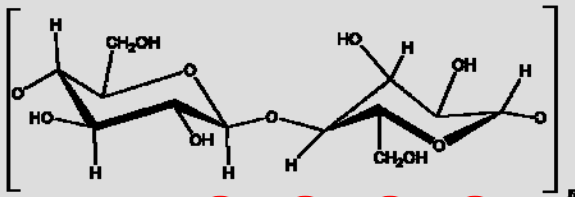
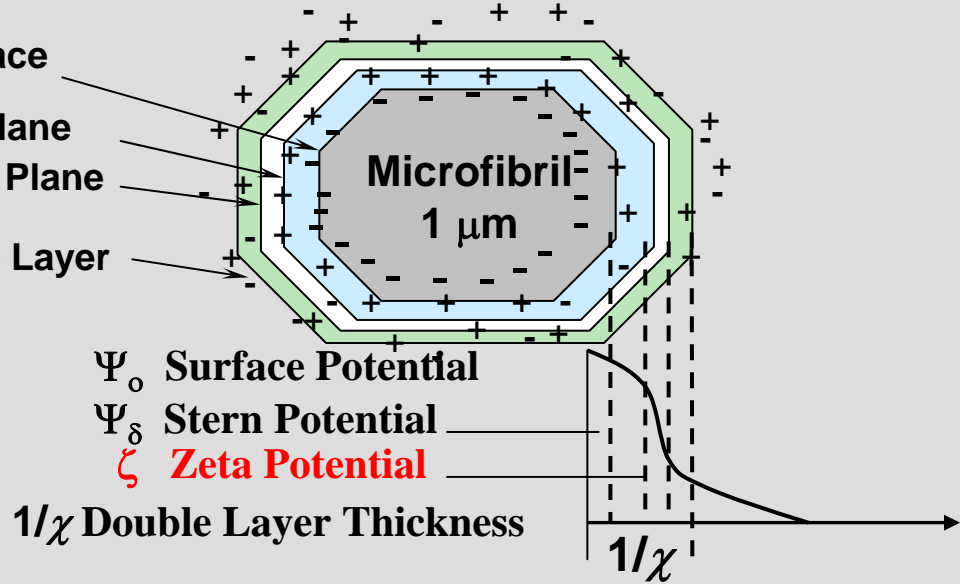


Microfibril Surface

Stern Plane

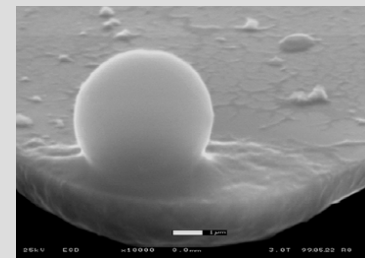
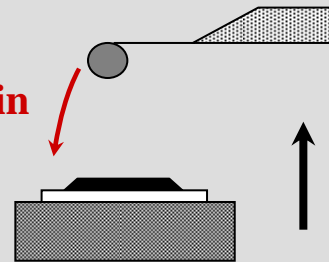
Shear Plane

Diffuse Layer



AFM Adhesion Measurement

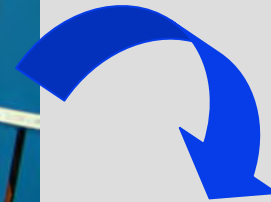
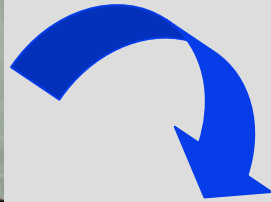
Snap-in



Holbery, J.D., et.al., J. of Micromechanics and Micromaterials, 10, 85-92 (2000)

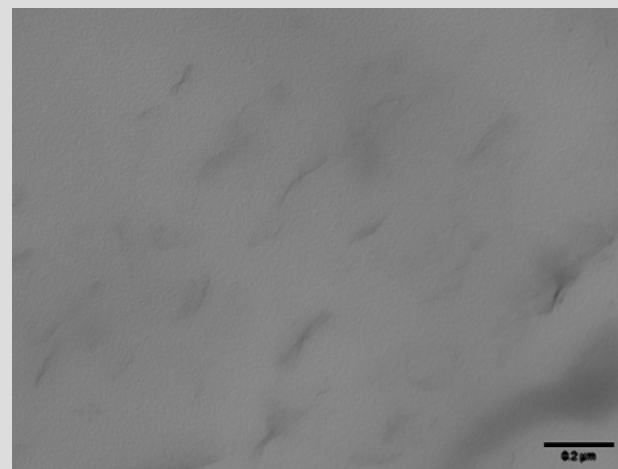
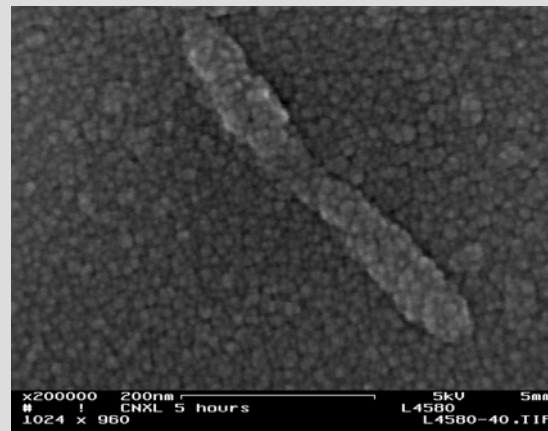
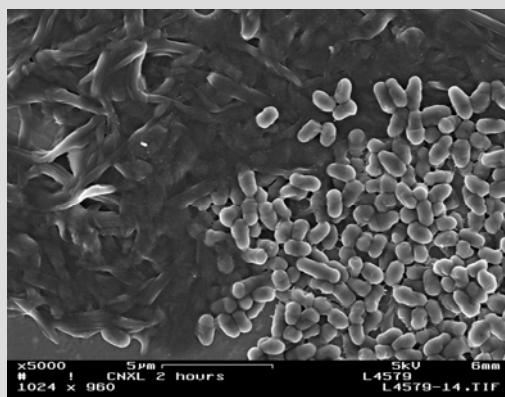
Holbery, J.D., et.al. Phys. Rev. D, Oct. (2000)

# Hemp-Kenaf Compression Molding



# 4. Nano-Scale Composites

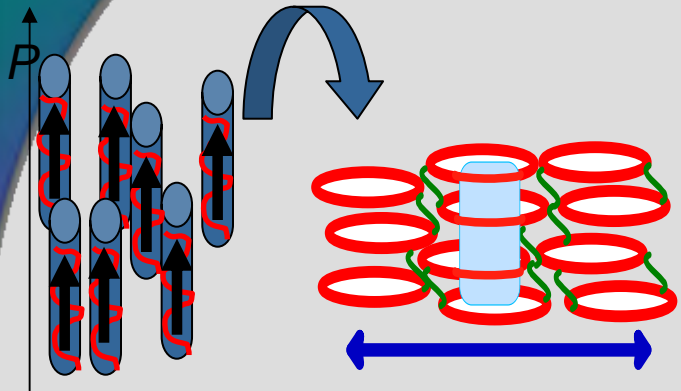
## Bio-based Materials



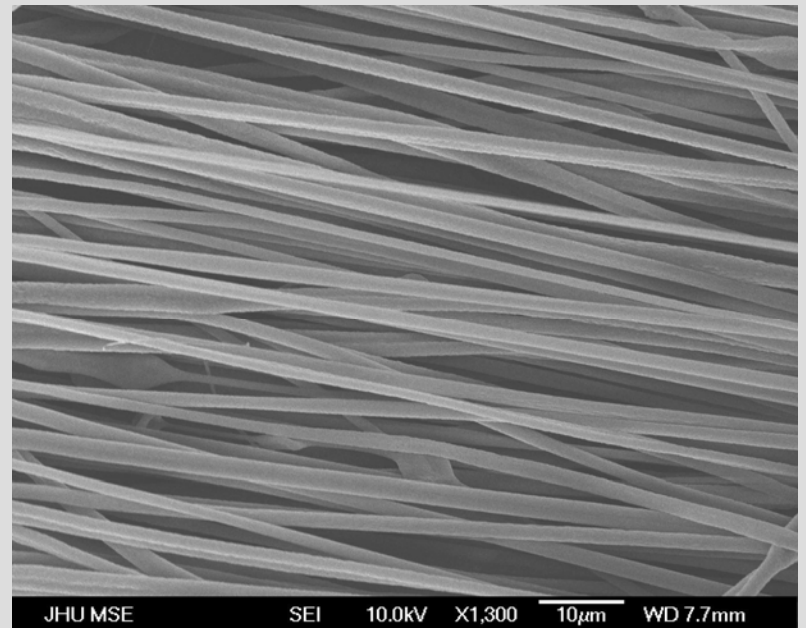
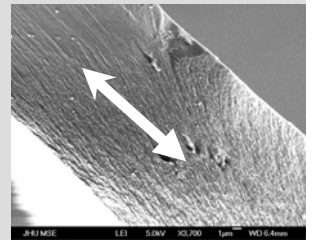
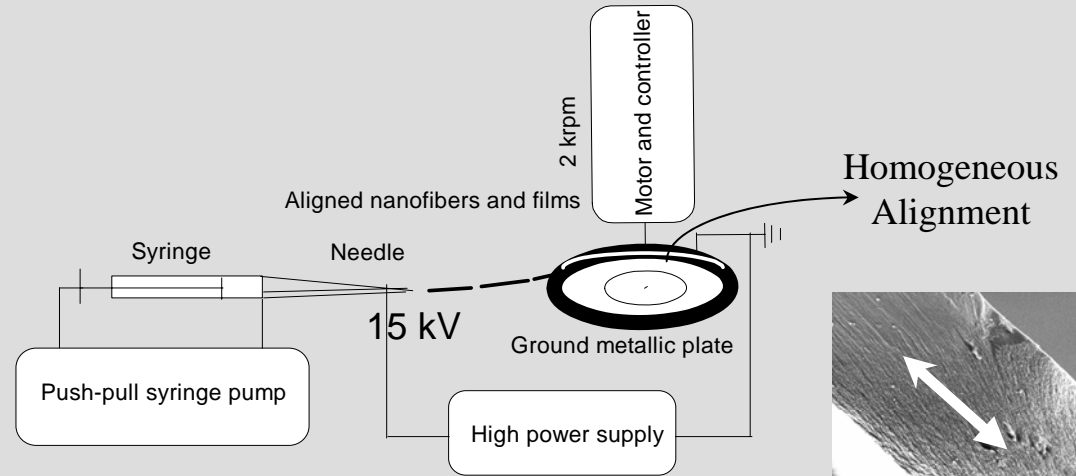
### Synthesis of Cellulose Nanocrystals

Collaborate with Kaichang Li, OSU and Mike Wolcott, WSU  
Holbery et al., Amer. Soc. Composite Proceedings, 2007  
Holbery et al., J. of Comp. Matls., in review

# Specialty Composites Electro-spinning Polymer Fibers and Textile Composites



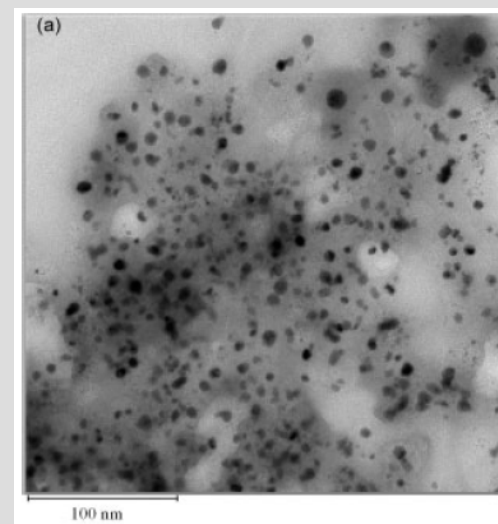
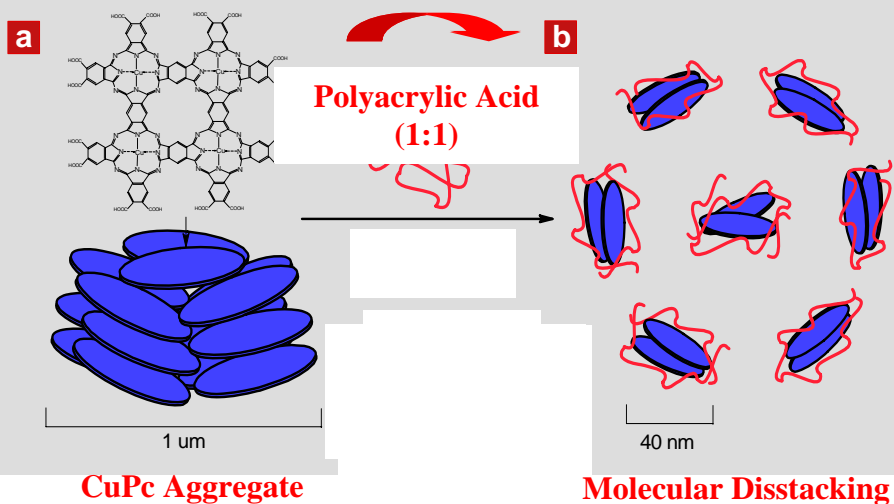
Aligned helical Rod-like LC Peptide Biopolymers



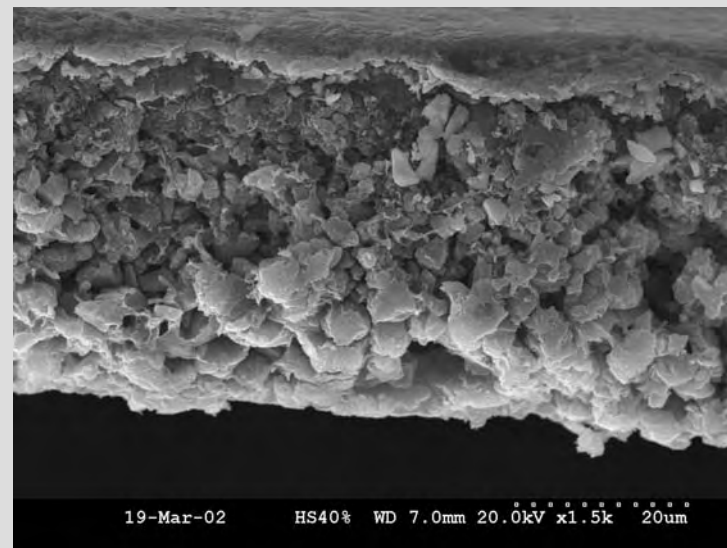
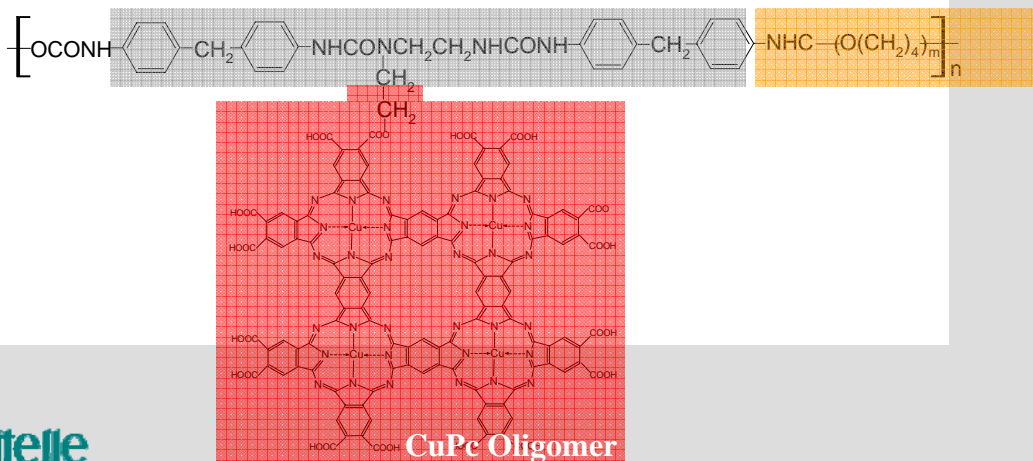


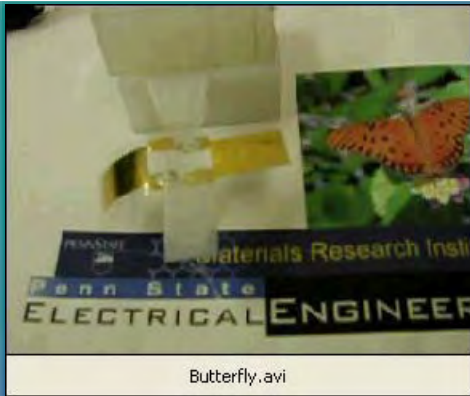
# 5. All-Organic High-Dielectric-Constant Composite Actuator Materials

## • Filler Surface and Interface Modification

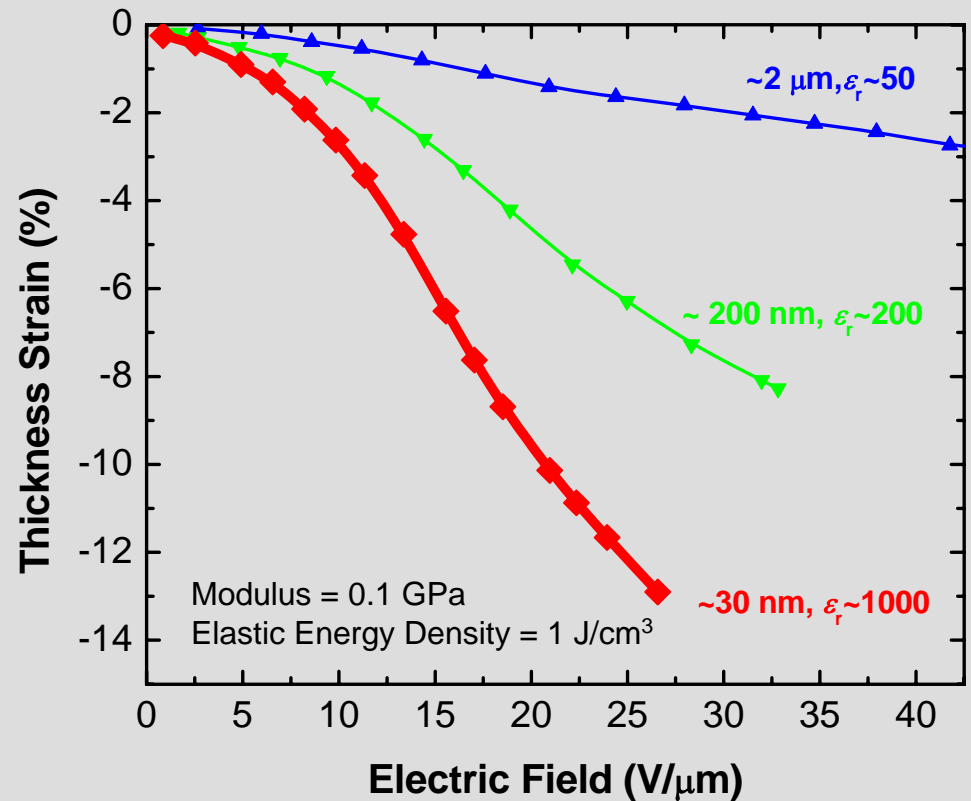
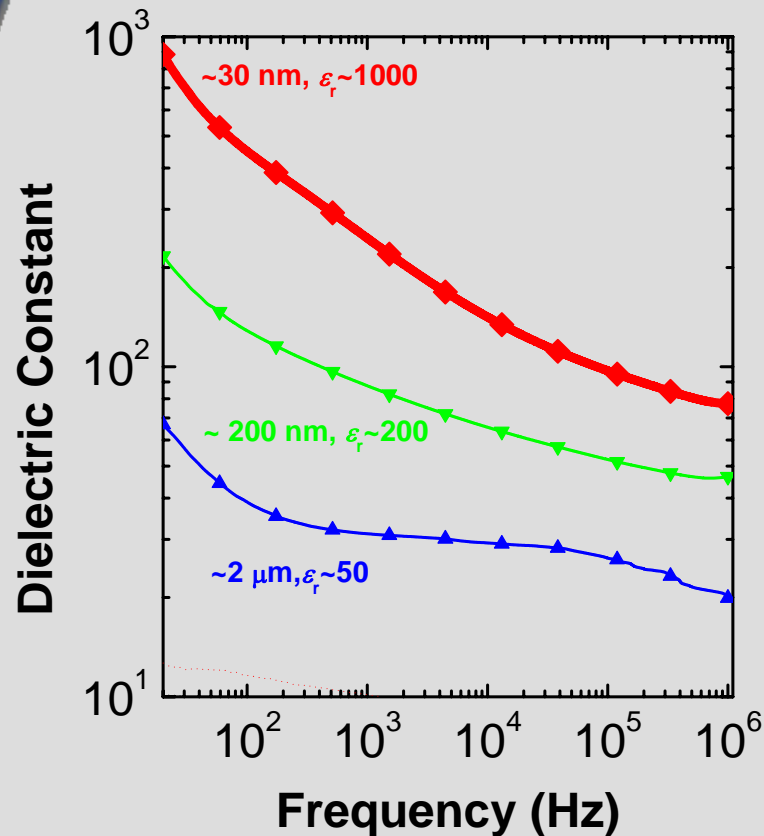


## • Filler Chemical Grafting and Polymerization



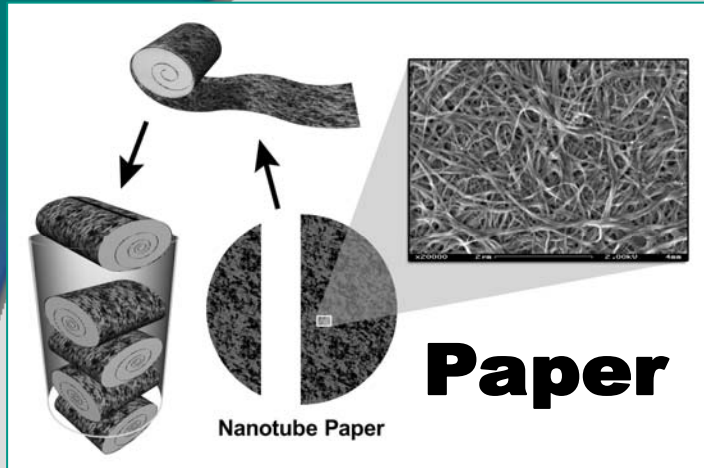


# High-Dielectric-Constant Nano-Phase Polymers and NanoComposites in Low-Voltage Actuation

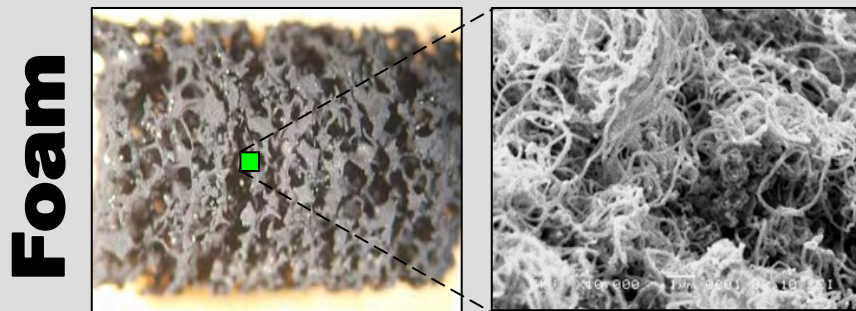
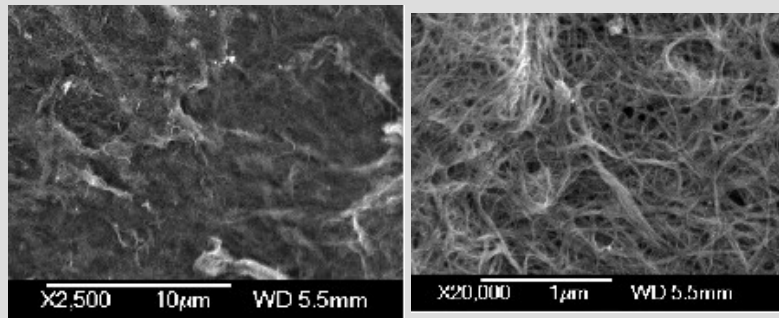


# Nano-Scale Fiber Composites

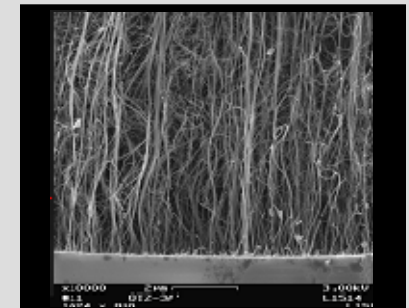
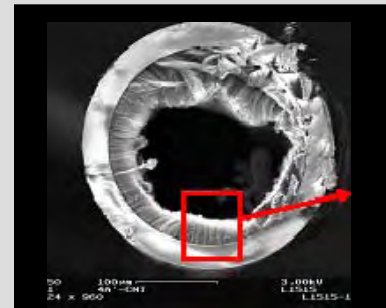
## Carbon Nanotube Research



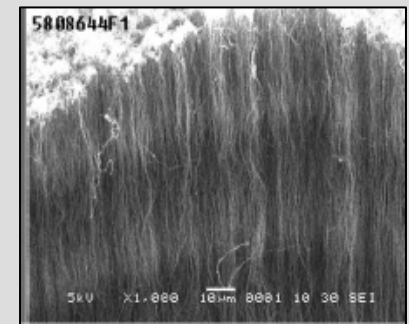
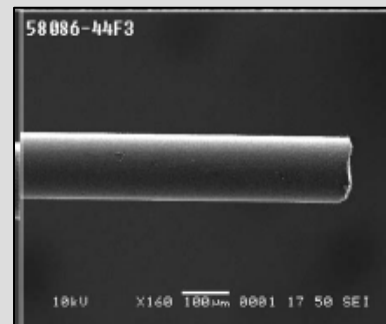
- Commercial CNT's – PNNL has grown CNTs from various sources
- Developed inexpensive process to produce CNT paper
- Produced paper from carbon fibrils
- Potential sensor, industrial applications



**Capillary**



**Fiber**



# Collaborators

1. **Manufacturers**
  - Ford Motor Company
  - General Motors
  - Chrysler
  - Hewlett - Packard
  - Material Innovation Technology
  - Meridian Automotive
  - Continental Automotive Systems
  - Moldflow
  - Albany International
2. **Materials Suppliers**
  - KenGro Inc. (MS)
  - Stemergy Inc. (Ontario, Canada)
  - Ashland Chemical
  - AOC
  - Reichold Chemicals
  - Hexcel
3. **Universities**
  - Prof. Kaichang Li – Oregon State Univ.
  - Prof. Mike Wolcott – WSU
  - Prof. Chuck Tucker – U. Illinois
  - Prof. Don Baird – VA Tech

# Acknowledgements

1. The DOE and US Automotive Composite Consortium for their generous support.
2. DOE Office of Energy Efficiency and Renewable Energy
  1. Vehicle Technology
  2. Biomass
  3. Buildings and Infrastructure
3. The Boeing Company
4. American Plastic Council

# Thank You



*Pierce Alan Holbery – Bringing joy since August 3, 2007*