NIST – The New Steel
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CNT Composites for High Performance Structural Applications: Development, Measurement, and Scale-up

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Talk Outline

1. ***Development:*** ***Mechanical needs, dispersion, functionalization***
2. ***Measurement:*** ***Direct and indirect methods, testing, characterization***
3. ***Scale-up:*** ***Value chain, materials to finished product***

Mechanical Needs- Sporting Goods, Aerospace

- Strength
  - Dent, bending, shear, compression, impact
- Toughness
  - Durability, safe failure mode, external flaw trauma
- Consistency
  - Enhances player performance
  - Maximum performance that always meets industry limits
- Environmental
  - Corrosion, sun, chemicals, "the hot trunk"

Nanotech Technology Landscape

- **The Good**: Very Strong and light
  - CNTs have awesome strength – 20x to 100x that of steel … and that's conservative
  - 1/6 the weight of steel
- **The Bad**: Severe technical challenges
  - Consistency – variable quality, hard to detect
  - Volume of supply – able to meet demand
  - Tendency to agglomerate – dispersion issues
  - Bond strength – how to bond without destroying
- **The Ugly**: Problems can kill you
  - Poor quality CNTs or dispersion can lower strength
  - Health and safety – reality, unknowns

Development Approach

- Identify an Industry partner for strategic needs
  - CNT sourcing – volume, quality, measurement
  - CNT dispersion – at various stages
  - Supply arrangement
- Focus the R&D effort
  - Maximize material properties; loading curve
  - Define the envelope, for allowable variation
  - Focus on elevating minimum properties
  - Develop effective analysis tools
- Leverage the results over wide product base
  - Establish designs that exploit the advantage
  - Maintain/ observe quality limits – materials, processes

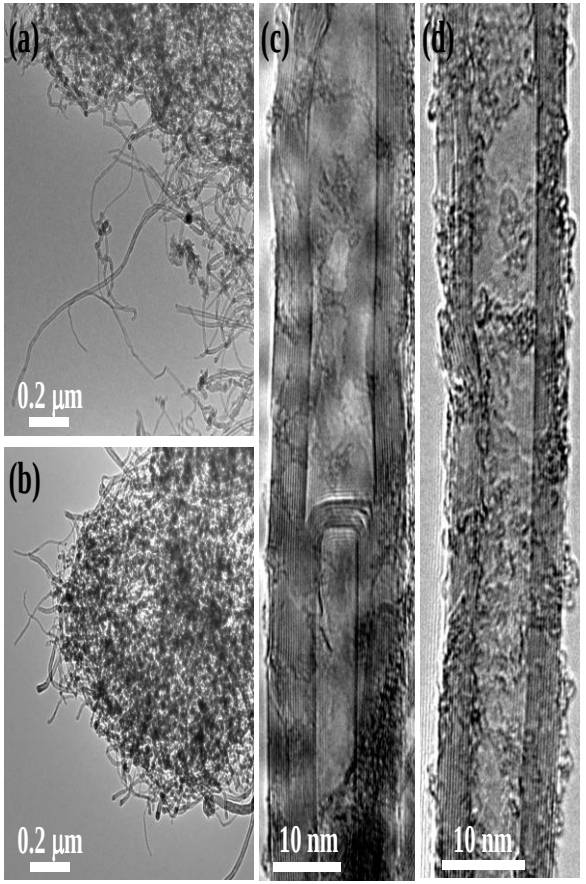
Shear processes for CNT dispersion

(a) Parallel – Statistical treatment

- High-shear rotor/ stator
- Ultrasonic bath
- Sonication probe
- Shakers

(b) Serial – All elements see shear field

- 3-Roll Milling
- Microfluidic high pressure labyrinth

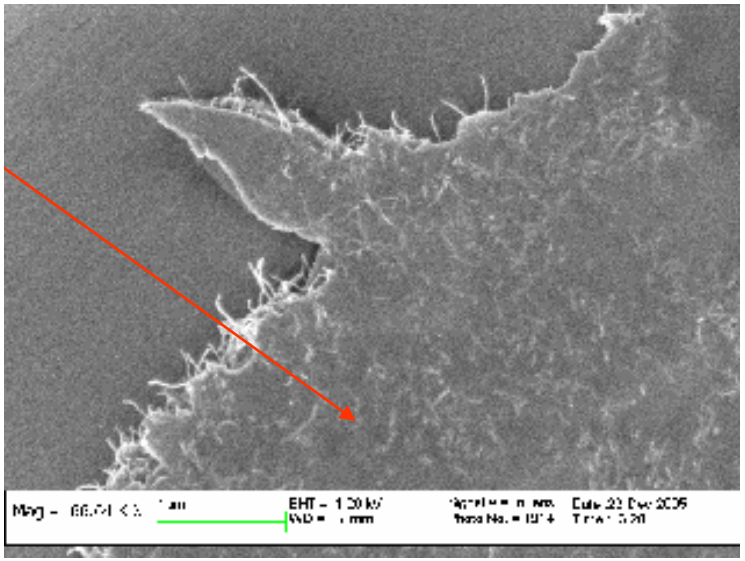


You <u>can</u> have too much…

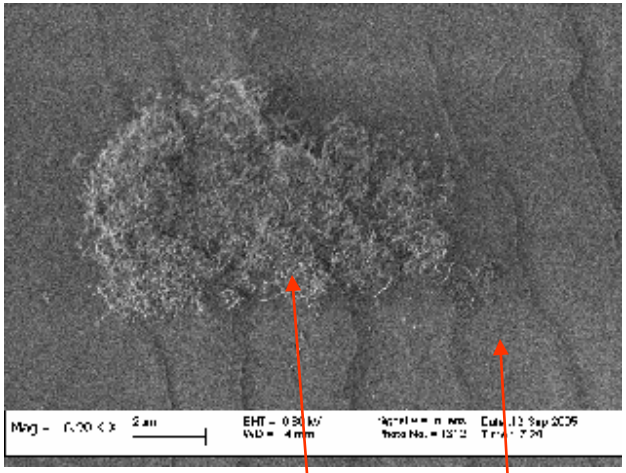
CNT in Epoxy Resin: Bad Vs. Good Dispersion

Well dispersed/Distributed MWNT In Epoxy Resin

Good dispersion

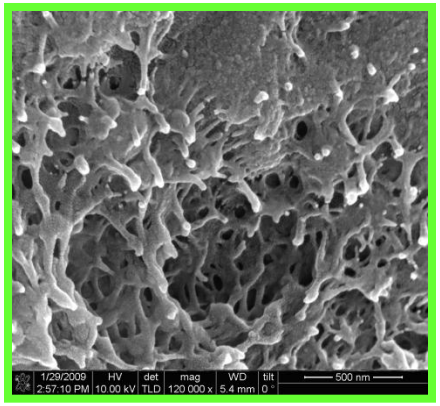


Bad dispersion



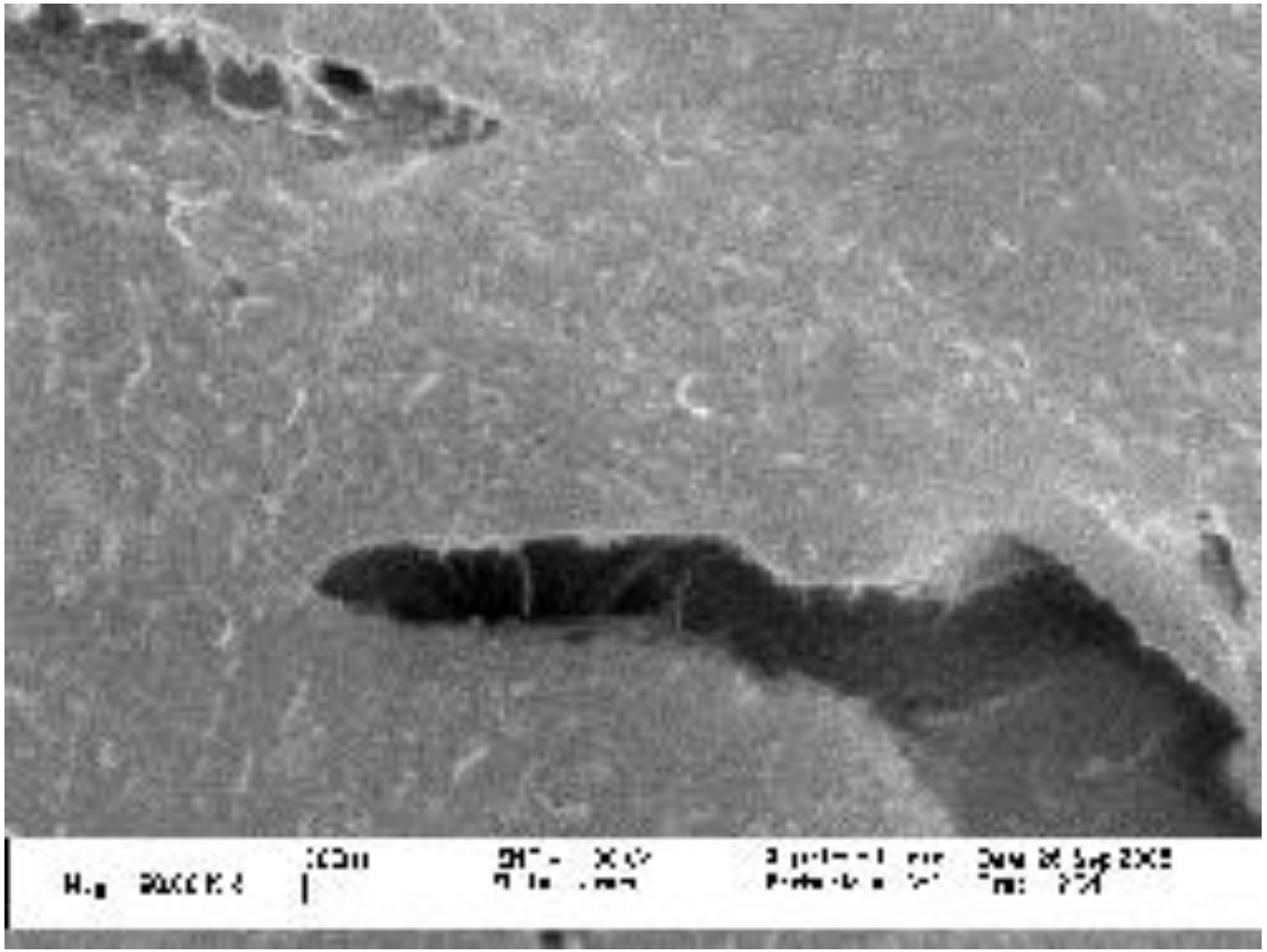
Island of MWNT In Epoxy Resin (very stiff)

Nothing but Resin (not stiff)

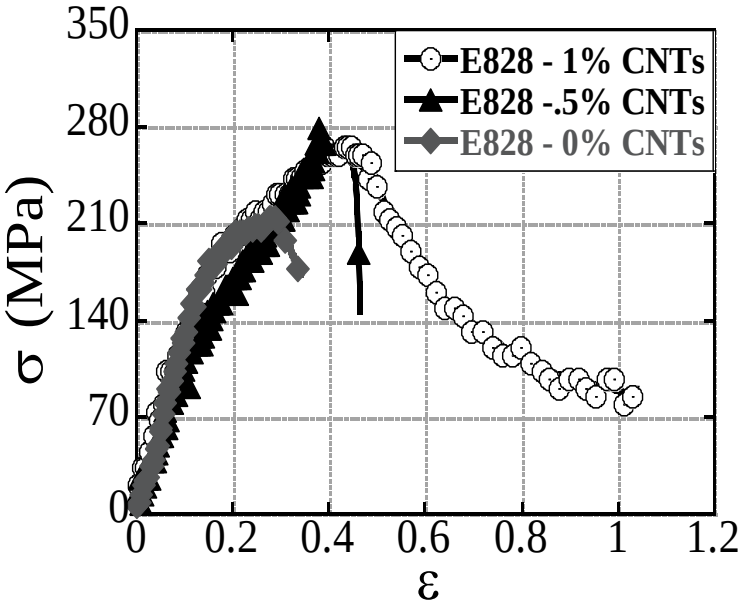


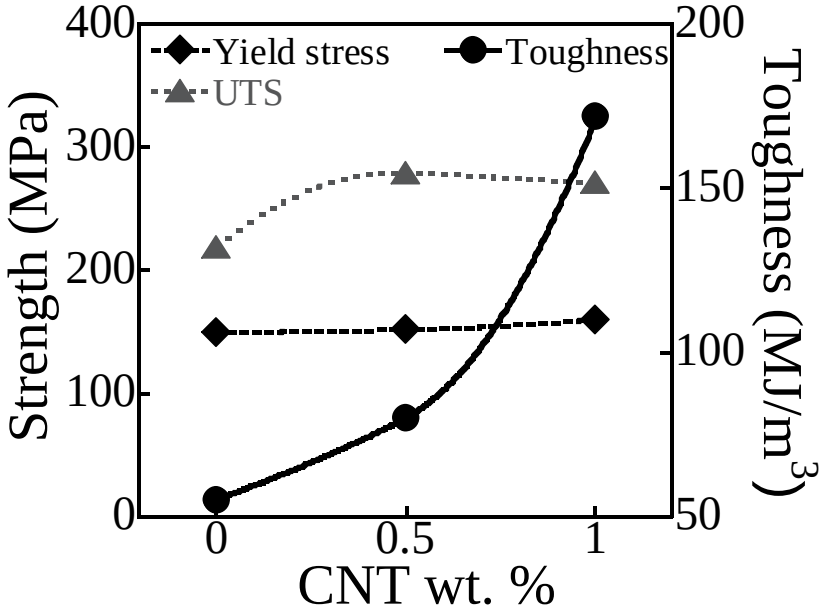
Good dispersion – Fracture surface showing CNT pullout

Fracture – CNTs span and stop cracks



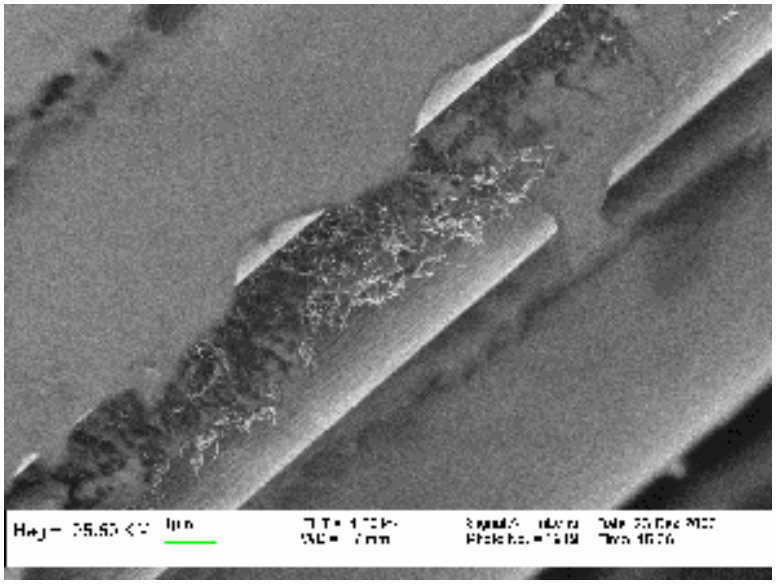
Loading curve: MWNT in E828 at 0%, .5%, 1%





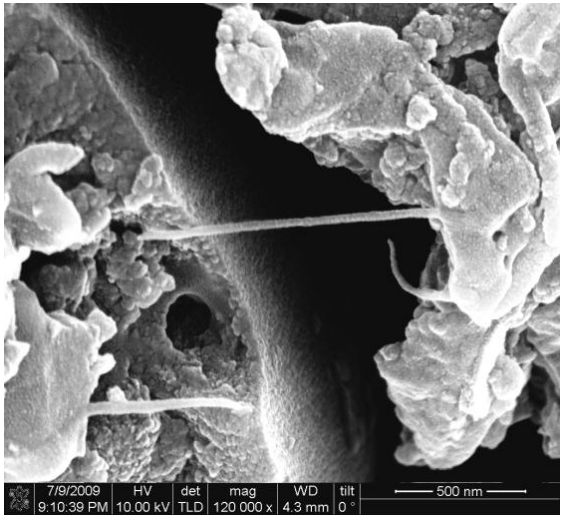
Premix Method – Substantial toughness gains

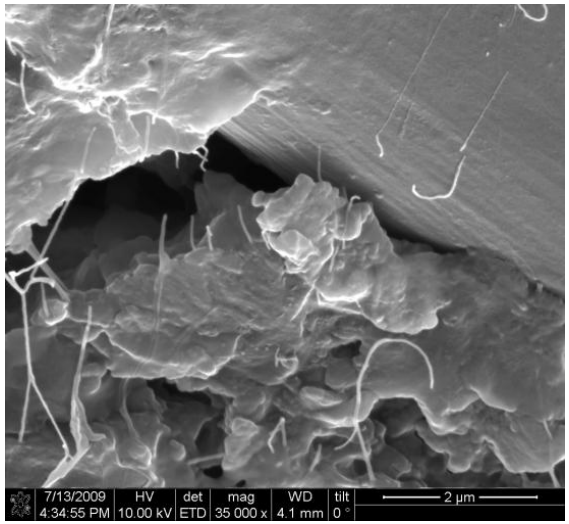
Composite view – CNT and carbon fiber



CNT fortifies resin areas, provides shear bridging

Functionalization provides attachment to CF, alignment and pullout under strain

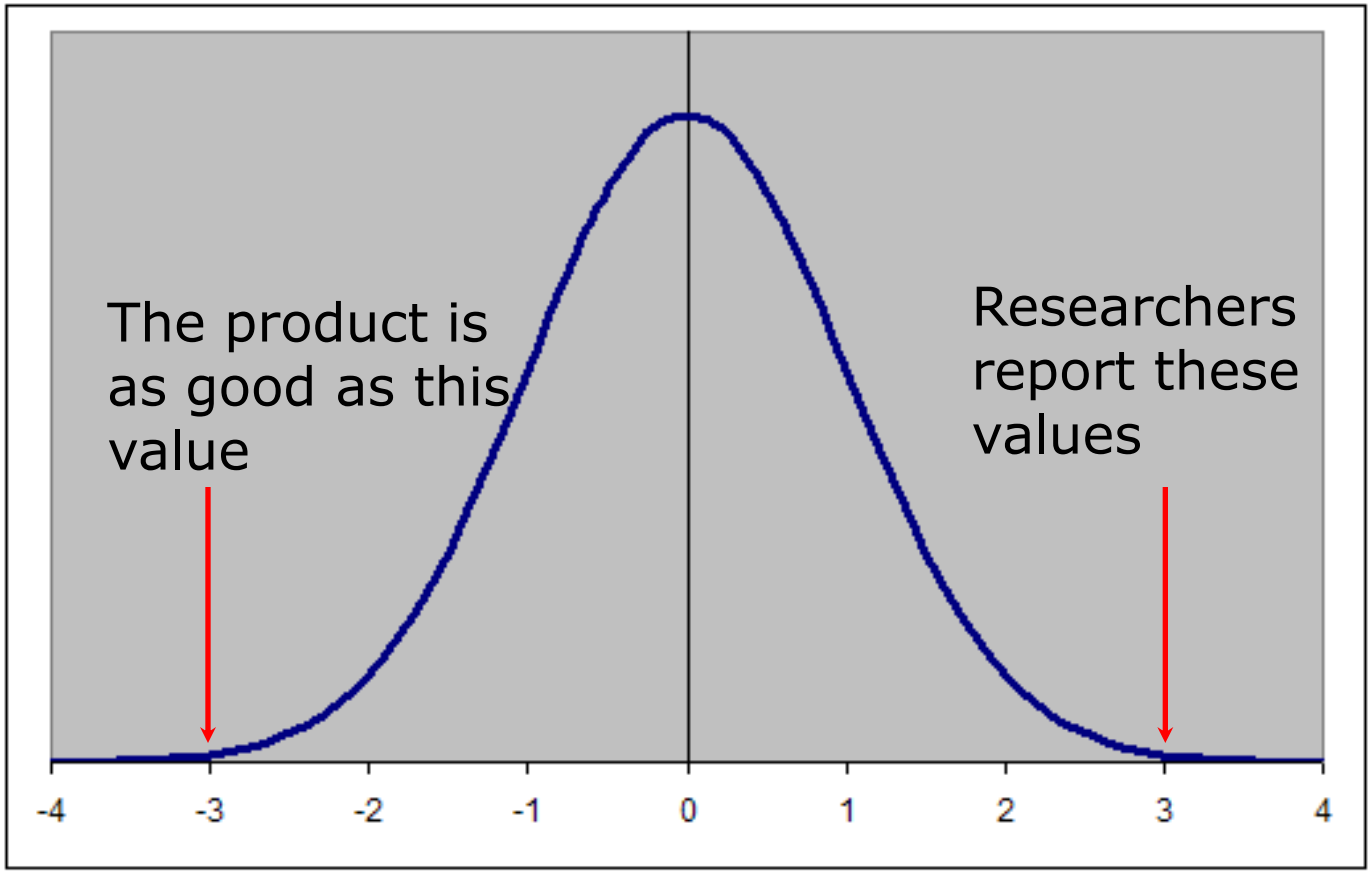




Measurement

Direct:	Hegman gage – Particle size progression
	SEM Evaluation – scale feature size
	Strand test – scale diameter ratio
	Laser measurement
Indirect:	Tensile or Short beam shear
	Miniature Disk Bending Test

The Technical Reality of Nano

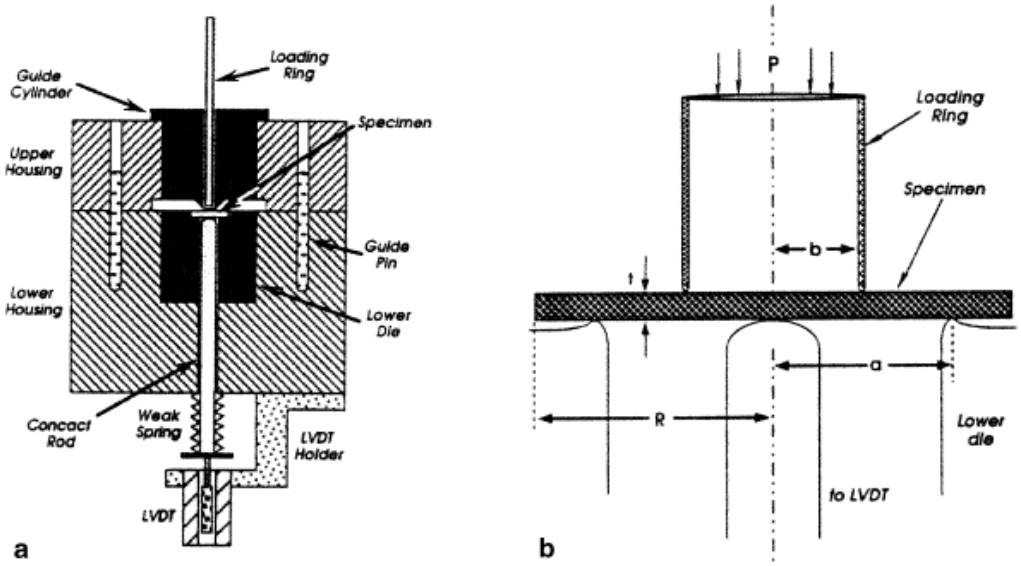


Strength and Toughness Range

Put another way:
If there is one bad flaw in every million nanotubes…
There are 30 in a kg-size object

Miniaturized Disk-Bend Test (MDBT)

Schematic diagrams of the MDBT: (a) The entire fixture; (b) Close-up view illustrating the specimen and the important geometric parameters.

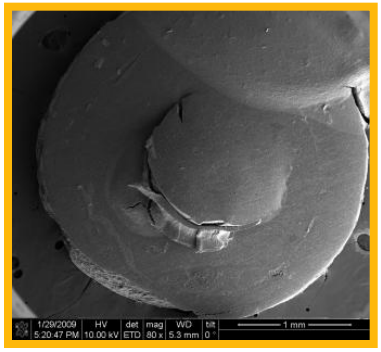


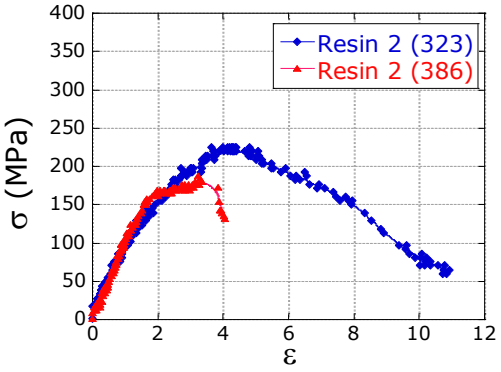
$$\sigma = \frac{3P}{2\pi t^2}\left\{(1+\nu)\ln\frac{a}{b} + \frac{(1-\nu)}{2}\left[1-\frac{b^2}{a^2}\right]\frac{a^2}{R^2}\right\}$$

P is the applied load, ν is Poisson's ratio, t is the specimen thickness and a, b, and R are the radii of the lower die, the loading ring, and the specimen respectively.

1) Slices are cut from resin samples cured in glass tubes
2) 3 mm disks are punched out, and ground to a thickness of 250-500 μm using 600 carbide paper.
3) Both sides of the disks are polished to a mirror finish , then tested.

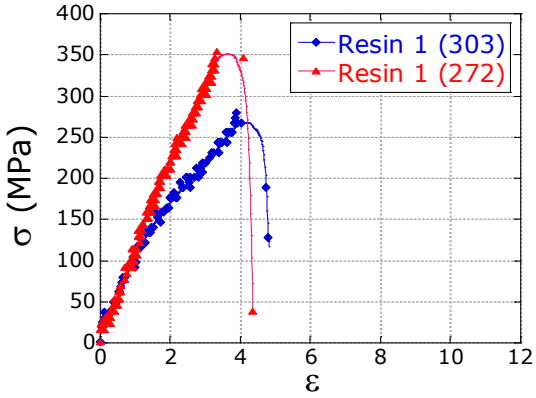
Miniaturized Disk-Bend Test (MDBT)

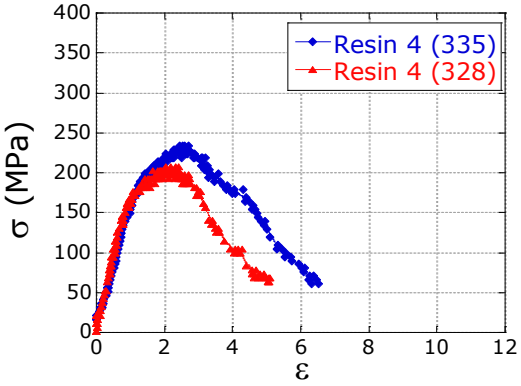




•Stress-strain curves are plotted to the same scale on both of the samples for each resin. The thickness of each sample (in μm) is indicated by the numbers in parenthesis.

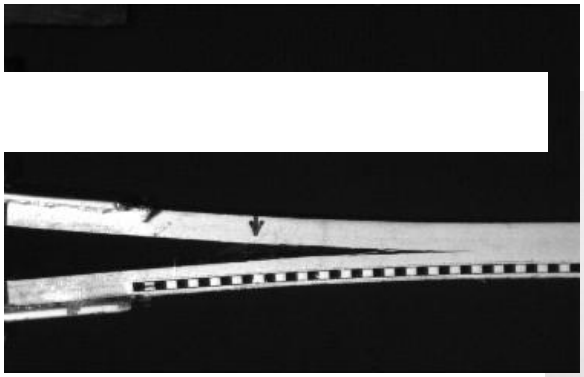
•Ultimate and yield strength are noted; Slope of elastic region gives a measure of modulus.





•Curves are integrated to the point of 10% drop from the peak, to give a measure of mechanical toughness

Materials Testing



G1C Measurement

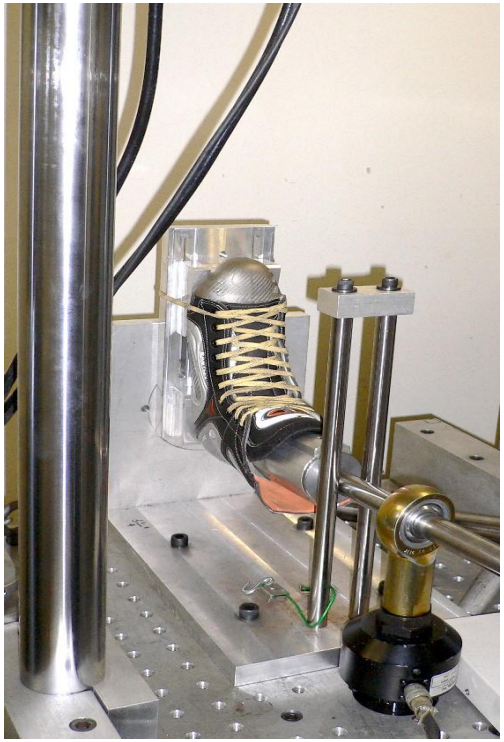
Beam and Charpy Impact

Short Beam Shear

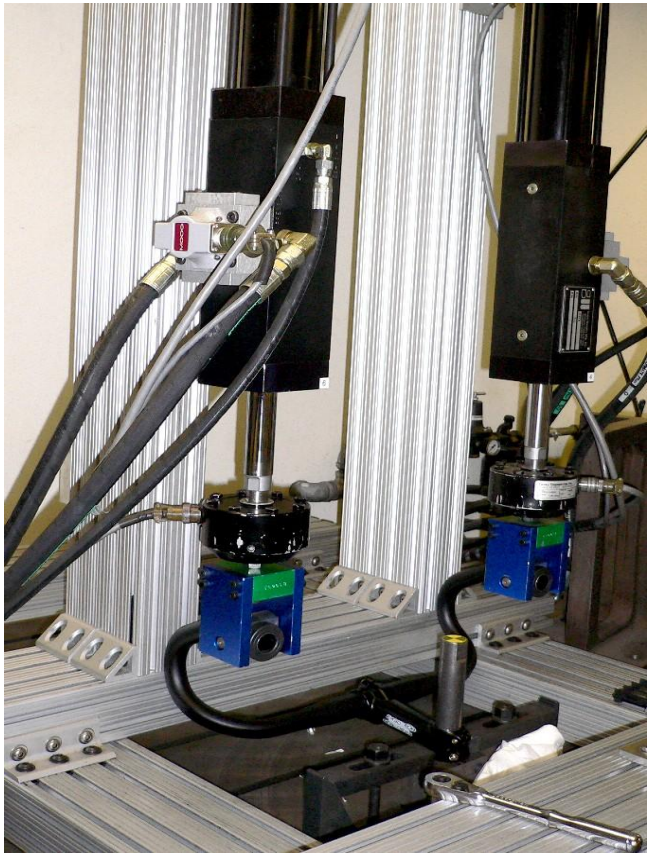
Materials Testing → Product Testing

Product Testing

Composite wheel static and fatigue testing



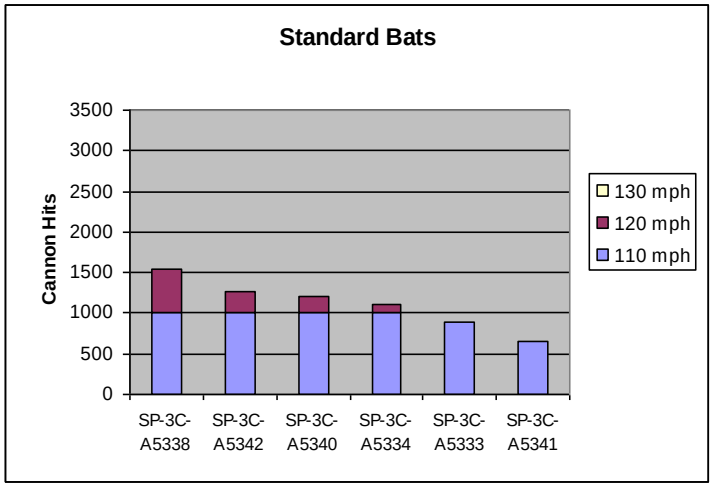
Composite hockey boot fatigue and wear

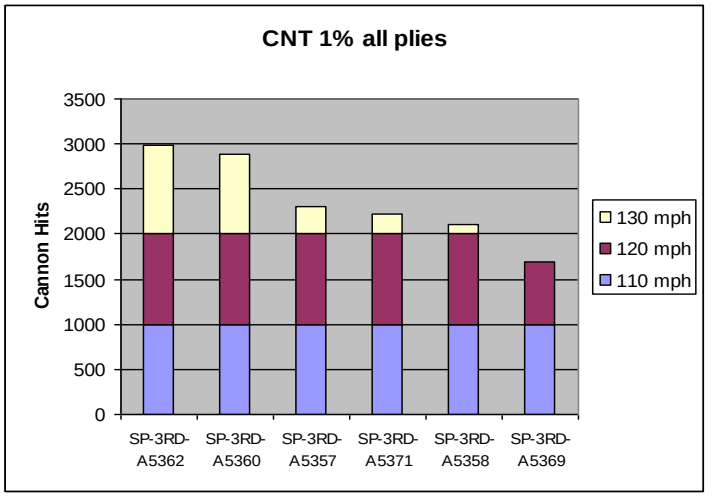


Handle bar fatigue

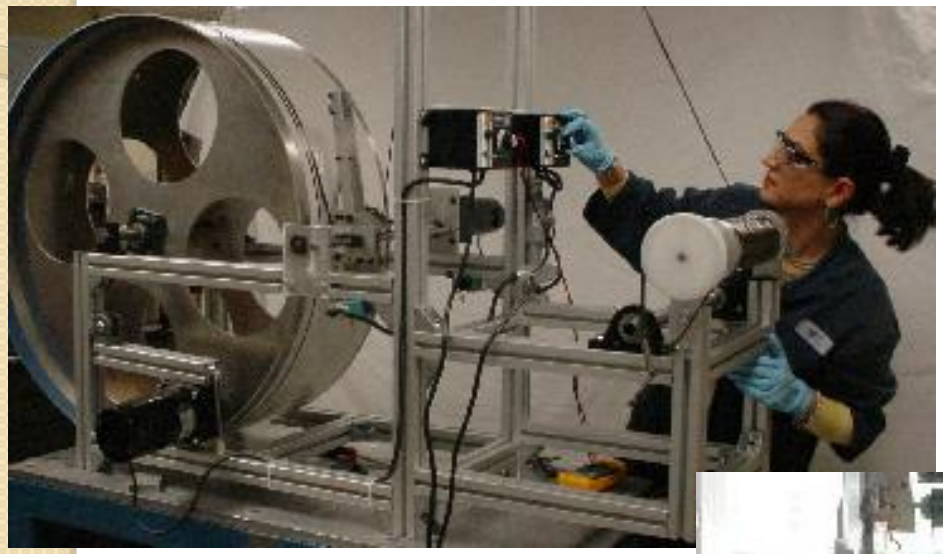
Product Testing

Bat Cannon –Repetitive impact at 110, 120, 130 mph





Scale Up: Lab CNT Material Development Linked to Production



Laboratory Prepreg operation, Van Nuys, CA

Production Prepreg Guangdong, China

CNT Scale up 101:

Disperse – CNT production set to control length, straightness, end closure, catalyst control and removal.

Disperse – CNT supplier separates prior to providing functionalization; controlled bond opening by acid treatment, plasma, etc; or VDW polymer treatment.

Disperse – Resin formulation. Chemical bonding to resin or other constituents. Pot time is known by aging study. Solvent additions are limited by formulation tolerance.

Sample CNT Composites' Value Chain

Carbon Nanotube Supplier

SWNT
DWNT
MWNT

Zyvex functionalizes with Kentera™

Zyvex Additives

Processed Nanotubes

Easton puts additive into composite matrix

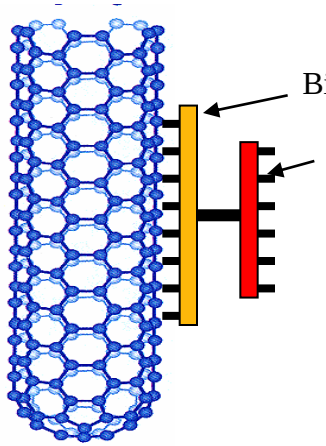
Enhanced Materials

Epoxy Prepreg, VARTM infusion

Easton design, testing, manufacturing

Finished Goods

Baseball Bats
Softball Bats
Bike Accessories
Hockey Sticks



Functionalization technology

Thanks for Listening.

Questions?

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