DOE Lignin to Carbon Fiber Workshop

Detroit, MI

Presented by:

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Bioenergy Program
Environmental Sciences Division

Oak Ridge National Laboratory

June 4, 2013
Substantive importance discussions

• What are the larger substantive issues?
• What are the questions we need to be asking?
• What is the State of Technology?
• What is currently researchable?
• What is deployable? At what scale? With what quality?
Substantive importance discussions

• What are our capabilities?
  – Carbon Fiber (materials) State of Technology

• What are our processing capabilities? At what scale?
  – Carbon Fiber Technology Facility (CFTF)

• What are our analytical capabilities?
  – Manufacturing Demonstration Facility (MDF)

• What about precursor sources?
  – Bioenergy Science Centers (BESC)

• What are our partnership possibilities?
  – Carbon Fiber Technology Consortium (CFTC)
The Energy Independence and Security Act (EISA) of 2007 sets aggressive goals:

- Move renewable fuels into the marketplace
- Reduce the nation’s dependence on foreign sources of energy
- Reduce GHG emissions from the transportation sector.

EISA established production volumes for the Renewable Fuel Standard Program (RFS), increasing the supply of renewable fuels to 36 billion gallons by 2022.

The U.S. Department of Energy’s (DOE) Biomass Program focuses on developing advanced biofuels to help meet the RFS goals.
Overall picture: Biomass for fuels and bioproducts

Cellulose and hemicellulose

Chemical building blocks: Plastics, industrial additives, biomedical applications

Biofuels

Cellulose and hemicellulose

Lignin

Up to 50% reduction in finished carbon fiber production cost

Melt spinner

Carbon fiber production line

Conversion cost: 50% of conventional

Robotic preforming

Biofuels

Lignin and innovative processing: ~50% less than PAN and conventional processing

Overall picture: Biomass for fuels and bioproducts
Biofuel and Lightweight Materials from Renewable Resources

Chemical building blocks for
- Plastics
- Industrial additives
- Biomedical applications

Cellulose & Hemi-cellulose

Lignin
Estimated reduction of 30 to 40% of finished carbon fiber production cost

30% to 60%

Carbon Fiber Production Line

Robotic preforming

Ethanol Fuel

Estimated conversion cost reduction of at least 30% of conventional conversion cost, or 15% of finished carbon fiber cost

Lignin and innovative processing technologies together yield estimated manufacturing cost reduction of 50% or more over PAN and conventional processing base case
Biomass feedstocks for energy

- Primary forestland resources
  - Fuelwood (Currently used)
  - Composite operations
    - Logging residue
    - Thinnings (timberlands)
  - Other removal residue
  - Thinnings (other forestlands)
  - Conventional products

- Primary cropland resources
  - Grain crops (Currently used)
  - Oil crops (Currently used)
  - Agricultural crop residues
  - Perennial grasses
  - Woody crops
  - Annual energy crops

- Secondary residues and waste resources
  - Pulping liquors (Currently used)
  - Mill residues (Currently used)
  - Unused mill residue
  - Crop processing residues
  - Waste oils and greases
  - Animal manures
  - Urban wood wastes
Bioscience and biotechnology spans resource to end use

**Center for BioEnergy Sustainability**
Land use logistics

**Feedstock selection:** Switchgrass, *Populus*

**BioEnergy Science Center**
Molecular biology, chemical and structural analysis and characterization, modeling and simulation

**Biomass deconstruction/conversion**

**Catalysis and Separations S&T**
Catalytic conversion, technology development for biomass separation

**Carbon Fiber Technology Facility**
Manufacturing Scale-up

**National Transportation Research Center**
Engine Research

**Lighter weight vehicles**

**Blendable biofuels**

**Lignin**

*BioEnergy Science Center*
Molecular biology, chemical and structural analysis and characterization, modeling and simulation

*Carbon Fiber Technology Facility*
Manufacturing Scale-up
BioEnergy Science Center
A multi-institutional, DOE-funded center performing basic and applied science dedicated to improving yields of biofuels from cellulosic biomass

300+ People in 18 Institutions

Oak Ridge National Laboratory
University of Georgia
University of Tennessee
Dartmouth College
Georgia Institute of Technology
West Virginia University
ArborGen, LLC
Ceres, Incorporated
Mascoma Corporation

Samuel Roberts Noble Foundation
National Renewable Energy Laboratory
Cornell University
University of California—Riverside
North Carolina State University
University of California—Los Angeles
DuPont
GreenWood Resources
University of North Texas

2012–2013
Consolidated bioprocessing (CBP) will revolutionize how biomass is processed and converted.
Genetic block in lignin biosynthesis in switchgrass increases biofuel yields

Phenylalanine → PAL

Agrobacterium-mediated transformation of switchgrass

Ethanol yield

Wild-type switchgrass

Noble Foundation transgenic switchgrass

25% more ethanol

X. Fu and Z. Wang (Noble), J. Mielenz (ORNL), support from USDA/DOE
Genetic manipulation of lignin improves biofuel production from switchgrass

- Down-regulation of a single gene reduces recalcitrance with no apparent growth defects and leads to:
  - Increase in ethanol production by over one-third
  - Reduction in needed severity of pre-treatment
  - Evidence that biofuel processing costs can be reduced by at least 20% with 300-400% lower enzyme costs

Fu et al. PNAS, 2011
Lignin: A renewable low-cost feedstock

- **Characteristics**
  - Can be derived from trees (20–30% lignin) and other biomass
  - Currently a by-product of pulping and biorefineries
  - Readily available non-petroleum-based CF precursor
  - Potentially lowest cost precursor

Biomass selection → Plant genetics → Refining → Chemical processing → Material processing → Application
The case for lignin

- Abundant: 40M tpy can be extracted from Kraft mills with no adverse effect on mill balance (source: Peter Axegard, Innventia)
- Inexpensive: $0.03−$0.08/lb fuel value
- Procured sustainably
- Renewable and carbon rich
- Domestic
- Rural jobs
- Enhances biorefinery economics
## Potential lignin-derived products

<table>
<thead>
<tr>
<th>Product</th>
<th>Current technology status</th>
<th>Market risk</th>
<th>Challenges</th>
<th>Market volume</th>
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</thead>
<tbody>
<tr>
<td><strong>Hydrocarbon and aromatic chemicals</strong></td>
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<tr>
<td>Benzene/toluene/xylene</td>
<td>Partially developed</td>
<td>Low</td>
<td>Catalytic challenges: Selective dehydroxylation, demethoxylation and dealkylation</td>
<td>High</td>
</tr>
<tr>
<td>Phenol/substituted phenols</td>
<td>Partially developed</td>
<td>Low</td>
<td>-do- and Secondary derivatization of BTX chemicals</td>
<td>High</td>
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<tr>
<td>Aromatic polyols</td>
<td>Emerging</td>
<td>?</td>
<td>-do-</td>
<td></td>
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<tr>
<td>Biphenyls</td>
<td>Emerging</td>
<td>?</td>
<td>-do-</td>
<td>Moderate</td>
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<tr>
<td>Cyclohexane</td>
<td>Emerging</td>
<td>Low</td>
<td>-do-</td>
<td>High</td>
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<tr>
<td>Aromatic monomers</td>
<td>Emerging</td>
<td>?</td>
<td>Selective hydrogenolysis</td>
<td>?</td>
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<tr>
<td>Oxidized products (vanillin/DMSO)</td>
<td>Developed</td>
<td>High</td>
<td>Biocatalytic route for selective oxidation</td>
<td>Low</td>
</tr>
<tr>
<td>C1-C7 gases and mixed liquid fuels</td>
<td>Emerging</td>
<td>Low</td>
<td>Catalyst life, reduce process steps, process scale-up</td>
<td>High</td>
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<tr>
<td><strong>Macromolecules and their derivatives</strong></td>
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<tr>
<td>Carbon fiber</td>
<td>Partially developed</td>
<td>Moderate</td>
<td>Economic challenges: Isolation of lignin, spinning rate, carbon yield, varied lignin sources</td>
<td>High</td>
</tr>
<tr>
<td>Polymer extender</td>
<td>Partially developed</td>
<td>Moderate</td>
<td>Modification of lignin to compatibilize with polymer matrices, color of lignin-extended product</td>
<td>Moderate</td>
</tr>
<tr>
<td>Thermosets</td>
<td>Emerging</td>
<td>Moderate</td>
<td>Molecular weight and viscosity control of the product, varied lignin sources</td>
<td>?</td>
</tr>
<tr>
<td>Formaldehyde free adhesives</td>
<td>Partially developed</td>
<td>Moderate</td>
<td>Consistency of lignin to ensure constant cure rate</td>
<td>High</td>
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<tr>
<td><strong>Syngas products</strong></td>
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<tr>
<td>Methanol/dimethyl ether</td>
<td>Developed</td>
<td>Low</td>
<td>Technological challenges: Economic syngas purification, process scale-up</td>
<td>High</td>
</tr>
<tr>
<td>Ethanol/mixed alcohols</td>
<td>Emerging</td>
<td>Moderate</td>
<td>Economic syngas purification, catalyst and process improvement to produce 2-C alcohols, process scale-up</td>
<td>High</td>
</tr>
</tbody>
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Source: Holladay et al., Top Value-Added Chemicals from Biomass, PNNL-16983, October 2007
Current carbon fiber technology: From lab discovery to commercialization

**DOE Office of Science**
- Precursor chemistry and microstructure
- Process science: Microwaves create internal heating through material dipole vibrations

**DOE EERE**
- Melt spinning increases throughput with less energy and solvent
- Microwave/plasma-based conversion processes reduce residence time and energy demand

**Industry partnerships**
- CFTF: 25 ton/year pilot line facility
- Large IP portfolio available for licensing
- Developing key partnerships in multiple industries
### Carbon fiber product forms

<table>
<thead>
<tr>
<th>Continuous fibers</th>
<th>Unidirectional tapes and prepregs</th>
<th>Woven fabrics and prepregs</th>
<th>Chopped or milled fibers</th>
<th>Non-wovens</th>
</tr>
</thead>
</table>

Petroleum-based polyacrylonitrile (PAN)  
- Most common precursor  
- Cost fluctuates with crude oil prices

Textile PAN and polyolefin-based precursors  
- Lower cost  
- Petroleum-based

Lignin: A sustainable resource material  
- Commonly derived from wood via Kraft pulping  
- By-product of cellulosic ethanol production: Supply will increase as biorefineries are built  
- Cost is largely independent of oil prices

Today, almost half of carbon fiber manufacturing costs are associated with precursors
Keys to maximizing lignin carbon fiber value

- Tendencies:
  - Hardwood lignin melt spins well and stabilizes slowly
  - Softwood lignin stabilizes well but doesn’t readily melt spin
  - High purity is needed for melt spinning

Scalable achievement of fast conversion, high-rate spinning, and high yield has proven to be very elusive
Carbon fiber market

Automotive, wind energy, pressure vessels, oil and gas, and all other high-volume energy applications of carbon fiber composites are in the “industrial” market sector.

Cumulative carbon fiber demand by market sector

Costs and properties of structural carbon fibers

Automotive target
Strength: 1.72 GPa, modulus: 172 GPa

Best lignin CF (strength: 1.07 GPa, modulus: 83 GPa)

Typical ORNL lignin CF (strength: 0.48 GPa, modulus: 48 GPa)

DOE price target: $5−$7/lb

Fiber tensile strength, GPa

Fiber modulus, GPa

$15−$20/lb

$30−$40/lb

~$80/lb

~$10/lb
# Other potential markets for low-cost carbon fiber

<table>
<thead>
<tr>
<th>Market</th>
<th>Potential Benefits</th>
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</thead>
<tbody>
<tr>
<td>Civil infrastructure</td>
<td>Rapid repair and installation, time and cost savings</td>
</tr>
<tr>
<td>Biomass materials</td>
<td>Alternative revenue, waste minimization</td>
</tr>
<tr>
<td>Nontraditional energy</td>
<td>Geothermal, solar, and ocean</td>
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<tr>
<td>Non-aerospace defense</td>
<td>Light weight, higher mobility</td>
</tr>
<tr>
<td>Aerospace</td>
<td>Secondary structures</td>
</tr>
<tr>
<td>Power transmission</td>
<td>Less bulky structures, zero CLTE</td>
</tr>
<tr>
<td>Oil and gas</td>
<td>Offshore structural components</td>
</tr>
<tr>
<td>Vehicle technologies</td>
<td>Necessary for &gt;50% mass reduction</td>
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<tr>
<td>Wind energy</td>
<td>Needed for longer blade designs</td>
</tr>
<tr>
<td>Energy storage</td>
<td>Flywheels, Li-ion batteries, supercapacitors</td>
</tr>
<tr>
<td>Electronics</td>
<td>Light weight, EMI shielding</td>
</tr>
<tr>
<td>Pressurized gas storage</td>
<td>High specific strength</td>
</tr>
</tbody>
</table>

**Common issues**

- Fiber cost
- Fiber availability
- Design methods
- Manufacturing methods
- Product forms
Potential automotive market is huge for low-cost carbon fiber

Carbon fiber potential in 2017 at 50% of current price

<table>
<thead>
<tr>
<th>Global automotive production by car type</th>
<th>Expected vehicle production</th>
<th>Expected use of CF in cars</th>
<th>Demand for CF at 50% of current price (pounds)</th>
<th>Market for CF at 50% of current price ($M)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Super cars</td>
<td>6,000</td>
<td>100%</td>
<td>1.3 million</td>
<td>$7M</td>
</tr>
<tr>
<td>Super luxury cars</td>
<td>600,000</td>
<td>10%</td>
<td>101.2 million</td>
<td>$506M</td>
</tr>
<tr>
<td>Luxury cars</td>
<td>4 million</td>
<td>10%</td>
<td>202.4 million</td>
<td>$1,012M</td>
</tr>
<tr>
<td>Other/regular cars</td>
<td>92 million</td>
<td>1%</td>
<td>305 million</td>
<td>$1,525M</td>
</tr>
<tr>
<td><strong>Total</strong></td>
<td><strong>97 million</strong></td>
<td></td>
<td><strong>305 million</strong></td>
<td><strong>$1,525M</strong></td>
</tr>
</tbody>
</table>

Source: Lucintel, ACMA Composites 2012

3× current global CF demand for **all applications**; 10B lb potential automotive demand at full market penetration
State of structural lignin carbon fiber technology

- Mechanical properties:
  - Best strength and modulus to date ~ 155 ksi / 12 Msi (with softwood)
  - Typical strength and modulus ~ 70 ksi / 7 Msi
- Multi-filament tows not despoolable
- Single filaments that are moderately stretchable have been produced
- Lignin carbon fiber filaments exhibit evolving graphitic that is isotropic
  - Aligned crystallite morphology has not been reported
- Earliest development work dates to 1960s; DOE funding initiated in late 1990s
Lignin-based carbon fiber (LCF) meets performance requirements for high-temperature thermal insulation

18 in. diameter lignin GRI™ prototypes

Various GRI™ products machined into shapes

Other potential functional applications: Batteries, capacitors, sorbents (filtration, natural gas storage), fireproof fabrics

Figures courtesy GrafTech
Keys to structural properties: Possible solutions to the problem

Feedstock selectivity

- Identify preferred lignin characteristics (currently not well understood)
- Select and/or genetically engineer plants that produce lignin with preferred characteristics
- Mill selectivity
- Selective isolation/extraction/purification processes
- Molecular weight control (fractionation)
Chemistry is the “master key” to structural properties

Extensive chemical characterization is needed

<table>
<thead>
<tr>
<th>Blending</th>
<th>Doping</th>
<th>Derivatization</th>
<th>Modification</th>
<th>Cracking</th>
</tr>
</thead>
</table>

PAN based precursor is not 100% PAN

Accepted hyperbranched structure of lignin

Recently proposed linear structure of lignin (Crestini et al., *Biomacromolecules* 2011)

Lignin structure is a matter of debate
What is a composite?

- Combination of 2 or more constituent materials, each retaining its distinct phase, working together to deliver properties that cannot be achieved by either alone

- Common examples:
  - Paper (cellulose fibers in a binder)
  - Concrete (steel rods reinforcing cement matrix)
  - Particle board (wood chips reinforcing polymeric matrix)
  - Trees (cellulosic reinforcement in lignin matrix)

![Chopped fiber, random composite](courtesy Rutgers University)
![Woven carbon fabric]
![Continuous fiber composite with unidirectional lamina]
Manufacturing Matters

U.S. Manufacturing Sector

• Contributes about 12% of gross domestic product (GDP)
• Directly employs ~12 million people
• Accounts for 60% of U.S. engineering and science jobs
• Supplies about 57% of U.S. exports
• Produces nearly 20% of the world's manufacturing output

Manufacturing is the most diverse end-use sector—in terms of energy services required, sources of energy used, technologies needed, and product output.
We are focusing ORNL resources to support manufacturing initiative

• Manufacturing and materials R&D to:
  – Reduce the energy intensity of U.S. industry
  – Support development of new products
  – Strengthen our nation’s competitiveness and economic vitality

• Leveraging ORNL’s distinctive core capabilities
  – Neutron scattering
  – High-performance computing
  – Advanced materials
  – Advanced characterization

*MDF*: a multidisciplinary DOE-funded facility dedicated to enabling demonstration of next-generation materials and manufacturing technologies for advancing the US industrial economy

[www.ornl.gov/manufacturing](http://www.ornl.gov/manufacturing)
ORNL has exceptional resources for materials and manufacturing R&D

<table>
<thead>
<tr>
<th>National user facilities</th>
<th>Specialized capabilities</th>
<th>Secure work space</th>
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<tbody>
<tr>
<td>• Building Technologies Research and Integration Center</td>
<td>• Carbon Fiber Technology Center</td>
<td>• Multiprogram Research Facility</td>
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<tr>
<td>• Center for Nanophase Materials Sciences</td>
<td>• Center for Advanced Thin-film Systems</td>
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<tr>
<td>• High Flux Isotope Reactor</td>
<td>• Materials Processing</td>
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<tr>
<td>• High Temperature Materials Laboratory</td>
<td>• Metrology</td>
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<tr>
<td>• National Center for Computational Sciences</td>
<td>• Robotics and Energetic Systems</td>
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<tr>
<td>• National Transportation Research Center</td>
<td>• Sensors and Signals Research</td>
<td></td>
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<tr>
<td>• Spallation Neutron Source</td>
<td>• Thin Film Deposition and Analytical Facility</td>
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</table>

End-to-end research at a single site to reduce costs, accelerate innovation, and maximize capital investment culminating in prototypes.
MDF – integration of cutting-edge manufacturing technologies

Additive Manufacturing
- Metal Powder Bed Consolidation
- Direct Metal Deposition
- Polymer Extrusion
- Ultrasonic Consolidation

Carbon Fiber
- Precursor to Filament
- Filament to Carbon Fiber
- Carbon Fiber to Composite Material

Advanced Materials & Processing
- Lightweight Metals
- Roll-to-Roll
- Low-Temperature
- Transient Field

Automation & Controls
- Modeling & Simulation
- Sensing, Tracking & Measurement
- Communicating Wirelessly
- Systems Engineering

Manufacturing Demonstration Facility

Technology Impacts:
- Market Driven Adoption of New Materials and Processes
- Transformational & Cross-Cutting for Next-Generation Products

Market
Carbon Fiber Technology Center (CFTF) Delivers Unmatched Flexibility

- Highly instrumented, highly flexible conventional carbon fiber line for “any precursor in any format”
- Melt-spun fiber line to produce precursor fibers
- Provisions for additional future equipment
- Produce up to 25 tonnes/year of carbon fibers
- Demonstrate technology scalability
- Train and educate workers
- Work in partnerships with industry
Building a bridge from R&D to deployment and commercialization

Carbon Fiber Technology Facility (CFTF) roles

| Demonstrate low-cost carbon fiber (LCCF) technology scalability with the last scaling step before full-scale commercial production | Produce quantities of LCCF needed for large-scale material and process evaluations and prototyping |
CFTF Engages the Entire Composites Value Chain

RAW MATERIALS → PRECURSOR FIBERS → CONVERSION → COMPOSITE FORMULATION → TESTING & PROTOTYPING → APPLICATIONS

- **PAN**
  - Textile, HMW, SAF
  - **Solution Spinning**
  - **Highly Flexible Thermal Process**
  - **Atmospheric Pressure Plasma**
  - **Microwave Assisted Plasma**
  - **Plasma Surface Treatment**

- **PAN**
  - Melt-spun
  - **Melt Spinning**

- **Polyolefin**

- **Lignin**

CFTF in dashed area

Advanced Conversion Processes

- **Resin Design**
  - Matrix Formulation
  - Pre-pregging
  - Weaving
  - Pre-forming
  - Molding
  - Filament Winding
  - Curing
  - Etc.

Testing And Prototyping

End Users provide ORNL with cost & performance specs

STRUCTURAL
  - Vehicles
  - Wind
  - Defense
  - Aerospace
  - Oil & Gas
  - Infrastructure

NON STRUCTURAL
  - Flame Retardant
  - Energy Storage
  - Filtration
  - Thermal Mgmt.
  - Electrodes

OAK RIDGE CARBON FIBER COMPOSITES CONSORTIUM
Collaboration in Workforce Training

Pool of Candidates
- DOL grant funded
- Located at ORNL
- Industry focused training
- For qualified unemployed or under-employed

Technician Internship Program
- High-quality STEM learning experience
- Collaboration with researchers in field of interest
- Growth of S&T talent
- Hands-on experience on complex CF line
- Learn S&T underpinning ORNL research
- Develop skills directly transferrable to industry

Longer term Vision:
(i) Develop workforce training system for future carbon fiber manufacturing partners
(ii) Develop internship and other training programs from high school through university graduate level

Photo courtesy of Michael Patrick & Knoxville News-Sentinel
Our Mission

To accelerate the development and deployment of new lower cost carbon fiber composite materials, creating a new generation of strong, light-weight materials to enhance America’s economic competitiveness.

Established July, 2011
Value proposition

- Serves as a platform for industry to develop a pre-competitive technology roadmap to accelerate the development and deployment of strong, light-weight materials

- Enables industry to collaborate across the value chain to develop new precursors, explore new conversion technologies, and create new applications for low-cost carbon fiber composites
Currently at 50 Members and continuing to grow!

- 3M Company
- ABC Group Sales & Engineering
- Advanced Composites Group
- Alpha Industries
- Ashland
- ATK Launch Systems
- Barge Waggoner
- BASF Corporation
- Chomarat NA, LLC
- CIMV
- Composite Applications Group
- Continental Structural Plastics
- Cytec Carbon Fibers
- Domtar
- Dow Chemical Company
- DowAksa
- Despatch Industries
- Faurecia
- Fibria
- Ford Motor Company
- General Electric
- Georgia-Pacific
- Global Composites Solutions
- Graftech International
- Hanwha Azdel
- Harper International
- Hills, Inc.
- Innovation Valley Inc.
- Innventia
- INOAC USA
- Lignol Innovations
- Materials & Chemistry Laboratory
- Metalsa Structural Products
- Metso Power
- NovusFolium
- Plasan Carbon Composites
- Renmatix
- Sabic Innovative Plastics
- SGL Carbon Fibers
- Sodra
- Southern University
- SSOE Group
- Steelcase
- TennEra
- Toho Tenax America
- United Technologies Research Center
- USEC
- UT-Battelle
- Virdia, Inc.
- Volkswagen Group of America
# Grand vision

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<tbody>
<tr>
<td>Extensive characterization</td>
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<td>Extensive characterization</td>
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<td>10 tons of structural bio-CF produced</td>
<td>The vision is realized</td>
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<tr>
<td>Biomass engineering and extensive chemistry</td>
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<tr>
<td>Process and property optimization</td>
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<td>Bio-CF production and component/system prototyping</td>
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## Milestones

<table>
<thead>
<tr>
<th>Papers in high-impact journals</th>
<th>Prototype suppliers selected</th>
<th>Raw materials: Tensile properties</th>
<th>Bio-CF produced</th>
<th>10 tons of vehicles and blades</th>
</tr>
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<tbody>
<tr>
<td></td>
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<td>1% strain 14 Msi modulus</td>
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<td></td>
<td>1% strain 22 Msi modulus</td>
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<tr>
<td></td>
<td></td>
<td>1% strain 32 Msi modulus</td>
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</tbody>
</table>

- 14 Msi modulus
- 22 Msi modulus
- 32 Msi modulus

- VIPs drive bio-CF intensive vehicle(s) to Washington, DC
- Field test a set of wind turbine blades with bio-CF structure