Project Objective

Goal:
• Develop and demonstrate lignin-based carbon fibers in high-temperature applications. Focus on melt processing of fibers.
  • Rigid insulation, felt insulation, graphite electrodes, filtration

Problem:
• No one has yet developed or commercialized scalable and cost-effective lignin precursor technology and demonstrated that fibers are drop-in high-value replacement for fibers in an existing application

Challenge:
• Lignin has a complicated chemical structure and is difficult precursor
• Developed technology must work with multiple lignin types
• Technology must be scalable, cost-effective, and be better than current
• Insulation is cost driven product. Innovation needed to compete
Today there are no commercial lignin-based carbon fibers
Only academic studies reported with focus on improving strength
Lignin is being sold into primarily chemical and agricultural applications. Off-take agreements needed to advance lignin supply

Our technical approach works with and not against the lignin structure
- **Application-driven approach**: focus on “functional” properties not structural properties
- **Cost-driven approach**: focus on meeting cost metrics
- **Process-driven approach**: focus on scalable technology
Technical Approach

- Focus low-cost chemical modification of lignin structure
  - BP1 – develop and demonstrate lab scale precursors and fibers
  - BP2 – produce 500 lbs and demonstrate in product prototypes
  - BP3 – produce 5000 lbs and demonstrate in end use environment
- GTI
  - Over 130 years of carbon and graphite product experience
  - USA manufacturing base for insulation but global customer base
- ORNL
  - Over 20 years experience with lignin precursor R&D and scale-up
  - World class precursor labs and fiber spinning capabilities
  - ORNL CFTF key component of scale-up assessment
- Risks
  - Technology does not work – pursue and downselect from parallel paths
  - Technology does not meet cost – focus on low cost chemical treatment
  - Technology does not scale-up – focus on scalable chemical treatment
Technical Approach

Lignin powder → Lignin pellets → Lignin fiber mat by melt processing

Lignin chemical treatment

Treatment enhances stabilization throughput

Lignin mat after stabilization → Lignin fiber after carbonization and milling

Insulation
Graphite electrodes
Filtration
Transition and Deployment

- Carbon fiber is expensive. Many applications are cost driven.
- Selected applications need functional not structural properties
- Lignin carbon fibers could be low cost and high performance

Applications
- High-temp insulation (rigid and felt)
- Graphite electrodes
- Filtration (activated fibers)

Commercialization approach
- Develop scalable lignin precursor technology
- Demonstrate high throughput melt processing of lignin
- Demonstrate lignin carbon fibers in prototype products
- Validate products in customer environment

Sustainment
- Development of scalable lignin fiber technology platform
- Qualification of multiple lignin types (hardwood, softwood)
GRI™ Insulation
- Rigid, high-temperature insulator
- 100% bonded carbon fiber
- Standard and purified grades
- Optional anti-dust coating
- Panels and machined parts

Applications
- Vacuum furnaces
- Brazing furnaces
- Sintering furnaces
- Crystal growth furnaces

Preliminary Lignin-based Carbon Fiber Cost Modelling (Production Cost)

Main Effects:
- Carbon yield
- Precursor cost
- Stabilization time

Transition and Deployment
Measure of Success

- Insulation is price driven market
  - Target for est. lignin carbon fiber production cost is $5.00/pound
  - Customers expect low cost and high thermal efficiency
  - Customers expect high purity (ash in product less than 500 ppm)

- Existing insulation product lines targeted for immediate impact
  - Drop-in replacement for current rayon and pitch foreign-sourced fibers

- Lower-cost lignin fibers could have several impacts:
  - Displacing rayon and pitch fibers with USA-based fibers
  - Lower cost product, increasing market competitiveness
  - Better insulation product, increasing energy efficiency
  - Lower CO2 vs PAN: lignin is already polymerized and can be melt processed
  - Jump starting lignin fibers for other applications

- Benefits: cost, higher performance, energy efficiency, CO2 avoidance
Project Management & Budget


- BP1 has 4 main tasks and milestones. Ending 6/21/2016
  - Identify and screen at least 10 different lignin samples
  - Show at least 4 that meet process and cost criteria ($5.00/pound)
  - Also must meet purity level (ash < 500 ppm in final fiber product)

- BP 2 has 4 mains tasks and milestones

- BP3 has 4 main tasks and milestones

<table>
<thead>
<tr>
<th>Total Project Budget</th>
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<tbody>
<tr>
<td>DOE Investment - GTI</td>
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<tr>
<td>DOE Investment - ORNL</td>
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<tr>
<td>Cost Share</td>
</tr>
<tr>
<td>Project Total</td>
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</table>
Results and Accomplishments

- **BP 1** will finish 6/21/2016
- Significant accomplishments on precursor and process at lab scale
  - Developed Low-cost scalable chemical treatment to accelerate stabilization
  - Works on hardwood, softwood, and grasses, but to different degrees
  - Accelerated thermal stabilization of less than 3 hours achieved for all
  - Stabilized fibers can be carbonized with acceptable yield
  - Carbonized fibers can be milled to suitable fibers for insulation
- Currently evaluating scale up from gram scale to kilogram scale

<table>
<thead>
<tr>
<th>ID</th>
<th>Sample</th>
<th>Chemical Treatment</th>
<th>Melt Spin Temp</th>
<th>Best Stabilization Time To Date</th>
<th>Carbon Yield</th>
<th>Est. Production Cost</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>HW orgnsolv</td>
<td>YY + ZZ</td>
<td>155 - 170 °C</td>
<td>3 hours</td>
<td>44%</td>
<td>$4.99/lb</td>
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<tr>
<td>4</td>
<td>SW kraft purified</td>
<td>WW + YY + ZZ</td>
<td>130 °C</td>
<td>1.5 hours</td>
<td>41%</td>
<td>$5.00/lb</td>
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<td>5</td>
<td>Grass kraft purified</td>
<td>WW+ZZ + YY</td>
<td>127-133 °C</td>
<td>2 hours</td>
<td>42%</td>
<td>$5.03/lb</td>
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<tr>
<td>DD</td>
<td>DD</td>
<td>YY + ZZ</td>
<td>136 °C</td>
<td>2.5 hours</td>
<td>44%</td>
<td>$4.94/lb</td>
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</tbody>
</table>
Results and Accomplishments

Microscopy of Stabilized Fibers at Lab Scale

HW organosolv – 3 hours

Softwood kraft purified – 1.5 hours

Grass kraft purified – 2 hours

Lignin #DD – 2.5 hours
Results and Accomplishments

Microscopy of Carbonized and Milled Fibers at Lab Scale

- Hardwood organosolv
- Softwood kraft purified
- Grass kraft purified
- Lignin #DD
Results and Accomplishments

Microscopy of Carbonized and Milled Fibers at Lab Scale

- Hardwood organosolv
- Softwood kraft purified
- Grass kraft purified
- Lignin #DD