2017 CROSSCUTTING Research Project Review
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REDUCED MODE SAPPHIRE FIBER AND DISTRIBUTED SENSING SYSTEM
DE-FE0012274

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Overview

- Motivation, Objectives, and Technical Challenges
- Research Approach and Technology
  - LMV Sapphire Fiber Design and Fabrication
  - Distributed Temperature Sensing System
- Milestones and Schedule
- Impact and Achievements
- Next Steps
Motivation

• Eliminate barriers to the seamless integration of fiber optic sensing technologies in power plants

• Improve the operating efficiencies and safety of power plants via the real time and distributed sensing of temperature
Project Objectives

• **Goal**: Develop a Raman scattering distributed temperature sensing system based on a low modal volume (LMV) sapphire fiber sensor.

• **Objective**: Design, fabricate and characterize a sapphire fiber that limits the number of guided modes.

• **Objective**: Develop a prototype, fully-distributed sensing system and evaluate its performance in a laboratory test environment for operation at temperatures over 1000ºC.

• **Benefit**: The proposed sapphire fibers and sensors will allow for the seamless integration of mature fiber optic sensing technologies in new power plant control systems.
Technical Challenges

• Performance of single crystal sapphire fibers
  • Large “core” diameters
  • High numerical aperture (NA)
  • High loss
  • Weak Raman signal in sapphire fiber
• High operating temperatures
  • Thermal radiation generated by the sapphire fiber
  • Thermal radiation coupled into the fiber end
• Achievable spatial resolution
  • Pulse width
  • Modal dispersion
TECHNOLOGY & APPROACH
Research Approach

• Design and fabricate a single crystal sapphire fiber with a modal volume optimized for sensor applications
  • Wet acid etching at elevated temperatures
  • “Bundled” photonic crystal sapphire fiber design
  • Sapphire fiber growth via LHPG

• Design and construct a Raman scattering distributed temperature sensing system
  • Interrogation at 532 nm
  • Design and component optimization
  • Performance testing
RESEARCH PROGRESS:
LMV SAPPHIRE FIBER DESIGN
AND FABRICATION
LMV Sapphire Fiber Fabrication

Fabrication via Wet Acid Etching

- Sulfuric/phosphoric acid solutions
  - Studied and optimized concentrations
- Elevated temperatures (>200°C)
  - Determined etch rates
  - Determined activation energies
  - Studied a-plane vs. c-plane
- Extended lengths (~ 1m)
- Improved surface quality
  - Eliminated surface deposits
- Simple, cost effective, scalable
- Potential new applications
  - Gas sensing, inclined tip sensing
LMV Sapphire Fiber Fabrication

Equipment and Techniques

Custom Etching System

Excellent Temperature Control

Temperature and Etching Uniformity

<table>
<thead>
<tr>
<th>Sample</th>
<th>Top Diameter (µm)</th>
<th>Bottom Diameter (µm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>A</td>
<td>82.0</td>
<td>73.1</td>
</tr>
<tr>
<td>B</td>
<td>80.9</td>
<td>73.4</td>
</tr>
<tr>
<td>C</td>
<td>79.6</td>
<td>74.4</td>
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<tr>
<td>Average</td>
<td>80.8</td>
<td>73.6</td>
</tr>
</tbody>
</table>

Standard Deviation

1.2

0.7
LMV Sapphire Fiber Fabrication
Understanding and Control

Optimization of Etching Solution

Etch Rates

[Graph showing etch rate vs. temperature and etch rate vs. time]
LMV Sapphire Fiber Fabrication
Characterization of Surface Quality

Optical Microscopy
(fiber diameter, contamination, polarization states, non-uniformities)

Scanning Electron Microscopy (SEM)
Energy-dispersive X-ray spectroscopy (EDAX)
(fiber diameter, contamination, composition/elemental analysis, defects, non-uniformities)
LMV Sapphire Fiber Testing

Modal Volume Measurement

- Three different wavelengths (532nm, 782.9nm, 982.9nm)
- Focused into connector using direct free-space coupling
  - Overfilled using objective lens with NA=0.66
- Sample mounted on 3-axis stage
- CCD camera beam profiler mounted on 3-axis stage
- Polished fiber tip (100 nm lapping film)
LMV Sapphire Fiber Testing

Far Field Analysis Method

- Far-field intensity patterns capture
  - Prior to etching
  - Post etching and polishing
- Modal interference and superposition yields a “speckled” appearance
- Reduction in diameter and modal volume
  - Number of power peaks (speckles) decreases
  - Relative diameter of individual speckles increases
  - Modal interference and superposition due a decrease in the number of supported modes
- Qualitative analysis of modal volume
- Low order mode profiles are visible
The trend in modal volume reduction with a reduction in fiber diameter and increase wavelength agrees with theoretical predictions.
LMV Sapphire Fiber Testing

NA Characterization Techniques

- Vary angle of waveguide tip with stationary photodetector (TIA standard)
  - Requires both ends of fiber to be connected
  - Requires decent fiber length (>1/3m)
- Vary input NA and measure output power
  - Assumes all intensity effects are NA-dependent
- Beam diameter differential
  - Overfill fiber (all modes are excited)
  - Measure beam width twice with known distance between
  - Vergence angle calculated from beam width differential
  - Requires consistent beam projection to be accurate (single mode)
LMV Sapphire Fiber Testing

Theoretical vs. Effective NA

- The measured (“effective”) NA can deviate significantly from the theoretically calculated value
  - Non-ideal geometry (i.e. non-circular cross section)
  - Small core diameter
  - Inefficient coupling, surface scattering, angled end faces

- Beam width differential method
  - CCD camera beam profiler (Thorlabs BC106-VIS)

<table>
<thead>
<tr>
<th>Wavelength</th>
<th>$\text{NA}_{\text{th}}=1.4$</th>
<th>$\text{NA}_{\text{eff}}=0.09$</th>
<th>6.5μm</th>
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</thead>
<tbody>
<tr>
<td>532nm</td>
<td>1614</td>
<td>2</td>
<td></td>
</tr>
<tr>
<td>783nm</td>
<td>736</td>
<td>1</td>
<td></td>
</tr>
<tr>
<td>983nm</td>
<td>461</td>
<td>1</td>
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</table>
Laser Heated Pedestal Growth

Basic Components

- Beam Steering Optics
  - Imaging System
  - HeNe Alignment Laser
  - Polarizer-Attenuator-Analyzer
  - Gold Coated Copper Mirrors
  - Beam Expander

- Growth Chamber Optics
  - Aluminum Optics
  - In-house design and polishing
  - Reflaxicon, Scraper Mirror, Spherical Mirror

- Mechanical Drawing System
  - Synchronized Linear Stages
Laser Heated Pedestal Growth
Automatic Diameter Control System

Diameter variations ~1.7% were readily achieved
LMV Sapphire Fiber

*Summary of Results*

- **For the first time**, a submicron single crystal sapphire fiber for the propagation of lower order modes was fabricated via wet-acid etching.
- Few mode operation was demonstrated, for the *first-time*, in a single crystal sapphire fiber.
- Reduction of the “effective” NA and modal volume was verified via an array of characterization techniques and test parameters.
- A fully operational LHPG system was designed and constructed in-house for the fabrication of unique sapphire fiber structures.
RESEARCH PROGRESS: DISTRIBUTED TEMPERATURE SENSING SYSTEM
Operating Wavelength Selection

Blackbody Radiation

The Raman intensity of the Anti-Stokes and Stokes components is proportional to its differential cross section given by (M. Hobel, Applied Optics, 1995)

\[
\frac{d\sigma_{AS}}{d\Omega} \left| \right. x \approx \frac{1}{\lambda_{AS}^4} \frac{1}{\exp \left[ \frac{hc\Delta\nu}{K_BT(x)} \right] - 1}
\]

\[
\frac{d\sigma_{S}}{d\Omega} \left| \right. x \approx \frac{1}{\lambda_{S}^4} \frac{1}{1 - \exp \left[ -\frac{hc\Delta\nu}{K_BT(x)} \right]}
\]

According to Planck’s law, the radiation can be calculated as followed (M. Planck, P. Blakiston’s Son & Co, 1914)

\[
B(T) = \frac{2hc^2}{\lambda^5} \frac{1}{\frac{hc}{e^{\frac{hc}{\lambda k_BT}} - 1}}
\]
Scattering in Sapphire Fiber

Experimental Set-Up

- Experimental setup for Raman scattering detection.
- Temperature distribution along the sapphire fiber.

B. Liu et al, Optics Letters, 2015
Scattering in Sapphire Fiber

Temperature Dependence

Spectrum

Peak Intensity

B. Liu, Optics Letters, 2015
Scattering in Sapphire Fiber

Temperature Dependence

Peak Frequency

Temperature dependence of sapphire Raman position

Peak Width

Temperature dependence of sapphire Raman width

B. Liu, Optics Letters, 2015
Raman DTS System Design

Experimental Set-Up

B. Liu et al, Optics Letters, 2016
Raman DTS System Performance

1 meter Sapphire Fiber

B. Liu et al, Optics Letters, 2016
Raman DTS System Performance

1 meter Sapphire Fiber

Stokes & AntiStokes VS Temperature

Normalized Intensity (a.u.)

Temperature (°C)

Stokes

AntiStokes

Raman Ratio

Temperature (°C)

Temperature-rise

Temperature-fall

Curve fitting

Standard deviation

<table>
<thead>
<tr>
<th>Temperature-rise</th>
<th>Temperature-fall</th>
<th>Curve fitting</th>
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</thead>
<tbody>
<tr>
<td>3.4466</td>
<td>3.5239</td>
<td>3.5211</td>
</tr>
<tr>
<td>4.5177</td>
<td>3.5828</td>
<td>4.3905</td>
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<tr>
<td>4.2505</td>
<td>3.5218</td>
<td>4.0517</td>
</tr>
</tbody>
</table>

Standard Deviation: 3.7°C (3.0°C)

B. Liu et al, Optics Letters, 2016
Raman DTS System Performance

1 meter Sapphire Fiber

Temperature profile along the fiber:
- Blue: 30 °C
- Green: 100 °C
- Red: 200 °C
- Cyan: 300 °C
- Pink: 400 °C
- Yellow: 500 °C
- Black: 600 °C
- Blue: 700 °C
- Magenta: 800 °C
- Red: 900 °C
- Teal: 1000 °C
- Magenta: 1100 °C
- Yellow: 1200 °C

Demodulated signal at heating center:
- Red: Raman DTS
- Blue: Thermal couple

Time (minutes)

Distance (m)

Temperature profile along the fiber

Demodulated temperature (°C)
Raman DTS System Performance

2 meter Sapphire Fiber

Stokes VS Temperature

Normalized Stokes (a.u.)

Distance (m)

AntiStokes VS Temperature

Normalized AntiStokes (a.u.)

Distance (m)
Raman DTS System Performance

3 meter Sapphire Fiber

Stokes VS Temperature

AntiStokes VS Temperature

Joint reflection
Raman DTS System Performance

Spatial Resolution

- Sensing length: 3 meters
- Temperature: 1400ºC
- Spatial resolution: 16.4 cm
  - Determined via 10% to 90% response distance
Raman DTS System

Summary of Results

• Raman fully-distributed ultra-high temperature sensing technique, a **first-of-its-kind** technology, was successfully demonstrated.

• A temperature standard deviation of 3.0°C (0.2% of full scale) was demonstrated in a 1 meter sapphire fiber.

• A maximum operating temperature of 1400°C was demonstrated (upper limit has yet to be determined)

• A spatial resolution <20 cm was achieved with a fiber sensing length of 3 m.
MILESTONES AND SCHEDULE
Project Milestones

- LMV sapphire fiber
  - Demonstrated design feasibility
  - Developed fabrication processes
  - Demonstrated fiber performance

- Fully-distributed temperature sensing system
  - Demonstrated design feasibility
  - Characterized Raman scattering response in sapphire fibers
  - Demonstrated system performance in fused silica optical fibers
  - Demonstrated prototype system in single crystal sapphire fibers

<table>
<thead>
<tr>
<th>M. #</th>
<th>Title/Description</th>
<th>Planned Completion Date</th>
<th>Actual Completion Date</th>
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<tr>
<td>3</td>
<td>Demonstration of LMV Sapphire Fabrication</td>
<td>6/30/2015</td>
<td>6/30/2015</td>
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<tr>
<td>4</td>
<td>Demonstration of Sensing System</td>
<td>12/31/2015</td>
<td>12/31/2015</td>
</tr>
<tr>
<td>6</td>
<td>Final Report</td>
<td>12/31/2016</td>
<td>85%</td>
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Milestone Success Criteria

- **LMV sapphire fiber**
  - Demonstrated significantly reduce modal volume (<< 50%)
    - Few mode and single mode operation
  - Minimum bend radius << 4 mm
    - Bend radius < 100 µm
- **Fully-distributed temperature sensing system**
  - Sapphire sensing lengths of 1, 2, and 3 meters
  - Spatial resolutions < 17 cm
  - Operating temperature ~ 1400°C

<table>
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<tr>
<th>ID</th>
<th>Title</th>
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<th>Result</th>
<th>M.S.</th>
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<th>Actual Completion</th>
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<tr>
<td>SC1</td>
<td>System Modeling</td>
<td>1. 50% modal volume reduction</td>
<td>1. &gt;&gt; 50% modal volume reduction</td>
<td>M2</td>
<td>12/31/2014</td>
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<tr>
<td></td>
<td></td>
<td>2. Sensing length of 3 m</td>
<td>2. Sensing length of 3 m</td>
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<td>3. Resolution of &lt; 20 cm</td>
<td>3. Resolution &lt; 17 cm</td>
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<tr>
<td>SC2</td>
<td>LMV Sapphire Fiber</td>
<td>1. 40% reduction in modal volume</td>
<td>1. &gt; 50% modal volume reduction</td>
<td>M3</td>
<td>6/30/2015</td>
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<td></td>
<td></td>
<td>2. Attenuation &lt; 6 dB/m @ 355 nm</td>
<td>2. Attenuation &lt; 8 dB/m @ 532 nm</td>
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<td></td>
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<td>3. Minimum bend radius &lt; 25 mm</td>
<td>3. Minimum bend radius &lt; 4 mm</td>
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<tr>
<td>SC3</td>
<td>Distributed Sensing System</td>
<td>1. Sensing length of 2 m</td>
<td>1. Sensing length of 3 m</td>
<td>M4</td>
<td>12/31/2015</td>
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<tr>
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<td>2. Resolution &lt; 20 cm</td>
<td>2. Resolution &lt; 10 cm</td>
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<tr>
<td></td>
<td></td>
<td>1. Sensing length of 3m</td>
<td>1. Sensing length of 3 m</td>
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<tr>
<td></td>
<td></td>
<td>2. Resolution &lt; 20 cm</td>
<td>2. Resolution &lt; 17 cm</td>
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</table>
Review Panel Recommendations

- All Recommendations have been addressed
- On schedule and budget

<table>
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<th>RPR #</th>
<th>Title/Description</th>
<th>Planned Completion Date</th>
<th>Actual Completion Date</th>
<th>Verification Method</th>
<th>Comments (progress toward achieving milestone, explanation of deviation from plan, etc.)</th>
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<tr>
<td>R1</td>
<td>Material Characterization of Pre- and Post-Etched Sapphire Fibers</td>
<td>9/30/2016</td>
<td>9/30/2016</td>
<td>DOE Approval</td>
<td>Completed</td>
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<tr>
<td>R2</td>
<td>&quot;Back of the Envelope&quot; Calculations to Predict Fiber and System Performance</td>
<td>3/30/2016</td>
<td>3/30/2016</td>
<td>DOE Approval</td>
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<td>R3</td>
<td>Identify a &quot;Back-up Approach&quot;/Alternative Strategies</td>
<td>12/30/2016</td>
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<td>DOE Approval</td>
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<td>R4</td>
<td>Engage Crystal Growth Experts for LHPG</td>
<td>9/30/2016</td>
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<td>R5</td>
<td>Evaluate Consistencies between Theoretical Analyses and Experimentation/Manufacturability</td>
<td>12/30/2016</td>
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<td>DOE Approval</td>
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## Project Schedule

### GANT CHART

<table>
<thead>
<tr>
<th>Task #</th>
<th>Task Name</th>
<th>Project Year 1</th>
<th>Project Year 2</th>
<th>Project Year 3</th>
<th>Ext. Year</th>
<th>Planned Start Date</th>
<th>Planned End Date</th>
<th>Actual Start Date</th>
<th>Actual End Date</th>
<th>Comments</th>
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<tbody>
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<td>M1</td>
<td><strong>MILESTONE 1</strong>*</td>
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<td>Q4</td>
<td>1/1/2014</td>
<td>5/15/2014</td>
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<td>12/30/2014</td>
<td>4/1/2014</td>
<td>12/30/2014</td>
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<td>4.2</td>
<td>Distributed Sensing System Building and Demonstrating with Silica Fiber</td>
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<td></td>
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<td>5/1/2014</td>
<td>12/29/2015</td>
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<td>5.1</td>
<td>Construct Prototype LMV Sapphire Fiber</td>
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<td>9/1/2015</td>
<td>1/4/2016</td>
<td>9/1/2015</td>
<td>5/1/2016</td>
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<td>5.2</td>
<td>Demonstrate Distributed High Temperature Sensing with LMV Sapphire Fiber</td>
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<td>1/14/2016</td>
<td>5/18/2016</td>
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<td>6</td>
<td>Construct &amp; Evaluate Refined Prototype Sensing System</td>
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<td>6/30/2016</td>
<td>11/30/2016</td>
<td>7/16/2016</td>
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<td>On Schedule</td>
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</tbody>
</table>

### KEY PROJECT DATES

- **Completed**: Task is completed as planned.
- **On Schedule**: Task is on schedule.
- **No Cost Extension Granted**: No cost extension granted for the task.

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[Image: GANT CHART diagram]
IMPACT, ACHIEVEMENTS, AND NEXT STEPS
Research Impact

• Technical Achievements
  • Fabrication of sub-micron single crystal sapphire fiber
  • Observation of Raman Stokes and Anti-Stokes peaks in sapphire fiber
  • Measurement of fiber attenuation in the time domain in sapphire fiber
  • Distributed Raman temperature measurements in sapphire fiber
  • Demonstrated few to single mode operation in sapphire fiber

• Student Support
  • Full Support: Cary Hill (Ph.D., ’16), Bo Liu (Ph.D., ’17), Yujie Cheng (Ph.D., ‘17)
  • Partial: Adam Floyd (Ph.D., ‘17), Jiaji He, (Ph.D., TBD), Hanna Heyl (Ph.D., TBD), Shuo Yang, (Ph.D., ‘19), Amiya Behera (Ph.D, ‘17) Chennan Hu (Ph.D., TBD), Sunny Chang (M.S., ‘16), Elizabeth Bonnell (M.E., ‘16)

• Faculty Training & Development
  • Zhihao Yu (Post-doc)
  • Daniel Homa (Research Scientist)
  • Haifeng Xuan (Research Associate)
  • Chenyuan Hu (Post-doc)
Research Products

• Peer Reviewed Publications

• Intellectual Property
Project Performance

• All Project Milestones Met On Time and On Budget
• All Success Criteria Met On Time and On Budget
• “First of Its Kind” Technologies
  – Fabrication of sub-micron single crystal sapphire fiber
  – Fabrication and demonstration of single mode sapphire fiber
  – Observation of Raman Stokes and Anti-Stokes peaks in sapphire fiber
  – Measurement of fiber attenuation in the time domain in sapphire fiber
  – Distributed Raman temperature measurements in sapphire fiber
• Dissemination of Findings
  – 5 peer reviewed publications, 1 submitted, 2 planned
  – 2 provisional patents filed
• Graduate Student Support (11)
• Faculty Training and Development (4)
Next Steps

- Compose manuscripts and submit for publication
- Generate and submit Final Report
- DTS system integration with LMV sapphire fiber
- Process development and optimization of LHPG system
- Submit continuation application
- Evaluate additional research opportunities for fiber and sensing technologies
Acknowledgements

**Virginia Tech**
- Center for Photonics Technology (CPT)
- Gary Pickrell
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- Yujie Cheng
- Sunny Chang
- Elizabeth Bonnell
- Hann Heyl
- Zhiting Tian
- Haifeng Xuan
- Robert Blackwell
- Amy Hill, Jiaji He, Shuo Yang, Amiya Behera, Chennan Hu, Cindy Purdue, Chenyuan Hu, Nevada Davis

**Department of Energy**
- National Energy Technology Laboratory
- Project Manager: Jessica Mullen
- Sydni Credle
- Susan Maley*

*Now with Electric Power Research Institute*
THANK YOU FOR YOUR TIME