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
  • Field Deployable Fiber Optic Sensing System
• Milestones and Schedule
• Impact, Achievements and 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, 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
Research Approach

• Design and fabricate a single crystal sapphire fiber with a modal volume optimized for sensor applications
  • Perform waveguide analysis of fiber designs
  • Wet acid etching at elevated temperatures

• Design and construct distributed temperature sensing system(s)
  • Develop complimentary distributed sensing schemes
    • Raman backscatter
    • Fiber Bragg gratings
  • Conduct performance testing

• Field trails testing in operating environment
  • Design and fabrication of harsh environment sensor packaging
  • Sensor deployment and operation
RESEARCH PROGRESS:
LMV SAPPHIRE FIBER DESIGN
AND FABRICATION
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

Optimization and Control

<table>
<thead>
<tr>
<th>Sample</th>
<th>Top Diameter (µm)</th>
<th>Bottom Diameter (µm)</th>
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<tbody>
<tr>
<td>A</td>
<td>82.0</td>
<td>73.1</td>
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<td>1.2</td>
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Sample Temperatures (°C)

- [Graph showing etch rate vs. 1/T (K)]
- [Graph showing temperature vs. time (hr:min:sec)]
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
Mechanical Strength via Bend Radius

<10μm bend radius

100μm
LMV Sapphire Fiber Testing

Far Field Analysis Method

- Far-field intensity patterns capture
  - Prior to etching
  - Post etching and polishing
  - Three different wavelengths (532nm, 782.9nm, 982.9nm)
- 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
- NA measurements performed via the beam width differential method
LMV Sapphire Fiber Testing

Far Field Analysis of RDSF

The trend in modal volume reduction for a reduction in fiber diameter and increase in wavelength agrees with theoretical predictions.
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>$N_{A_{\text{theor.}}}=1.4$</th>
<th>$N_{A_{\text{eff.}}}=0.09$</th>
<th>6.5$\mu$m</th>
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<tr>
<td>532nm</td>
<td>1614</td>
<td>2</td>
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<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

Single crystal sapphire fiber fabrication

Diameter variations ~1.7% were readily achieved
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 SYSTEMS
RAMAN BACKSCATTERING BASED TEMPERATURE SENSING
Operating Wavelength Selection

Signal Quality and 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)

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

\[
\frac{d\sigma_S}{d\Omega} \bigg|_x \approx \frac{1}{\lambda_S^4} \frac{1}{1 - \exp \left[ - \frac{hc\Delta \nu}{K_B T(x)} \right]}
\]

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

\[
B(T) = \frac{2hc^2}{\lambda^5} \frac{1}{\exp \left( \frac{hc}{\lambda k_B T} \right) - 1}
\]
Scattering in Sapphire Fiber

Characterization of Raman Spectra

Bo Liu, Zhihao Yu, Zhipeng Tian, Daniel Homa, Cary Hill, Anbo Wang, and Gary Pickrell.
Raman DTS System Design

Experimental Set-Up

Raman DTS System Performance

1 meter Sapphire Fiber

Temperature profile along the fiber

Demodulated signal at heating center

Raman DTS System Performance
Sensing Length, Temperature and Spatial Resolution

- Sensing length: 3 meters
- Temperature: 1400ºC
- Spatial resolution: 16.4 cm
  - Determined via 10% to 90% response distance
FIBER BRAGG GRATING BASED TEMPERATURE SENSING
FBG Sensor Fabrication

Point-by-Point Method

- FBG created by localized refractive index changes distributed along the fiber
  - Inscription via 780 nm (IR-fs) laser
  - Phase matching condition:
    \[ m\lambda_{\text{Bragg}} = 2n_{\text{eff}}\Lambda \]

- Pitch controlled by the relation between the moving speed and the repetition rate
- Length adjusted by the total number of laser pulses
- Unique advantages over phase mask method
  - Geometrical and design flexibility
  - Wavelength division multiplexing (WDM) can be readily implemented
FBG Sensor Fabrication

FBG Interrogation Technique

FBG Sensor Fabrication
Enhancement of Reflectivity via Thermal Annealing

FBG reflectivity was permanently enhanced by about 5.5 times

FBG Sensor Fabrication

Wavelength Multiplexed FBG Array
FBG Sensor Fabrication

Reduced Diameter Sapphire Fiber Bragg Grating

Step 1: Point-by-Point FBG Fabrication

- Pulse Chain, $f_{rep}$
- Index Matching Oil
- Moving, $v_s$

\[ \Lambda = \frac{v_s}{f_{rep}} \]

Step 2: Hot-Wet Acid Etching

- Glass Shield
- Glass Shielded Thermocouple
- Heating Elements
- $\text{H}_2\text{SO}_4 + \text{H}_3\text{PO}_4$ (3:1 Molar)
- Glass Container
- $330 ^\circ\text{C}$
- ~4 cm

Top view

- 3 μm
- 1.77 μm
- 1.81 μm

Side view

- 3.28 μm

Diameter (μm) vs. Time (hour)

- Slope: -2.7 μm/h

Measured Points

Linear Fitting
**FBG Sensor Performance**

*Reduced Diameter Sapphire Fiber Bragg Grating*

**FBG spectral response**

Reduced modal volume improves FBG peak fidelity

Distributed Temperature Sensing Systems

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
- Ultra-high temperature sensing was demonstrated, for the first time, with FBGs inscribed in single crystal sapphire fiber via the point-by-point method
  - FBG reflectivity enhancement and stabilization technique was demonstrated
  - A maximum operating temperature of 1400°C was demonstrated (upper limit has yet to be determined)
HARSH ENVIRONMENT SENSOR
PACKAGING AND DEPLOYMENT
Harsh Environment Packaging

Fiber in Ceramic Tube (“FICT”)

- Inherent design and material flexibility for application specificity
- Constructed from commercially available “off-the-shelf” components
Performance Testing

High Temperature Exposure

Laboratory Scale Test Set-Up

Temperature Sensor(s) Response

- Furnace
- Data Recording
- Fiber Coupler
- SLED
- Optical Spectrum Analyzer

Graph:
- FBG 1
- FBG 2
- FBG 3

Measured Temperature (°C) vs. Time (hour)

600 700 800 900 1000 1100
0 10 20 30 40 50 60 70 80 90 100 110
Field Trial Testing
Virginia Tech Power Plant

- Virginia Tech Power Plant
  - Commercial entity that provides electricity to the residents of Blacksburg, VA, as well as steam for the Virginia Tech main campus
  - Annual steam output greater than 943 billion BTUs
  - A 6,250-kilowatt, 12,470-volt steam-turbine-powered generator produces nearly 27 million kilowatt-hours of electricity annually
  - Five boilers, each outfitted with superheaters rated at 80,000 or 100,000 lbs of steam per hour

- Successful sensor deployment
  - Coal-fired boiler (~1000ºC anticipated)
  - Installed during boiler operation
  - Operating for 2 weeks
  - Remote and mobile data access and monitoring
Field Trial Testing

Preliminary Results

- Installed successfully and currently operating at 2 weeks
- Optical interrogator was “turned off” and caused observed data drops
- High operating temperatures anticipated in coming weeks
MILESTONES AND SCHEDULE
Project Milestones

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

- **Fully-distributed temperature sensing system**
  - Demonstrated prototype FBG sensing system in single crystal sapphire fibers
  - Demonstrated prototype Raman distributed sensing system in single crystal sapphire fibers

- **Field Testing**
  - Demonstrated harsh environment sensor packaging
  - Performance tested prototype sensing system
  - Successful deployment and operation of sensing system

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<th>Title/Description</th>
<th>Planned Completion Date</th>
<th>Actual Completion Date</th>
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<td>1</td>
<td>Management and Qualification Plan</td>
<td>5/15/2014</td>
<td>5/15/2014</td>
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<td>3</td>
<td>Demonstration of LMV Sapphire Fabrication</td>
<td>6/30/2015</td>
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<td>4</td>
<td>Demonstration of Sensing System</td>
<td>12/31/2015</td>
<td>12/31/2015</td>
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<td>6</td>
<td>Project Management Plan</td>
<td>9/30/2017</td>
<td>9/30/2017</td>
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<td>7</td>
<td>Optimized Sensing System</td>
<td>12/31/2017</td>
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<td>8</td>
<td>Harsh Environment Sensor Packaging</td>
<td>12/31/2017</td>
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<td>9</td>
<td>Field (or Performance) Testing of Prototype Sensor</td>
<td>12/31/2018</td>
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<td>10</td>
<td>Final Report</td>
<td>3/30/2019</td>
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**Milestone Success Criteria**

- All Project Milestones Met On Time and On Budget
- All Success Criteria Met On Time and On Budget

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<th>ID</th>
<th>Title</th>
<th>Description</th>
<th>Result</th>
<th>M.S.</th>
<th>Planned Completion</th>
<th>Actual Completion</th>
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</table>
| SC1  | System Modeling                | 1. 50% modal volume reduction  
2. Sensing length of 3 m  
3. Resolution of < 20 cm | 1. >> 50% modal volume reduction  
2. Sensing length of 3 m  
3. Resolution < 17 cm | M2   | 12/31/2014          | 12/31/2014          |
| SC2  | LMV Sapphire Fiber             | 1. 40% reduction in modal volume  
2. Attenuation < 6 dB/m @ 355 nm  
3. Minimum bend radius < 25 mm | 1. > 50 % modal volume reduction  
2. Attenuation < 8 dB/m @ 532 nm  
3. Minimum bend radius < 4 mm | M3   | 6/30/2015           | 6/30/2015           |
| SC3  | Distributed Sensing System     | 1. Sensing length of 2 m  
2. Resolution < 20 cm | 1. Sensing length of 3 m  
2. Resolution < 10 cm | M4   | 12/31/2015          | 12/31/2015          |
| SC4  | Prototype Test Results         | Distributed Temperature Sensing:  
1. Sensing length of 3m  
2. Resolution < 20 cm | Distributed Temperature Sensing:  
1. Sensing length of 3 m  
| SC5  | Harsh Environment Sensor       | 1. Maximum Temperature: 1000°C  
2. Testing Duration: 100 hours | 1. Maximum Temperature: 1000°C  
2. Testing Duration: 100 hours | M8   | 12/31/2017          | 12/31/2017          |
| SC6  | Temperature Sensing System     | 1. Maximum Temperature: 1000°C  
2. Temperature Accuracy: < 5% F.S. | TBD | M9   | 12/31/2018          | TBD               |
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
  • Fabrication of FBGs in sapphire fiber via the point-by-point method
  • Measurement of fiber attenuation in the time domain in sapphire fiber
  • Distributed Raman temperature measurements in sapphire fiber
  • Quasi-distributed FBG based temperature measurements in sapphire fiber
  • Demonstrated few to single mode operation in sapphire fiber
  • Developed harsh environment fiber optic sensor packaging

• 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), Logan Theis (Ph.D., TBD)

• 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
  – Point-by-point fabrication of single crystal sapphire fiber FBGs
  – 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
  – Quasi-distributed FBG based temperature measurements in sapphire fiber
• Dissemination of Findings
  – 8 peer reviewed publications
  – 2 provisional patents filed
• Graduate Student Support (12)
• Faculty Training and Development (4)
Next Steps

- Field trial of prototype sensor in coal-fired boiler
  - Continue operation until facility shut-down
  - Conduct full-scale data analysis
  - Review “lessons learned” and develop “action plan”
- Field trial of prototype sensor in natural gas-fired boiler
  - Implement action plan and improvements for sensor deployment
- Generate final report and “media-ready” documentation
- Evaluate additional research opportunities for fiber and sensing technologies
Acknowledgements

Virginia Tech
Center for Photonics Technology (CPT)
Gary Pickrell
Anbo Wang
Zhihao Yu
Bo Liu
Cary Hill
Di Hu
Adam Floyd
Yujie Cheng
Sunny Chang
Elizabeth Bonnell
Hanna Heyl
Zhiting Tian
Haifeng Xuan
Logan Theis
Amy Hill, Jiaji He, Shuo Yang, Amiya Behera, Chennan Hu, Cindy Purdue, Chenyuan Hu, Nevada Davis, Robert Blackwell

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
Review Panel Recommendations

- All Recommendations have been addressed
- On schedule and budget

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<tr>
<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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<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>
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<td>R2</td>
<td>&quot;Back of the Envelope&quot; Calculations to Predict Fiber and System Performance</td>
<td>3/30/2016</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>R5</td>
<td>Evaluate Consistencies between Theoretical Analyses and Experimentation/Manufacturability</td>
<td>12/30/2016</td>
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LMV Sapphire Fiber Fabrication

Equipment and Techniques

Custom Etching System

Excellent Temperature Control

Temperature and Etching Uniformity

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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)
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)
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

*Growth Chamber Optics*

Spherical (Parabolic) Mirror
(Focus beam to melt zone)

Reflxicon
(Create “doughnut” shaped beam)

Scraper Mirror
(Reflect beam to spherical mirror)
Laser Heated Pedestal Growth

Automatic Diameter Control System

Diameter variations ~1.7% were readily achieved
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

Peak Frequency

Temperature dependence of sapphire Raman position

Peak Width

Temperature dependence of sapphire Raman width

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

1 meter Sapphire Fiber

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

2 meter Sapphire Fiber

![Stokes VS Temperature Graph](image)

![AntiStokes VS Temperature Graph](image)
Raman DTS System Performance

3 meter Sapphire Fiber

Stokes VS Temperature

AntiStokes VS Temperature

Joint reflection

Normalized Stokes (a.u.)

Distance (m)

Normalized AntiStokes (a.u.)

Distance (m)
Raman DTS System Performance

Spatial Resolution

- Sensing length: 3 meters
- Temperature: 1400°C
- Spatial resolution: 16.4 cm
  - Determined via 10% to 90% response distance