2017 CROSSCUTTING
Research Project Review
Omni William Penn Hotel
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REDUCED MODE SAPPHIRE FIBER *AND* DISTRIBUTED SENSING SYSTEM

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Gary Pickrell, Anbo Wang, Daniel Homa
Virginia Tech Center for Photonics Technology
Blacksburg, VA 24061
pickrell@vt.edu, awang@vt.edu, dan24@vt.edu
http://photonics.ece.vt.edu/

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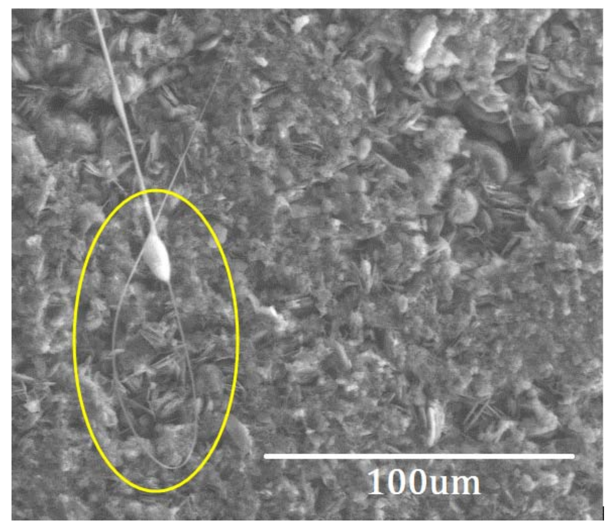
Overview

- 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



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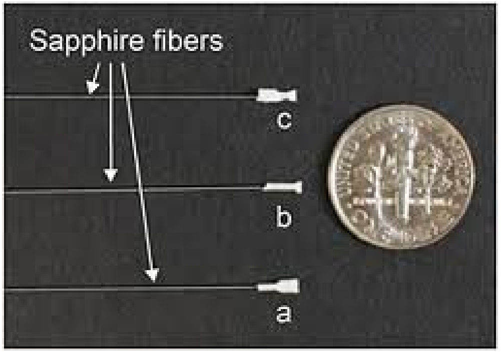
Motivation

- 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

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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

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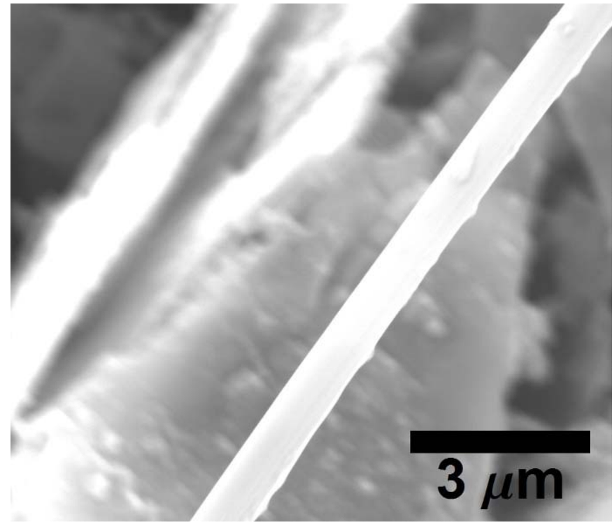
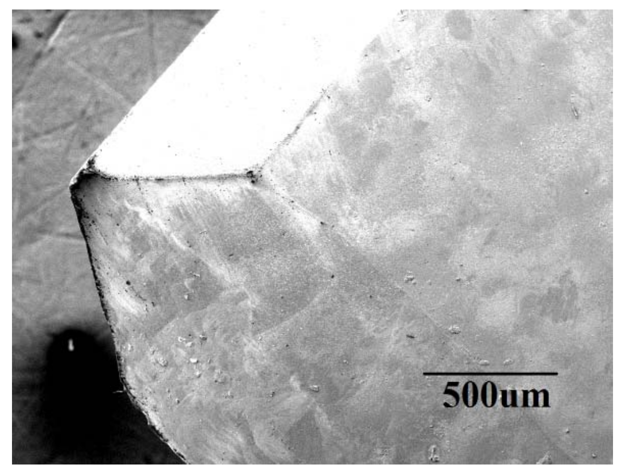
RESEARCH PROGRESS:
LMV SAPPHIRE FIBER DESIGN AND FABRICATION

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





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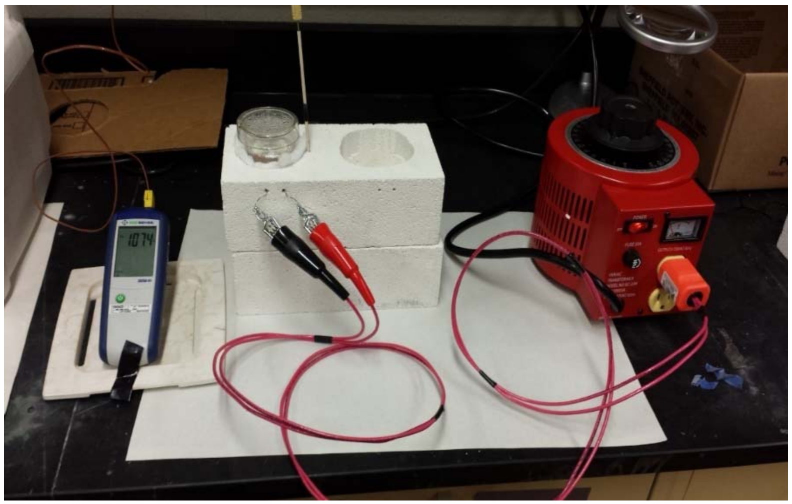
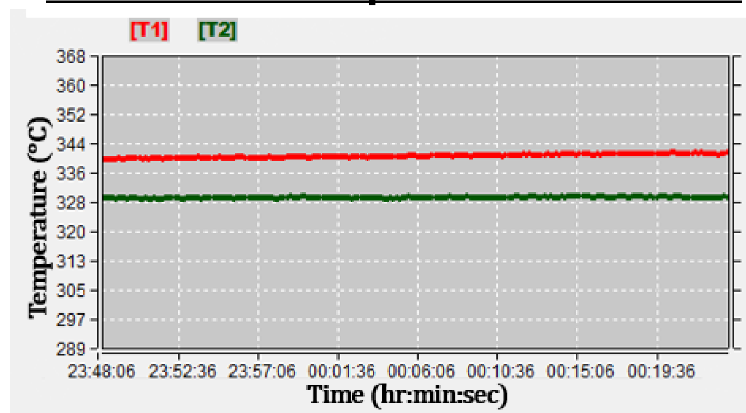
LMV Sapphire Fiber Fabrication

Equipment and Techniques

Custom Etching System



Excellent Temperature Control



Temperature and Etching Uniformity

Sample	Top Diameter (μm)	Bottom Diameter (μm)
A	82.0	73.1
B	80.9	73.4
C	79.6	74.4
Average	80.8	73.6
Standard Deviation	1.2	0.7

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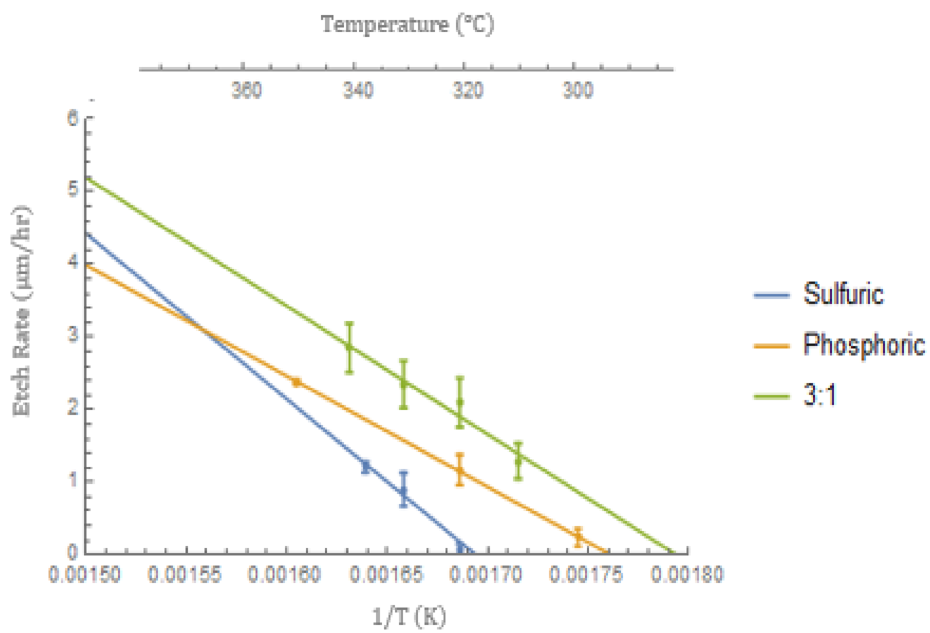
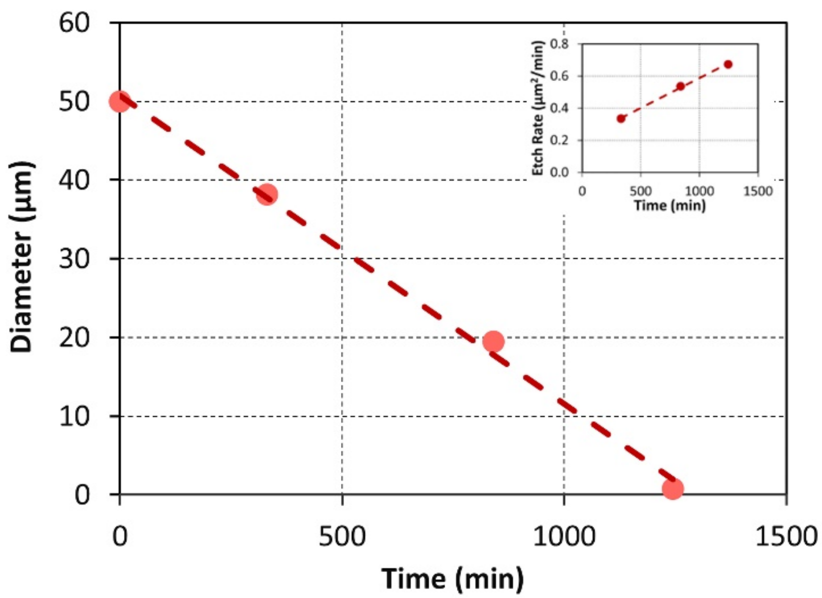
LMV Sapphire Fiber Fabrication

Understanding and Control

Optimization of Etching Solution



Etch Rates



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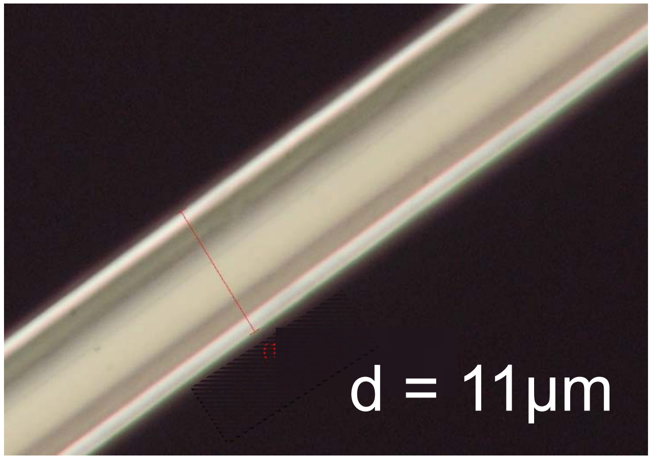
LMV Sapphire Fiber Fabrication

Characterization of Surface Quality

Optical Microscopy

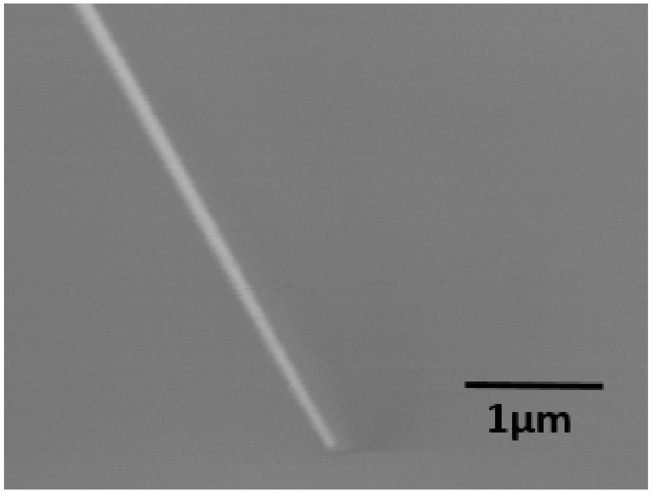
(fiber diameter, contamination, polarization states, non-uniformities)



Scanning Electron Microscopy (SEM)

Energy-dispersive X-ray spectroscopy (EDAX)

(fiber diameter, contamination, composition/elemental analysis, defects, non-uniformities)



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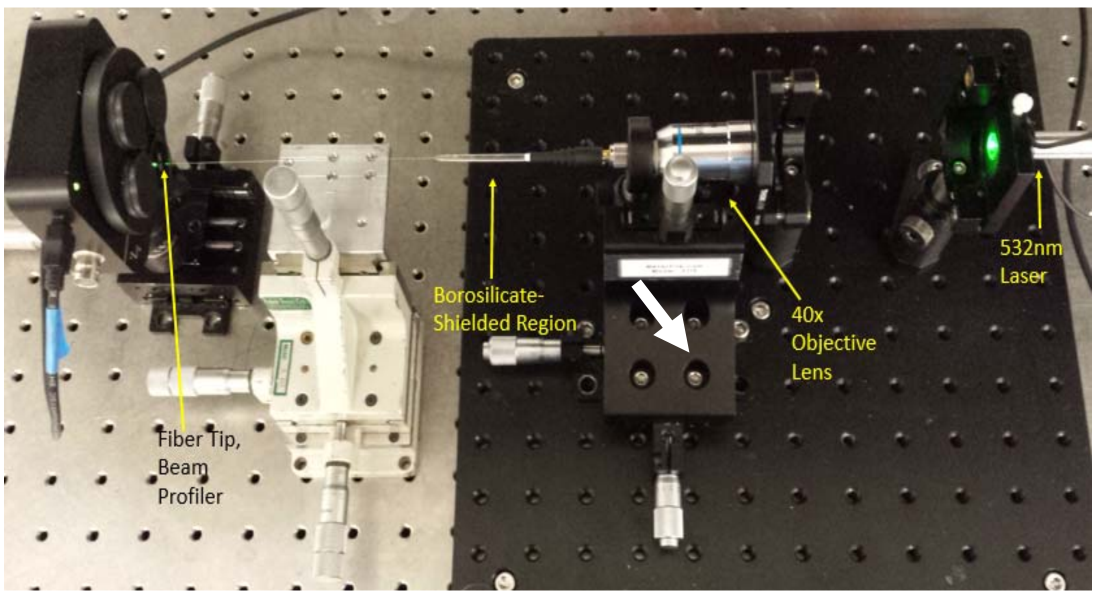
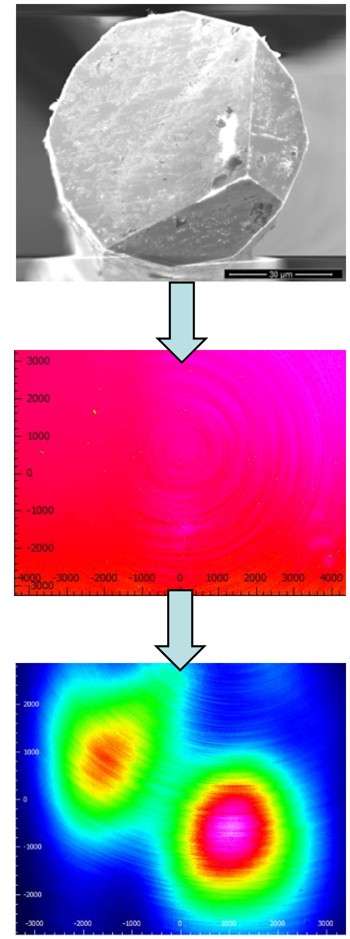
LMV Sapphire Fiber Testing

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)



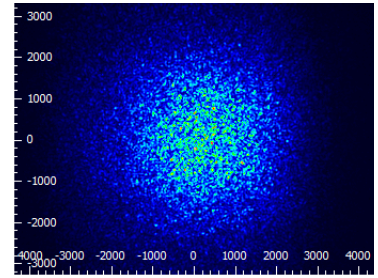


LMV Sapphire Fiber Testing

Far Field Analysis Method

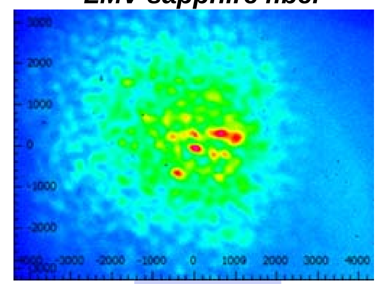
- Far-field intensity patterns capture
  - Prior to etching
  - Post etching and polishing
- Modal interference and superposition yields a "speckled" appearance
- Reduction in diameter and modal volume
  - Number of power peaks (speckles) decreases
  - Relative diameter of individual speckles increases
  - Modal interference and superposition due a decrease in the number of supported modes
- Qualitative analysis of modal volume
- Low order mode profiles are visible

"As-received" sapphire fiber



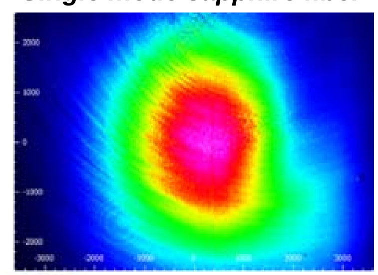
Etching

LMV sapphire fiber



Etching

Single mode sapphire fiber



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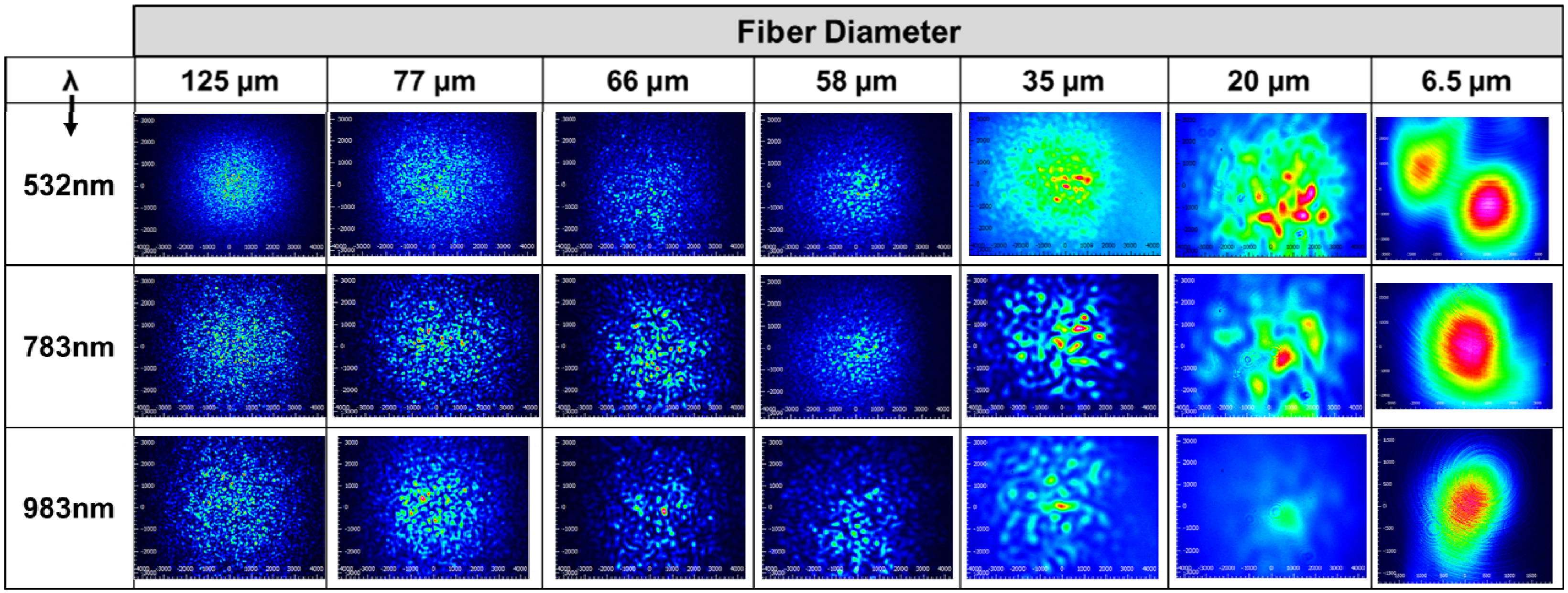
LMV Sapphire Fiber Testing

Far Field Analysis of RDSF



The trend in modal volume reduction with a reduction in fiber diameter and increase wavelength agrees with theoretical predictions

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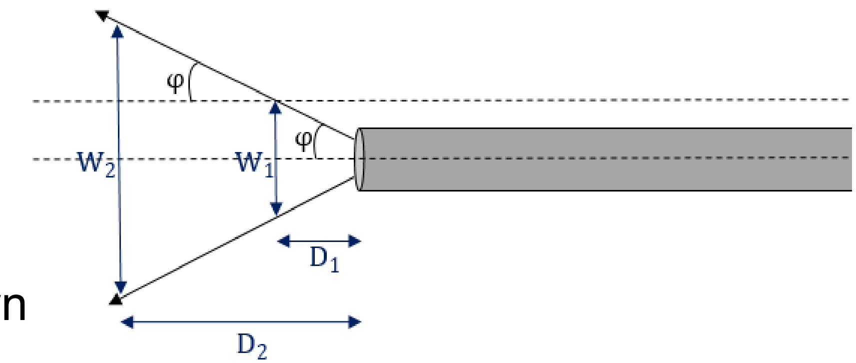
LMV Sapphire Fiber Testing

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)

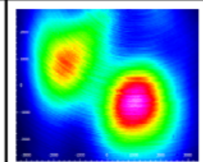
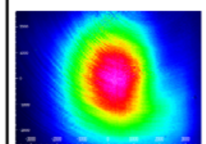
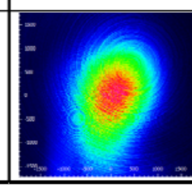


LMV Sapphire Fiber Testing

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

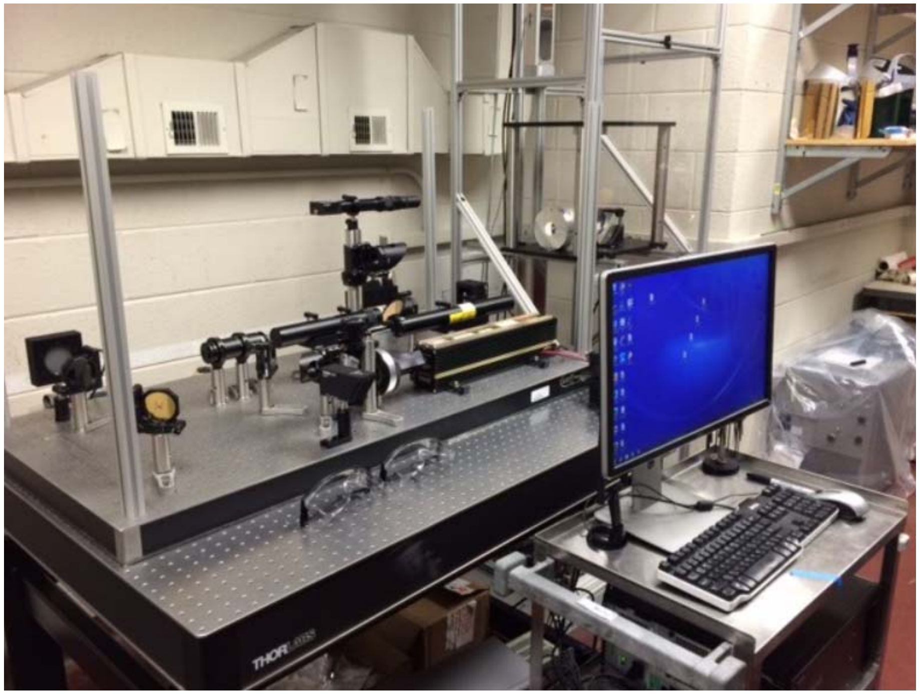
- Beam width differential method
  - CCD camera beam profiler (Thorlabs BC106-VIS)

	Calculated Modal Volume		
Wavelength	**$NA_{Theor.}$=~1.4**	**NA_{eff}=0.09**	**6.5µm**
532nm	1614	2	
783nm	736	1	
983nm	461	1	

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



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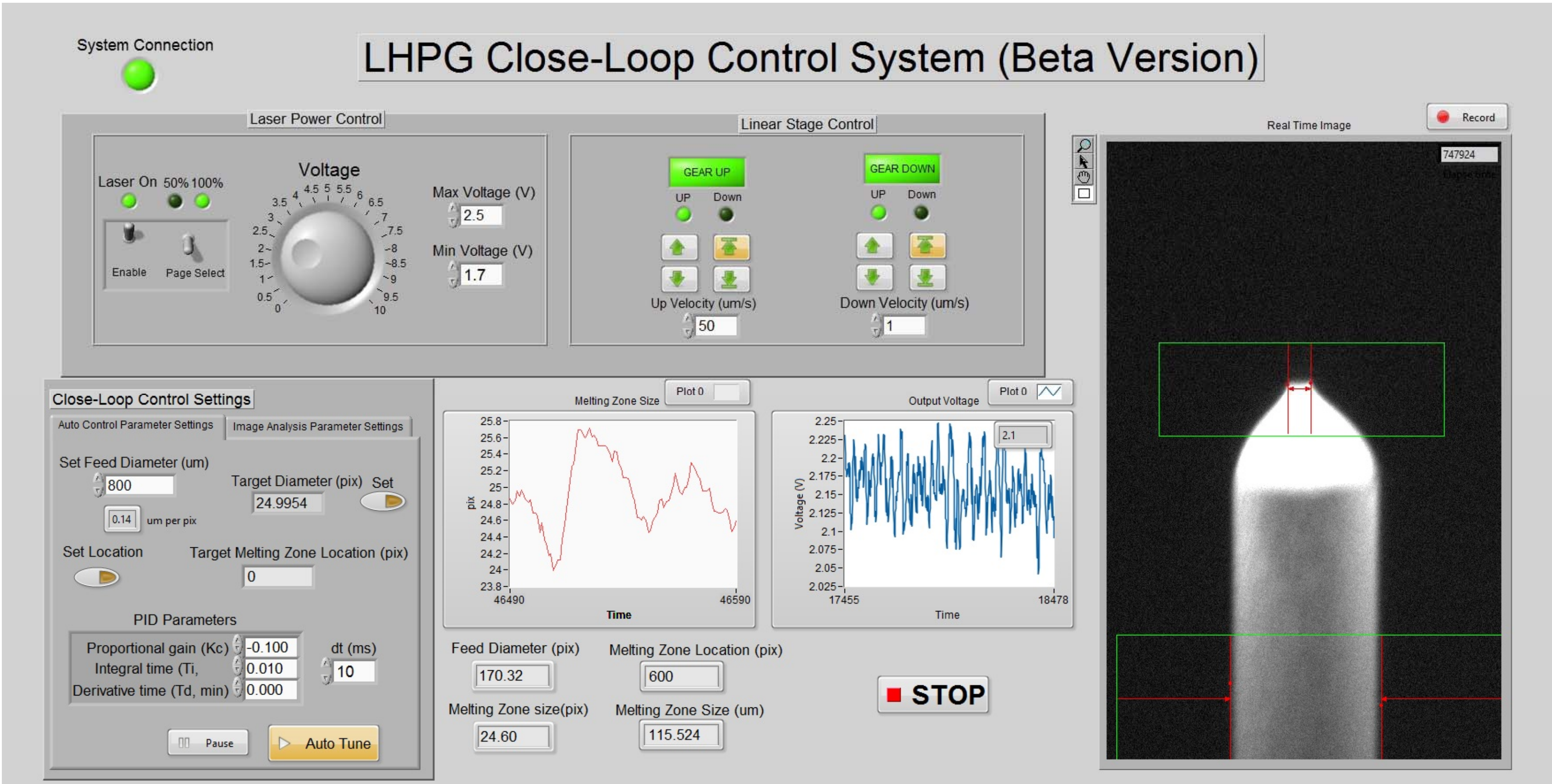
Laser Heated Pedestal Growth

Automatic Diameter Control System



Diameter variations ~1.7% were readily achieved

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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

<u>RESEARCH PROGRESS</u>: *DISTRIBUTED TEMPERATURE SENSING SYSTEM*

Operating Wavelength Selection

Blackbody Radiation

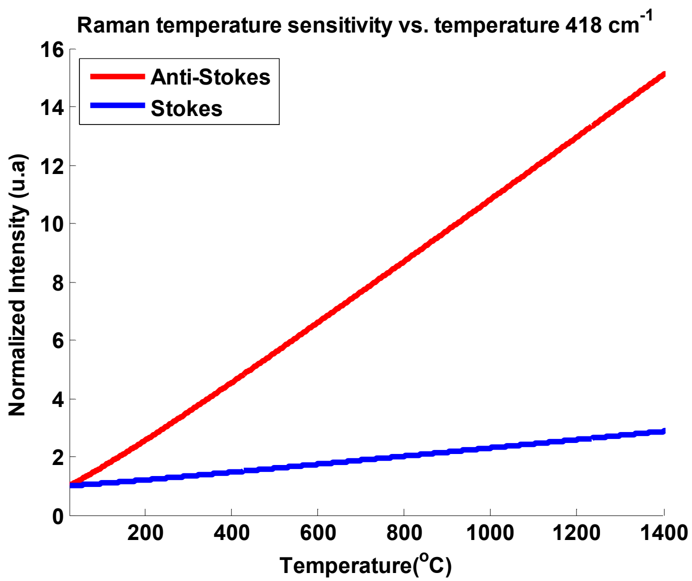
- The Raman intensity of the Anti-Stokes and Stokes components is proportional to its differential cross section given by *(M. Hobel, Applied Optics, 1995)*

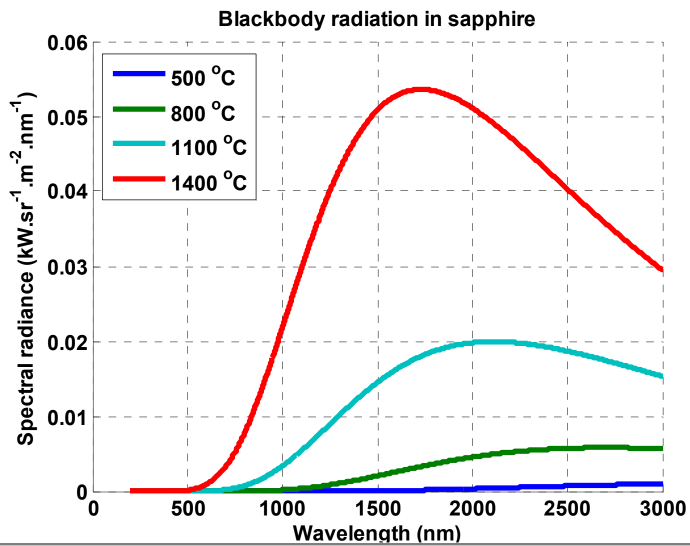
$$\left.\frac{d\sigma_{AS}}{d\Omega}\right|_x \cong \frac{1}{\lambda_{AS}{}^4}\frac{1}{\exp\left[\frac{hc\Delta\upsilon}{K_B T(x)}\right]-1}$$

$$\left.\frac{d\sigma_{S}}{d\Omega}\right|_x \cong \frac{1}{\lambda_{S}{}^4}\frac{1}{1-\exp\left[-\frac{hc\Delta\upsilon}{K_B T(x)}\right]}$$

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

$$B(T) = \frac{2hc^2}{\lambda^5}\frac{1}{e^{\frac{hc}{\lambda k_B T}}-1}$$

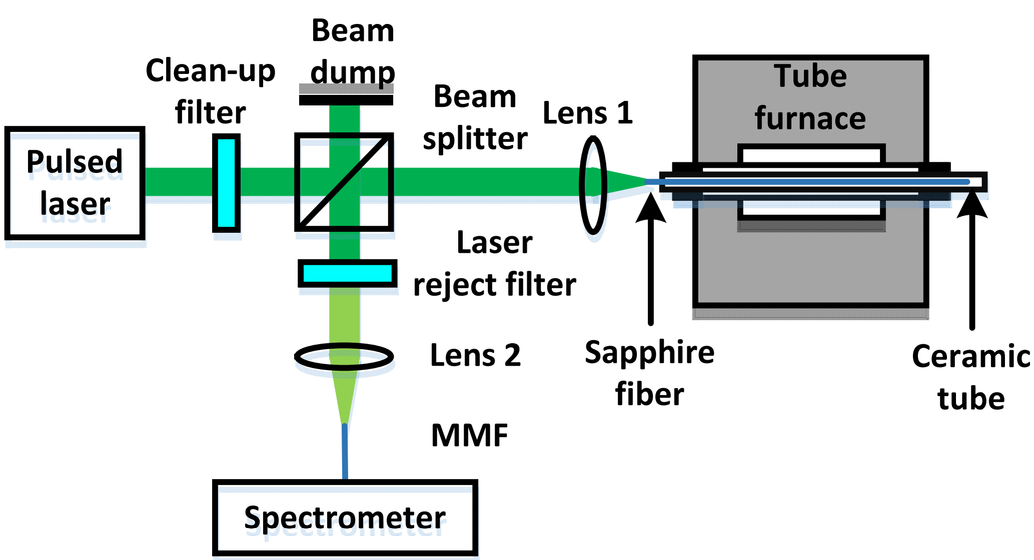


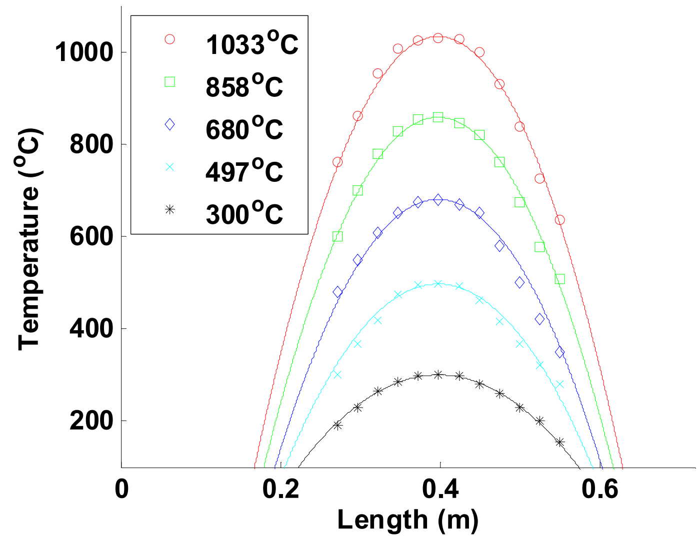


Scattering in Sapphire Fiber

Experimental Set-Up

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



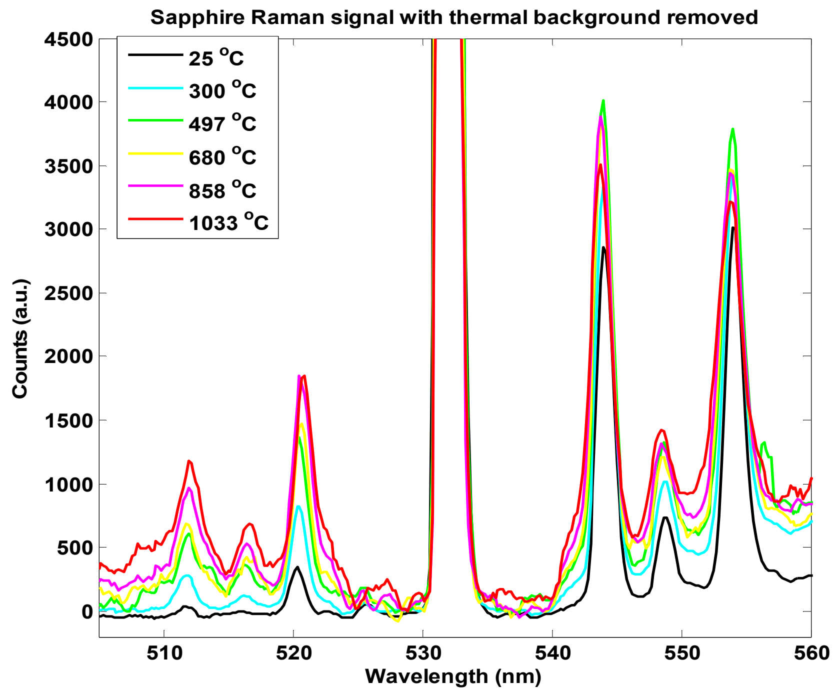


B. Liu et al, Optics Letters, 2015

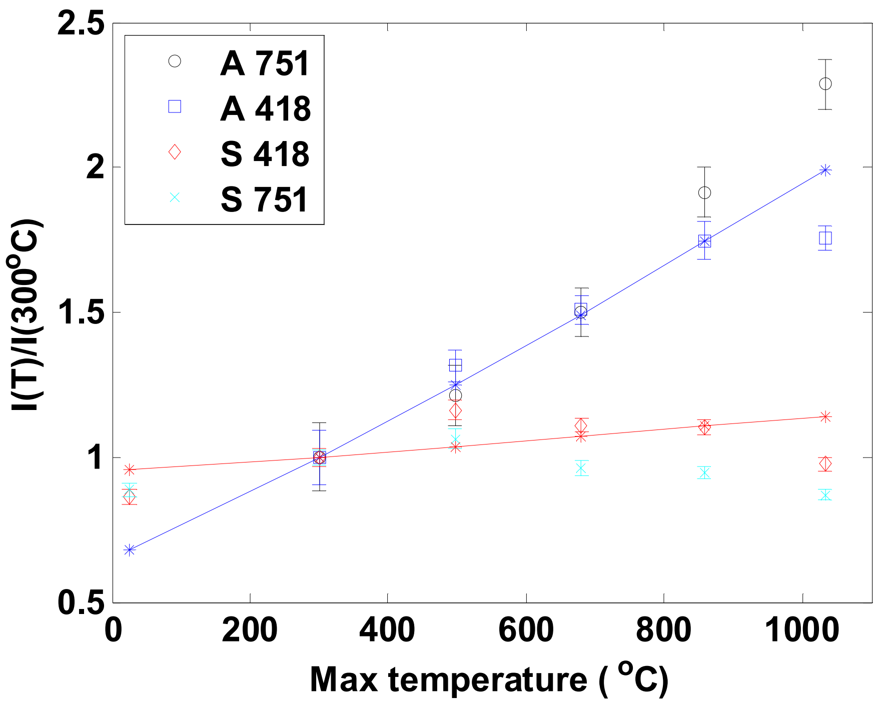
Scattering in Sapphire Fiber

Temperature Dependence

Spectrum



Peak Intensity

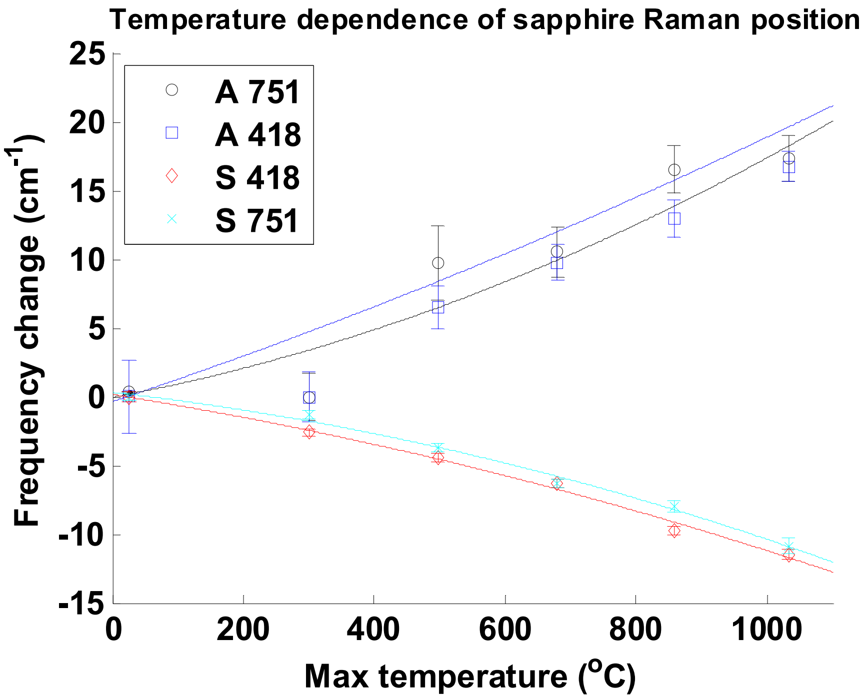


B. Liu, Optics Letters, 2015

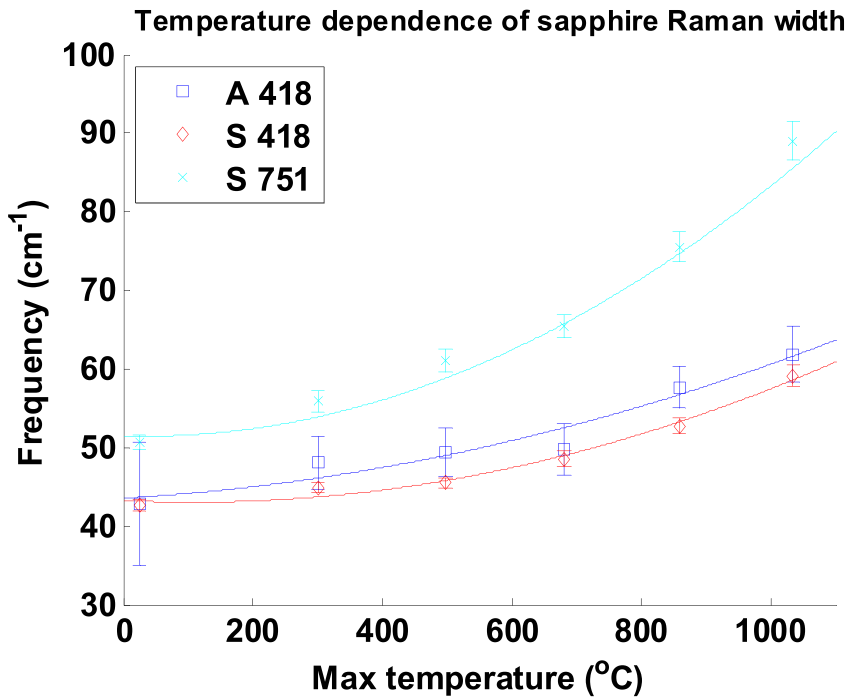
Scattering in Sapphire Fiber

Temperature Dependence

Peak Frequency



Peak Width



B. Liu, Optics Letters, 2015

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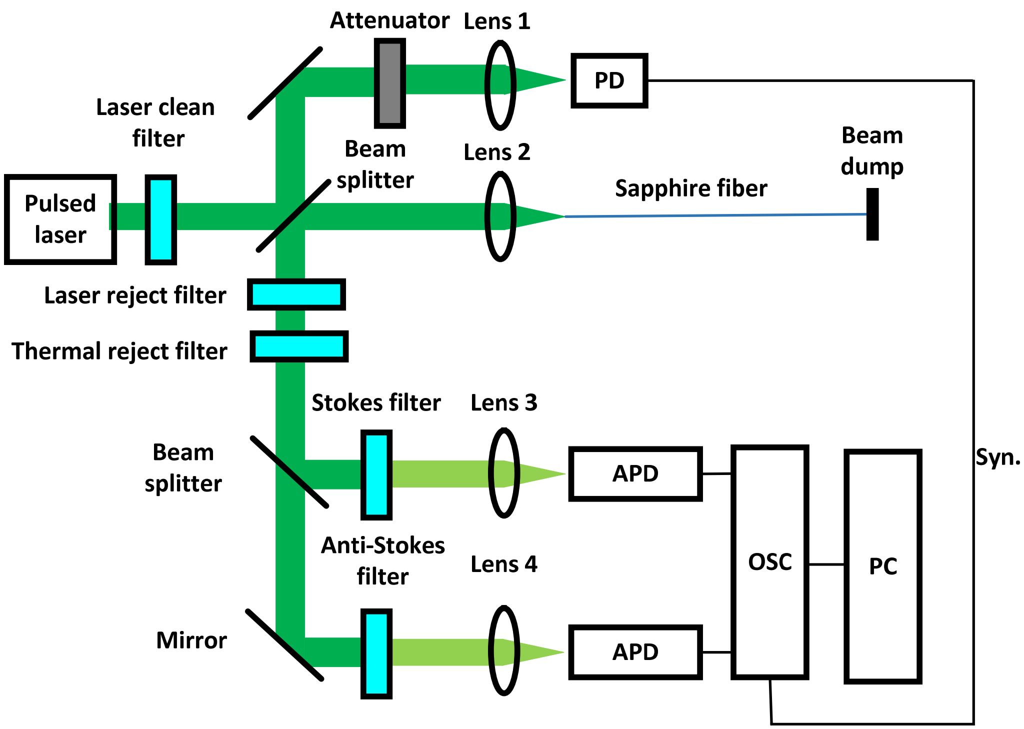
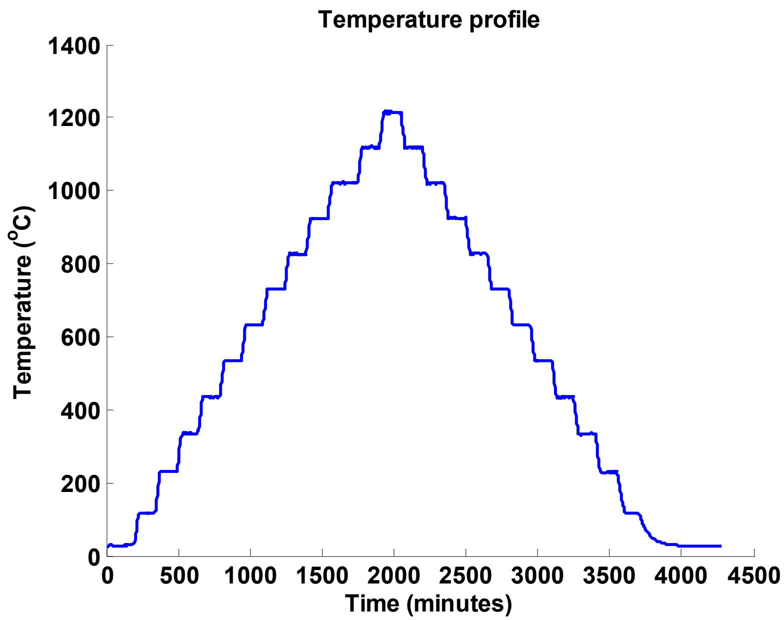
Raman DTS System Design

Experimental Set-Up





B. Liu et al, Optics Letters, 2016

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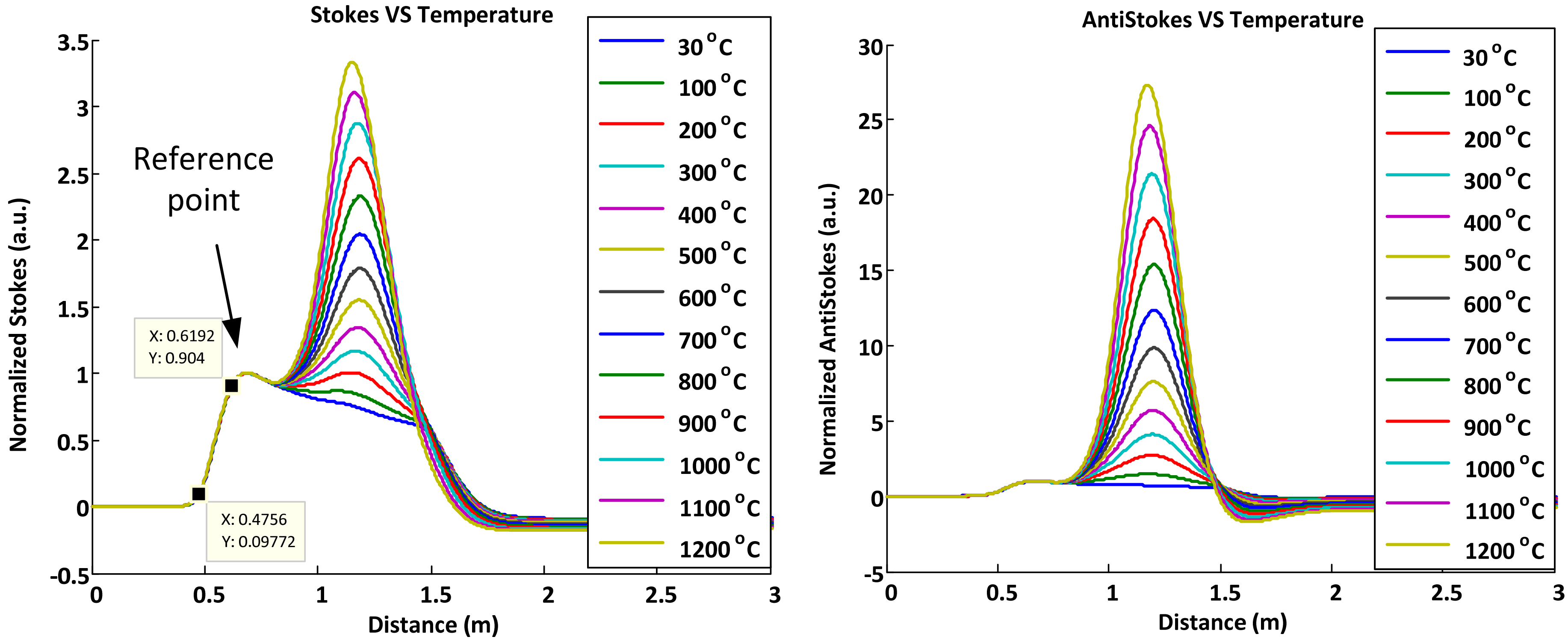
Raman DTS System Performance

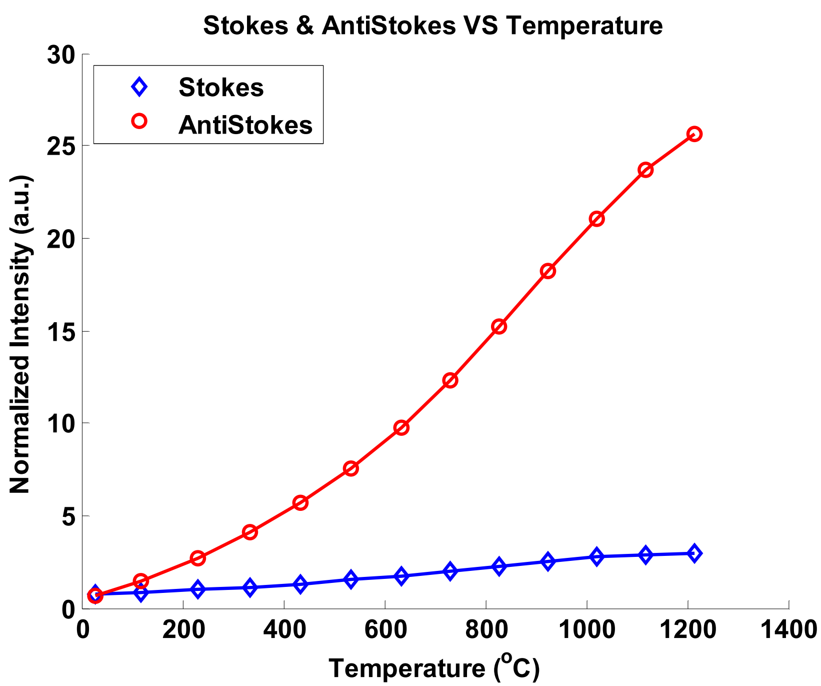
1 meter Sapphire Fiber

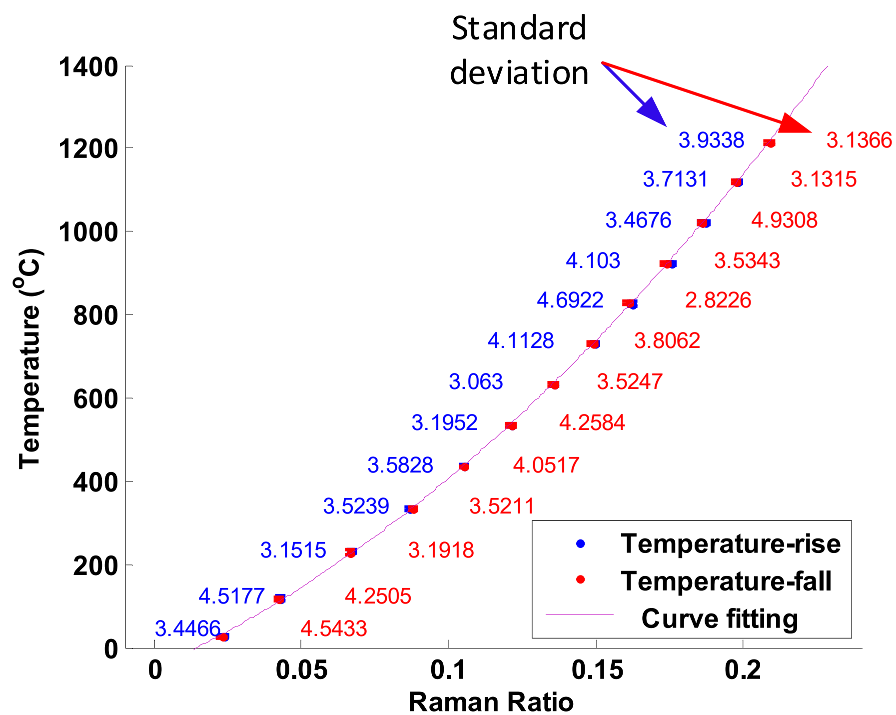


B. Liu et al, Optics Letters, 2016

Raman DTS System Performance

1 meter Sapphire Fiber





Standard Deviation: 3.7ºC (3.0ºC)

B. Liu et al, Optics Letters, 2016

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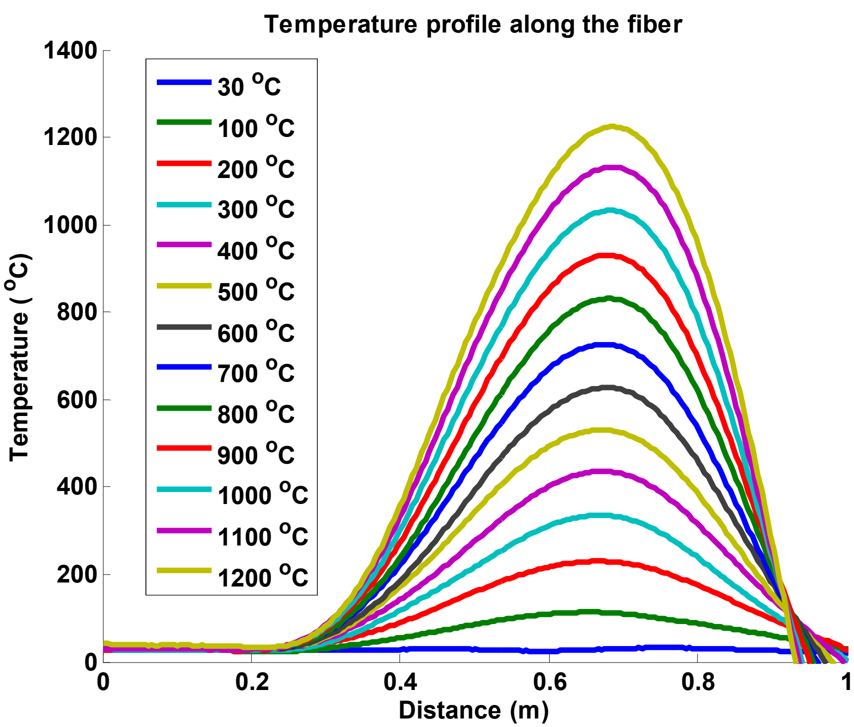
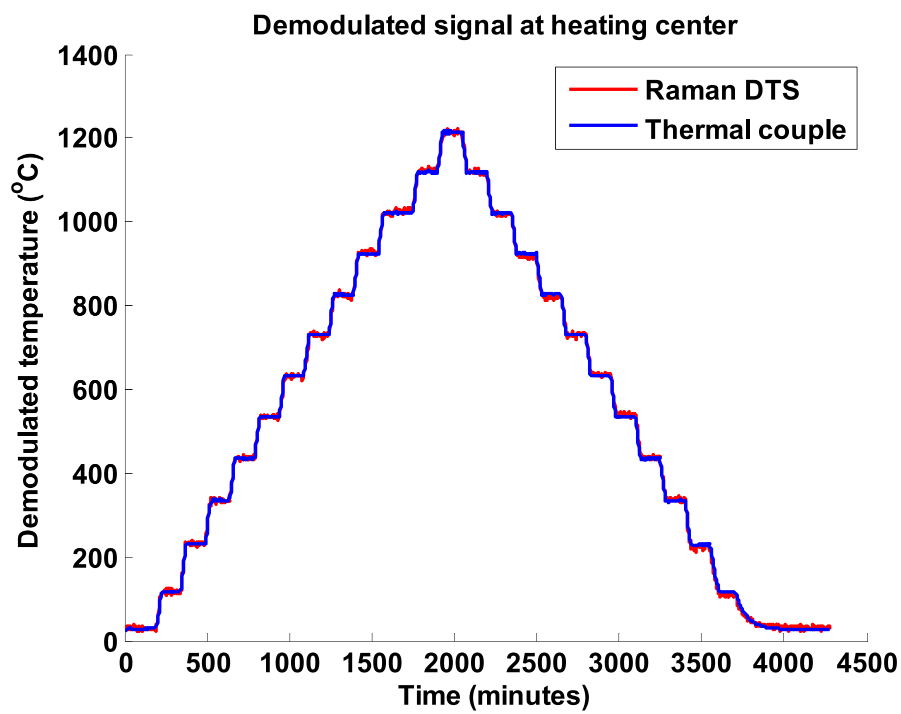
Raman DTS System Performance

1 meter Sapphire Fiber





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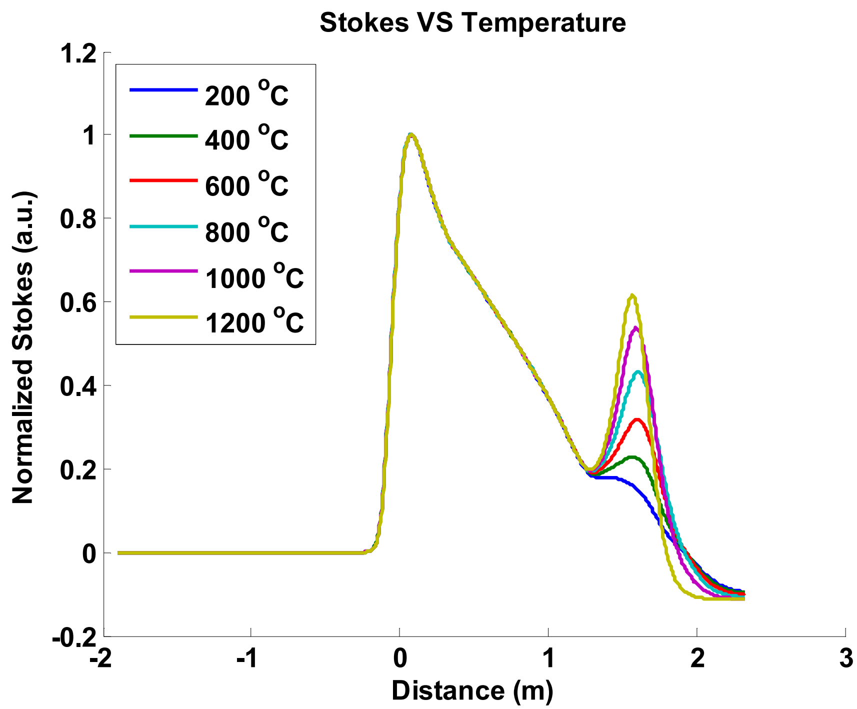
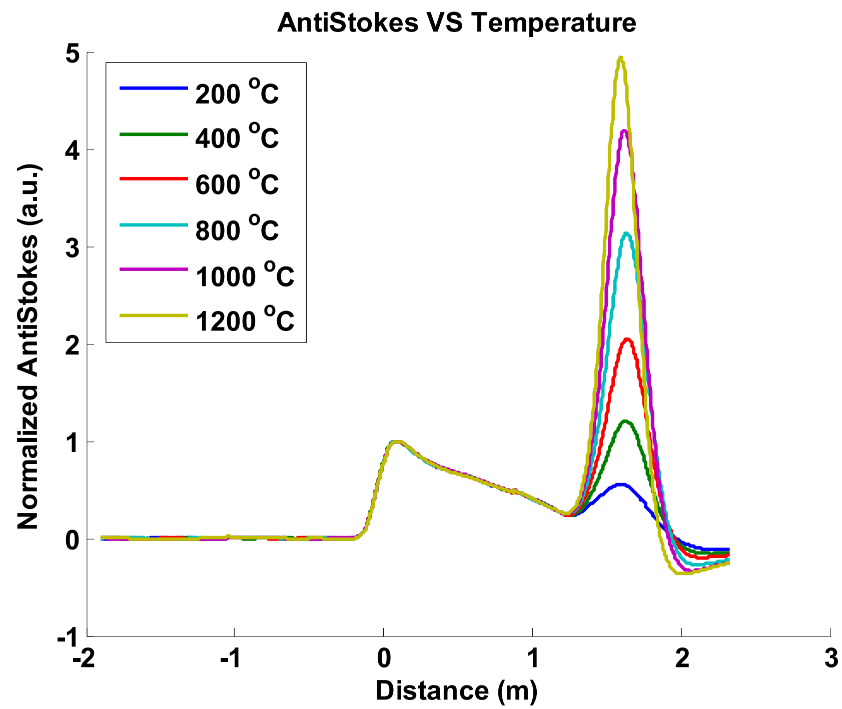
Raman DTS System Performance

2 meter Sapphire Fiber





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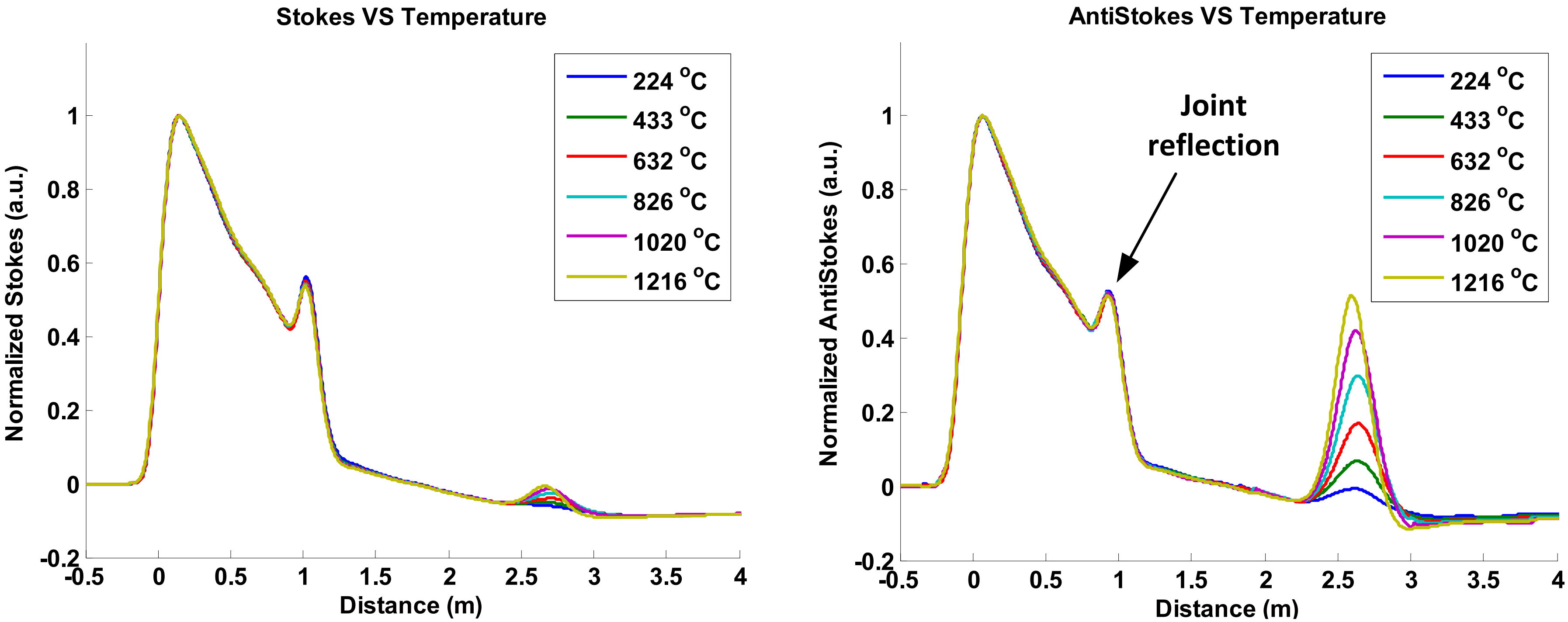
Raman DTS System Performance

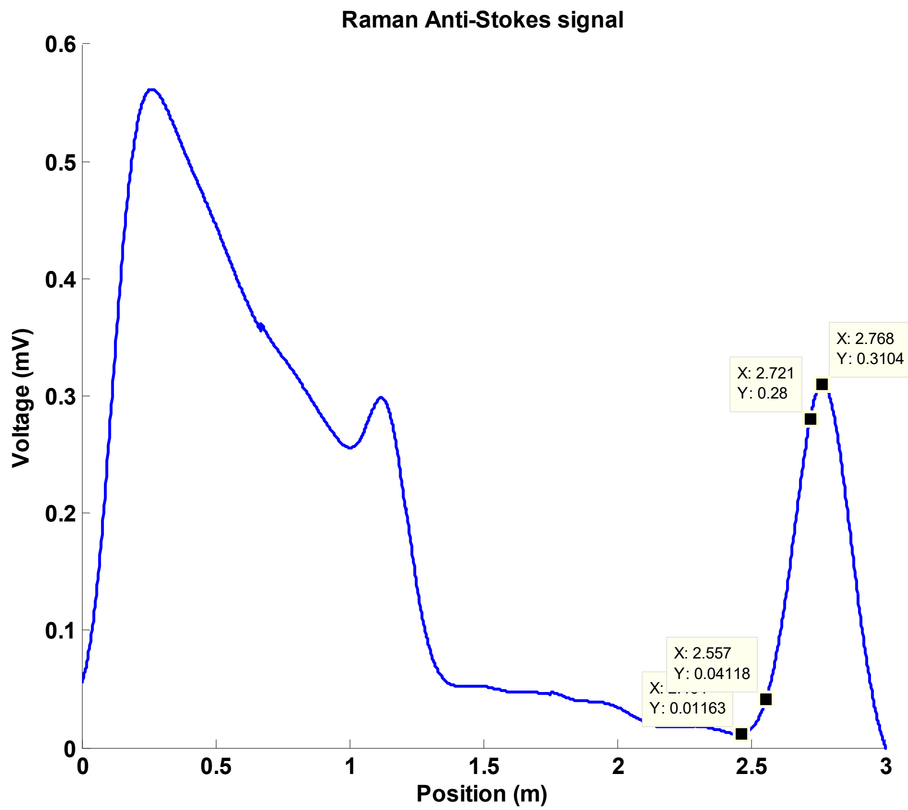
3 meter Sapphire Fiber



Raman DTS System Performance

Spatial Resolution

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



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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 3.0ºC (0.2% of full scale) was demonstrated in a 1 meter sapphire fiber.
- A maximum operating temperature of 1400ºC 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

M. #	Title/Description	Planned Completion Date	Actual Completion Date
1	Project Management Plan	5/15/2014	5/8/2014
2	Modeling of LMV Sapphire Fiber & Sensing System	12/31/2014	12/31/2014
3	Demonstration of LMV Sapphire Fabrication	6/30/2015	6/30/2015
4	Demonstration of Sensing System	12/31/2015	12/31/2015
5	Prototype System Test Results	6/29/2016	6/29/2016
6	Final Report	12/31/2016	85%

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 ~ 1400ºC

ID	Title	Description	Result	M.S.	Planned Completion	Actual Completion
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 2. Resolution < 17 cm	M5	6/29/2016	6/29/2016

Review Panel Recommendations

- All Recommendations have been addressed
- On schedule and budget

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

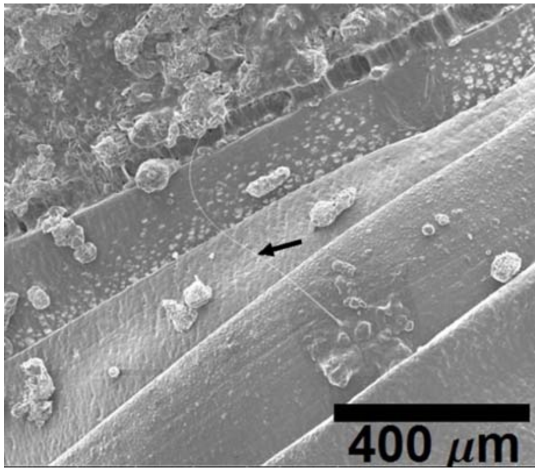
Project Schedule

GANT CHART		BUDGET PERIOD 1						BUDGET PERIOD 2						Ext.	KEY PROJECT DATES				
		Project Year 1				Project Year 2				Project Year 3				Y4	Planned Start Date	Planned End Date	Actual Start Date	Actual End Date	Comments
Task #	Task Name	Q1	Q2	Q3	Q4	Q1	Q2	Q3	Q4	Q1	Q2	Q3	Q4	Q1					
1	Project Management and Planning														1/1/2014	5/15/2014	1/1/2014	5/8/2014	Completed
2	Determine Technical Requirements														1/1/2014	5/15/2014	1/1/2014	5/8/2014	
M1*	**MILESTONE 1***		√												**5/15/2014 *(4/1/2014)****		**4/28/2014***		
3	Optical Modeling of LMV Sapphire and Distributed Sensor System														2/3/2014	12/30/2014	2/3/2014	12/30/2014	Completed
3.1	LMV Sapphire Fiber Modeling														2/3/2014	12/30/2014	2/3/2014	12/30/2014	
3.2	Sapphire Fiber Based High Temperature Distributed Sensing Modeling														4/1/2014	12/30/2014	4/1/2014	12/30/2014	
M2	**MILESTONE 2**				√										**12/31/2014**		**12/31/2014**		
4	Demonstrate Feasibility of LMV Sapphire and Distributed Sensing System														4/2/2014	12/31/2015	2/1/2014	12/31/2015	Completed
4.1	LMV Sapphire Fiber Fabrication														4/1/2014	6/26/2015	2/1/2014	6/30/2015	
M3	**MILESTONE 3**						√								**6/29/2015**		**6/29/2015**		
4.2	Distributed Sensing System Building and Demonstrating with Silica Fiber														5/1/2014	12/29/2015	2/1/2014	12/31/2015	Completed
M4	**MILESTONE 4**								√						**12/31/2015**		**12/31/2015**		
5	Develop LMV Sapphire Fiber Temperature Sensing System														9/1/2015	6/30/2016	7/1/2015	6/16/2016	Completed
5.1	Construct Prototype LMV Sapphire Fiber														9/1/2015	1/4/2016	9/1/2015	5/1/2016	
5.2	Demonstrate Distributed High Temperature Sensing with LMV Sapphire Fiber														1/14/2016	5/18/2016	10/1/2015	6/16/2016	
5.3	Perform Sensor Calibration and Verification														5/19/2016	6/28/2016	6/1/2016	6/29/2016	
M5	**MILESTONE 5**										√				**6/29/2016**				
6	Construct & Evaluate Refined Prototype Sensing System														6/30/2016	11/30/2016	7/16/2016		On Schedule No Cost Extension Granted
6.1	Fabricate Refined Version of LMV Sapphire Fiber														6/30/2016	8/22/2016	7/20/2016		
6.2	Construct Refined Version of LMV Sapphire Fiber Distributed Sensing System														6/29/2016	9/15/2016	7/20/2016		
6.3	Test Sensor and Evaluate Performance														9/16/2016	10/28/2016	11/20/2016		
7	Prepare and Submit Final Report														11/11/2016	11/11/2016	10/20/2016		
M6	**MILESTONE 6**														**12/31/2016**				

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)



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Research Products

- Peer Reviewed Publications
  - Hill, Cary, Daniel Homa, Bo Liu, Zhihao Yu, Anbo Wang, and Gary Pickrell. ***Submicron Diameter Single Crystal Sapphire Optical Fiber***, Materials Letters 138, no. 0 (2015): 71-73.
  - Bo Liu, Zhihao Yu, Zhipeng Tian, Daniel Homa, Cary Hill, Anbo Wang, and Gary Pickrell. ***Temperature dependence of sapphire fiber Raman scattering***, Opt Lett. 2015; 40(9):2041-4.
  - Cheng, Yujie, Cary Hill, Bo Liu, Zhihao Yu, Haifeng Xuan, Daniel Homa, Anbo Wang and Gary Pickrell. ***Modal Reduction in Single Crystal Sapphire Optical Fiber***, Optical Engineering 54, no. 10 (2015): 107103.
  - Yujie Cheng, Cary Hill, Bo Liu, Zhihao Yu, Haifeng Xuan, Daniel Homa, Anbo Wang, and Gary Pickrell. ***Design and analysis of large-core single-mode windmill single crystal sapphire optical fiber***, Optical Engineering, 55 (2016) 066101-066101.
  - Liu, Bo, Zhihao Yu, Daniel Homa, Yujie Cheng, Gary Pickrell, and Anbo Wang. ***Sapphire-Fiber-based Distributed High Temperature Sensing System***, Optics Letters, 41(18), 4405-4408.
  - Hill, Cary, Dan Homa, Zhihao Yu, Yujie Cheng, Bo Liu, Anbo Wang and Gary Pickrell. ***Single Crystal Sapphire Optical Fiber***, Applied Sciences (submitted for publication).
- Intellectual Property
  - U.S. Patent Application No. 62/057,291; ***Processing Technique for the Fabrication of Sub-micron Diameter Sapphire Optical Fiber***, G. Pickrell, D. Homa, W. Hill, filed Sept. 30, 2014.
  - U.S. Patent Application No. 62/264,659. ***Distributed Temperature Sensing System Using Optical Sapphire Waveguide***, A. Wang, G. Pickrell, B. Liu, Z. Yu., Dec. 2015.

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

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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