NETL-HBCU/UCR
Project Joint Kick-Off Meeting
Materials Science and Engineering Department
Electrical and Computer Engineering Department
Center for Photonics Technology
Virginia Tech, Blacksburg VA
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INVESTIGATION OF HIGH TEMPERATURE SILICA BASED FIBER OPTIC SENSOR MATERIALS

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Overview

- Project Team
- Motivation
- Technical Background
- Potential Significance
- Relevancy to Fossil Energy
- Statement of Project Objectives (SOPO)
- Milestones, Budget, and Schedule
- Risks and Management Plan
- Project Status

Project Team

- PI : Dr. Gary Pickrell
  - Provide executive management for all phases of the project, and oversee the selection, fabrication, and characterization of the fibers.
- Co-PI : Dr. Anbo Wang
  - Support all phases of the project, and provide expertise in all technical aspects of the project and efforts pertaining to the testing and analysis of the selected optical fibers.
- Research Scientist: Dr. Daniel Homa
  - Daily project management, to include technical insights, under the guidance of the PI and Co-PI.
- Graduate Student: TBD, ECE
  - Construct the testing facilities, conduct all testing, and compile the data for interpretation.

Interdisciplinary collaboration between the Materials Science and Engineering and Electrical and Computer Engineering Departments at Virginia Tech

Motivation

- Need for robust and reliable sensing and monitoring systems
- Need for sensing system integration at a realistic cost point
- Need for an alternative to sapphire fiber optics, which are more expensive and require additional expertise

Power Generation Technology Needs

	Coal Gasifiers	Combustion Turbines	Solid Oxide Fuel Cells	Advanced Boiler Systems
Temperature	< 1600°C	< 1300°C	< 900°C	< 1000°C
Pressures	< 1000 psi	Ratios 30:1	Atmospheric	Atmospheric
Atmosphere(s)	Highly Reducing, Erosive, Corrosive	Oxidizing	Oxidizing and Reducing	Oxidizing
Examples of Important Gas Species	H_2, O_2, CO, CO_2, H_2O, H_2S, CH_4	O_2, Gaseous Fuels (Natural Gas to High Hydrogen), CO, CO_2, NO_x, SO_x	Hydrogen from Gaseous Fuels and Oxygen from Air	Steam, CO, CO_2, NO_x, SO_x

Ohodnicki, Jr., Paul R. "Embedded Sensors for Extreme Temperature and Harsh Environments." NETL-RUA Commercial Opportunity Summary, (2013)

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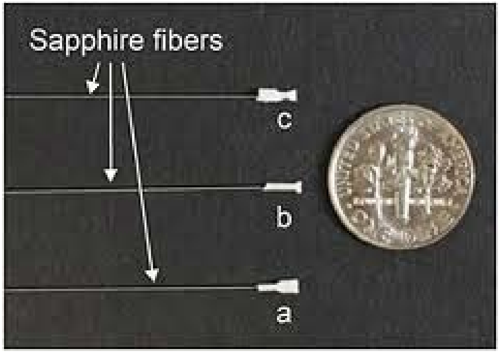
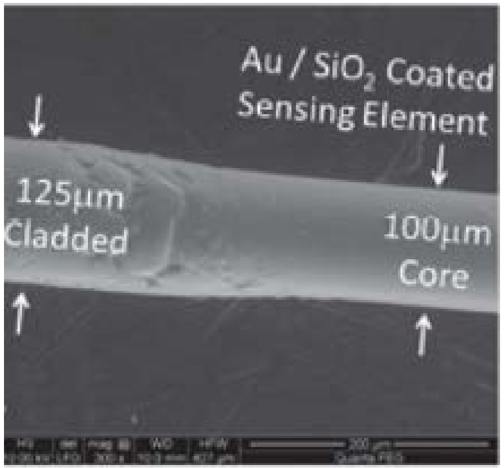
TECHNICAL BACKGROUND

Technical Background

FIBER OPTIC SENSING IN HARSH ENVIRONMENTS

- Mature single point and distributed fiber sensing systems
  - Raman distributed temperature
  - Bragg grating based strain and temperature
  - Single point Fabry Perot sensors
- FO sensing technologies have shown great promise in many applications
  - Structural health monitoring
  - Downhole pressure and temperature
- Fused silica optical fiber applications are limited by temperature
  - Reportedly between ~800-900ºC
- Single crystal sapphire FO sensors are used almost exclusively at high temperatures
  - Exotic and expensive





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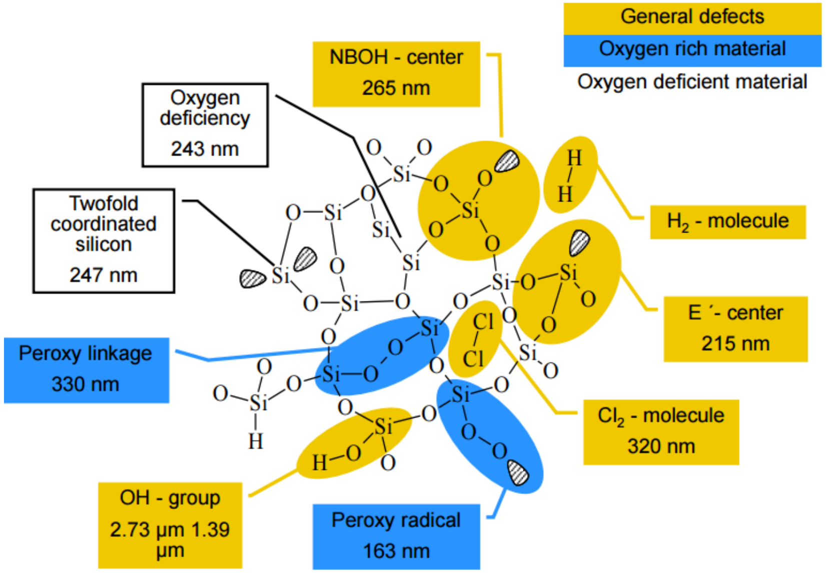
Technical Background

Fused Silica Optical Fiber

- Fused silica is an amorphous material with "defects"
- Defects are disruptions in coordination and ordering of the idealized structure
- Many pathways to defects
  - Fabrication (oxygen stoichiometry, impurities, draw conditions, etc.)
  - Environment (thermal, radiation, etc.)
- Optically active
  - Induced absorptions at specific wavelengths
- Potentially reactive
  - Energy favorable defect sites



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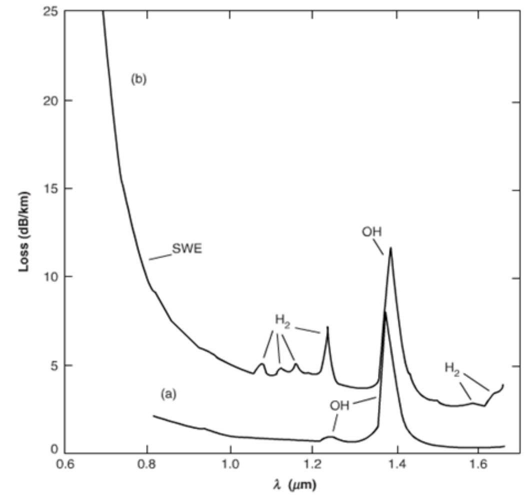
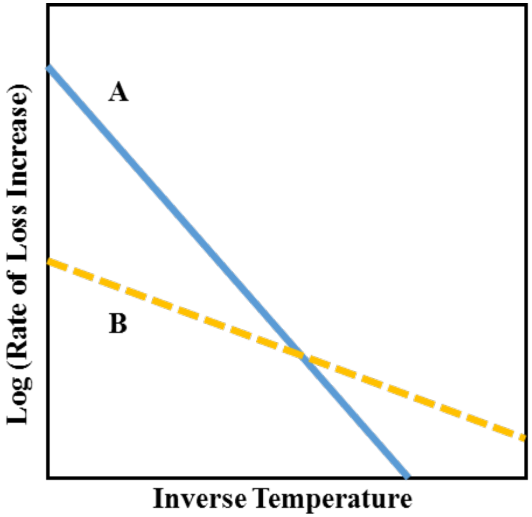
Technical Background

INDUCED SPECTRAL CHANGES

- Susceptibility to chemical constituents can induce absorption at specific wavelengths
- Hydrogen induced attenuation
  - Molecular hydrogen
  - Reactions with glass network
- Complex behavior of loss mechanisms
  - Temperature, time, environment
  - Dopant and defect chemistry
- Deep understanding required to predict
  - Extremely difficult with no knowledge of manufacturing processes
- Temperature induced attenuation
  - Structural relaxation
  - Dopant diffusion

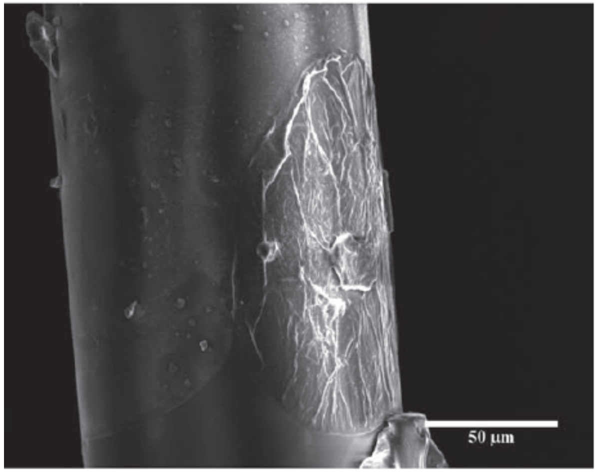


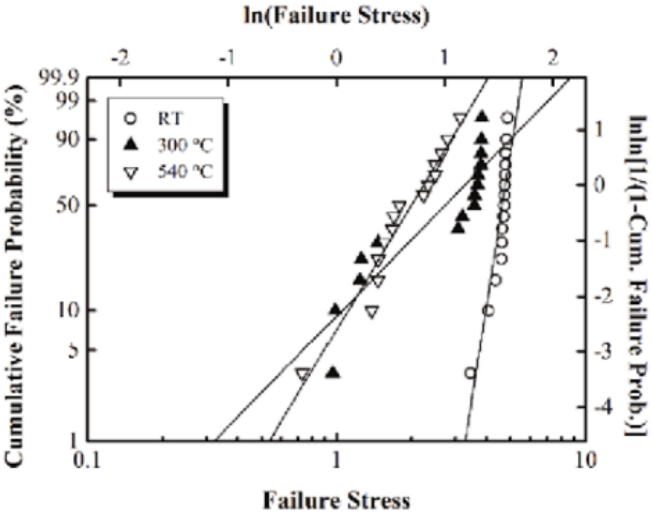


Technical Background

DEVITRIFICATION AND RELIABILITY

- Cristobalite crystallization
  - Stress concentrating flaws
  - Induced thermal mismatch
- Water diffusion controlled pitting
- Strength degradation
  - Micro-cracking
- Optical degradation
  - Broadband scattering loss
- Dependent on material properties

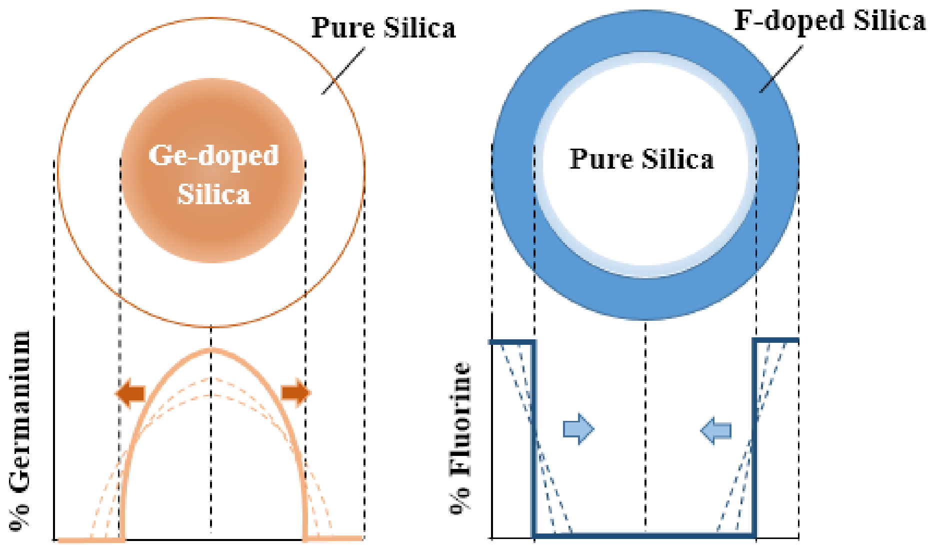




Technical Background

Dopant Migration

- Thermal diffusion
  - Core (Ge, P) to cladding
  - Cladding (F) to core
- Broadening of the RI profile with exposure time
  - Alters light propagation
  - Alters sensor response
- Characterization
  - Fiber refractive index profile
  - Time of flight SIMS



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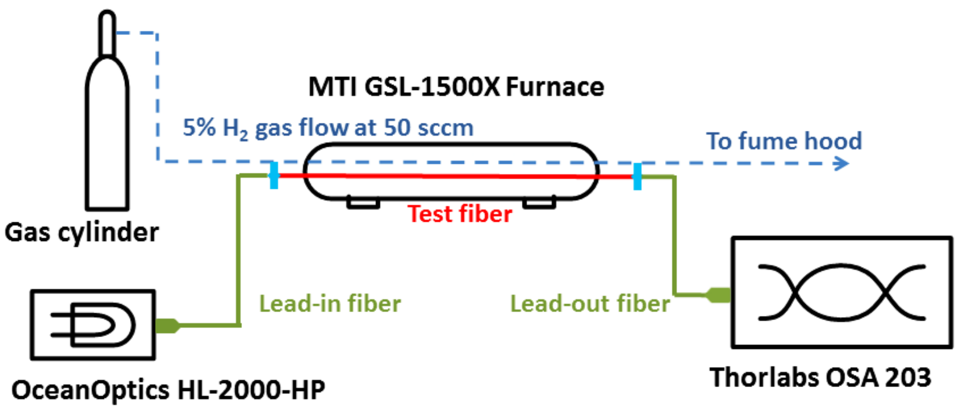
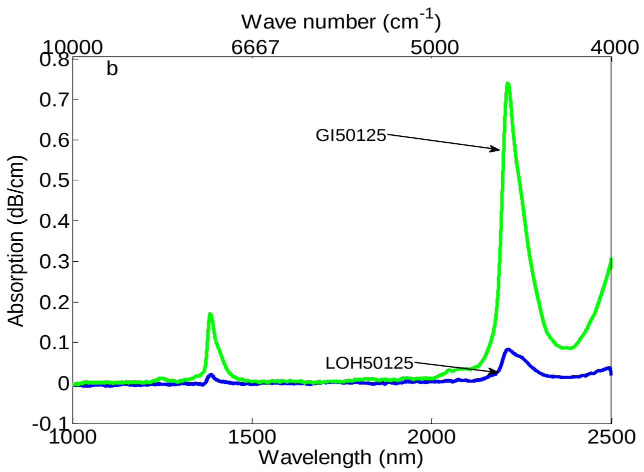
Technical Background

PRIOR COLLABORATION WITH NETL

Schematic of Fiber Test System



In-Situ Optical Spectrum

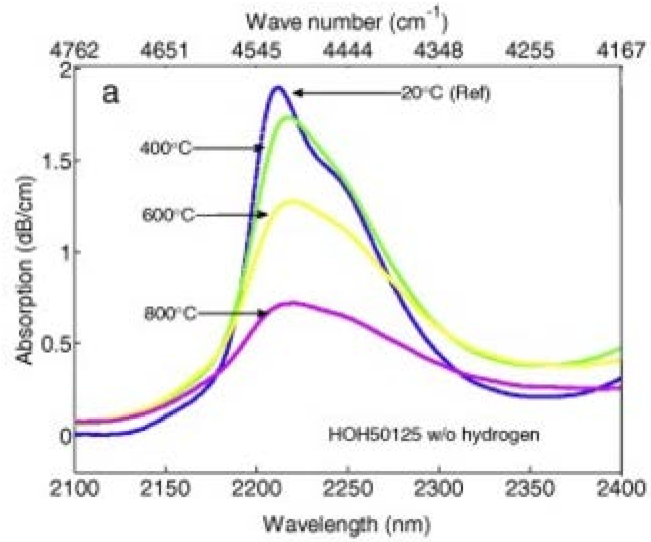


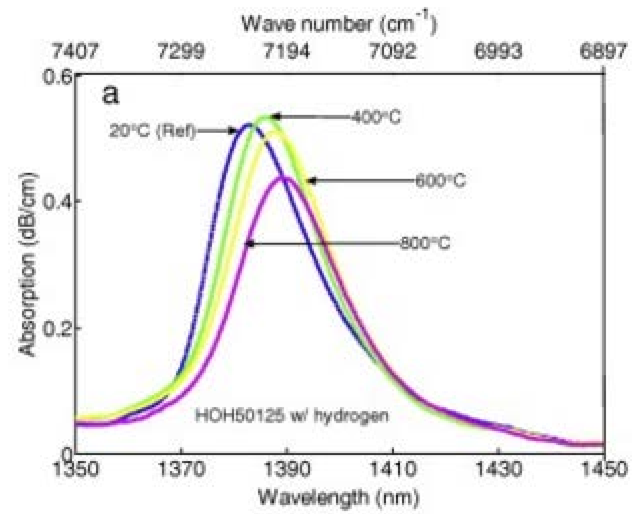
Technical Background

PRIOR COLLABORATION WITH NETL

- Characterized OH absorption in silica optical fiber from 20ºC to 800ºC in-situ.
- Observed decreasing intensity in the 2200 nm band with temperature
- Observed peak wavelength shift in the 1390 nm band with temperature
- Both absorption bands demonstrated a temperature dependence
- It was surmised that the observed phenomena were related to the conversion of the OH spectral components and structural relaxation.

Yu, L., Bonnell, E., Homa, D., Pickrell, G., Wang, A., Ohodnicki, P., Chorpening, S., and Buric, M. (2016). Observation of temperature dependence of the IR hydroxyl absorption bands in silica optical fiber. Optical Fiber Technology, 30, 1-7.





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Potential Significance

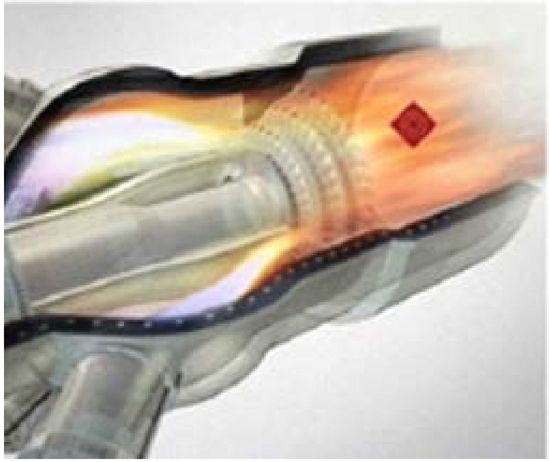
- Understanding of the mechanisms that dictate the behavior of fused silica optical fibers exposed to harsh environments that are not currently understood and severely limit their current use
- Provide a cost effective alternative to sapphire based optical fibers over the intermediate temperature ranges (500 - 1000ºC)
- First-of-its-kind predictive models that accurately describe performance upon exposure to different environments which will allow for selection of the proper fiber type for deployment
- Novel optical fiber cladding materials that are cost effective and readily integrated into current manufacturing techniques
- Enable sensing and monitoring techniques in applications that have yet to be explored due perceived limitations

Relevancy to Fossil Fuel

- Expand the platform of sensing and monitoring technologies that can be integrated into critical components of gasifiers, turbines, and boilers

- Increase operational efficiencies, improve the safety and reliability, and reduce the emissions of power generation

- Provide operators with an alternative to sapphire based fiber optic sensing systems which are often exotic and expensive



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Statement of Project Objectives

- **Goal:** Develop a comprehensive and complete understanding of the optical and mechanical stability of fused silica optical fibers at high temperatures under various gaseous species conditions.
- **Objective:** Conduct comprehensive testing and analysis of the interactions of fuel gas stream species with state-of-the-art optical fibers to understand the induced devitrification, chemical reactions, and the diffusion of chemical species on the mechanical and optical performance.
- **Objective**: Novel and traditional fused silica fibers will be drawn under various process conditions to fully understand the fundamental mechanisms that govern performance.
- **Benefit**: The developed predictive models will enable the selection of optical fibers for specific operating environments.

Project Tasks

- Task 1.0 – Project Management and Planning
- Task 2.0 – Literature and Optical Fiber Market Review
  - Preform Literature Review
  - Perform Commercial Optical Fiber Market Review
  - Generate Literature and Market Review Report
- Task 3.0 – Develop Test Plan

Deliverables

**Included in corresponding Quarterly Report(s)*

Task	Title/Description	Deliverable
1	Project Management and Planning	Revised Project Management Plan
2	Literature and Commercial Optical Fiber Market Review	Optical Fiber Literature Review and Market Report
3	Develop the Test Plan	Environmental Test Plan
5	Evaluate the Mechanical Stability of Comercial Optical Fibers	Mechanical Stability Test Report
6	Evaluate the Dopant Stability of Commercial Optical Fibers	Dopant Stability Test Report
7	Evaluate the Optical Stability of Commercial Optical Fibers	Optical Stability Test Report
8	Prepare and Submit Final Report	Final Report

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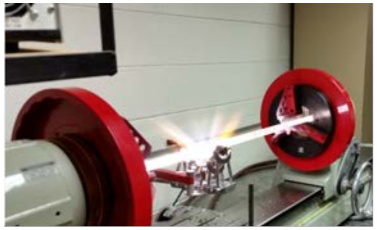
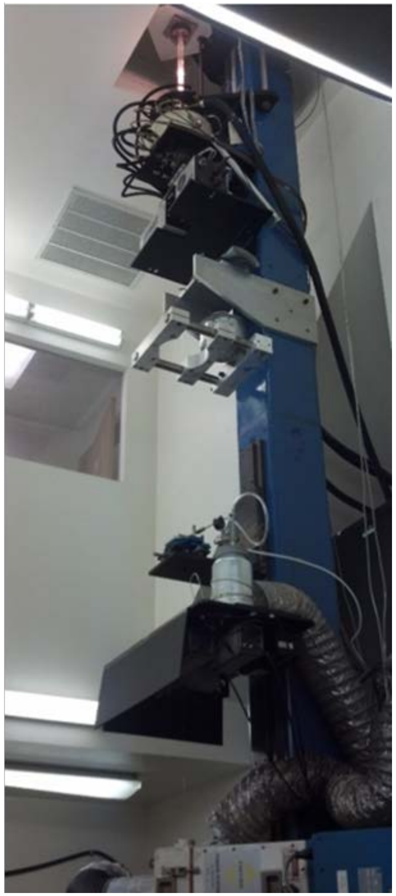
Project Tasks

- Task 4.0 – Prepare for Optical Fiber Stability Testing
  - Acquire Materials and Supplies
  - Construct and Commission the Environmental Testing Facilities
- Task 5.0 – Evaluate the Mechanical Stability of Commercial Optical Fibers and Drawn Fibers
  - Draw Fused Silica Fibers on Virginia Tech Draw Tower
  - Conduct Environmental Testing
  - Perform Data Analyses and Interpretation
  - Generate Mechanical Stability Test Report





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Project Tasks

- Task 6.0 – Evaluate the Dopant Stability of Commercial Optical Fibers
  - Conduct Environmental Testing
  - Perform Material Characterization
  - Perform Data Analysis and Interpretation
  - Generate Dopant Diffusion Test Report
- Task 7.0 – Evaluate the Optical Stability of Commercial Fibers
  - Conduct Environmental Testing
  - Perform Data Analyses and Interpretation
  - Generate Optical Stability Test Report
- Task 8.0 – Prepare and Submit Final Report

Project Schedule

GANTT CHART		Project Year 1				Project Year 2				Key Dates	
Task #	Task Name	Q1	Q2	Q3	Q4	Q1	Q2	Q3	Q4	Start Date	End Date
1	Project Management and Planning									10/1/2016	12/30/2016
M1	MILESTONE 1									12/31/2016	
2	Literature and State of the Art Optical Fiber Market Review									10/15/2016	3/30/2017
2.1	Perform Literature Review									10/15/2016	3/15/2017
2.2	Perform Commercial Optical Fiber Market Review									10/15/2016	3/15/2017
2.2	Generate Literature Review and Market Report									1/1/2016	3/30/2017
M2	MILESTONE 2									2/28/2016	
3	Develop Test Plan									11/15/2016	3/30/2017
M3	MILESTONE 3									3/30/2017	
4	Prepare for Optical Fiber Stability Testing									12/15/2016	3/30/2017
4.1	Acquire Materials and Supplies									12/15/2016	4/15/2017
4.2	Construct and Commission the Environmental Test Facilities									4/1/2017	6/30/2017
M4	MILESTONE 4									6/30/2017	
5	Evaluate the Mechanically Stability of Commercial Optical Fibers and Drawn Fibers									4/1/2017	3/30/2018
5.1	Draw Fused Silica Fibers on Virginia Tech Draw Tower									4/1/2017	9/15/2017
M5	MILESTONE 5									9/30/2017	
5.2	Conduct Environmental Testing									6/15/2017	11/15/2017
5.3	Perform Data Analyses and Interpretation									9/16/2017	12/30/2017
5.4	Generate Mechanical Stability Report									12/1/2017	12/30/2017
6	Evaluate the Dopant Stability of Commercial Optical Fibers									10/1/2017	2/15/2018
6.1	Conduct Environmental Testing									10/15/2017	1/15/2018
6.2	Perform Material Characterization									11/1/2017	2/15/2018
6.3	Perform Data Analyses and Interpretation									2/1/2018	3/15/2018
6.4	Generate Dopant Diffusion Test Report									3/1/2018	3/30/2018
M6	MILESTONE 6									3/30/2018	
7	Evaluate the Optical Stability of Commercial Fibers									7/15/2017	8/1/2018
7.1	Conduct Environmental Testing									7/16/2017	5/15/2018
7.2	Perform Data Analyses and Interpretation									4/1/2018	8/15/2018
7.3	Generate Optical Stability Test Report									8/15/2018	9/15/2018
8	Prepare and Submit Final Report									9/1/2018	9/30/2018
M7	MILESTONE 7									9/30/2018	

Project Milestones

- Technology review
- Performance testing
  - Test plan
  - Test facilities
- Novel fiber development
  - Design and fabrication
- Characterization
  - Optical
  - Mechanical
  - Chemical
- Final Report

M. #	Title/Description	Completion Date
1	Project Management Plan	Q1
2	Literature and State of the Art Optical Fiber Review	Q2
3	Optical Fiber Test Plan	Q2
4	Commission Optical Fiber Test Facilities	Q3
5	Virginia Tech Optical Fiber Fabrication	Q4
6	Fiber Mechanical and Dopant Stability	Q6
7	Final Report	Q8

Milestone Success Criteria

- High temperature fiber test facilities
  - Design and construct fiber testing facilities
- Fused silica fiber fabrication and testing
  - Fabricate optical fibers design for high temperature operation
  - Demonstrate adequate lengths and tensile strength
- Testing of commercial optical fibers
  - Evaluate the performance of commercial optical fibers
  - Develop comprehensive understanding of fundamental mechanisms

ID	Title	Description	M.S.	Completion
SC1	High Temperature Fiber Test Facilities	1. Maximum Temperature = 900°C 2. Minimum Fiber Length > 0.5 m 3. Minimum two gases: H_2, O_2, N_2, CO, CO_2	M4	Q3
SC2	Fused Silica Fiber Fabrication and Testing	1. Minimum Length of 1 m 2. Minimum Tensile Strength of 100 kpsi	M5	Q4
SC3	Testing of Commercial Fibers	1. Minimum: 3 fiber types 2. Minimum Temperature Maximum: 800°C	M6	Q7

Risk Management Plan

- Risk Management Planning
  - Addresses potential risks relative to (1) project scope and complexity, (2) technical challenges, (3) resources, (4) research team dynamics and (5) overall project management.
- Risk Identification
  - Continuously conducted at the bi-weekly meetings. The PI shall be responsible for further analyses and prioritization of the risks and communicating this information to DOE. Risk
- Analysis
  - Includes “negative” classifications based on (a) Impact, (b) Probability and (c) Schedule. The levels of risk are: “High”, “Medium”, and “Low”.
- Risk Planning / Mitigation
  - The PI and research team will continuously evaluate risks and identify the appropriate solutions at the bi-weekly meetings and/or specific project meetings.
- Risk Monitoring and Control
  - All risks identified and associated actions will be documented in the bi-weekly meeting agendas

Project Risks

- Adequate Signal from Tested Optical Fibers
  - The short fiber lengths (~ 1m) that are tested at high temperatures (>500ºC) tend to yield low signal to noise (or background) ratios for attenuation measurements. Therefore, induced loss mechanisms may not be identified or analyzed inaccurately.
  - (1) High Impact Risk, (2) Low Risk Probability and (3) Low Schedule Risk.
  - Risk is mitigated through the preliminary testing that has been conducted, in our prior collaborative work with a NETL research team. An exhaustive literature review will also be performed to identify potential absorption peaks for particular chemical species. Two different light sources and optical spectrum analyzers will be employed to assure stability and perform measurements over a broad wavelength range (400 – 2500 nm)
- Adequate Strength for "As-Drawn" Fused Silica Fibers
  - The tensile strength of fused silica fibers fabricated on the Virginia Tech Draw Tower may not possess the tensile strength required for mechanical testing.
  - Categorized as a (1) Medium Impact Risk, (2) Low Probability Risk and (3) Low Schedule Risk.
  - Risk is mitigated through preliminary work that has demonstrated our ability to draw a number of fiber types and preliminary bend tests performed on "as-drawn" fibers has shown a minimum tensile strength in excess of 300 kpsi.

Project Status

- Project start date: October 1, 2016
- Project management planning
- Identification of commercial optical fibers
- Test facilities and training
- Graduate student selection

Acknowledgements

Virginia Tech

Center for Photonics Technology (CPT)

Gary Pickrell

Anbo Wang

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Project Manager: Barbara A. Carney

Jessica Mullen

THANK YOU FOR YOUR TIME