Investigation of High Temperature Silica Based Fiber Optic Sensor Materials

Motivation

To meet performance metrics of energy conversion systems such as advanced boiler systems, combustion turbines and solid oxide fuel cells, operators are looking towards advanced sensor and monitoring systems to improve their reliability, efficiency, safety and security. The high temperature and harsh environments that are required to operate these systems often necessitate the use of fiber optic sensors over more traditional sensing technologies. To this end, there is a need for fiber optic sensing system integration at a realistic cost point and an alternative to sapphire fiber optics, which are more expensive and require additional expertise

Objectives

- Investigate and characterize the performance of commercially available optical fibers at elevated temperatures in replicated fuel gas streams
- Perform a comprehensive study of the interactions with the chemical constituents with respect to structural, optical, and mechanical stability
- Develop design strategies to assure reliable performance of deployed optical fibers and sensors.

Scope

- Study fused silica optical fiber sensor materials by elucidating performance dependencies on the stoichiometry of fiber fabrication, incorporated chemical species, thermal history, material grades, dopants, and fiber design.
- Fiberize several types of fused silica to evaluate the impact of stoichiometry and chemistry on thermal stability

Start Date

October 1, 2016

Researchers

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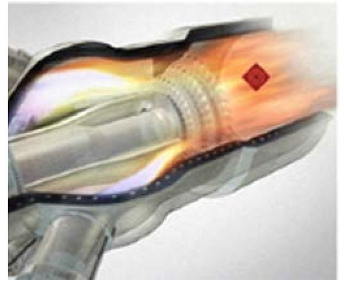
Sponsors

National Energy Technology Laboratory of the U.S. Department of Energy (DOE)

1 Fiber Optic Sensing in Harsh Environments

- The lack of commercial solutions for these demanding applications, coupled with drivers such as improved energy efficiency, and reduced emissions, has created a growing market opportunity that is anticipated to reach $4.5 billion by 2018 .

Monitoring of energy conversion systems.



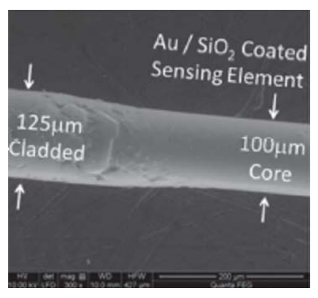
Schematic of gas turbine (NETL).

- Energy conversion systems such as advanced boiler systems, combustion turbines and solid oxide fuel cells have become increasingly more complex and subject to harsher and higher temperature environments.

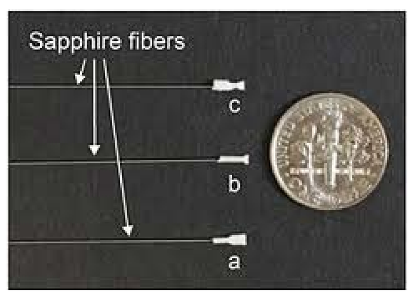
Power Generation Technology Needs [1]

	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

- Mature fiber optic sensing technologies, discrete and distributed, have the potential to meet these market demands, but to date, the daunting material challenges associated with the extremely high temperature and harsh environments have slowed implementation in power generation system



SEM image of novel fused silica based fiber optic sensor [2].



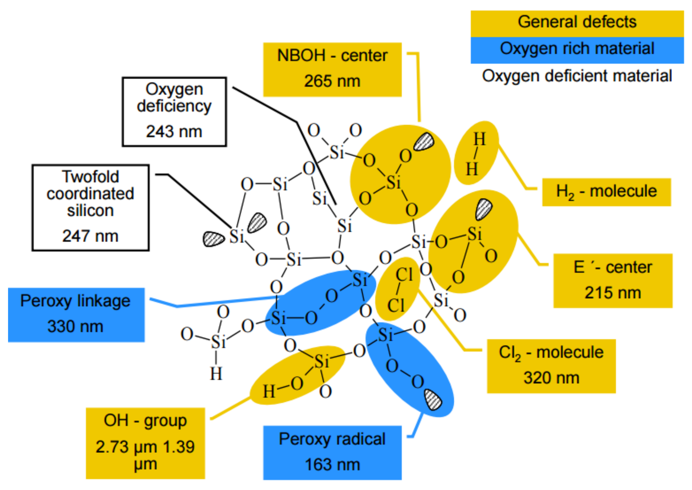
Sapphire based single point FO sensors.

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

[2] Ohodnicki, P.R, M.P. Buric, T.D. Brown, C. Matranga, C. Wang, J. Baltrus and M. Andio. "Plasmonic Nanocomposite Thin Film Enabled Fiber Optic Sensors for Simultaneous Gas and Temperature Sensing at Extreme Temperatures." Nanoscale 5, no. 19 (2013): 9030-9039.

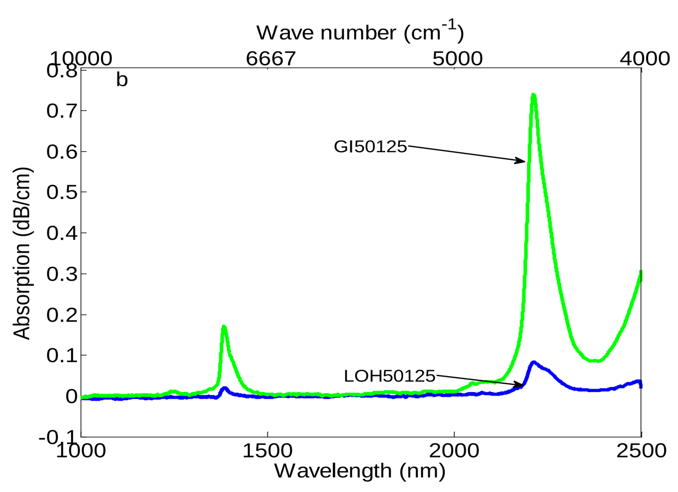
2 Fused Silica Based Optical Fiber

- Although there is some ambiguity in defining the concept of a defect in an amorphous material, a working definition is a disruption in coordination and ordering of the idealized structure of the amorphous silica network.
- There are a number of types of defects that are present in the fused silica glass upon manufacture, to include those that are more prevalent in glasses that are fabricated under oxygen deficient or rich conditions, those that include impurities such as chlorine and hydroxyl, and specific dopants such as germanium and fluorine.



Schematic of common defects in fused silica glass [3]

- Several types of defects that are created or become exacerbated upon fiber draw such as "drawing induced" E centers and non-bridging oxygen hole centers (NBOHC) depending on parameters such as draw speed, tension, and temperature, as well as the dopant type and profile in the preform.
- Optically active defects will induce absorptions at specific wavelengths and act as reaction sites for impurities, such as H_2, during and post fabrication. The defects sites are thermally active and, the distribution of the defect population can become transient upon exposure to an energy source, such as UV and nuclear radiation



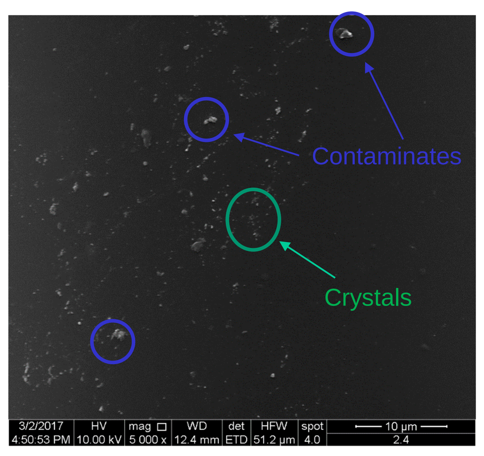
Induced attenuation for silica core fiber and graded index multimode fiber upon H_2 exposure at elevated temperatures (800ºC) [4].

[3] Nürnberg, Frank. "Fused Silica Challenges", CERN, October 20, 2015.

[4] Yu, Li, Elizabeth Bonnell, Daniel Homa, Gary Pickrell, Anbo Wang, PR Ohodnicki, Steven Woodruff, Benjamin Chorpening and Michael Buric. "Observation of Temperature Dependence of the Ir Hydroxyl Absorption Bands in Silica Optical Fiber." Optical Fiber Technology 30, (2016): 1-7.

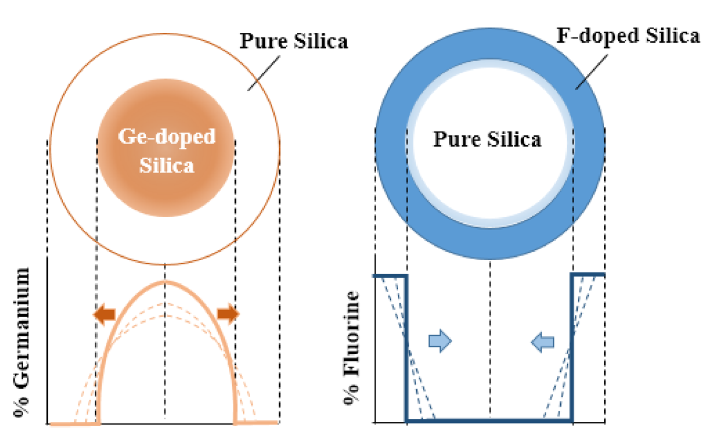
3 Devitrification and Dopant Migration

- Mechanical weakening of fused silica optical fibers is governed by the surface crystallization at high temperatures (~800ºC) and the diffusion of water below this temperature.
- Cristobalite crystals on the surface of the fused silica fiber act as stress concentrating flaws and the resulting thermal mismatch between the glass and crystals induces micro crack formation upon changes in temperature.
- Water diffusion-controlled pitting of the surface governs the degradation of strength.



SEM image of optical fiber surface with crystal formation and contamination upon thermal treatment.

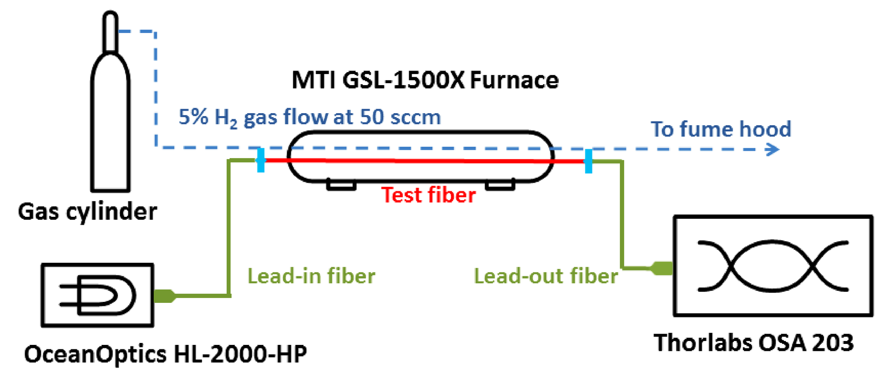
- Thermal diffusion of dopant ions will broaden the refractive index profile and alter the characteristics of light propagation in the fiber.
- Germanium is the most common dopant used in traditional single and multimode optical fibers; Ge atoms, in silica fiber, diffuse substantially over short durations at elevated temperatures; ~4 x10^{-16} m^2/s in the range of 1200ºC >T >.1400ºC.
- "Pure silica core" fibers typically maintain a cladding glass that is doped with fluorine to reduce the refractive index; F atoms, in fused silica, maintain a diffusion coefficient of ~5 x10^{-7} m^2/s at 1300ºC.



Schematic of (a) germanium diffusion in fiber optic and (b) fluorine diffusion in pure silica core fiber.

4 Induced Optical Attenuation

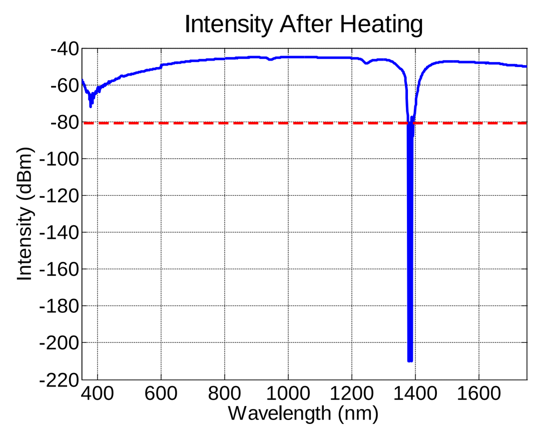
- Broadband Light Source
- VIS to Mid IR optical detection (0.4µm – 2.5µm)
- Controlled atmosphere (N_2, CO_2, CO, H_2O, Air)
- Elevated temperatures (RT - 1200ºC)

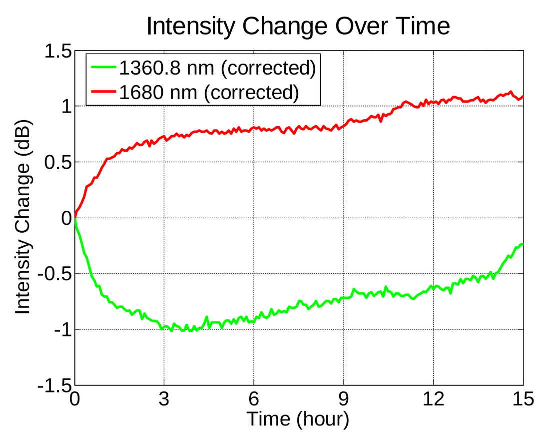


Schematic of heated H_2 susceptibility test facilities [4].

Monitoring of spectral changes as a function of time and temperature

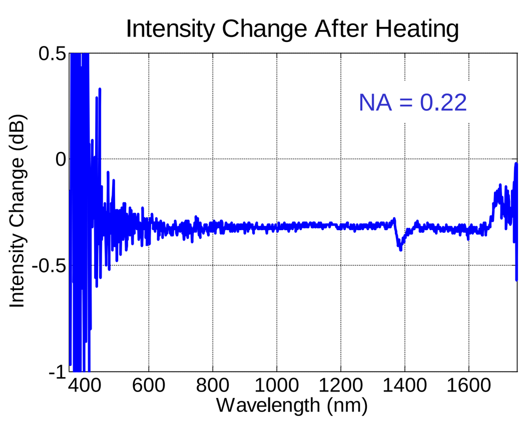
- Evaluation of high OH step index pure silica multimode fiber at ~900ºC in air

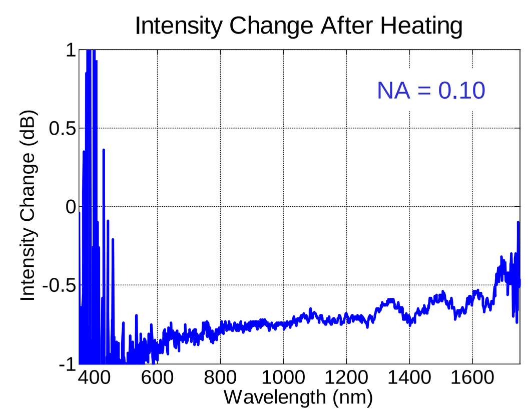




Influence of dopant concentration and refractive index profile on performance

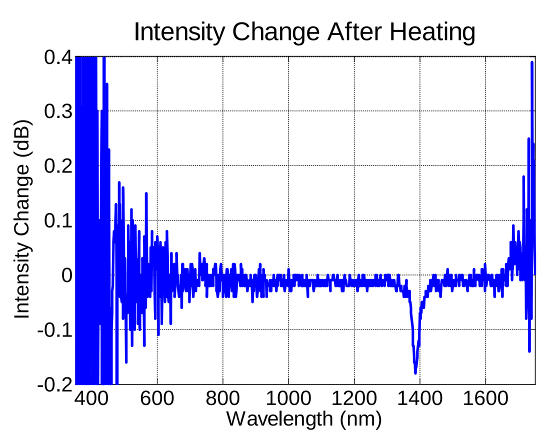
- Evaluation of two pure silica core fiber types with different NA (0.10 vs. 0.22) at ~1200ºC in air





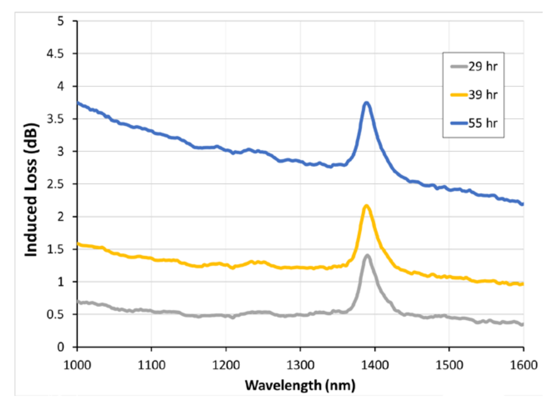
Exposure to different gas constituents

- Evaluation of low OH pure silica core multimode fiber in 5% CO_2/95% N_2 at ~1200ºC.



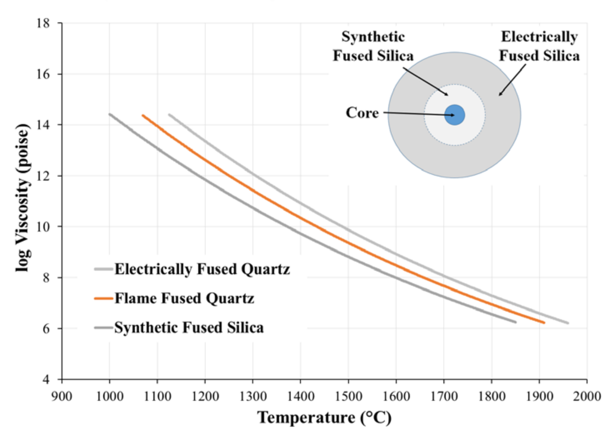
Performance of single mode fiber

- Evaluation of SMF28 single mode fiber at ~1200ºC in air for 55 hours.



5 Thermally Tolerant Optical Fiber

- Commercial optical fibers are not optimized for application in energy related environments
- Fibers with several different types of silica glass will be drawn on our draw tower to evaluate the extent and rate of devitrification, as compared to commercial optical fibers.
- The influence of the draw parameters will be investigated with the ultimate goal of producing an optical fiber specifically designed for high temperature stability.



Viscosity of different silica based glass types as a function of temperature. Proposed fiber design (insert).

Glass working lathe (top left) and optical fiber draw facilities (bottom left and right) at Virginia Tech.

NEXT STEPS

NEAR TERM TASKS

- Perform literature and commercial optical fiber market review
- Selected commercial optical fiber types and develop test plan
- Design, construct and commission the high temperature optical fiber test facilities.
- Conduct preliminary testing of selected optical fibers in N_2, air, and CO_2 at elevated temperatures (900-1200ºC)
- Perform optical and scanning electron microscopy on the thermally treated fibers

MILESTONE 2 : Optical Fiber Test Plan	***3/30/17***
MILESTONE 3 : Commission Optical Fiber Test Facilities	***3/30/17***
MILESTONE 4 : Commercial Fiber Test Report	***12/31/17***
MILESTONE 5 : Prototype and Commercial Fiber Test Report	***6/30/18***
MILESTONE 6 : Final Report	***12/31/18***

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