# Reduced Mode Sapphire Optical Fiber and Sensing System

## Motivation

Monitoring systems have been shown to improve the reliability, efficiency, safety and security of advanced combustion and gasification. The ability to tailor the structure of single crystal sapphire fibers has the potential to enhance the performance of high temperature fiber optic sensors. The proposed sapphire fiber waveguide design will overcome the harsh environment challenges that severely limit the integration of mature optical fiber sensing technologies in new power plant control systems.

## Objective

- Develop a micro-structured sapphire fiber sensor with low modal volume (LMV).
- Demonstrate a Raman scattering based distributed temperature sensing system.

## Scope

- Evaluate the improvement introduced by the Low Mode Volume sapphire fiber in a wafer-based sensor system.
- Develop a prototype sensing system with fully-distributed sensing capacity and evaluate its performance in a laboratory test environment for operation at temperatures over 1100°C.

## Start Date

January 1, 2014

## Researchers

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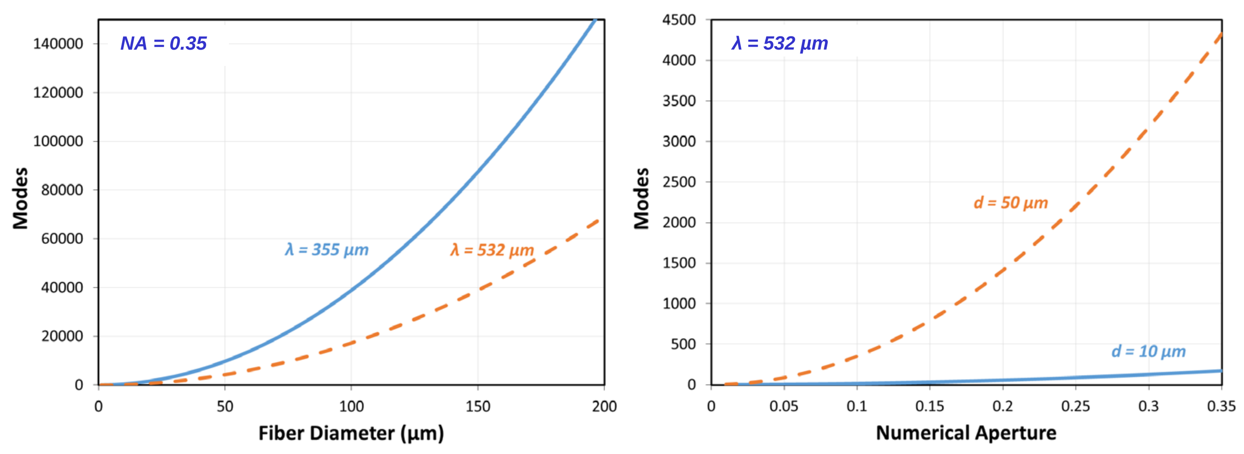
## Sponsors

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

## 1 Low Modal Volume Sapphire Fiber



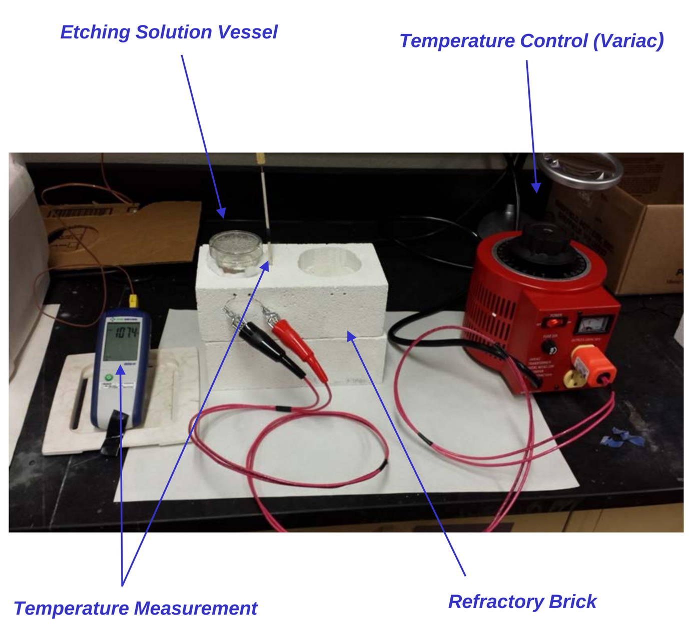

*The number of modes as a function of the sapphire fiber diameter (left) and Numerical Apertures (right).*

- The large modal volume of sapphire fibers limits the performance of Raman scattering FO sensing systems.
- The number of modes, M, in fibers with large V parameters can be estimated by $M = \left({}^{4}/_{\pi^2}\right) V^2$ .
- The number of modes can be modified by reducing the core size and/or the NA, as seen in the following relationship, where a is the fiber radius, and $\lambda$ is the operating wavelength, $n_{co}$ and $n_{cl}$ are the refractive indices of the core and the cladding, respectively.

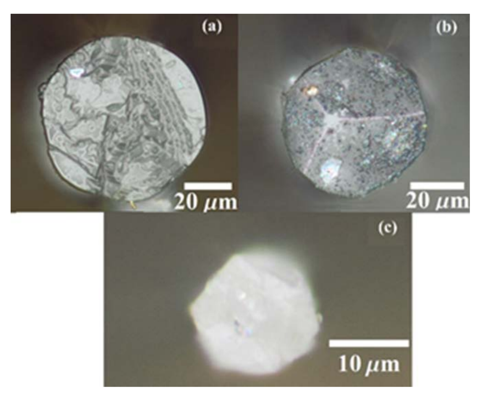
$$V = \frac{2\pi}{\lambda}\, a\, NA = \frac{2\pi}{\lambda}\, a \sqrt{n_{co}^2 - n_{cl}^2}$$

- A reduction in fiber diameter and/or numerical aperture will reduce the V number, and in turn, significantly reduce the number of modes propagating in the fiber.

## 2 Single Crystal Sapphire Etching




*Single Crystal Etching Station.*




*VT Etching Technique Reduces Sapphire Fiber Diameter . (a) "As-Received" Fiber: 70 μm, (b) Etched Fiber : 50 μm, (c) Etched Fiber: 15 μm.*

*Sapphire wafer masked with Teflon™ during etching process.*

- The microstructure and size of single crystal sapphire fibers are tailored via a wet acid etching technique. Fibers are exposed to sulfuric ($H_2SO_4$) and phosphoric ($H_3PO_4$) at elevated temperatures (300°C - 500°C)

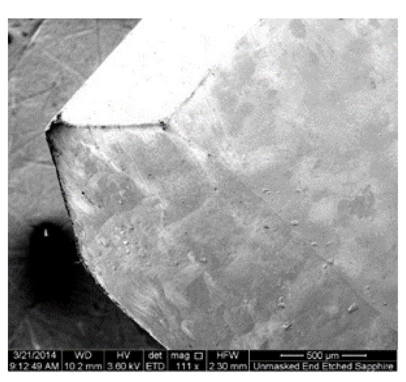
$$Al_2O_3 + 6H_3PO_4 \leftrightarrow 2Al(H_2PO_4)_3 + 3H_2O \quad (1)$$

$$Al(\,H_2PO_4\,)_3 + Al_2O_3 \leftrightarrow 3AlPO_4 + 3H_2O \quad (2)$$

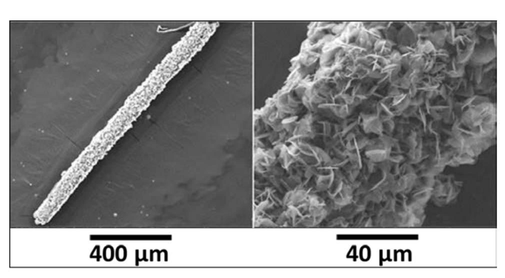
$$Al^{+3} + nSO_4^{-2} \leftrightarrow Al_2(SO_4)_n^{\,3-2n} \quad (3)$$

$$2Al^{+3} + 3SO_4^{\,-2} \leftrightarrow Al_2(SO_4)_3 + xH_2O \quad (4)$$

## 3 Sapphire "Microstructuring"



*Angled Edge on Single Crystal Sapphire Wafer via VT Etching Process*




*Conversion of Single Crystal Sapphire Fiber to mullite via VT SiC masking techniques.*

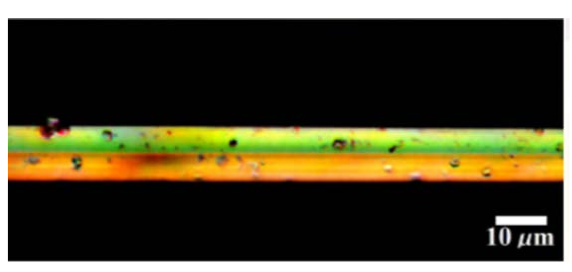
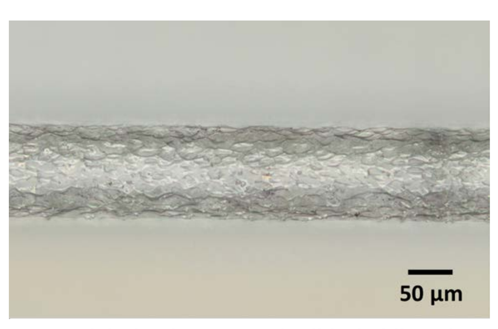


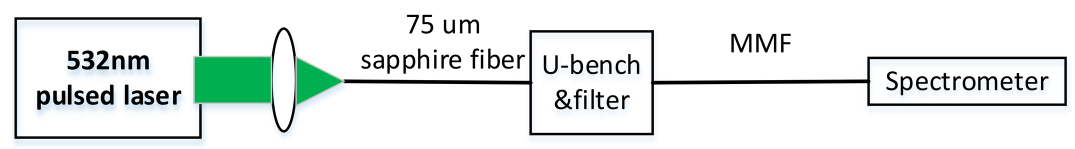

*Image of etched sapphire fiber via polarized light; the birefringence is due to the attenuated hexagonal structure of the material.*

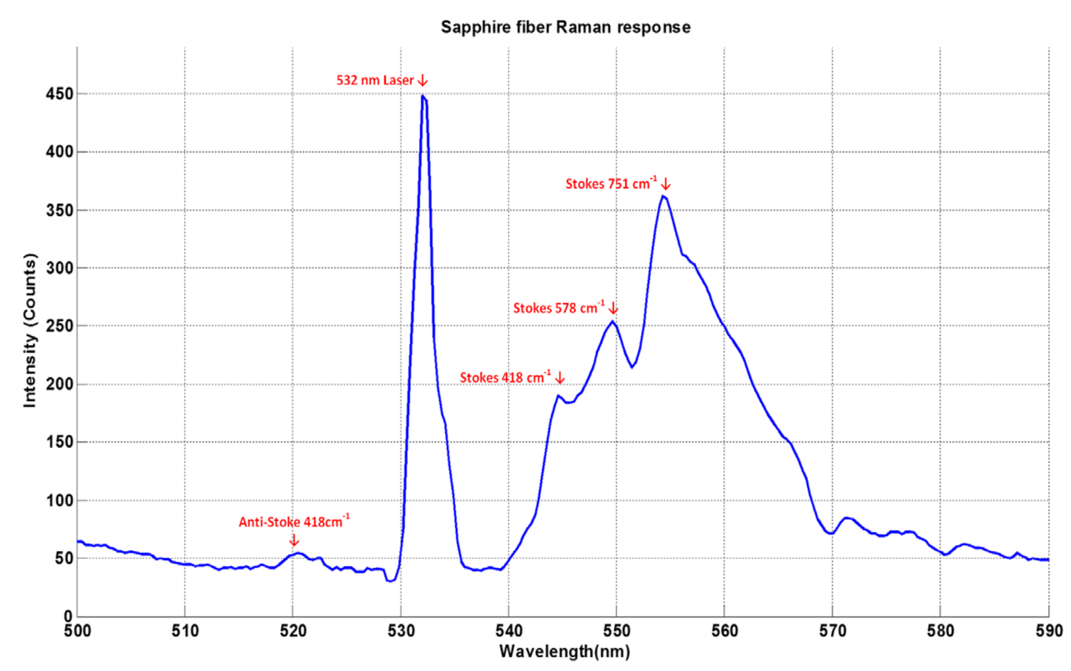



*Potential Alumina Porous/Solid Cladding via Virginia Tech "Conversion" Technology.*

- Masking techniques are currently being explored to form features in the sapphire fiber via Teflon ™ and silica masking techniques.
- Reflective edge features and attenuated hexagon shaped fibers have been demonstrated via selected processing parameters.
- Novel silicon carbide masking and "conversion" techniques are currently being investigated as potential cladding processes.

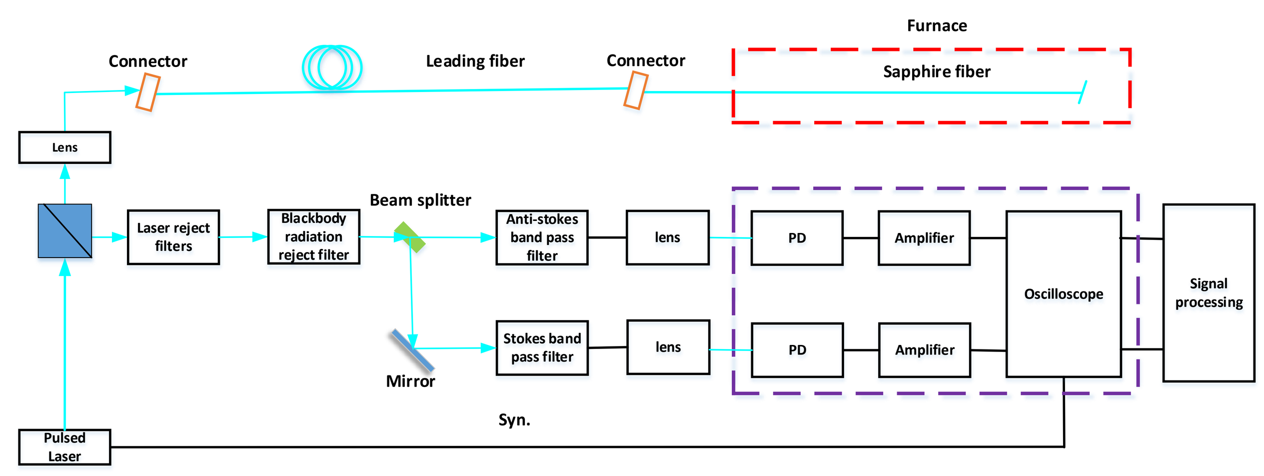
## 4 Raman Scattering in Sapphire Fibers



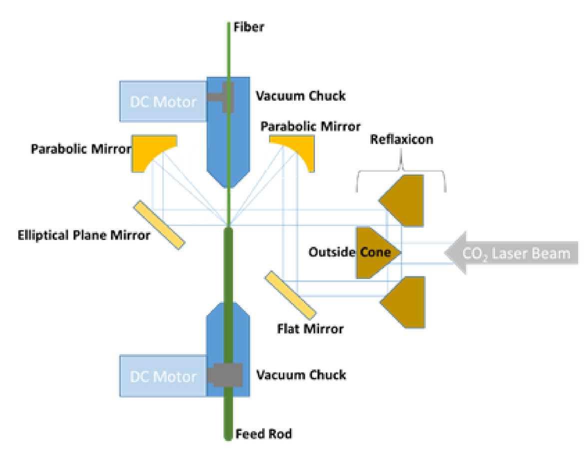

- The temperature information is deduced from the intensity ratio of anti-Stokes signal to Stokes signal, which changes significantly as temperature increases. At room temperature, anti-Stokes signal is relatively week.
- The Stokes peaks overlap due to relatively low spectrometer resolution.

## 5 Distributed Sensing System design

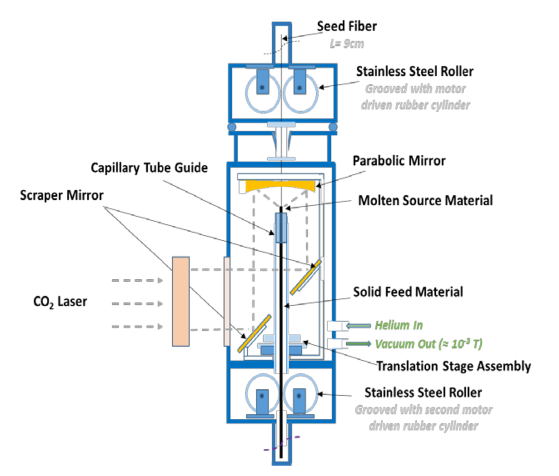



- High speed time based measurement system records the Stokes and anti-Stokes signal from the sapphire fiber simultaneously to deduce the temperature distribution along the sapphire fiber.
- The influence of blackbody radiation at high temperature will be suppressed by using a short wavelength laser source and applying narrow band pass filters.

## 6 Micro-structured Sapphire Fiber Growth




*Laser Heated Pedestal Growth system design with reflaxicon optic (U.S. Patent 4,421,721)*




*Laser Heated Pedestal Growth system design with graded reflectivity mirror optic (U.S. Patent 5,607,506).*

- A Laser Heated Pedestal Growth (LHPG) system will be designed and installed to support the optimization of geometries, structures, chemistries, and optical properties of the fibers specific to the Raman scattering sensing technology.
- The stable $CO_2$ laser beam is guided and transformed into a "Super-Gaussian" or "top-hat" shape and/or "ring" prior to focus onto the seed crystal.

## NEXT STEPS

**NEAR TERM TASKS**

- Measure the intensity of blackbody radiation experimentally in a sapphire fiber at high temperature.
- Compare the intensity of Raman signal quality using 532nm and 355nm laser sources for system optimization.
- Evaluate the dependency of the sapphire etching process per temperature, solution chemistry, and time.
- Compare the transmission properties of etched and pristine single crystal sapphire fibers.
- Investigate Teflon™ , silica and newly developed SiC masking techniques.
- Design and construct of the first generation (Gen 1) LHPG system

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| **MILESTONE 2 : Model Low Modal Volume (LMV) Sapphire Fiber and Sensing System** | ***12/31/14*** |
| **MILESTONE 3 : Demonstrate the Fabrication of LMV Sapphire Fiber** | ***6/30/15*** |
| **MILESTONE 4 : Demonstrate a Raman Scattering Fiber Optics Sensing System** | ***12/31/15*** |
| **MILESTONE 5 : Evaluate the Prototype Fiber Optic Sensing System** | ***6/30/16*** |
| **MILESTONE 6 : Generate Final Report** | ***12/31/16*** |