Novel Optical Fibers for High Temperature In-Situ Miniaturized Gas Sensors

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NETL CrossCutting Review Meeting

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Motivation

- Gas Species Sensor for harsh environments
  - Coal Gasification products
  - Advanced power generation
- Gas species determination
  - Process optimization
  - Process fault/Failure identification

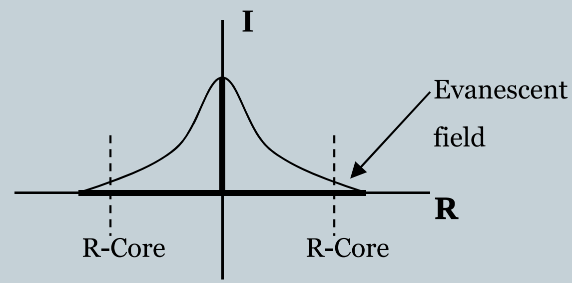
Scope of Work

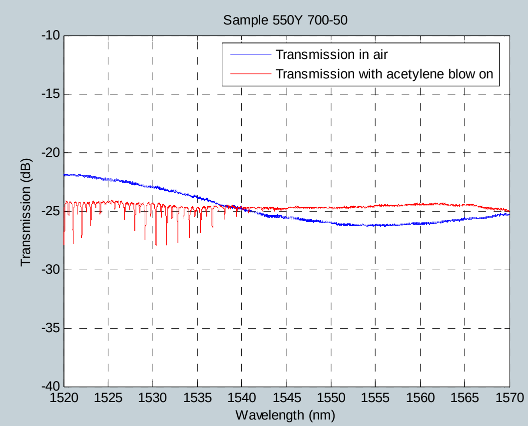
- Scope:
  - The objective of the proposed program is to develop novel modified fiber-based materials for miniaturized optical sensors for in-situ detection of a wide range of gaseous species at high temperatures and in harsh environments.

Background: Technical Approach

- Transmission of light is not 100%
  - Loss from impurities
  - Loss from evanescent field
  - Loss due to direct absorption
- The Evanescent field is a portion of the EM field that extends beyond the core/cladding boundary

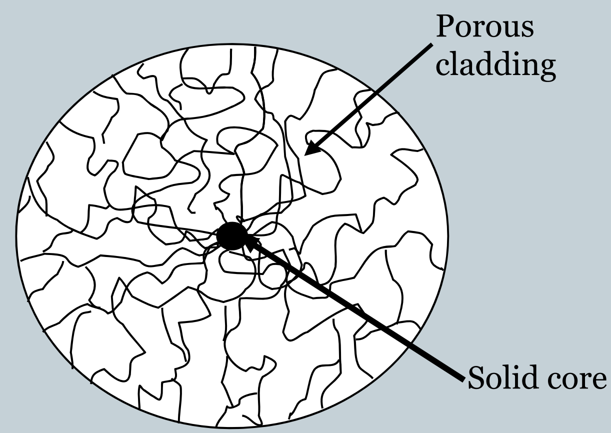
- The interaction between the evanescent field or injected light and a gas provides an absorption spectra
- This spectra is specific to the gas

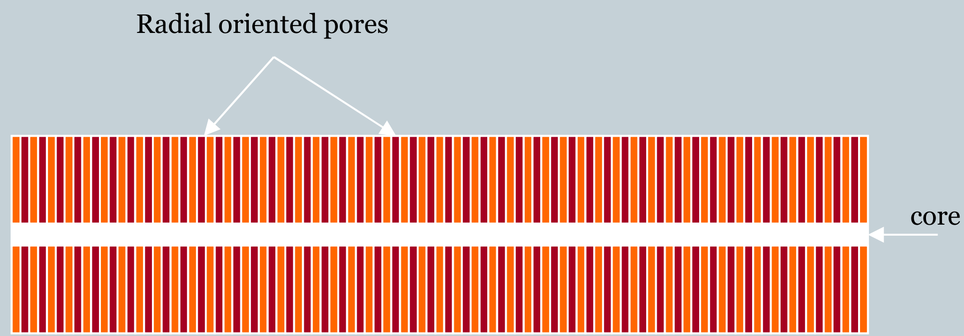




Background: Technical Approach

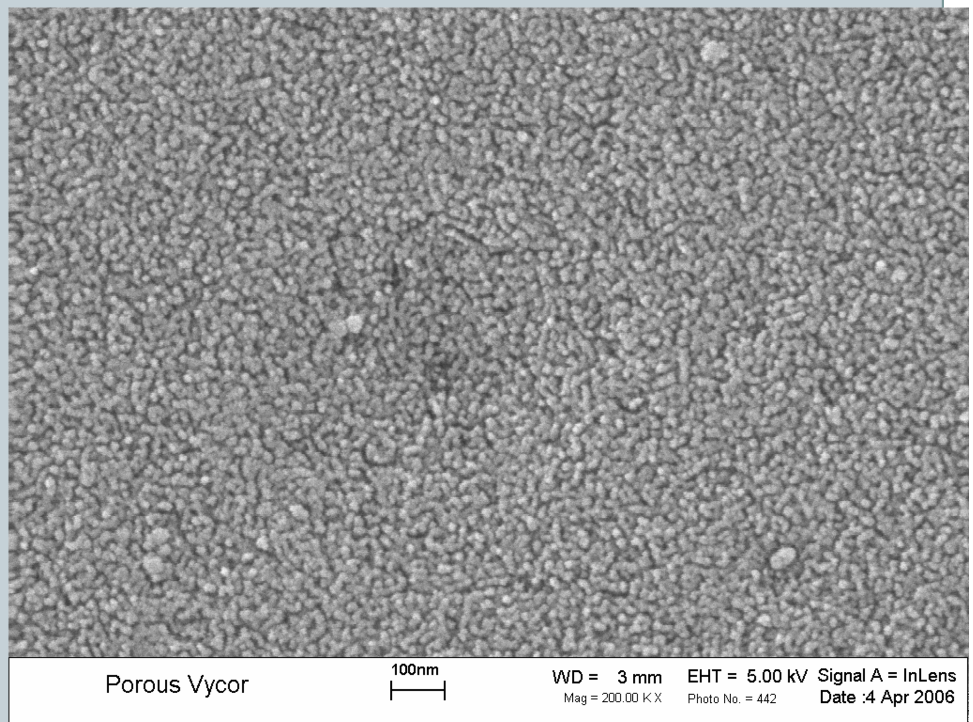
- Design is fabricated by
  - A spinodally phase separating cladding glass
    - Produces a 3-D interconnected cladding
    - Porosity has a radial orientation
    - Porosity connects the fiber surface to the core





Background: Porous Glass

- Porous glass are made from phase separable glass compositions
  - Article is made desired shape
  - Article is annealed
  - Annealing induces phase separation so two interconnected phases are present
  - One of the phases is removed
  - Porous skeleton remains with a 3-D interconnected porosity



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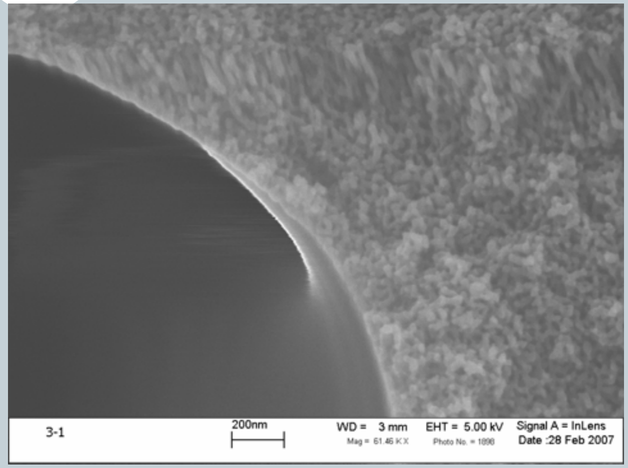
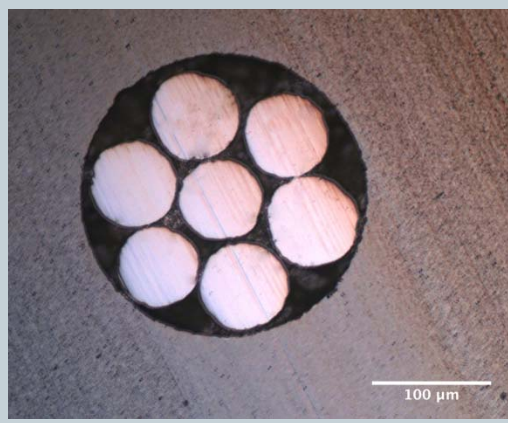
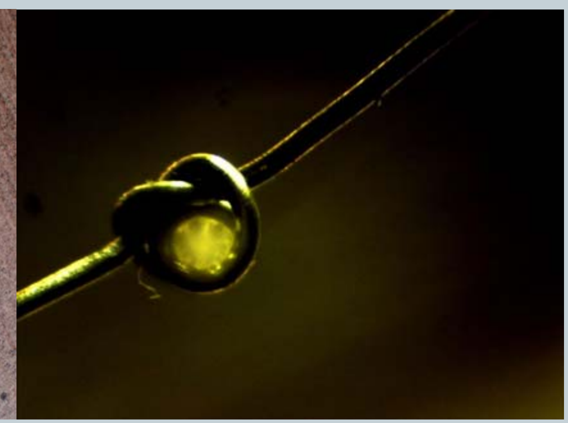
Background: Prior Work

- Two part structure
  - Cladding glass- Porous glass
    - Core glass- solid borosilicate glass
    - Hollow core
  - Pore size range
    - 10-30 nm avg.
    - Range tunable
      - 4 nm to 100 nm
- Sapphire Photonic Crystal
  - Sapphire bundle based optical structure

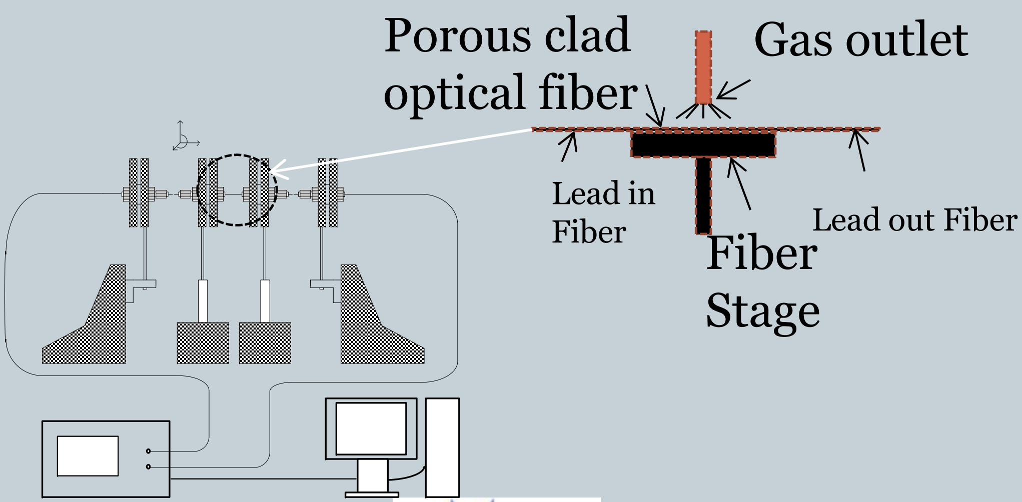


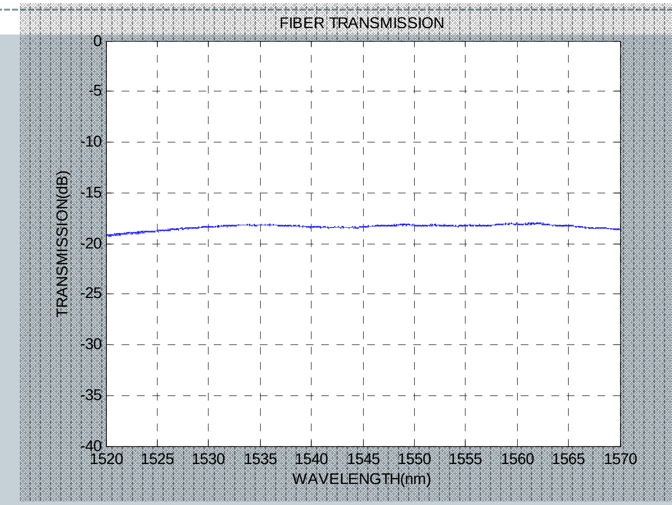


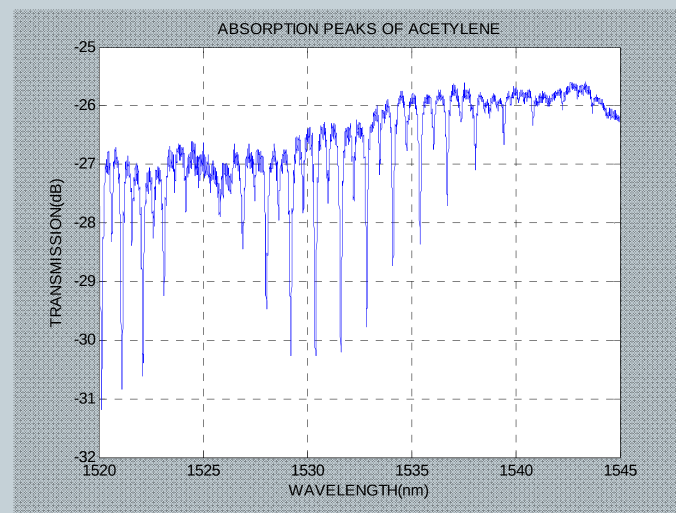


Background: Prior Work

- Sensing
  - ~ 1 second response time
  - Stable up 500° C







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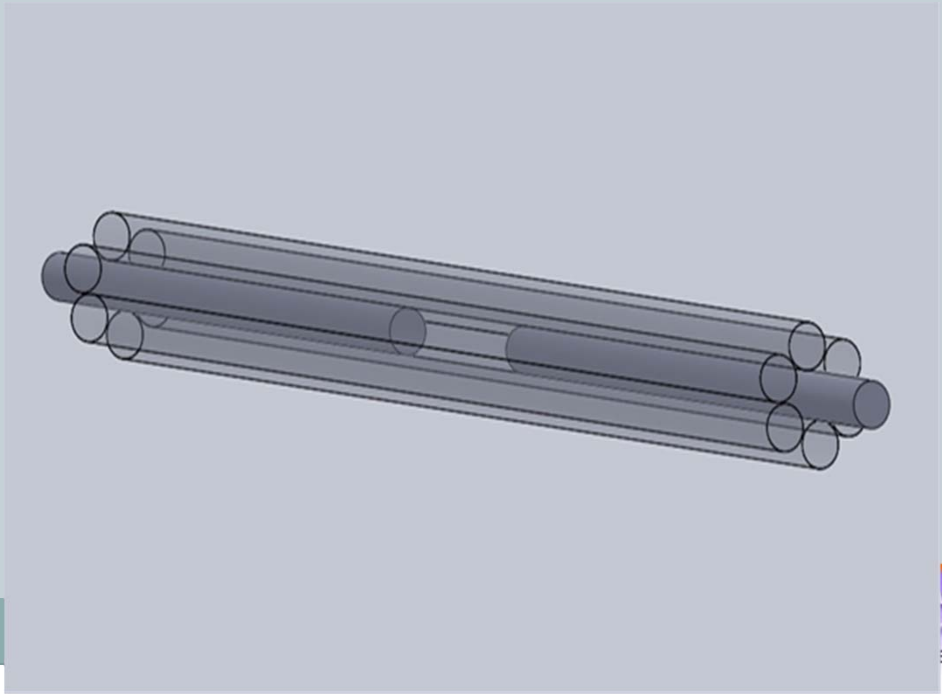
Current Tasks

- Sapphire Photonic Crystal Fiber (SPCF) gas sensor
  - Fabrication
  - Sapphire bonding
  - Testing
- Porous Glass Optical Fiber Gas Sensor
  - Sensor integration in optical system
  - Transmission Loss reduction
  - Gas sensing sensitivity and resolution characterization
  - Field test prototype
- Long wavelength laser system development

Center Gap Sensor

- Center gap between highly polished sapphire fibers enables measurement of gas absorption peaks
- Center fibers and gap held in place using six surrounding fibers in hexagonal formation



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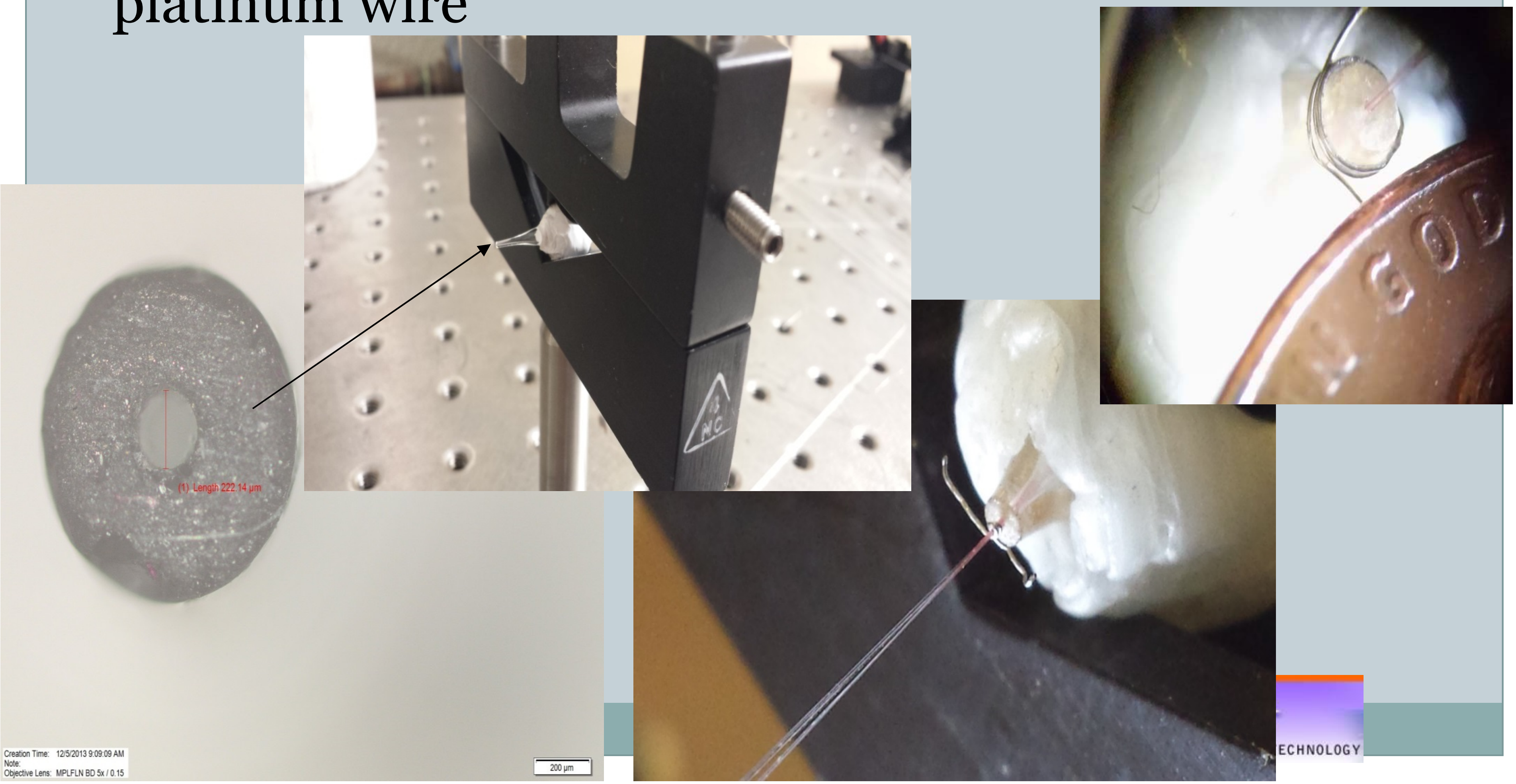
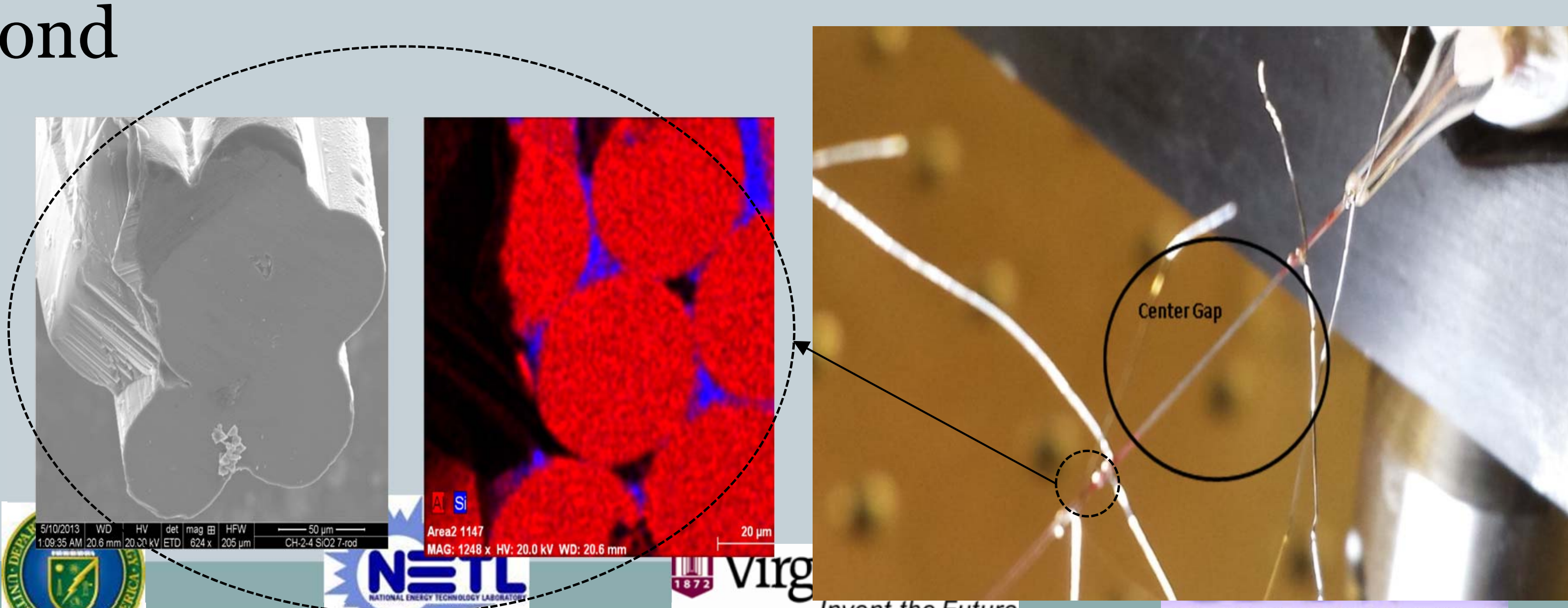
Center Gap Sensor Construction

- Sensor is assembled using tapering funnels and platinum wire

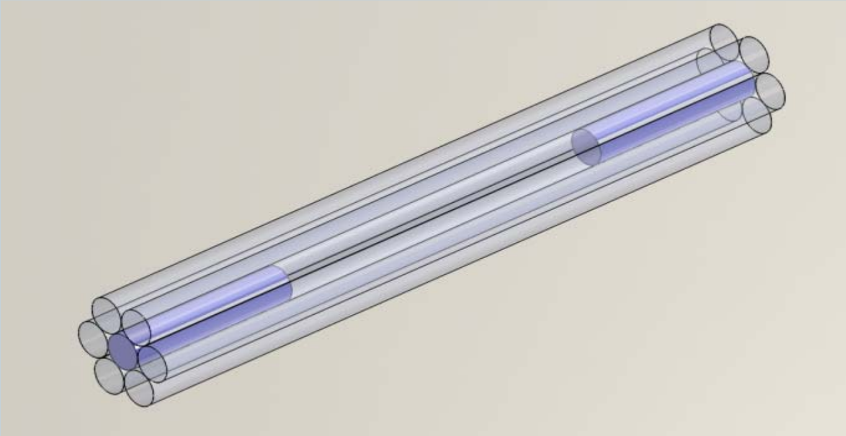


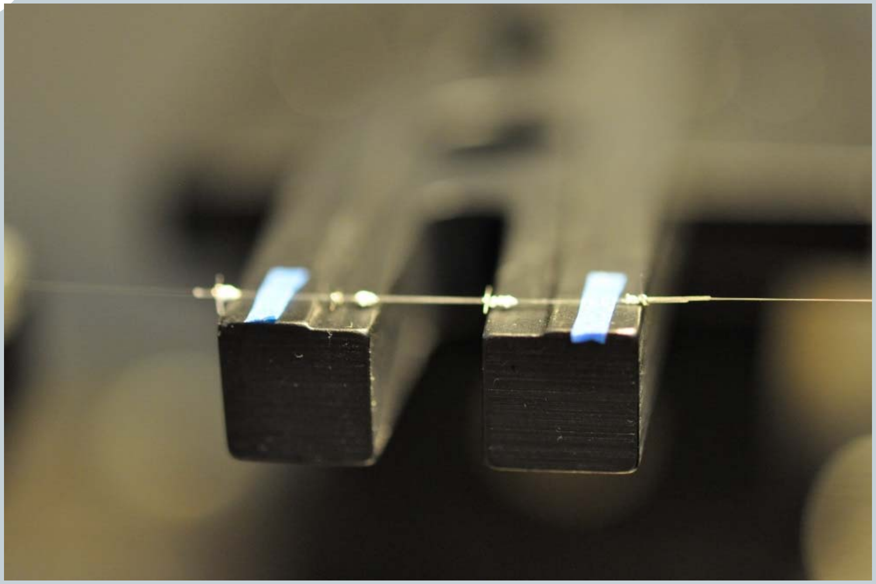
Sensor Bonding- Center Gap Design

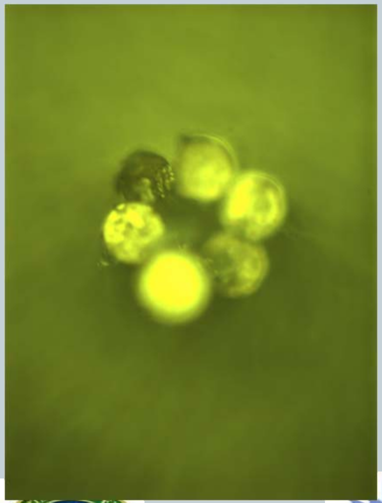
- Bonding colloid is applied to areas outside of the gap area
  - Fired at 1600°C for 12-72 hours to encourage diffusion bonding
- Colloidal particles infiltrate fibers, forming strong bond



Sapphire bundle gas sensor

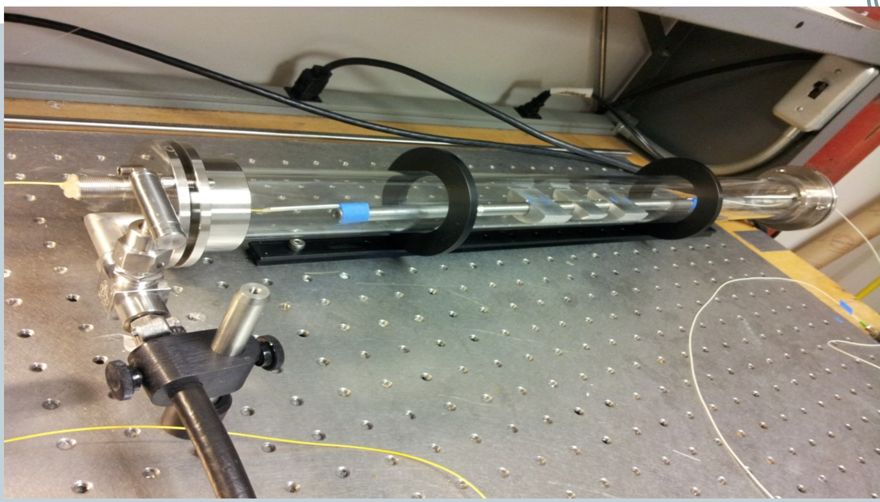


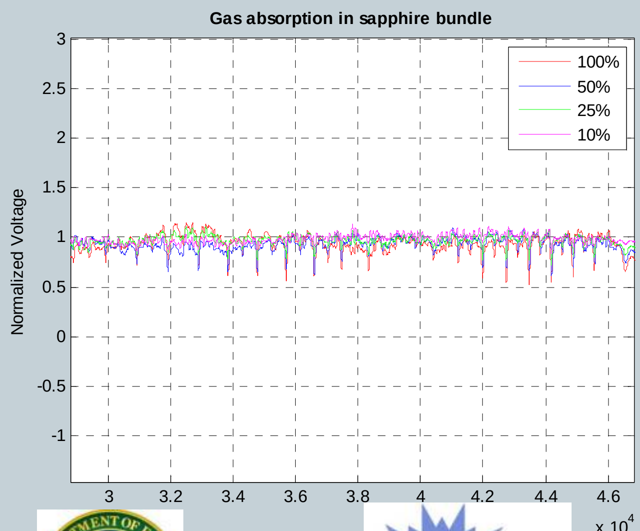


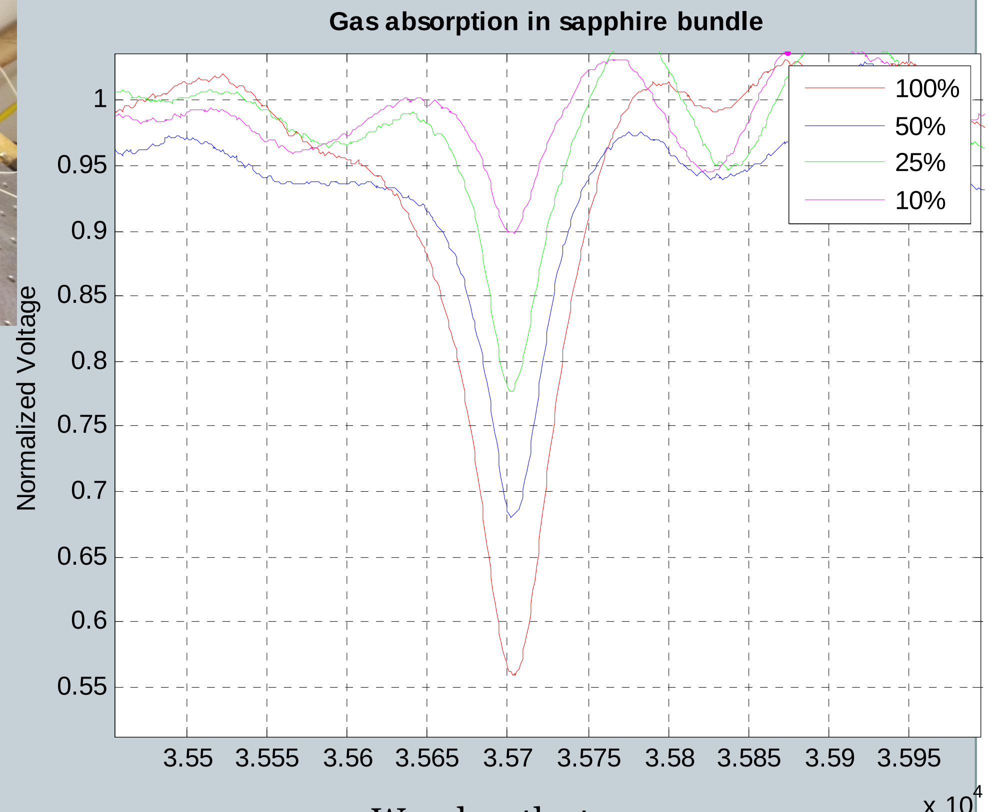


- Diameter of the outer sapphire fibers is 70μm and center sapphire fiber is 50μm
- Air cavity = 10mm

Sapphire bundle with different gas concentration



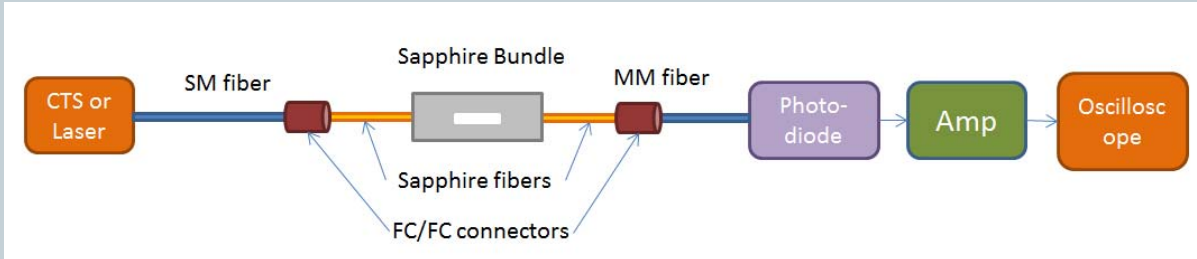


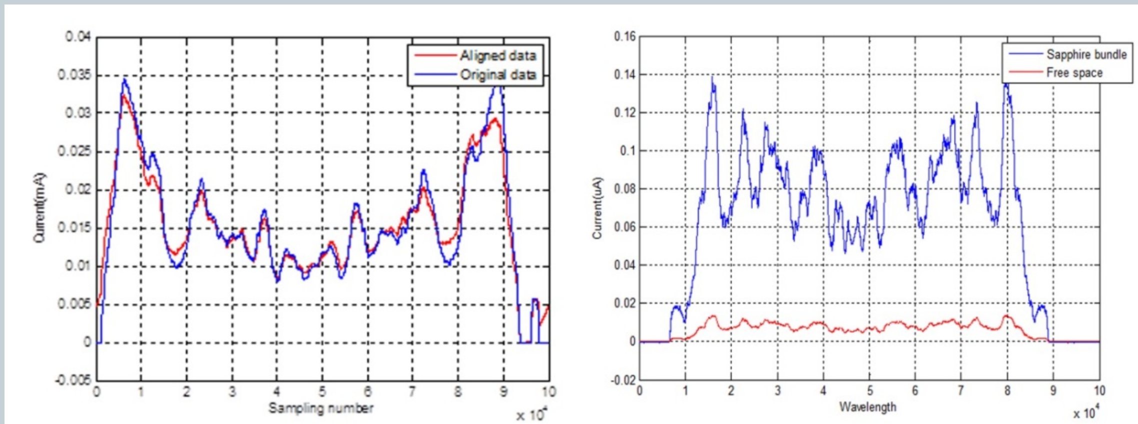


- Wavelength at 1530.4nm

New sapphire bundle with FC connector

- Sapphire bundle sensor with (a) FC connectors and (b) splicing points (Both air cavity=1cm)
- Transmitted power of the sapphire bundle with FC connector has two orders of magnitude stronger than one with splicing point
- Sapphire bundle with FC connector has less modal noise





(a) (b)

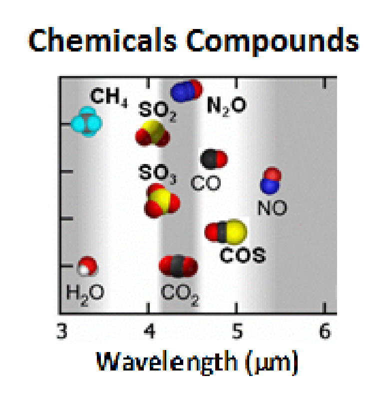
Long Wavelength System

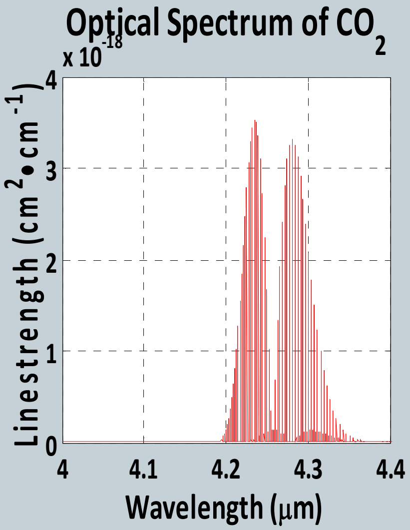
Why the mid-infrared?

The mid-infrared, or mid-IR, is a portion of the electromagnetic spectrum spanning wavelengths between 3-15µm

The Long Wavelength System operates between 4-4.42µm

- This range falls inside a transparent atmospheric window where light transmits well
- This is also where chemical compounds exhibit fundamental vibrational modes
- Optical spectrum measurements are used to observe the effects of these modes to both uniquely *identify & quantify* chemical compounds, *e.g.* CO_2, CO, N_2O, SO_3





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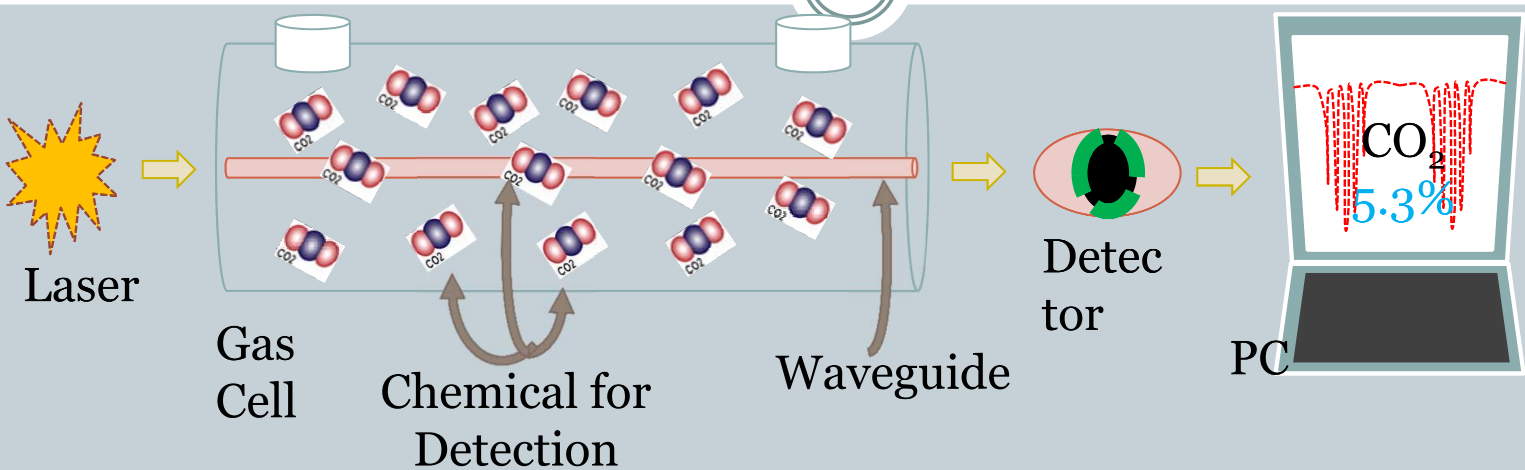
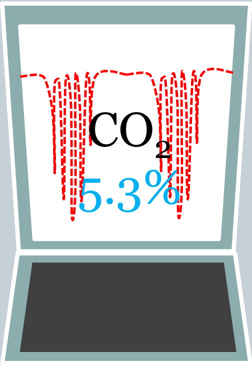
Direct absorption detection technique



- Main advantage is simplicity
- Light passes through a gas cell that contains the chemical for detection
- Light interacts with the chemical as it traverses a porous silica or sapphire waveguide
- Light intensity values are recorded for different wavelengths
- These values form an absorption spectrum from which the gas is both identifiable chemically and quantifiable in terms of concentration

Signal Processing

Identifying and quantifying gases



The detection technique provides an absorption spectrum that appears as multiple dips across a slowly varying background

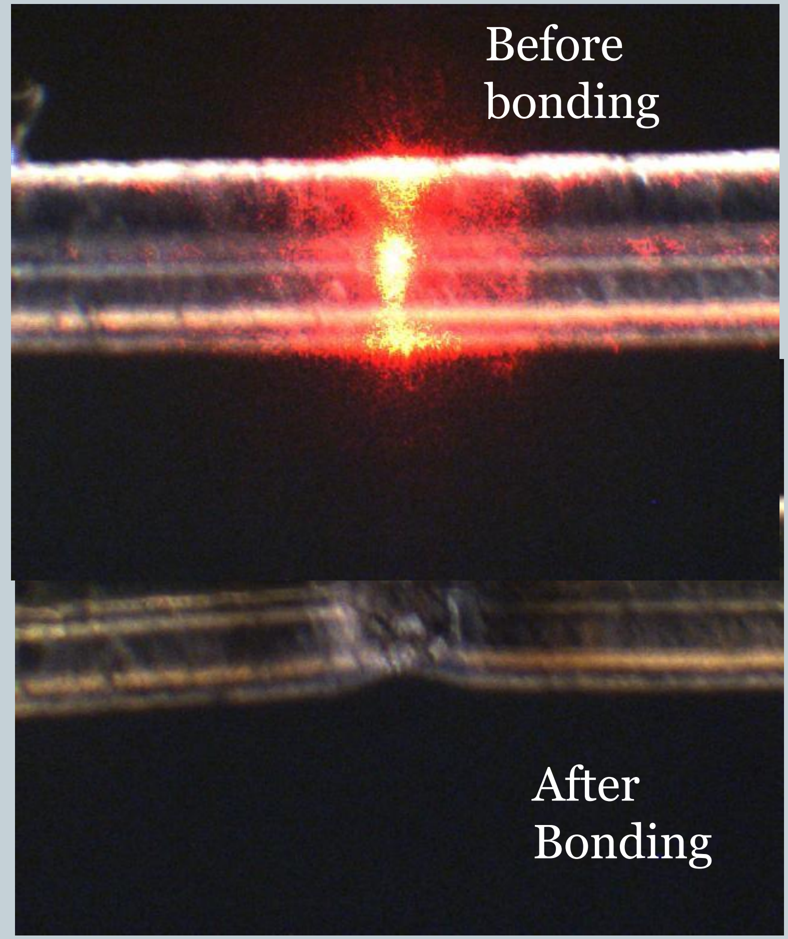
To understand the significance of these dips requires some signal processing that assigns numbers to their magnitudes & wavelength positions

Mathematical techniques such as correlation are used to relate these numbers to the identity of a specific gas

Measurements of unknown concentrations can be determined by comparison versus data of known concentrations

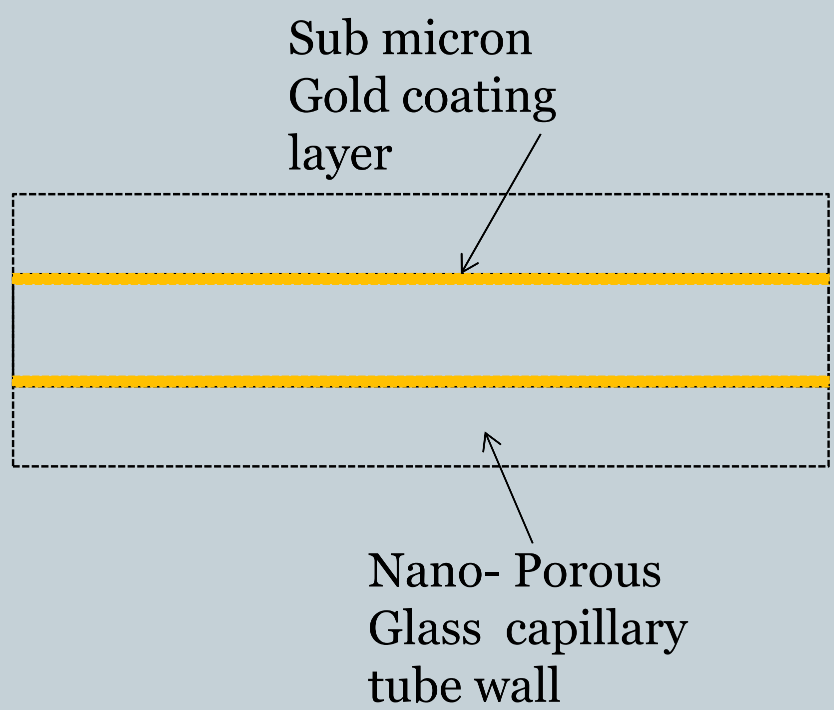
Porous Glass Sensor Joining Techniques

- Trial methods
  - Fusion Splicing
  - CO_2 laser bonding
  - Ceramic adhesive
- Trial evaluation
  - Transmission
  - Bond stability
- Best method
  - CO_2 laser bonding



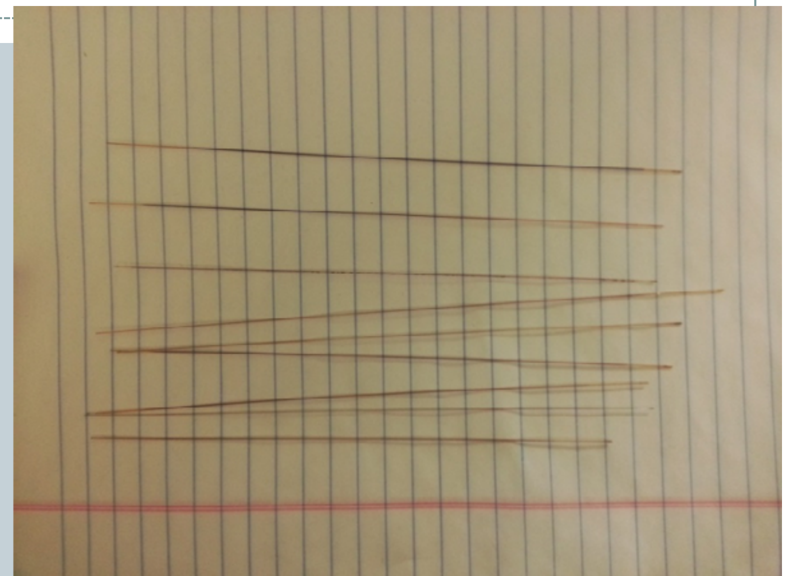
Porous Glass Sensor

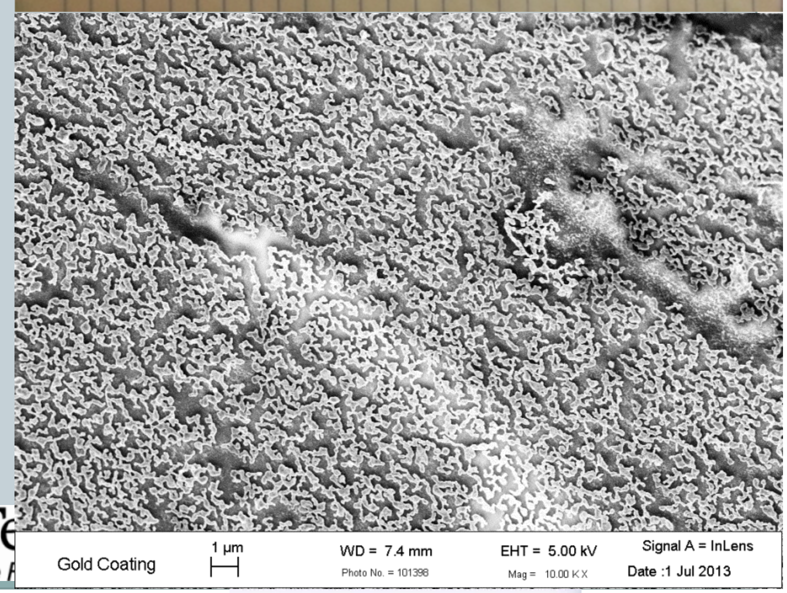
- Transmission improvement
  - Gold coating sensor interior
  - Increased boundary reflection
  - Reduced loss



Signal Improvement Methods

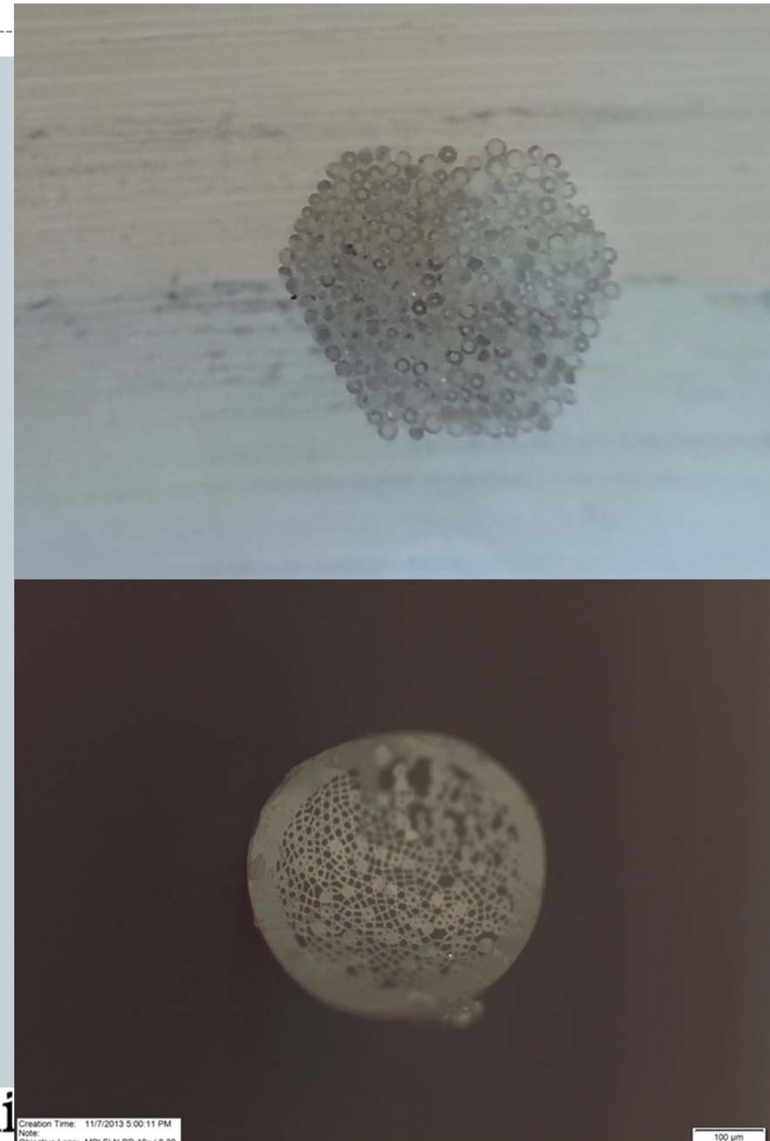
- Gold plating of inner surface of glass prior to leeching.
- Porous gold coating obtained on surface.
- High variation, so many samples needed to obtain best results.
- Plated using commercially available electroless solutions





Alternative Structures

- Ordered tube arrays using vycor to obtain Photonic Crystal Fiber Structure.
- Tubes stacked from pulled fibers using machined mold
- Pulled using mini draw tower.
- Very sensitive to heat profile of furnace.



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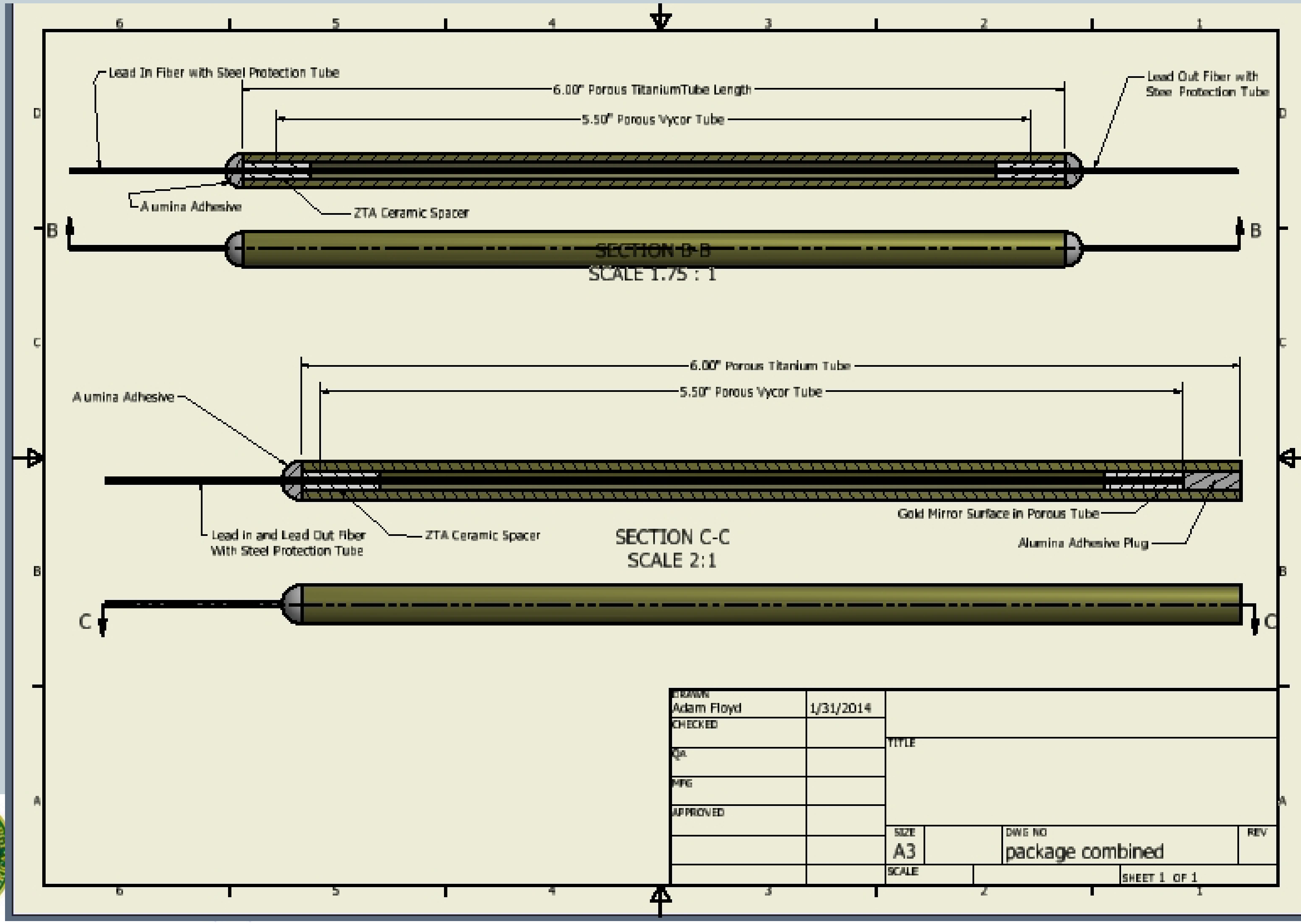
Potential Packaging Design

- Two potential designs
- Provide protection to the fragile nature of the glass.



Future work

- Testing of SPCF gas
  - Optical properties
  - Gas sensing capability
- Continuing evaluation of LW system
  - Long wavelength system gas sensing demonstration
- Sensitivity measurement of SPCF and porous glass sensors
- Construction of porous glass gas sensor prototype
- Porous glass photonic crystal fiber prototype

Thank you

- Questions?