Optical Fiber Sensor Development for Harsh Fossil Energy Applications

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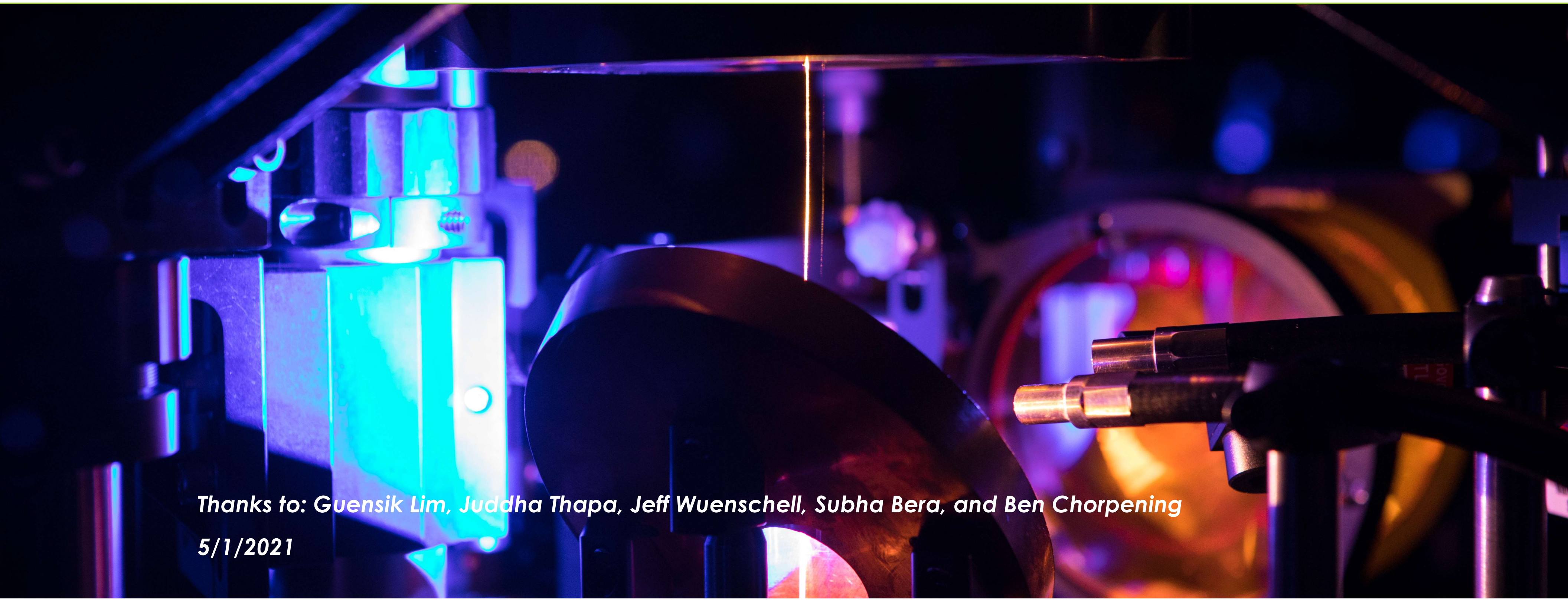
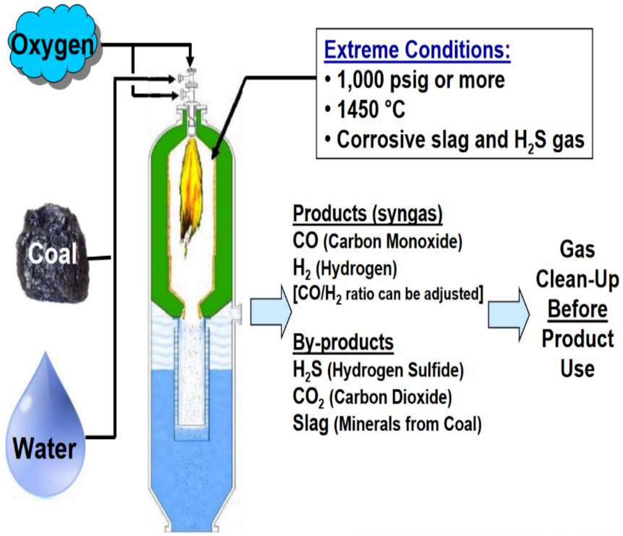
Research and Innovation Center



Thanks to: Guensik Lim, Juddha Thapa, Jeff Wuenschell, Subha Bera, and Ben Chorpening

5/1/2021

Why single crystal fibers in FE sensing?



	Coal Gasifiers	Combustion Turbines	Solid Oxide Fuel Cells	Advanced Boiler Systems
Temperatures	Up to 1600°C	Up to 1300°C	Up to 900°C	Up to 1000°C
Pressures	Up to 1000psi	Pressure Ratios 30:1	Atmospheric	Atmospheric
Atmosphere	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	CO, CO_2, NO_X, SO_X

❖ **Why optical fiber?**
1. No electrical interference
2. Medium temperature (~800c)
3. Single Feedthrough
4. Inexpensive
5. Easily functionalized
6. Distributed!

❖ **Single crystal fiber**
1. High melting point (sapphire: 2054°C)
2. Corrosion resistant
3. Compact size (100 microns)
4. Wide transmission window
5. Benefits of silica ++

Research Breakdown

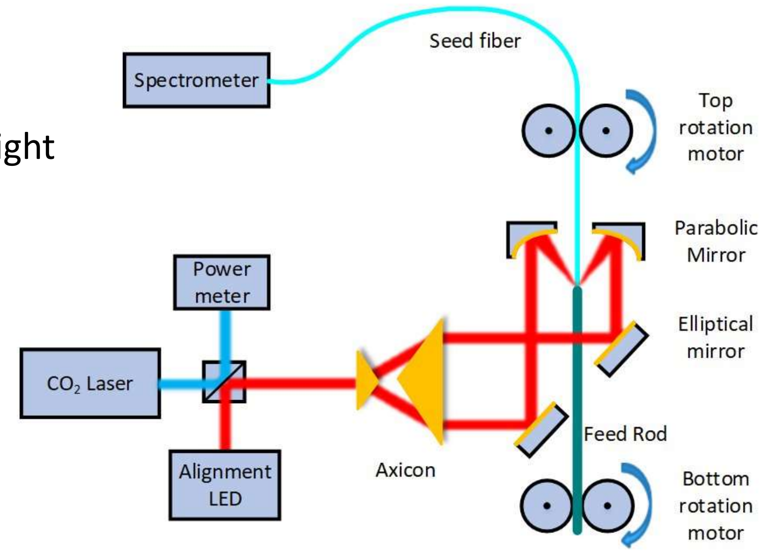
- Increase data-visibility for energy-system operators through high-value distributed measurements (replacing single-point)
  - "Toughest environments provide the highest value"
  - Enable predictive capabilities through data-analytics and AI/ML
- Methods: Produce novel single-crystal fibers for harsh-environment sensor applications
- Design Novel fiber-optic interrogators that work with SC-fiber
- Add – novel parameters like gas composition, flow, radiation, or others
- Market – complete sensor solutions for specific applications/customers
- Control processes for efficiency ($$, fuel, CO_2), Predict failures for maintenance

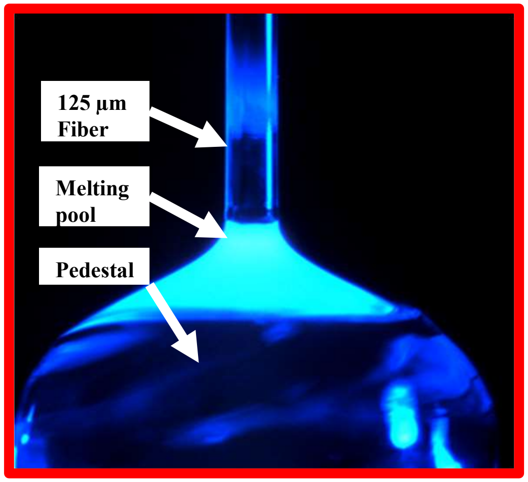
Research Breakdown

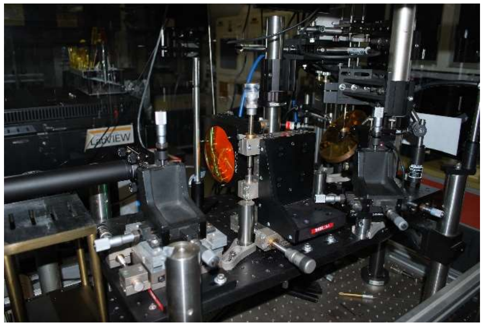
- Advanced Sensors Supporting Tasks:
  - 21: Optical fiber Oxygen Sensor material development
  - 24: Single-crystal fiber substrate and Cladding development
  - 33: Raman distributed optical fiber temperature sensing interrogator development
- Work for others support:
  - ARPA-e “Molten salt loop development acceleration using single crystal distributed optical fiber sensors ($2M, 2 years)

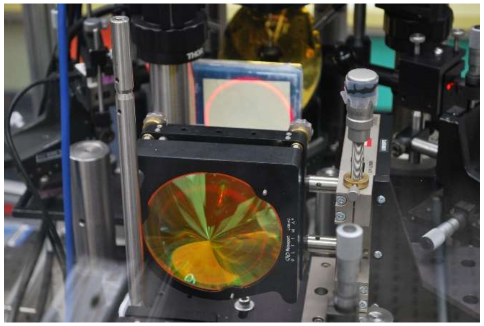
Making Single-crystal fiber with LHPG

- CO_2 laser source for heating
- "Doughnut" beam shaper surrounds molten zone with light
- Motors advance feedstock (pedestal) and fiber
- Slow process (mm/min)
- Grows purse crystals (no cladding)





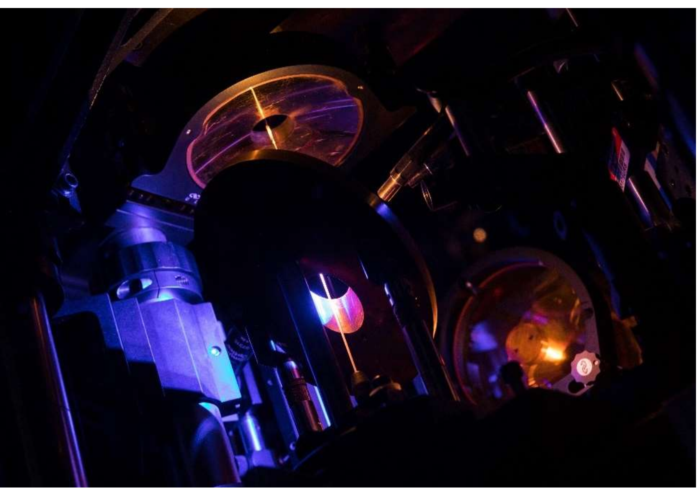


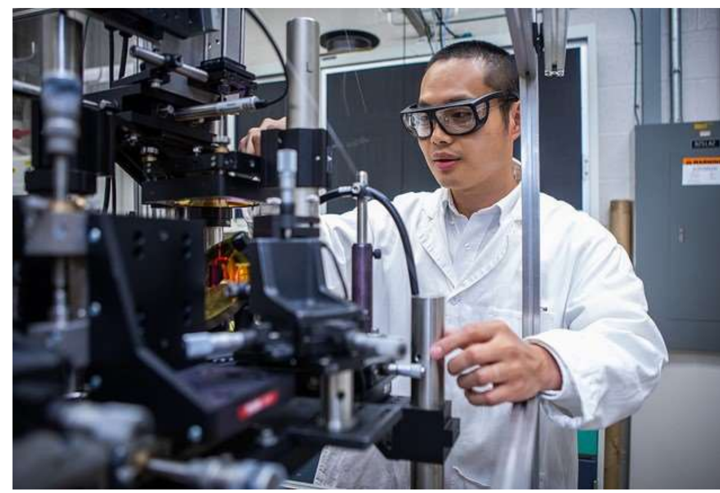


NETL LHPG Capabilities and features

Some NETL LHPG stats:

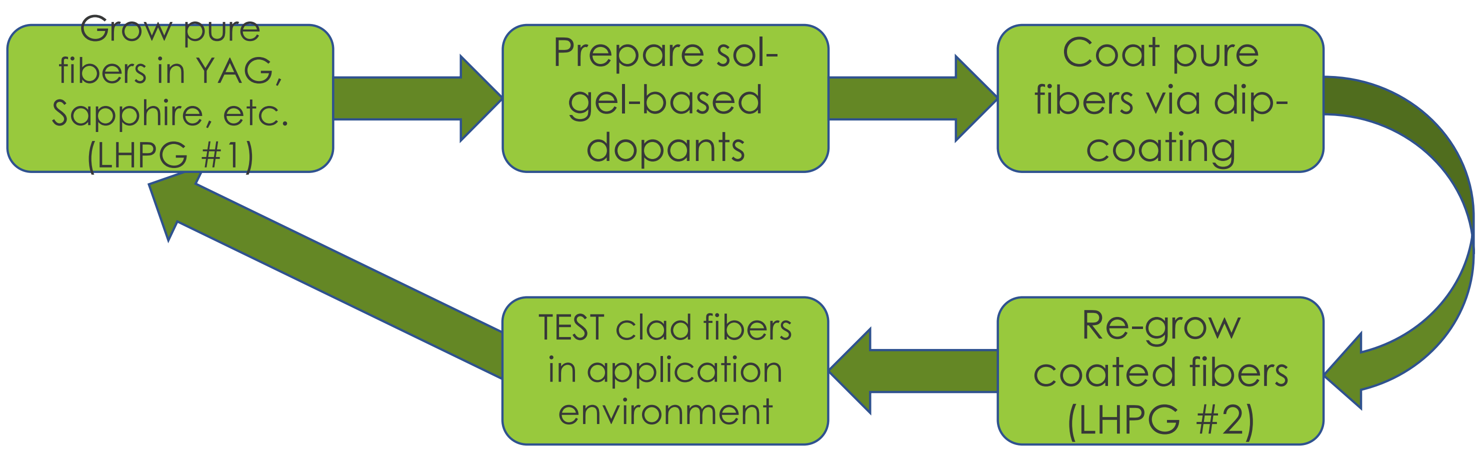
- Minimum diameter variation <2um
- Minimum fiber diameter <55um
- 50W laser power available (<1.5mm pedestals)
- Automatic fiber centering (+/-2mm)
- Continuous growth of any length with start/stop algorithm
- Error Erasing Algorithm





Experimental SC fiber Cladding

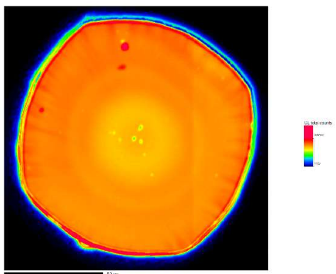
- Grow cladded fibers with 2-stage LHPG
  - Sapphire or YAG
  - Sol-gel (or other) dopant additions
- Evaluate materials compatibility in FE (or nuclear) systems
- Improve fiber performance

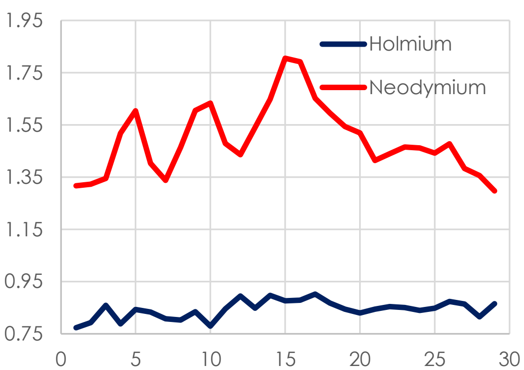


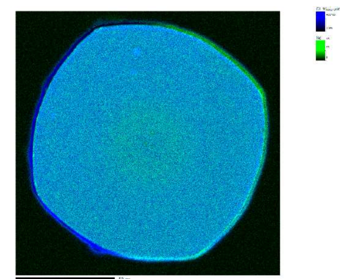
Cladding Application

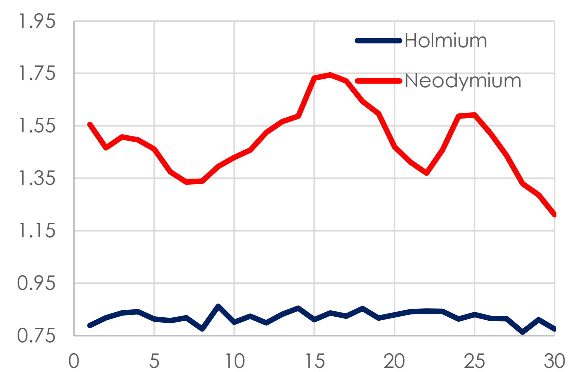
Dopant Species Made to Date:

Dopant species	Host crystal
Cr (chromium)	Sapphire
Nd (neodymium)	YAG
Ho (holmium)	YAG
Er (erbium)	YAG
Yb (ytterbium)	YAG
Ce (cerium)	YAG/ LuAG
Gd (gadolinium)	YAG/ LuAG





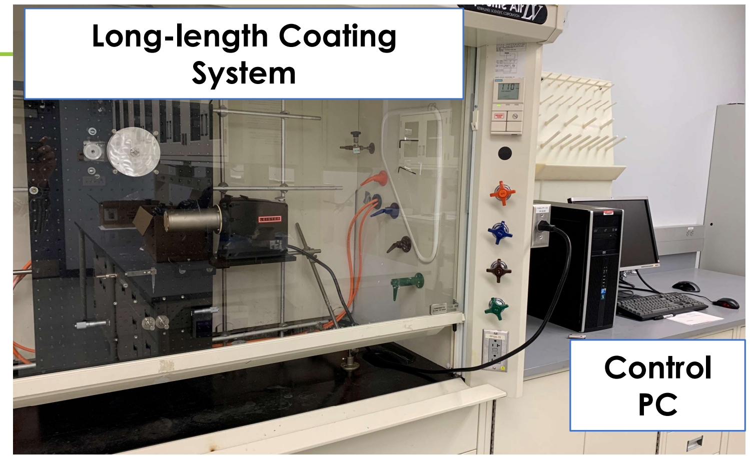


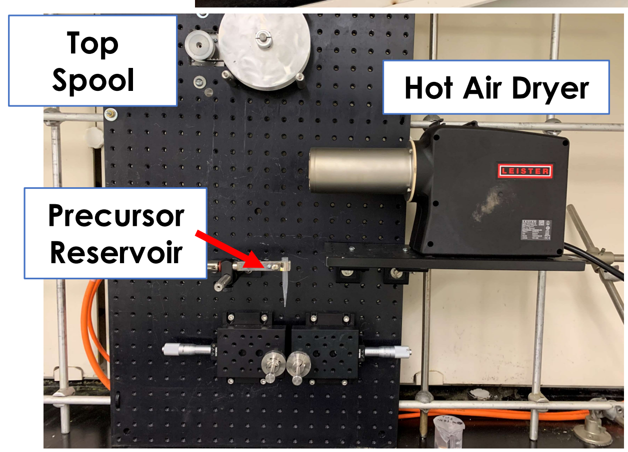


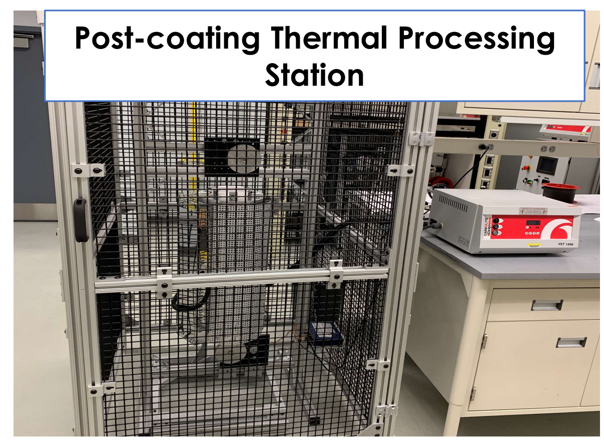
Automatic Dopant Segregation through LHPG: Top left: Visible light guiding in GRIN YAG fiber, Top right: EMPA map of Nd concentration in a GRIN YAG fiber, Bottom plots: Co-doped Nd and Ho: YAG fiber dopant concentrations in X (left) and Y (right)

Major Accomplishment: Reel-to-Reel sol-gel processing system completed (Task 23)

- Coater designed to coat long lengths of single crystal fiber (~3-5 m) in sol gel solution and "soft bake" with hot air dryer.
- Post-coating thermal processing – vertical furnace with 1200°C max temperature.
- Processed fiber used for re-growth and dopant distribution

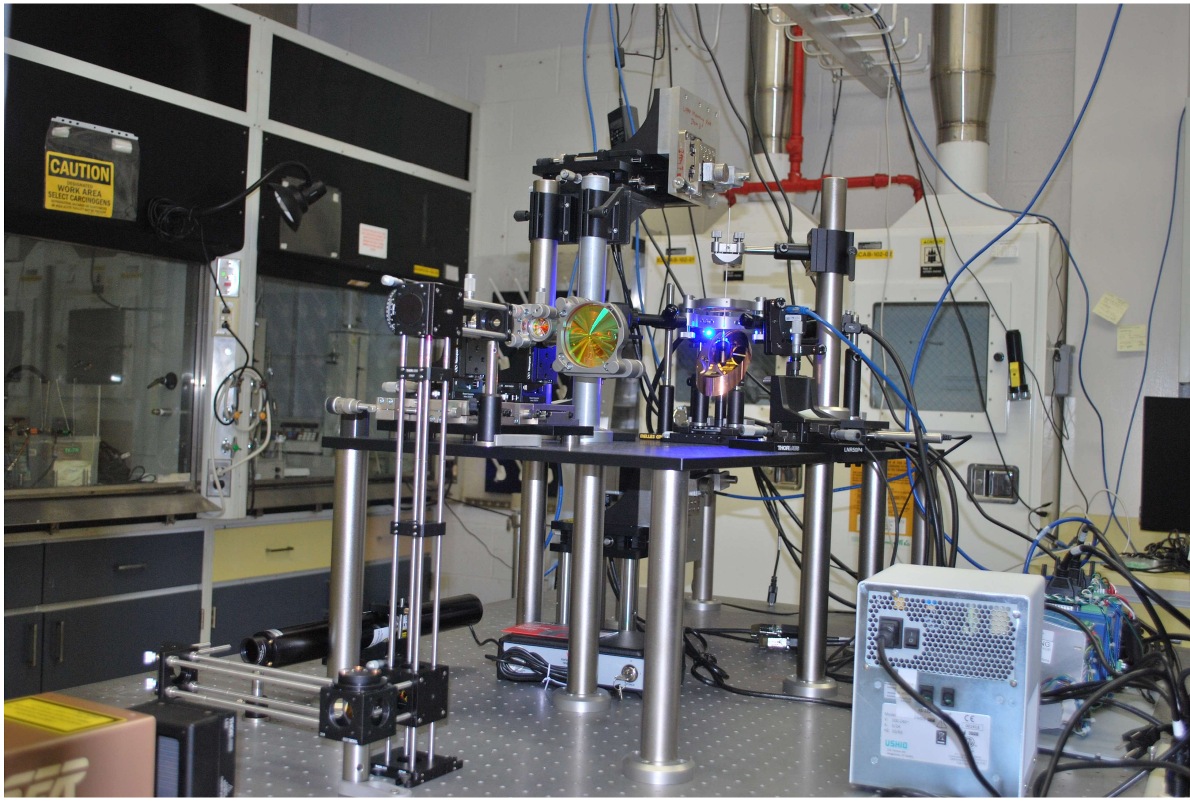






Major Accomplishment: LHPG #2

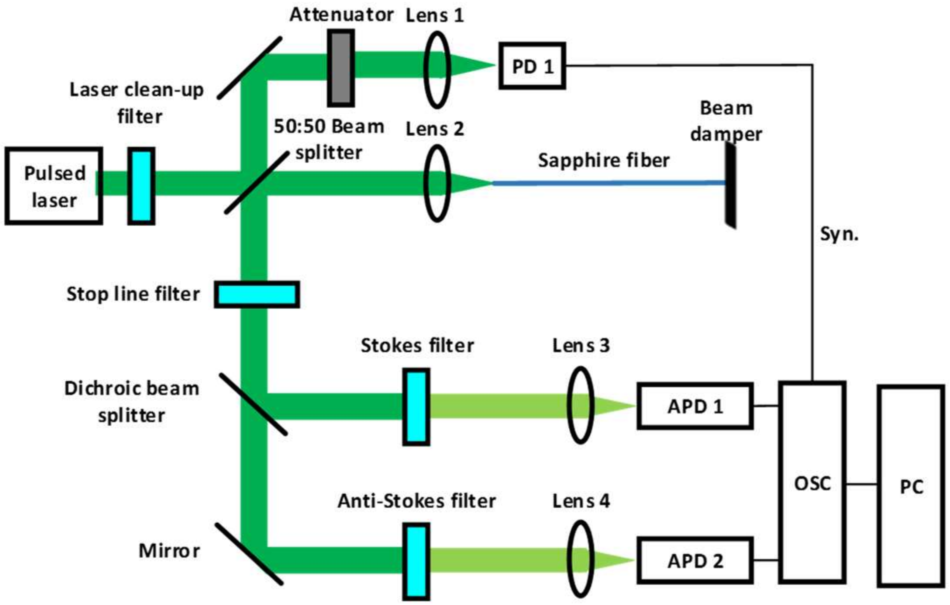
- Constructed in-house
- Mechanical components machined @ NETL/MGN
- >$200k investment (FE/ARPA-e)
- Enables novel 2-stage procedure
  - growth followed by cladding
  - 1mm -> 300um -> 100um (or smaller)
- More than double throughput
- Unique capability/facility



How an SC-fiber becomes a T-sensor (Task 33)

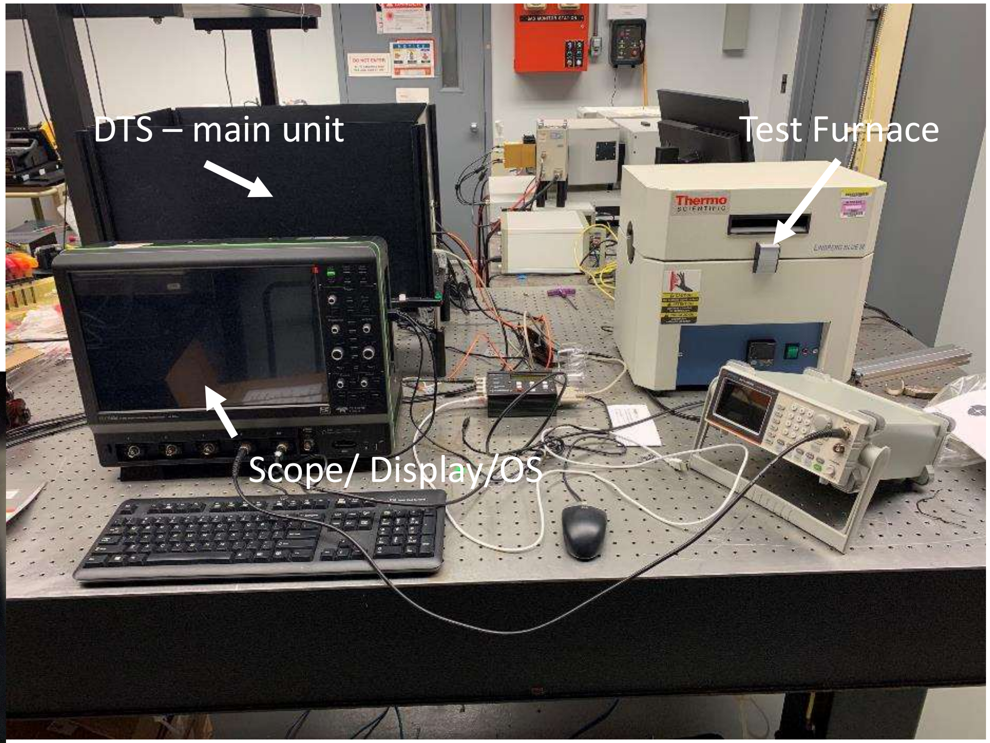
- Introducing the NETL Raman DTS (distributed temperature sensor)
- Pulsed ~350ps 532nm green laser
- Excites Raman Scattering as pulse propagates
- Collects Raman with Fast avalanche photodiodes
- Optics designed for sapphire or YAG fiber
- First interrogator for SC-fiber
- First interrogator produced by NETL Interrogator Development Effort

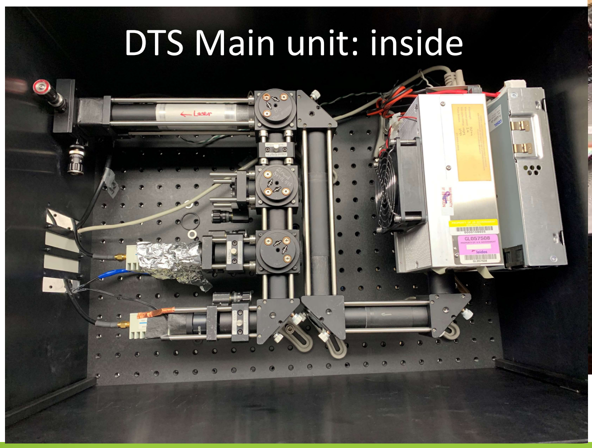
Raman OTDR, Liu et al Opt. Lett., 2016



Raman DTS – Lab Prototype

- Off-the-shelf components
- Breadboard construction
- Enabled design optimization/tinkering
- Improved prototype used for field-testing / product version





Major Accomplishment: DTS Field Prototype design

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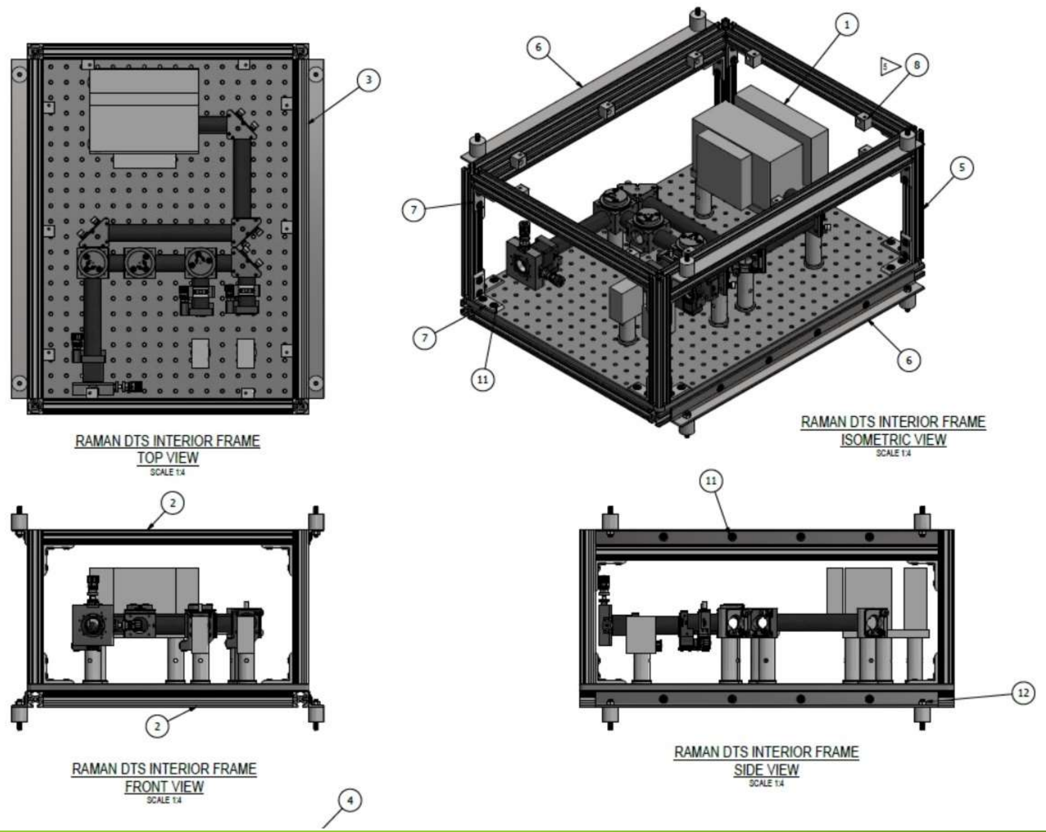
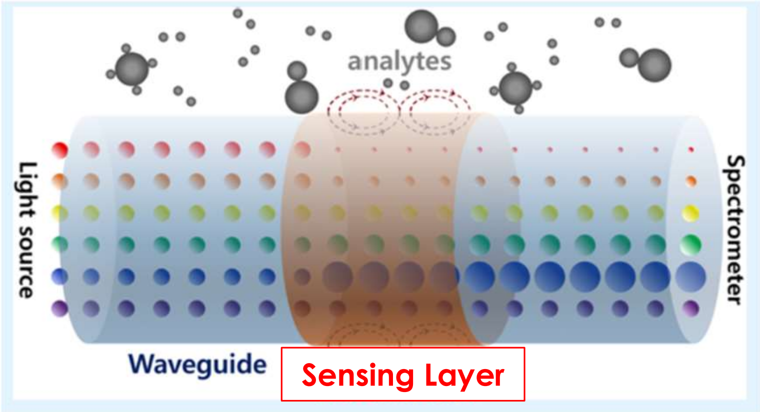
- Flight case design
- Shock-mounted optics
- Laser safety – electrical interlocks
- Software for lead-in fiber
- YAG or Sapphire fibers
- Simplified operator controls
- Field tests in July at INL!
- (even more important) field test in October at MITRR



Adding chemical sensor capabilities (fundamental research): Development of Functional Sensor Materials for Oxygen Sensing (Task 21)

- **Goal** – high temperature distributed or point sensor for oxygen concentration under conditions relevant for SOFC or post-combustion applications.
- Oxygen sensing functional thin films (LSCF) were tested on silica fiber under post-combustion relevant conditions (500-975°C, 1-19% O_2, humid conditions).
- Computational model was developed to better understand dynamic sensing response of model oxide film (LSTO) based on physics and defect chemistry.
- Reactor modification was developed to set up spatially varying oxygen concentration for distributed sensor testing.



Key Deliverables EY20

- Wuenschell, et al. "Combined plasmonic Au-nanoparticle and conducting metal oxide high-temperature optical sensing with LSTO." Nanoscale 12.27 (2020): 14524-14537.
- Wuenschell, et al. "The role of oxide defect chemistry in the Drude and plasmonic response of optical fiber-based sensing layers for high-temperature gas sensing." Oxide-based Materials and Devices XII. Vol. 11687. International Society for Optics and Photonics, 2021.
- Li, Jiayu, et al. "Fiber Coupled Near-Field Thermoplasmonic Emission from Gold Nanorods at 1100 K." Small (2021): 20072

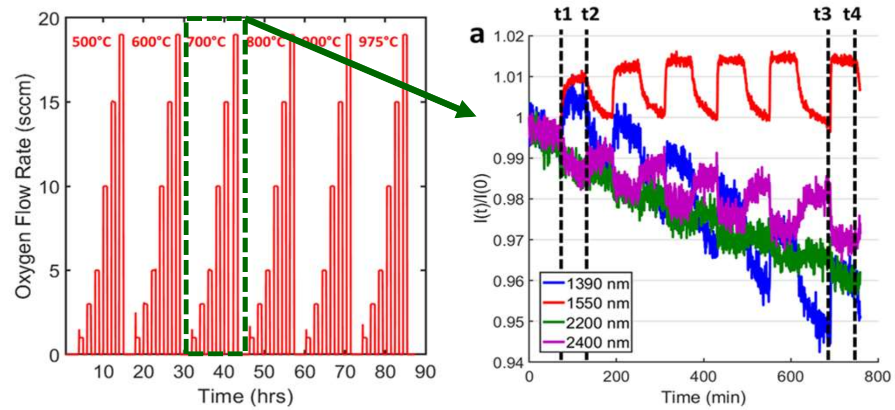
Adding chemical sensor capabilities: LSCF as an O_2 sensor

Tests

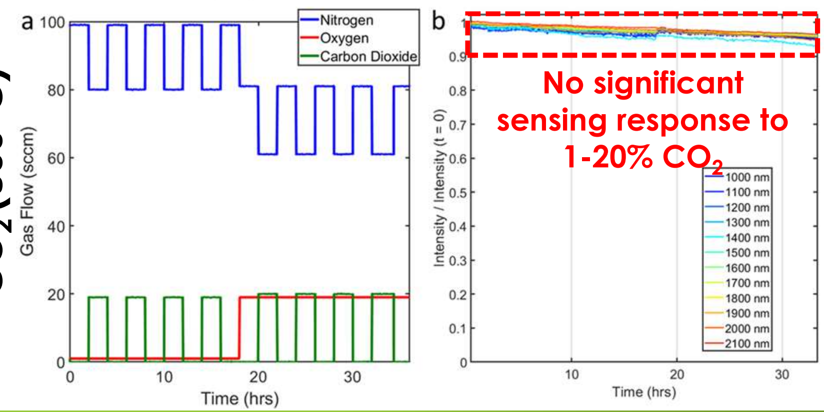
- Oxygen sensing layer tested under conditions for operation in post-combustion environment
- High temperature (500-975°C)
- 1-20% O_2 input gas stream passed through bubbler humidifier
- Cross-sensitivity of oxygen sensing response tested with CO_2

Conclusions

- Majority of drift at high temperature corresponds to hydroxyl defect lines of silica (fiber).
- Broadband response of film (LSCF) found to be more stable.
- Silica as limiting factor bodes well for future with single crystal fiber as platform.



Response to CO_2 (800°C)



How to test a distributed gas sensor (spatially varying gas test rig)

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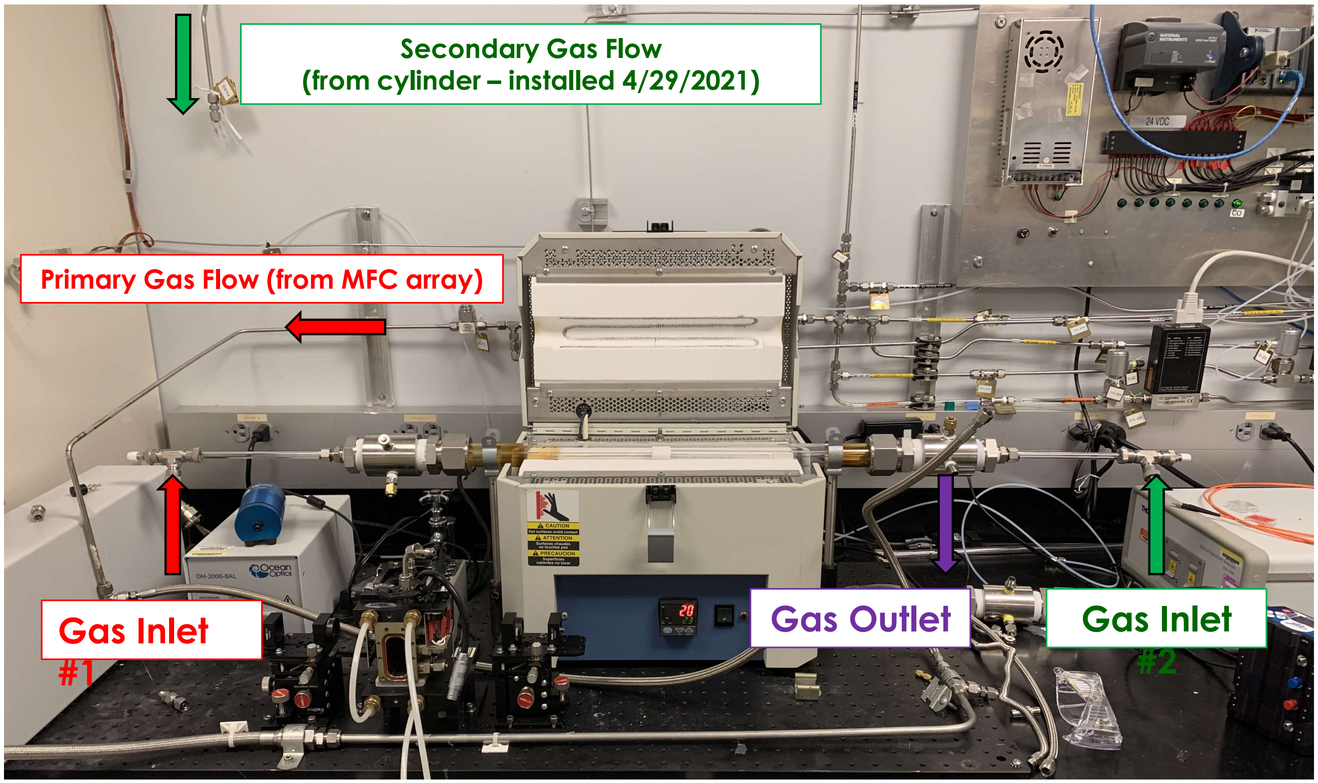

- Reactor system designed / built in EY20 feeds two gas flows into tube furnace reactor (max temp. 1000°C) with partition.
- **Goal** – to establish spatially varying oxygen concentration for testing distributed oxygen sensors.
- Primary gas flow: Controllable 0-20% O_2, N_2 balance.
- Secondary gas flow: fixed, based on cylinder selected (both N_2 and air permitted).
- Final construction / testing to commence upon arrival of MFC (COVID related shipping delay from supplier).

Major Conclusions

- Distributed Fiber-optic sensing will enable amazing new capabilities
- The toughest (and highest value) sensor locations are becoming accessible
- Single-crystal fiber will enable measurements where silica is problematic
- Interrogators can be developed at lower cost, for specific applications
- Functional materials can enable novel parameters like gas composition
- NETL can offer a complete solution with fiber, coatings, and interrogators

Measure where it counts!

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