Fiber Optic Sensors for Harsh Environment Applications (AS Tasks 21, 24, and 33)

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FECM Spring R&D Project Review Meeting

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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

Task/Topic Overview

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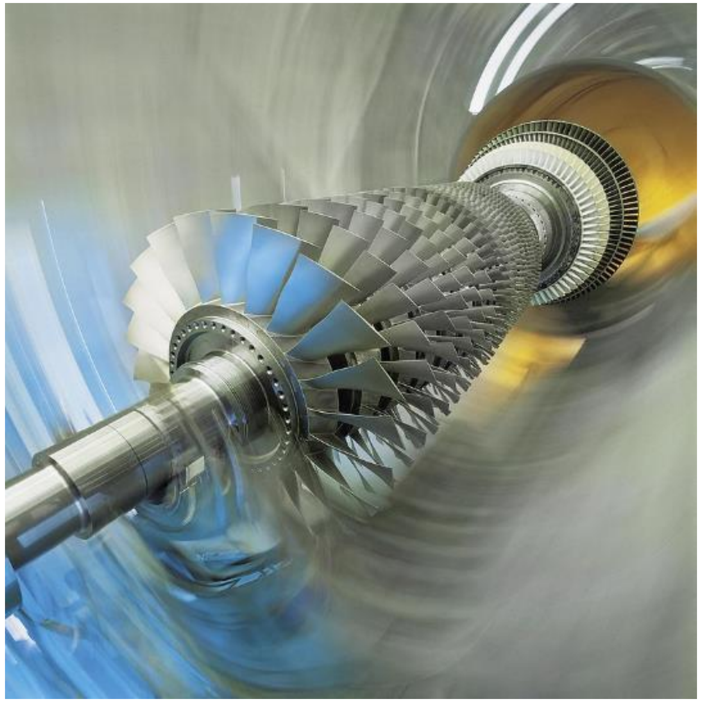
- Task 24: Laser Heated Pedestal Growth/ Fiber Cladding
- Task 33: Distributed Raman Temperature Sensing with single-crystal optical fiber (ultra-high temperature)
- Overlap with 2019-2022 ARPA-e nuclear sensing project
- Task 21: Novel Optical Oxygen Sensing Materials

Why single crystal fibers in FE sensing?



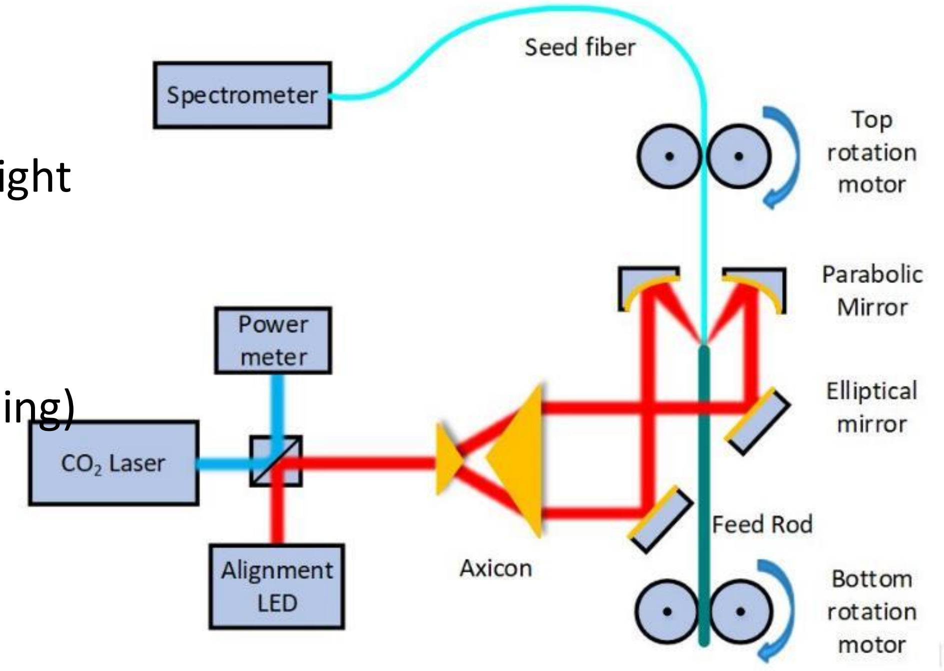
❖ **Why optical fiber?**
1. No electrical interference
2. Medium temperature (~800c)
3. Single Feedthrough
4. Inexpensive
5. Easily functionalized
6. Distributed!

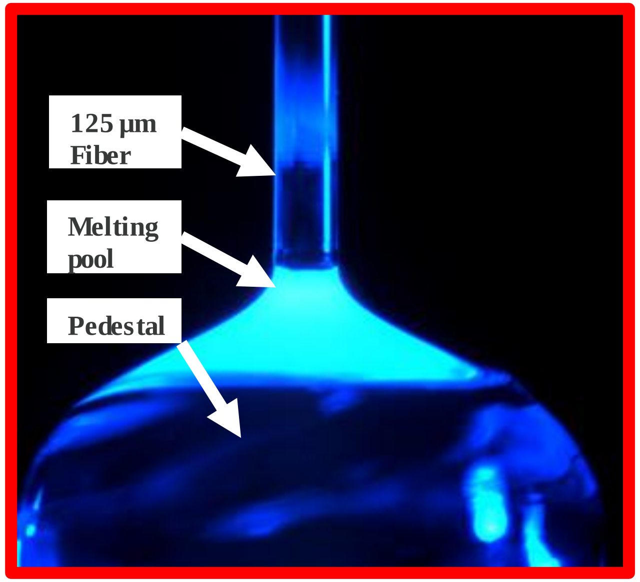
❖ **Single crystal fiber**
1. High melting point (sapphire: 2054°C)
2. Corrosion resistant
3. Compact size (100 microns)
4. Wide transmission window
5. Benefits of silica ++

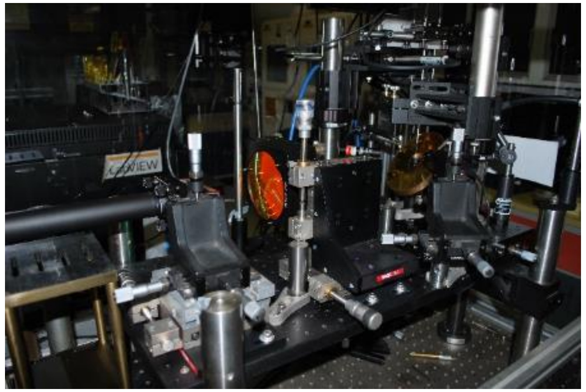
	Coal / Waste plastic biomass Gasifiers	**Combustion Turbines (H2 or NG)**	**Solid Oxide Fuel Cells / Electrolyzers**	**Hybrid systems**
Temperatures	Up to 1600°C	Up to 1300°C	Up to 900°C	Up to 1000°C
Pressures	Up to 1000psi	Pressure Ratios 30:1	Atmospheric	System dependent
Atmosphere	Highly Reducing, Erosive, Corrosive	Oxidizing	Oxidizing and Reducing	Oxidizing and reducing
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	H2, NG components, contaminants

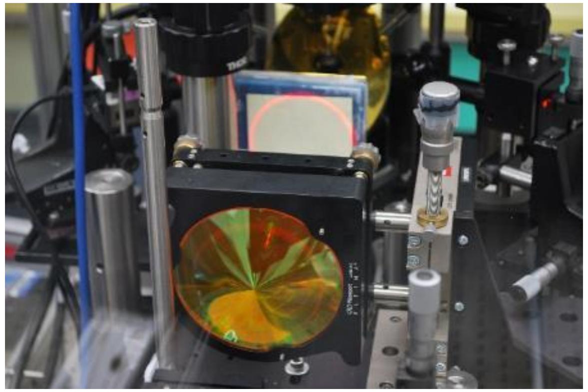
Making Single-crystal fiber with LHPG

- 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 pure crystals (no cladding)





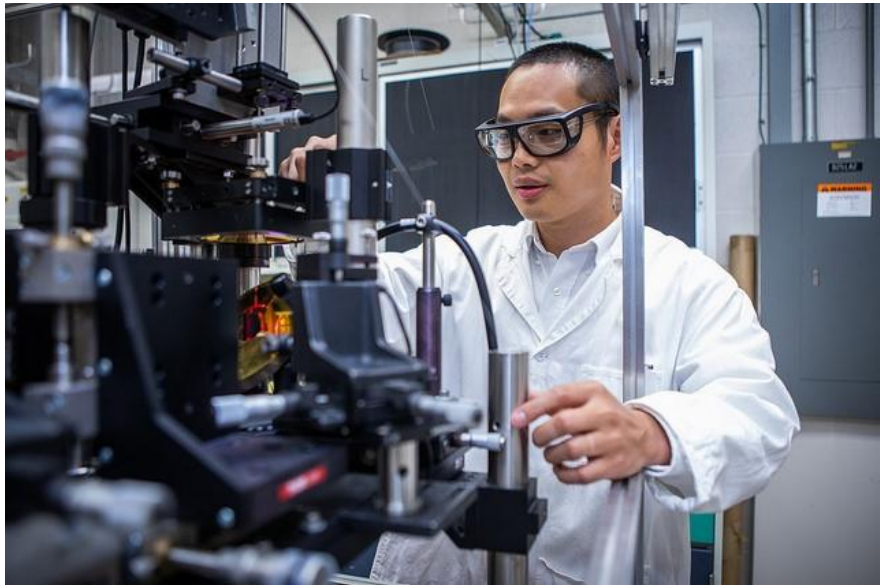




NETL LHPG Capabilities and features

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
- Second LHPG brought online in 2021
- High temperature claddings



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

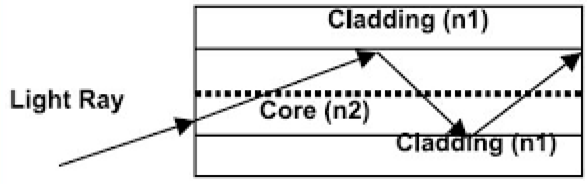
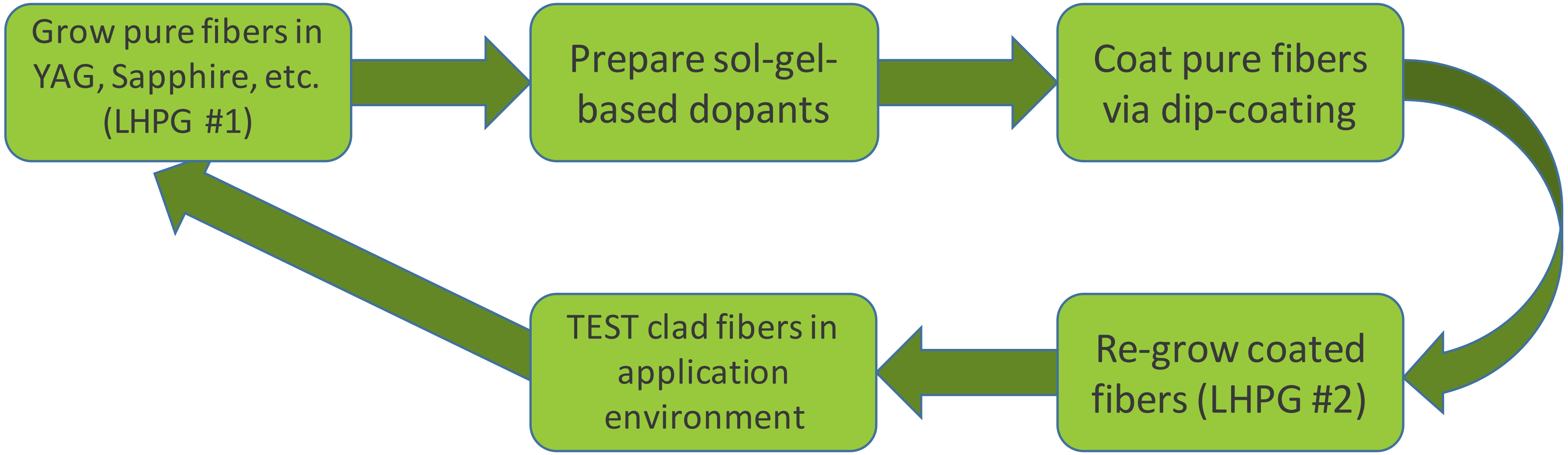


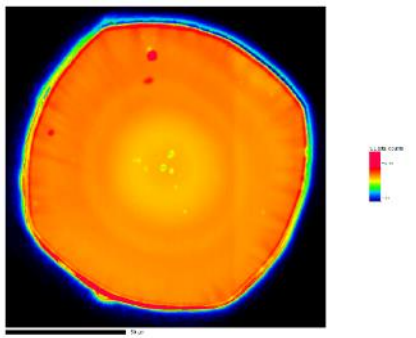
Figure 1: Light launched into the fiber core

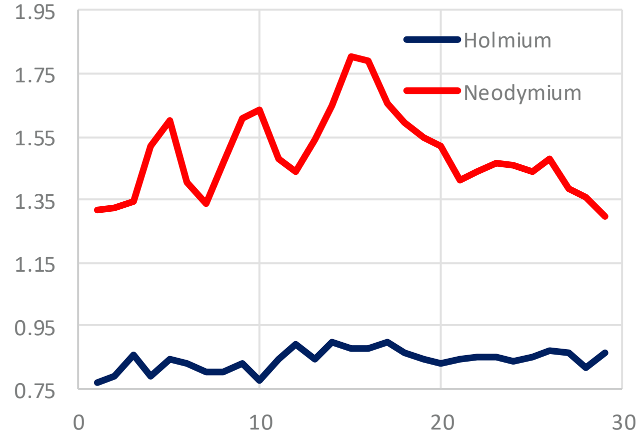


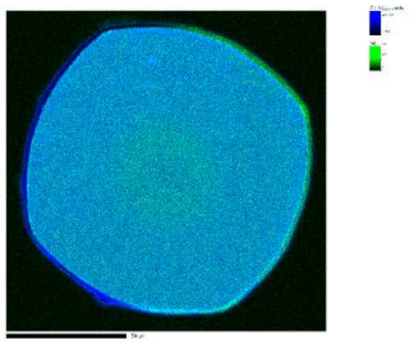
Cladding Application

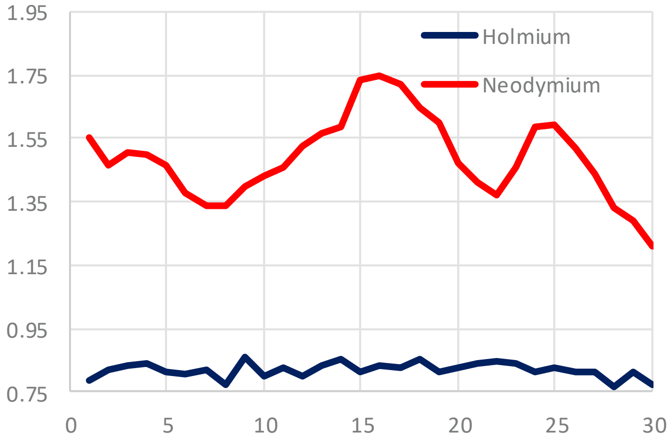
Dopant Species Made to Date:

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





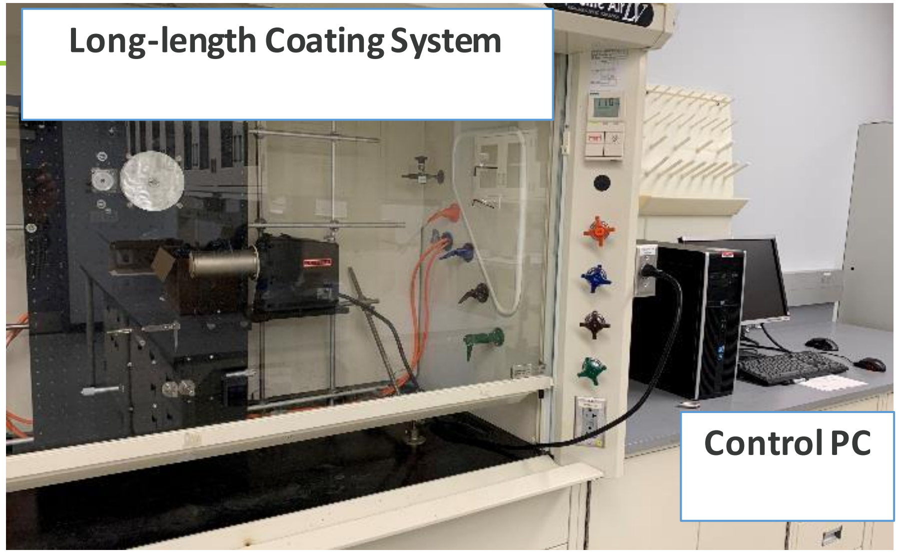


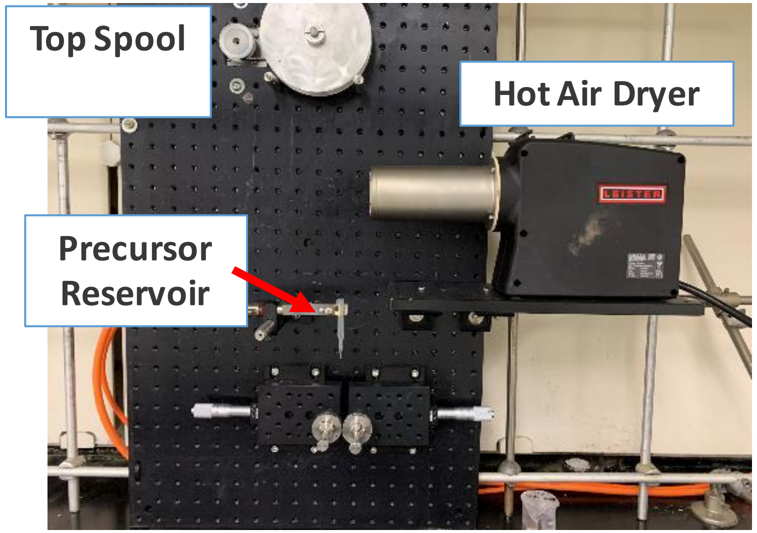


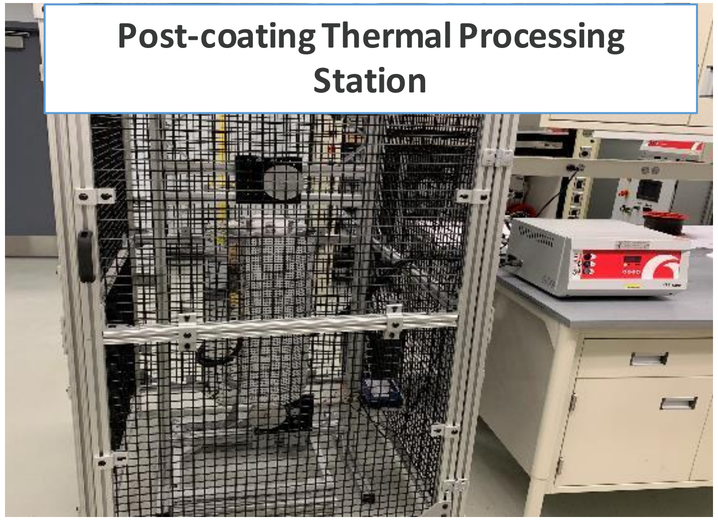
Automatic Dopant Segregation through LHPG: Top left: Visible light guiding in GRIN YAG fiber, Top right: EMPA map of Nd concentration in a GRIN YAG fiber, Bottom plots: Co-doped Nd and Ho: YAG fiber dopant concentrations in X (left) and Y (right)

Reel-to-Reel sol-gel processing system for cladding dopant additions (Task 24)

- 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







Novel dual LHPG system for clad sc-fiber

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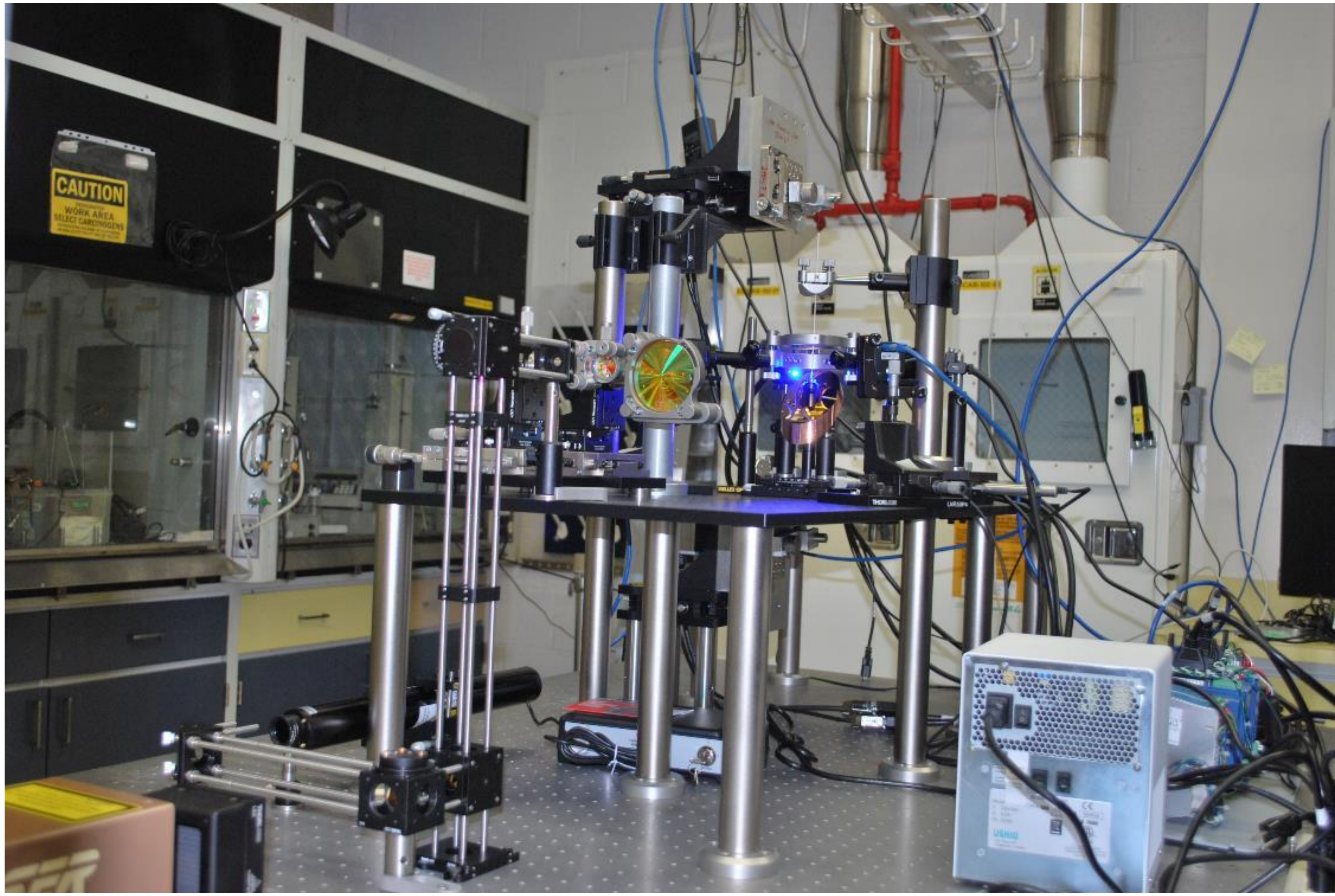
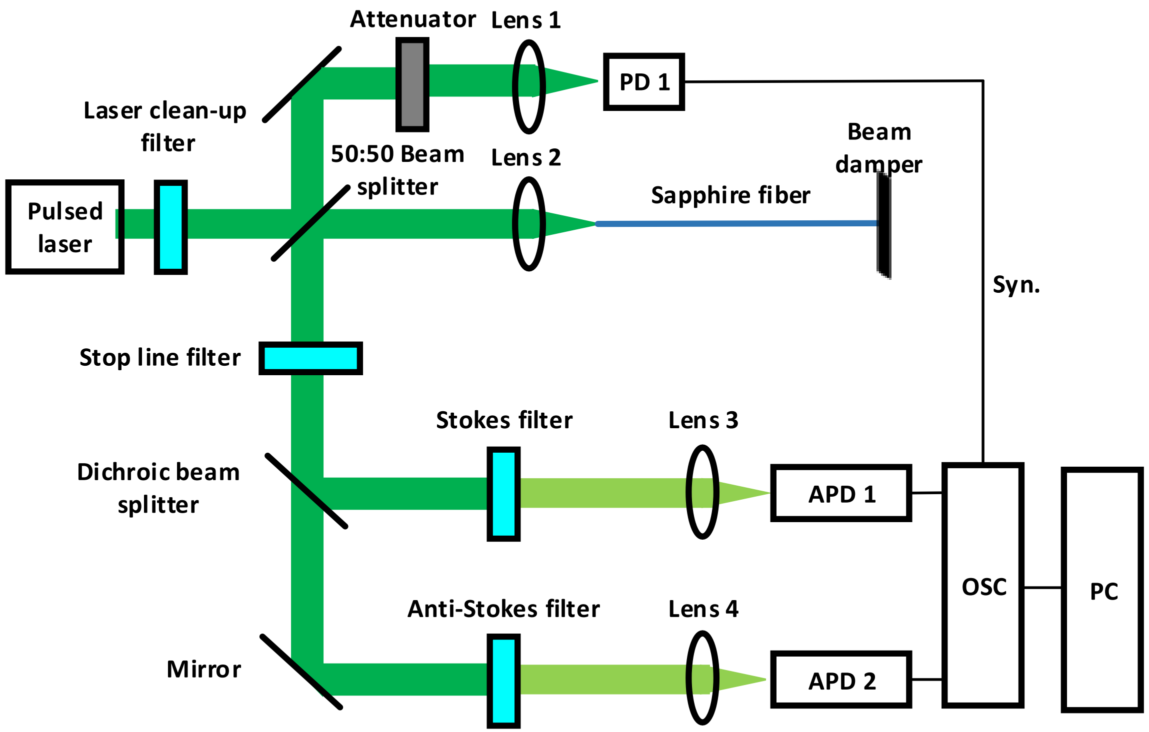
- Constructed in-house
- Mechanical components machined @ NETL/MGN
- >$200k investment (FE/ARPA-e)
- Enables novel 2-stage procedure
  - growth followed by cladding
  - 1mm -> 300um -> 100um (or smaller)
- More than double throughput
- Unique capability/facility



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

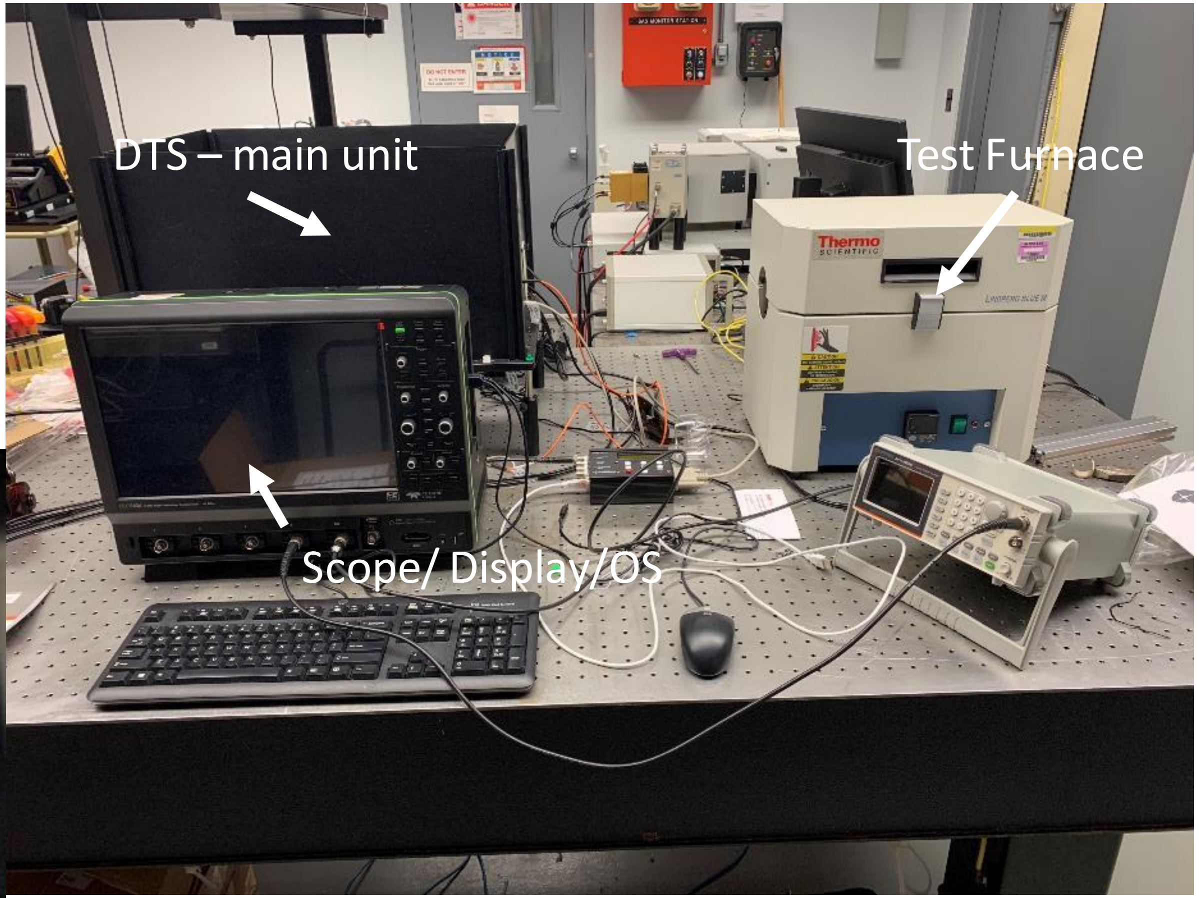
- Introducing the NETL Raman DTS (distributed temperature sensor)
- Pulsed ~350ps 532nm green laser
- Excites Raman Scattering as pulse propagates
- Collects Raman with Fast avalanche photodiodes
- Optics designed for sapphire or YAG fiber
- First interrogator for SC-fiber
- First interrogator produced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

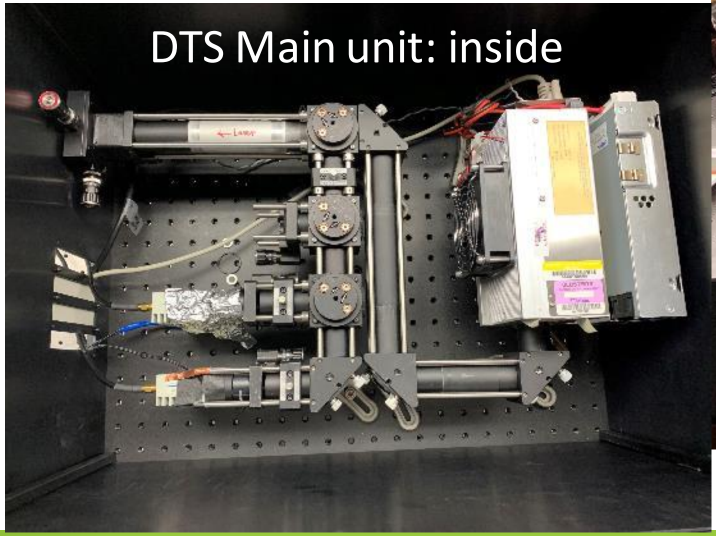
Raman OTDR, Liu et al Opt. Lett., 2016



Raman DTS – Lab Prototype

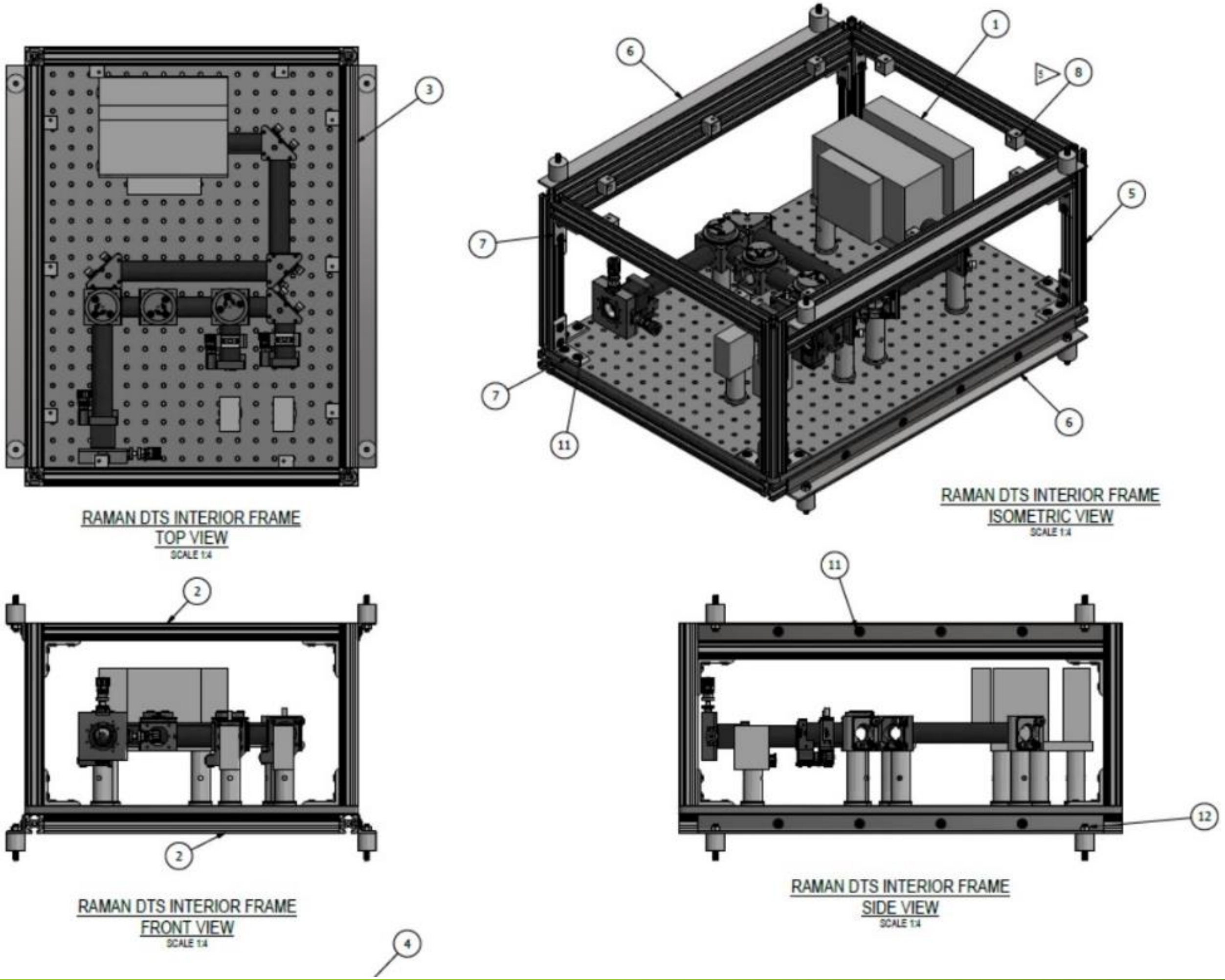
- Off-the-shelf components
- Breadboard construction
- Enabled design optimization/tinkering
- Improved prototype used for field-testing / product version





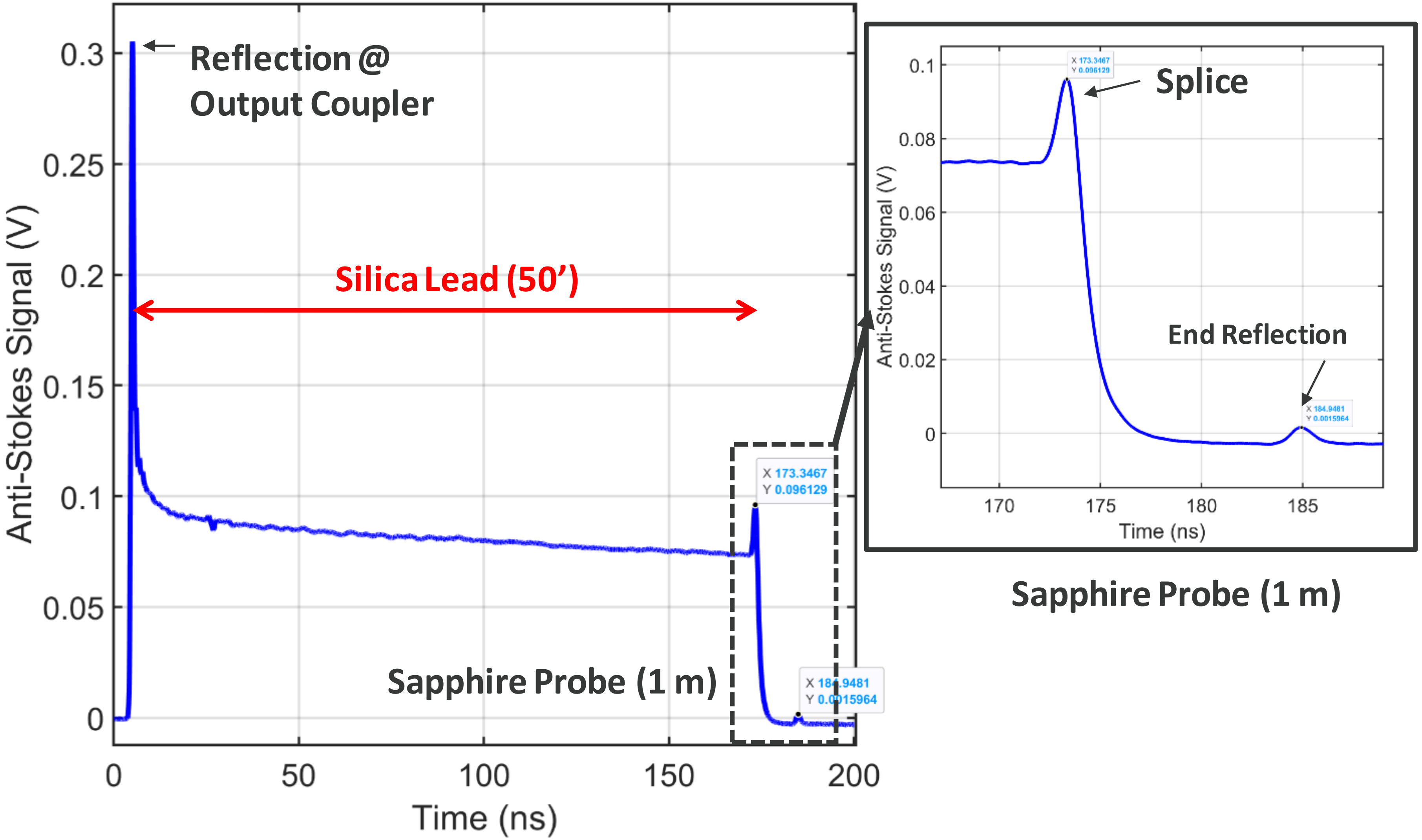
DTS Field Prototype design

- Flight case design
- Shock-mounted optics
- Laser safety – electrical interlocks
- Software for lead-in fiber
- YAG or Sapphire fibers
- Simplified operator controls
- First field test at MITR
- Second Field test at INL



Probe Design for remote measurement

- 50' silica multimode fiber (105 μm), Thorlabs low-OH content silica.
- Fusion spliced to 1 m long single crystal sapphire probe (100 μm diameter).
- Single-crystal probe covers entirety of hot zone.

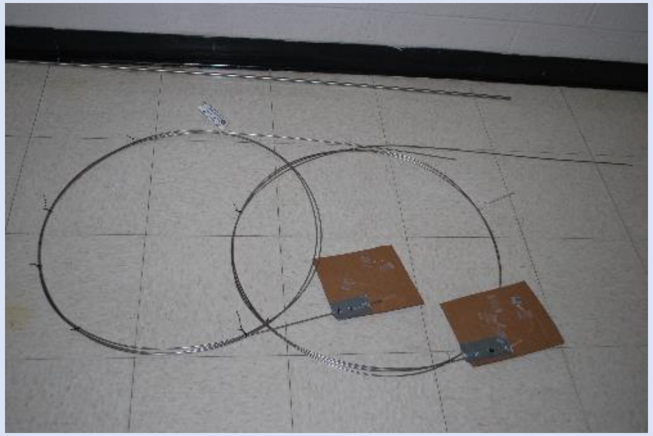


Sapphire Probe (1 m)

MIT Research Reactor Temperature Measurement

- Molten salt-loop development acceleration with distributed single-crystal harsh-environment optical fiber-sensors (ARPA-e 2019-2022)

Fiber-optic probe
(spliced radiation-resistant lead fiber to sapphire single-crystal fiber was inserted into protective stainless-steel tube)



Probe installed in Light Water Reactor

Installed fiber-optic probe into the dummy fuel element

Data acquisition station

Molten Salt temperature measurement with INL

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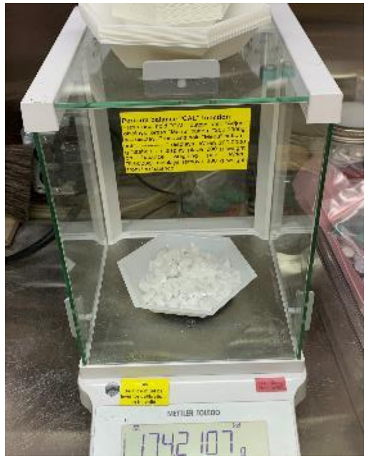


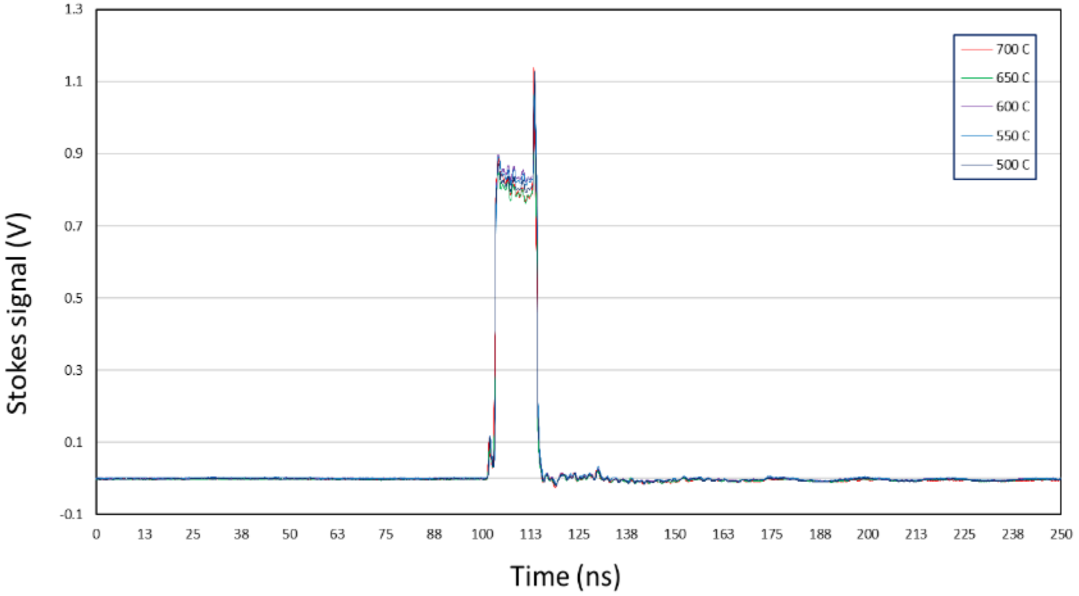
Chunks of NaCl-$MgCl_2$ eutectic salt-mixture

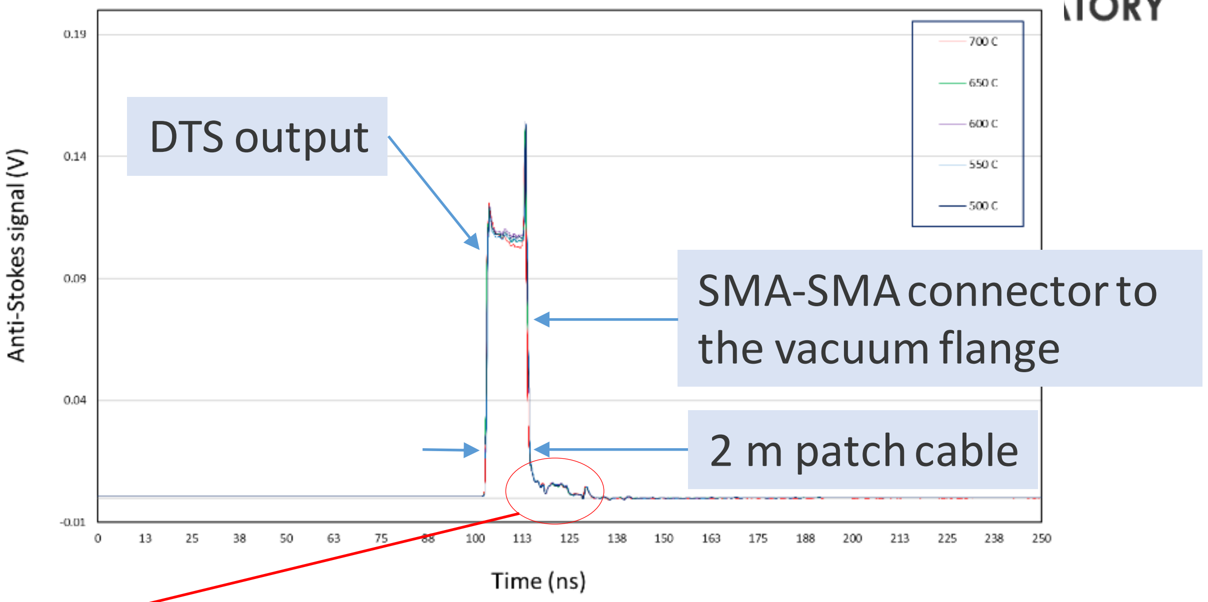
Salt-mixture melted at 500^{o}C

Chloride salt-mixture loaded in a pre-cleaned and dried glassy carbon crucible, inside a top loading Kerr furnace

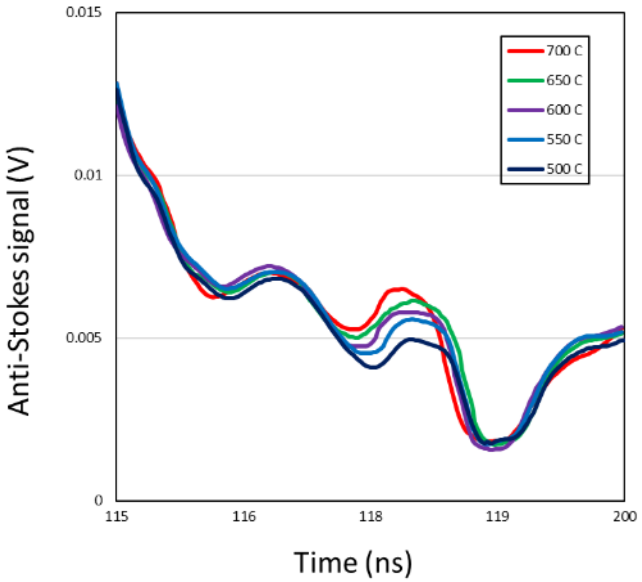
Ar-atmosphere glovebox with metallic cover and OD 6 window - designed as Class 1 enclosure for laser work

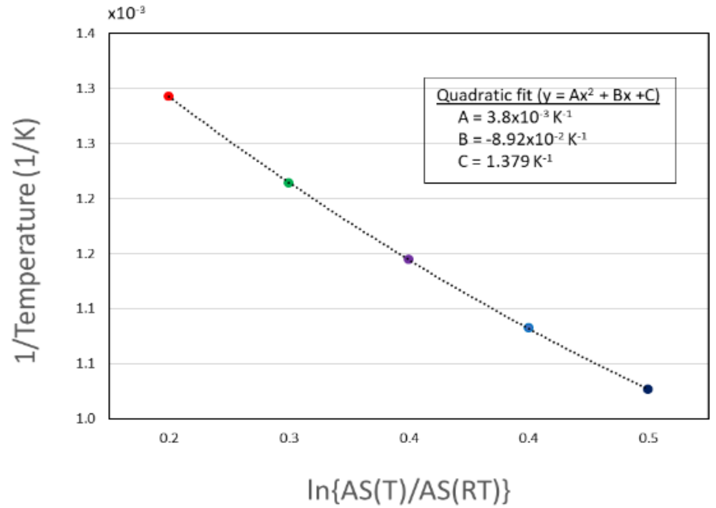
Molten Salt temperature measurement results





- **Salt temperature range**
  - From 500 to 700^{o}C, 50^{o}C increments

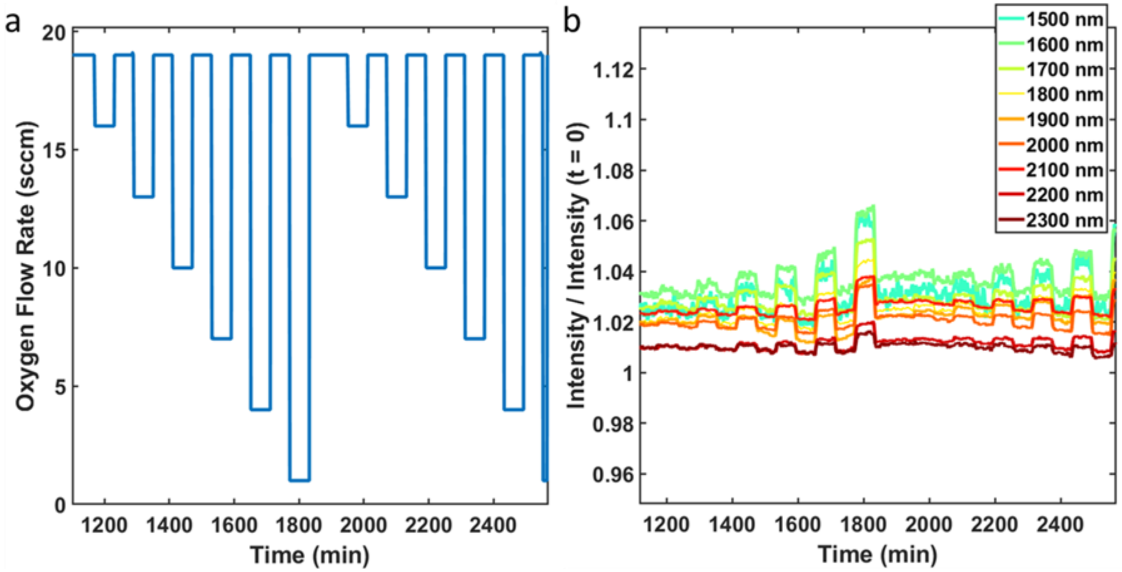




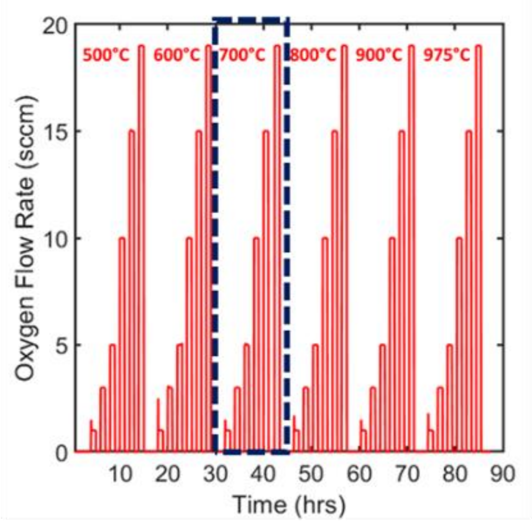
Temperature calibrated to AS signal relative to room temperature at peak temperature

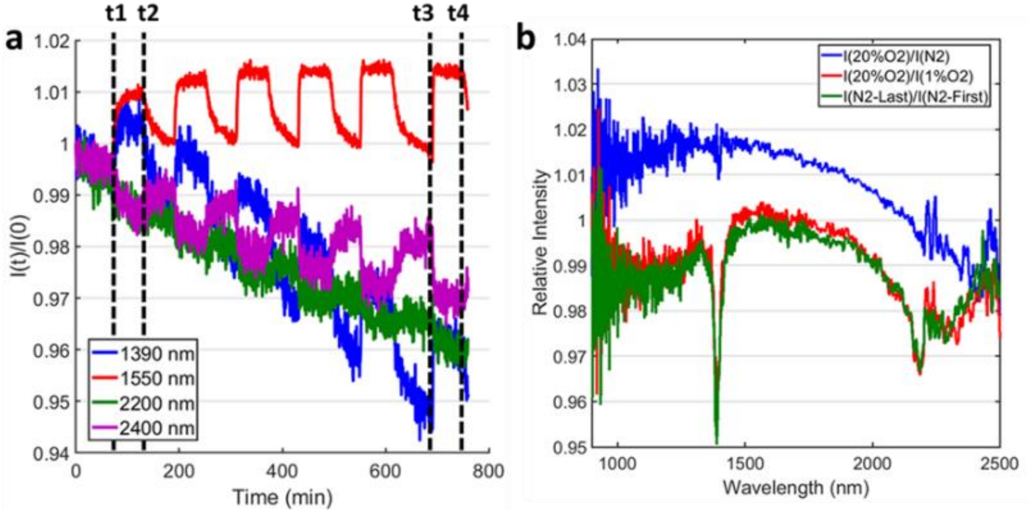
Task 21 –Development of Functional Sensor Materials for Oxygen Sensing

Fe-doped Strontium Titanate Sensing Layer



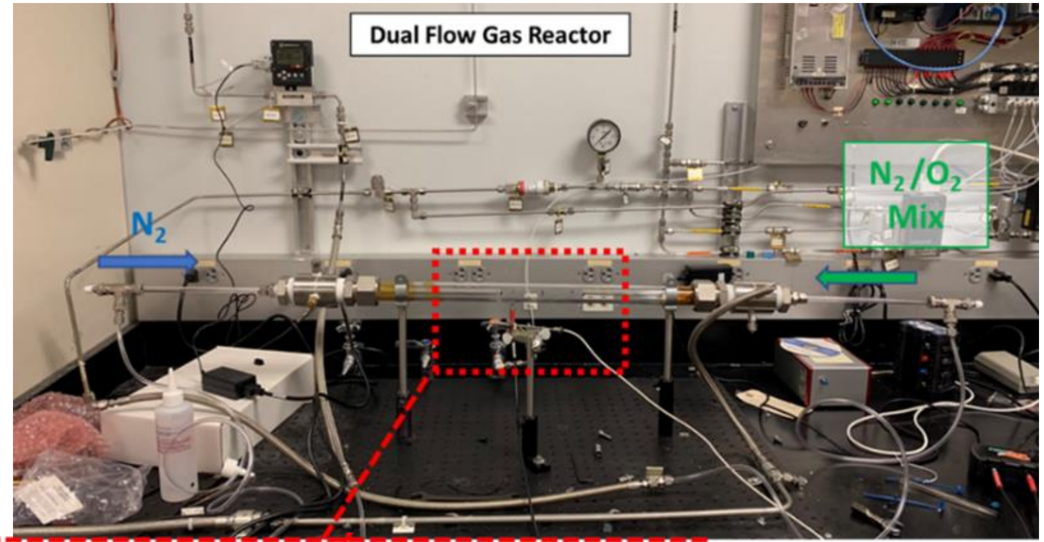
LSCF Sensing Layer

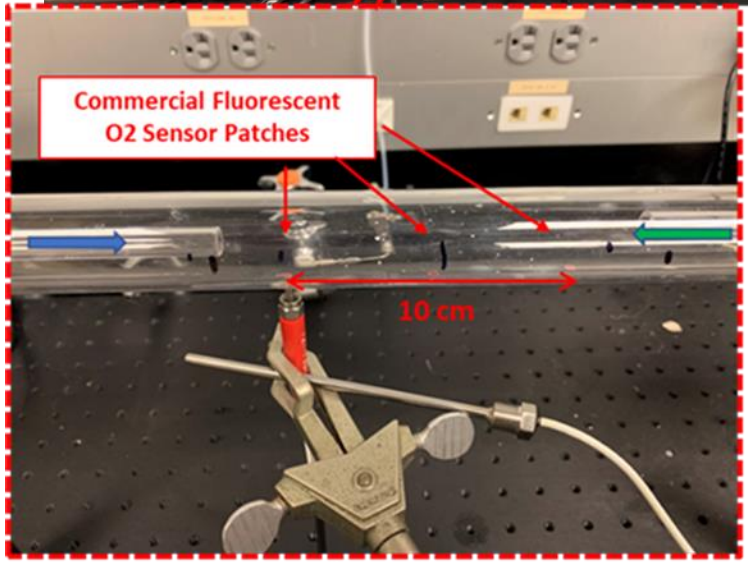


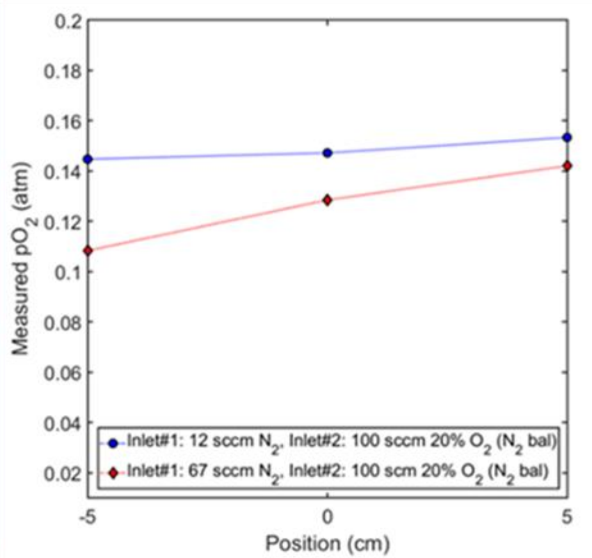


- Main candidates for oxygen sensing tested in EY21:
    - LSCF – sputtered ($La_{0.8}Sr_{0.2}Co_{0.2}Fe_{0.8}O_3$)
    - SFT – sol gel ($SrFe_{0.35}Ti_{0.65}O_3$)
- Tested at 500-900°C, 1-19% O_2, under humid conditions.
- **Technical challenge** – drift of point sensor response too large relative to 1-19% O_2 response. Approach for improving response, lowering drift (e.g., utilizing single crystal fiber) explored near end of EY21.

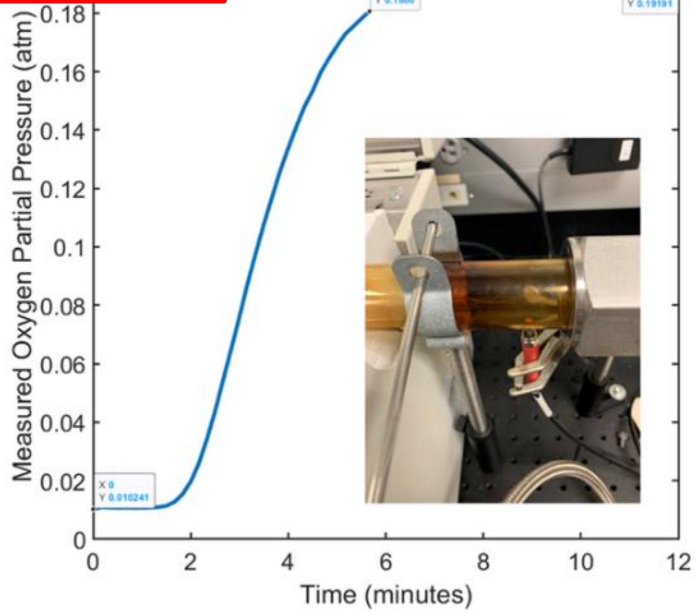
Gas flow concentration gradient reactor for oxygen sensing





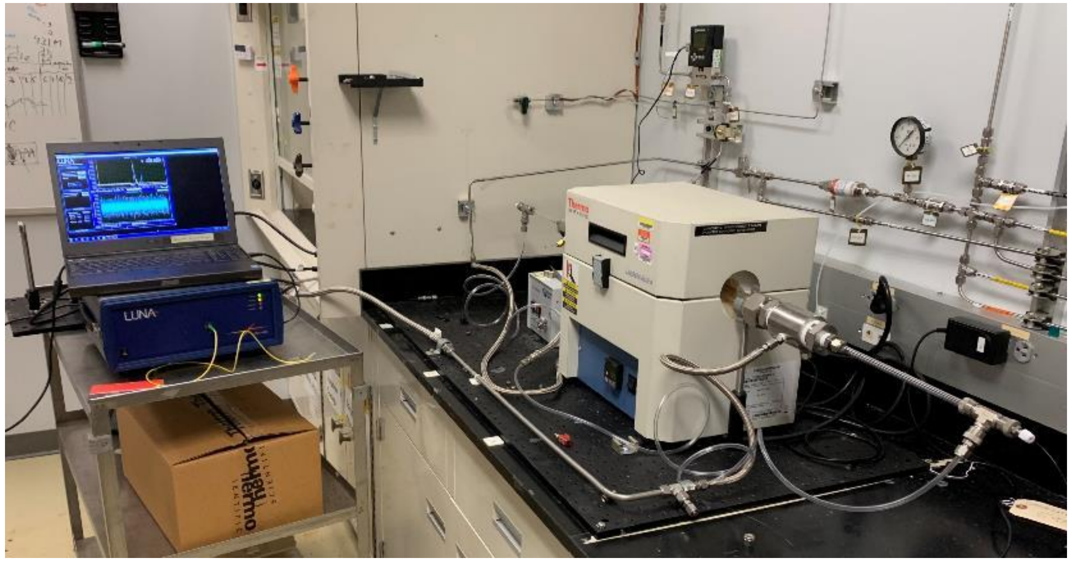


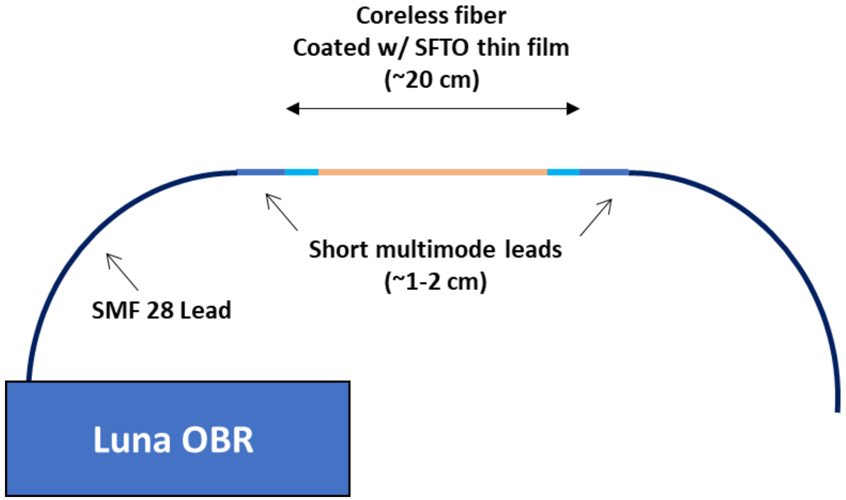
Measurement of oxygen partial pressure equilibration time in tube.

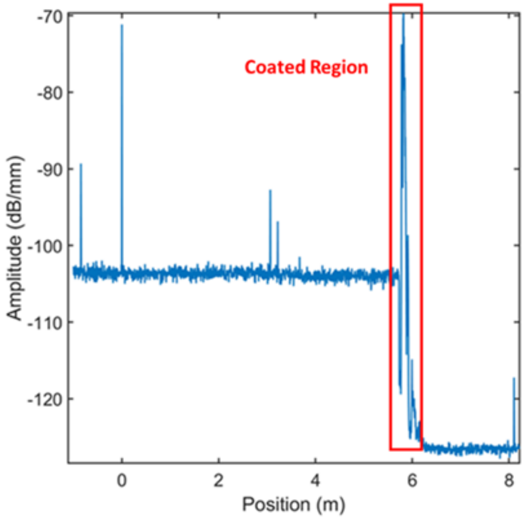


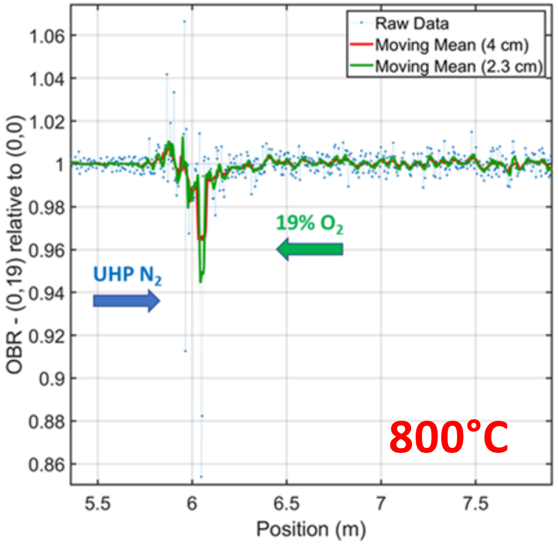
- System completed in EY21 for creating controllable gas concentration gradient (oxygen / nitrogen) at high temperature (up to 1000°C).
- Calibrated with commercial room temperature oxygen sensing system.
- Can be utilized in future for any gas available in ASDL across other projects.

Distributed oxygen measurement









- Dual gas flow system was tested with SMF / MMF hybrid sensor using Luna Optical Backscatter Reflectometer (OBR) for distributed sensing tests.
- Fiber sensor utilizing sol-gel based Fe-doped Strontium Titanate sensing layer tested for distributed oxygen sensing at high temperature (500-900°C).
- Initial results demonstrated strong backscatter from sensing layer and measurable variation under oxygen gradient.

Plasmonic materials for H2 and O2 – Advanced Sensors/ SOFC FWPs

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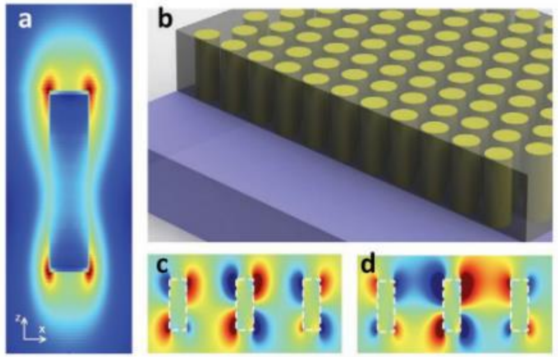
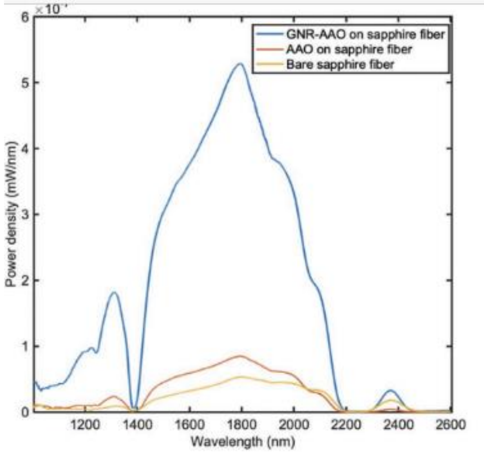
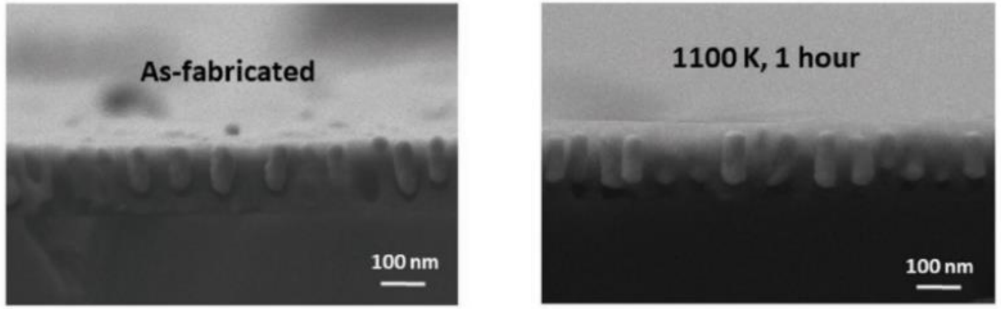
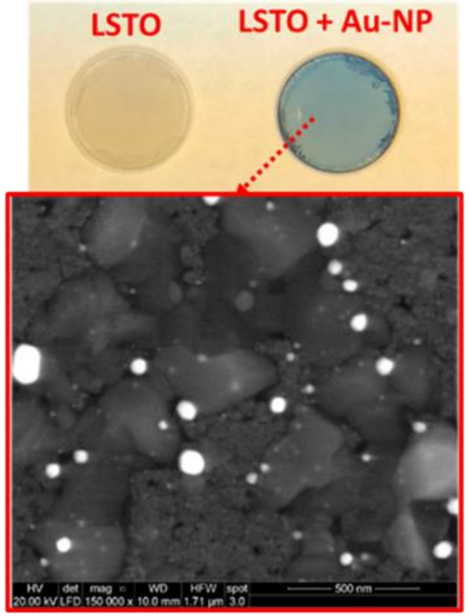
J. Li, et al. Small 17.17 (2021)

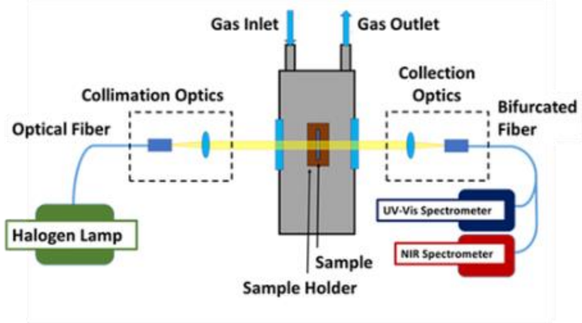


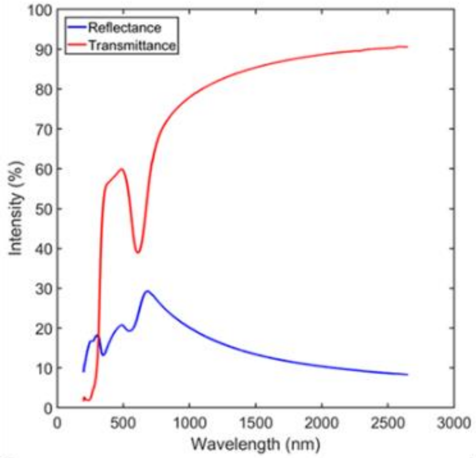


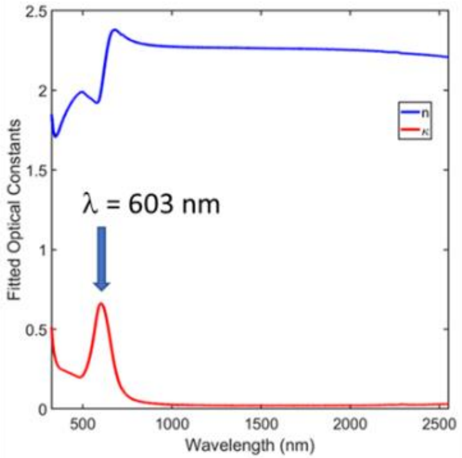


- Progress on development of plasmonic sensing layers for high-temperature gas sensing.
- Modeling of gold-nanoparticle incorporated oxides for O_2 and H_2 sensing (right).
- Sapphire embedded with gold nanorods for passive thermal (Blackbody) based sensing (top).









Wuenschell, et al. MRS Communications (2022).

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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