Single crystal fiber growth and sensing applications in energy

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NETL, RIC-MEM

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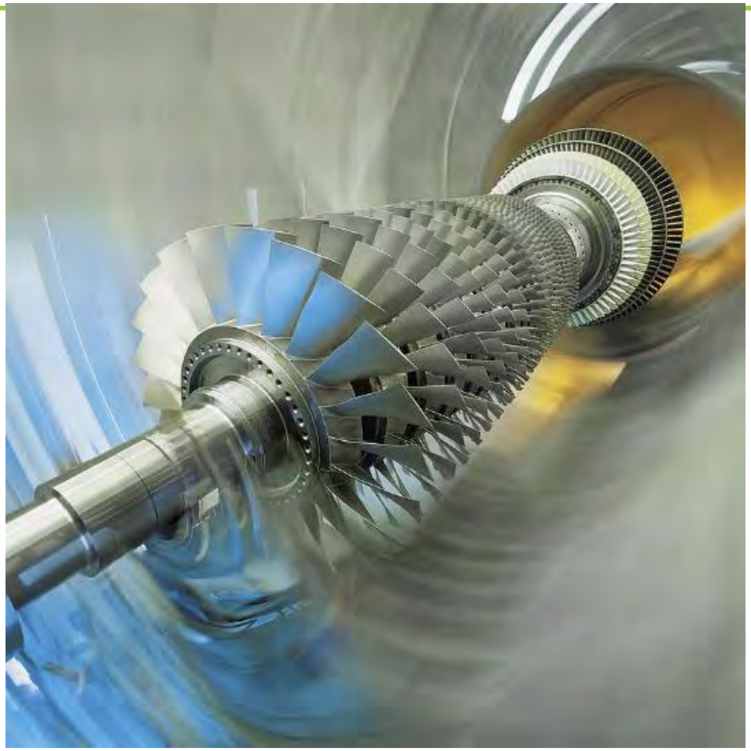
(LEIDOS contractor scientists)

U.S. DEPARTMENT OF ENERGY | NATIONAL ENERGY TECHNOLOGY LABORATORY

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 with harsh-environment sensing needs (fossil, nuclear, solar-thermal, etc)
- Control processes for efficiency ($$, fuel, CO_2), Predict failures for maintenance

Why use single crystal fibers in energy applications?



	Coal / Waste plastic biomass Gasifiers	Combustion Turbines (H2 or NG)	Solid Oxide Fuel Cells / Electrolyzers	Hybrid systems	Nuclear	Solar Thermal
Temperatures	Up to 1600°C	Up to 1300°C	Up to 900°C	Up to 1000°C	Up to 1000°C	Up to 700°C
Pressures	Up to 1000psi	Pressure Ratios 30:1	Atmospheric	System dependent	High pressure steam	High pressure steam
Atmosphere	Highly Reducing, Erosive, Corrosive	Oxidizing	Oxidizing and Reducing	Oxidizing and reducing	Gamma and Neutron radiation	Daily heating/cooling
Examples of Important 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	Head-space gases, water, molten salt	Water,brine, molten salts

❖ **Why optical fiber?**

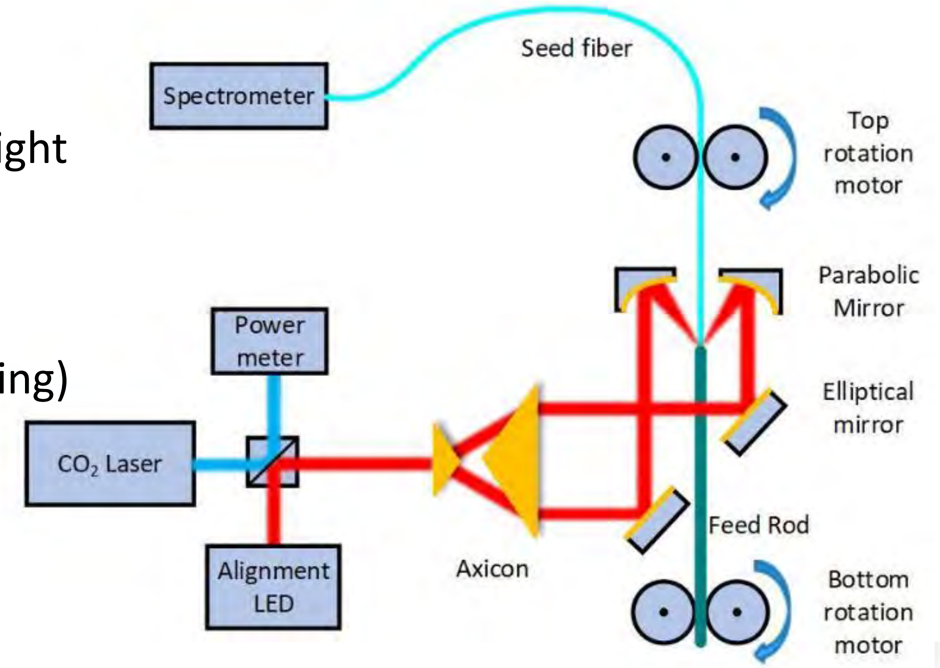
1. No electrical interference
2. Medium temperature (~800c)
3. Single Feedthrough
4. Inexpensive
5. Easily functionalized
6. Distributed!

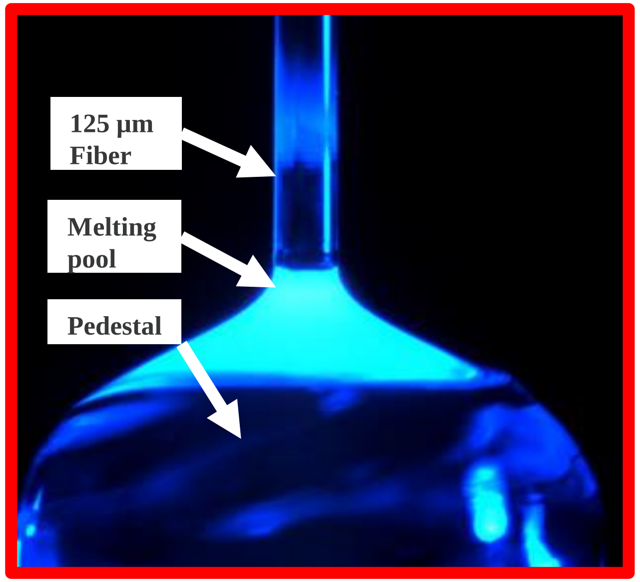
❖ **Single crystal fiber**

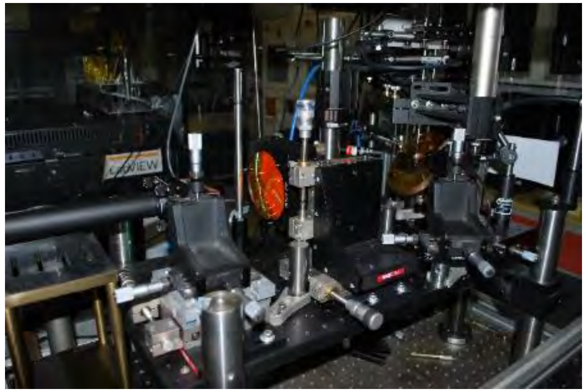
1. High melting point
2. Corrosion resistant
3. Compact size (100 microns)
4. Wide transmission window
5. Benefits of silica +low-OH absorption

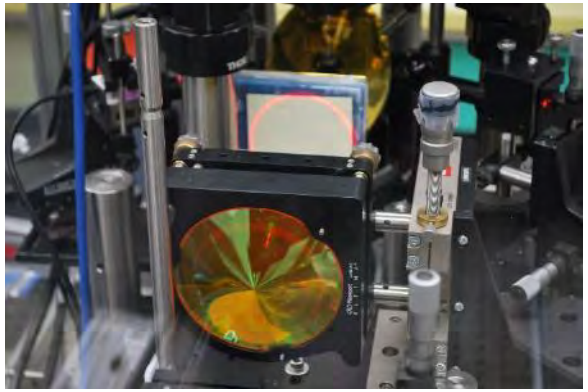
Making Single-crystal fiber with LHPG

- 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)





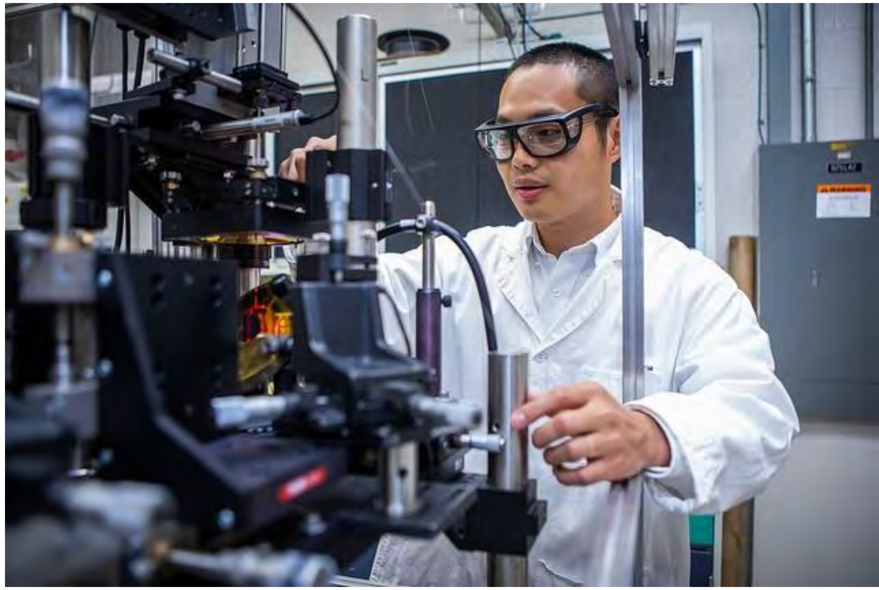




NETL LHPG Capabilities and features

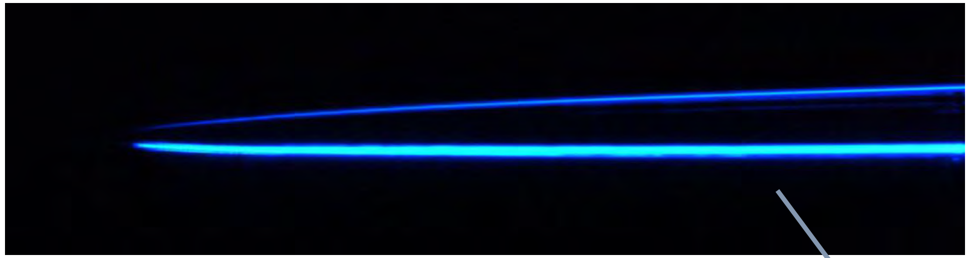
Some NETL LHPG stats:

- 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

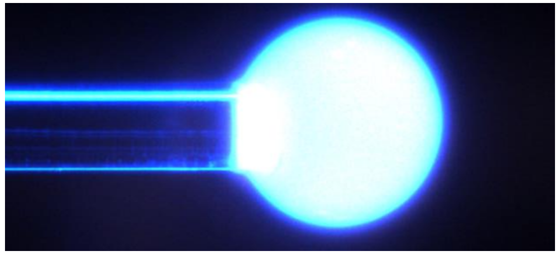


Active diameter variation for sensing

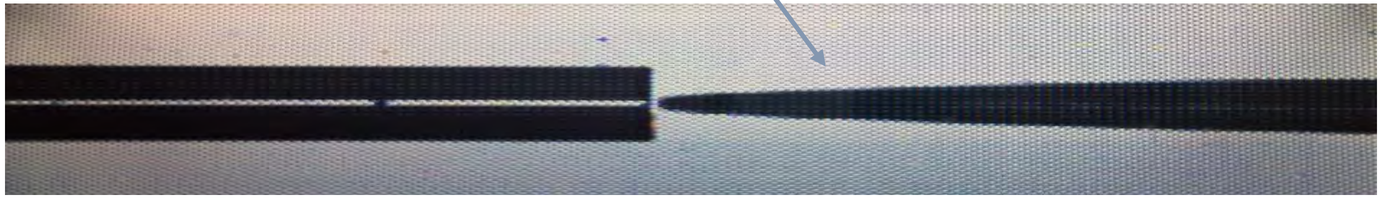
Sapphire fiber taper



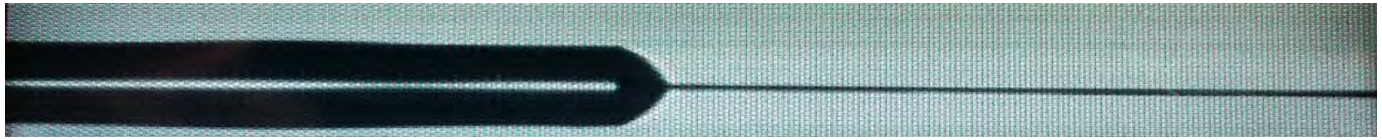
Sapphire ball lens (reduces end reflections)



Light coupling between single mode fiber (Left) and sapphire fiber (Right) with the taper tip



Light coupling between single mode fiber (Left) and silica multimode fiber (Right) with the taper tip



*SPIE Rising Researcher Award 2018

Collaboration / U Pitt LHPG

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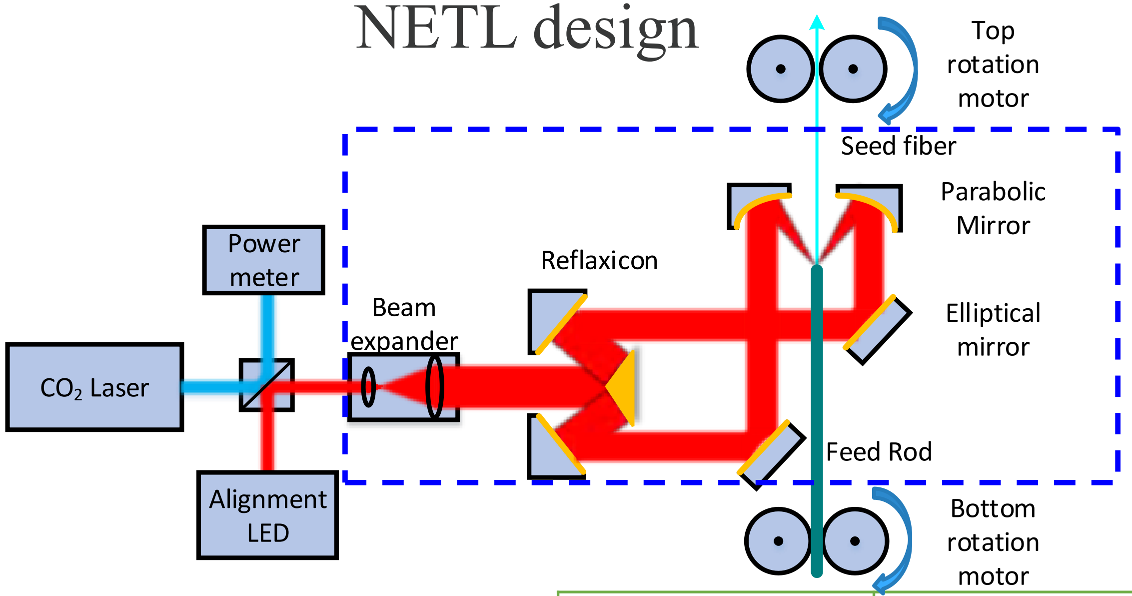
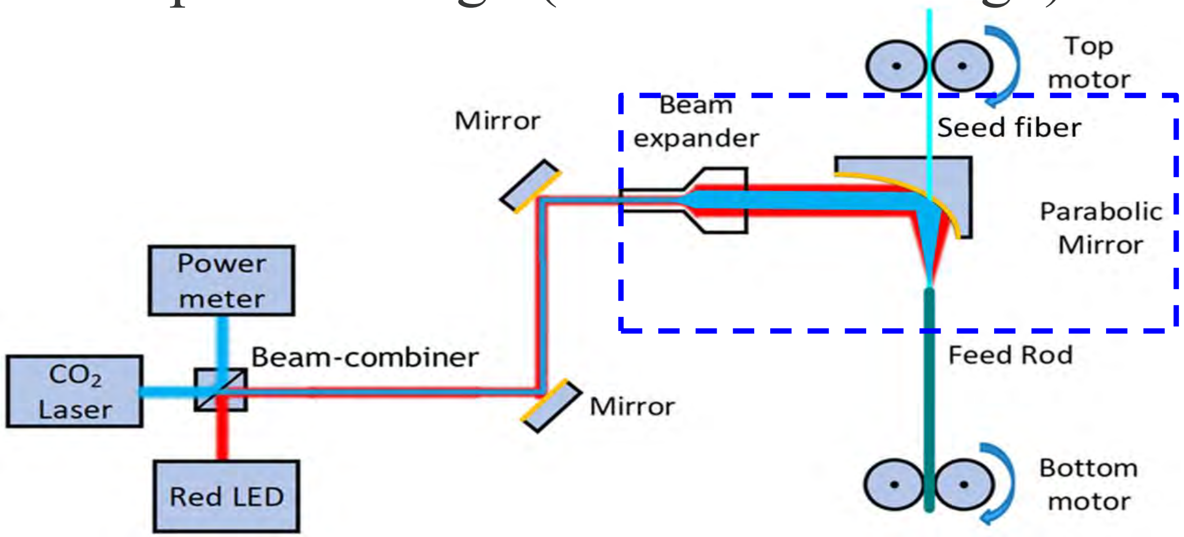
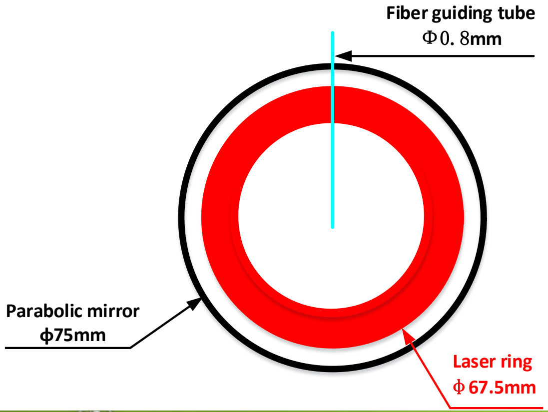
Long-length/continuous NETL design



Simplified design (Univ. of Pittsburgh)

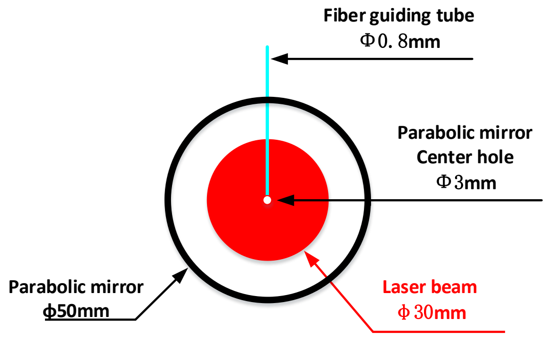


Continuous design



	Continuous design	Upitt. simplified design
Focusing optics	Axicons + turning mirror + paraboloidal mirror	Beam expander + 90 degree off-axis mirror
built	Customization	Off-the-shelf
Alignment DOF	27	18
Beam profile	Ring/Donut shape (axially symmetric)	Gaussian/flat top (axially asymmetric)

Simplified design



Contributed by Paul Ohodnicki, U Pgh.

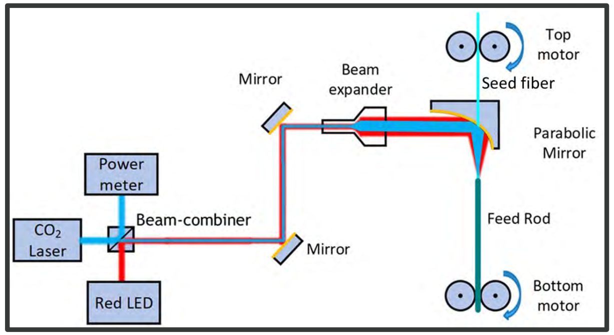
Growth of novel compositions (U of Pgh)

Material Uses
- ❖ Crystal Fibers with Enhanced Stability and/or New Functional Performance

Feedstock Manufacturing
- ❖ Powder Processing Techniques with Optimized Compositions / Structure

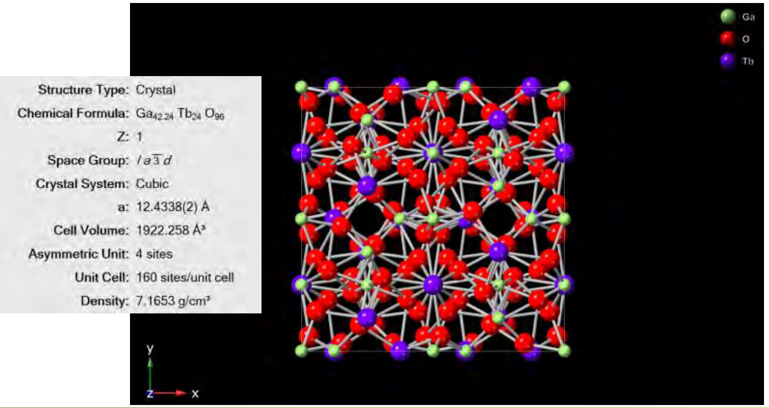
Growth Optimization
- ❖ Optimize Growth Processes Considering Thermodynamics and Kinetics

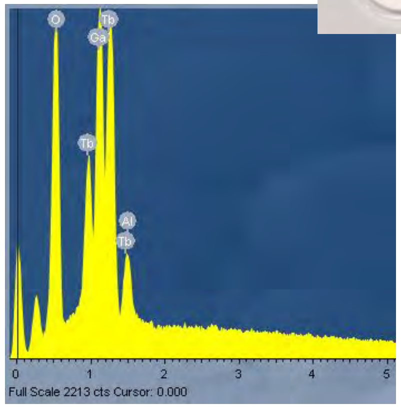
LHPG System Operation



Material Characterization

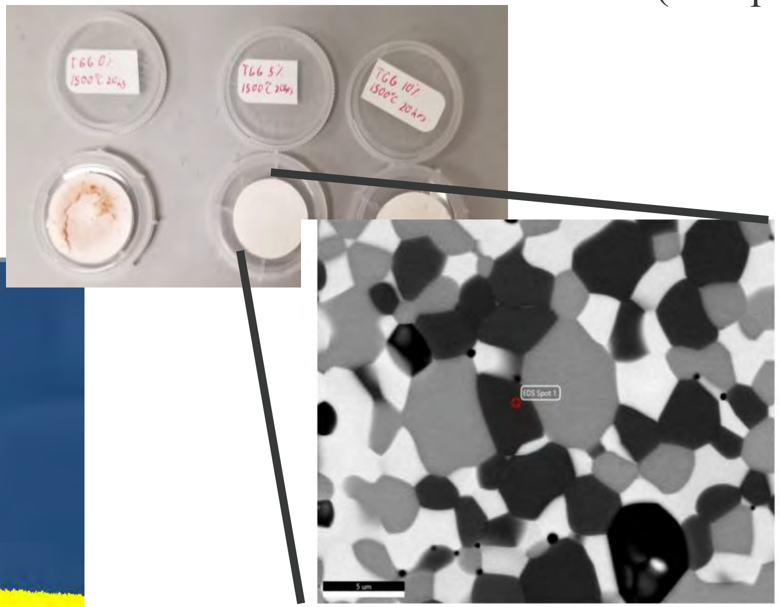
- ❖ Single Crystal XRD: structure determination
- ❖ EDS / EPMA: chemical composition





Feedstock Manufacturing

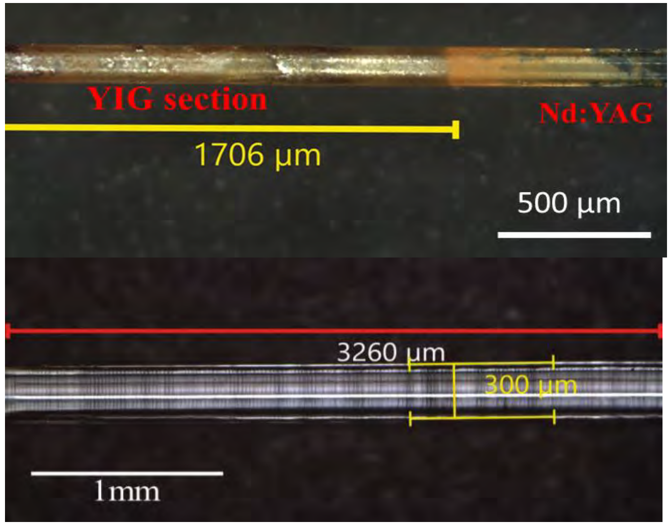
- ❖ Powder based manufacturing of different polycrystalline feedstocks for source rods in LHPG
- ❖ Allows for experimentation with varying compositions



Feedstock Microstructure

New Material Growth utilizing LHPG

- ❖ Versatility in growing refractory oxides (Including new functional oxides)
- ❖ Crucible free, high purity, diameter > 100 μm
- ❖ Expanding capabilities of crystal fiber sensors (new parameters, enhanced stability, etc.)



Fiber Growth

Contributed by Paul Ohodnicki, U Pgh.

Experimental SC fiber Cladding

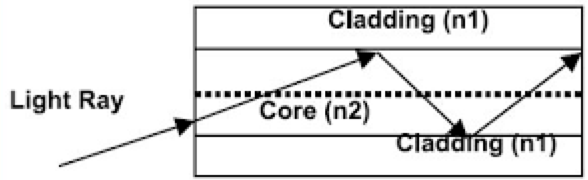
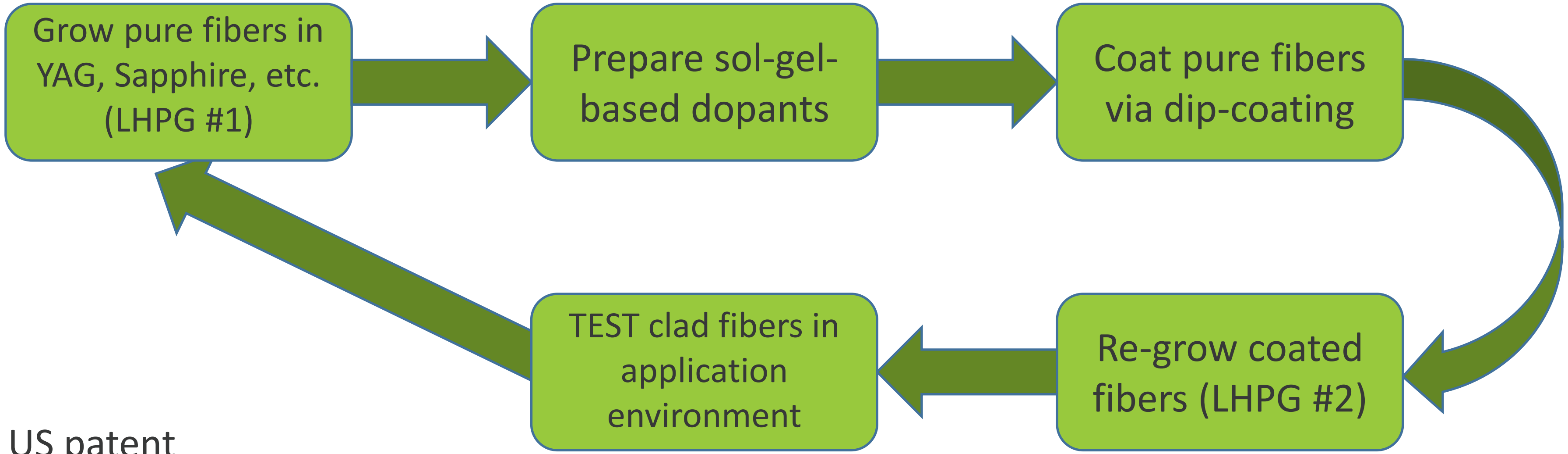


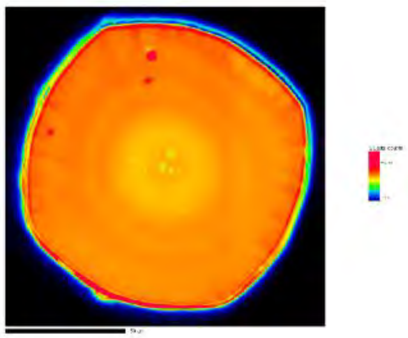
Figure 1: Light launched into the fiber core

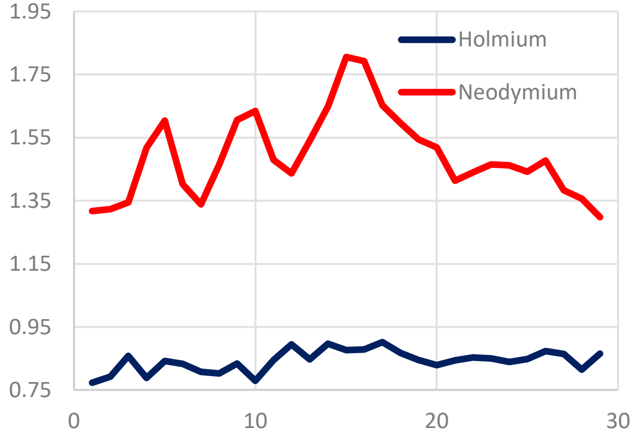
- Grow cladded fibers with 2-stage LHPG
  - Sapphire or YAG
  - Sol-gel (or other) dopant additions
- Evaluate materials compatibility in energy systems
- Improve fiber performance



*provisional US patent

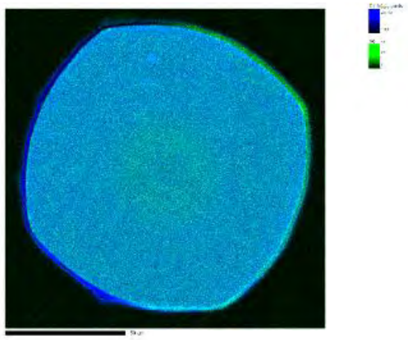
Cladding Application

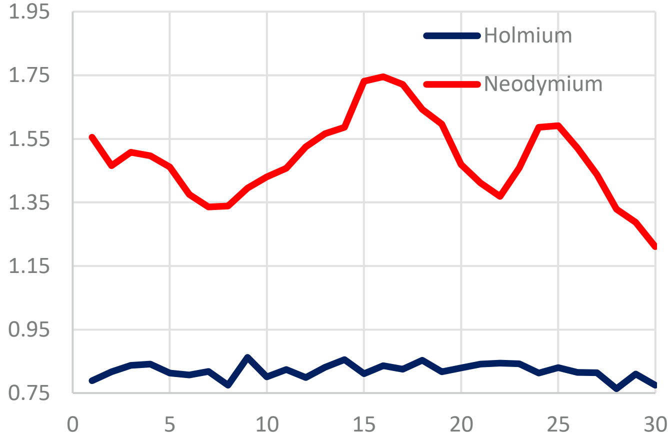




Dopant Species Made to Date:

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

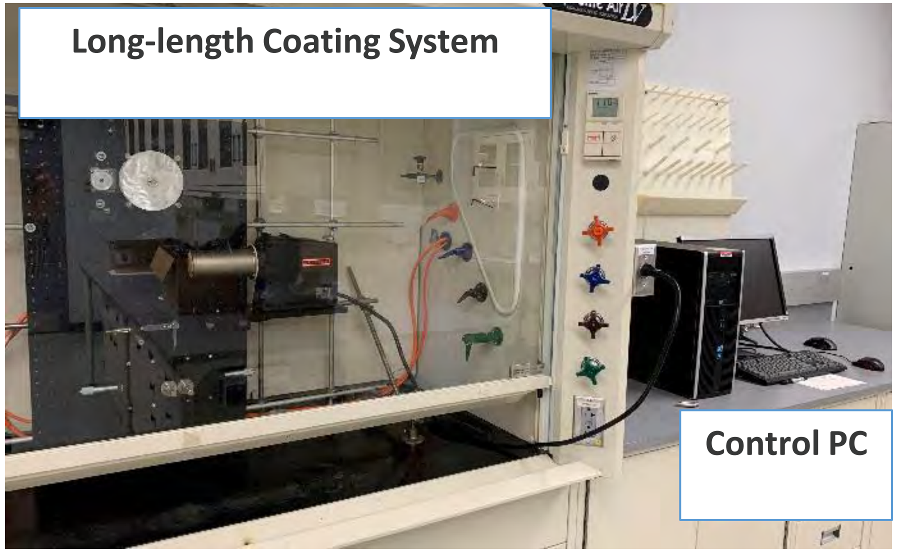


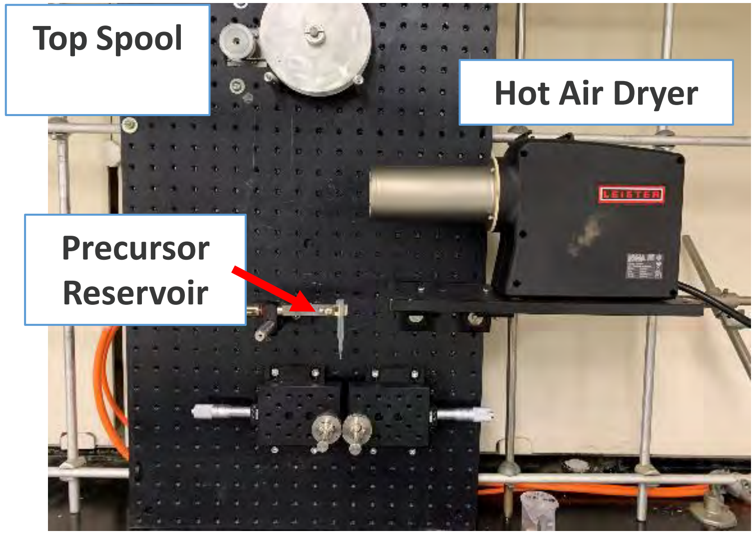


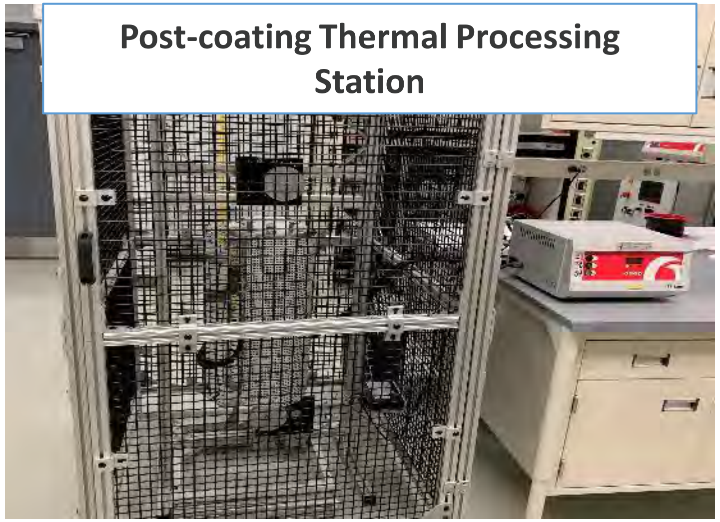
Automatic Dopant Segregation through LHPG: Top left: Visible light guiding in GRIN YAG fiber, Top right: EMPA map of Nd concentration in a GRIN YAG fiber, Bottom plots: Co-doped Nd and Ho: YAG fiber dopant concentrations in X (left) and Y (right)

Reel-to-Reel sol-gel processing system for cladding dopant additions

- Coater designed to coat long lengths of single crystal fiber (~several meters) 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

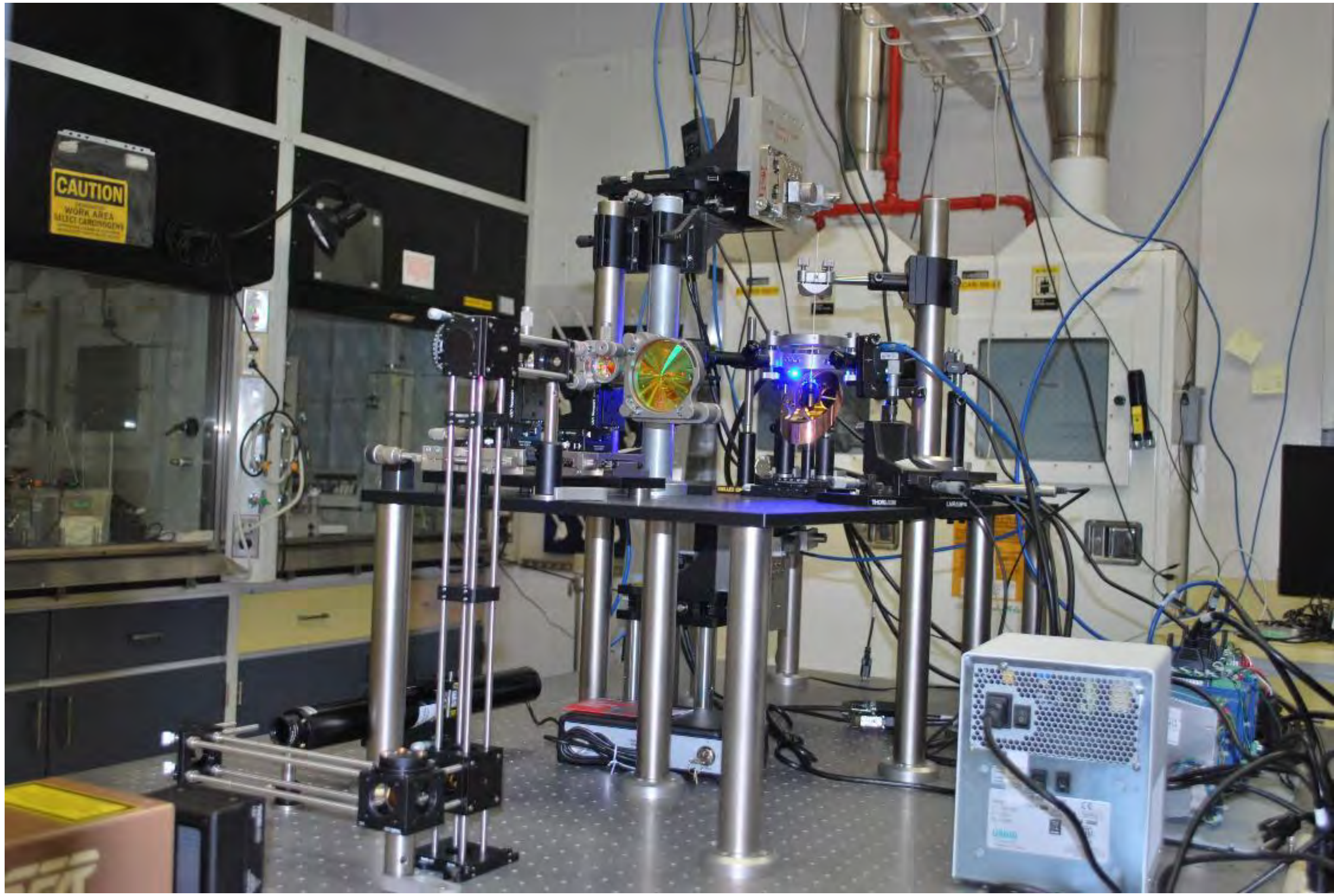






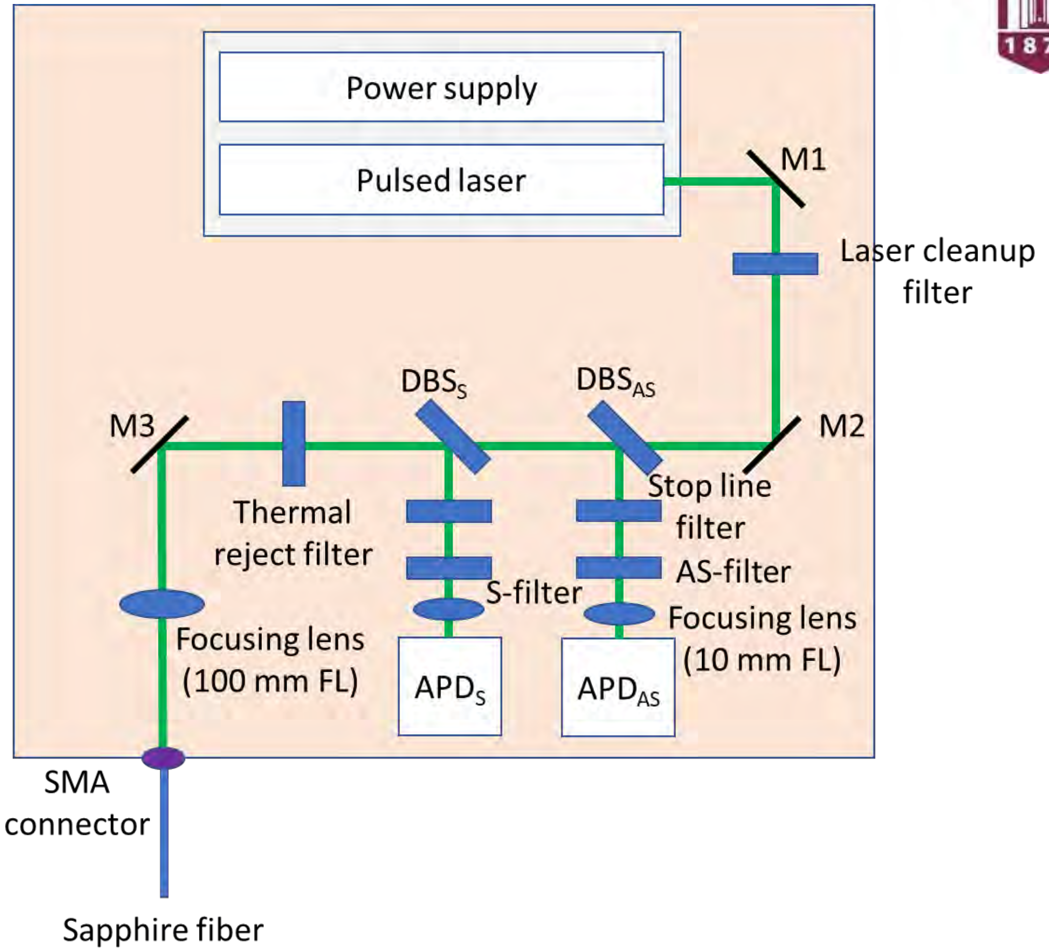
Novel dual LHPG system for clad sc-fiber

- 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



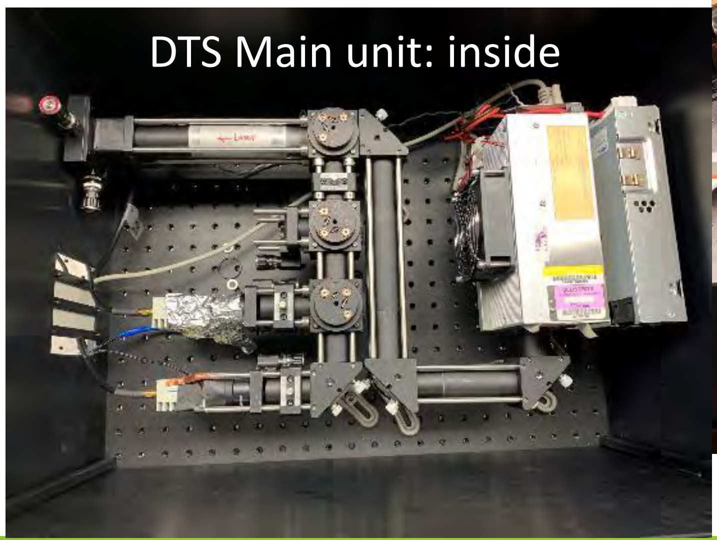
How an SC-fiber becomes a T-sensor

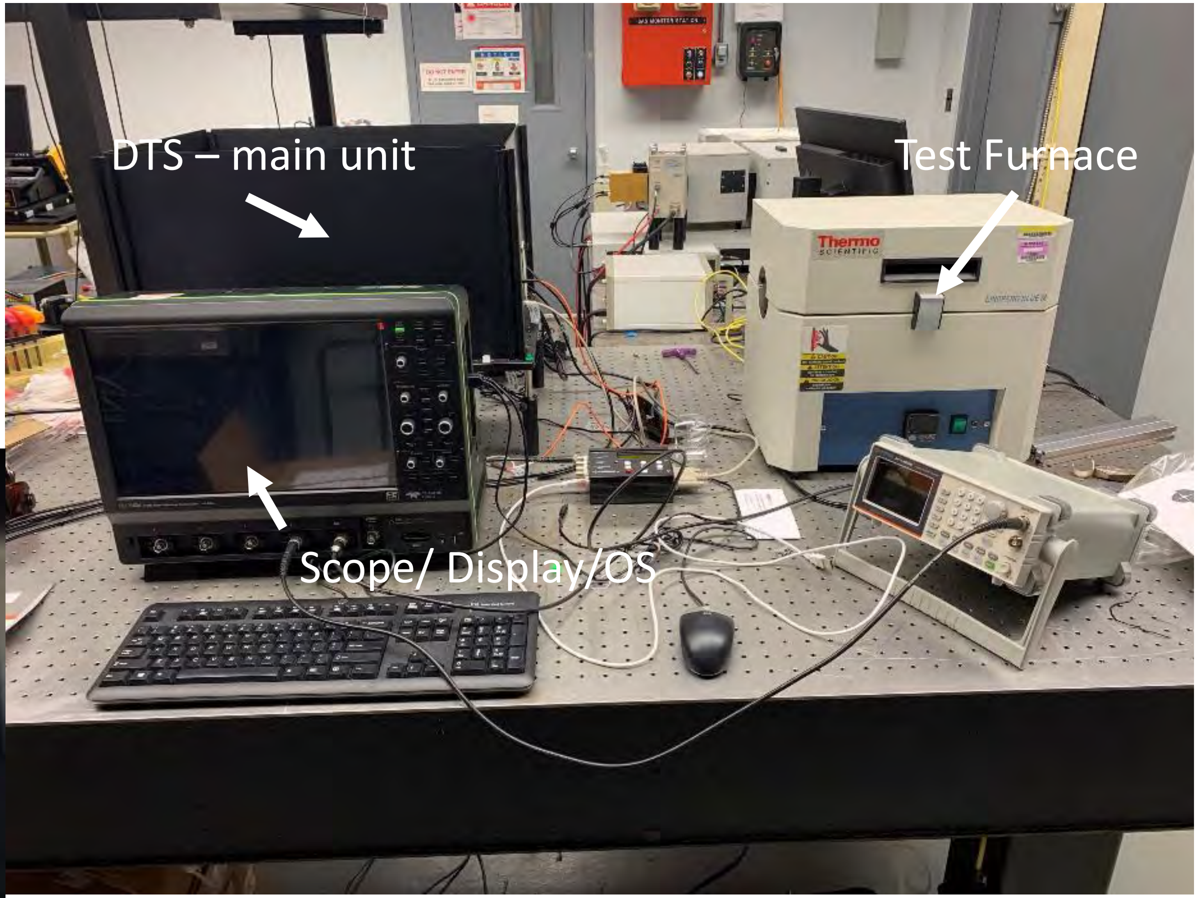
- Introducing the NETL Raman DTS (distributed temperature sensor)
- Pulsed ~350ps 532nm green laser
- Excites Raman Scattering as pulse propagates
- Collects Raman with Fast avalanche photodiodes
- Optics designed for sapphire or YAG fiber
- First interrogator for SC-fiber
- First interrogator produced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



Raman DTS – Lab Prototype

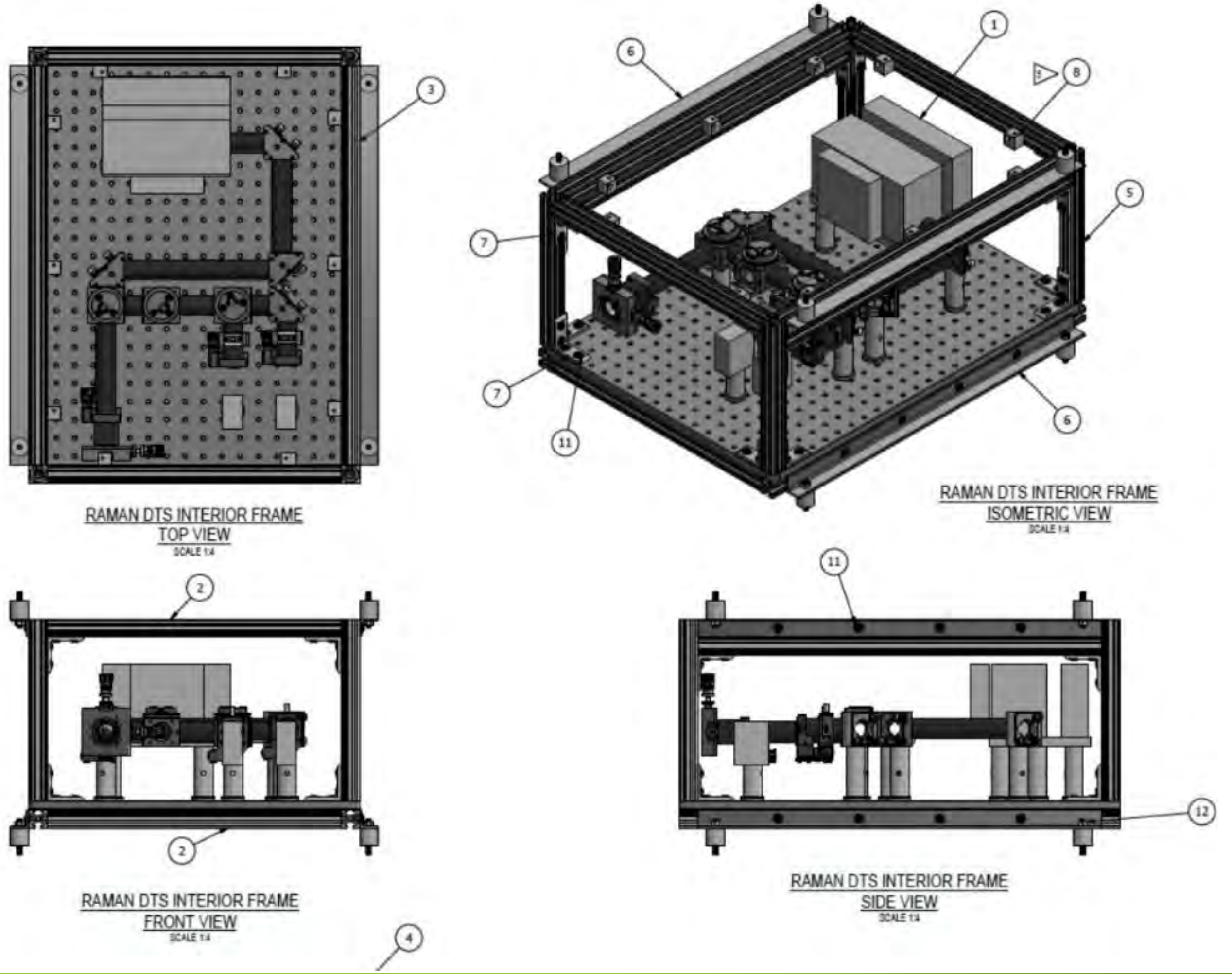
- Off-the-shelf components
- Breadboard construction
- Enabled design optimization/tinkering
- Improved prototype used for field-testing / product version





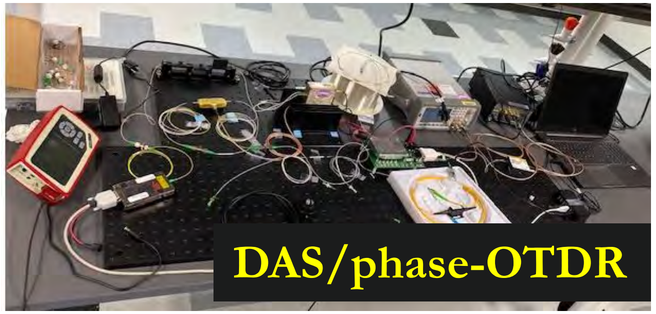
DTS Field Prototype design

- 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



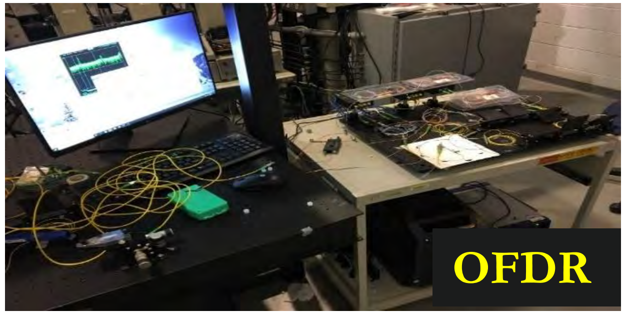
NETL Custom Build Interrogators and their performance

Cost: <40k



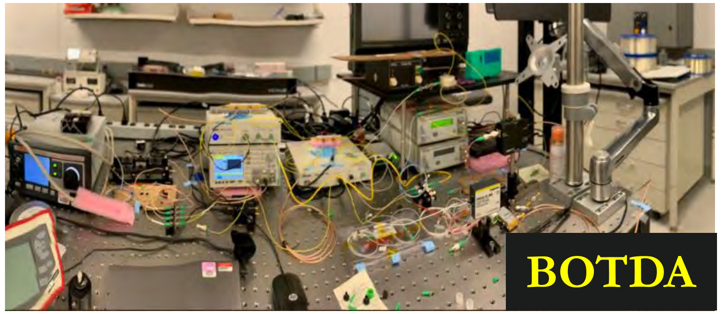
Sensing range = >50 km;
Spatial resolution = 1-2m;
Acoustic frequency range ≤20 kHz (depends on the fiber length);
Frequency resolution <2 Hz;
Laser safety: Class 3B

Cost: <35k

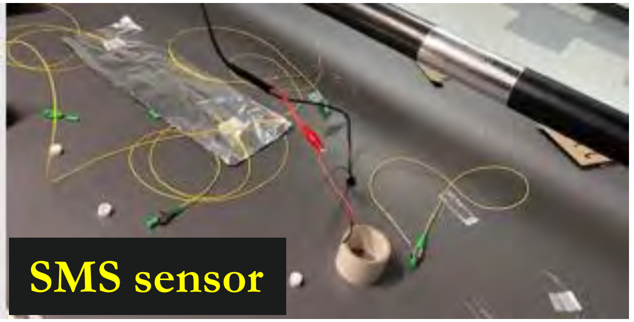


Sensing range = ≤1km;
Spatial resolution = <1mm;
Temperature resolution: 0.1°C
Strain resolution: 2με
Laser safety: Class 1

Cost: <70k



Sensing range = ≤150 km;
Spatial resolution = <5m;
Temperature resolution: ±1 to 2°C
Strain resolution: 10 to 20 με
Laser safety: Class 3B



Cost: <3k

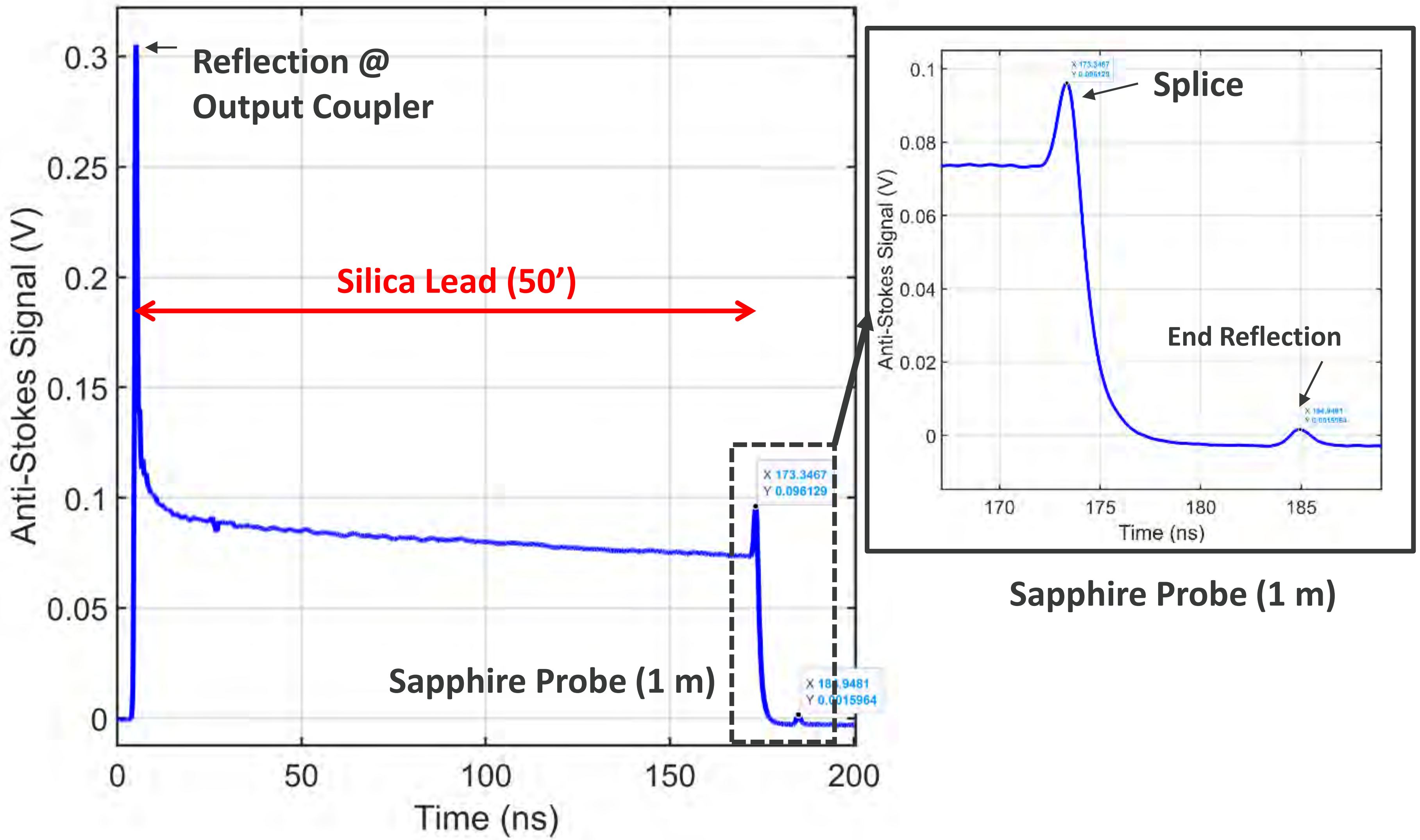
Acoustic frequency range: 5Hz to 1MHz;
Frequency resolution <1 to 2 Hz;
Laser safety: Class 1

Cost: <8k

Acoustic frequency range: 1Hz to 500kHz;
Frequency resolution <1 to 2 Hz;
Laser safety: Class 3B

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.

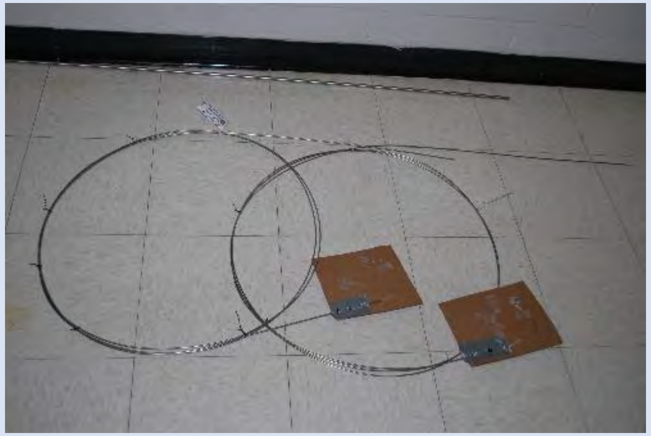


Sapphire Probe (1 m)

MIT Research Reactor Temperature Measurement

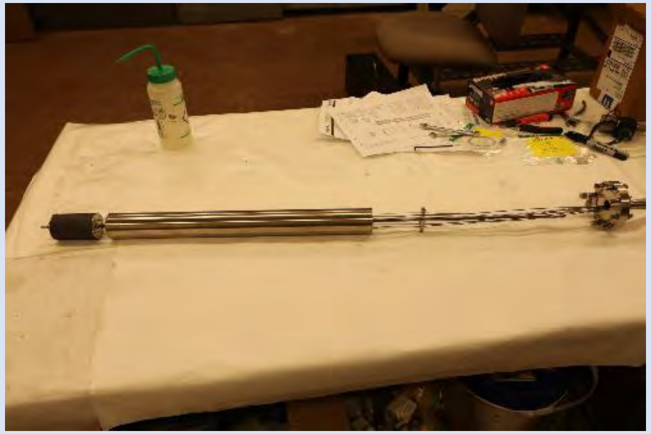
- Molten salt-loop development acceleration with distributed single-crystal harsh-environment optical fiber-sensors (ARPA-e 2019-2022)

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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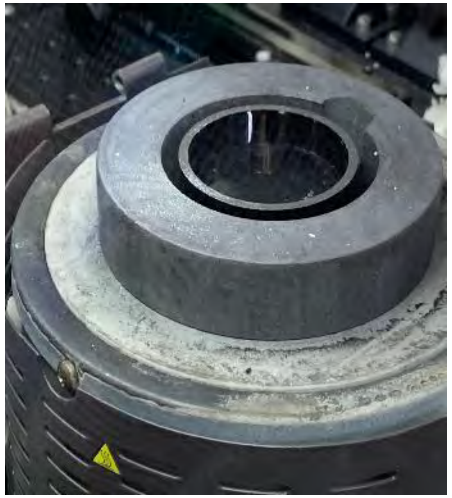
(with Calderoni, Gakhar, McCary)

Chunks of NaCl-$MgCl_2$ eutectic salt-mixture

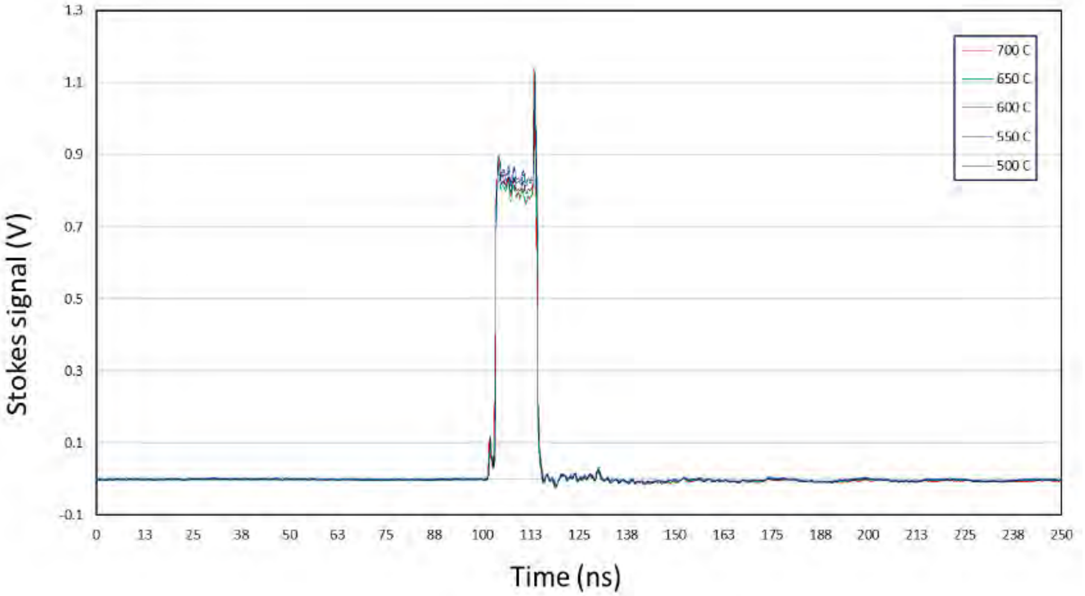


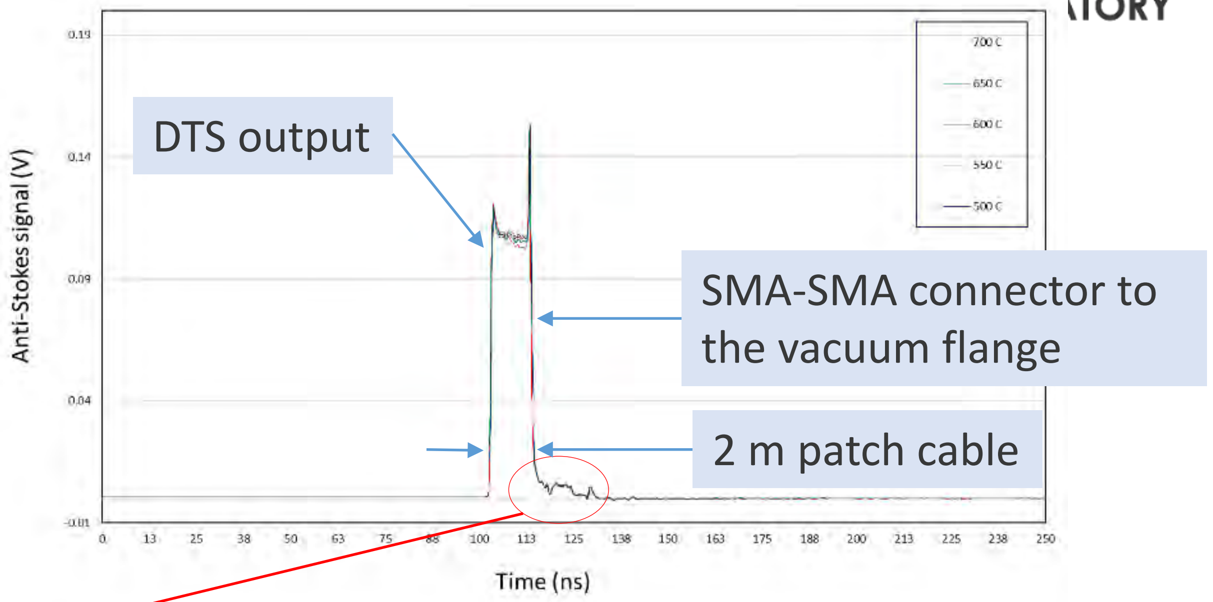
Salt-mixture melted at 500°C

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

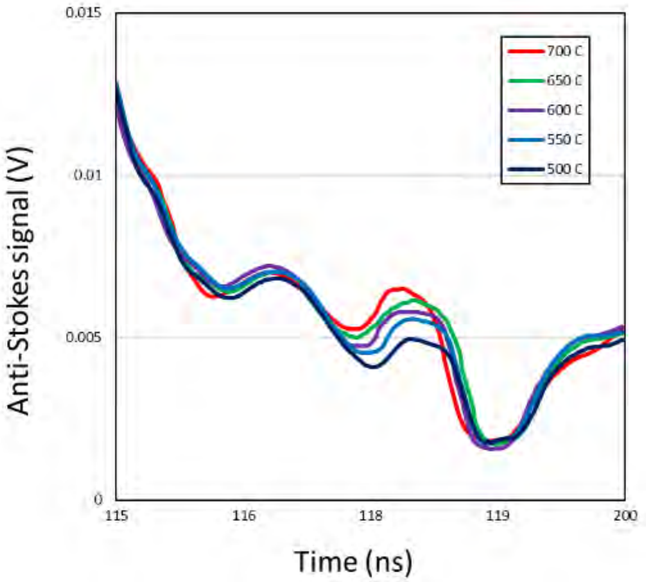
Ar-atmosphere glovebox with metallic cover and OD 6 window - designed as Class 1 enclosure for laser work

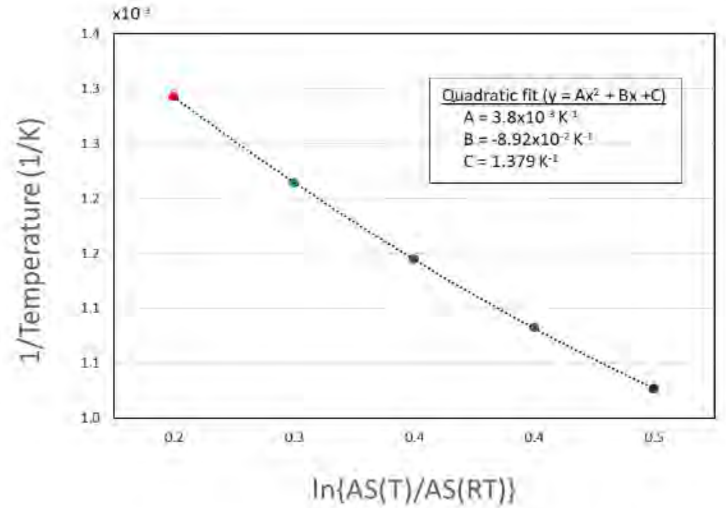
Molten Salt temperature measurement results





- **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

Special considerations for nuclear sensing

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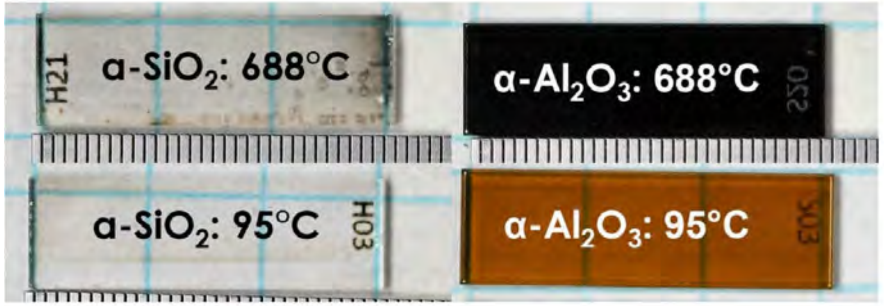

Fig. 5: Optical transparency of silica and sapphire after accumulating a fast neutron fluence of 2.4×10^{21} n_{fast}/cm^2

Chen and Petri, Nuclear Energy 2022

Increased packaging/cladding requirements/options:

- Novel glass/ceramic compositions (tubing or cladding) to deflect or slow neutrons
- Correlated temperature/ radiation limits
- Alternate crystal species (other than sapphire)
- “Protective” dopants (higher neutron cross section near surface)
- Rayleigh enhancement

DTS in fossil – Pressurized Pulse Combustor

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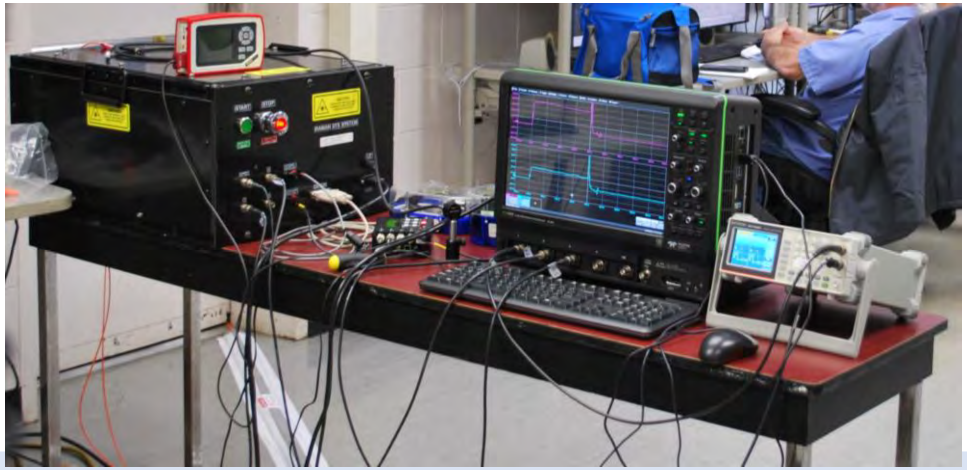
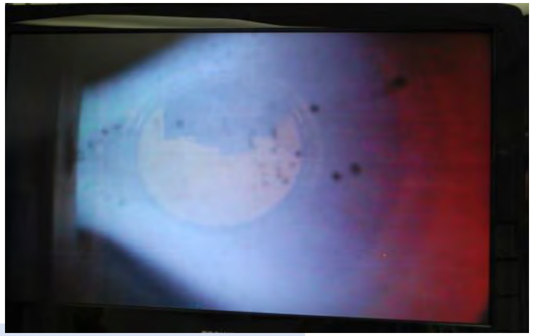
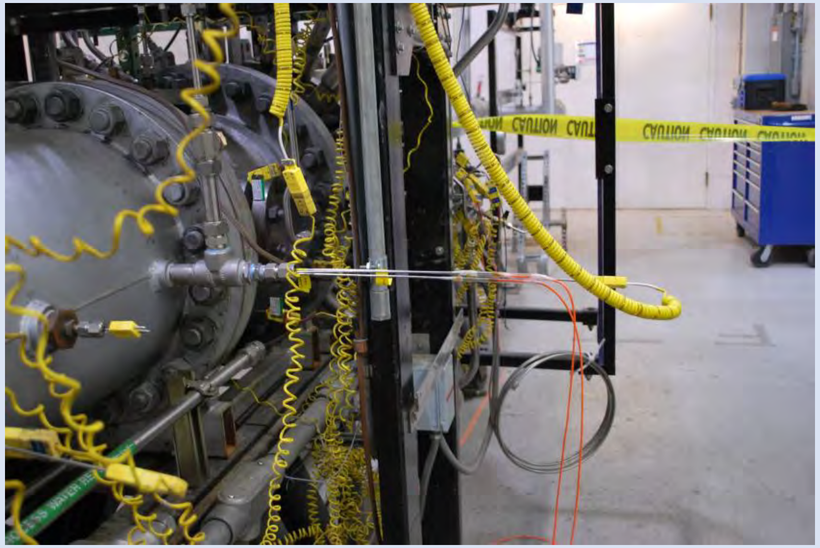
- Fully distributed sensing – 5cm resolution
- Temperature measurements above 1100C
- Multiple probes deployed (sapphire and YAG)
- Transients observed easily







Two fiber-optic probes (sapphire and YAG) & thermocouple (left) and its installation on PPC test rig (right)

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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