High-Temperature Sapphire Pressure Sensors For Harsh Environments

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Project Information

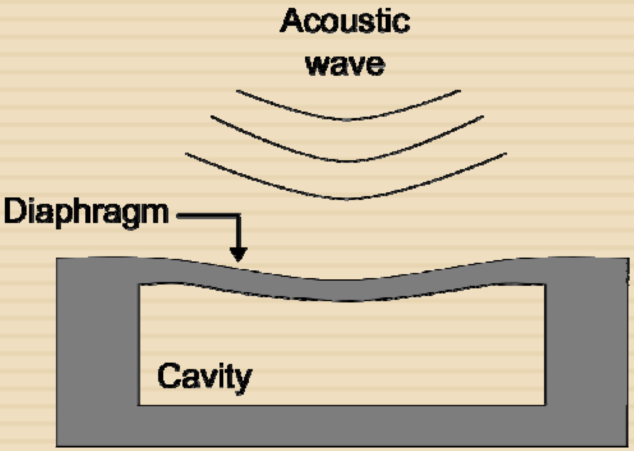
- Focus: Development of novel machining methods for the fabrication of harsh environment pressure sensors
- Award information
  - Project title: “High-temperature sapphire pressure sensors for harsh environments”
  - Award #: DE-FE0012370
  - Program manager: Sydni Credle
  - Duration: 3 years (1 year NCE) started Jan 2014
- Project team
  - UF (Project lead)
  - FSU

Technical Objectives

1. Novel sapphire fabrication processes
    - Subtractive machining: ultrashort pulse laser (modeling + experimental)
    - Additive manufacturing: spark plasma sintering
2. Characterize and mitigate thermo-mechanical damage
    - Statistical modeling of laser pulse-material interactions
3. Fabricate, package, calibrate, and demonstrate sapphire optical pressure sensor
    - Application for harsh environments (> 1000 °C and > 1000 psi)

Outline

- Introduction
- Thermal Damage Modeling
- Micro and Nano-indentation Mechanics
- Sensor Fabrication
- Acoustic Characterization
- Conclusion/Future work



Introduction

Motivation

- Next generation advanced energy systems will require harsh environment instrumentation:
  - Process control/closed loop feedback
  - Increase efficiency, reduce emissions & cost
- Sensor operational requirements
  - Temperature: >1000 °C and dynamic pressure: up to 1000 psi
  - Atmosphere: corrosive and/or erosive
- Conventional pressure sensor instrumentation limited to ~500 °C
- Current temperature mitigation techniques:
  - Stand-off tubes, water cooling

Transduction Mechanism Selection

	Capacitive	**Piezoresistive**	**Optical**
Thermal drift	✓	X	✓
DC measurement	✓	✓	✓
EMI insensitivity	X	X	✓
Harsh environment capability (>500 °C)	X	X	✓
Packaging simplicity	✓	✓	X

- Optical transduction (intensity modulation – optical lever) is selected given our constraints

Pro	**Con**
Simple/robust fabrication	Lower sensitivity
Incoherent source	
Single or multimode fibers	

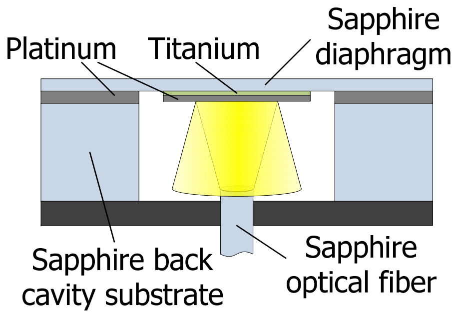
Material Selection

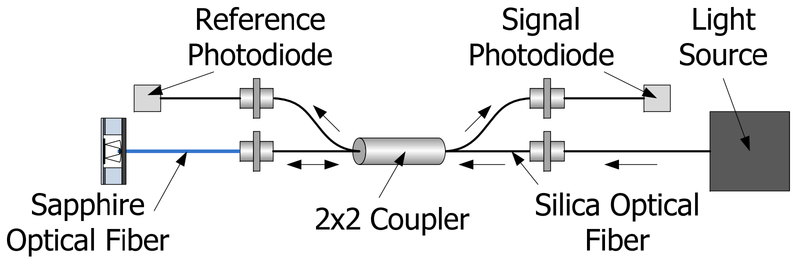
	Silicon Carbide	Diamond	Sapphire
Transparency	✓	X	✓
Bulk substrate availability	✓	X	✓
Optical fiber availability	X	X	✓
Minimal film stress	X	X	✓
Well-established μ-machining processes	X	X	X

- Benefits of sapphire
  - High melting point (2053 °C)
  - Resistance to chemical corrosion
  - Excellent hardness
  - Large transmission window (200 nm – 5 μm)
  - Multimode optical fibers available

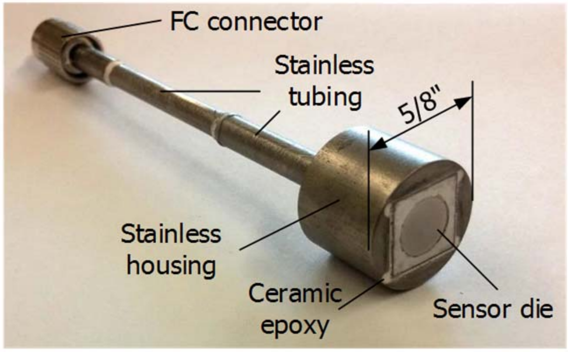
Proof-of-Concept Device (UF)

- Diaphragm
  - 8 mm diameter, 50 μm thick
  - Platinum reflective surface
- Configuration
  - Single send/receiver fiber
  - Sapphire/silica fiber connection
  - Reference photodiode



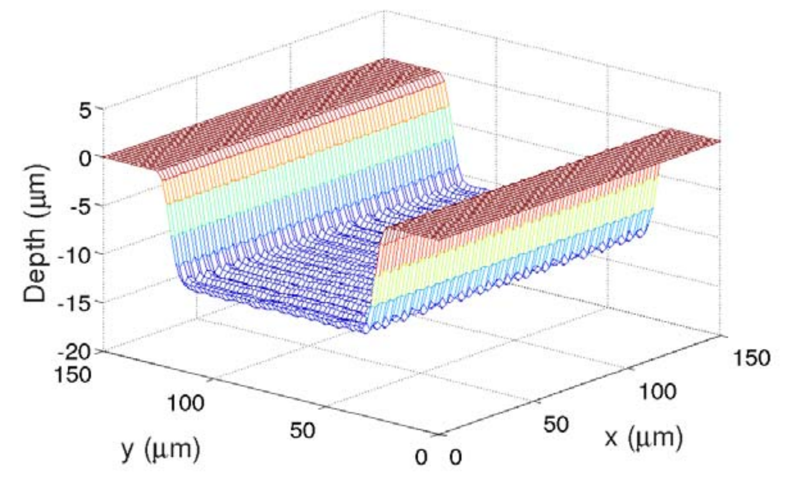


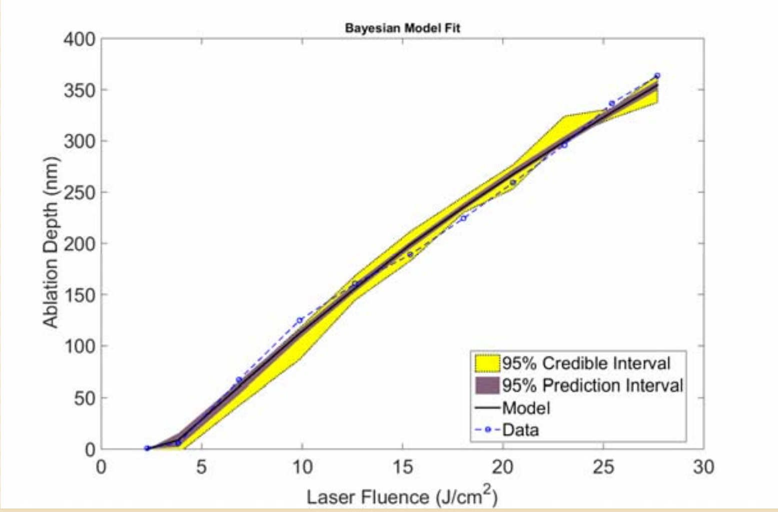
D. Mills et al, *Proc. SPIE*, vol. 9113, Apr 2014



Achievements

- Quantified structure-properties relations in laser machined sapphire
    - Enhanced fracture resistance and nominal strength increase
    - Dislocation formation and amorphous material structure
- Model formulation
    - Continuum light-matter predictions and uncertainty quantification of sapphire laser ablation
    - Finite element estimation of single crystal nanoindentation in sapphire
- Laser micromachining
    - Model prediction matches experimental data
    - Higher fluence and number of cut passes reduces sidewall angle
    - Increasing number of passes improves uniformity





Laser Ablation Modeling and Uncertainty Analysis

Laser Ablation Modeling

- Quantified key material physics associated with pulsed picosecond laser ablation of sapphire
- Maxwell equations coupled to time-dependent internal state electronic structure variable
  - Gaussian laser pulse excitation evaluated over a range of intensities
  - Electromagnetics coupled to rate dependent electronic material excitation

$$L = L_F + L_I + L_M$$

Lagrangian of free space (L_F), electromagnetic interaction energy (L_I), and material energy (L_M)

$$\Pi_D = -\sum \frac{1}{2} \beta^{\alpha} \dot{y}_i^{\alpha} \dot{y}_i^{\alpha}$$

Entropy generation function, β^{α}– inverse mobility, $\dot{y}_i^{\alpha}$ – rate of change of electronic material structure

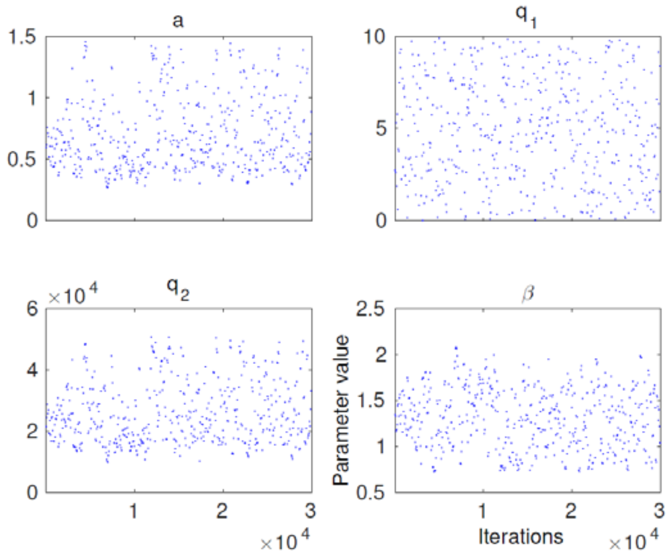
- Minimization of Lagrangian and entropy generation leads to balance equations

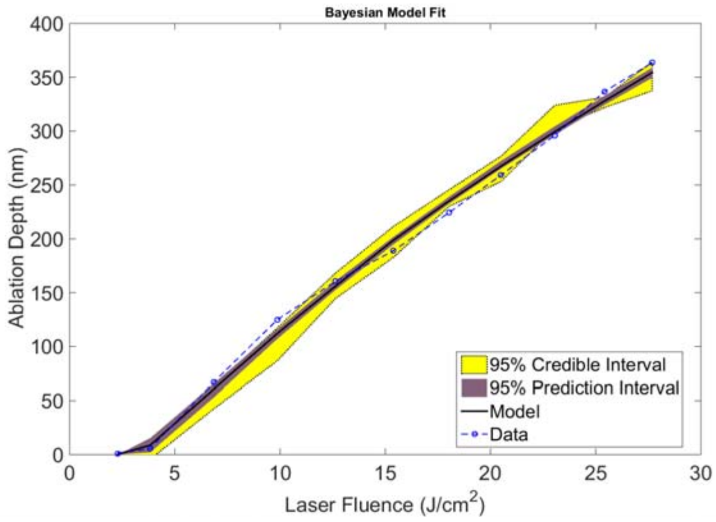
Numerical Implementation

- Balance equation for electronic structure
  - Phase field type equation→sharp interface limit
  - Tracks irreversible laser ablation on sapphire material surface
- Finite difference formulation
  - Error convergence of spatial and time discretization conducted
- Interaction energy within Lagrangian
  - Two light-matter constitutive models formulated
  - Robust model estimates of excited state model parameters
    - Quantified correlations between electron density of sapphire and complex permittivity governing light absorption

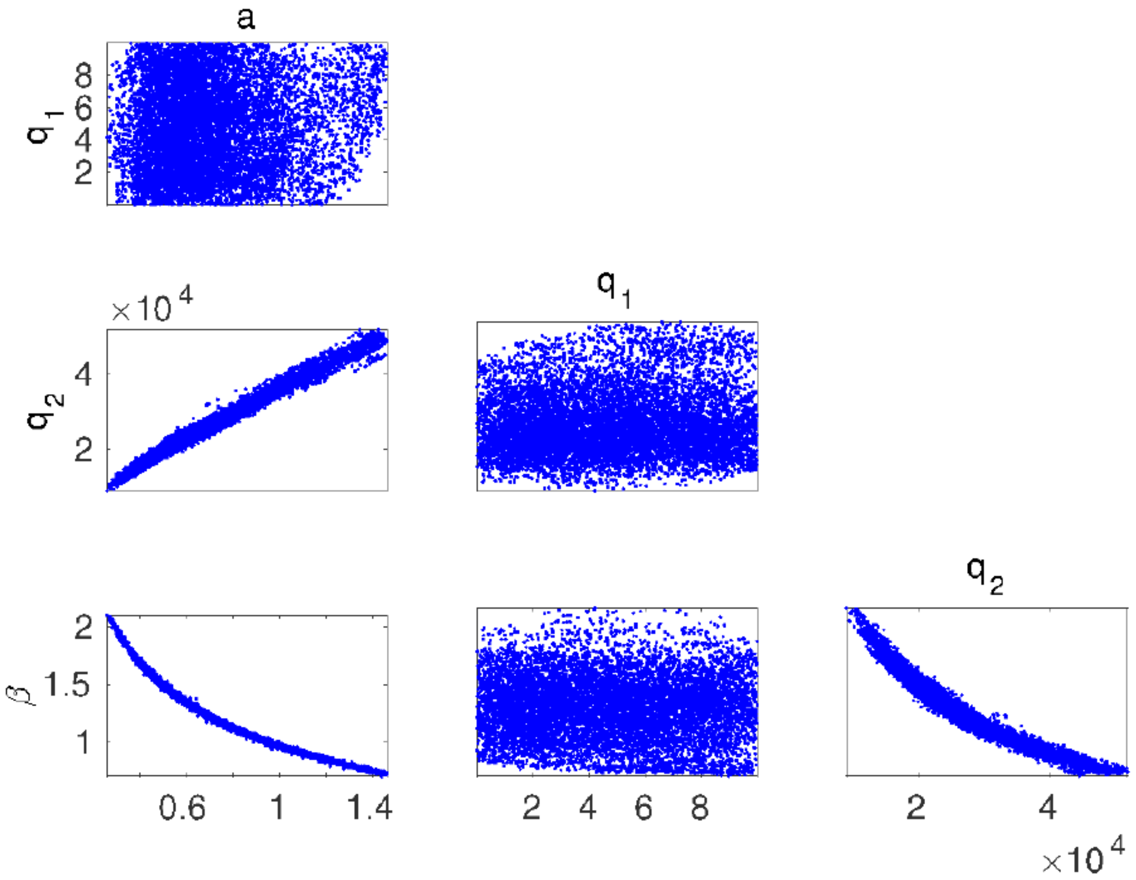
Uncertainty Quantification (UQ)

- Bayesian UQ used to validate the model in light of data
    - Markov Chain Monte Carlo (MCMC) numerically implemented using delayed rejection adaptive Metropolis (DRAM)
- 1D model approximation due to computational limits
    - Model sampled 3×10^4 times to achieve converged posterior densities





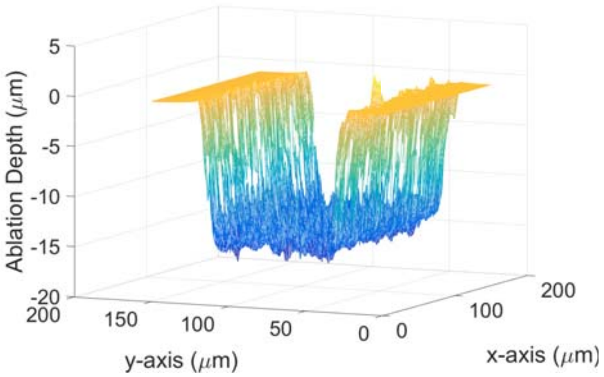
Parameter Correlation



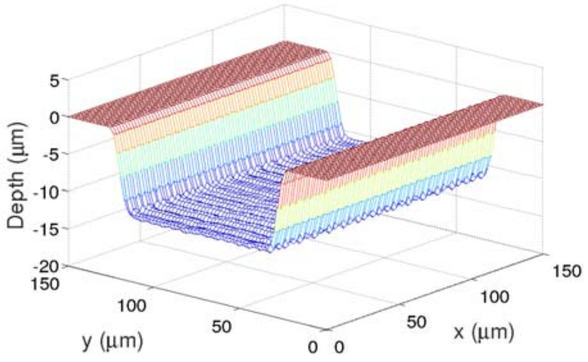
Three Dimensional Extrapolation

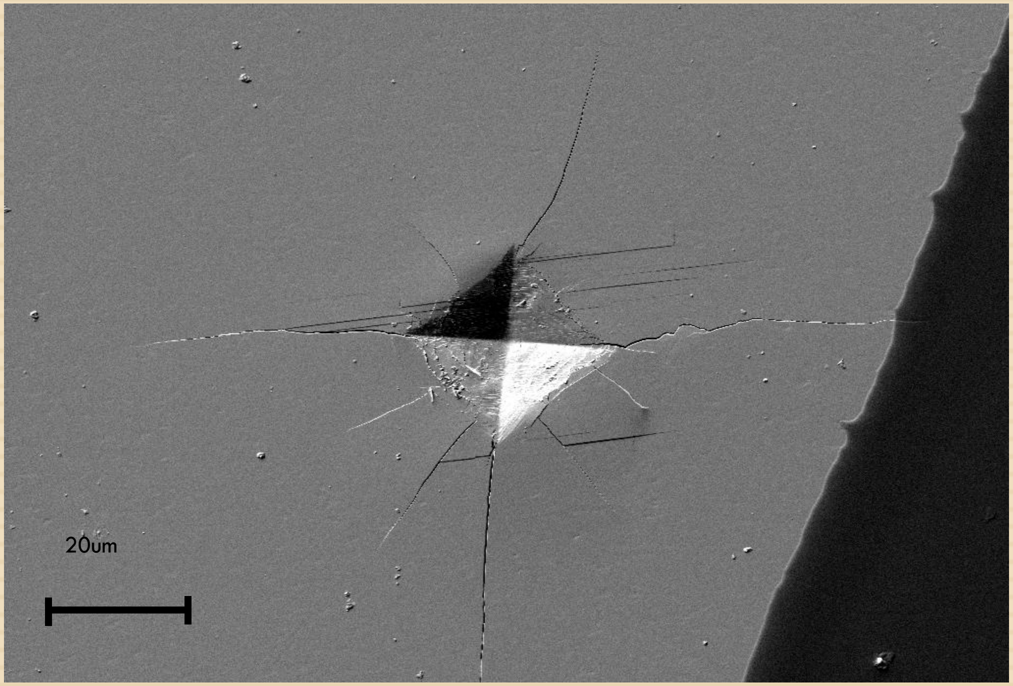
- One dimensional model calibration extrapolated to 3D
  - Reasonable correlation with data
  - Electronic relaxation: τ~4 fs
- Sensitivity and error analysis
  - Parameter sensitivity critical for 3D predictions
  - Average experimental depth of ablation: 13.8 μm
  - Average simulated depth of ablation: 11.3 μm

Experimental Ablation Surface



Simulation of Ablation

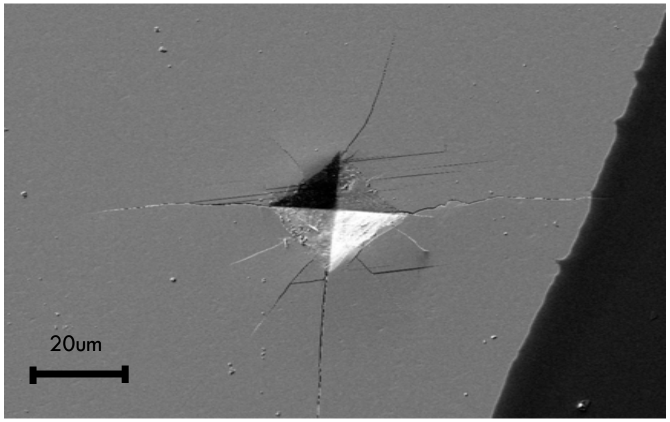


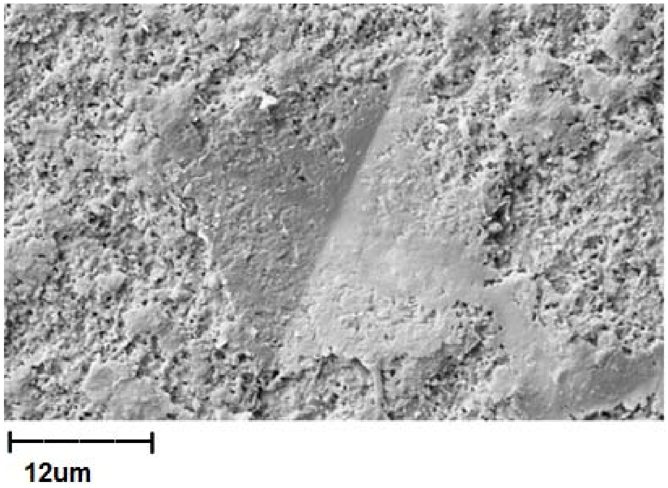


Micro and Nanoindentation Mechanics

Laser Ablated Fracture Resistance

- Enhanced toughness in laser machined sapphire
  - No observable cracks from microindentation
- Prior nanoindentations (UF group) illustrate differences in force-displacement curves
- Dislocations induced by laser ablation process
  - Confirmed from transmission electron microscopy (FSU group)





Model Development

- Solid mechanics of sapphire coupled to single crystal dislocation slip system model
- Kinematics broken into elastic (**H**^{e}) and plastic (**H**^{p}) components

$$\nabla \mathbf{u} = \mathbf{H}^e + \mathbf{H}^p$$

- Plastic strain dependent upon a set of single crystal slip systems (α)

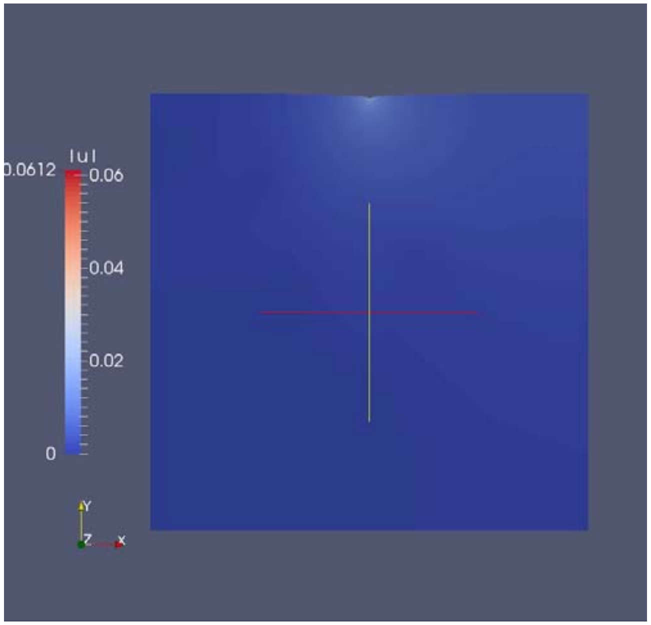
$$\mathbf{H}^p = \sum_\alpha \gamma^\alpha \mathbf{m}^\alpha \mathbf{s}^\alpha$$

- Time evolution equation for slip magnitude

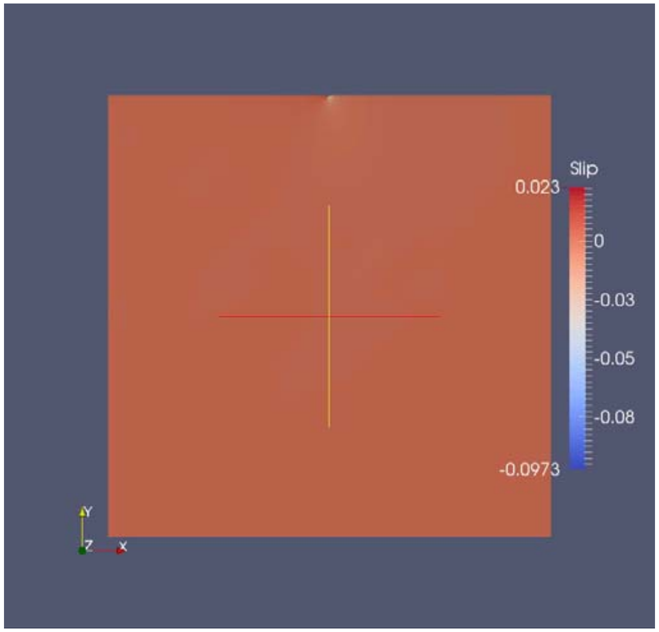
$$\dot{\gamma}^\alpha = A\mathbf{m}^\alpha \cdot \boldsymbol{\sigma} \cdot \mathbf{s}^\alpha$$

- Finite element model implemented in FEniCS
  - Penalty method introduced to accommodate nanoidentation contact mechanics

Results



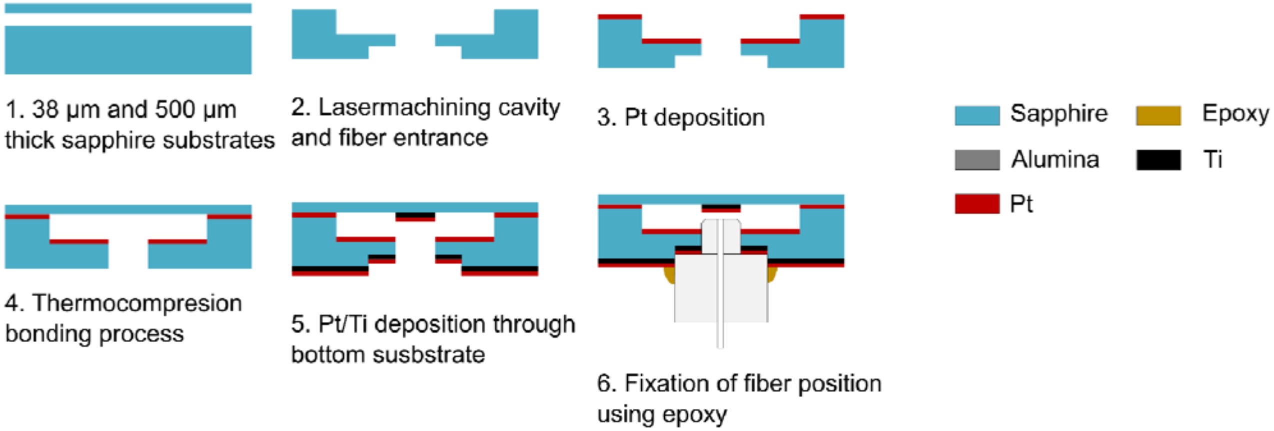
Displacement Field



Slip magnitude

Sensor Fabrication

Sensor Fabrication – Process Flow



1. 38 µm and 500 µm thick sapphire substrates

2. Lasermachining cavity and fiber entrance

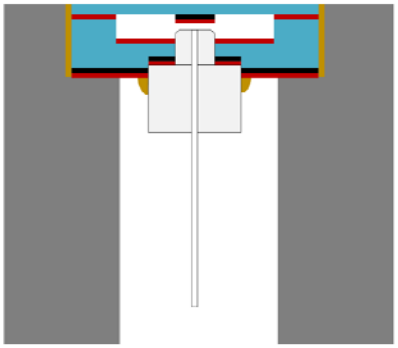
3. Pt deposition

4. Thermocompresion bonding process

5. Pt/Ti deposition through bottom susbstrate

6. Fixation of fiber position using epoxy

→ Issue 1:
In-house laser machining tool was down for most of the year
→ Solution 1:
Externally contracted the laser machining of cavity substrate

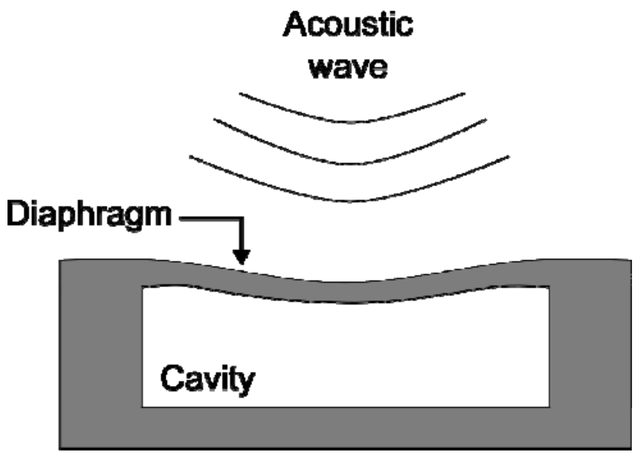


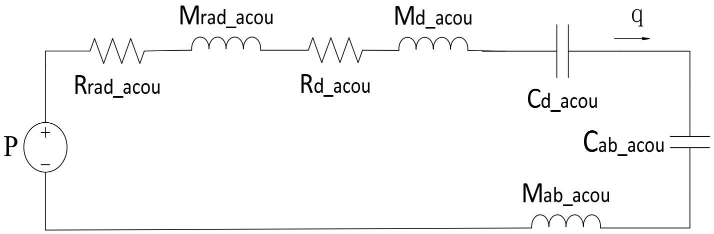
7. Packaging of the sensor

→ Issue 2:
Thermocompression bonding tool is down
→ Solution 2:
Use of ceramic epoxy for bonding the two substrates together

Sensor Fabrication – Mechanical Sensitivity Optimization

- Aim: optimize diaphragm diameter for best acousto-mechanical sensitivity
- Using lumped element modeling:





Sensor Fabrication – Mechanical Sensitivity Optimization

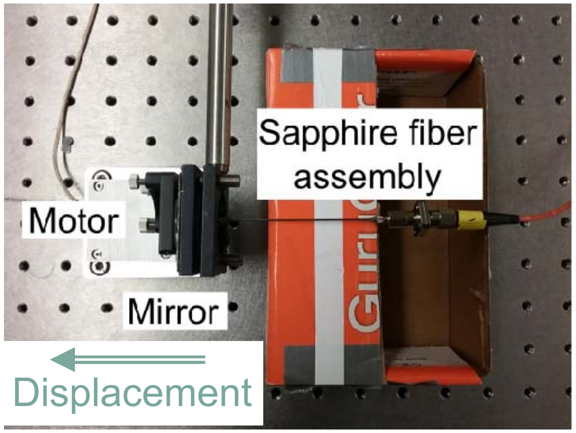
- Aim: optimize diaphragm diameter for best acousto-mechanical sensitivity
- Using lumped element modeling
- Assuming 200 kPa_{max}, and a 38 ± 22 μm thick substrate, f_{mincon} solutions:

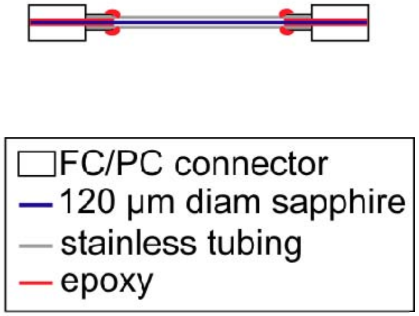
Thickness (μm)	Diameter (mm)	Flat-band Sensitivity (nm/Pa)	Maximum deflection (μm)	Resonant Frequency (kHz)
60	5.4	0.12	24.5	31.3
50	4.4	0.093	18.7	38.1
38	3.4	0.076	15.1	46.0
16	1.4	0.029	5.8	90.7

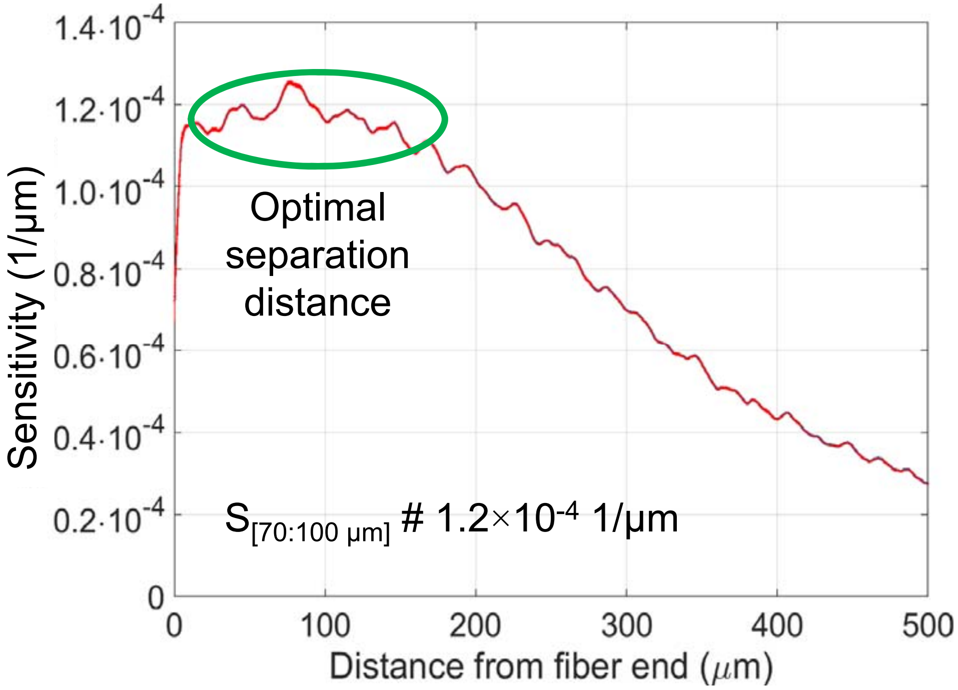
Resulting diameter = 5 mm

Sensor Fabrication – Optical Sensitivity Optimization

- Aim: Find the distance between end of fiber and reflective Pt layer for linear optical response and optimal sensitivity







Sensor Fabrication – Membrane/Cavity Substrates

- Laser machining of trenches in both substrates for improved adhesion
- Next step: using alumina ceramic, bond the two wafers together

3. Lasermachining of trenches in both substrates

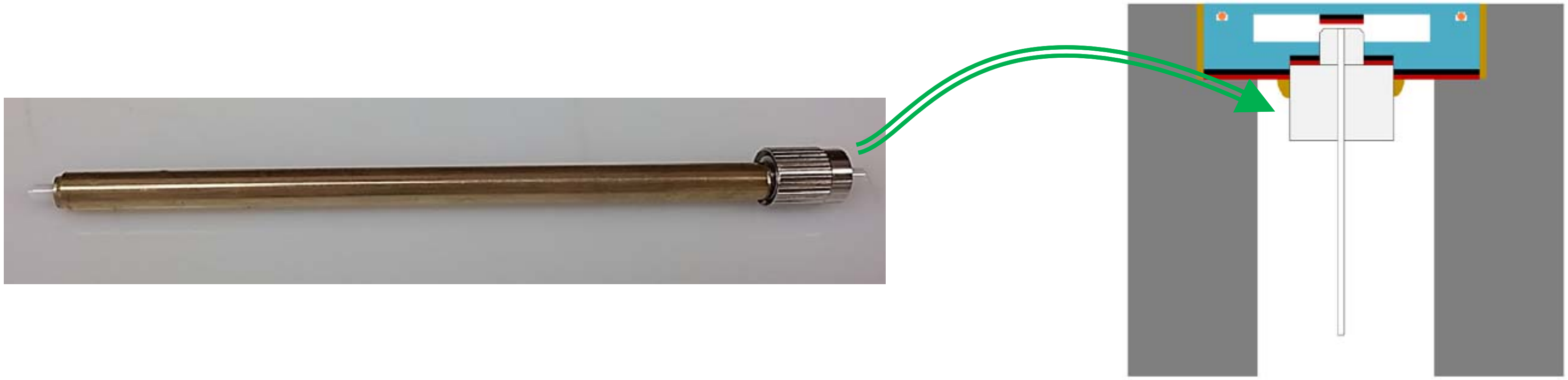
Zoom in (x10)

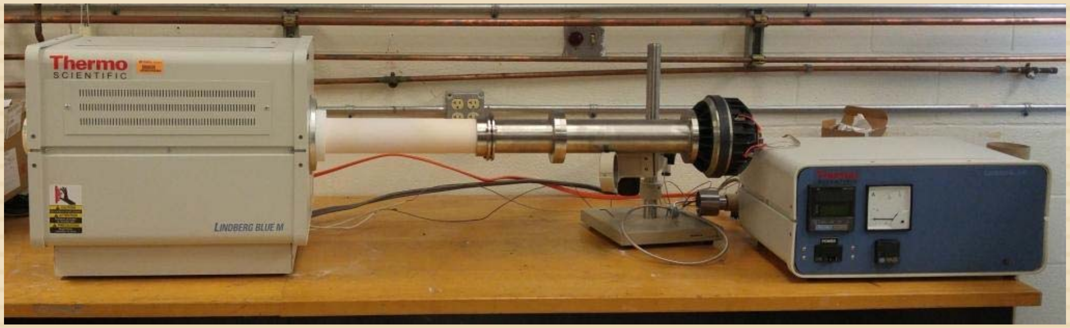
Cavity Substrate

Membrane Substrate

Sensor Fabrication – Optic Fiber Structure

- Sapphire optic fiber was mounted on:
  - Stepped ferule
  - FC connector
  - Brass tubing for rigidity
    - In the future, to be replaced by alumina

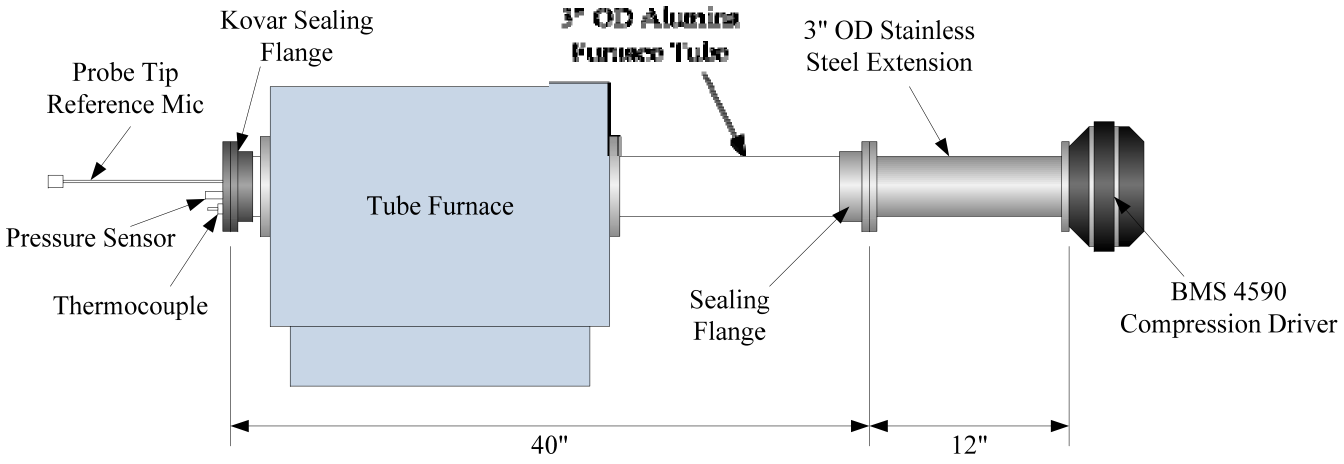




Acoustic Characterization

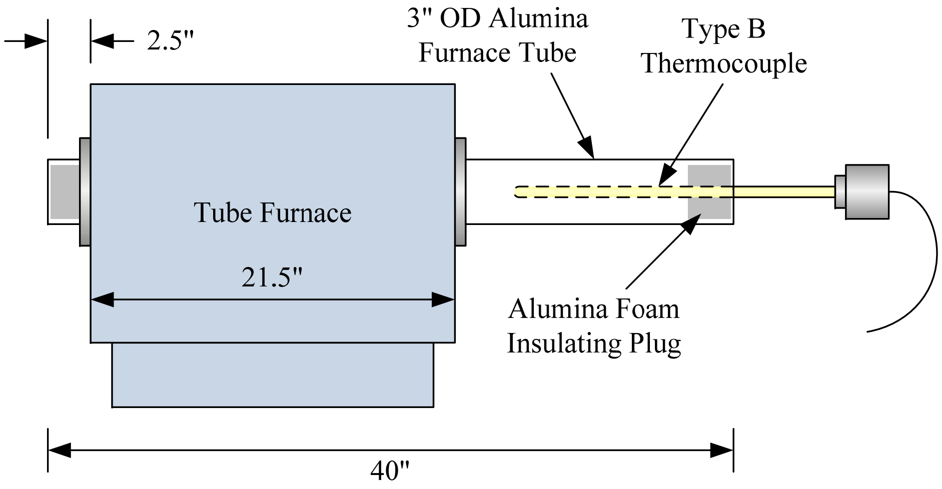
High Temperature Testing Facility

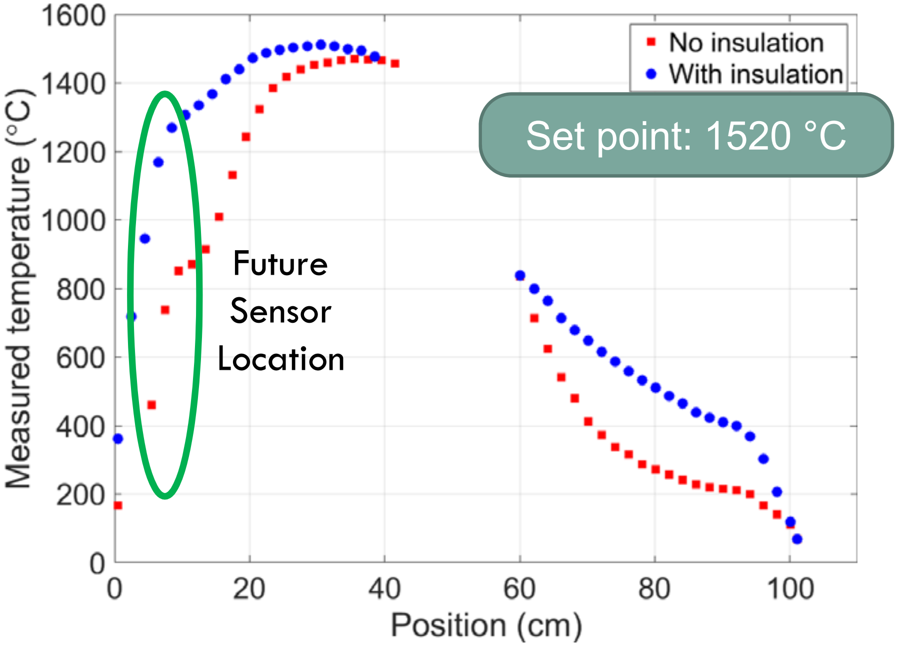
- Plane Wave Tube (PWT) for acoustic characterization
  - Speaker generates acoustic pressure waves
  - Propagate as plane acoustic waves through the tube furnace
  - Option: tube furnace ON ➔ high temperature capability
  - Pressure sensor characterized *in situ*



Step 1: Characterization of Temperature

- ☐ Aim: acoustic characterization up to 1200 °C
- ☐ Measurement of temperature along the PWT
- ☐ Added insulation to prevent thermal leak

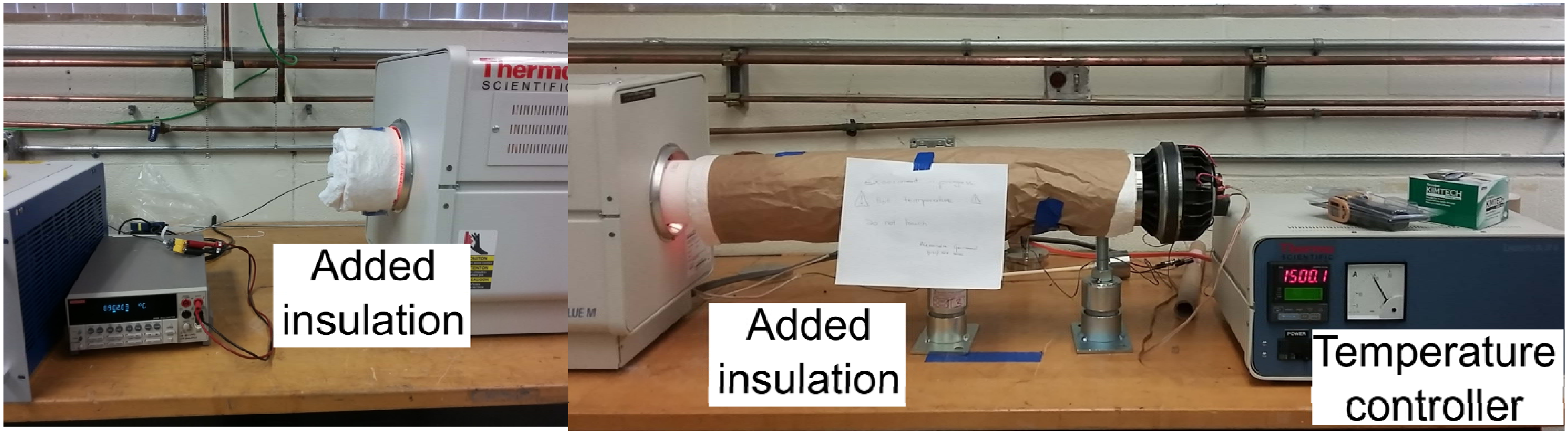




Set point: 1520 °C

Step 1: Characterization of Temperature

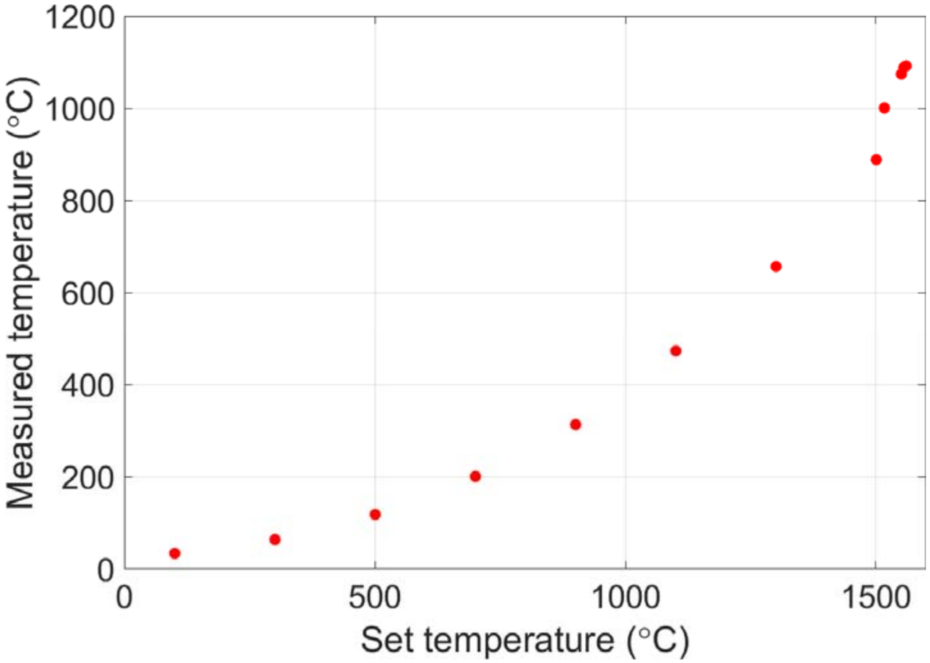
- ☐ Aim: acoustic characterization up to 1200 °C
- ☐ Measurement of temperature along the PWT
- ☐ Added insulation to prevent thermal leak



Step 1: Characterization of Temperature

☐ Aim: acoustic characterization up to 1200 °C

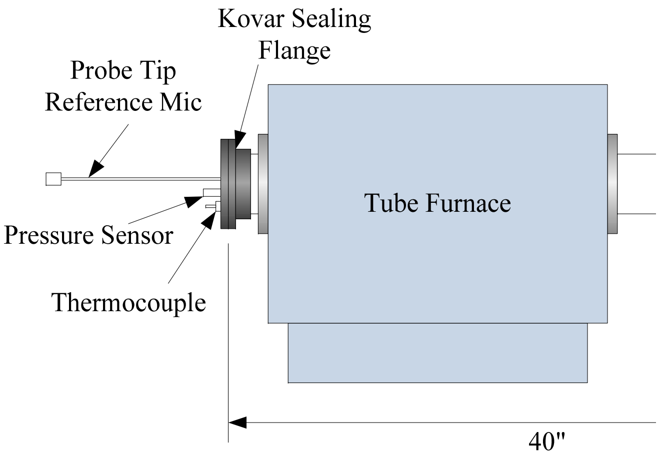
☐ Measurement of temperature at the future sensor location

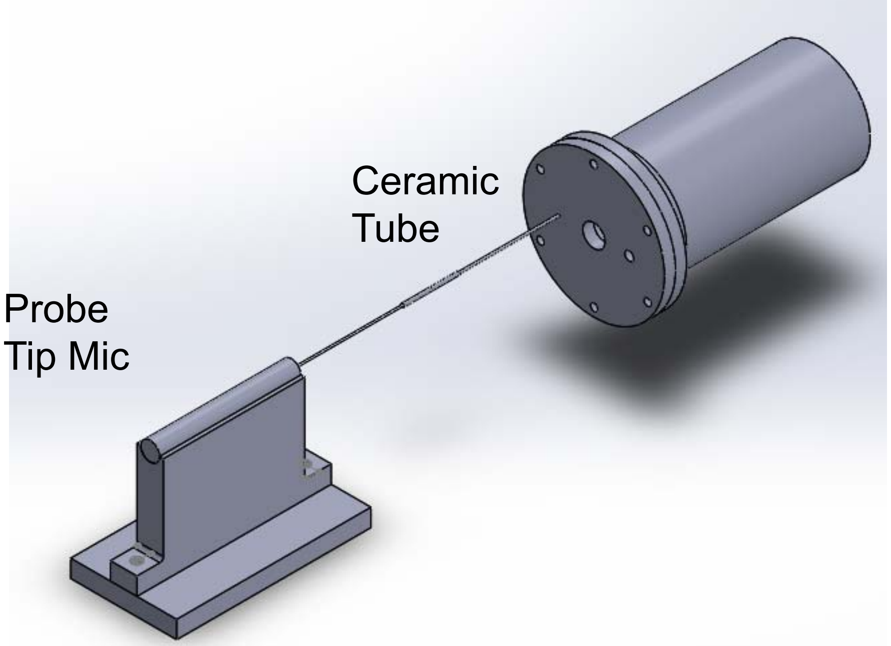


Highest measured temperature: 1092 °C

Step 2: Acoustic Characterization

- Aim: Acoustic characterization at high temperatures
- Issue: the acoustic response varies with temperature
- Solution: use of a remote reference microphone with probe tip





Conclusions – Future work

Conclusions

- Laser Ablation Modeling
  - Successfully predicted ps laser ablation in 1D and extrapolated 3D to simulate milling
  - Developed a 2D model of elastic+slip mechanics for nano-indentation in sapphire
- Sensor Fabrication
  - Determined the separation between fiber and reflective layer for optimal optical sensitivity
  - Designed cavity and membrane substrates for optimal acousto-mechanical sensitivity
  - Initiated sensor fabrication
- Acoustic Characterization
  - Optimized high temperature plane wave tube setup to reach 1100 °C at sensor location
  - Designed mounting support to hold all the equipment necessary for characterization
    - Thermocouple, remote reference microphone with probe tip, sensor

Future Work

- Laser Ablation Modeling
  - Inferring material property changes in laser machined sapphire via nanoindentation and FEA
  - Developing UQ tools and planning x-ray measurements to understand laser induced fracture properties
- Sensor Fabrication
  - Finish fabrication, package and demonstrate proof of concept at room temperature
- Acoustic Characterization
  - Calibrate PWT acoustic response at high temperatures
  - Demonstrate sensor capabilities at elevated temperatures

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