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

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

- **Pro**
  - Simple/robust fabrication
  - Incoherent source
  - Single or multimode fibers

- **Con**
  - Lower sensitivity

<table>
<thead>
<tr>
<th></th>
<th>Capacitive</th>
<th>Piezoresistive</th>
<th>Optical</th>
</tr>
</thead>
<tbody>
<tr>
<td>Thermal drift</td>
<td>✓</td>
<td>×</td>
<td>✓</td>
</tr>
<tr>
<td>DC measurement</td>
<td>✓</td>
<td>✓</td>
<td>✓</td>
</tr>
<tr>
<td>EMI insensitivity</td>
<td>×</td>
<td>×</td>
<td>✓</td>
</tr>
<tr>
<td>Harsh environment capability (&gt;500 °C)</td>
<td>×</td>
<td>×</td>
<td>✓</td>
</tr>
<tr>
<td>Packaging simplicity</td>
<td>✓</td>
<td>✓</td>
<td>×</td>
</tr>
</tbody>
</table>
Material Selection

- 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

<table>
<thead>
<tr>
<th></th>
<th>Silicon Carbide</th>
<th>Diamond</th>
<th>Sapphire</th>
</tr>
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<tbody>
<tr>
<td>Transparency</td>
<td>✓</td>
<td>×</td>
<td>✓</td>
</tr>
<tr>
<td>Bulk substrate availability</td>
<td>✓</td>
<td>×</td>
<td>✓</td>
</tr>
<tr>
<td>Optical fiber availability</td>
<td>×</td>
<td>×</td>
<td>✓</td>
</tr>
<tr>
<td>Minimal film stress</td>
<td>×</td>
<td>×</td>
<td>✓</td>
</tr>
<tr>
<td>Well-established μ-machining processes</td>
<td>×</td>
<td>×</td>
<td>×</td>
</tr>
</tbody>
</table>

- Transparency
- Bulk substrate availability
- Optical fiber availability
- Minimal film stress
- Well-established μ-machining processes
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

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
    \[
    L = L_F + L_I + L_M
    \]
    \[
    \Pi_D = -\sum \frac{1}{2} \beta^\alpha \dot{y}_i^\alpha \dot{y}_i^\alpha
    \]
    Lagrangian of free space \(L_F\), electromagnetic interaction energy \(L_I\), and material energy \(L_M\)
    Entropy generation function, \(\beta^\alpha\) – 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 $3 \times 10^4$ times to achieve converged posterior densities
Parameter Correlation
Three Dimensional Extrapolation

- One dimensional model calibration extrapolated to 3D
  - Reasonable correlation with data
  - Electronic relaxation: $\tau \sim 4$ fs
- Sensitivity and error analysis
  - Parameter sensitivity critical for 3D predictions
  - Average experimental depth of ablation: 13.8 $\mu$m
  - Average simulated depth of ablation: 11.3 $\mu$m
Micro and Nanoindentation Mechanics
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
  \[ \nabla u = H^e + H^p \]
- Plastic strain dependent upon a set of single crystal slip systems ($\alpha$)
  \[ H^p = \sum_\alpha \gamma^\alpha m^\alpha s^\alpha \]
- Time evolution equation for slip magnitude
  \[ \dot{\gamma}^\alpha = A m^\alpha \cdot \sigma \cdot s^\alpha \]
- Finite element model implemented in FEniCS
  - Penalty method introduced to accommodate nanoidentation contact mechanics
Results

Displacement Field

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

→ 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 $200 \text{kPa}_{\text{max}}$, and a $38 \pm 22 \mu m$ thick substrate, $f_{\text{mincon}}$ solutions:

<table>
<thead>
<tr>
<th>Thickness ($\mu m$)</th>
<th>Diameter (mm)</th>
<th>Flat-band Sensitivity (nm/Pa)</th>
<th>Maximum deflection ($\mu m$)</th>
<th>Resonant Frequency (kHz)</th>
</tr>
</thead>
<tbody>
<tr>
<td>60</td>
<td>5.4</td>
<td>0.12</td>
<td>24.5</td>
<td>31.3</td>
</tr>
<tr>
<td>50</td>
<td>4.4</td>
<td>0.093</td>
<td>18.7</td>
<td>38.1</td>
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<tr>
<td>38</td>
<td>3.4</td>
<td>0.076</td>
<td>15.1</td>
<td>46.0</td>
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<tr>
<td>16</td>
<td>1.4</td>
<td>0.029</td>
<td>5.8</td>
<td>90.7</td>
</tr>
</tbody>
</table>

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.

![Graph showing sensitivity vs. distance](image)

- **Optimal separation distance**

- **Sensitivity** \( S_{[70:100 \, \mu m]} \approx 1.2 \times 10^{-4} \, 1/\mu m \)
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
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**

![Diagram showing temperature measurement setup with a tube furnace, 3" OD Alumina Furnace Tube, Type B Thermocouple, Alumina Foam Insulating Plug, and graph showing measured temperature vs. position with no insulation and with insulation. Set point: 1520 °C.](image)
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

- **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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For Harsh Environments

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