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 (1.5 year NCE) started Jan 2014

- **Project team**
  - University of Florida
  - Florida State University
Outline

- Introduction
- Thermal Damage Modeling (FSU)
- Sensor Fabrication (UF)
- Acoustic Characterization (UF)
Motivation

- Next generation advanced energy systems will require harsh environment dynamic instrumentation:
  - Process control/closed loop feedback
  - Increase efficiency, reduce emissions & cost
- Sensor operational requirements
  - Temperature: >1000 °C and dynamic pressure: up to 1000 psi, 10s kHz
  - Atmosphere: corrosive and/or erosive
- Conventional pressure sensor instrumentation limited to ~500 °C
  - Temperature mitigation techniques: stand-off tubes, water cooling
  - Oxsensis claims 750 °C using sapphire interferometry technique
Technical Objectives

- **Novel sapphire fabrication processes**
  - Subtractive machining: ultrashort pulse laser micromachining
  - Additive manufacturing: thermocompression bonding via spark plasma sintering

- **Modeling and characterization of laser machined sapphire**
  - High temperature experimental characterization, modeling, and Bayesian uncertainty quantification

- **Fabricate, package, calibrate, and demonstrate sapphire optical pressure sensor**
Previous Work

- **Sapphire fabrication processes**
  - Developed empirical based path planning simulation for pico-second laser micromachining
  - Proved concept of thermal compression bonding via spark plasma sintering

- **Modeling and characterization of laser machined sapphire**
  - High temperature (1500°C) bend bar characterization
  - Light-matter ablation physics and Bayesian uncertainty quantification

- **Fabricated various pressure sensor components**
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Nanoindentation analysis

Nanoindentations: Experimental Results

![Graph showing force vs. indenter depth for different samples: Virgin 1, Virgin 2, Virgin 3, Virgin 4, Laser 1, Laser 2, Laser 3, Laser 4. The graph plots force in nN on the y-axis against indenter depth in nm on the x-axis. Each sample type is represented by a different line style and color.](image-url)
Finite Deformation FEA Modeling

Deformation Decomposition
\[ \mathbf{F} = \mathbf{F}^p \cdot \mathbf{F}^e \]

Stress-Strain Relation
\[ \mathbf{S} = \mathbf{C} : \mathbf{E}^e \]
- \( \mathbf{C} \) - Isotropic elastic moduli

Yield Stress Evolution
\[ Y(e^p) = Y_0 + H_{iso}e^p \]

Assume plasticity law with hardening
- Amorphous zone near surface
- No distinct dislocation pattern

Key Results

Virgin Sapphire

Laser Machined Sapphire
## Plastic Hardening Parameter Relations

<table>
<thead>
<tr>
<th>Experiment</th>
<th>Linear Hardening Parameter (MPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Virgin 1</td>
<td>60</td>
</tr>
<tr>
<td>Laser Machine 1</td>
<td>28</td>
</tr>
<tr>
<td>Laser Machine 2</td>
<td>19</td>
</tr>
<tr>
<td>Laser Machine 3</td>
<td>46</td>
</tr>
<tr>
<td>Laser Machine 4</td>
<td>37</td>
</tr>
</tbody>
</table>

Model fits apply $E = 450$ GPa, $Yield Stress = 1$ MPa
Sapphire Crystal Structure

- Specimens cut with r-plane normal to surface.
  - $\phi_0$: angle between specimen normal and r-plane surface normal
  - Assumes uncertainty in crystal cut & x-ray measurements
- X-ray diffraction scans were done in the pristine and laser machined regions
  - Both normal and in-plane measurements were taken

Figure 1: Sapphire crystal structure

Figure 2: Specimen geometry
X-Ray Diffraction

Figure 3: Diffraction of x-rays by planes of atoms.

\[ d_{hkl} = \frac{\lambda}{2 \sin \theta} \]
Normal (r-plane) Peak Comparisons
X-Ray Kinematic Model

\[ I = |F_{hkl}|^2 = \left( \sum_{i=1}^{N} f_i e^{2\pi i (P_j x_i \cdot R(\phi_0))} \right)^2 \]

- \( f_i \): Scattering factor
- \( P_j \): Miller indices (hkl) of plane of interest j
- \( x_i \): Cartesian coordinates of atom i
- \( R(\phi_0) \): Rotation Matrix dependent on \( \phi_0 \)
- \( N \): number of atoms

\[ I(\theta) = |F_{hkl}|^2 G(\theta) = I_{\text{scale}}|F_{hkl}|^2 e^{-\sqrt{2\pi}(\theta-\theta_b)^2/2\sigma^2} \]

- \( \theta \): Angle of incident x-ray
- \( \theta_b \): Angle at which maximum intensity occurs (Bragg Angle)
- \( \sigma \): Standard deviation of peak about \( \theta_b \)
- \( I_{\text{scale}} \): Scaling factor

Parameter uncertainty quantified using Bayesian statistics
- Markov Chain Monte Carlo algorithm
- Delayed Rejection Adaptive Metropolis (DRAM)
Spectra Predictions Normal (012) to R-Plane (Pristine Sapphire)

- Normal (012) to r-plane
- Normal (202) plane
Strain Inference from X-Ray Spectra

\[ \epsilon_{hkl} = \frac{\sin \theta_{\text{pristine}} - \sin \theta_{\text{laser}}}{\sin \theta_{\text{laser}}} \]

Equation to calculate strain in (hkl) plane direction

\[ \epsilon^R = \begin{bmatrix} ? & ? & ? \\ X & ? & ? \\ X & X & \epsilon^R_{33} \end{bmatrix} \]

Strain tensor relative to R coordinate system \((x^R, y^R, z^R)\)

- \(\epsilon^R_{33} = (-1.366 \pm 0.019) \times 10^{-3}\) (90% credible interval)

\[ \epsilon^O = \begin{bmatrix} A^O & C^O & D^O \\ C^O & B^O & E^O \\ D^O & E^O & \epsilon^O_{33} \end{bmatrix} \]

Strain tensor relative to O coordinate system \((x^O, y^O, z^O)\)

- \(\epsilon^O_{33} = (-0.098 \pm 0.922) \times 10^{-3}\) (90% credible interval)
- \(A^O, B^O, C^O, D^O, E^O\) are unknown parameters

If \(\epsilon^O\) is rotated from the O coordinate system to the R then: \(\bar{\epsilon}^O = \epsilon^R\)
Model Assumptions

- Unknown strain parameters inferred using:
  \[
  t_i = \sigma_{ji} n^R_j = 0 \\
  n^R = i_z \\
  \sigma_{ij} = C_{ijkl} \left( \varepsilon_{kl}^{Pristine} - \varepsilon_{kl} \right) \approx C_{ijkl} \varepsilon_{kl}
  \]

Assumptions:
1. Pristine reference state has zero residual strain
2. Traction on r-plane is zero
3. \( \varepsilon_{11} = \varepsilon_{22} \) and modulus is isotropic
Residual Strain Estimations

Assuming isotropy ($\varepsilon^R_{11} = \varepsilon^R_{22}$) and the relation $\varepsilon^O_{33} = \varepsilon^R_{33} = \text{const.}$

- $D^O = f(A^O, B^O)$
- $E^O = f(A^O, B^O, C^O)$

Using Bayesian methods coupled with the above relations the strain tensor is determined:

$$\varepsilon^R = \begin{bmatrix} 4.4 & 2.1 & -1.2 \\ 2.1 & 4.4 & -7.6 \\ -1.2 & -7.6 & -1.4 \end{bmatrix} \cdot 10^{-3}$$

Where:

$$\varepsilon^R_{11} = \varepsilon^R_{22} = (4.447 \pm 0.530) \cdot 10^{-3}$$

$$\varepsilon^R_{33} = (-1.366 \pm 0.019) \cdot 10^{-3}$$

*High confidence laser machined zone has in-plane compression which produces higher toughness*
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Optical transduction (intensity modulation – optical lever) is selected given the constraints

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

- **Pro**
  - Incoherent source
  - Thermally stable

- **Con**
  - Lower sensitivity
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
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 $38 \pm 22 \text{ µm}$ thick substrate, $f_{mincon}$ solutions:

<table>
<thead>
<tr>
<th>Thickness ($\mu$m)</th>
<th>Diameter (mm)</th>
<th>Flat-band Sensitivity $S_{AM}$ (n m/Pa)</th>
<th>Flat-band Sensitivity $S_{AM}$ (dB)</th>
<th>Maximum deflection (µm)</th>
<th>Resonating Frequency (kHz)</th>
</tr>
</thead>
<tbody>
<tr>
<td>60</td>
<td>5.4</td>
<td>$1.2 \times 10^{-1}$</td>
<td>-198.2</td>
<td>24.5</td>
<td>31.3</td>
</tr>
<tr>
<td>50</td>
<td>4.4</td>
<td>$9.3 \times 10^{-2}$</td>
<td>-200.6</td>
<td>18.7</td>
<td>38.1</td>
</tr>
<tr>
<td>38</td>
<td>3.4</td>
<td>$7.6 \times 10^{-2}$</td>
<td>-202.4</td>
<td>15.1</td>
<td>46.0</td>
</tr>
<tr>
<td>16</td>
<td>1.4</td>
<td>$2.9 \times 10^{-2}$</td>
<td>-210.7</td>
<td>5.8</td>
<td>90.7</td>
</tr>
</tbody>
</table>

Design diameter choosen = 5 mm
Sensor Fabrication – Optical Sensitivity Optimization

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

![Diagram of optical sensor setup]

![Graph showing normalized reflected intensity vs distance]

- Optimal separation distance $S = 1.24 \times 10^{-4} \text{ V/(V/μm)}$

- Graph annotations:
  - FC/PC connector
  - 120 μm diam sapphire
  - Stainless tubing
  - Epoxy

- Graph x-axis: Distance from fiber end (μm)
- Graph y-axis: Normalized reflected intensity ($V_{\text{ref}}$)
Sensor Fabrication – Initial Process Flow

→ Issue: Thermocompression bonding tool is down
→ Solution: Use of ceramic epoxy for bonding the two substrates together
1. Diaphragm substrate (8mm) with deposited Ti/Pt film and laser machined corrugation around edge

2. Cavity substrate (8mm) with laser machined cavity (5mm) and optical entry hole

3. Bonding two substrates using ceramic adhesive on the edge trench

4. Installing the sensor into the packaging tube
Sensor Fabrication – Bonding

- Laser machining for both substrates
- Laser machining corrugation for adhesion improvement

Microscope 100X
Sensor Fabrication – Membrane/Cavity Substrates

- Alignment on flip-chip bonder
- Manual application of alumina ceramic adhesive into the edge trench
Sensor Fabrication – Optic Fiber Structure

- Sapphire optic fiber was mounted on:
  - Laser micromachined stepped ferrule
  - FC connector
  - Stainless steel 304 tubing for rigidity
  - High-temperature alumina ceramic adhesive for bonding
Sensor Fabrication – Packaging

- Stainless steel housing
- High-temperature alumina ceramic adhesive for bonding
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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
- **Issue:** measured temperature is too low
- **Solution:** adding insulation to prevent thermal leak

![Diagram of tube furnace with labeled dimensions and thermocouple placement.]

- **Highest measured temperature:** 1092 °C
- **Setpoint:** 1520 °C

![Graph showing the relationship between setpoint temperature and endplate temperature. The graph includes a trend line with data points.]
Step 2: Acoustic Characterization

- High temp PWT test set up
- Sensor (DUT,1), probe tip microphone (reference,2), thermocouple(3)
Step 2: Acoustic Characterization Results

- SPL(90-160dB) Sweep at 1kHz
- Temperature from 23°C (room temp) to 600°C with 100°C step size
- Sensitivity drops dramatically after 500°C
  - Failure?
Step 2: Acoustic Characterization Results

- SPL(90-160dB) sweep at 1kHz
- Temperature from 23°C (room temp) to 600°C with 100°C step size
- Sensitivity drops after 500°C
Step 2: Acoustic Characterization Results

- Frequency response function via multisine acoustic waves (300-2200Hz)
- Temperature from 23°C to 600°C
- FRF magnitude drops after 500°C
Conclusions

- **Material Characterization and Modeling**
  - Changes in plasticity of laser machined sapphire quantified using finite deformation contact mechanics & FEA
  - X-ray diffraction and Bayesian UQ used to identify source of increase in fracture toughness--in-plane compression

- **Sensor Fabrication**
  - Determined the separation between fiber and reflective membrane for optimal sensitivity
  - Designed, fabricated and packaged a prototype sapphire pressure sensor using a ceramic adhesive

- **Acoustic Characterization**
  - Optimized high temperature plane wave tube setup to reach 1100 °C at sensor location
  - Set up all the equipment necessary for characterization
    - Thermocouple, remote reference microphone with probe tip, sensor
  - Characterized sensor up to 500 °C (failure point)
Future Work

- **Additive Manufacturing Process and Mechanics**
  - Develop bonding technology to bond sapphire substrates with and without intermediate layers
  - Experimentally characterize bonding strength and fracture as a function of temperature
  - Develop a fundamental understanding of high temperature (>1000C) fracture mechanics and interfacial material physics
  - Understand laser processing parameters on subsurface material properties to enhance fatigue resistance and additive/subtractive manufacturing

- **Sensor Fabrication**
  - Improve the fabrication and packaging process including thermal compression bonding and packaging sealing

- **Acoustic Characterization**
  - Calibrate PWT acoustic response at high temperatures for a new sensor with improved fabrication and packaging
  - Hot jet test using high temperature sapphire sensor
  - Test long term stability of the sensor
Questions?

Thank you!