COE CST Third Annual Technical Meeting:

High-Temperature Pressure Sensors for Hypersonic Vehicles

David Mills
Mark Sheplak

October 28 – 30, 2013

Center of Excellence for
Commercial Space Transportation

Overview

- Team Members
- Purpose of Task
- Research Methodology
- Results
- Next Steps
- Contact Information

Team Members

- **University of Florida**
  - **Mark Sheplak** – Professor, Dept. of Mechanical and Aerospace Engineering
  - **David Mills** – Graduate Research Assistant
  - **Daniel Blood** – Graduate Research Assistant
- **Florida State University**
  - **William Oates** – Asst. Professor, Dept. of Mechanical Engineering
  - **Justin Collins** – Graduate Research Assistant

Purpose of Task

- Conventional instrumentation is unsuitable for continuous measurement in high-temperature environments such as:
  - High-speed reentry vehicles
  - Hypersonic transports
  - Gas Turbines
  - Scramjets
- Pressure sensors capable of high-temperature operation (>1000°C) will improve understanding of shock-wave/boundary layer interactions which directly influence critical vehicle characteristics such as lift, drag, and propulsion efficiency

Objectives

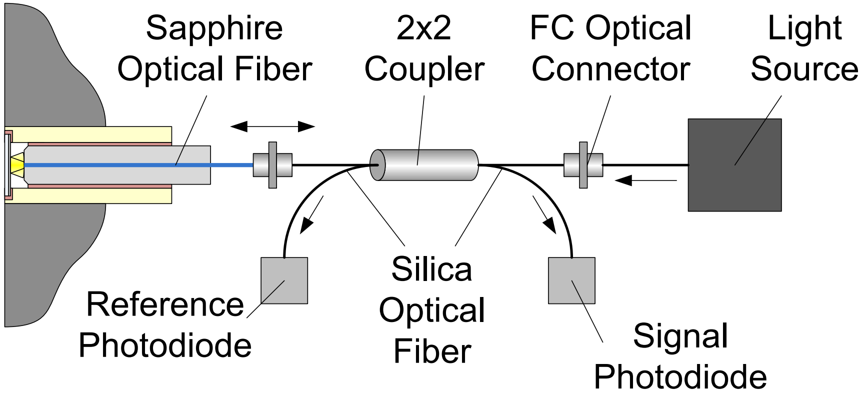
- Identify a suitable sensing method, material, and fabrication process for a high-bandwidth pressure sensor capable of continuous operation in temperatures in excess of 1000°C
- Fabricate a prototype sensor and create a robust high-temperature package
- Characterize the packaged sensor at room temperature and in high-temperature environments
- Implement the packaged sensor in a hypersonic or hot jet flow facility and/or a gas turbine

Research Methodology

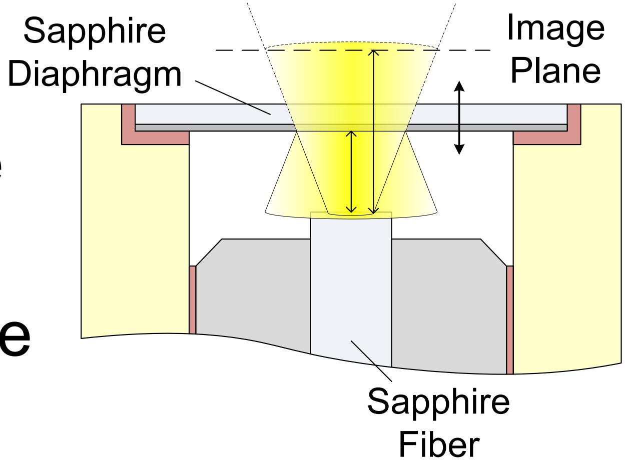
- Sapphire fiber-optic sensors provide the following advantages over traditional silicon-based electrical sensors:
  - Electrically passive
  - Highly chemically inert
  - Immune to EMI
  - Non-conductive
- Requires development of the following processes:
  - Ultra-short pulse laser micromachining
  - Thermocompression bonding via spark plasma sintering (SPS) technology

Research Methodology

- Transduction Method: Fiber-optic lever
  - Intensity modulation via diaphragm deflection
  - Single send/receive fiber

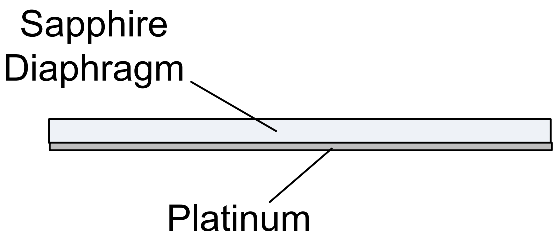


- Optical Configuration
  - LED source with multimode fibers eliminates interferometric effects
  - Silica optical fiber components reduce back-end packaging costs
  - Reference photodiode eliminates noise from source

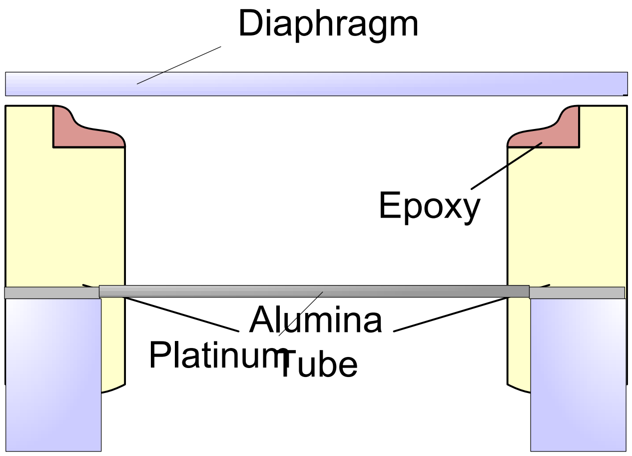


Process Flow

- Initial prototype sensor
  - Machine 4.5 mm diameter diaphragm from 50 μm thick sapphire
  - Deposit 200 nm platinum reflective layer with 20 nm titanium adhesion layer
  - Machine 4.5 mm recess in 3 mm ID alumina tube
  - Epoxy diaphragm inside recess



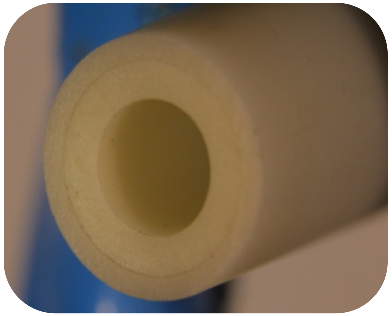
- Bonded prototype sensor
  - Machine 7 mm diameter hole in 1 mm thick sapphire substrate to form back cavity
  - Deposit 500 nm platinum bonding layer on back cavity substrate
  - Align and bond 50 μm sapphire diaphragm to back cavity substrate
  - Deposit 200 nm platinum reflective layer with 20 nm titanium adhesion layer in center

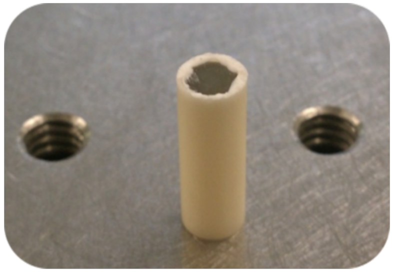


Fabrication Challenges

- Picosecond laser micromachining of sapphire
  - Thermal damage to surrounding material affects material properties and reliability
  - Understand relationship to machining parameters
- Spark plasma sintering (SPS) bonding of sapphire
  - Reduced temperatures and holding time compared to traditional vacuum hot press
  - Understand relationship between bond parameters and bond strength, thermal damage
- High-temperature packaging
  - Provide robust packaging solution while minimizing thermal stress effects

Prototype Fabrication & Packaging

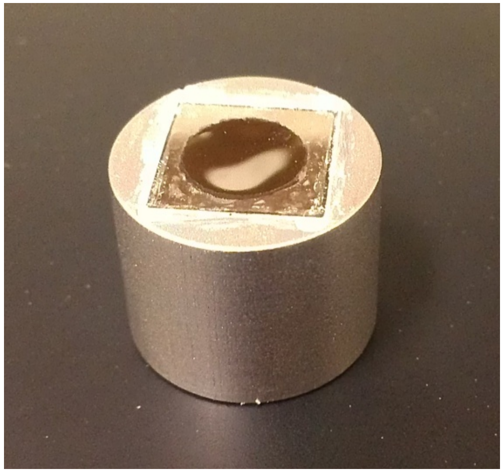




- Developed laser machining processes for alumina and sapphire
- Poor definition of diaphragm shape and boundary condition due to application of epoxy
- Demonstrated method to determine optimal fiber distance from diaphragm
- Stainless steel package capable of 600°C operation

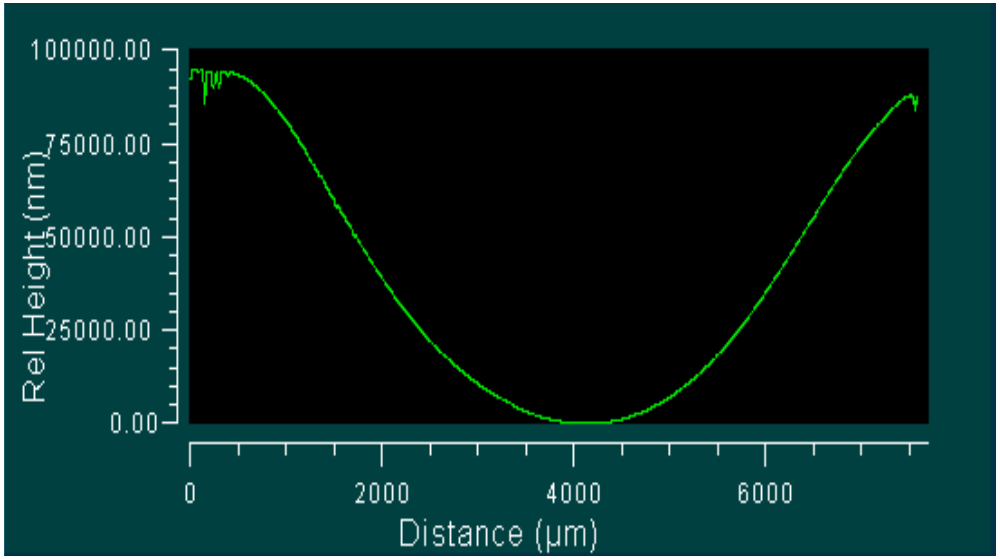
Bonded Sensor Design & Fab

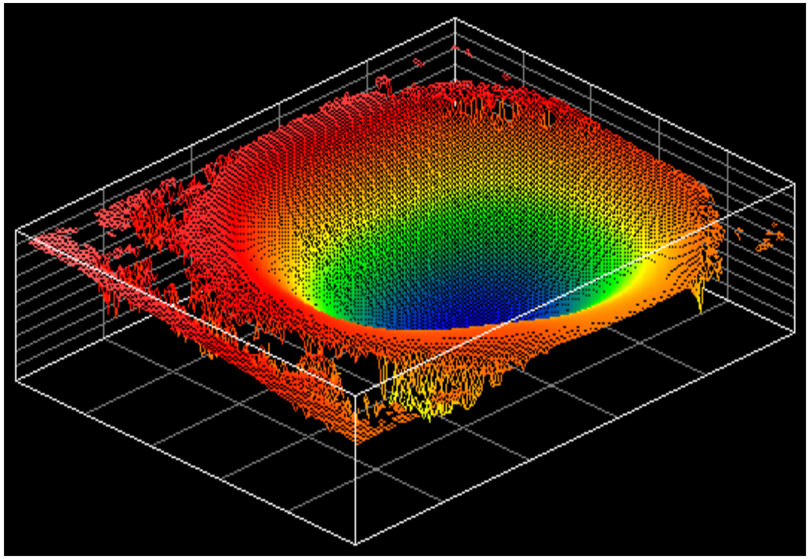
- Larger diameter – improved pressure sensitivity
  - Diaphragm size: 7 mm diameter, 50 μm thick
  - Resonant frequency: 19.6 kHz
  - Mechanical sensitivity: 0.55 nm/Pa
- SPS Bond – better control of boundary
  - Heat/Cool Rate: 50°C/min
  - Temperature: 1200°C
  - Hold Time: 5 min
  - Diaphragm buckled during process



Post-bond Buckling Analysis

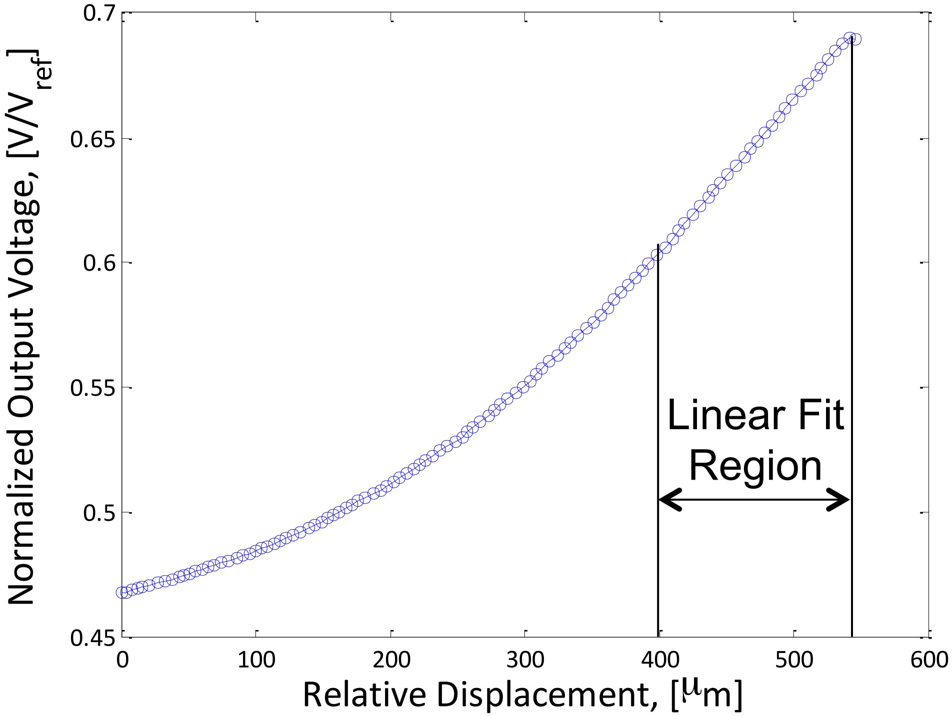
- Buckled diaphragm analyzed using scanning white light interferometer (SWLI)
- Measured center deflection of 90 μm corresponds to ~275 MPa residual compressive stress

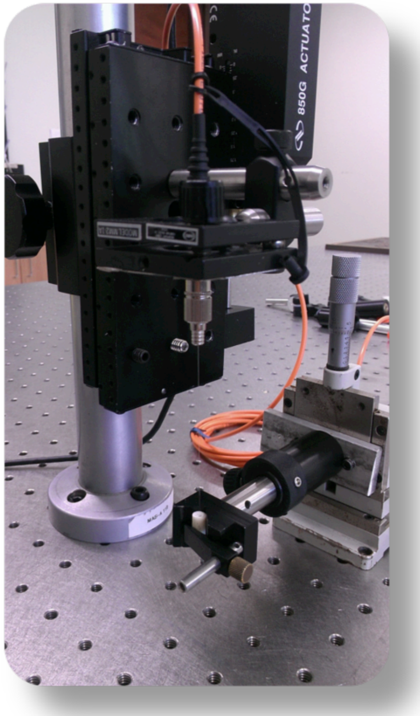




Sensitivity Calibration

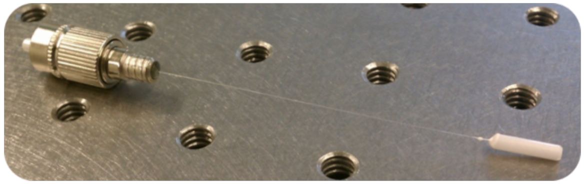
- Optimal distance between fiber and diaphragm determined based on deflection sensitivity
- Polyfit to linear region of normalized output gives a sensitivity of 0.62 mV/V/µm



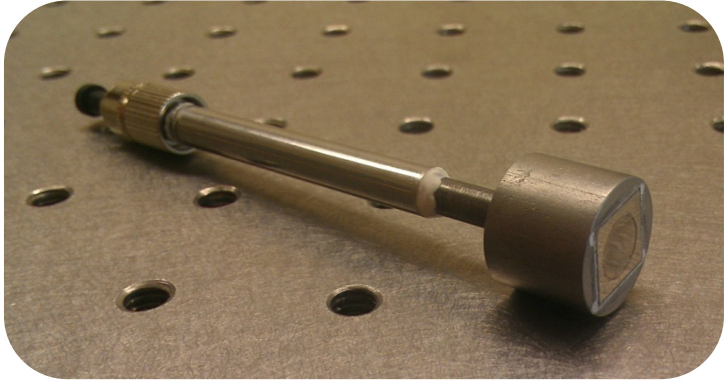


High-Temperature Packaging

- Sapphire optical fiber packaged in FC connector on one end with bare zirconia ferrule on other end

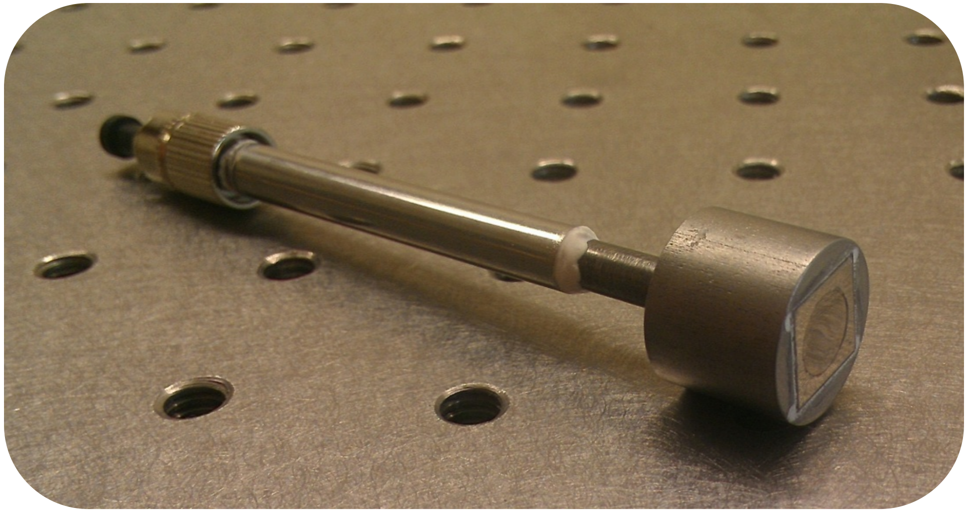


- Zirconia ferrule epoxied into stainless steel housing in position determined by sensitivity calibration
- Stainless steel tubing used to protect sapphire optical fiber attached using high-temp ceramic epoxy



Results

- Measured sensor resonance of 22.1 kHz
- Theoretical sensitivity of 0.12 μV/V/Pa based on estimated sensitivity of buckled diaphragm
- Max continuous operating temperature of 900°C



Next Steps

- Complete SPS bond process development and characterization of bond interface
- Room-temperature plane wave tube characterization
  - Sensitivity
  - Frequency response
  - Linearity
- High-temperature characterization
  - Demonstrate survivability
  - Determine thermal drift
- Testing of the sensor in a high-temperature flow facility or gas turbine

Contact Information

- David Mills – dm82@ufl.edu
- Mark Sheplak – sheplak@ufl.edu

Interdisciplinary
Microsystems
Group
UNIVERSITY OF FLORIDA

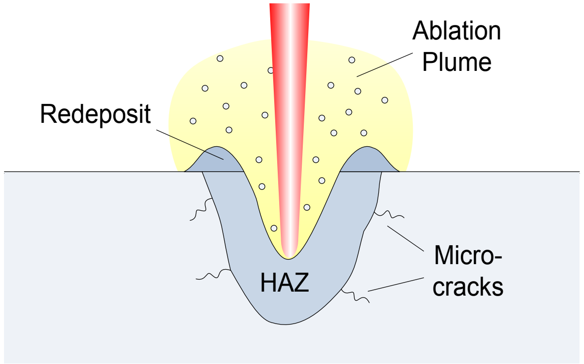
FCAAP
FLORIDA CENTER FOR ADVANCED AERO-PROPULSION

Backup Slides

Interdisciplinary
Microsystems
Group
UNIVERSITY OF FLORIDA

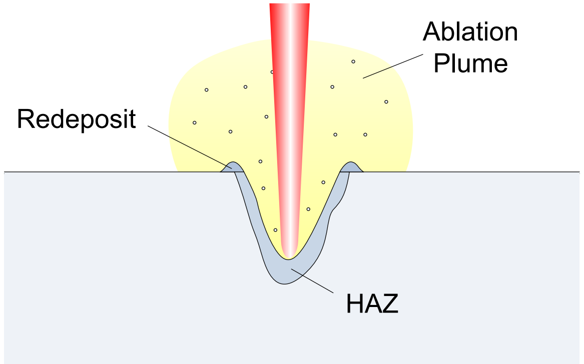
Laser Micromachining

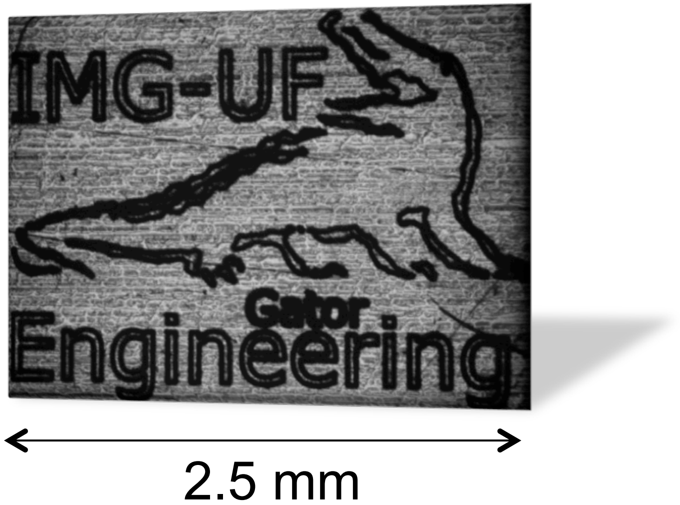
- “Long” Pulsewidths (>10 ps)
  - Industry standard
  - High reliability
  - Large heat affected zone (HAZ)
  - Micro-cracking and redeposit



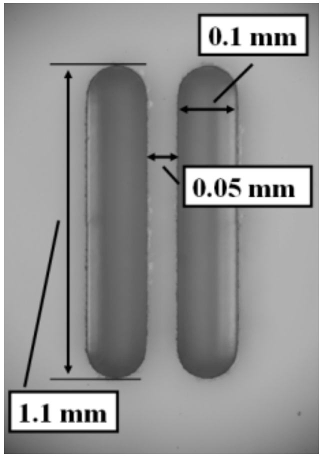
Laser Micromachining

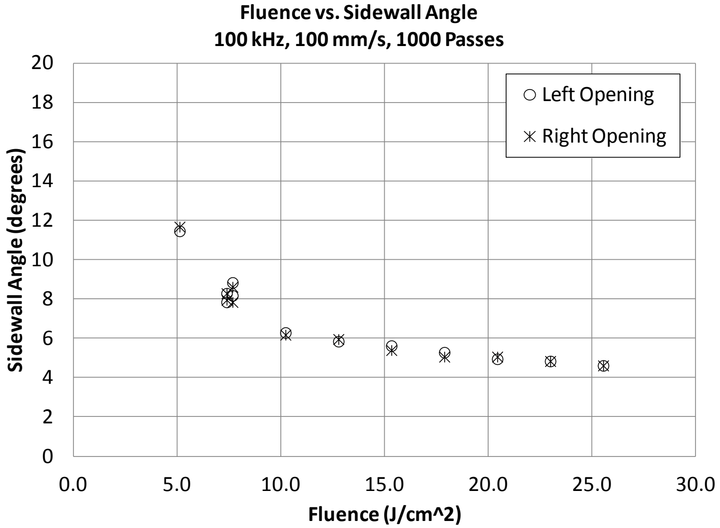
- Ultrashort Pulsewidths (<10 ps)
  - Direct solid-vapor transition
  - Reduced HAZ and micro-cracking
  - Lower fluence required
  - Deterministic material removal rate
  - Research tools
- Oxford Lasers J-355PS Laser Micromachining Workstation
  - Coherent Talisker 355 nm DPSS laser
  - Pulse length <10 – 15 ps
  - Pulse frequency up to 200 kHz
  - Power adjustable from ~0.05 – 4.5 W
  - XYZ stages & galvonometer

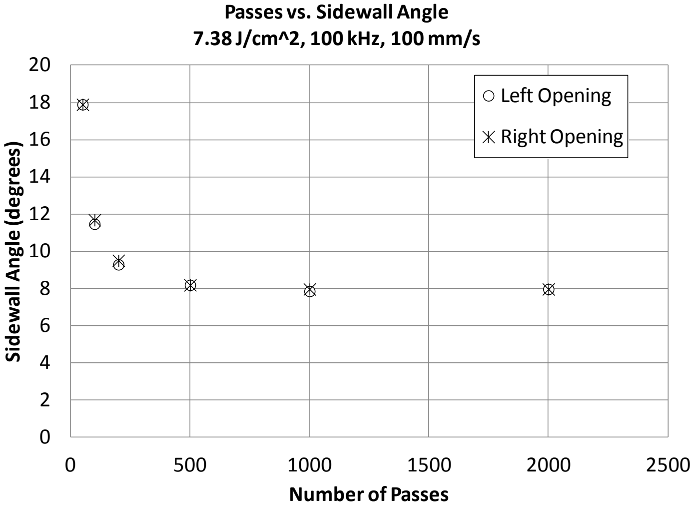


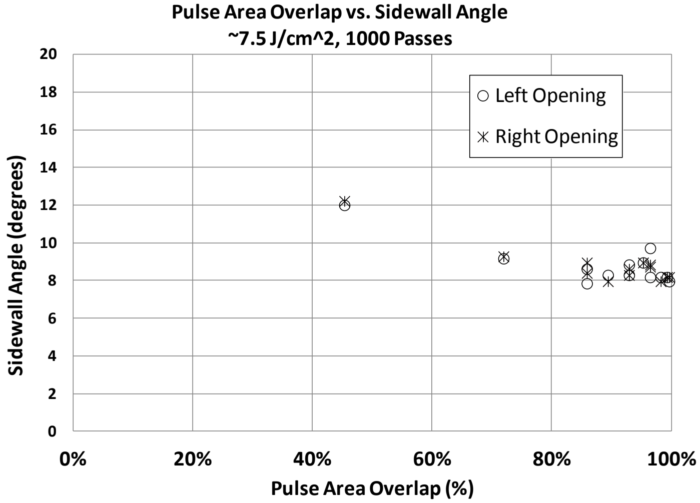


Laser Micromachining Trends







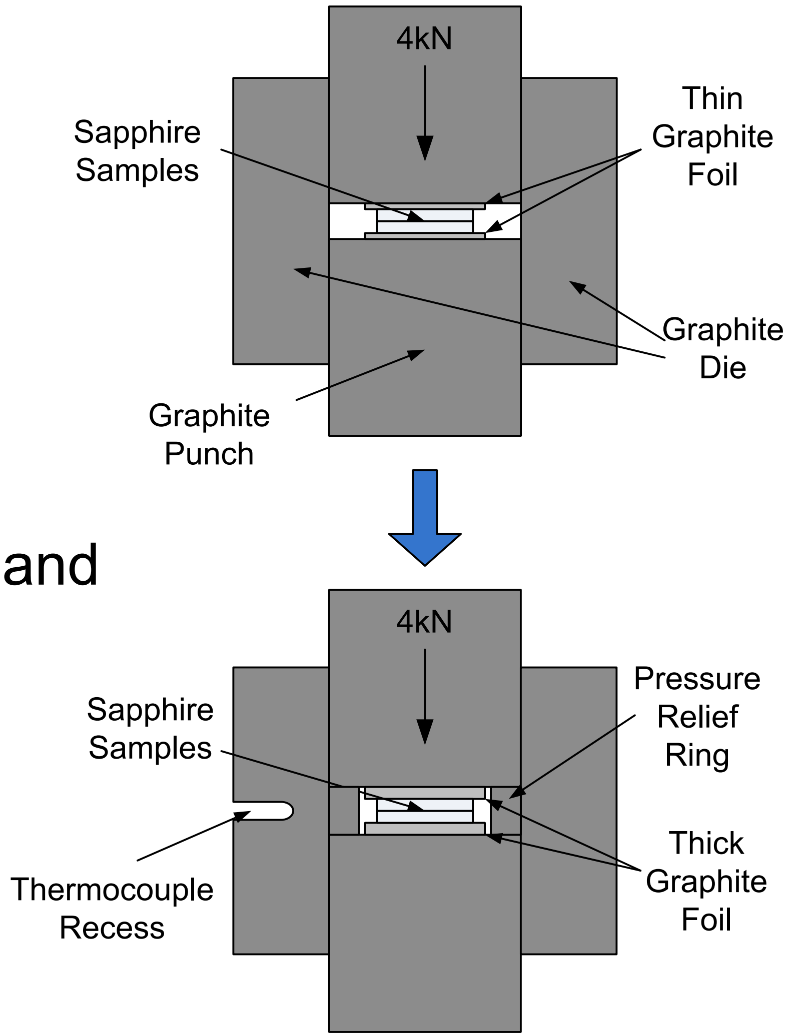


Thermocompression Bonding

- High temperature bonding process
  - 70-90% of melting point (up to 1450°C for sapphire & Pt)
  - 1-10 MPa substrate pressure
  - Up to 24 hour hold time – issues with survivability of patterned features
- Spark Plasma Sintering (SPS) process
  - Large current density (~1000 A/cm^2) causes rapid resistive heating of substrates
  - Faster heating and cooling rates than hot press
  - Reduced temperature and holding time for similar performance

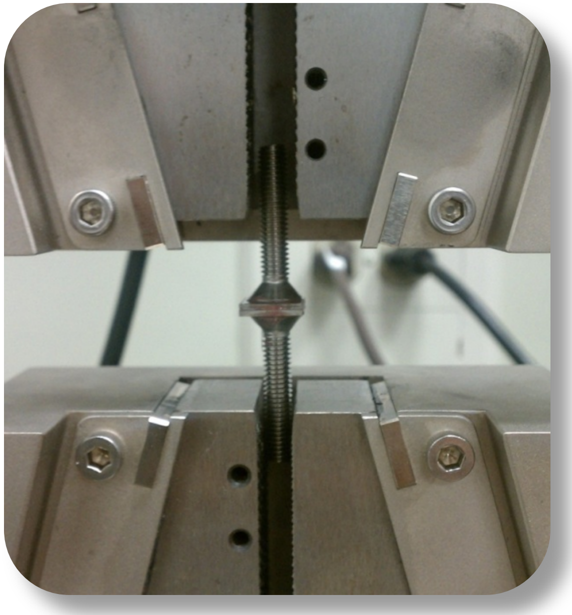
SPS Bonding Process

- Original Process
  - Bond parameters
    - Max temp: 800°C
    - Heating rate: 25°C/min
    - Hold time: 5 minutes
  - Low bond strength
  - Substrate cracking issues
- Modified Process
  - Reduced pressure load via spacer and compressible graphite foil
  - Bond parameters
    - Max temp: 1200°C
    - Heating rate: 50°C/min
    - Hold time: 5 minutes
  - Improved bond strength via higher temps
  - No visible cracks observed



Bond Characterization

- Tensile test
  - Studs bonded to substrates using Hysol 9309.3NA adhesive
  - Original SPS sample tensile strength: ~350 kPa
  - Samples created using modified SPS process: >12 MPa
    - Adhesive joint failed before the bond interface
    - Need improved method for characterization

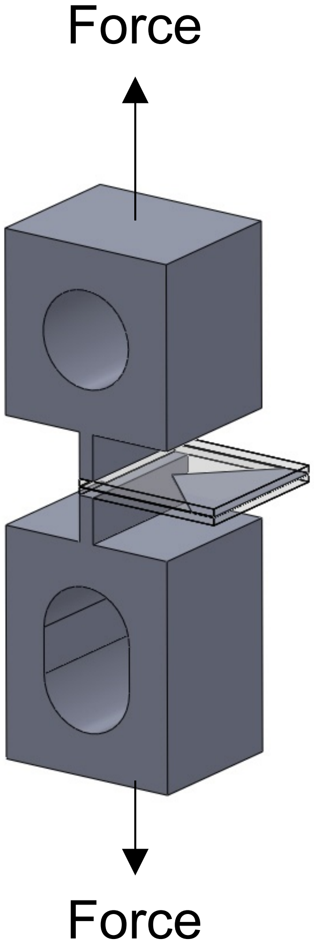


Bond Characterization

- Chevron test
  - Based on SEMI Standard MS5-1211
  - Platinum bonding layer patterned in chevron geometry on sapphire substrate
  - Blocks are attached at the free ends of the bonded specimen
  - Chevron tip creates a pre-crack to initiate failure
  - Max load related to fracture toughness, K_c , and critical wafer bond toughness, G_c

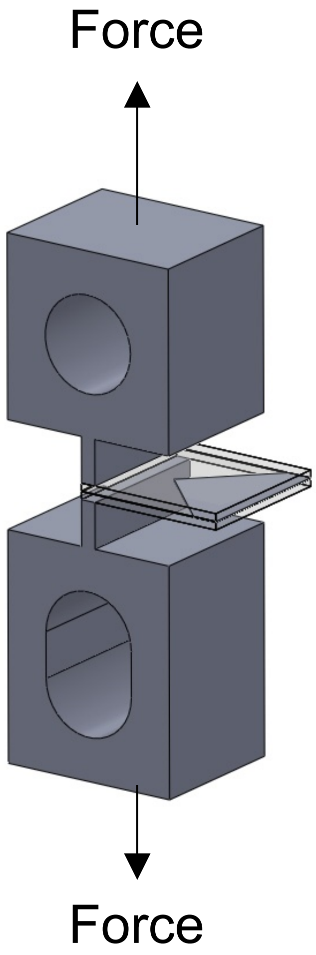
$$K_c \propto F_{max}$$

$$G_c \propto K_c^2$$



Bond Characterization

- Chevron test
  - Based on SEMI Standard MS5-1211
  - Fracture toughness, $K_c = \frac{F_{max}}{B\sqrt{w}} Y_{min}$ where $B = w = 10\ mm$, and Y_{min} is a geometry function determined using FEM simulations
  - Critical wafer bond toughness, $G_c = \frac{K_c^2}{\bar{E}}$ where $\bar{E} = \frac{E}{1 - \nu^2}$ for an isotropic material



Sensor Design

- Mechanical Sensitivity

$$\frac{w_0}{P} = \frac{3}{16}\frac{a^4}{h^3}\frac{1-v^2}{E}$$

- Resonant Frequency

$$f_{res} = \frac{1}{2\pi}\sqrt{\frac{1}{C_{me}M_{me}}}$$

$$C_{me} = \frac{9a^2(1-v^2)}{16\pi E h^3}$$

$$M_{me} = \frac{\rho\pi a^2 h}{5}$$

Residual Stress Estimate

- Pressure drop determined as a function of center deflection[1] (solved using Ritz method)
  - Assumed deflection profile: $w(r) = w_0\left(1 - \frac{r^2}{a^2}\right)^2$

$$\Delta P = \frac{4hw_0}{a^2}\left(\frac{4}{3}\frac{h^2}{a^2}\frac{E}{1-\nu^2} + \sigma_0 + \frac{64}{105}\frac{w_0^2}{a^2}\frac{E}{1-\nu^2}\right)$$

- Solve for σ_0 assuming no pressure drop (ΔP = 0)

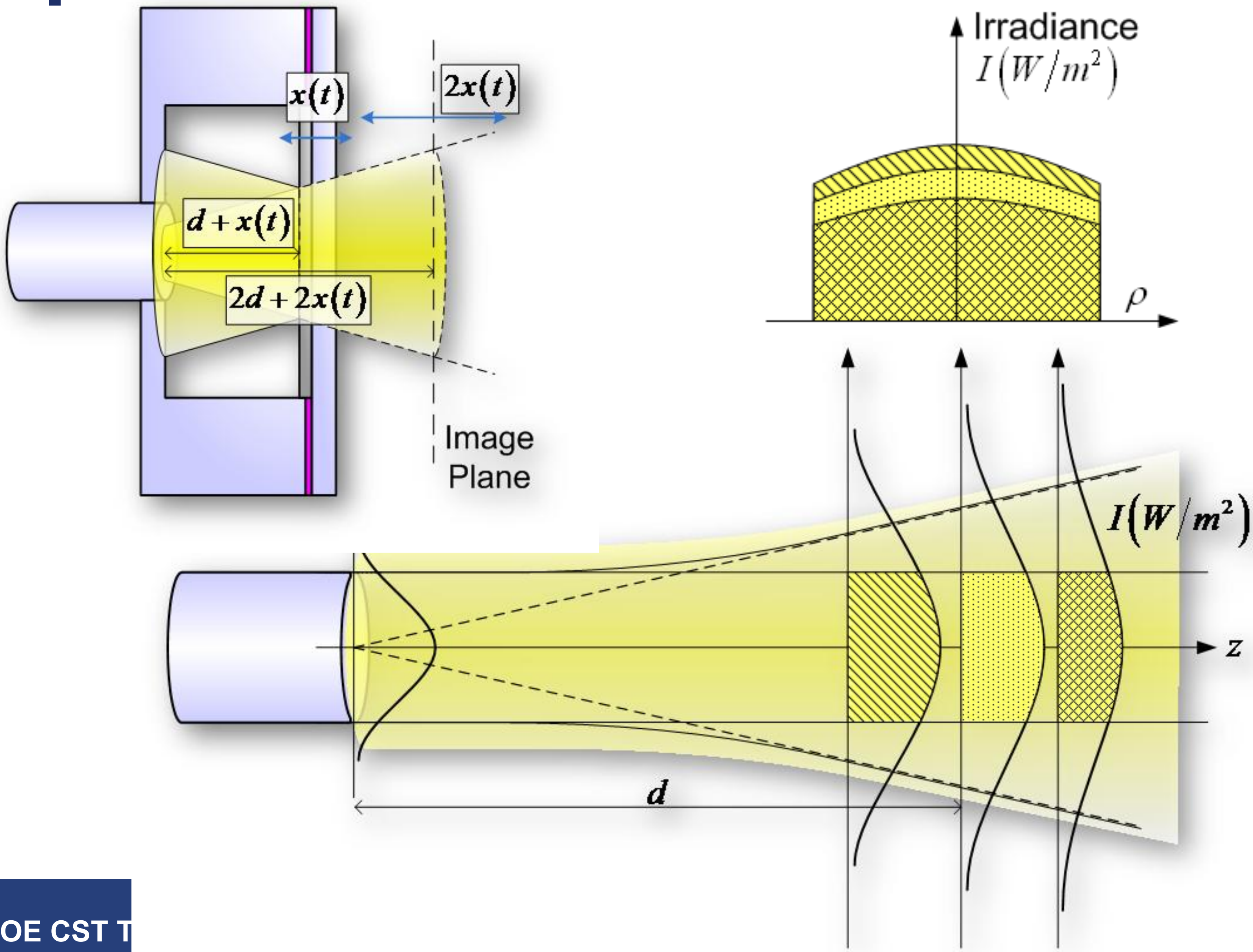
$$\sigma_0 = -\left(\frac{4}{3}\frac{h^2}{a^2} + \frac{64}{105}\frac{w_0^2}{a^2}\right)\frac{E}{1-\nu^2}$$

[1] W.K. Schomburg, *Introduction to Microsystem Design*, Springer, New York, NY, pp. 29-50, 2011.

Choosing a Transduction Scheme

- Factors Influencing Choice of Transducer Concept
  - Specifications: "what do you want to measure?"
    - Physics related: dynamic range, bandwidth, spatial resolution, single sensor versus arrays, fundamental vs. control, etc.
  - Environment: "where do you want to measure it?"
    - Wind tunnel, flight test, gas versus liquid, etc.
      - **Temperature, pressure, humidity, dirt, rain, EMI, shocks, cavitation, fouling, etc.**
  - Packaging Requirements: "where do you mount device?"
    - Application dependent: flush-mounting, single sensor versus arrays (packing density), etc.
  - Other Factors:
    - Budget, time-scale for test, risk tolerance, etc.

Opto-mechanical Transduction



Towards High-Temperature

- Somewhat Uncharted Territory in MEMS
  - Silicon starts to plastically deform at 650 °C
  - Any circuit devices will be temperature limited (diodes, ICs, etc.)
- High-Temperature Limits Transducer Choices
  - Piezoresistive:
    - Leakage current and resistor noise increase with temperature
    - Limited to around 200 °C or must be cooled
  - Capacitive:
    - Low capacitance requires buffer amplifier close to sensor
      - **High-temperature, low noise, high-input impedance amplifiers do not exist**
- Optical is best if you can get it off optical bench
  - Detection electronics are remotely located
  - High temperature sapphire fibers and substrates exist

Oxsensis “Wavephire” Sensor

- Micro-machined sapphire pressure sensor with sapphire fiber-optic
  - Extrinsic Fabry Perot interferometer using at least two wavelengths
  - Diaphragm is micromachined using proprietary process
    - Limitations prevents further miniaturization to sub-millimeter size

- Specifications
  - Temperature range
    - -40 to 600°C (continuous)
    - -40 to 1000°C (research and development)
  - 100 dB dynamic range
  - Uncertainty <±10%