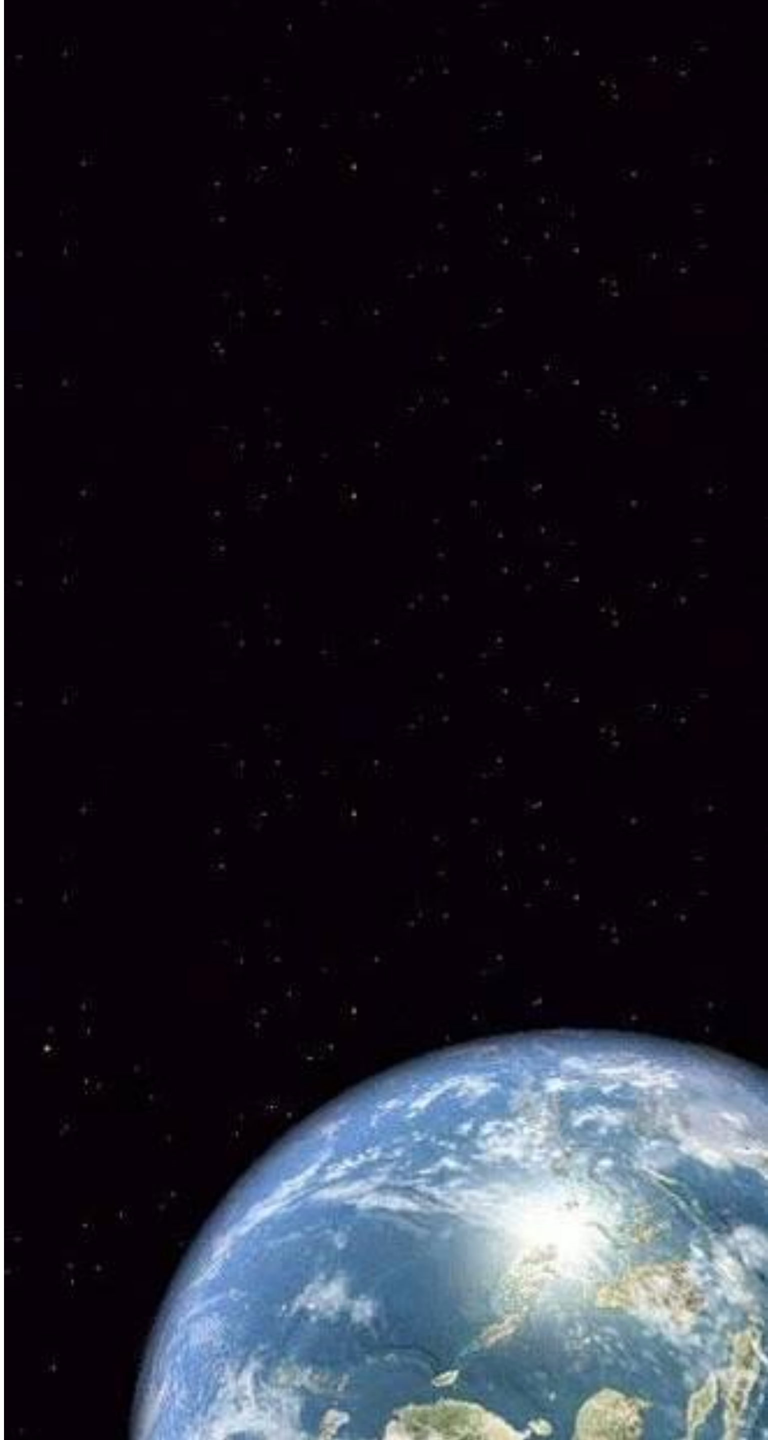
COE CST 2nd Annual Technical Meeting:

High Temperature Pressure Sensors for Hypersonic Vehicles

David Mills

October 31 – November 1, 2012



Overview

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

Team Members

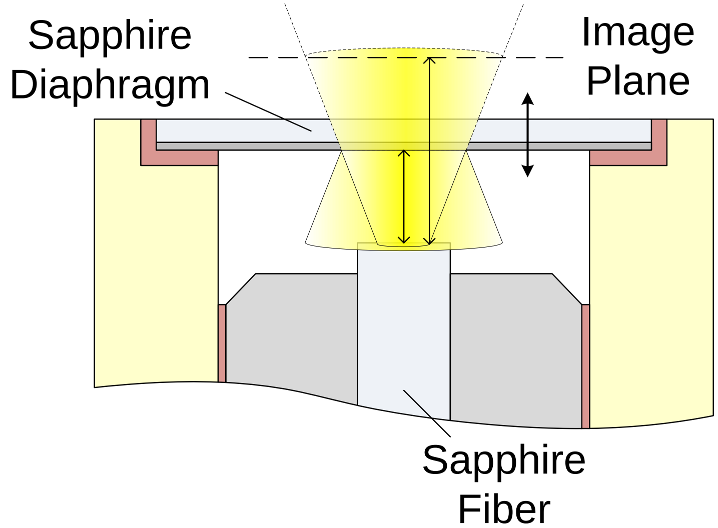
- **University of Florida**
  - **Mark Sheplak** - Professor, Dept. of Mechanical and Aerospace Engineering
  - **David Mills** - Graduate Research Assistant
  - **Daniel Blood** - Graduate Research Assistant
- **Florida State University**
  - **William Oates** - Asst. Professor, Dept. of Mechanical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

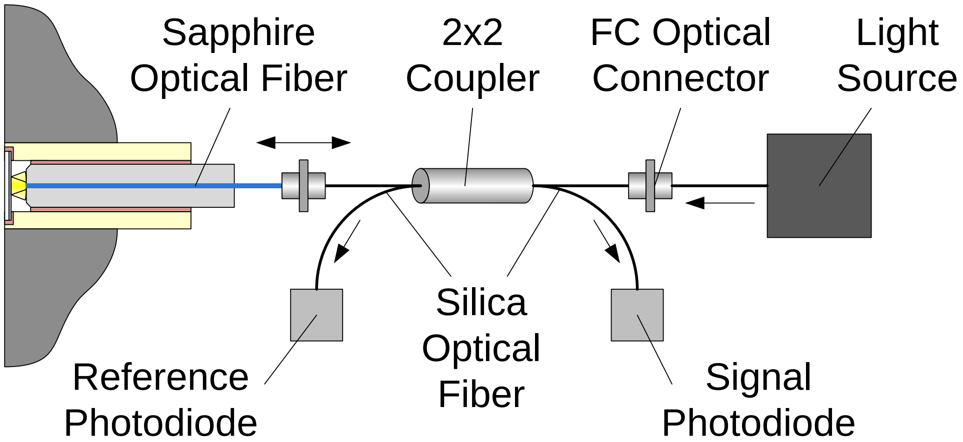
Purpose of Task

- Design, fabricate, and characterize a robust, high-bandwidth micromachined pressure sensor for harsh environments
  - Applications
    - High speed reentry vehicles
    - Hypersonic transports
    - Gas turbines
    - Scramjets
  - Performance Metrics
    - Temperature: >1000 C
    - Bandwidth: >10 kHz
- Develop novel processing techniques for the fabrication of high temperature sensors
  - Laser micromachining processes for patterning of structures in sapphire and alumina
  - Bonding process to for fabrication of multi-wafer sensors enabling three-dimensional structures

Research Methodology

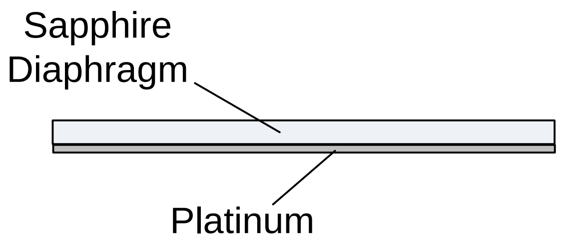
- Fiber optic lever
  - Intensity modulation
  - Single fiber in/fiber out
- Optical configuration
  - Multimode silica fibers
    - More efficient coupling to sapphire fiber
  - Incoherent LED light source
  - Reference photodiode to monitor source drift

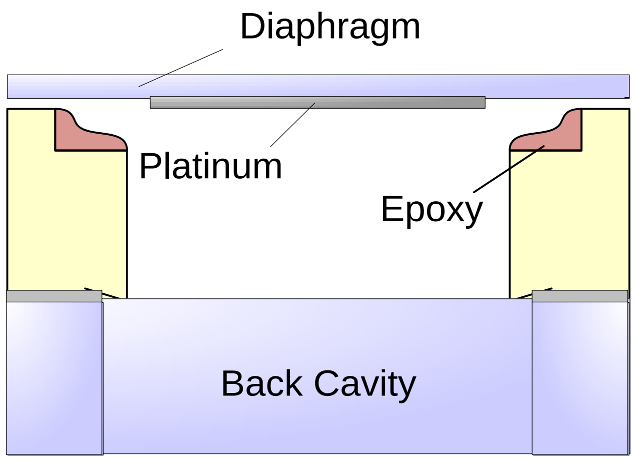




Device Fabrication

- 3mm tube sensor
  - 50 µm sapphire diaphragm
  - Deposit platinum reflective layer w/ titanium adhesion layer
  - Laser machine 4.5 mm recess in alumina tube
  - Epoxy diaphragm inside recess
- 7mm flat sensor
  - 50 µm sapphire diaphragm
  - Deposit platinum reflective layer w/ titanium adhesion layer in center
  - 1 mm thick sapphire substrate
  - Machine 7 mm diameter hole in 1 mm thick sapphire to form back cavity
  - Deposit 500 nm platinum bonding layer on 1 mm thick substrate
  - Align and bond diaphragm to cavity substrate



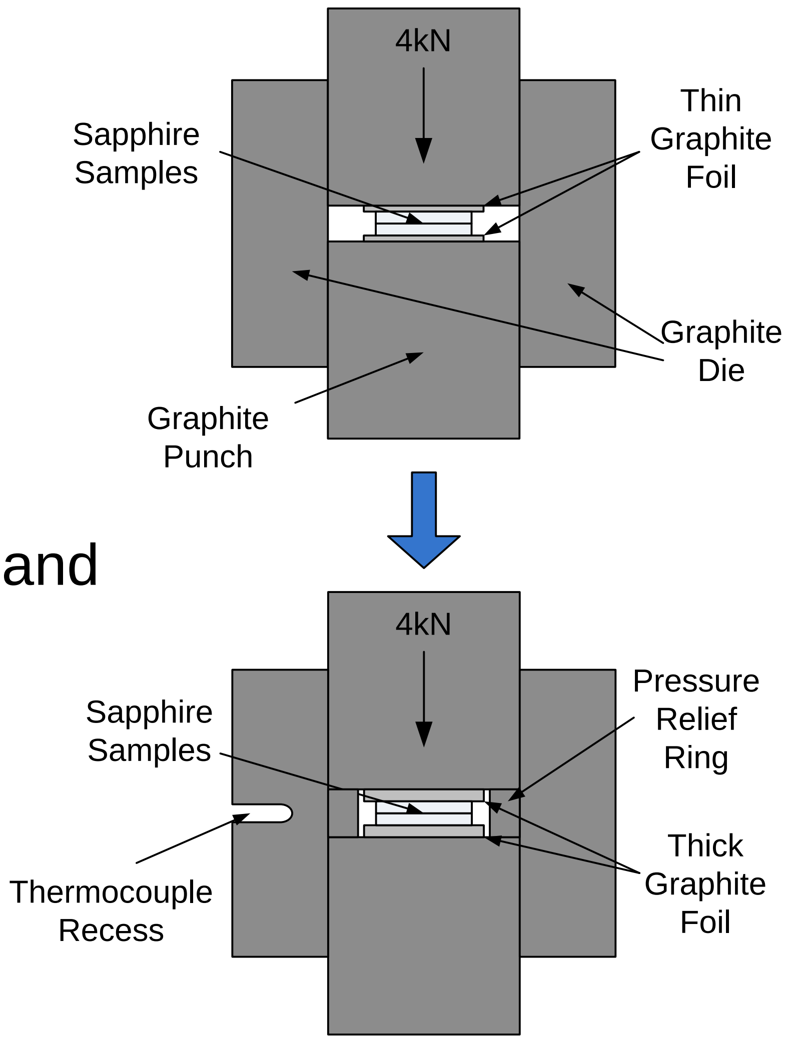


Fabrication Challenges

- 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
  - Minimize thermal stress effects

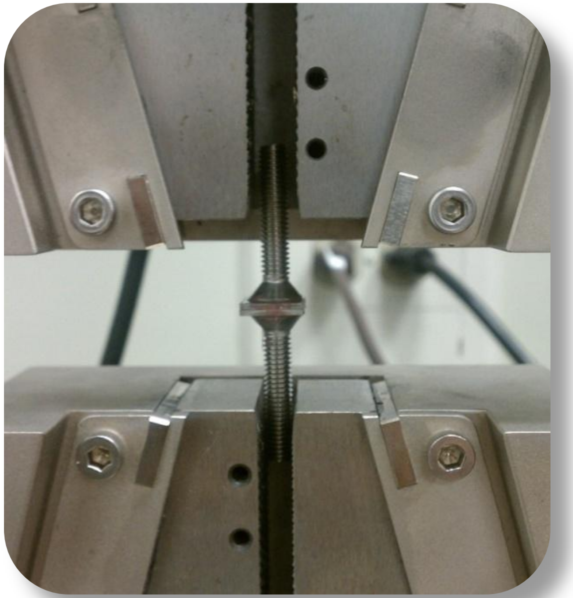
SPS Bonding Process

- 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

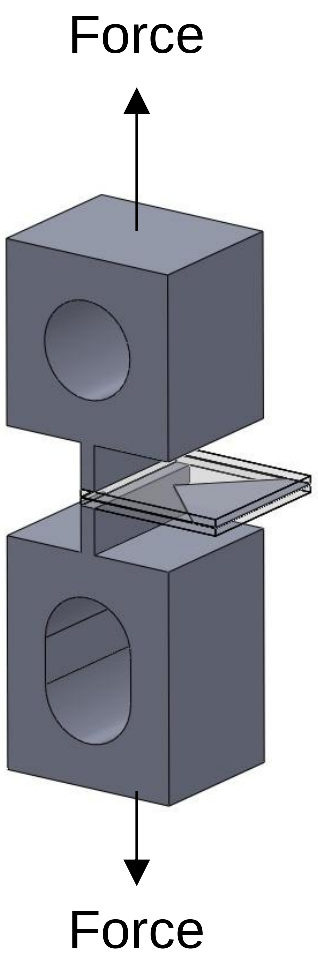


Bond Characterization

- 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

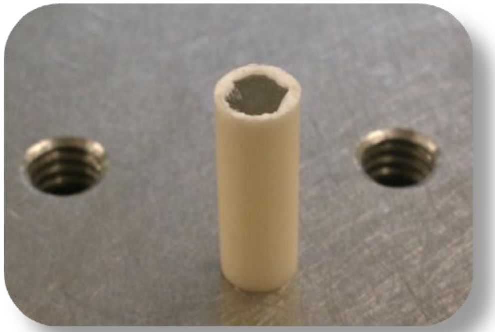
$$K_c \propto F_{max}$$

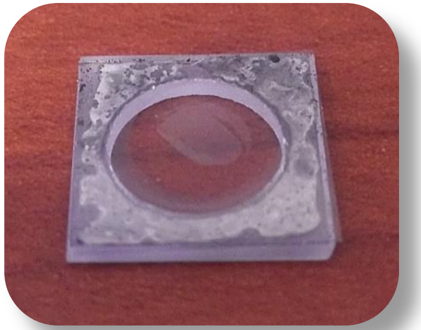
$$G_c \propto K_c^2$$

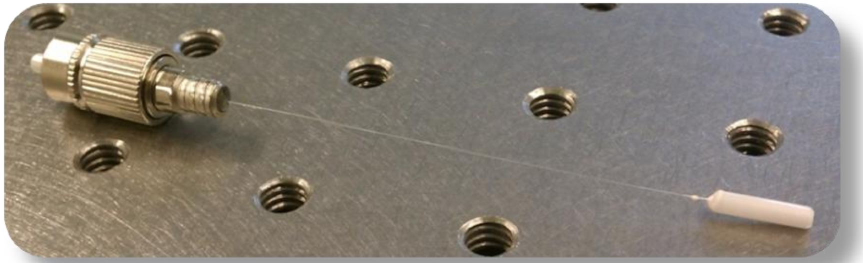


Sensor Fabrication

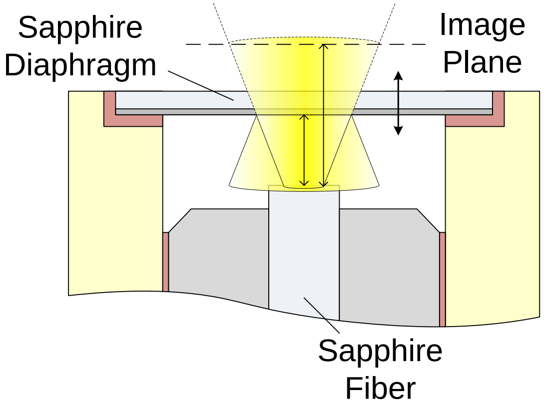
- High-temp prototype sensors
  - 3mm tube sensor
    - Ti/Pt-coated sapphire diaphragm epoxied to alumina housing
  - Sapphire fiber w/ zirconia optical ferrule
  - 7mm flat sensor
    - 50um sapphire diaphragm attached to 1mm thick back-cavity using SPS bond process
    - Reflective film degradation, buckling



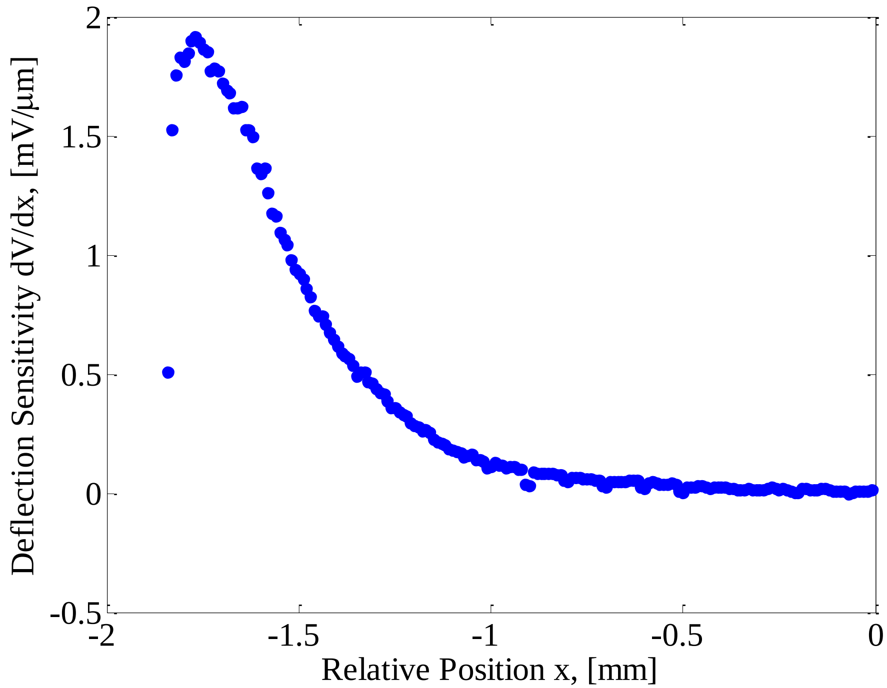


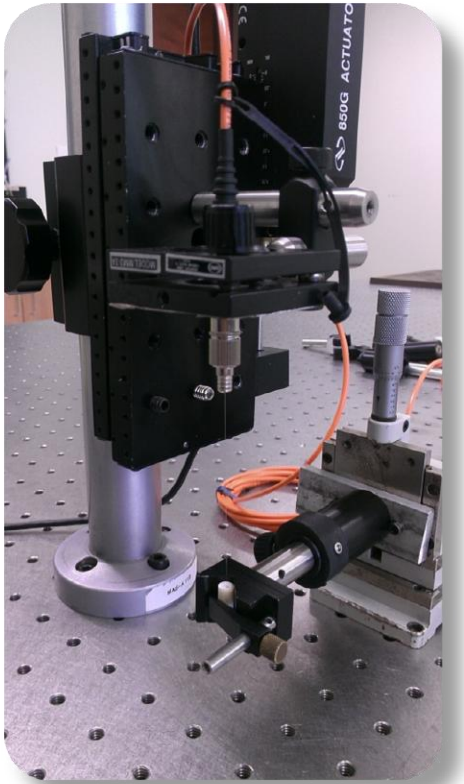


Sensor Packaging



- Sensitivity Calibration
  - Experimentally determined optimal distance from fiber to diaphragm
  - Max deflection sensitivity of 1.92 mV/µm



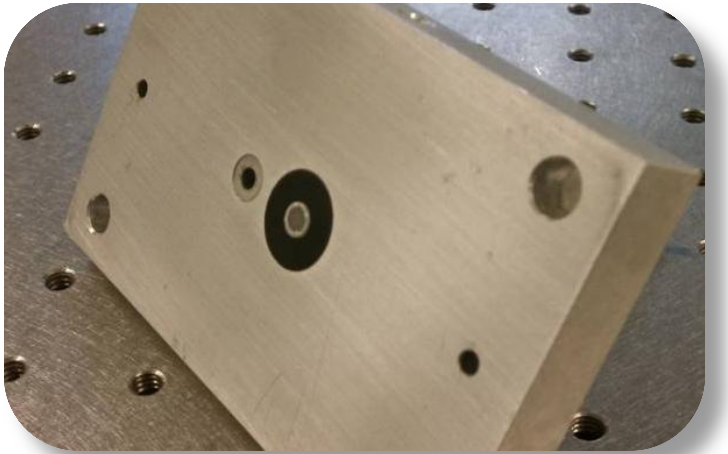


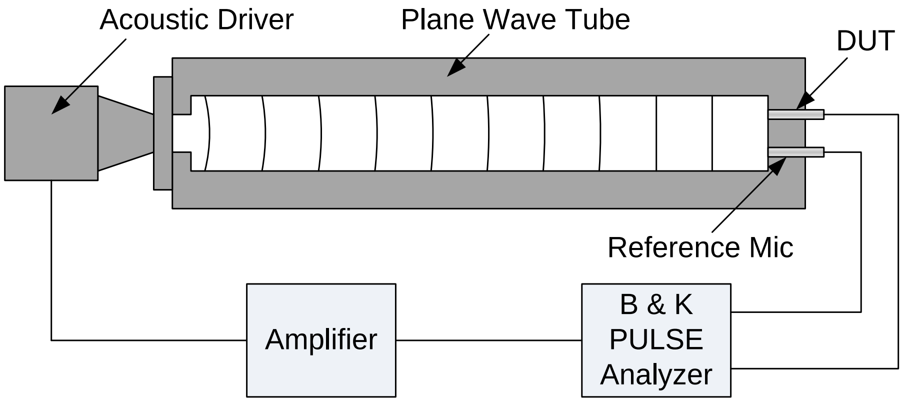
Sensor Packaging

- High-temp epoxy used on all connections
- Stainless steel braid and crimps
- Standard FC optical connector couples to traditional silica optical fiber components
- Package capable of operation up to 600 C

Next Steps

- Process development
  - Laser machining parameters for thinning sapphire diaphragms
  - Evaluate SPS bonding process using chevron test specimens
  - Improve metal film survivability during bonding
- Package 7mm flat sensor
- Static pressure calibration
- PWT calibration
  - Frequency response
  - Linearity
- High-temperature calibration
  - Temperature drift
  - Environmental chamber



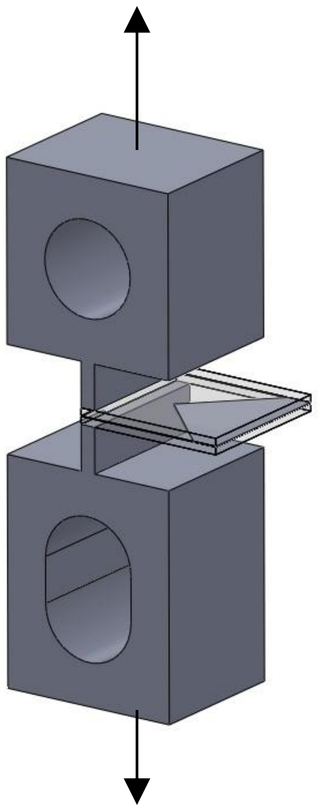


Contact Information

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

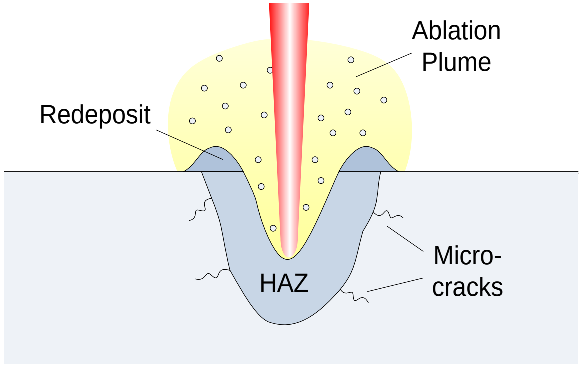
Bond Characterization

- Chevron test
  - 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



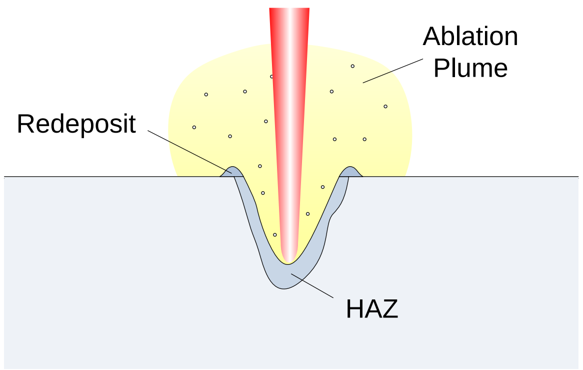
Laser Micromachining

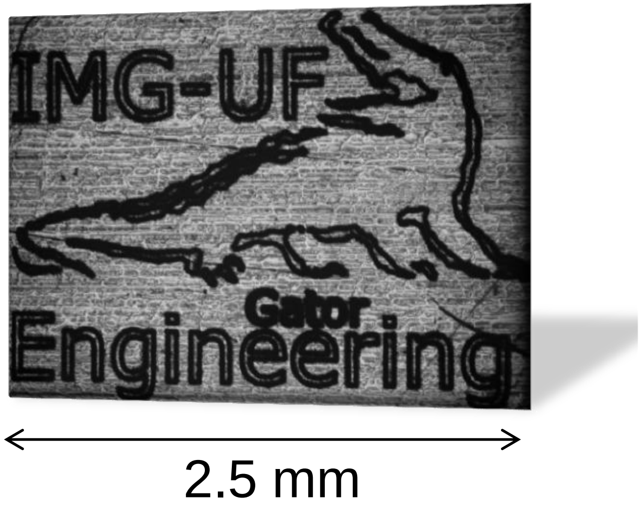
- "Long" Pulsewidths (>10 ps)
  - Industry standard
  - High reliability
  - Large heat affected zone (HAZ)
  - Micro-cracking and redeposit



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



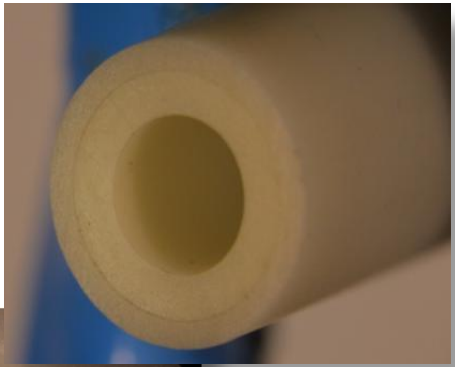


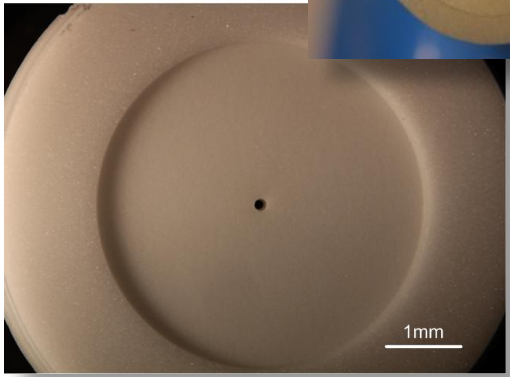
Thermocompression Bonding

- 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

Process Development Results

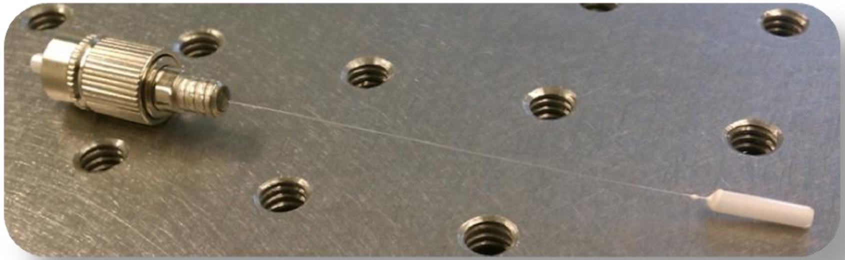
- Laser Machining
  - Cutting speed: 100 mm/s
  - Frequency: 100 kHz
  - Pulse overlap: ~86%
  - Laser fluence
    - Alumina: 2.45 J/cm^2
    - Sapphire: 4.48 J/cm^2
- Bonding
  - Bond parameters
    - Max temp: 800 C
    - Heating rate: 25 C/min
    - Hold time: 5 minutes
  - Tensile strength: ~350 kPa
  - Substrate cracking issues

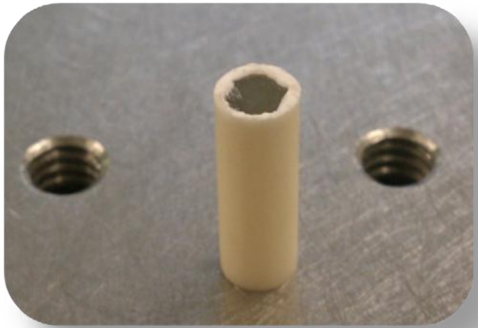




Fabrication Results

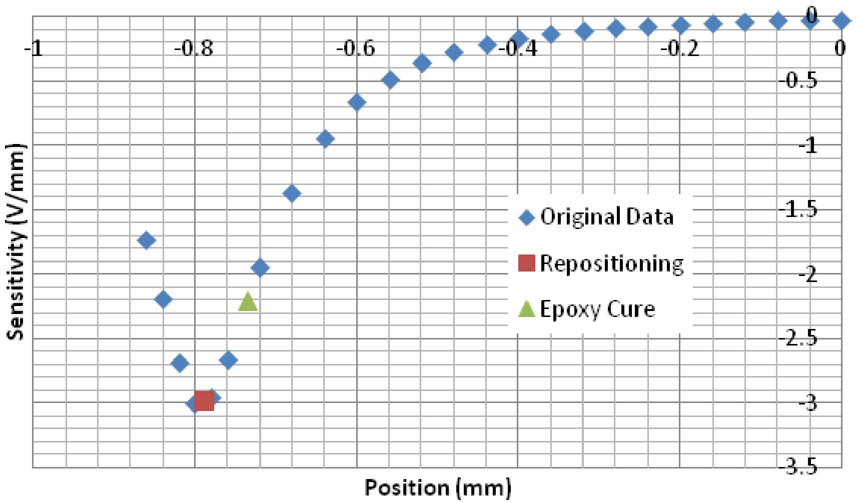
- Low Temperature Prototype
  - Silicon diaphragm
  - Silica fiber and low temp epoxy
- High Temperature Sensor
  - Pt-coated sapphire diaphragm
  - Sapphire fiber w/ zirconia optical ferrule

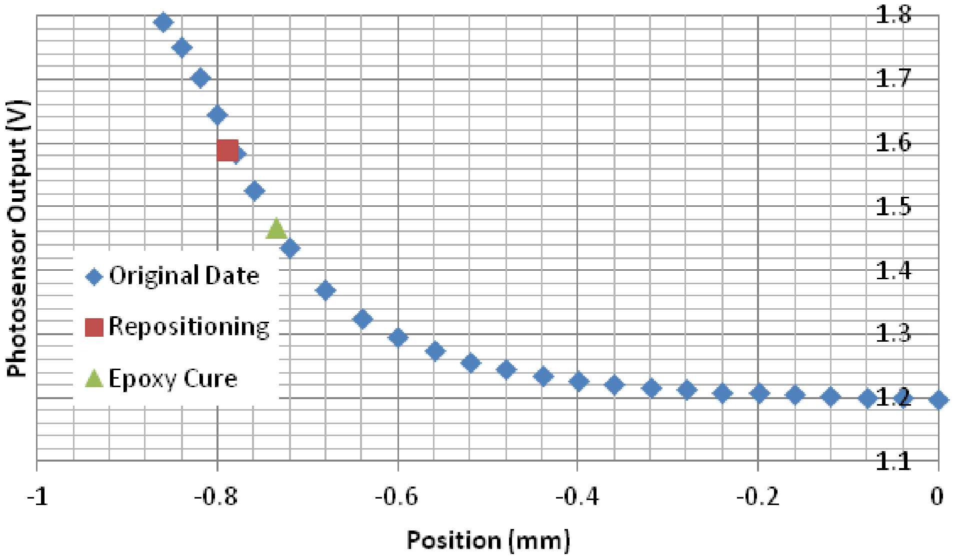




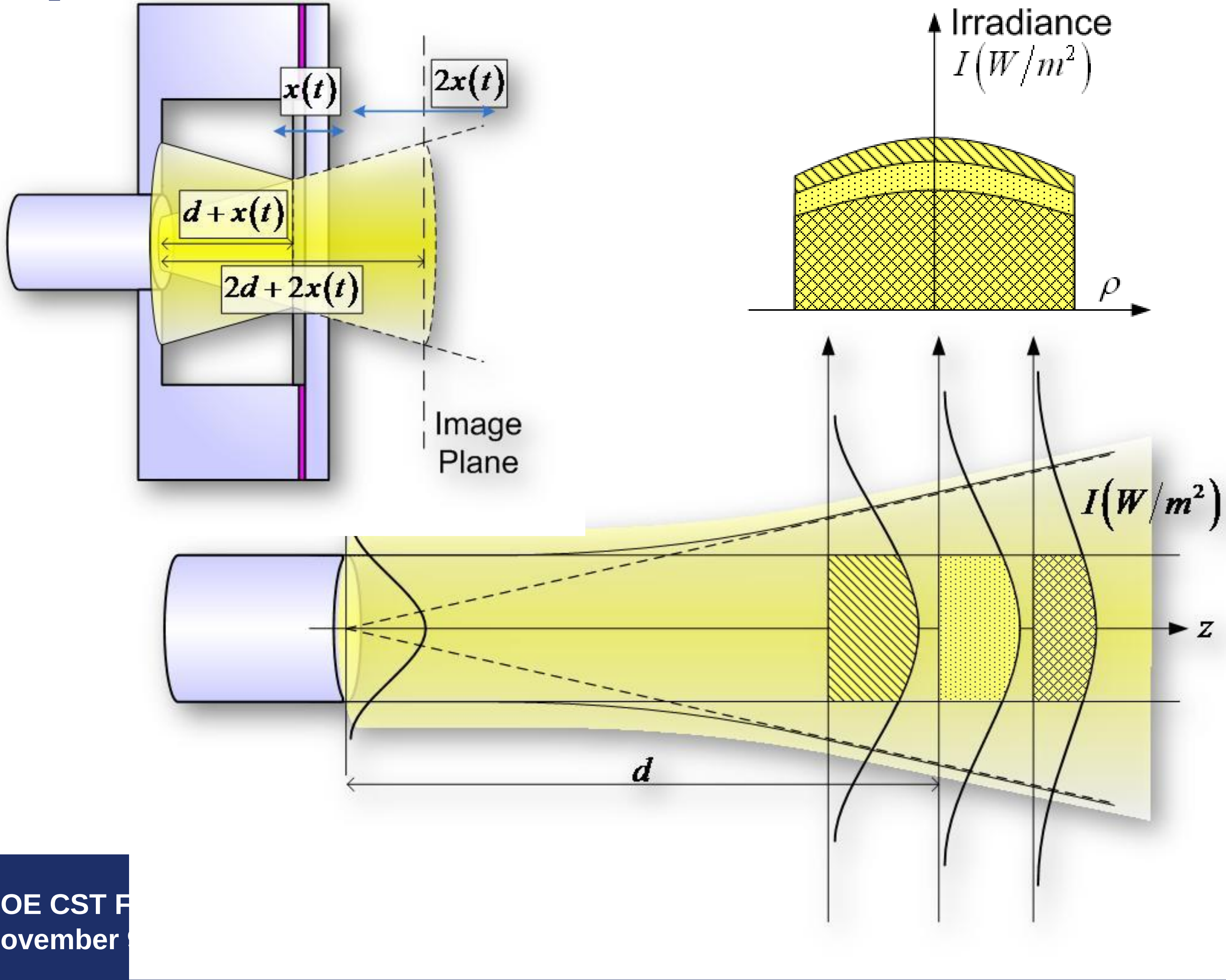
Backup Slides

- Prototype Sensor Static Calibration

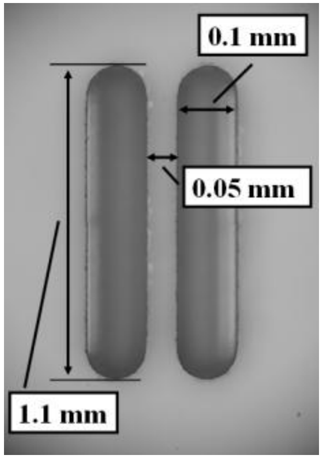


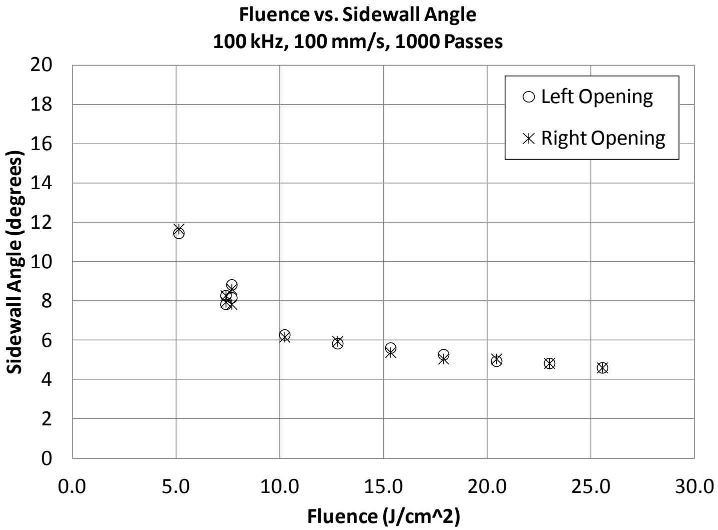


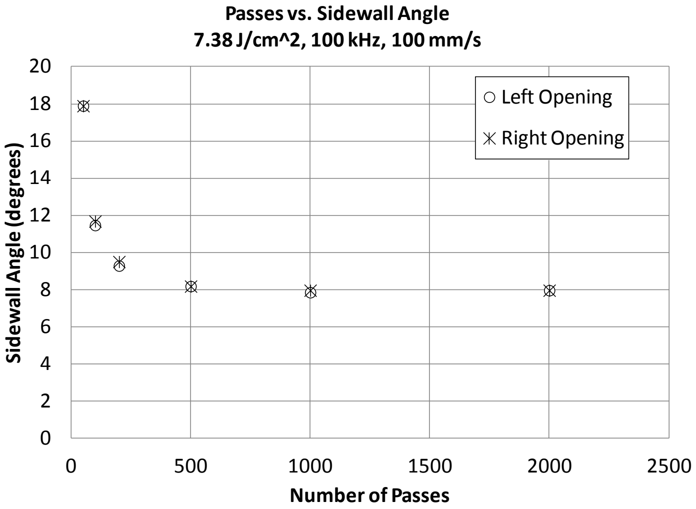
Opto-mechanical Transduction

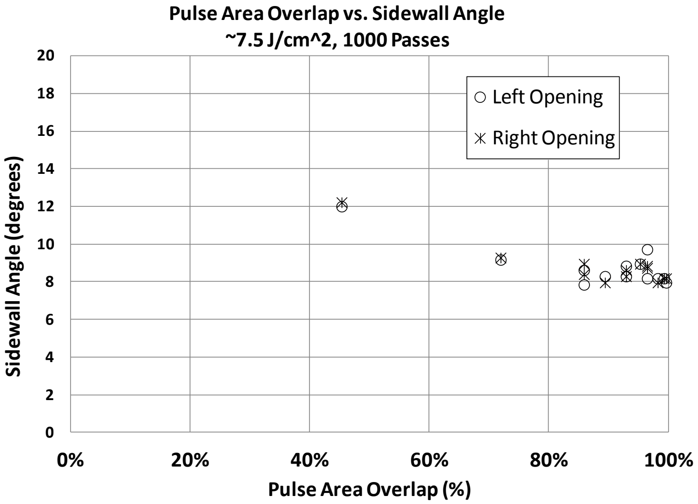


Laser Micromachining Trends

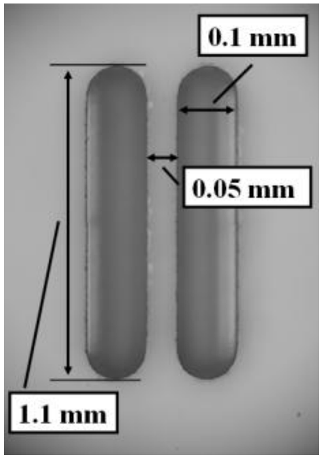


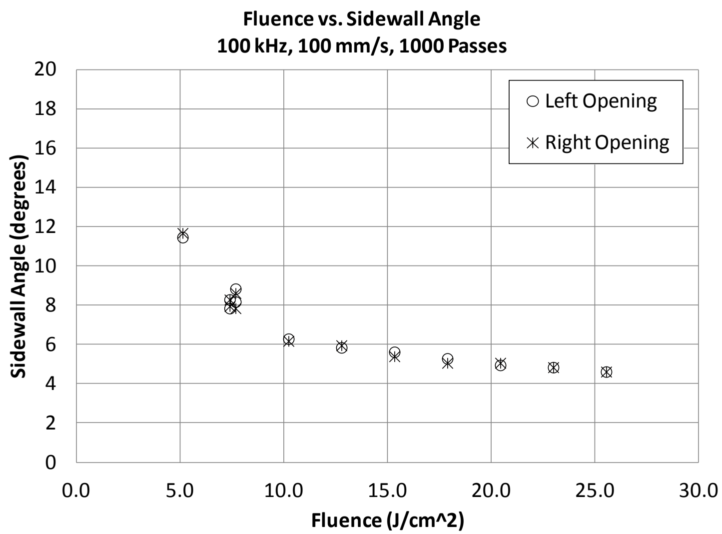


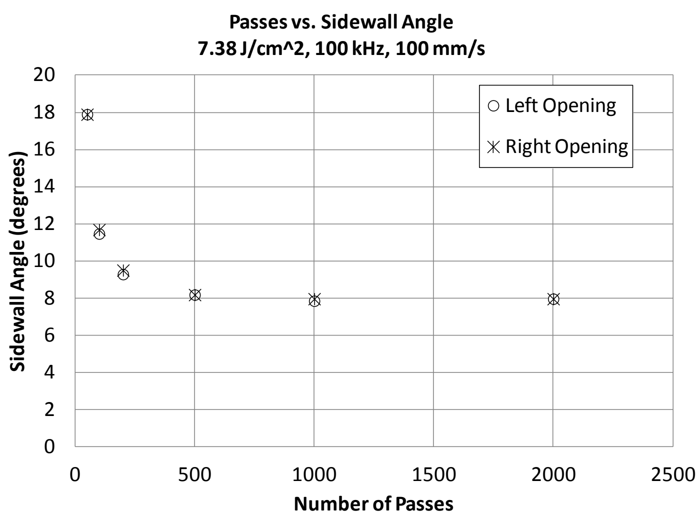


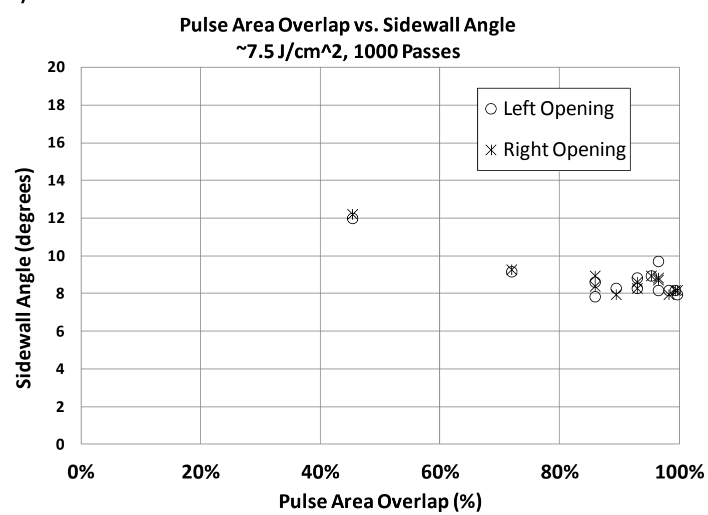


Laser Micromachining Trends









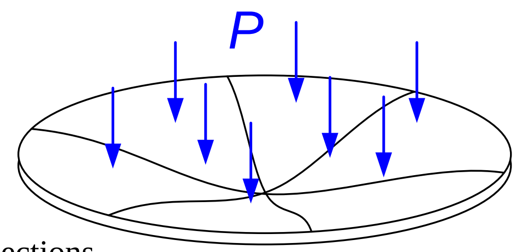
Oxsensis "Wavephire" Sensor

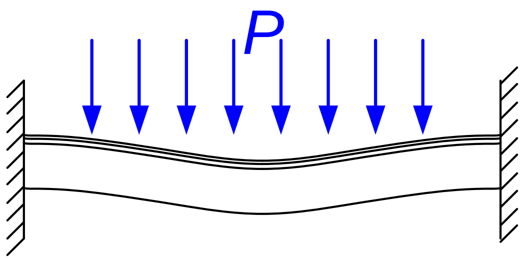
- Micro-machined sapphire pressure sensor with sapphire fiber-optic
  - Extrinsic Fabry Perot interferometer using at least two wavelengths
  - Diaphragm is micromachined using proprietary process
    - Limitations prevents further miniaturization to sub-millimeter size

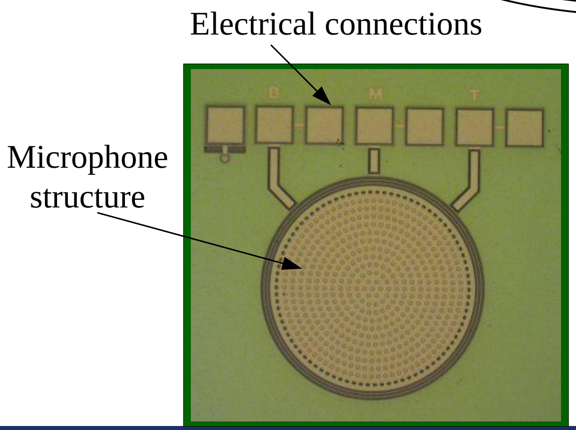
- Specifications
  - Temperature range
    - -40 to 600°C (continuous)
    - -40 to 1000°C (research and development)
  - 100 dB dynamic range
  - Uncertainty <±10%

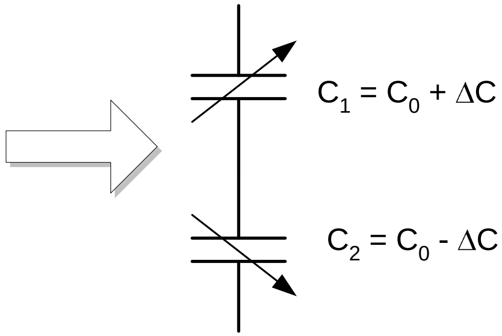
Dynamic Pressure Sensors

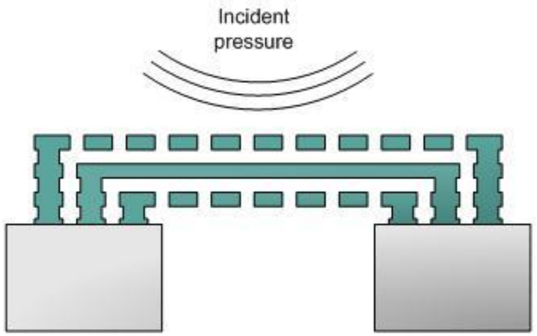
- **Diaphragm Sensors**
  - **Diaphragm deflects vertically due to incoming pressure**
  - **Displacement sensed via transduction method**
- **Transduction Schemes**
  - **Capacitive, optical, piezoresistive, piezoelectric, etc.**











Choosing a Transduction Scheme

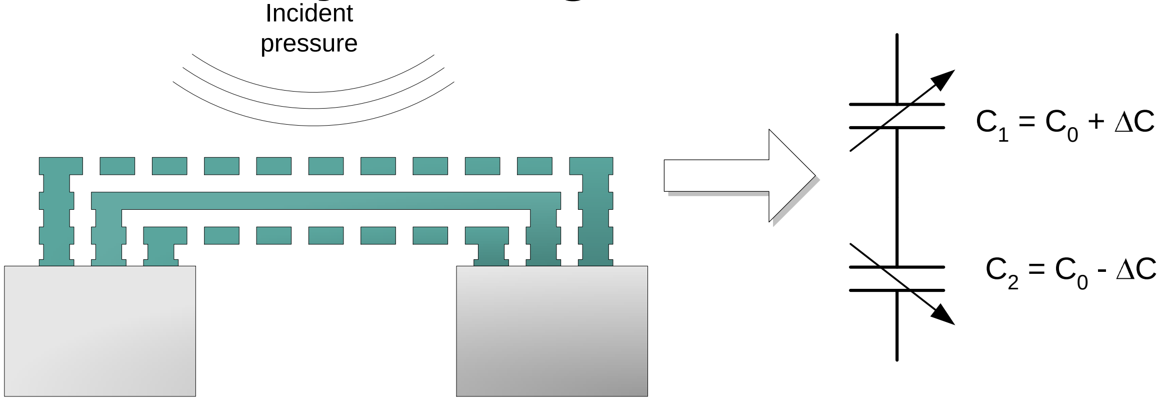
- Factors Influencing Choice of Transducer Concept
  - Specifications: “what do you want to measure?”
    - Physics related: dynamic range, bandwidth, spatial resolution, single sensor versus arrays, fundamental vs. control, etc.
  - Environment: “where do you want to measure it?”
    - Wind tunnel, 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.

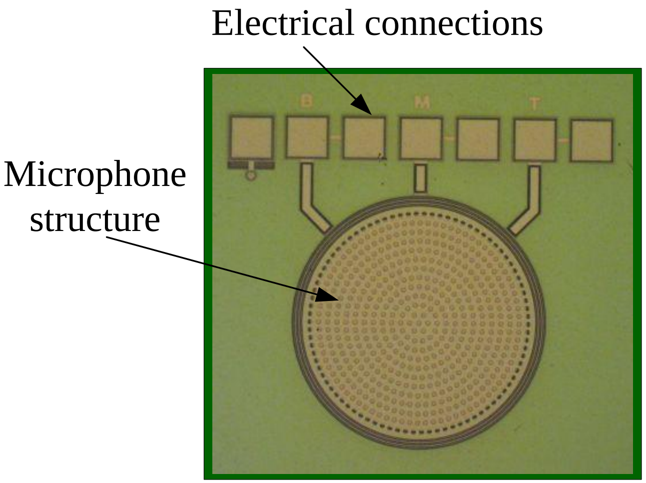
Towards High-Temperature

- Somewhat Unchartered 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

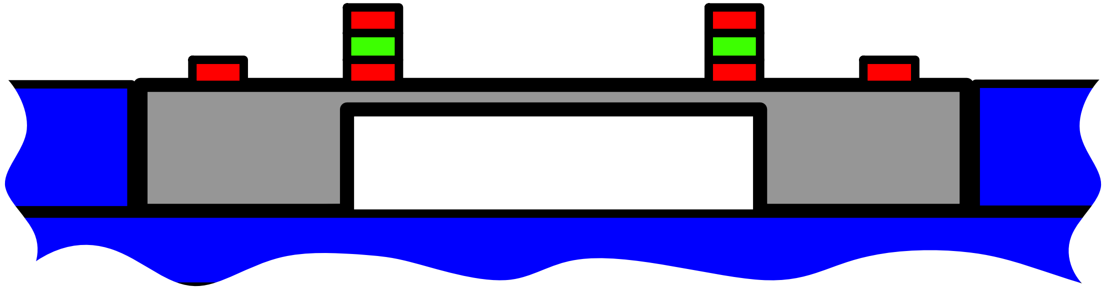
Microphones / Pressure Sensors

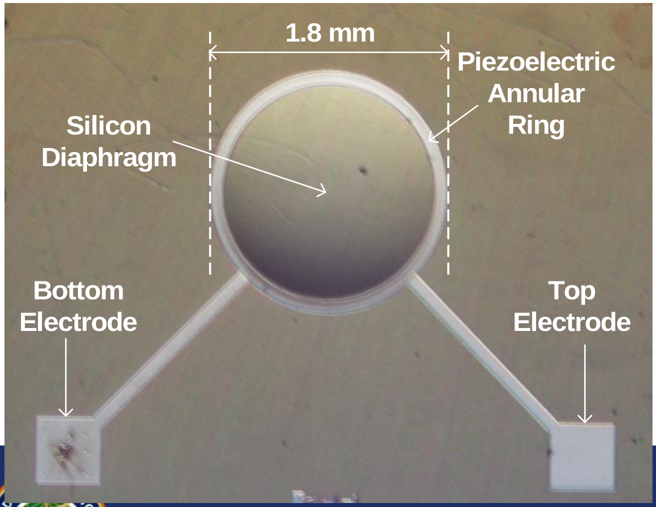
- **Capacitive**: Sensitivity= 0.28 mV/Pa, DR= 22-160 dB, f_{res} = 158 kHz
  - Arrays, benign environments





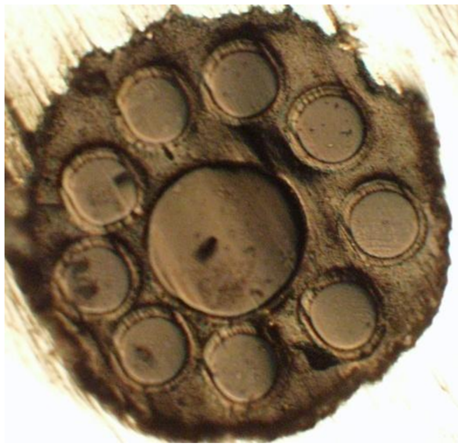
- **Piezoelectric**: Sensitivity= 0.75 mV/Pa, DR= 48-169 dB, f_{res} = 50 kHz
  - Fuselage TBL studies

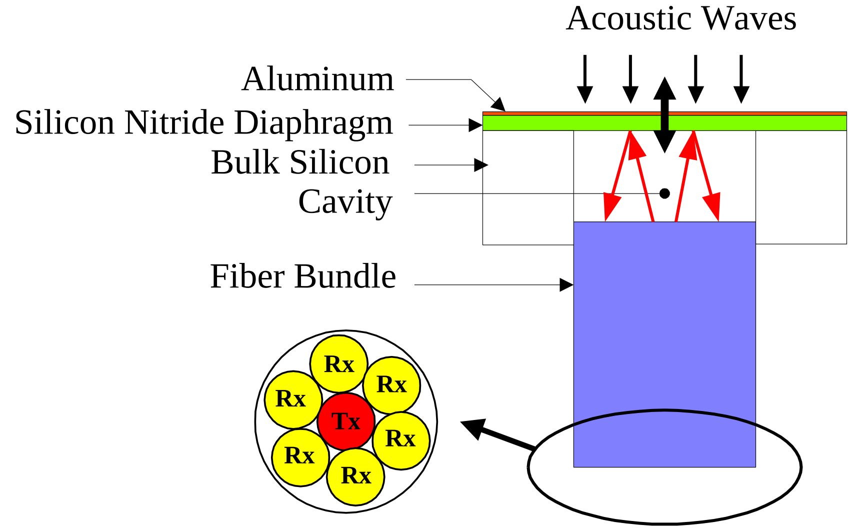




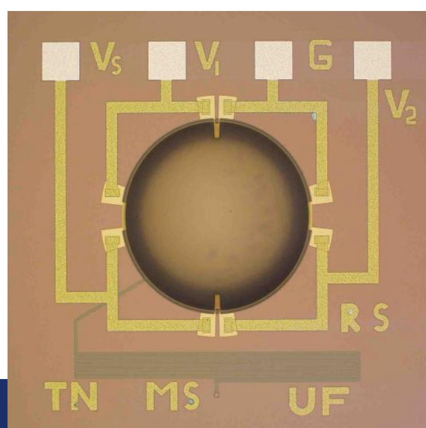
Microphones / Pressure Sensors

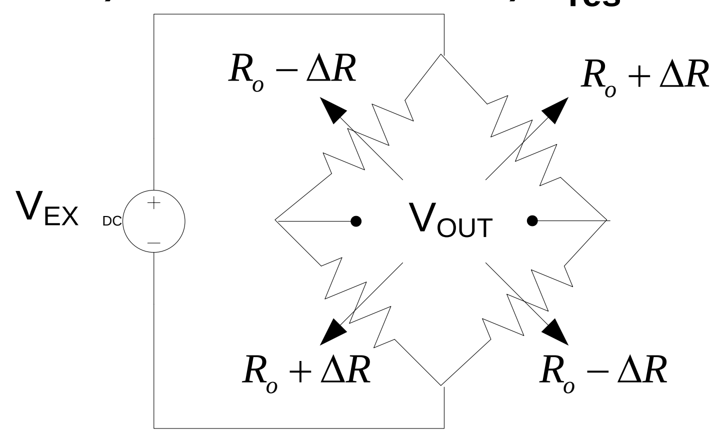
- **Fiber Optic: Sensitivity= 0.5 mV/Pa, DR= 70-160 dB, f_{res} > 100 kHz**
  - **Hostile environments**





- **Piezoresistive: Sensitivity= 1.8 μV/Pa, DR= 52-160 dB, f_{res} > 100 kHz**
  - **Directional acoustic arrays**





Material Properties

		Units	Silicon	Silica	Sapphire	Diamond	6H SiC
Material Properties	Melting Temp	°C	1412 [1]	1650	2040 [2]	3650 - sublimes	2830 - sublimes [1]
	Max Use Temp	°C	650 - strain point	1100 - no load [7]	1800 - no load [2]	650 - Si substrate	1650 - no load [5]
	Tensile Strength	GPa	7.0 [6]	8.4 [6]	15.4 [6]	53.0 [6]	21.0 [6]
	Poission's Ratio	-	0.28 - [100] plane, 0.26 - [110] plane [1]	0.14 - 0.17 [9]	0.25 - 0.3 [2]	0.1 [1]	0.14 [5]
	Young's Modulus	GPa	130 - [100] plane, 170 - [110] plane [1]	73 [6]	530 [6]	1035 [6]	700 [6]
	CTE, 20°C	µm/m-°C	2.6 [1]	0.55 [9]	5 - ⊥ to C-axis [2]	0.8 [1]	4.7 - ∥ to C-axis, 4.3 - ⊥ to C-axis [1]
	Thermal Conductivity, 20°C	W/m-°C	130 [1]	1.4 [9]	41.9 [2]	600-2000 [1]	490 [1]
	Thermal Shock Parameter [8]		1.52E+06	2.52E+05	1.83E+05	3.46E+07	2.94E+06
	Optical Transmission, UV-NIR	%	~0 - λ < 1.05µm, 50 - λ > 1.05µm [4]	86-93 [7]	80-90 [3]	60-70 [9]	70-80 [1]
	Refractive Index	-	3.42 (IR) [1]	1.45 @ 589 nm [7]	1.8 - 1.6, UV-IR [2]	2.4 (IR) [1]	2.59 - ∥ to C-axis, 2.55 - ⊥ to C-axis (IR)[1]
Transducer Issues	Optical Fiber Availability		no	yes	yes	no	no
	Substrate Availability		excellent	excellent	excellent	poor	limited
	Patternability / Process		Standard MEMS Processes		Laser Micromachining	Liftoff	SiC specific DRIE process, micromolding
	Transduction Mechanisms						