High-Temperature Sapphire Pressure Sensors for Harsh Environments

Justin Kiehne[1], Peter Woerner[2],
William Oates[2], Mark Sheplak[1]

[1]Interdisciplinary Microsystems Group, University of Florida
[2]Florida State University

DE-FE0012370
2016 NETL Crosscutting Research Review Meeting
April 18, 2016

Interdisciplinary Microsystems Group
UNIVERSITY OF FLORIDA

Outline

- Introduction
- Laser Ablation Modeling
- Thermal Damage Analysis
- Thermocompression Bonding
- High Temperature Testing Facility
- Conclusion

Outline

- Introduction
  - Project overview
  - Motivation
  - Approach
  - Proof-of-Concept Device
  - Objectives and Summary
- Laser Ablation Modeling
- Thermal Damage Analysis
- Thermocompression Bonding
- High Temperature Testing Facility
- Conclusion

Project Overview

- 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: 4 years, 1 year NCE, beginning Jan 2014
- Project team
  - UF (Project lead)
  - FSU

Motivation

- Advanced energy systems require harsh environment instrumentation:
  - Process control/closed loop feedback
  - Increased efficiency
  - Reduced emissions & cost
- Applications
  - Coal gasification
  - Gas turbines
  - Solid oxide fuel cells
  - Deep oil and geothermal drilling

Motivation

- Sensor operational requirements
  - Temperature: >1000°C
  - Dynamic pressure: up to 1000 psi
  - Atmosphere: corrosive and/or erosive

- Conventional pressure sensor instrumentation is limited to ~500°C
- Current temperature mitigation techniques:
  - Stand-off tubes
  - Water cooling

Outline

- Introduction
  - Project overview
  - Motivation
  - Approach
  - Proof-of-Concept Device
  - Objectives and Summary
- Laser Ablation Modeling
- Thermal Damage Analysis
- Thermocompression Bonding
- High Temperature Testing Facility
- Conclusion

Approach

- Transduction mechanisms
  - Capacitive
  - Optical
  - Piezoelectric
  - Piezoresistive
- Benefits of fiber optic transduction
  - DC measurement
  - Immunity to EMI
  - Passive
  - Non-conductive
  - Remote electronics
  - Multiplexing

Approach

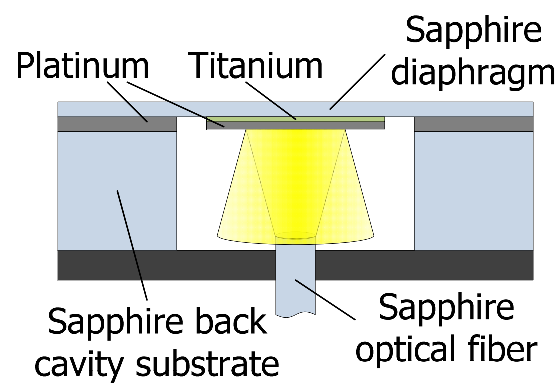
- Sensor/optical fiber materials
  - ~~Silicon~~
  - ~~Silica~~
  - Silicon carbide
  - Sapphire
  - Diamond
- Benefits of sapphire
  - High melting point (2053°C)
  - Resistance to chemical corrosion
  - Excellent hardness
  - Large transmission window (200 nm – 5 μm)
  - Multimode optical fibers available

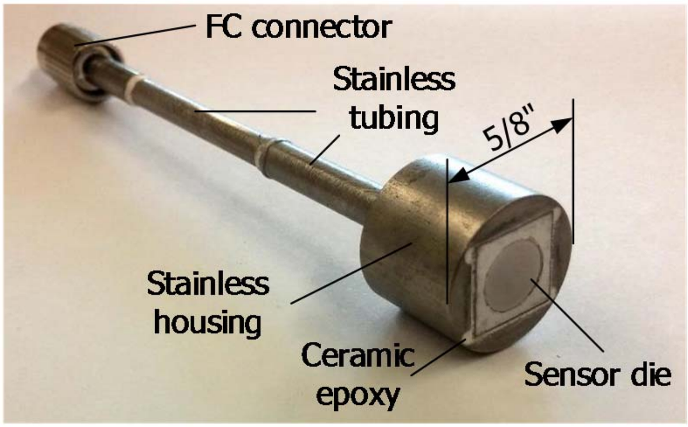
Approach

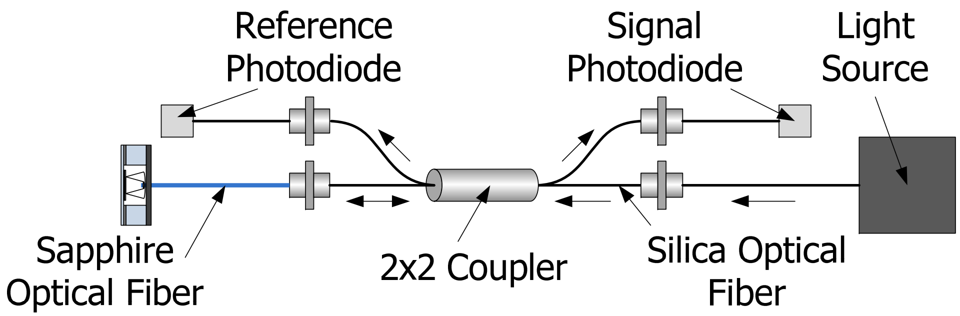
- Common fiber optic measurement techniques
  - Phase modulation – interferometer
    - Pros
      - High sensitivity
    - Cons
      - Environmental sensitivity
      - Coherent source
      - Single mode fibers
  - Intensity modulation – optical lever
    - Pros
      - Simple/robust fabrication
      - Incoherent source
      - Single or multimode fibers
    - Cons
      - Less sensitive

Proof-of-Concept Device (UF)

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





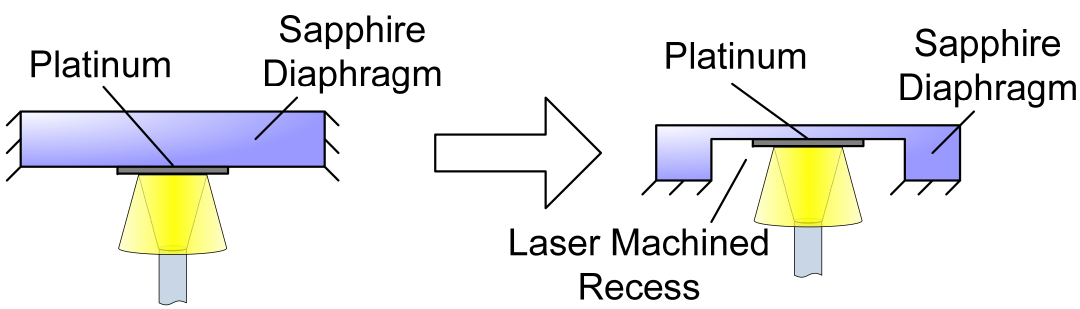


D. Mills et al, *Proc. SPIE*, vol. 9113, Apr 2014

Proof-of-Concept Device

- Performance issues
  - High stiffness – low sensitivity
  - ~300 MPa Residual stress

- Proposed Improvements
  - Increased sensitivity – ultrashort pulse laser micromachining



  - Residual stress – characterize thermocompression bonding

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

Technical Objectives

- Novel sapphire fabrication processes
  - Subtractive machining: ultrashort pulse laser
  - Additive manufacturing: spark plasma sintering
- Characterize and mitigate thermo-mechanical damage
- Fabricate, package, calibrate, and demonstrate sapphire pressure sensor

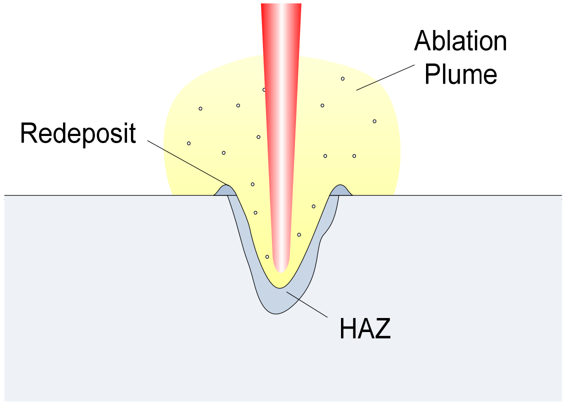
Technical Objectives

- Phase I
  - Laser machining process development
  - SPS thermocompression bonding process development
  - Laser machining thermal damage modeling & analysis
- Phase II
  - Sensor design & fabrication
  - High-temperature packaging
- Phase III
  - Room- and high-temperature characterization
  - Hot jet testing

Previous Work – Pulsed Laser Micromachining (UF)

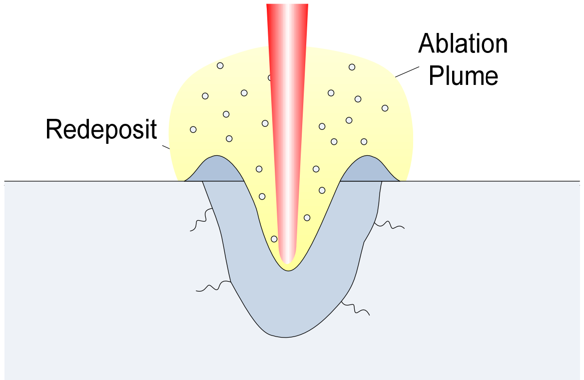
- Ultrashort Pulsed Laser Machining
  - Thermal diffusion depth less than optical penetration depth
  - Reduced damage, redeposit

Ultrashort Pulsewidths

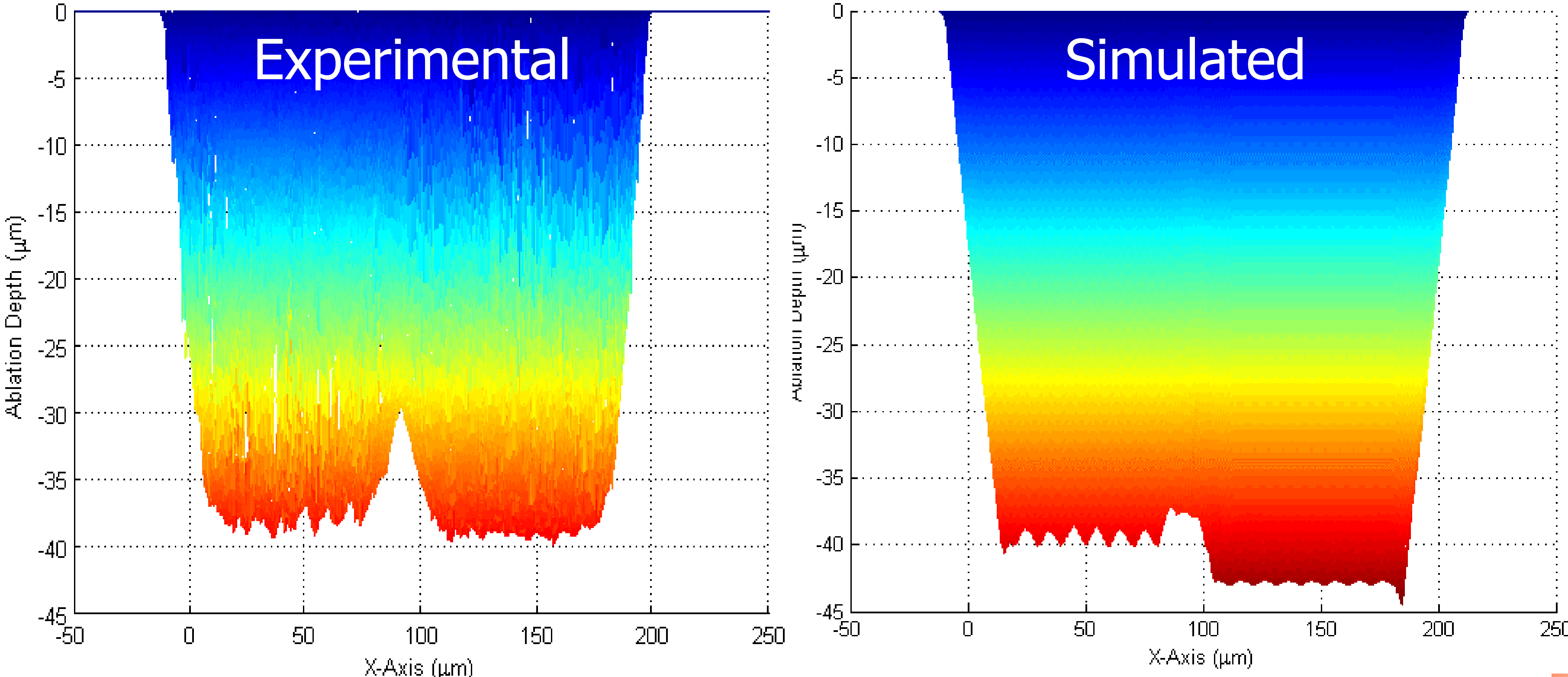


- Four key machining parameters:
  1. Pulse spacing (μm)
  2. Pulse repetition rate (Hz)
  3. Pulse fluence (J/cm^2)
  4. Cut passes (#)

Long Pulsewidths



Previous Work – Pulsed Laser Micromachining (UF)



- Higher fluence, number of passes reduces sidewall angle
- Increasing passes in a region of pulse overlap improves depth uniformity
- Ablation type dependent on laser fluence and pulses/area

Outline

- Introduction
- Laser Ablation Modeling
  - Model
  - Model Validation
- Thermal Damage Analysis
- Thermocompression Bonding
- High Temperature Testing Facility
- Conclusion

Laser Ablation Modeling (FSU)

- One dimensional model approximation
  - Scalar order parameter governing electron density

$$\rho(x,t) = \sum_{\alpha} \sqrt{y_i^{\alpha}(x,t)\, y_i^{\alpha}(x,t)}$$

  - Balance law governing ρ(x,t) obtained from minimization of energy functions
  - Leads to a phase field or sharp interface model driven by electric field (laser) pulses
- Key governing equations

Multi-well energy

$$\frac{\partial E}{\partial x} = -\kappa(\rho) E$$

Electromagnetic nonlinear Beer's Law

$$\beta \frac{\partial \rho}{\partial t} = -\frac{\partial \psi}{\partial \rho} - qE$$

Sharp interface based order parameter model

Laser Ablation Modeling

- Material physics modeling of laser ablation
  1. Laser input: time dependent Maxwell's equations
  2. Material evolution: electronic structure balance equation[1]
- Different light-matter constitutive relations[2]

Standard Force Model

Light attenuation depends on electronic structure

$$\kappa(\rho) = \kappa(\rho; \kappa_1, \kappa_2)$$

Parameters are independent of each other

Coupled Force Model

Couples light attenuation to total charge and damping

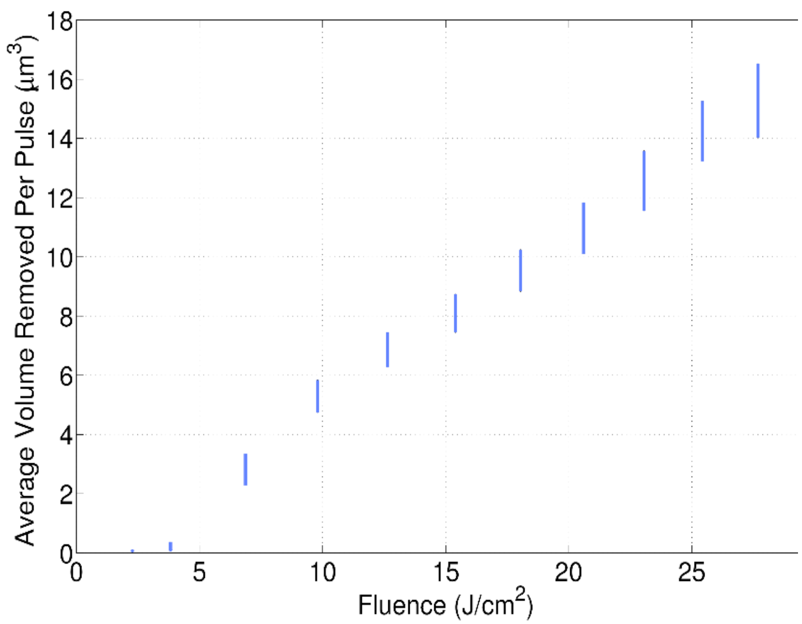
$$\kappa = \kappa(\beta, q)$$

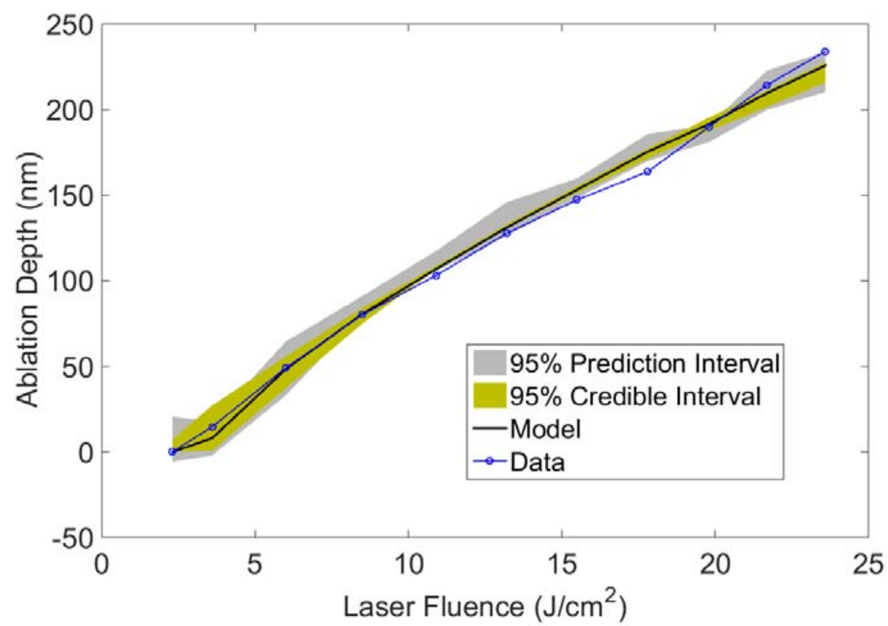
Total charge depends on electronic structure

$$q(\rho) = q(\rho; q_1, q_2)$$

1. Nelson, D., Phys. Rev. A, v. 44(6), 1991.
2. Woerner, P. et al., AIAA SciTech, 2016.

Model Validation (UF/FSU)





- Ablation of material predicted as a function of picosecond pulsed laser excitation
- Laser intensity dependence model parameters identified via Bayesian statistics

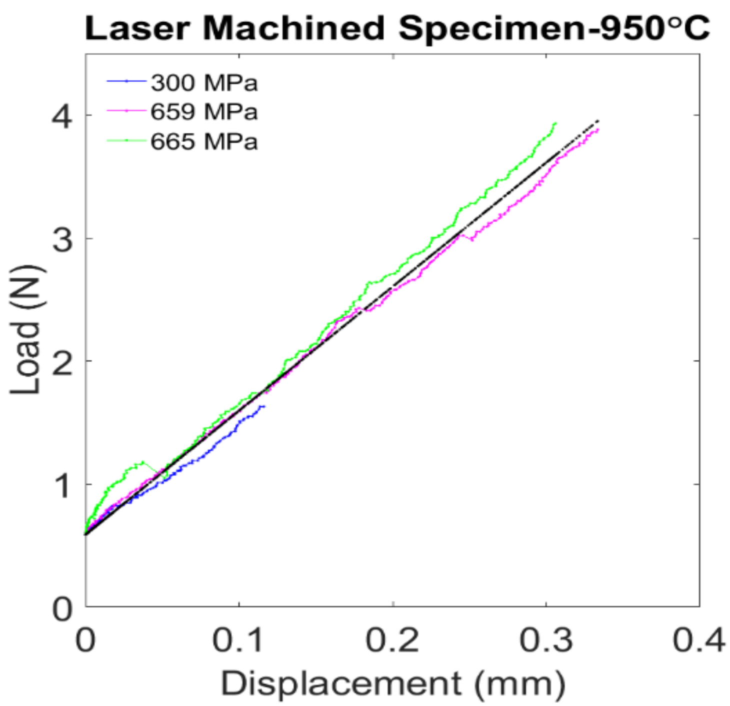
*Daniel Blood, "Simulation, Part Path Correction, and Automated Process Parameter Selection for Ultrashort Pulsed Laser Micromachining of Sapphire", University of Florida, PhD Thesis, directed by Profs. M. Sheplak & T. Schmitz, 2014.

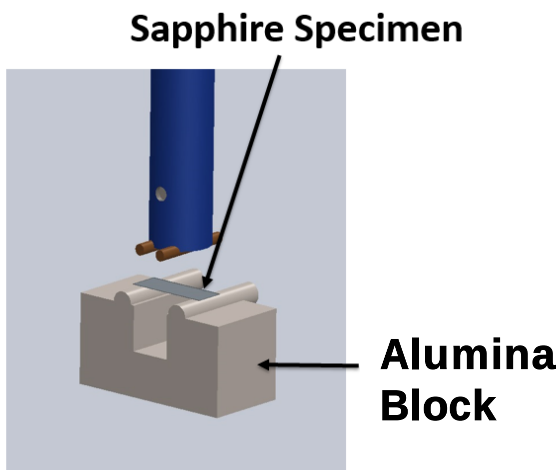
Outline

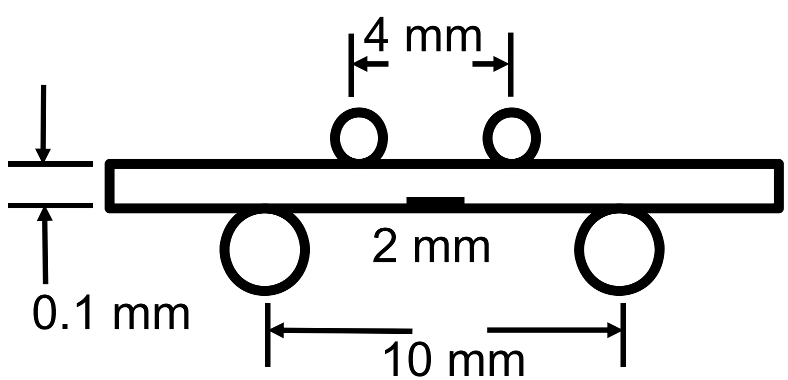
- Introduction
- Laser Ablation Modeling
- Thermal Damage Analysis
  - Four point bend bar test
  - Flexural strength
- Thermocompression Bonding
- High Temperature Testing Facility
- Conclusion

Thermal Damage Analysis (FSU)

- Four point bend bar test for flexural strength
  - Pristine, laser machined (6 × 16 × 0.1)
  - 0.02 mm × 2 mm notch at neutral axis
  - 25°C, 950°C, 1300°C

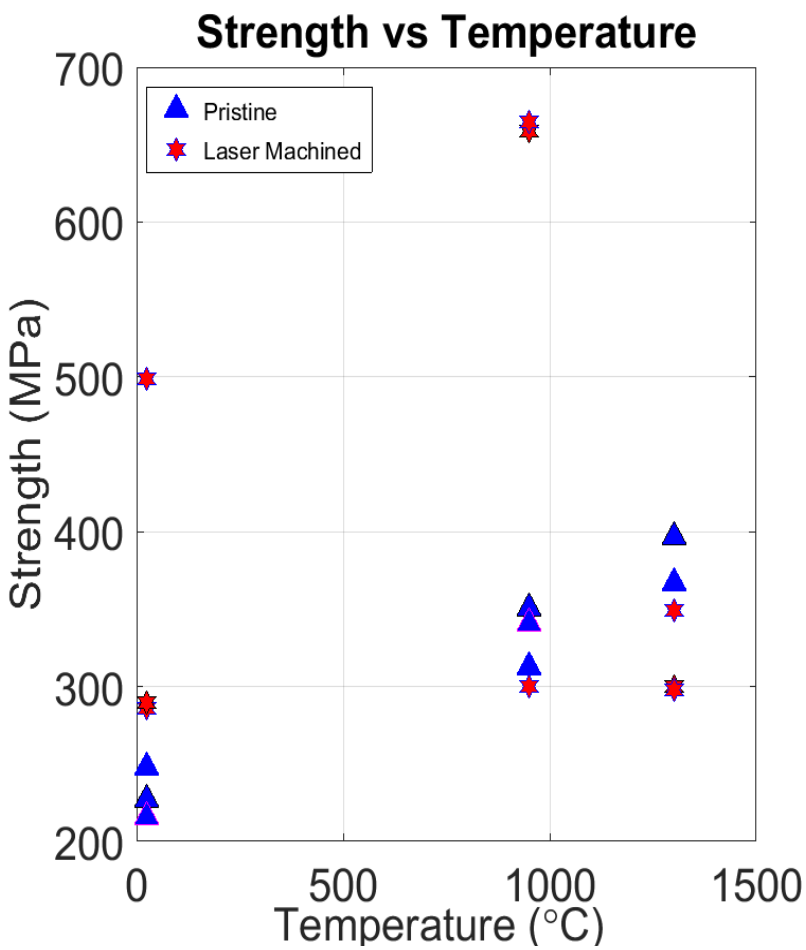






Thermal Damage Analysis

- Preliminary results:
  - Pristine specimen strength increase
  - Machine specimen strength inconsistent
- Further evaluation necessary



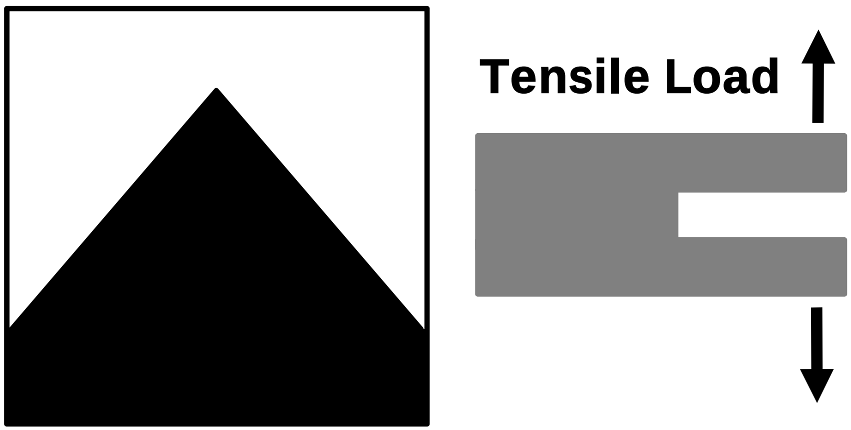
Outline

- Introduction
- Laser Ablation Modeling
- Thermal Damage Analysis
- Thermocompression Bonding
  - Bond characterization
  - Laser machining
- High Temperature Testing Facility
- Conclusion

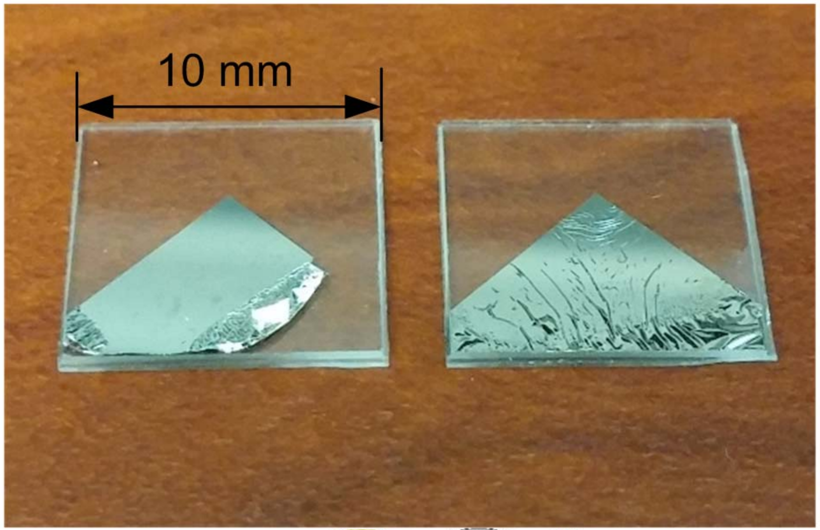
Thermocompression Bonding – Characterization (UF)

- Chevron test for bond strength characterization
  - Increasing tensile load
  - Chevron shape nucleates brittle failure
- Conventional platinum lift-off process unsuccessful

Chevron test

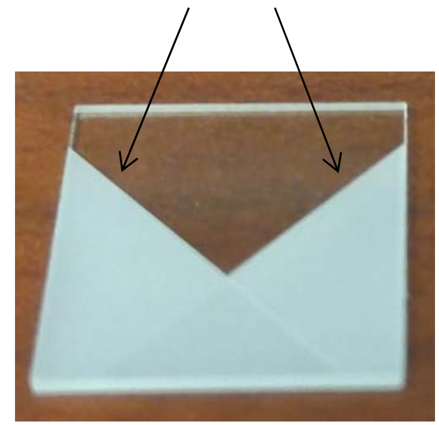


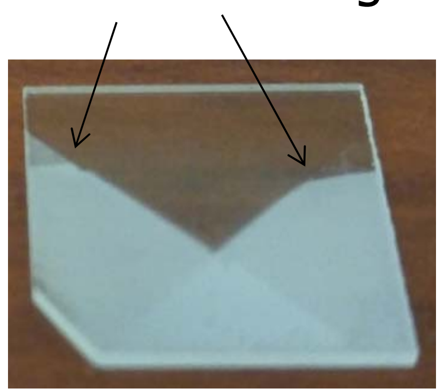
Failed lift-off technique



Thermocompression Bonding – Laser Machining (UF)

- Laser machine chevron shape
  - Deposit platinum
  - Eliminates lift off process
- High power machining
  - Redeposit buildup
  - Additional roughness
- Low power machining
  - Inconsistent cut depth



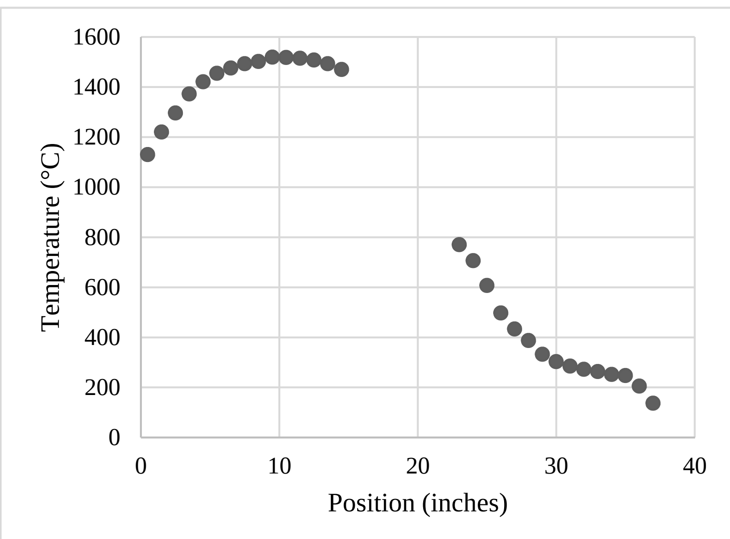


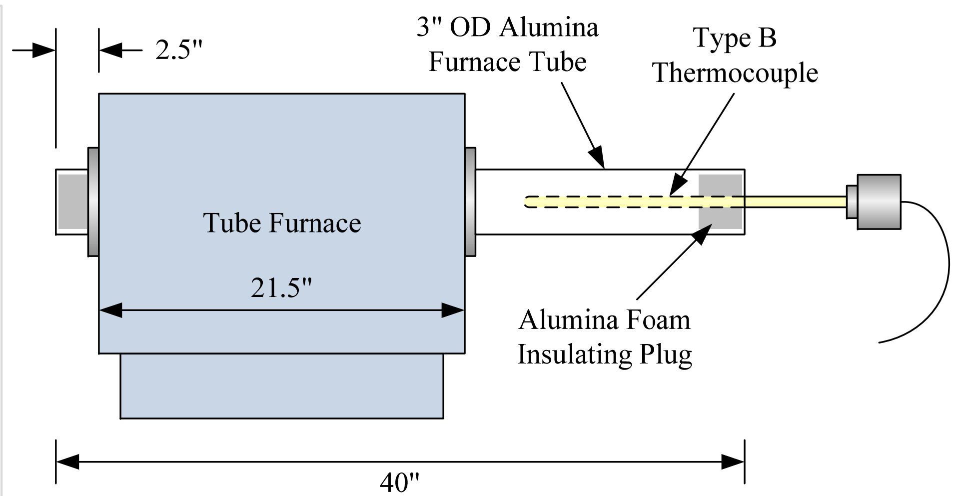
Outline

- Introduction
- Laser Ablation Modeling
- Thermal Damage Analysis
- Thermocompression Bonding
- High Temperature Testing Facility
- Conclusion

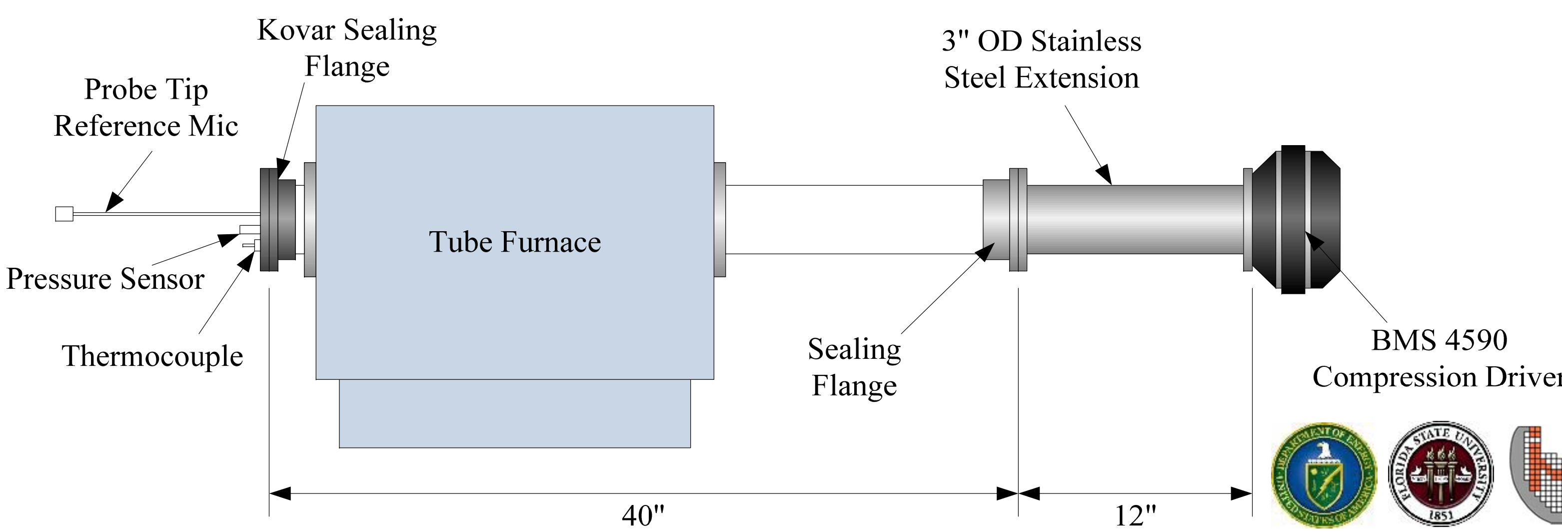
High Temperature Testing Facility – Temperature Profile

- Temperature profile at 1550°C
  - Establish temperature limits
  - >1000° C at sensor
  - Removable external mounts





High Temperature Testing Facility



Outline

- Introduction
- Laser Ablation Modeling
- Thermal Damage Analysis
- Thermocompression Bonding
- High Temperature Testing Facility
- Conclusion
  - Summary
  - Future work

Summary

- Laser machining characterized
  - Simulations validated
- Laser ablation model validated
  - Agreement with empirical data
- High temperature plane wave tube operational
  - Temperature profile
  - Mounting assembly
- Bonding characterization method established

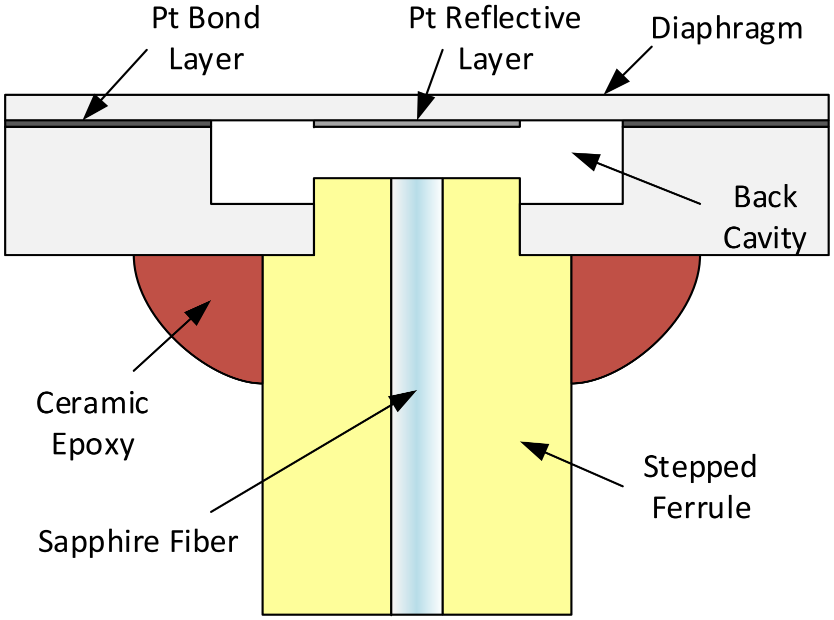
Future Work

- Resolve laser troubles
  - In talks with Oxford Lasers
- Extend laser ablation model for sub-surface laser damage
  - Strength, fracture
- Sensor fabrication
  - Optimal sensor design
- High-temperature package development
- Packaged sensor calibration
  - Hot jet testing

Questions?

Contingency Sensor Design

- Non-optimal sensor design
  - conventional machining
  - 30-40 μm thickness substrates
  - Stepped tip optical ferrules
  - Larger back cavity



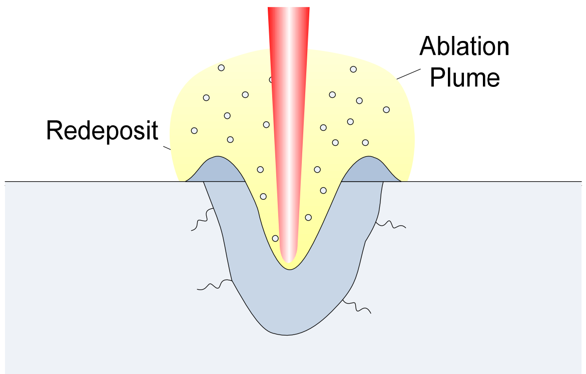
Model Analysis–Global Sensitivity(FSU)

- Global sensitivity analysis using Morris sampling identifies β as the most sensitive parameter and κ_1 as insensitive.

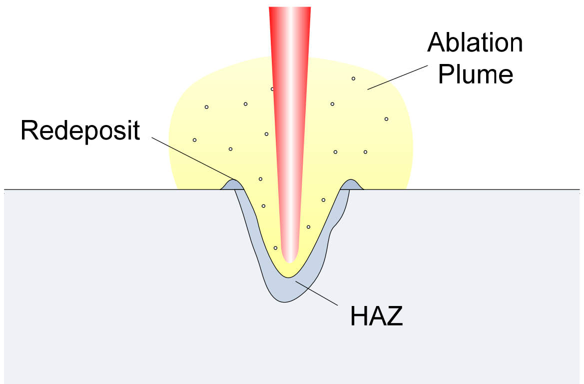
- Parameters considered:

$\kappa(\rho) = \sigma(\rho; \kappa_1, \kappa_2)$	Electromagnetic attenuation factor: κ_1 (room temperature) κ_2 (excited state)
β	Inverse electron mobility parameter
q	Total electric charge

Previous Work – Pulsed Laser Micromachining



Long Pulsewidths



Ultrashort Pulsewidths

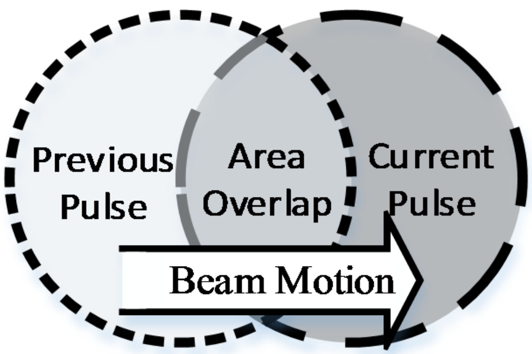
- Ultrashort pulse laser micromachining
  - Classification based on relation between thermal diffusion depth, *d*, and optical penetration depth, δ

$$d = 2\sqrt{\frac{k\tau}{\rho c_p}} \qquad \delta = \frac{2}{\alpha}$$

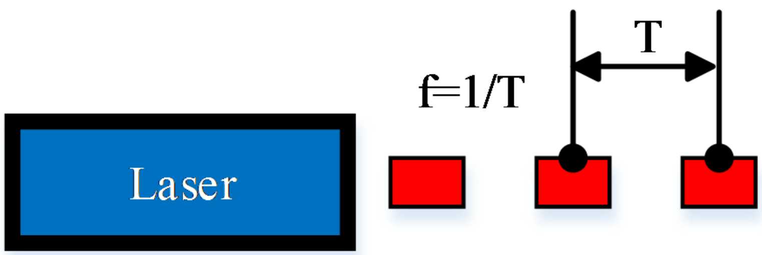
  - $d < \delta$, material removal is dominated by photochemical processes and is considered ultrashort

Pulsed Laser Micromachining

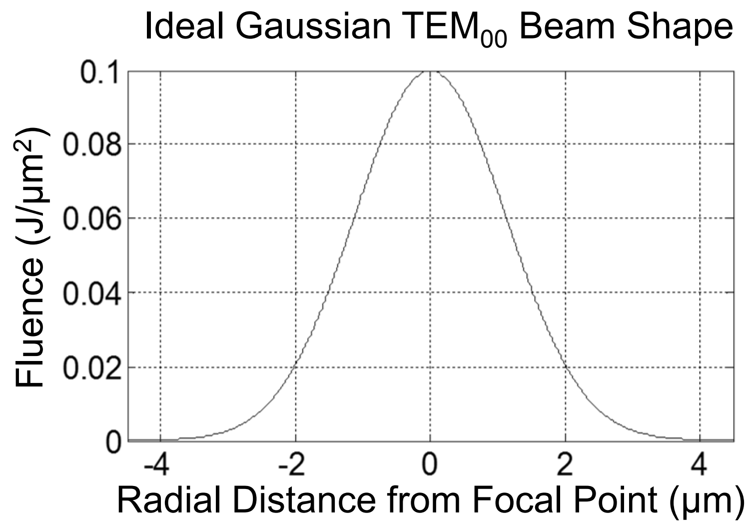
- Four key machining parameters of interest:



1. Pulse Spacing (μm)



2. Pulse Repetition Rate (Hz)

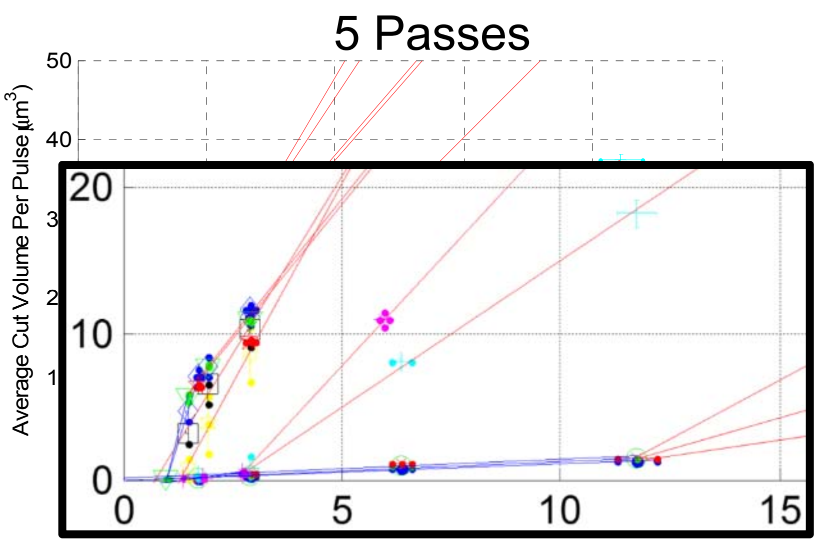


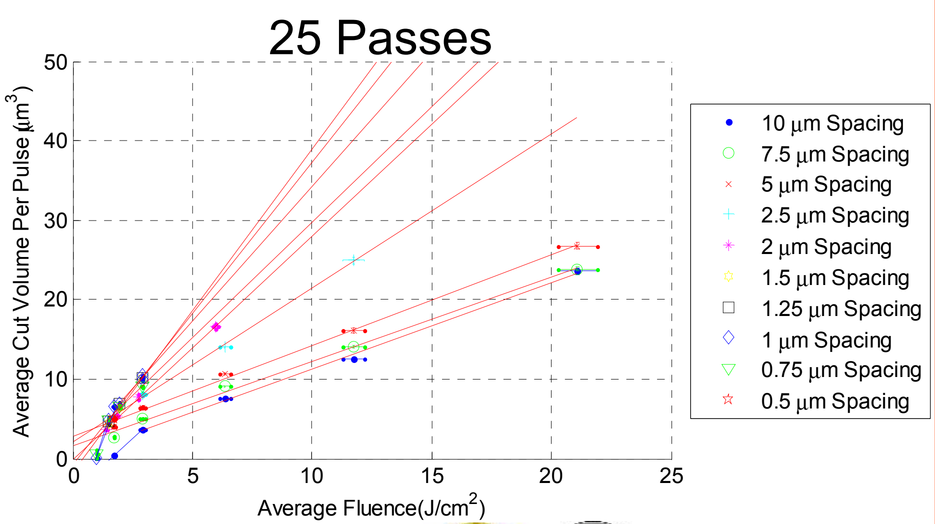
3. Pulse Fluence (J/cm^2)

4. Cut Passes – Number of times the cut path is repeated

Gentle vs. Strong Ablation

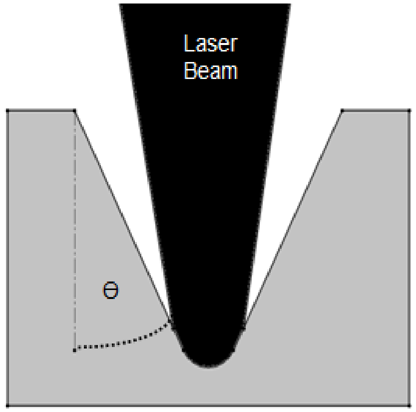
- Transition from gentle to strong ablation is dependent on the number of laser pulses in a given area and the laser fluence
- Machining parameters
  - Feature size: 400 µm x 250 µm
  - Laser fluence: 1.2 – 21.5 J/cm^2
  - Number of passes: 1-50
- Linear fits to gentle (blue) and strong (red) ablation regimes
- Threshold laser fluence: ~1 J/cm^2

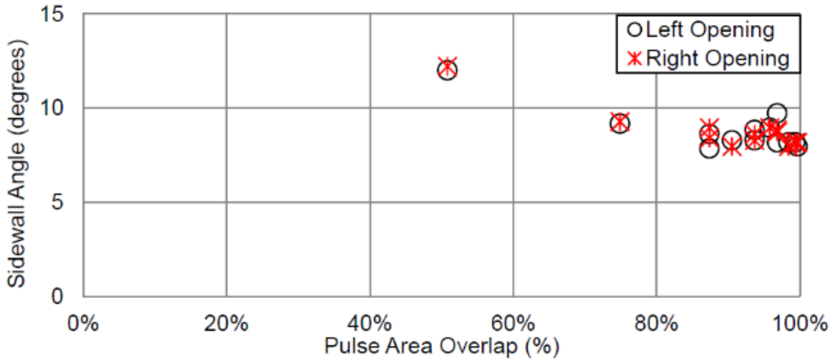


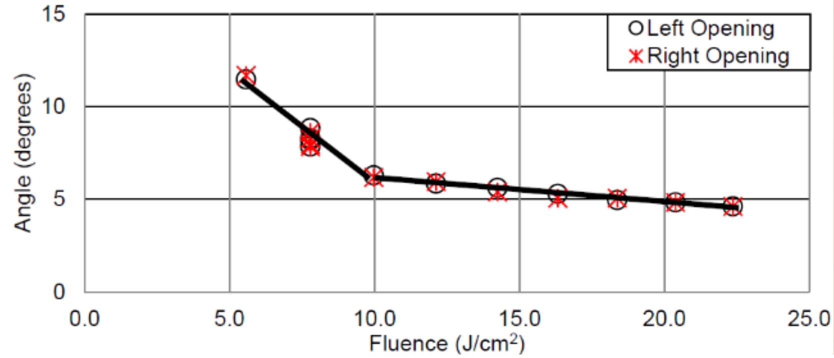


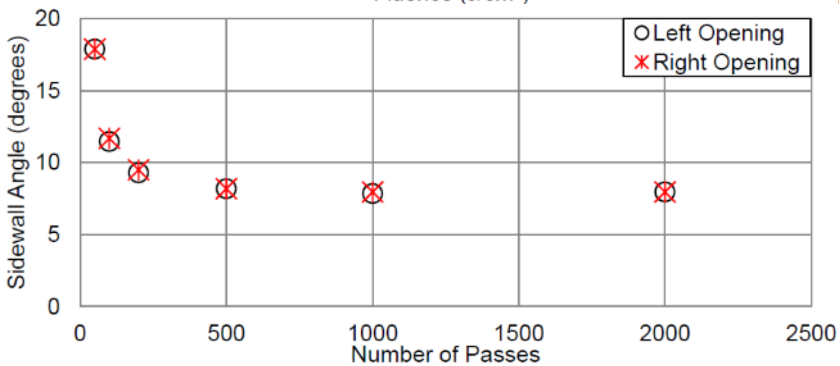
Sidewall Angle

- Machining parameters
  - Fluence: 5.1-25.5 J/cm^2
  - Pulse area overlap: 45-99%
  - Number of passes: 50-2000
- Sidewall angle is constant above ~75% pulse area overlap
- Higher fluence and number of passes reduce sidewall angle



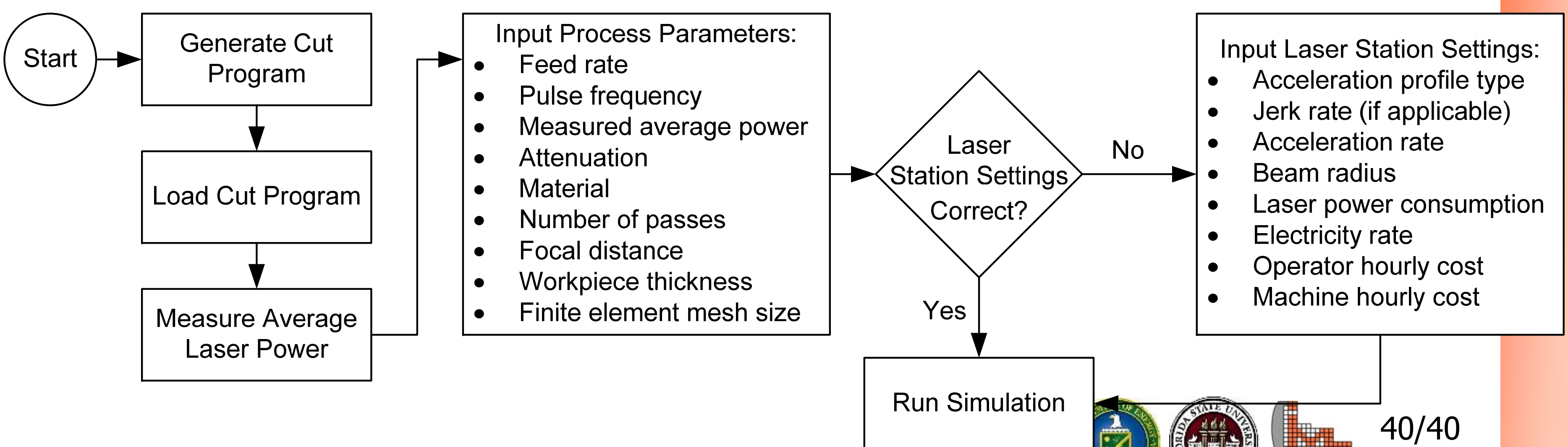




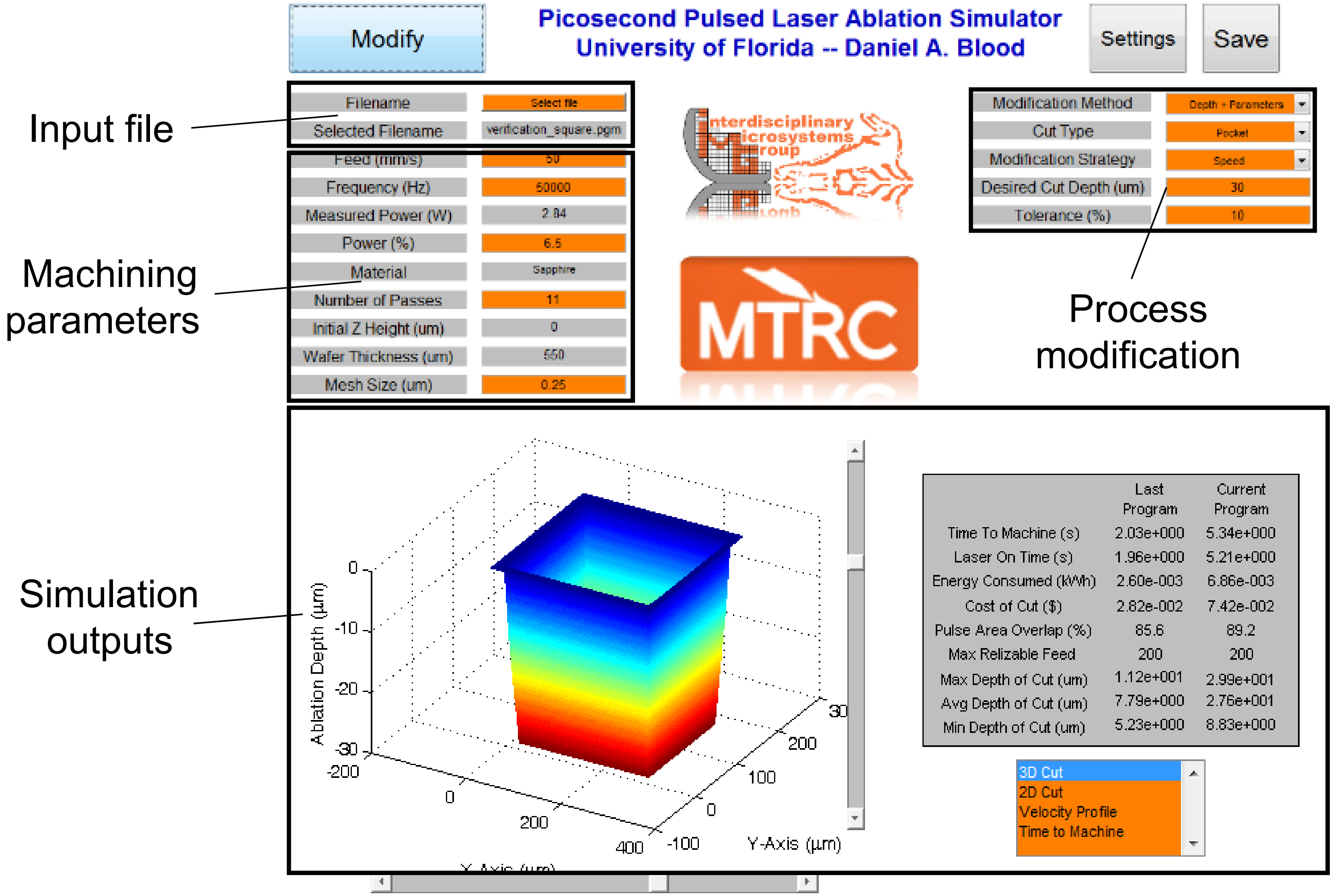


Laser Machining Simulation

- User inputs
  - Cut program (G code)
  - Process parameters
  - Laser station settings
- Program outputs
  - Results table
  - 2D and 3D simulated depth of cut plots
  - 2D velocity plot
  - Input feedrate vs machining time plot

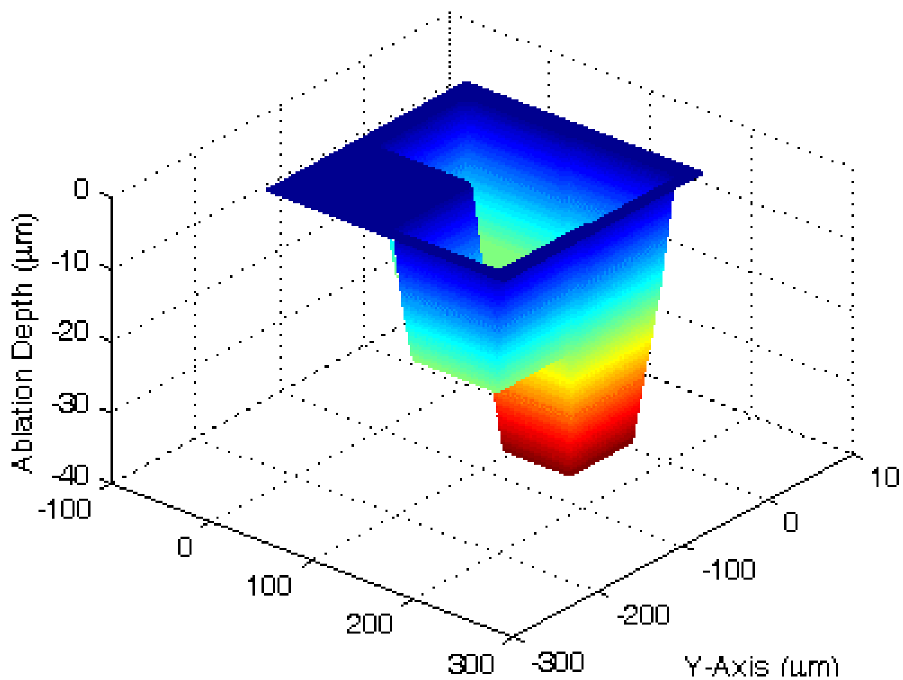


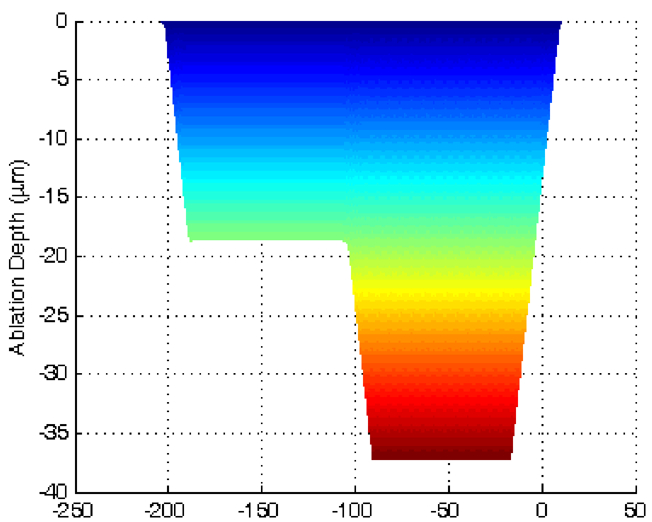
Laser Machining Simulation



Part Path Modification

- Test geometry – overlapping rectangles
  - Creates deeper machined region
  - Goal: add passes in specific areas to create a single region of consistent depth

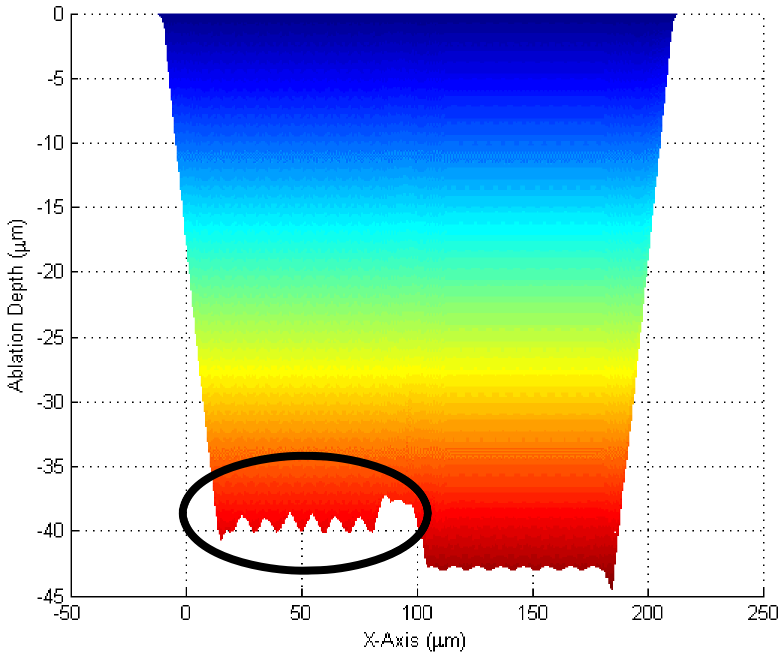




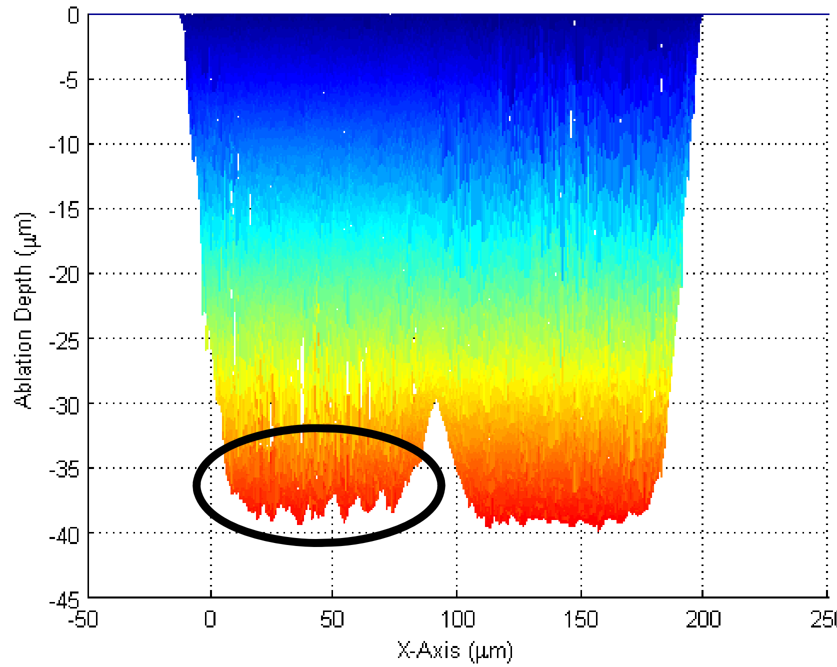
Part Path Modification Results

- Additional passes in region of single overlap improves the depth uniformity
- Good agreement with simulation including capture of periodic structures in the machined recess

Simulated



Measured



Laser Ablation Modeling

- Material physics modeling of laser ablation
  1. Laser input: time dependent Maxwell's equations
  2. Material evolution: electronic structure balance equation

Lagrangian energy formulation

$$L = L_F + L_I + L_M$$

Free space — Electronic interactions — Kinetic & stored energy

Energy losses to ablation

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

y_i^{α} --vector order parameters (α=1,…n) defining homogenized electronic structure

Nelson, D., Phys. Rev. A, v. 44(6), 1991.
Nelson, D., *Electric, Optic, and Acoustic Interactions in Dielectrics*, Wiley, 1979.

Laser Ablation Modeling

- One dimensional model approximation
  - Scalar order parameter governing electron density

$$\rho(x,t) = \sum_{\alpha} \sqrt{y_i^{\alpha}(x,t)\, y_i^{\alpha}(x,t)}$$

  - Balance law governing ρ(x,t) obtained from minimization of energy functions
  - Leads to a phase field or sharp interface model driven by electric field (laser) pulses
- Key governing equations

Multi-well energy

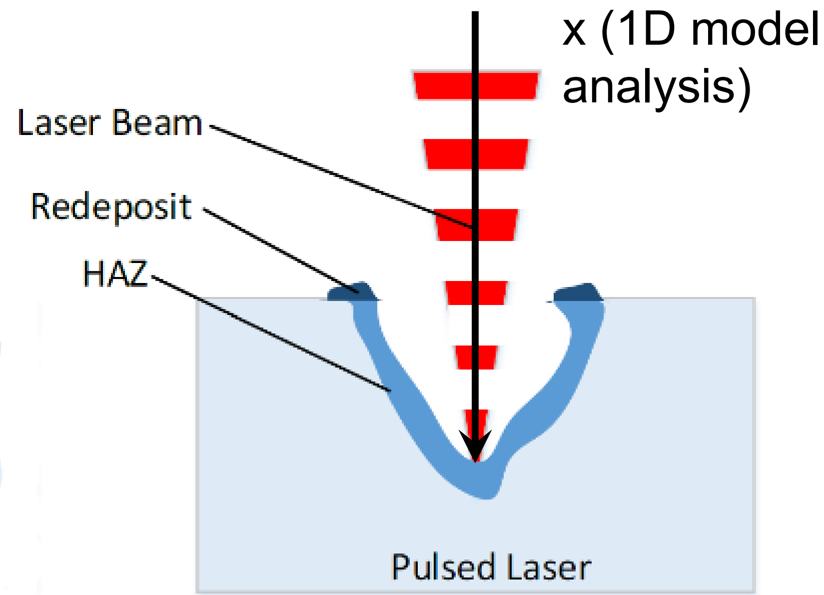
$$\sigma(\rho)\mu_0 \frac{\partial E}{\partial t} = \nabla^2 E$$

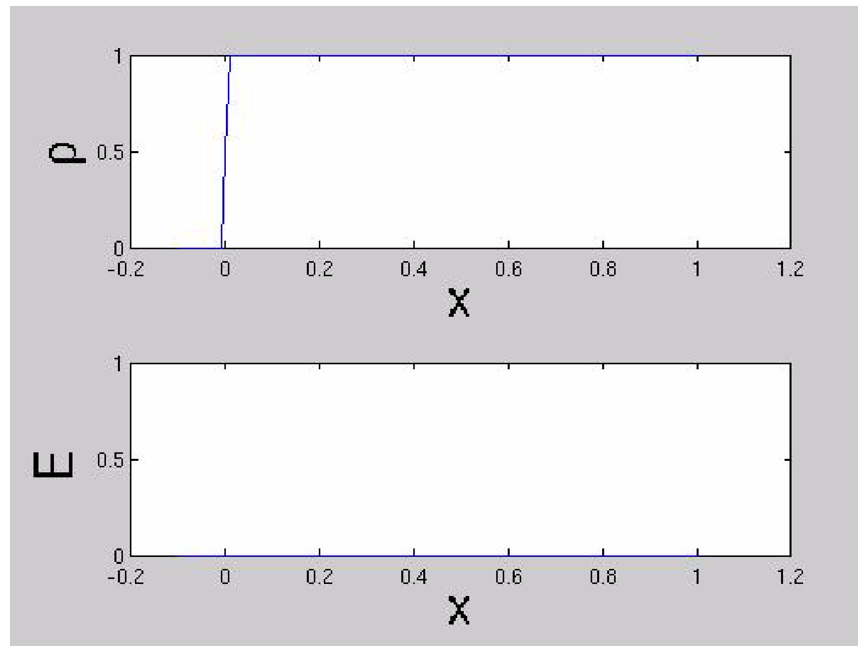
Electromagnetic equation

$$\beta(E)\frac{\partial \rho}{\partial t} = a_0 \nabla^2 \rho - \frac{\partial \psi}{\partial \rho} - \gamma(E)$$

Phase field based order parameter model

Model Validation





- Ablation of material predicted as a function of picosecond pulsed laser excitation
- Laser intensity dependence model parameters identified via Bayesian statistics

*Daniel Blood, "Simulation, Part Path Correction, and Automated Process Parameter Selection for Ultrashort Pulsed Laser Micromachining of Sapphire", University of Florida, PhD Thesis, directed by Profs. M. Sheplak & T. Schmitz, 2014.

Model Analysis – Parameter Sensitivity

$$\sigma(\rho)\mu_0 \frac{\partial E}{\partial t} = \nabla^2 E$$

<u>Electromagnetic equation</u>

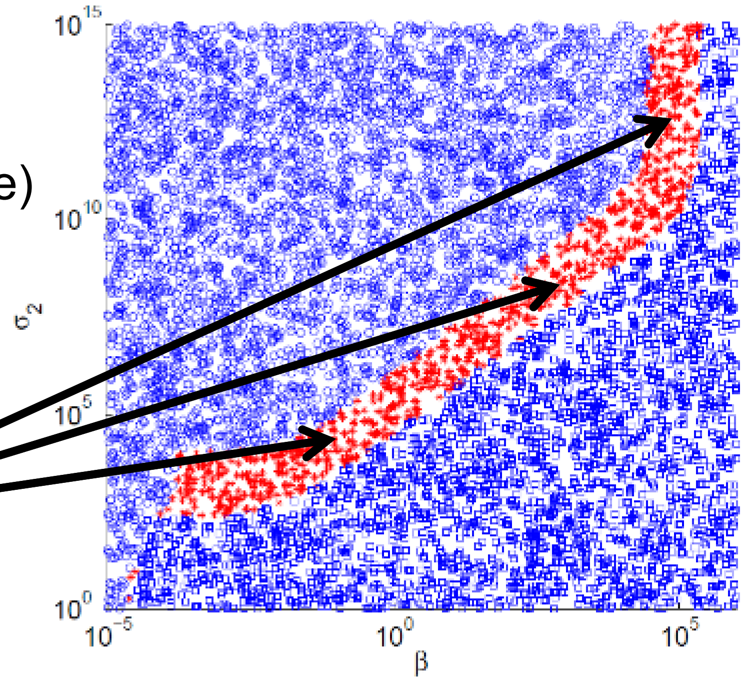
$$\beta(E)\frac{\partial \rho}{\partial t} = a_0 \nabla^2 \rho - \frac{\partial \psi}{\partial \rho} - \gamma(E)$$

<u>Phase field based order parameter model</u>

- Critical parameters considered

$\sigma(\rho) = \sigma(\rho; \sigma_1, \sigma_2)$ Electric conductivity:
σ_1 (room temperature)
σ_2 (excited state)

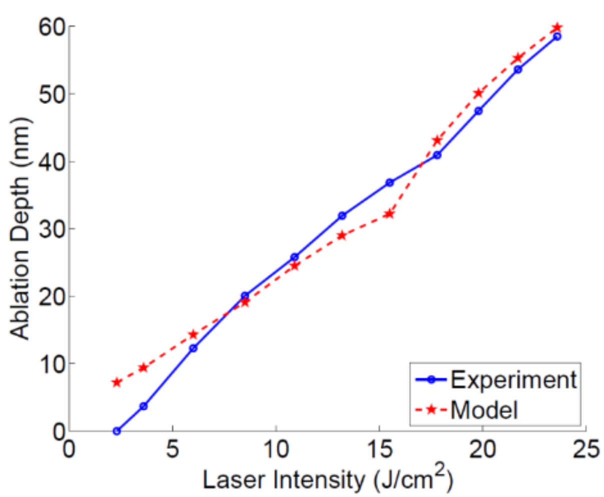
$\beta(E)$ Inverse electron mobility parameter

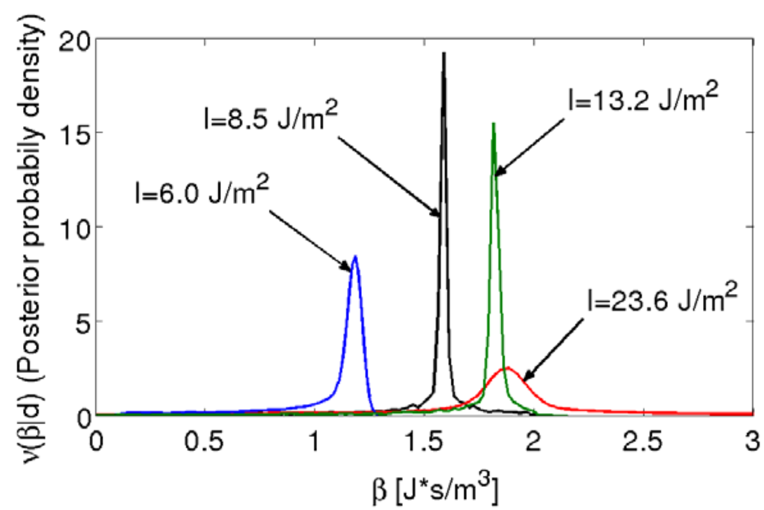


Region of <u>finite</u> machined depth giving potentially valid numerical correlation with laser ablation experiments

Model Analysis – Uncertainty Quantification

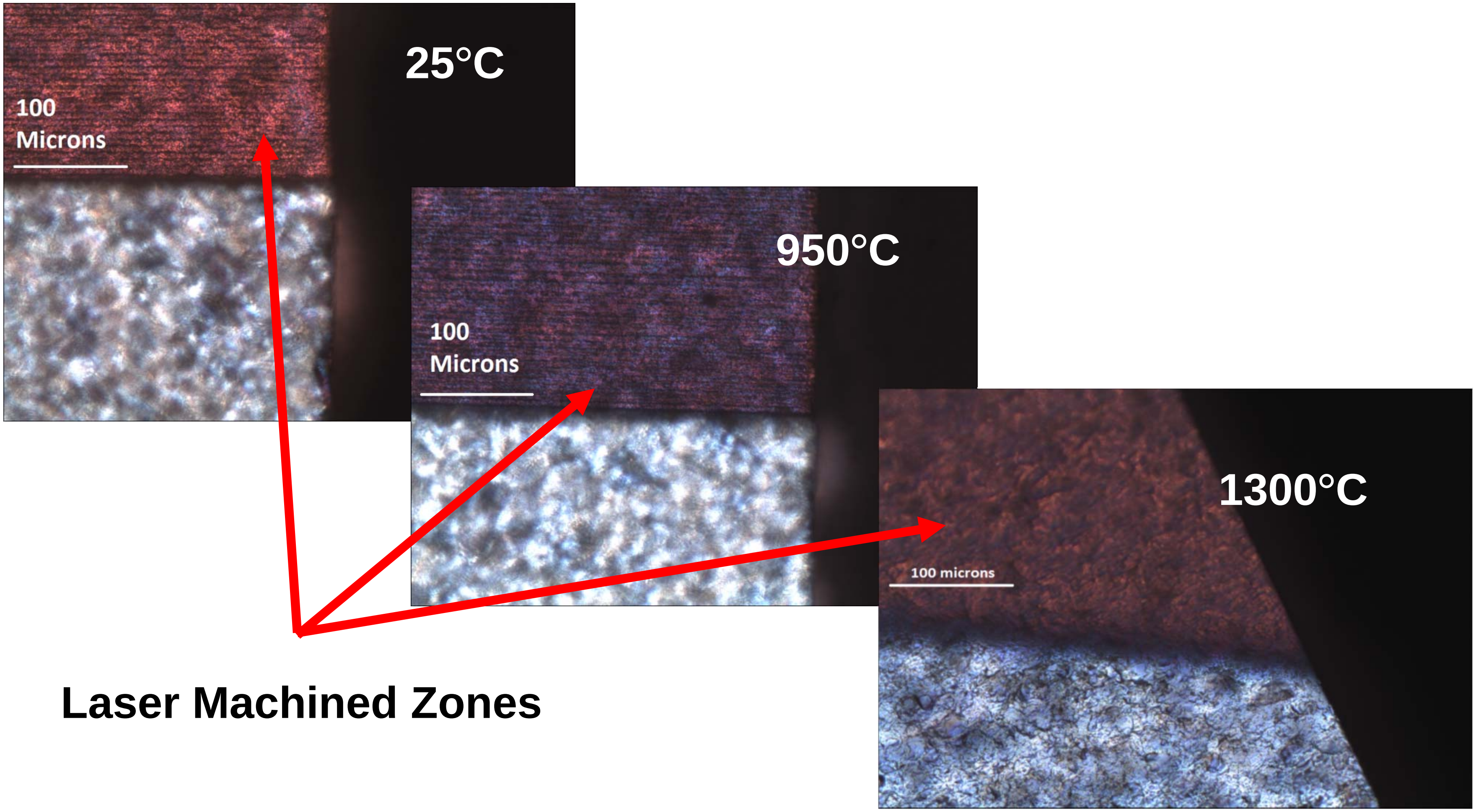
- Bayesian statistics applied to quantify reduced order model uncertainty
  - Kinetic parameter (β) found to increase approximately linearly with picosecond pulsed laser intensity
  - Illustrated in terms of the probability of β given a machined depth *d*





*Daniel Blood, "Simulation, Part Path Correction, and Automated Process Parameter Selection for Ultrashort Pulsed Laser Micromachining of Sapphire", University of Florida, PhD Thesis, directed by Profs. M. Sheplak & T. Schmitz, 2014.

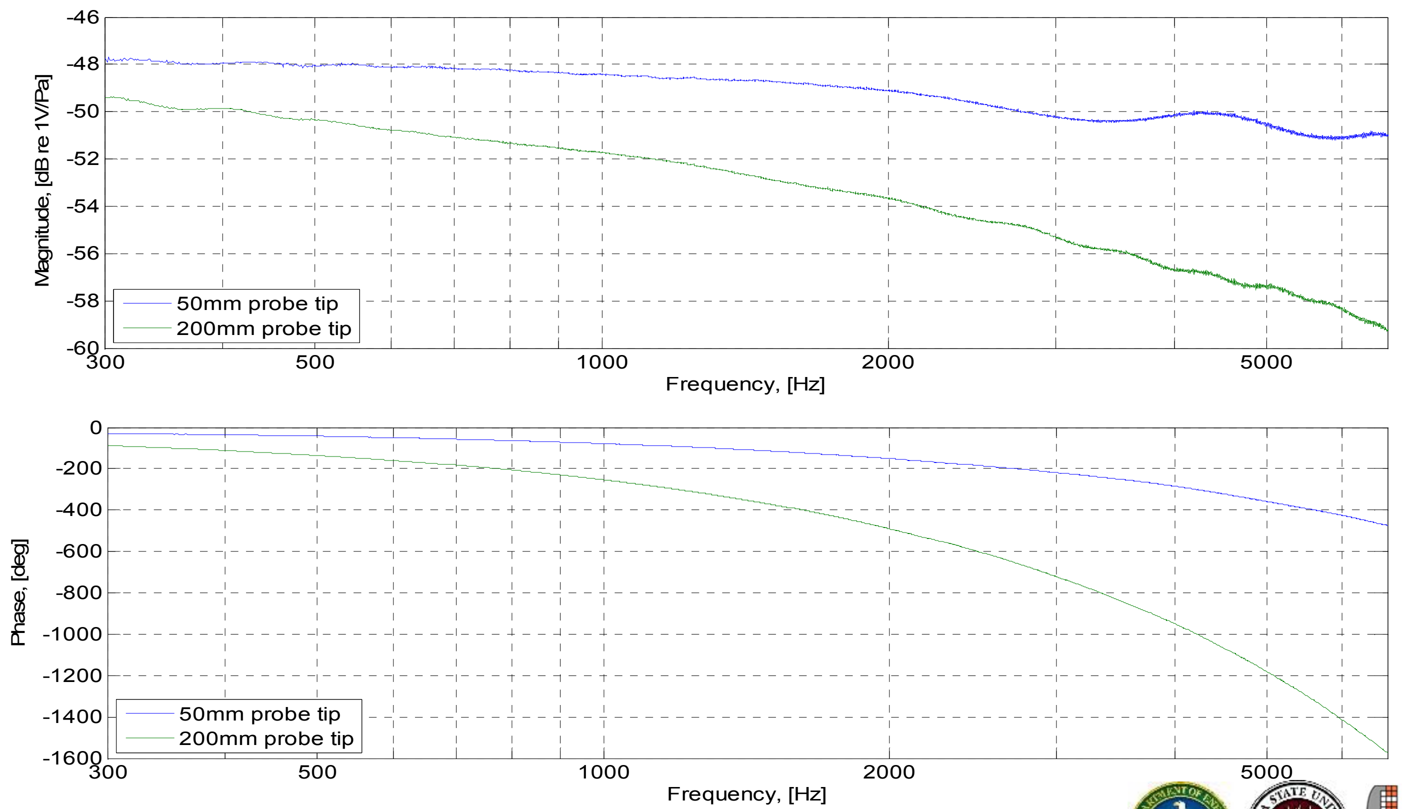
Birefringence Characterization



Laser Machined Zones

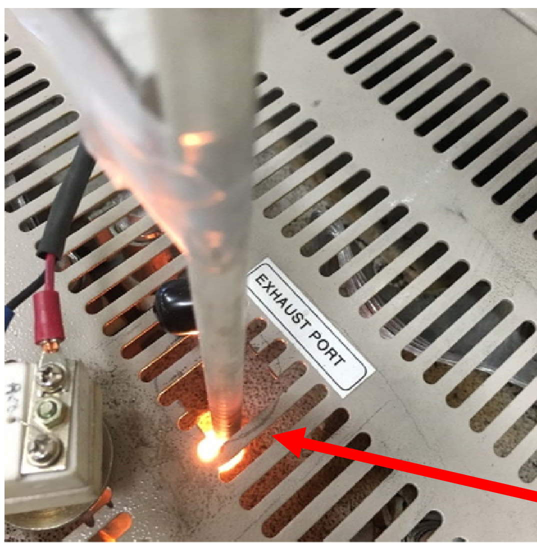
High Temperature Testing Facility – Probe Reference Mic

- Brüel & Kjær probe tip microphone selected for reference
- Smoot FRF out to 6.7 kHz in acoustic plane wave tube



Experimental Setup

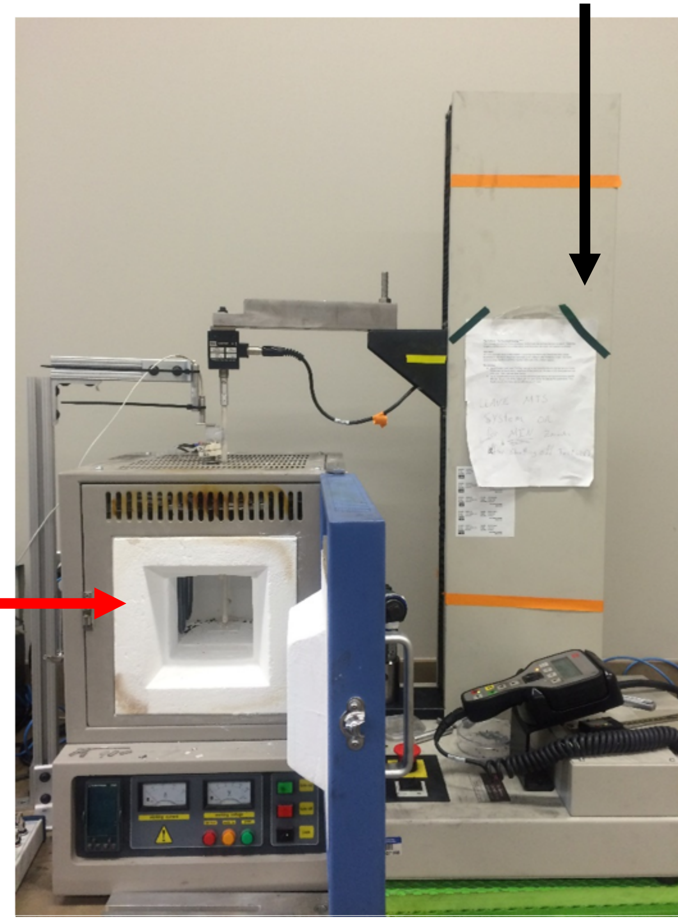
- Box furnace integrated with a 1kN MTS load frame
- Flexural strength measurements
  - Quantify affect of laser machining



Exhaust port

Box furnace (1600°C)

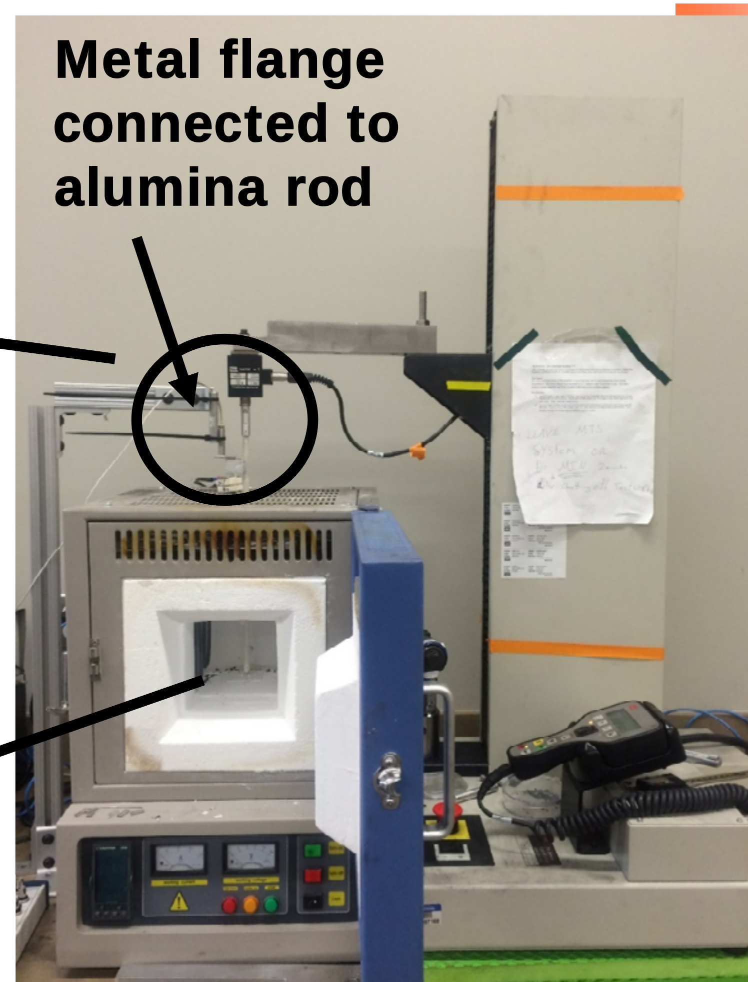
MTS-1kN

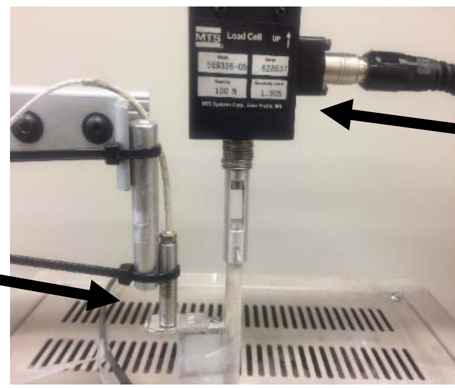


Bend Bar Configuration

- System compliance minimized using non-contact capacitor probe

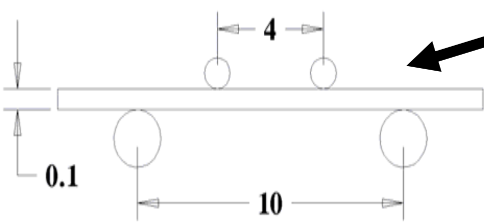
Metal flange connected to alumina rod

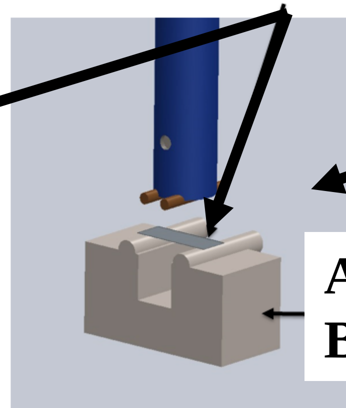




Capacitor Probe

Sapphire Specimen





Alumina Block