High-Temperature Sapphire Pressure Sensors for Harsh Environments

David Mills[1], Daniel Blood[1,3], Harman Singh Bal[2], Peter Woerner[2], William Oates[2],Mark Sheplak[1]

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

DE-FE0012370
2015 NETL Crosscutting Research Review Meeting
April 30, 2015

Outline

- Introduction
- Approach
- Proof-of-Concept Device
- Objectives
- Ultrashort Pulse Laser Micromachining
- Laser Ablation Modeling
- Conclusions

Project Overview

- Focus: Development of novel fabrication methods for the synthesis of high-temperature sapphire optical pressure sensors
- Award information
  - Project title: "High-temperature sapphire pressure sensors for harsh environments"
  - Award #: DE-FE0012370
  - Program manager: Sydni Credle
  - Duration: 3 years, beginning Jan 2014
- Project team
  - UF (Project lead)
  - FSU

Motivation

- Development and implementation of advanced energy systems will require novel harsh environment sensors and instrumentation for:
  - Advanced process control/closed loop feedback systems
  - Increased efficiency
  - Reduced emissions & cost
- Applications
  - Coal gasification
  - Advanced gas turbine systems
  - 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
- Temperature mitigation techniques:
  - Stand-off tubes - cause signal attenuation and degradation
  - Water cooling - imparts unknown aerothermal effects on the surrounding flow

Approach

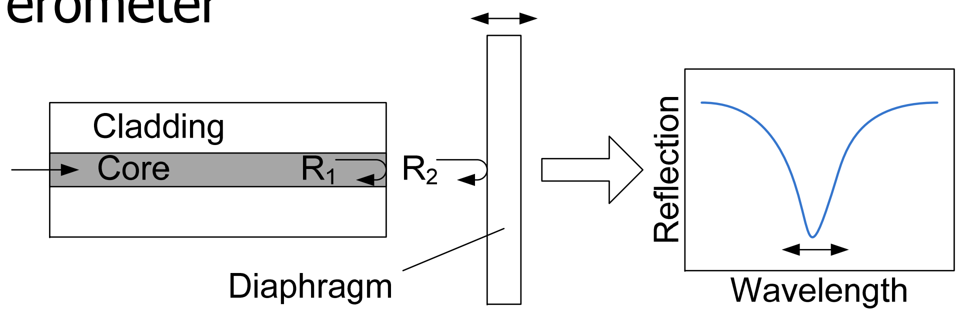
- Transduction mechanisms
  - Capacitive
  - Optical
  - Piezoelectric
  - Piezoresistive
- Benefits of fiber optic transduction
  - DC measurement
  - Immunity to EMI
  - Passive
  - Non-conductive
  - Remote electronics
  - Multiplexing

Approach

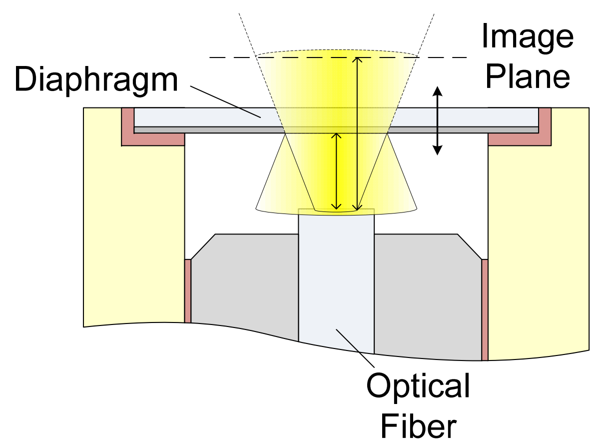
- Sensor/optical fiber materials
  - ~~Silicon~~
  - ~~Silica~~
  - Silicon carbide
  - Sapphire
  - Diamond
- Benefits of sapphire
  - High melting point (2053°C)
  - Resistance to chemical corrosion
  - Excellent hardness
  - Large transmission window (200 nm – 5 μm)
  - Multimode optical fibers &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

Approach

- Common fiber optic measurement techniques
  - Phase modulation – interferometer
    - High sensitivity
    - Environmental sensitivity
    - Coherent source
    - Single mode fibers

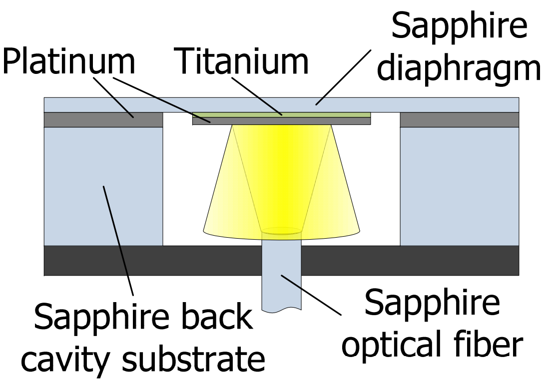


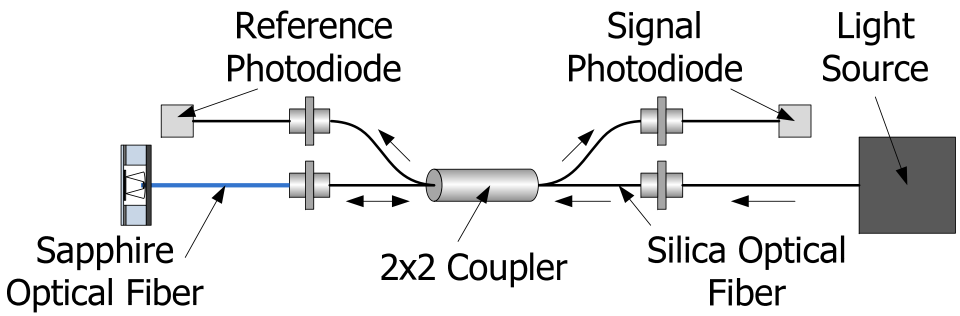
  - Intensity modulation – optical lever
    - Less sensitive to environmental changes
    - Incoherent source
    - Single or multimode fibers
    - Relaxed fabrication/packaging tolerances
    - Multiple send/receive configurations

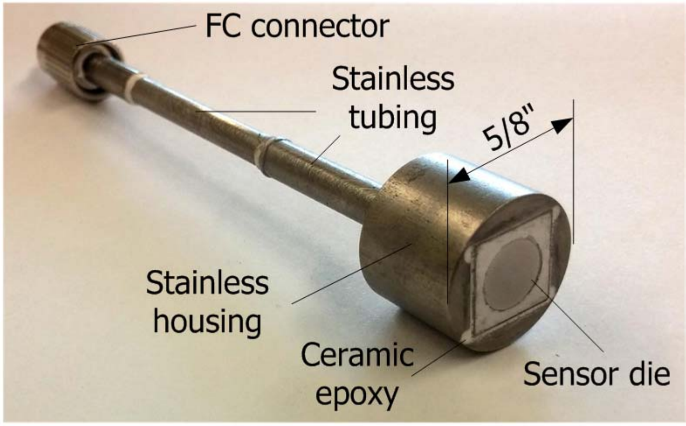


Proof-of-Concept Device

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



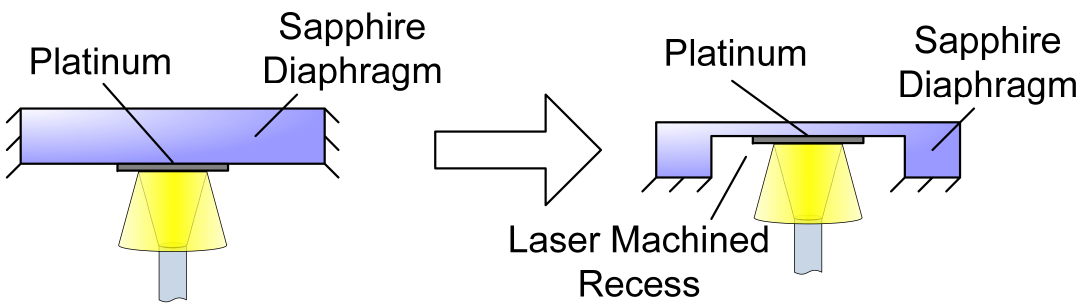




D. Mills et al, *Proc. SPIE*, vol. 9113, Apr 2014

Proof-of-Concept Device

- Performance issues
  - High stiffness – low sensitivity
  - Large residual stress (~300 MPa) resulted in buckled diaphragm
- Improvements
  - Sensitivity – utilize ultrashort pulse laser micromachining to fabricate thinner diaphragm structures



  - Residual stress – improve thermocompression bond process through additional testing and characterization of bond interface

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

Technical Objectives

- Implement novel sapphire fabrication processes for fabrication of 3-dimensional structures
  - Subtractive machining: ultrashort pulse laser micromachining
  - Additive manufacturing: thermocompression bonding via spark plasma sintering (SPS) technology
- Characterize and mitigate thermo-mechanical damage imparted by manufacturing processes via statistical modeling of laser pulse-material interactions
- Fabricate, package, calibrate, and demonstrate in the field a high-temperature sapphire dynamic pressure capable of operation up to 1000°C and 1000 psi

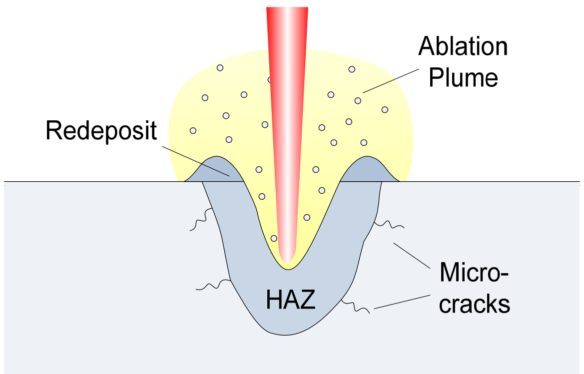
Technical Objectives

- 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

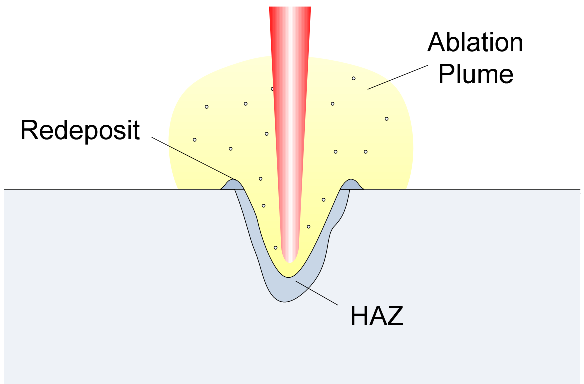
Outline

- Introduction
- Approach
- Proof-of-Concept Device
- Objectives
- Ultrashort Pulse Laser Micromachining
- Laser Ablation Modeling
- Conclusions

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

$$d = 2\sqrt{at} \qquad \delta = \frac{2}{\alpha}$$

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

Pulsed Laser Micromachining

- Oxford Lasers Micromachining Station
  - Laser: Coherent Talisker Ultra (DPSS)
  - Pulse duration: 10-15 ps nominally
  - Wavelength: 355 nm
  - Beam diameter: 8.8 μm
  - Max output: 4 W at laser head (20 μJ pulse energy)
  - Beam attenuator from 0 -100%
  - Pulse frequency: up to 200 kHz

- For sapphire,

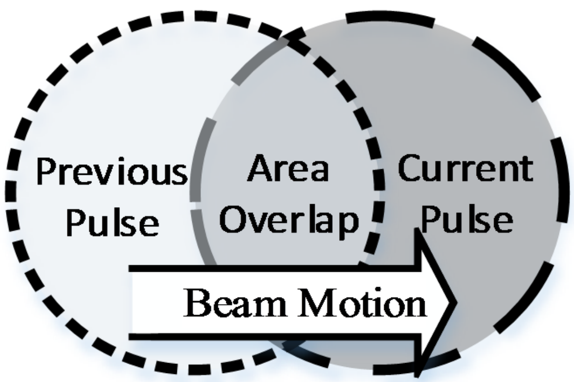
$$\delta \approx 72.4\ \mu m$$

$$d \approx 24.4\ nm$$

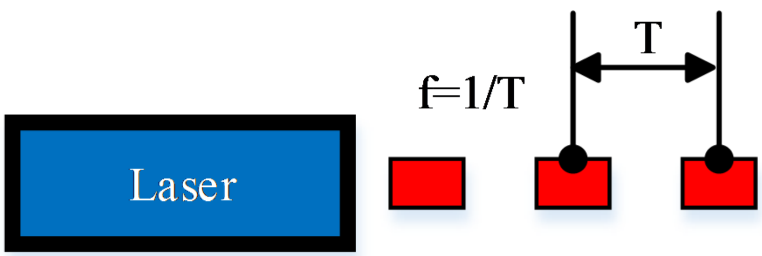
10ps pulse is considered ultrashort

Pulsed Laser Micromachining

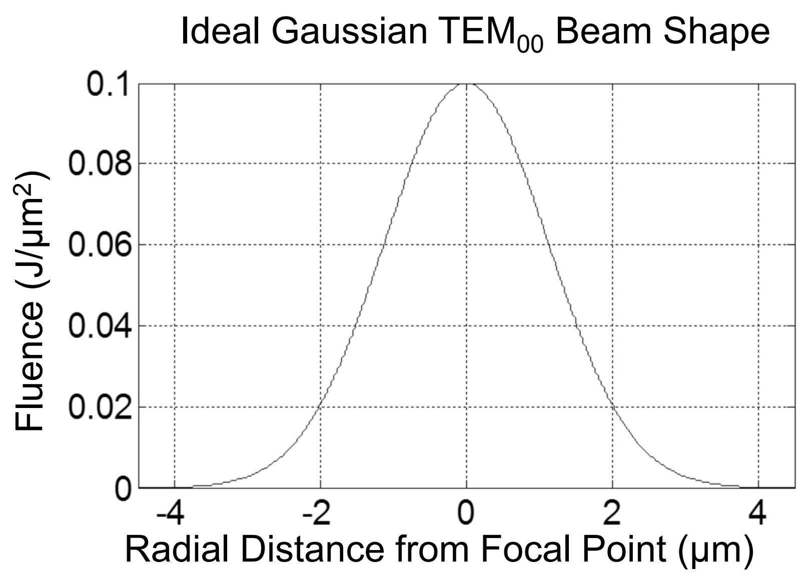
- Four key machining parameters of interest:



1. Pulse Spacing (μm)



2. Pulse Repetition Rate (Hz)



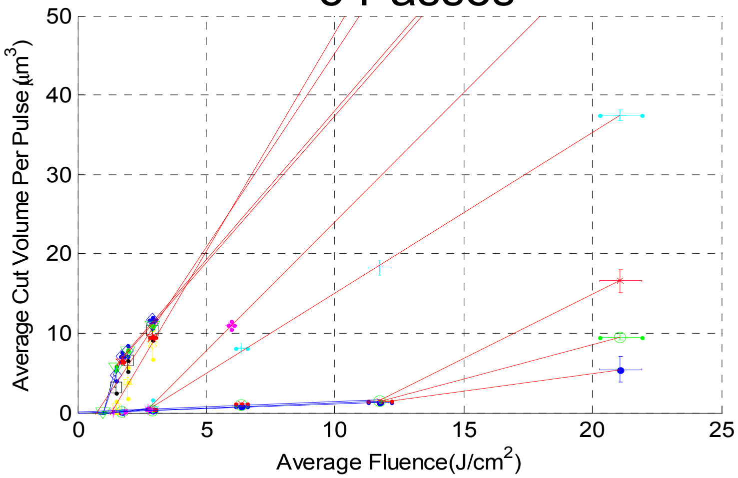
3. Pulse Fluence (J/cm^2)

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

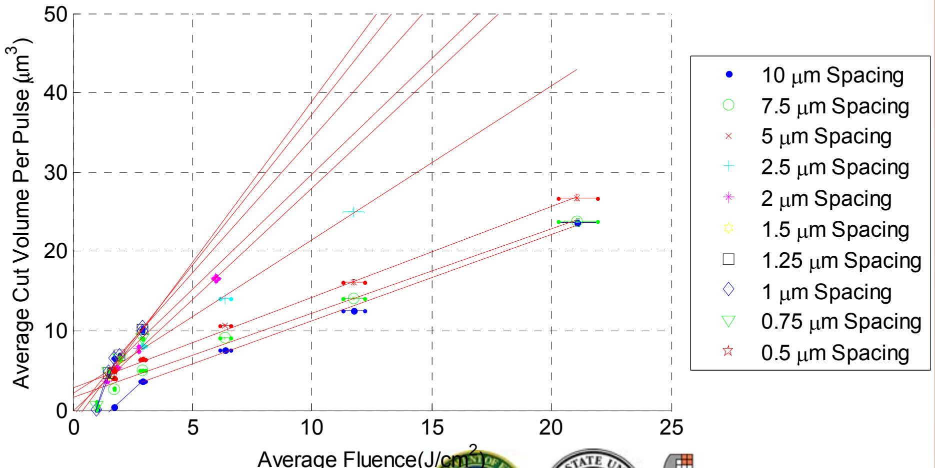
Gentle vs. Strong Ablation

- 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

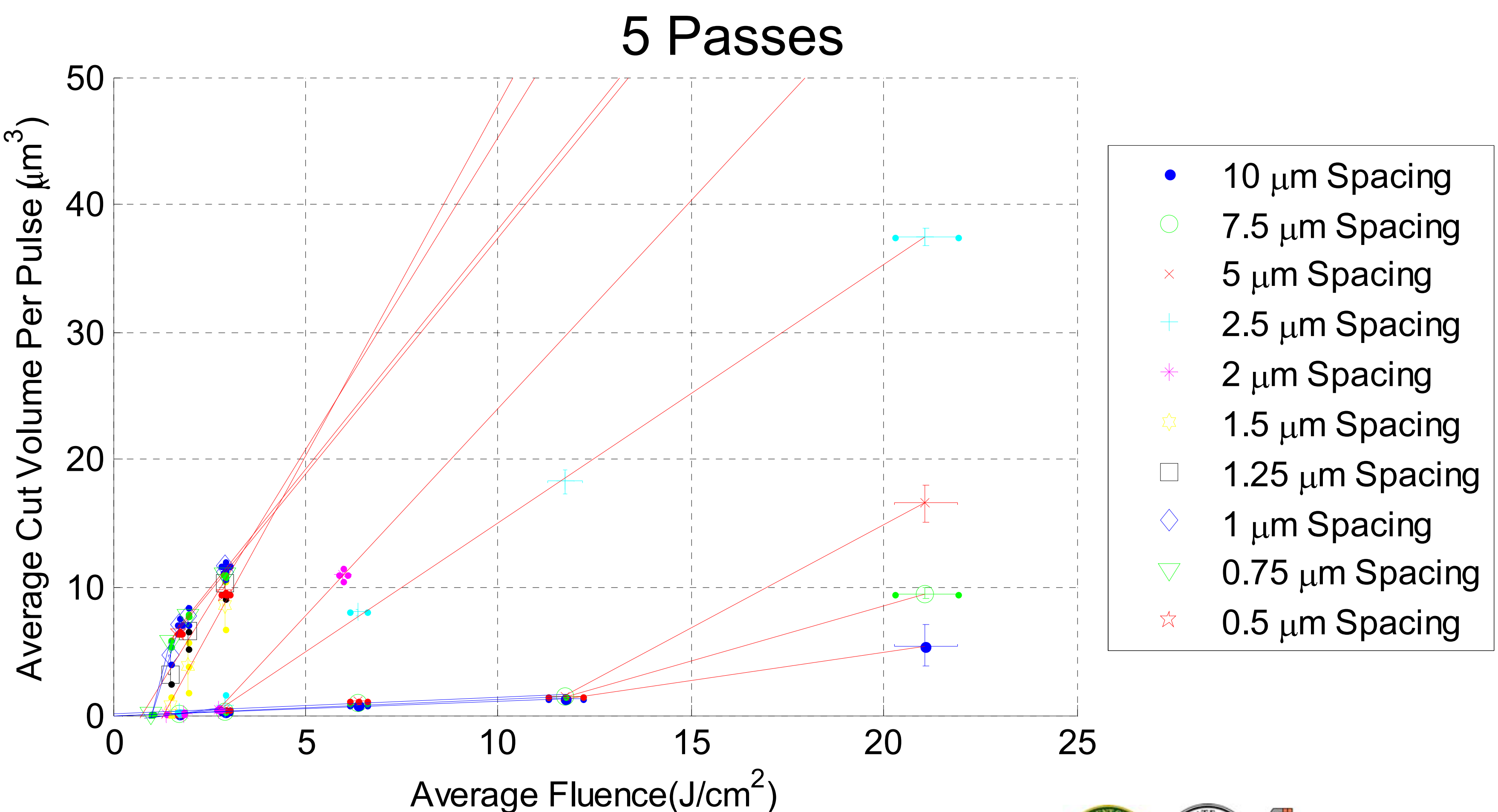
5 Passes



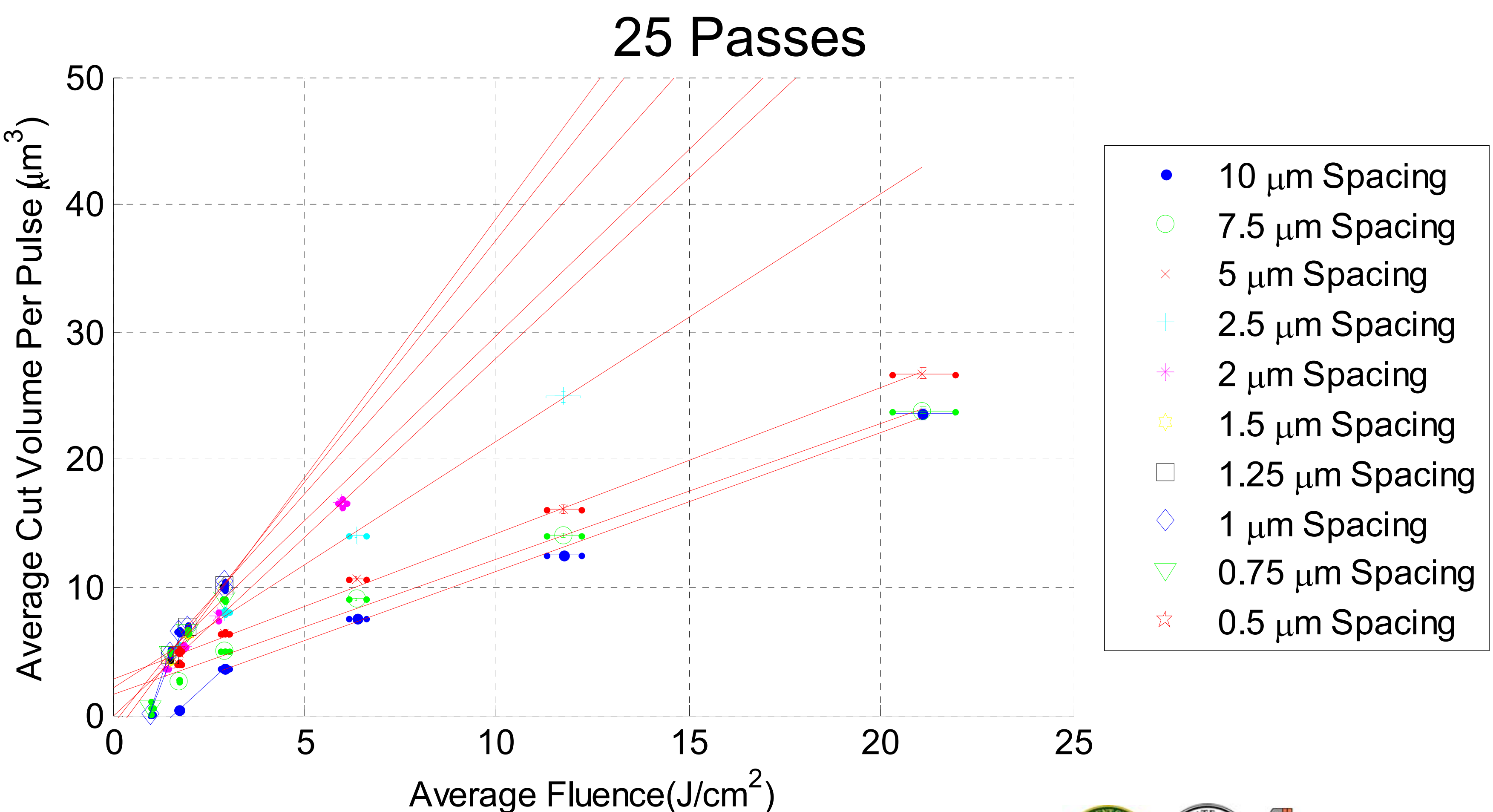
25 Passes



Gentle vs. Strong Ablation

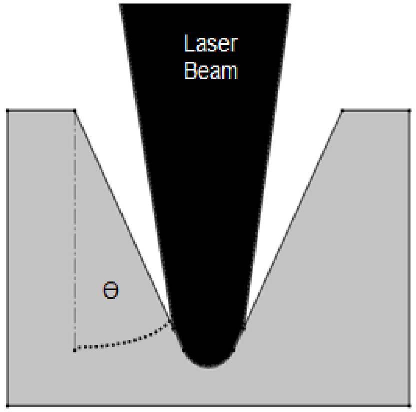


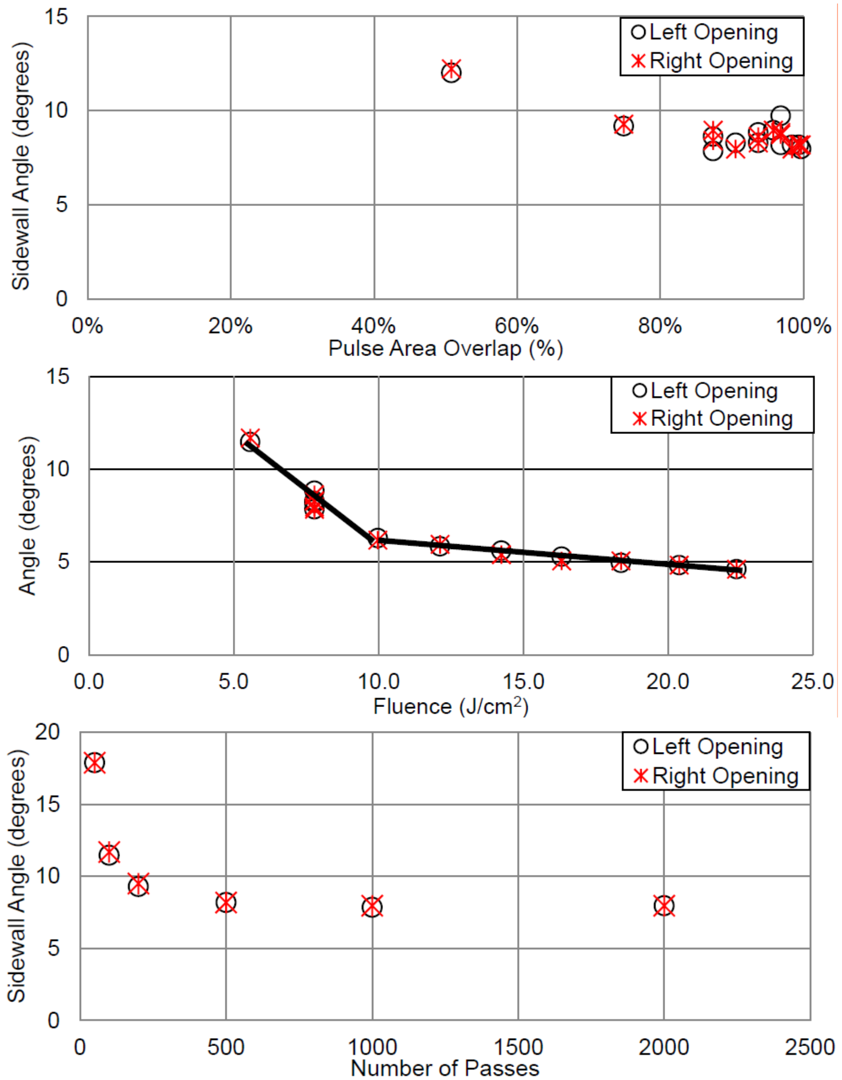
Gentle vs. Strong Ablation



Sidewall Angle

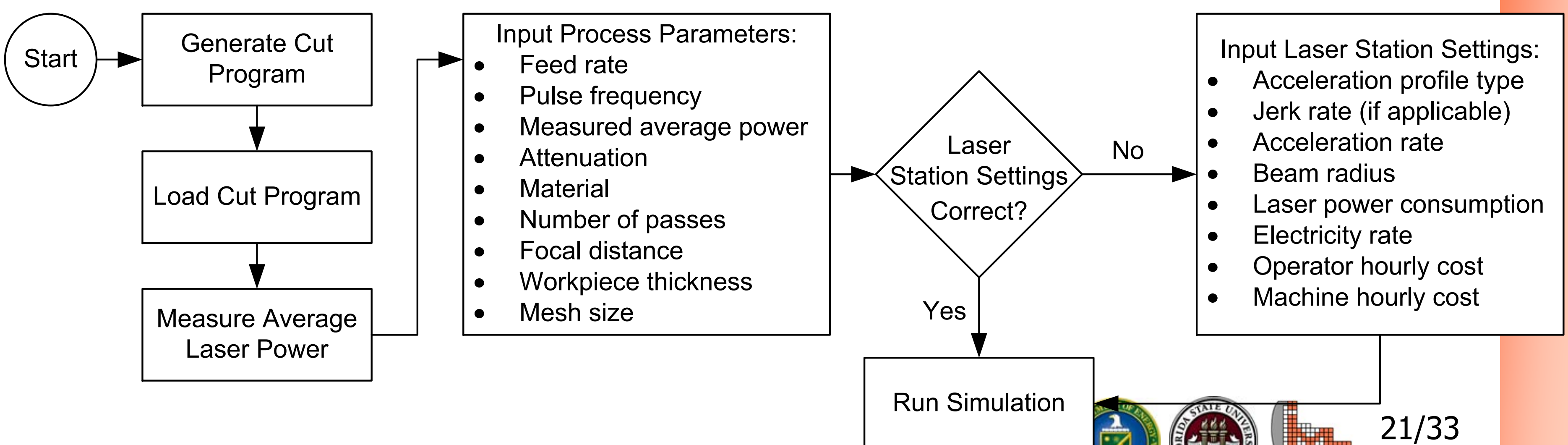
- Machining parameters
  - Fluence: 5.1-25.5 J/cm^2
  - Pulse area overlap: 45-99%
  - Number of passes: 50-2000
- Sidewall angle is constant above ~75% pulse area overlap
- Higher fluence and number of passes reduce sidewall angle





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

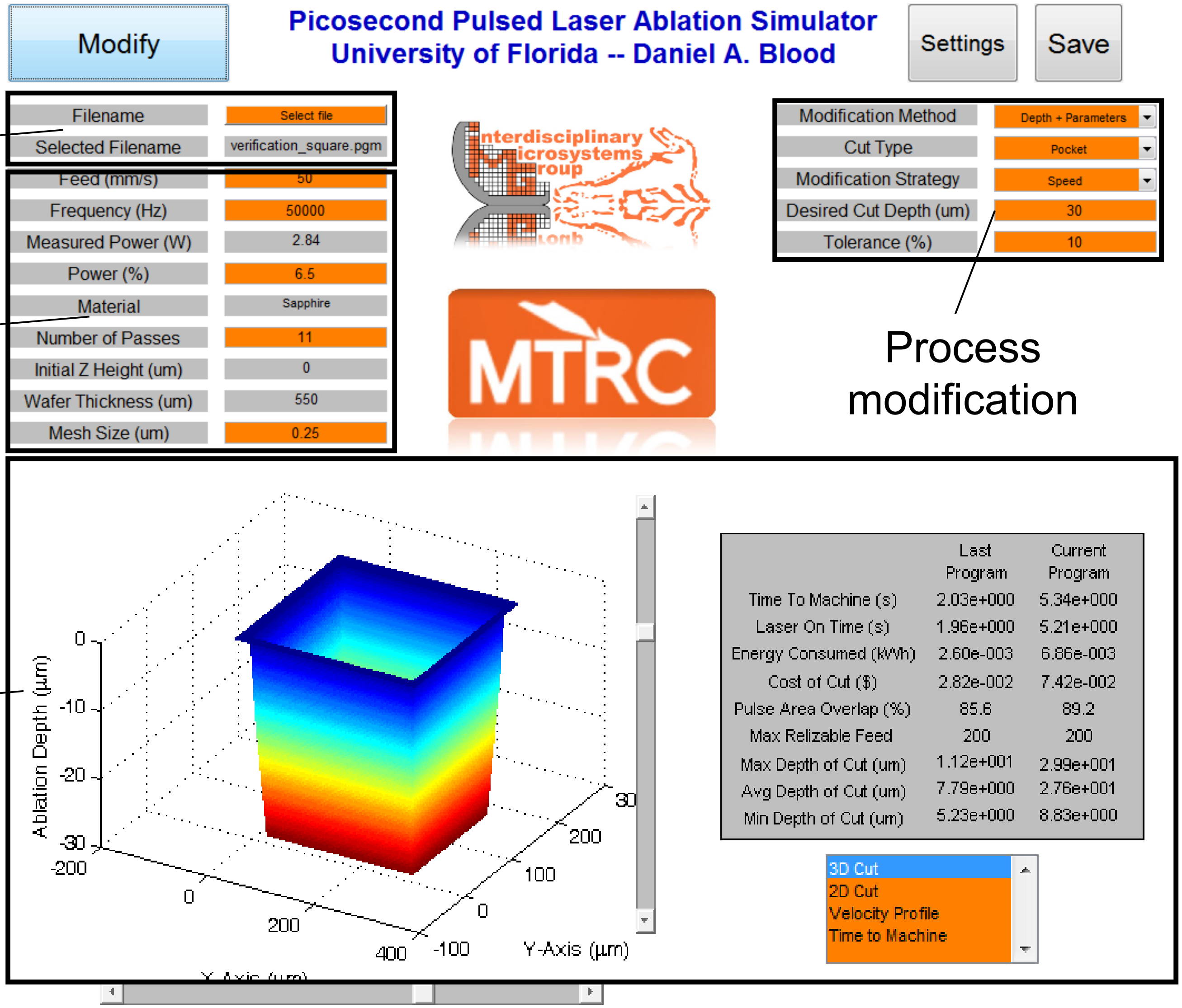


Laser Machining Simulation

Input file

Machining parameters

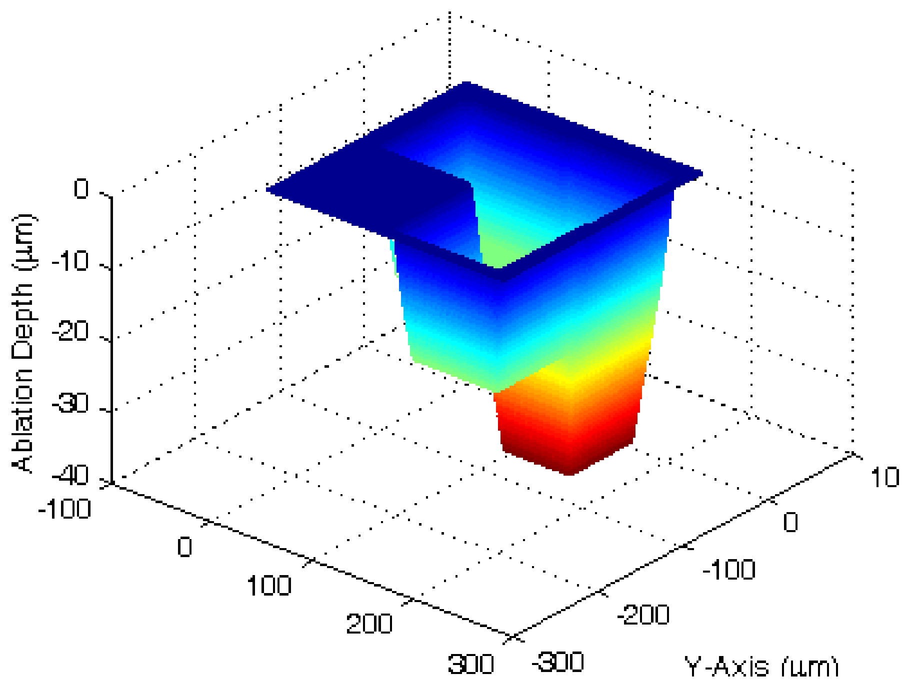
Simulation outputs

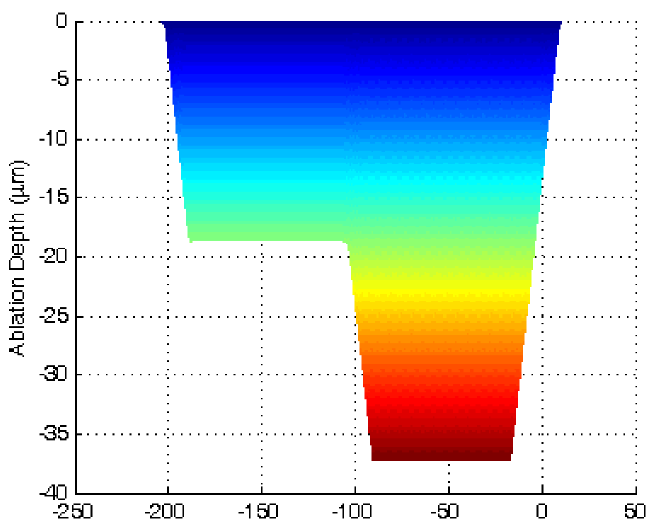


Process modification

Part Path Modification

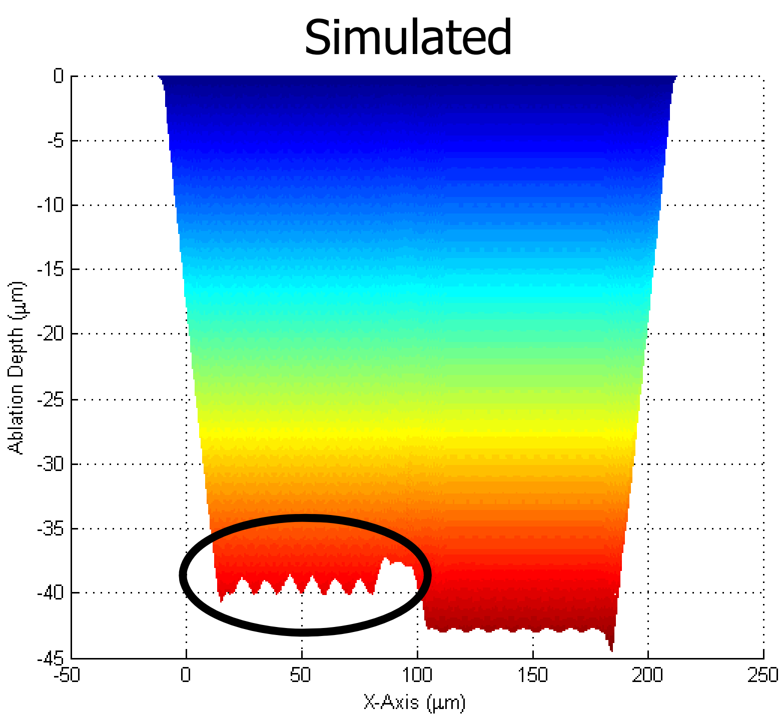
- Test geometry – overlapping rectangles
  - Creates deeper machined region
  - Goal: add passes in specific areas to create a single region of consistent depth

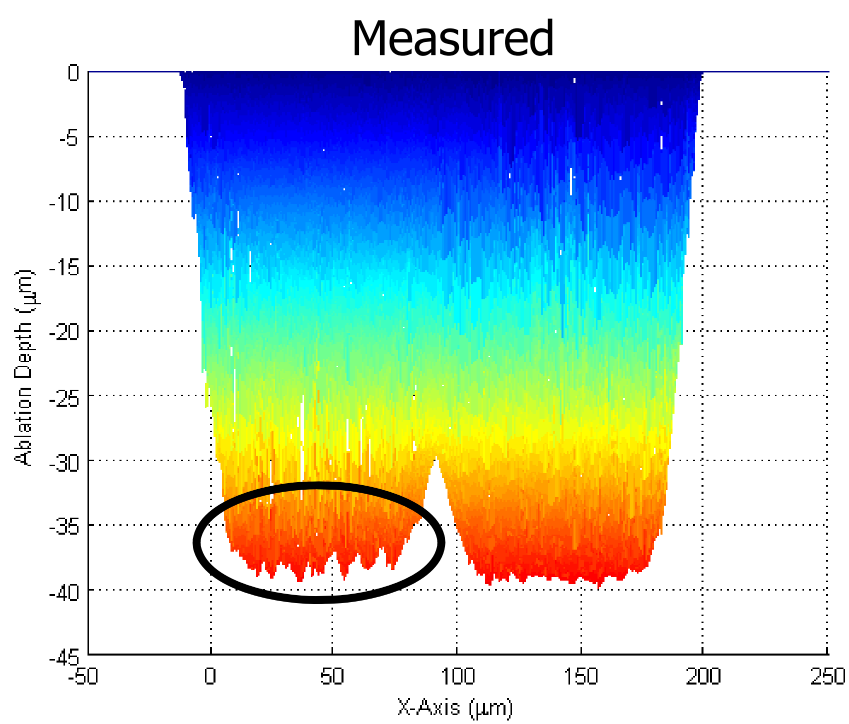




Part Path Modification Results

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





Outline

- Introduction
- Approach
- Proof-of-Concept Device
- Objectives
- Ultrashort Pulse Laser Micromachining
- Laser Ablation Modeling
- Conclusions

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 $\rho(x,t)$ 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

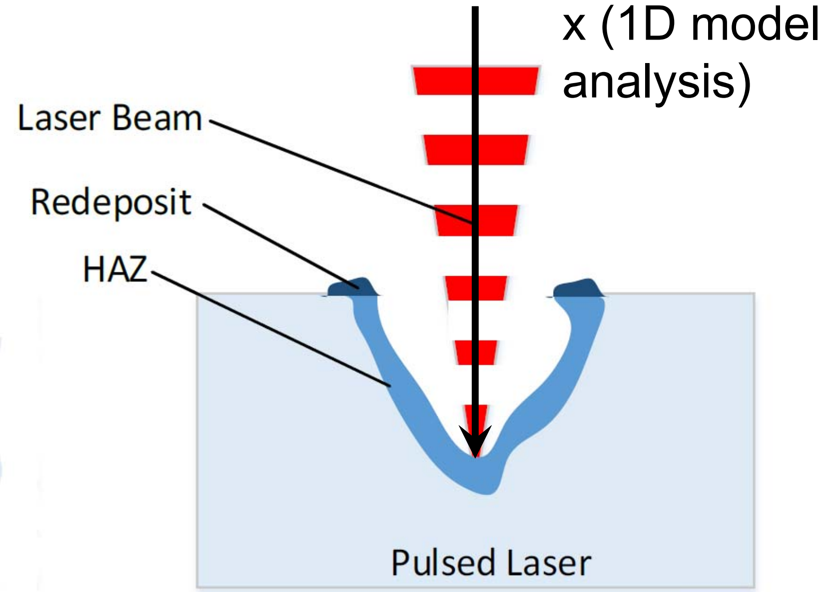
$$\sigma(\rho)\mu_0 \frac{\partial E}{\partial t} = \nabla^2 E$$

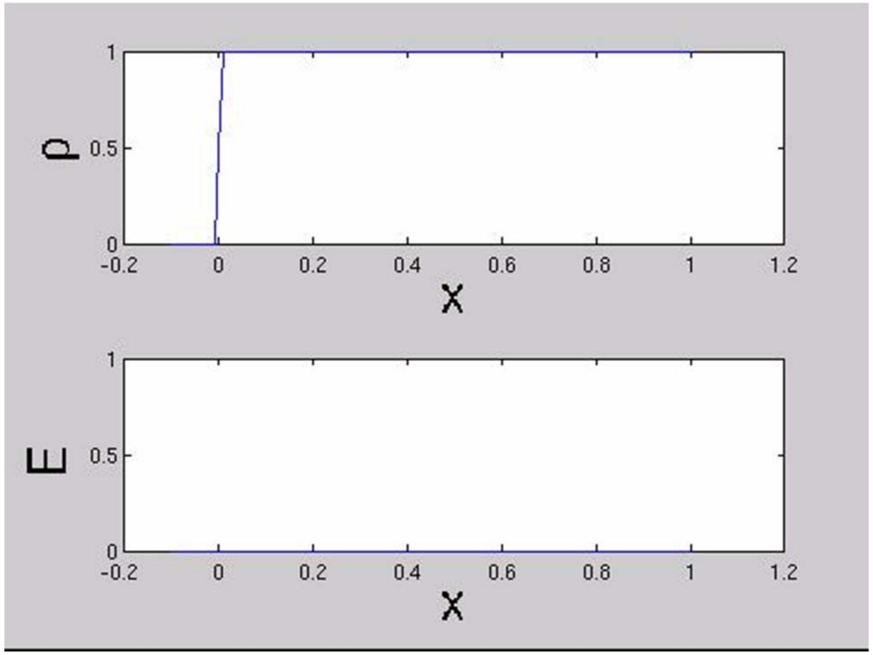
Electromagnetic equation

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

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

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

Electromagnetic equation

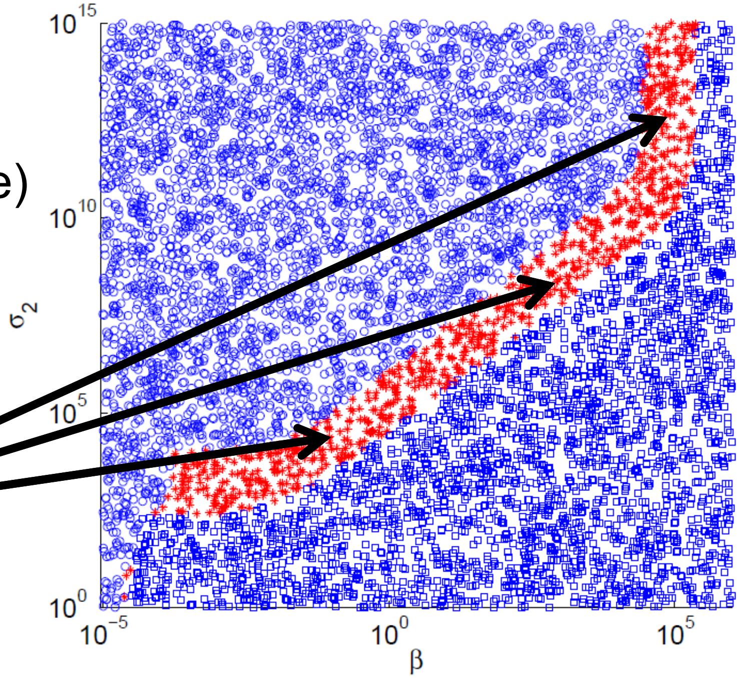
$$\beta(E)\frac{\partial \rho}{\partial t} = a_0 \nabla^2 \rho - \frac{\partial \psi}{\partial \rho} - \gamma(E)$$

Phase field based order parameter model

- Critical parameters considered

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

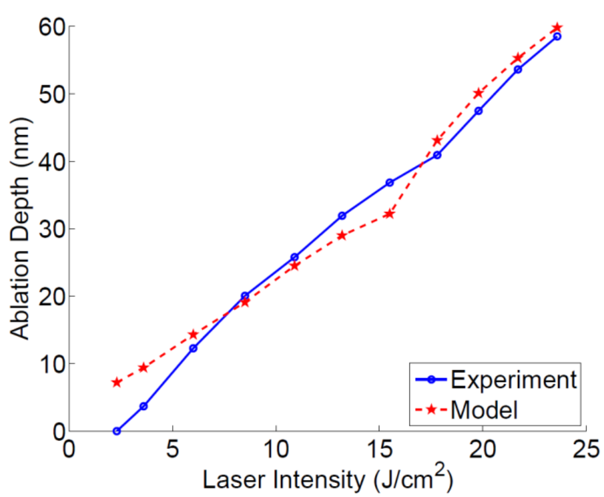
$\beta(E)$ Inverse electronic mobility parameter

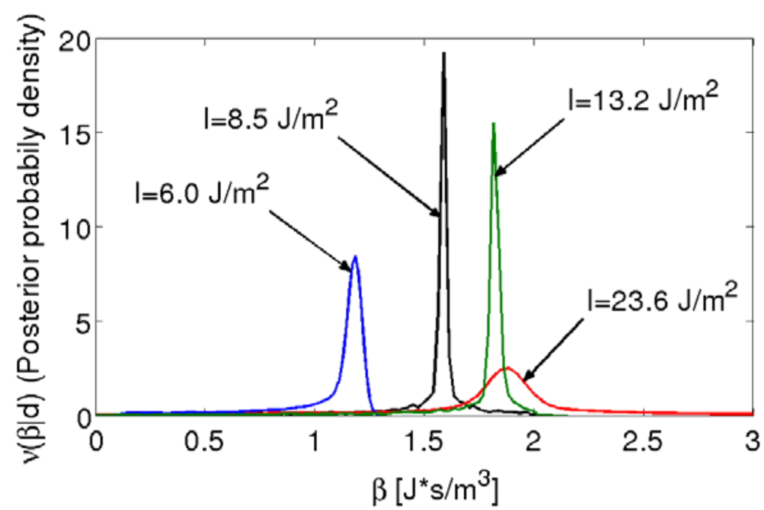


Region of finite machined depth giving potentially valid numerical correlation with laser ablation experiments

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.

Outline

- Introduction
- Approach
- Proof-of-Concept Device
- Objectives
- Ultrashort Pulse Laser Micromachining
- Laser Ablation Modeling
- Conclusions

Summary

- Laser machining process for the sapphire-UF laser system characterized
- Simulator developed and validated based on empirical data
- Laser ablation model developed
  - Coupling among laser exciation and electronic structure evolution
  - Uncertainty and sensitivity analysis conducted on a reduced order model approximation
  - Parameter dependence on laser intensity identified

Future Work

- Quantification of laser damage via four point bend testing at elevated temperatures
- Extension of the laser ablation model to include effects of sub-surface laser damage on strength and fracture
- Fabricate high-temperature plane wave tube for dynamic pressure calibration
- Sensor fabrication
- High-temperature package development
- Packaged sensor calibration & hot jet testing

Questions?