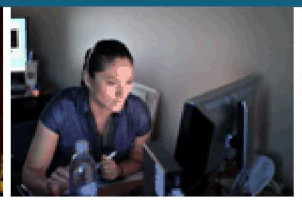
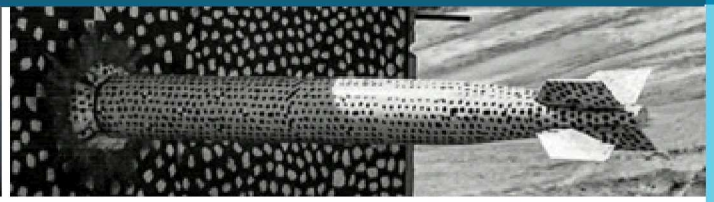
SAND2020-5222PE

Final Presentation





May 6, 2020

Emily Huntley

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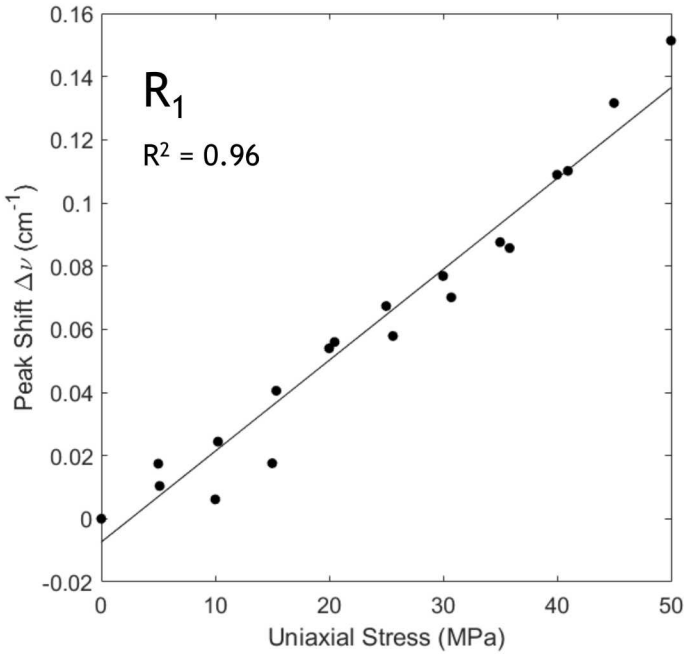
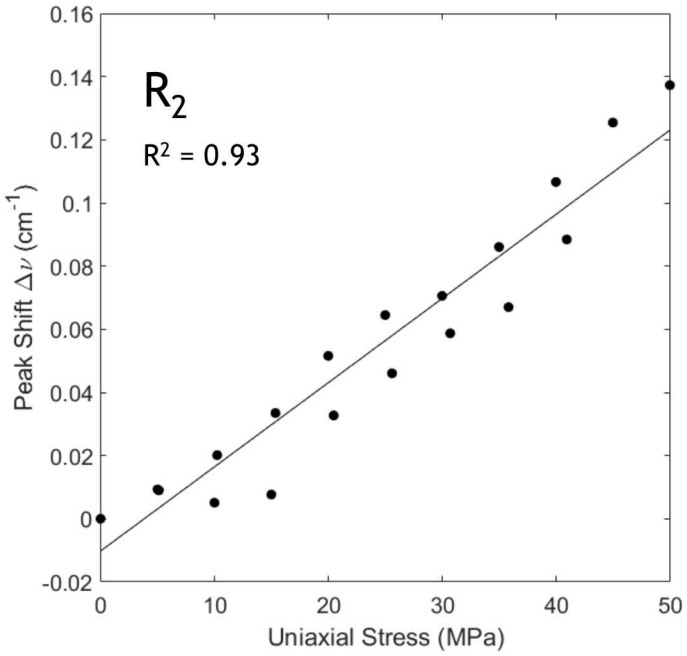
Photoluminescent Spectroscopy of Alumina Filled Glass

Determination of Piezospectroscopic Coefficient

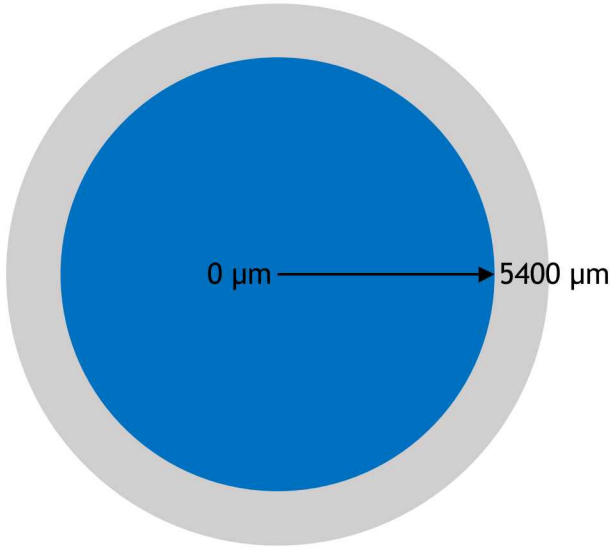
- Samples were loaded in 4-pt bending, uniaxial stress range of 0 to 50 MPa
- Different chromium concentrations can affect the coefficient –same material was used throughout
- New piezospectroscopic coefficients were determined from slopes of applied stress vs peak shift
  - $\Pi_{ave,R1}$=2.88 $cm^{-1}GPa^{-1}$
  - $\Pi_{ave,R2}$=2.66 $cm^{-1}GPa^{-1}$
- Therefore:
  - $\Pi_{tr,R1}$=8.64 $cm^{-1}GPa^{-1}$
  - $\Pi_{tr,R2}$=7.98 $cm^{-1}GPa^{-1}$
  - These are used in the seal calculations

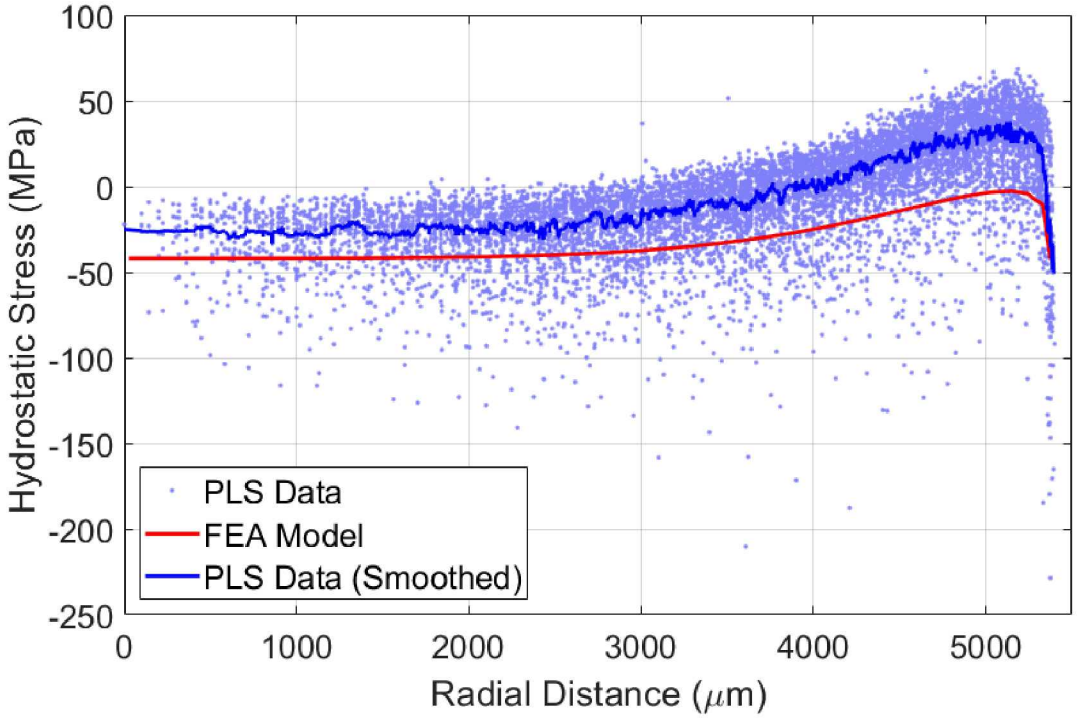




Applying Piezospectroscopy to Simple Seal

- Overall Scan
  - Power: 35 mW, spot size: 100x100 μm
  - Showed qualitative agreement with *shape* of model predictions, not necessarily with the absolute stress values
  - Different power setting than all other scans (including bend bars) may be cause of large discrepancy from other scans

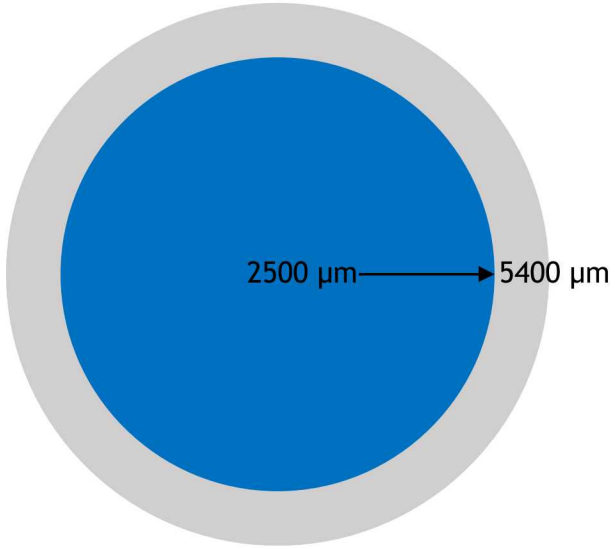


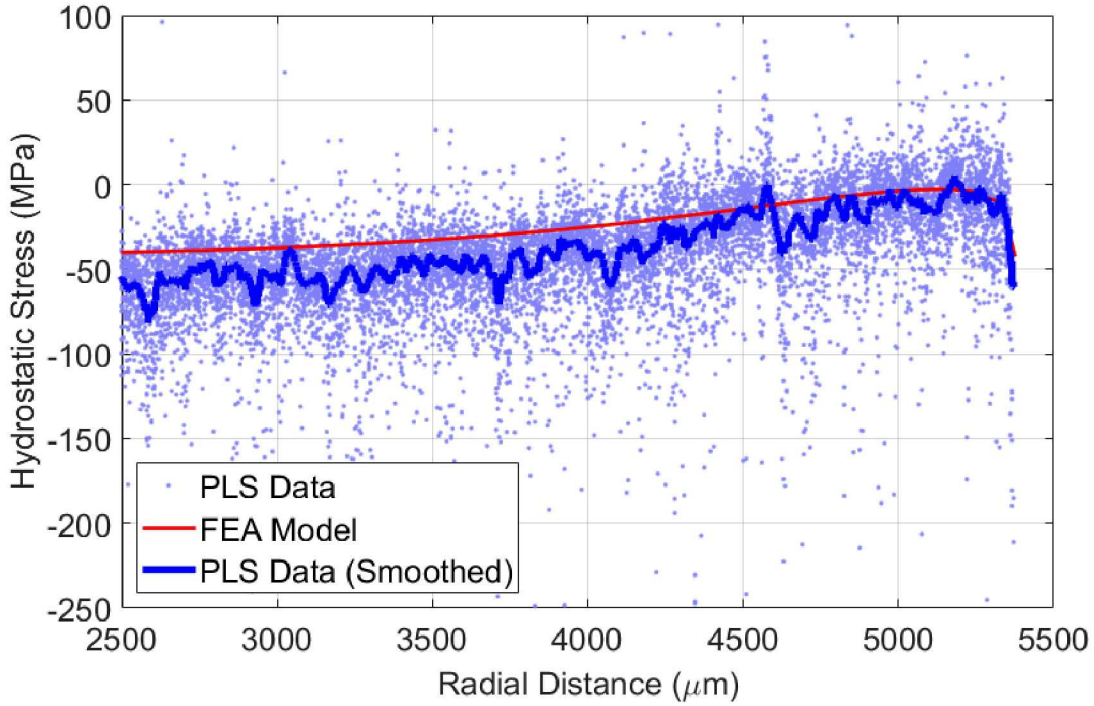


*data was smoothed using a moving median with a window of 100 data points to visualize the data

Applying Piezospectroscopy to Simple Seal (con't)

- Large Area Scan
  - Power: 50 mW, spot size: 5x5 μm
  - Did not scan region of constant stress
  - Absolute stress values closer to those predicted by model
  - Large scatter and regions of high or low values (ex: near 4580 microns) are clustered around pores





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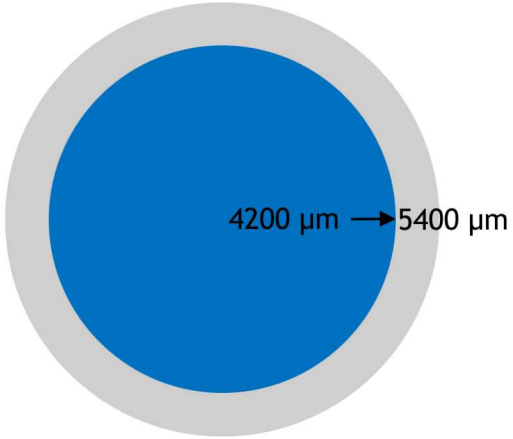
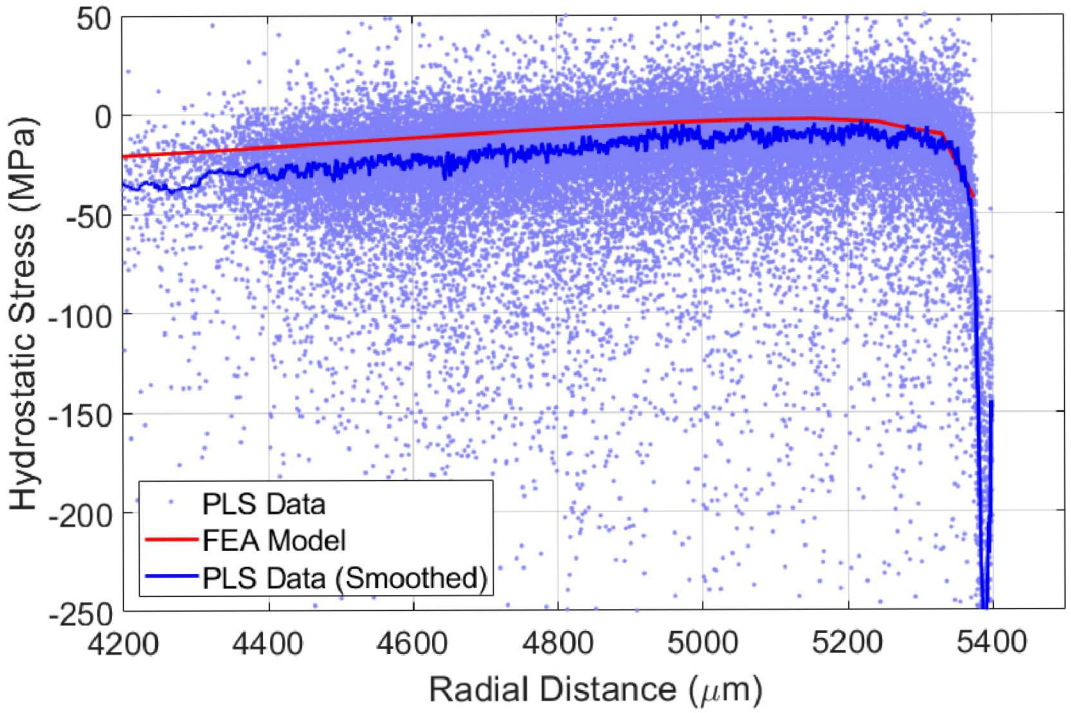
Applying Piezospectroscopy to Simple Seal (con't)

- Near Edge Scan
  - Power: 50 mW, spot size: 10x10 μm
  - Conducted to investigate sudden spike in compressive stress near edge (starting ~100 μm from the edge), as predicted by the model
  - Spike is captured in this data!
  - Overall, data shows strong agreement with model near edge
  - Interesting find: within ~10μm of the edge of the seal, the compressive stress decreases suddenly

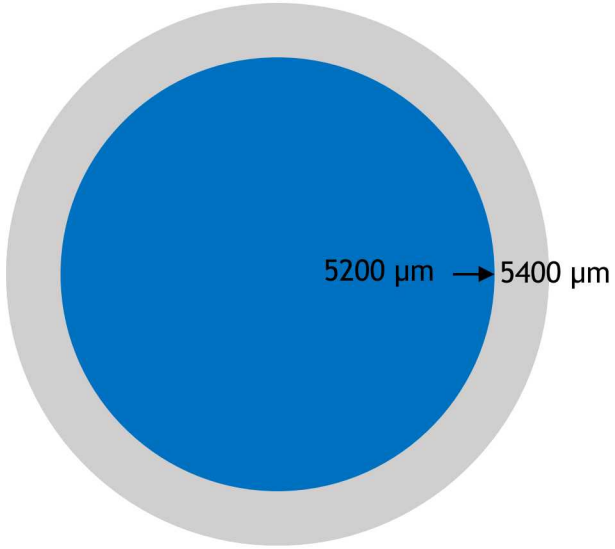


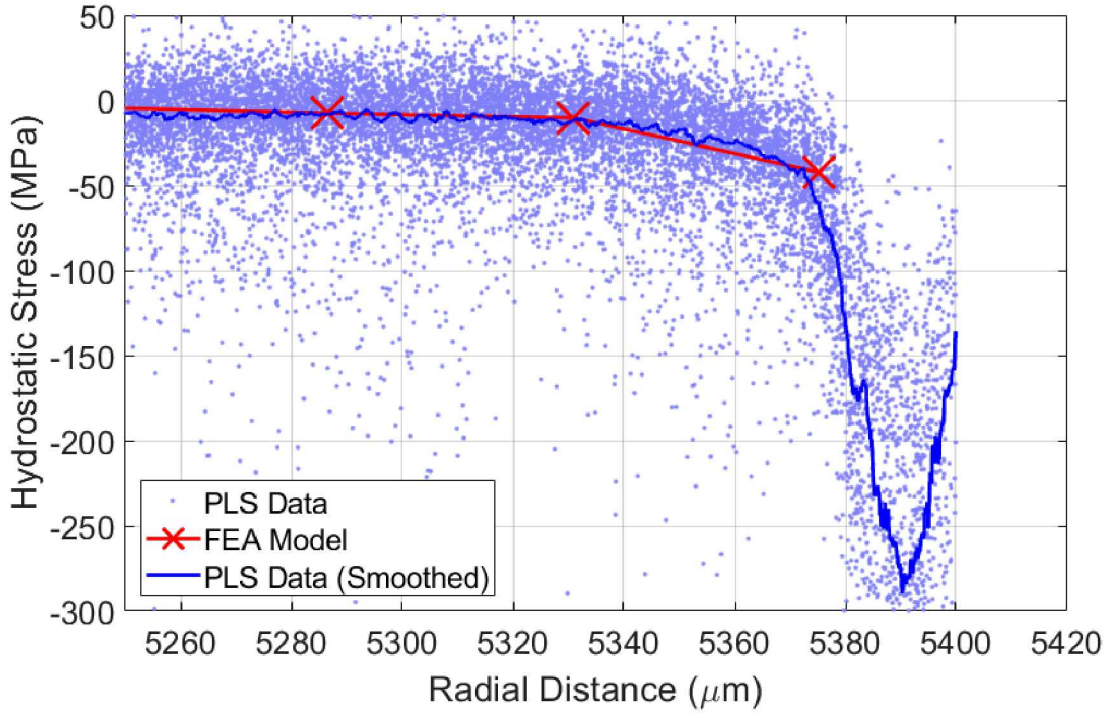


*data was smoothed using smoothdata function in MATLAB on moving median mode in order to visualize the data

Applying Piezospectroscopy to Simple Seal (con't)

- At Edge Scan
  - Power: 50 mW, spot size: 5x5 μm
  - Conducted to investigate apparent decrease in compressive stress within ~10 μm of the edge
  - Major overlap between model and experimental values
  - Decrease is evident! The mesh in the model will need refining to see if it also captures this effect fully

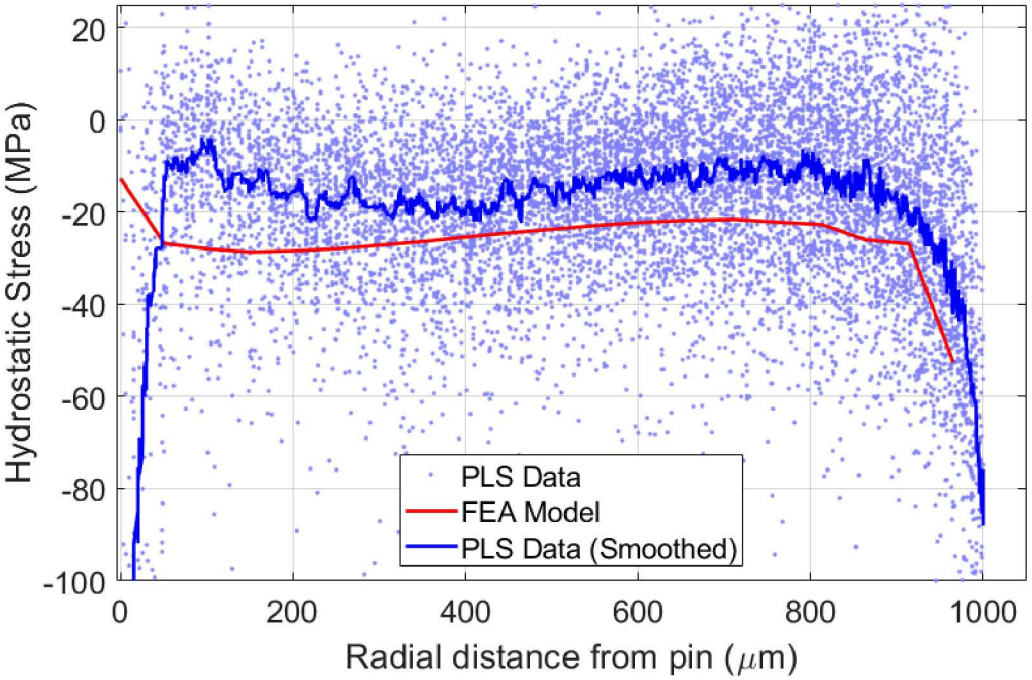


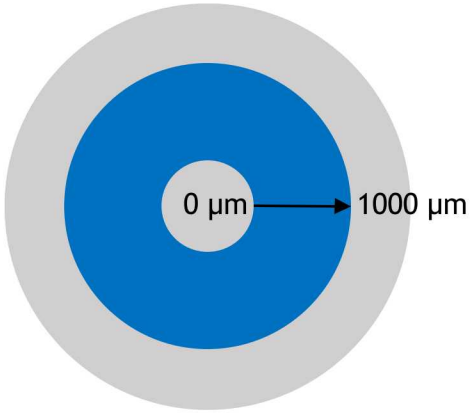


*data was smoothed using smoothdata function in MATLAB on moving median mode in order to visualize the data

Applying Piezospectroscopy to Single Pin Header

- Initial Scan:
  - Power: 50mW, Spot Size: 25x25 μm
  - Model behavior and experimental data follow the same pattern from the center of the glass and out to the edge of the shell (~400-1000 μm)
  - Model and data diverge near the pin, the model predicts a moderate decrease in compressive stress while the data shows a sharp increase in compressive stress.
  - Further scans of pin-glass interface in progress
  - High amount of noise due to poor surface finish, the sample has now been repolished before new scans

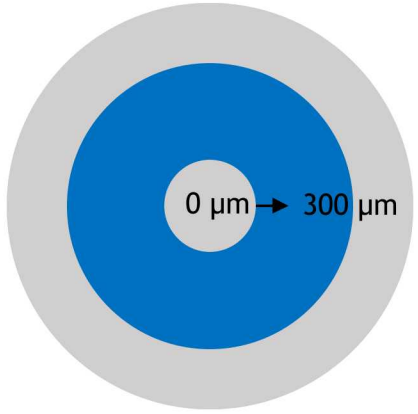


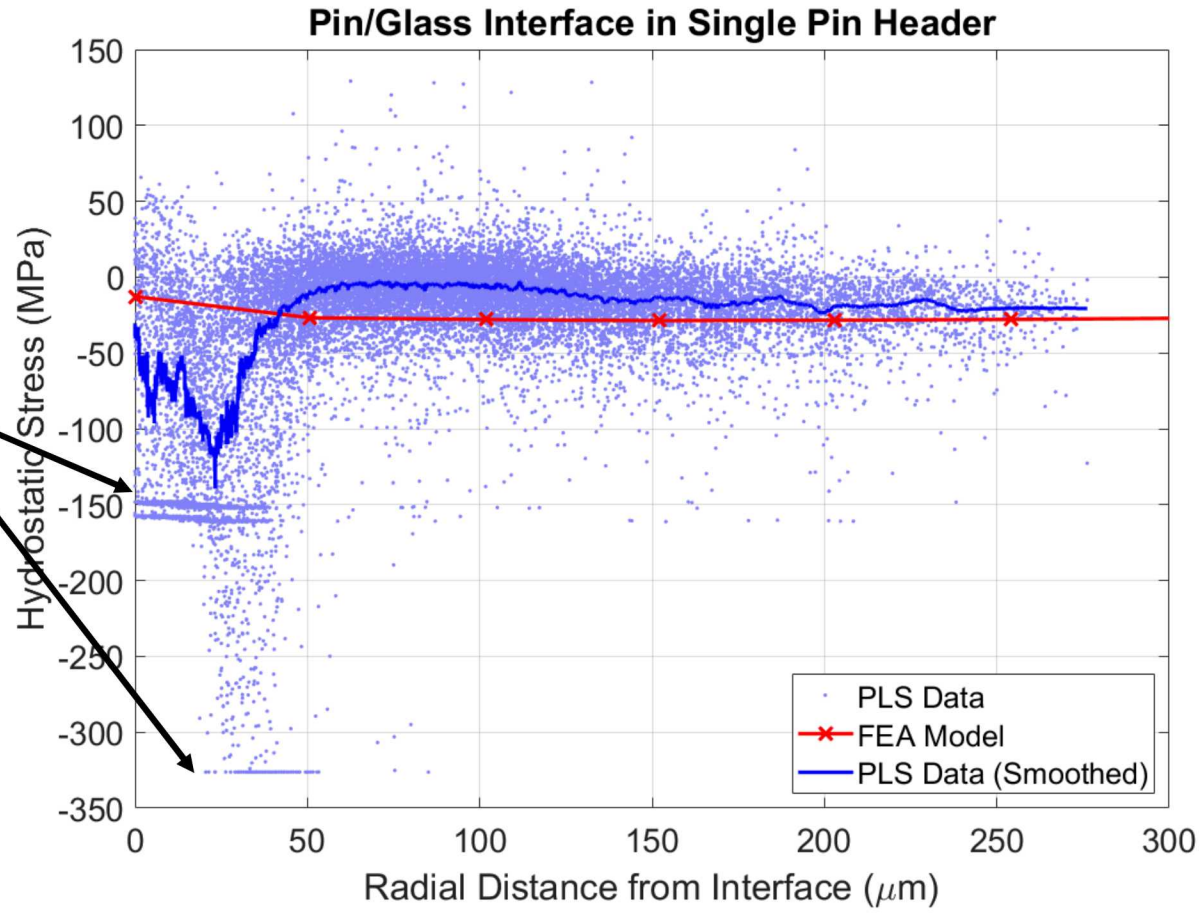


*data was smoothed using smoothdata function in MATLAB on moving median mode in order to visualize the data

Applying Piezospectroscopy to Single Pin Header (con't)

- Power: 50 mW, Spot Size: 5x5 µm
- Actual scans are rectangles that were tiled together
- Model and data diverge near the pin, the model predicts a moderate decrease in compressive stress while the data shows a sharp increase in compressive stress.
- Data has strange, minimum stress for each scan





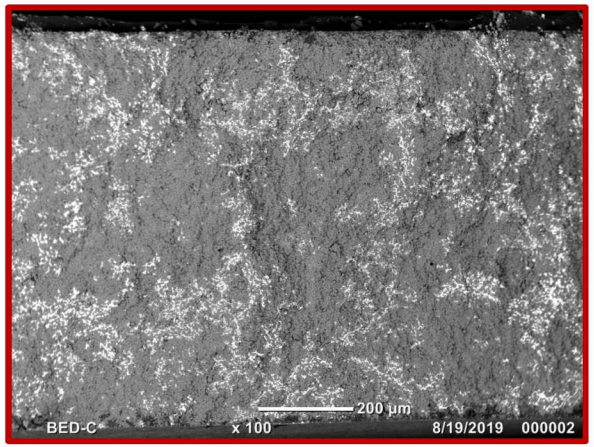
Future PLS Work

- Aging study – starting soon
- More work investigating the single pin stress pattern, the data we have so far seems to show discrepancy with model near pin interface, but is very noisy
- How to process peak data with MATLAB:
  - I made a function called peak2stress which simplifies the code A LOT
  - If you open the peak2stress.mlx file, there are instructions on how to use it (inputs & outputs)
  - Ensure that the peak2stress file is in your file path on MATLAB
  - Each of my PLS codes has comments on them to help with understanding how it works

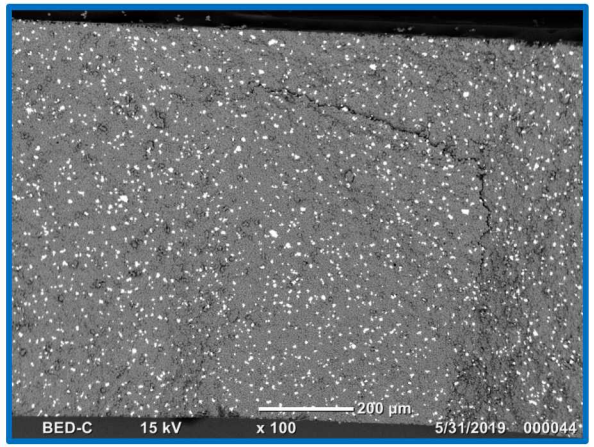
Other Projects

AlN Disks

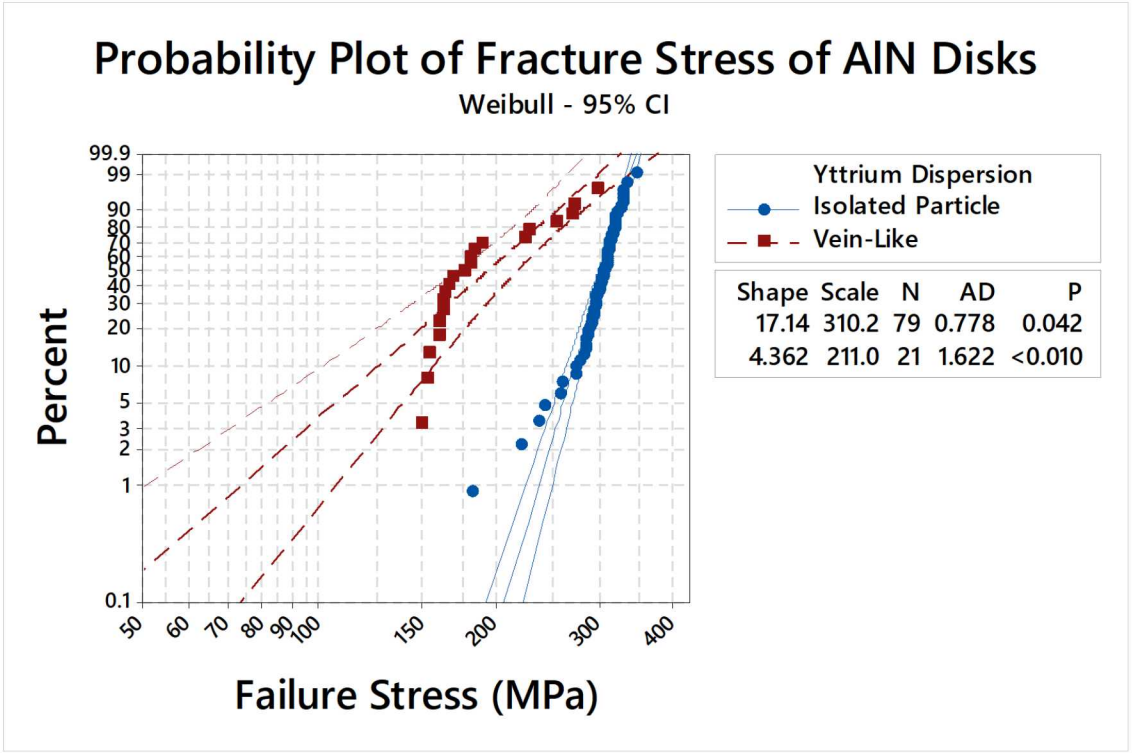
- Imaged 100 fracture surfaces in SEM
  - Received them after the manufacturer ran biaxial strength testing on them
- Split into "isolated particle" and "vein-like" yttria dispersion
- Found significant difference in strength between the different yttria dispersions
- Presented at RGSAM



Vein-Like

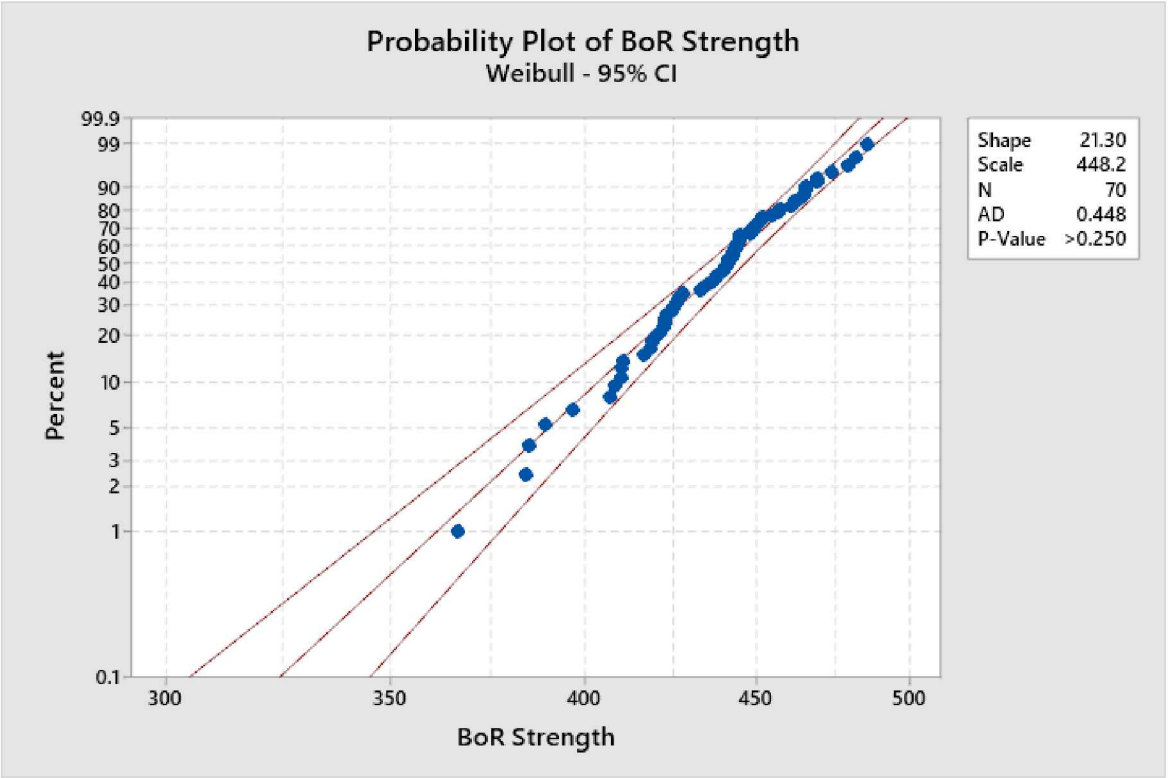


Isolated Particle



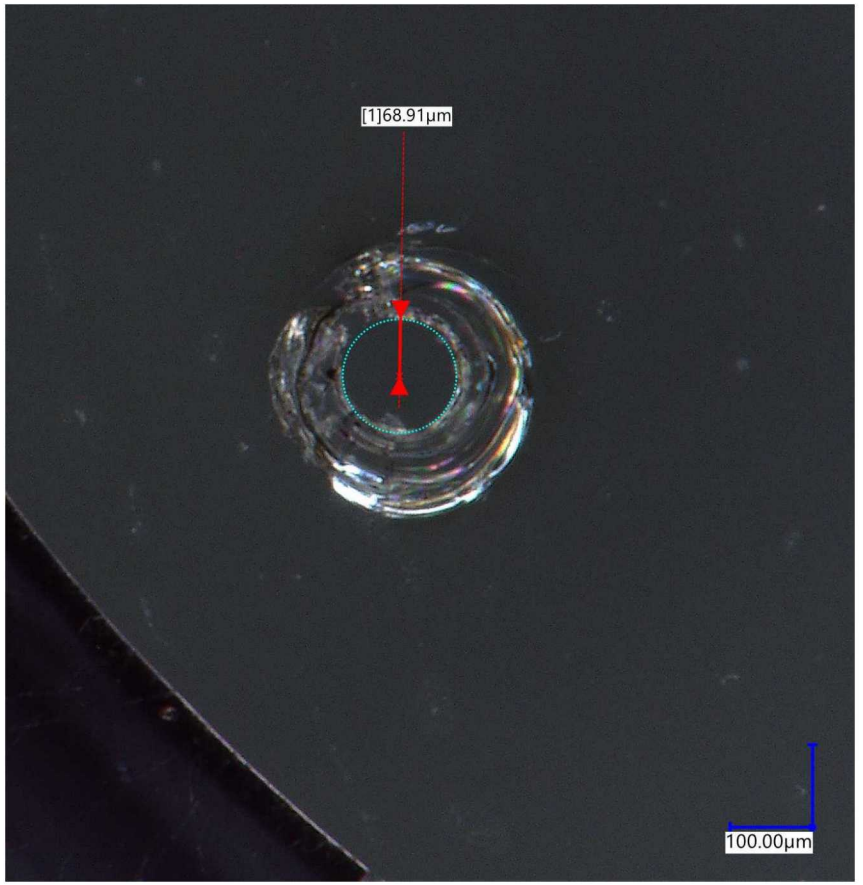
AlN Disks (con't)

- Ball-on-Ring strength tests on additional 70 samples
- This new batch of samples was made before the vein-like structure was discovered
- The intention was to test whether the structure affected strength in a smaller searched area, however…
- All 70 of these samples had isolated-particle structures on the tested side



Hertzian Indentation

- Collected data for Hertzian indentation at two different crosshead speeds
  - Approximately 30 samples at each speed
  - 0.2 mm/min vs 0.002 mm/min
- Measured ring radius in Keyence for each indentation
- The load and radius values can be used to calculate the estimated stresses at the crack
  - Still needs to be done
  - Can be a good virtual work project for new intern!

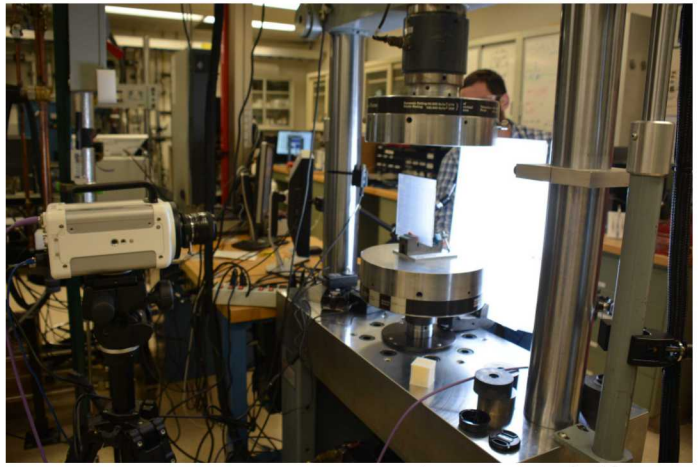


Frangible Glass Bonding

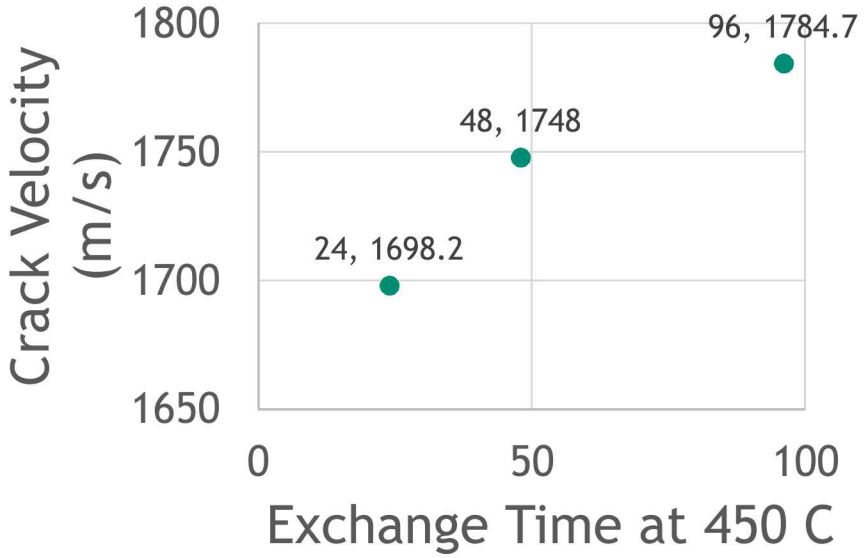
- After many tests, we decided the best process to get crack propagation between layers is…
  - Drop small amount of terpineol (5 drops for a 2”x2” square) on glass and spread out evenly
  - Place glass tape over terpineol layer, ensure there are no air bubbles
  - Burnout: 325°C for 8 hours
  - Glass-to-tape bonding: 390°C for 30 minutes
  - Stack glass pieces in desired layers, add weights on top
  - Glass-to-glass bonding: 390°C for 30 minutes and 450°C for 2 hours
- We were unable to get 90° bonded pieces to propagate cracks
  - Future work can look into this, perhaps make a fixture to hold pieces together at 90°

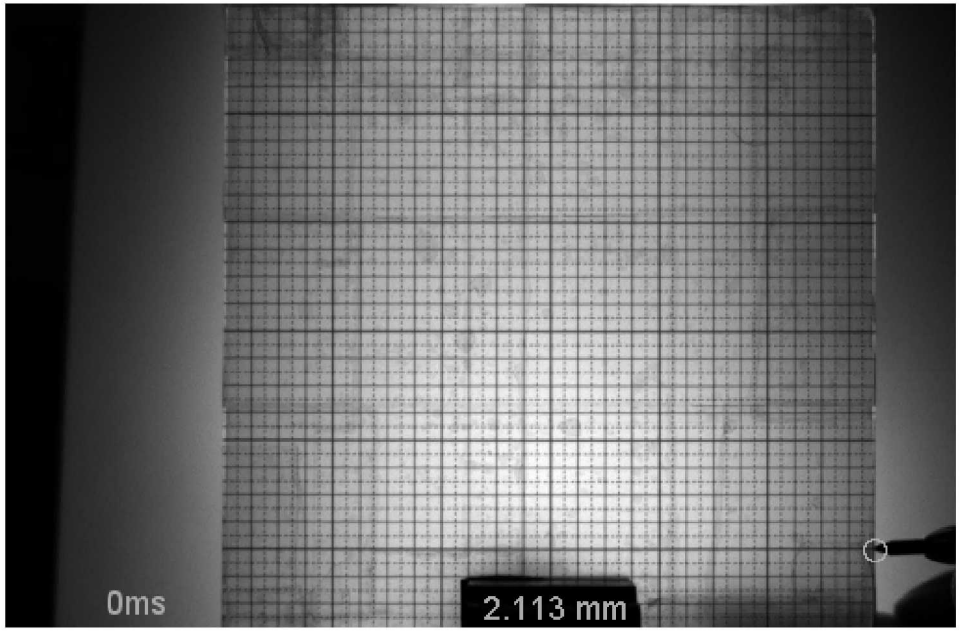
Slow Motion Frangible Glass

- Frame-by-frame analysis to find crack front velocity
- Can also use slow motion to analyze the pattern of the cracks
- Initial data shows that the crack velocity is dependent on exchange time
- The lighting flickers, special slow-motion lights could improve this if there's interest



Camera set up, opaque plastic to diffuse light

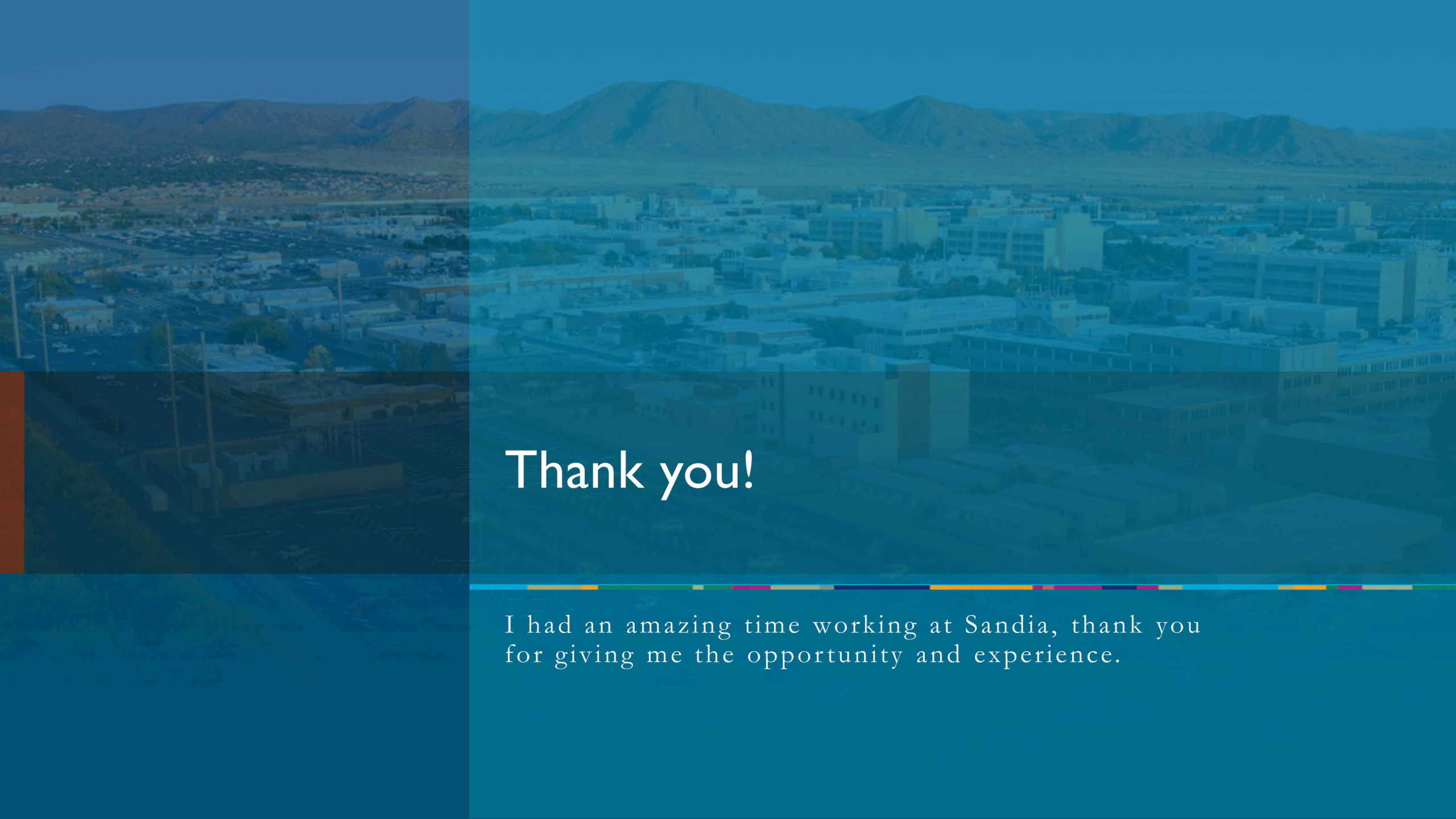




96 hour sample, annotated

Smaller, Miscellaneous Work

- These are projects I did some work on, but more as a helping hand:
  - Prepared a lot of frangible glass squares to build up pre-made stock for quick testing
  - PZT fracture toughness – notching to meet ISO standard
  - Studied effect of surface damage and extended etching on BPS bend bars
  - Helped Jonah edit the manual/instructions for the Centorr Hot Press
  - Inventoried the indenter supplies, inventoried and reorganized the glass-to-metal seal supplies



Thank you!

I had an amazing time working at Sandia, thank you for giving me the opportunity and experience.