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Micro-structural Stress Modeling of Brittle Materials for Enhanced Performance and Reliability

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U.S. DEPARTMENT OF ENERGY

NNSA National Nuclear Security Administration

Outline

- Motivation
- Background
- Experimental Measurements
- Modeling
  - Micro-structure meshing
  - Modeling of stresses
- Future Work

Brittle Materials Failure Presents A Reliability Concern in High Consequence Applications

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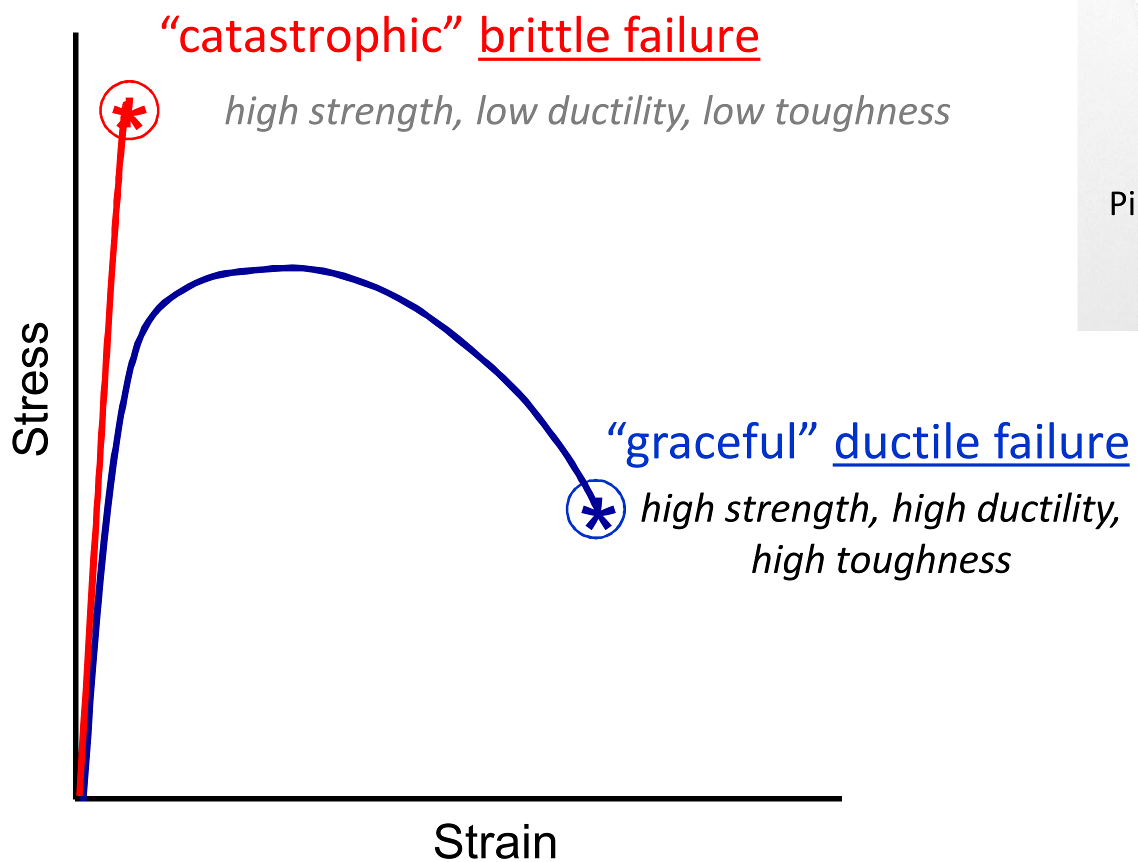
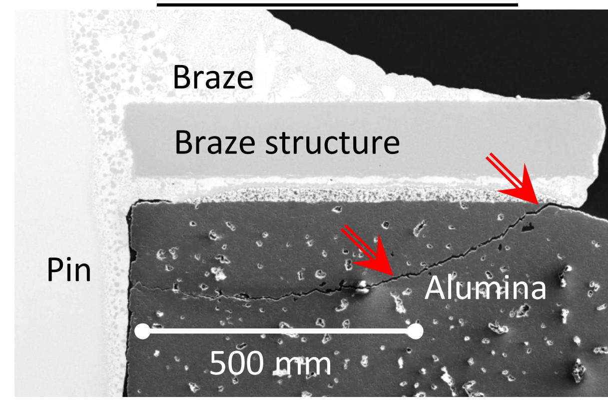
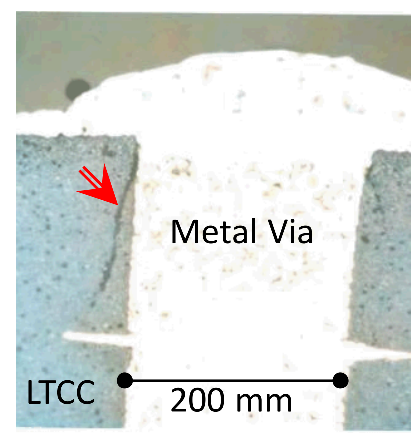


Electrical Feedthru



Electronic Substrate



Brittle materials are susceptible to sudden catastrophic failure

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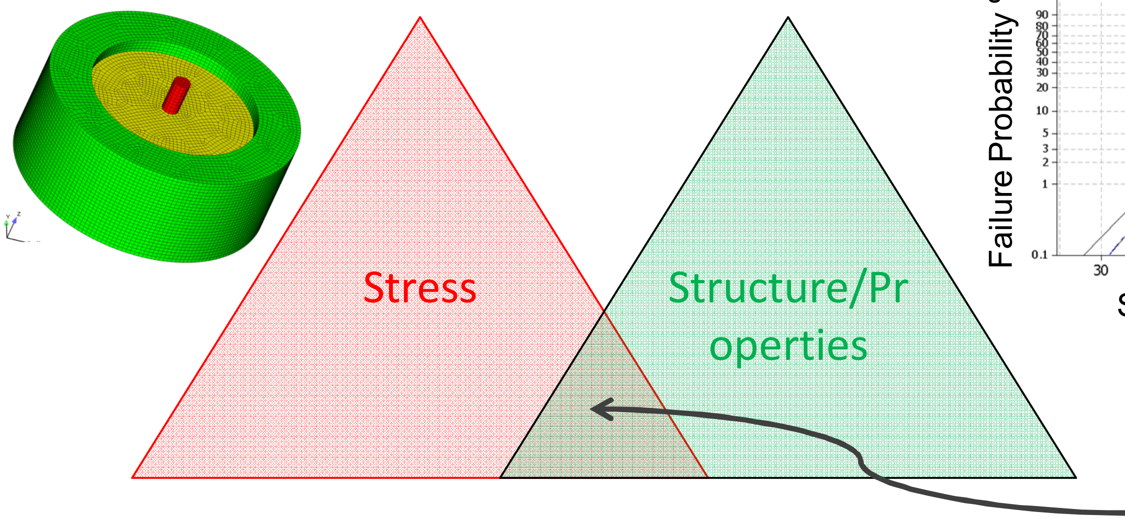
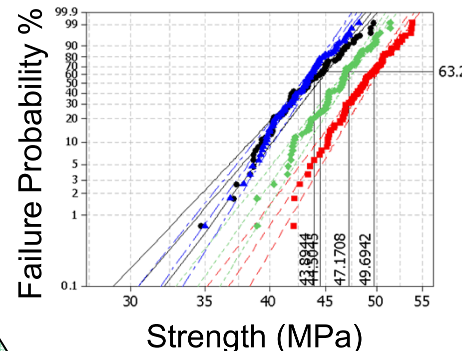
Current State: Qualitative Stress-Based Predictions

$$\sigma \sim \sigma_{crit}$$



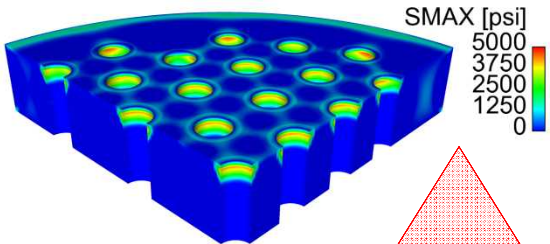


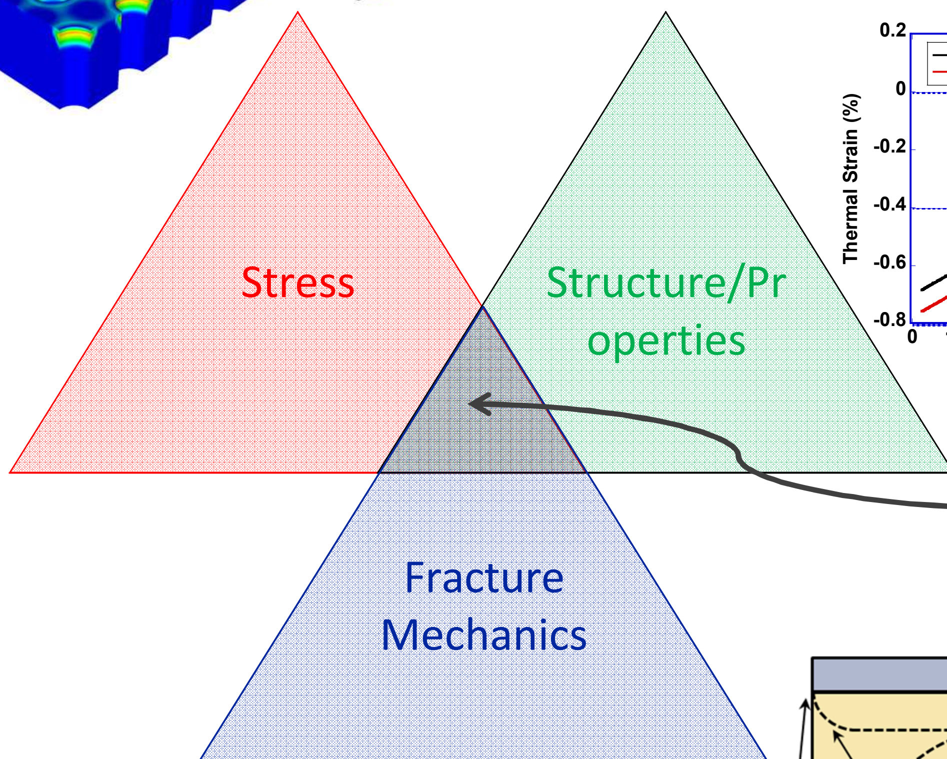
Qualitative prediction of brittle failure based on engineering judgment/experience

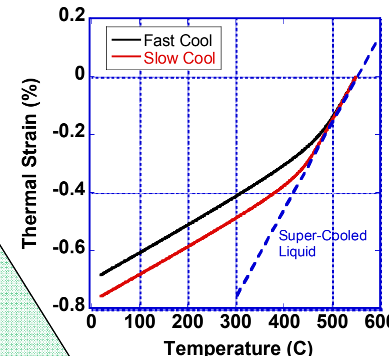
- *We Design To Avoid High Stress*
- Engineering Judgment Has Deficiencies
  - Limited by practical experience
  - Neglects flaws/flaw populations
  - Does not incorporate fracture mechanics

Future State: Quantitative Mechanics-Based Prediction of Brittle Failure & Reliability

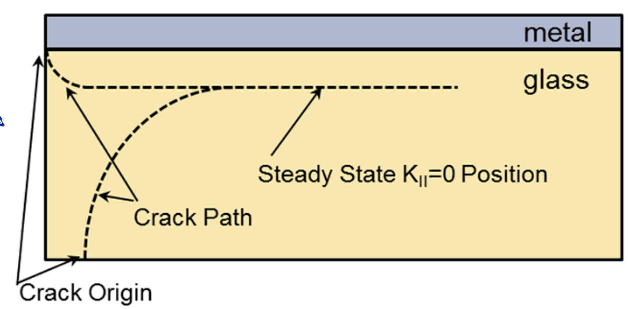
$$K \sim \sigma a^{1/2} \sim K_{IC}$$







Quantitative mechanics-based brittle failure prediction



Sandia Has A Research & Development Program That Addresses The Gaps To Predict Brittle Failure

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Vision

- Transition from **Qualitative** stress-based engineering judgment to **Quantitative** mechanics-based failure prediction.

Approach

- Develop foundational materials **characterization & modeling** S&T in:
  1) **Stress/Loading** – physically-based models, materials & processing data, & model validation
  2) **Fracture Mechanics** - crack initiation & propagation, and statistical bounds & variability
  3) **Structure/Properties** – understand & control of process-structure-property relations

Coordinated Stress, Fracture, & Structure/Props Experiments & Modeling Are Being Conducted

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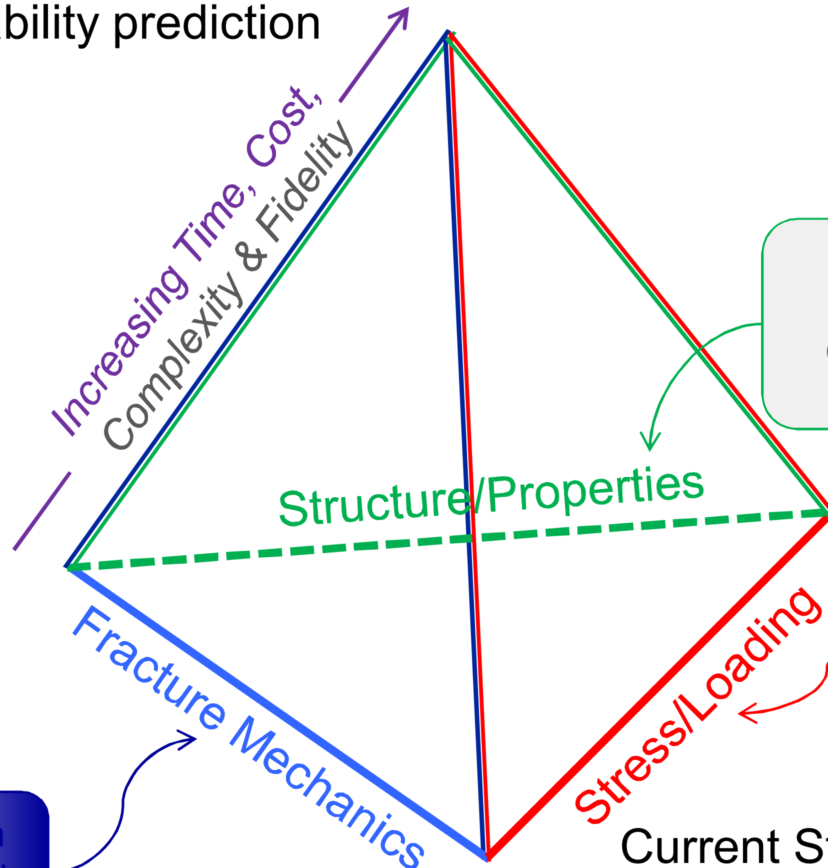
Vision: **Quantitative** mechanics-based failure & reliability prediction



Microstructure & Property Characterization & Modeling

Micro- to Continuum-Scale Stress Characterization & Modeling

Characterization & Modeling Crack propagation

Current State: **Qualitative** stress-based failure analysis (engineering judgment)

Micro- To Continuum-Scale Stress Mapping Is Possible With SEM/EBSD And PL Spectroscopy

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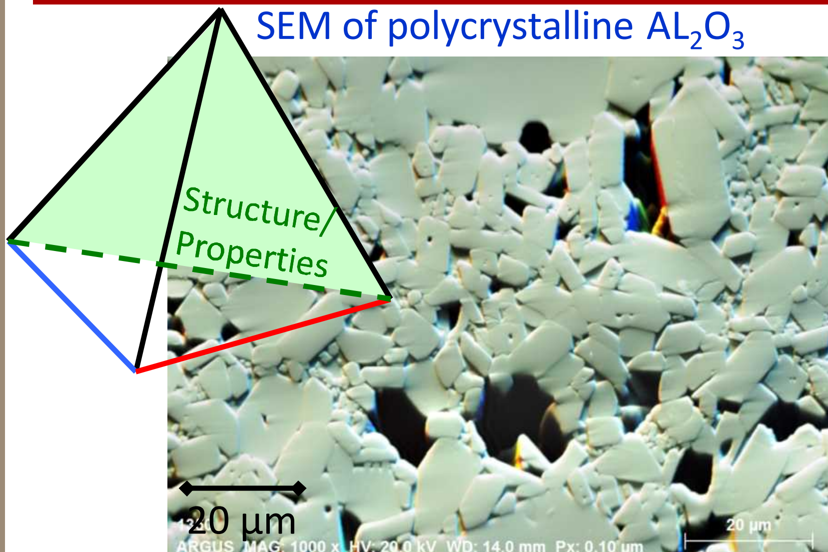
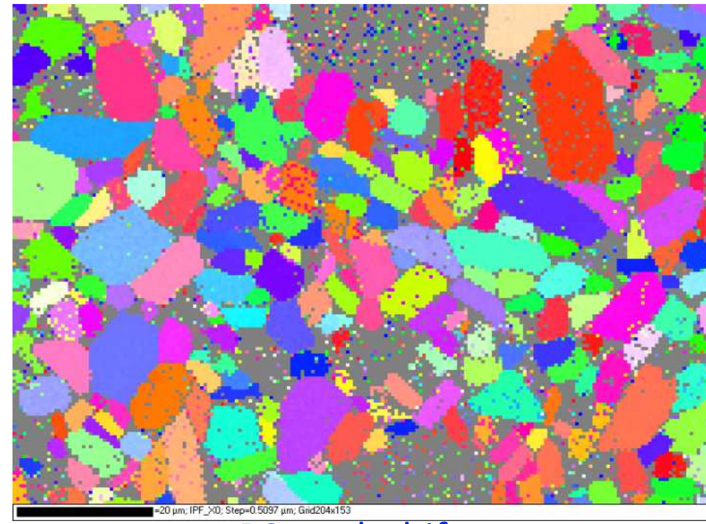
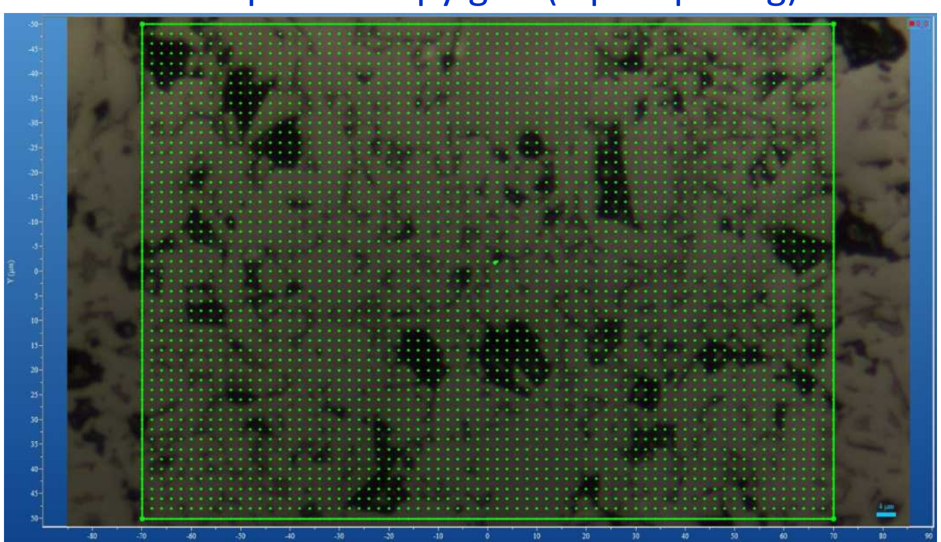
SEM of polycrystalline AL_2O_3



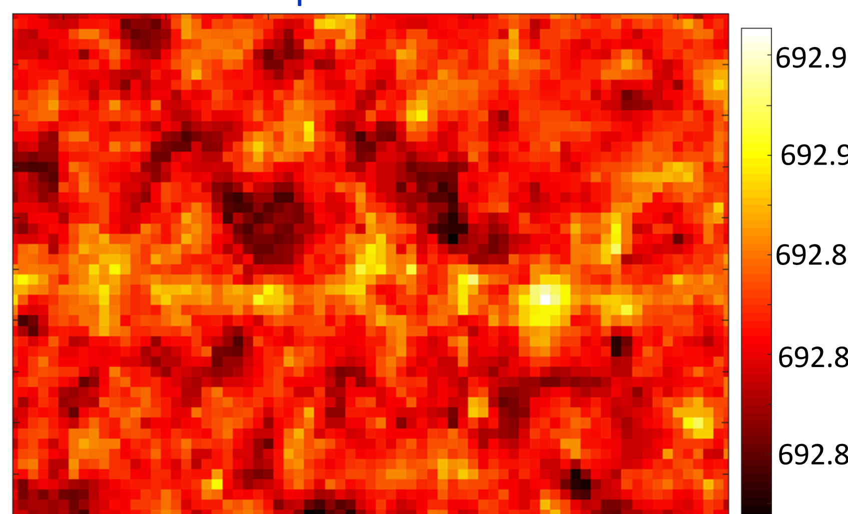
EBSD map (0.5 µm spacing)



PL Spectroscopy grid (2 µm spacing)



R2 peak shift



PL Spectroscopy Was Used To Measure Stress-Sensitive Cr Emission Band Peak Shifts

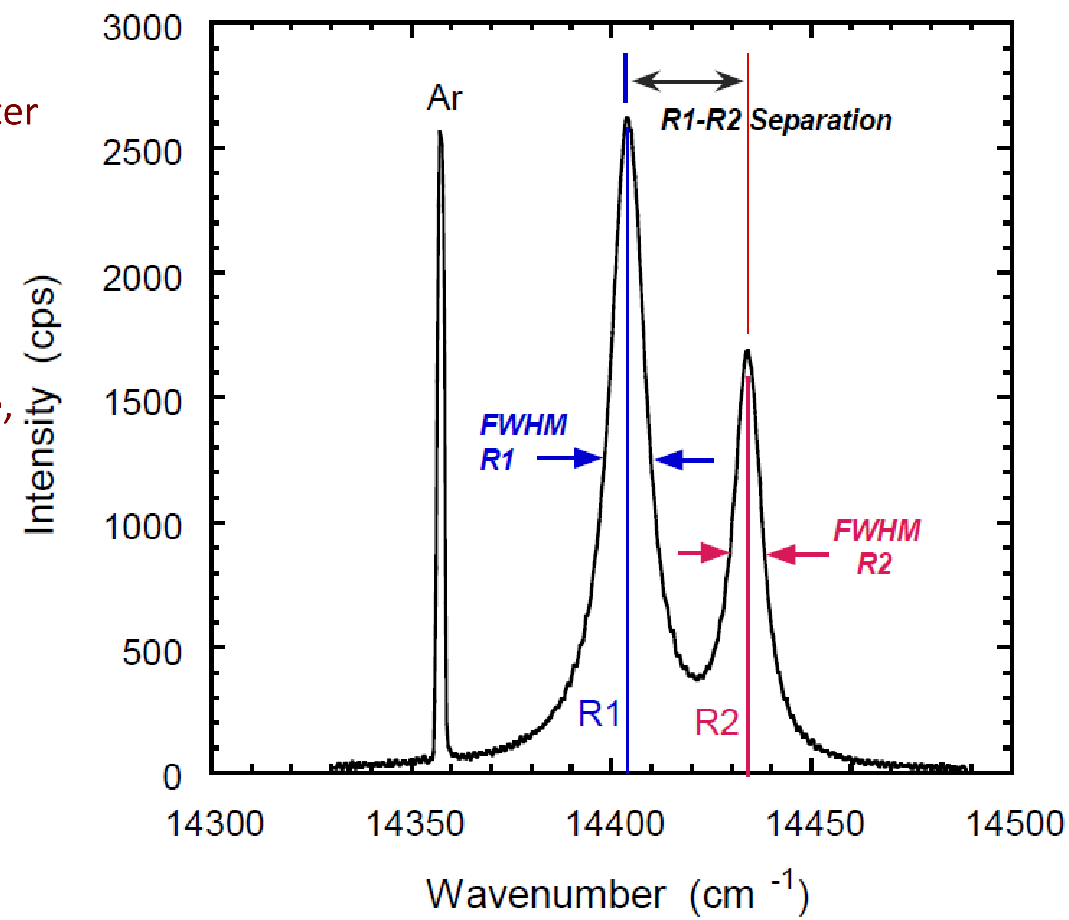
Photoluminescence (PL) Spectroscopy

Equipment

- Horiba Labram HR Raman Spectrometer
- 532 nm, 15 mW Laser
- Confocal microscope

Measurement

- Peaks shift due to stress, temperature, & Cr concentration
- Crystal orientation dependent signal
- R1 – R2 separation variation provides additional stress-state information
- Peak width provides stress-state information
- Ar line corrected instrument drift



A State-Of-The-Art PL Spectroscopy Capability Has Been Developed To Map Multi-Scale Stress

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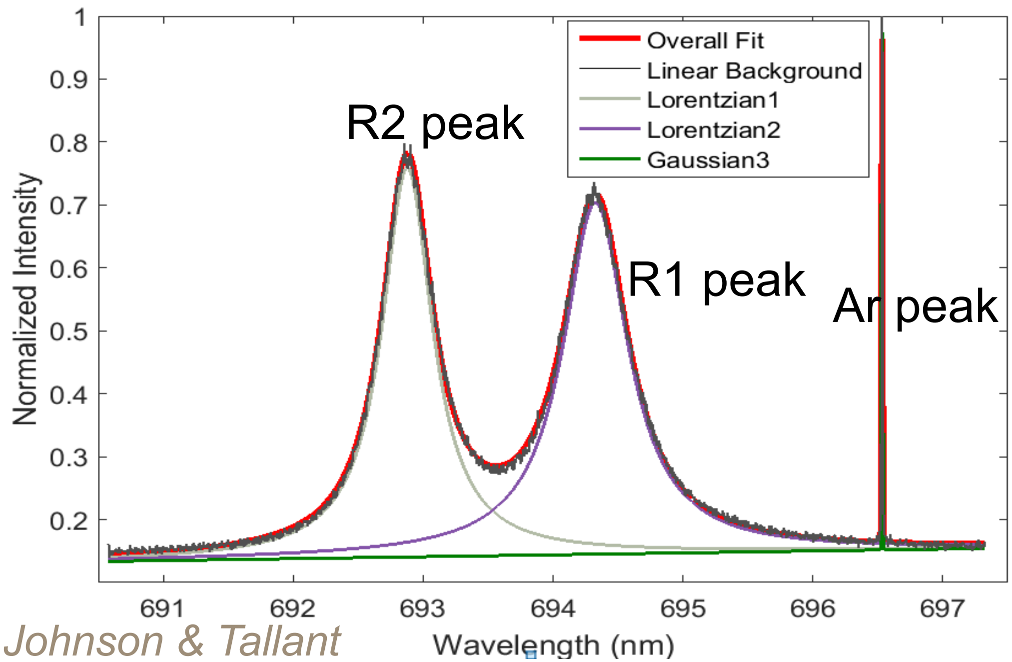
Map mm^2 areas using thousands of spectra with micrometer-scale spatial resolution

- Focused laser beam for micro-scale spatial resolution
- Determine stress sensitive R1 (~694.24 nm) and R2 (~692.84 nm) Ruby ($Al_2O_3:Cr^{3+}$) PL bands
- Automated specimen repositioning and spectra acquisition
- Process thousands of spectra in minutes (MATLAB, GRAMS, & LABSPEC)

Wavelength resolution and stability to resolve peak shits to 0.001 nm (~3MPa)

- Instrument drift corrected to Ar lamp emission line NIST reference (696.5431 nm)
- Temperature stable to 0.1°C (0.0007 nm peak shift) and temperature corrected
- Working to correct for Cr concentration (1 wt.% Cr – 4.8 nm peak shift)



- R1 & R2 fluorescence peaks (positions, widths, and separations) are best fit with a Lorentzian or pseudo-Voigt fit
- The Ar peak is best fit with a Gaussian fit

Johnson & Tallant

Converting Peak Shifts to Stress

$$\Delta\nu\ (1/\text{cm}) = \Pi_{ij}\left(\frac{\text{cm}^{-1}}{\text{GPa}}\right)\sigma_{ij}(\text{GPa})$$

if $\Pi_{11} = \Pi_{22} = \Pi_a$ and $\Pi_{33} = \Pi_c$ this can be put in the form

$$\begin{bmatrix}\Delta\nu^{(1)} \\ \Delta\nu^{(2)}\end{bmatrix} = \begin{bmatrix}\Pi_M^{(1)} & \Pi_S^{(1)} \\ \Pi_M^{(2)} & \Pi_S^{(2)}\end{bmatrix}\begin{bmatrix}\sigma_M \\ \sigma_S\end{bmatrix} \quad \text{where} \quad \begin{matrix}\Pi_M^{(i)} = \Pi_c^{(i)} + 2\Pi_a^{(i)} & \Pi_S^{(i)} = \Pi_c^{(i)} - \Pi_a^{(i)} \\ \sigma_M = (\sigma_{11}+\sigma_{22}+\sigma_{33})/3 & \sigma_S = (2\sigma_{33}-\sigma_{11}-\sigma_{22})/3\end{matrix}$$

C. A. Michaels, R. F. Cook, “Determination of residual stress distributions in polycrystalline alumina using fluorescence microscopy” *Materials and Design*, 107, (2016), 478-490

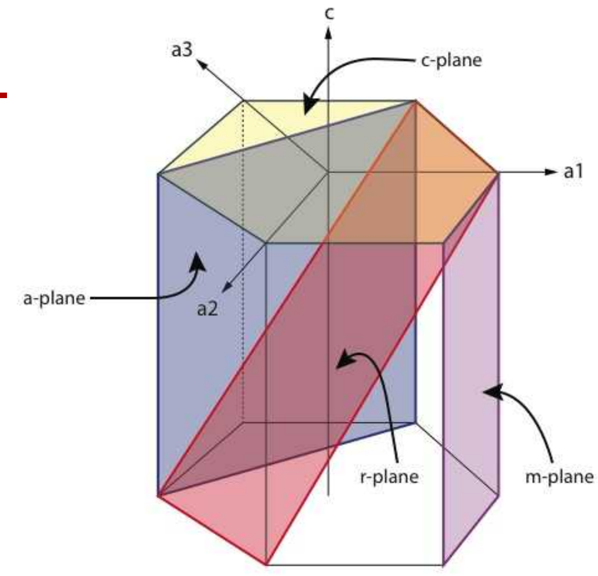
Table I. Piezospectroscopic Coefficients for Ruby

	Piezospectroscopic coefficients (cm⁻¹/GPa)						
	Previous results			Our results			
	Uniaxial loading		Hydrostatic loading pressure coeff P[†]				
	Π_{11}	Π_{33}		Π_{11}	Π_{22}	Π_{33}	$\Pi_{11} + \Pi_{22} + \Pi_{33}$
R1	3.0[‡] 3.2[§] 2.7[¶]	1.8[‡] 1.4[§] 1.8[¶]	7.59[††]	2.56	3.50	1.53	7.59
R2	2.8[‡] 2.8[§] 2.4[¶]	2.3[‡] 1.9[§] 2.2[¶]	7.615[††]	2.65	2.80	2.16	7.61

[†] $P = \Pi_{11} + \Pi_{22} + \Pi_{33}$ [‡]Reference 4, room temperature(?). [§]Reference 7, 77 K. [¶]Reference 6, 77 K. [††]Reference 12, room temperature.

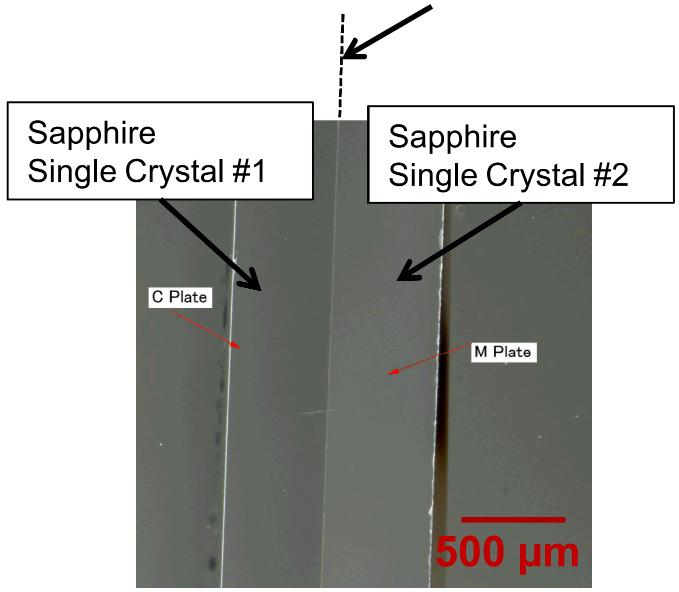
Bi-crystal samples for Spectroscopic stress measurement

- **Sputtered ~100nm of glass on polished surface of single crystals (Eagle glass from corning)**
- **Crystals were then placed glass side together on alumina boat**
- **Crystals were heated to 1600°C at 20°C/min and held for 1 hr followed by air cool in furnace**



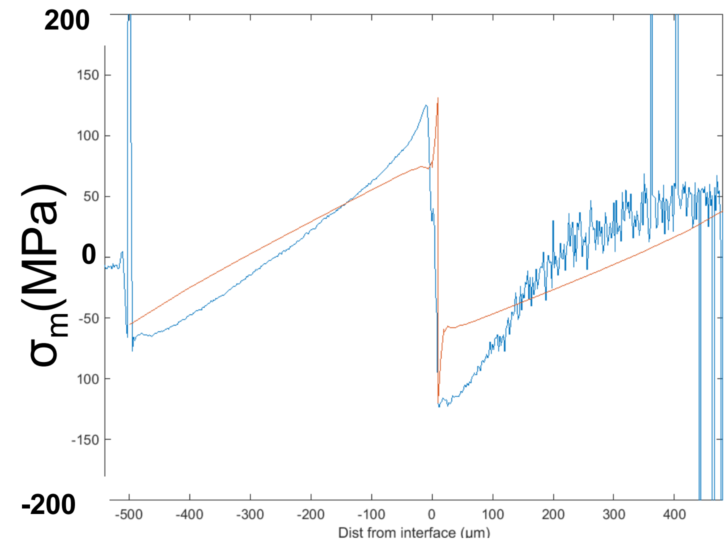
<u>Successfully bonded Bi-crystals:</u>

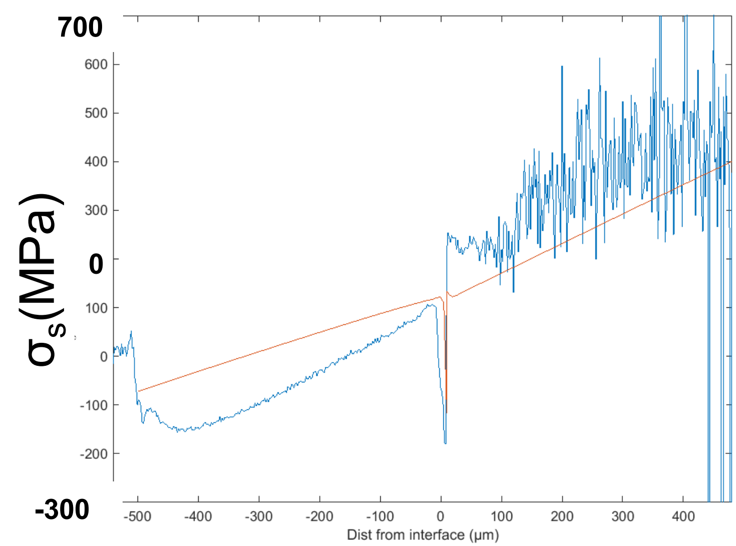
- **C-plane to C-plane, rotated 30°**
- **A-plane to C-plane**
- **A-plane to M-plane**



Stress results, compare to FE

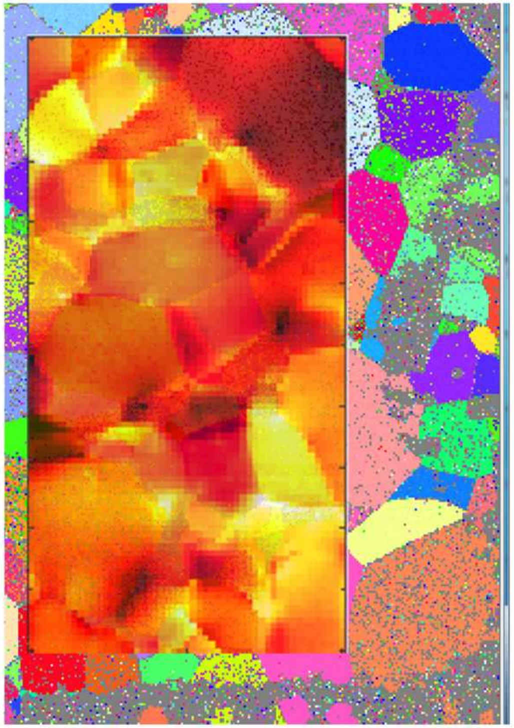
- σ_M calculated from PL data has same shape as that from FE model
- There is currently a scaling uncertainty
  - What temperature change should be applied in FE model?
  - This plot uses ΔT=1600 °C but glass layer doesn't support stress for high temperatures
  - Currently only using linear, room temperature CTE values





Microstructural Meshing <u>REAL</u> microstructures

- Generated microstructures are often insufficient for model validation with experiments
- Equivalent microstructures is vaguely defined, in order to do first order model validation need microstructure upon which experiments performed

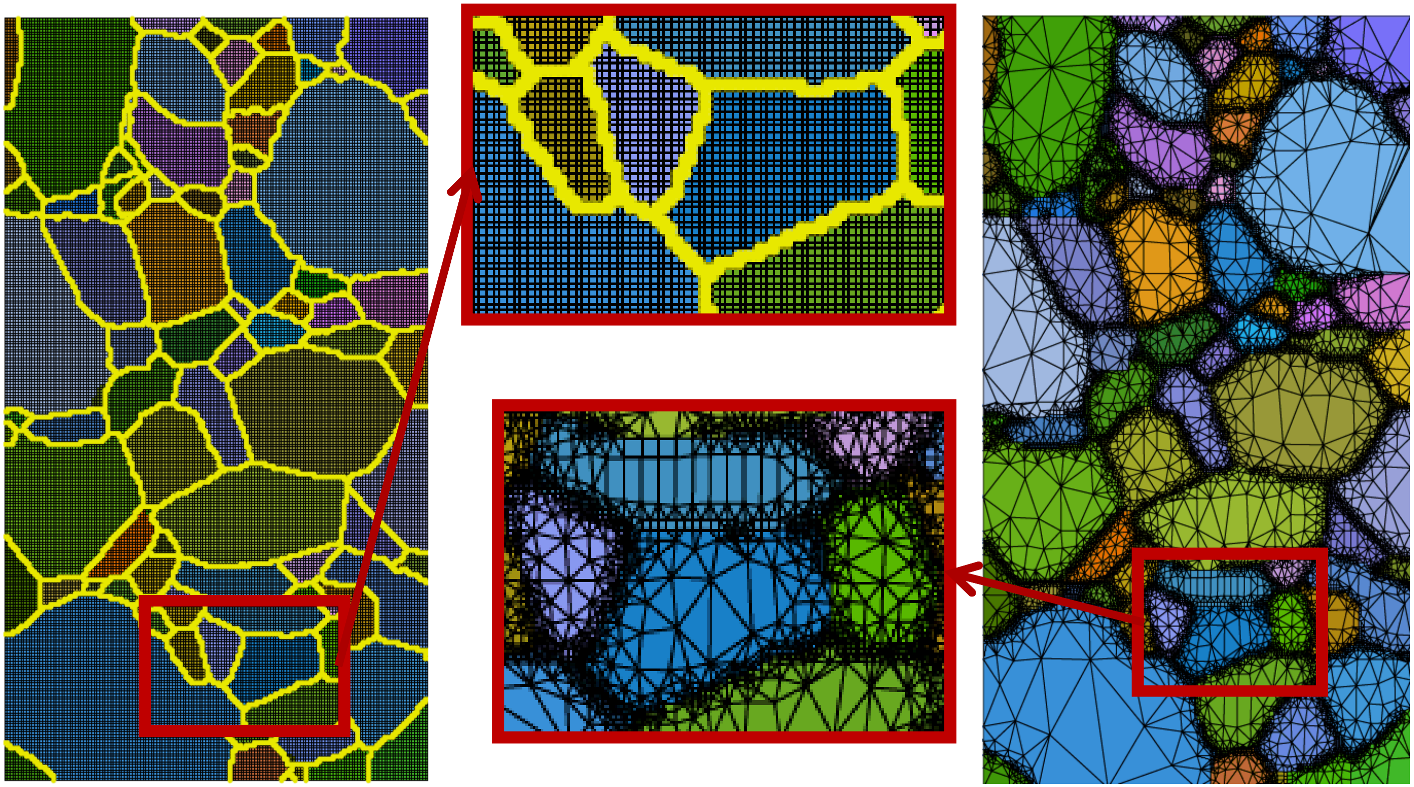


EBSD map aligned with experiment mapped for stress

Meshing Schemes

- Multiple commercial and open source meshing software's available
- Common issue is number of elements to capture microstructure can become extremely large
- Fall into two basic categories
  - Point-by-point, or element for every pixel/data point
    - Cubit Sculpt, MOOSE EBSD/Image reader, etc
  - Software that enables input from multiple data sources
    - OOF2/3D, Avizo, etc

Comparisons of Meshing Schemes

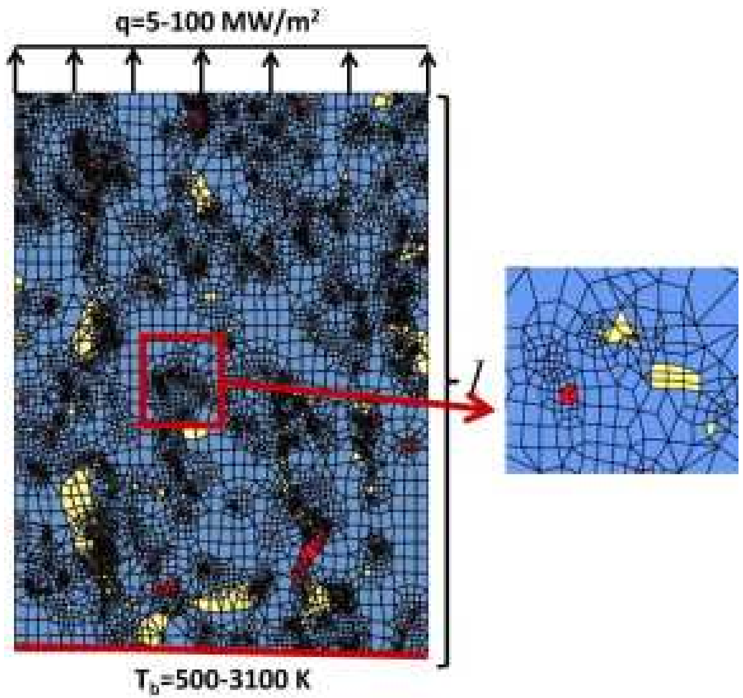


	Element per point	OOF2
$N_{elements}$	44274	18090
N_{nodes}	43836	27987

Significant reduction in computational power for OOF2 meshes

Have your mesh now what?

- Need material properties, including anisotropy
- Need to be able to rotate elasticity and other tensor properties by orientation
- MOOSE is great option for adaptive rapid development



Mesoscale Modeling with the MOOSE framework

- All of the code required to easily create your own phase field application is in the open source MOOSE modules (MOOSE-PF).

MOOSE

Framework

All of the code that forms the basis of the MOOSE framework

Modules

Phase Field
- Cahn-Hilliard and Allen-Cahn equations
- Free energy based development

Tensor Mechanics
- Linear elasticity
- Eigenstrains
- J2 Plasticity
- Crystal plasticity

Heat Conduction
- Steady state
- Transient
- Locally varying thermal conductivity

Credit: Mooseframework.org

Model Set up

elastic constants

S_{11}= 497 GPa S_{33}= 501 GPa

S_{44}= 147 GPa S_{12}= 163 GPa

S_{13}= 116 GPa S_{14}= -22 Gpa

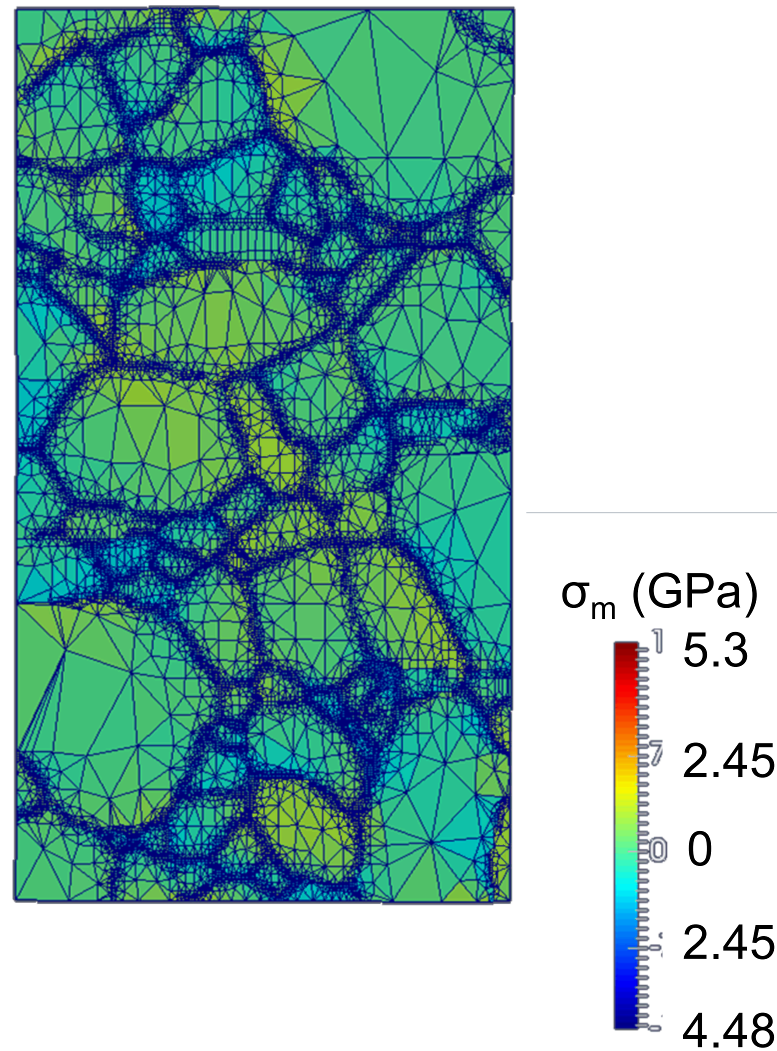
- Goto et. al. Journal of Geophysical Research, 1989

Coefficients of thermal expansion

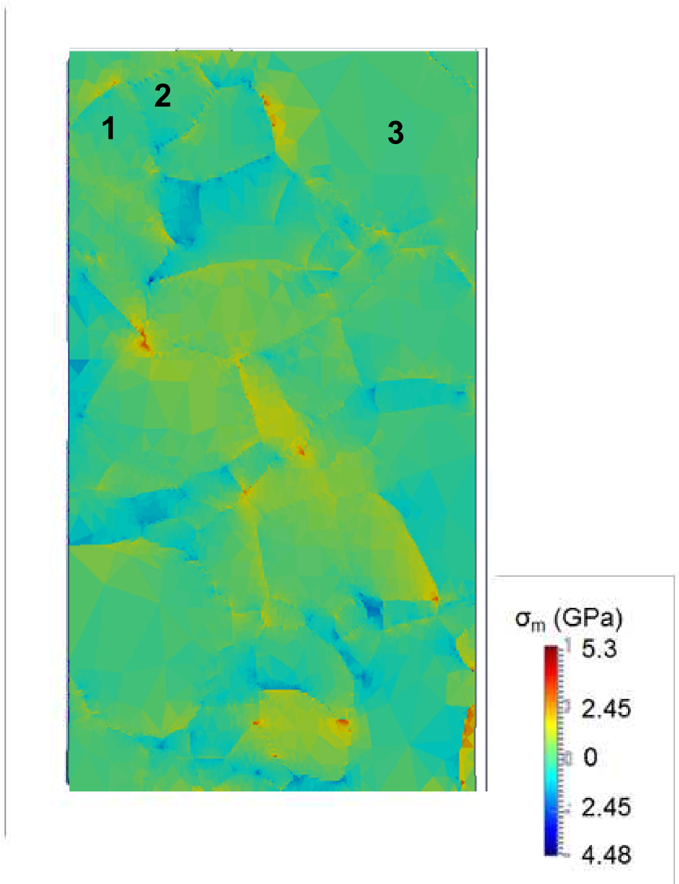
α_{11} = 7.3e-06 1/°C

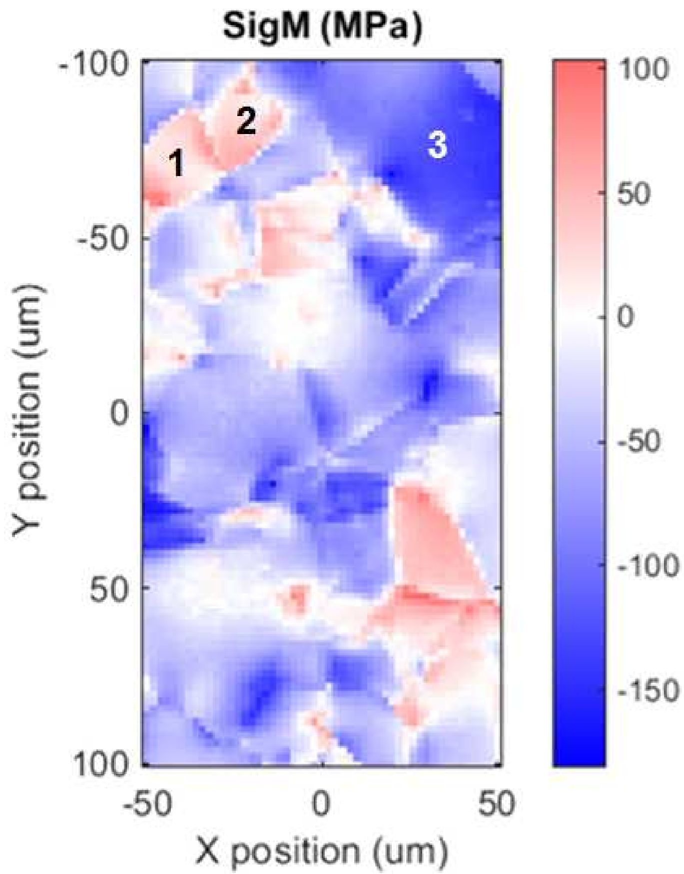
α_{33} = 8.2e-06 1/°C

ΔT=1500°C



Comparison to Experiment

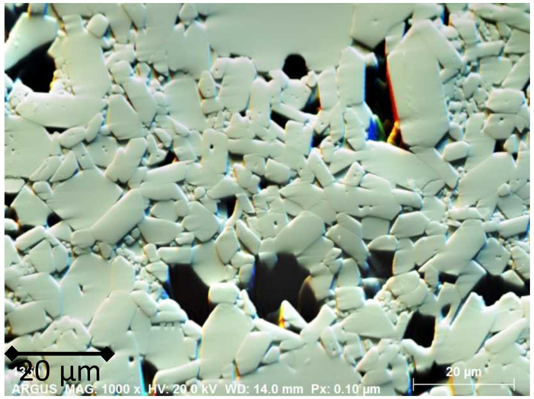




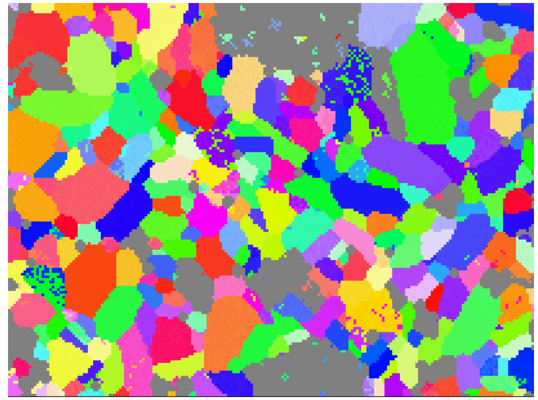
General Agreement-need to refine for resolution and 2D-3D reality

Grain	σ_m (MPa)
1	49
2	206
3	-52

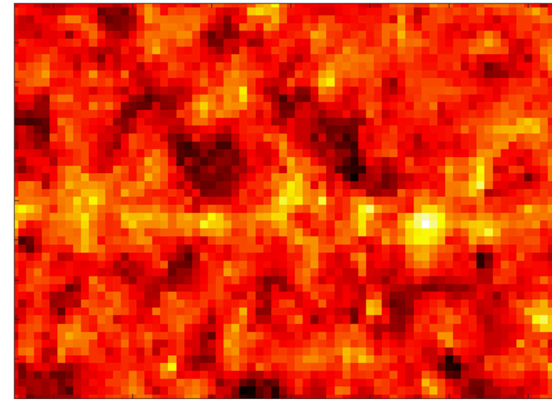
Experimentally-Informed Microstructure Modeling Capability Is Being Developed To Predict Stress



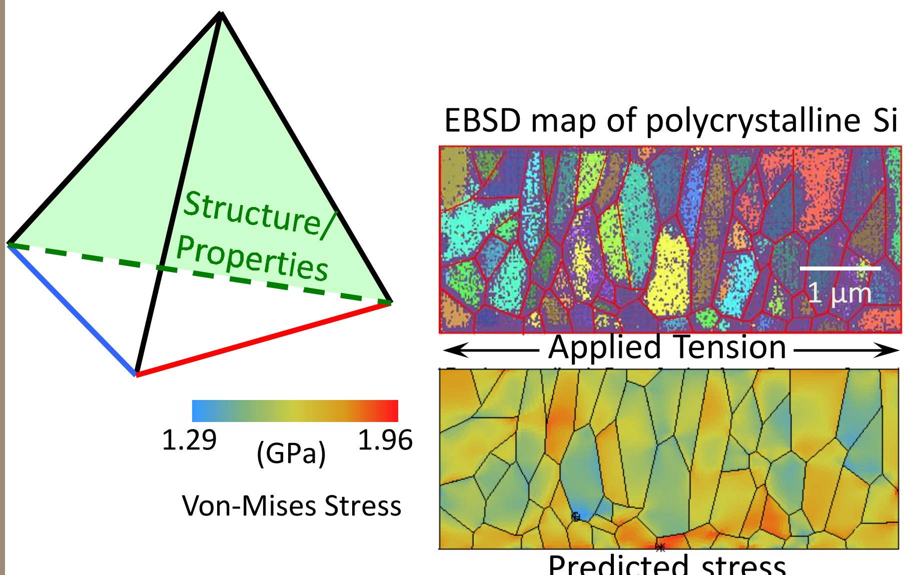
SEM of Al_2O_3



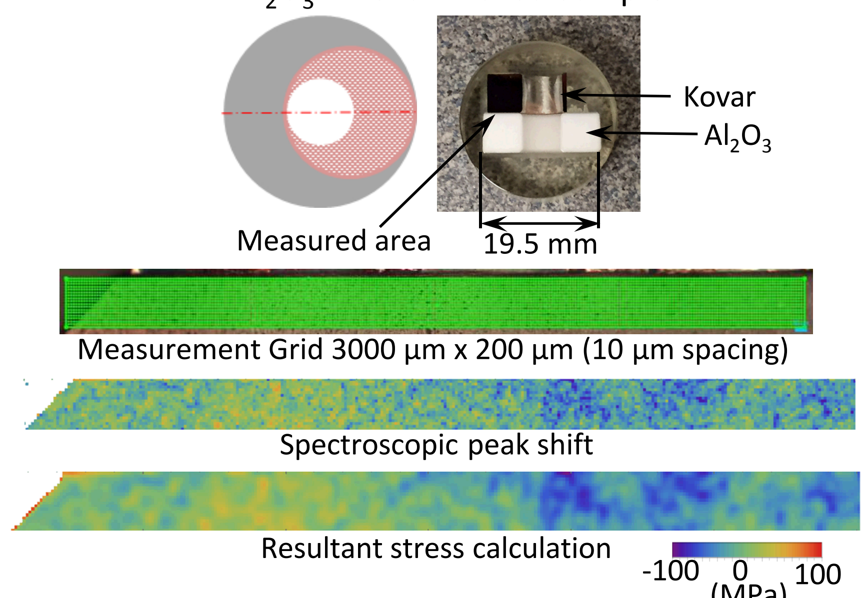
EBSD map of Al_2O_3



Stress map of Al_2O_3



FE simulation of micro-scale stress & variation in a brittle material microstructure



PL spectroscopy used for micro- to continuum-scale stress measurement

Buchheit, Teague, Johnson, Tallant, Meserole, & Tandon

Conclusions

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- Photo-luminesicne spectroscopy mapping is promising technique for enabeling validation of stress at critical microstructural level
- Ease/speed of experiments enables large areas to be measured
- Microstructure meshing and modeling are primed to take advantage of this new data to enable mechanistic based models of brittle failure

Future Work

- Perform external loading experiments on single crystals
- Perform loading experiments on tested microstructures and compare
- Move to more complicated microstructures
- Study interaction of loading and microstructure defects/properties
- Improve models with experimental validation

Questions

Backup Slides

Converting Peak Shifts to Stress

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C. A. Michaels, R. F. Cook, "Determination of residual stress distributions in polycrystalline alumina using fluorescence microscopy" *Materials and Design*, 107, (2016), 478-490

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R2	2.8‡ 2.8§ 2.4¶	2.3‡ 1.9§ 2.2¶	7.615††	2.65	2.80	2.16	7.61

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