

# Characterization and Modeling of Microstructural level Stresses in Brittle Materials

M.C. Teague, S.J. Grutzik, T.E. Buchheit,, R.L. Johnson,  
S.P. Meserole, and K.G. Ewsuk

Sandia National Laboratories  
Albuquerque, NM 87185

Sandia National Laboratories is a multi-program laboratory managed and operated by Sandia Corporation, a wholly owned subsidiary of Lockheed Martin Corporation, for the U.S. Department of Energy's National Nuclear Security Administration under contract DE-AC04-94AL85000.

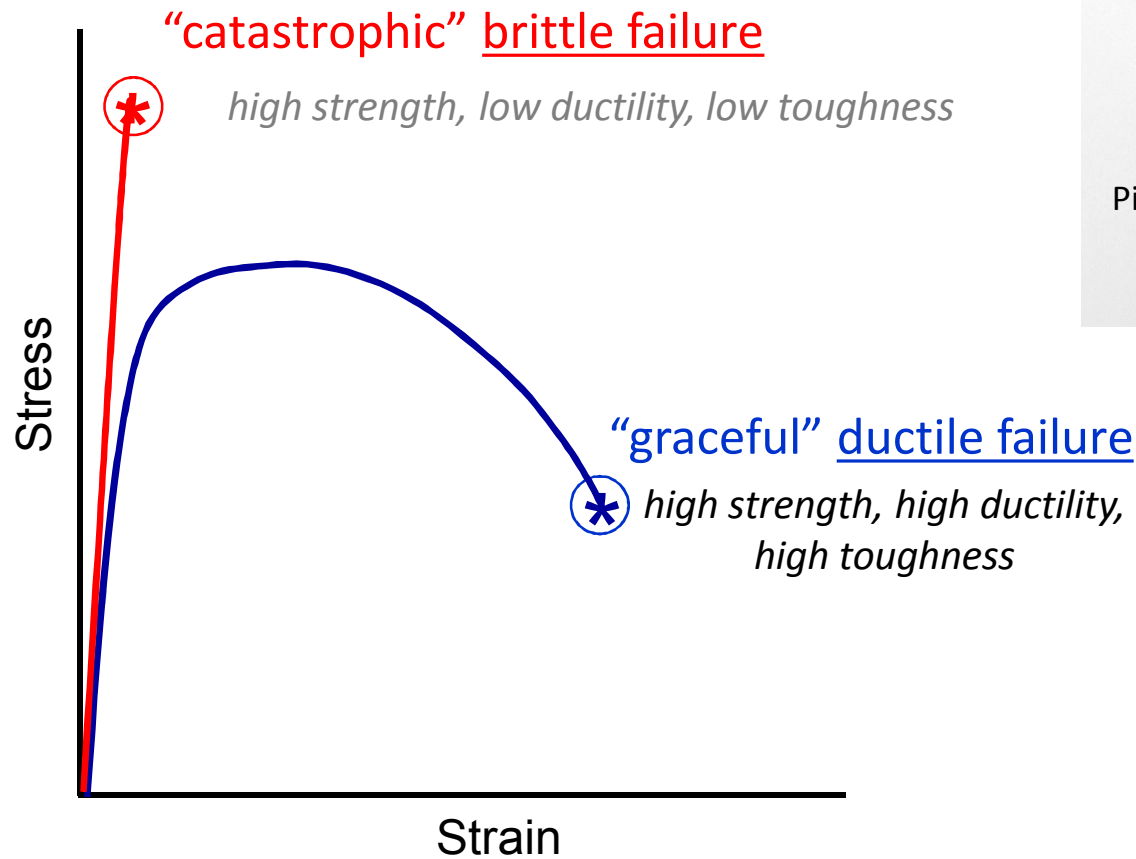


# Outline

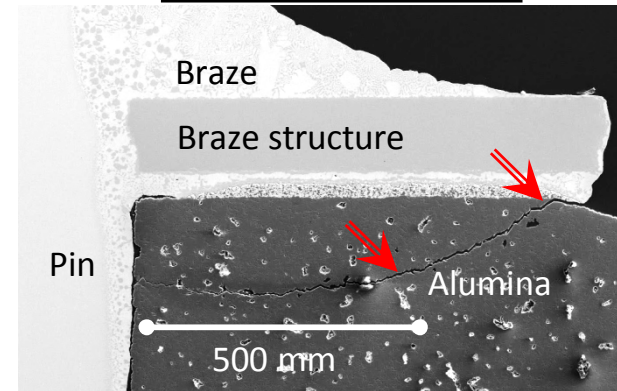
---

- Motivation
- Background
- Bi-crystal Work
- Polycrystalline work
  - Micro-structure meshing
  - Modeling of stresses
- Future Work

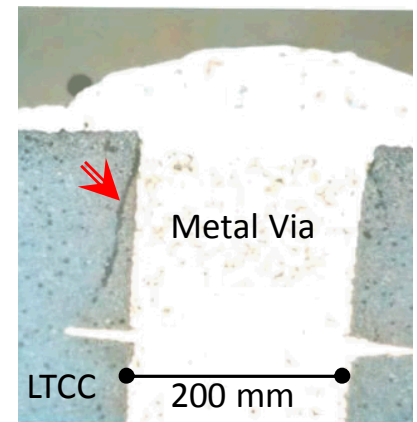
# Brittle Materials Failure Presents A Reliability Concern in High Consequence Applications



Electrical Feedthru



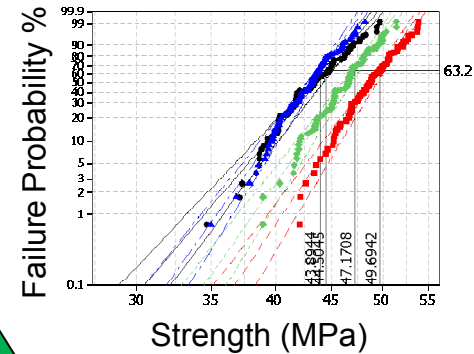
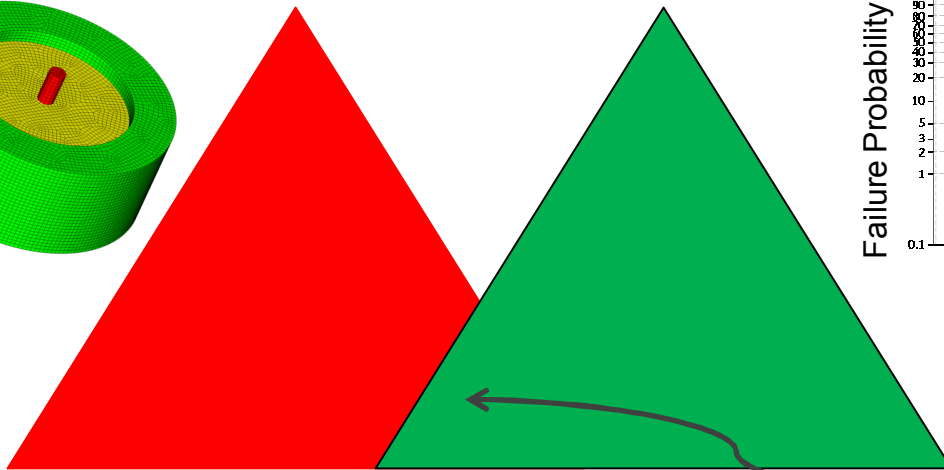
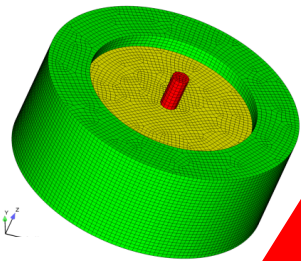
Electronic Substrate



Brittle materials are susceptible to sudden catastrophic failure

# Current State: Qualitative Stress-Based Predictions

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

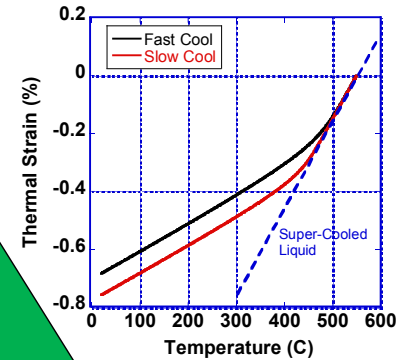
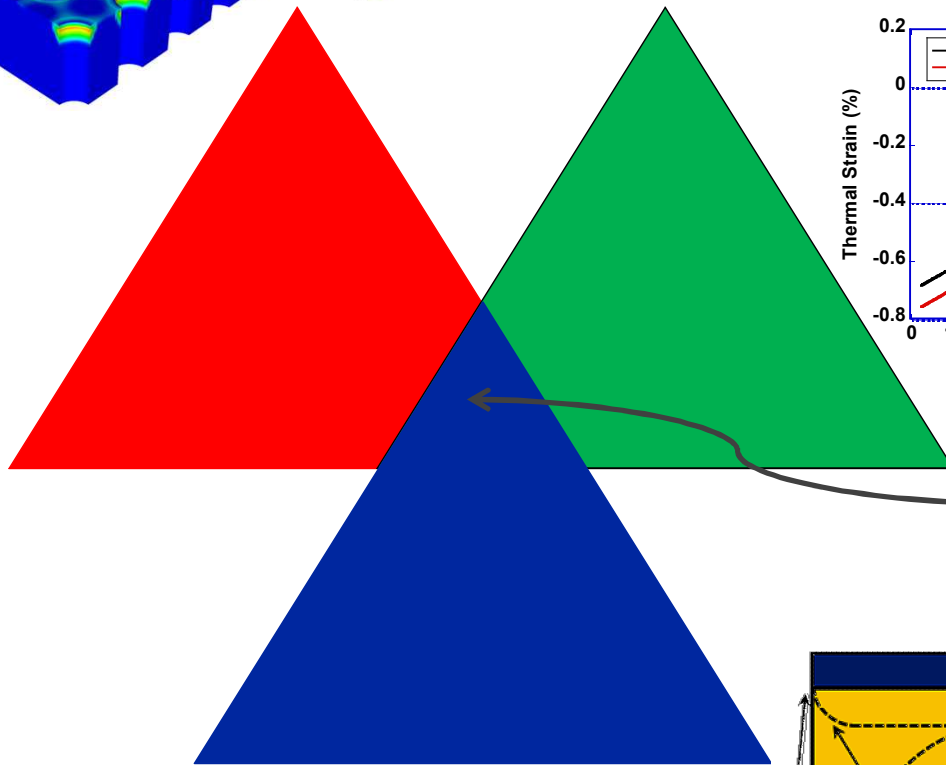
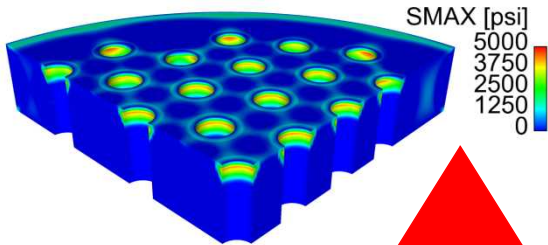


*Qualitative prediction  
of brittle failure based  
on engineering  
judgment/experience*

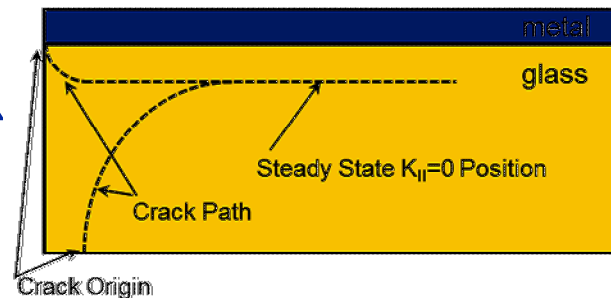
- We Design To Avoid High Stress
- Engineering Judgment Has Deficiencies
  - Limited by practical experience
  - Neglects flaws/flaw populations
  - Does not incorporate fracture mechanics

# 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 Sandia National Laboratories That Addresses The Gaps To Predict Brittle Failure

---

## Vision

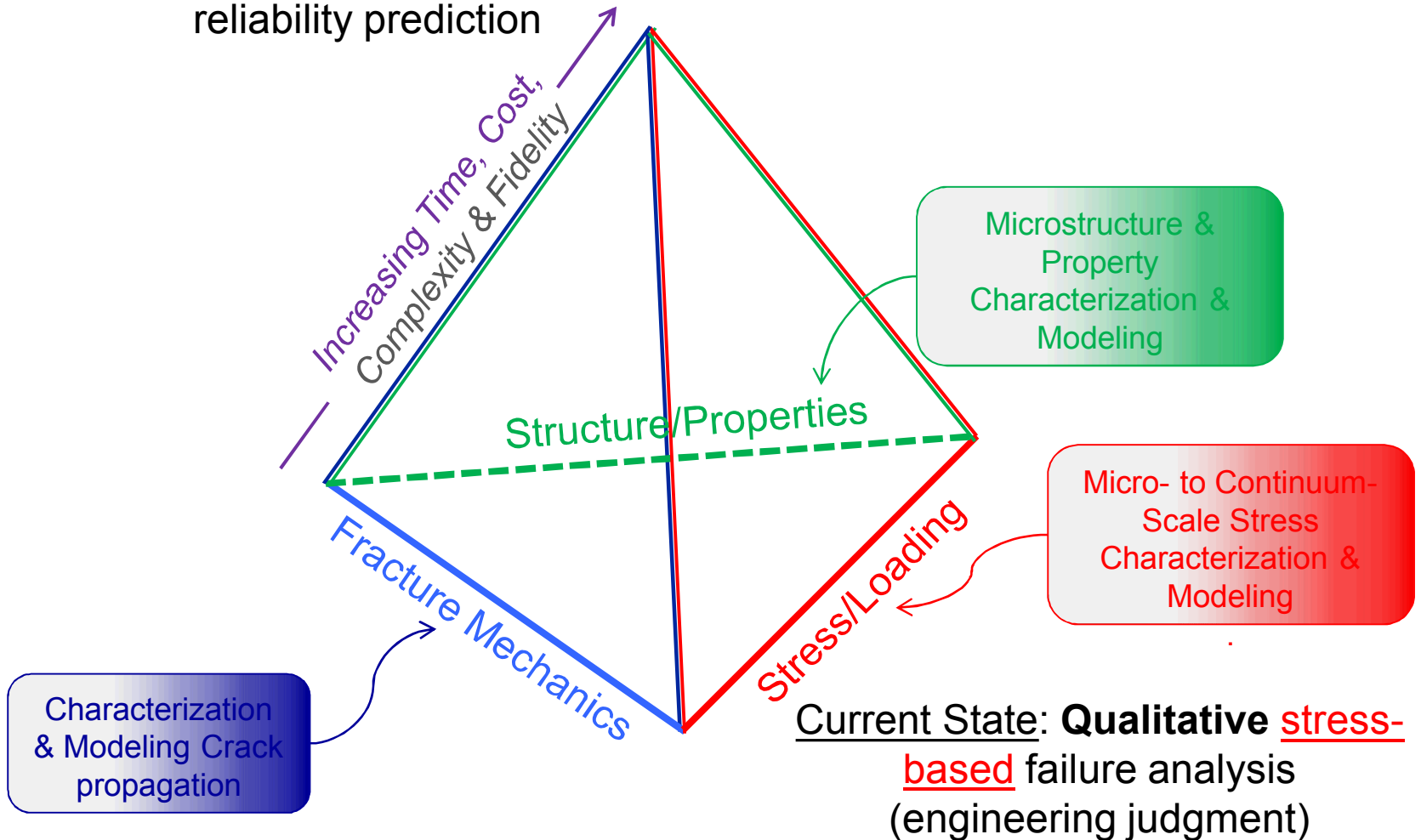
- Transition from **Qualitative** stress-based engineering judgment to **Quantitative** mechanics-based failure prediction.

## Approach

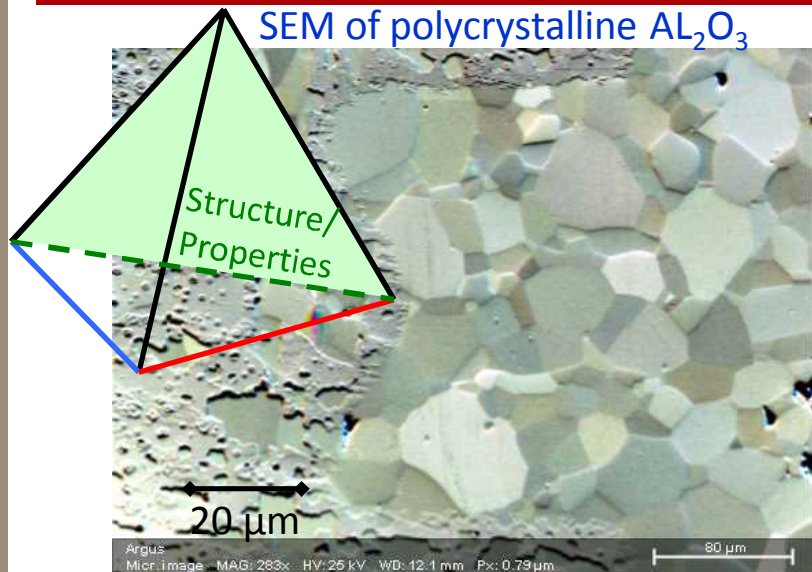
- Develop foundational materials **characterization & modeling** S&T in:
  - 1) **Stress/Loading** – physically-based models, materials & processing data, & model validation
  - 2) **Fracture Mechanics** - crack initiation & propagation, and statistical bounds & variability
  - 3) **Structure/Properties**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

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

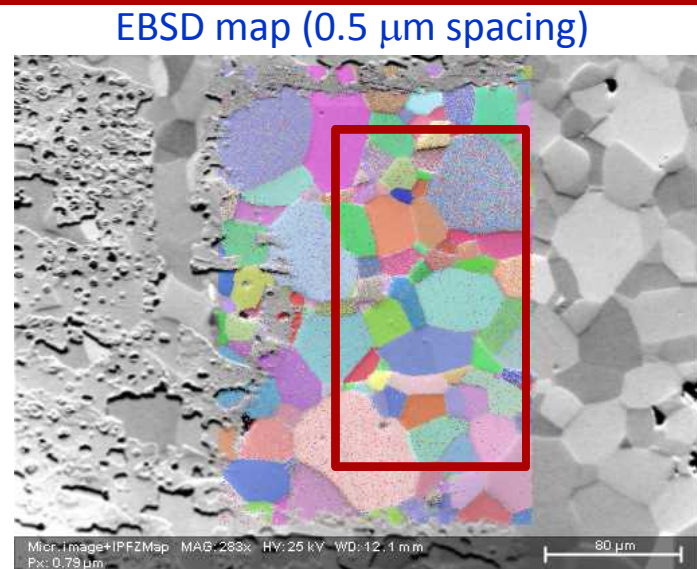
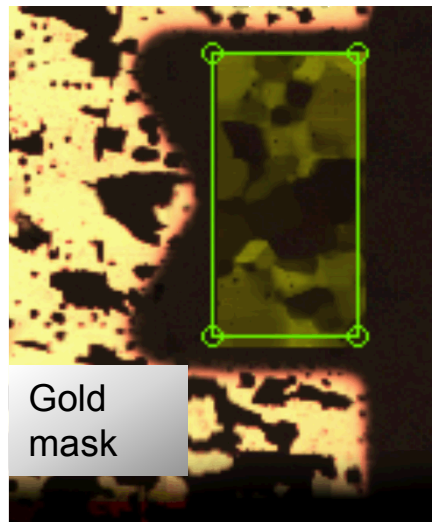
Vision: **Quantitative** mechanics-based failure & reliability prediction



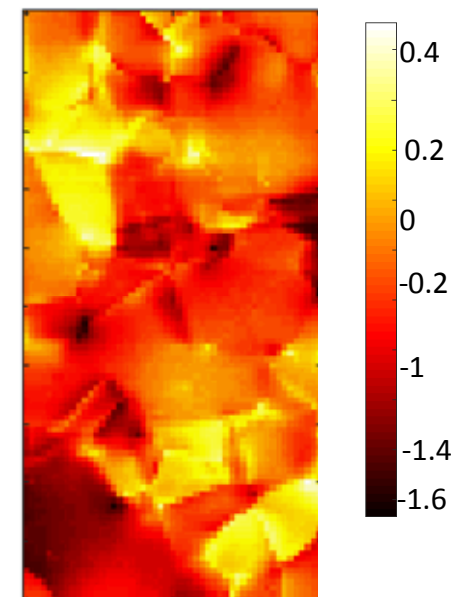
# Micro- To Continuum-Scale Stress Mapping Is Possible With SEM/EBSD And PL Spectroscopy



PL Spectroscopy grid (2  $\mu m$  spacing)



R2 peak shift



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

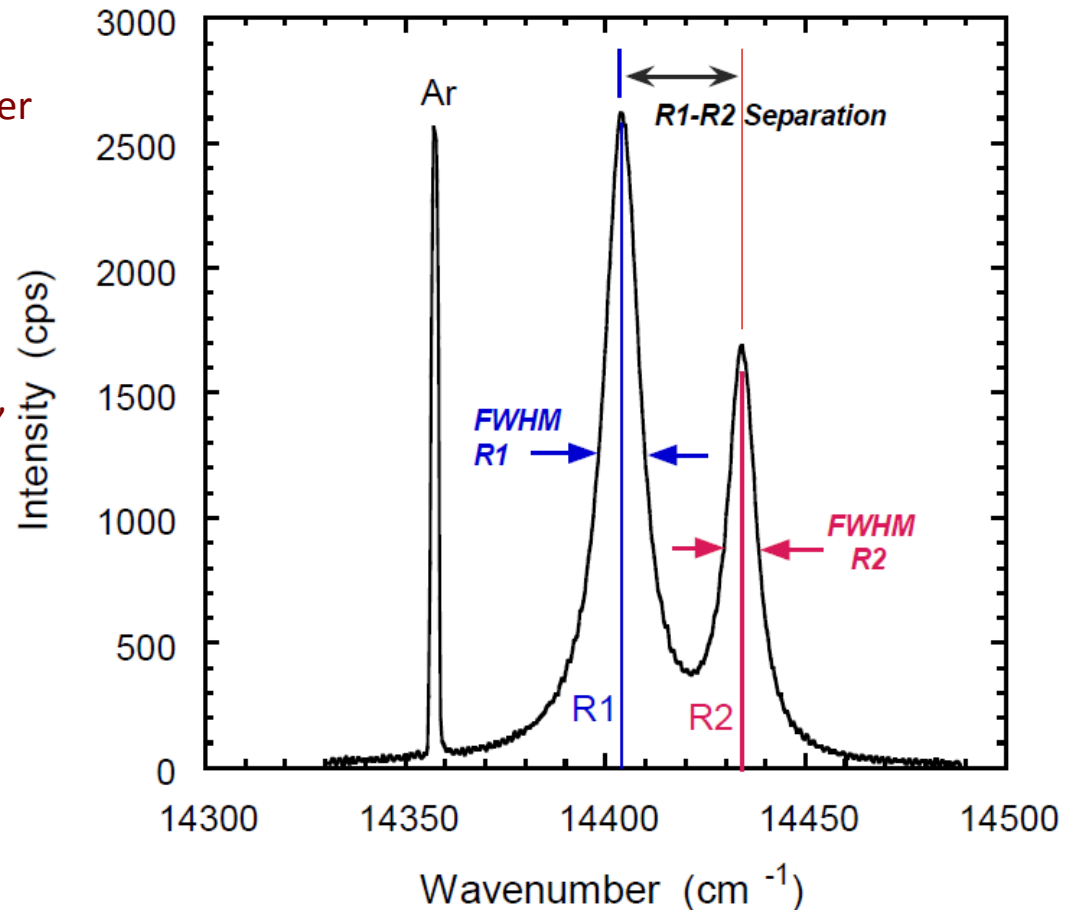
## Photoluminescence (PL) Spectroscopy

### 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



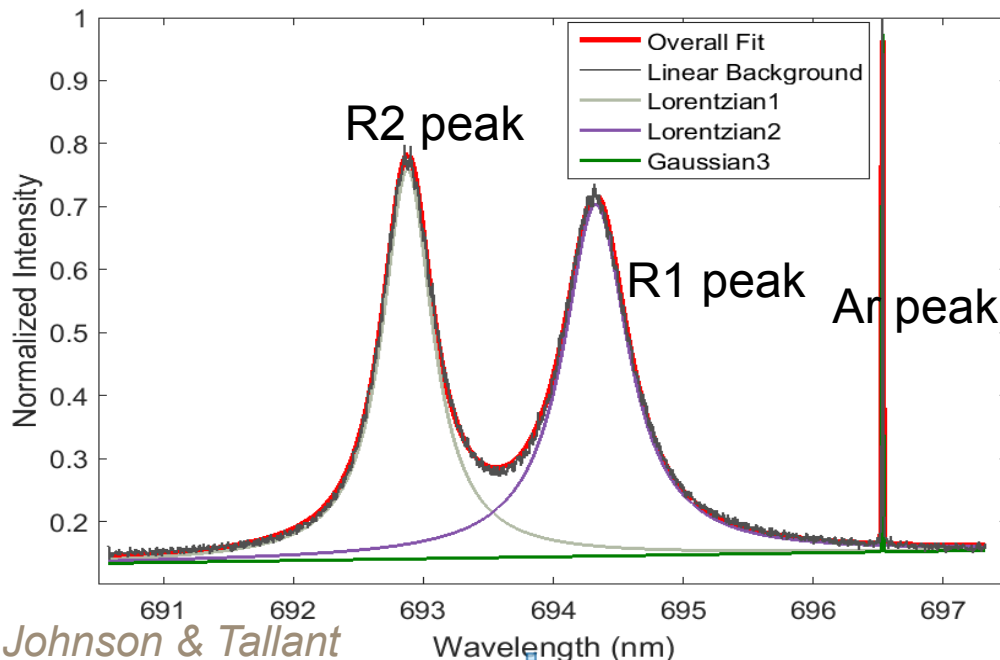
# A State-Of-The-Art PL Spectroscopy Capability Has Been Developed To Map Multi-Scale Stress

Map mm<sup>2</sup> 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<sub>2</sub>O<sub>3</sub>:Cr<sup>3+</sup>) PL bands
- Automated specimen repositioning and spectra acquisition
- Process thousands of spectra in minutes (MATLAB, GRAMS, & LABSPEC)

Wavelength resolution and stability to resolve peak shifts 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

# Converting Peak Shifts to Stress

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

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

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

$$\sigma_M = (\sigma_{11} + \sigma_{22} + \sigma_{33})/3 \quad \sigma_S = (2\sigma_{33} - \sigma_{11} - \sigma_{22})/3$$

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

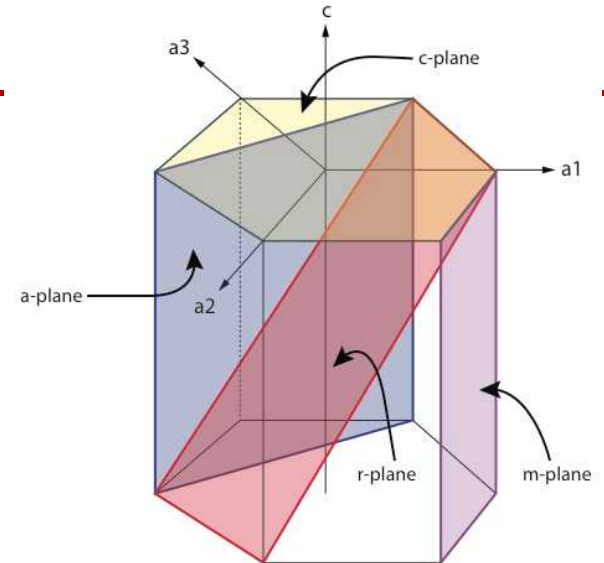
**Table I. Piezospectroscopic Coefficients for Ruby**

	Piezospectroscopic coefficients (cm <sup>-1</sup> /GPa)						
	Previous results			Our results			
	Uniaxial loading		Hydrostatic loading pressure coeff P <sup>1</sup>	Π <sub>11</sub>	Π <sub>22</sub>	Π <sub>33</sub>	Π <sub>11</sub> + Π <sub>22</sub> + Π <sub>33</sub>
Π <sub>11</sub>	Π <sub>33</sub>						
R1	3.0 <sup>‡</sup>	1.8 <sup>‡</sup>	7.59 <sup>††</sup>	2.56	3.50	1.53	7.59
	3.2 <sup>§</sup>	1.4 <sup>§</sup>					
	2.7 <sup>¶</sup>	1.8 <sup>¶</sup>					
R2	2.8 <sup>‡</sup>	2.3 <sup>‡</sup>	7.615 <sup>††</sup>	2.65	2.80	2.16	7.61
	2.8 <sup>§</sup>	1.9 <sup>§</sup>					
	2.4 <sup>¶</sup>	2.2 <sup>¶</sup>					

<sup>1</sup>P = Π<sub>11</sub> + Π<sub>22</sub> + Π<sub>33</sub> <sup>‡</sup>Reference 4, room temperature(?). <sup>§</sup>Reference 7, 77 K. <sup>¶</sup>Reference 6, 77 K. <sup>††</sup>Reference 12, room temperature.

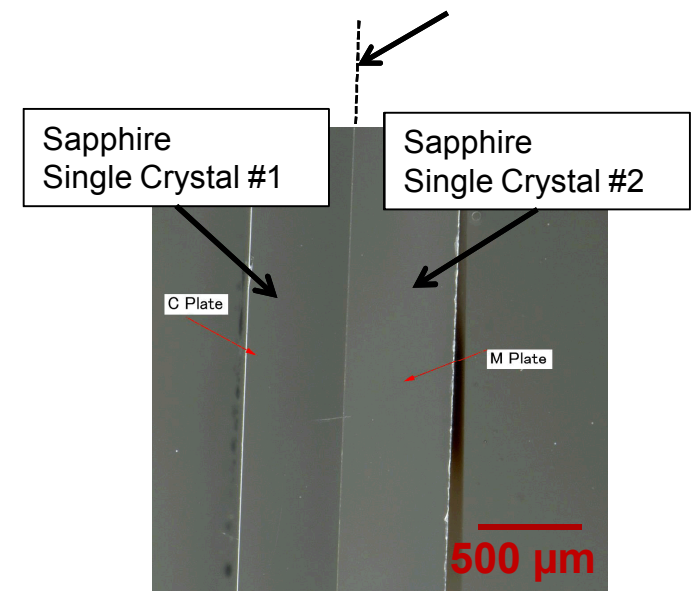
# Bi-crystal samples for Spectroscopic stress measurement

- 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



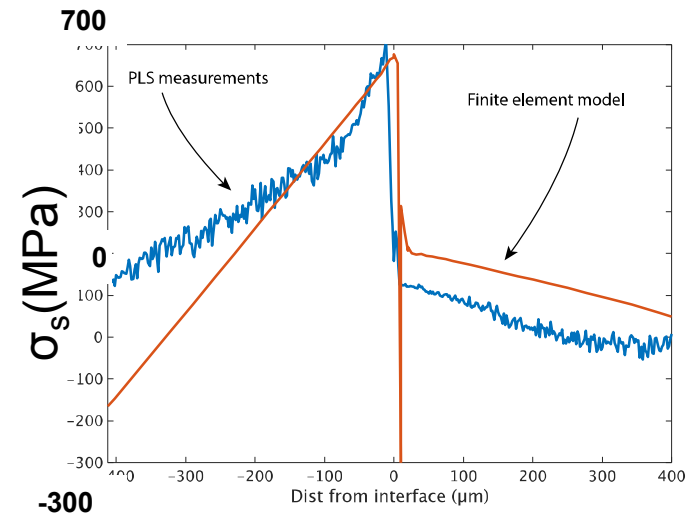
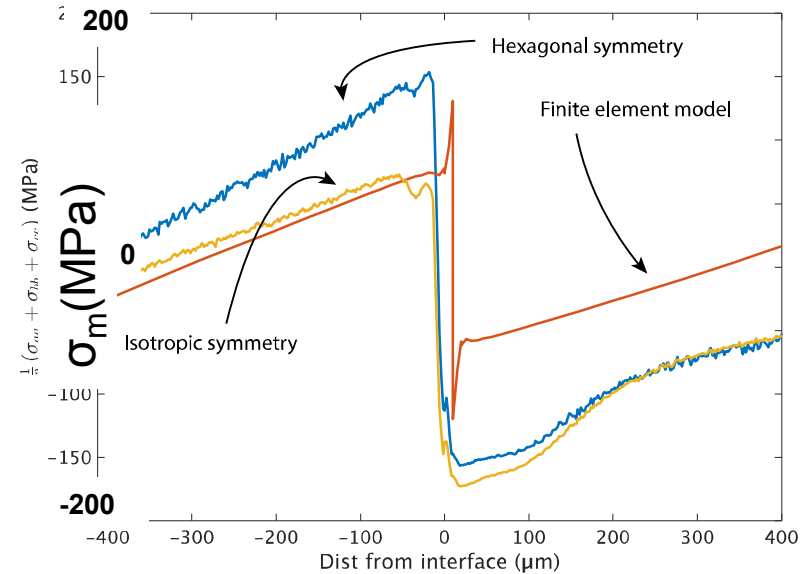
## Successfully bonded Bi-crystals:

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



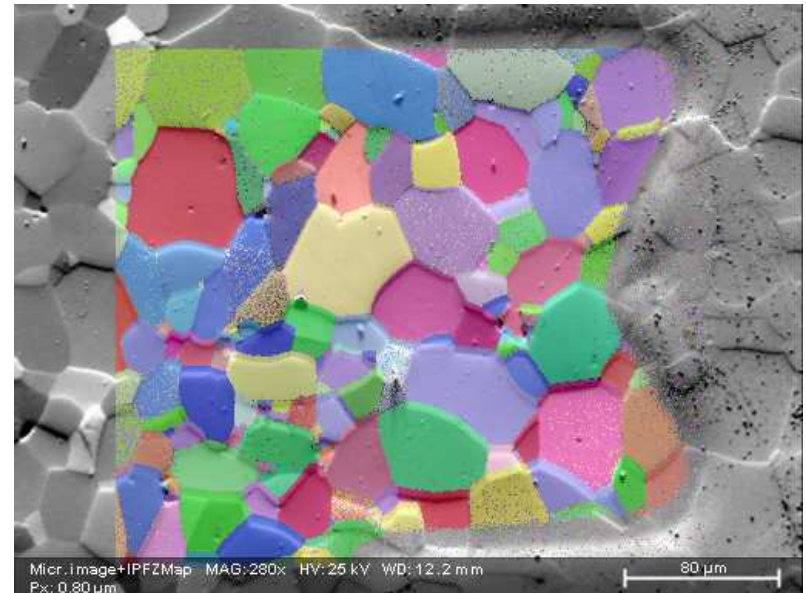
# Stress results, compare to FE

- $\sigma_M$  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  $\Delta T=1600$  °C but glass layer doesn't support stress for high temperatures
  - Currently only using linear, room temperature CTE values



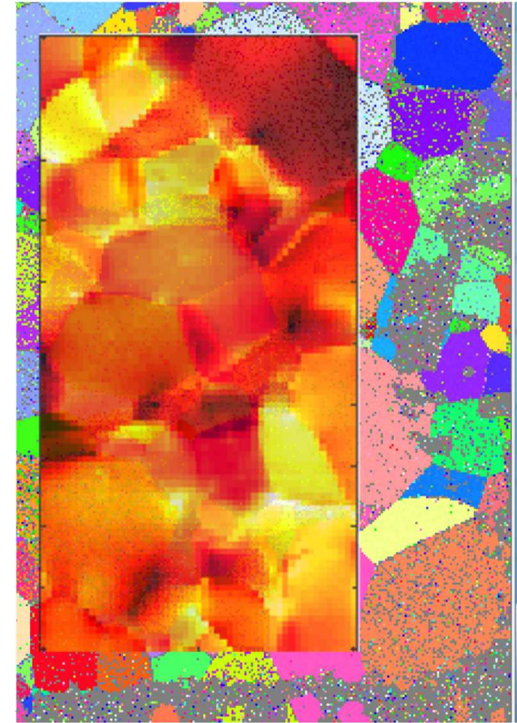
# Polycrystalline Samples

- Cr-doped to 0.025wt% Cr
- 99.5% dense
- Avg. Grain size 20  $\mu\text{m}$
- Polished to EBSD quality finish
- Masked with gold for fiducial markers



# Microstructural Meshing REAL 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

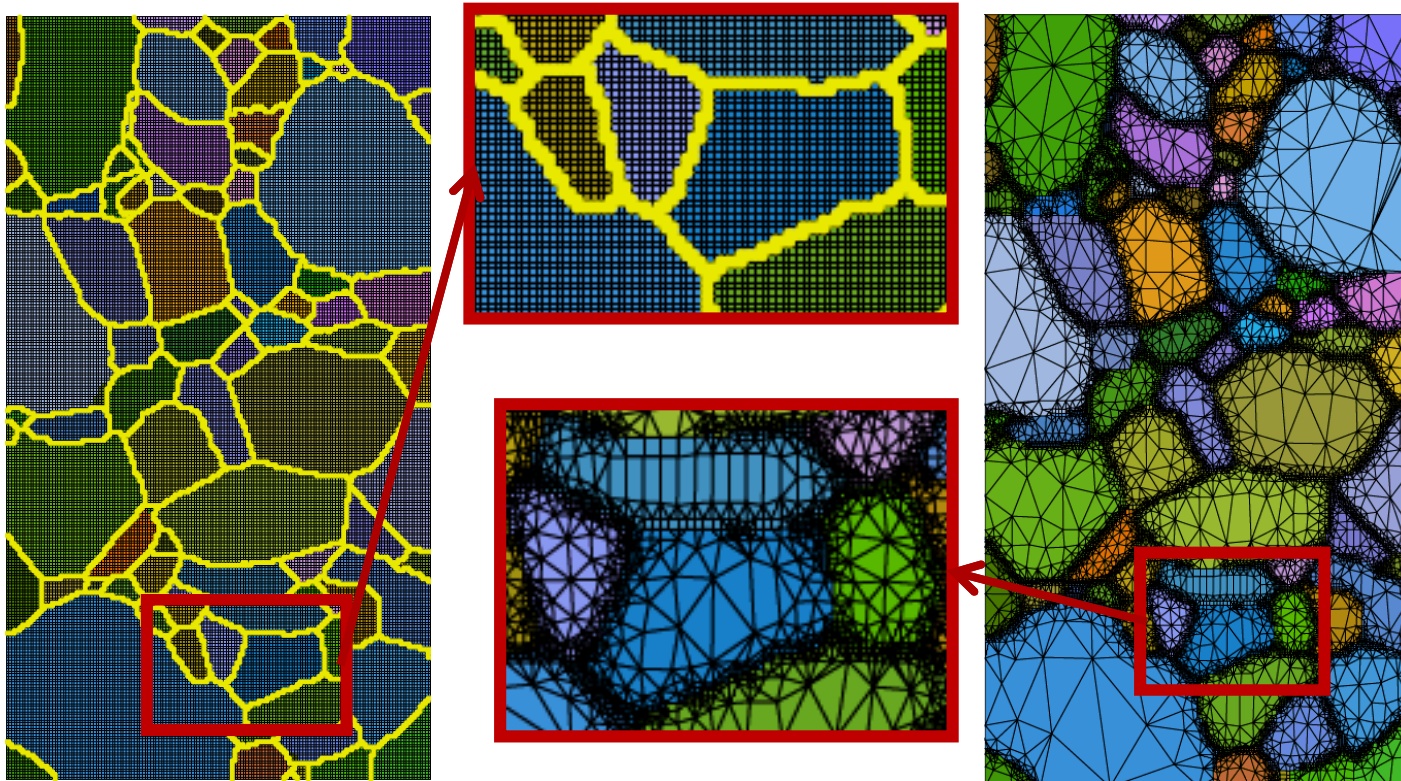


EBSD map aligned with experiment mapped for stress

# 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

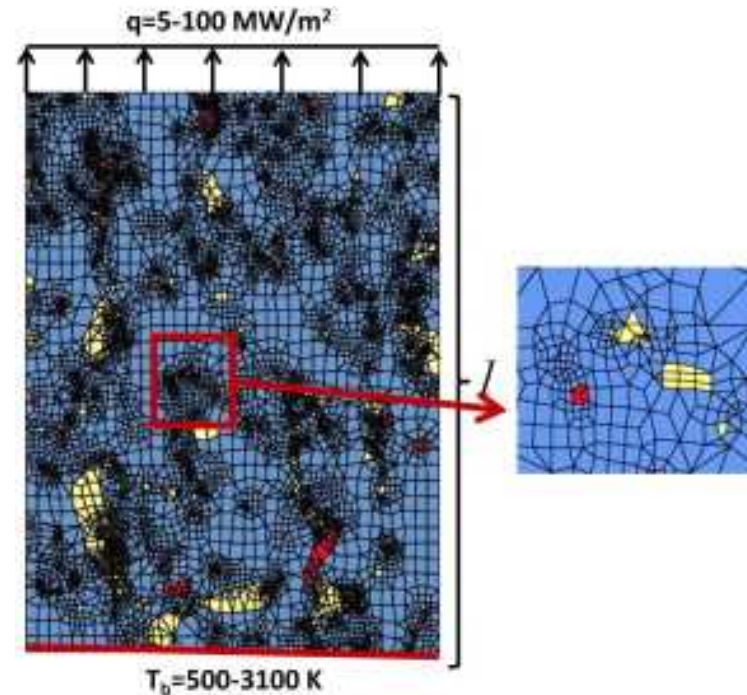


	Element per point	OOF2
$N_{\text{elements}}$	44274	18090
$N_{\text{nodes}}$	43836	27987

Significant reduction in computational power for OOF2 meshes

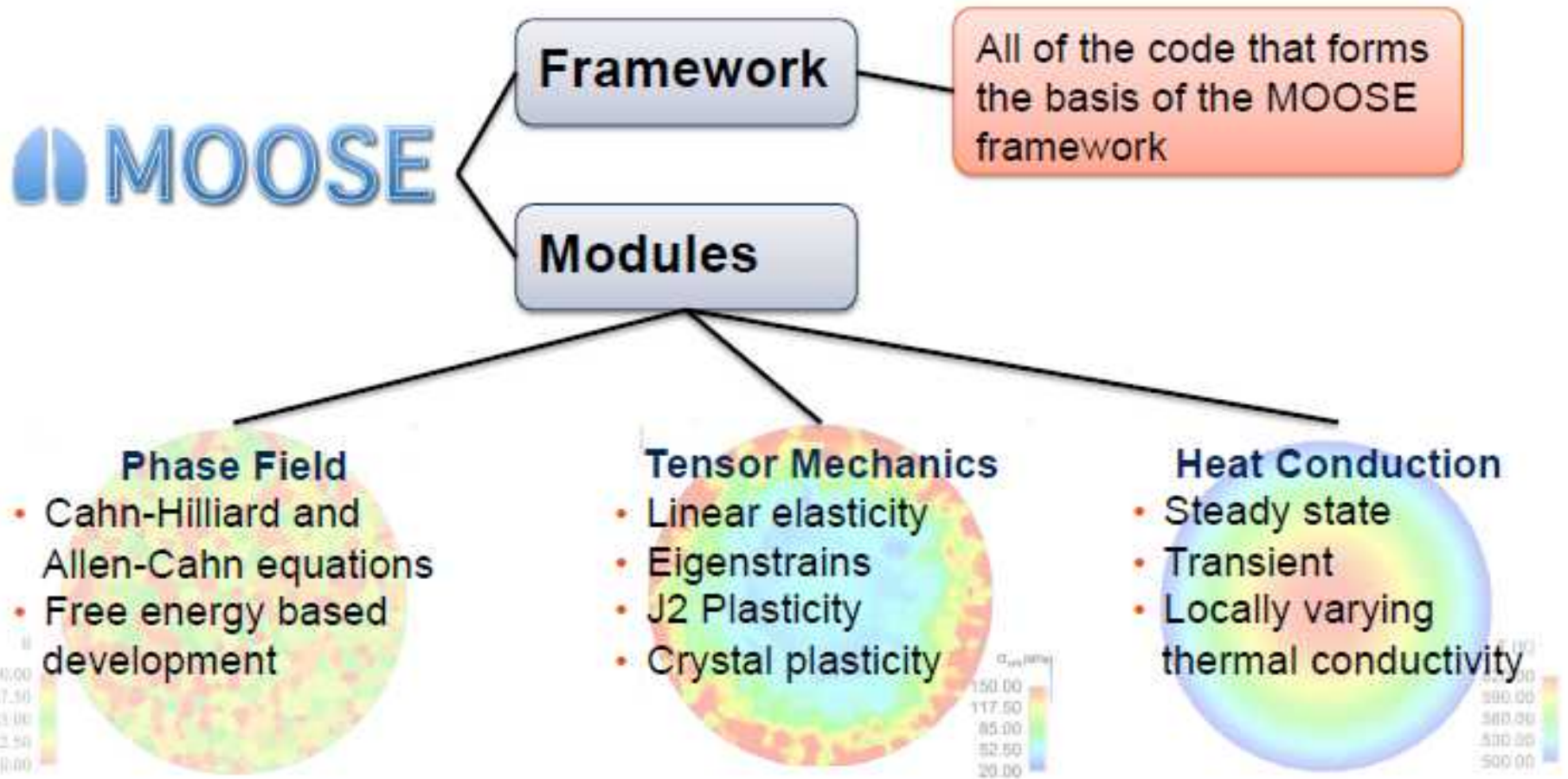
# Have your mesh now what?

- 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).



# Model Set up

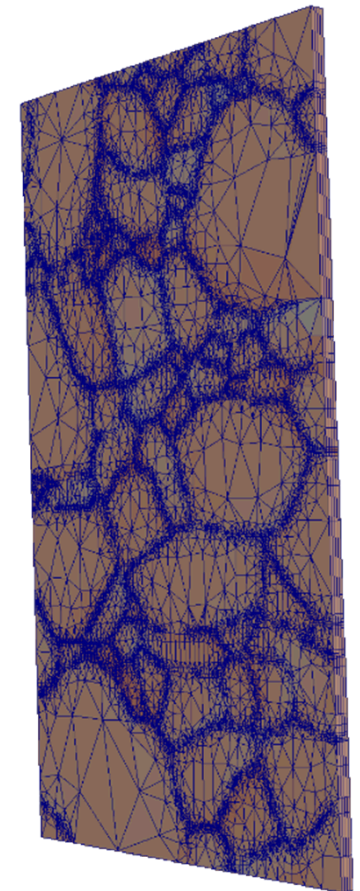
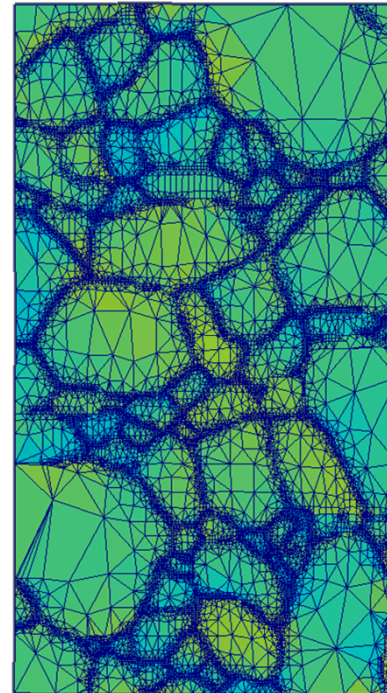
*Ran in 3 conditions*

*-2D Plane Strain-assumed zero strain in z direction*

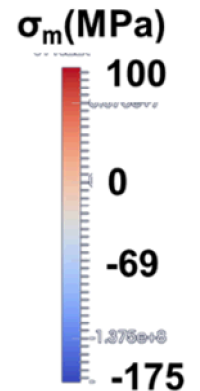
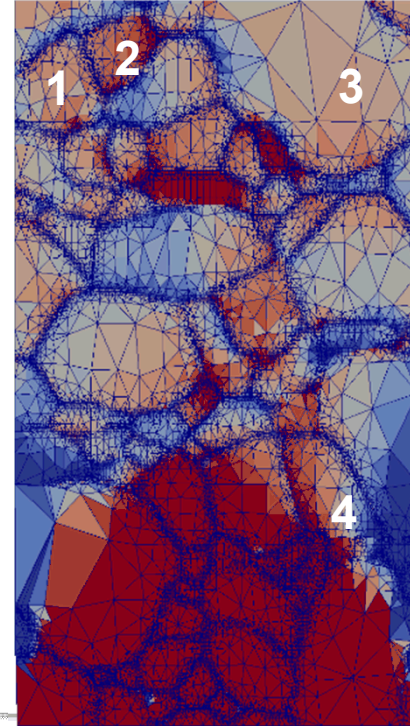
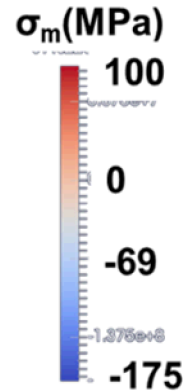
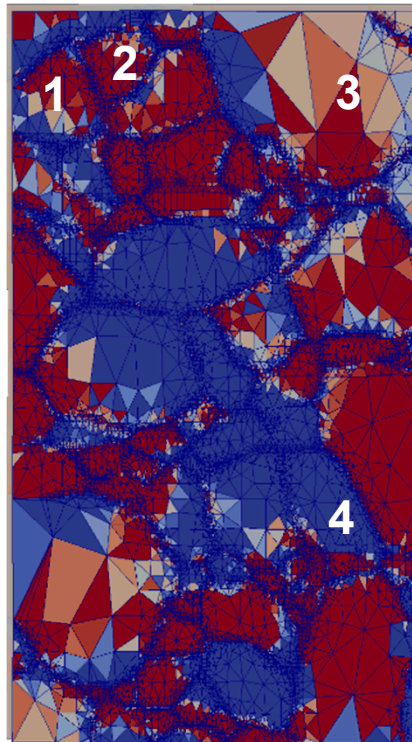
*-2D plane stress-assumes zero stress in z direction*

*-3D extruded mesh*

	Plane Strain	Plane Stress	3D Extruded
$\Delta T$	1500C	1500C	1500C
Constraint	None	Held in x and y on bottom edge	Back face held in z

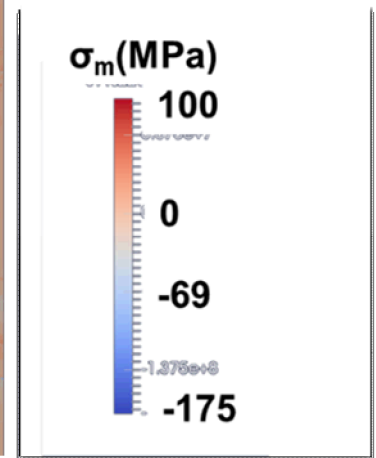
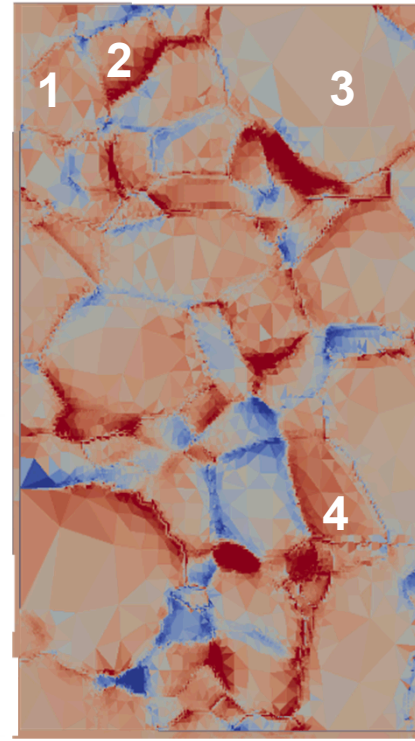
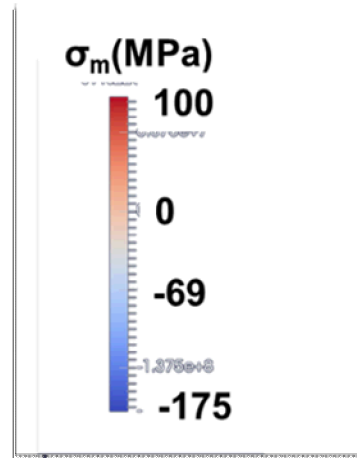
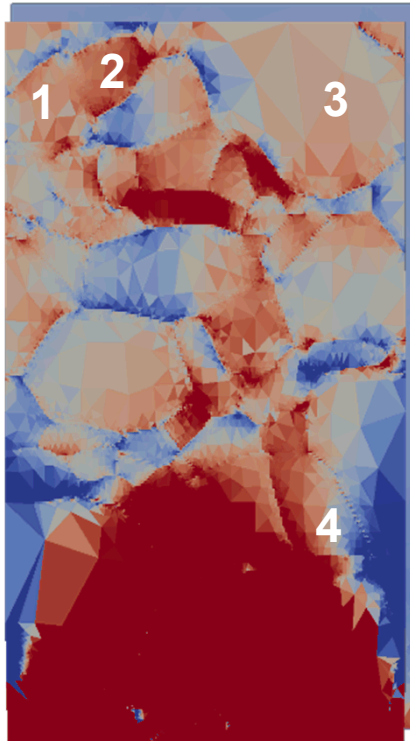


# Plane Strain vs Plane Stress



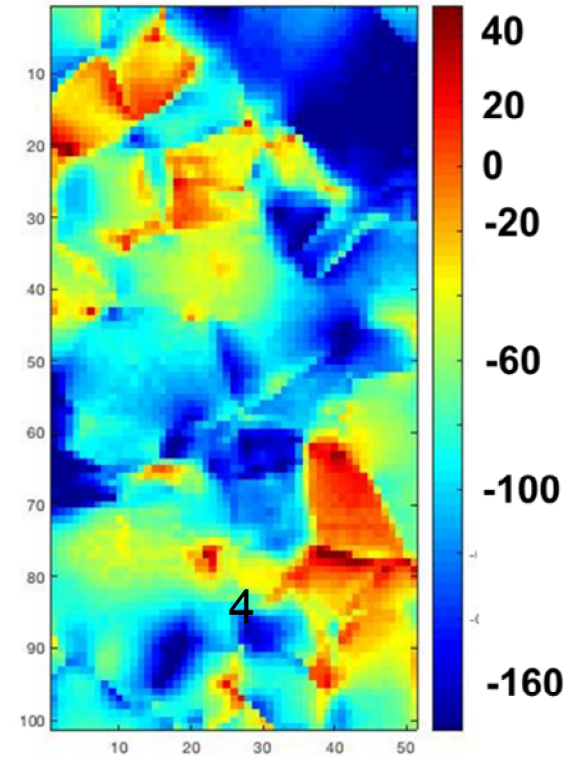
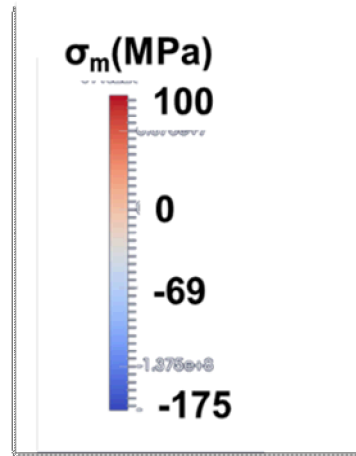
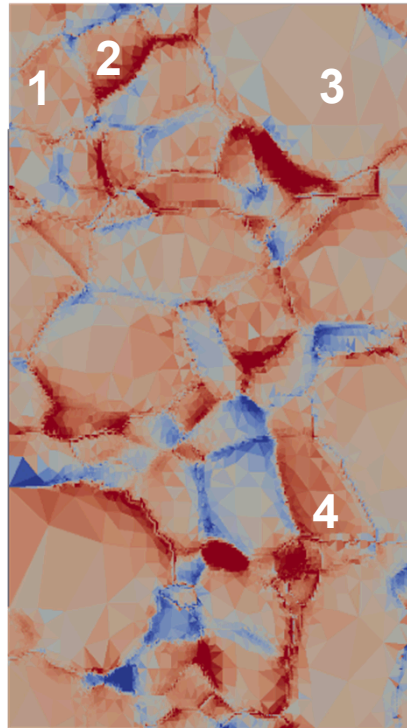
Grain ID	Plane Strain (MPa)	Plane Stress (MPa)
1	49	9.5
2	206	58
3	-52	-9.6
4	-633	-19.5

# Plane Stress vs 3D



Grain ID	Plane Stress (MPa)	3D (MPa)
1	9.5	5.2
2	58	35.8
3	-9.6	-7.5
4	-19.5	-29

# Comparison to Experiment

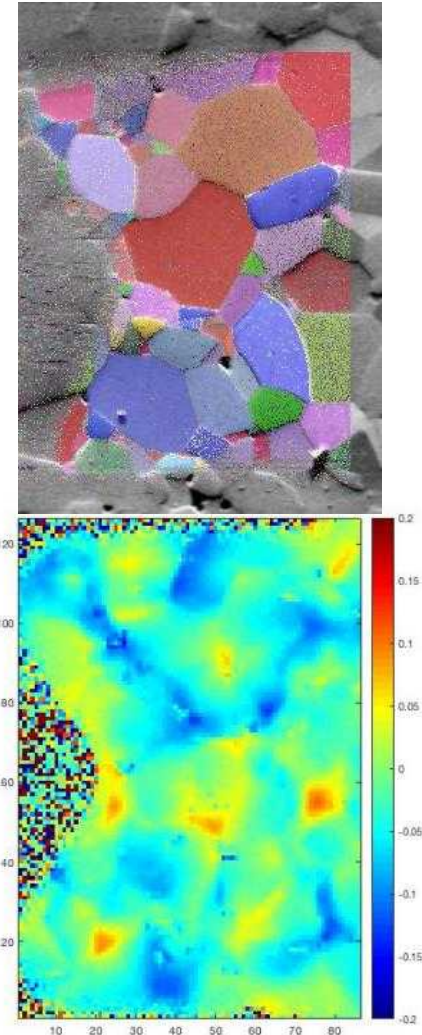


General Agreement-need to refine for resolution and 2D-3D reality

Grain	$\sigma_m$ (MPa)
1	5.2
2	35.8
3	-7.5
4	-29

# Conclusions

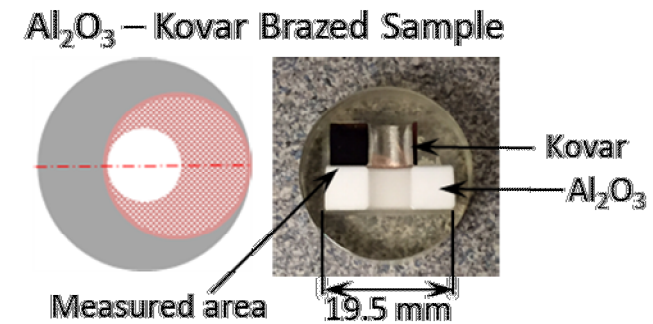
- PL-spectroscopy mapping is promising technique for enabling validation of stress at critical microstructural level
- Ease/speed of experiments enables large areas to be measured
- Microstructure meshing and modeling are primed to take advantage of this new data to enable mechanistic based models of brittle failure
- Plane stress provide better 2D approximation of residual stresses, than plain strain.
- Extruding mesh to 3D provides closest match to experimental values



EBSD and stress map of alumina

# Future Work

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



Brazed sample geometry

# Questions



# Converting Peak Shifts to Stress

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

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

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

$$\sigma_M = (\sigma_{11} + \sigma_{22} + \sigma_{33})/3 \quad \sigma_S = (2\sigma_{33} - \sigma_{11} - \sigma_{22})/3$$

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 <sup>-1</sup> /GPa)						
	Previous results			Our results			
	Uniaxial loading		Hydrostatic loading pressure coeff P <sup>1</sup>	Π <sub>11</sub>	Π <sub>22</sub>	Π <sub>33</sub>	Π <sub>11</sub> + Π <sub>22</sub> + Π <sub>33</sub>
Π <sub>11</sub>	Π <sub>33</sub>						
R1	3.0 <sup>2</sup>	1.8 <sup>‡</sup>	7.59 <sup>††</sup>	2.56	3.50	1.53	7.59
	3.2 <sup>§</sup>	1.4 <sup>§</sup>					
	2.7 <sup>¶</sup>	1.8 <sup>¶</sup>					
R2	2.8 <sup>‡</sup>	2.3 <sup>‡</sup>	7.615 <sup>††</sup>	2.65	2.80	2.16	7.61
	2.8 <sup>§</sup>	1.9 <sup>§</sup>					
	2.4 <sup>¶</sup>	2.2 <sup>¶</sup>					

<sup>1</sup>P = Π<sub>11</sub> + Π<sub>22</sub> + Π<sub>33</sub> <sup>2</sup>Reference 4, room temperature(?). <sup>3</sup>Reference 7, 77 K. <sup>4</sup>Reference 6, 77 K. <sup>5</sup>Reference 12, room temperature.