

ADVANCES IN HREBSD MEASUREMENT AND ITS IMPACT ON HIGH FIDELITY CRYSTAL PLASTICITY MODEL DEVELOPMENT

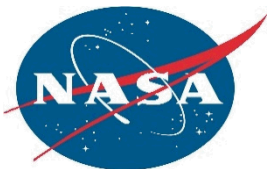
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INITIALISMS

- ◆ Electron backscatter diffraction – EBSD
- ◆ Scanning electron microscope – SEM
- ◆ Transmission electron microscope – TEM
- ◆ High (angular) resolution EBSD – HREBSD
- ◆ X-ray diffraction – XRD
- ◆ Digital image correlation – DIC
- ◆ Electron channeling contrast imaging – ECCI
- ◆ Geometrically necessary dislocation – GND
- ◆ Transmission Kikuchi diffraction – TKD
- ◆ High energy X-ray diffraction microscopy – HEDM
- ◆ Pattern center – PC
- ◆ Inverse compositional Gauss-Newton - ICGN



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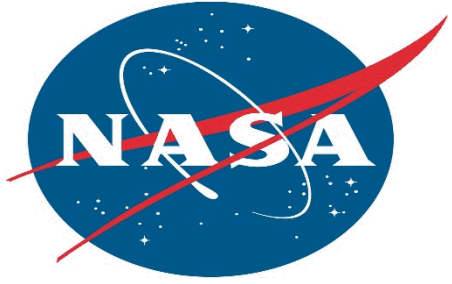
David Fullwood



OUTLINE

- ◆ Introduction
 - ◆ Motivation – Microstructural modeling
 - ◆ Background to HREBSD
 - ◆ Combining with DIC (Microstamping)
- ◆ Mitigating error
 - ◆ Pattern Center Error
 - ◆ Shift uncertainty
 - ◆ Cross-correlation error
- ◆ Accommodating Error
 - ◆ Inverse material point simulation
 - ◆ Non-deterministic model calibration
- ◆ Cross-validation studies
 - ◆ X-ray
 - ◆ ECCI
 - ◆ TEM
- ◆ Conclusions and future work



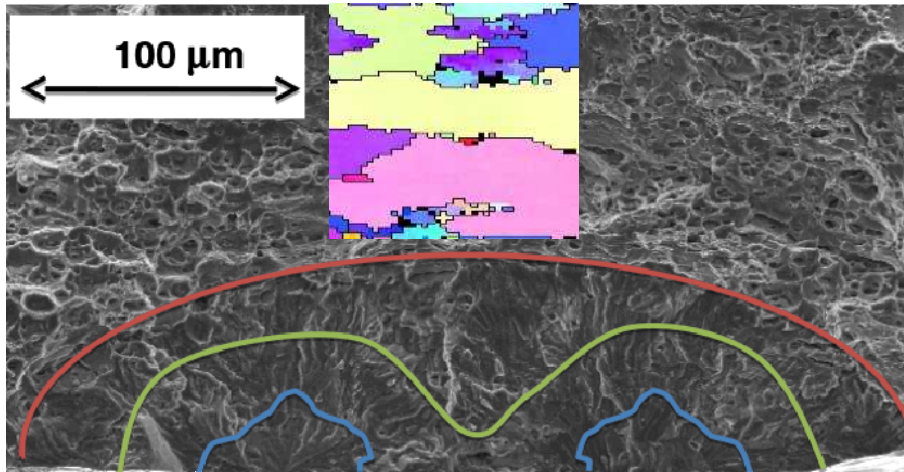


INTRODUCTION

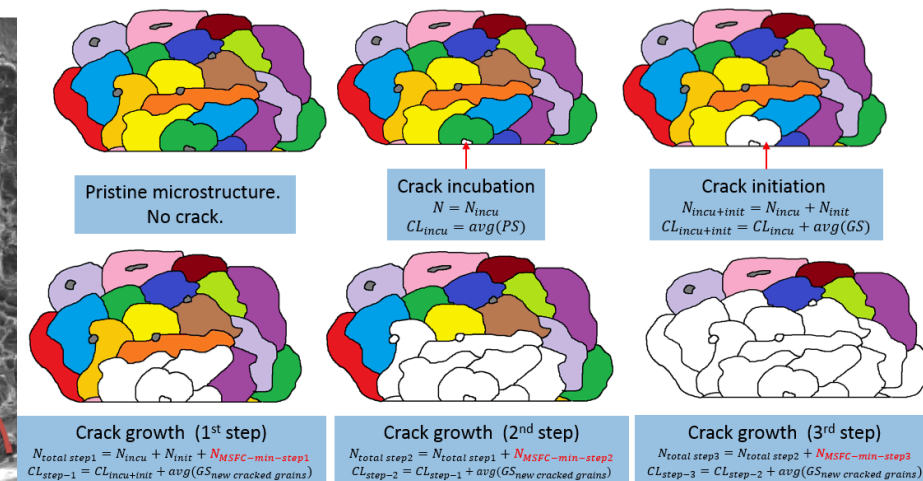
MOTIVATION

- ◆ Microstructurally small fatigue cracks
- ◆ Majority of component life can be spent here
- ◆ Need accurate models to understand/characterize uncertainty in life
- ◆ The key to higher fidelity is local characterization, i.e. microscopy as data

Evolution of a Crack Front

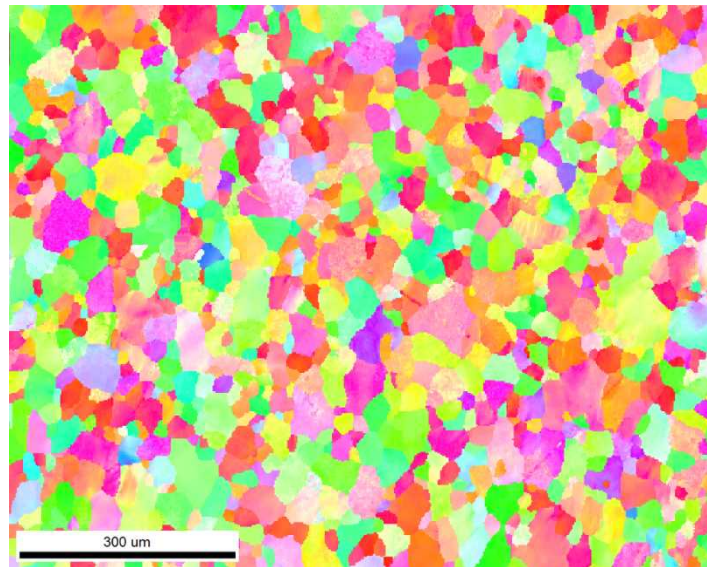
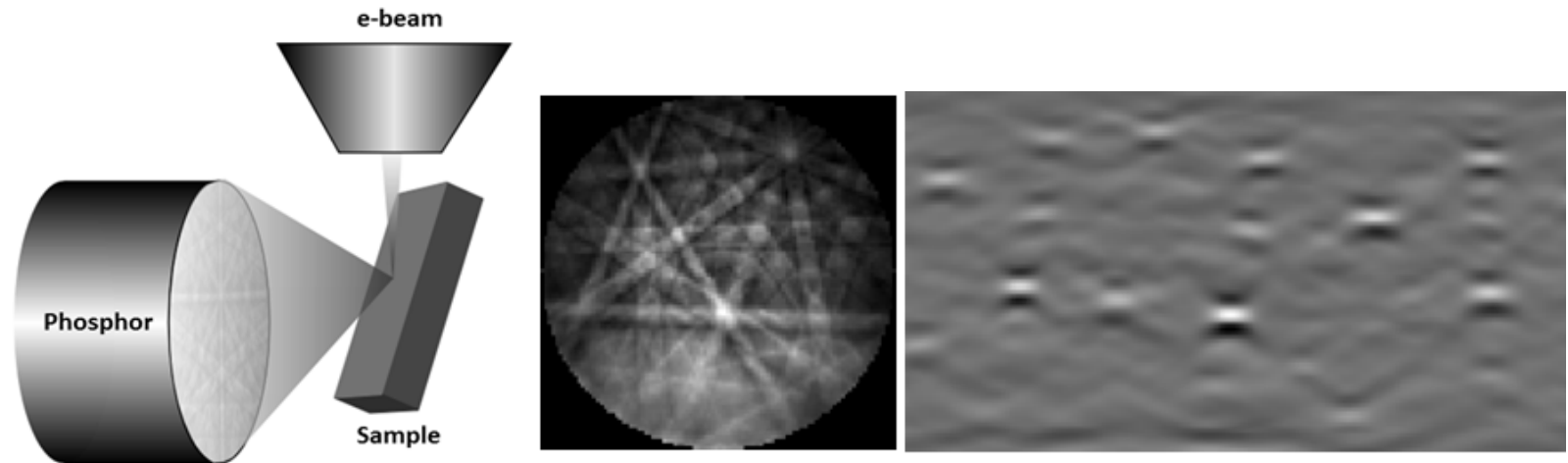


Small Crack Growth Model

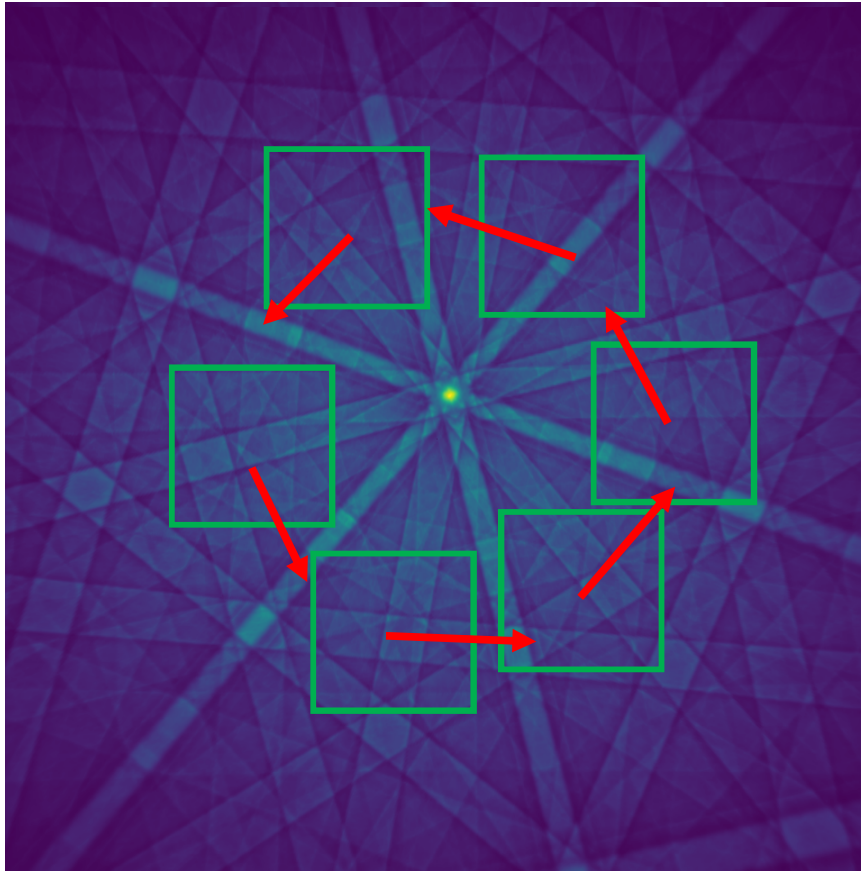


EBSD

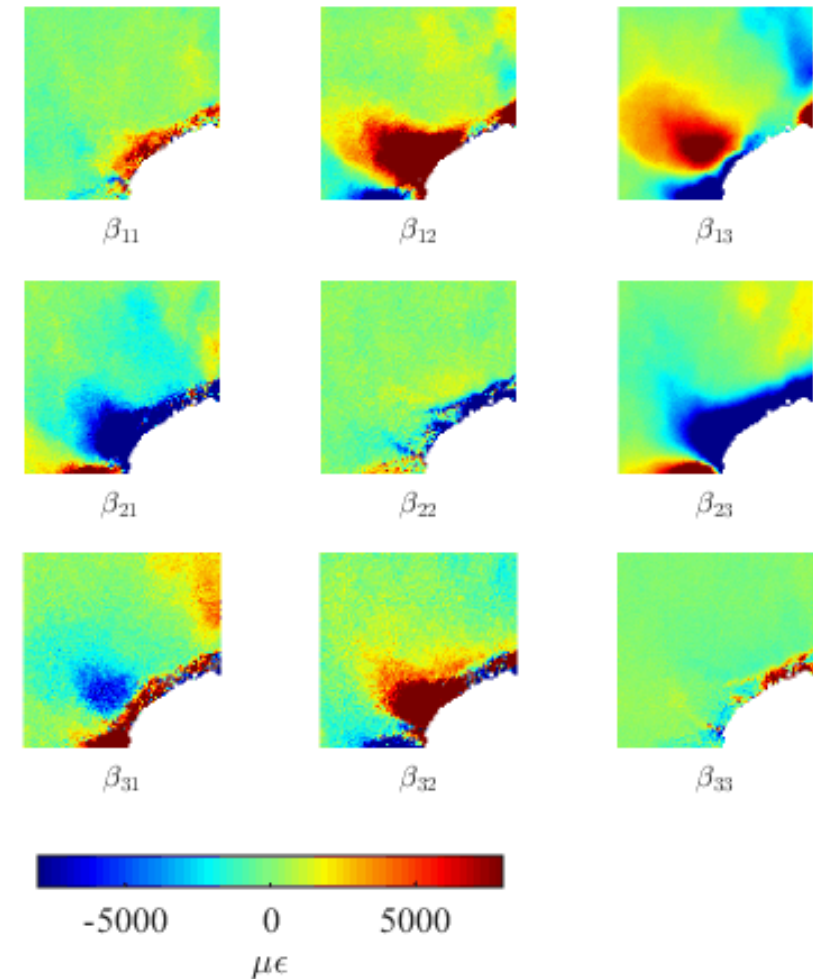
- ◆ SEM based technique
- ◆ Dynamical diffraction
- ◆ Interaction volume depth on the order of nanometers
- ◆ Indexed automatically via the Hough transform (0.1-0.5 degree accuracy)
- ◆ Commercial development of detectors has focused on speed, but has recently pivoted back to resolution and noise reduction



HIGH RESOLUTION EBSD (HREBSD) – STRESS AND STRAIN



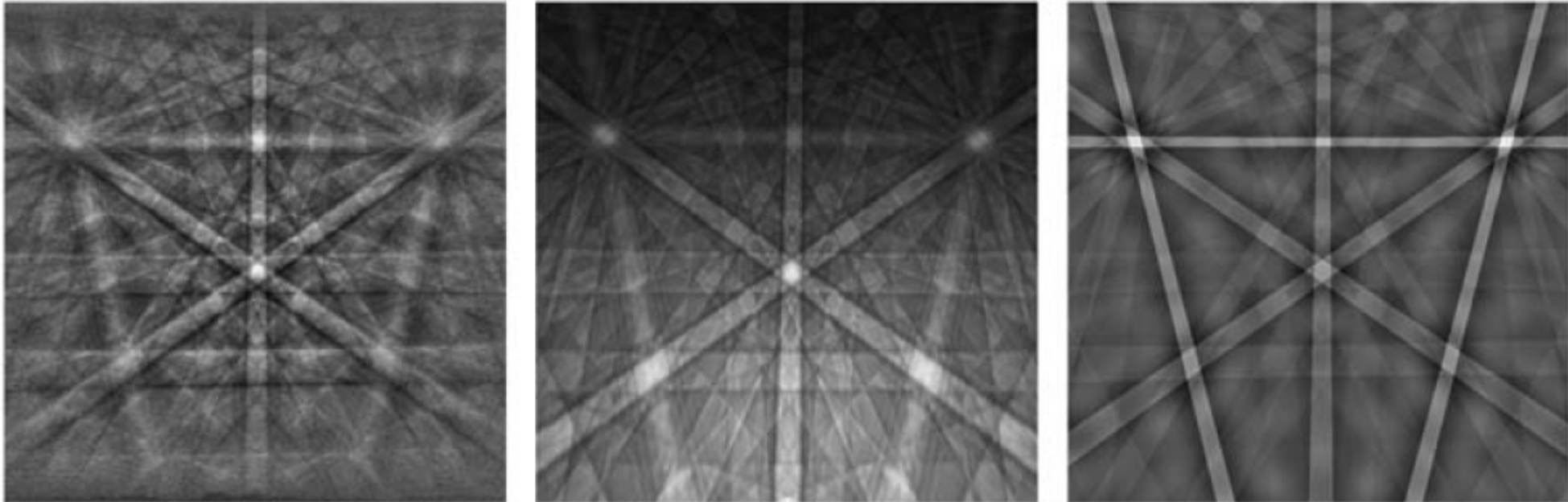
Lattice Distortion



◆ Output: Relative elastic distortion
(relative stresses with assumed elasticity parameters)

SIMULATED PATTERNS

- ◆ Absolute stresses and strains are recoverable when strain free, simulated patterns are used
- ◆ Use of simulated patterns is extremely sensitive to pattern center error.
- ◆ Simulated pattern HREBSD is easily iterated.



Jackson, B., Christensen, J., Singh, S., De Graef, M., Fullwood, D., Homer, E., & Wagoner, R. (2016). Performance of Dynamically Simulated Reference Patterns for Cross-Correlation Electron Backscatter Diffraction. *Microscopy and Microanalysis*, 22(4), 789-802. doi:10.1017/S143192761601148X

HREBSD – GND DENSITY

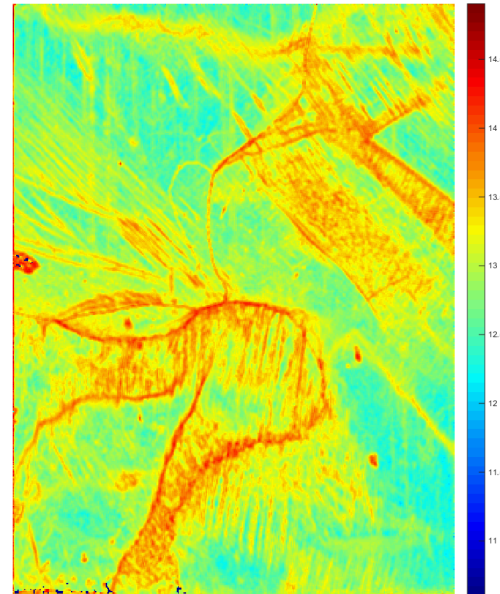
The fundamental relation of continuum dislocation theory may be coupled with the definition of Nye's tensor to measure dislocation densities on individual slip systems.

$$\min. f = \sum_{t=1}^N |w^t \rho^t| \quad \text{s.t.} \quad \alpha_{ij} = \sum_{t=1}^N \rho^t b_i^t v_j^t \quad \text{where} \quad \alpha^s = \nabla^s \times \mathbf{F}^s$$

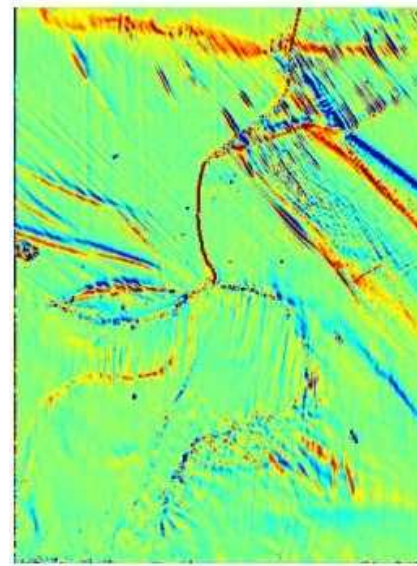
Orientation from EBSD



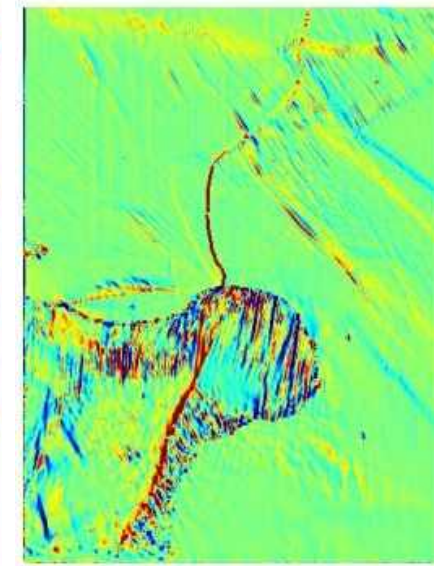
Total Dislocation Density



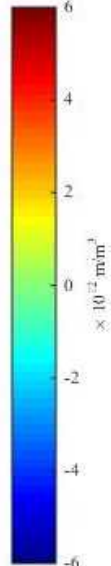
Individual Dislocation Densities



(111)[011]

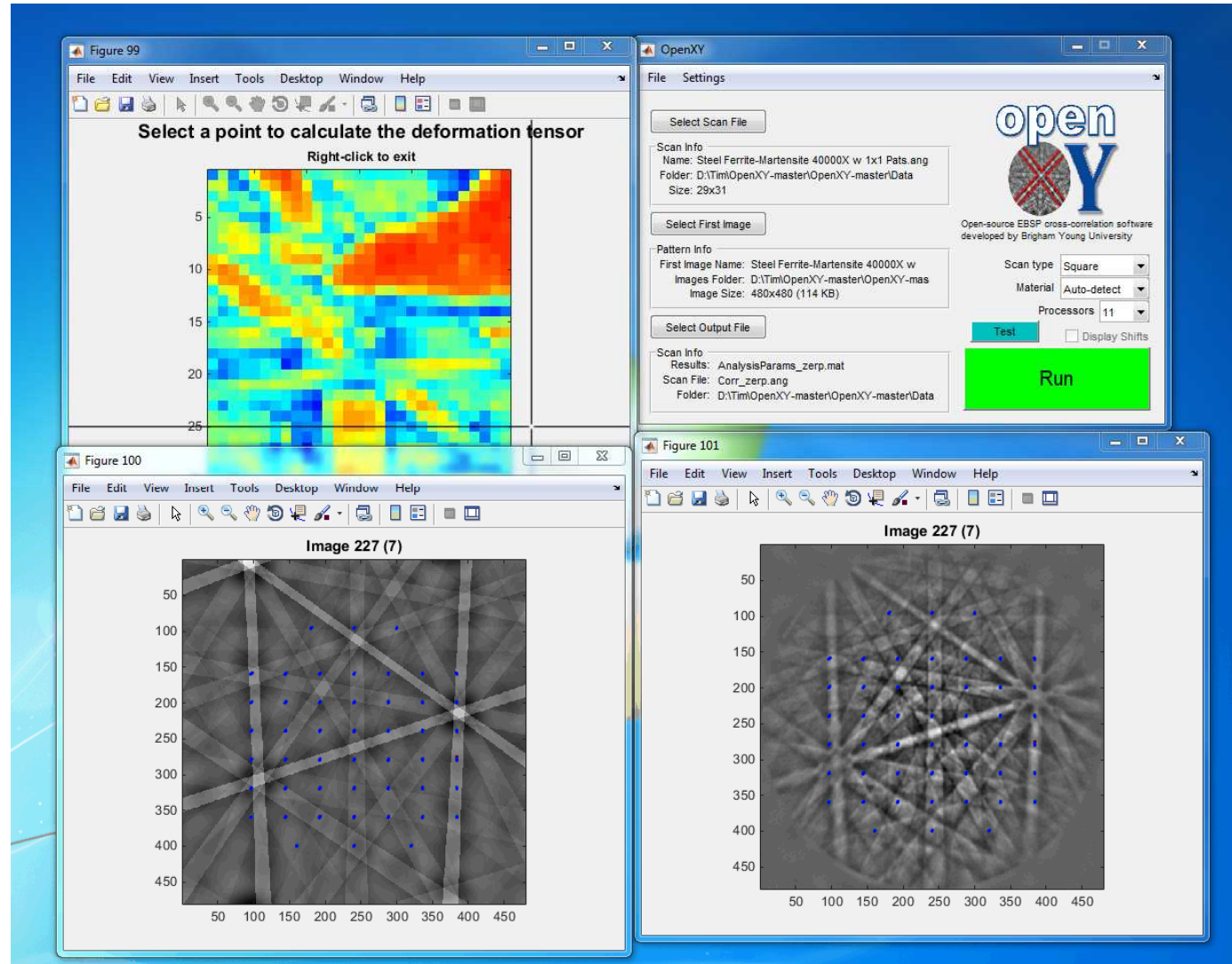


(111)[110]



◆ Output: GND Density

OPENXY



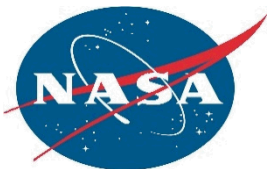
PROS AND CONS COMPARED TO OTHER DIFFRACTION TECHNIQUES

Pros

- ◆ Better spatial resolution than X-ray/HEDM
- ◆ Simple sample preparation compared to TEM
- ◆ Examines bulk material
- ◆ Simple, inexpensive, widely available
- ◆ Rapidly improving technology

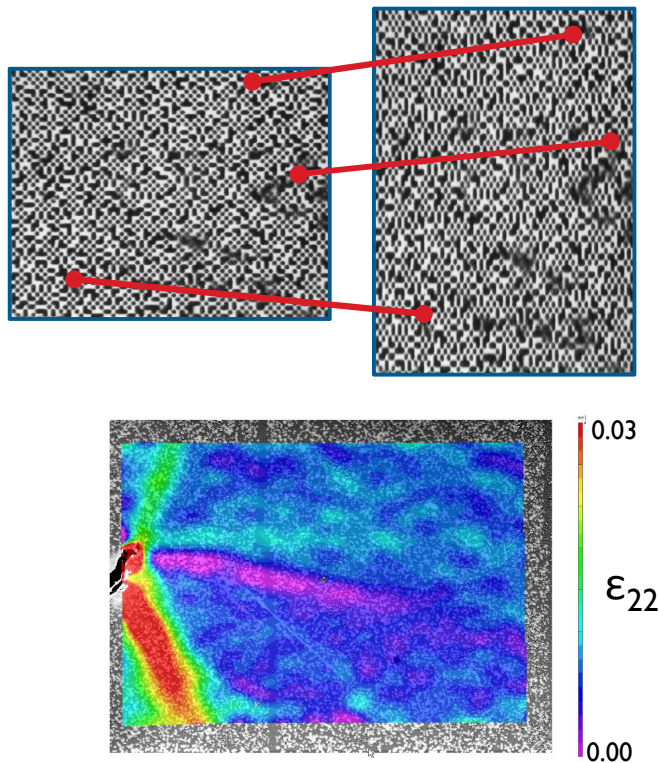
Cons

- ◆ Surface technique, instead of 3D like X-ray
- ◆ Worse resolution than TEM or ECCI
- ◆ Polishing makes it a somewhat destructive technique



DIGITAL IMAGE CORRELATION (DIC) IN THE SEM

Cross correlation



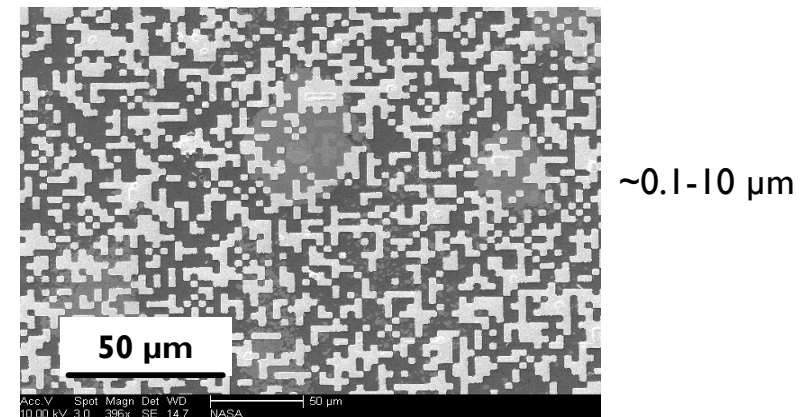
◆ Output: Total deformation

(in plane for 2D)

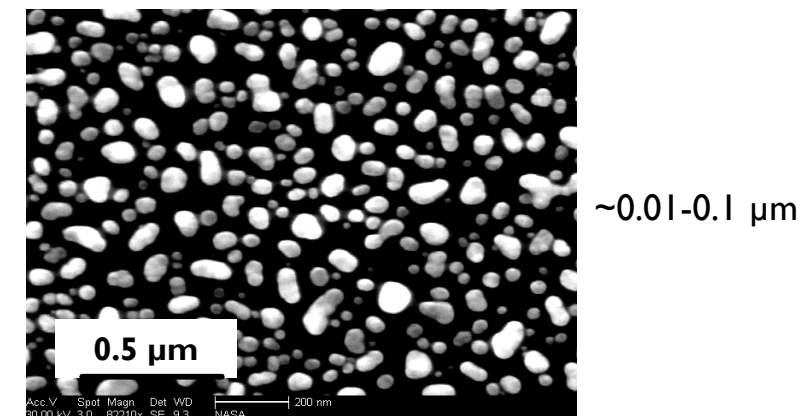
DIC performed with VIC2D

Conventional patterning methods for micro-DIC

E-beam lithography



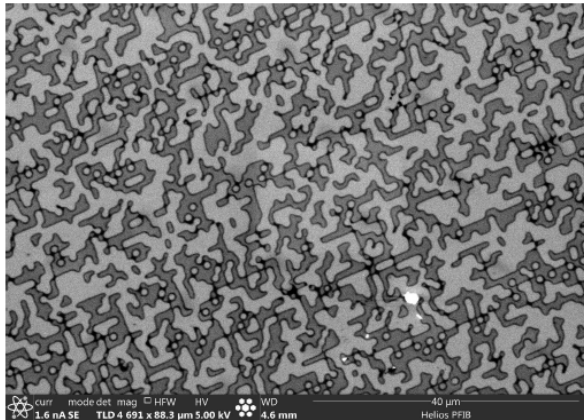
Nanoparticles



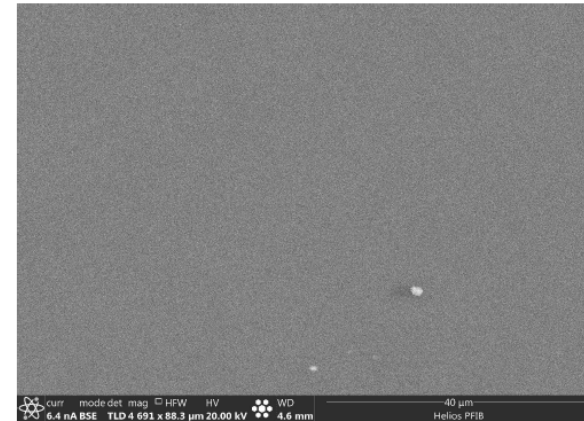
COMBINED DIC AND HREBSD

- ◆ Traditional surface preparation makes DIC and HREBSD mutually exclusive
- ◆ Microstamped urethane polymer patterns are selectively electron-transparent

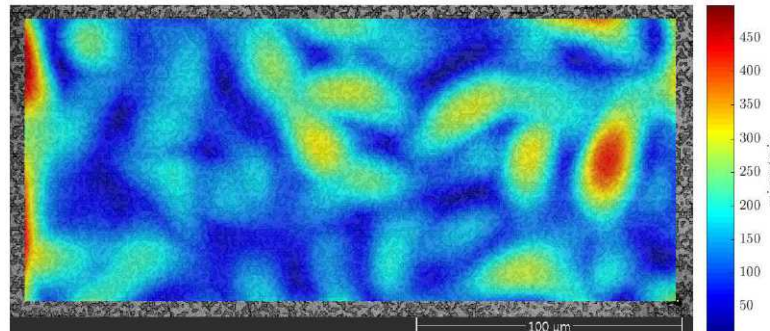
5kV SE



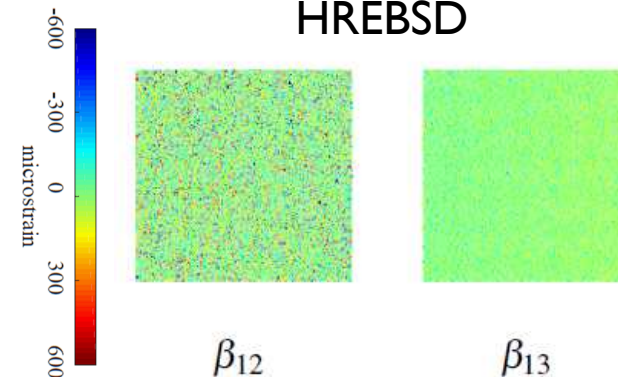
20kV BSE



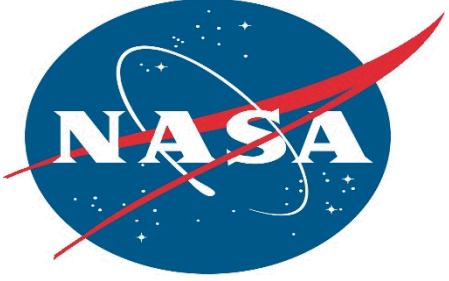
DIC



HREBSD

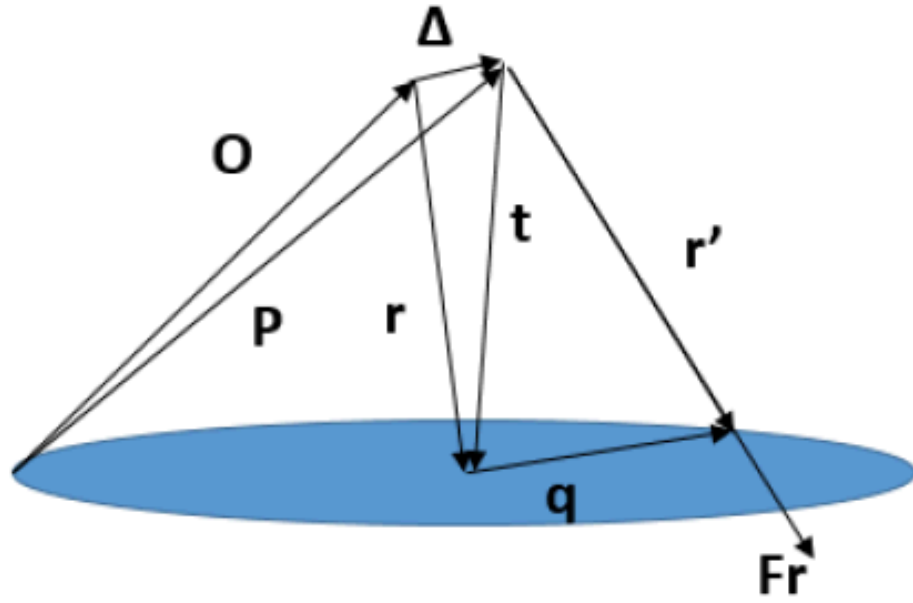


Ruggles, T. J., G. F. Bomarito, A. H. Cannon, and J. D. Hochhalter (2017). Selectively Electron-Transparent Microstamping Toward Concurrent Digital Image Correlation and High-Angular Resolution Electron Backscatter Diffraction (EBSD) Analysis. *Microscopy and Microanalysis* **23**(6), 1091–1095.



MITIGATING HREBSD MEASUREMENT ERROR

PATTERN CENTER ERROR

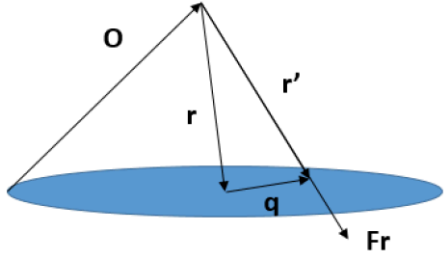


$$\beta_{phantom} = \frac{1}{O_3 + 2\Delta_3/3} \begin{bmatrix} \Delta_3/3 & 0 & -\Delta_1 \\ 0 & \Delta_3/3 & -\Delta_2 \\ 0 & 0 & -2\Delta_3/3 \end{bmatrix}$$

- ❑ Pattern center calibration techniques are necessary to reduce this error.
- ❑ The simplest methods involve strain minimization.
- ❑ These methods require a strain free calibration point (heavily annealed or epitaxial material FIB-welded to the sample surface)

PATTERN CENTER CALIBRATION

HREBSD



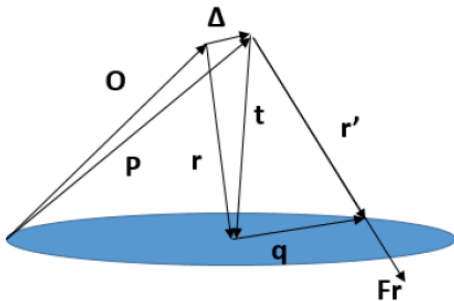
$$\vec{q} = \mathbf{F}\vec{r} \frac{-\vec{O} \cdot \hat{z}}{\mathbf{F}\vec{r} \cdot \hat{z}} - \vec{r}$$

8 degrees of freedom,
9 unknowns



Add in traction free OR
deviatoric constraint

PC calibration



$$\vec{q} = \vec{\Delta} + \mathbf{F}\vec{r} \frac{-(\vec{O} + \vec{\Delta}) \cdot \hat{z}}{\mathbf{F}\vec{r} \cdot \hat{z}} - \vec{r}$$

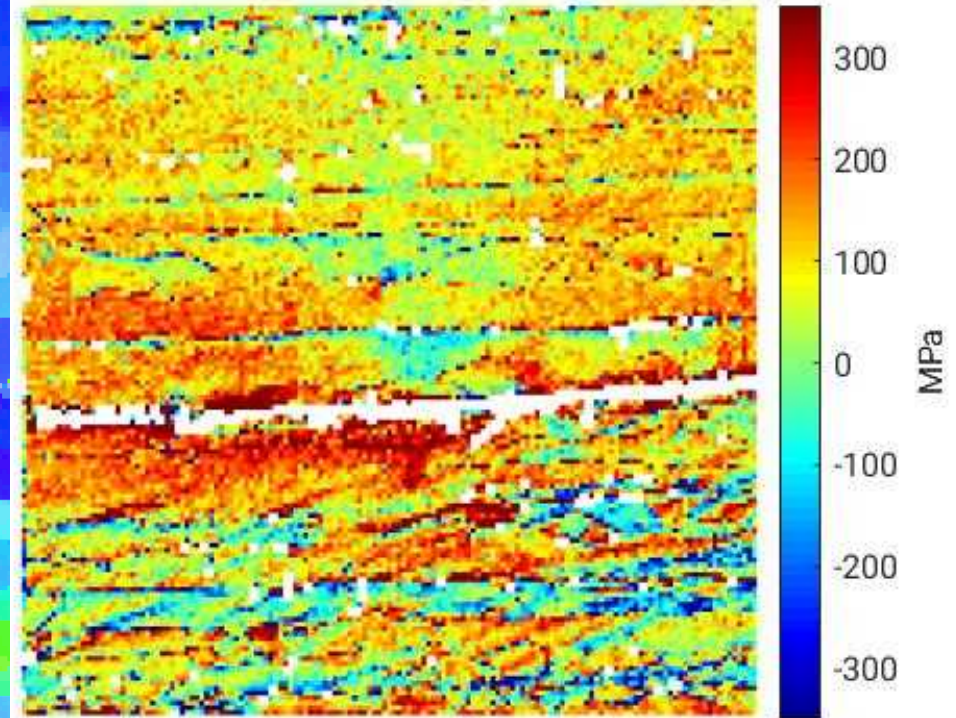
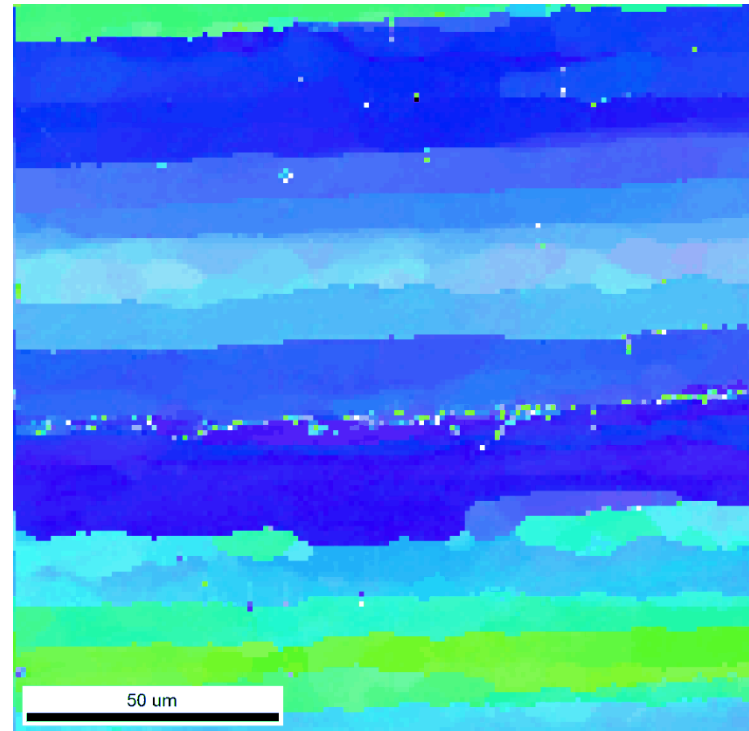
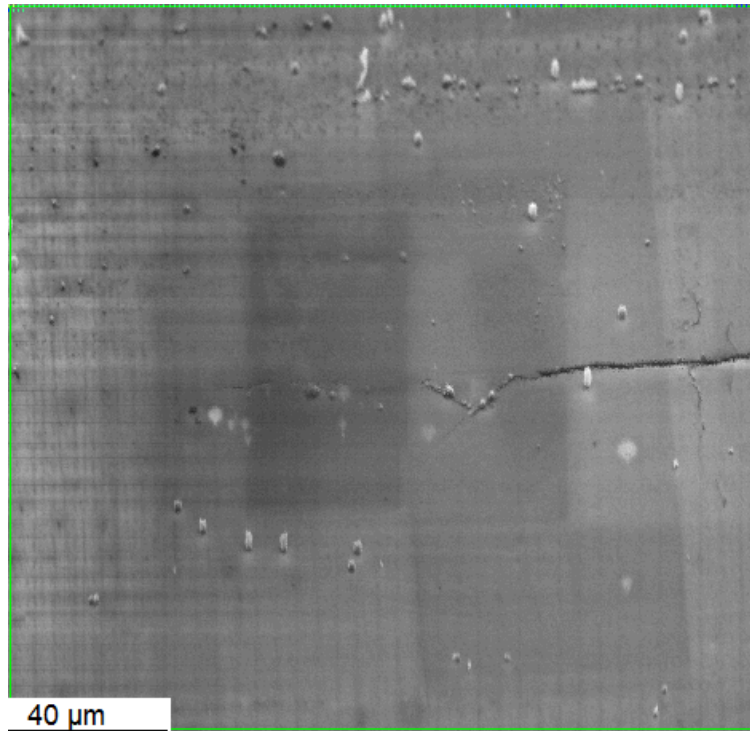
8 degrees of freedom,
12 unknowns



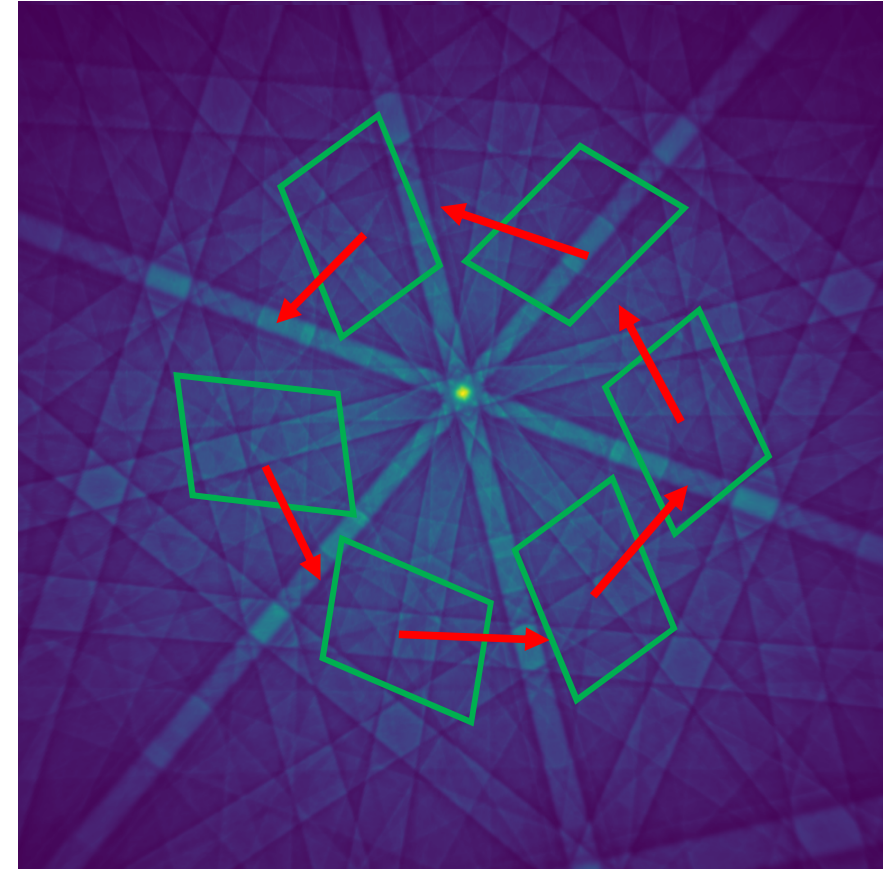
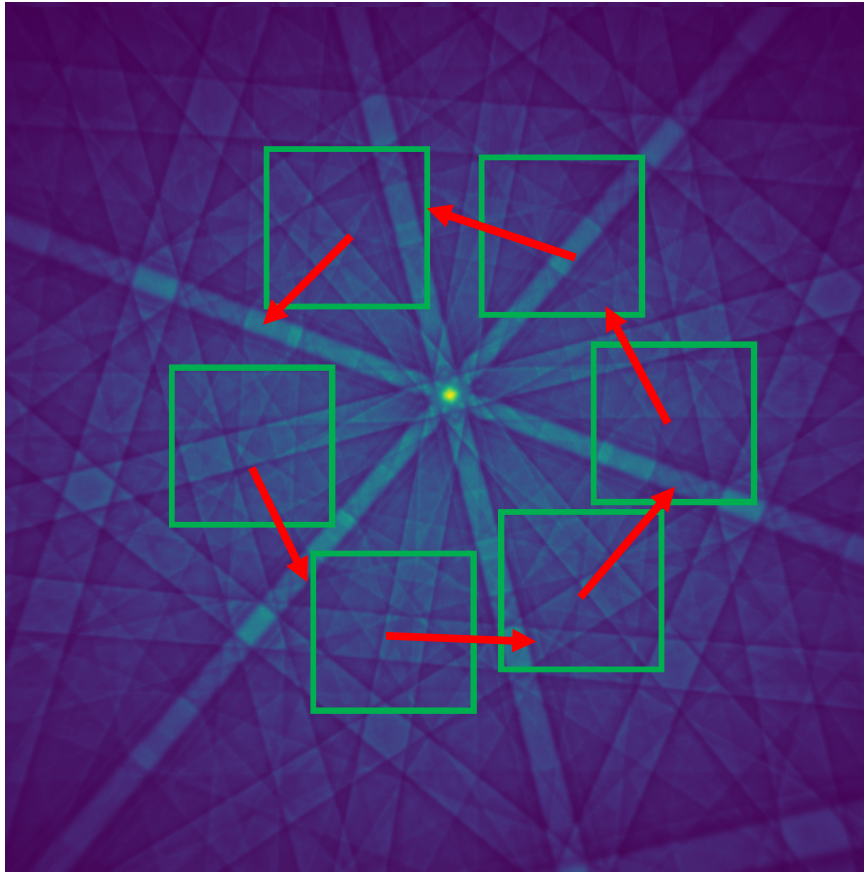
Add in traction free AND
deviatoric constraint

PC calibration and HREBSD are performed simultaneously

PATTERN CENTER CALIBRATION RESULTS



CROSS-CORRELATION ERROR



XASGO – INVERSE COMPOSITIONAL GAUSS NEWTON HREBSD

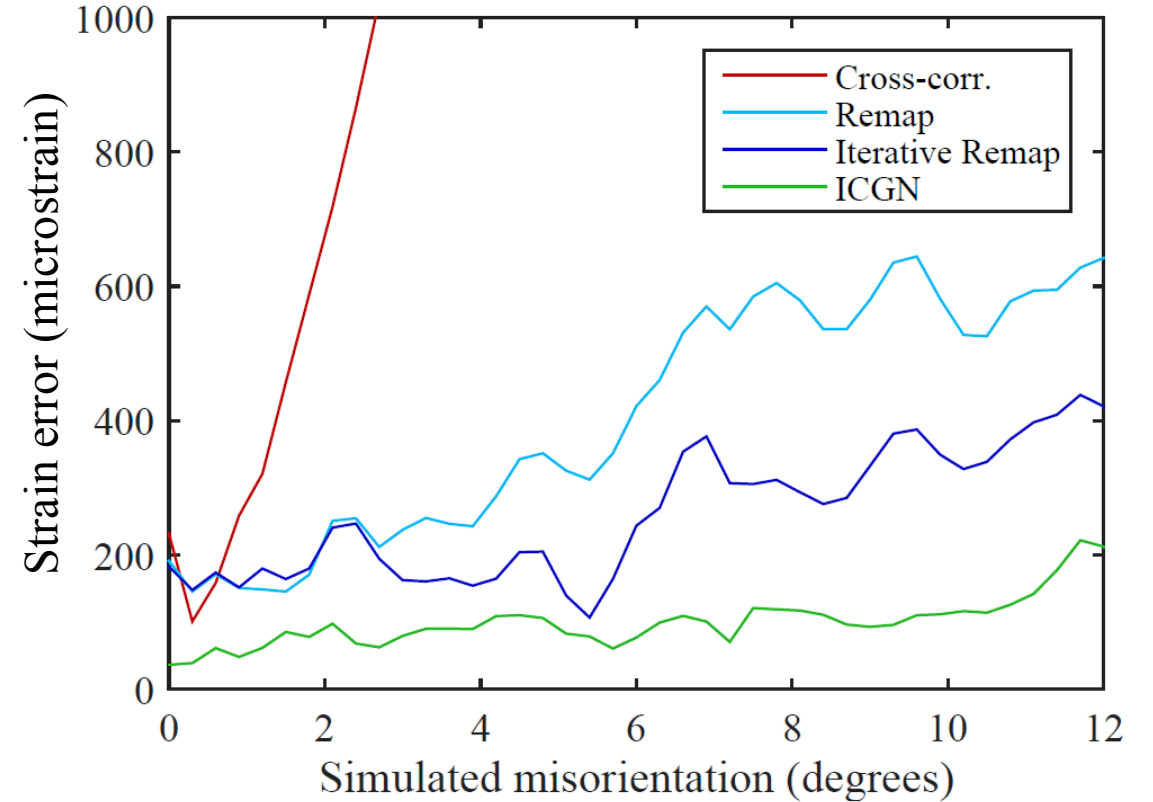
$$C_{LS} = \sum_{i=1}^N \left[\frac{f^{(i)} - f_m}{\sqrt{\sum_{i=1}^N [f^{(i)} - f_m]^2}} - \frac{g^{(i)} - g_m}{\sqrt{\sum_{i=1}^N [g^{(i)} - g_m]^2}} \right]^2$$

$$\nabla C_{LS} = \frac{dC_{LS}}{d\Delta\mathbf{F}} \Big|_{\Delta\mathbf{F}=\mathbf{I}} \approx \frac{2}{\sqrt{\sum [f^{(i)} - f_m]^2}} \sum_{i=1}^N \left[\left[\frac{f^{(i)} - f_m}{\sqrt{\sum [f^{(i)} - f_m]^2}} - \frac{g^{(i)} - g_m}{\sqrt{\sum [g^{(i)} - g_m]^2}} \right] \left[\frac{df^{(i)}}{d\Delta\mathbf{F}} \right] \right] \Big|_{\Delta\mathbf{F}=\mathbf{I}}$$

$$\nabla\nabla C_{LS} = \frac{d^2 C_{LS}}{d\Delta\mathbf{F}^2} \Big|_{\Delta\mathbf{F}=\mathbf{I}} \approx \frac{2}{\sqrt{\sum [f^{(i)} - f_m]^2}} \left[\frac{df^{(i)}}{d\Delta\mathbf{F}} \right] \otimes \left[\frac{df^{(i)}}{d\Delta\mathbf{F}} \right] \Big|_{\Delta\mathbf{F}=\mathbf{I}}$$

$$\vec{w}(\vec{x}^{(i)}, \mathbf{F}, \vec{P}, \Delta\vec{P}) = \vec{x}^{(i)} + \vec{q}^{(i)} = \Delta\vec{P} + \mathbf{F}(\vec{x}^{(i)} - \vec{P}) \frac{-(\vec{P} + \Delta\vec{P}) \cdot \hat{z}}{\mathbf{F}(\vec{x}^{(i)} - \vec{P}) \cdot \hat{z}} + \vec{P}$$

$$\frac{df^{(i)}}{d\Delta\mathbf{F}} = \frac{\partial f^{(i)}}{\partial w_1} \frac{dw_1}{d\Delta\mathbf{F}} + \frac{\partial f^{(i)}}{\partial w_2} \frac{dw_2}{d\Delta\mathbf{F}}$$

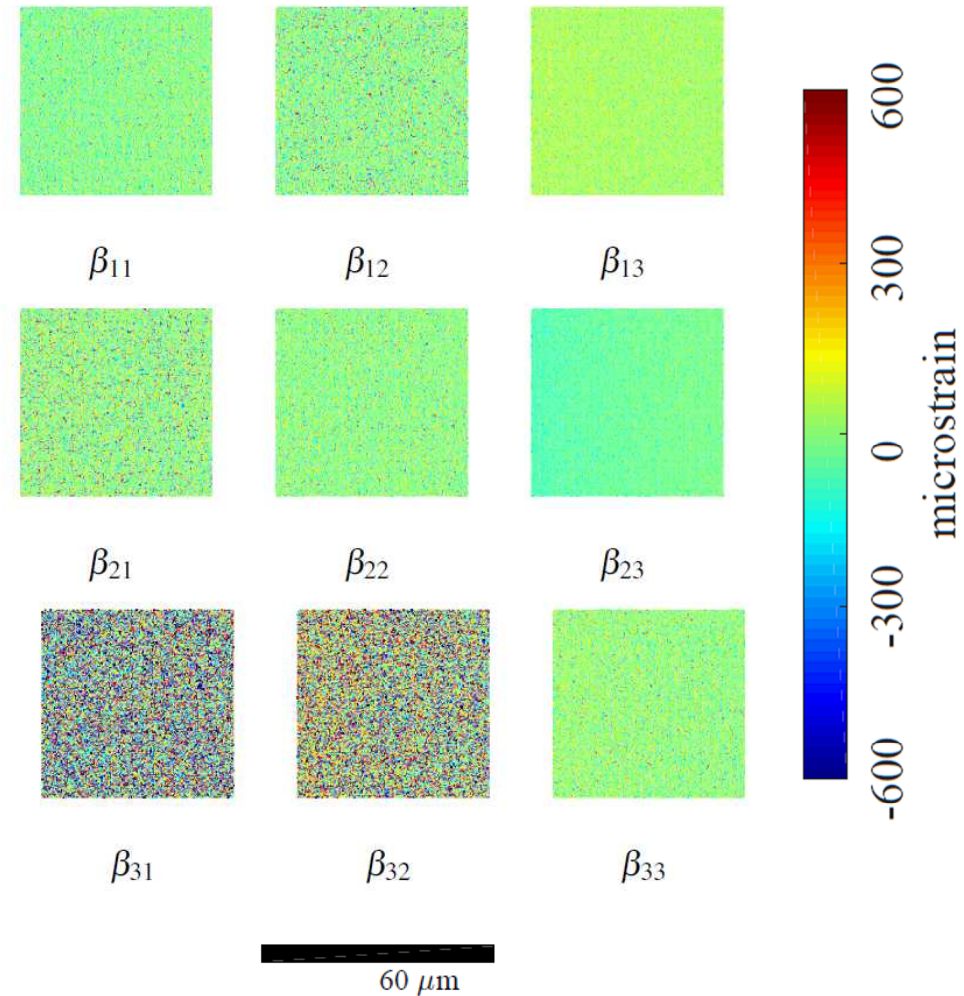


Ruggles, T., G. Bomarito, R. Qiu, and J. Hochhalter (2018). New levels of high angular resolution EBSD performance via inverse compositional Gauss-Newton based digital image correlation. *Ultra-microscopy*.

WHITE NOISE IN HREBSD

- ◆ Noise in HREBSD measurements is determined by:
 - ◆ Pattern quality
 - ◆ Pattern resolution
 - ◆ Diffraction geometry
- ◆ Noise of each term (in the detector reference frame) of the lattice distortion is not equal.

Erroneous strain in Si



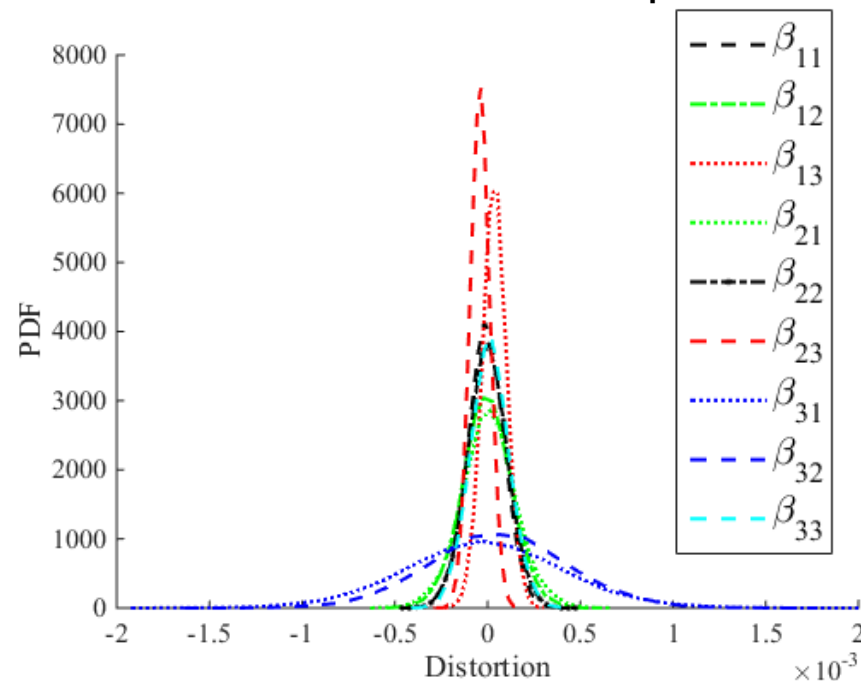
RELATIVE NOISE IN EACH DISTORTION TERM

- ◆ A simple Monte Carlo simulation was used to determine the relationship between the standard deviation of the noise in each term and the standard deviation of the noise in the measured shifts. The relevant variables were distance of the ROIs from the pattern center (r), detector distance (d), number of ROIs (N), and σ_q , the standard deviation of the noise in the shifts:

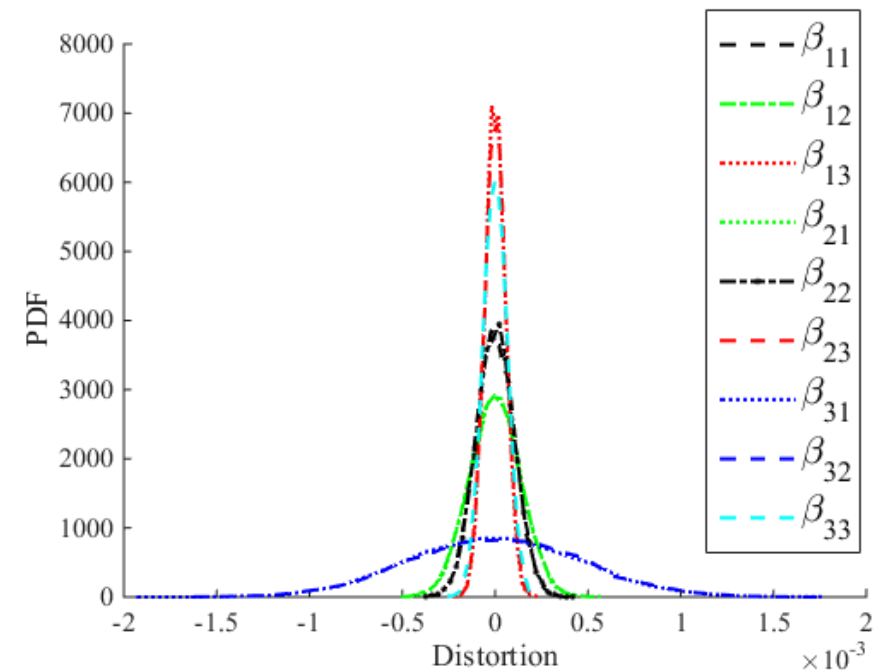
$$\sigma(\beta_{13}), \sigma(\beta_{23}) \propto \frac{1}{d\sqrt{N}} \sigma_q \quad \sigma(\beta_{31}), \sigma(\beta_{32}) \propto \frac{d}{r^2\sqrt{N}} \sigma_q \quad \sigma(\beta_{i=j}), \sigma(\beta_{12}), \sigma(\beta_{21}) \propto \frac{1}{r\sqrt{N}} \sigma_q$$

- ◆ Standard deviation of the shifts was about 1/13th of a pixel

Si before stamp



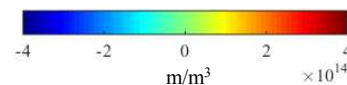
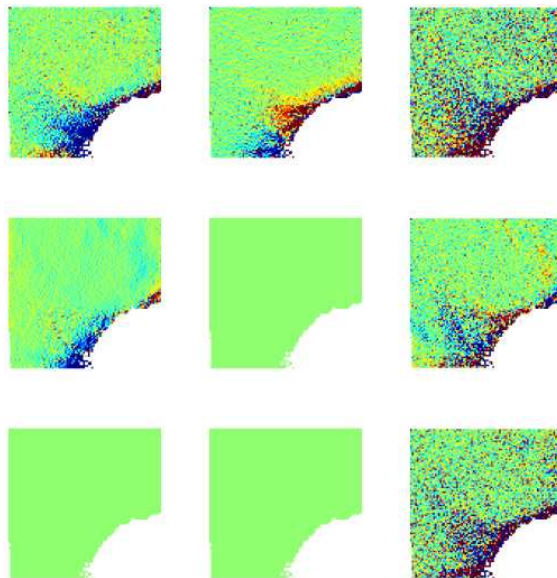
Monte Carlo using random noise



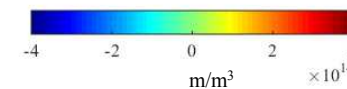
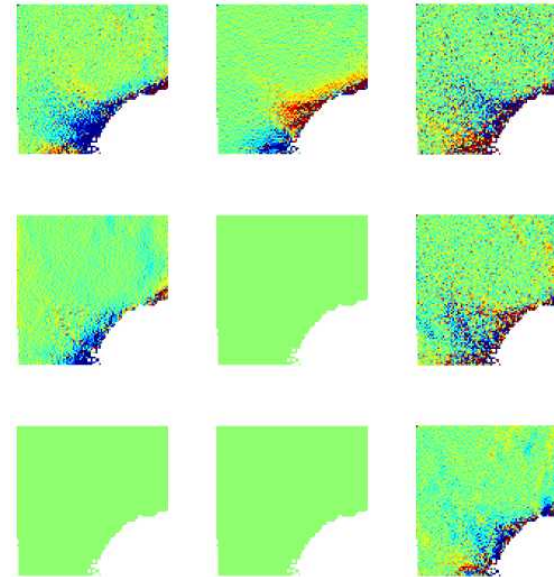
REDUCING NOISE IN GND MEASUREMENTS

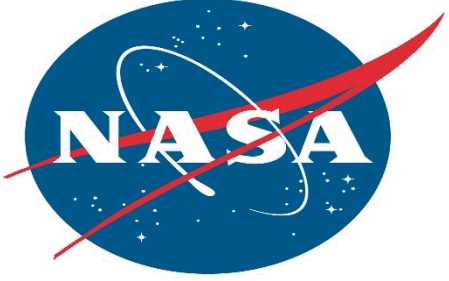
- ◆ While elastic strains measured via HREBSD are essential to measure stress, they represent a small fraction of the distortion gradient used to calculate GND density.
- ◆ Strains are typically ignored to get three more degrees of freedom from the Nye tensor when calculated with derivatives in only two orthogonal directions
- ◆ By imposing antisymmetry in the phosphor frame, the most problematic “noise” terms may be eliminated.

Known Nye Tensor Components



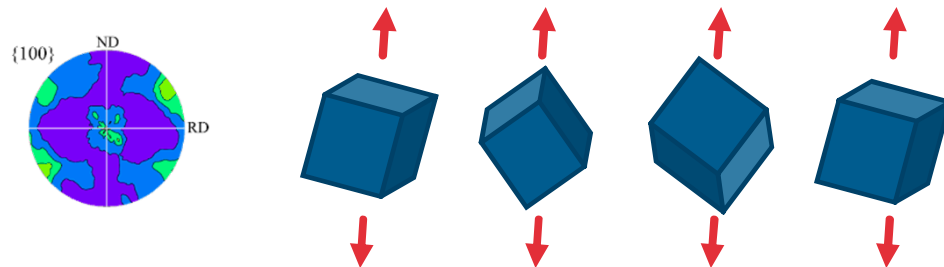
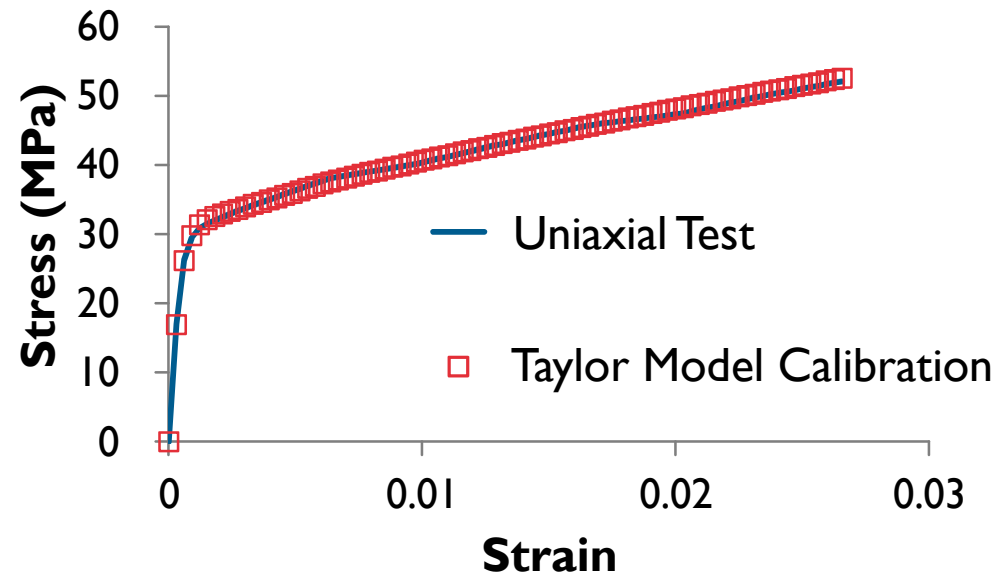
Nye Tensor with Enforced Antisymmetry



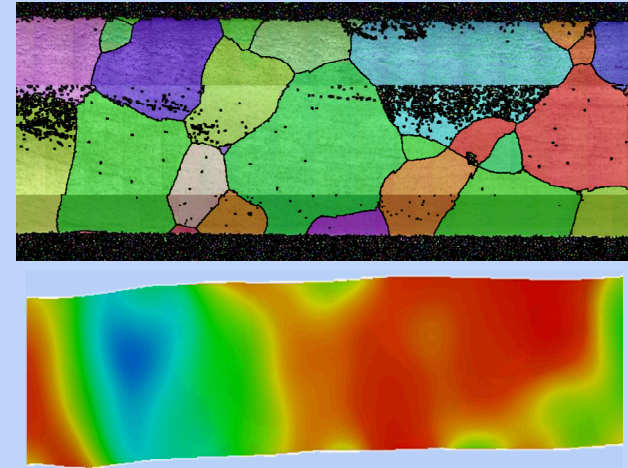


ACCOMMODATING HREBSD MEASUREMENT ERROR

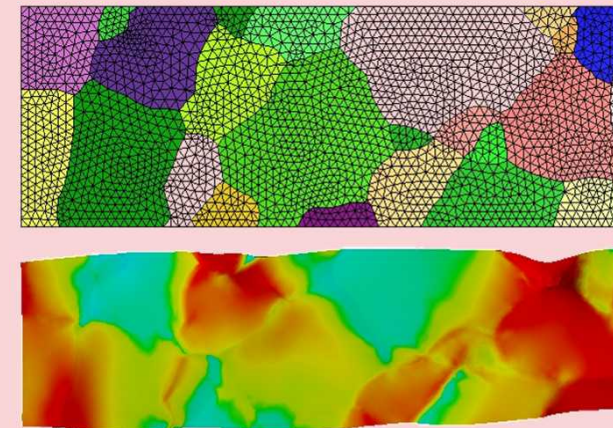
MODEL CALIBRATION - COMMON PRACTICE



Experiment

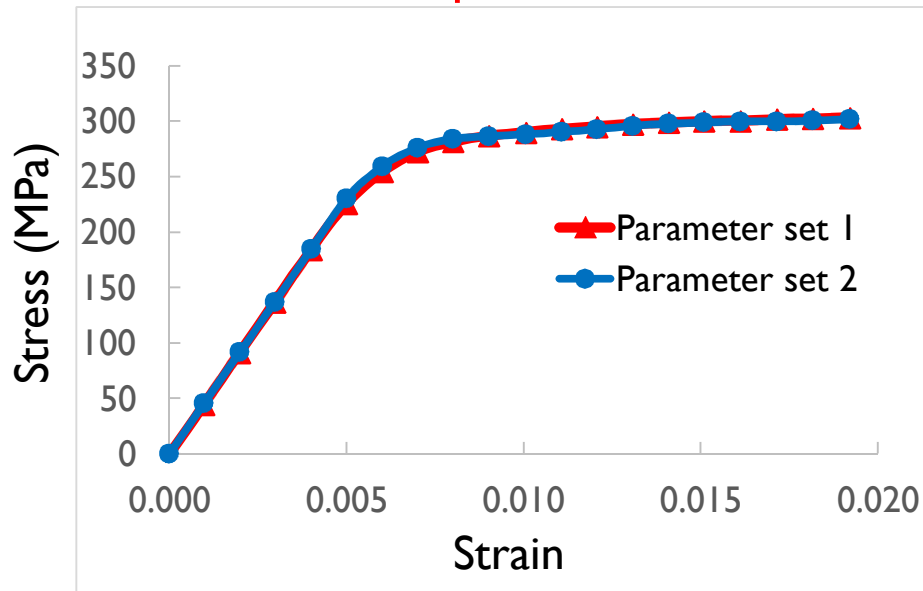


FE Model

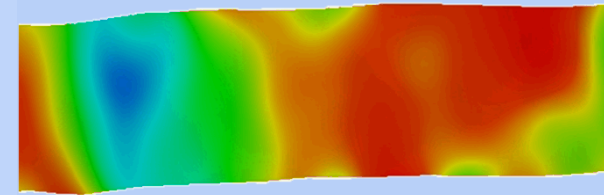
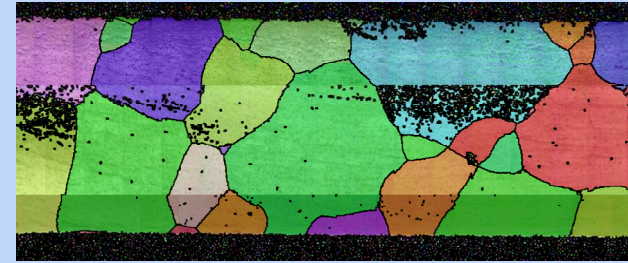


MODEL CALIBRATION - COMMON PRACTICE

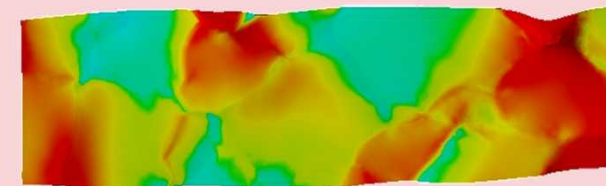
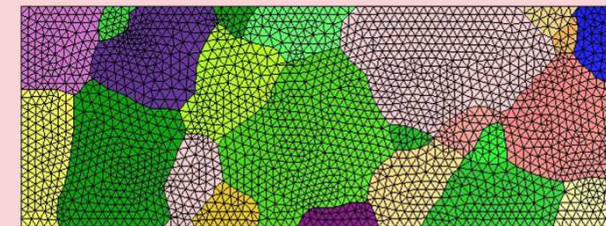
- ◆ Calibrated with uniaxial test:
under-constrained
- ◆ Compare local deformation/response
- ◆ Calibrate at scale it will be used!
- ◆ Utilize tools for measuring local deformation/response in calibration!



Experiment



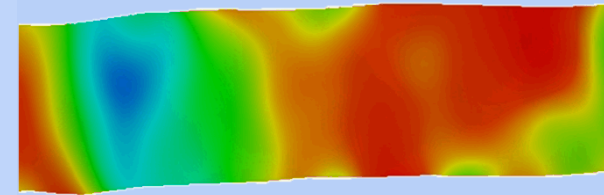
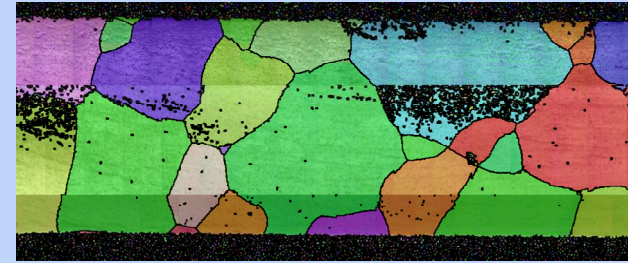
FE Model



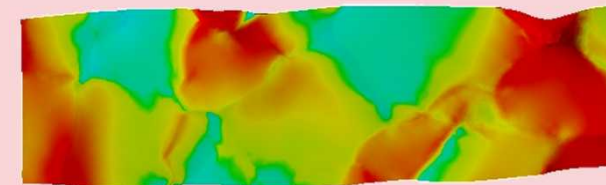
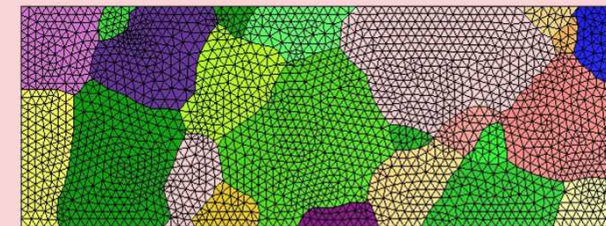
INVERSE FINITE ELEMENT SIMULATIONS

- ◆ Need to know grain structure throughout
- ◆ High computational cost (often intractable, especially for uncertainty quantification)

Experiment

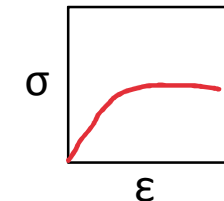
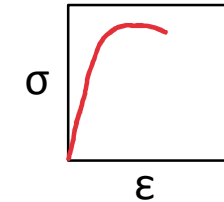
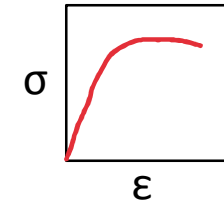
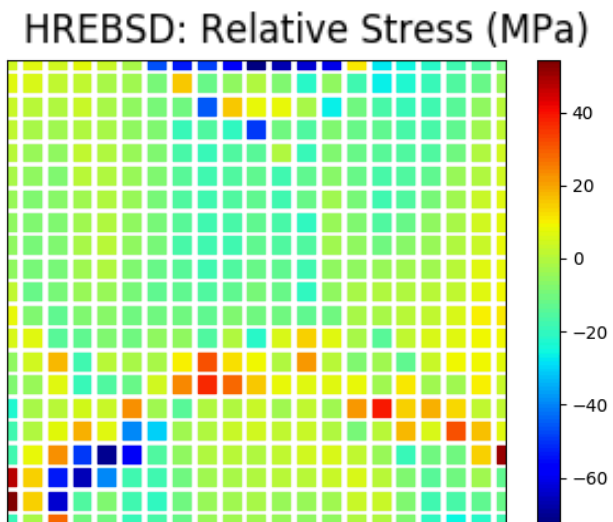
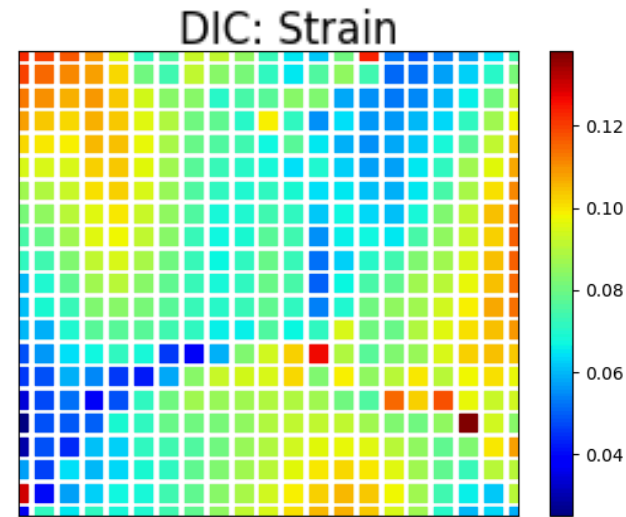


FE Model



INVERSE MATERIAL POINT SIMULATIONS (iMPS)

- ◆ Decouple material points
 - ◆ Local deformation from **DIC**
 - ◆ Local response from **HREBSD**
 - ◆ Only a material model to solve, no global stiffness matrix
-
- ◆ No need to know grain structure throughout
 - ◆ No need to solve computationally expensive FE problem! (3 orders of magnitude speed-up)

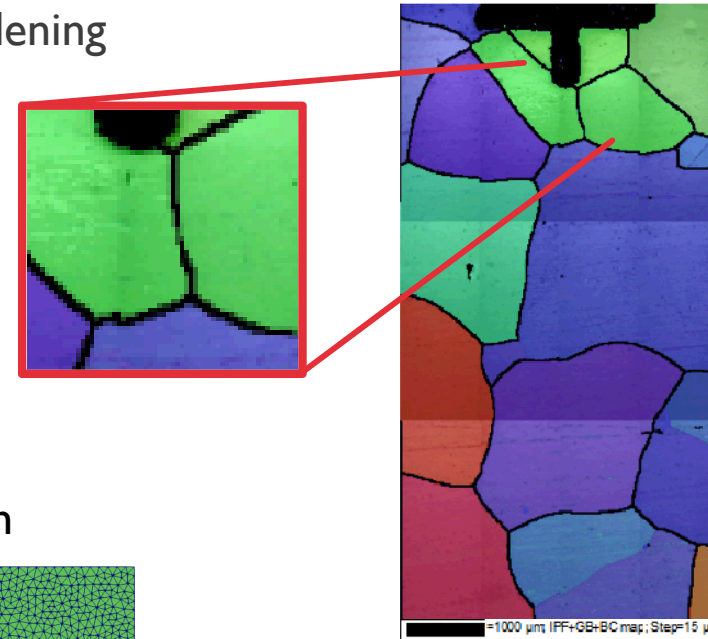


SIMULATED DIC AND HREBSD

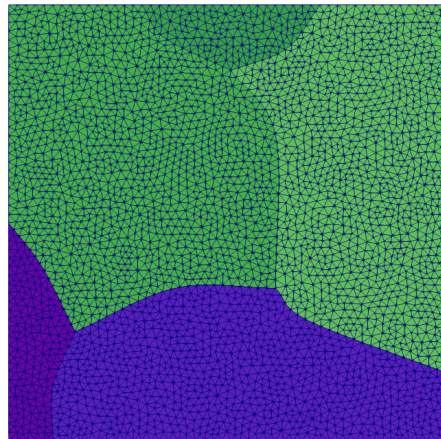
- ◆ Physical calibration experiment still underway
- ◆ Simulated experiment: FE Model with Voce Kocks hardening

$$\Theta = (g_0, m, G_0, g_s^*, \omega)$$
$$(18.0, 0.02, 270, 44.0, 5 \times 10^{-6})$$

Physical specimen



FE Simulation



SIMULATED DIC AND HREBSD

◆ Physical calibration experiment still underway

◆ Simulated experiment: FE Model

$$\Theta = (g_0, m, G_0, g_s^*, \omega)$$
$$(18.0, 0.02, 270, 44.0, 5 \times 10^{-6})$$

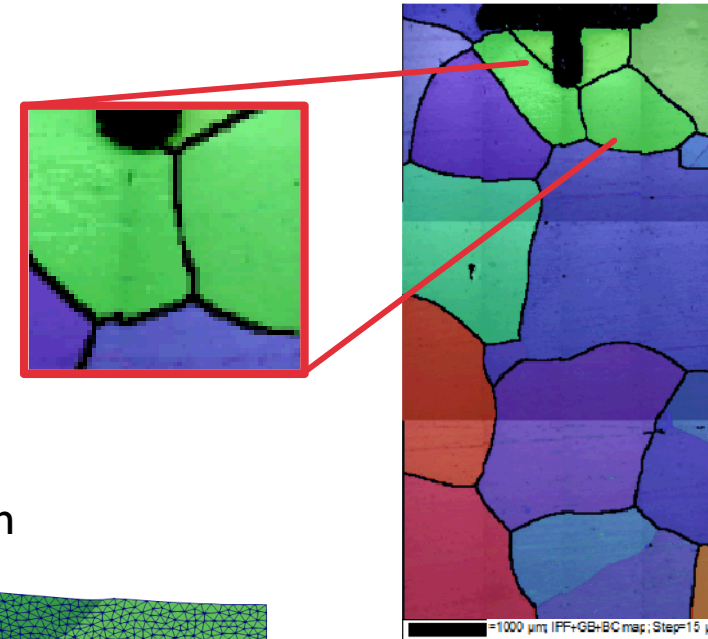
◆ Strains + noise => DIC

◆ Volume average stress => F-D

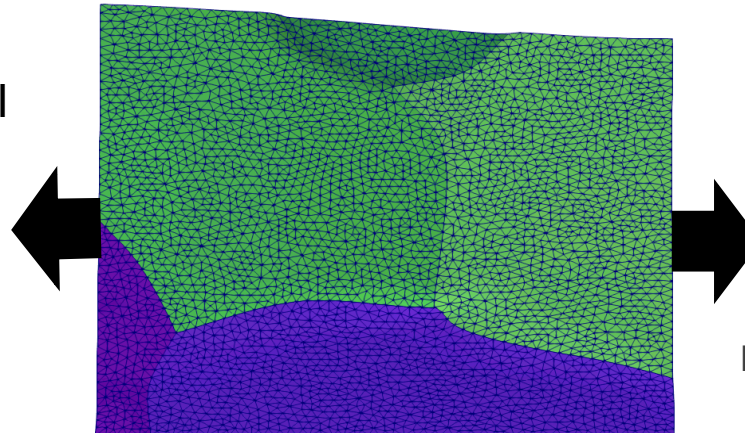
◆ Relative stresses + noise => HREBSD

◆ Determine distributions of model parameters with Markov Chain Monte Carlo

Physical specimen



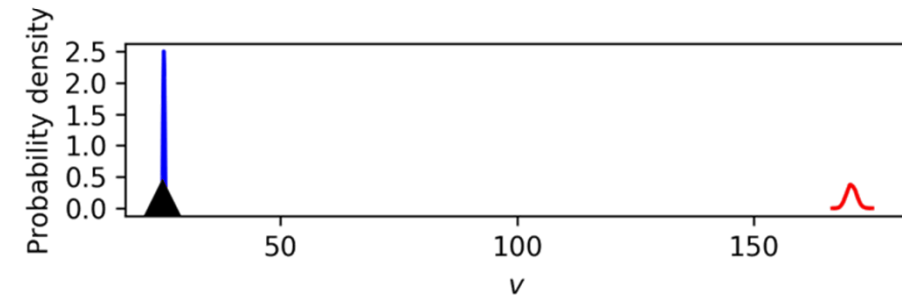
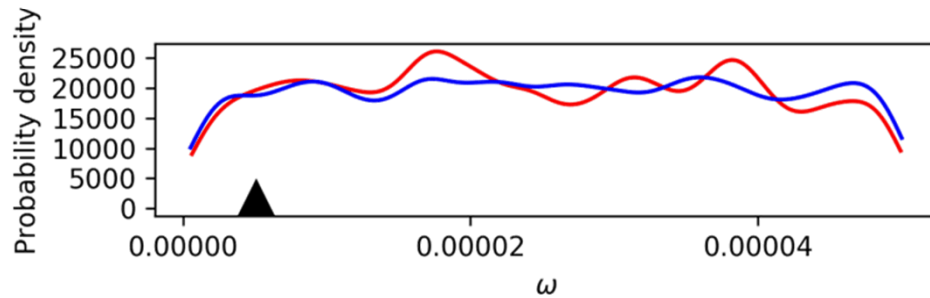
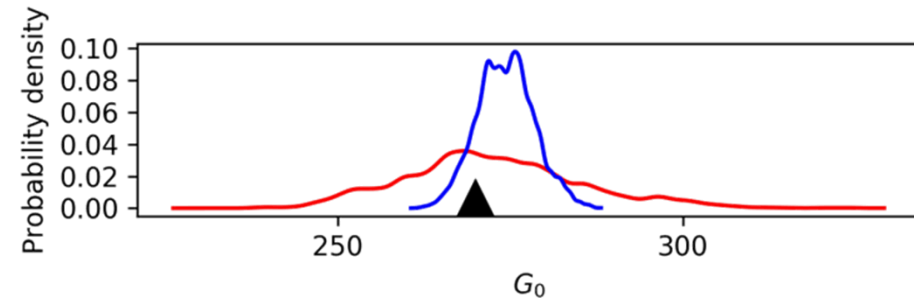
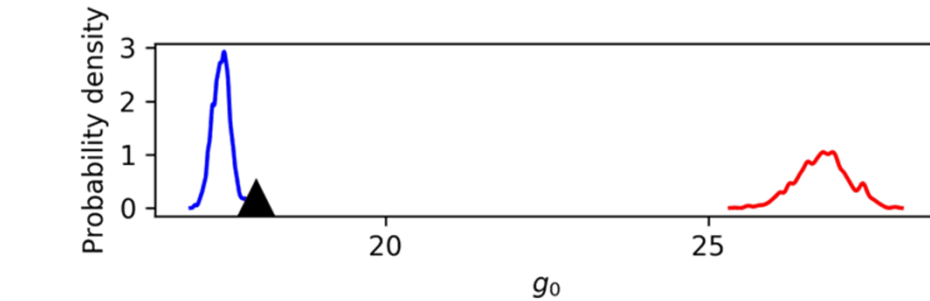
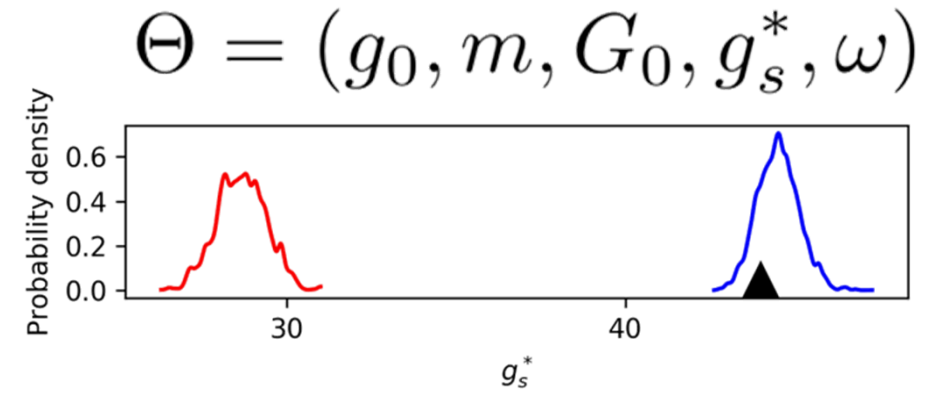
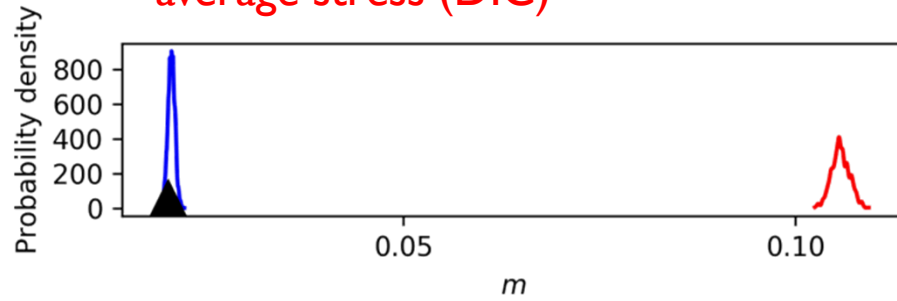
FE Simulation

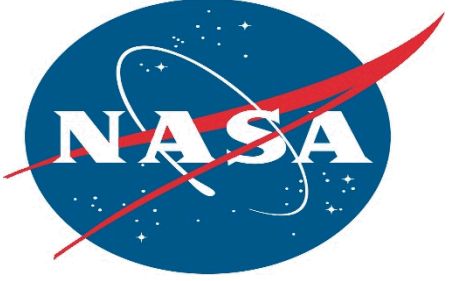


FE performed with ScifEN

RESULTS

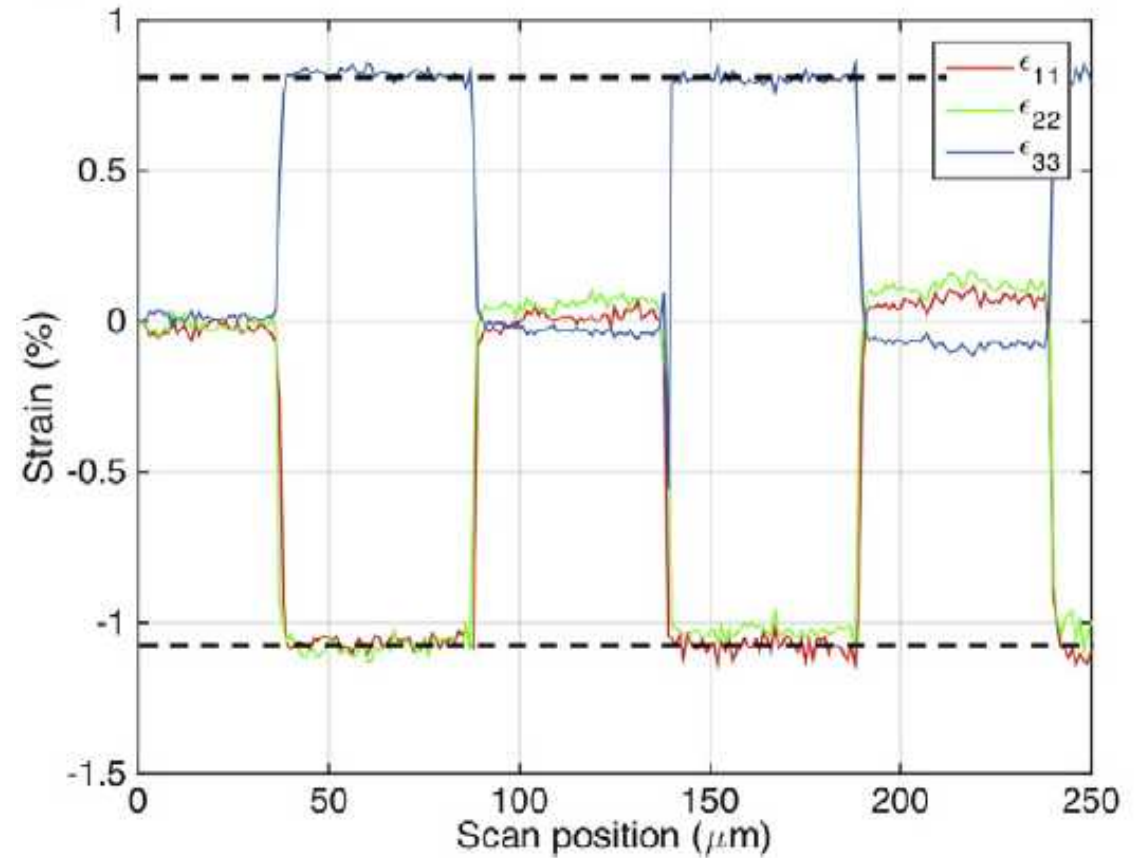
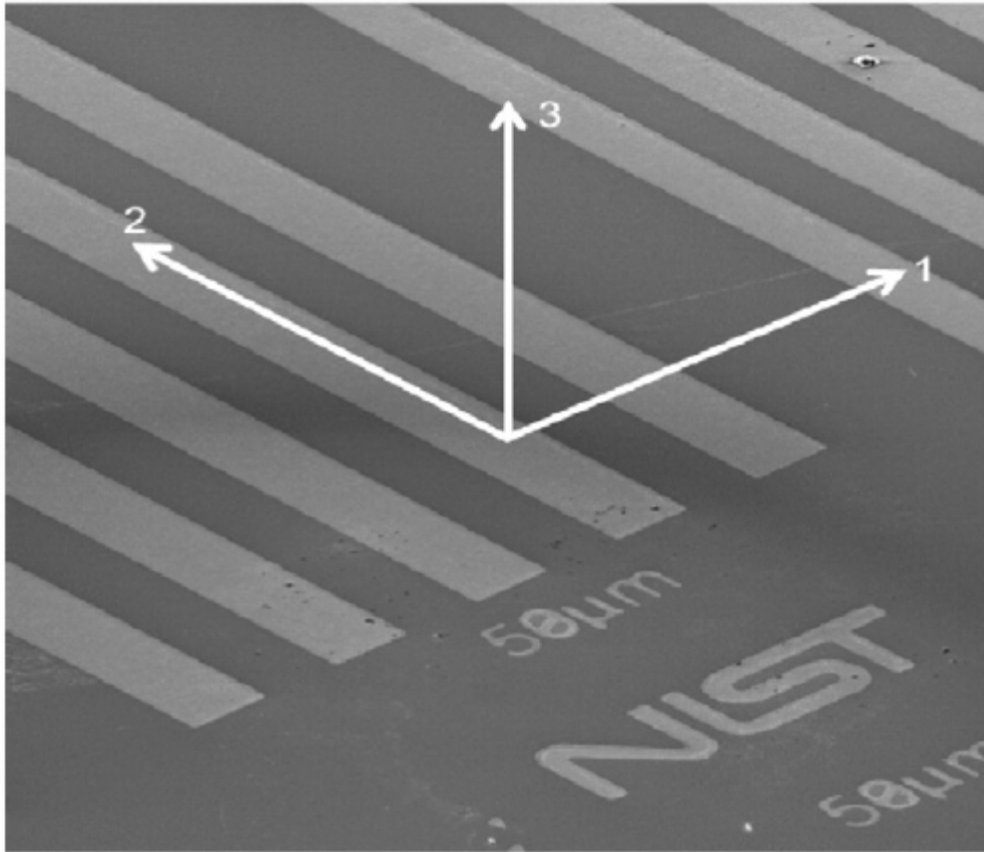
relative stress (DIC+HREBSD)
average stress (DIC)





CROSS-VALIDATION STUDIES

X-RAY



Fullwood, D., M. Vaudin, C. Daniels, T. Ruggles, and S. I. Wright (2015). Validation of kinematically simulated pattern HR-EBSD for measuring absolute strains and lattice tetragonality. *Materials Characterization* 107, 270 –277.

ECCI

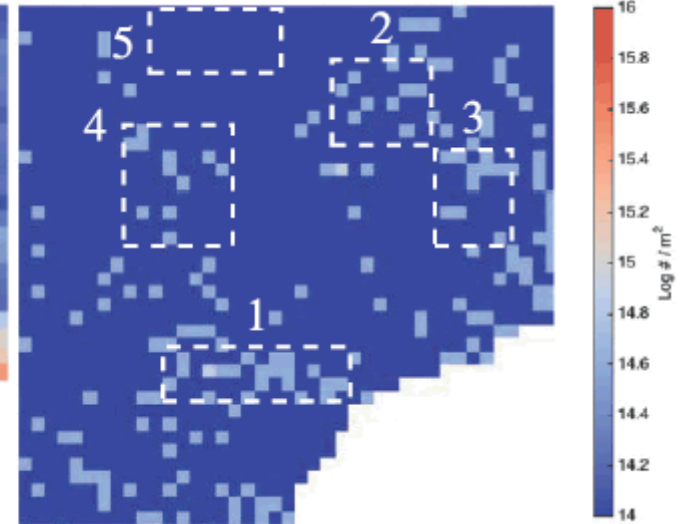
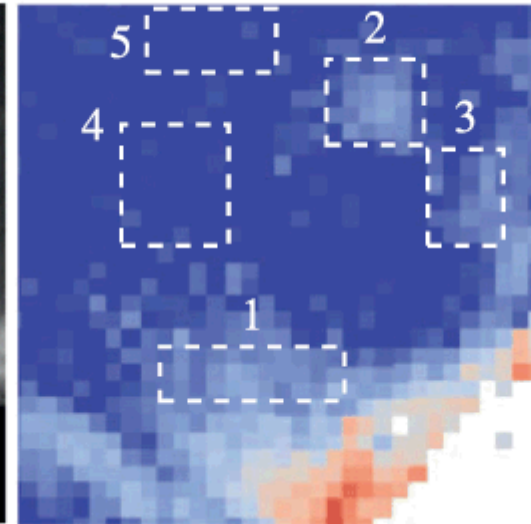
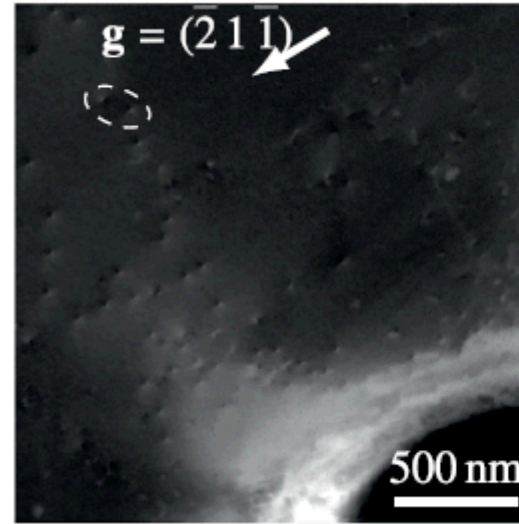
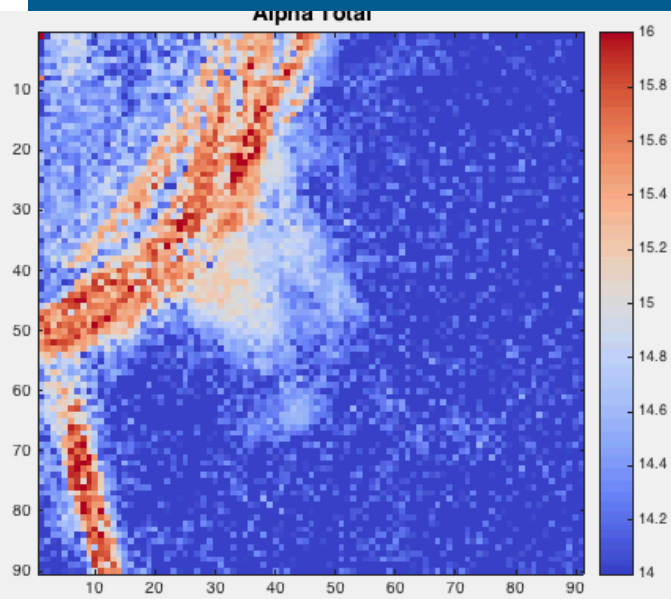


Table 1

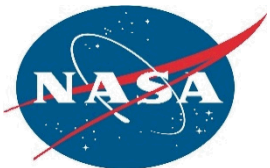
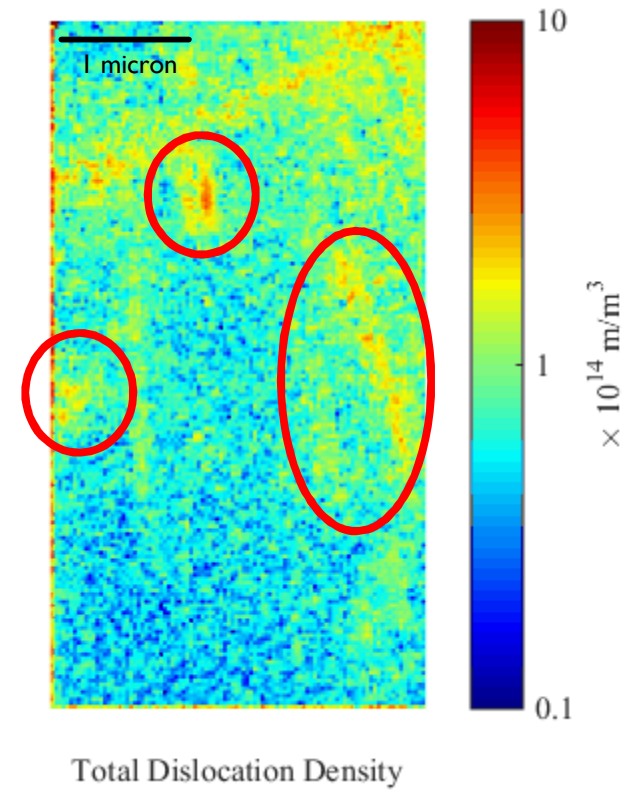
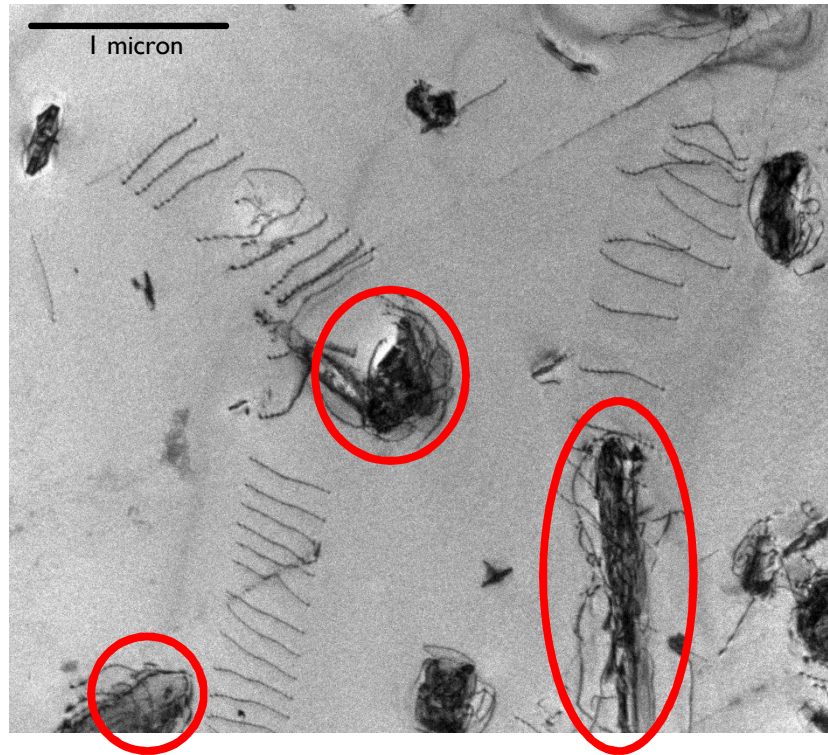
Comparison of CC-EBSD GND densities and ECCI dislocation densities for the 5 regions shown in Fig. 3.

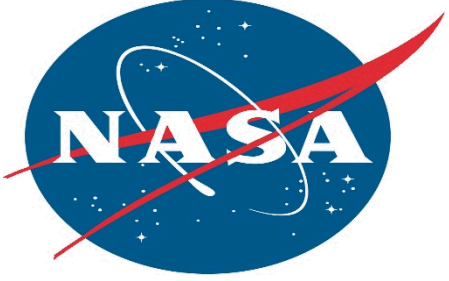
Region #	CC-EBSD GND Density	ECCI Density	ECCI (Line Direction Correction)	ECCI (Dipole Correction)
1	$2.6 \cdot 10^{14} \text{ m}^{-2}$	$1.6 \cdot 10^{14} \text{ m}^{-2}$	$2.1 \cdot 10^{14} \text{ m}^{-2}$	$1.9 \cdot 10^{14} \text{ m}^{-2}$
2	$2.1 \cdot 10^{14} \text{ m}^{-2}$	$8.6 \cdot 10^{13} \text{ m}^{-2}$	$1.6 \cdot 10^{14} \text{ m}^{-2}$	No Dipoles
3	$1.7 \cdot 10^{14} \text{ m}^{-2}$	$1.1 \cdot 10^{14} \text{ m}^{-2}$	$2.2 \cdot 10^{14} \text{ m}^{-2}$	No Dipoles
4	$6.4 \cdot 10^{13} \text{ m}^{-2}$	$6.1 \cdot 10^{13} \text{ m}^{-2}$	$8.0 \cdot 10^{13} \text{ m}^{-2}$	No Dipoles
5	$5.2 \cdot 10^{13} \text{ m}^{-2}$	0 m^{-2}	0 m^{-2}	No Dipoles

Dunlap, B. E., T. J. Ruggles, D. T. Fullwood, B. Jackson, and M. A. Crimp (2018). Comparison of dislocation characterization by electron channeling contrast imaging and cross-correlation electron backscattered diffraction. *Ultramicroscopy* **184**, 125–133.

TEM

Bulk GND measurements show qualitative agreement

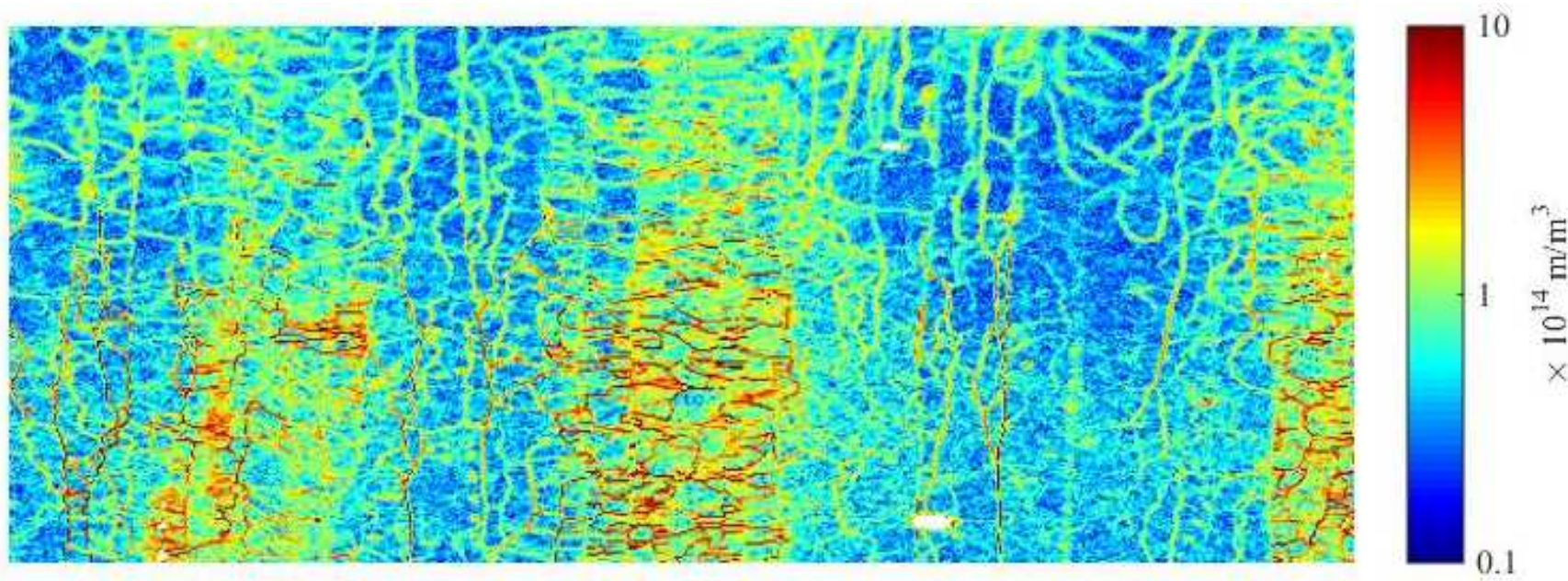




CONCLUSIONS AND FUTURE WORK

CONCLUSIONS

- ◆ HREBSD provides valuable local data about material state: elastic strain, stress and dislocation density
- ◆ Local data can be used to develop and calibrate high fidelity microstructure models
- ◆ Methods have been presented here to increase the accuracy of HREBSD measurements, including pattern center calibration, and the elimination of noisy terms in the distortion measurements.



FUTURE WORK

- ◆ Collect a combined DIC/HREBSD data set and use it to calibrate/develop a high fidelity model with uncertainty included
- ◆ Integrate ICGN HREBSD and PC calibration into OpenXY
- ◆ Incorporate HREBSD data into the Material Knowledge System (MKS)
- ◆ Validate GND measurements with TEM
- ◆ Apply these techniques to TKD

Questions?

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