STRAIN MAPPING BY HIGH ANGULAR RESOLUTION EBSD (AKA HR-EBSD)

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http://expmicromech.com

Acknowledgments: Angus Wilkinson, Angus Kirkland, Fionn Dunne, Jun Jiang, Vivian Tong, David Wallis, Lars Hansen, Aimo Winkelmann

Outline

- EBSD vs HR-EBSD
- Examples
- Strain and deformation
- HR-EBSD fundamentals
- Accuracy, precision and sensitivity
- Pattern Remapping
- Summary

EBSD vs HR-EBSD

	EBSD	HR-EBSD
Absolute Orientation	~2°	No
Misorientation	~0.1 to 0.5°	~0.006°
Deformation –		
GNDs @ 1µm step	> $3x10^{13}$	> $3x10^{11}$
GNDs @ 100nm step **in lines / m²** (b = 0.3nm)	> $3x10^{12}$	> $3x10^{10}$
Relative elastic strain	No	Deviatoric strain ± $1x10^{-4}$
Relative residual stress (Type III – within grain)	No	Anisotropic Hooke's law ± 20 MPa (E=200GPa)
Example tasks:	Microstructure, Texture, Grain size, etc.	**Deformation** i.e. elastic strain, misorientation & residual dislocation content

Strain and deformation

Strain and deformation

- Two types of strain tensor
  - Elastic (i.e. stress)
  - Plastic

- Elastic leads to a change in bond length / angle
  → HR-EBSD!

- Plastic more tricky…

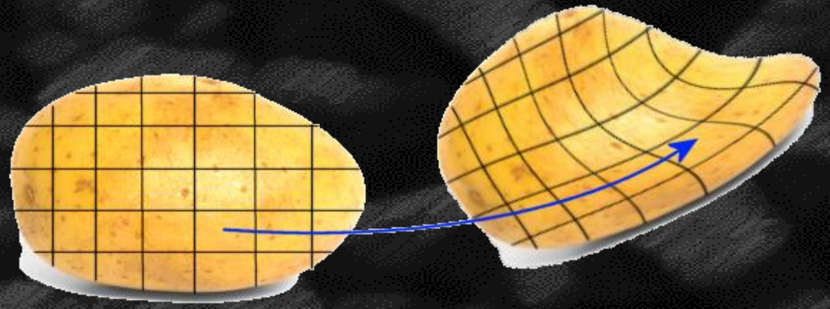
Strain and deformation

Plastic shape change
due to slip etc.
Lattice remains un-deformed

$$\boldsymbol{F} = \boldsymbol{F}^e \boldsymbol{F}^p$$

Elastic shape change
→ Bond stretch etc.
→ Lattice orientation
due to slip etc.

$$\boldsymbol{F} = \begin{pmatrix} F_{11} & F_{12} & F_{13} \\ F_{21} & F_{22} & F_{23} \\ F_{31} & F_{32} & F_{33} \end{pmatrix} = \begin{pmatrix} \frac{dx}{dX} & \frac{dx}{dY} & \frac{dx}{dZ} \\ \frac{dy}{dX} & \frac{dy}{dY} & \frac{dy}{dZ} \\ \frac{dz}{dX} & \frac{dz}{dY} & \frac{dz}{dZ} \end{pmatrix} + \boldsymbol{I}$$



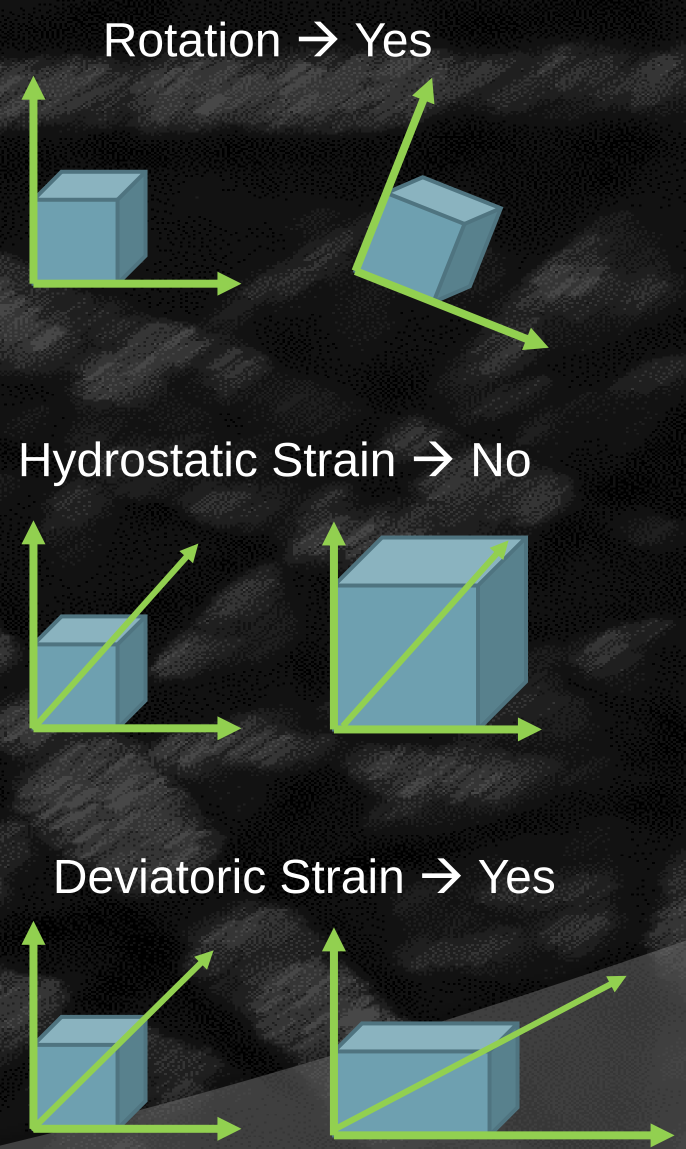
Strain is fundamentally a 2nd rank tensor

Many metrics are proxies
→ use with care!

Potato deformation
(from http://www.continuummechanics.org/cm/deformationgradient.html)

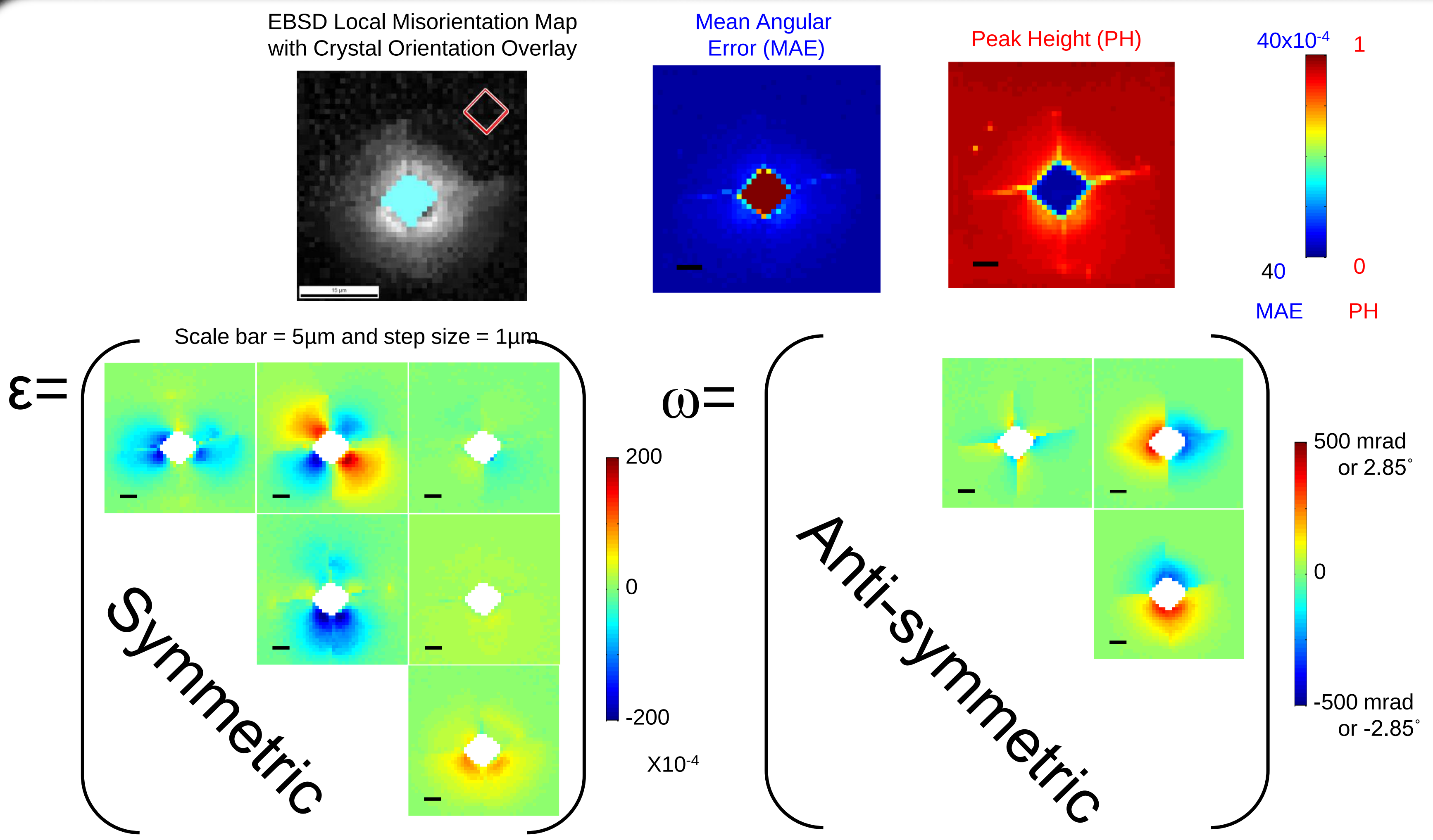
HR-EBSD and F^e

- Follow change in interplanar angles within the diffraction (with high precision)
- Apply simple geometry and extract F^e
- Split F^e into elastic strain (deviatoric) & lattice rotation
- Use lattice rotation gradients to evaluate GNDs (a symptom of plastic strain)



Examples

Strain & rotation



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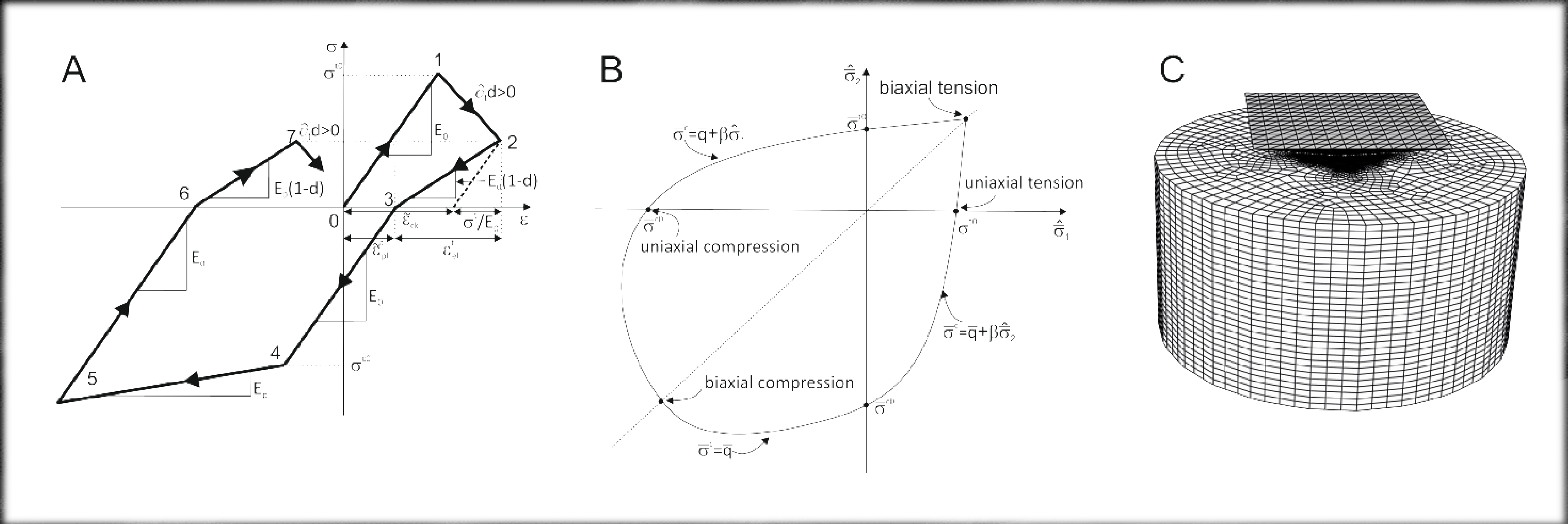
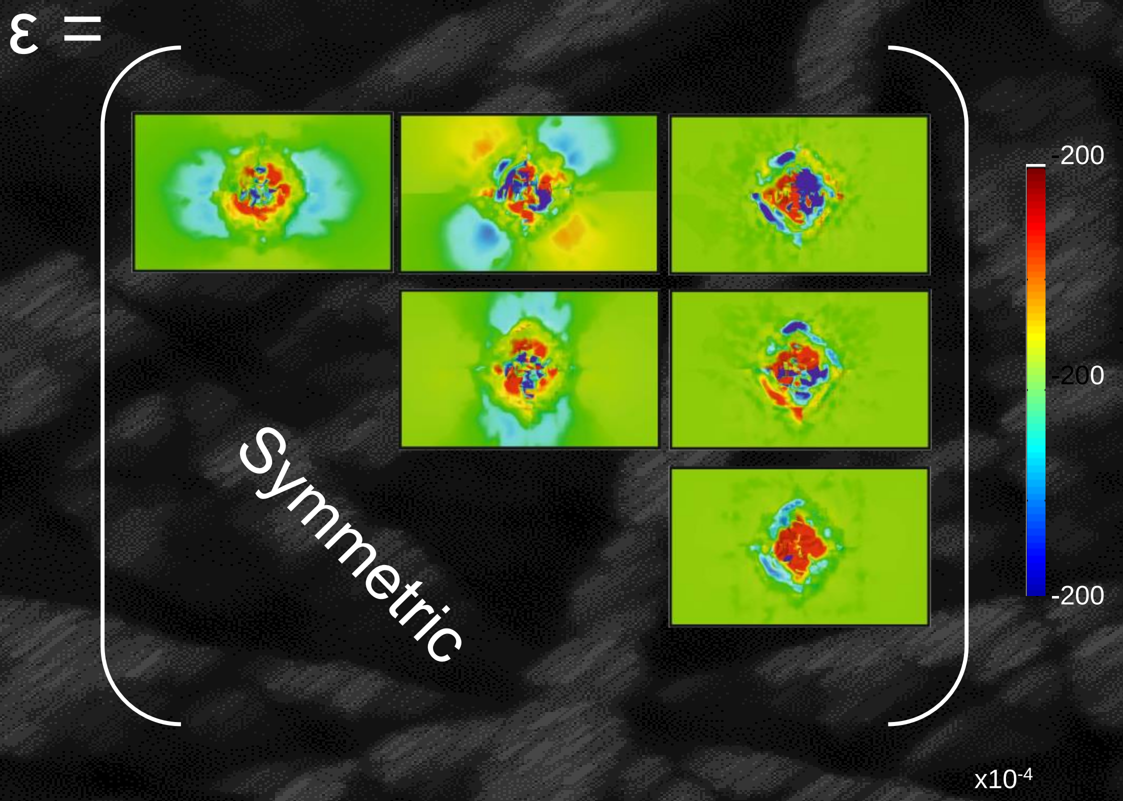
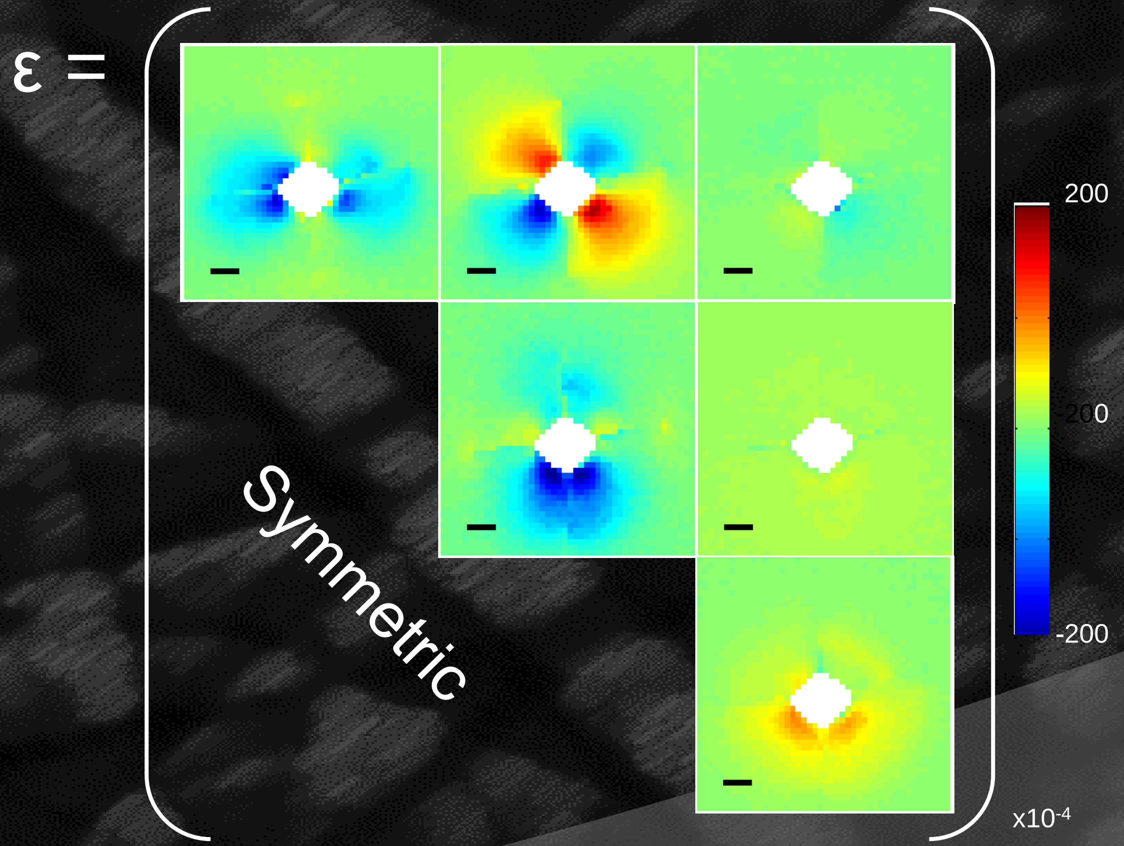
Comparison with models

- 50g force
- Concrete damage model + cohesive zones along <110>



Comparing Strain Fields





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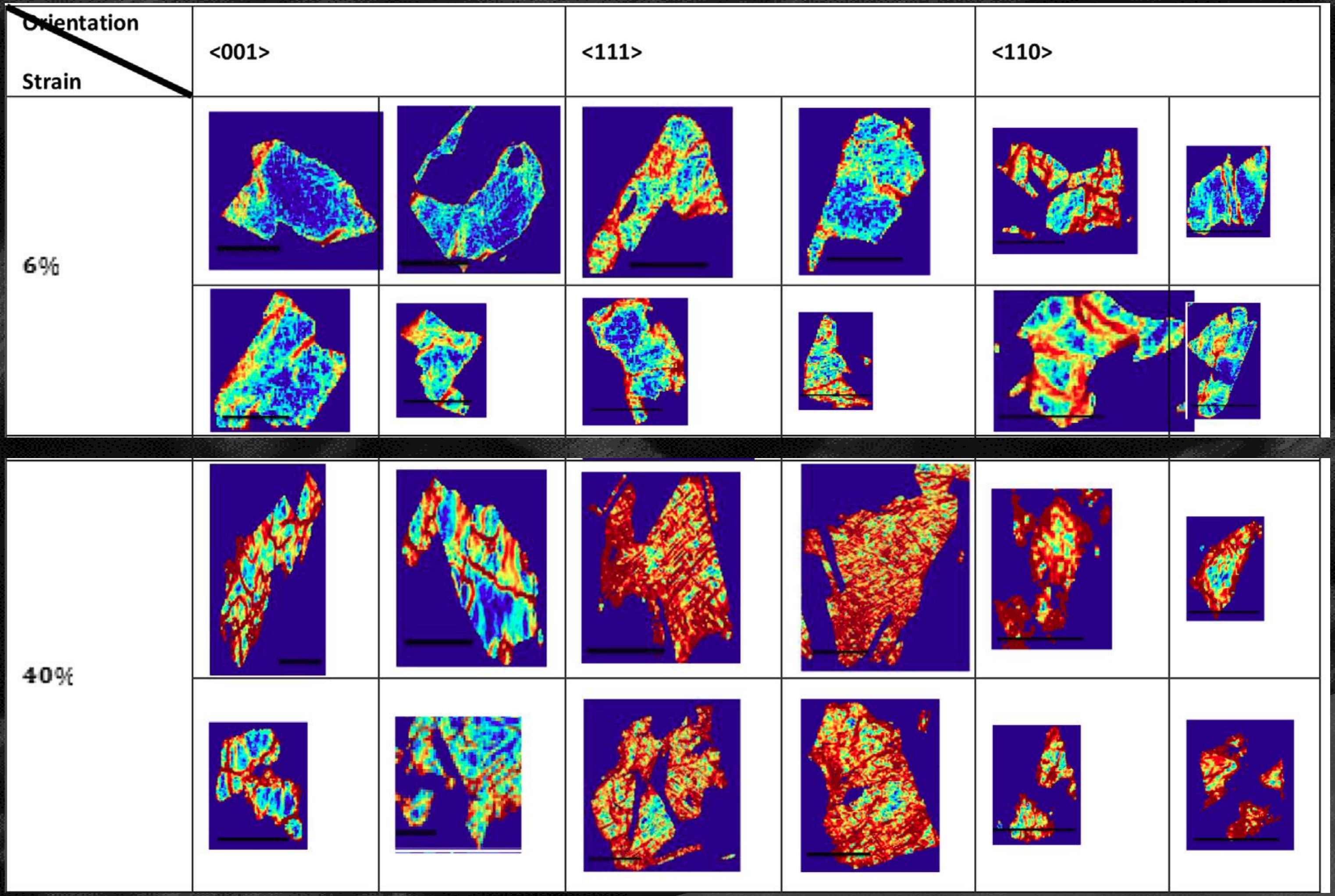
Jiang, Britton and Wilkinson (2015) IJP

Microstructure – dislocation structure correlations (Cu)

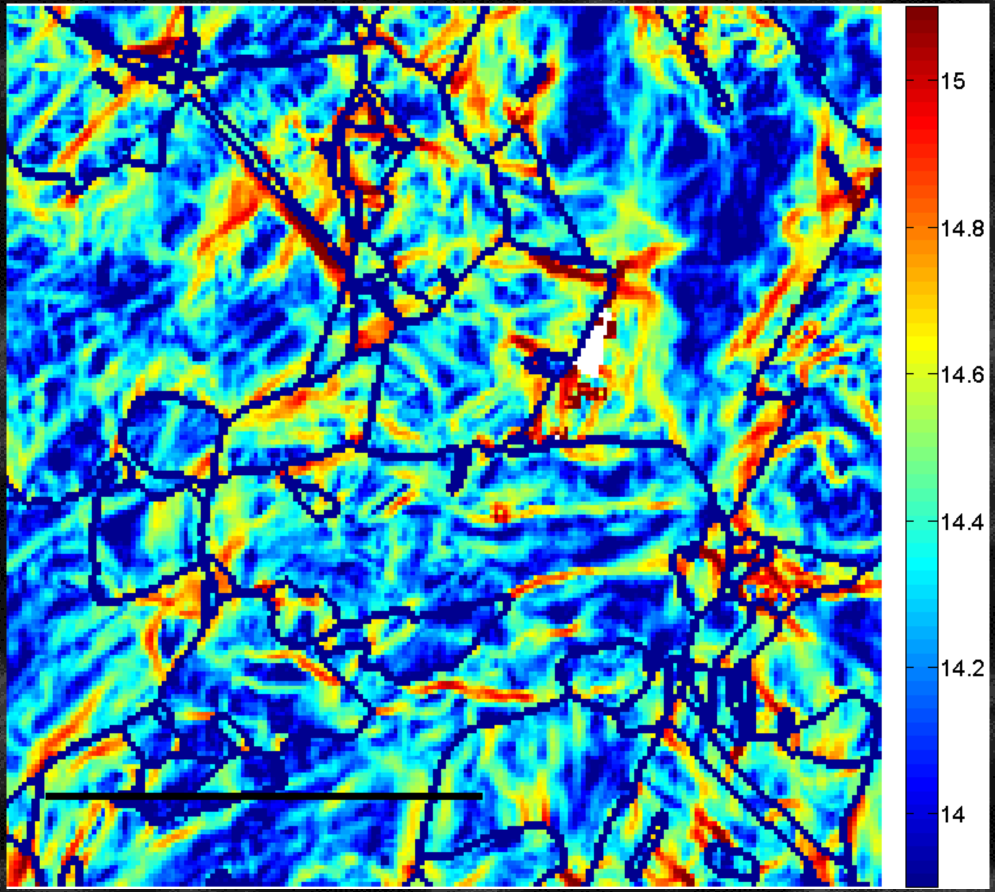
- Evolution of dislocation structure with uniaxial strain
  - Avoids thin films
  - Cover large areas
  - Access many grains

- Correlate with tensor quantities
  - Schmid factor, Taylor factor etc.

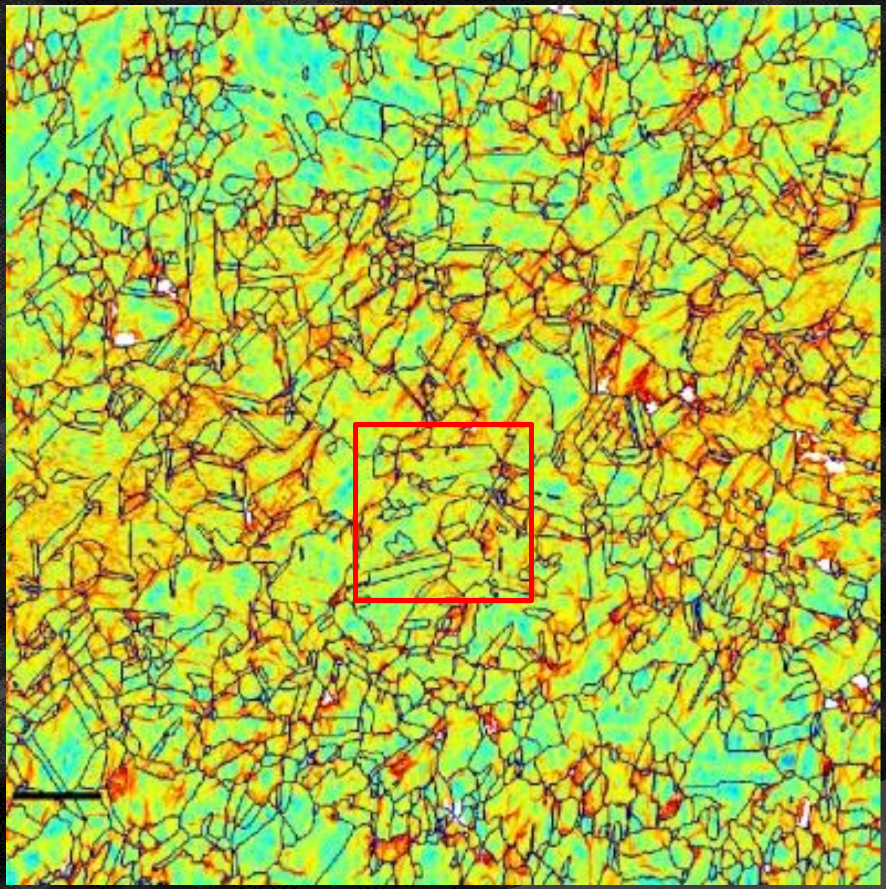
- Can also compare with crystal plasticity models



Big data…



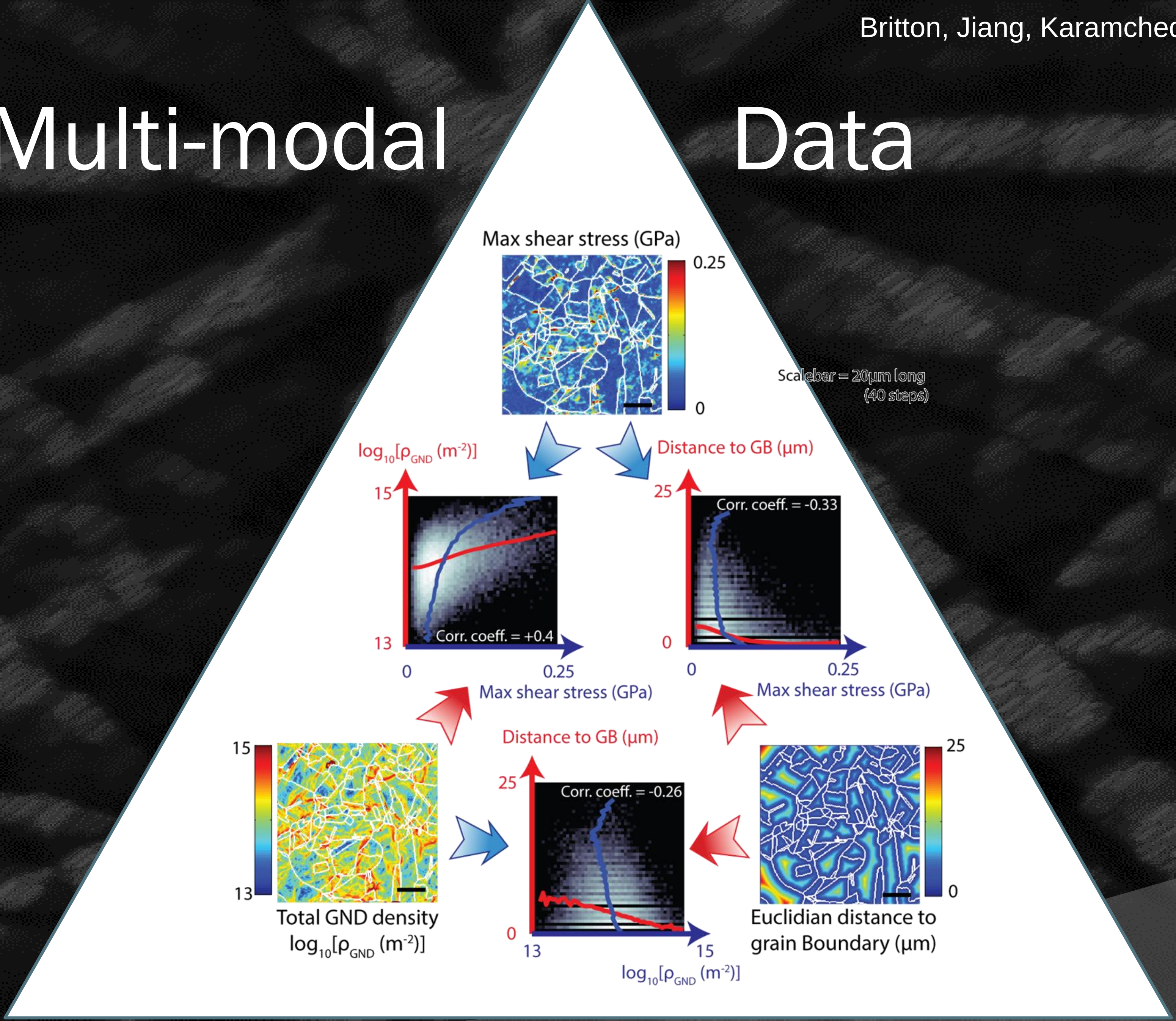
~110μm square = 30-60 grains



~500μm square = 1500 grains

Britton, Jiang, Karamched and Wilkinson (2013) *JOM*

Multi-modal Data

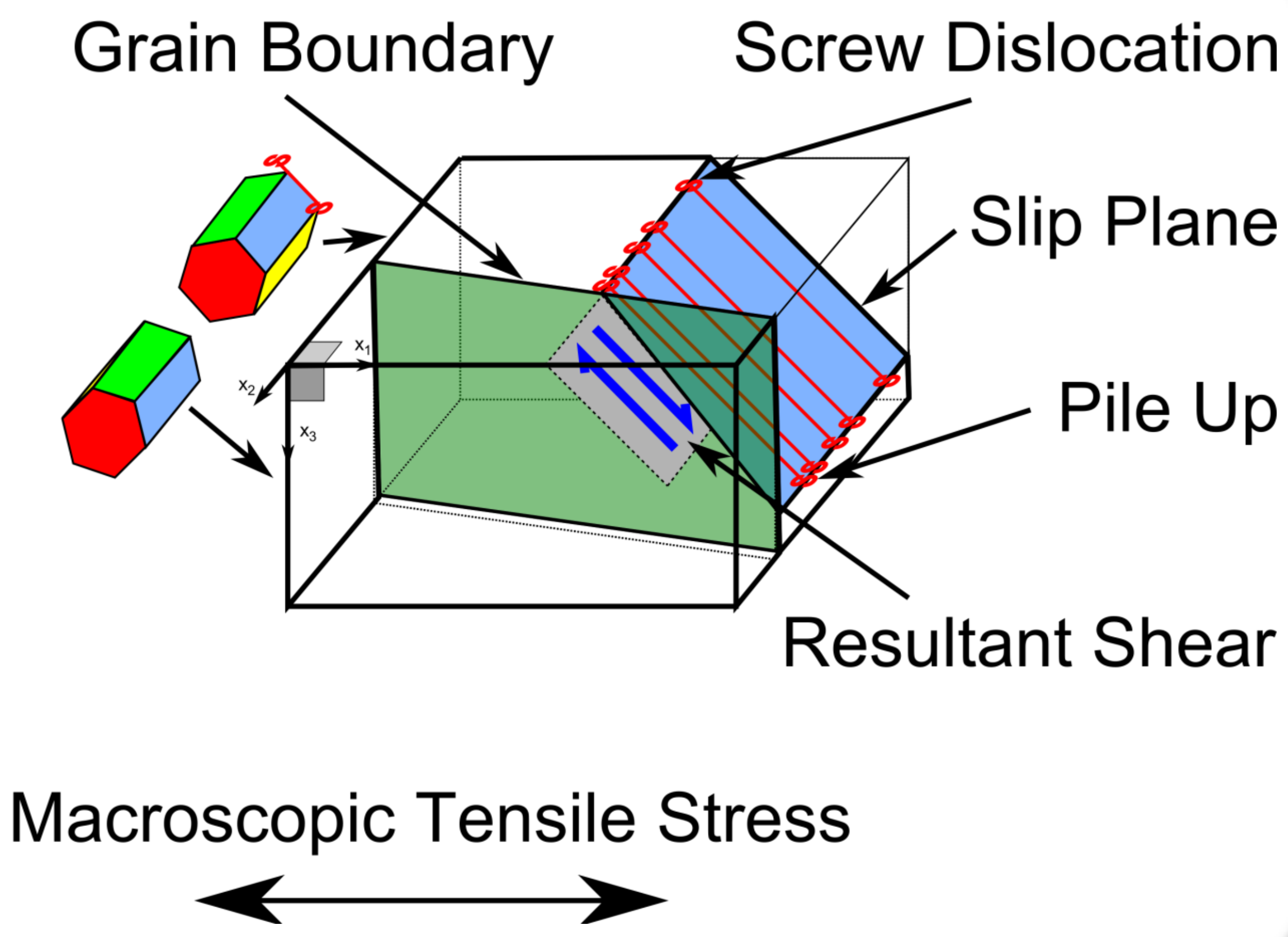


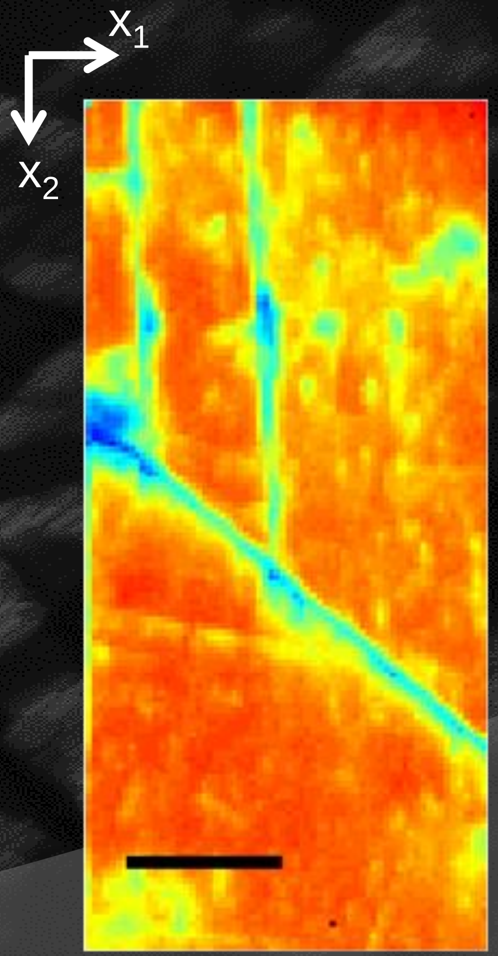
New insight – g.b. strength

Britton and Wilkinson (2013) *Acta Mat*

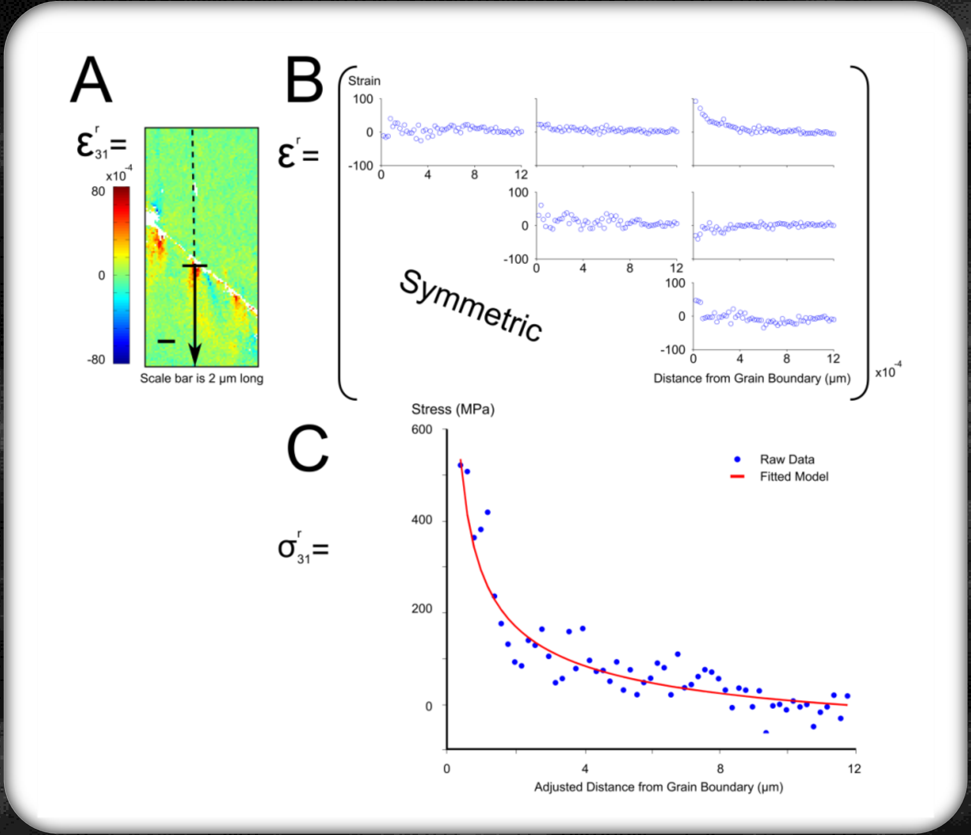
Guo, Y., Britton, T.B. and Wilkinson, A.J. (2014) *Acta Mat*

Guo, Y., Collins, D.M., Tarleton, E., Hoffman, F., Tischler, J., Liu, W., Xu, R., Wilkinson, A.J. and Britton, T.B. (2015) *Acta Mat*





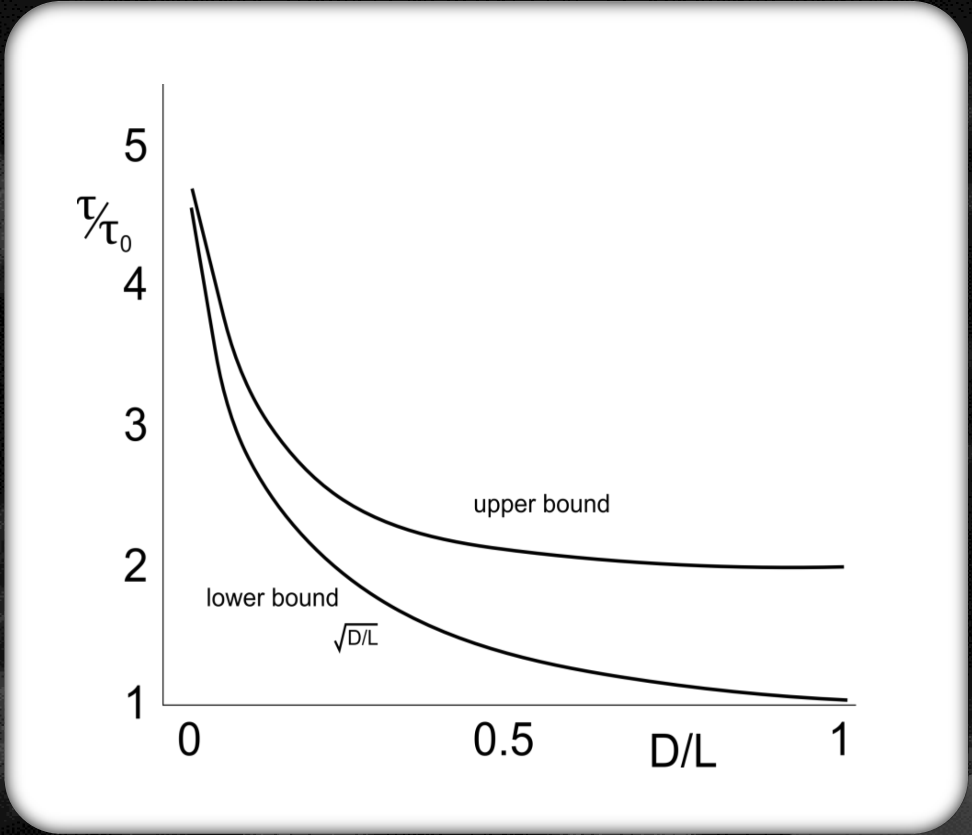
Verifying Eshelby, Frank and Nabarro



Measurement of stress and strain near g.b.

fitting: $\sigma_{31} = A + K / \sqrt{D}$

$K = 0.42\ \mathrm{MPa}\sqrt{m}$



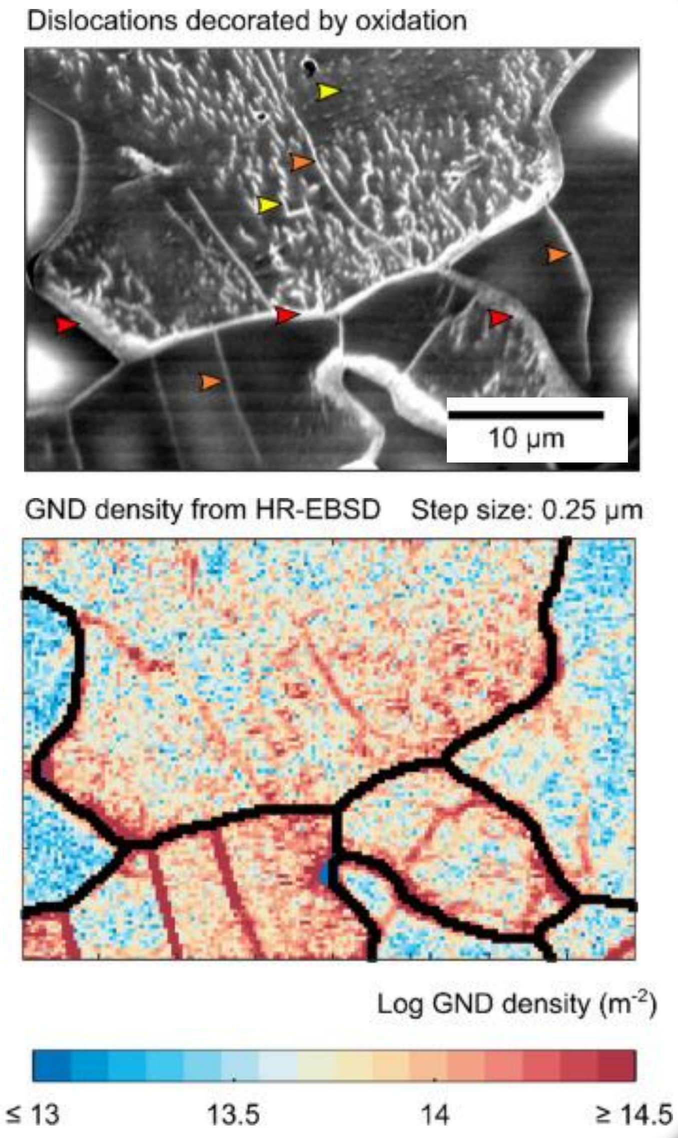
Normalised shear stress vs normalised distance from a screw dislocation pile up

Redrawn from Eshelby, Frank, Nabarro (1951)

Wallis, D., Hansen, L.N., Britton, T.B. and Wilkinson, A.J. (2016) *Ultramicroscopy*

HR-EBSD Rocks

- All we need are good EBSD patterns…
- Other material systems can be explored
  - e.g. Metals, Semiconductors, Rocks, Intermetallics etc.
- Example here – dislocations in olivine
  - Good correlation between dislocation decoration (oxidation) & GND measurements
  - Important for understanding earth formation

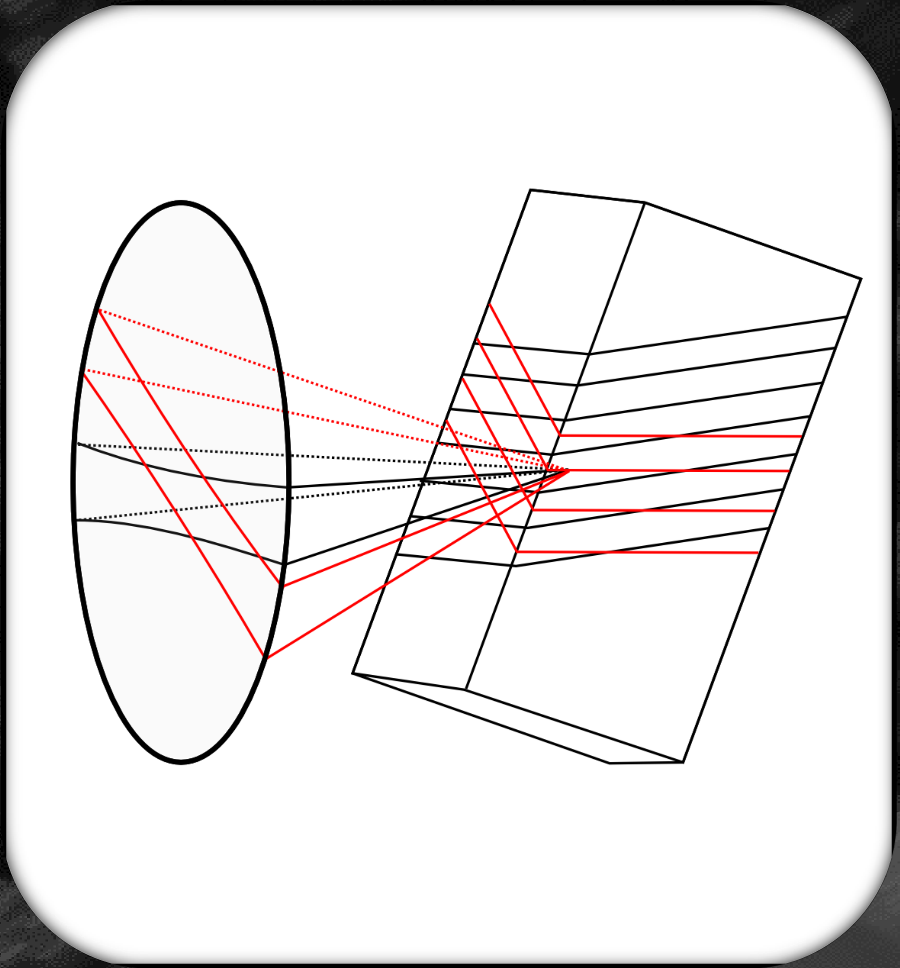


HR-EBSD Fundamentals

Method described in Britton, Jiang, Karamched and Wilkinson in *JOM* (2013)

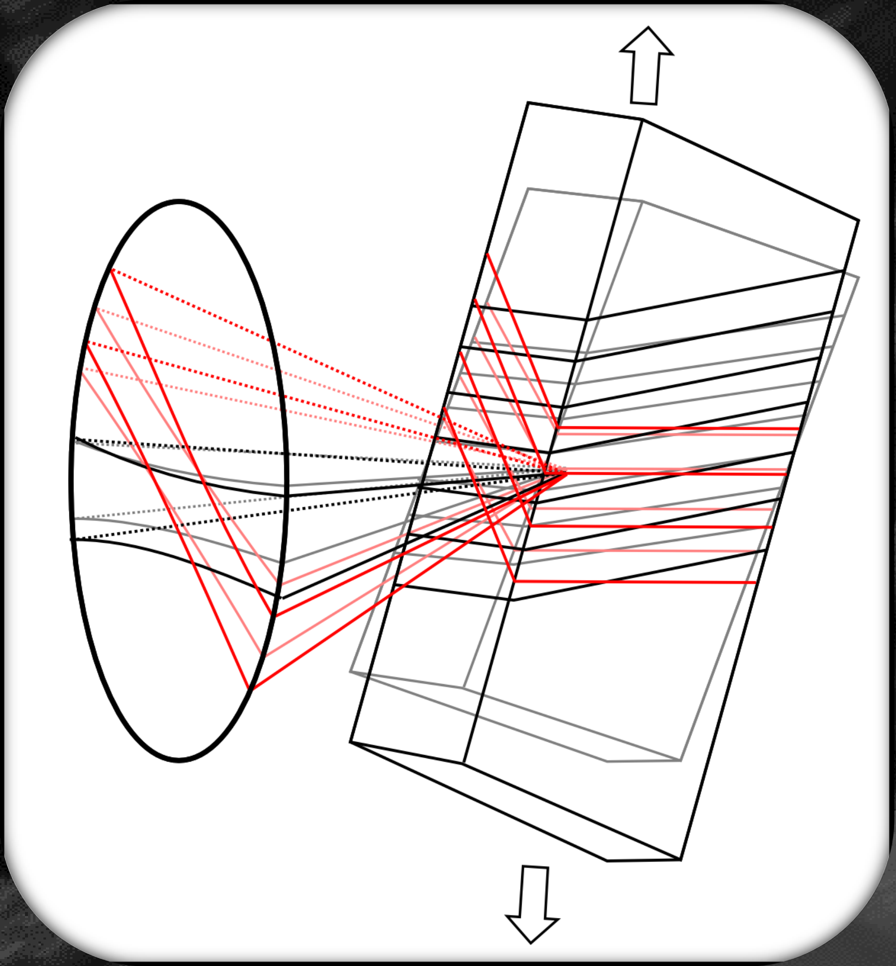
Accessing Elastic Strain – HR-EBSD

- EBSD pattern = direct projection of lattice planes



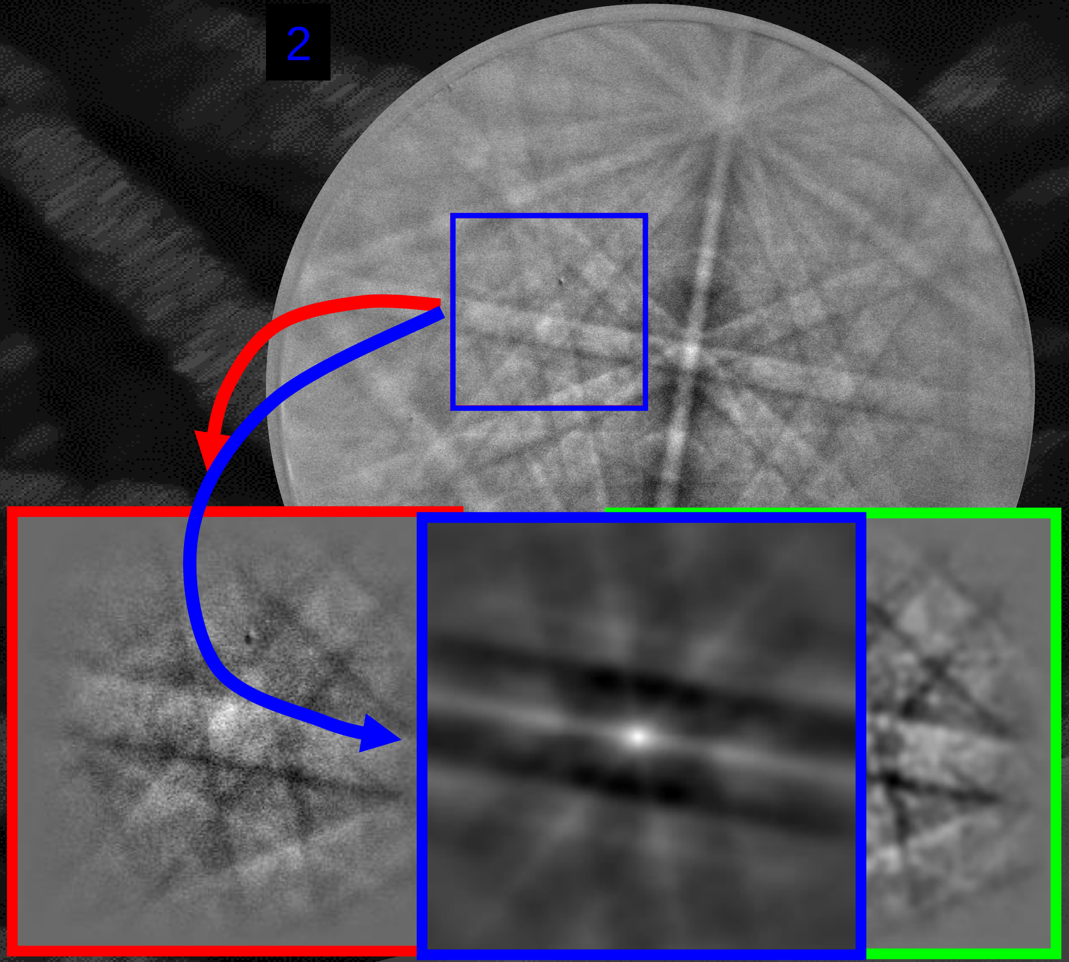
Accessing Elastic Strain – HR-EBSD

- EBSD pattern = direct projection of lattice planes
- Strain of crystal = movement of bands



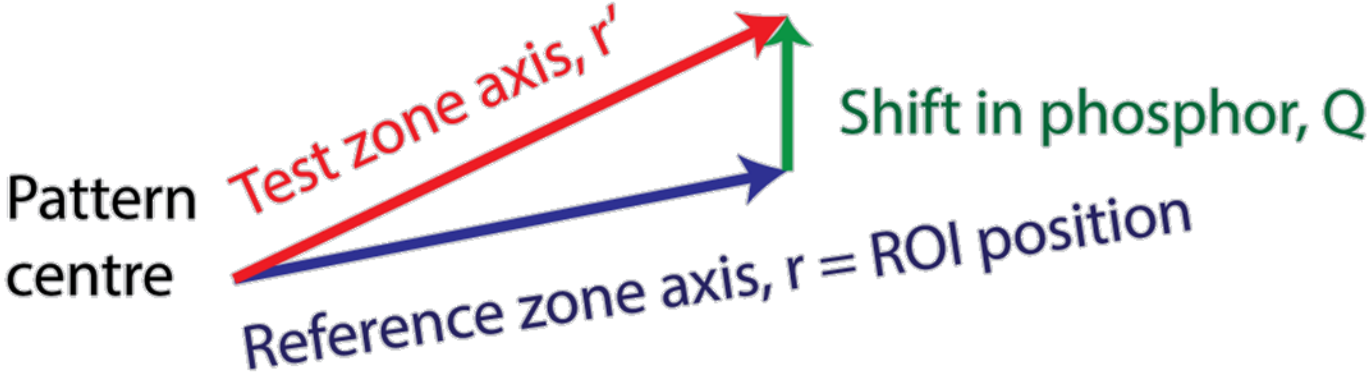
Measuring shifts with image correlation

- 20+ ROI used (offline)
  - Select ROIs → FFT
  - Apply filter
- Compare unstrained (1) vs strained (2) pattern
  - Upsample peak in XCF of ROI
    - xshift= -6.06 (pixels)
    - yshift= -4.59
- 'Just' an educated 'guess' of the translation vector between test & reference



Method described in Britton, Jiang, Karamched and Wilkinson in *JOM* (2013)

HR-EBSD – shifts to strains



$$r_x(A_{xx} - A_{zz}) + r_y A_{xy} + r_z A_{xz} + \frac{r_x r_x}{r_z} A_{zx} + \frac{r_y r_x}{r_z} A_{zy} = Q_x$$

$$r_x A_{yx} + r_y(A_{yy} - A_{zz}) + r_z A_{yz} + \frac{r_x r_y}{r_z} A_{zx} + \frac{r_y r_y}{r_z} A_{zy} = Q_y$$

Infinitesimal:

$$\omega = \frac{1}{2}(A - A^T)$$

$$\varepsilon = \frac{1}{2}(A + A^T)$$

Finite:

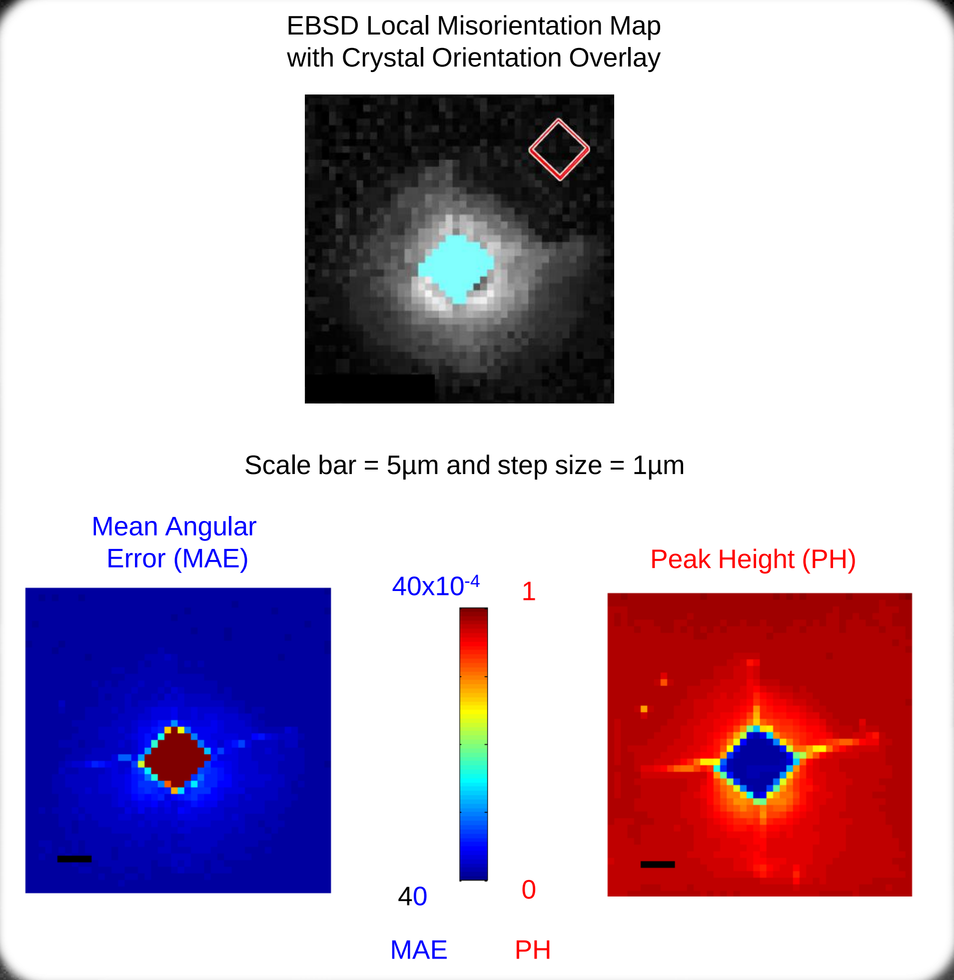
Deformation gradient → $F = A + I = U\Sigma V^*$ ← Displacement gradient (A)

$$\varepsilon = \frac{1}{2}(F^T.F - I)$$

Finite rotation → $R = UV^*$

Error Metrics* - measure precision

- Mean Angular Error (MAE)
  - Solve an over determined problem (shifts → deformation)
    - Only 4x regions of interest (ROI) needed
    - Typically use 20+ ROI
  - Tests how well shifts + remapping 'fit' a deformation gradient
  - Need a value < strains of interest
  - Low values can hide systematic errors
- Measure correlation peak height (PH)
  - Normalise with 1 (autocorrelation) and 0 = no correlation
  - Calculate geometric mean → 1 bad ROI reduces PH strongly
  - Typically values >0.3 are 'ok' (higher is better!)



*e.g. as found in CrossCourt, but not all HR-EBSD tools

Reference selection

- HR-EBSD measures difference in strain + orientation between test & reference pattern

- Reference within same grain
  & same sample
  & microscope cannot be disturbed

  - 1μm of misalignment = 1/20th pixel = 1×10^{-4} in strain error

- Simulations as reference patterns requires

  great patterns → dynamical patterns likely ok (e.g. EM Soft + Dynamics)
  & great knowledge of pattern centre → still not good enough
  & great knowledge of camera optics → not well tackled (yet?)

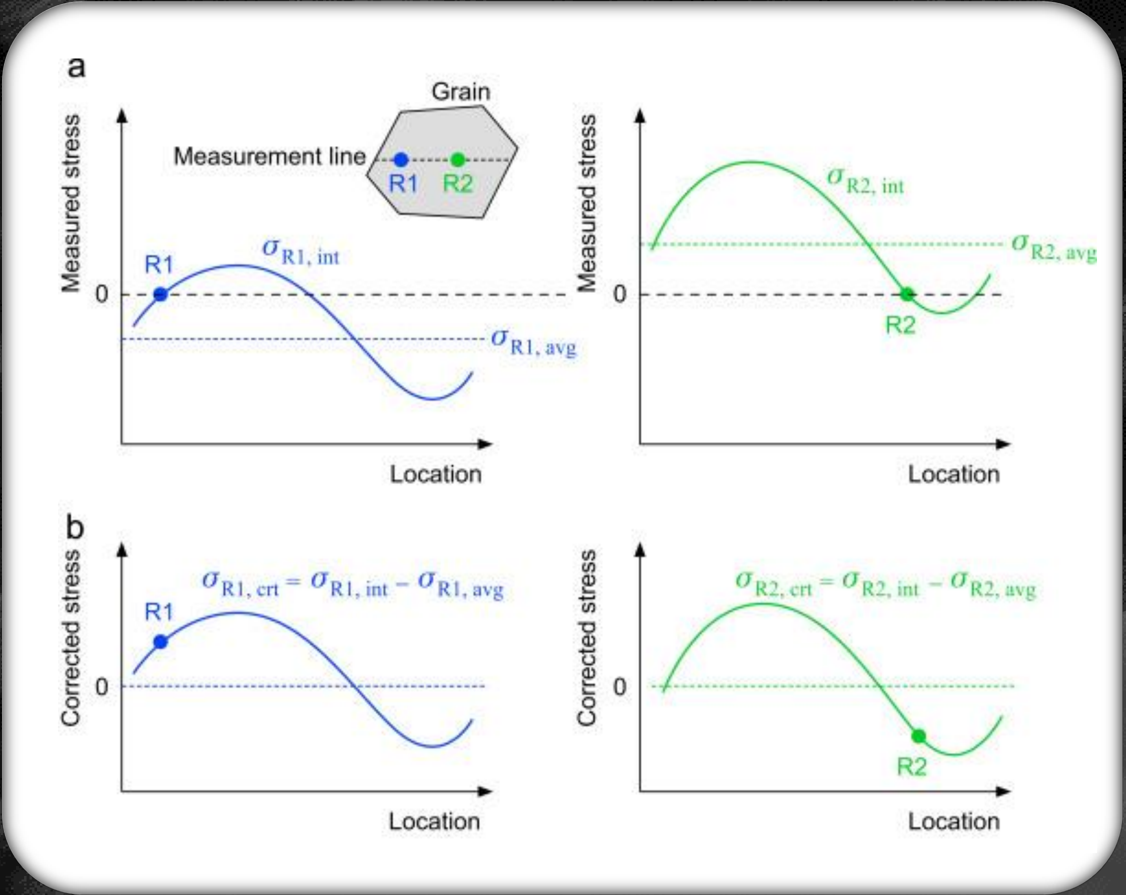
*see Britton et al. (2010) Ultramicroscopy for challenges to be overcome

Reference selection

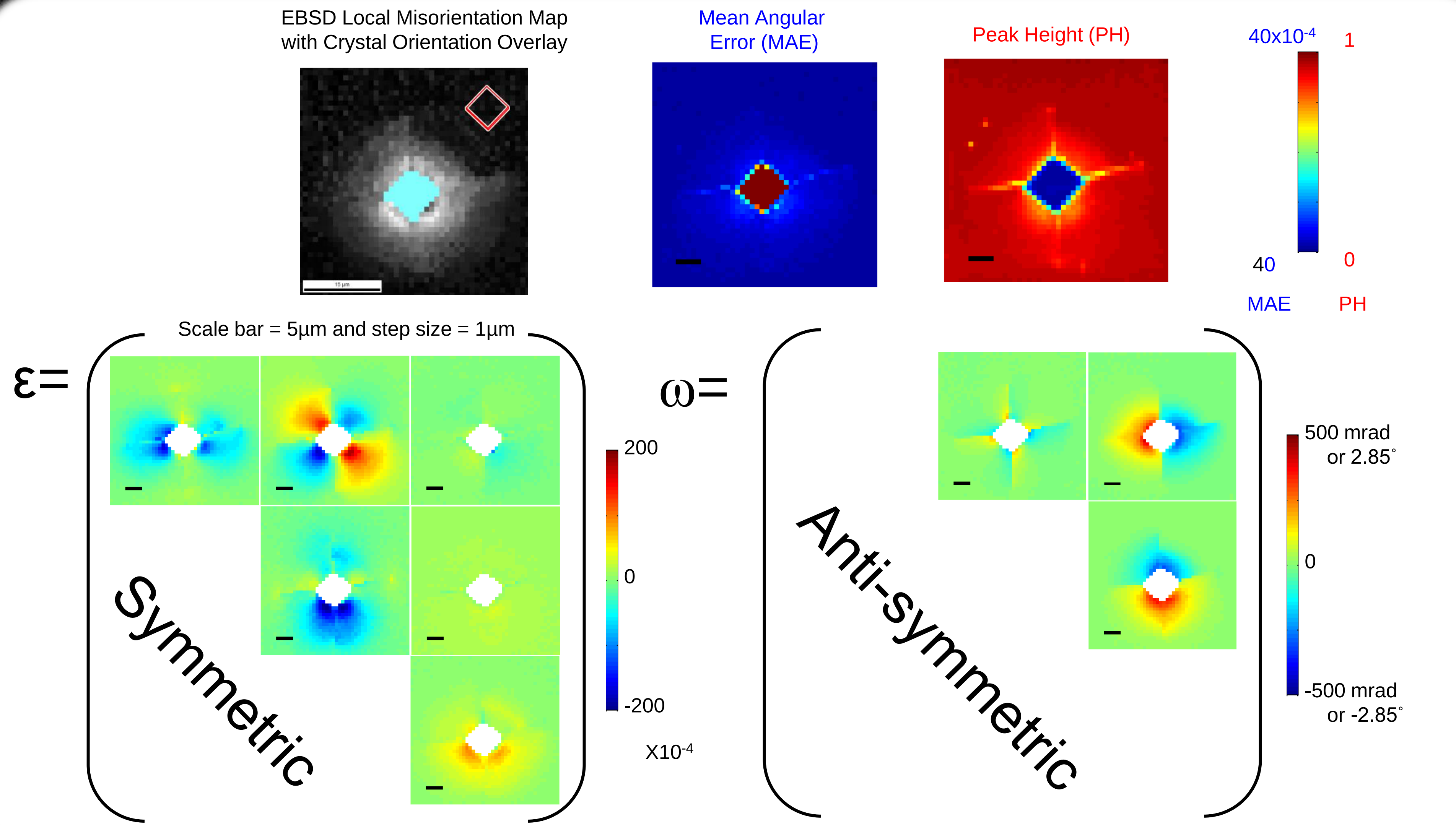
- Choose something clear (single pattern, crisp)
- Precise location does not matter
- Relative strain + rotation measured
- Can 're-zero' as needed

- Reference selection for GND measurements not important
  - Local curvature

Illustration of the effect of reference pattern selection
From Mikami et al. (2015) *Mat Sci Eng A*



Example – Si indentation



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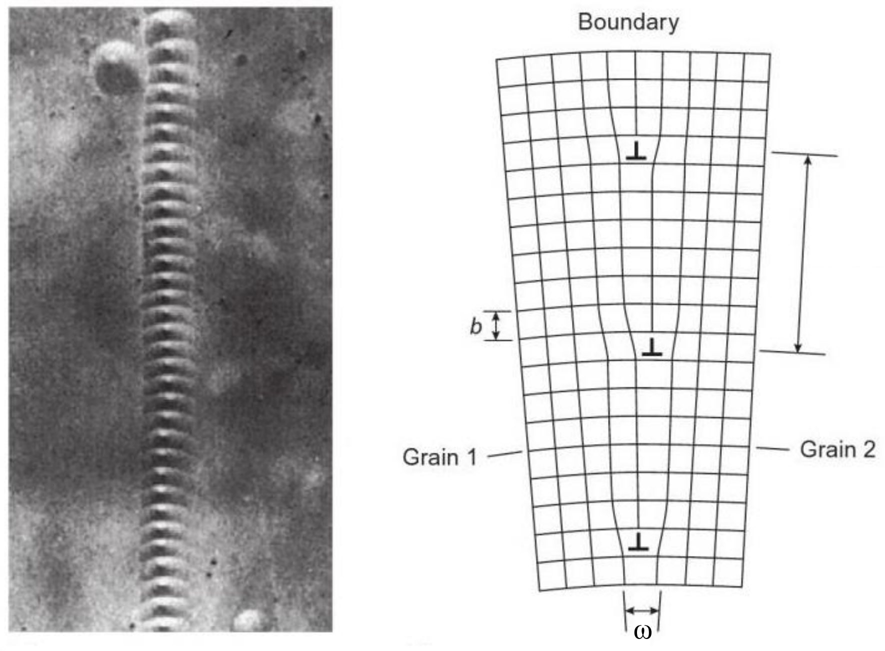
Closure, Nye Tensor and GNDs

- In a deformed crystal (w/o cracks) the displacement field is continuous
- Therefore: $curl(F^e) = -curl(F^p)$
- Evaluate $curl(F^e)$ to understand some plastic strain gradients
- Field of GND analysis – using the Nye tensor [1]

[1] Nye in Acta Met (1953)

Measuring dislocation content (GNDs)

- Map lattice rotations
- Calculate curvature
  - $K_n = d\omega_{ij}/dx_k$
- Nye's dislocation tensor [1] relates curvature to dislocation content



Etch pits revealing a low angle grain boundary containing an array of geometrically necessary dislocations (GNDs)

[From Hull and Bacon, Introduction to Dislocations]

[1] Nye (1953) *Acta Mat*

Curvatures & Densities

- FCC = 18 dislocation types
  - 6 screw, 12 edge
- (often*) overdetermined problem
- Solve with physically motivated minimisation:
  - use linprog & weight according to line energies

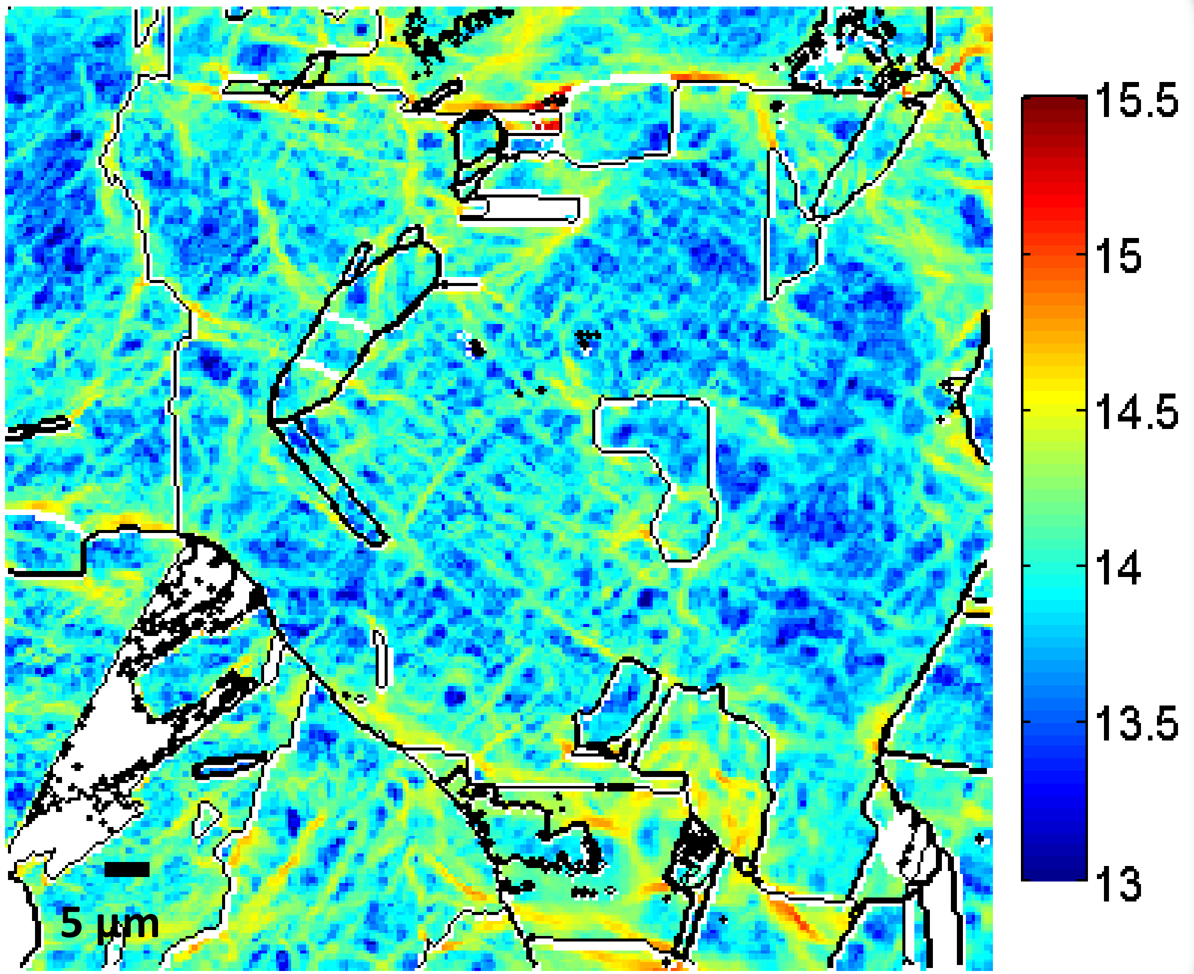
$$\begin{pmatrix} b_1 l_1 - \tfrac{1}{2}\boldsymbol{b}.\boldsymbol{l} & \rightarrow \boldsymbol{S}^{\mathrm{th}} \\ b_1 l_2 & \\ b_1 l_3 & \\ b_2 l_1 & \\ b_2 l_2 - \tfrac{1}{2}\boldsymbol{b}.\boldsymbol{l} & \\ b_2 l_3 & \end{pmatrix} \begin{pmatrix} \rho \\ . \\ \boldsymbol{S}^{\mathrm{th}} \end{pmatrix} = \begin{pmatrix} \partial\omega_{23}/\partial x_1 \\ \partial\omega_{31}/\partial x_1 \\ \partial\omega_{12}/\partial x_1 \\ \partial\omega_{23}/\partial x_2 \\ \partial\omega_{12}/\partial x_2 \\ \partial\omega_{23}/\partial x_2 \end{pmatrix}$$

$$A \begin{pmatrix} \rho \\ . \\ \boldsymbol{S}^{\mathrm{th}} \end{pmatrix} = K$$

Except in low symmetry materials, like rocks – see Wallis et al. (2016) *Ultramicroscopy*

Example map

- Cu – 2% plastic strain
- Reveals cell structure
- Relationship of GND distributions with microstructure…

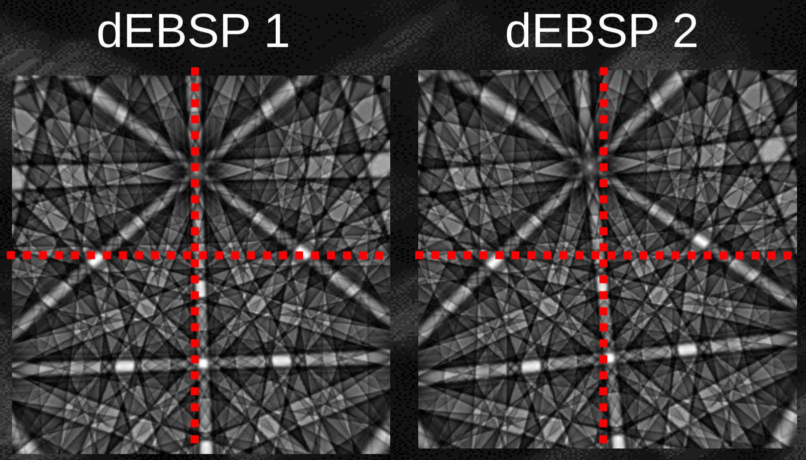


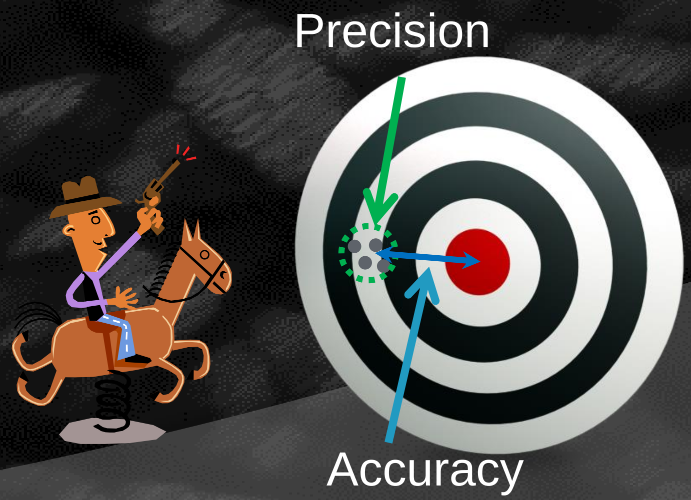
Accuracy, Precision & Sensitivity

Accuracy, Precision and Sensitivity

- Fundamentally image correlation used to compare diffraction patterns and extract ‘high quality’ data

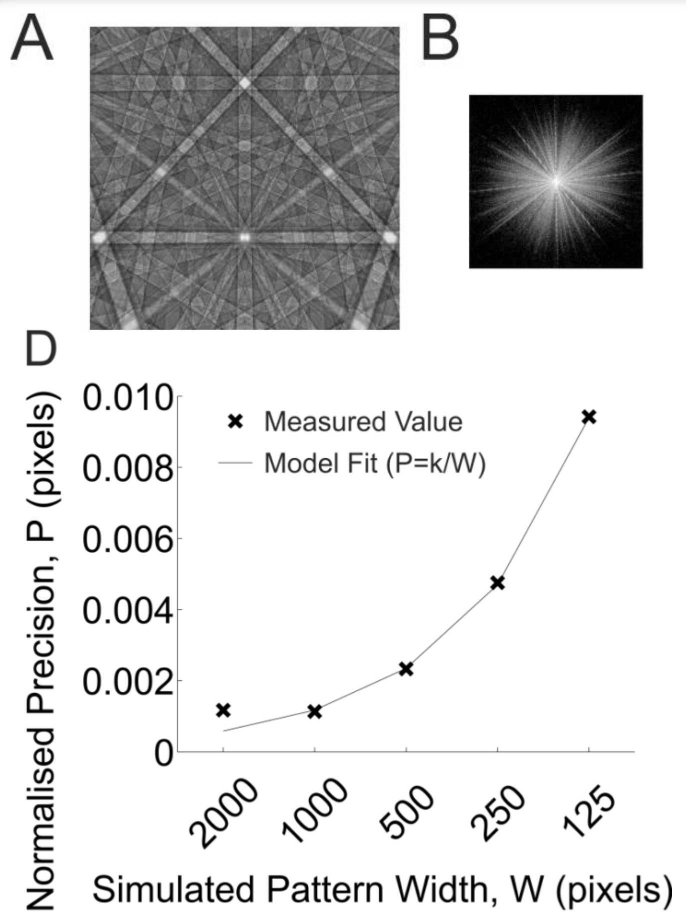
  - Precision
    – ability to recover same result many times
  - Accuracy
    – ability to recover correct answer
  - Sensitivity
    – what sort of changes can we observe





Measuring Precision – Dynamical Simulations

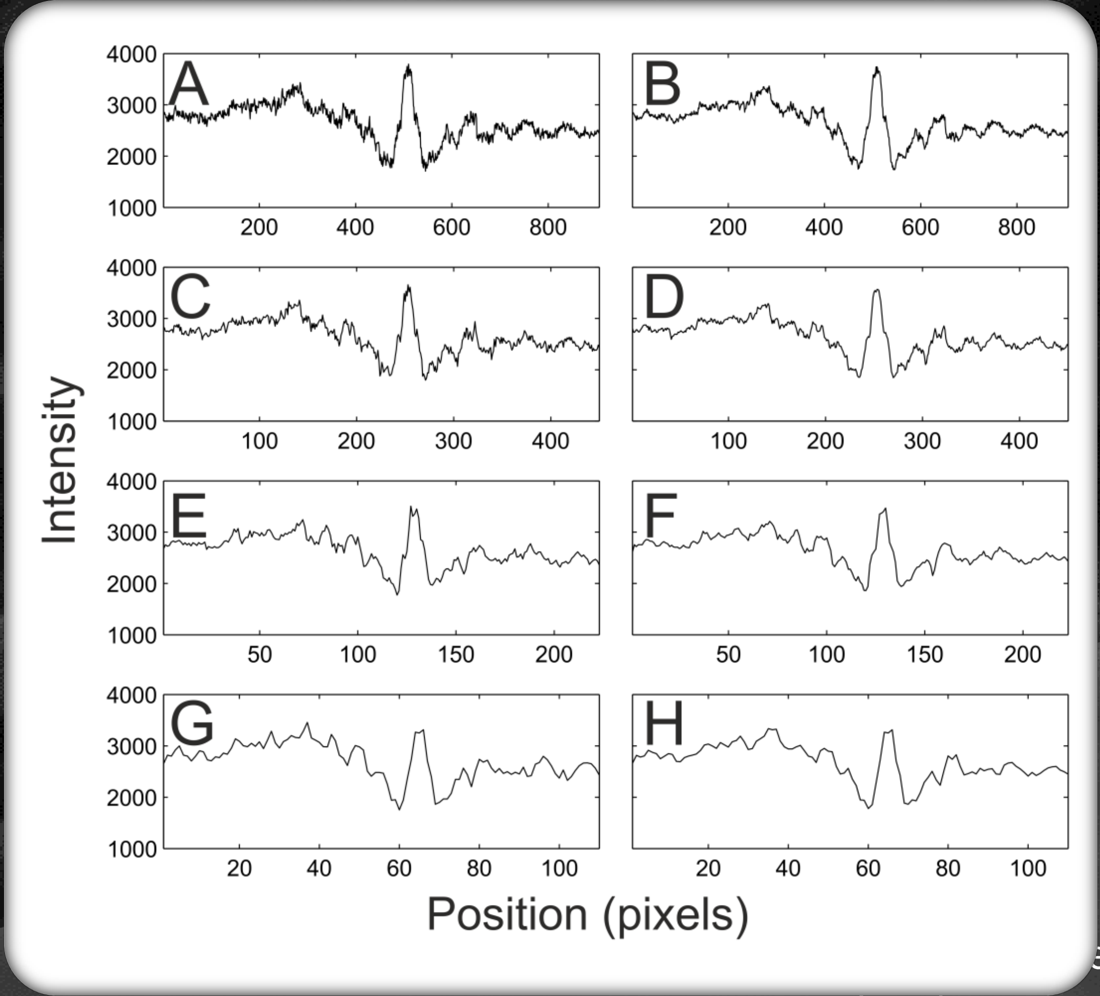
- Generate a high quality 4MG simulation
  - ecpdist courtesy of Dr Aimo Winklemann
- Beam shift virtually
- Bin the image
- Measure (normalised) precision for each binning level



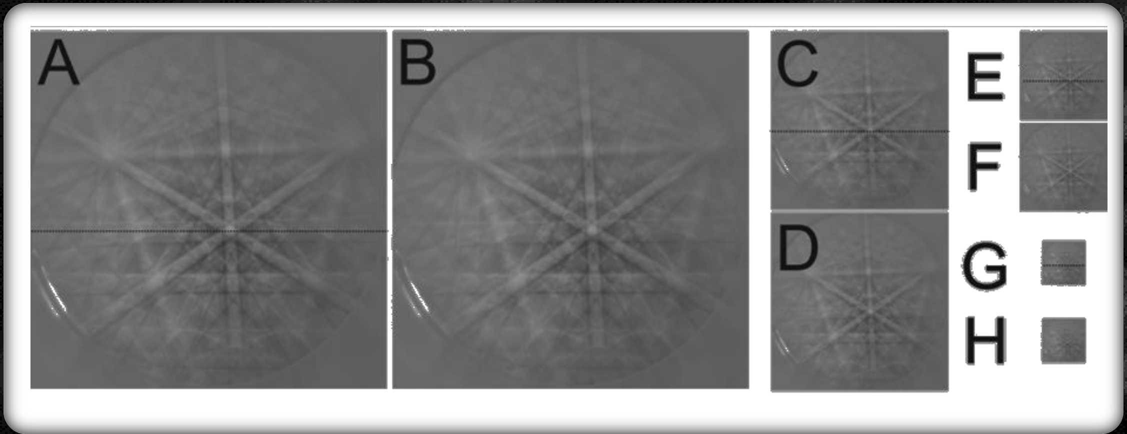
Capturing Diffraction Patterns

- Variables:
  - Hardware (~fixed)
  - Exposure time/probe current
    - i.e. electron budget
  - Camera binning

One Pattern → Five Patterns



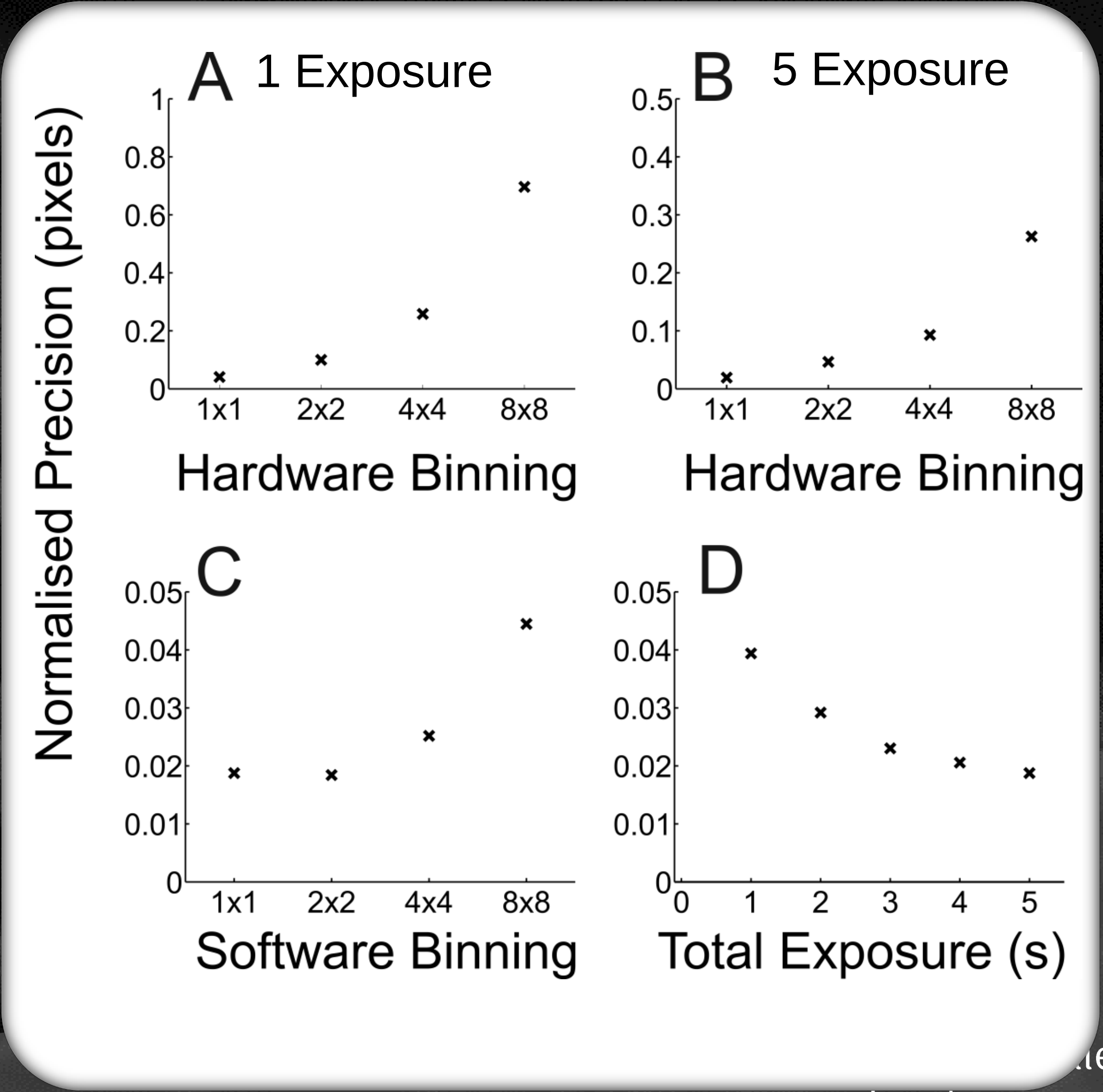
More Binning →



Precision vs budget and binning

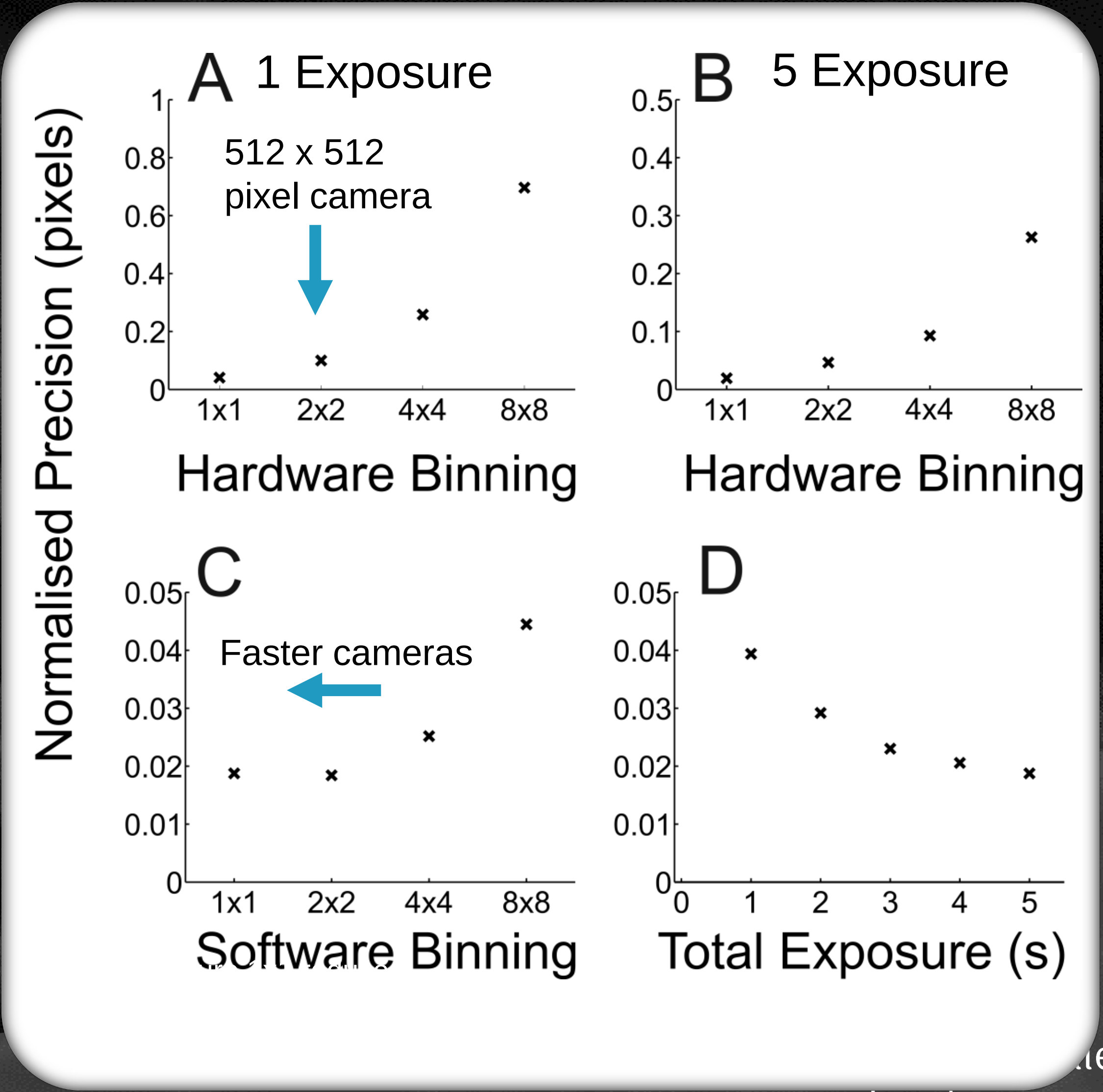
- Measure variation EBSP movement due to beam shift
  - 10 pairs of patterns
  - Different binning (hardware & software)
  - Different exposure times

- Measure precision
  - Averaging standard deviation of 50 ROIs for the difference



Implications

- Sensitivity proportional to precision
- 0.1 normalised pixels in 1000 pixels ~ 1×10^{-4} in sensitivity*
- 2x2 binning, faster exposures or 'fast cameras' ok!
  - Hough resolution:
    - 0.5° = 8 pixels shift
  - Just 0.5 pixels shift sensitivity?
    - 0.02° misorientation resolution!

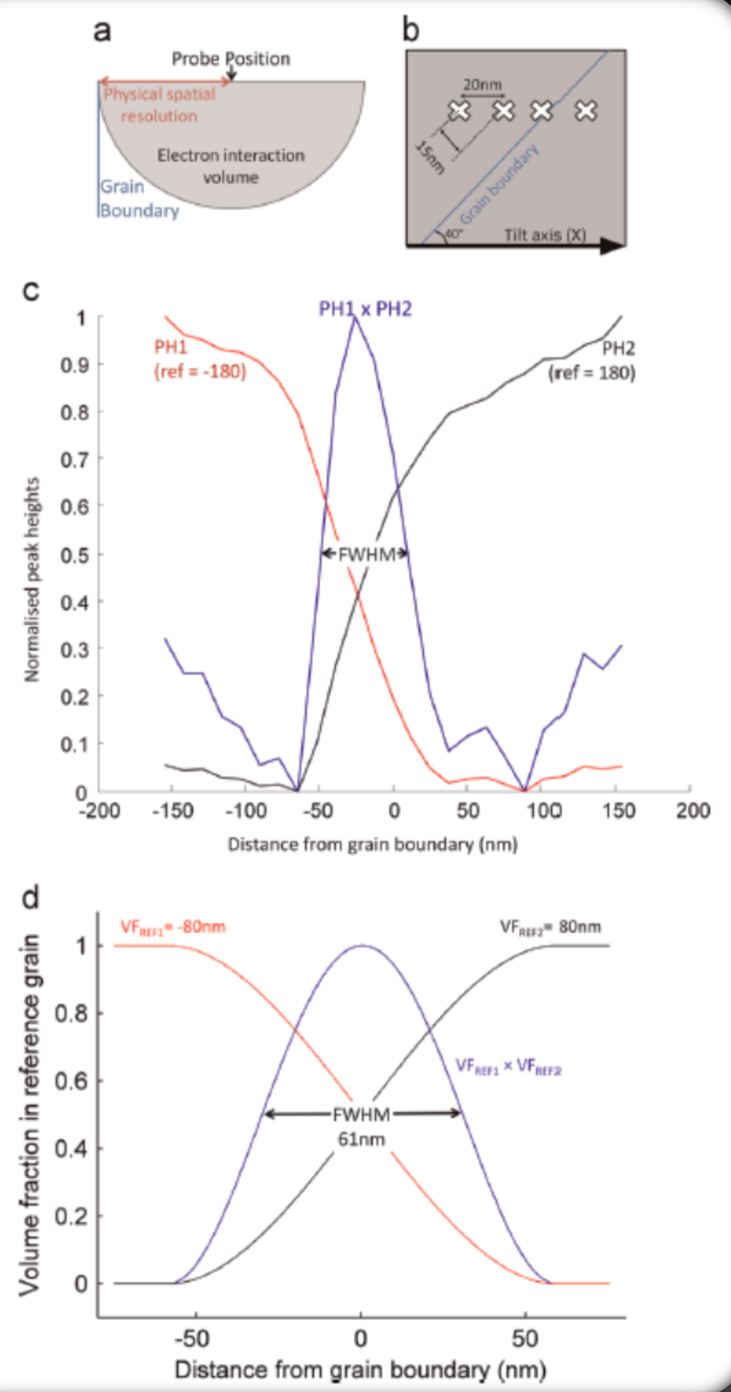


HR-EBSD resolution & precision

- (Effective) spatial resolution – ~typical EBSD
  - c. 20 x 60 x 20 nm^3 → see figure to right [1]

- Typical precision [2,3]
  - ~1x10^{-4} in strain
  - ~1x10^{-4} / 0.006° in rotation
    - vs Hough at 0.8x10^{-2} / 0.5°
    - Up to ~1x10^{-5} reported with great patterns

- Could be better with better hardware… [4]



[1] Tong, Jiang, Wilkinson and Britton (2015) Ultramicroscopy
[1] Wilkinson, Meaden and Dingley (2006) Ultramicroscopy
[2] Britton, Jiang, Karamched, Wilkinson (2013) JOM
[3] Britton et al. (2013) "Assessing the precision of strain measurements using electron backscatter diffraction" – Parts 1 and 2" in *Ultramicroscopy*

HR-EBSD: Remapping

Pattern remapping

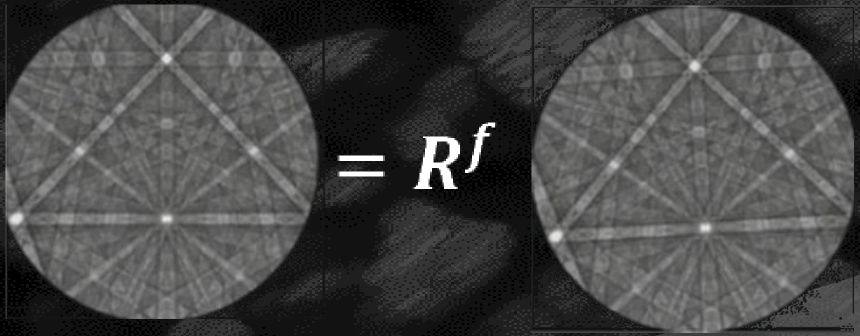
- 3° rotation → ~10s of pixels shifts
- 0.001 strain → ~1s pixel shifts

- Estimate finite rotation matrix, R^f, from measured (infinitesimal) rotations

- Interpolate test pattern into reference orientation
  - Bicubic in matlab (gridfit)

$$R^f = \begin{pmatrix} \cos\omega_{12} & \sin\omega_{12} & 0 \\ -\sin\omega_{12} & \cos\omega_{12} & 0 \\ 0 & 0 & 1 \end{pmatrix} \times \begin{pmatrix} 1 & 0 & 0 \\ 0 & \cos\omega_{23} & \sin\omega_{23} \\ 0 & -\sin\omega_{23} & \cos\omega_{23} \end{pmatrix} \times \begin{pmatrix} \cos\omega_{31} & 0 & -\sin\omega_{31} \\ 0 & 1 & 0 \\ \sin\omega_{31} & 0 & \cos\omega_{31} \end{pmatrix}$$

$$r' = R^f r$$

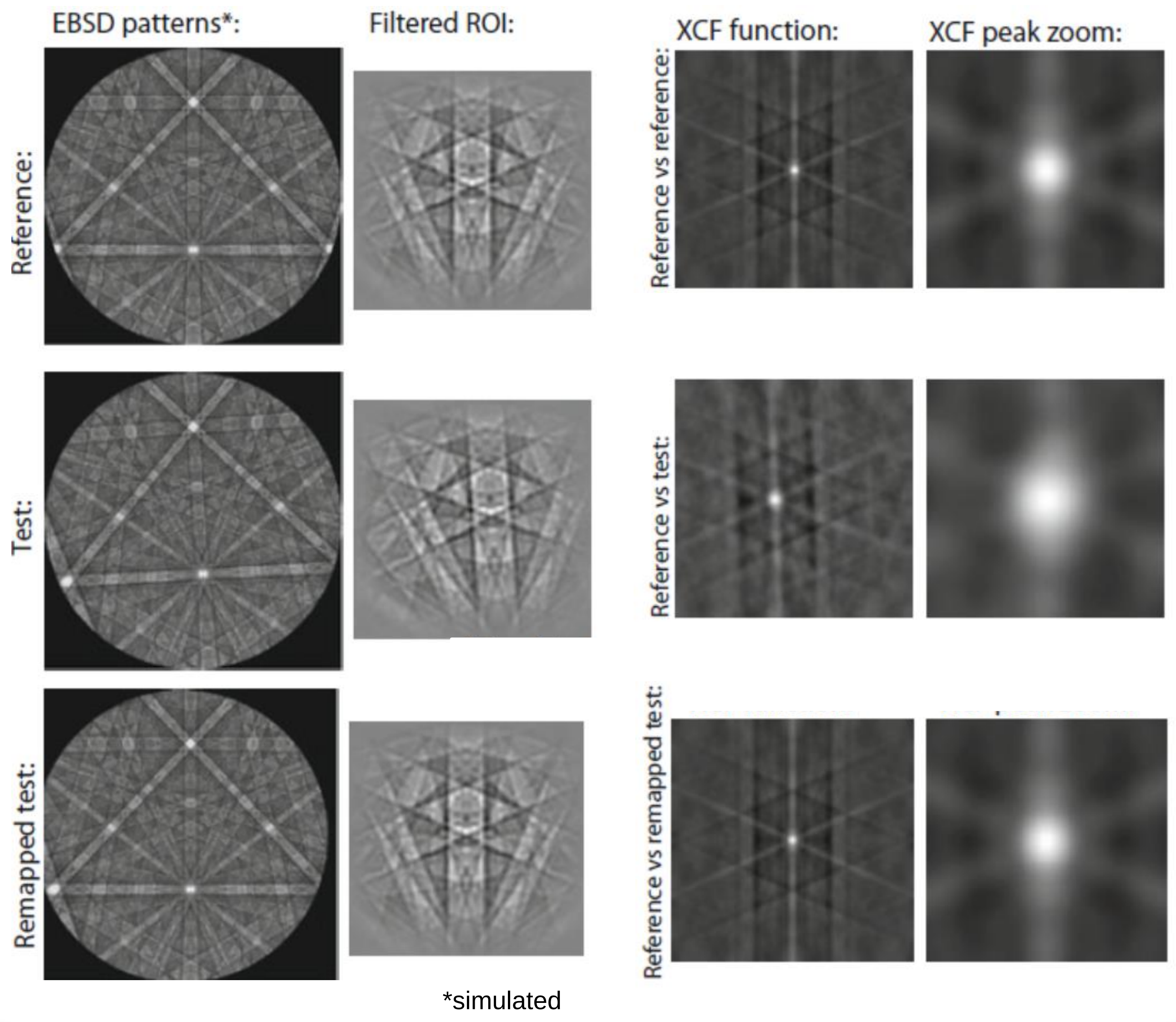


Remapped Test   Test

Figures from Britton, Jiang and Wilkinson (2013) *JOM*

Remapping

- 1st pass measurement of rotation
- Use to remap intensities
  - Need projection info
  - Improve XCF
- Cross correlation for strains + precise rotations
- Recombine the maths in finite deformation framework



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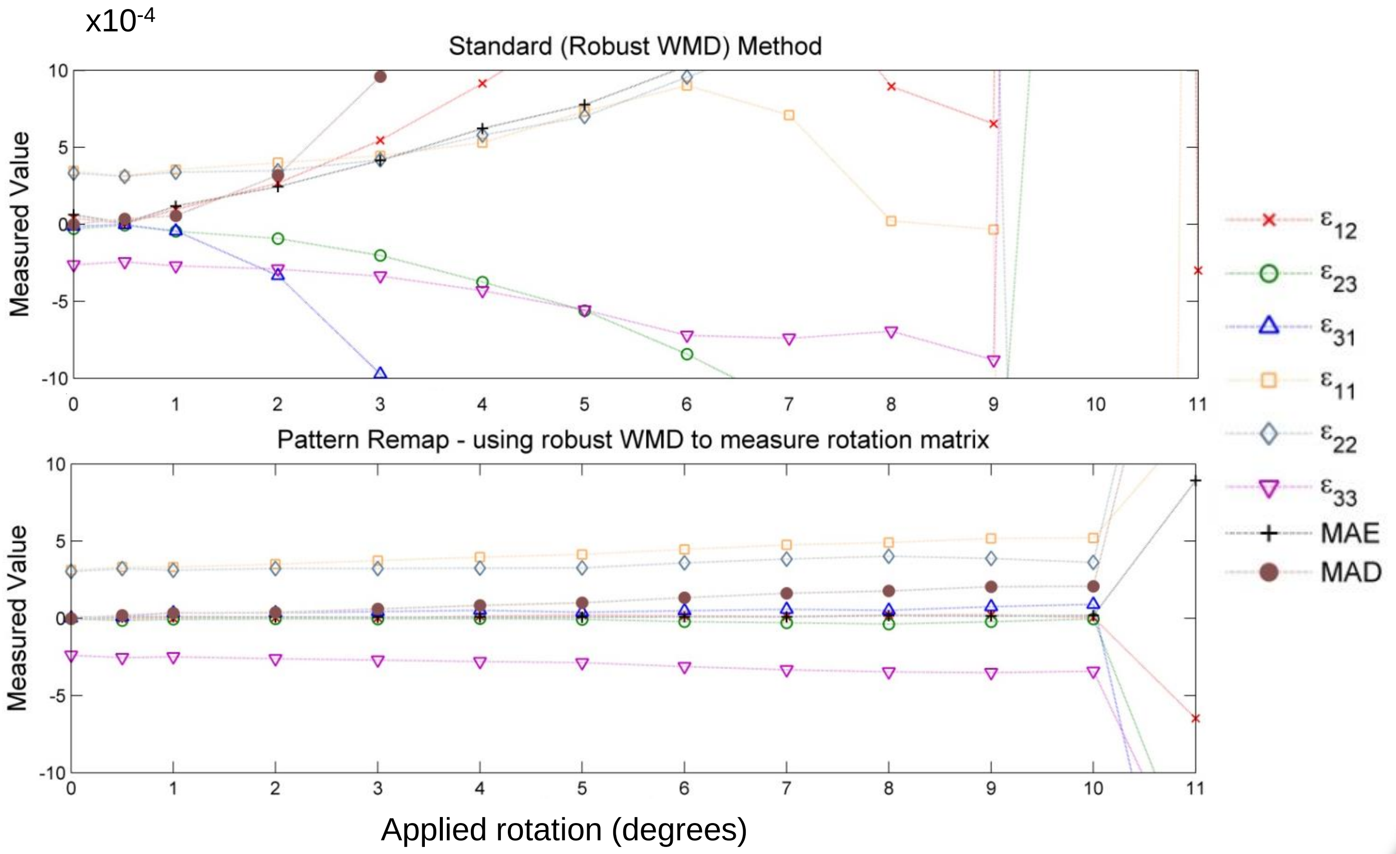
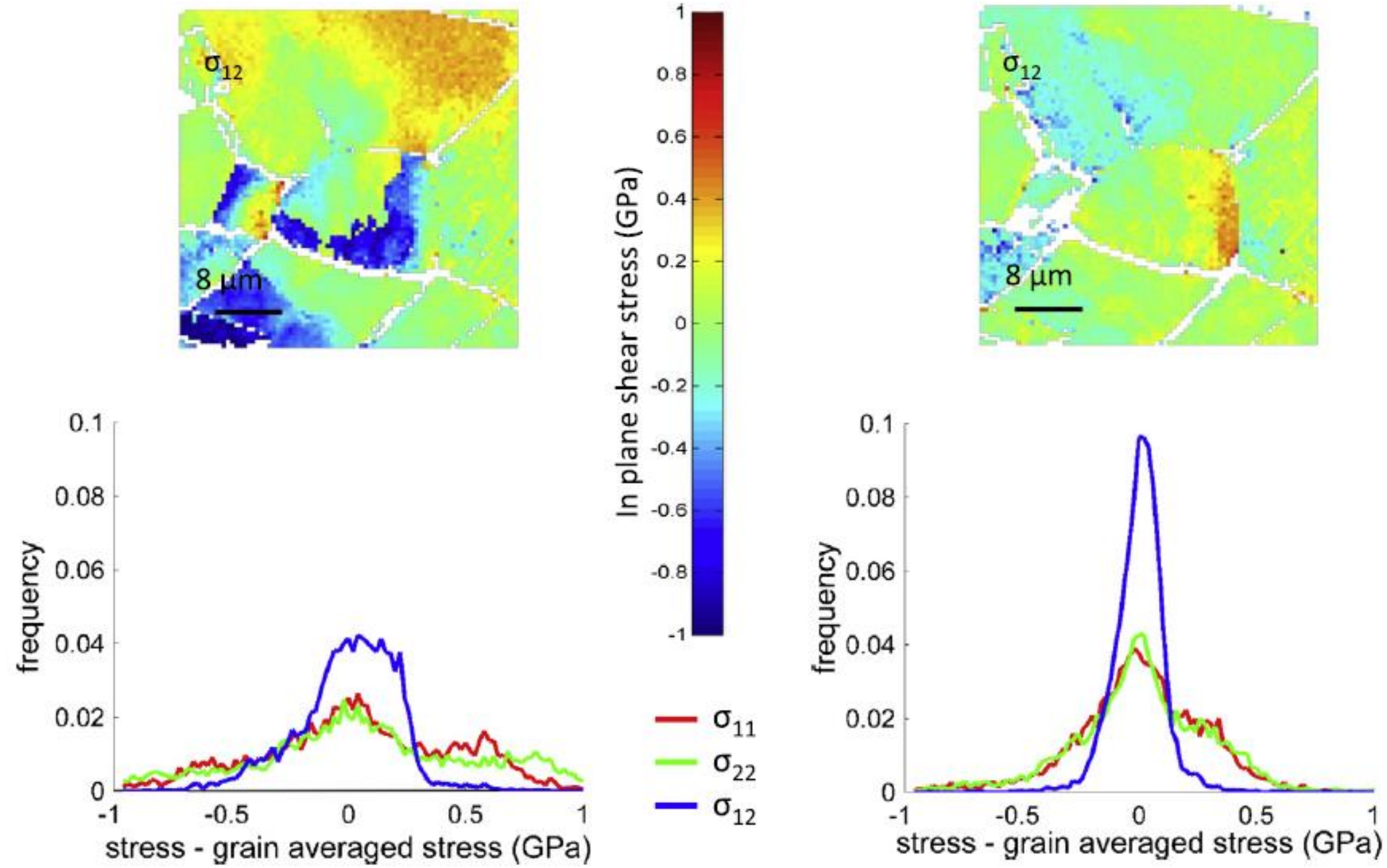
Measuring strains with large rotations

- Comparing simulated patterns (tetragonal distortion)
- Errors introduced when rotations large
- Due to non-translational distortions
- Use pattern remapping to fix
- Needs a good pattern centre measurement!



Wilkinson, A.J., Tarleton, E. Vilalta-Clemente, A., Jiang, J., Britton, T.B. and Collins, D.M. (2014) *PRL*

More reasonable stresses?



(a) first pass (b) second pass

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HR-EBSD: Summary

Summary

- Compare 2+ good patterns … many many times (an embarrassing problem)
- Software evaluates F^e
- Explore residual elastic strain tensor (i.e. stress) within grains
- Measure lattice rotation gradients → enables measurement of GND content via Nye's analysis
  - Only a symptom of part of plastic strain…
- Use of simulations, at present, is a limited to algorithm development*

*see Britton et al. (2010) Ultramicroscopy for challenges to be overcome