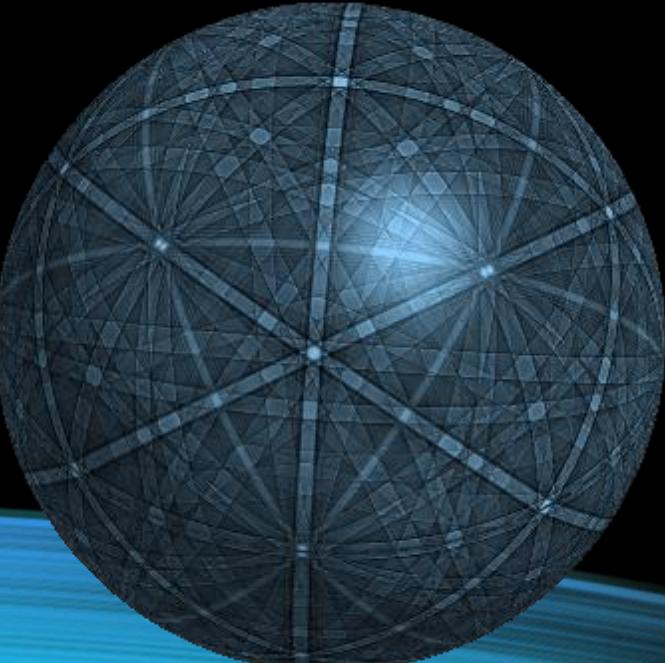


\*W: <http://www.expmicromech.com>; T: @bmatb

# ADVANCING ORIENTATION ANALYSIS



Vivian Tong<sup>1</sup>, Jim Hickey<sup>1</sup>, Euan Wielewski<sup>2</sup>, Jun Jiang<sup>1</sup>, Yi Guo<sup>3</sup>,  
Arantxa Vilalta-Clemente<sup>3</sup>, David Wallis<sup>4</sup>, Lars Hansen<sup>4</sup>,  
Aimo Winkelmann<sup>5</sup>, Angus Wilkinson<sup>3</sup>, and **T.B. Britton\***<sup>1</sup>

1. Department of Materials, Imperial College London, UK
2. Department of Engineering, University of Glasgow, UK
3. Department of Materials, University of Oxford, UK
4. Department of Earth Sciences, University of Oxford, UK
5. Bruker Nano, Berlin, Germany

# BIG QUESTIONS 🤔



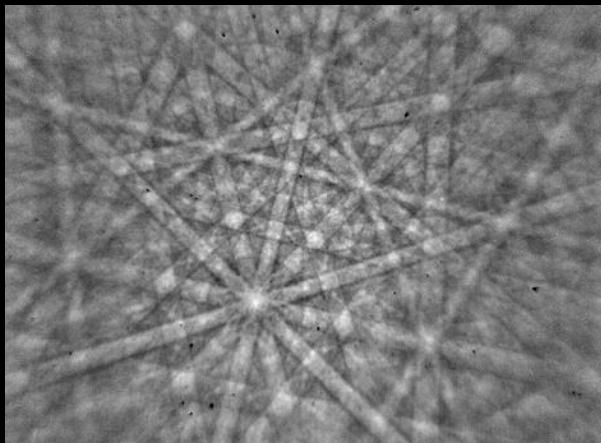
How does it squish/break?  
💪 & ❤️ → 💔



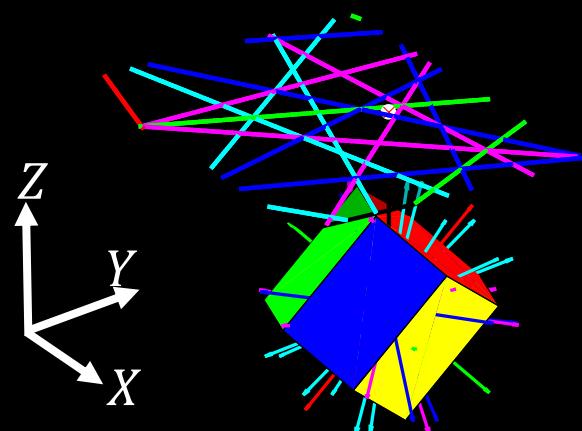
How does it change?  
❤️ & ❤️ ❤️ ❤️ & ❤️ → ❤️ ❤️

# WHAT DOES EBSD MEASURE?

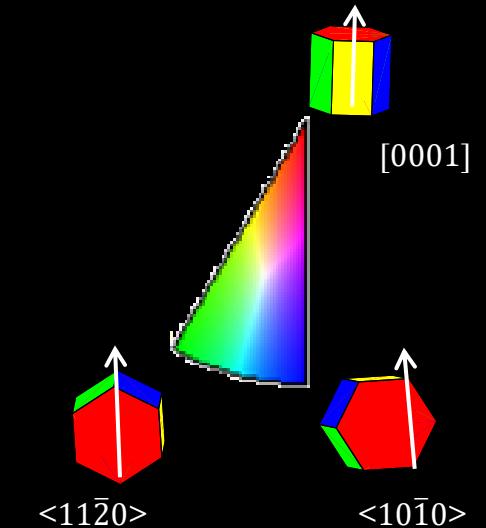
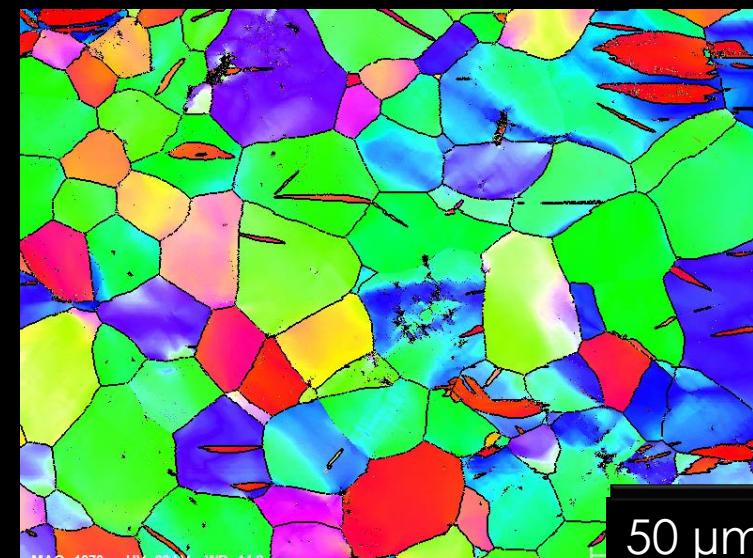
1. **Collect:** Diffraction pattern



2. **Measure:** Crystal information

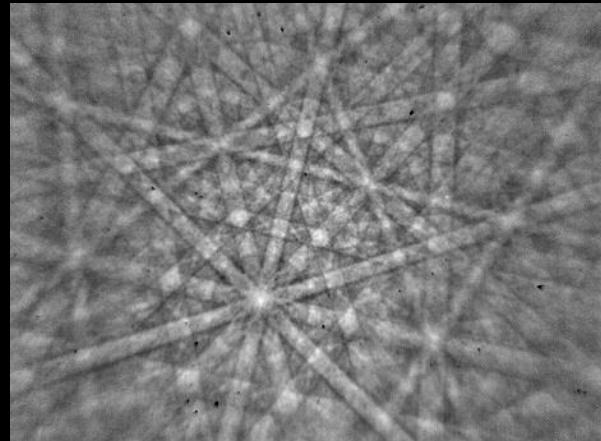


3. **Repeat & create map:**  
Orientations in microstructure



# WHAT DOES EBSD MEASURE?

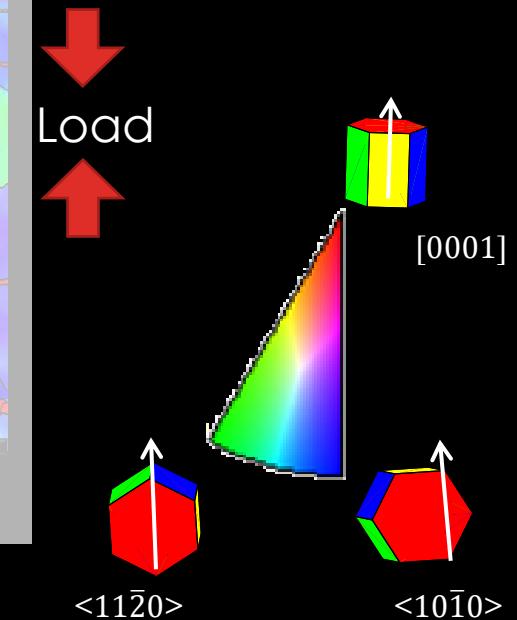
1. **Collect:** Diffraction pattern



2. **Measure:** Crystal orientation

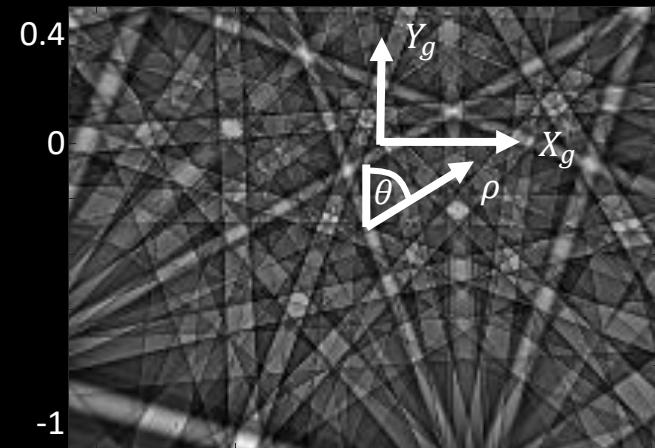


3. **Repeat & create map:**  
Orientations in microstructure

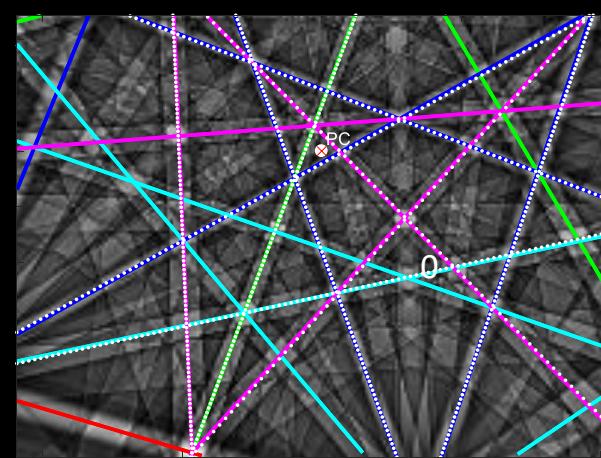


# CONVENTIONAL EBSD

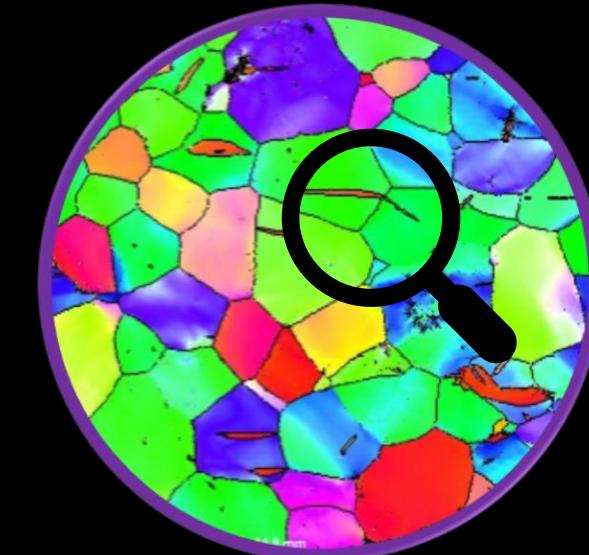
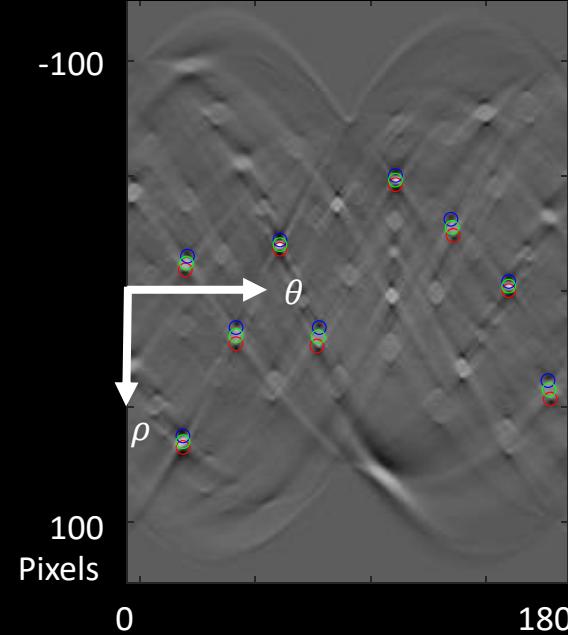
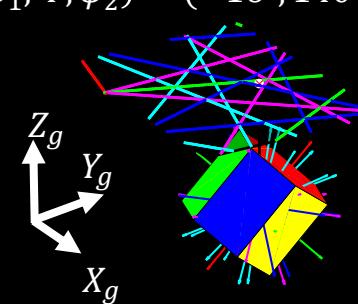
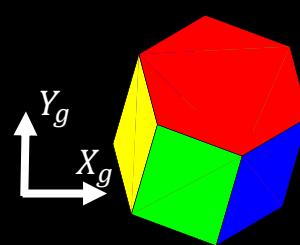
(a) Input Pattern &amp; Coordinate Systems



(c)



(b) Radon Transform, with Peaks

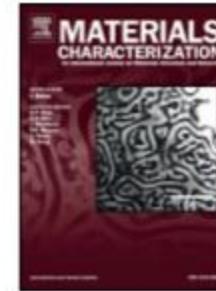
(d) Crystal Orientation:  $(\phi_1, \Phi, \phi_2) = (-16^\circ, 140^\circ, 36^\circ)$   
[in detector frame]



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## Materials Characterization

journal homepage: [www.elsevier.com/locate/matchar](http://www.elsevier.com/locate/matchar)



# Tutorial: Crystal orientations and EBSD – Or which way is up?



T.B. Britton <sup>a,\*</sup>, J. Jiang <sup>a</sup>, Y. Guo <sup>b,1</sup>, A. Vilalta-Clemente <sup>b</sup>, D. Wallis <sup>c</sup>, L.N. Hansen <sup>c</sup>,  
A. Winkelmann <sup>d</sup>, A.J. Wilkinson <sup>b</sup>

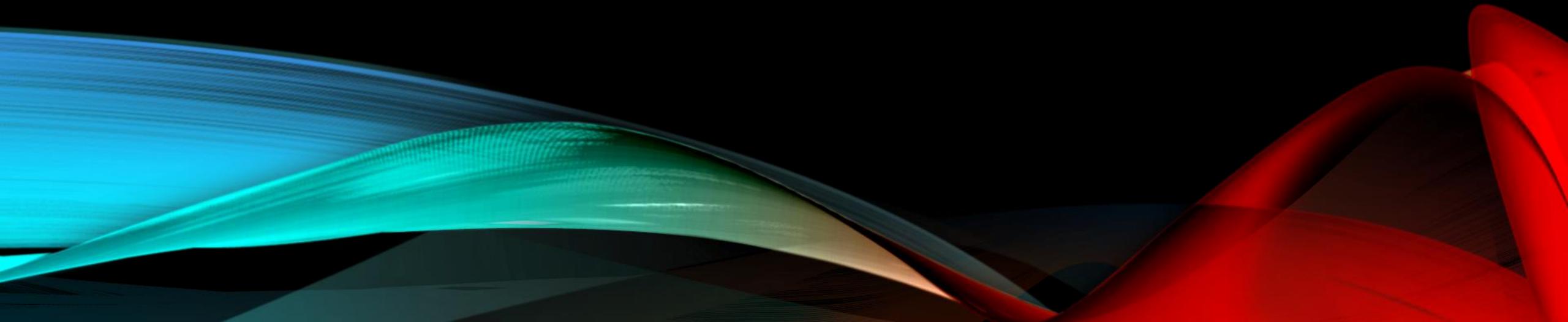
<sup>a</sup> Department of Materials, Imperial College London, Prince Consort Road, SW7 2AZ, United Kingdom

<sup>b</sup> Department of Materials, University of Oxford, Parks Road, OX1 3PH, United Kingdom

<sup>c</sup> Department of Earth Sciences, University of Oxford, South Parks Road, OX1 3AN, United Kingdom

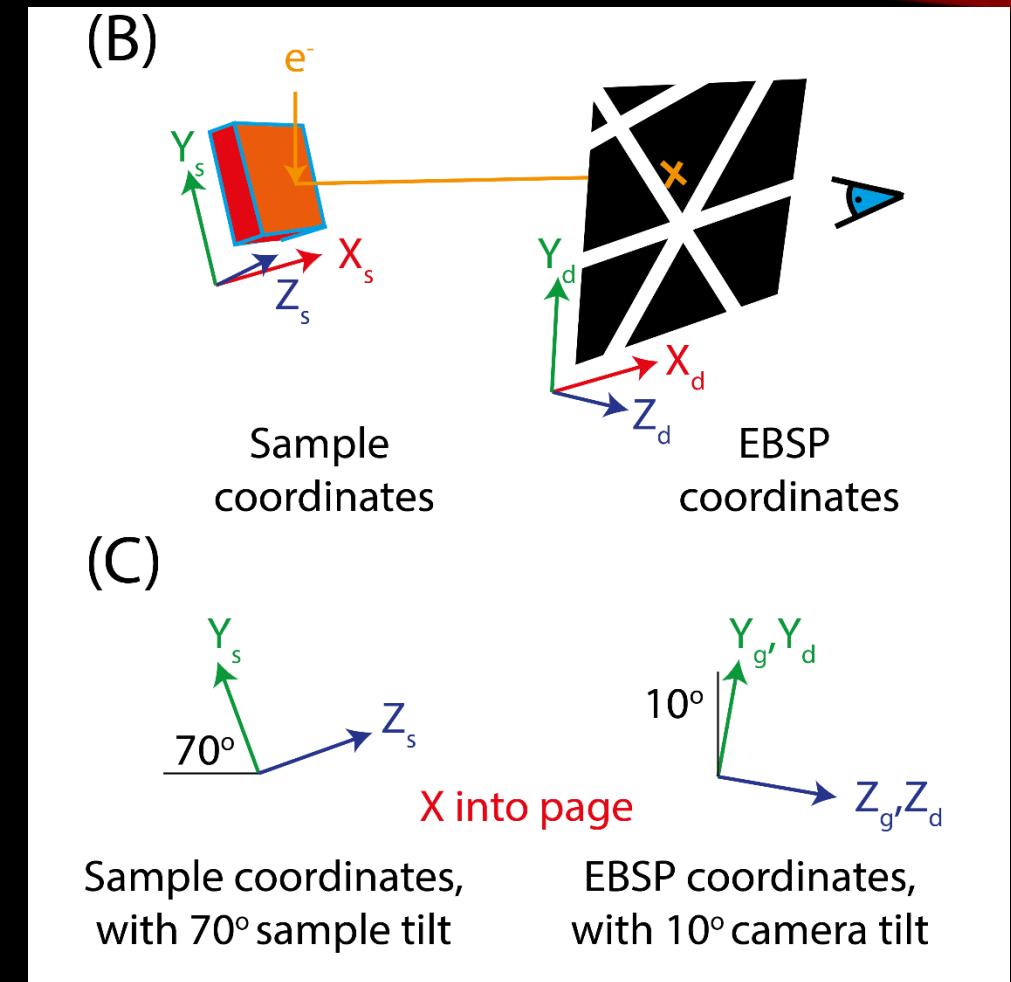
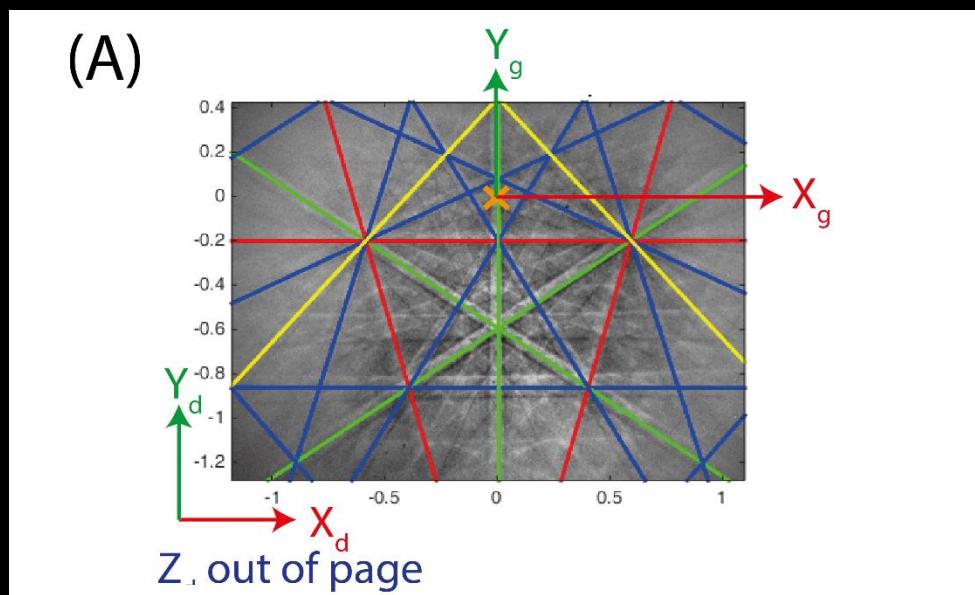
<sup>d</sup> Bruker Nano GmbH, Am Studio 2D, 12489 Berlin, Germany

WARNING: THE FOLLOWING IS SPECIFIC TO  
ONE SYSTEM – THE METHOD IS GENERAL

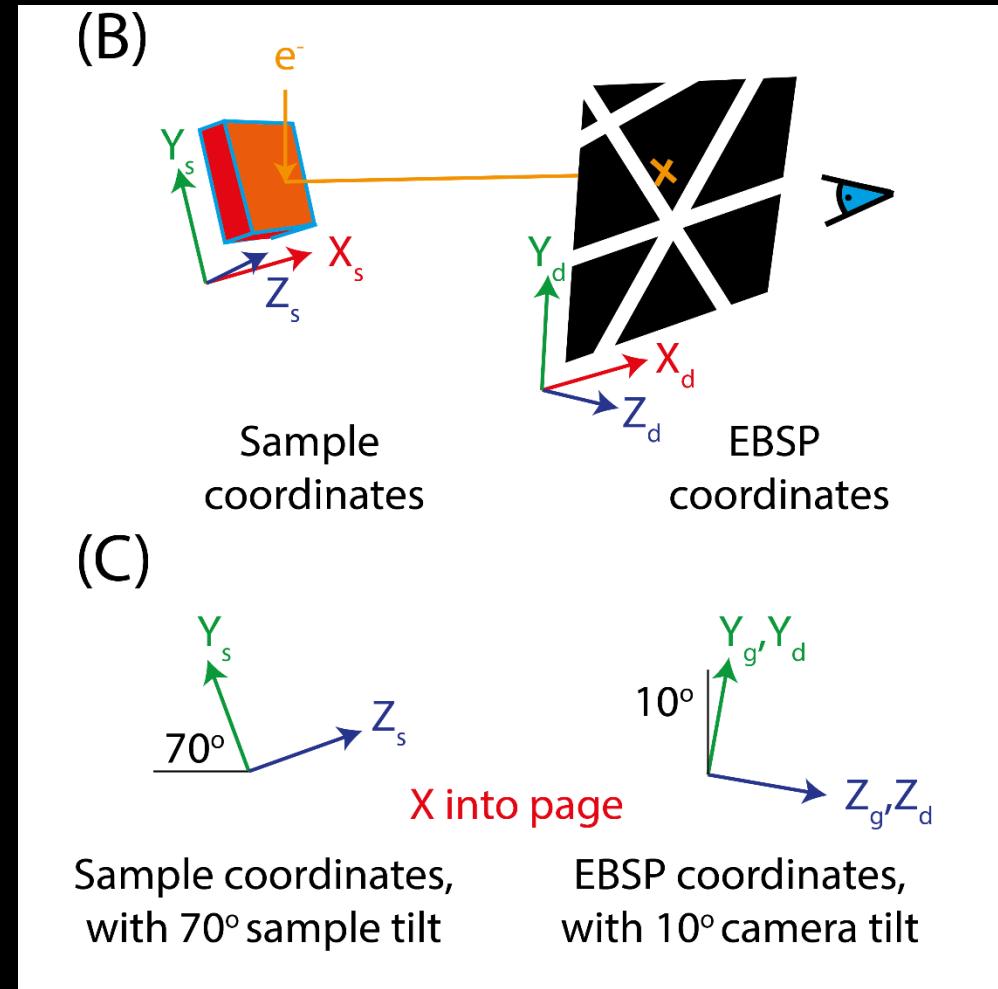
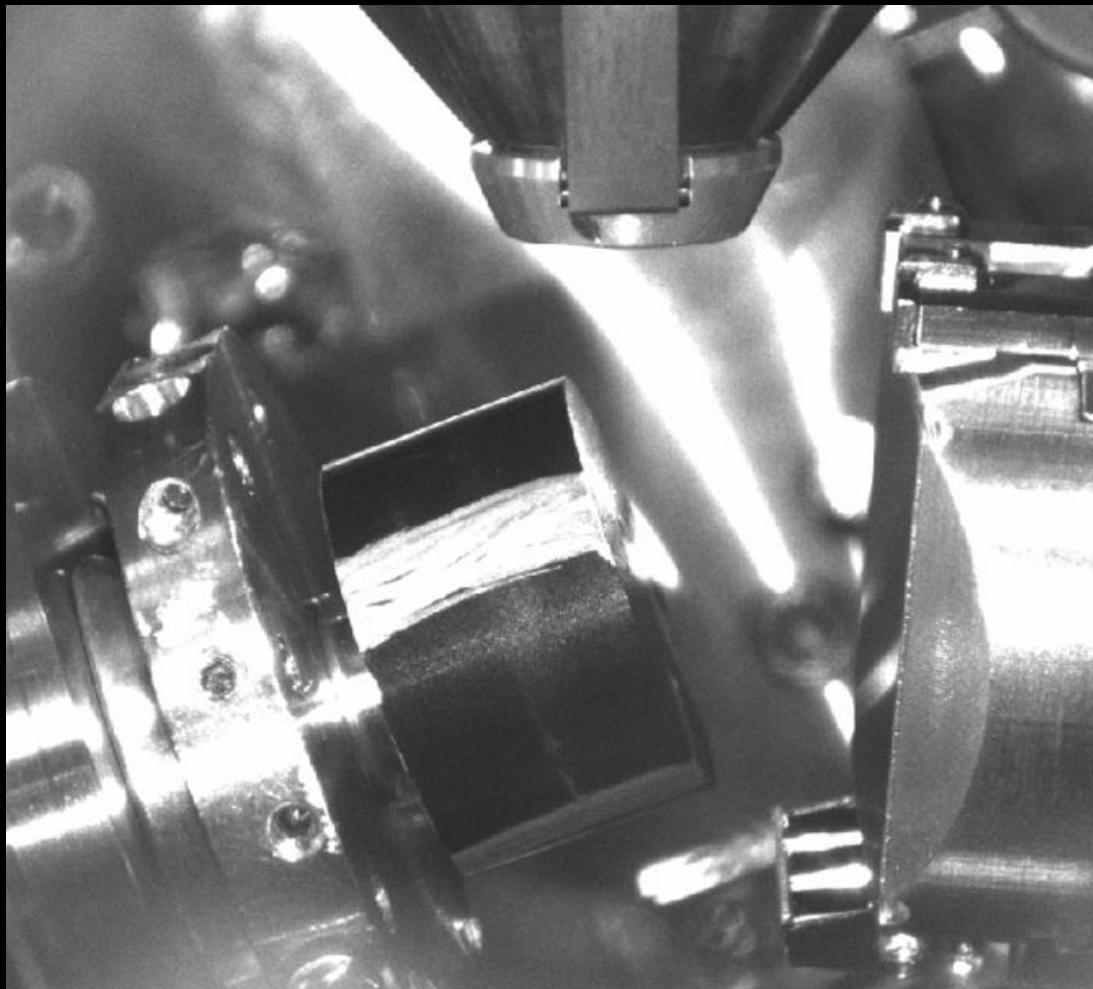


# COORDINATE SYSTEMS

- EBSD has many coordinate systems
  - Diffraction pattern (image, gnomonic)
  - The crystal to diffraction pattern
  - Sample coordinate system
  - Sample to crystal
  - Pole figures, tensors, properties...



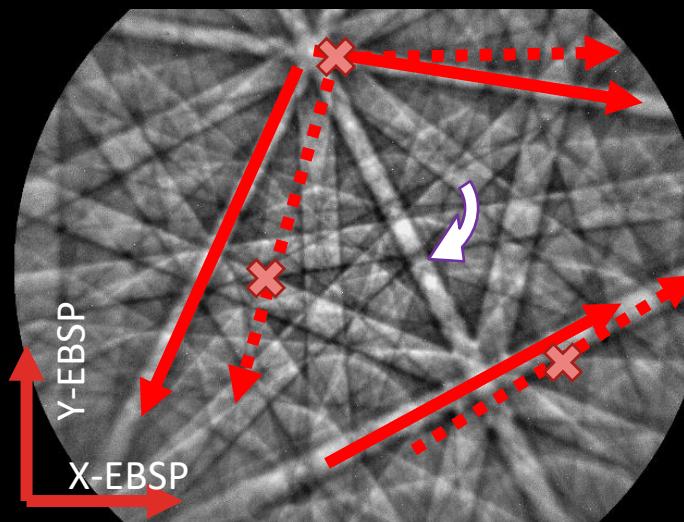
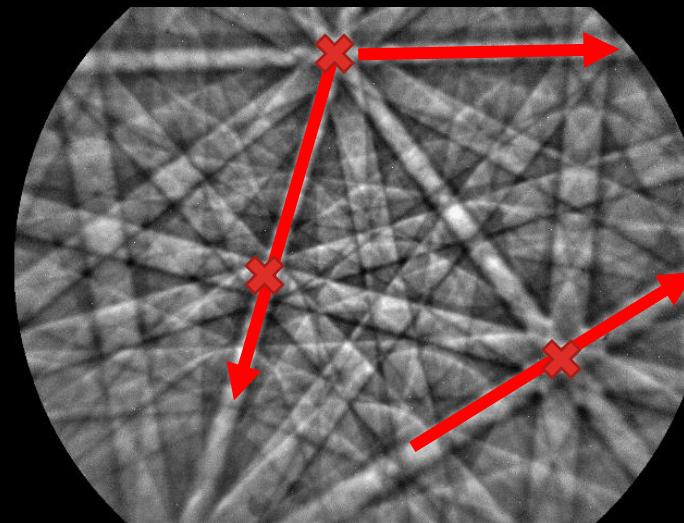
# MICROSCOPE AND EBSD CAMERA



# CONVENTIONS

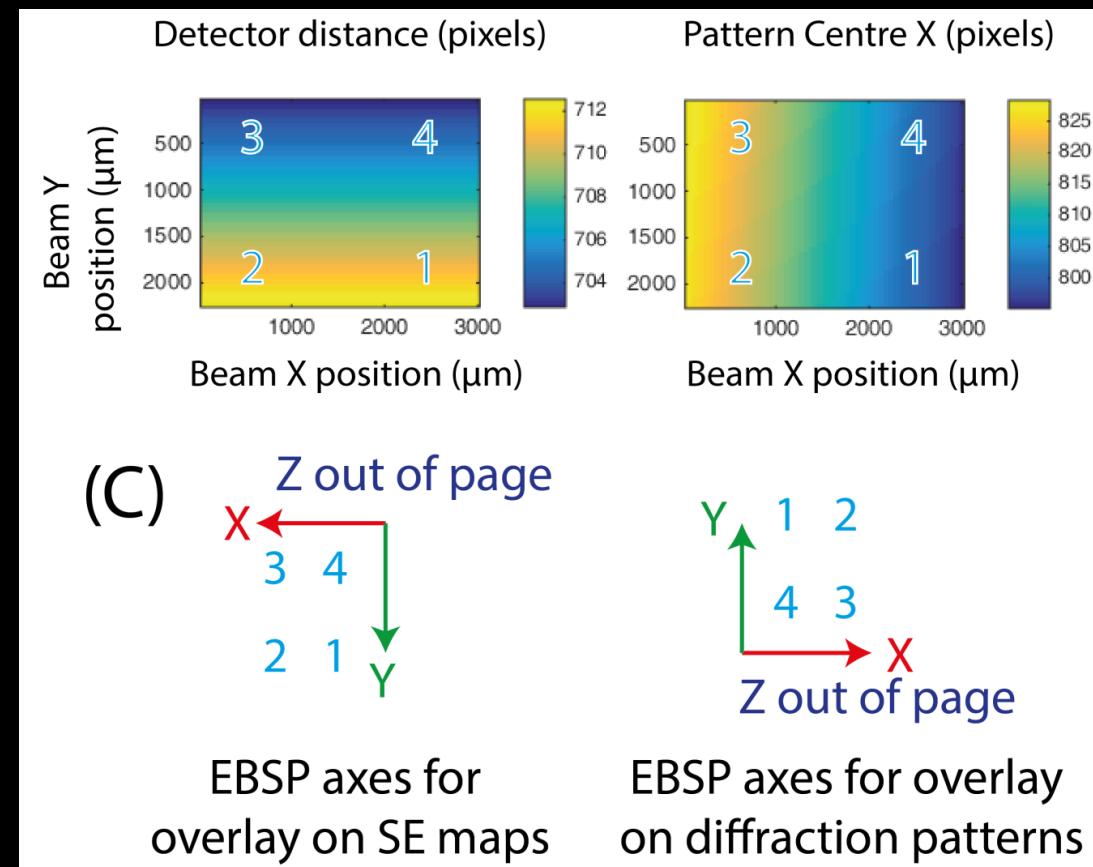
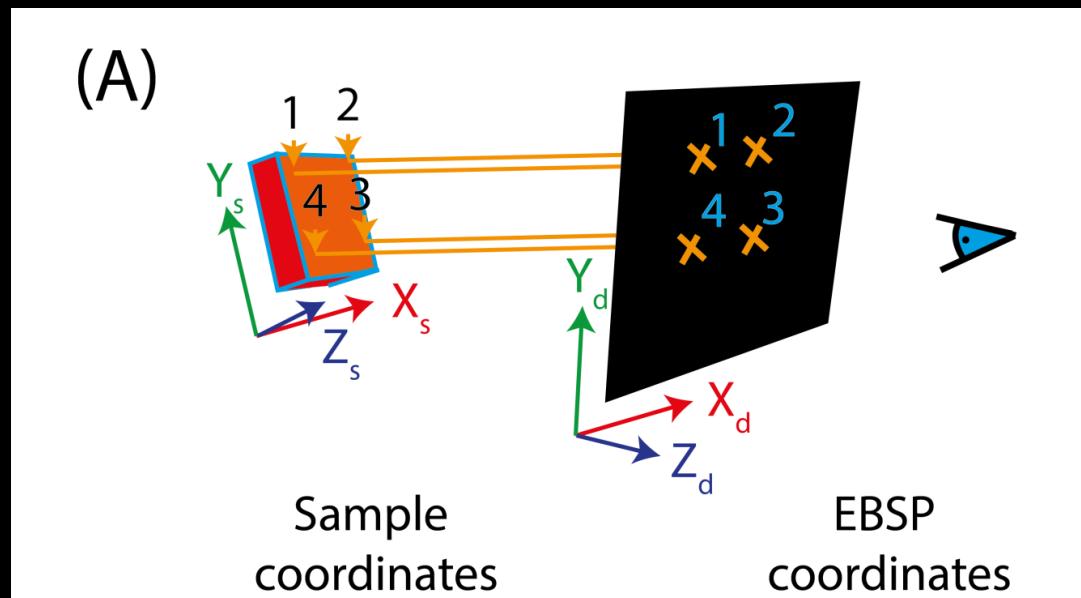
- Z points out of the sample
- The 2D map is orthogonal
- X, Y and Z form a right handed set
- Indexing happens with respect to a reference crystal
  - within the diffraction pattern
  - relate this to the beam scanning
  - can relate to features, i.e. the sample or a grain boundary
- How do we link beam scanning directions – i.e. the mapping settings – to the EBSD pattern?
  - Rotate the sample & see how the pattern + sample change

# ROTATION TEST



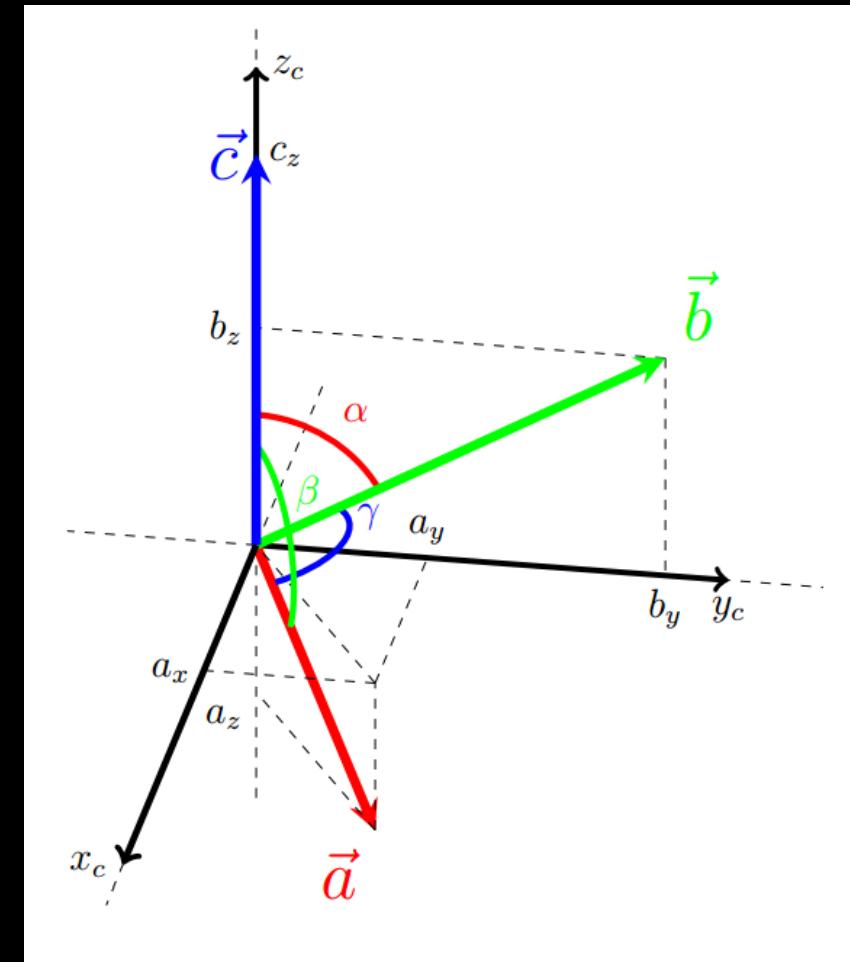
# CHECKING SCANNING

- We can follow the pattern centre movement
  - Pattern centre = shortest distance between sample & detector
  - Use PCx and DD



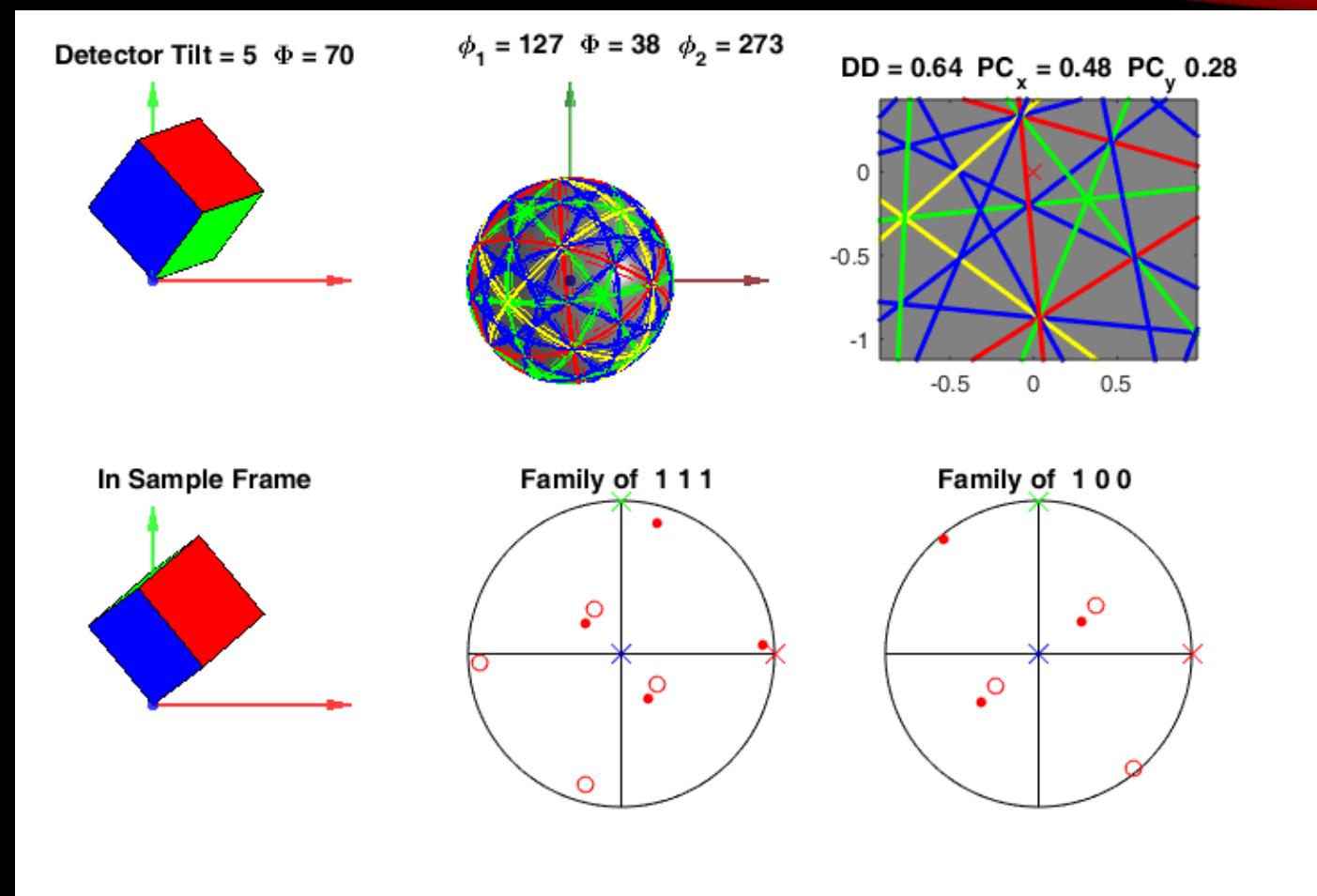
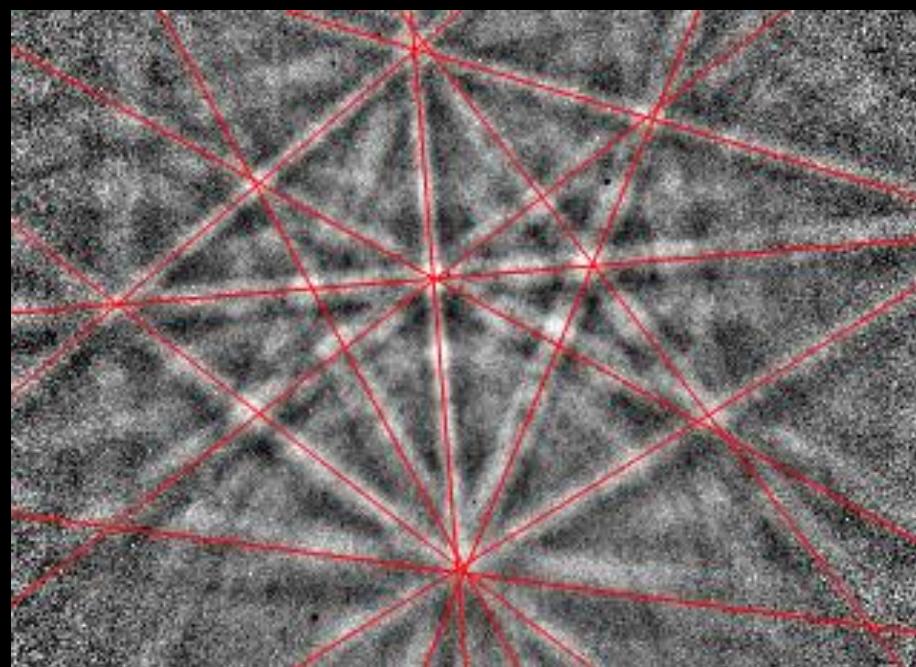
# ESTABLISH A CRYSTAL FRAME

- We need to describe the reference crystal
  - Link a cartesian axis system to the crystal axis system
  - i.e. relationship between X, Y, C and a, b, c (and  $a^*$ ,  $b^*$ ,  $c^*$ )
- In our convention, used by at least one manufacturer
  - **a** **b** and **c** form a right handed set.
  - **c** is parallel to the  $Z_c$  axis.
  - **b** lies in the  $y_c$ - $z_c$  plane, at an angle  $\alpha$  to **c**
  - **a** is pointed such that it is an angle  $\beta$  to **c** and  $\gamma$  to **b**.
- Equations are in the tutorial paper, and python + matlab scripts, and full maths is in the PDF supplementary article



# PATTERN + ORIENTATION CHECK

- X = R, Y = G, Z = B

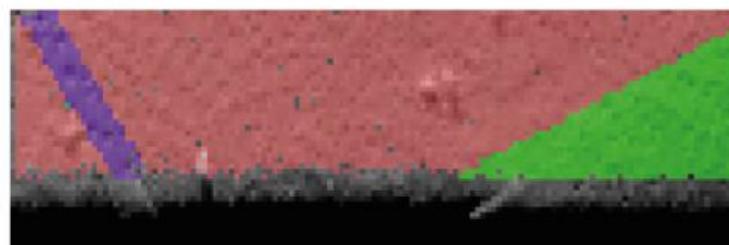


# CRYSTAL CHECKING

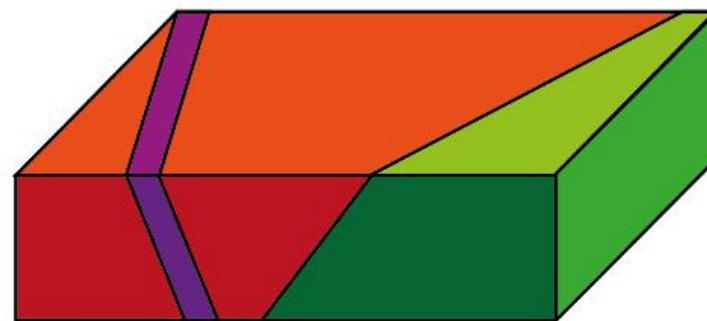
- The 180° rotation ‘problem’ will change the direction of axes out of plane
  - Even in cubic
- Need to have a crystallographic feature we know & can link between the sample + scanning frames, to the EBSD frame
- Use a coherent twin in FCC – 60°{111} twinning plane (ABCBC stacking)
  - Plot the plane trace & use a FIB to look at the subsurface trace
- Also need to check lower symmetry materials
  - does the cartesian to crystal axis work?

# OUT OF PLANE TESTING

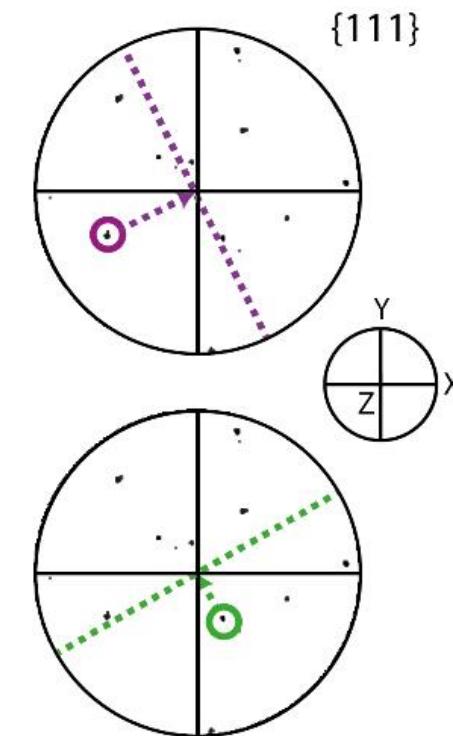
(A) Secondary electron micrograph and IPF Z



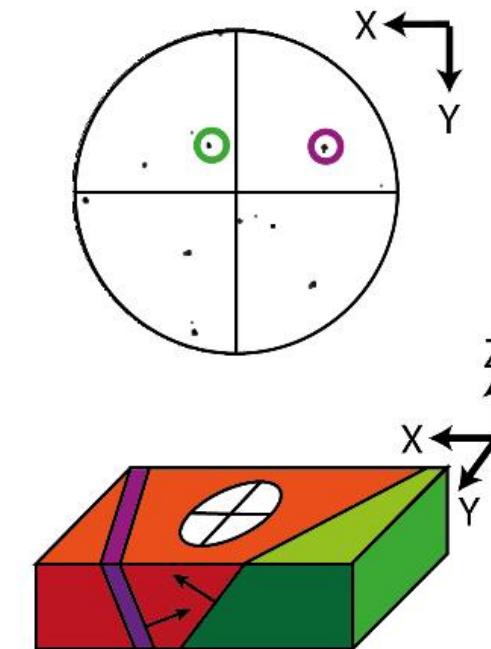
(B) 3D schematic of twinned region



(C) Surface trace analysis

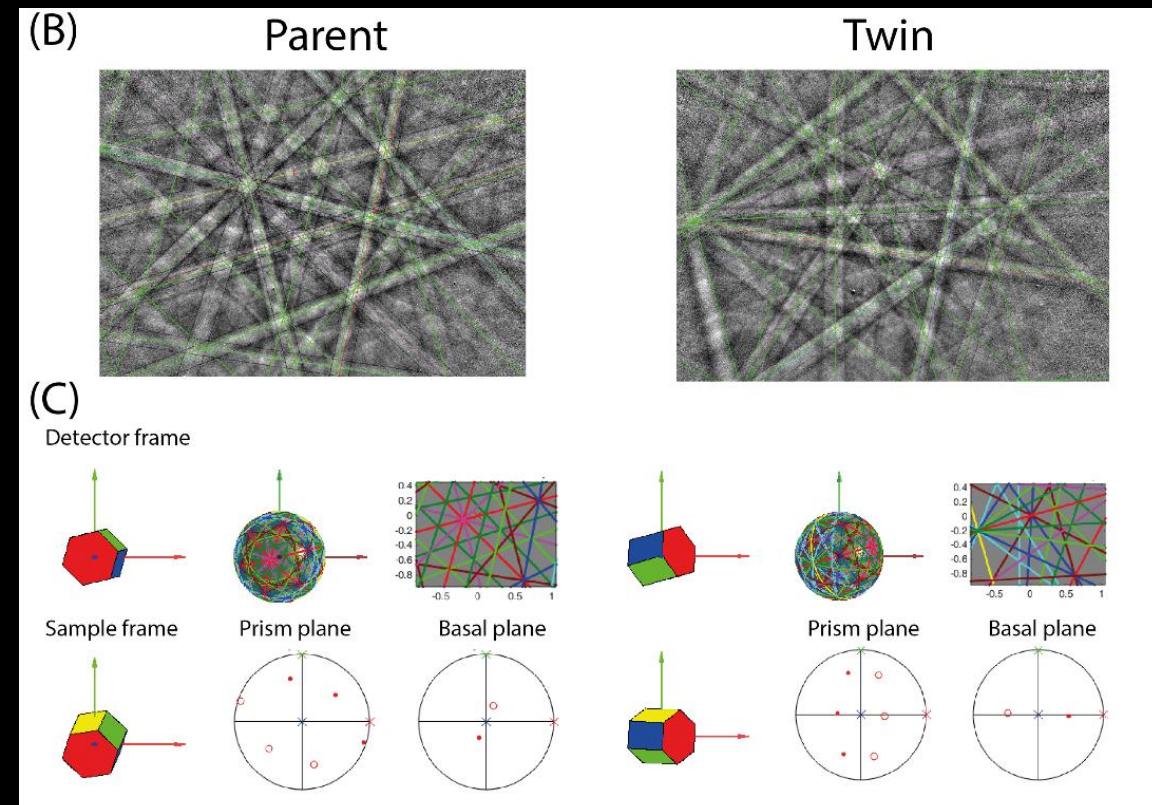
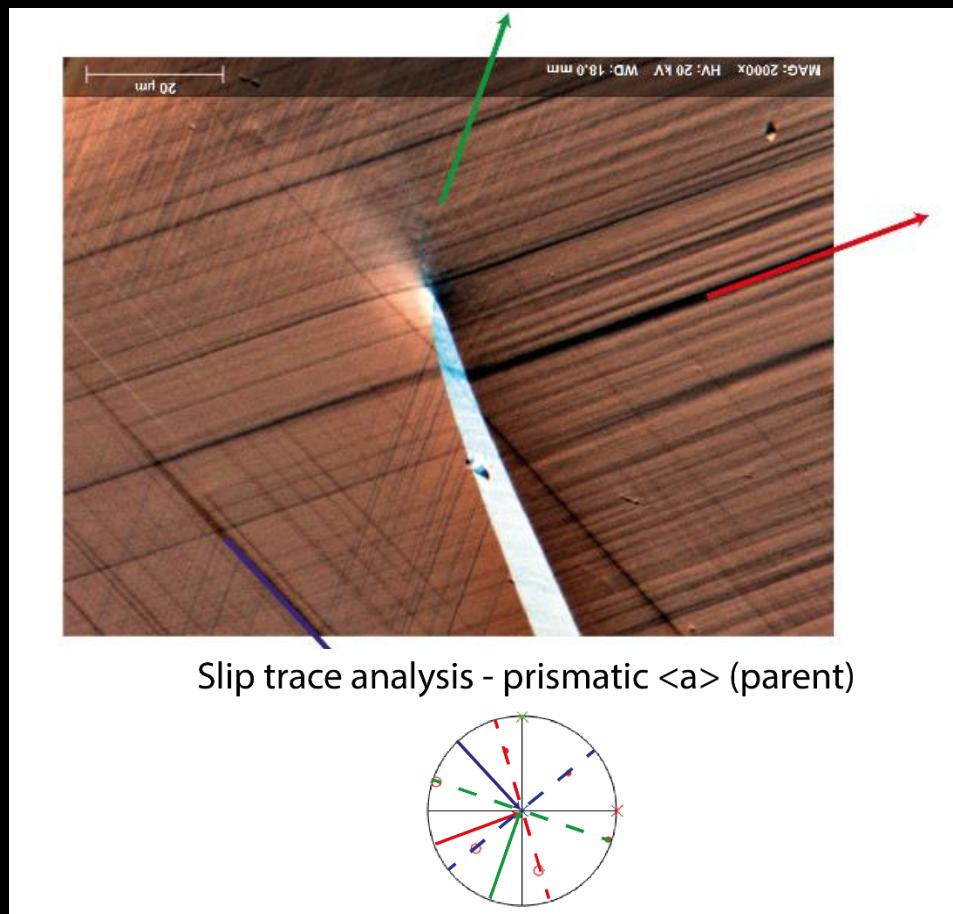


(D) Consistent description of subsurface trace (180° rotation of pole figure)



# CONSISTENCY CHECK – LOWER SYMMETRY

- Titanium T1 type twin – check the slip traces



**1**

Establish sampling coordinate system

- Scan silicon wafer and record pattern centre motion / pattern shifts
- Describe consistent coordinate system for detector & specimen

**2**

Establish consistent rotation of pattern and sample

- Rotate sample about specimen normal and record secondary electron micrograph and EBSPs
- Compare feature and diffraction pattern motion and confirm use of right handed set

**3**

Establish description of lower symmetry unit cell

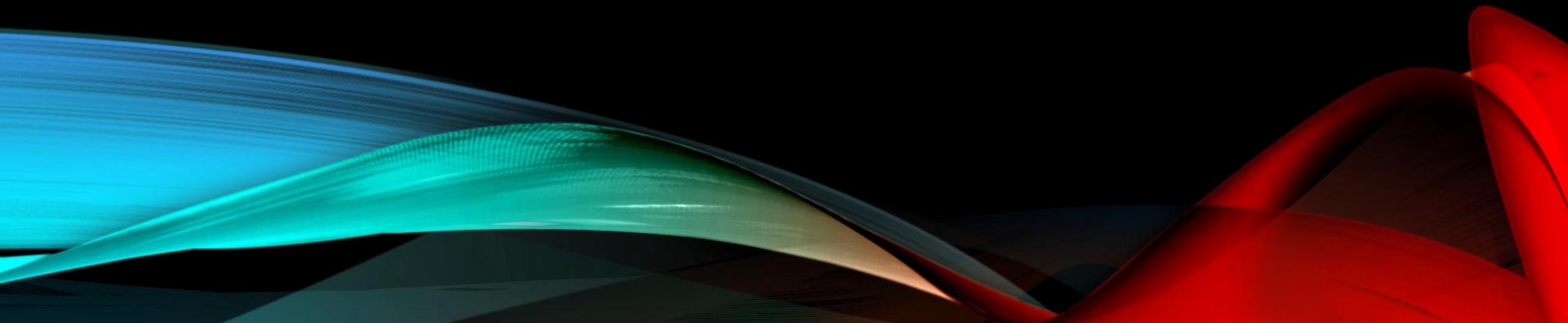
- Capture and record diffraction pattern from crystal and match with sample feature (e.g. slip band)
- Simulate diffraction pattern using low symmetry simulation code

**4**

Validate out of plane convention, pole figure use and crystallographic feature analysis

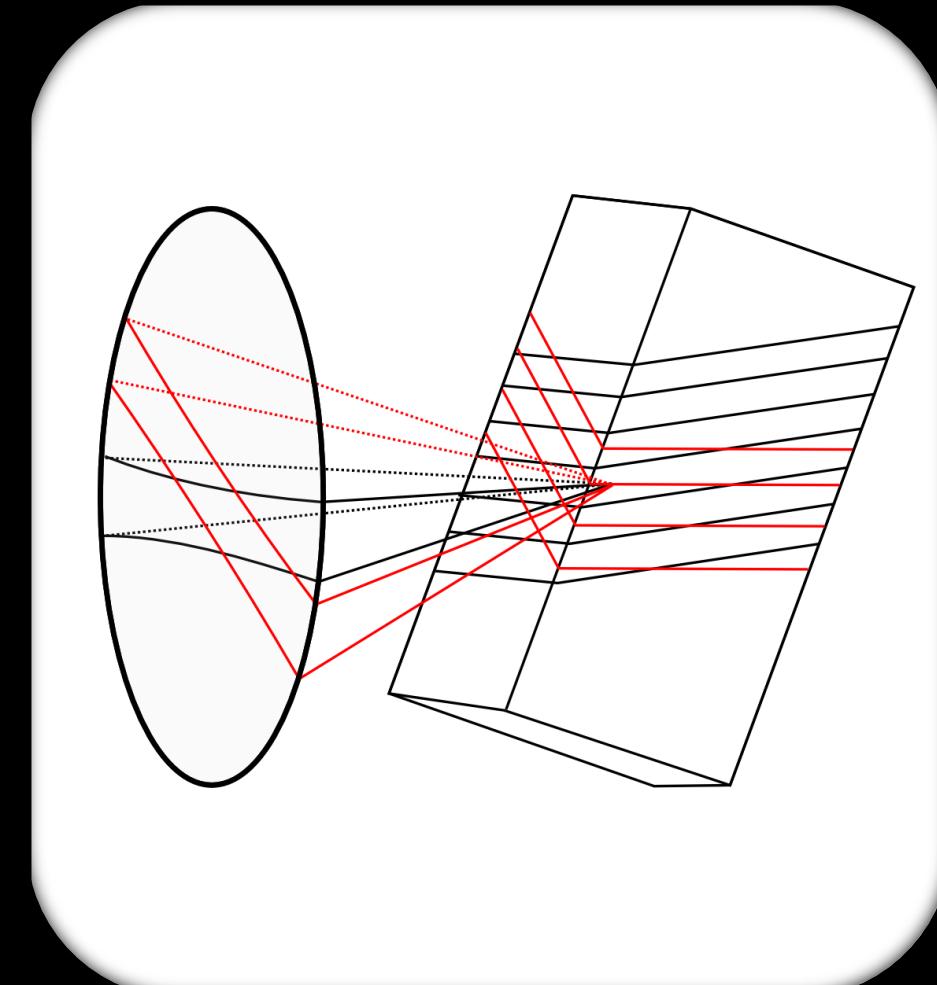
- Sample 3D microstructure with a specific crystallographic feature (e.g. annealing twins)
- Compare crystallographic feature with microstructure map (e.g. with pole figures)

# HIGH RESOLUTION EBSD



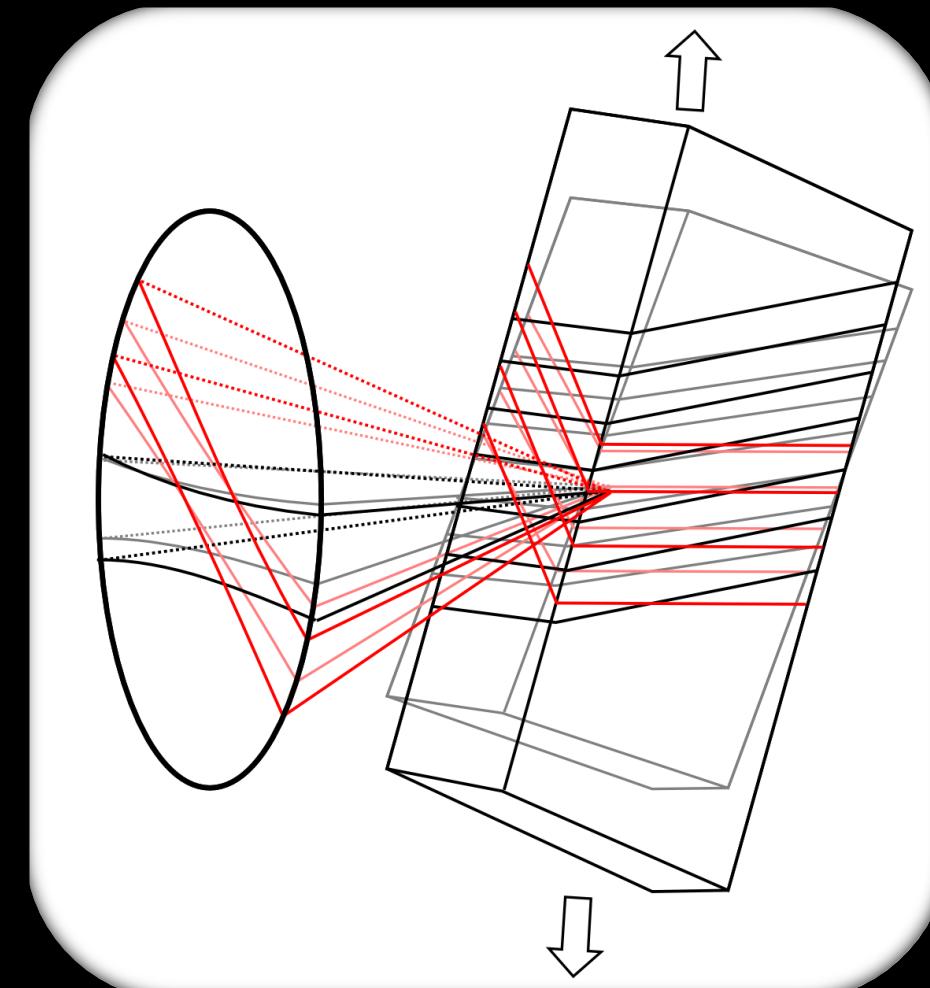
# HR-EBSD: ACCESSING STRAIN

- EBSD = direct projection of crystal lattice



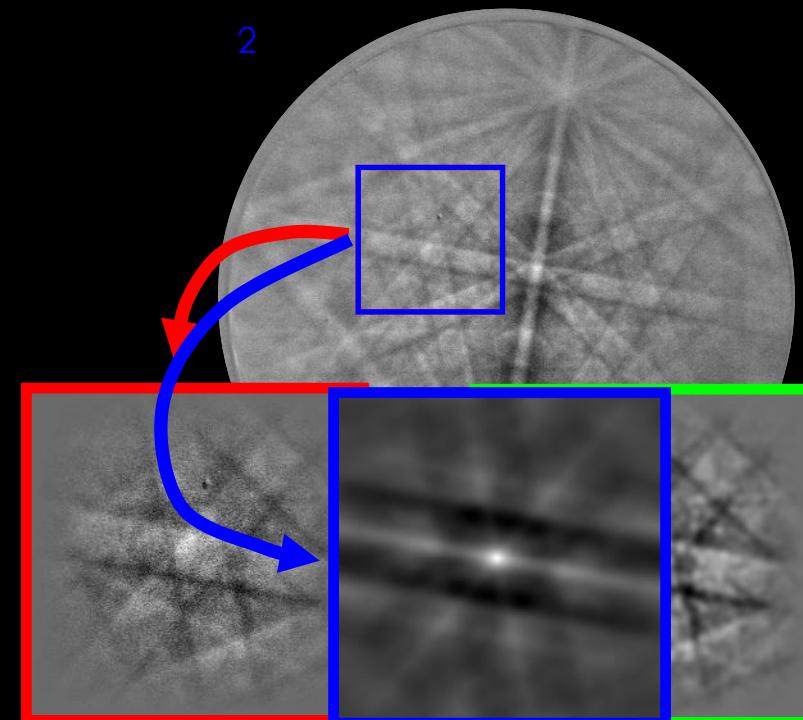
# HR-EBSD: ACCESSING STRAIN

- EBSD = direct projection of crystal lattice
- Strain crystal, pattern changes
  - Interplanar angles change

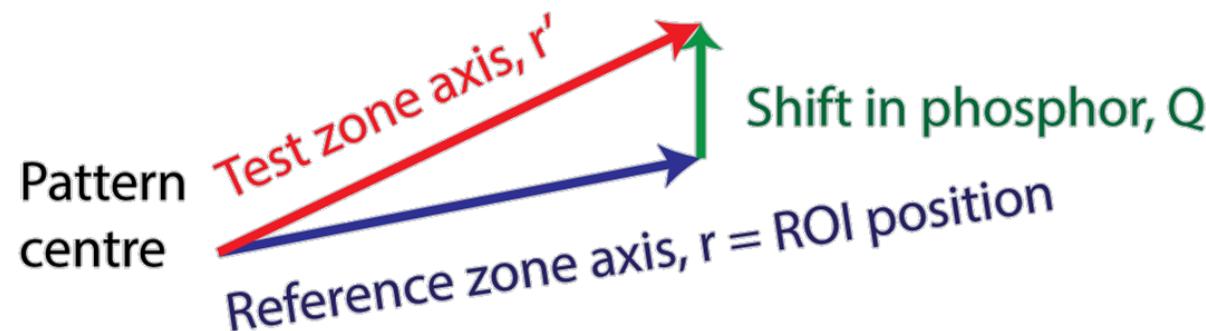


# HR-EBSD: MEASURING SHIFTS

- 20+ ROI used (offline)
  - Select ROIs → FFT
  - Apply filter
- Compare unstrained (1) vs strained (2) pattern
  - Upsample peak in XCF of ROI
    - $x\text{shift} = -6.06$  (pixels)
    - $y\text{shift} = -4.59$
- ‘Just’ an educated ‘guess’ of the translation vector between test & reference



# HR-EBSD: SHIFTS → STRAINS



$$\begin{aligned} r_x(A_{xx} - A_{zz}) + r_yA_{xy} + r_zA_{xz} + \frac{r_xr_x}{r_z}A_{zx} + \frac{r_yr_x}{r_z}A_{zy} &= Q_x \\ r_xA_{yx} + r_y(A_{yy} - A_{zz}) + r_zA_{yz} + \frac{r_xr_y}{r_z}A_{zx} + \frac{r_yr_y}{r_z}A_{zy} &= Q_y \end{aligned}$$

Infinitesimal:

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

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

Finite:

Deformation gradient  $\rightarrow F = A + I = U\Sigma V^*$

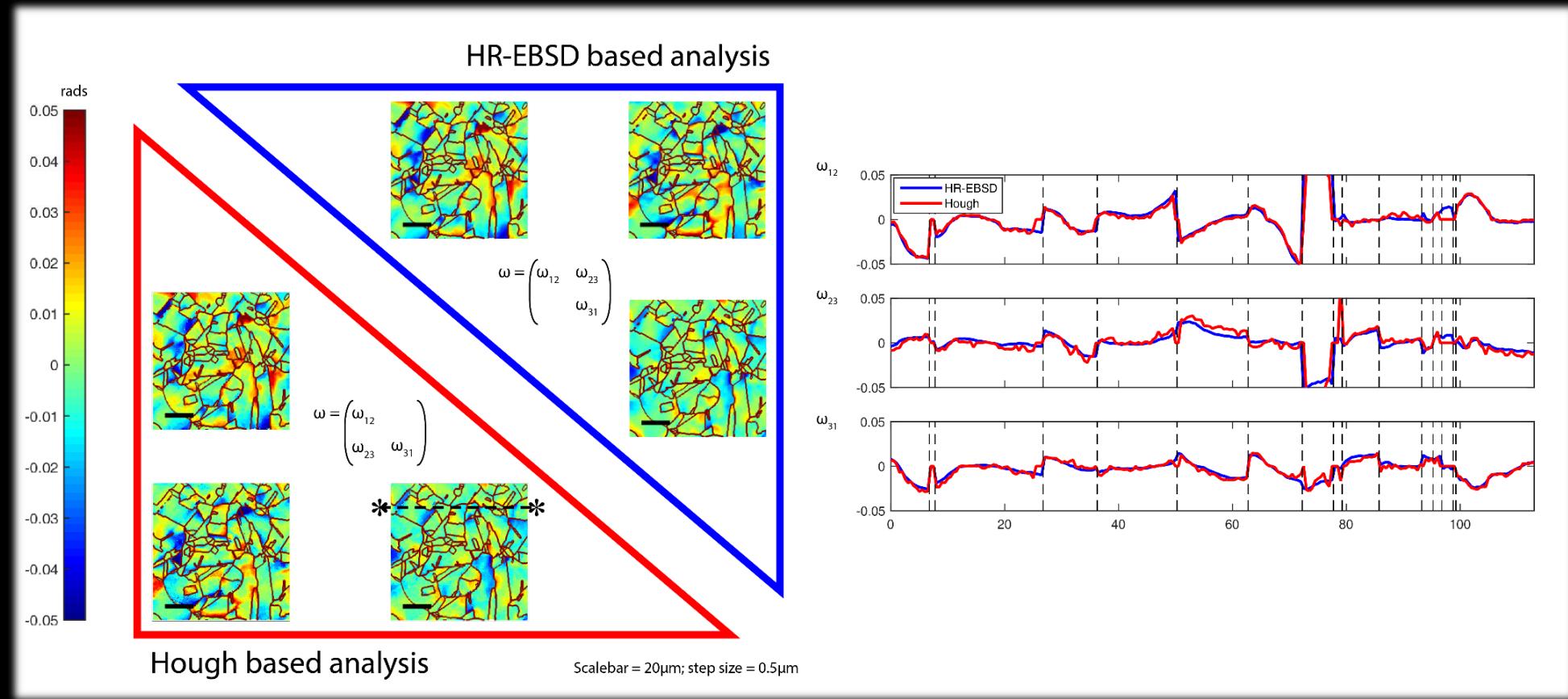
$$\varepsilon = \frac{1}{2}(F^T \cdot F - I)$$

Displacement gradient  
Finite rotation  $\rightarrow R = UV^*$

# CONVENTIONAL EBSD VS HR-EBSD

	Conventional-EBSD	HR-EBSD
<b>Absolute Orientation</b>	~2°	No
<b>Misorientation</b>	~0.1 to 0.5°	~0.006° ( $1 \times 10^{-4}$ rads)
<b>Deformation –</b>		
<b>GNDs @ 1µm step</b>	$> 3 \times 10^{13}$	$> 3 \times 10^{11}$
<b>GNDs @ 100nm step in lines / m<sup>2</sup> (b = 0.3nm)</b>	$> 3 \times 10^{14}$	$> 3 \times 10^{12}$
<b>Relative elastic strain</b>	No	Deviatoric strain $\pm 1 \times 10^{-4}$
<b>Relative residual stress (Type III – within grain)</b>	No	Anisotropic Hooke's law $\pm 20$ MPa (E=200GPa)
<b>Example tasks:</b>	Microstructure, Texture, Grain size, etc.	<b>Deformation</b> i.e. elastic strain, misorientation & residual dislocation content

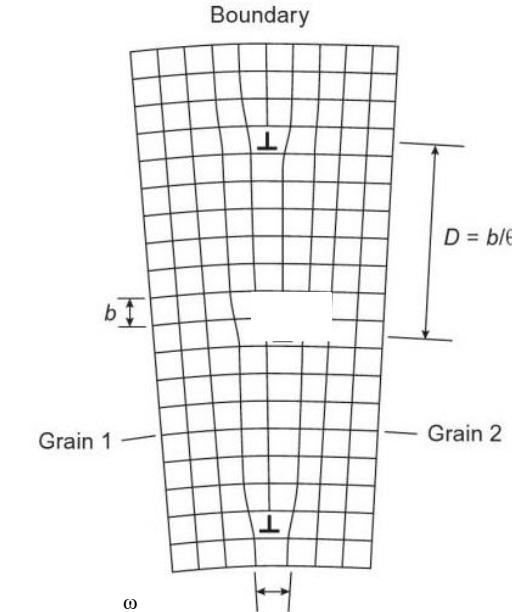
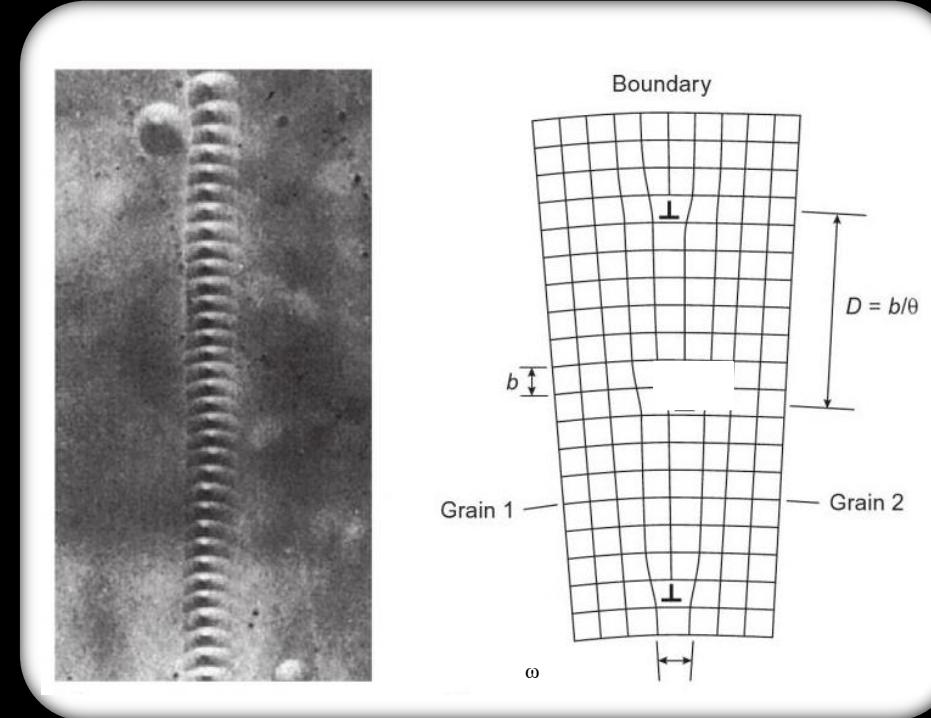
# EBSD VS HR-EBSD



- EBSD vs HR-EBSD  
= similar trends, but different details

# 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

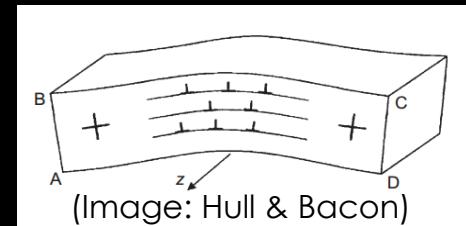
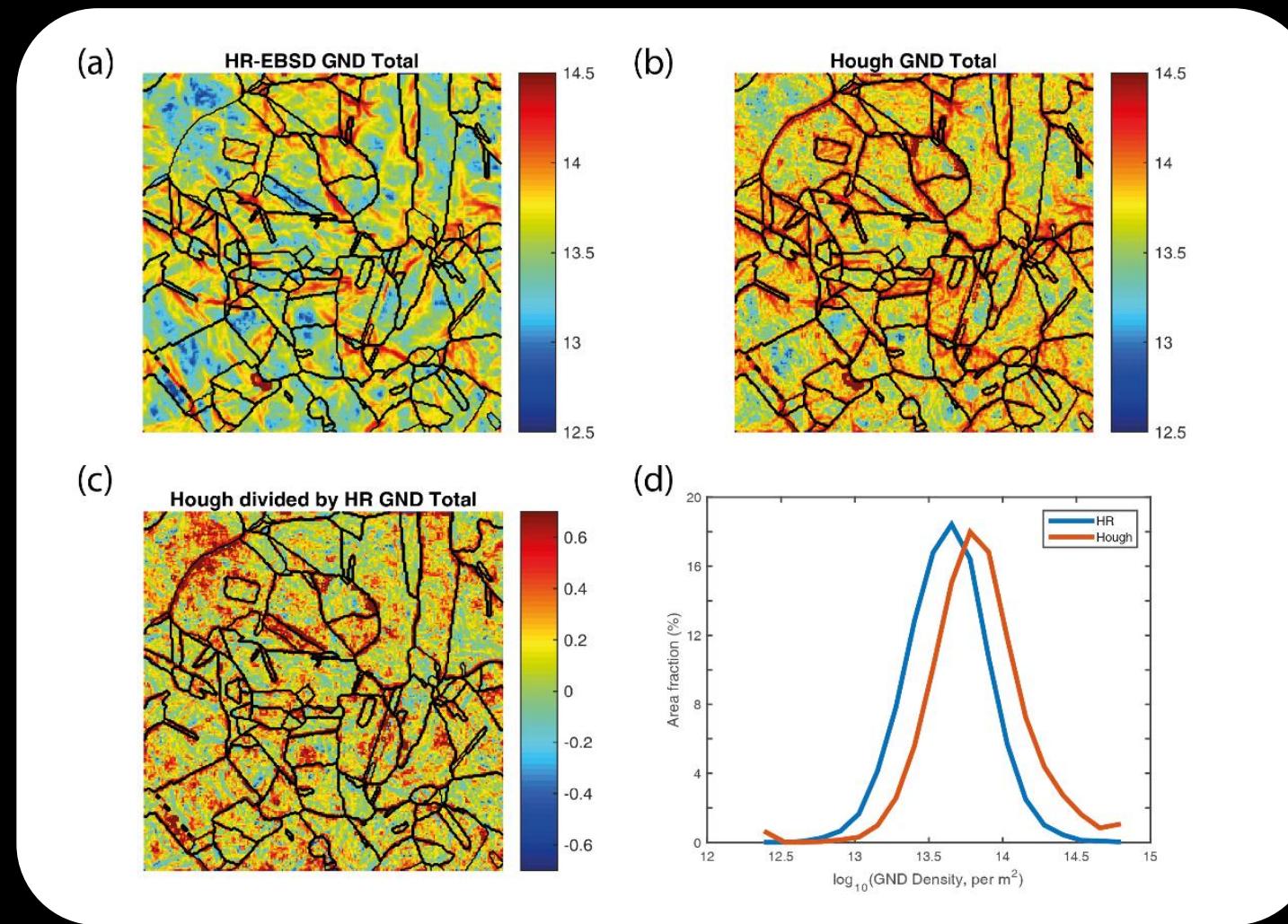
# CURVATURES & DENSITIES

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

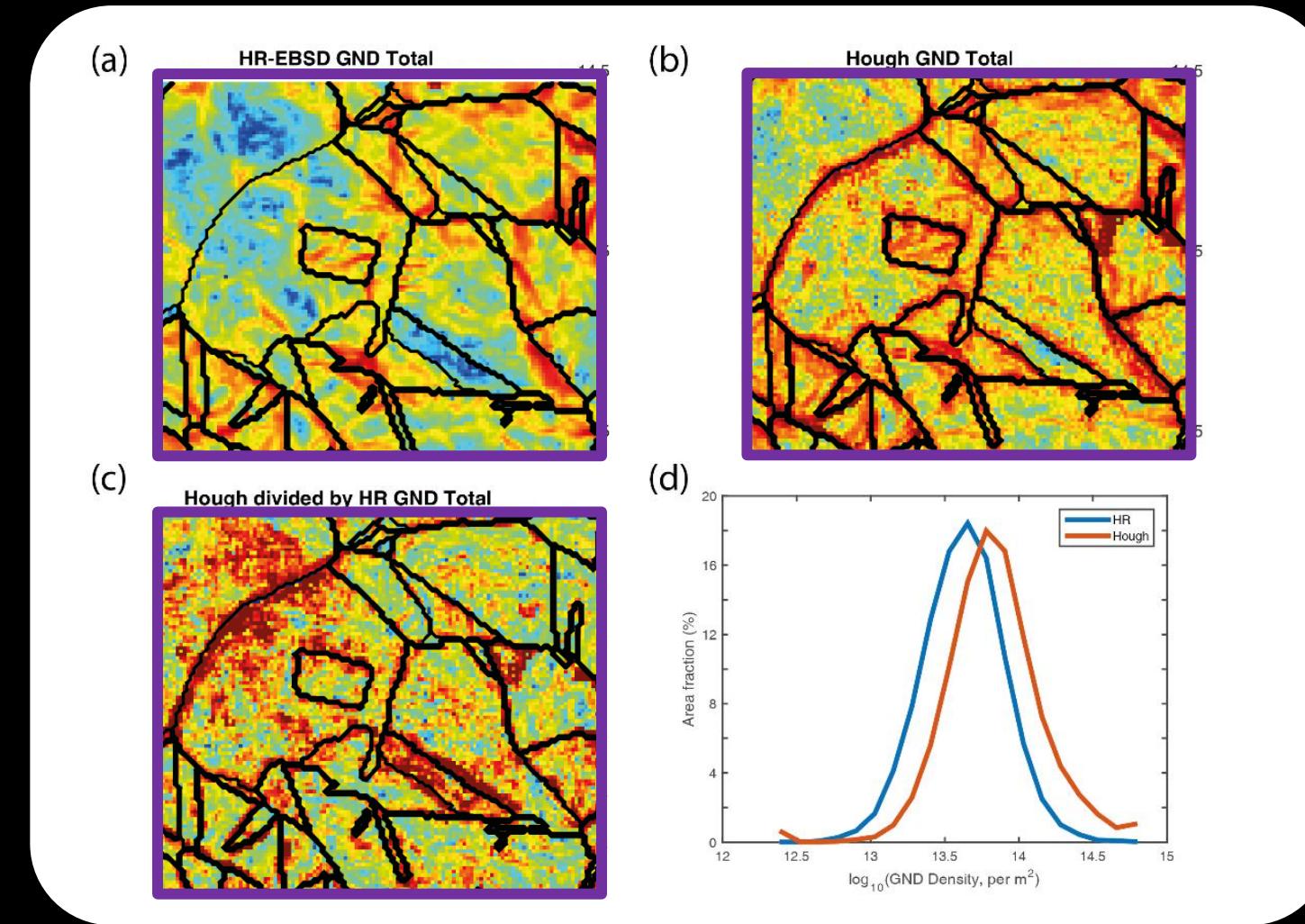
$$\begin{pmatrix} b_1 l_1 - \frac{1}{2} \mathbf{b} \cdot \mathbf{l} & \rightarrow S^{\text{th}} \\ b_1 l_2 \\ b_1 l_3 \\ b_2 l_1 \\ b_2 l_2 - \frac{1}{2} \mathbf{b} \cdot \mathbf{l} \\ b_2 l_3 \end{pmatrix} \begin{pmatrix} \rho \\ \cdot \\ S^{\text{th}} \end{pmatrix} = \begin{pmatrix} \partial \omega_{23}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

$$A \begin{pmatrix} \rho \\ \cdot \\ S^{\text{th}} \end{pmatrix} = K$$

# DEFORMATION - DETAILS MATTER



# DEFORMATION - DETAILS MATTER

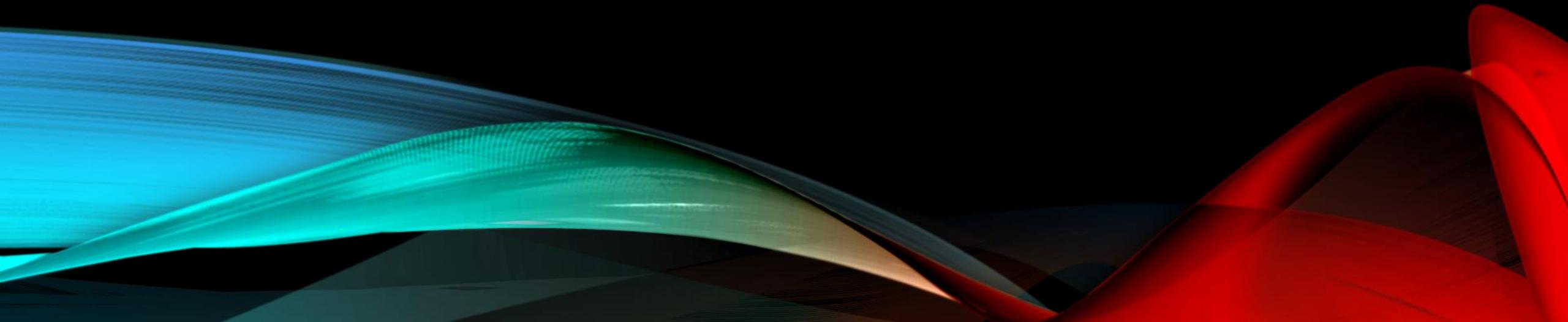


# SUMMARY – EBSD COMPARED

- EBSD = orientations + phase
  - Limited misorientation accuracy → issues for GND assessment
  - But easy to measure large misorientation + quick
- HR-EBSD = comparison of two+ patterns
  - Elastic strain
  - Lattice misorientation

# HIGH RATE ZR DEFORMATION

Vivian Tong, Euan Wielewski, and Ben Britton

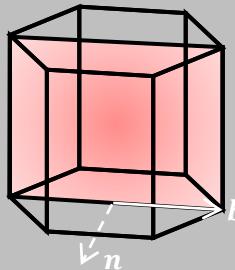


# SLIP IN ZR

Lowest CRSS (RT,  
quasi-static  
deformation)

$\langle a \rangle$

prismatic slip

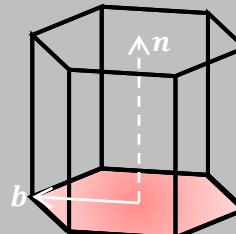


$$\mathbf{n} = (1\bar{1}00)$$

$$\mathbf{b} = [11\bar{2}0]$$

$\langle a \rangle$

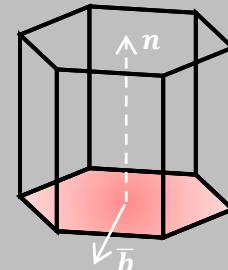
basal slip



$$\mathbf{n} = (0001)$$

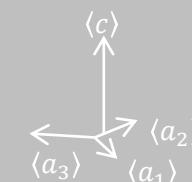
$$\mathbf{b} = [\bar{1}\bar{1}20]$$

$\langle a_1 + a_2 \rangle$   
basal slip



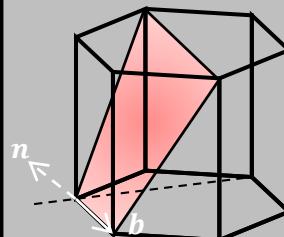
$$\mathbf{n} = (0001)$$

$$\mathbf{b} = [1\bar{1}00]$$



CRSS: 1.3×  
 $\langle a \rangle$  prismatic  
(RT, QS)

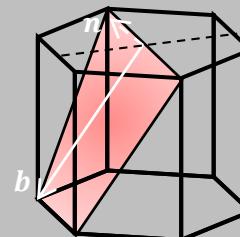
$\langle a \rangle$  1<sup>st</sup> order  
pyramidal slip



$$\mathbf{n} = (0\bar{1}11)$$

$$\mathbf{b} = [2\bar{1}10]$$

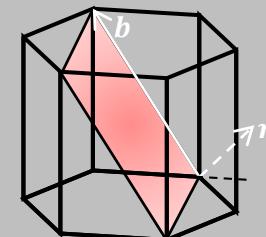
$\langle c + a \rangle$  1<sup>st</sup> order  
pyramidal slip



$$\mathbf{n} = (0\bar{1}11)$$

$$\mathbf{b} = [\bar{1}12\bar{3}]$$

$\langle c + a \rangle$  2<sup>nd</sup> order  
pyramidal slip



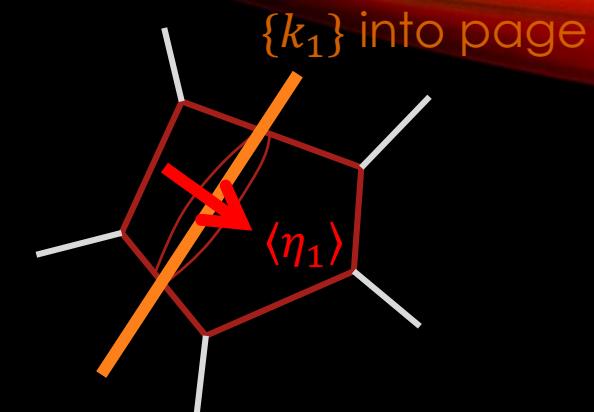
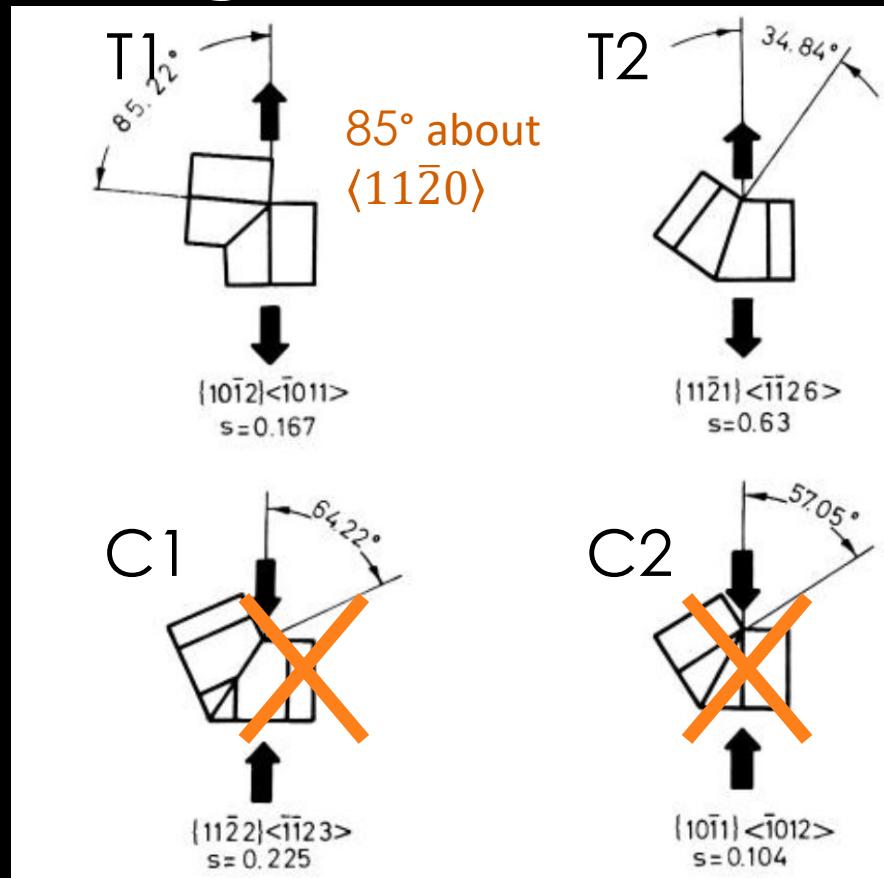
$$\mathbf{n} = (1\bar{1}\bar{2}2)$$

$$\mathbf{b} = [\bar{1}123]$$

CRSS: 3.5×  $\langle a \rangle$  prismatic (RT, QS)

<http://www.expmicromech.com>

# TWINNING IN ZR



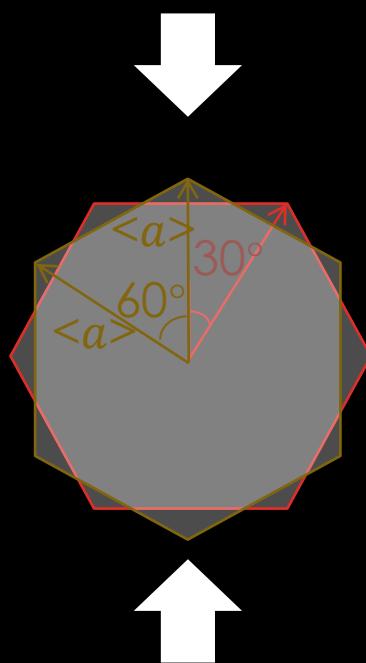
## Twin characteristics:

- $\langle c \rangle$ -axis extension/contraction
- Twin plane  $\{k_1\}$
- Twinning direction  $\langle \eta_1 \rangle$
- Twinning shear  $s$
- Shear plane  $\langle P \rangle$ 
  - (Lattice rotation axis)
- Lattice rotation angle  $\theta$

Easy to identify & characterise using EBSD intergranular misorientations

# DEFORMATION IN HCP CRYSTALS

**'Soft' sample**

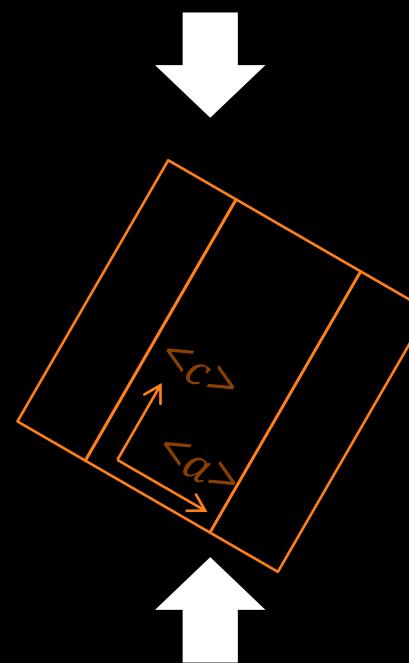


$\langle a \rangle$  prism slip  
 $\{10\bar{1}0\}<11\bar{2}0>$

T1 twinning  
 $\{10\bar{1}2\}<10\bar{1}1>$

T2 twinning  
 $\{11\bar{2}1\}<11\bar{2}\bar{6}>$

**'Harder' sample**



$\langle a \rangle$  prism slip  
 $\{10\bar{1}0\}<11\bar{2}0>$

$\langle a \rangle$  basal slip  
 $\{0001\}<11\bar{2}0>$

T2 twinning  
 $\{11\bar{2}1\}<11\bar{2}\bar{6}>$

T1 twinning  
 $\{10\bar{1}2\}<10\bar{1}1>$

& limited  $\langle c + a \rangle$  pyramidal slip

# RESULTS: MECHANICAL DATA

## Contribution to work hardening:

**Twinning:** Small contribution:

- Crystal could rotate to harder orientation
- Grain size refined

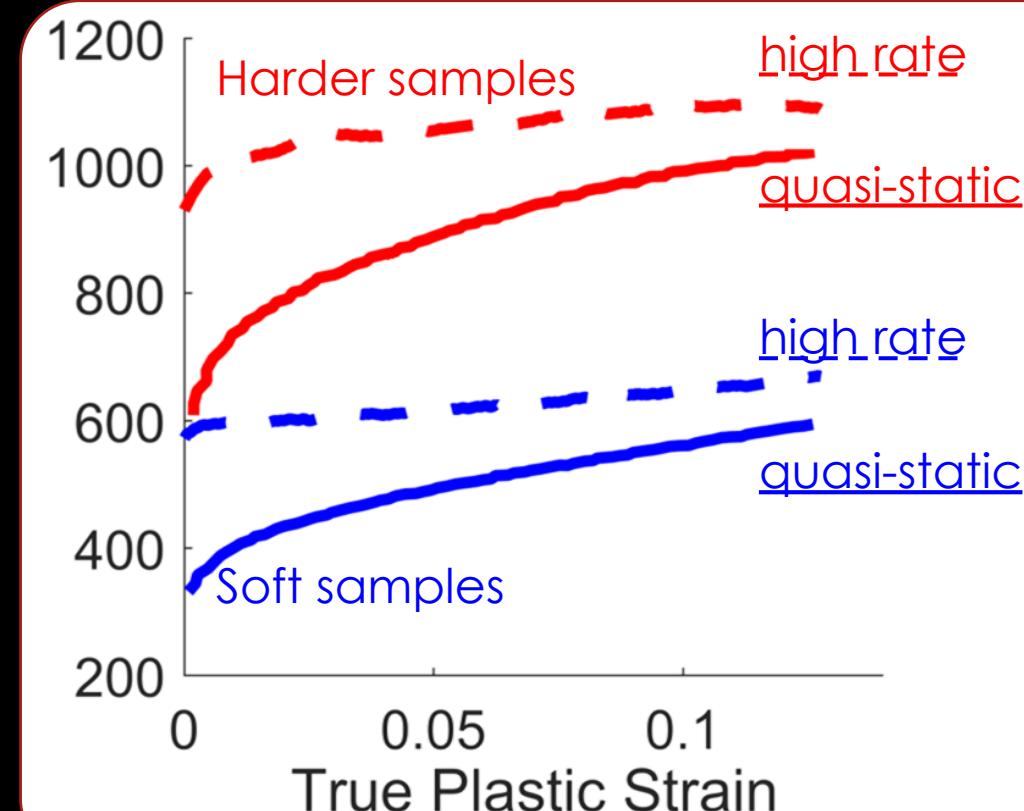
**Single slip:** Small contribution

- Slip blocked at grain boundaries
- Regions run into each other

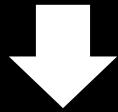
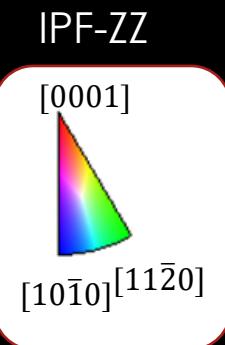
**Multiple slip:** large contribution

- Dislocation cell structures
- Forest hardening

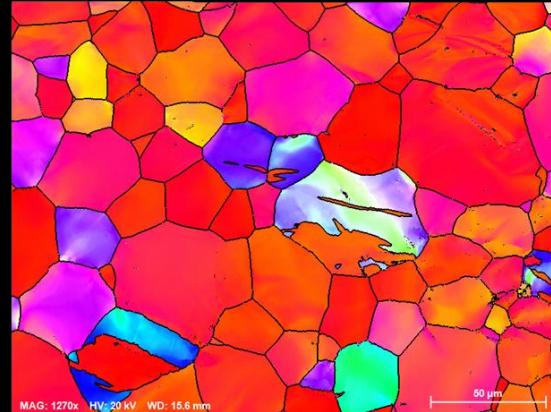
True Stress (MPa)



# RESULTS: ORIENTATIONS



Quasi-static

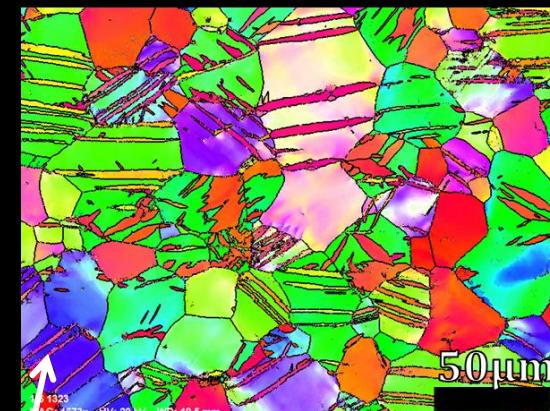
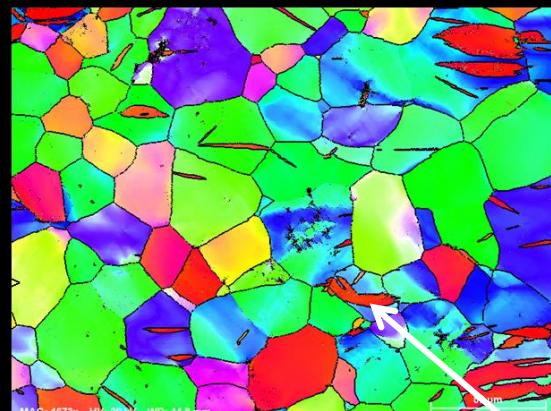
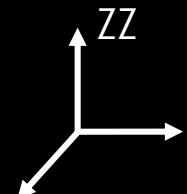


High strain rate



'Harder' samples

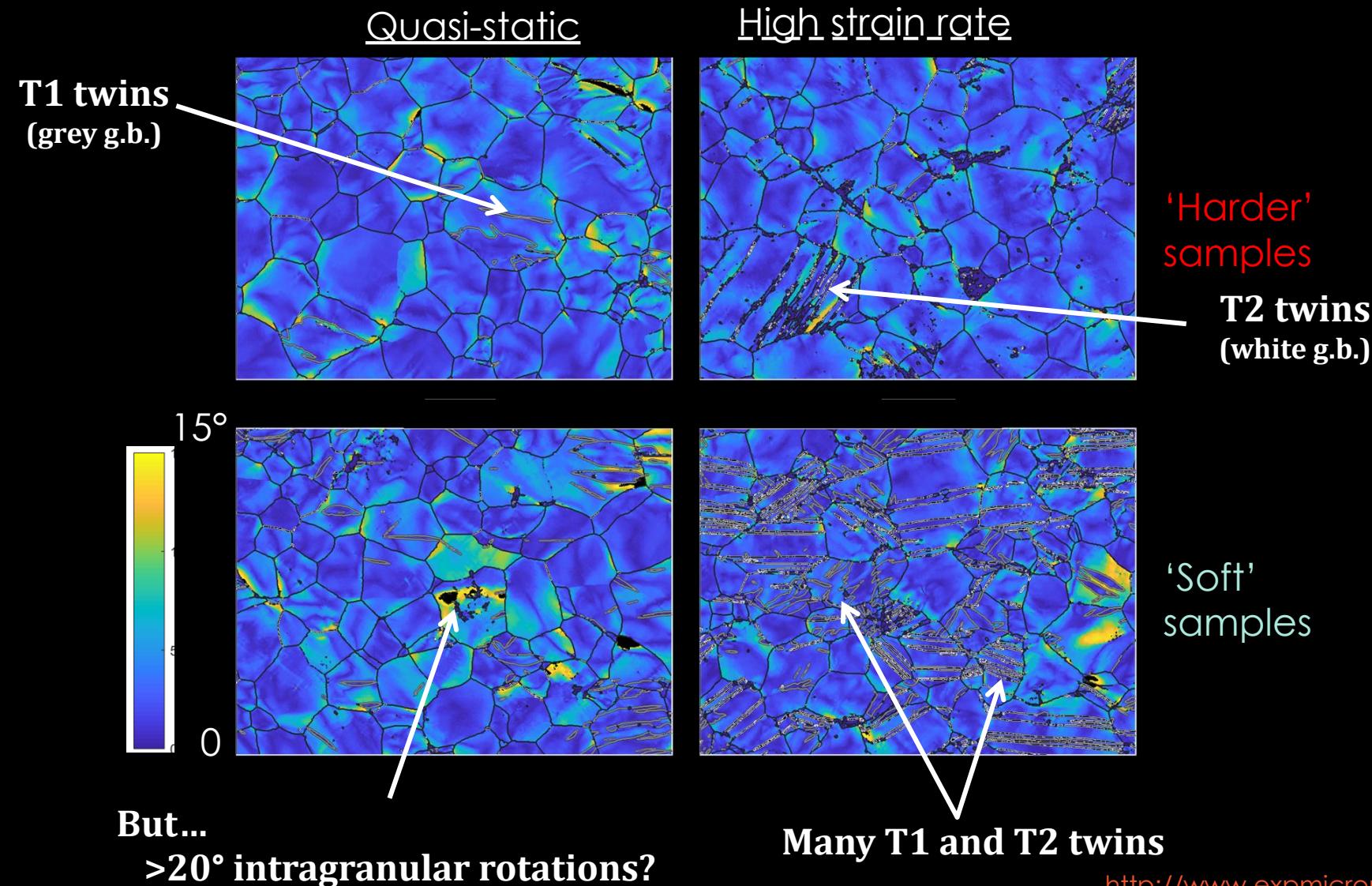
Texture difference



'Soft' samples

Hard twins, soft parents

# RESULTS: MISORIENTATION ANGLES



# TWIN ANALYSIS - QUANTITATIVE

	ND-QS		ND-HR		TD-QS		TD-HR	
	T1	T2	T1	T2	T1	T2	T1	T2
EBSD step size	0.3 µm							
Total grains (inc. twins)	136		233		231		920	
Grain diameter (exc. FOV edge)	21 µm		13 µm		14 µm		7 µm	
Twin area fraction	4 %	0 %	1 %	2 %	6 %	0 %	9 %	12 %
Strain from twinning	0.4 %	0 %	0.1 %	0.9 %	0.7 %	0 %	1 %	5 %
Num. twins	13	0	5	14	67	0	150	121
Num. parents	7	0	3	4	32	0	37	41
Mean twins/ parent	1.9		1.7	3.5	2.1		4.1	3.0
Max variants/ parent	2		1	1	2		5	2
Mean twin length/µm	17 µm		10 µm	36 µm	11 µm		8 µm	19 µm
Mean twin width	6 µm		3 µm	2 µm	2 µm		3 µm	2 µm
Mean twin area	76 µm <sup>2</sup>		26 µm <sup>2</sup>	62 µm <sup>2</sup>	18 µm <sup>2</sup>		17 µm <sup>2</sup>	22 µm <sup>2</sup>

# QUANTIFYING MICRO-DEFORMATION

## Method 1: Identify residual dislocations

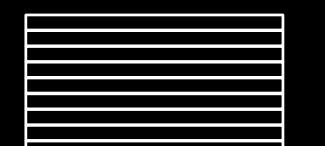
e.g. TEM diffraction, synchrotron XRD, HR-EBSD

- Measure elastic strain around dislocations
  - Residual stress
  - Image contrast
- Burgers vector  $\mathbf{b}$  & line direction  $\mathbf{l}$  resolved
- Slip plane not always known
- Glissile/sessile character not known

### IMPORTANT!

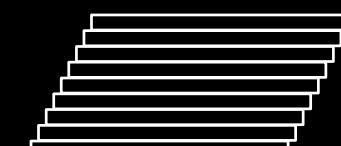
Residual dislocations  $\neq$  slip activation

This technique cannot measure plastic strain, e.g.

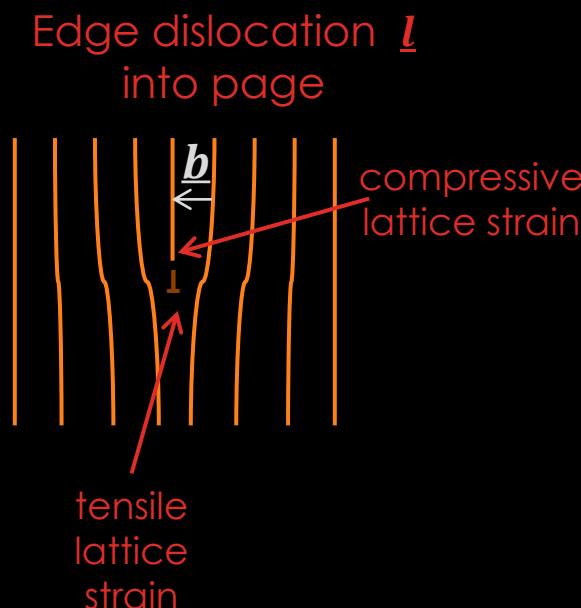


Undeformed

Single  
slip



Deformed

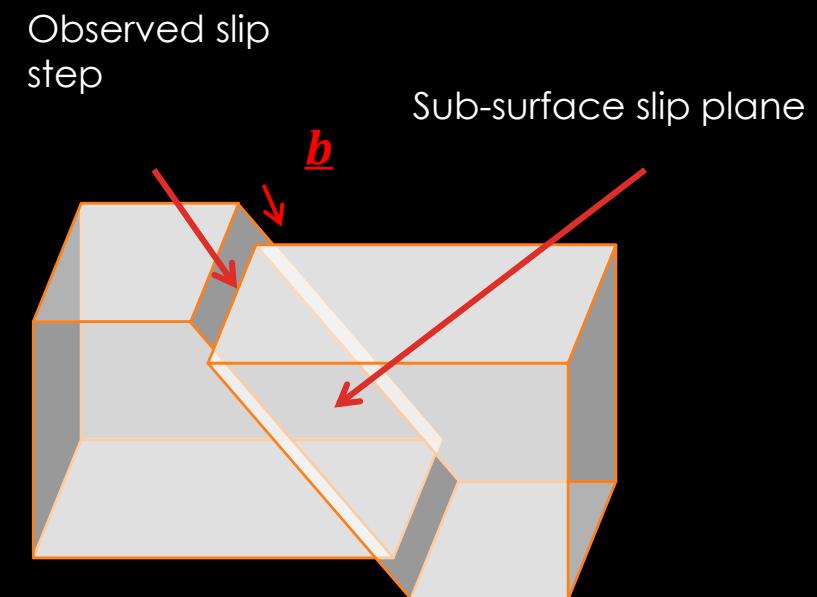


No residual dislocations

# QUANTIFYING MICRO-DEFORMATION

Method 2: Identify slip plane  
e.g. SEM-EBSD slip trace analysis [1]

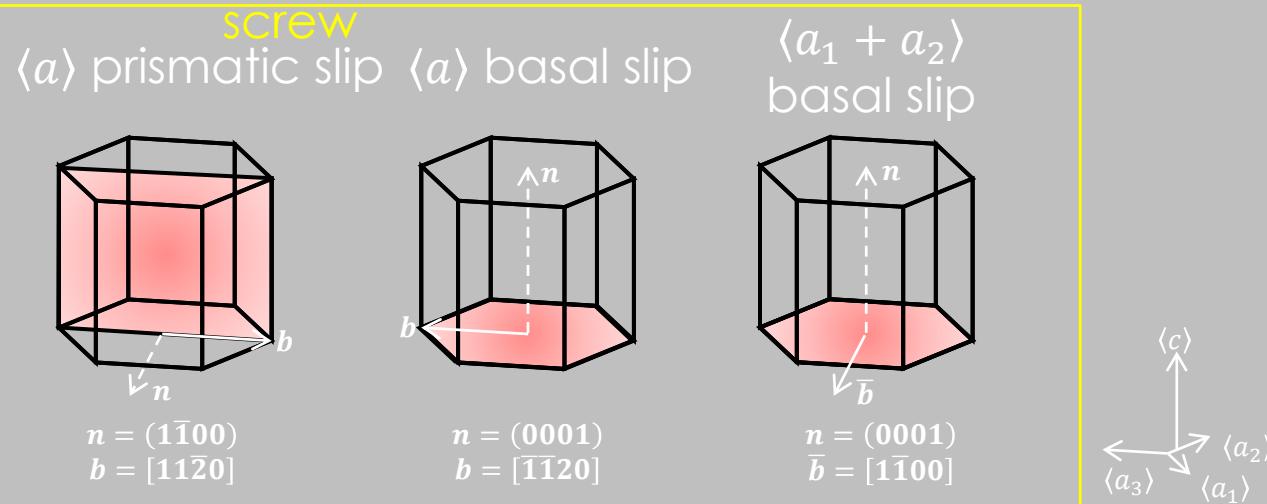
- Correlate surface trace (imaging, AFM or DIC) with crystal orientation (EBSD)
- Slip plane resolved
- Slip direction usually unknown



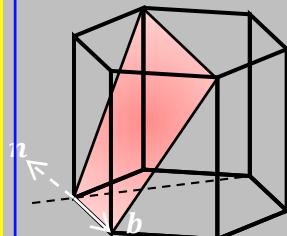
[1] A. Orozco-Caballero et al., "How magnesium accommodates local deformation incompatibility: A high-resolution digital image correlation study," *Acta Mater* 2017

# WHICH SLIP SYSTEM?

Diffraction methods:  $\langle a \rangle$



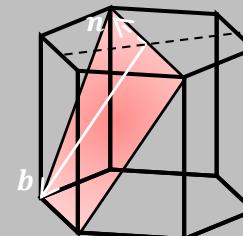
$\langle a \rangle$  1<sup>st</sup> order  
pyramidal slip



$$n = (0\bar{1}11)$$

$$b = [2\bar{1}10]$$

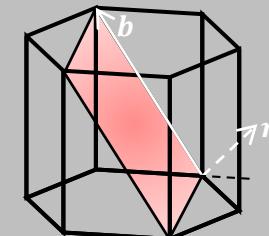
$\langle c + a \rangle$  1<sup>st</sup> order  
pyramidal slip



$$n = (0\bar{1}11)$$

$$b = [\bar{1}123]$$

$\langle c + a \rangle$  2<sup>nd</sup> order  
pyramidal slip



$$n = (11\bar{2}2)$$

$$b = [\bar{1}123]$$

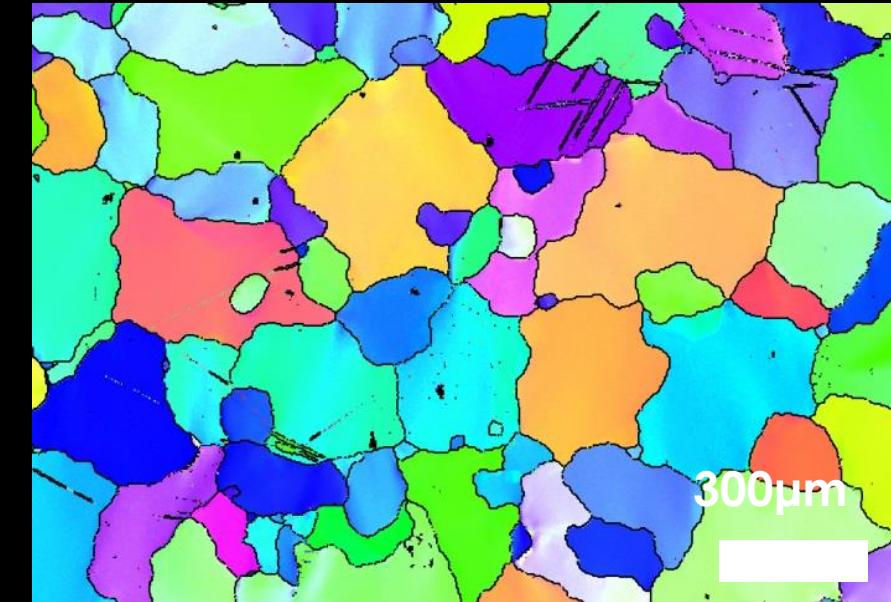
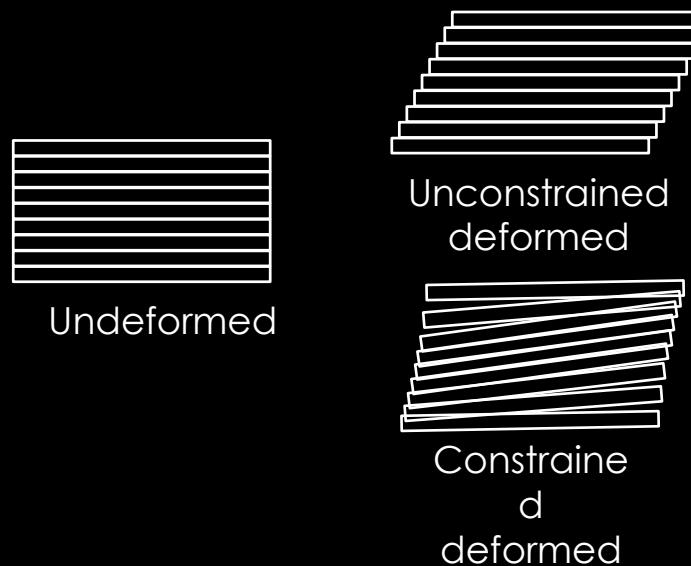
Slip trace analysis: 1<sup>st</sup> order pyramidal plane

<http://www.expmicromech.com>

# QUANTIFYING MICRO-DEFORMATION

Method 3: Identify rotation axis from constrained slip

- Slip in **constrained polycrystal** → long range lattice rotations
  - Plastic rotation balanced by lattice rotation in constrained geometry
- Must assume constraint, i.e. grain shape does not change before/after deformation

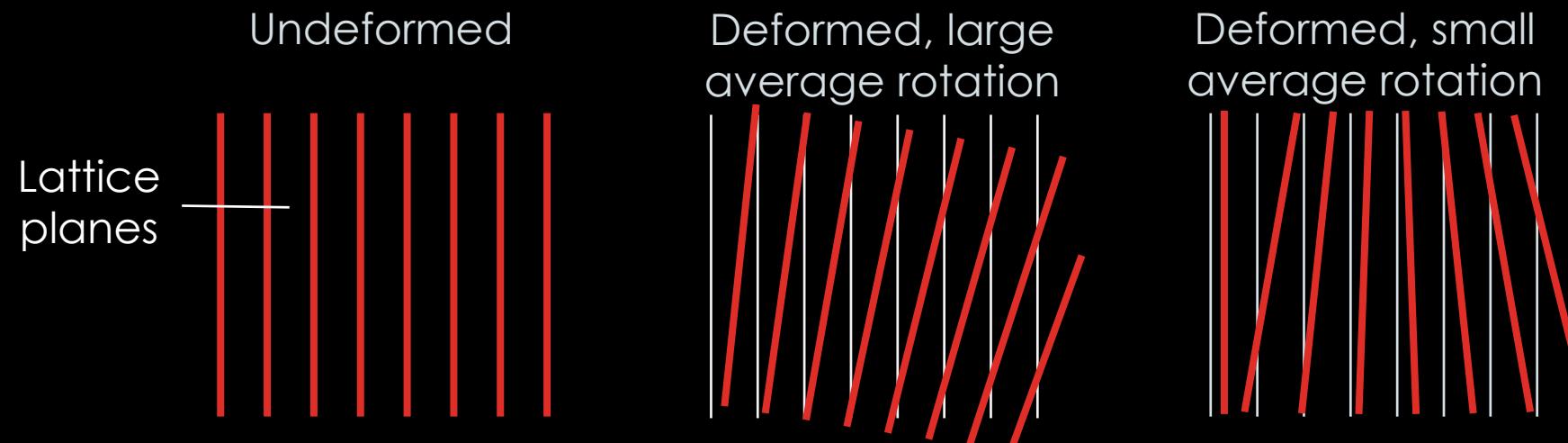


**~1% deformed, large grained Zr**  
Orientation gradients caused by slip shown as colour changes in this IPF map

Use EBSD to characterise long range lattice rotations

# QUANTIFYING MICRO-DEFORMATION

- Pre-deformation: uniform orientation ( $<2^\circ$  intragranular misorientation)
- Deformed: large orientation gradients
- Ideally, measure lattice rotation w.r.t. undeformed orientation ← not easily accessible
- Small deformations (10% strain)
  - Small total lattice rotation
  - Check: negligible crystallographic texture change before/after



# QUANTIFYING SLIP

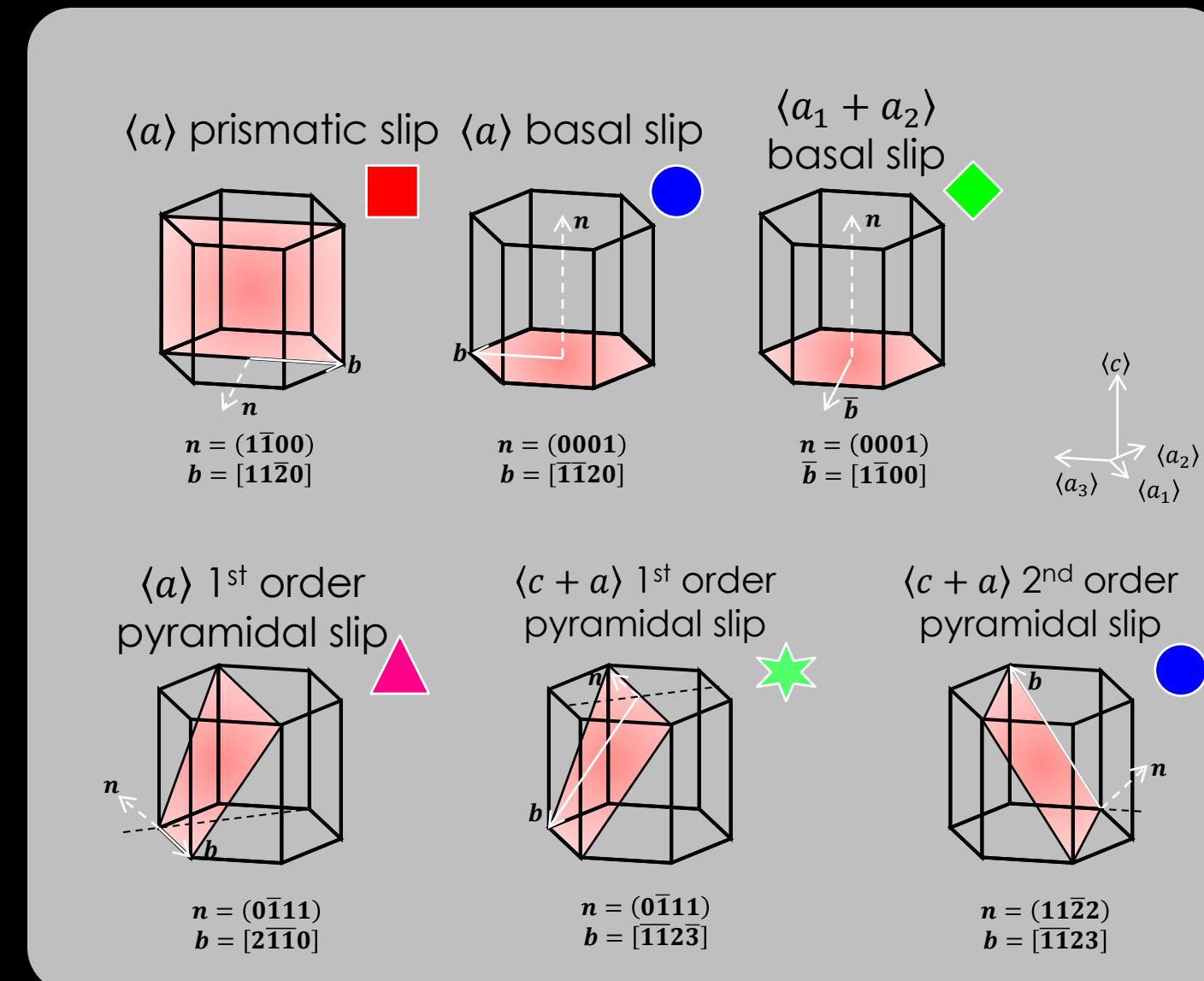
- Intragranular misorientations relative to grain mean orientation
- Approximate that mean orientation  $\approx$  undeformed state
- Misorientation: both angle & **axis**
- Axis in **sample** or **crystal** coordinate frame



45° angle  
[1̄100] axis into the page

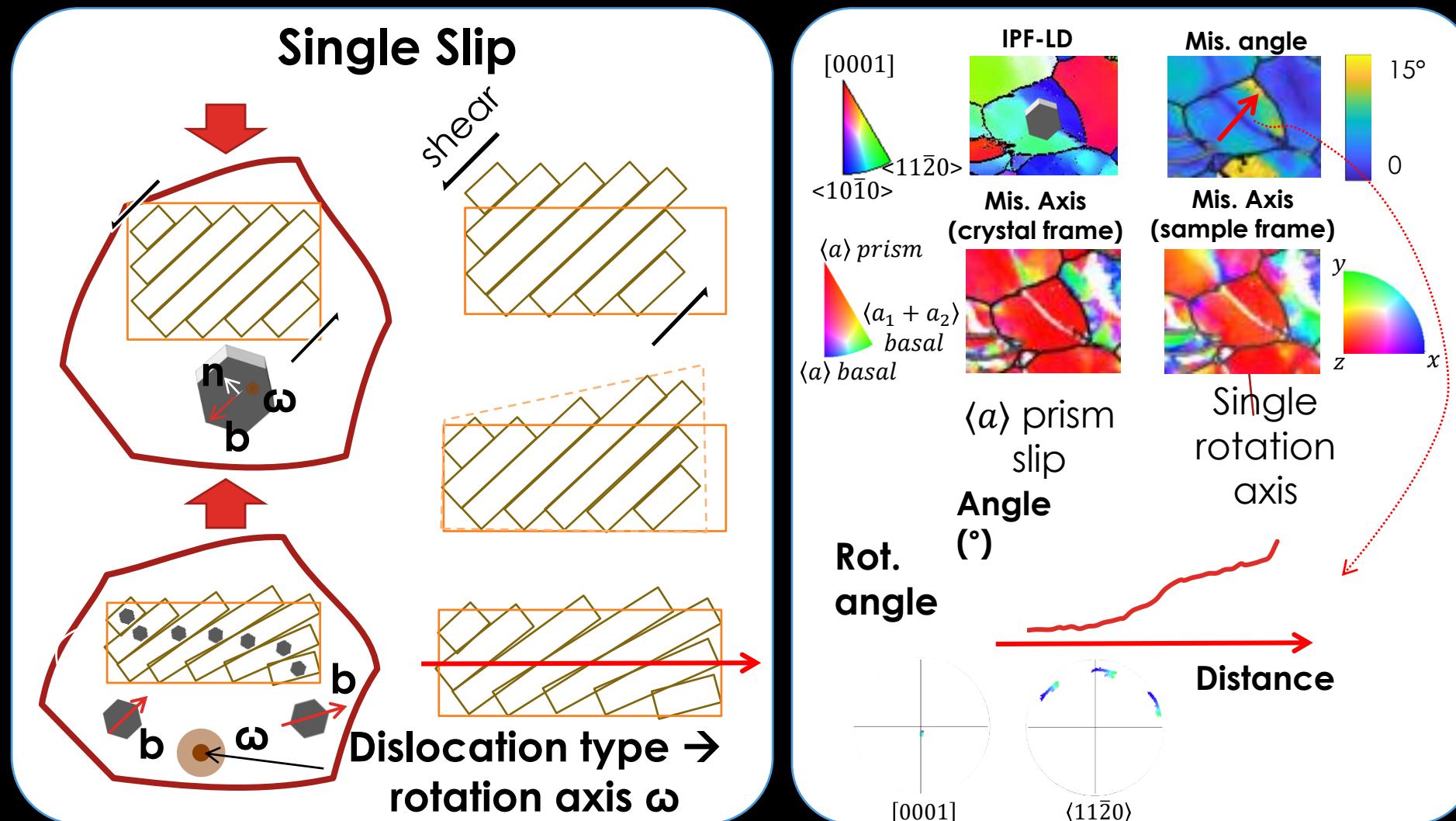
180° angle  
[112̄3] axis vertical in the page

# MISORIENTATION AXES (CRYSTAL FRAME)



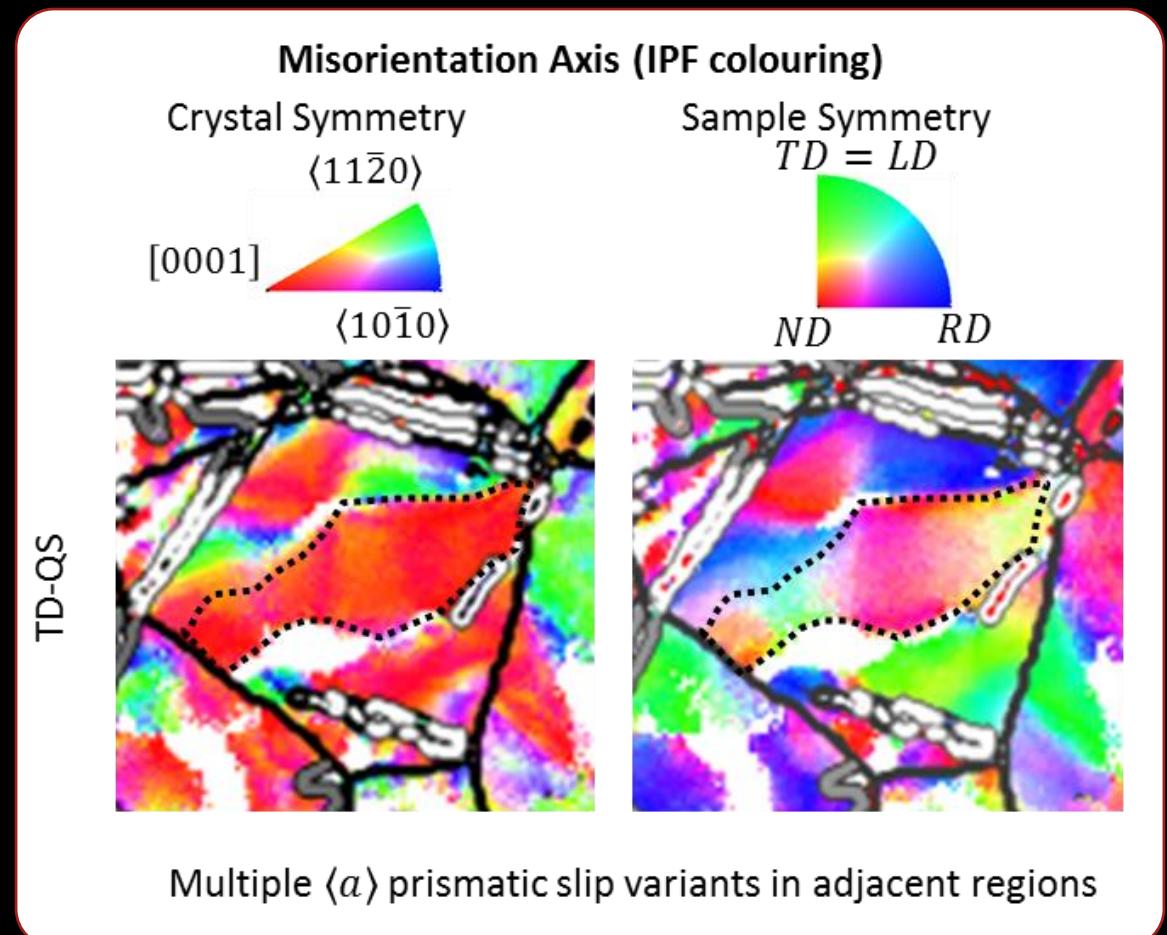
[1] A. J. Wilkinson, "A new method for determining small misorientations from electron back scatter diffraction patterns," *Scr. Mater.* 2001.

# SINGLE SLIP IN A CONSTRAINED GRAIN

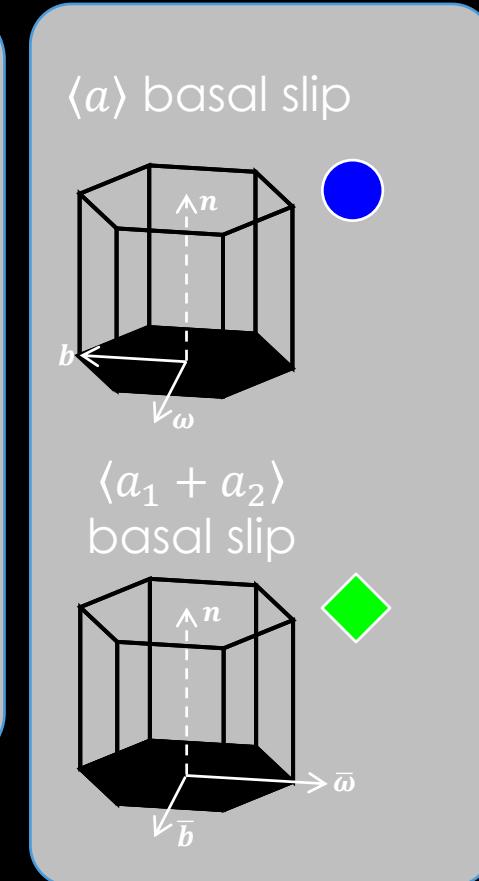
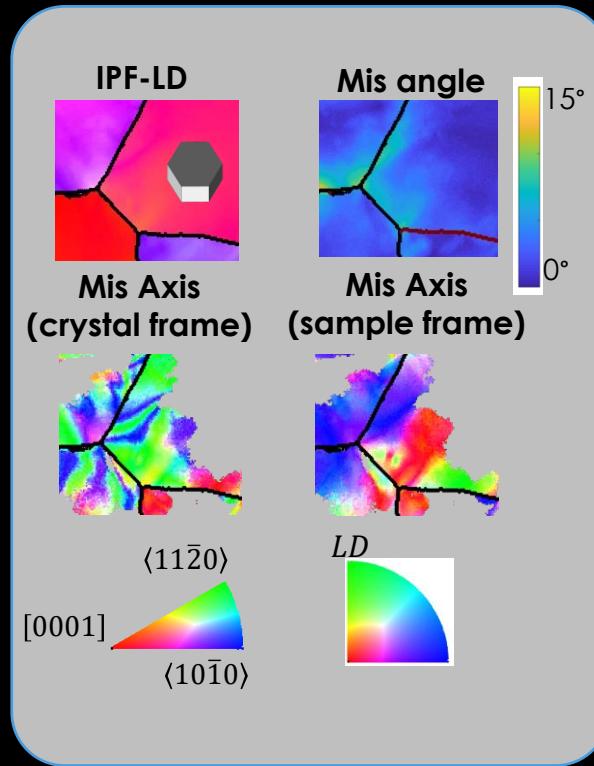
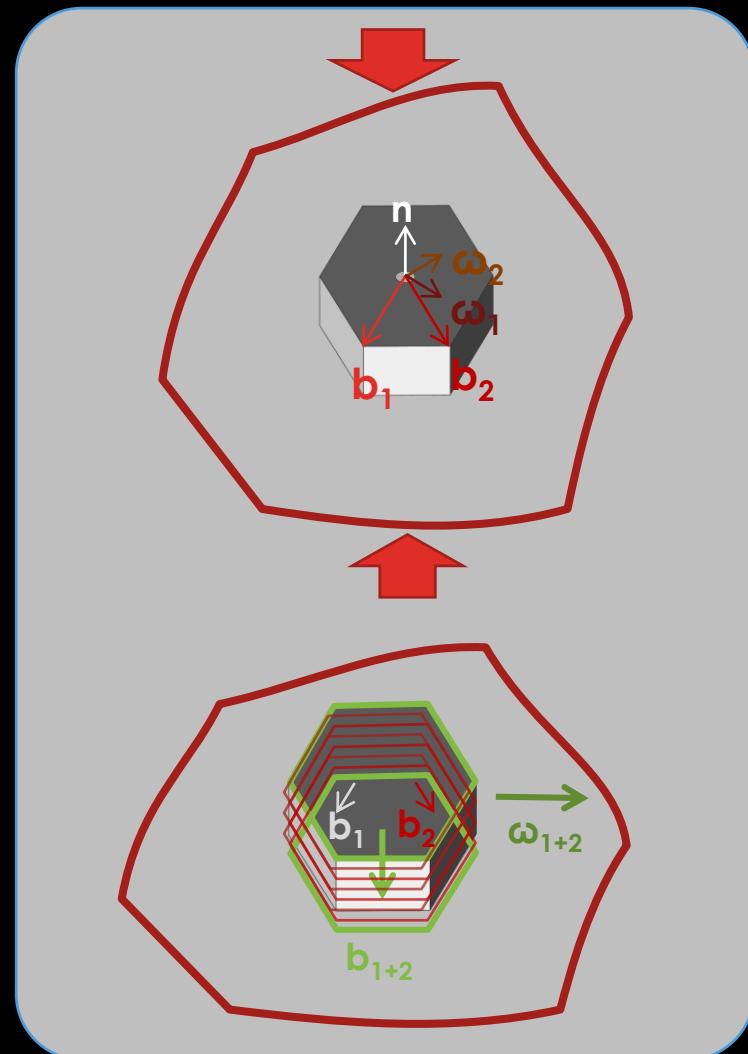


# MULTIPLE PRISMATIC SLIP

- Misorientation axis using crystal = red = [0001] = prism
- Variation in sample colouring (rainbow) = multiple (spatially resolved) crystal systems operating



# BASAL SLIP



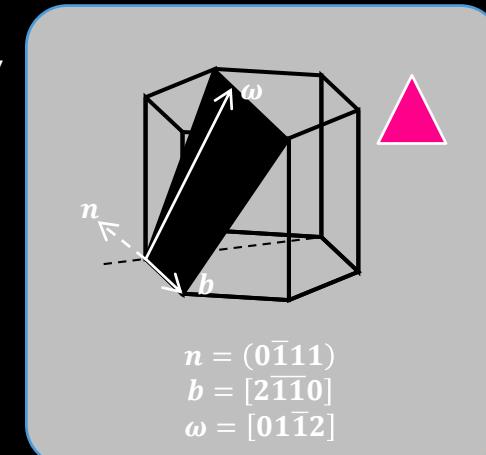
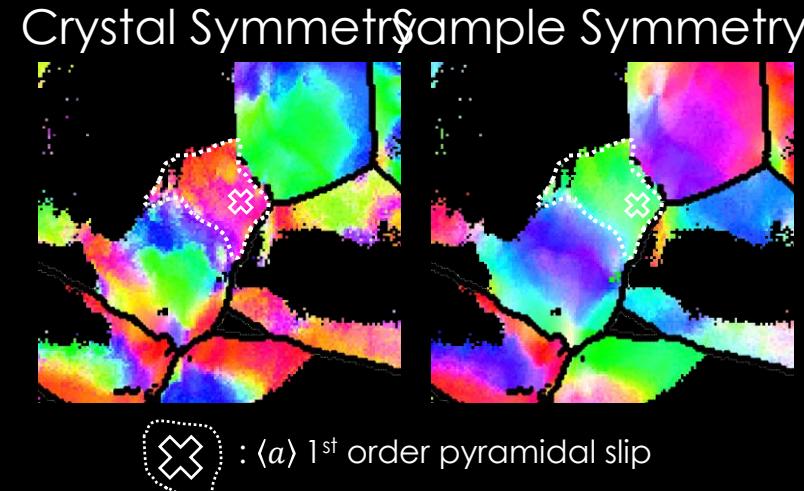
'Double' basal slip = special multiple slip mode → cooperative motion of  $\langle a \rangle$  basal slip dislocations

# $\langle a \rangle$ 1<sup>ST</sup> ORDER PYRAMIDAL SLIP

Colour resolution  
too coarse →  
plot points on IPF  
key



Allow 15° deviation locus  
(measurement uncertainty)

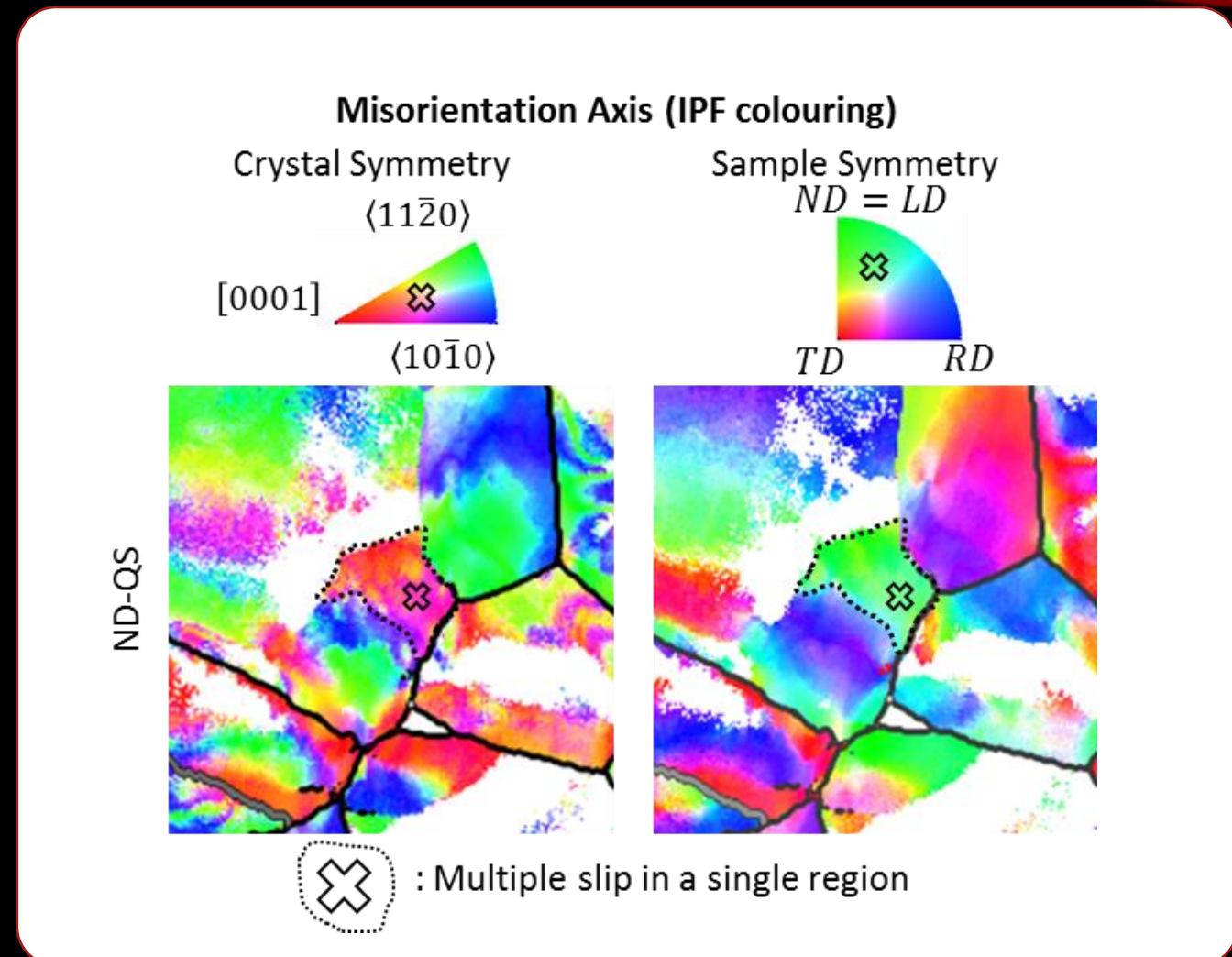


Sample orientation

Check expected misorientation axis in sample symmetry (know crystal orientation)

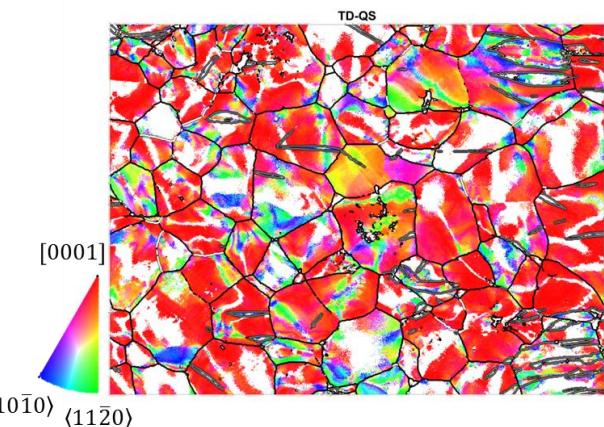
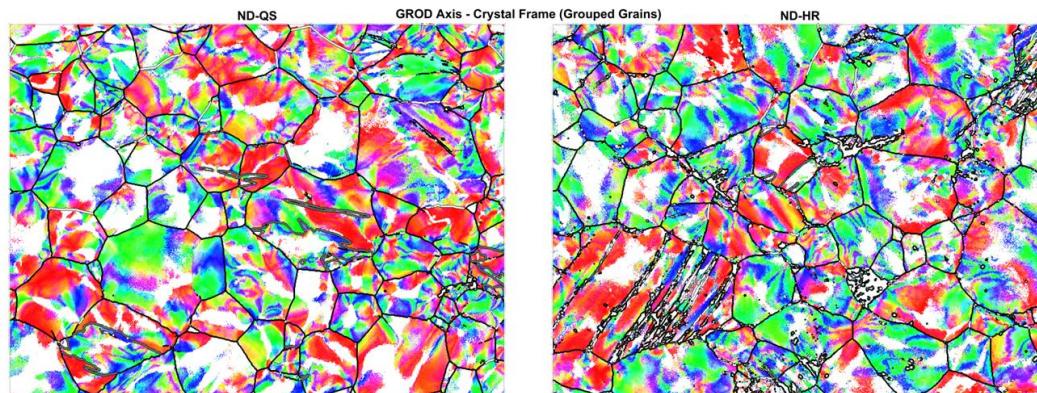
# MULTIPLE SLIP SYSTEMS

- Multiple slip mechanisms = difficult to allocate slip system in crystal frame
- In sample frame – patchy, but consistent in colour
  - Indicates ~similar strain state

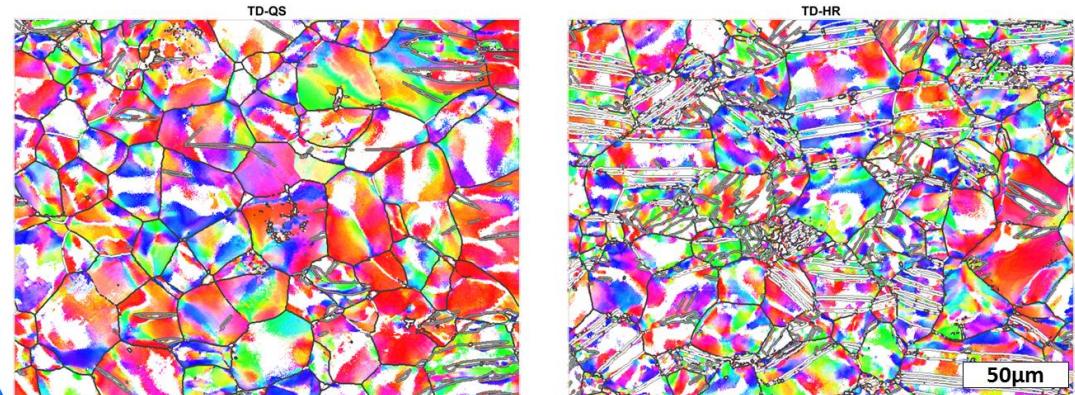
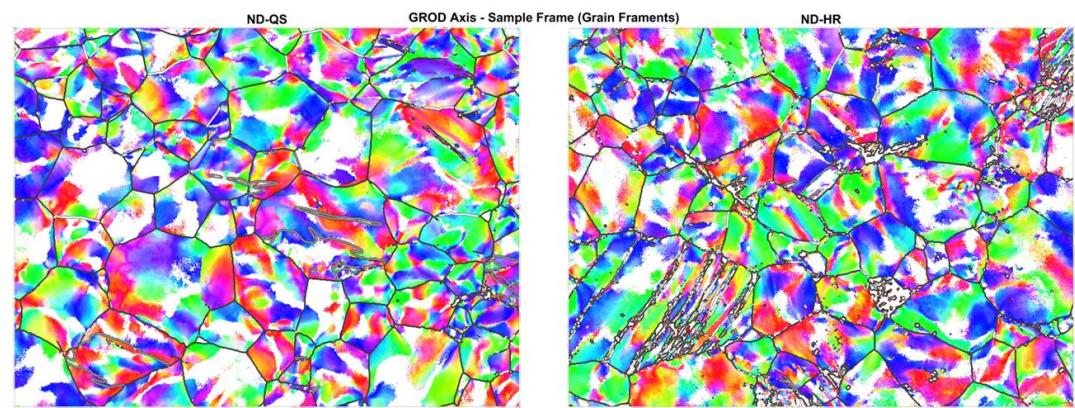


# ORIENTATION AXIS - FRAMES

Crystal

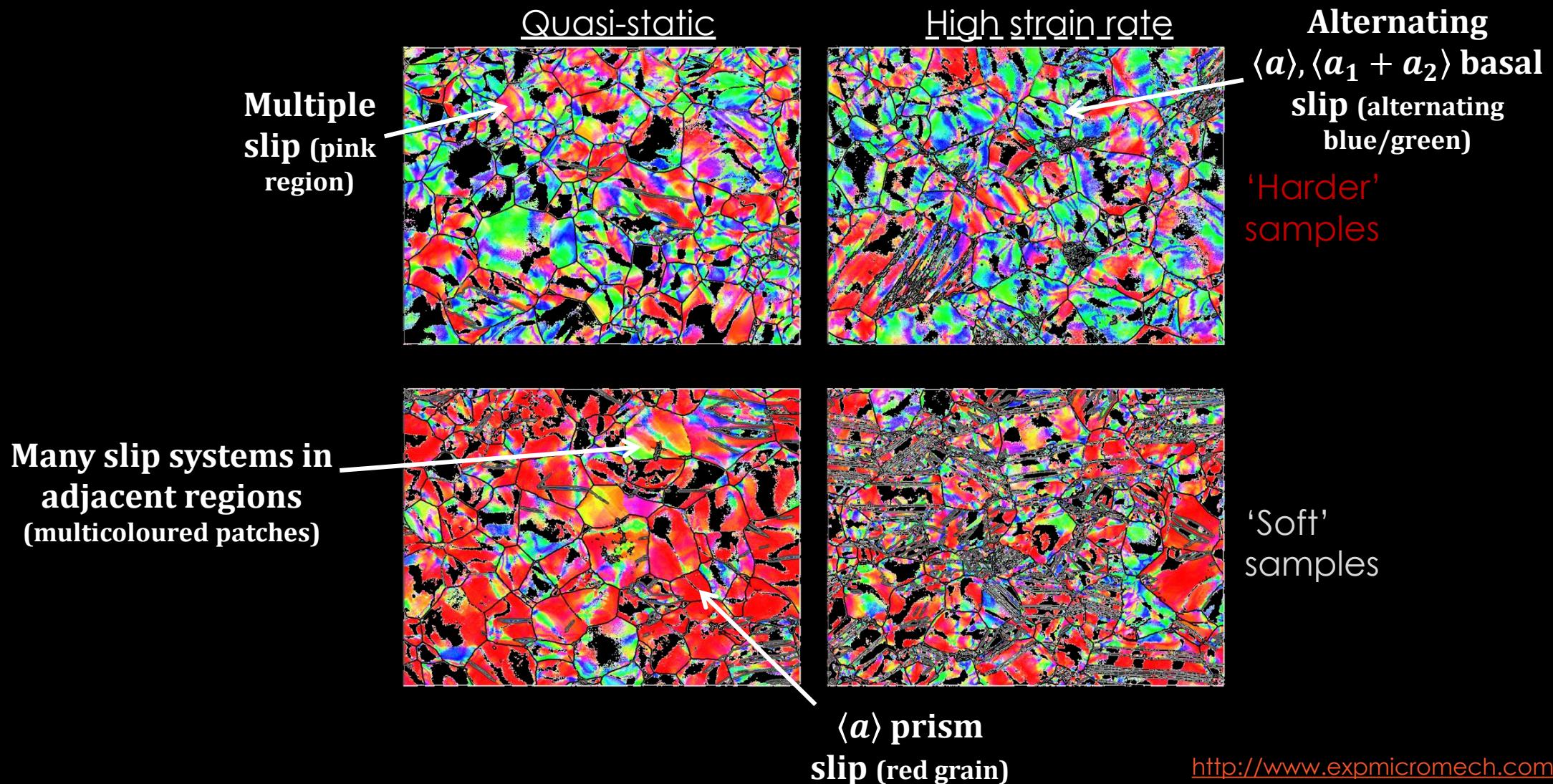


Sample



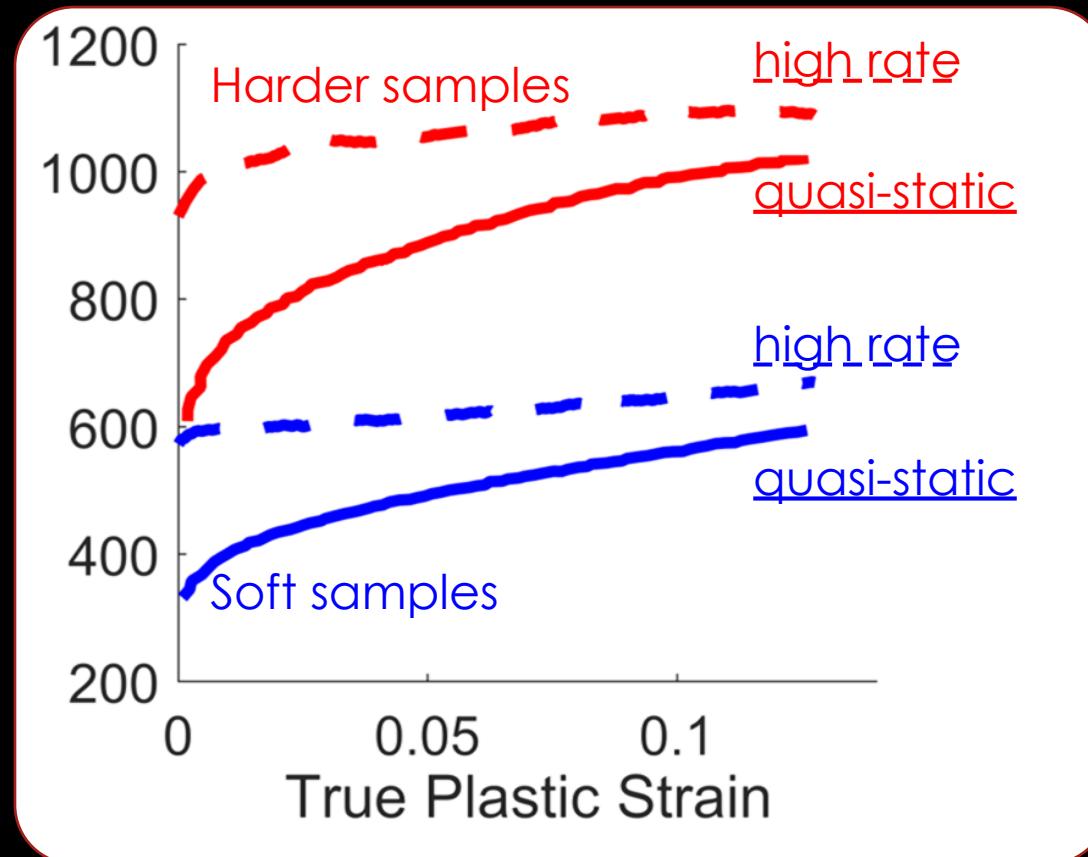
White regions < 2° from mean orientation

# MAP-WIDE SLIP SYSTEM ANALYSIS (CRYSTAL FRAME)



# SLIP SYSTEM ACTIVITY

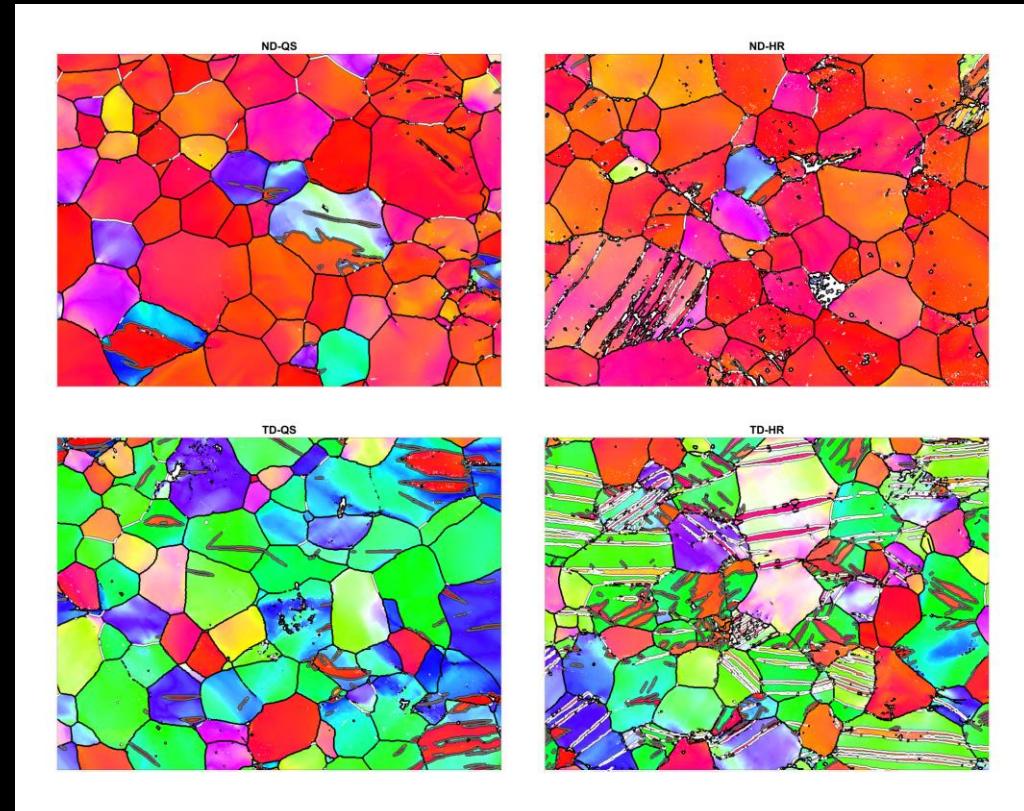
True Stress (MPa)



GROD20	Prismatic	Basal	Mix	Twining
Hard-QS	8%	34%	58%	T1
Hard-HR	6%	42%	51%	Many T2+T1
Soft-QS	34%	12%	54%	T1
Soft-HR	19%	20%	60%	T2+T1

# SUMMARY – HIGH RATE

- Texture + strain rate changes deformation modes
- EBSD enables twin AND slip system assessment
- Slip = Assumption of constraint
  - Requires large deformations
  - Assesses long range rotation gradients



# IF STEEL DEFORMATION

Jim Hickey and Ben Britton

(Work in process – linked to MTEX tutorial...)

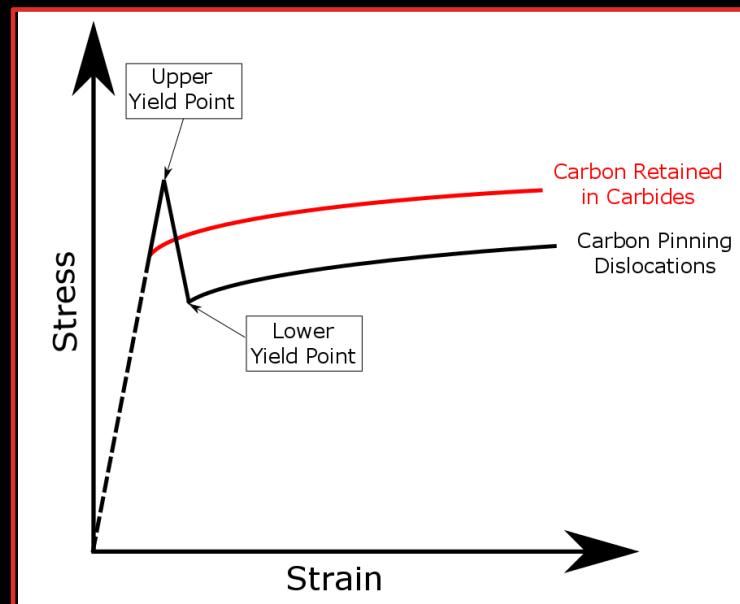
# CASE STUDY & INITIAL HYPOTHESIS: IF STEEL

**Interstitial Free (IF) Steel:** Carbon atoms located in **carbides** as opposed to ferrite matrix

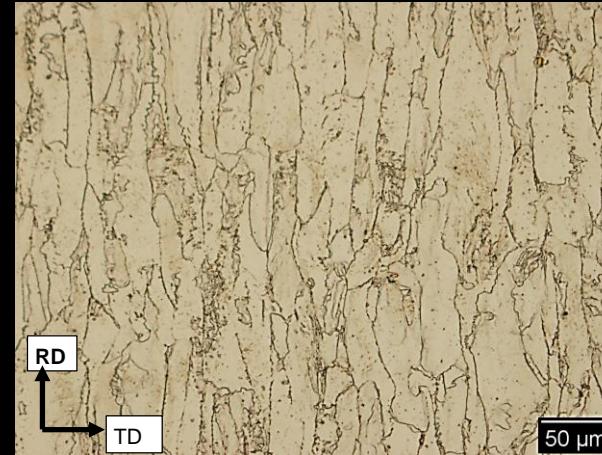
**Sample:** Cold rolled, Ti/ Nb (carbide forming) sheet  
IF steel 0.8 mm thick

**Task:** Design two heat-treatments: one to dissolve carbides, the other to retain them

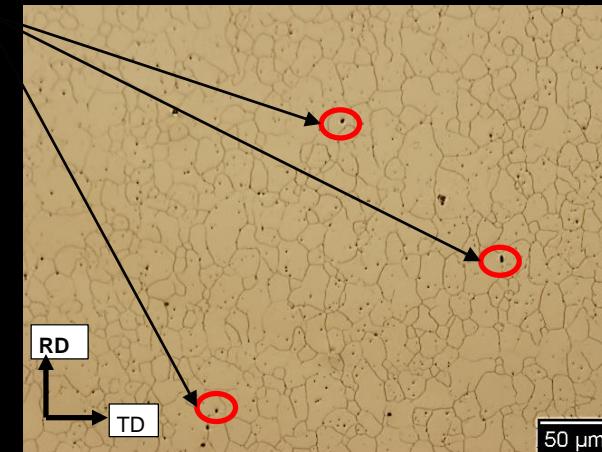
**Hypothesis:**



Carbides

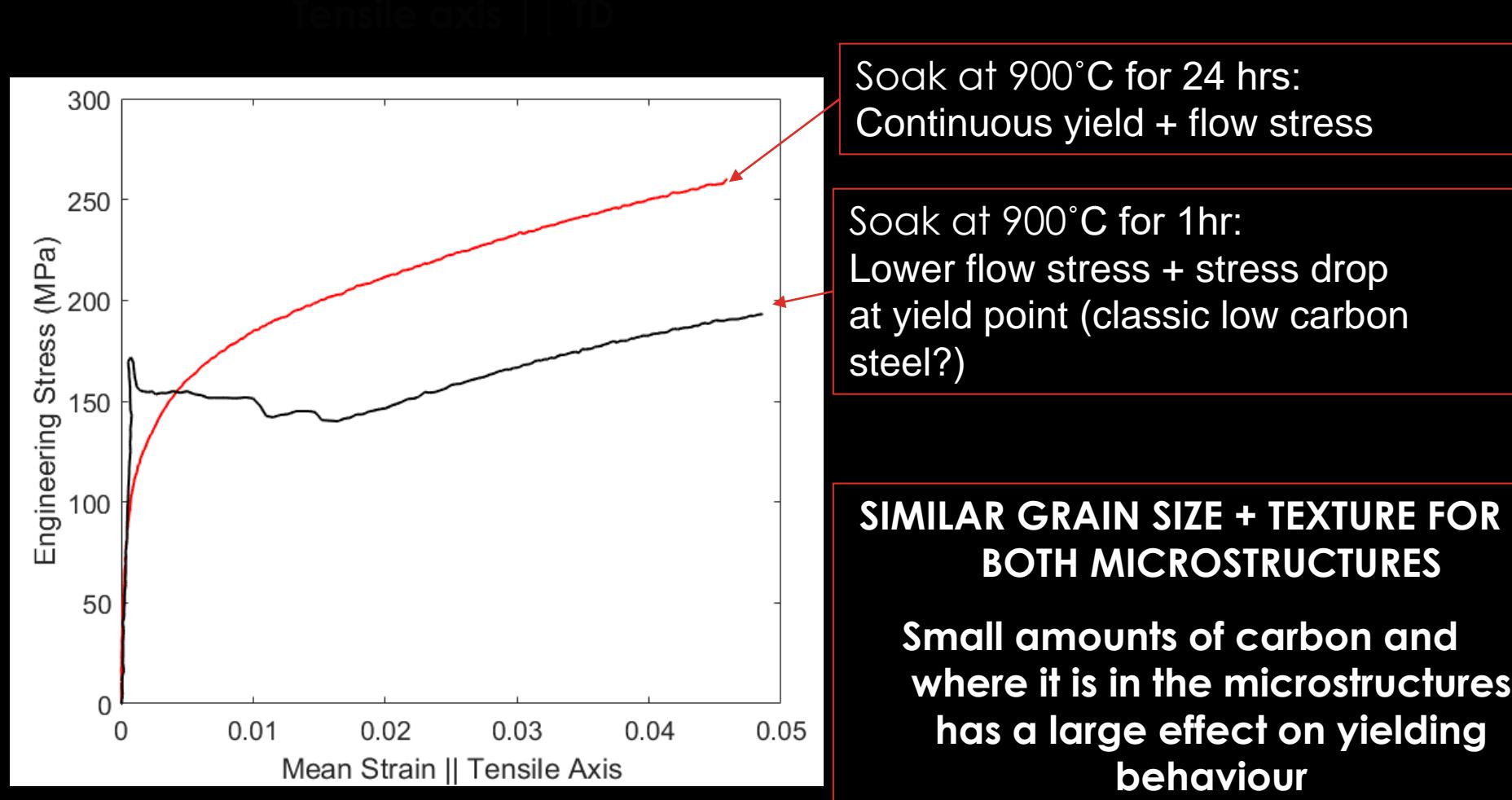


Micrograph of as-received rolled surface of IF steel sheet

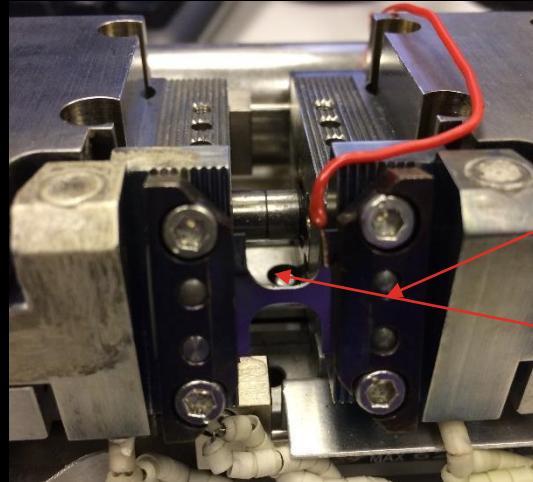


Micrograph of as-received rolled surface of IF steel sheet showing carbides highlighted by red circles

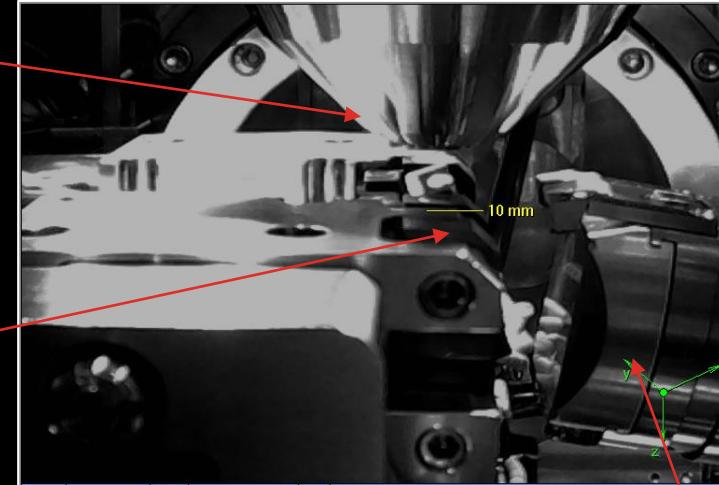
# EX-SITU TENSILE RESULTS I: RECRYSTALLIZATION FROM AUSTENITE



# IN-SITU I: HETEROGENEOUS YIELDING SAMPLE

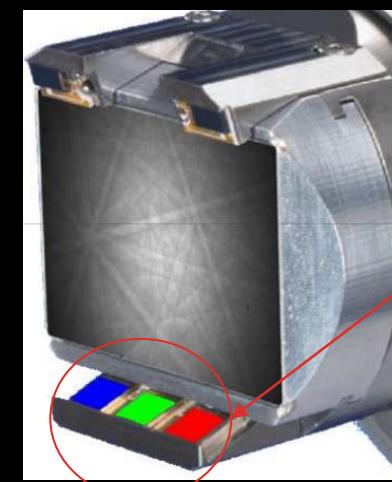


Pole Piece  
Tensile Stage Jaws  
Sample



EBSD Detector

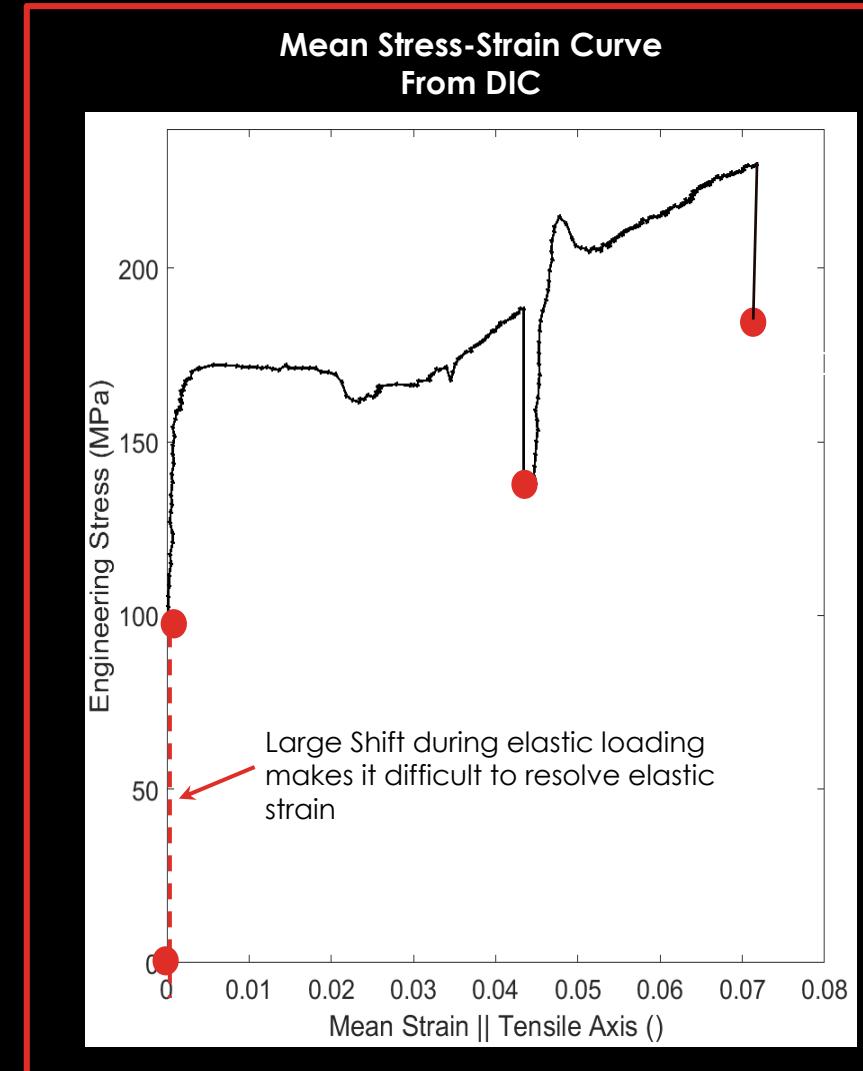
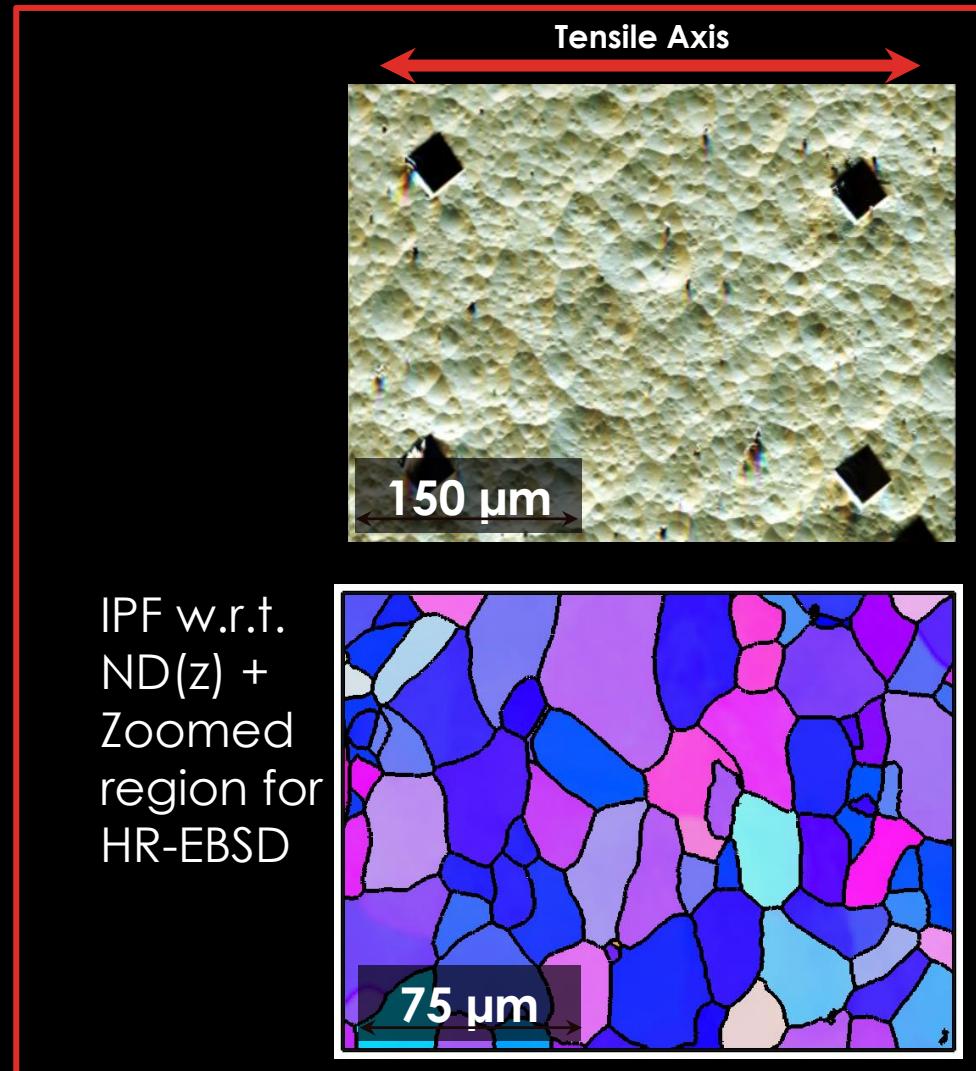
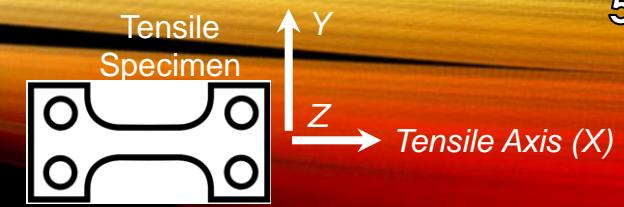
FSD Diodes:  
Topographical  
Contrast



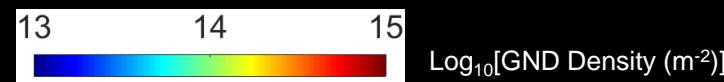
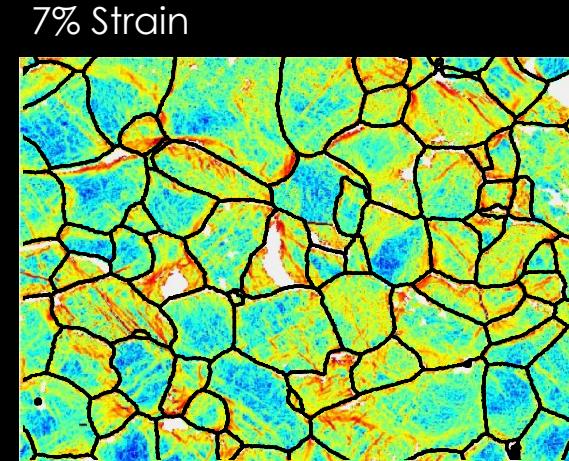
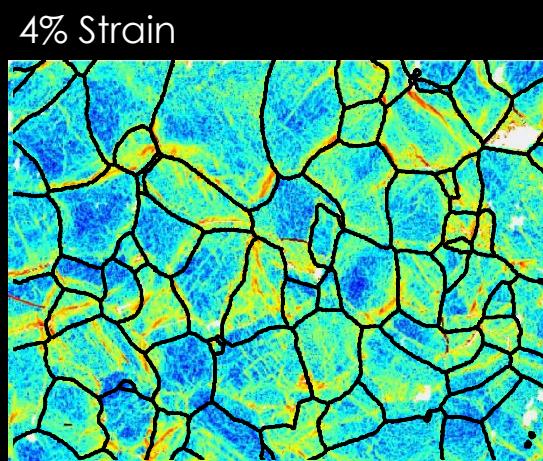
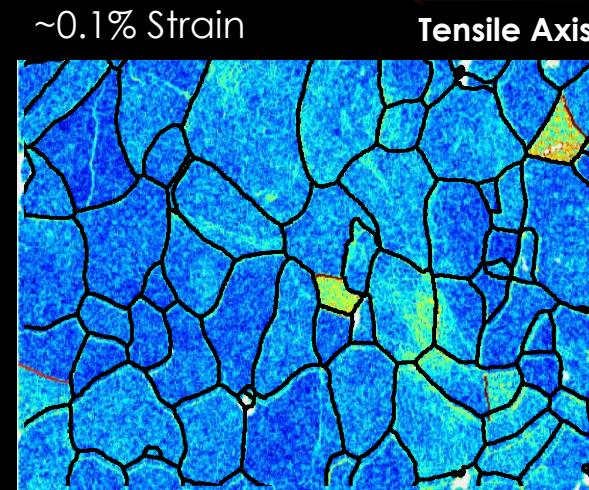
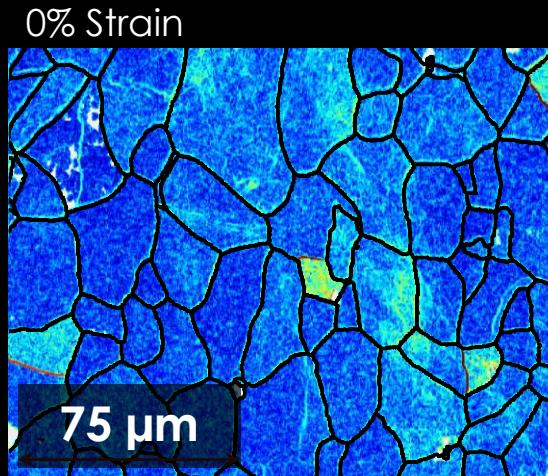
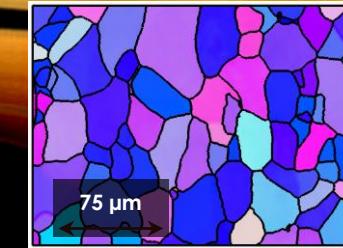
FSE diodes image courtesy  
of Daniel Gaily

- Experiment to:
  - Measure Geometrically Necessary Dislocation (GND) density using HR-EBSD
  - Measure strain using DIC + Markers (Indents + Roughness)

# IN-SITU II: DIC



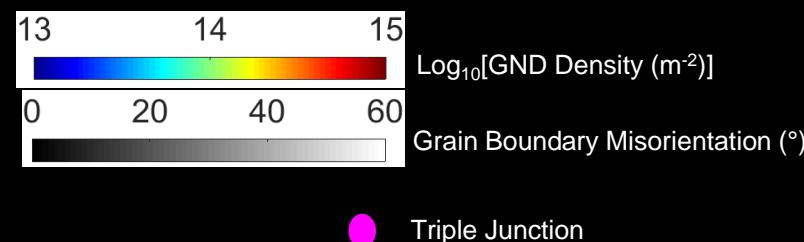
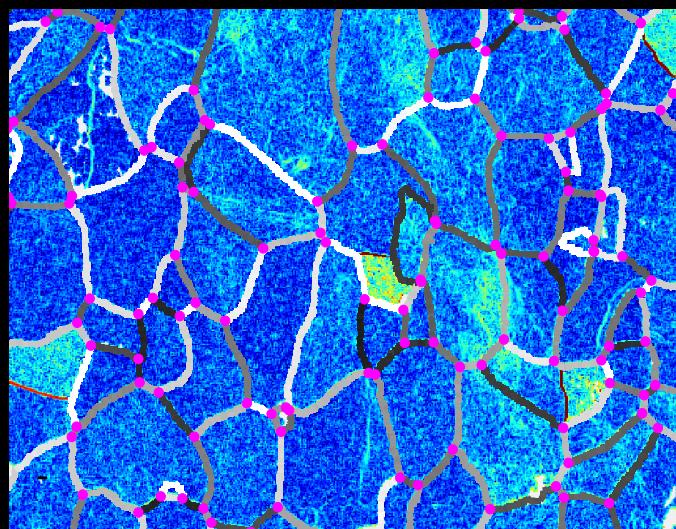
# HR-EBSD RESULTS: GEOMETRICALLY NECESSARY DISLOCATIONS (GND) DENSITY



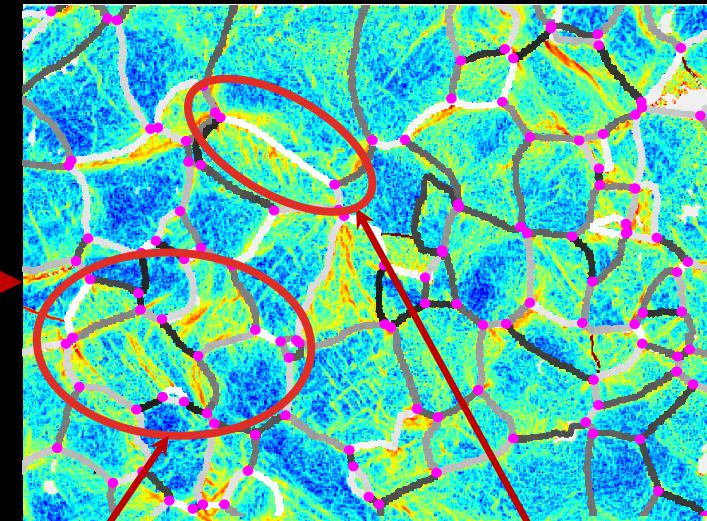
- Step Size: 0.4  $\mu\text{m}$
- $\rho_{\text{GND}}$  increases with strain
- Heterogeneous  $\rho_{\text{GND}}$  field
- Intragranular bands of high  $\rho_{\text{GND}}$  at  $\sim 45^\circ$  to the tensile axis.  
Maximum shear strain
- Challenge in identifying an EBSP per grain to act as a reference point
- **High  $\rho_{\text{GND}}$  at some grain boundaries**

# GRAIN BOUNDARY MO & TRIPLE JUNCTIONS CORRELATIONS

0% Strain



4% Strain



High GND density in regions with high concentration of triple junctions

Development of high GND density at grain boundaries with high MO

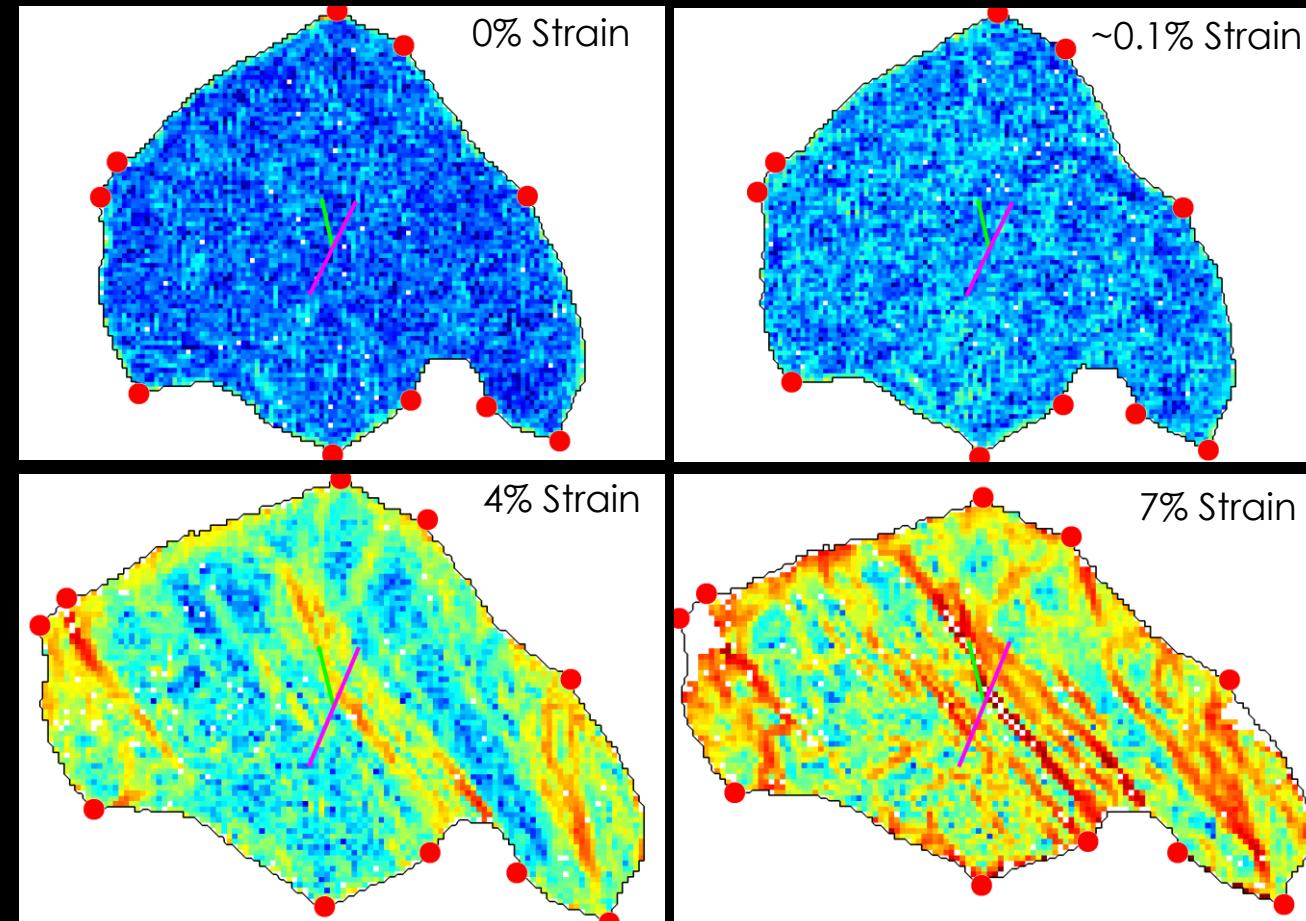
Can these supposed relationships be quantified?

# GRAIN TRACKING – GND EVOLUTION

Does the GND network track with:

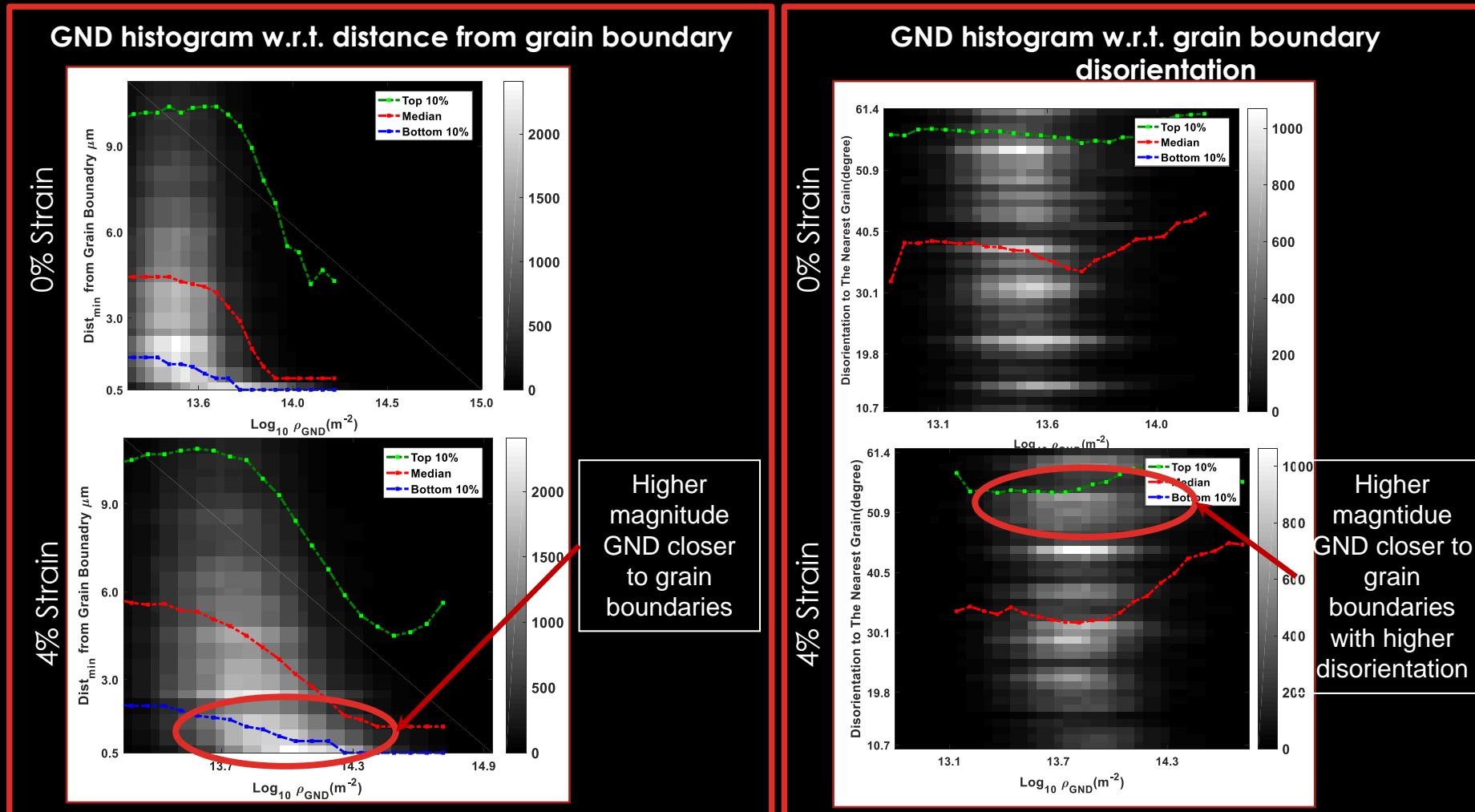
- (a) Crystal orientation,
- (b) Constraint  $\rightarrow$  GB or Triple Junction

Can focus our analysis within a map...

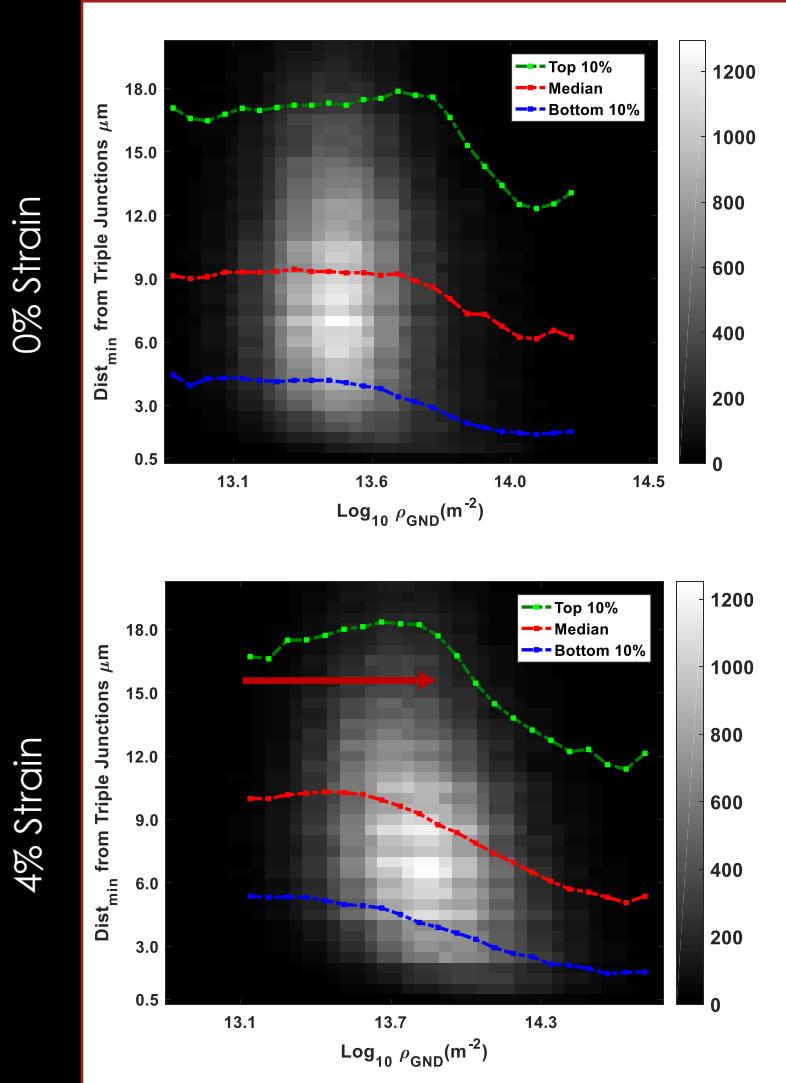


# GRAIN BOUNDARY CORRELATIONS

- Bin GND Data and calculate Euclidian distance from grain boundaries



# TRIPLE JUNCTION CORRELATIONS



- Higher magnitude GND closer to triple junctions
- Despite texture of sample, distribution of GNDs appears to be determined by grain boundaries & triple junctions
- Sample texture does not appear to have a large effect on GND (when sample is pulled parallel to the initial plate TD)
- Cannot observe effect of carbides

# IF STEEL SUMMARY

Observed the same field during deformation

Enables evolution of GND density (+more) to be correlated with microstructure

Quantitative assessment of correlative fields enables mechanistic insight  
Correlate microstructure features & mix EBSD data to provide new information

# ADVANCING EBSD ANALYSIS

EBSĐ data is a quantitative stepped measurement of the crystal shape, phase, and crystal orientation

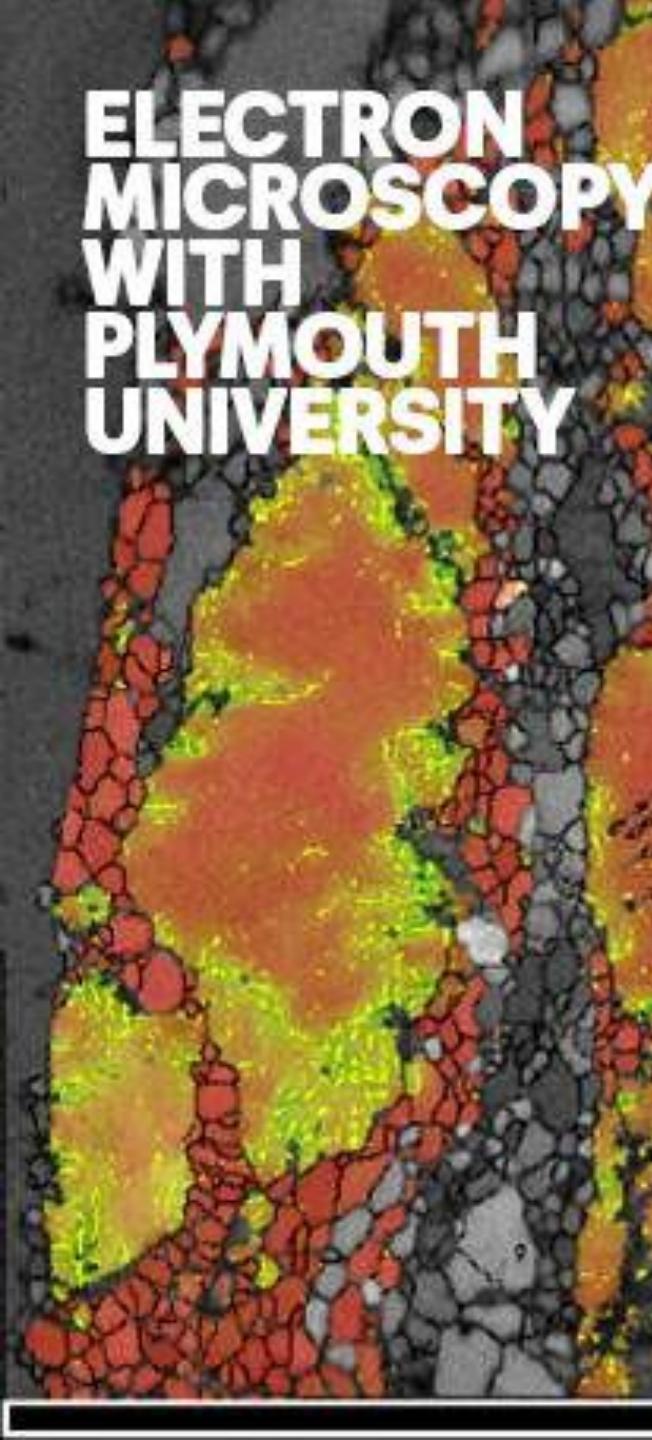
Care with understanding what your data looks like, what it can tell you, and its accuracy + precision

HR-EBSD enables probing of the lattice strain variations & precise measurement of the stored GND density

For high quality analysis → use quantitative, data-based, analysis for your advantage

Often MTEX solutions will help but you can mix signals & perform new calculations if needed...

Build your analysis to probe a mechanisms (ha ha!)



# ELECTRON MICROSCOPY WITH PLYMOUTH UNIVERSITY

## RMS EBSD 2018

10<sup>th</sup> & 11<sup>th</sup> April

University of Plymouth, UK

Confirmed speakers:

- Sandra Piazolo (Leeds)
- Paul Midgley (Cambridge)
- Mike Zolensky (NASA, Johnson)
- Ralf Hielscher (TU Chemitz)



# QUESTIONS?

