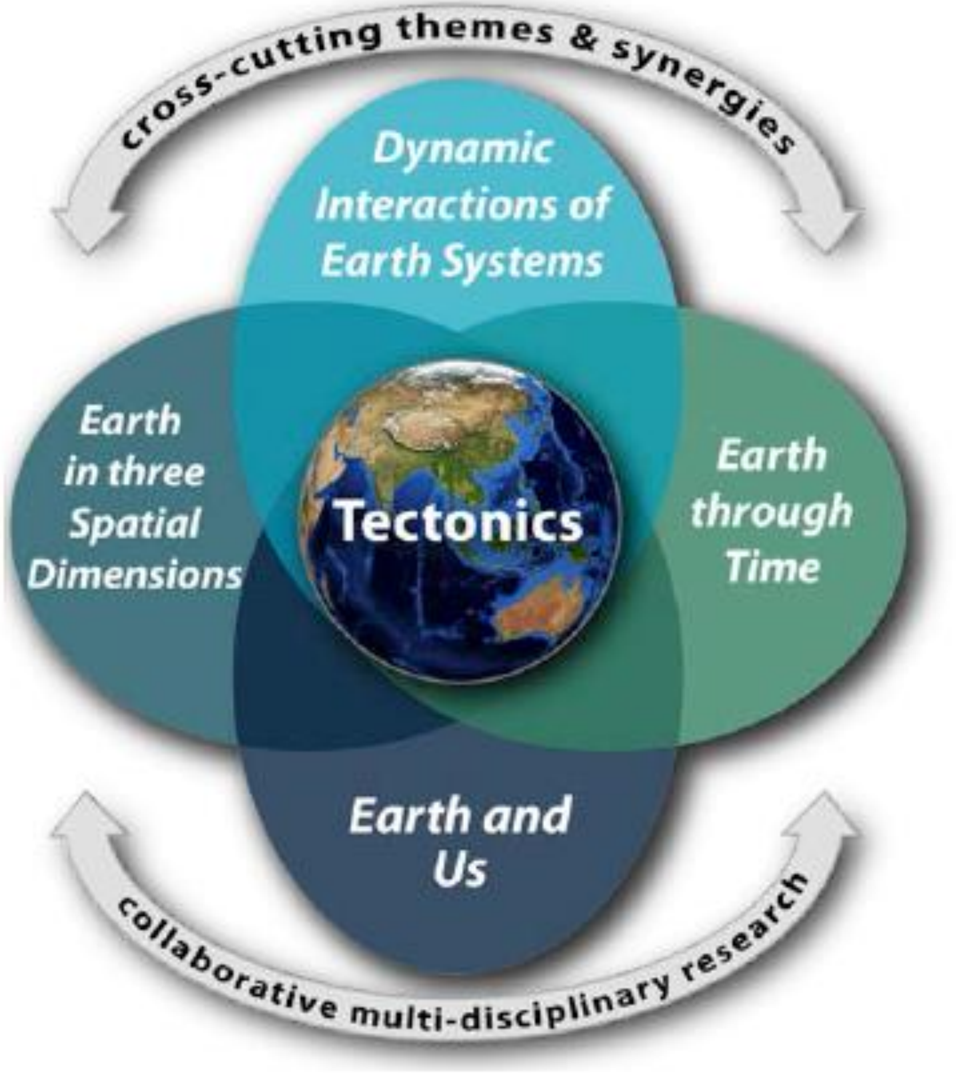
Rheology of the Lower Crust:
Concepts, Methods, Observations

1) Lecture 1: Rheology of the Lower Crust: General importance & Recap of Solid state deformation mechanism and flow laws

2) Quantitative Orientation Analysis: How does it work? How can it help me to understand the Lower Crust – rheology

3) Quantitative Orientation Analysis: Examples and Opportunities - Rheology and evolution of the Lower Crust

4) Rheology of the Lower Crust: Other measurements and considerations



Modified after Huntington & Klepeis et al. 2017

Lecture 2: Quantitative Orientation Analysis: How does it work? How can it help me to understand the Lower Crust – rheology and evolution

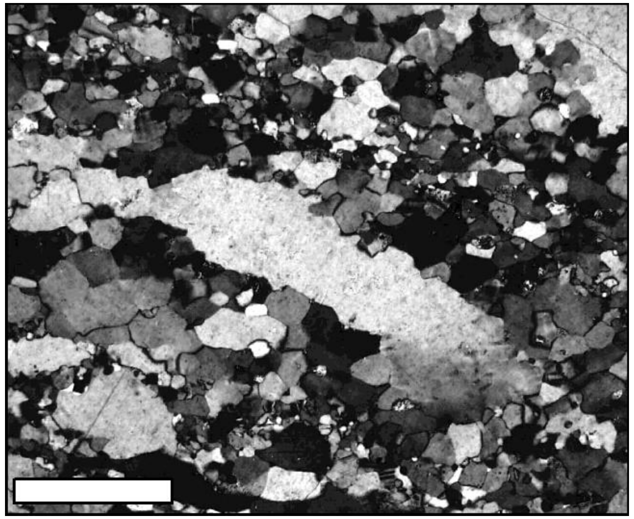
1. Quantitative Orientation Analysis – Why?
2. Quantitative Orientation Analysis – Measurement methods
3. Focus on EBSD - What is EBSD?
4. How does it work?
5. What can EBSD tell us?
6. Examples - general

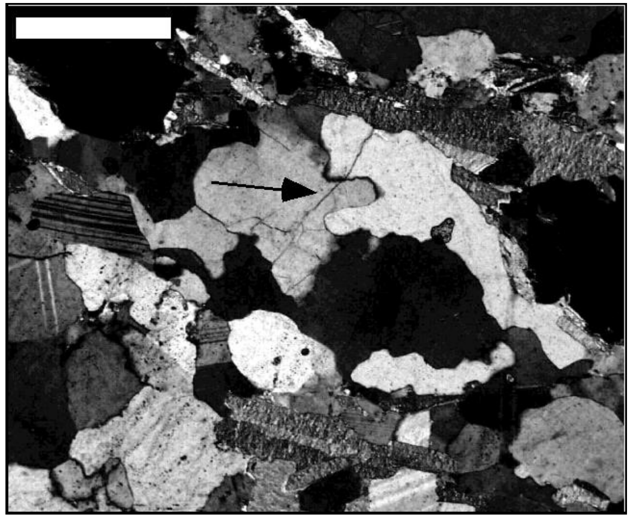
Recap

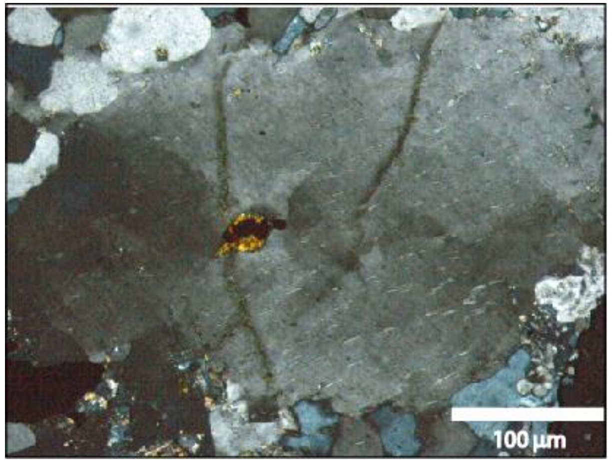
Why do we study microstructures?

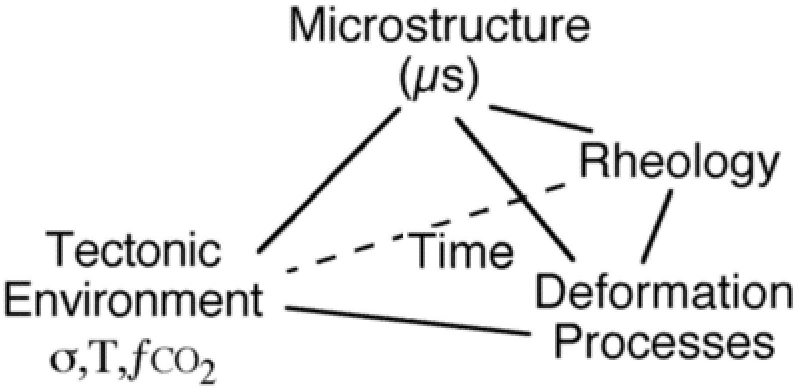
1) By understanding/interpreting how a microstructure forms we can derive the conditions under which it has formed

2) As grain scale processes control the macroscopic behaviour of rocks, microstructures can tell us about the processes that led to the microstructures -> flow law/rheology of a rock









Remember: Deformation by movement of lattice defects

Know your processes

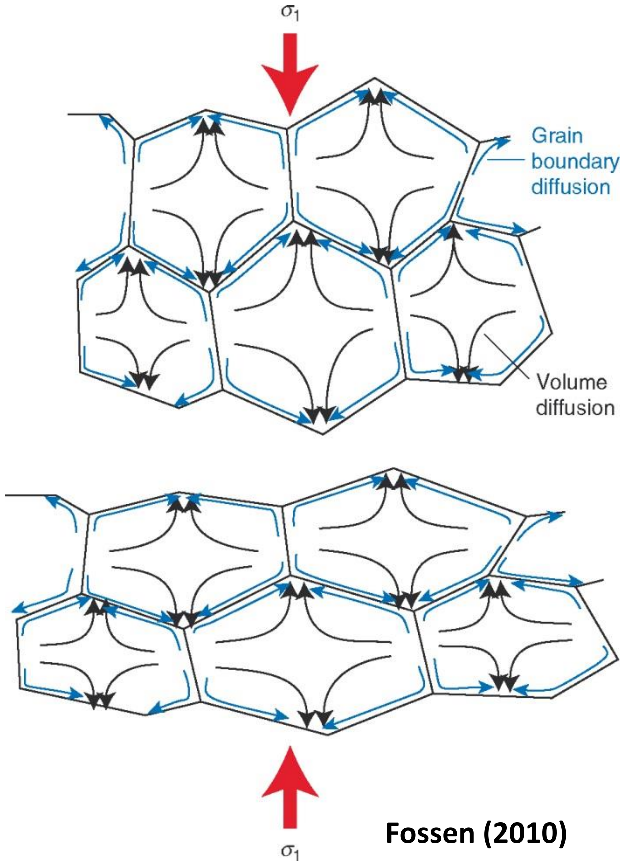
- Point defects /Vacancies

  Diffusion creep

  - **Nabarro-Herring creep** - through
  - **Cobble creep** – around
  - **Pressure Solution**

  / Dissolution-precipitation

- Line defects
  - **Dislocation glide/creep**



Fossen (2010)

Effect on microstructure

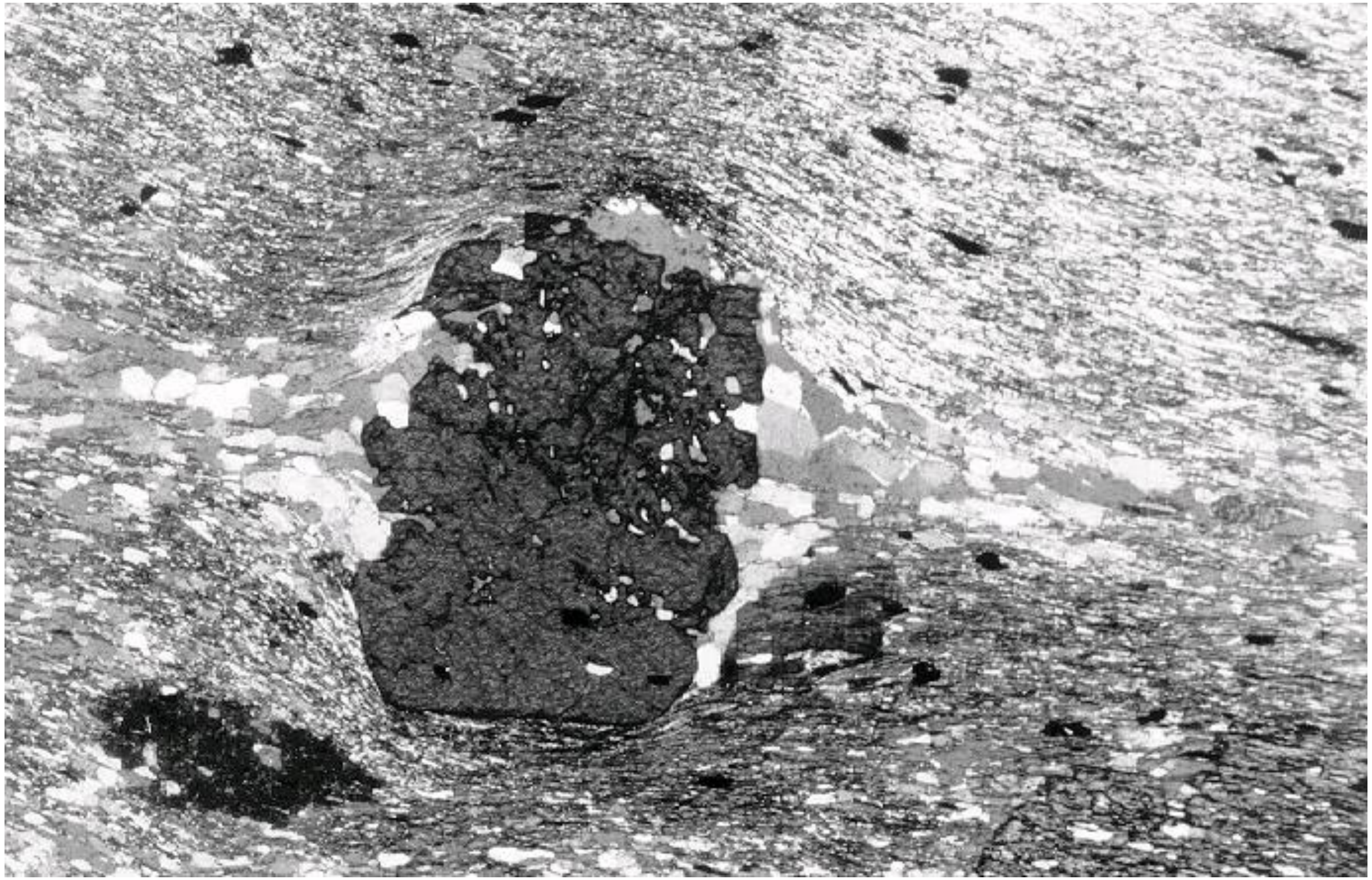
(Physical & chemical)

Examples

Local stress variations during deformation -> dissolution-percipitation creep?

(Winsch et al., 2001)

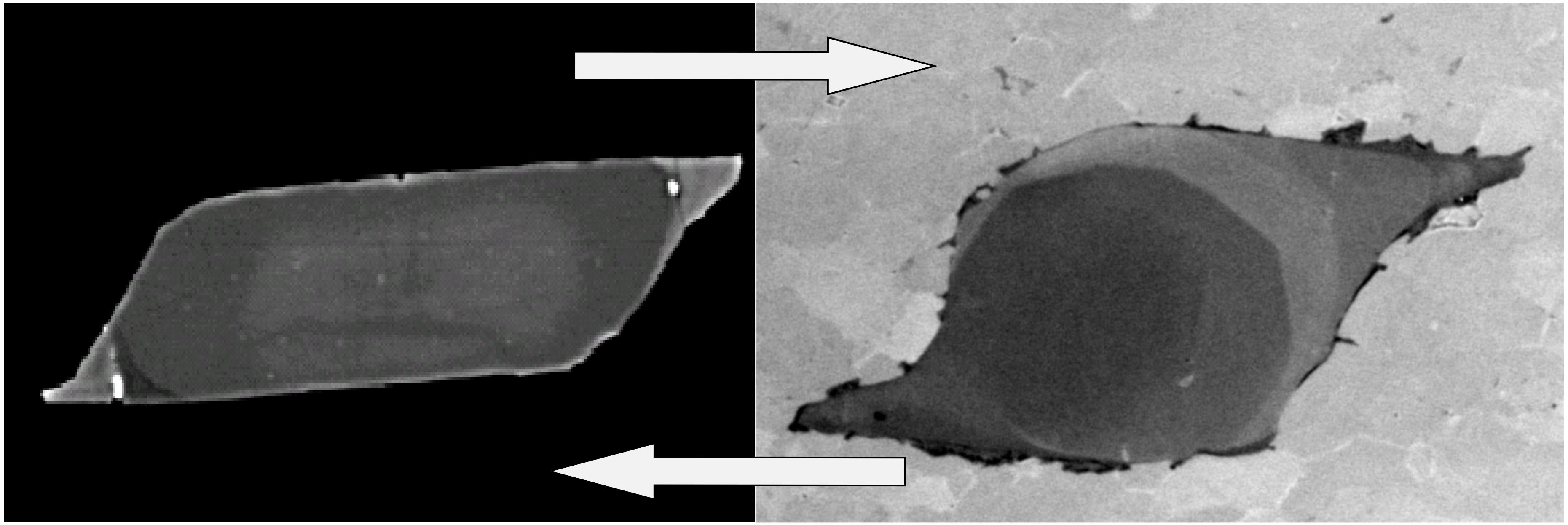
mm
to
cm
scale



mm
to
cm
scale

Tourmaline / Qtz – fish:
Formation due to differential stress
-> dissolution-percipitation creep?

CL images



Tourmaline

Quartz

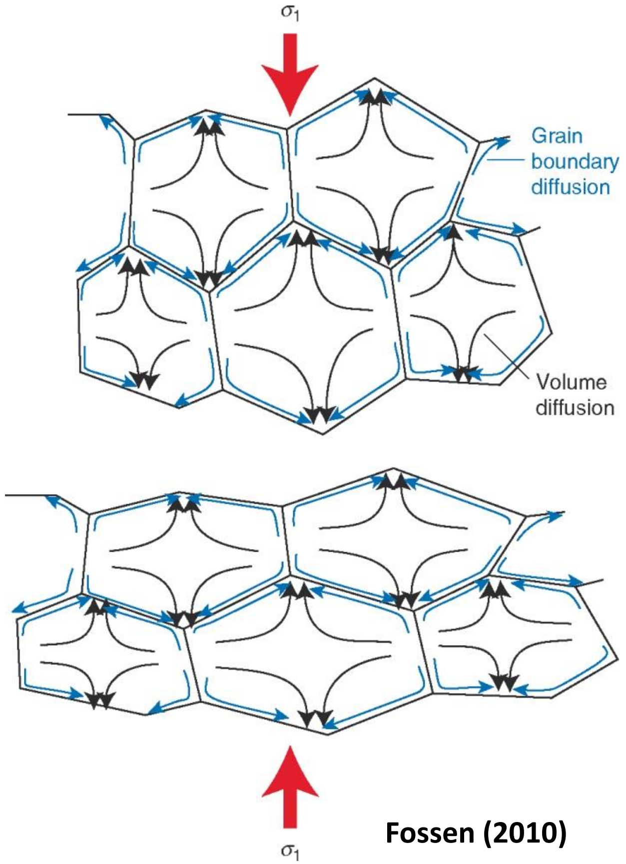
Bestmann et al. JSG, 2003

Note: If you date -> where to put your spot analysis for original and deformation??

Remember: Deformation by movement of lattice defects

Know your processes

- Point defects /Vacancies
  - ***Diffusion creep***
  - **Nabarro-Herring creep** - through
  - **Cobble creep** – around
  - **Pressure Solution / Dissolution-precipitation**
- Line defects
  - **Dislocation glide/creep**



Fossen (2010)

Effect on microstructure

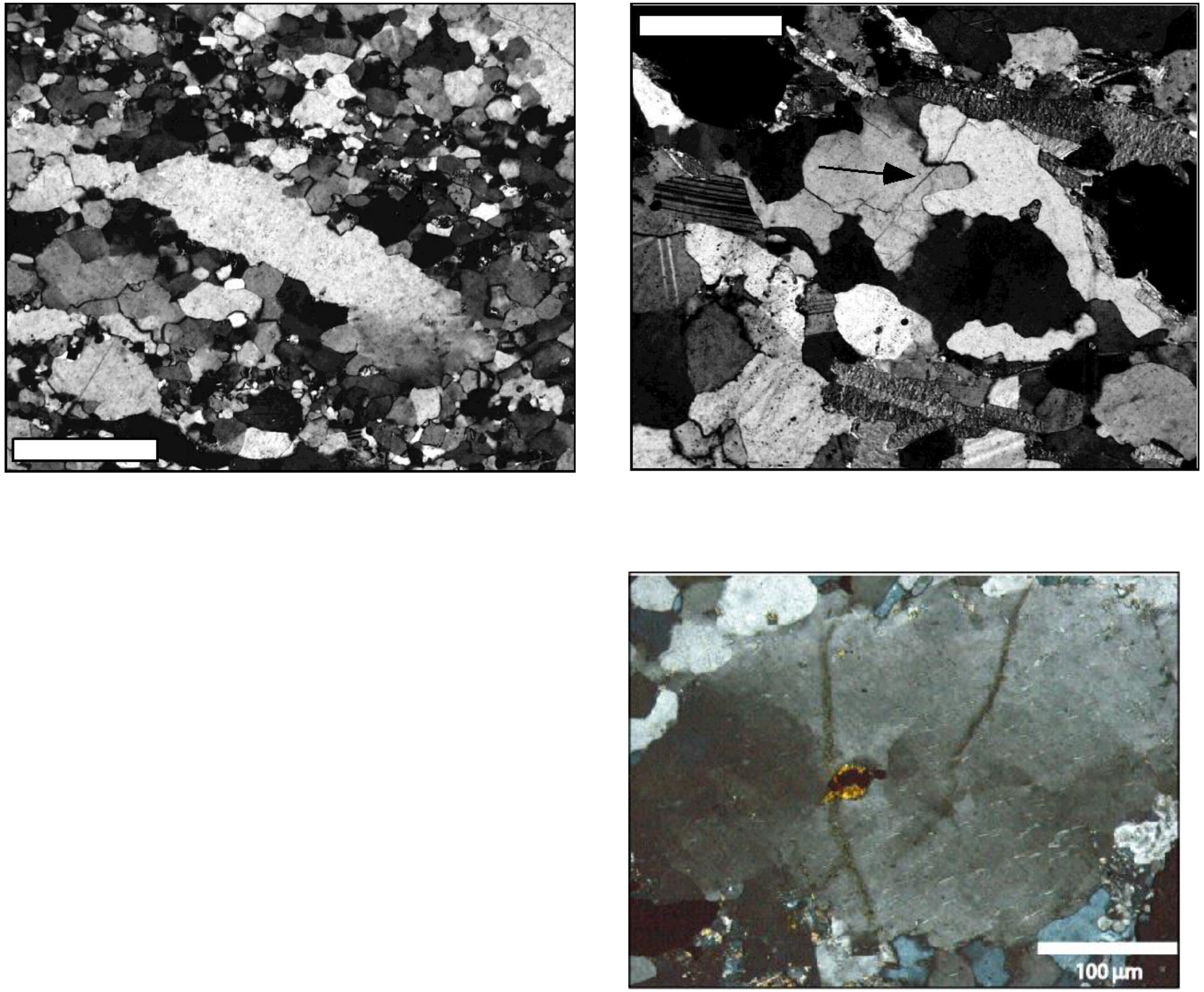
(Physical & chemical)

Different effects on microstructure -> quantification needed

Going beyond description

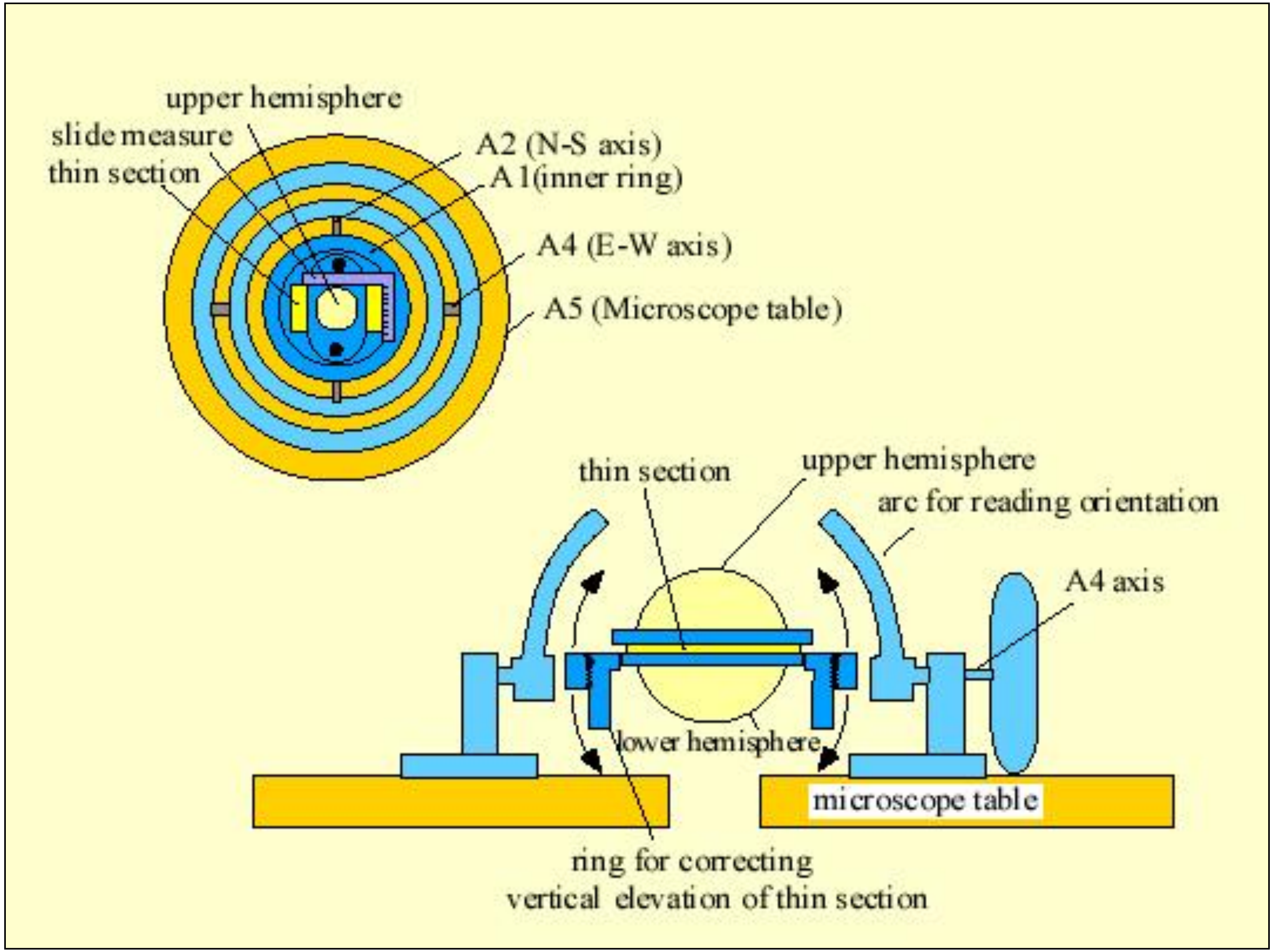
How can we measure crystallographic orientations: Measurement methods

- Universal stage
- CIP
- Texture Goniometry
- TEM
- EBSD (detail later)



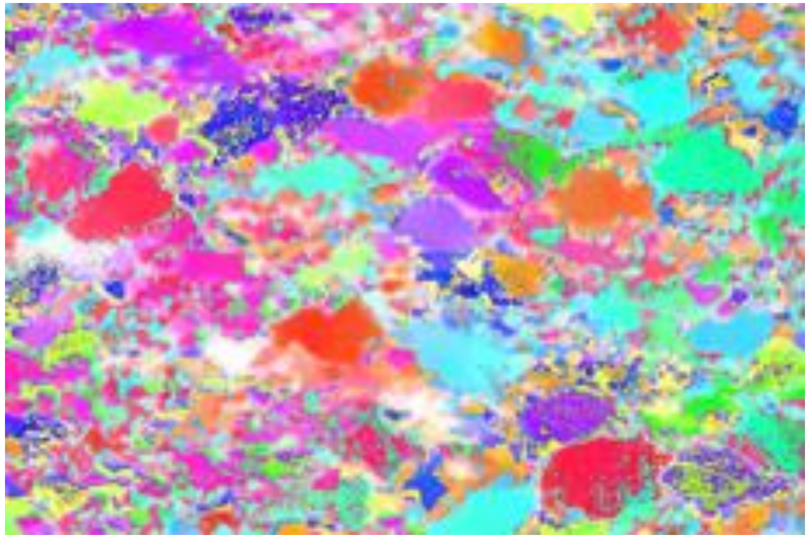
U(universal) -stage

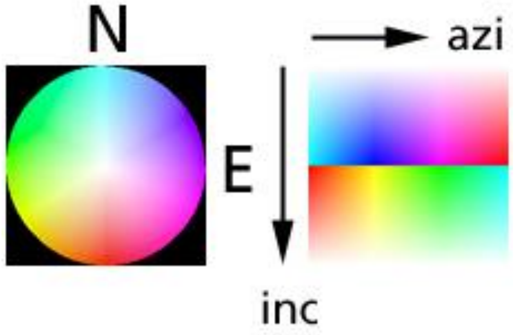
- traditional way: - uses extinction angle of crystal



Computer Integrated Polarization Microscopy (CIP)

Renee Heilbronner – several papers 1995-2005





- Images are taken every 15 degrees rotation
- Colour-coding can be obtained that is unique with respect to c-axis orientation, i.e. the orientation halfspace.
- To map the two-dimensional orientation space, i.e., to uniquely colour-code c-axis orientations, azimuth and inclination images are calculated and treated as two channels of a colour image.
- Two-dimensional colour look-up tables (CLUTs) are used to assign unique colours to any given pixel depending on the azimuth and inclination values of the c-axis at that point.
- Orientation of its c-axis varies gradually from dipping above the plane of the section to dipping below it.

Texture Goniometry

2 Types: - X-ray texture goniometers
- neutron texture goniometers

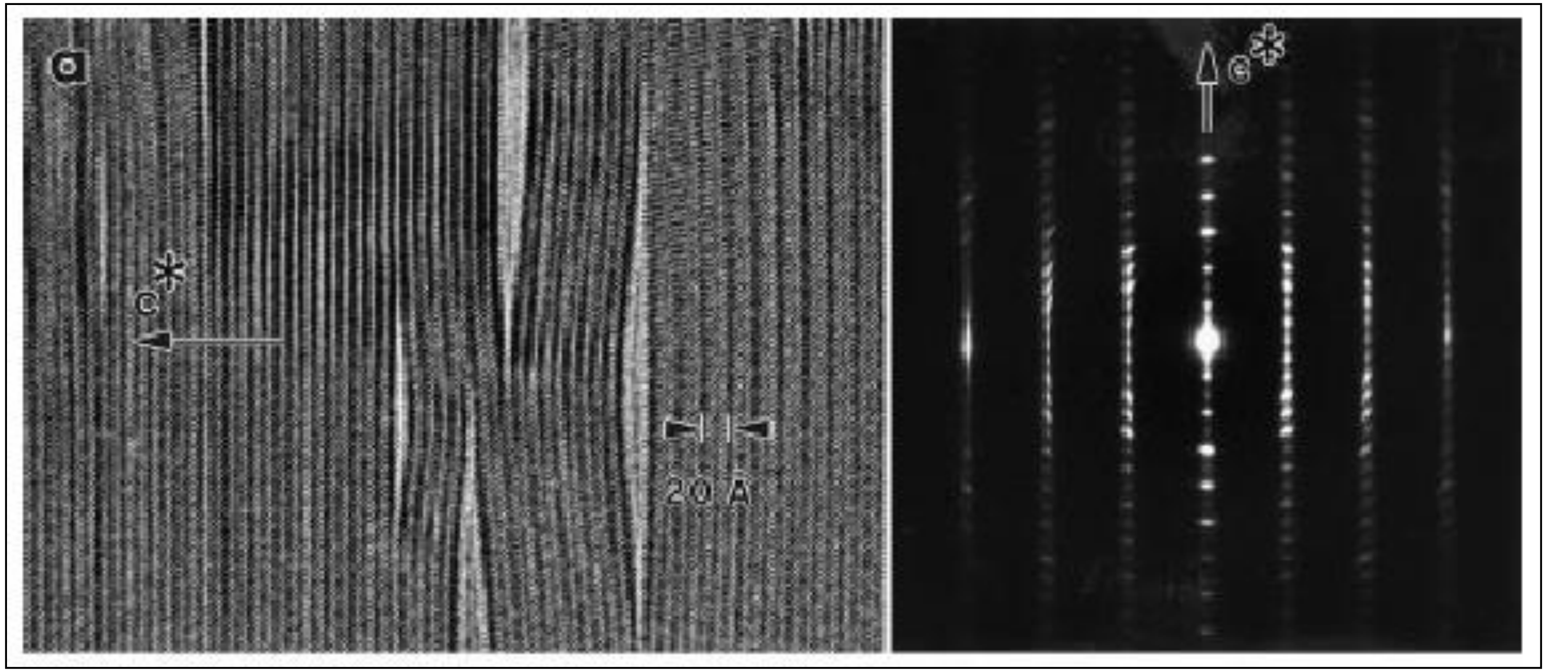
Advantages: - CPOs of large samples can be done
(Crystallographic Preferred Orientation)

Disadvantage: - no spatial relationship of crystallographic orientation and microstructure

TEM

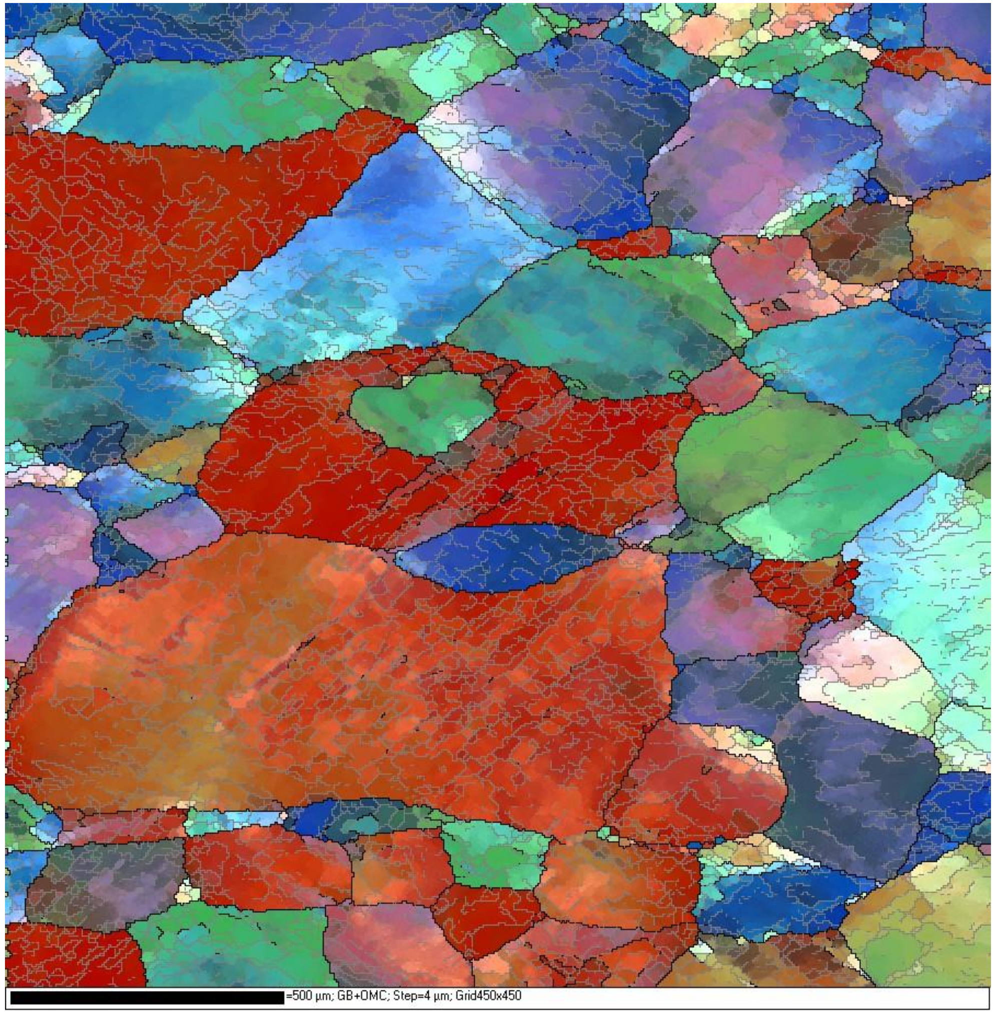
Transmission Electron Microscope

Higher resolution than SEM – but very very local – lack of statistics



EBSD

Electron BackScatter Diffraction



Special instrument in a SEM
(Scanning Electron Microscope)

Electrons are diffracted according to the crystallography of the crystal, the image of the diffraction is characteristic for the orientation of the crystal.
Automated analyses allows very rapid aquisition of crystallography data
(up to 3000pt/s (2019))

Gives spatial relationship of crystallography

Summary: Measurement methods

	Spatial Information (CPO linked to microstructure)	Full orientation (all axes)	Statistically robust
Universal stage	✗	✗	✗
CIP	✓	✗	✓
Texture Goniometry	✗	✓	✓
EBSD	✓	✓	✓
TEM	✓	✓	✗

What is EBSD?

EBSD – Electron Backscatter Diffraction

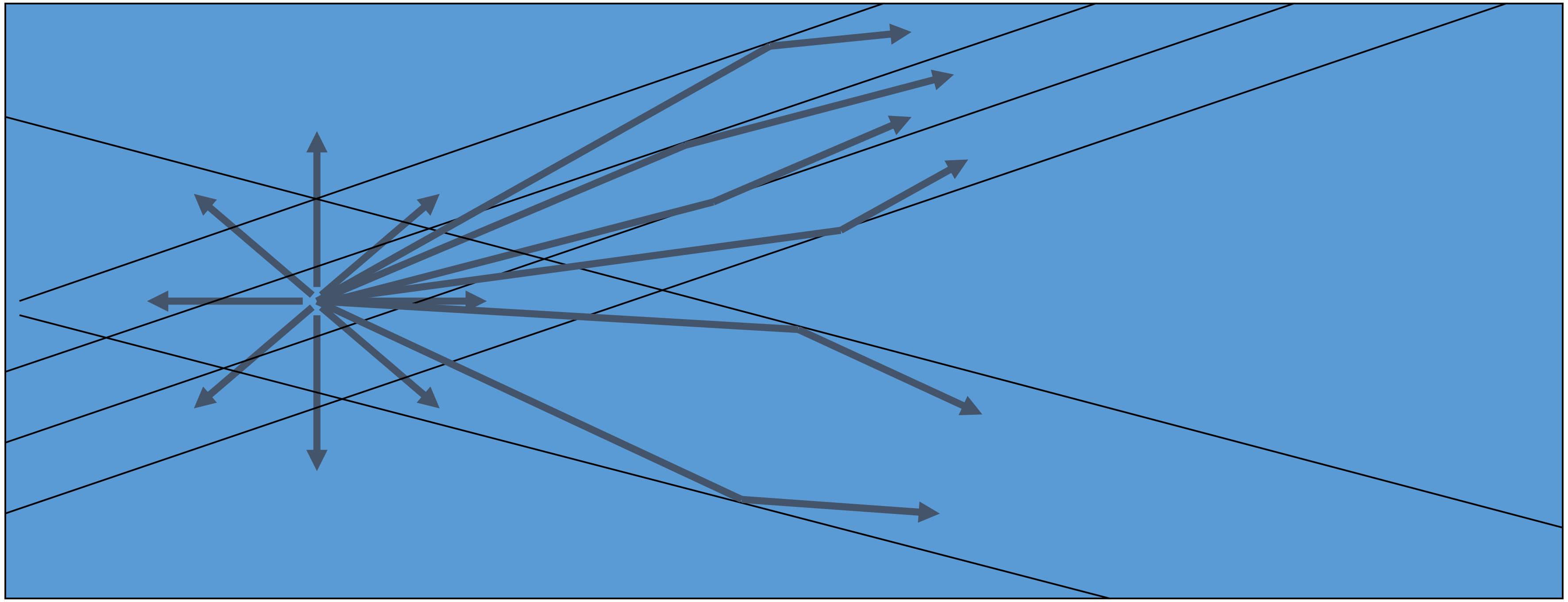
Other names:

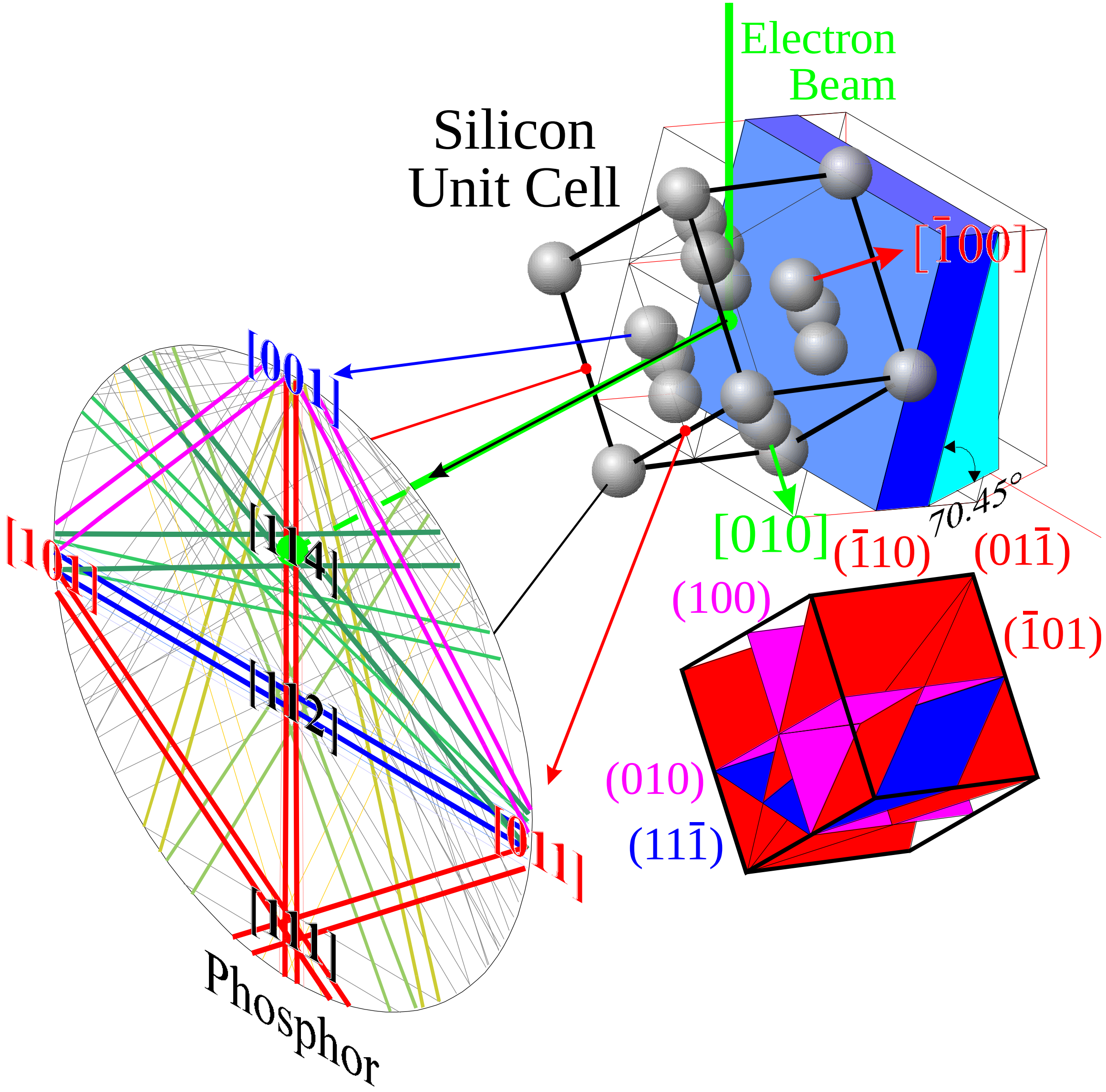
- EBSP – Electron Backscatter (Diffraction) Pattern
- BKD – Backscatter Kikuchi Diffraction
- OIM™ - Orientation Imaging Microscopy
- OM – Orientation Mapping
- COM – Crystal Orientation Mapping

EBSD is:

- a Scanning Electron Microscope (SEM) based technique
- a surface analysis technique
- a technique that can be used to analyse any crystalline material (e.g. metals, rocks, ceramics...)

If the material is crystalline, electrons will be diffracted by lattice planes where the Bragg condition is satisfied.



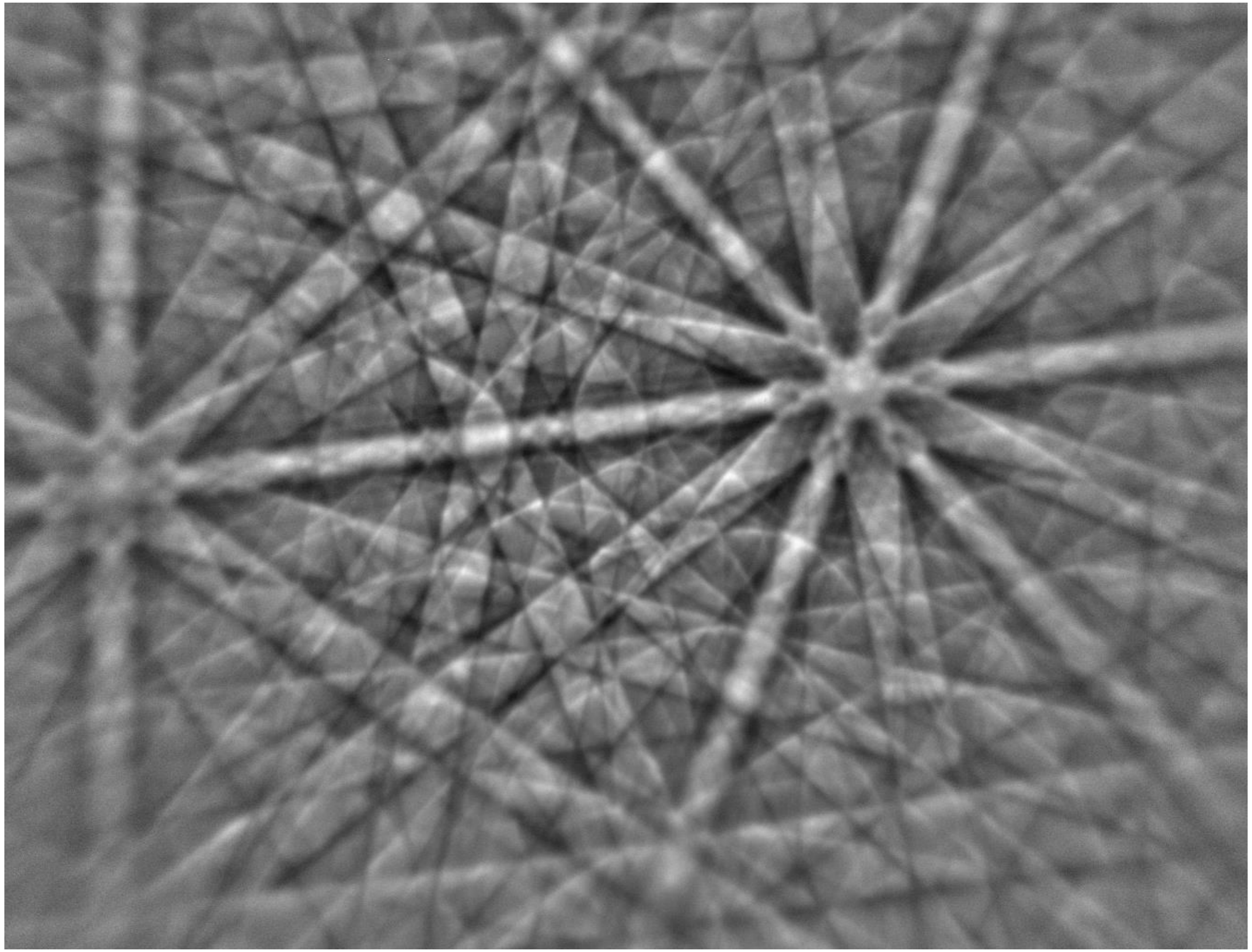


3D: each lattice plane (hkl) will give rise to two diffraction cones

For wavelengths associated with electron beams the cone opening angles are close to 180 degrees.

Austin Day 2000

Resulting EBSP...



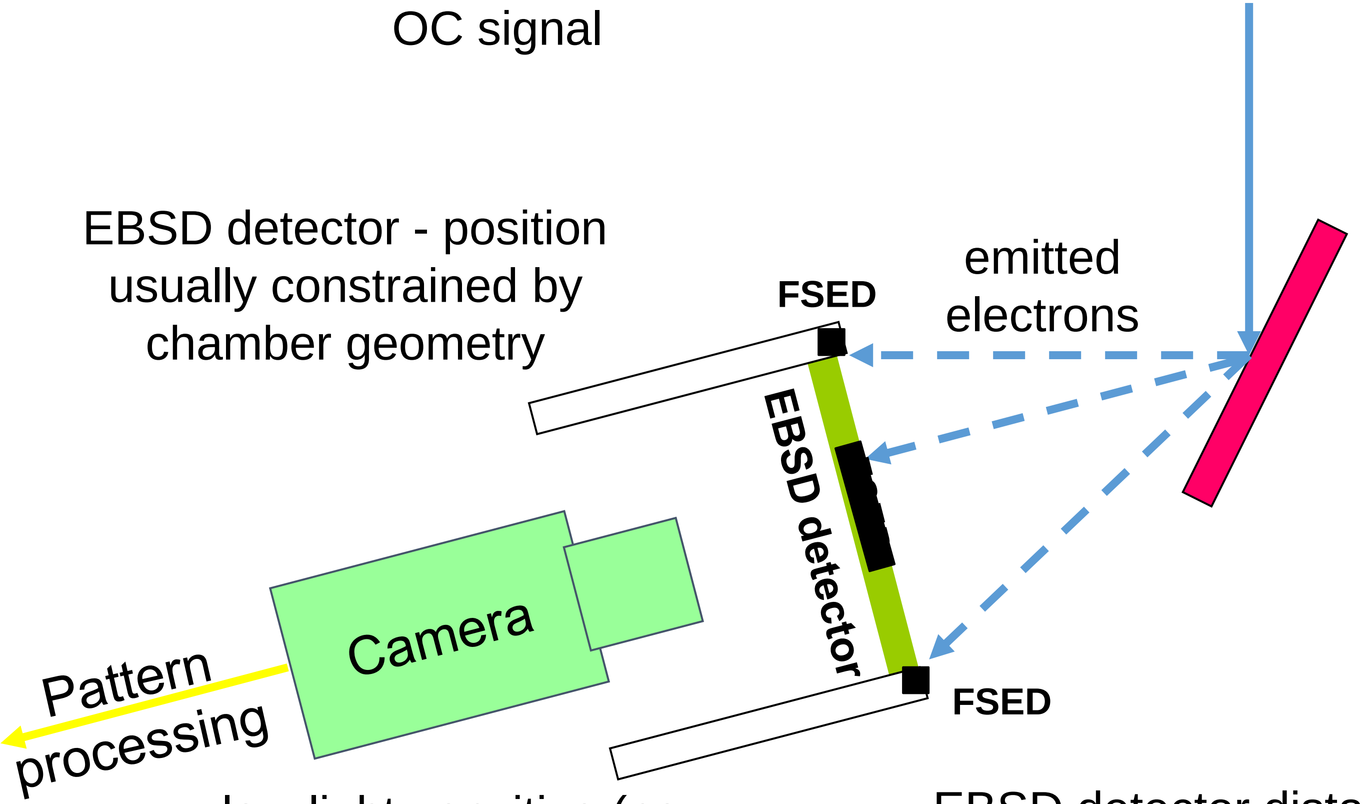
EBSD PATTERN RECOGNITION

- EBSD patterns are *unique* for a specific crystal orientation
- The pattern is controlled by the crystal structure: space group symmetry, lattice parameters, *precise* composition
- Within each pattern, specific ‘bands’ (i.e. pairs of ‘cones of diffraction’) represent the spacing of specific lattice planes (i.e. d_{hkl})
- EBSD pattern recognition compares the pattern of bands with an ‘atlas’ of all possible patterns in order to index the crystal orientation depicted
- This process WAS manual – it is NOW *automated* !

Typical SEM EBSD set-up

FSED: fore-scattered electron detectors capture OC signal

incident electron beam: 8-40kV, 0.01-50nA

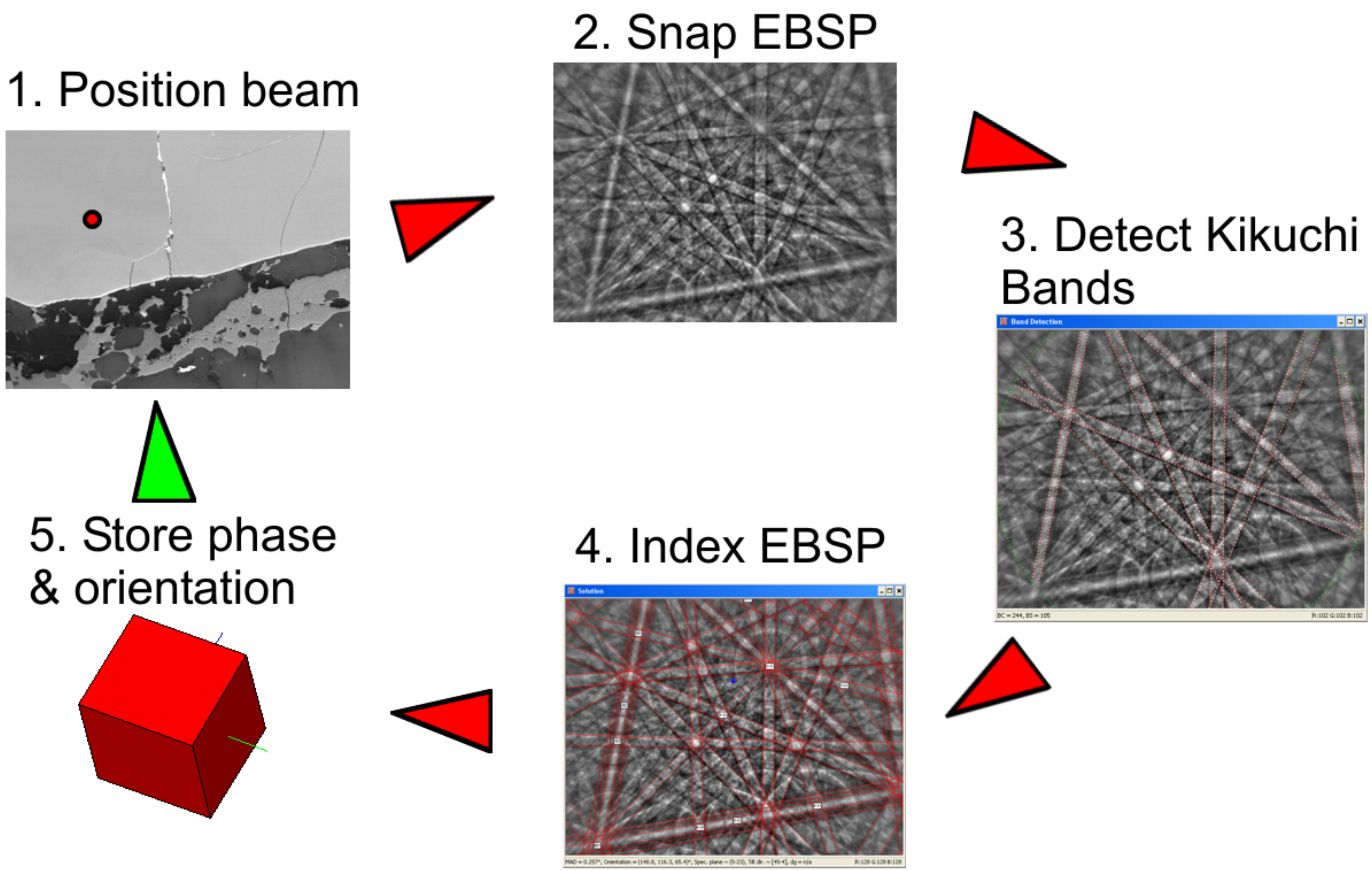


EBSD detector - position usually constrained by chamber geometry

Specimen: Surface normal typically inclined 60°-80° to horizontal

low-light sensitive (now digital; originally analogue)

EBSD detector distance set to give ~90° angular range in EBSP



1. Position beam

2. Snap EBSP

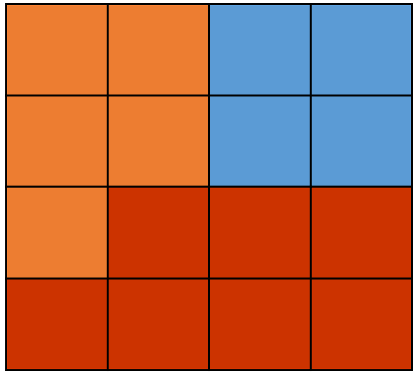
3. Detect Kikuchi Bands

4. Index EBSP

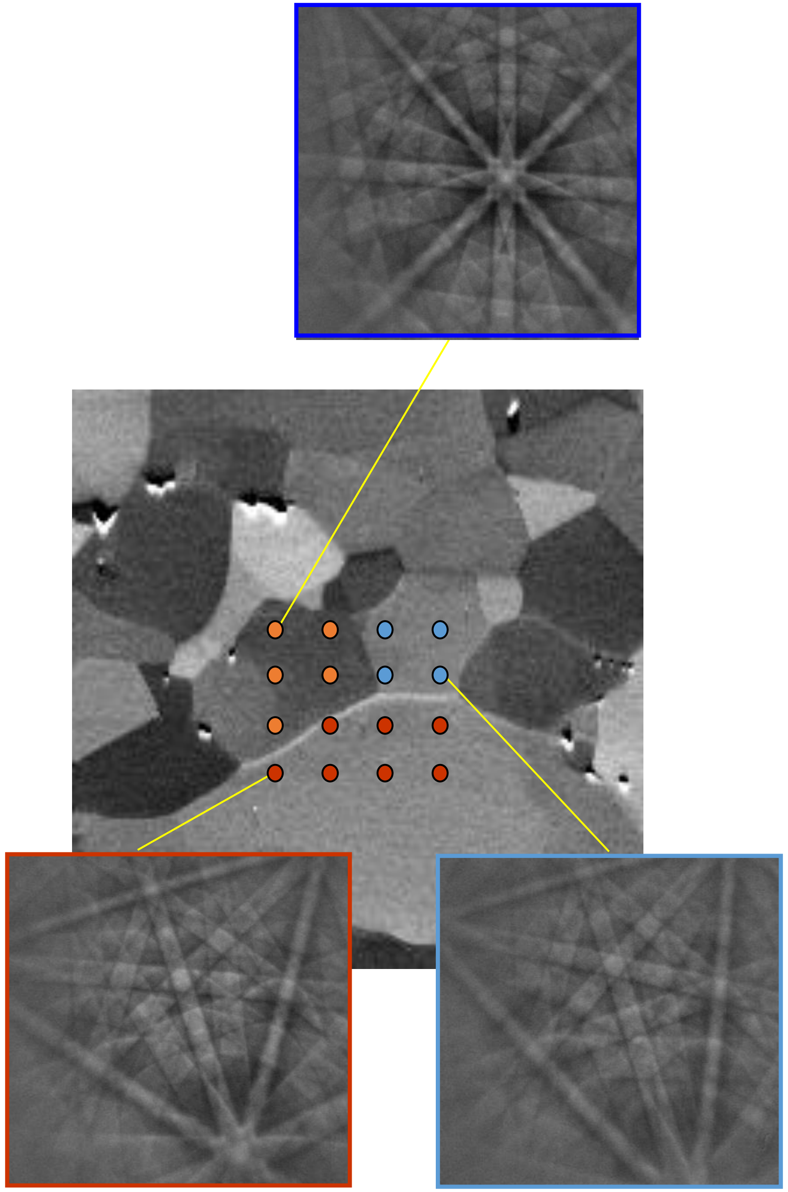
5. Store phase & orientation

The EBSD Cycle

Automated EBSD



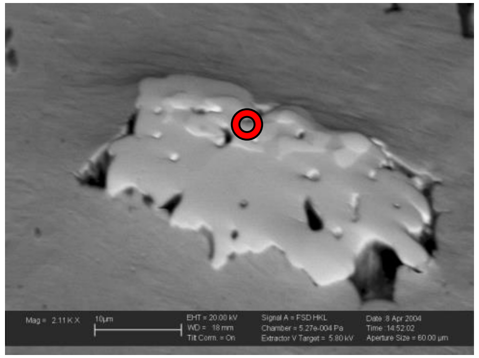
EBSD analysis on a grid of points, from which the complete microstructure can be reconstructed.



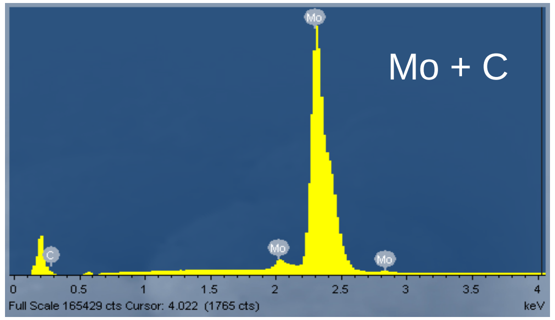
EBSD Automation

- Most EBSD analyses involve automation – "orientation mapping"
- The electron beam is stepped across a grid on the surface (orthogonal or hexagonal) and at each point the EBSD Cycle is repeated
- The data can then be represented in a map form (orientation map, phase map, boundary map etc.)
- Automated EBSD is very attractive as it can run for many hours unattended, and allows a near-complete characterisation of the microstructure

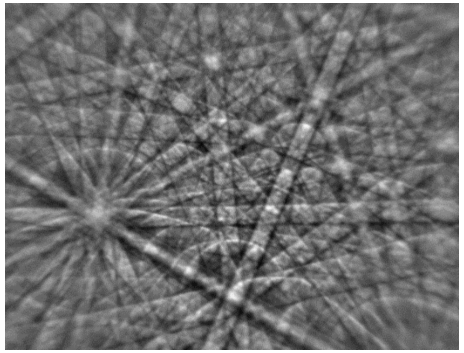
The Phase ID Cycle



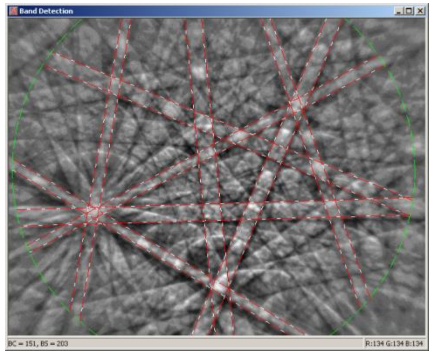
1. Position Beam



2b. Collect EDX Spectrum + identify peaks



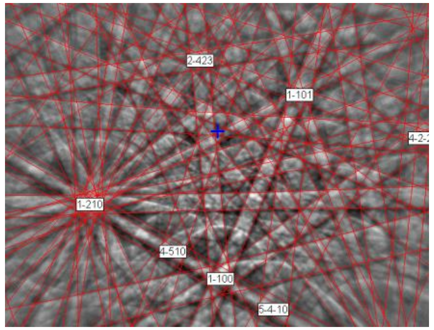
2a. Snap EBSP



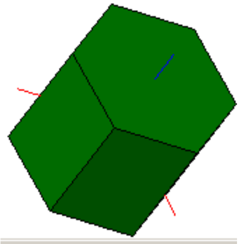
3a. Detect Bands

3b. Search database(s) for matching phases

Mo_2C - Space Group 60, orthorhombic
Mo_2C - Space Group 194, hexagonal
Mo_2C - Space Group 187, hexagonal
Mo_2C - Space Group 162, trigonal
Mo_2C - Space Group 29, orthorhombic



4. Index EBSP



Mo_2C – Hexagonal (SG 194)

5. Save result

What can EBSD tell us?

EBSD provides 3 main types of information:

- The absolute 3D orientation of the crystal lattice
- Discrimination between phases based on their crystallographic differences (e.g. austenite (FCC) and ferrite (BCC) in steel; α (HCP) and β (BCC) Titanium)
- Identification of unknown phases (Phase ID) when used in conjunction with EDX

EBSD cannot:

- analyse non-crystalline (amorphous) materials, such as glass, plastics, wood etc.
- provide information about the crystal orientations within the volume of a sample – it is purely a surface technique
- analyse samples with poor surface preparation or thick coats (e.g >10nm thick)
- Discriminate between phases with similar crystallography without EDX info (e.g. Fe-FCC and Al)

Typical Measurements with EBSD

- Phase distribution
- Phase identification
- Texture/CPO strength ("Texture" = degree of alignment of the crystal lattices in a sample)
- Grain size
- Boundary properties (e.g. twin boundary frequency)
- Misorientation data (difference in orientation between different points in the sample)
- Recrystallised / deformed fraction (proportion of the sample that has been recrystallised and deformed)
- Intra-granular deformation
- Plus much, much more

EBSD Peformance

EBSD performance can be measured using 3 criteria:

- Accuracy of Data
- Spatial Resolution
- Acquisition Speed

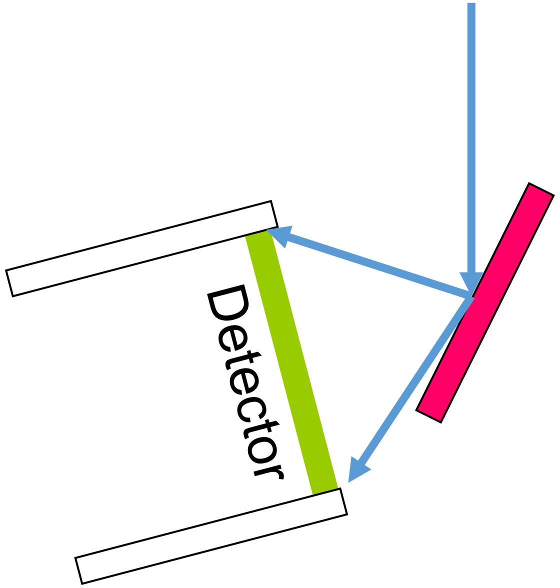
Data Accuracy I

- Angular Resolution – depends on many factors:
    - EBSP resolution
    - EBSP sharpness (usually inversely related to the degree of strain in the crystal lattice)
    - the resolution of the Hough transform (used to detect the Kikuchi Bands)
    - the system calibration
    - the parameters used to index the EBSP (e.g. number of bands detected)
    - the crystal orientation
    - the sample preparation
    - the capture angle (i.e. how close the phosphor screen is to the sample)

Angular resolution :

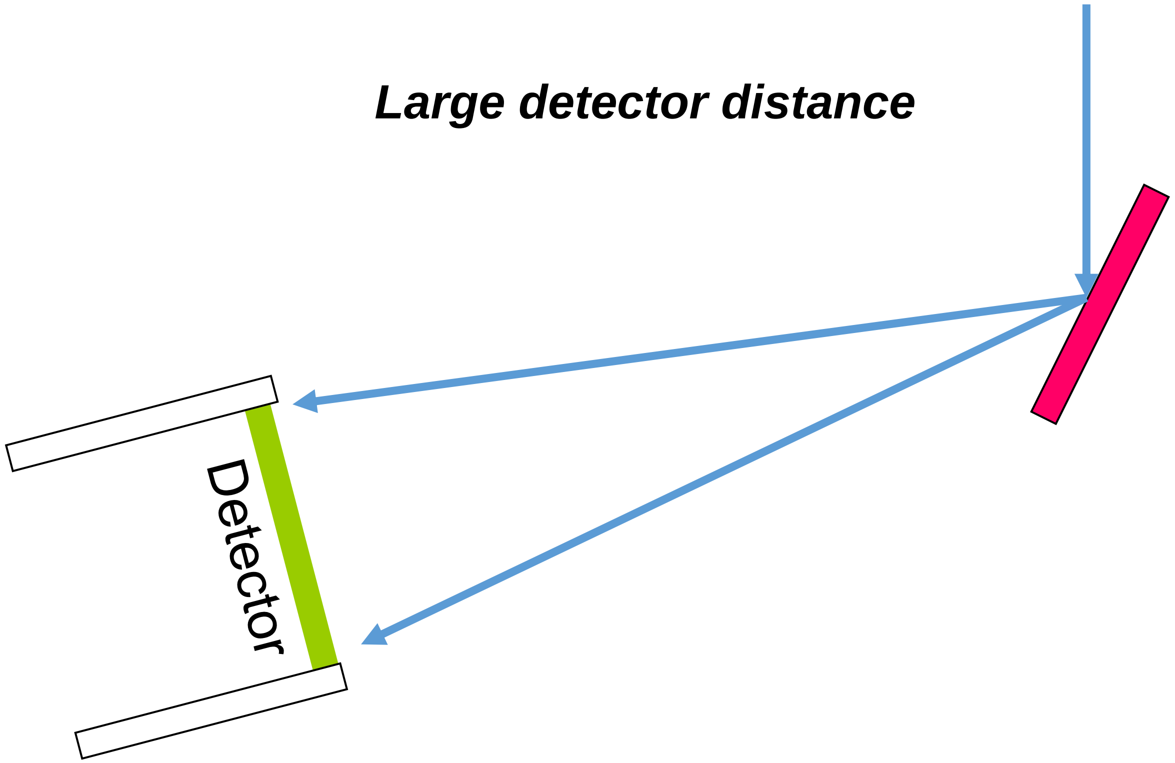
example sample-detector considerations

Small detector distance



good for indexing but
poor angular resolution

Large detector distance



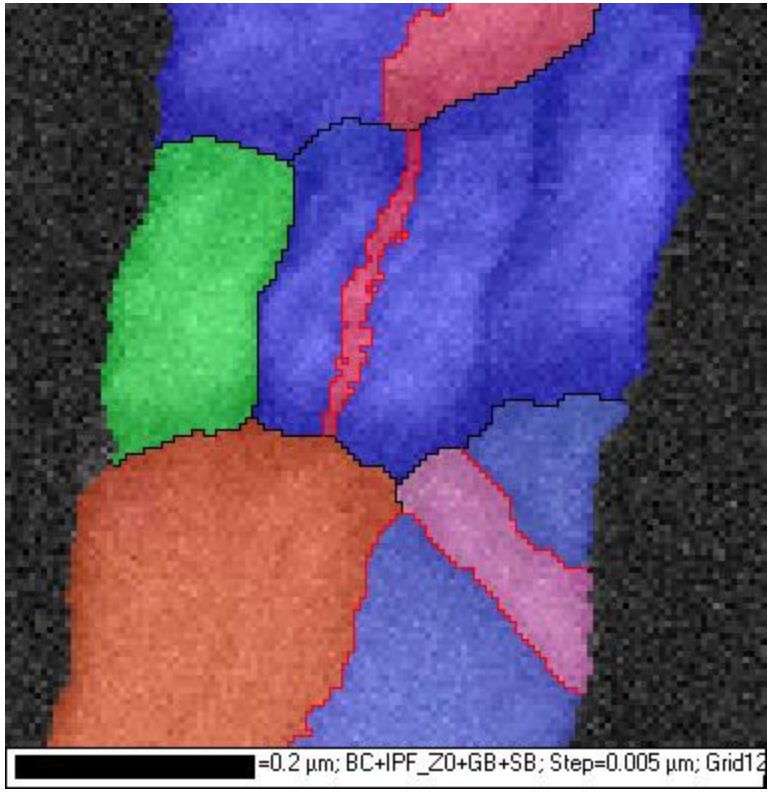
poor for indexing but good angular resolution

important for constraining misorientation axes.

Data Accuracy II

Data Reliability – the most critical of factors

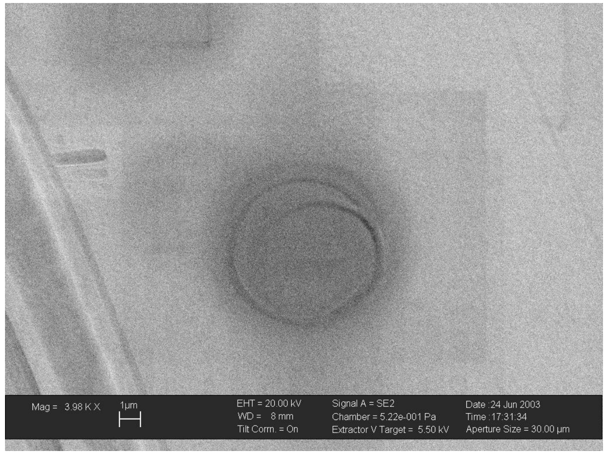
- Some materials have relatively simple crystallography and rarely produce errors (e.g. Al, Ni, austenite)
- But, many materials suffer from "pseudosymmetry": this means that there appears to be a higher order symmetry than really exists.
- Pseudosymmetry can lead to misindexed points even in the most carefully calibrated system
- Poor quality EBSPs (such as from a deformed sample) can also lead to misindexing
- There is often a balance between "hit rate" (% of points indexed) and data reliability



The limit of spatial resolution – twin domains in Cu-interconnects in the region of 20nm wide, mapped with a spacing between analysis points (step size) of 5nm.

Similar or better resolution has been achieved in Pt and Au thin films, with step sizes as low as 2nm.

BUT – at these resolutions, other factors usually become significant (such as contamination – see SE image below of a Pt-thin film after a 2nm step EBSD map)

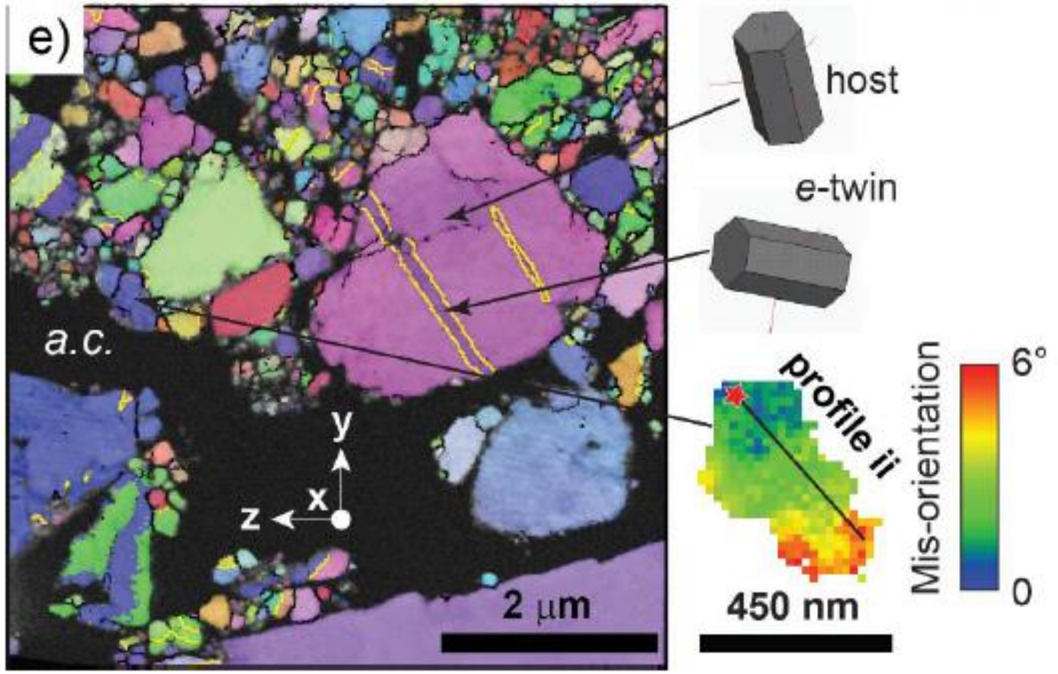


However, now we have a remedy!!
TKD (Transmission Kikuchi
Diffraction (Trimby 2012)
(EBSD on TEM sections

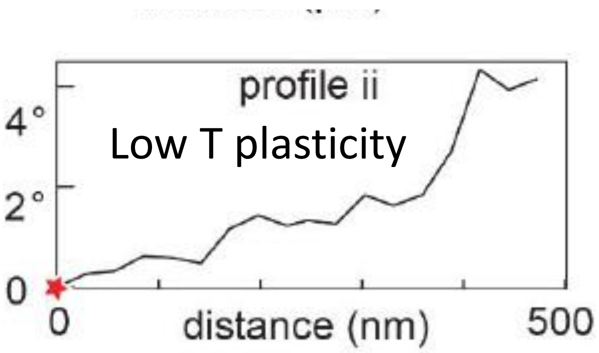
Example of TKD use

Shallow Earthquakes

Sub-seismic slip in nano calcite fault gouge generates amorphous carbon and crystallographic texture at low temperature



a.c. = amorphous carbon



Low T plasticity

- Low T plasticity
- e-twin generation
- Amorphous carbon

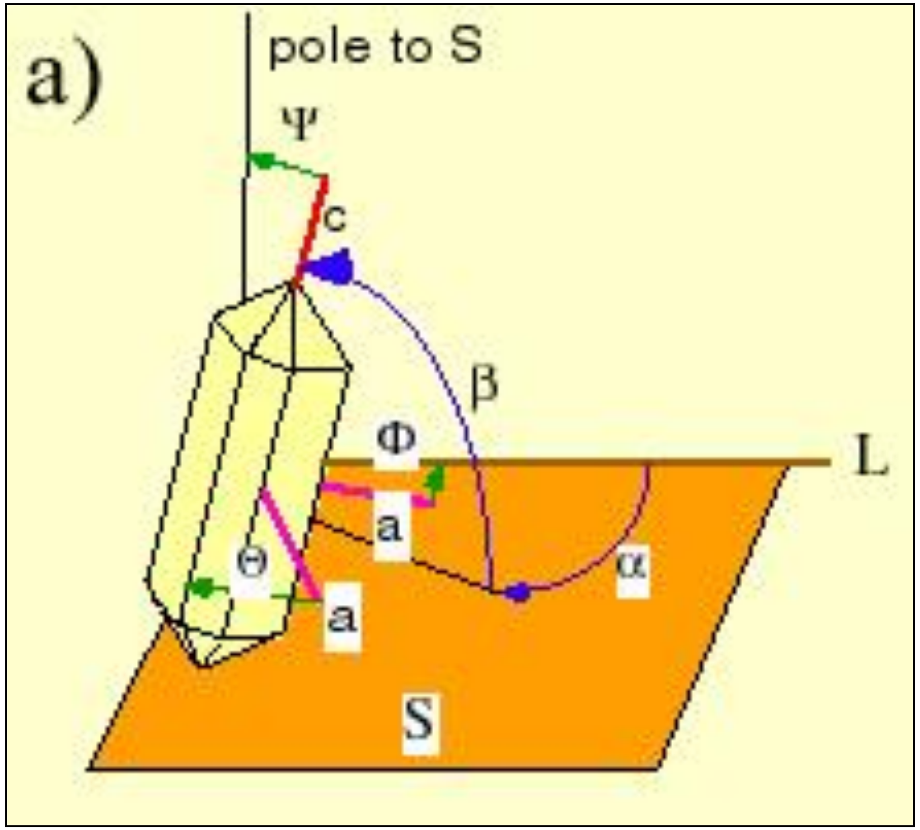
DelPiane, Piazolo et al., Geology, 2018

Acquisition Speed

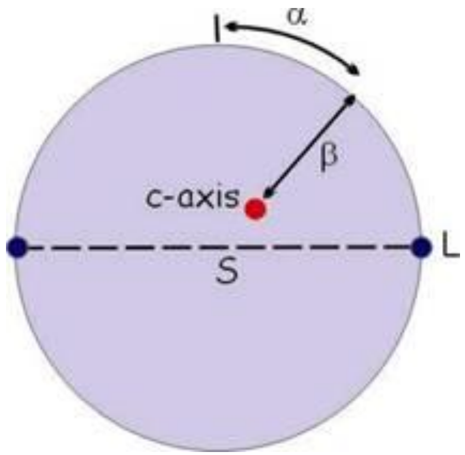
- Speed of data acquisition has increased dramatically in the last 10-15 years
- Now some systems can perform at 3000 pts/s
- Increases are due to software improvements, detectors computer processor improvements
- But, rarely is the maximum speed used for real analysis (the emphasis is best placed on data reliability) especially for rocks
- High speed analyses are generally not appropriate for work on complex samples (e.g. rocks, multi-phase layers) or for combined EDS-EBSD work

Display of Crystallographic Orientation Data

- Pole figures
- Inverse pole figures
- ODFs
- Orientation Maps



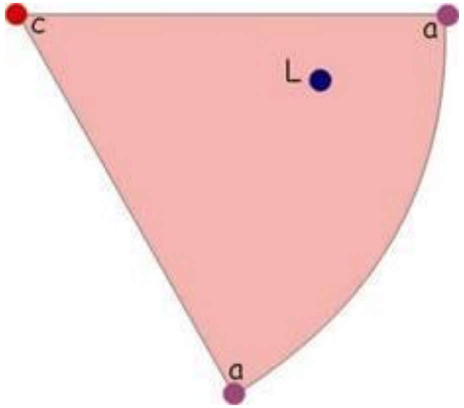
How to represent CPO Measurement



Optic axis (e.g. quartz c-axis) is a line – defined by plunge & azimuth & plots as point on a stereographic projection.

Two types from optical/universal stage:

1. *pole figure* - crystal direction (e.g. quartz ***c***-axis) in sample orientation (e.g. tectonic space - i.e. X,Y,Z or S, L);



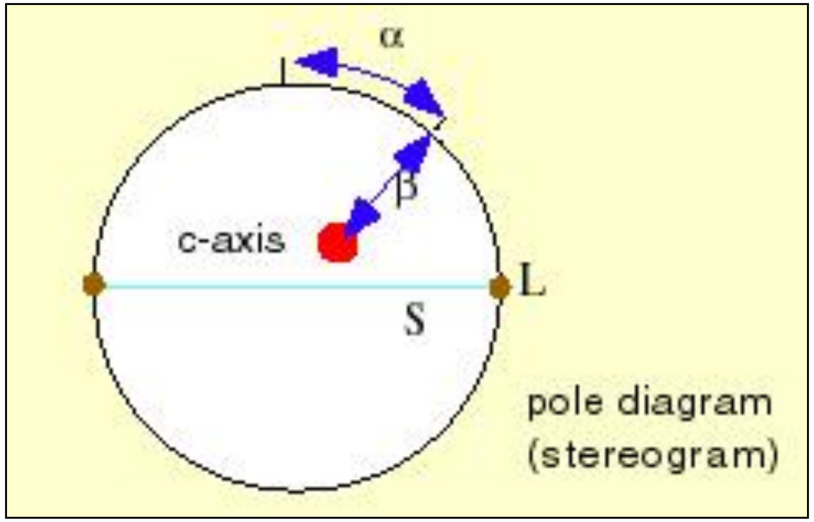
2. *inverse pole figure* – sample direction (e.g. tectonic X,Y, Z) in crystal space (e.g. crystal unit triangle).

Both 2D representations of 3D - assist interpretation but loose information (i.e. only 1 CPO element considered).

3D orientation defined by 3 unique directions relative to external (e.g. tectonic) reference frame (e.g. foliation & lineation; X, Y, Z; etc.).

Pole Figures

A pole figure shows the projected position of a particular set of crystallographic planes which have been projected on to a sphere and then on to a circle. There are two main methods for doing this, the stereographic and equal area projection.



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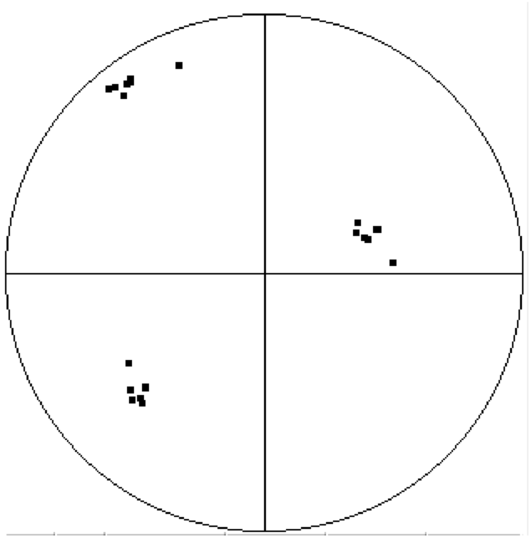
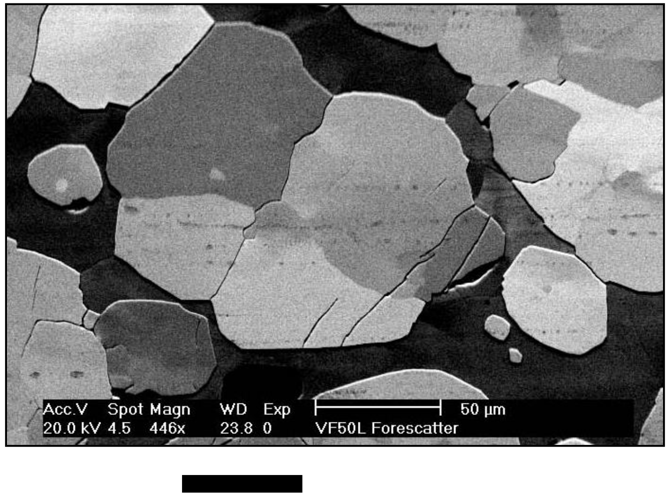
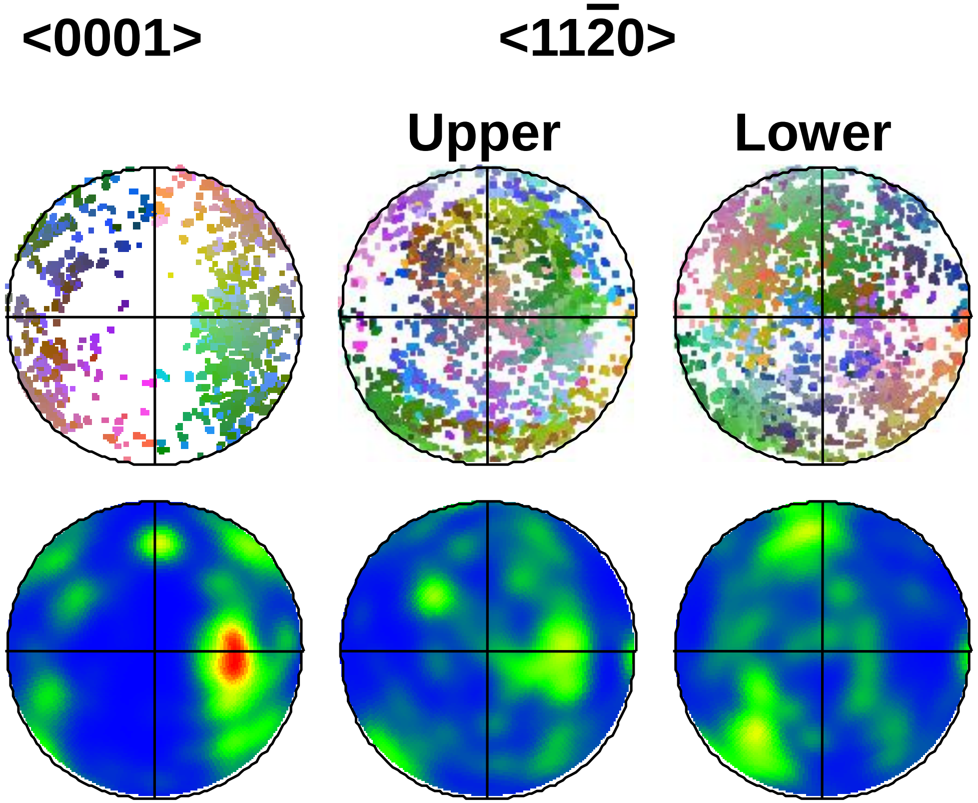


Garnet



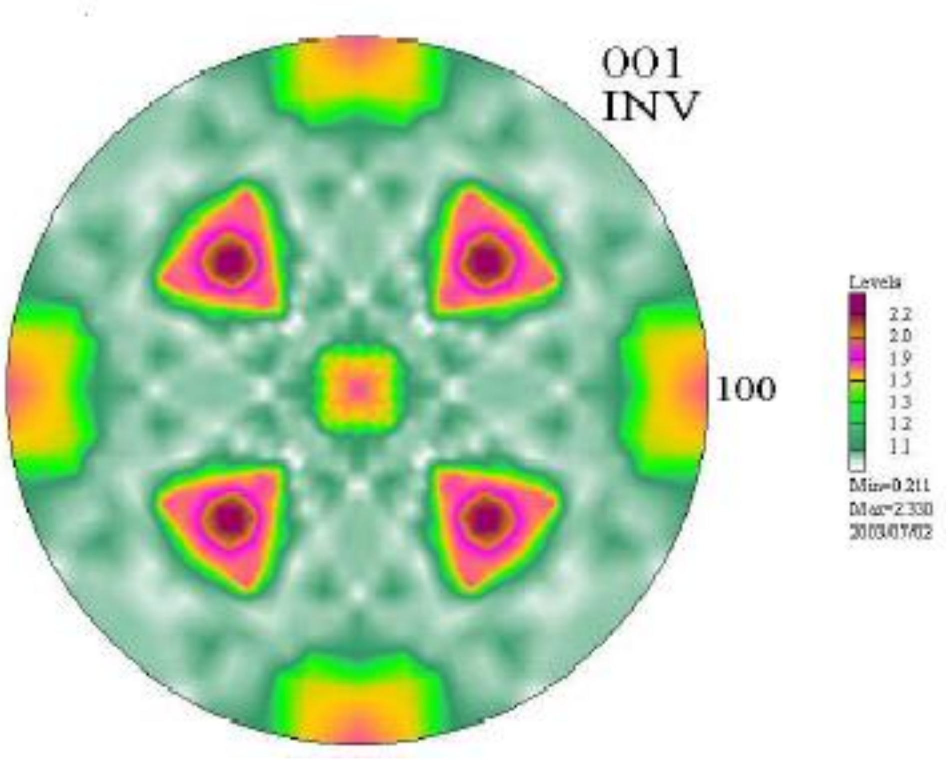
50µm

Quartz Pole Figures



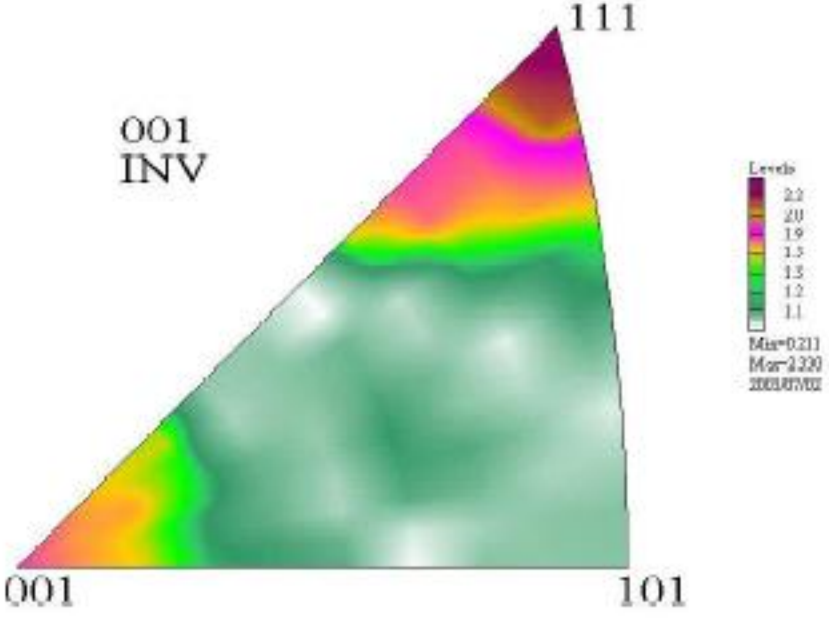
Inverse Pole Figures

Complete Inverse Pole Figure – orientation distribution of a sample axis or XYZ direction on a sterogram plotted with respect to crystallographic orientation (axis and direction)

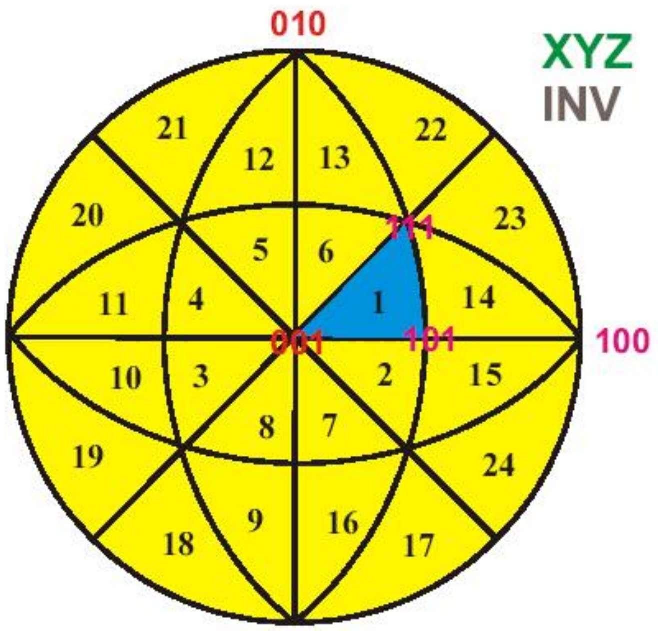


Inverse Pole Figures

Partial Inverse Pole Figure – orientation distribution of a sample axis or XYZ direction on a standard stereographic triangle. The area covered by the partial inverse polefigure is dependent on the crystallographic system.

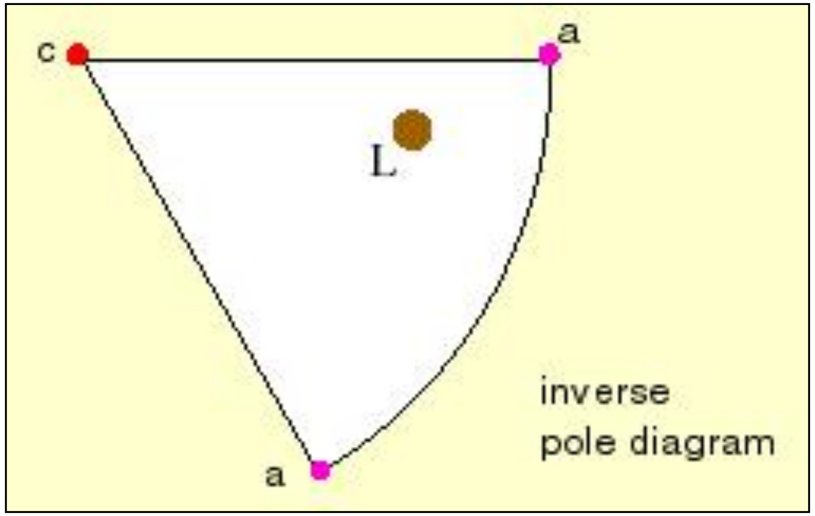


24 Equivalent regions of complete
inverse pole figure
for cubic system

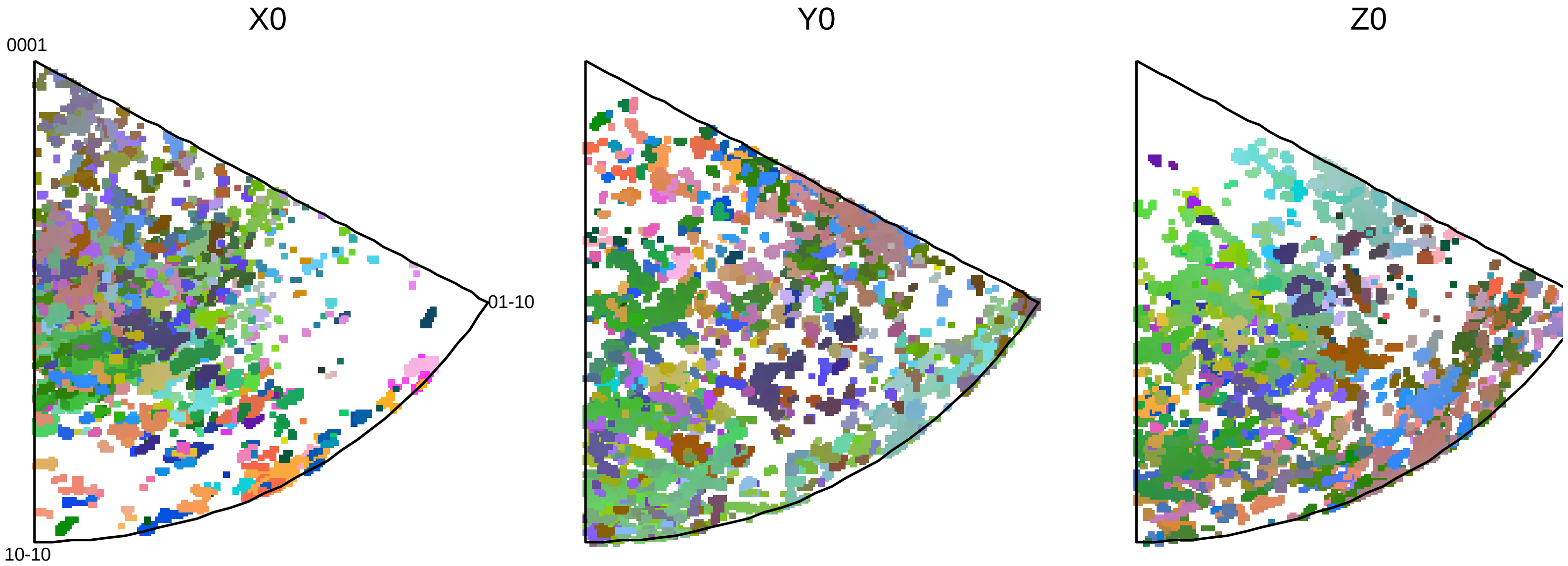


Inverse Pole Figures

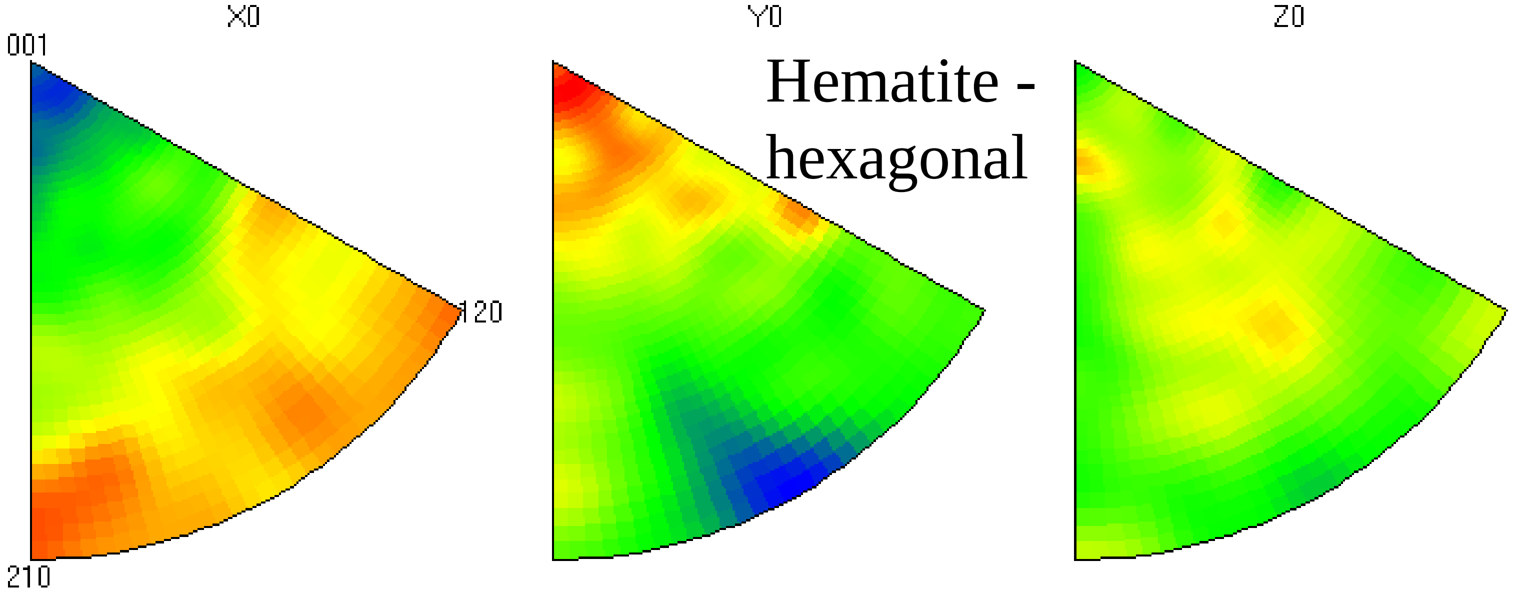
- Relatively easy to understand
- Need more than one IPF to show the full orientation



Quartz - trigonal



Hematite - hexagonal

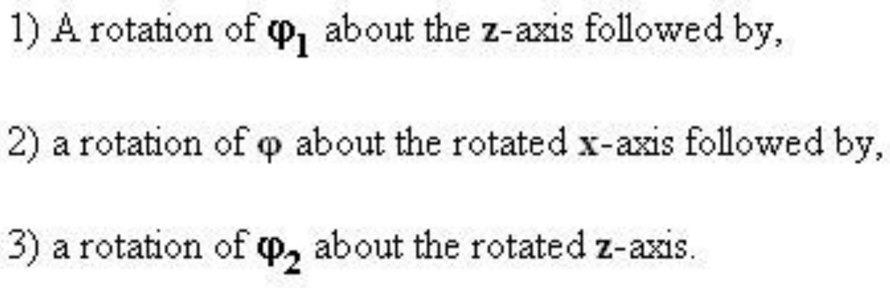


Orientation distribution functions (ODFs)

- Difficult to understand
- Individual orientations plot as single points using Euler angles
- Used extensively in material science

Need to measure 3 angles
– not possible optically

Euler angles

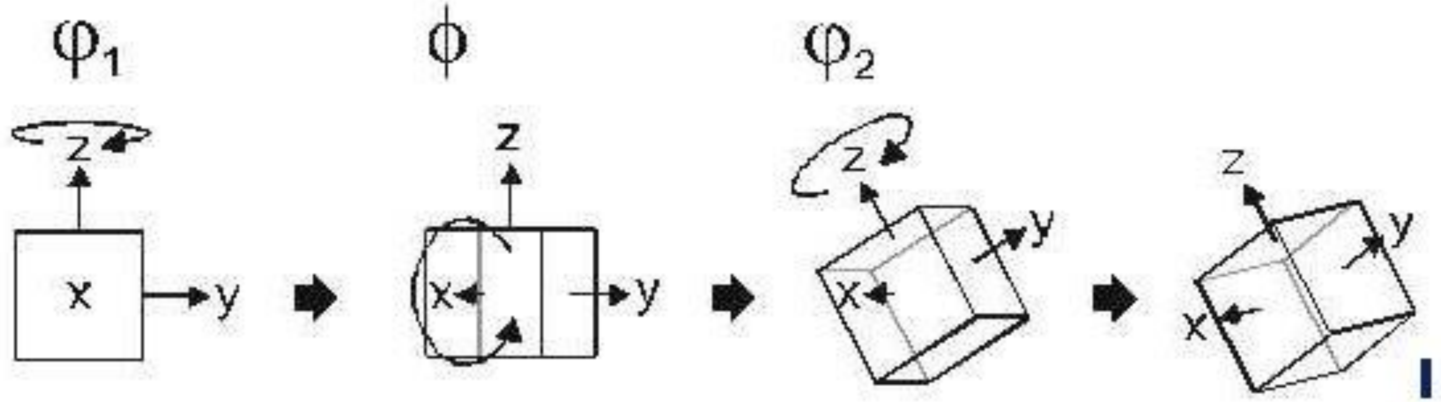


1) A rotation of φ_1 about the **z**-axis followed by,

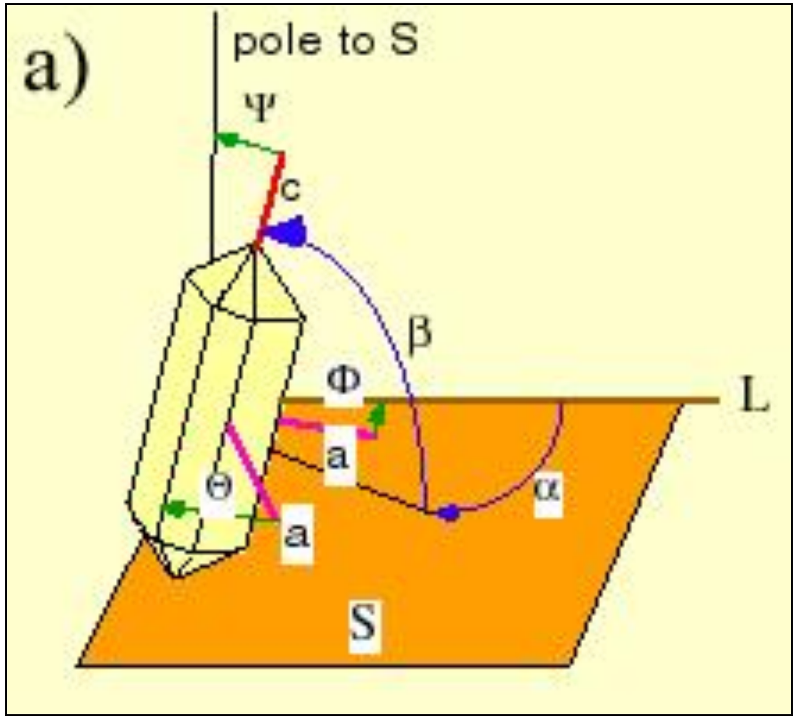
2) a rotation of φ about the rotated **x**-axis followed by,

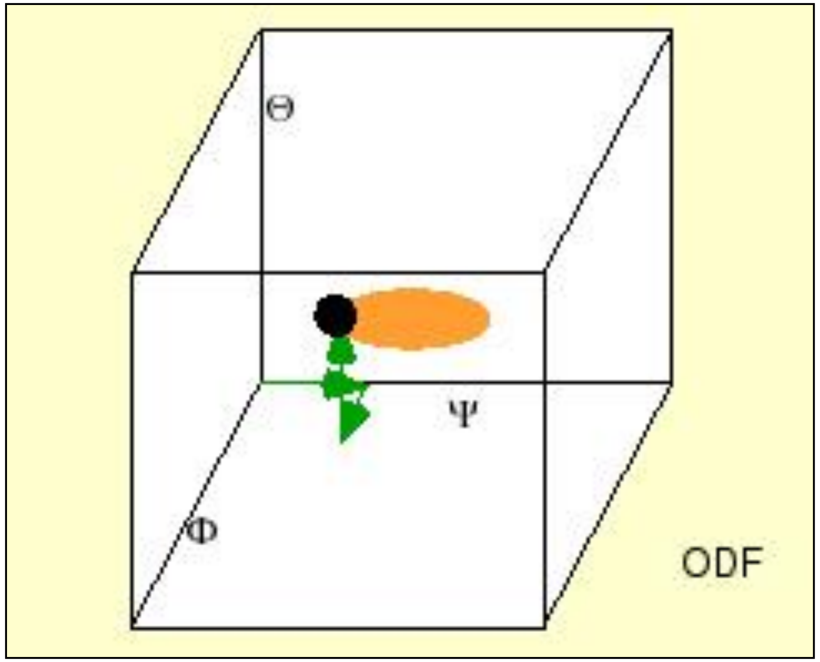
3) a rotation of φ_2 about the rotated **z**-axis.

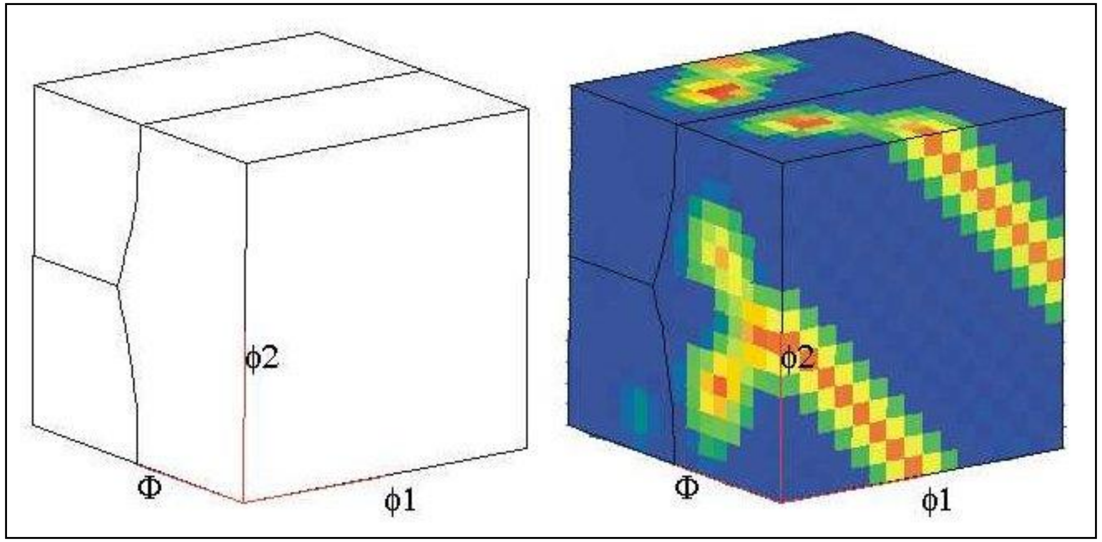
Watch out!!!
Several definitions of Euler angles in use!!



ODFs



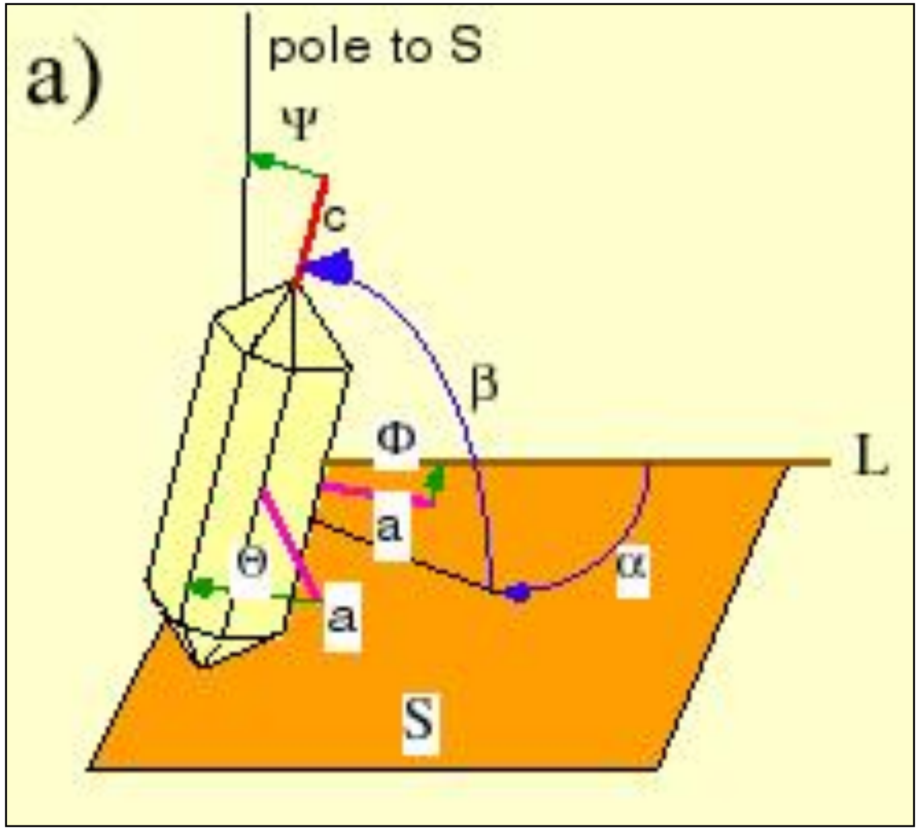




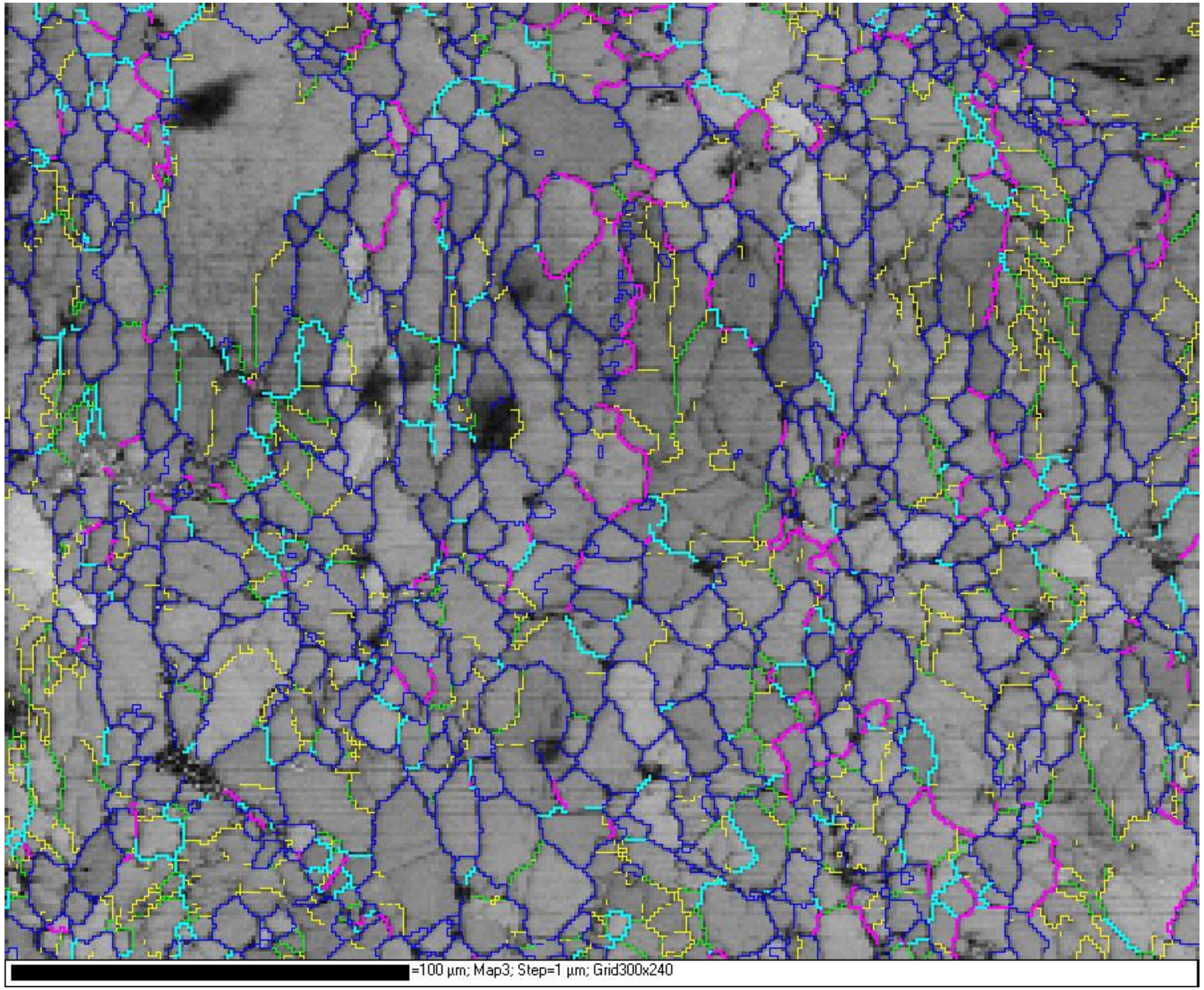
Display of Data

Spatial
Information

- ✗ Pole figures
- ✗ Inverse pole figures
- ✗ ODFs
- ✓ Orientation Maps



Boundary Maps



>2°

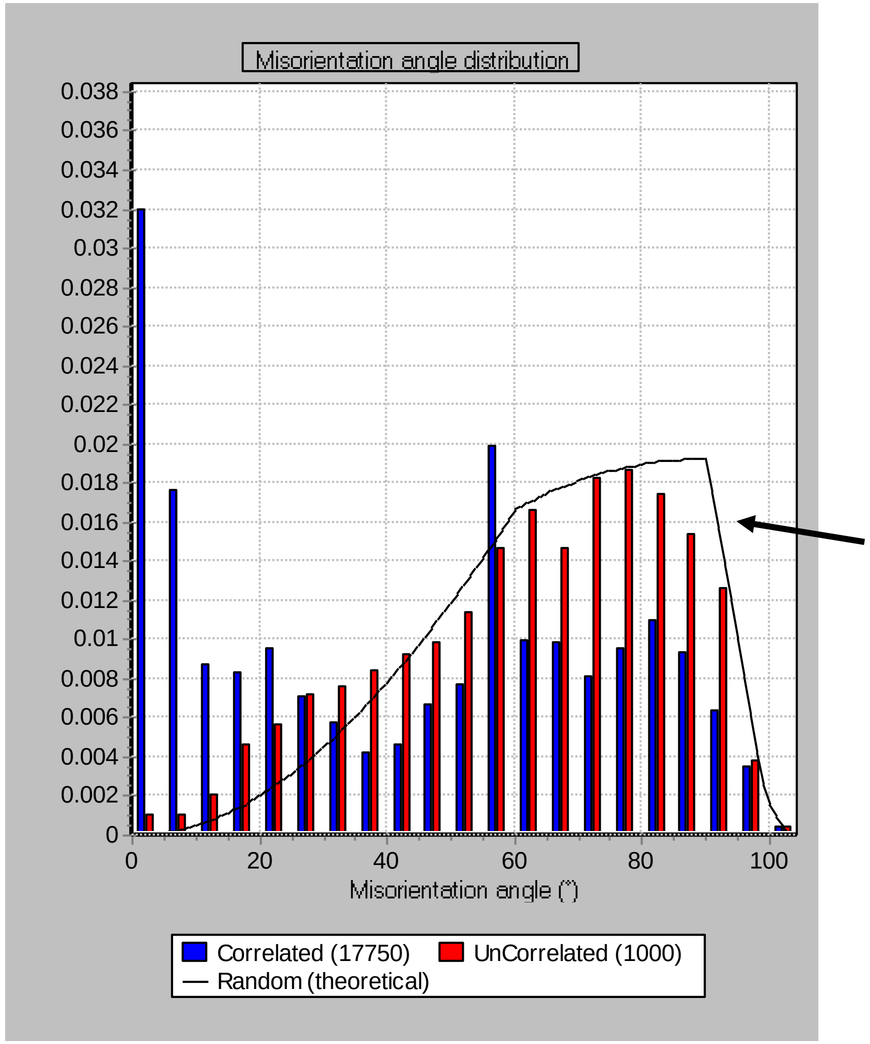
>5°

>10°

>20°

>30°

Misorientation angle distribution



🟦 **Neighbour pair**

🟥 **Random pair**

Disorientation probability

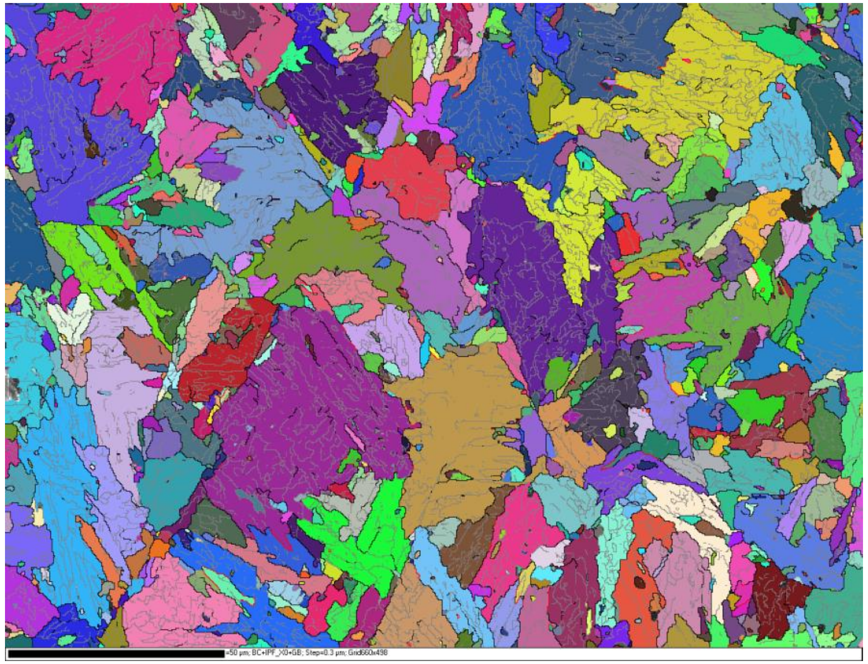
EBSD Application Fields

In general there are 3 main application areas:

1. Materials sciences
2. Microelectronics
3. Earth and planetary sciences

Materials Sciences I – very similar to Geology

- **Metals Processing**
  – the main application field of EBSD.
  - quantify physical properties (such as strength, ductility, corrosion resistance, toughness etc.) of many metals are controlled by key characteristics such as grain size, phase distribution, deformation…

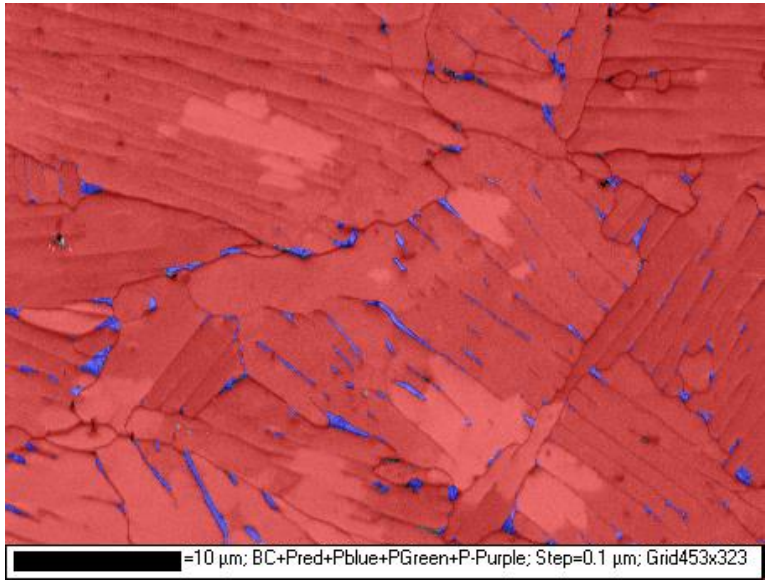


EBSD grain map of a bainitic steel

Courtesy of P. Trimby

Materials Sciences II

- **Ceramics**
  – for high T applications.
  - phase discrimination and standard texture/grain size measurements
- **Phase distribution**
  - knowing the proportion of different phases is essential in many materials, such as in duplex steels. EBSD nice quantitative method & location



Phase map of a Ti sample:

Red = α-Ti (hexagonal – 97.3%

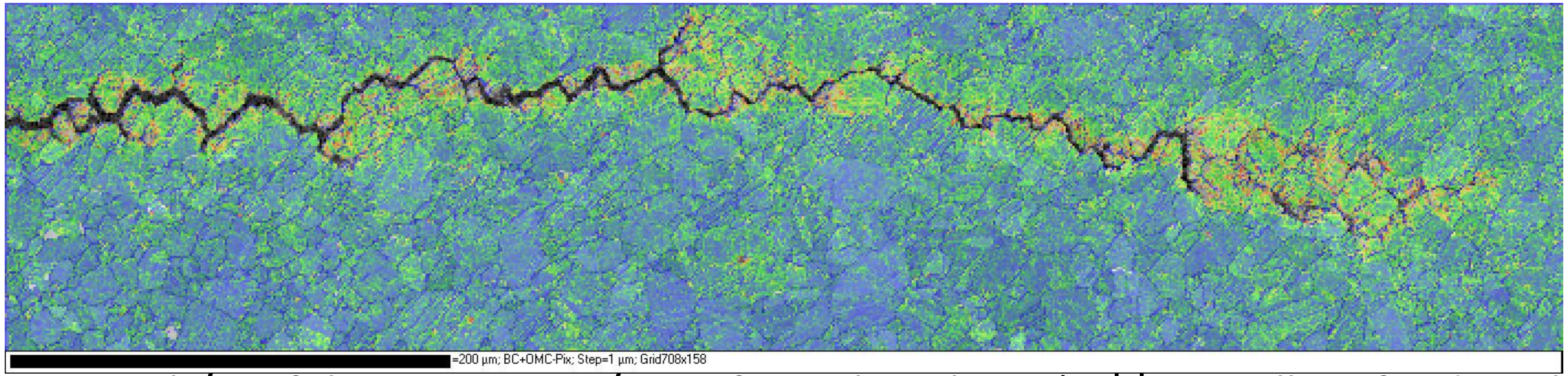
Blue = β-Ti (cubic – 2.7%)

Courtesy of P. Trimby

Materials Sciences III

- **Fatigue / strain analyses**
  – to measure intragranular deformation, such as localised deformation associated with cracks.
  - used in studies of fatigue, crack growth and strain analysis.

Red = high intragranular deformation



analysis of the microstructures of metal implants (e.g. Ti-Ta alloys for dental implants).

Courtesy of P. Trimby

Microelectronics I

- **Grain Boundary Engineering**
  – knowledge need to refine grain boundary characteristics in order to enhance a material’s properties.
  - e.g. development of special boundary properties in Cu thin films and Cu interconnects, enabling percolative networks across the material (conductivity is increased by an abundance of low angle and “special” (e.g. CSL) boundaries).

Grain map of 500nm Cu interconnects, showing best fit ellipses

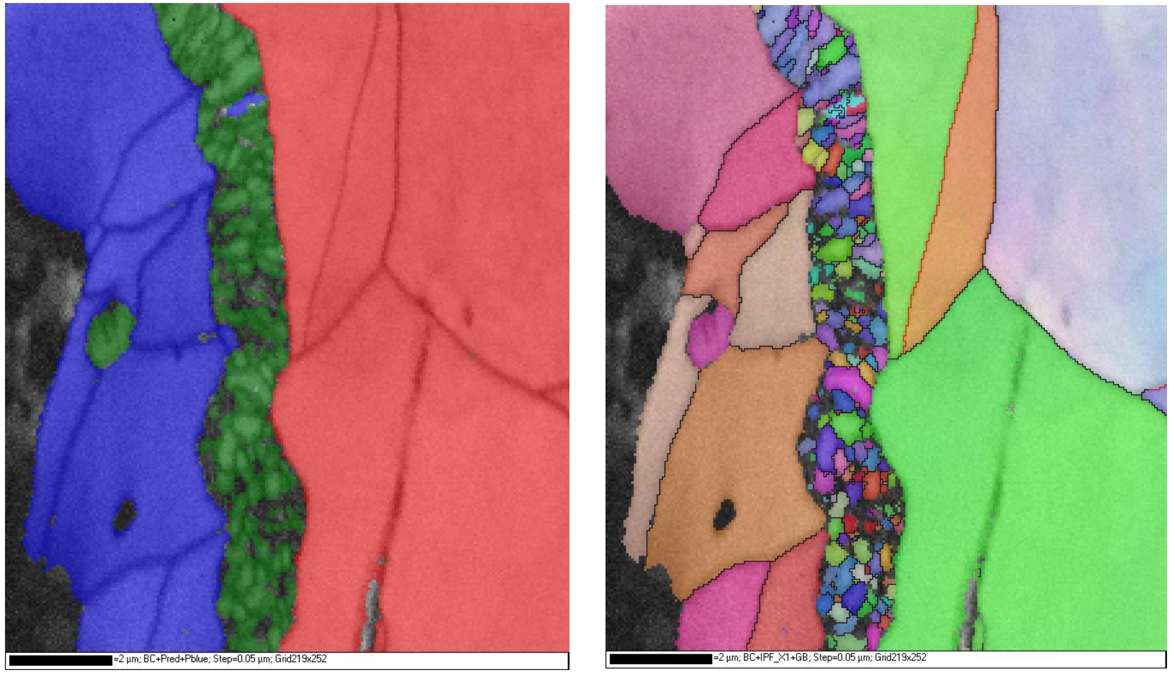
Left – excluding twin boundaries

Right – including twin boundaries

Courtesy of P. Trimby

Microelectronics II

- **Solders**
  -> very small changes in chemistry but significant changes in crystallography on a small scale.



Phase map and orientation map of layers in a Pb-free solder:

Red = Cu
Green = Cu3Sn (orthorhombic)
Blue = Cu6Sn5 (hexagonal)

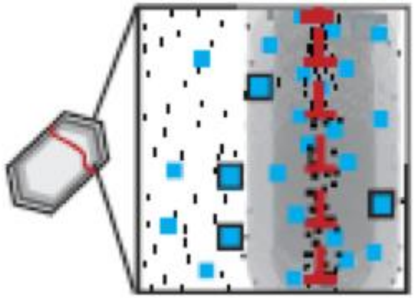
Courtesy of P. Trimby

Earth and Planetary Sciences I

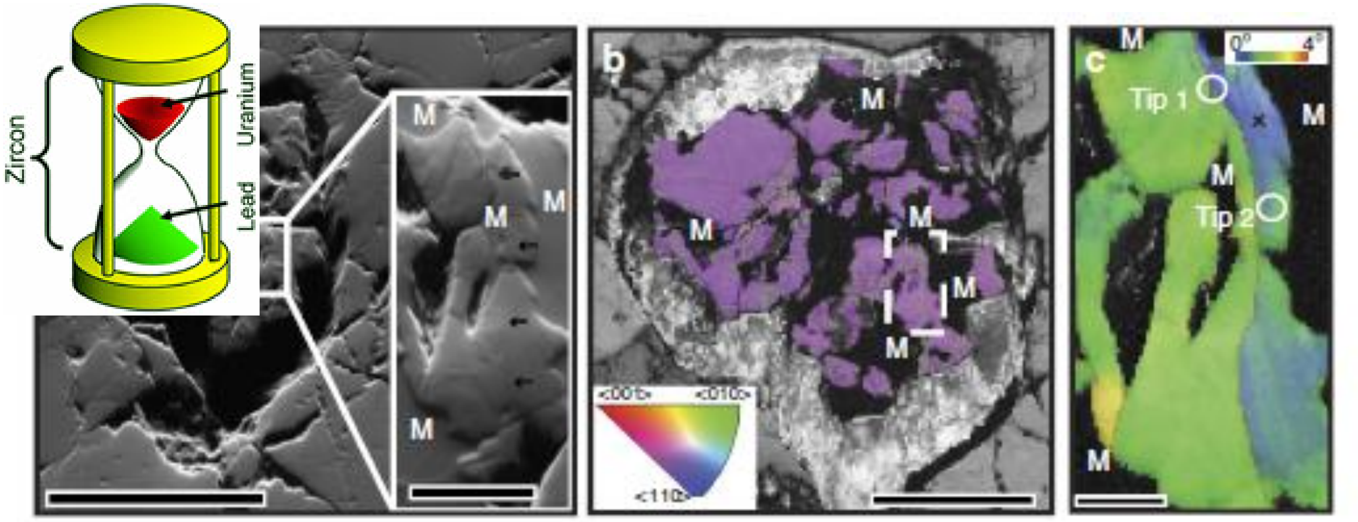
Piazolo et al. Nat. Comms. 2016, Atoms on the move – Deformation induced element redistribution

- **Igneous Geology**
  - e.g. orientations of olivine and chromite grains in layered intrusions -> clues about the mechanism of layer formation.
- **Metamorphic Petrology**
  - accurate phase identification of very small metamorphic minerals
  - studies on the effect of deformation on the accuracy of dating techniques & link between deformation and composition.

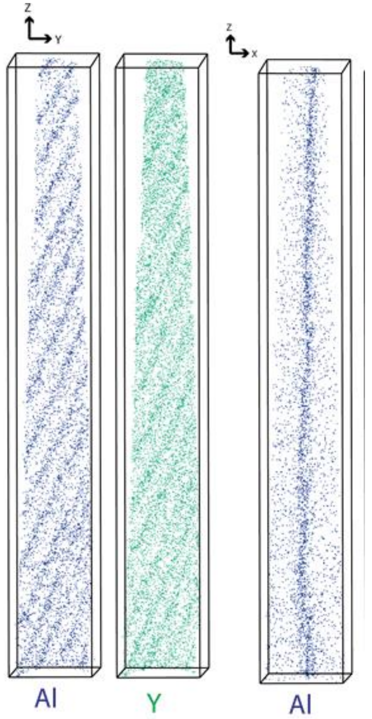
Formation of dislocation array



Subgrain boundaries -> bullet trains for elements *“pipe diffusion”*



Atom probe
Atomic scale
Element
distribution

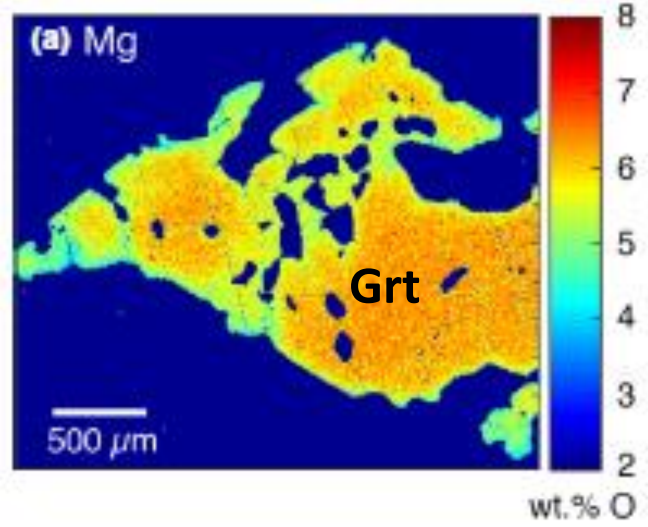


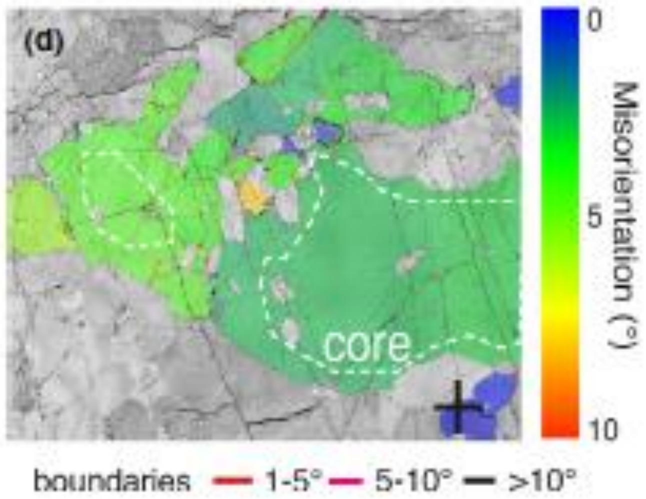
Napier Complex, Antarctica – Zircon Dating

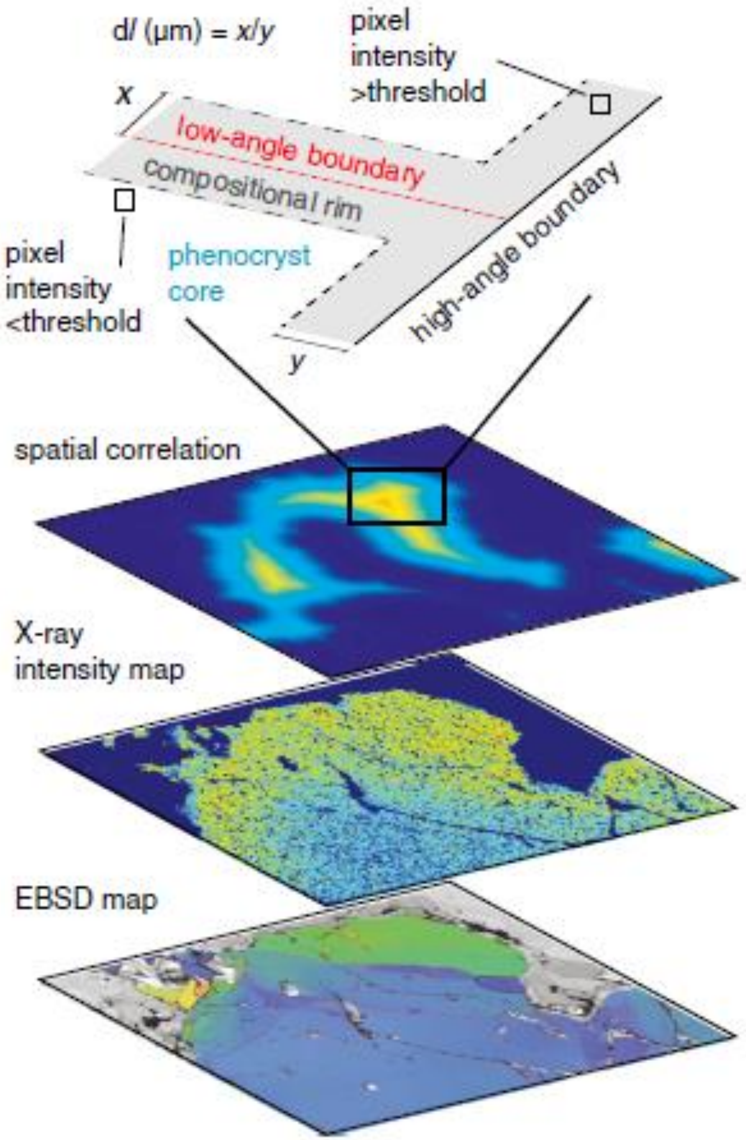
>2 Ga, high T deformation, >1.6 Ga quiet, metamict zones, reverse discordance

Earth and Planetary Sciences II

- **Igneous Geology** For example, studying the orientations of olivine and chromite grains in layered intrusions -> clues about the mechanism of layer formation.
- **Metamorphic Petrology** - accurate phase identification of very small metamorphic minerals & studies on the effect of deformation on the accuracy of dating techniques & **link between deformation and composition.**







Chapman et al. 2019

Earth and Planetary Sciences III

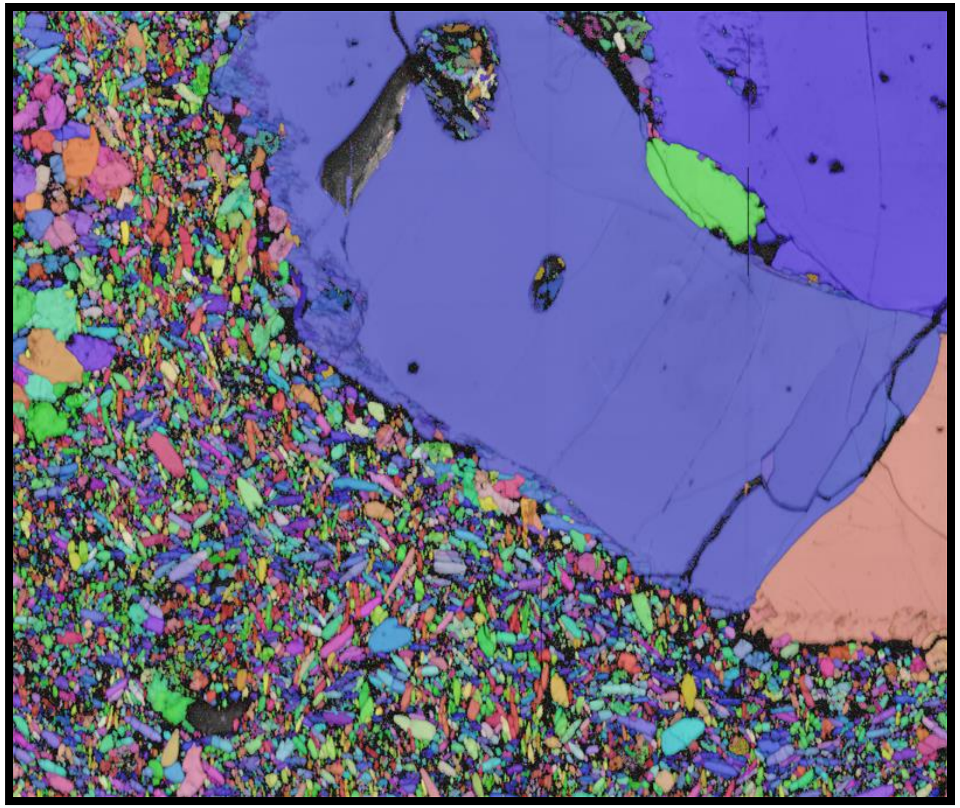
- **Palaeontology / Biomineralogy**
  – Now really possible: as many of the minerals in shell structures (e.g. calcite and aragonite) are very beam sensitive and many of the structures are extremely fine grained.

Grain structure in aragonite nacre in a modern gastropod shell. The layers are 1-2 μm wide

Courtesy of P. Trimby

Earth and Planetary Sciences IV

- **Sedimentology** – provide clues into the mechanism of overgrowth formation in sandstones (a key feature in the suitability of sandstones for petroleum reservoirs/ CO2 sequestration),
  - to correlate the texture with seismic anisotropy. (more later)
- **Meteorites** – Note: meteorites similar to many terrestrial rocks (e.g. the olivine-pyroxene grains in the chondritic meteorite shown),
  -show many interesting phase transformation / phase relationship characteristics (more later)

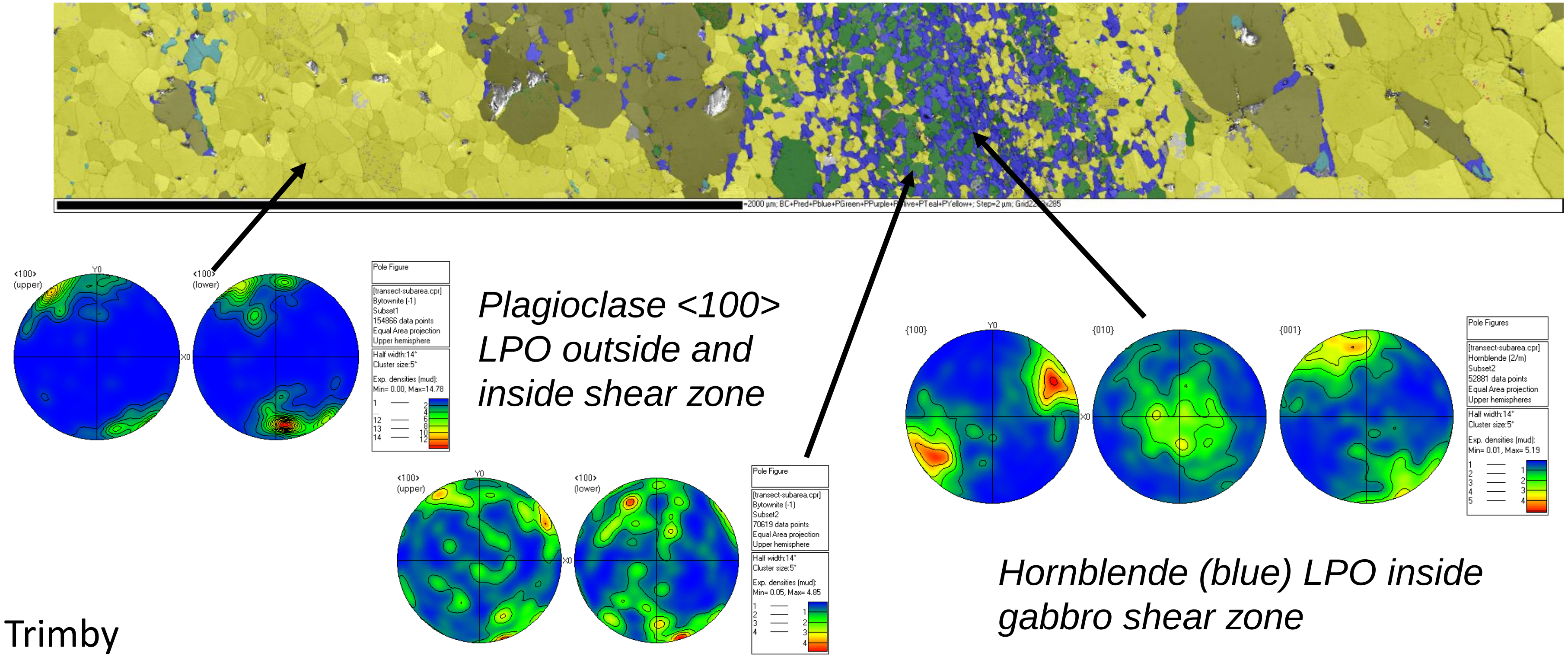


EBSD map of Allende meteorite

Courtesy of P. Trimby/L. Daly

Earth and Planetary Sciences - some examples here

- **Structural Geology** – accurate determination of the deformation processes active during a rock's history using microstructural characterisation and lattice preferred orientations (LPOs).
  -EBSD is not limited to just a few phases, and is providing important breakthroughs in a range of rocks types and minerals (e.g. quartz, carbonates, feldspars, oxides, olivine etc.).



Plagioclase <100> LPO outside and inside shear zone

Hornblende (blue) LPO inside gabbro shear zone

Courtesy of P. Trimby

Example: Crystal plastic Deformation behaviour of the mantle
Olivine

in terms of: flow direction,
stress & wet/dry conditions

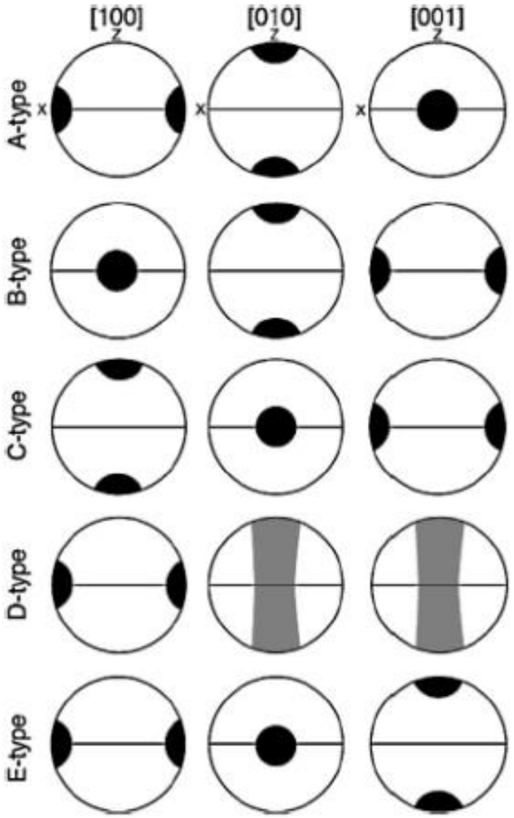
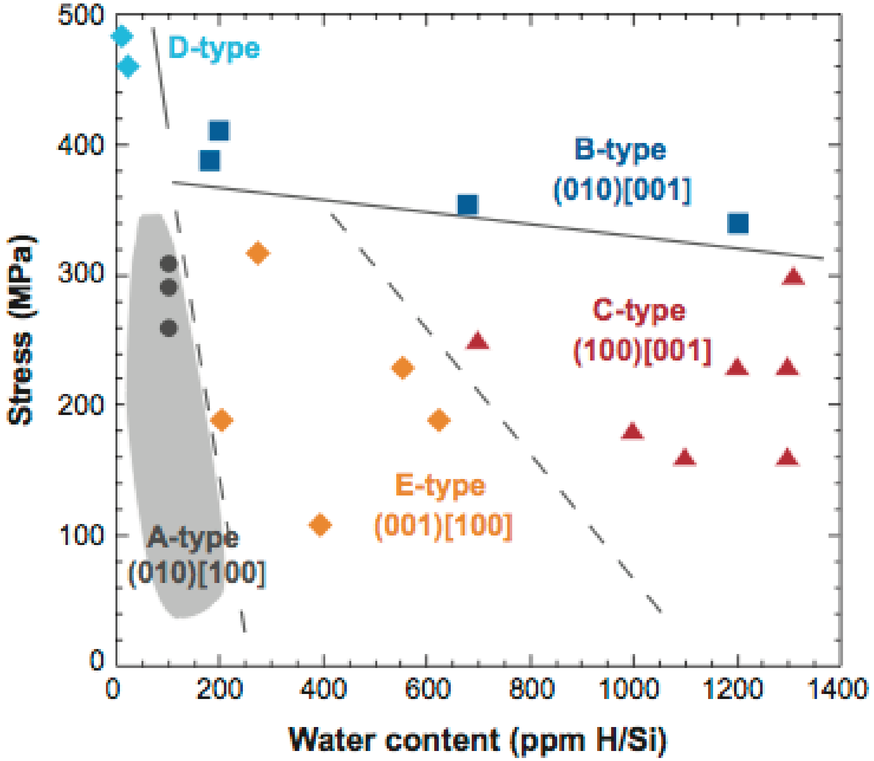


Fig. 1. Schematic olivine fabric types proposed by Jung and Karato (2001) and Katayama et al. (2004).

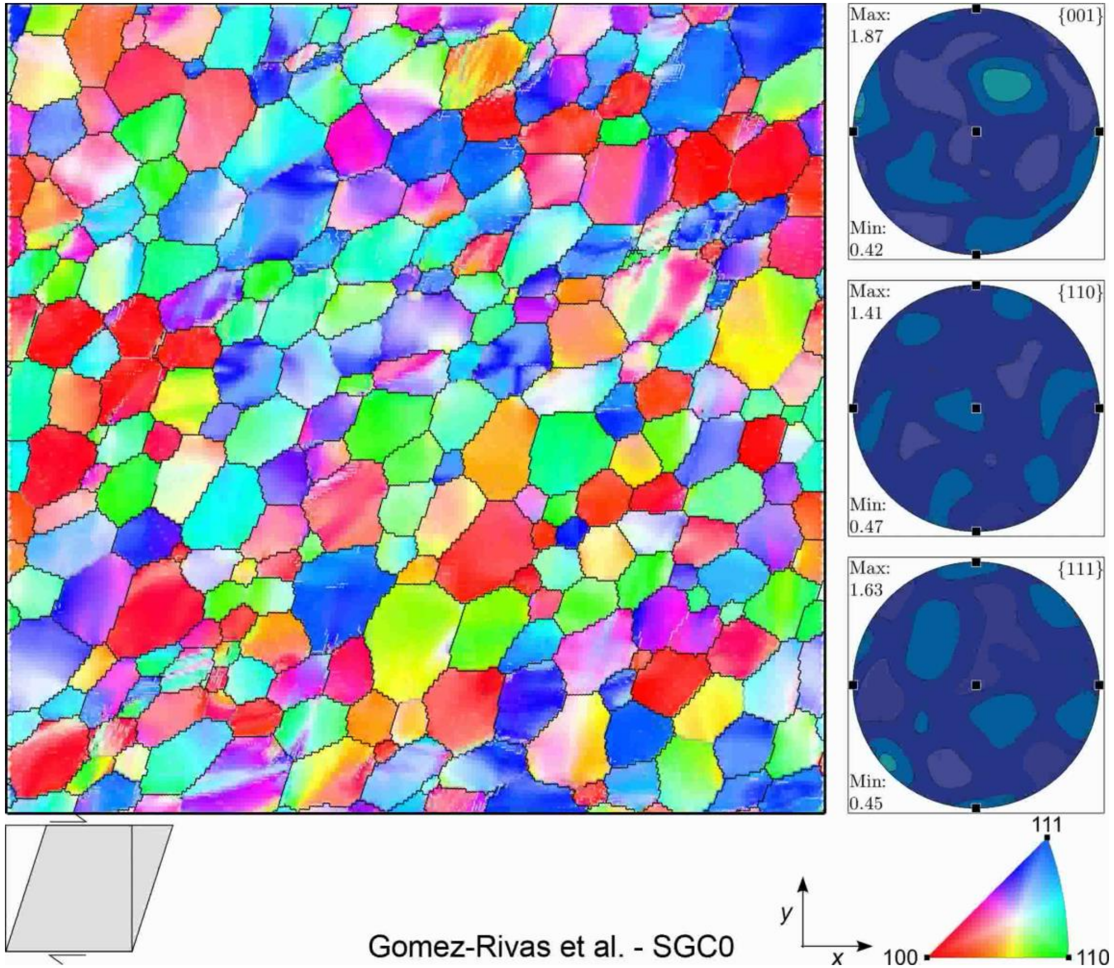


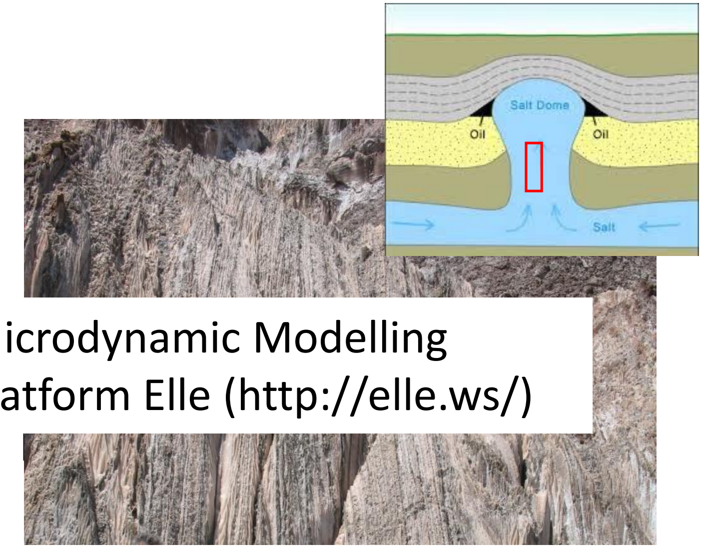
Karato et al. 2008

Compare to numerical models – e.g. salt deformation modelling

Forensic – reading the rock record, succession of events is pivotal

Predictive – future behaviour and material properties for hazard management, input for large scale models





Microdynamic Modelling platform Elle (http://elle.ws/)

Thank you & Questions

Salt Diapir neck: Cardona, Spain

Operator splitting: FFT viscoplastic deform. – subgrain formation – gbm- recovery