Carnegie Mellon

Understanding the TSL EBSD Data Collection System

27-750

Texture, Microstructure & Anisotropy

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MSE

Overview

- ## Understanding the diffraction patterns
  - Source of diffraction
  - SEM setup per required data
  - The makeup of a pattern

- ## Setting up the data collection system
  - Environment variables
  - Phase and reflectors

- ## Capturing patterns
  - Choosing video settings
  - Background subtraction

- ## Image Processing
  - Detecting bands: Hough transform
  - Enhancing the transform: Butterfly mask
  - Selecting appropriate Hough settings
  - Origin of Image Quality (I.Q.)

Overview (cont'd)

- ## Indexing captured patterns
  - Identifying detected bands: Triplet method
  - Determining solution: Voting scheme
  - Origin of Confidence Index (C.I.)
  - Identifying a solution in multi-phase materials

- ## Calibration
  - Physical meaning
  - Method and need for tuning

- ## Scanning
  - Choosing appropriate parameters

- General reference on orientation mapping: "Orientation Mapping" by Anthony D Rollett & Katayun Barmak; uploaded to Box as CH11-Orientation_Mapping-final_proofs.pdf.

Questions (1)

- Why do we need to position the specimen at the eucentric point?
- Why does the specimen need to be tilted at a steep angle of incidence (70°) to the electron beam?
- Why is it so important to avoid contact between a specimen and the phosphor screen?
- What is the function of the phosphor screen?
- What is the characteristic appearance of a diffraction pattern in EBSD?
- Why is specimen surface preparation so important?
- What are reflectors and how do you choose them?
- What is the Hough Transform?
- What does "binning" refer to (in connection with Hough Transforms)?
- What is a "sharpening mask"?
- What does "frame averaging" do for acquisition?

Questions (2)

- What does background subtraction do?
- What is image quality?
- What are the coordinates of the image after the Hough transform has been applied?
- Why is the Hough transform effective for detecting lines?
- What are interzonal angles (in the context of an EBSD diffraction pattern)?
- What is the "confidence index" and how is it calculated?
- Why is it important to have a flat surface for the specimen?

SEM Schematic Overview
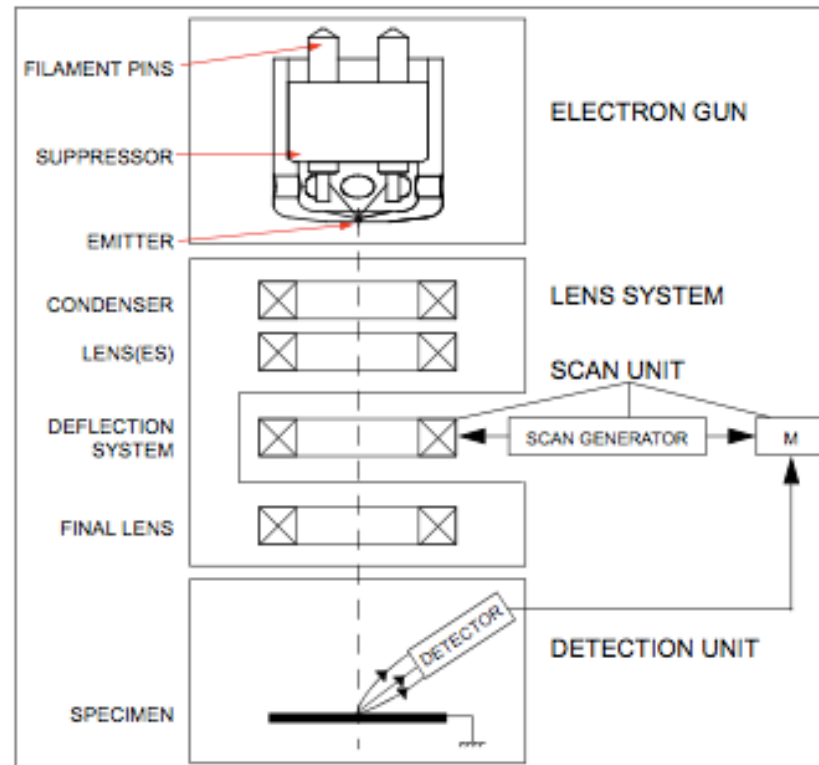
- All students using this system need to know how to use SEM. It is recommended that all users take SEM courses offered by the MSE department

Sample Size effect
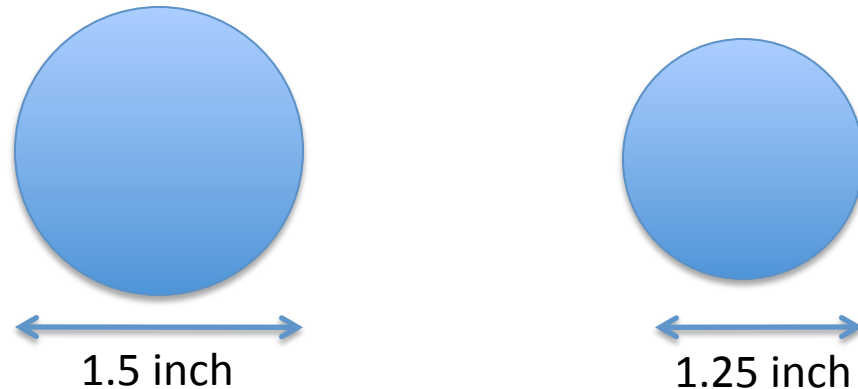
1.5 inch   1.25 inch

- All the samples needs to be prepared (polished) before EBSD data collection. As most samples are mounted before polishing, it is recommended to use smaller size mount (1.25 inch preferred)
- It is difficult to work with large mounted samples (with 1.5 inch) in OIM as the edge of the mount may touch either the camera or the SEM emitter after tilting
- It is critically important that the specimen does NOT touch the phosphor screen because this is easily damaged

Diffraction Pattern-Observation Events

- OIM computer asks Microscope Control Computer to place a fixed electron beam on a spot on the sample

- A cone of diffracted electrons is intercepted by a specifically placed phosphor screen

- Incident electrons excite the phosphor, producing photons

- A Charge Coupled Device (CCD) Camera detects and amplifies the photons and sends the signal to the OIM computer for indexing
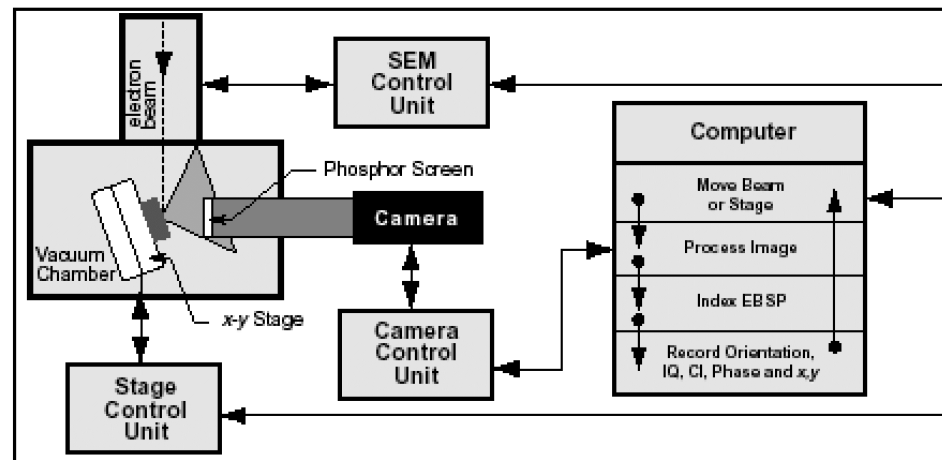
Electron backscatter diffraction (EBSD)

incoherent incoming wave field (produced by inelastic scattering)

coherent outgoing wave field (backscatter diffraction)

sample

detector (with directional sensitivity)
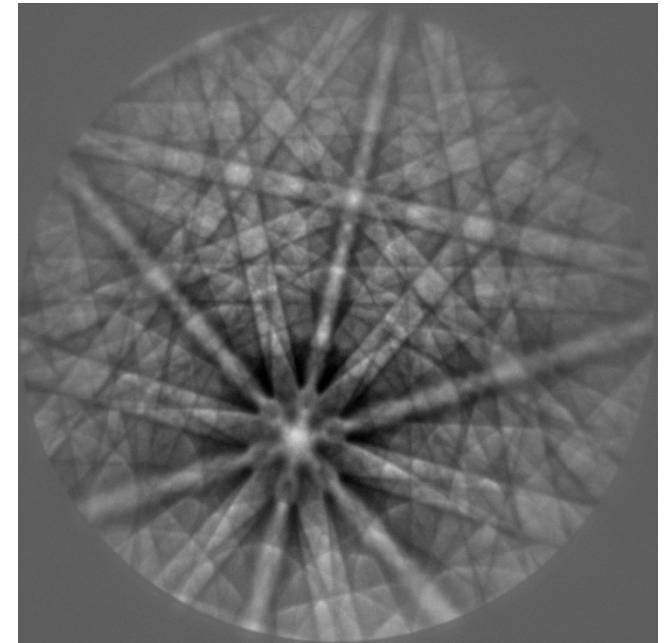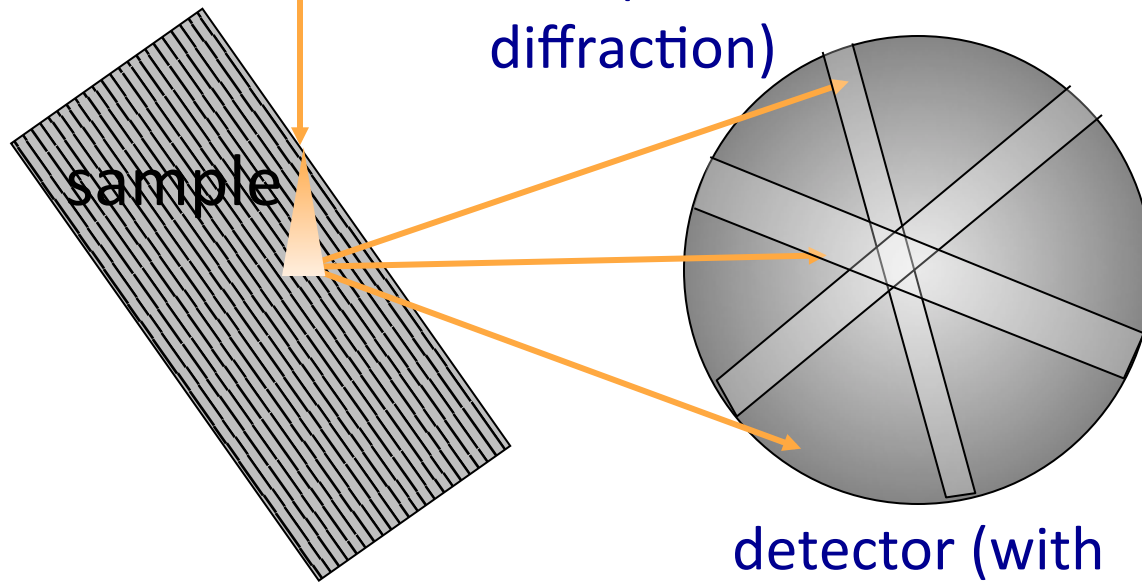
A typical EBSD pattern (Niobium, 15 kV)

9

EBSD-based orientation microscopy

e⁻

1 1 1

TD

RD

deformed

9.000
5.800
3.737
2.408
1.552
1.000
0.644

1 1 1

TD

RD

recrystallised grains

10.00 μm = 100 s

COMPO 15.0kV X450 10μm

15° 180°
2° 5°
5° 10°
10° 15°

BSEI of a partially
recrystallised IF steel

inverse pole
figure map

grain boundary
character

pole figures
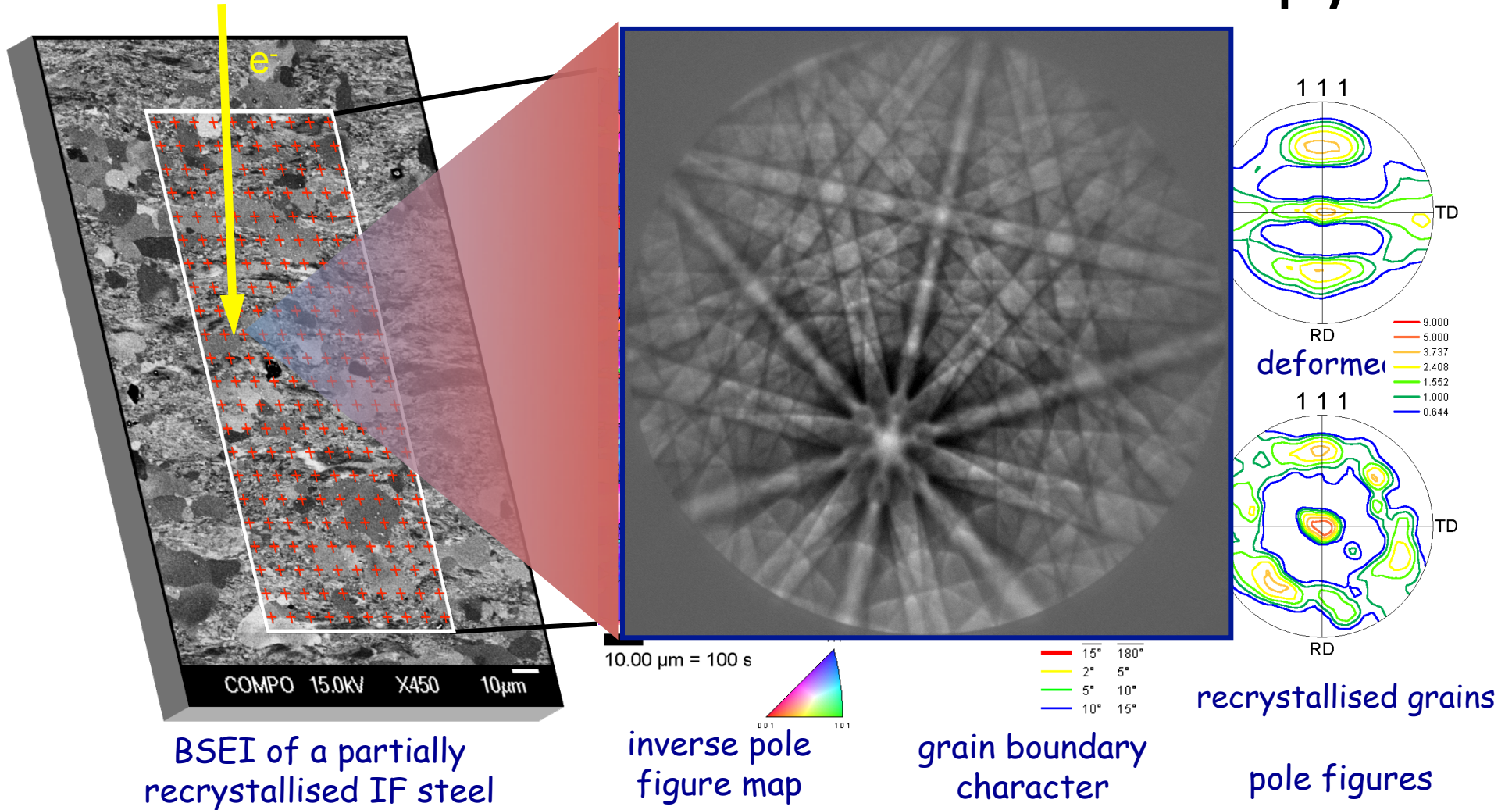
- By nature a "quantitative" technique!
- Spatial resolution: lateral ~ 20 ... 80 nm, depth ~ 10 nm
- Angular resolution: conventional 0.5°, special > 0.01°
- Measured area: μm² ... cm²

Measurement of macroscopic textures using EBSD

Advantages:

⇨ direct measure of orientation distribution

⇨ no corrections for absorption, defocussing etc. required

⇨ no peak overlaps in multiphase materials
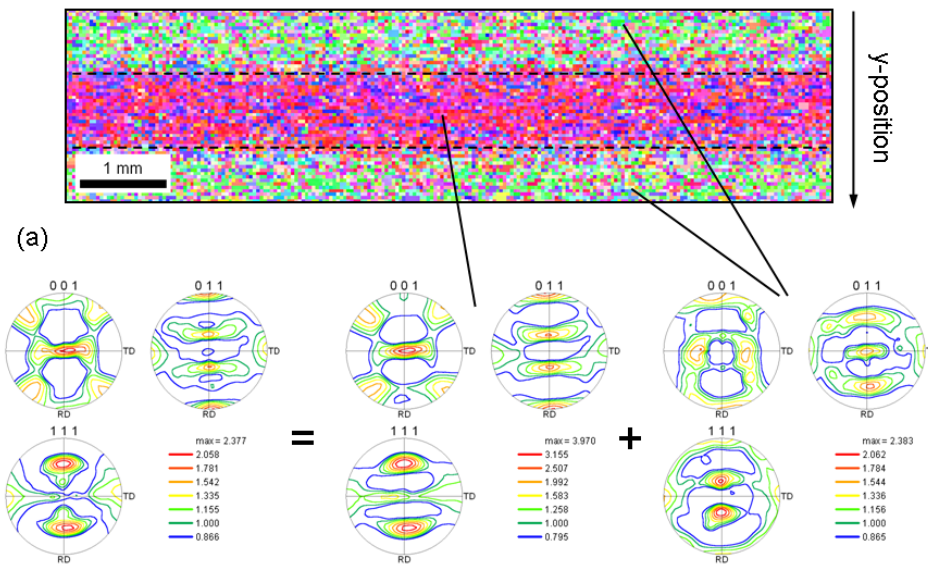
Texture field in a hot-rolled Si-steel

Challenges:

⇨ statistical representativeness

⇨ spatial resolution on highly deformed or sub-μ material
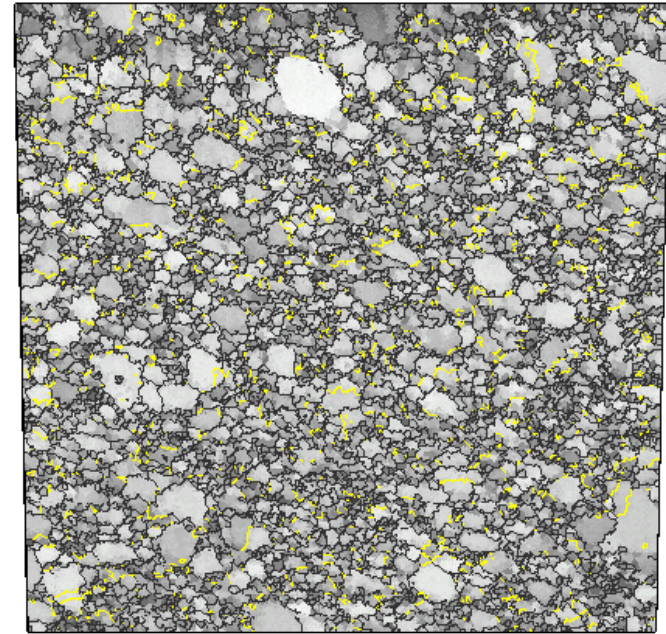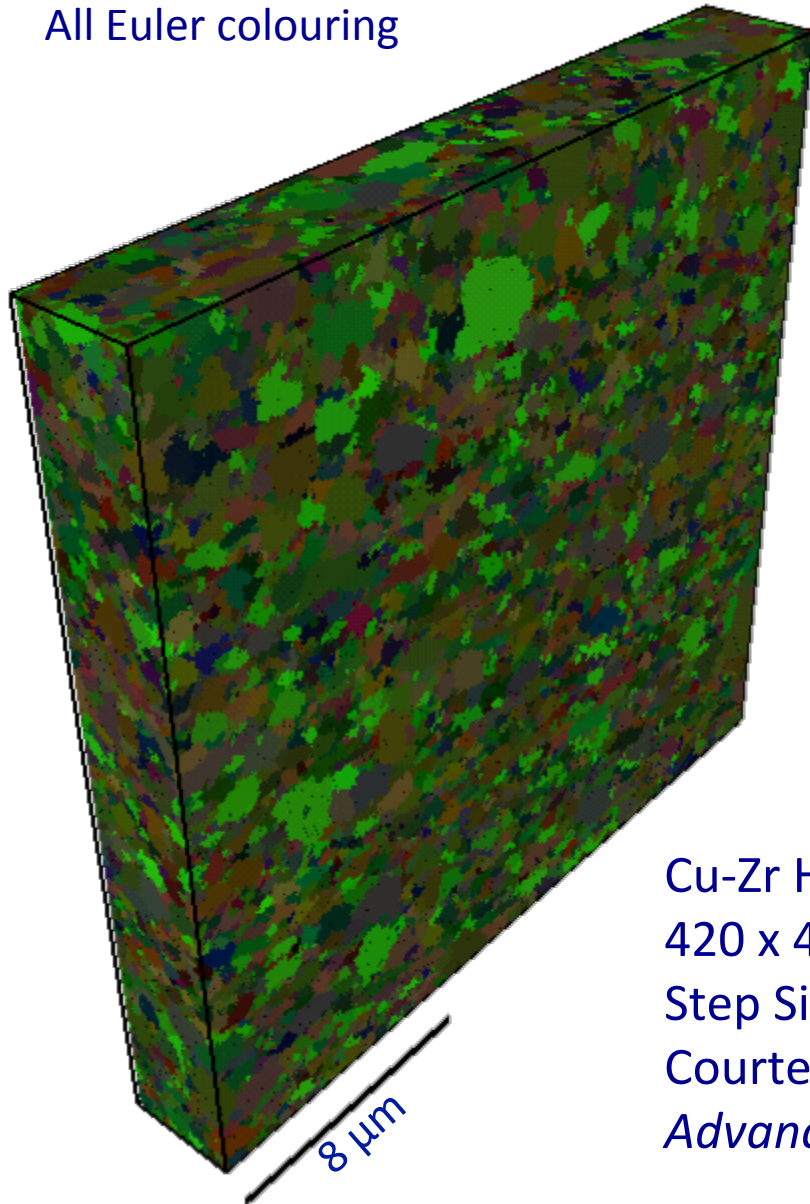
Spatial resolution of EBSD

primary electron energy: 15 kV

length: 60...100 nm

half width: 10...17 nm

depth: 5...10 nm
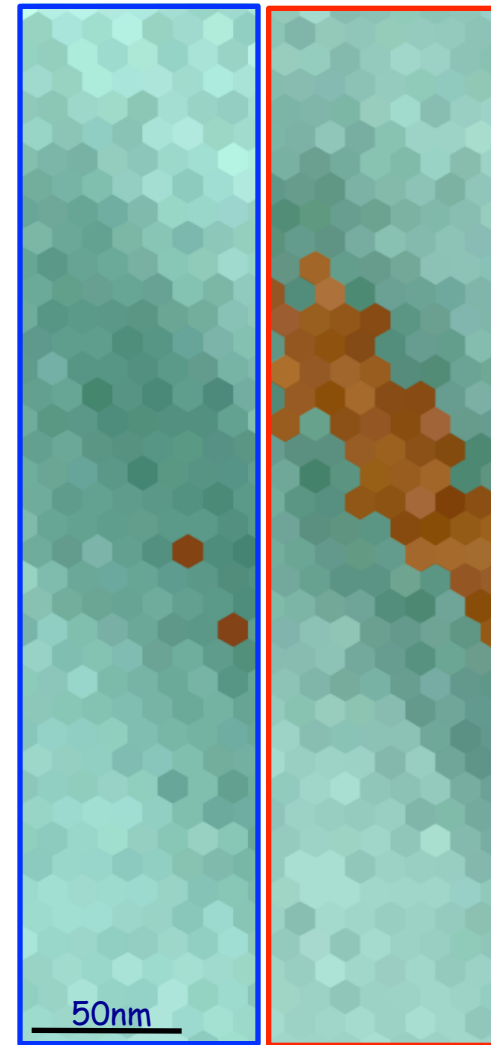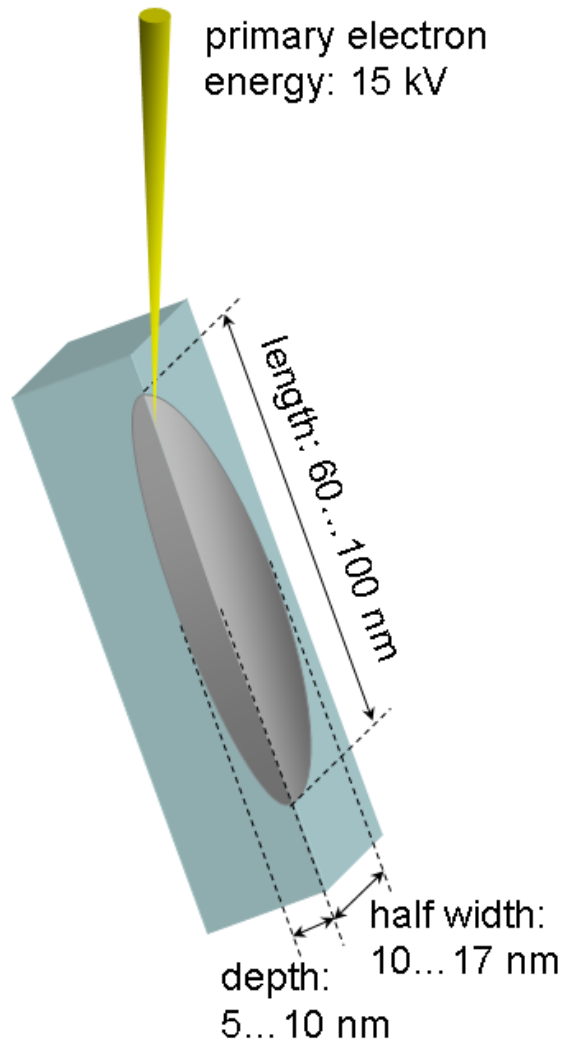
50nm

15kV   7.5kV

A word on spatial resolution of SEM and TEM

$20 \cdot 10^3$ nm^3

15 keV

30 nm

75 nm

10 nm

EBSD-based
orientation microscopy

200 keV

$2 \cdot 10^3$ nm^3

75 nm

TEM-based orientation microscopy
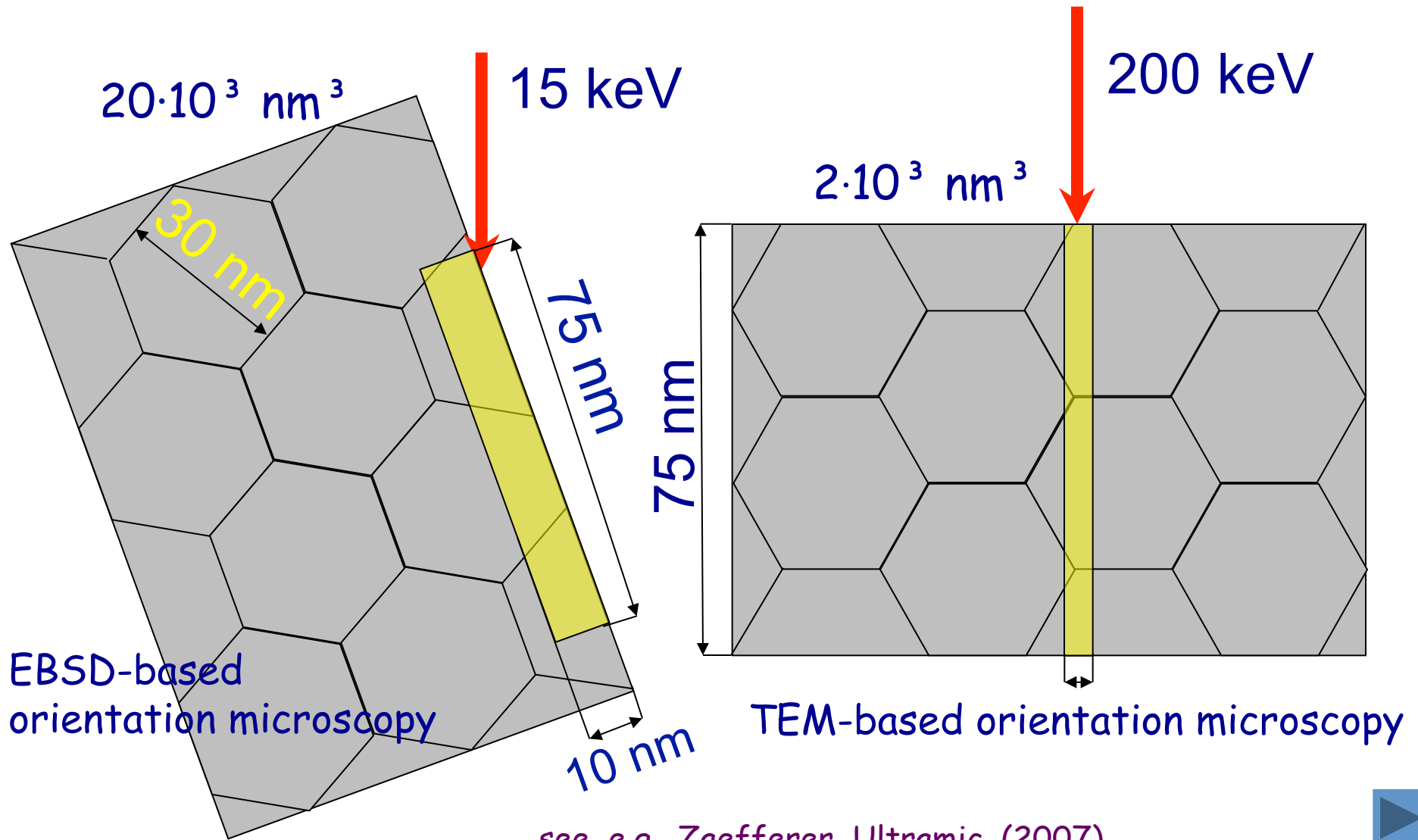
see, e.g., Zaefferer, Ultramic. (2007)

An example data set

All Euler colouring

Cu-Zr HPT-deformed & annealed
420 x 420 x 60 voxels; approx. 40,000 grains
Step Size 50 nm
Courtesy A. Khorashadizadeh, MPIE
Advanced Engineering Materials **13** 237 (2011)

8 μm

14

Applications of 3D orientation microscopy

- 3D morphology

- 5-parameter grain boundary characterization

- Calculation of GND densities using the Nye-tensor approach

- Coupling with modelling

28 μm

28 μm

17 μm

ED
ND
TD

111

001   101
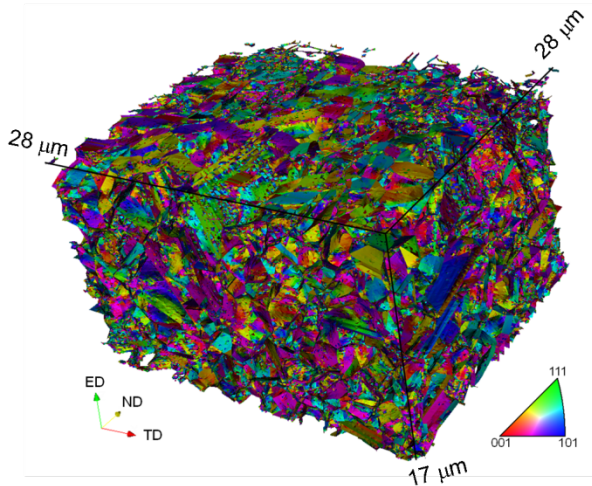
Interfaces colored according to their miller indices

17.60
16.68
15.76
14.84
13.92
13.00

Total GND density (log $1/m^2$)

15

Grain Boundaries

3D EBSD: serial sectioning &
reconstruction with software QUBE
(Konijnenberg, Bruker Nano)

$(hkl)_{gb}$

$<uvw>$

53°<323>

57° <110>

Tilt
boundary

Twist
boundary

$1\ \mu m$

ω
$[uvw]$

Grain boundary
normal vectors
$(hkl)_{gb}$
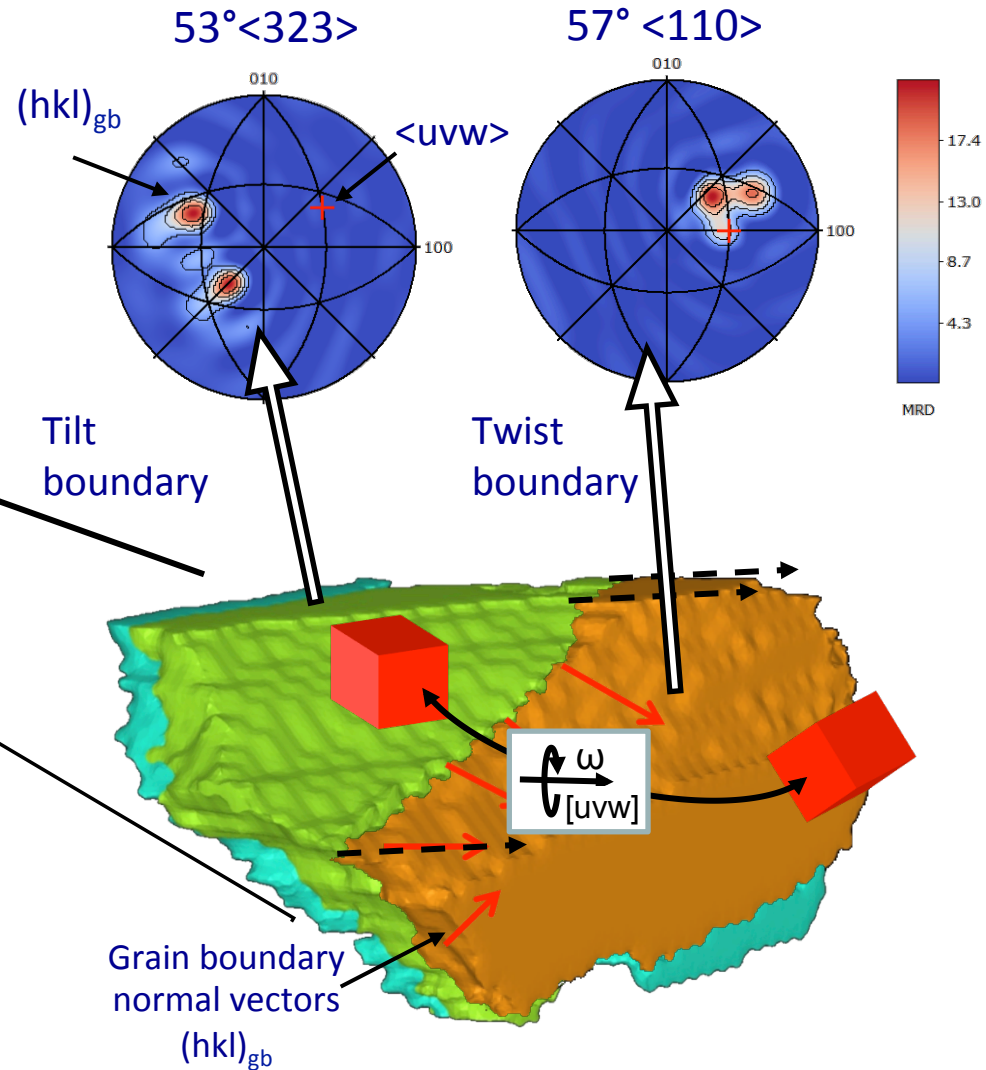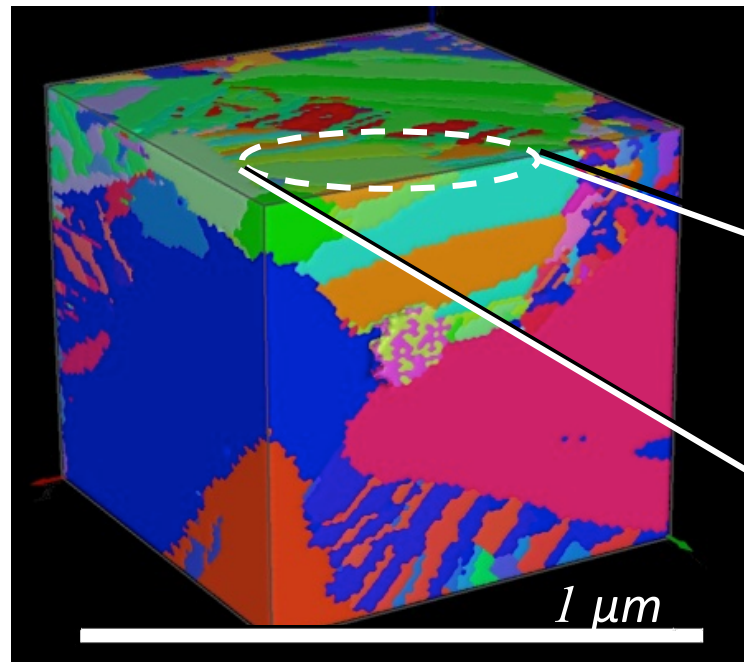
5 rotational parameters: ω (1), <uvw> (2), $(hkl)_{gb}$ (2)

Phase and structure determination

FeNdB(-O)Material, Courtesy of T. Woodcock, IfW Dresden

Phases:
Nd2O3 – Red
NdO – Green
FeNdB - Gray

Unknown

cubic

trigonal
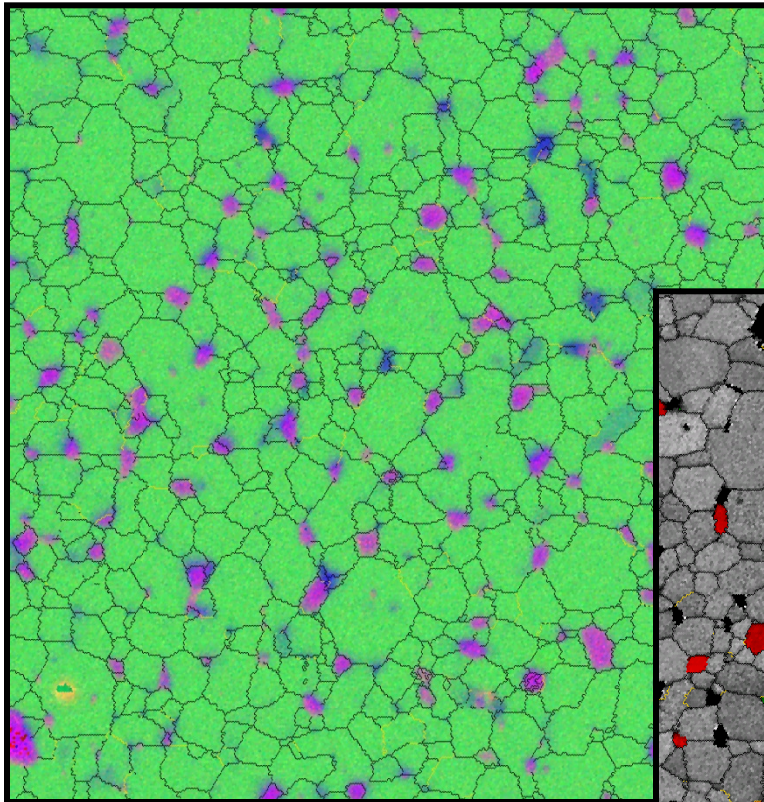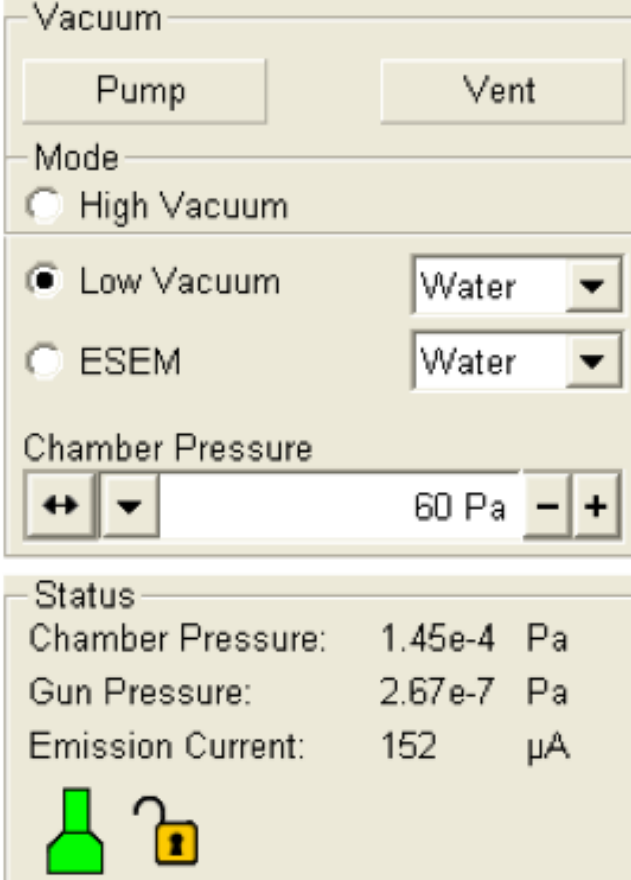
EDS Counts:
O-Red
Fe-Green
Nd-Blue

S. Zaefferer: Introduction to EBSD
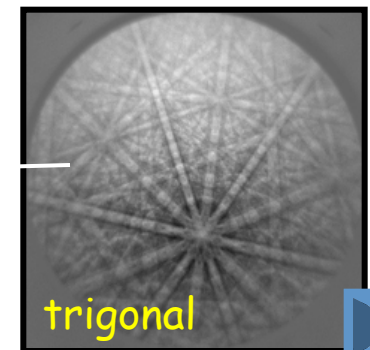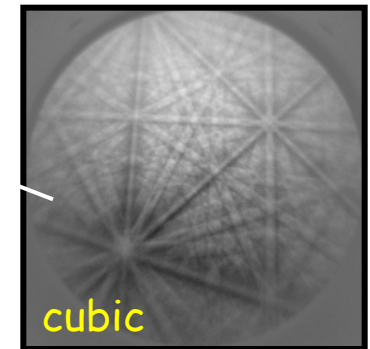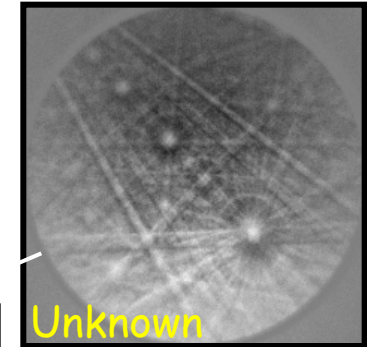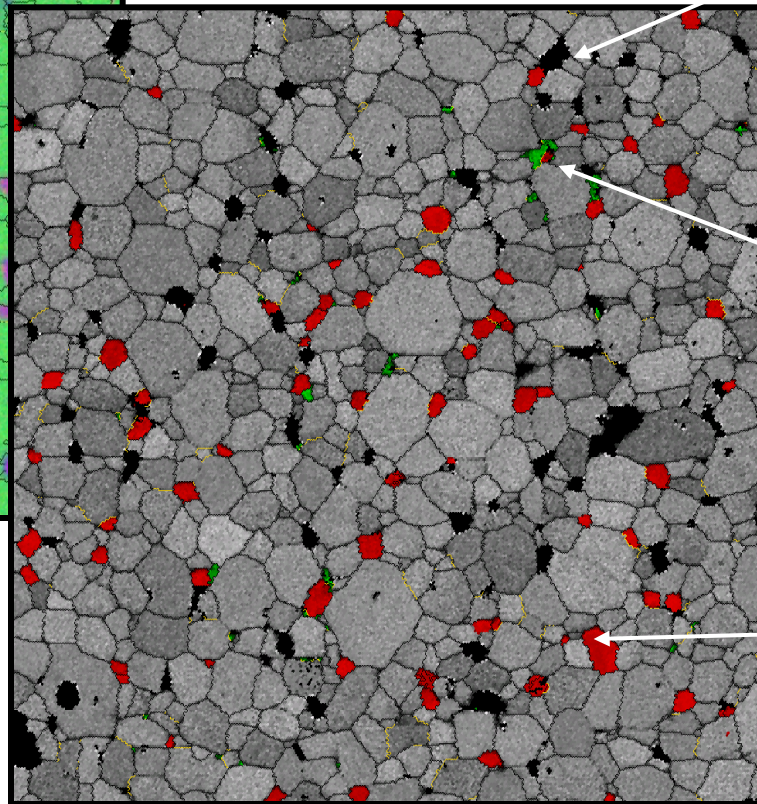
Vacuum System

- The Quanta FEG has 3 operating vacuum modes to deal with different sample types:
  - High Vacuum
  - Low Vacuum
  - ESEM (Environmental SEM)

- Low Vacuum and ESEM can use water vapours from a built-in water reservoir which is supplied by the user and connected to a gas inlet provided.

- Observation of outgassing or highly charging materials can be made using one of these modes without the need to metal coat the sample.

Vacuum Status

Icon	Status
	Column and Chamber Vented (Complete Shutdown - see Chapter 3)
	Column and chamber Pumping or Venting
	Column and Chamber Vacuum (ready for the microscope operation)
	Column Vacuum and Chamber Pumping or Venting
	Column Vacuum and Chamber Vented (ready for a sample and/or detector exchange)
	Stage axes – Lock (any one) / Unlock (all)
	Displays when Dynamic Focus is On
	Displays when Scan Rotation is not zero
	Displays when External scanning mode is On

Vacuum

Pump	Vent

Mode

○ High Vacuum

● Low Vacuum   Water ▼

○ ESEM   Water ▼

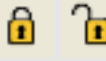**Chamber Pressure**

↔ | ▼ | 60 Pa   − | +

Status

Chamber Pressure: 1.45e-4   Pa
Gun Pressure: 2.67e-7   Pa
Emission Current: 152   µA

- **Green:** PUMPED to the desired vacuum mode
- **Orange:** TRANSITION between two vacuum modes (pumping / venting / purging)
- **Grey:** VENTED for sample or detector exchange
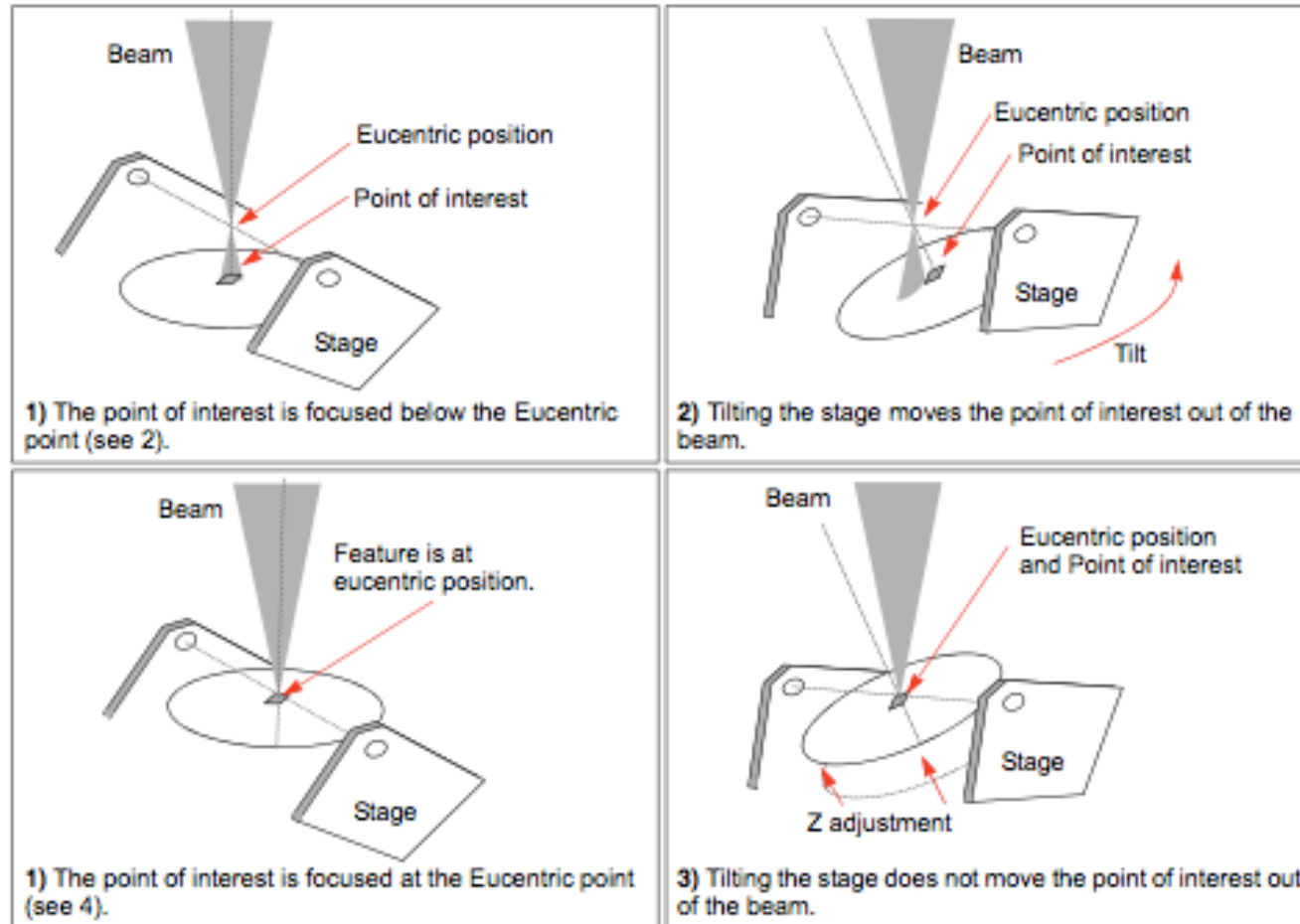
The Tool Bar

Surface Positioning detector
(automatically detect working
Distance)

Image Refreshing rate
Turtle: lower refresh rate (higher resolution)
Rabbit: Higher refresh rate (lower resolution)

3 µs

Automatic Contrast and Brightness (short key F9)

Eucentric Position

Top left panel:
Beam
Eucentric position
Point of interest
Stage
1) The point of interest is focused below the Eucentric point (see 2).

Top right panel:
Beam
Eucentric position
Point of interest
Stage
Tilt
2) Tilting the stage moves the point of interest out of the beam.

Bottom left panel:
Beam
Feature is at eucentric position.
Stage
1) The point of interest is focused at the Eucentric point (see 4).

Bottom right panel:
Beam
Eucentric position and Point of interest
Stage
Z adjustment
3) Tilting the stage does not move the point of interest out of the beam.

Note that eucentric position only occurs when the working distance is 10.
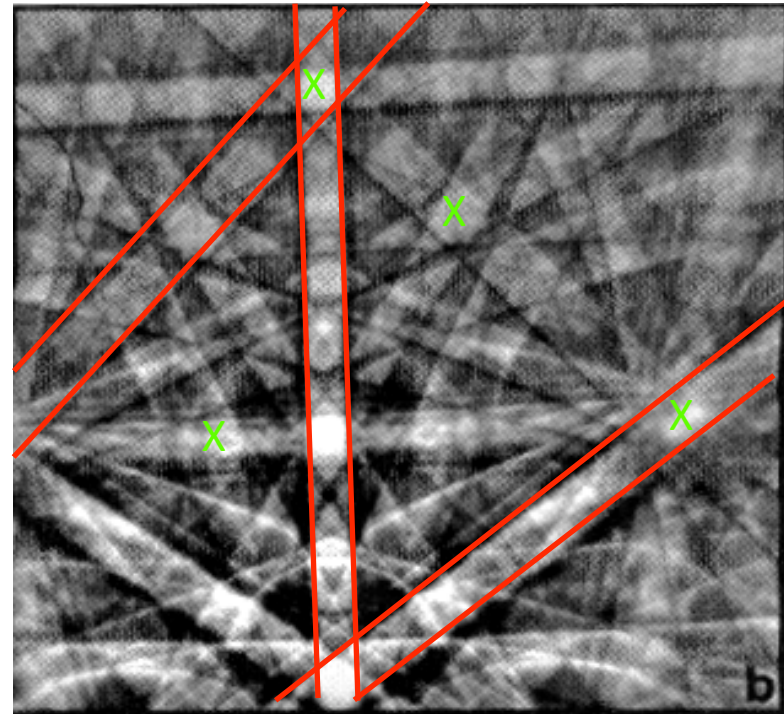
Diffraction Patterns-Source

- Electron <u>B</u>ack<u>s</u>catter Diffraction <u>P</u>atterns (EBSPs) are observed when a fixed, focused electron beam is positioned on a tilted specimen

- Tilting is used to reduce the path length of the backscattered electrons

- To obtain sufficient backscattered electrons, the specimen is tilted between 55-75°, where 70° is considered **ideal** because it maximizes the yield of backscattered electrons in the direction of the scintillation screen

- The backscattered electrons escape from 30-40 *nm* underneath the surface, hence there is a diffracting **volume**

- Note that $\delta x \approx$ 2 times spot size

   and $\delta y \approx$ 2.5 to 3 times spot size
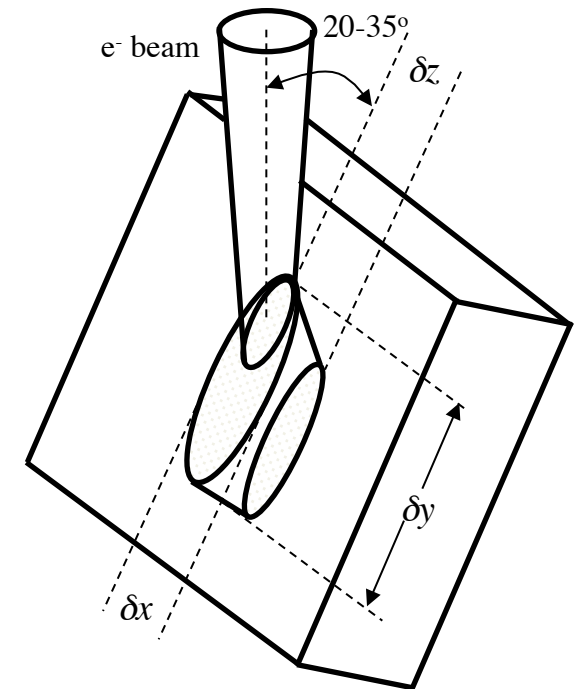
Diffraction Patterns-Anatomy of a Pattern

- There are two distinct features:
  - Bands
  - Poles

- Bands are intersections of diffraction *cones* that correspond to a family of crystallographic planes
- The small Bragg angles mean that the lines of intersection of the cones with the scintillation screen are effectively straight lines
- Band *widths* are proportional to the inverse interplanar spacing
- Intersection of multiple bands (planes) correspond to a pole of those planes (vector)
- Note that while the bands are bright, they are surrounded by thin dark lines on either side

Diffraction Pattern-SEM Settings

- Increasing the Accelerating Voltage increases the energy of the electrons Increases the diffraction pattern intensity

- Higher Accelerating Voltage also produces narrower diffraction bands (*a vs. b*) and is necessary for adequate diffraction from coated samples (*c vs. d*)

- Larger spot sizes (beam current) may be used to increase diffraction pattern intensity

- High resolution datasets and non-conductive materials require lower voltage and spot size settings

- For insulators (most ceramics), consider using a low-vacuum "environmental" SEM.

40 kV   10 kV
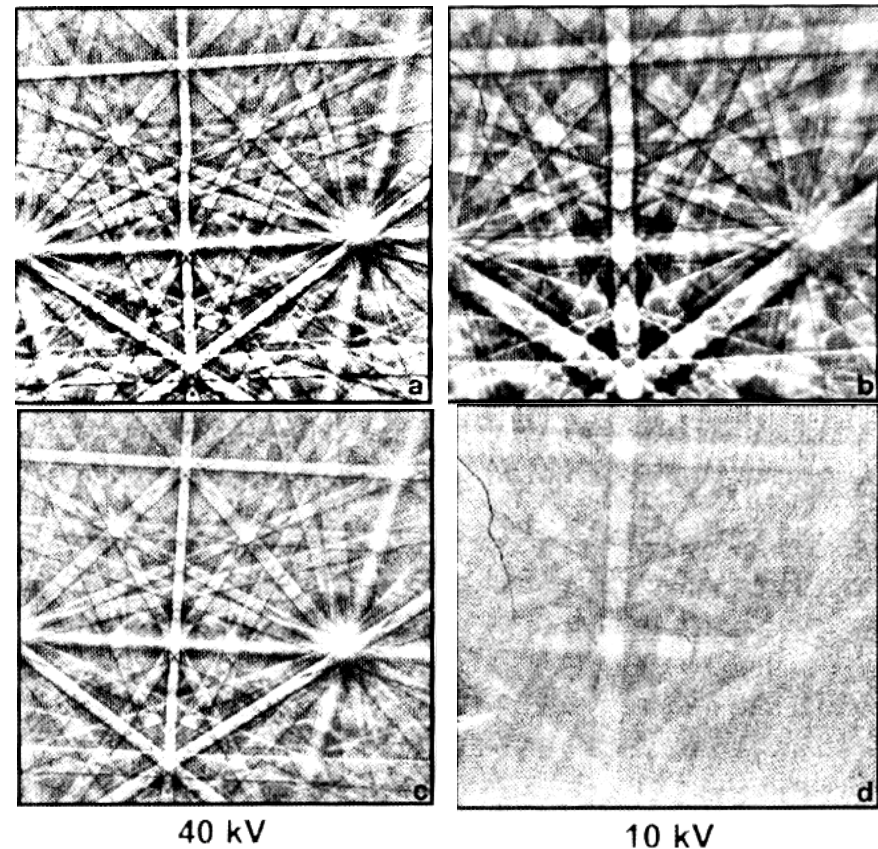
System setup-Material data

- In order for the system to index diffraction patterns, three material characteristics need to be known:
  - Symmetry
  - Lattice parameters
  - Reflectors

- "Reflector" means a particular set of lattice planes ("hkl" values)

- Information for most materials exist in TSL .mat files

- "Custom" material files can be generated using the ICDD **powder diffraction data files**; try searching http://www.crystallography.net/new.html to find information. For example, the MTEX software reads CIF files to determine crystal structure and symmetry.

- Symmetry and Lattice parameters can be readily input from the ICDD data

- Reflectors with the highest intensity should be used (4-5 reflectors for high symmetry; up to 12 reflectors for low symmetry)

Phase Parameters

Material Name
Titanium

Formula

Symmetry Point Group
Hexagonal (D6h) [6/mmm]   Categories...

Lattice Parameters

a [Å]	2.95	alpha	90
b [Å]	2.95	beta	90
c [Å]	4.68	gamma	120

Reflectors

	hkl	F_hkl	d-spacing
☑ ●	0 0 0 2	0.0	2.340
☑ ●	1 0 -1 1	0.0	2.242
☑ ●	1 1 -2 0	0.0	1.475
☑ ●	1 0 -1 0	0.0	2.555
☑ ●	1 1 -2 2	0.0	1.248
☑ ●	2 0 -2 1	0.0	1.333

Apply

System setup-Material data

Phase Parameters

Material Name
Barium Titanium Oxide

Formula
BaTiO3

Symmetry Point Group
Tetragonal (D4h) [4/mmm] ▼ | Categories... |

Lattice Parameters

a [Å] 3.994   alpha 90
b [Å] 3.994   beta   90
c [Å] 4.038   gamma 90

Reflectors

	hkl	F_hkl	d-spacing
●	1 0 1	999.0	2.840
●	1 1 0	598.0	2.824
●	1 1 1	329.0	2.314
●	2 0 0	292.0	1.997
●	2 1 1	291.0	1.633
●	0 0 2	153.0	2.019

Apply

PDF # 831880, Wavelength = 1.54060 (A)

| 83-1880 | Quality: C |
| CAS Number: | |

Molecular Weight: 233.23
Volume[CD]:   64.36
Dx: 6.018   Dm:

Sys: Tetragonal
Lattice: Primitive
S.G.: P4mm (99)
Cell Parameters:
a 3.994   b   c 4.033
α   β   γ

I/Icor: 7.46
Rad: CuKa1
Lambda: 1.54060
Filter:
d-sp: calculated
ICSD # : 100804
Mineral Name:
Perovskite (Ba), syn

Ba Ti O3
Barium Titanium Oxide
Ref: Calculated from ICSD using POWD-12++, (1997)
Ref: Waesche, R., Denner, W., Schulz, H., Mater. Res. Bull., 16, 497 (1981)

d(A)	Int-f	h k l	d(A)	Int-f	h k l	d(A)	Int-f	h k l
4.0335	106	0 0 1	1.7863	54	2 1 0	1.2643	59	3 0 1
3.9945	201	1 0 0	1.6413	151	1 1 2	1.2631	60	3 1 0
2.8382	999	1 0 1	1.6333	291	2 1 1	1.2139	23	1 1 3
2.8245	598	1 1 0	1.4191	138	2 0 2	1.2054	45	3 1 1
2.3136	329	1 1 1	1.4122	71	2 2 0	1.1568	54	2 2 2
2.0167	153	0 0 2	1.3445	4	0 0 3	1.1153	7	2 0 3
1.9972	292	2 0 0	1.3372	27	2 1 2	1.1111	7	3 0 2
1.8003	39	1 0 2	1.3329	20	2 2 1	1.1078	8	3 2 0
1.7898	43	2 0 1	1.2742	51	1 0 3			

- Enter appropriate material parameters
- Reflectors should be chosen based on:
  - Intensity (higher intensity is better)
 - The number per *zone*

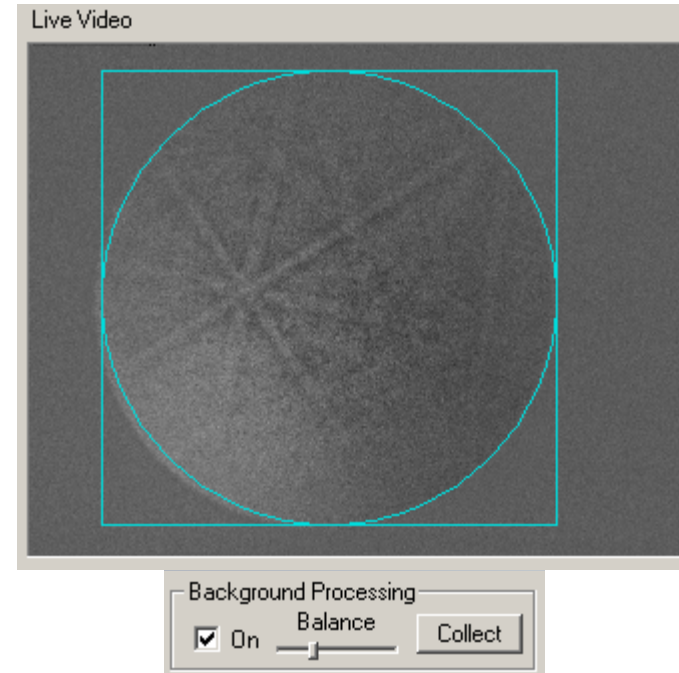# Pattern capture-Background
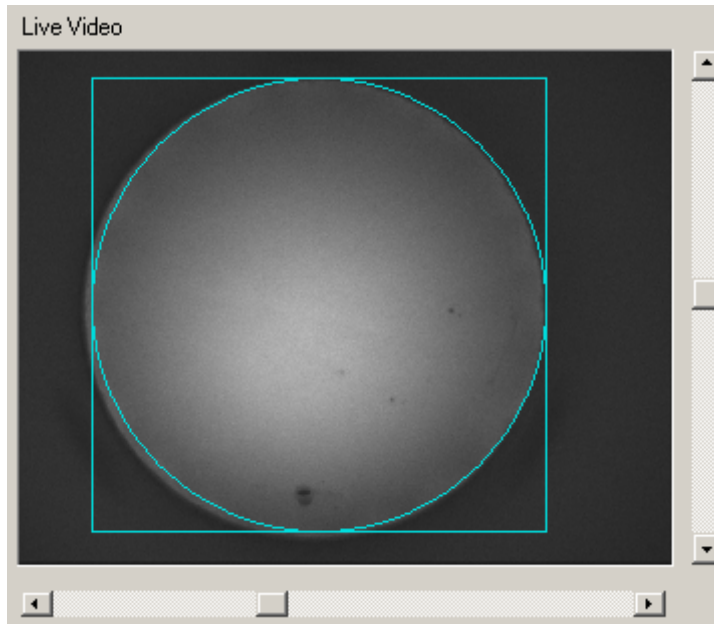
Live signal
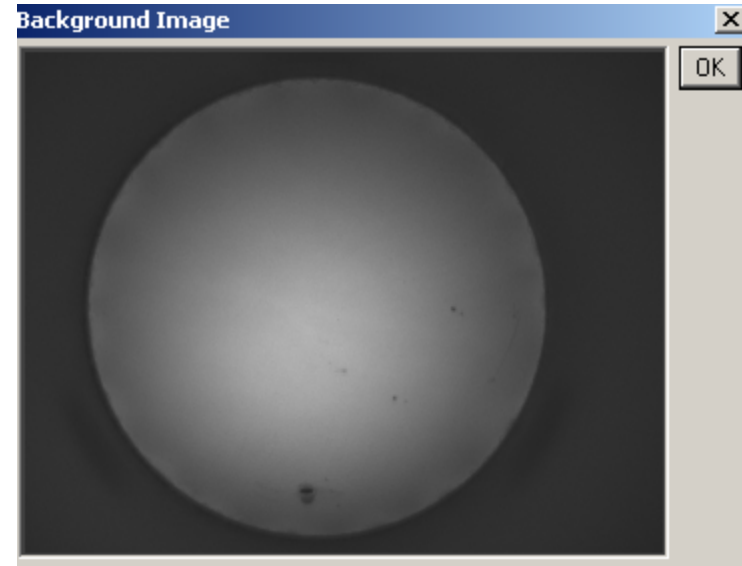
Averaged signal

- The background is the fixed variation in the captured frames due to the spatial variation in intensity of the backscattered electrons
- Removal is done by averaging 8 frames (SEM in TV scan mode)
- Note the variation of intensity in the images. The brightest point (marked with X) should be close to the center of the captured circle.
- The location of this bright spot can be used to indicate how appropriate the Working Distance is. A low bright spot = WD is too large and vice versa

Pattern capture-Background Subtraction
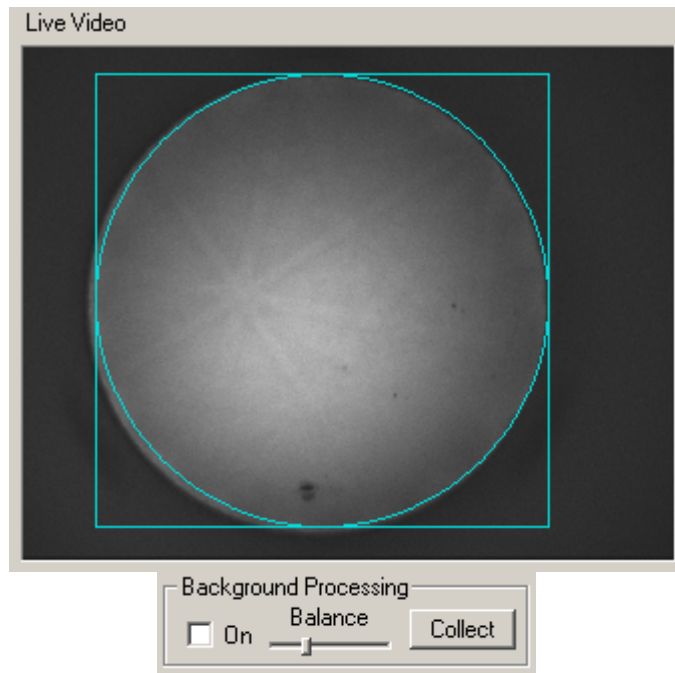
Without subtraction   With subtraction

- The background subtraction step is critical as it "brings out" the bands in the pattern
- The "Balance" slider can be used to aid band detection. Usually a slightly lower setting improves indexing *even though it may not appear better to the human eye*

Hough: Accumulator Diagram (1)

- The most basic idea behind the Hough transform is to take each point in the image and "spread it out" along the line defined by the transform in Hough space (angle vs. radius).

- See: Illingworth and Kittler Computer Vision, Graphics and Image Processing, **44**, 87-116 (1988); Duda & Hart (1972), "Use of Hough transform to detect lines and curves in picture", *Communications of the ACM*, **15** 11-15.
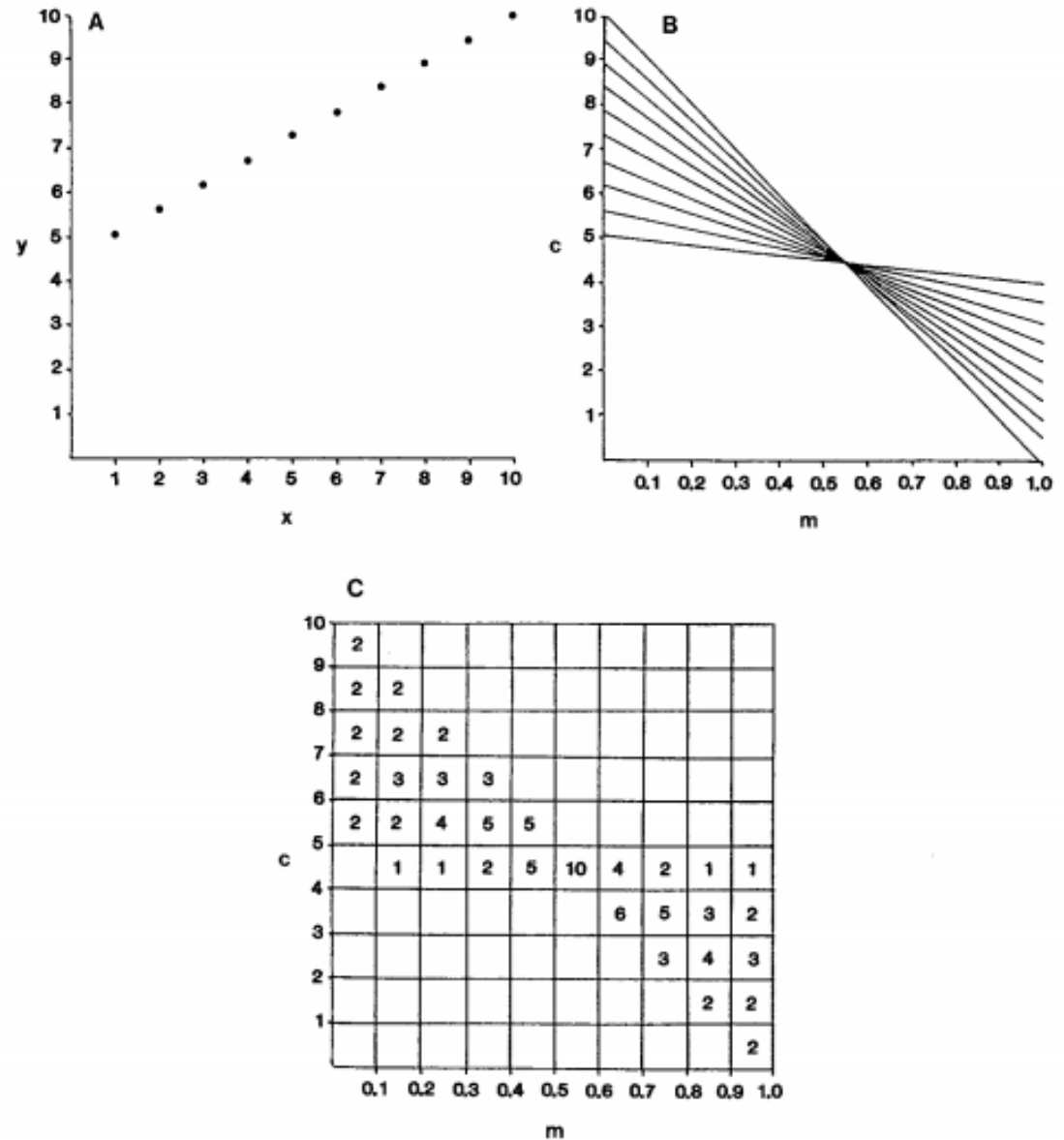
FIG. 1. The basis of the Hough transform for line detection: (A) (x, y) point image space; (B) (m, c) parameter space; (C) accumulator space corresponding to (B).

Hough: Accumulator Diagram (2)

- The following is quoted (12 iv 14) from:
  http://www.ebsd-image.org/documentation/reference/ops/hough/op/houghtransform.html
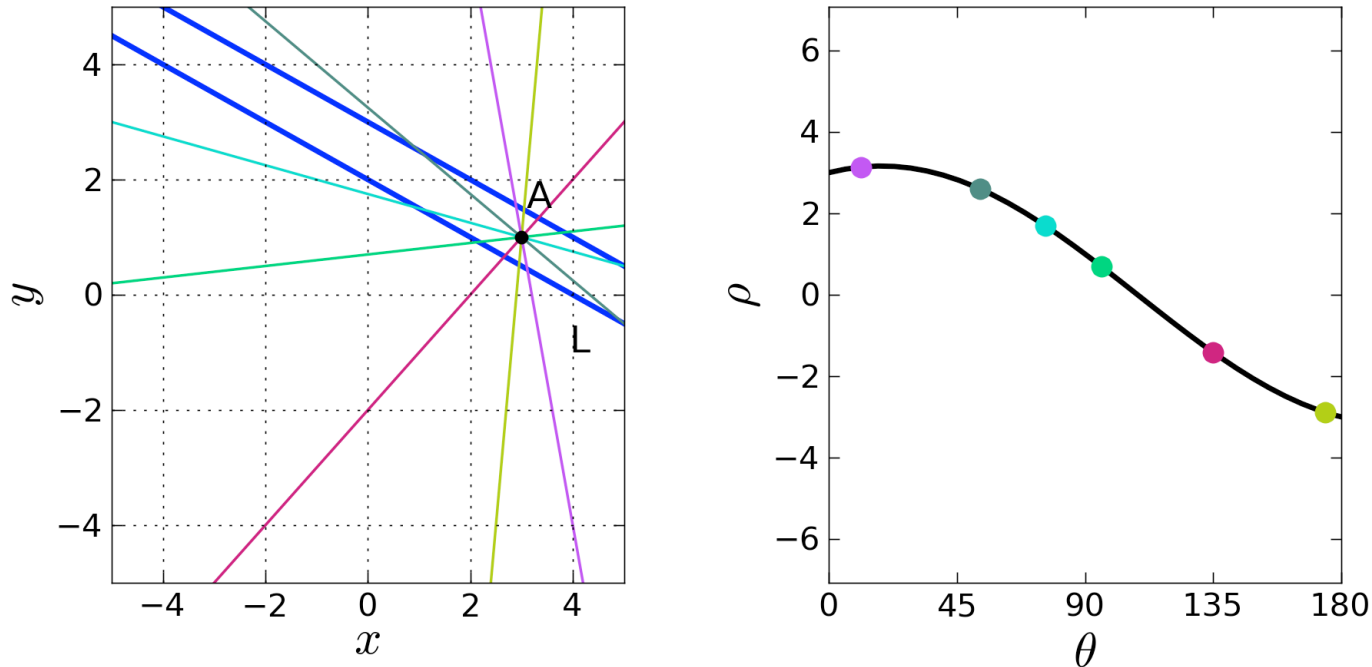
"Effectively, this transformation converts each pixel of the image space into a sinusoidal curve in the Hough space. The calculated ρ value is rounded to the closest pixel ρ_j. The intensity of the pixels (θ_j, ρ_j) that are part of the sinusoidal curve are augmented by the intensity of the corresponding pixel (x_i,y_i) in the image space. The accumulation of these intensities give rise to peaks in the Hough space which corresponds to the θ and ρ coordinates of the bands in the image space."

$$\rho = x_i \ cos\theta_j + y_i \ sin\theta_j$$

Hough: Accumulator Diagram (3)

- The following is quoted from:
  http://www.ebsd-image.org/documentation/reference/ops/hough/op/houghtransform.html
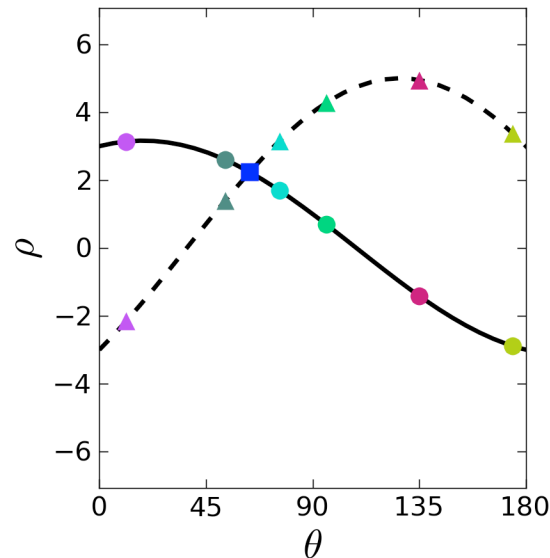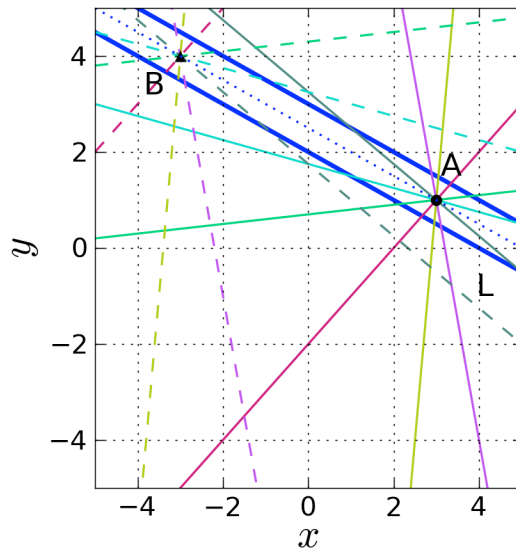
Additional Refs:
• Krieger Lassen (1994). Automated determination of crystal orientations from electron backscattering patterns. (Unpublished doctoral dissertation). The Technical University of Denmark.
• Tao & Eades (2005). Errors, artifacts, and improvements in ebsd processing and mapping. *Microscopy Microanalysis*, **11** 79-87.

"From the definition of the Hough transform, each pixel in the image space is transformed into a sinusoidal curve in the Hough space. The curve represents all the possible uni-dimensional lines that can pass through that pixel in the image space. A few lines are drawn in the figure above with their corresponding position in Hough space represented by circle markers. Only a small fraction of the lines are fully contained in the band, the rest of the lines cross it, but most of their pixels are outside the band.
If this geometrical construction is repeated for another pixel, B, of the band L, the same result is obtained. In the figure above, the lines passing by B and their equivalent representation in Hough space using triangular marker. All the lines or curves related to pixel B are drawn as dashed lines.
The lines inside of band L and passing by pixel B are the same lines that are also passing by pixel A. In Hough space, these lines end up having the same coordinates θ and ρ, forming a peak. The intersection of the sinusoidal curves therefore corresponds to the lines that are fully inscribed inside the band in the image space. The intensity at this intersection is higher than the background because of two interlinked reasons: a) the sinusoidal curve of the pixels in the band have a higher intensity that the one of the pixels outside of it; and b) the intensity of many sinusoidal curves is added at this intersection."

Detecting Patterns-Hough Transform

- A modified Hough Transform is used, and transforms the pattern so that it has a reference frame that is akin to polar coordinates
- Lines in the captured pattern with points (x_i, y_i) are transformed into the length of the orthogonal vector, ρ and an angle θ
- The **average** grayscale of the line (x_i, y_i) in Cartesian space is then assigned to the point (ρ, θ) in Hough space

Cartesian space   *Transformed (Hough) space*

I: $0 \leq \rho \leq n$; $0 \leq \theta \leq \pi/2$ II: $0 \leq \rho \leq n$; $\pi/2 < \theta \leq \pi$

III: $-n \leq \rho < 0$; $0 \leq \theta \leq \pi/2$ IV: $-n \leq \rho < 0$; $\pi/2 < \theta \leq \pi$

2n = Hough bin size
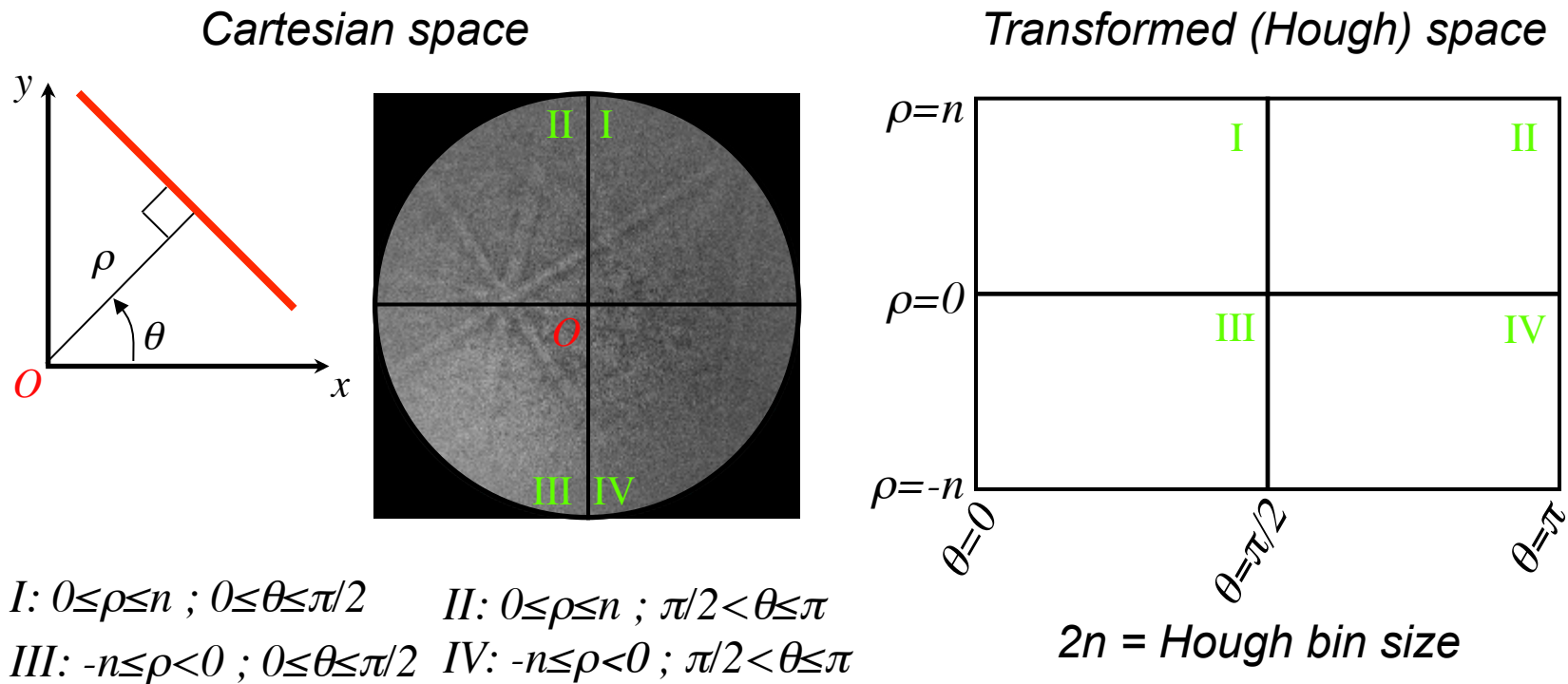
Detecting Patterns-The Hough of one band

Cartesian space
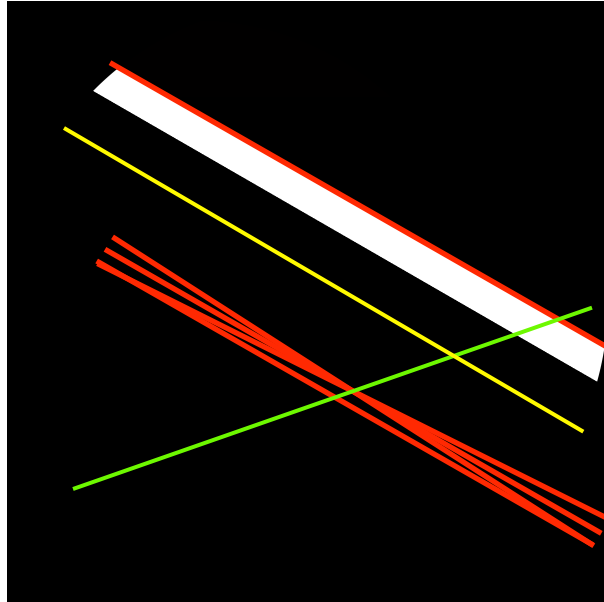
Transformed (Hough) space
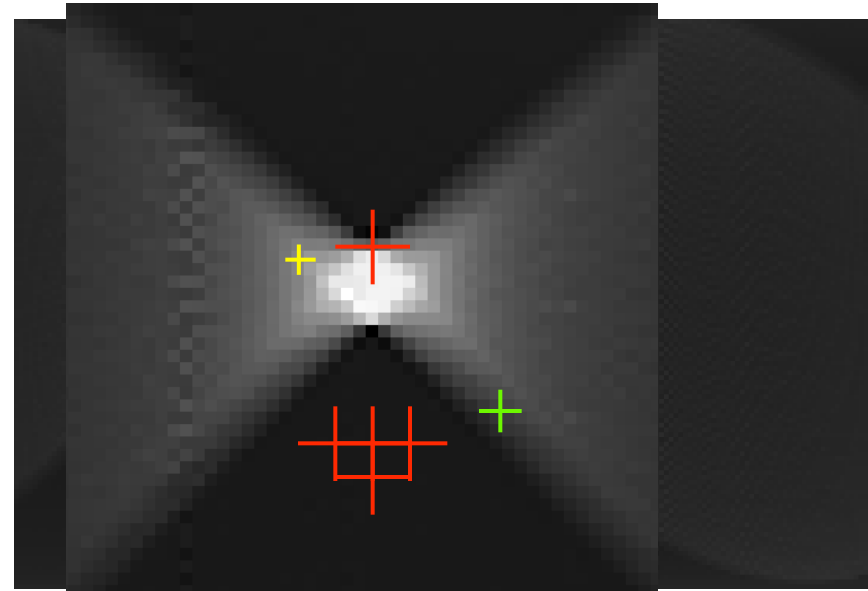
- Since the patterns are composed of bands, and not lines, the observed peaks in Hough space are a collection of points and not just one discrete point
- Lines that intersect the band in Cartesian space are on average higher than those that do not intersect the band at all

Setting up binning/mask

- Due to the shape of a band in Hough space, a multiplicative mask can be used to intensify the band grayscale

- Three mask sizes are available: 5 x 5, 9 x 9, 13 x 13. These numbers refer to the *pixel size* of the mask

-2	-6	-8	-6	-2
0	3	-1	3	0
1	8	20	8	1
0	3	-1	3	0
-2	-6	-8	-6	-2

5 x 5 mask

- A 5 x 5 block of pixels is processed at a time
- The grayscale value of each pixel is multiplied by the corresponding mask value
- The total value is added to the grayscale value at the center of the mask
- Note that the sum of the mask elements = zero
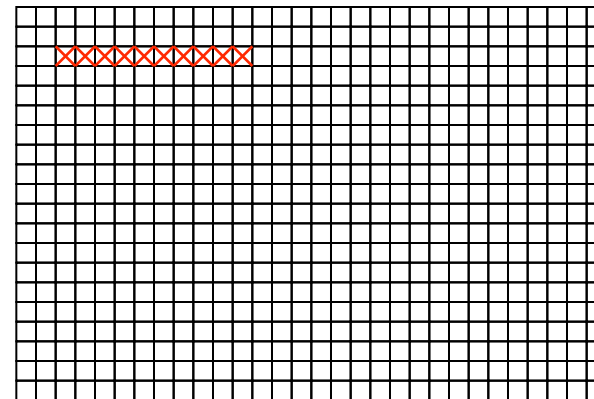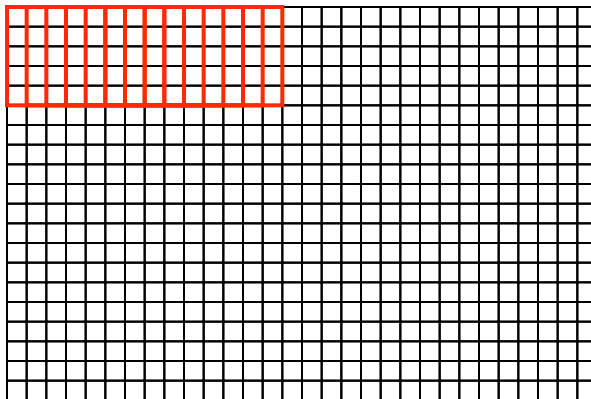
Detecting Patterns-Hough Parameters

More peaks	*Less peaks*
Use with low symmetry	Use with cubic materials
Increases the number of solutions	Decreases the number of solution

Symmetry ➜ 0   Symmetry ➜ 1
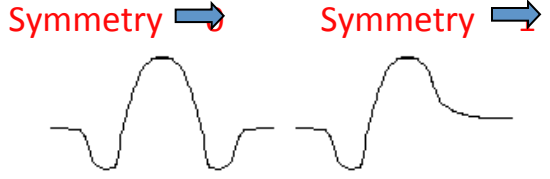
Smaller distance	*Larger distance*
Closely spaced bands	Sparsely distributed bands

Smaller mag.	*Larger mag.*
Band intensity is low	Band intensity is high

Binned Pattern Size=Hough resolution in ρ

Smaller size	*Larger size*
Use with broad bands	Use with narrower bands
Use for faster speed	Use with low symmetry materials

I.Q.=*Average* grayscale value of detected Hough peaks

Indexing Patterns-Identifying Bands

- ## Procedure:
  - Generate a lookup table from given lattice parameters and chosen reflectors (planes) that contains the **inter-planar angles**
  - Generate a list of all triplets (sets of three bands) from the detected bands in Hough space
  - Example (cubic): 100^111 = 54.7°; 111^110 = 35.3°; 100^110 = 45°
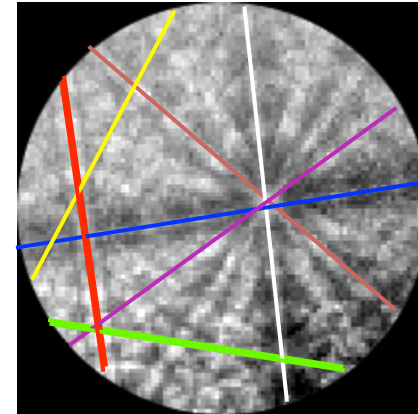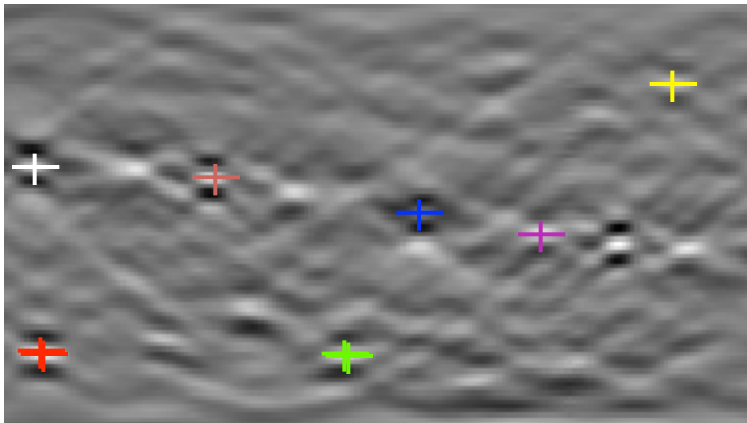
  - Calculate the inter-planar angles for each triplet set
  - Since there is often more than one possible solution for each triplet, a method that uses **all** the bands needs to be implemented

Indexing Patterns-Settings
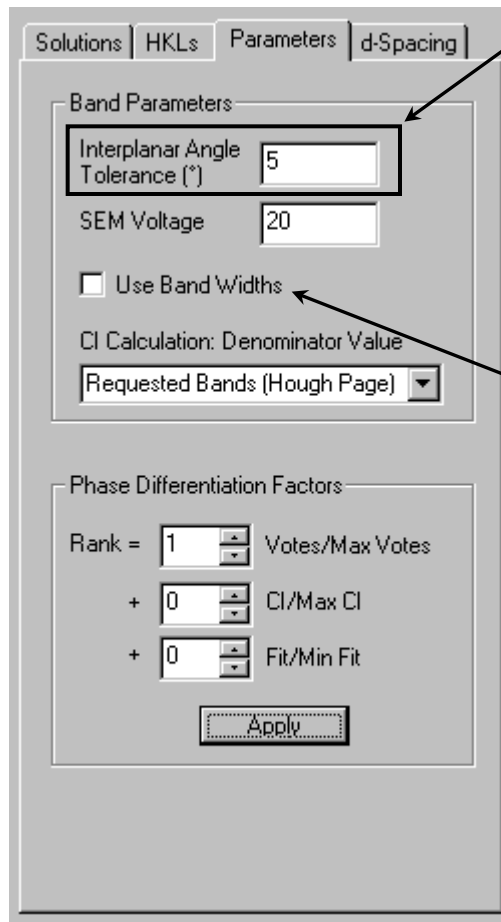
Tolerance = How much angular deviation a plane is allowed while being a candidate

Lower Tolerance	Larger Tolerance
Use if many bands are tightly bunched	Use with poorer patterns
Higher speed (eliminates possible solutions)	Lower speed (more possible solutions)

Band widths: check if the *theoretical* width of bands should be considered during indexing

If multi-phase indexing is being used, a "best" solution for *each* phase will be calculated. These values assign a weight to each possible factor:
- Votes: based on total votes for the solution/largest number of votes for all phases
- CI: ratio of CI/largest CI for all phases
- Fit: fit for the solution/best (smallest) fit between all phases

The indexing solution of the phase with the largest Rank value is chosen as the solution for the pattern

Indexing Patterns-Voting Scheme

- Consider an example where there exist:
  - Only 10 band triplets (i.e. 5 detected bands)
  - Many possible solutions to consider, where each possible solution assigns an *hkl* to each band. Only 11 solutions are shown for illustration
- Triplets are illustrated as 3 colored lines
- If a solution yields inter-planar angles within tolerance, a vote or an "x" is marked in the solution column
- The solution chosen is that with most number of votes
- Confidence index (CI) is calculated as

$$CI = \frac{\textbf{\textit{\# votes of S1 - \# votes of S2}}}{\textbf{\textit{number of band triplets}}} = \frac{10-4}{10} = 0.6$$

- Once the solution is chosen, it is compared to the Hough and the angular deviation is calculated as the fit

Solution #

Band triplets

votes

S1 (solution w/most votes)

S2 (solution w/ 2nd most votes)

Scanning

- The selection of scanning parameters depends on some factors:
  - Time allotted
  - Desired area of coverage (scan size)
  - Desired detail (step size)
- To determine if the scan settings are acceptable time-wise you must:
  - Start the scan
  - Use a watch and note how many patterns are solved per minute (n)
  - Divide the total number of points by n to get the total time
- To decide if the step size is appropriate for your SEM settings, use the following rough guide:

Spot Size Selection	Approximate Spot Size
1	5 nm
2	10 nm
3	20 nm
4	50 nm
5	100 nm
6	200 nm
7	500 nm

Supplemental

INTERPLANAR ANGLES (IN DEGREES) IN CUBIC CRYSTALS BETWEEN PLANES OF THE FORM $\{h_1k_1l_1\}$ AND $\{h_2k_2l_2\}$

$\{h_2k_2l_2\}$	$\{h_1k_1l_1\}$						
	100	110	111	210	211	221	310
100	0 90						
110	45 90	0 60 90					
111	54.7	35.3 90	0 70.5 109.5				
210	26.6 63.4 90	18.4 50.8 71.6	39.2 75.0	0 36.9 53.1			
211	35.3 65.9	30 54.7 73.2 90	19.5 61.9 90	24.1 43.1 56.8	0 33.6 48.2		
221	48.2 70.5	19.5 45 76.4 90	15.8 54.7 78.9	26.6 41.8 53.4	17.7 35.3 47.1	0 27.3 39.0	
310	18.4 71.6 90	26.6 47.9 63.4 77.1	43.1 68.6	8.1 58.1 45	25.4 49.8 58.9	32.5 42.5 58.2	0 25.9 36.9
311	25.2 72.5	31.5 64.8 90	29.5 58.5 80.0	19.3 47.6 66.1	10.0 42.4 60.5	25.2 45.3 59.8	17.6 40.3 55.1
320	33.7 56.3 90	11.3 54.0 66.9	61.3 71.3	7.1 29.8 41.9	25.2 37.6 55.6	22.4 42.3 49.7	15.3 37.9 52.1
321	36.7 57.7 74.5	19.1 40.9 55.5	22.2 51.9 72.0 90	17.0 33.2 53.3	10.9 29.2 40.2	11.5 27.0 36.7	21.6 32.3 40.5
331	46.5	13.1	22.0				
510	11.4						
511	15.6						
711	11.3						

Cubics: interplanar angles