



Understanding the TSL EBSD Data Collection System

27-750

Texture, Microstructure & Anisotropy

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Bassem El-Dasher, Gregory Rohrer

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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.

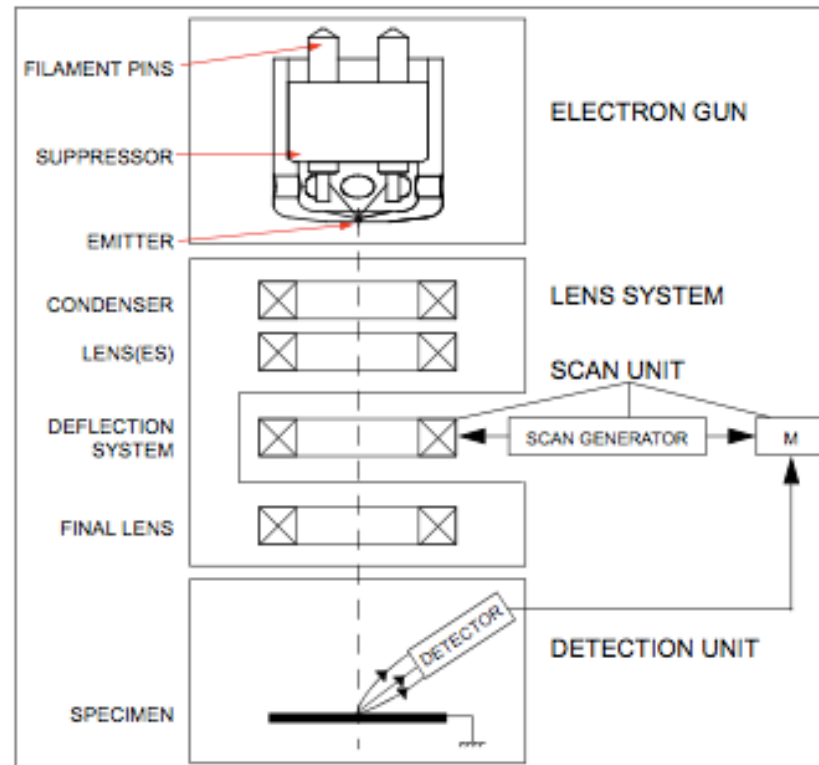
Questions (1)

- 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)

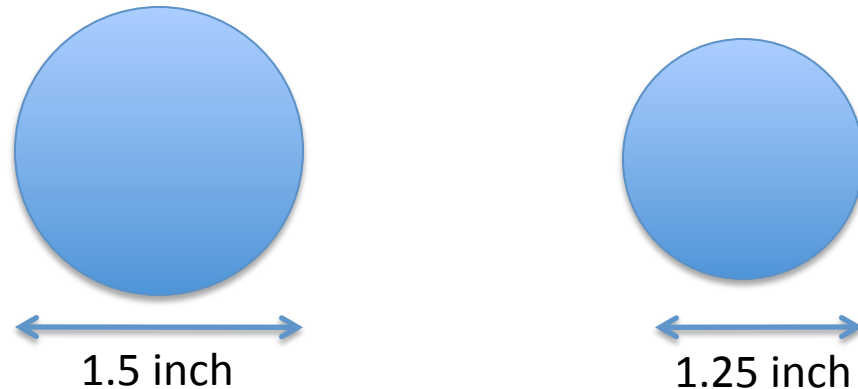
- What does background subtraction do?
- What is image quality?
- What are the coordinates of the image after the Hough transform has been applied?
- Why is the Hough transform effective for detecting lines?
- What are interzonal angles (in the context of an EBSD diffraction pattern)?
- What is the “confidence 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

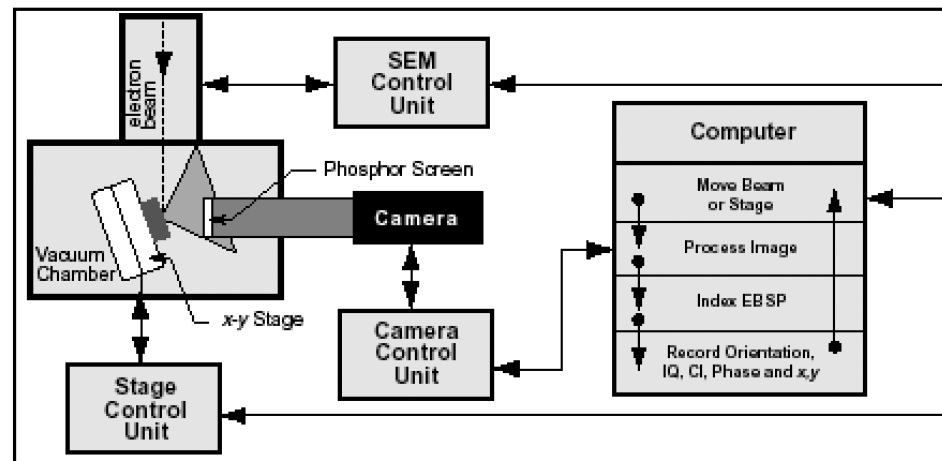
Sample Size effect



- All the samples need 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

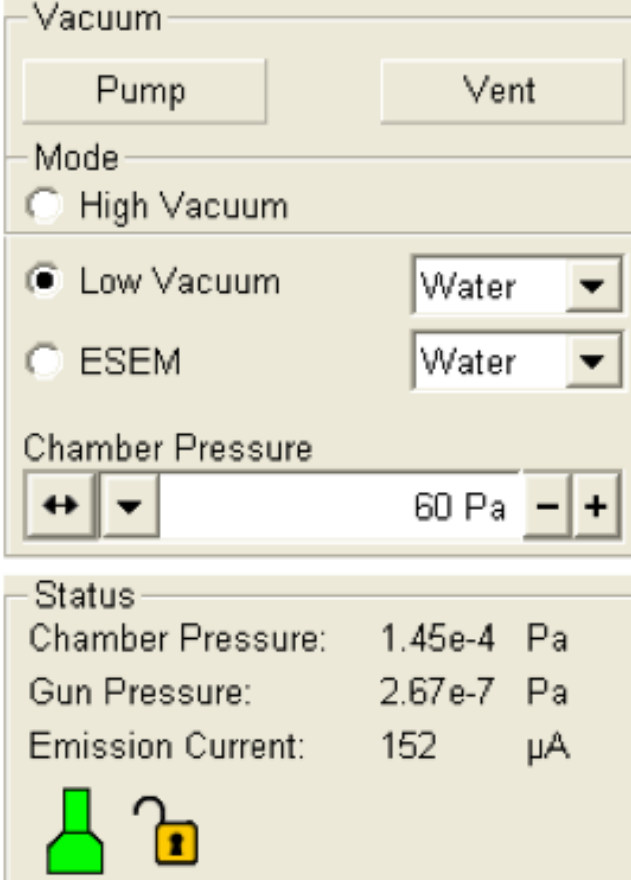
Diffraction Pattern-Observation Events

- 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



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

Pump Vent

Mode

High Vacuum

Low Vacuum Water

ESEM Water

Chamber Pressure



60 Pa

Status






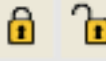



Chamber Pressure: 1.45e-4 Pa

Gun Pressure: 2.67e-7 Pa

Emission Current: 152 µA

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 μ A



- **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

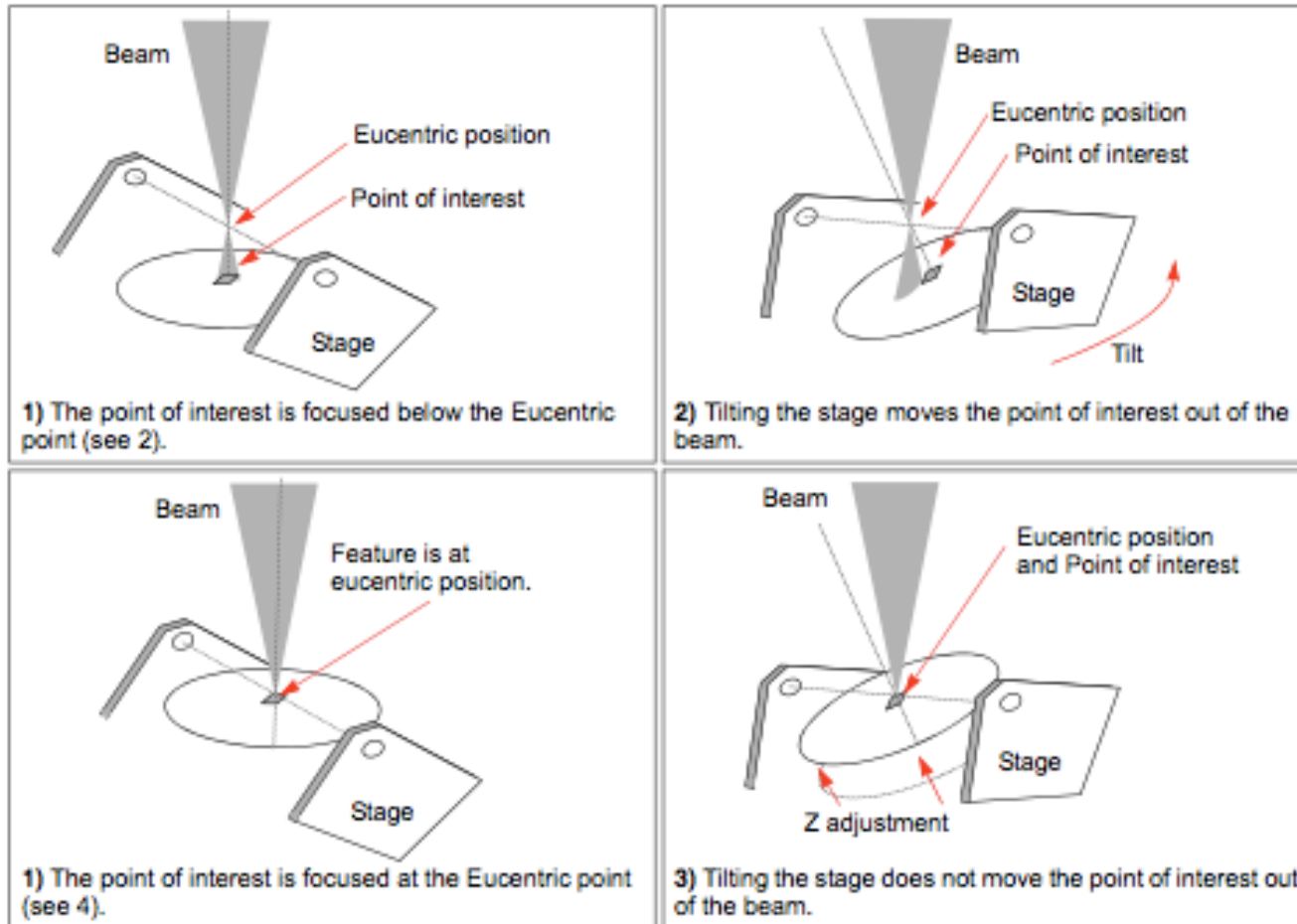
Surface Positioning detector
(automatically detect working
Distance)

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



Automatic Contrast and Brightness (short key F9)

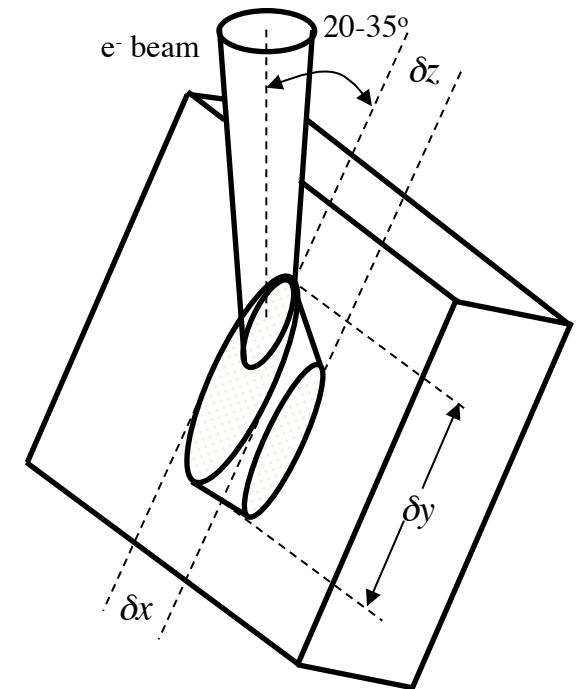
Eucentric Position



Note that eucentric position only occurs when the working distance is 10.

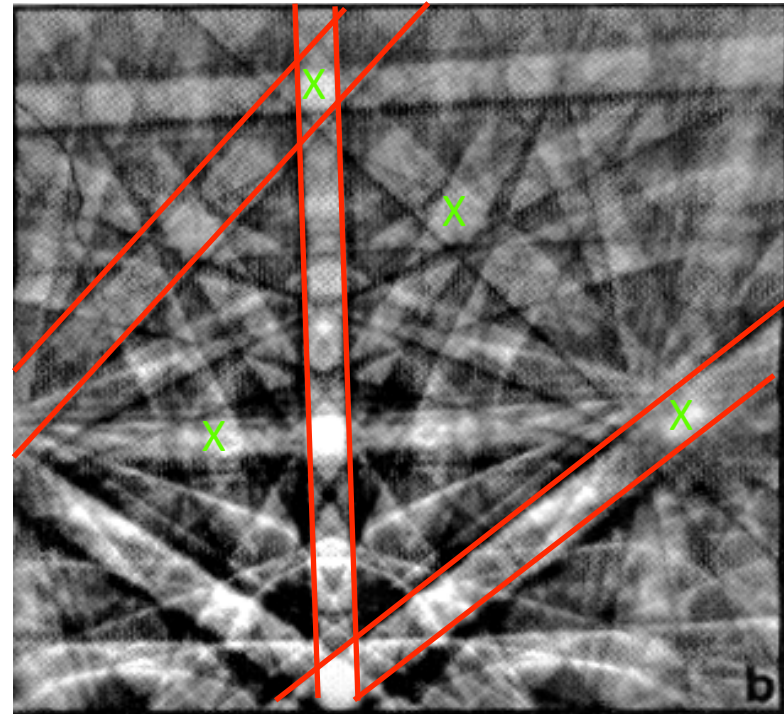
Diffraction Patterns-Source

- Electron Backscatter Diffraction Patterns (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 $55-75^\circ$, 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 $30-40\text{ nm}$ 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



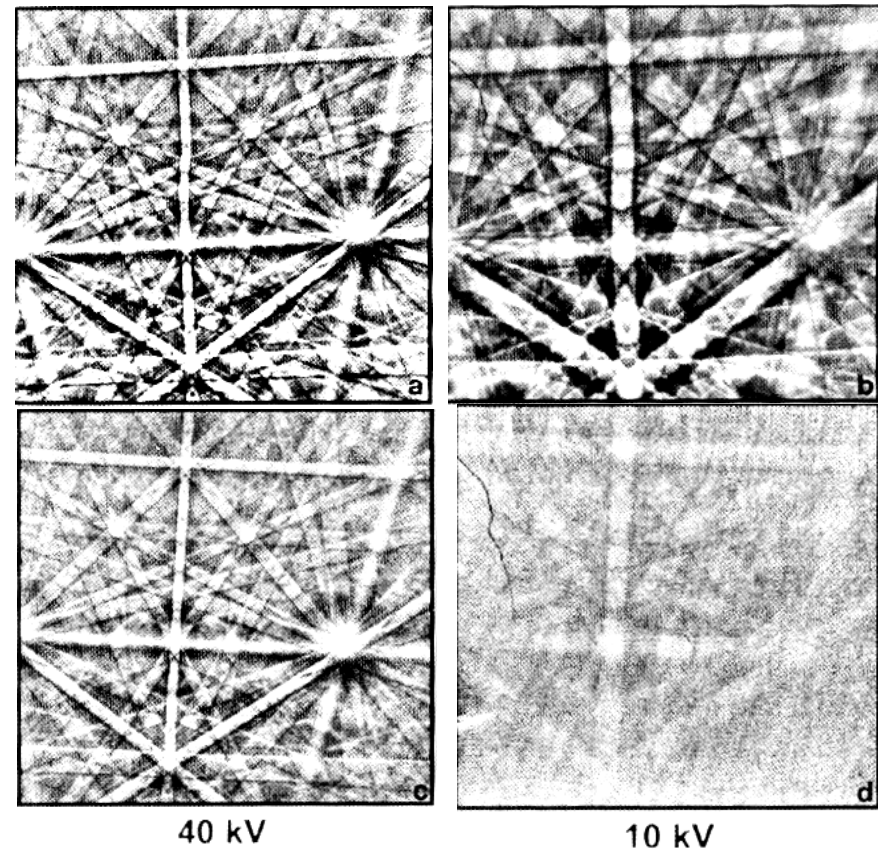
Diffraction Patterns-Anatomy of a Pattern

- 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.



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**
- 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
<input checked="" type="checkbox"/>	0 0 2	0.0	2.340
<input checked="" type="checkbox"/>	1 0 -1 1	0.0	2.242
<input checked="" type="checkbox"/>	1 1 -2 0	0.0	1.475
<input checked="" type="checkbox"/>	1 0 -1 0	0.0	2.555
<input checked="" type="checkbox"/>	1 1 -2 2	0.0	1.248
<input checked="" type="checkbox"/>	2 0 -2 1	0.0	1.222

Apply

System setup-Material data

Phase Parameters

Material Name
Barium Titanium Oxide

Formula
BaTiO₃

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

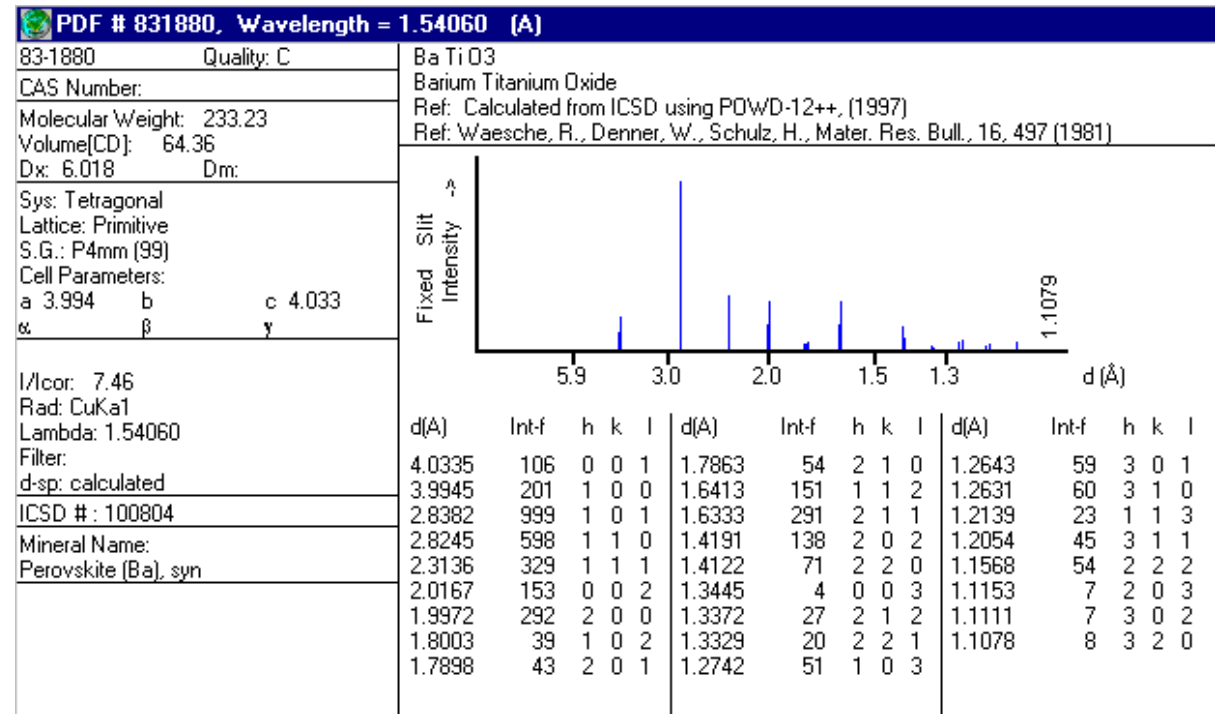
Lattice Parameters

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

Reflectors

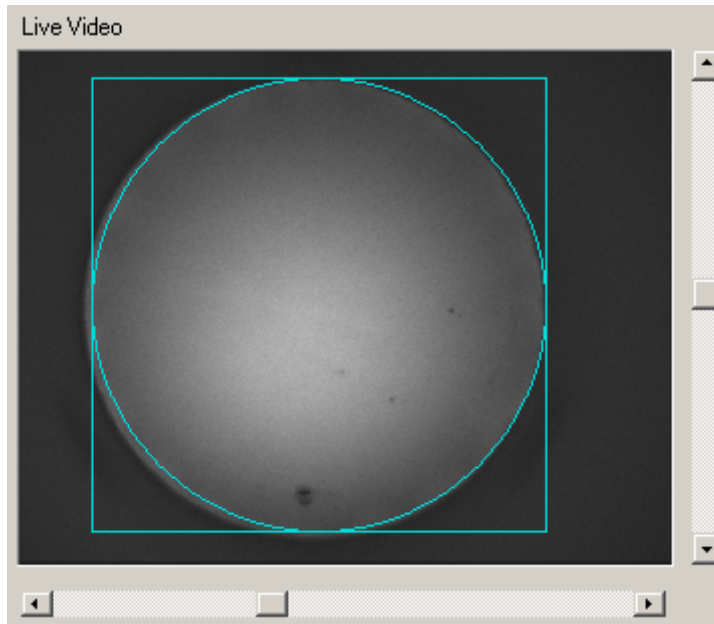
	hkl	F_hkl	d-spacing
<input checked="" type="checkbox"/>	1 0 1	999.0	2.840
<input checked="" type="checkbox"/>	1 1 0	598.0	2.824
<input checked="" type="checkbox"/>	1 1 1	329.0	2.314
<input checked="" type="checkbox"/>	2 0 0	292.0	1.997
<input checked="" type="checkbox"/>	2 1 1	291.0	1.633
<input checked="" type="checkbox"/>	0 0 2	152.0	2.010

Apply

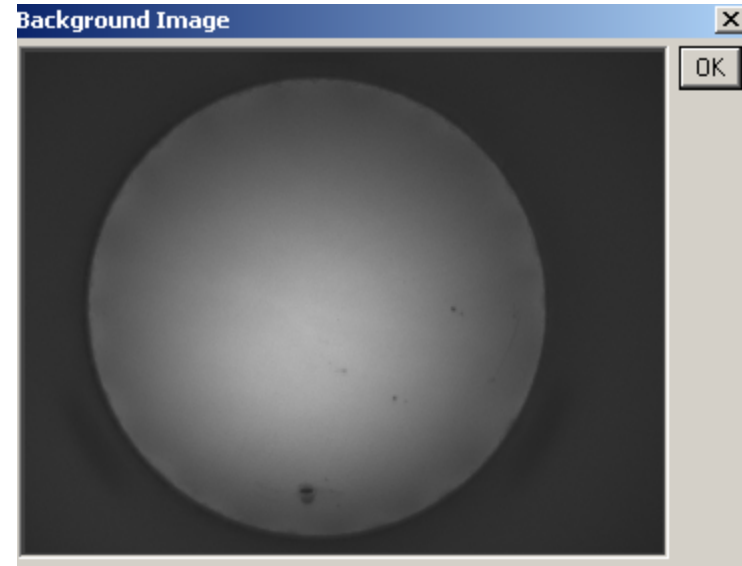


- Enter appropriate material parameters
- Reflectors should be chosen based on:
 - Intensity (higher intensity is better)
 - The number per *zone*

Pattern capture-Background



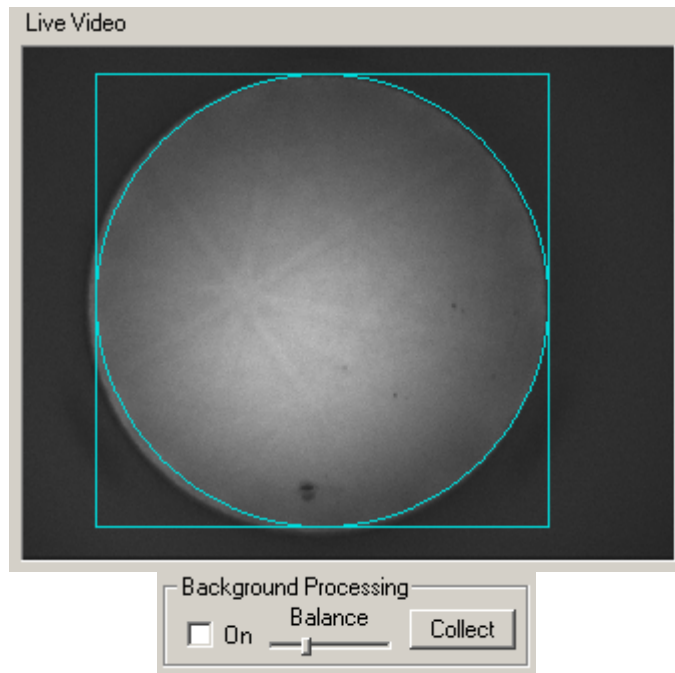
Live signal



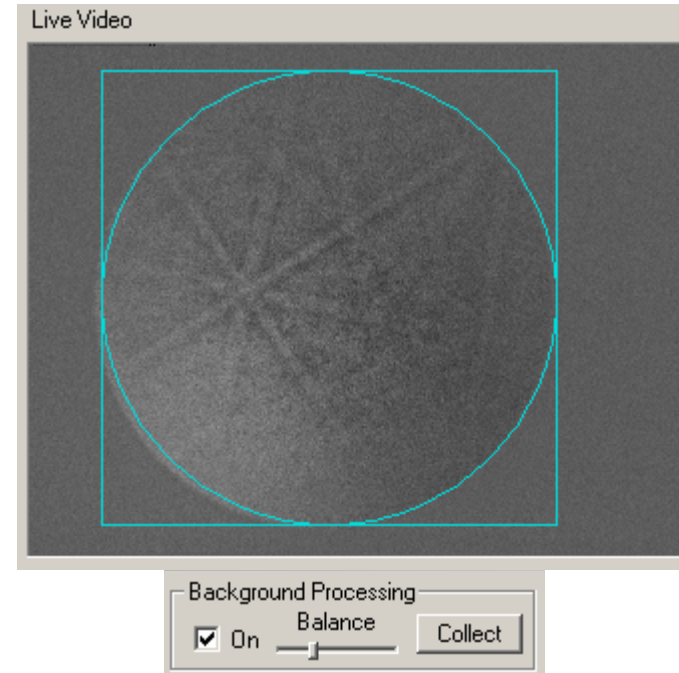
Averaged signal

- 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, R. O., & Hart, P. E. (1972), “Use of Hough transform to detect lines and curves in picture”, *Communications of the ACM*, **15**(1), 11-15.

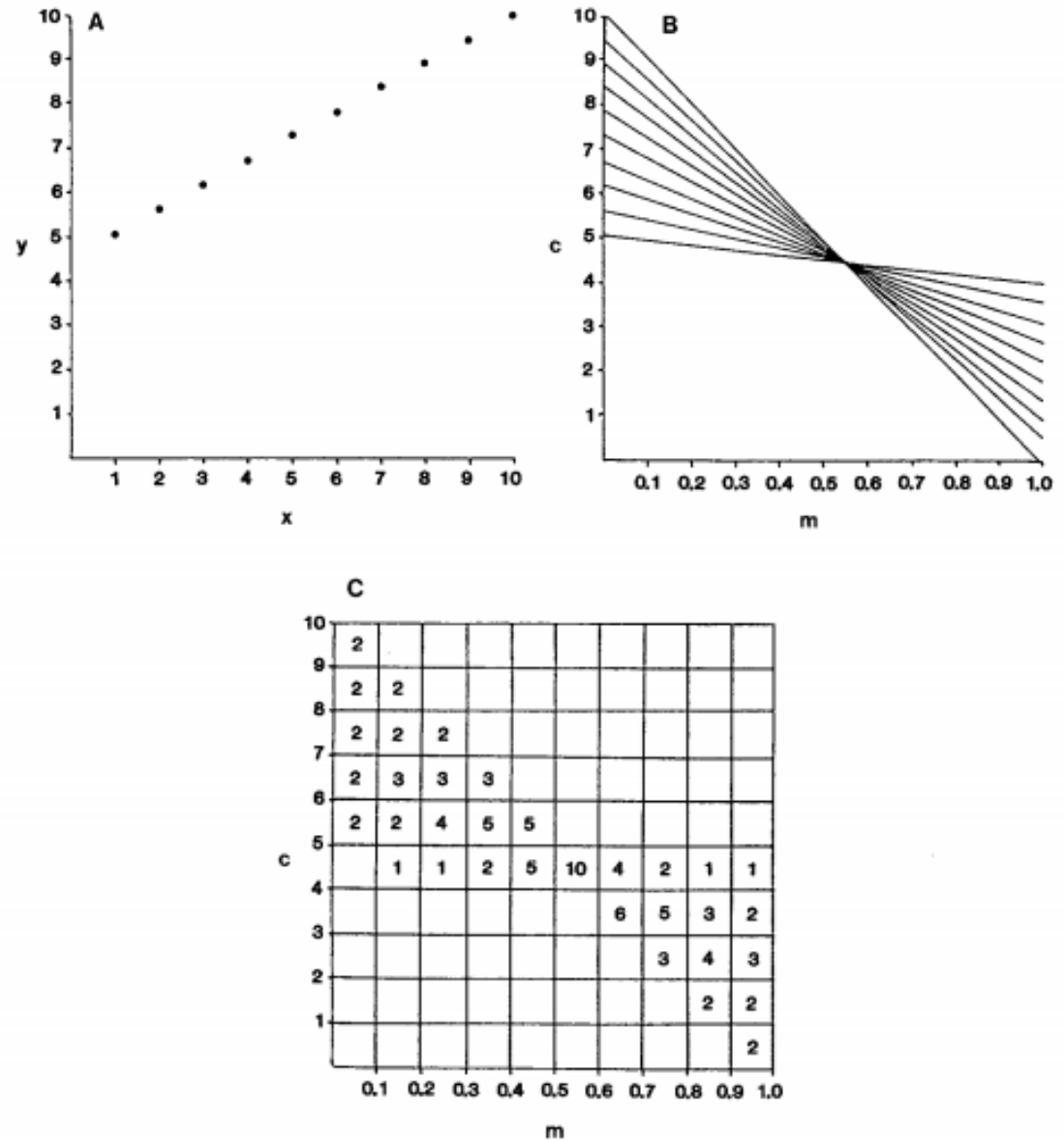
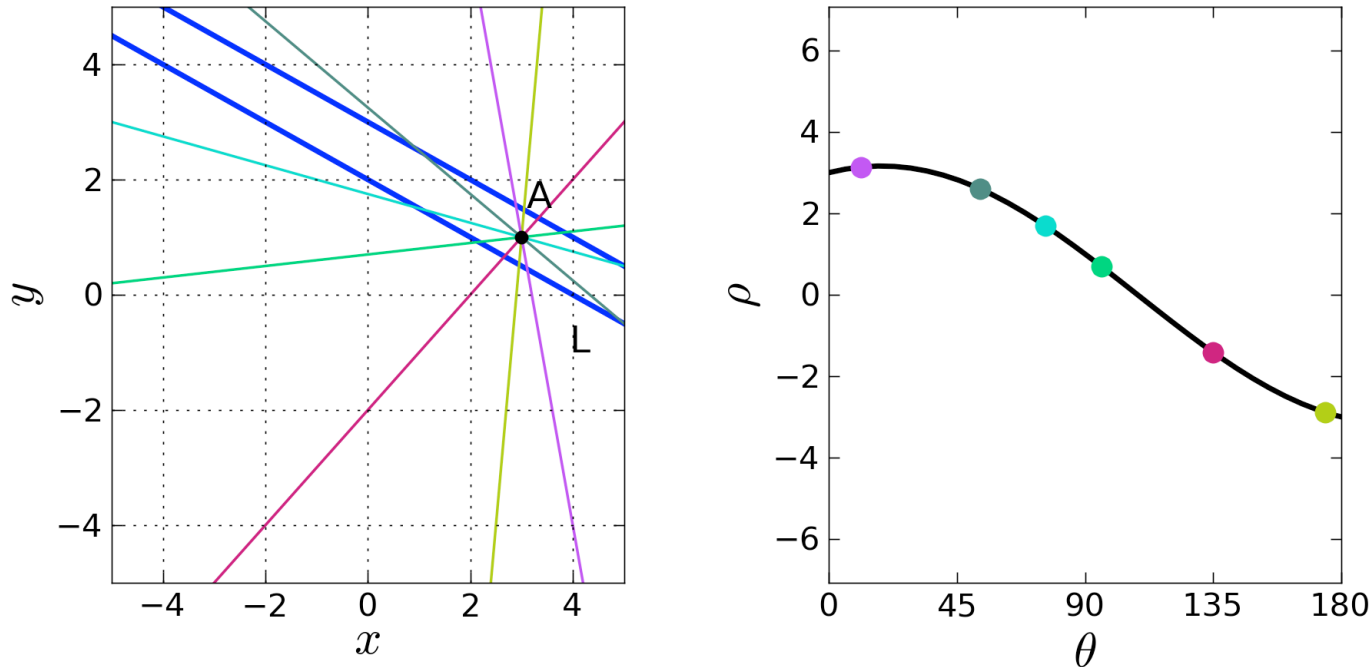


FIG. 1. The basis of the Hough transform for line detection: (A) (x, y) point image space; (B) (m, c) parameter space; (C) accumulator space corresponding to (B).

Hough: Accumulator Diagram (2)

- The following is quoted (12 iv 14) from:
<http://www.ebsd-image.org/documentation/reference/ops/hough/op/houghtransform.html>



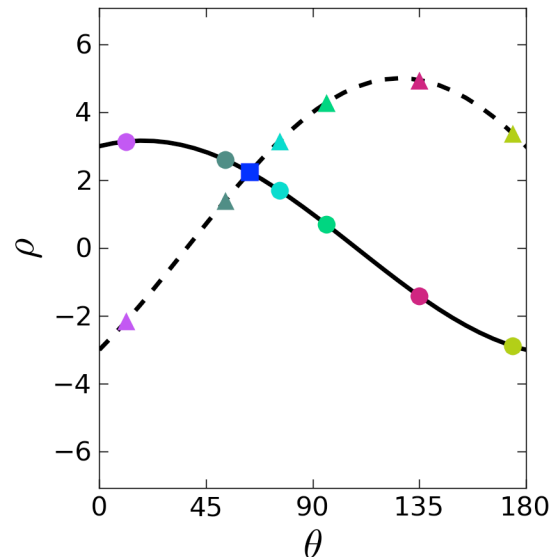
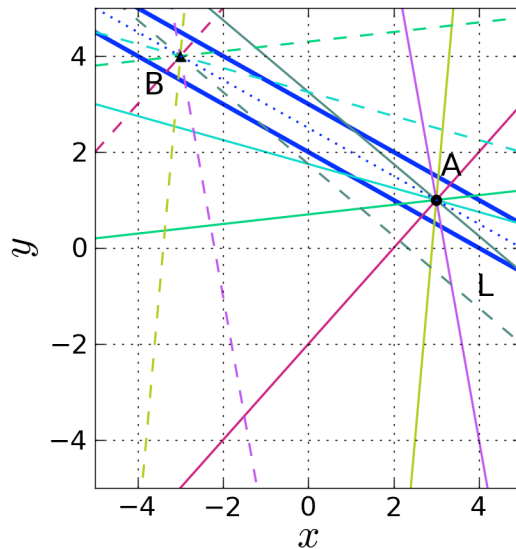
“Effectively, this transformation converts each pixel of the image space into a sinusoidal curve in the Hough space. The calculated ρ value is rounded to the closest pixel ρ_j . The intensity of the pixels (θ_j, ρ_j) that are part of the sinusoidal curve are augmented by the intensity of the corresponding 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:

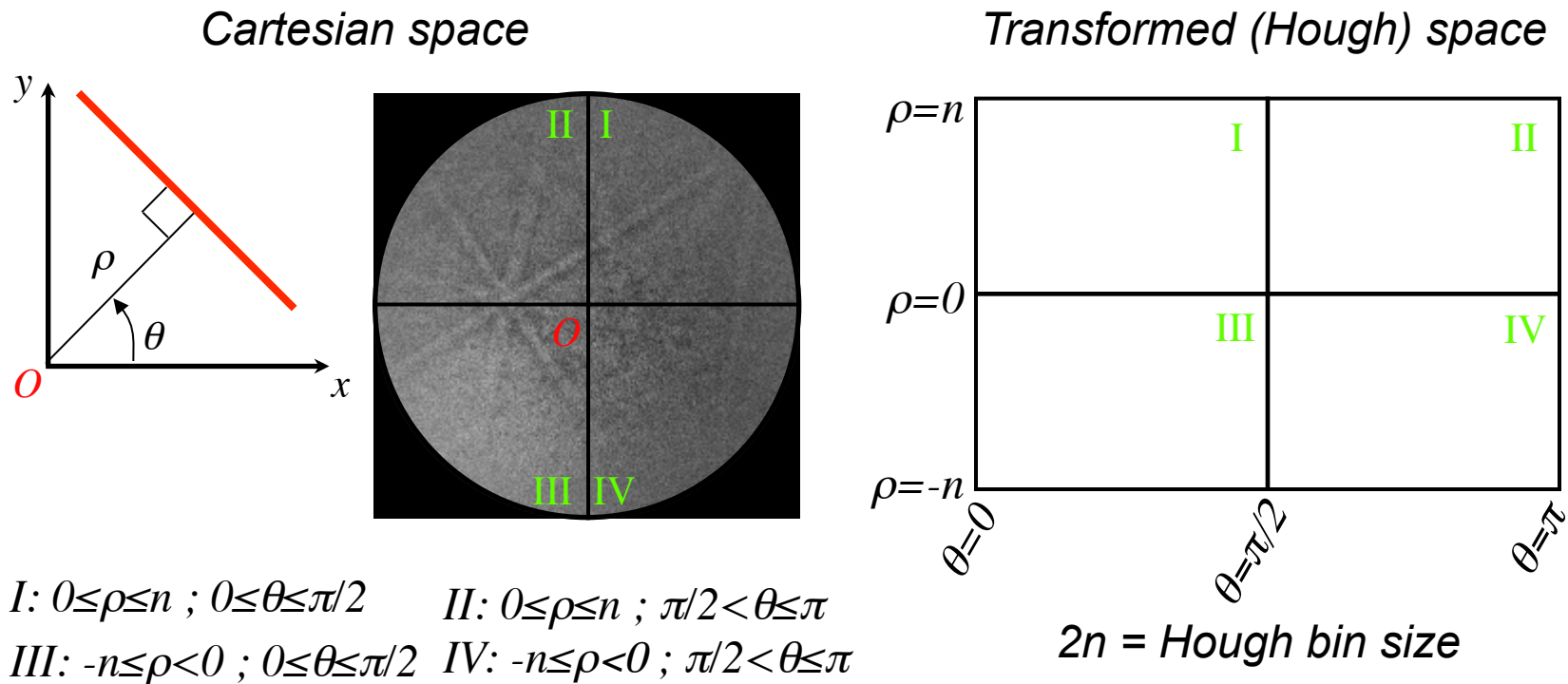
- Krieger Lassen, N. C. (1994). Automated determination of crystal orientations from electron backscattering patterns. (Unpublished doctoral dissertation). The Technical University of Denmark.
- Tao, X., & Eades, A. (2005). Errors, artifacts, and improvements in ebsd processing and mapping. *Microscopy 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 than the one of the pixels outside of it; and b) the intensity of many sinusoidal curves is added at this intersection.”

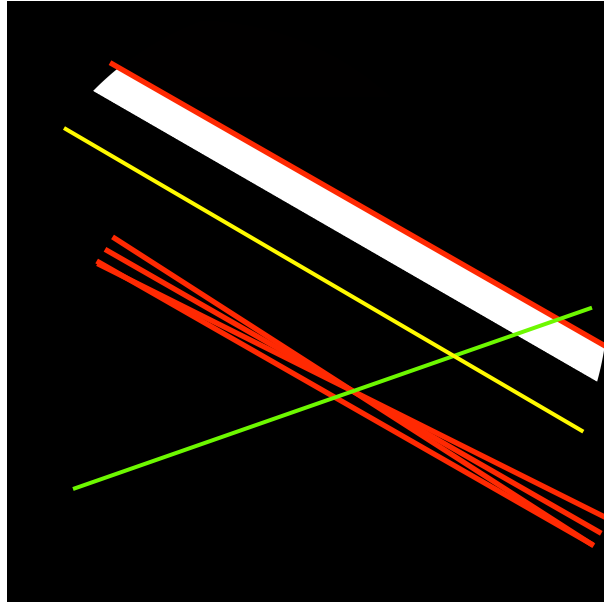
Detecting Patterns-Hough Transform

- 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

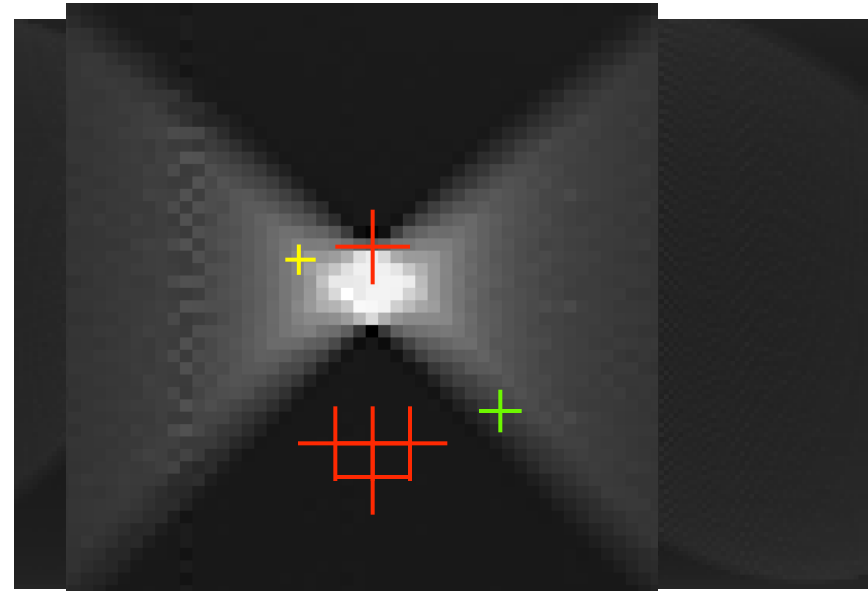


Detecting Patterns-The Hough of one band

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

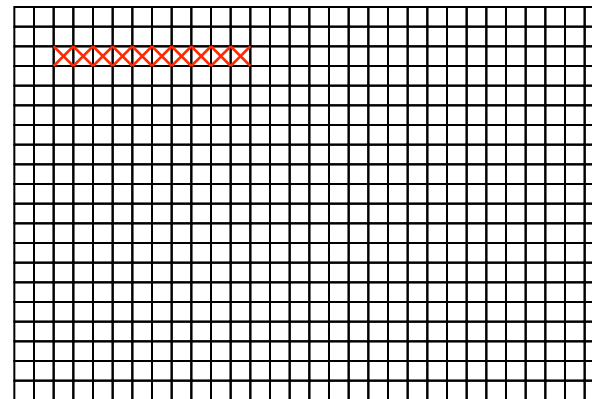
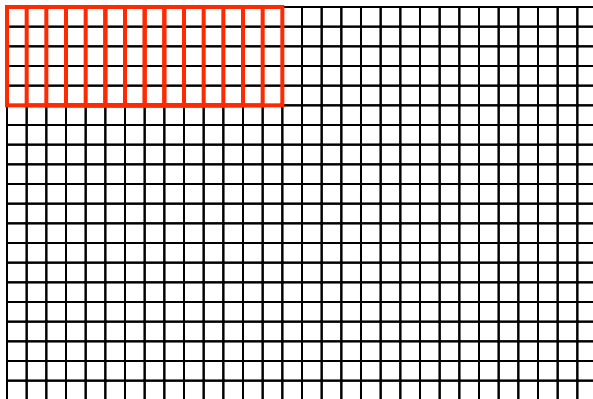
Setting up binning/mask

- 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

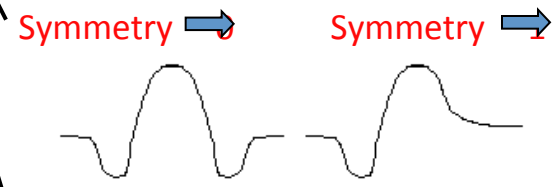


Detecting Patterns-Hough Parameters

The interface shows a grayscale image with several peaks marked by colored crosses. The Hough space plot shows multiple intersecting lines in various colors. The control panels include:

- Image Parameters:** Binned Pattern Size (96), Rho Fraction (100%), Image Filter (0%), and a Hide Bands button.
- Peak Parameters:** Maximum Peak No. (8), Minimum Peak No. (4), Peak Symmetry (0.76), Min Peak Distance (15), Min Peak Magnitude (100), and a Hide Peaks button.
- Hough Parameters:** Convolution Mask (Medium [9x9]), Theta Step Size (1), Hough Time (0.11), and Image Quality (93.9).

<i>More peaks</i>	<i>Less peaks</i>
Use with low symmetry	Use with cubic materials
Increases the number of solutions	Decreases the number of solution



<i>Smaller distance</i>	<i>Larger distance</i>
Closely spaced bands	Sparsely distributed bands

<i>Smaller mag.</i>	<i>Larger mag.</i>
Band intensity is low	Band intensity is high

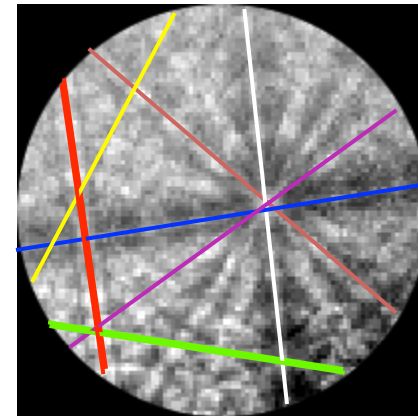
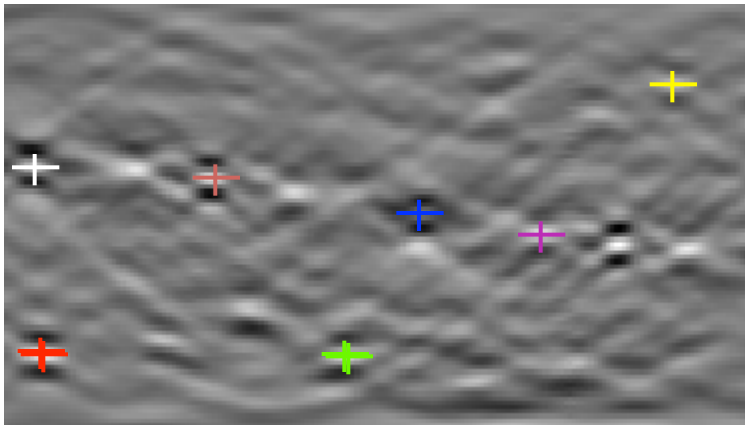
Binned Pattern Size=Hough resolution in ρ

<i>Smaller size</i>	<i>Larger size</i>
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

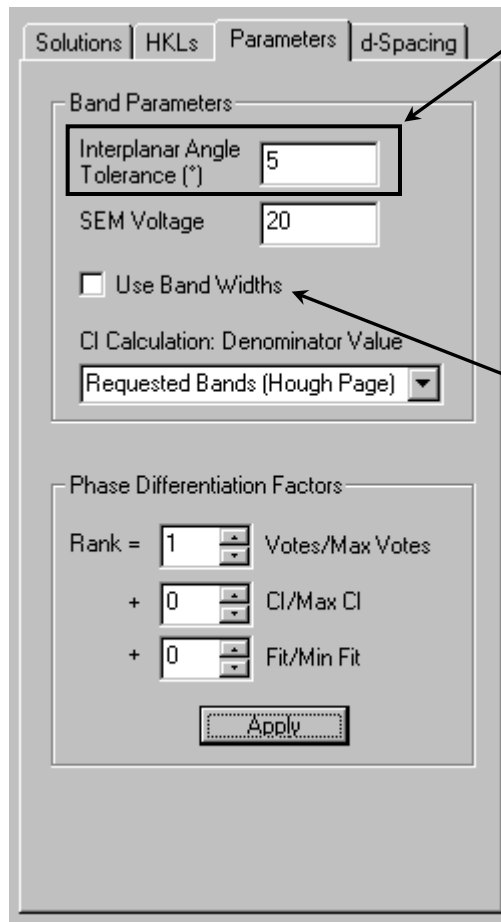
Indexing Patterns-Identifying Bands

- 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



- 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

<i>Lower Tolerance</i>	<i>Larger Tolerance</i>
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

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

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

		Solution #										
		1	2	3	4	5	6	7	8	9	10	11
Band triplets		x	x	x								
				x								
				x								
		x	x	x	x	x						
			x	x	x		x					
			x	x				x				
									x			
					x							
					x							
										x		
				x						x	x	
										x	x	
Σ		2	4	10	2	1	1	1	1	1	1	1

 S2 (solution w/ 2nd most votes)
  S1 (solution w/ 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