

# Texture and Anisotropy

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## **Part III:**

### **Chapter 7. Scanning electron microscopy-based techniques**

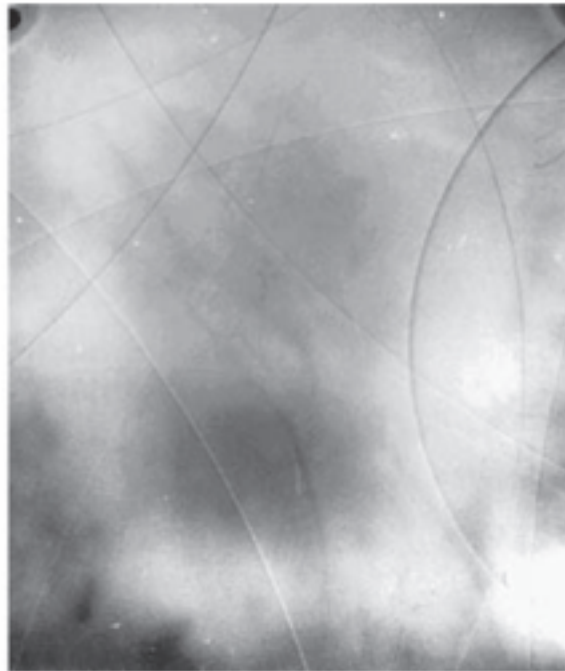
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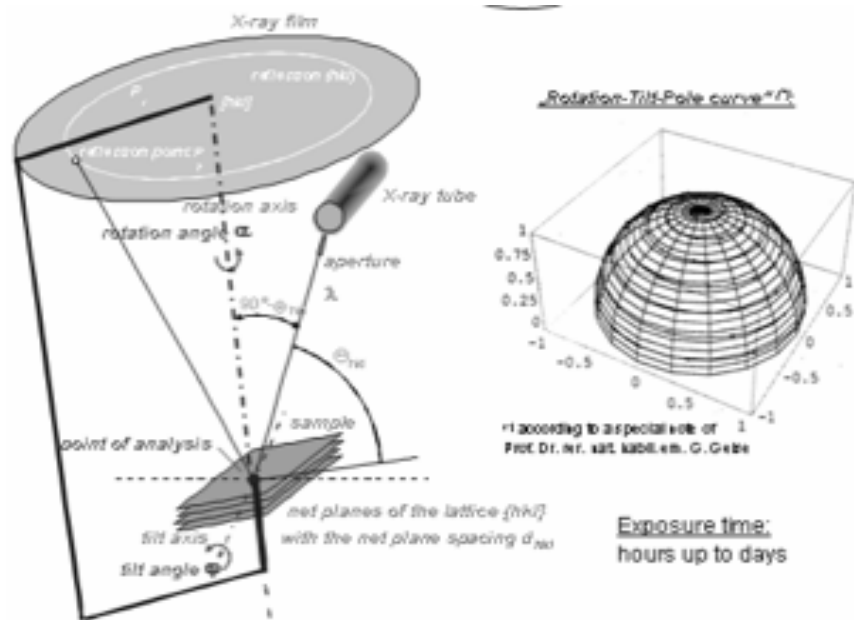
# Micro-Kossel technique

The Kossel technique is based on the diffraction of x-rays at crystal lattice.

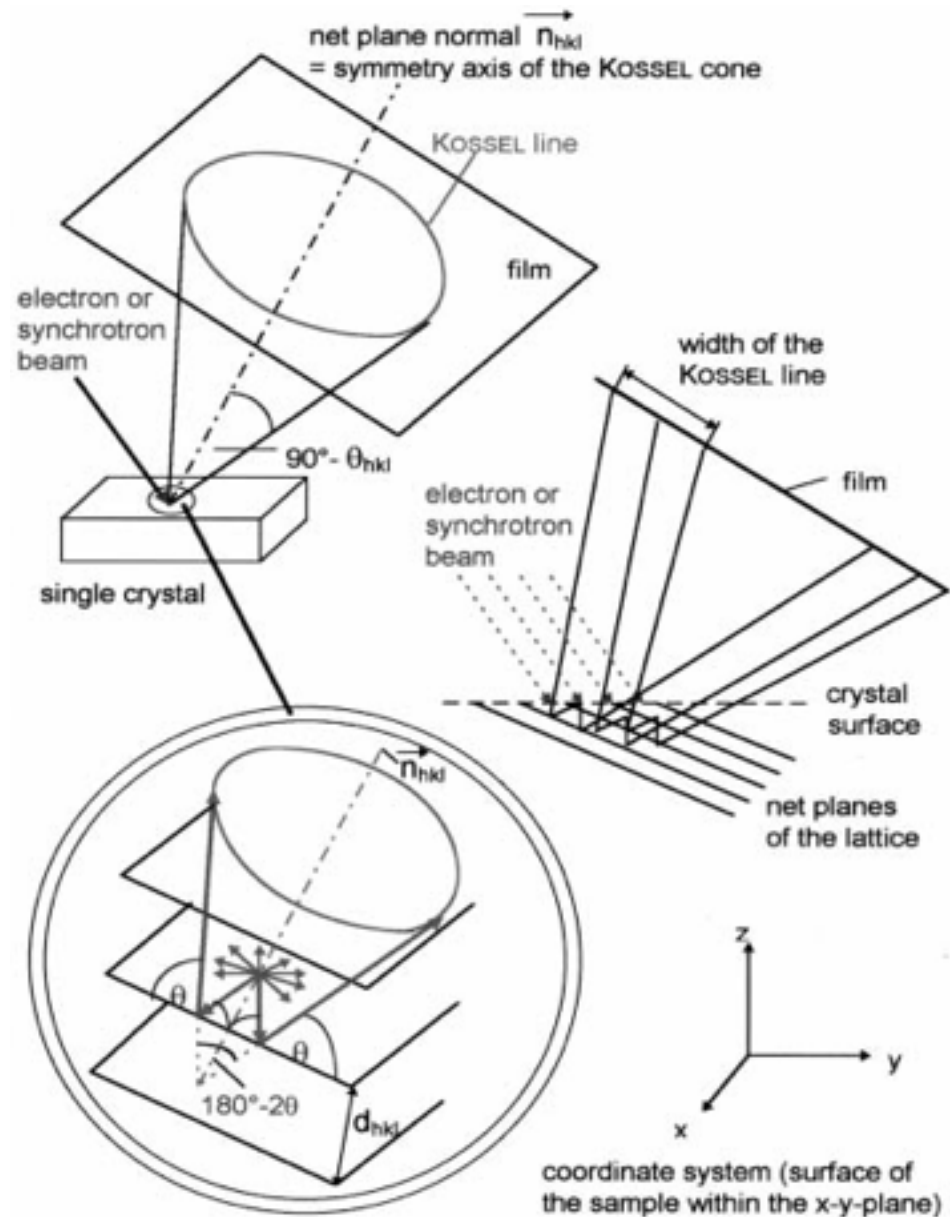
The Kossel cones, the reflected x-rays form cone-shaped planes, because the lattice planes are irradiated with x-rays with all directions. The wavelength is much larger than Kikuchi electron diffraction.



# Principle of Kossel tech.



Exposure time:  
hours up to days



BRAGG's law:

$$\lambda = 2 \cdot d_{hkl} \cdot \sin \theta_{hkl}$$

( $\lambda$  ... wavelength;  $d_{hkl}$  ... net plane spacing;  $\theta_{hkl}$  ... diffraction angle)

# Kossel patterns

The wavelength is much larger than Kikuchi electron diffraction. The resulting projection lines on the film plane are strongly curved.

In order to obtain Kossel patterns, the accelerating voltage of the electron must be about 2-3 times larger than the critical voltage to produce the characteristic x-ray  $K\alpha$  radiation.

The spatial resolution is limited to 5-10  $\mu\text{m}$ .

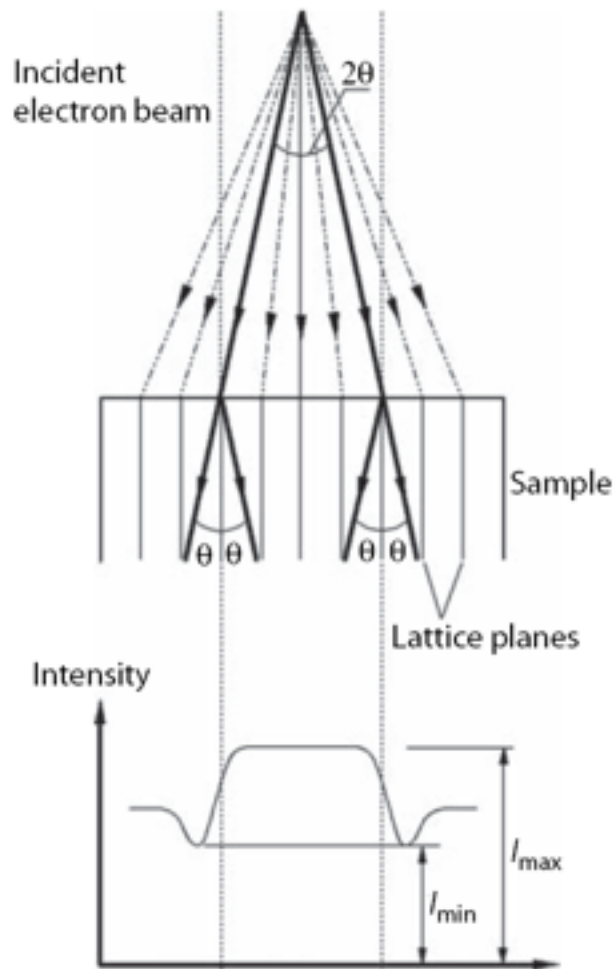
A low sign-to-noise ratio caused by the nonreflected x-rays as well as the continuous radiation

High-precision internal stress measurement ( $\Delta a/a = 10^{-5}$ )

# Electron channeling diffraction

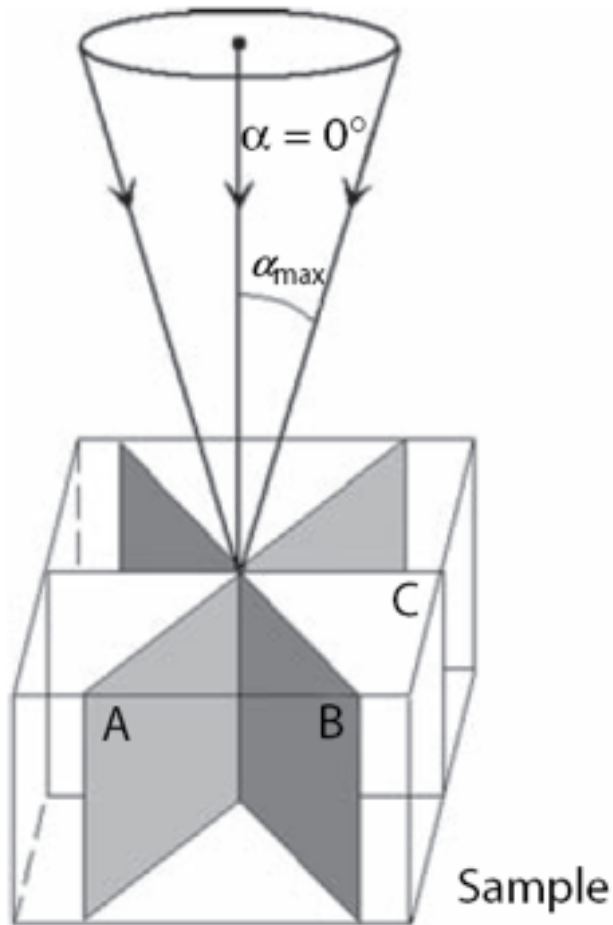
It is based on the diffraction of the electron beam in an SEM at the crystal lattice. The intensity of the reflected electrons depends not only on the site but also on the angular direction of the incident beam.

By changing the direction of the incident beam, electron channeling patterns (ECP) is formed due to the anisotropy of backscattered electrons.

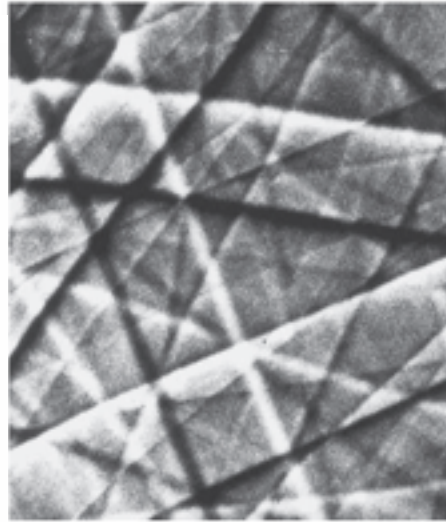


(a) SAC pattern of a recrystallized grain in partially recrystallized aluminum. (Courtesy of H.W. Erbslöh.) (b) Formation of pseudo-Kikuchi bands in SAC.

# Formation of ECP patterns

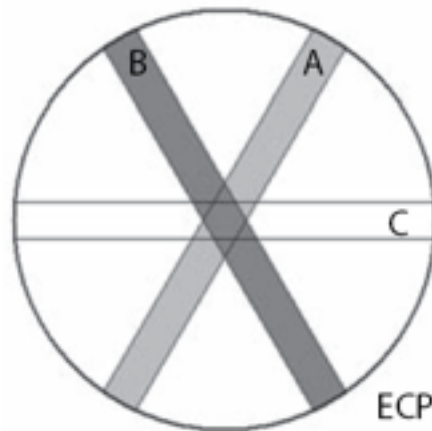


(a)



In order to achieve optimum spatial resolution, probe diameter is reduced to few microns (selected area electron channeling diffraction) ECP

ECP bands is called “pseudo-Kikuchi bands”

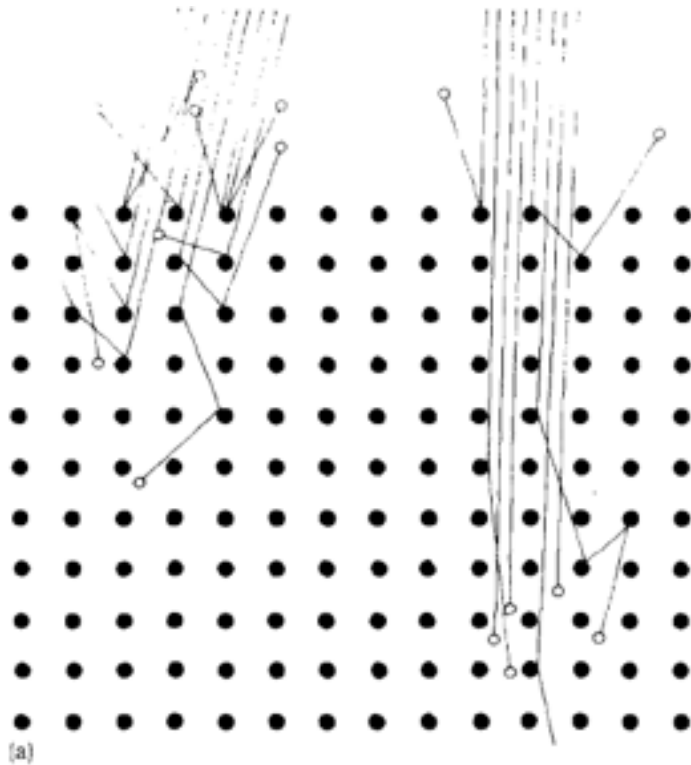


(b)

# Principle of ECP patterns

Bloch wave I

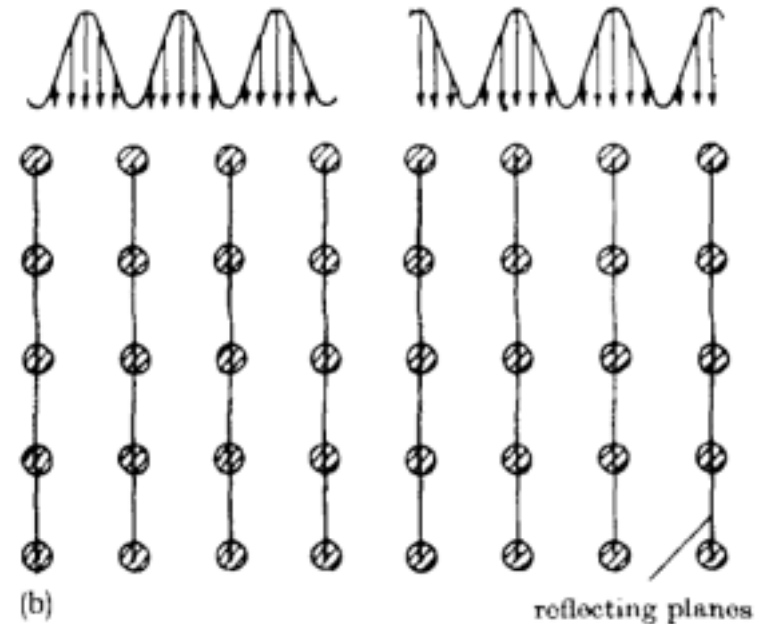
Bloch wave II



Bloch wave I: strongly interaction with the column of atoms, and will be absorbed.

Bloch wave II

Bloch wave I



Bloch wave II: be channeled through the specimen

# Principle of ECP patterns: conti.

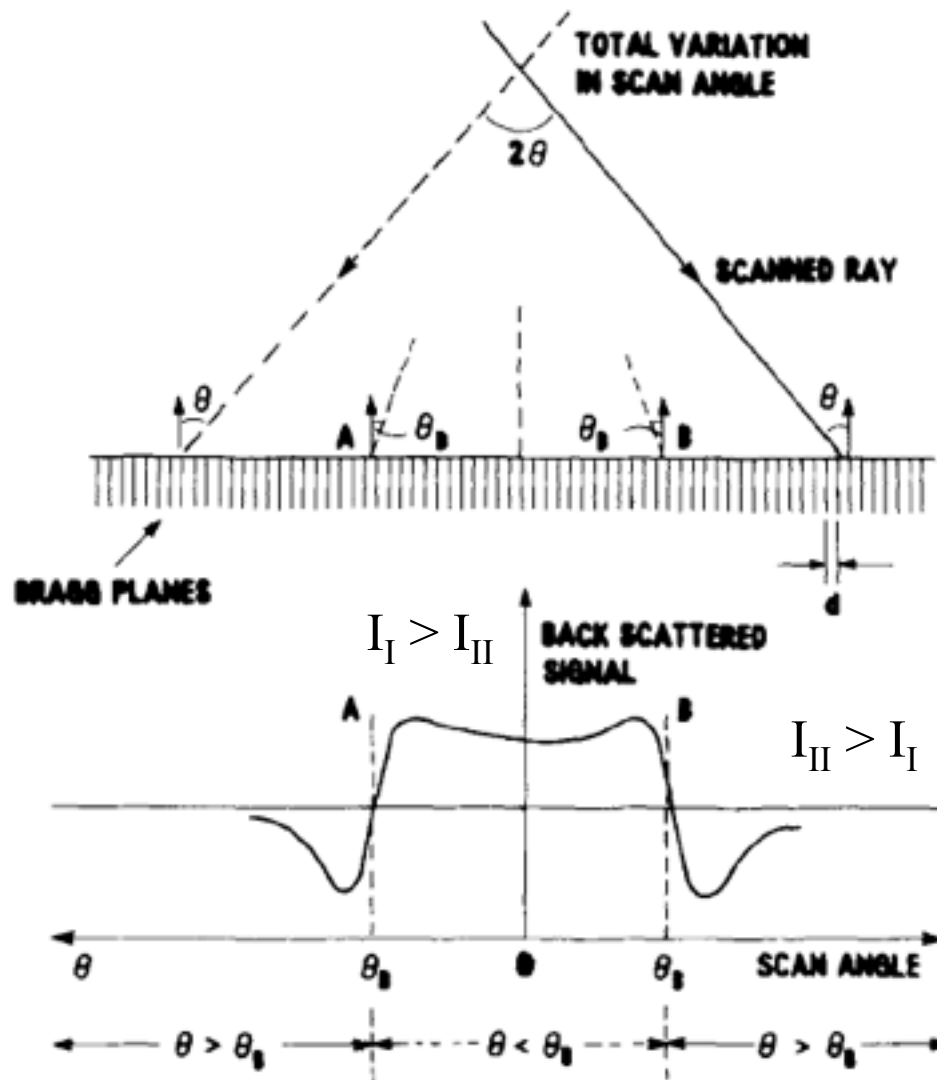


FIG. 2. Changes in the geometry of an incident beam during scanning. During any one line scan the incidence angle  $\theta$  will vary from being greater than  $\theta_B$  to being smaller than  $\theta_B$ . At two symmetric positions **A** and **B**,  $\theta = \theta_B$ . This variation gives the changes in backscattering signal intensity shown.



# Special Topics on EBSD

# EBSD Developments: I

1930-1950s:

First observations of  
high-angle Kikuchi pattern

1970s:

Electron back-scattering  
pattern recorded in the  
SEM

Early 1980s:

Computer routines for  
interactive EBSD pattern  
evaluation

# EBSD Developments: II

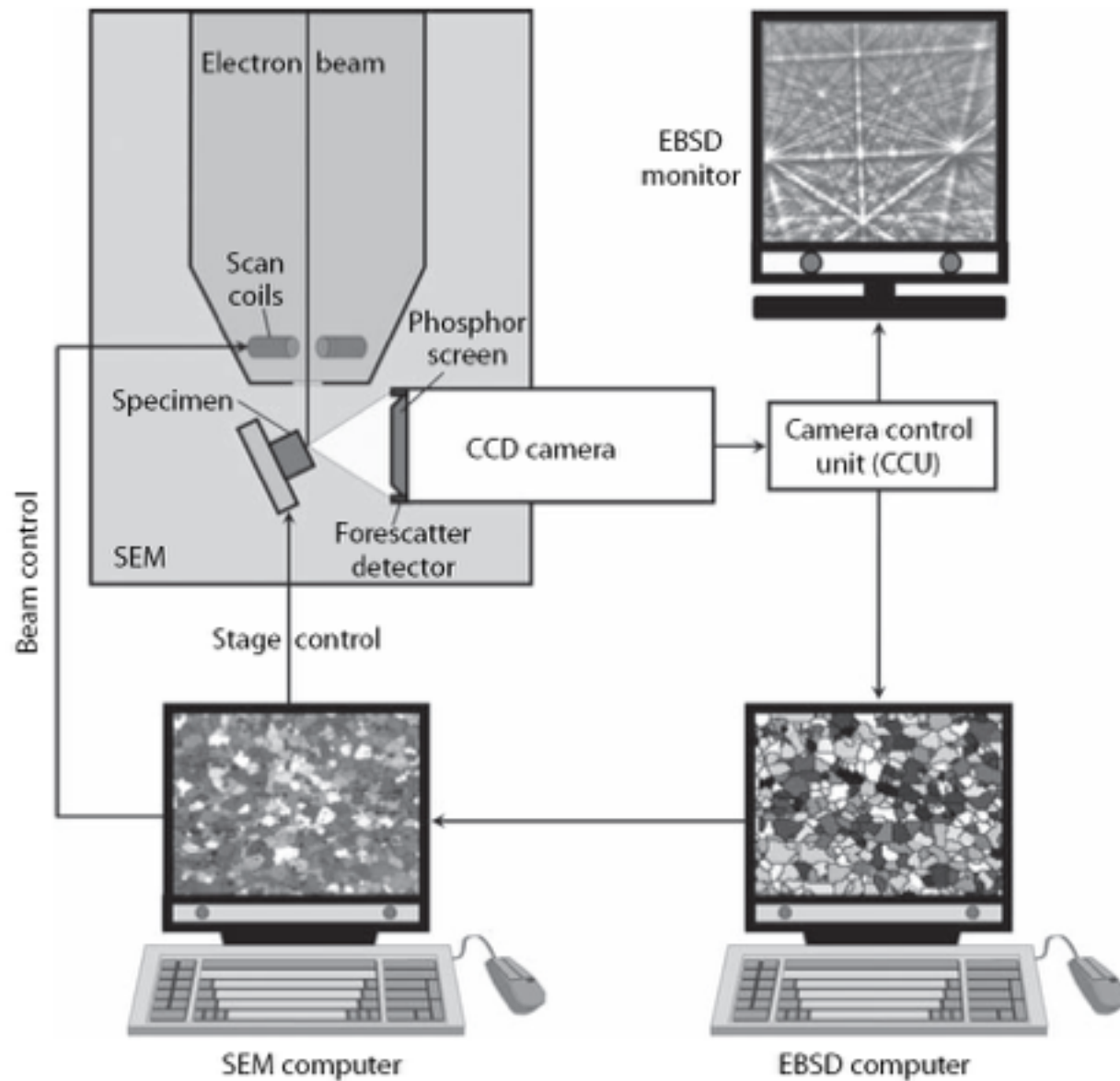
Later 1980s:

Automated EBSD pattern  
analysis

1990s:

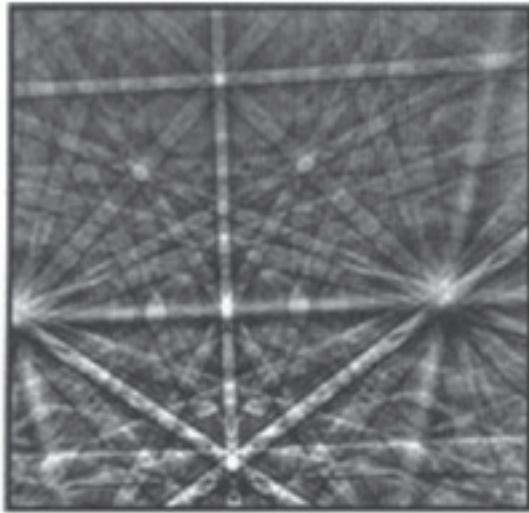
Orientation mapping

# EBSD system

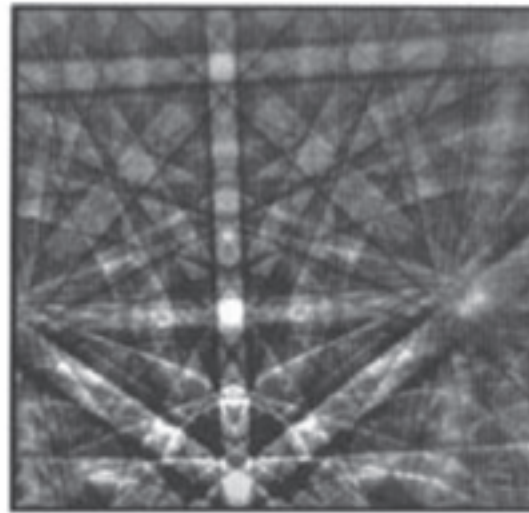


# EBSD sample preparation

Si

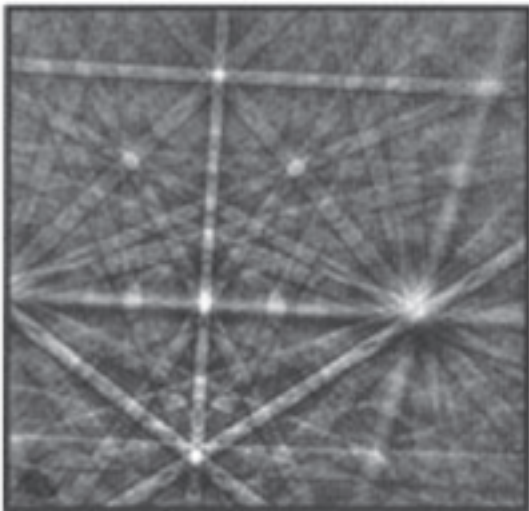


(a)



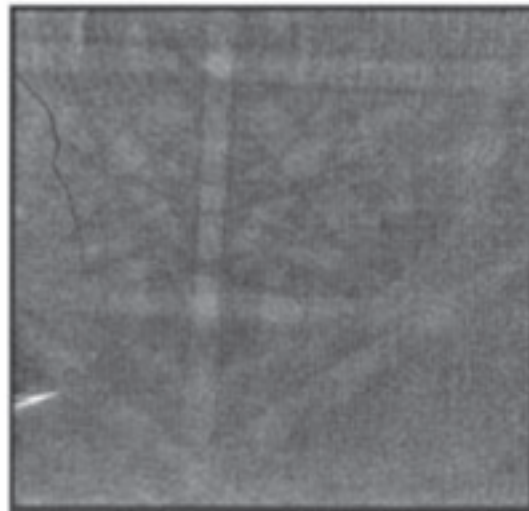
(b)

No coating



(c)

40 kV



(d)

10 kV

5 nm Nickel  
coating

Beam voltage

# EBSD sample preparation techniques

## I. Mechanical Polish

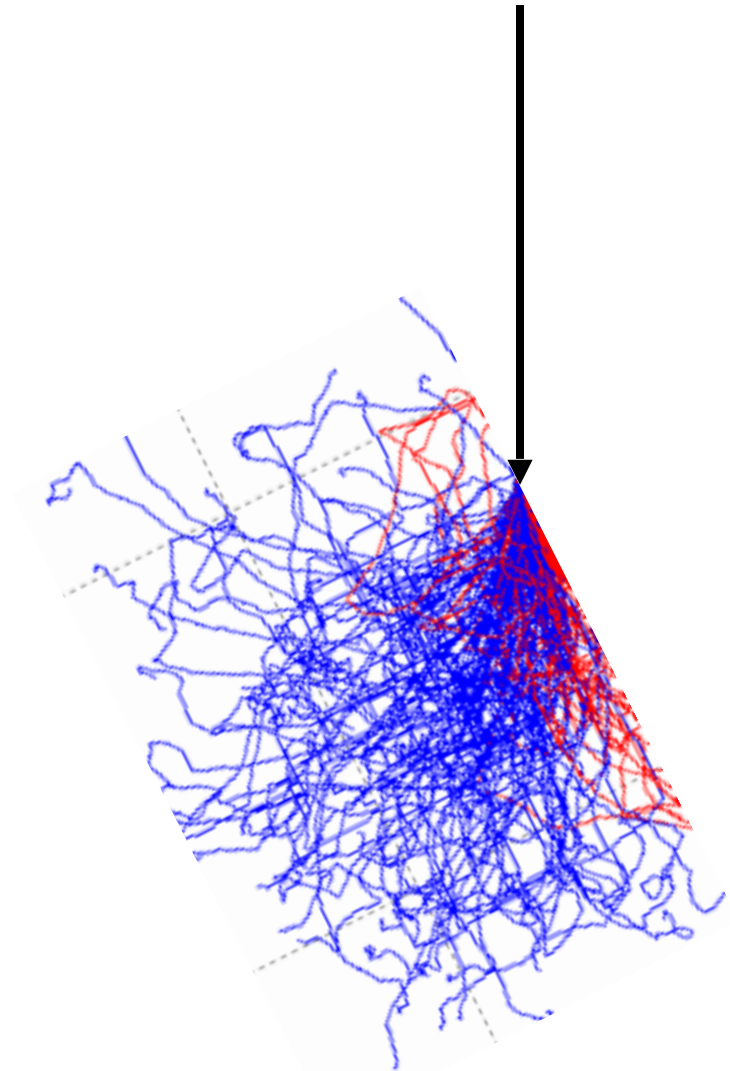
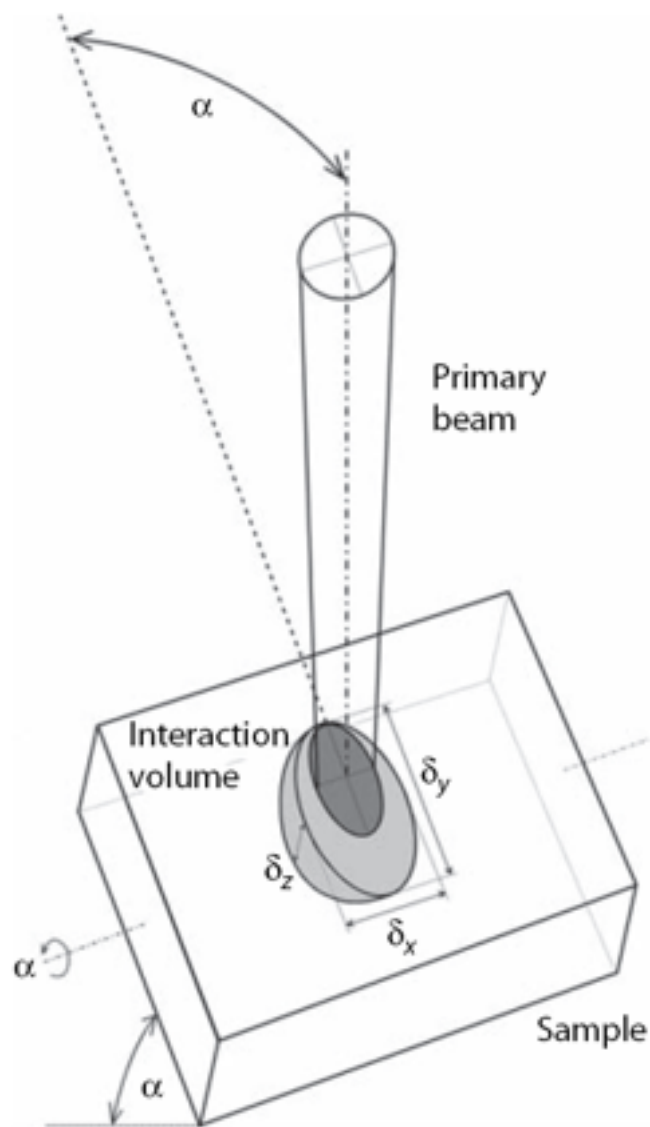
1. Sectioning
2. Mounting
3. Grinding
4. Polishing
5. Colloidal Silica

## II. Electropolish

## III. Chemical Etch

## IV. Ion Etch

# Specimen-beam interaction for EBSD



# Specimen Tilt for EBSD

- The main effect of tilting the specimen is to reduce the path length of electrons, which have been backscattered by lattice planes.
- Therefore, when compared to flat specimen, more of these electrons undergo diffraction, and escape from the specimen before being absorbed.

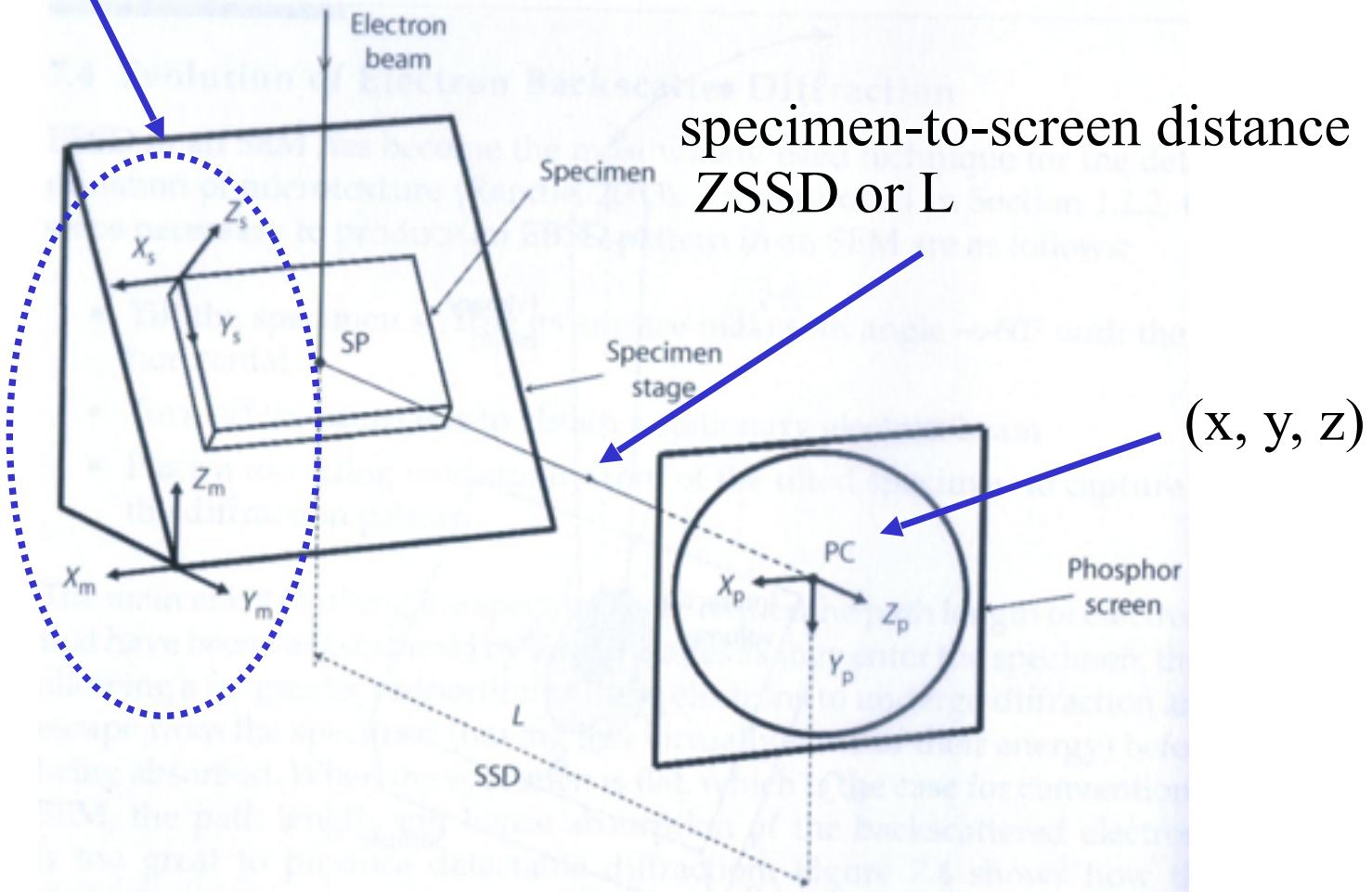


# Parameters for EBSD

- The coordinates of the pattern center, PC
- The specimen-to-screen distance  $Z_{SSD}$  or L
- The relationship between reference directions in the microscope ( $X_m$ ,  $Y_m$ ,  $Z_m$ ), and screen/pattern ( $x$ ,  $y$ ,  $z$ )

# Schematic of EBSD parameters

$(X_m, Y_m, Z_m)$



# Introduction to EBSD patterns

- The EBSD pattern results from diffraction of a divergent source of electrons generated within the sample just beneath the point where the primary electron strikes the specimen.
- The electrons that contribute to the pattern are only those:
  - That have lost no more than a few electron volts of energy.
  - Emerge from a depth in the specimen of no more than 30 to 40 nanometers, which is a considerably smaller depth than that reached by the primary beam.

# Introduction to EBSD patterns

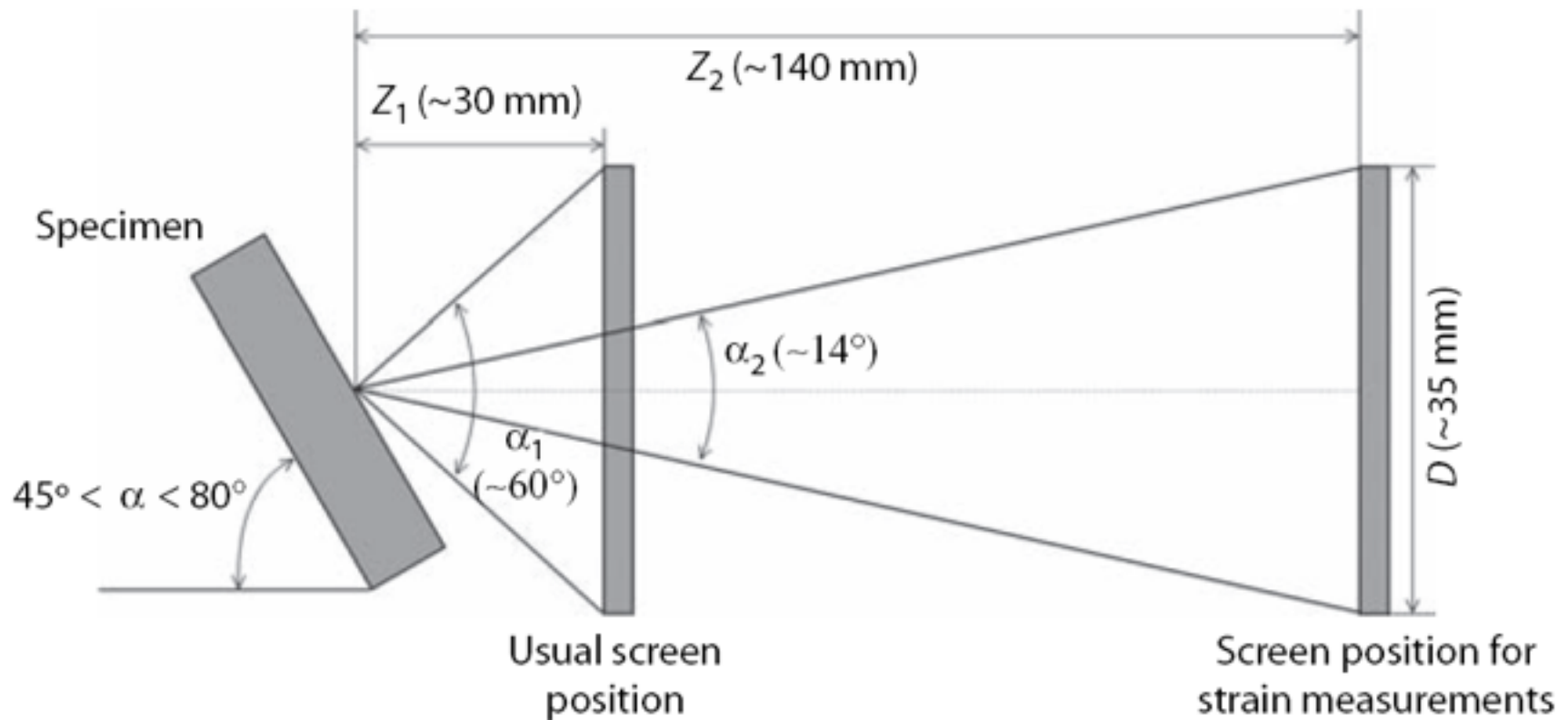
- The lower the primary beam voltage, the better the resolution, because the beam spread is smaller.
- This benefit may be offset, because at lower primary voltages, the probe diameter for a given current is larger.
- The large beam current can be countered by using:
  - Field emission source, or
  - Lower beam currents. However, lower beam current leads to a poorer signal to noise ratio.

# Introduction to EBSD patterns

- The diffraction pattern can be captured on a recording medium or device placed in front of the tilted specimen.
- Examples of detecting or recording medium are:
  - a phosphor screen, viewed by a low light television camera. The luminosity of a phosphor screen for a beam current of 100 pA at 20 kV is of the order of  $10^{-4}$  lux (10 times greater than that detectable in live TV by a SIT camera).
  - CCD camera, which produces about 50 times more luminosity than a phosphor screen.

# EBSD hardware: camera

A slow-scan charge-coupled device (SSCCD) video camera with solid-state sensors is now used for EBSD, replacing SIT camera.



Effect of retracting the phosphor screen in an EBSD system to give a smaller convergence angle  $\alpha$  and hence better resolution in the diffraction pattern. (Courtesy of A. Wilkinson.)

# Data collection efficiency

The total time required per map pixel is the sum of the time moving to the map coordinates and to site the electron probe, collecting a diffraction pattern, analyzing the pattern.

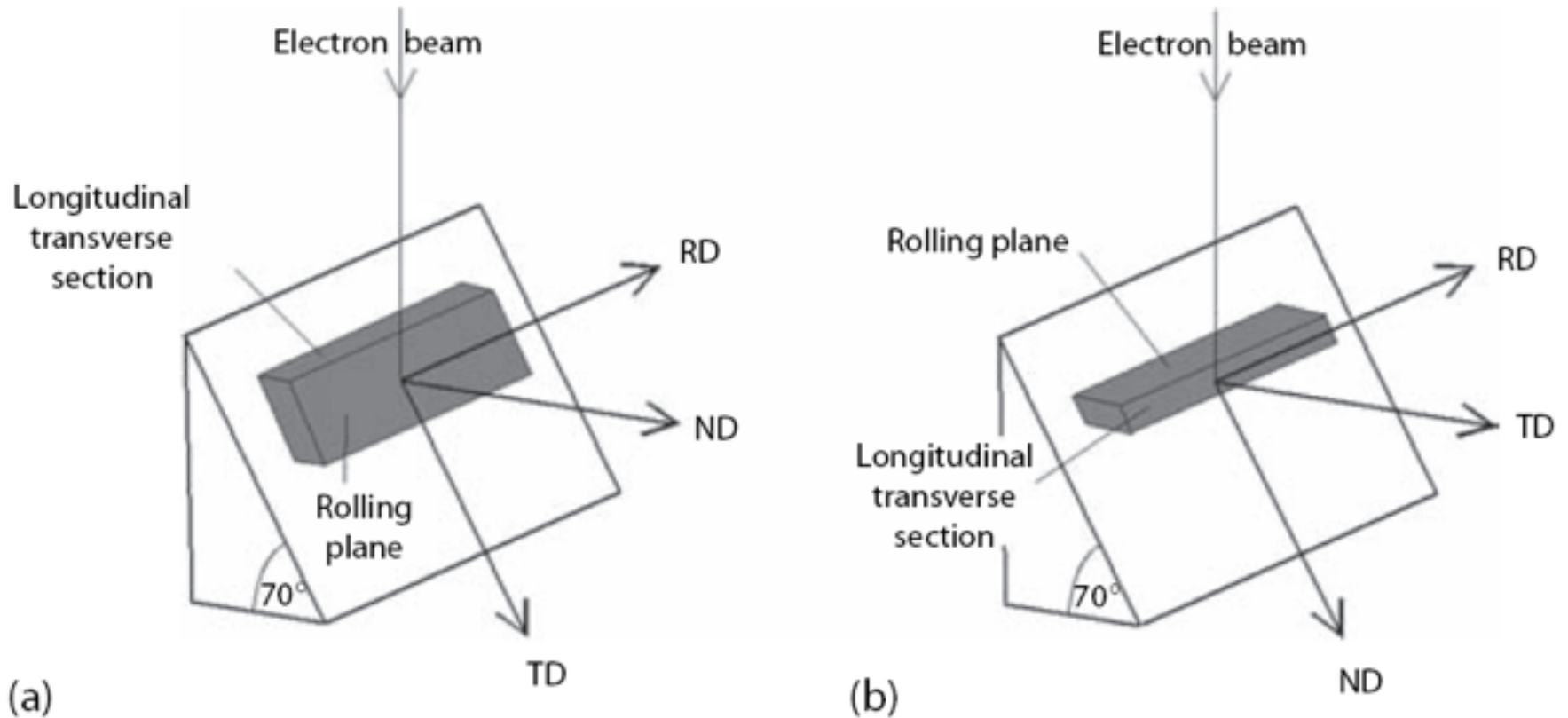
Data collection speed depends on the improvement in camera technology (dynamic range, 10 bits of gray scale), in group of pixels in the diffraction pattern, in electron processing (frame average and amplification), in computer and software improvements.

# Experiment design philosophy

1. Selection of a suitable candidate material and specimens for analysis
2. Optimum specimen preparation
3. Manual checking of diffraction patterns for clarity and expected phase match
4. Application of microstructure for grain size and phase distribution
5. Test run of data collection
6. Data collection, via automated orientation map



# Experiment design philosophy



Examples of choices for mounting an EBSD specimen to sample either (a) the rolling plane or (b) longitudinal-transverse section.

# Phase identification

The principle of phase identification by EBSD is:  
the symmetry of the diffraction patterns of the specimen,  
chemical composition analysis of the phase by energy-dispersive  
spectroscopy.

# Influences on EBSD spatial resolution

Microscopy parameters

Material

Specimen/microscopy geometry

Accelerating voltage

Probe current

Pattern clarity

# Resolution: Microscopy parameter

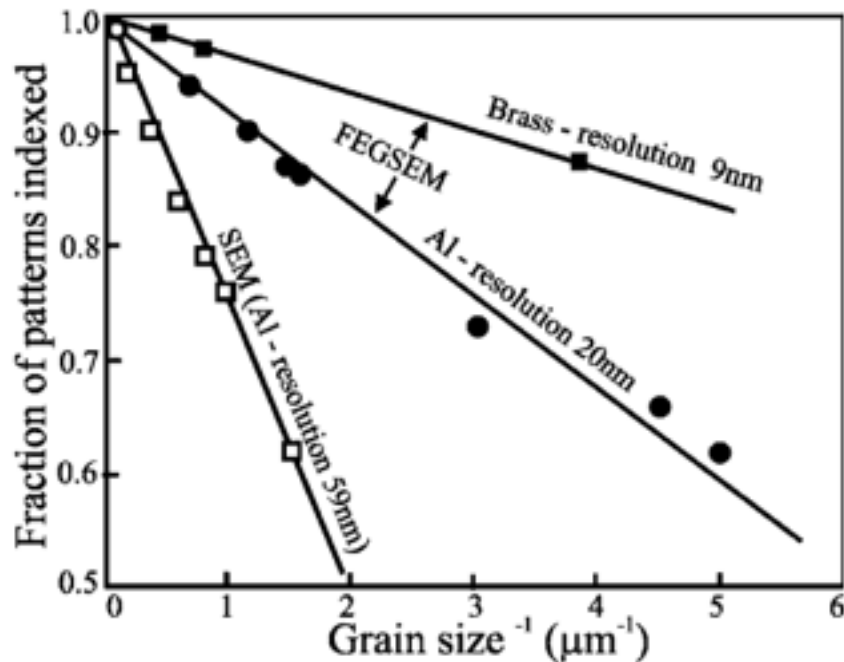


Fig. 1. The effect of grain size on the fraction of diffraction patterns indexed in polycrystalline aluminium alloys and  $\alpha$ -brass at 20 keV in a FEGSEM. The results are compared with those obtained in a W-filament SEM.

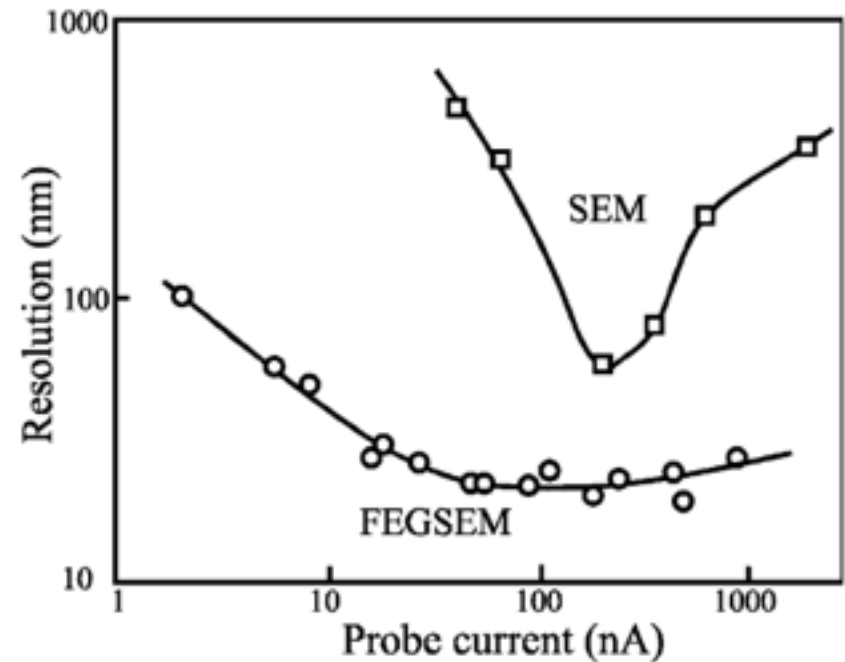
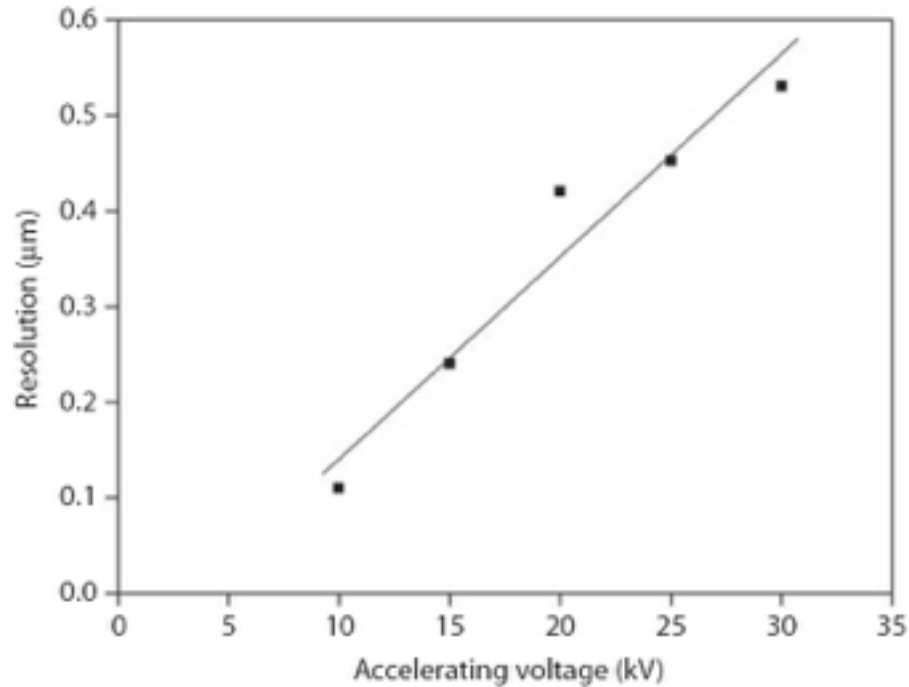


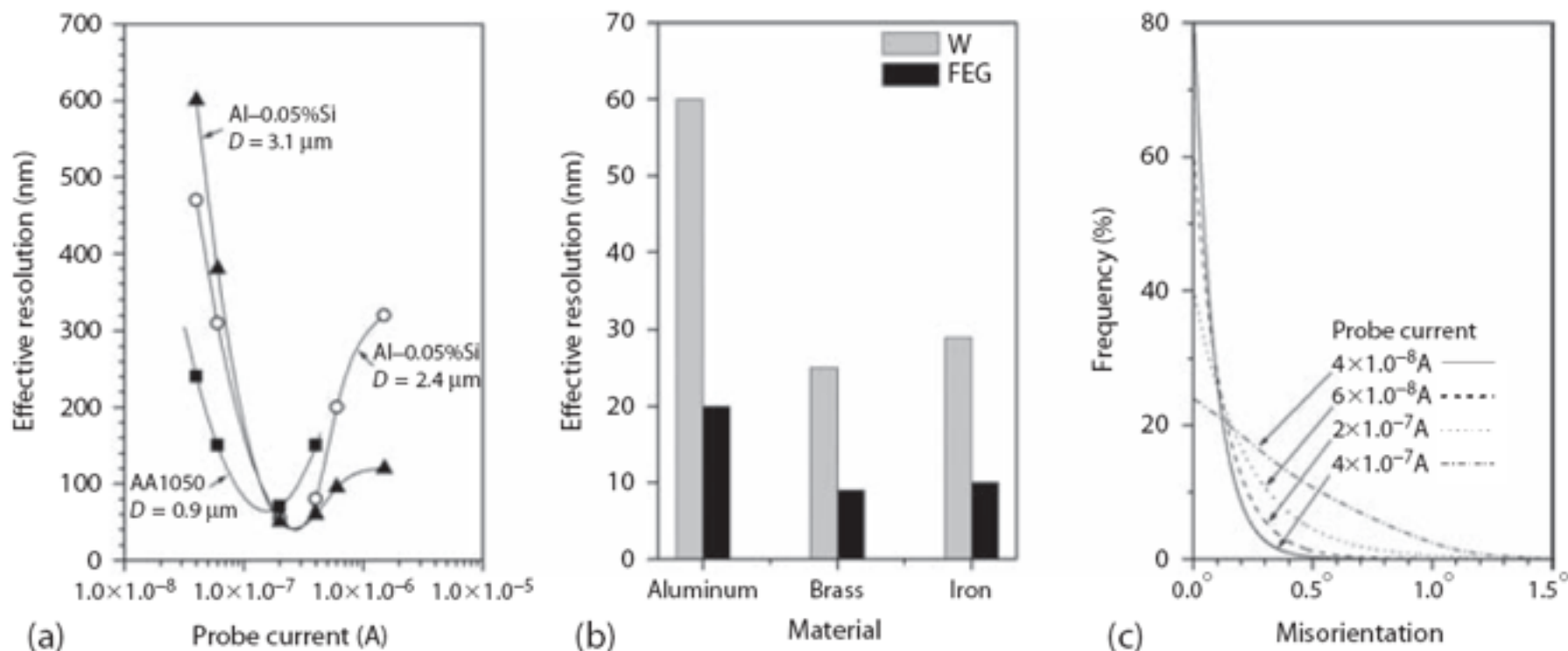
Fig. 2. The effect of probe current on the effective spatial resolution in aluminium at 20 keV. Data for the FEGSEM are compared with previous results (Humphreys *et al.*, 1999) from a W-filament SEM.

# Resolution: accelerating voltage



Spatial resolution of EBSD in nickel as a function of accelerating voltage. (Adapted from Drake, A. and Vale, S.H., *Inst. Phys. Conf. Ser.* 147, 137, Inst. Phys. Pub. Inc., Bristol, 1995.)

# Resolution: probe current



(a) The effect of probe current on effective resolution for several aluminum specimens. The minima in the plots are caused by the reduced pattern-solving accuracy at low probe currents. (From Humphreys, F. J. et al., *J. Microsc.*, 195, 212, 1999.) (b) Effective EBSD spatial resolution for various metals in tungsten filament and FEG SEMs. (From Humphreys, F.J., *Scripta Mater.*, 51, 771, 2004.) (c) Misorientation measurements between adjacent points on a single-crystal silicon specimen for four different probe currents (in amperes). The highest-probe current provides the most accurate result. (Courtesy of F. J. Humphreys.)

# Resolution: pattern clarity

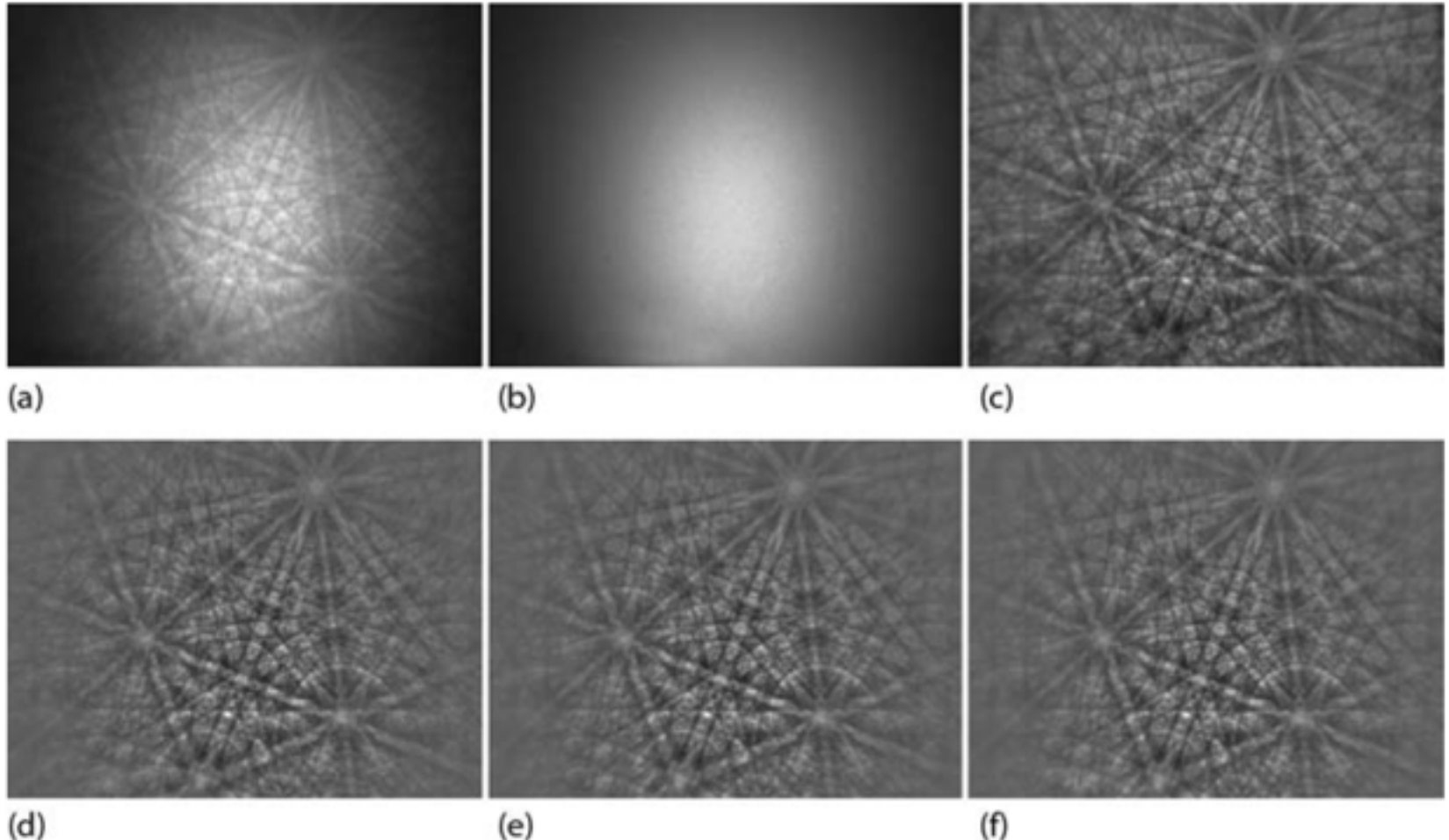
Pattern clarity is influenced by the quality of pattern arising from the sampled volume of specimen and by “noise” introduced during pattern capture and processing.

This is due to that the pattern is averaged over too few frames or too few points, when the backscattered electron signal is reduced.

The backscattered electron signal is proportional to the atomic number.

Increasing in probe currents results in high backscattered electron signal.

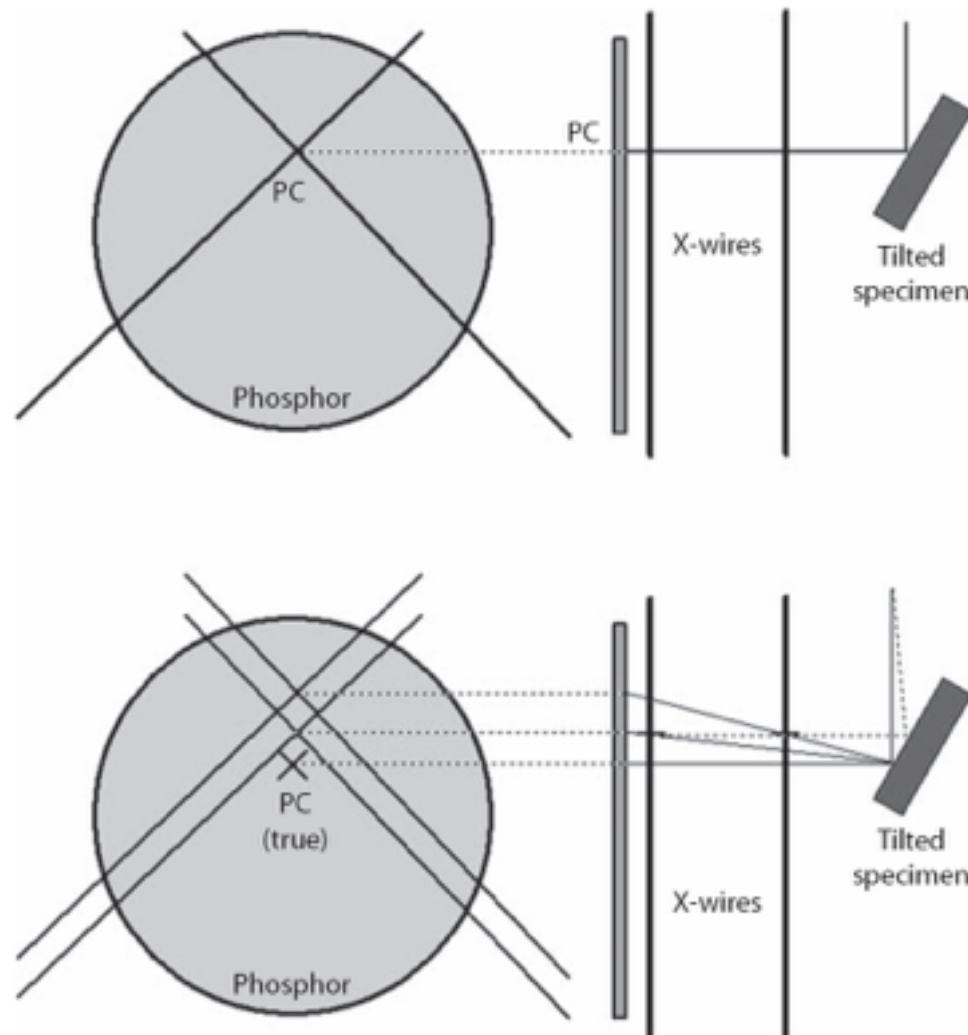
# Resolution: diffraction pattern enhancement



Example of EBSD image processing. (a) A raw EBSD pattern from tungsten carbide. (b) The EBSD pattern background signal. (c) The corrected EBSD pattern produced by dividing the raw pattern by the background and then adjusting the brightness and contrast of the resulting image to fit the available gray-level range. (d) Pattern after single frame averaging. (e) Pattern after frame averaging over three frames. (f) Pattern after frame averaging over 20 frames. (Courtesy of K. Mingard and A. Day.)



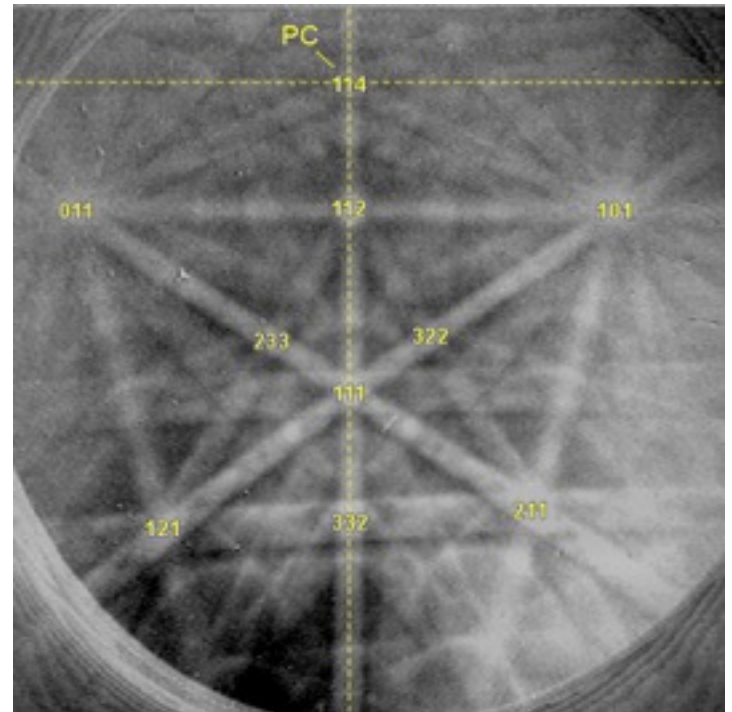
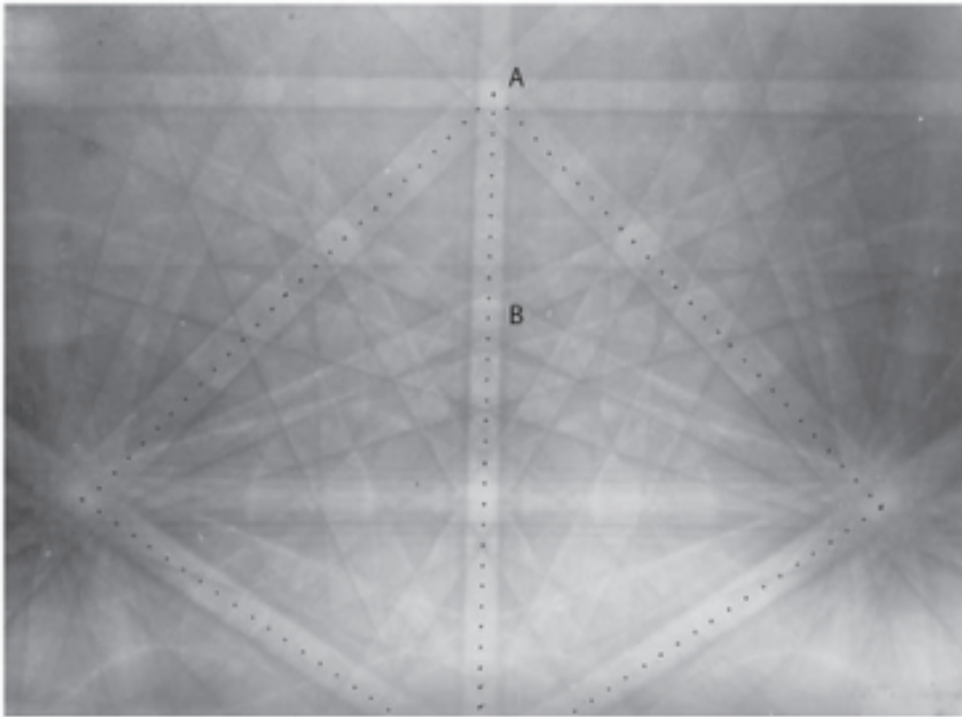
# Calibration procedures: shadow casting



Location of the pattern center, *PC*, on the phosphor screen by alignment of two sets of cross wires located between the specimen and the screen. (Courtesy of A. Day.)

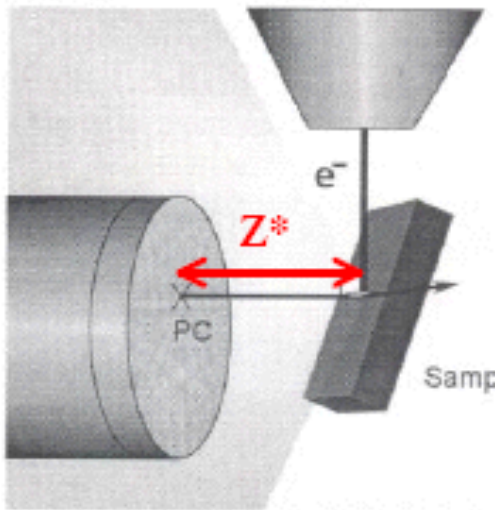
# Calibration procedures: known crystal orientation

(001) Si



# Calibration procedures: known crystal orientation

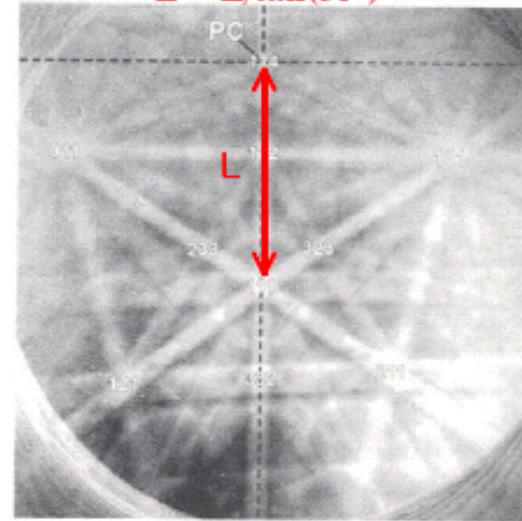
Pattern center(PC)



[114] direction is normal to screen  
Angle bet. [114] and [111] : 35°

specimen to screen distance

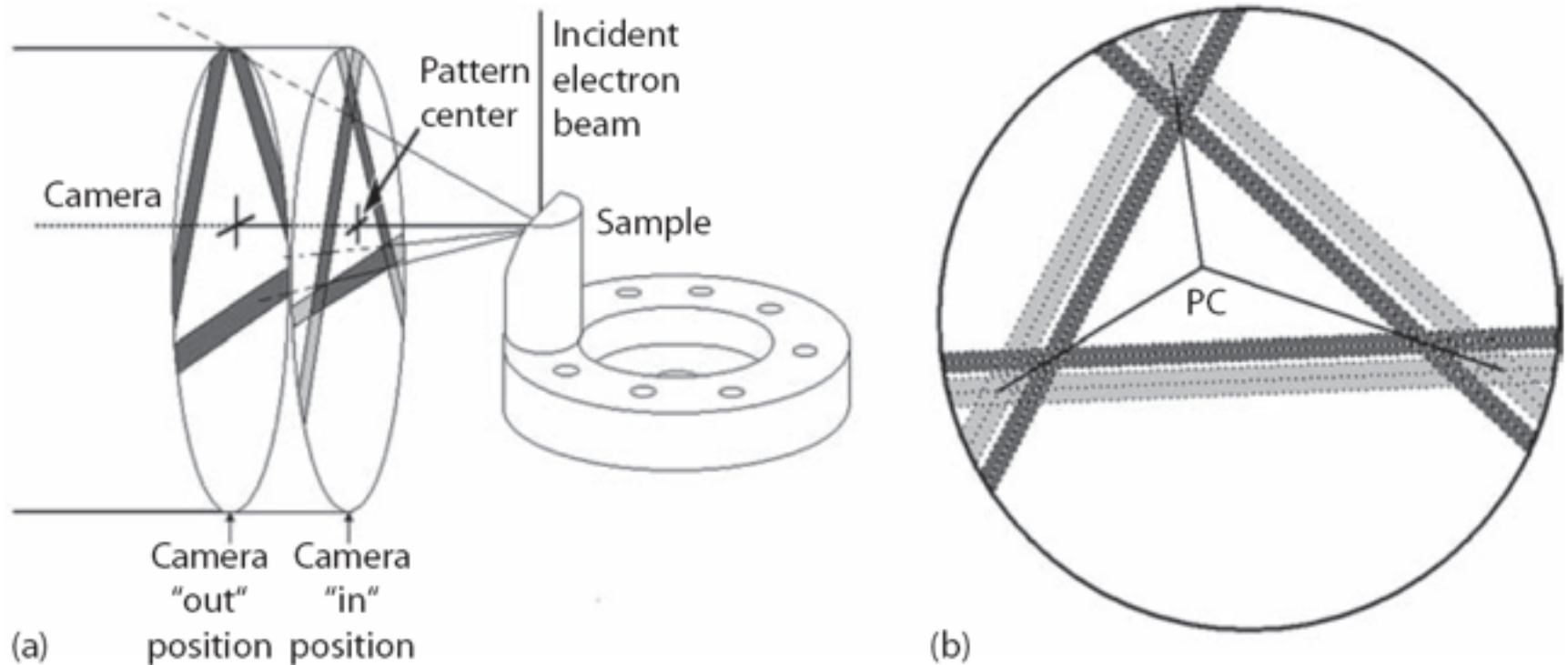
$$Z^* = L / \tan(35^\circ)$$



$$\cos\phi = \frac{\begin{pmatrix} x_1 - x_{PC} \\ y_1 - y_{PC} \\ Z_{SSD} \end{pmatrix} \cdot \begin{pmatrix} x_2 - x_{PC} \\ y_2 - y_{PC} \\ Z_{SSD} \end{pmatrix}}{\sqrt{(x_1 - x_{PC})^2 + (y_1 - y_{PC})^2 + Z_{SSD}^2} \cdot \sqrt{(x_2 - x_{PC})^2 + (y_2 - y_{PC})^2 + Z_{SSD}^2}}$$

$\phi$  is the angle between the two chosen zone axes,  $(x_1, y_1)$  and  $(x_2, y_2)$  are the coordinates  $(x_{PC}, y_{PC})$  are the coordinates of the PC

# Calibration procedures: pattern magnification



Principle of the "pattern magnification" method of EBSD calibration. (Courtesy of K. Dicks.)

# Summary of EBSD calibration methods

## Summary of EBSD Calibration Options

Method	Advantages	Disadvantages
Shadow casting	Accurate and repeatable; some protection against screen collision	Requires precision hardware that interferes with automatic pattern recognition
Known crystal orientation	Simplicity	Errors associated with crystal and mounting; some pattern recognition required
Pattern magnification	No special equipment or standards required	Some pattern recognition required
Iterative pattern fitting	No special equipment or standards required	Requires sophisticated programming and a “first guess” solution

# EBSD developments

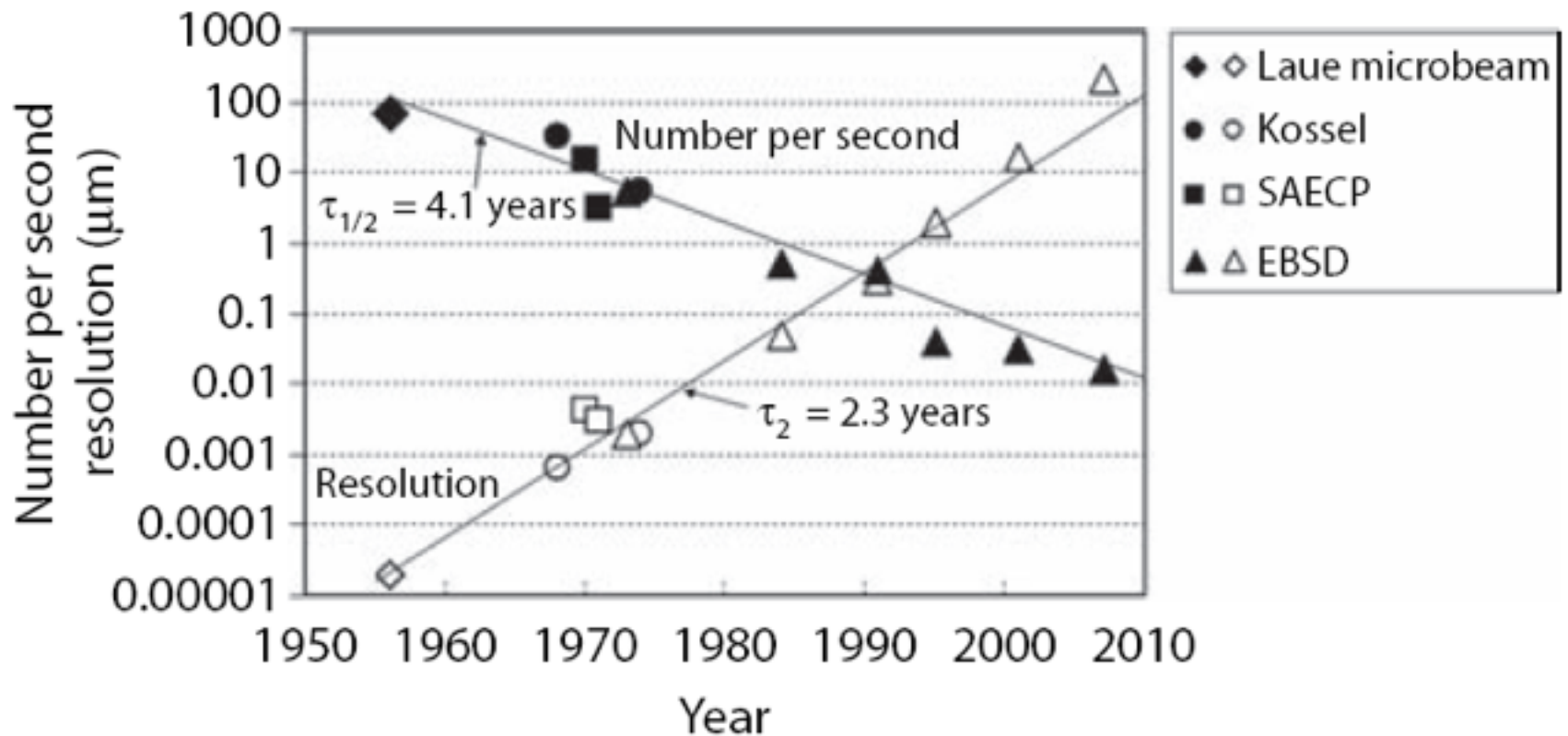


FIGURE 7.17

Improvements in spatial resolution and indexing speed as a function of year for several microdiffraction techniques. (Courtesy of W.B. Hutchinson.)

# EBSD operation: automation indexing

Recording the pattern

Contrast enhancement of the pattern

Correct the pattern background

Pre-processing

Hough transformation

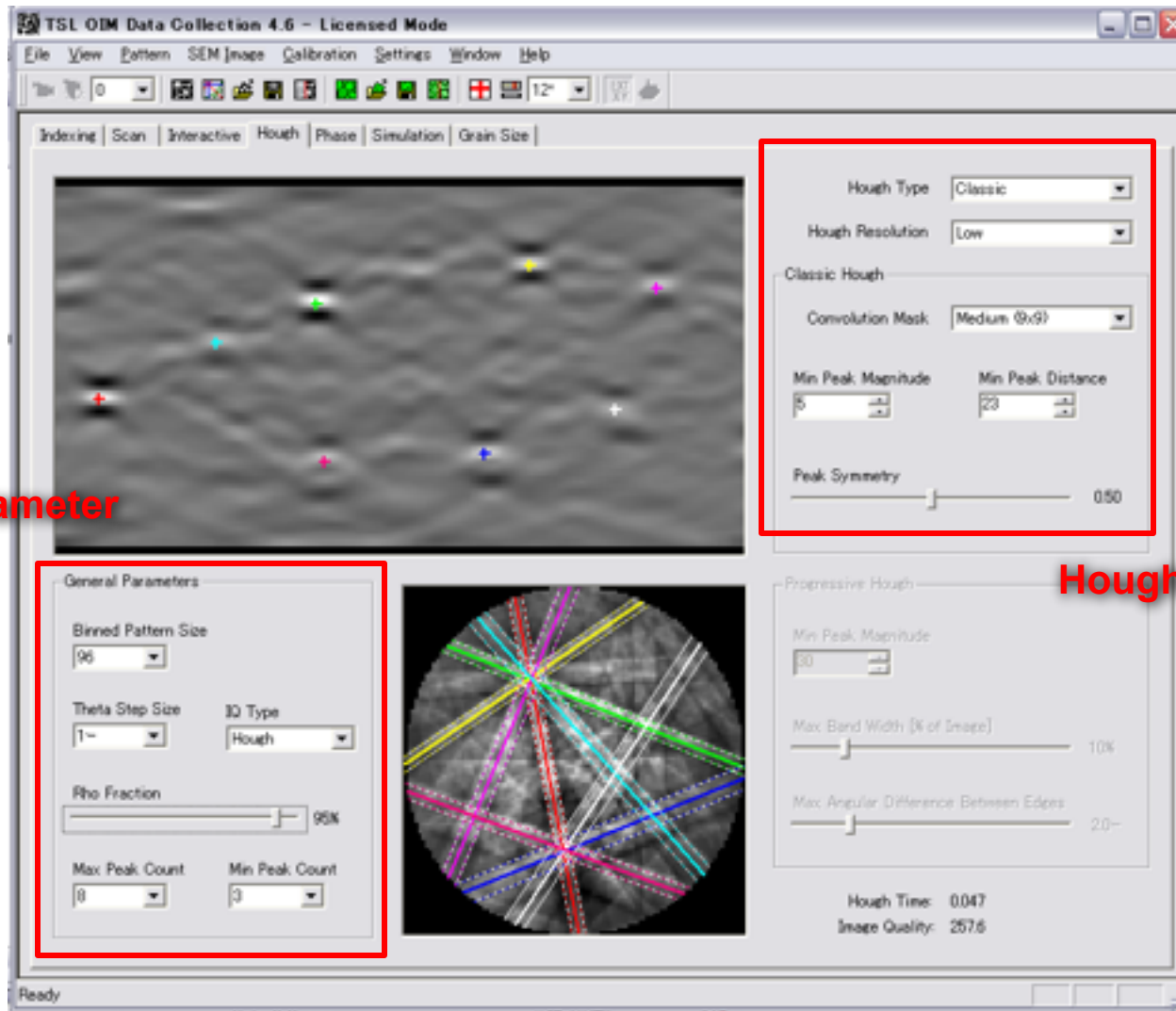
Transformation

Identification of the peaks in Hough space

Identification

# Adjusting parameters for Hough transformation

General Parameter



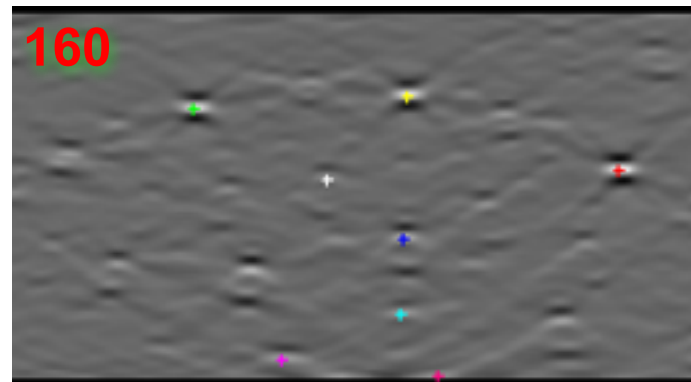
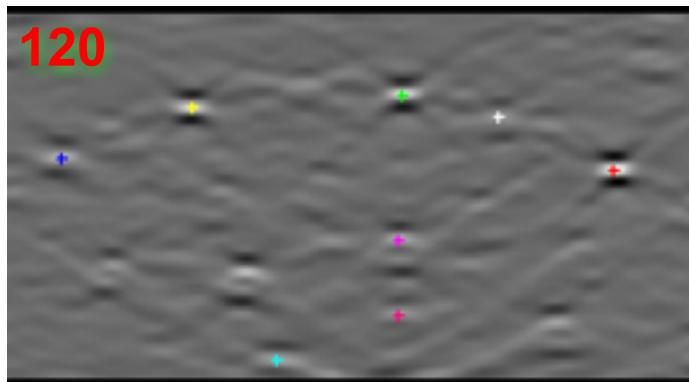
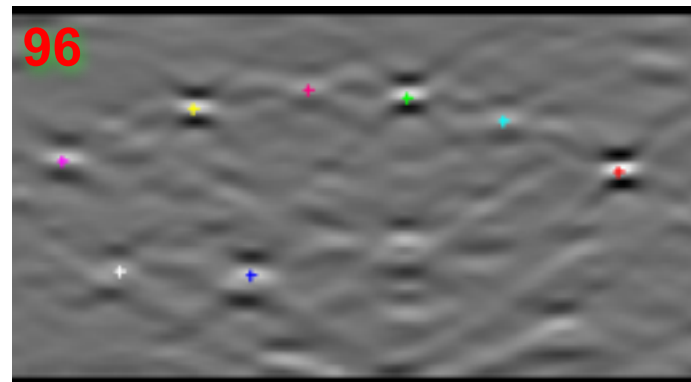
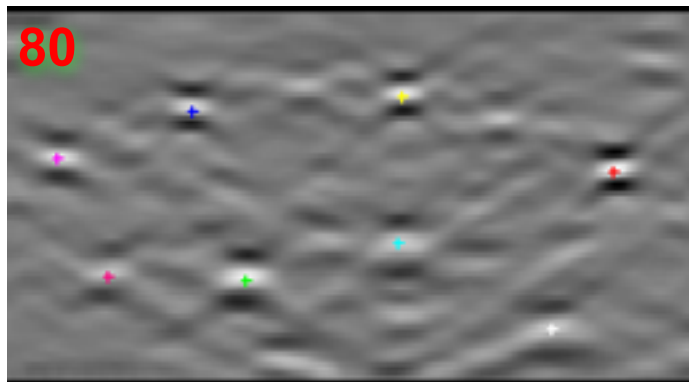
Hough Parameter



# General Parameter

## Binned Pattern Size:

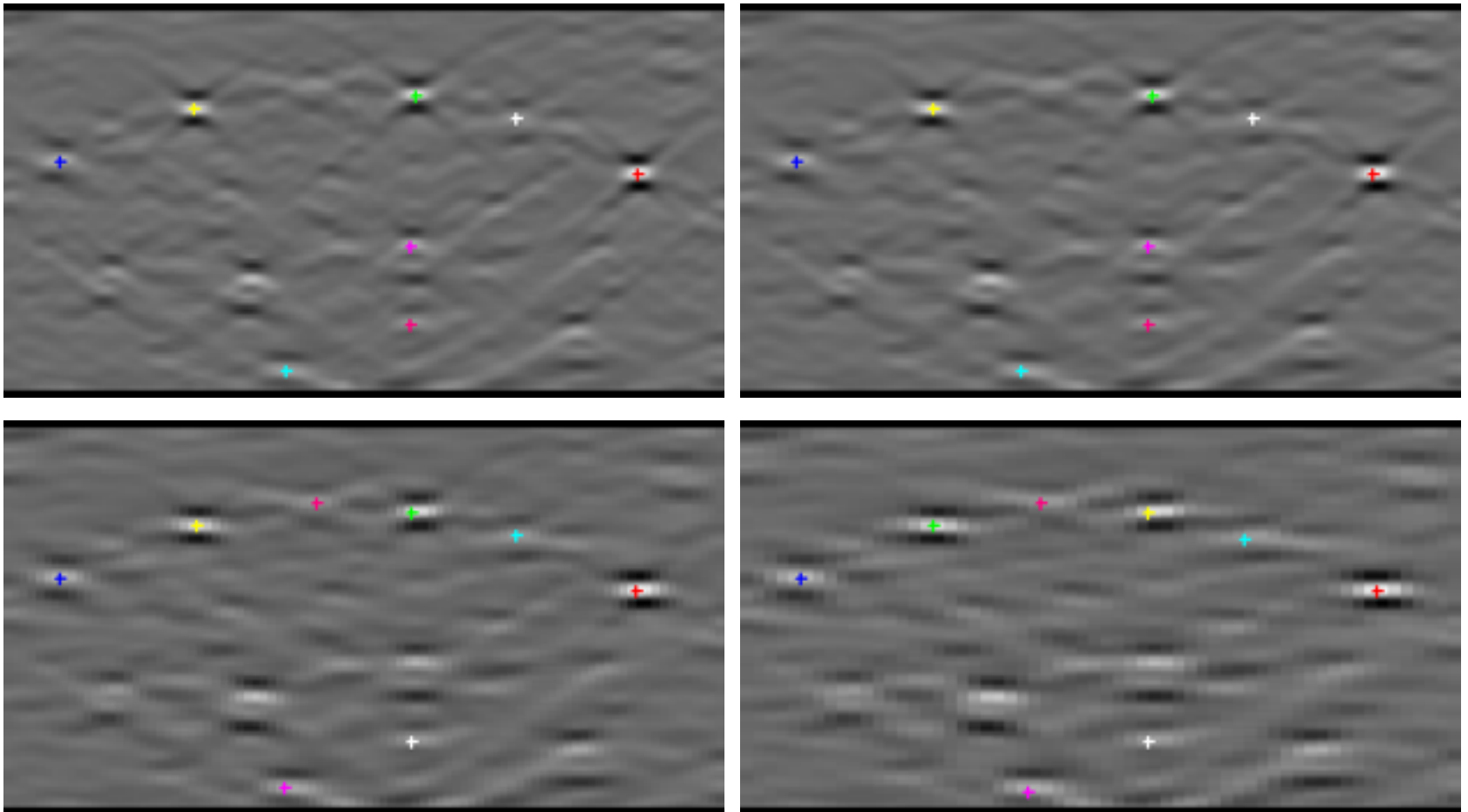
It sets the size of the patterns used by the low resolution Hough transform. The default setting is 96x96. Smaller settings result in faster processing times, while larger settings provide greater pixel density for line measurements.



# General Parameter

## Theta Step Size:

It sets the  $\theta$  value for Hough calculation. The default setting is 1. Smaller settings result in slower processing times, while larger settings provide faster calculation but rough image.

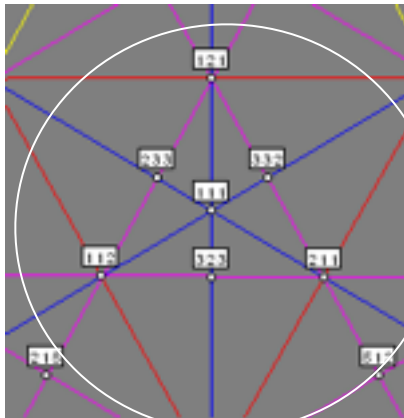


# General Parameter

## How many bands should be detected for indexing?

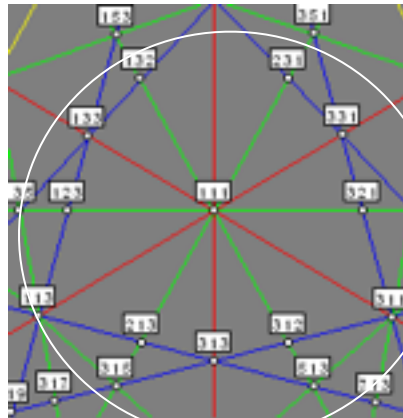
**Number of the bands necessary for indexing the pattern depends on the symmetry of the crystals.**

### Cubic : FCC



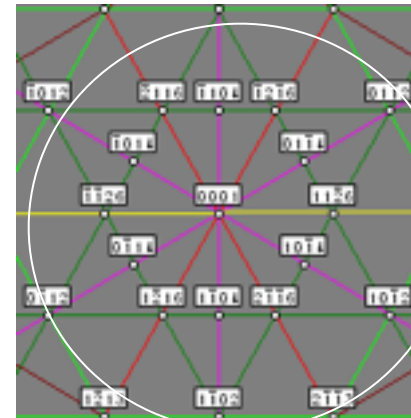
**7 bands are good enough to index this highly symmetry pattern.**

### Cubic : BCC



**7 bands are not good enough to decide the orientation when the  $\langle 111 \rangle$  axis becomes nearly center of the screen. 8 bands are necessary in this case.**

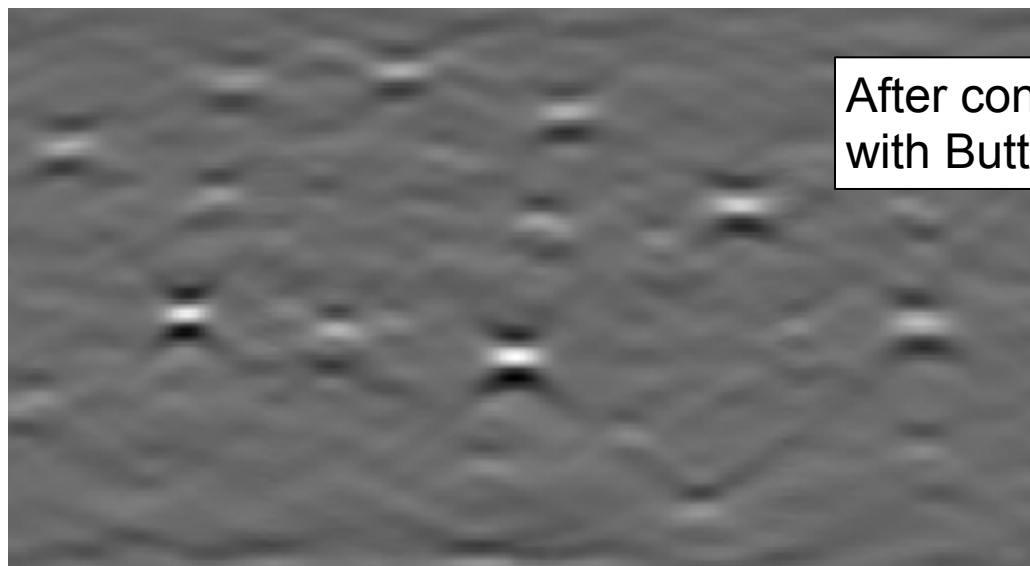
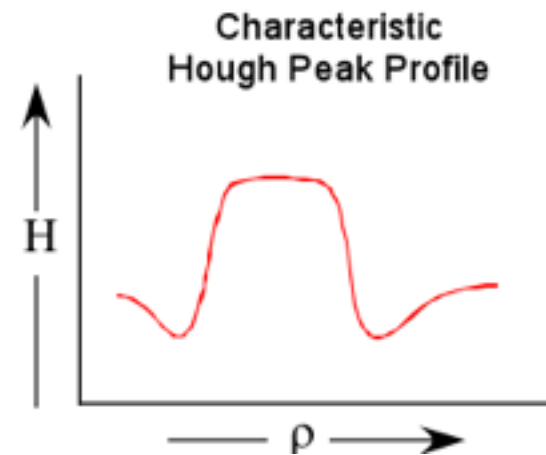
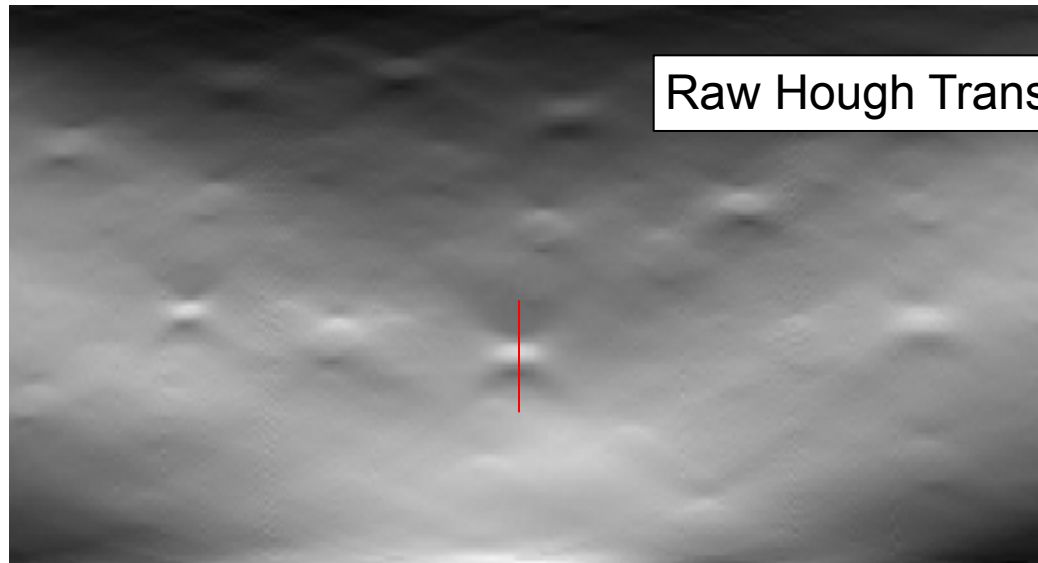
## Hexagonal



**<111>axis has 6 holds rotation symmetry. So additional one more band is good enough to decide the orientation of this pattern.**

**In actual case, the situation depends on also calibration and acquisition solid angle. Check in simulation page.**

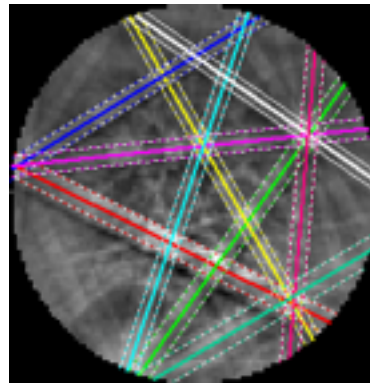
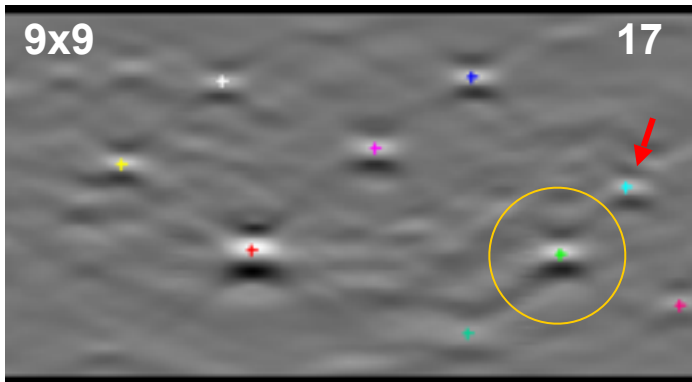
# Hough Parameter



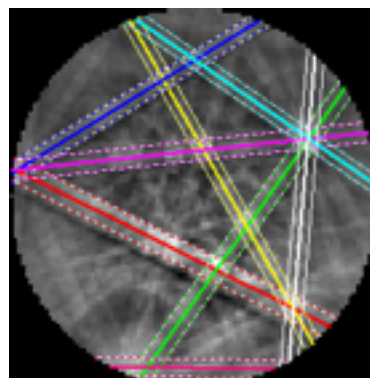
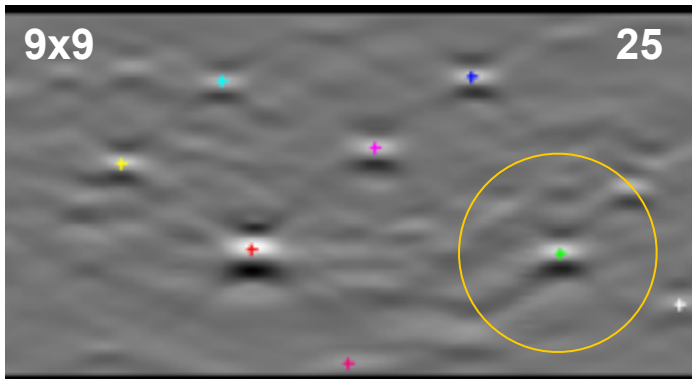
# Hough Parameter

## Minimum peak distance :

It prohibits to detect a peak within this pixel value from the detected peak.



The arrowed peak is detected in the Hough of 17, but it is not detected in the Hough of 25.



This parameter should be decided after thinking the symmetry of the crystal.

# Identifying by interplanar angle

1) Identify the  $hkl$  of the high contrast bands (bands likely to be detected by the Hough transform).

200  
111  
220  
311

2) Determine all of the symmetrically equivalent  $hkl$ 's.

200, 020, 002  
111,  $11\bar{1}$ ,  $1\bar{1}1$ ,  $\bar{1}11$   
220,  $2\bar{2}0$ , 202,  $20\bar{2}$ , 022,  $02\bar{2}$   
311,  $\bar{3}11$ ,  $3\bar{1}1$ ,  $31\bar{1}$ ,  $131$ ,  $\bar{1}31$ ,  $1\bar{3}1$ ,  $13\bar{1}$ ,  $113$ ,  $\bar{1}13$ ,  $1\bar{1}3$ ,  $11\bar{3}$

3) Form all possible pairs.

200, 020	200, 002	200, 111	200, $11\bar{1}$	?
020, 002	020, 111	020, $11\bar{1}$	020, $1\bar{1}1$	?
002, 111	002, $11\bar{1}$	002, $1\bar{1}1$	002, $\bar{1}11$	?
?				

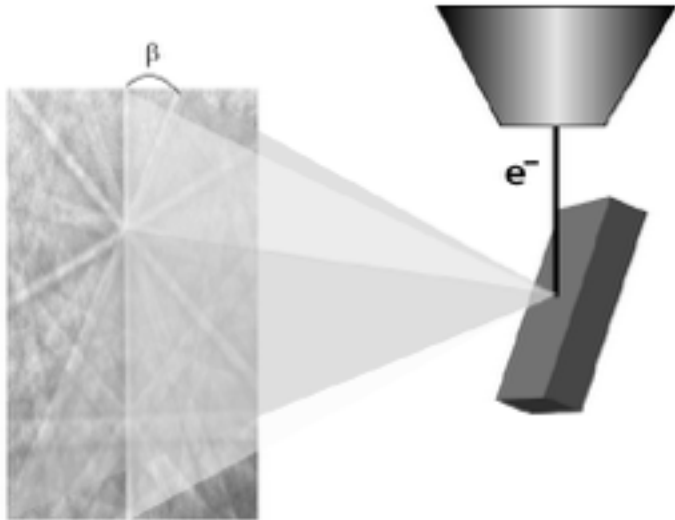
4) Calculate the angles between the plane pairs.

$200 \angle 020 = 90^\circ$	$200 \angle 002 = 90^\circ$	$200 \angle 111 = 54.7^\circ$	$200 \angle 11\bar{1} = 54.7^\circ$	?
$020 \angle 002 = 90^\circ$	$020 \angle 111 = 54.7^\circ$	$020 \angle 11\bar{1} = 54.7^\circ$	$020 \angle 1\bar{1}1 = 54.7^\circ$	?
$002 \angle 111 = 90^\circ$	$002 \angle 11\bar{1} = 90^\circ$	$002 \angle 1\bar{1}1 = 90^\circ$	$002 \angle \bar{1}11 = 90^\circ$	?
?				

5) Throw out duplicates and sort.

$200 \angle 311 = 25.2^\circ$	$111 \angle 220 = 35.3^\circ$	$111 \angle \bar{3}11 = 58.5^\circ$	$111 \angle 11\bar{1} = 70.5^\circ$
$111 \angle 311 = 29.5^\circ$	$200 \angle 220 = 45.0^\circ$	$220 \angle 202 = 60^\circ$	$200 \angle 131 = 72.5^\circ$
$220 \angle 311 = 31.5^\circ$	$311 \angle \bar{3}11 = 50.5^\circ$	$311 \angle 13\bar{1} = 63.0^\circ$	$111 \angle \bar{3}11 = 80^\circ$
$311 \angle \bar{3}11 = 35.1^\circ$	$200 \angle 111 = 54.7^\circ$	$220 \angle 3\bar{1}1 = 64.8^\circ$	?

# Identifying by interplanar angle



If the  $hkl$  of two bands in the pattern can be identified then the corresponding orientation can be calculated. By comparing the angle between two bands with an inter-planar angle look up table, the  $hkl$  pair associated with the band pair can be identified. However, unless the band widths are taken into account, which  $hkl$  belongs with which band is indeterminate. However, if 3 bands are used then the  $hkl$ s associated with all 3 bands can be determined.

# Identifying by inter-planar angle

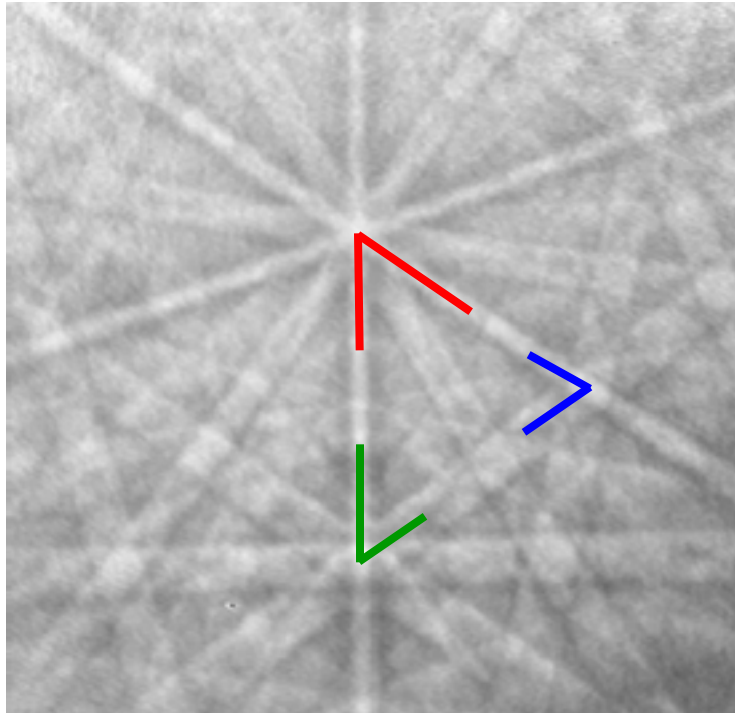
**Table 5.1.** Look-up table of interplanar angles for face-centered cubic crystals

Angle	$(hkl)_1$	$(hkl)_2$	Angle	$(hkl)_1$	$(hkl)_2$
25.2	200	311	64.8	220	$3\bar{1}1$
29.5	111	311	70.5	111	$11\bar{1}$
31.5	220	311	72.5	200	131
35.1	311	$31\bar{1}$	80.0	111	$3\bar{1}\bar{1}$
35.3	111	220	84.8	311	$1\bar{3}1$
45.0	200	220	90.0	111	$2\bar{2}0$
50.5	311	$3\bar{1}\bar{1}$	90.0	200	020
54.7	111	200	90.0	200	022
58.5	111	$31\bar{1}$	90.0	200	$1\bar{1}3$
60.0	220	202	90.0	200	$2\bar{2}0$
63.0	311	$13\bar{1}$			



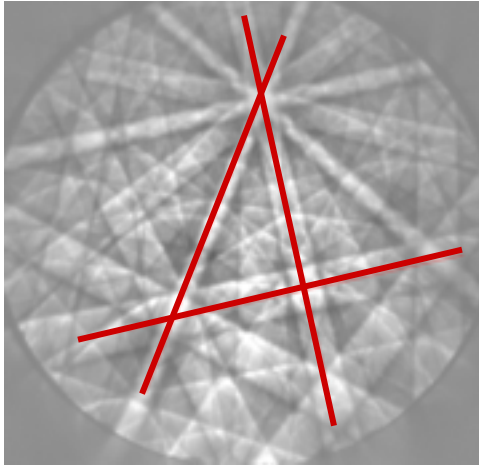
# Indexing: Bands – Triplet Solutions

A set of orientations is obtained from a triplet of bands by comparing the interplanar angles against a look-up table.



Angle	(hkl) <sub>1</sub>	(hkl) <sub>2</sub>
25.2	200	311
29.5	111	311
31.5	220	311
35.1	311	311
35.3	111	220
45.0	200	220
50.5	311	311
54.7	111	200
58.5	111	311
60.0	220	202
63.0	311	131
64.8	220	311
70.5	111	111
72.5	200	131
80.0	111	311
84.8	311	131
90.0	111	220
90.0	200	020
90.0	200	022
90.0	220	113
90.0	220	220

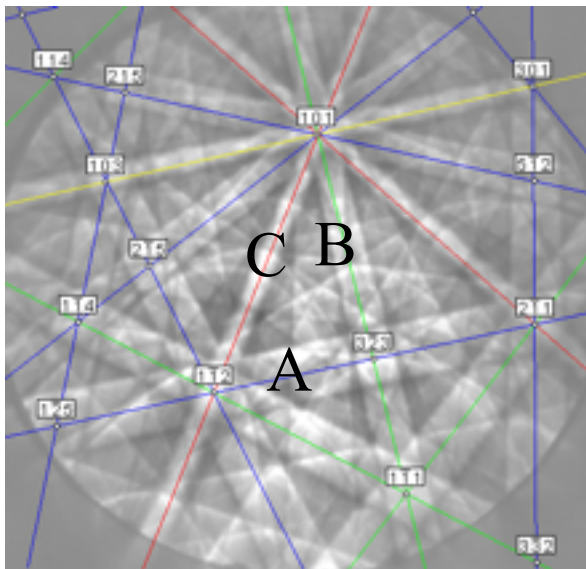
# Indexing: Bands – Triplet Solutions



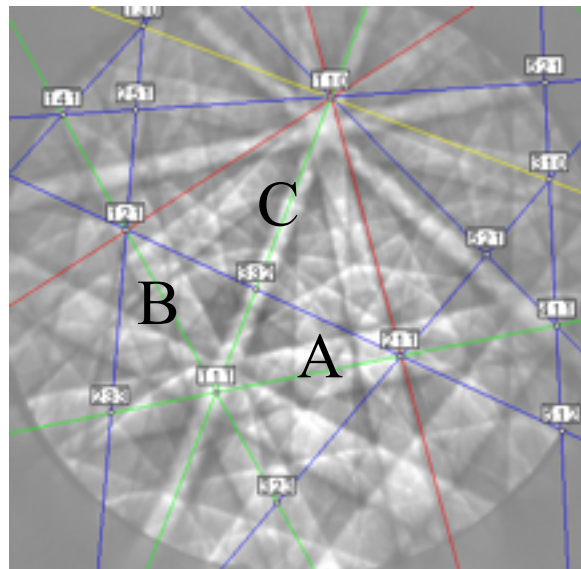
For a set of three bands, compare the interplanar angles against the LUT and determine all possible indexing solutions.

Even with 3 bands more than one solution is possible especially if the tolerance angle is wide.

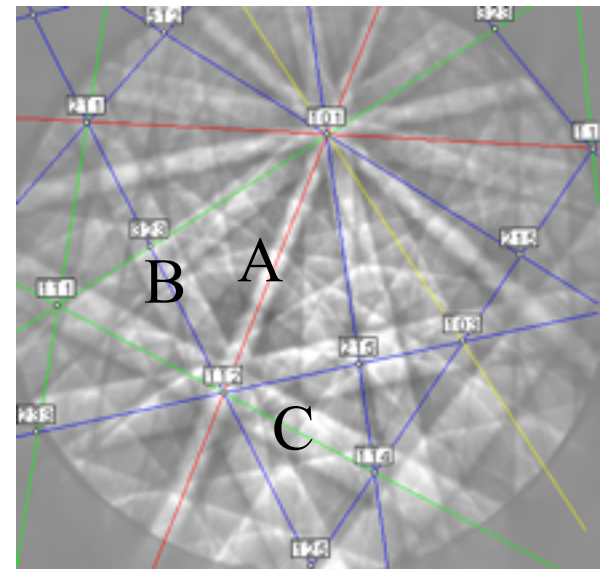
Solution 1



Solution 2



Solution 3



# Indexing: Bands – Triplet Solutions

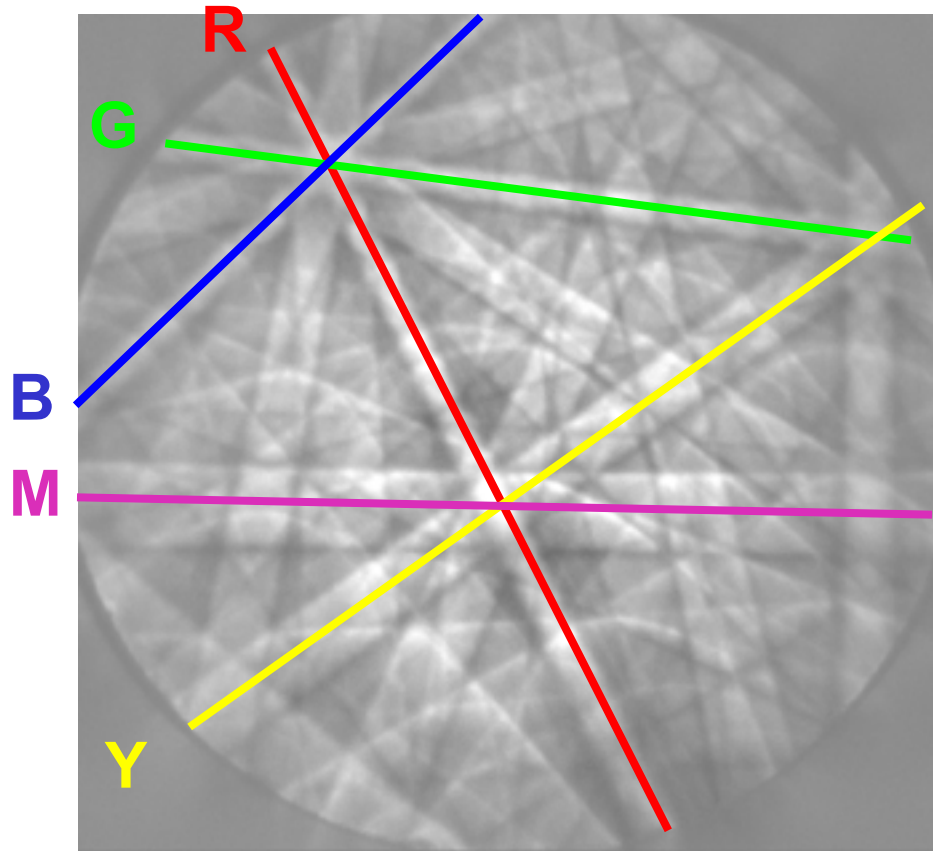
$$\# triplets = \frac{n!}{(n-3)! \cdot 3!}$$

For a given number of bands,  $n$ , used for pattern indexing, the number of band triplets is determined by this formula.

Typically 7 to 9 detected bands are used for automatic indexing.

$n$	$\# triplets$
3	1
4	4
5	10
6	20
7	35
8	56
9	84

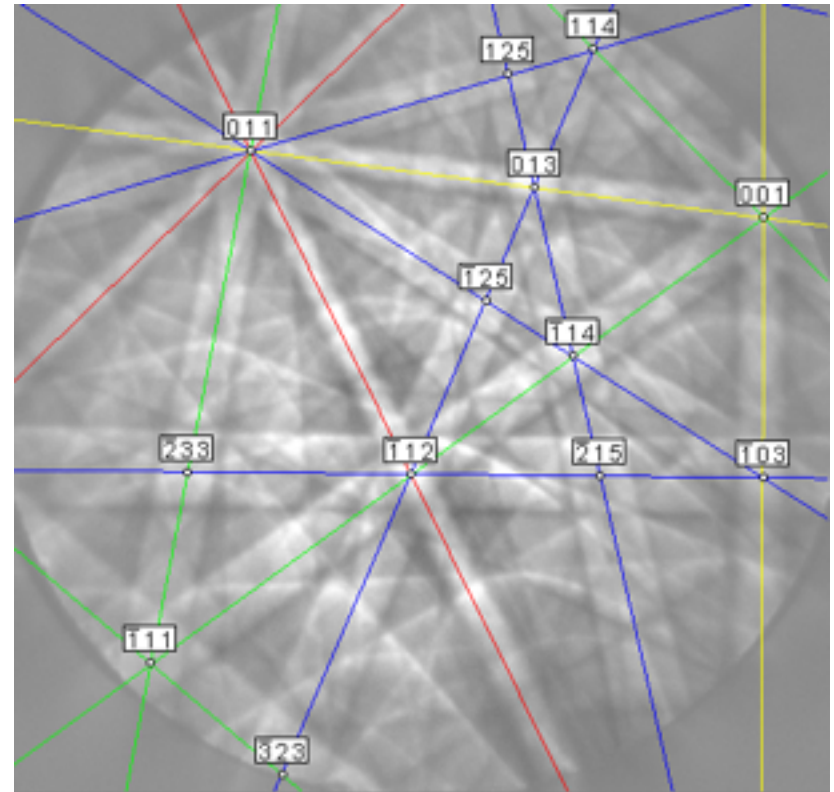
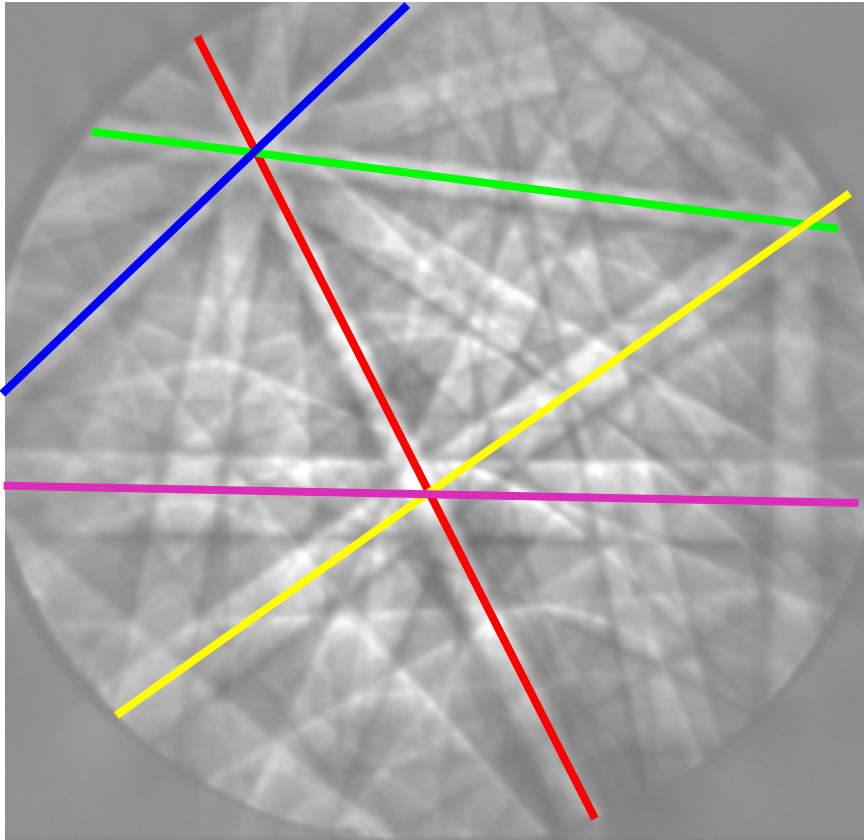
# Indexing: Bands – Triplet Solutions



Triplet	Solution 1 ( $V_1$ )	Solution 2 ( $V_2$ )	Solution 3 ( $V_3$ )
R, G, Y	X		
R, G, B	X		
R, G, M	X		
R, Y, B	X		
R, Y, M	X	X	
R, B, M	X		
G, Y, B	X		
G, Y, M	X		
G, B, M	X		
Y, B, M	X		X
Total	10	1	1

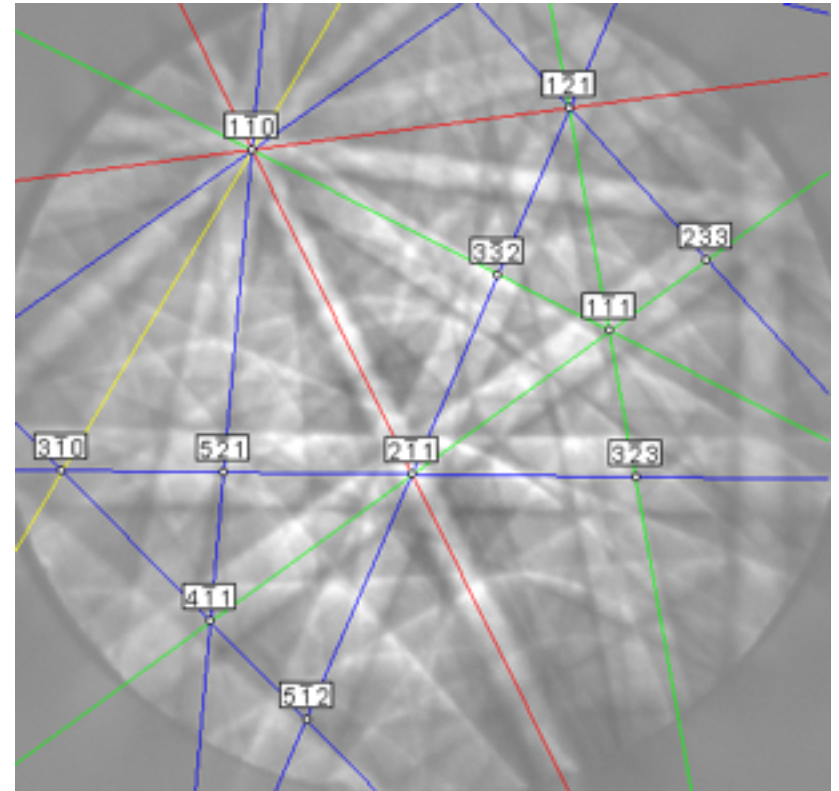
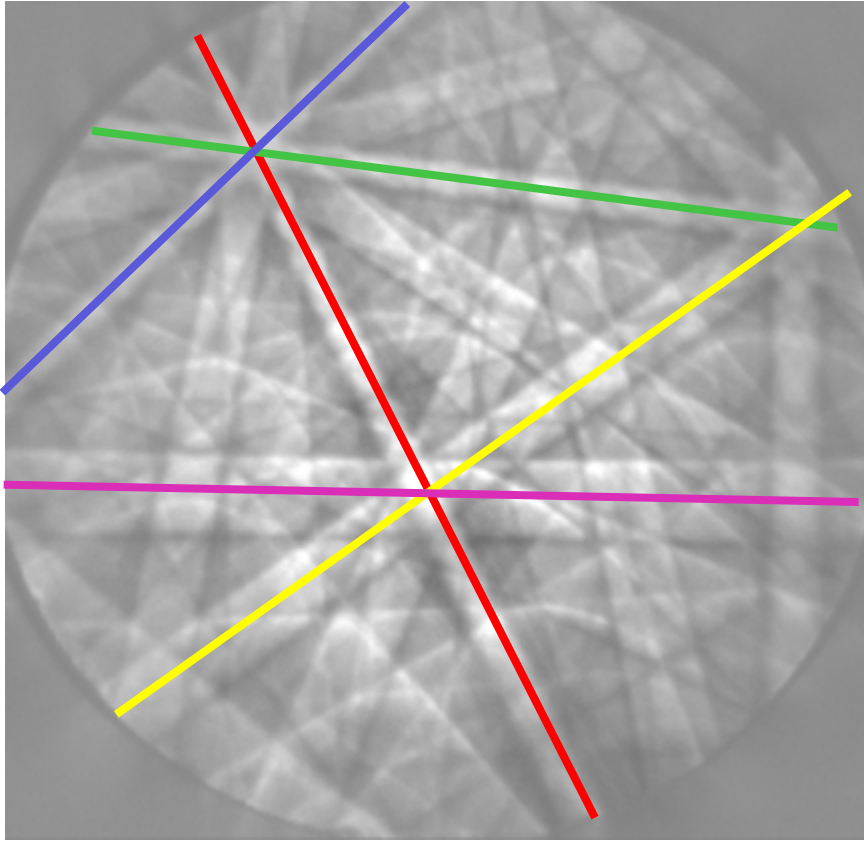
For this set of 5 detected bands, 10 triplet combinations are possible. For each of these 10 triplets, solution  $V_1$  matched. Solutions  $V_2$  and  $V_3$  each matched one triplet only.

# Indexing: Bands – Triplet Solutions



Solution  $V_1$ . Notice how each of the 5 bands match bands in the indexing solution overlay.

# Indexing: Bands – Triplet Solutions

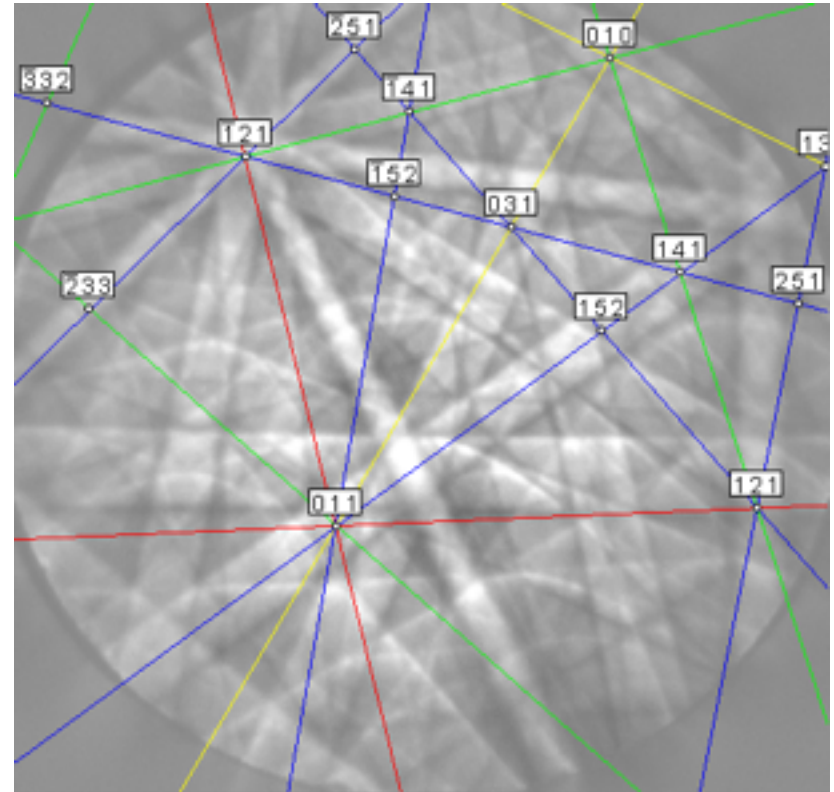
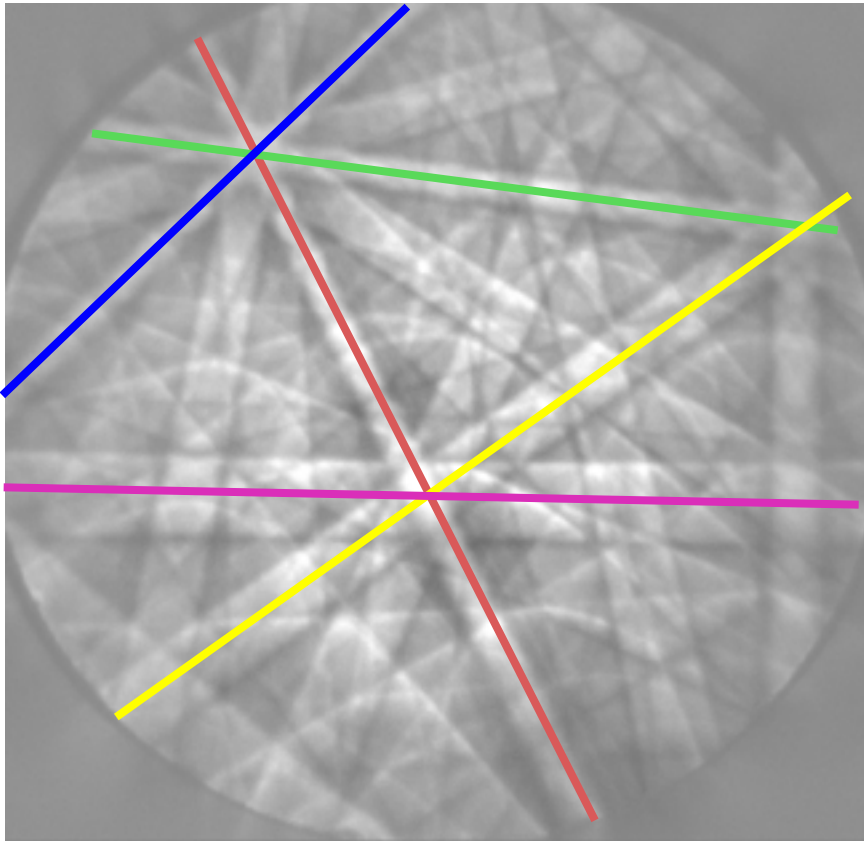


Fit = 1.30°

Solution  $V_2$ . Only the red, yellow, and magenta band triplet now match, producing a higher fit value.



# Indexing: Bands – Triplet Solutions



Fit = 1.91°

Solution  $V_3$ . Only the blue, yellow, and magenta band triplet now match, producing a higher fit value.

# Indexing: Bands – Confidence Index

Triplet	Solution 1 ( $V_1$ )	Solution 2 ( $V_2$ )	Solution 3 ( $V_3$ )
R, G, Y	X		
R, G, B	X		
R, G, M	X		
R, Y, B	X		
R, Y, M	X	X	
R, B, M	X		
G, Y, B	X		
G, Y, M	X		
G, B, M	X		
Y, B, M	X		X
Total	10	1	1

$$CI = \frac{V_1 - V_2}{V_{ideal}}$$

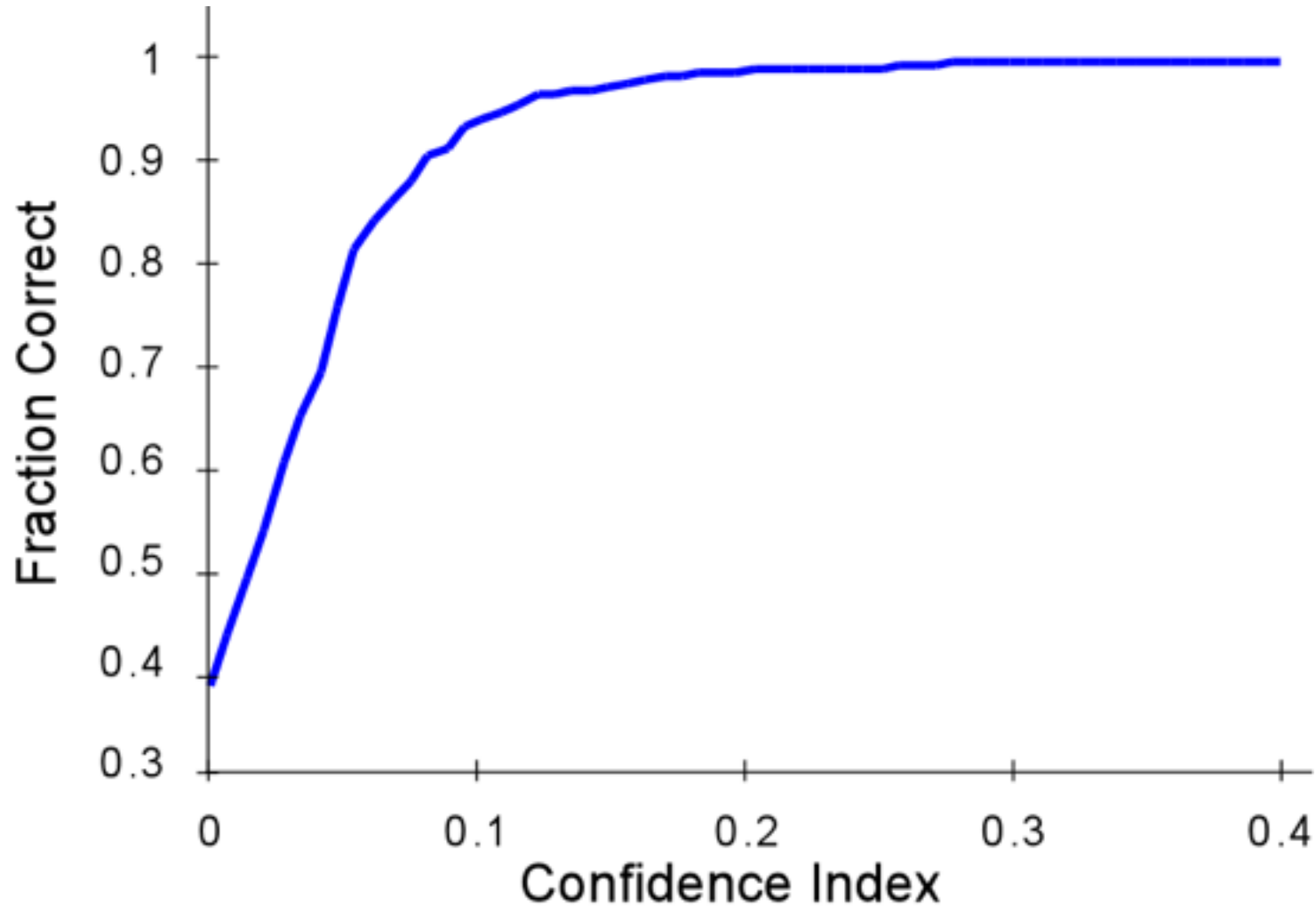
$$V_{ideal} = \frac{n!}{(n-3)! \cdot 3!}$$

$$CI = \frac{10 - 1}{10} = 0.9$$

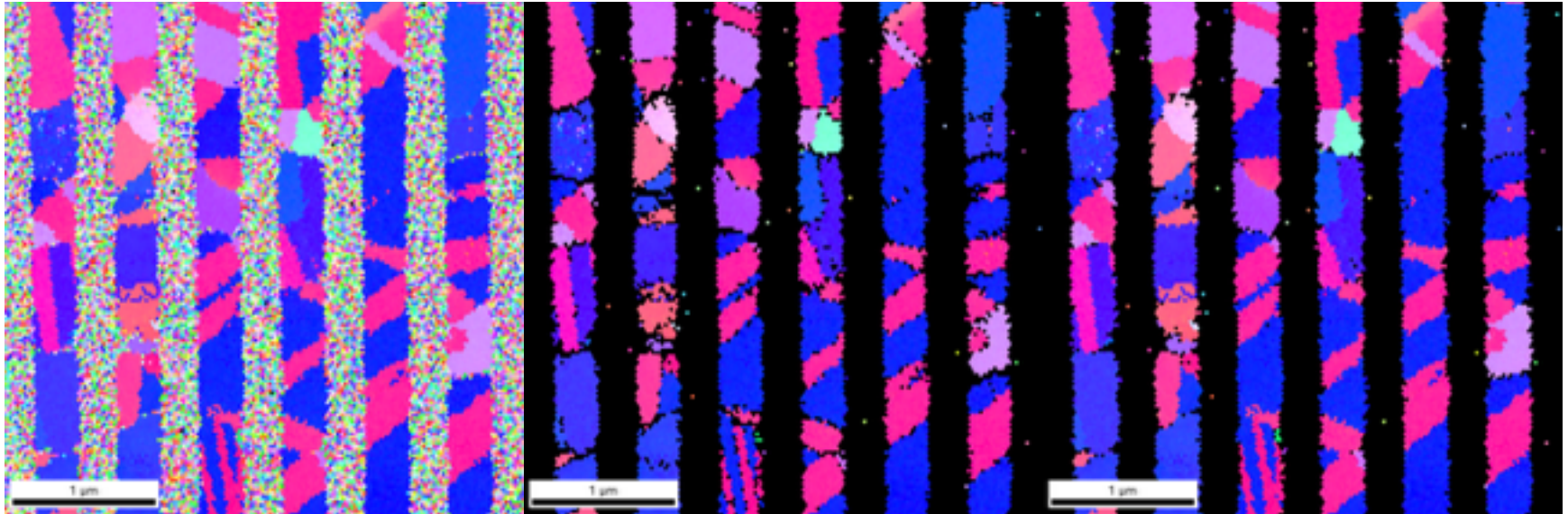
The confidence index,  $CI$ , measures the uniqueness of an orientation solution relative to the total number of possible votes.



# Indexing: Bands – Confidence Index



# Indexing: Bands – Confidence Index



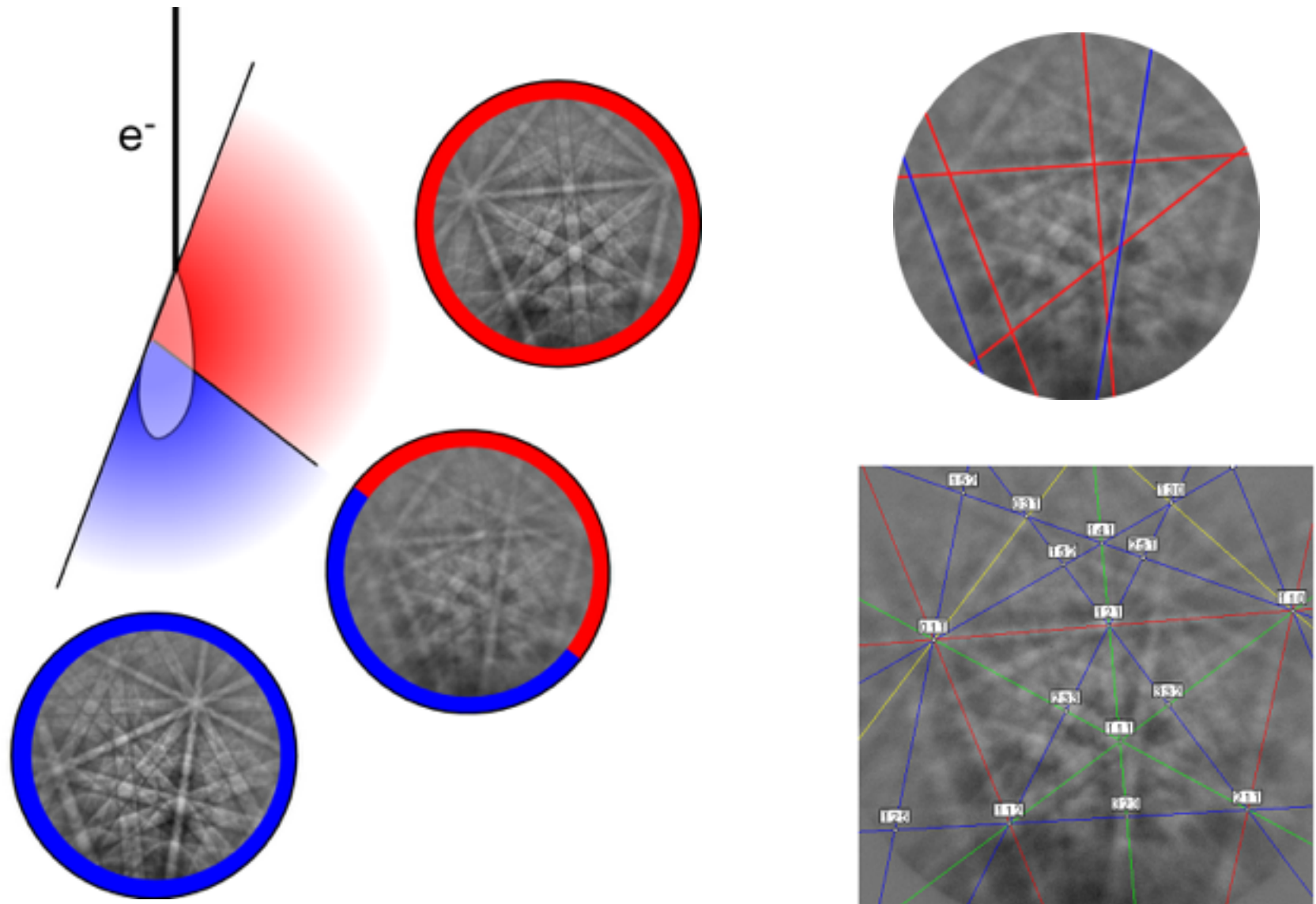
All Data

$C/>0.3$

$C/>0.3$  After CIS

The Confidence Index Standardization (CIS) feature recovers the low confidence data points that have the correct orientation measurements. This example shows the results from Cu IC lines, where the area between the lines is filled with amorphous SiO<sub>2</sub>. In the all data image, the SiO<sub>2</sub> area is noisy. In the  $C/>0.3$  area, this area is black, indicating a low confidence as expected. However, some of the grain boundary areas are also black. These are recovered using the CIS feature.

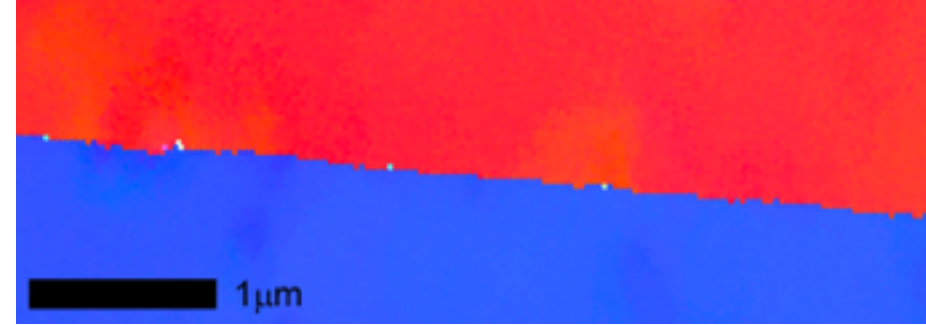
# Indexing: Deconvoluting Patterns



# Indexing: Deconvoluting Patterns & CI

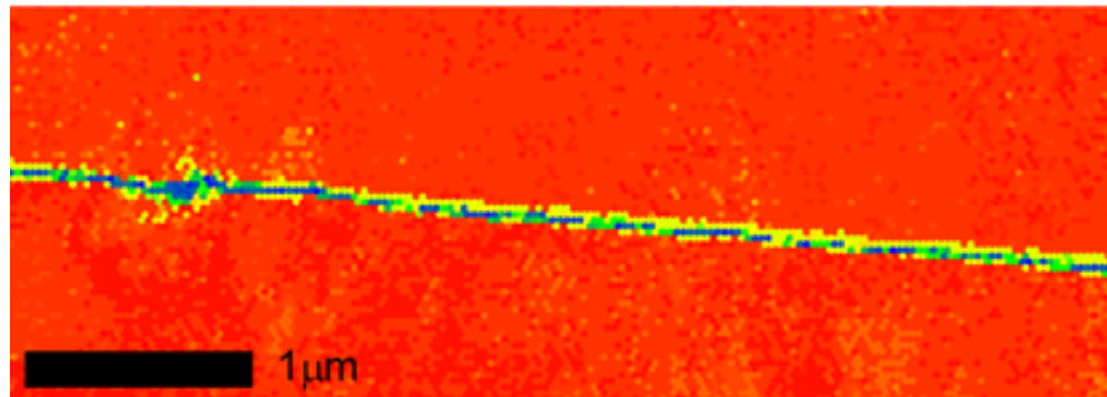


**IQ map**



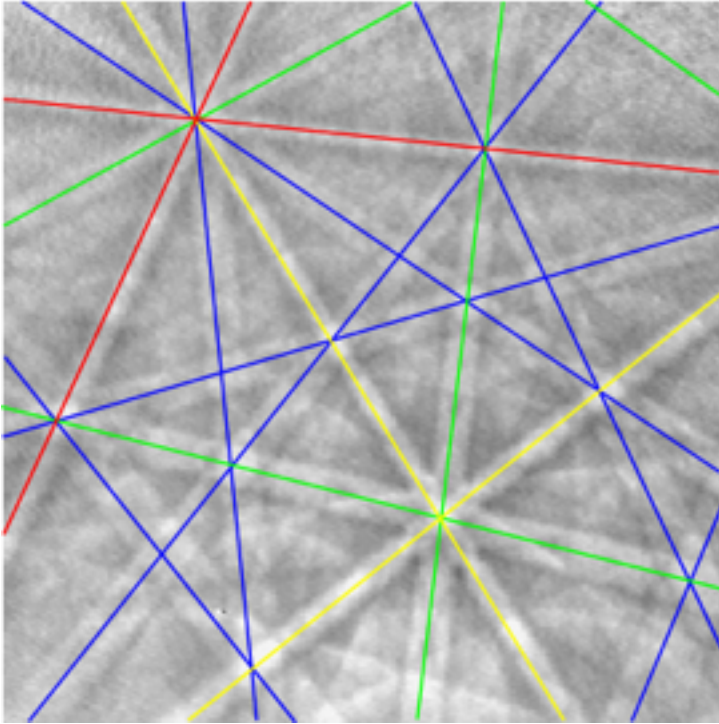
**IPF map**

**CI map**

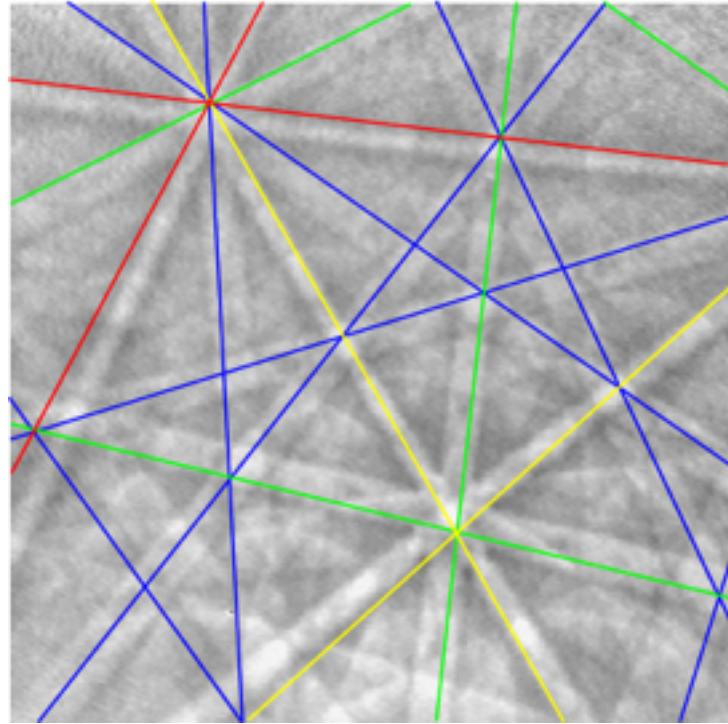


# Indexing: Fit

Misalignment between the detected bands and the recalculated bands.



Fit =  $0.28^\circ$



Fit =  $2.08^\circ$



# Calibration: Working Distance

