

EBSD SYSTEM

EDAX™

FEI Quanta 3D SEM/FIB

EBSD SYSTEM

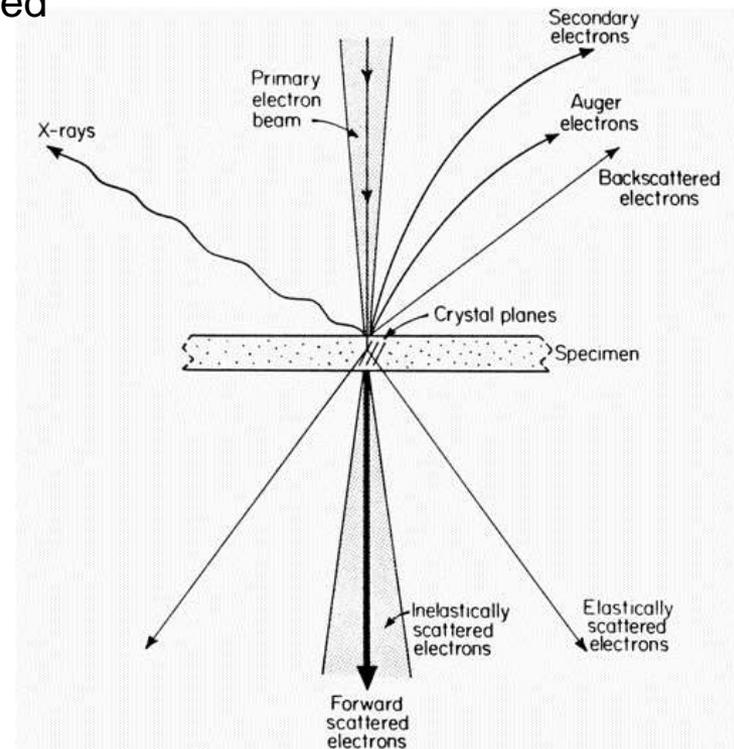
Between "products" caused by electron-matter interactions:

Elastically scattered electrons: No energy loss, the change in direction is in the order of degree. In the case of a crystalline material, the direction is determined by Bragg's law.

TEM diffraction, TEM dark field image, used by high resolution electron microscopy (HREM)

and

EBSD Electron BackScattered Diffraction
(visszaszórt elektron diffrakció)



EBSD – results of multiple processes: multiple scattering and interference

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How It Works?

In case of a thin single crystal sample - diffraction dots (for slightly thicker - Kikuchi lines)

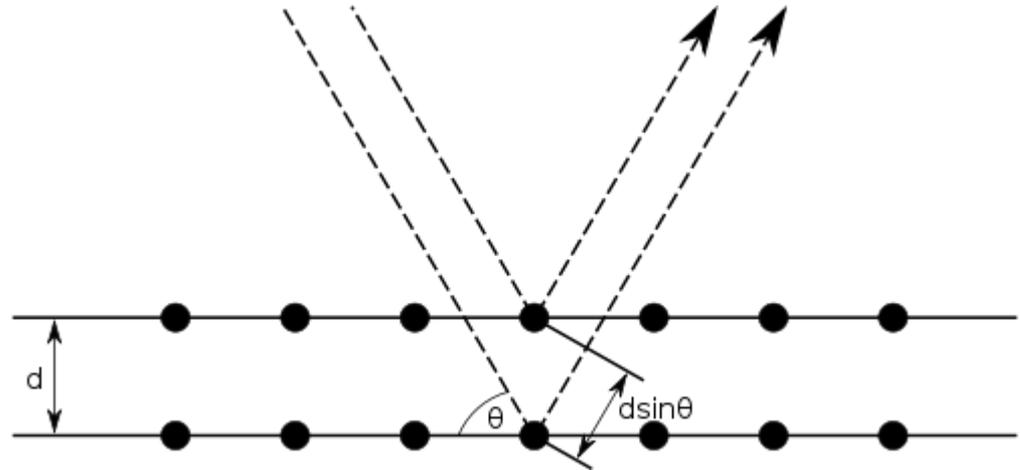
For thick sample and focused beam - Kikuchi bands

- elastic and low energy loss inelastic electron-electron collisions;
- electron Bragg scattering on the lattice planes

elastic scattering

interference

$$n\lambda = 2d \sin \Theta$$



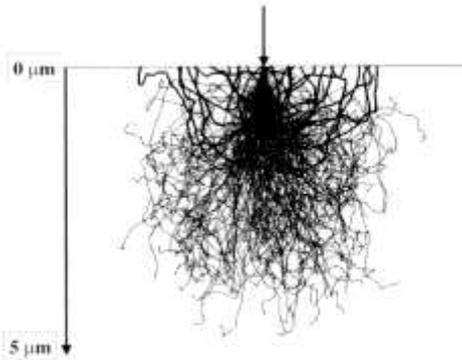
For 20 – 30 keV electrons λ is small $\Rightarrow \Theta$ is small too

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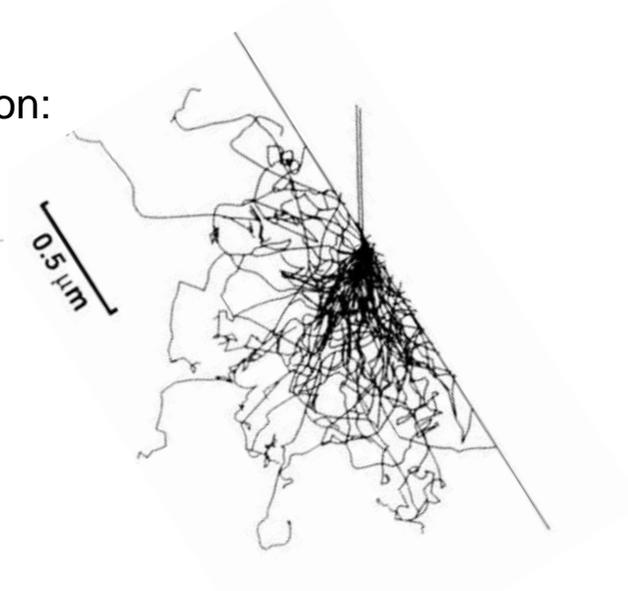
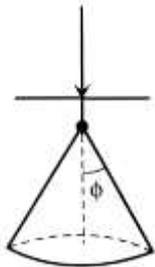
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Optimization of excitation

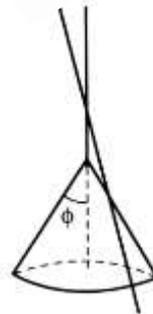
Monte-Carlo simulation:



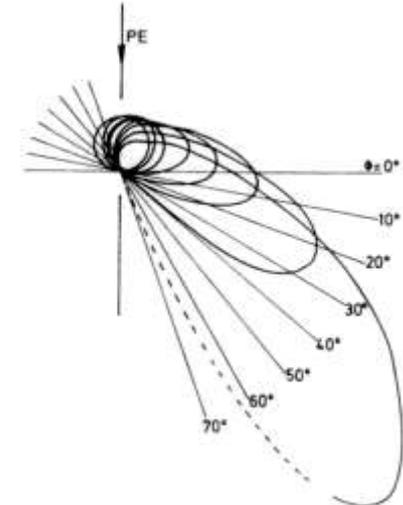
perpendicular excitation



oblique excitation



BSE yield :

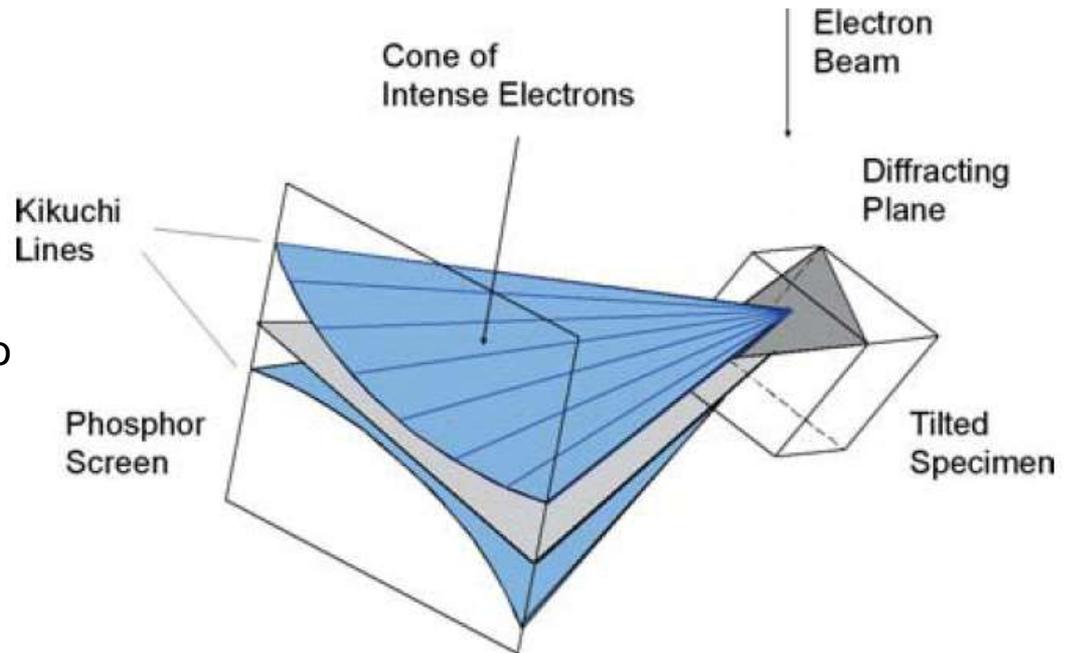


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EBSD imaging

- Bragg diffraction in the upper 20 nm of the surface
- Kossel cones corresponding to the direction of Bragg reflections
- one band belongs to one diffraction plane system
- small phosphor screen relative to the radius of the cone
- Kikuchi bands
- it is a kinematic model - the dynamic one provides a more detailed explanation



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Hikari camera + software

➤ **Hikari camera**

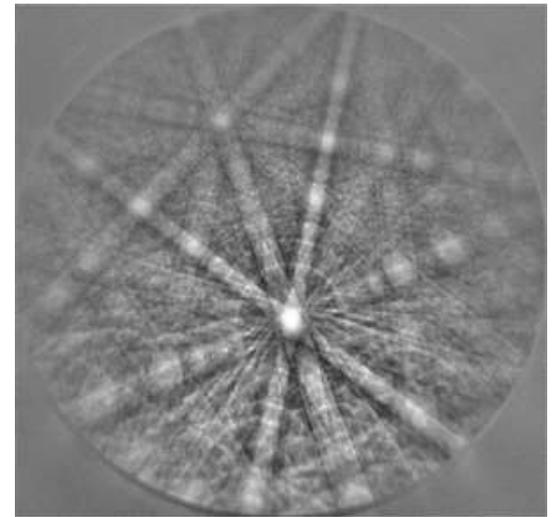
- *vacuum side phosphor surface*
- *special glass imaging optics;*
- *advanced CCD chip.*



➤ **Software (OIM 7.3)**

- *fast EBSD data collection;*
- *triplet indexing option - more accurate indexing;*
- *integrated camera console;*
- *adjustable image processing.*

➤ **450 pps (indexed patterns per second) image capturing**



EBSD pattern – EBSP

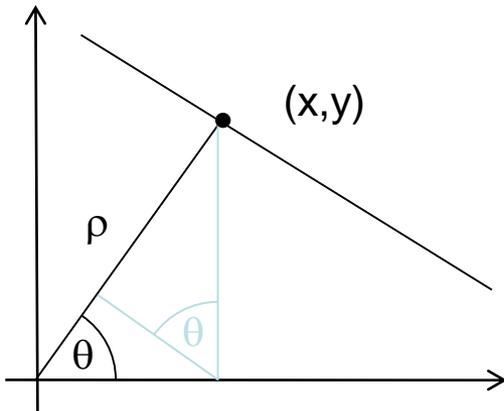
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Band Detection

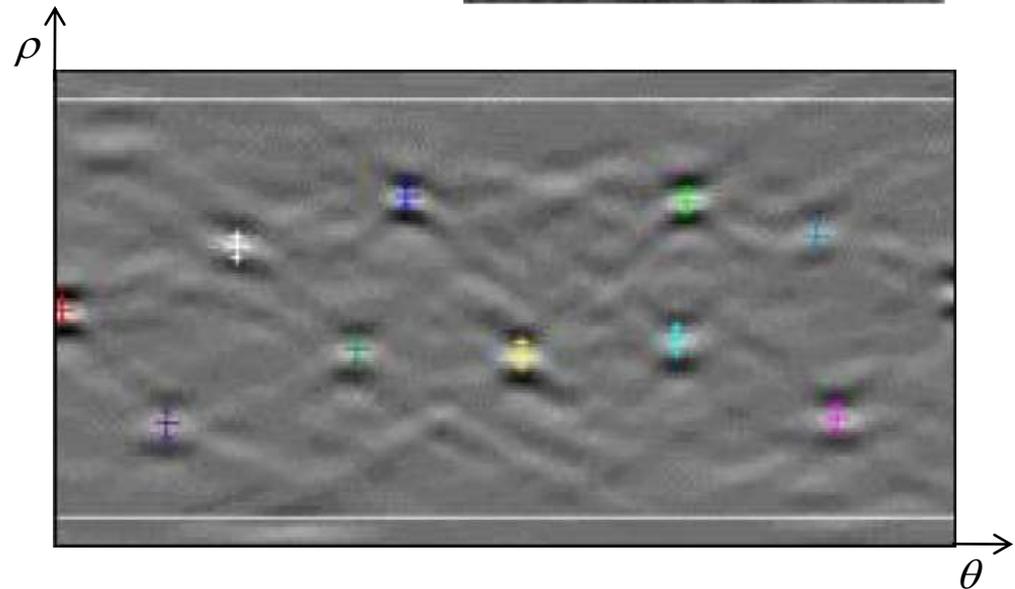
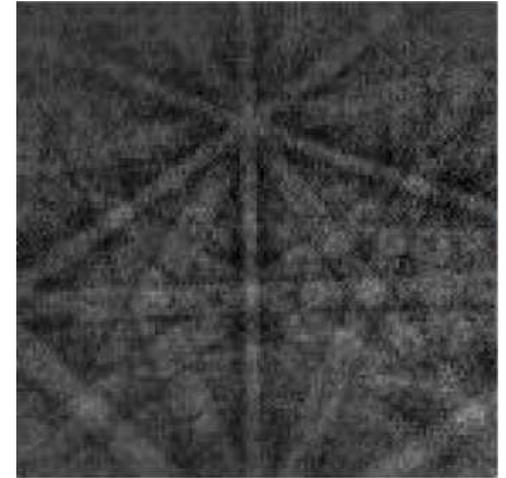
looking for bands – *Hough transformation*:

(x,y) plane \rightarrow (ρ,θ) rectangle



$$\rho_i = x_k \cos \Theta_i + y_k \sin \Theta_i$$

$$I_{(\rho,\Theta)} = \sum_k I_{(x_k, y_k)}$$



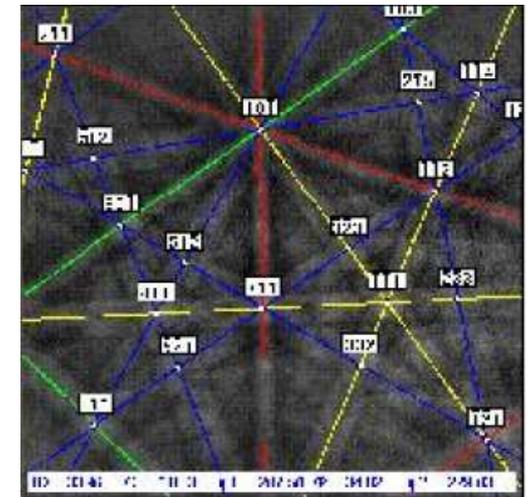
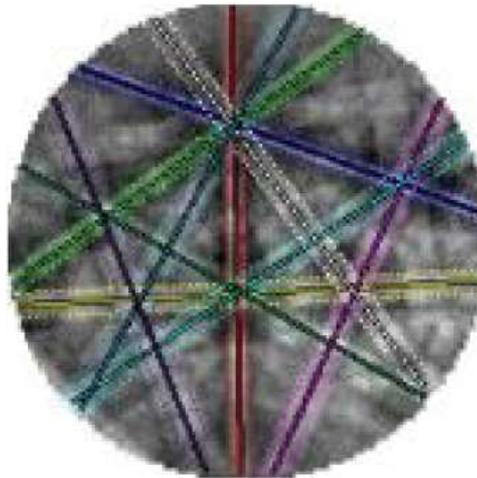
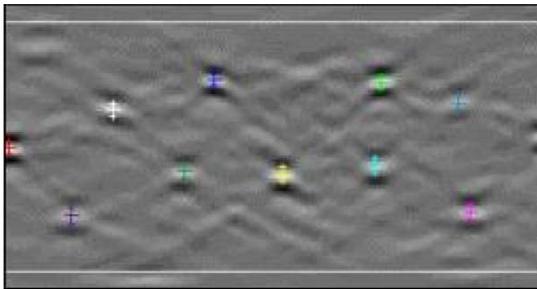


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Phase identification I.

- Hough transformation;
- maxima -> bands;
- indexing (crystallographic data)

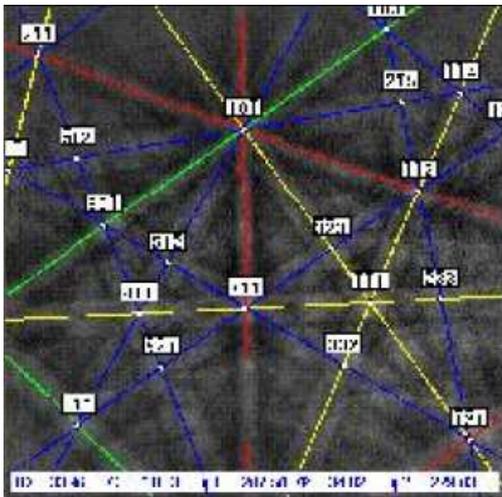


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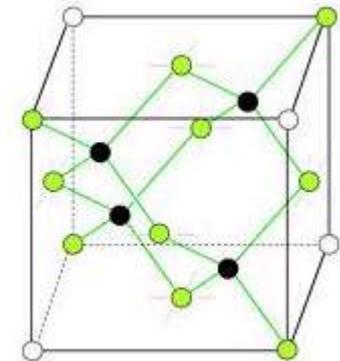
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Phase identification II.

- indexing (crystallographic data);
- weighting of possible solutions;
- crystal orientation and phase determination



		Indexing Solutions										
		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			
Σ		2	4	10	2	1	1	1	1	1	1	1



Voting Table

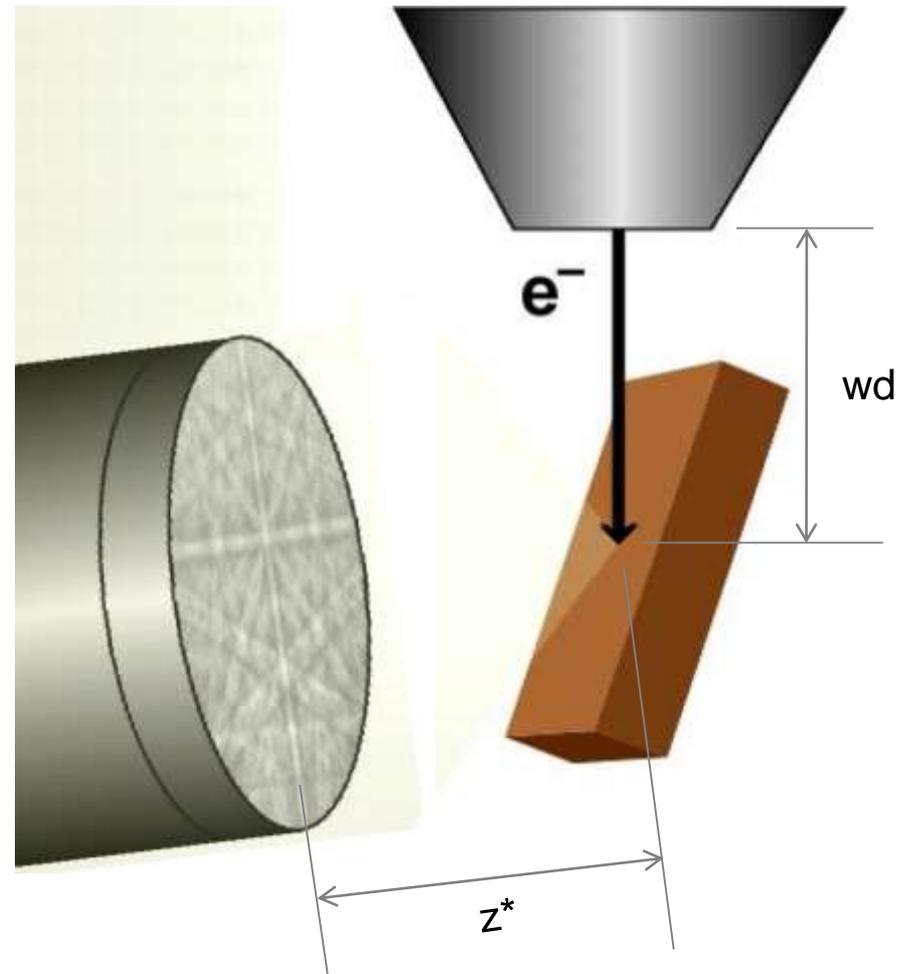
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EDAX – FEI setup

Geometric layout :

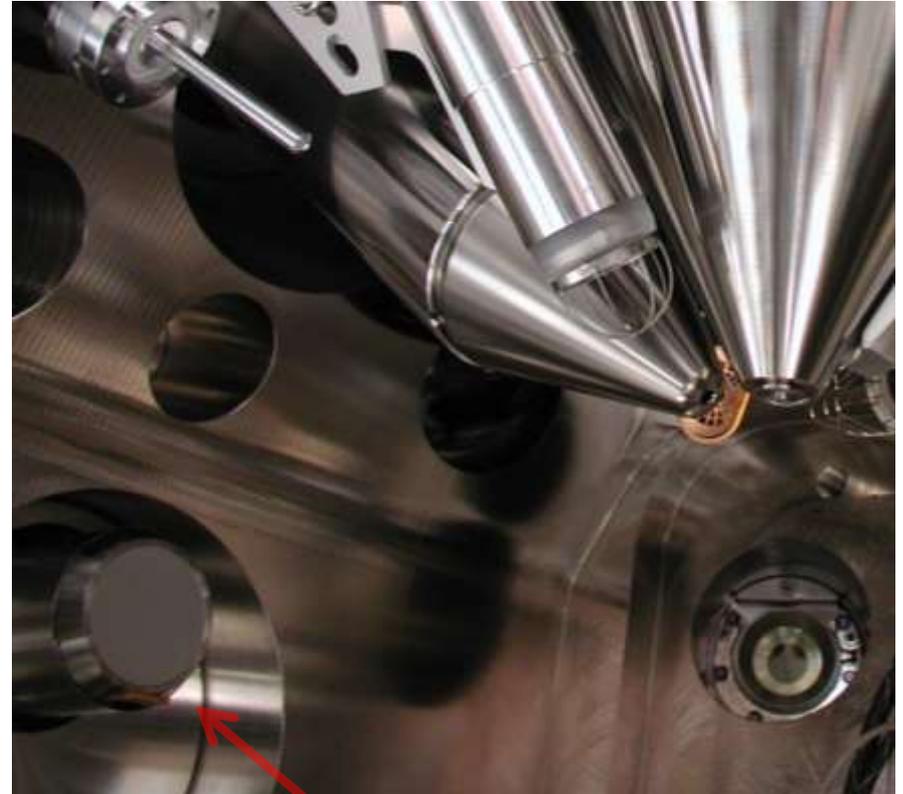
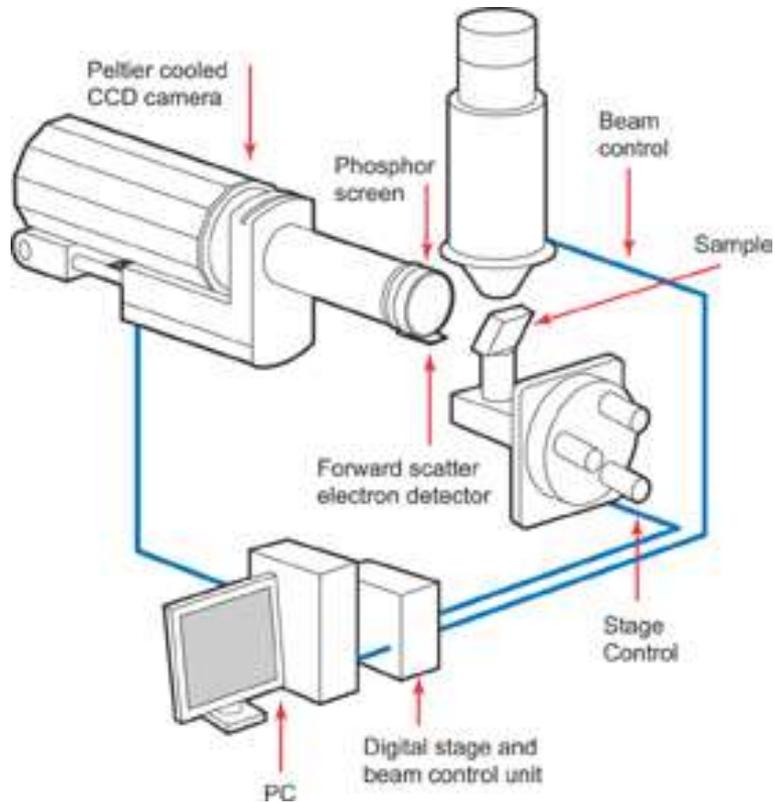
- 70° tilted sample;
- -7° tilted camera;
- working distance: $wd = 5-25\text{ mm}$
(atomic number dependent);
- $z^* = 30-150\text{ mm}$ camera distance



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EDAX – FEI setup



Hikari camera

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ion column

electron column

Omniprobe
nanomanipulator

gas injectors

EDX detector

sample
chamber door
sample holder with
mechanical operating
buttons

continuous
dynode electron
multiplier = CDEM
detector (SE, SI)

EBSD detector

GSED preamplifier



FEI Quanta 3D SEM/FIB

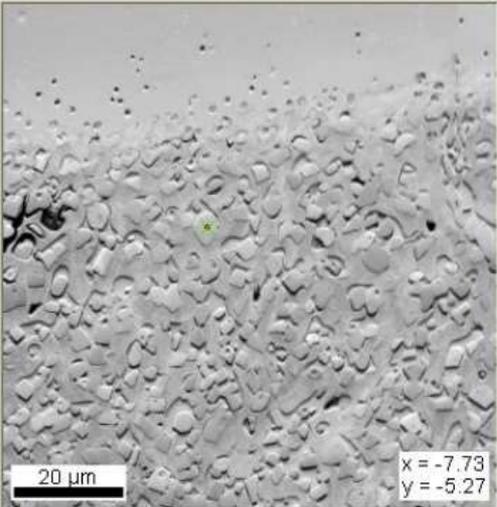
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TSL OIM Data Collection 5.1 - Licensed Mode

File View Camera Pattern SEM Image Calibration Settings Window Help

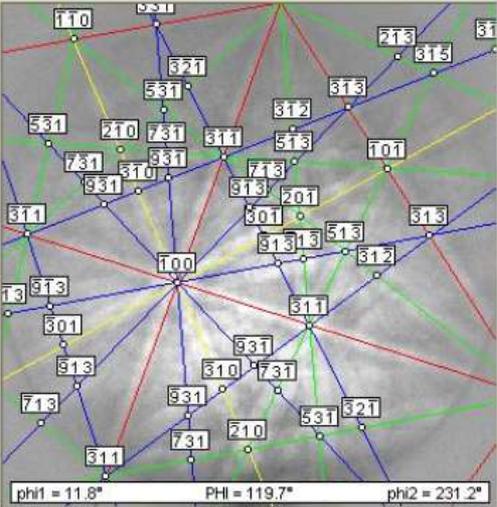
SED [Icons] 16" [XY]

Indexing | Scan | Interactive | Hough | Phase | Simulation | Grain Size | Video



Capture
Load
Save
Save As

Overlay
Color █
Data



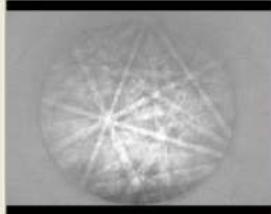
Index
Hide
Record

fit = 1.25
CI = 0.61

phi1 = 11.8° PHI = 119.7° phi2 = 231.2°

Instrument Console

Camera



5.4 fps Full View

Presets
 A B C D

Camera | Image Processing

Binning
2x2 (320 x 240)

Gain
0.00

Black
-0.28

Exposure
185.62

Exp Range
0.047 -> 1000 (Ms)

Auto Contrast

Snapshot Avg 5

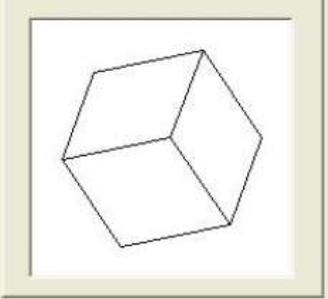
Camera Control

Analysis

Show	phi1	PHI	phi2	IQ	CI	fit	x	y	phase
1	11.8	119.7	231.2	23...	0.607	1....	-7.73	-5.27	Ferrite

Misorientation (rotation about [uvw]):

PF IPF Unit Cell



(-1 -1 0) NUM

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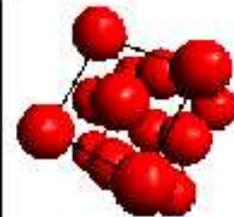
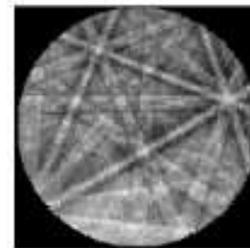
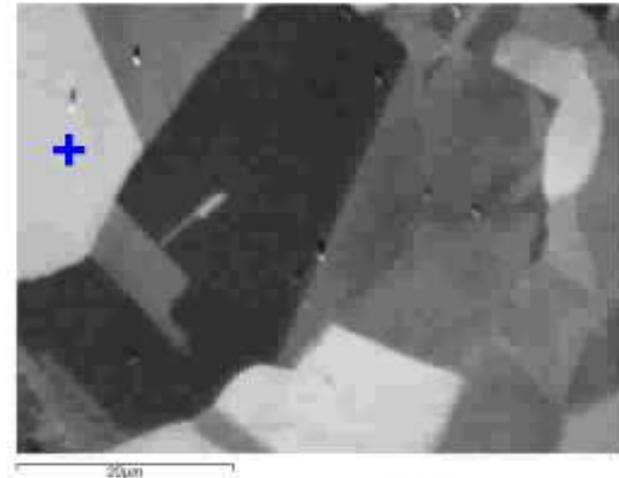
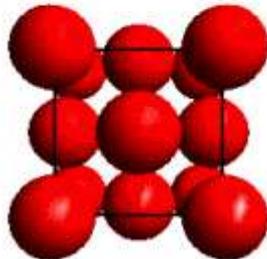
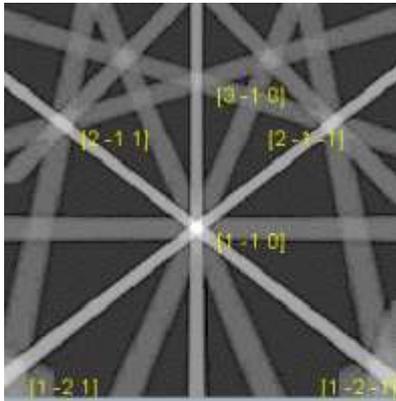
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What can it be used for? I.

Materials testing technique in SEM:

surface test of bulk polycrystalline (clear and smooth sample)

- Determining the orientation of grains.
- Examination of the distribution of grains over the surface of the whole sample.



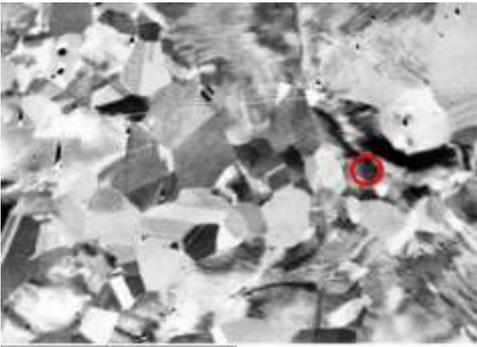
Texture: $\{3-17\}\langle-493\rangle$

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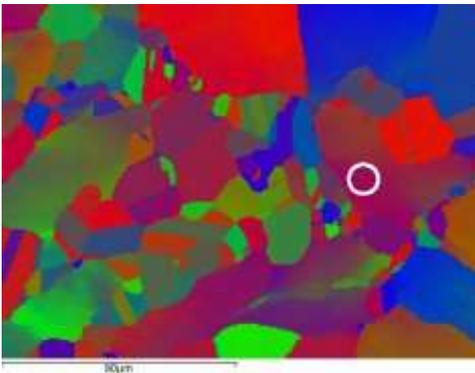
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What can it be used for? II.

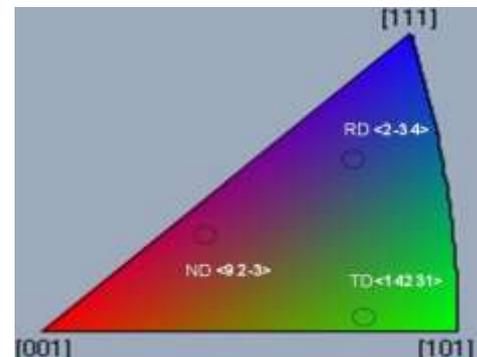
- local texture;
- point-to-point orientation change / correlation;
- phase identification and distribution determination;
- quantitative structure analysis;
- 3D structure discovery (with FIB);
- ~ 50 nm spatial resolution



SE image of rolled steel sample



and grain orientation map



inverse pole figure

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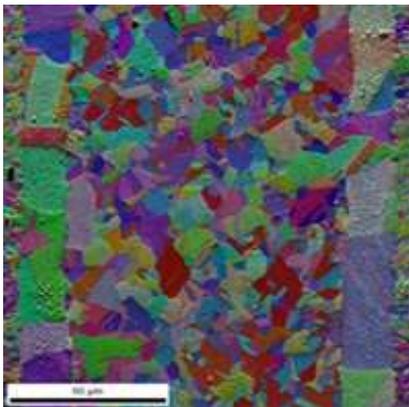
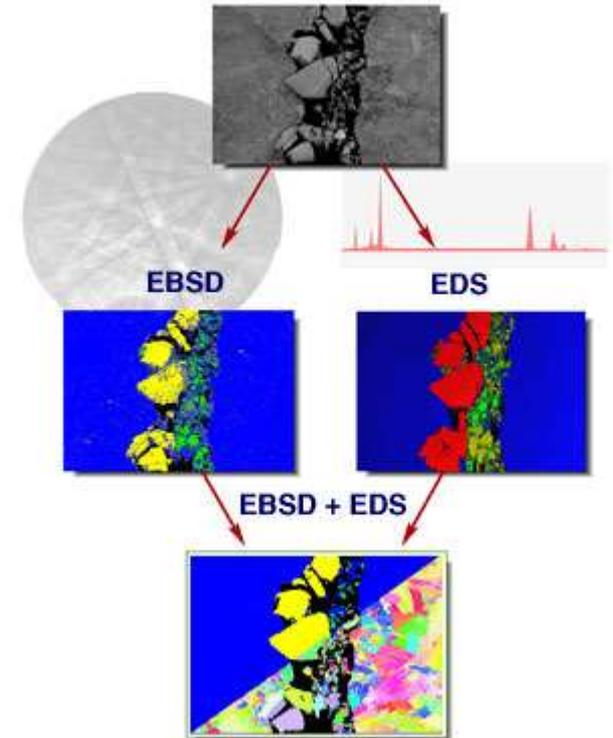
If indexing is not sufficient

e.g. same phase but different composition

Parallel use of EDS detector:

- recording X-ray intensity of selected elements simultaneously with EBSD data acquisition
- in the case of the same crystal structure, the particle can be determined by its composition

copper-nickel two-phase cubic system (austenite / ferrite)



Orientation map

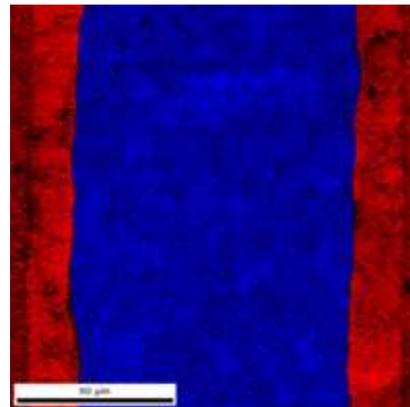


Image Quality and phase map