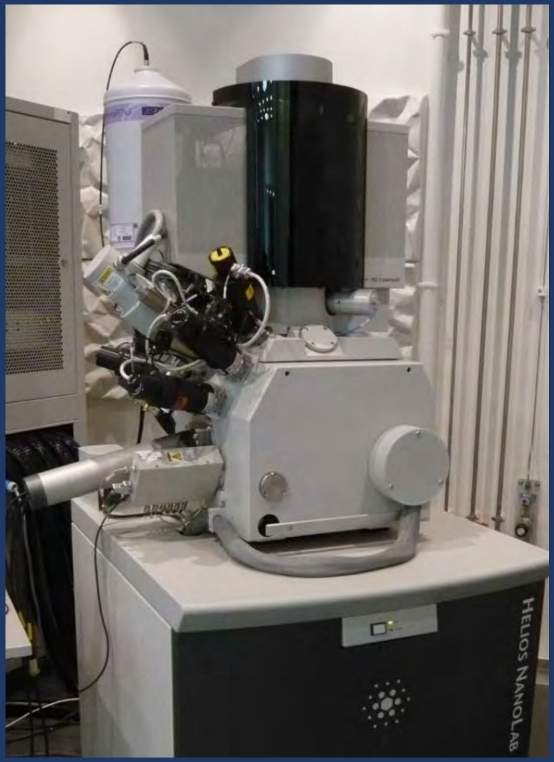
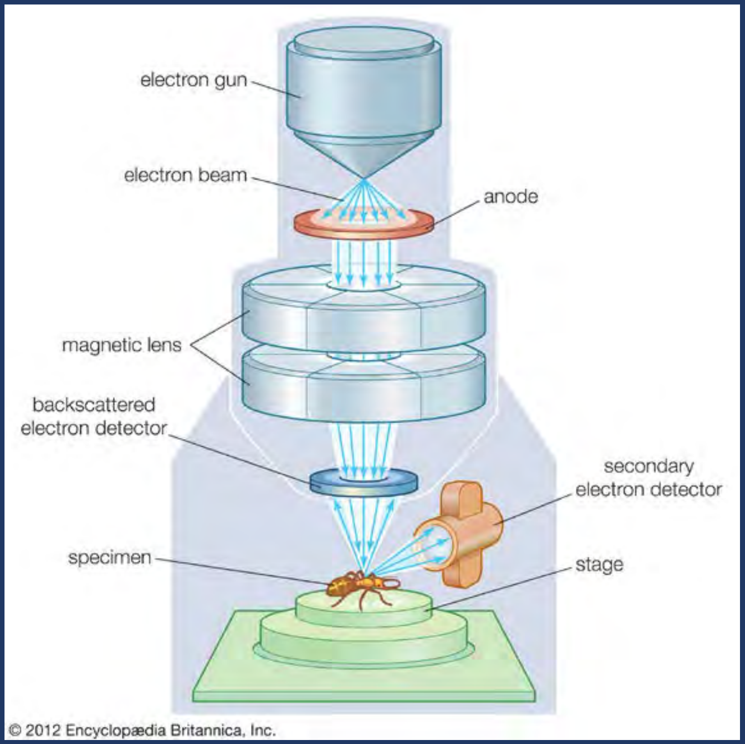
Diffraction techniques in the Scanning Electron Microscope (SEM)

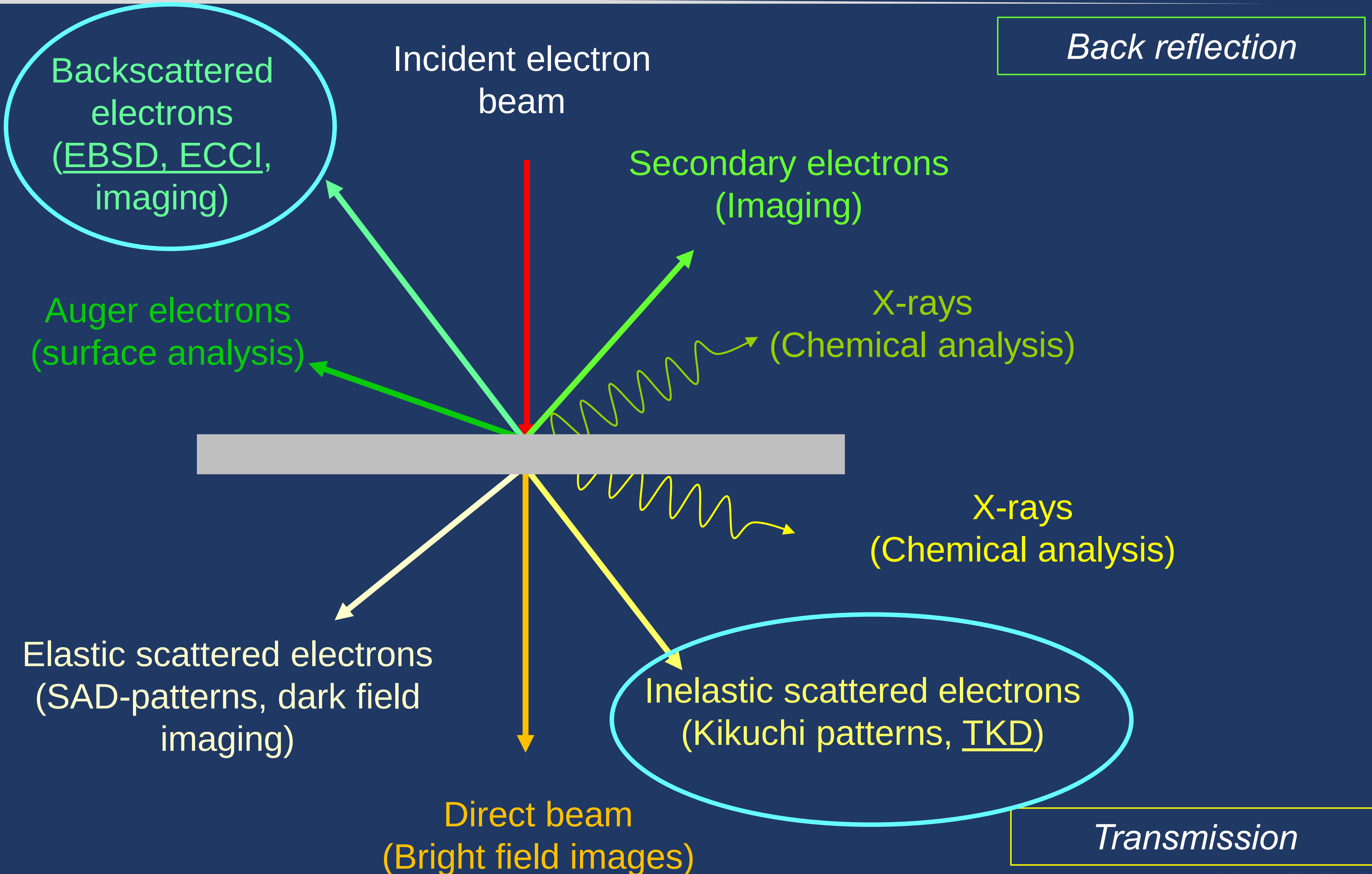
Alice Bastos da Silva Fanta
DTU-Nanolab

Scanning electron microscopy (SEM)





Scattering and diffraction

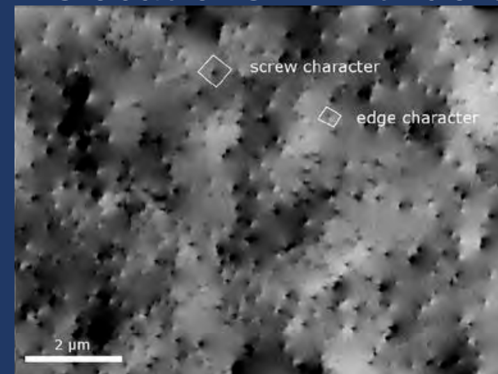


Electron Diffraction Techniques in the SEM

1. **ECCI –Electron channelling contrast images**

   Observation of crystal defects (dislocation, stacking faults and grain boundaries)

Dislocations in nitride thin films

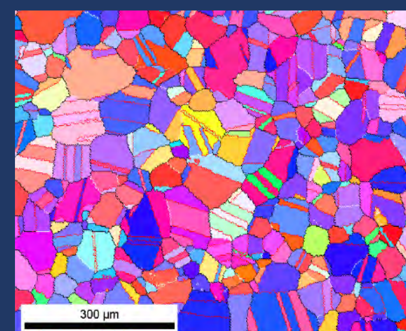


C. Trager-Cowan: http://ssd.phys.strath.ac.uk/index.php/Electron_channeling_contrast_imaging

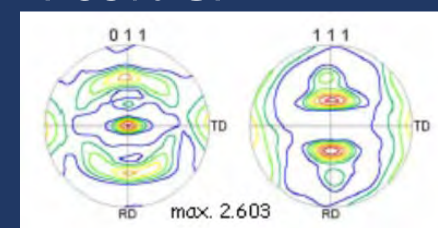
2. EBSD - Electron Backscatter Diffraction

   Microstructural – crystallographic characterization technique for <u>bulk samples</u>

TWIP steel

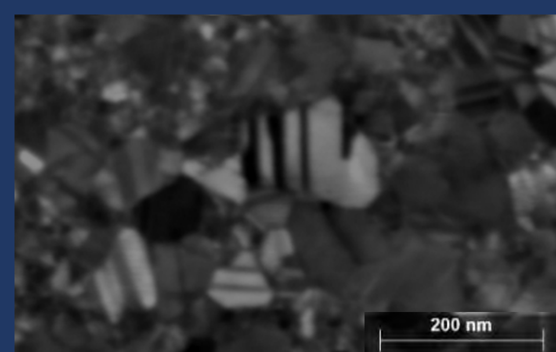


Fe3% Si



3. TKD - Transmission Kikuchi Diffraction

   Microstructural – crystallographic characterization technique for <u>thin samples</u>



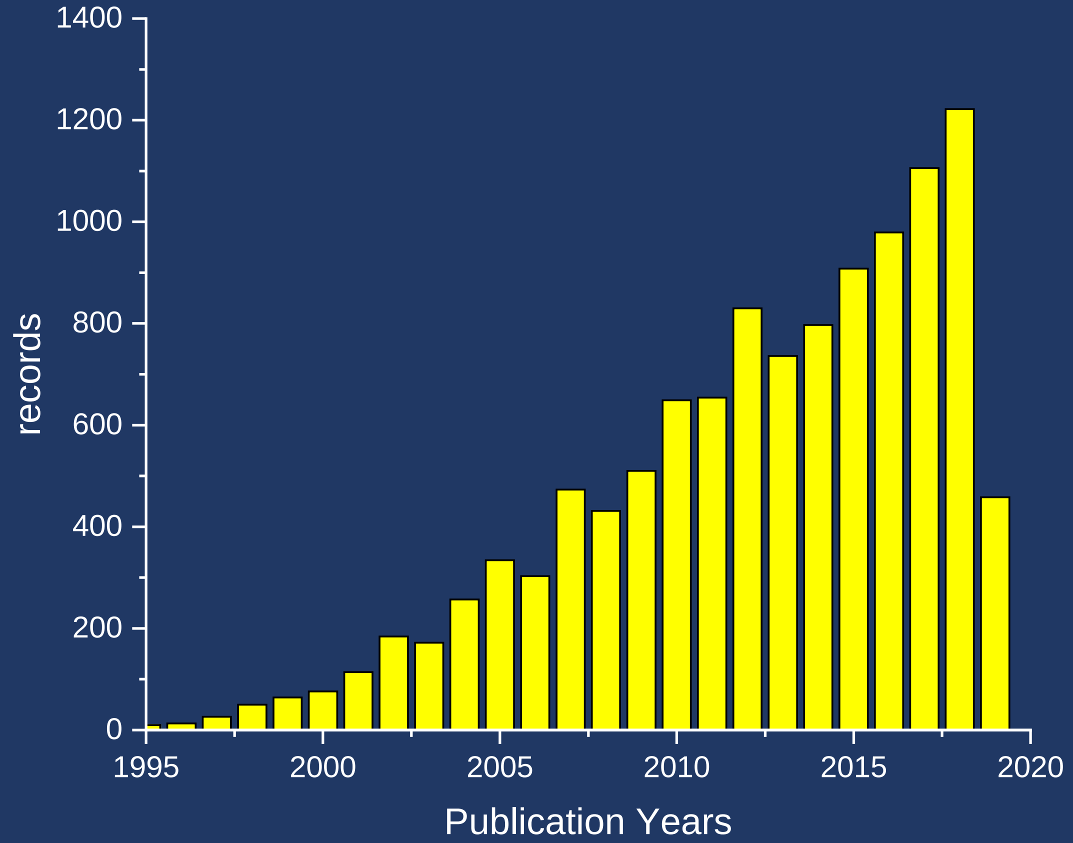
50 nm

Nordic Nanolab Network

EBSD
Electron Backscatter Diffraction

Electron backscatter diffraction - history

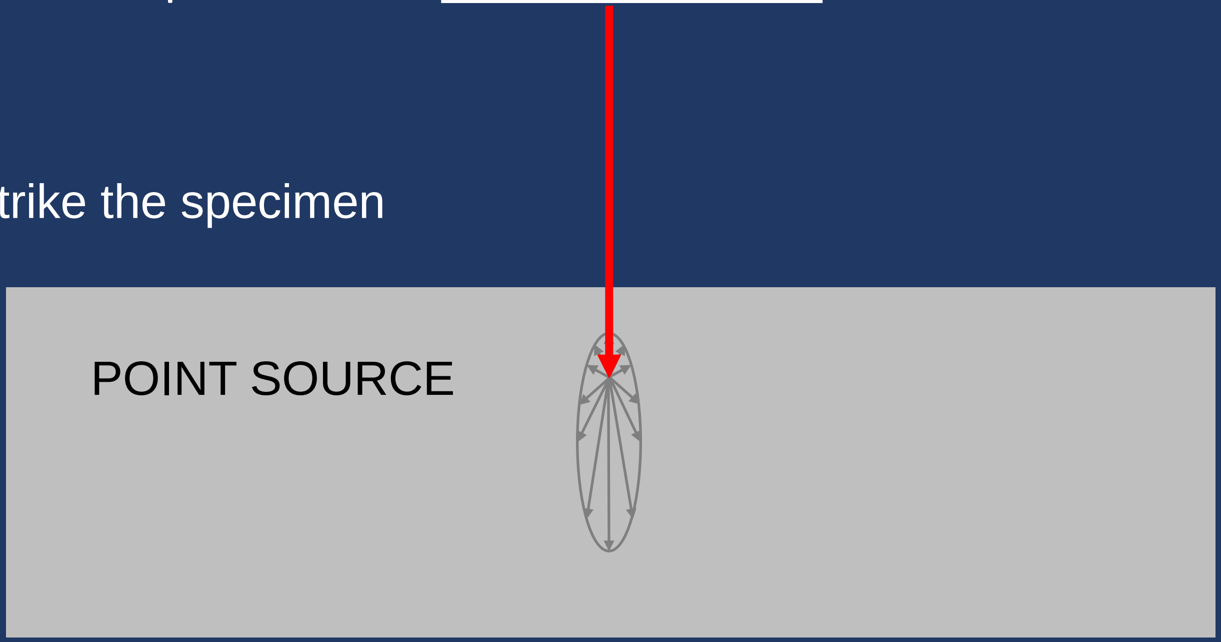
- 1928 - First observation of BKP by Nishikawa& Kikuchi
- 1973 - Observation of BKP in a SEM by Venables and Harland
- 1984 - Dingley started using TV camera and computer software for orientation determination
- 1992- Introduction of the Hough transform by Krieger Lassen et al.
- 1993 - Introduction of OIM (Orientation image microscopy) by Adams et al.



Formation of Kikuchi pattern – step 1

The formation of EBSD patterns is a two-step process

- Electrons strike the specimen



1. They are then inelastic scattered from the point source in all directions

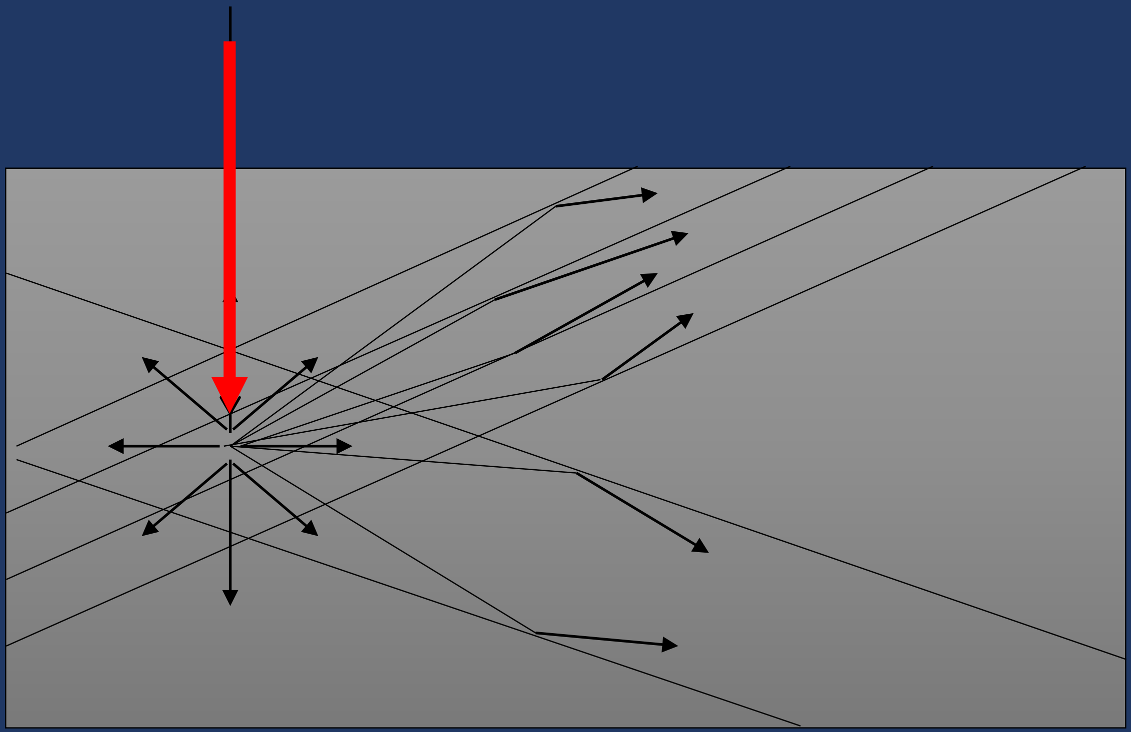
Inelastic: some loss of energy

Formation of Kikuchi pattern – step 2

2. *crystalline materials: those* electrons (from inside the point source) are diffracted by the crystal lattice planes when the Bragg condition is satisfied

Bragg equation: $n\lambda = 2d \sin\theta$

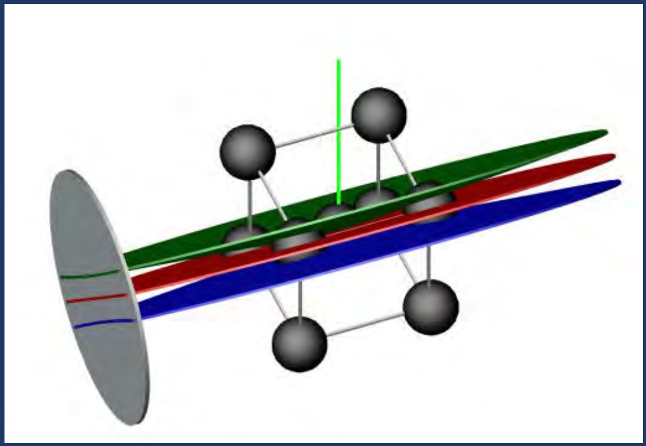
λ: wavelength of the electrons
d : spacing of the crystal planes
n: is an interger
θ : angle of incidence



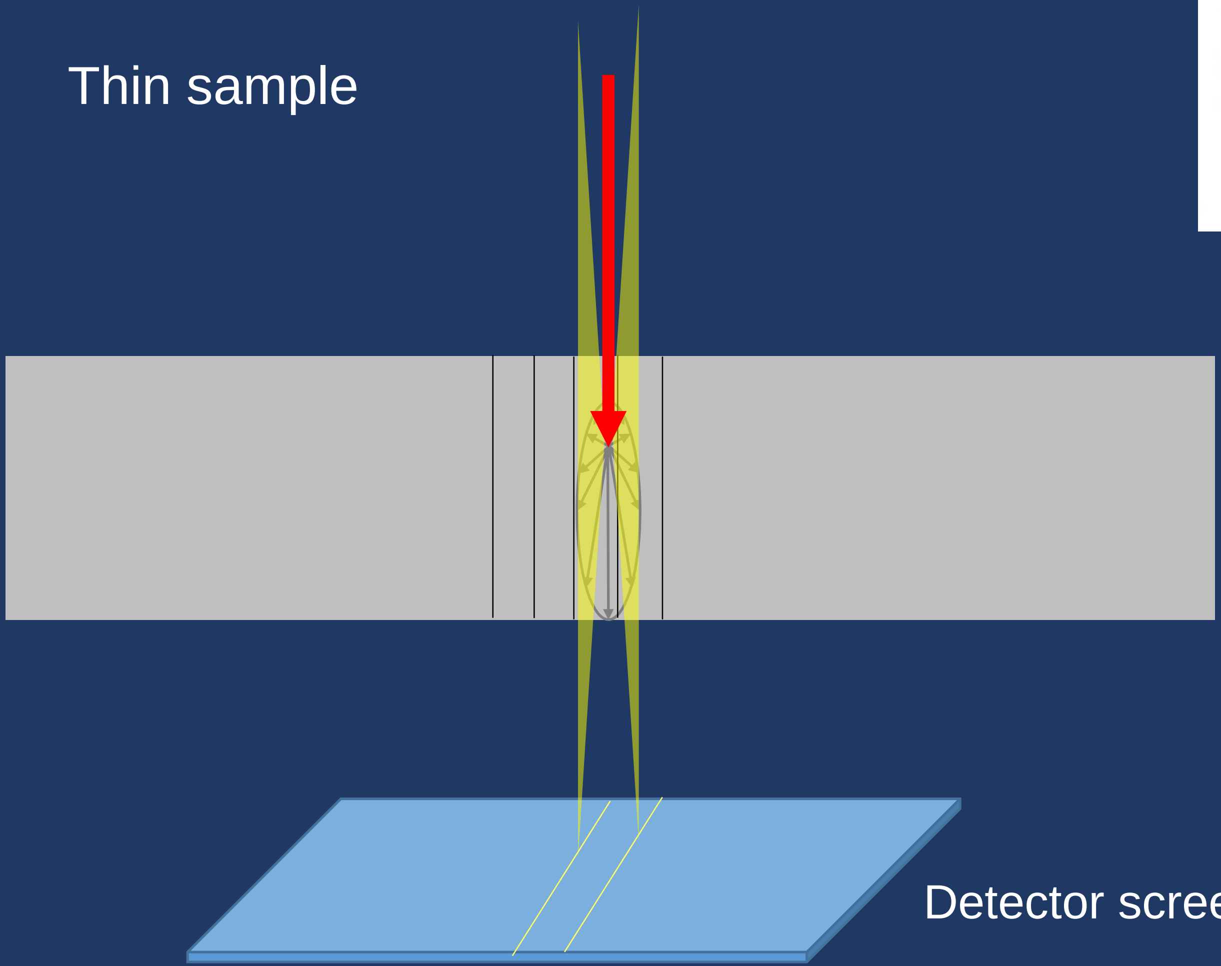
2 lattice plane

Simplified illustration – one electron and one lattice plane

- Since the scattered electrons are travelling in all direction, the diffracted beam will lie on one of two cones.



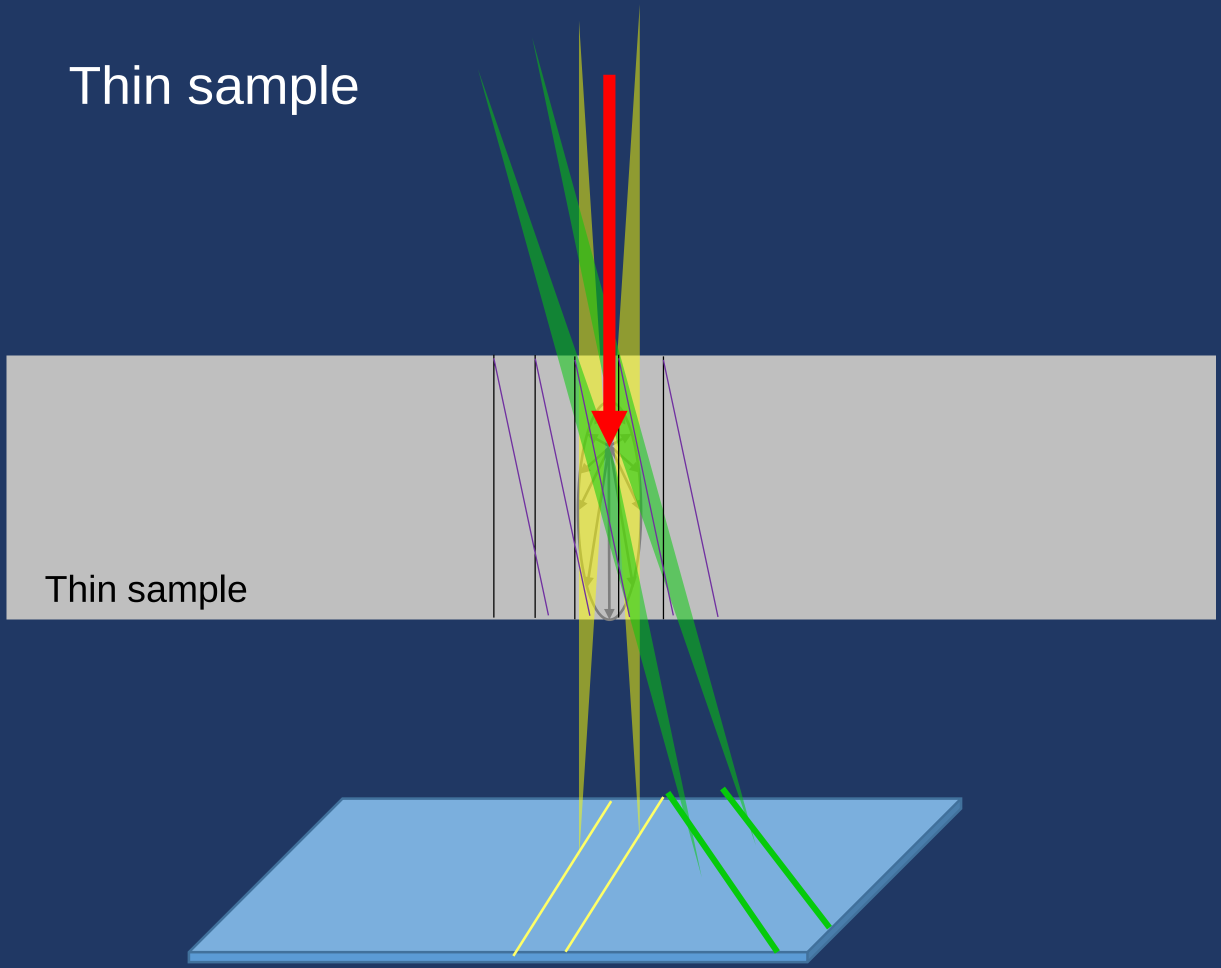
Oxford instrument



Adapted from:
Ref.:http://ssd.phys.strath.ac.uk/index.php/Electron_backscatter_diffraction_(EBSD)

Two electron and two lattice plane

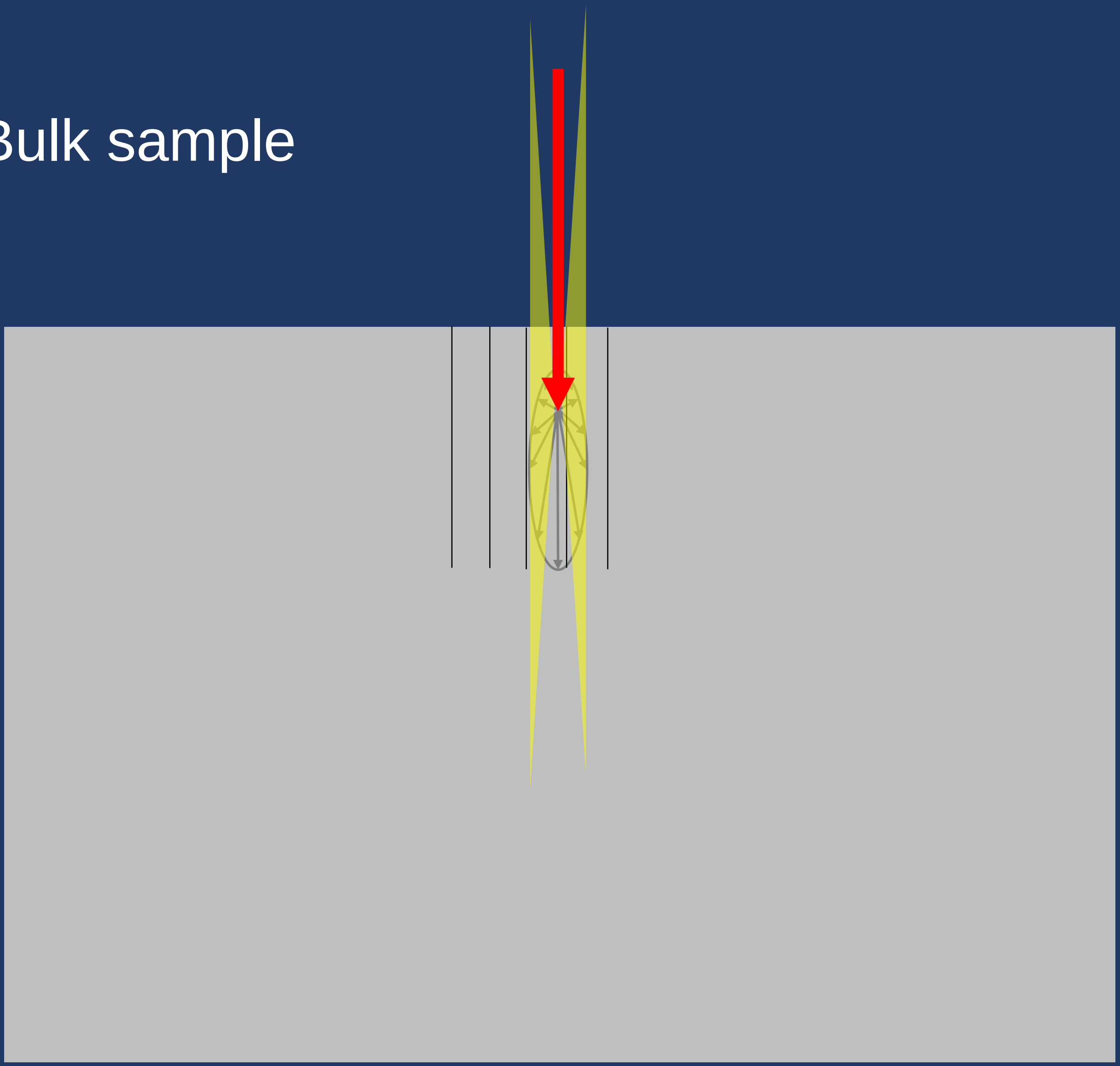
Thin sample



Adapted from:
Ref.:http://ssd.phys.strath.ac.uk/index.php/Electron_backscatter_diffraction_(EBSD)

Simplified illustration – one electron and one lattice plane

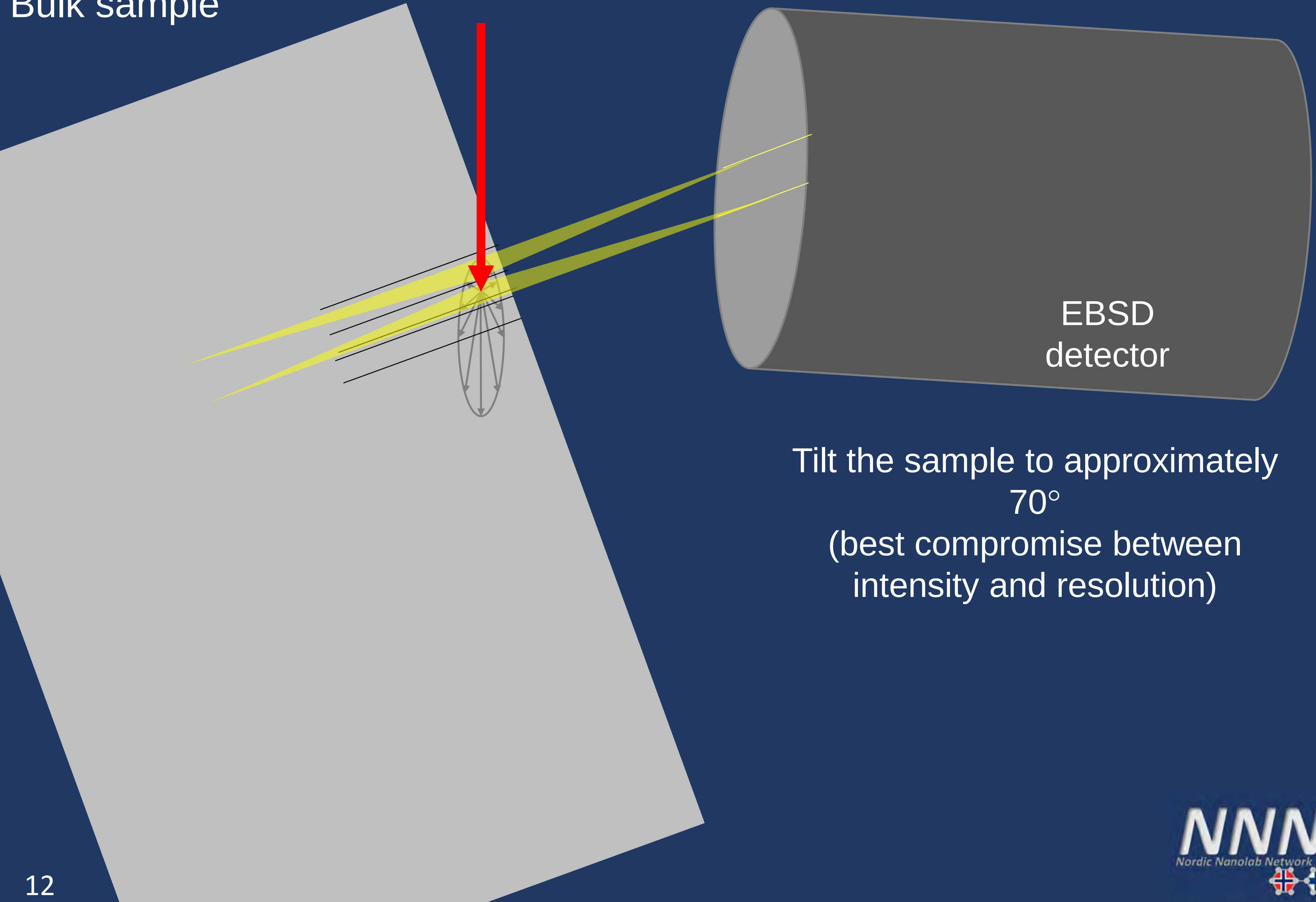
Bulk sample



Adapted from:
Ref.:http://ssd.phys.strath.ac.uk/index.php/Electron_backscatter_diffraction_(EBSD)

EBSD – tilted sample

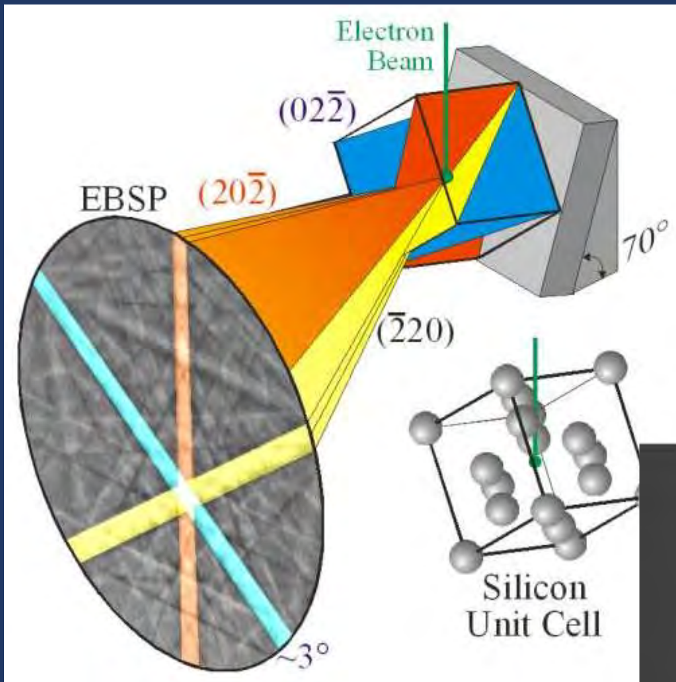
Bulk sample



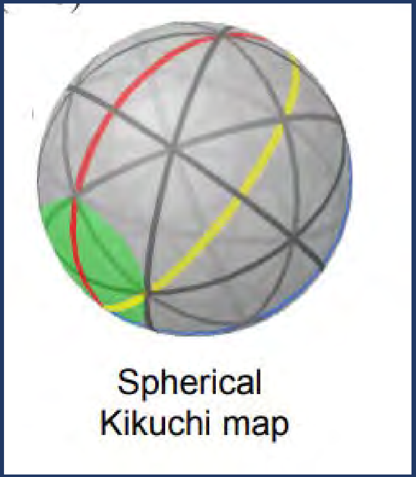
Tilt the sample to approximately 70°
(best compromise between intensity and resolution)

EBSD – tilted sample

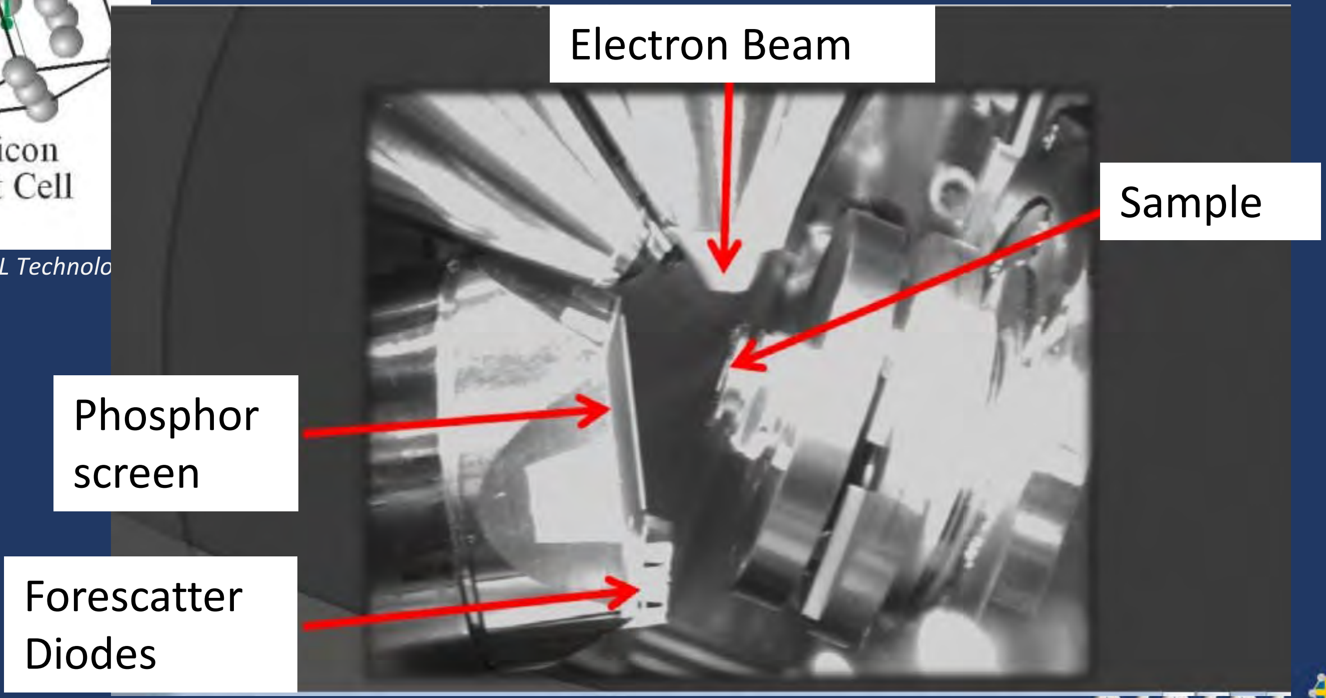
Scattering from single lattice planes



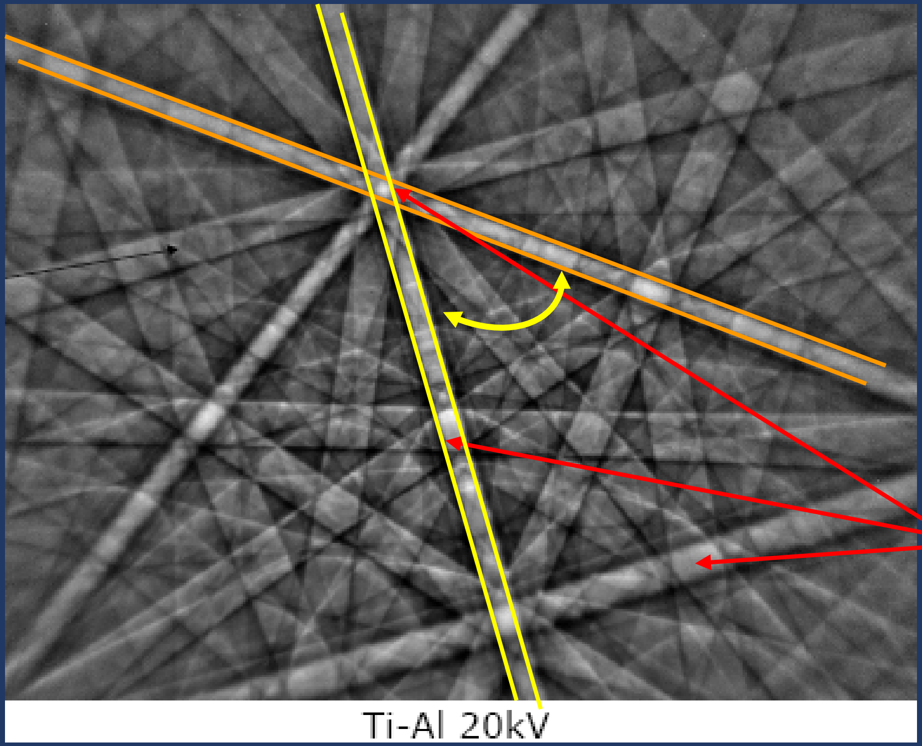
Ref.: A. P. Day et al., *Channel 5 User Manual, HKL Technolo A/S, Hobro, Denmark (2001).*



View from the SEM



EBSD patterns



Ref.: Dr. Emmanuelle Boehm Courjault; Introduction to EBSD (Electron BackScatter Diffraction) :Principle and Applications

Position of bands <u>directly linked</u> to the crystallographic orientation

Diffraction from a specific lattice plane

Intersections of bands = intersections of planes = zone axes

Angles between bands = angles between planes

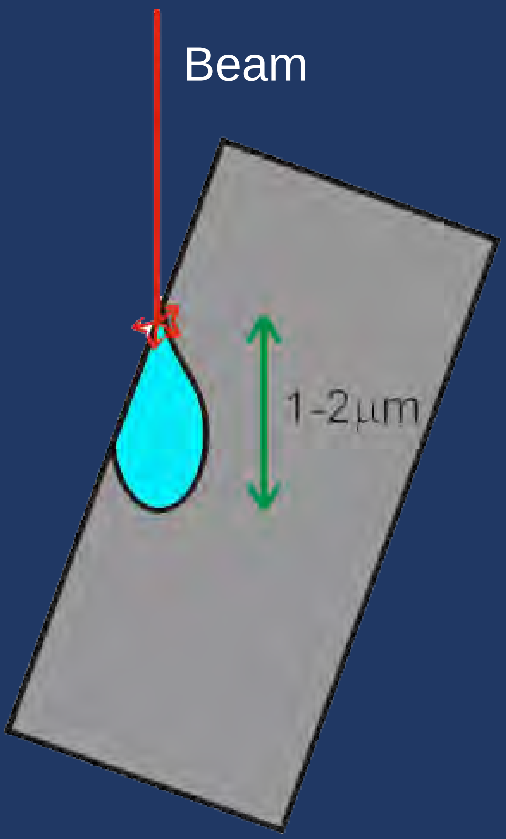
EBSD patterns are:

- *<u>unique</u>* for a specific crystal orientation
- is controlled by the crystal structure: space group symmetry, lattice parameters, *precise* composition

Surface sensitive technique

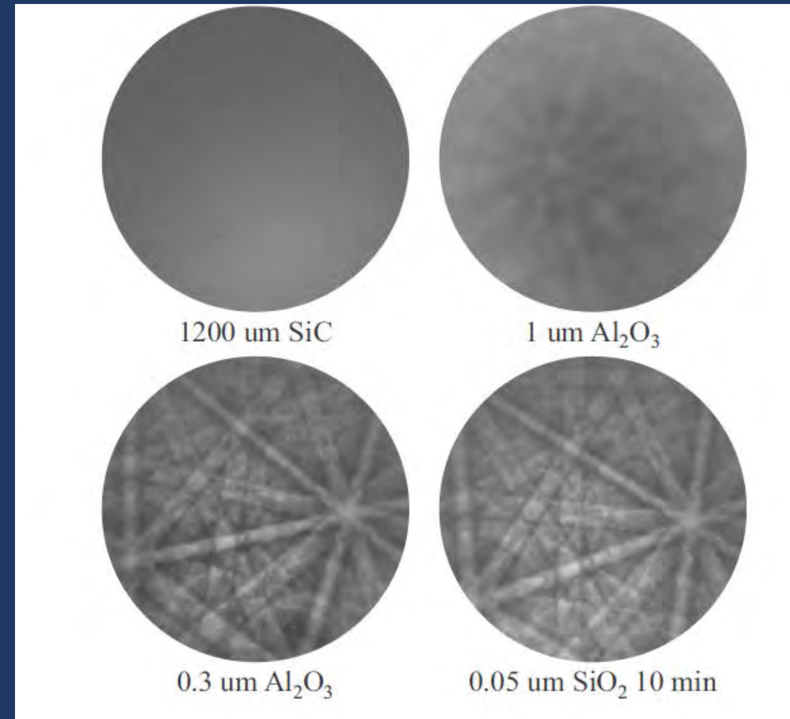
- Although EBSPs are created by **backscattered electrons**, the signal does not come from the whole BSE interaction volume

- Instead, the diffraction signal originates from a "POINT SOURCE" $\rightarrow$ 5-10nm under the surface



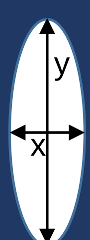
Surface sensitive technique

Surface sensitive technique

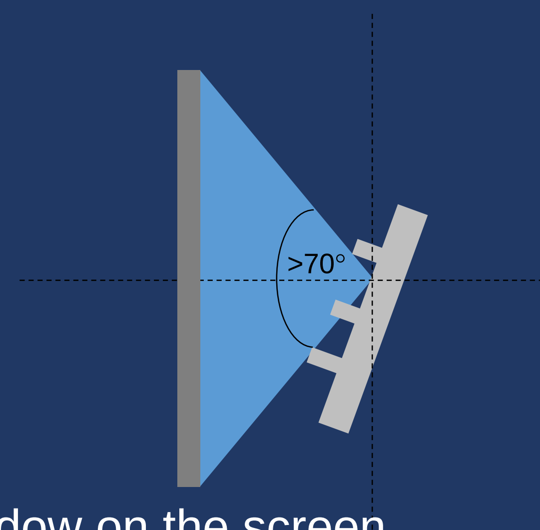


- The top layer
  - Free from damage
  - Free from contamination
  - or oxidation layers
  - in case of non conductive samples → the coat must be kept very thin – typically in the range of 2-5 nm.

- Due to high tilt angles (typically 70°),
  - surface topography must be kept to an absolute minimum.

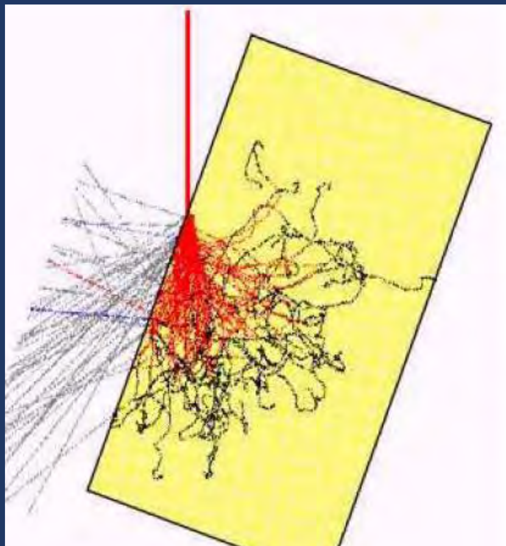


Sample strongly tilted –
Resolution y axis is 3x worst



Shadow on the screen

Signal intensity



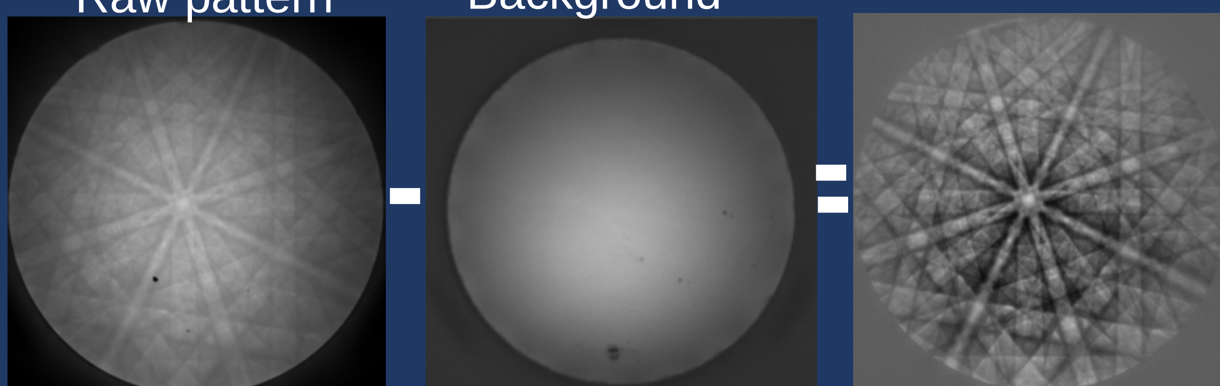
From: S. Baeck, TSL tutorial S. Wright

➡ Only a small fraction of the electron arriving at the phosphor screen are diffracted

Background intensity supresses both the contrast and sharpness.

Raw pattern

Background

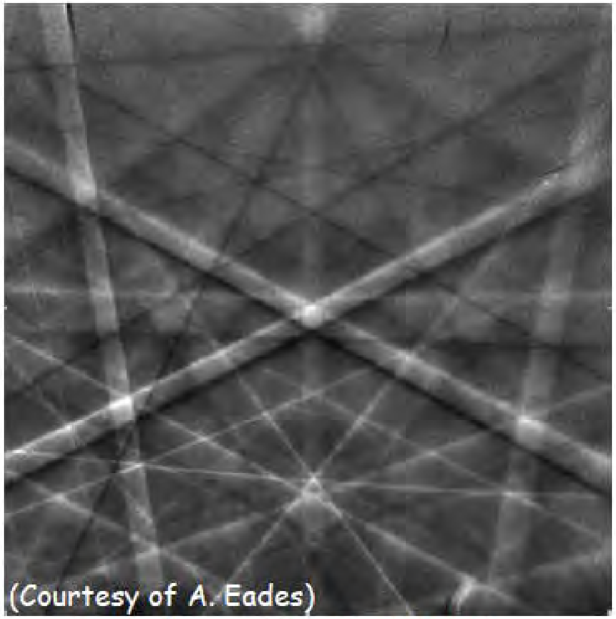


Nb-pattern from S. Zaefferer

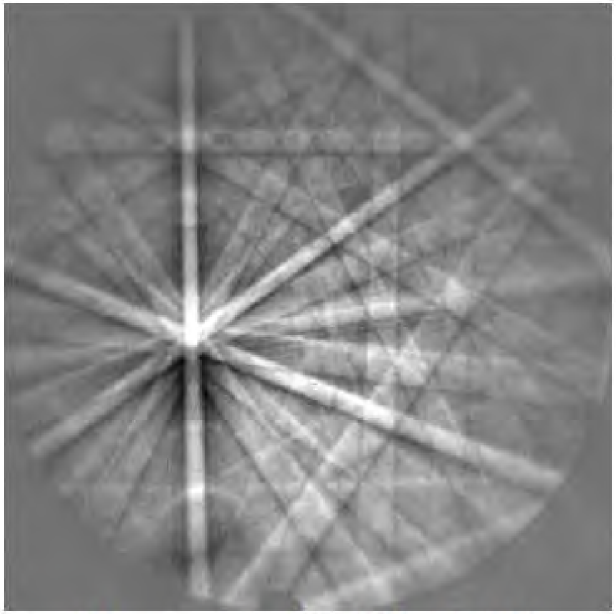
Signal is material dependent

Increasing atomic number (↑ Z):

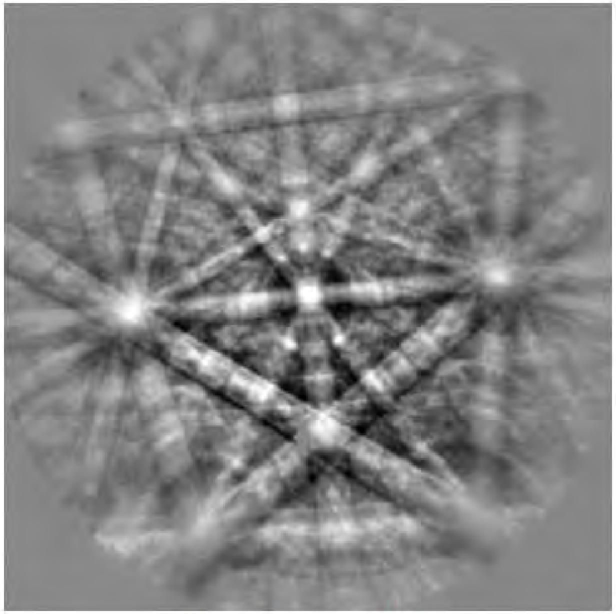
- Increase the amount of backscatter electrons – Pattern quality
- Decreases the interaction volume - improve spatial resolution



Beryllium (Z=4), 30 kV
exposure time ~ 15 s



Magnesium (Z=12), 15 kV
exposure time ~ 6 s



TiCr Laves phase
(Z=22...24), 15 kV

From S. Zaefferer

Short take home message

- It is not easy to prepare the sample for EBSD
- Once you get patterns, it runs fully automated

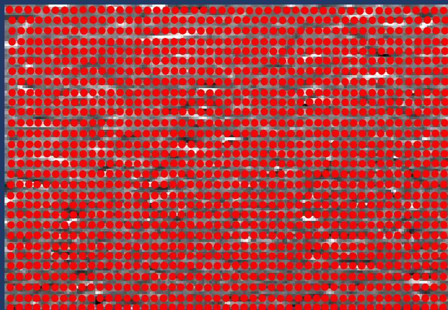
Automate data acquisition, pattern indexing and orientation determination

In one automate run you get:

- Grain size,
- texture,
- grain boundary distribution,
- phase distribution,
- …

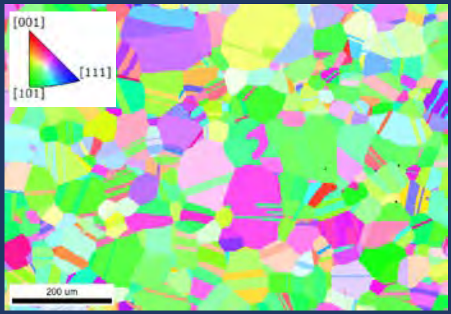
Nordic Nanolab Network

Choose the step size and the area

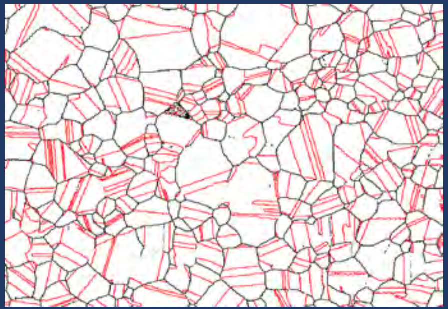


*Runs very fast: Now a days up to **3000** patterns per seconds*

Automated indexing during the acquisition



Orientation map



Grain boundary map (misorientation)

```
#Phase0:
#  Name: Austenite, fcc (New)
#  Spacegroup: F m#ovl3m
#  A: 3.66
#  B: 3.66
#  C: 3.66
#  Alpha: 9E1
#  Beta: 9E1
#  Gamma: 9E1
#Orientations:
#Index  Phase  x(Px)  y(Px)  x(µm)  y(µm)  phi1  PHI  phi2  Bands  BC  GrainIndex
0  0  0  0  0  0  0  0  0  0  0  -1
1  0  1  0  -2.717E-1  0  0  0  0  0  0  -1
2  0  2  0  -5.434E-1  0  0  0  0  0  0  -1
3  0  3  0  -8.151E-1  0  0  0  0  0  0  -1
4  0  4  0  -1.0868 0  0  0  0  0  0  -1
5  0  5  0  -1.3585 0  0  0  0  0  0  -1
6  0  6  0  -1.6302 0  0  0  0  0  0  -1
7  0  7  0  -1.9019 0  0  0  0  0  0  -1
8  0  8  0  -2.1736 0  0  0  0  0  0  -1
9  0  9  0  -2.4453 0  0  0  0  0  0  -1
10  0  10  0  -2.717  0  0  0  0  0  0  -1
11  0  11  0  -2.9887 0  0  0  0  0  0  -1
12  0  12  0  -3.2604 0  0  0  0  0  0  -1
13  0  13  0  -3.5321 0  0  0  0  0  0  -1
14  0  14  0  -3.8038 0  0  0  0  0  0  -1
15  0  15  0  -4.0755 0  0  0  0  0  0  -1
16  0  16  0  -4.3472 0  0  0  0  0  0  -1
17  0  17  0  -4.6189 0  0  0  0  0  0  -1
18  0  18  0  -4.8906 0  0  0  0  0  0  -1
19  0  19  0  -5.1623 0  0  0  0  0  0  -1
20  0  20  0  -5.434  0  0  0  0  0  0  -1
21  0  21  0  -5.7057 0  0  0  0  0  0  -1
22  0  22  0  -5.9774 0  0  0  0  0  0  -1
23  0  23  0  -6.2491 0  0  0  0  0  0  -1
24  0  24  0  -6.5208 0  0  0  0  0  0  -1
25  0  25  0  -6.7925 0  0  0  0  0  0  -1
26  0  26  0  -7.0642 0  0  0  0  0  0  -1
27  0  27  0  -7.3359 0  0  0  0  0  0  -1
28  0  28  0  -7.6076 0  0  0  0  0  0  -1
29  0  29  0  -7.8793 0  0  0  0  0  0  -1
30  0  30  0  -8.151  0  0  0  0  0  0  -1
31  0  31  0  -8.4227 0  0  0  0  0  0  -1
32  0  32  0  -8.6944 0  0  0  0  0  0  -1
33  0  33  0  -8.9661 0  0  0  0  0  0  -1
34  0  34  0  -9.2378 0  0  0  0  0  0  -1
35  0  35  0  -9.5095 0  0  0  0  0  0  -1
36  0  36  0  -9.7812 0  0  0  0  0  0  -1
37  0  37  0  -1.00529E1  0  0  0  0  0  0  -1
38  0  38  0  -1.03246E1  0  0  0  0  0  0  -1
39  0  39  0  -1.05963E1  0  0  0  0  0  0  -1
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41  0  41  0  -1.11397E1  0  0  0  0  0  0  -1
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44  0  44  0  -1.19548E1  0  0  0  0  0  0  -1
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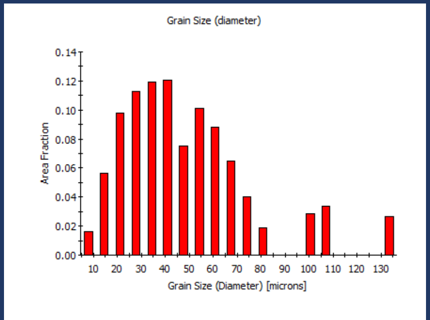
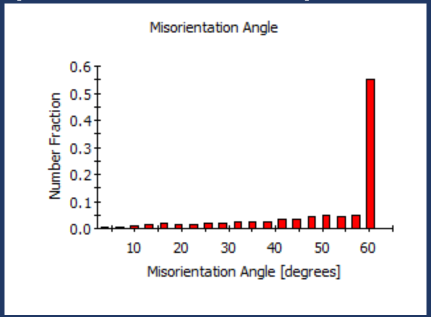


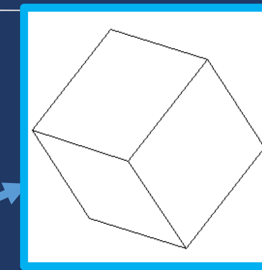
Grains size distribution



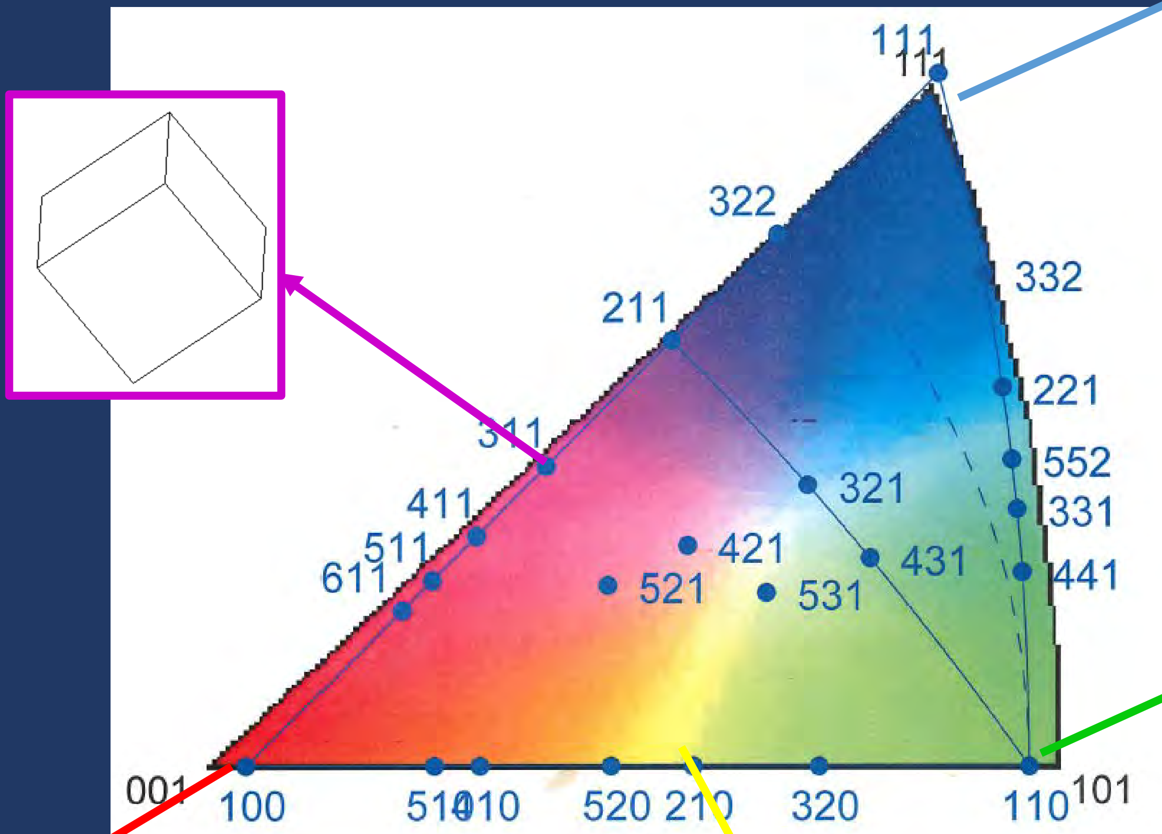
Misorientation angles

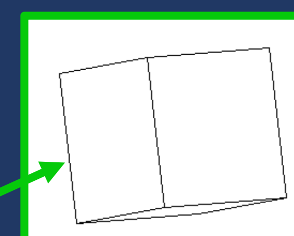
Nordic Nanolab Network

Colourful image

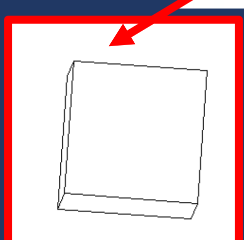


111 plane // to the surface

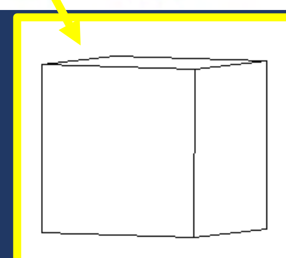




110 plane // to the surface

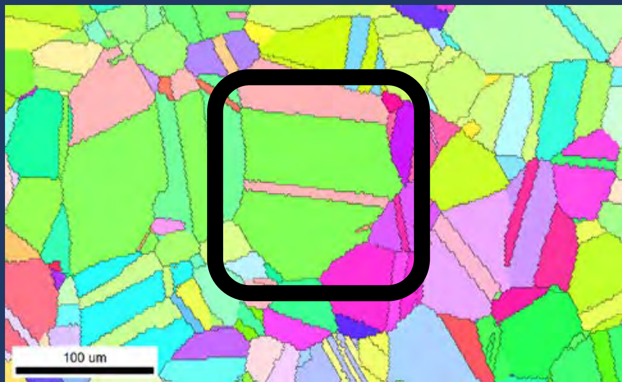


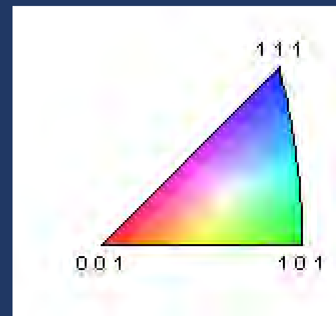
100 plane // to the surface



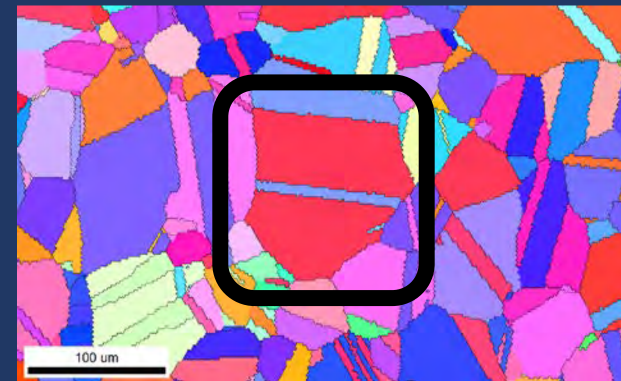
Orientation with respect to the sample coordinate system

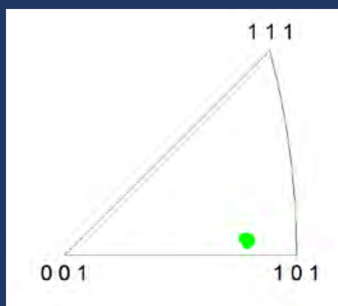
Z-direction

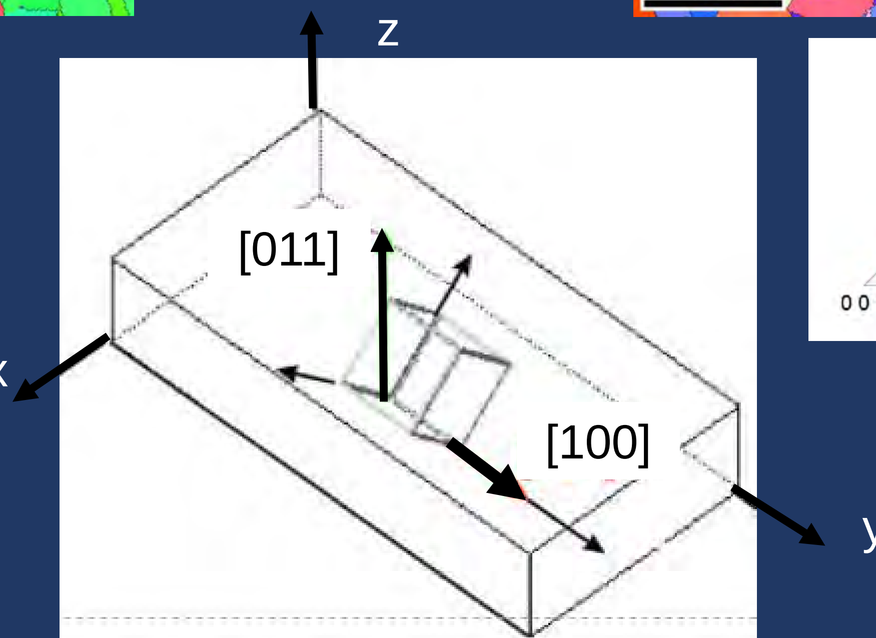


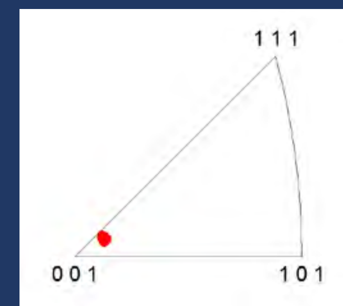


Y-direction



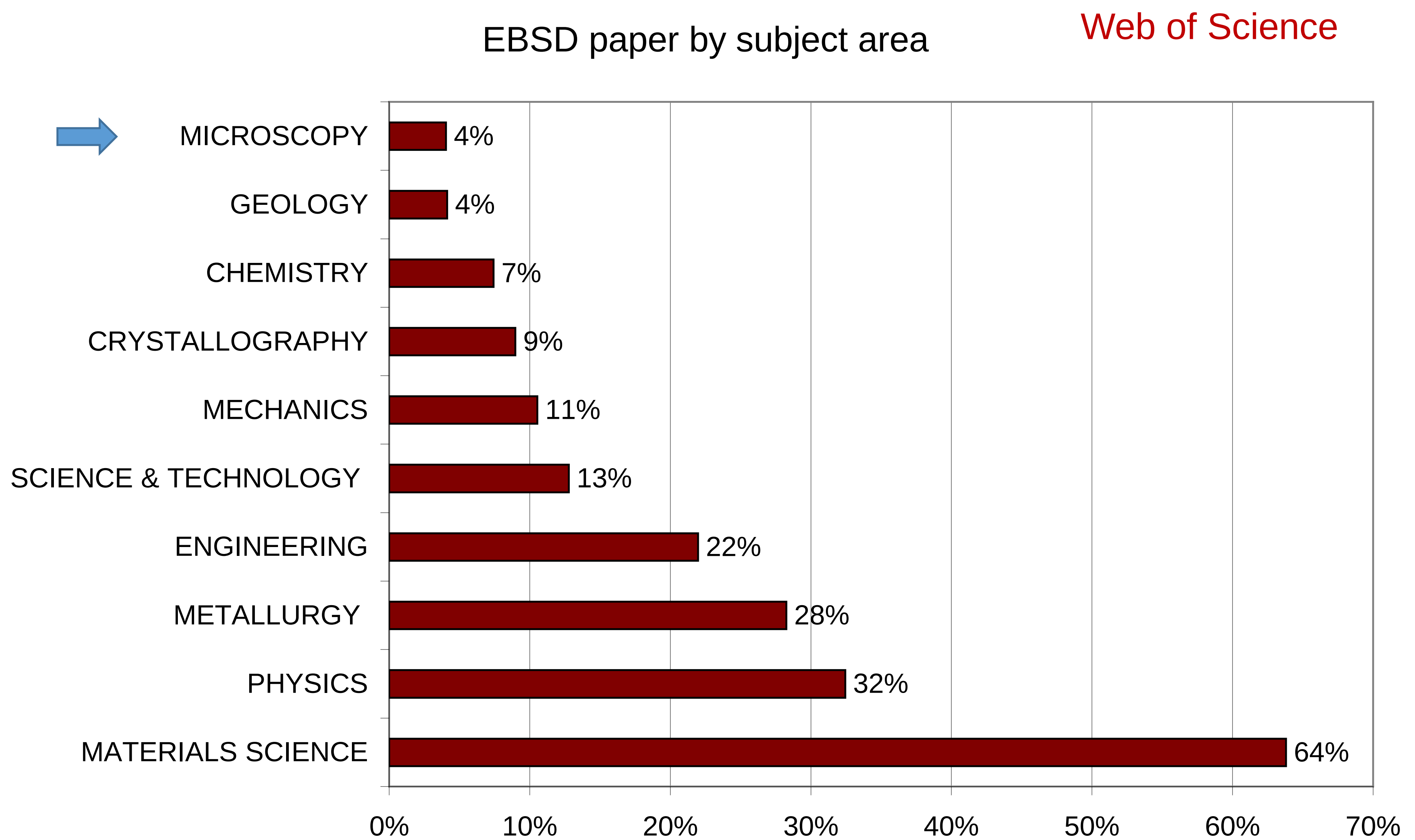






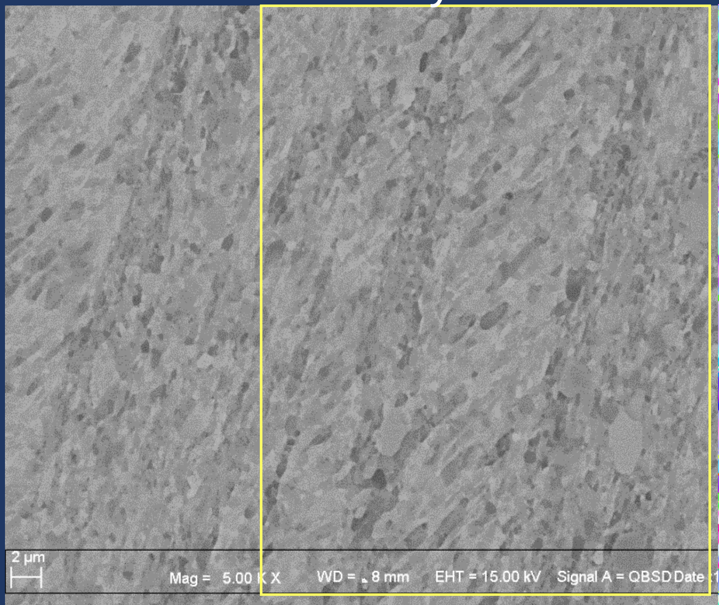
Adapted from: *Channel 5 User Manual, HKL Technology A/S, Hobro, Denmark (2001).*

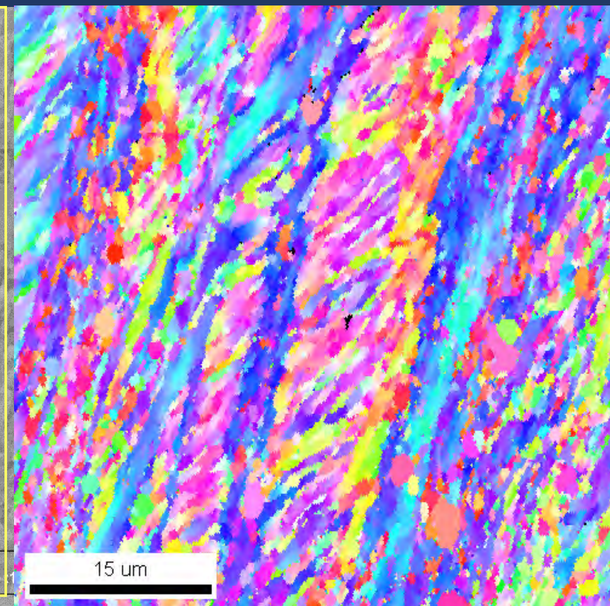
EBSD is a materials characterization tool

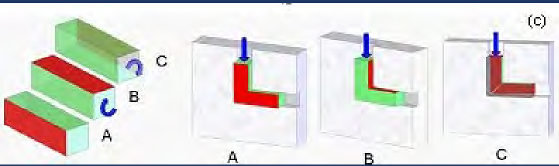


Application example -1

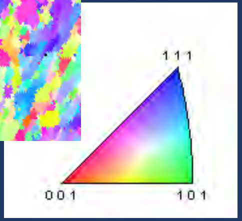
Niobium deformed by ECAP and heat treated for 15 min



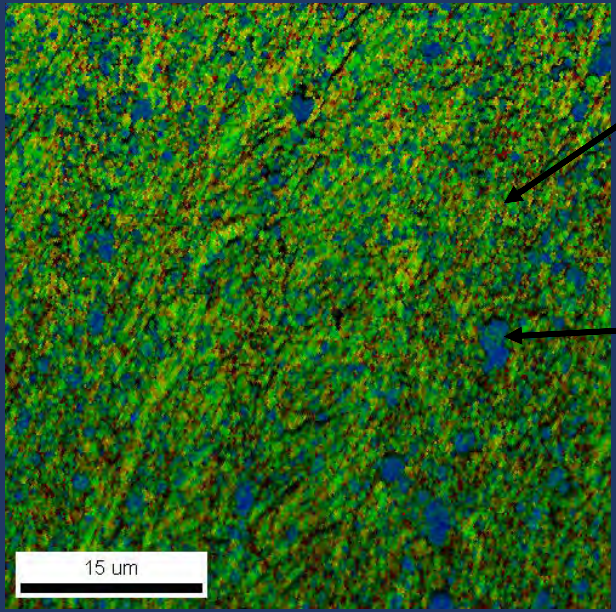




Orientation bands – related to the type of deformation - **shear**

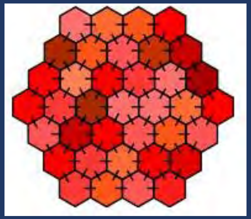


Local misorientation map



Deformed areas

Recrystallized areas



	Min	Max	Total Fraction	Partition Fraction
	0	3	0.999	0.999

H. Sandim; www.imim.pl