EBSD

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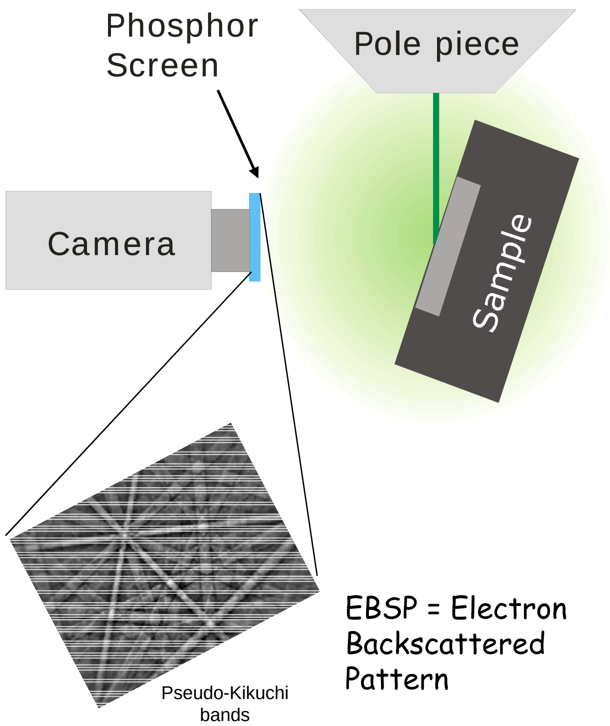
EBSD Basics

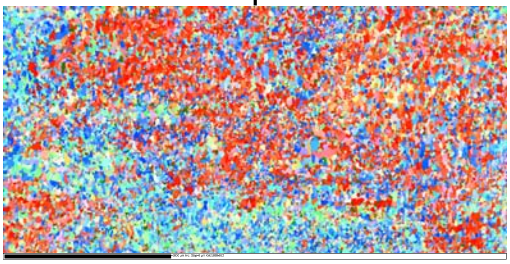
- Quantitative, general microstructural characterization in the SEM based on diffraction effects !
- Orientation measurements, phase identification
- Near-surface technique
  - Using diffraction patterns originating 20 nm – 100 nm below the surface
  - Surface preparation is critical
- Materials analyzed
  - Crystalline materials
  - Metals, ceramics, minerals
  - Conductors (and insulators)



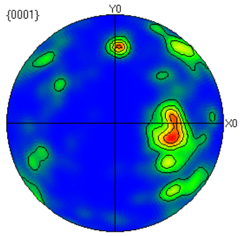
EBSP = Electron Backscattered Pattern

Uses of EBSD

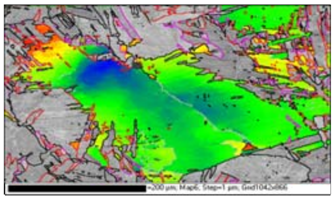
Maps

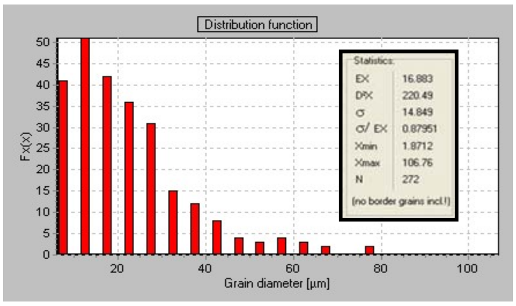


Pole figures
(orientation analysis)



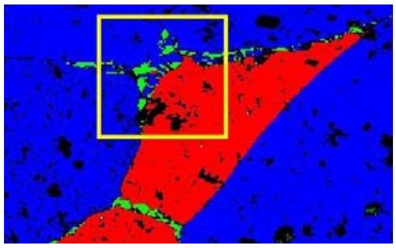
Strain analysis



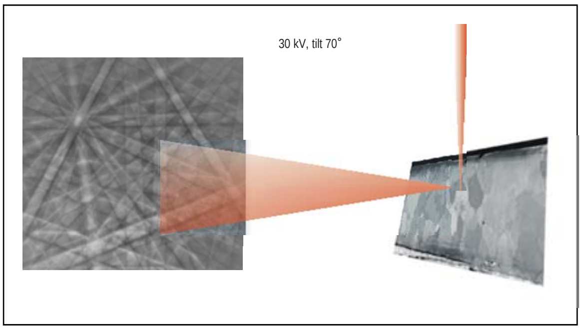


Quantitative
microstructural data

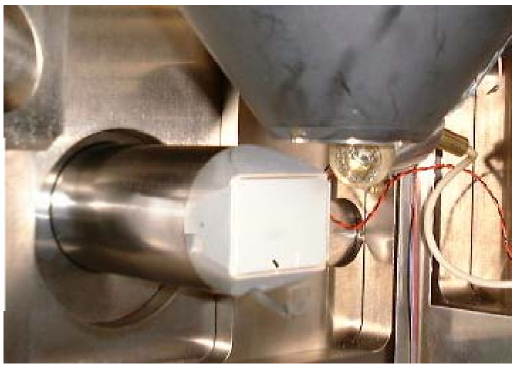
Phase identification



How EBSPs are obtained ?



Tilt 70° - Spot mode - 30 kV - 10 nA



Phosphor screen + CCD camera

EBSD : electron diffraction in the SEM: 2 step process !

1) Electrons of the incident beam spread beneath the surface in all direction due to elastic interactions (*backscattered* electrons) :
small divergent source of electron behind (~ 100 nm) the sample surface.

2) These electrons are ***diffracted*** **by crystal planes** according to the Bragg condition.

Sample preparation

Requirements

The backscattering volume (100 nm) below the surface sample must be crystalline and without excessive plastic deformation.

Problems :
- → plastic deformation due to mechanical polishing
- → foreign layers (oxide)
- → internal strain

Preparations methods

✓ Mirror quality polishing (→ 0.25 µm diamond grade), **and** :

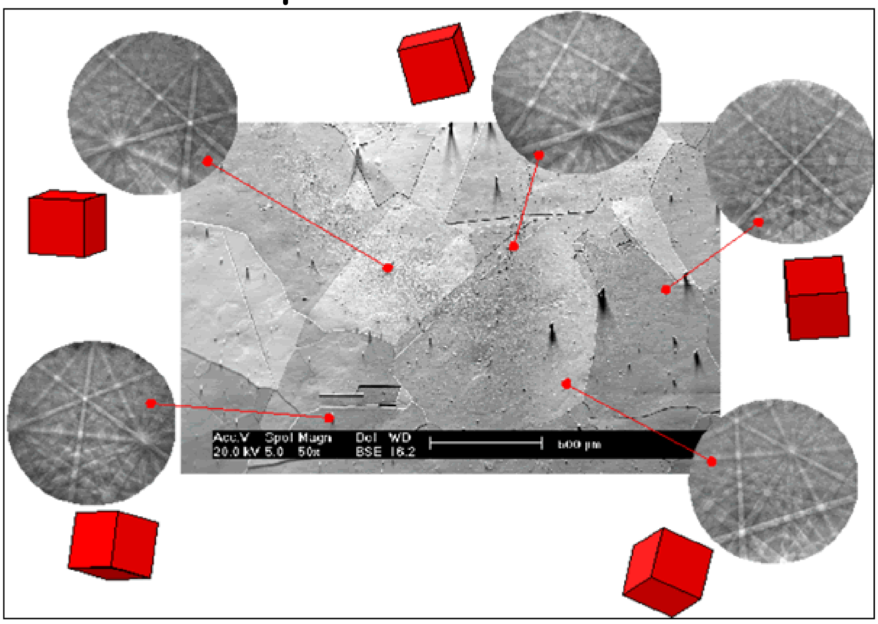
- → chemical-mechanical polishing (silica or alumina suspension)
- → electro-polishing or chemical polishing/etching
- → ion-milling or plasma etching

✓ cleaved surface, growth surface

✓ Insulating materials

- → carbon coating (< 100 Å) (degrade pattern quality)
- → low-vacuum SEM (a few Pa)

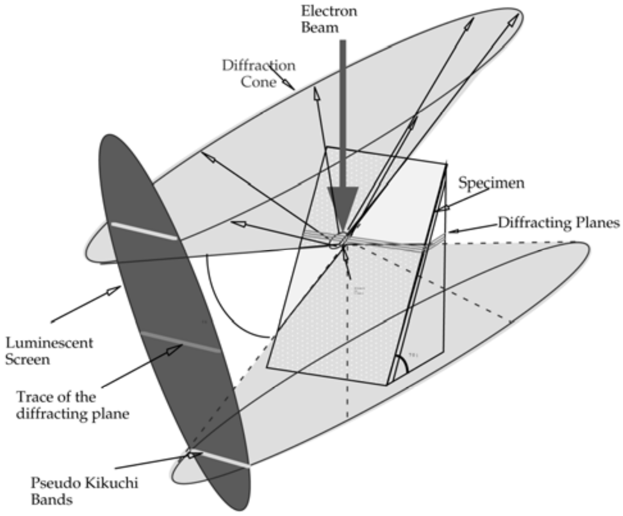
Acquisition of EBSPs

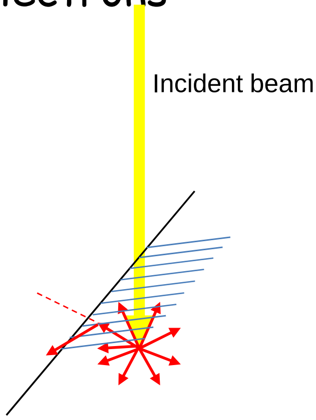


Positioning of beam on tilted surface and analyse the EBSD pattern !

EBSD consist of **Kikuchi bands** corresponding to the various diffracting planes. Intersections of these bands correspond to crystal zone axis.
The geometrical arrangement of Kikuchi bands depends of **crystal symmetry** and **crystal orientation**.

Diffraction of backscattered electrons

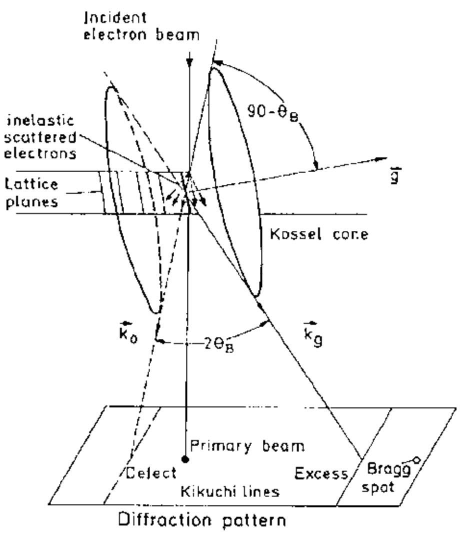




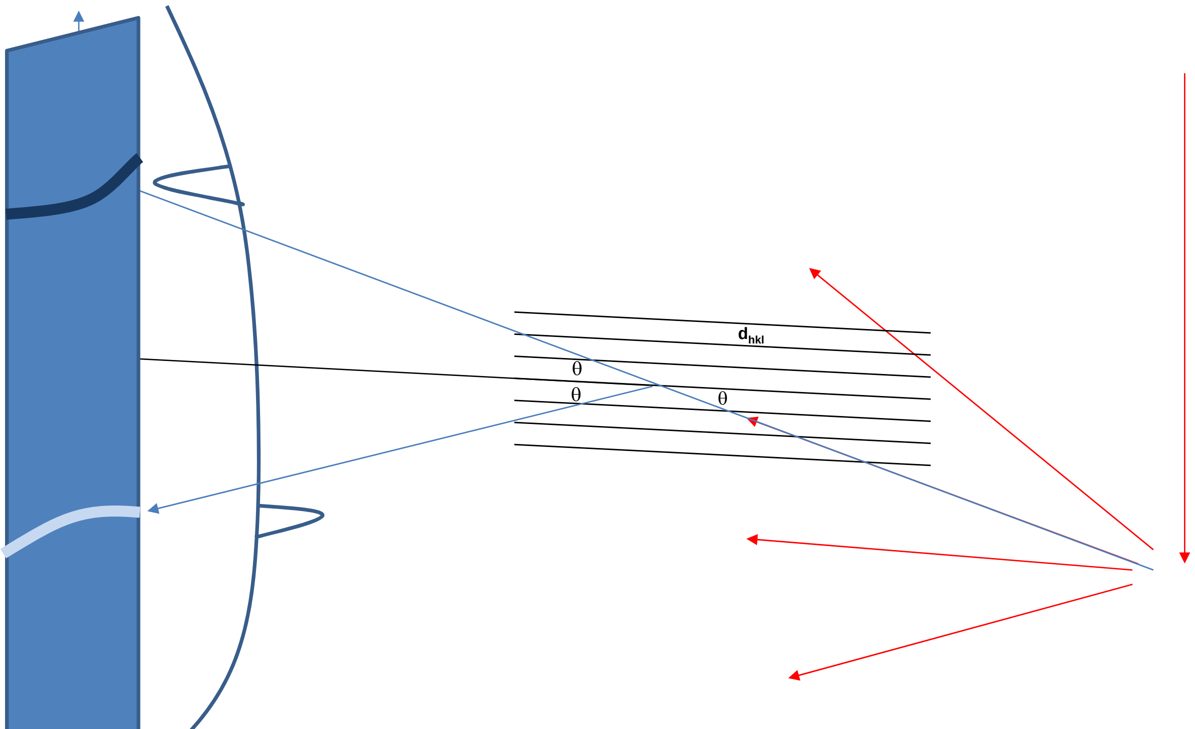
1) elastically scattered electrons
(forward scattered / back scattered)
&
2) Diffraction by crystal planes

= Bragg diffraction

Kikuchi lines (bands) in TEM



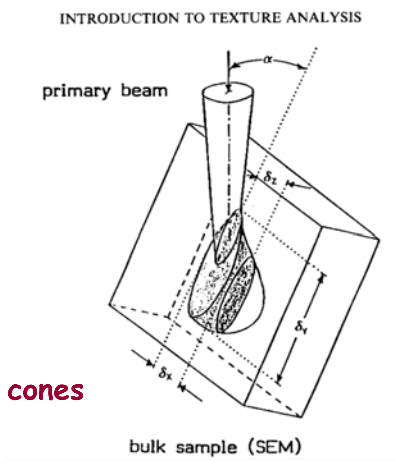
Thick sample: inelastic scattering followed by elastic scattering (= diffraction)



Bragg : 2 d_{hkl} sin θ = n λ

20 keV → λ ≈ 7 10^{-3} nm → θ ≈ 0.5°

Formation of EBSPs



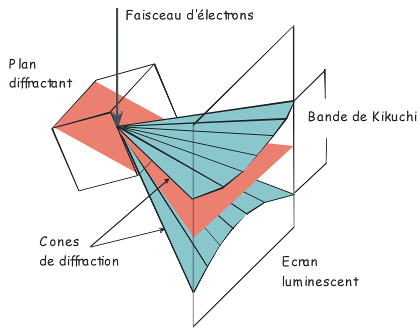
- **Backscattering** : due to mainly elastic (ΔE< 200 eV) interactions (plasmons, phonons) the electron beam is spread in all directions.
  The emission volume corresponds to a **small divergent source of electrons below the sample surface** (100 nm).
  - **Scattering** by crystallographic planes : **2 diffraction cones**

Bragg : 2 d_{hkl} sin θ = n λ

20 keV → λ ≈ 7 10^{-3} nm → θ ≈ 0.5°

- **Gnomonic projection** on the screen: **2 hyperbolas (Kikuchi bands)**. The middle of a band corresponds to the trace of the diffracting plane.

- Relatives intensities : structure factors (dynamical effects)



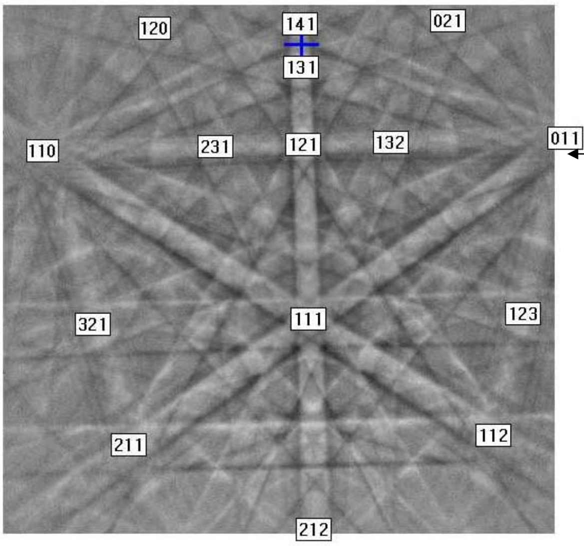
Indexation of EBSPs

Each band = diffraction of a family of planes

Intersections of bands = intersections of planes = zone axes

Angles between bands = angles between planes

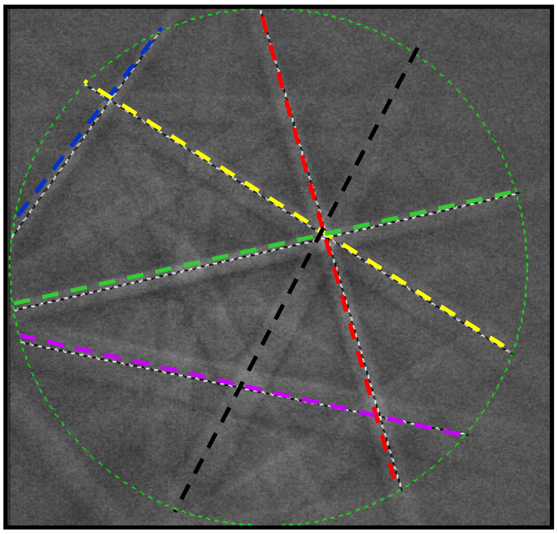
Position of bands directly linked to the crystallographic orientation

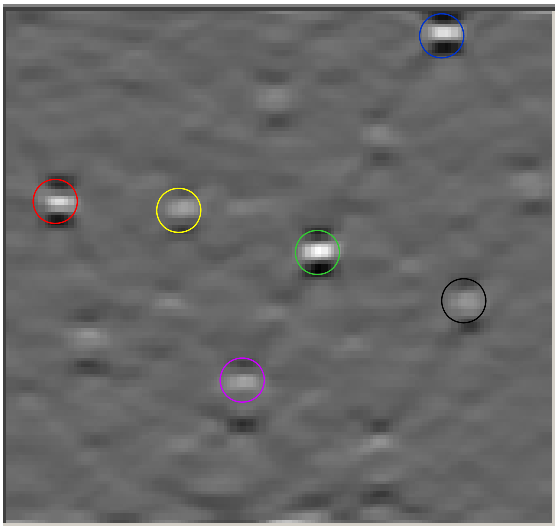


Zone axis

Si 40 kV

Hough transform





EBSP : the computer doesn't manage to distinguish between the grey levels

Hough transform : it is easier for the computer to detect the clear spots and dark areas top and bottom

Line positions with a common intersection (zone axis) will lie along a line/sine curve

Hough transform

The transform between the coordinates (x, y) of the diffraction pattern and the coordinates (ρ, θ) of Hough space is given by (Figure 8):

$$\rho = x \cos \theta + y \sin \theta \qquad (3)$$

A straight line is characterized by ρ, the perpendicular distance from the origin and θ the angle made with the x-axis and so is represented by a single point (ρ, θ) in Hough space. Kikuchi bands transform to bright regions in Hough space which can be detected

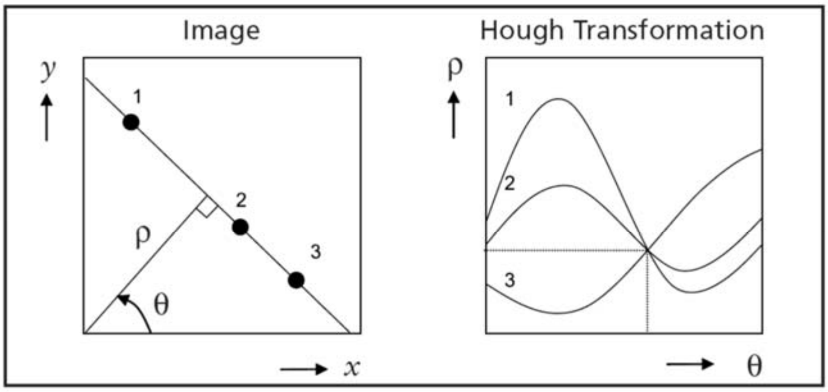
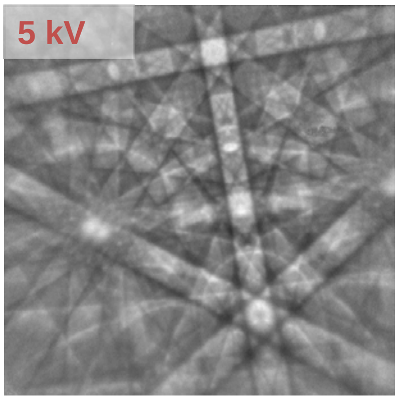
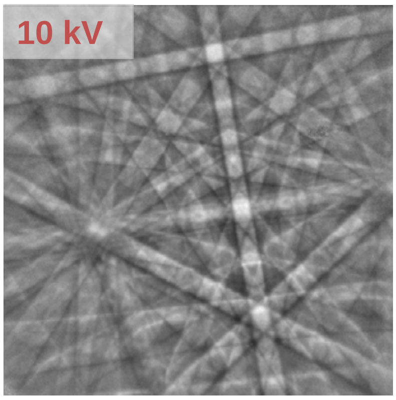
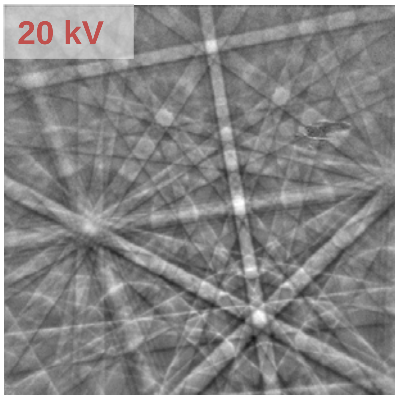


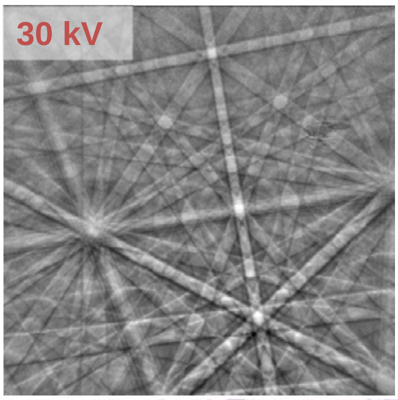
Figure 8: The Hough transform converts lines into points in Hough space

Influence of accelerating voltage









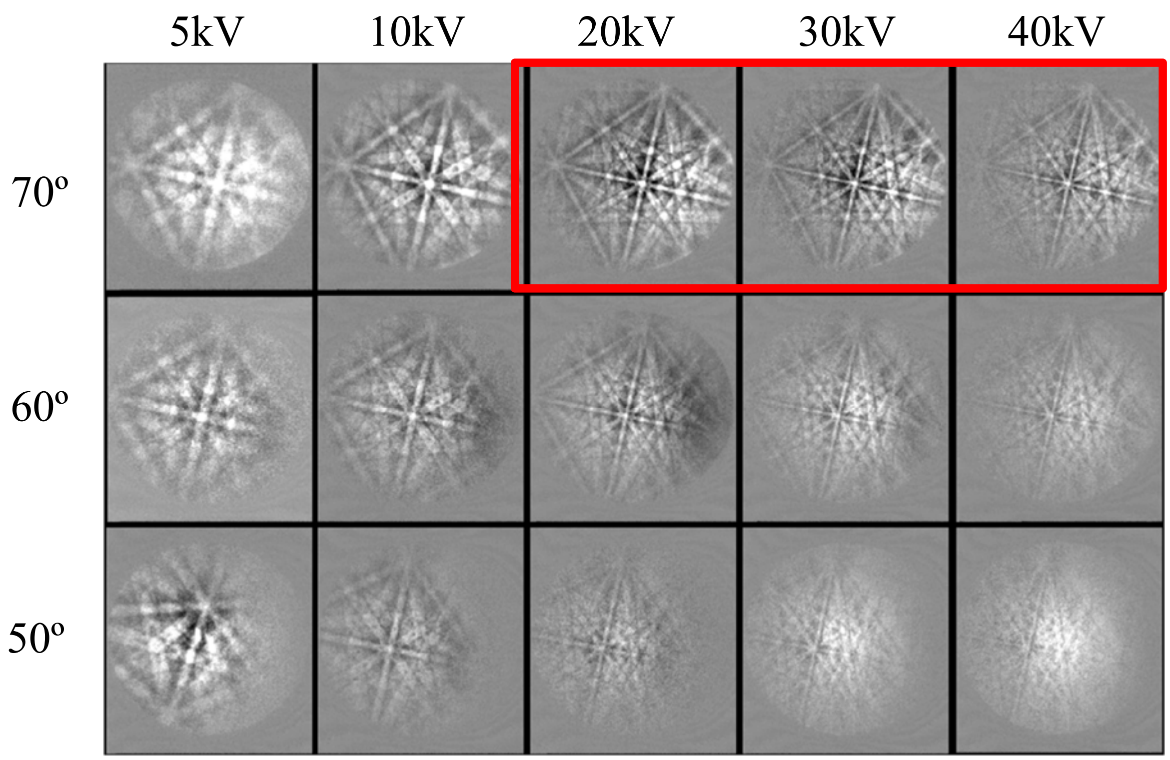
Si [100], same WD and camera length

- The phosphor screen efficiency drops at reduced voltages.

Bragg : $2\, d_{hkl} \sin \theta = n\, \lambda$

θ = width of bands

EBSP vs. voltage and tilt



Spatial and Angular accuracy

0.1 - 1° relative
≈ 2° absolute

20 nm - 1 µm

- **Limited by :**
  - ✓ Accuracy on the localization of Kikuchi bands (Hough, …).
  - ✓ The weak signal/noise ratio of the images, and blur of Kikuchi bands.
  - ✓ Geometrical fluctuations of the conditions of diffraction, and calibration.
  - ✓ absolute accuracy : sample position in the chamber.

Limited by the overlapping of diffraction patterns in the vicinity of a boundary

- **Depend on :**
  - ✓ interaction volume (energy and Z)
  - ✓ size of the beam spot (probe current, focus, astigmatism)
- **How to improve resolution :**
  - ✓ FEG SEM : higher brightness, stable and reproducible beam.

Applications of EBSD

EBSD patterns depend mainly on - **crystal structure** (symmetry)
- **crystal orientation**

■ Micro-crystallography

Determination of zone-axis symmetries :

1, 2, 3, 4, 6, m, 2mm, 3m, 4mm ou 6mm

→ identification of the crystal point group.
→ Possible indeterminations : (1 / -1, 3 / -3, 4 / -4, 6 / -6)

Seldom used

■ Orientation measurements

Main application

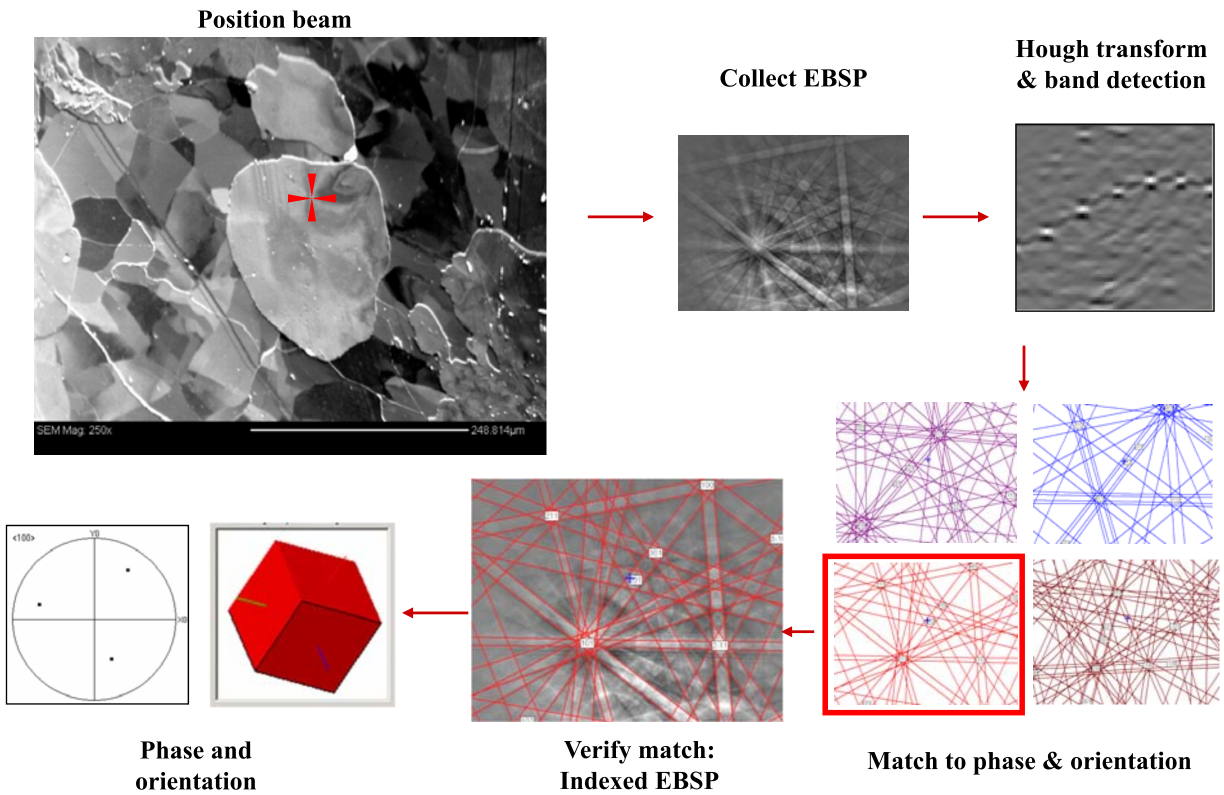
■ Phase identification / discrimination

✓ Chemical analysis (EDS, WDS)
✓ Rough lattice parameter measurement (~5%, depend on calibration)
✓ Symmetry analysis, or investigation with a crystallographic data base.

	X-ray	EBSD (SEM)	TEM
Sample preparation	easy	easy/moderate	difficult
Ease of use	moderate	easy	difficult
Speed	minutes	minutes	hours
Spatial resolution	~ 0.1 mm	~ 0.1 μm	~ 1 nm

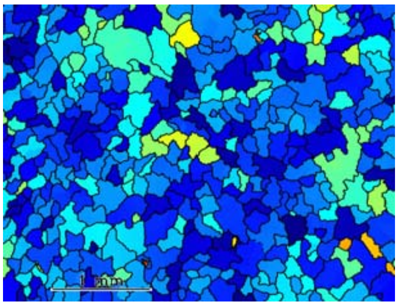
Document Oxford Instruments

Indexing Cycle



Data analysis and orientation representations
Directional solidification of a Ni-base alloy

Orientation image

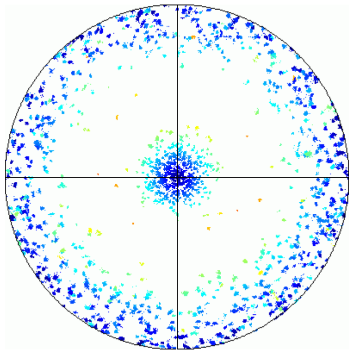


Angle (<001>, ND>)

0°

55°

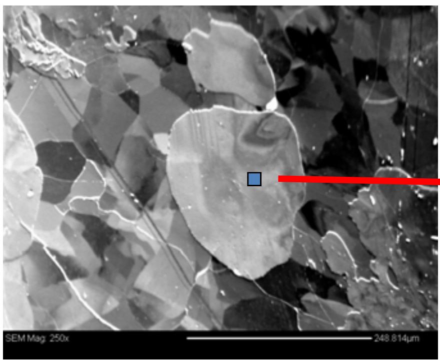
The scale is coloured considering the misorientation between the points and the <001> direction oriented along the normal direction (i.e. Z)

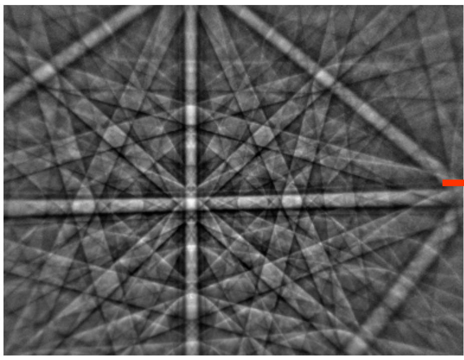


Pole figure

→ Stereographic projection

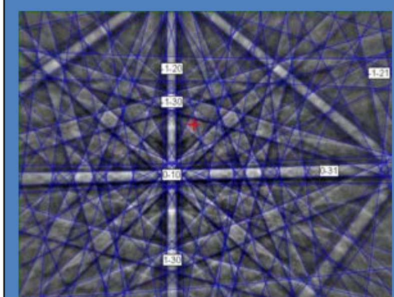
Phase identification





Acquire EBSP

List of possible phases (determined previously by EDS, XRD…)



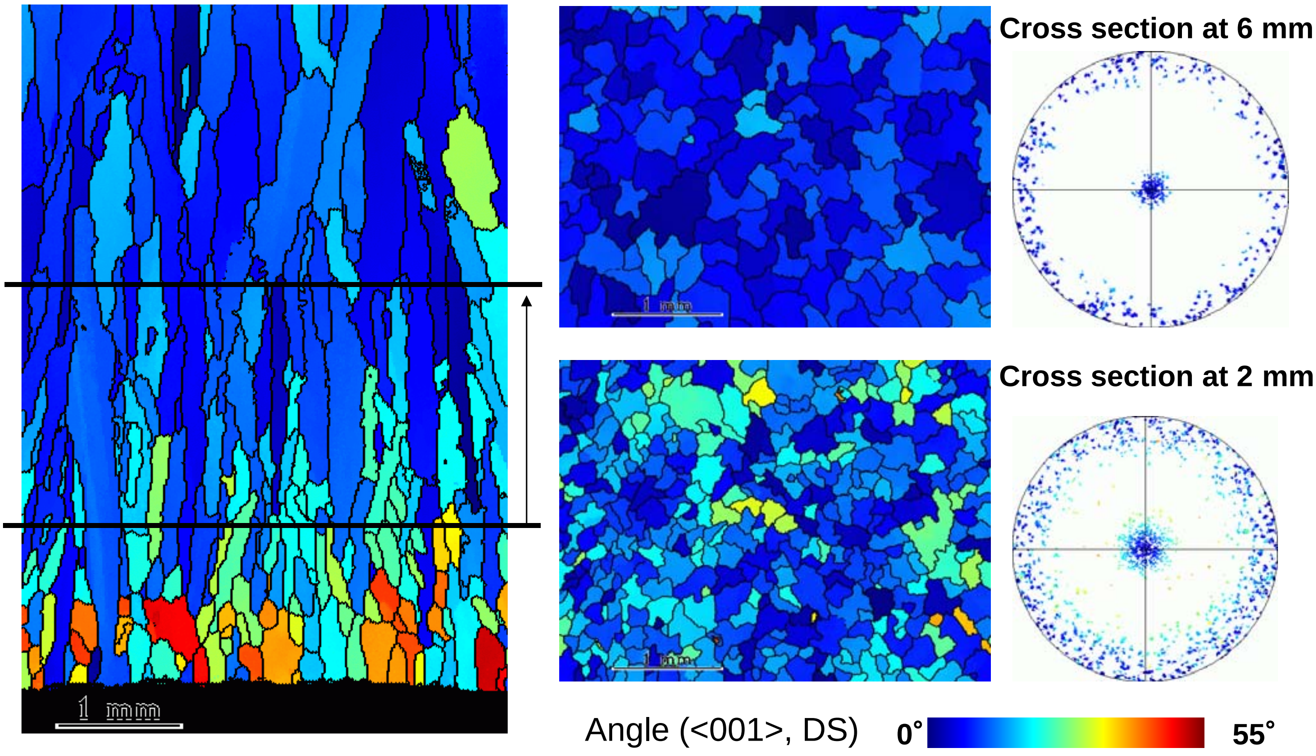
Index…

Solutions:

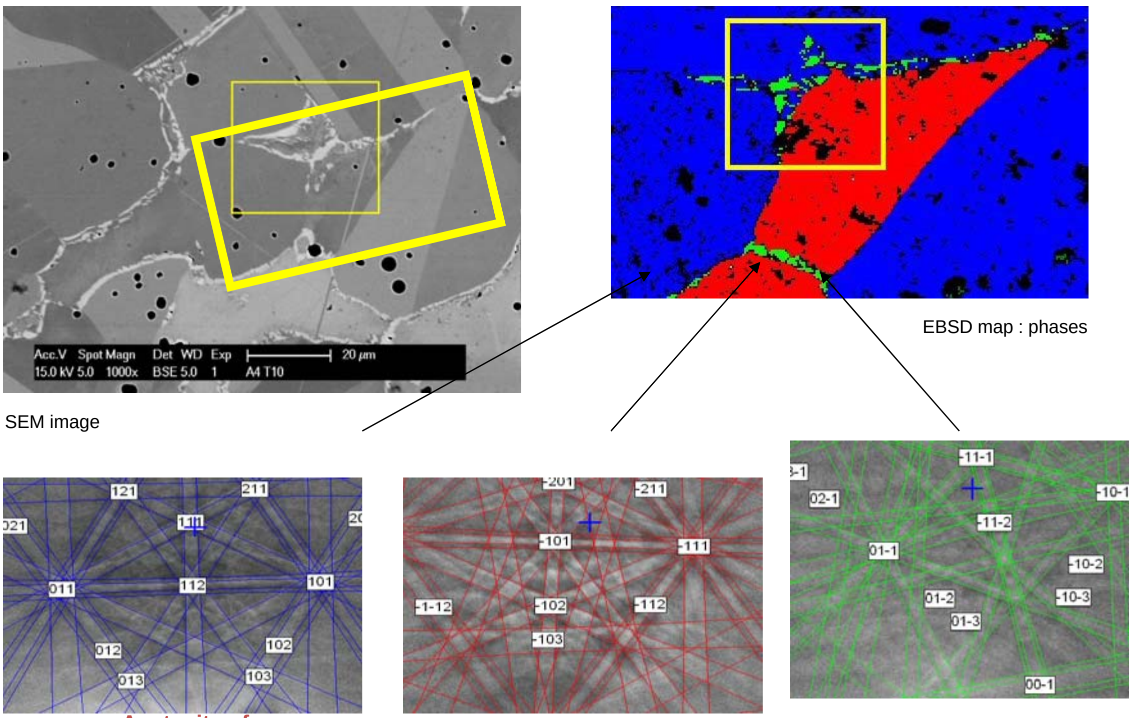
Phase	MAD	Bands
Aluminium	0.0521	6
Al As2 Cs3	1.9693	6
Al As2 Cs3	1.9697	6
Al As2 Cs3	1.9829	6

Phase identified

1. Directional solidification of a Ni-base alloy



2. Identification of chi phase in a duplex steel



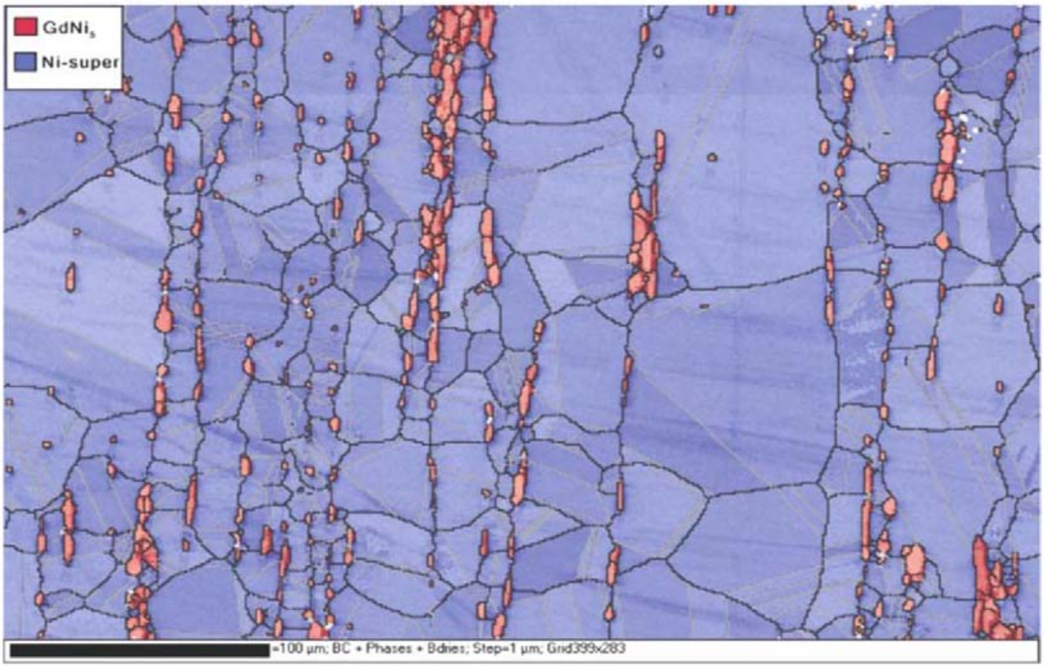
Austenite : fcc
Space group : F m 3 m

Ferrite : bcc
Space group : I m 3 m

Chi phase ($Cr_6Fe_{18}Mo_5$) : bcc
Space group : I -4 3 m

J. Friedli, E. Boehm Courjault, A. Jacot LSMX

3. $GdNi_5$ precipitates in a Ni-based superalloy



Phase map : Ni-alloy (blue),
$GdNi_5$ (red, 7% of area)

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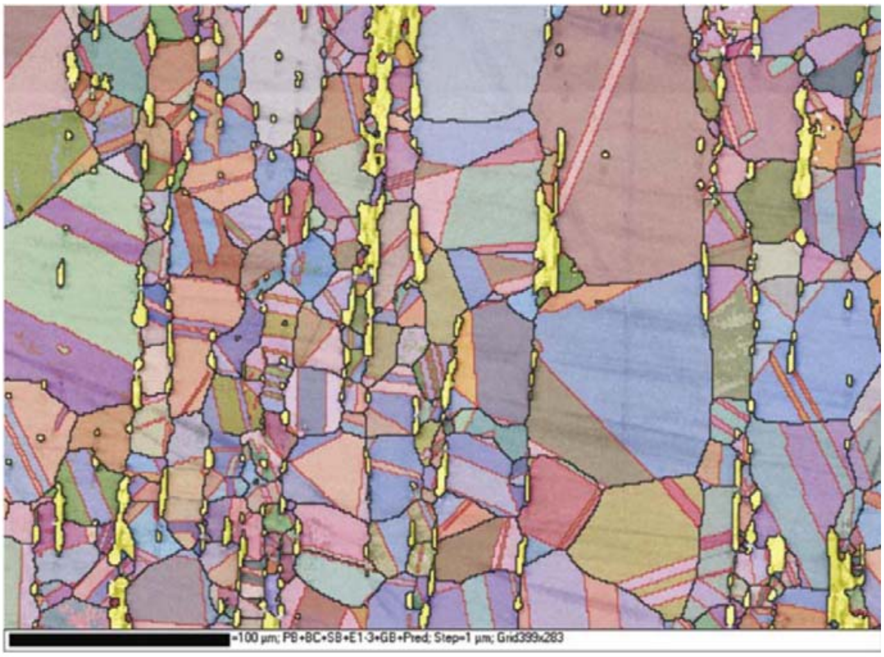
4. $GdNi_5$ precipitates in a Ni-based superalloy



Orientation map (black = grain or phase boundary ; red = twins)
$GdNi_5$ precipitates have all the same orientation