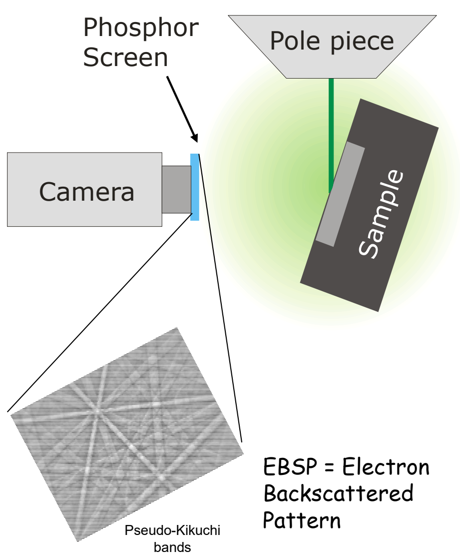
EBSD
electron backscatter diffraction

Marco Cantoni
021/693.48.16

Centre Interdisciplinaire de Microscopie Electronique
CIME

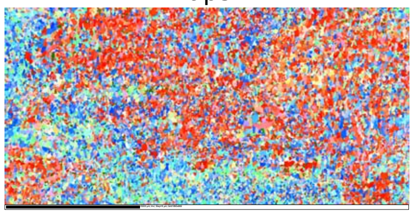
EBSD Basics

- Quantitative, general microstructural characterization in the SEM
- 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 that survive under the beam
  - Metals, ceramics, minerals
  - Conductors and insulators
  - Pharmaceuticals and polymers generally damage too quickly to work on, although...

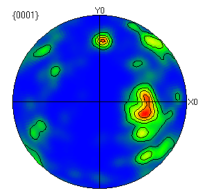


Uses of EBSD

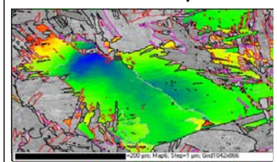
Maps

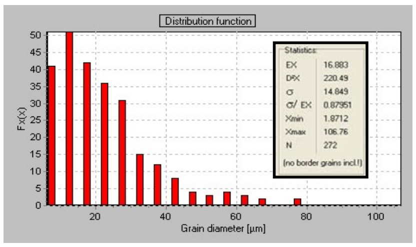


Pole figures
(orientation analysis)



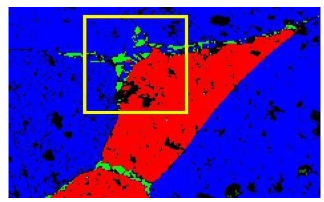
Strain analysis



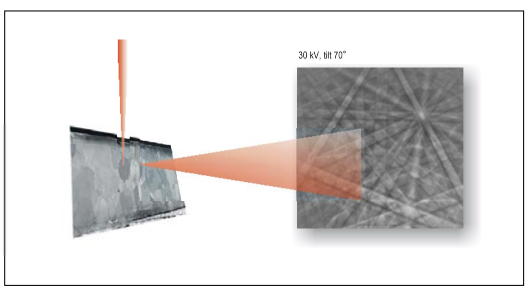


Quantitative microstructural data

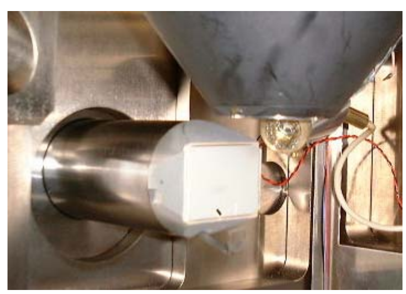
Phase identification



How EBSPs are obtained ?



Tilt 70° - Spot mode - 30 kV - 10 nA



Phosphor screen + CCD camera

EBSD : electron diffraction in the SEM

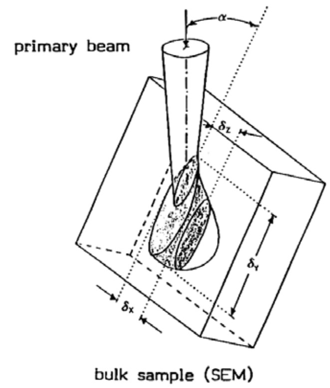
- ✓ Electrons of the incident beam spread beneath the surface in all direction due to elastic interactions (backscattered electrons) :
  small divergent source of electron behind (~ 100 nm) the sample surface.
- ✓ These electrons are **diffracted by crystal planes** according to the Bragg condition.

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** :

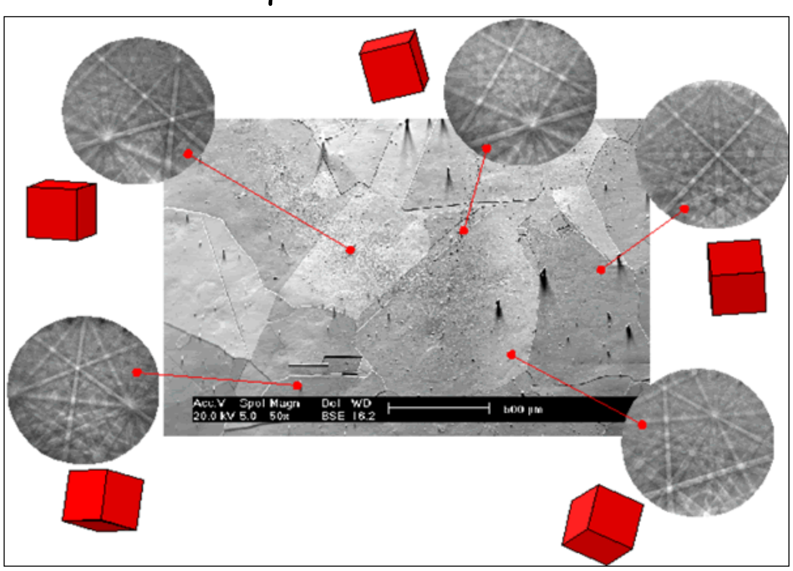
→ chemical-mechanical polishing (silica or alumina suspension)
→ electro-polishing or chemical polishing/etching
→ ion-milling or plasma etching

✓ cleaved surface, growth surface

✓ Insulating materials

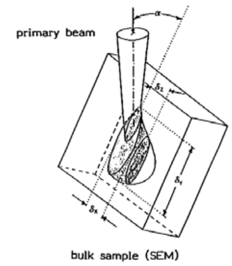
→ carbon coating (< 100 Å) (degrade pattern quality)
→ low-vacuum SEM (a few Pa)

Acquisition of EBSPs



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**.

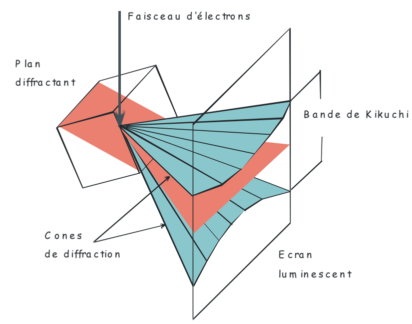
Formation of EBSPs



- **Backscattering** : due to slightly inelastic (< 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**

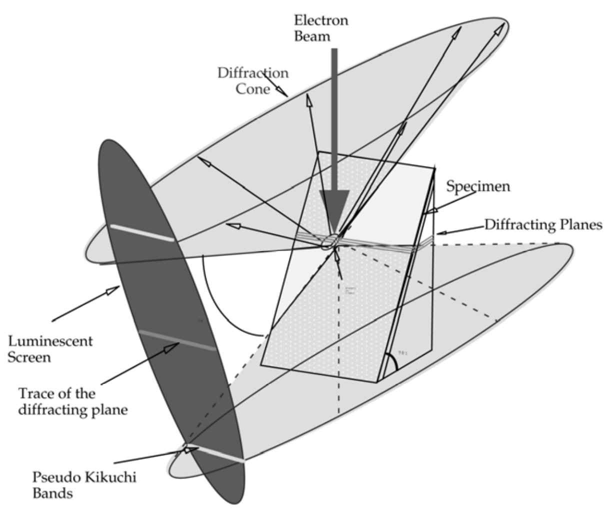
Bragg : $2\ d_{hkl} \sin\theta = n\lambda$
20 keV $\rightarrow \lambda \approx 7\ 10^{-3}$ nm $\rightarrow \theta \approx 0.5°$

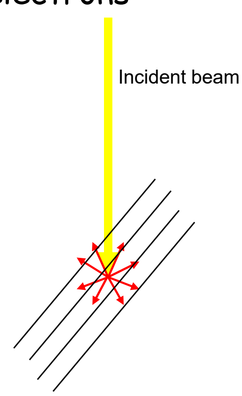


- **Gnomonic projection** on the screen: **2 hyperbolas (Kikuchi bands)**. The middle of a band corresponds to the trace of the diffracting plane.

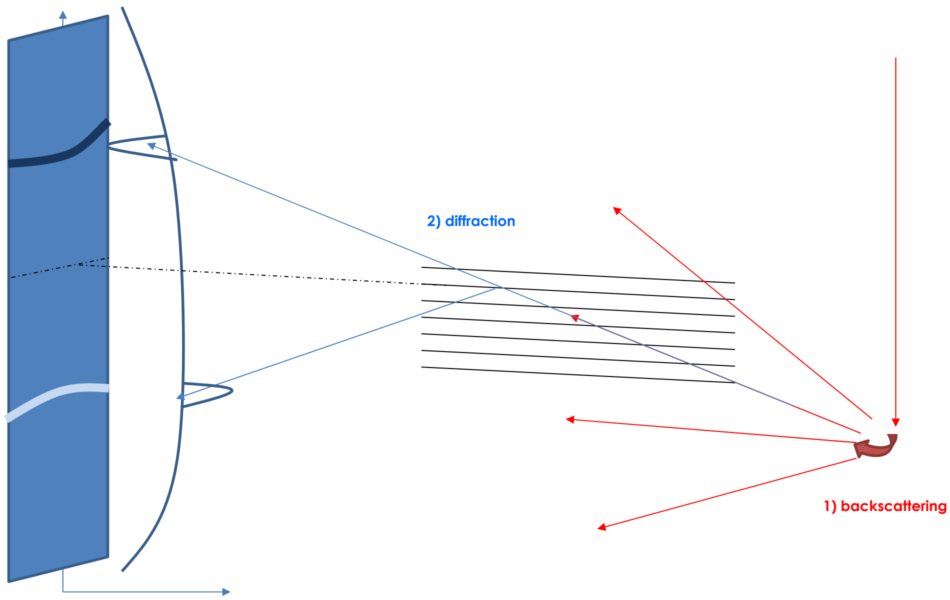
- Relatives intensities : structure factors (dynamical effects)

Diffraction of backscattered electrons





a) Pseudo-elastically (small energy loss) scattered electrons (forward scattered / back scattered)
&
b) crystal planes = Bragg diffraction



Intensity of BSE on screen

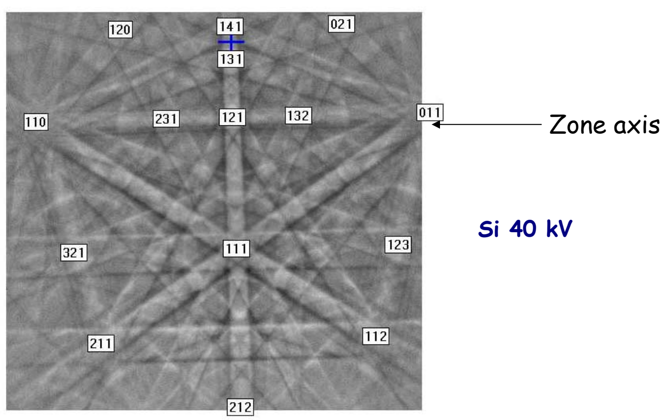
Indexation of EBSPs

Each band = diffraction of a family of planes

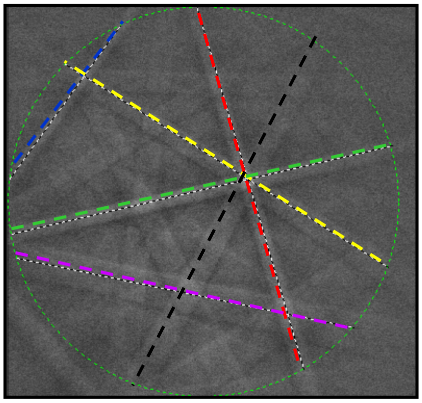
Intersections of bands = intersections of planes = zone axes

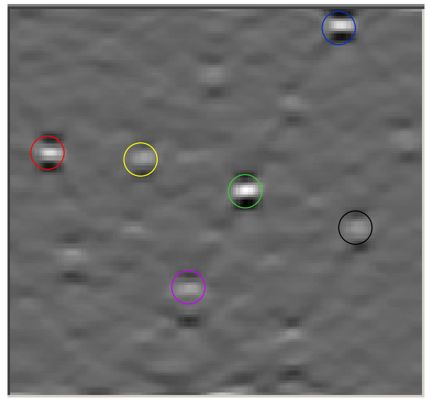
Angles between bands = angles between planes

Position of bands directly linked to the crystallographic orientation



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

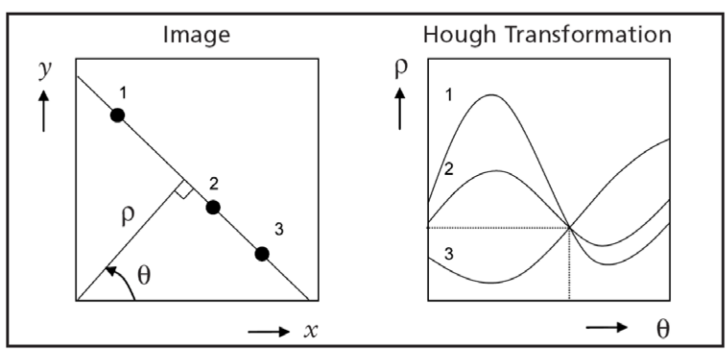
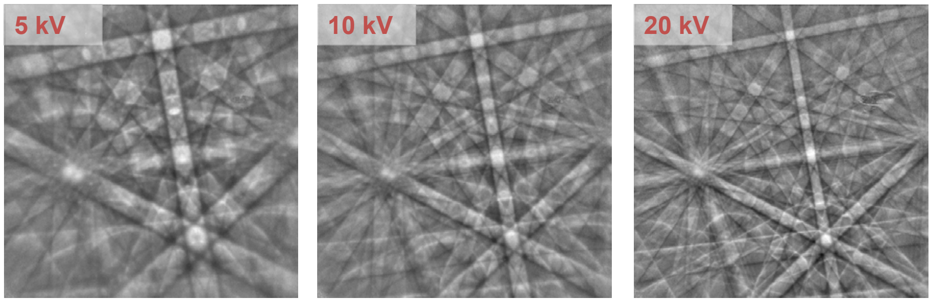
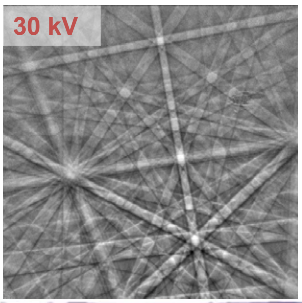


Figure 8: The Hough transform converts lines into points in Hough space

Influence of accelerating voltage

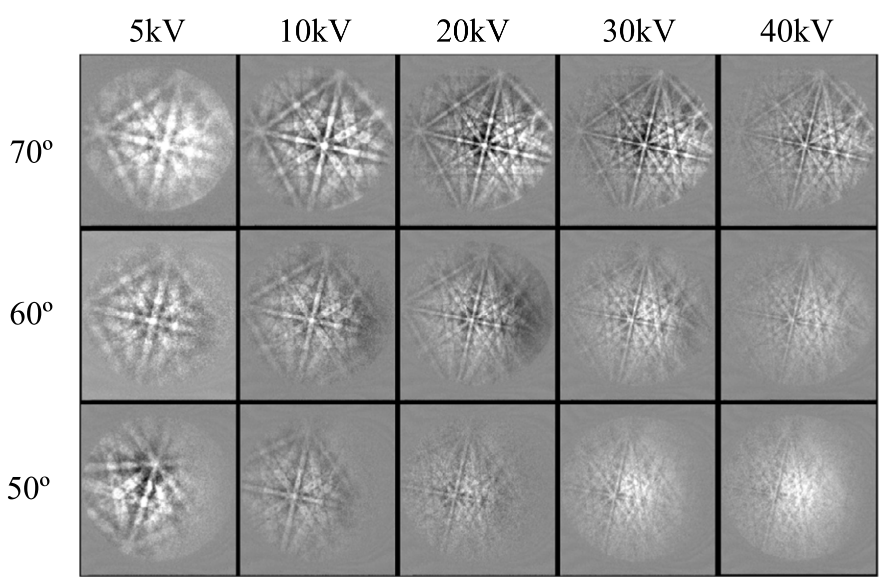




Si [100], same WD and camera length

- The phosphor screen efficiency drops at reduced voltages.

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**

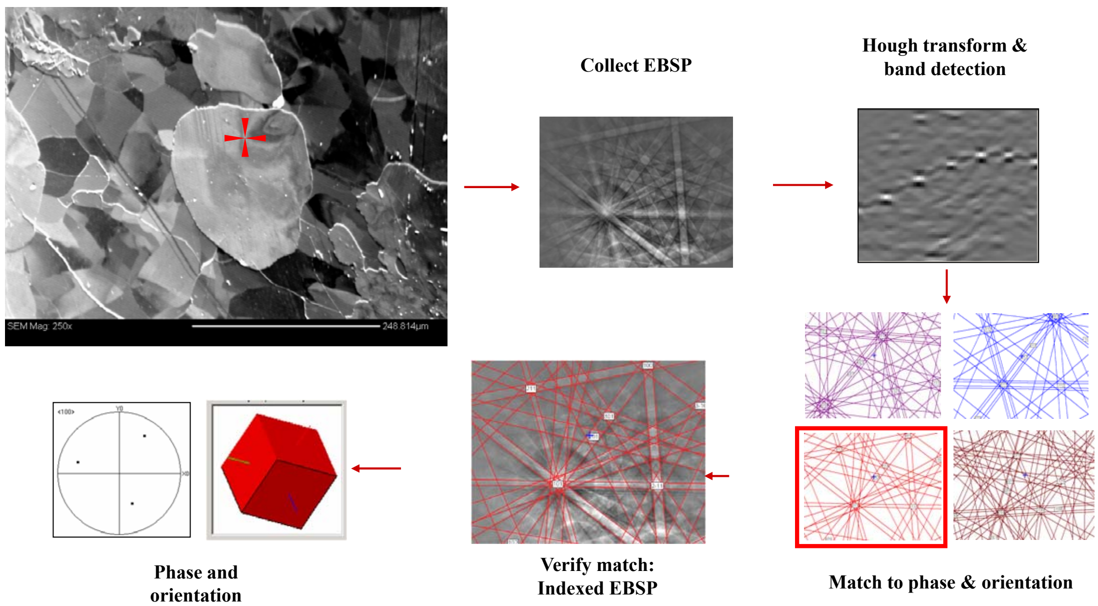
Main application

- **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

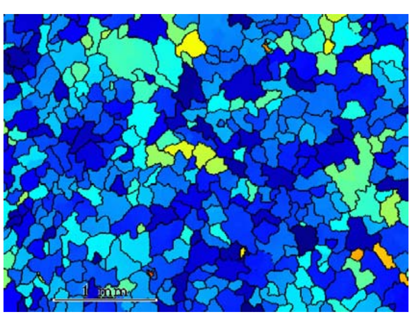
Indexing Cycle



Data analysis and orientation representations

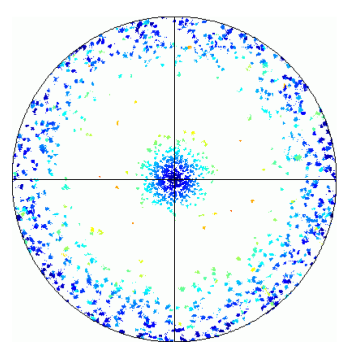
Directional solidification of a Ni-base alloy

Orientation image



Angle (<001>, ND>)

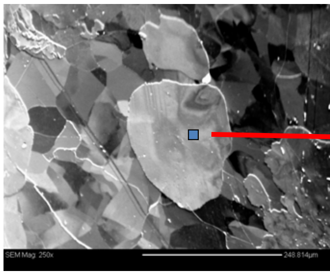
The scale is coloured considering the misorientation between the points and the <001> direction oriented along the normal direction (i.e. Z)

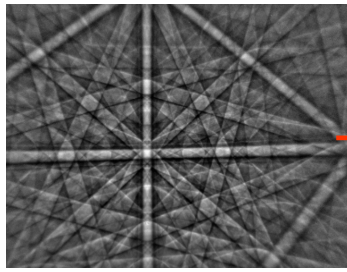


Pole figure

→ Stereographic projection

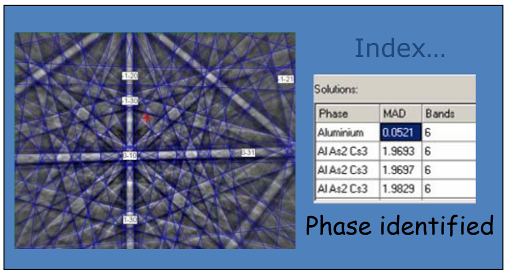
Phase identification



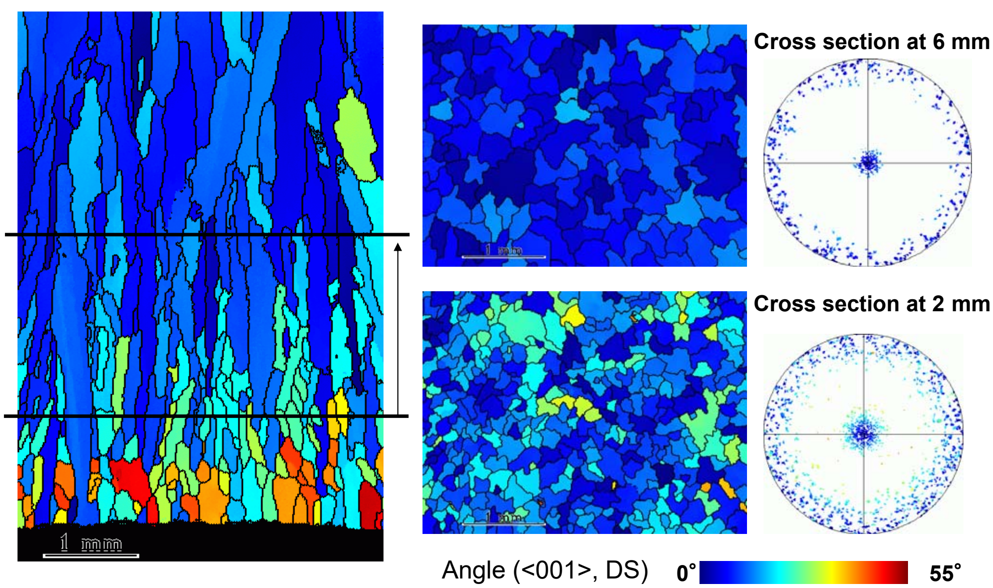


Acquire EBSP

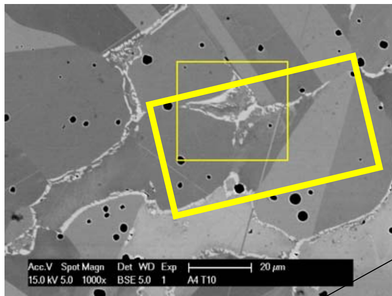
List of possible phases (determined previously by EDS, XRD...)



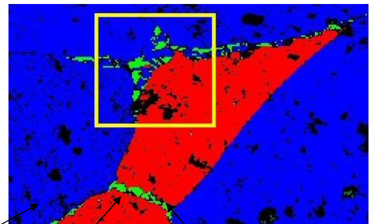
1. Directional solidification of a Ni-base alloy



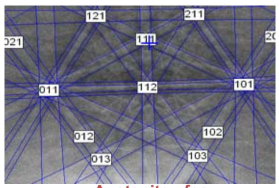
2. Identification of chi phase in a duplex steel



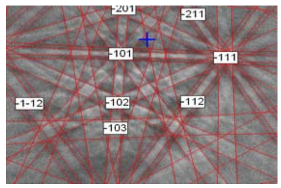
SEM image



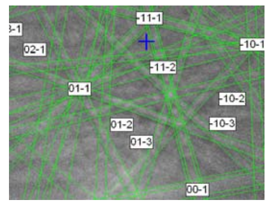
EBSD map : phases



Austenite : fcc
Space group : F m 3 m



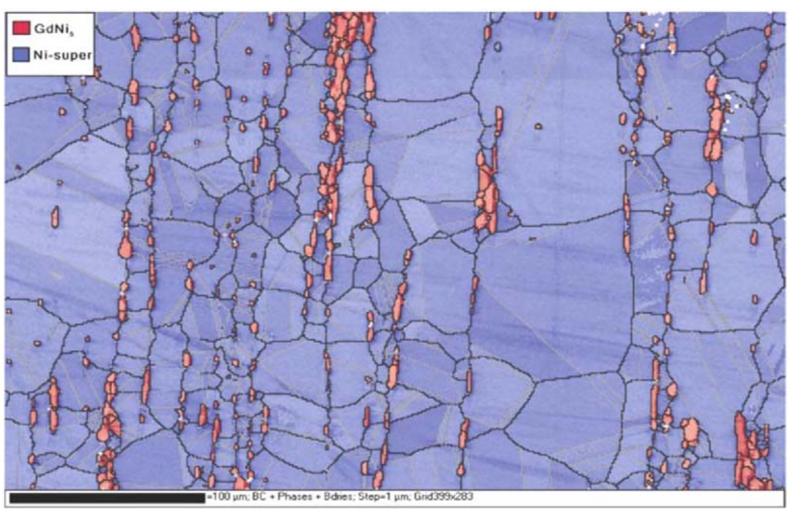
Ferrite : bcc
Space group : I m 3 m



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

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

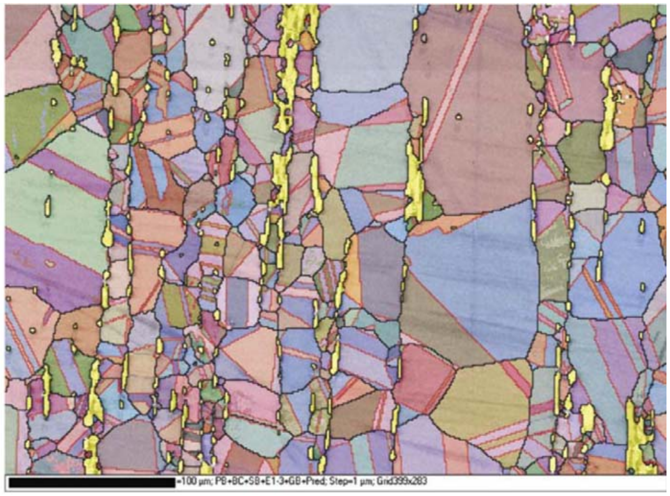
3. $GdNi_5$ precipitates in a Ni-based superalloy



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

Document Oxford Instruments

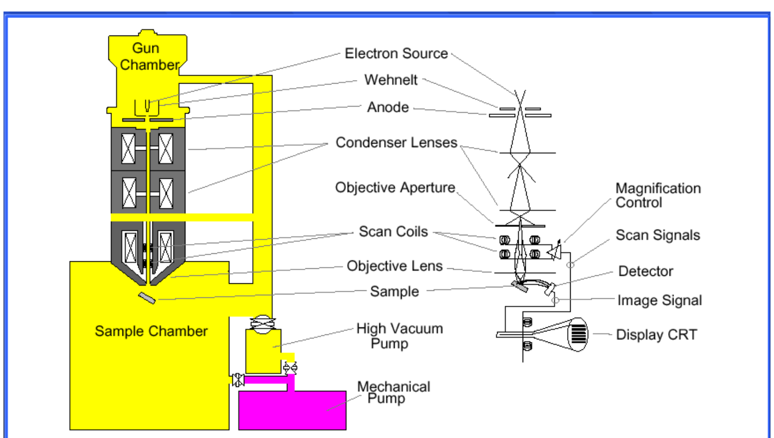
4. $GdNi_5$ precipitates in a Ni-based superalloy



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

ESEM

Acknowledgements to:



An SEM forms an image by scanning a finely focused beam of electrons over the sample surface.

ESEM - Environmental SEM

PHILIPS ElectroScan

- Investigate samples in a variety of environments manipulating pressure, temperature, humidity, and composition of ambient gas or liquid.
- Observe non-conductive, wet, dirty, outgassing, dynamic samples without cleaning or coating.

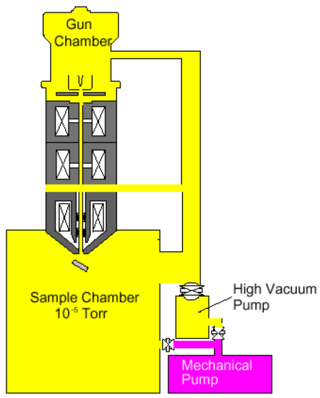
Seeing Things You've Never Seen Before

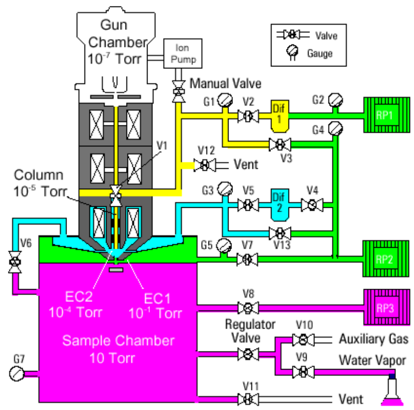
PHILIPS ElectroScan

Uncoated Silicon Nitride | Dissolving Table Salt | Living Aphid

Oxidizing Iron 800° C | Crystallizing KCL 600° C | Oil and Water Droplets

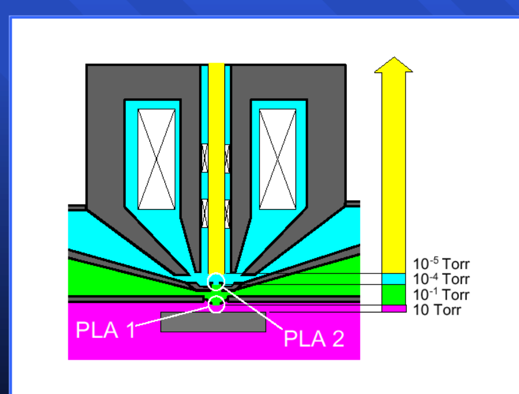
Conventional SEM "(C)SEM" *Environmental SEM* "ESEM"





Multiple Pressure Limiting Apertures

ElectroScan



Multiple PLAs permit larger aperture diameters and shorter gas path lengths.

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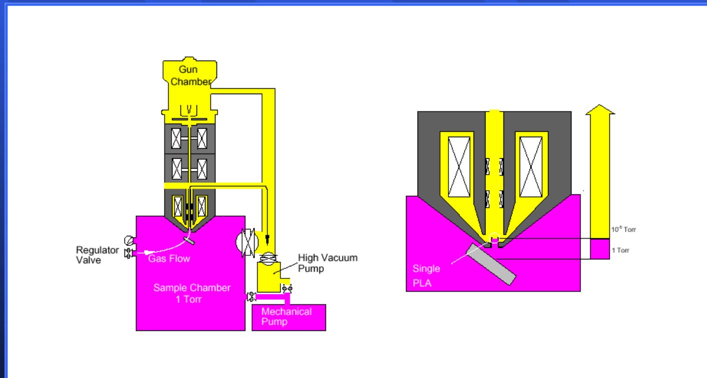
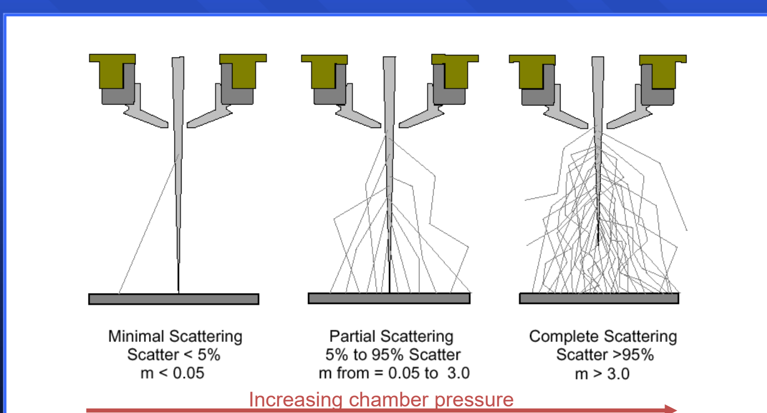
LV-CSEM Vacuum System

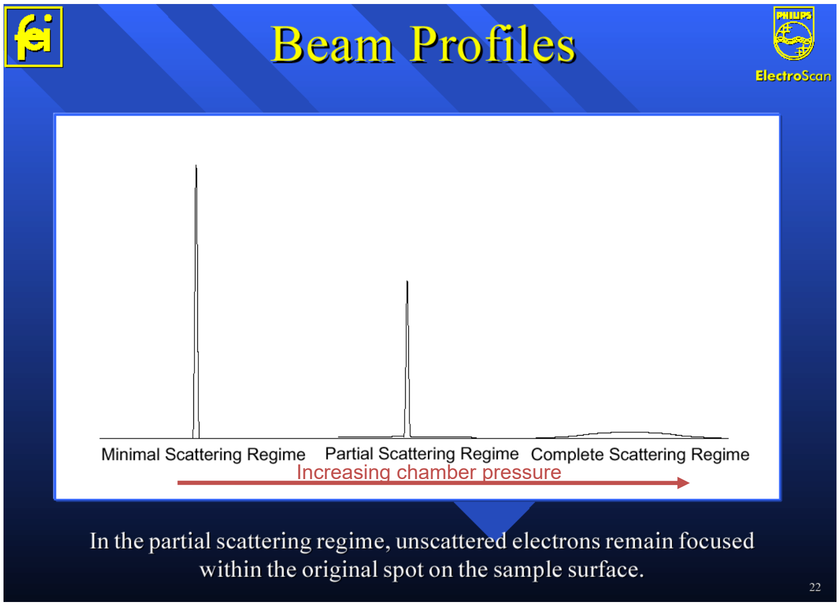
(LV: Low Vacuum)

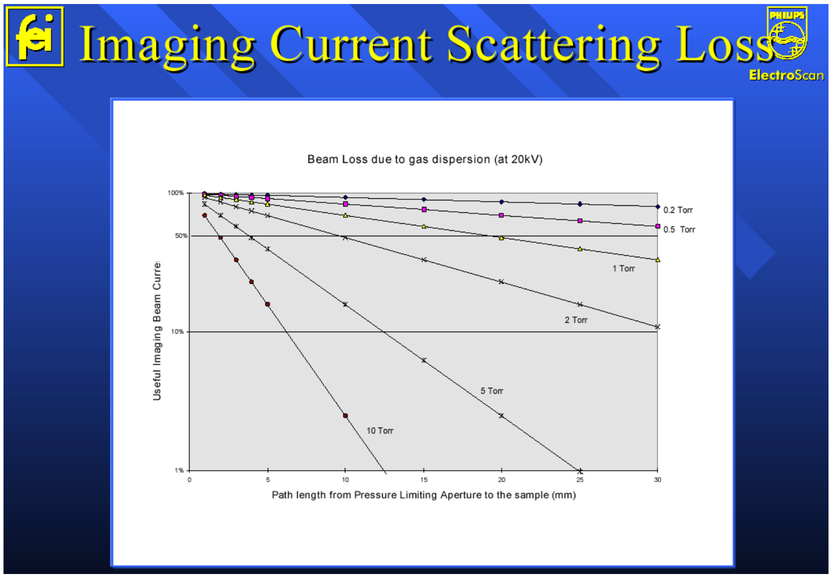


LV-CSEMs are restricted to a single Pressure Limiting Aperture.
It must be large enough to pass the beam and small enough to limit gas flow.

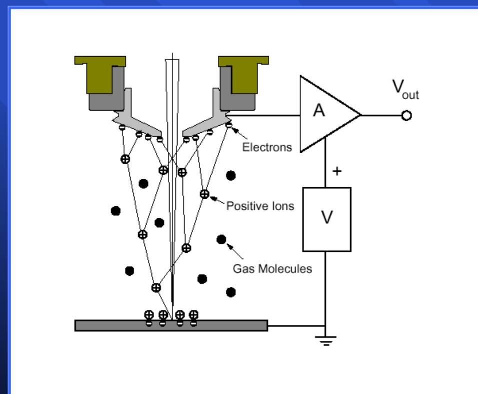
Electron Scattering




Beam Profiles
PHILIPS
ElectroScan
Minimal Scattering Regime
Partial Scattering Regime
Complete Scattering Regime
Increasing chamber pressure
In the partial scattering regime, unscattered electrons remain focused within the original spot on the sample surface.
22


Imaging Current Scattering Loss
PHILIPS
ElectroScan
Beam Loss due to gas dispersion (at 20kV)
Useful Imaging Beam Curre
100%
50%
10%
1%
0
5
10
15
20
25
30
0.2 Torr
0.5 Torr
1 Torr
2 Torr
5 Torr
10 Torr
Path length from Pressure Limiting Aperture to the sample (mm)

Environmental Secondary Electron Detector



The ESD uses the gas in the environment to amplify the secondary electron signal

LEO's solution for SE detector in Low Vapor Pressure SEM
(VP detector for VP mode in SEM)

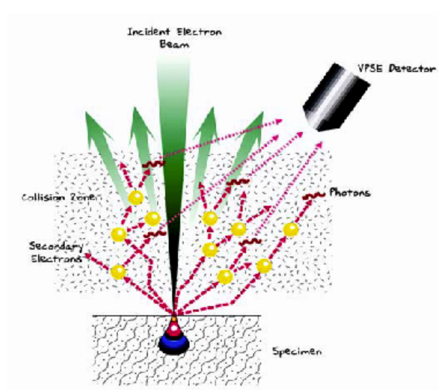
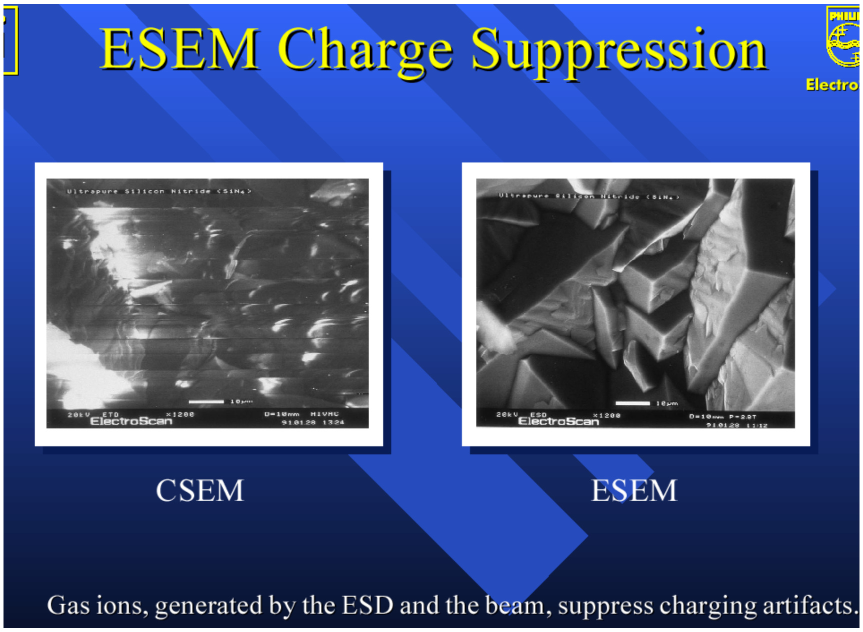
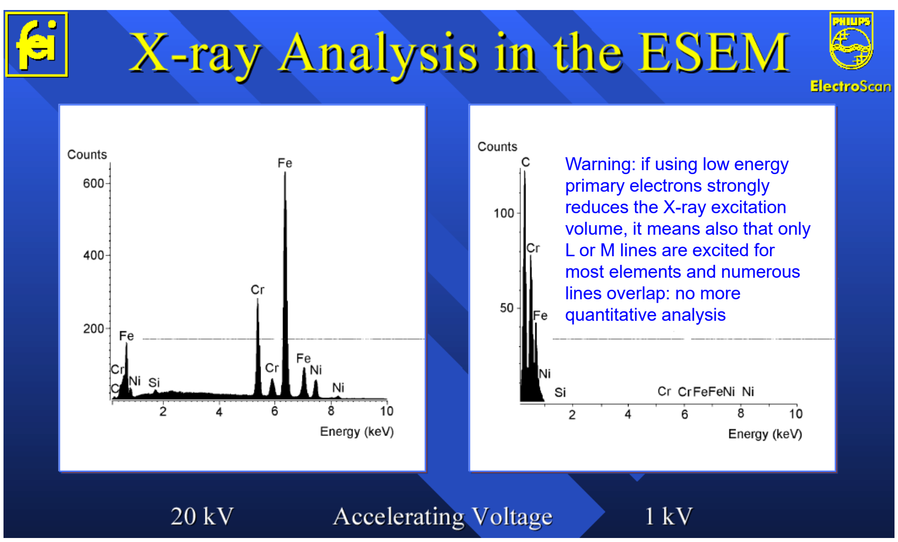
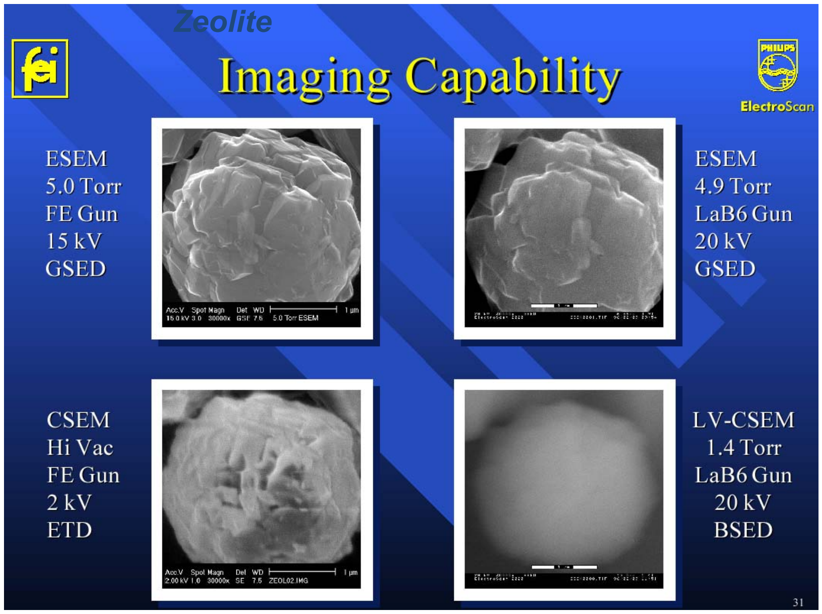
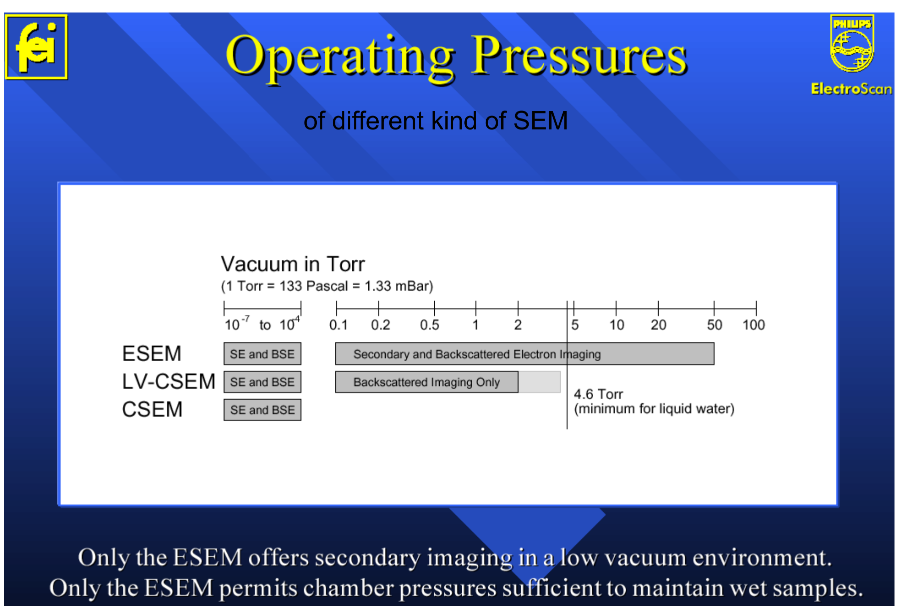


Figure 3: Principle of SE detection in VP mode by using the VPSE detector

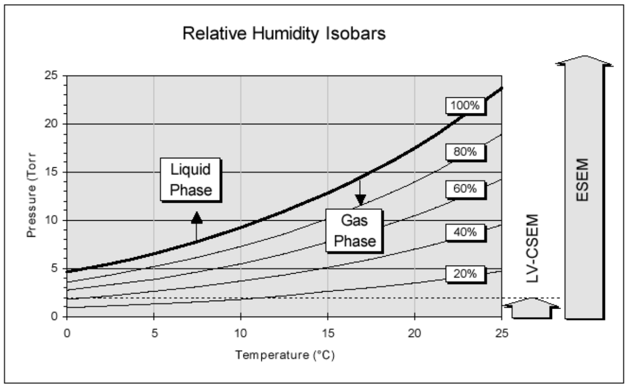

ESEM Charge Suppression
CSEM
ESEM
Gas ions, generated by the ESD and the beam, suppress charging artifacts.


X-ray Analysis in the ESEM
ElectroScan
Counts
Energy (keV)
Warning: if using low energy primary electrons strongly reduces the X-ray excitation volume, it means also that only L or M lines are excited for most elements and numerous lines overlap: no more quantitative analysis
20 kV
Accelerating Voltage
1 kV


Zeolite
Imaging Capability
ElectroScan
ESEM
5.0 Torr
FE Gun
15 kV
GSED
ESEM
4.9 Torr
LaB6 Gun
20 kV
GSED
CSEM
Hi Vac
FE Gun
2 kV
ETD
LV-CSEM
1.4 Torr
LaB6 Gun
20 kV
BSED
31


Operating Pressures
of different kind of SEM
ElectroScan
Vacuum in Torr
(1 Torr = 133 Pascal = 1.33 mBar)
10^-7 to 10^-4
0.1 0.2 0.5 1 2 5 10 20 50 100
ESEM
LV-CSEM
CSEM
SE and BSE
Secondary and Backscattered Electron Imaging
Backscattered Imaging Only
4.6 Torr
(minimum for liquid water)
Only the ESEM offers secondary imaging in a low vacuum environment.
Only the ESEM permits chamber pressures sufficient to maintain wet samples.

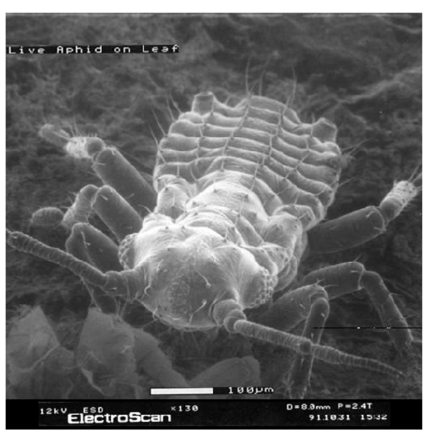
Relative Humidity Isobars



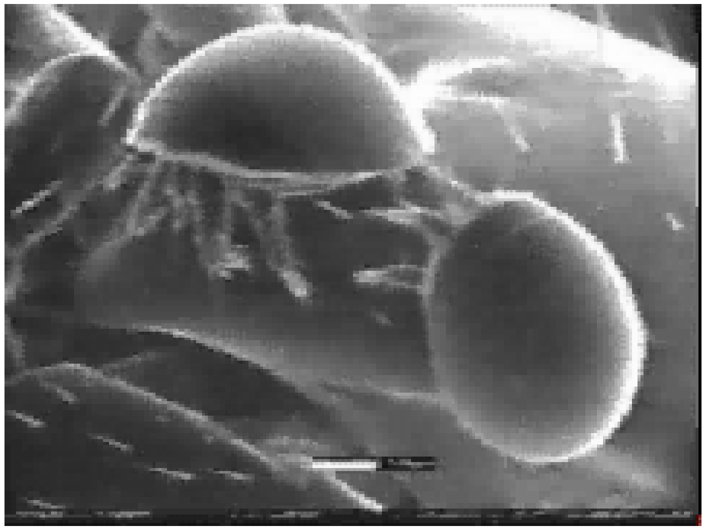
temperature just above 0° C reduces as much as possible the chamber pressure while still preventing sample freezing (Ice formation may destroy the microstructure)

The sample chamber pressure must be at least 4.6 Torr to sustain liquid water.

Hydrated Samples

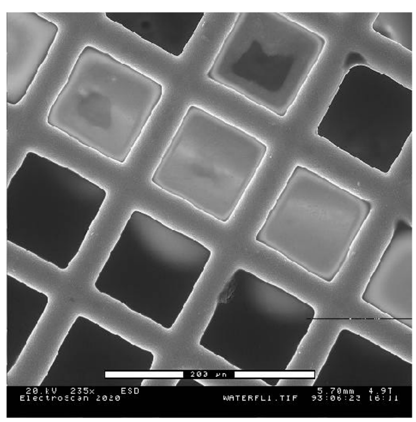


Live Aphid



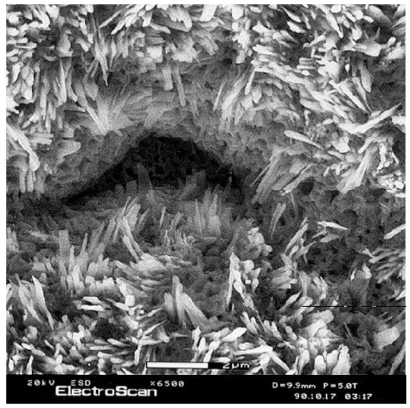
Dust Mite

Hydrated Samples



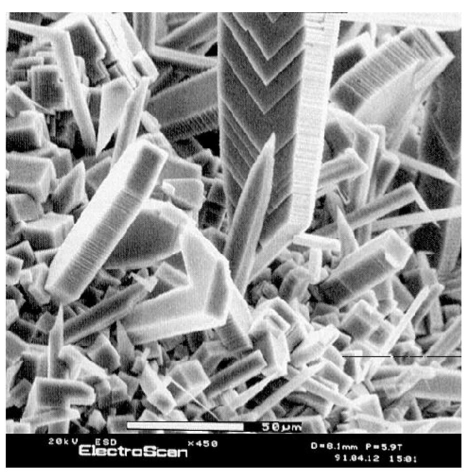
Water Film on Copper Grid

Oxidation/Corrosion



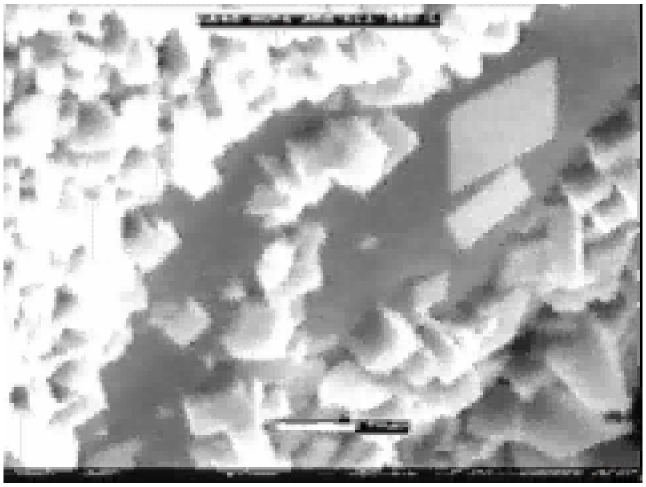
Iron Oxidizing in the ESEM

Phase transitions



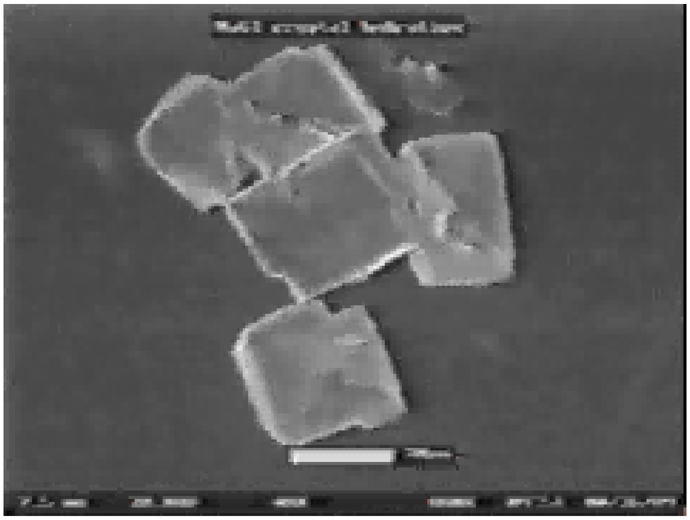
KCL Crystals grown from gas in ESEM at 600° C

Phase transitions



KCL Crystals grown from gas in ESEM at 600° C

HydrationProcesses



Salt dissolving in water condensed from the ESEM environment

PASTA Party