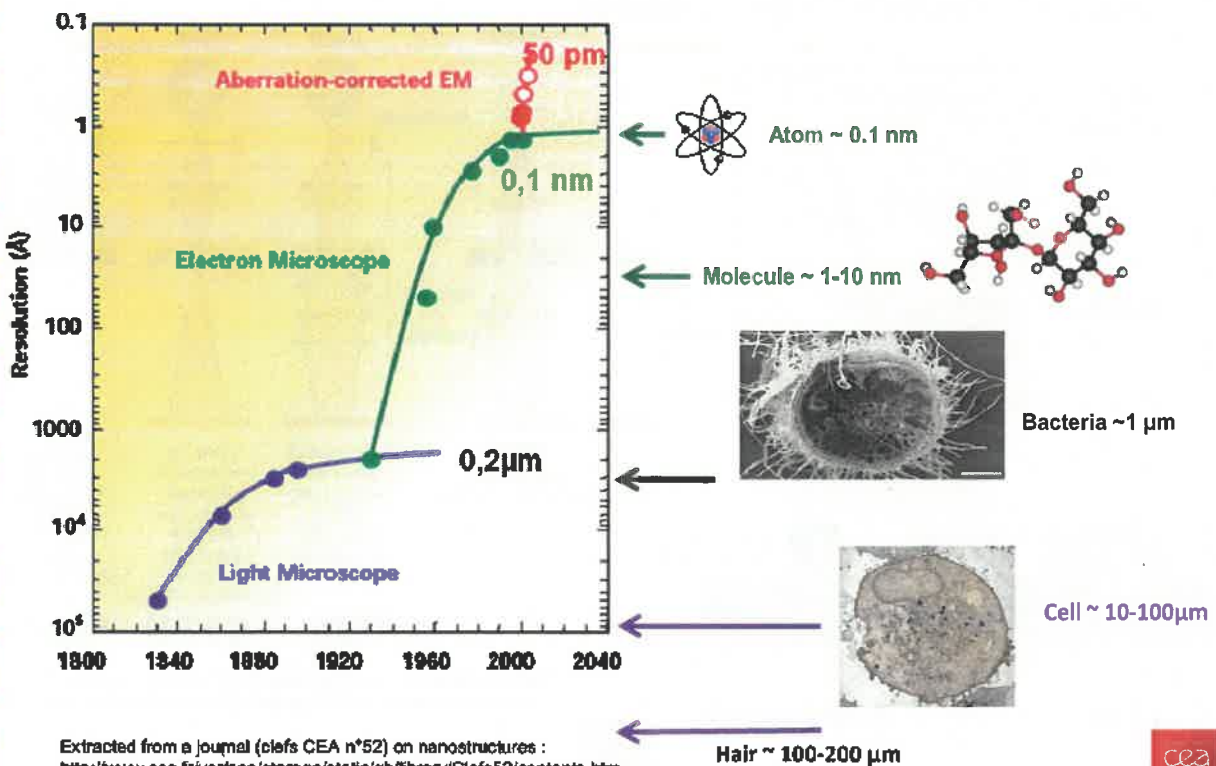


# Electron Microscopy for materials science

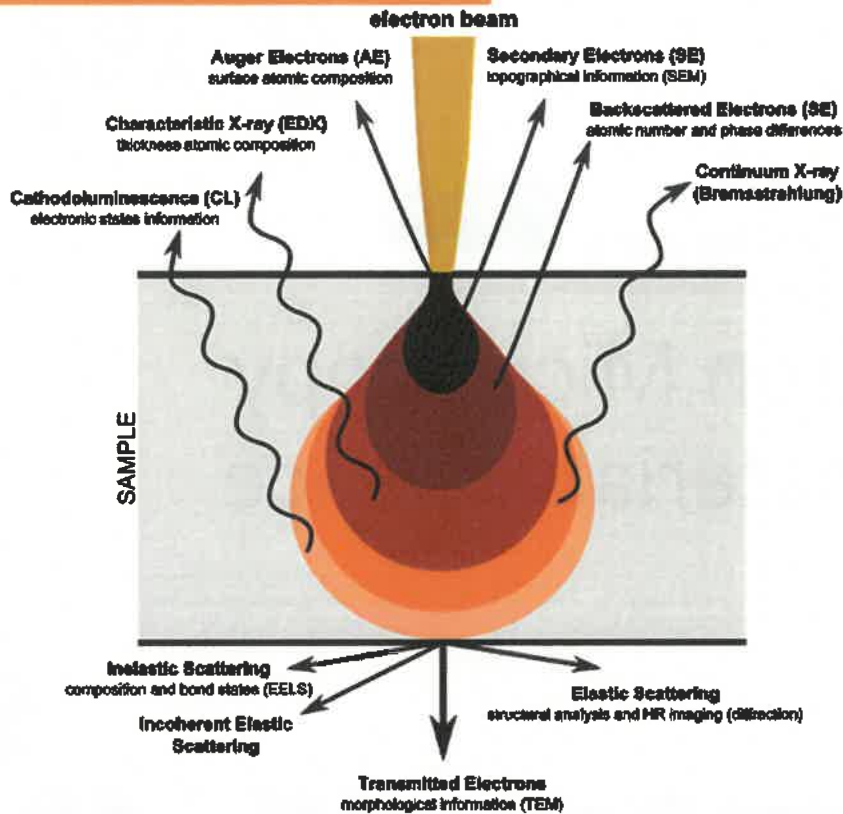
NANO-ELEC.



## Electron microscope: magnifying glass ?



# Much more than a magnifying glass

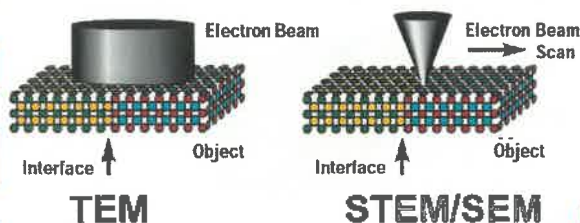
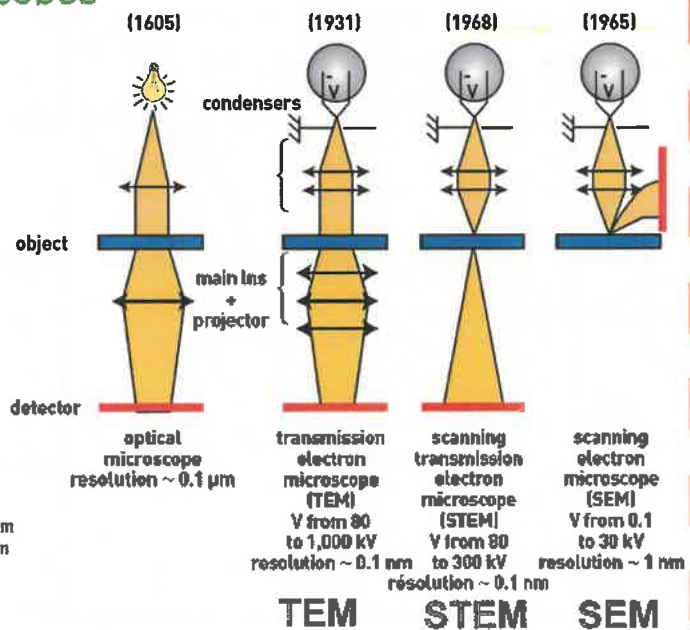
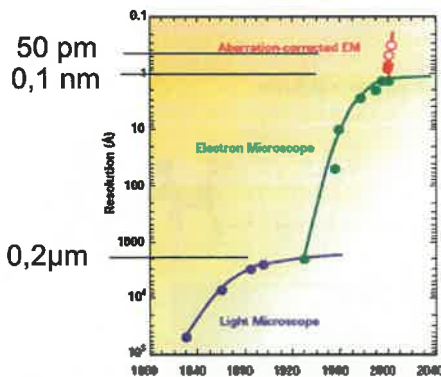


Extracted from a journal (clefs CEA n°52) on nanostructures : <http://www.cea.fr/var/cea/storage/static/gb/library/Clefs52/contents.htm>



# Much more than a magnifying glass

## Optical and electron microscopes



Extracted from a journal (clefs CEA n° 52) on nanostructures : <http://www.cea.fr/var/cea/storage/static/gb/library/Clefs52/contents.htm>

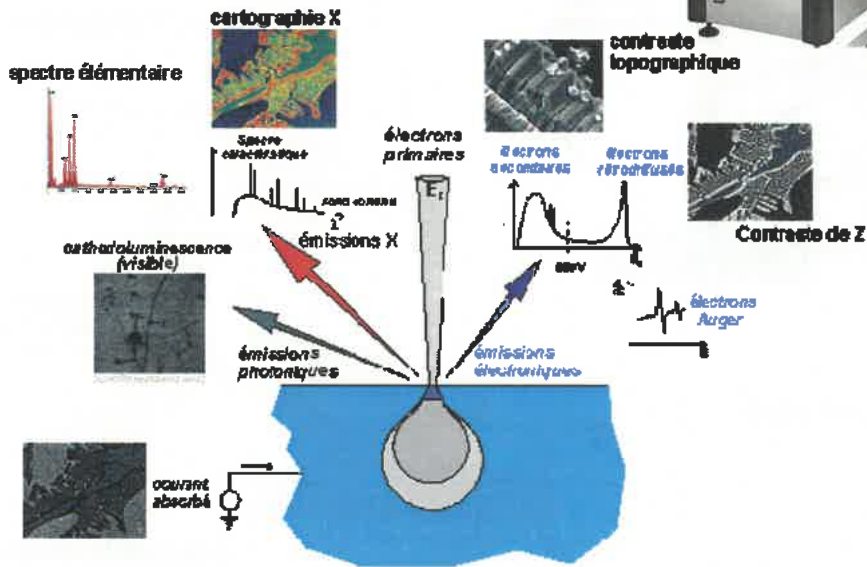


REM : reflection Electron Microscope

# The Scanning Electron Microscope (SEM)

An Amazing Tool for...

- Observation : SE – BSE
- Texture
- Chemical Analysis (EDS – WDS)
- Cathodoluminescence
- EBIC...

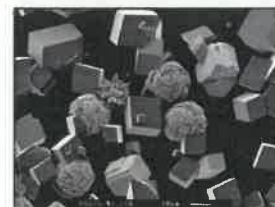
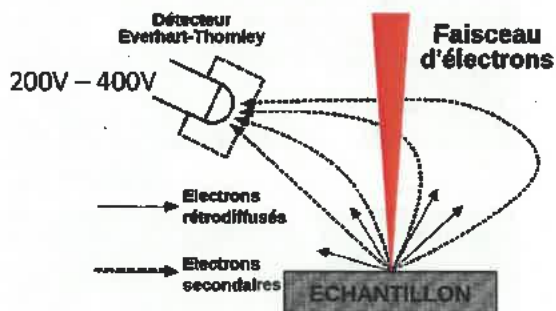
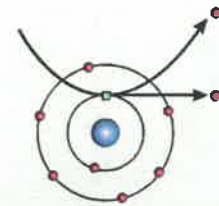


5

# The Scanning Electron Microscope (SEM)

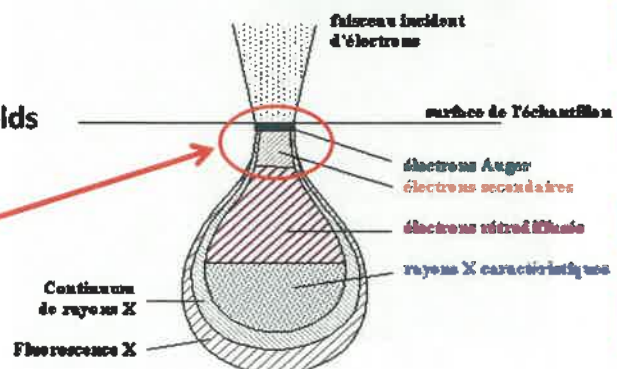
## Secondary electrons (SE)

- Generated as ionization products
- low energy (up to 50 eV)



Sensitive to electric and magnetic fields

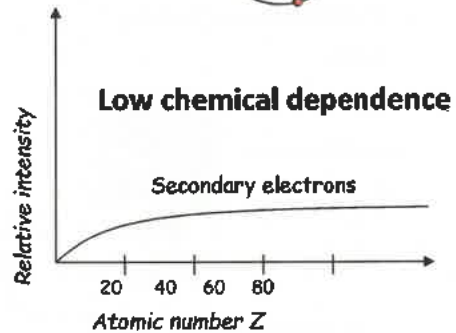
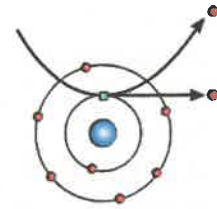
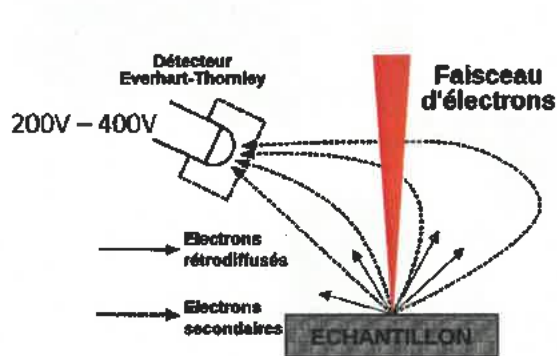
Only electrons emitted to the near surface reach the detector  
Surface electrons  
Resolution 1 nm à 15 keV  
(MEB FEG Zeiss Ultra Plus)



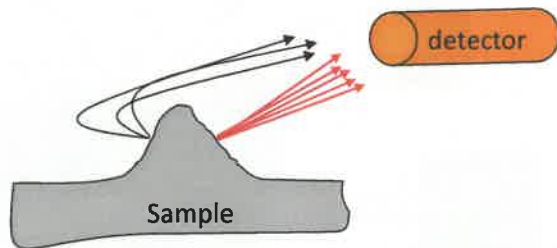
6

# The Scanning Electron Microscope (SEM)

## Secondary electrons (SE)



isotropic emission

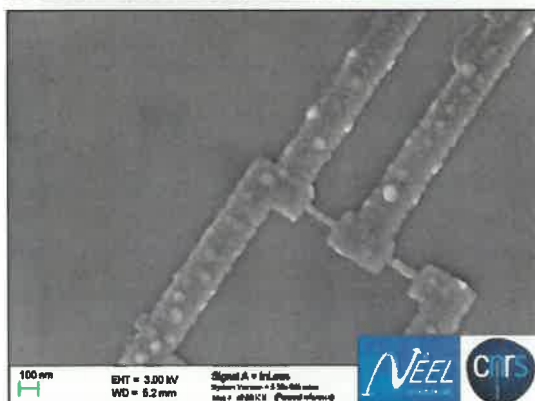


topographic contrast

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# The Scanning Electron Microscope (SEM)

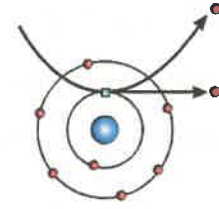
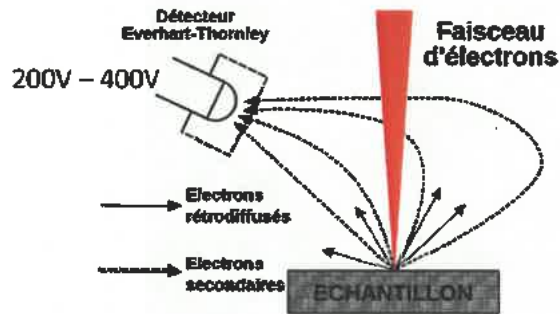
## Secondary electrons



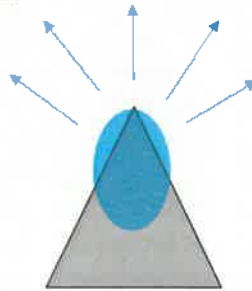
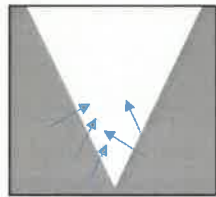
8

# The Scanning Electron Microscope (SEM)

## Secondary electrons (SE)



Side effect



ULP, Strasbourg

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# The Scanning Electron Microscope (SEM)

## Secondary electrons (SE)

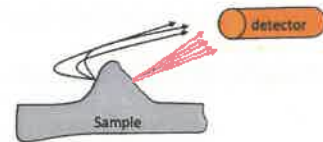


Image 1



Image 2

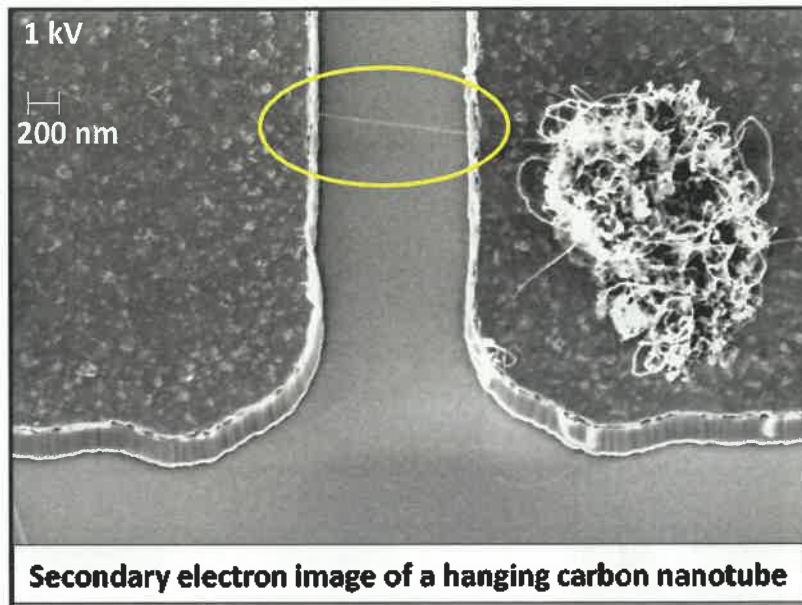
What do you see ?

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# The Scanning Electron Microscope (SEM)

## Secondary electrons (SE)

Morphological observations of conductive samples



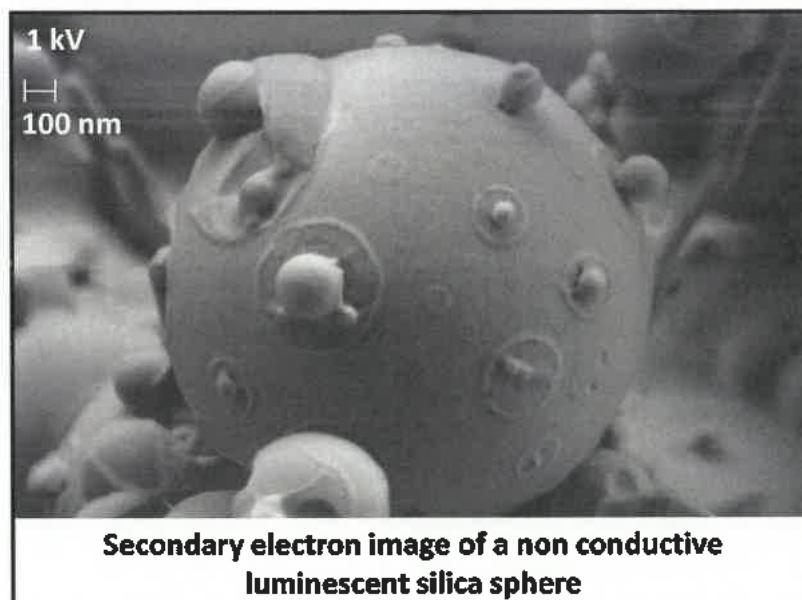
S. Paris

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# The Scanning Electron Microscope (SEM)

## Secondary electrons (SE)

Morphological observations of non conductive samples



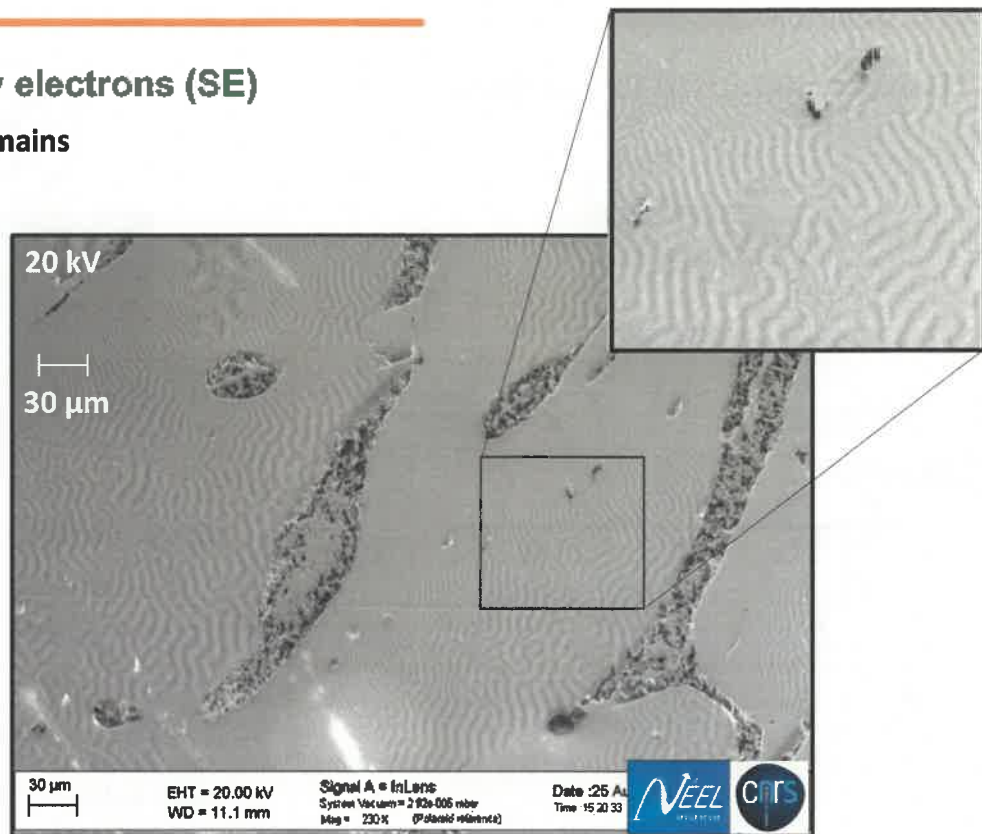
S. Paris

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# The Scanning Electron Microscope (SEM)

## Secondary electrons (SE)

Magnetic domains



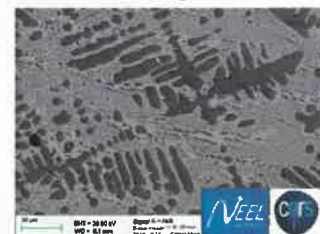
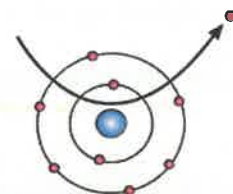
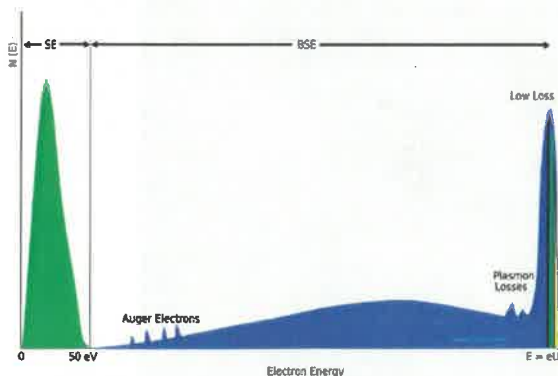
S. Pairis

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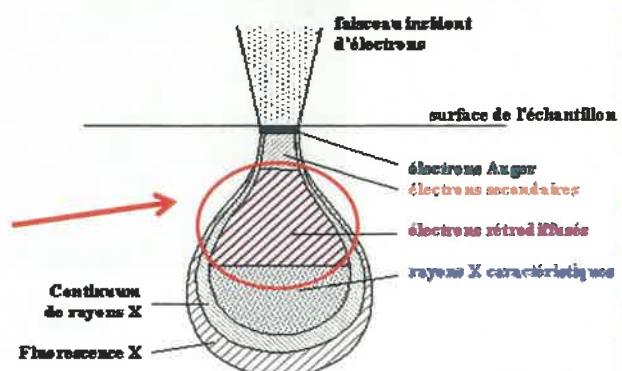
# The Scanning Electron Microscope (SEM)

## Backscattered electrons (BSE)

- Originating in the electron beam
- No energy loss (elastic diffusion)
- High energy



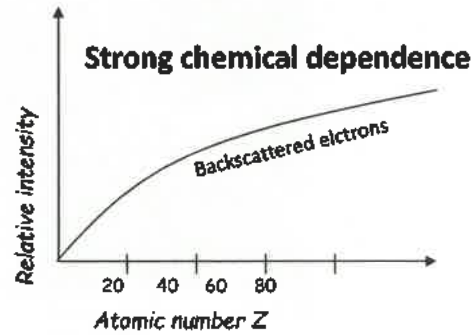
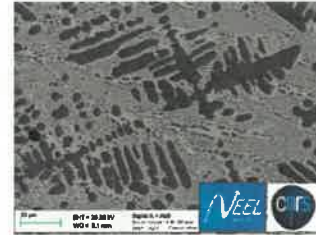
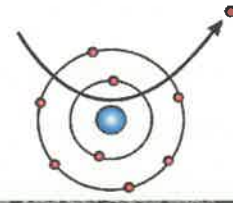
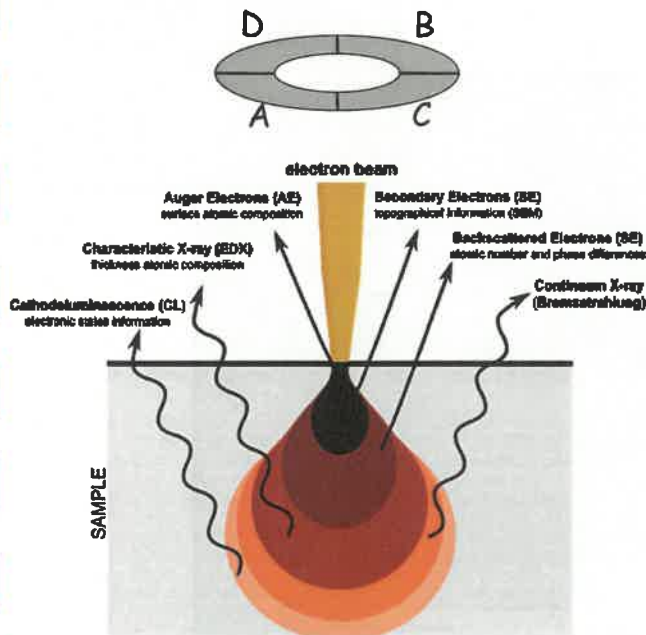
Backscattered electrons:  
penetration depth :  $10^{\text{th}}$  -  $100^{\text{th}}$  nm or more  
=> Resolution > 1 nm à 15 keV  
(MEB FEG Zeiss Ultra Plus)



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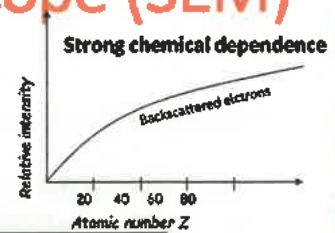
# The Scanning Electron Microscope (SEM)

## Backscattered electrons (BSE)

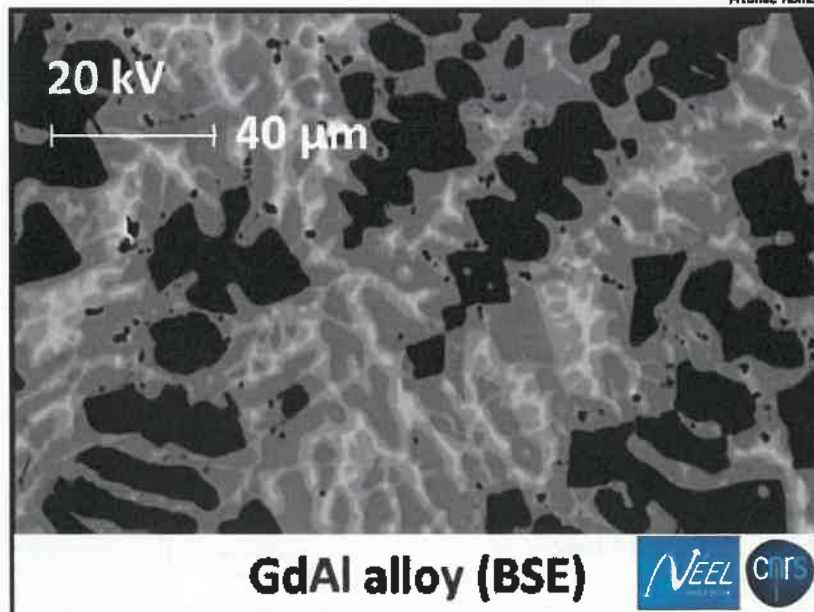


# The Scanning Electron Microscope (SEM)

## Backscattered electrons (BSE): chemical contrast



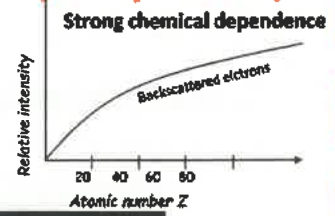
Al : Z = 13  
Gd : Z = 64





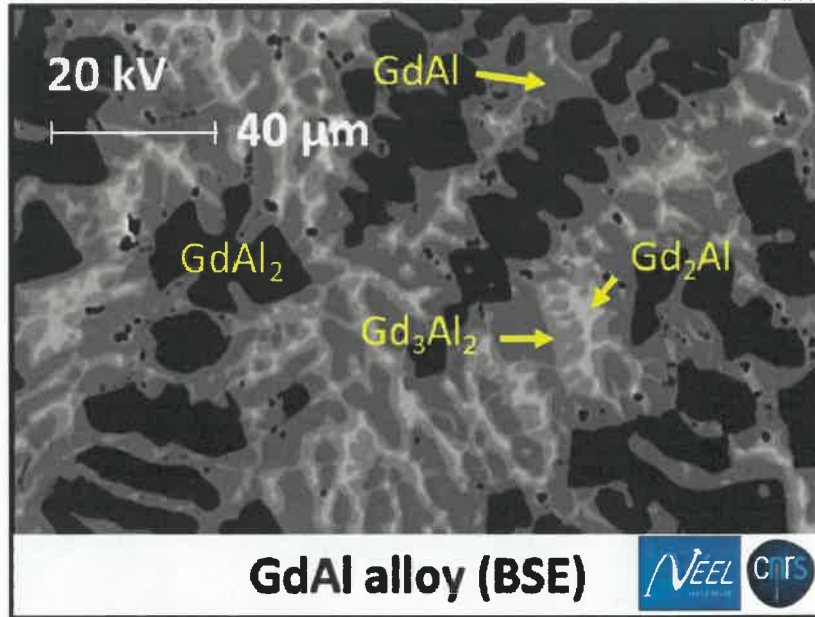
# The Scanning Electron Microscope (SEM)

## Backscattered electrons (BSE): chemical contrast



Al : Z = 13  
Gd : Z = 64

$Z(\text{Gd}_2\text{Al}) = 47$   
 $Z(\text{Gd}_3\text{Al}_2) = 43,6$   
 $Z(\text{GdAl}) = 38,5$   
 $Z(\text{GdAl}_2) = 30$



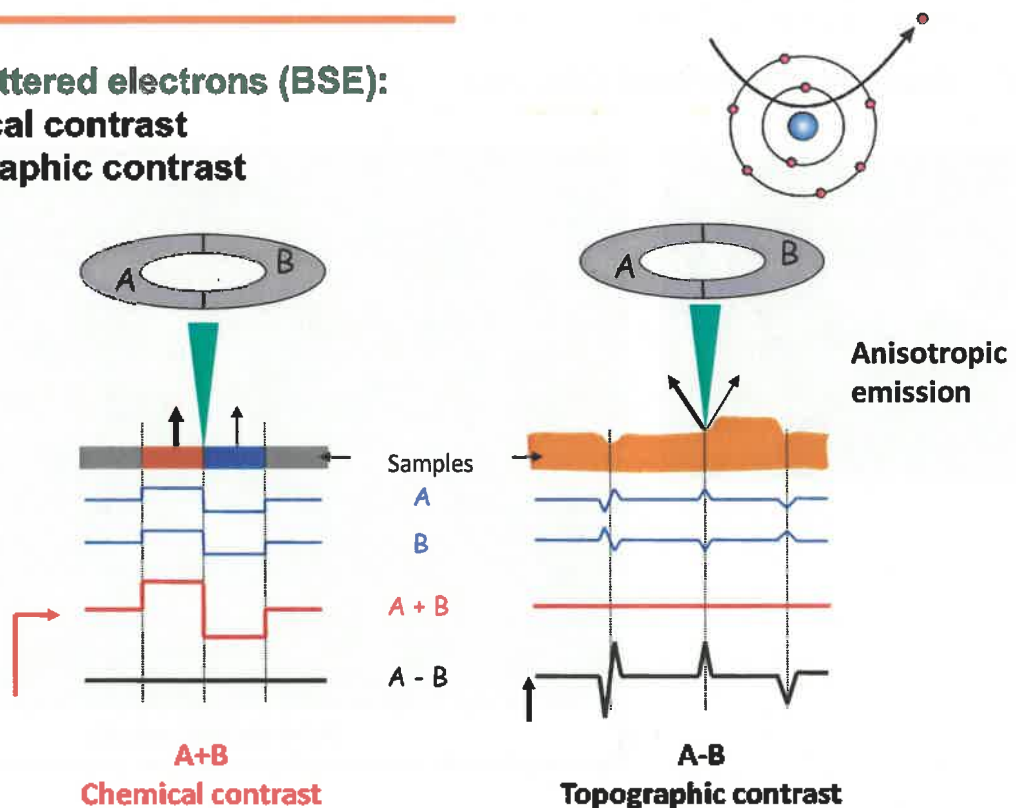
S. Pairis

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# The Scanning Electron Microscope (SEM)

## Backscattered electrons (BSE):

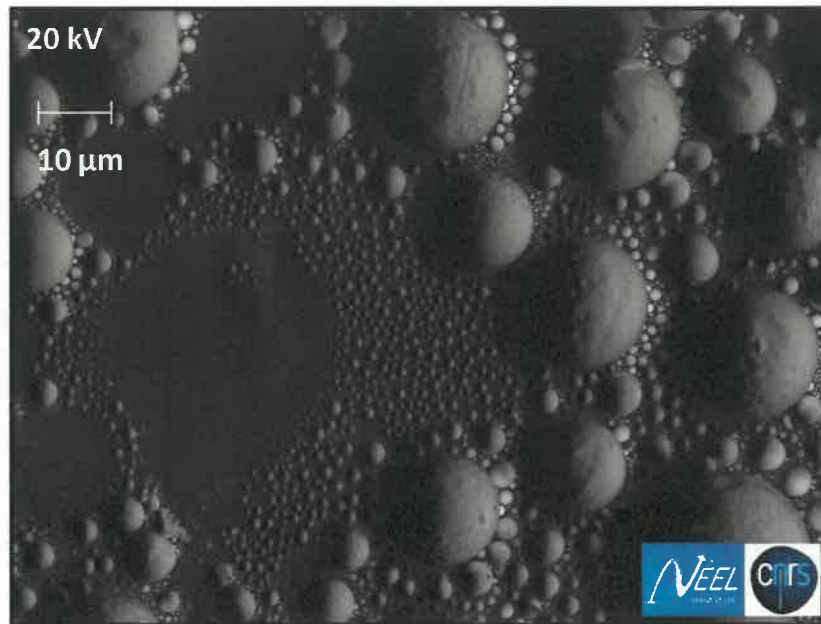
- Chemical contrast
- Topographic contrast



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# The Scanning Electron Microscope (SEM)

**Backscattered electrons (BSE): topographic contrast**

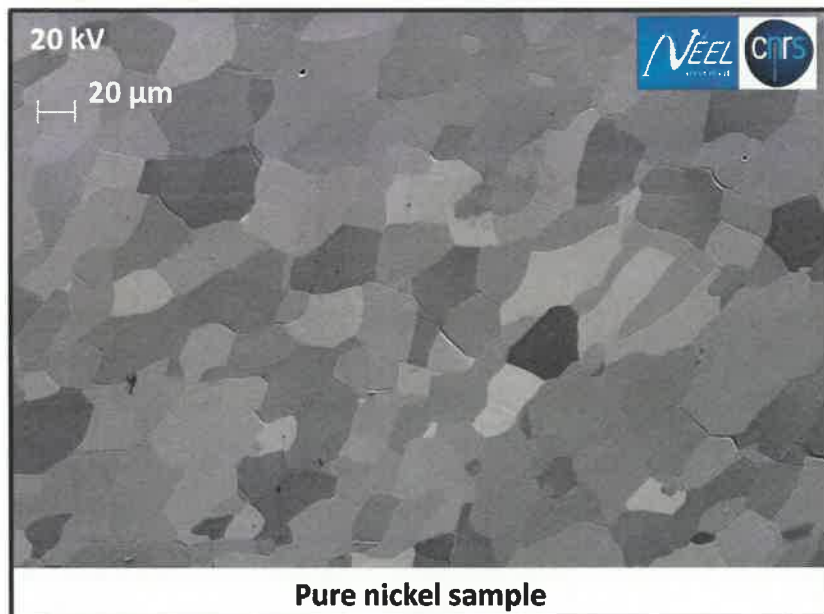
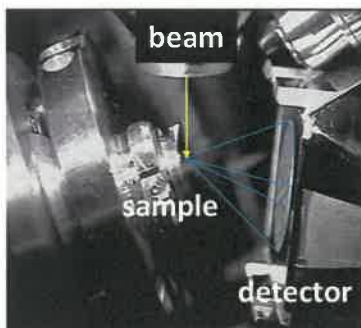


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# The Scanning Electron Microscope (SEM)

**Electron BackScattered Diffraction (EBSD): cristallographic orientation**



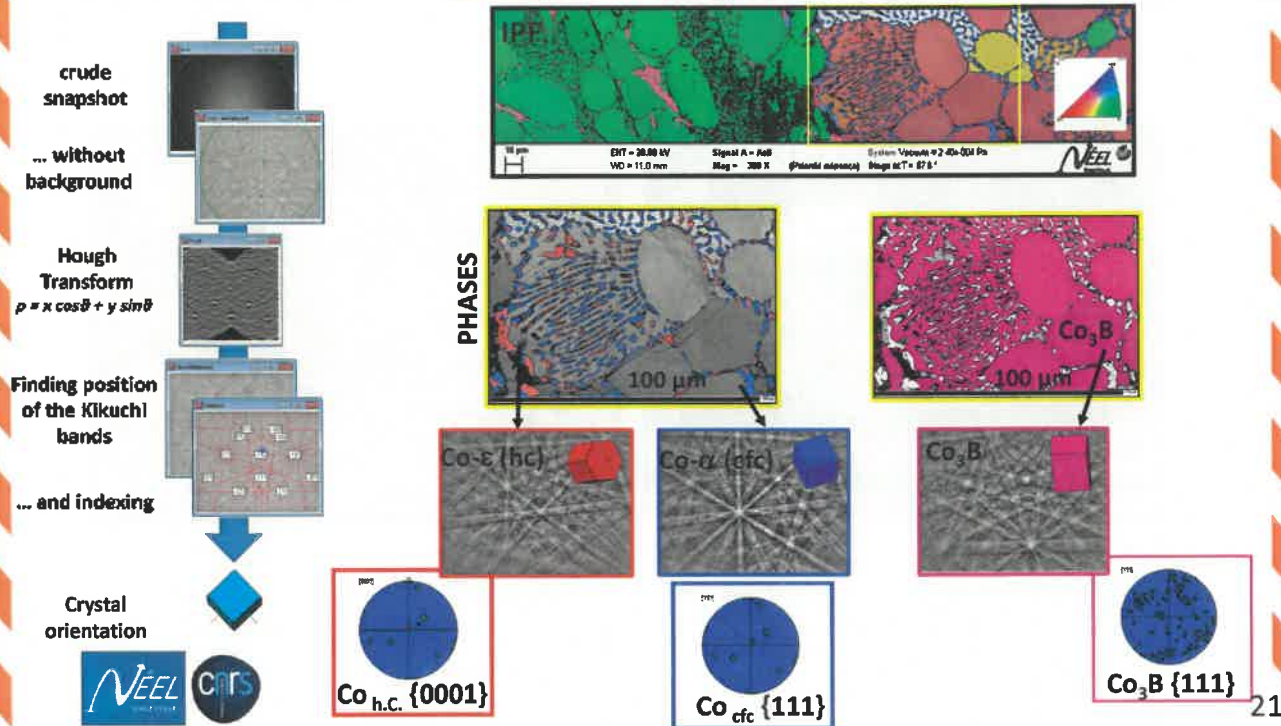
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# The Scanning Electron Microscope (SEM)

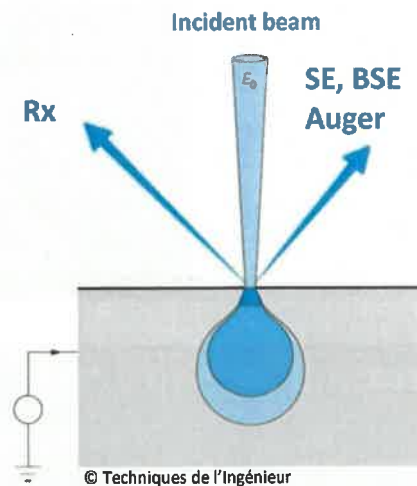
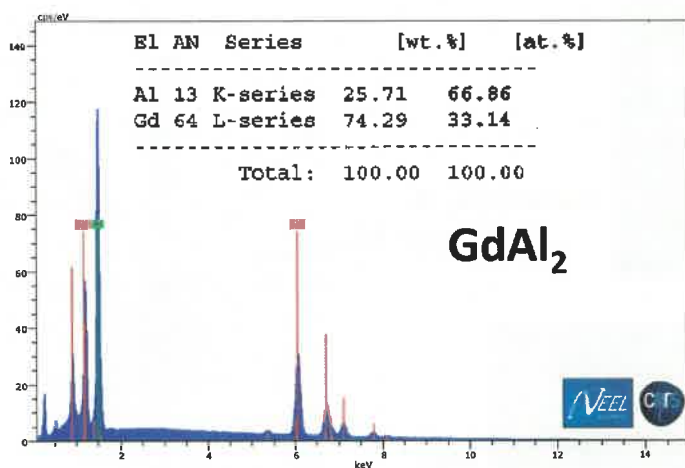
## Electron BackScattered Diffraction (EBSD): microstructural analysis (texture)

Temperature-induced structure transition in a liquid Co-B eutectic alloy [Yixuan He Jinshan Li Jun Wang Eyub Yildiz Sébastien Pairis Eric Beaugnon Materials Letters, Elsevier, 2019, 234, pp.351-353. \(10.1016/j.matlet.2018.09.138\)](#)



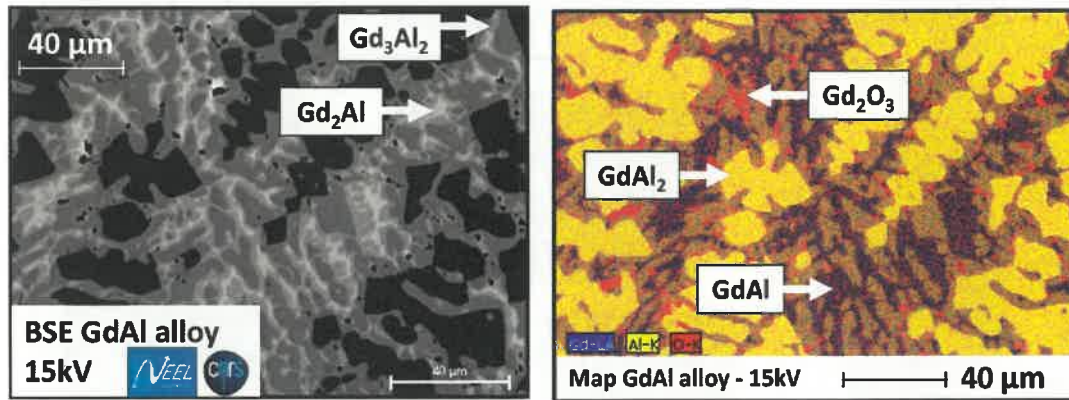
# The Scanning Electron Microscope (SEM)

## Chemical analysis: Energy Dispersive Spectroscopy (or WDS)



# The Scanning Electron Microscope (SEM)

Chemical analysis: Energy Dispersive Spectroscopy (or WDS)  
Hypermap (spectra on each pixel)

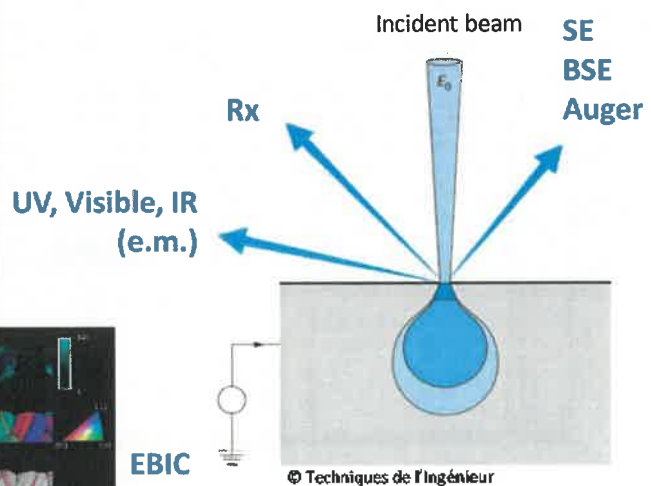


S. Pairis

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# The Scanning Electron Microscope (SEM)

Cathodoluminescence and Electron Beam Induced Current (EBIC)

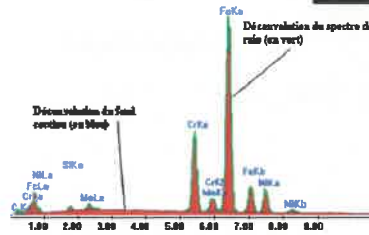
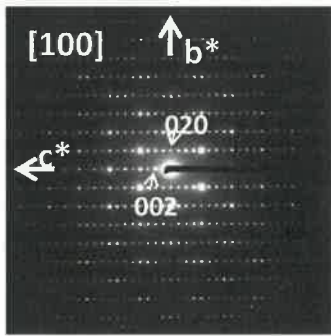
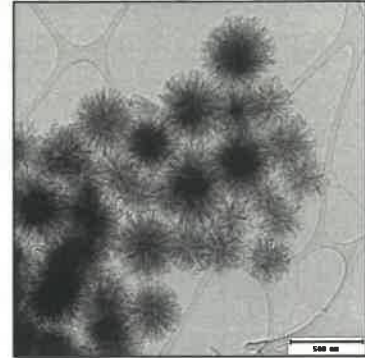
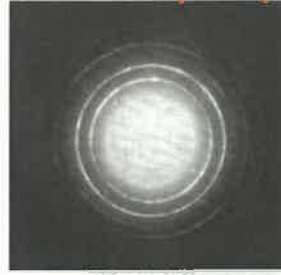
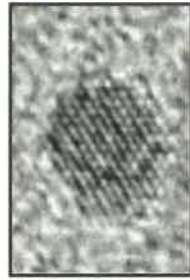
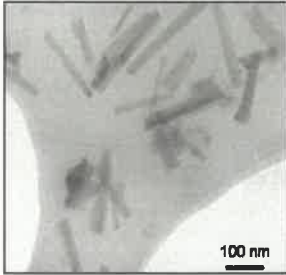


Dr. J. Poplawsky, Oak Ridge National Laboratory

S. Pairis

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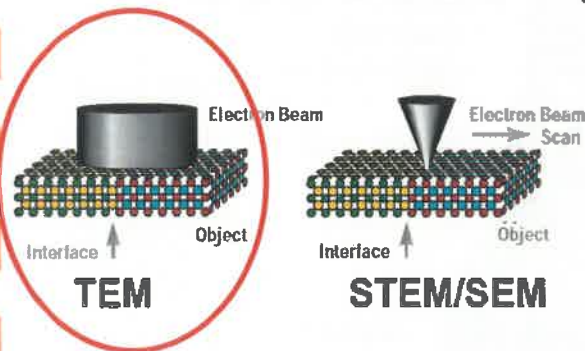
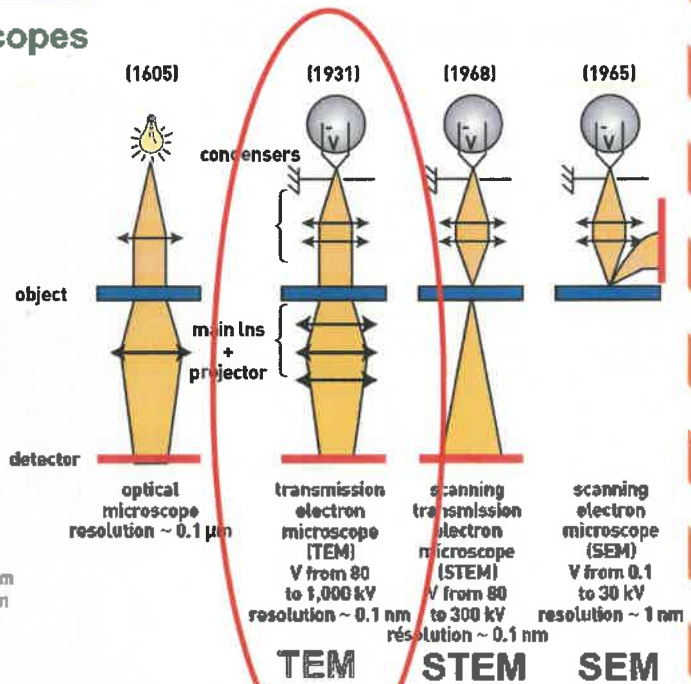
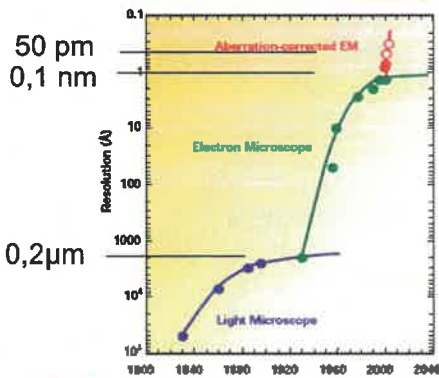
# The Transmission Electron Microscope (TEM)



30

# The Transmission Electron Microscope (TEM)

## Optical and electron microscopes



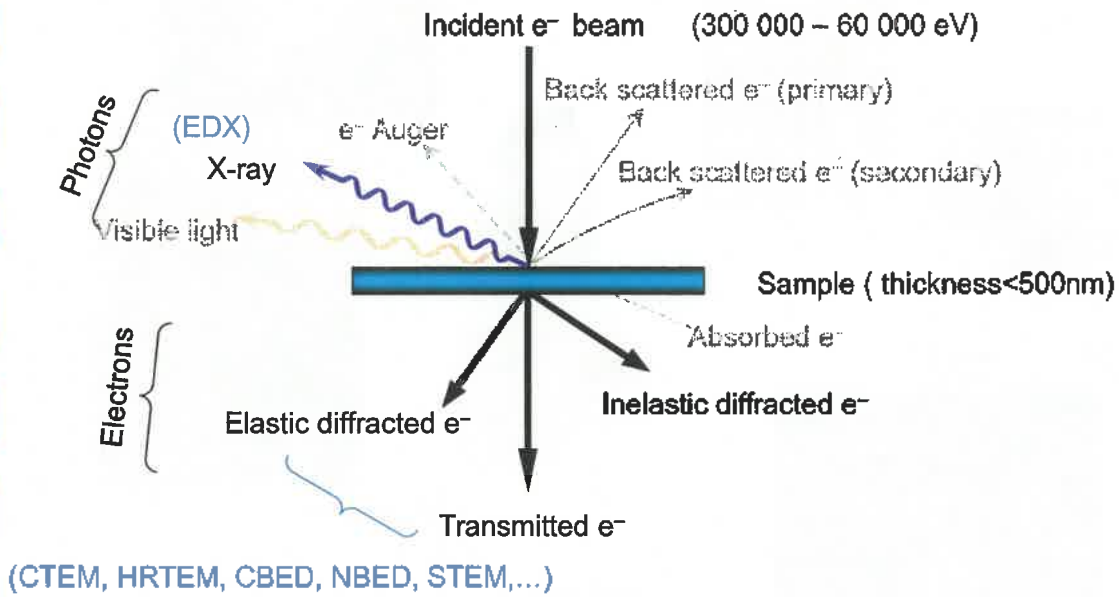
Extracted from a journal (clefs CEA n° 52) on nanostructures : <http://www.cea.fr/var/cea/storage/Static/gb/library/Clefs52/contents.htm>

REM : reflection Electron Microscope

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# The Transmission Electron Microscope (TEM)

## What is it ?

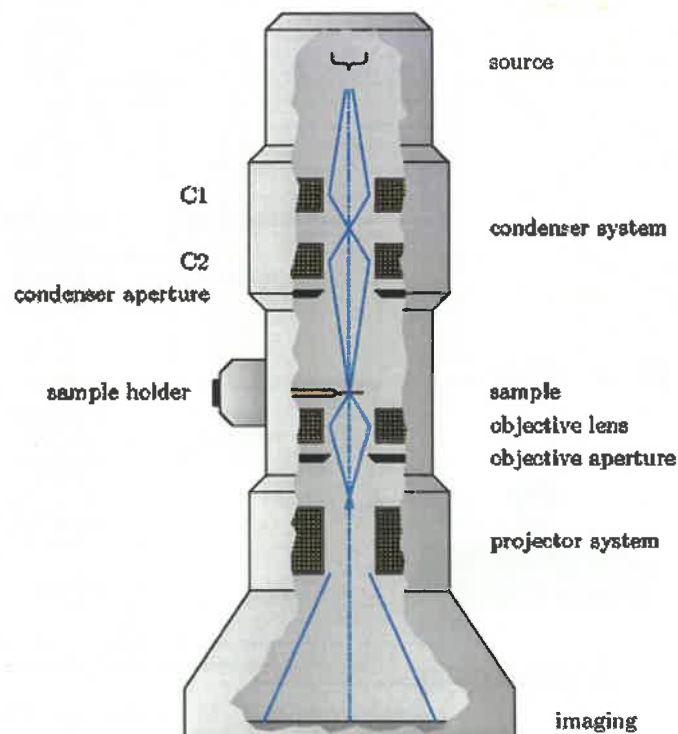


M. Den Hertog

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# The Transmission Electron Microscope (TEM)

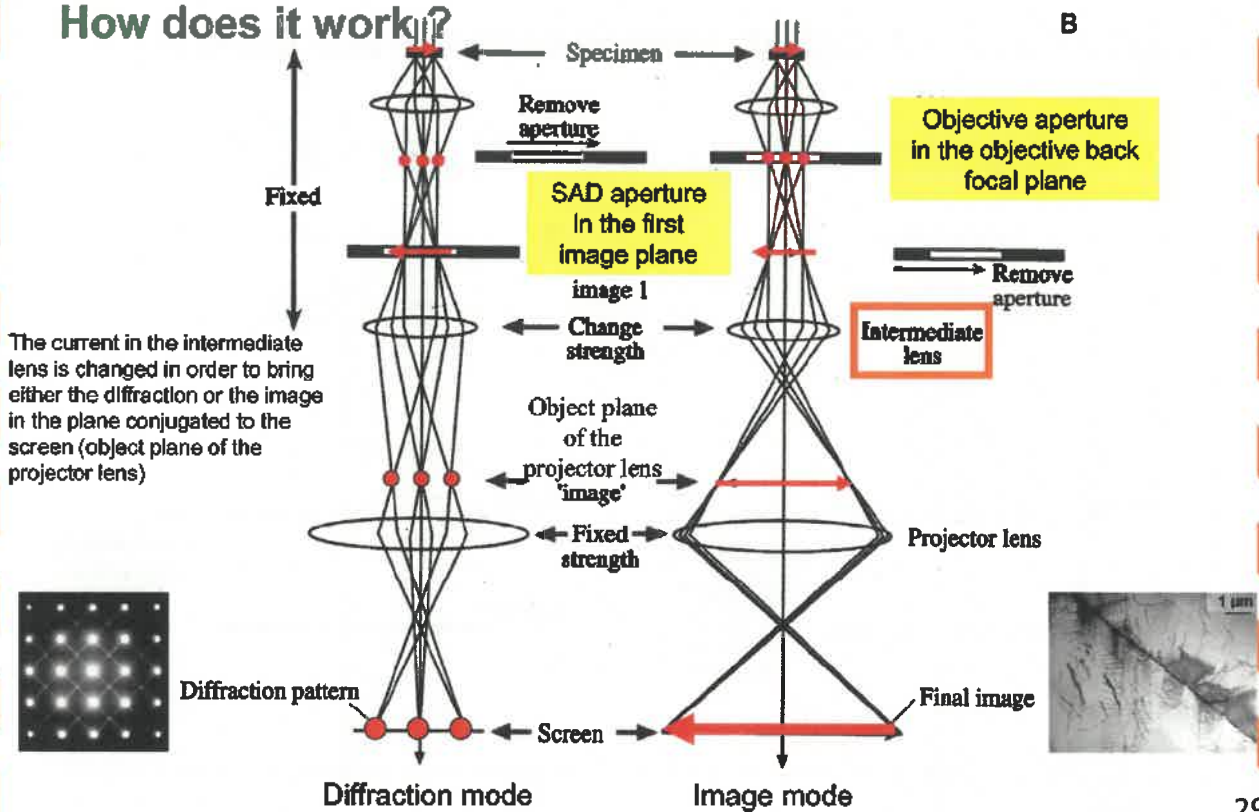
## How does it work ?



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# The Transmission Electron Microscope (TEM)

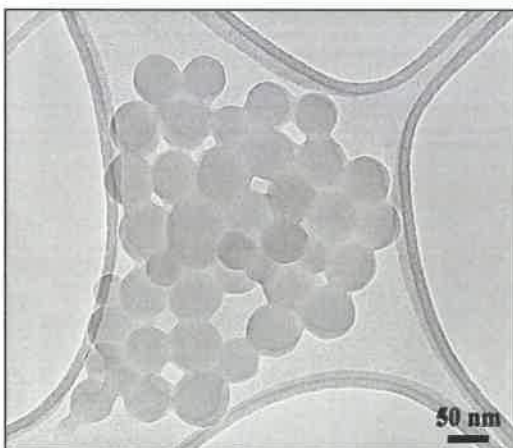
## How does it work?



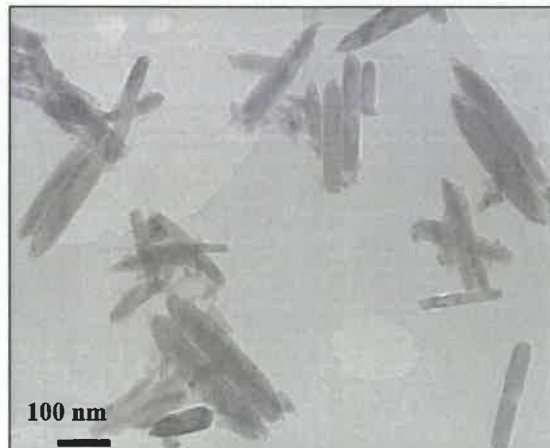
29

# The Transmission Electron Microscope (TEM)

## Conventional imaging: morphology



Amorphous latex particles

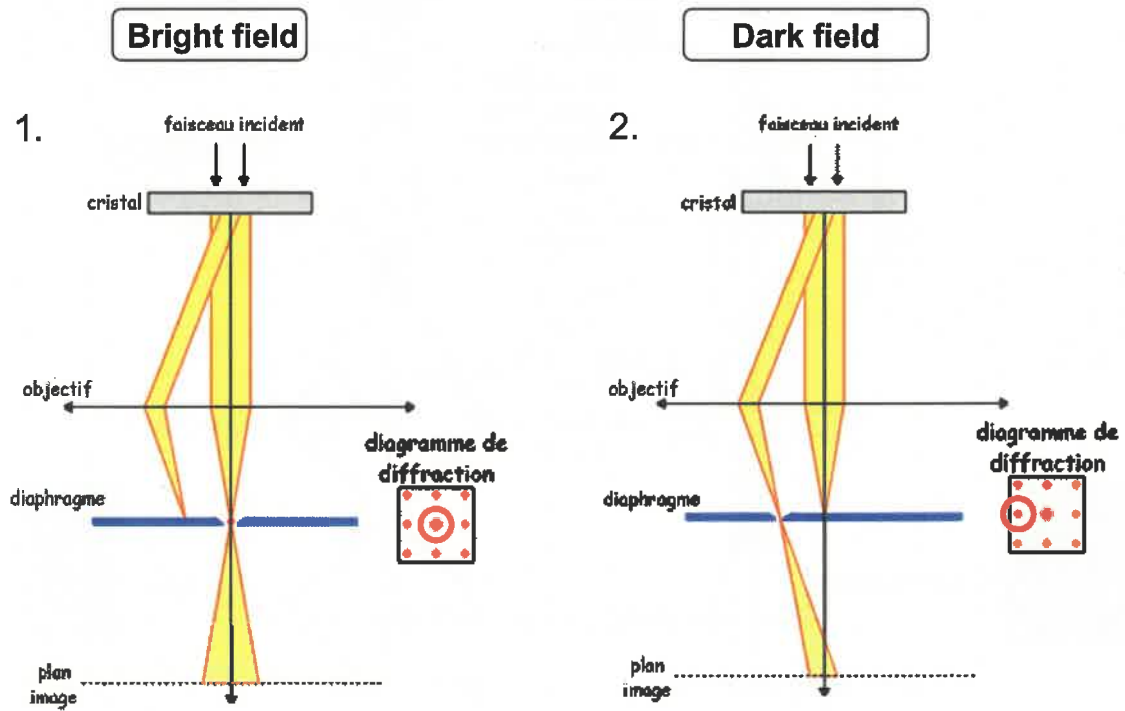


Cristallized particles of akaganeite  $\beta$ -FeOOH

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# The Transmission Electron Microscope (TEM)

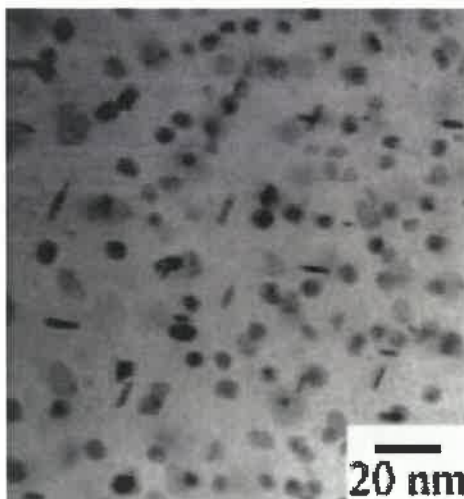
## Conventional imaging: How to enhance the contrast ?



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# The Transmission Electron Microscope (TEM)

## Conventional imaging: bright field



AlZnMg alloy  
2 types de precipitates,  
globular / platelets.



Evangelista et al.,  
Can. Metal. Qu. 43, 339 (04)

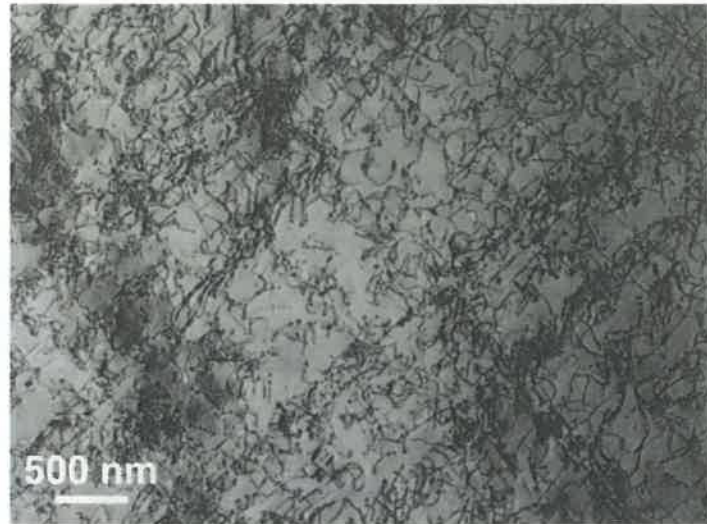
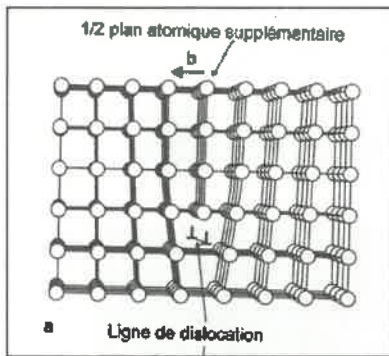
**Crystallized: diffraction contrast**  
**Amorphous: diffusion contrast**

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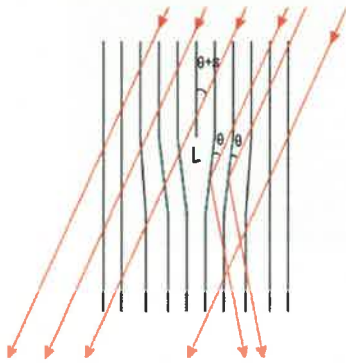


# The Transmission Electron Microscope (TEM)

## Conventional imaging: bright field



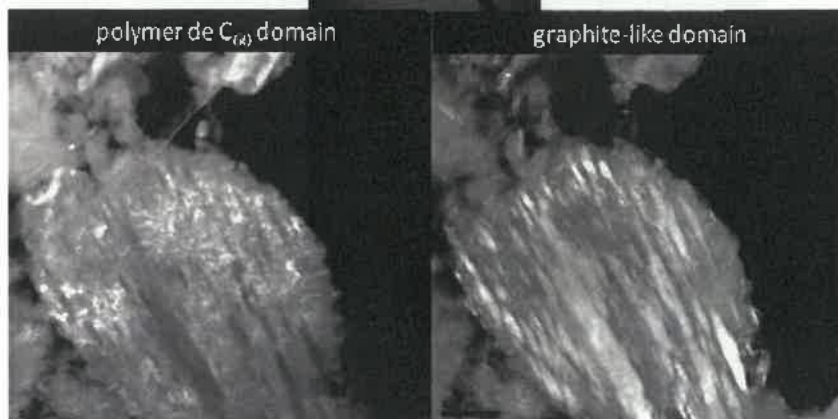
Dislocations in a Ni-Fe-Mn-Nb alloy



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# The Transmission Electron Microscope (TEM)

## Conventional imaging: dark field



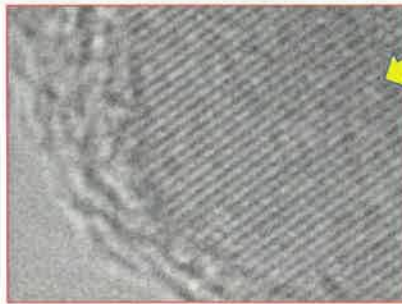
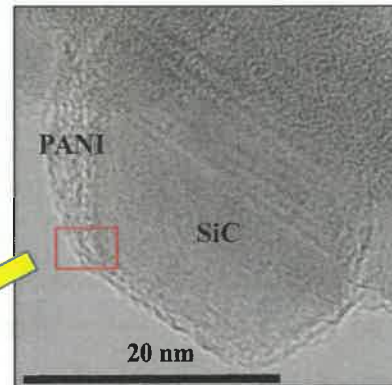
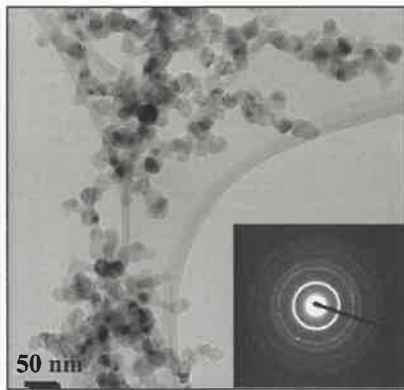
2 phases "graphite-like" and polymer C<sub>60</sub> coexist in the same particle.  
C. LEPOITTEVIN and M. ALVAREZ-MURGA

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# The Transmission Electron Microscope (TEM)

## Conventional imaging to High resolution imaging

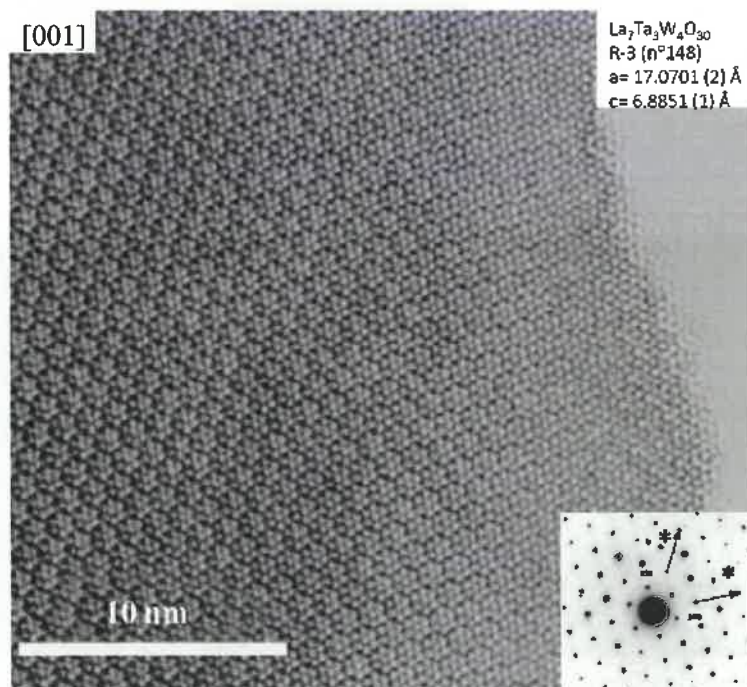
SiC Nanoparticles



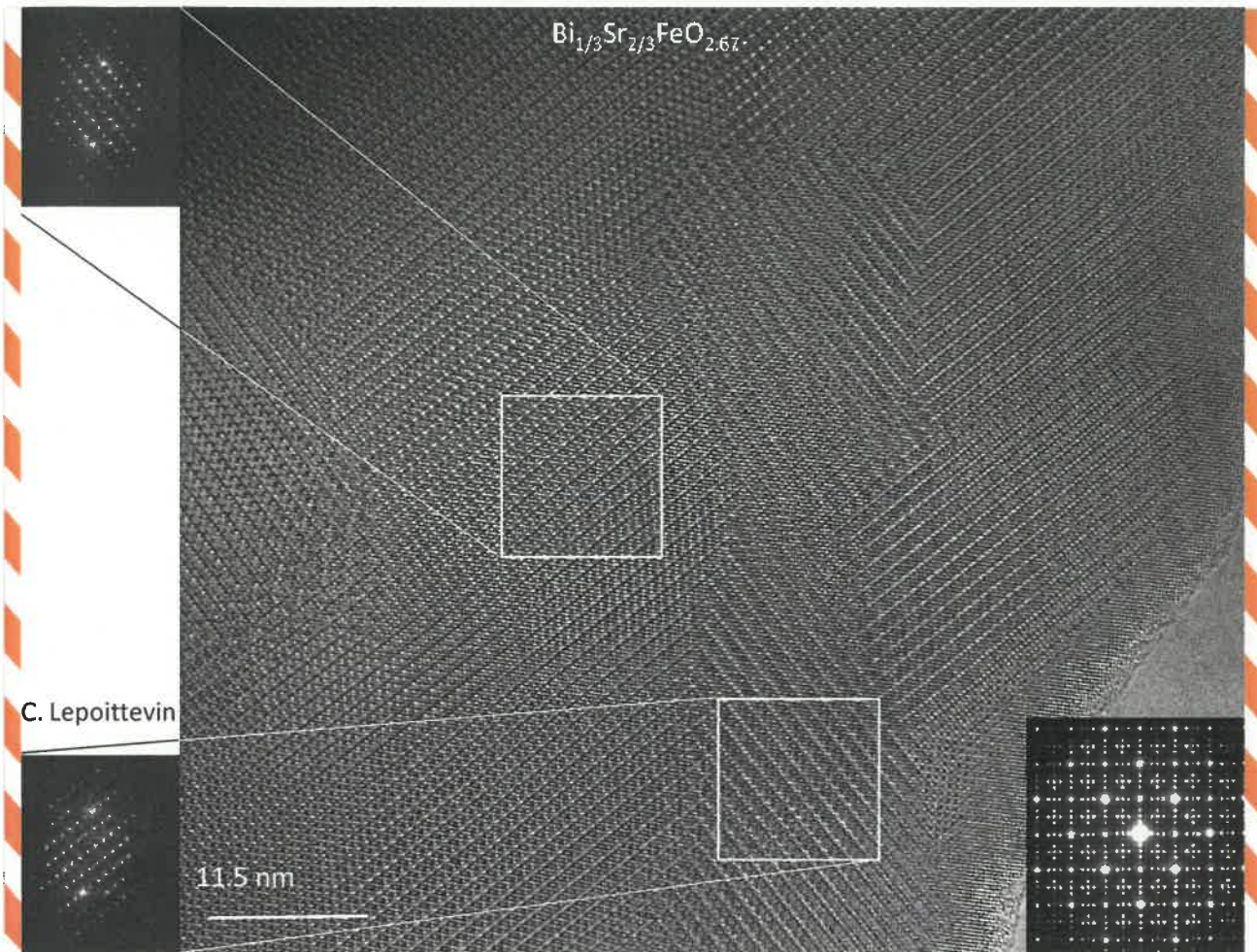
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# The Transmission Electron Microscope (TEM)

## High resolution imaging

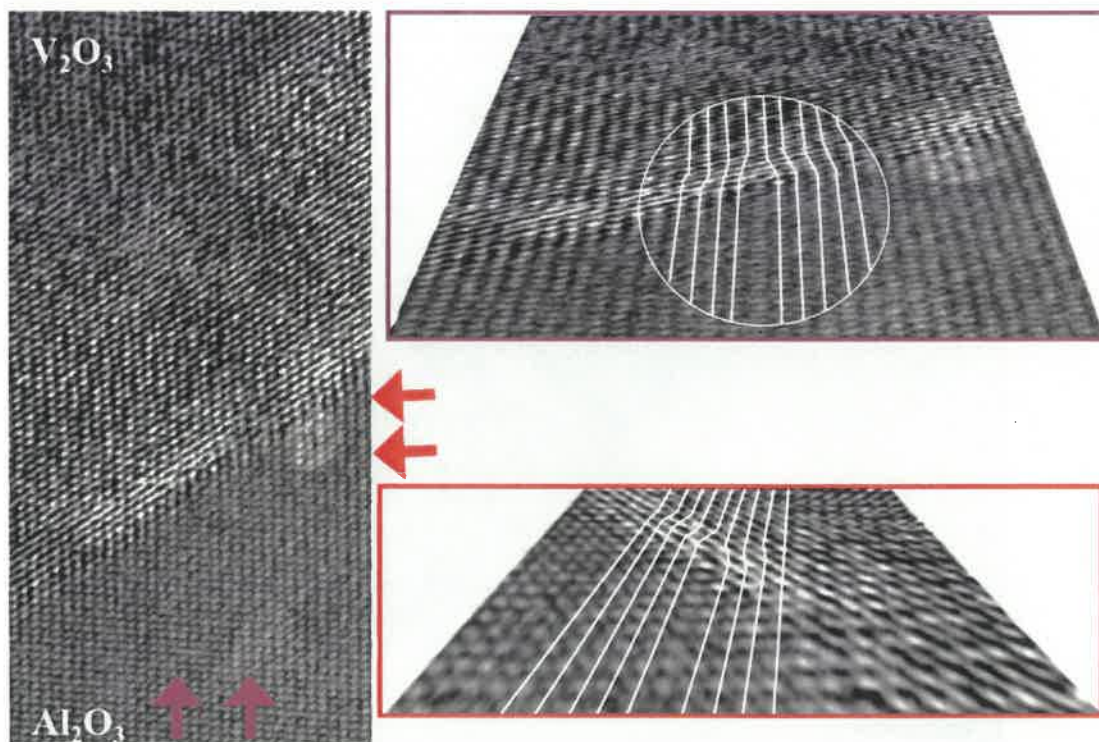


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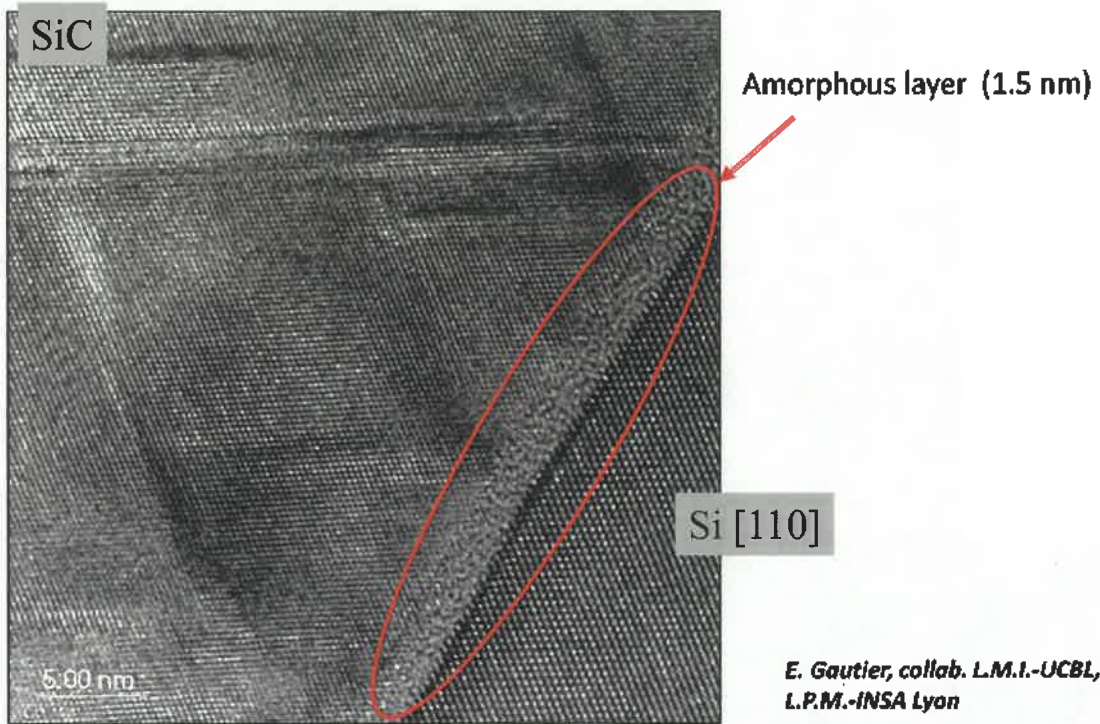
## The Transmission Electron Microscope (TEM)

### High resolution imaging



# The Transmission Electron Microscope (TEM)

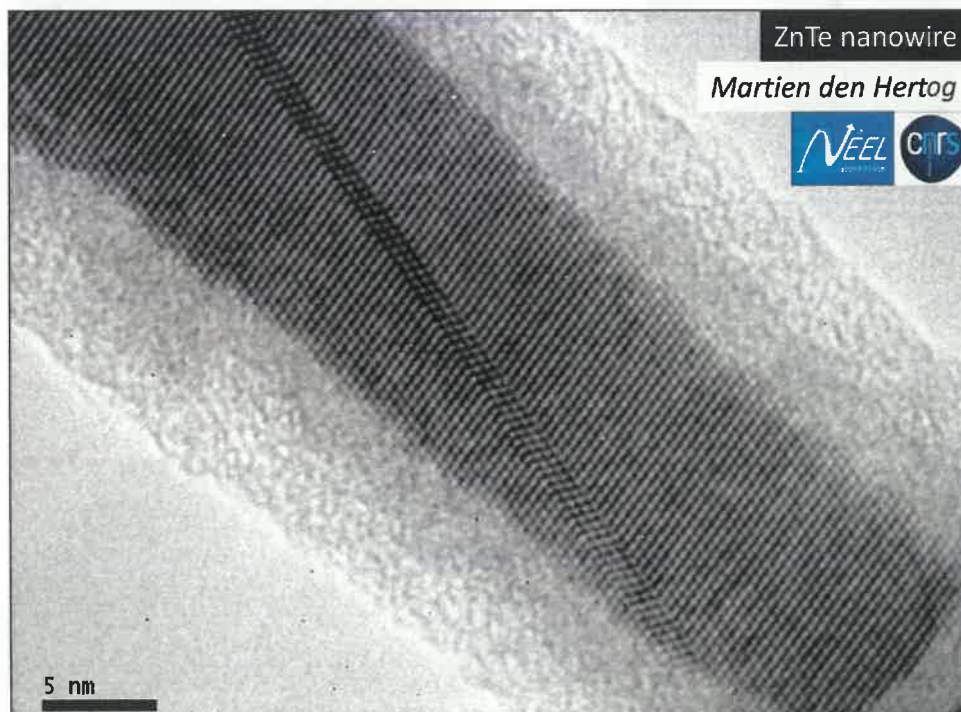
## High resolution imaging



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# The Transmission Electron Microscope (TEM)

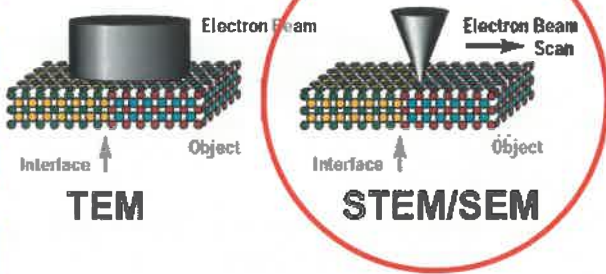
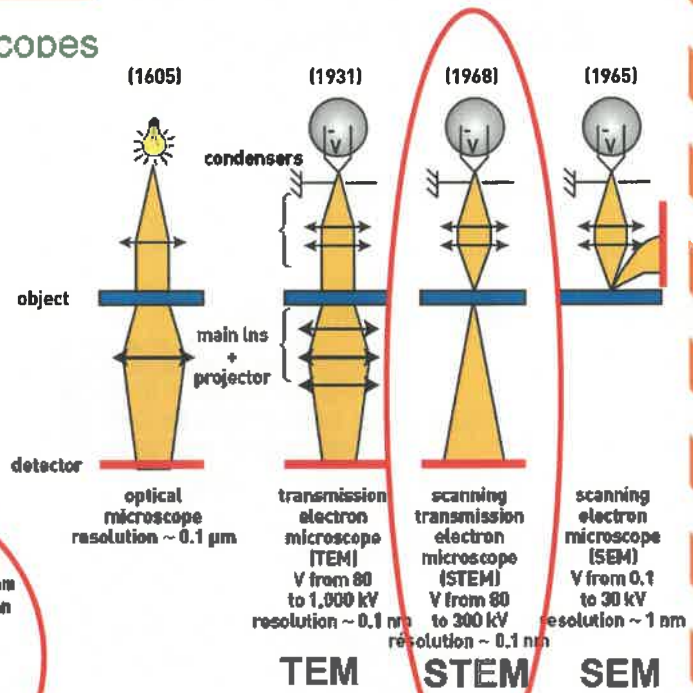
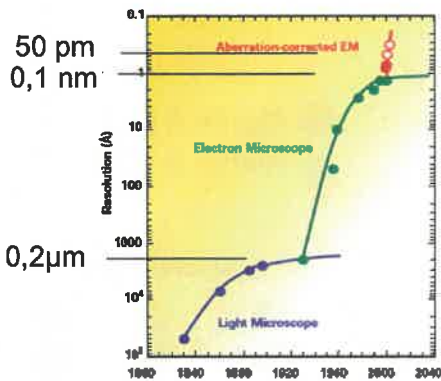
## High resolution imaging



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# The Scanning Transmission Electron Microscope (STEM)

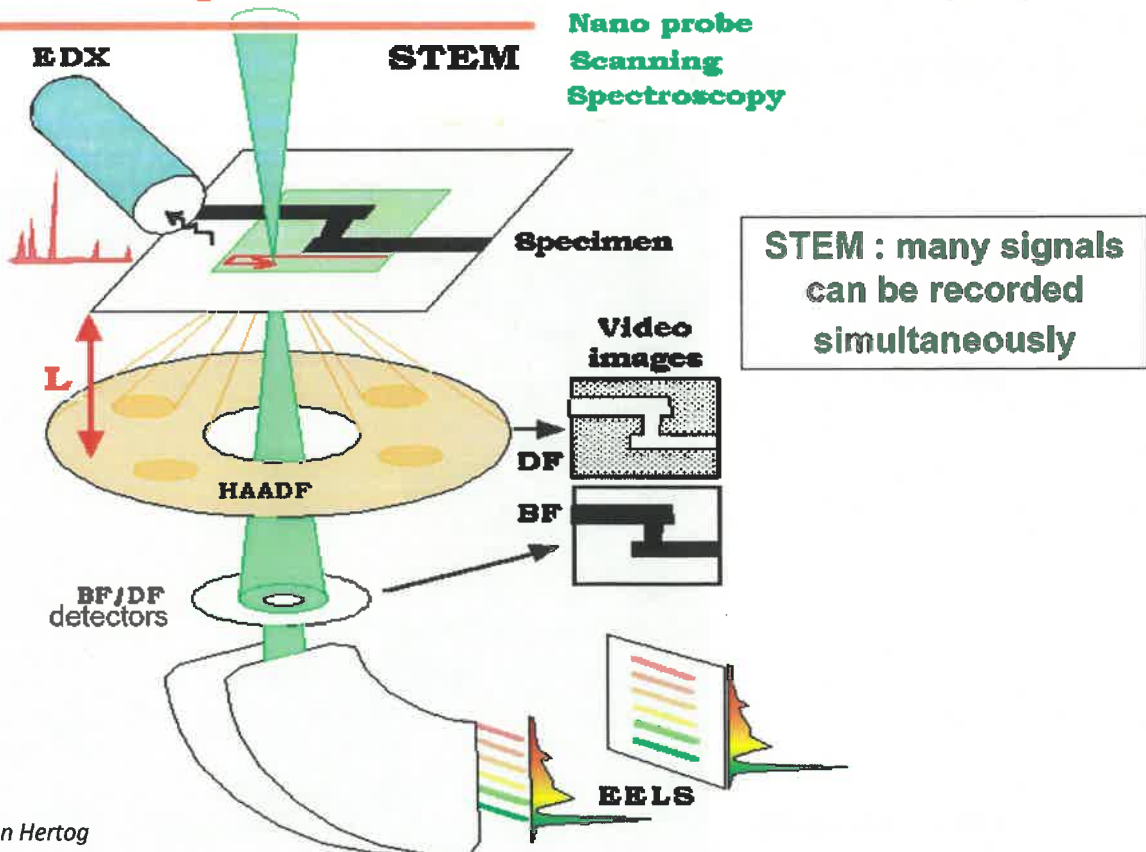
## Optical and electron microscopes



Extracted from a journal (clefs CEA n° 52) on nanostructures : <http://www.cea.fr/var/cea/storage/attachments/library/Clefs52/contents.htm>

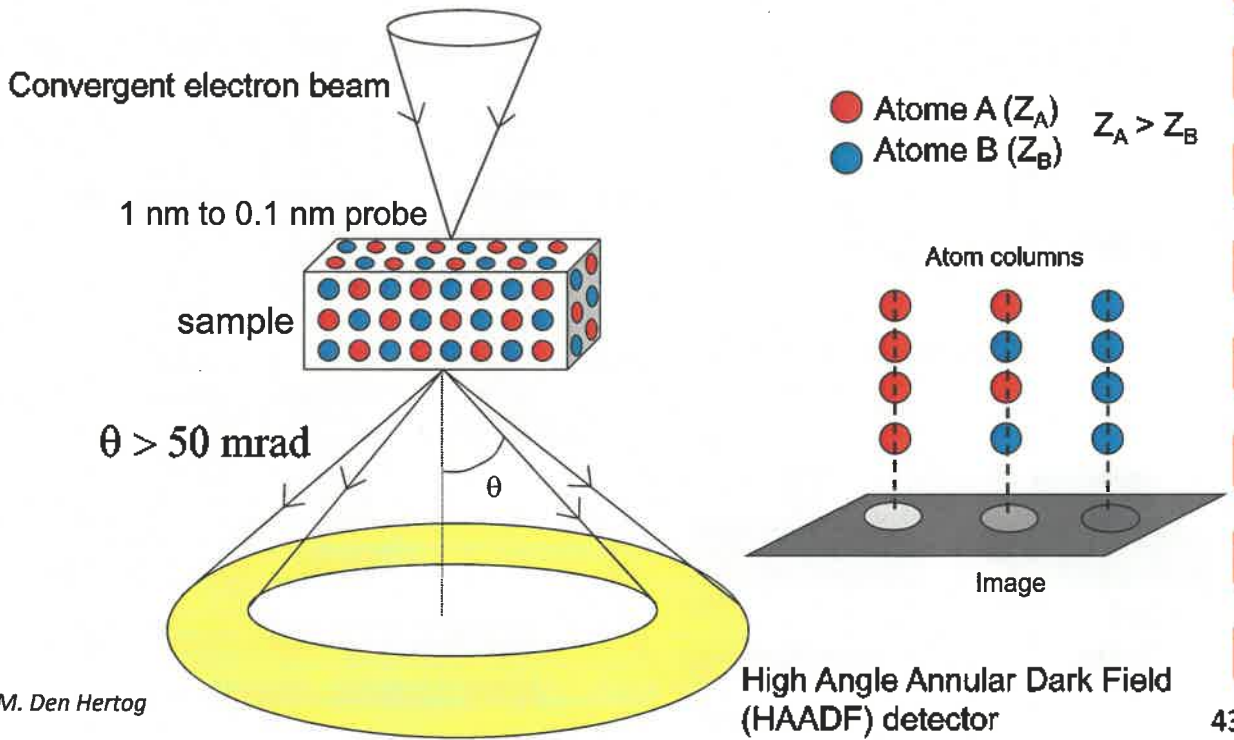
REM : reflection Electron Microscope

# The Scanning Transmission Electron Microscope (STEM)



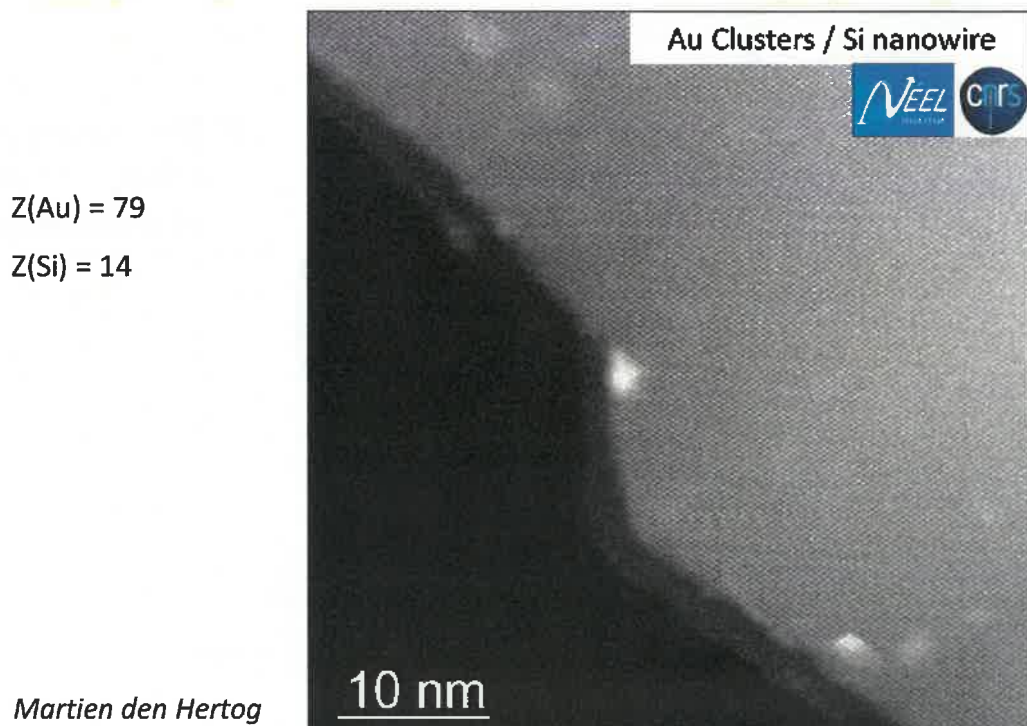
# The Scanning Transmission Electron Microscope (STEM)

## Z contrast imaging



# The Scanning Transmission Electron Microscope (STEM)

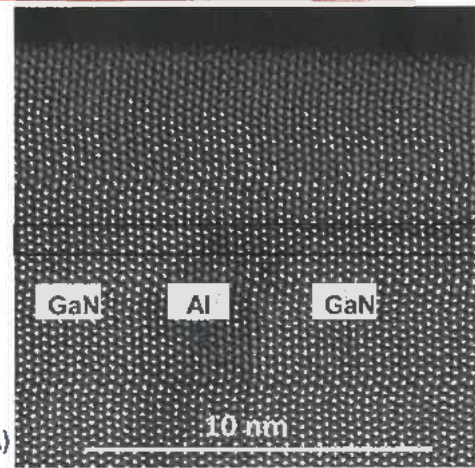
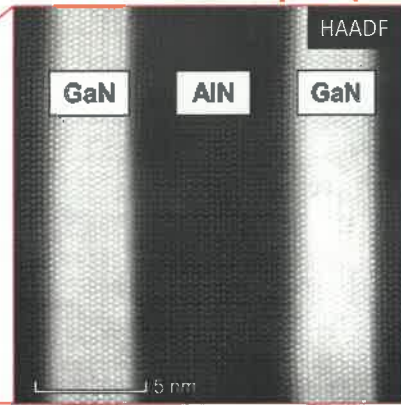
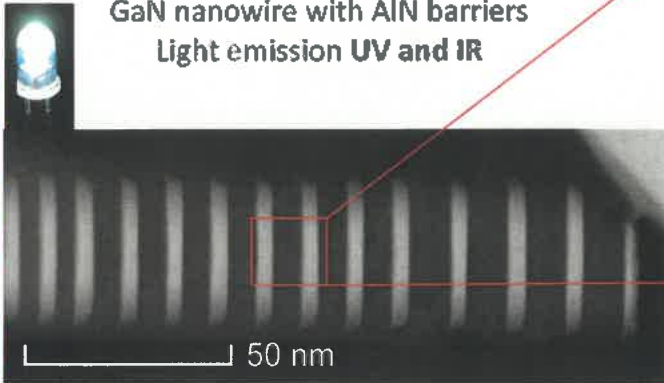
## Z contrast imaging



# The Scanning Transmission Electron Microscope (STEM)

## Z contrast imaging

GaN nanowire with AlN barriers  
Light emission UV and IR



### Challenges:

- Understand opto-electrical properties

### Information:

Size, orientation of NW, heterostructure, interfaces

### Perspectives:

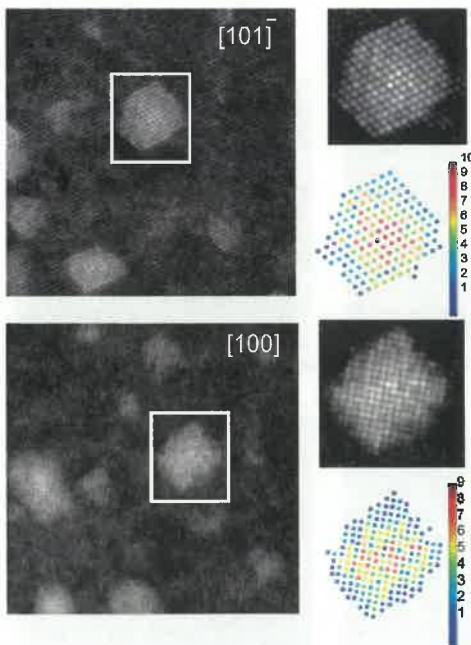
- Single atom spectroscopy (Mn, rare earths)
- Dopant characterization (EDX – holography)



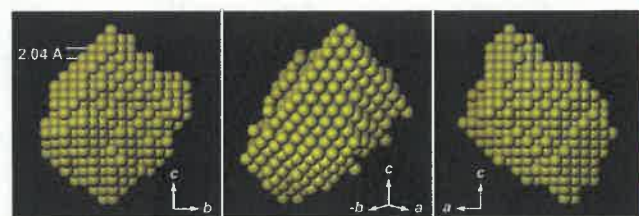
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# The Scanning Transmission Electron Microscope (STEM)

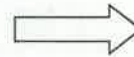
## Z contrast imaging: 3D reconstruction



## Ag nanoparticles in Al

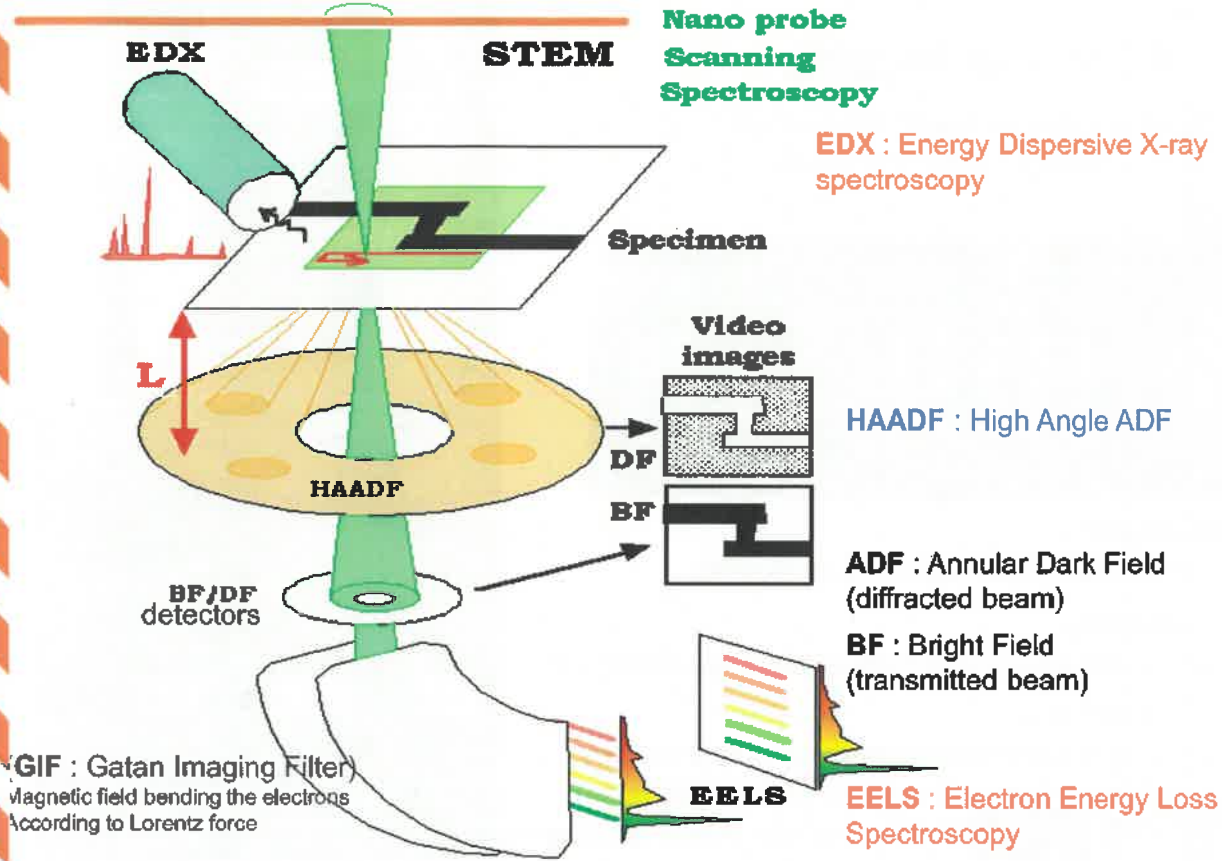


HR STEM HAADF  
2 orientations  
of the same particle



3D reconstruction  
of Ag nanoclusters

# The Scanning Transmission Electron Microscope (STEM)



# The Scanning Transmission Electron Microscope (STEM)

## Chemical analysis at atomic resolution: EDX

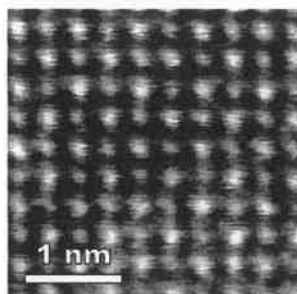
Energy Dispersive X-Ray Spectroscopy at each beam position → chemical map

### NaLaCoWO<sub>6</sub> (multiferroic material)

- Data storage
- Sensors
- Spintronics



HAADF-STEM



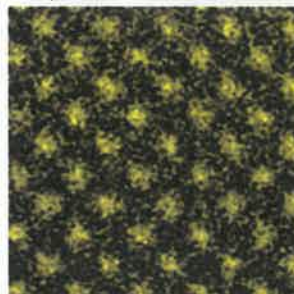
Na, EDX



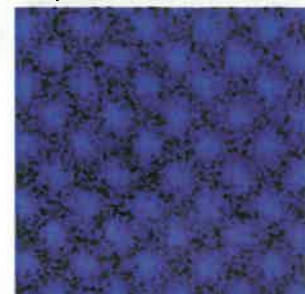
Co, EDX



La, EDX



W, EDX

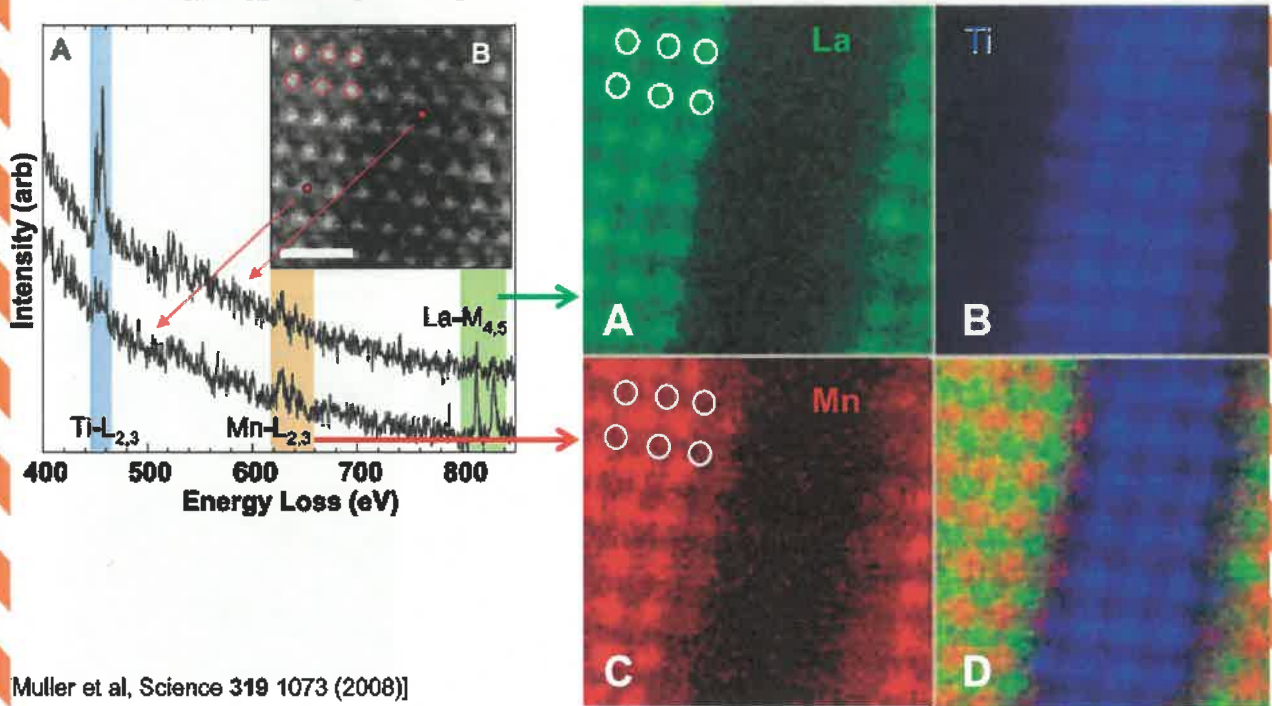




# The Scanning Transmission Electron Microscope (STEM)

## Chemical analysis at atomic resolution: EELS

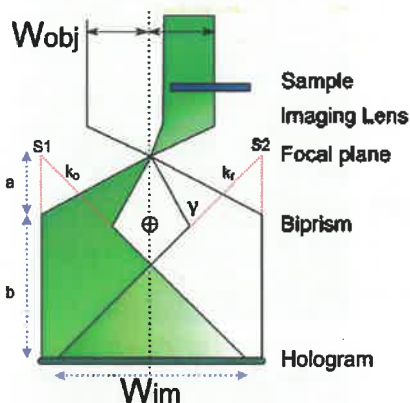
- Electron Energy Loss spectrum (EELS) at each beam position → chemical map
- $\text{La}_{0.7}\text{Sr}_{0.3}\text{MnO}_3/\text{SrTiO}_3$  interface



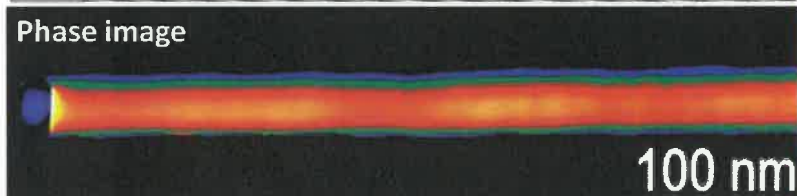
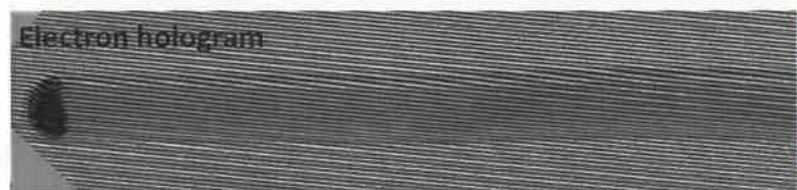
Muller et al, Science 319 1073 (2008)]

# The Scanning Transmission Electron Microscope (STEM)

## Imaging of electric and magnetic fields: holography



⇒ doping, magnetic domains (nano / optoelectronics)



### Challenges:

- Quantitative electrical / magnetic characterization

### Perspectives:

→ In-situ electrical TEM

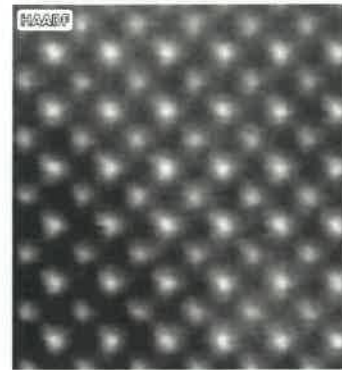
# The Scanning Transmission Electron Microscope (STEM)

## Summary

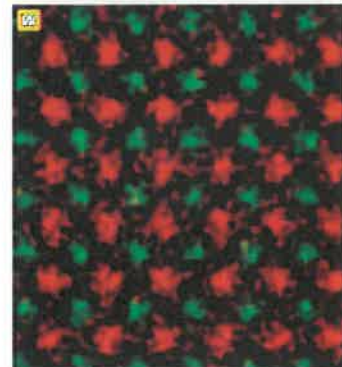
TEM : a bunch of techniques

- 1) Visualisation of the object and measurement of its **sizes** (layer thickness, cluster size)
- 2) Visulation of **defects** (linear and planar) and phase (volume) in 2D or 3D
- 3) **Crystal characterisation** (symmetry, polarity, atomic structure ...or amorphous)
- 4) **Chemical composition**
- 5) **Atomic structure** determination (interface, quasicrystal, which atom is where)
- 6) Measurement of **local lattice parameters** (translation, rotation, **strain**)
- 7) **Magnetic and electric fields** in-side and around the object
- 8) In-situ experiments (heating, cooling, growth, electrical ...)

high resolution STEM



Chimical analysis (EDX)



W  
La

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# The Scanning Transmission Electron Microscope (STEM)

## Summary

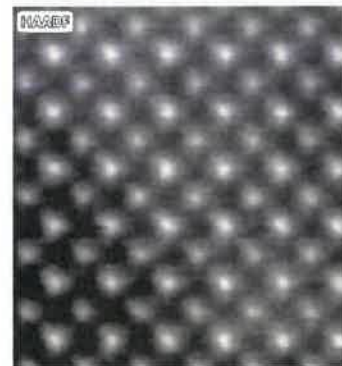
- Atomic Resolution
- Chemical Information
- Study of crystals and defects
- Access to electric and magnetic fields
- In-situ (temperature, in situ electrical biasing)

→ Indispensable tool for Nanoscience

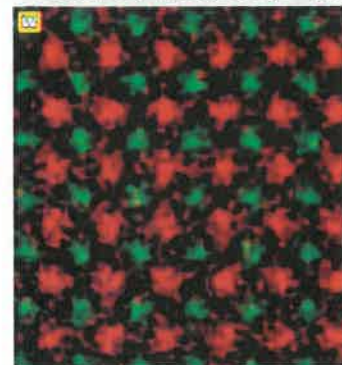
Project new TEM: MEET@Néel



high resolution STEM



Chimical analysis (EDX)

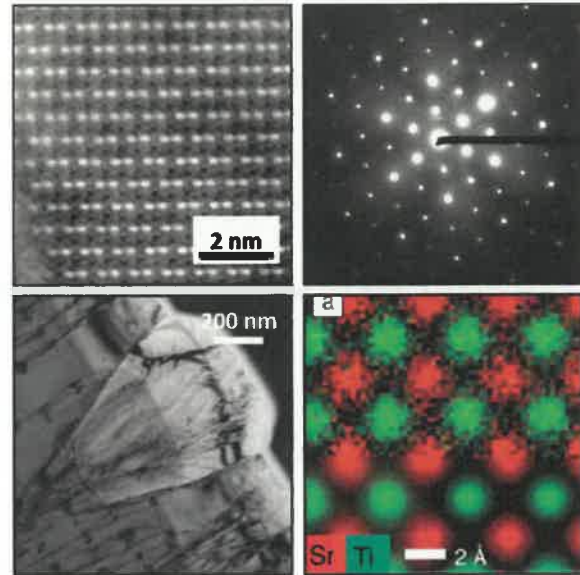


W  
La

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Acquisition of a state of the art STEM

- academic research projects
- industrial technological developments
- associated training operations



Probe corrected STEM, HAADF, ABF, EDX, EELS, Precession, Biprism



JEOL  
JEM-ARM200F NEOARM  
Cold FEG Cs STEM

Operating: april 2022

Characterisations at a very low scale: microstructure mapping down to nanometric / atomic scale:

- crystallography
- chemical composition
- quantitative analysis of light elements

Characterisation of thin films: thicknesses, surfaces and interfaces, adherence, diffusion of elements, doping, crystalline quality, preferential orientation / epitaxy, mapping of elements at the atomic scale

Measurements of local electric and magnetic fields at a nanometric scale

*In situ* studies with temperature or electric stimuli

3 D tomography analysis:

- grain atomic structure
- size of crystalline domains

# 3D Electron Diffraction at Institut Néel

## THE LOW-DOSE ELECTRON DIFFRACTION TOMOGRAPHY METHOD (LD-EDT)

S. Kodjikian, H. Klein



### Physical properties

Electrical conduction



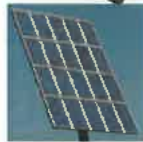
Thermal conduction



Magnetism



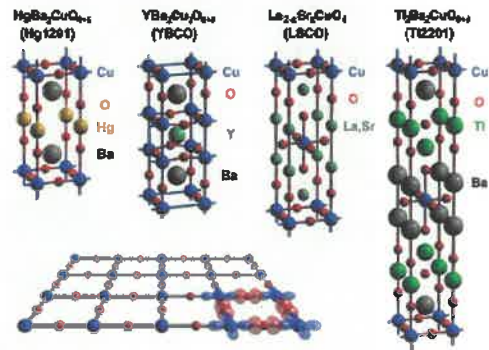
Energy



Optics



### Atomic structure

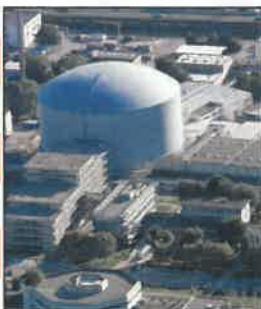


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# 3D Electron Diffraction at Institut Néel

## THE LOW-DOSE ELECTRON DIFFRACTION TOMOGRAPHY METHOD (LD-EDT)

Electrons interact strongly with matter, an extraordinary advantage for studying nano-sized materials ...



Neutrons  
1 mm



Laboratory X-Rays  
100 μm



Synchrotron X-Rays  
1 μm



TEM  
100 nm



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# 3D Electron Diffraction at Institut Néel

## THE LOW-DOSE ELECTRON DIFFRACTION TOMOGRAPHY METHOD (LD-EDT)

S. Kodjikian and H. Klein, 2019, *Ultramicroscopy*, 200, 12-19, <https://doi.org/10.1016/j.ultramic.2019.02.010>

**ELECTRON DIFFRACTION TOMOGRAPHY:**  
a non-oriented crystal is tilted around the goniometer axis in small steps.

Electron beam  
Precession  
+50°  
Rotation of the crystal (step=1°)  
Non-oriented crystal  
-50°

NEEL CNRS

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# 3D Electron Diffraction at Institut Néel

## THE LOW-DOSE ELECTRON DIFFRACTION TOMOGRAPHY METHOD (LD-EDT)

Acquisition (ex : 100 ms)

Non oriented crystal

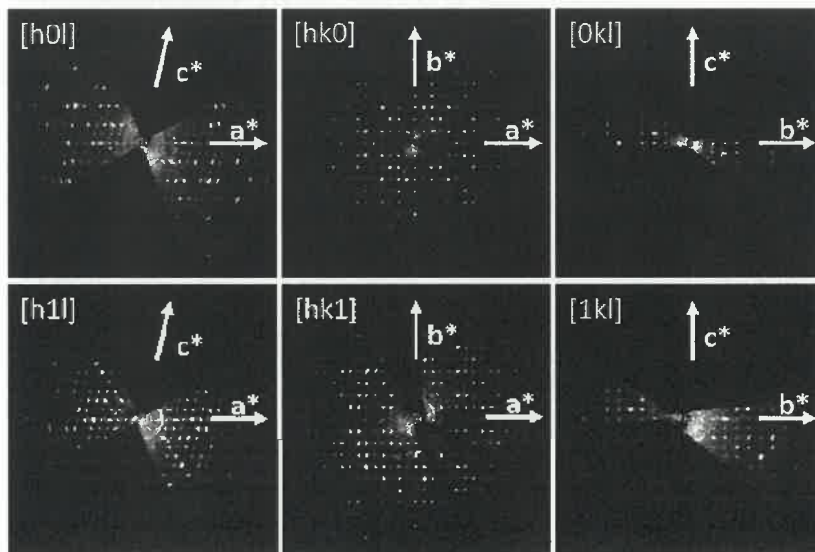
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# 3D Electron Diffraction at Institut Néel

## THE LOW-DOSE ELECTRON DIFFRACTION TOMOGRAPHY METHOD (LD-EDT)

### 3D reconstruction of the reciprocal lattice



Unit cell  
Symmetry  
Atomic positions



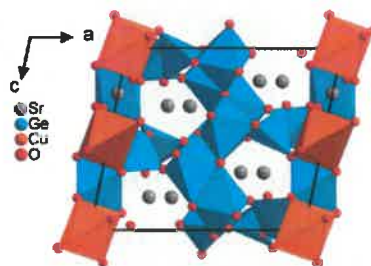
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# 3D Electron Diffraction at Institut Néel

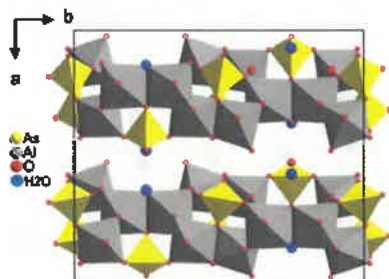
## THE LOW-DOSE ELECTRON DIFFRACTION TOMOGRAPHY METHOD (LD-EDT)



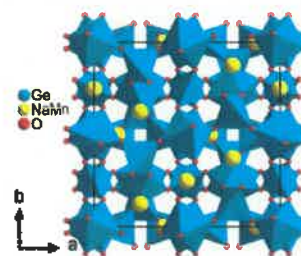
Some structures  
we solved



H. Klein, S. Kodjikian, R. Philippe, L. Ding, C.V. Colin, C. Darie & P. Bordet, 2020, *Acta Cryst.* B76, 727-732, <https://doi.org/10.1107/S2052520620008914>



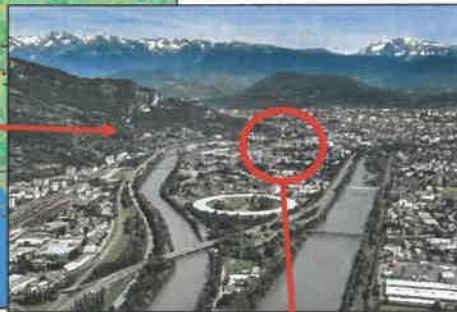
I.E. Grey, E. Yoruk, S. Kodjikian, H. Klein, C. Bougerol, H.E.A. Brand, P. Bordet, W.G. Mumme, G. Favreau and S.J. Mills, 2020, *Mineralogical Magazine*, 84, 608-615, doi:10.1180/mgm.2020.52



H. Klein, S. Kodjikian, Emre Yörük, Pierre Bordet  
To be published

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# Thanks for your attention



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**Institut Néel**

