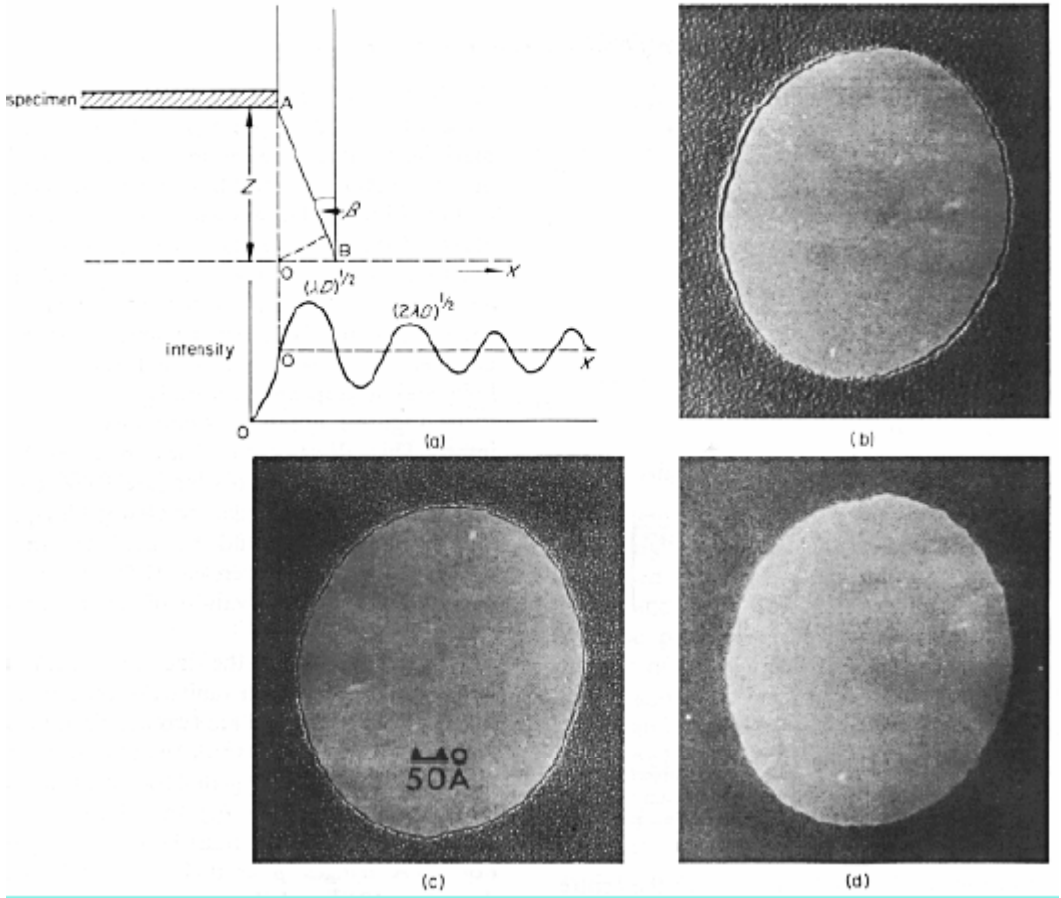
Phase microscopy: Fresnel fringes in the TEM

Interferences between waves not being in phase

overfocussed



underfocussed in focus

2 waves propagating through different media (sample, vacuum)

=> difference of mean inner potentials

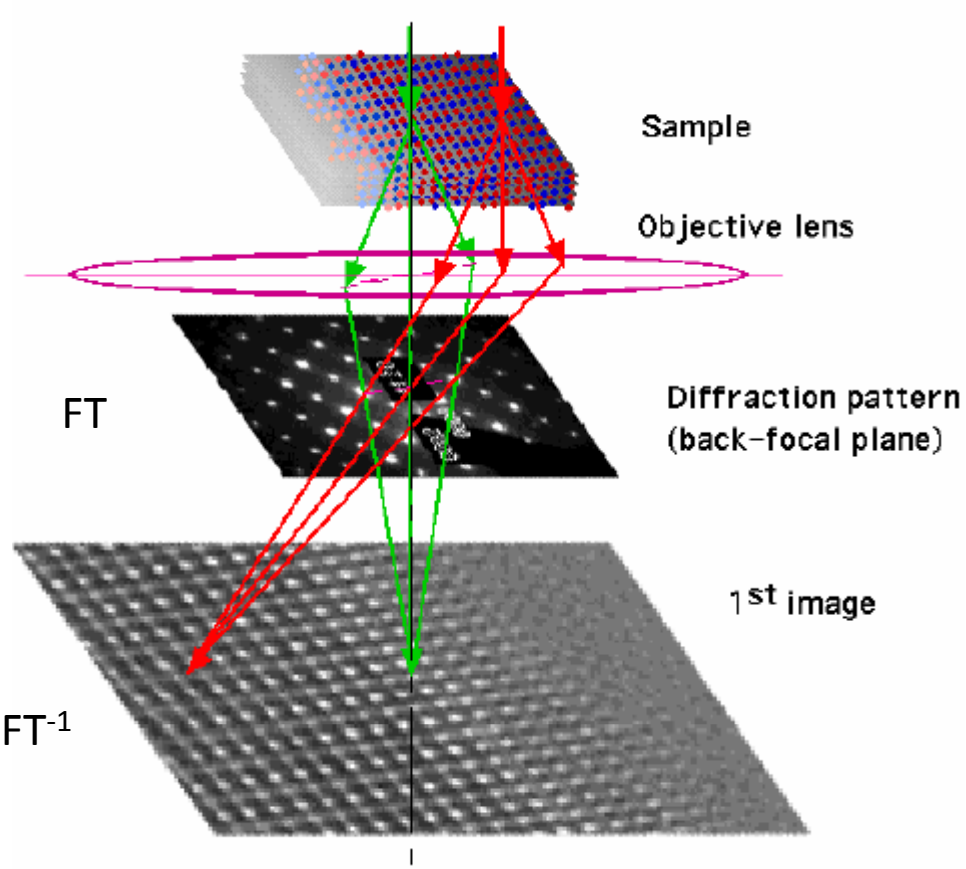
=> difference of optical paths

=> phase differences

Transformed into intensity contrast by the objective lens

Phase TEM relies on coherency of incident beam (energy spread, point source) => FEG

Phase microscopy: High Resolution Electron Microscopy



Multi-beam mode image: transmitted + several diffracted beams interfere
=> contrast = f (phase relationships of these beams)

- image "represents" the crystalline structure of the specimen => structural characterization at the atomic scale

- phase differences result from:
(i) interactions between the e-beam and the electrostatic potential in the object (periodical for a crystal)
(ii) phase shift induced by the objective lens (transfer function)

specimen thin enough => beam amplitude variations can be neglected **("phase object")**
=> phase shift proportional to the electrostatic potential at every point of the exit surface

High Resolution Electron Microscopy: WPO approx

- Transmission function of the sample:

if small phase shift, Weak Phase Object (WPO) approximation

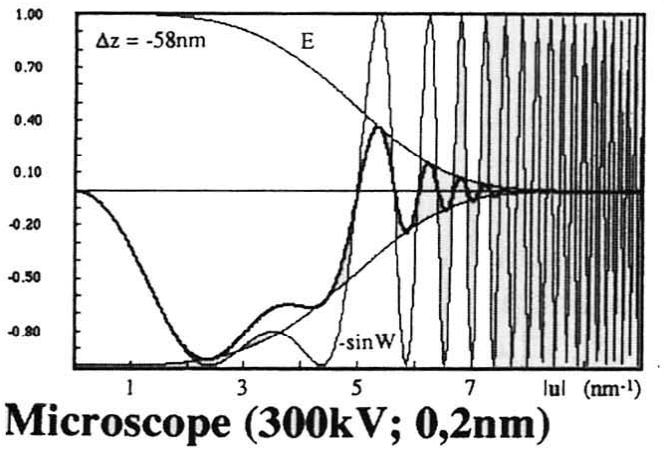
$q(x,y)= 1-i\sigma\Phi(x,y)$ ← Projected potential

- Image directly related to the projected potential of the object (atoms) after modification by the objective lens (filter)

$$I(x,y)= |\psi_i(x,y)|^2 = |q(x,y)*t(x,y)|^2$$
$$= 1+2\sigma\Phi(x,y) *s(x,y)$$

Projected potential (object) — Im. of t(x,y) (objective lens)

$s(x,y)=TF\{O(u,v)\sin(\chi(u,v)\}$ is the imaginary part of $t(x,y)$



Microscope (300kV; 0,2nm)

- best "reading" of the projected potential when

-largest possible frequency bandwith

-objective lens is underfocussed by $\Delta f = -1.2(Cs\lambda)^{1/2}$ (Scherzer condition)

High Resolution Electron Microscopy

• In all practical cases, WPO approximation is not valid
=> comparison HREM images/numerical simulation of the images for ≠ Df is necessary to access to the real structure of the sample (ex: where are the atomic columns?)

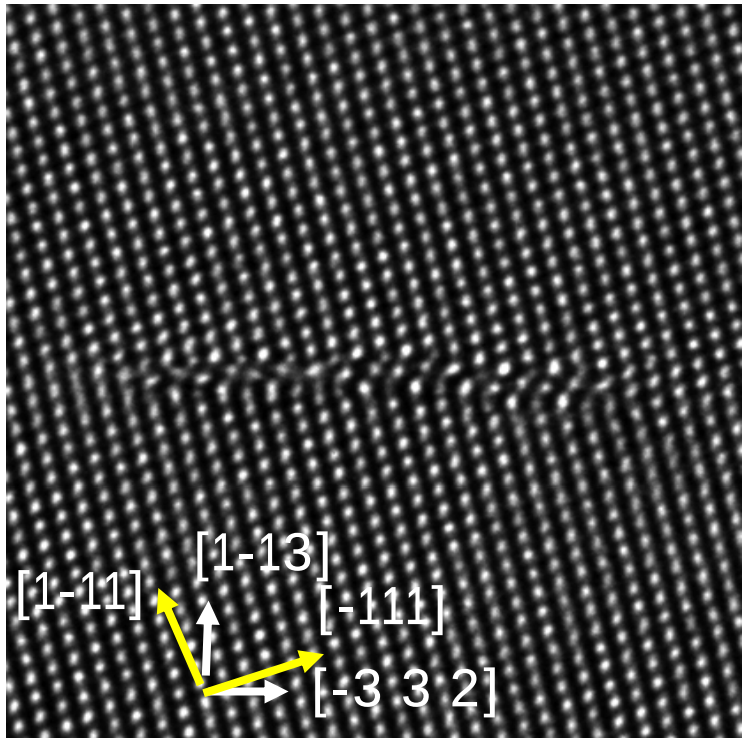
• However, although non linear, information regarding the atomic structure can be directly obtained in some cases: stacking sequences, extra plane, orientation relationship, presence of dislocations…

• Good experimental conditions:
-thin specimen (10 nm range)
- incident beam // to a simple (low index) direction of the crystal => diffraction= known plane of the RS
- column perfectly aligned
- appropriate defocus of the objective lens (Schertzer condition)
- microscope resolution smaller than the distance between the atomic columns that you want to image!

Never trust ONE image !

High Resolution Electron Microscopy

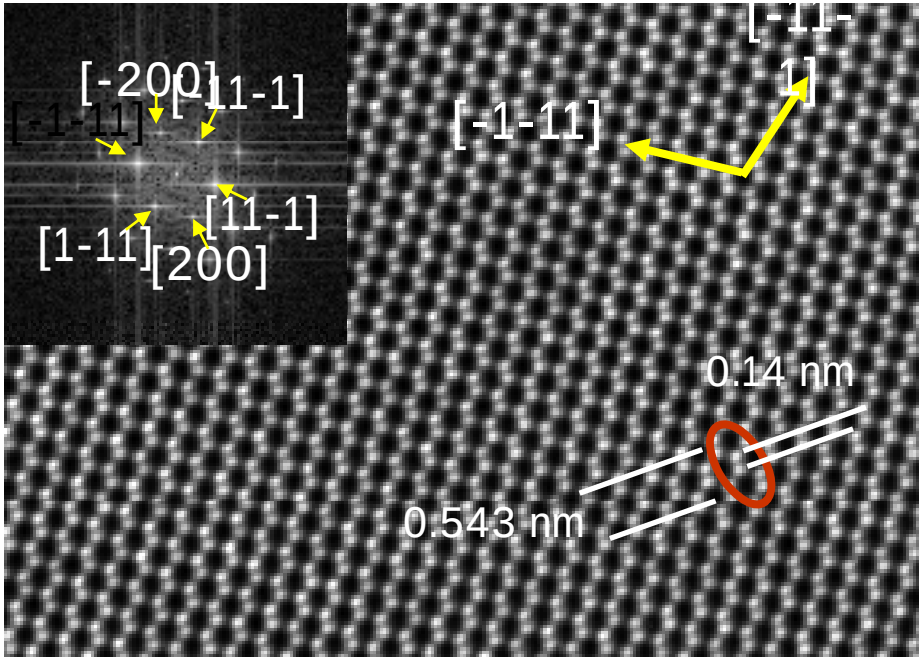
Rod-like defect in silicon



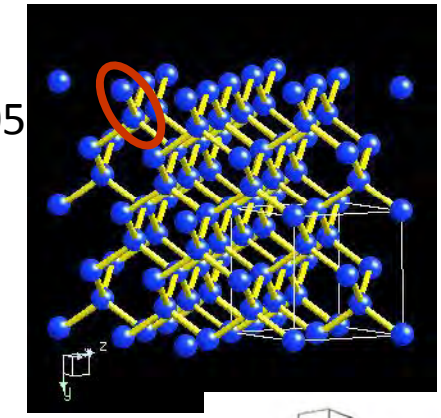
Cherkashin 2005

B=[011]

Si « dumbells »



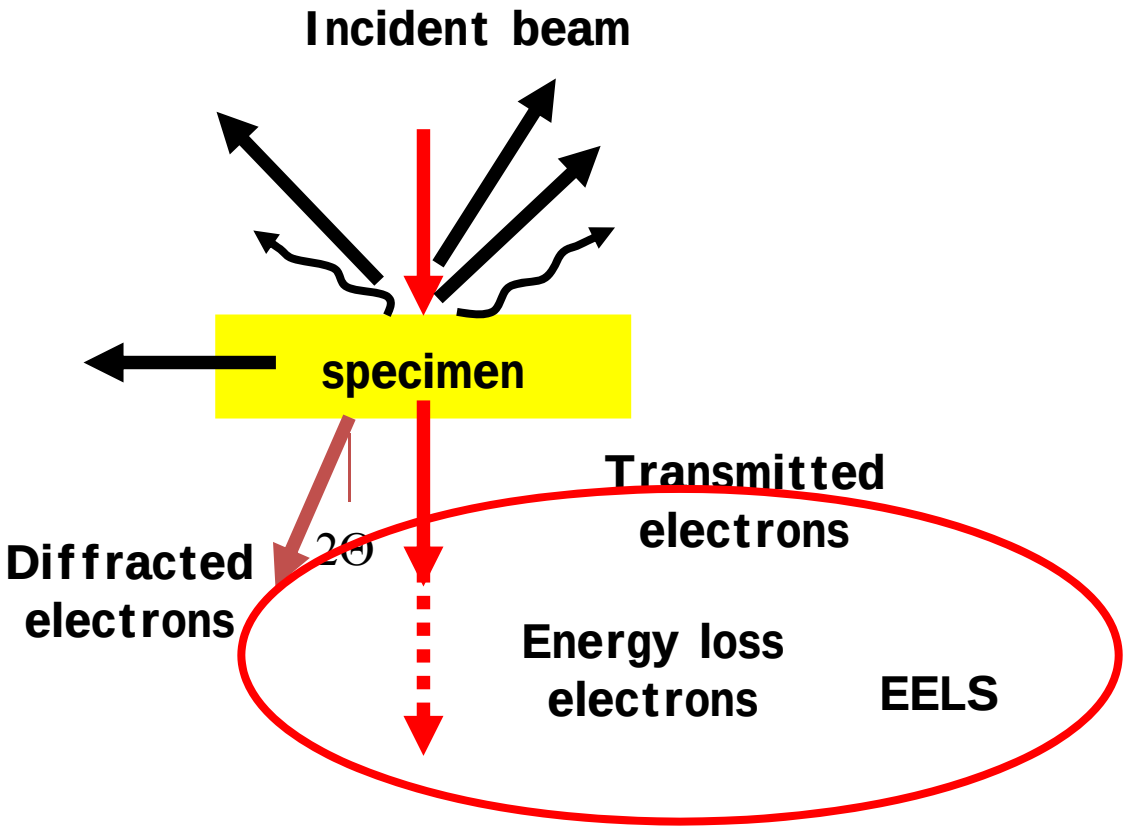
with Cs corrector



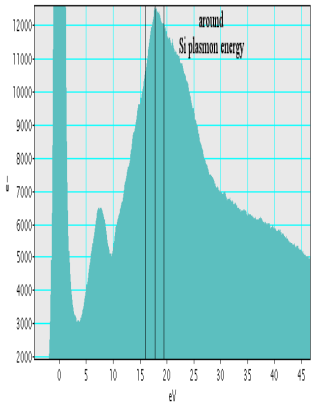
Houdellier 2005

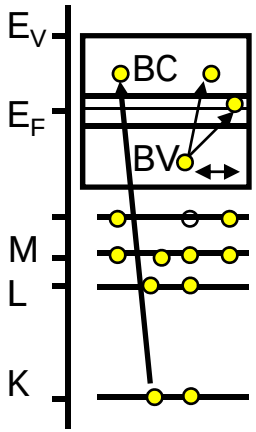
Interfaces, precipitates, defects, phase identification, defects, strain, etc…

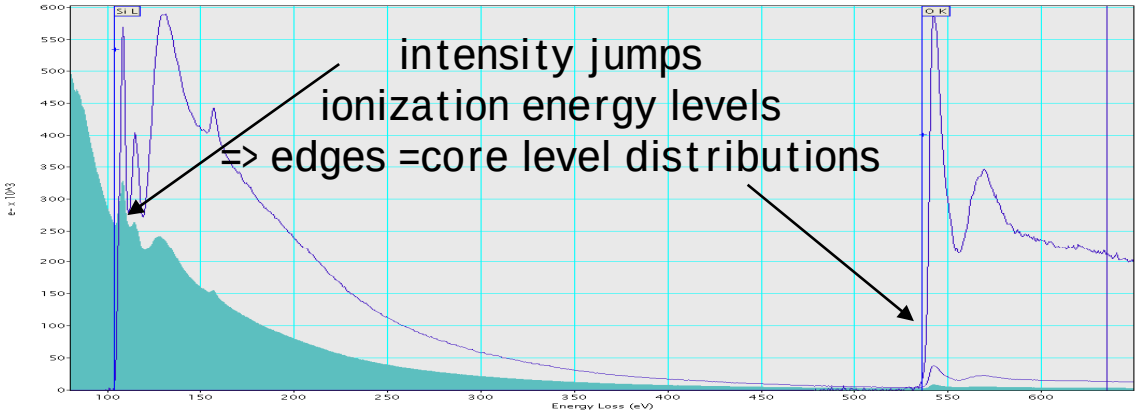
What else ?


Incident beam
specimen
Transmitted electrons
Diffracted electrons
2Θ
Energy loss electrons
EELS

Electron Energy Loss Spectroscopy







$$\mathrm{Im}(-1/\varepsilon) = \varepsilon_2 / (\varepsilon_1^{\,2} + \varepsilon_2^{\,2})$$

Low-losses (the first few ten eV)

Valence electrons (VEELS)

Collective excitation : **Plasmon**
Individual excitation : **Interband Transition**

Thickness: $I_t / I_{zl} = t / \lambda_p$

Dielectric function

$\varepsilon = \varepsilon_1 + i\,\varepsilon_2$

Optical parameters

$n, k, \mu, R = f(\varepsilon_1, \varepsilon_2)$

Electronic structure

$\varepsilon_2 \propto$ JDOS

Core-losses (up to a few thousand eV)

Inelastic interactions with inner or core level electrons

Elemental quantitative analysis

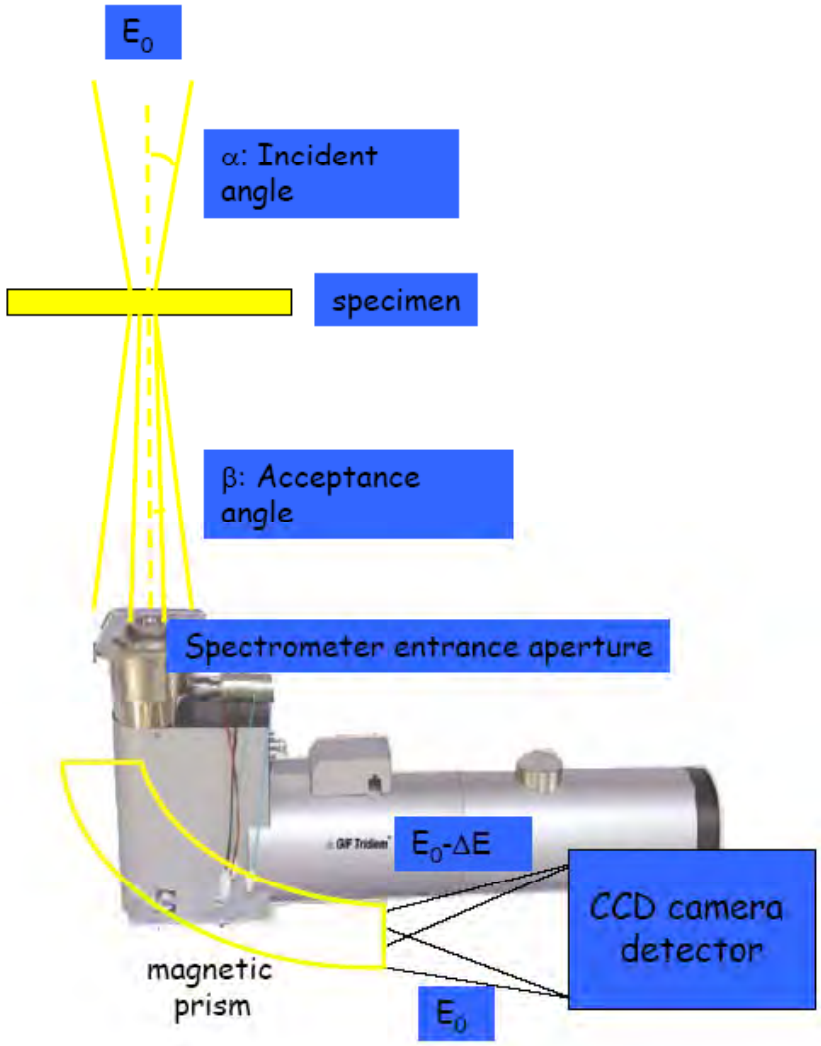
Electronic structure

$I \sim \mathrm{Im}(-1/\varepsilon) = \varepsilon_2 \propto$ unoccupied DOS

Energy **L**oss **N**ear **E**dge fine **S**tructure

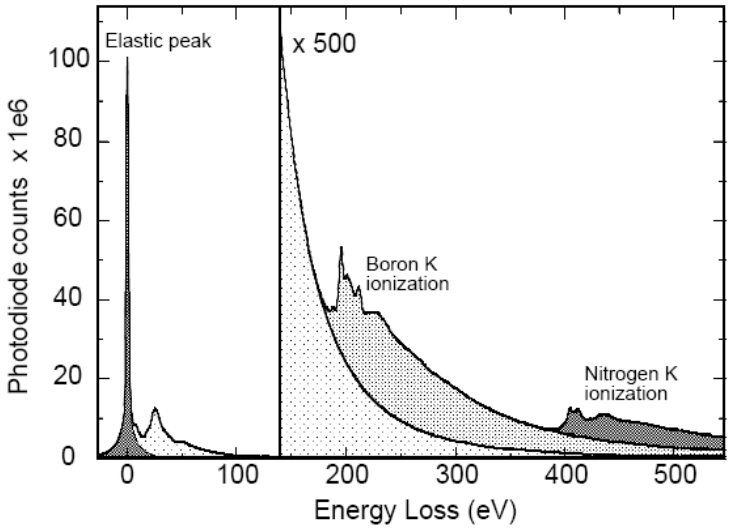
ELNES

Electron Energy Loss Spectroscopy

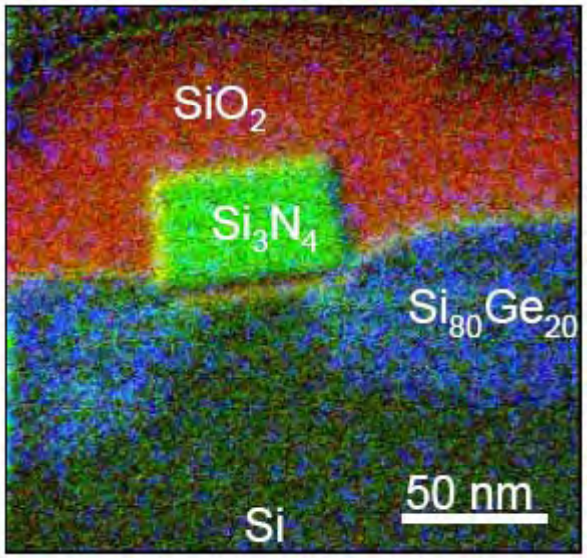


Electron Energy Loss Spectroscopy

. Electron Energy Loss Spectroscopy



Energy filtered TEM



TEM/EELS

EELS adds one dimension to TEM imaging

By probing the excitations of electrons bounded to the solid with the electrons from the incident beam

Gives information on:

-specimen thickness
-elemental chemical composition and spatial distribution of elements
-spatial distribution of first neighbors relative to an atomic site
-chemical bonding
-electronic band structure
-dielectric function

with an energy resolution ranging between 1 eV and 0.3 eV

with a spatial resolution ranging between 10 nm and 1 nm

Concept of holography

Complex wave : amplitude and phase

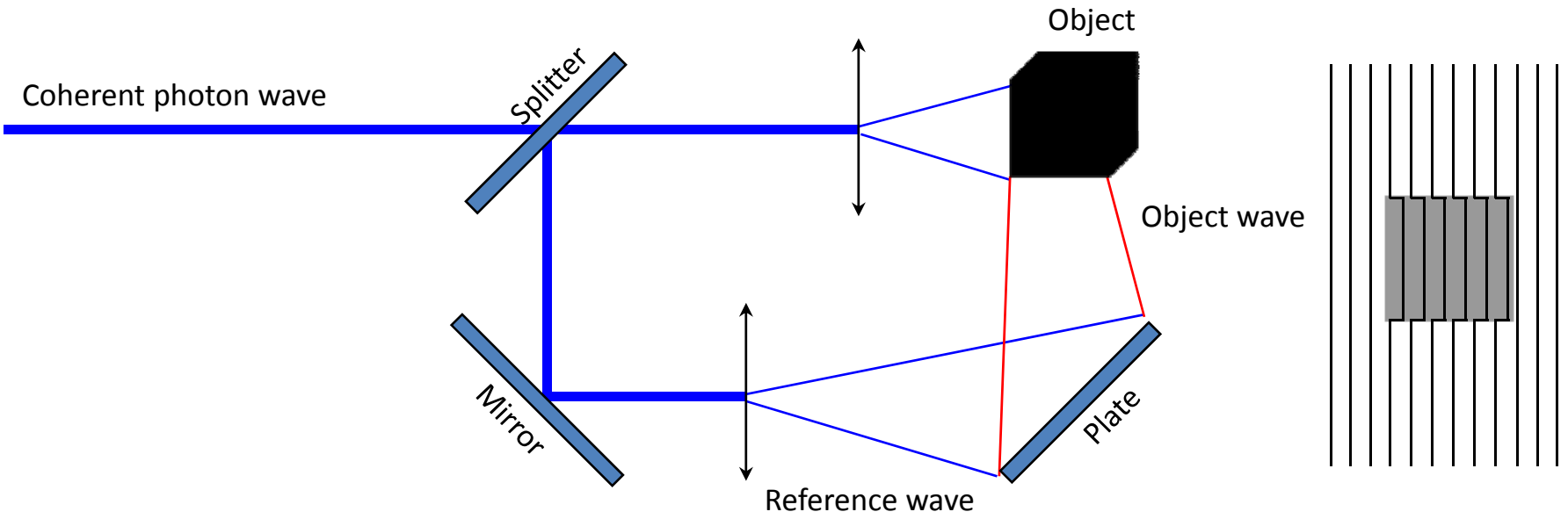
$$\Psi = Ae^{i\varphi}$$

Take a picture : 

$$I = |\Psi|^2 = A^2$$

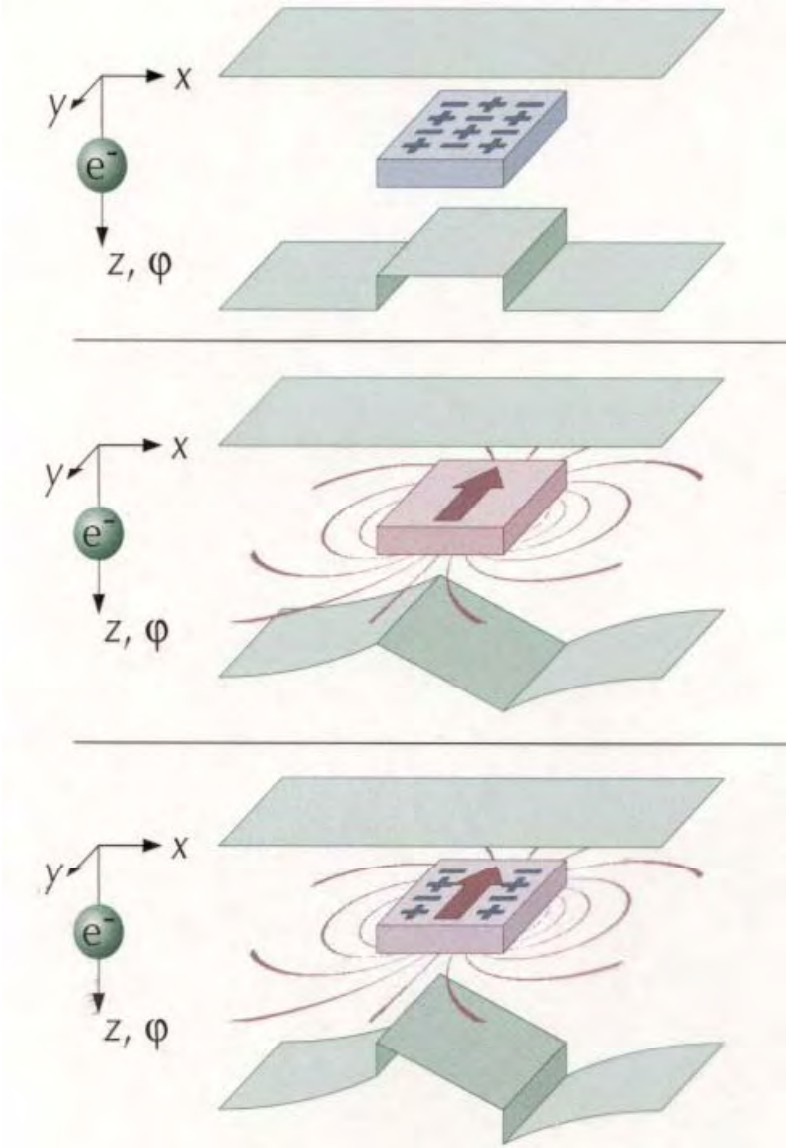
You loose the phase φ !!!

We want to retrieve the phase !



$$I = |\Psi_{ref} + \Psi_{object}|^2 = A_{ref}{}^2 + A_{object}{}^2 + 2A_{ref}A_{object}\cos(2\pi R_0 x + \varphi_{object} - \varphi_{ref})$$

Information in the phase

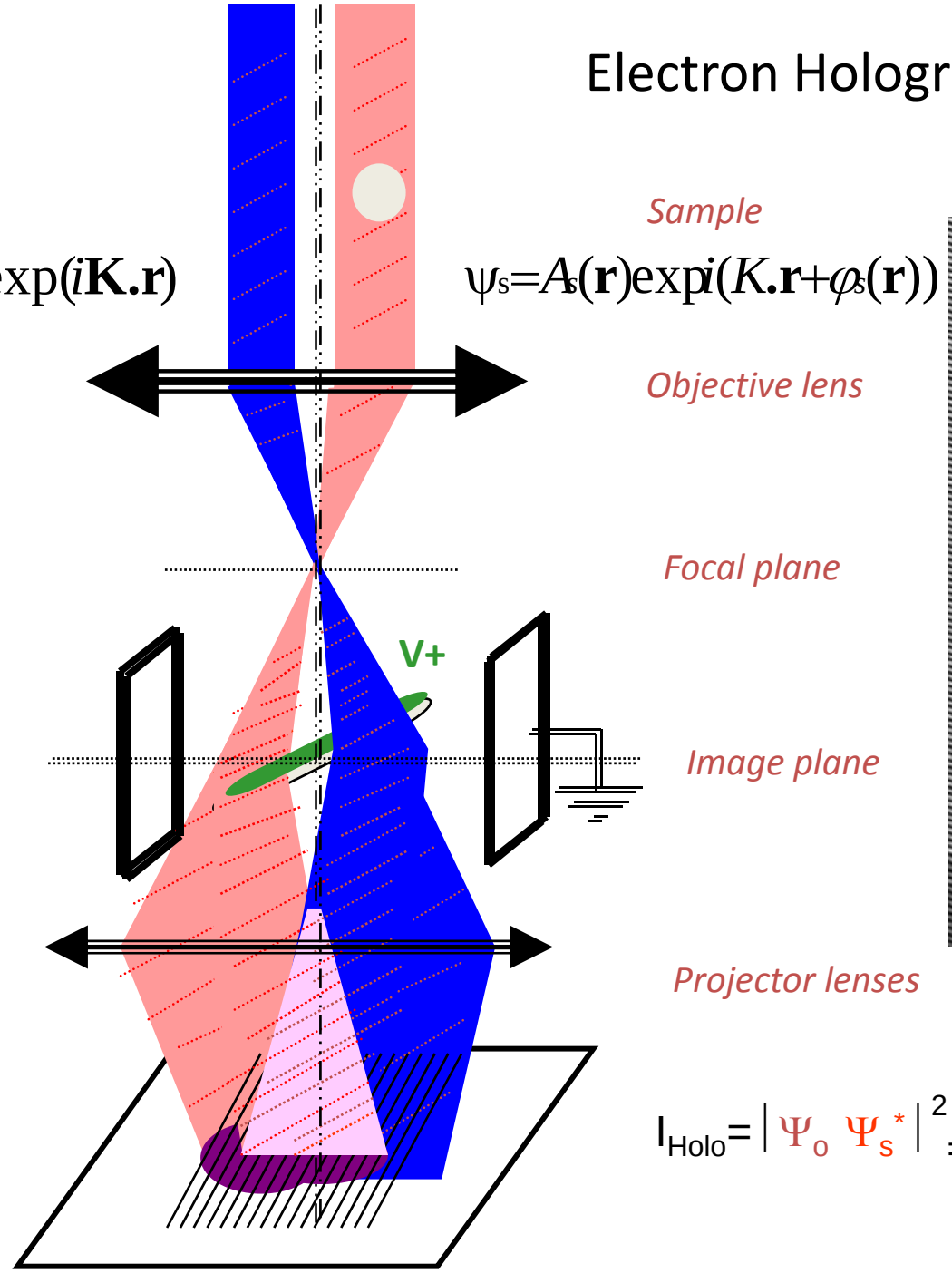


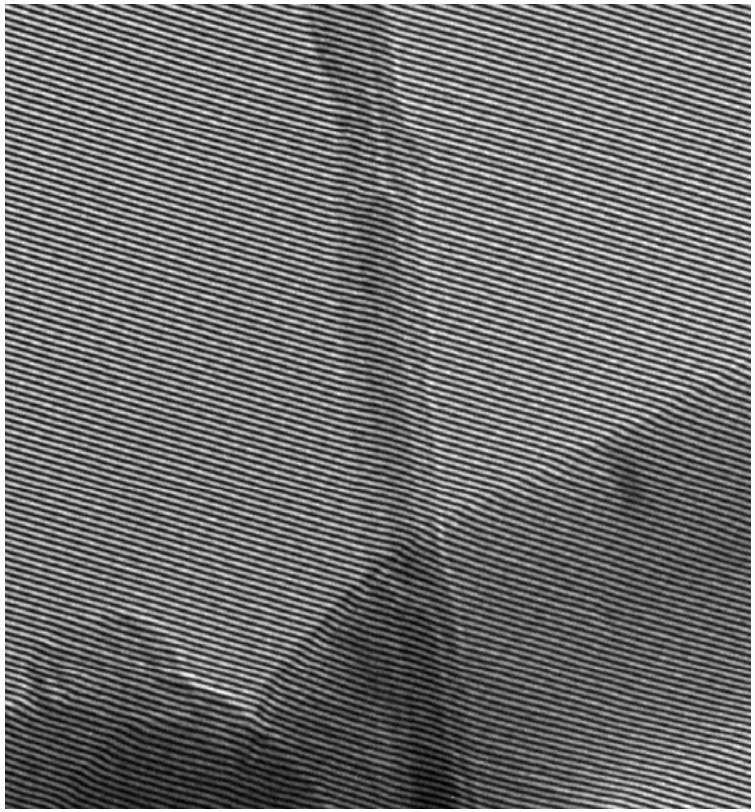
$$\phi^{E}(\mathrm{x}) = \mathrm{C_E} \int V(x,z)dz$$

$$\phi^{M}(\mathrm{x}) = -\frac{e}{\hbar} \iint B_{\mathrm{n}}(\mathrm{x},\mathrm{z})\mathrm{dxdz}$$

$$\phi^{T} = \phi^{M} + \phi^{E}$$

Electron Holography in a TEM

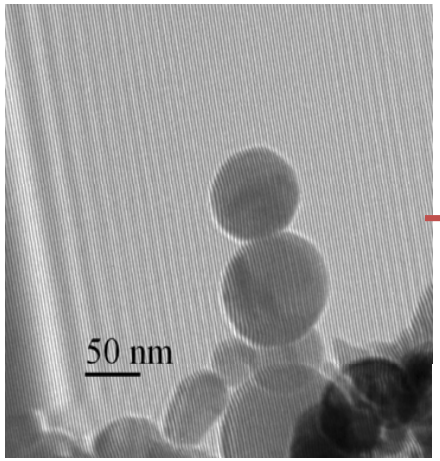




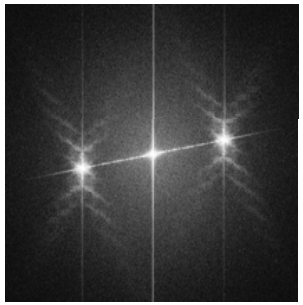
$$I_{Holo} = |\Psi_o \Psi_s^*|^2 = 1 + A_S^2(x,y)$$

$$+2A_S(x,y)\cos[2\pi R_0.x+\varphi_S(x,y)]$$

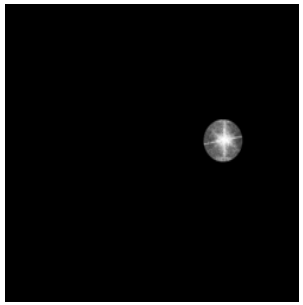
GPA analysis of the hologram



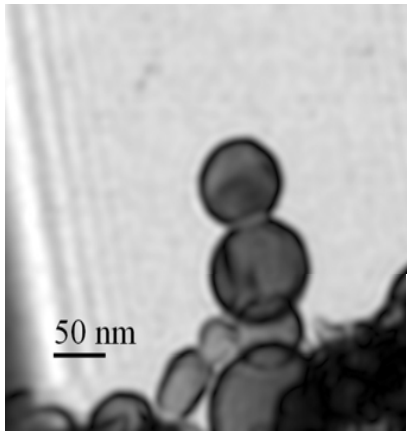
FT

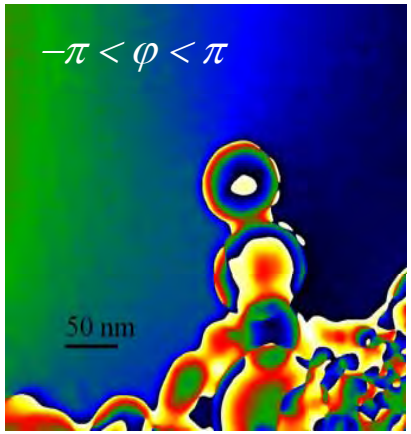


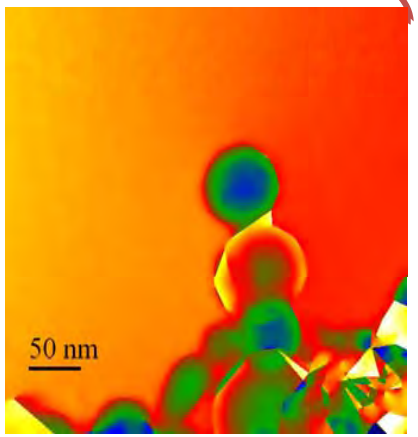
Mask



FT^{-1}







2π jumps

unwrapped

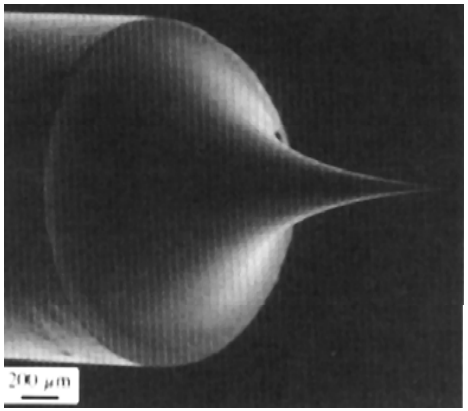
Image d'amplitude
$A_S(x,y)$

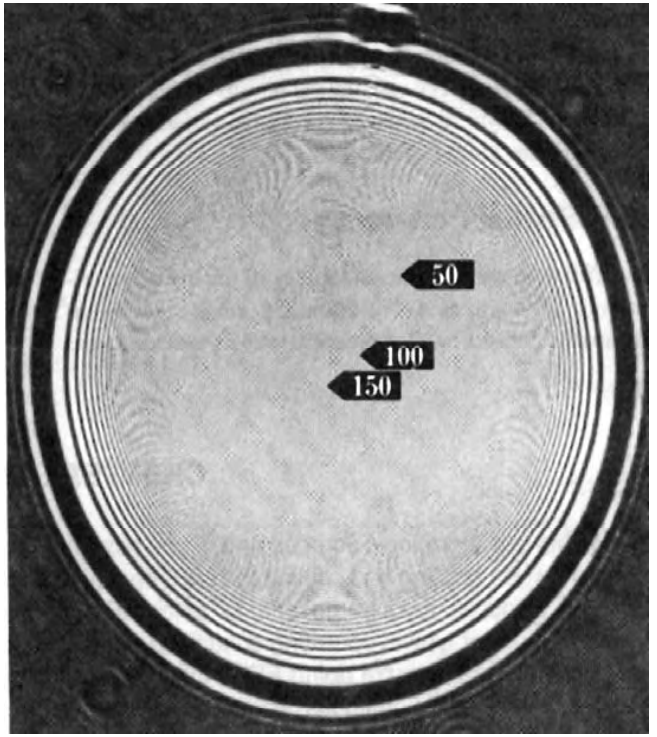
Image de phase
$\varphi_S(x,y)$

Experimental aspects

- Coherent Electron Beam

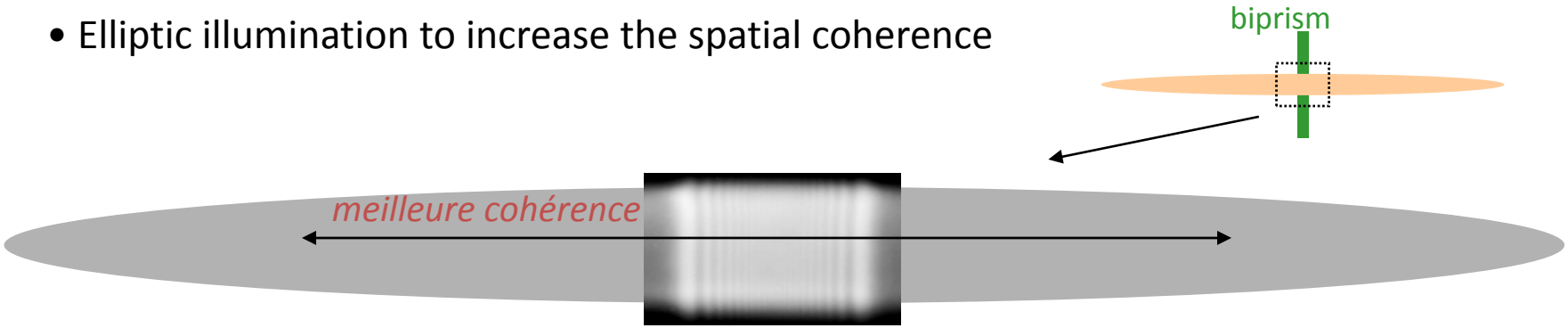
Field Emission Gun

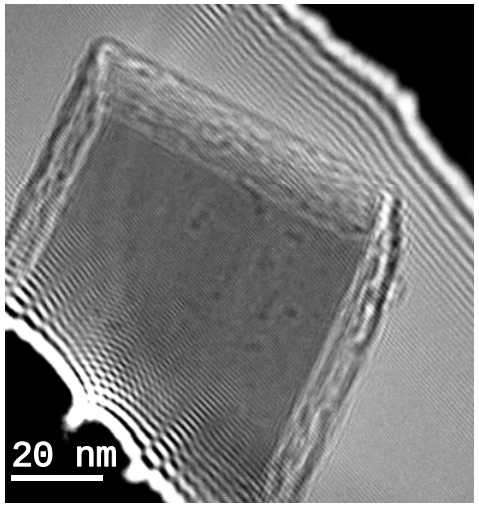




Fresnel fringes due to the high coherence of the beam

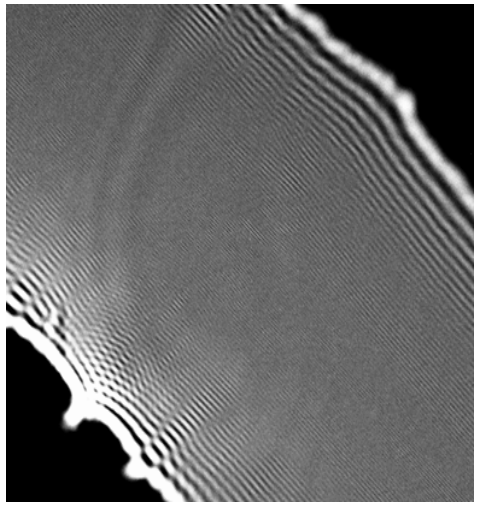
- Elliptic illumination to increase the spatial coherence





Hologram with the object

Reference hologram (without object)



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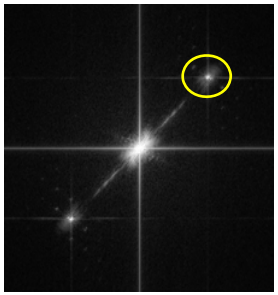

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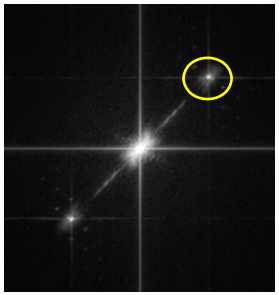

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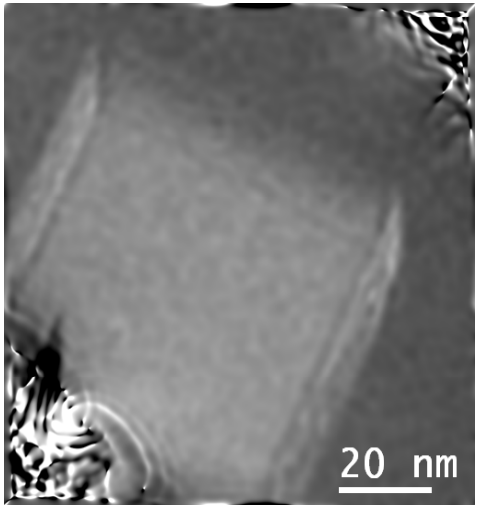
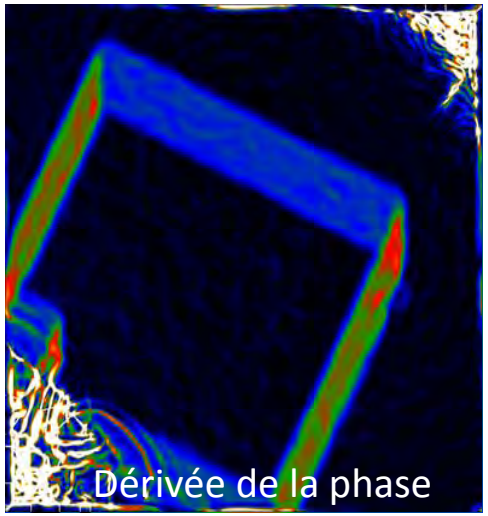


$I_S = A_S e^{i\varphi_S}$

$I_R = A_R e^{i\varphi_R}$

$$\frac{I_S}{I_R} = \frac{A_S}{A_R} e^{i(\varphi_S - \varphi_R)}$$



Normalised Amplitude $\frac{A_S}{A_R} = A_N$

Phase shift due to the object only $\varphi_S - \varphi_R = \Delta\varphi$

Non magnetic sample (B = 0)

$$\varphi(x,y) = C_E \int V(x,y,z)dz - \frac{e}{\hbar}\iint B_n(x,y,z)dxdz$$

$$\varphi_{elect}(x,y) = C_E \int V(x,y,z)dz$$

with : $C_E = \left(\frac{2\pi}{\lambda}\right)\left(\frac{E + E_0}{E(E + 2E_0)}\right)$

$V(x,y,z) = V_i(x,y,z) + V(x,y,z)$

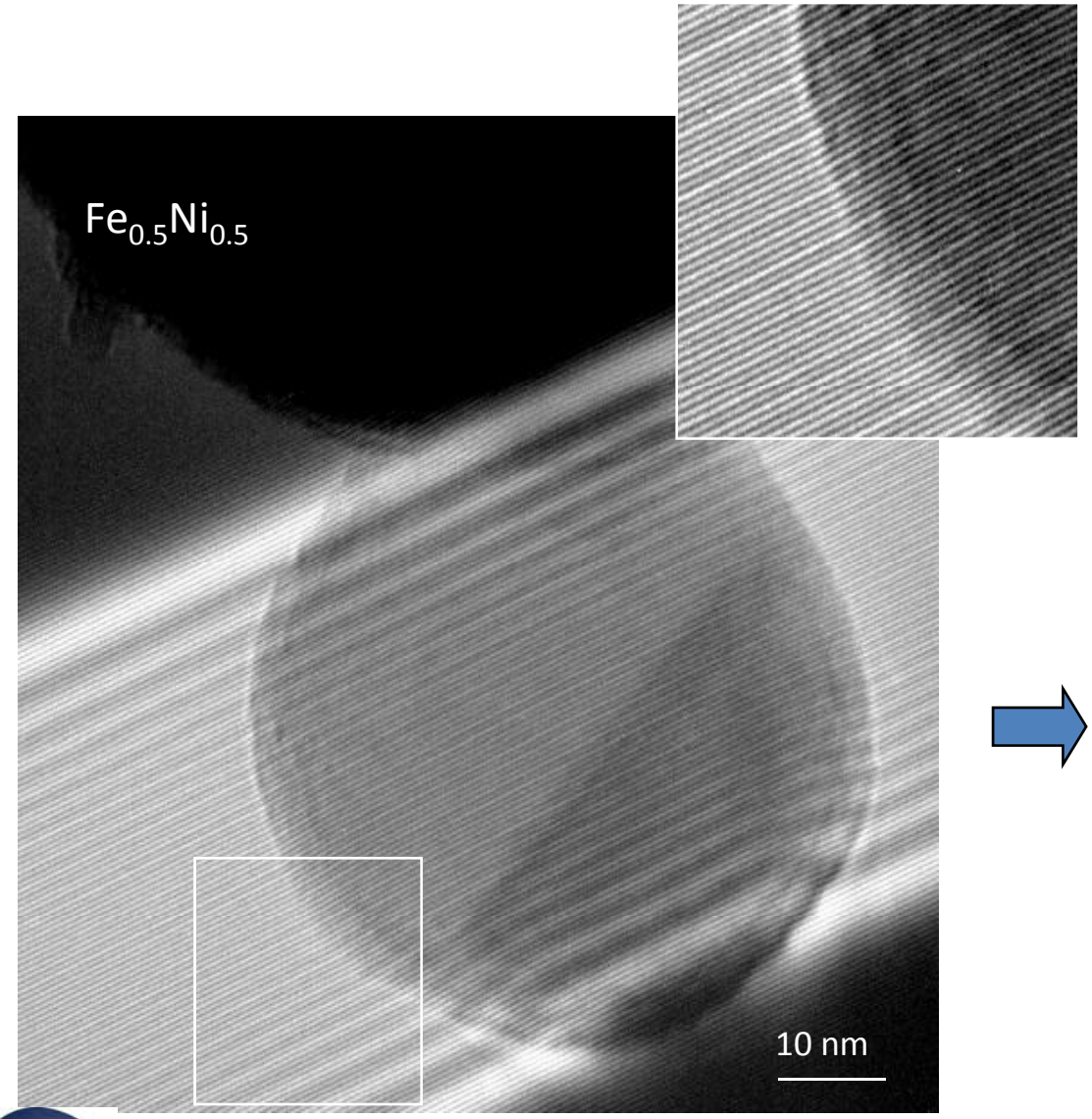
- $V_i(x,y,z)$ = Mean Inner Potential (MIP)
- $V(x,y,z)$ = Local electric potential (junctions, CMOS, diode ..)
- if V_i et V are constants along the e-beam direction « z » :

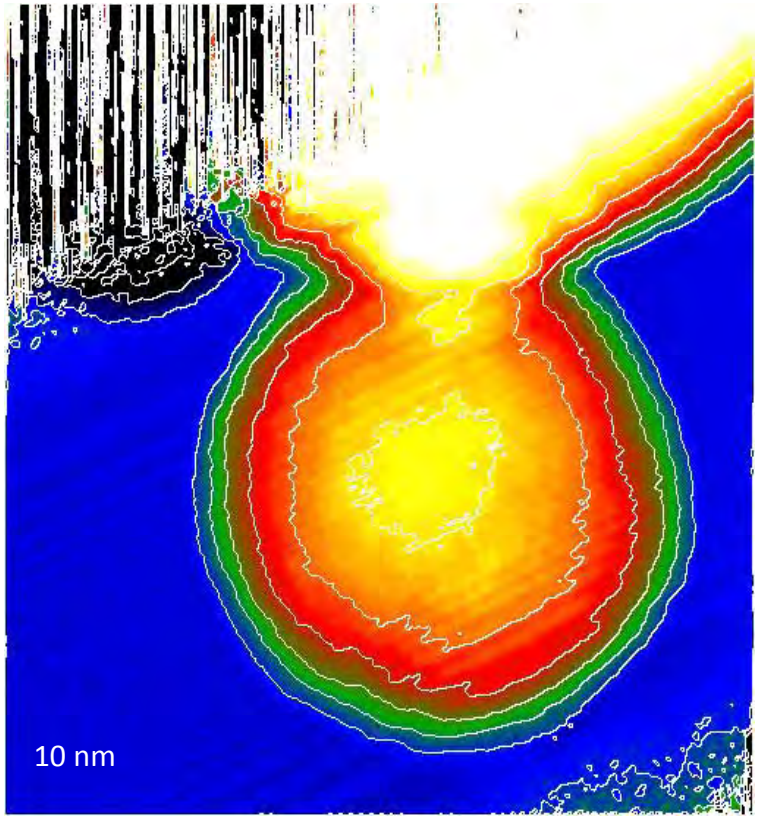
$$\varphi_{elect} = C_E (V_i + V)\, t \qquad t = \text{sample thickness}$$

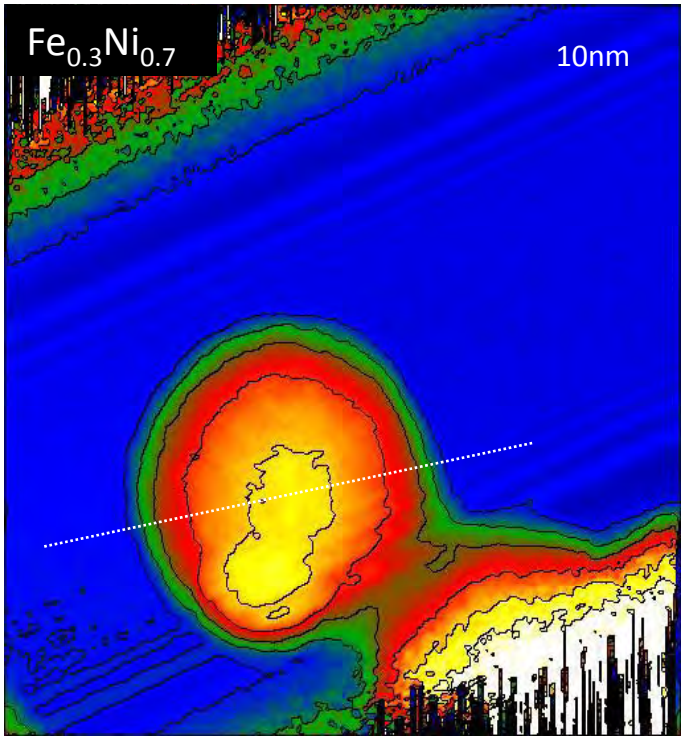
➔ Need to perfectly know the sample thickness (sphere, cleaved sample, CBED thickness measurement...) to quantify V_i and V

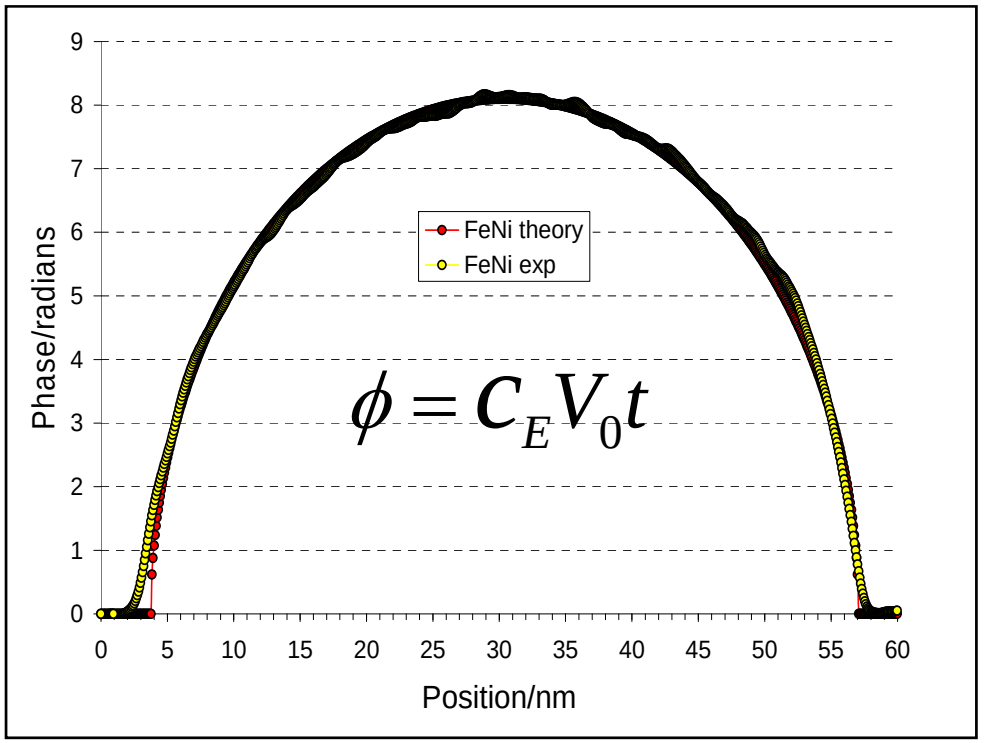
Mean Inner Potential (MIP) measurement

$$\varphi_{elect} = C_E V_i t$$









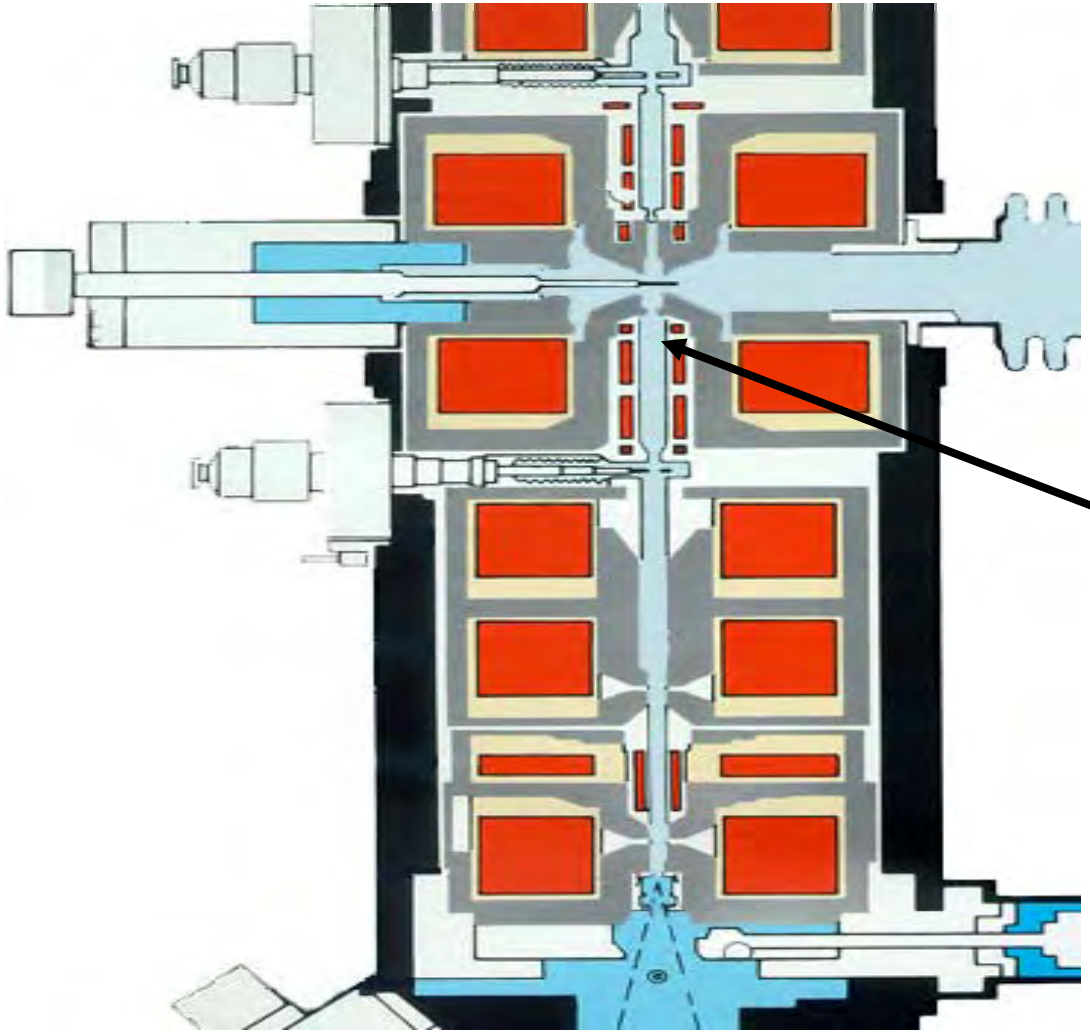
- MIP measurement
- Composition analysis
- Morphological studies

CEMES TOULOUSE

Magnetic samples

$$\varphi(x) = C_E \int V(x,z)dz - \frac{e}{\hbar}\iint B_n(x,z)dxdz$$

Pb. 1: The objective must be witched off. => use of Lorentz lens to get sufficient resolution



- Object in a magnetic field free area
- Field of view up to 1µm

f = 30 mm

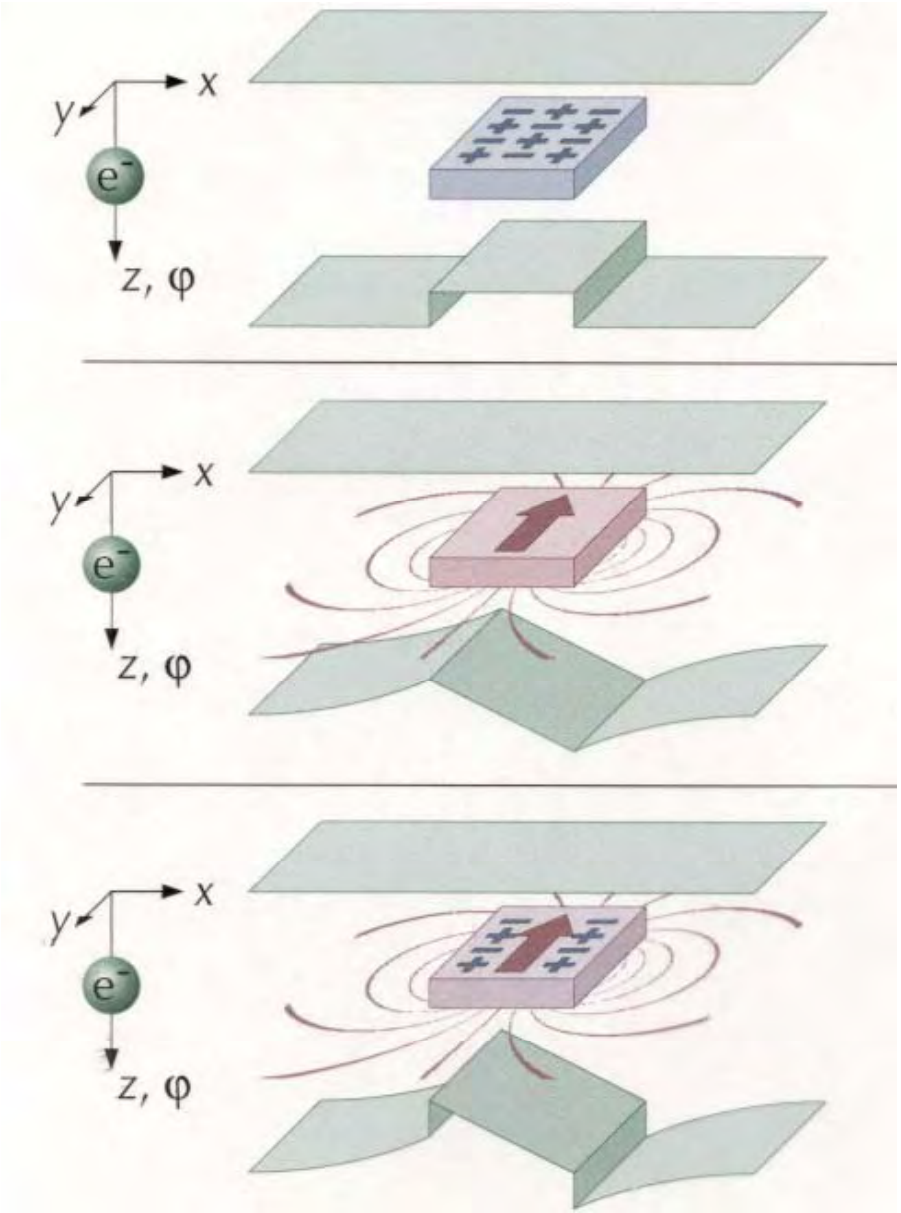
Cs = 7430 mm (!!)

Cc = 39 mm

Point resolution # 2 nm

Magnetic samples

Pb. 2: How to separate the magnetic and electrostatic contributions B and $C_E V_i t$?



Phase shift induced bt the local electrostatic potential:

$$\varphi_{elect}(\mathrm{x}) = \mathrm{C_E} \int V(x,z)dz$$

Phase shift induced by the local magnetic field :

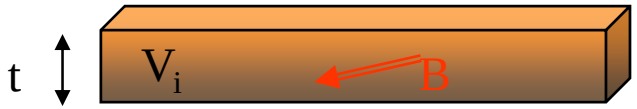
$$\varphi_{mag}(\mathrm{x}) = -\frac{e}{\hbar} \iint B_{\mathrm{n}}(\mathrm{x},\mathrm{z})\mathrm{dxdz}$$

Total phase shift

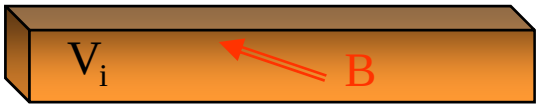
$$\varphi_{Tot}(\mathrm{x}) = \varphi_{elect}(\mathrm{x}) + \varphi_{mag}(\mathrm{x})$$

Pb. 2: How to separate the magnetic and electrostatic contributions B and $C_E V_i t$?

1/ Taking two holograms (4 with the reference holograms) switching upside down the sample
=> The sign of the magnetic contribution is reversed and the electrostatic one remains.



$$\varphi_1 = C_E V_i . t - \frac{e}{\hbar}\iint B_n ds$$



$$\varphi_2 = C_E V_i . t + \frac{e}{\hbar}\iint B_n ds$$

$$\varphi_2 - \varphi_1 = 2\frac{e}{\hbar}\iint B_n ds \qquad \varphi_2 + \varphi_1 = 2C_E V_i t$$

Problem : finding back the same area of interest

2/ Taking two holograms with two different high voltages

$$C_E = \left(\frac{2\pi}{\lambda}\right)\left(\frac{E + E_0}{E(E + 2E_0)}\right)$$

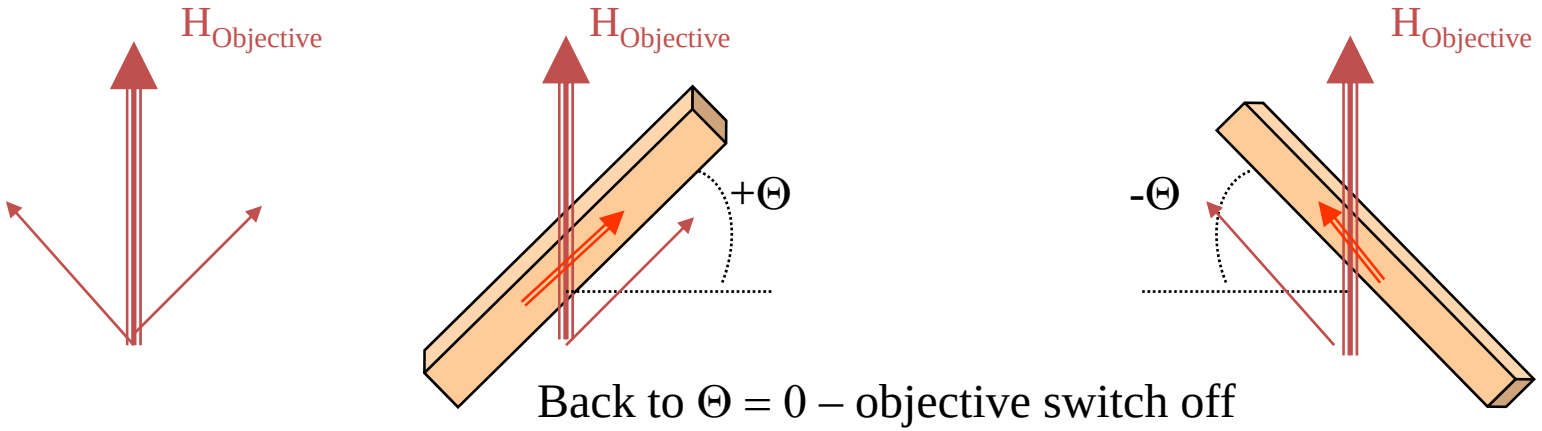
$$\varphi_1 = C_{E_1} V_i . t - \frac{e}{\hbar}\iint B_n ds$$

$$\varphi_2 = C_{E_2} V_i . t - \frac{e}{\hbar}\iint B_n ds$$

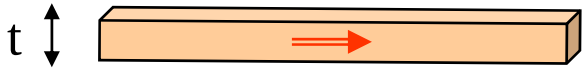
$$V_i t = \frac{\varphi_2 - \varphi_1}{(C_{E_2} - C_{E_1})}$$

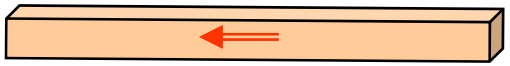
Problem : keeping the same acquisition conditions (magnification, alignment…) when changing the high voltage.

3/ Using the high magnetic field of the objective (~2 Tesla) to saturate the magnetic sample in two opposite directions



Back to Θ = 0 – objective switch off





$$\varphi_1 = C_E V_i.t - \frac{e}{\hbar}.t.\int B_n(x).dx$$

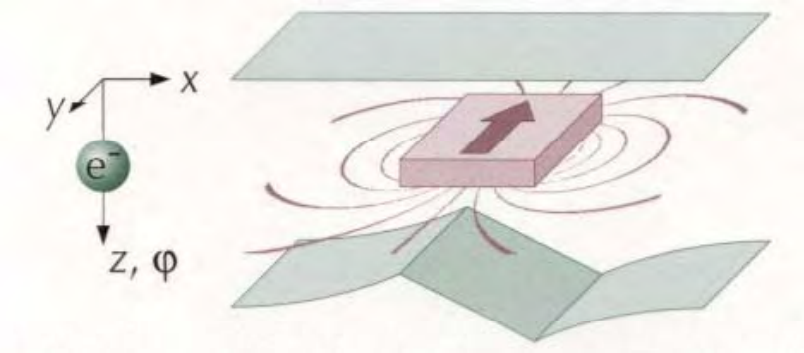
$$\varphi_2 = C_E V_i.t + \frac{e}{\hbar}.t.\int B_n(x).dx$$

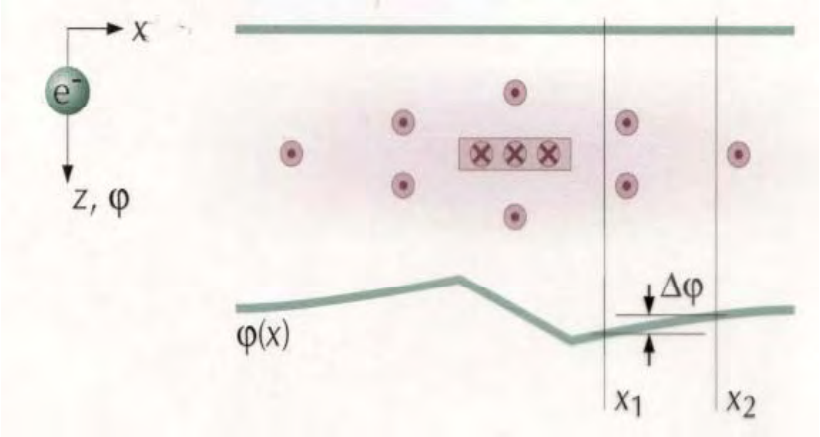
$$\frac{e}{\hbar}.t.\int B_n(x).dx = \frac{\varphi_2 - \varphi_1}{2}$$

Magnetic contribution to the phase shift

$$C_E V_i t = \frac{\varphi_2 + \varphi_1}{2}$$

Electrostatic contribution to the phase shift



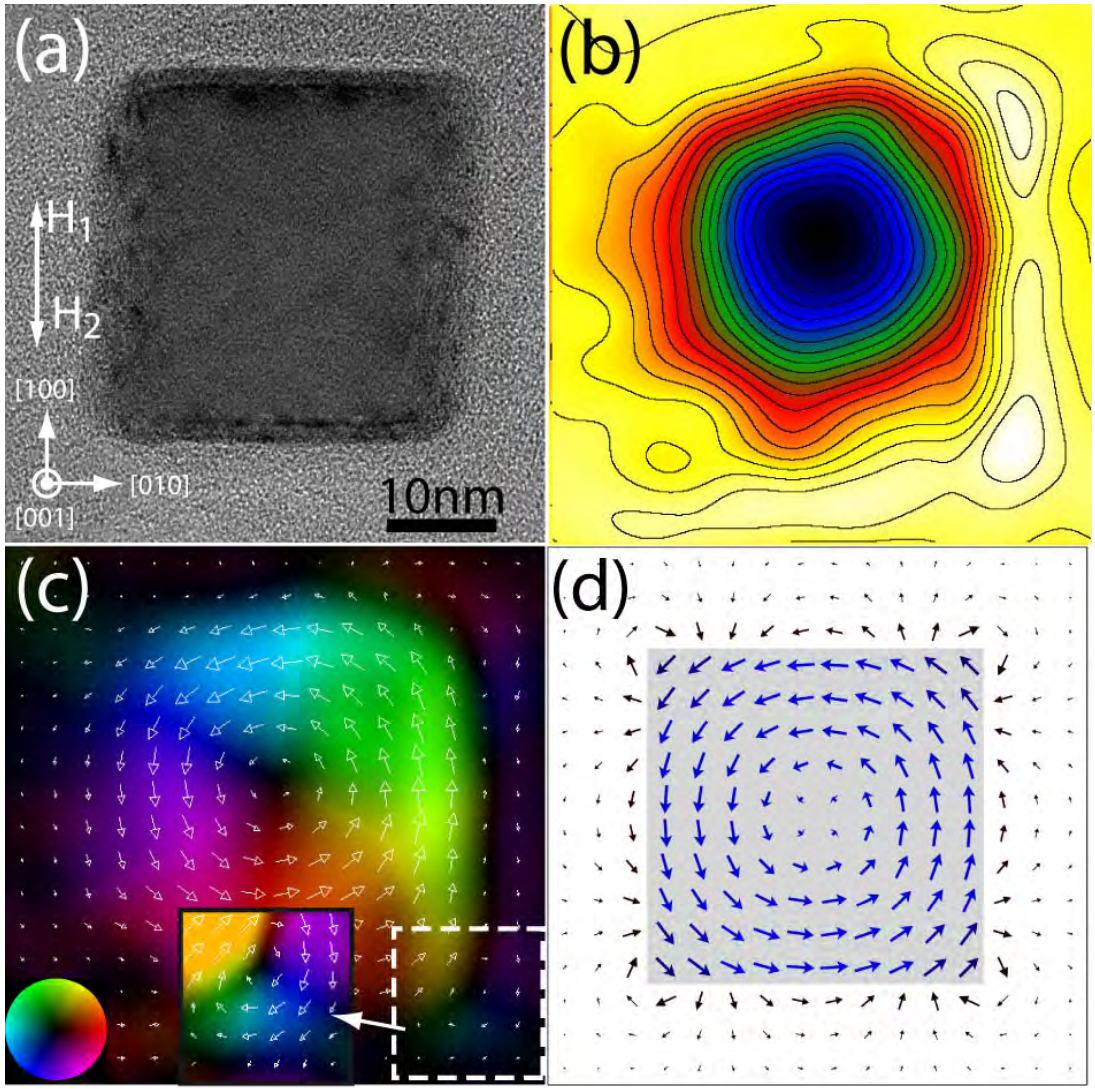


$$\Delta\varphi = \varphi(x_2, y) - \varphi(x_1, y) = -\frac{e}{\hbar} \int_{\xi=x_2}^{x_1} \int B_n(\xi, y, z) d\xi dz$$

$$\frac{\partial \varphi(x)}{\partial x} = \frac{e}{\hbar} B_n(x).t$$

- The gradient of the phase is proportional to the in-plane component of the magnetic induction B_n
- The equiphase contour gives the direction of the magnetic induction B

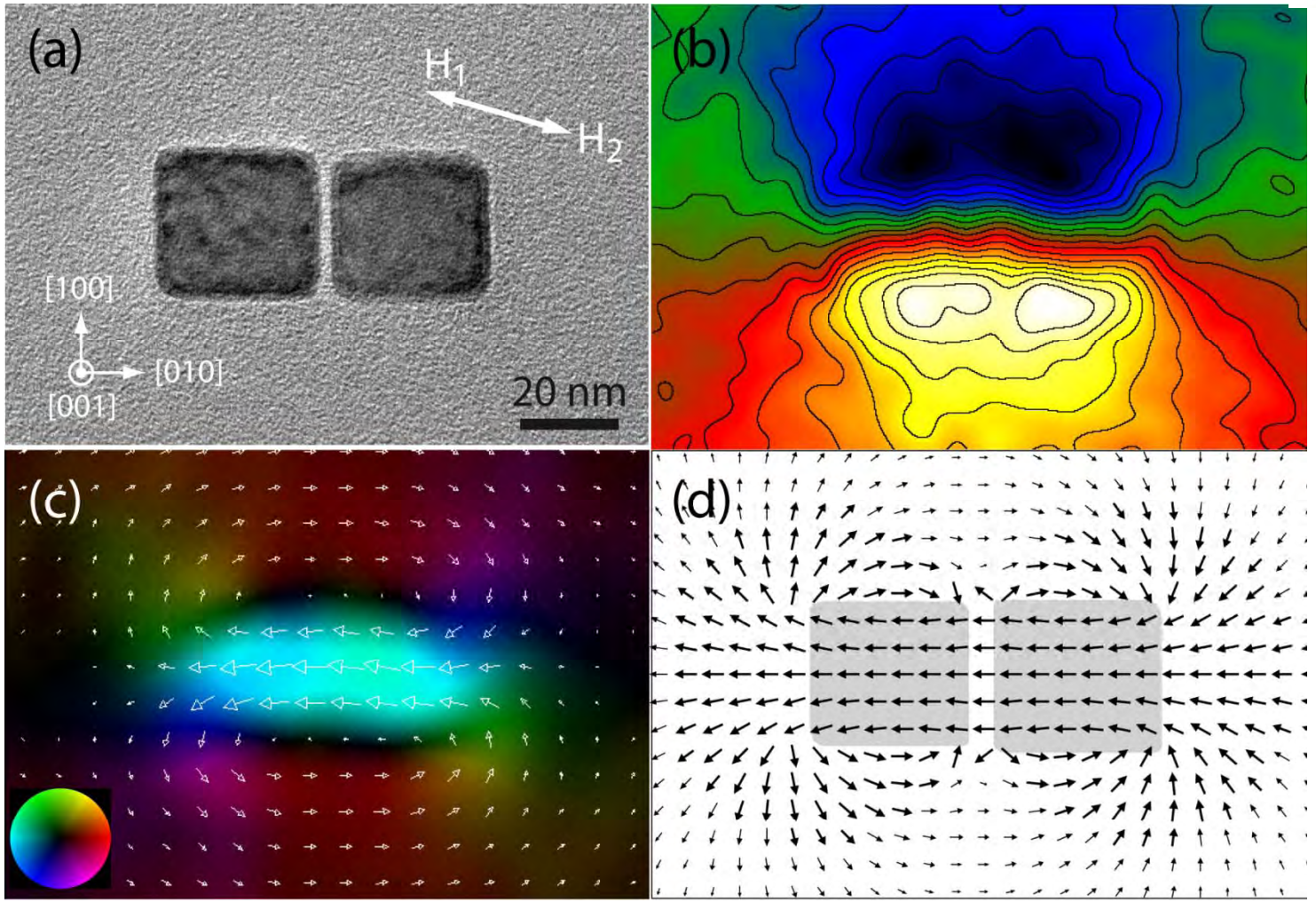
Magnetic Configuration of an isolated Fe Nanocube



(a) TEM image, (b) Magnetic contribution to the phase shift, (c) Magnetic induction mapping, (d) micromagnetic simulation (OOMMF)

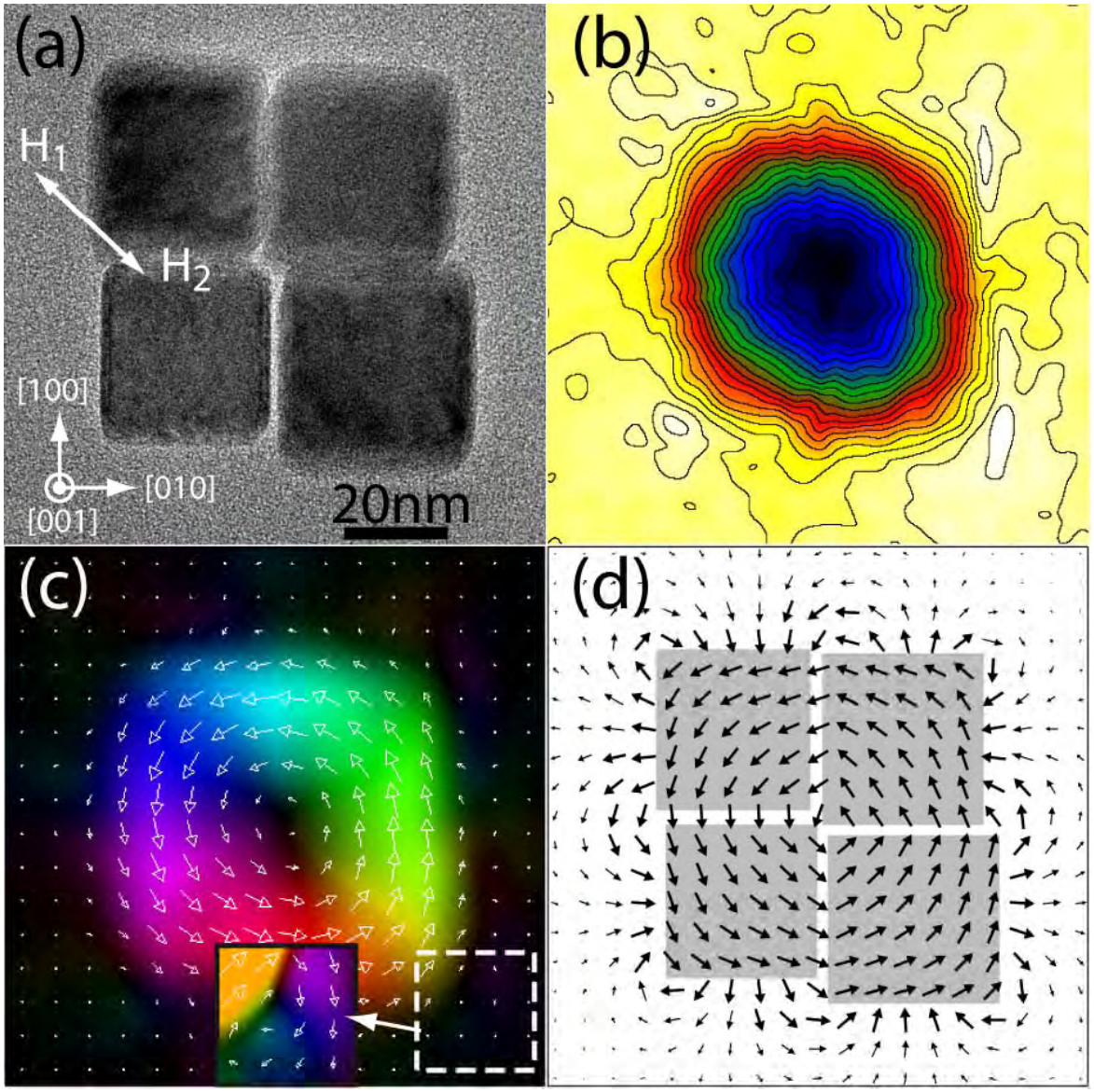
Snoeck et al., Nano Lett., **2008**, *8* (12), pp 4293–4298

Magnetic Configuration of two neighbouring Fe Nanocubes



(a) TEM image, (b) Magnetic contribution to the phase shift, (c) Magnetic induction mapping, (d) micromagnetic simulation (OOMMF)

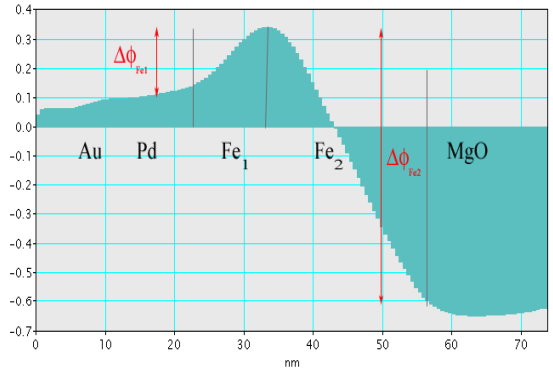
Magnetic Configuration of four neighbouring Fe Nanocubes



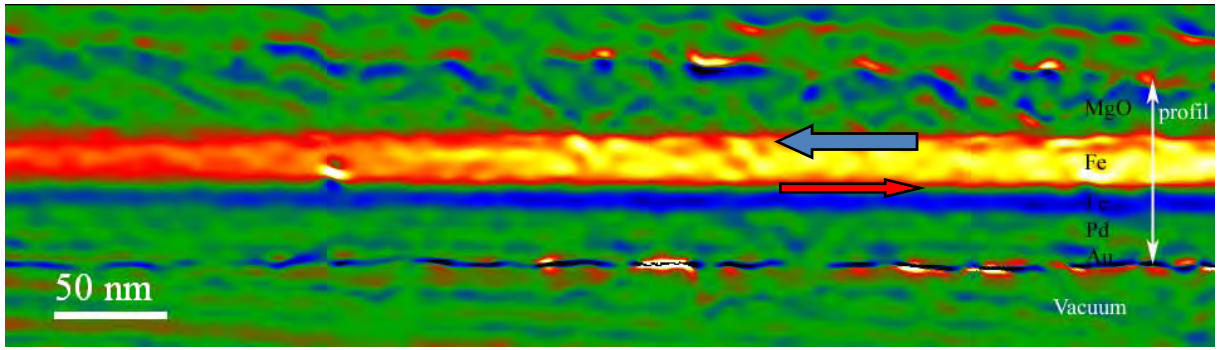
(a) TEM image, (b) Magnetic contribution to the phase shift, (c) Magnetic induction mapping, (d) micromagnetic simulation (OOMMF)

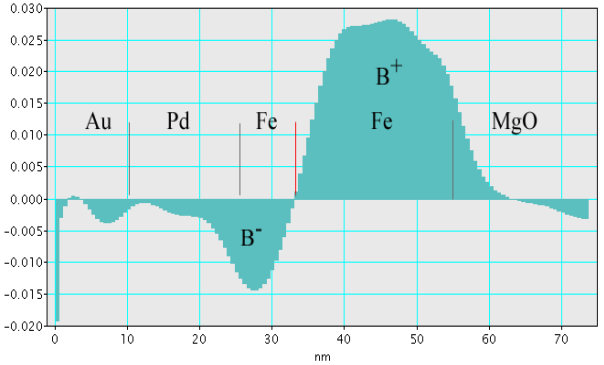
Magnetic multilayers

$\phi(x) = e/\hbar \int B_\perp(x)\, t(x)\, dx$



$d\phi(x)/dx = (e / \hbar) \,.\, t \,.\, B_\perp(x)$



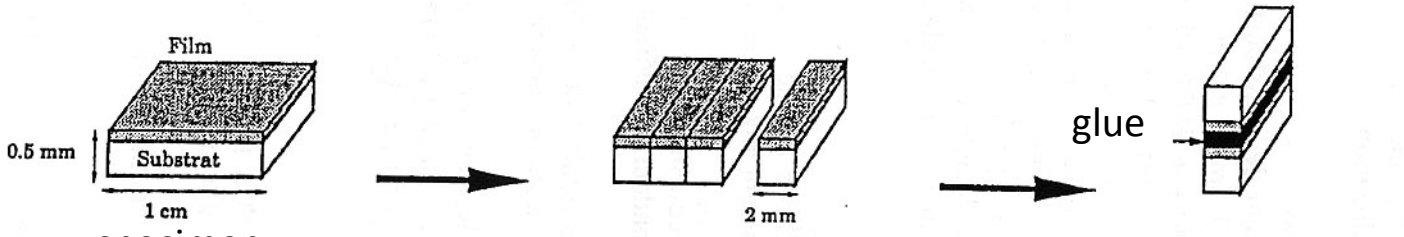


E. Snoeck, P. Baules, G. BenAssayag, C. Tiusan, F. Greullet, M. Hehn, A. Schuhl

J. Phys.: Condens. Matter 20 (2008) 055219.

Sample preparation

Cross Section

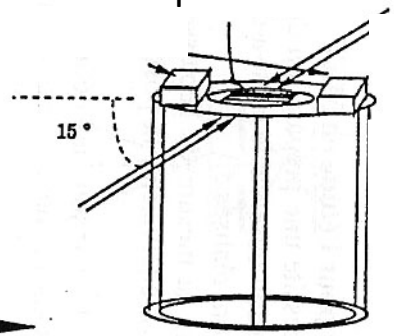


specimen

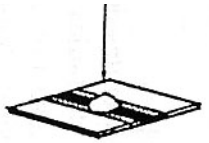
1st cut

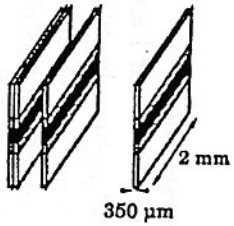
specimen

Ar+ ions



electrons





2nd cut

Mechanical grinding

(abrasive papers + dimpler)

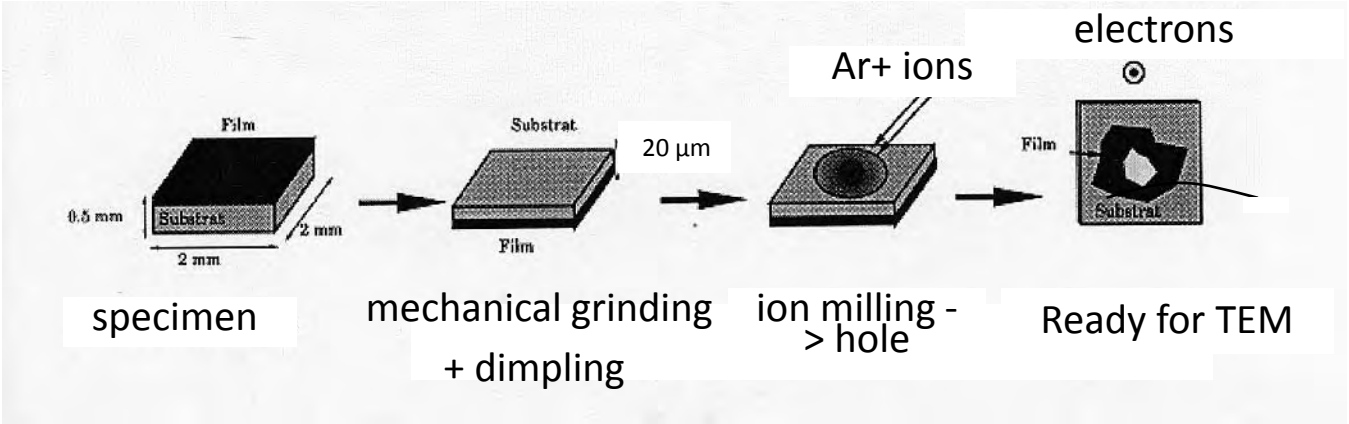
-> 10-20 µm

+ ion milling -> hole

Ready for TEM

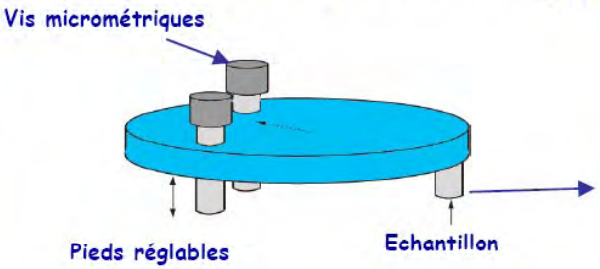
Sample preparation

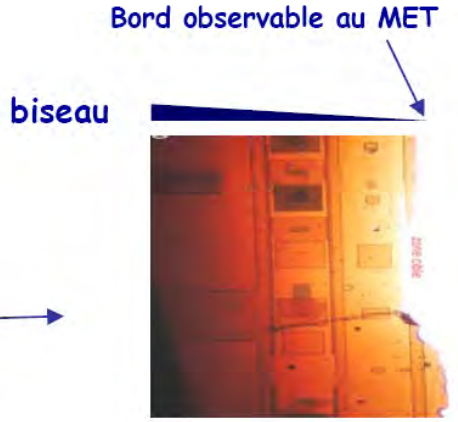
Plan view



Sample preparation

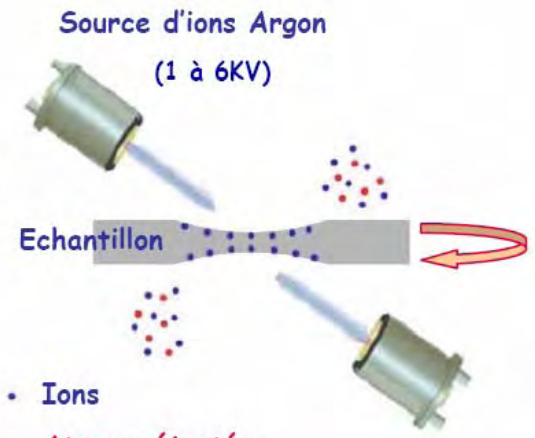
Le tripode





Amincisseurs ioniques DUAL ION MILL et PIPS (Gatan)

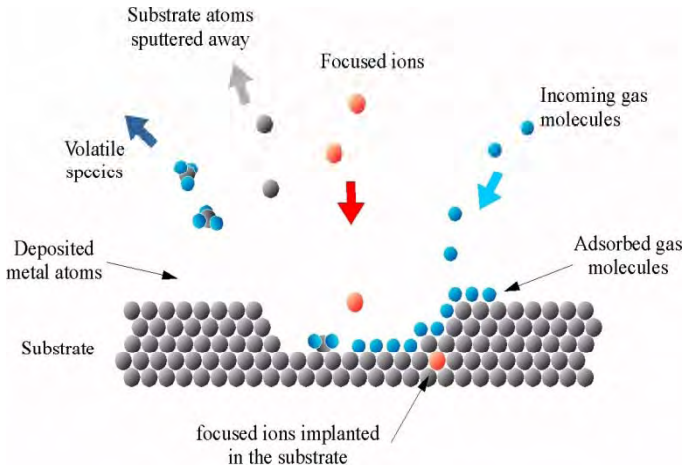
Principe

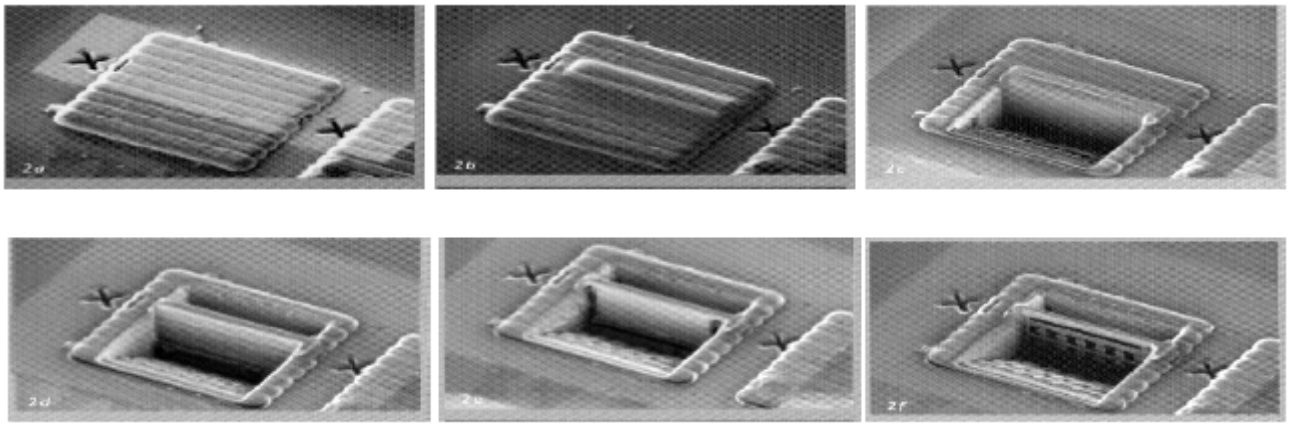


- Ions
- Atomes éjectés

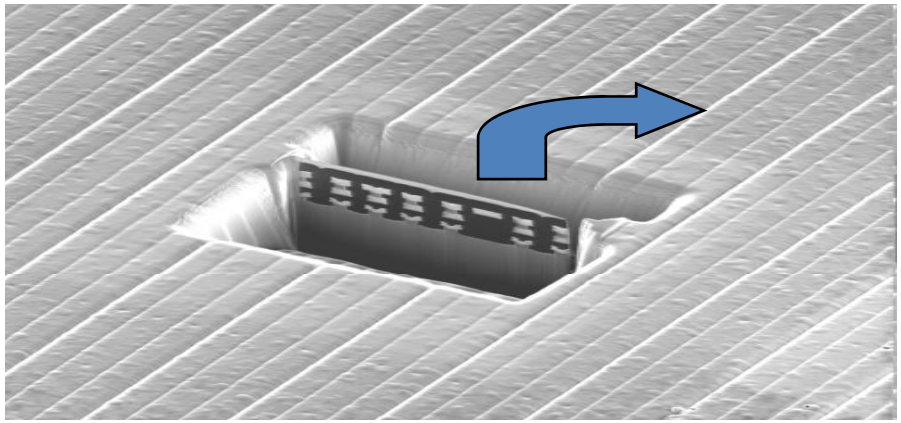
Jacques Crestou
Service préparation

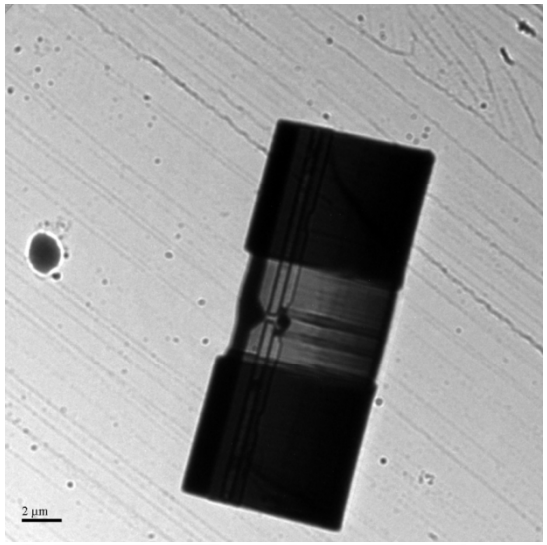
Sample preparation: FIB

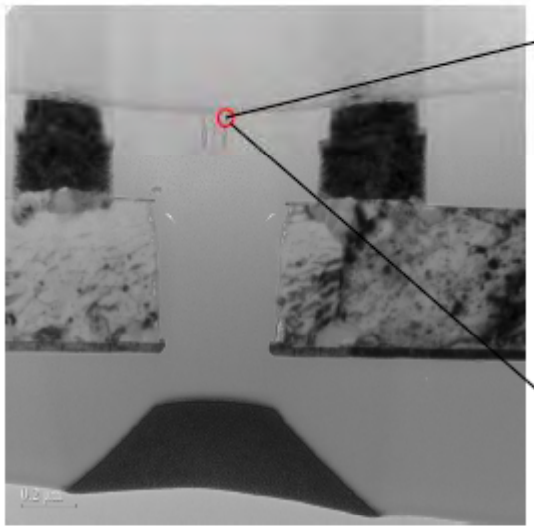


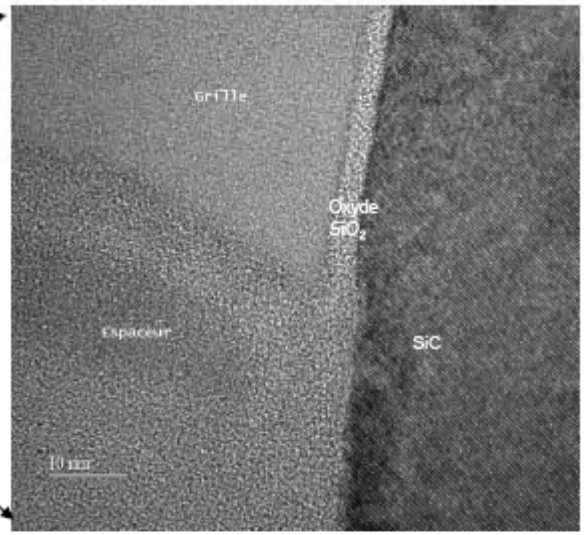


Sample preparation: FIB









Summary

Good TEM results from

50% specimen preparation,

20% TEM’s price

30% guy’s experience…