Préparation de pointes pour la sonde atomique par FIB

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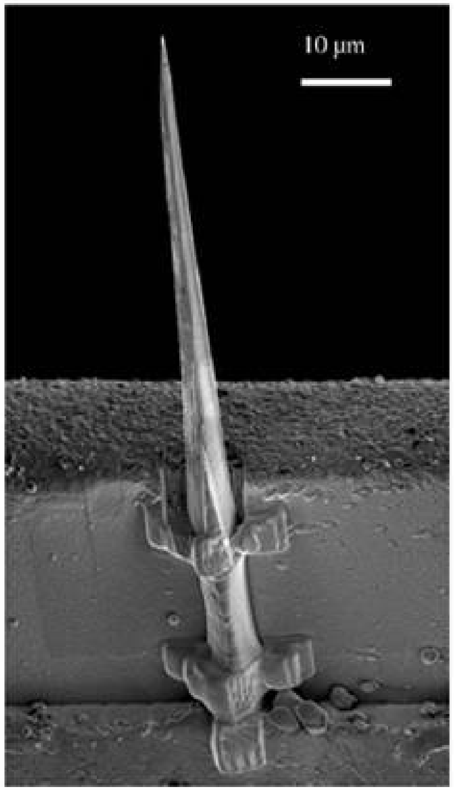
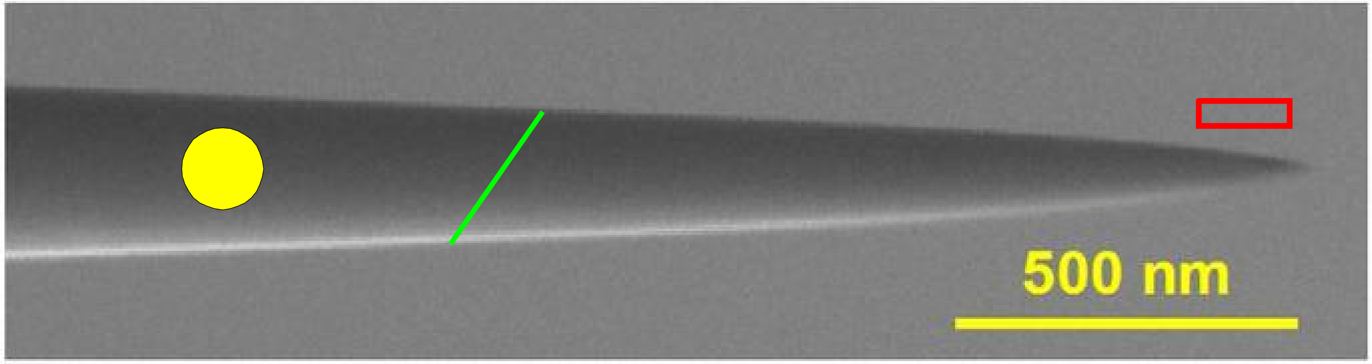


Fig. 9 – Secondary electron image of an atom probe post produced by ion milling. The post has been subject to 'lift-out' and then welded on to a Cu grid via platinum deposition.

L’échantillon de sonde atomique :



Forme de pointe

Section circulaire uniforme

Surface lisse

Faible angle de cône

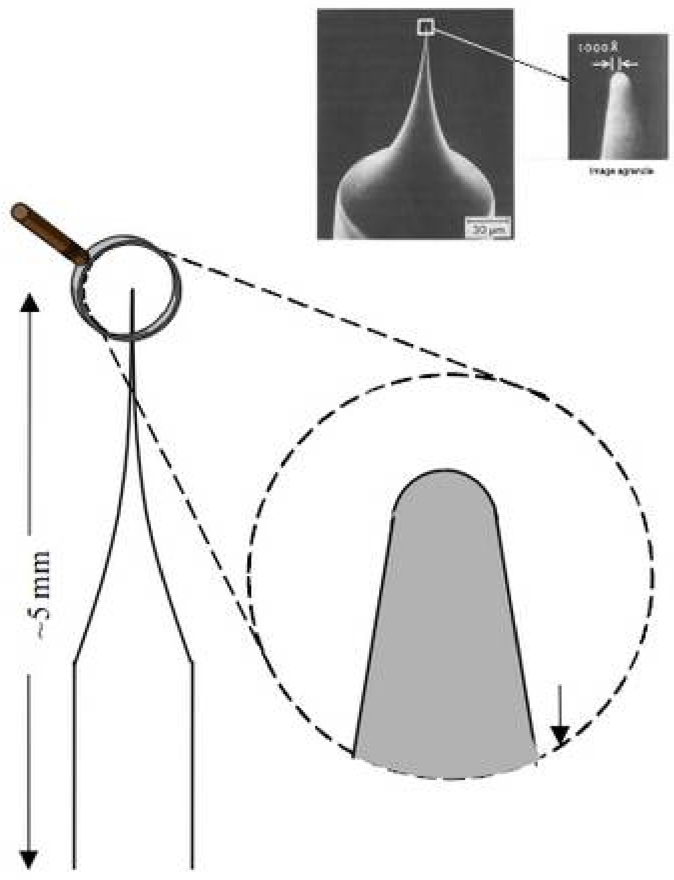
Rayon de courbure à l’apex < 50 nm

Longueur suffisante à partir de la base pour éviter tout écrantage du champ électrique > ~ 2 µm

Les méthodes de préparation ne doivent pas introduire d’artefacts ni modifier la microstructure. Les échantillons doivent être compatibles avec l’ultravide.

Le volume d’analyse : les 100-200 premiers nm…

Effet de pointe



$$F = \frac{V}{\beta R}$$

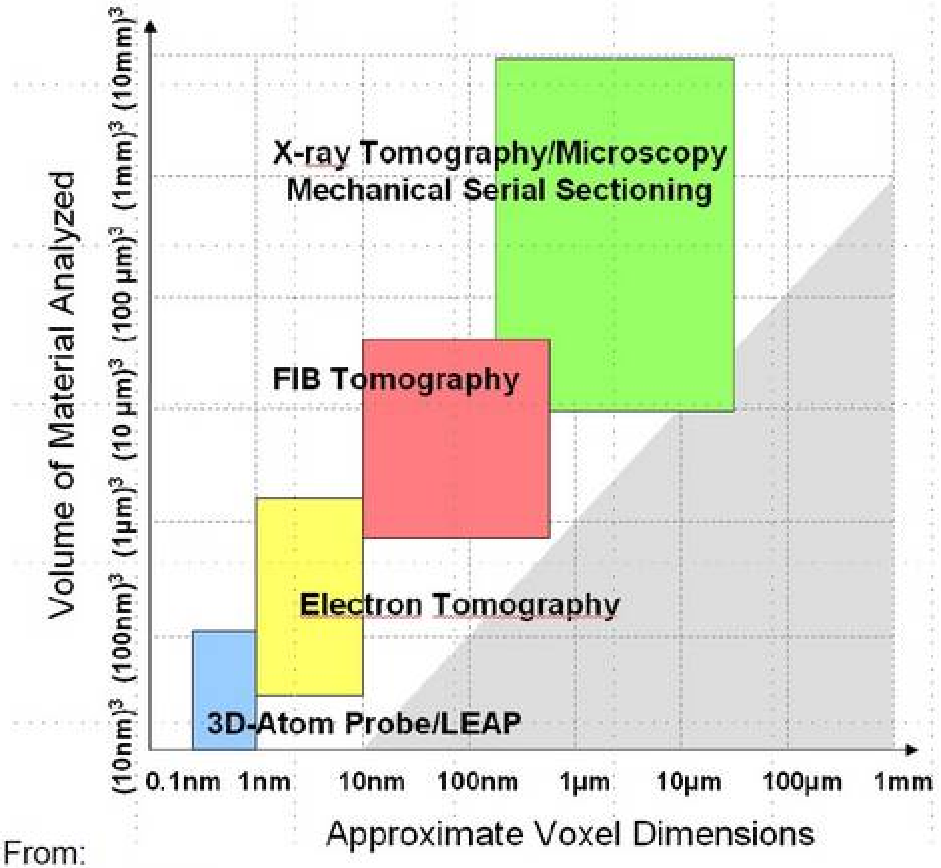
R~50 nm,
V~10 kV,
β~5.

F~ 40 V/nm !!!
à la surface de la pointe

β a shape factor (electrostatic)
β = 1 for a sphere
β = 2 for a hemisphere on a cylinder
β < 8 generally

Sonde atomique Tomographique (APT)

= **3D analytical high resolution microscope**



From:
Uchic and Holzer
MRS Bulletin, 2007

Avantages

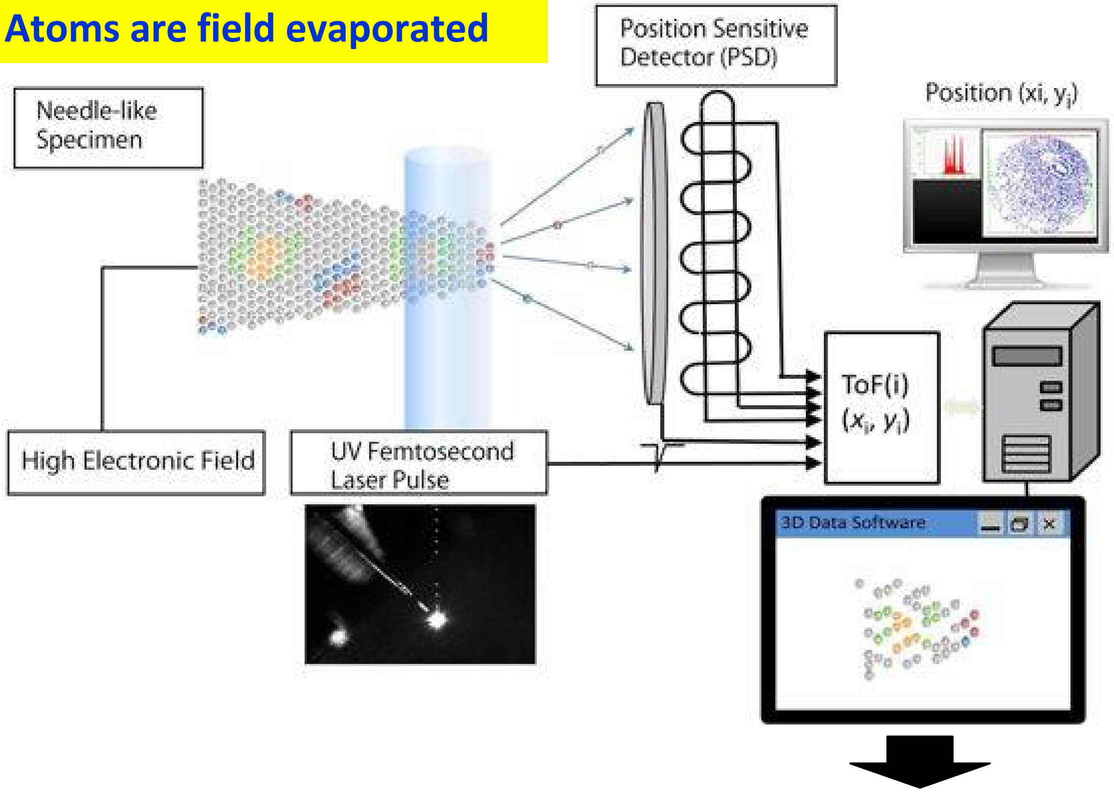
Résolution spatiale
Même efficacité de détection pour tous les éléments (même les légers)
Tomographie 3D

Inconvénients

Petit volume d'analyse
Conductivité électrique
Fragilité échantillon
Préparation échantillon

Sonde atomique Tomographique (APT)

Atoms are field evaporated



Position sensitive detector

→ 3D mapping of atoms

Time of flight mass spectrometry

→ Chemical information

Mapping at the atomic scale of the 3D spatial distribution of atoms in the sample

(Depth resolution = 0,1 nm and lateral resolution ~ 0,3 nm)

Grandissement G ~ x 10^7.

Aire d'analyse ϕ^2, ϕ = D/G ~ 10nm (D detector size = 10 cm)

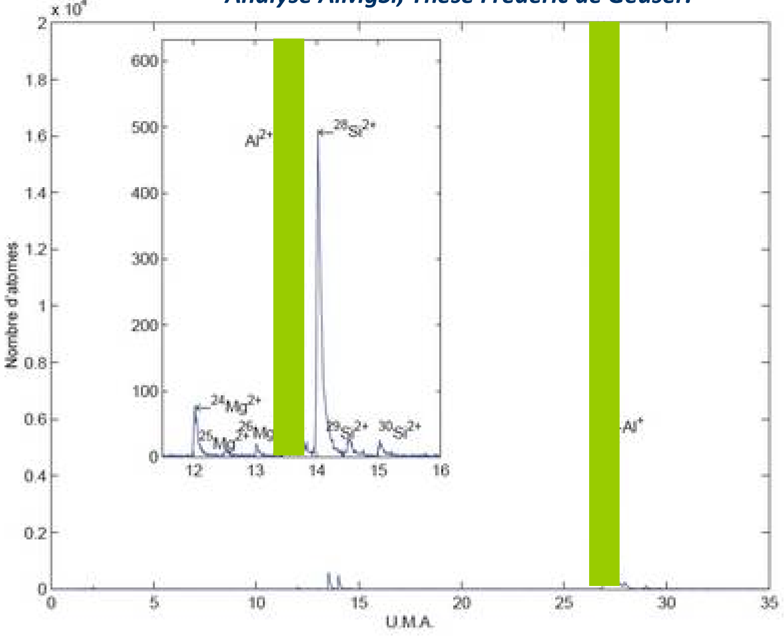
Sonde atomique Tomographique (APT)

Time of flight mass spectrometry

+

Position sensitive detector

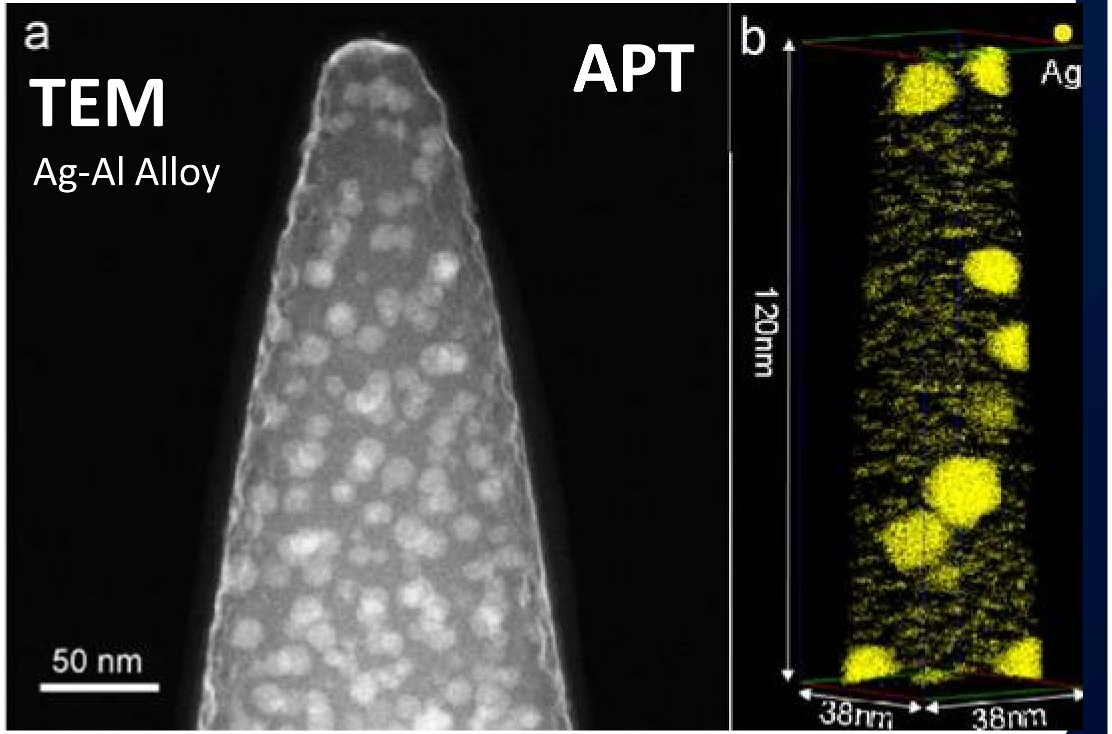
Analyse AlMgSi, Thèse Frederic de Geuser.



rapport masse/état de charge

Al^+ : 27/1= 27

Al^{++} : 27/2=13.5



Volume d'analyse = 80 x 80 x 200 nm^3

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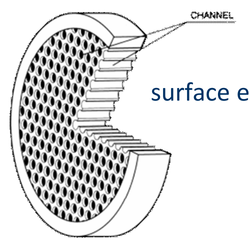
Limite de détection



surface effective MCP = 60 %

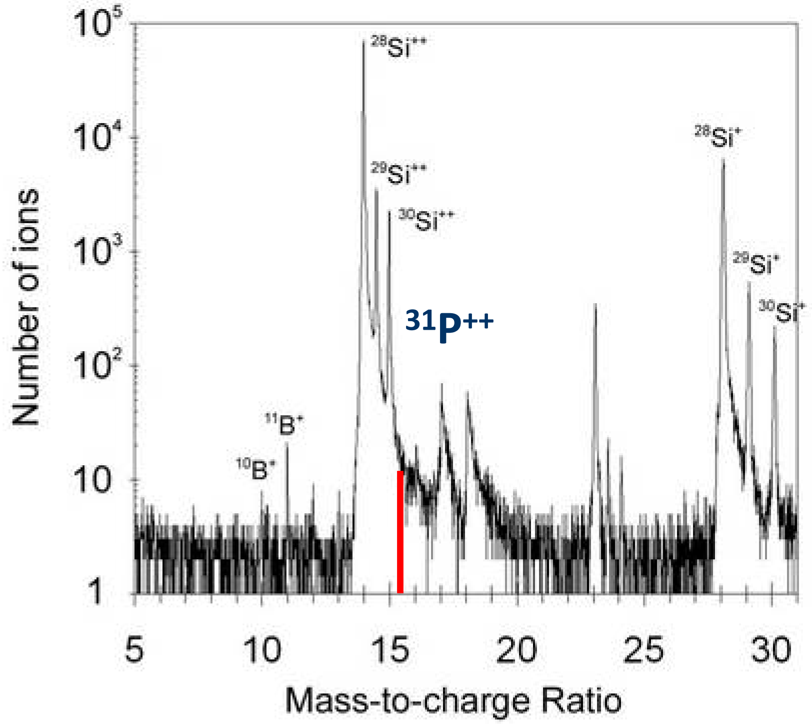
Sensibilité de la sonde atomique dépend :

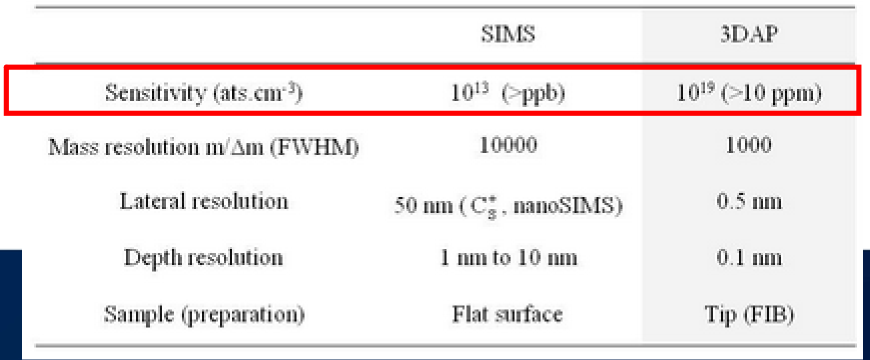
- de la résolution en masse
- du rendement de détection
- de la composition de l'alliage

Limite de détection (éléments d'addition)

- autour de 10 ppm at. si loin des pics principaux.
- autour de <u>50-100 ppm at.</u> à proximité des pics principaux

Bore dans Silicium

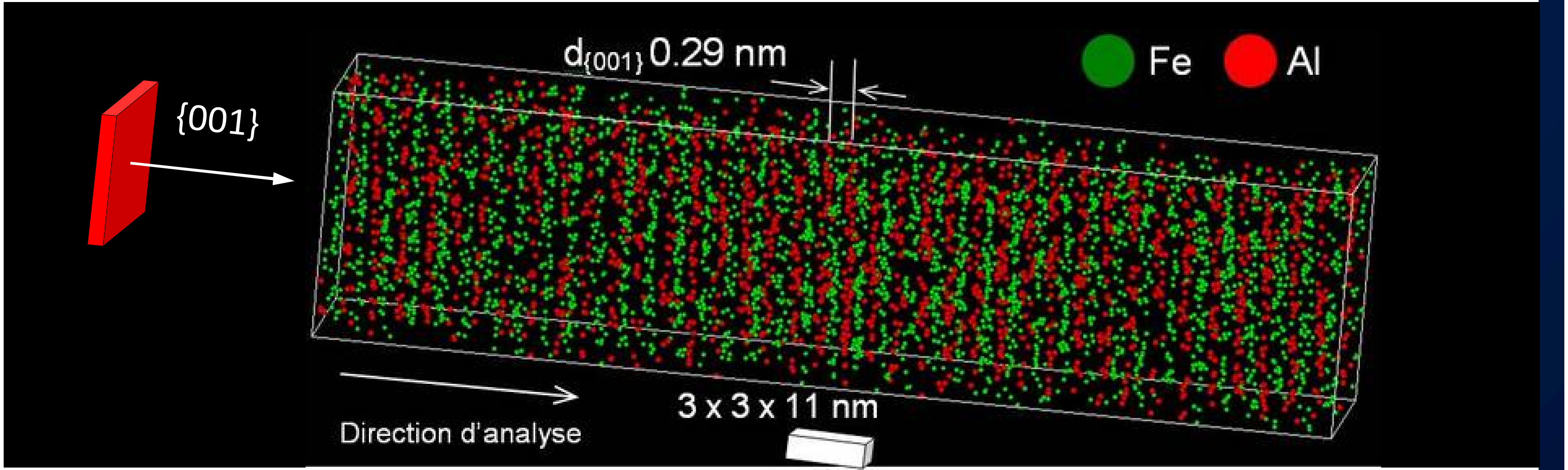


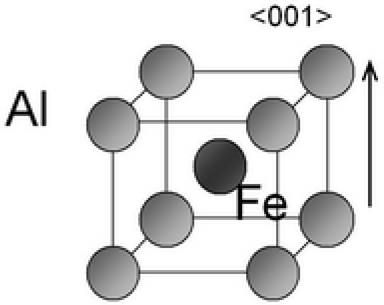


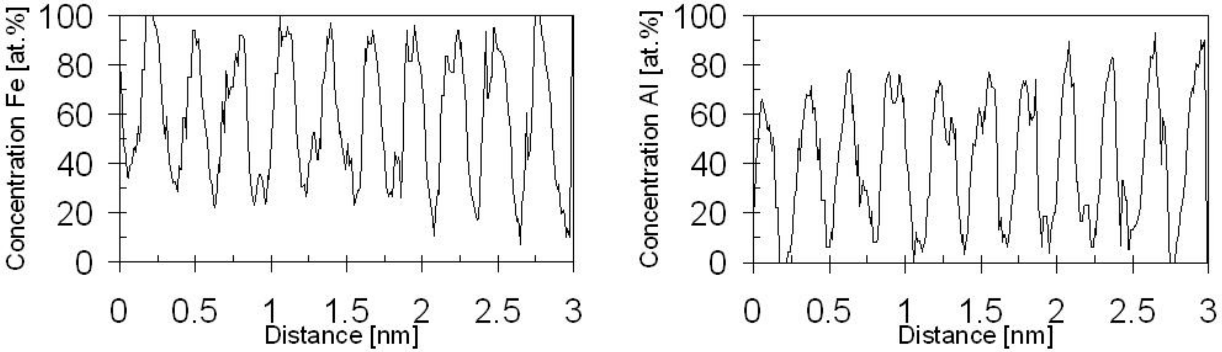
	SIMS	3DAP
Sensitivity (ats.cm^{-3})	10^{13} (>ppb)	10^{19} (>10 ppm)
Mass resolution m/Δm (FWHM)	10000	1000
Lateral resolution	50 nm (C_s^+, nanoSIMS)	0.5 nm
Depth resolution	1 nm to 10 nm	0.1 nm
Sample (preparation)	Flat surface	Tip (FIB)

Structure ordonnée B2

Plans {001} de l'alliage Fe-40Al (B2)





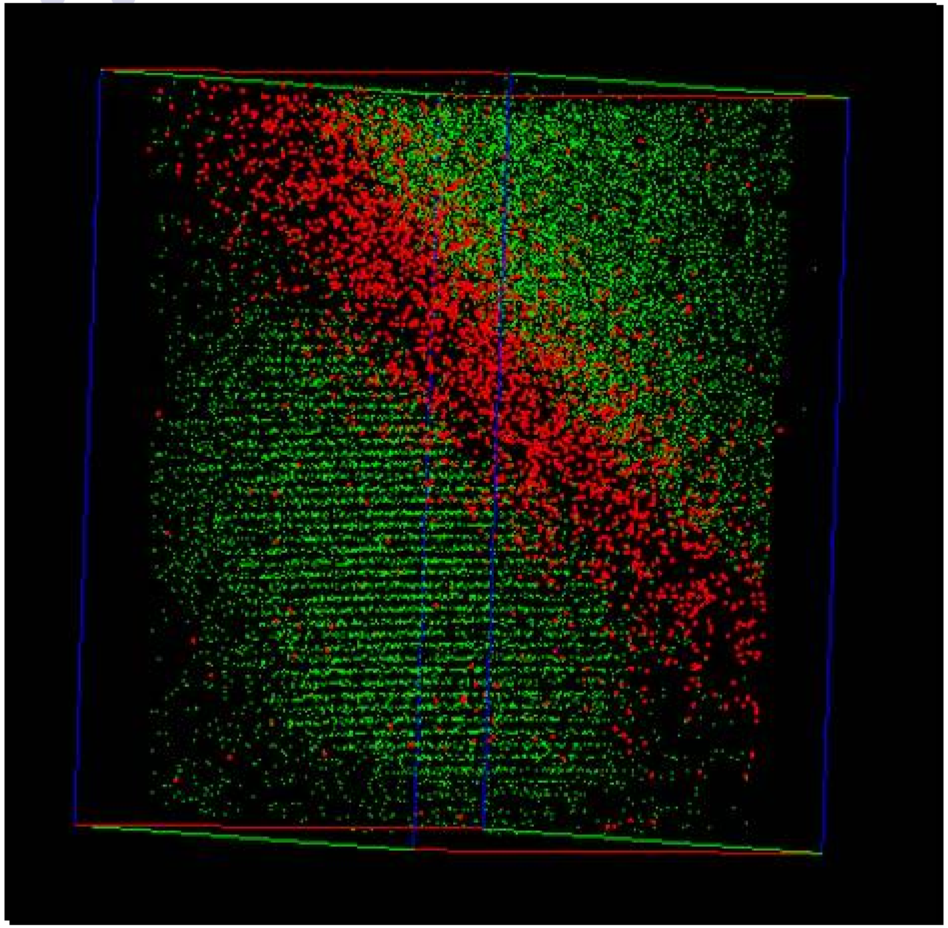


Profils de concentration (Fe, Al) suivant une direction de type <001>.

Depth resolution = 0,1 nm and lateral resolution ~ 0,3 nm

Ségrégation aux Joints de Grains

Superalliage à base de nickel N18



Observation of two grain riched in Al atoms

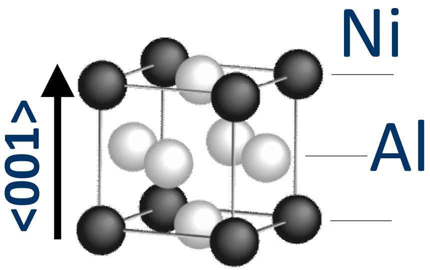
Observation of atomic planes

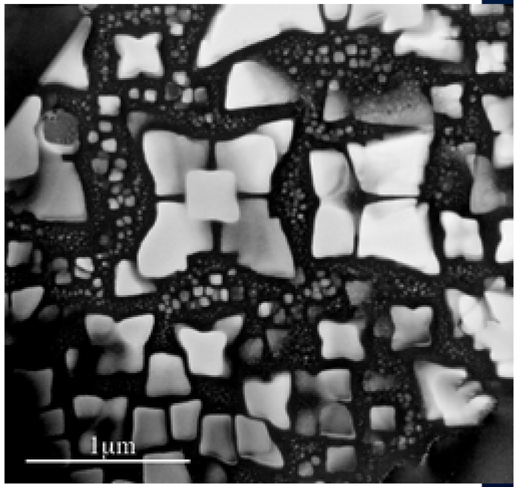
Segregation of B atoms at grain boundaries

Ni atoms are not represented

🟢 Al

🔴 B

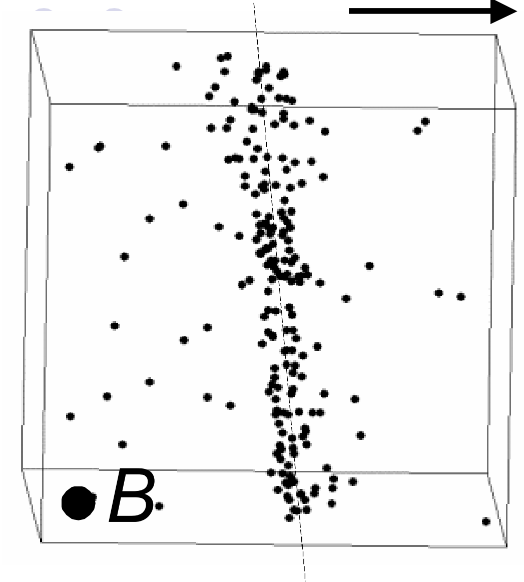




Dislocation coin

« Boron Cloud »

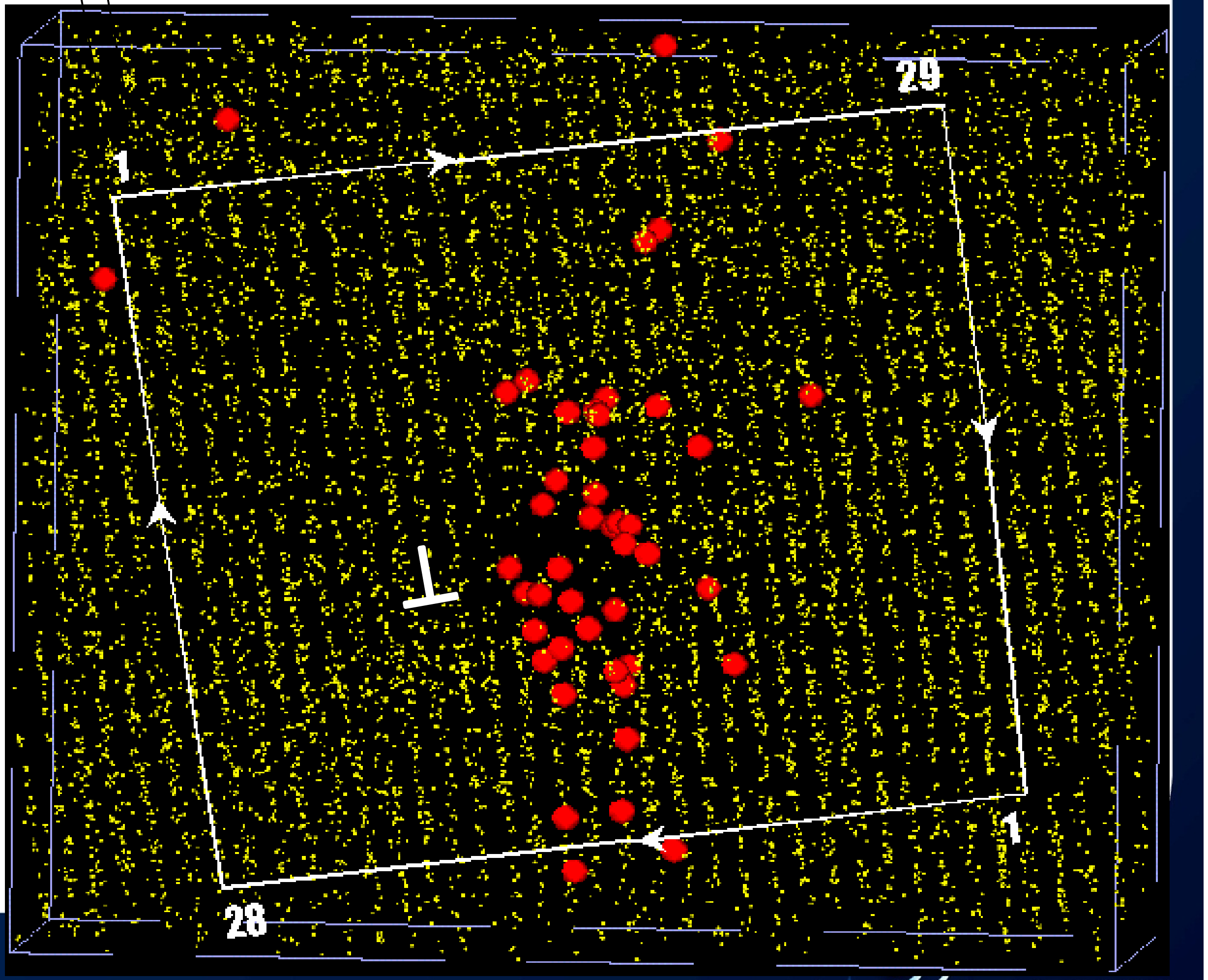
{001}



TOP VIEW

$d_{100} = 0.29$ nm

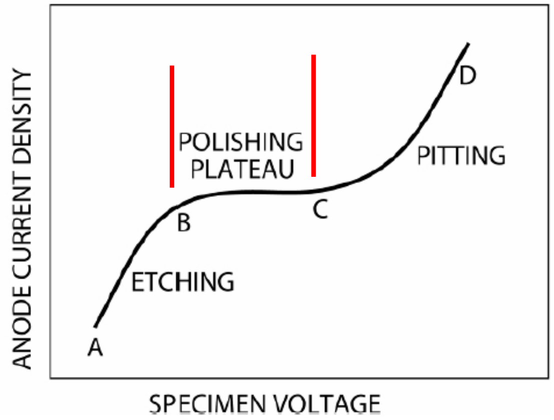
$\vec{b} \perp \{001\}_{FeAl}$



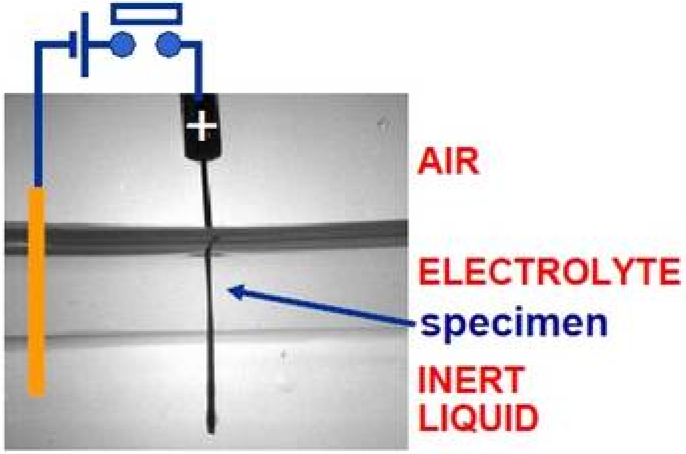
🔴 B

🟡 Al

Electropolissage

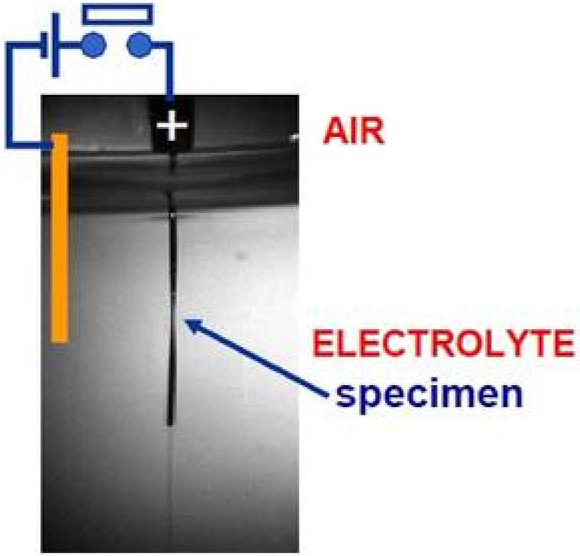


Double layer method



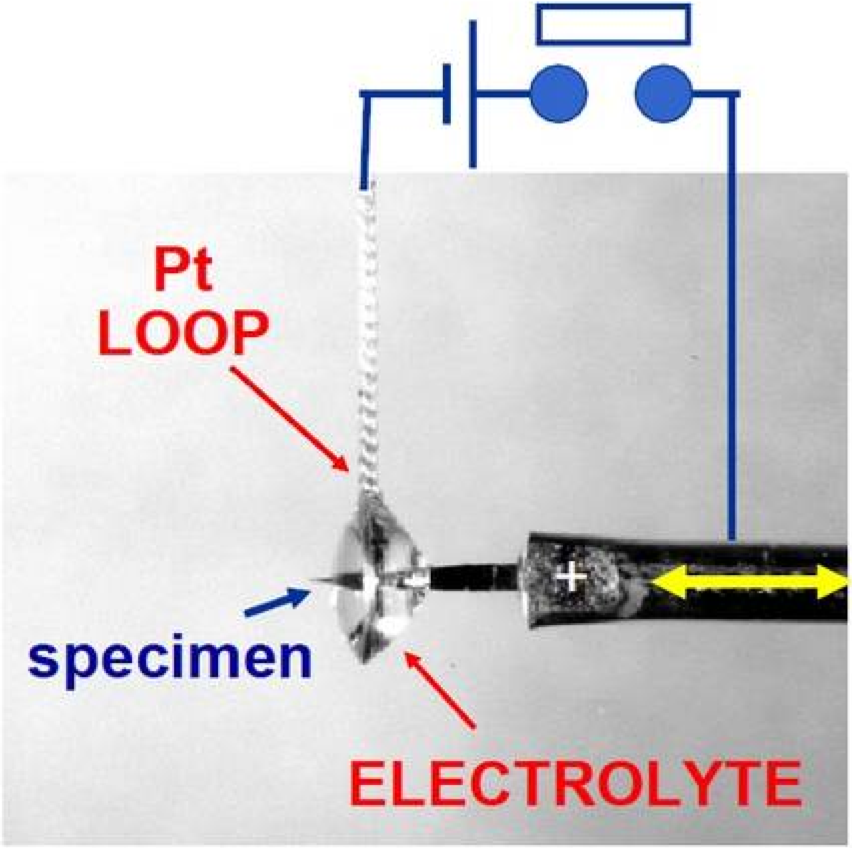
Electropolishing only occurs in the electrolyte producing a necked region.

Stage 2



Continued until the neck cannot support the weight of the lower portion.

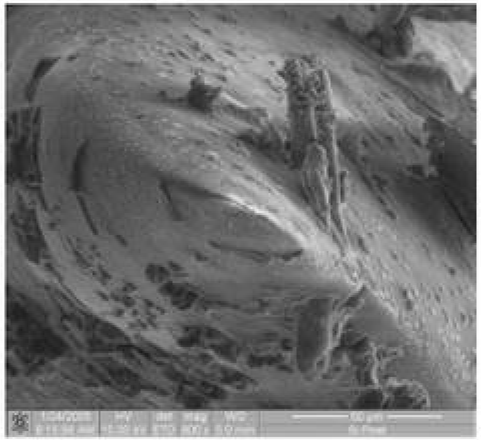
Electropolissage : micro-loupe

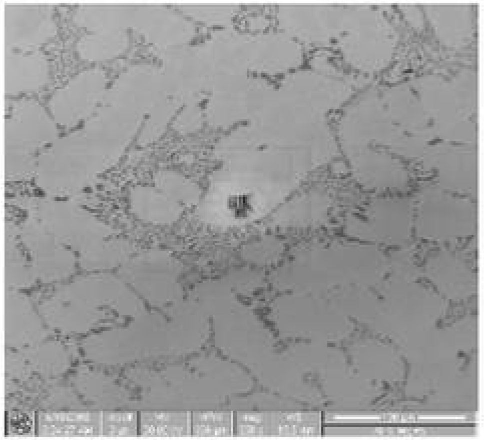


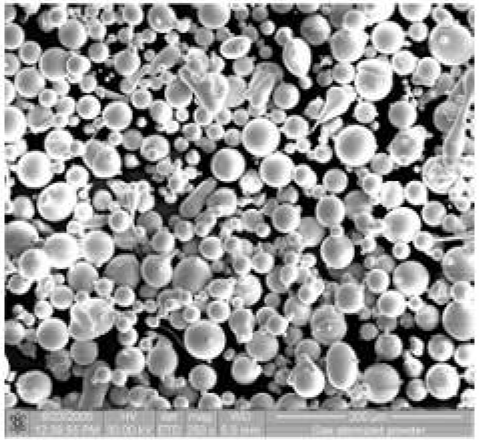
Specimens can be resharpened by slowly moving the specimen in a drop of electrolyte while applying voltage pulses.

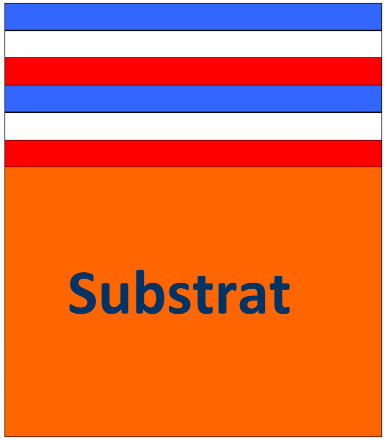
Procedure monitored under low power optical stereoscope

Pourquoi utiliser le FIB ?


Preferential polishing


Inhomogeneous


powder



Thin films

Electropolissage

pas conseillée pour certains matériaux (Zircaloy,…)
pas utilisable pour films minces, couches de surface/implantées, poudres, rubans, prélèvement d’une zone particulière (JG,…).

Focused Ion Beam (FIB)

alternative efficace pour un large spectre de matériaux et de géométrie

Accessoires Cross-Beam

Pt - GIS

FEG SEM column

Micromanipulator

1-30 keV
FIB column

SESI
Electron detector

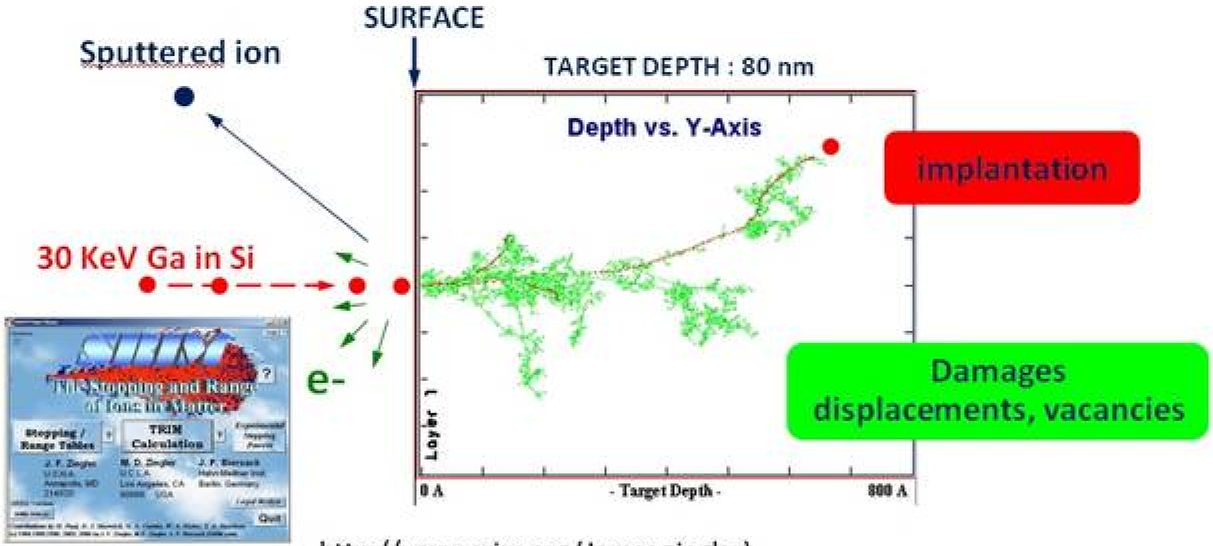
Stage

ZEISS – NVISION 40

Procédure de préparation

Localiser la zone	SE2, ESB, EBSD. SI…
Protection	PECS, EBID
Lift-out	FIB : 30kV (Rough Mill)
Soudure sur pointe	IBID (FIB : 30kV)
Annular milling	FIB : 30kV à 1 kV
AP experiment	Atom probe

Amorphisation et implantation



Sputtered ion

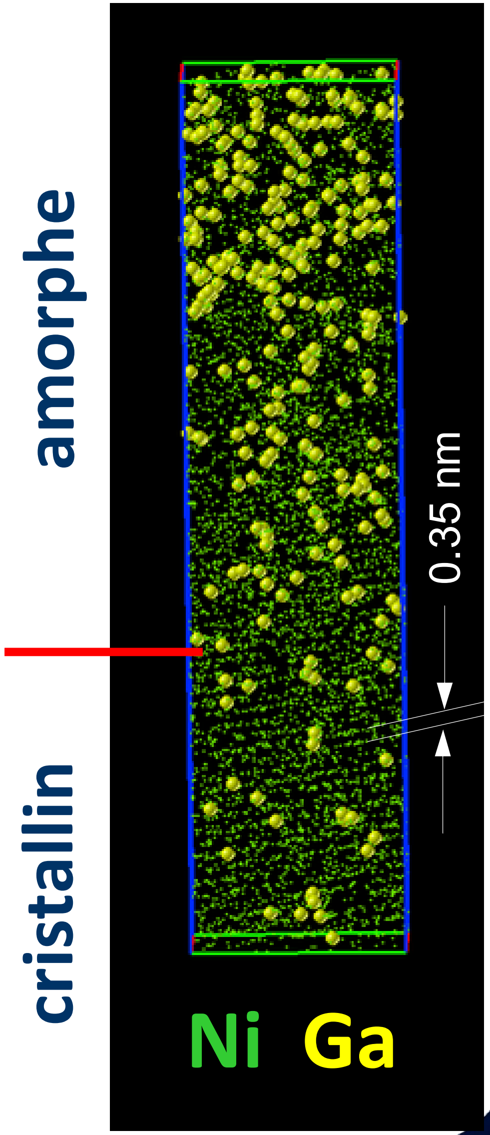
30 KeV Ga in Si

e-

implantation

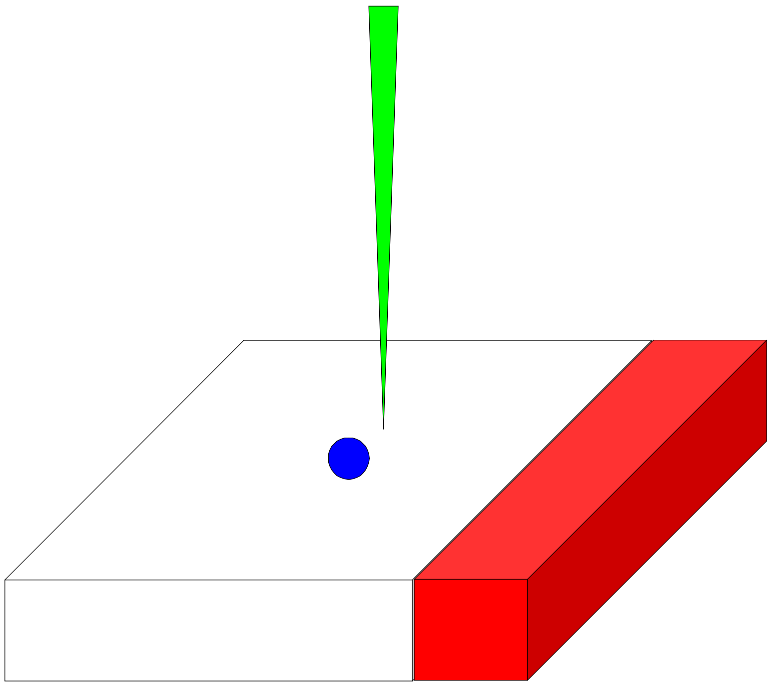
Damages
displacements, vacancies

http://www.srim.org (James ziegler)

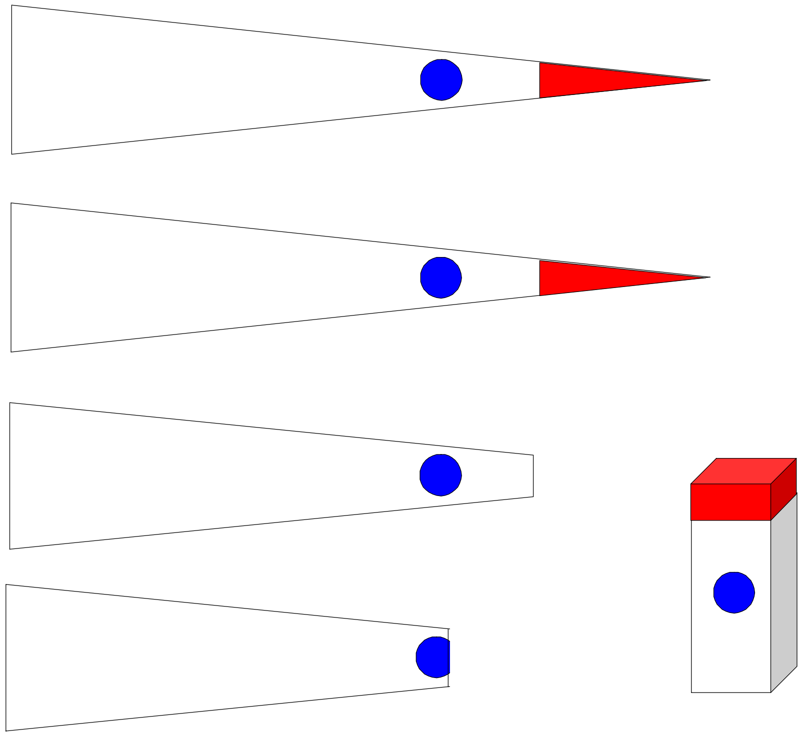


Couche de protection

MET



APT



Couche de protection

Model 682 PECS™ GATAN
Precision Etching Coating System

Element	Evaporation Field	Range (nm)
Ag	24 V/nm	10.4
Cu	30 V/nm	9.8
Ni	35 V/nm	9.2
Cr	29 V/nm	11.2
Si	33 V/nm	26.9

Pt Evaporation Field : 45 V/nm !

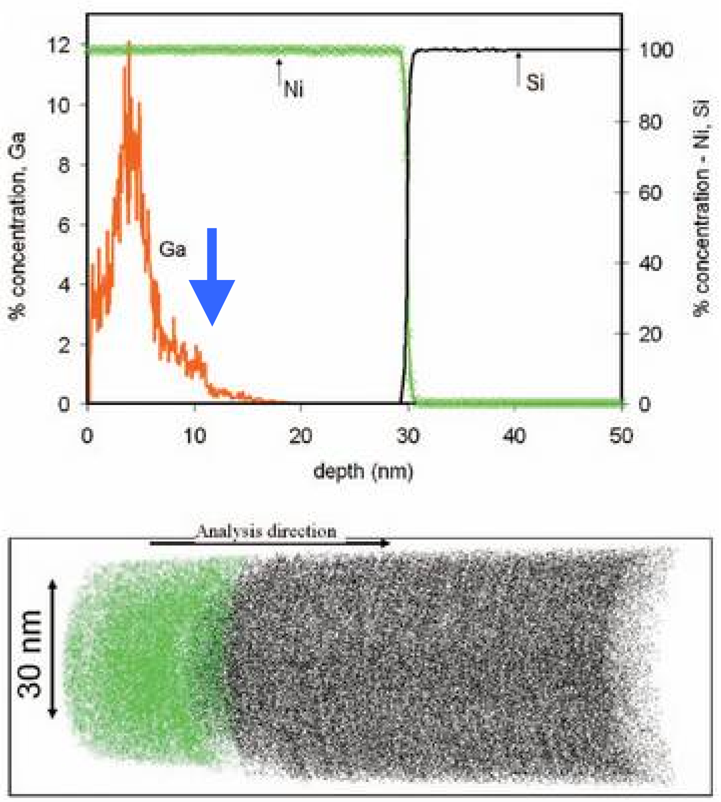
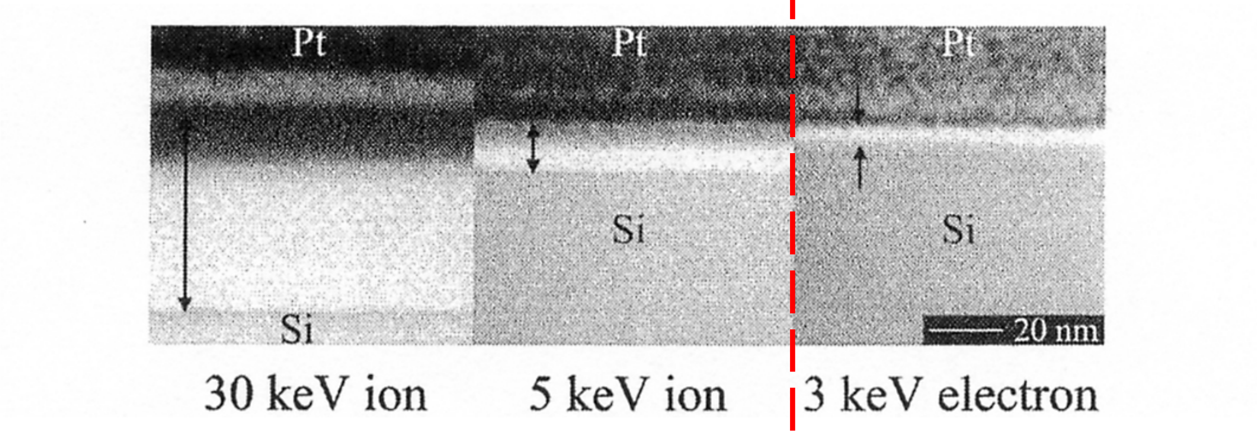
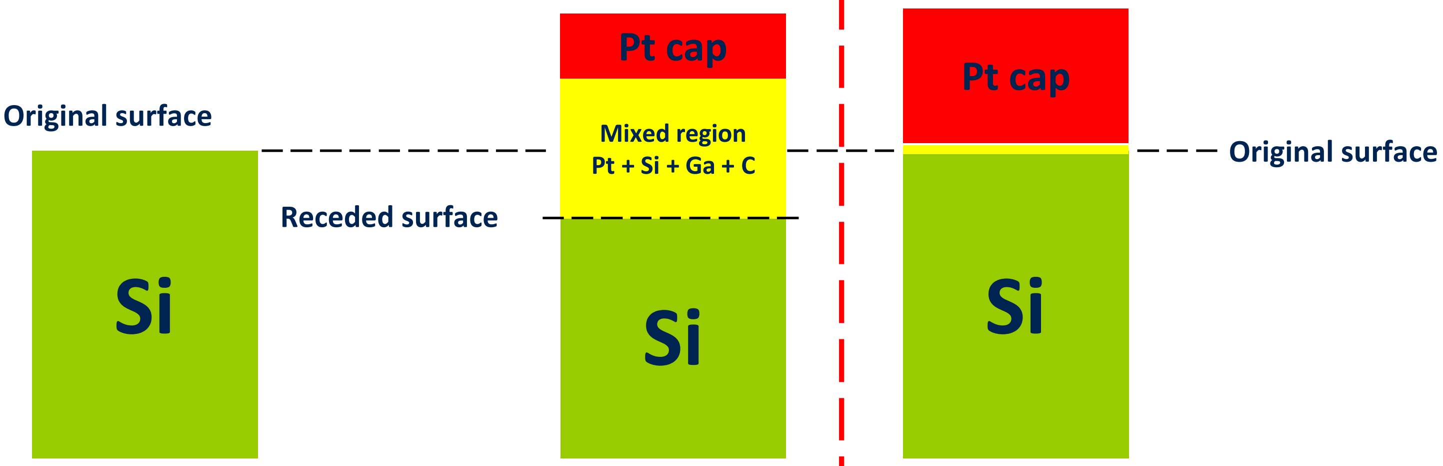


FIG. 3. (Top) Ga profile in the Ni cap for the FIB-sharpened sample whose atom map is shown. (Bottom) 3D atom map showing Si (black) that was protected with a Ni cap (green) during the FIB sharpening process.

Pt IBID vs Pt EBID

Pt : IBID/EBID





Original surface

Pt cap

Mixed region
Pt + Si + Ga + C

Receded surface

Pt cap

Original surface

Si

Si

Si

Lift Out

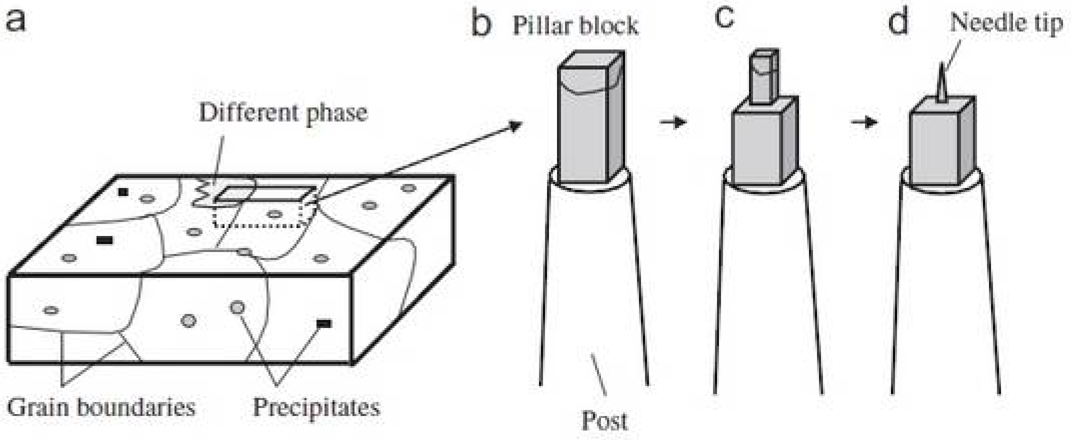
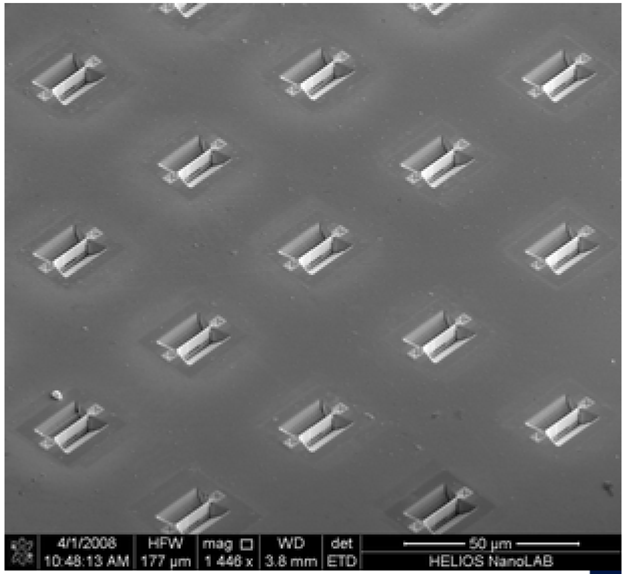
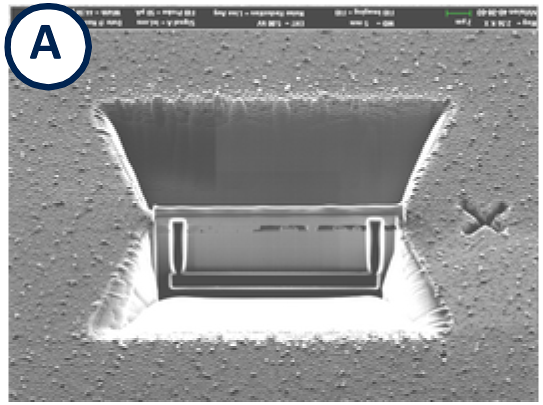


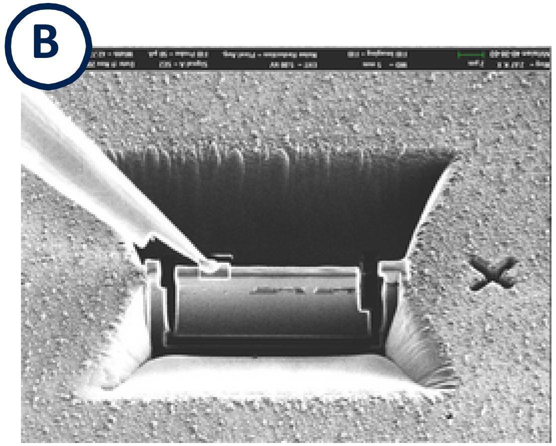
Fig. 3. Schematic drawing of needle specimen preparation flow for the site-specific region analysis: (a) sampling from the object region of interest (the phase interface), (b) pillar block mounted on the needle stage, (c) square rod formed in upper potion of the block, and (d) final specimen form with a tip containing the object region.

Lift Out





Sample after rough milling



The tip of the micromanipulator is welded to the lamella.

The sample is cut out

The sample is cut out and lifted out of the substrate

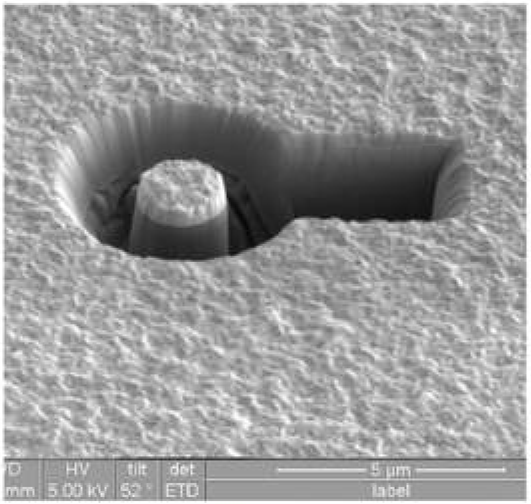


Fig. 9. Region of interest isolated by an annular mill pattern. Behind the annular milled regions is a trench that allows micromanipulator access to the tip.

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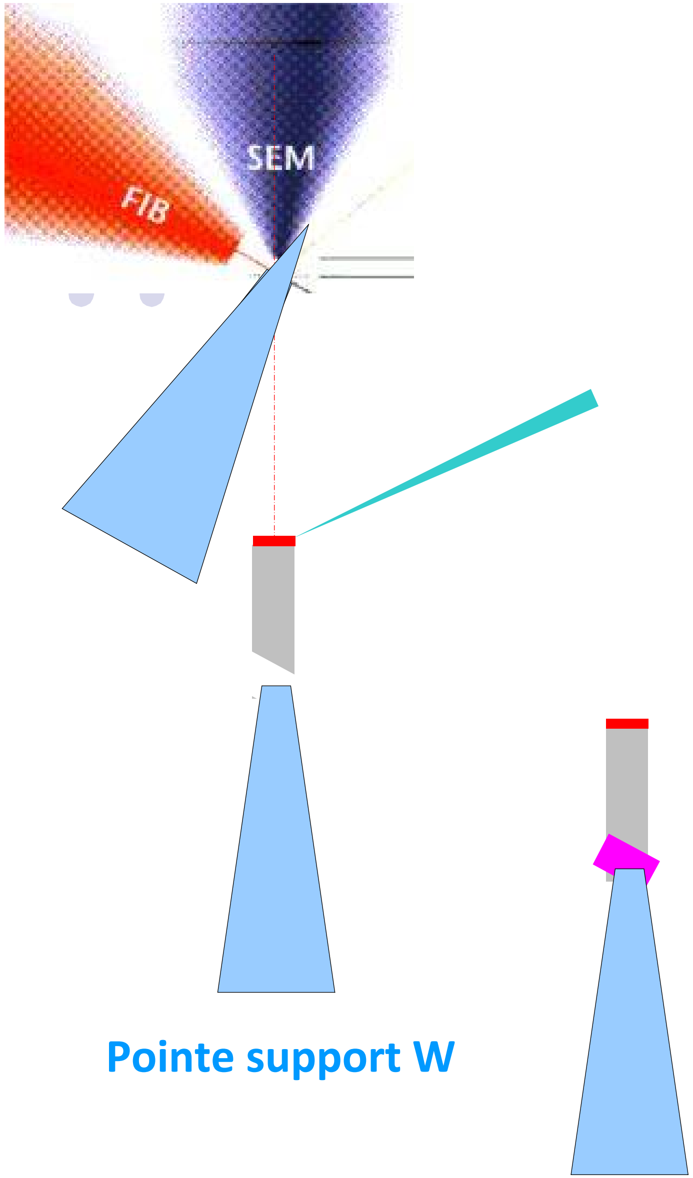
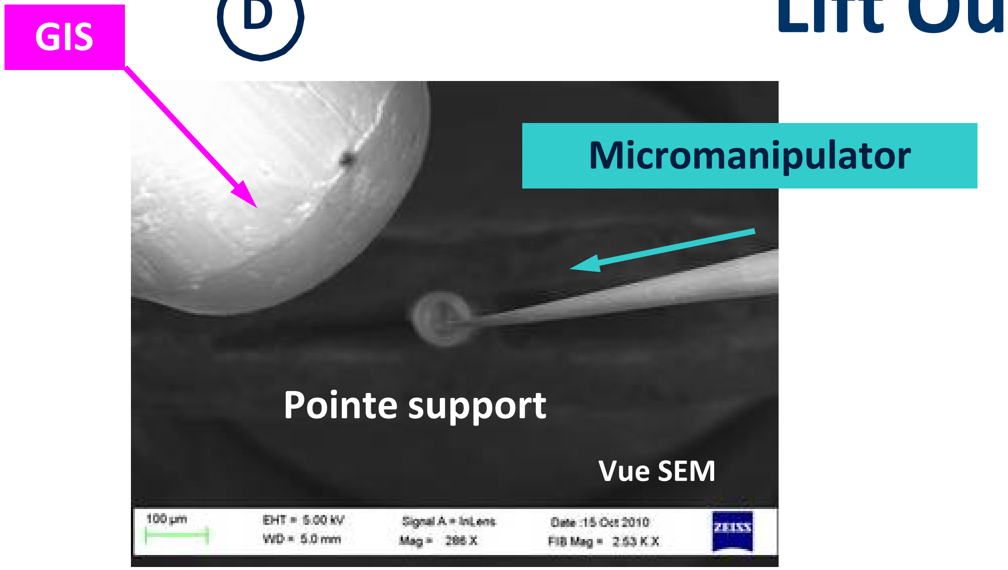
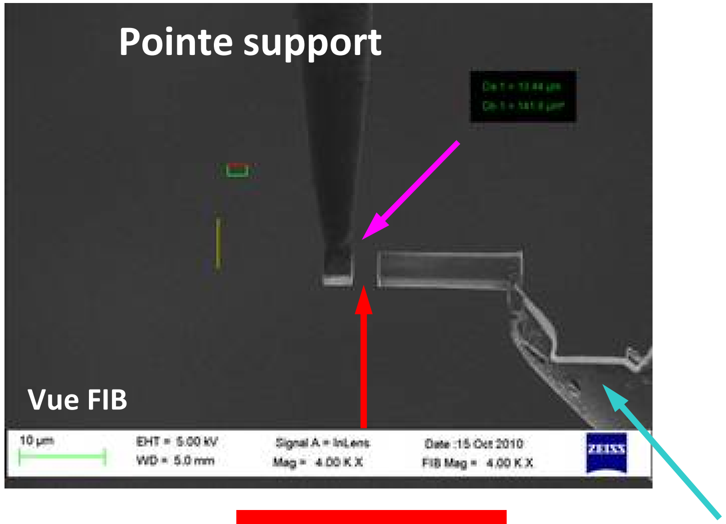
Lift Out

D



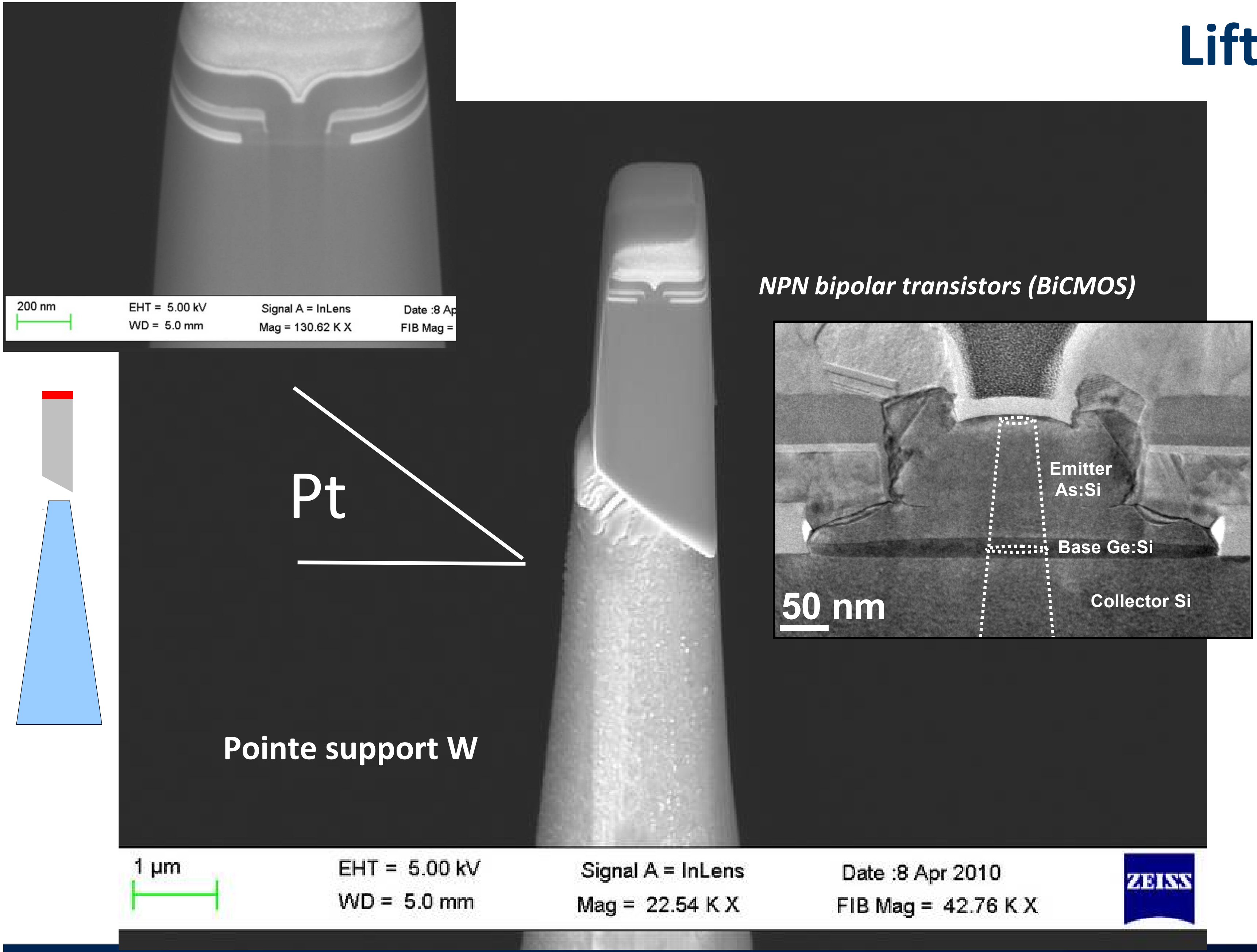
Pointe support W





FIB cut

Lift Out

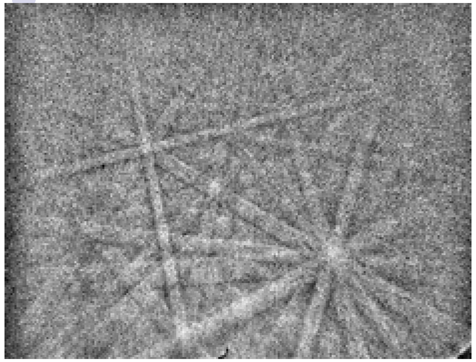


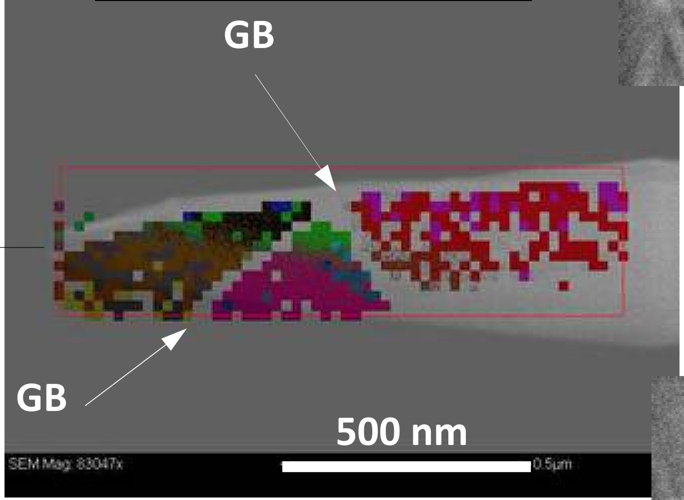
NPN bipolar transistors (BiCMOS)

Pointe support W

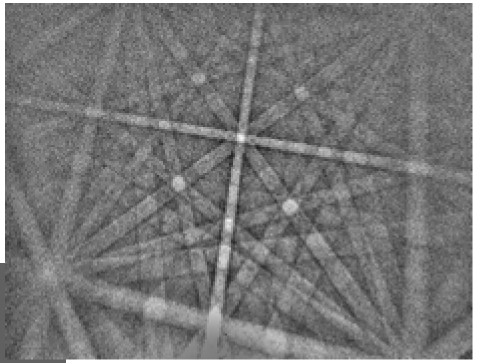
Détection de joints de grains par EBSD

G1





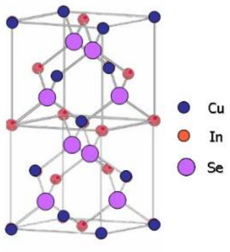
G3



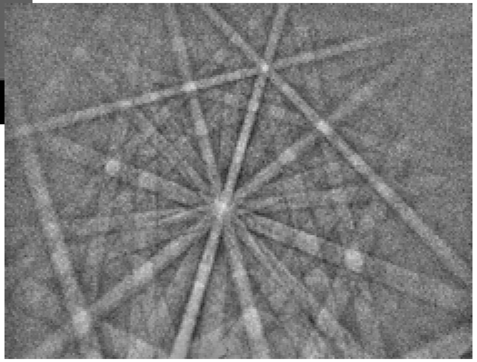
Chalcopyrite, tétragonal
binning 2 x 2, 20 kV, aperture 60 µm
Grid distance X,Y : 20 nm

RD

axe de la pointe



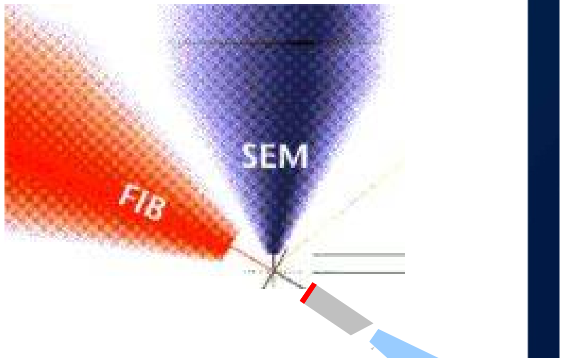
G2

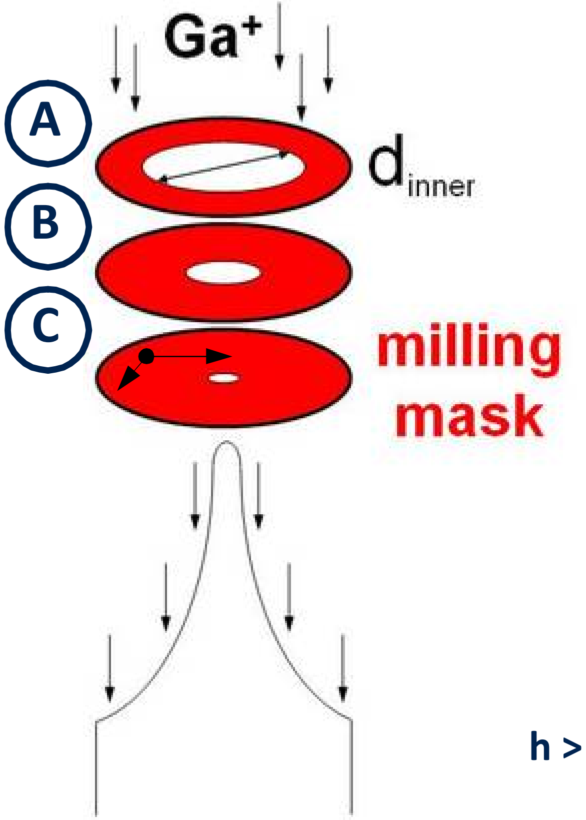


- Localisation des joints de grains
- Désorientation des joints (R, θ), plan du joint $\vec{n}$
- Direction cristallographique dans l'axe de la pointe

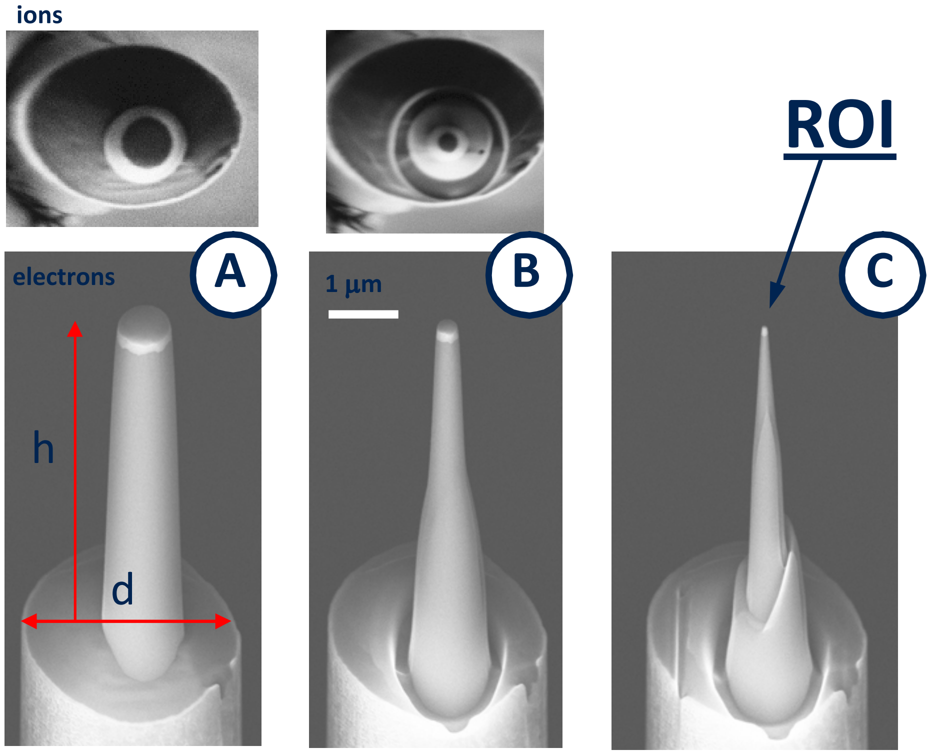
Annular Milling : Masque

The sample is aligned along the beam direction, the inner diameter of the circular mask and the milling current are reduced after each milling stage.





h > 2 x d



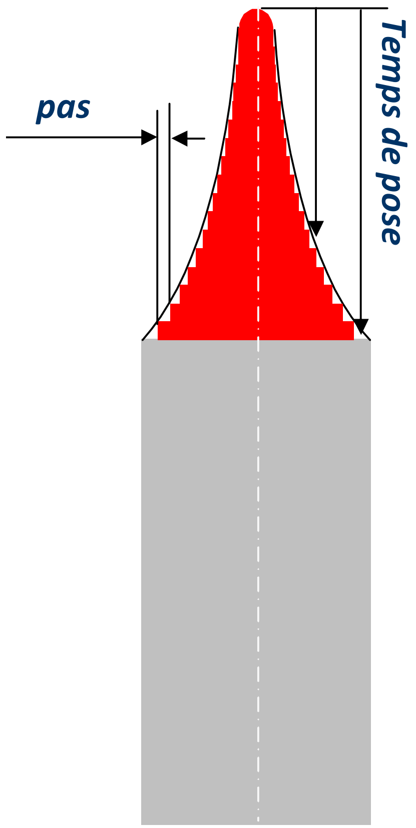
Rough Mill
few nA,30 keV

Sharpening
100-500 pA, 30keV

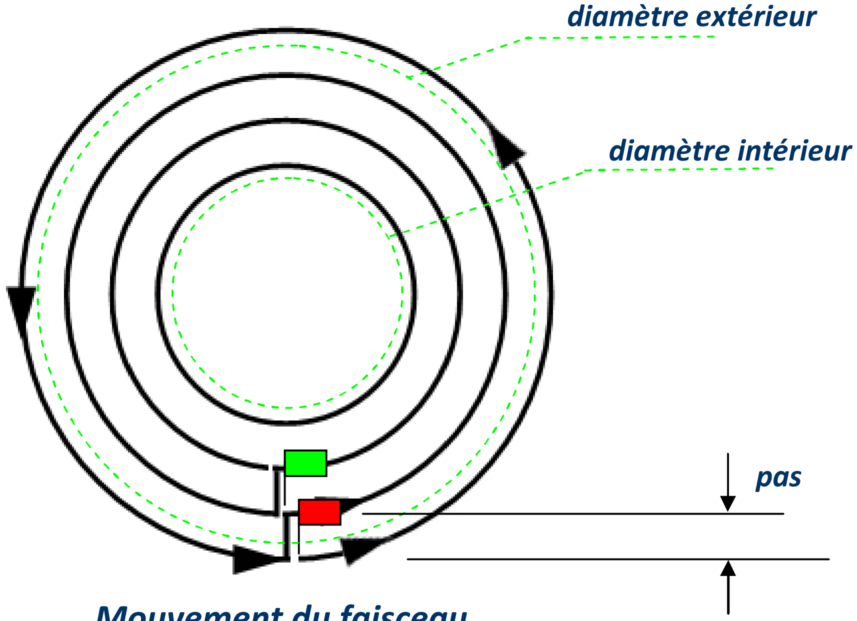
Final
few pA

Annular Milling : FIB spot

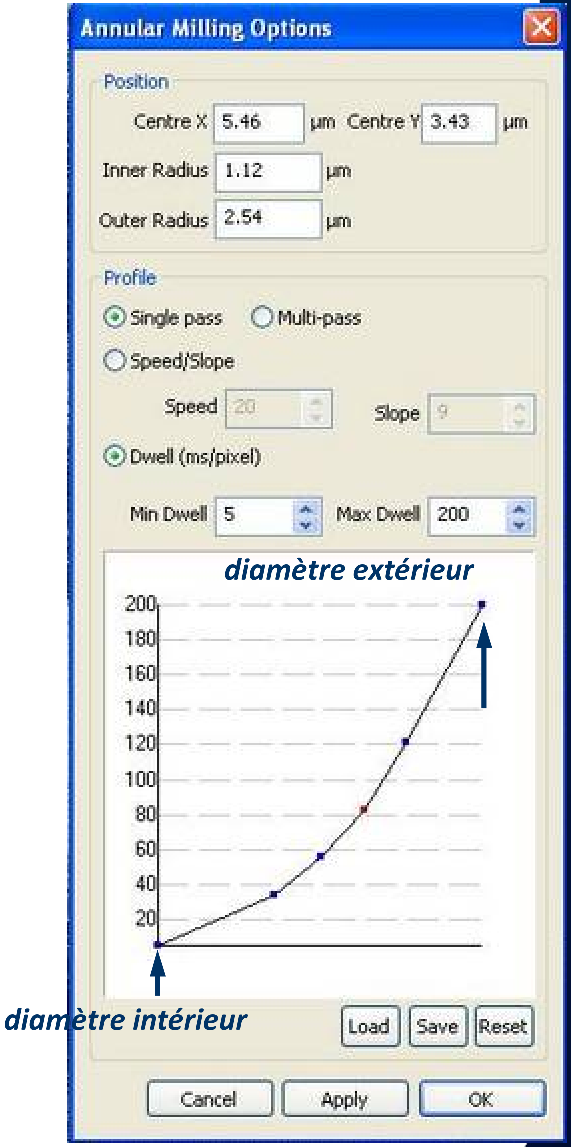
Variation du dwell time avec le rayon d’usinage : plus on se rapproche du diamètre interne plus la vitesse d’usinage augmente; Variations linéaire, en 1/sin….



Forme de pointe souhaitée



Mouvement du faisceau
Départ - Arrivée



Annular Milling Options

Position

Centre X 5.46 µm Centre Y 3.43 µm

Inner Radius 1.12 µm

Outer Radius 2.54 µm

Profile

● Single pass ○ Multi-pass

○ Speed/Slope

Speed 20 Slope 9

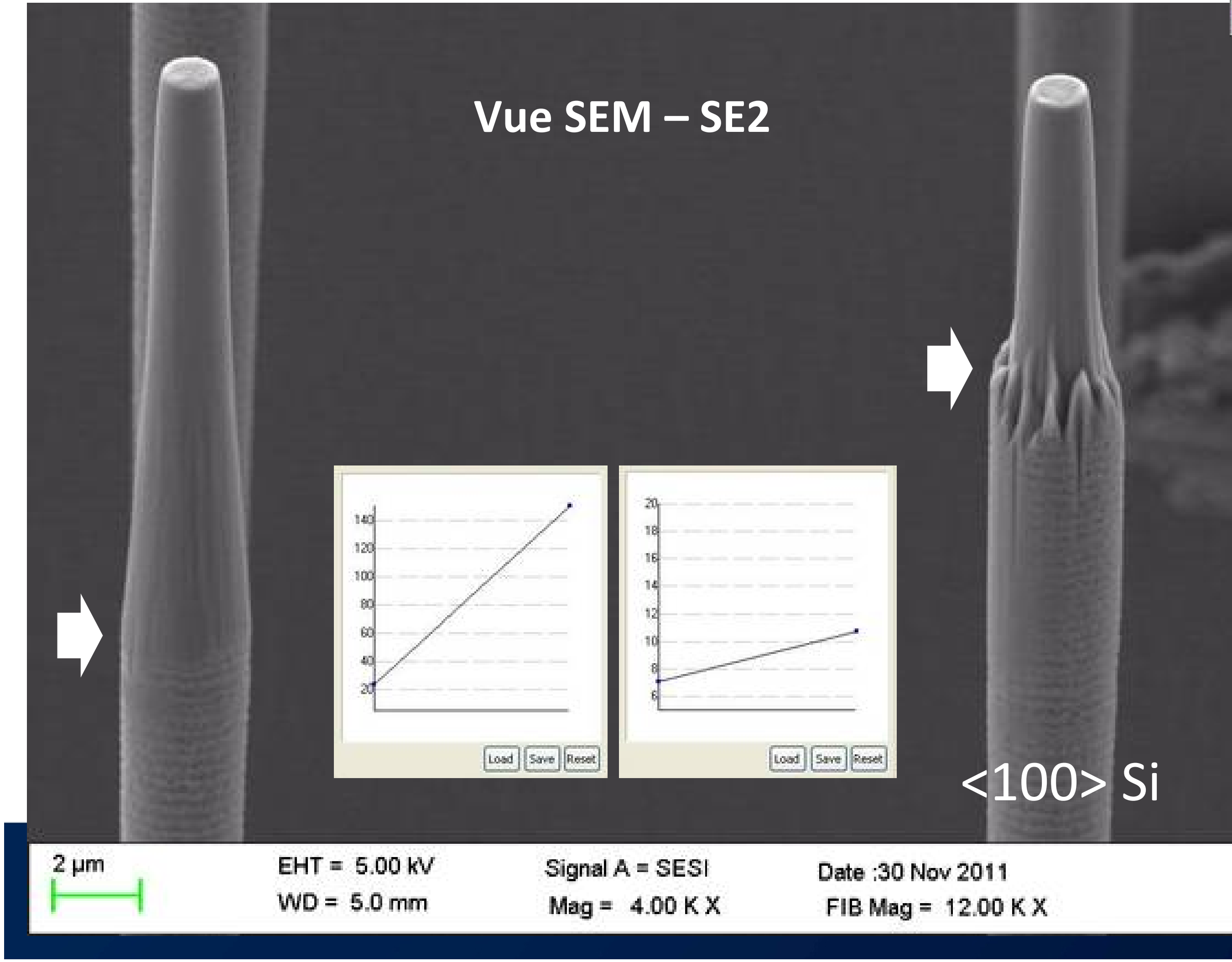
● Dwell (ms/pixel)

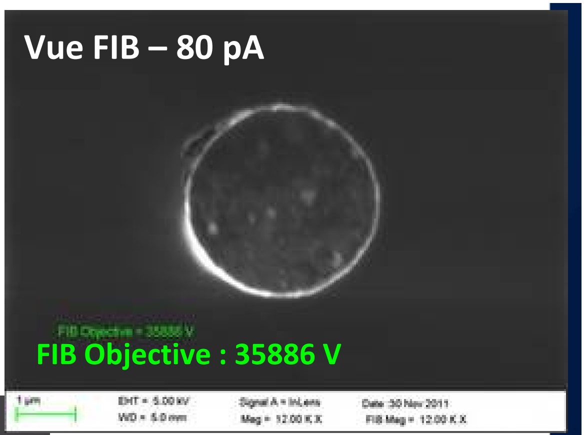
Min Dwell 5 Max Dwell 200

Annular Milling : FIB spot

→ Effet du dwell time

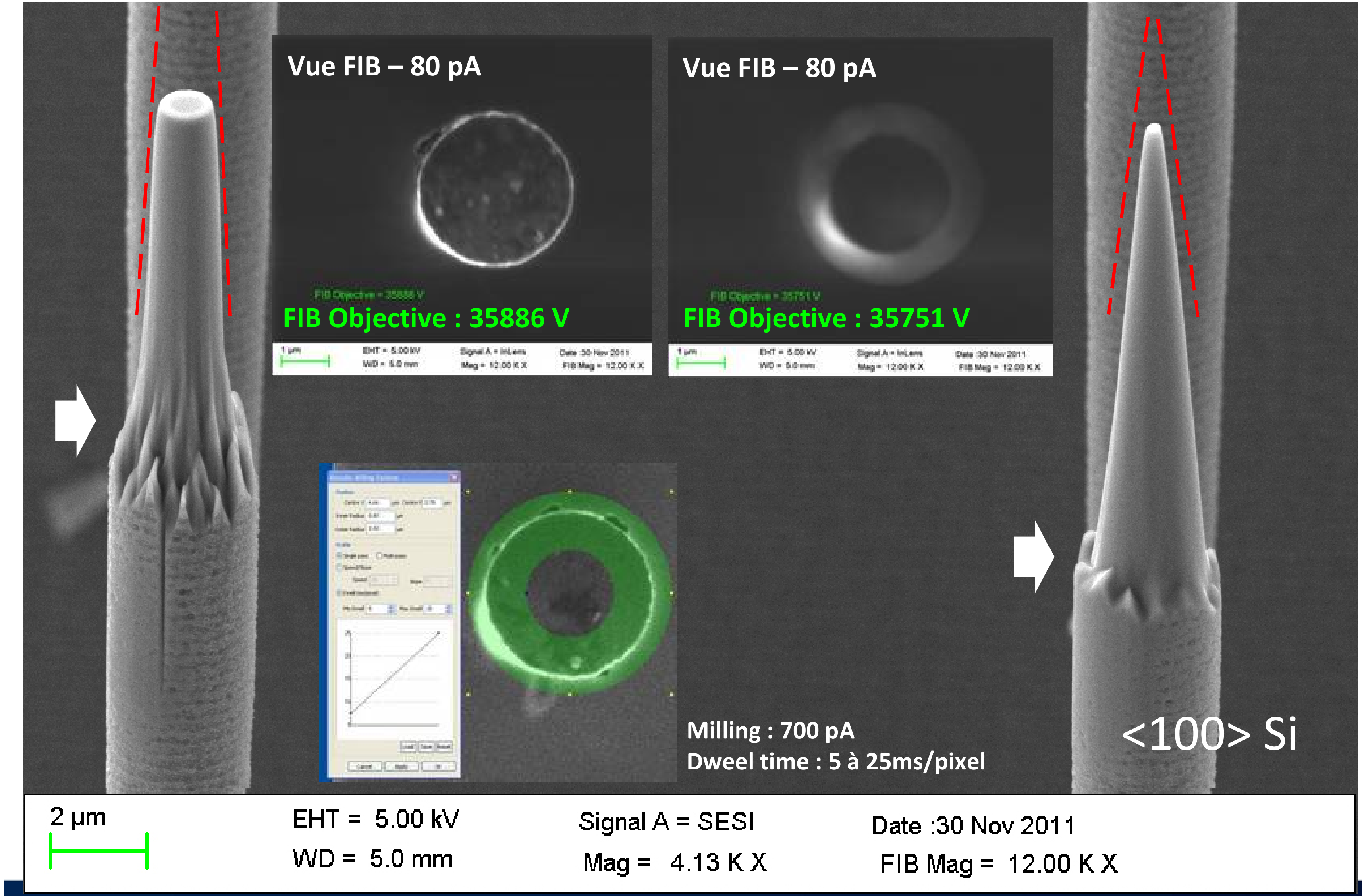
Milling : 700 pA





Annular Milling : FIB spot

→ Effet du FIB focus



Vue FIB – 80 pA

FIB Objective : 35886 V

Vue FIB – 80 pA

FIB Objective : 35751 V

Milling : 700 pA
Dweel time : 5 à 25ms/pixel

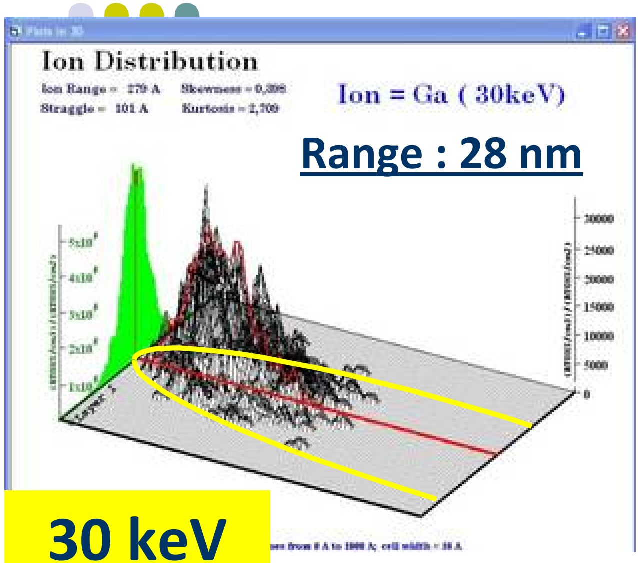
<100> Si

Low kV cleaning

Low kV cleaning

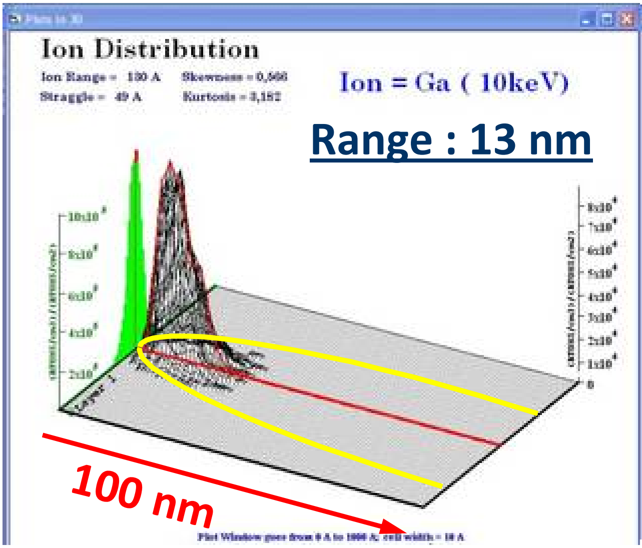
http://www.srim.org/

= reduce acceleration of Ga to reduce amorphous layer



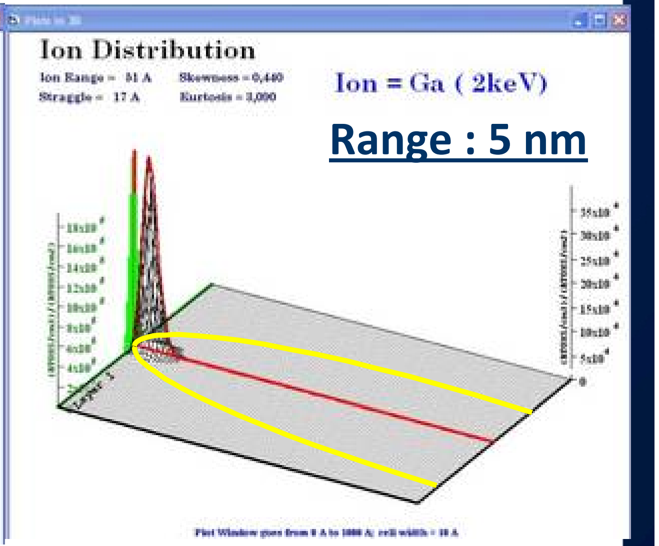
Range : 28 nm

30 keV



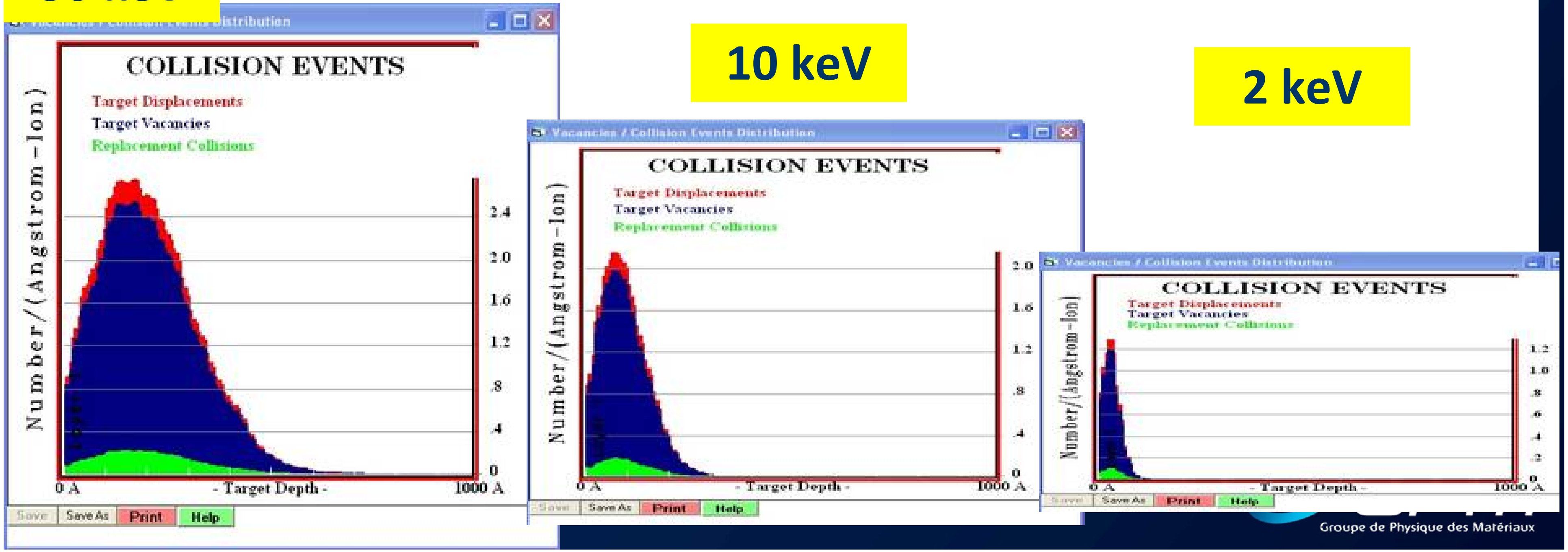
Range : 13 nm

10 keV



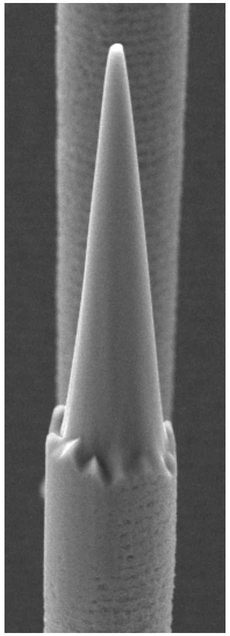
Range : 5 nm

2 keV



Low kV cleaning

Atom Probe



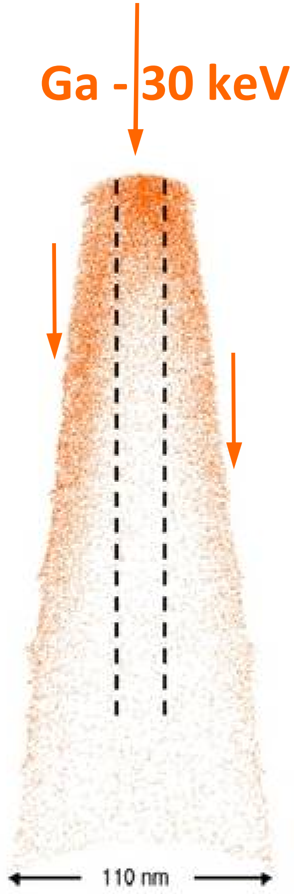
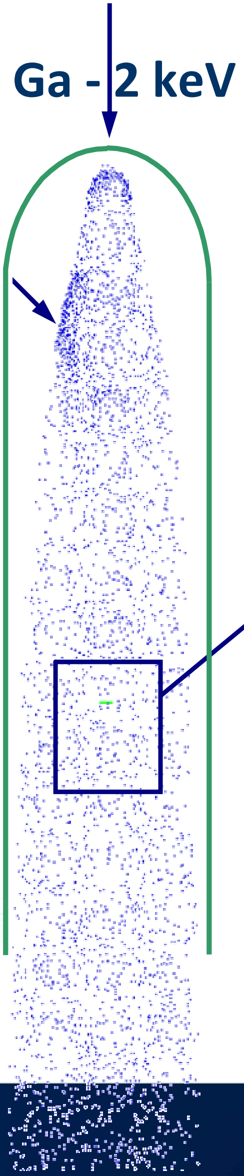


Fig. 11. Atomic-map of a Si specimen sharpened in a FIB with 30 keV Ga ions. For clarity, only the Ga atoms are shown. The dashed lines represent the field of view attainable with a 40 mm detector.



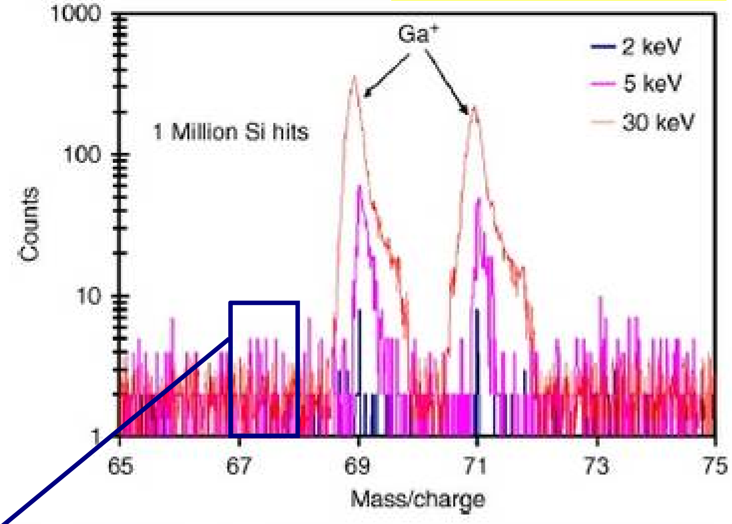
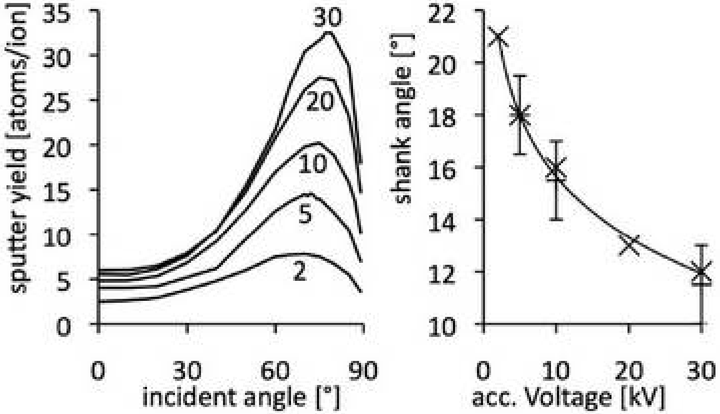
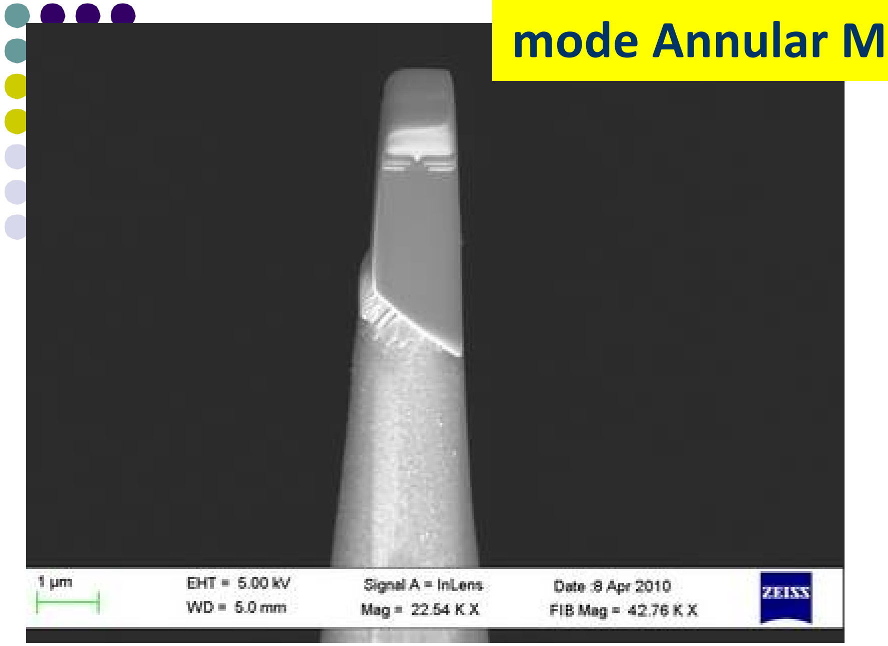


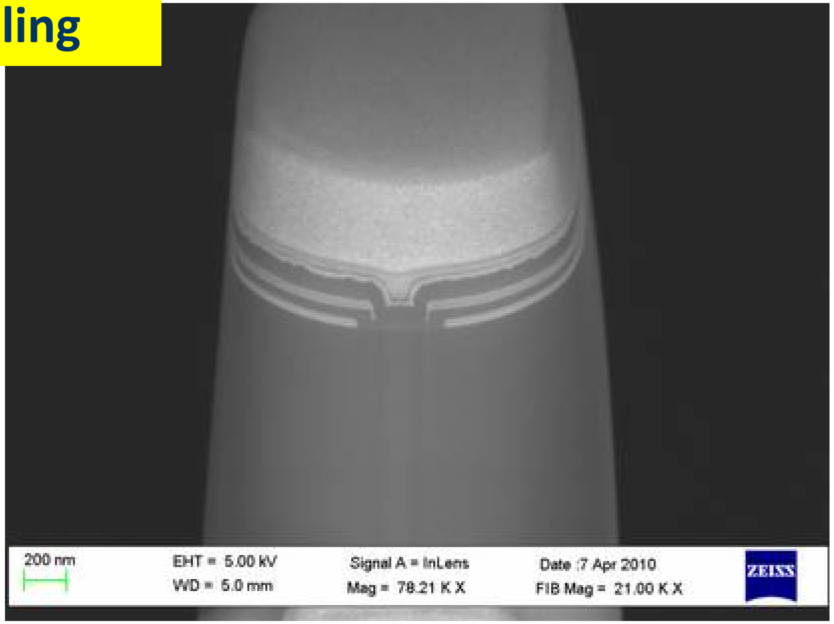
Fig. 13. Mass spectrum in the Ga region for the 2, 5 and 30 keV accelerating energies. One million Si atoms were sampled for each spectrum.

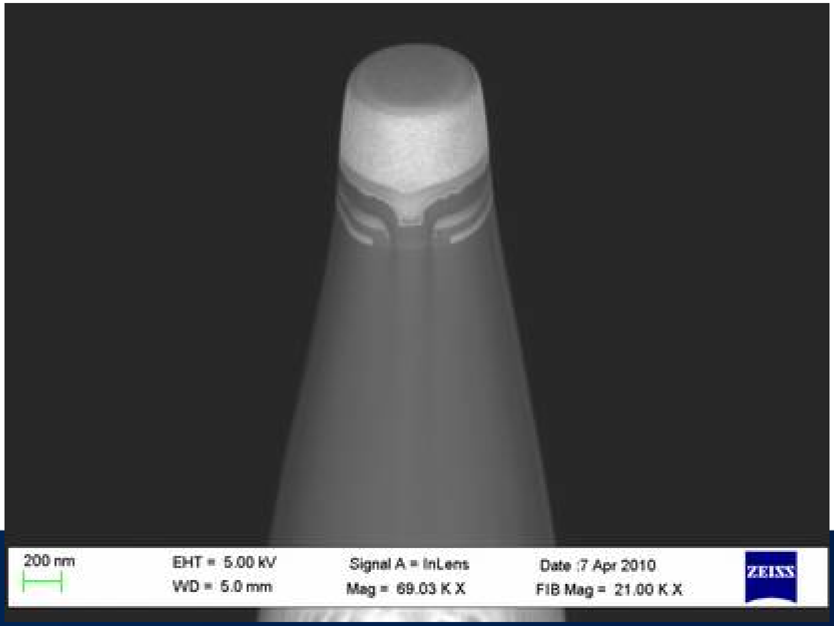


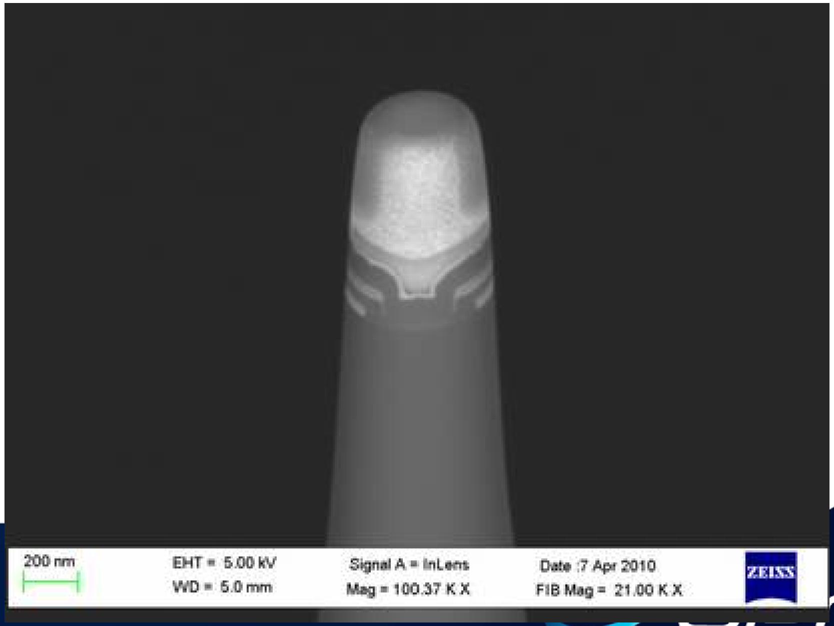
(a) Angular dependence of sputter yield for Ga in Fe for various accelerating voltages calculated by SRIM. (b) The angle of highest sputter yield from SRIM calculations (×) compared to the measured shank angle of AP samples (+).

mode Annular Milling



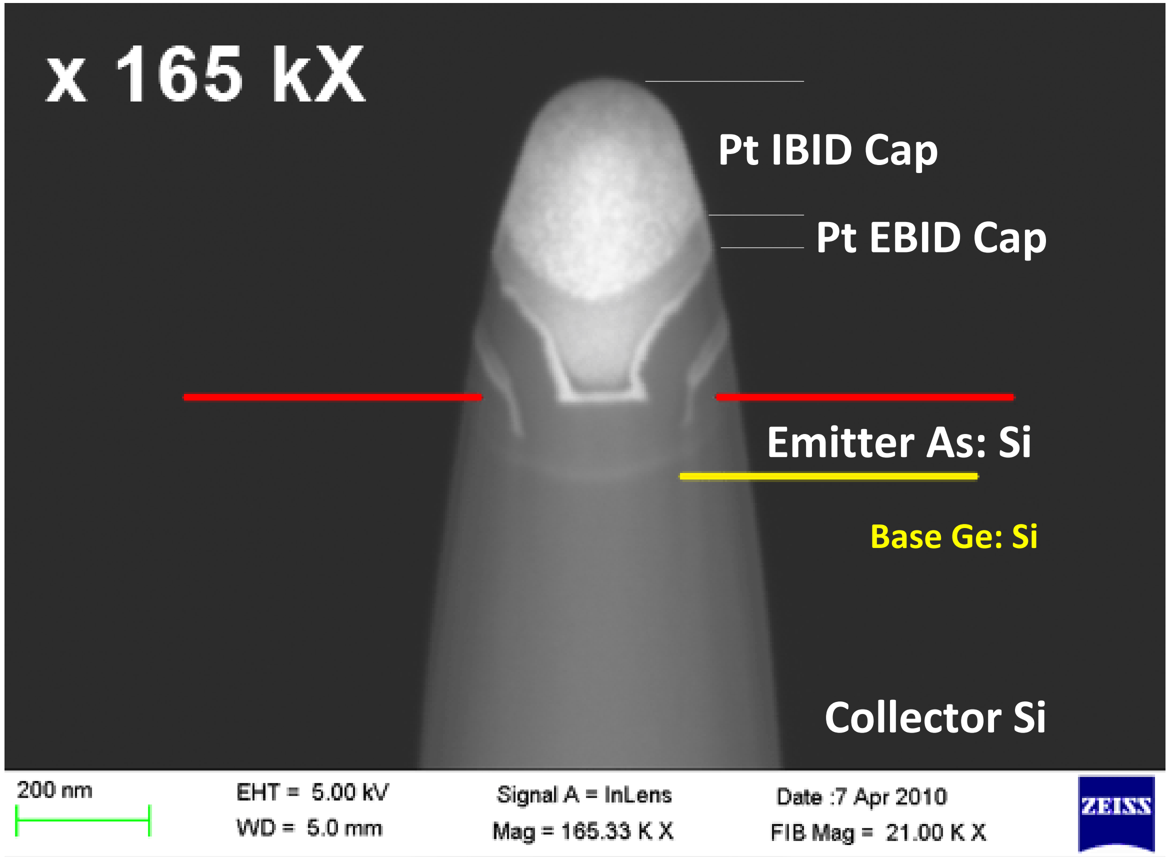




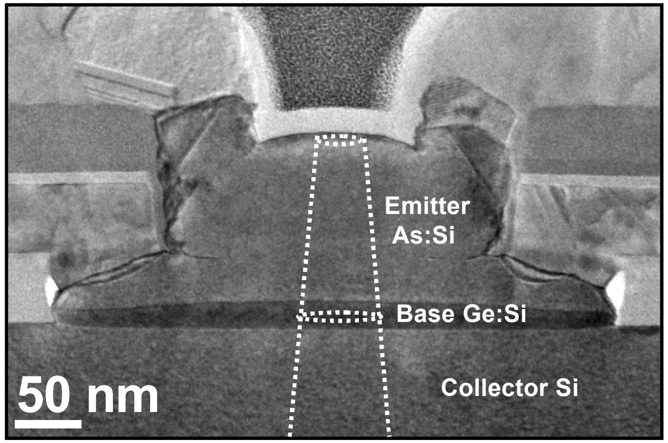


Low kV cleaning : mode SEM + FIB

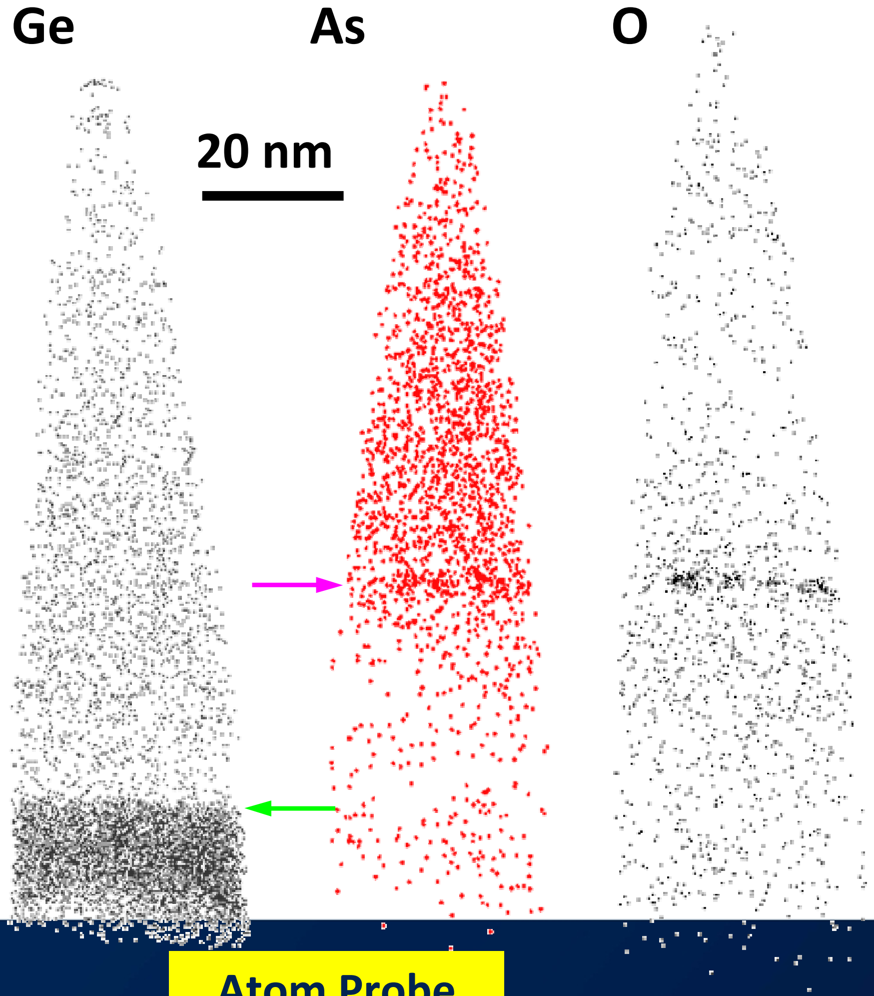
mode SEM + FIB



NPN bipolar transistors (BiCMOS)

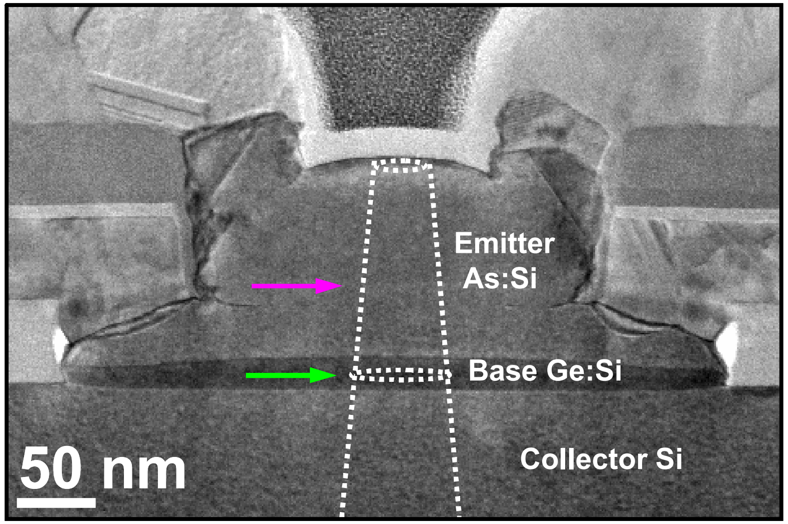


Distribution of Ge and As atoms from APT: presence of As clusters



Atom Probe

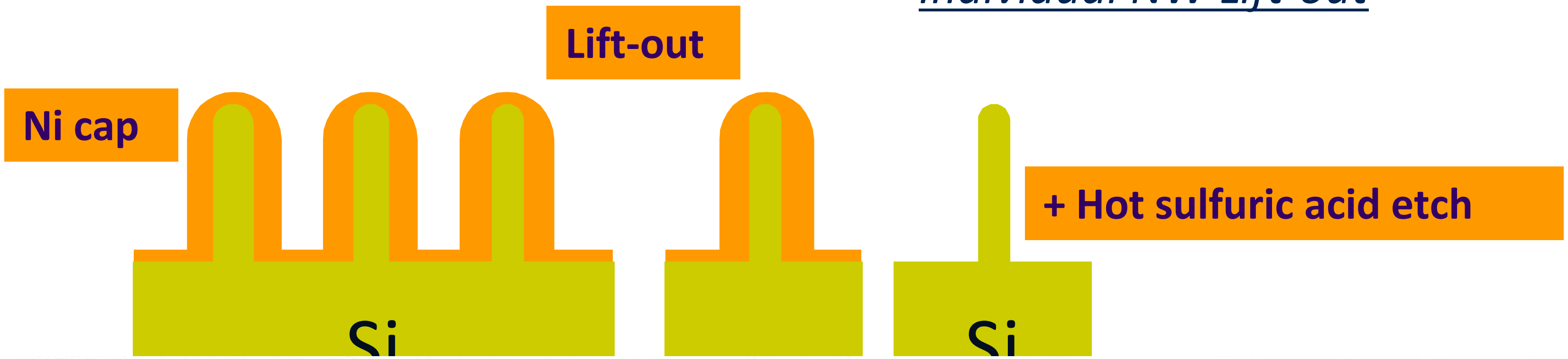
NPN bipolar transistors (BiCMOS)



Segregation of As on small oxygen clusters :
clear influence of a bad oxide removal between two epitaxies

Nanofils de Silicium

Individual NW Lift-out



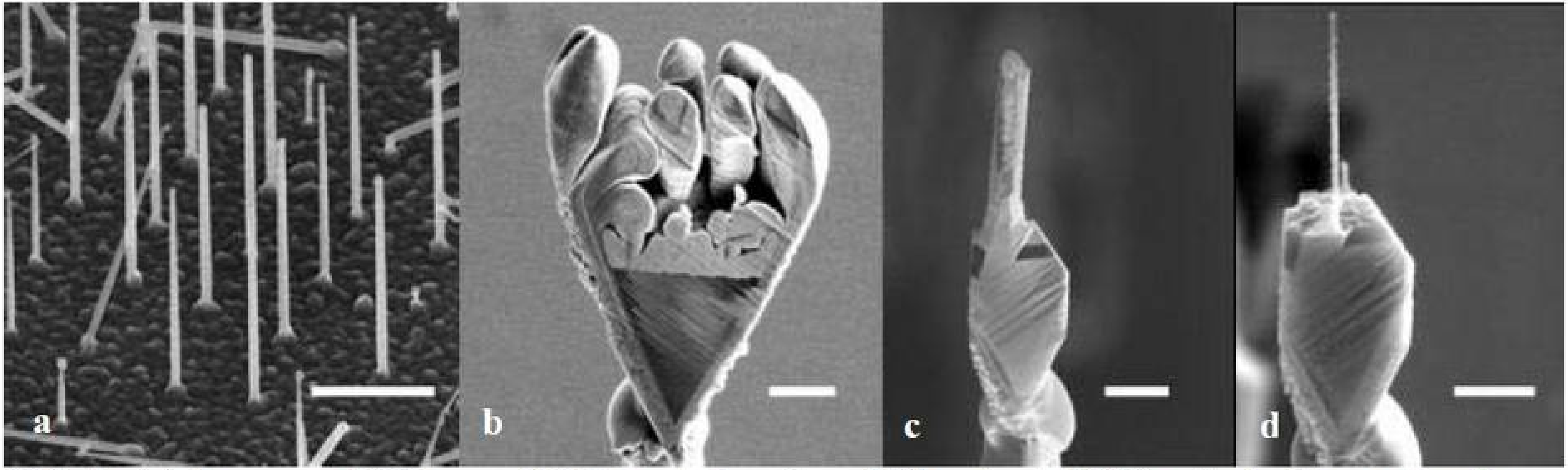
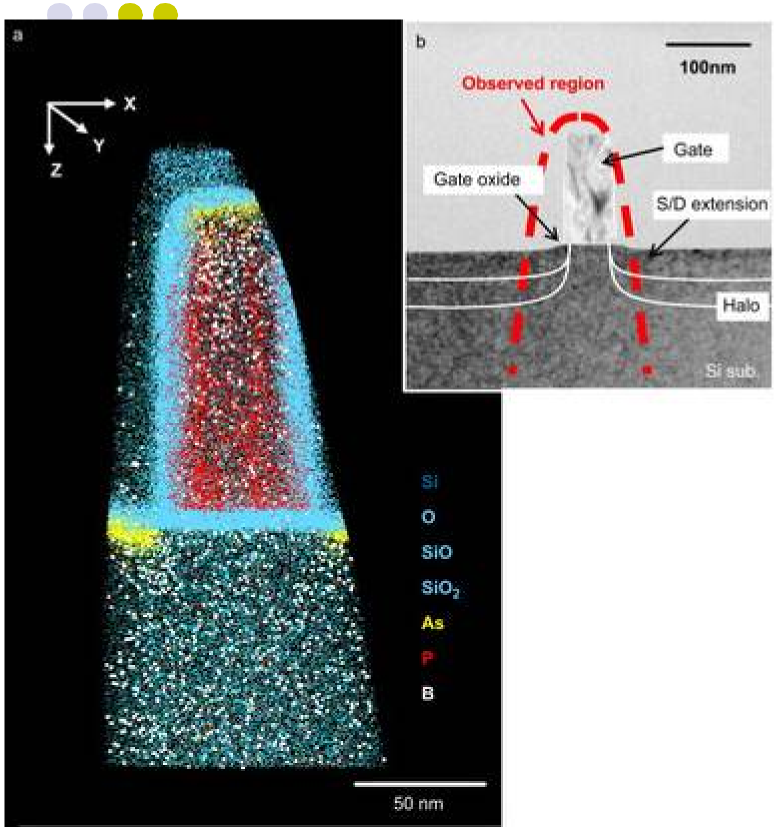
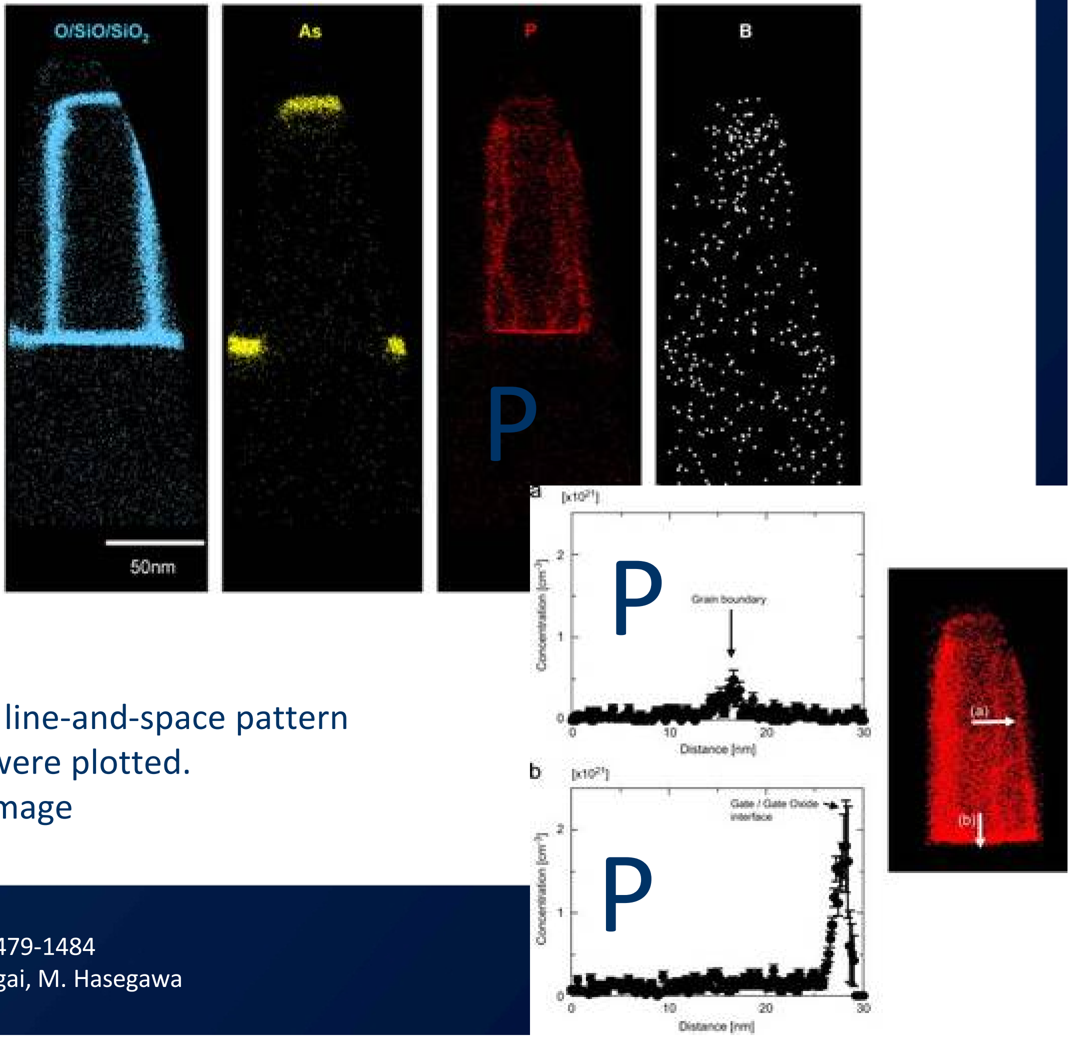


FIG. 1. (a) As-grown NW array. (b) Extracted wedge of Ni protected NWs attached to a microtip post. (c) Same wedge after FIB milling to isolate single NW for analysis. (d) NW after Ni removal via sulfuric acid etch. Note: All scale bars 2μ in length.

Dopant distributions in n-MOSFET structure



Elemental maps of O/*SiO*/*SiO*2, As, P, and B in a slice 10 nm thick, parallel to the *X*–*Z* plane.

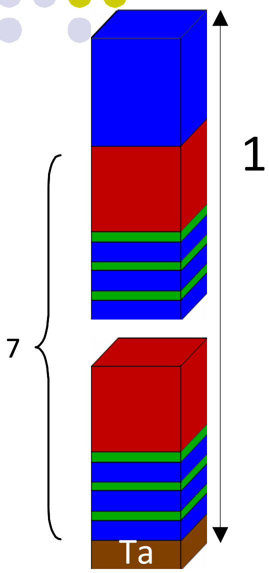


(a) The 3D elemental map of an n-type MOSFET from a line-and-space pattern sample. For visual clarity, only 0.1% of Si atoms were plotted.

(b) Cross-sectional transmission electron microscopic image

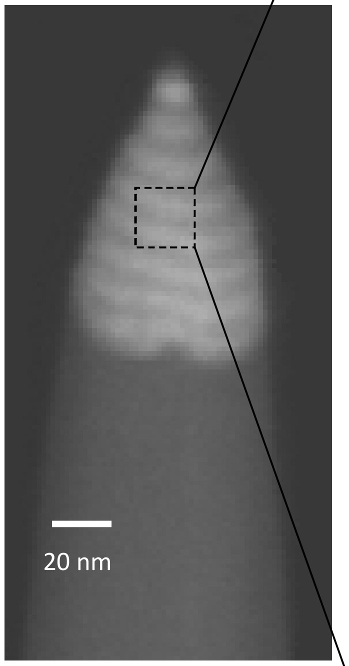
Ultramicroscopy, Volume 109, Issue 12, November 2009, Pages 1479-1484
K. Inoue, F. Yano, A. Nishida, H. Takamizawa, T. Tsunomura, Y. Nagai, M. Hasegawa

Multicouches magnétiques (Pt/Co)$_3$/Pt$_x$/IrMn

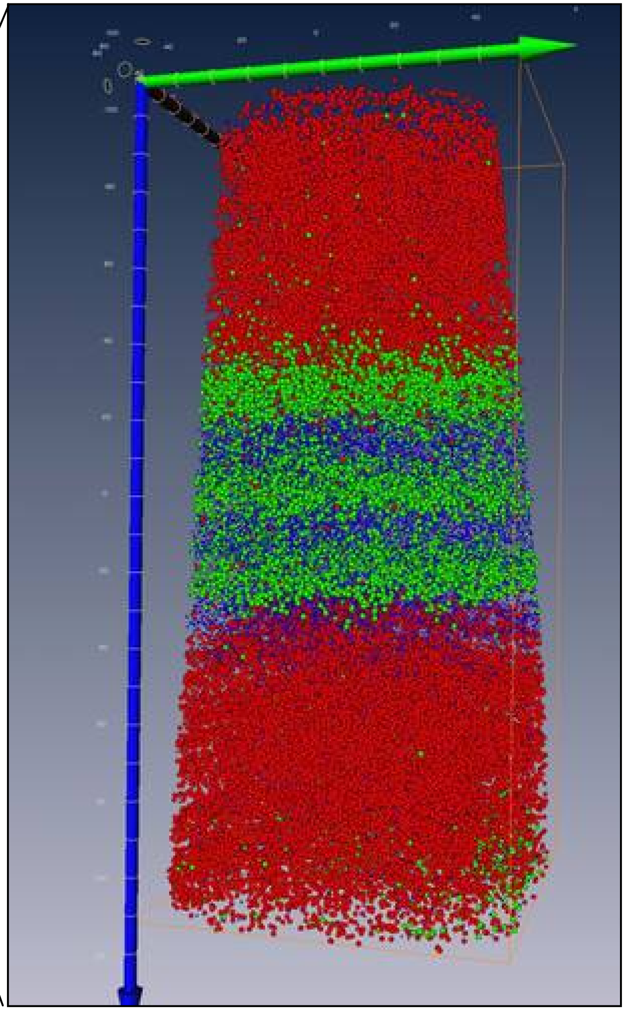


Deposited layers

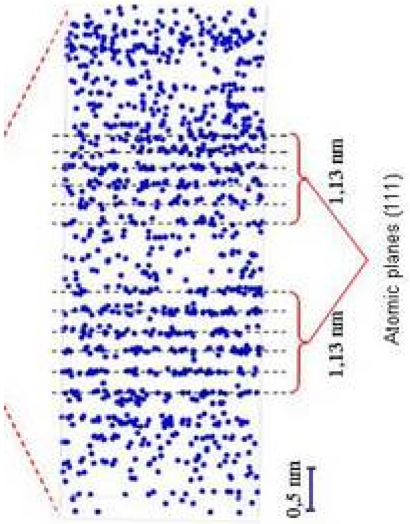
🟩 Co
🟦 Pt
🟥 IrMn



SEM image

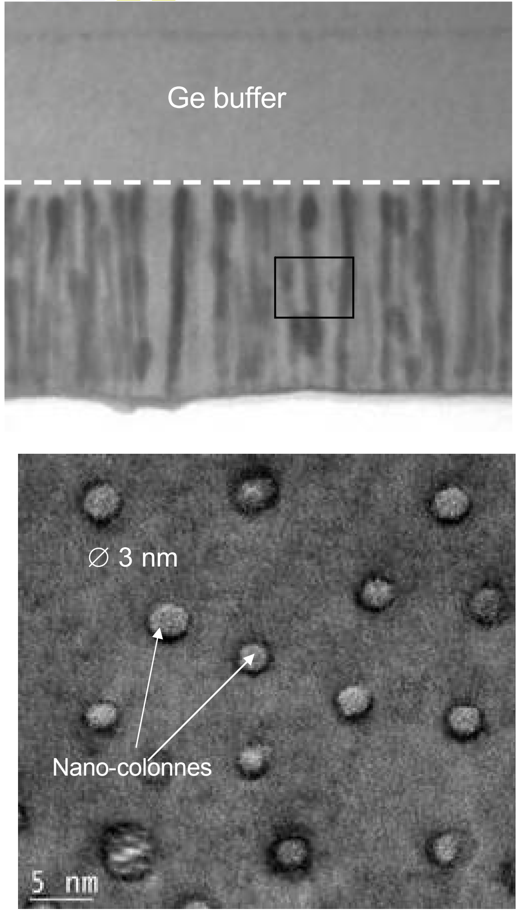


Co
Pt
IrMn

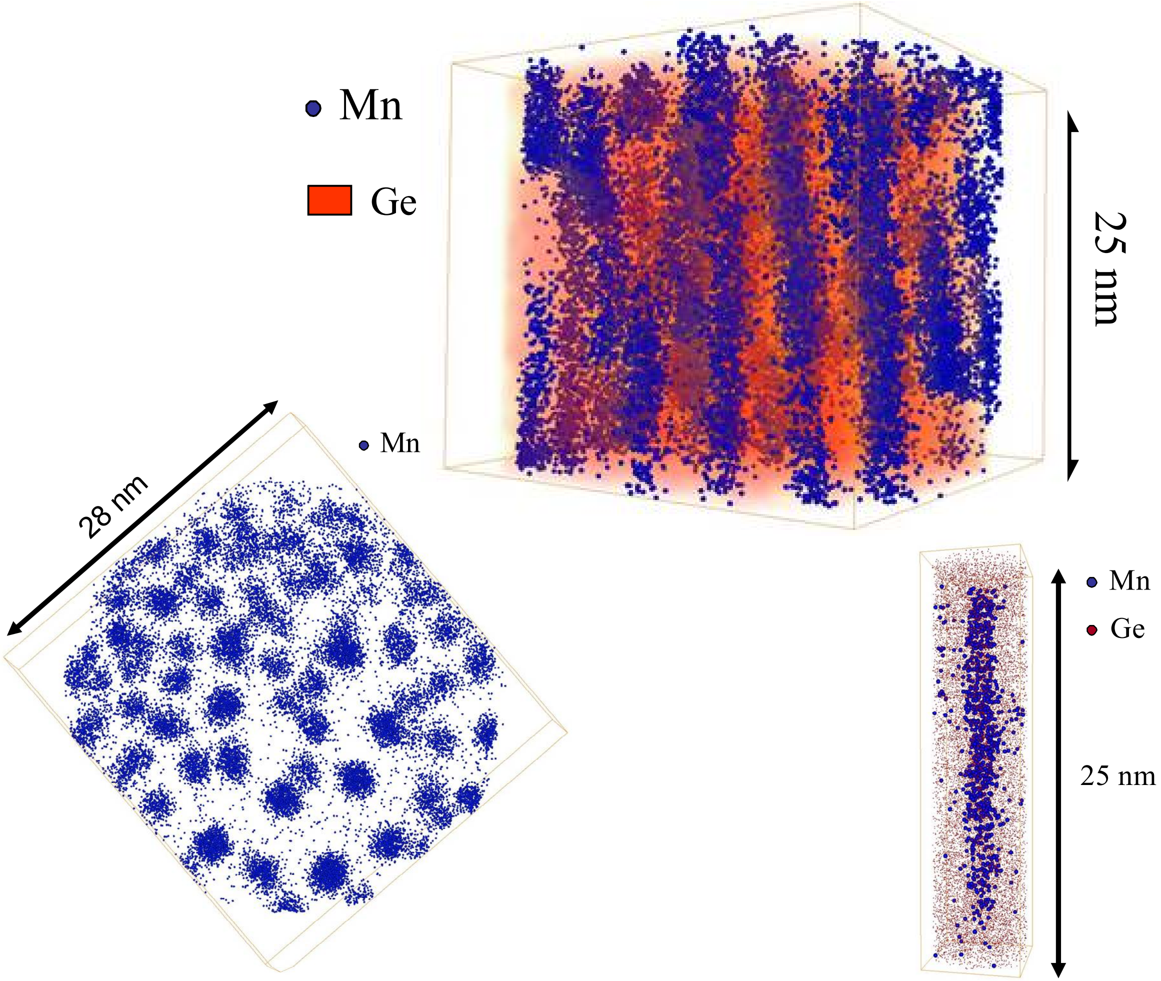


A. Zarefy, R. Lardé, L. Lechevallier, F. Cuvilly, J. M. Le Breton, V. Baltz,B. Rodmacq and B. Dieny .
JOURNAL OF APPLIED PHYSICS **105**, 103912 2009

Self organized Mn rich nanocolumns in Ge



TEM images (INAC)



Collaboration with CEA-INAC/Spintec and Néel institute (Grenoble)

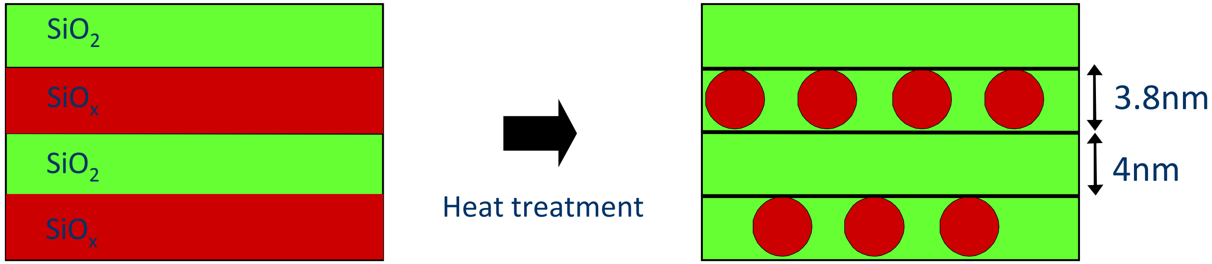
Si nanoparticles growth in SiO_2

many applications : Solar cells, optical amplifier , wave guide…

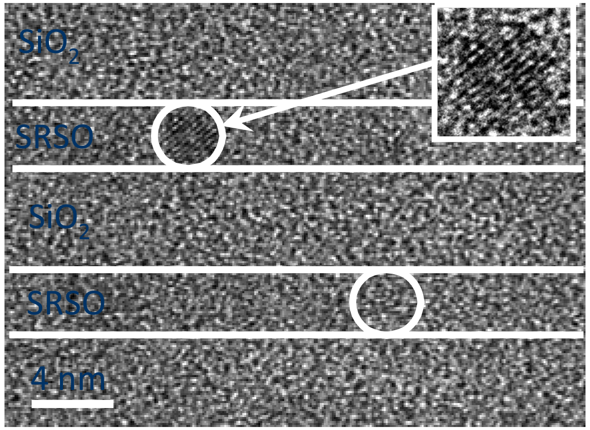
Elaboration by phase separation from Si riche SiO_x :

$$SiO_x \rightarrow \frac{x}{2} SiO_2 + (1 - \frac{x}{2}) Si$$

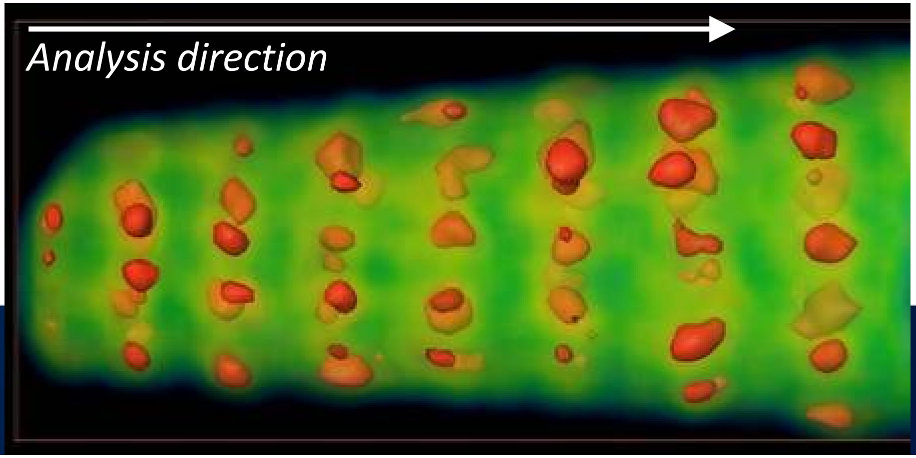
Multilayered structure ➔ control of the size distribution

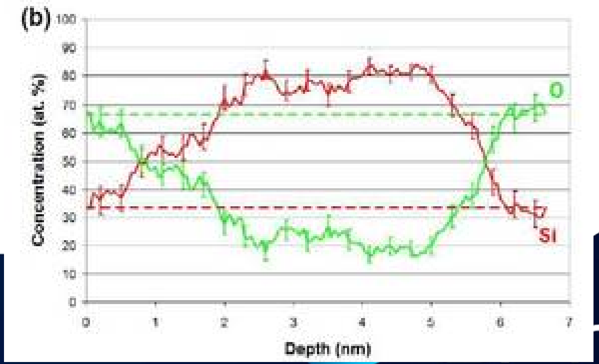


Si excess in SiO_x ~25%



25x25x70 nm³



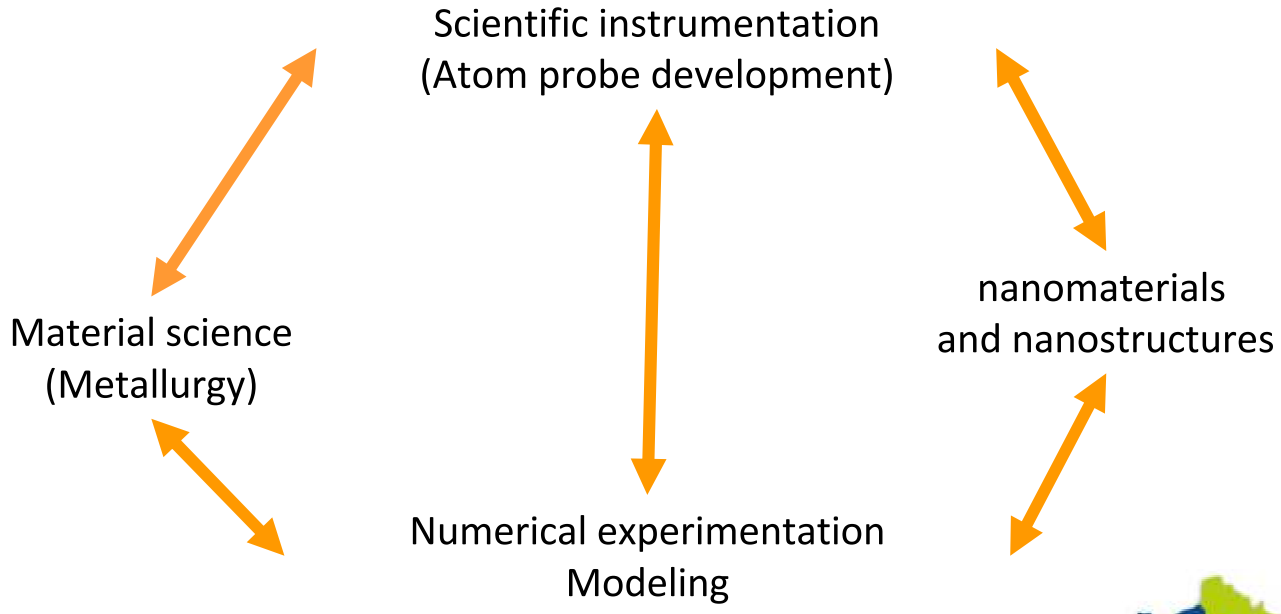


Groupe de Physique des Matériaux

Groupe de Physique des Matériaux

Director : Pr. P. Pareige

GPM UMR 6634 CNRS, Université de Rouen, INSA de Rouen
52 enseignants chercheurs titulaires, 13 ingénieurs, 40 doctorants, 7 post-doctorants, 4 techniciens, 5 administratifs



Scientific instrumentation
(Atom probe development)

Material science
(Metallurgy)

nanomaterials
and nanostructures

Numerical experimentation
Modeling

Fundamental research on technological interest materials

Merci de votre attention