GNMEBA – Journées pédagogiques 2023
Le MEB-FIB : état de l'art, principe et applications

Principes physiques mis en jeu au FIB, influence des conditions opératoires

Emmanuel CADEL

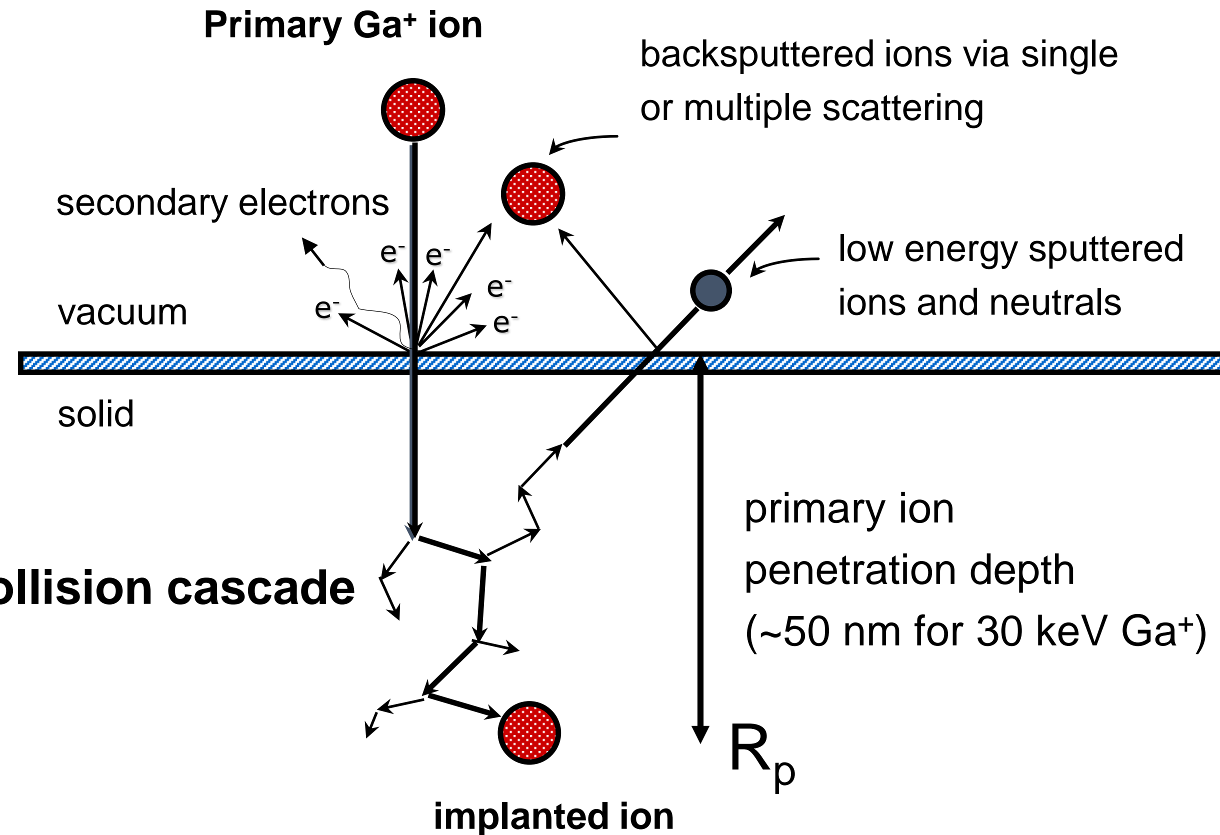
Groupe de Physique des Matériaux
UMR CNRS 6634

Sommaire

- Bref introduction du principe physique de l'interaction ion-matière
- Conditions opératoires :
    - Réglage du faisceau
    - Alignement du faisceau
    - Influence de l'angle d'incidence
    - Influence de la nature de la cible
    - Influence de la tension d'accélération
- Dommages et tentative de minimisation :
    - Baisser la tension d'accélération
    - Changer la nature de l'ion incident
    - Le mode Cryo FIB-SEM…

Gamme d'énergie utilisée pendant l'usinage FIB : 0.5 - 30 keV

- Éjection d'un atome de surface si l'énergie cinétique reçu > énergie de liaison
  (Two-body billiard ball collision model)
- Diffusion de l'ion incident dans la cible
  (Rutherford Scattering)
- Perte d'énergie de l'ion et implantation
- Production de phonons
- Emission d'électrons secondaires (SE).



Fundamentals of Ion-Surface Interaction, W. Möller, http://www.hzdr.de/FWI/GENERAL/Fundam_ISI.pdf

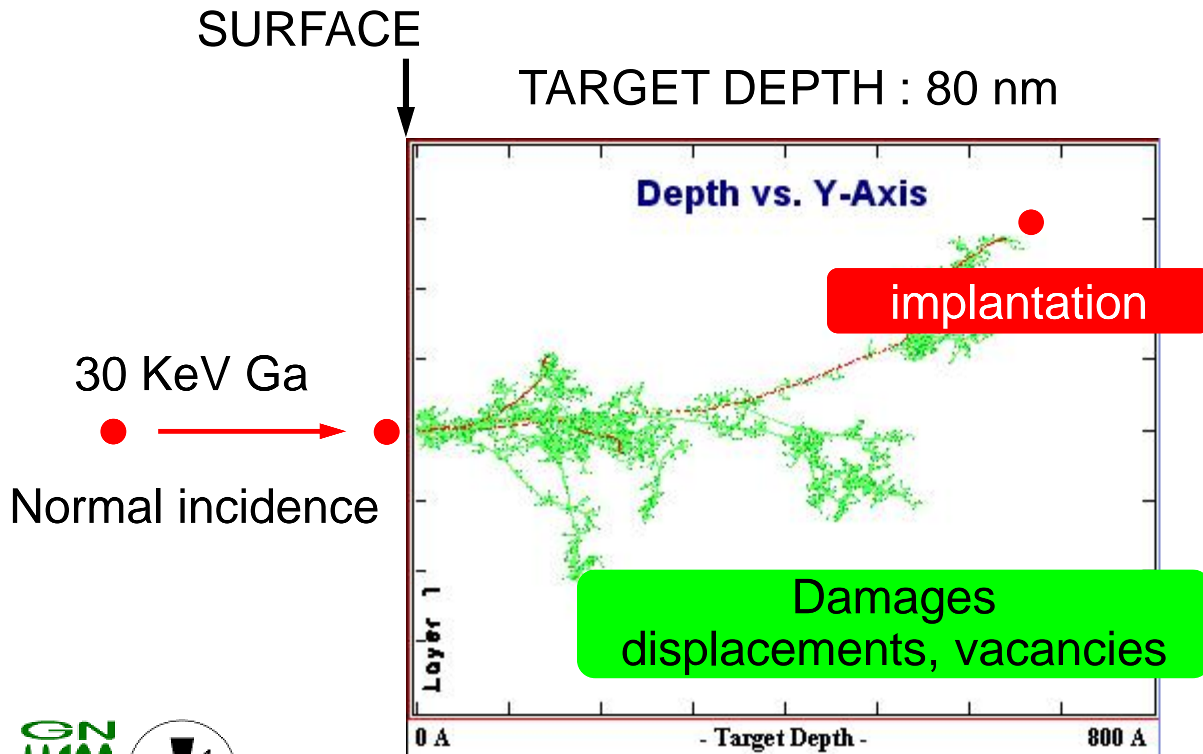
Simulation of collision volume : SRIM (TRIM)

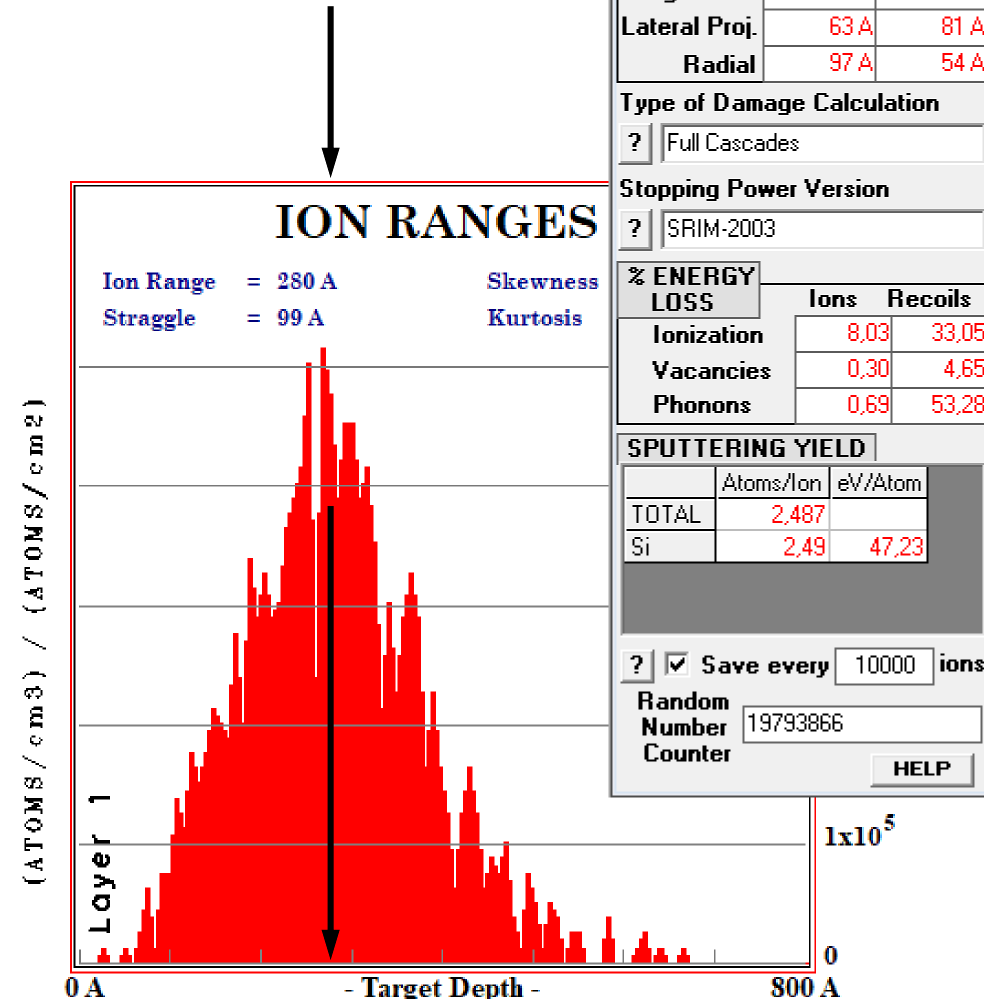
http://www.srim.org

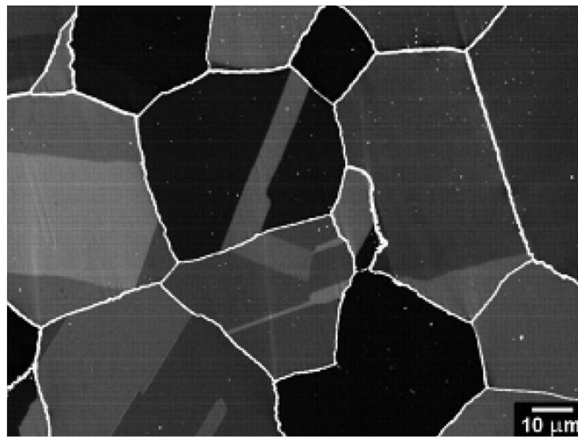
The target is treated as an amorphous material.

1000 Ga-30 KeV in amorphous Si

Projected Range R_p = 28 nm

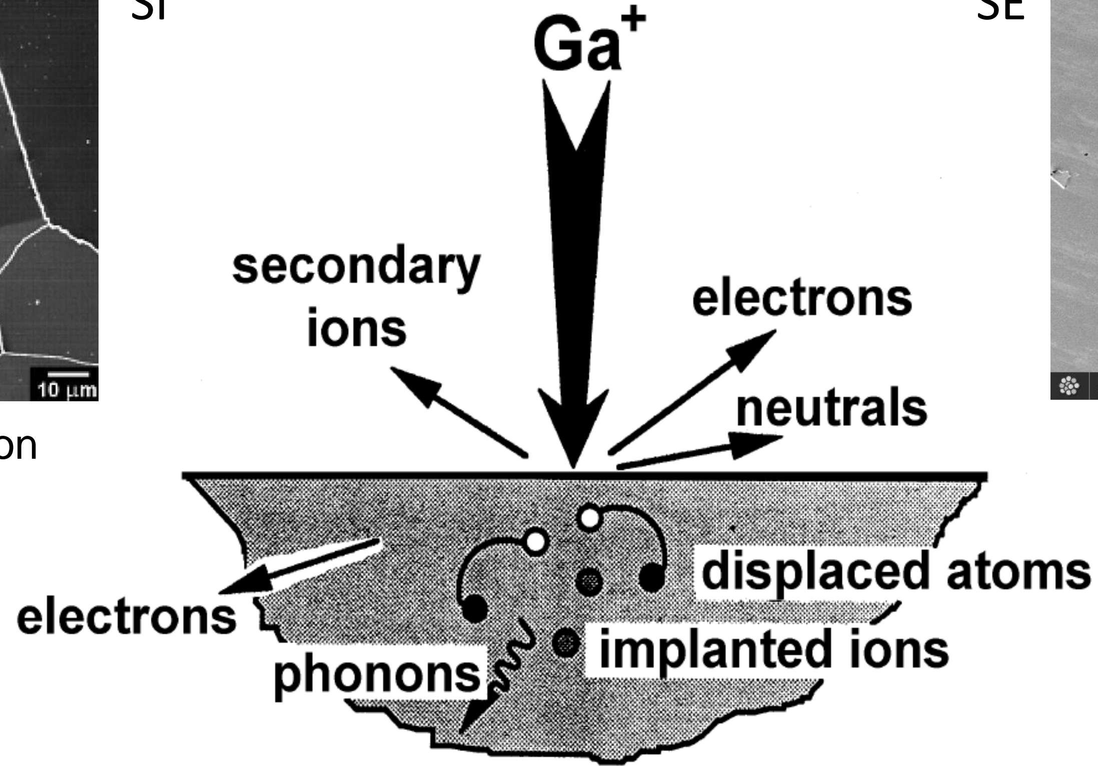




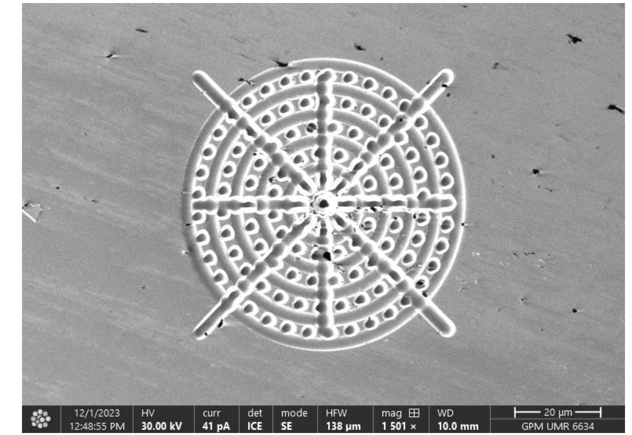


Contraste de canalisation

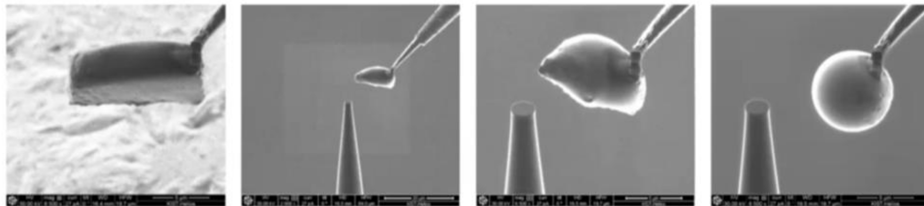
SI

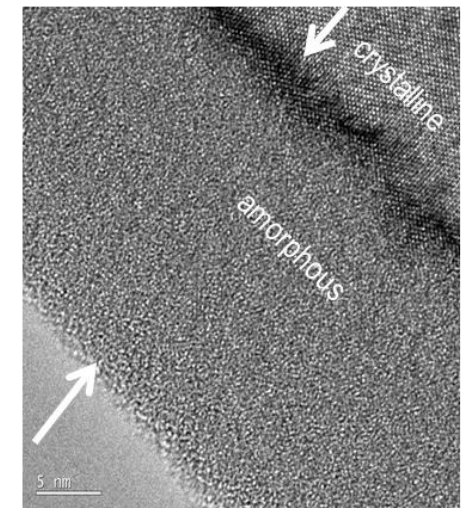


SE



InGa





Sommaire

- Bref introduction du principe physique de l'interaction ion-matière
- Conditions opératoires :
  - Réglage du faisceau
  - Alignement du faisceau
  - Influence de l'angle d'incidence
  - Influence de la nature de la cible
  - Influence de la tension d'accélération
- Dommages et tentative de minimisation :
  - Baisser la tension d'accélération
  - Changer la nature de l'ion incident
  - Le mode Cryo FIB-SEM…

Vérifier la qualité des spots FIB

80 pA | 148 pA | 293 pA | 681 pA | 1.51 nA

0.25 | 0.39 | 0.52 | 0.63 | 1.19 µm

2.96 nA | 6.26 nA | 11.67 nA | 25.88 nA

1.09 | 1.64 | 2.18 | 4.45 µm

43.3 nA

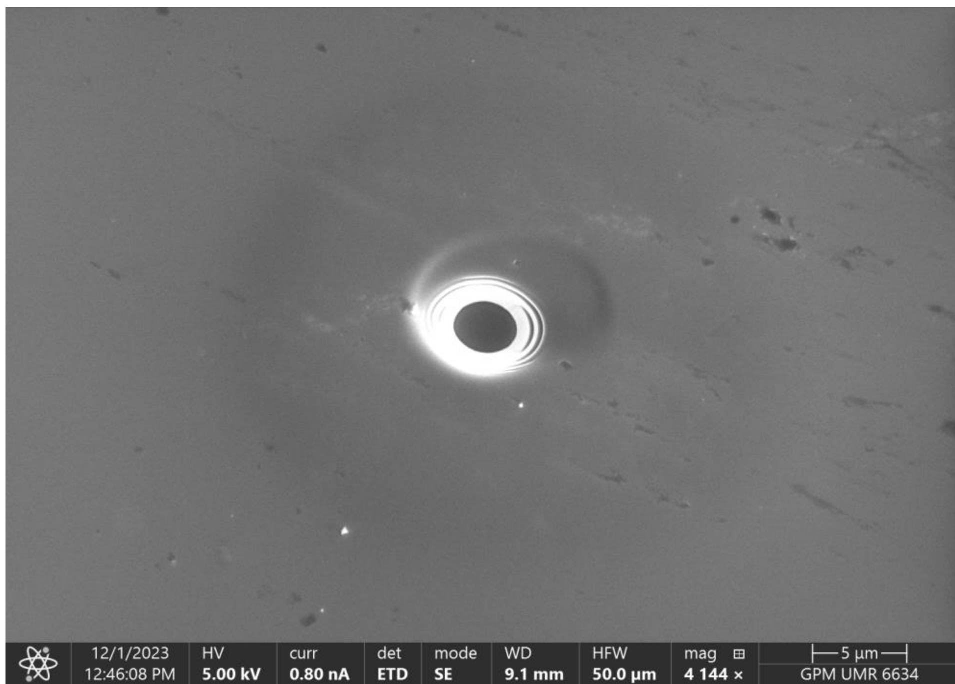
7.18 µm

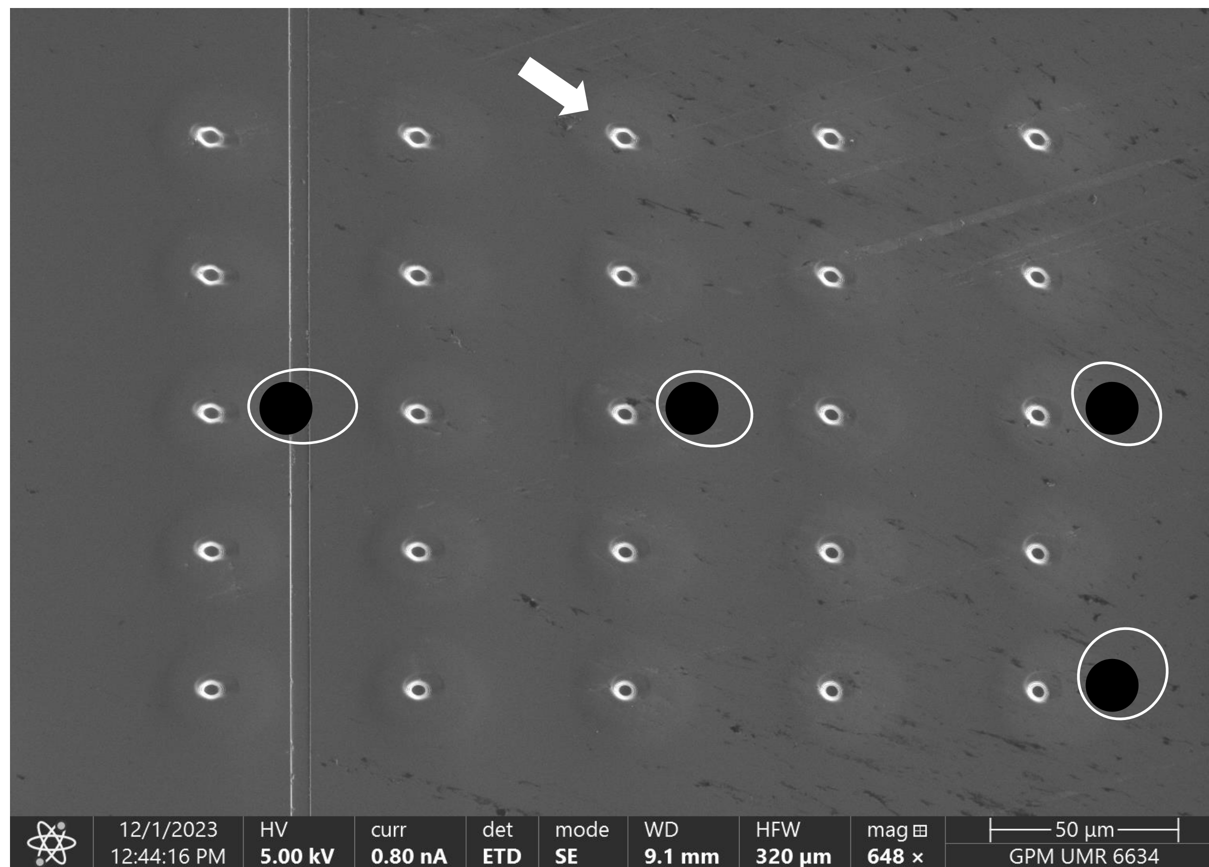
V 2 = 6.999 µm

NVision 40
s:n 38-51
2 µm
Mag = 3.00 K X
EHT = 5.00 kV
WD = 5.6 mm
FIB Lock Mags = No
FIB Probe = 30KV:80 pA
Signal A = SESI
ESB Grid = 306 V
System Vacuum = 2.96e-006 mbar
Gun Vacuum = 1.53e-009 mbar
Date :27 Jun 2022
Time :14:20:42
ZEISS

Alignement des diaphragmes / courant

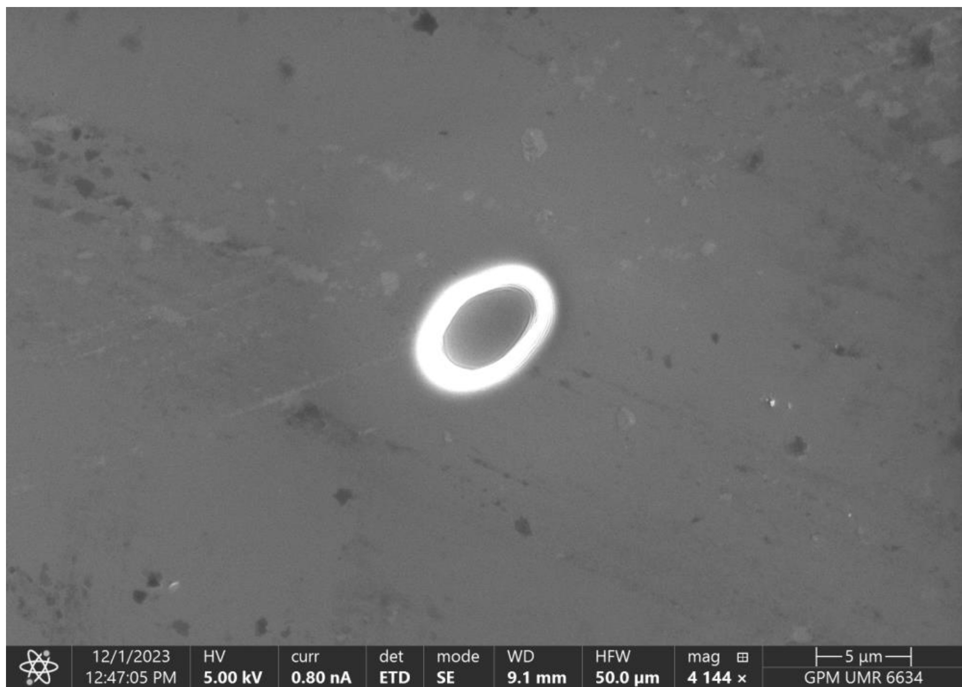
Spot burns de quelques secondes

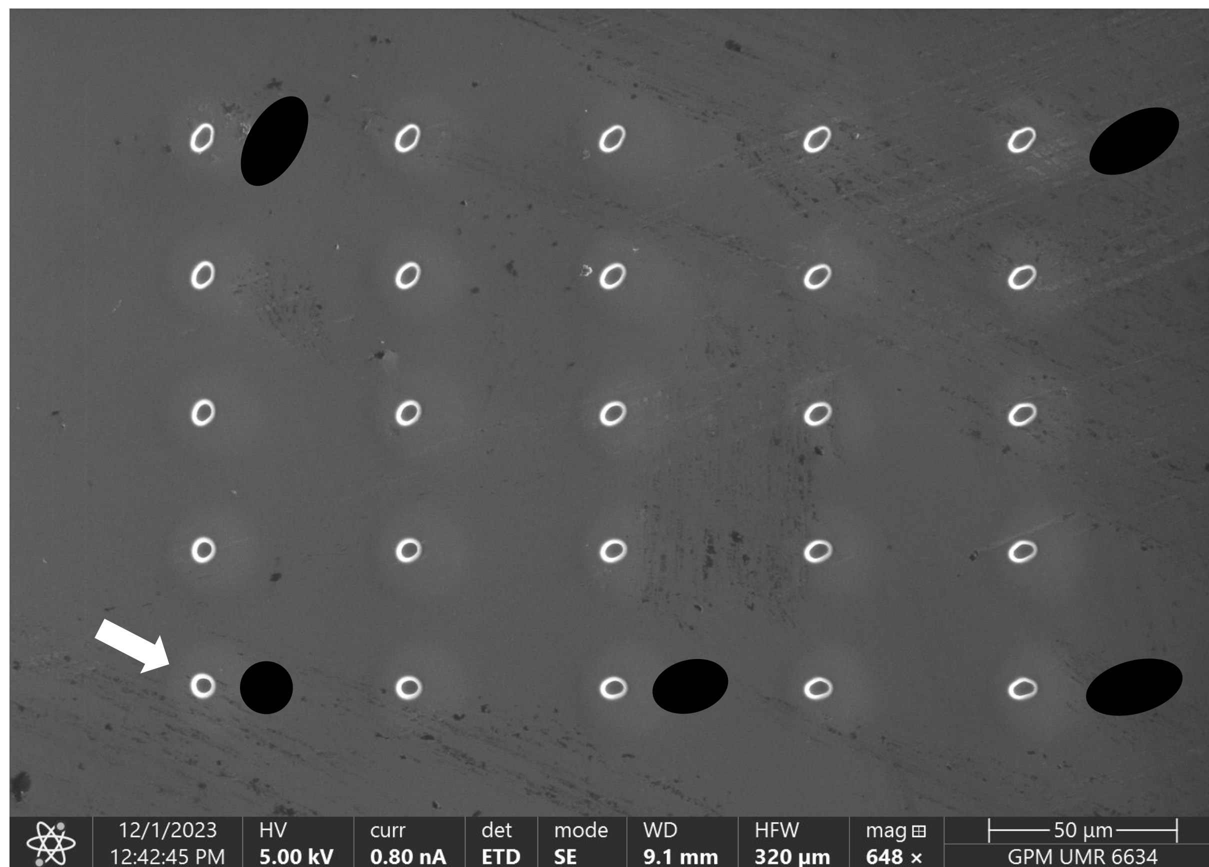




Correction de l'astigmatisme

Spot burns de quelques secondes

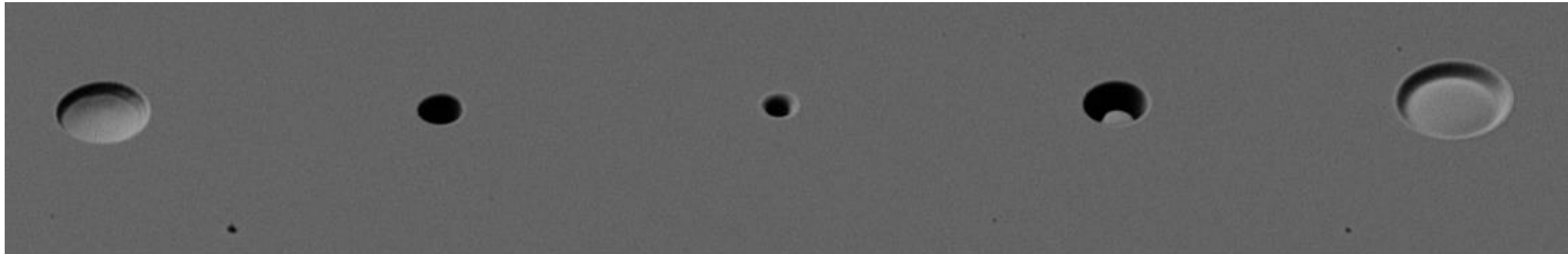




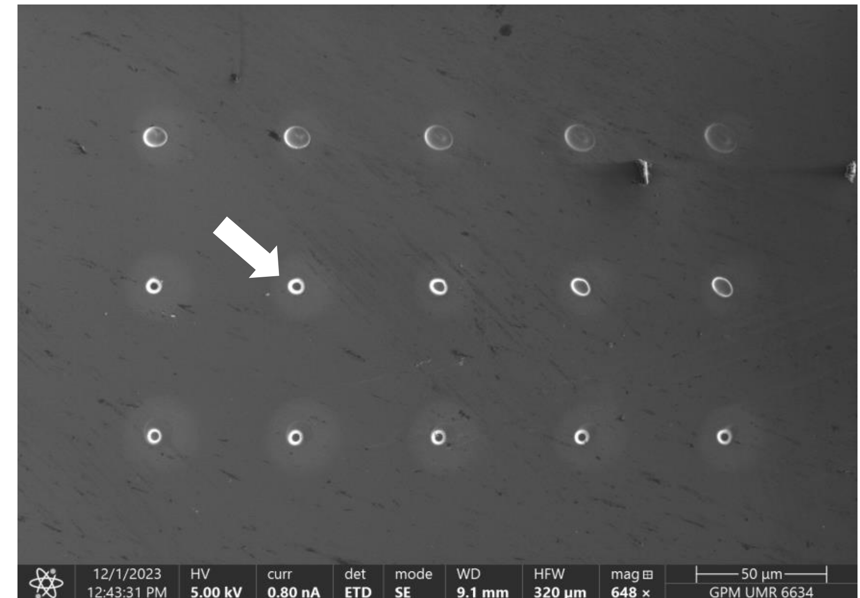
Optimisation du faisceau FIB

Réglage du defocus pour les forts courants

Divergent beam **Focus** **Convergent beam**

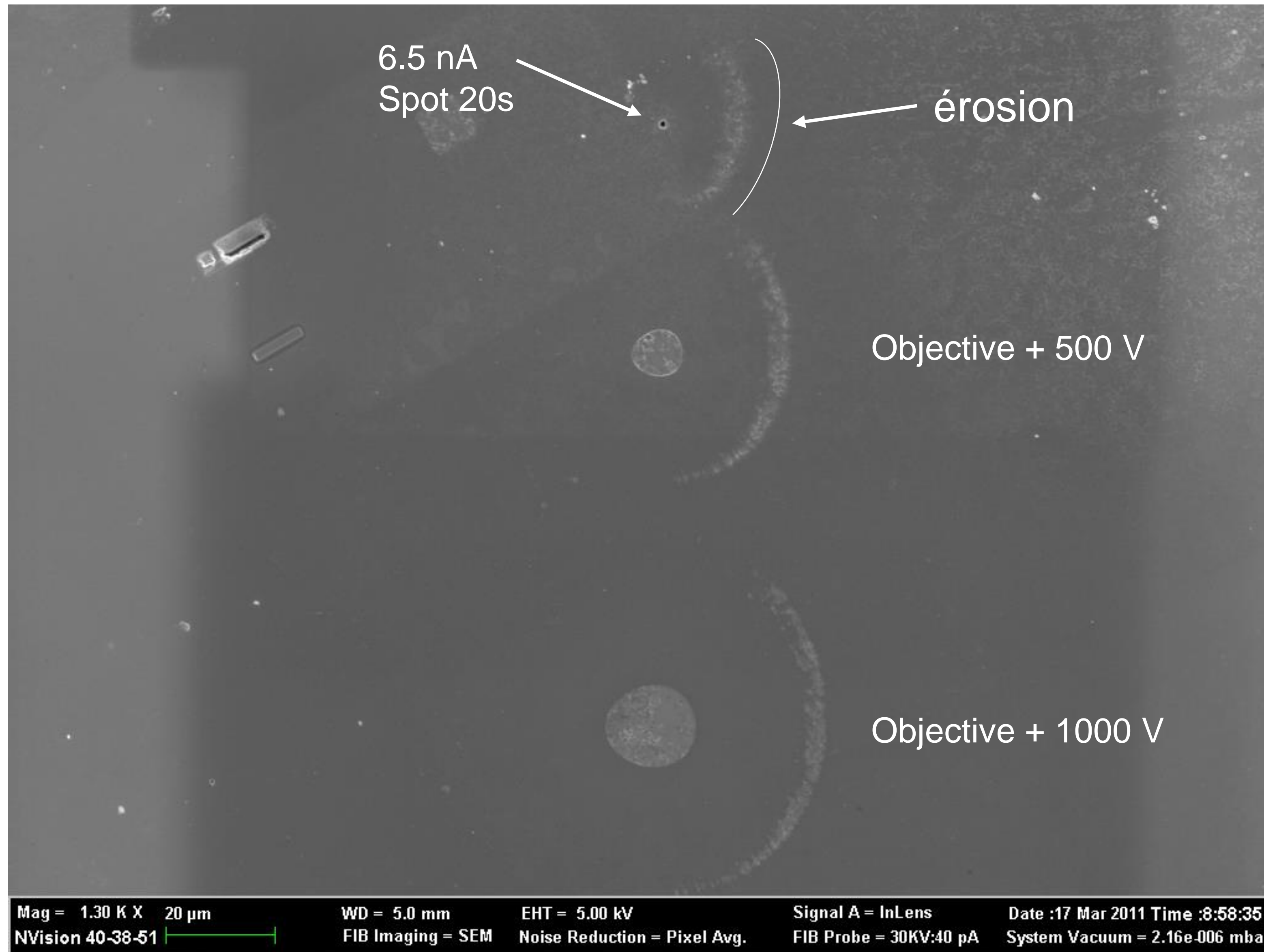


Probe I = 30kv : 1.5nA / DwellTime = 10 s
Nominal Fib objective voltage = 35400 V
Delta U_{obj} = 150 V



Spot burns de quelques secondes

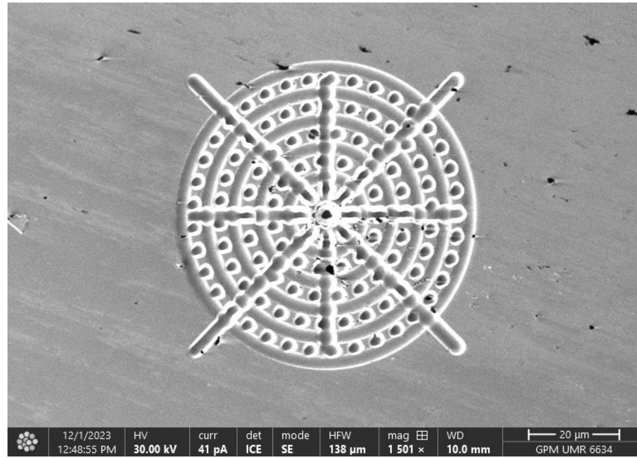
Point de vigilance sur le diaphragme

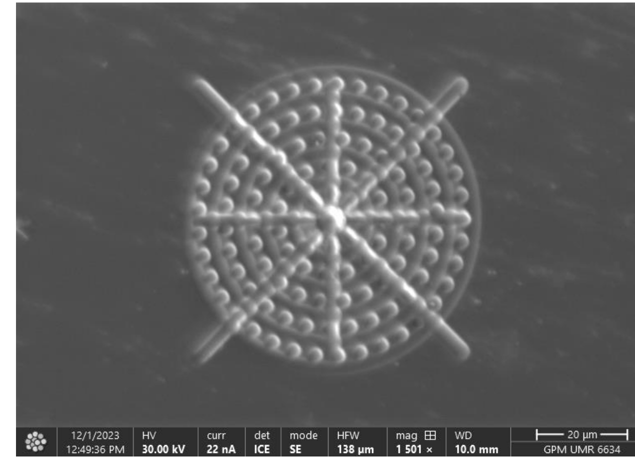


Alignement des courants de faisceau FIB

En courant de faisceau à une valeur de tension donnée…

30 kV – 41 pA

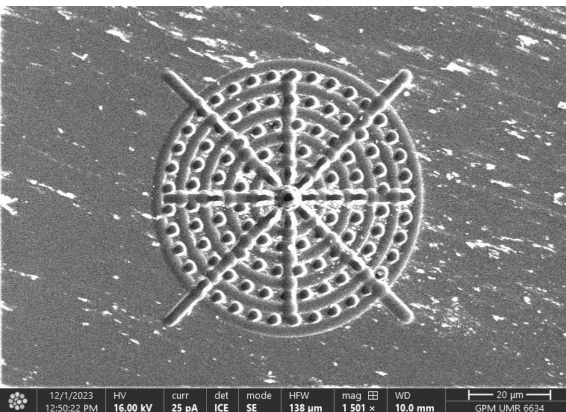




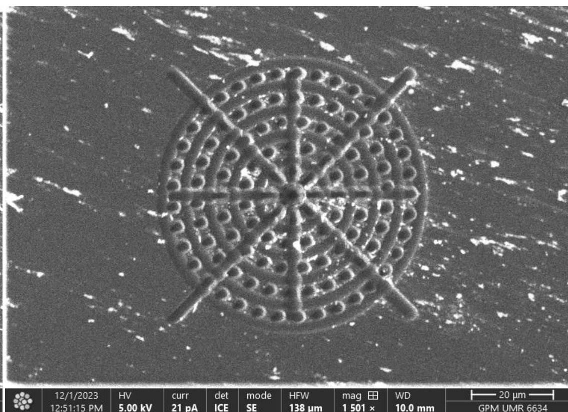
30 kV – 22nA

…et en tension d'accélération

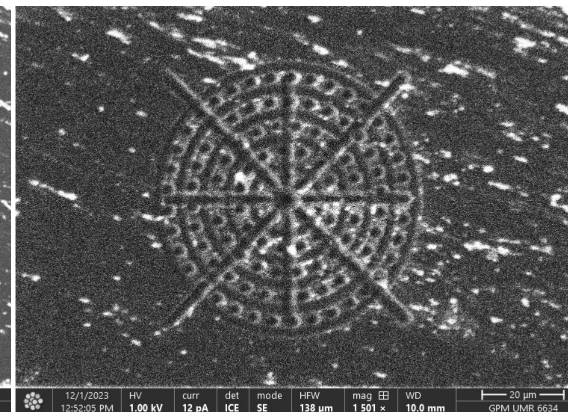
16 kV – 25 pA



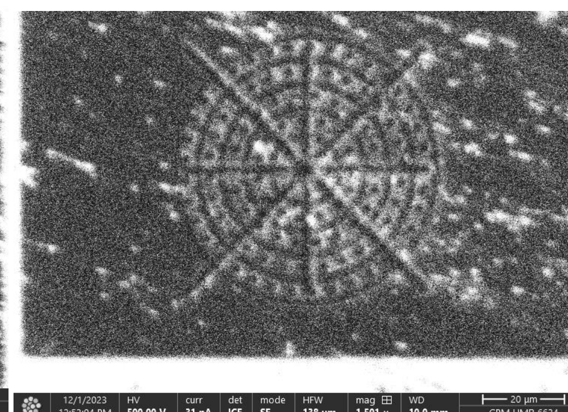
5 kV – 21 pA



1 kV –12 pA



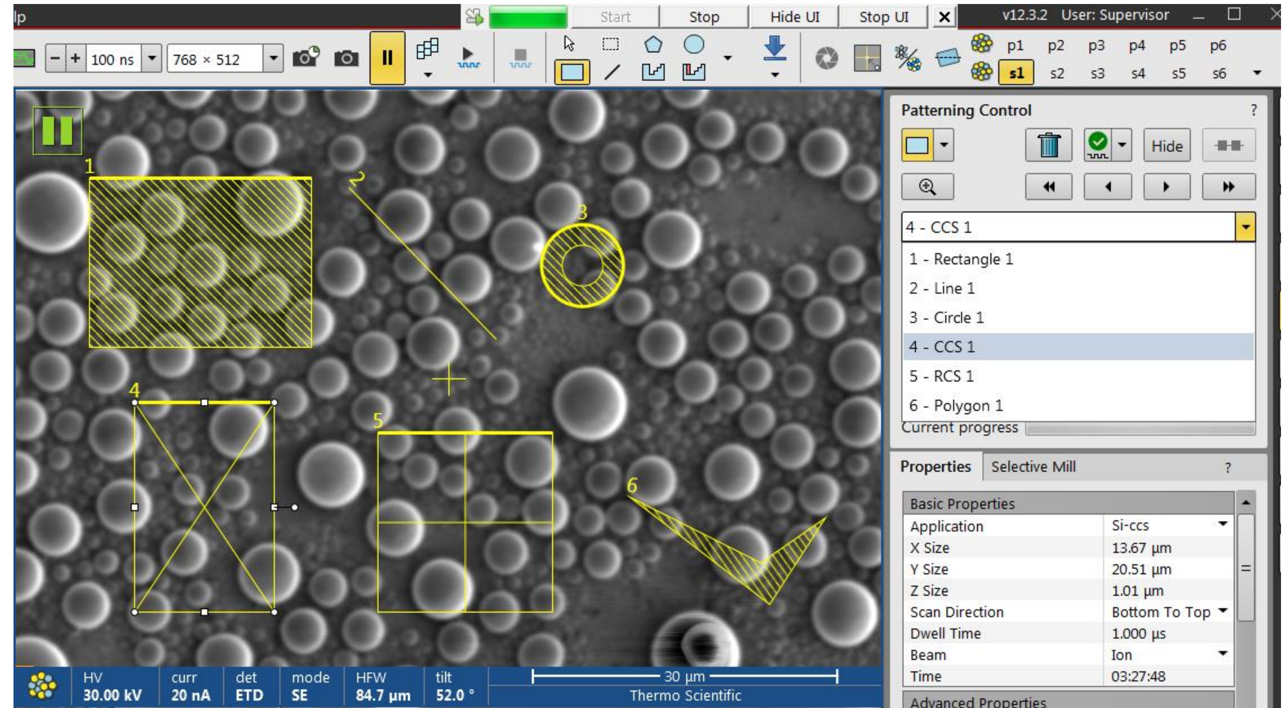
0.5 kV – 31 pA



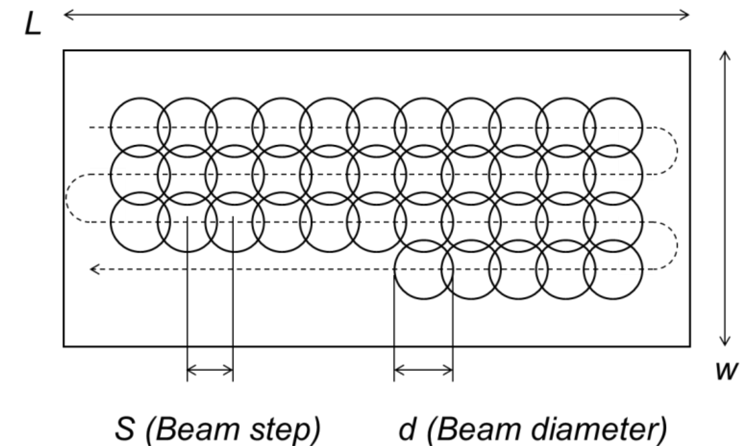
Mode de balayage du faisceau

Formes variées…

- Line
- Box
- Circle (with adjustable inner diameter)
- Polygons
- 24-bit bitmap files
- Regular cross section
- Cleaning cross section

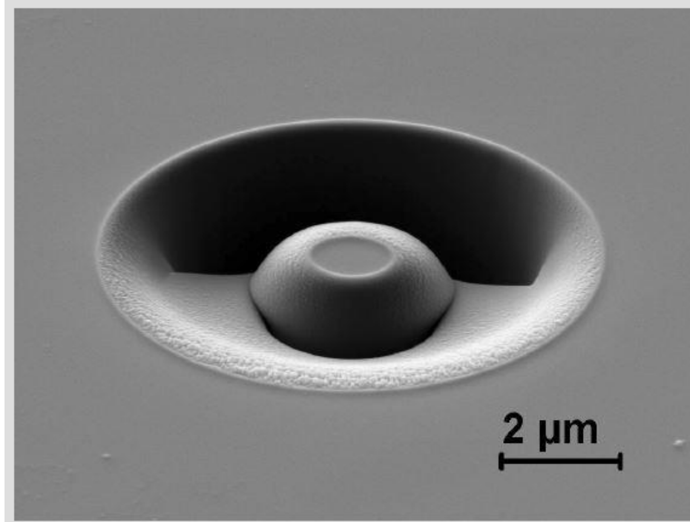


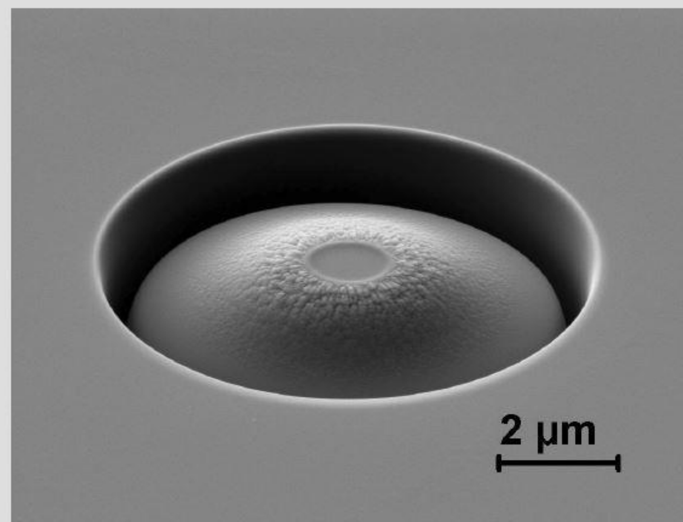
- Recouvrement du faisceau
- Dwell time (µs à ms)
- Direction du balayage (top to bottom,…)
- Mode de balayage (raster, serpentine,…)

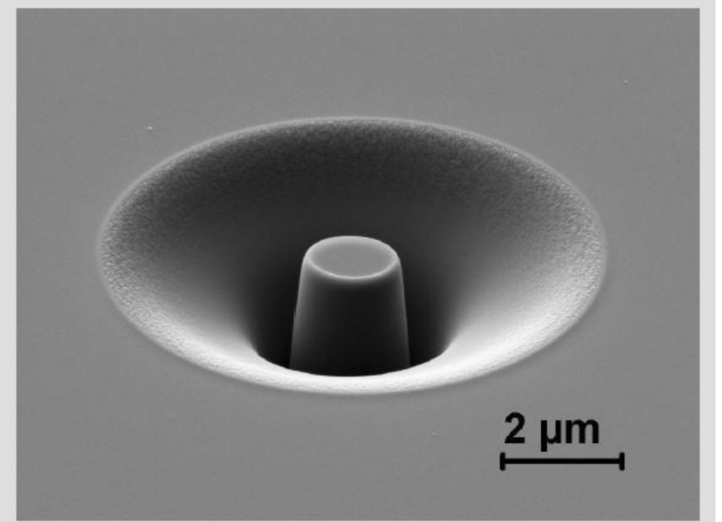


Mode de balayage

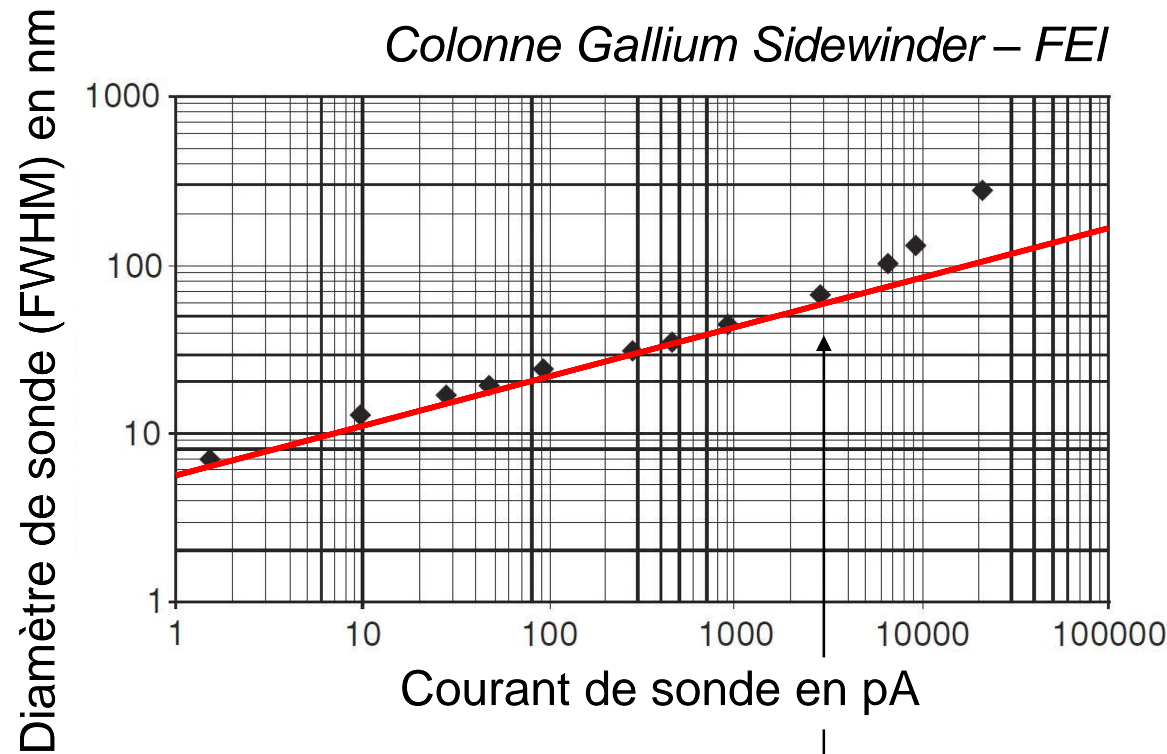
Balayage XY | **Annulaire In / Out** | **Annulaire Out / In**



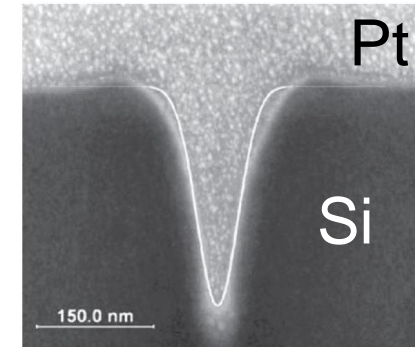




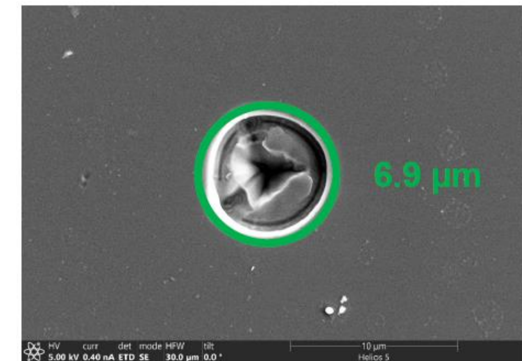
Courant - diamètre faisceau FIB



Ga - 30 kV - 93 pA



Ga - 30 kV - 100 nA



⬅ faibles courants

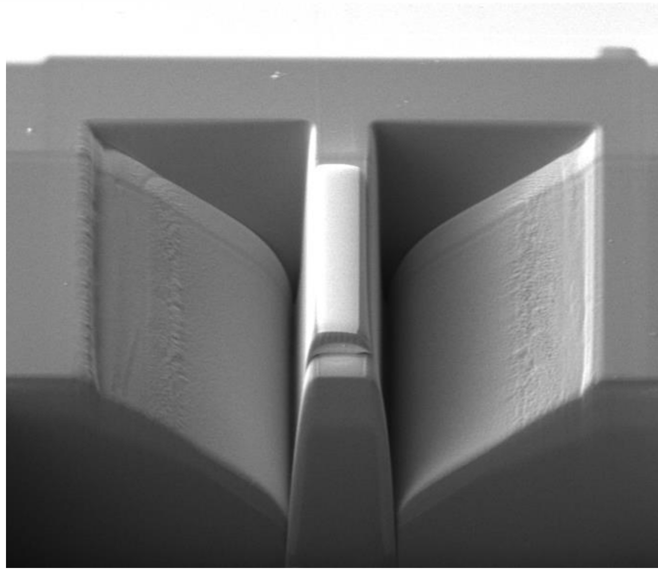
Taille de sonde petite
Gain en résolution
2.5 nm à 1 pA (30 kV)

➡ forts courants

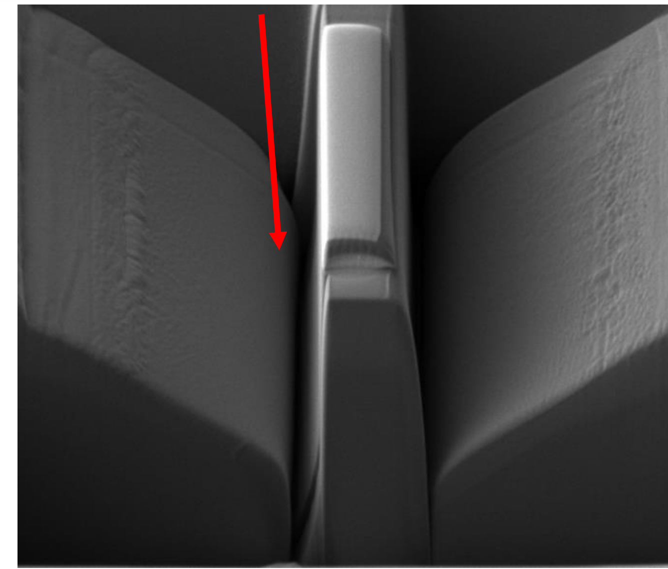
Taille de sonde devient très grande
Perte de la résolution

Limite actuelle (FIB Ga) > 100 nA

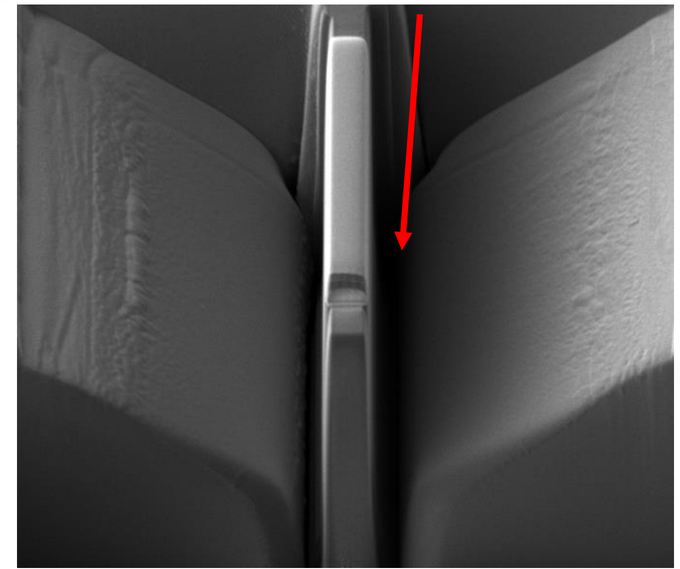
Utilisation du tilt de la platine échantillon



52°



53.5°



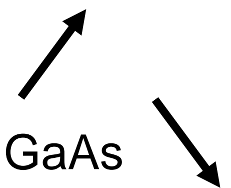
50.5°

Silicium

28 pA	48 pA	93 pA	0.28 nA	0.48 nA	0.92 nA	2.8 nA	6.5 nA	9.3 nA	21 nA
0.5^{0}	0.8^{0}	1.2^{0}	1.2^{0}	1.5^{0}	1.5^{0}	2^{0}	2^{0}	2^{0}	2^{0}

Materials harder than Silicon (lower sputter rate), i.e. Steel

Materials softer than Silicon (higher/faster sputter rate), i.e. GaAs

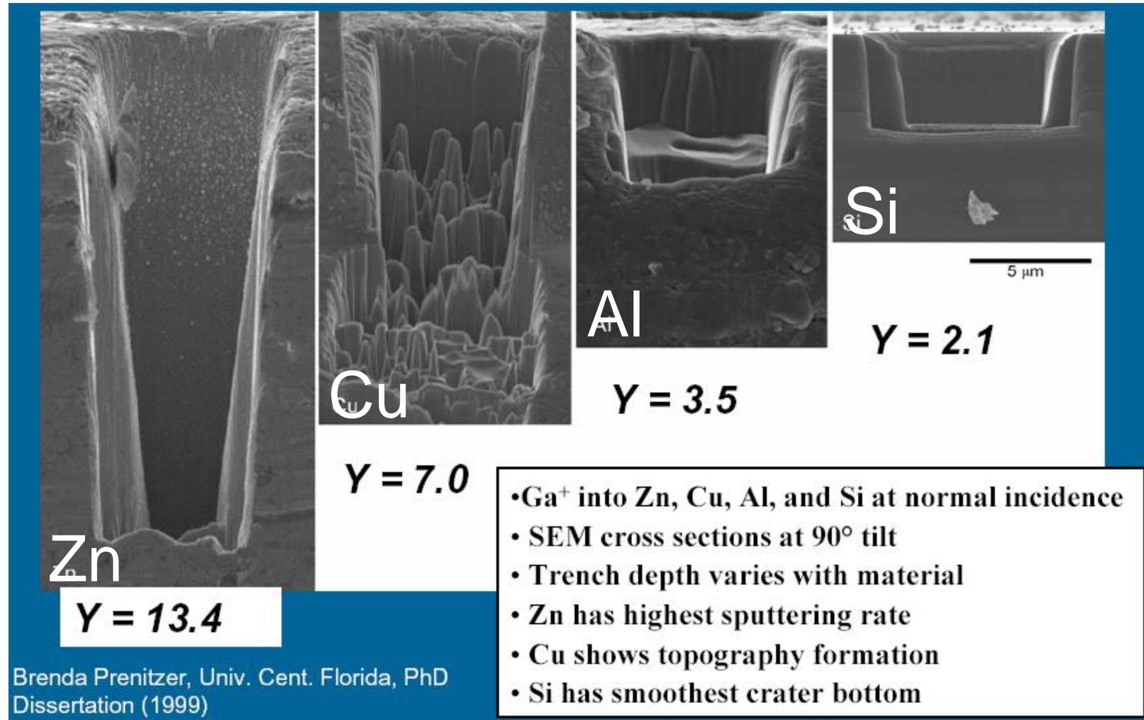


Sommaire

- Bref introduction du principe physique de l'interaction ion-matière
- Conditions opératoires :
  - Réglage du faisceau
  - Alignement du faisceau
  - Influence de l'angle d'incidence
  - Influence de la nature de la cible
  - Influence de la tension d'accélération
- Dommages et tentative de minimisation :
  - Baisser la tension d'accélération
  - Changer la nature de l'ion incident
  - Le mode Cryo FIB-SEM…

Rendement de pulvérisation Y

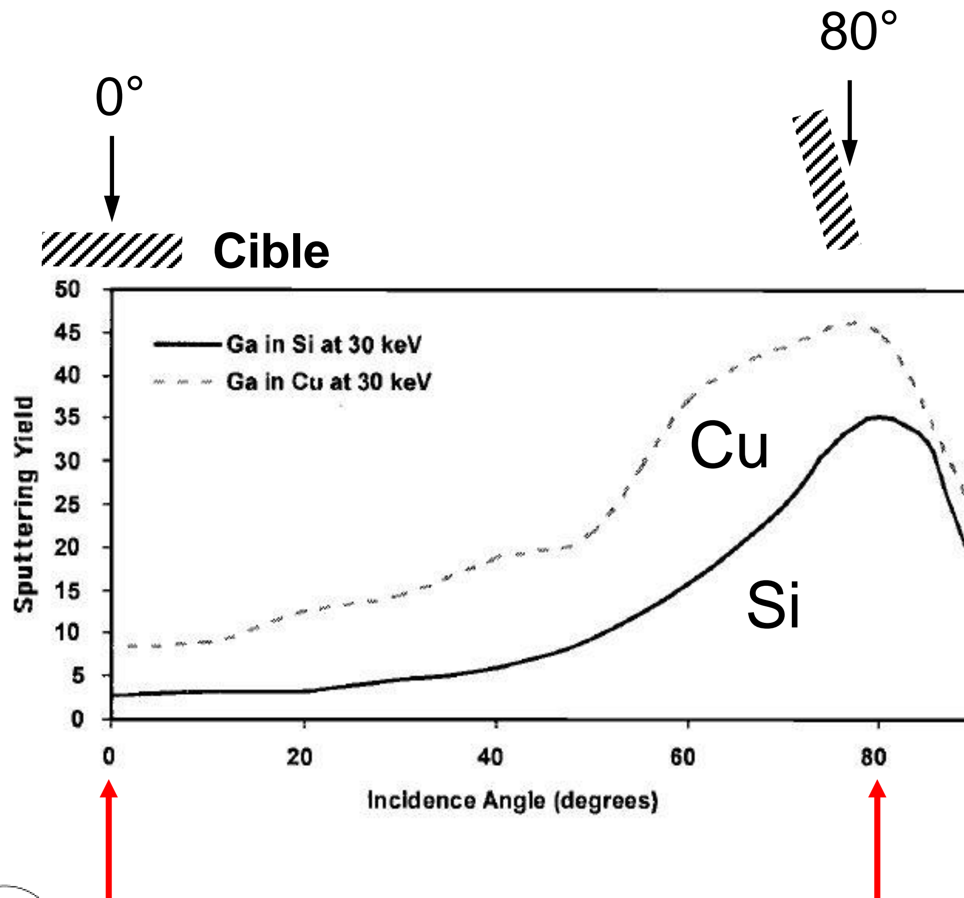
Dépendant du numéro atomique de la cible



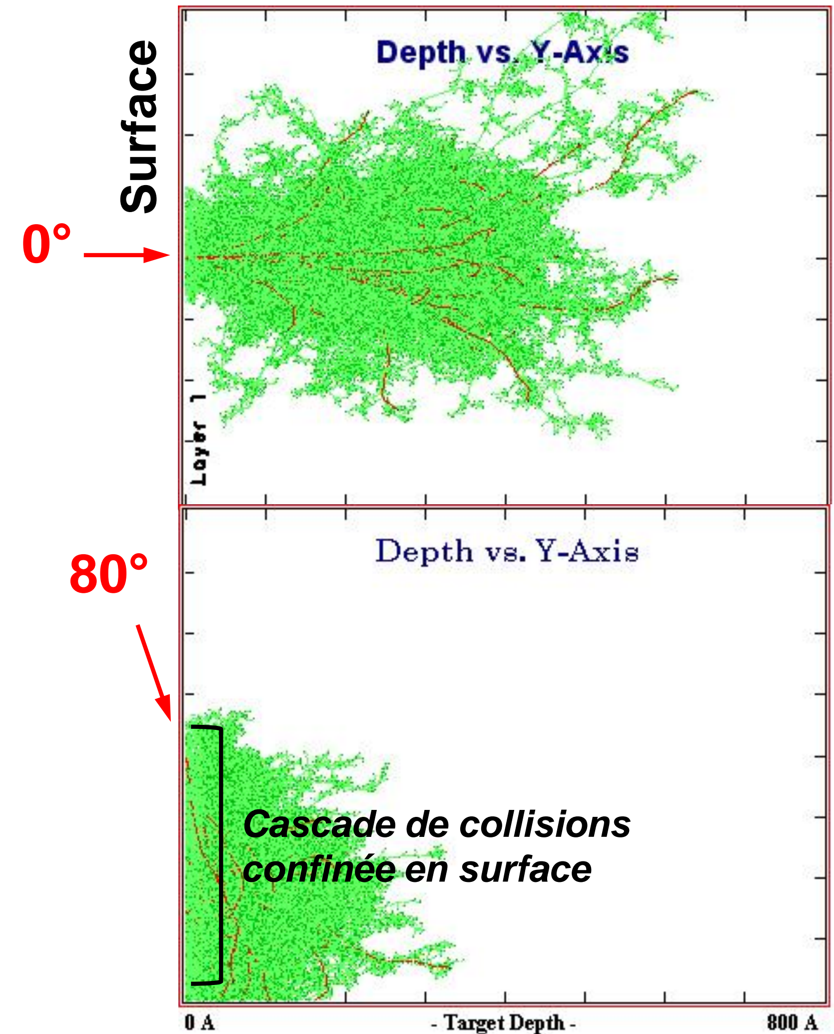
Substrate	Ion	Energy (keV)	Yield (atoms/ion)
Si	Ga^+	30	3.1 ± 0.8
Si	Ga^+	30	1.92 ± 0.16
Si	Ga^+	25	2.6
Si	Ga^+	25	3.9 ± 0.4
Si	Kr^+	25	3.1
Diamond (100)	Ga^+	20	2
Diamond	Ga^+	50	2.3
Hard amorphous carbon	Ga^+	50	2.6
GaN	Ga^+	15	5.2
		30	6.2
		50	6.8
		70	6.7
InP	Ga^+	30	7.5
GaAs	Ga^+	30	4.8
SiC	Ga^+	30	2.5
Al_2O_3	Ga^+	30	1.9
Au	Ga^+	100	32
Au	Ga^+	40	15.7 ± 1.3
Au (plated)	Ga^+	25	18 ± 3
Au (evap.)	Ga^+	25	23 ± 5
Au	Kr^+	25	20
W (RF-sputt.)	Ga^+	25	5 ± 0.7
W	Kr^+	22	4.1
Al	Ga^+	30	1.1–4.5
Al	Si^+	30	–0.26
		120	–0.09
SiO_2	Ga^+	68	2.0 molec./ion
SiO_2	Ga^+	25	0.84
SiO_2	Ga^+	30	0.85
SiO_2	Ga^+	30	2.0

Rendement de pulvérisation Y number of ejected particles per incident ion

Dépendant de l'angle d'incidence du faisceau

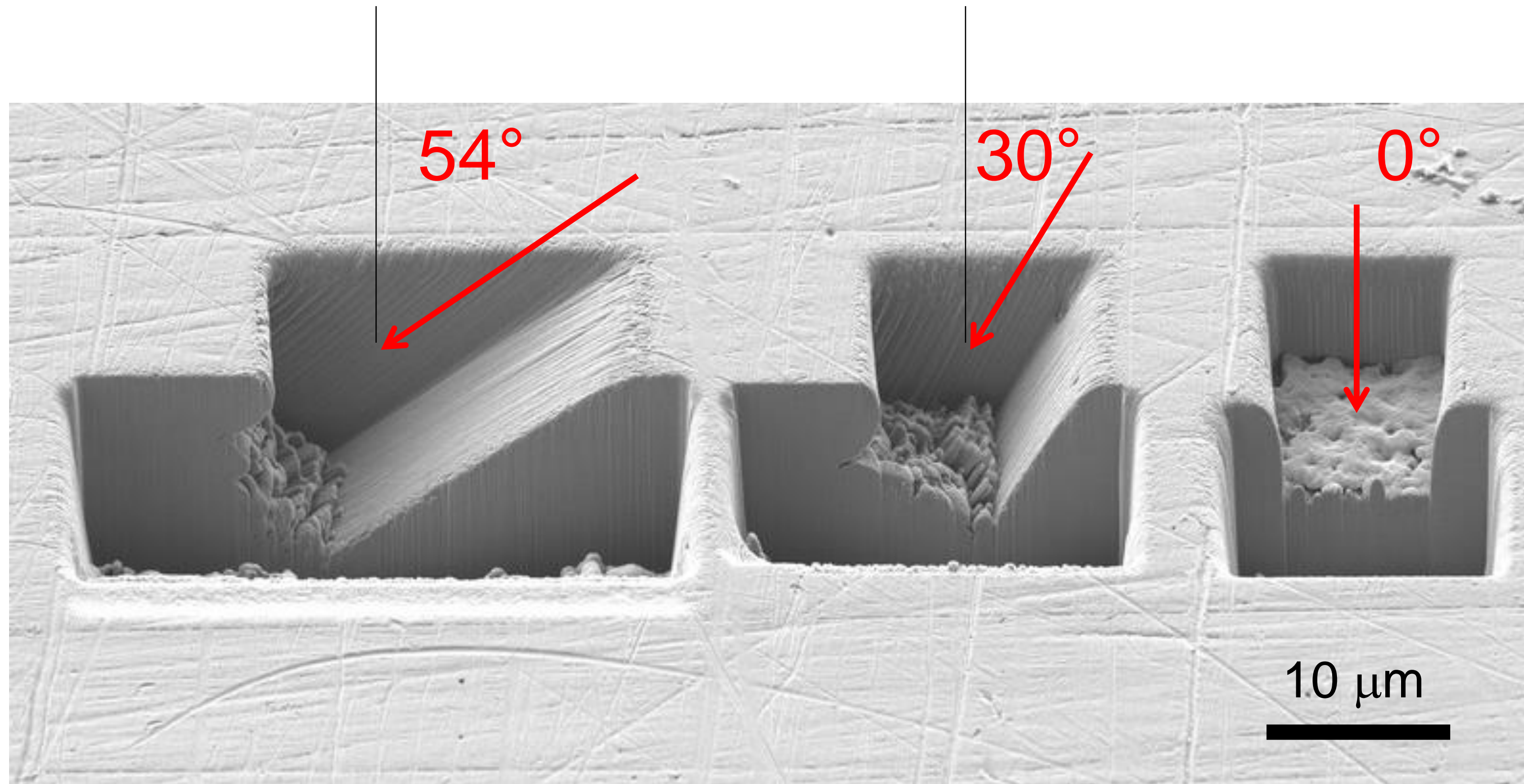


Ga - 30 kV dans Si



Rendement de pulvérisation Y

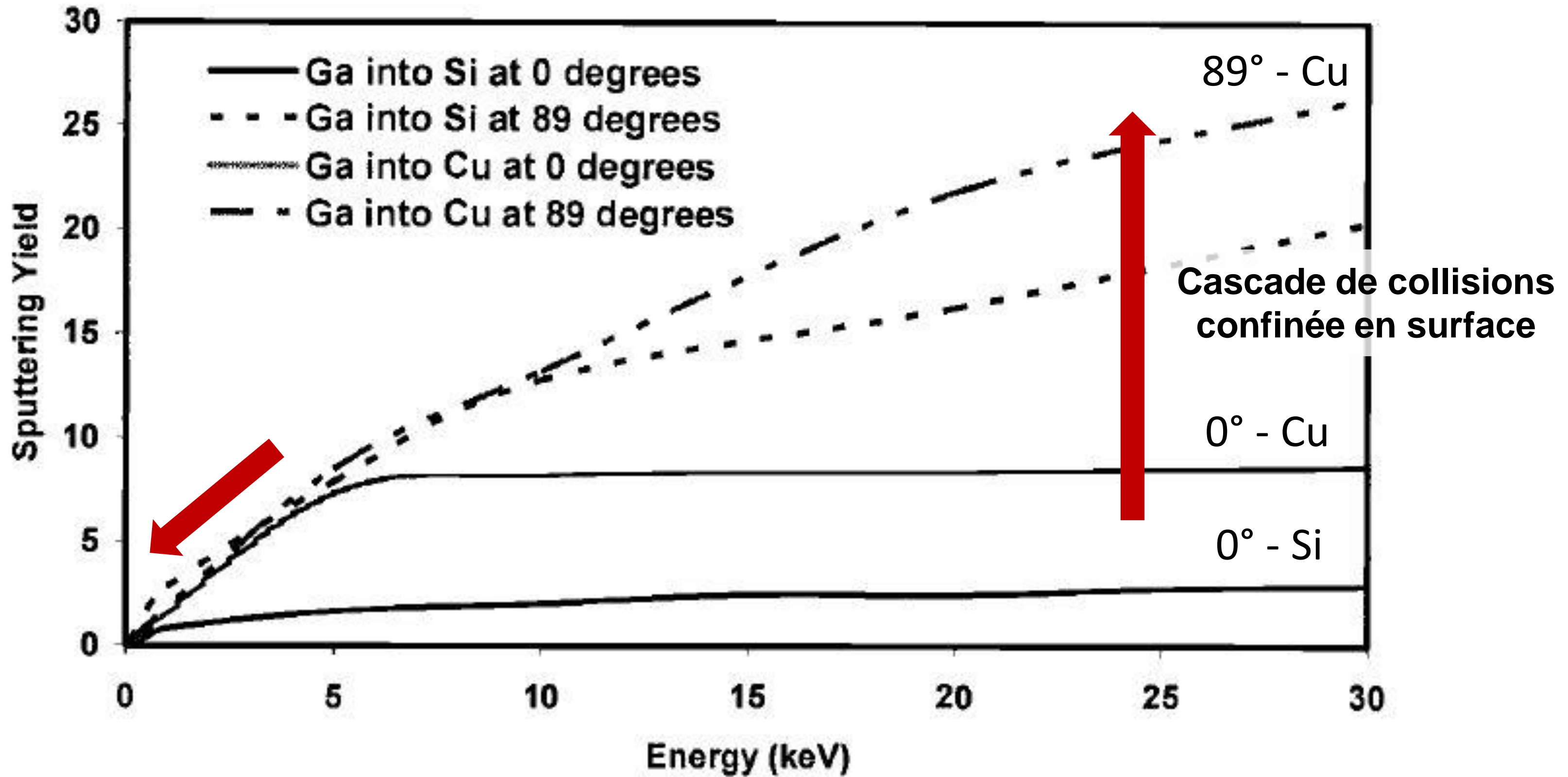
Dépendant de l'angle d'incidence du faisceau



même dose et pattern 10 x 10 µm - Ga @ 30 kV sur Nickel

Rendement de pulvérisation Y

Dépendant de la tension d'accélération du faisceau

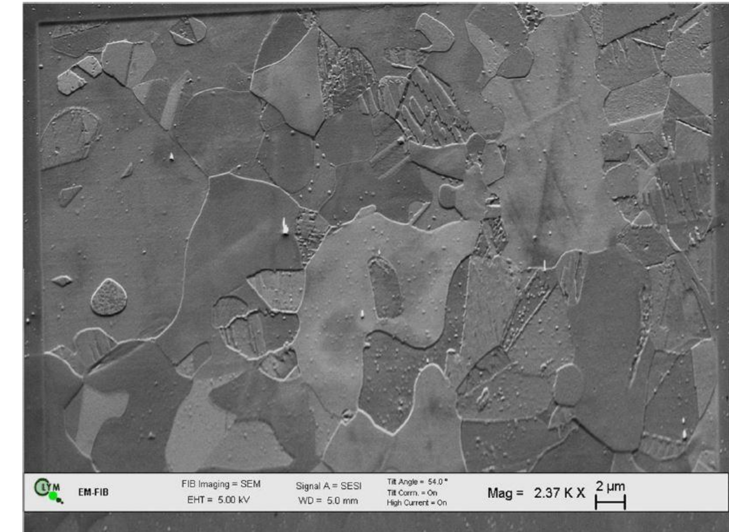


- Bref introduction du principe physique de l'interaction ion-matière
- Conditions opératoires :
    - Réglage du faisceau
    - Alignement du faisceau
    - Influence de l'angle d'incidence
    - Influence de la nature de la cible
    - Influence de la tension d'accélération
- Dommages et tentatives de minimisation :
    - Baisser la tension d'accélération
    - Changer la nature de l'ion incident
    - Le mode Cryo FIB-SEM…

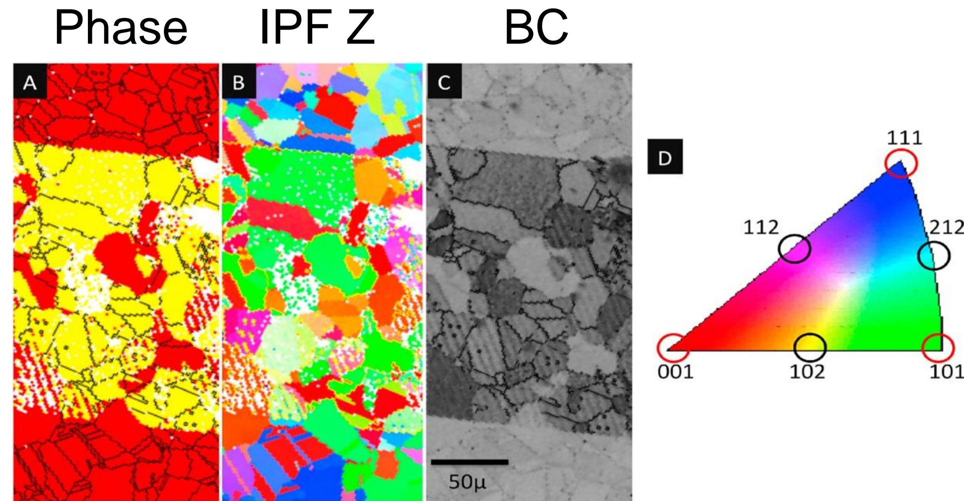
Modification du matériau sous faisceau d'ions

Pour minimiser le phénomène :

- adapter le courant d'imagerie à l'étendue de la surface balayée par le faisceau FIB,
- augmenter la vitesse de balayage (dwell time),
- éviter de balayer continuellement (blank, freeze,…).



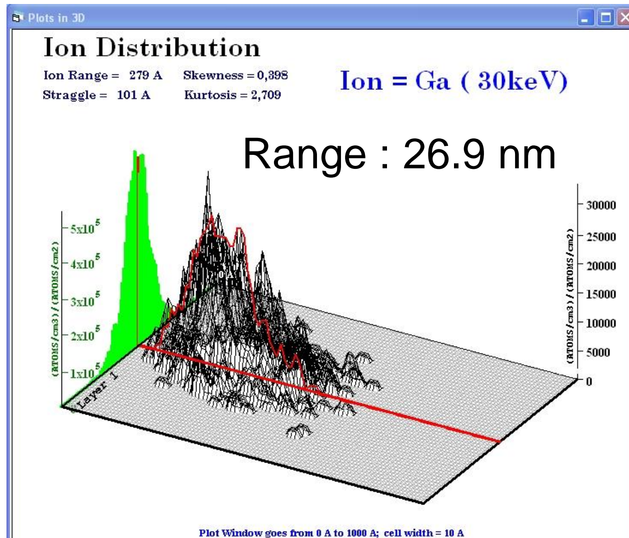
Transformation Austenite γ (rouge) en Ferrite α (jaune)



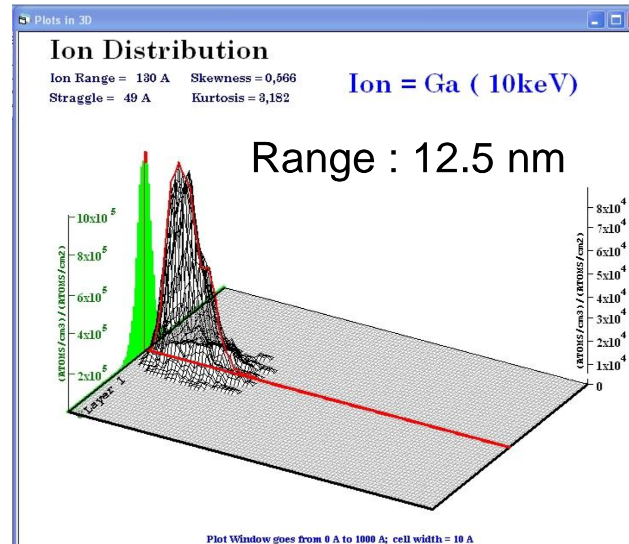
Acta Materialia 120 (2016) 391 / Journal of Microscopy, Vol. 245 (2012) 288

Minimiser l'implantation : baisser la tension d'accélération

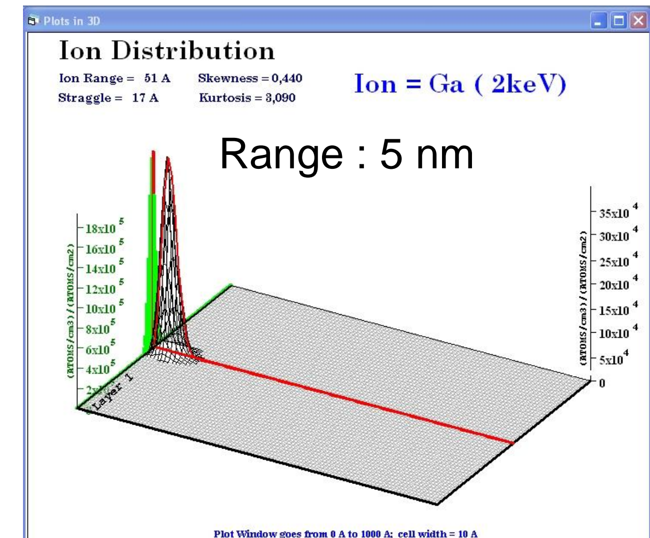
30 keV



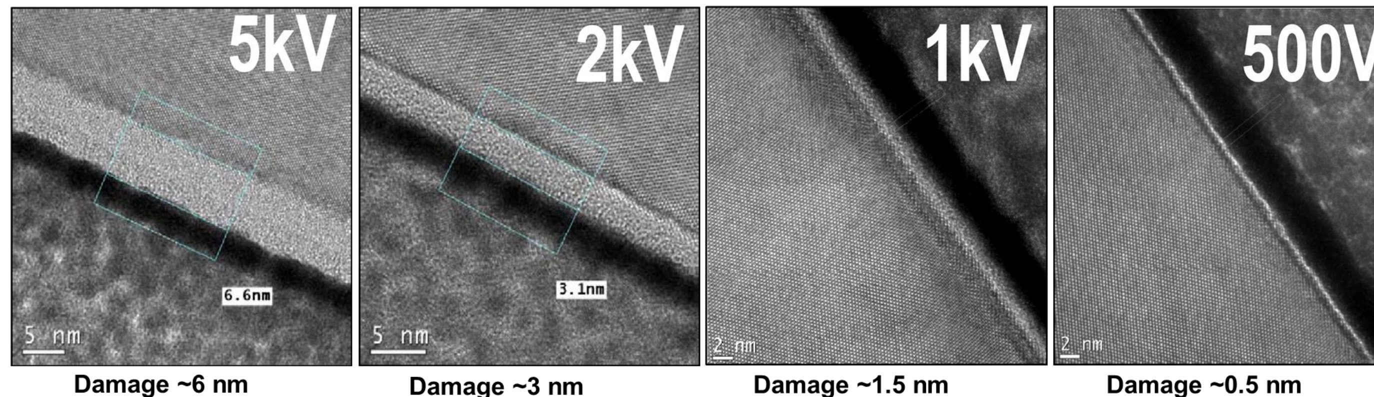
10 keV



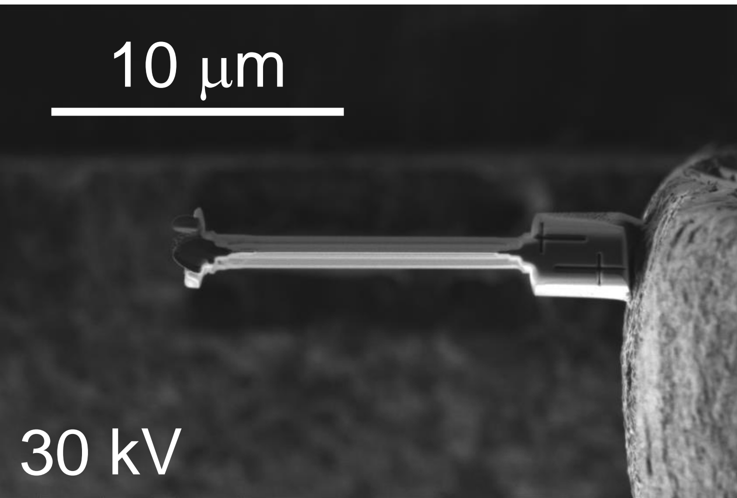
2 keV

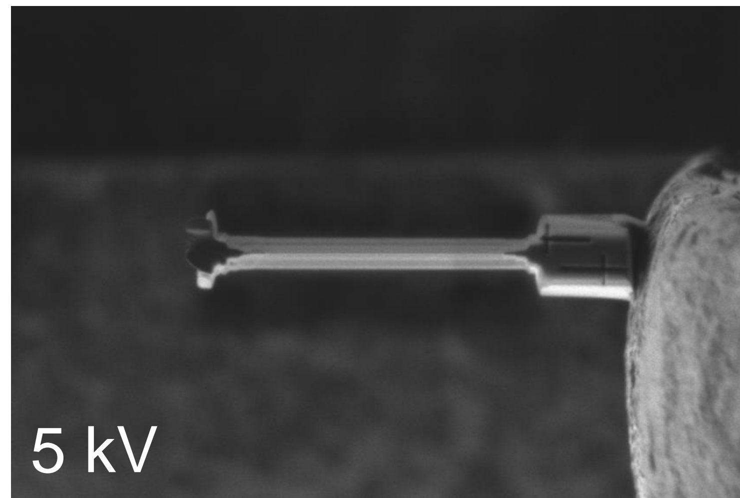


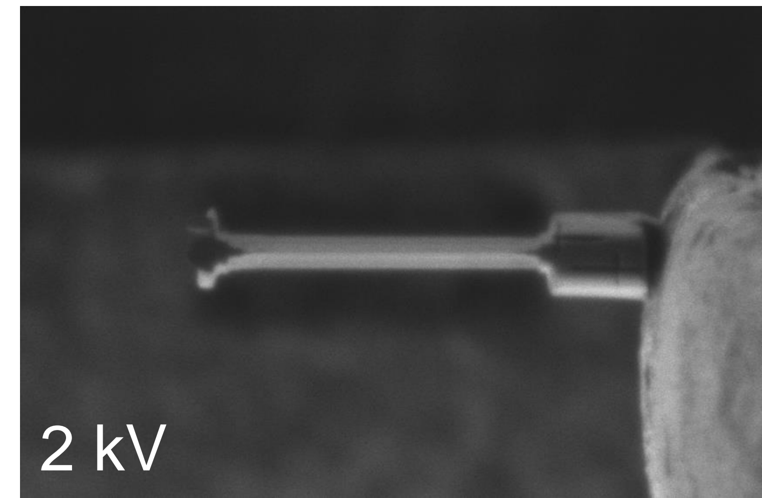
Cross section of a Si thinned sample at various kV

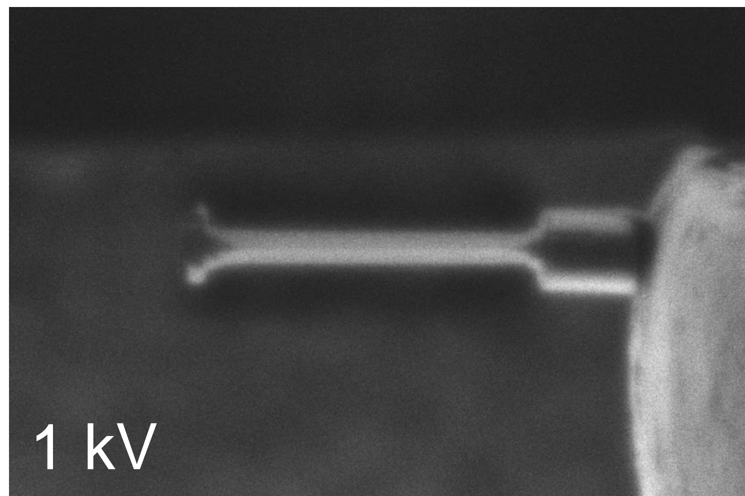


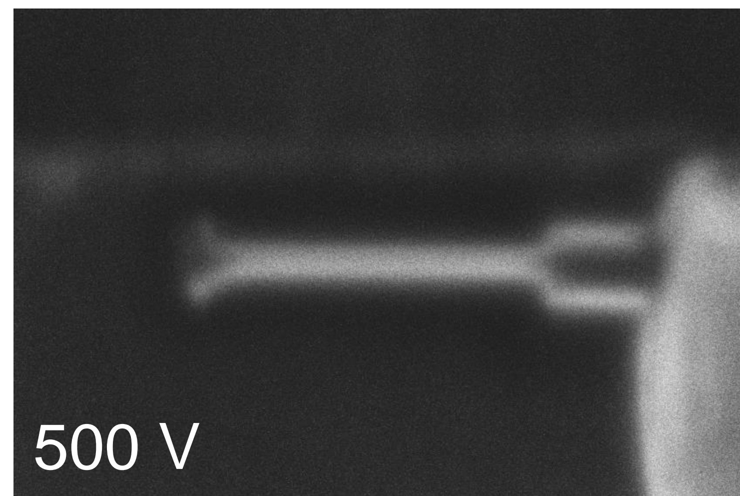
Résolution basse tension

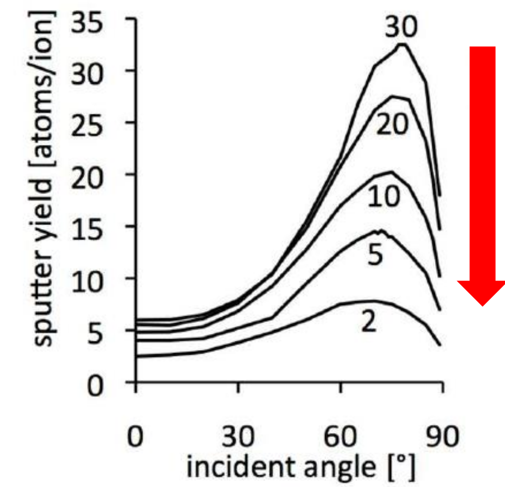






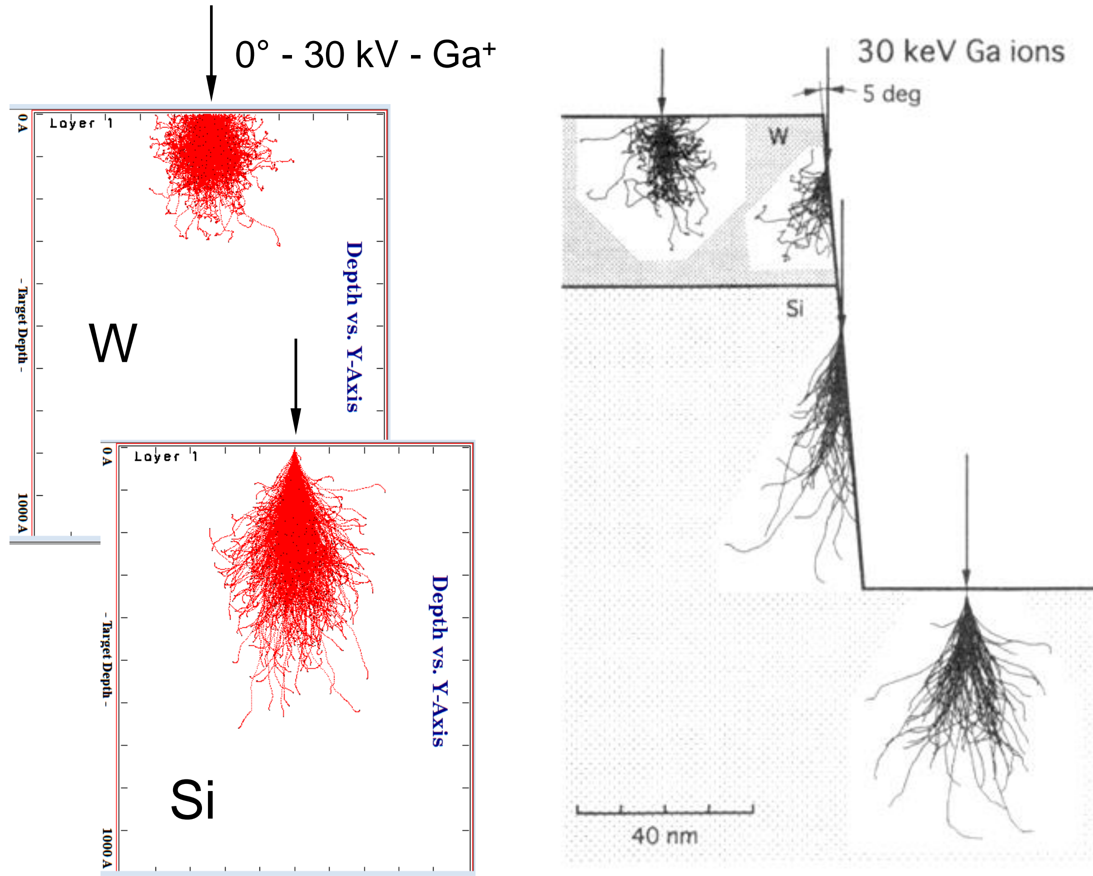


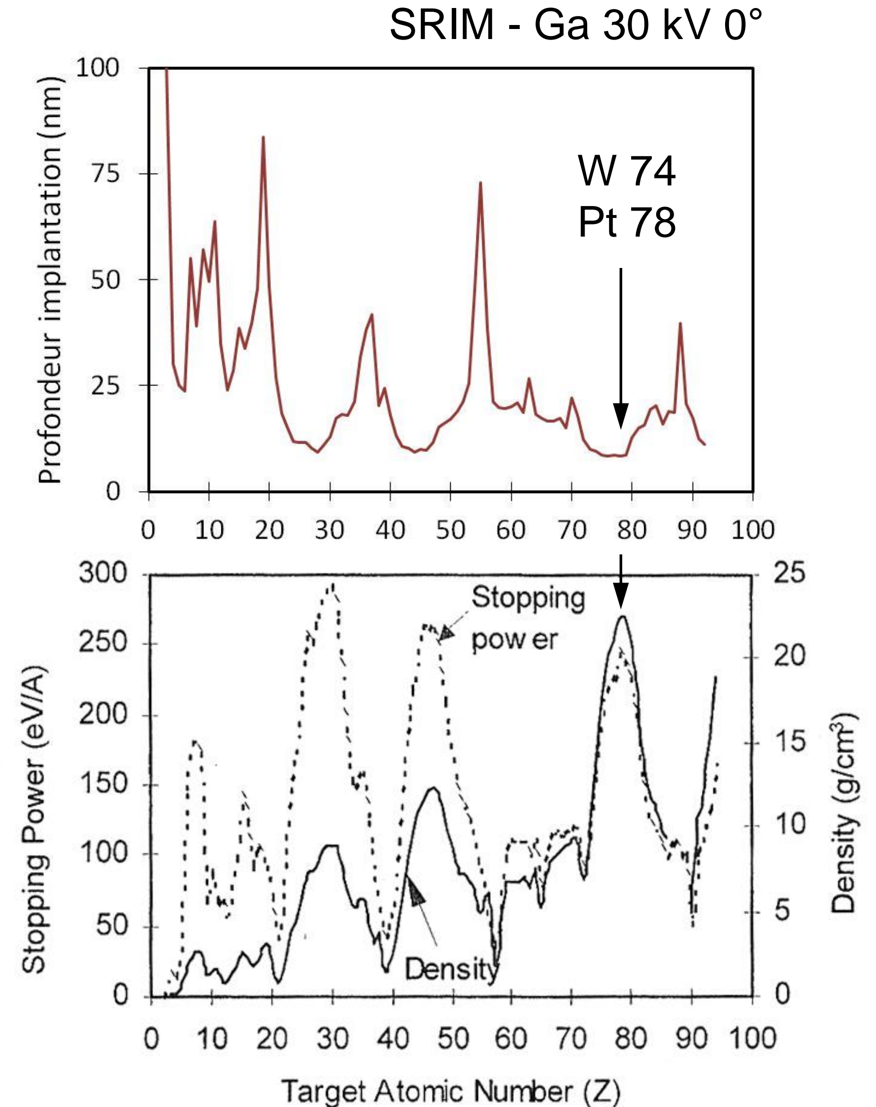




Implantation du Gallium : Protection Pt ou W

Lié au pouvoir d'arrêt du matériau cible

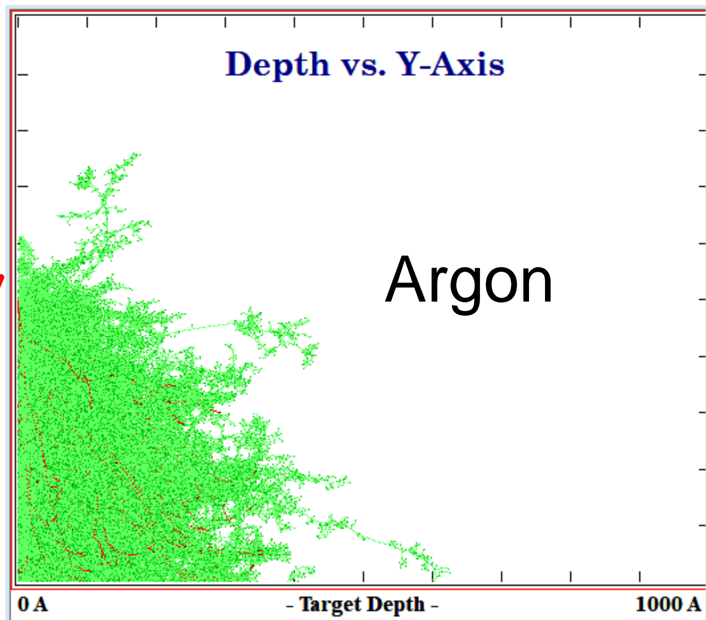


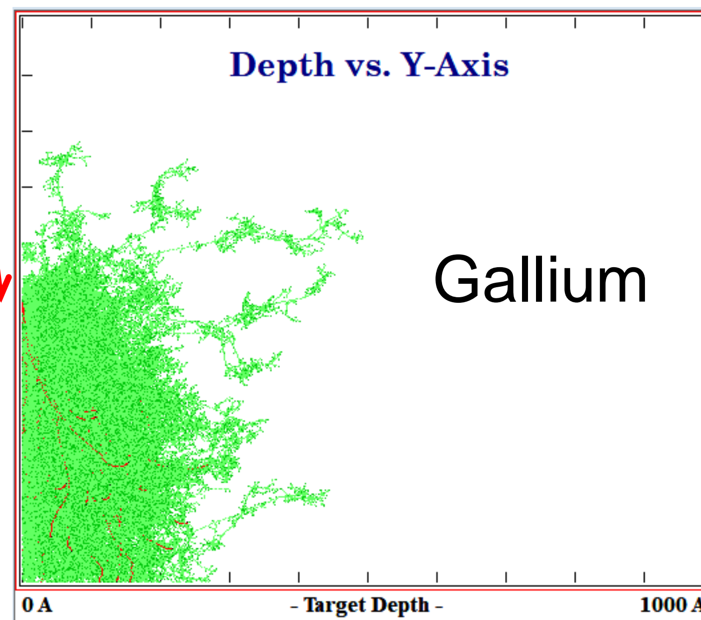


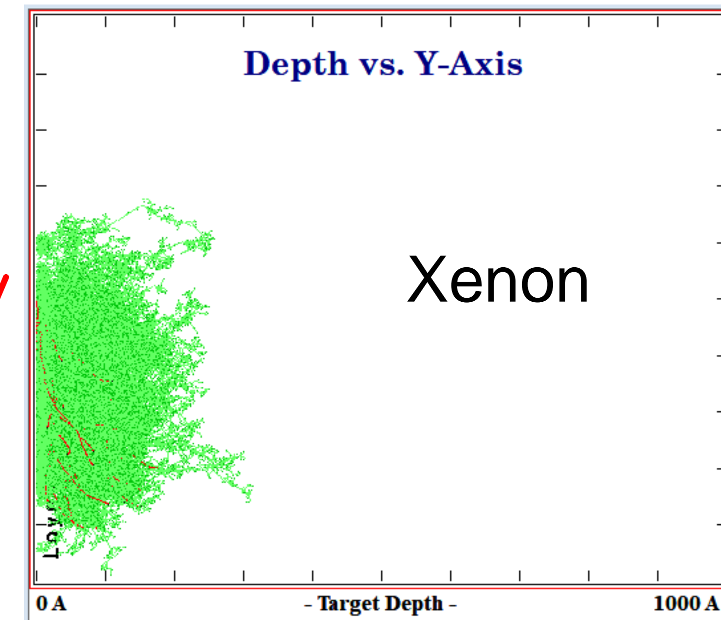
Implantation latérale

Ions incidents @ 30kV, 88°

Ar 371 lacunes / ion	Ga 330 lacunes / ion	Xe 356 lacunes / ion
lateral proj 37.1 nm	lateral proj 28.5 nm	lateral proj 23.8 nm







Damage-free Si layer :

Argon 26 %
Gallium 43 %
Xenon 53 %

Rp % changes by switching ion at different ion energy

● Ga vers Xe ● Ga vers Ar ● Xe vers Ar

Rp % changes by switching ion

60%
50%
40%
30%
20%
10%
0%
-10%
-20%

0 5 10 15 20 25 30

Ion energy keV

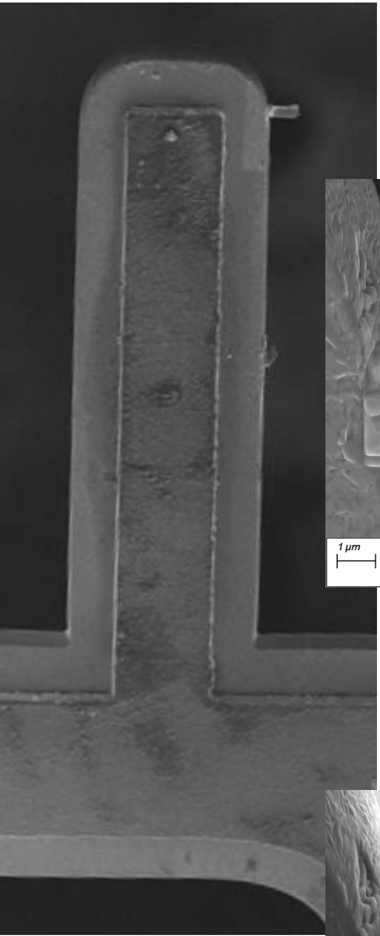
< 1.5 keV $Ga^+ \longrightarrow Ar^+$

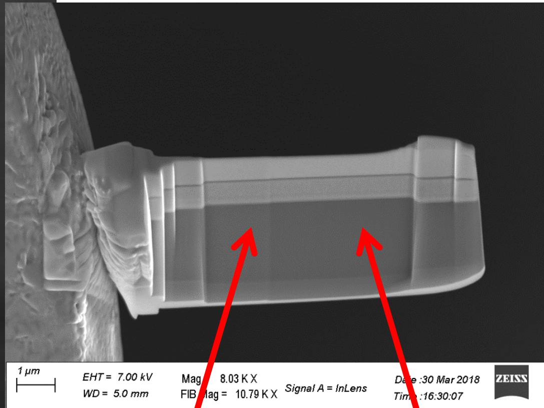
> 6.5 keV $Ga^+ \longrightarrow Xe^+$

< 3 keV $Xe^+ \longrightarrow Ar^+$

Argon : pour des applications < 1.5 kV
Xenon : pour des applications à haute tension

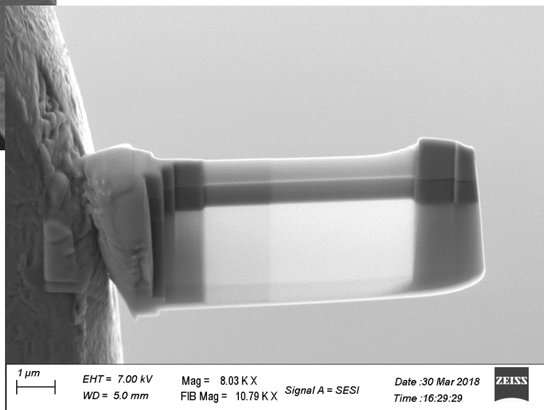
Amincissement Post-FIB : 0.5 keV Argon "broad beam"



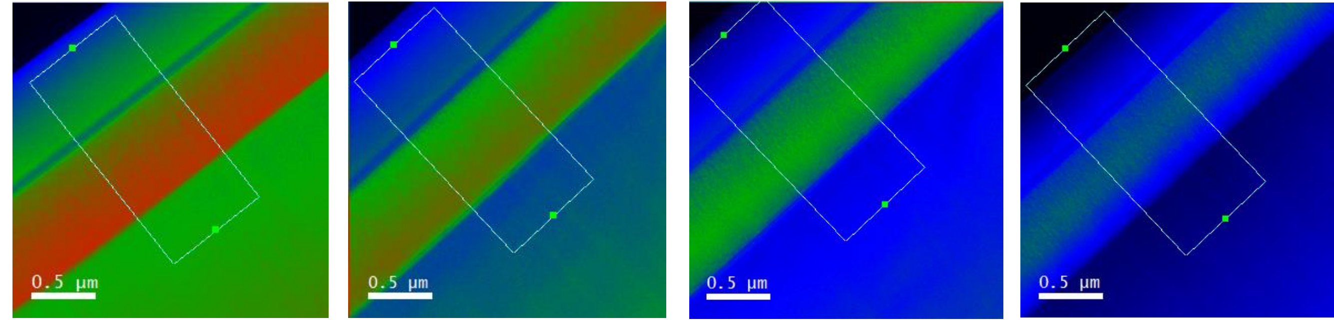


+8°
0.5 keV Ar+

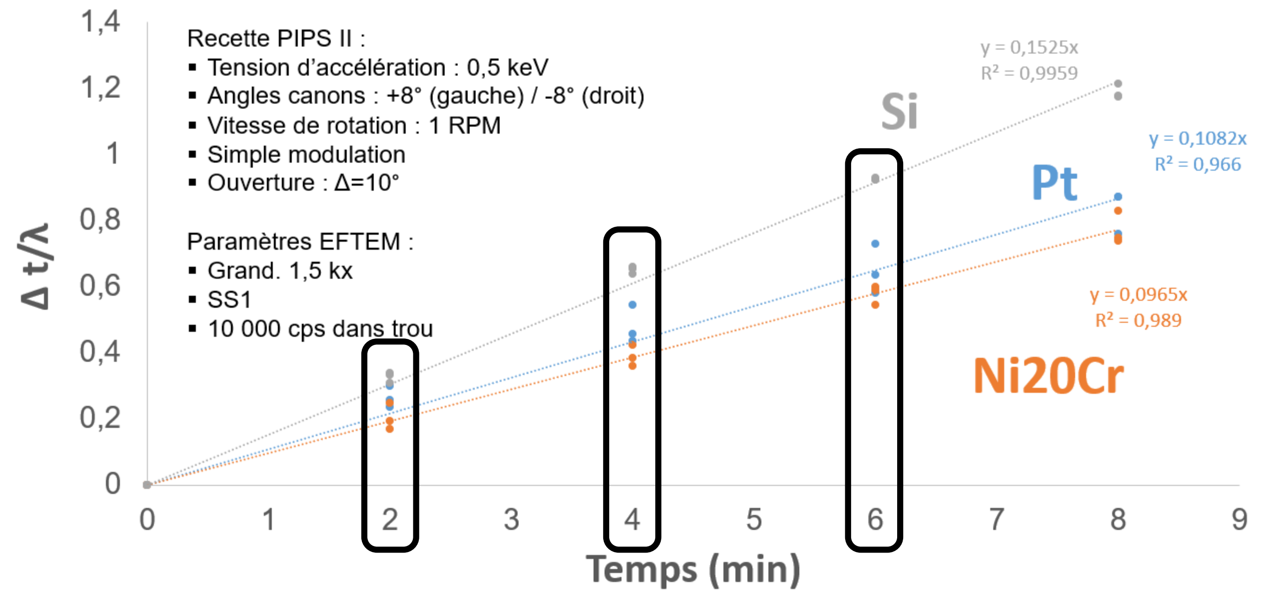
-8°
0.5 keV Ar+



EFTEM

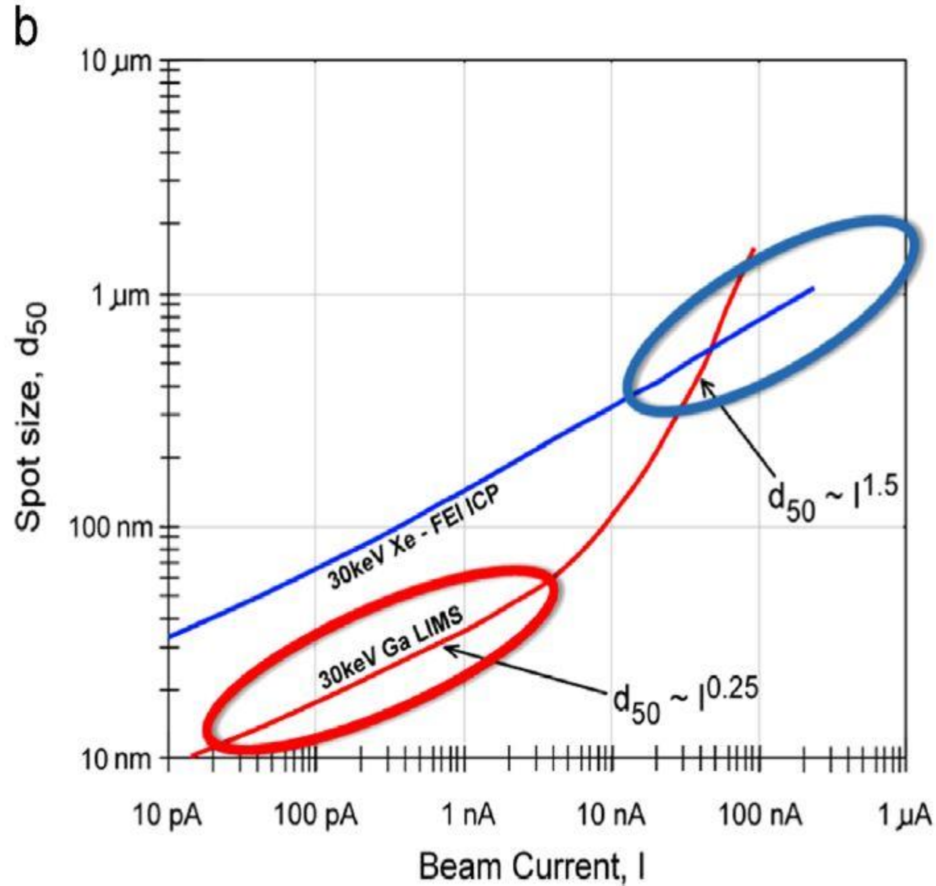


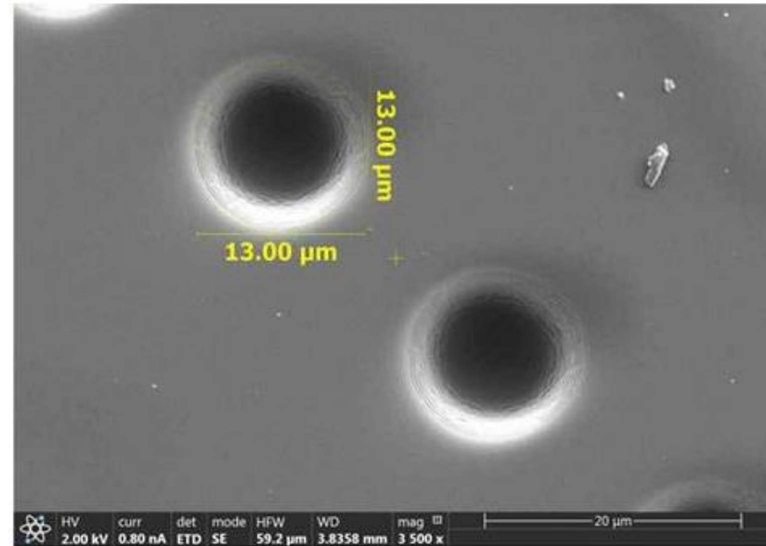
Post-FIB | PIPS II 2 min | PIPS II 4 min | PIPS II 6 min



Choix de la technologie Plasma-FIB

- Fort courant disponible (> 2.5 µA, si le matériau résiste…)
- Diamètre de faisceau > faisceau Ga
- Interaction ion / matière (Al, Mg, …)



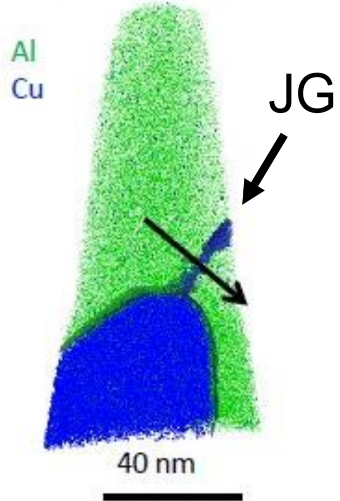


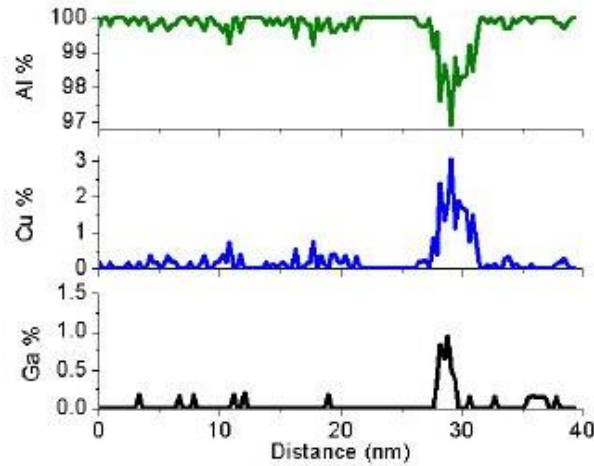
PFIB Xe 2.5 µA

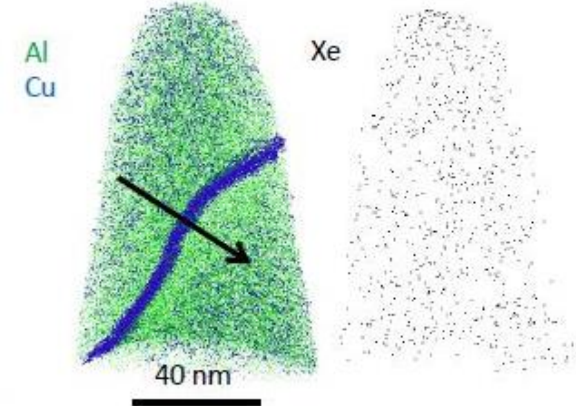
➡ Xe, Ar, N, O

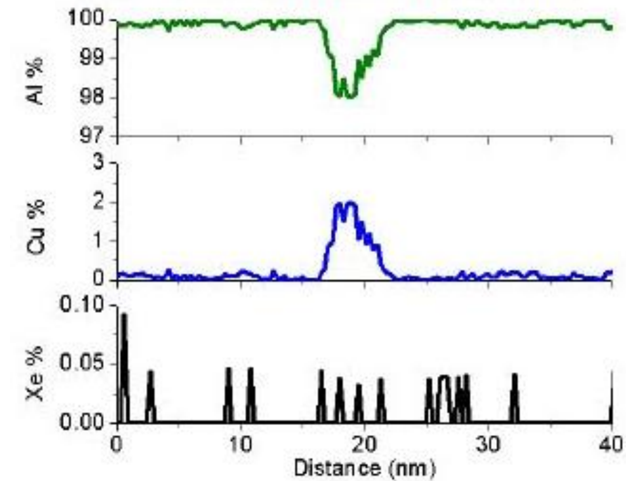
Choix de la technologie Plasma-FIB

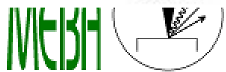
Diffusion du Gallium dans les défauts (JG, dislocation,…)











Ga-FIB dans Aluminium

Plasma-FIB

Bénéfice du Cryo FIB-SEM

- Indispensable pour les matériaux cryo-fixés

- Réduire l'échauffement local :
  lié à l'utilisation du faisceau d'ions ou d'électrons

Low melting temperature materials

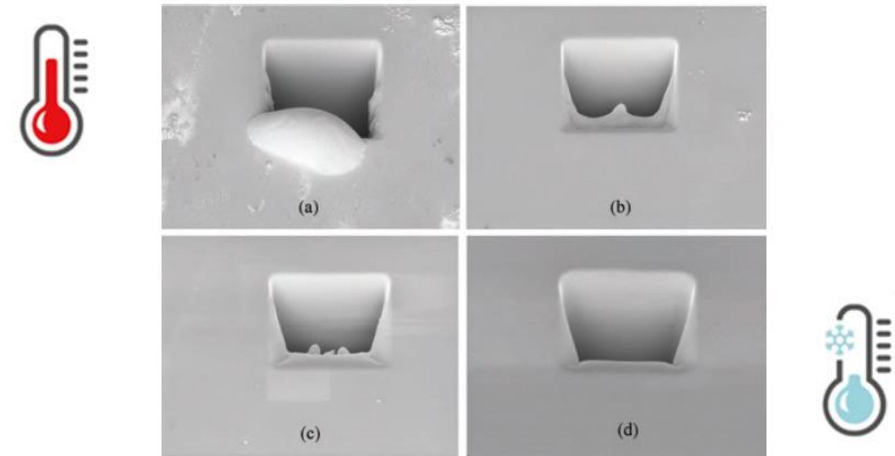


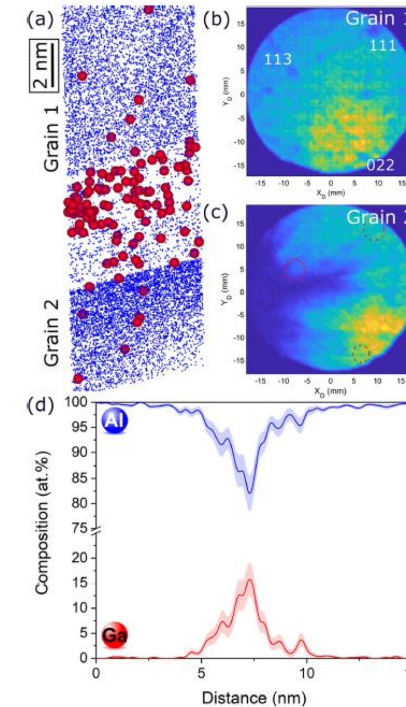
Fig. 12 GaN exposed to 30 keV Ga^+, dose: 5.5 nC/μm^2 at (a) 248, (b) 223, (c) 173, and (d) 128 K

Failure Analysis of Electronic Material Using Cryogenic FIB-SEM EDFAAO (2013) 3:12-19

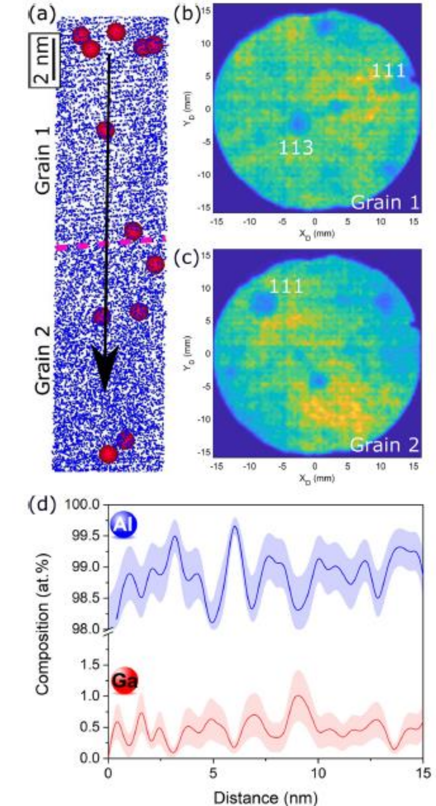
Bénéfice du Cryo FIB-SEM

- Indispensable pour les matériaux cryo-fixés
- Réduire l'échauffement local :
  lié à l'utilisation du faisceau d'ions ou d'électrons
- Réduire les artefacts :
  implantation Ga, +H sur Zr et Ti

Analyse de JG en APT



Préparation T_{amb}

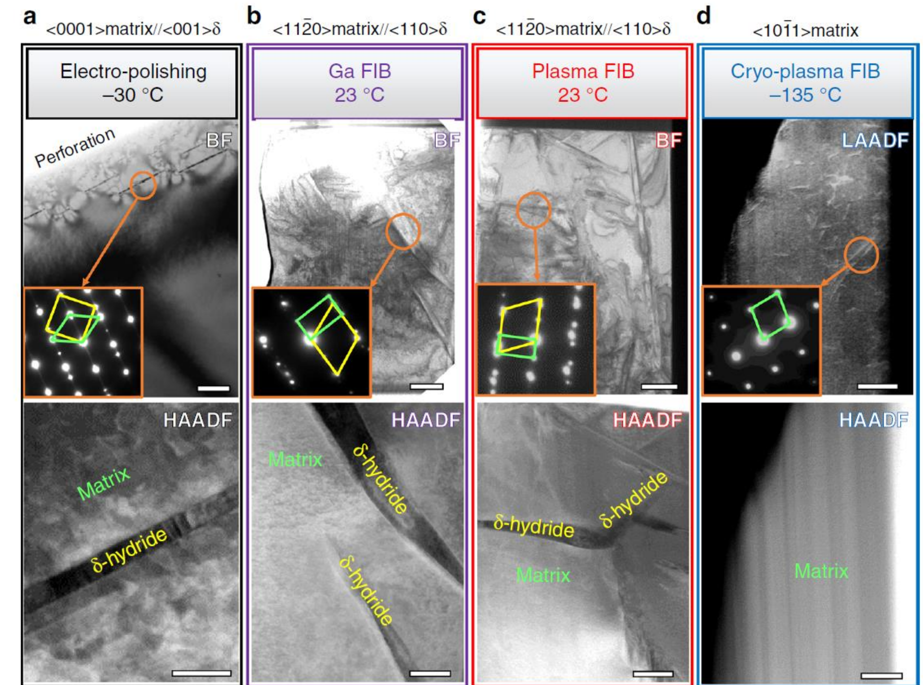


Préparation T_{cryo}

New approach for FIB-preparation of atom probe specimens for aluminum alloys PLOS ONE https://doi.org/10.1371/journal.pone.0231179 April 2, 2020

Bénéfice du Cryo FIB-SEM

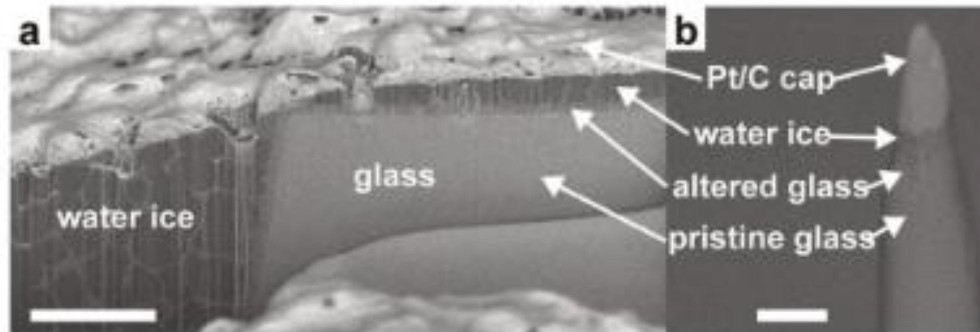
- Indispensable pour les matériaux cryo-fixés
- Réduire l'échauffement local :
  lié à l'utilisation du faisceau d'ions ou d'électrons
- Réduire les artefacts :
  implantation Ga, +H sur Zr et Ti

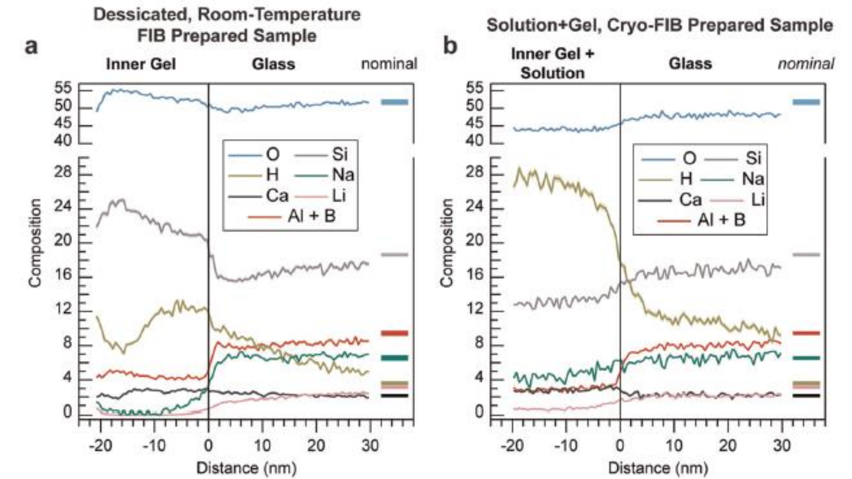


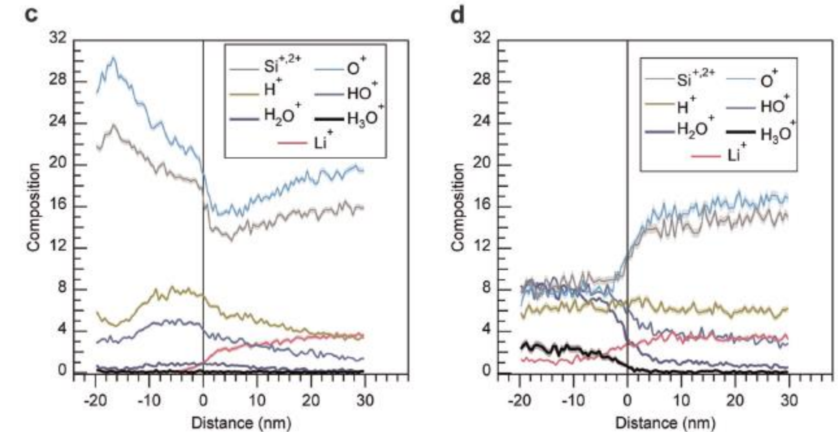
Ti and its alloys as examples of cryogenic focused ion beam milling of environmentally-sensitive materials NATURE COMMUNICATIONS | (2019) 10:942

Bénéfice du Cryo FIB-SEM

- Indispensable pour les matériaux cryo-fixés
- Réduire l'échauffement local :
  lié à l'utilisation du faisceau d'ions ou d'électrons
- Réduire les artefacts :
  implantation Ga, +H sur Zr et Ti
- Stabiliser les éléments mobiles (Li, H,…)





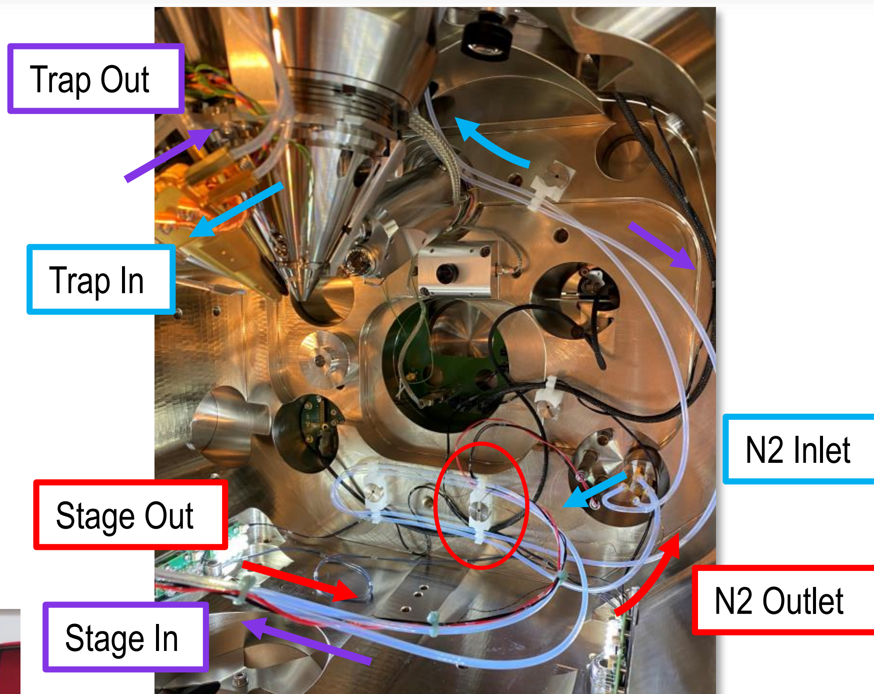


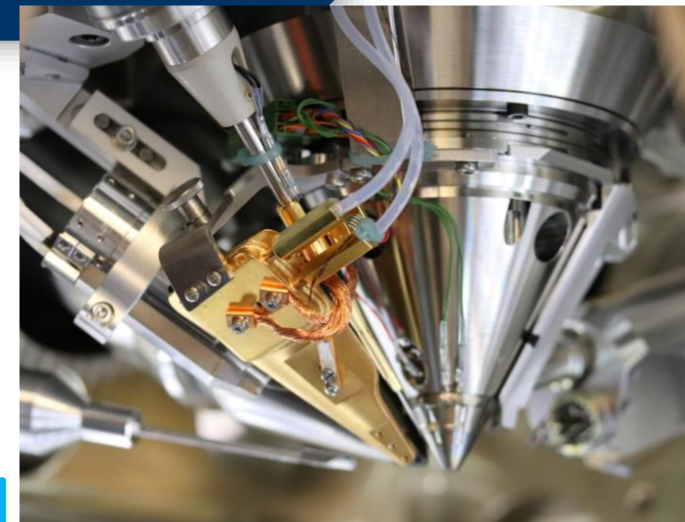
Tomographic mapping of the nanoscale water-filled pore structure in corroded borosilicate glass Materials Degradation (2020) 4:8 ; https://doi.org/10.1038/s41529-020-0110-5

Accessoires Cryo- SEM FIB

Cryo-Easylift

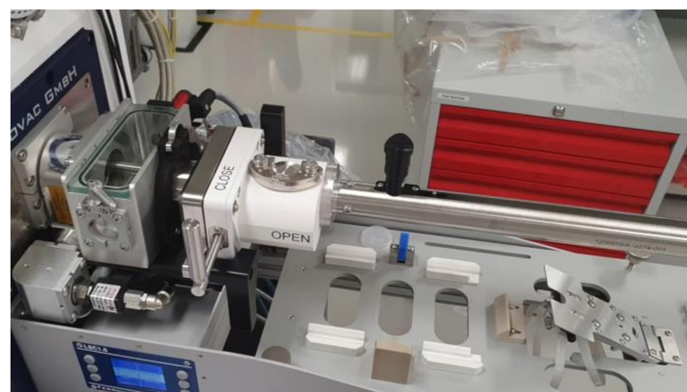
Production N2 – 5 bars

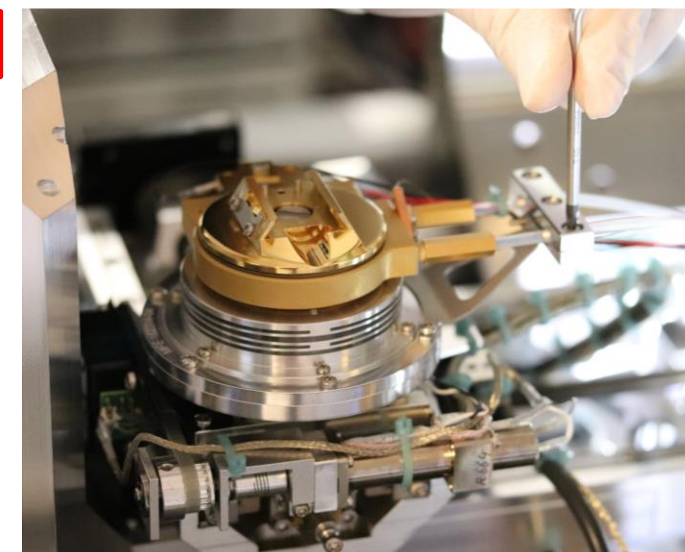




Cold Trap

Aquilos like / IGST transfer rod





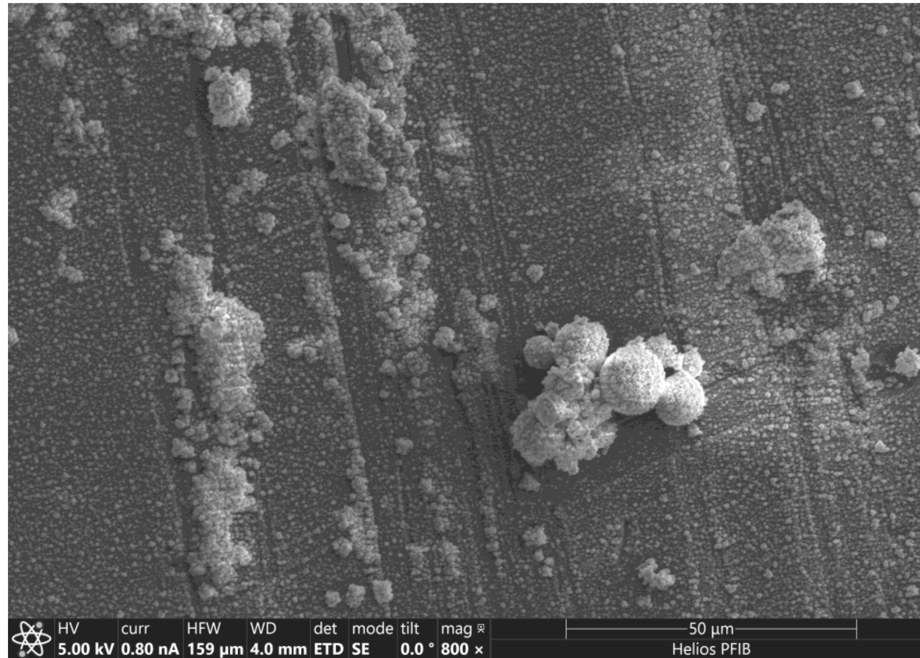
Cold stage **Rot 360°, T + 55° + Heater**

Heat exchanger LN2 dewar

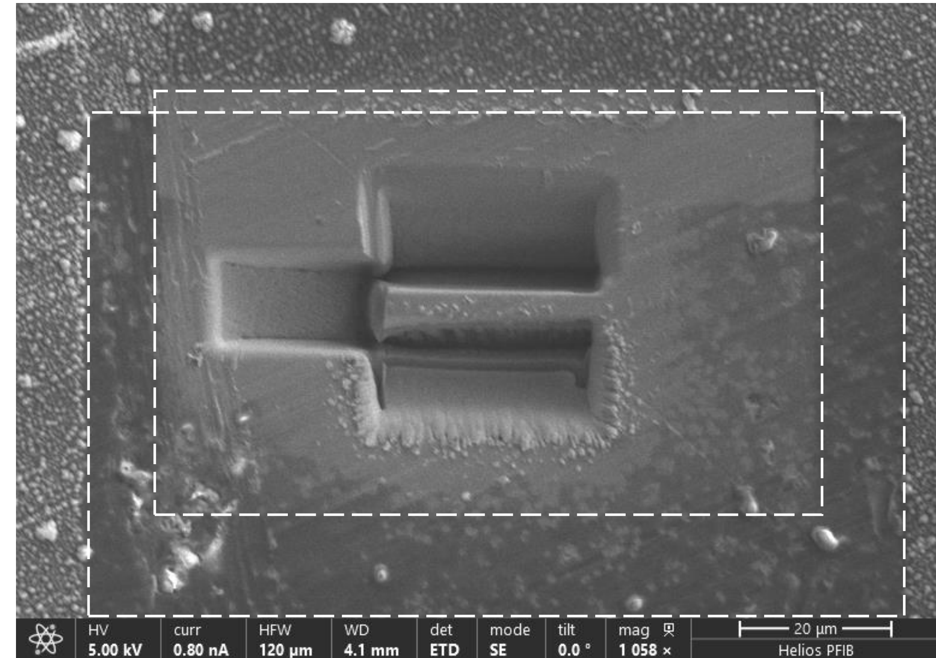
Imagerie SEM et Usinage FIB

SEM 5 kV 0.8 nA (ETD SE)

Préparation Lift-Out @ temp Cryo
Xenon PFIB : 60 nA – 15 nA – 4 nA
Tilt stage 52°

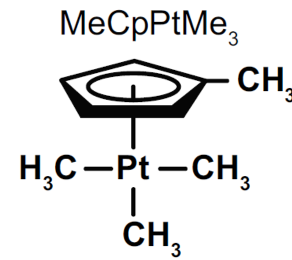


Givre en surface



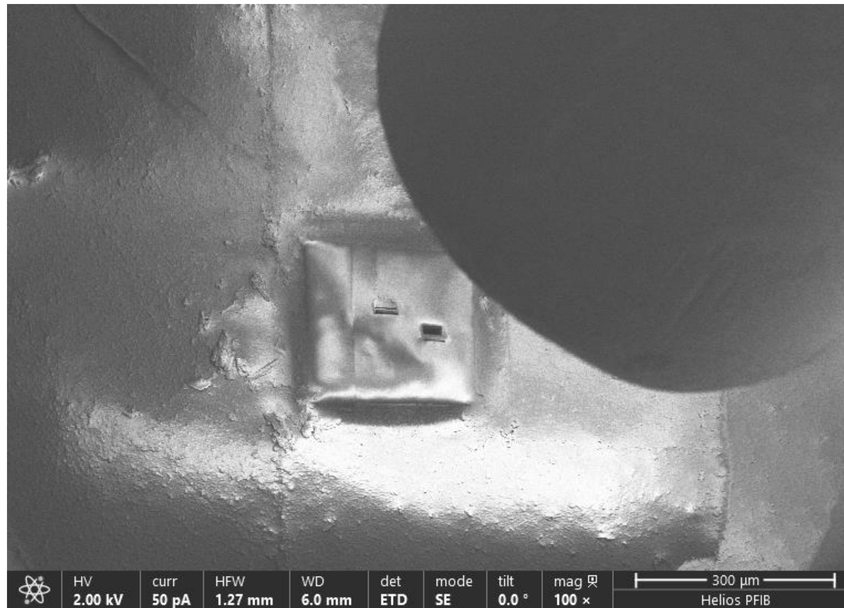
Sublimation du givre sous faisceau ionique

Uncontrolled deposition of Pt GIS

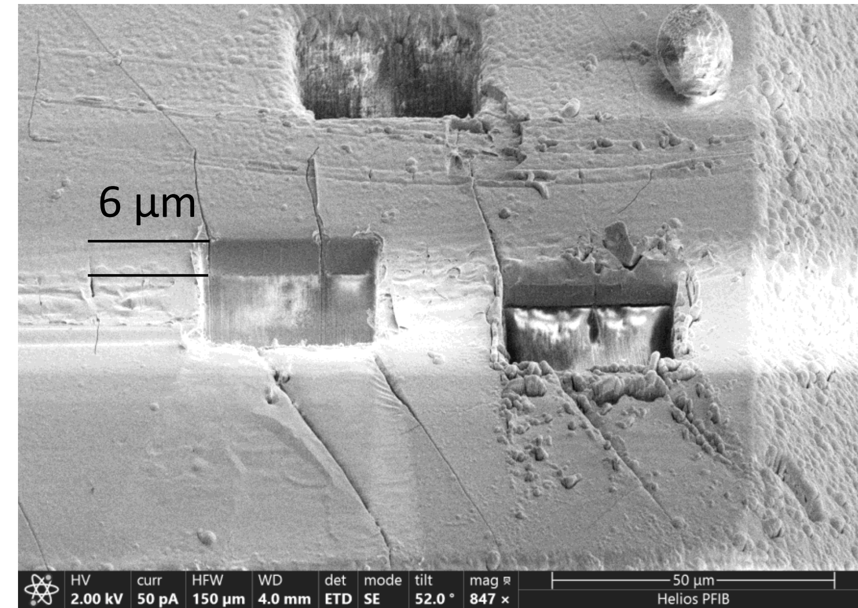


- Faster deposition at cryo temperatures : condensed Pt Layer, the thermal gradient between the deposition gas and the cold specimen surface drives the deposition process,
- **Nozzle – sample distance of several mm (WD 6 mm),**
- For 1 minute deposition time at a GIS temperature of 60°C, 1-µm thicknesses of Pt were achieved,
- Modification of Pt deposit layer using 30kV ion beam rectangular scanning.

Dépôt 3 min - Pt-GIS 2% WD 6 mm

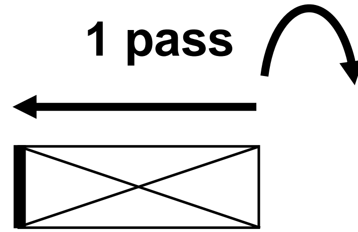


+ Dépôt 3 min = 6 min au total



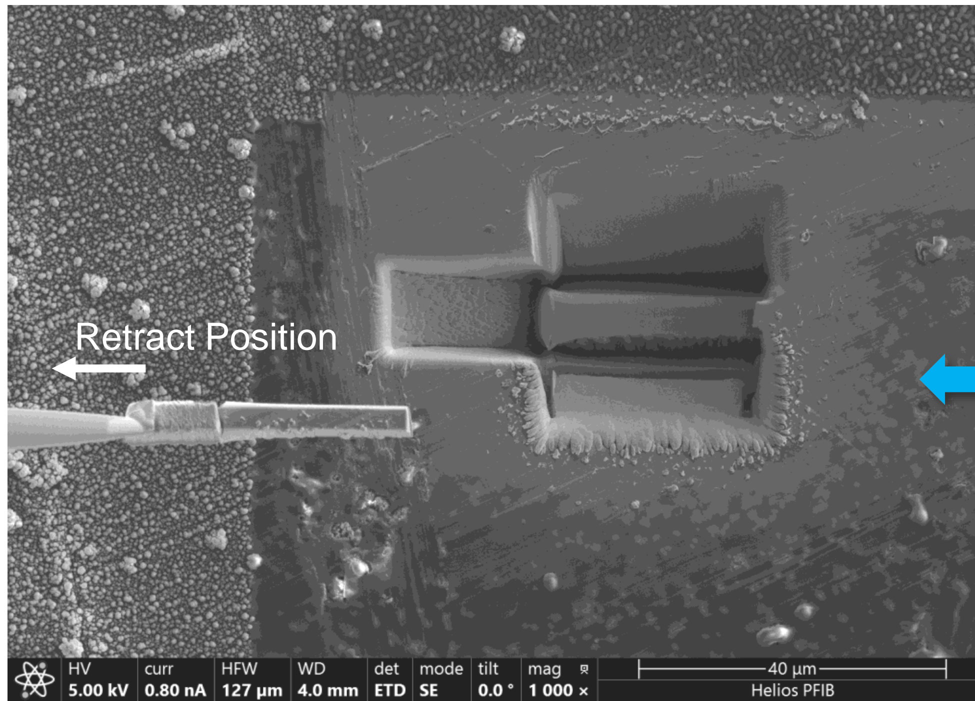
Procédure de Lift-out : redeposition

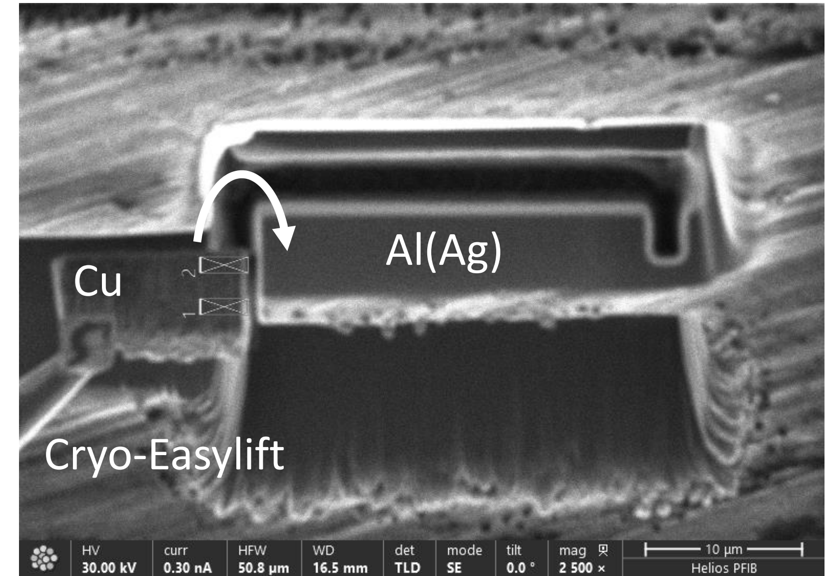
Single-pass milled rectangle
Ion beam redeposition cuts

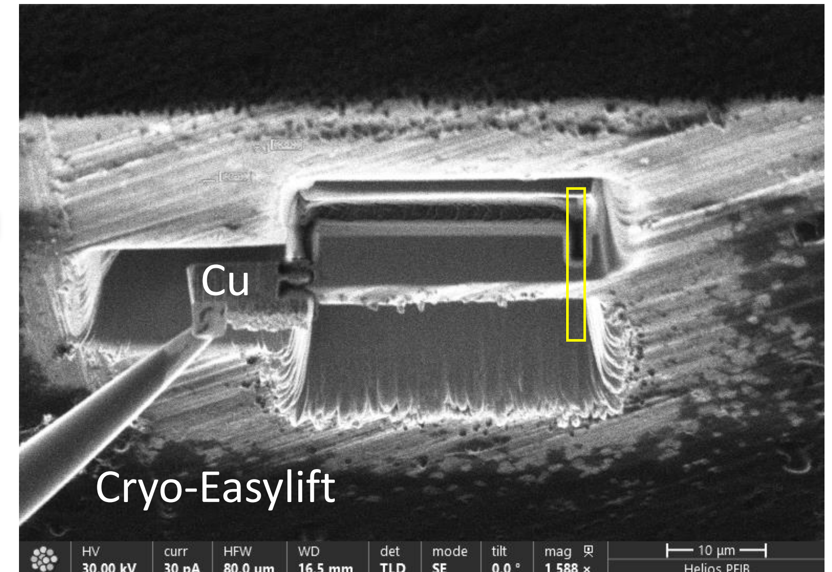


Lift- Out @ cryo temp

Tilt 0°

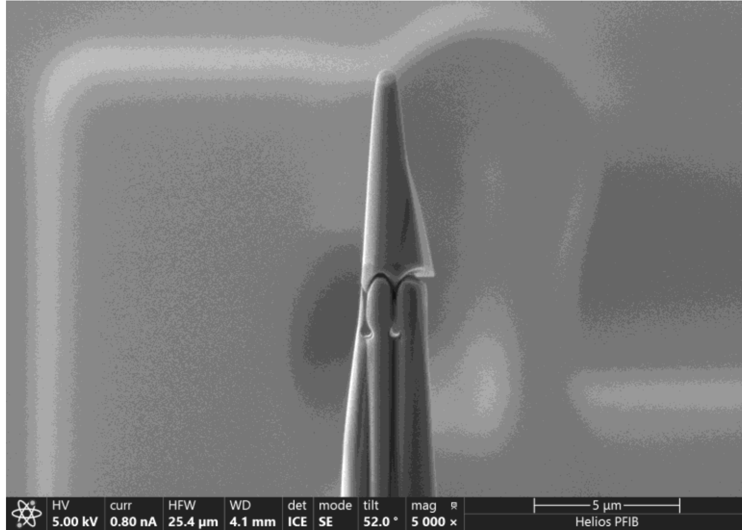




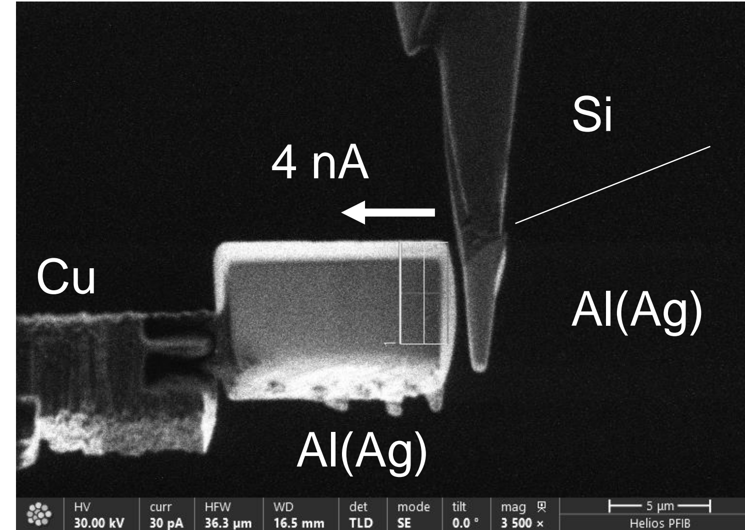


Renforcement @ cryo temp

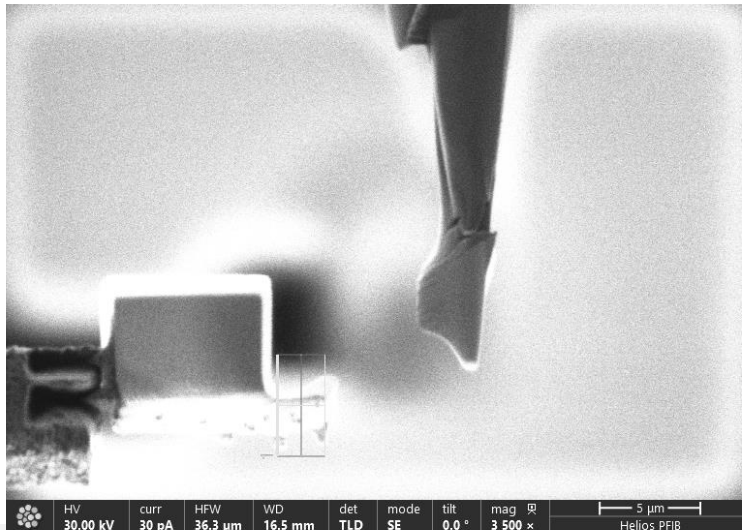
Pré – Annular Milling



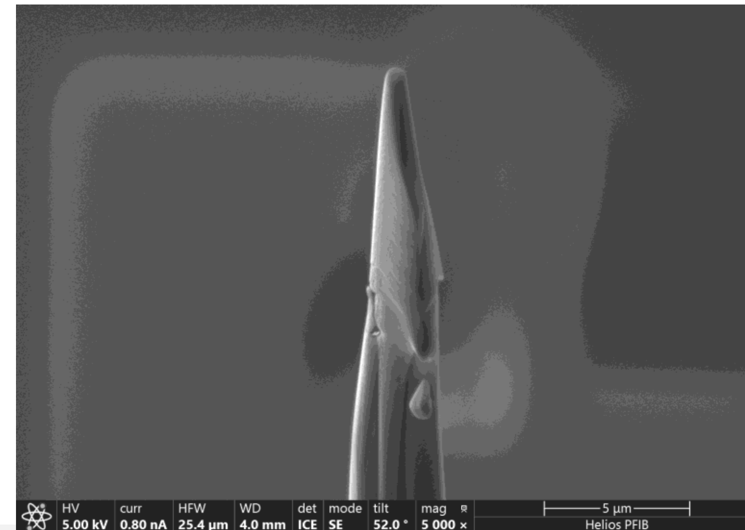
Rotation stage 90° + approche Easylift Cryo



Single-pass milled rectangle @ 4nA

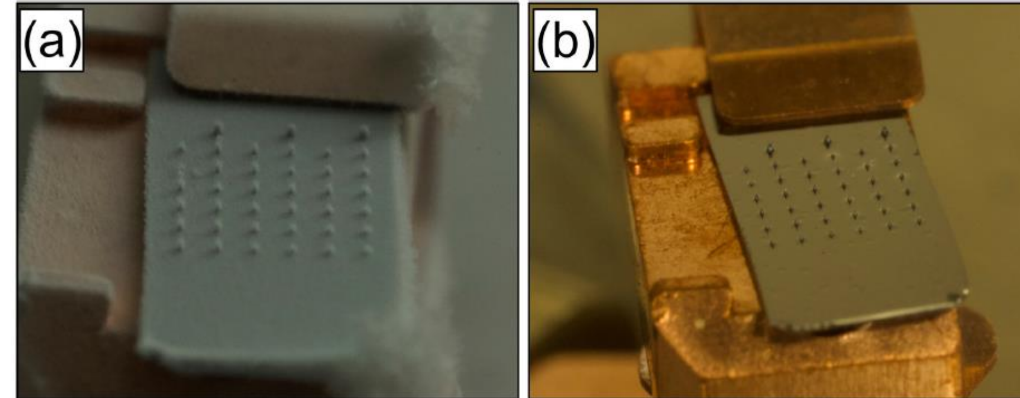


Rotation stage -90° pour amincissement final

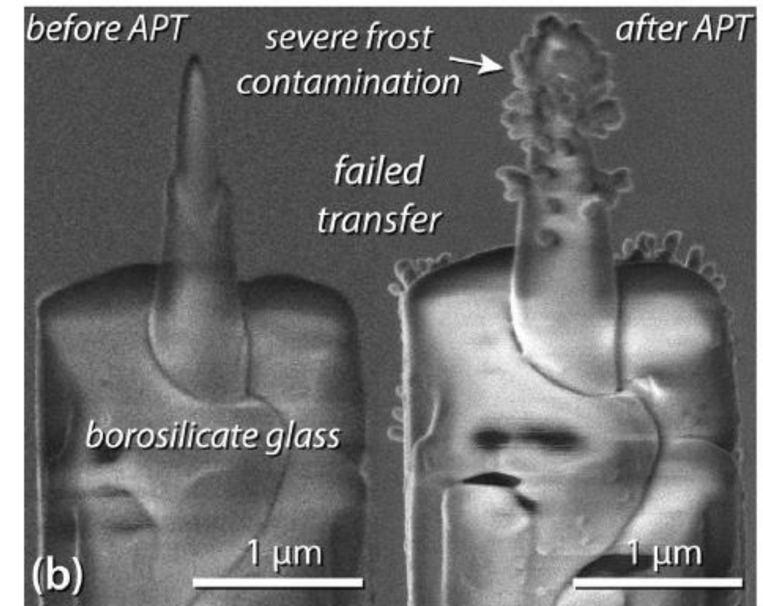


Points de vigilance

Leigh T. Stephenson et al. PLOS ONE
https://doi.org/10.1371/journal.pone.0209211



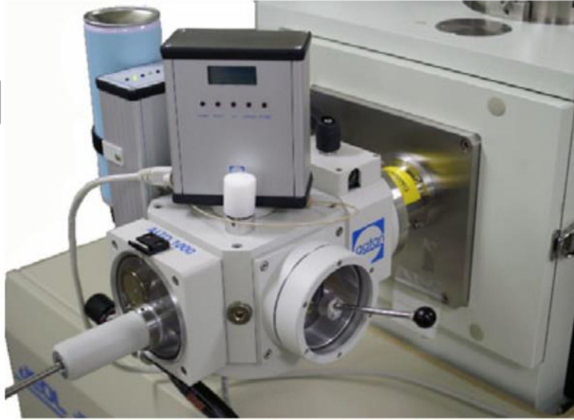
PEREA et al. - Ultramicroscopy 194 (2018) 89–99

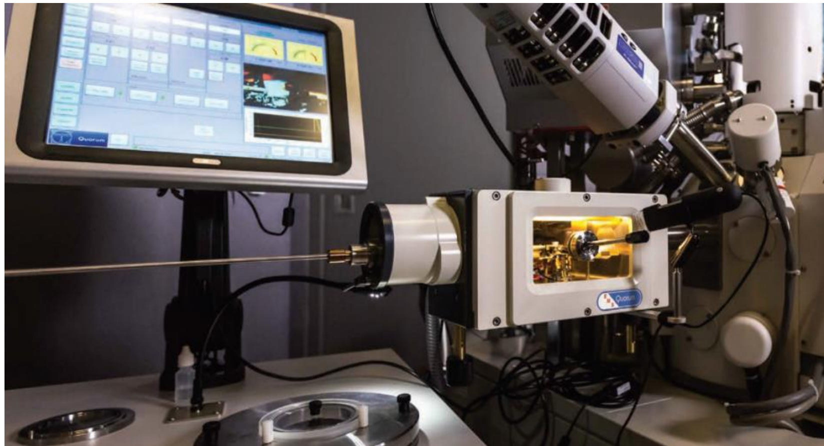


- Keep a dry environment around the sample,
- Maintains high vacuum < 10^{-6} mbar,
- Maintains -160 °C during transfer,
- Short transfer times into SEM / APT,
- Avoid frost formation !!!

GN MEBA

Système de transfert





Procédure de transfert

UHV/CRYO

Atom probe loadlock Chamber

UHV Booster

VCTM Gate Valve

LN2 dewar for suitcase

Wobblestick transfer arm

Puck storage chamber

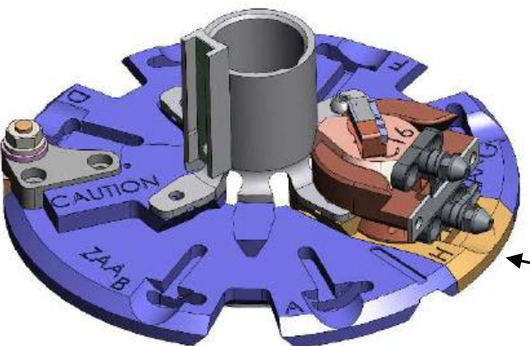
Battery-operated ion pump with NEG

Roughing pump

Cryo carousel

Préparation Cryo des échantillons
Valise de transfert connectée au FIB

"Cryo carrier puck (Cu)"
refroidi @ 30K dans la chambre d'analyse



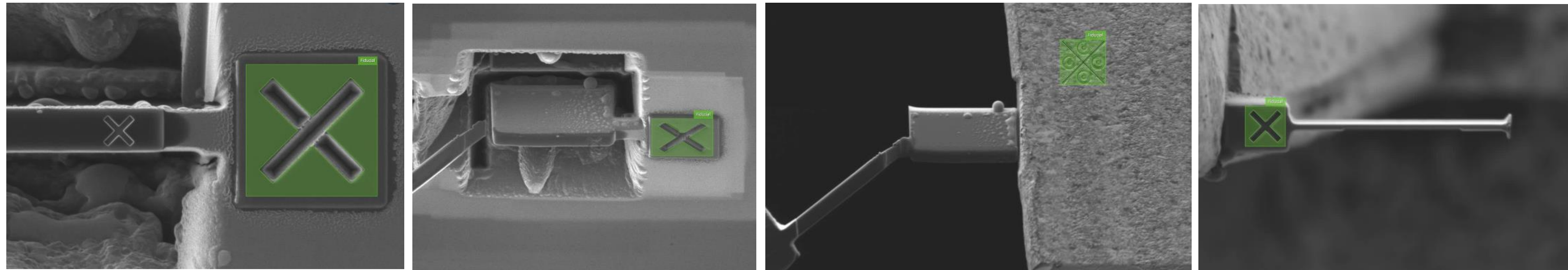
PEEK

Cryo puck

Cryo carrier puck (Cu)

En conclusion

- Quelques pistes de compréhension sur les paramètres qui influencent le comportement d'un matériau sous faisceau d'ions focalisés,
- L'utilisateur dispose d'outils pour découper, graver, usiner, déposer,… et de paramètres tension, courant, dwell time, dose (temps, surface,…), tilt Stage, mode de balayage (redéposition) pour adapter le comportement de chaque matériau sous le faisceau,
- Savoir-faire à acquérir pour chaque "classe" de matériau,
- Un faisceau d'ions endommage toujours l'échantillon : baisser la tension d'accélération, changer la nature de l'ion, réduire l'échauffement,…choisir une autre solution…
- Aujourd'hui, procédures d'alignement du faisceau FIB automatisées et des procédures de préparation d'échantillons (lames minces) "full automated".



GN MEBA