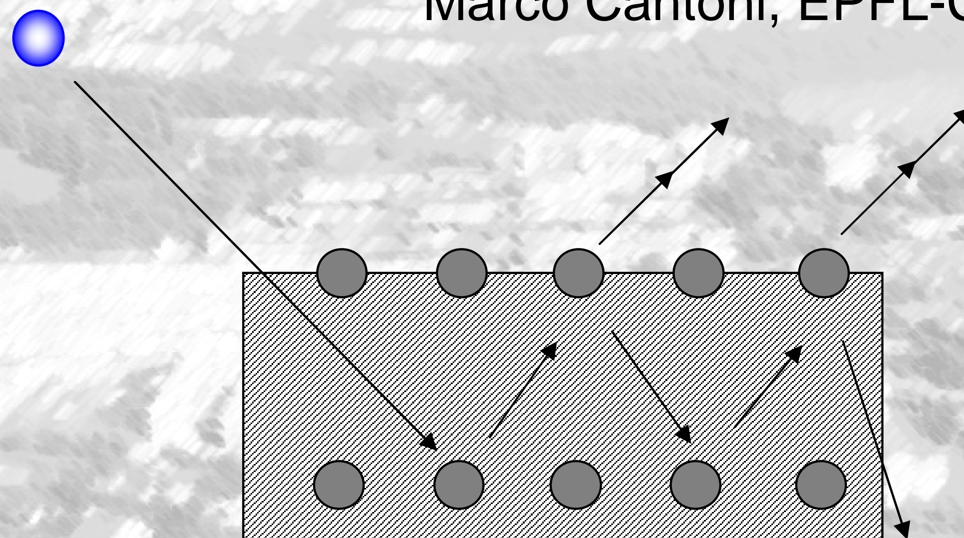
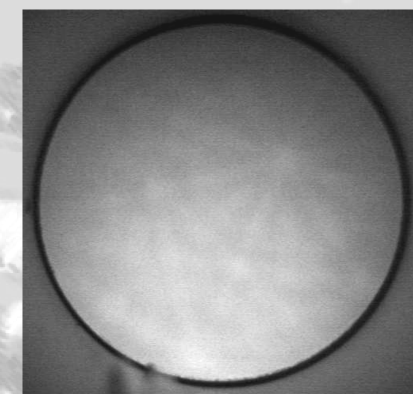
CENTRE INTERDISCIPLINAIRE
DE MICROSCOPIE ELECTRONIQUE

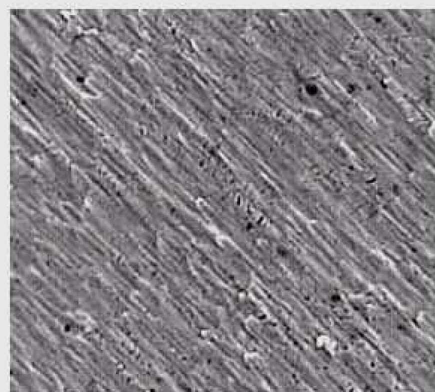
ÉCOLE POLYTECHNIQUE FÉDÉRALE DE LAUSANNE

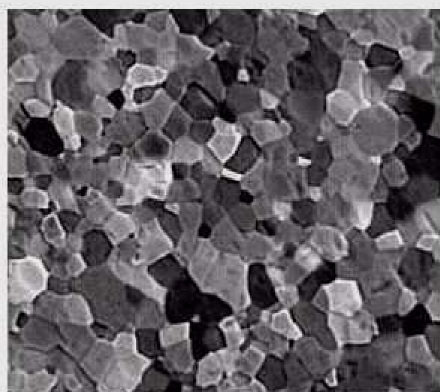
Polissage Ionique

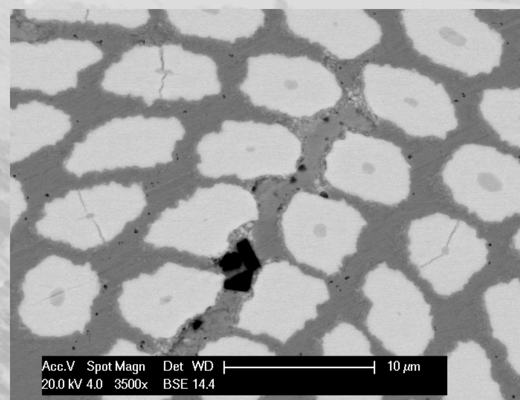
Marco Cantoni, EPFL-CIME

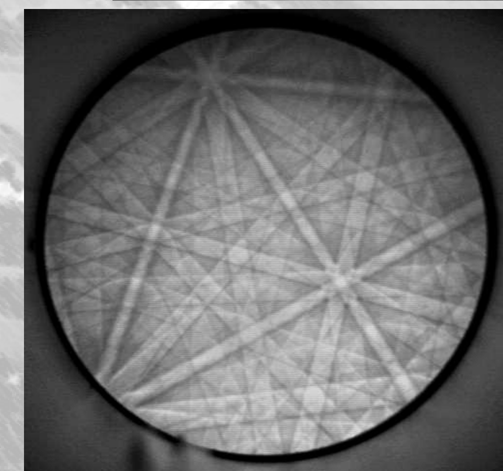












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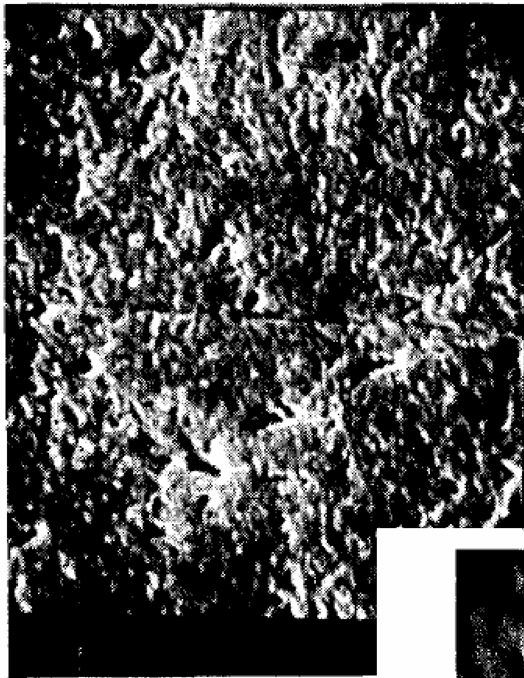
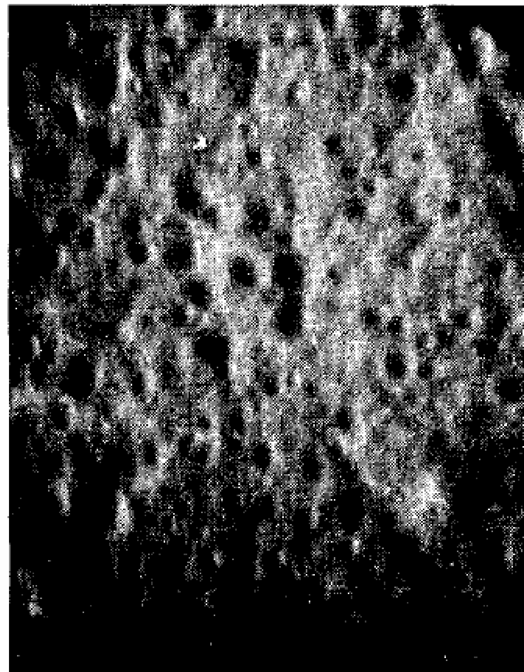


(a) α = 0.915%



(d) α = 0.762%

polissage
ionique
avec Xe

Ion polishing of copper: some observations

R. A. Hoffman, W. J. Lange, and W. J. Choyke

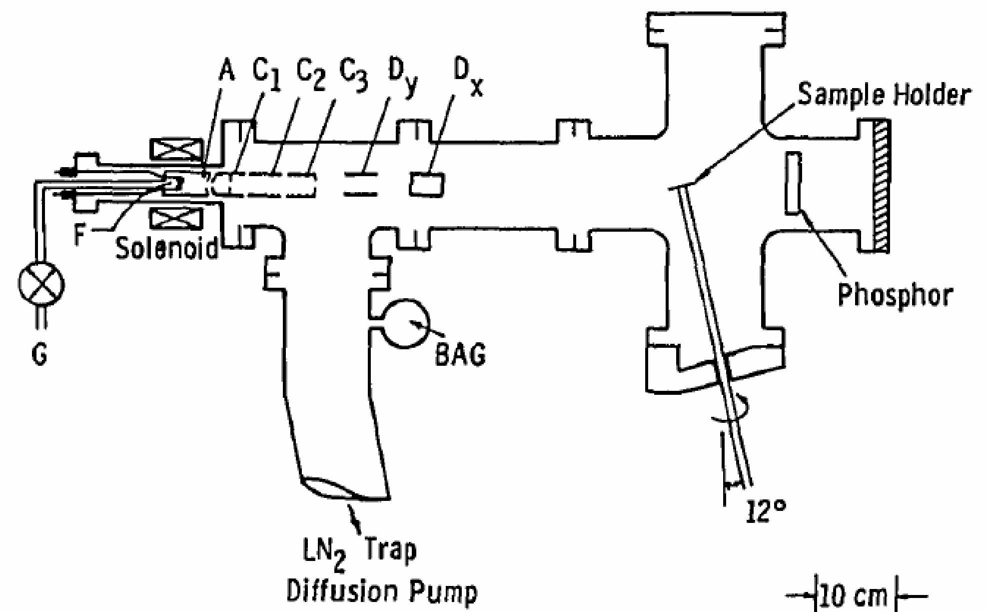
August 1975 / Vol. 14, No. 8 / APPLIED OPTICS 1803

I. Introduction

Mirrors for high power laser application must of necessity exhibit very low optical absorptivity at the appropriate wavelength(s). The mirrors must have the proper optical figure, be smooth to minimize light scatter, and have high thermal conductivity to prevent surface damage and undesirable mechanical stresses. To obtain near-theoretical absorptivity the surface must also be smooth and damage free.[1] Certain metals that have the appropriate properties, such as copper and molybdenum, are candidates for laser mirror substrates. However, conventional polishing of metals usually damages the surface being polished; the depth of the damage depends upon the hardness of the material and the hardness and size of the polishing grit used. In addition, polishing grit may be imbedded in or just below the surface. The

Ions are extracted by a 6-kV potential drop across a 1.25-mm gap into the first element of a three-member, 2.54-cm diam, cylindrical, decelerating electrostatic lens whose resultant focal length is ≈45 cm. Aperture stops, 6.25-mm diam, located in the first lens cylinder and on the exit end of the third assure an ion beam of low divergence. Two orthogonal sets of deflection plates permit the ion beam to be positioned and rastered on the sample by application of suitable dc and ac potentials (~100 V). A phosphor screen, mounted on a movable probe, permits convenient alignment and focusing of the beam. Thus the ion spot and raster can be visually positioned on the sample and any spillover observed. The ion spot is approximately 3 mm in diameter for a 5-μA beam. Attempts to obtain higher currents without increasing the spot size by using electrons to cancel space charge in the ion beam were unsuccessful.

The ion beam is incident, typically with 4 keV, on the sample, which is inclined at an angle of 12° to the beam. Using Xe ions under these conditions the penetration depth of ions into the sample is estimated from LSS theory[2] to be ~10 Å. Other useful features incorporated in the apparatus include provision for thermal evaporation and thickness monitoring to enable *in situ* deposition of films on samples and an electron source to provide charge neutrality when working with insulating samples.



« low kV »: 6kV (4kV)

Balayage: FIB !
Spot: 3mm

Source d’électrons:
neutralisation des charges

V. Conclusions

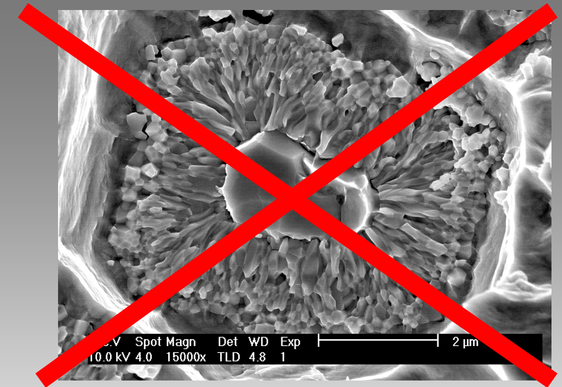
It is shown that ion polishing of single crystal copper samples is capable of removing damage layers of material and contamination from surfaces that had been prepared by other polishing methods, and that the resulting surfaces are smoother than those produced by the initial polishing. Furthermore, the combination of ion polishing and 450°C vacuum annealing yields values of absorptivity that are significantly lower than those before ion polishing.

Aujourd'hui:

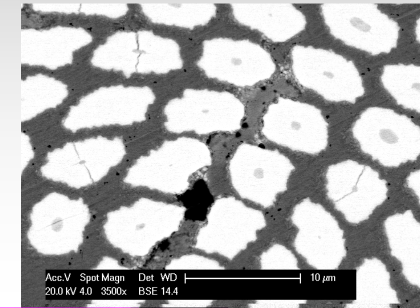
BIB: Broad Ion Beam: faisceau « large », 1-5mm (200V-10kV)

FIB: Focused Ion Beam: faisceau focalisé, >4nm (30kV – 500V), souvent combiné avec un MEB

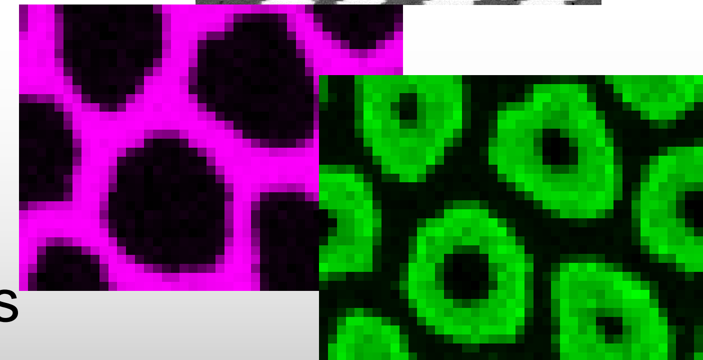
Polir un échantillon:
adieu, les électrons secondaires….
profondeur de champs, contraste de topographie, résolution…



- Imagerie avec des électrons rétrodiffusés, contraste de masse, channeling

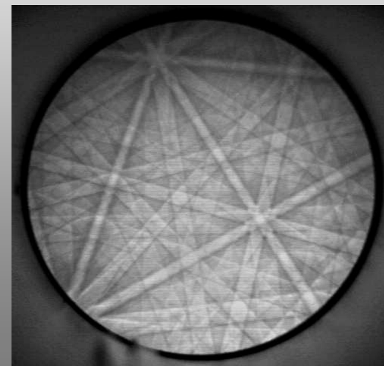


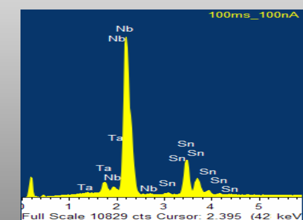
- Méthodes de spectroscopie:
  EDX, WDX, Auger….
  Corrections ZAF, PROZA, XPP etc.



- EBSD, diffraction des électrons retrodiffusés

Préparation de l'échantillon:
techniques de polissage mécanique et électrochimique élaborées





Quand le polissage mécanique échoue…

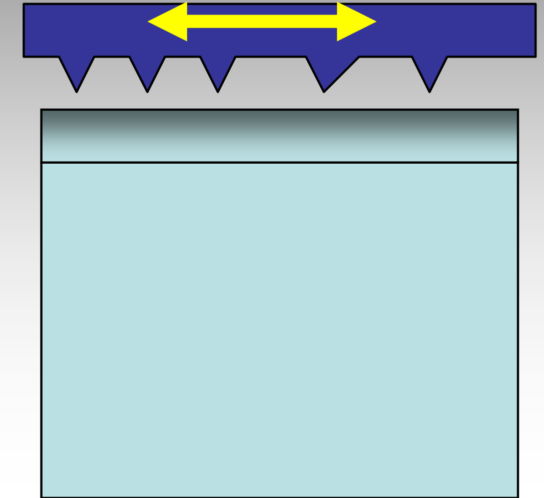
Métaux doux: Au, Al, Cu, Pb etc.

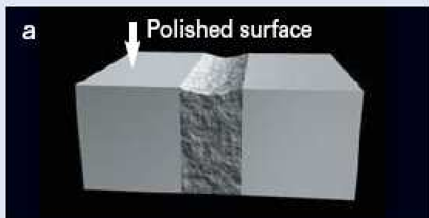
Matériaux durs: Céramiques, Carbides, Composites

Semi-conducteurs (circuits imprimés)

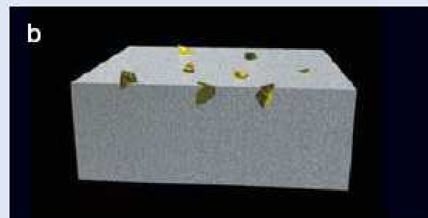
Polymères, matériaux mous

Matériaux hétérogènes, poreux

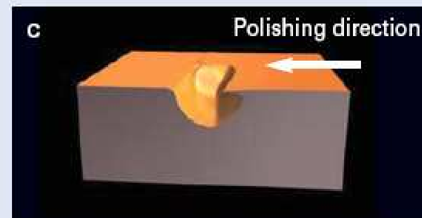




a) On material with different hardnesses, the polished surface becomes uneven as the soft portion is cut faster and easier.



b) On soft material, pieces of hard polisher can be buried in the material being polished.



c) On soft material with a void, the material around the void stretches and deforms.

d) On material with a void, pieces of polisher get stuck in the void.

e) On metals, due to distortion caused by mechanical polishing on the polished surface, information about the crystal becomes difficult to obtain.

zone endommagée,
distorsion,
grains arrachés,
rugosité de surface,
remplissage de pores
etc.

Cable supraconducteur, multi-filaments Nb_3Sn

Superconducting Nb_3Sn cables for high magnetic fields 10-20T:

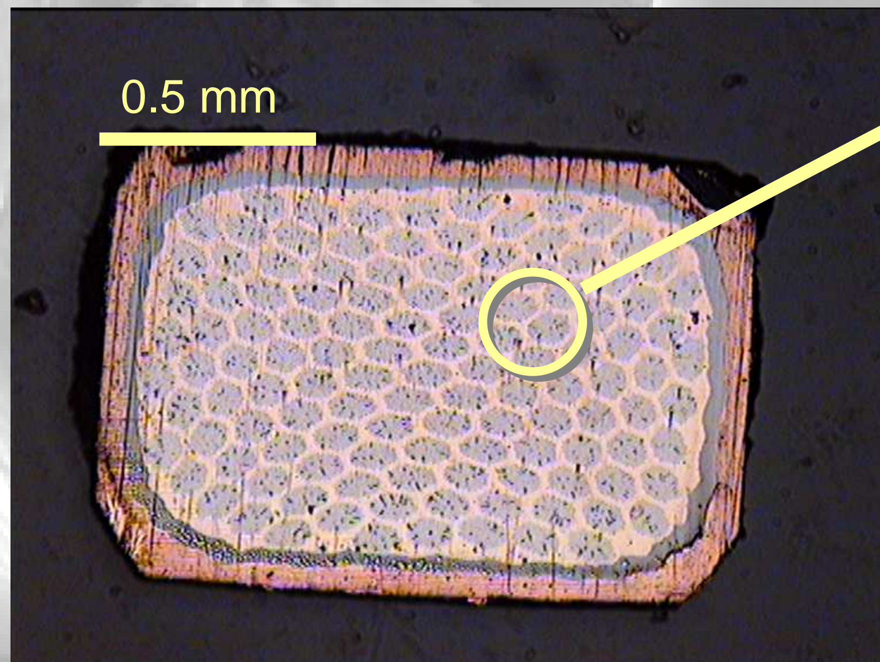
increase current density, lower cost

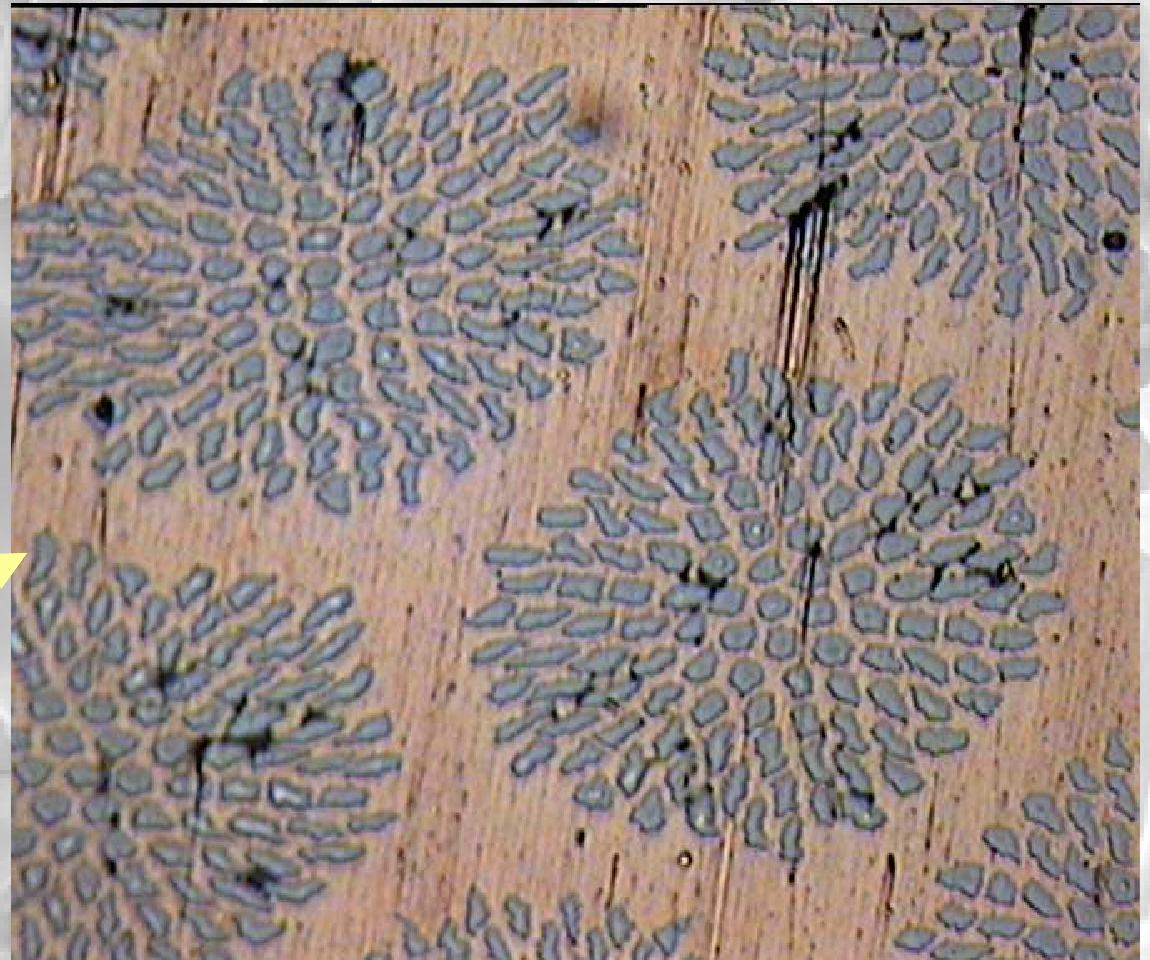
Potential Applications:

NMR, Tokamak fusion reactors

Large Hadron Collider (LHC), CERN

Typical cable:
1 x 1.5mm cross-section
121x121 filaments of Nb_3Sn
in a bronze (Cu/Sn) matrix





Prof. R. Flükiger, V. Abächerli, D. Uglietti, B. Seeber
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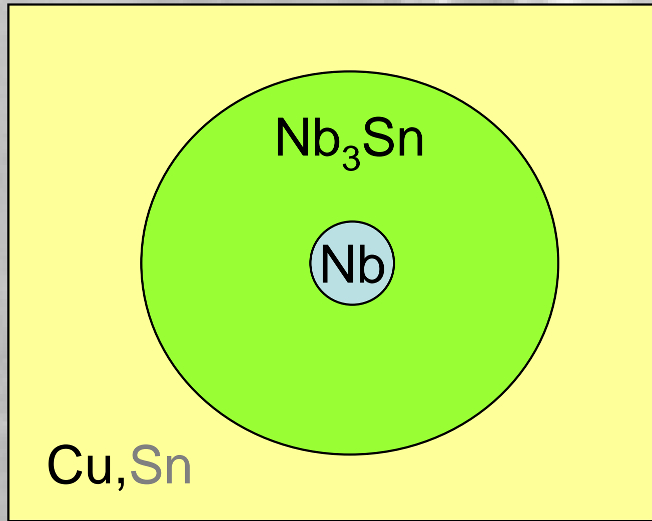
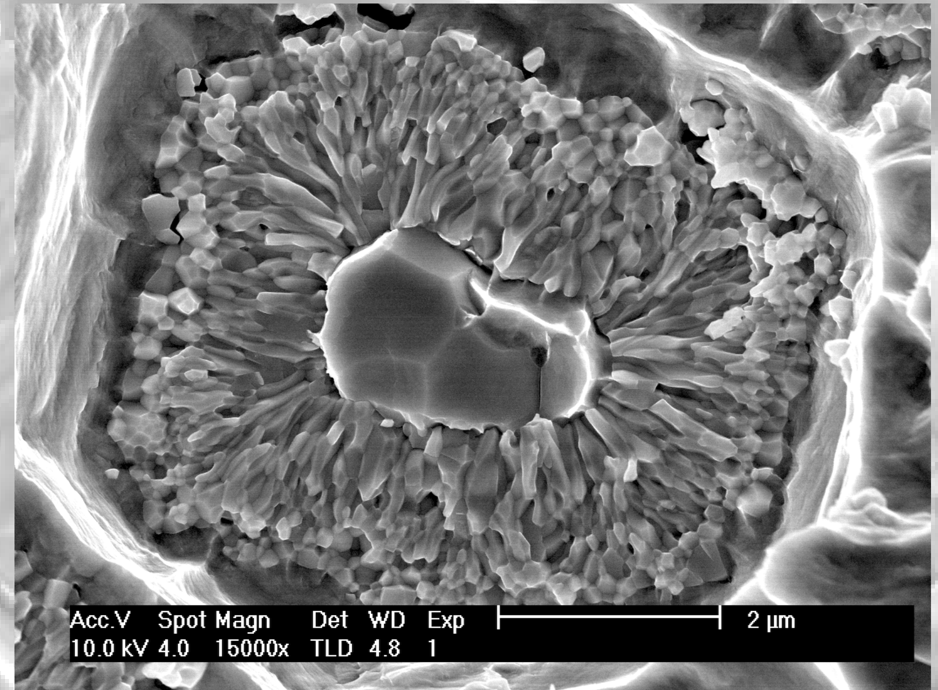
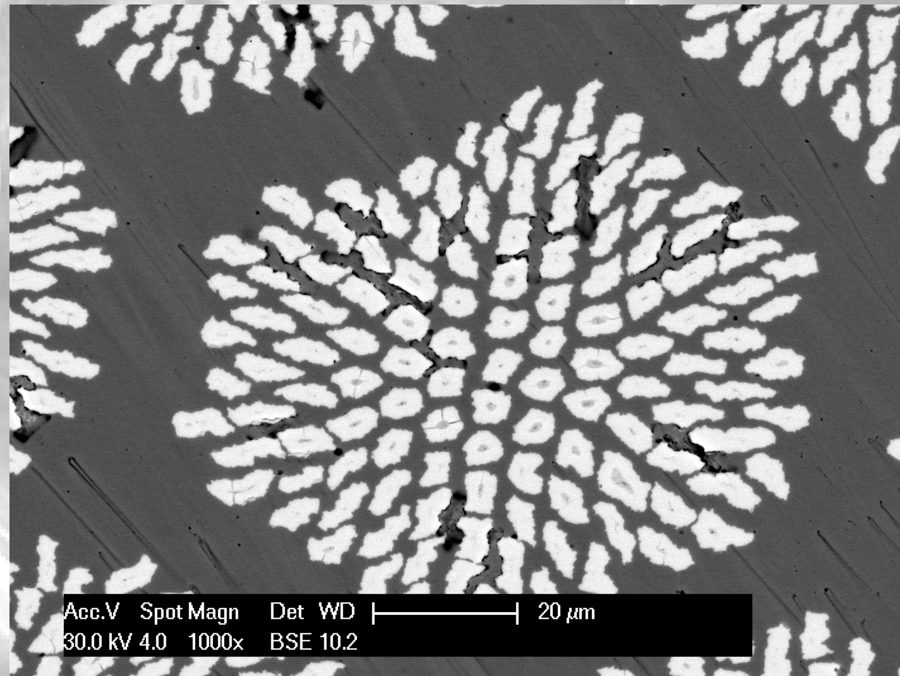
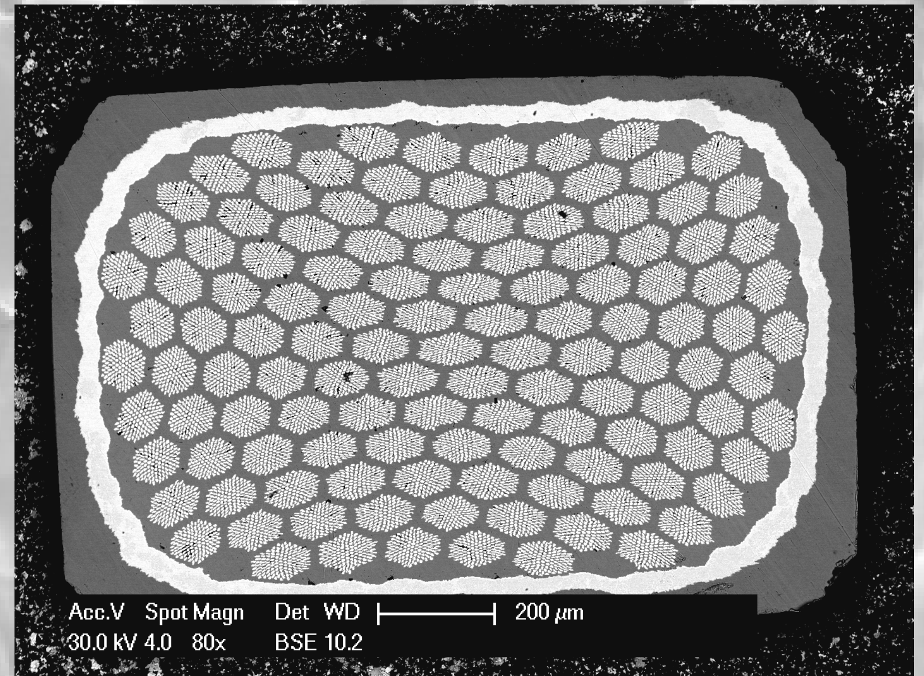
University of Geneva

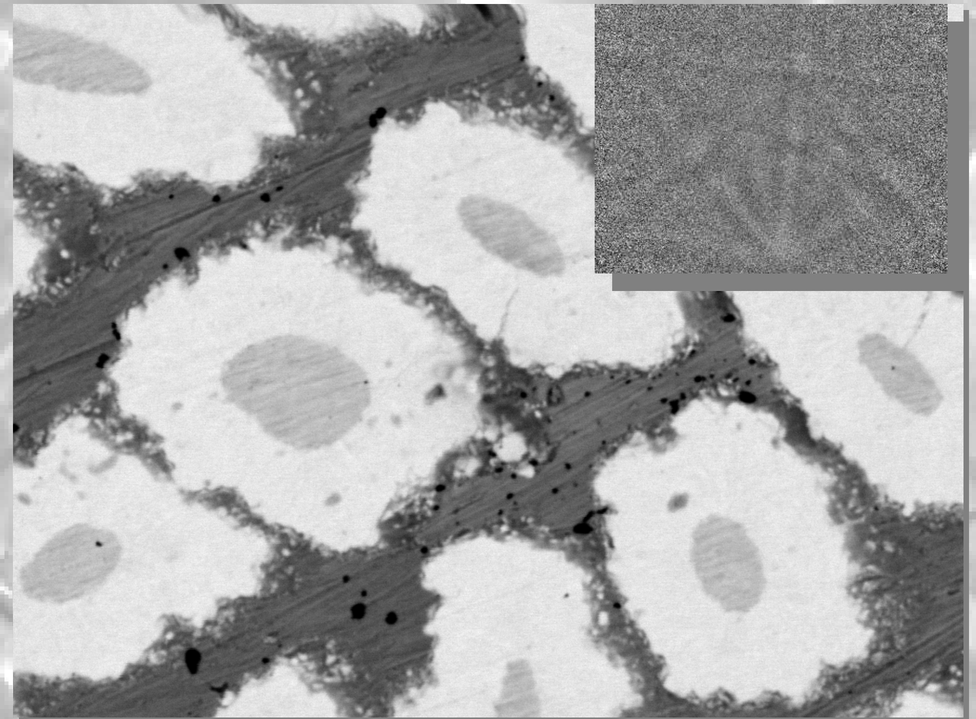

Nb_3Sn
Nb
Cu,Sn


Acc.V Spot Magn Det WD Exp
10.0 kV 4.0 15000x TLD 4.8 1
2 µm

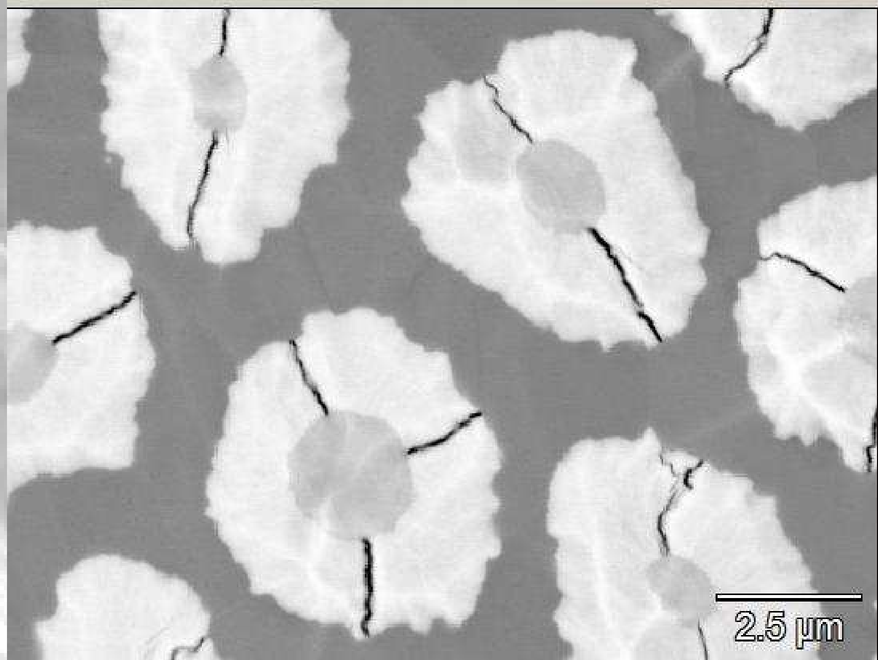

Acc.V Spot Magn Det WD
30.0 kV 4.0 1000x BSE 10.2
20 µm

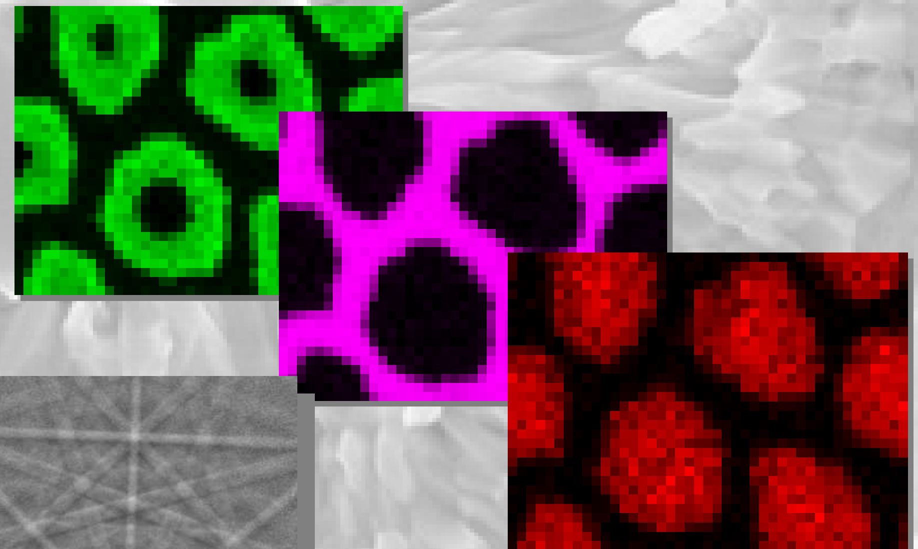

Acc.V Spot Magn Det WD
30.0 kV 4.0 80x BSE 10.2
200 µm

Grains de Nb3Sn incrusté dans la matrice de Cu.
Trace de polissage
Filaments déplacés dans la matrice
Ecrouissage empèche la formation des patterns de lignes de Kikuchi (EBSD)



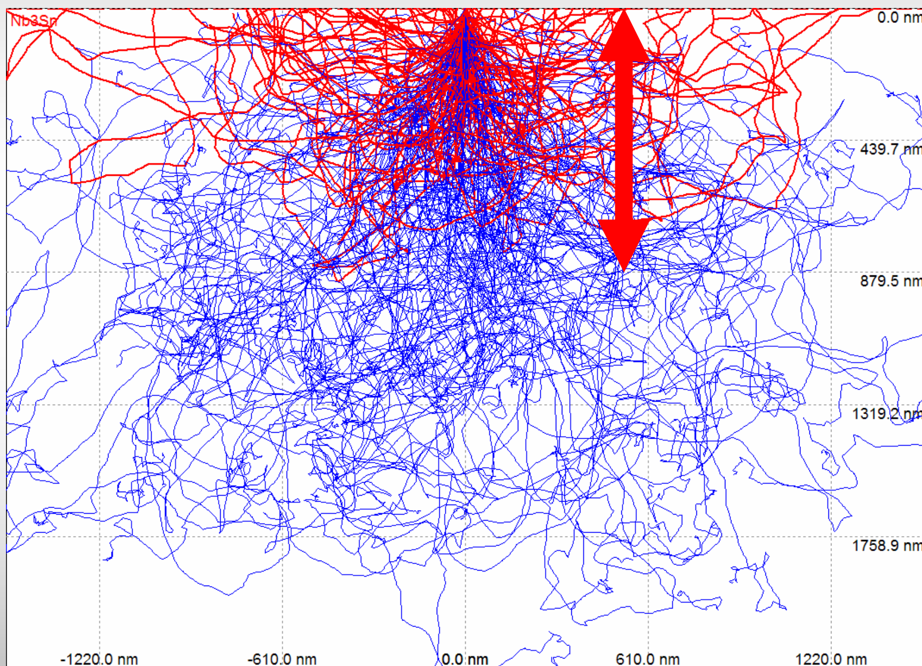
Après bombardement ionique (pour echantillon TEM, Gatan PIPS)

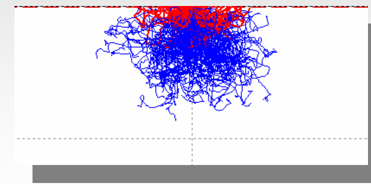


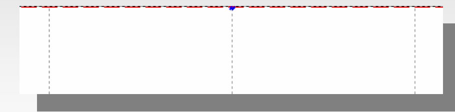


But du polissage (décapage) finale:

Enlever la couche endommagée par la préparation mécanique
et réduire (minimaliser) les artefacts de la préparation ionique…







Nb_3Sn
Profondeur max. électrons rétrodiffusés

30kV: 800nm
15kV: 300nm
1kV: 5nm

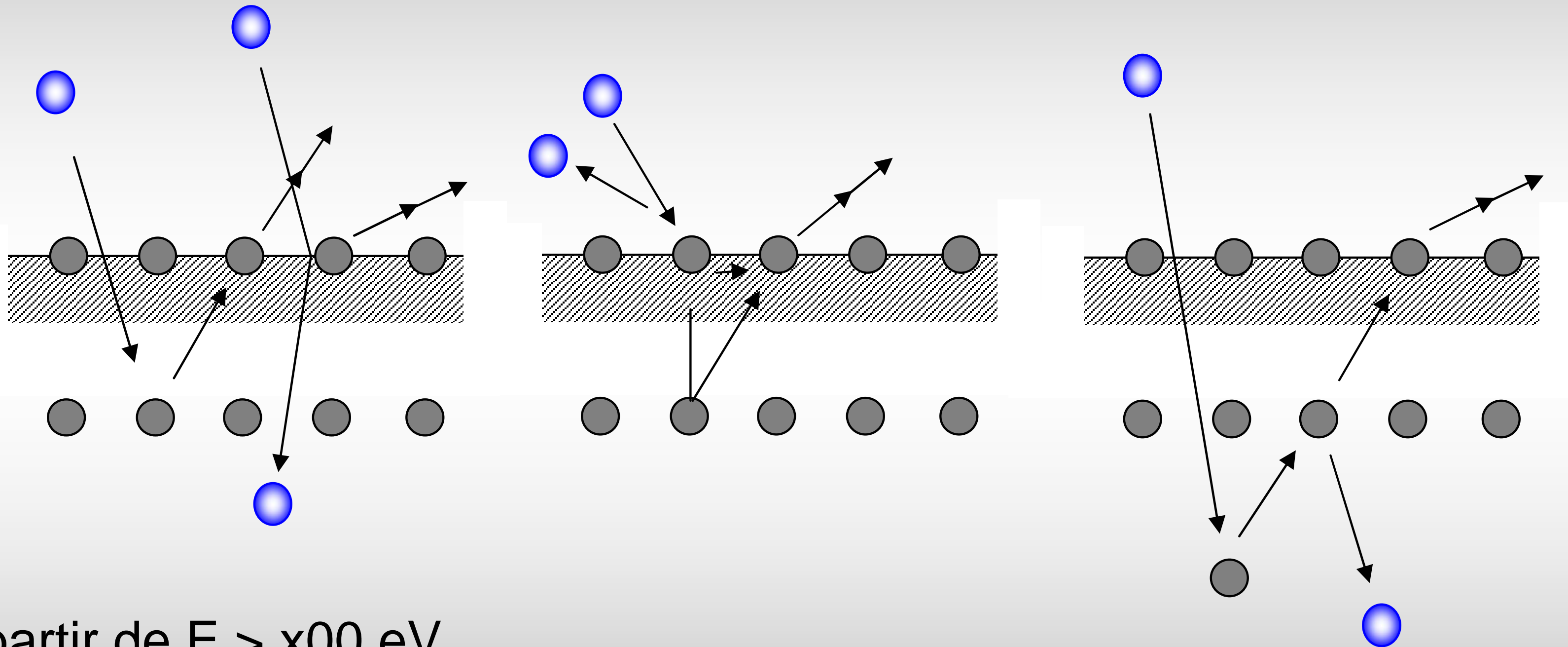
Critère: volume d'interactions

Monte-Carlo Simulation CASINO v2.42

Interaction ion <-> matière

attaque, polissage

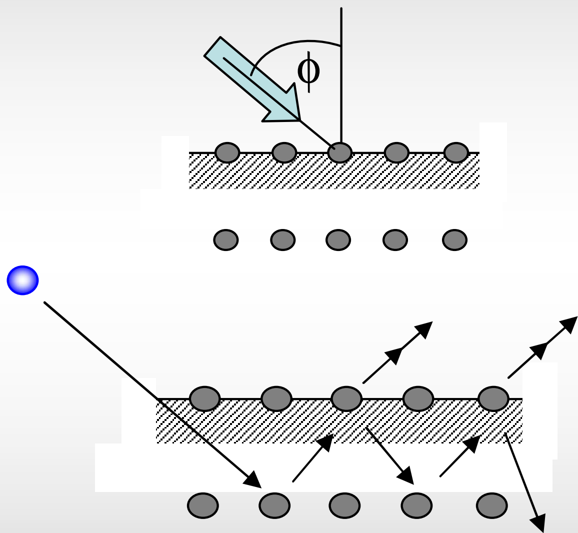
- Attaque physique: éjections d' atomes de la « cible » par des cascades de collisions



- À partir de E > x00 eV
- Energie des ions: 200V – 30kV
- Origine des atoms ejectés: premières mono-couches atomiques
- Effets non-désirés: implantations, amorphisation, échauffement

Ion-Solid interaction
Sputtering Yield

- Sputtering yield depends on incident angle ϕ



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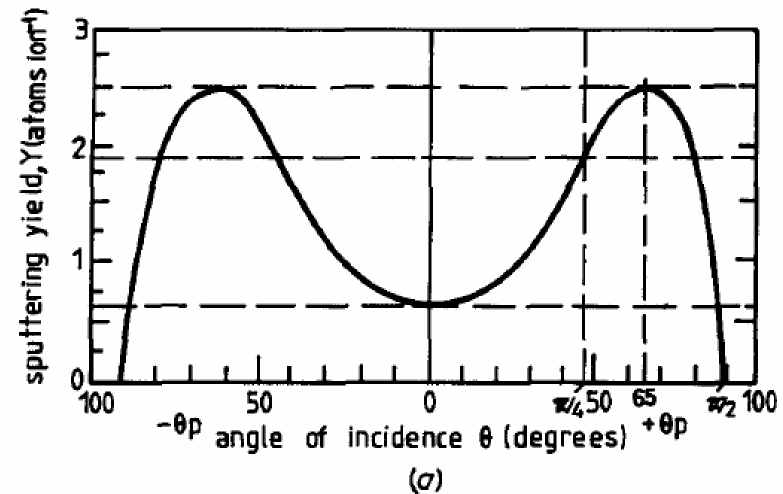
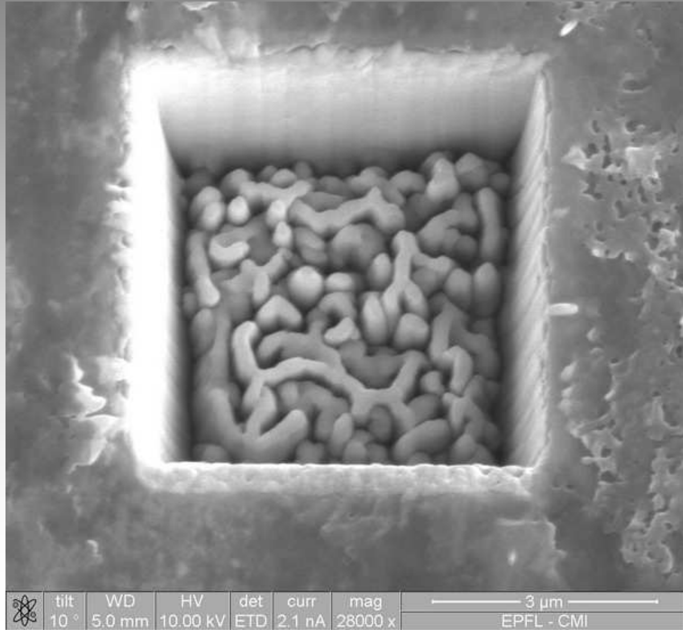
The theory of ion beam polishing and machining

G Carter and M J Nobes, *Department of Electronic and Electrical Engineering, University of Salford, Salford M5 4WT, UK*
and
I V Katardjiev, *Department of Technology, School of Engineering, Uppsala University, Box 534, S-751 21 Uppsala, Sweden*

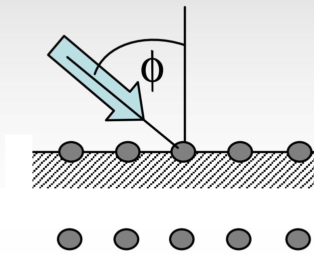


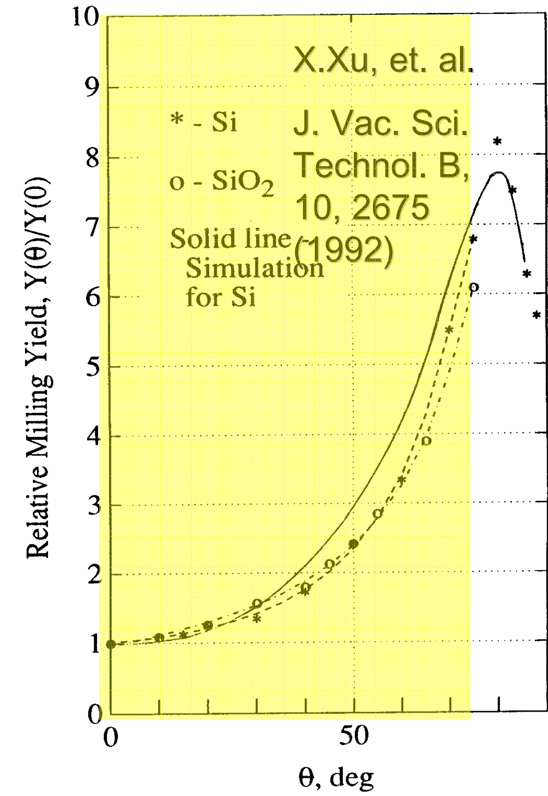
- Higher probability of collision cascades near the surface at higher ϕ
- Sputtering yield has maximum for ϕ = 75°

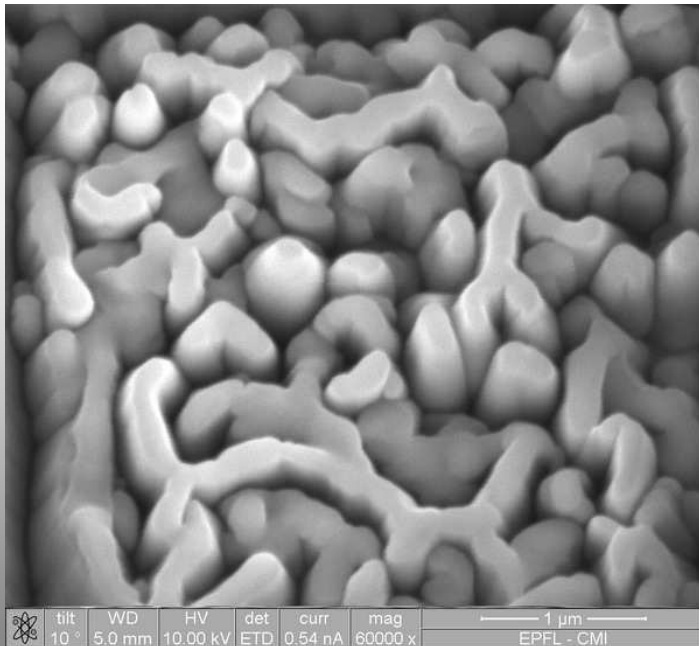
Attaque, usinage

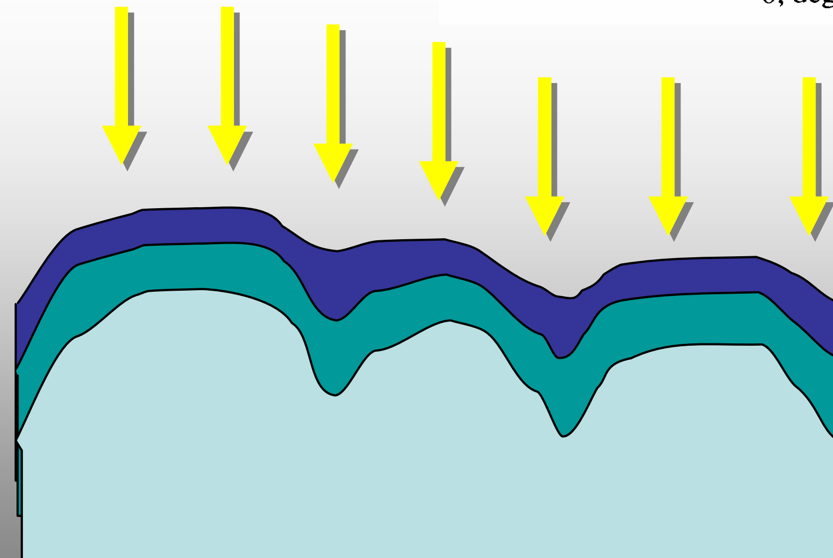


Attaque par FIB, acier

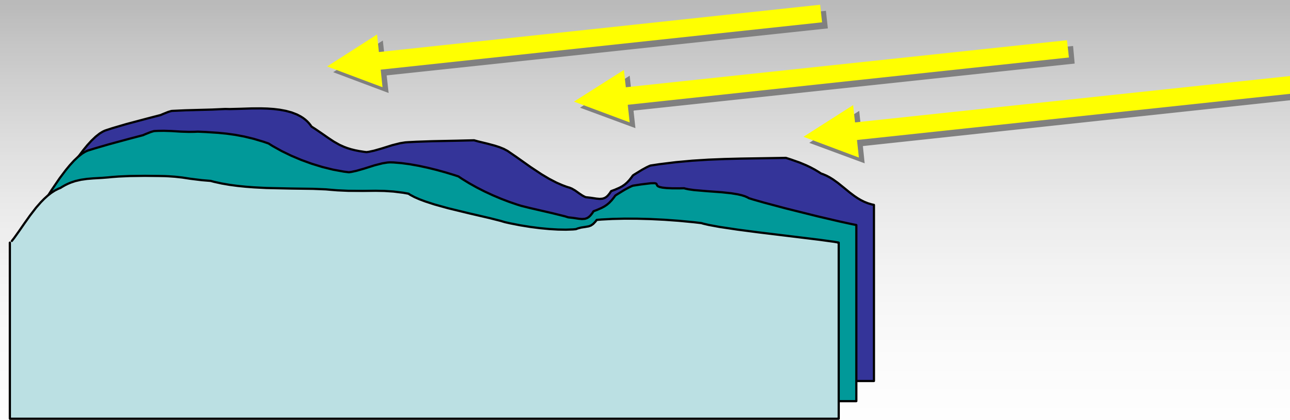


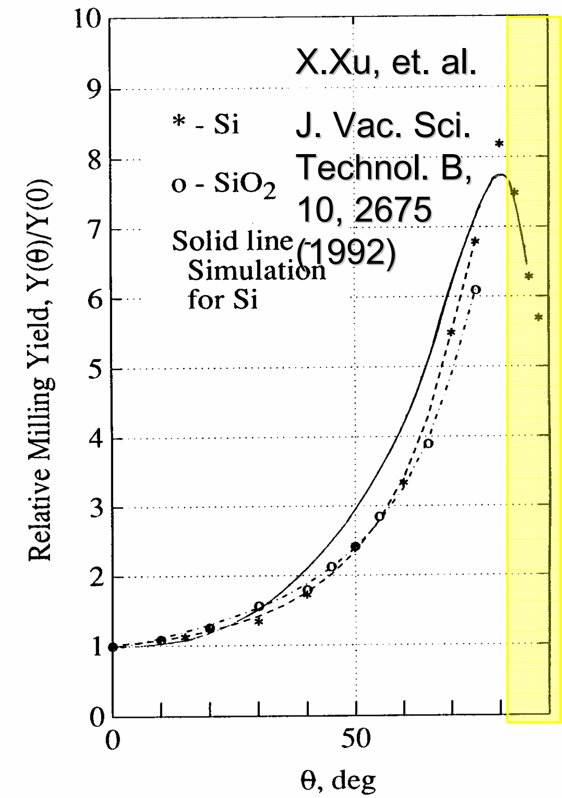






Polissage, angle d'incidence faible





Computer simulation of the topography evolution on ion bombarded surfaces

M. Zier *, W. Hauffe

Department of Physics, Dresden University of Technology, Dresden, Germany

Nuclear Instruments and Methods in Physics Research B 202 (2003) 182–187

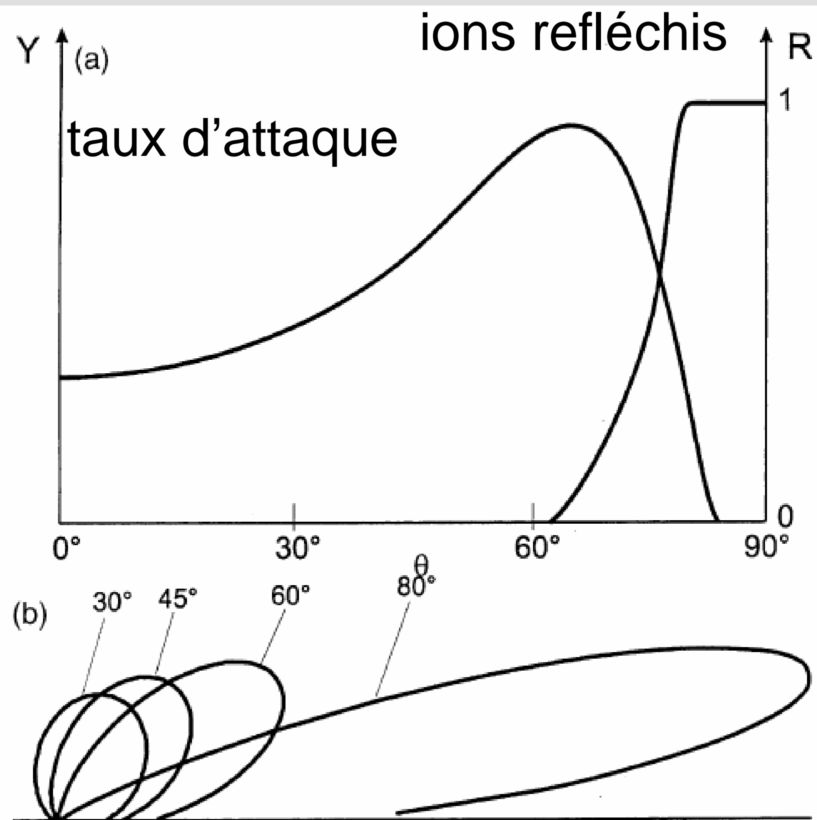
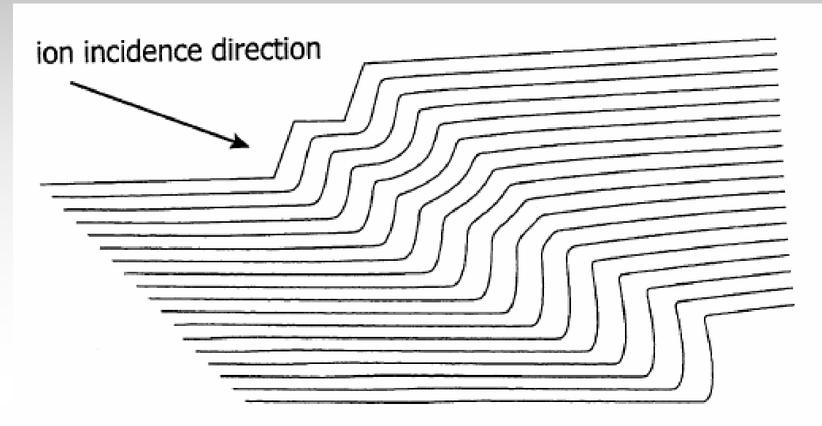


Fig. 2. (a) Typical forms of $Y(\theta)$ and $R(\theta)$. (b) Assumed angular distribution of scattered ions.



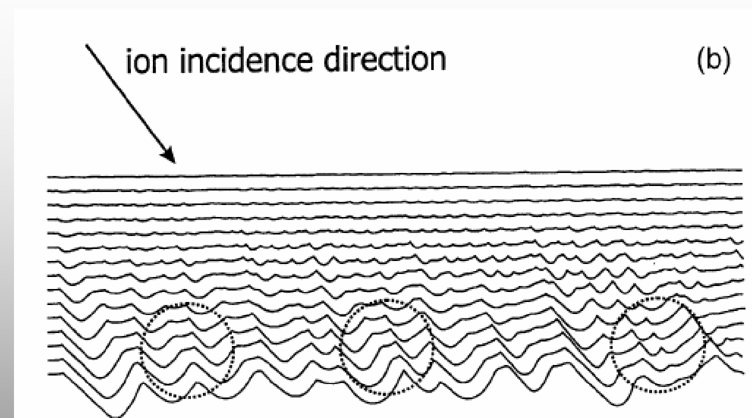


Fig. 5. Simulation of a noisy line profile (a) without ion reflection and redeposition, (b) with ion reflection and redeposition.

polissage ionique avec Ar pour MET

http://temsamprep.in2p3.fr/

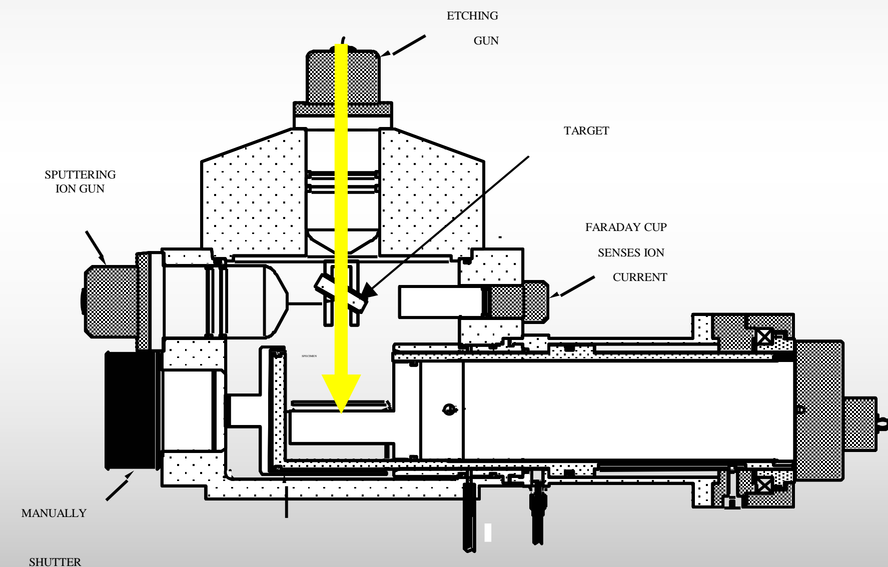
- bien établi
- polissage a faible angle (jusqu'à 0 dégrée)
- très faible tension 4kV, 2kV, x00V

couche amorphe à la surface / épaisseur de l'échantillon
refroidissement possible, rotation de l'échantillon
taux d'attaque faible
zones minces petites, optimisé pour échantillons MET

CENTRE NATIONAL DE LA RECHERCHE SCIENTIFIQUE
IN2P3 | CNRS | Annuaires | sites CNRS | Autres sites
TEM SAM PREP
Microscopie Electronique en Transmission (MET) : guide de préparation des échantillons
guides méthodologiques | techniques | photothèque | lexique | livre | observations | actualité | partenaires | contactez nous | liens

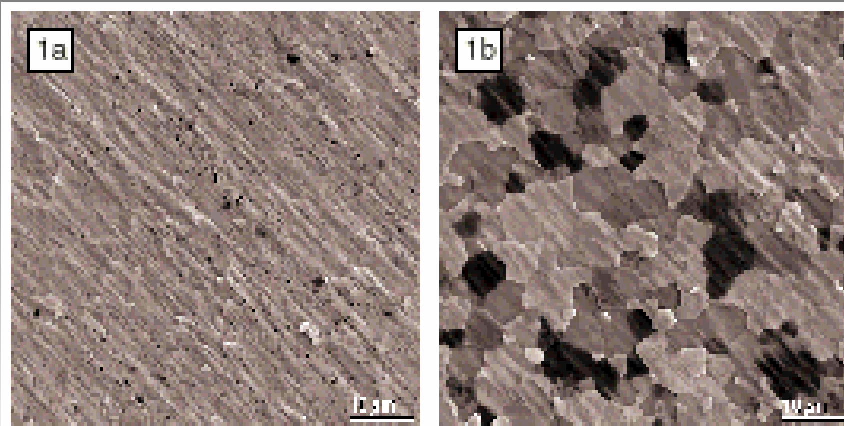
PRECISION ETCHING COATING SYSTEM
GATAN modèle 682 PECS

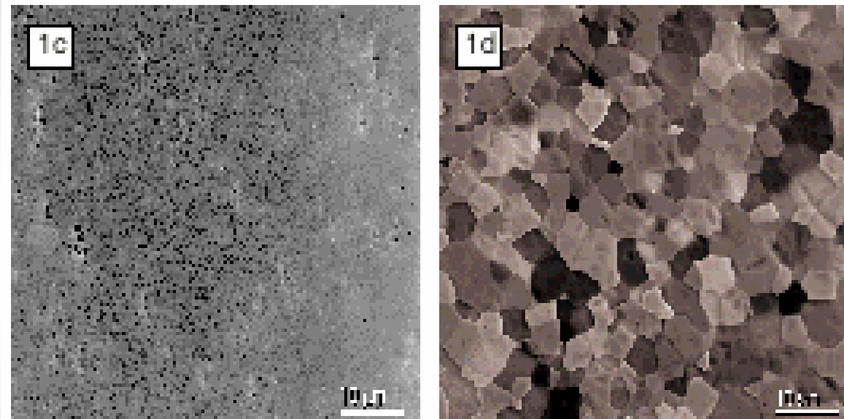
Energie des ions : 1kV à 10 kV
Diamètres Faisceau (FWHM) : Canon décapage ≈ 1 cm
Densité de courant : 10 A/cm² par source (pic maximum)

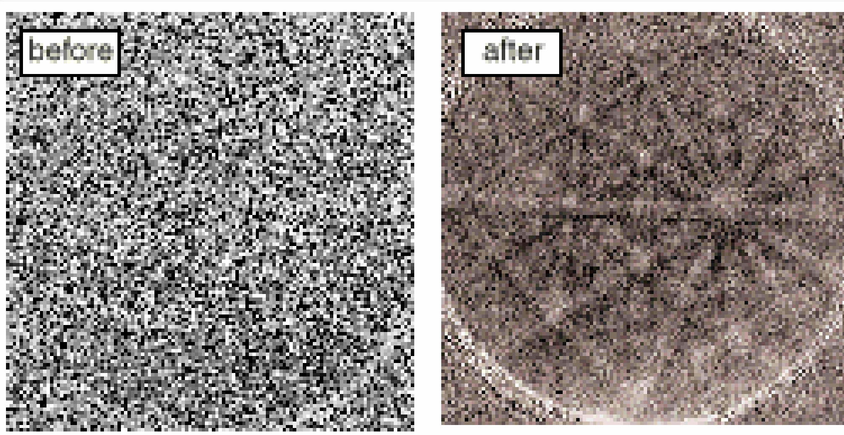


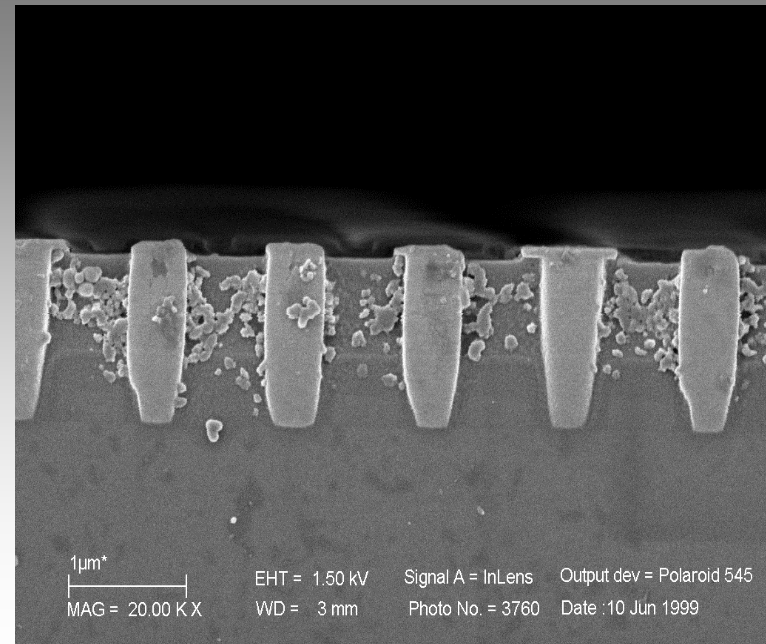
- Le PECS offre les avantages suivants :
- décapage et métallisation dans la même chambre sous vide,
- Métallisation haute résolution avec tout type de dépôt,
- Temps de manipulation substantiellement réduit,
- Contamination de l'échantillon réduite au minimum,
- Introduction de taille d'échantillon élevée,
- Grande facilité d'utilisation.
- Gaz réactive (I)

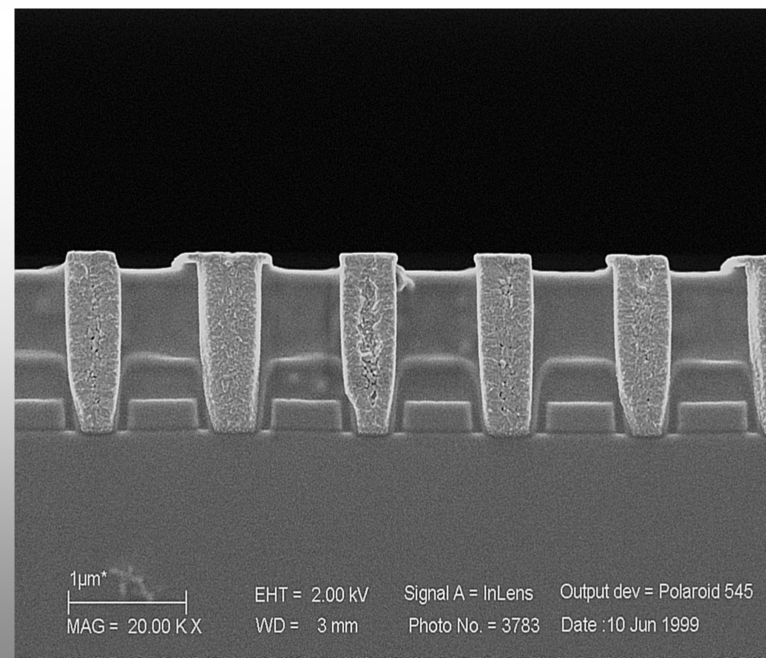
Pour obtenir une surface lisse:
rotation et inclinaison de l'échantillon


1a
1b


1c
1d


before
after

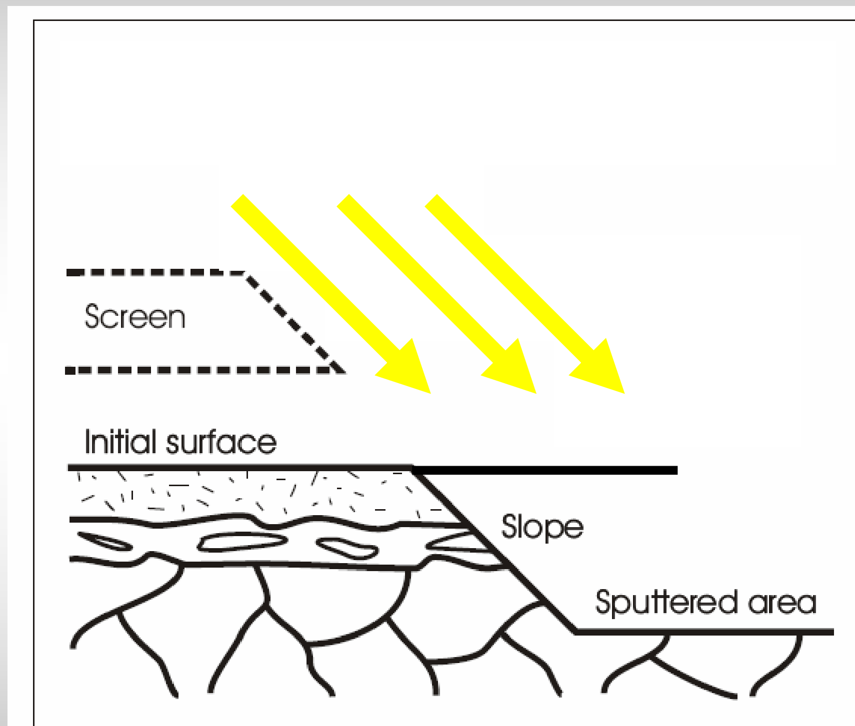

1µm*
MAG = 20.00 K X
EHT = 1.50 kV
WD = 3 mm
Signal A = InLens
Photo No. = 3760
Output dev = Polaroid 545
Date :10 Jun 1999


1µm*
MAG = 20.00 K X
EHT = 2.00 kV
WD = 3 mm
Signal A = InLens
Photo No. = 3783
Output dev = Polaroid 545
Date :10 Jun 1999

Advances in Broad Ion Beam Processing for 3D Microscopy and Microanalysis of Ceramic Composites

W. Hauffe* and R.J. Mitro**

Microsc Microanal 9(Suppl 2), 2003



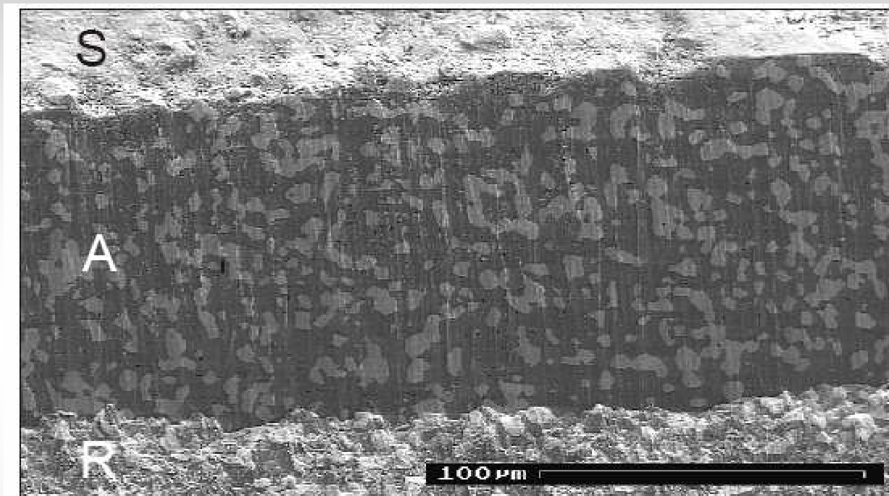


FIG. 2. FESEM image of an ion beam cut through BN/TiB_2 ceramic composite.
S Initial surface; A Slope cut area; R Sputtered region

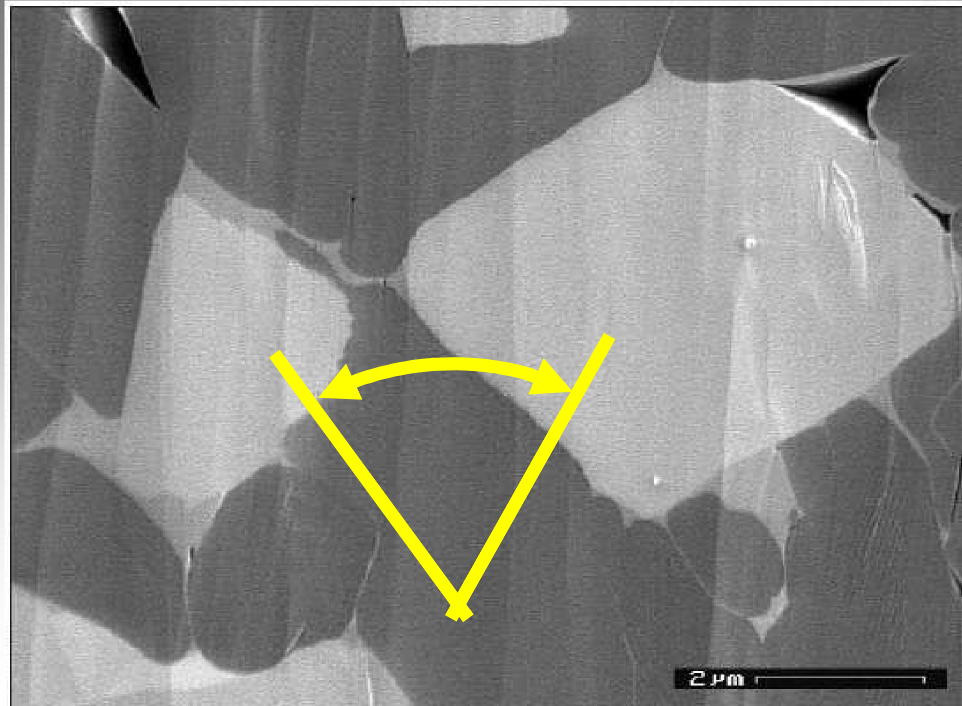
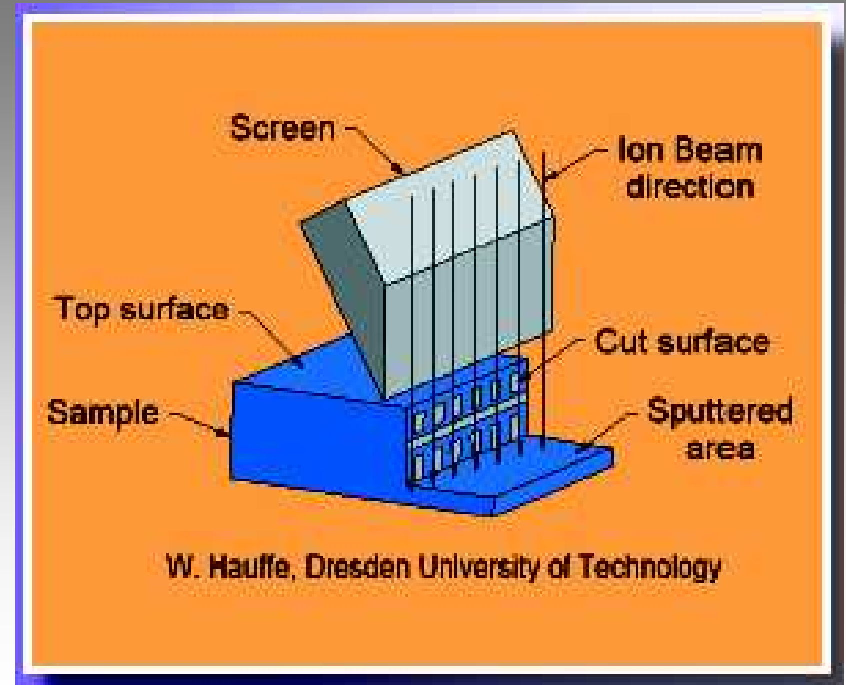


FIG. 3. FESEM image of the ion beam cut area in in FIG. 2 (Detail) with atomic number contrast between the components BN (dark), TiB_2 (bright) and the glassy phase (grey)



+ Cross-sections de grand envergure

A New Method of Surface Preparation for High Spatial Resolution EPMA/SEM with an Argon Ion Beam

Hideyuki Takahashi[1,*], **Ayako Sato**[1], **Masaru Takakura**[1], **Norihisa Mori**[1], **Juergen Boerder**[2], **Walter Knoll**[2], and **John Critchell**[3]

[1] Application and Research Group, Electron Optics Division, JEOL Ltd., Tokyo 196-8558, Japan
[2] JEOL Germany GMBH; Oskaer-von-Miller Strasse 1, D-85386 Eching, Germany
[3] JEOL UK Ltd., JEOL House, Silver Court, Watchmead, Welmyn Garden City, Herts, AL7 1LT, United Kingdom

Microchim Acta 155, 295–300 (2006)

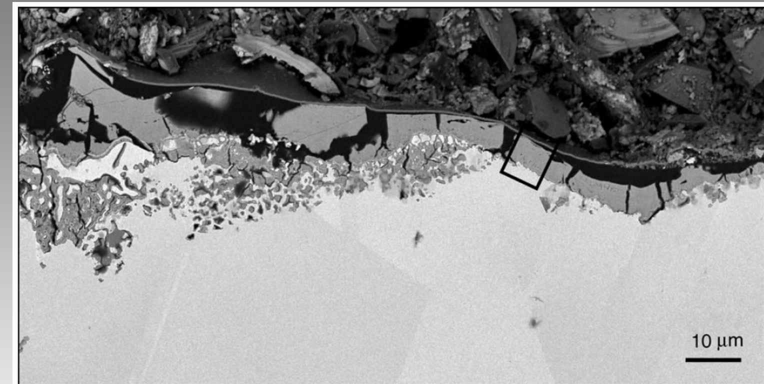
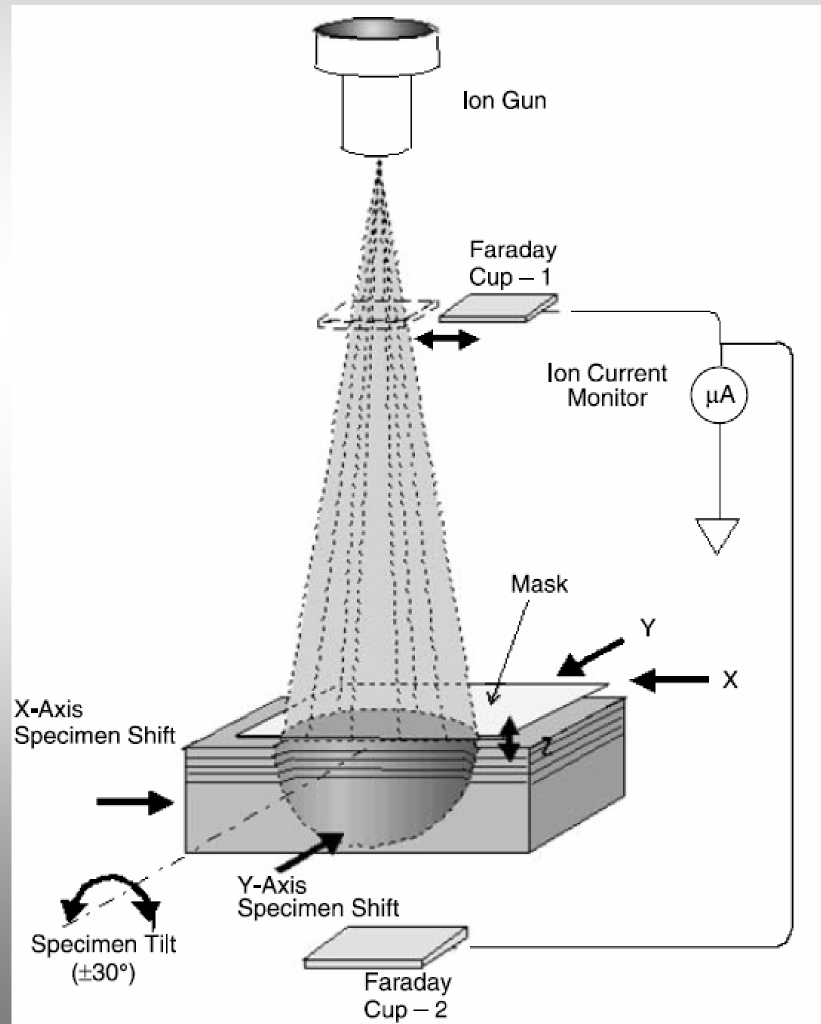
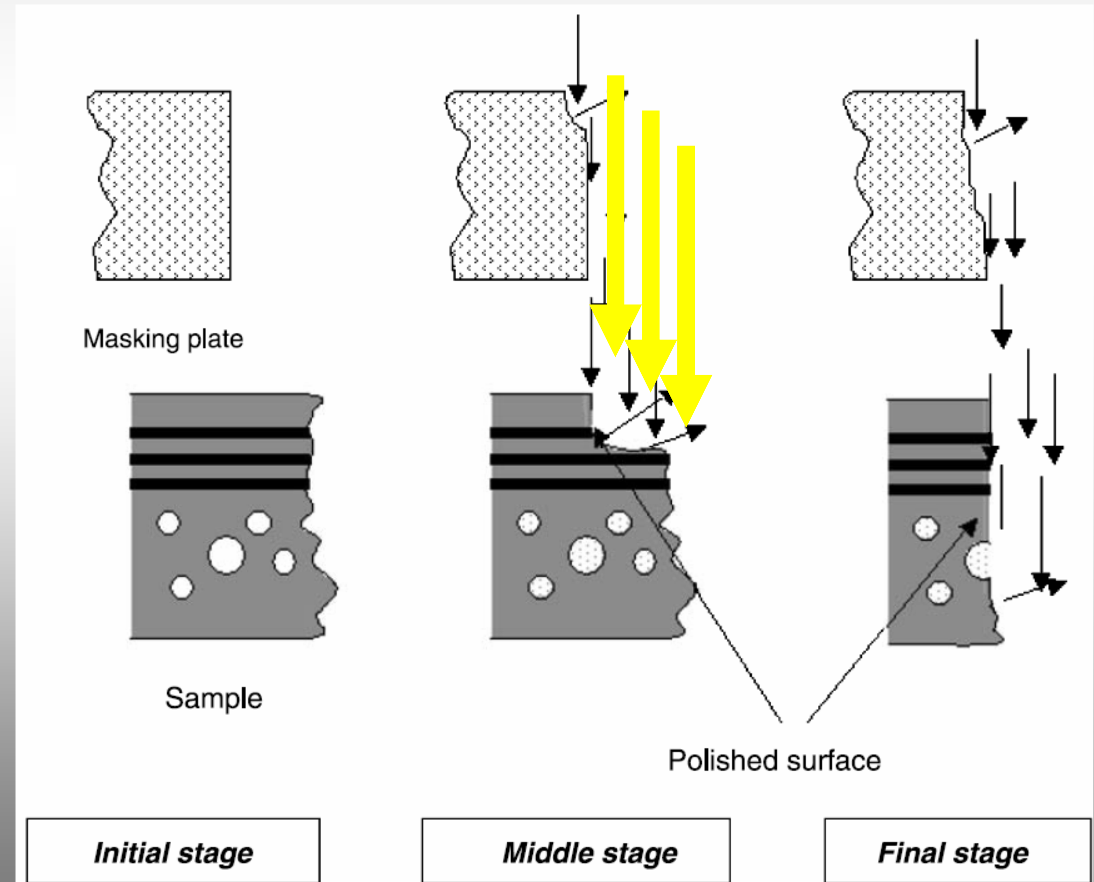


Fig. 3. BSEI of a cross section of a corroded surface zone in Alloy 825 (10 kV)





JEOL

Fig. 2 Cross section polisher (CP) SM-09010

Table 1 Principal specifications

Ion gun	Penning type Argon ion gun
Accelerating voltage	2 to 6 kV
Ion beam diameter	500 μm (FWHM)
Milling rate	0.3 μm/min (6kV, silicon, 100μm from edge)
Maximum specimen size	11mm(W)×10mm(D)×2mm(H)
Specimen stage	X : ±3mm, Y : ±3mm
Specimen alignment	±5°
Vacuum system	TMP, RP

Fig. 11 Surface of ceramic powder (Secondary electron image)

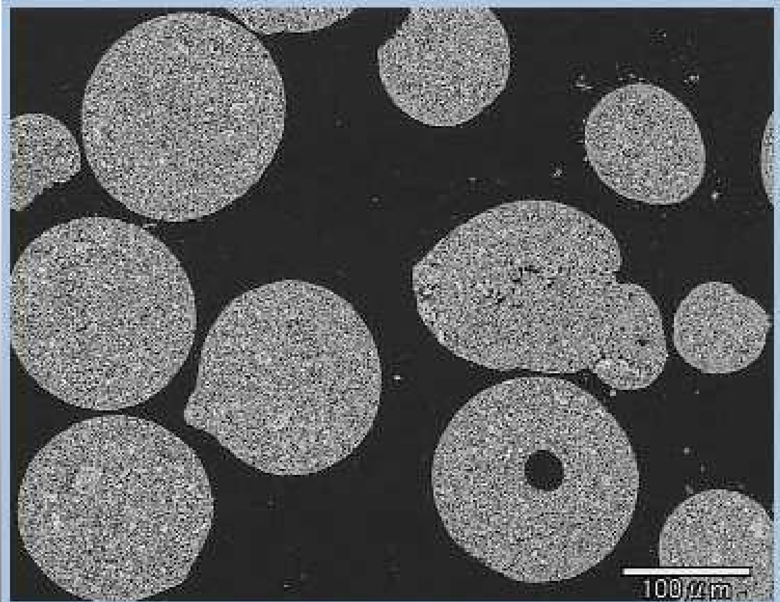
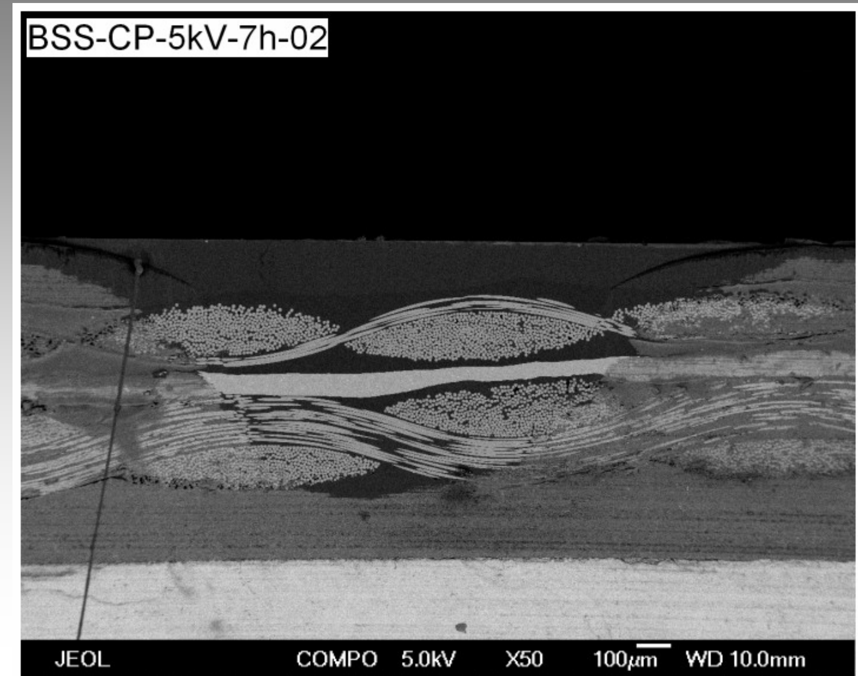
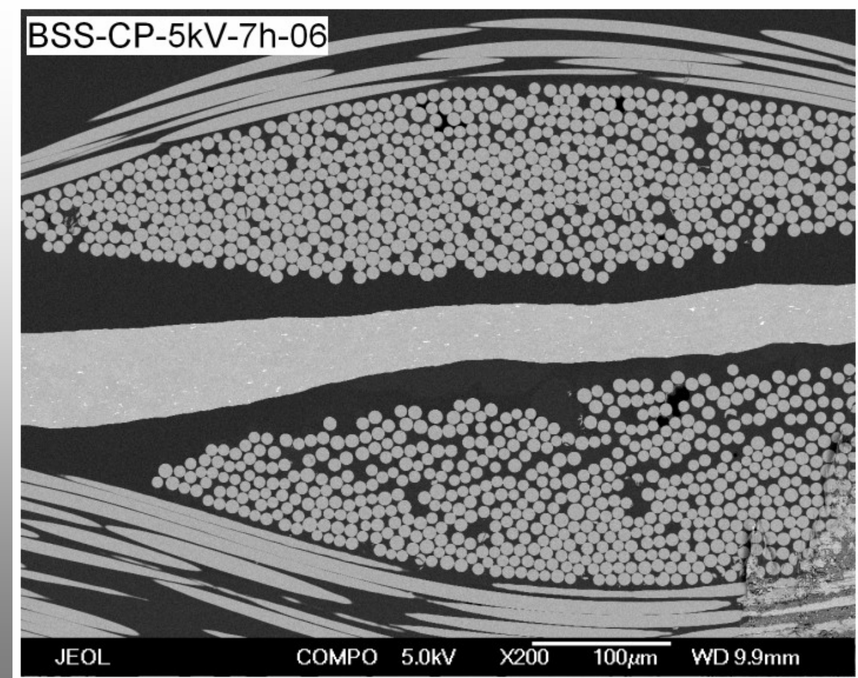
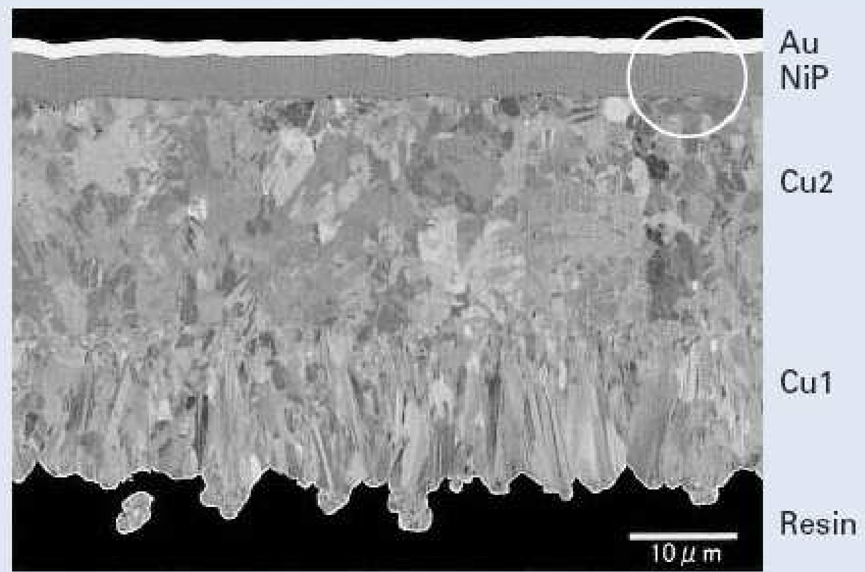


Fig. 12 Cross section of ceramic powder (Backscattered electron image)







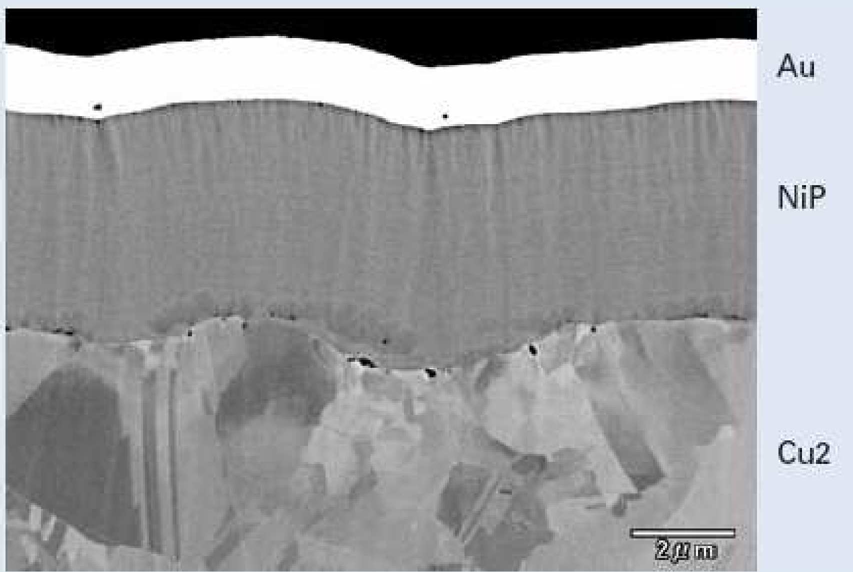
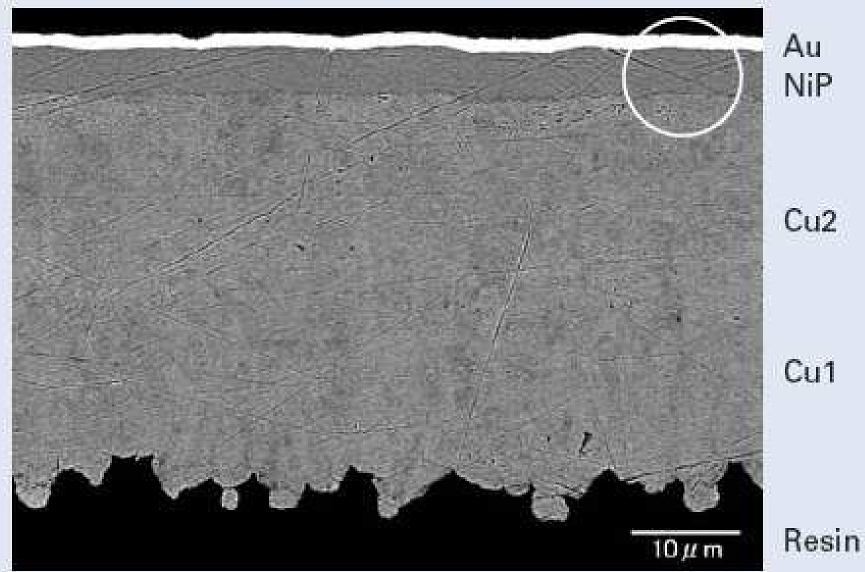


Fig. 3 CP method (Backscattered electron image)



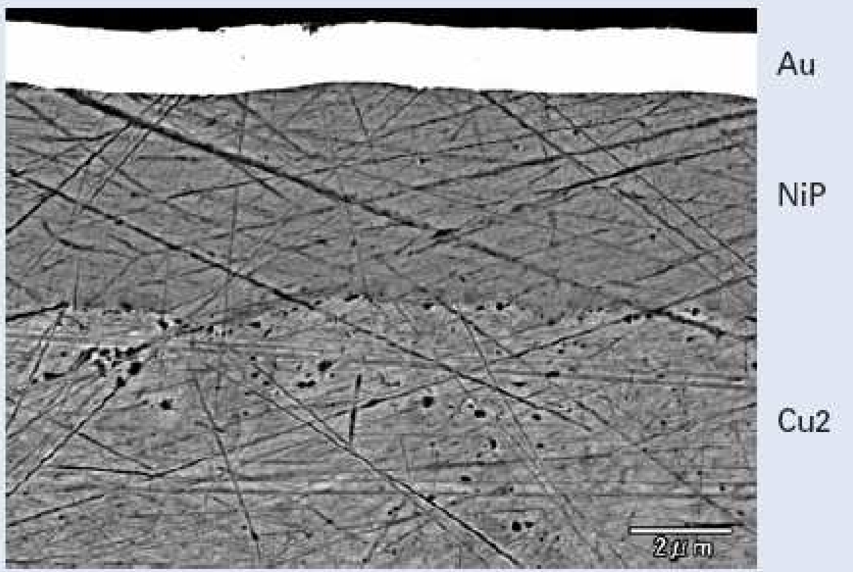
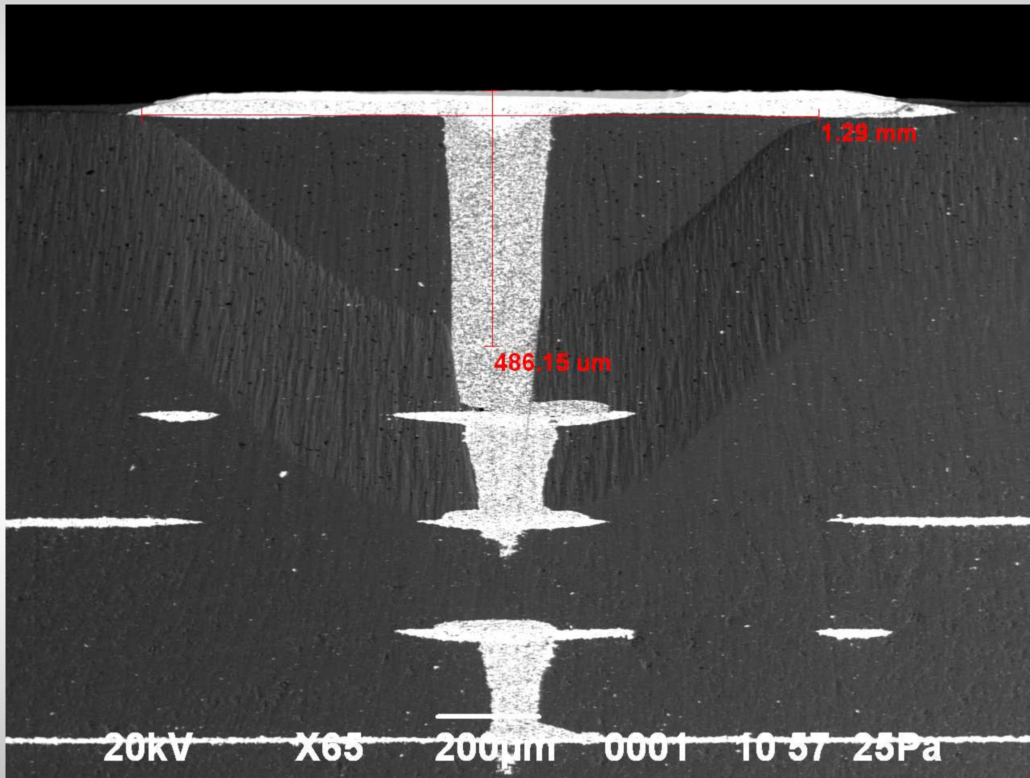
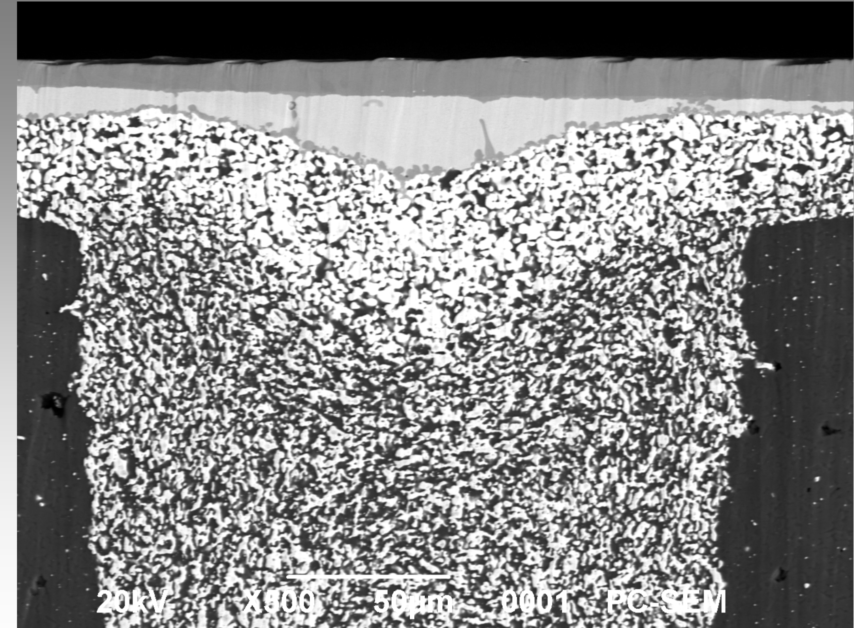


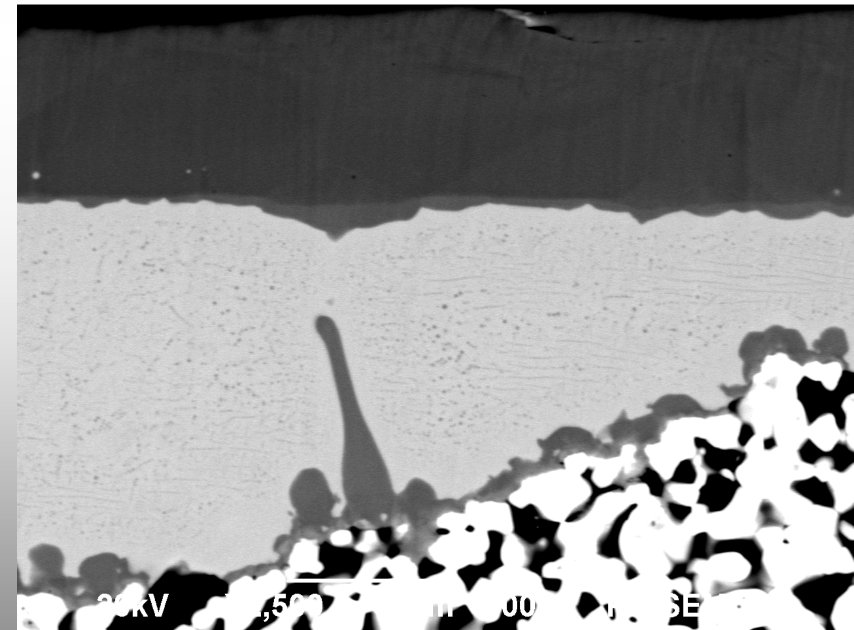
Fig. 4 Mechanical polish (Backscattered electron image)

Sample – Pentium chip, milling time – 5 hrs



Overview of the cut.
Depth ~ 490 µm
Width ~ 800 µm





Fischione

ASaP functions in one chamber

- *Plasma Cleaning (PC)*
- *Ion Beam Etching (IBE)*
- *Reactive Ion Beam Etching (RIBE)*
- *Reactive Ion Etching (RIE)*
- *Ion Beam Sputter Coating (IBSC)*

Model 1030

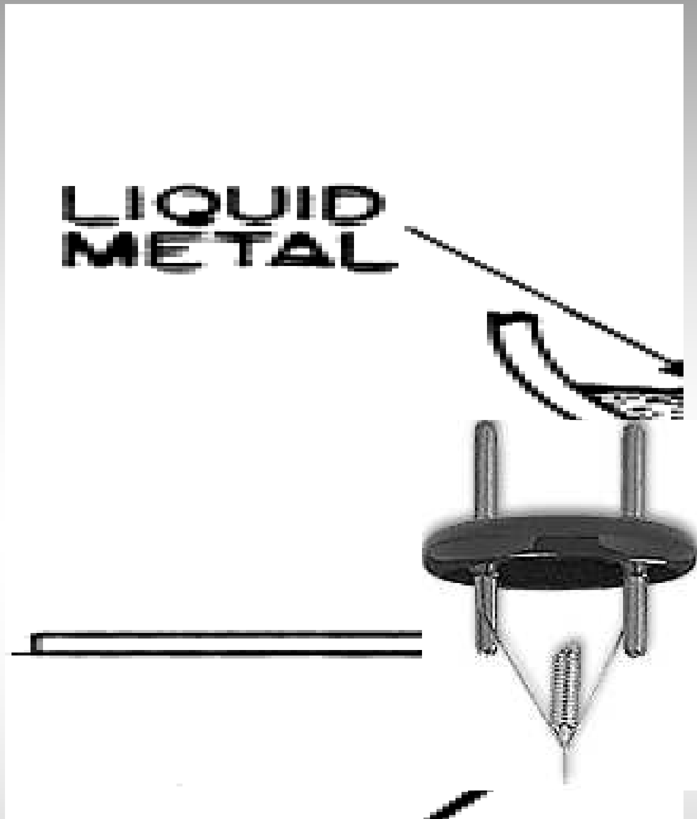
Automated Sample Prep (ASaP) System

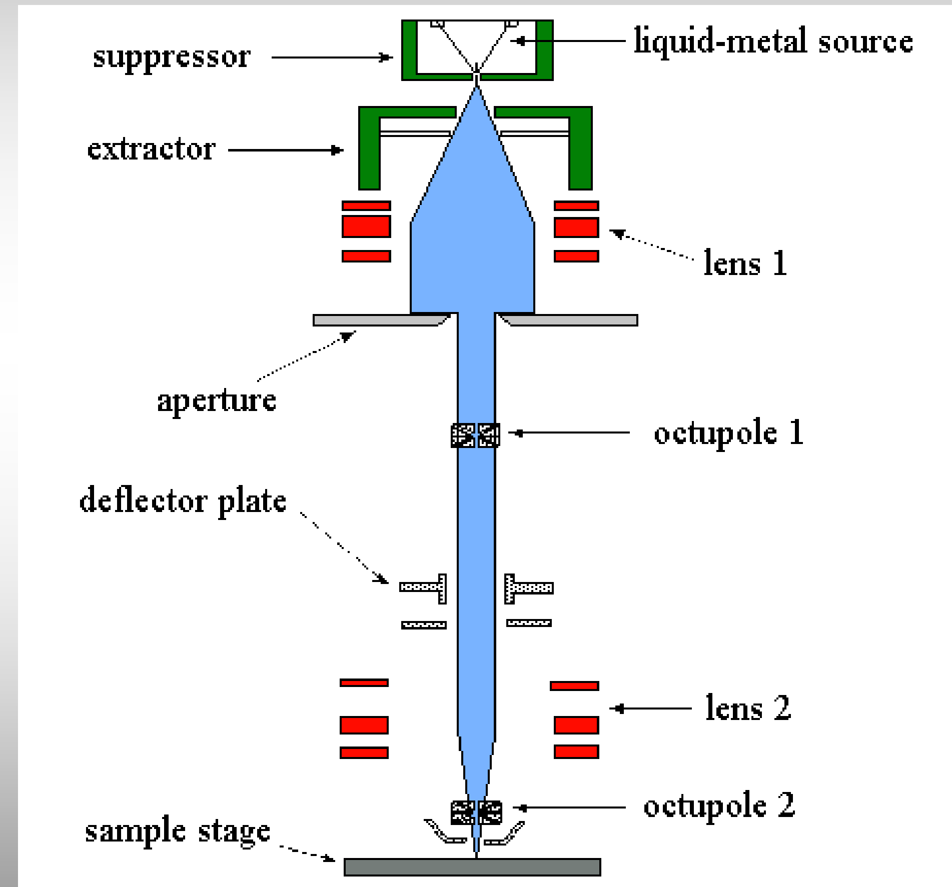
(Patent Pending)

BAL-TEC RES120 SEM

Faisceau ionique focalisé

Focused Ion Beam (FIB)





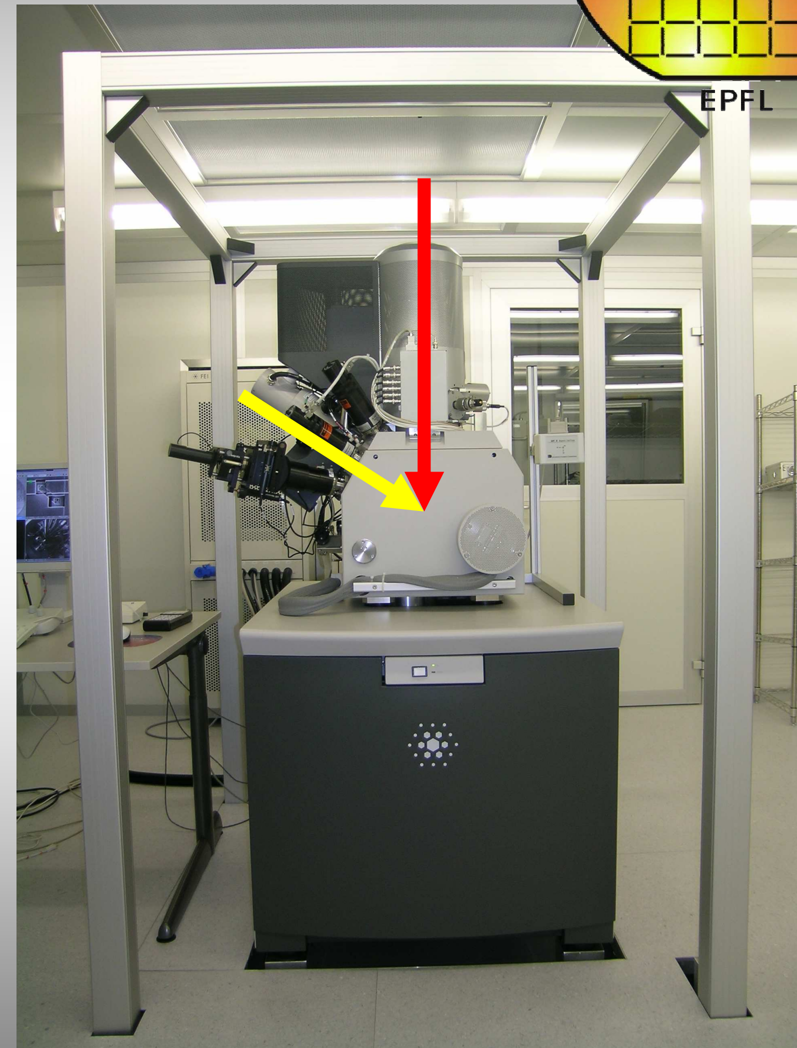
Schematic diagram of a FIB ion column
Source: IBM Almaden Research Center

- Développé: 1970's and 80's (Escovitz, Levi-Setti, Orloff, Swanson…)
- MEB-> « MIB »
- Source: Liquid Metal Ion Source (LMIS).
  Ex: Ga, Au, Be, Si, Pd,B, P, As, Ni, Sb,alloys …
- Principe: émission à effet de champs

Depuis avril 2004:

FIB au CMI/EPFL

- **Dual Beam Nova 600 Nanolab** FEI
- **Dual Beam = SEM + FIB**
- SEM: Schottky thermal field emitter
- FIB: Ga LMIS
- Samples, 4 and 6 inch wafers
- 4 Gas Injector Systems
  - Pt deposition (C9H16Pt)
  - SiO2 deposition (TEOS)
  - Insulator Enhanced Etch (XeF2)
  - Selective Carbon Mill (MgSO4)
- Charge neutralizer system
- Omniprobe (in situ TEM lamella lift out)
- Fast Electron Beam Blanker (basic e beam lithography)



3 “ modes” du faisceau ionique

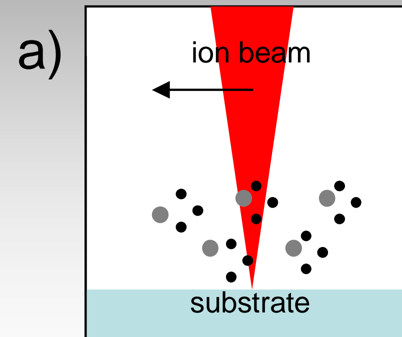
- Emission de ions secondaires et d’électrons secondaire imagerie ionique

- Usinage et polissage
  - Attaque ionique currants élevés

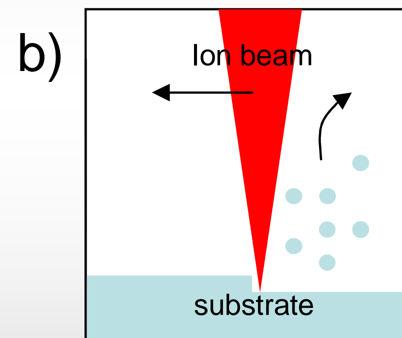
- Reactions chimique (assisté de gaz)
  - FIB **deposition**
  - Attaque préférentielle

- autres:
  Implantation d’ions
  amorphisation

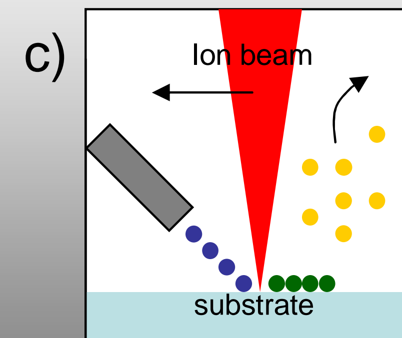
a)

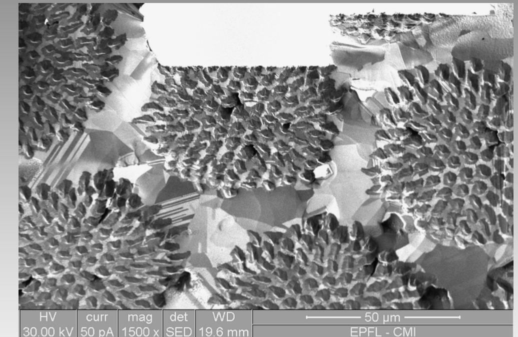


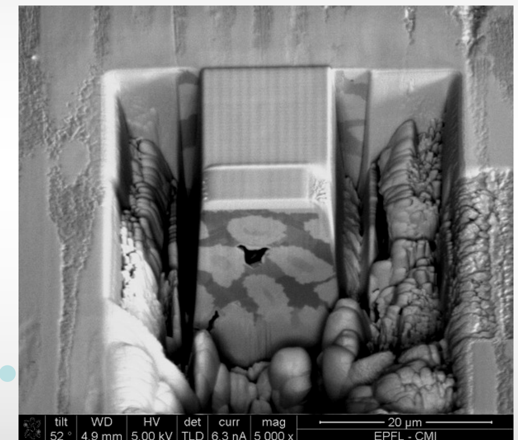
b)

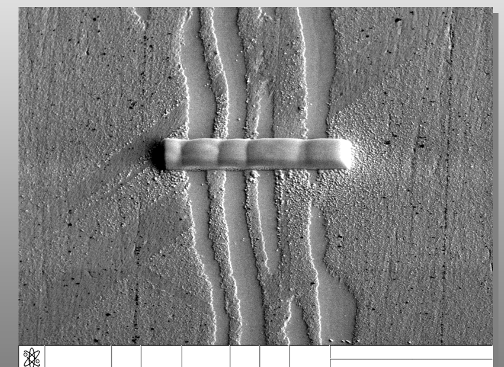


c)

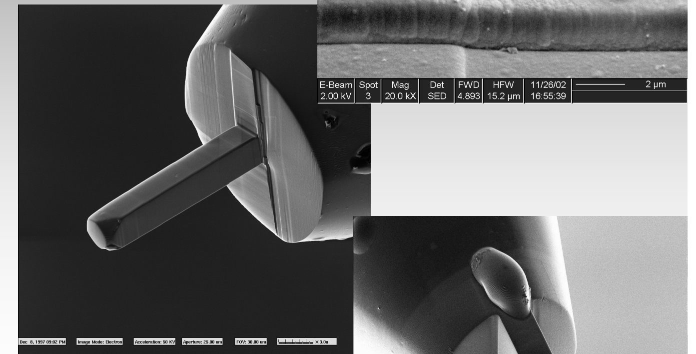








Applications

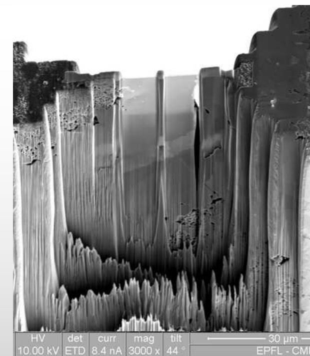


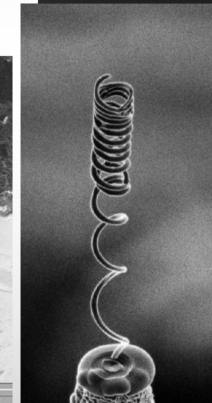
“Industrial” applications
(semiconductor industry)

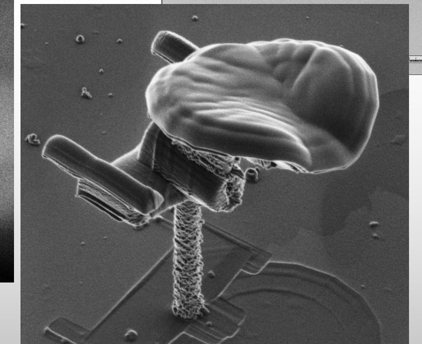
- sectioning for failure analysis
- prototype circuit rewiring
- mask repair
- **TEM sample preparation**

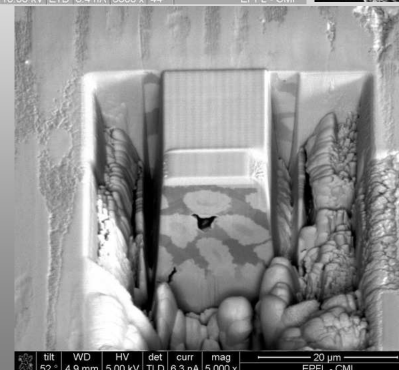
Research

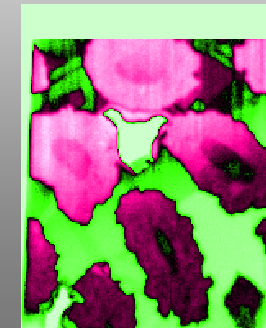
- Micromachining
- Nanofabricated structures
- **TEM sample preparation**
- **FIB Nano-Tomography**





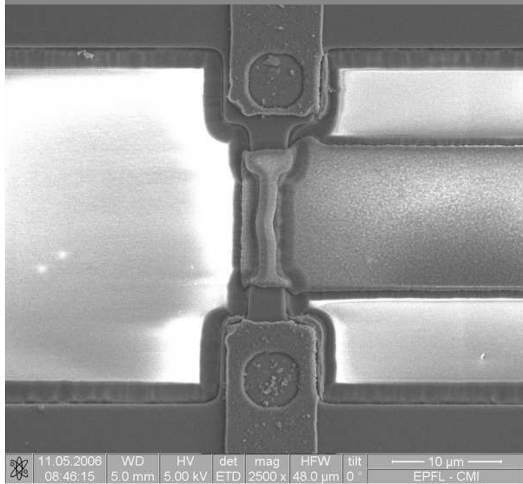


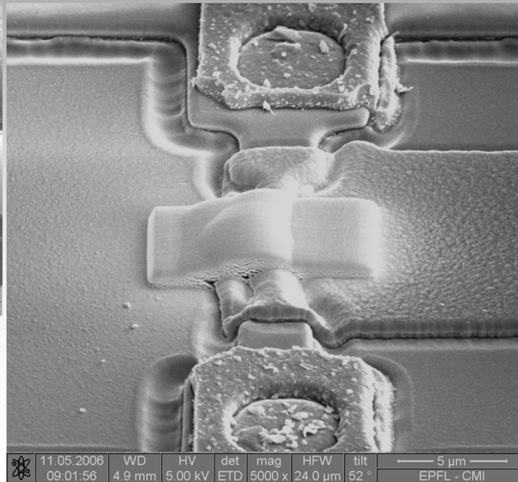


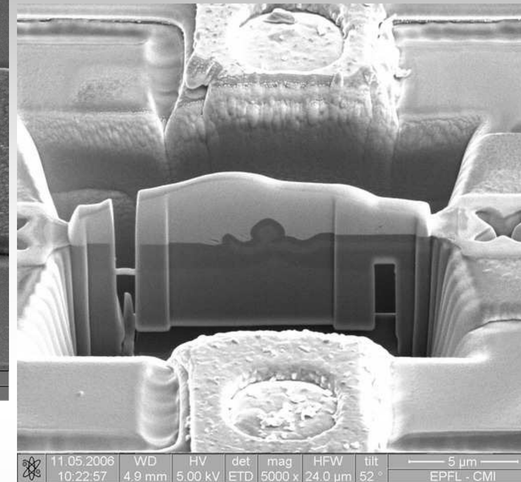


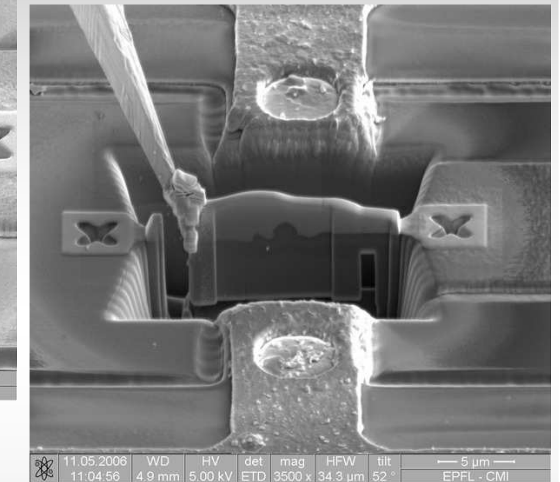
nano-fil de Si

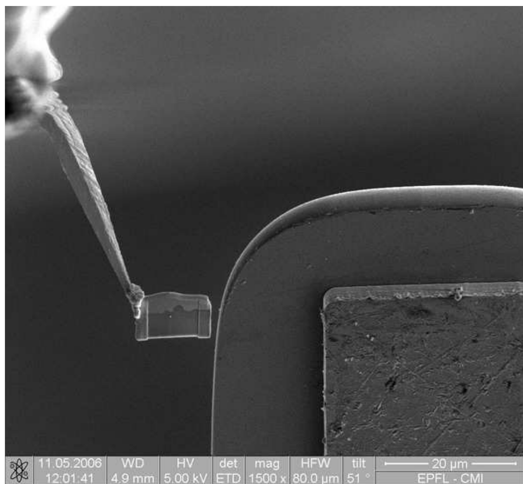
M. Pavius, V. Pott, CMI

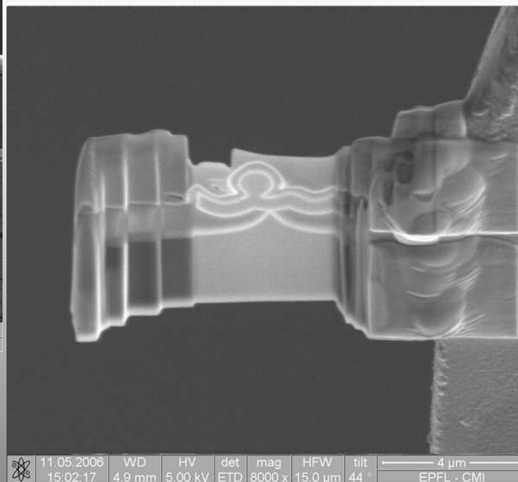


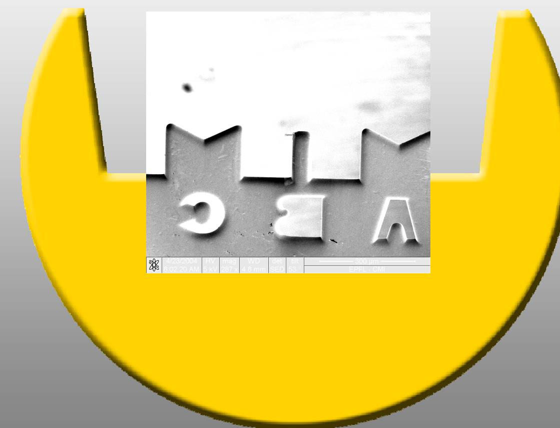






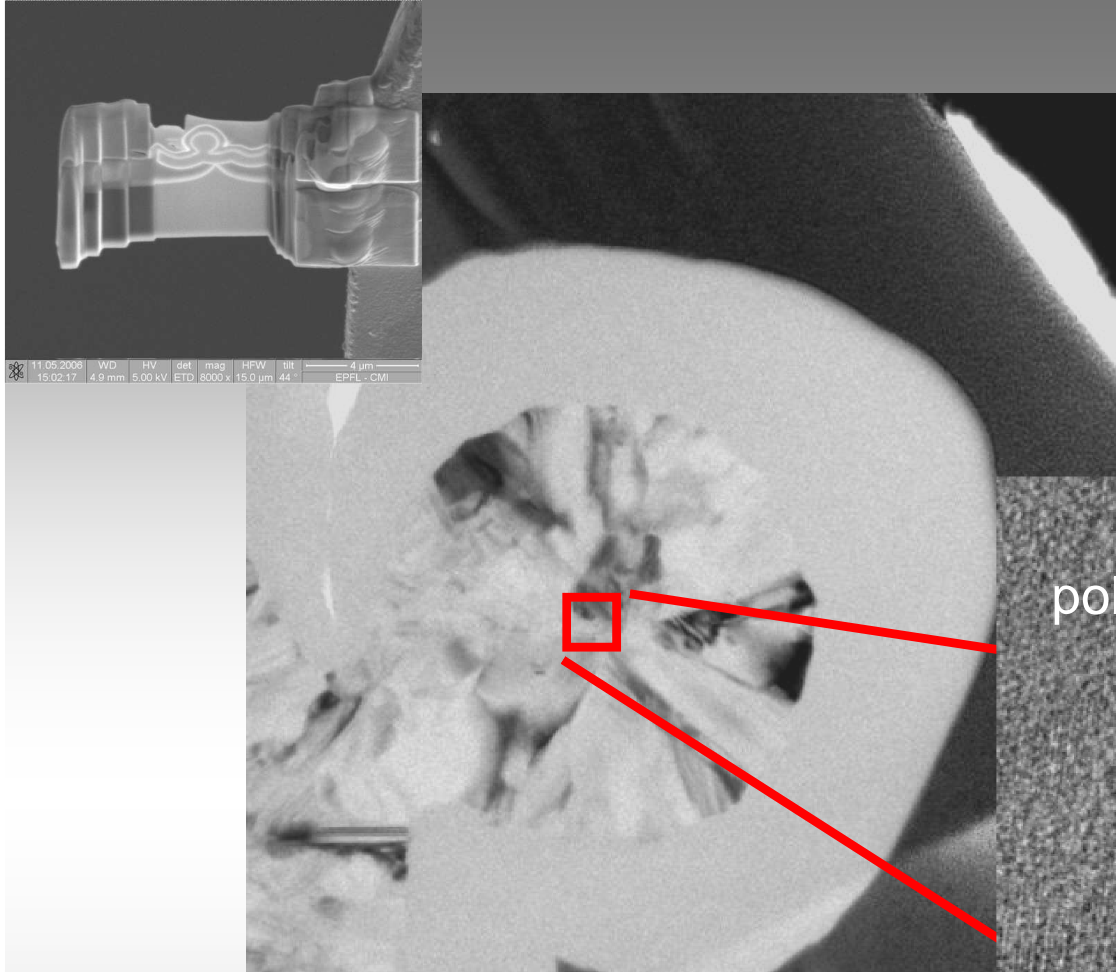


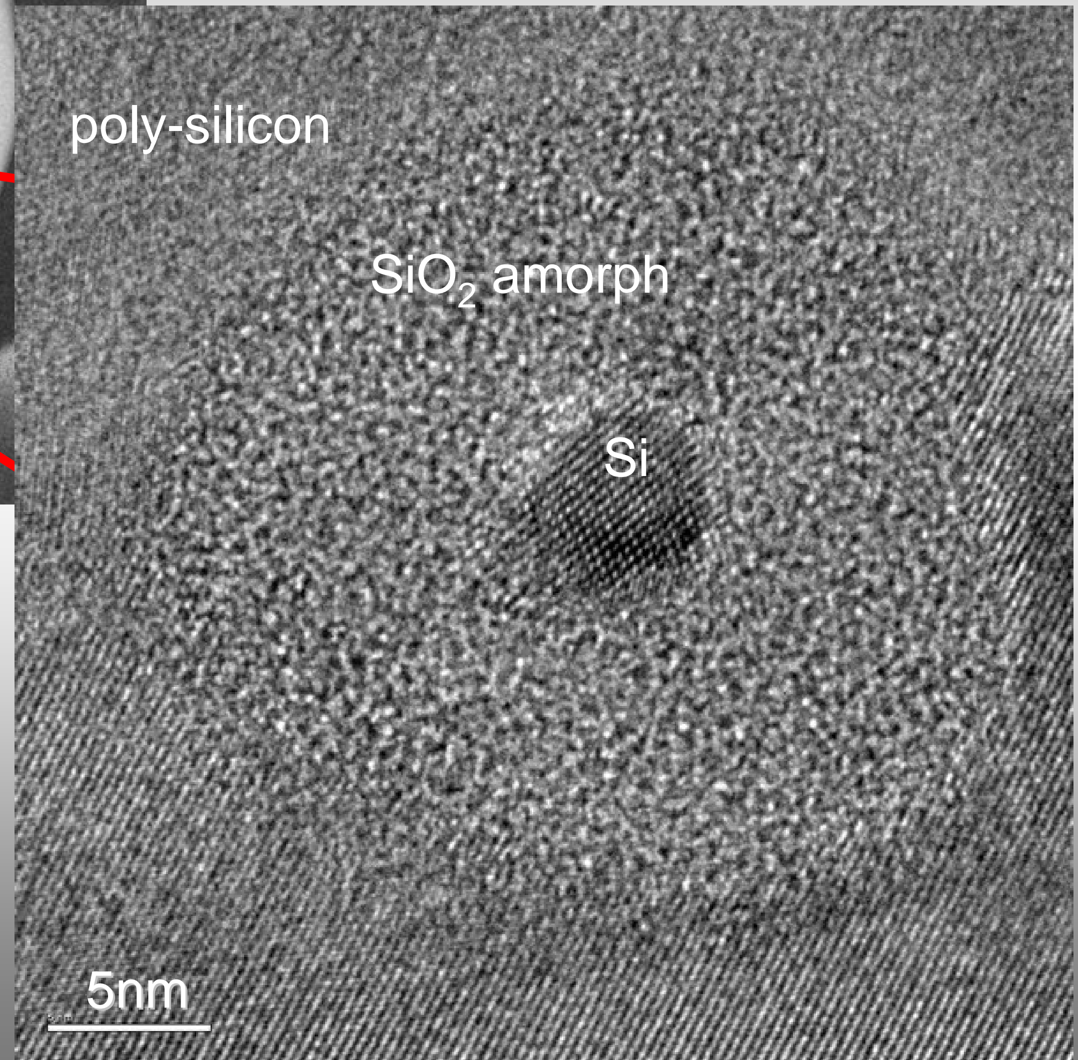




Porte-échantillon MET, diametre 3mm

TEM, HRTEM

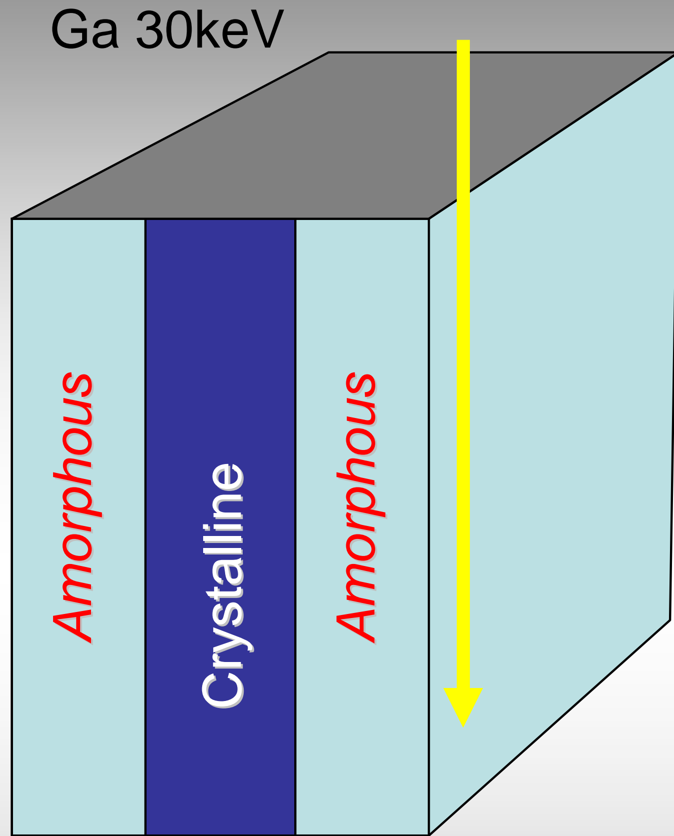




- Zone mine (10x5um) avec épaisseur constante (typ. 50-80 nm)
- Préparation d'échantillons hétérogènes
- Précision dans la sélection d'endroit.

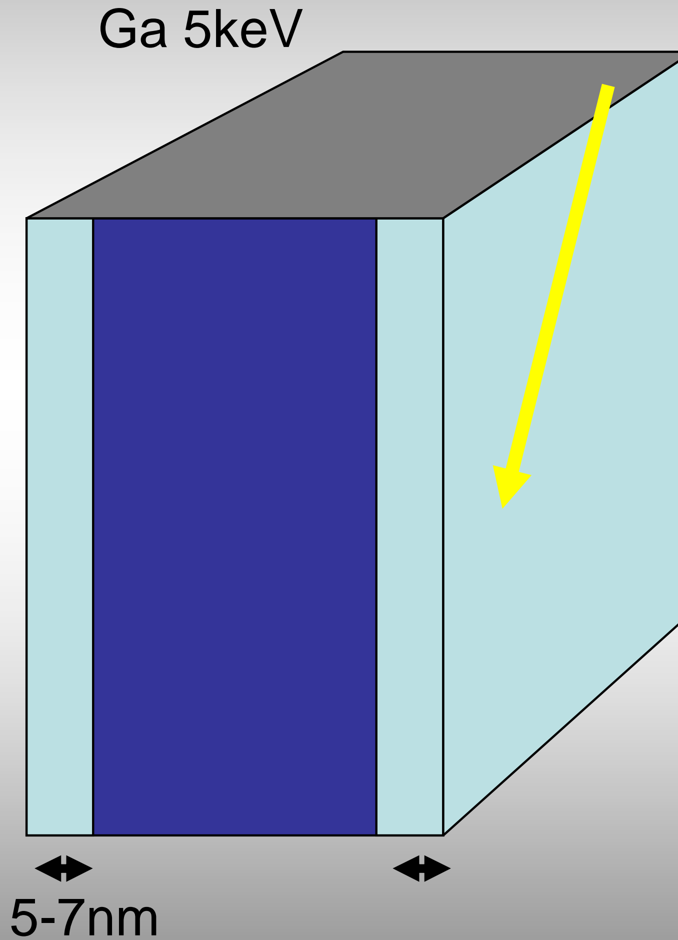
Amorphisation

« low-kV cleaning »

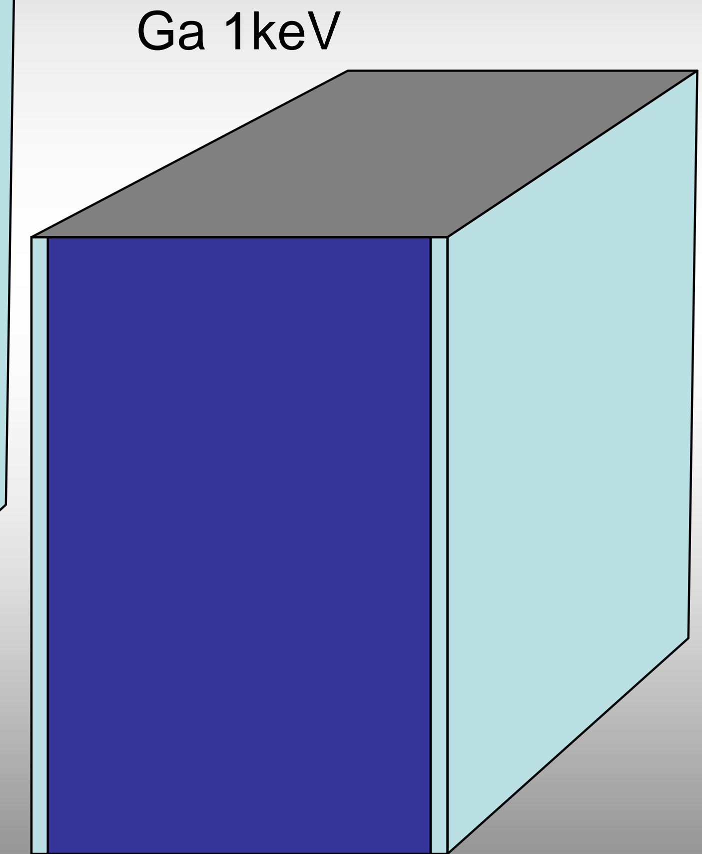


25-30nm 25-30nm
Zone amorphisée

Valeurs typiques pour Si

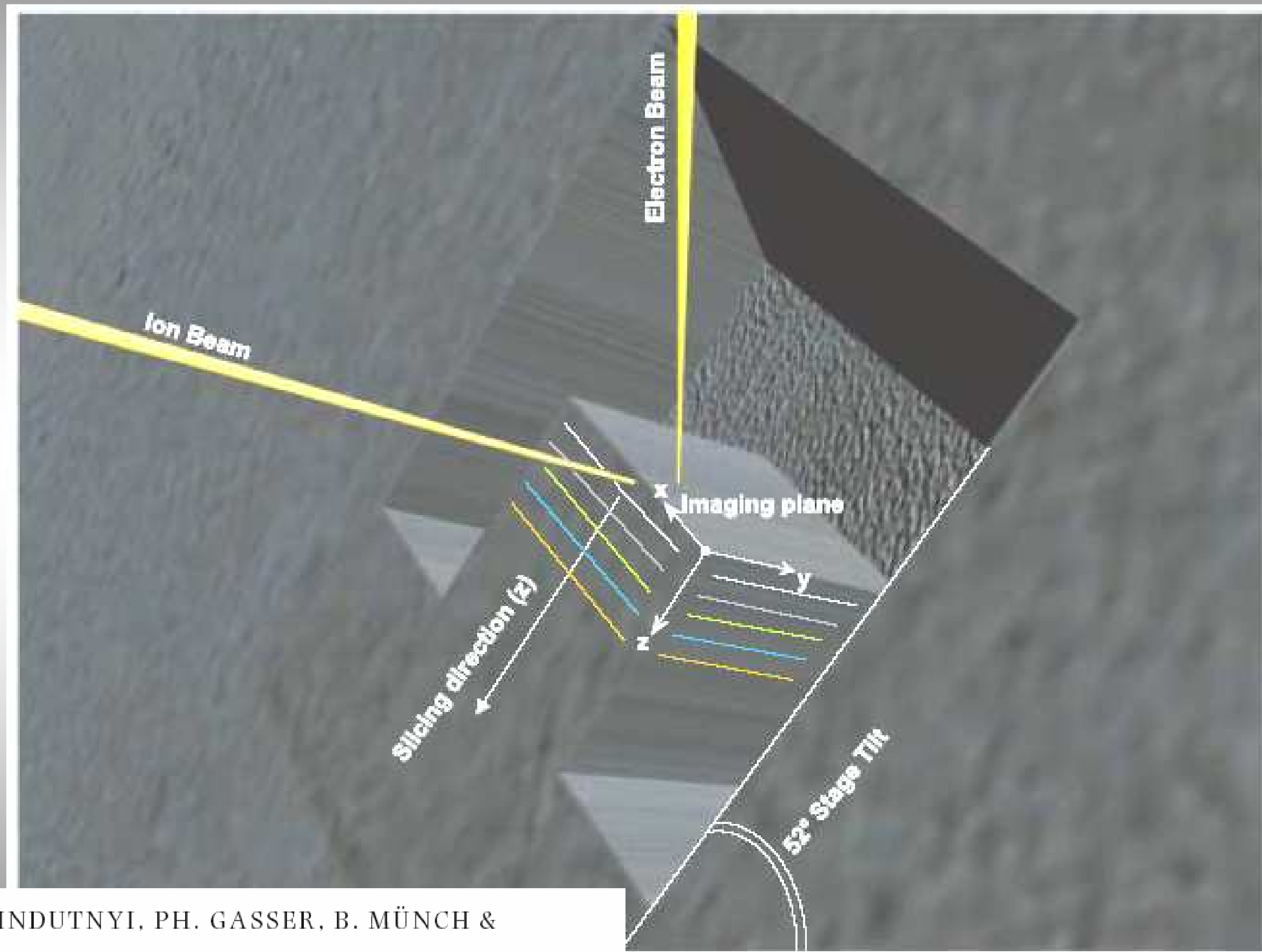


5-7nm



2-3 nm

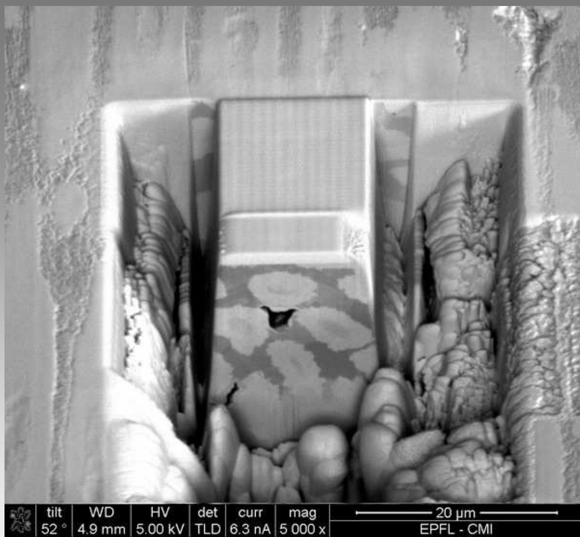
Microscopie 3D: FIB Nanotomography



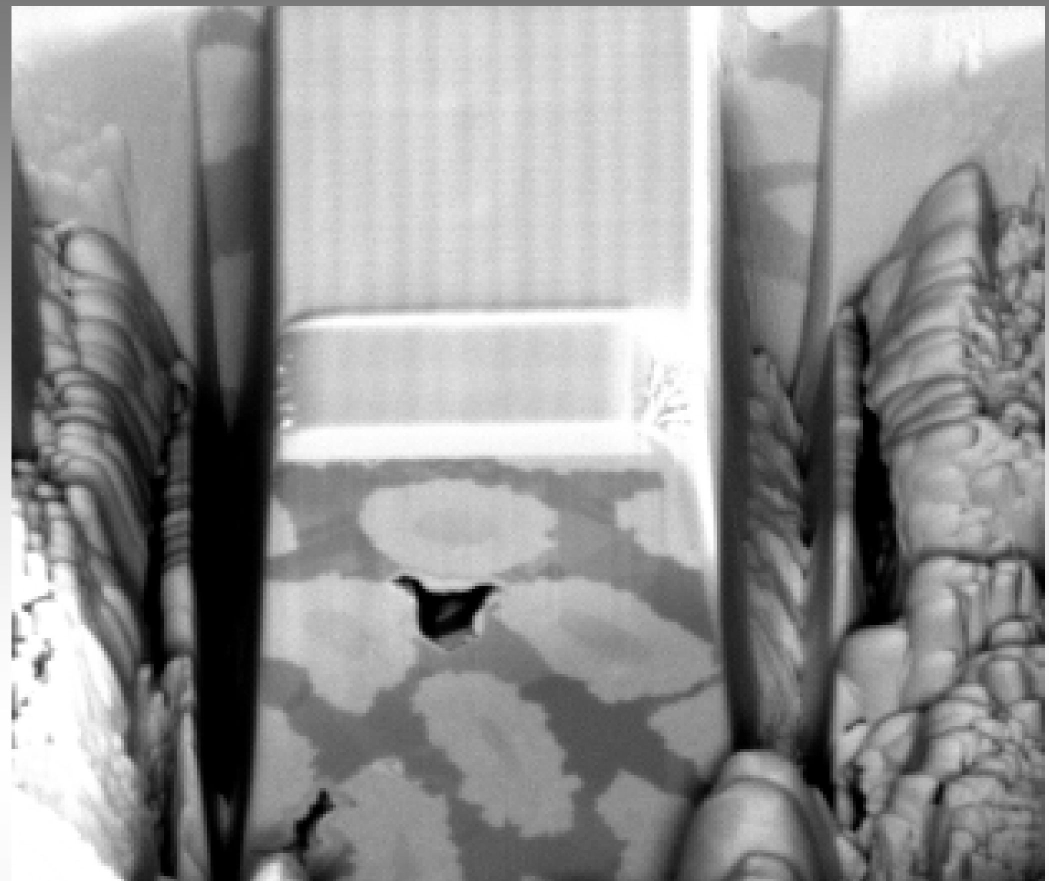
L. HOLZER, F. INDUTNYI, PH. GASSER, B. MÜNCH & M. WEGMANN

EMPA, Swiss Federal Laboratories for Materials Testing and Research, Ueberlandstrasse 129, 8600 Dübendorf, Switzerland

Journal of Microscopy, Vol. 216, Pt 1 October 2004, pp. 84–95

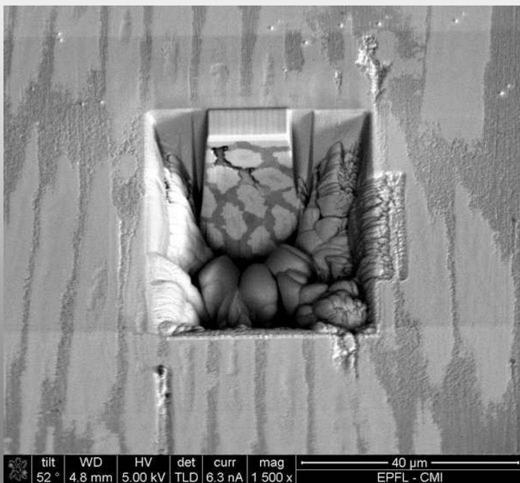


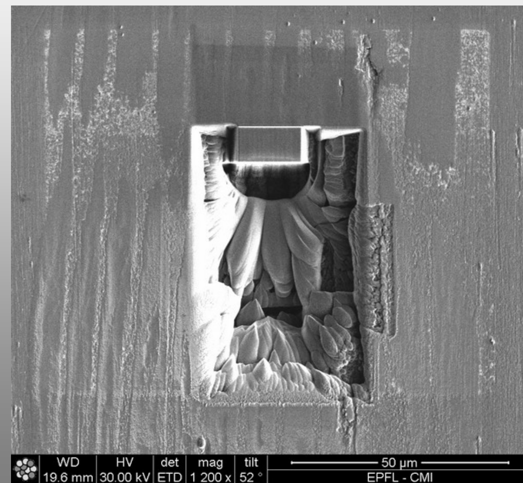
Preparation



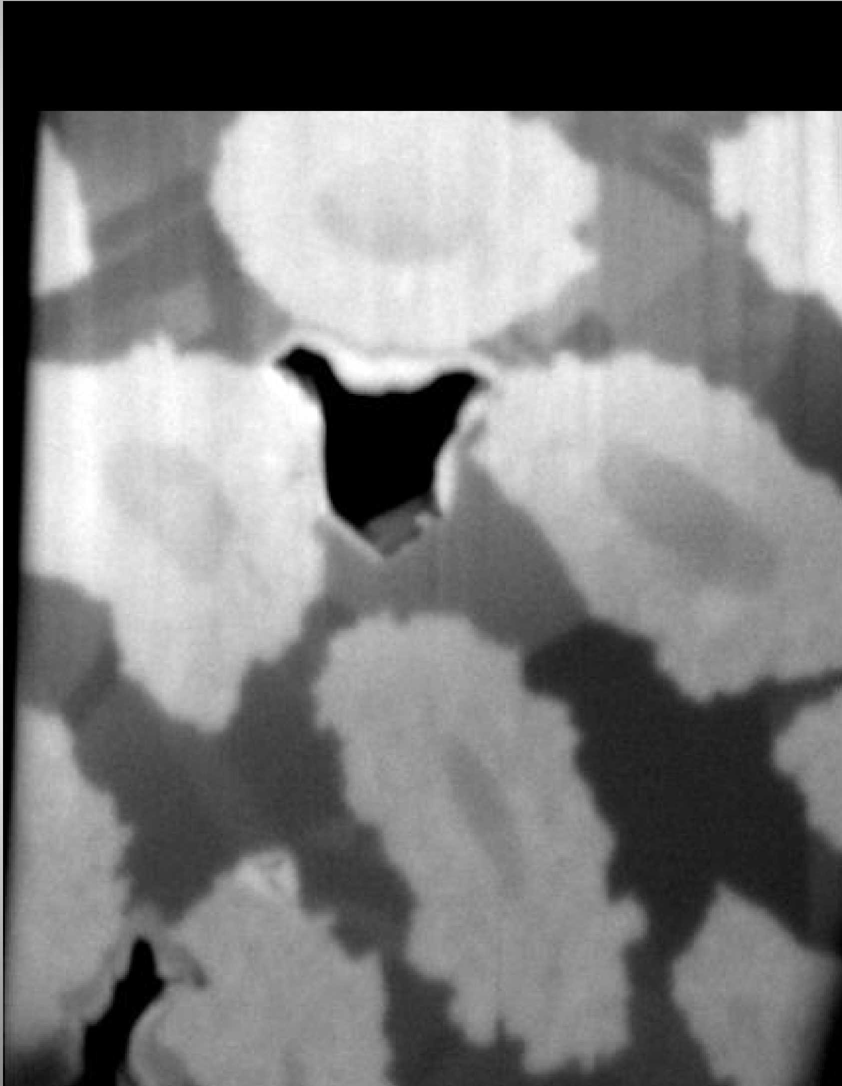
Usinage et imagerie automatisé: 170 sections (10h)

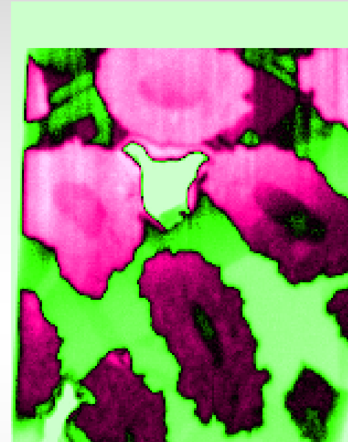
la fin:





alignement des images
et selection de la zone interessante



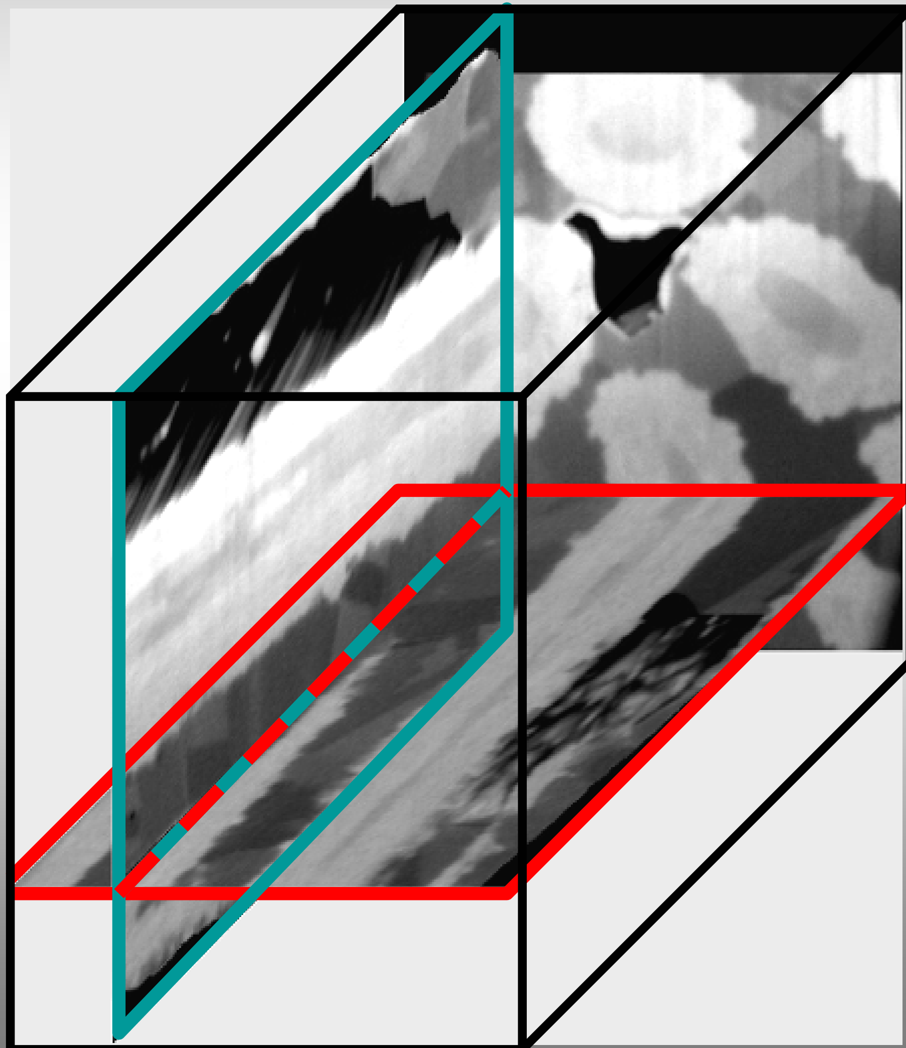


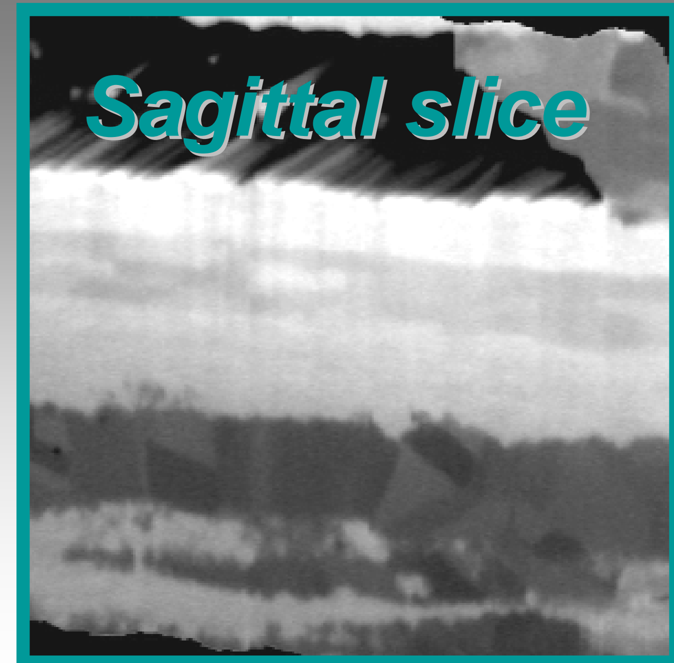
ImageJ
Image Processing and Analysis in Java

http://rsb.info.nih.gov/ij/index.html

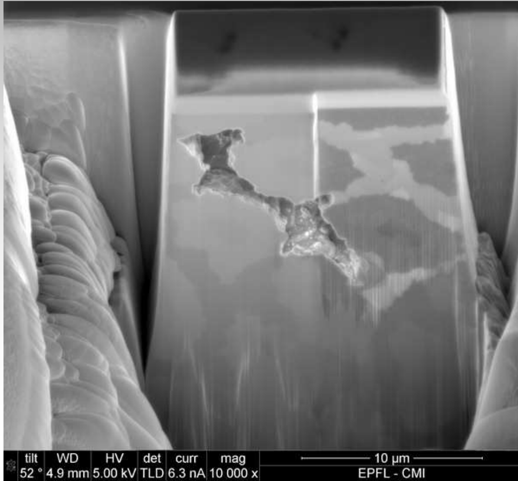
3D volume rendering, reconstruction:

Orthogonal slices

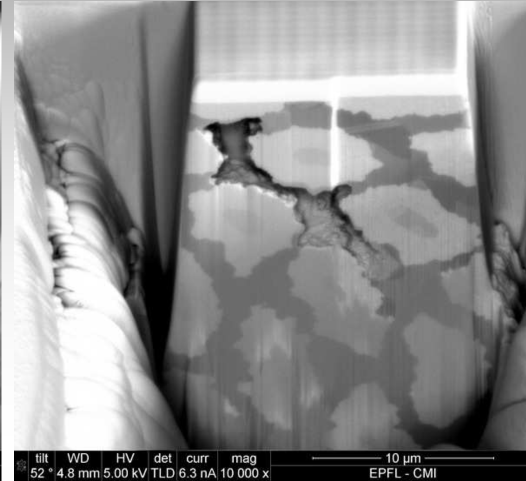




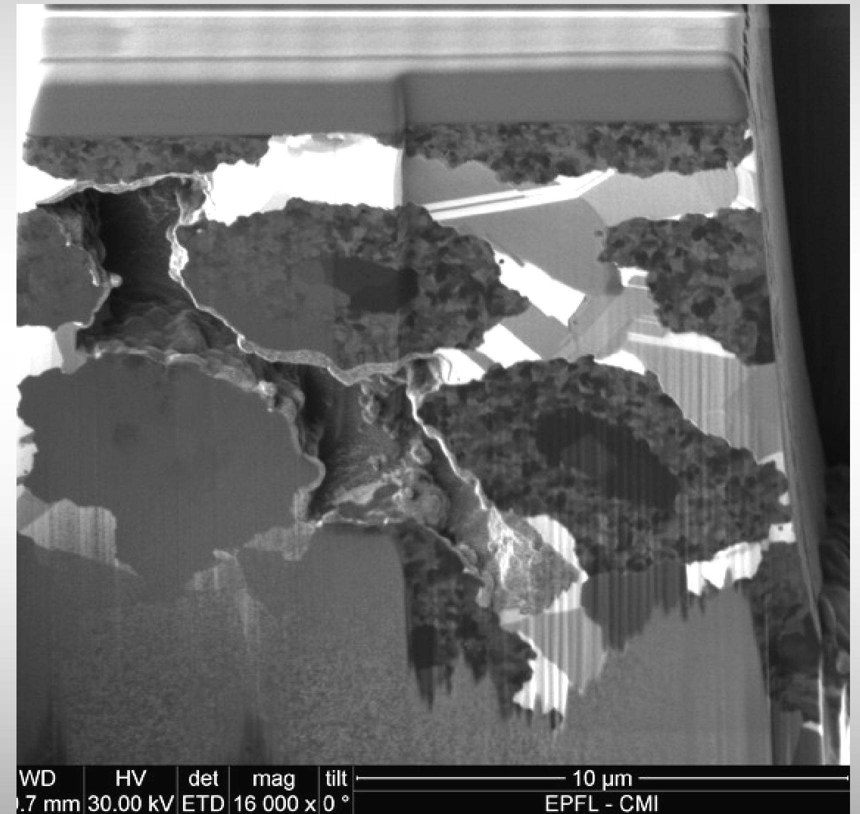
Le choix du detecteur



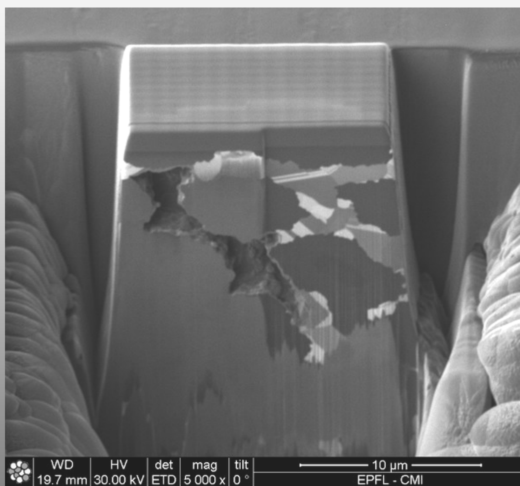
SE detector (TLD)



BSE detector (TLD)

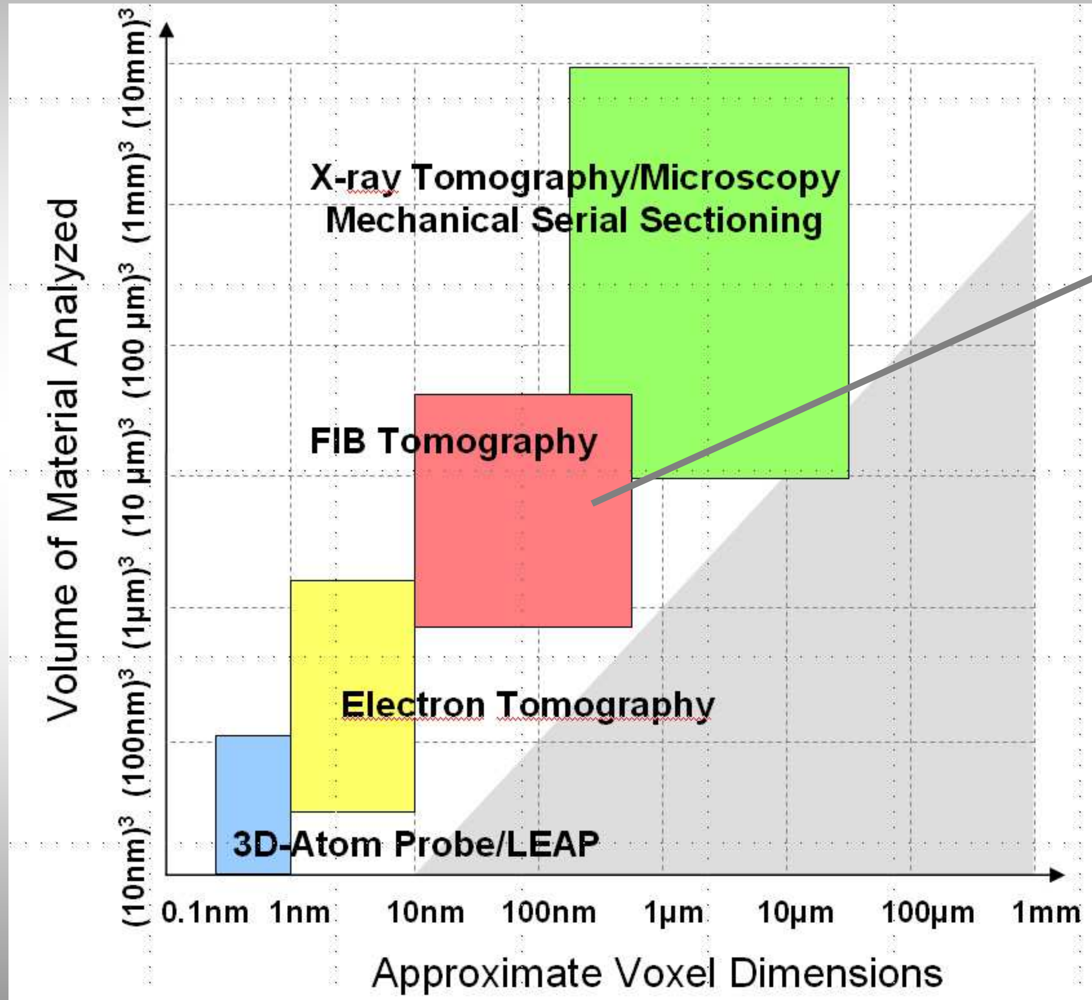


Imagerie ionique nécessite tilts et rotations…!



Ion beam imaging (SE)

FIB-NT en relation avec les autres techniques „3D“

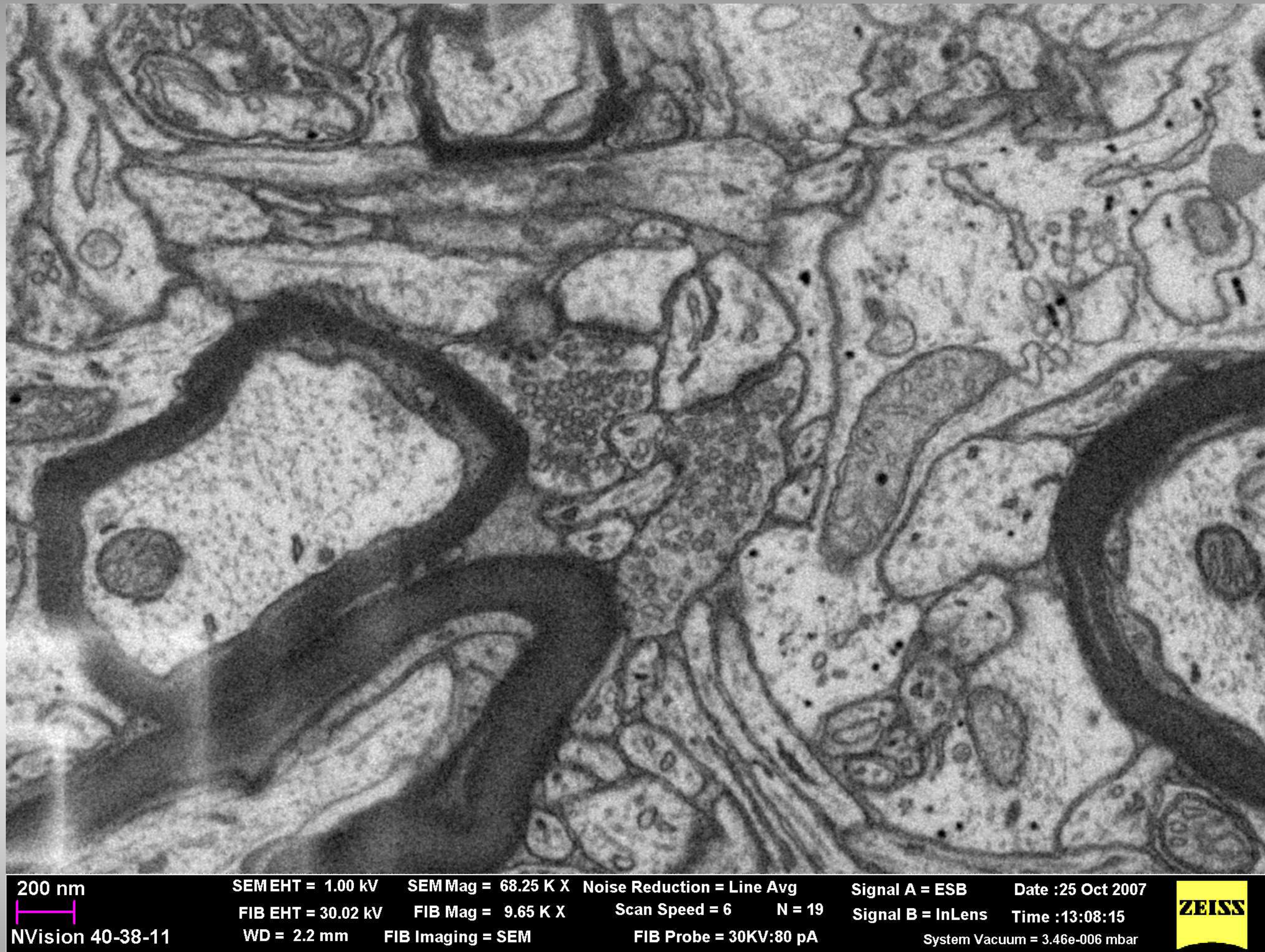


From: Uchic, Holzer and Inkson
Mat. Res. Bul., subm.

Possibilités nouvelles dans la microscopie 3D:
Combination de FIB-nt
avec Cryo, EBSD, EDX

SEM goes 3D

Echantillons biologiques….



Graham Knott UNIL / CIME (dès 1.12.2007)

Conclusions

- **BIB: Broad Ion Beam: faisceau « large », 1-5mm (200V-10kV)**
  « nettoyage », décapage, polissage des surfaces relativement larges pour imagerie rétrodiffusée, spectroscopie et EBSD
  facile à utiliser
- **FIB: Focused Ion Beam: faisceau focalisé, >4nm (30kV – 500V)**
  Usinage et polissage avec haute précision pour préparation des échantillons MET
  Combiné avec toute la palette des techniques MEB: une nouvelle dimension !