

Préparations d'échantillons pour microscopie analytique de 0.05 à 30 keV

Pr. Raynald Gauvin, Ph.D.,

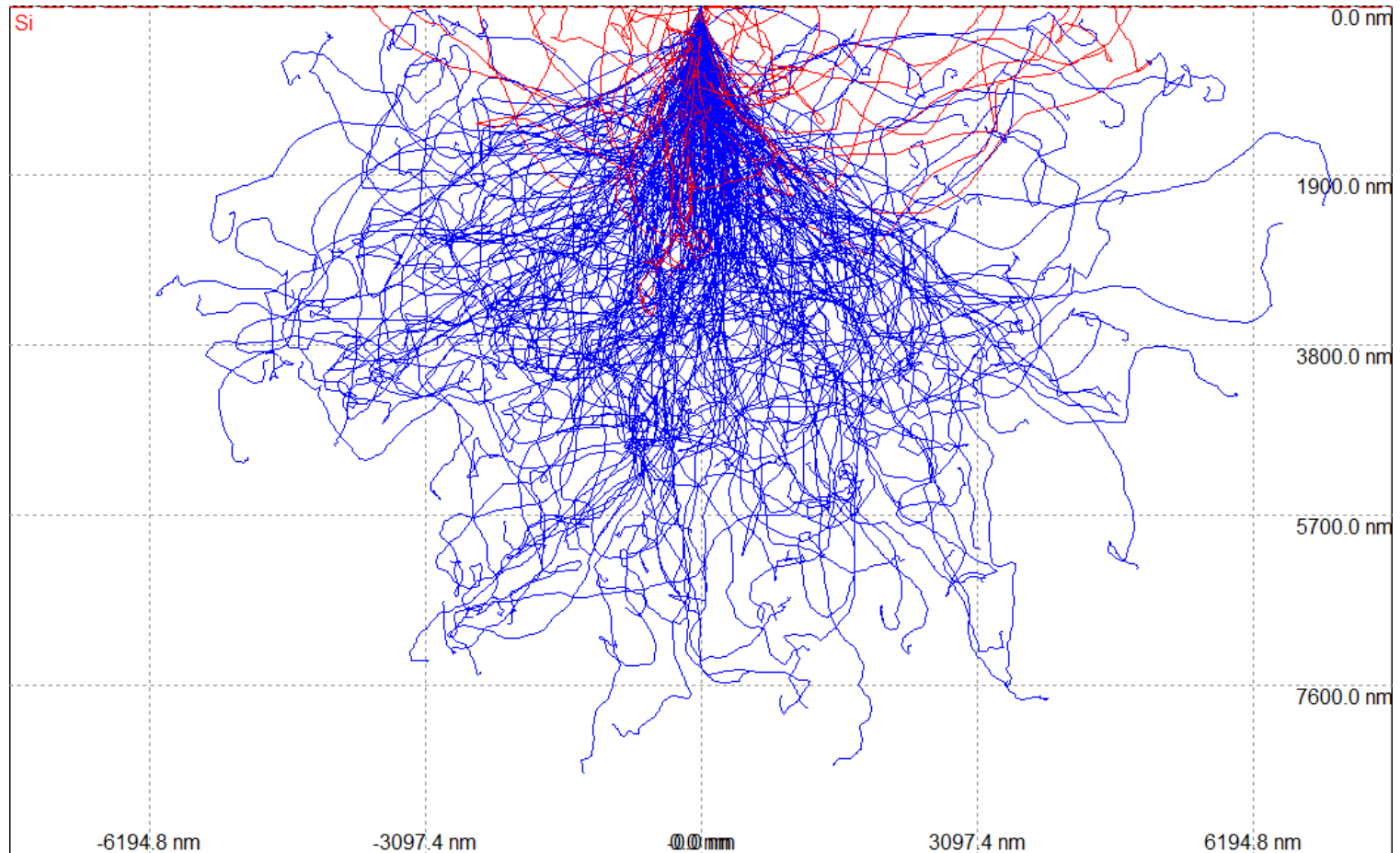
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Canada.

Préparation d'échantillons pour la microscopie électronique à balayage à haute résolution

- Zone SEM pour la décontamination au carbone
- Zone SEM pour sections polies de polymères composites
- Liquide ionique
 - Échantillon biologique
 - Roche
- Polissage ionique Ar⁺
 - Matériaux mous
 - Imagerie bas voltage à haute résolution
- Imagerie des dislocations
- Préparation des lames minces – STEM dans le MEB

CASINO



[CASINO: A new Monte Carlo code in C language for electron beam interaction .1. Description of the program](#)

By: Hovington, P; Drouin, D; Gauvin, R

SCANNING Volume: 19 Issue: 1 Pages: 1-14 Published: JAN 1997 **355 Citations**

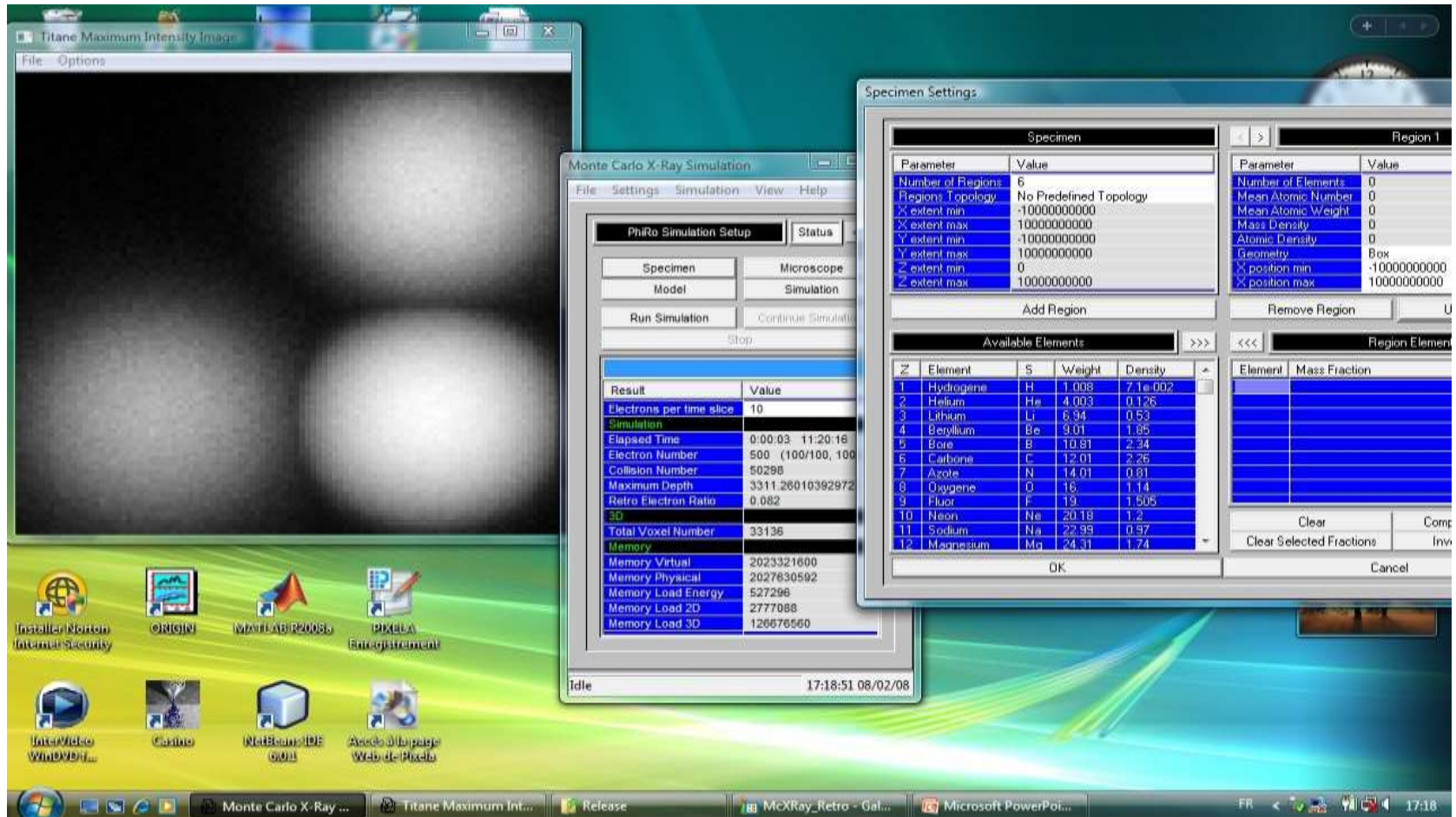
[CASINO V2.42 - A fast and easy-to-use modeling tool for scanning electron microscopy and microanalysis users](#)

By: Drouin, D.; Couture, A. R.; Joly, D. and Gauvin, R.

SCANNING Volume: 29 Issue: 3 Pages: 92-101 Published: MAY-JUN 2007 **479 Citations**

MC X-Ray: Nouveau Programme

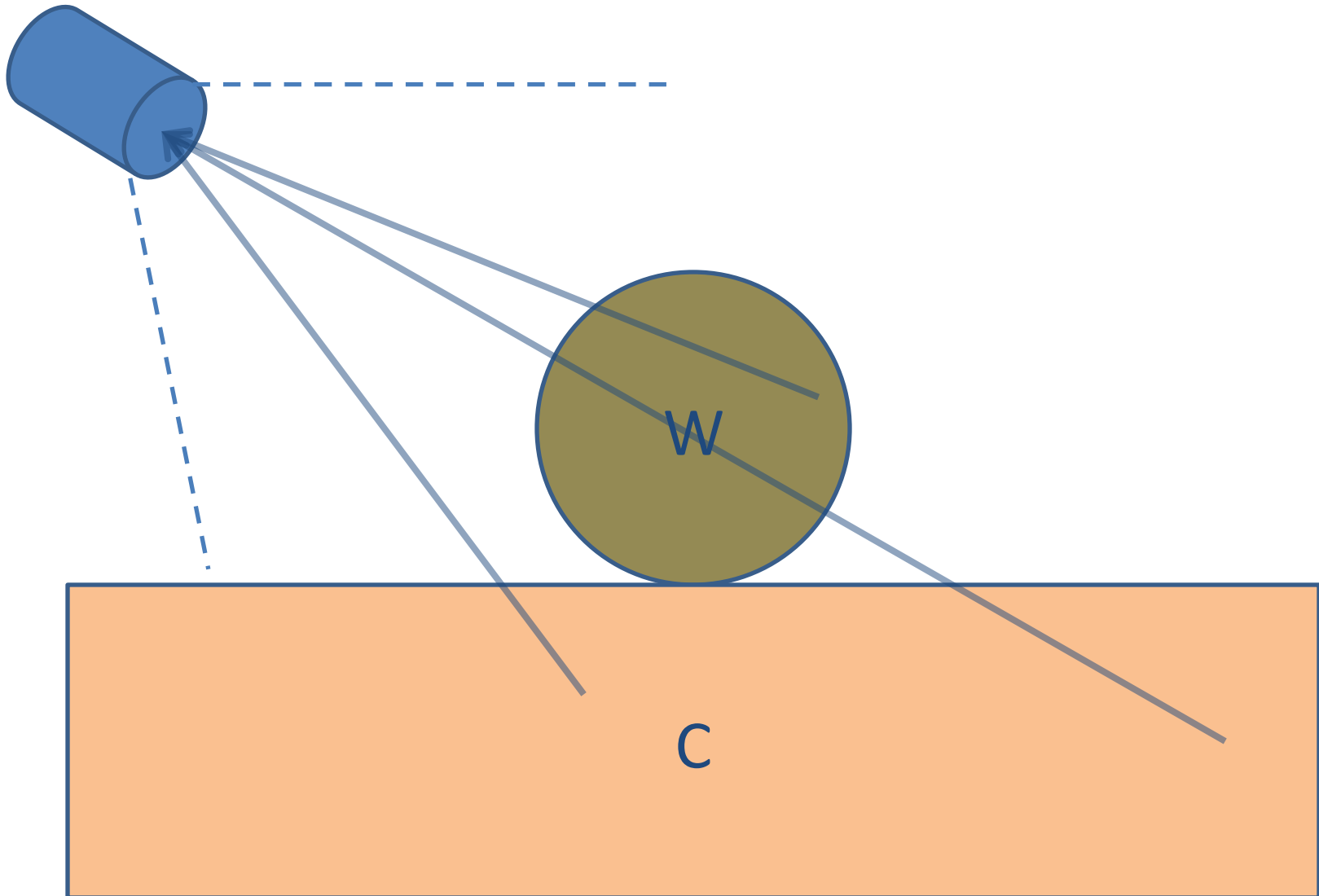
Monte Carlo memrg.com



R. Gauvin and P. Michaud (2009), "MC X-Ray, a New Monte Carlo Program for Quantitative X-Ray Microanalysis of Real Materials", *Microscopy and Microanalysis*, 15 (Supp.2), p. 488-489.

Results – Map – W on C

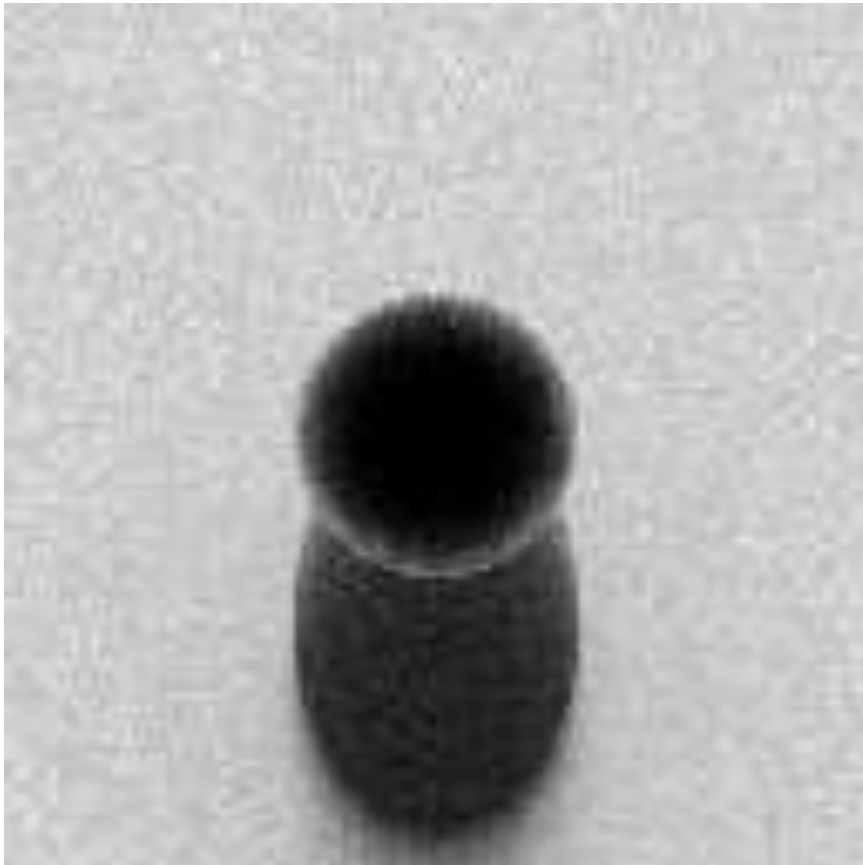
1 μm W Sphere on C, 20 keV, 40° TOA



Results – Map – W on C

1 μm W Sphere on C, 20 keV, 40° TOA

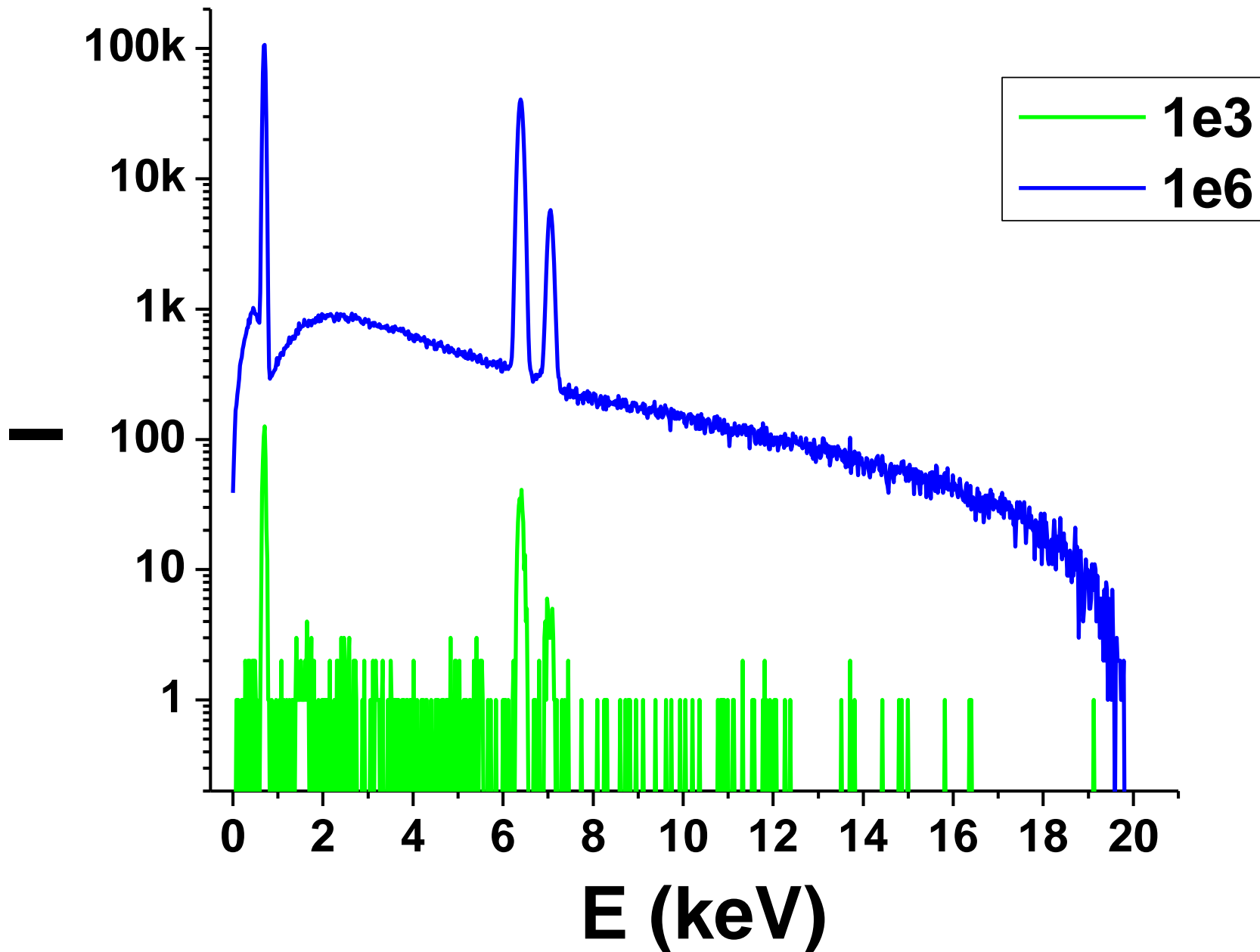
C K α 1



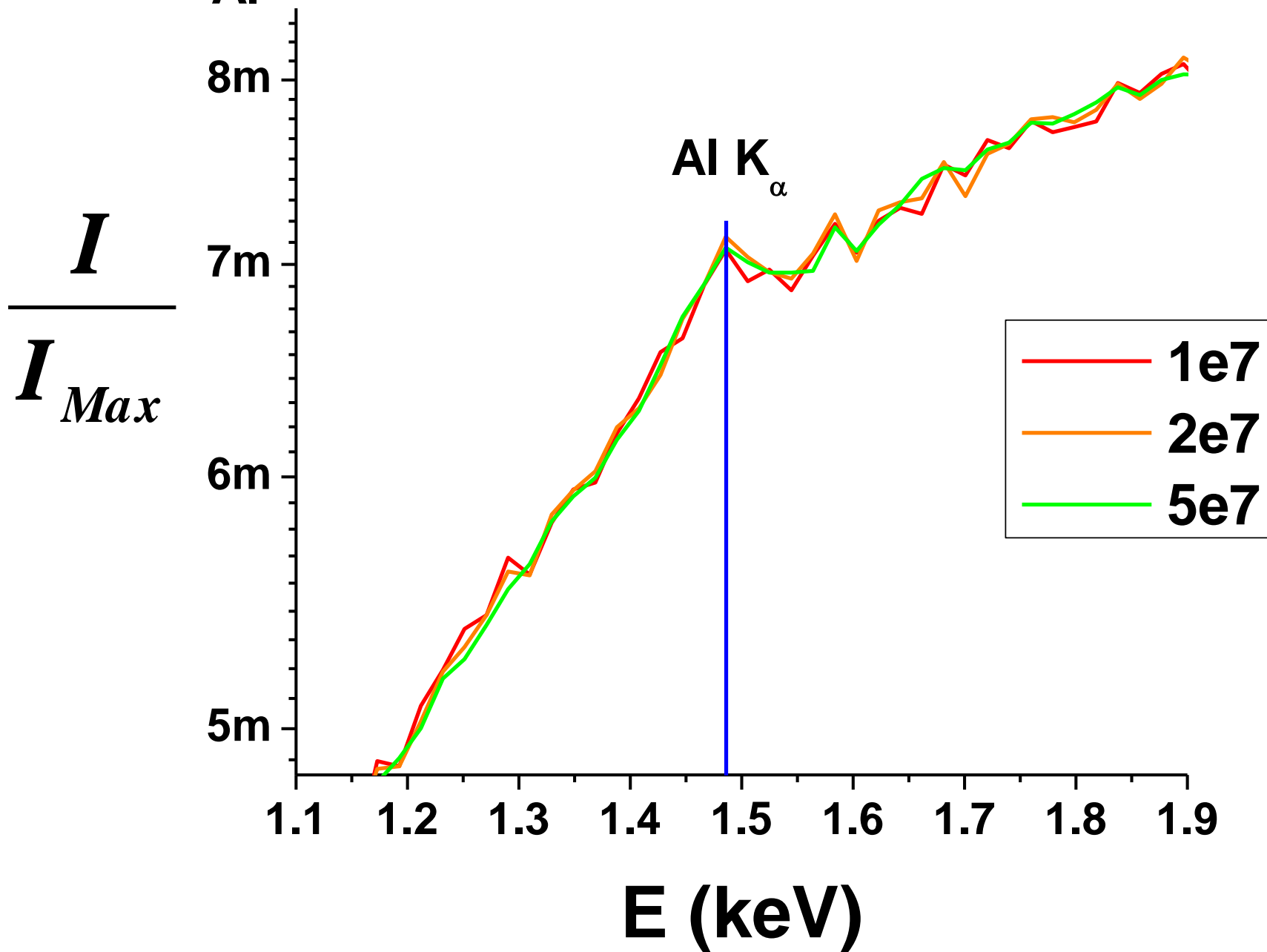
W M α



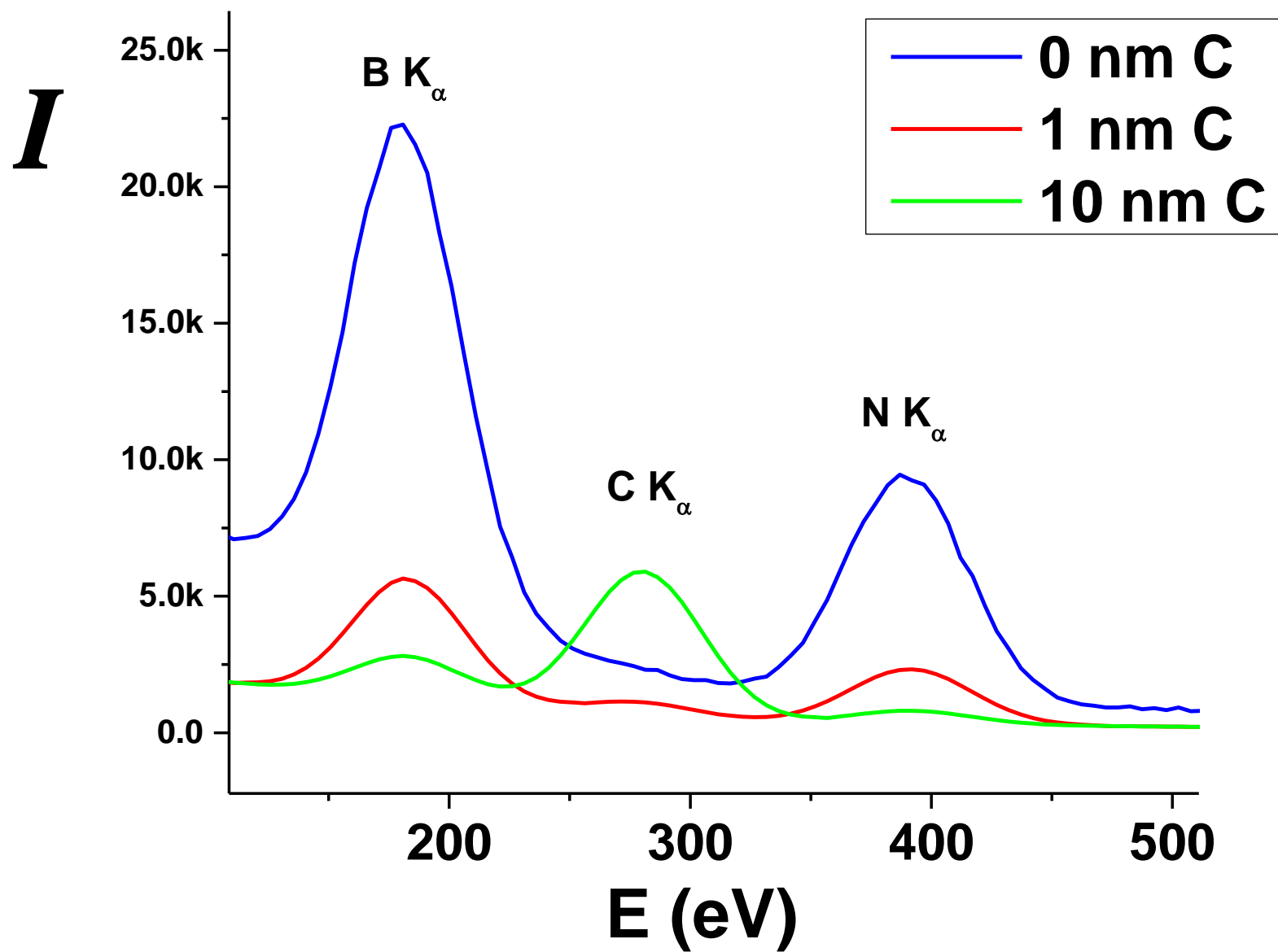
$C_{Al} = 0.0005$, Al - Fe, 20 keV



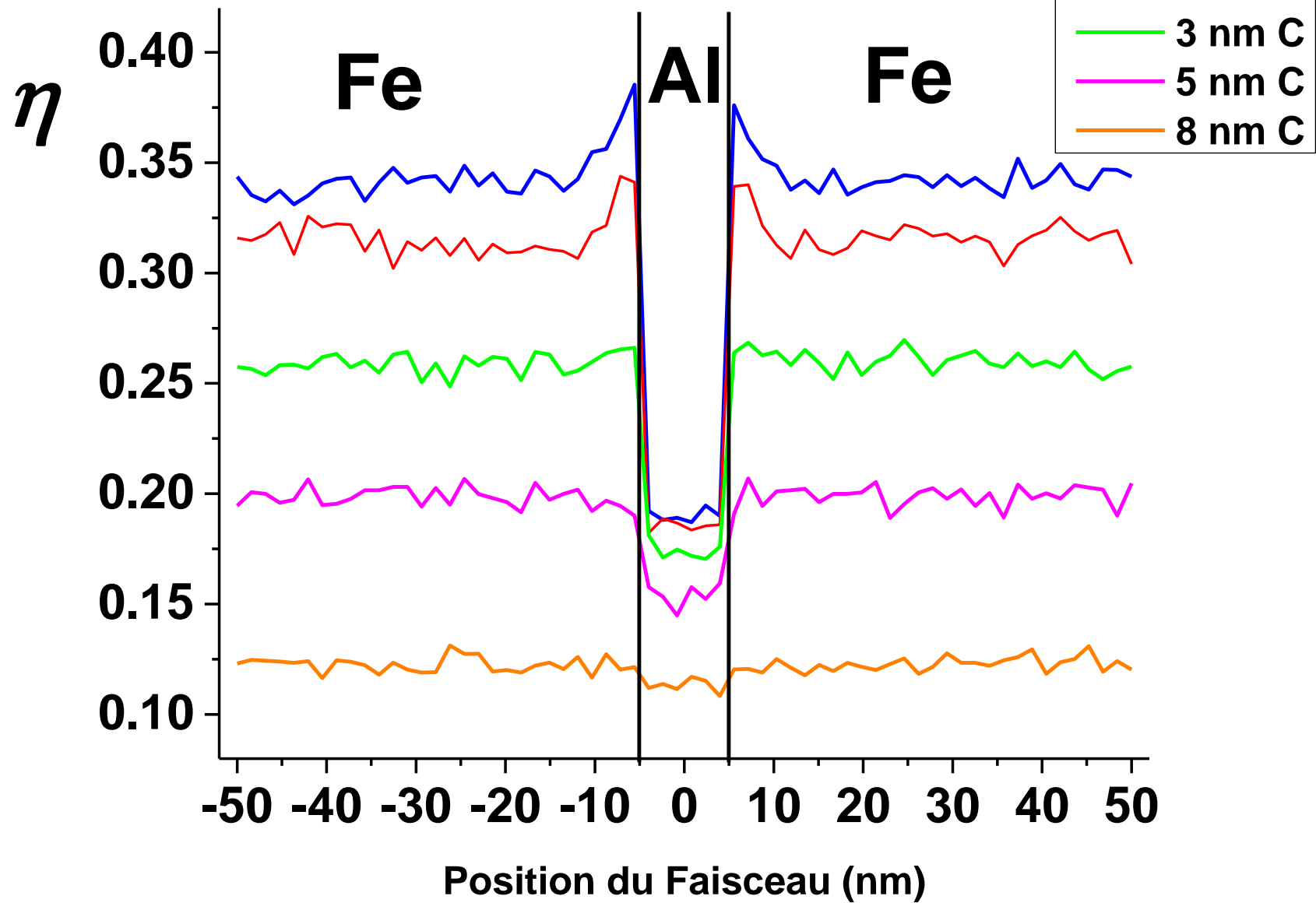
$C_{Al} = 0.0005$, Al - Fe, 20 keV



BN, 1 keV



10 nm Al in Fe, 1 keV, D = 4.3 nm



Low Voltage SEM

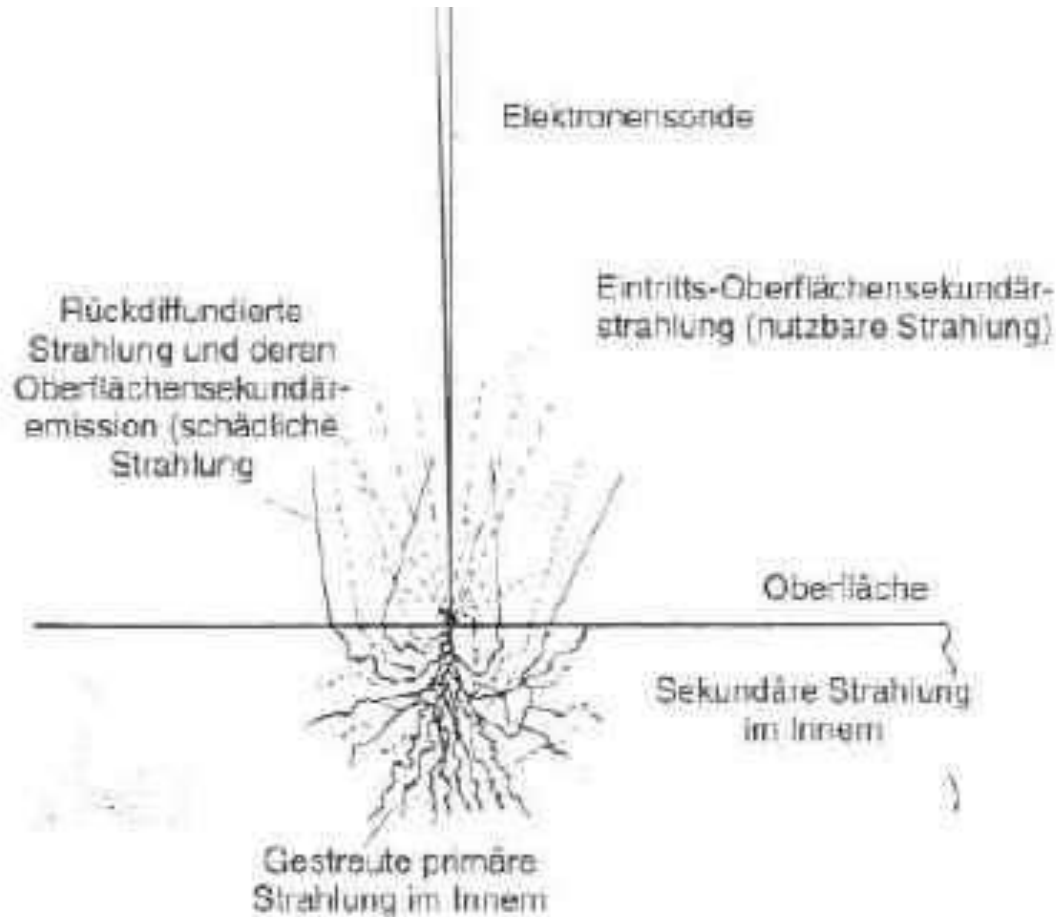


FIG. 6 Diagram illustrating von Ardenne's (1940) discussion of secondary electron imaging of a surface.

Manfred
Von Ardenne
1940

Proposed for High
Resolution

1. Low voltage
SEM
2. High Voltage
SEM

Microscopes à Émission de Champs Froid – Groupe du Pr. Gauvin

Hitachi SU-8000 (2010)



0.5 nm à 30 keV
2 nm à 0.2 keV

Hitachi SU-8230 (2014)

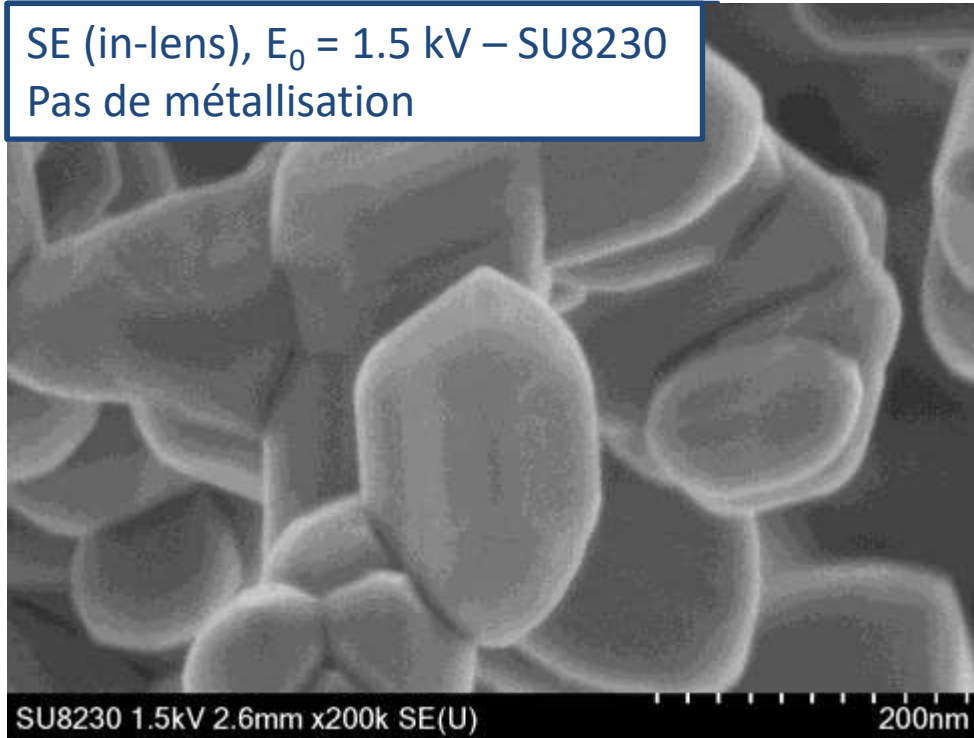


0.4 nm à 30 keV,
1.8 nm à 0.2 keV

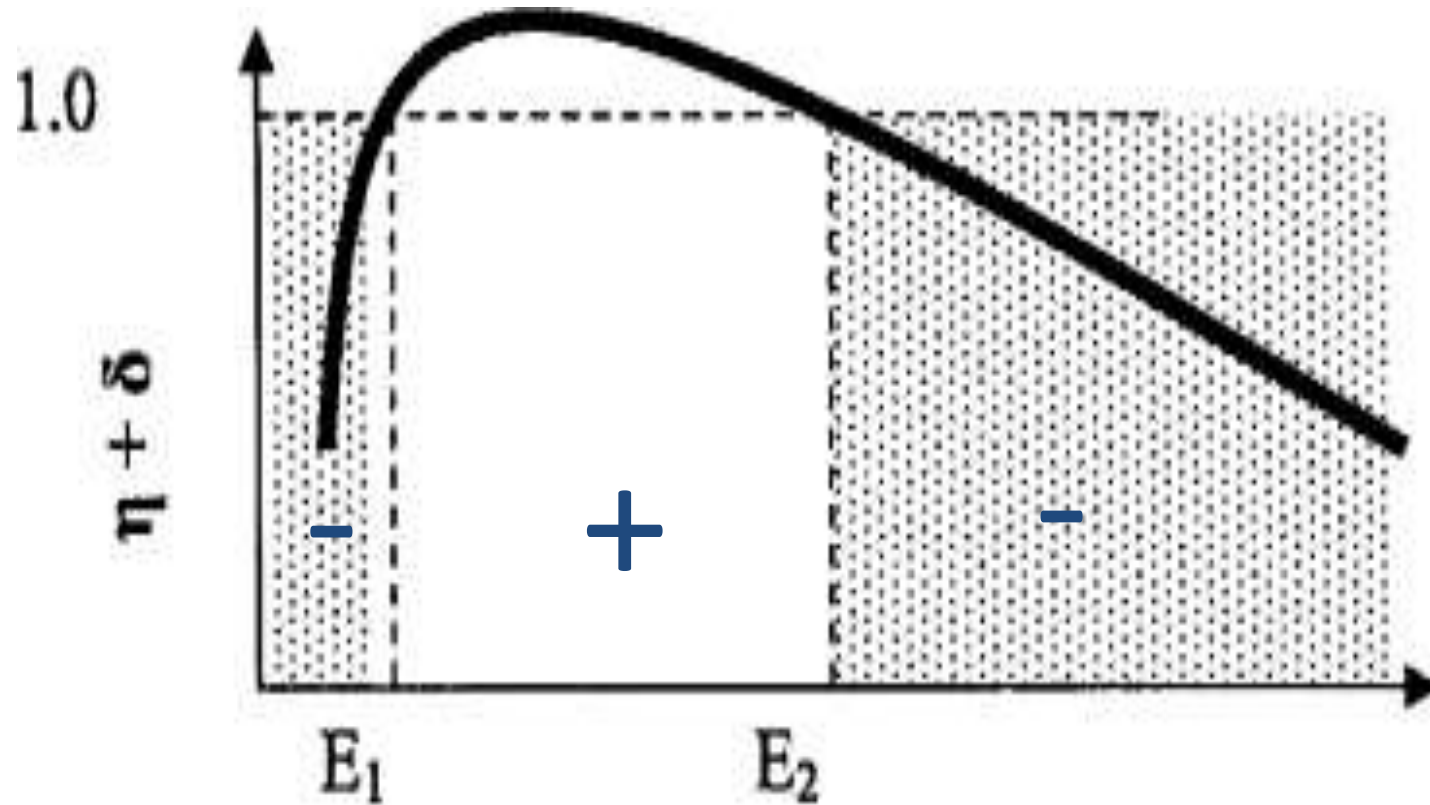
**LA MÉTALLISATION DE
SURFACE POUR
L'IMAGERIE À HAUTE-
RÉSOLUTION?**

Pourquoi il ne faut pas métalliser les échantillons pour l'imagerie haute résolution?

SE (in-lens), $E_0 = 1.5 \text{ kV}$ – SU8230
Pas de métallisation



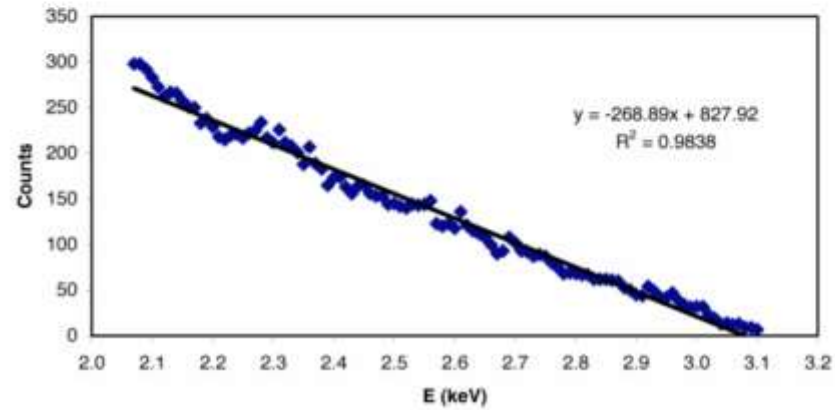
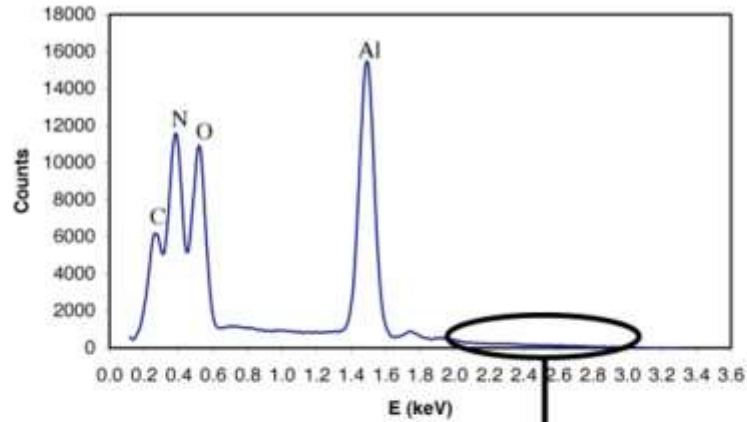
No – Charging E_2 Energy



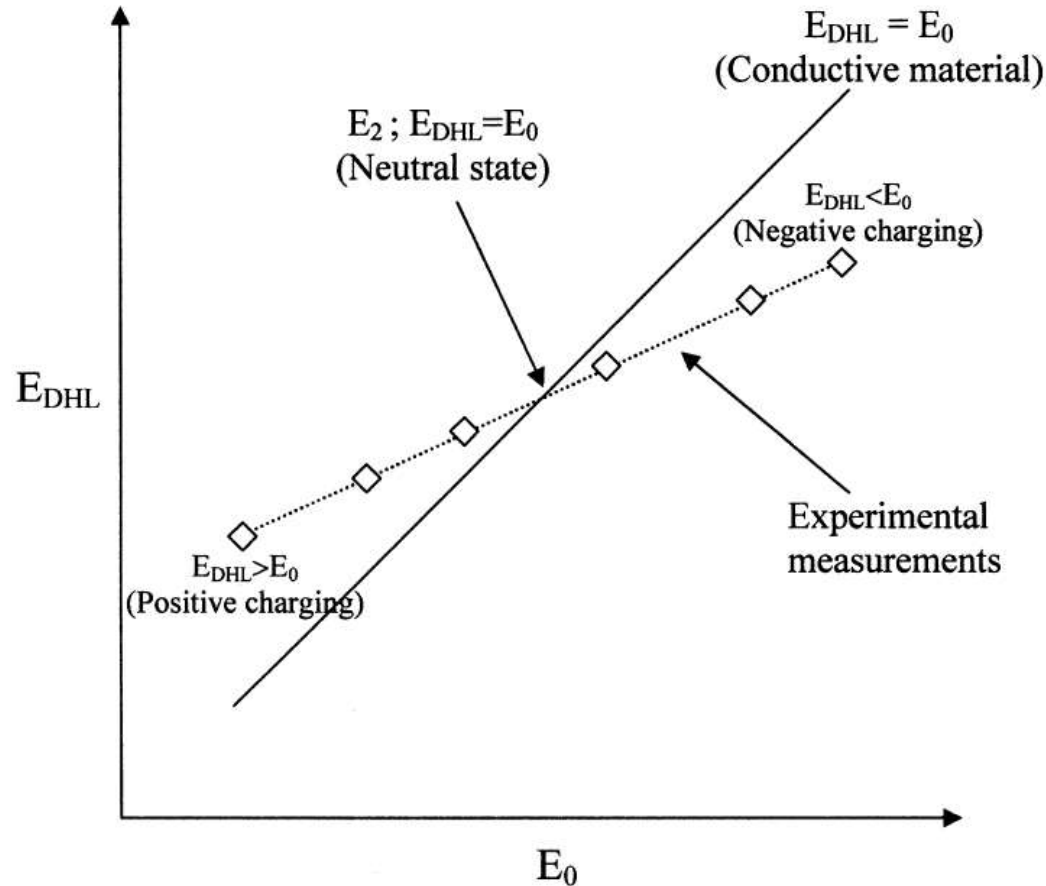
Beam Energy, keV

**E_2 Energies – Flat Surface
Polymers 0.4 – 2 keV
Ceramics 2 – 4 keV**

Duane – Hunt Limit



E_2 Measurement with Duane – Hunt Limit

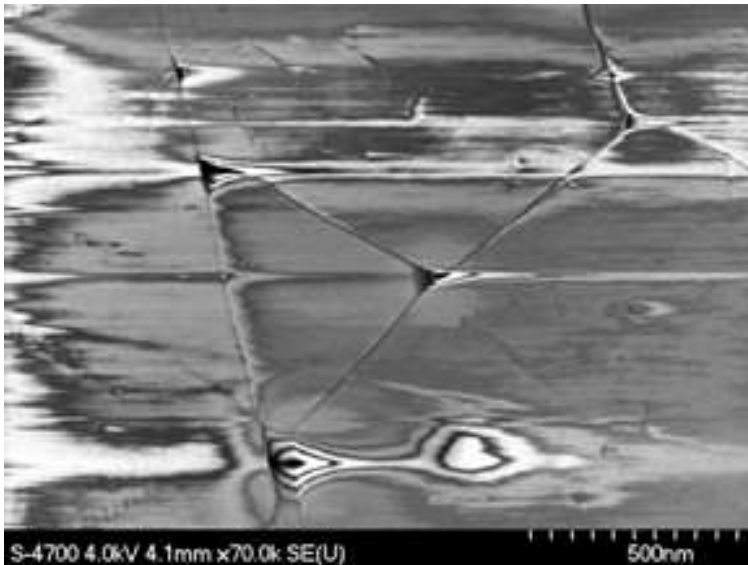
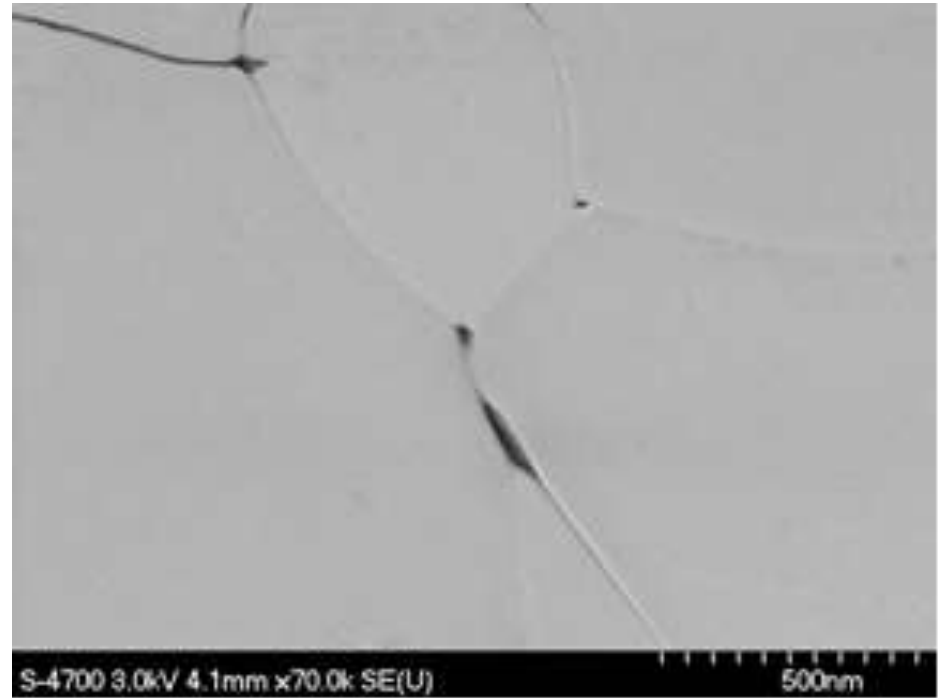


M. Brochu, H. Demers and R. Gauvin (2005), *Microscopy & Microanalysis*, 11, pp. 56 – 65.

2.5 keV

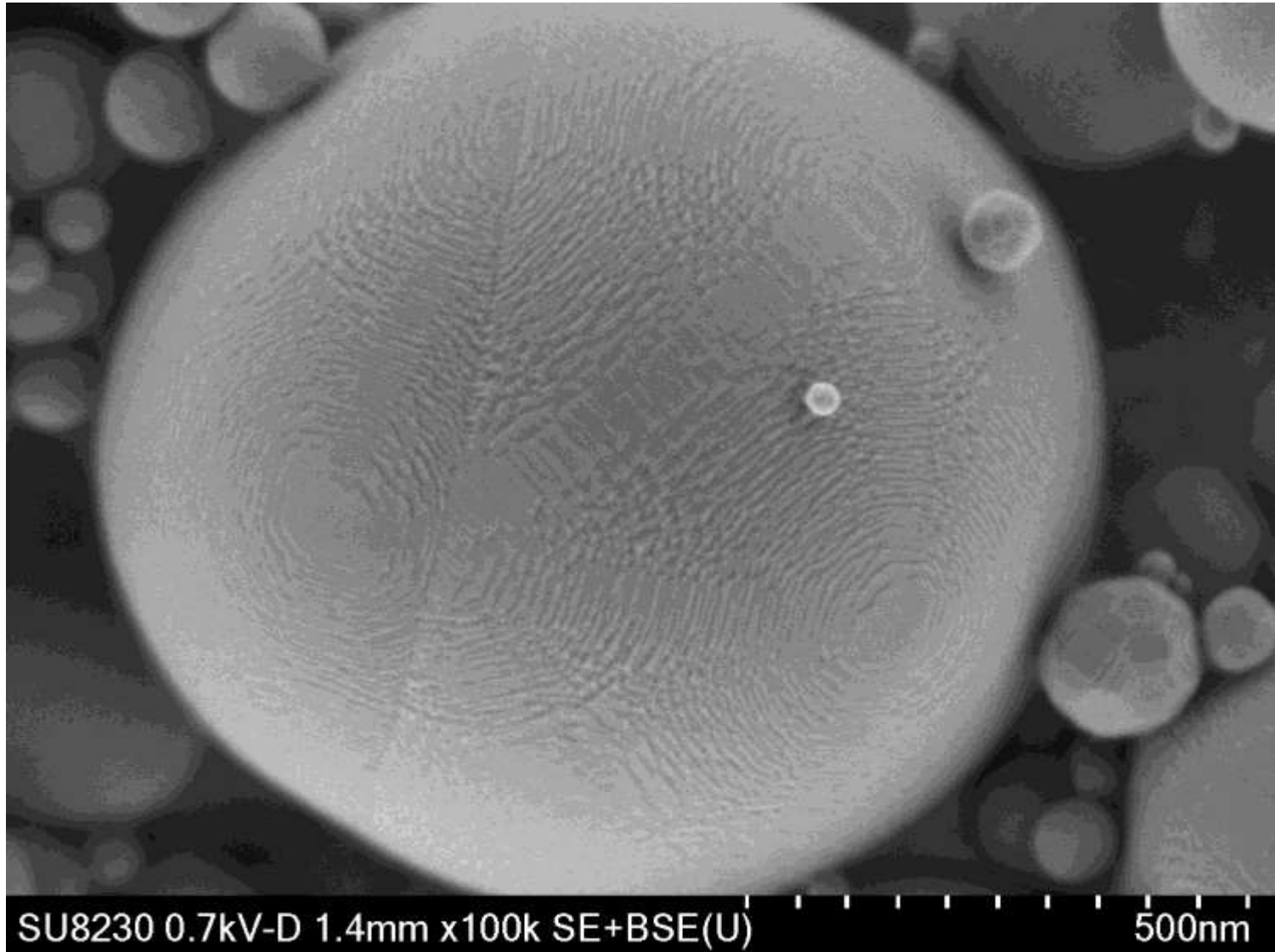


3 keV



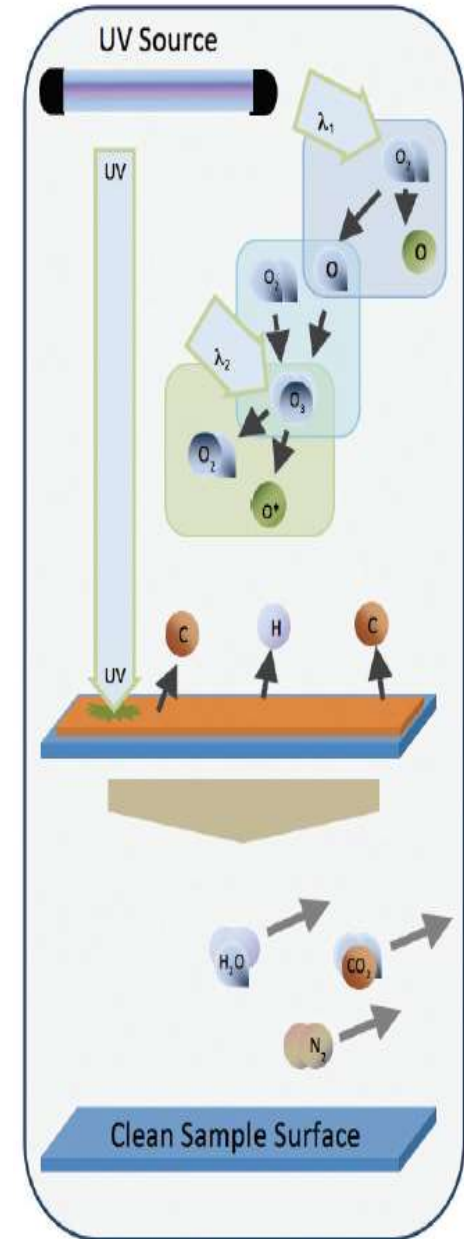
4 keV

Alumina, 0.7 keV



TECHNOLOGIE OZONE / UV (ZONECLEANER)

ZoneCleaner: Décontamination par rayons UV



¹ Soong, C., Woo, P., & Hoyle, D. (2012). Contamination cleaning of TEM/SEM samples with the ZONE cleaner. *Microscopy Today*, 20(06), 44-48.

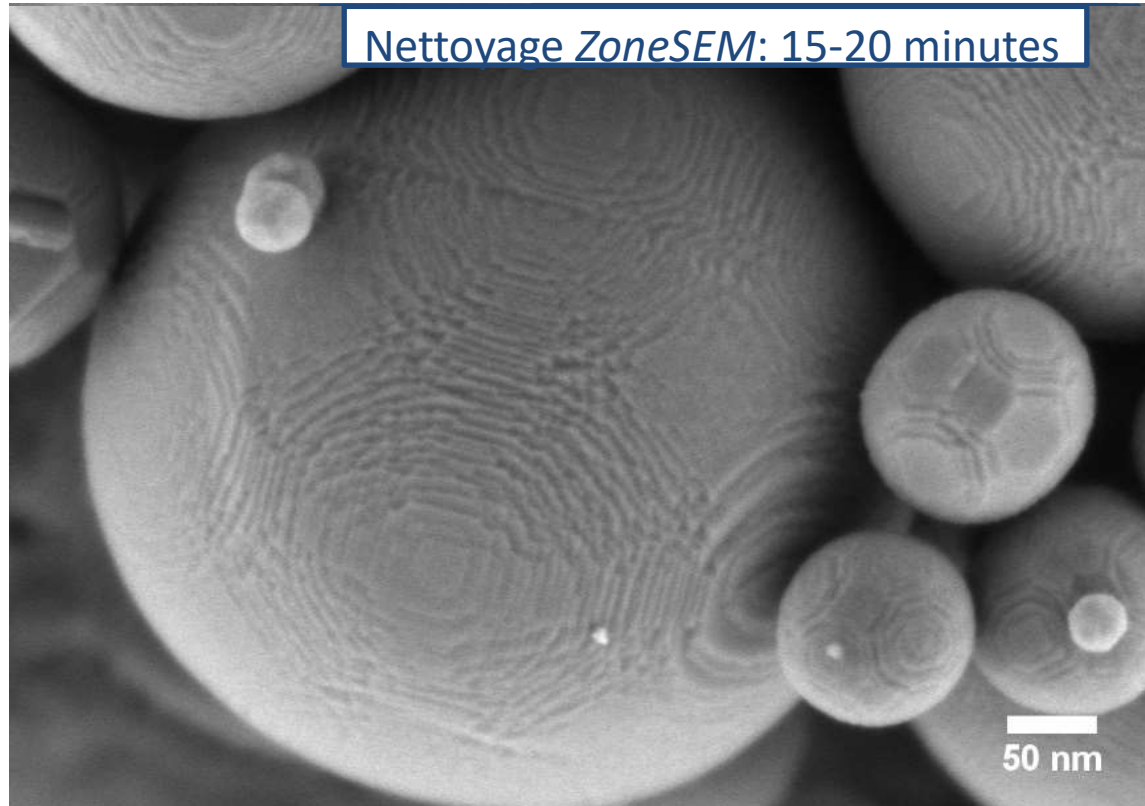
ZoneCleaner (*ZoneSEM*, Hitachi): Décontamination par rayons UV

Échantillon:

Nano-sphères
d'alumine

Préparation:

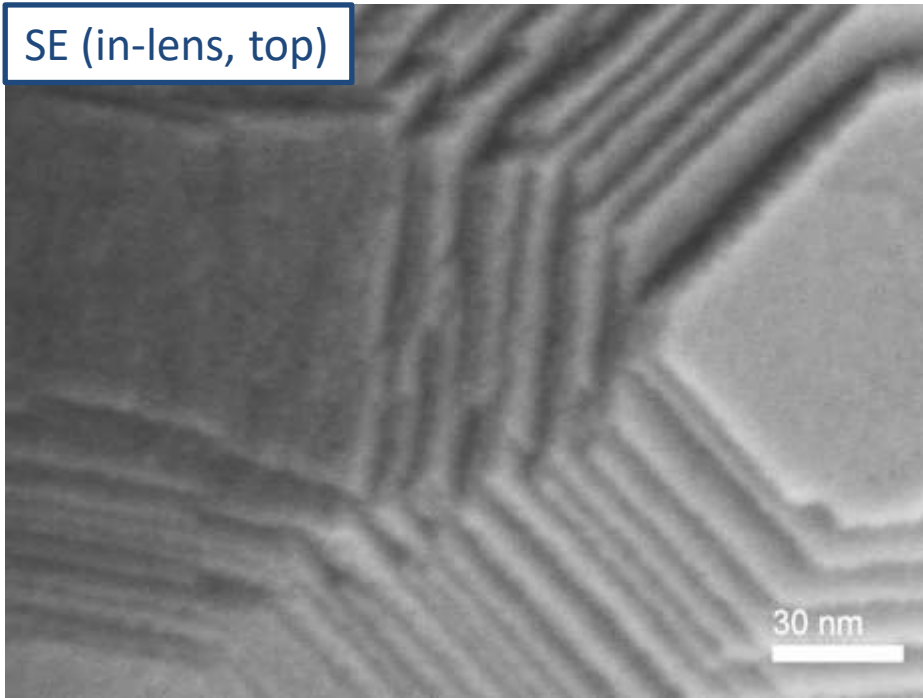
Dispersion dans
l'éthanol +
ultrasons



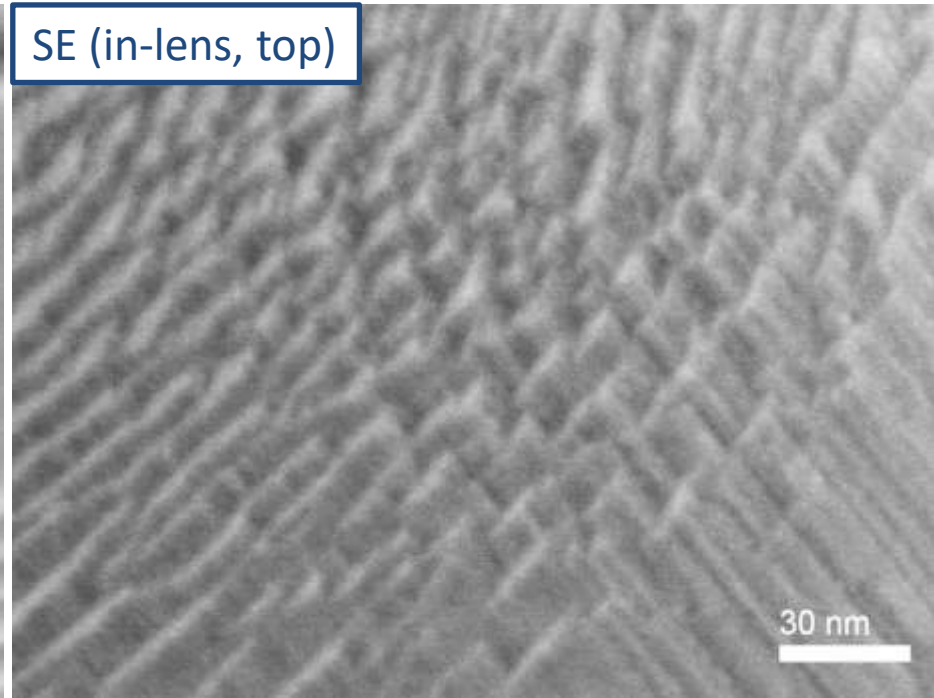
ZoneCleaner (*ZoneSEM*, Hitachi): Décontamination par rayons UV

Imagerie bas voltage en mode décélération, $E_L = 700$ V

SE (in-lens, top)



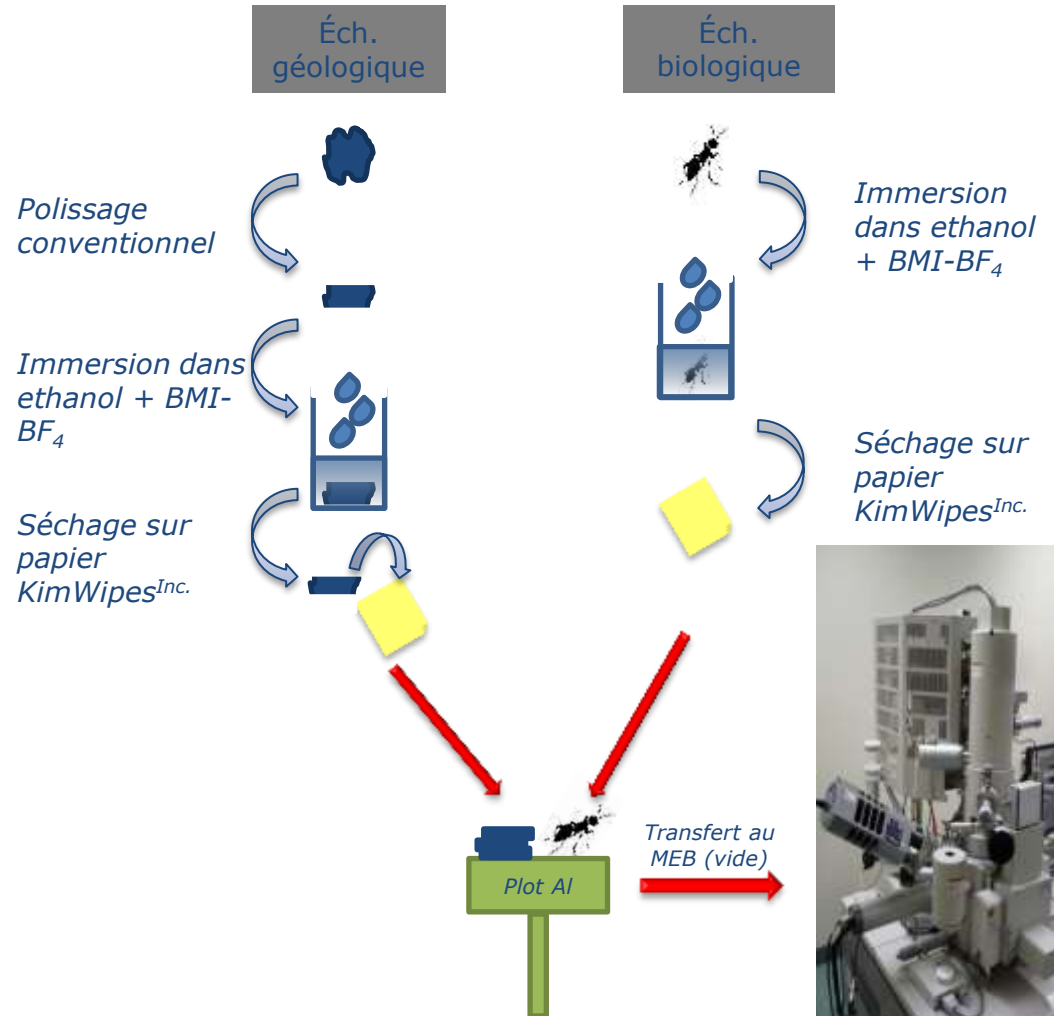
SE (in-lens, top)



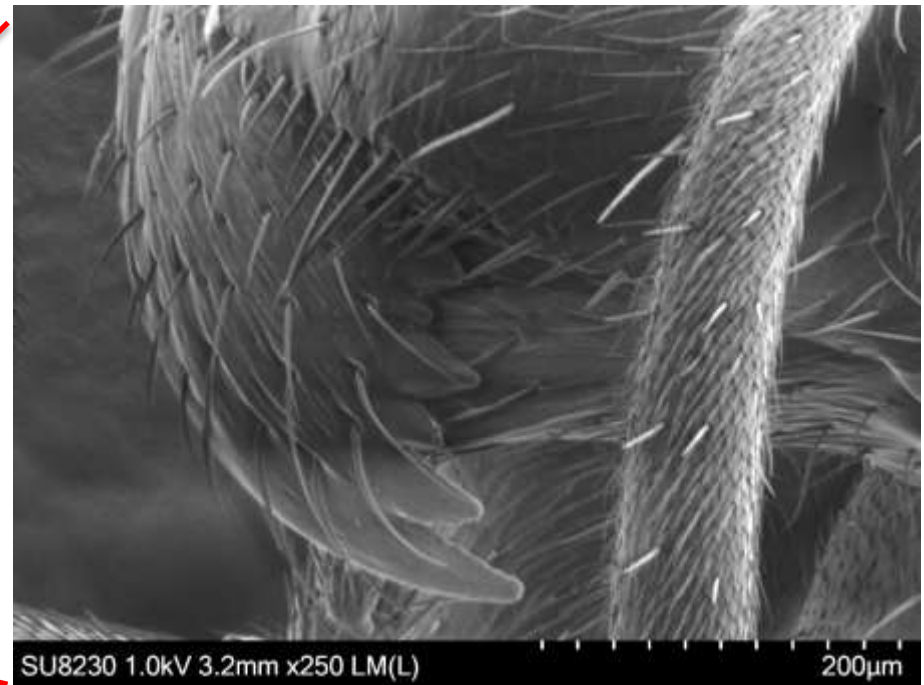
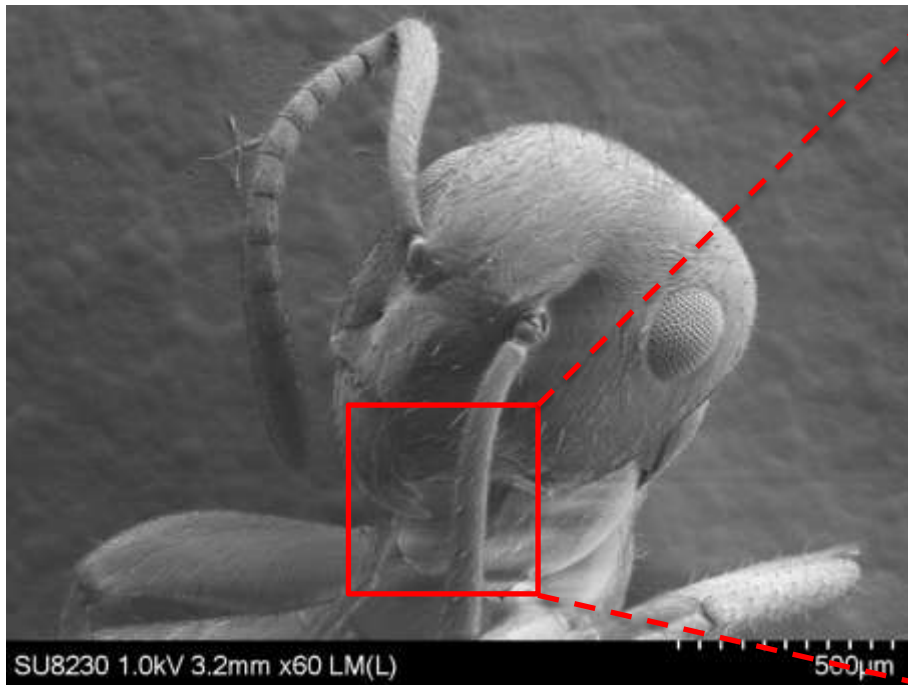
**APPORT DU LIQUIDE IONIQUE
POUR LA PRÉPARATION
D'ÉCHANTILLONS NON-
CONDUCTEURS ET HUMIDES**

Liquide ionique pour la préparation d'échantillons: Principe

- Liquide ionique: 1-butyl-3-méthylimidazolium tetrafluoroborate ($BMI-BF_4$)
- Hydrophile = Bon pour échantillons biologiques humides
- Peu de préparation
- Bon conducteur = Pas besoin de métallisation
- Faible tension de vapeur = Stable sous vide secondaire



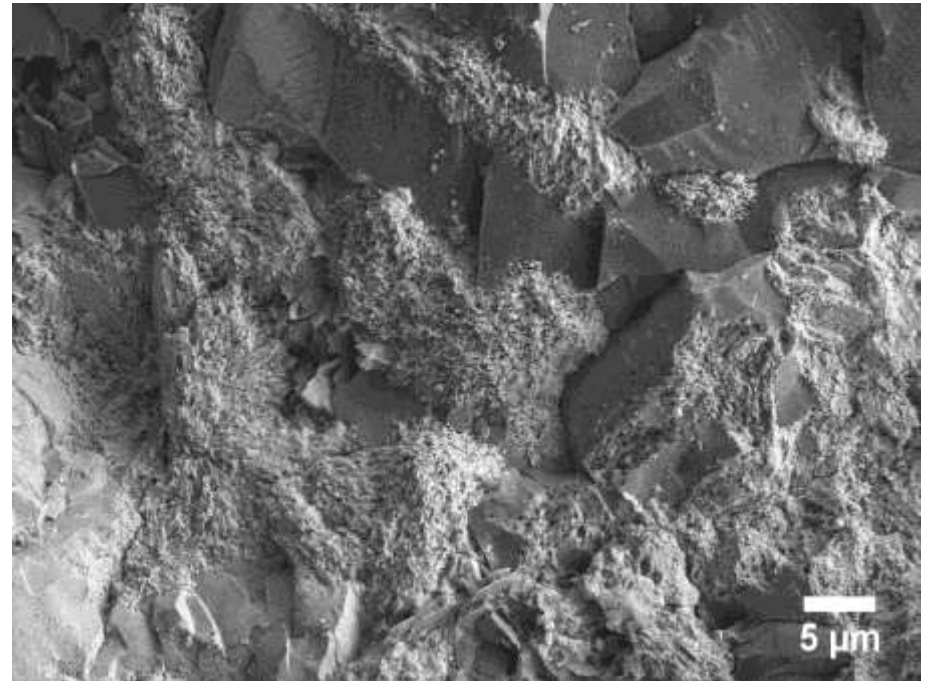
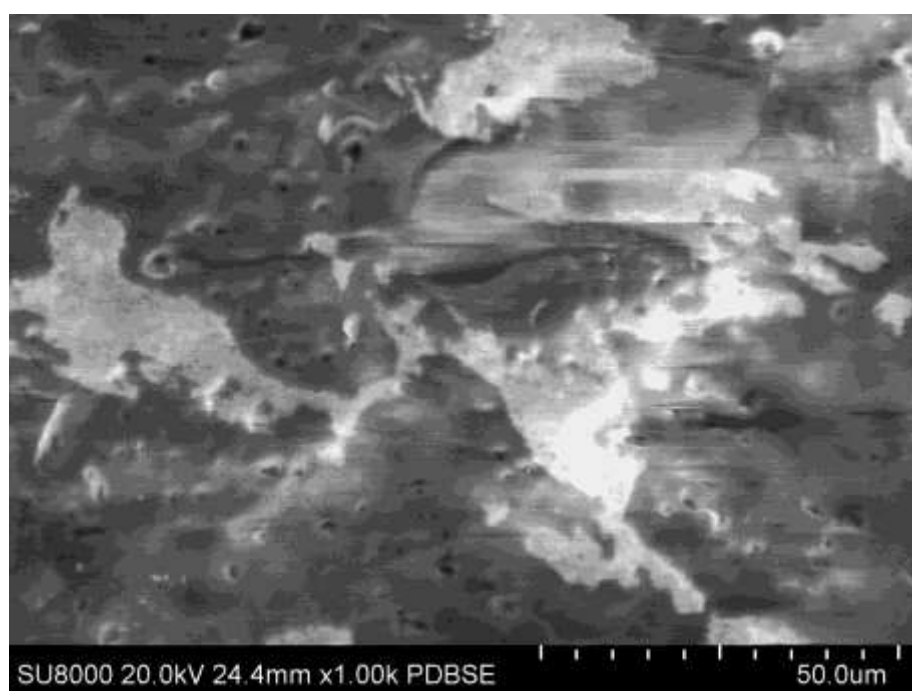
Liquide ionique pour la préparation d'échantillons: Imagerie d'une fourmi



Liquide ionique pour la préparation d'échantillons: Imagerie d'une roche de la mine Nechalacho (Thor Lake, Canada)

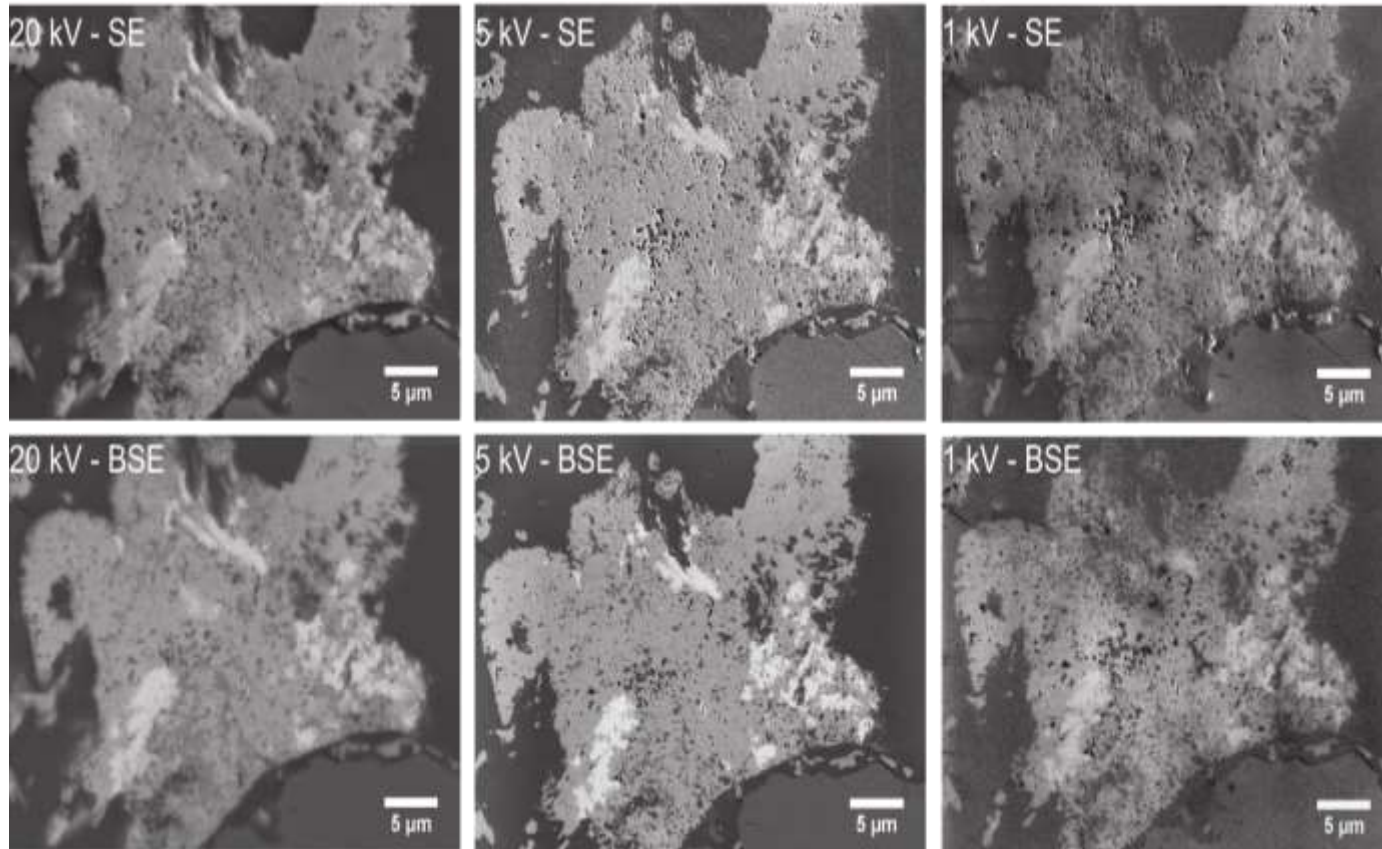
Sans traitement, ni métallisation

Avec traitement au BMI-BF₄



Brodusch, N., Waters, K., Demers, H., & Gauvin, R. (2014). *Microscopy Research and Technique*, 77(3), 225-235.

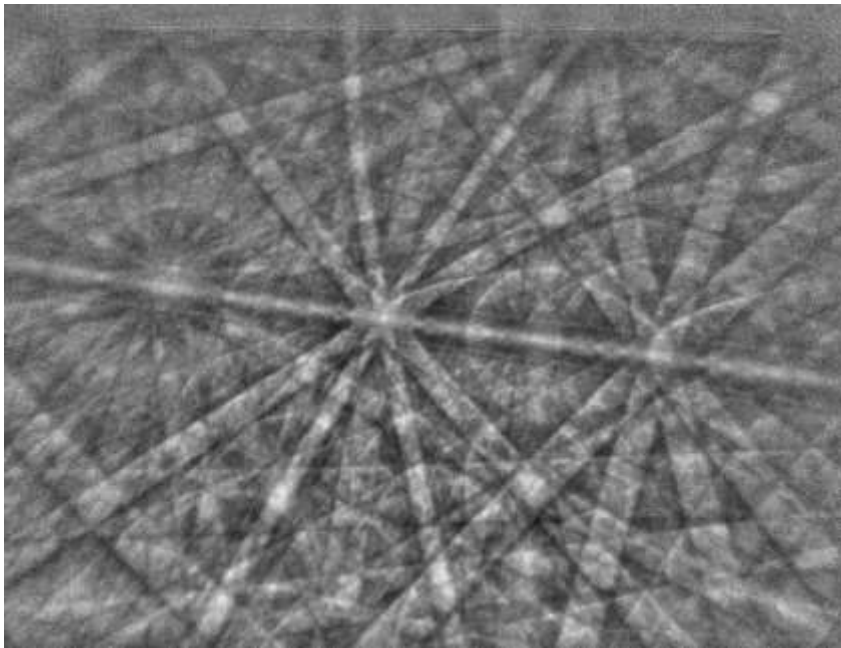
Liquide ionique pour la préparation d'échantillons: Imagerie d'une roche de la mine Nechalacho (Thor Lake, Canada)



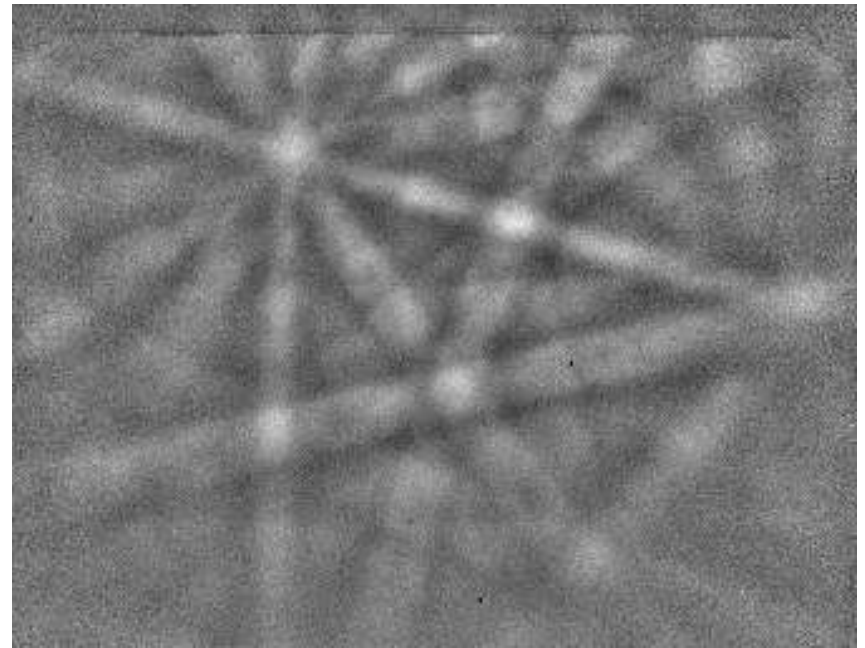
Pas de traces de charge avec 1 kV $< E_0 < 20$ kV!

Liquide ionique pour la préparation d'échantillons: Imagerie d'une roche de la mine Nechalacho (Thor Lake, Canada) - EBSD

Zircone, $E_0 = 20$ kV

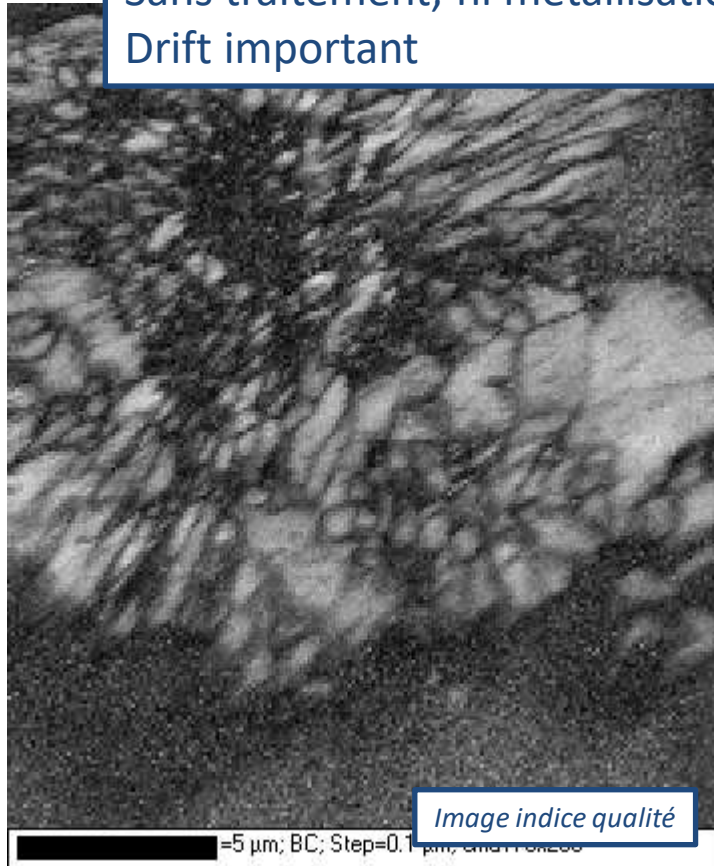


Fergusonite, $E_0 = 5$ kV

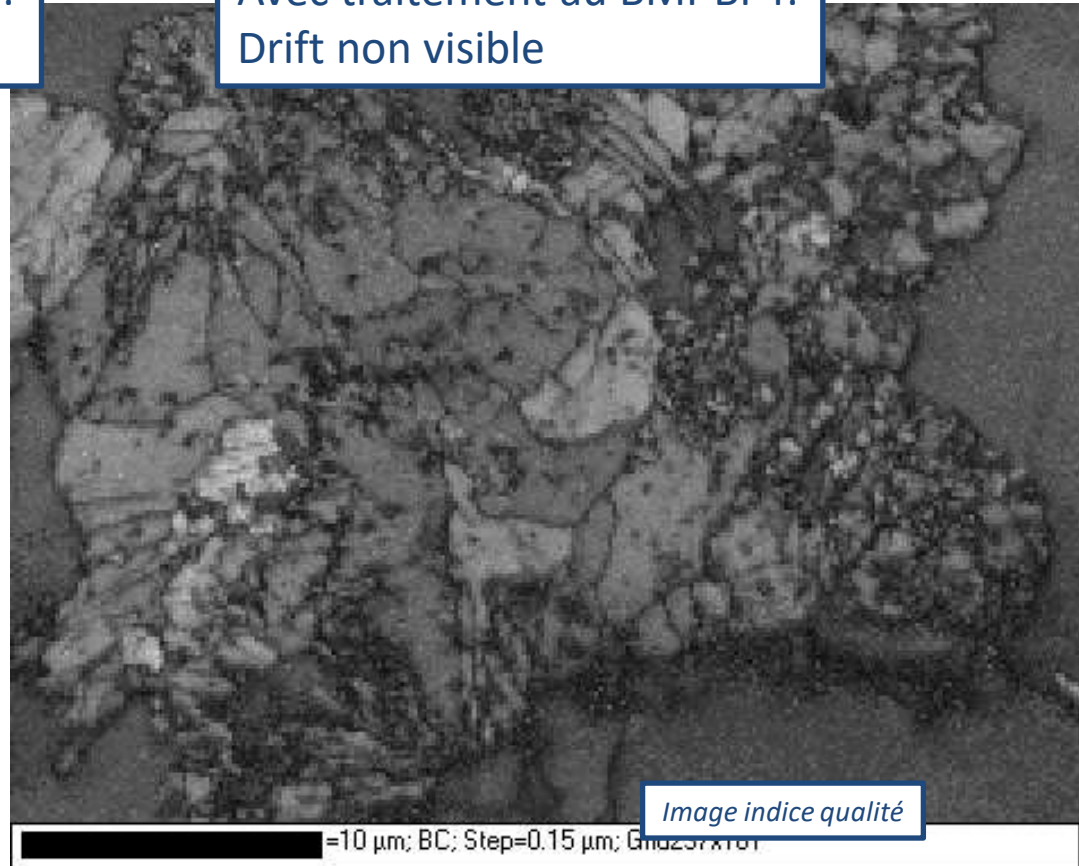


Liquide ionique pour la préparation d'échantillons: Imagerie d'une roche de Nechalacho (Thor Lake, Canada) - EBSD

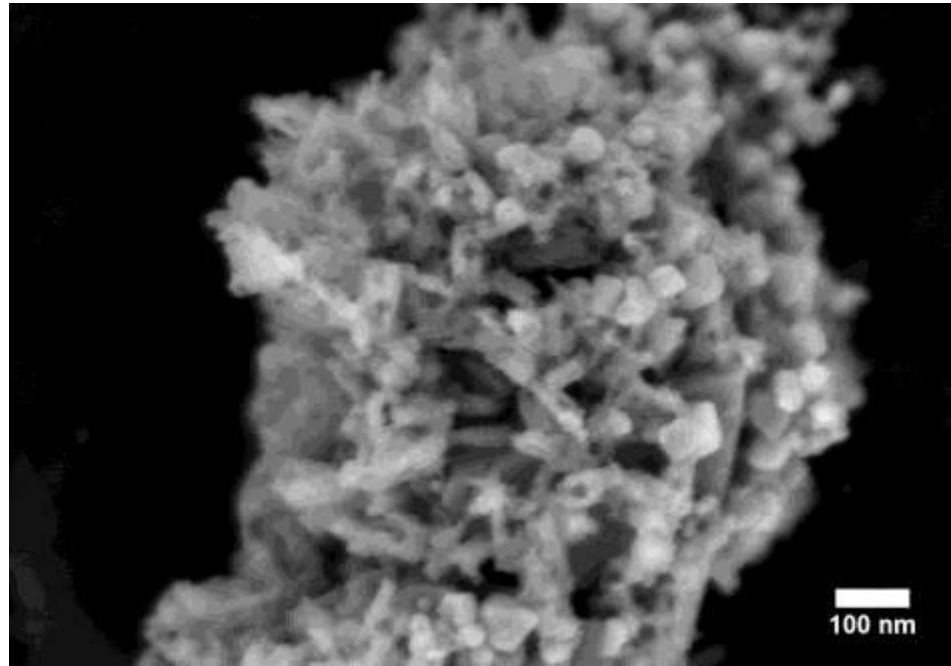
Sans traitement, ni métallisation:
Drift important



Avec traitement au BMI-BF4:
Drift non visible

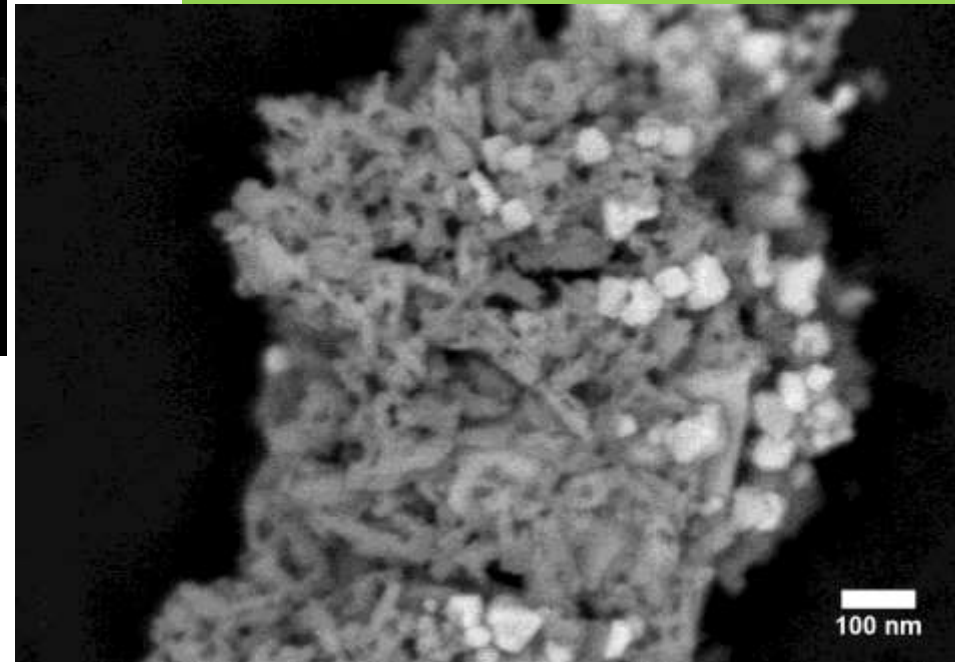


$\text{Li}_2\text{FeSiO}_4$ with filtration ($E_0 = 2.2$ kV) and ionic liquid preparation



Upper detector (filter bias = 8-9 V)

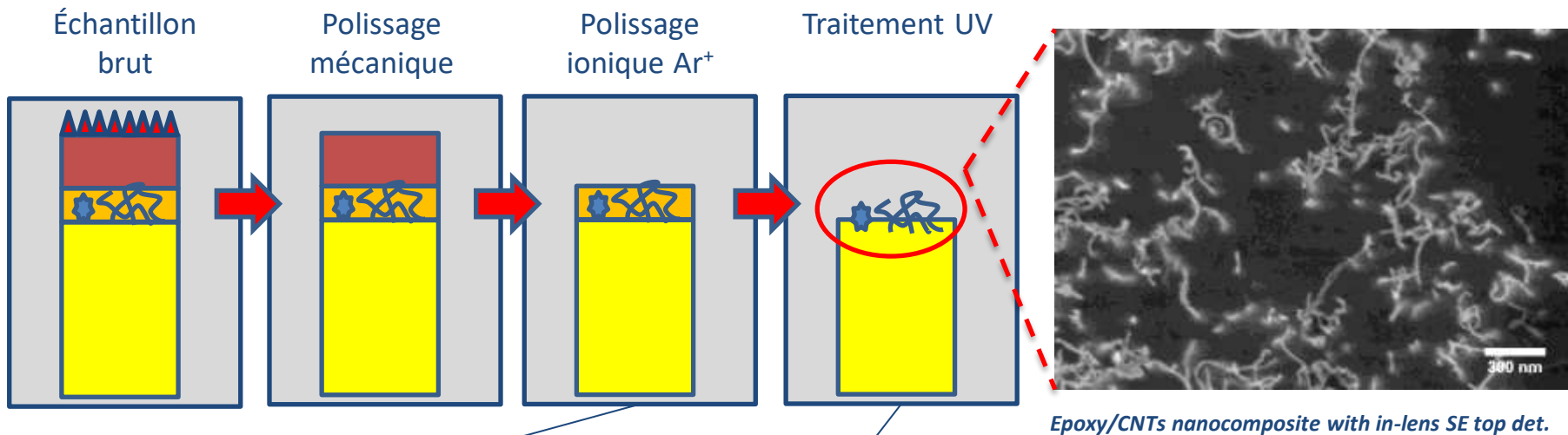
Top detector with filtration (80%)



Ion Milling



ZoneCleaner (*ZoneSEM*, Hitachi): Attaque chimique par rayons UV - Principe



Ion milling IM4000 (Hitachi)



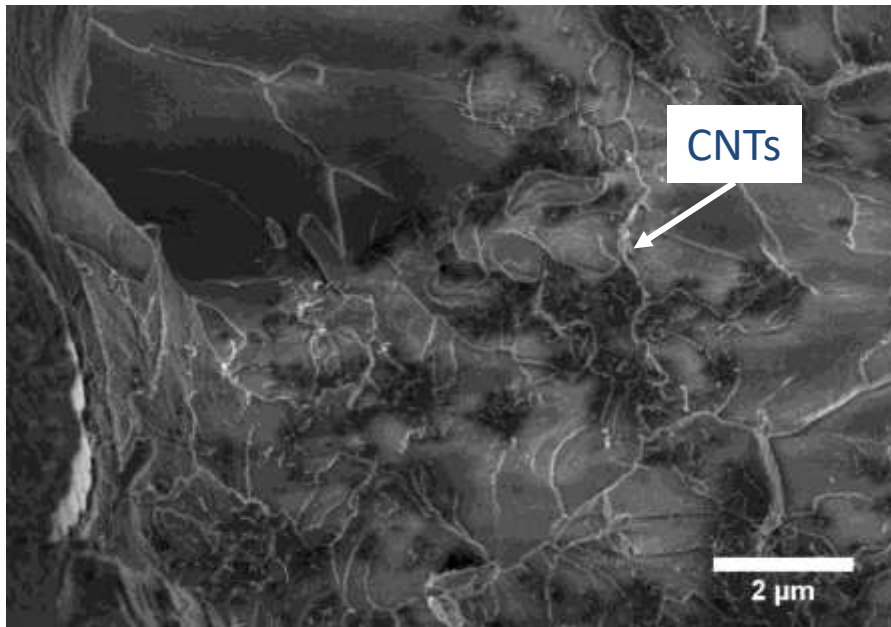
ZoneSEM ozone cleaner (Hitachi)



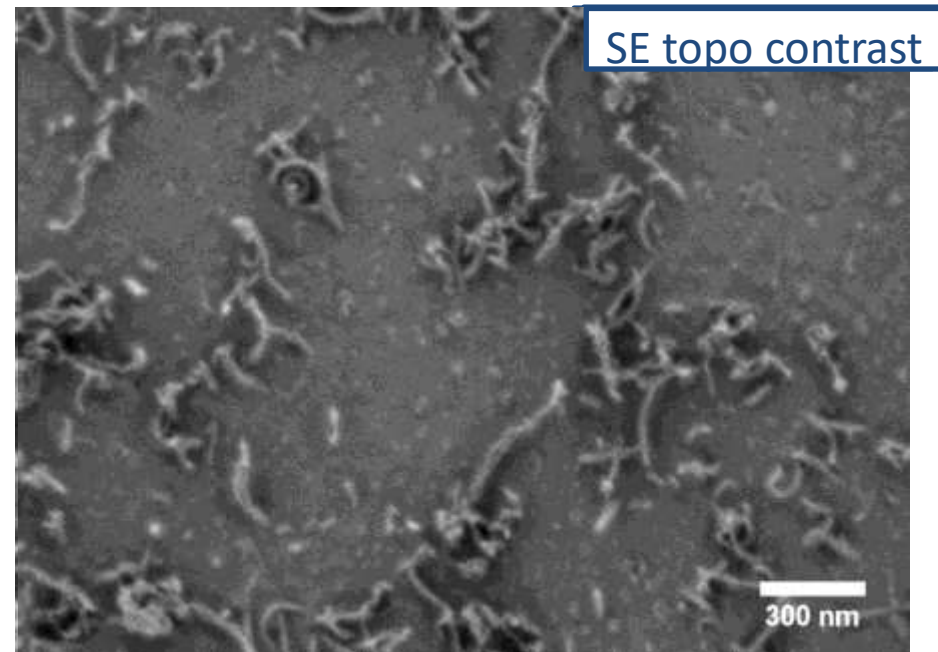
SU8230 cold-field emission SEM (Hitachi)

ZoneCleaner (*ZoneSEM*, Hitachi): Attaque chimique par rayons UV – Epoxy / CNT

Cryo-fracture fragile conventionnelle



Section polie avec notre méthode

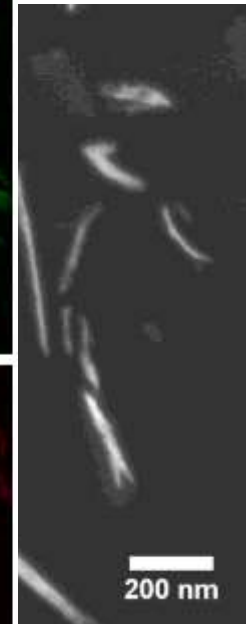
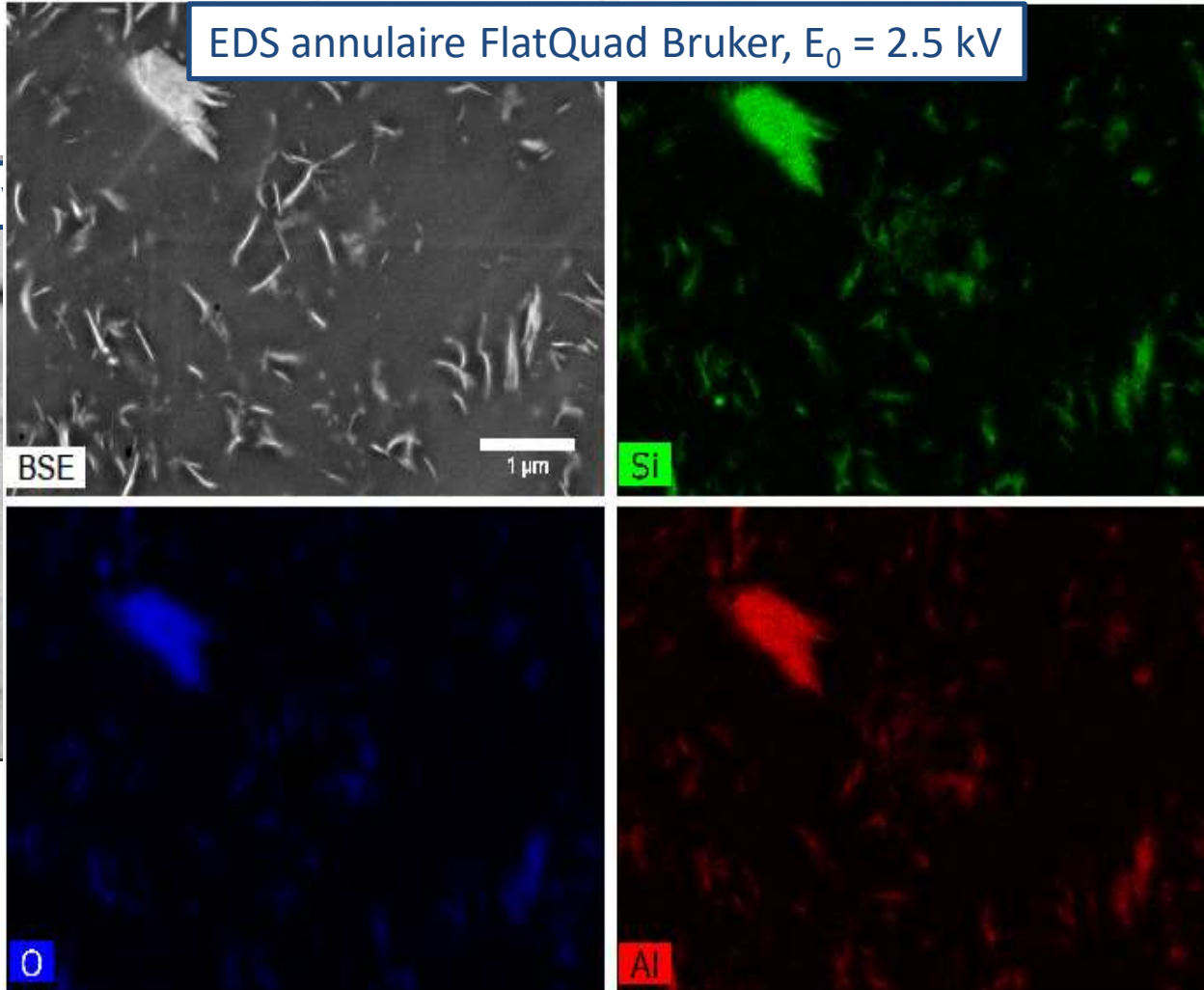


Brodusch, N., Yourdkhani, M., Hubert, P., & Gauvin, R. (2015). *Journal of Microscopy*, 260(2), 117-124.

ZoneCleaner (*ZoneSEM*, Hitachi): Attaque chimique par rayons UV – Polylactic acid (PLA) / nanoclay

EDS annulaire FlatQuad Bruker, $E_0 = 2.5$ kV

STEM-BF, $E_0 = 30$ kV



**LE POLISSAGE IONIQUE
POUR L'IMAGERIE DE
SURFACES À HAUTE
RÉSOLUTION SPATIALE**

Imaging Precipitates at Low E_0

Monte Carlo Predictions (1995)

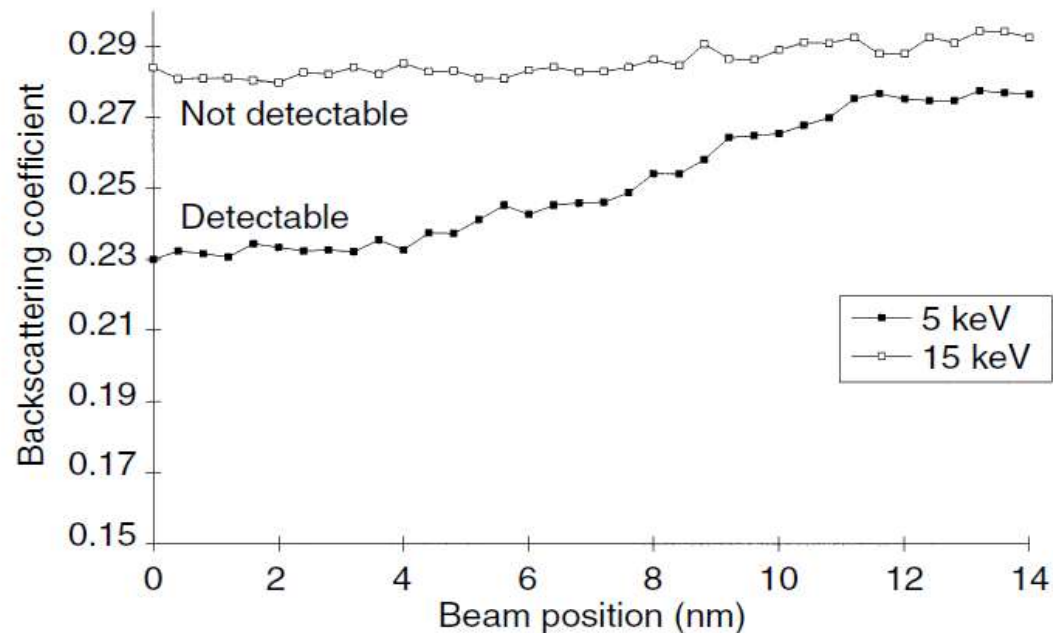
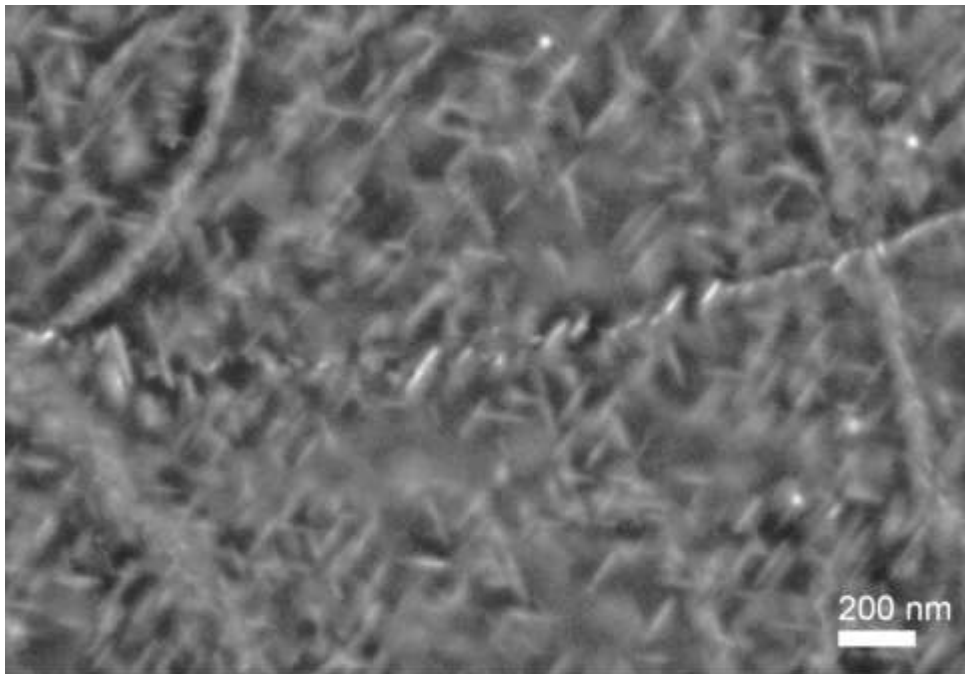
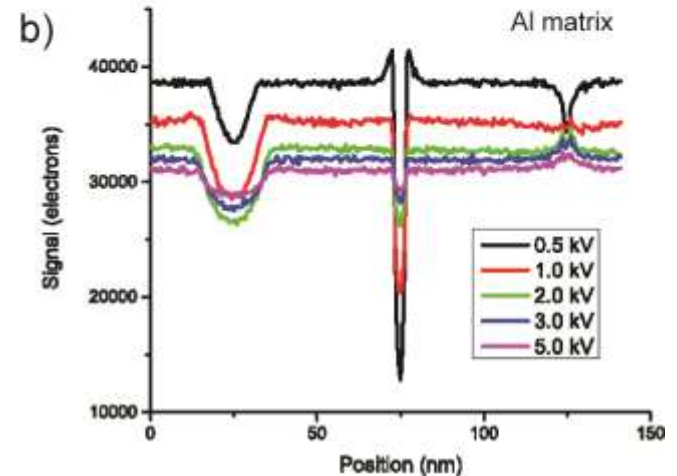
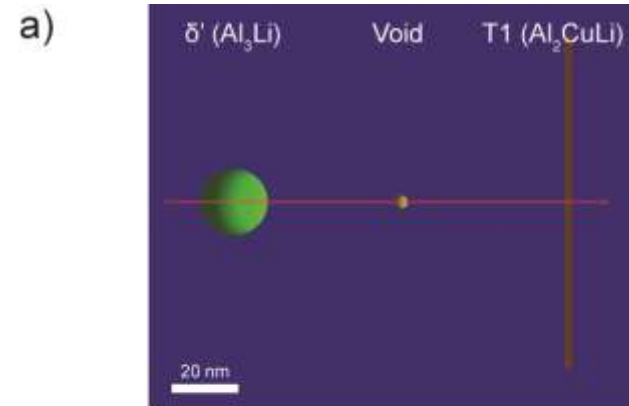


FIG. 24 Simulated profile for a 10 nm radius hemispherical inclusion of the MnS/Fe system at $E_0 = 5$ and 15 keV.

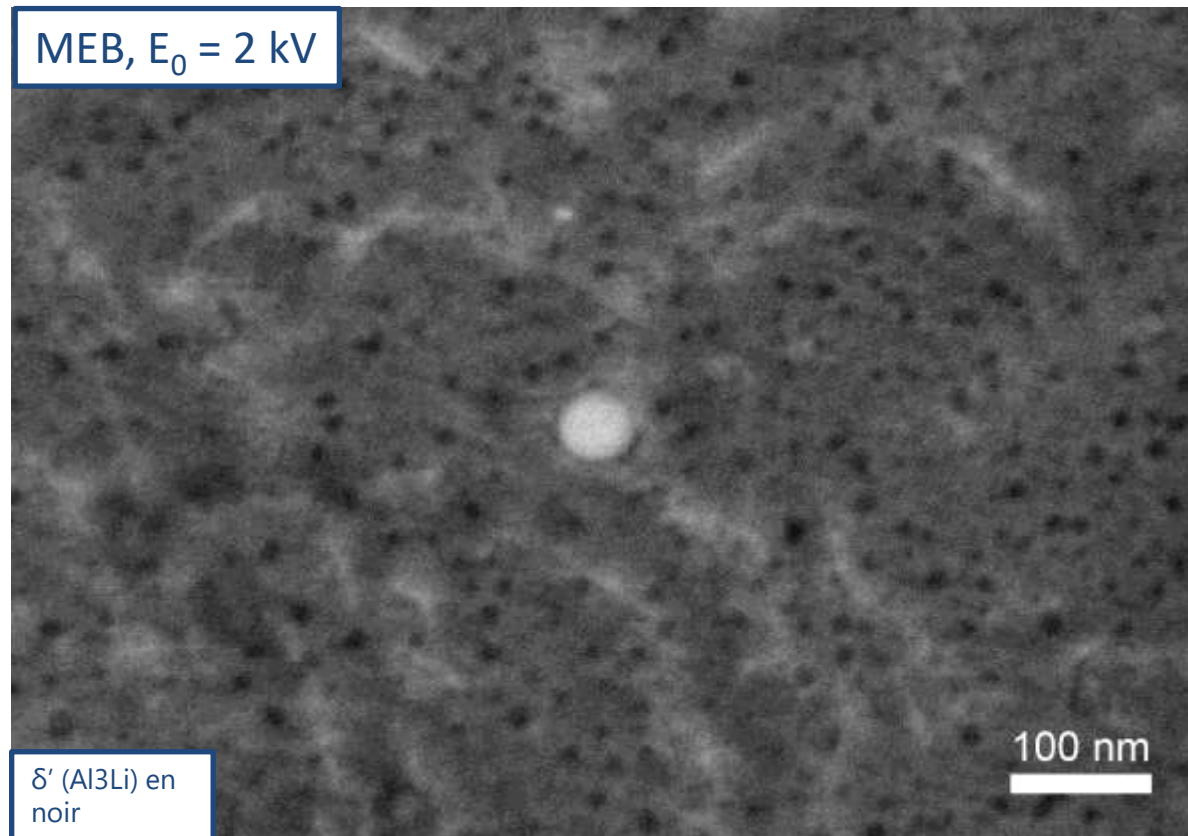
Polissage ionique pour l'imagerie BSE à bas voltage: Alliage Al-Li-Cu AA2099-T83

- Simulations Monte Carlo = Contraste entre δ' (Al_3Li) et matrice Al
- Pas observé avec polissage mécanique et ionique conventionnels
- Polissage ionique bas voltage nécessaire ($< 1 \text{ kV}$)

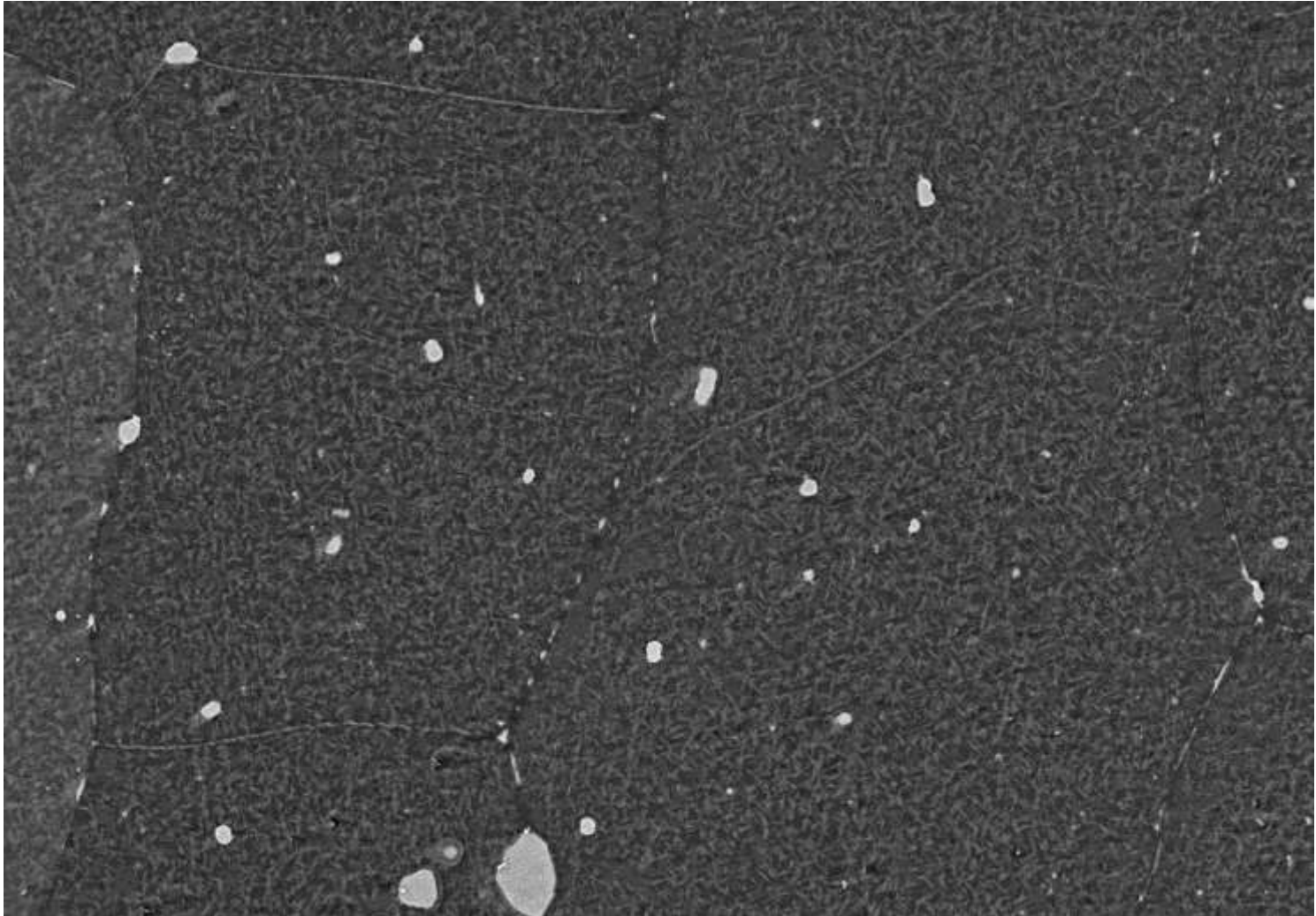


Brodusch, N., Voisard, F., & Gauvin, R. (2017), *J Microsc*, 268(2), 107-118.

Polissage ionique pour l'imagerie BSE à bas voltage: Alliage Al-Li-Cu AA2099-T83

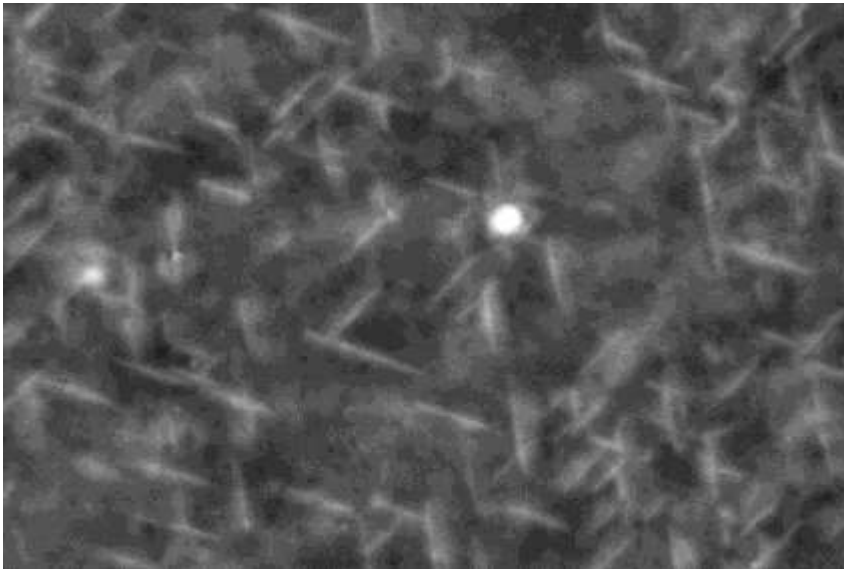


Al – Li alloy, 2099



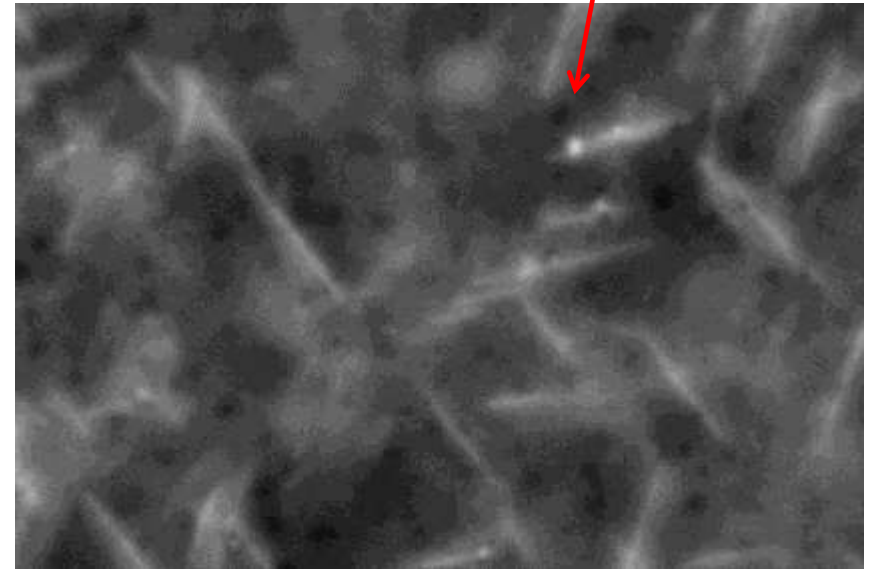
Al - Li Alloy 2099

LA100(U)



FOV 1270 nm
100 kX
SNR: 1.5
Resolution: 10.0 nm

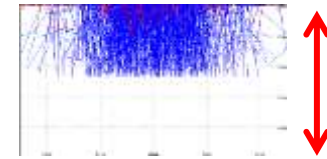
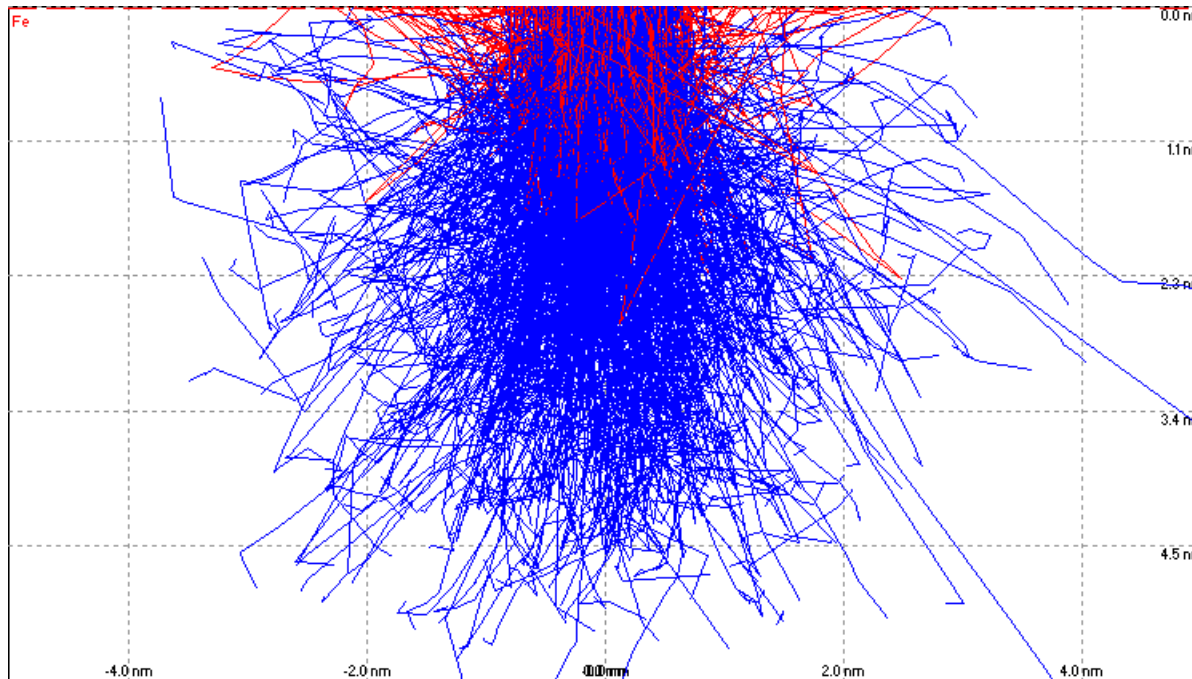
LA100(U)



δ' (Al_3Li)

FOV: 635 nm
200 kX
SNR: 1.6
Resolution: 6.6 nm

MEB Très basse Énergie



0.1 kV

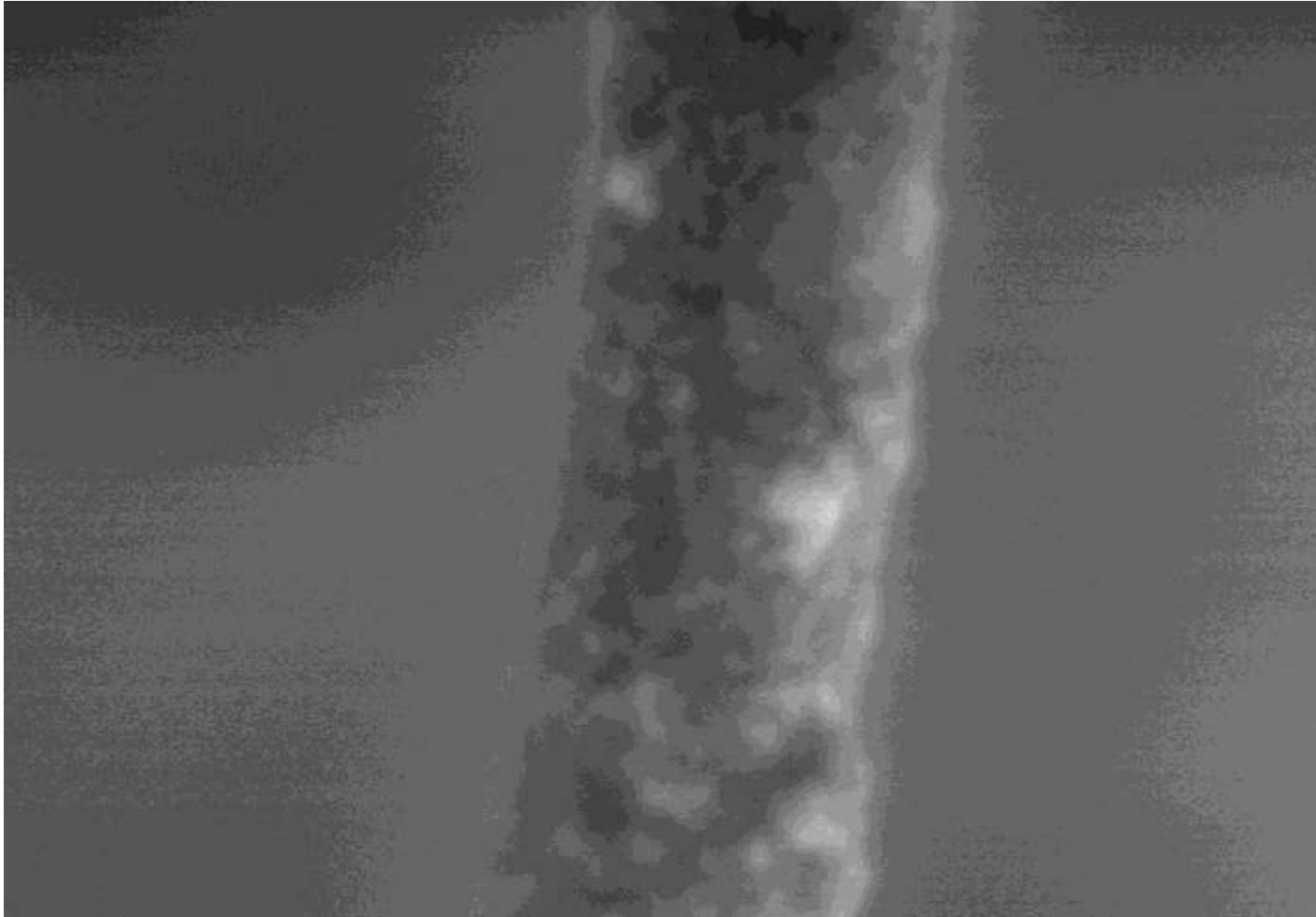
0.5 kV

10 nm

Ultra low voltage imaging:

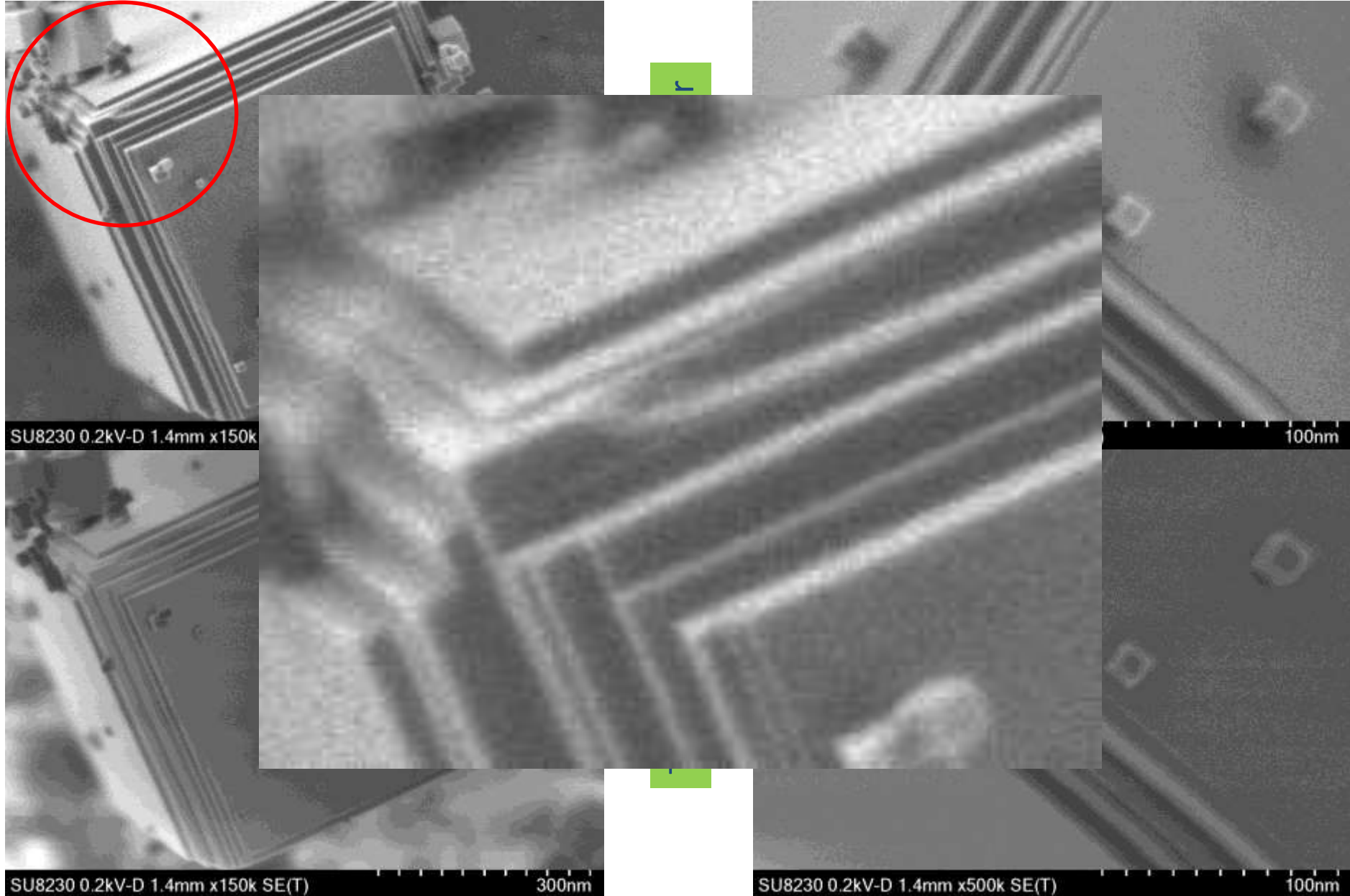
- Close to atomic scale interaction volume
- SE/BSE signals from the same interaction volume

Top Detector, 50 V



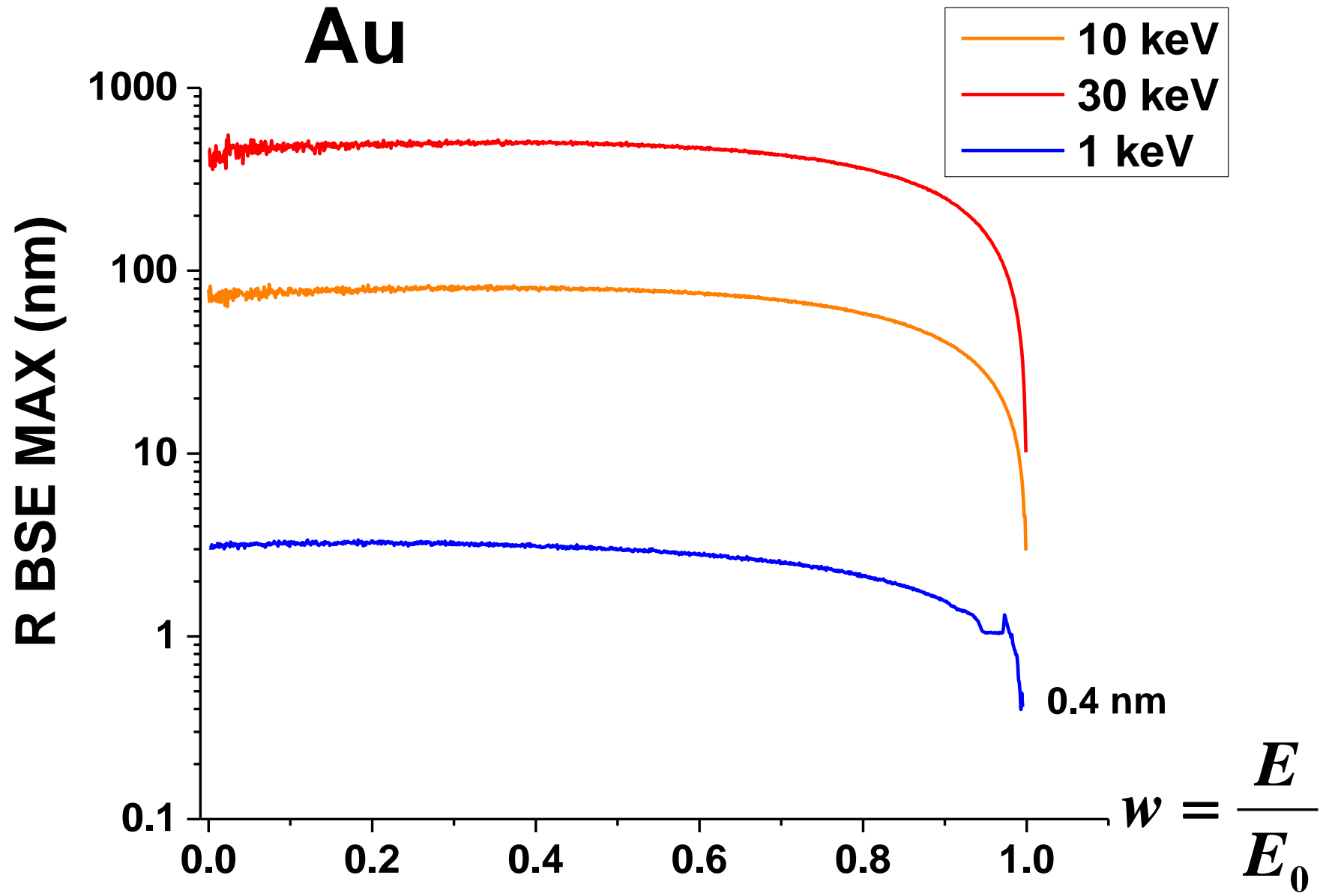
500 kX
SE(T)
FOV: 254 nm
SNR: 1.0
Resolution: 2.8 nm

MgO ash on Al substrate

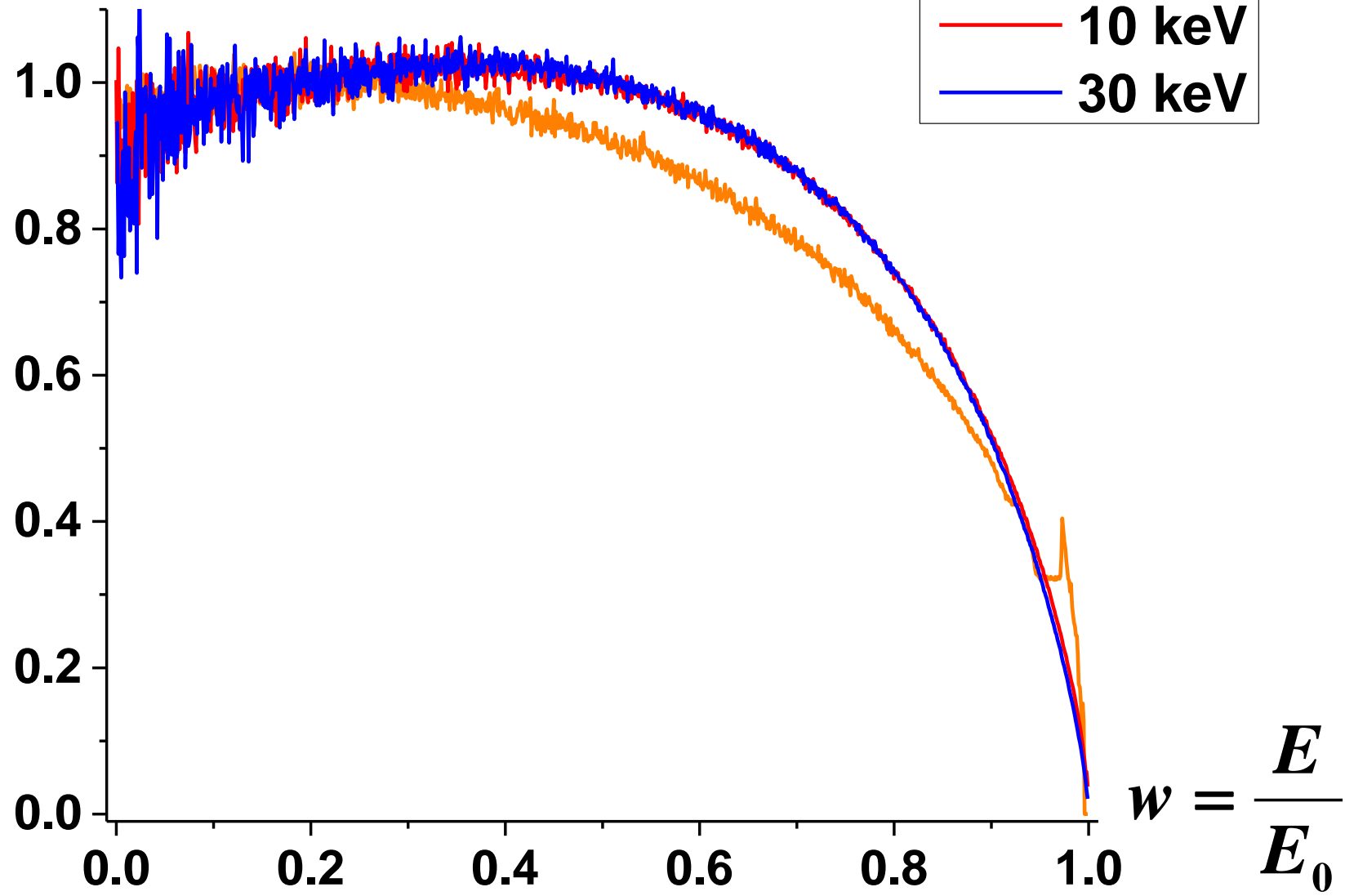


Filtration en Énergie des Électrons Rétrodiffusés

Au



Au

$$\frac{R}{R_{Max}}$$


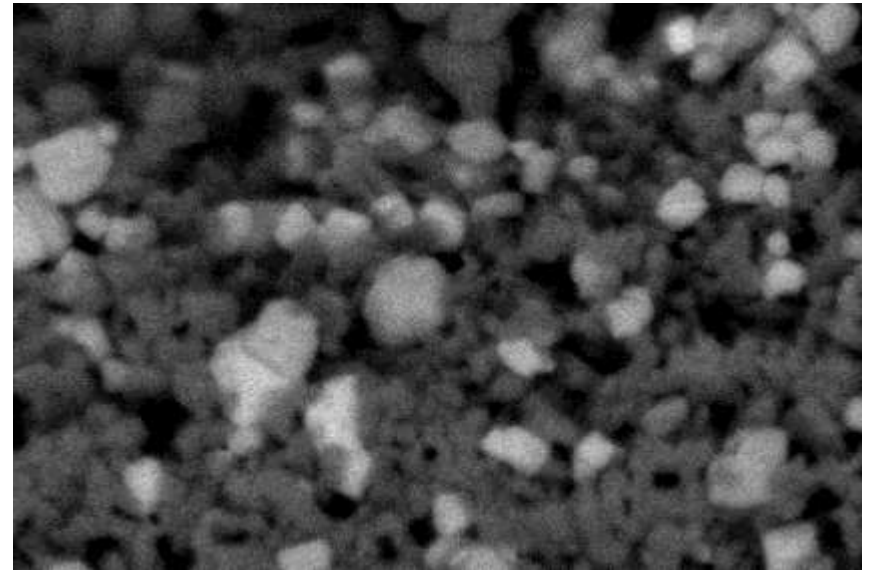
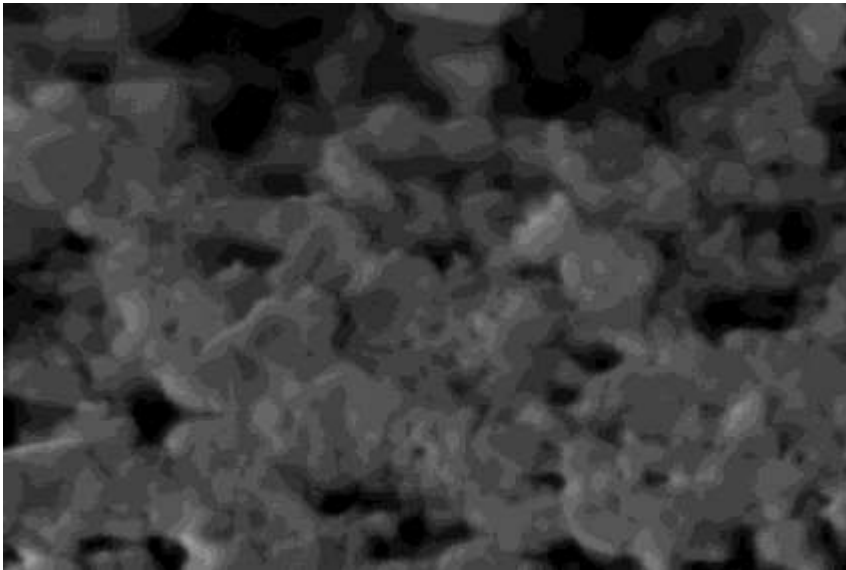
- 1 keV
- 10 keV
- 30 keV

$$w = \frac{E}{E_0}$$

Li powder

SE(U)

HA(T)F95



SNR: 36

Resolution: 3.713 nm

FOV: 706 nm

180 kX

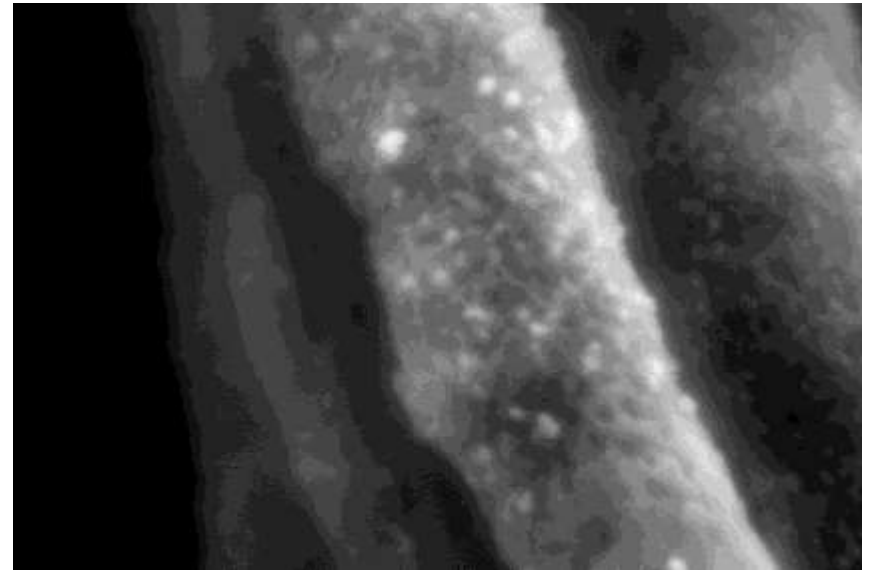
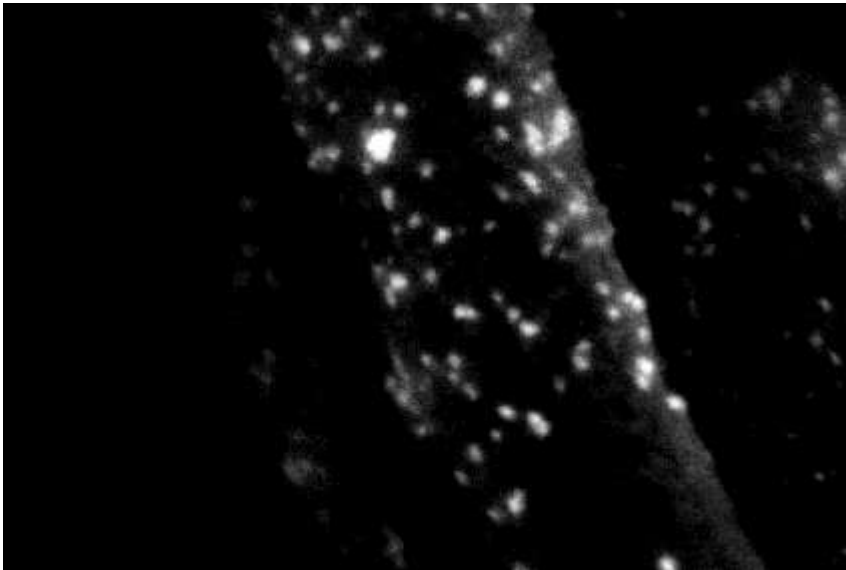
SNR: 1.5

Resolution: 4.388 nm

CNT 1 kV Deceleration

BSE(T)F150

SE+BSE(U)



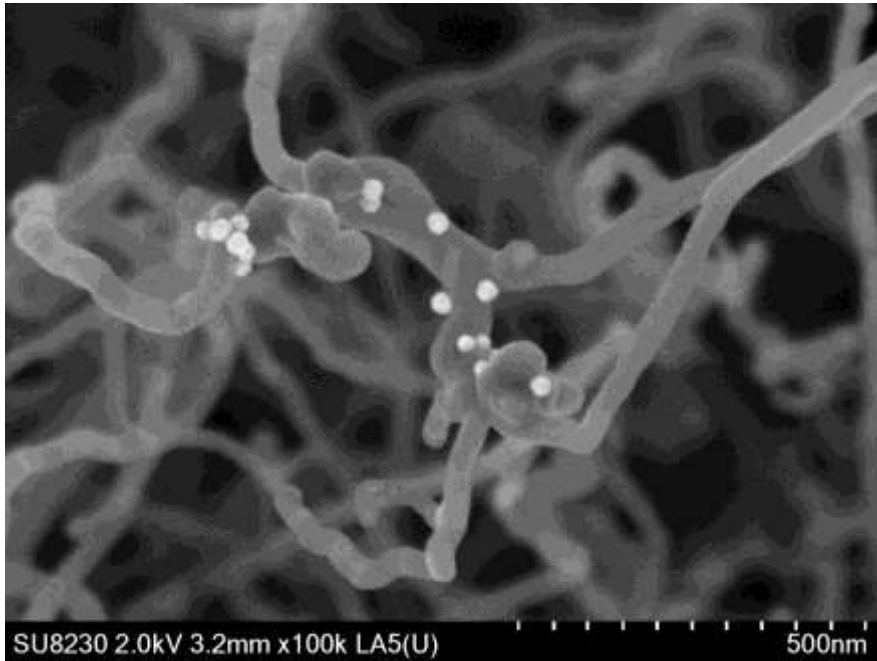
SNR: NaN
Resolution: 1.9 nm

FOV 254 nm
500 kX

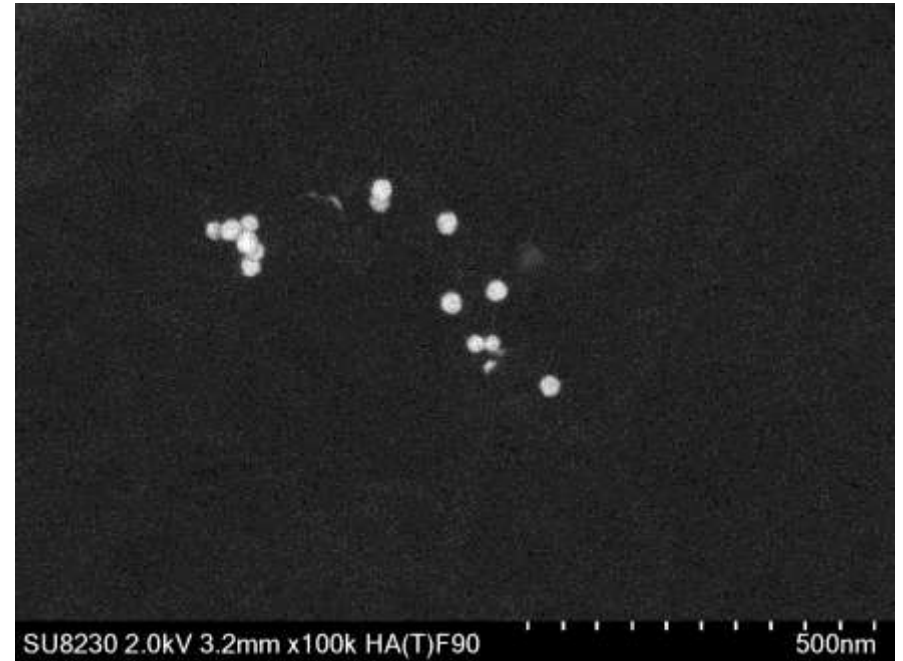
SNR: 6.4
Resolution: 2.5 nm

Au on MCNT, 2 keV

LA5(U)

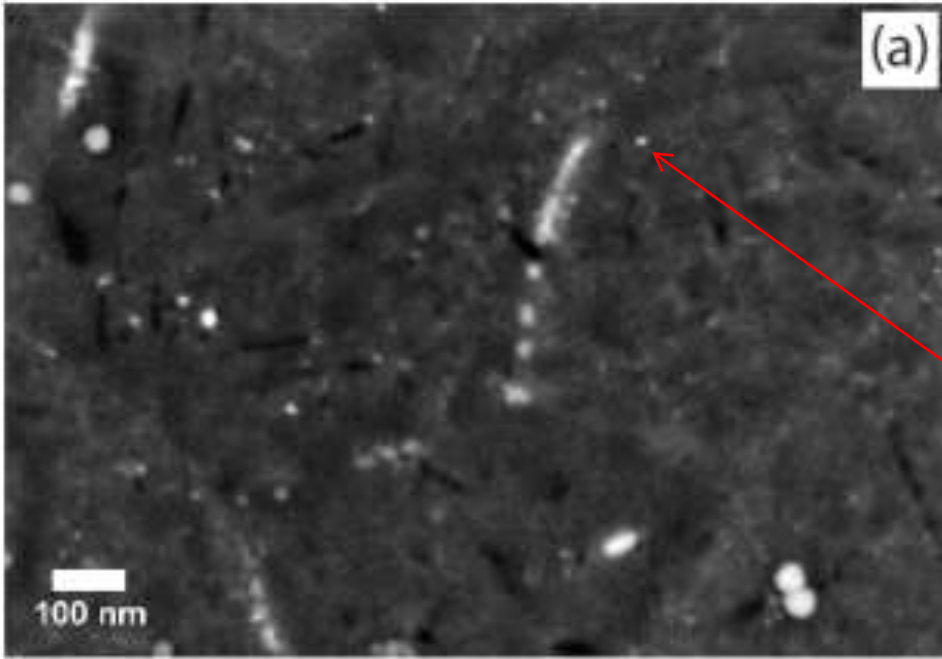


HA(T)F90



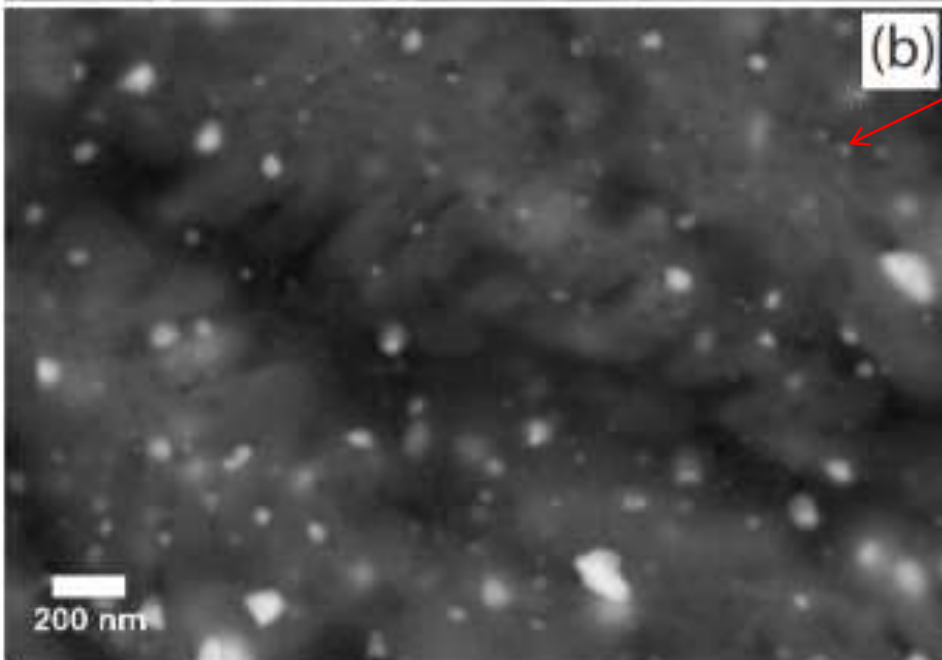
Manfred Von Ardenne





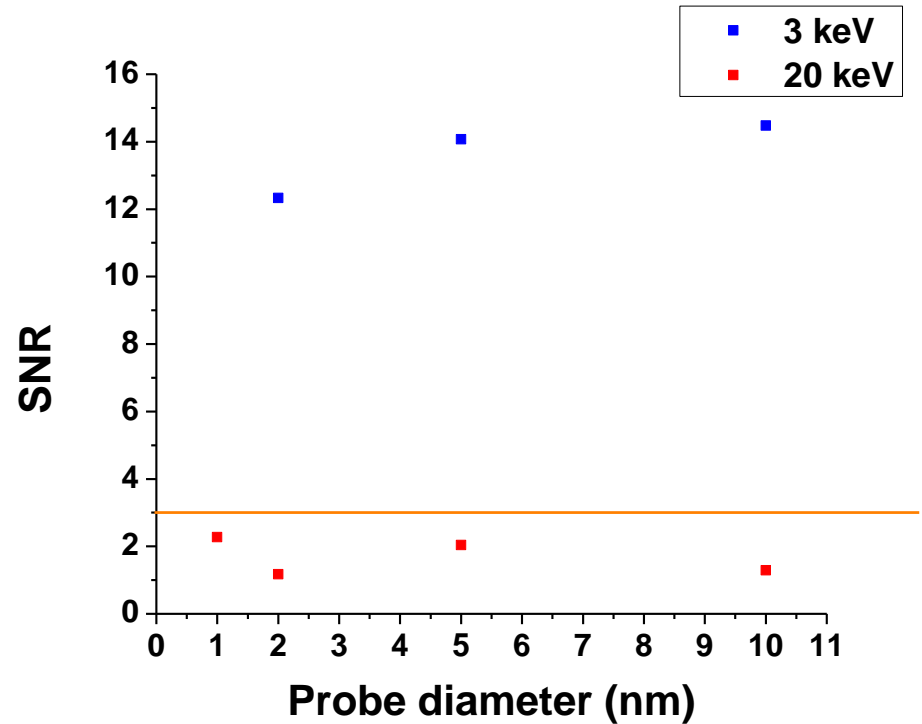
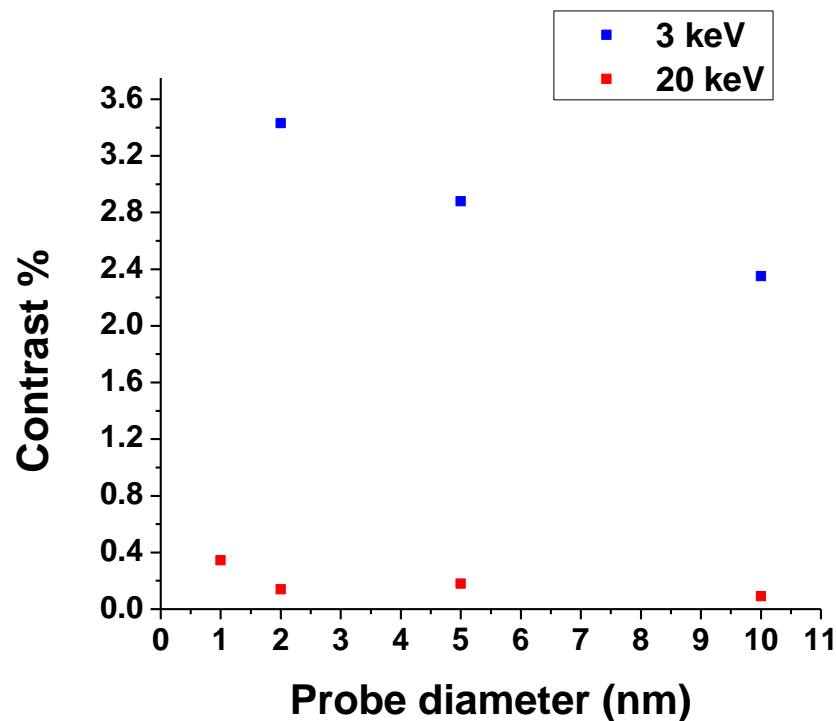
Al – Li alloy
3 keV, SE upper Detector

10 nm Inclusions



PbS inclusion in Slag
20 keV, BSE 5 Quadrant Detector

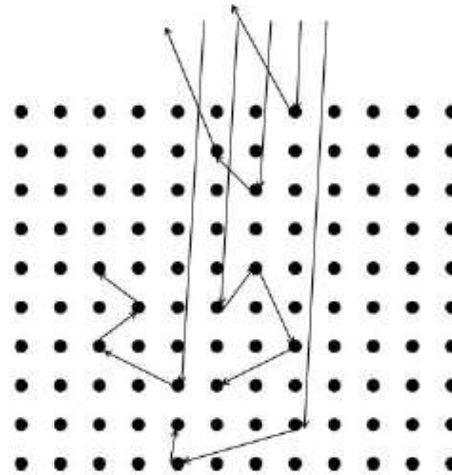
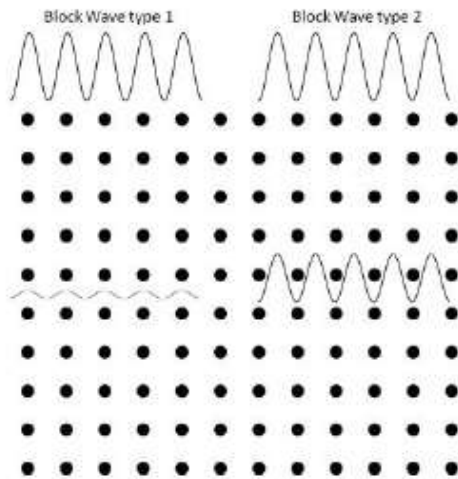
10 nm PbS Inclusion in Si



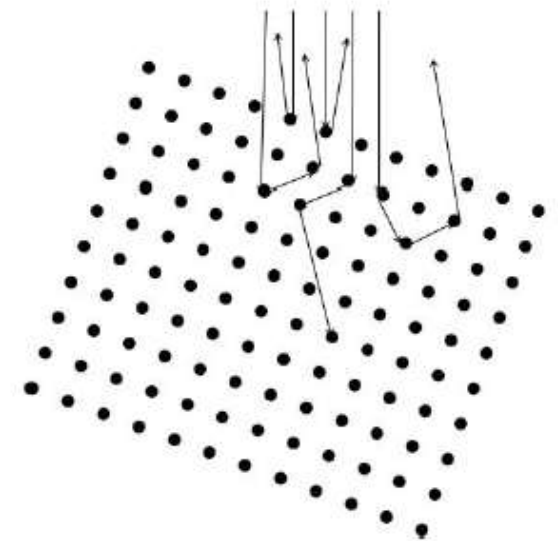
$$i \propto E_0$$
$$i \propto d^{0.67}$$

$i = 5 \text{ nA}$ for $d = 1 \text{ nm}$ at 20 keV

Electron Channelling Ion Milling

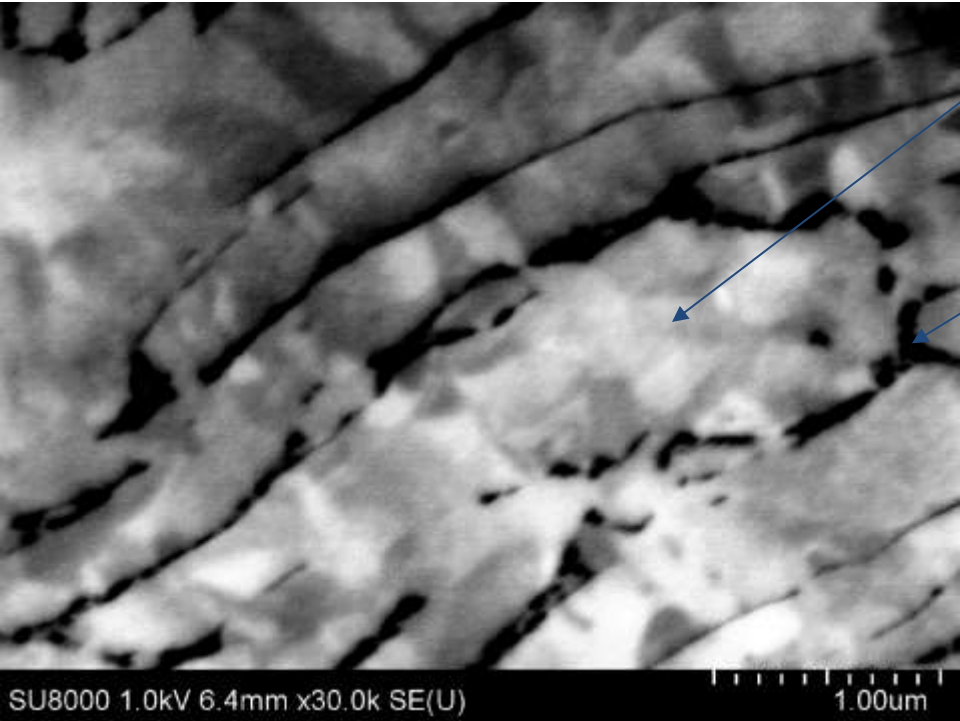


Low retrodiffusion yield



High retrodiffusion yield

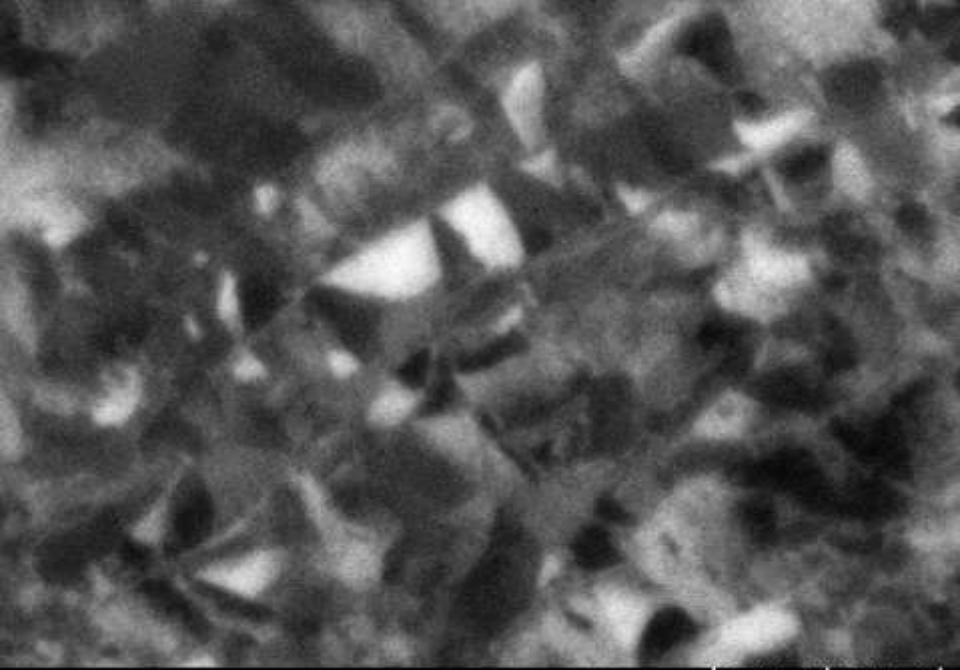
Channeling contrast 1 kV (Zr Alloy)



α phase
 β phase
BackScattered Electrons (PD-BSE detector)

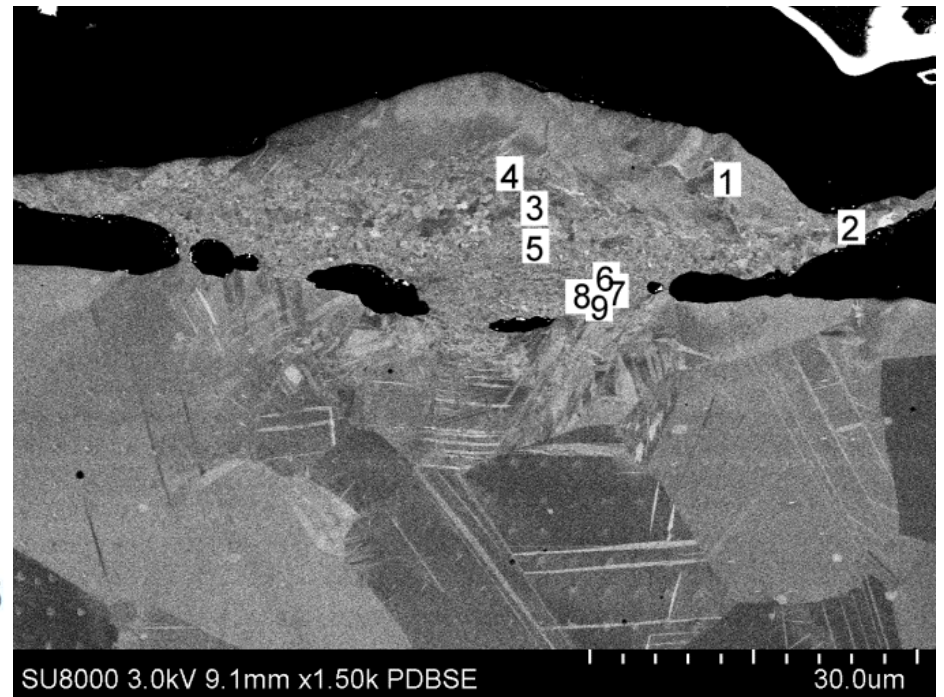
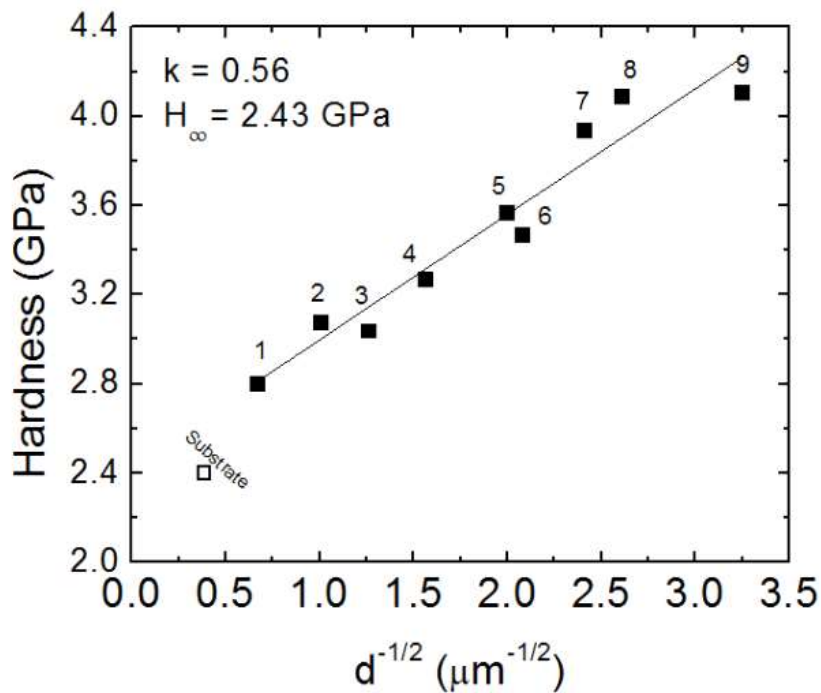


Secondary Electrons (Upper detector)



SU8000 1.0kV 6.4mm x30.0k PDBSE 1.00um

Ex-Situ Nano Indentation

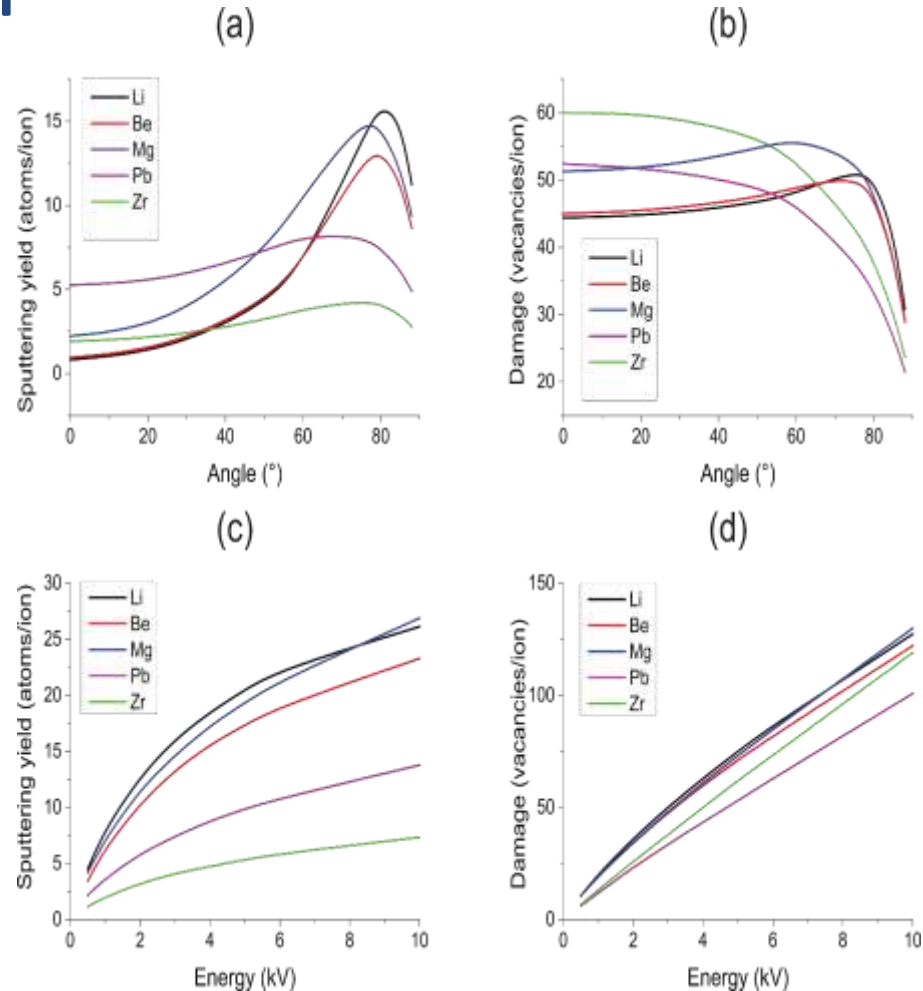


Dina Goldbaum, Richard R. Chromik, Nicolas Brodusch and Raynald Gauvin (2015),
Microscopy and Microanalysis, 32, 6, pp. 1 – 12.

**LE POLISSAGE IONIQUE
POUR L'IMAGERIE DE
SURFACES DES
MATÉRIAUX MOUS**

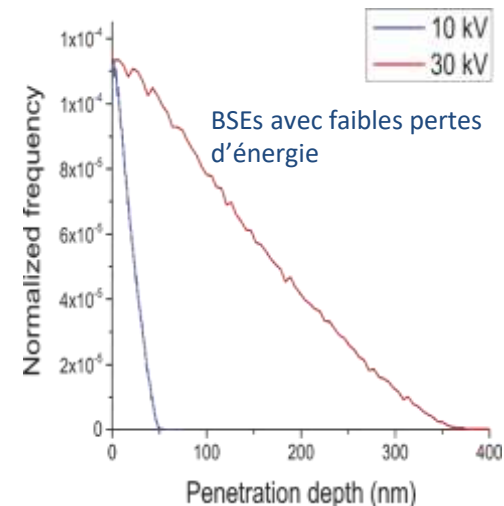
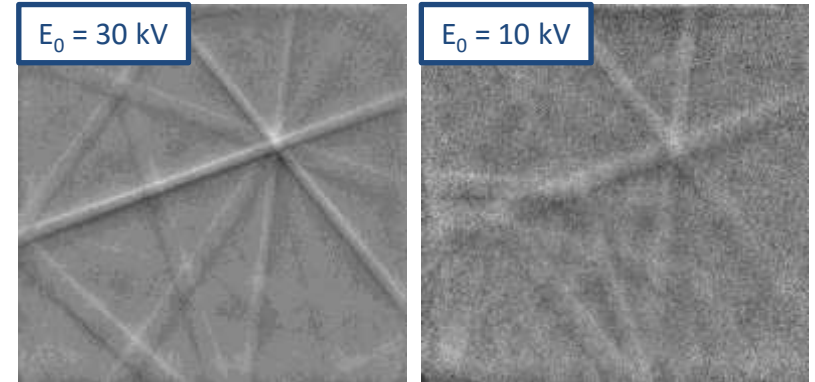
Polissage ionique pour matériaux mous: Exemple du lithium

- Matériaux mous impossibles à polir de façon conventionnelle
- Polissage ionique = Pas d'action mécanique
- Inconvénient = Dommage par irradiation des ions Ar^+
- Taux d'arrachement et dommage = Fonction de l'angle d'incidence et de la tension
- Méthode = angle d'incidence / tension d'accélération variée pour limiter le dommage



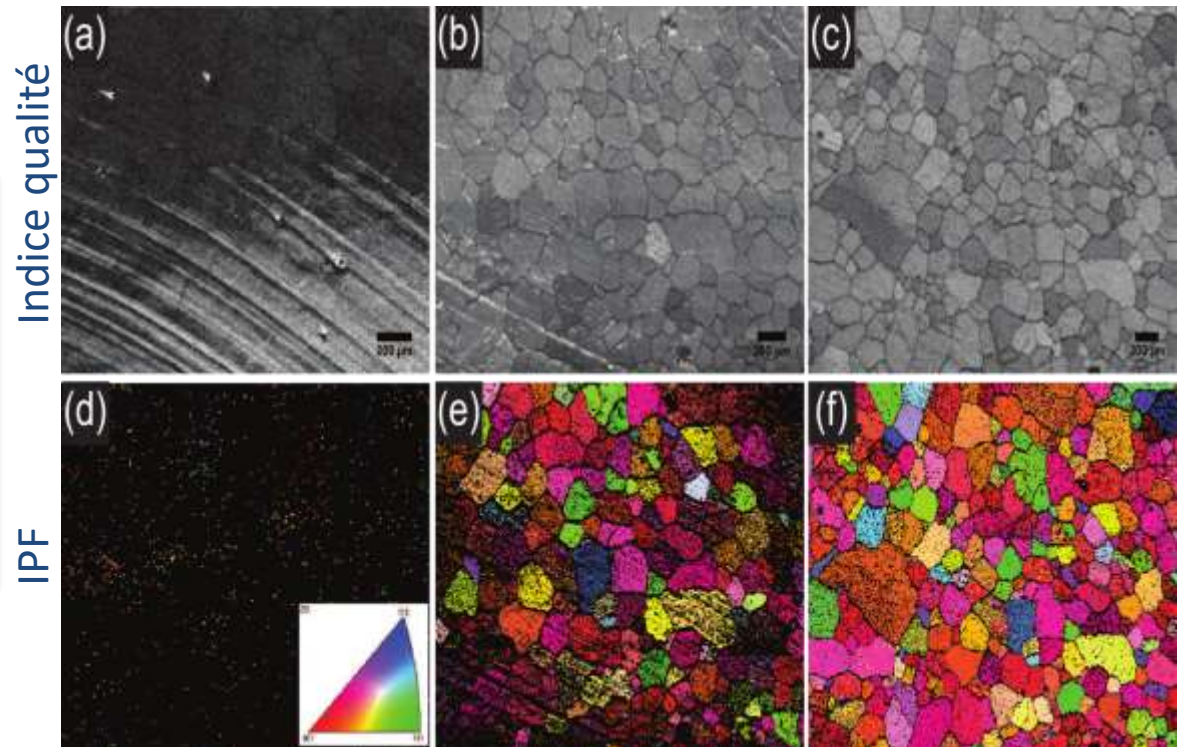
Polissage ionique pour matériaux mous: Exemple du lithium pur extrudé

- Surface marquée mais de bonne qualité
- Clichés EBSD peu intenses mais suffisant pour indexage
- Clichés à $E_0 = 10$ kV = Peu de dommage de surface
- Monte Carlo = Estimation pénétration en profondeur
 - 50 nm pour $E_0 = 10$ kV
 - 350 nm pour $E_0 = 30$ kV

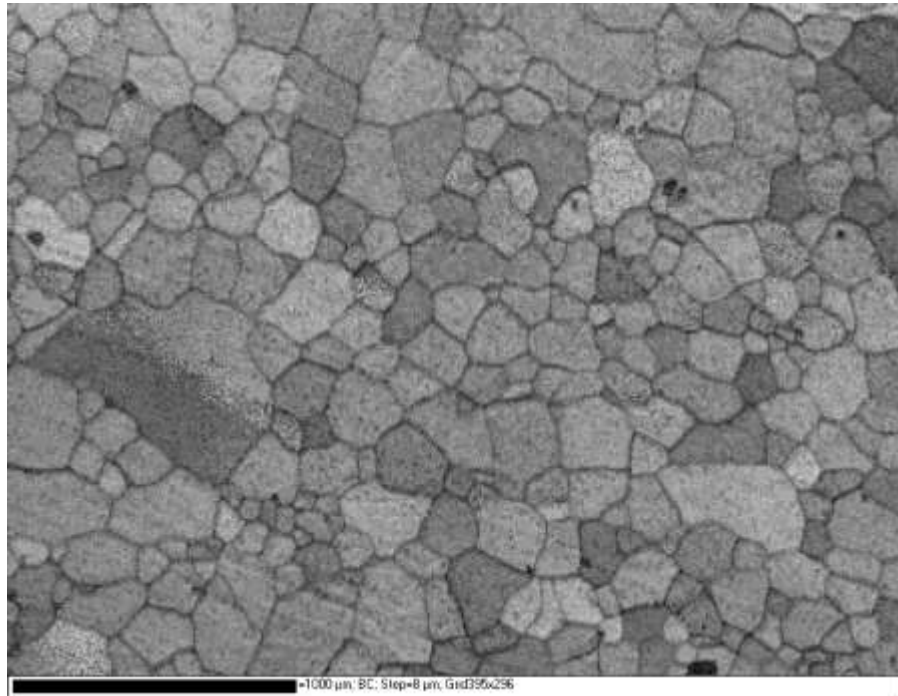


Polissage ionique pour matériaux mous: Exemple du lithium pur extrudé

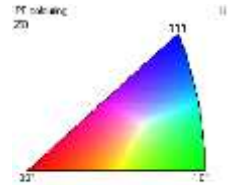
- Nette amélioration de la qualité de surface (indice de qualité) avec le temps de polissage
- Meilleur taux d'indexage (IPF) avec le temps
- Grains facilement identifiables et quantifiables (orientation, taille)



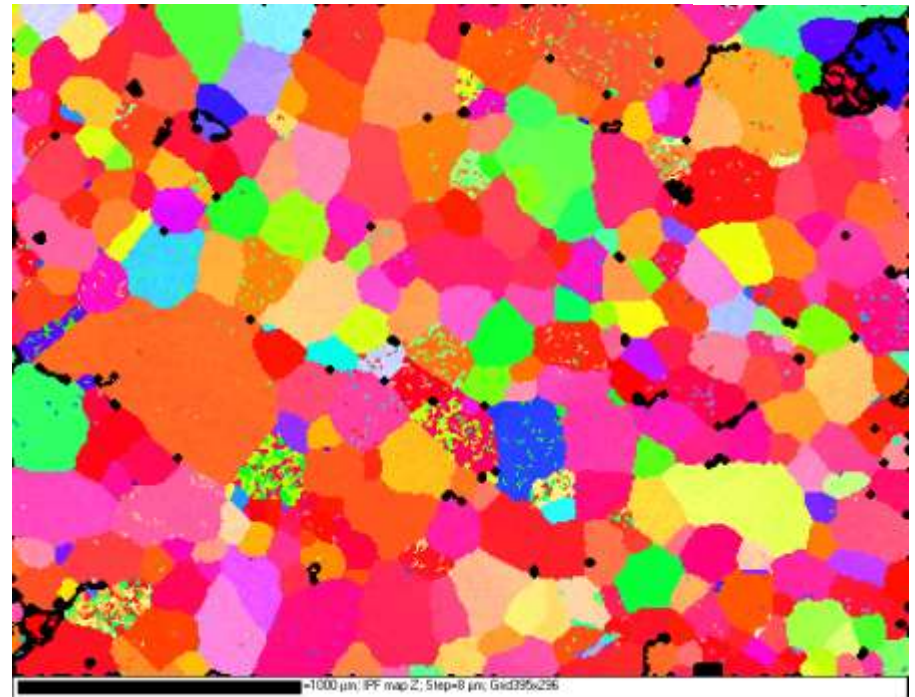
EBSD Feuilles de Li



Band contrast map



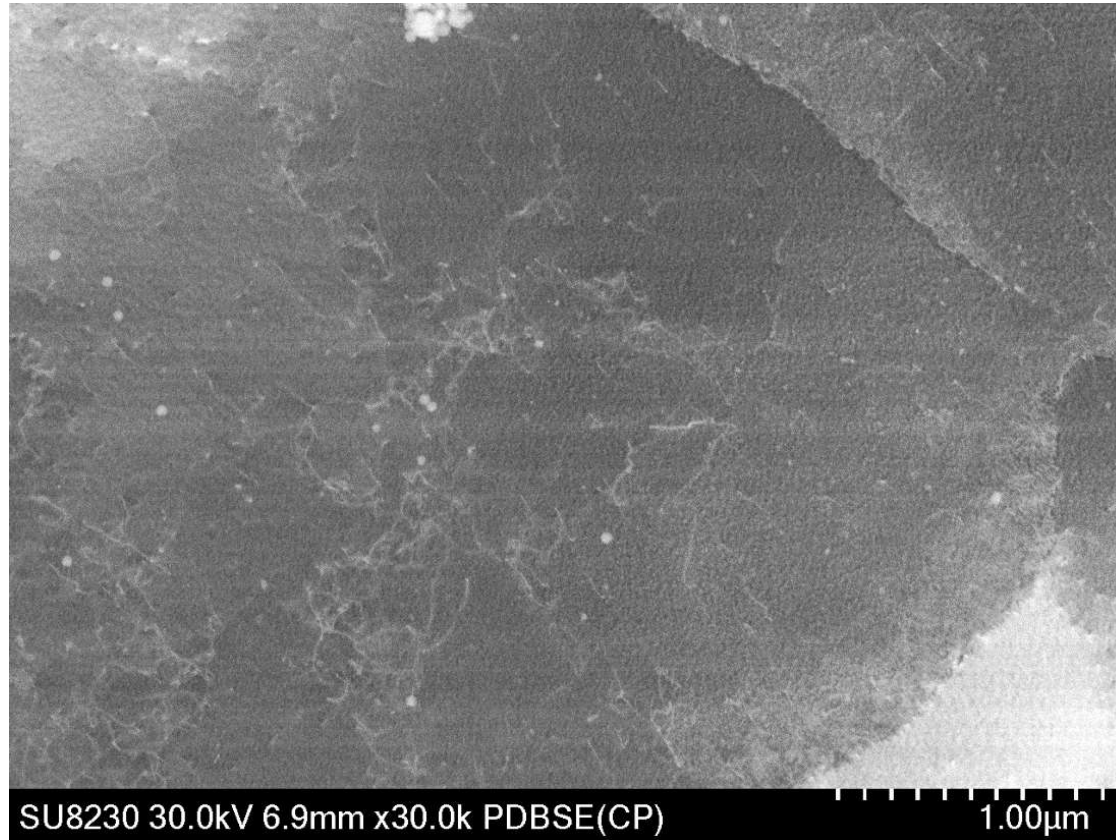
Inverse pole figure map



PRÉPARATION DE SURFACES POUR L'IMAGERIE DES DISLOCATIONS

Préparation d'échantillons: Un pré-requis à l'imagerie des dislocations

- Polissage conventionnel + Silice colloïdale 50 nm
- Silice colloïdale 20 nm
- Polissage ionique:
 - 2 min, 4 kV, 85°
- Attaque chimique NH_3 5% + Polissage finition silice colloïdale 20 nm (2 min)



**Attaque chimique
ou électropolissage
nécessaires**

Quantitative X-Ray Microanalysis

Quantitative X-Ray Microanalysis

$$\frac{C_i}{C_{(i)}} = Z_i A_i F_i \frac{I_i}{I_{(i)}} \frac{(i_p \tau)_{(i)}}{(i_p \tau)_i}$$

$$Z_i = \frac{\int_0^\infty \varphi(\rho z)_i d\rho z}{\int_0^\infty \varphi(\rho z)_{(i)} d\rho z}$$

$$A_i = \frac{f(\chi_{(i)})}{f(\chi_i)}$$

$$f(\chi_i) = \frac{\int_0^\infty \varphi(\rho z)_i e^{-\chi_i \rho z} d\rho z}{\int_0^\infty \varphi(\rho z)_i d\rho z}$$

$$F_i = \frac{(1 + \{ \sum_{j=1}^m f_{c,j} \} + f_{Br})_{(i)}}{(1 + \{ \sum_{j=1}^m f_{c,j} \} + f_{Br})_i}$$

Quantitative X-Ray Microanalysis

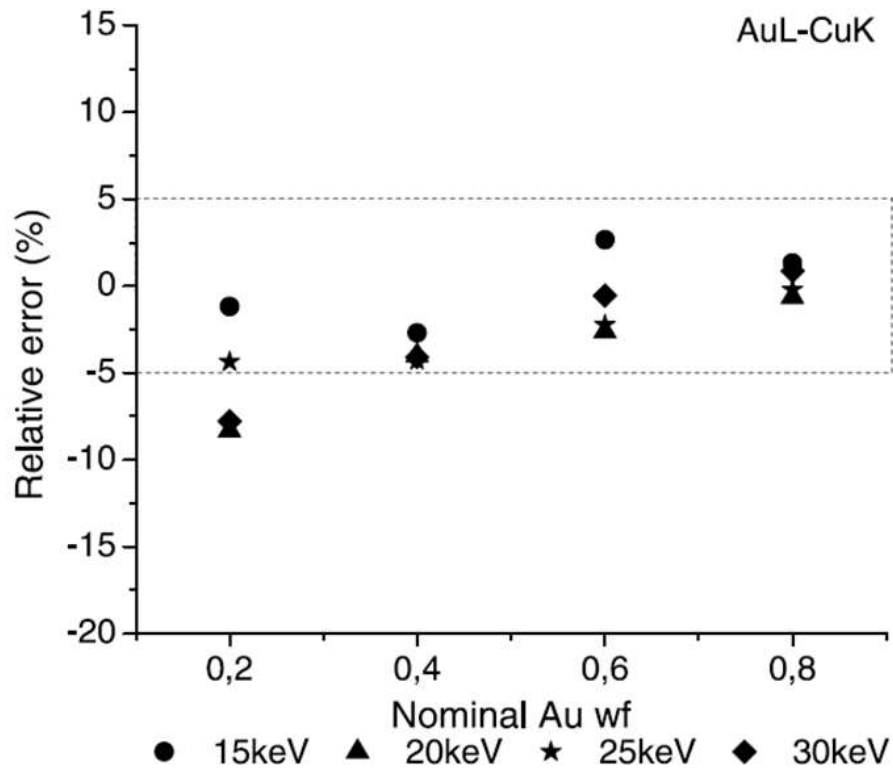
The f Ratio Method, same Spectrum

$$f_A = \frac{I_A}{I_A + I_B}$$

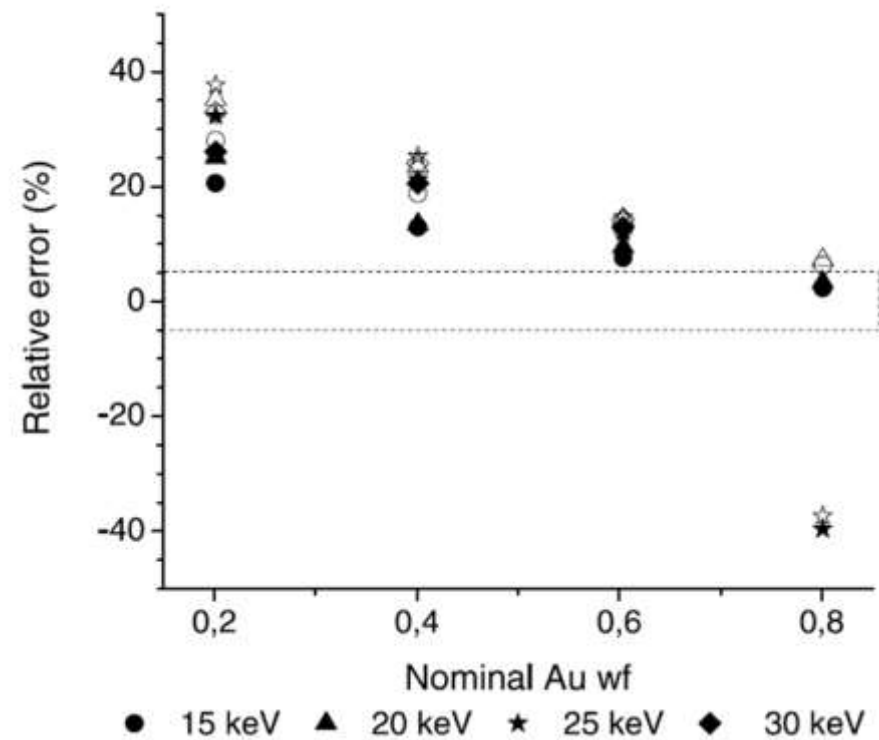
$$f_A = \frac{1}{1 + \Lambda_{A-B} K_{A-B} \frac{\int \varphi_B(\rho z) e^{\chi_B \rho z} d\rho z}{\int \varphi_A(\rho z) e^{\chi_A \rho z} d\rho z} \frac{F_B}{F_A} \frac{c_B}{c_A}}$$

P. Horny, E. Lifshin, H. Campbell and R. Gauvin (2010), "Development of a New Quantitative X-Ray Microanalysis Method for Electron Microscopy", *Microscopy & Microanalysis*, Vol. 16, No. 6, pp. 821-830.

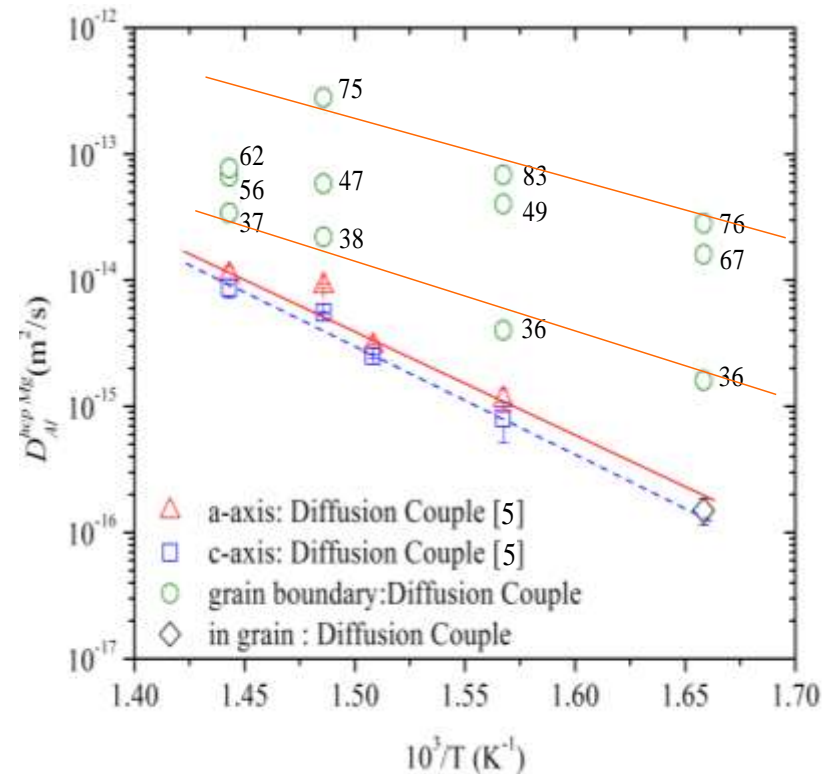
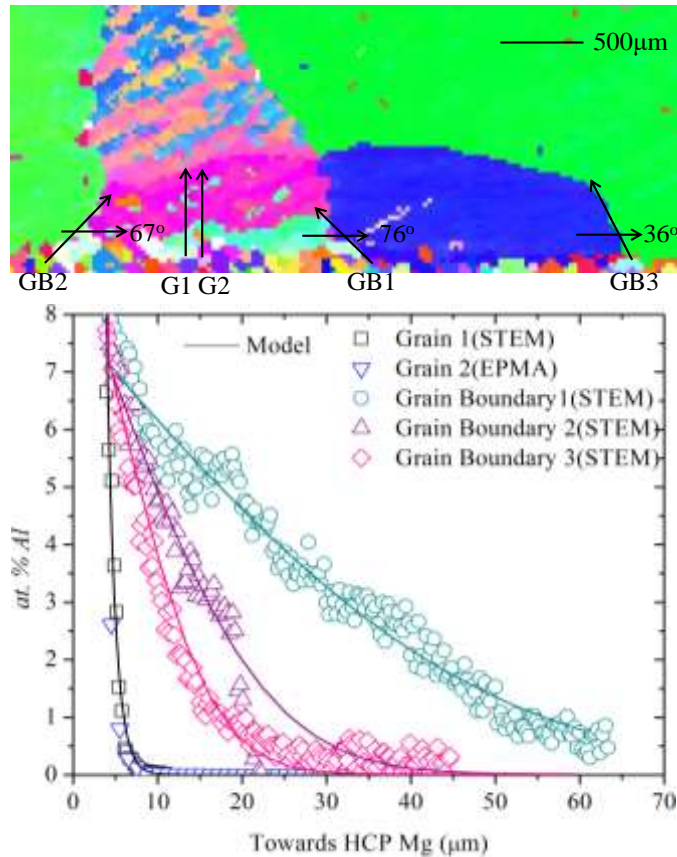
f Ratio Method



Standardless

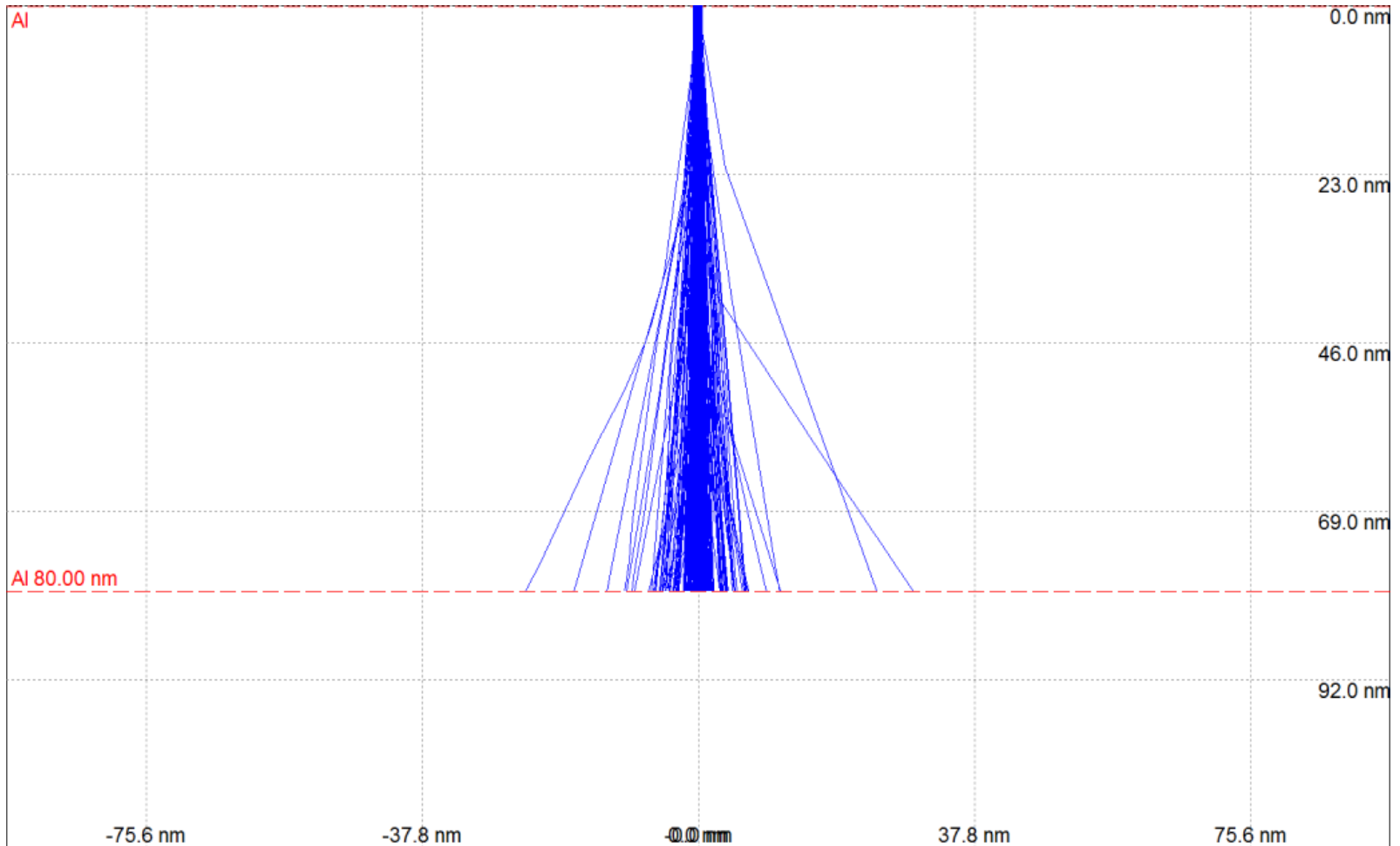


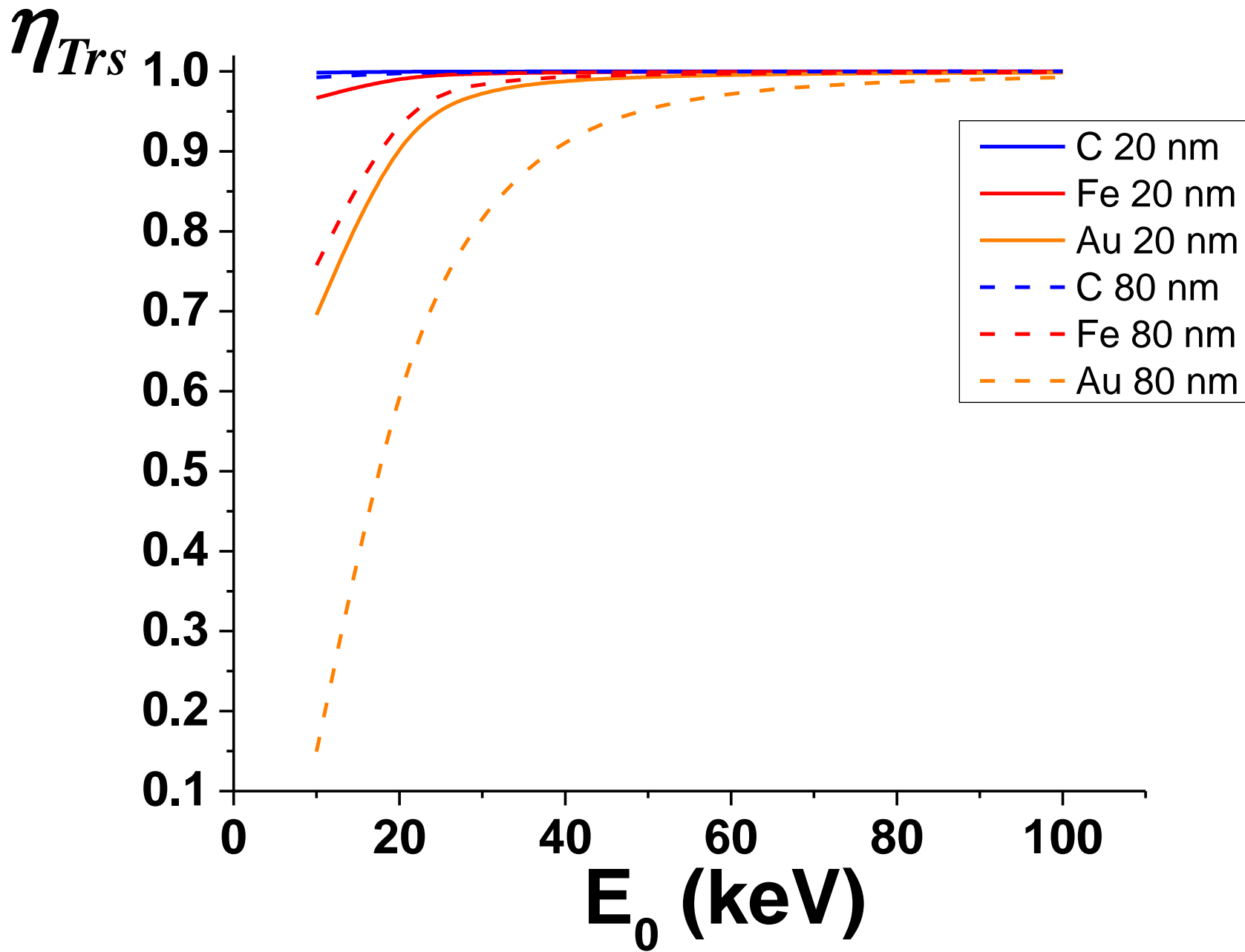
Grain Boundary Diffusion

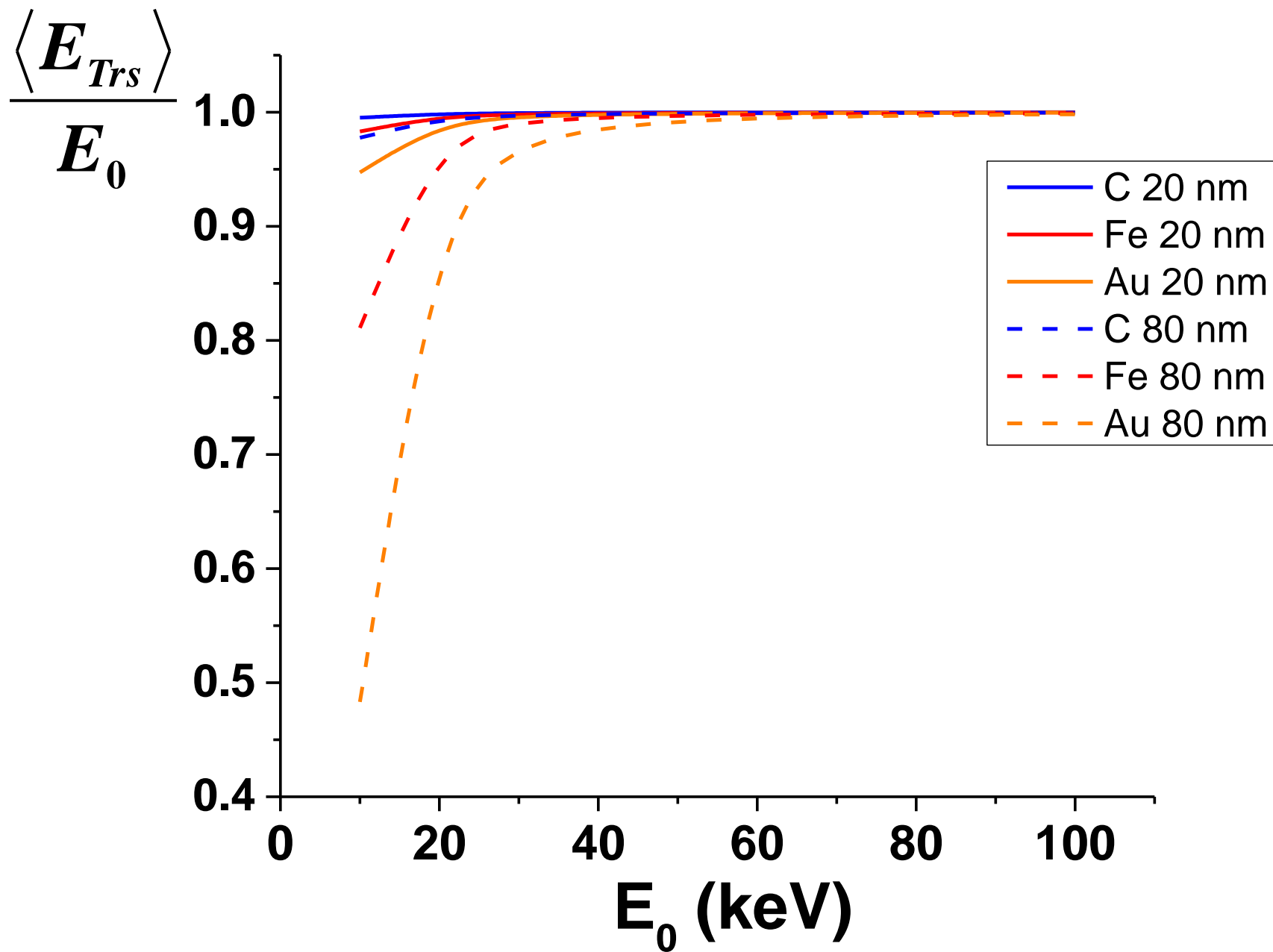


[S. K. Das, N. Brodusch, R. Gauvin and I.-H. Jung \(2014\), "Grain boundary diffusion of Al in Mg", Scripta Materialia, 80, pp. 41-44.](#)

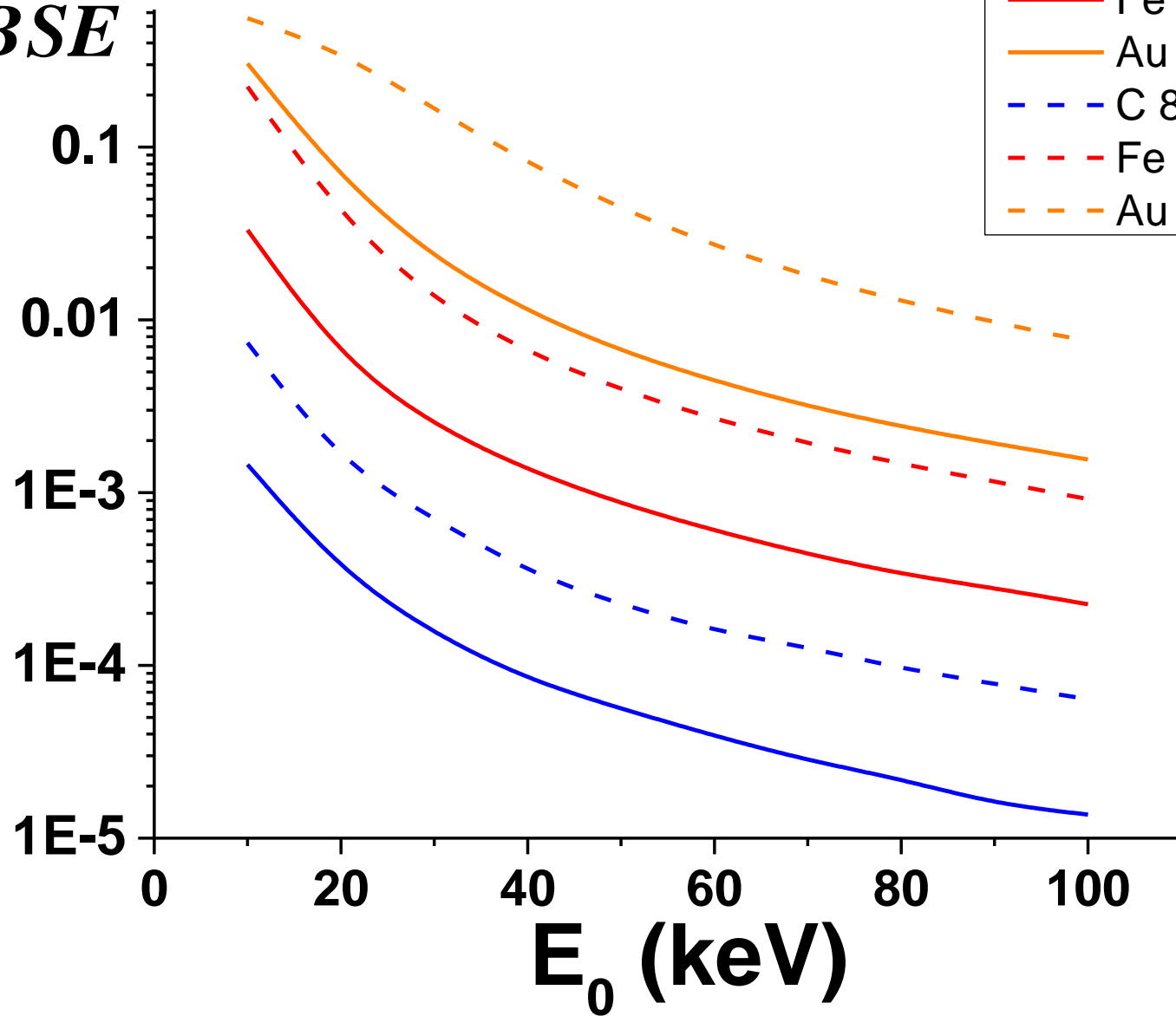
STEM, Al, 30 kV, 80 nm







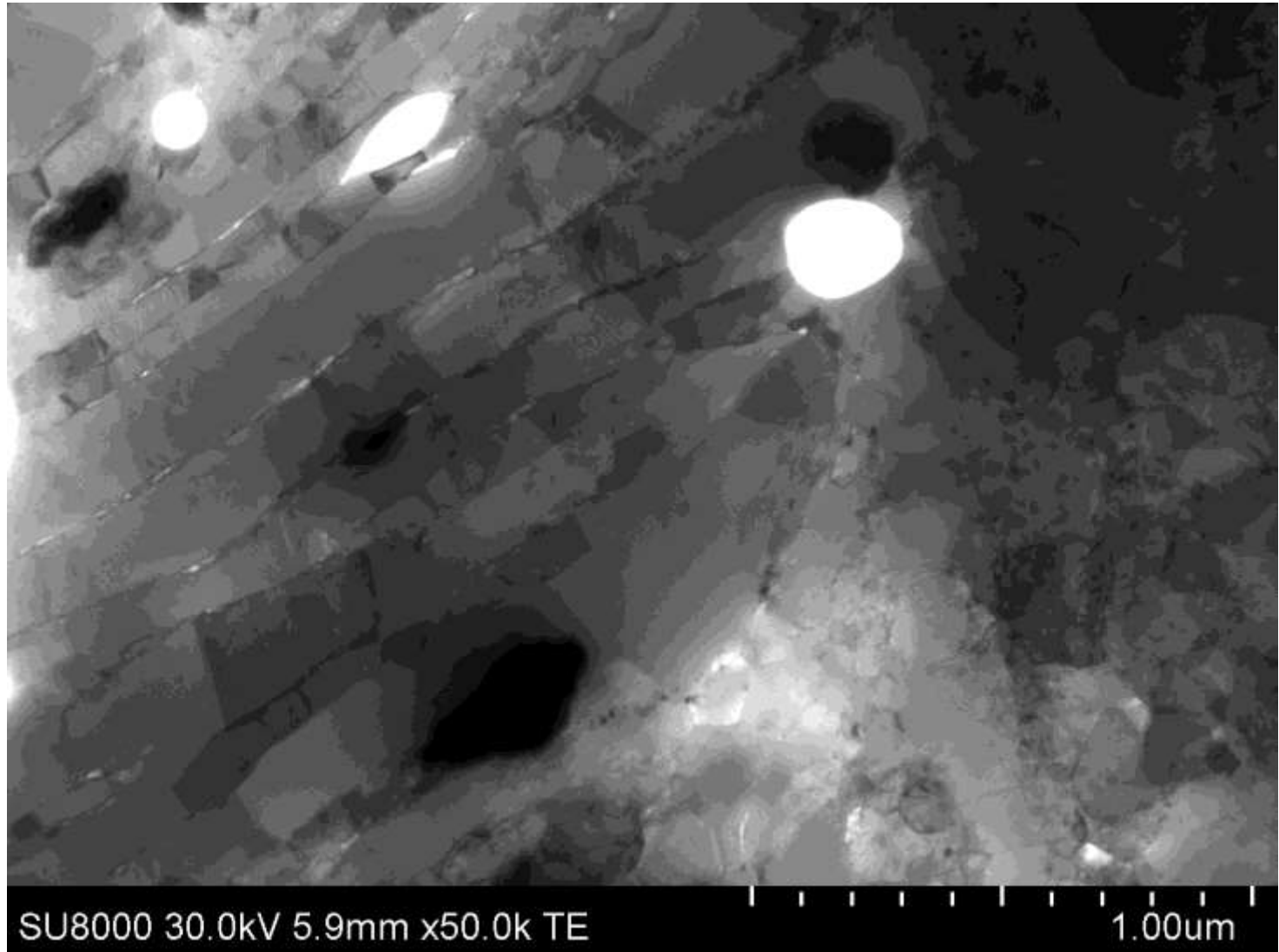
η_{BSE}



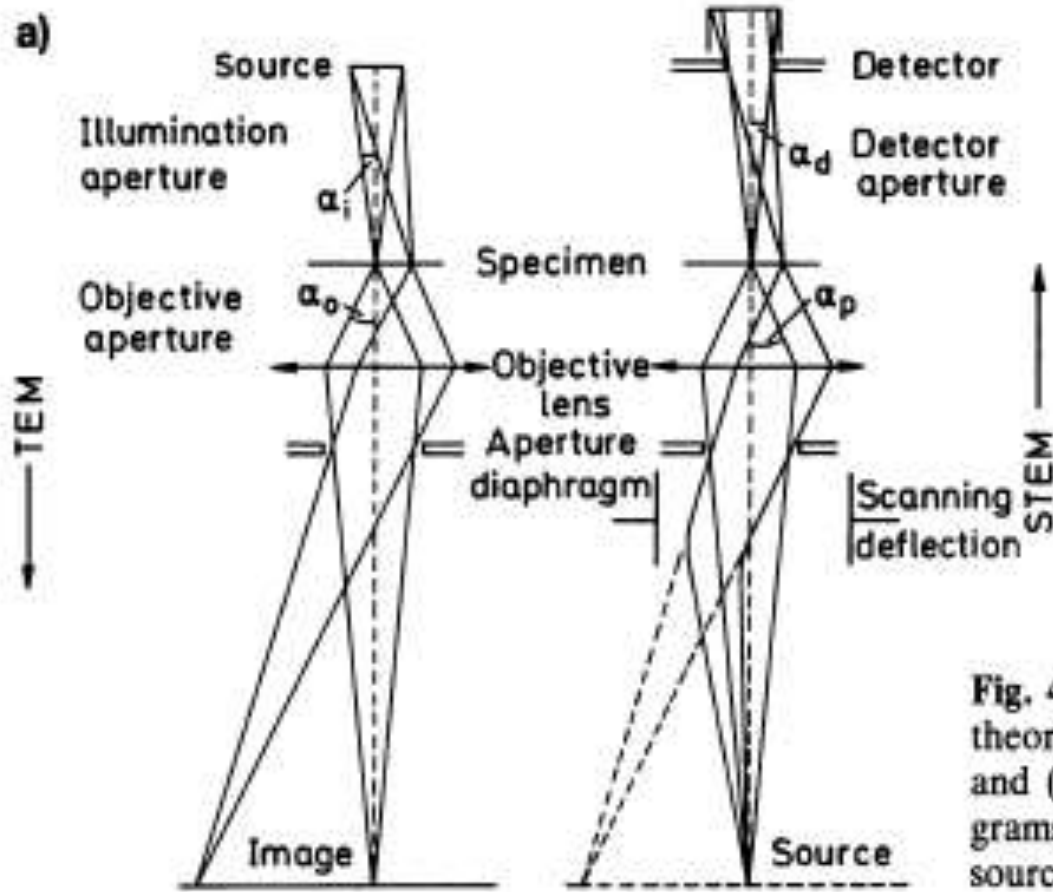
STEM dans le MEB

- Éliminer (Minimiser) la contamination par le Carbone.
- Préparations des lames minces comme au Microscope Électronique en Transmission (MET).
- Focus Ion Beam.
- Jet Electropolishing.
- Ultramicroton.
- Particules/Nanotubes sur grilles Be/Cu recouvertes de Carbone.

Al –Mg Chips



Reciprocity STEM - CTEM



$$\alpha_i = \alpha_d$$

Fig. 4.26 a, b. Demonstration of the theorem of reciprocity for (a) TEM and (b) STEM in terms of ray diagrams connecting the intermediate source and image

From Reimer (1984)

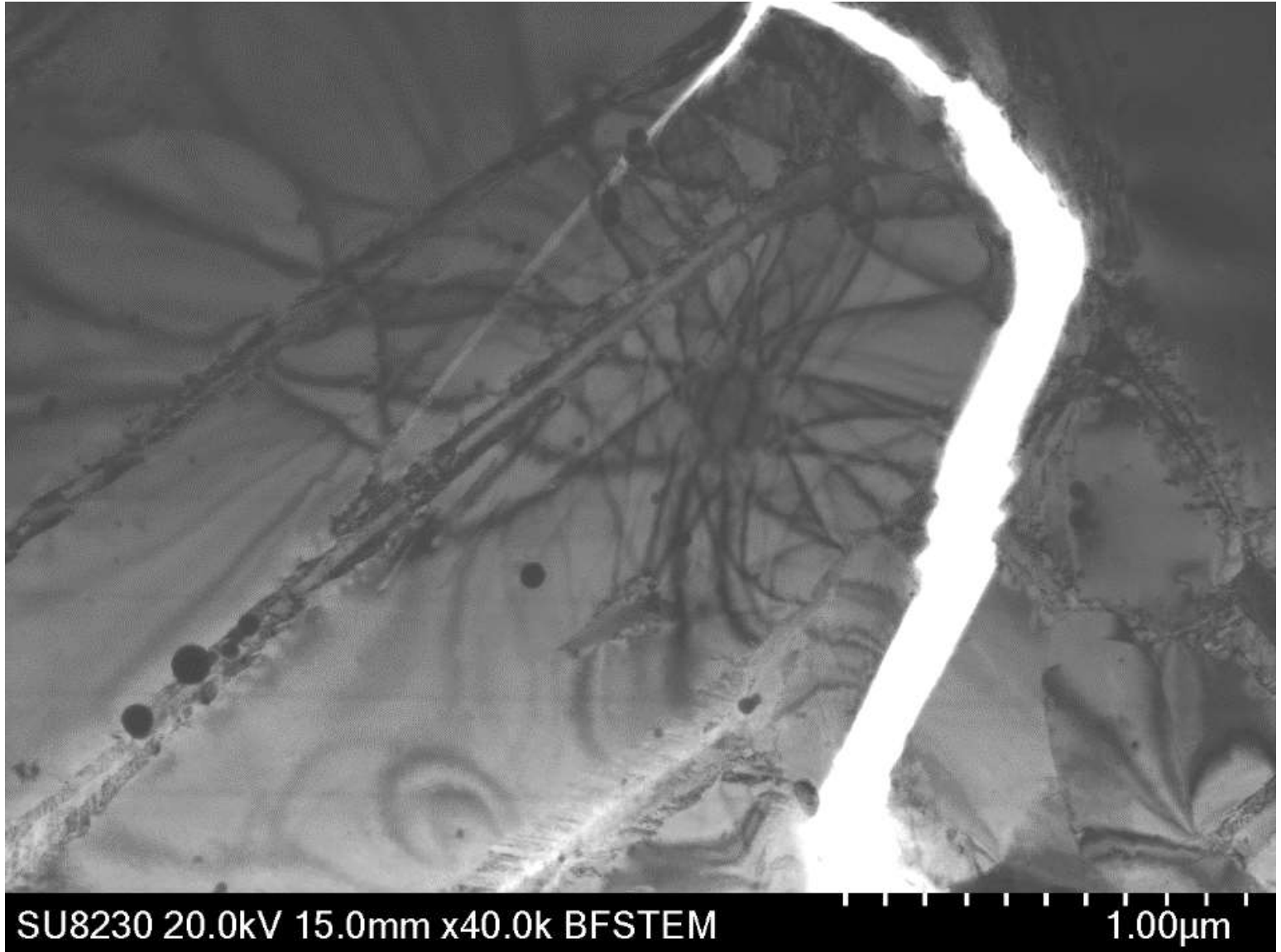
Williams and Carter (2009)

There is an obvious drawback to making α_d small. The signal falling on the STEM detector becomes very small and the STEM image becomes noisy. So STEM diffraction-contrast images become noisier as we attempt to increase the amount of diffraction contrast

Having an FEG helps to offset this increase in noise, but in general, **STEM diffraction-contrast images** (in both BF and DF) **compare so unfavorably with TEM images...**

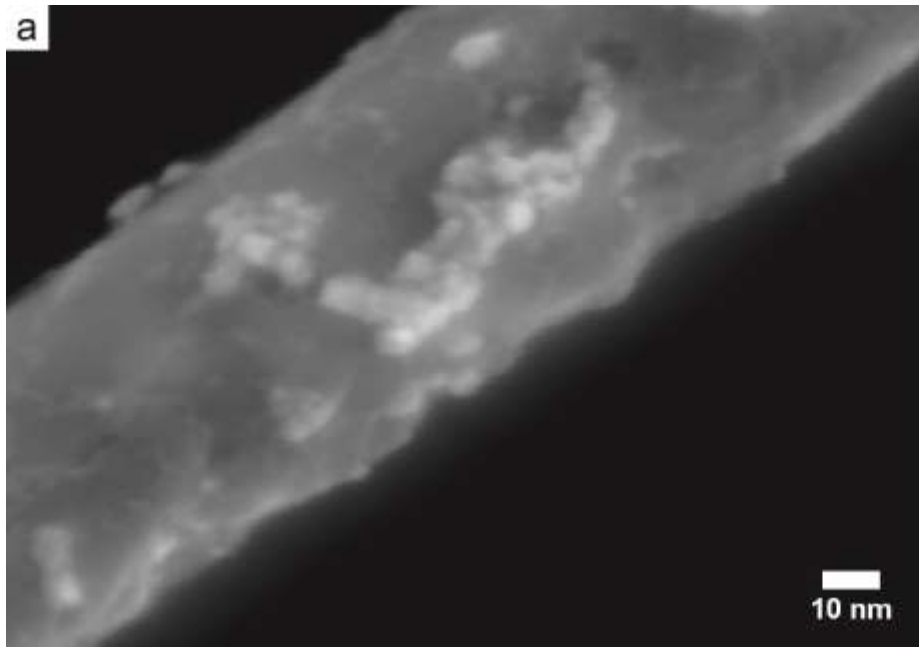
Aperture 3 ($\beta = 3.3$ mrad)

WD = 15 mm

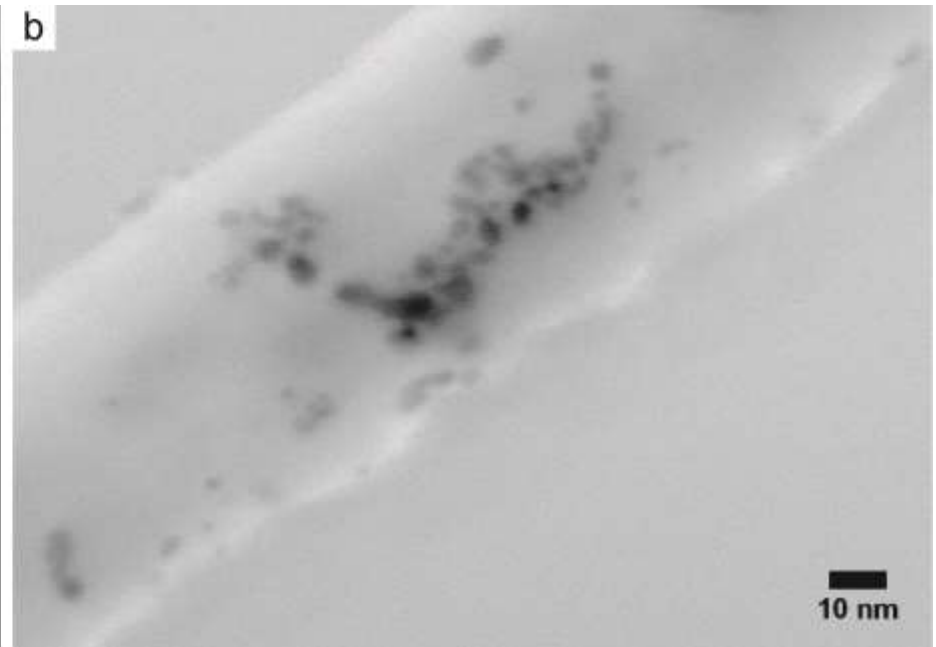


CNT covered with Pt/Ni nanoparticles

$E_0 = 10 \text{ kV}$

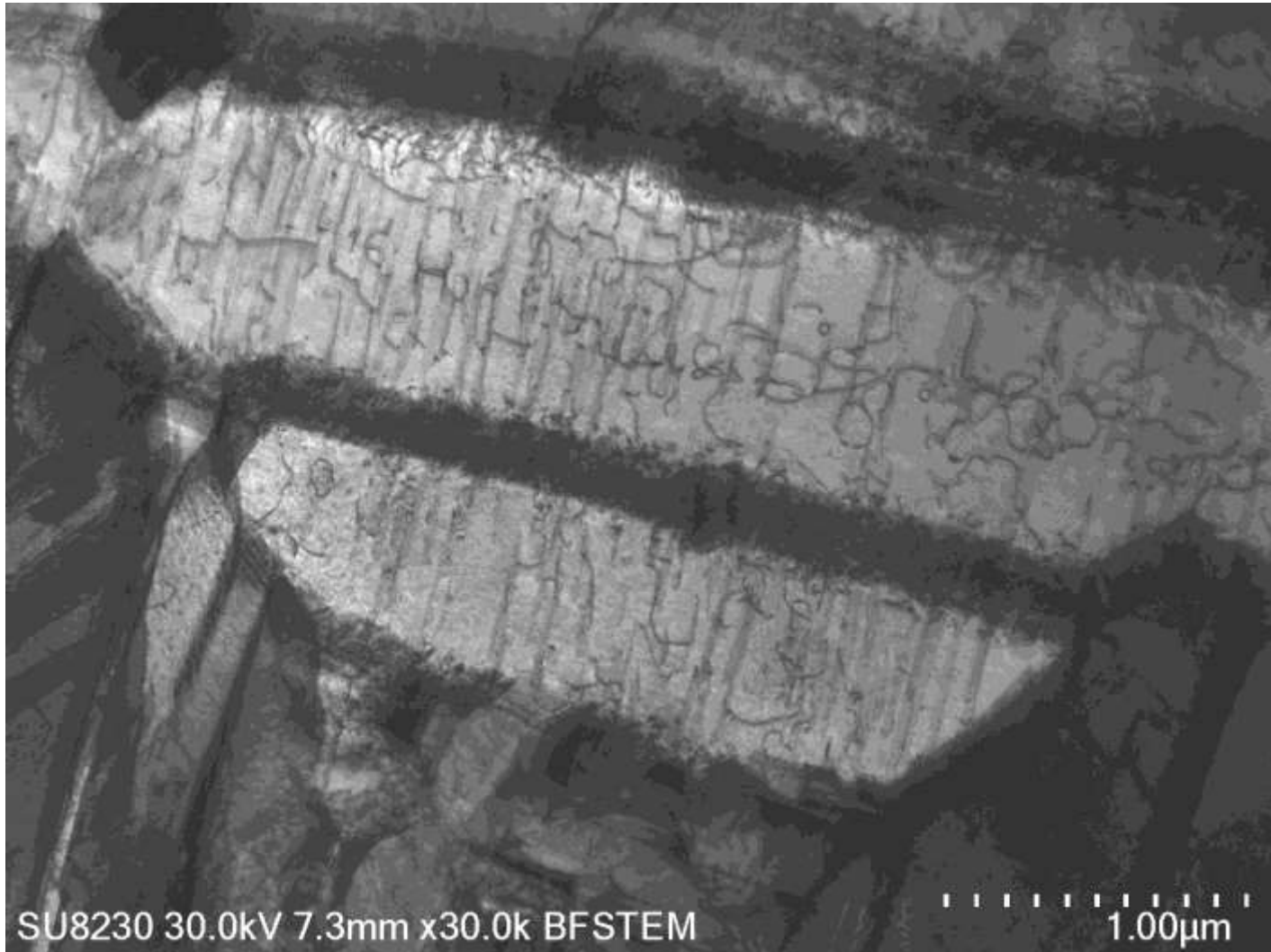


SE



BF

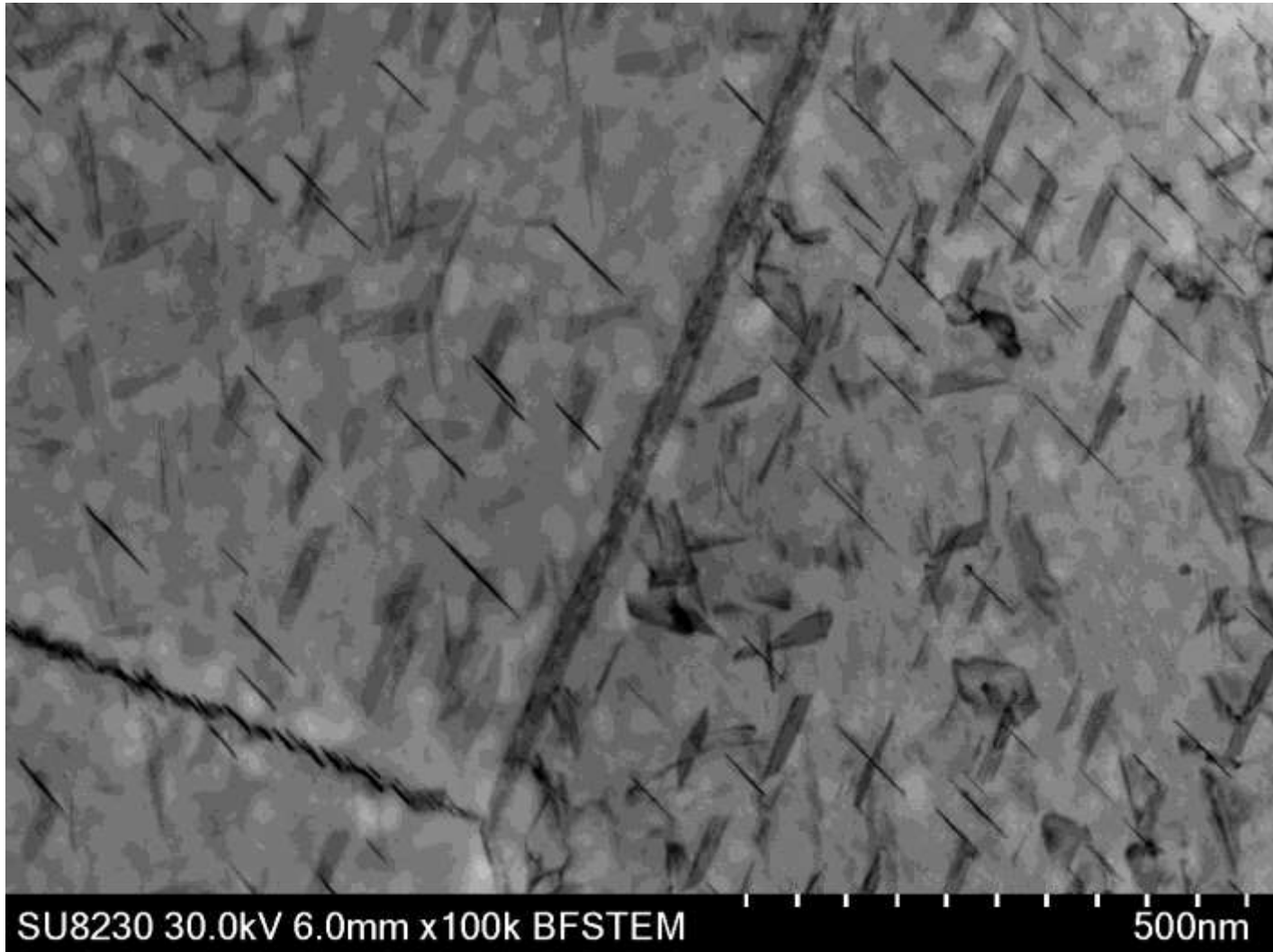
Dislocations in Ti-6Al-4V



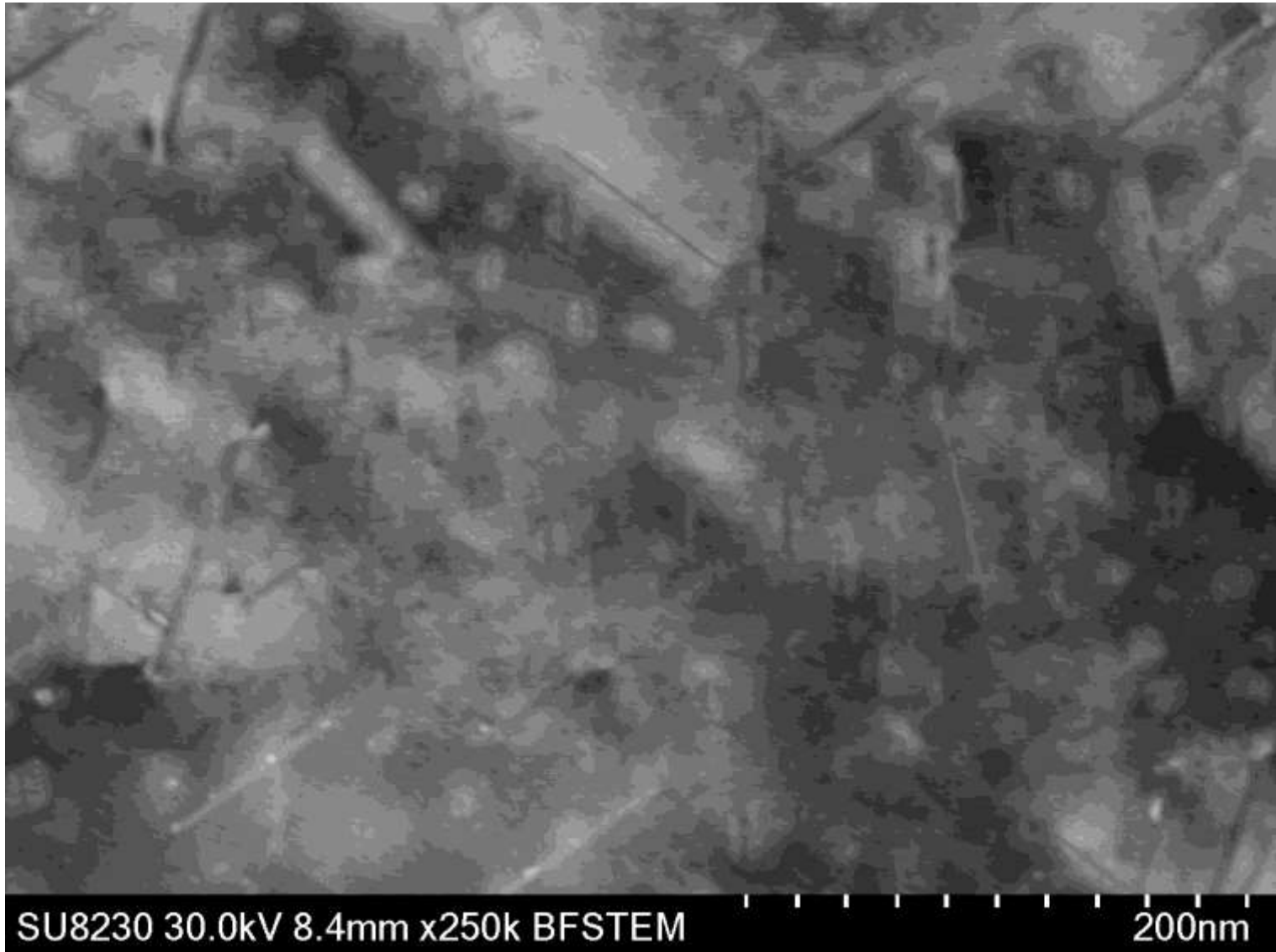
Dislocations in Ti-6Al-4V



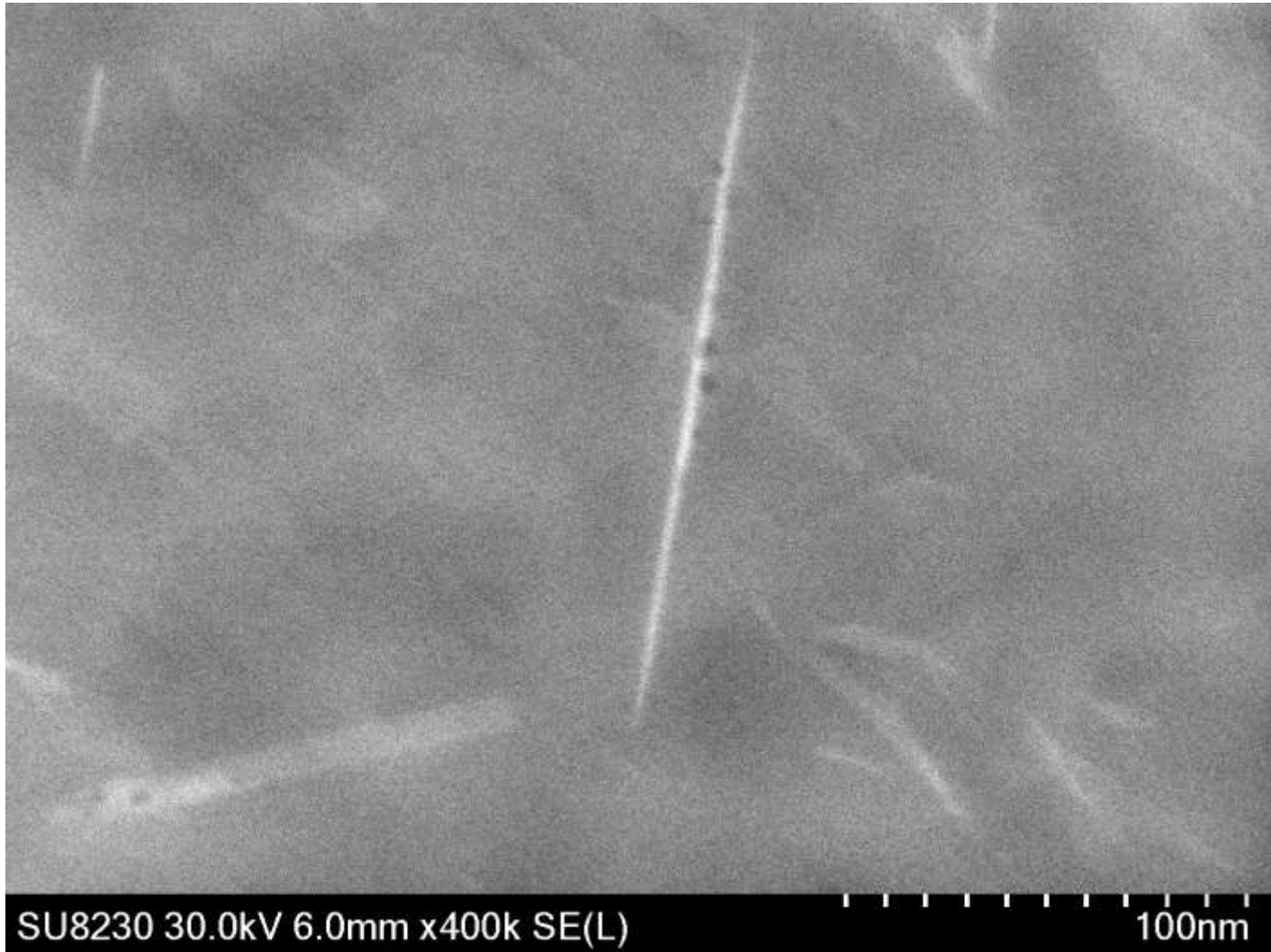
Al 2099, 30 keV, BF



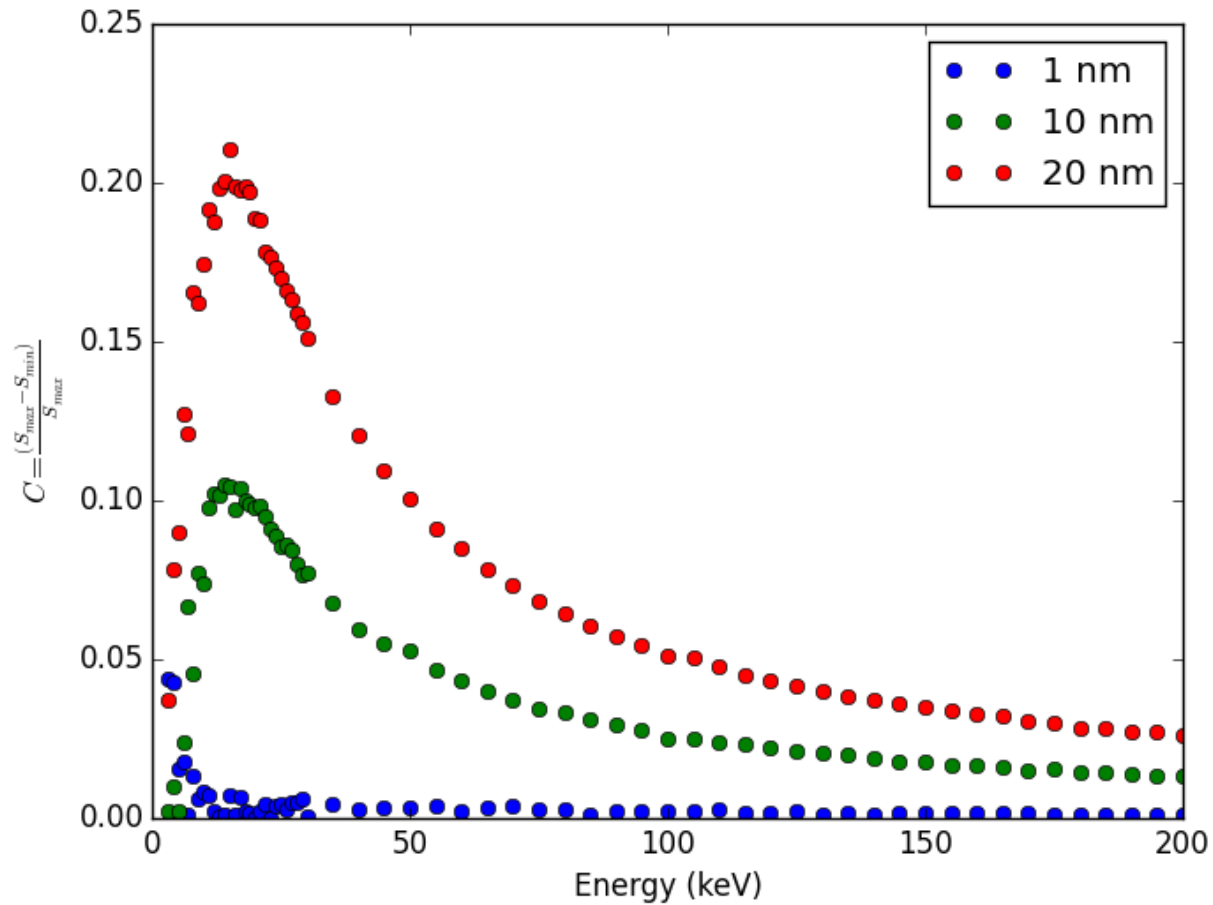
Strain Field around δ' ?



Al 2099, 30 keV, DF



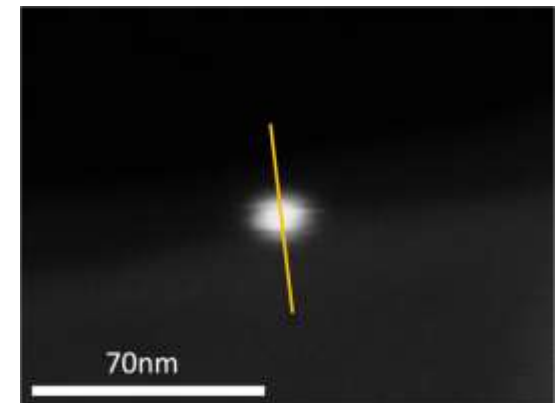
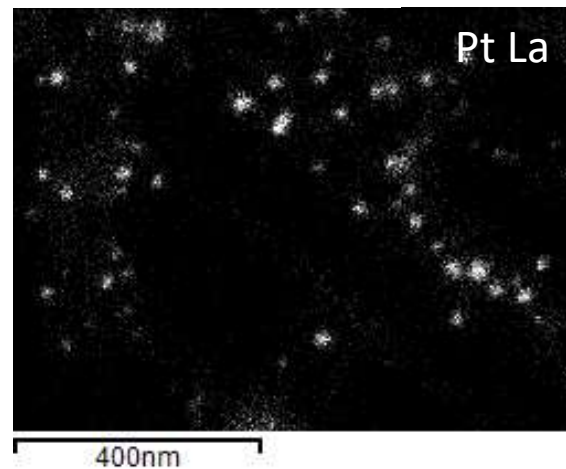
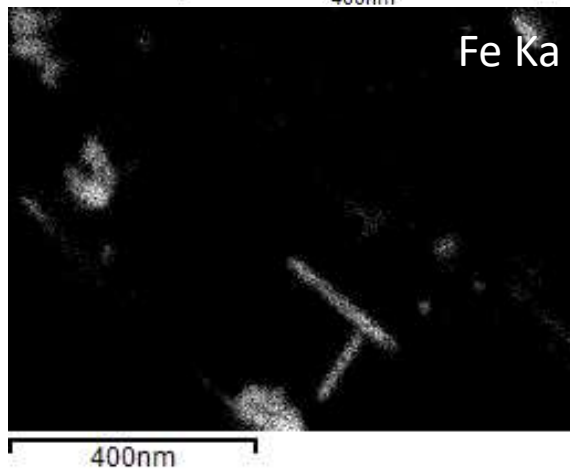
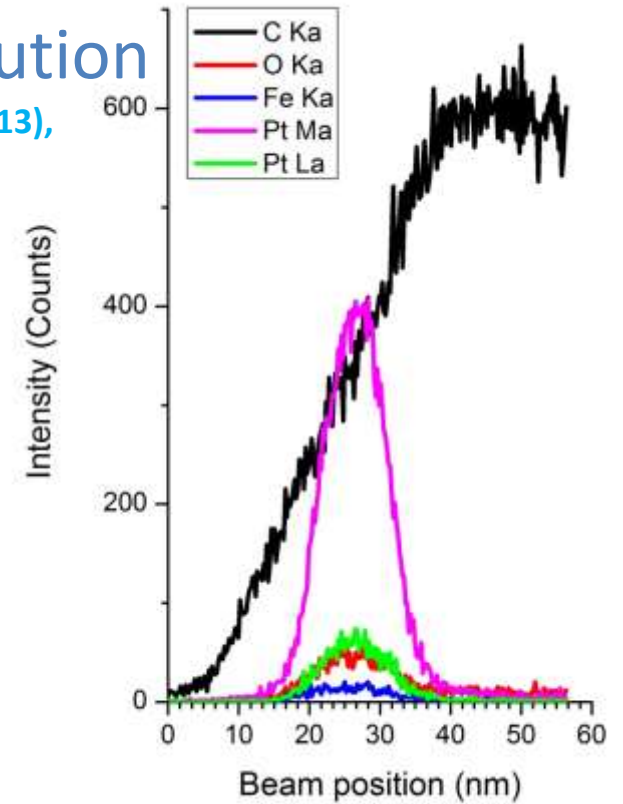
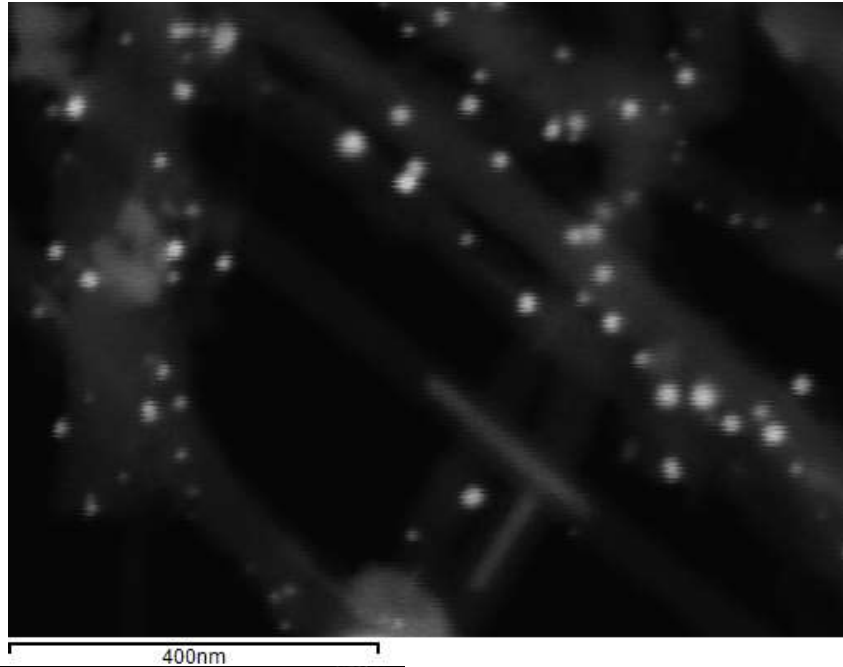
δ' (Al_3Li) in 80 nm Al, 11 MeV



Pt particulates on Carbon nanotubes

BSE – 30 keV – EDS analysis at high resolution

M. J.-F., Guinel, N. Brodusch, Y. Verde-Gomez, B. Escobar-Morales, R. Gauvin (2013),
Journal of Microscopy, Vol. 252, No. 1, pp. 49 – 57.



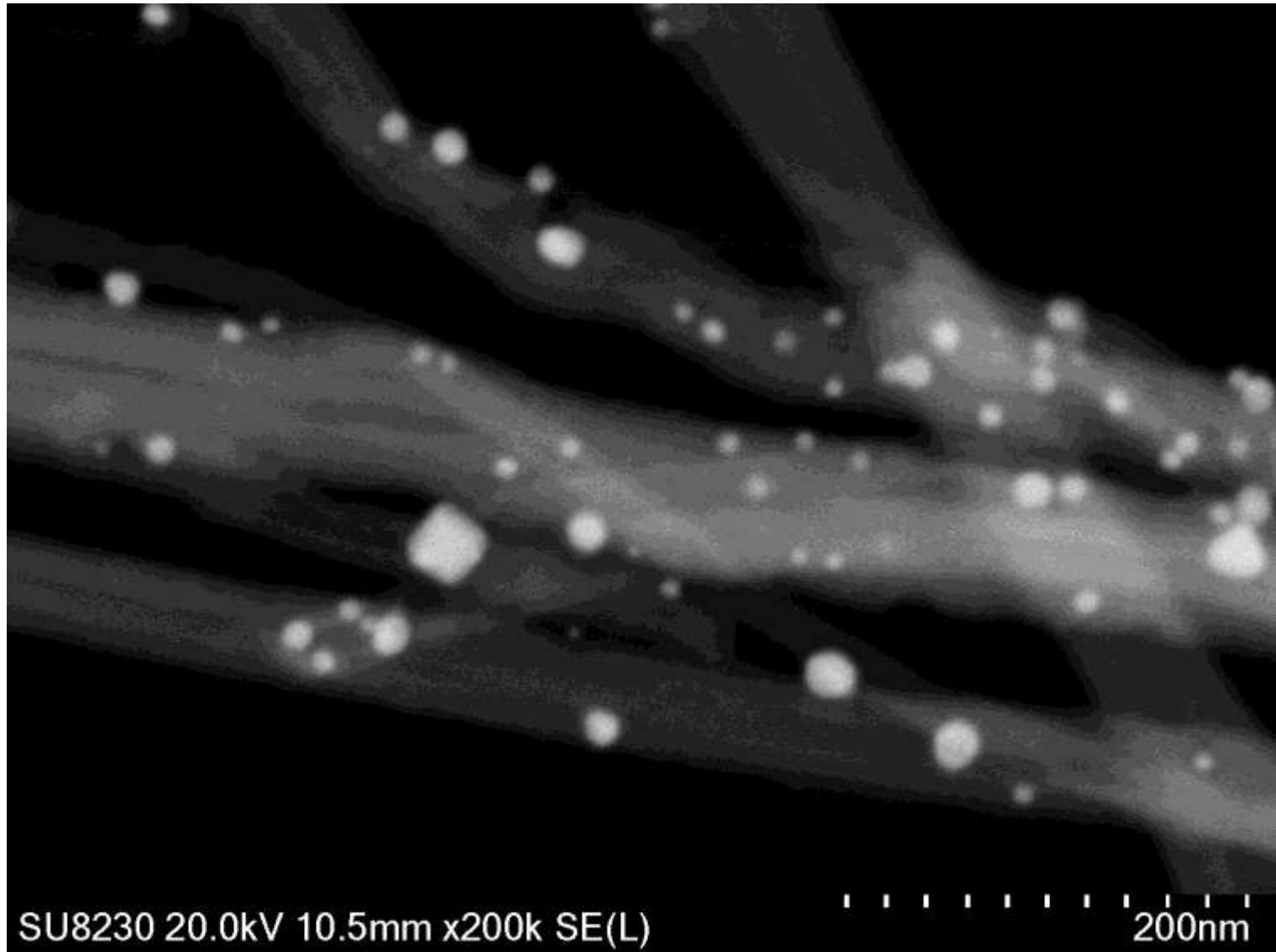
Bruker Flat Quad XFlash 5060F (BFQ)

- Quad detector
 - $4 \times 15 \text{ mm}^2 = 60 \text{ mm}^2$
- Annular design (below objective lens)
- Equipped with Mylar windows
 - Internal window ($1 \mu\text{m}$)
 - Additional $2 \mu\text{m}$ or $6 \mu\text{m}$ windows (on a special changer)
- High count rate capabilities with high collection efficiency



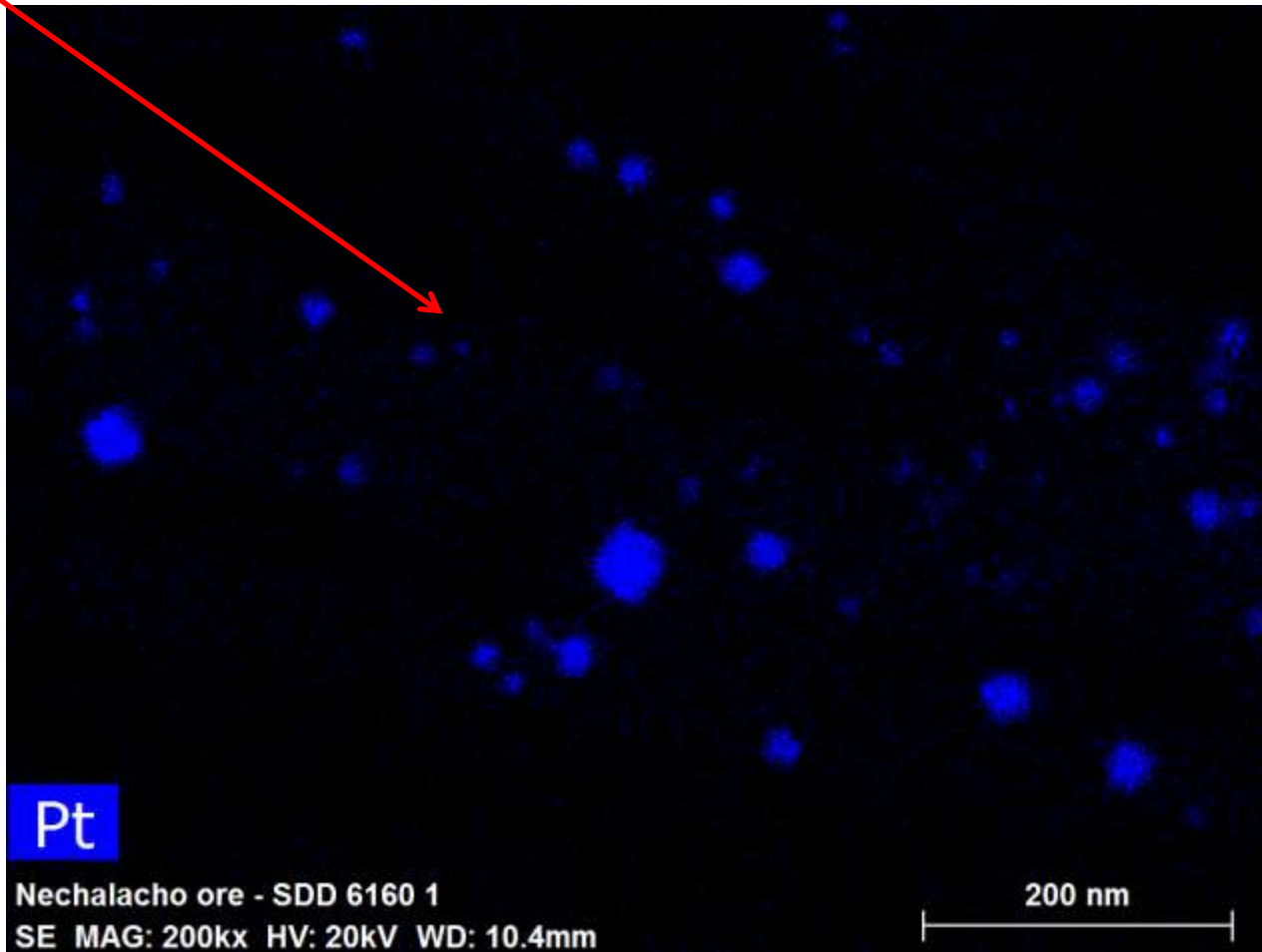
Image from Bruker Nano GmbH.

Pt Particulates on WCNT

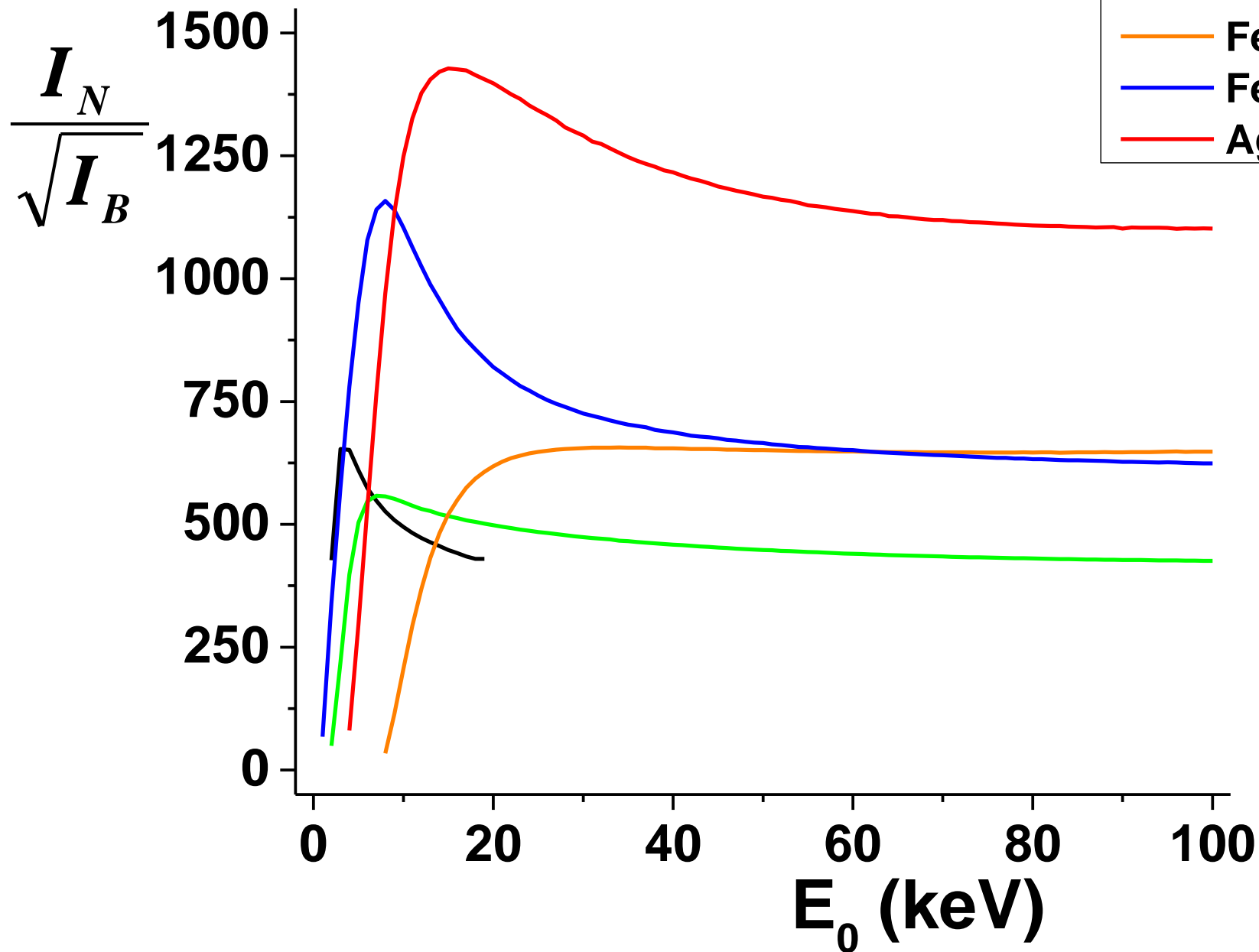


Pt Particulates on WCNT, 7 minutes

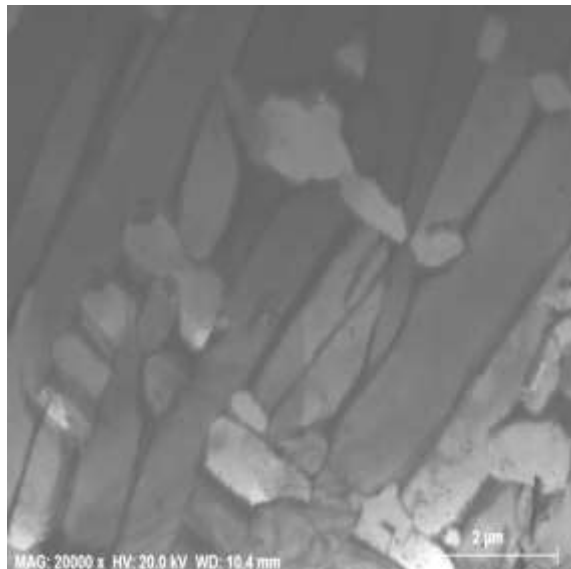
6 nm



80 nm, 50 000 e, same dose

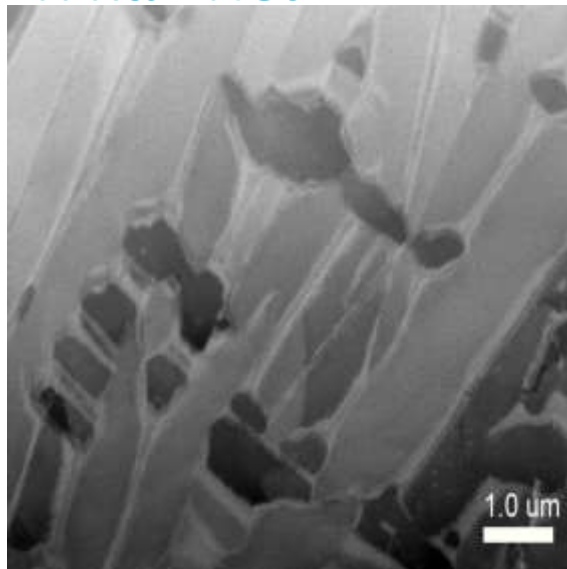


STEM-BF



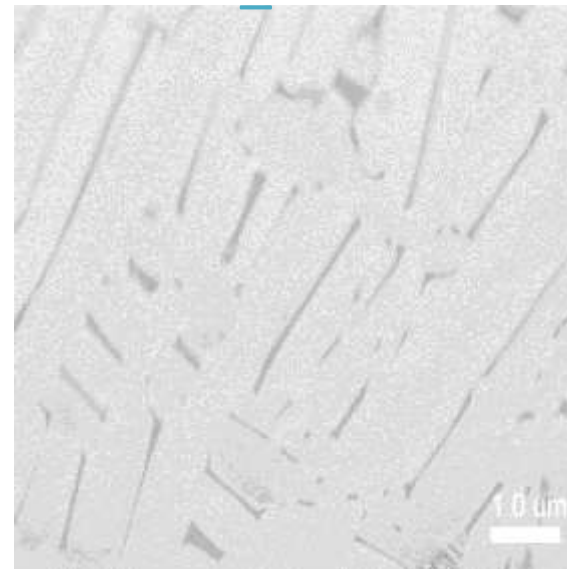
a

Ti K α - Net

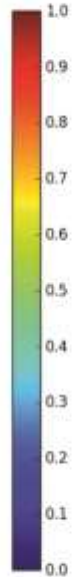
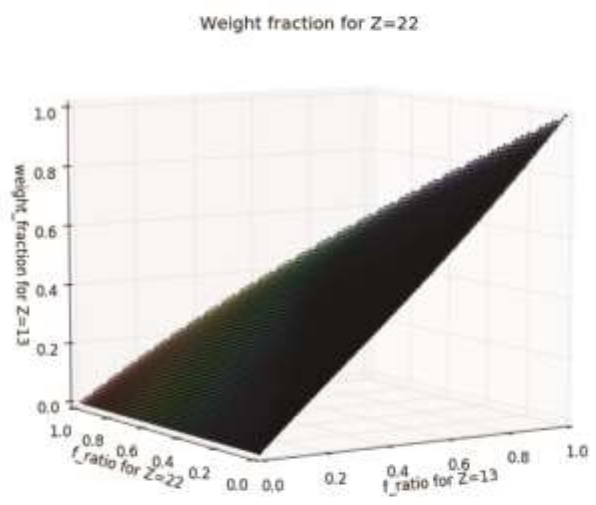


b

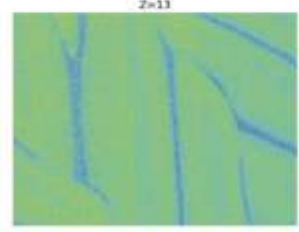
Ti K α - f_ratio



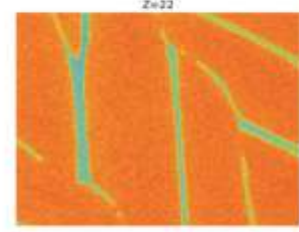
c



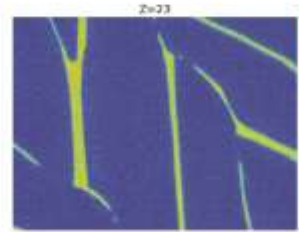
Al



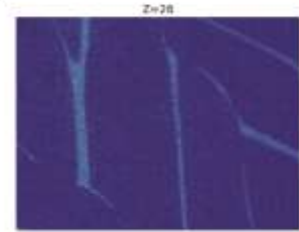
Ti



V



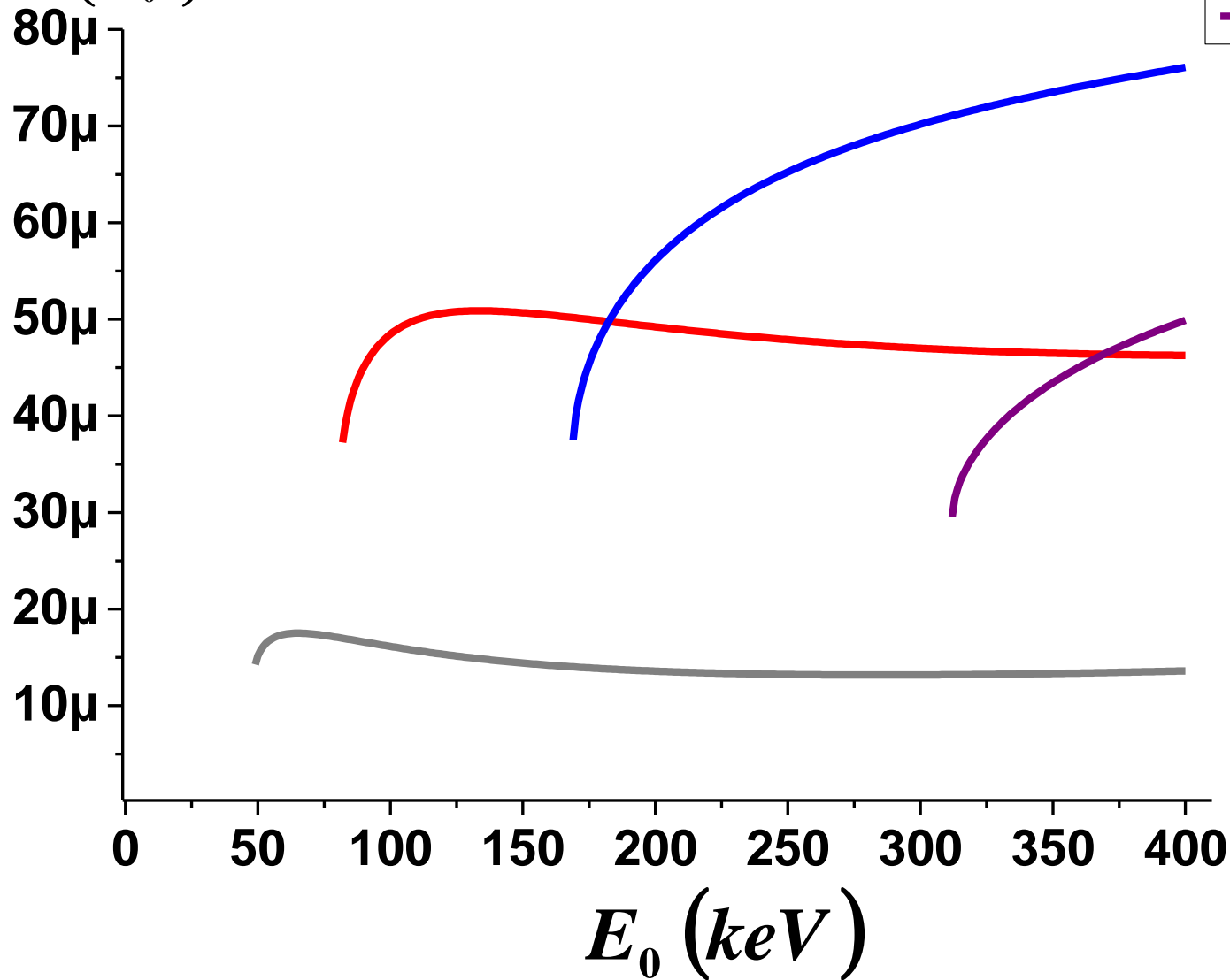
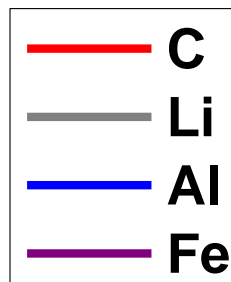
Fe



e

$$P_{Disp} = \frac{1 - e^{-t/\lambda}}{1 + \left(\frac{\theta_{min}}{\theta_0}\right)^2}$$

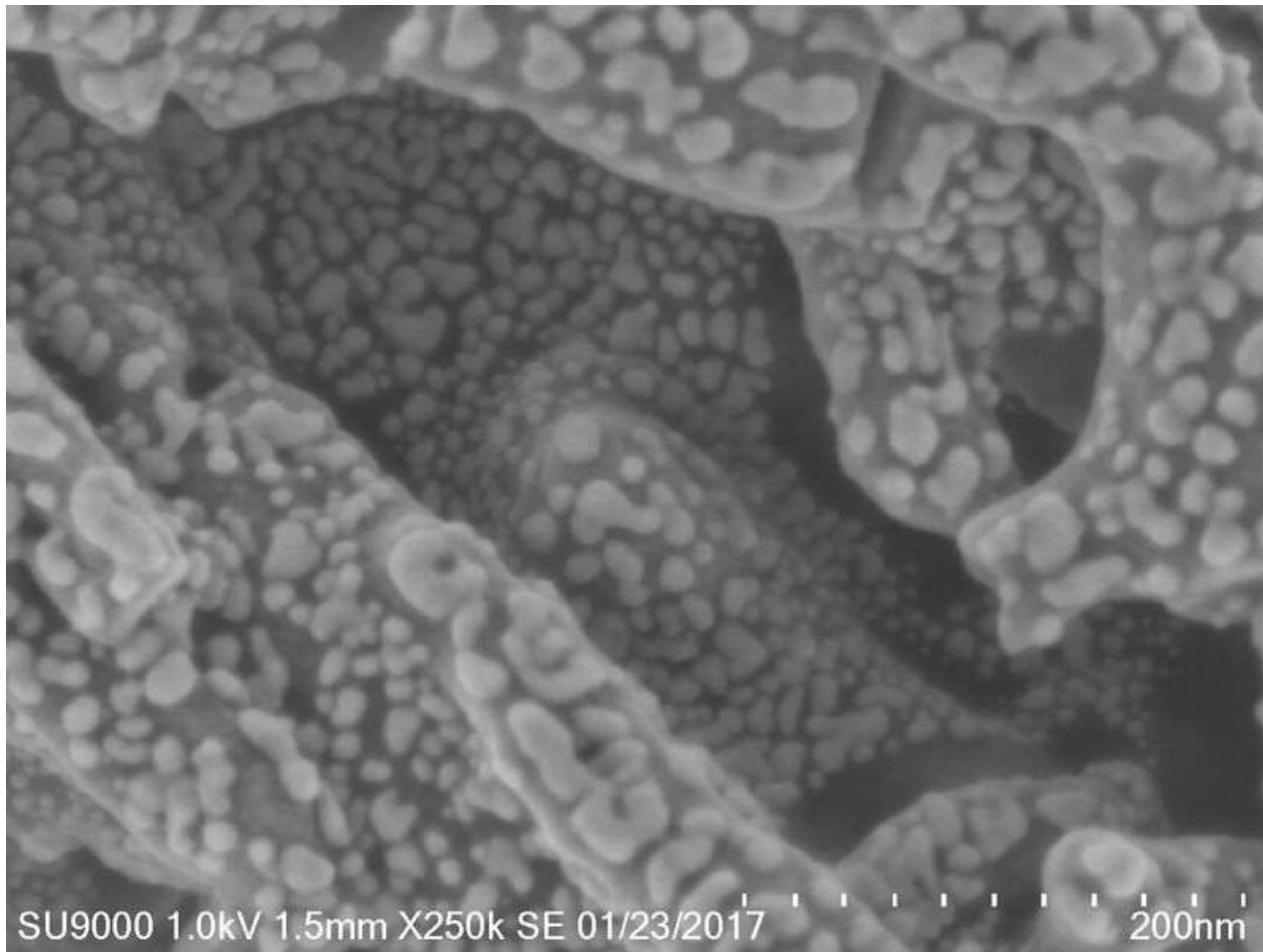
100 nm, $\Delta E_{Disp} = 15$ eV



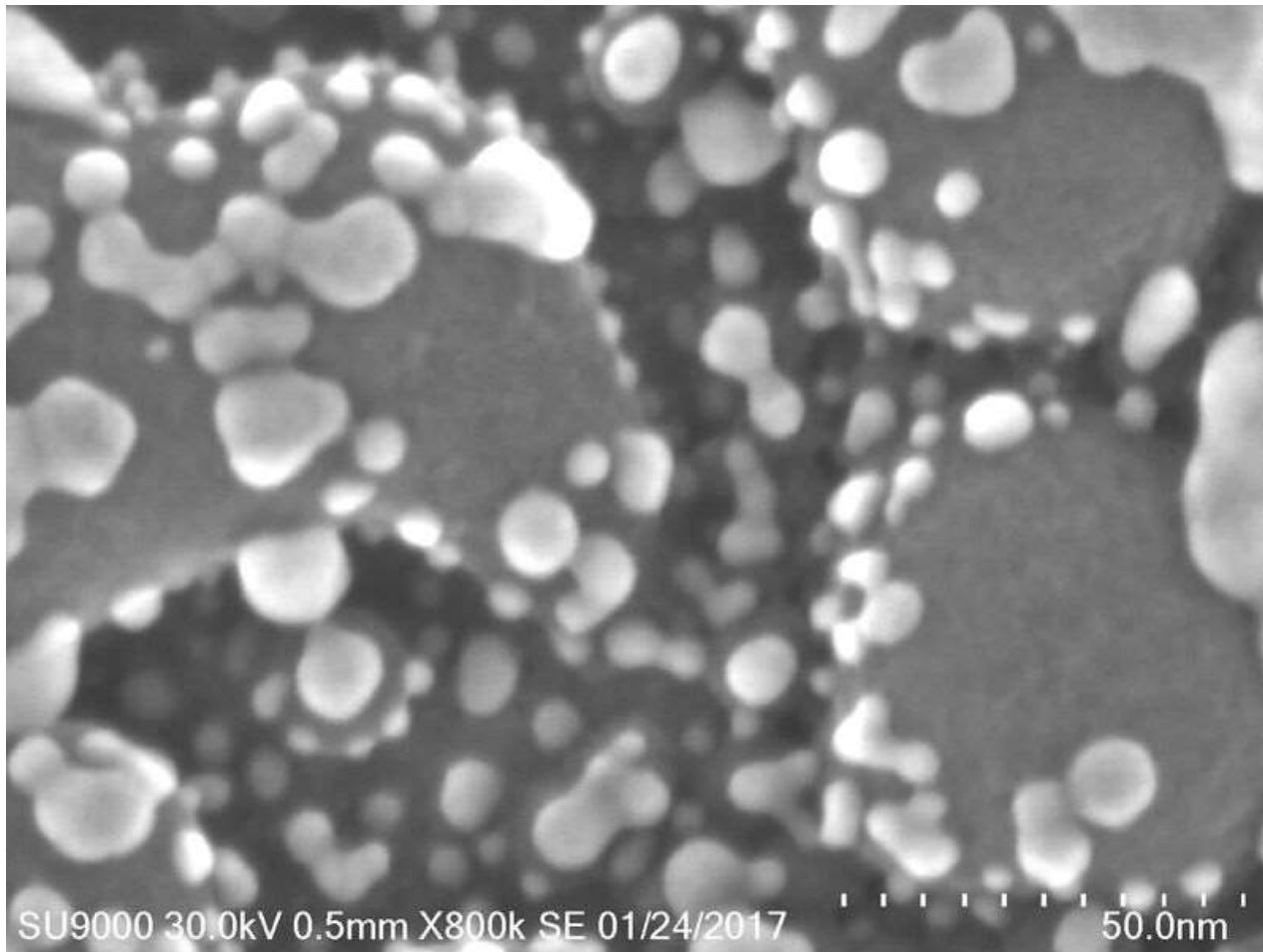
SU-9000EA STEM



Magnetic Tape, 1 keV, SE

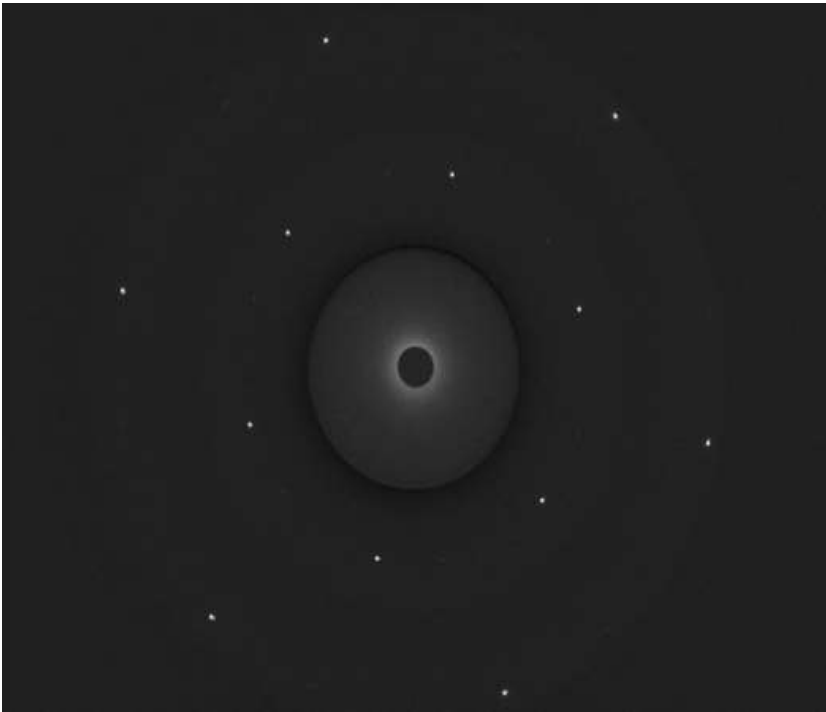


Magnetic Tape, 30 keV, SE

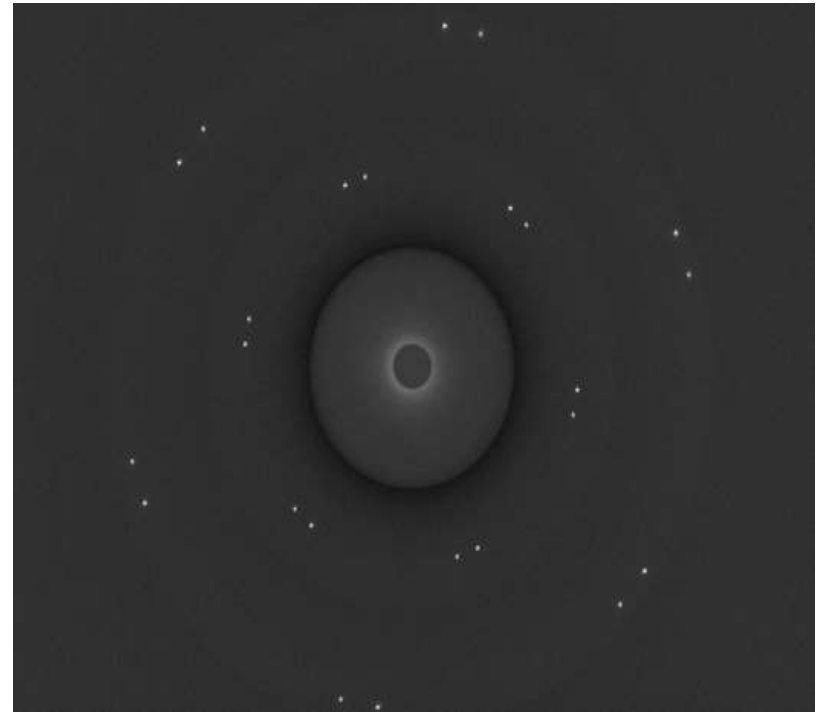


Graphene Spot Diffraction

1 Layer

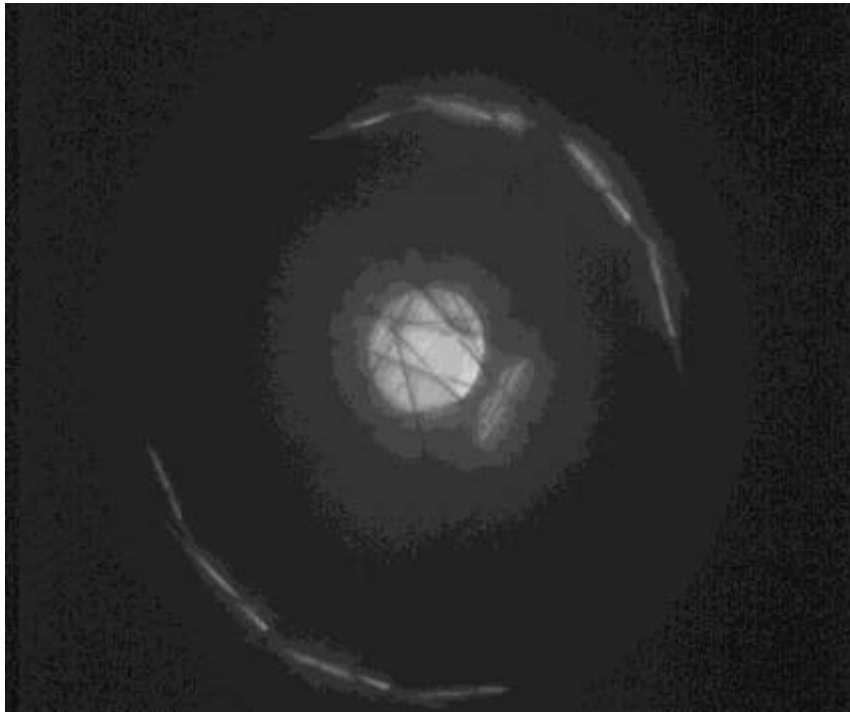


2 Layers

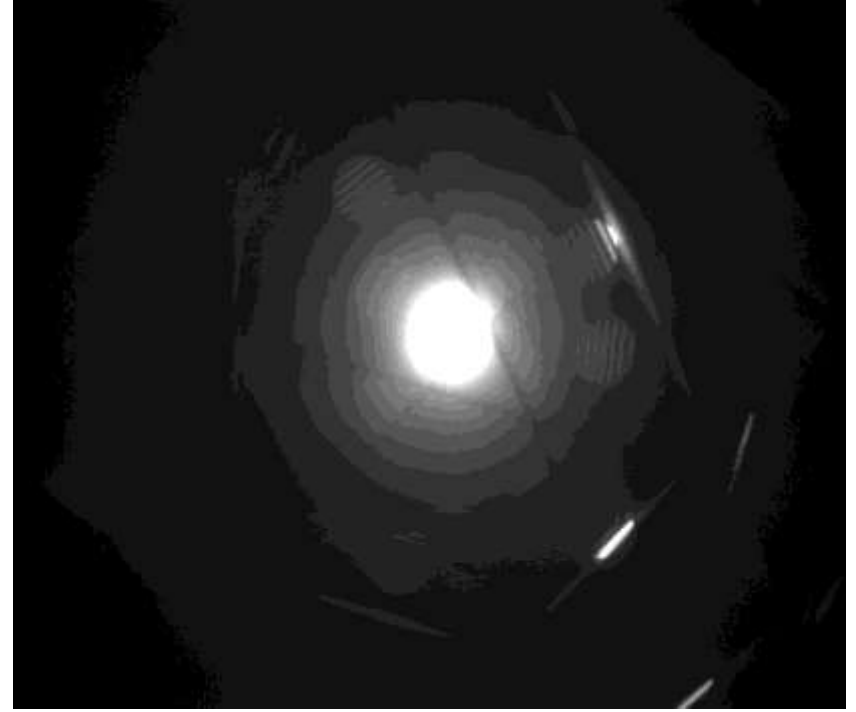


CBED Diffraction of Thicker Samples

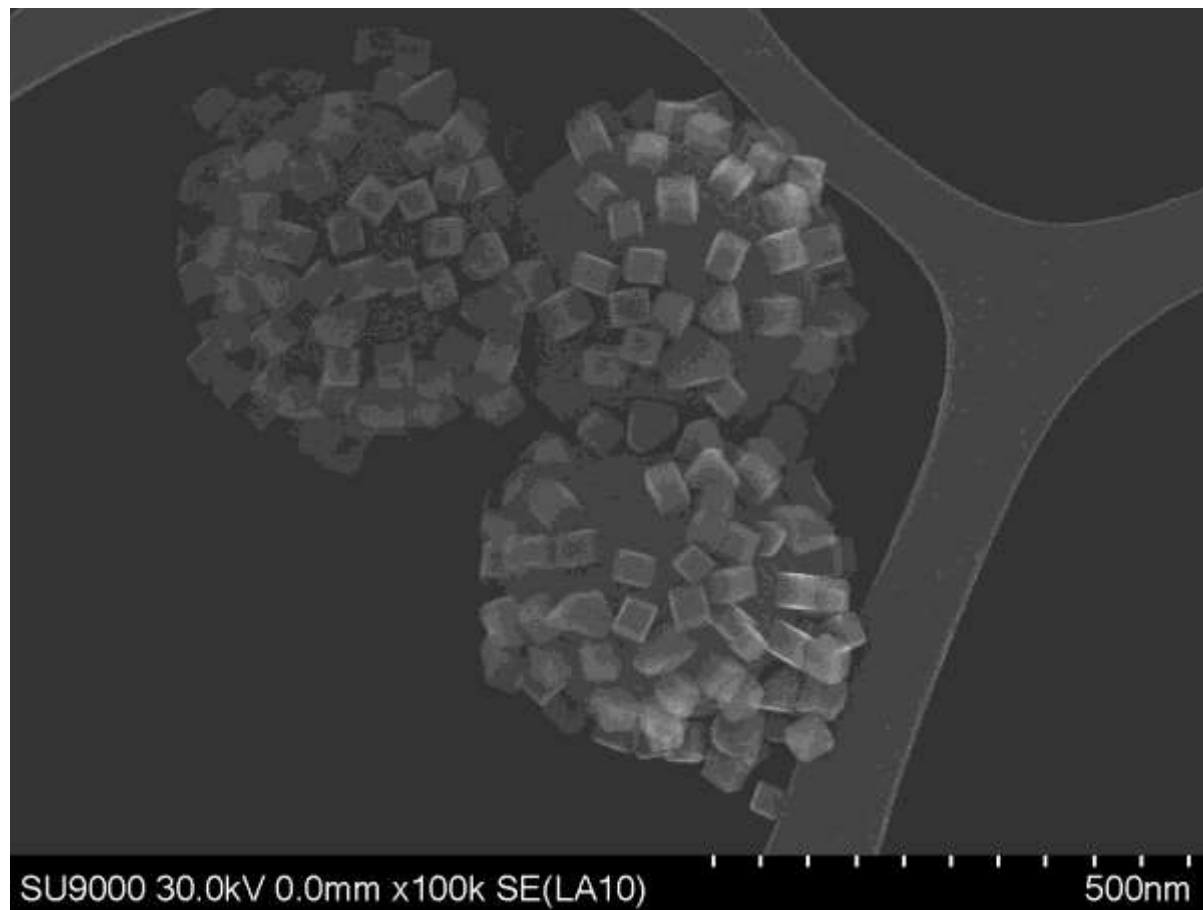
MgO



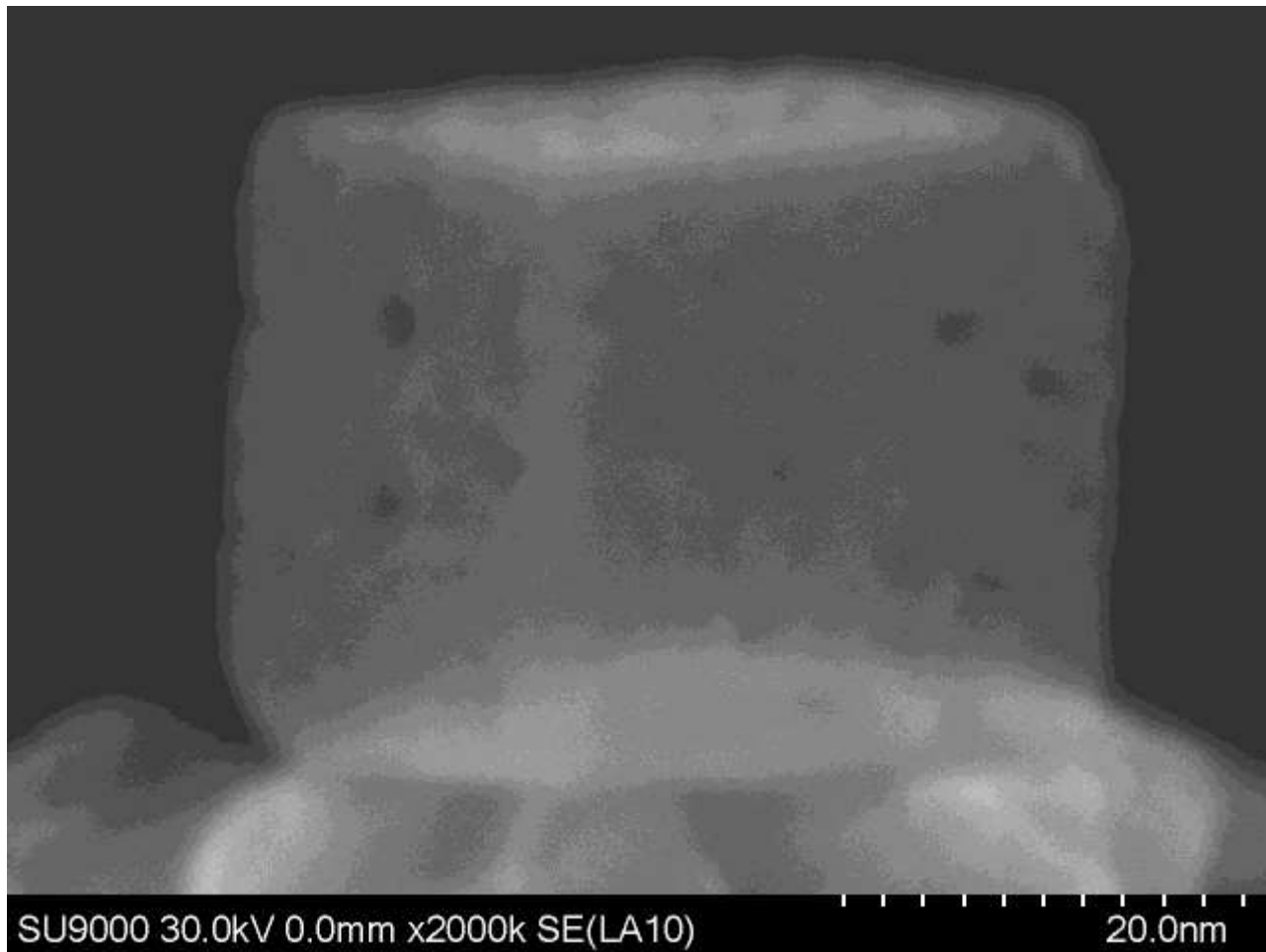
Al-Li alloy



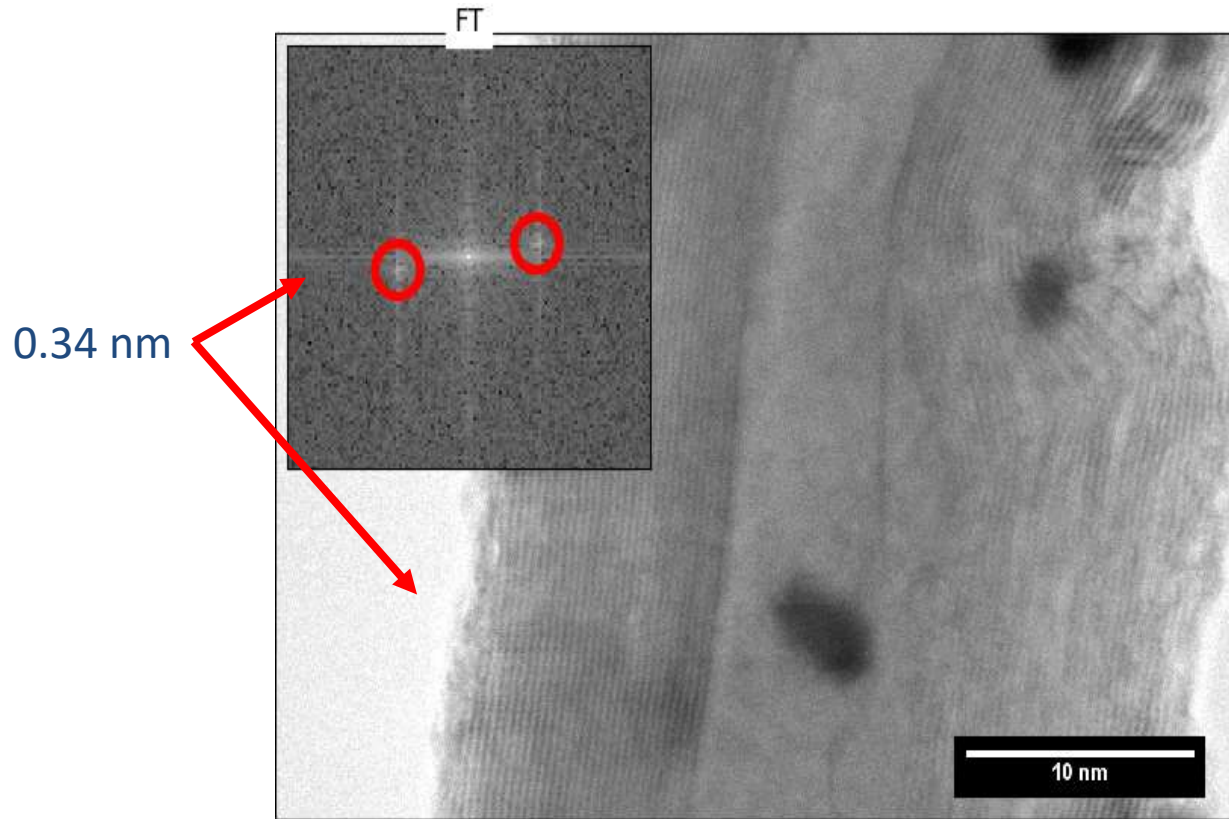
Fe₂O₃ Nanocubes



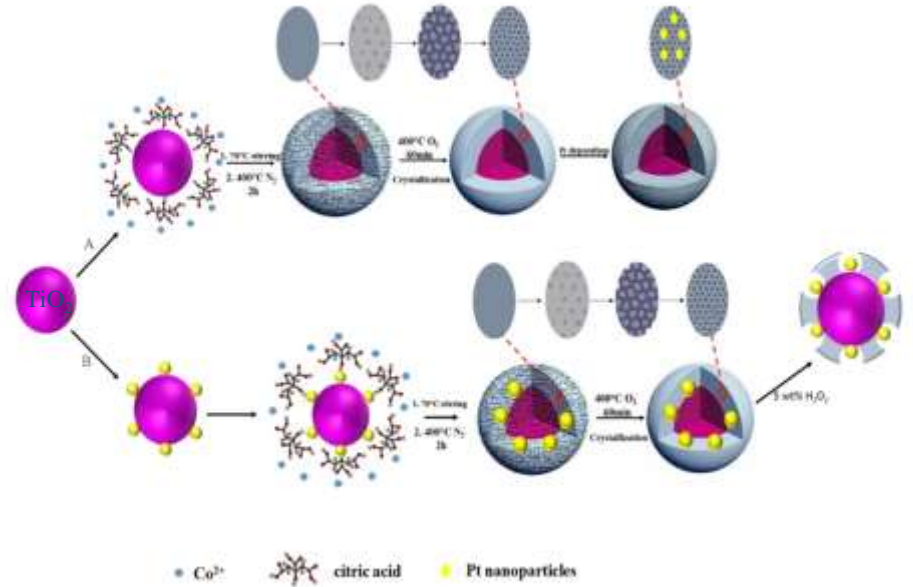
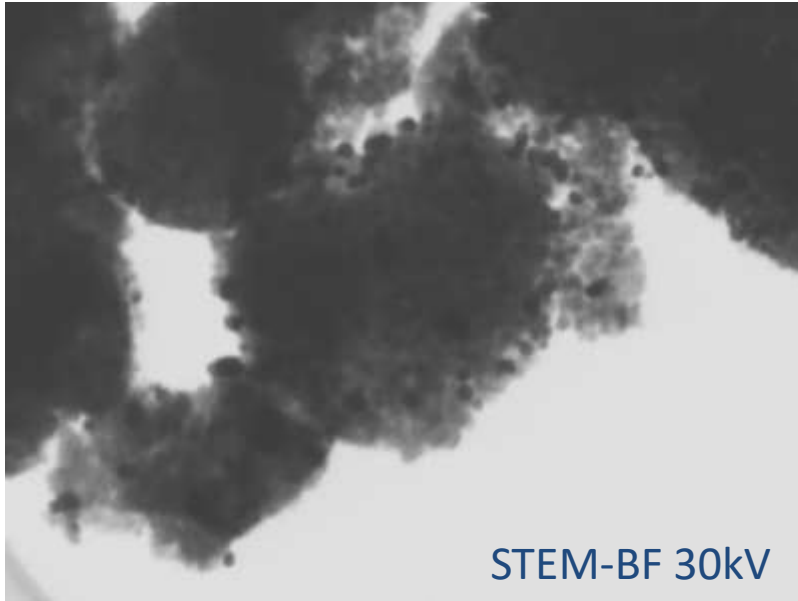
Fe₂O₃ Nanocubes



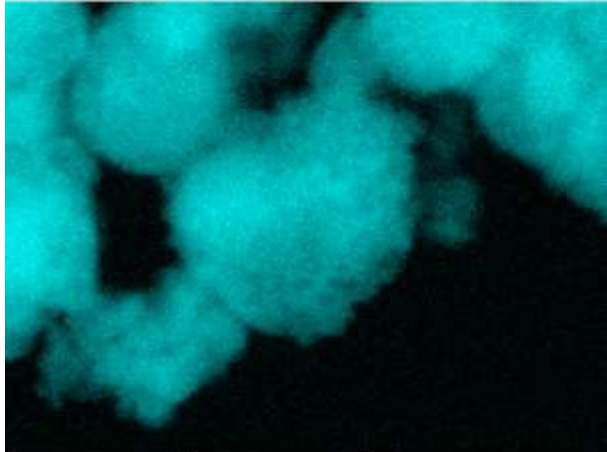
Lattice Imaging of Ni-Pt CNT



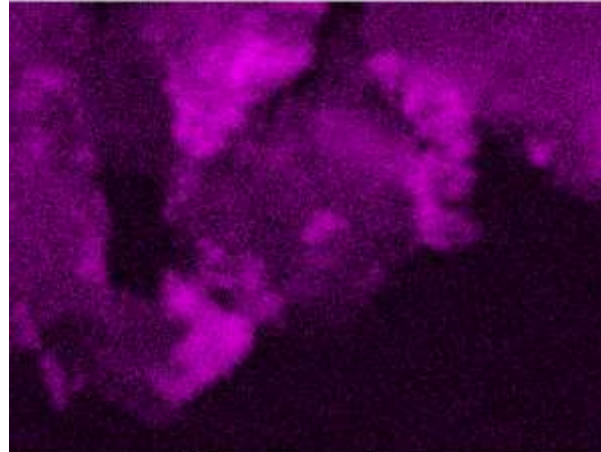
Core-shell nanocomposite



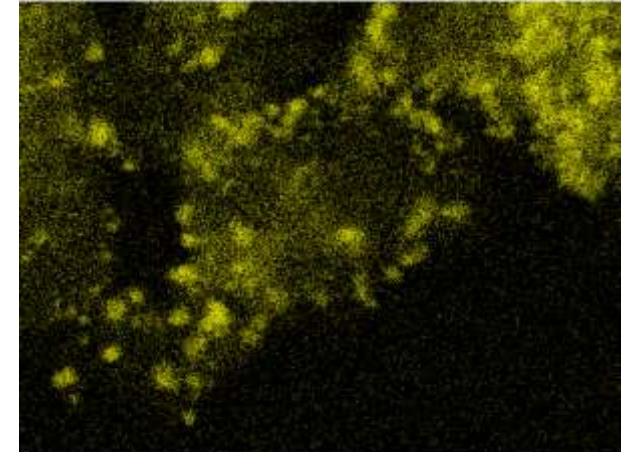
Ti K series



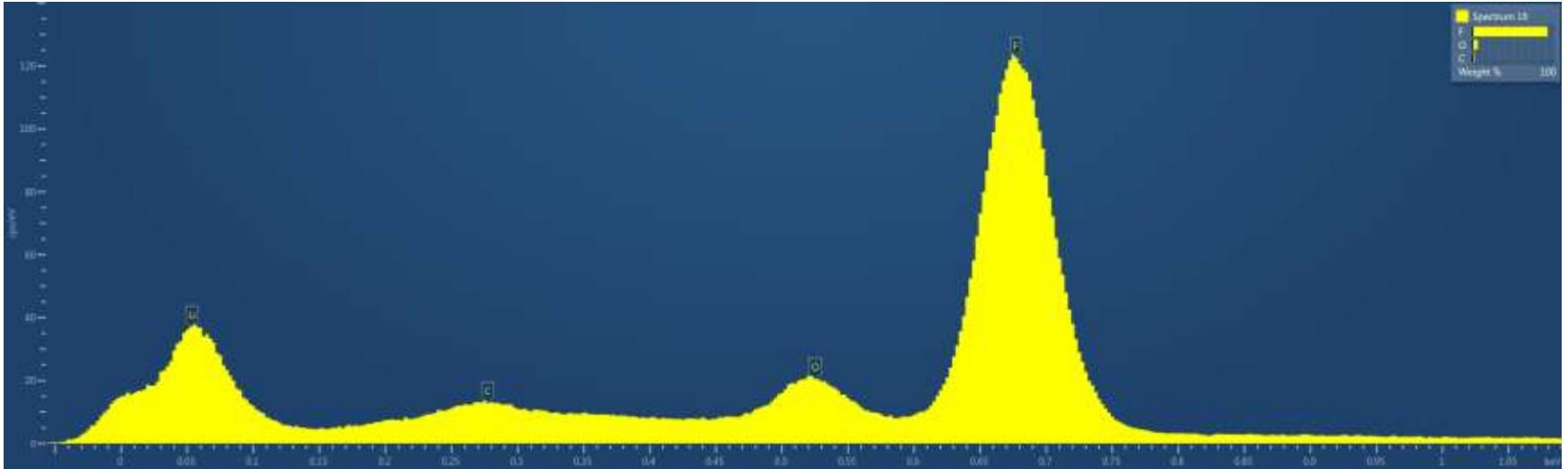
Co K series



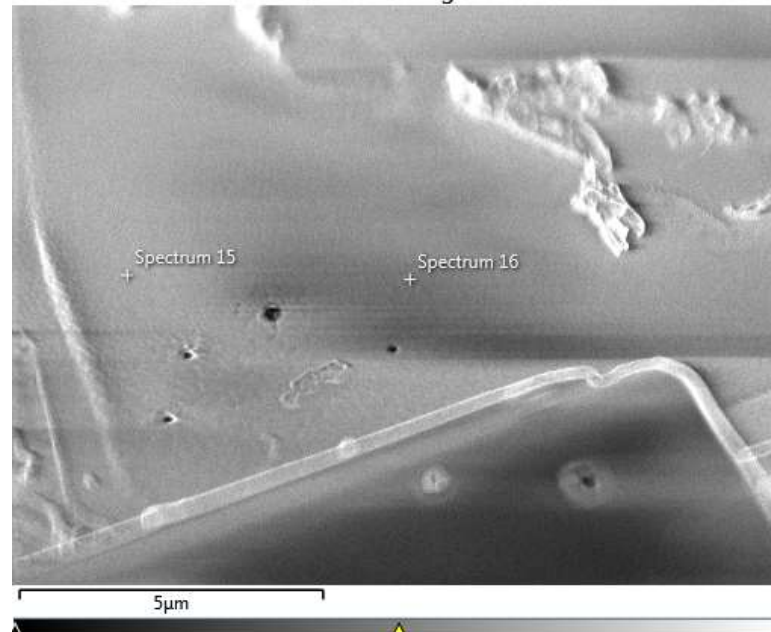
Pt L series



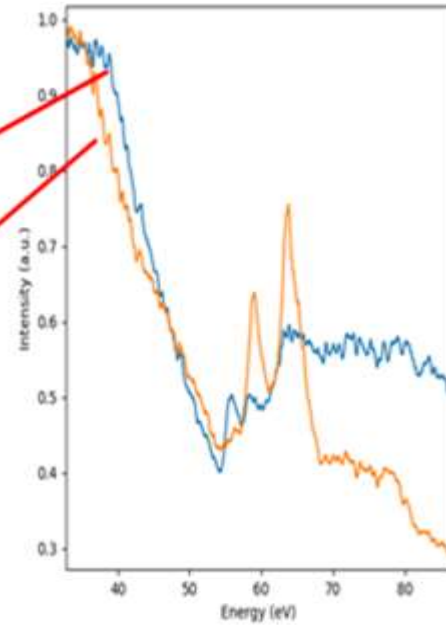
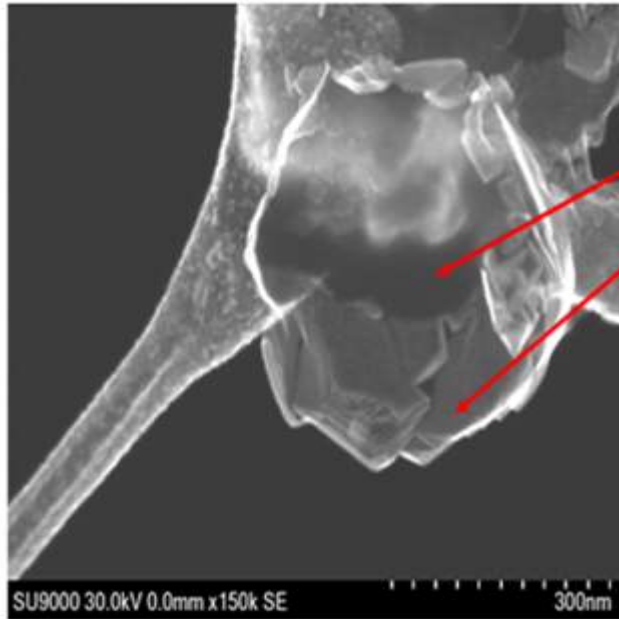
LiF with Extreme EDS



Electron Image 5

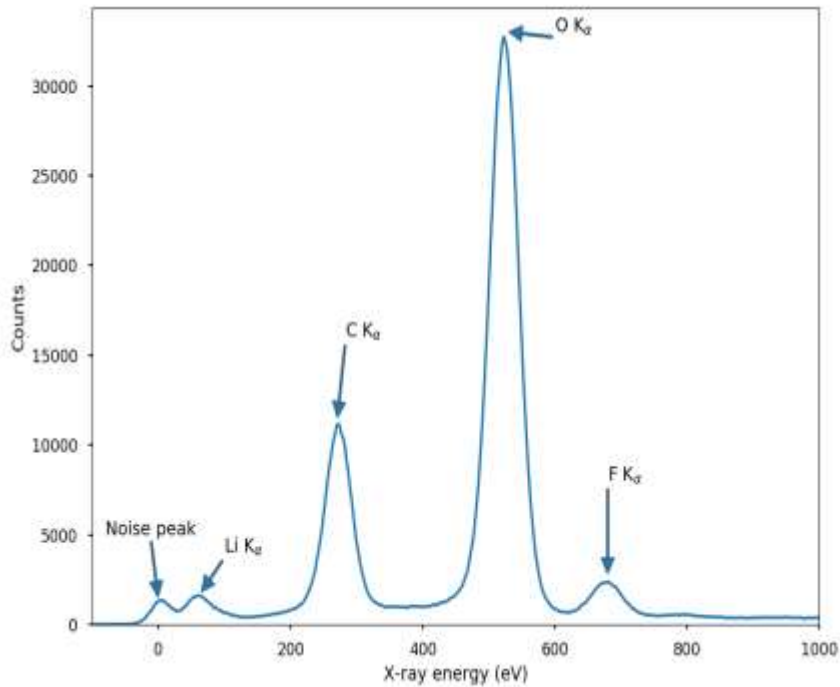


Li Detection in LiO_2

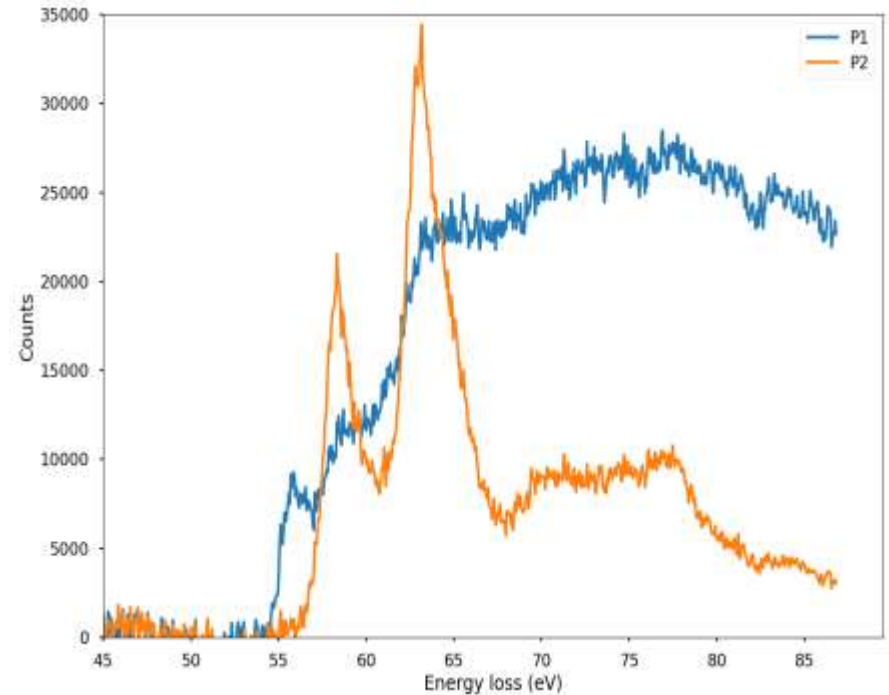


Lithium Analysis

2 kV Windowless SDD

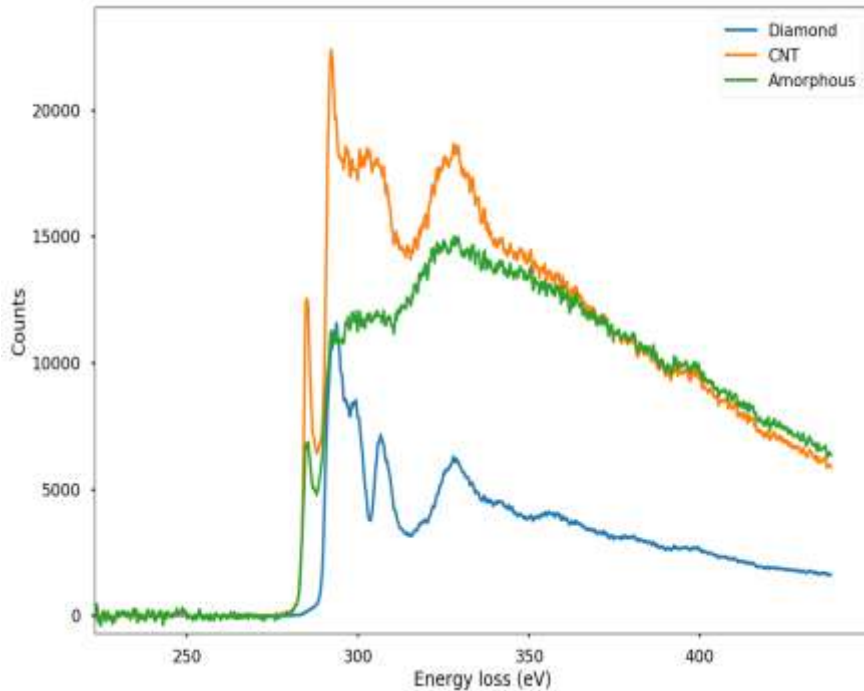


30 kV EELS

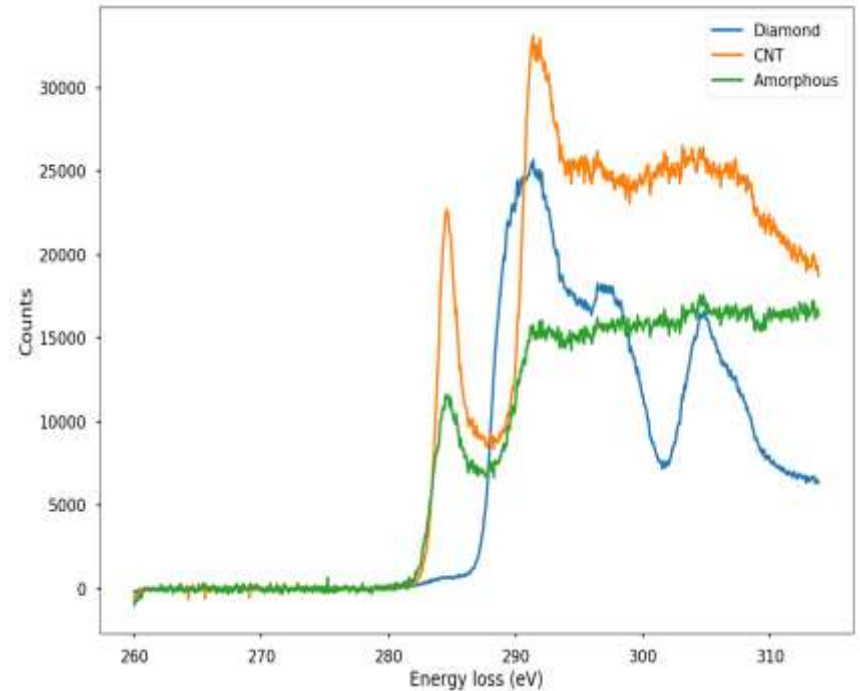


Carbon K Edge Shape

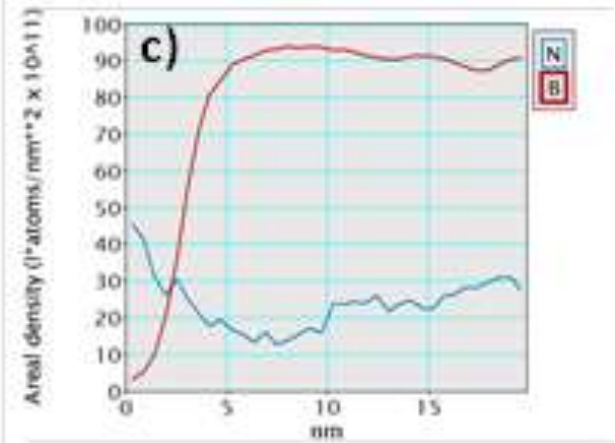
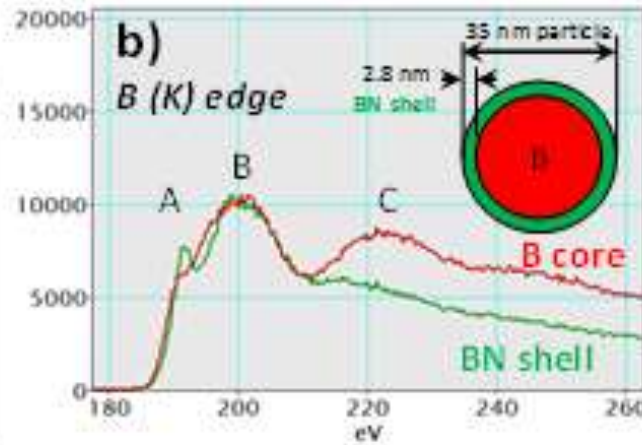
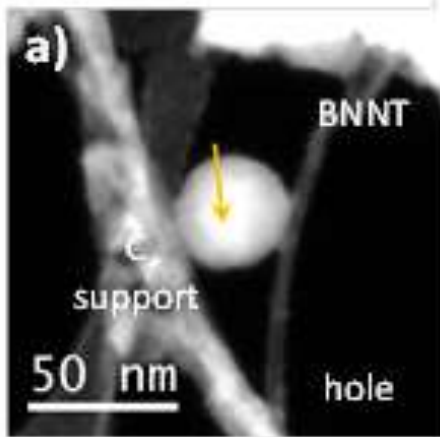
Energy range: 231 eV



Energy range: 54 eV

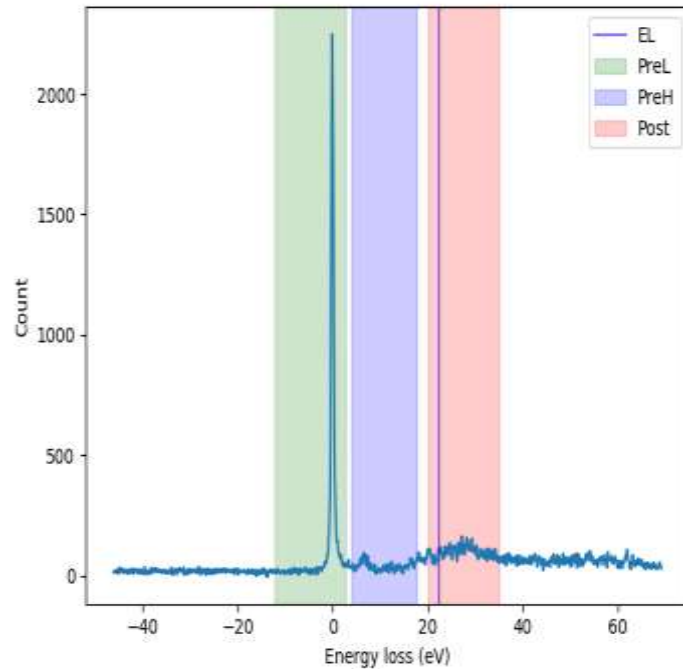


BN on B Nanotubes



EF-STEM of Zero Loss and Plasmon

EELS Spectrum



Zero Loss Image



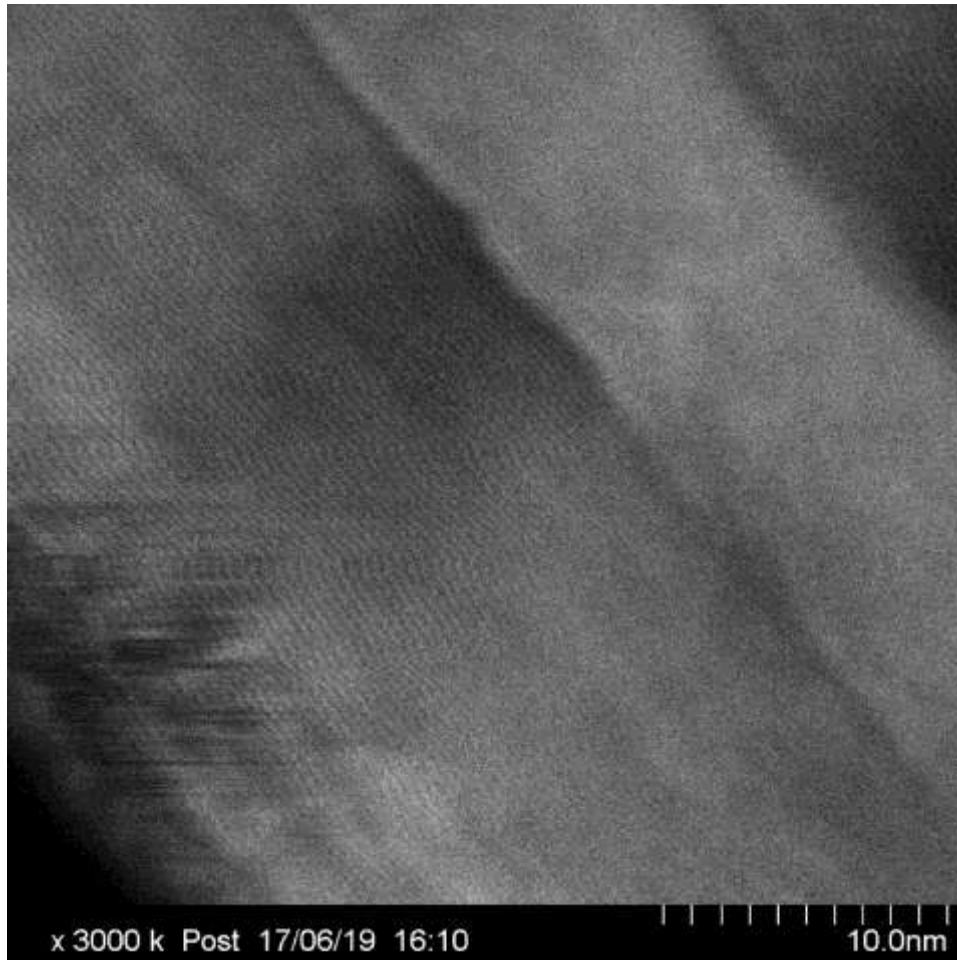
Plasmon image

post window (red)

x 350 k Post 17/04/27 15:01



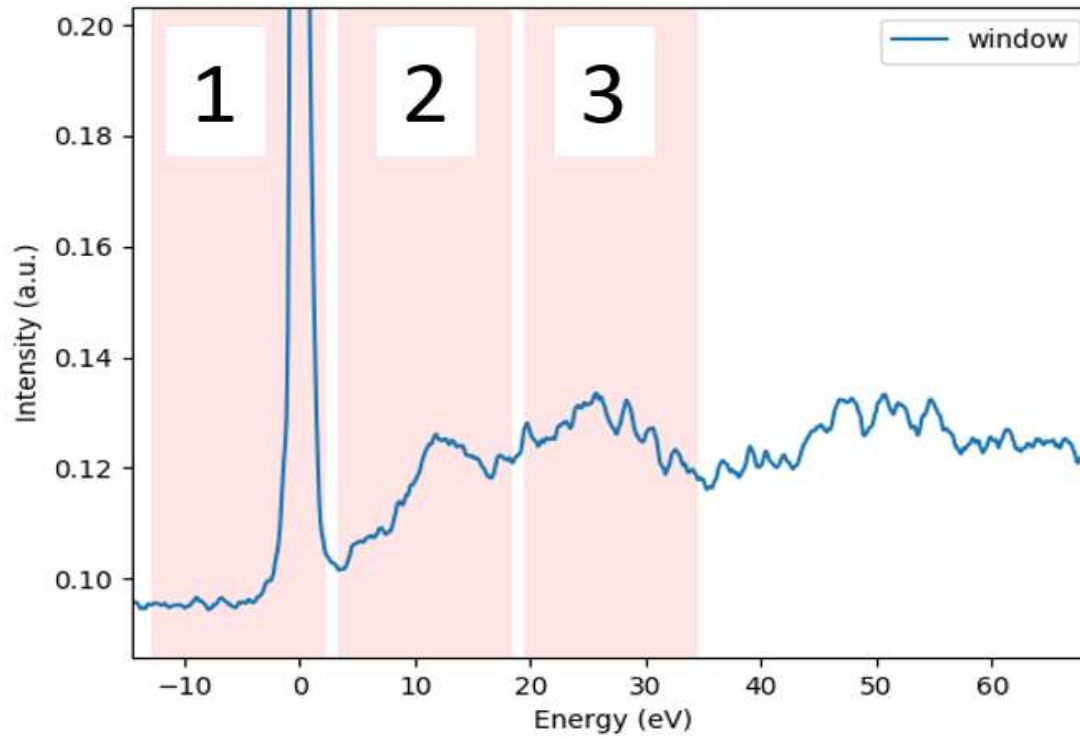
EELS Lattice Imaging of CNT



Plasmon
image

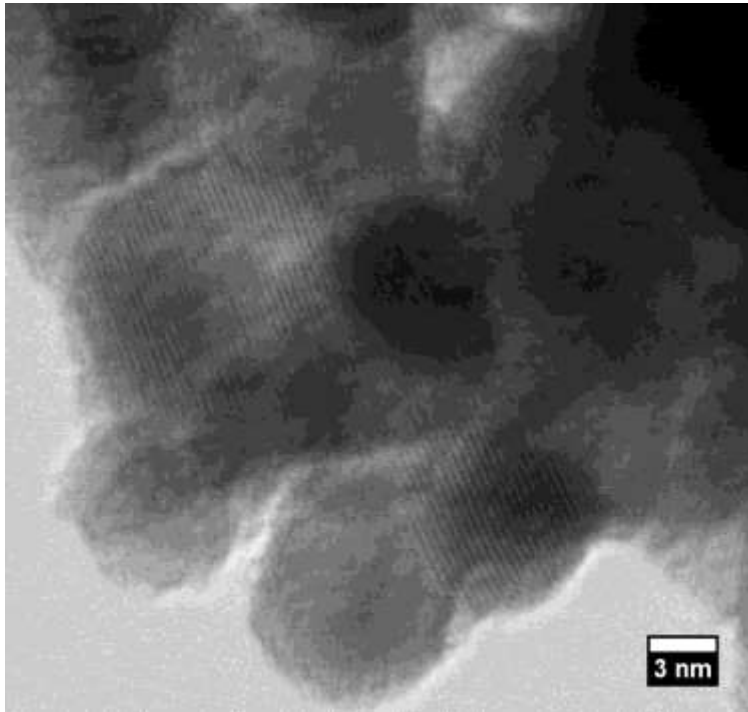
post window (red)

EELS Lattice Imaging of TiO_2 EFTEM Windows

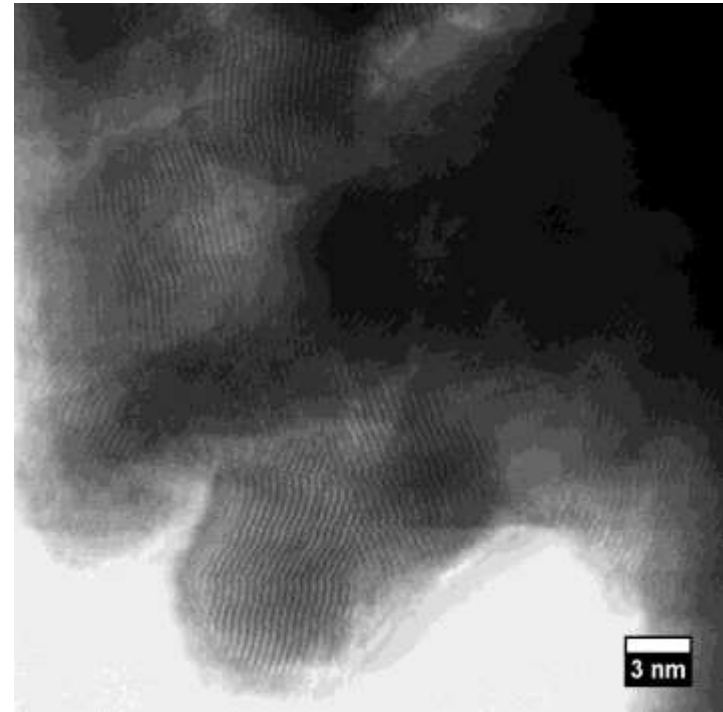


EELS Lattice Imaging of TiO₂

Bright-Field

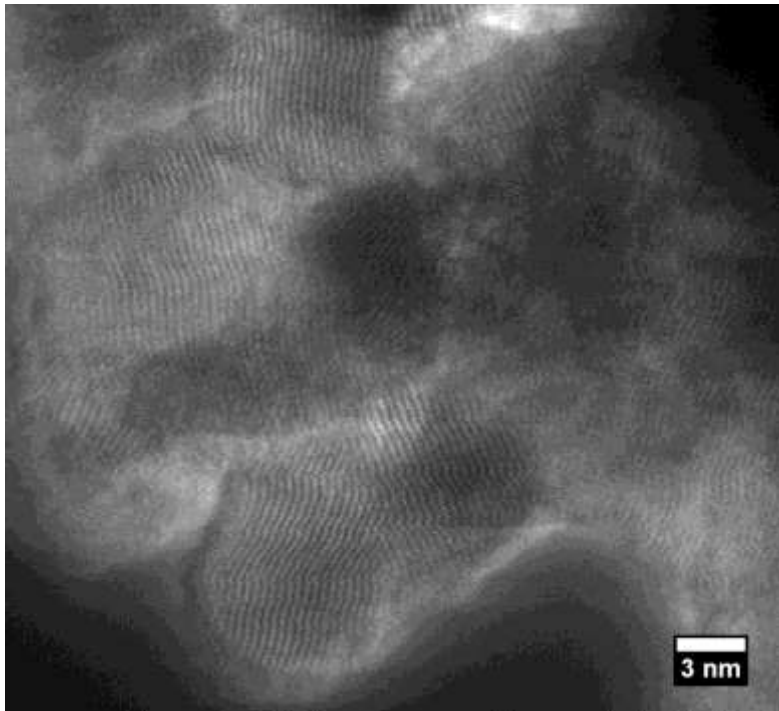


Zero Loss (1)

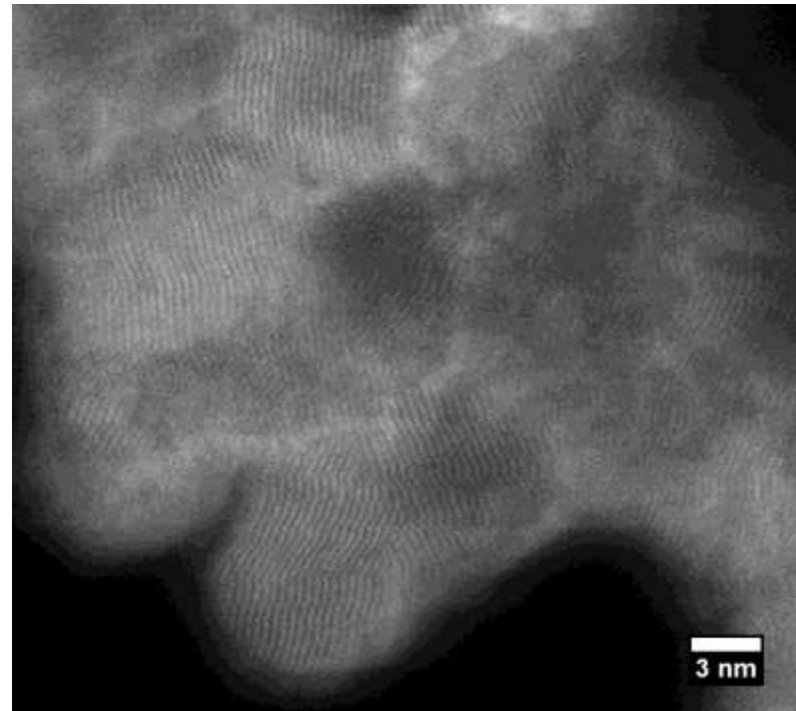


EELS Lattice Imaging of TiO₂

Surface Plasmon (2)

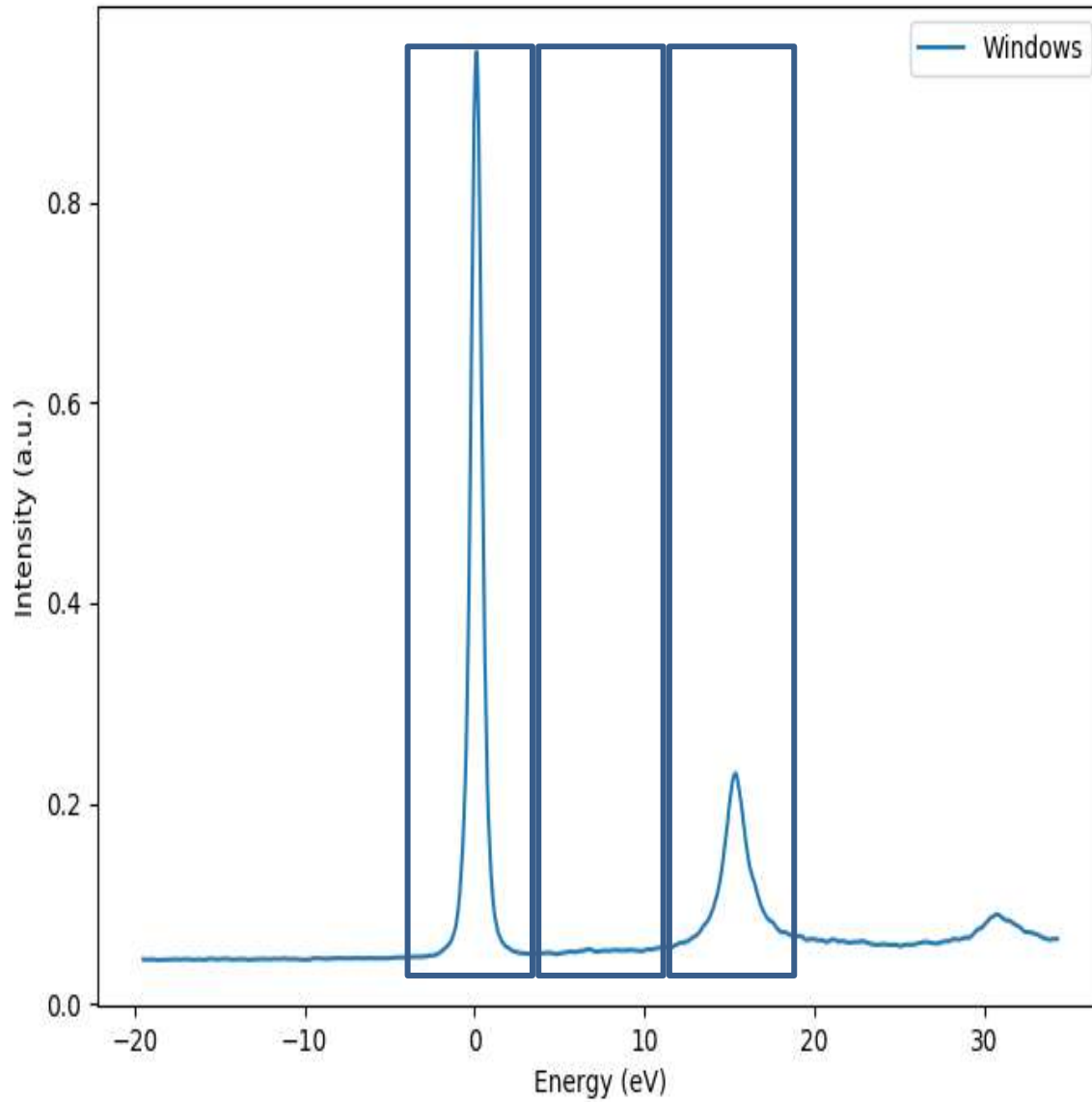


Bulk Plasmon (3)



Imaging of a Al 2190 Alloy

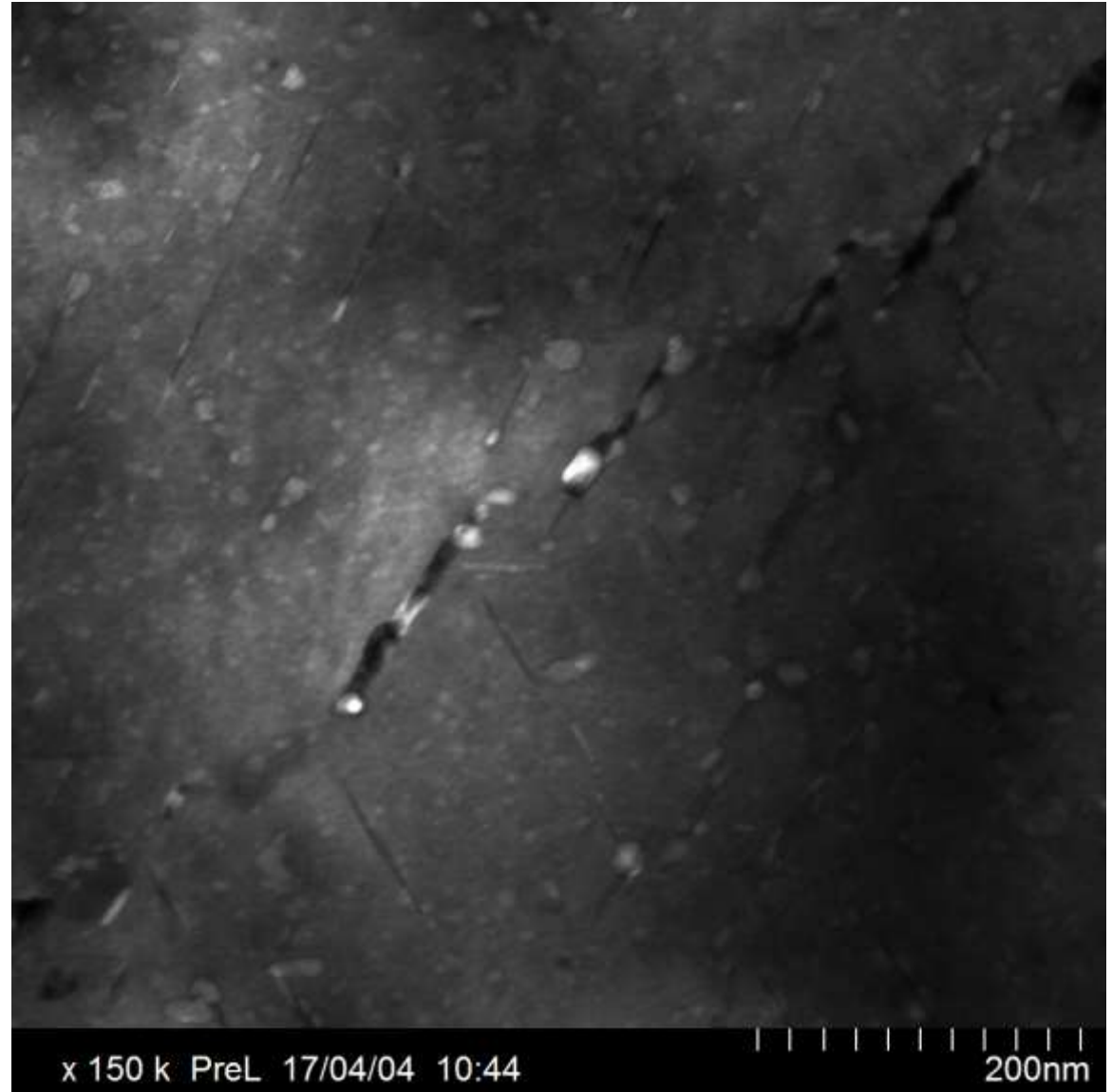
PreL PreH Post



Energy-filtered STEM - SU9000 (30 kV)

AA2099 – Al-Li-Cu

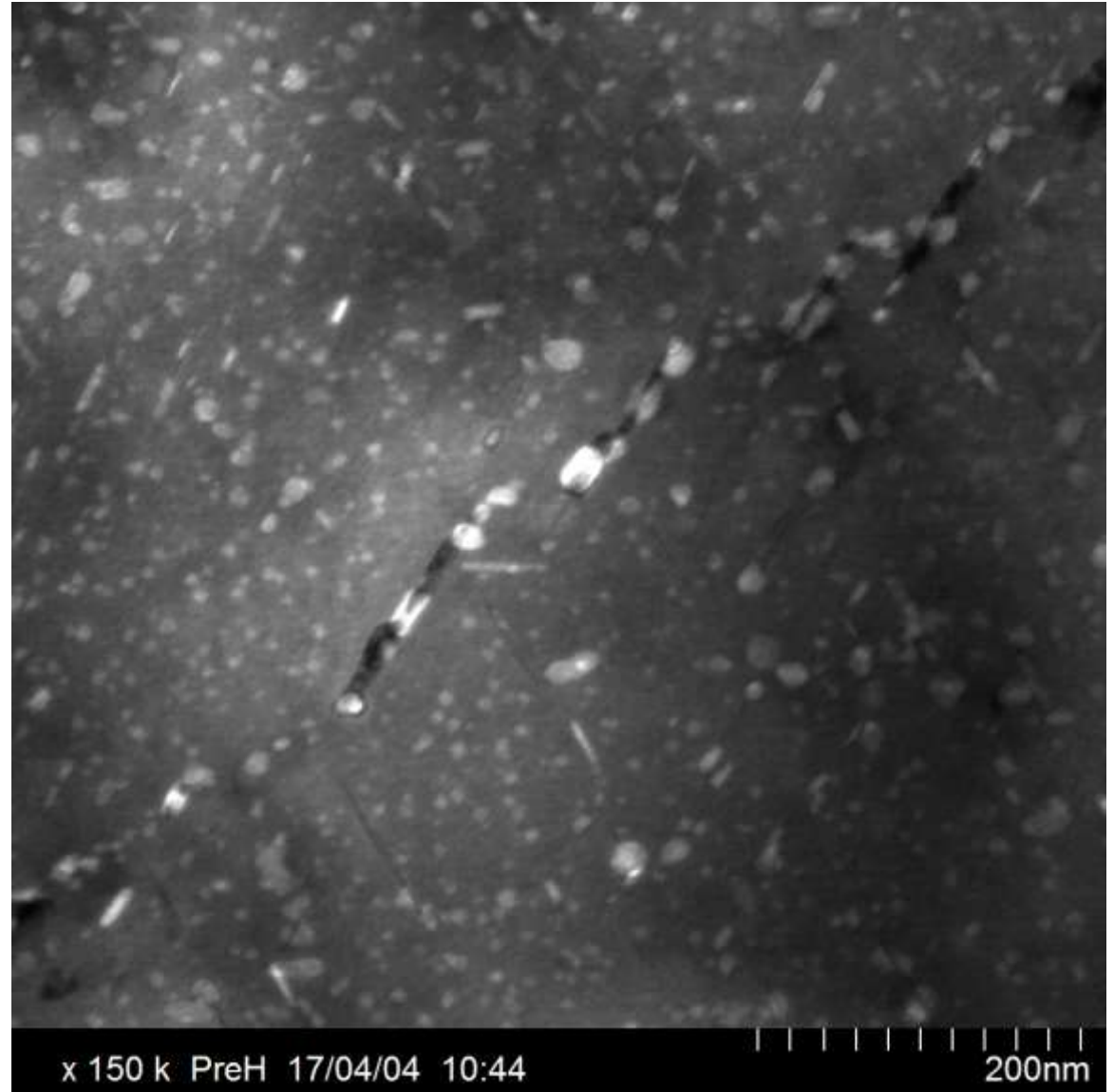
Zero-loss peak



Energy-filtered STEM - SU9000 (30 kV)

AA2099 – Al-Li-Cu

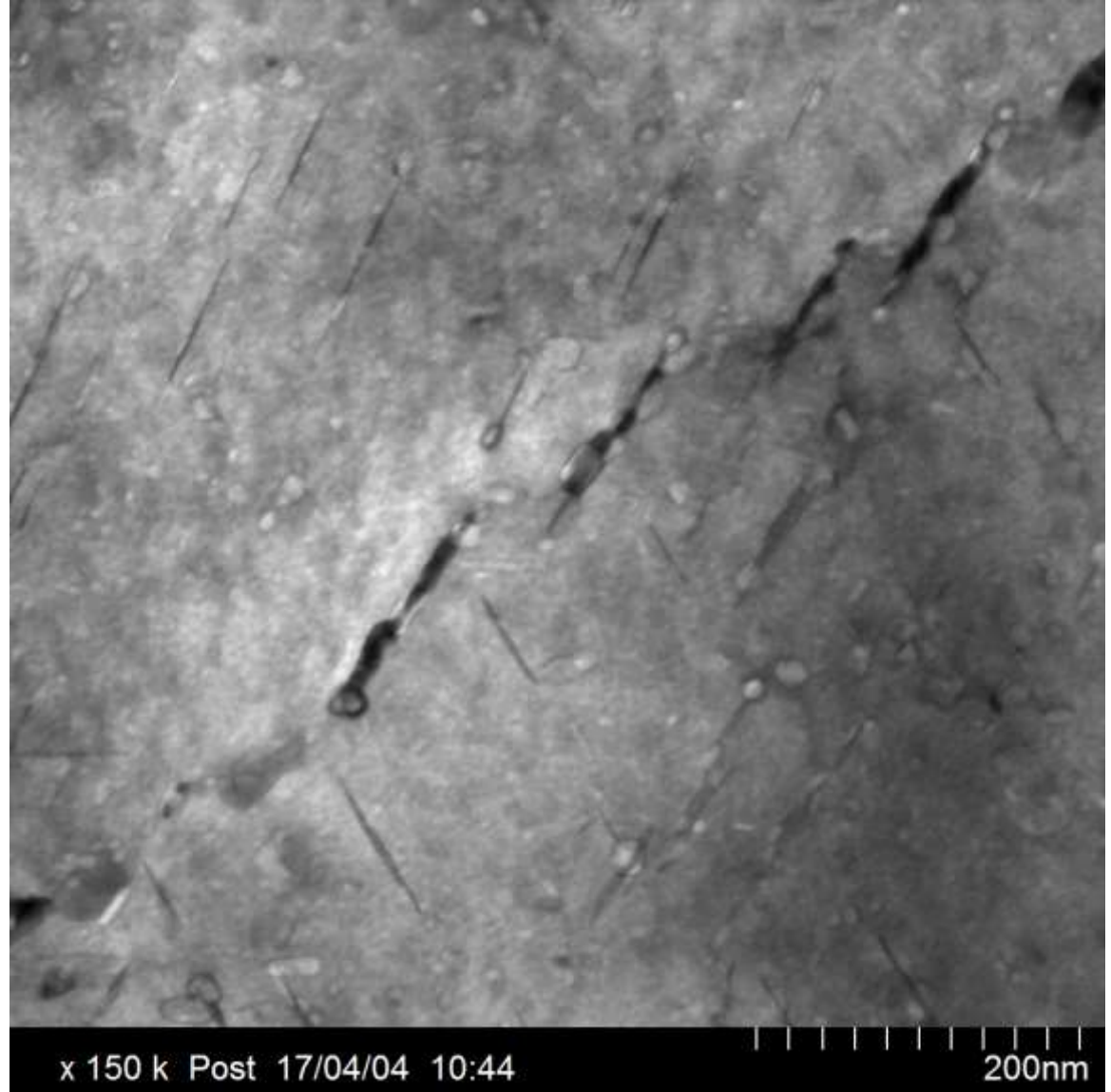
Plasmon 1

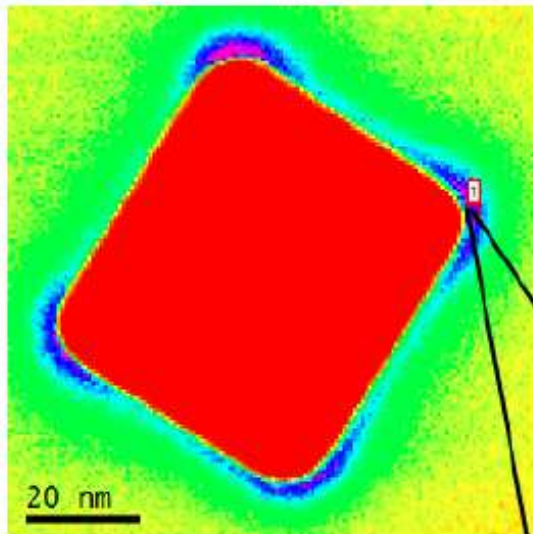


Energy-filtered STEM - SU9000 (30 kV)

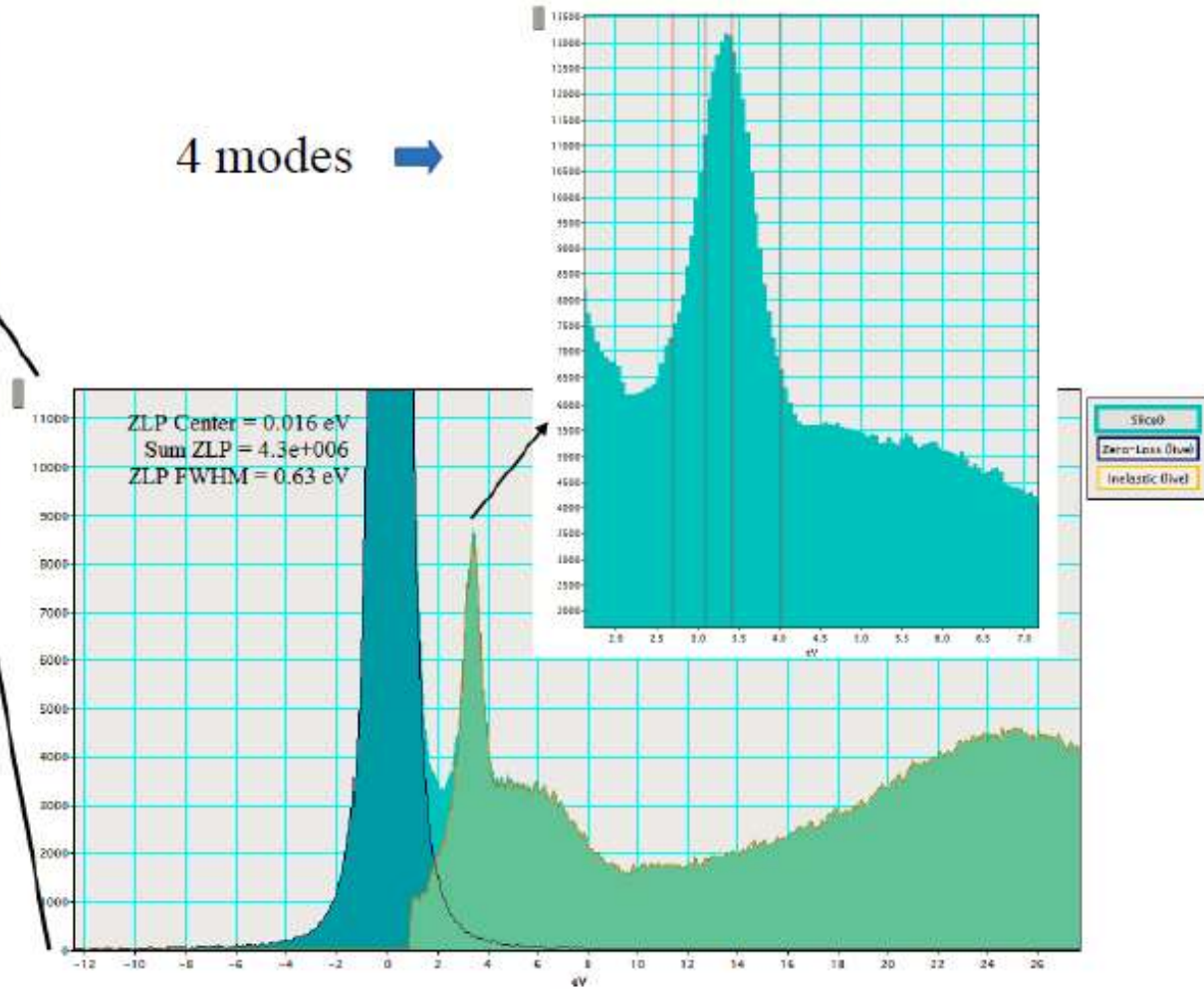
AA2099 – Al-Li-Cu

Plasmon 2

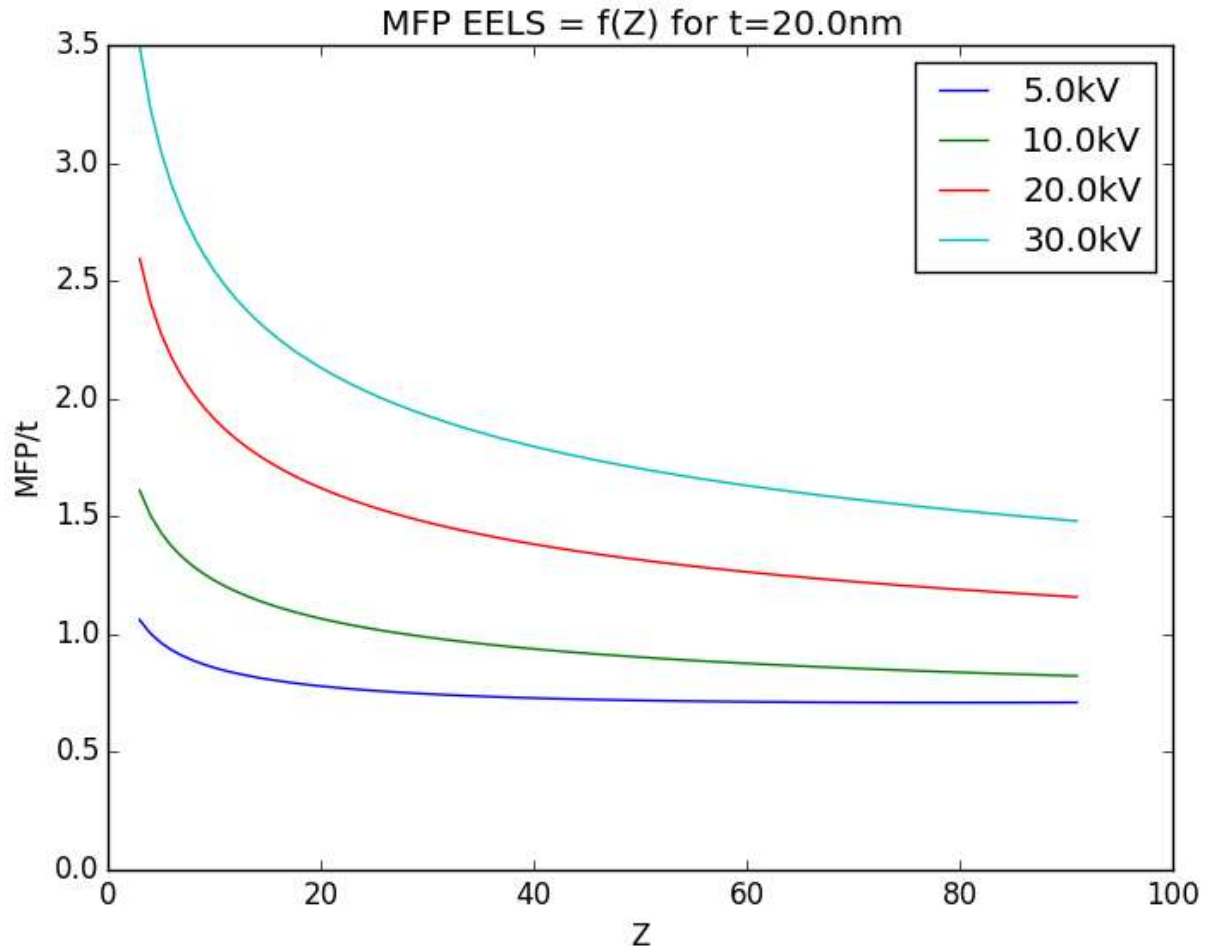




4 modes →

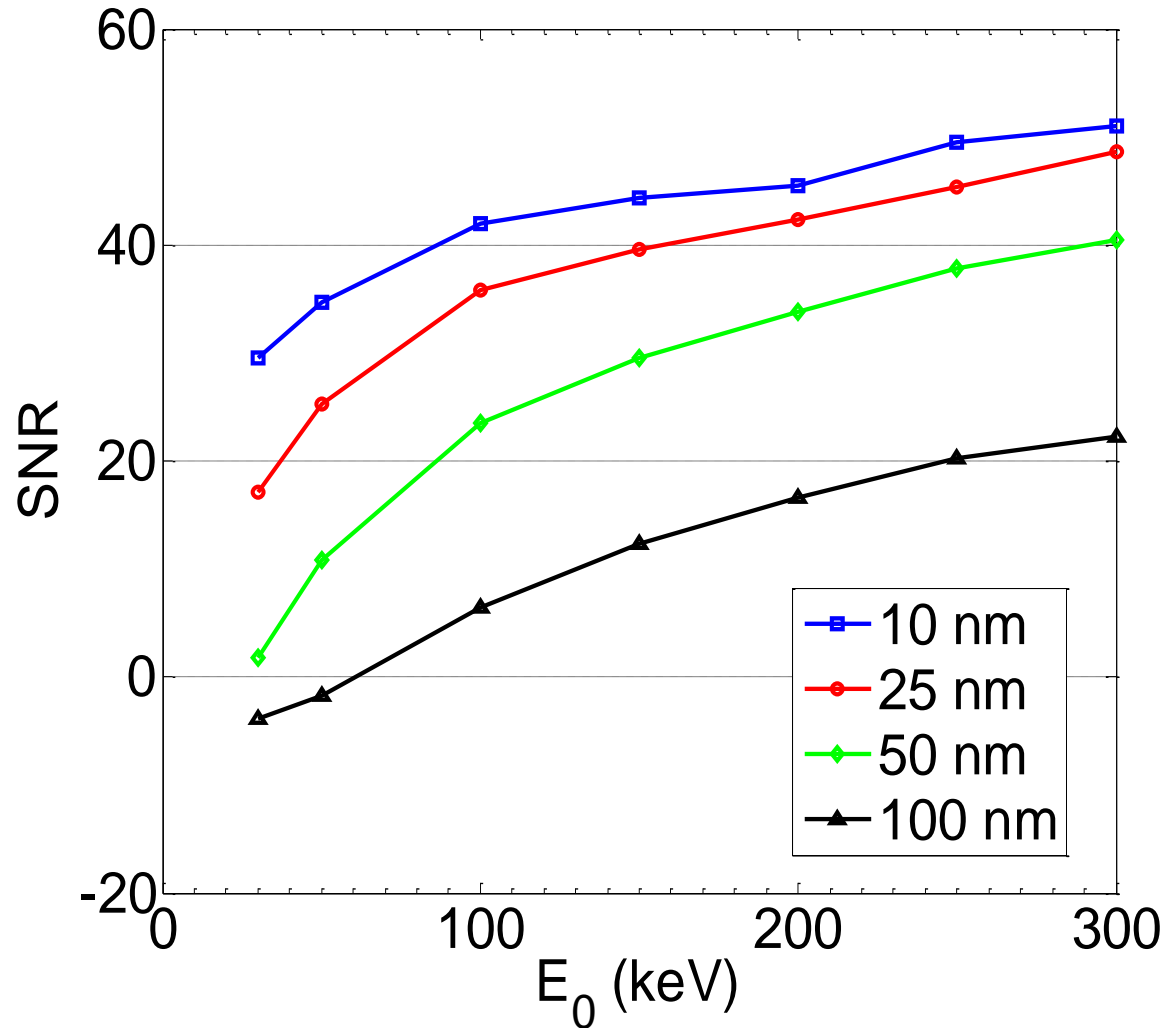


$\lambda/t, t = 20 \text{ nm}$



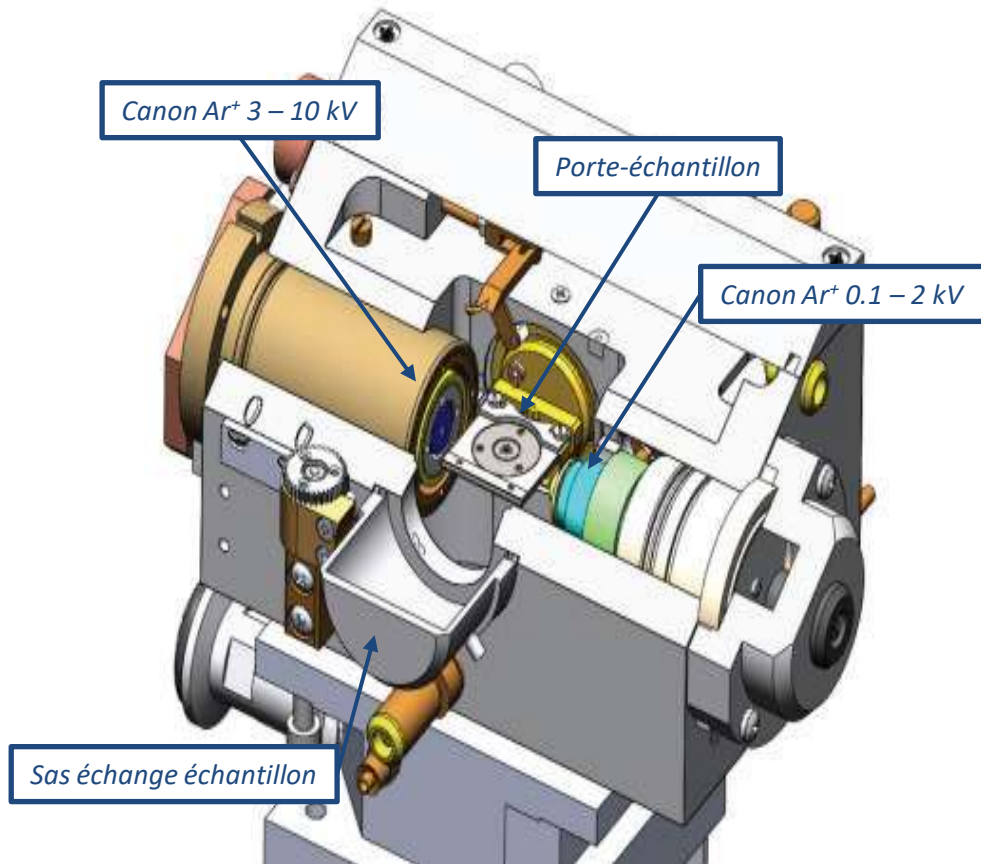
EELS Spectrum Simulation, GaN

$\Gamma=150\text{eV}, \Delta=50\text{eV}, \text{N K}$

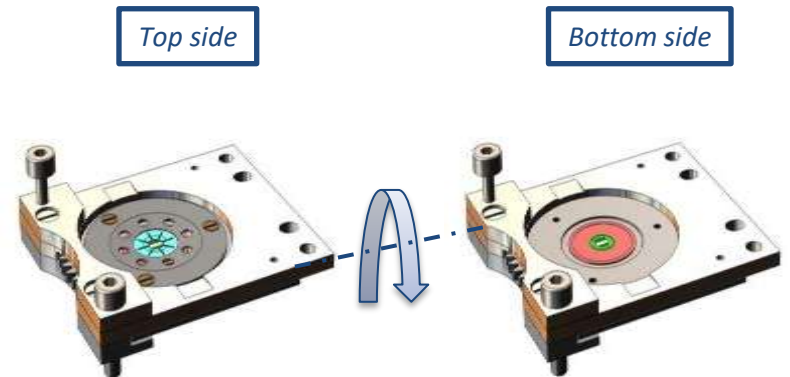


LE POLISSAGE IONIQUE POUR LA PRODUCTION D'ÉCHANTILLONS MINCES

Polissage ionique pour échantillons minces: UniMill de Technoorg-Linda (<http://www.technoorg.hu>)

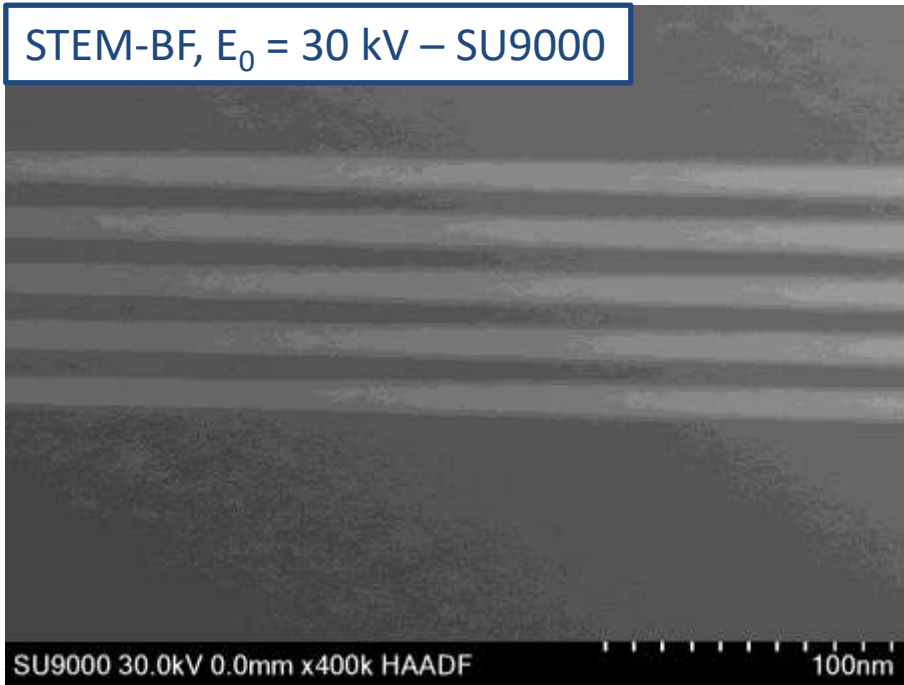


- Deux canons à Ar⁺, haute énergie (3 – 10 kV) et basse énergie (0.1 – 2 kV)
- Porte-échantillon réversible (deux faces)
- Très haute qualité de finition due aux basses énergies

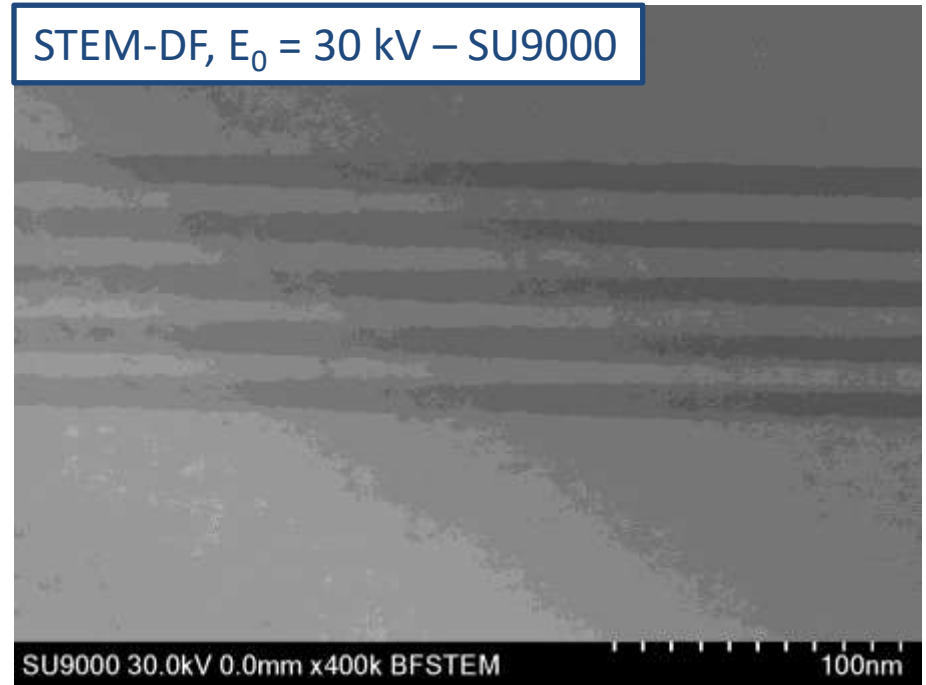


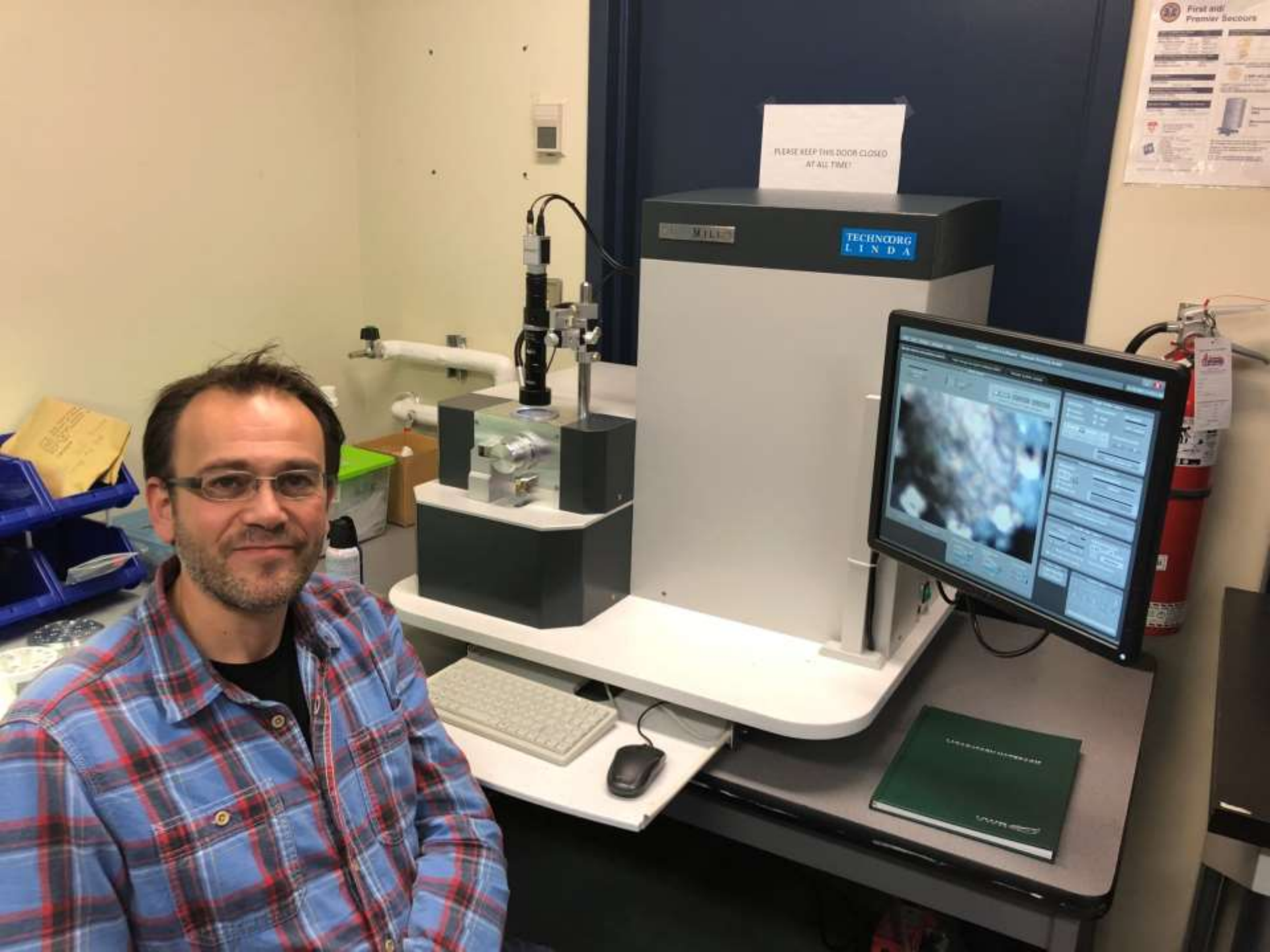
Polissage ionique pour échantillons minces: Couches de GaAs dans du silicium

STEM-BF, $E_0 = 30$ kV – SU9000



STEM-DF, $E_0 = 30$ kV – SU9000



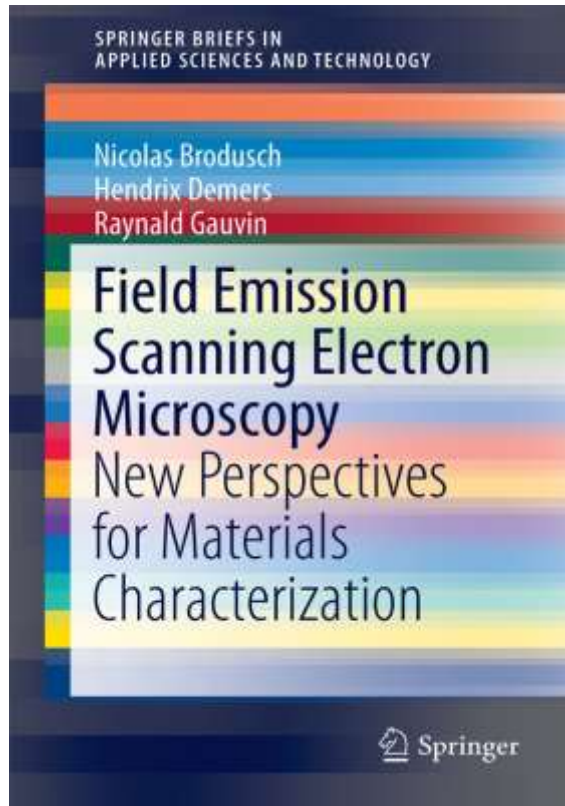


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