

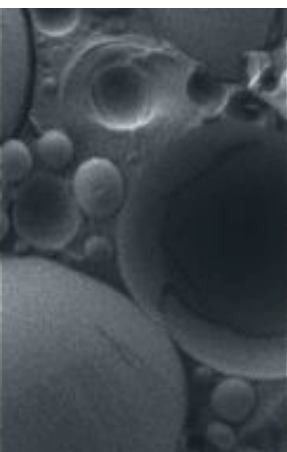
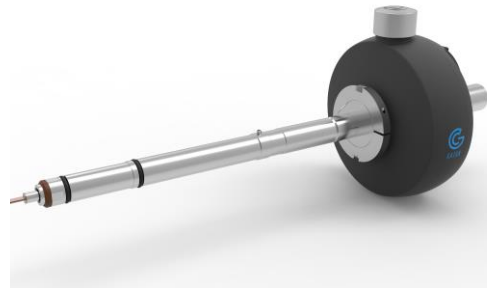
# Préparation ionique pour observations MEB & analyses EBSD

[Daniel.Monville@ametek.com](mailto:Daniel.Monville@ametek.com)

[Vincent.Richard@ametek.com](mailto:Vincent.Richard@ametek.com)

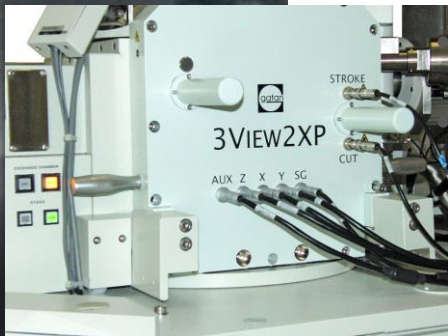
Journées pédagogiques GN-MEBA 3 & 4 décembre 2020





## **GATAN : accessoires pour MEB & MET** **... from Specimen preparation to EELS analysis**

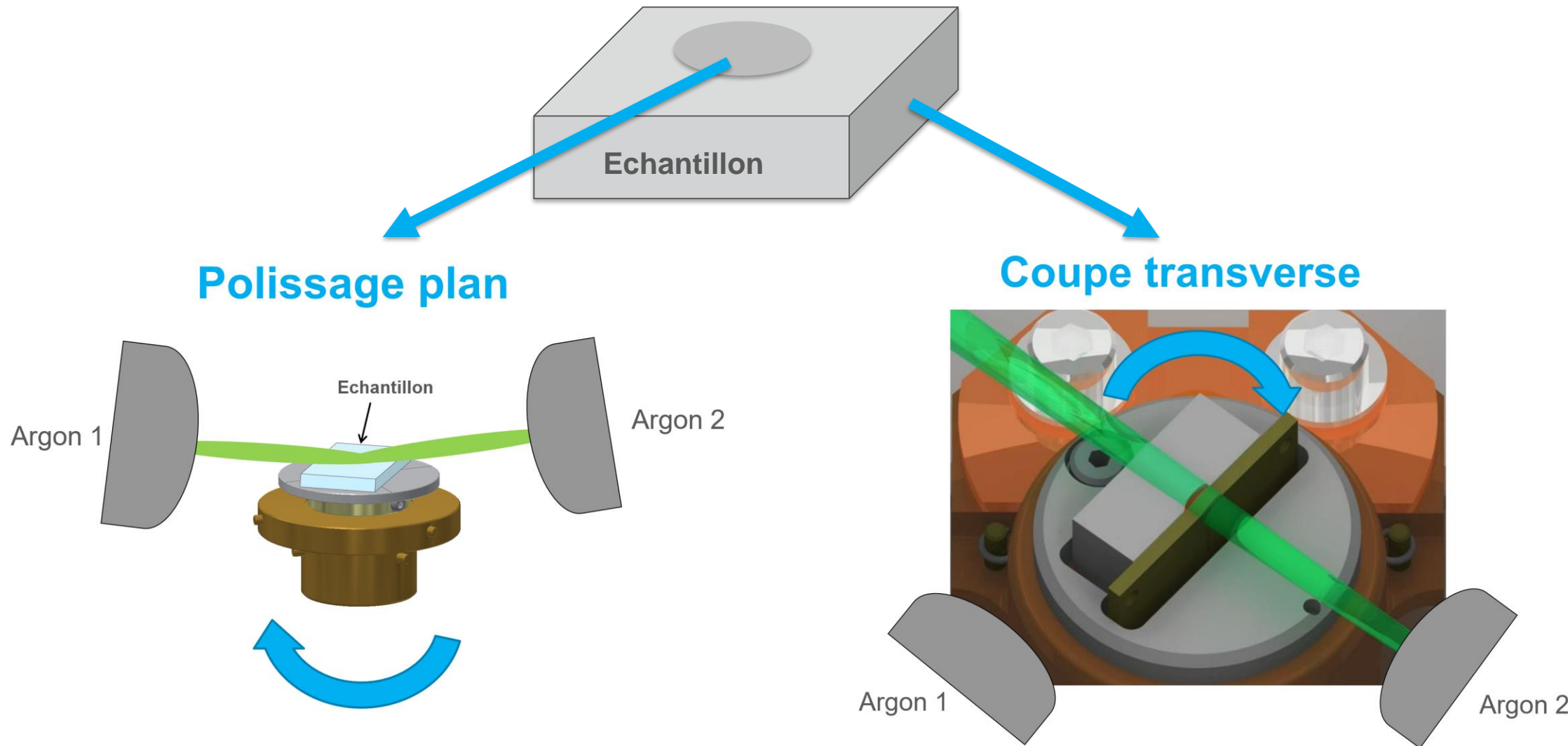
- Specimen Prep : PIPS, PECS, Ilion 2, Solarus, ...
- Holders : Analytical, CryoTransfer, Cooling/heating, Tensile, Tomography, multi-axes, ...
- Imaging : Direct and undirect detection : Rio, Oneview, K2, K3
- Analytical : EELS spectrometers and filters, CL Systems
- SEM accessories : 3View, CL Systems (Monarc), Heating/cooling stages, ...



# Préparation ionique pour observations MEB & analyses EBSD

# Techniques préparations ioniques

(Par faisceaux d'ions Argon non focalisés)



# Ilion II & PECS II spécifications

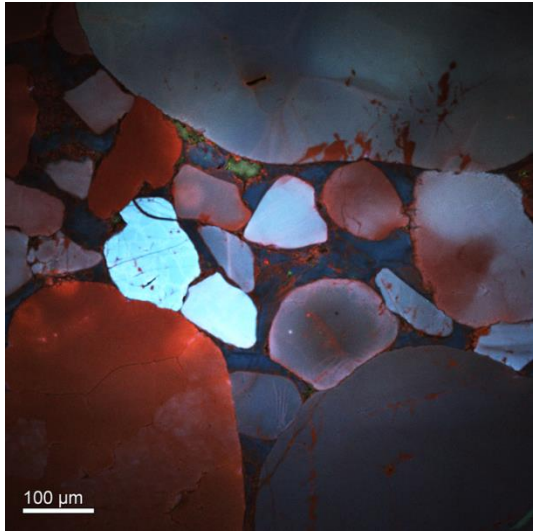
- Polissage plan et coupe transverse
- 2 faisceaux d'ions Argon
- Tension d'accélération : 100 Volts - 8000 Volts
- Recettes
- Polissage sectoriel
- Gestion automatique des canons
- Microscope et caméra avec Digital Micrograph
- Platine froide (Azote liquide)
- Sas d'introduction



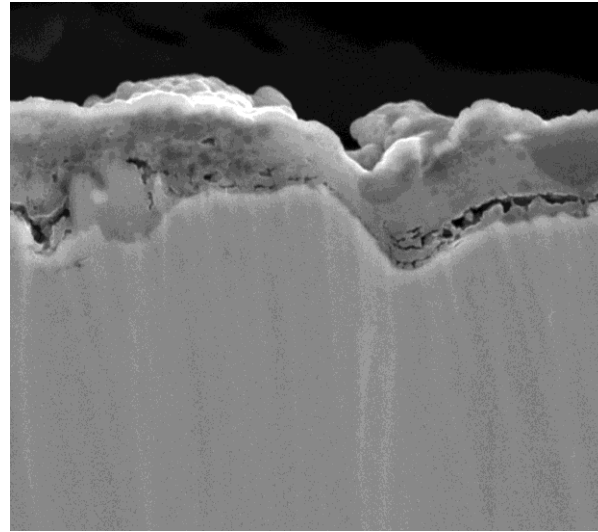
- Métallisation In-Situ
- Echantillons plus grands
- Transfert sous vide ou atmosphère

# Coupes transverses par faisceaux d'ions

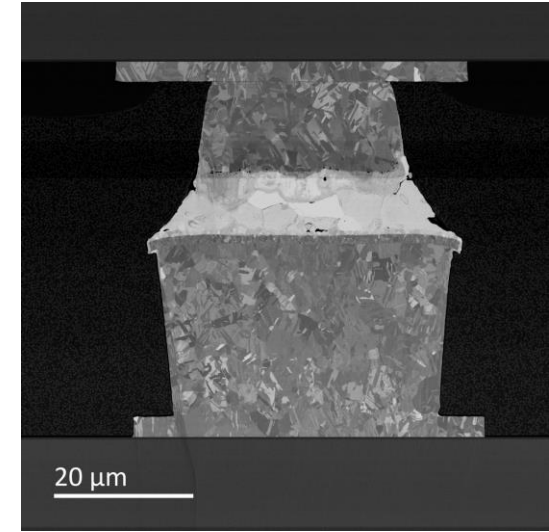
Shale



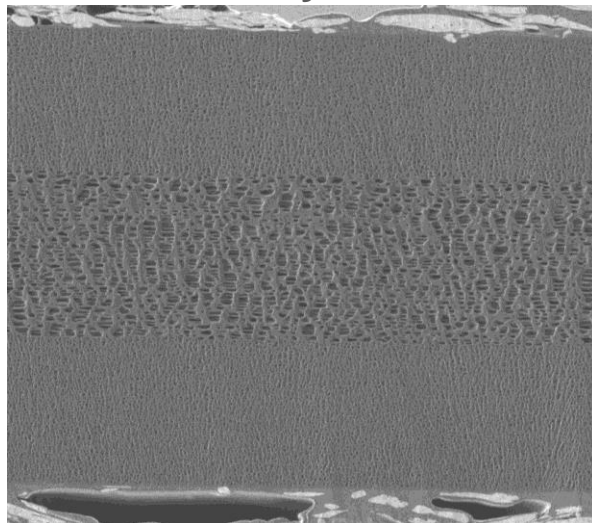
Ceramic coating on metal



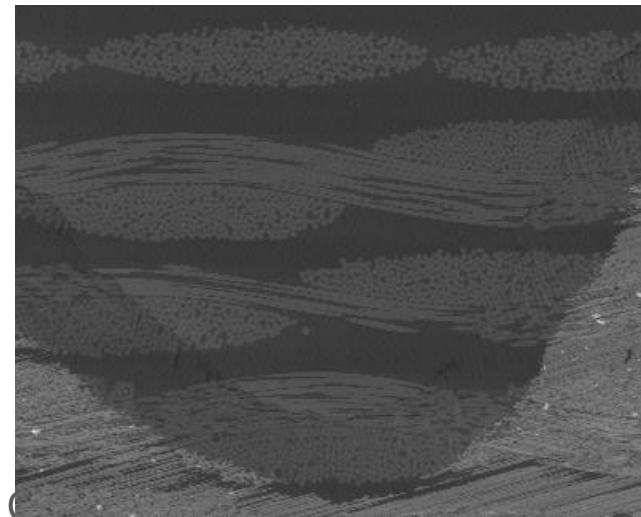
Semiconductor



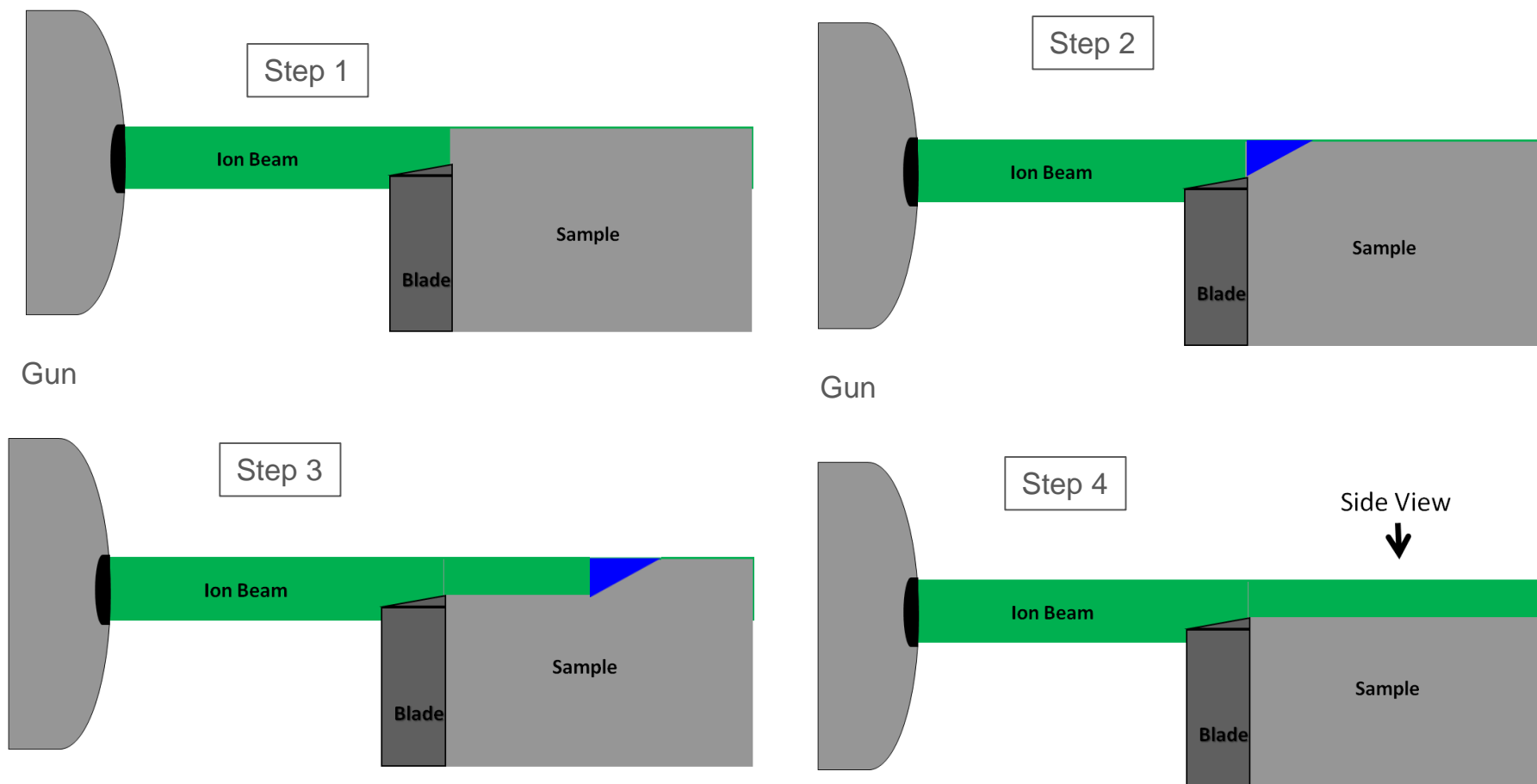
Polymer



Composite

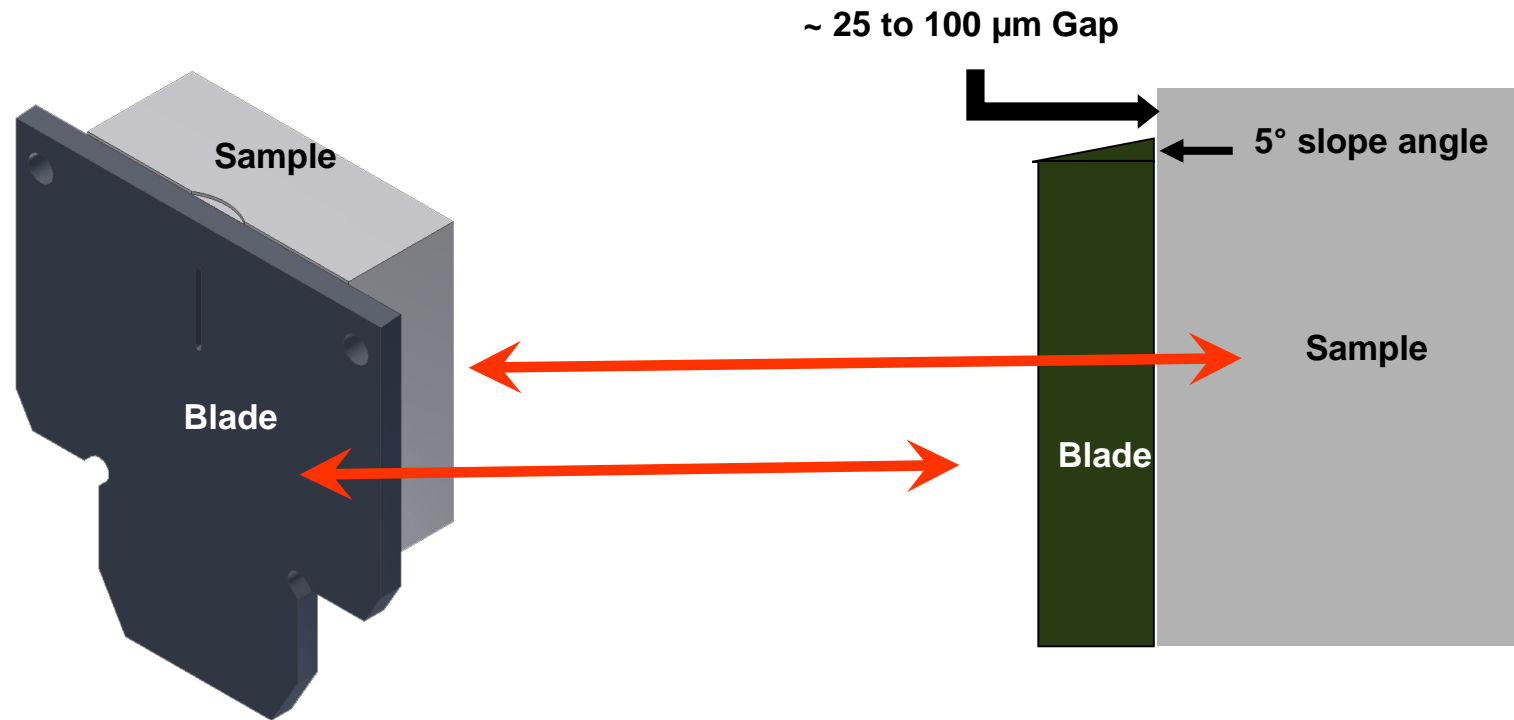


# Coupes transverses par faisceaux d'ions (principe)

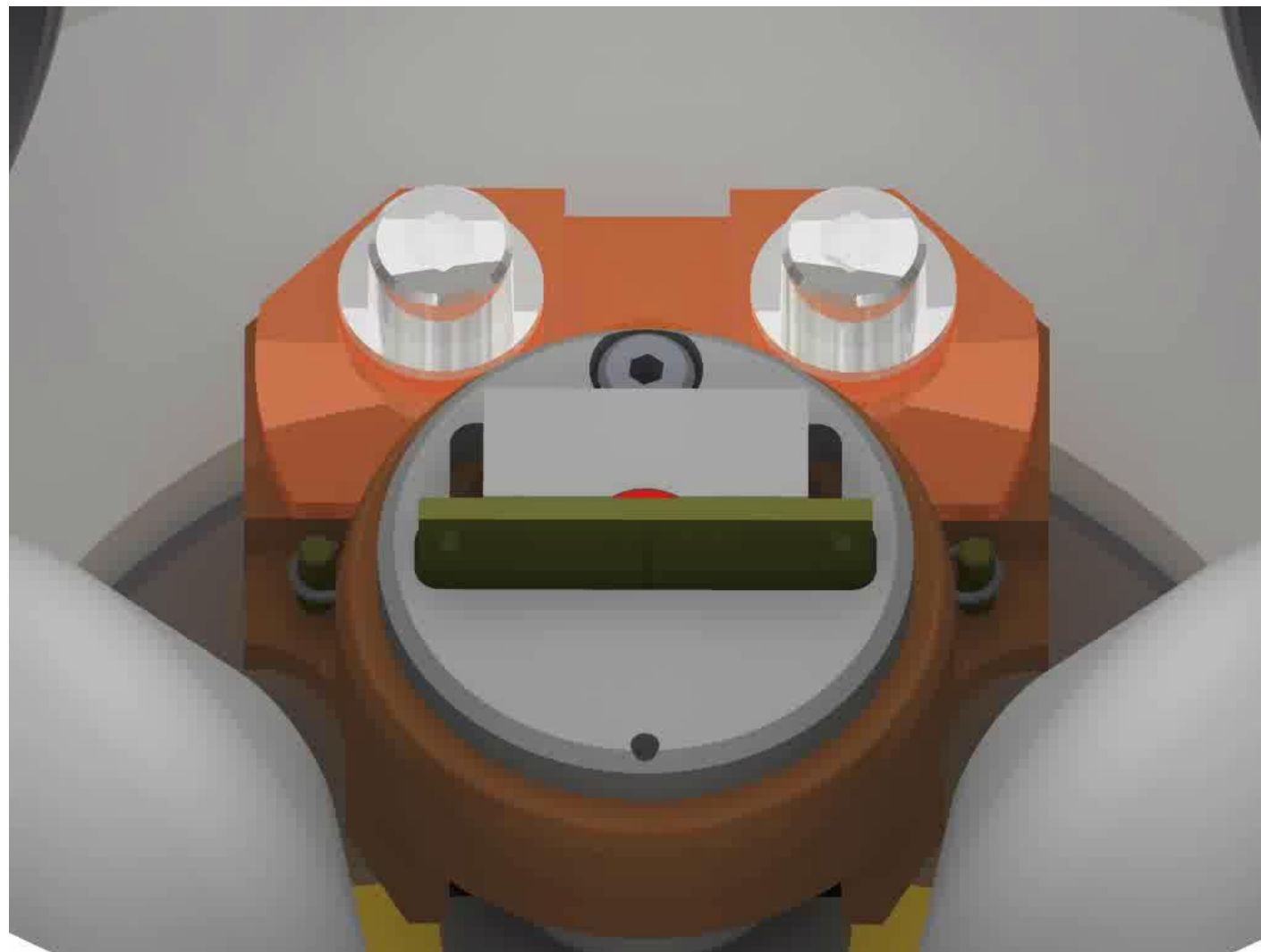


# Coupes transverses par faisceaux d'ions (montage de l'échantillon)

Un masque est fixé à l'échantillon de 25 à 100  $\mu\text{m}$  sous la surface supérieure (cet écart va déterminer l'épaisseur enlevée)

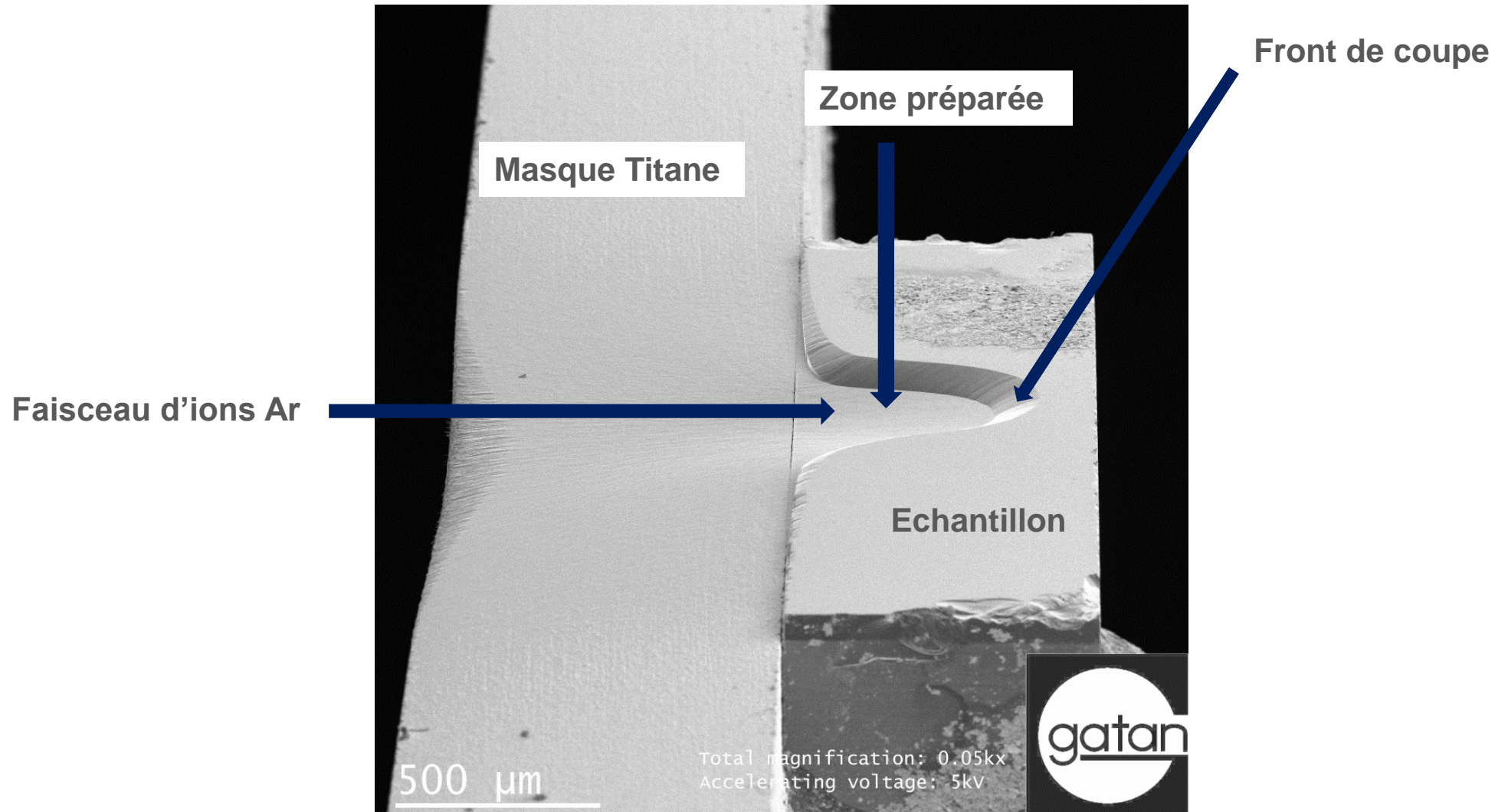


# Coupes transverses par faisceaux d'ions (polissage sectoriel)



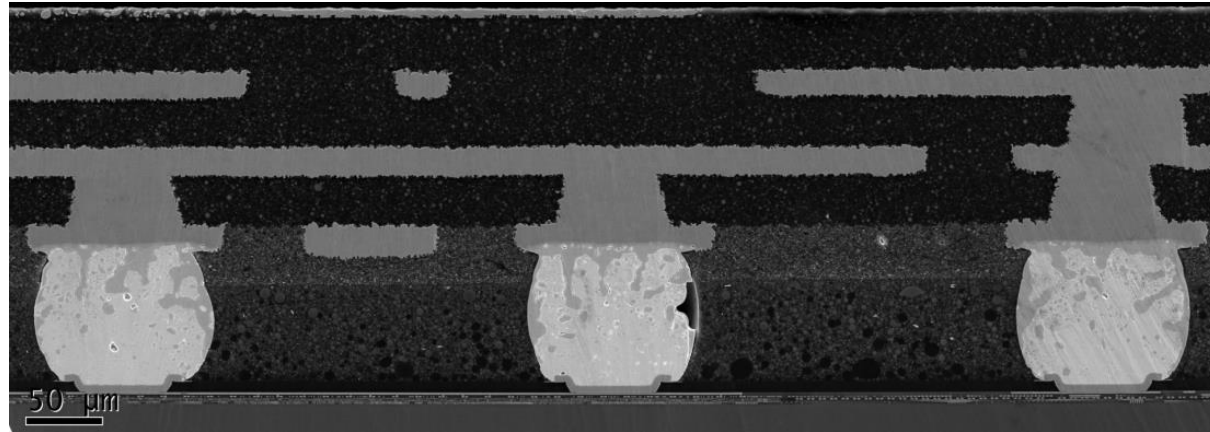
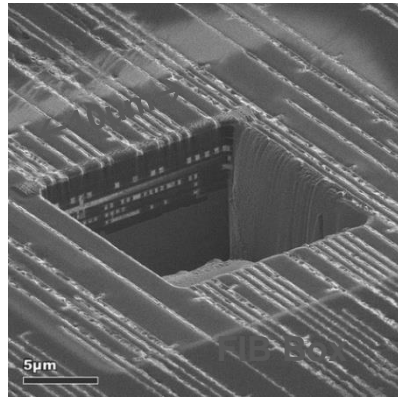
Confidential

# Coupes transverses par faisceaux d'ions (Ex Silicium)

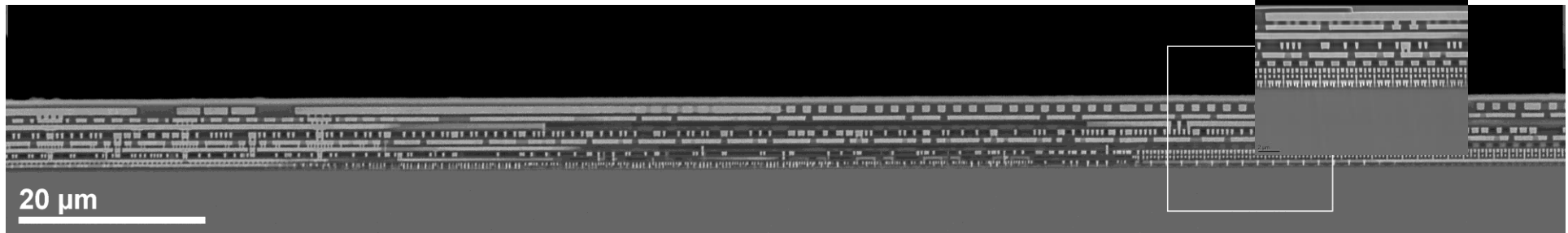


# X-Sections

Wide horizontal view of sample is ~200X that of FIB



C4 (Flip Chip) Ball Grid Array

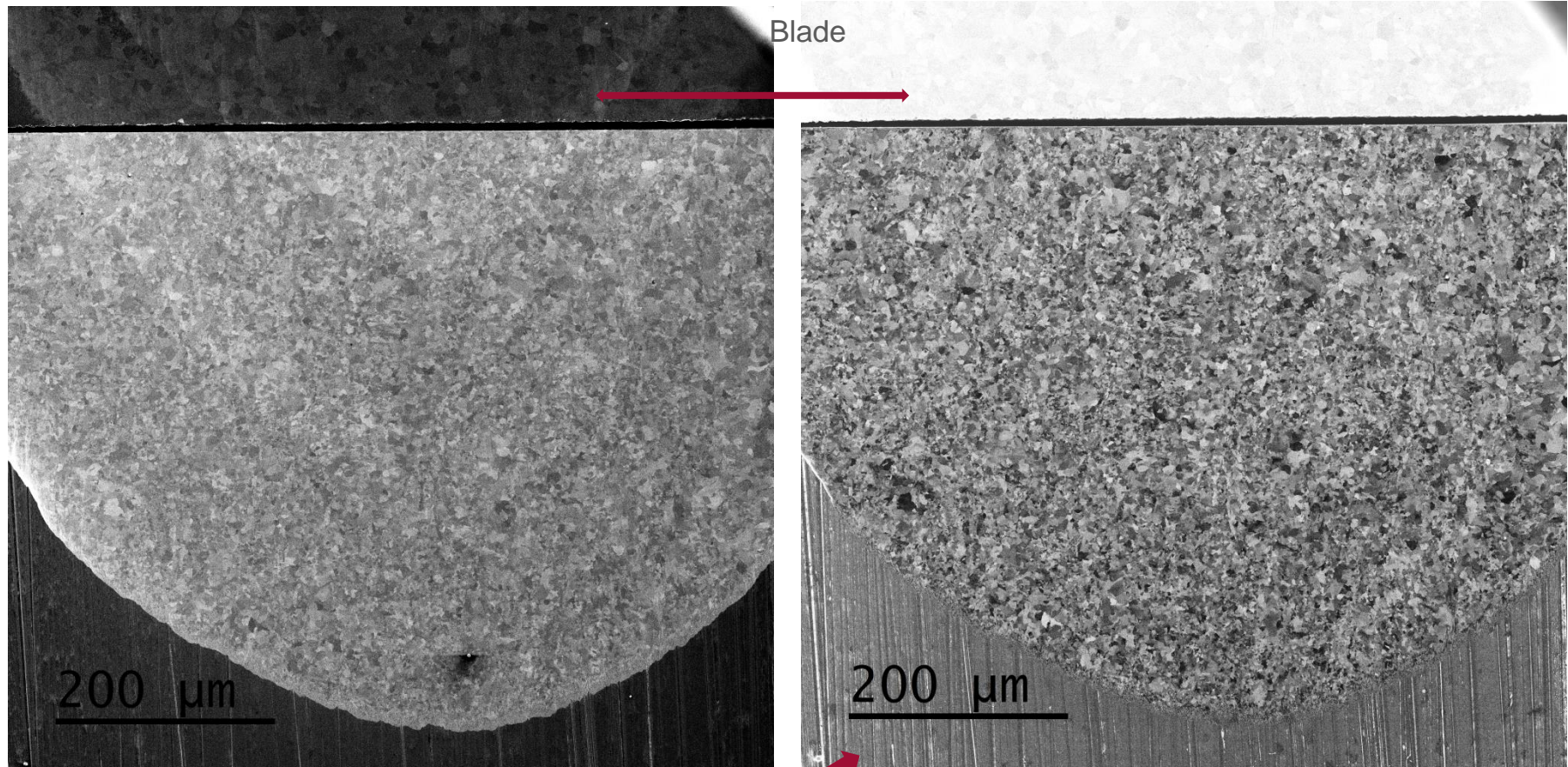


Large area cross section on CMOS facilitates fault/defect surveys

# Coupe transverse sur acier

SE Image

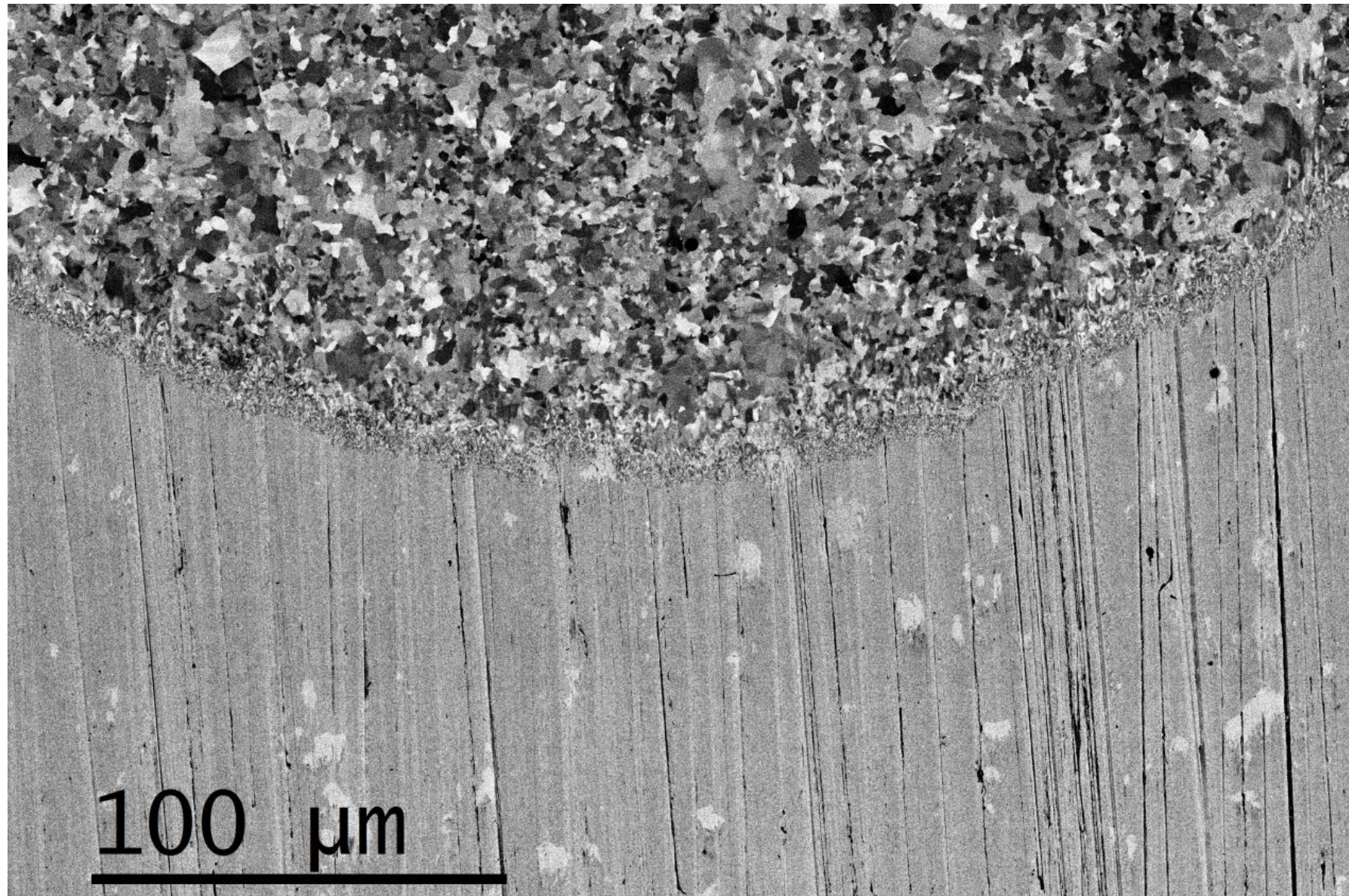
BSE Image



Mechanical Polish

Confidential

# Interface entre coupe ionique et polissage mécanique



# Acier martensitique (BSE)



2  $\mu\text{m}$

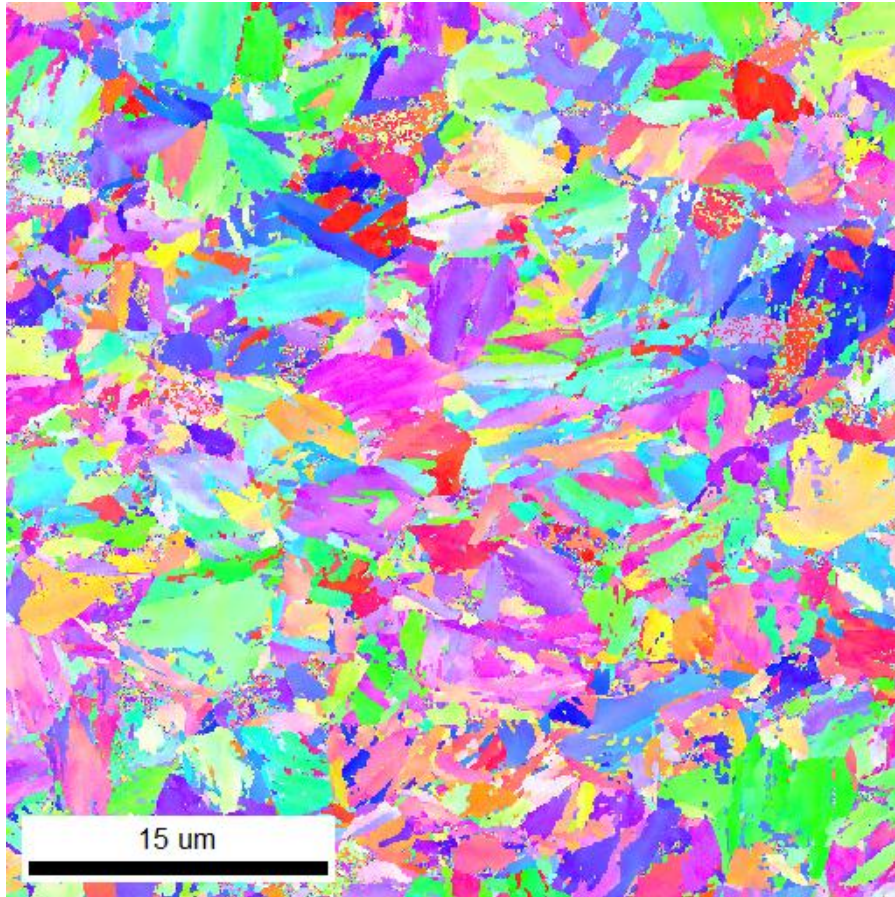
EHT = 5.00 kV

WD = 6.2 mm

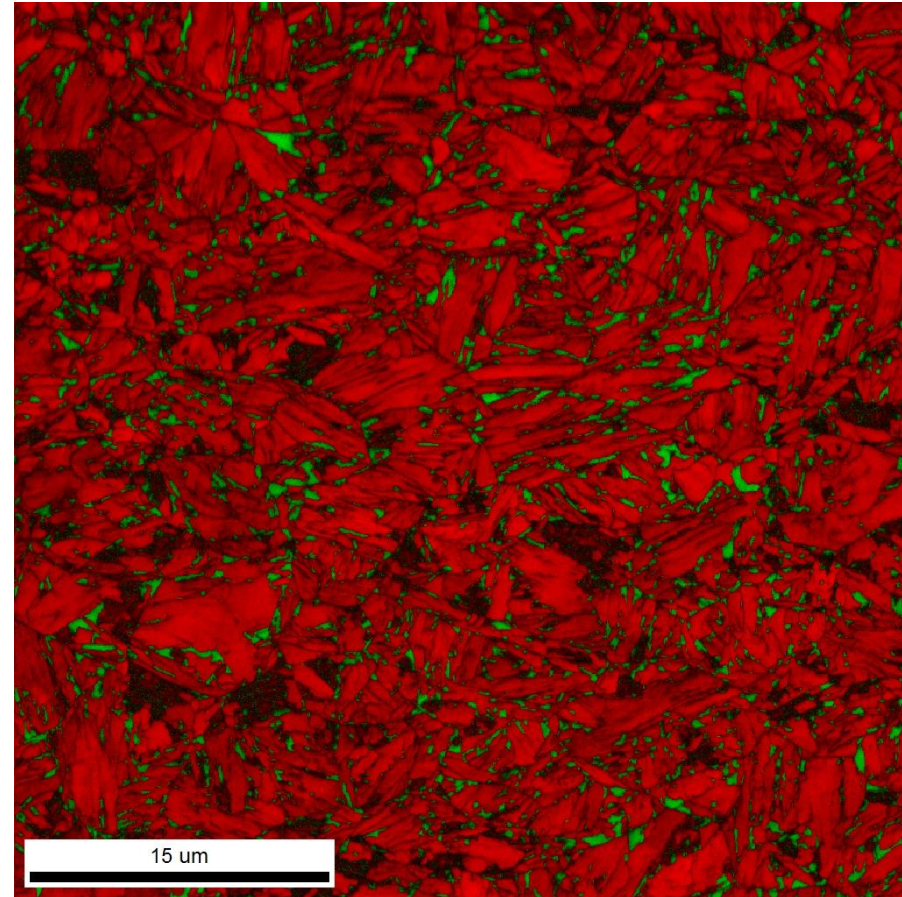
NTS BSD 4.24e-004 Pa



# EBSD on Martensite



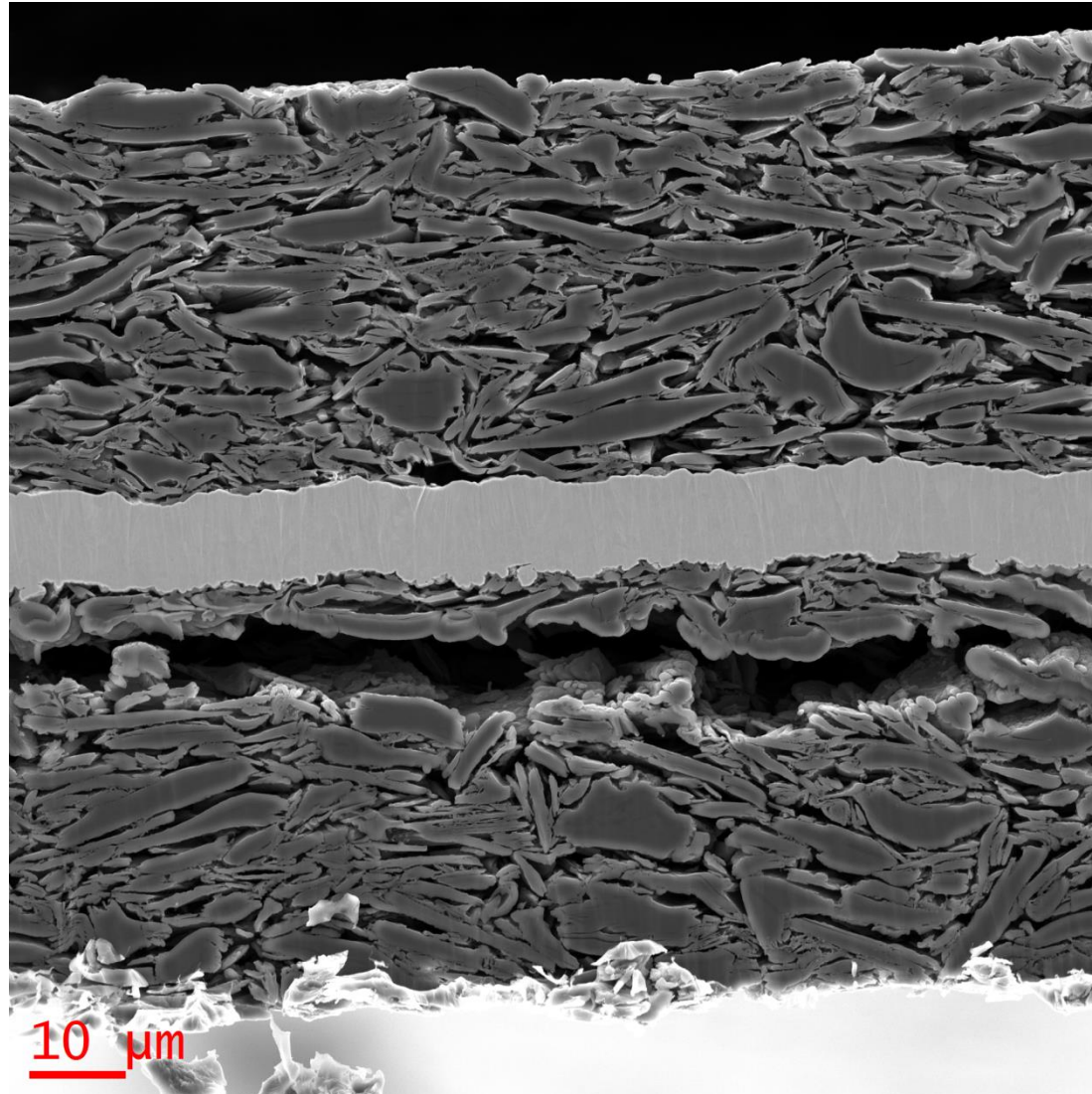
IPF



Phases +IQ

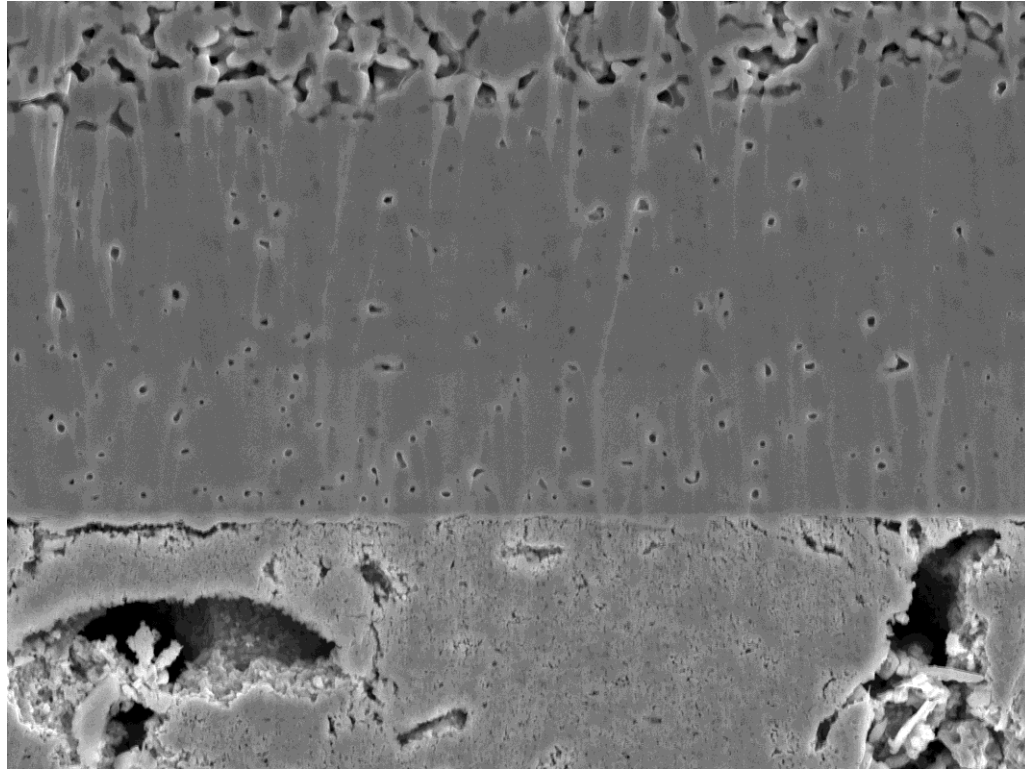
Rouge CC  
Vert CFC

# Li Battery Prep

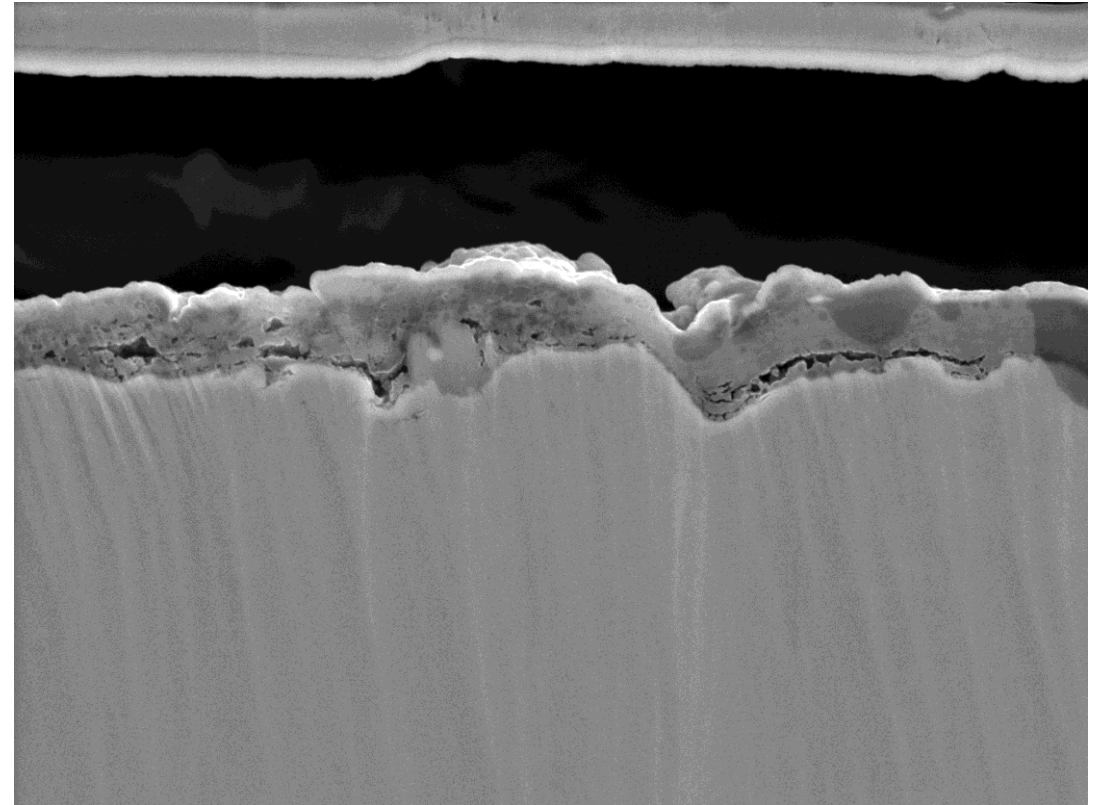


Confidential

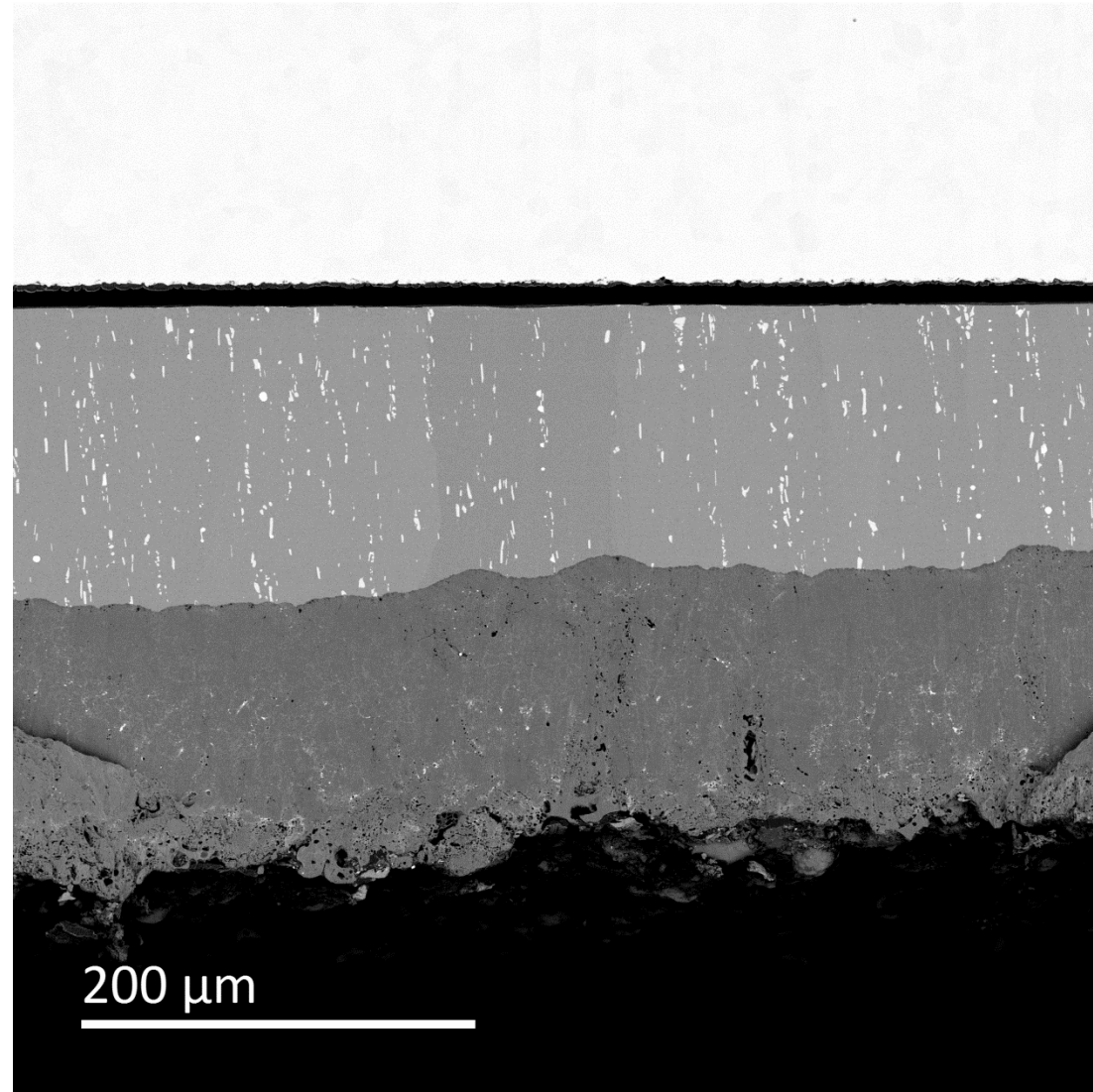
# Ceramic Coating



## Ceramic Coating on Metal



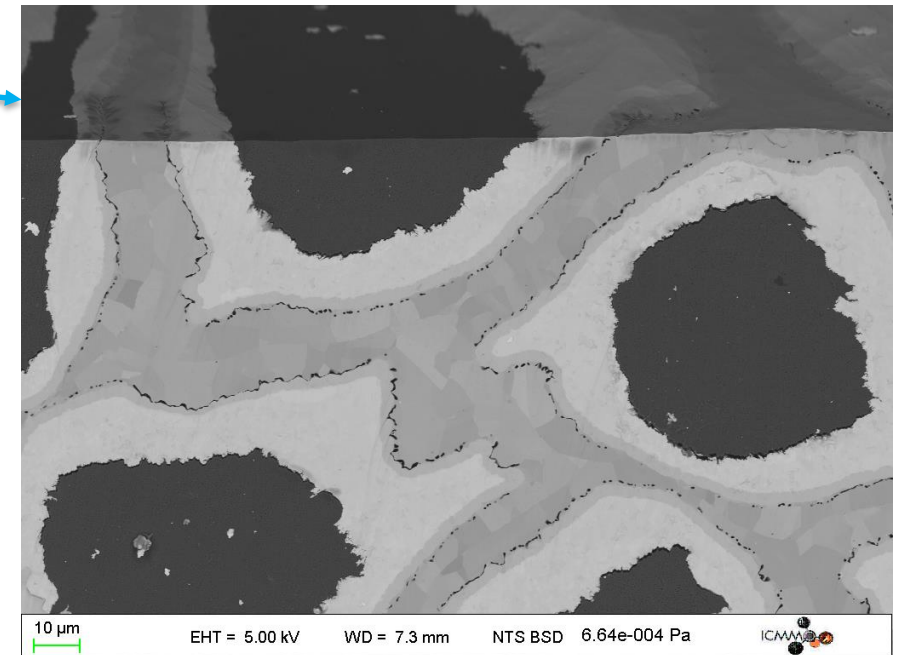
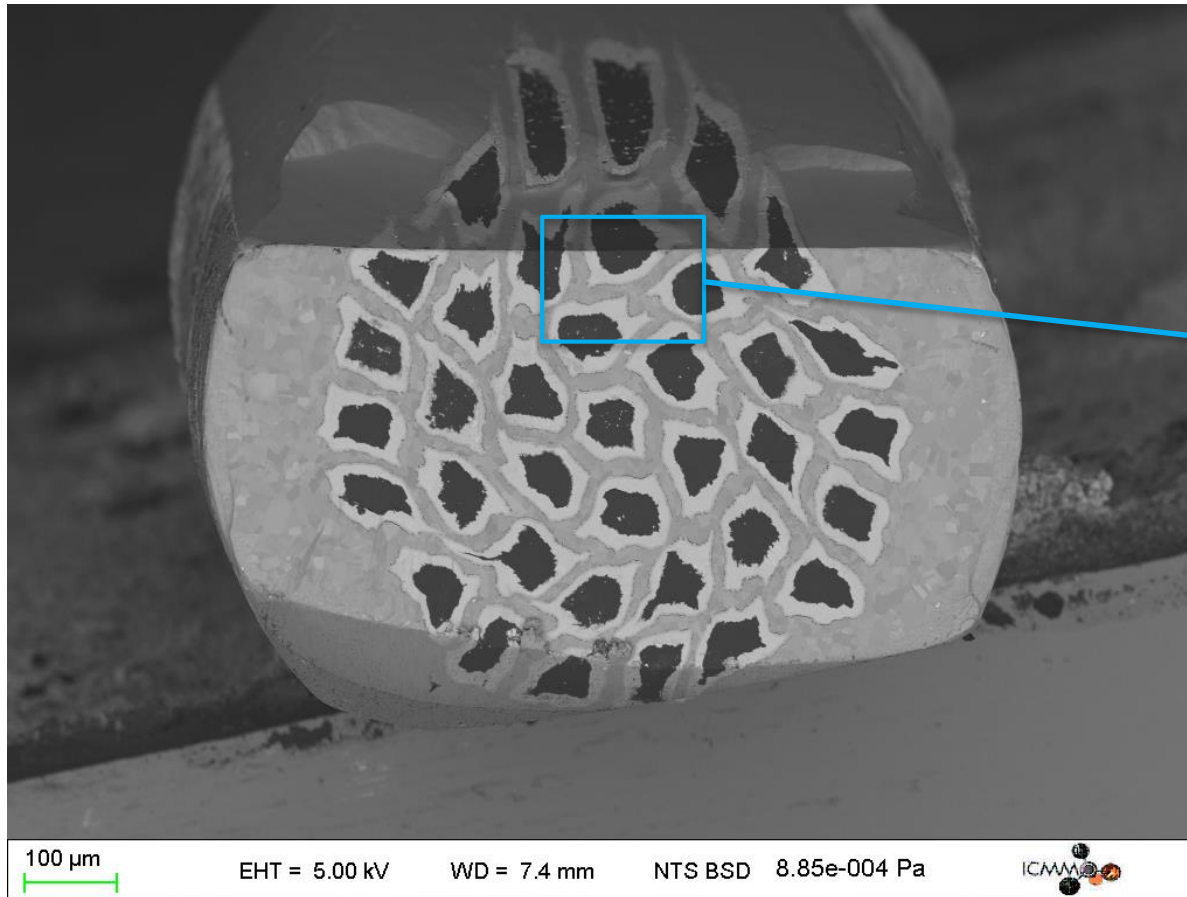
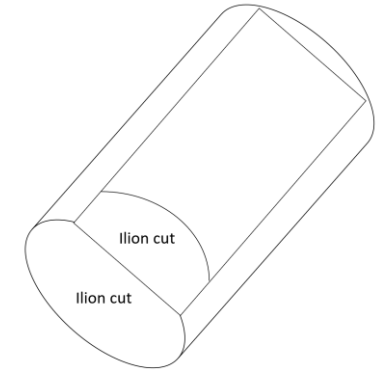
# Alumina on Aluminum



BSE Image

# CABLE SUPRA CONDUCTEUR (CERN) ILION 2

## Préparation pour 3D EBSD FIB

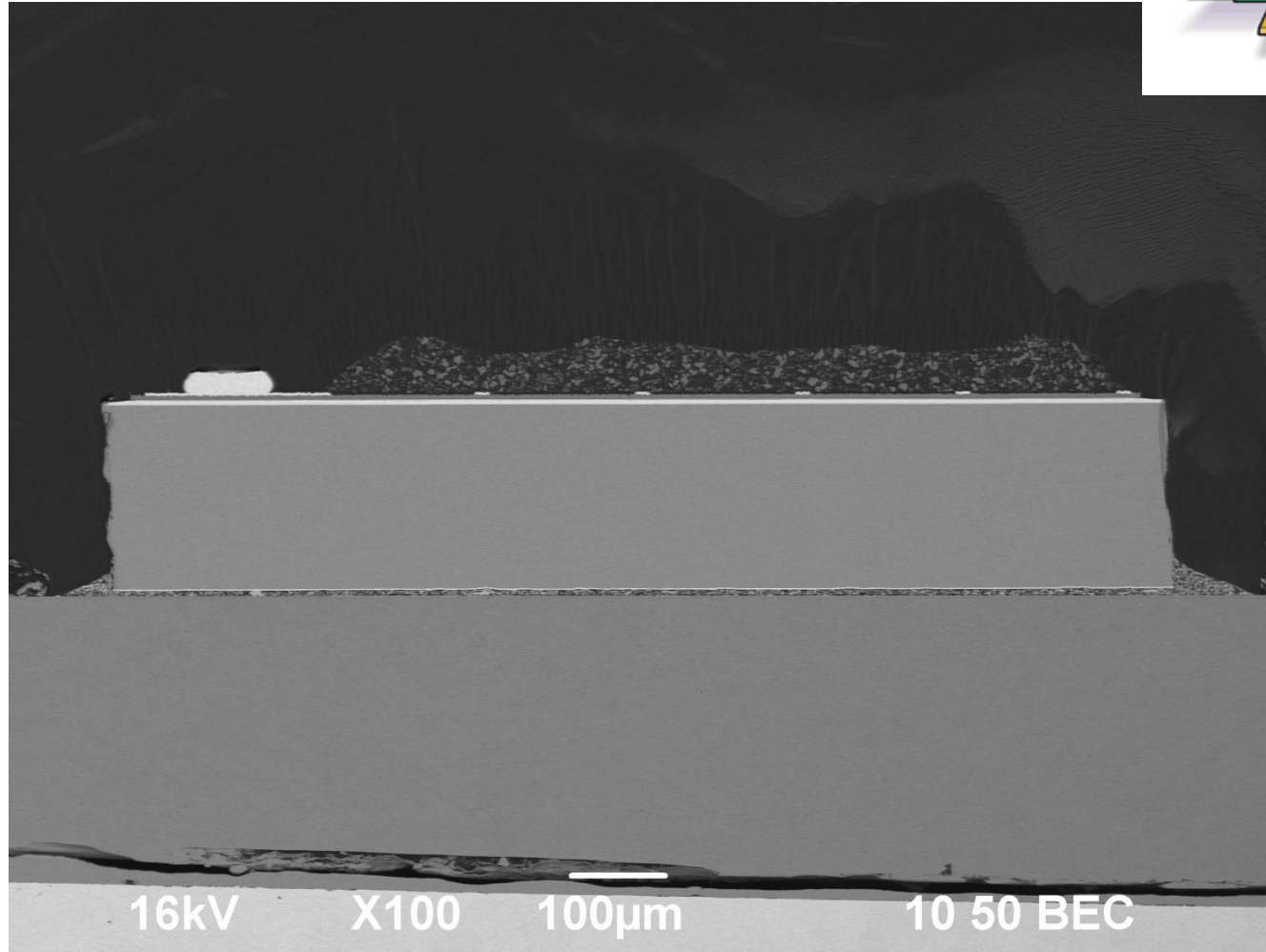
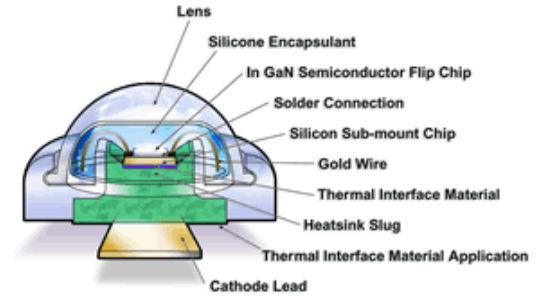


A vertical strip on the left side of the slide shows a microscopic view of numerous bubbles of varying sizes, some with distinct shadows and highlights, suggesting a liquid or gaseous medium.

# Platine froide (N<sub>2</sub> liquide):

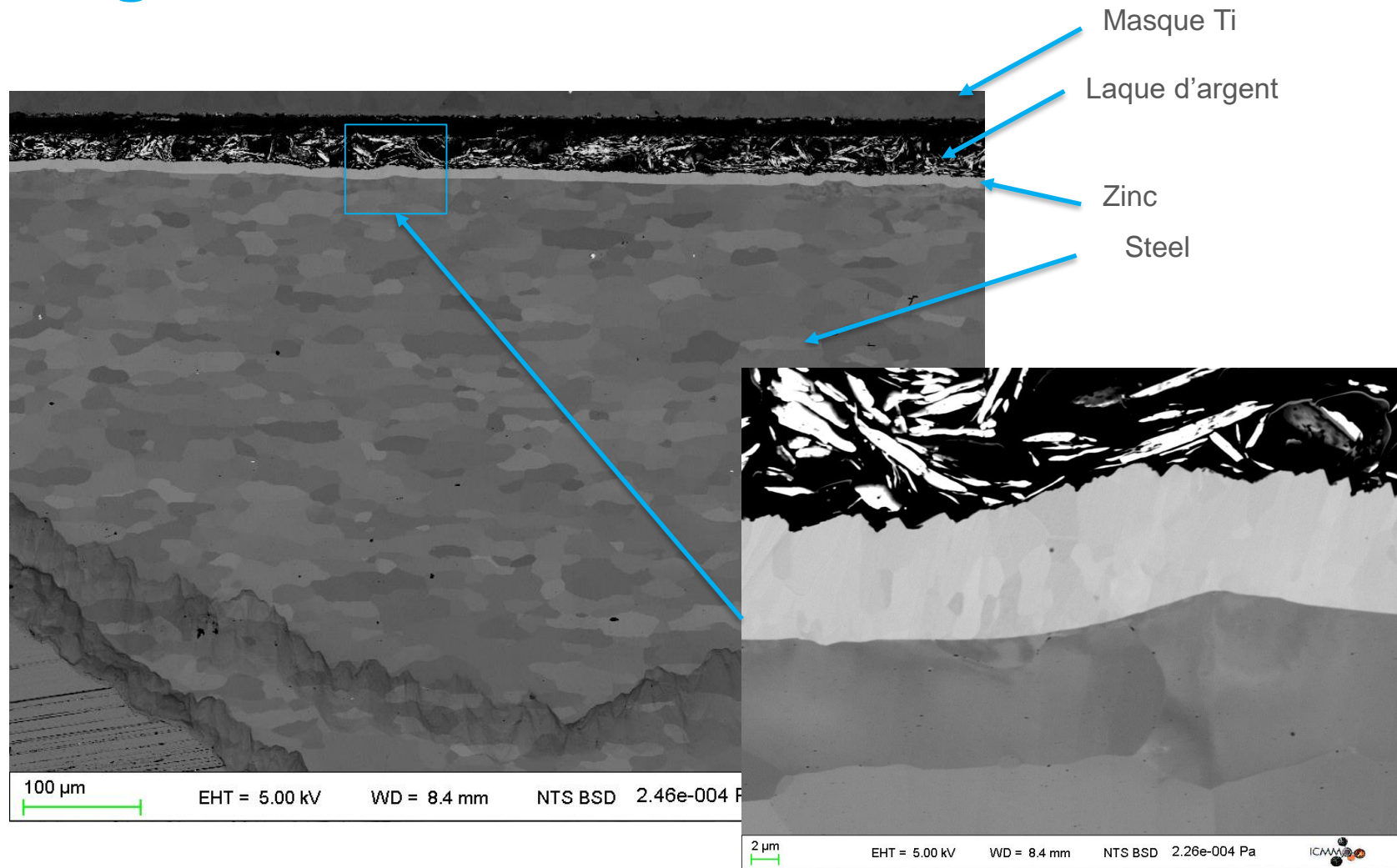
Polymers, CIGS, Paints, LED, zinc, ...

# Complete HB LED

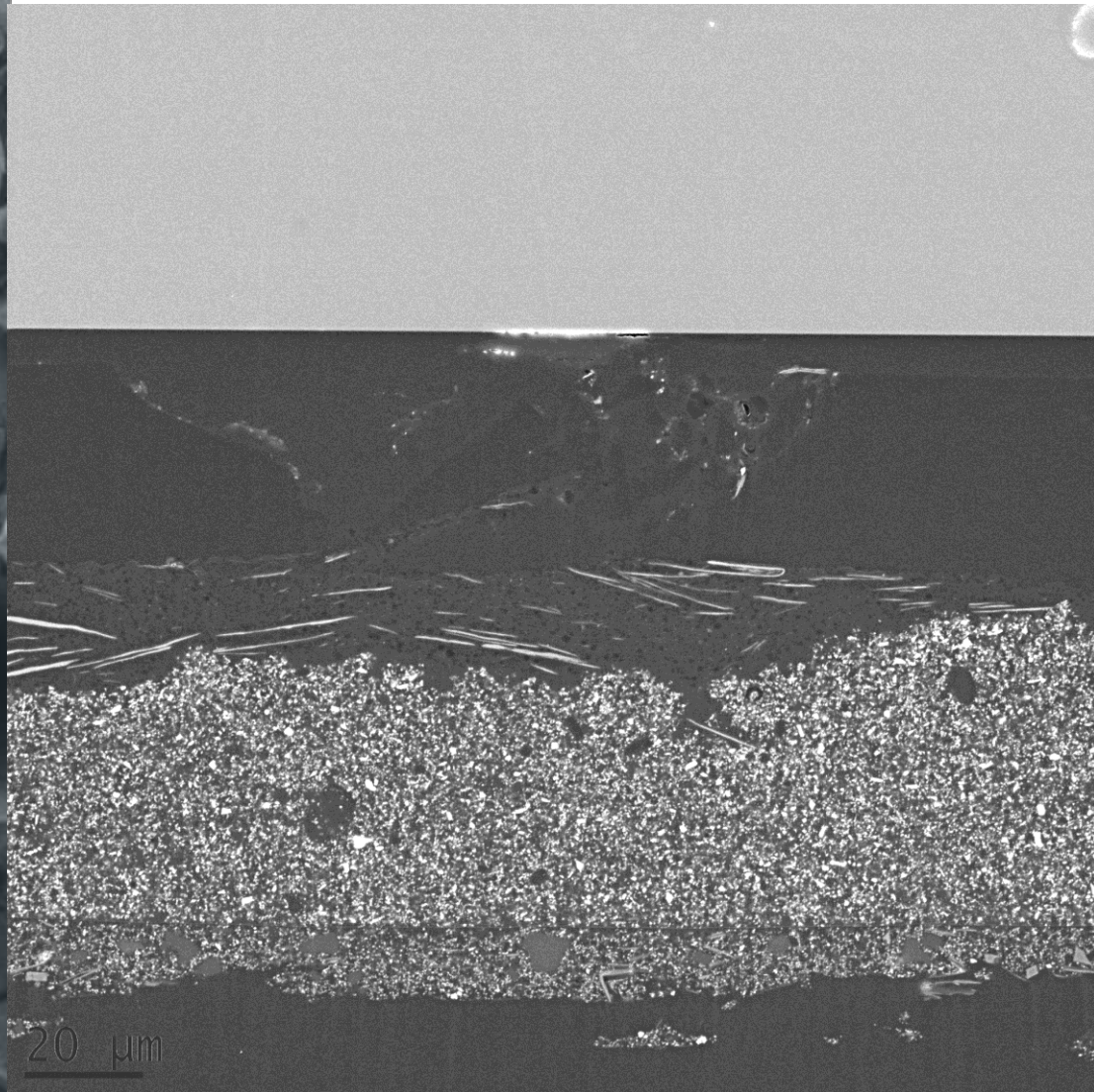


- 1mm Cut Width
- 4 hr cut time
- Materials from Plastic, Sapphire, Phosphor, Gold, Silicone Gel, Silicate
- Great for CL as well
- 20 interfaces of interest in this one sample

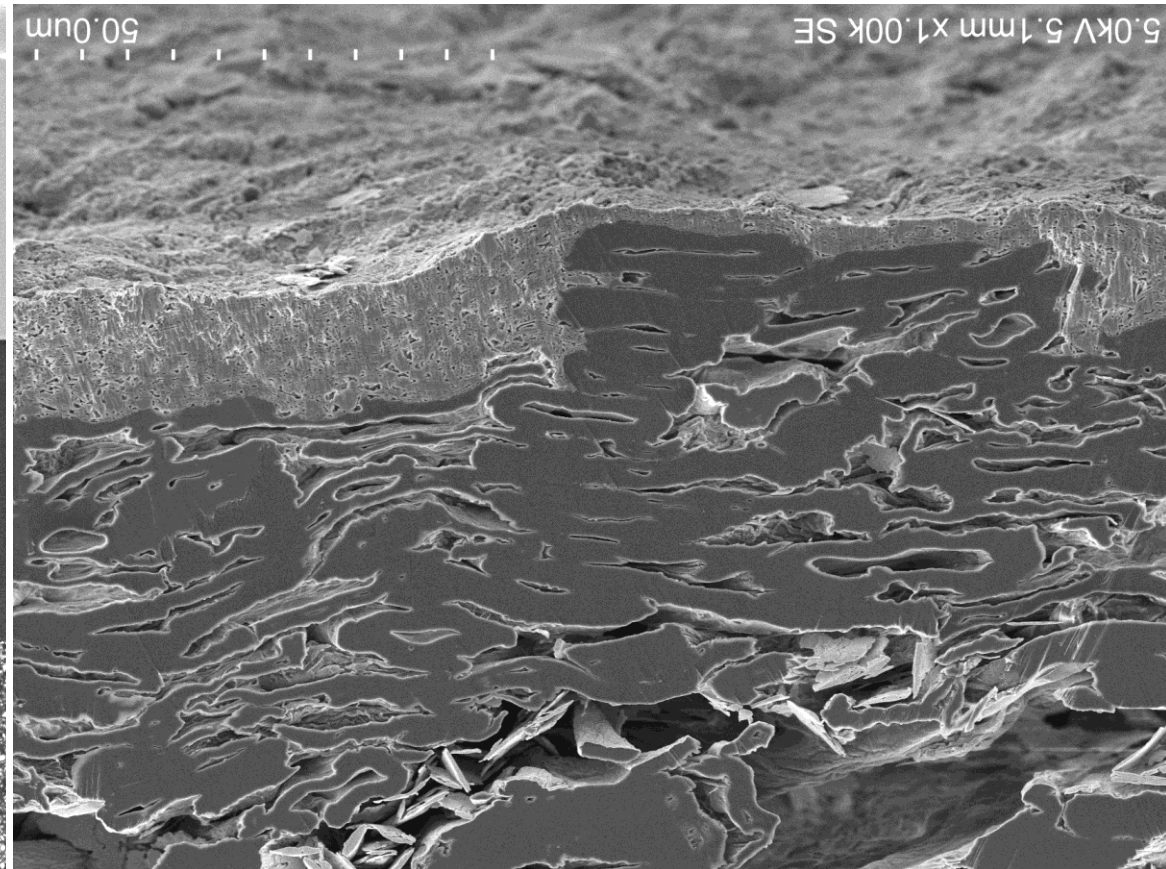
# Acier galvanisé



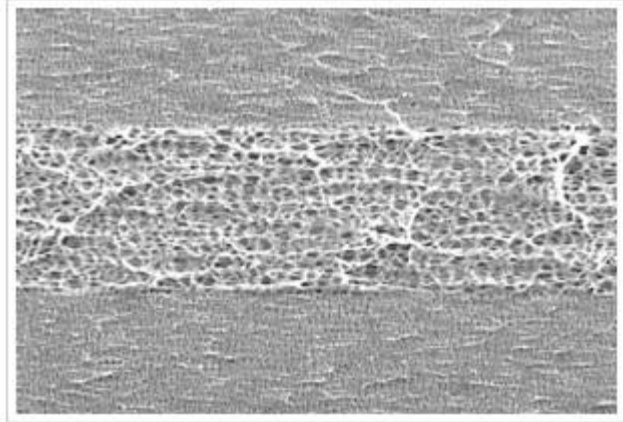
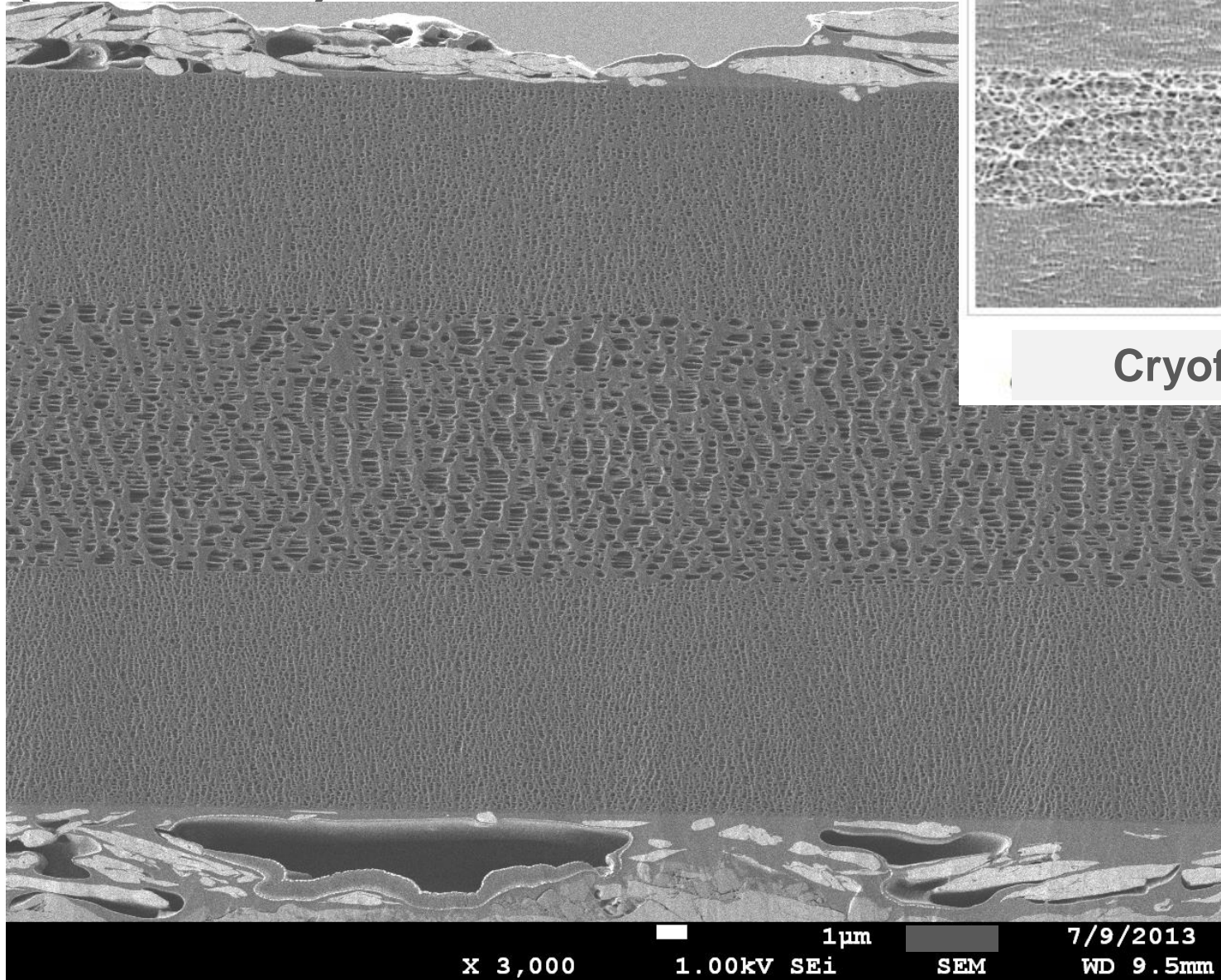
# Peinture



# Papier

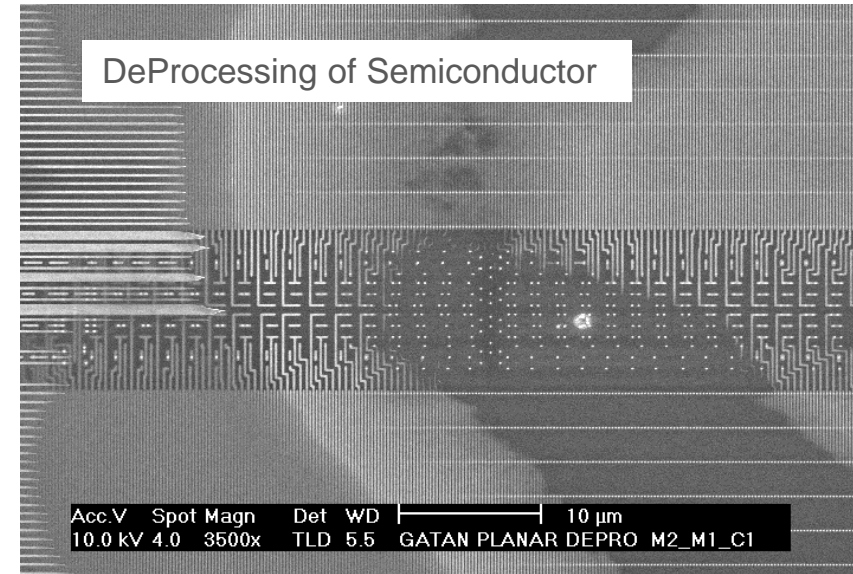
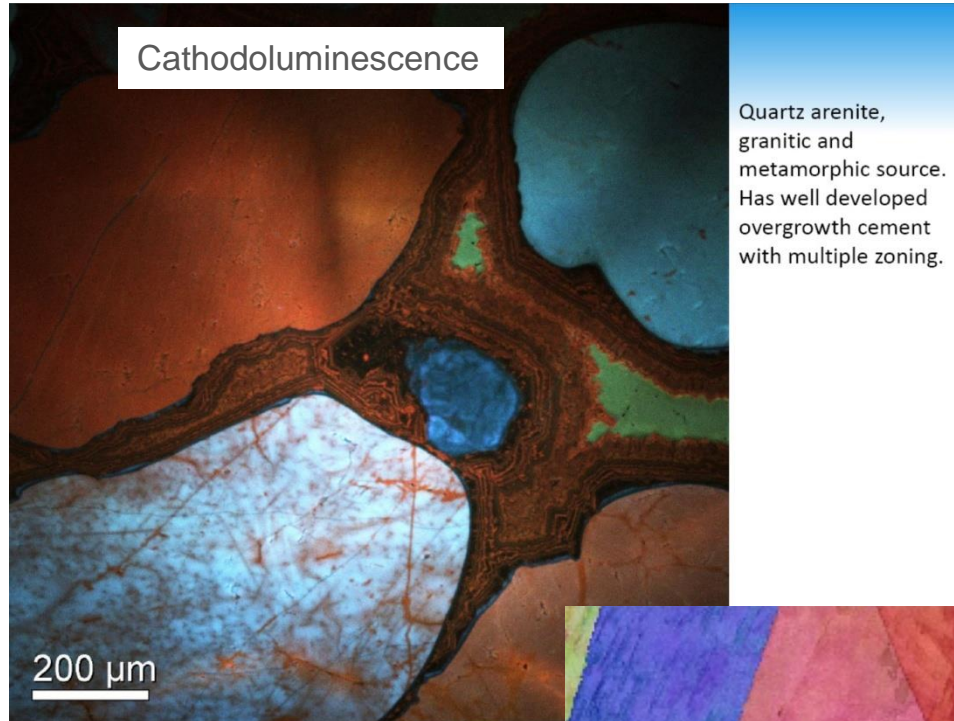


# Trilayer Polypropylene / Polyethylene (PP/PE/PP)

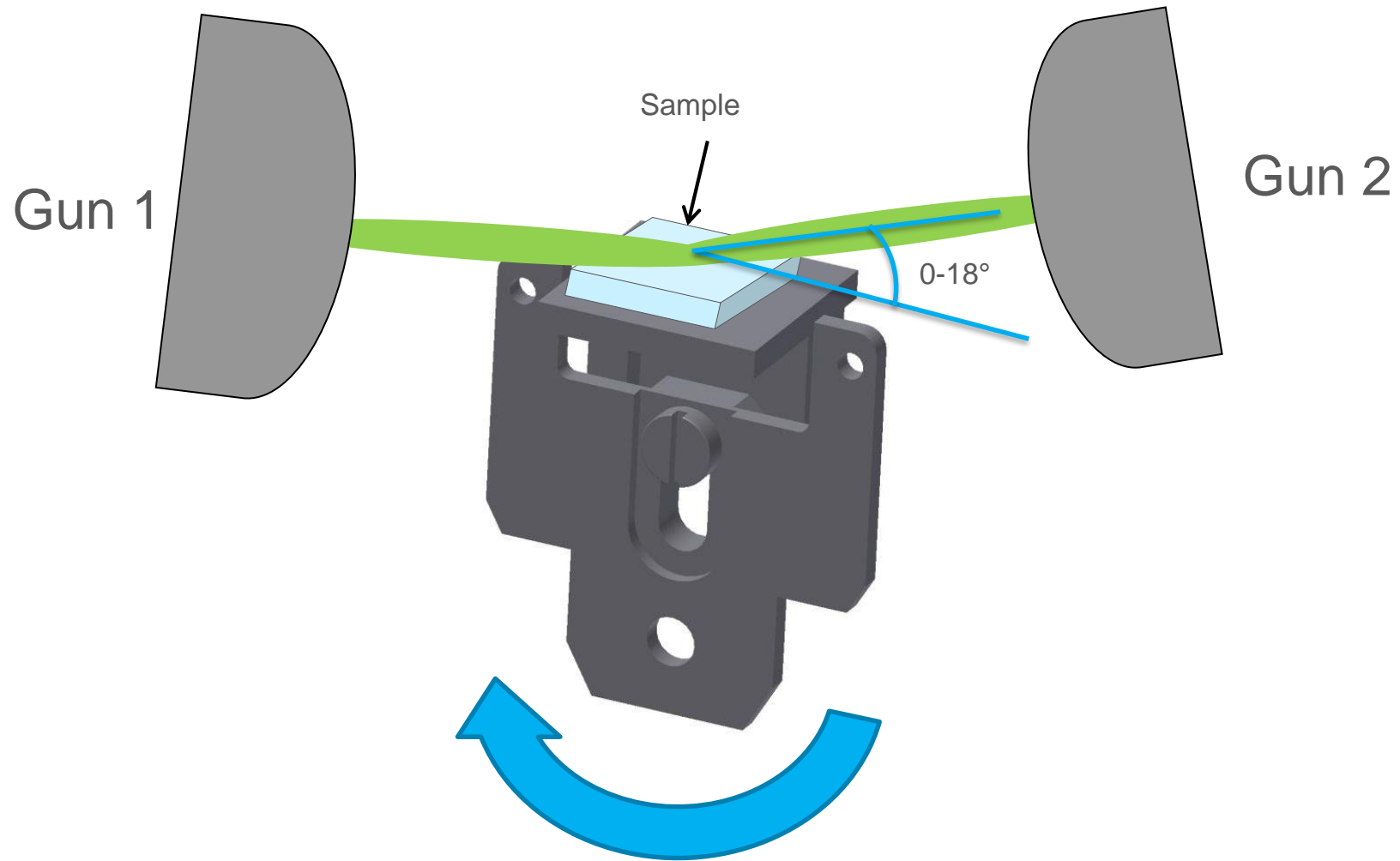


Cryofracture

# Polissage plan par faisceaux d'ions

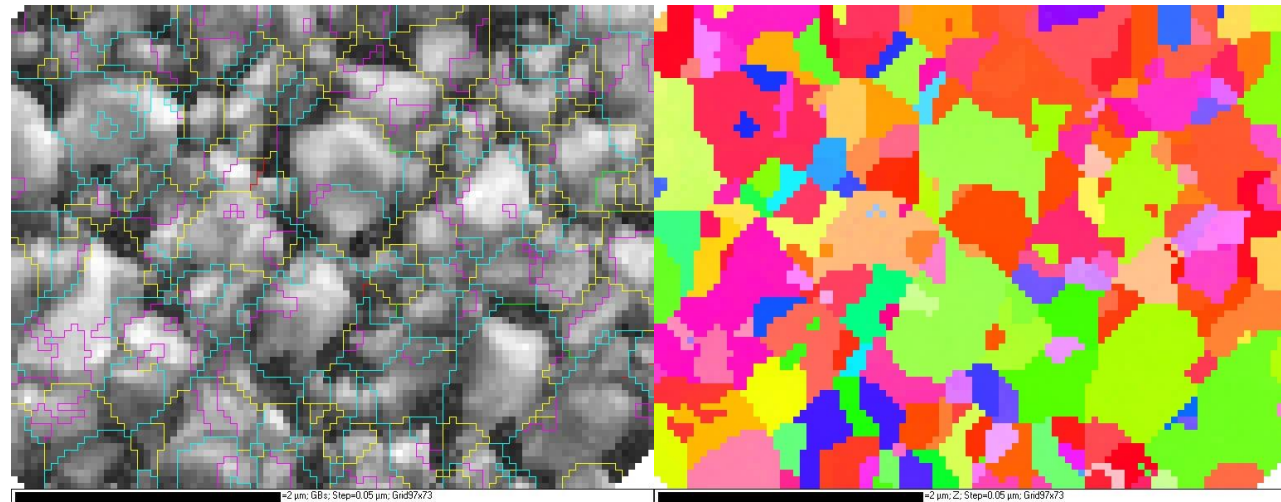
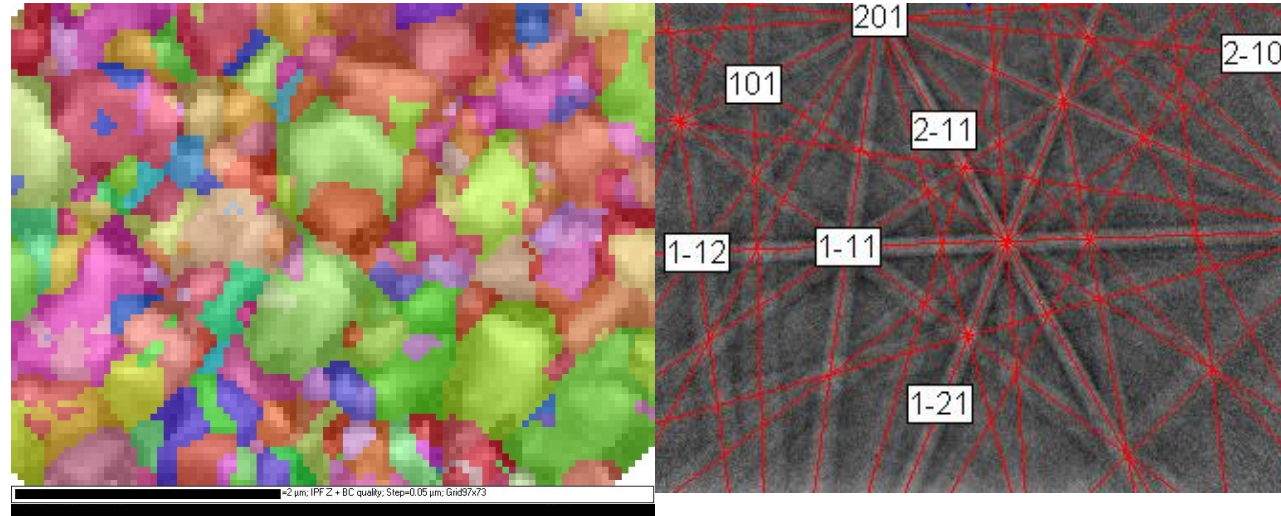
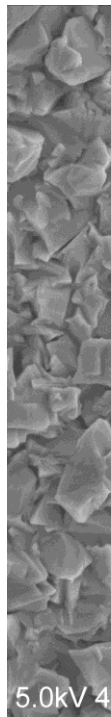


# Polissage plan par faisceaux d'ions (principe)

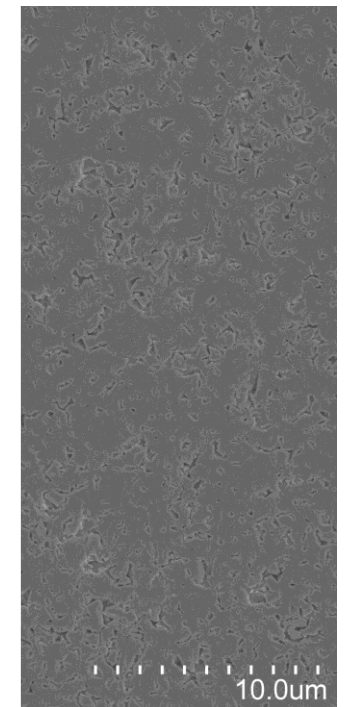


## Dépôt CuInSe<sub>2</sub>

EBSD on CuInSe<sub>2</sub> sample prepared on Gatan Ilion+ polisher

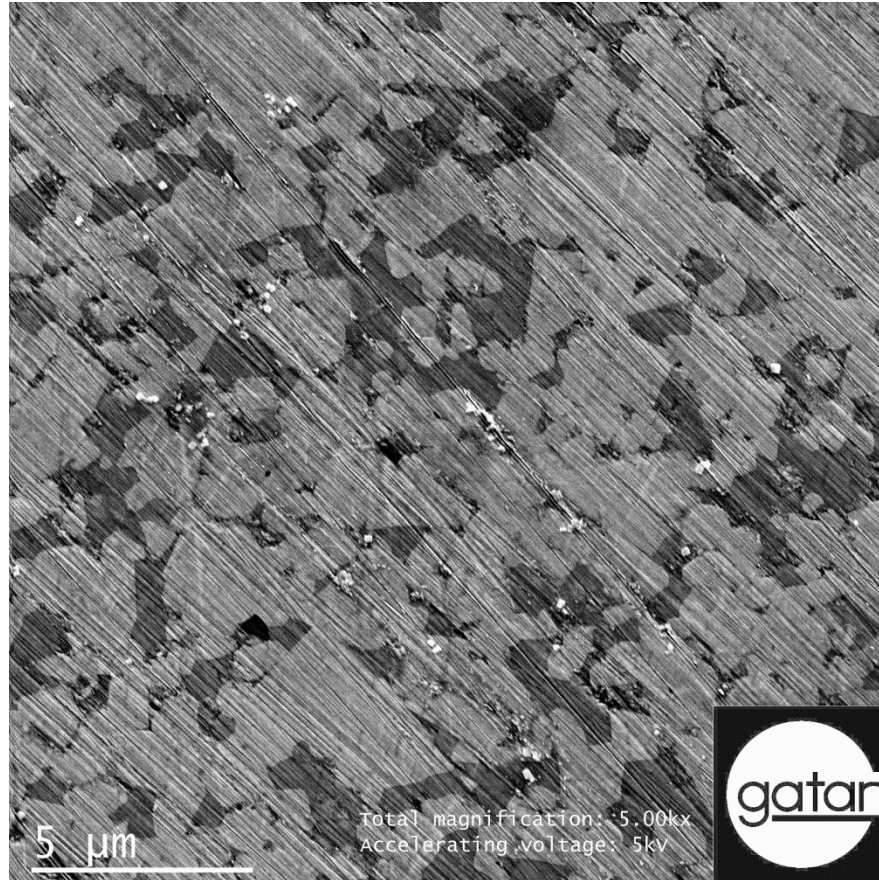


30 Seconds

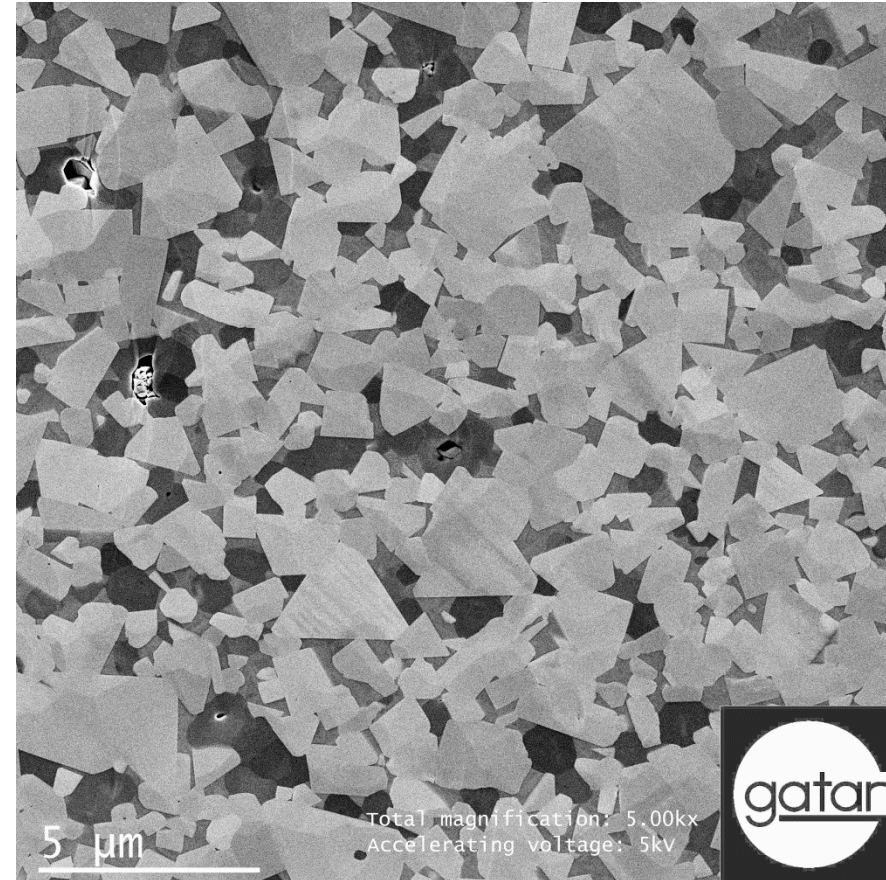


# WC-Co

Mechanical Polished to 3  $\mu\text{m}$  diamond

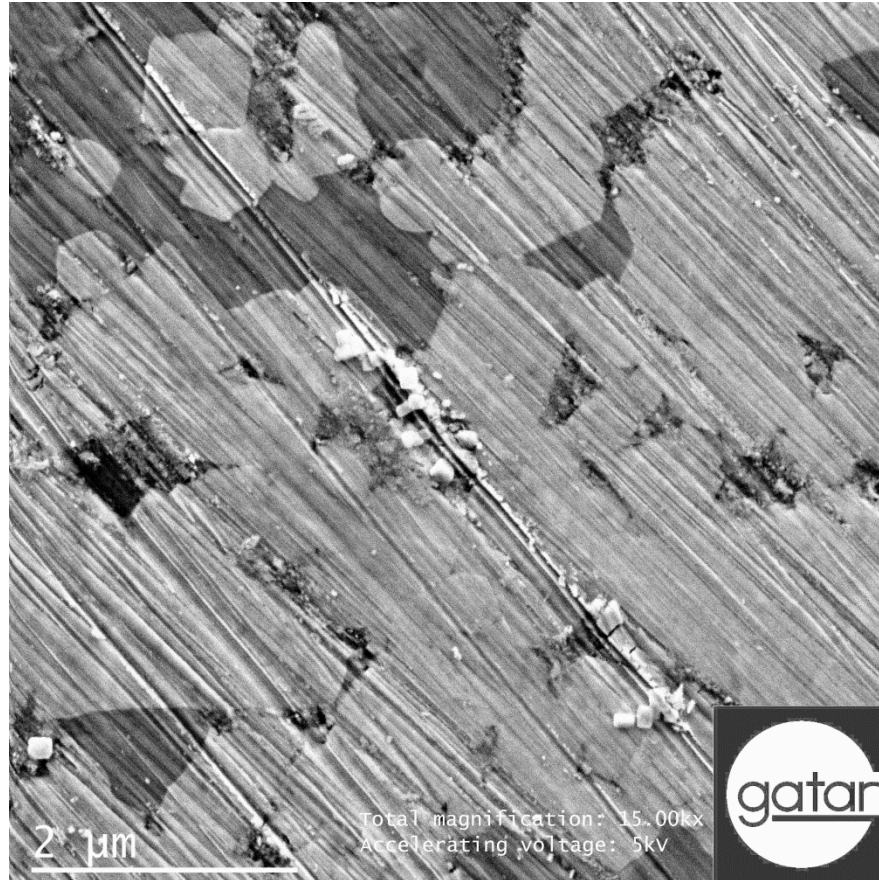


Planar Polish In Ilion

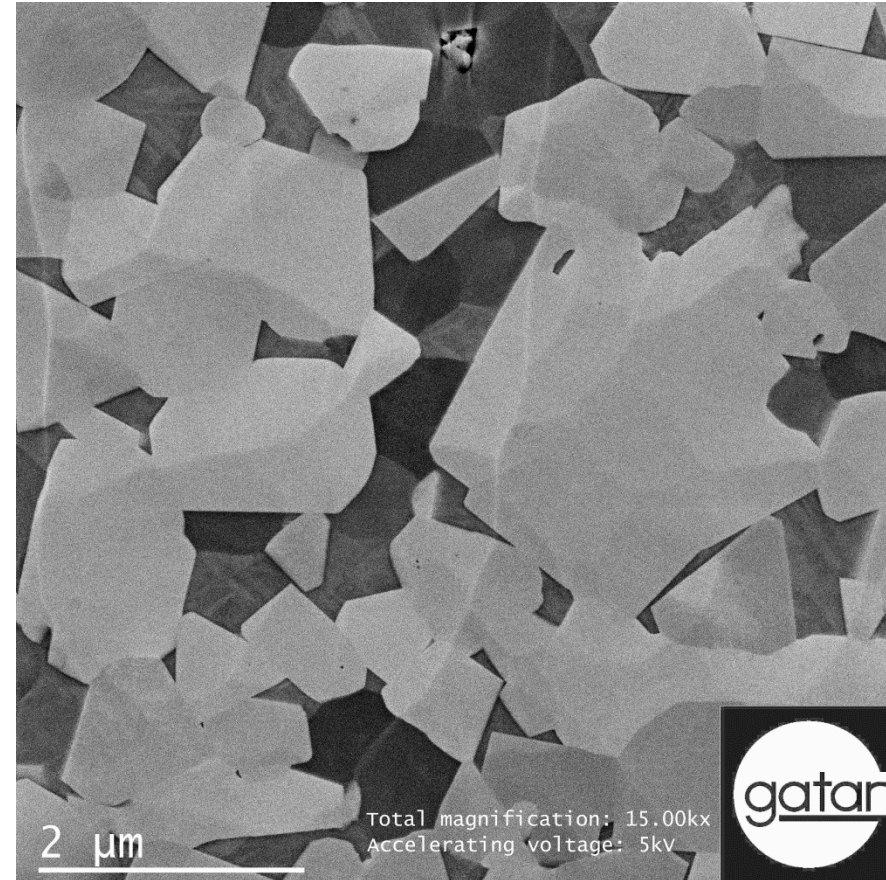


# WC-Co

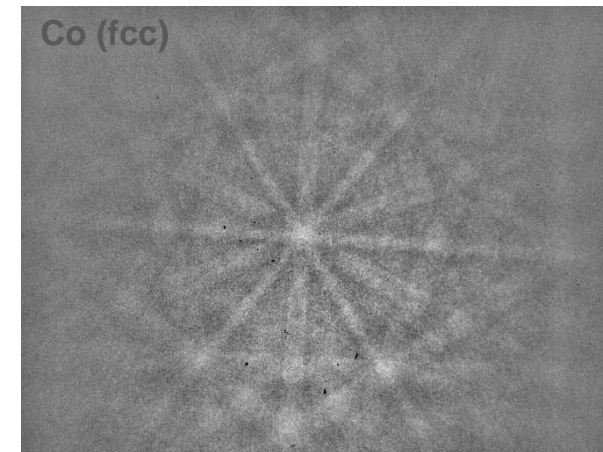
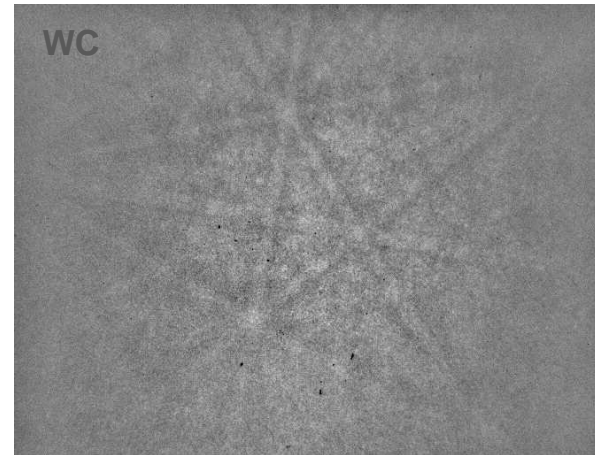
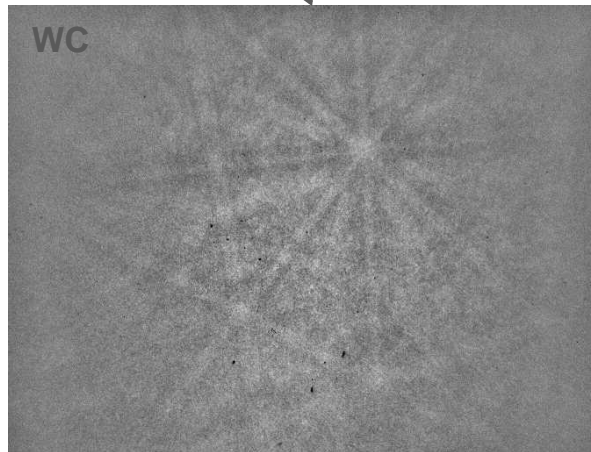
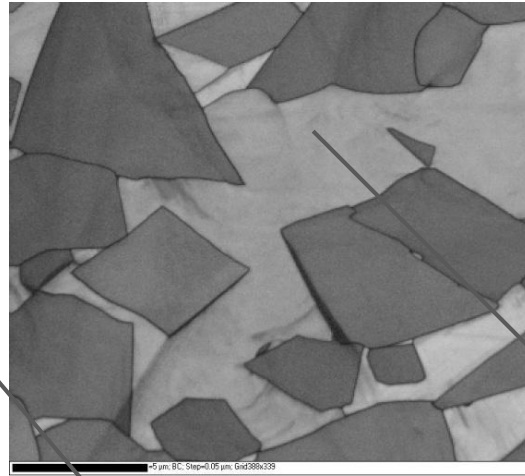
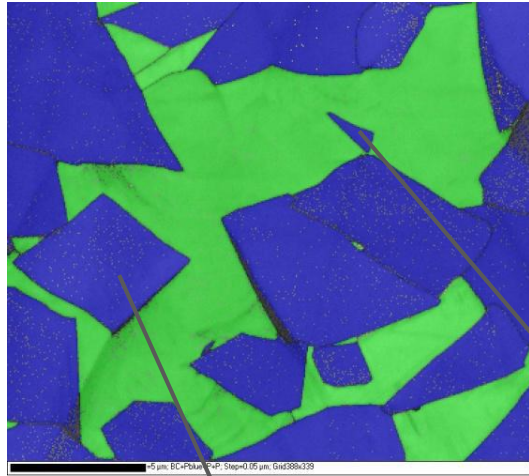
Mechanical Polished to 3 $\mu$ m diamond



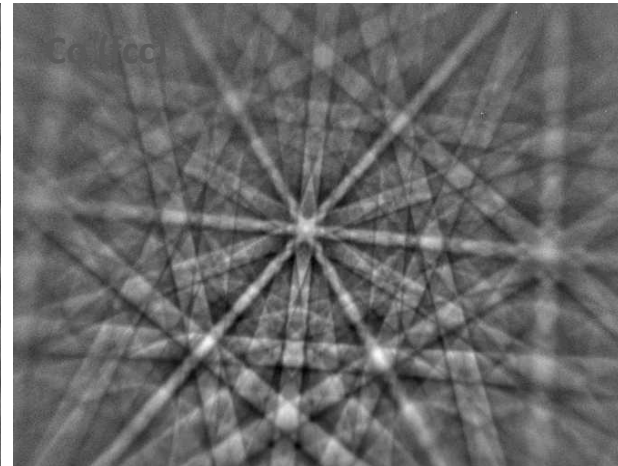
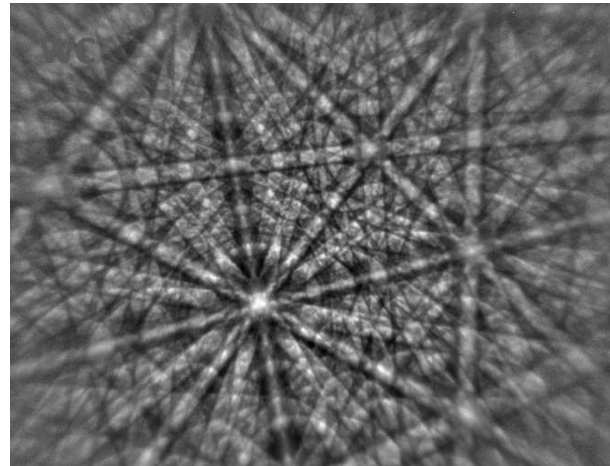
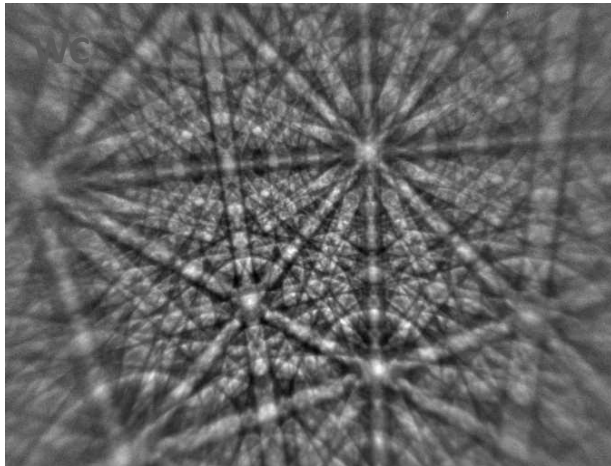
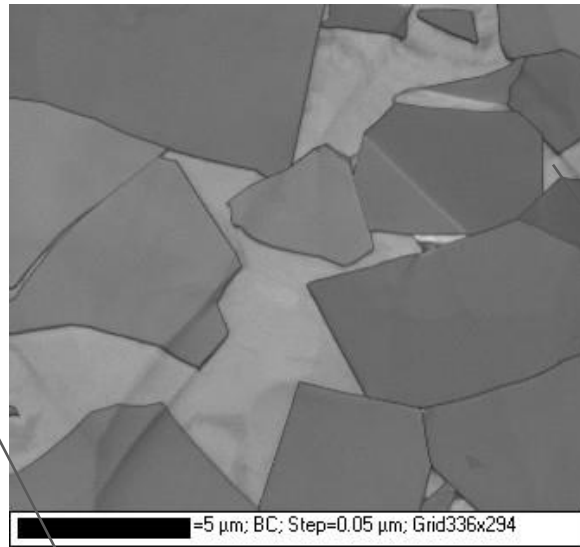
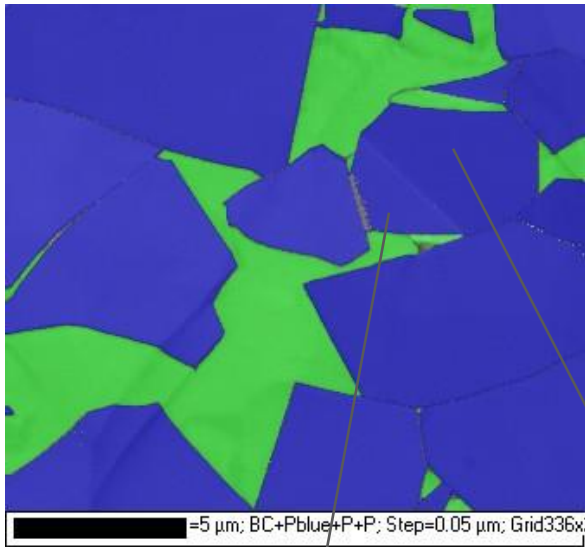
Planar Polish In Ilion



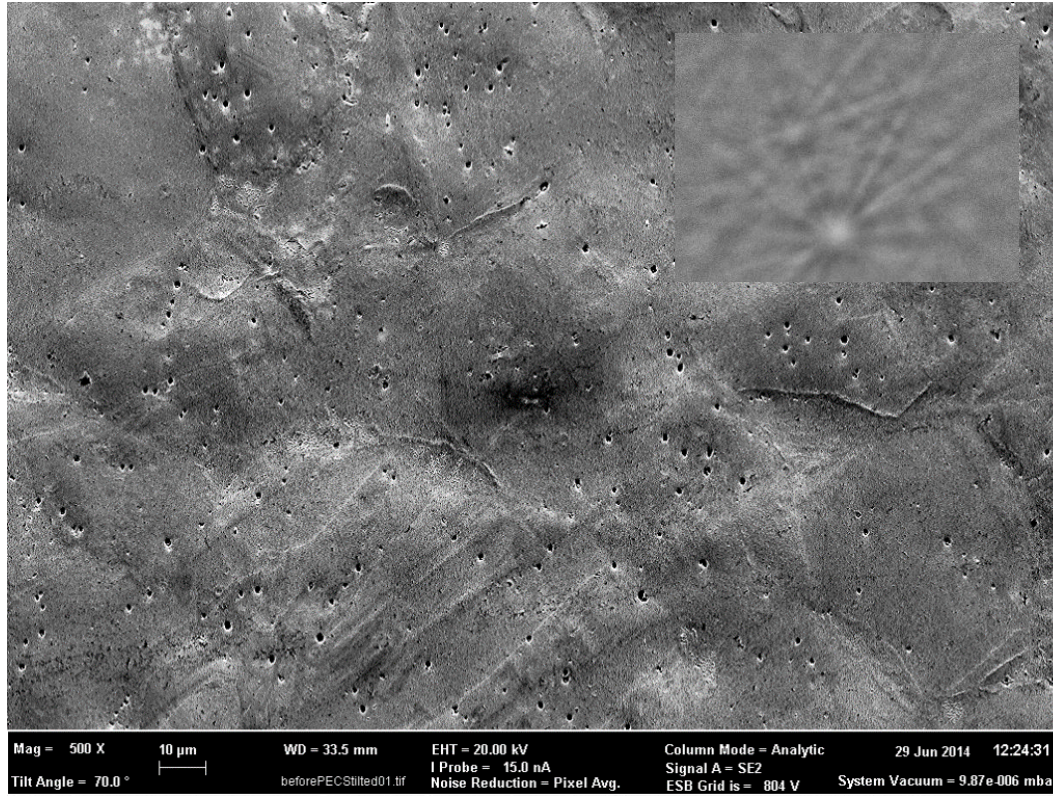
# WC- Co (Argon 8KV)



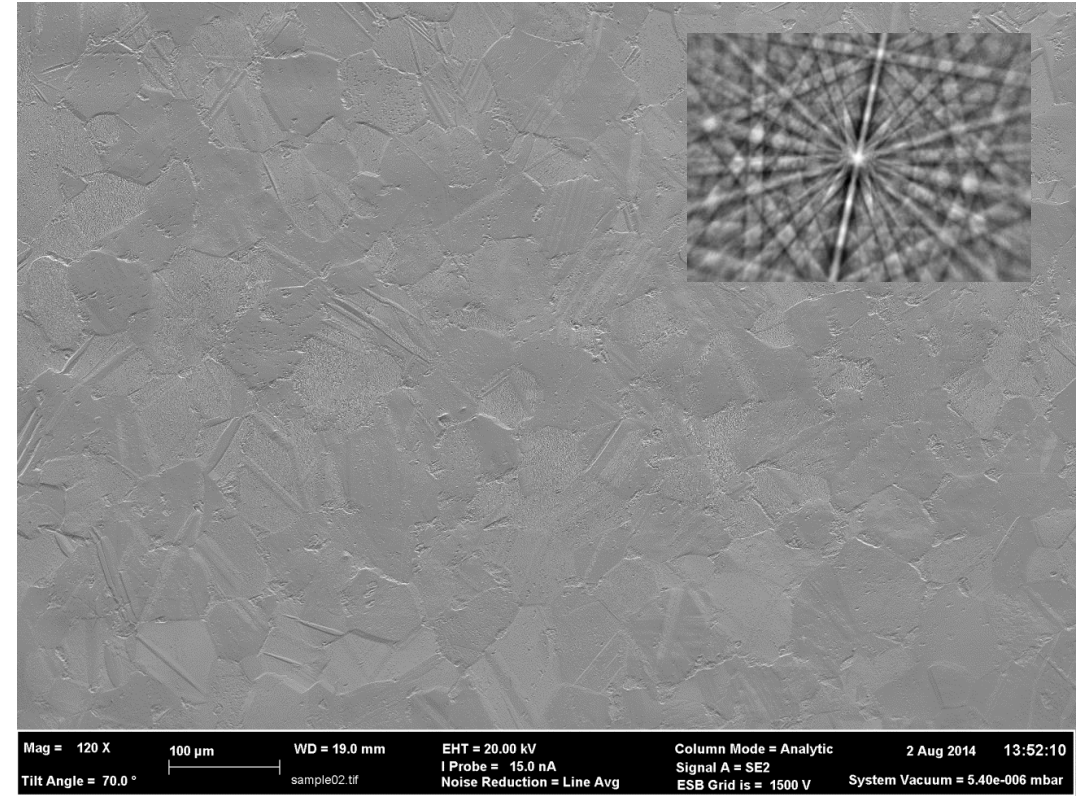
# WC- Co (Argon 1KV)



# PECS II Zircaloy

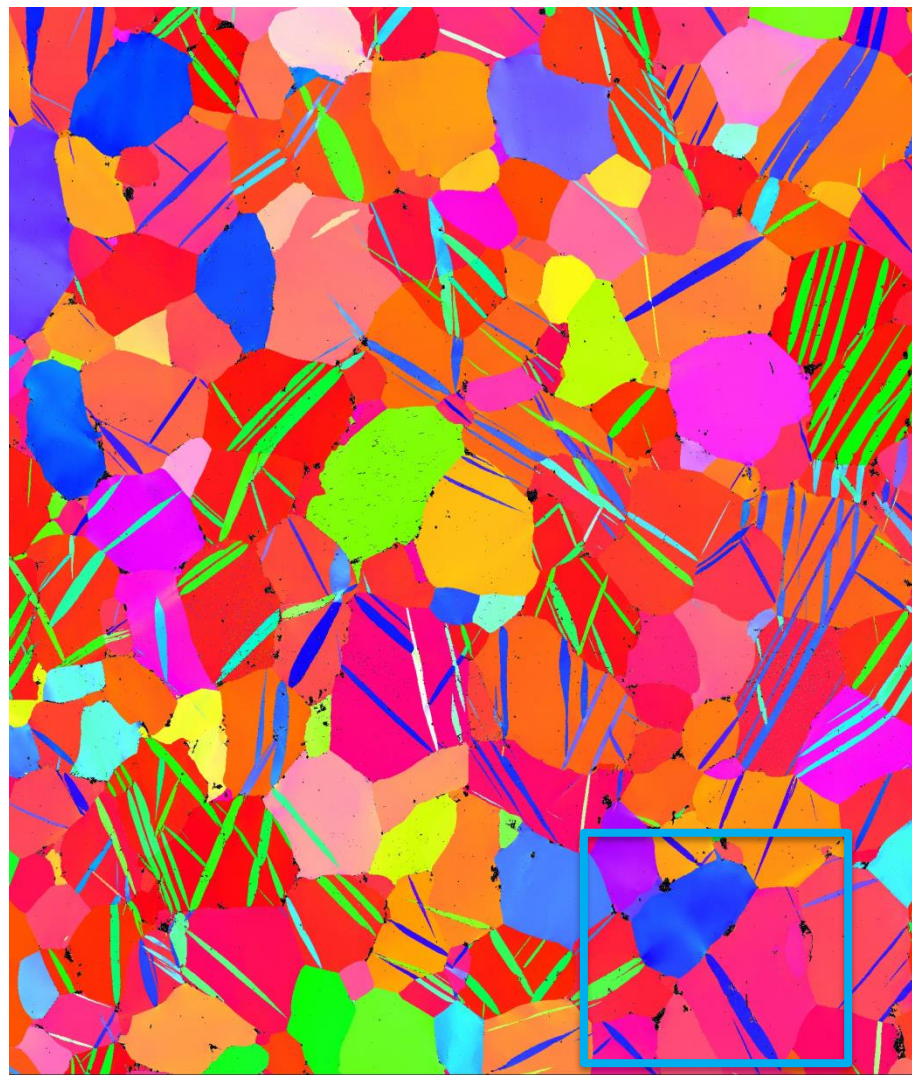


Mech. Polish + etch colloidal silica



PECS 2 8key, 15 minutes

# IPFZ Map: Pixel size 0.31 $\mu\text{m}$ Acquisition Time 18 hr



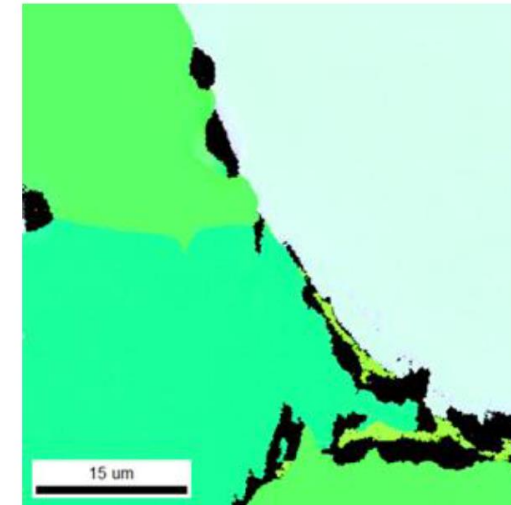
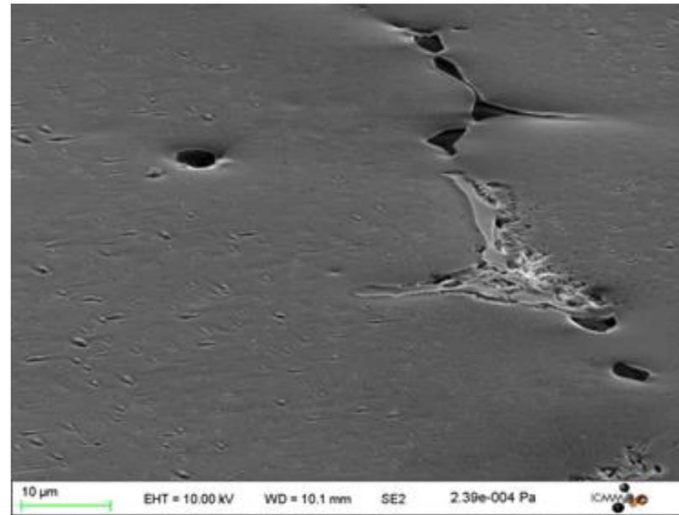
ARGUS: IPFXMap  
Px: 0.31  $\mu\text{m}$  MapSize: 3000 x 2250



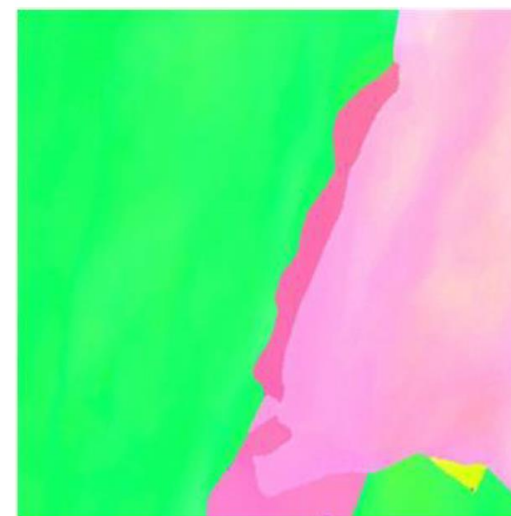
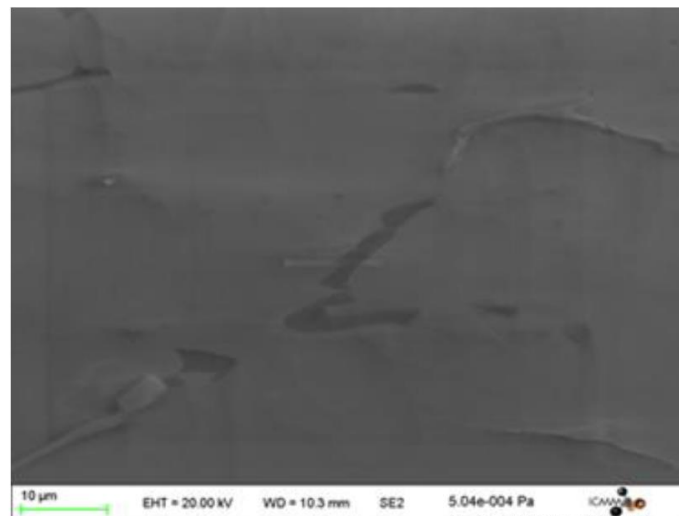
ARGUS: IPFXMap  
Px: 0.44  $\mu\text{m}$  MapSize: 512 x 384

# Al 6061 : polissage électrolytique versus PECS II

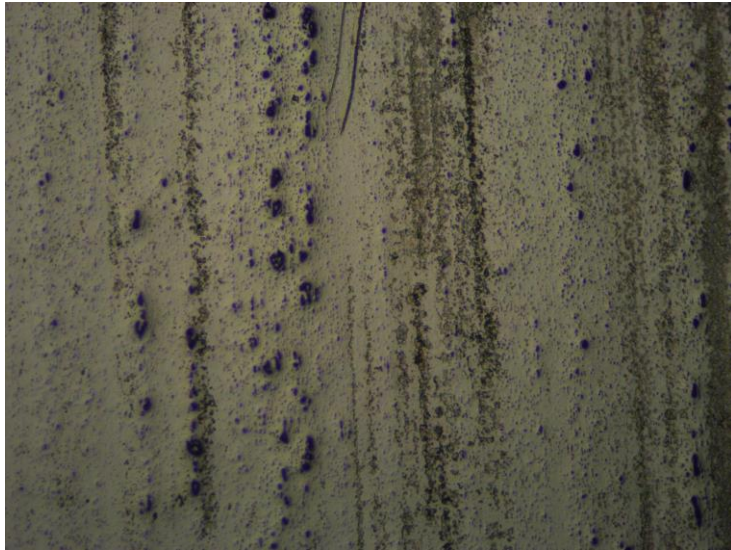
Polissage électrolytique



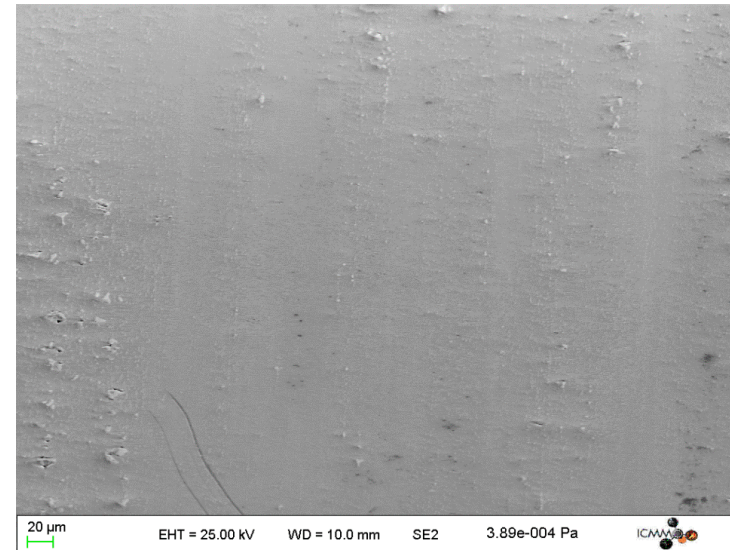
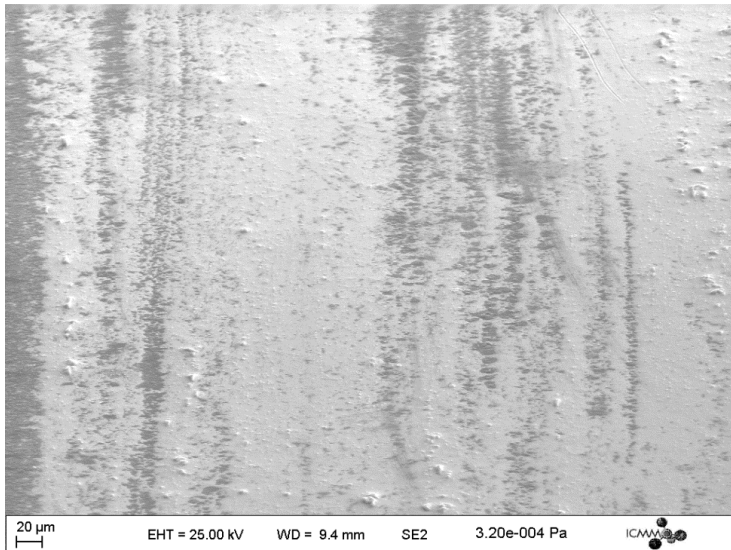
Polissage ionique  
(PECS 2)



# Alliage aluminium: polissage OPS versus PECS II



Optical microscope (Ilion2)

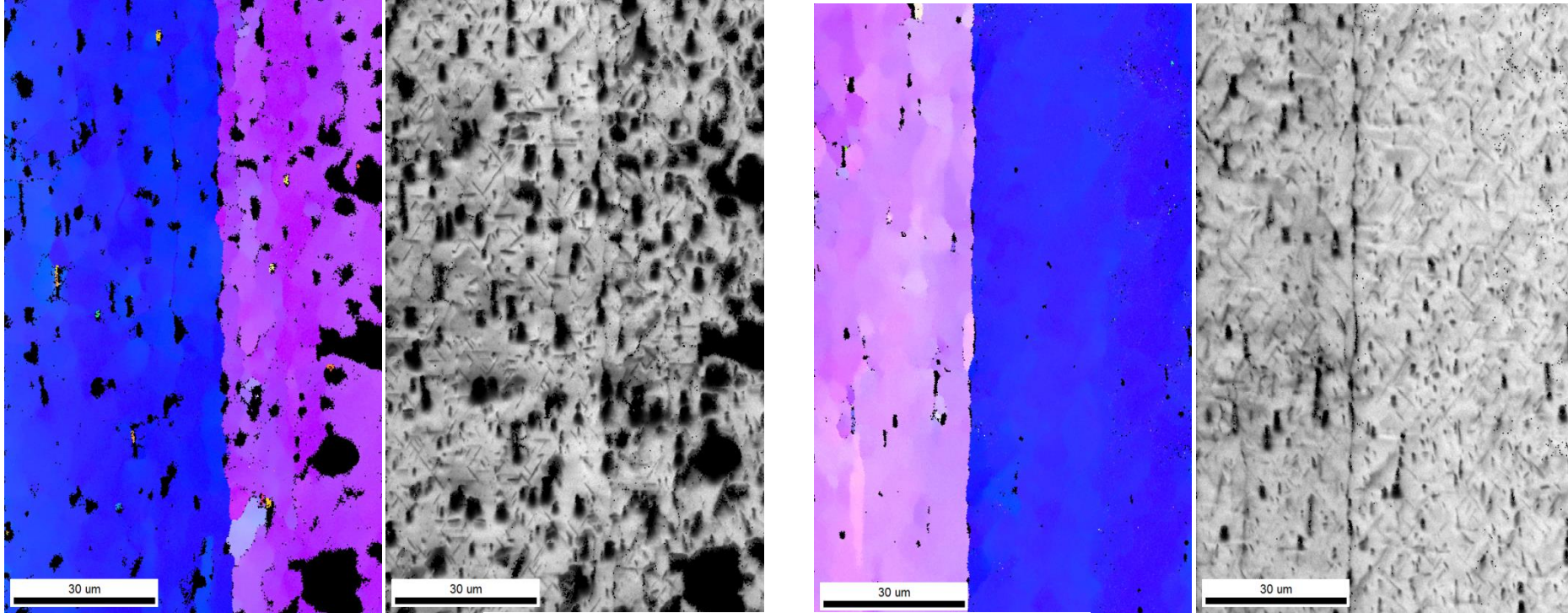


MEB Zeiss Supra 55

Finition silice colloïdale

Reprise PECS 2

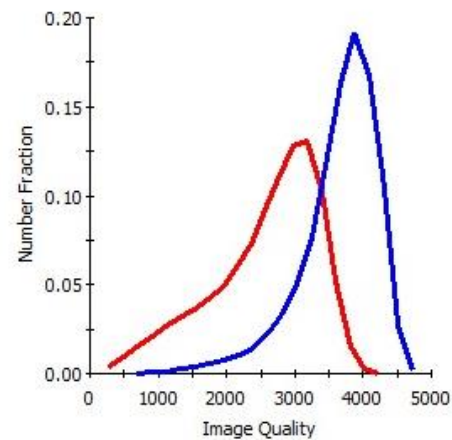
# Alliage aluminium



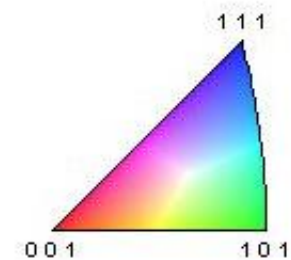
IPF map (suppression des points avec indice de confiance < 0.1)

Image Quality

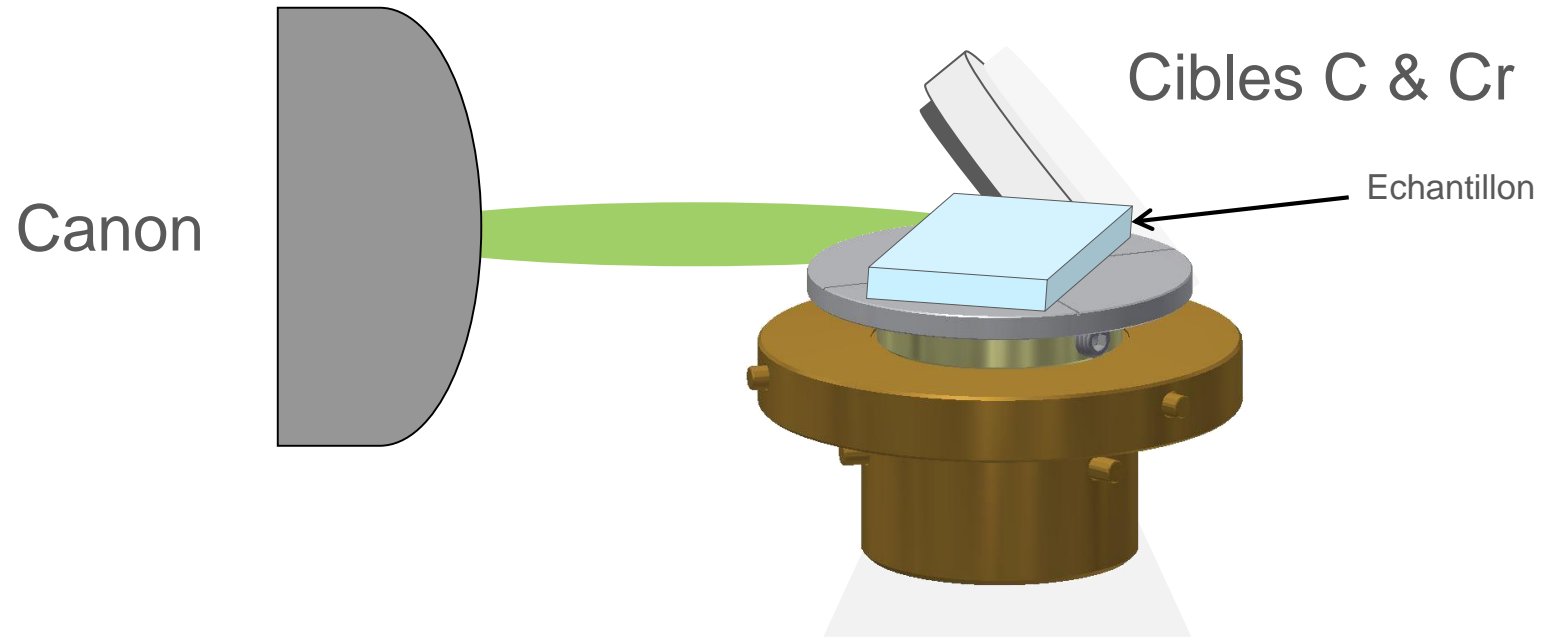
Quality map



— brut  
— 3kv-1kv Ilion2



# Métallisation In Situ



Cibles disponibles: C, Cr, Au, Pt, Ir, PD-Au, Si, Al<sub>2</sub>O<sub>3</sub> ...

Confidential

# Conclusion

- Les systèmes Gatan permettent le polissage et la découpe par faisceaux d'ions tout en préservant la surface de l'échantillon.
- Les spécificités des systèmes Gatan sont :
  - Montage des échantillons en dehors de la chambre de préparation
  - SAS offre la possibilité de transférer et reprendre l'échantillon rapidement
  - Microscope optique pour l'observation en cours de polissage
  - Platine froide pour les échantillons sensibles
  - Coupe transverse (>2mm)
  - Polissage plan ( >10 mm<sup>2</sup>)
  - Métallisation In-Situ
  - Transfert sous vide ou atmosphère de l'échantillon
- La préparation ionique est une technique de choix pour les analyses suivantes : CL, EDS, EBSD, WDS.