



Complémentarité des imageries MEB et MET

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Etudier la matière avec des électrons

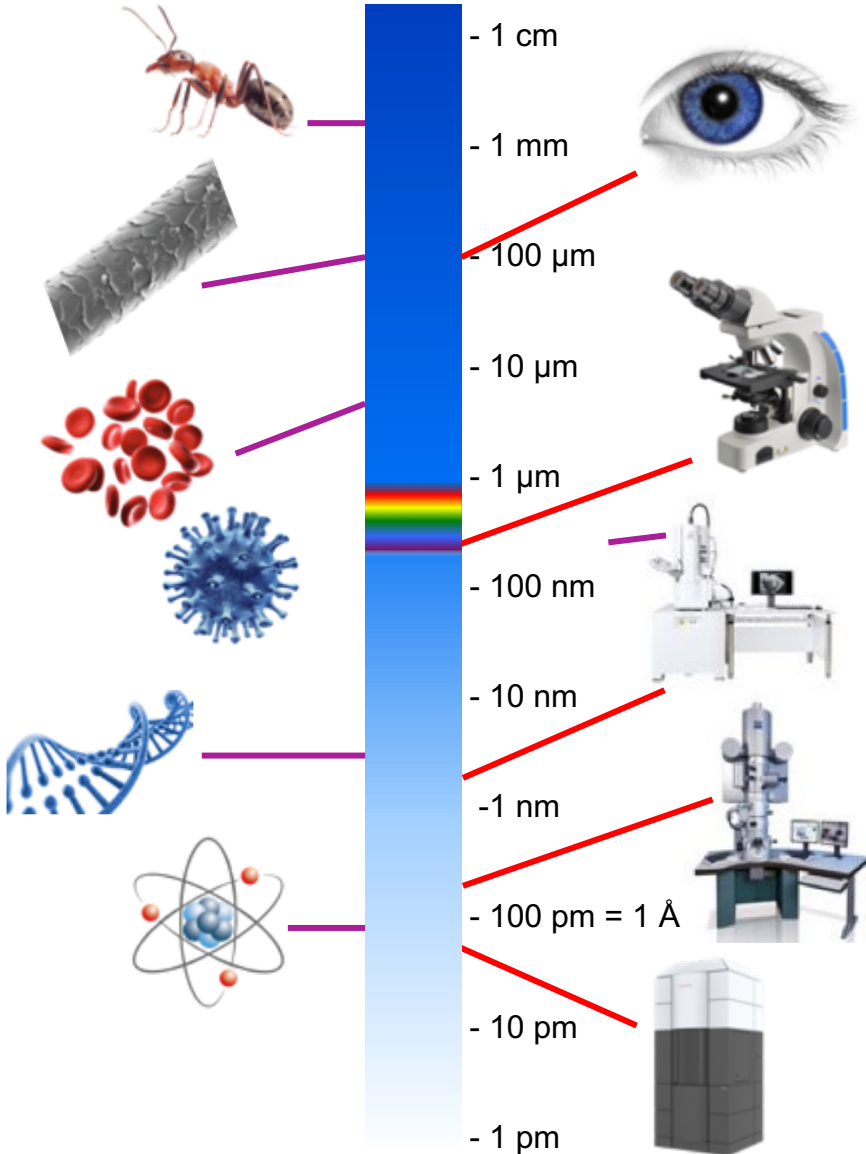
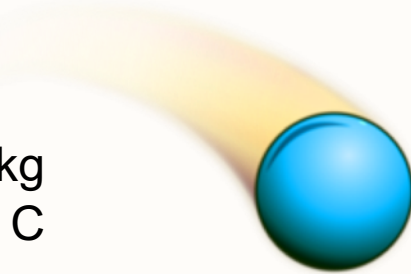
Nom : ELECTRON
Symbole : e^- (β^-)
Date de découverte : 1897

famille : Fermion
groupe : lepton

Masse : $9,1 \cdot 10^{-31}$ kg
Charge : $1,6 \cdot 10^{-19}$ C
Spin : $\frac{1}{2}$

Signes particuliers :

- facile à accélérer et à manipuler
- onde et particule
- forte interaction avec la matière



A l'heure de l'apéro



Les bons ingrédients

Le bon matériel



Le savoir faire

De belles réalisations

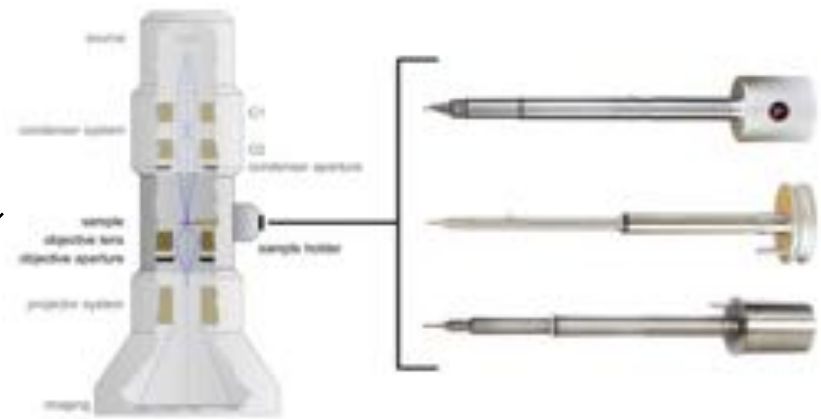


A l'heure de l'apéro



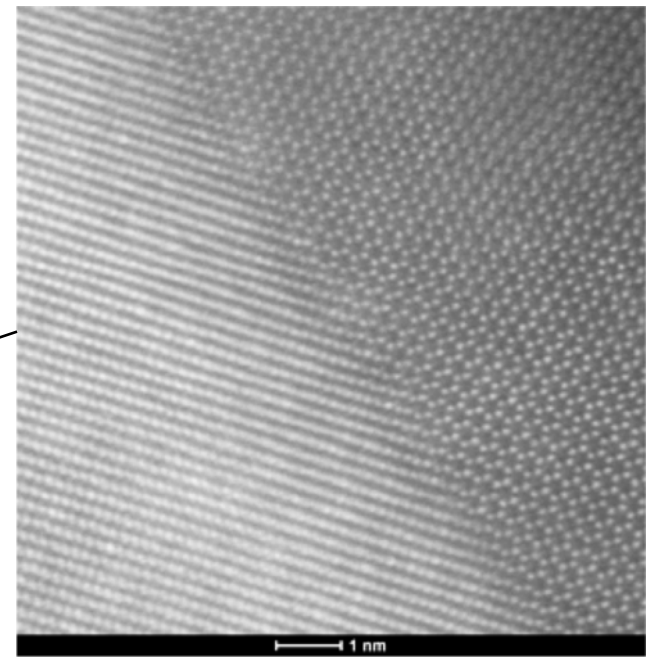
Les bons ingrédients

Le bon matériel

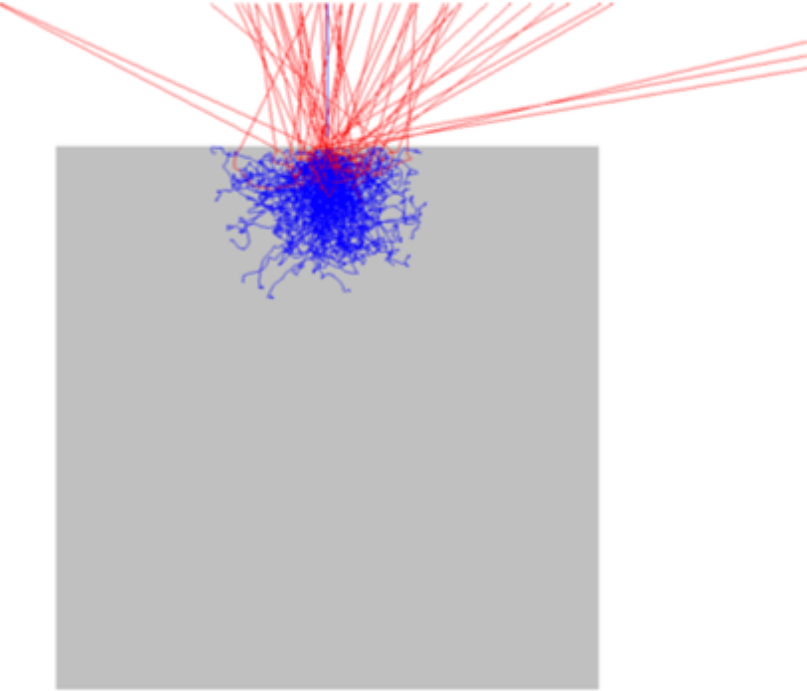
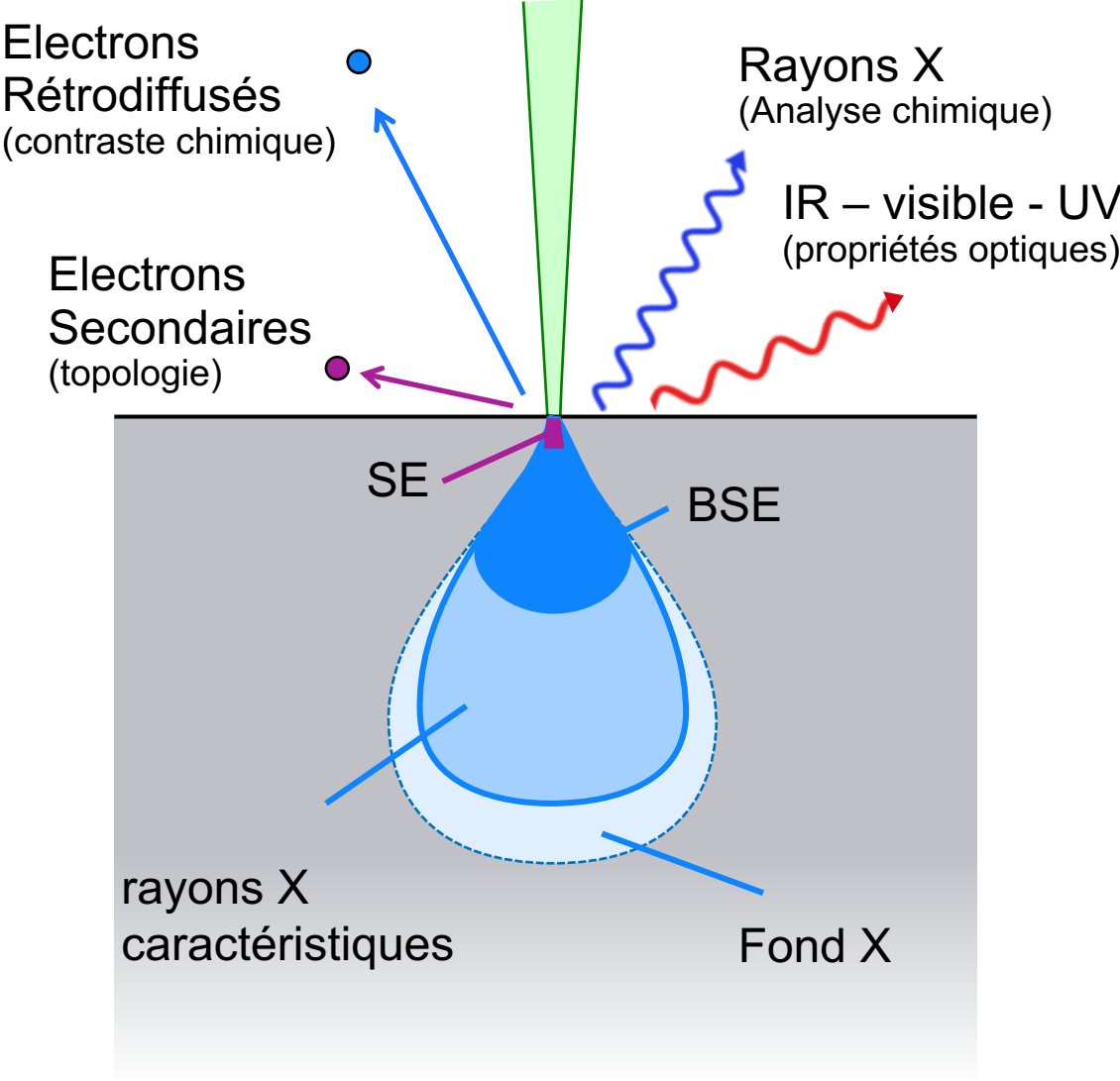


Le savoir faire

De belles réalisations

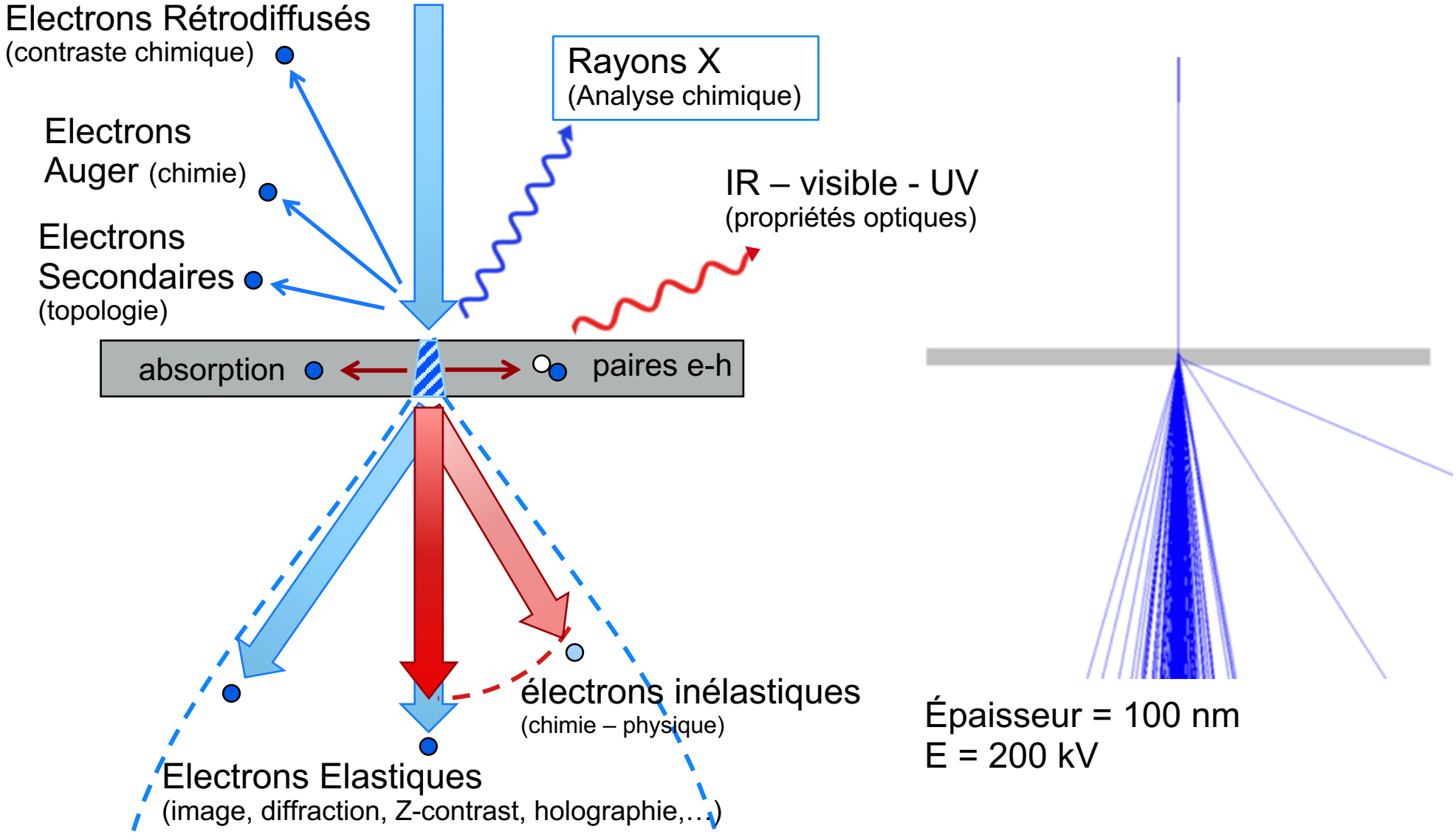


Quels signaux

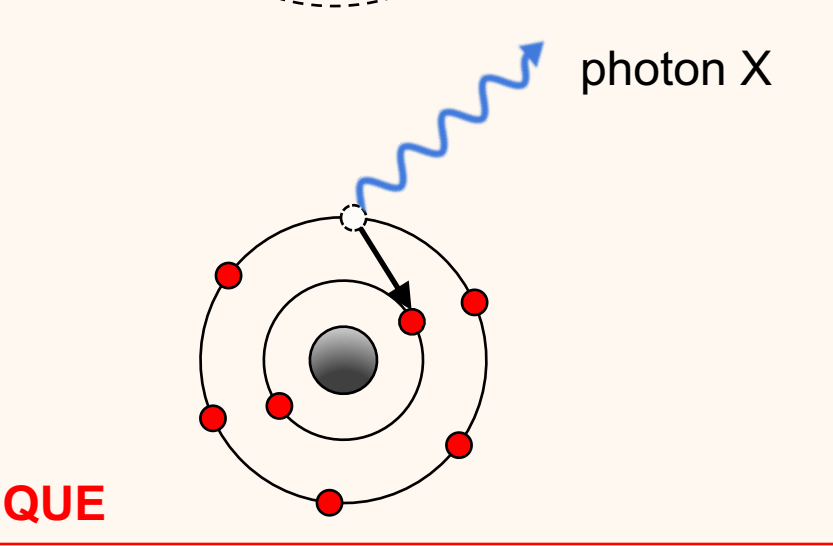
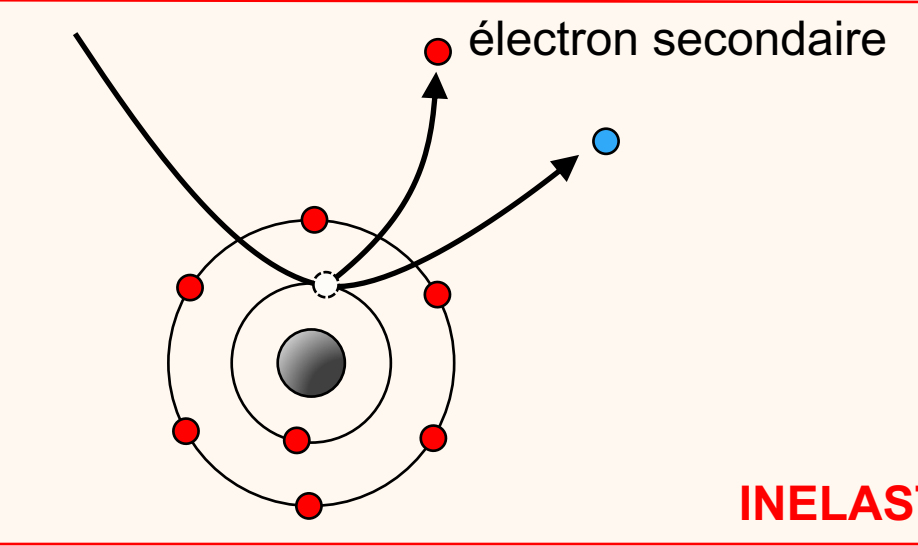
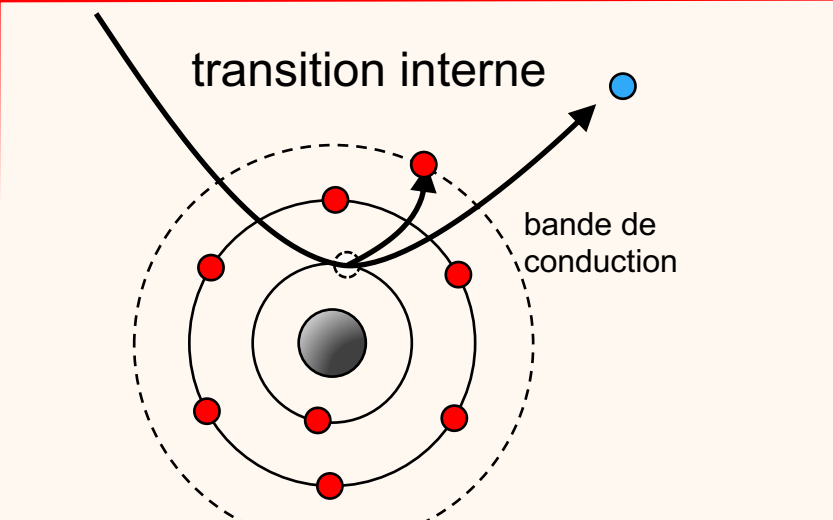
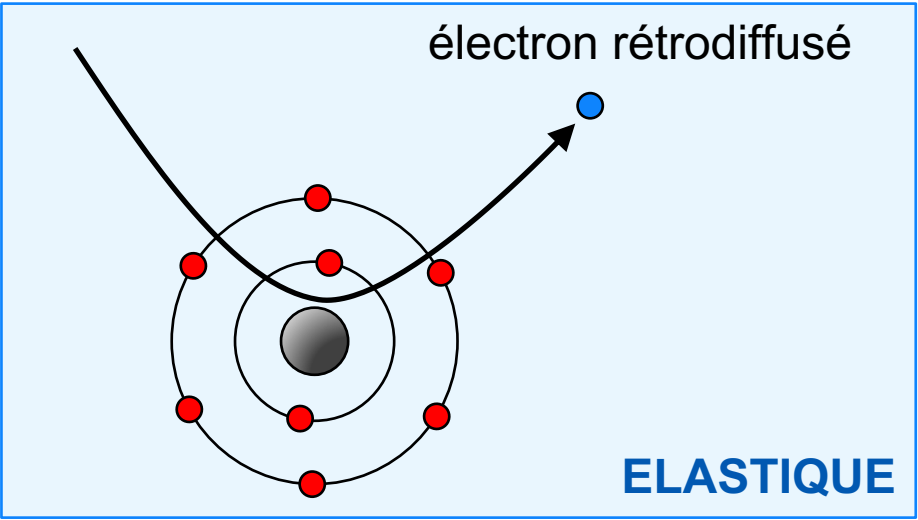


Épaisseur = 3 μm
E = 15 kV

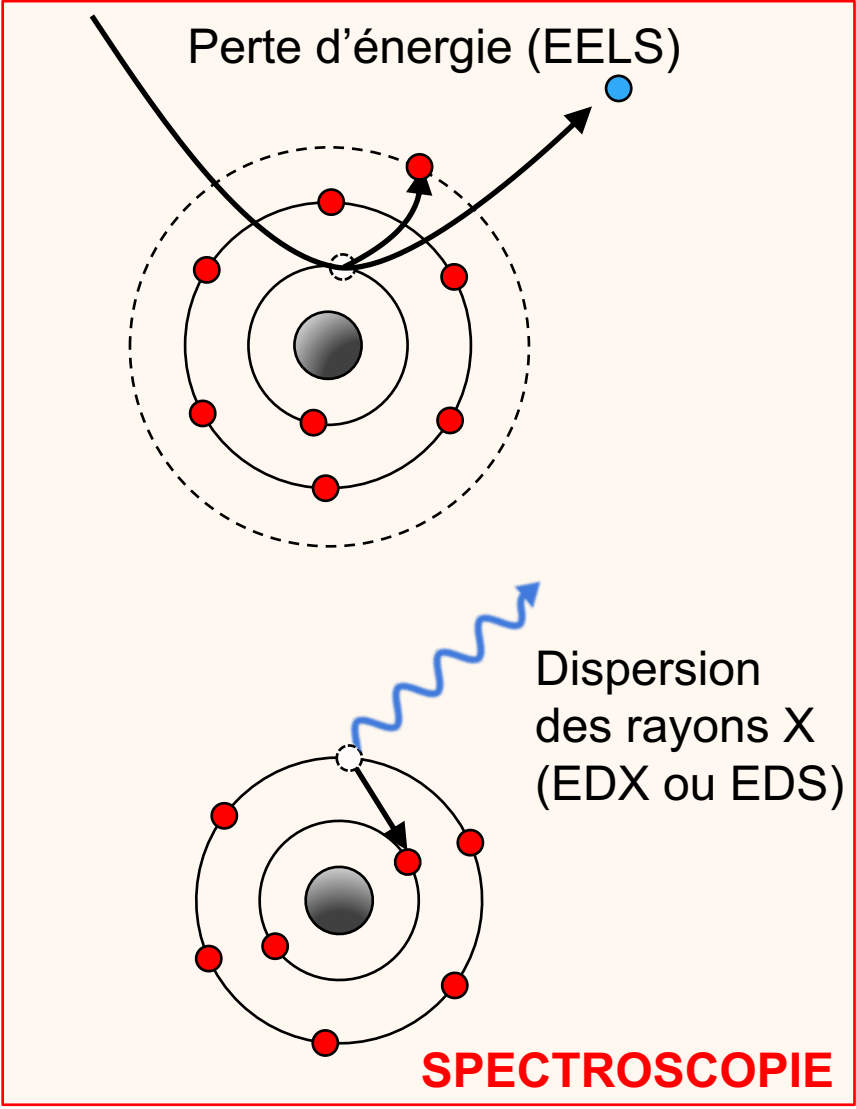
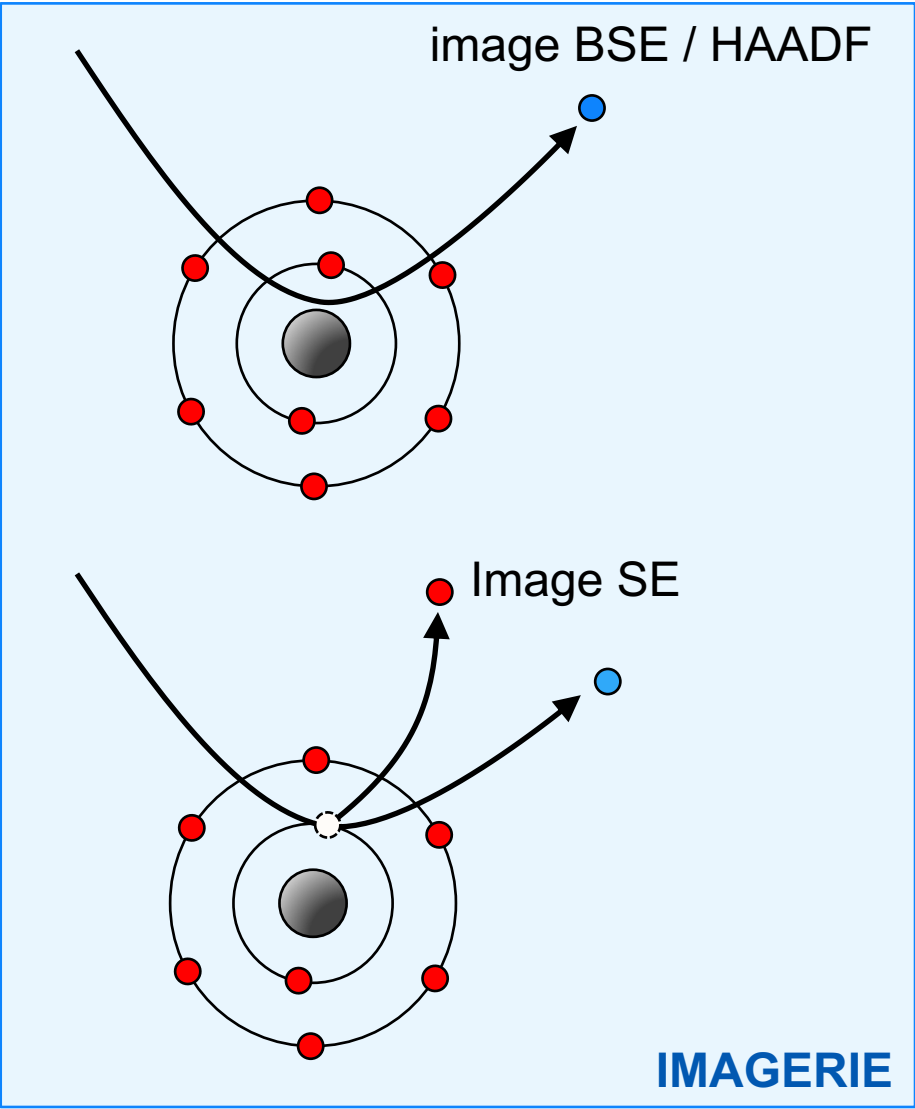
Quels signaux



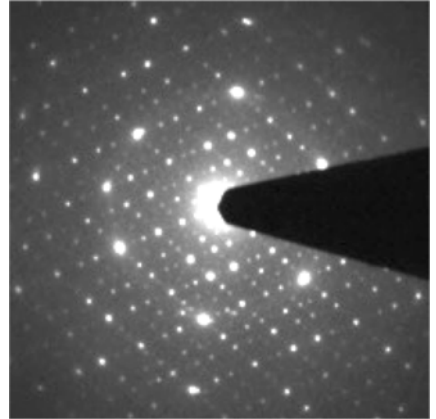
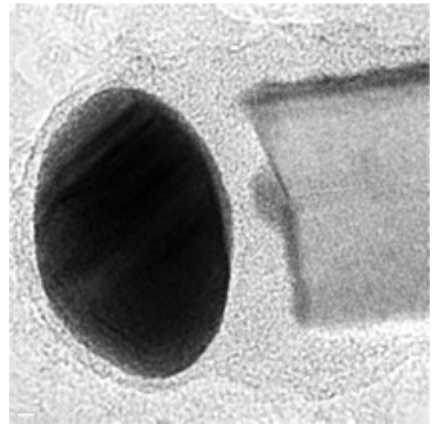
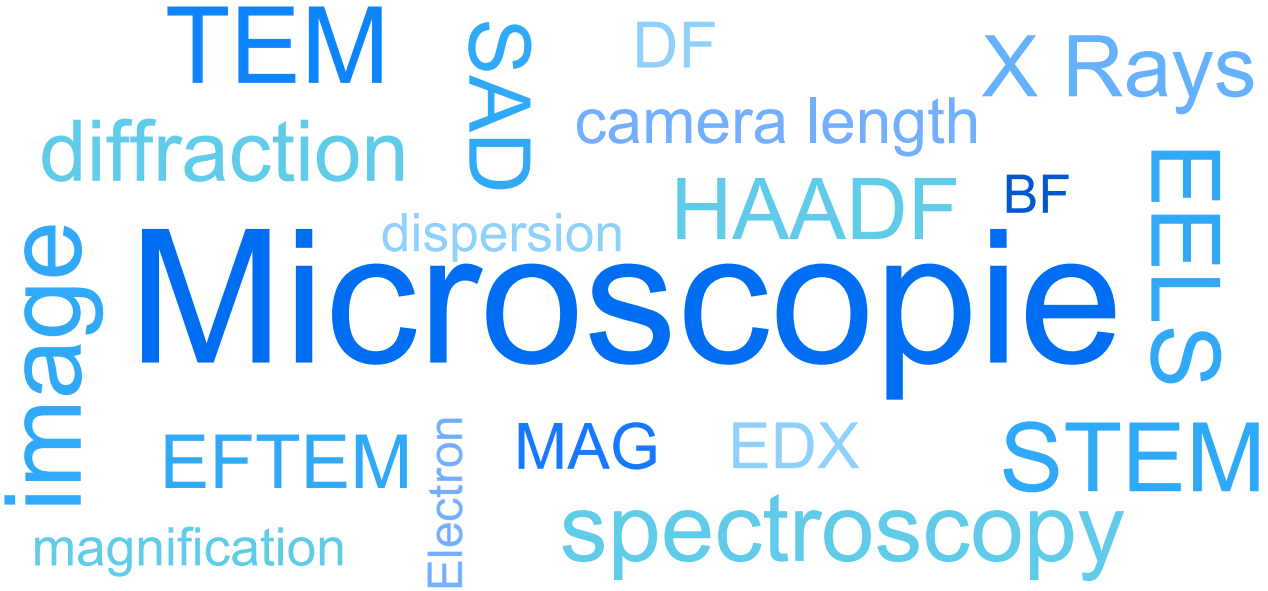
Interactions électrons/matière



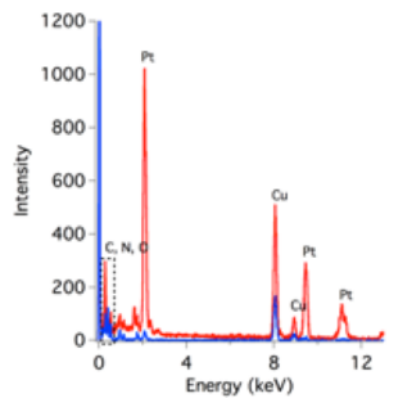
Interactions électrons/matière



Acronymes



- Image
 - Cristallographie
 - Analyse



2 modes de fonctionnement

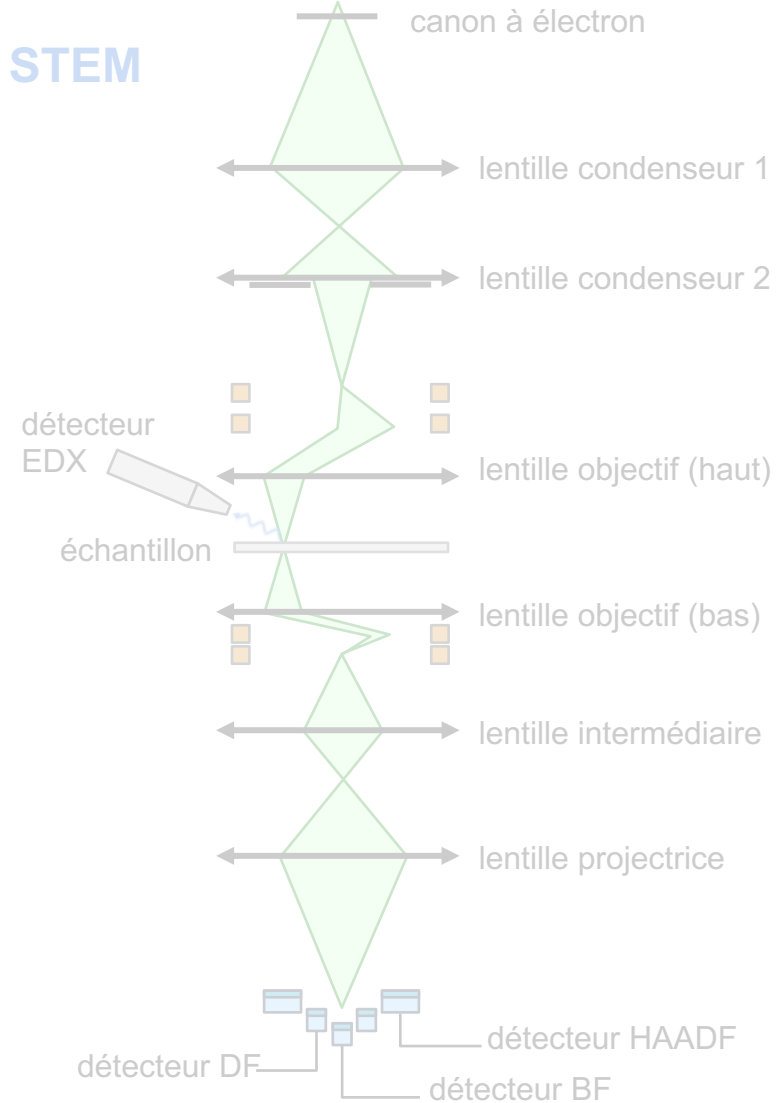
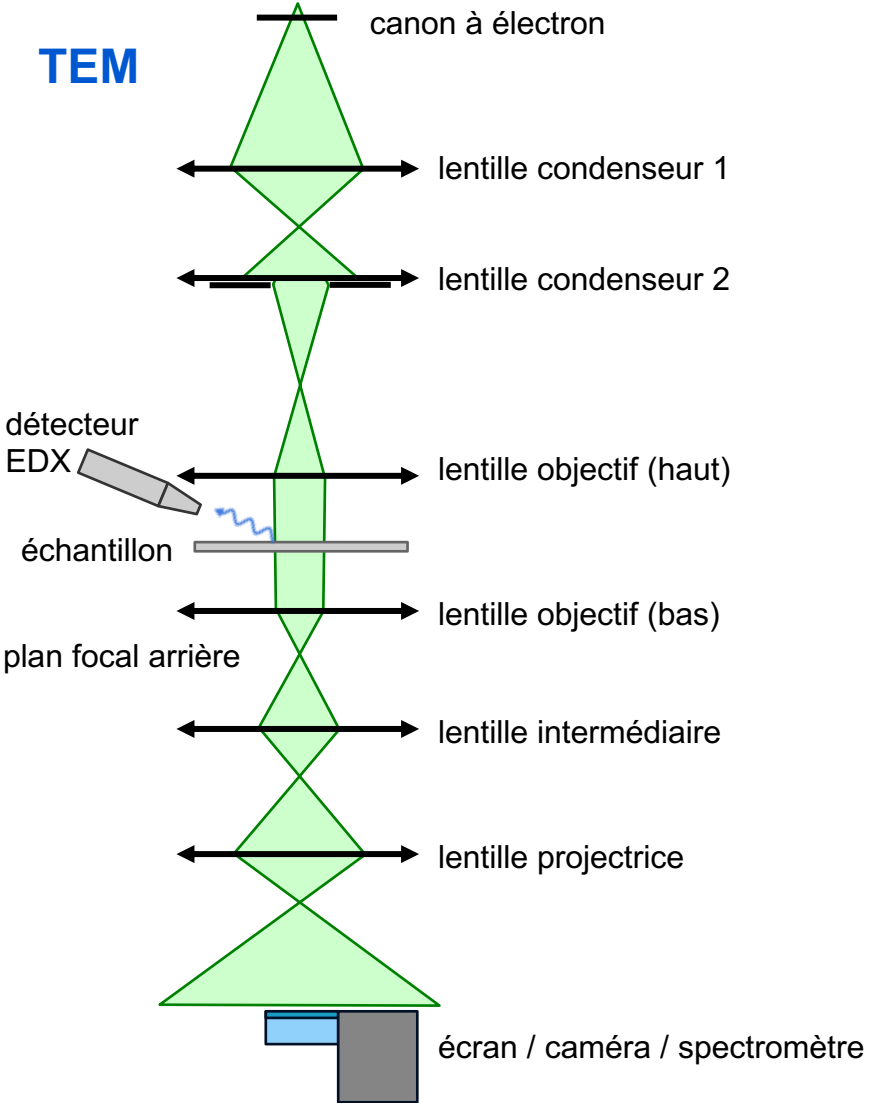


Image et Diffraction

chaque famille de plans cristallins donnent une tâche dans le plan focal arrière de la lentille objectif

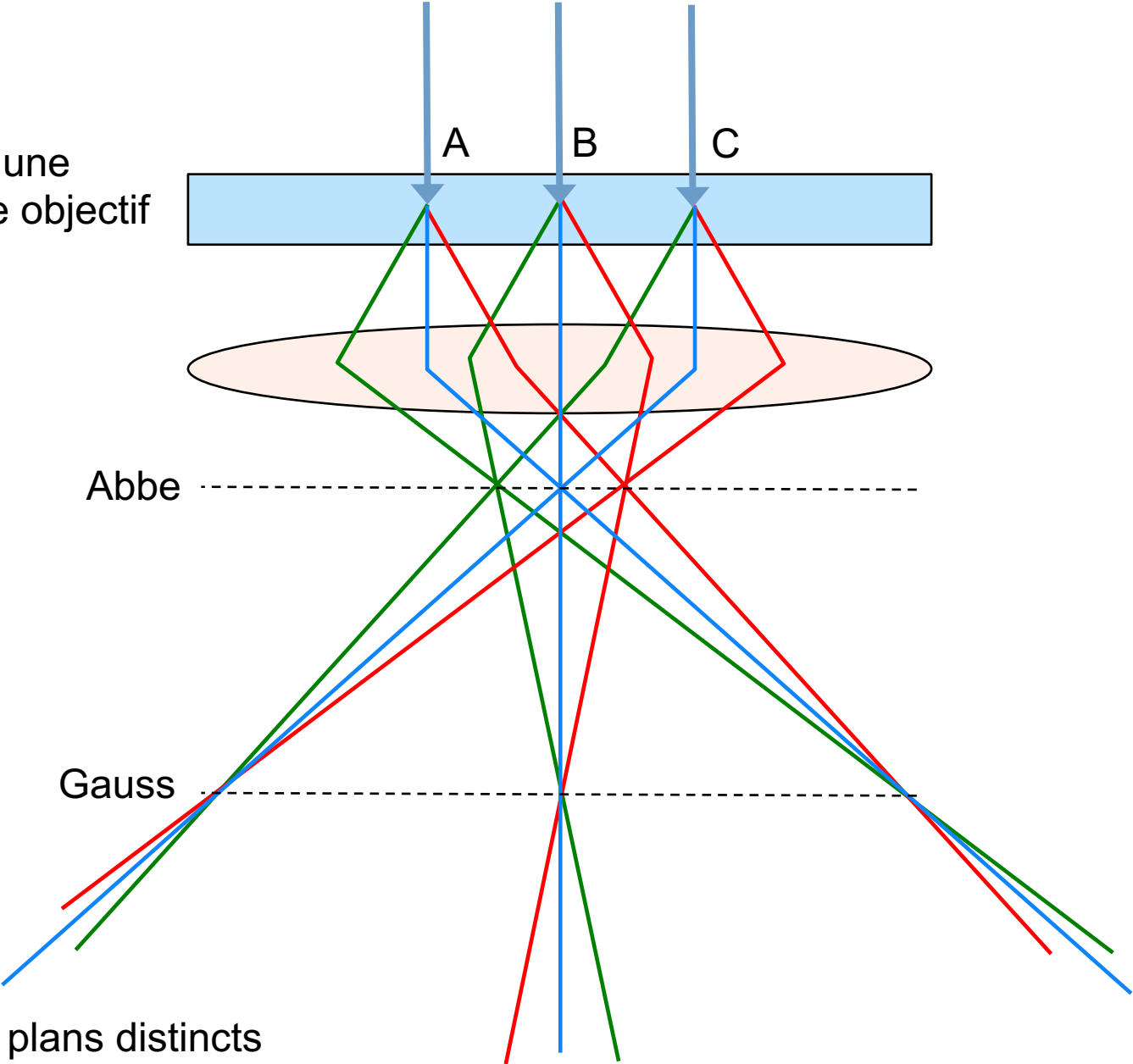
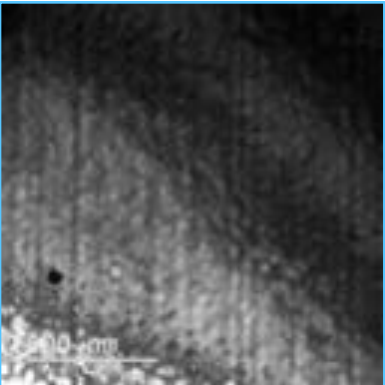
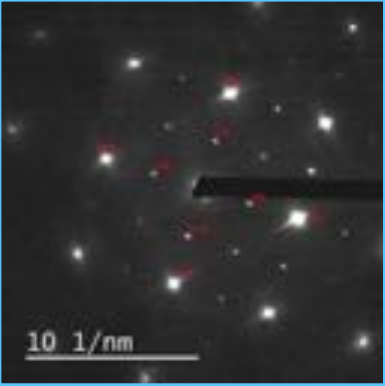


Image et diffraction sont formées dans des plans distincts

Image et Diffraction

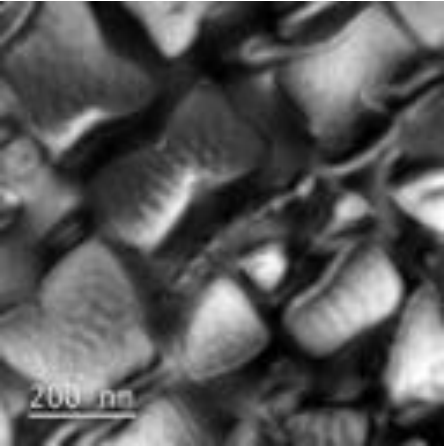
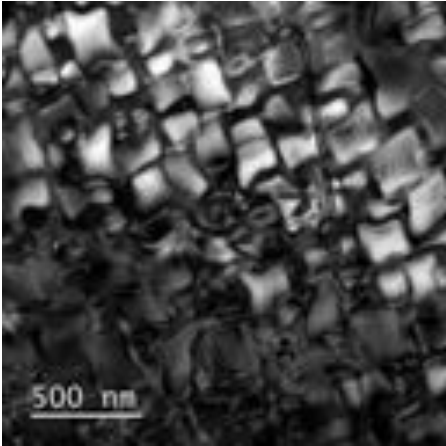
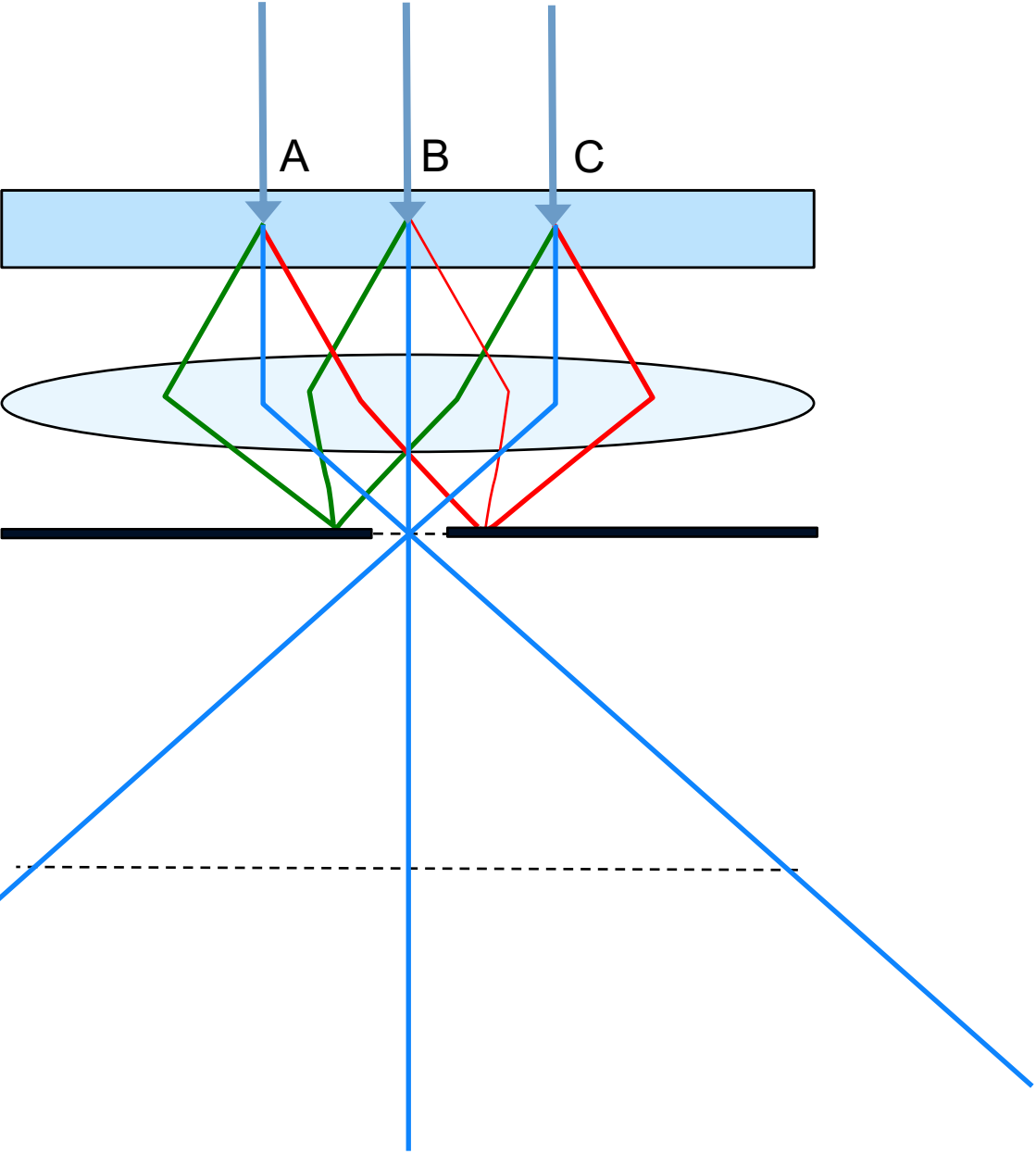
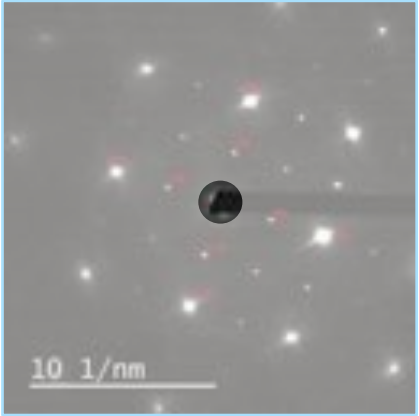


Image et Diffraction

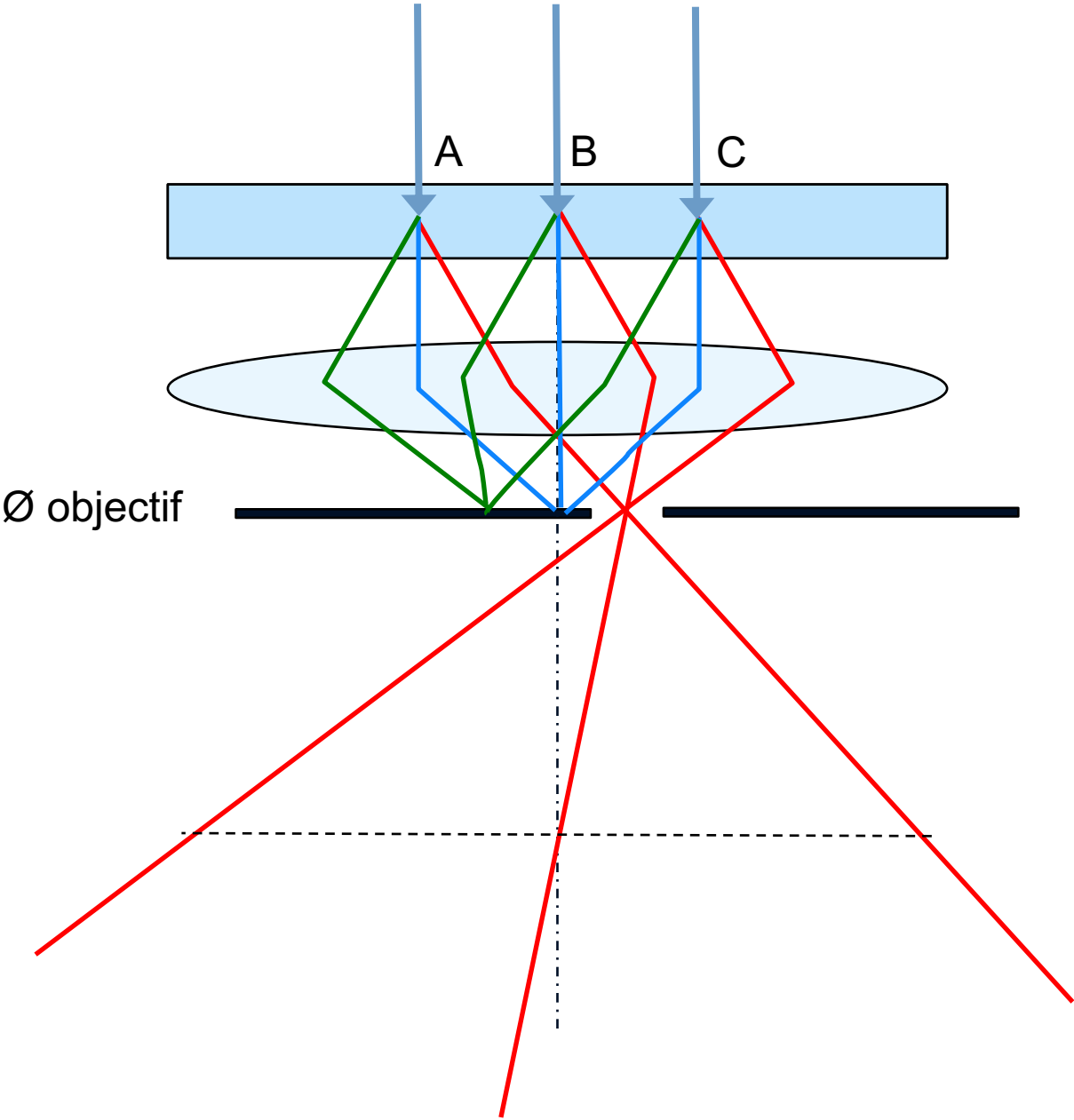
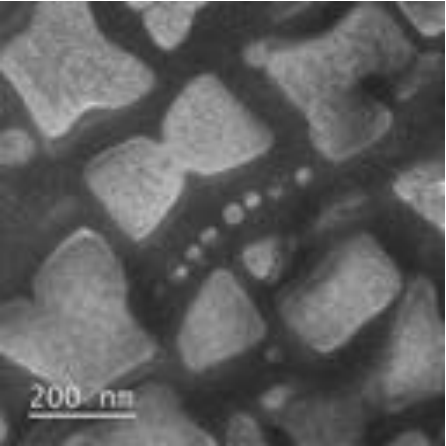
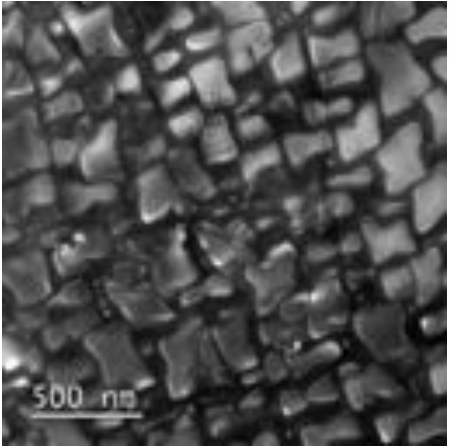
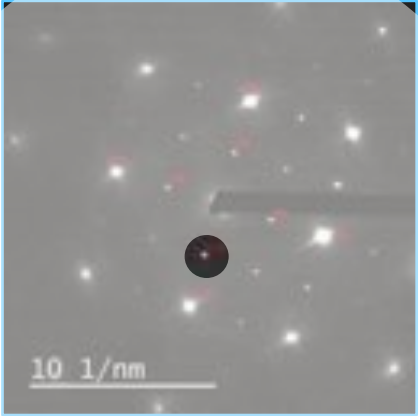
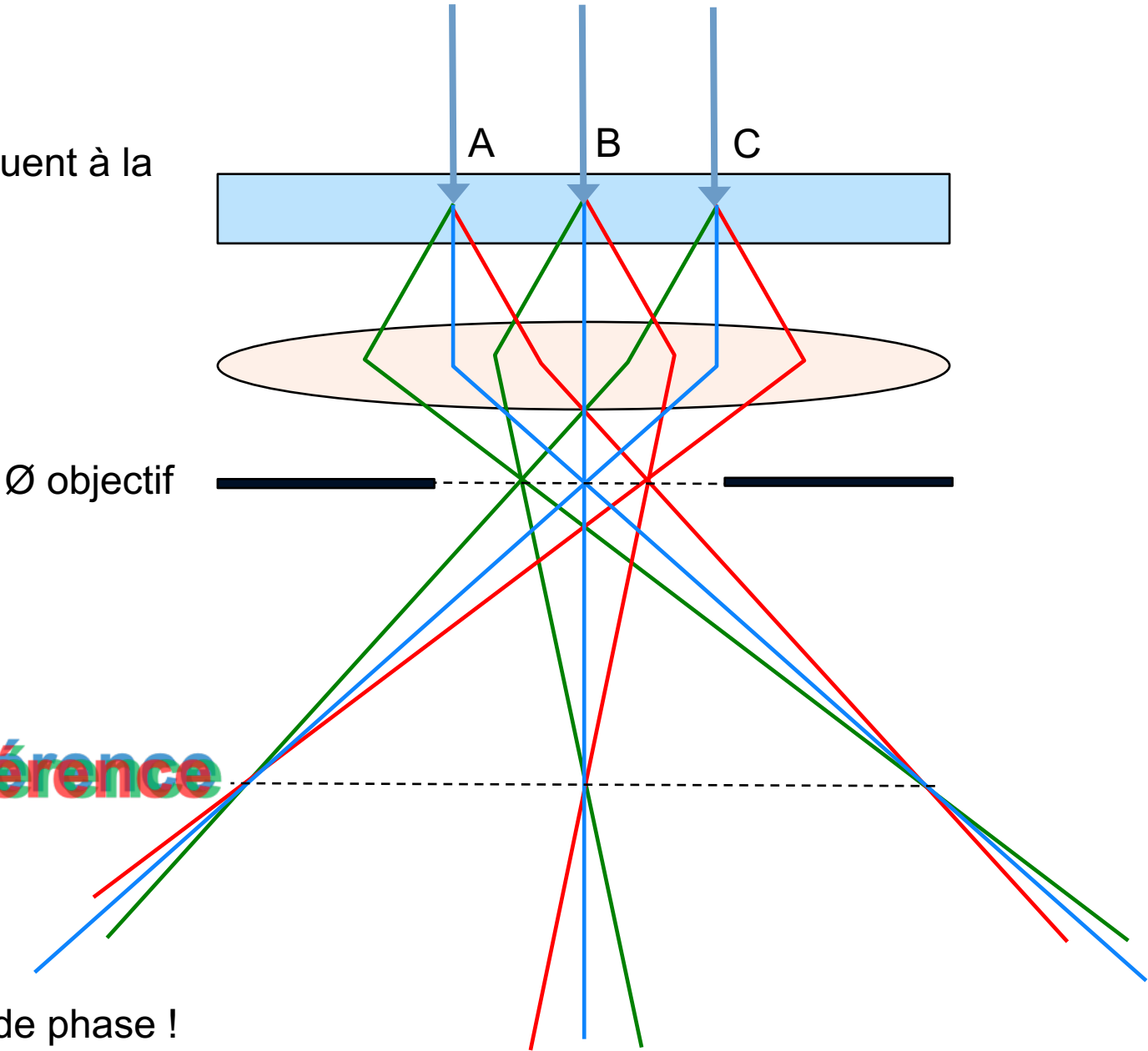
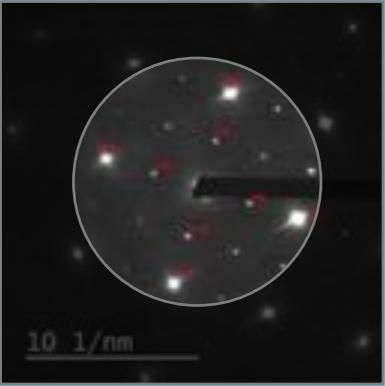


Image de phase

chaque famille de plans cristallins contribue à la formation d'une image interférentielle

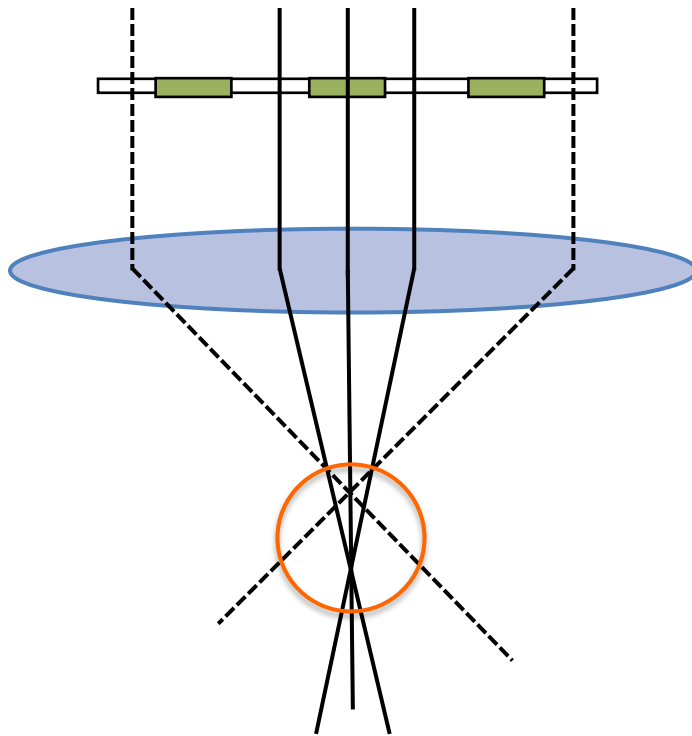
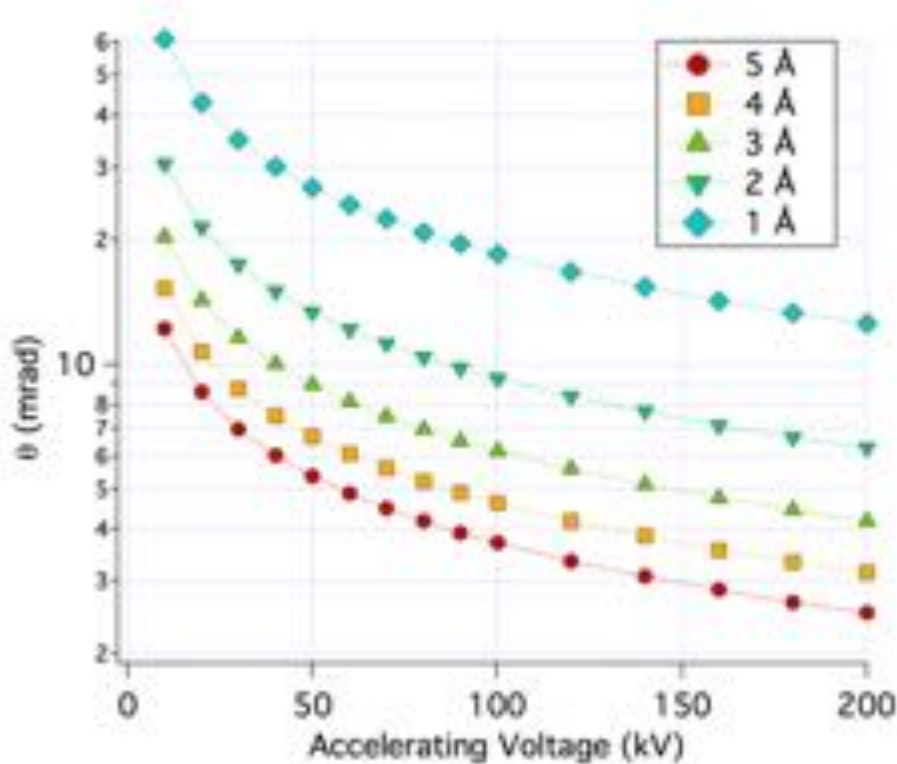


L'image résultante contient un contraste de phase !

Aberration sphérique

$$n\lambda = 2d \sin \theta$$

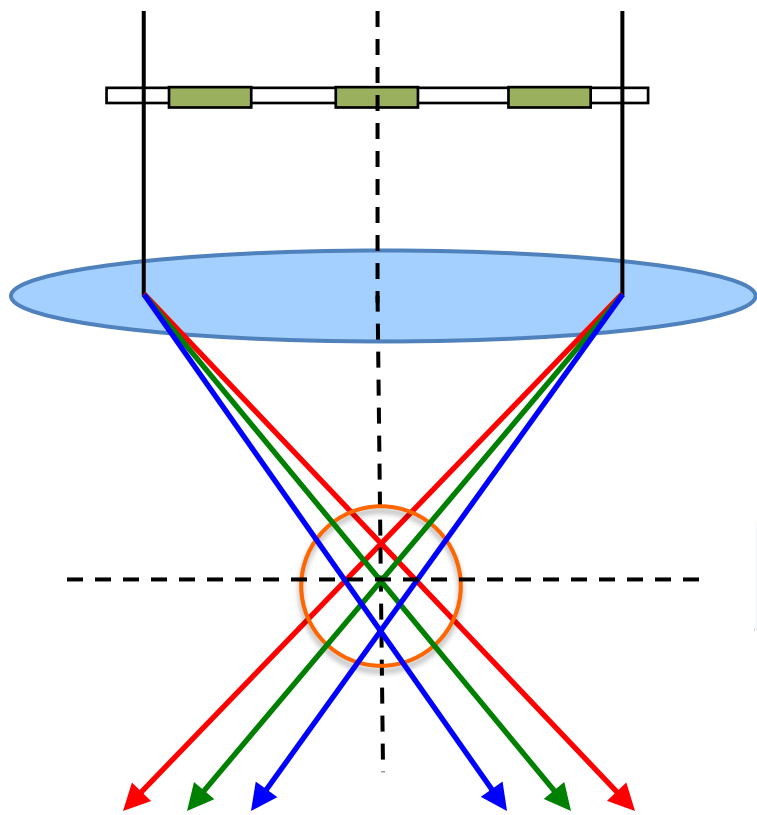
loi de Bragg



$$D_{res} = 0.66 \lambda^{3/4} C_S^{1/4}$$

L'aberration sphérique limite la résolution en introduisant un décalage de phase
 L'ouverture d'un TEM est d'environ 1 degré.

Aberration chromatique



$$d_c = \frac{1}{2} C_c \frac{dE}{E} \theta$$

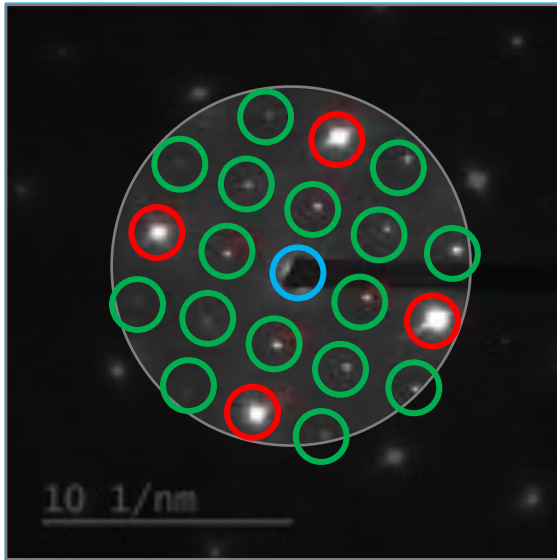
Aberration chromatique C_c

source	W	LaB6	FEG	Cold FEG	mono
dispersion énergétique (eV)	3	1.5	0.7	0.4	< 0.1

Impact plus important de ce paramètre en cas :

- **d'élargissement spectral important (EFTEM)**
- **de basses tensions (E faible)**

Image de phase



Limite de fréquence spatiale acceptée

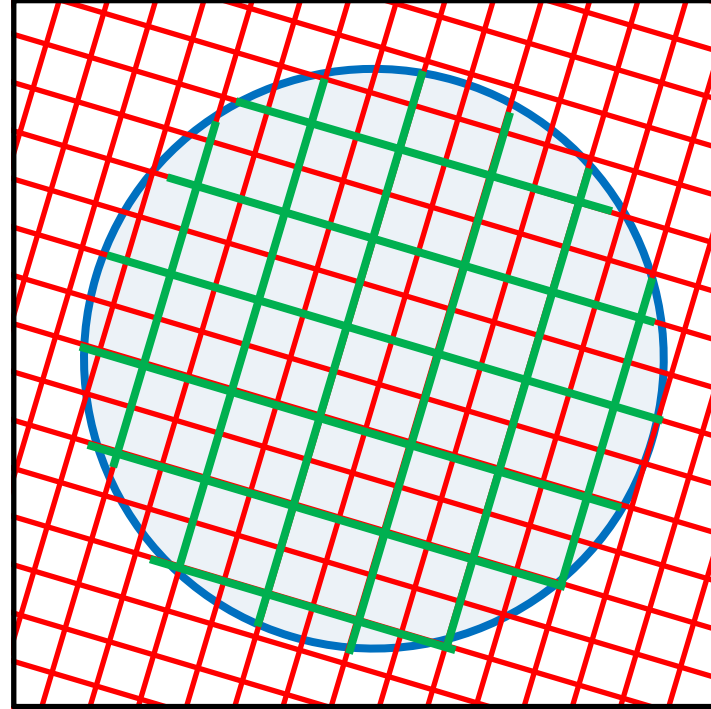
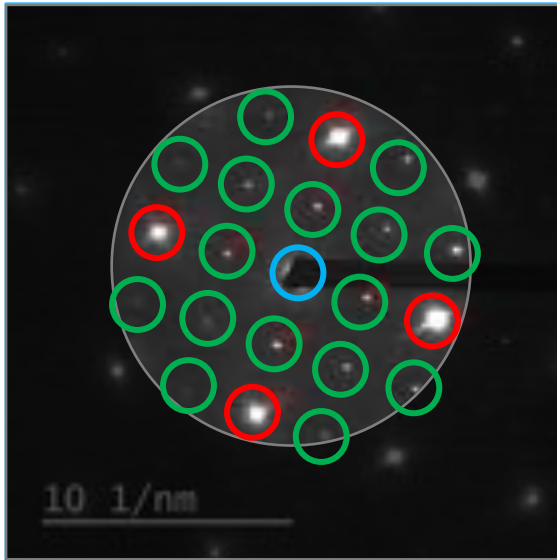
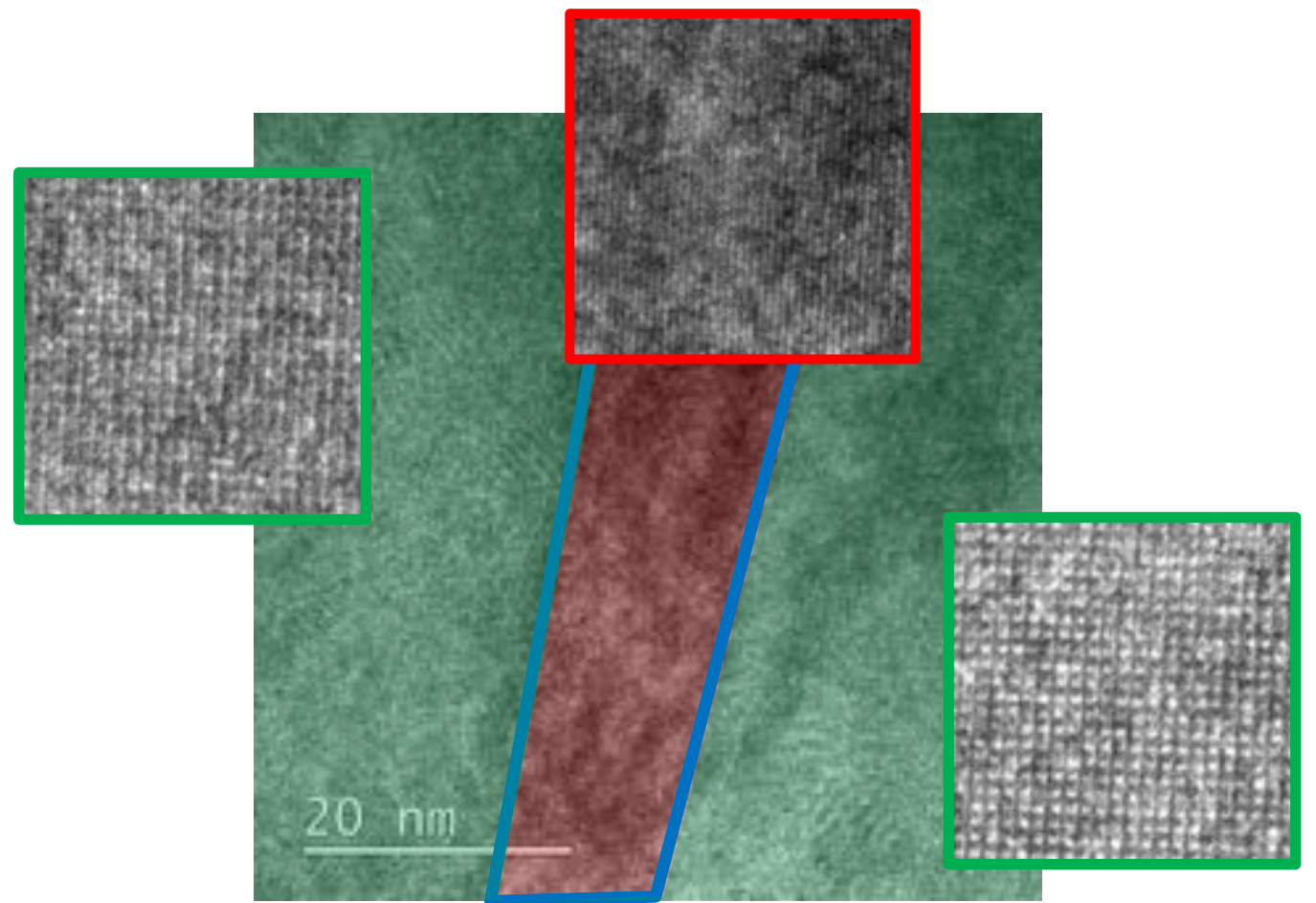


Image de phase

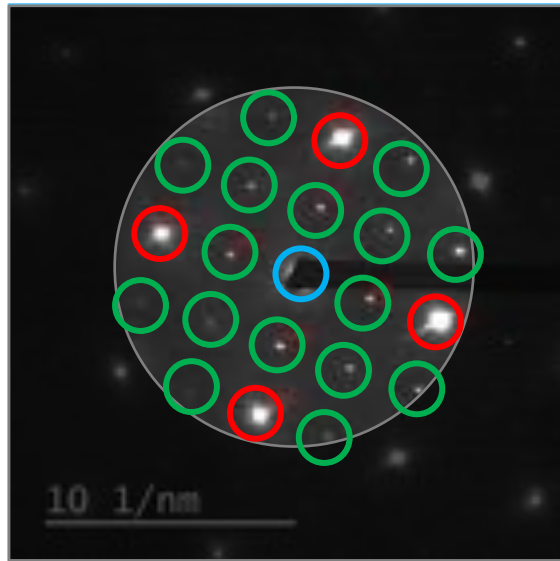


Limite de fréquence spatiale acceptée

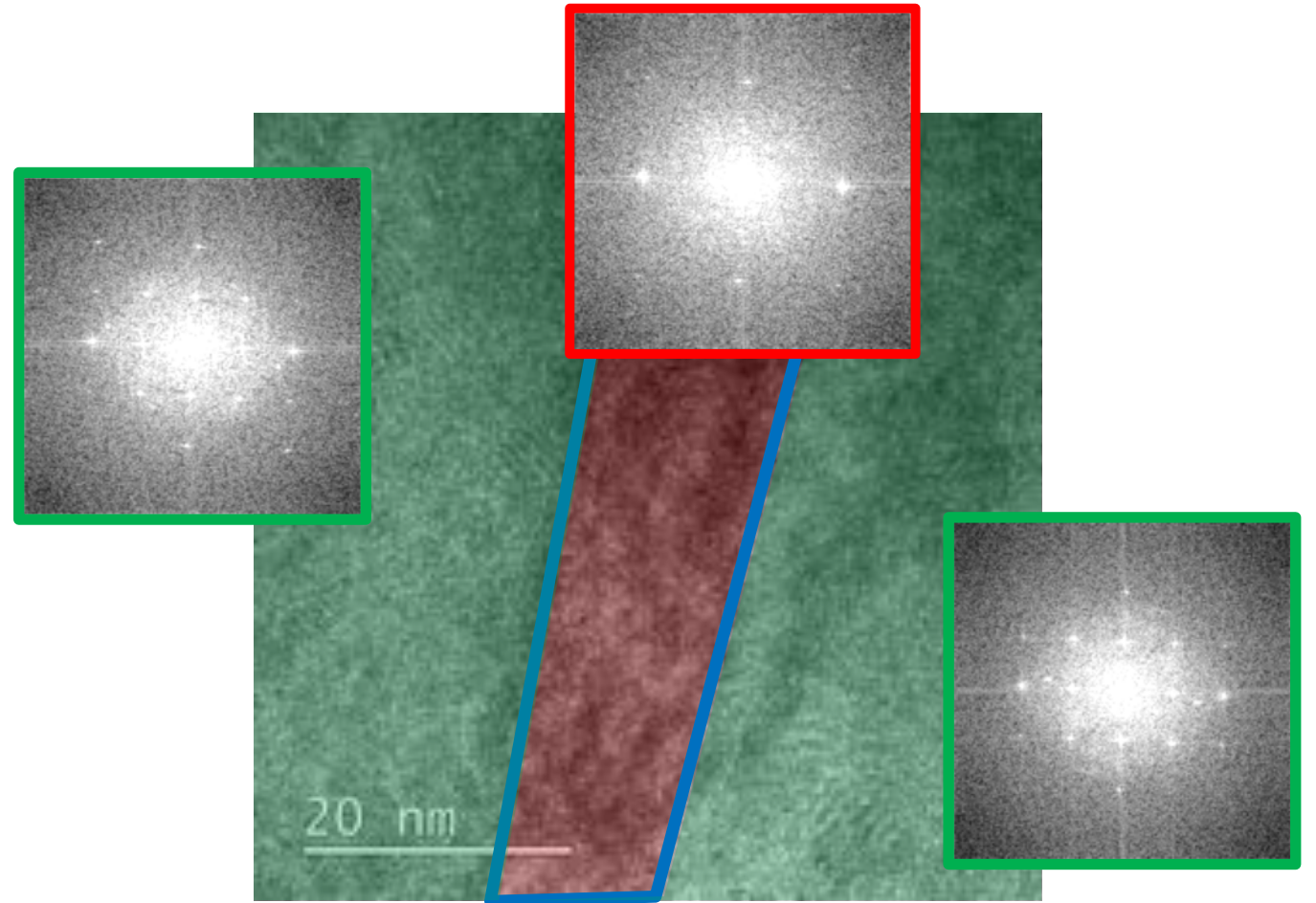


Dans un MET standard $d = 2.3 \text{ \AA}$

Image de phase

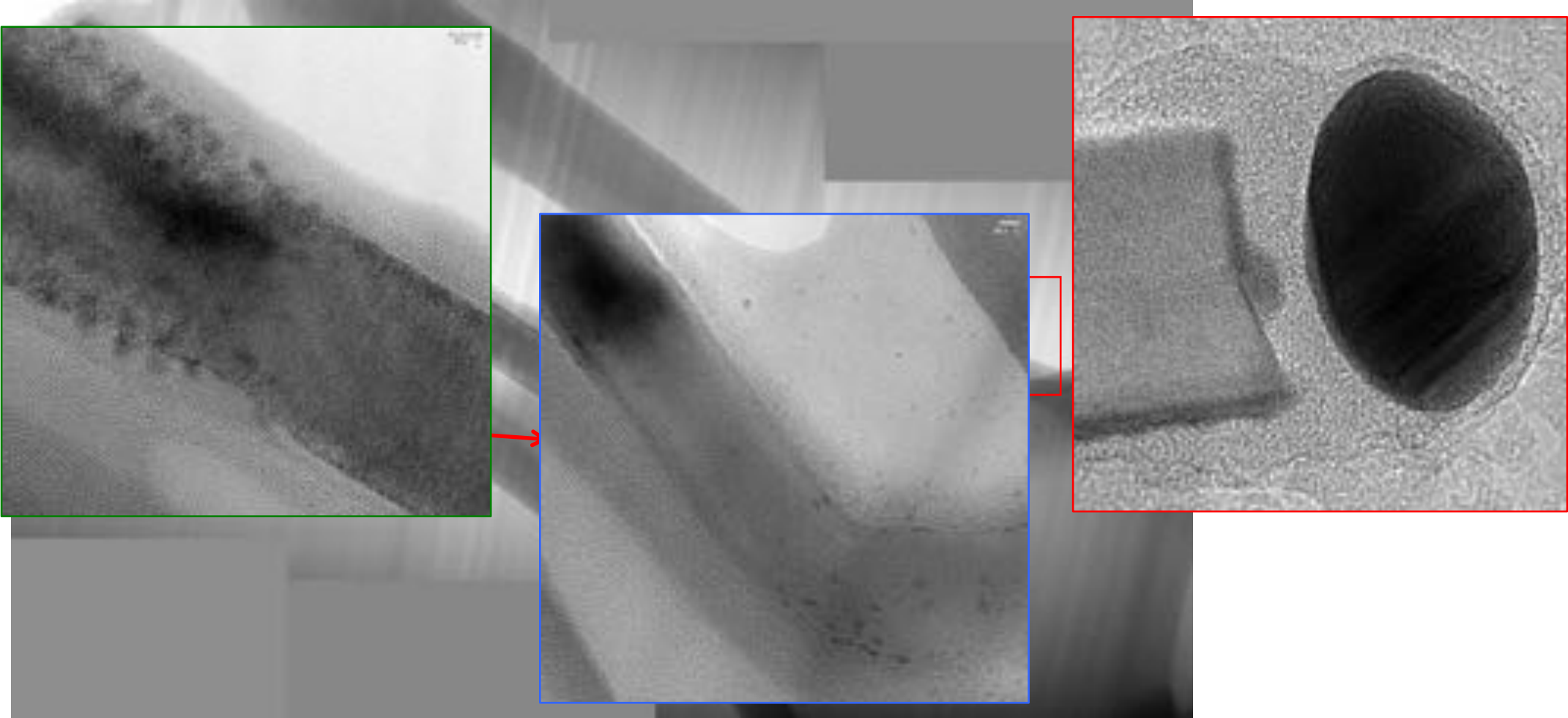


Limite de fréquence spatiale acceptée

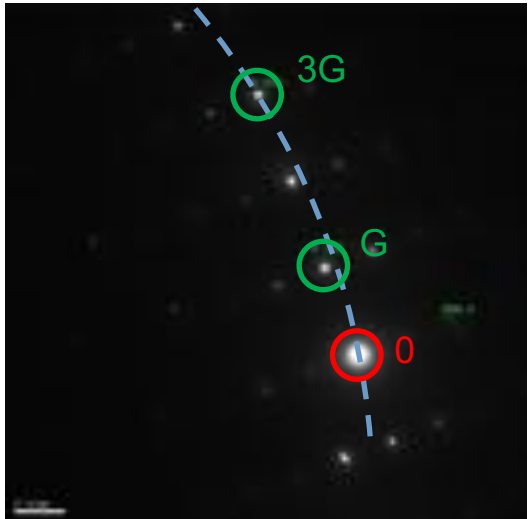
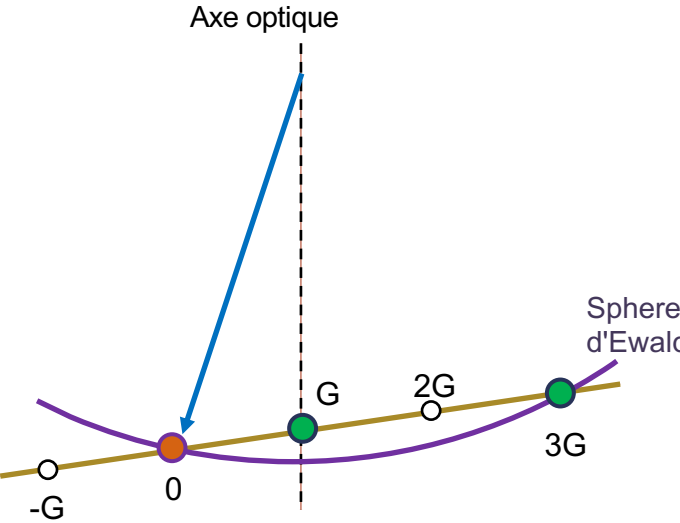
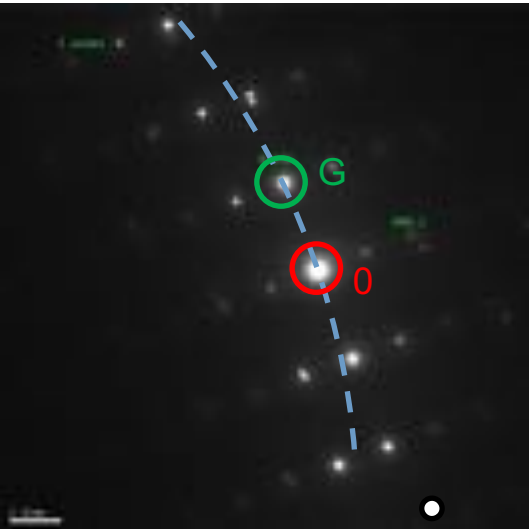
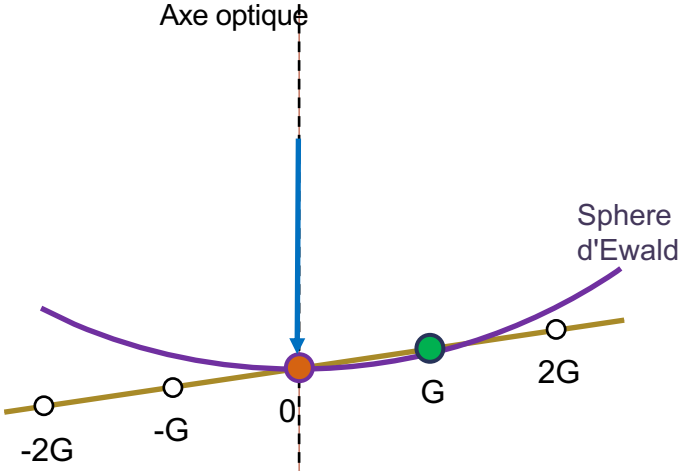


Dans un MET standard $d = 2.3 \text{ \AA}$

Imagerie cohérente

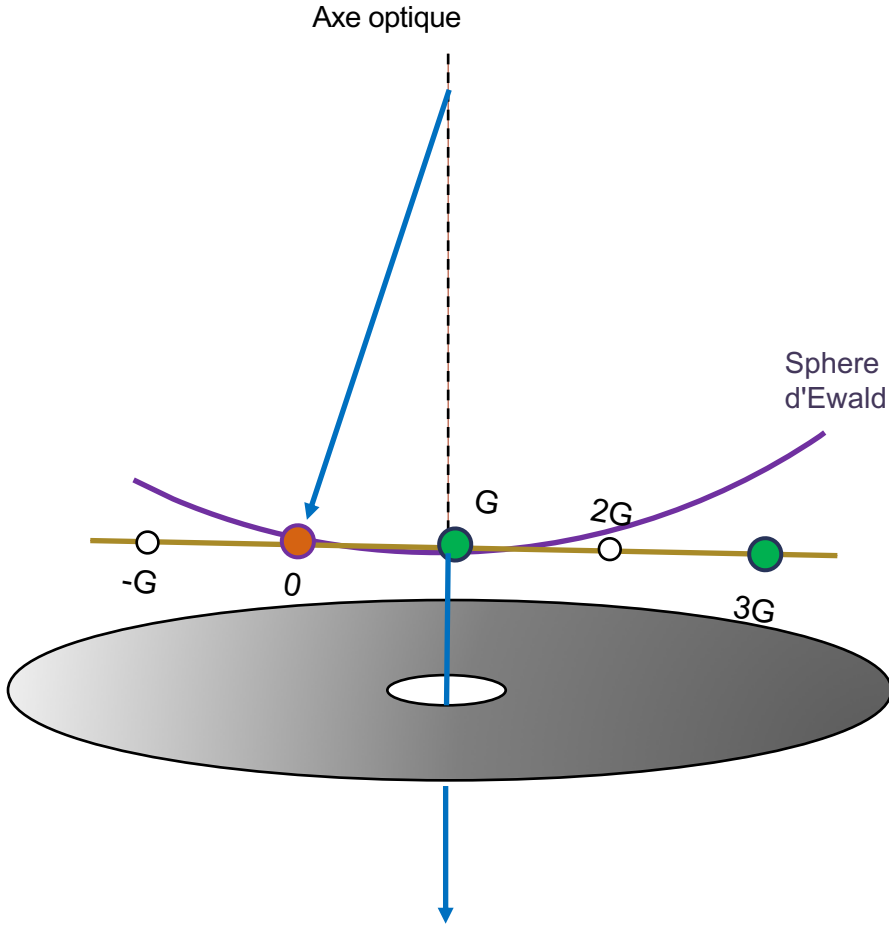
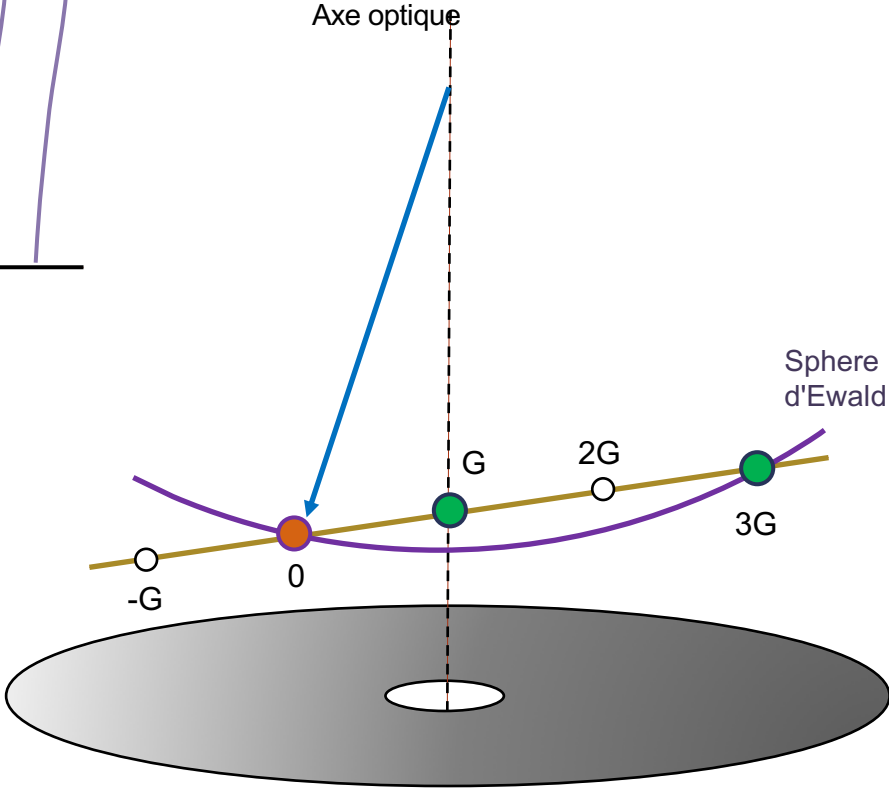
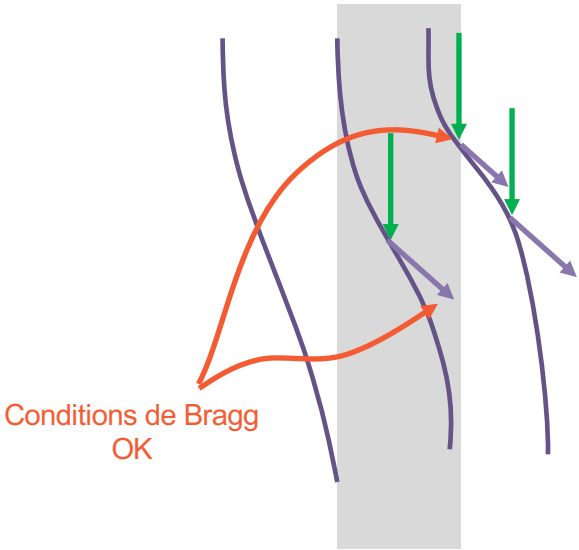
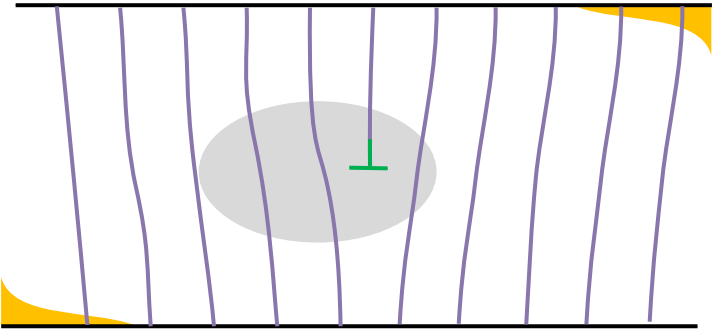


Voir les dislocations

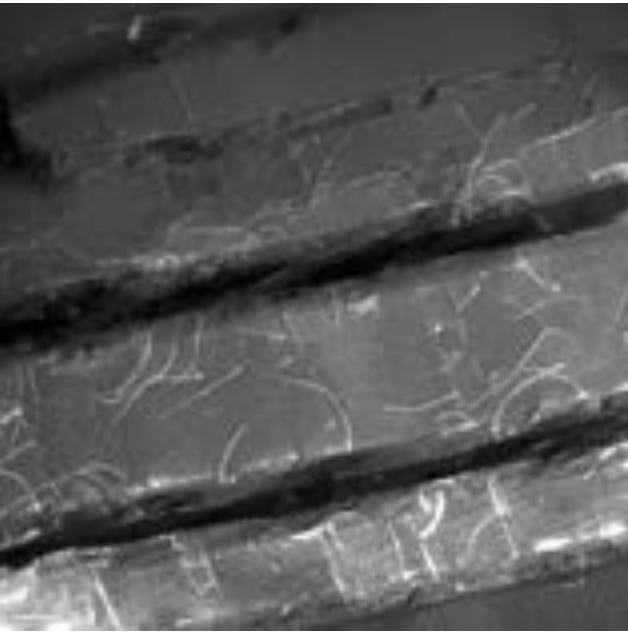
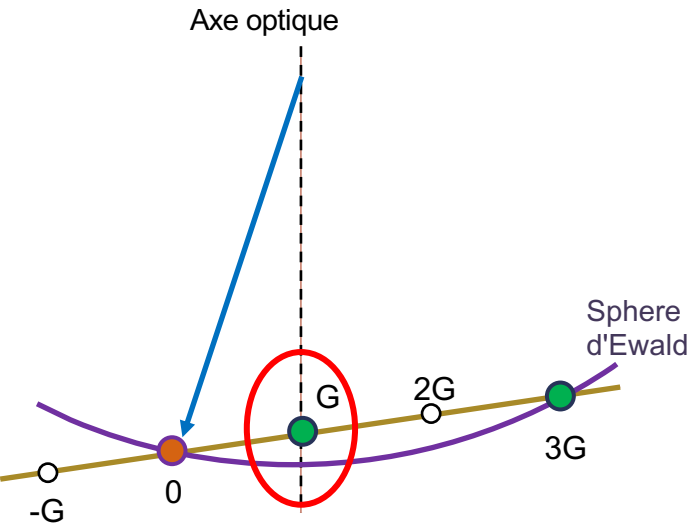
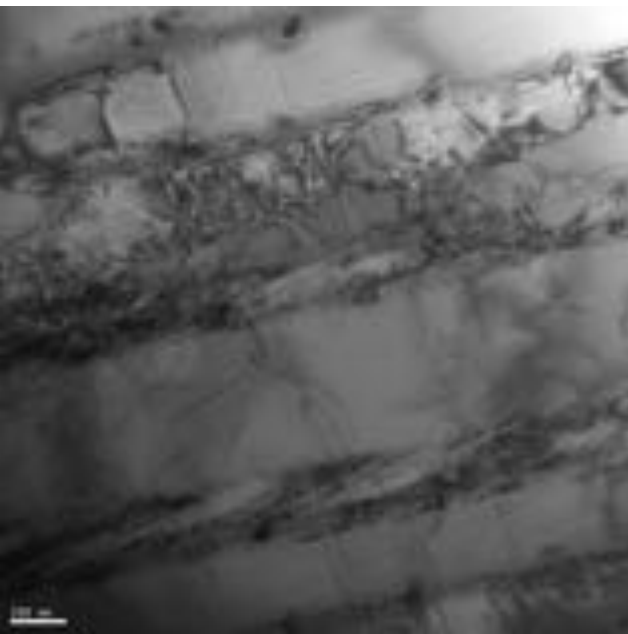
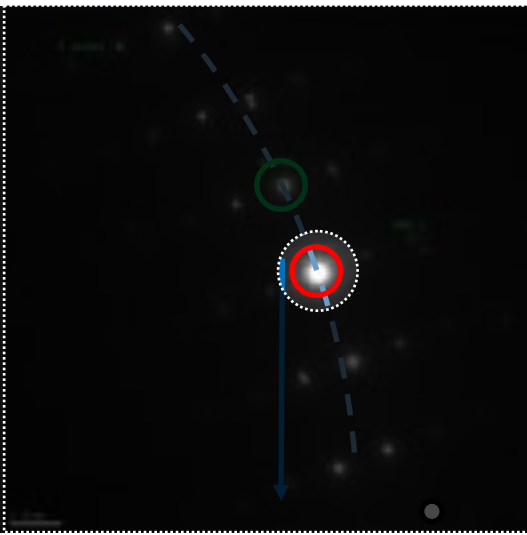
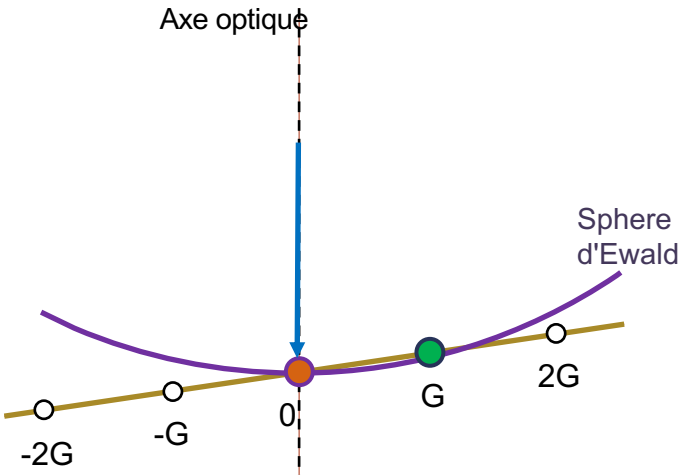


Champ sombre en faisceau faible (WBDF)

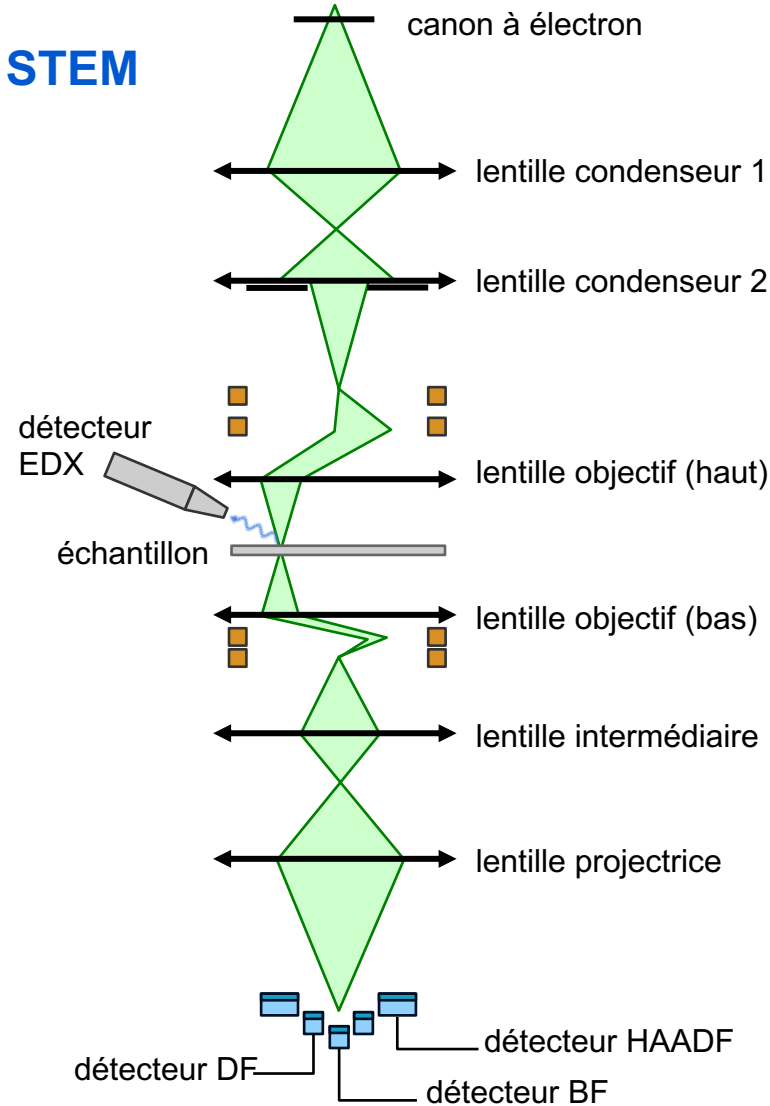
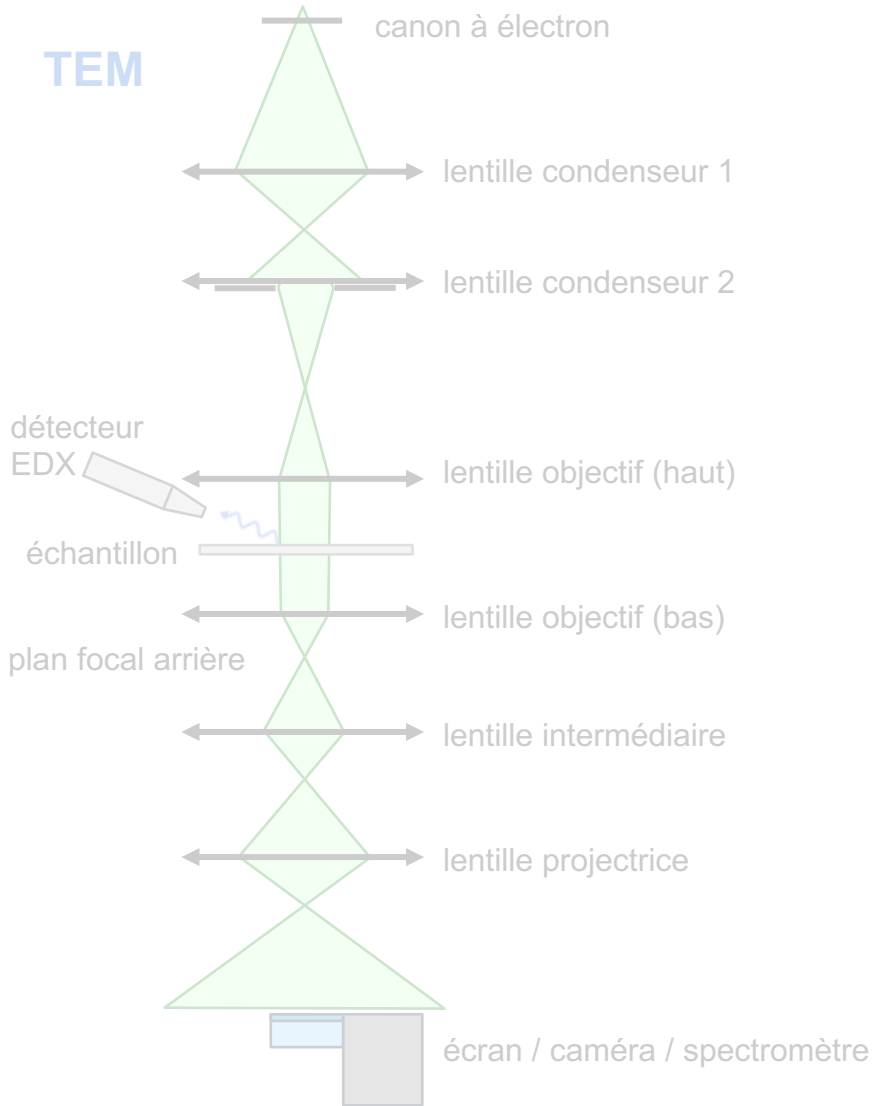
Utiliser la distorsion au cœur de la dislocation pour l'imager



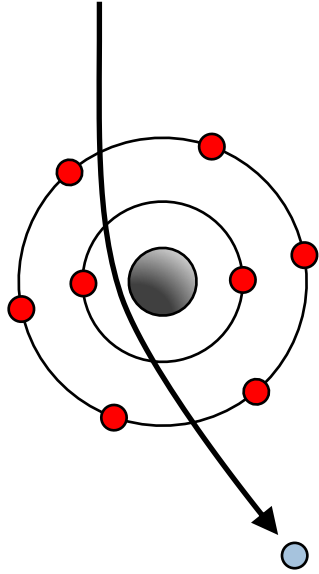
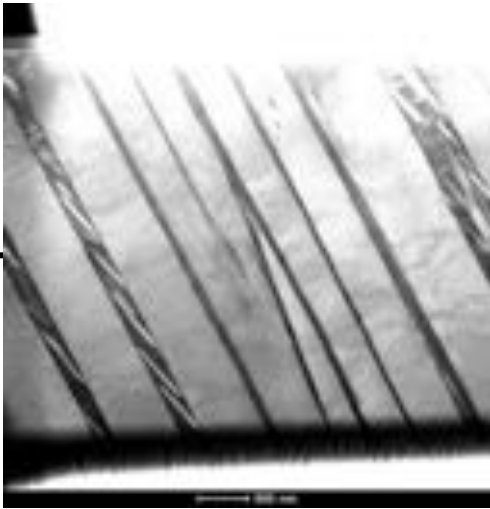
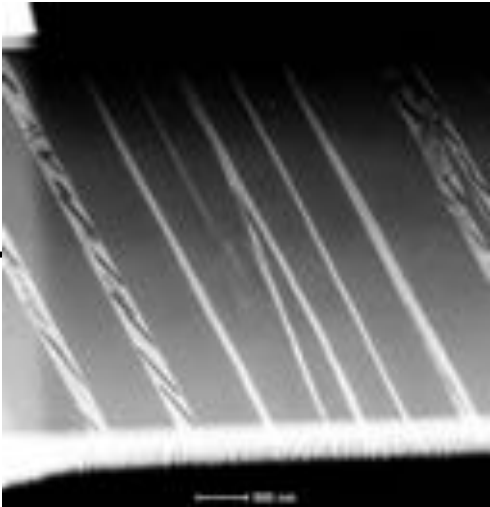
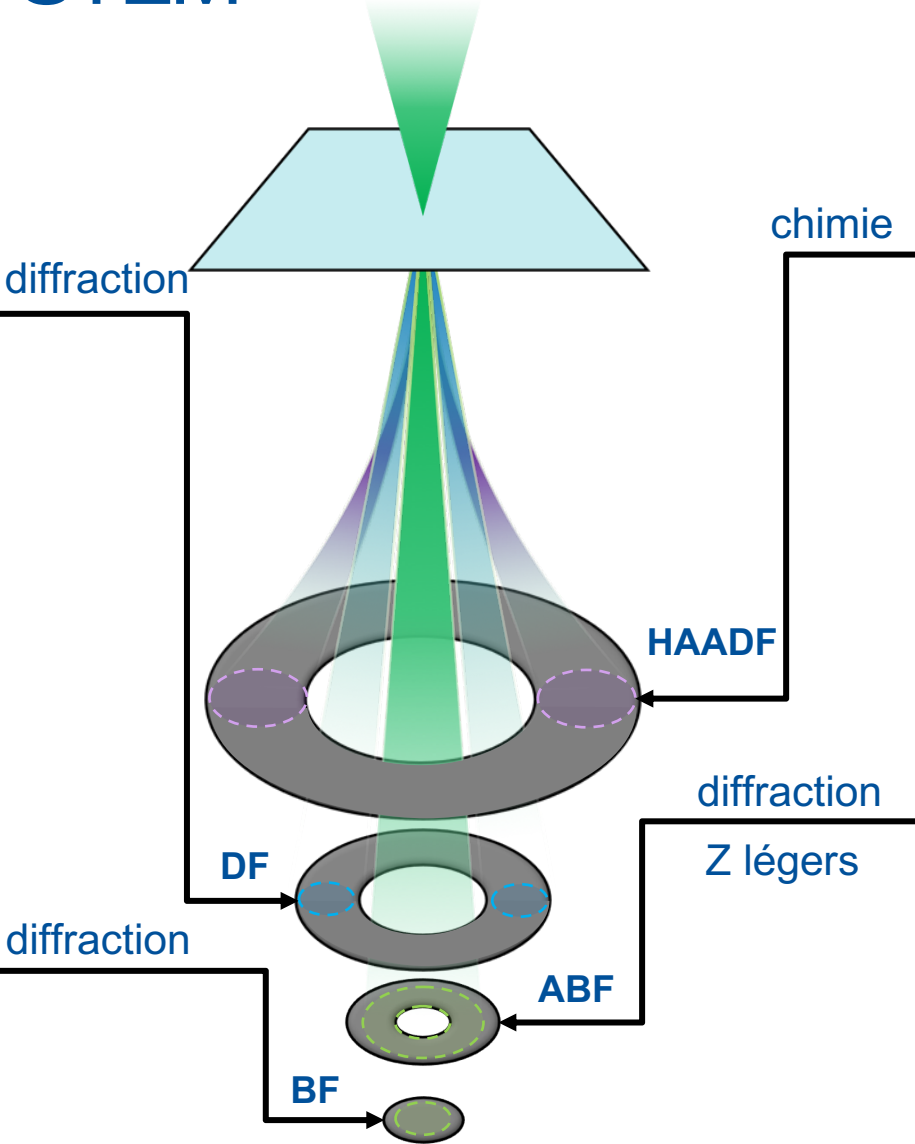
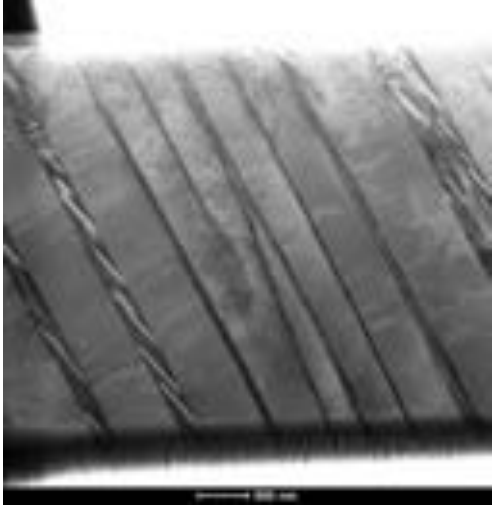
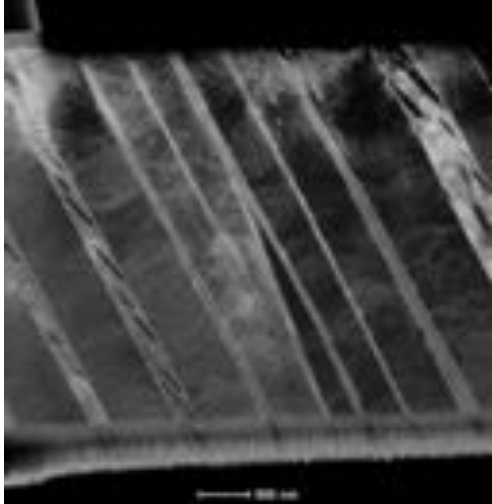
Voir les dislocations



2 modes de fonctionnement



Contrastes en STEM



electronic diffusion factor

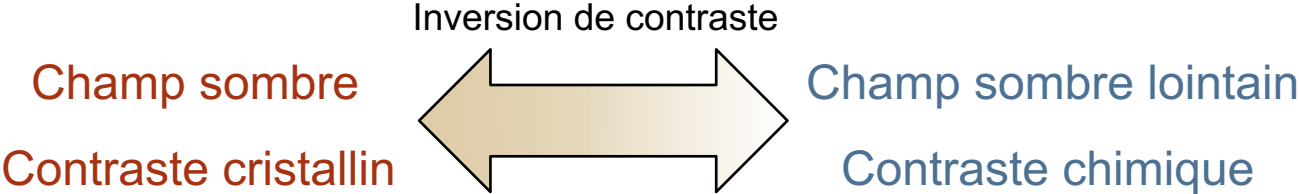
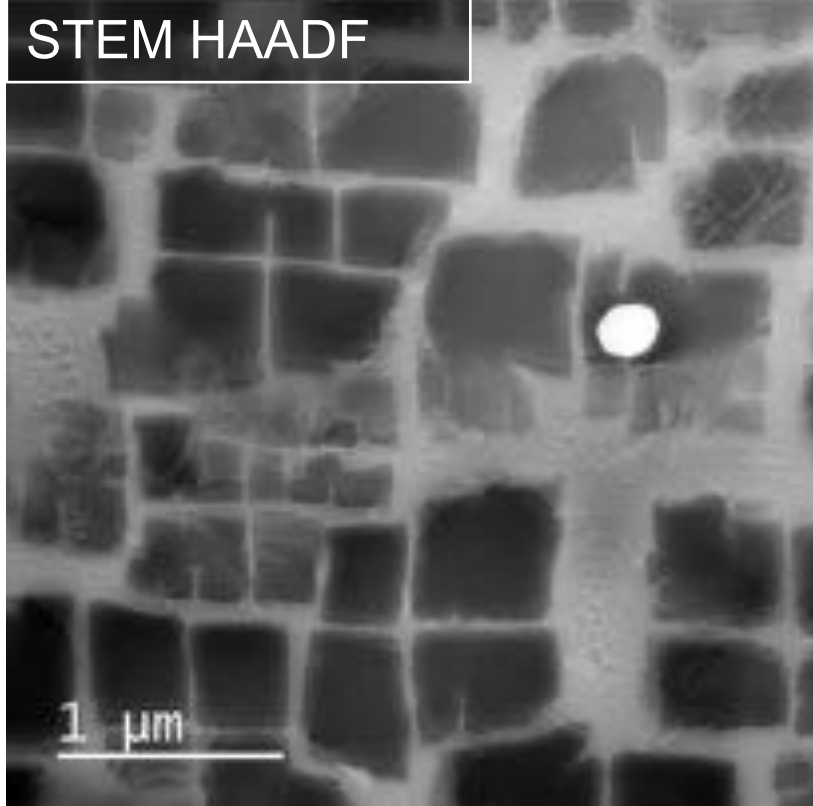
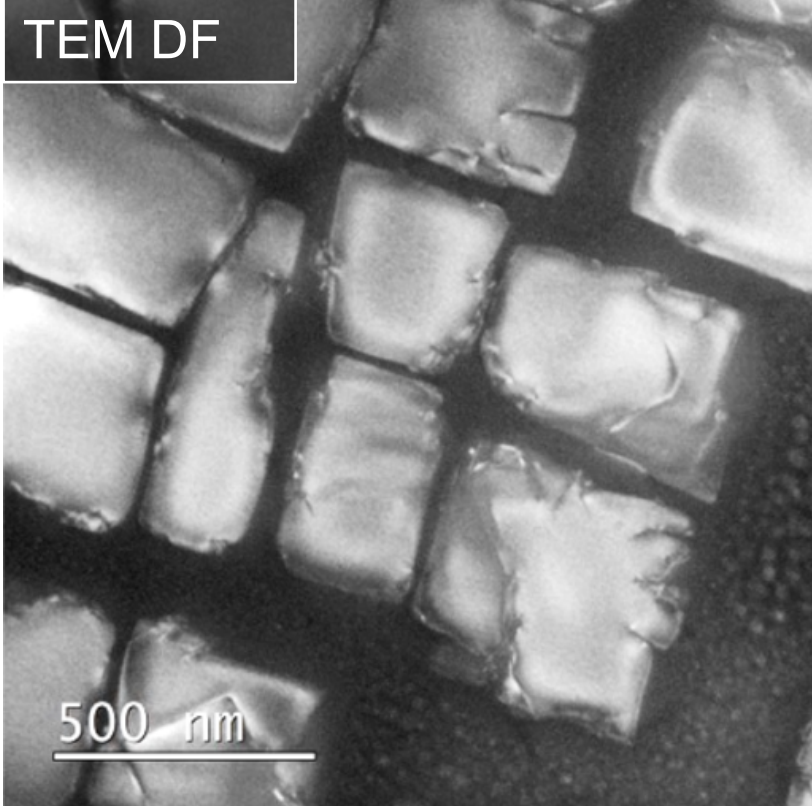
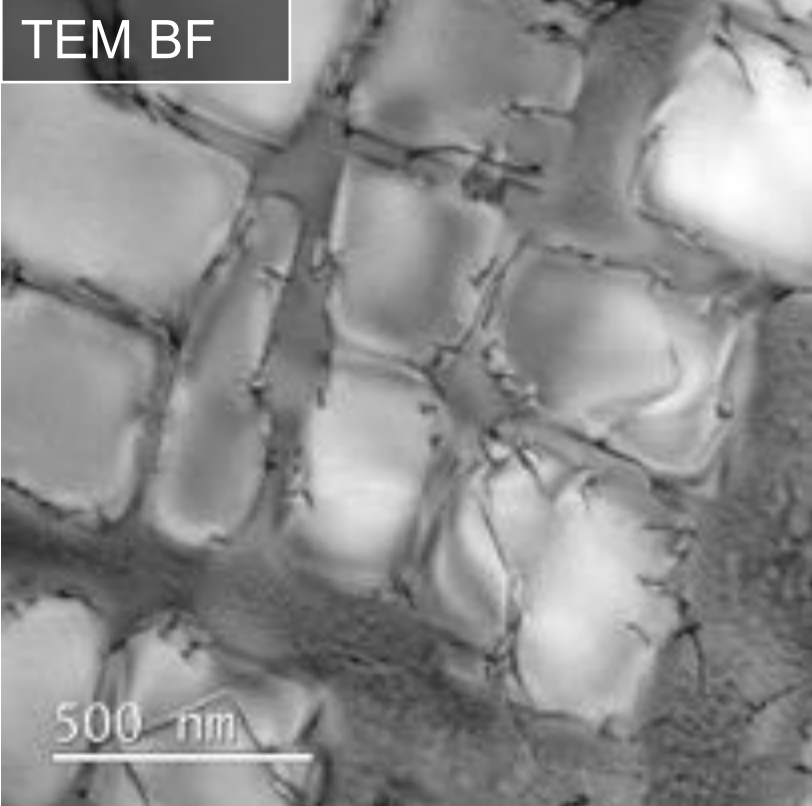
$$f_e(q) = 1 \frac{1}{4\pi\epsilon_0} \times \left(\frac{\lambda}{\sin\theta}\right)^2 [Z - f_x(q)]$$

but screening in $Z^{-1/3}$

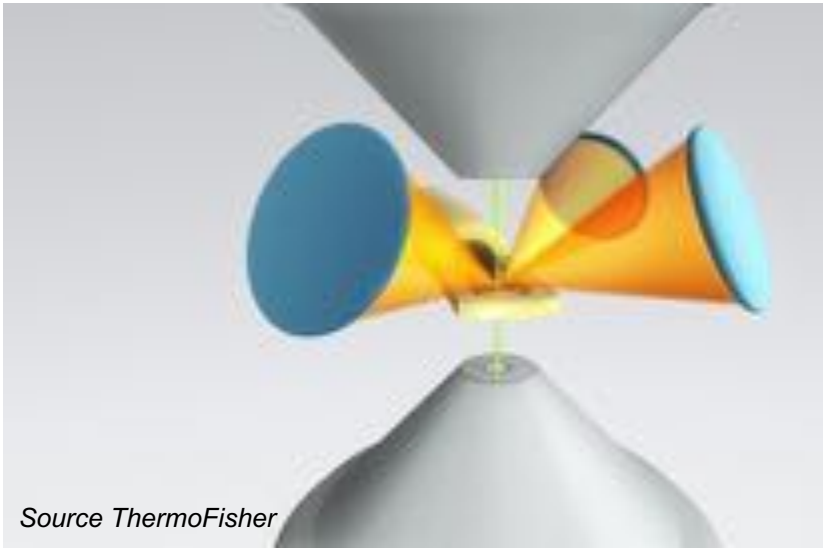
$$Z^{4/3} \leq I \leq Z^2$$

Imagerie cohérente vs imagerie incohérente

microstructure γ/γ' dans un alliage base nickel

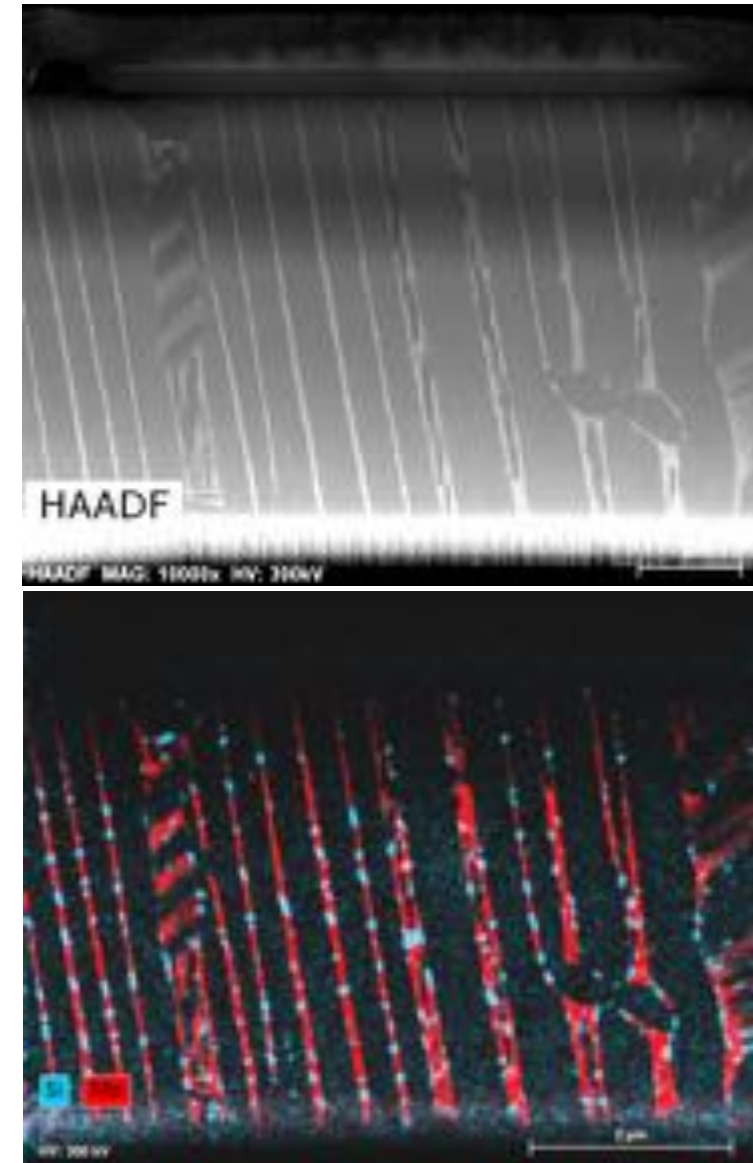
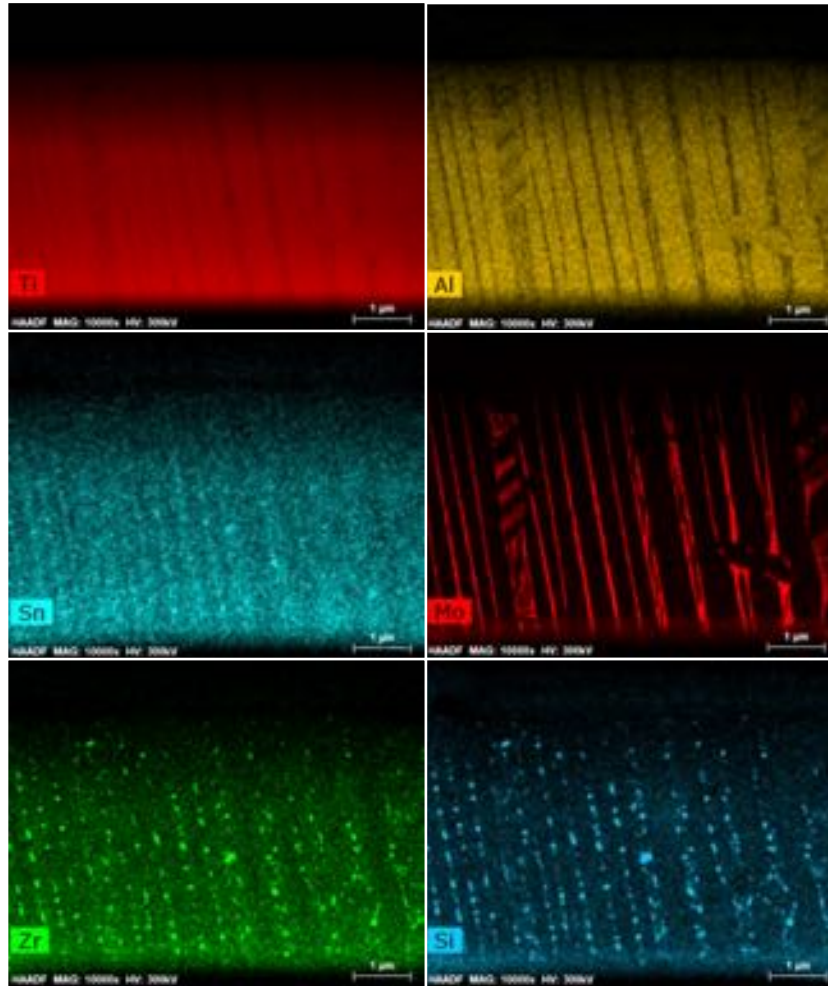


EDS



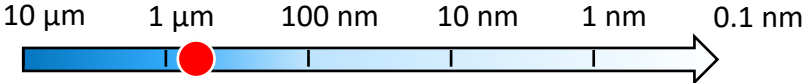
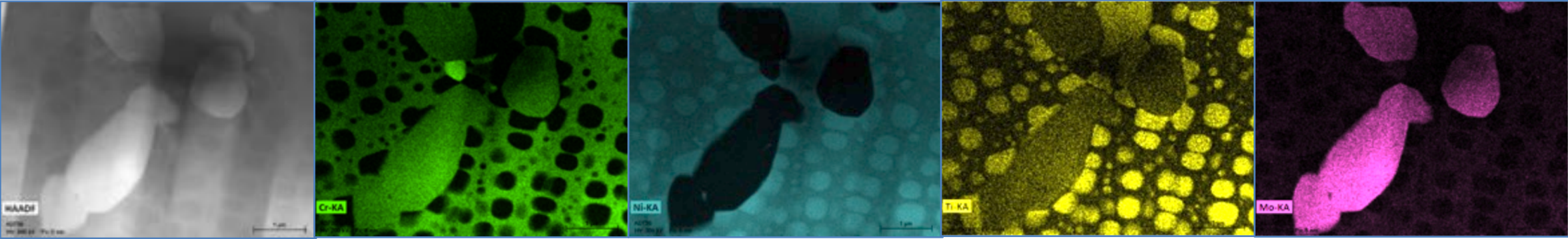
Titan G2 corrigé sonde - 400pA
LMPS - CentraleSupélec

- Mo marqueur de la phase β
- Présence de Si et Zr dans des précipités aux interfaces α/β

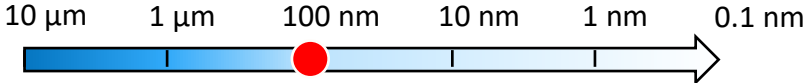
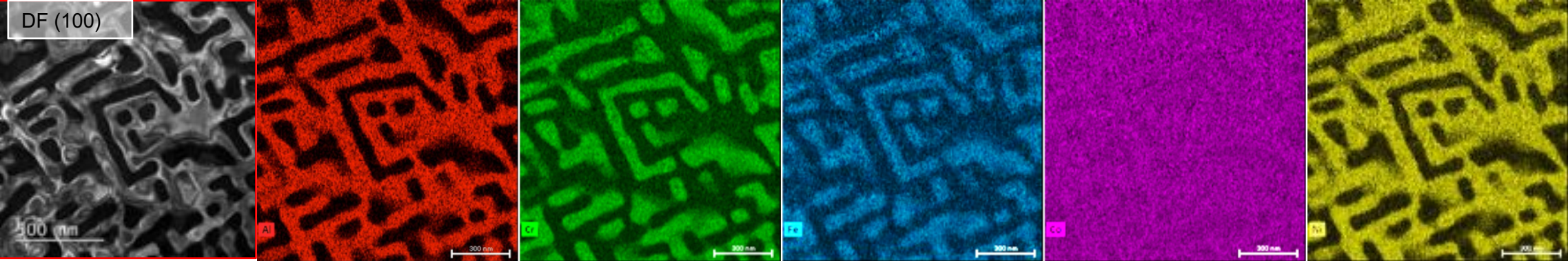


Quelles limites d'analyse !

Superaliage base-Ni

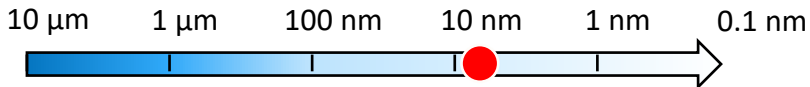
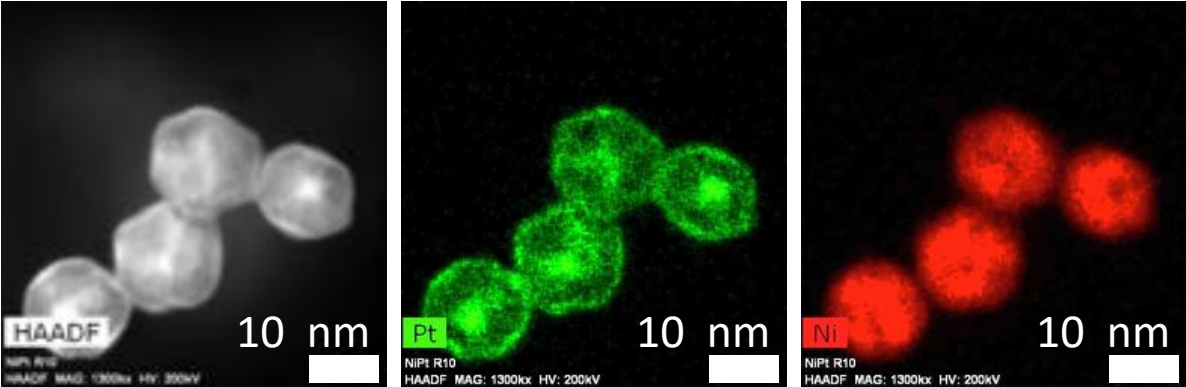


Alliage haute entropie

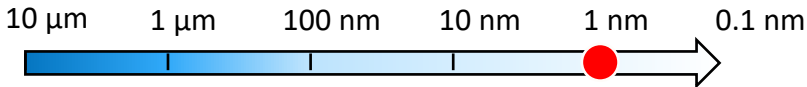
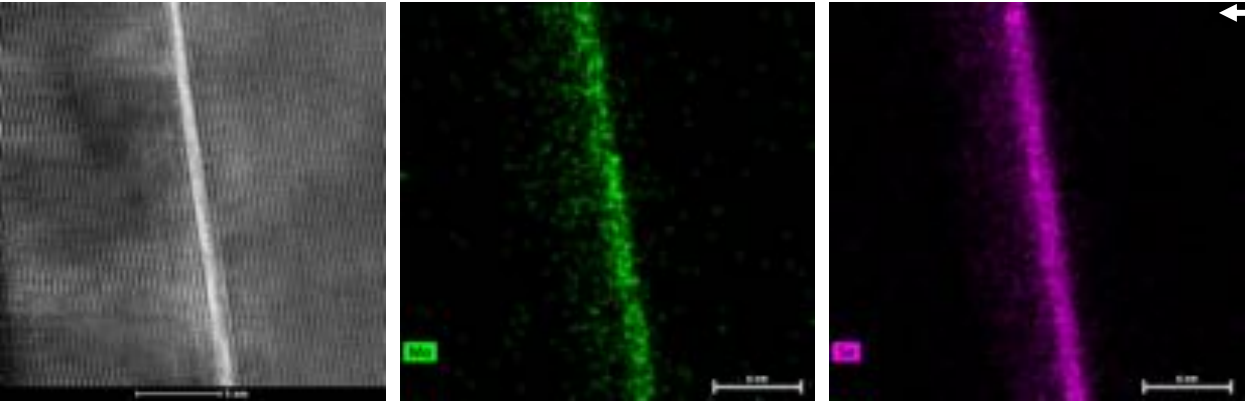


Quelles limites d'analyse !

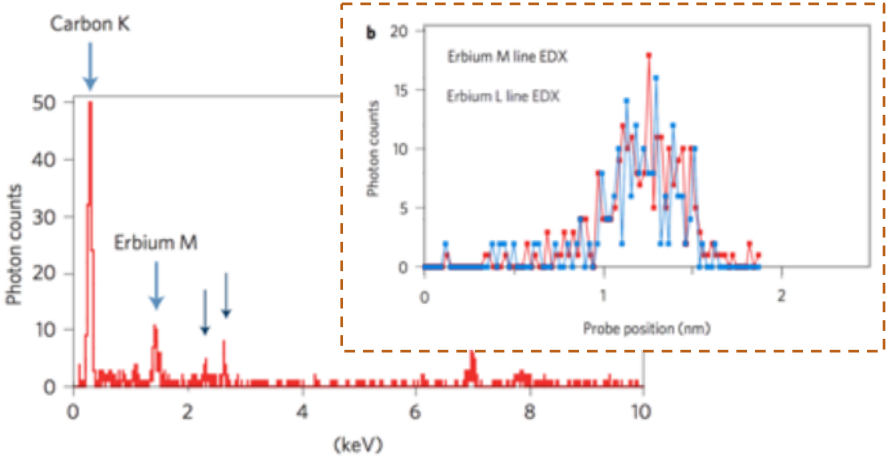
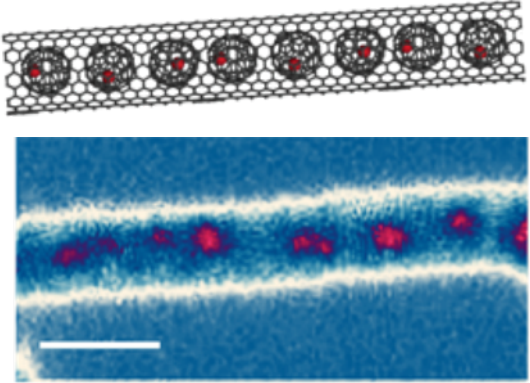
Nanoparticules NiPt



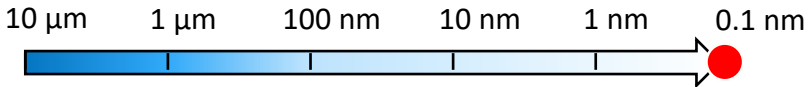
Feuillet MoSe₂ entre deux couches de BN



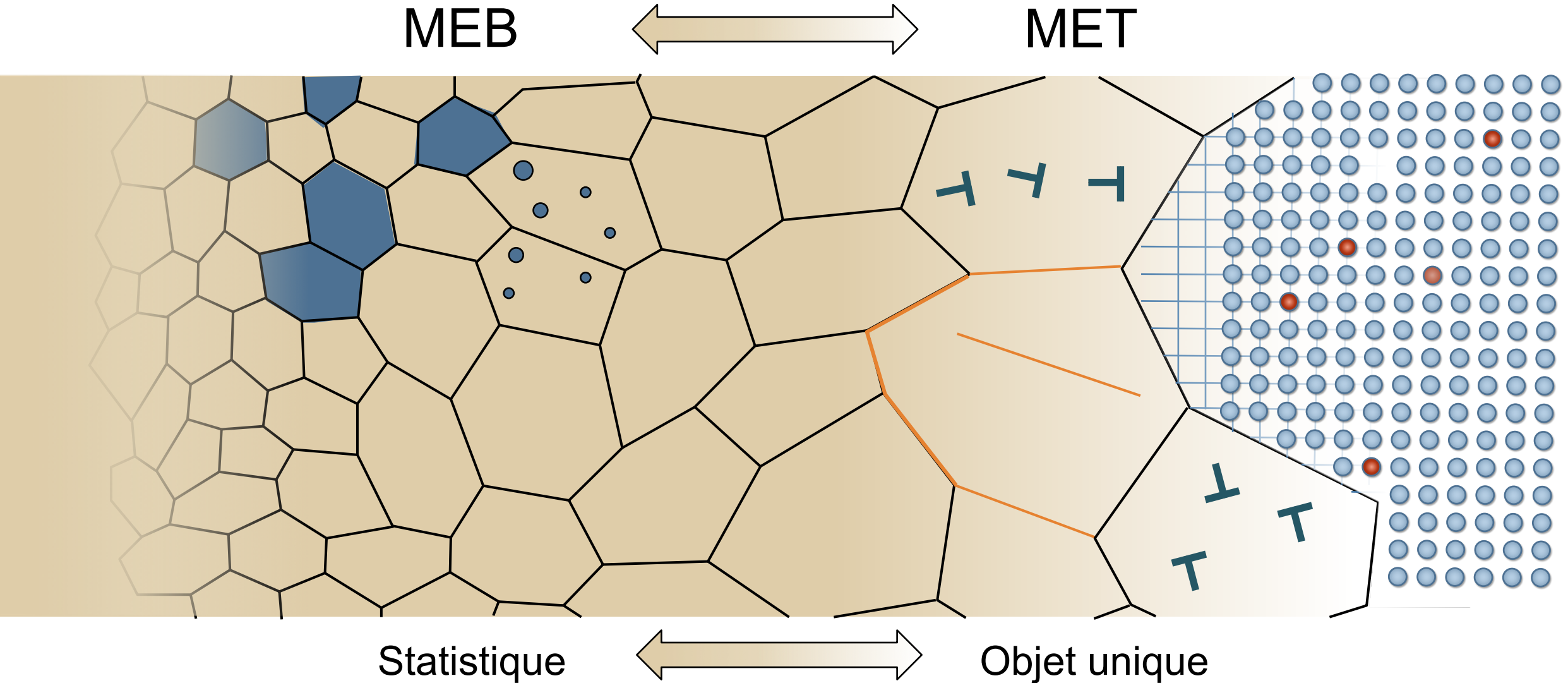
Et 1 atome ?



K. Suenaga et al., Nature Photon 6, 545 (2012)

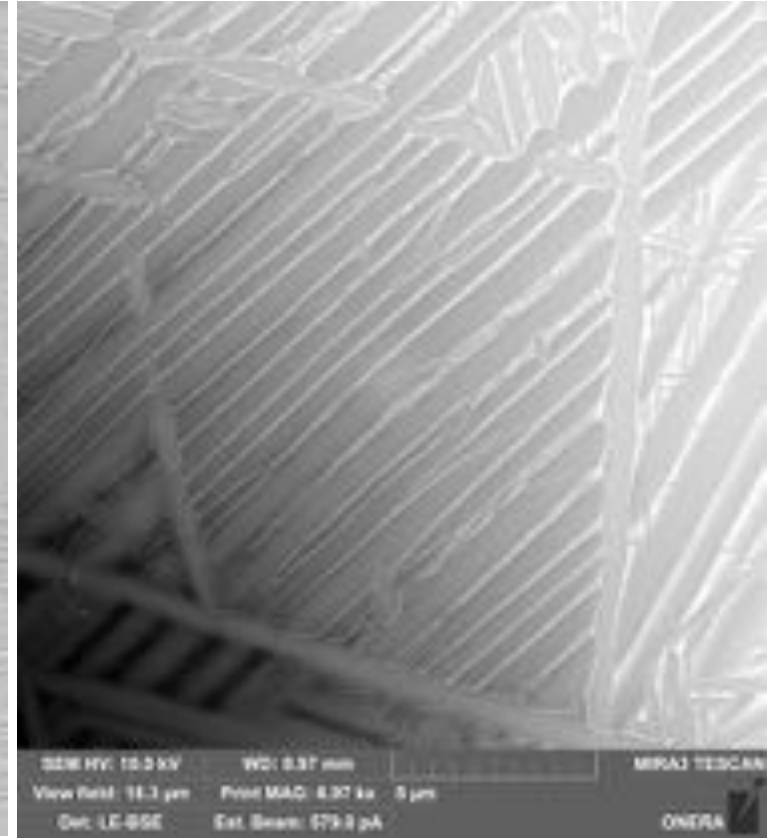
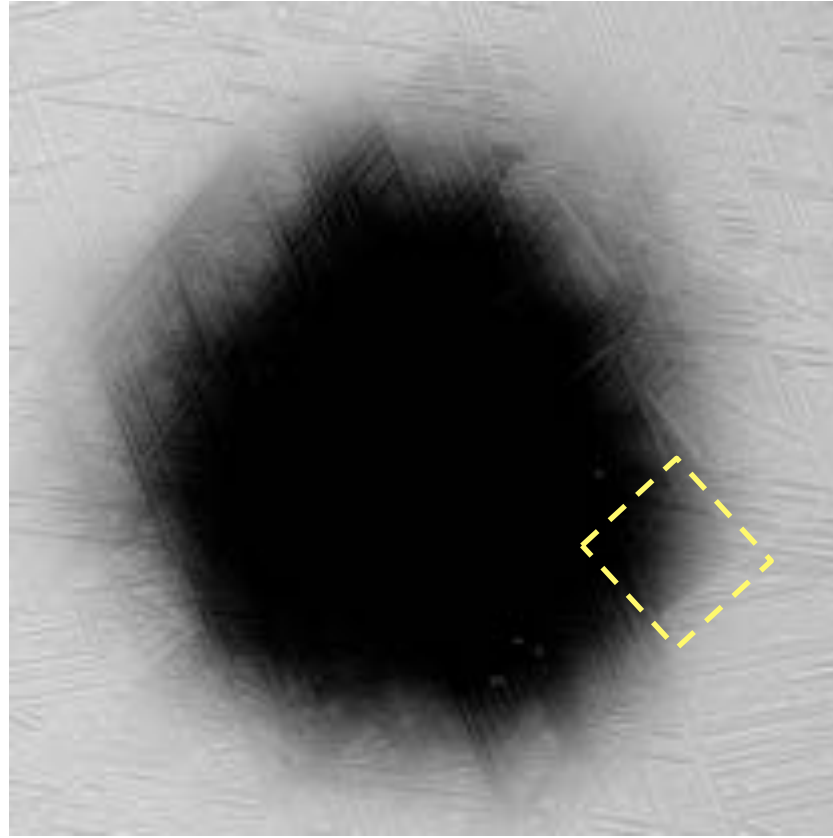
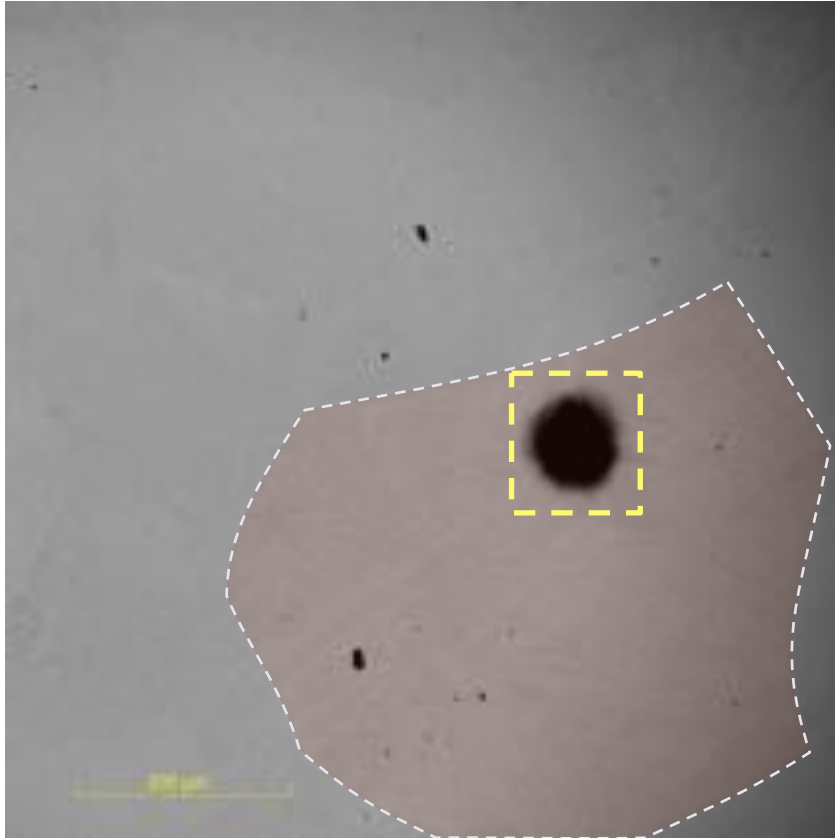


Transition d'échelle : du grain à l'atome



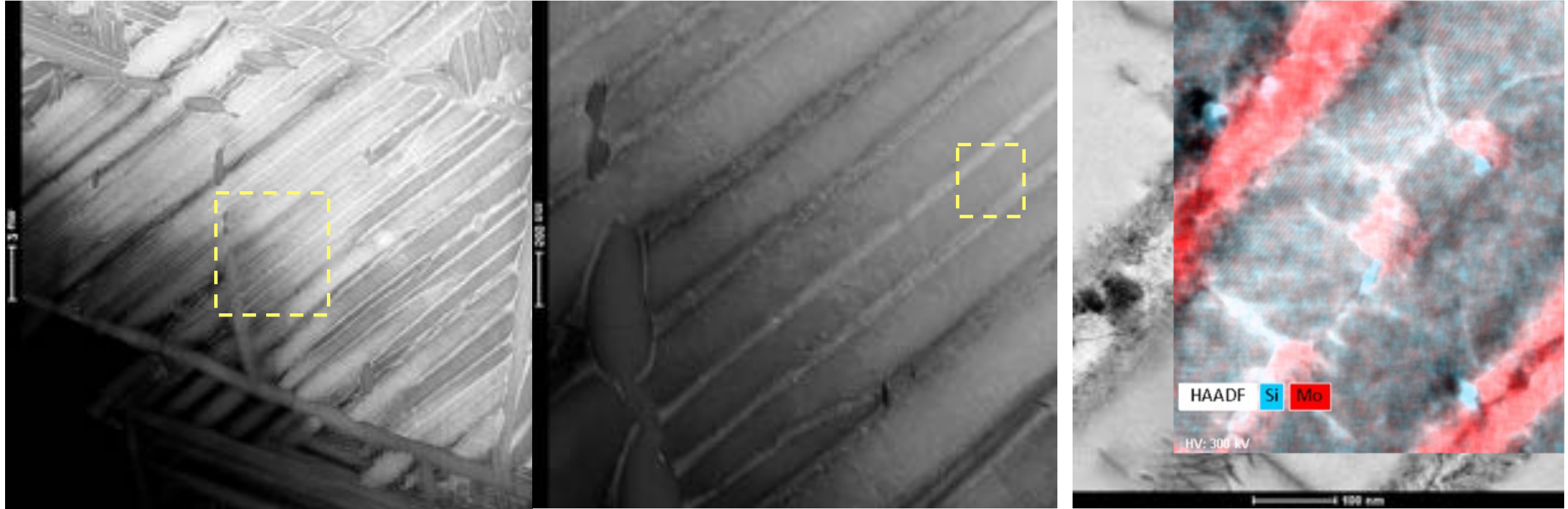
Transition d'échelle : du grain à l'atome (MEB)

Alliage aéronautique Ti quasi α



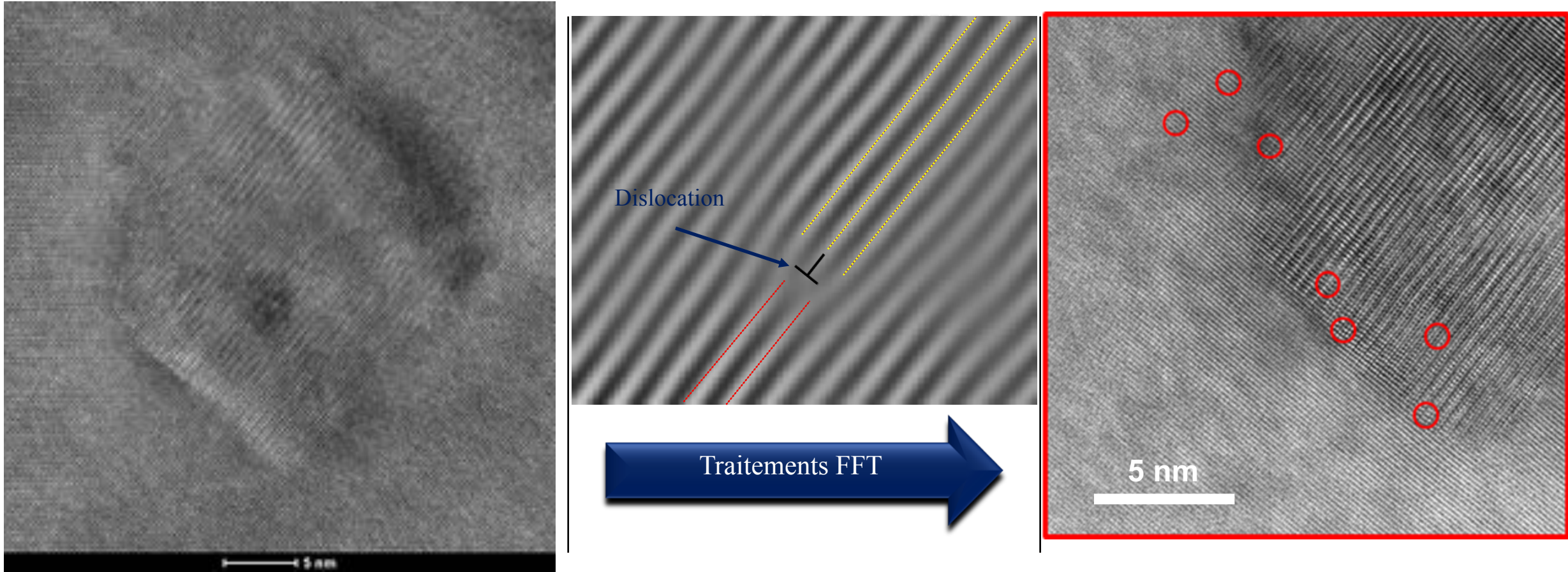
Lames MET préparée par gravure électrolytique

Transition d'échelle : du grain à l'atome (MET)



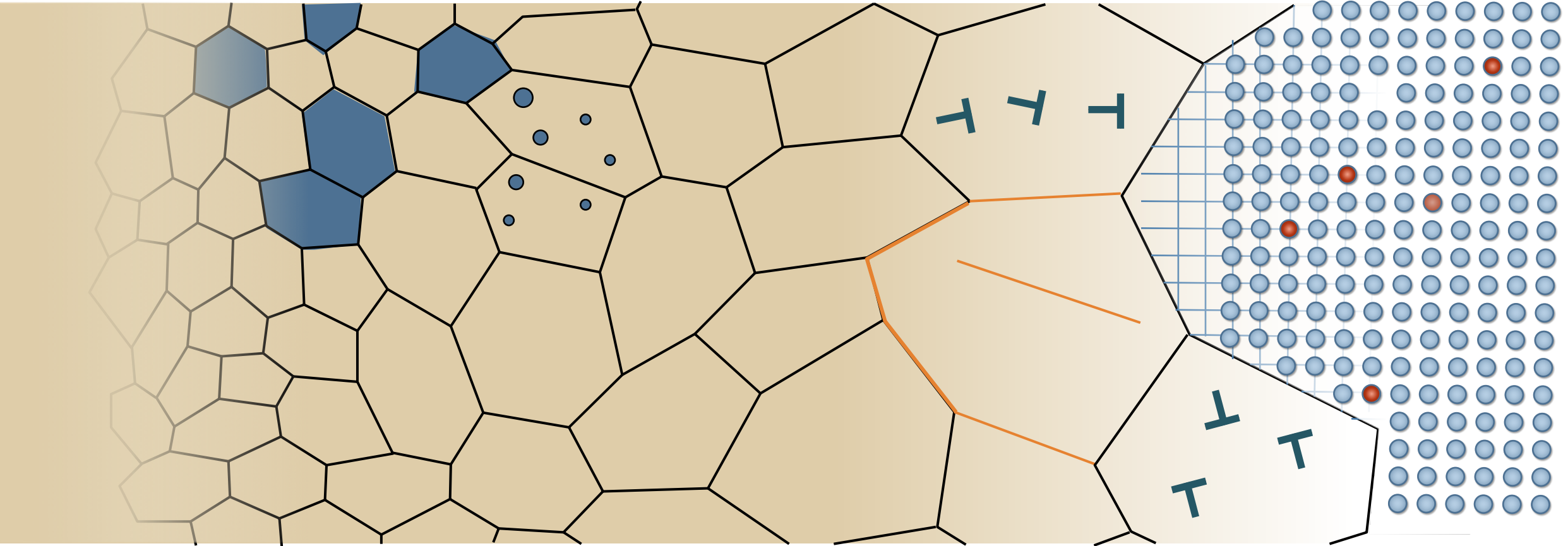
- Ancrage des dislocations sur les précipités dans la phase α
- Déstabilisation de la phase α par un enrichissement local en Mo

Transition d'échelle : du grain à l'atome (HR-STEM)



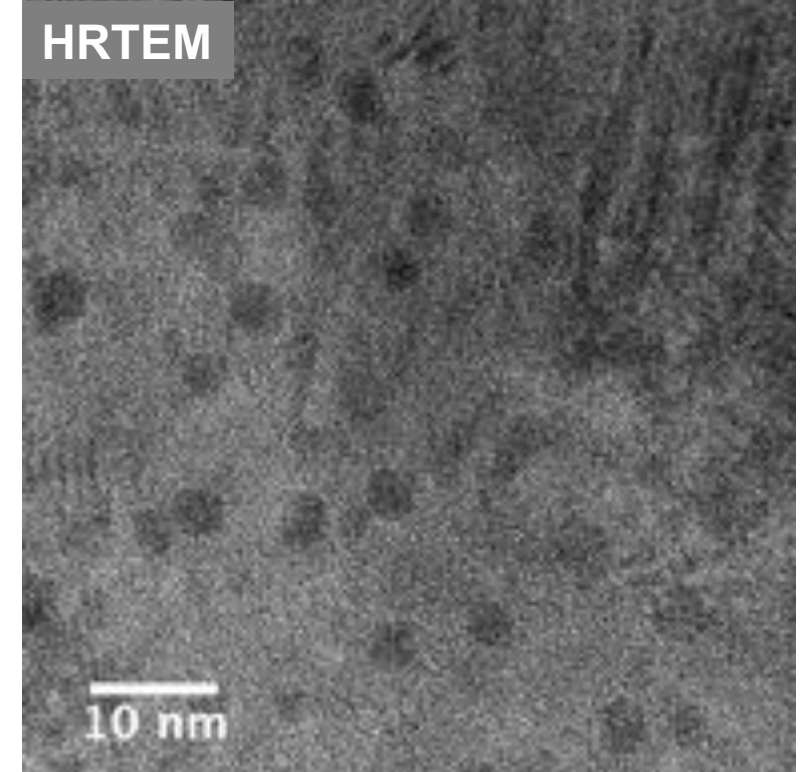
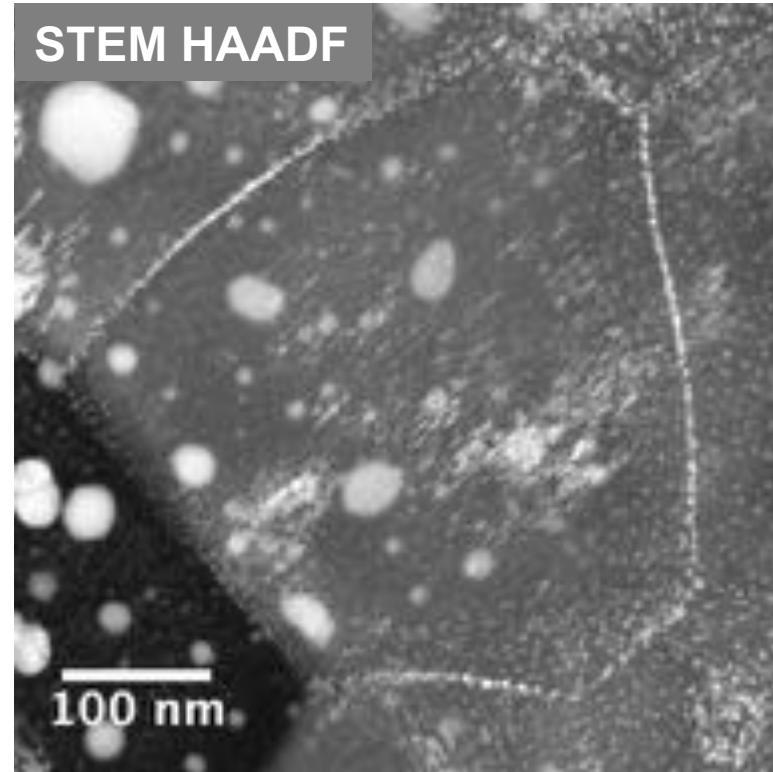
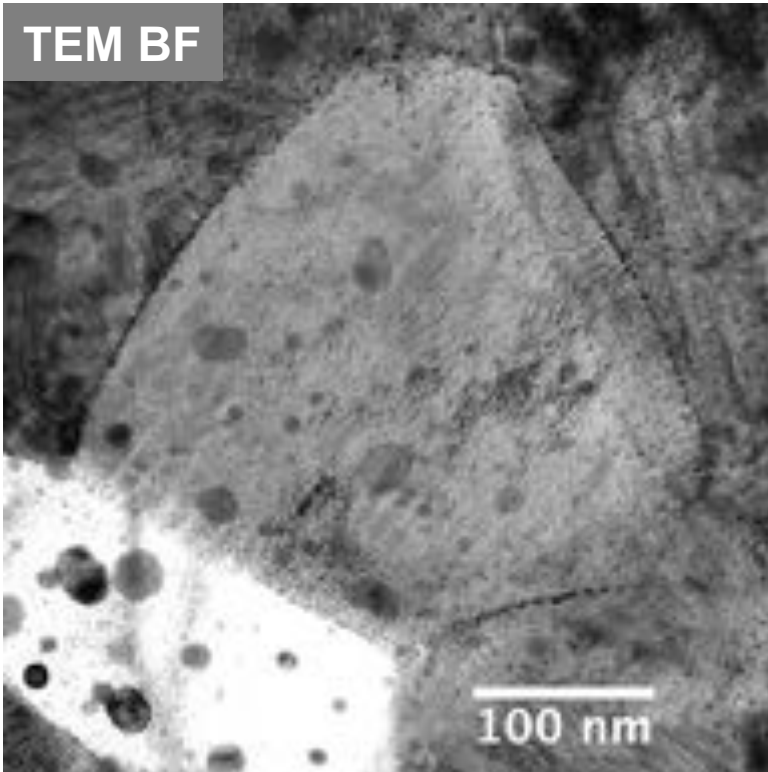
- Semi-cohérence des précipités M_xSi_y

Voir en 3D



Voir en 3D

Oxydation d'un alliage TiAlW

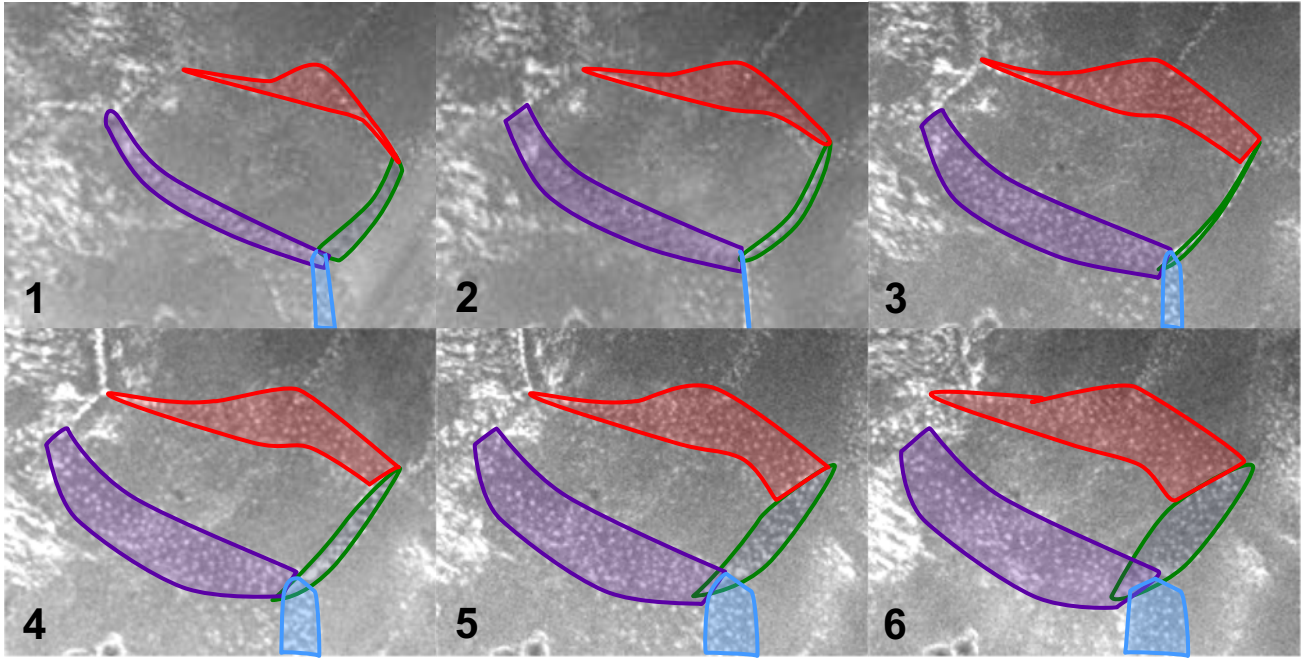
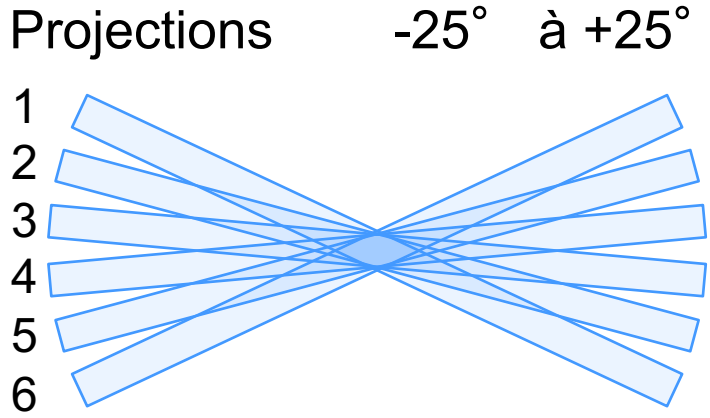


→ Origine des nanoparticules : intrinsèque, pollution ?

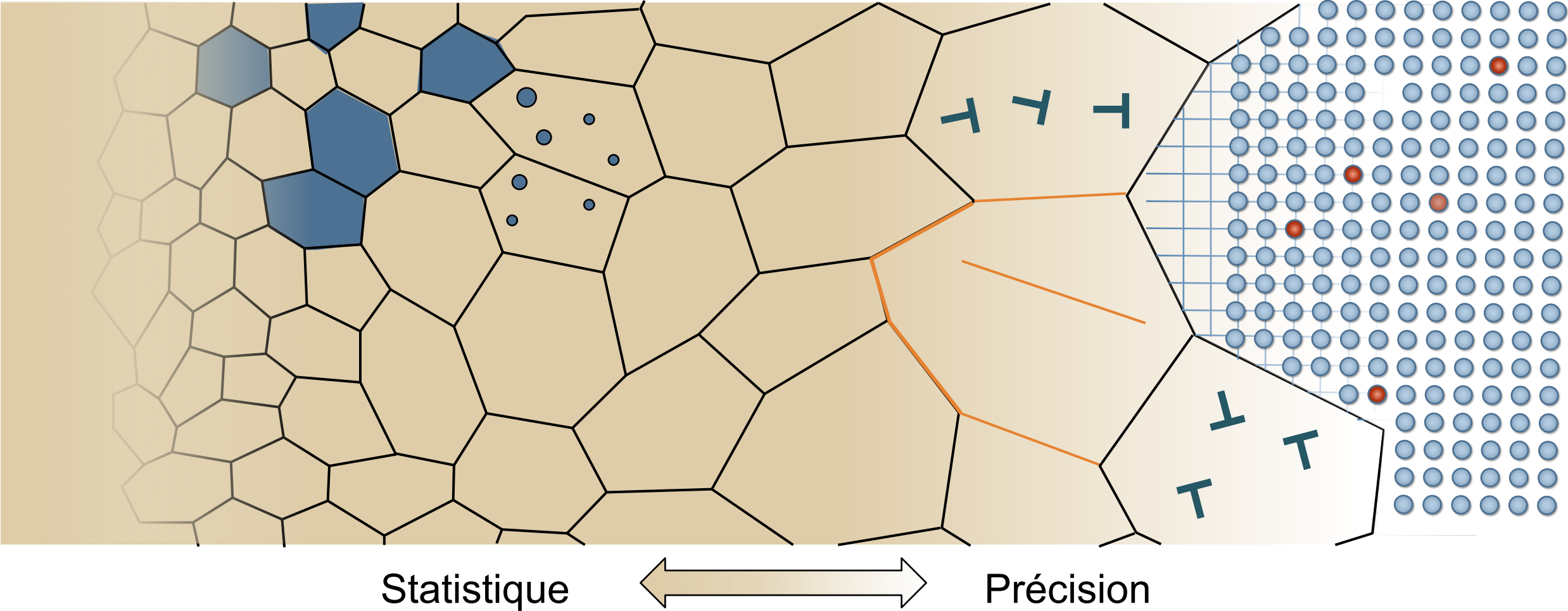
Tomographie



Le MET fait des projections
→ Incertitude sur la nature du volume traversé

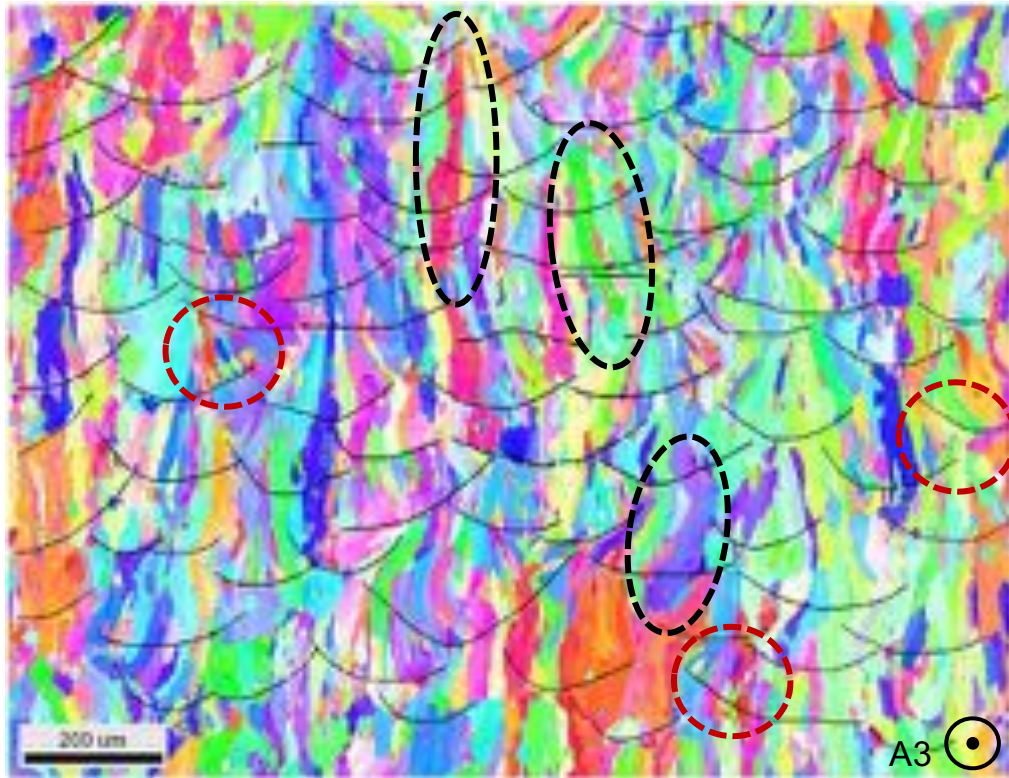


Croiser les techniques

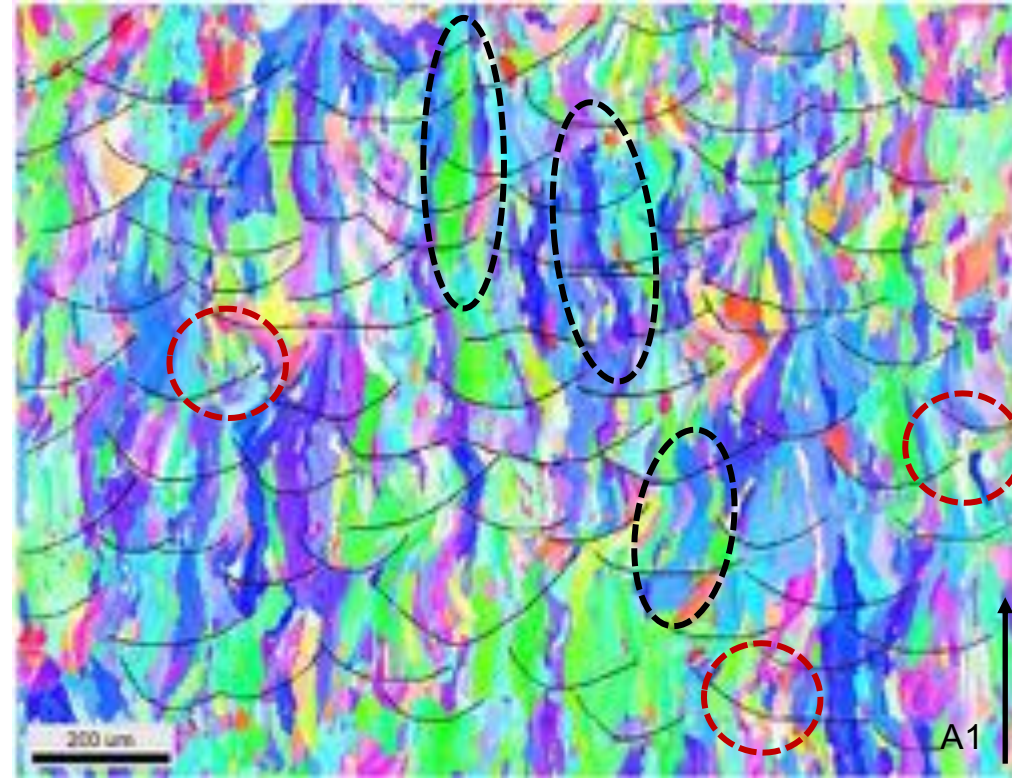


Microstructure brute de AlFe obtenu par F.A.

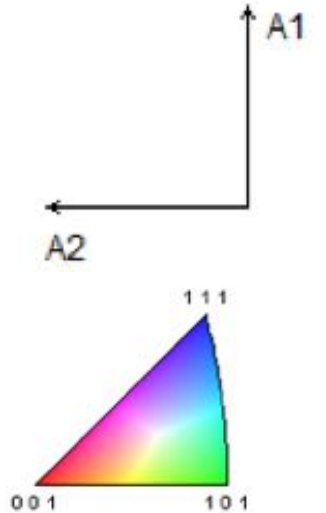
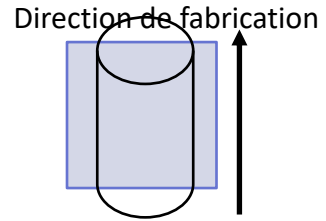
❖ Morphologie des grains dans les bains de fusion



IPF [001]



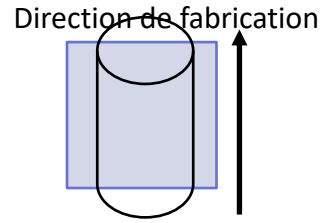
IPF [100]



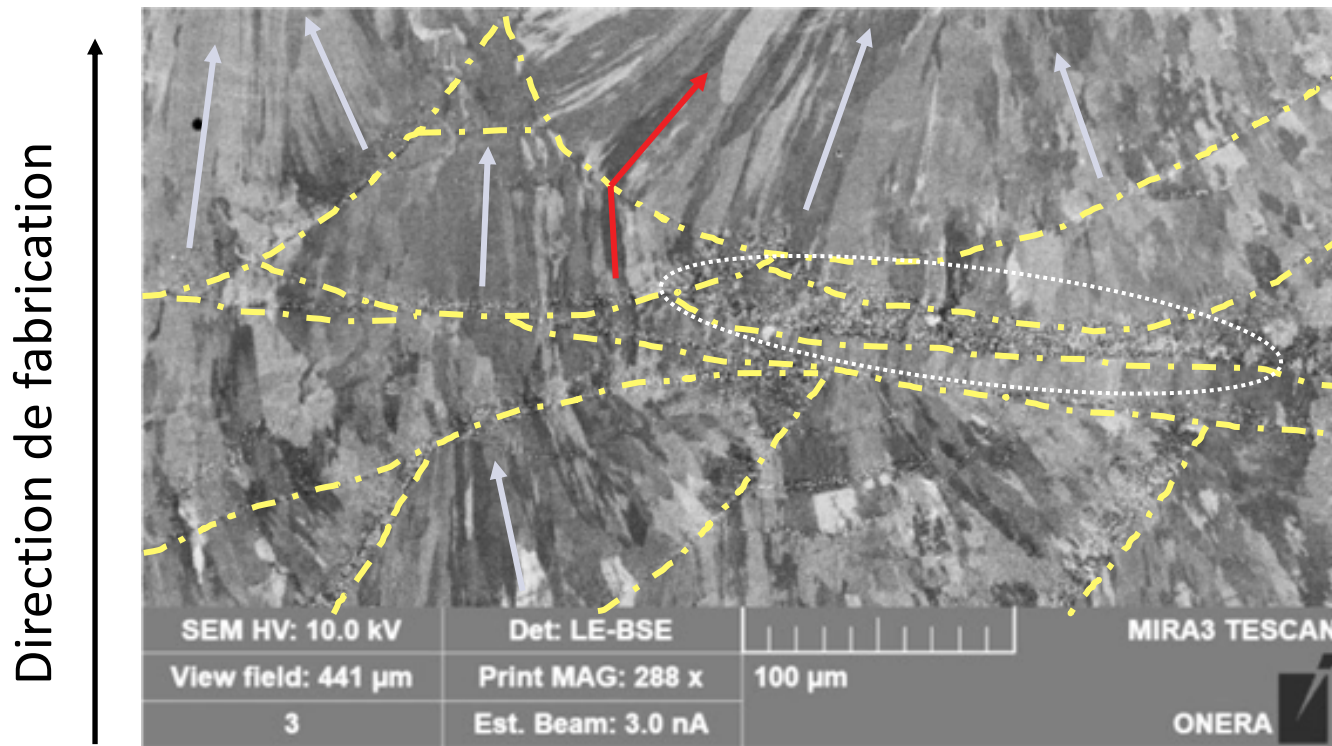
- Grains colonnaires traversant plusieurs bains de fusion
- Grains équiaxes aux frontières des bains de fusion

Microstructure brute de AlFe obtenu par F.A.

❖ Morphologie des grains dans les bains de fusion

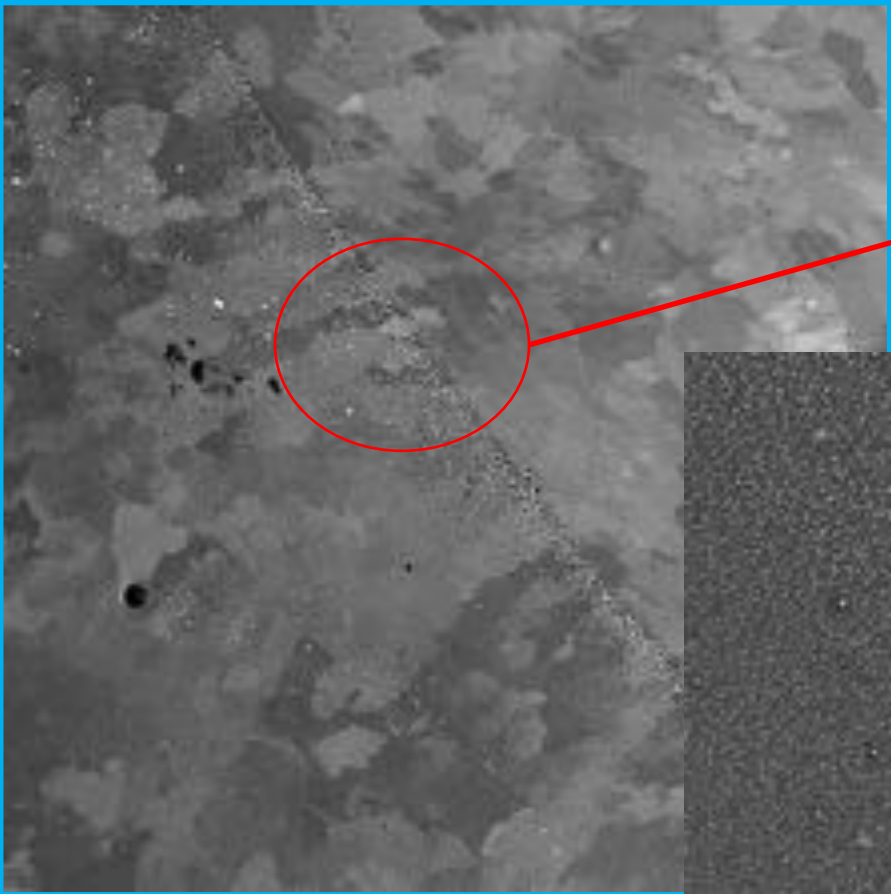


- ⇒ Croissance vers le centre des bains
- ⇒ Précipités en bas des bains de fusion

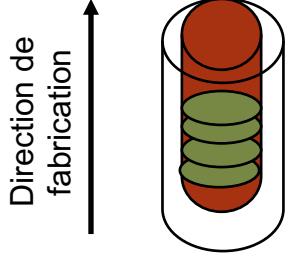
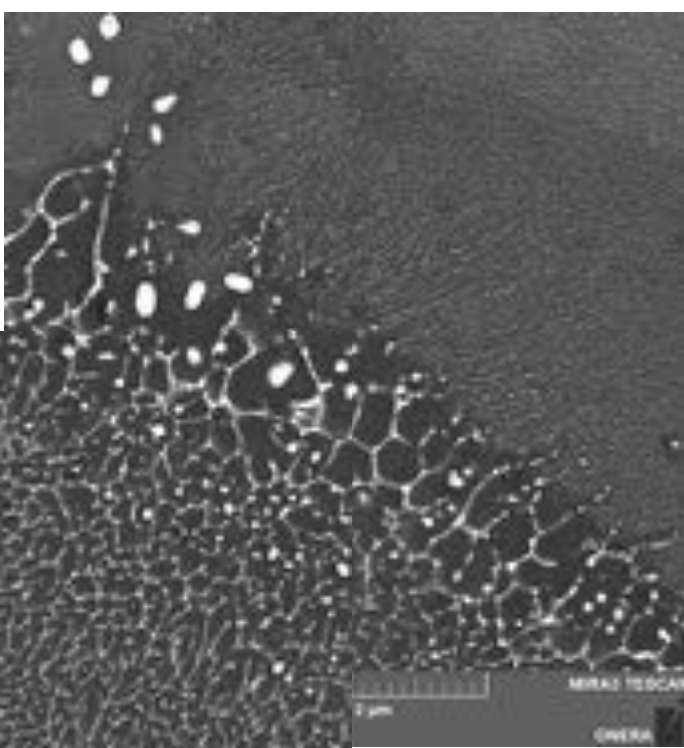
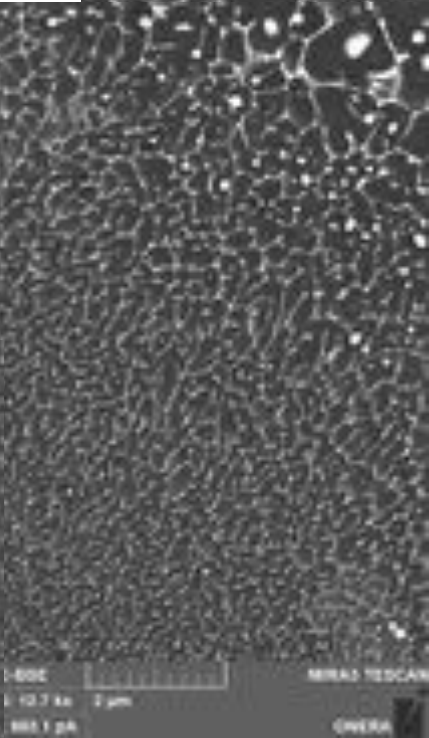
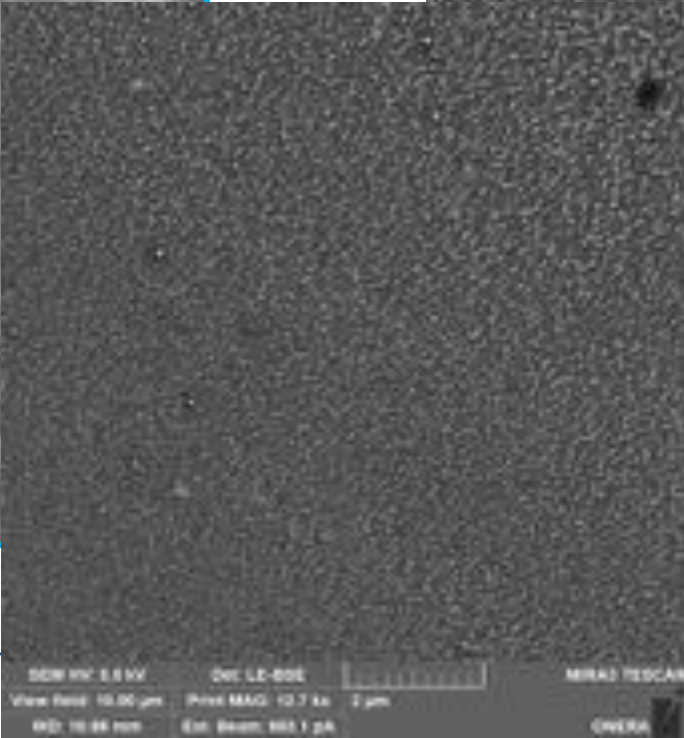


Lames minces observées au MEB

❖ Transition zone grossière/fine



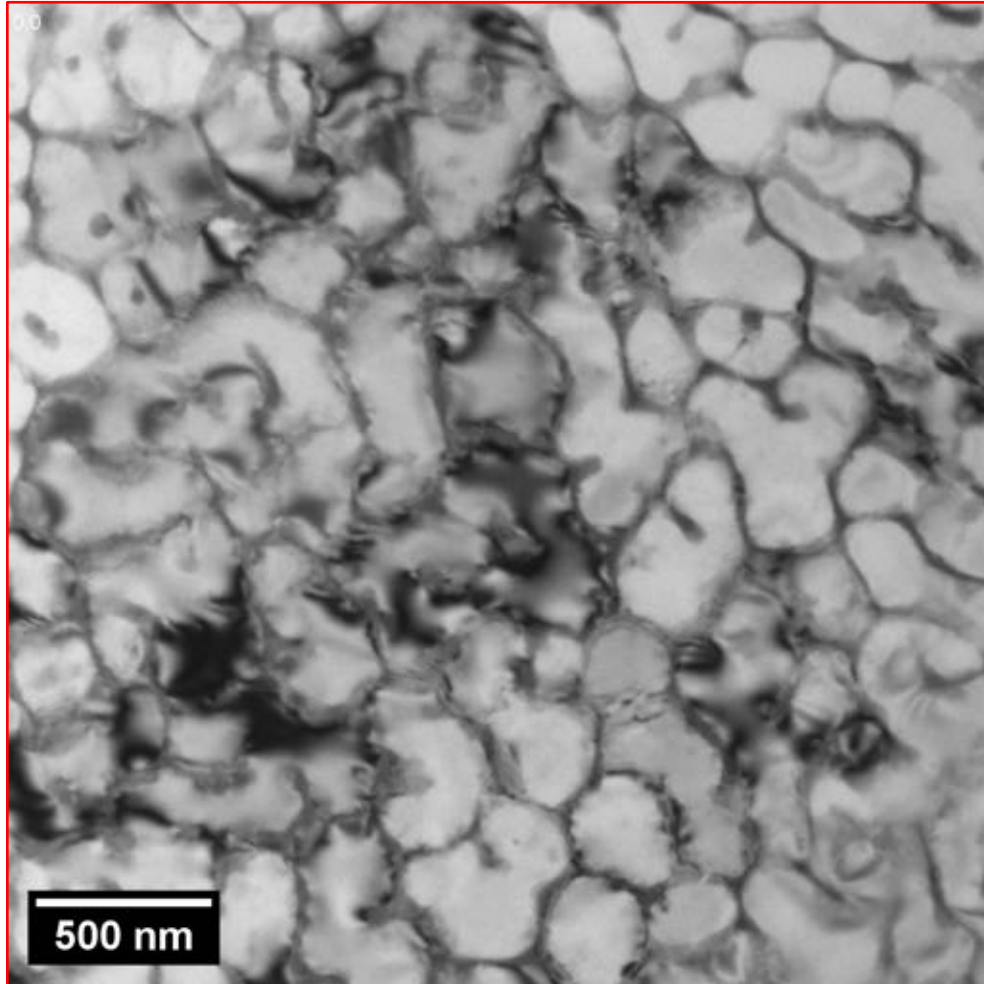
P200-110-14, L3



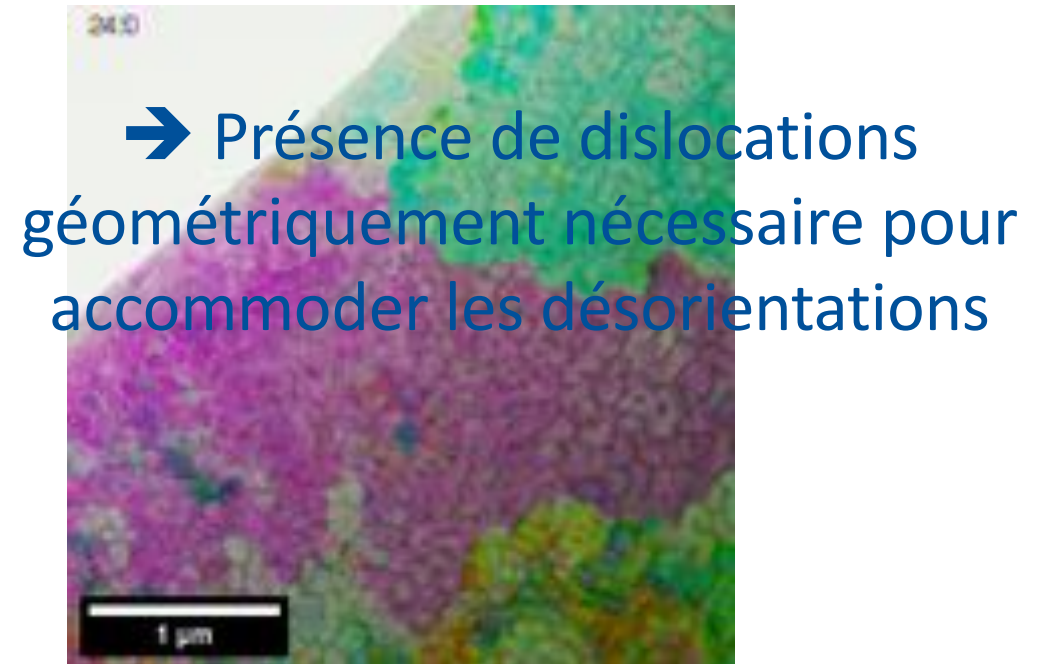
Affinement progressif de la sous-structure vers le centre du cordon

Lame mince observée au MET

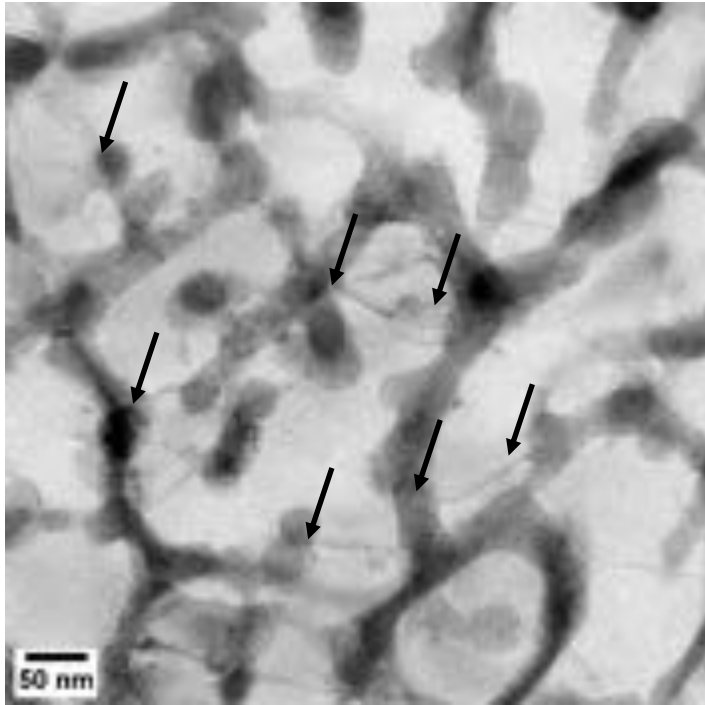
❖ Désorientations entre cellules



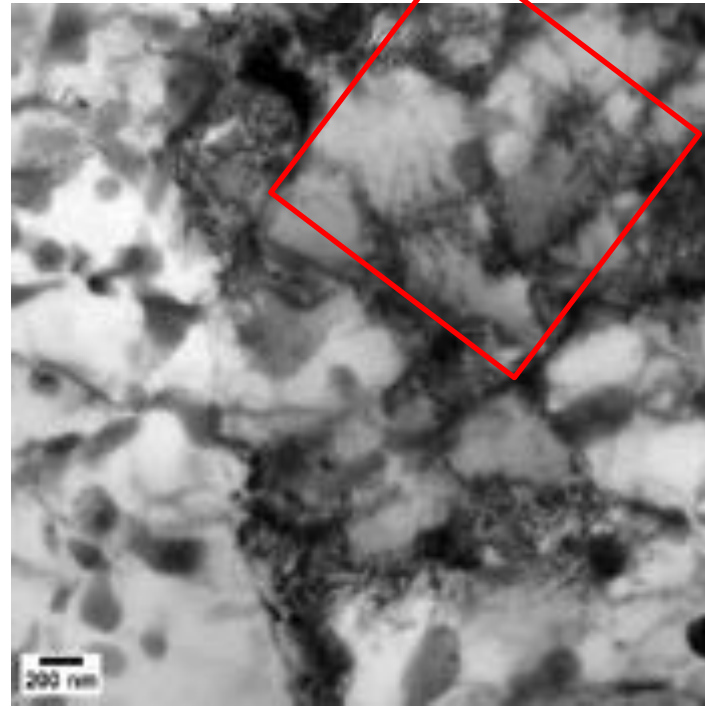
- Désorientations entre groupes de cellules → grains
- Désorientations au sein d'un même groupe de cellules



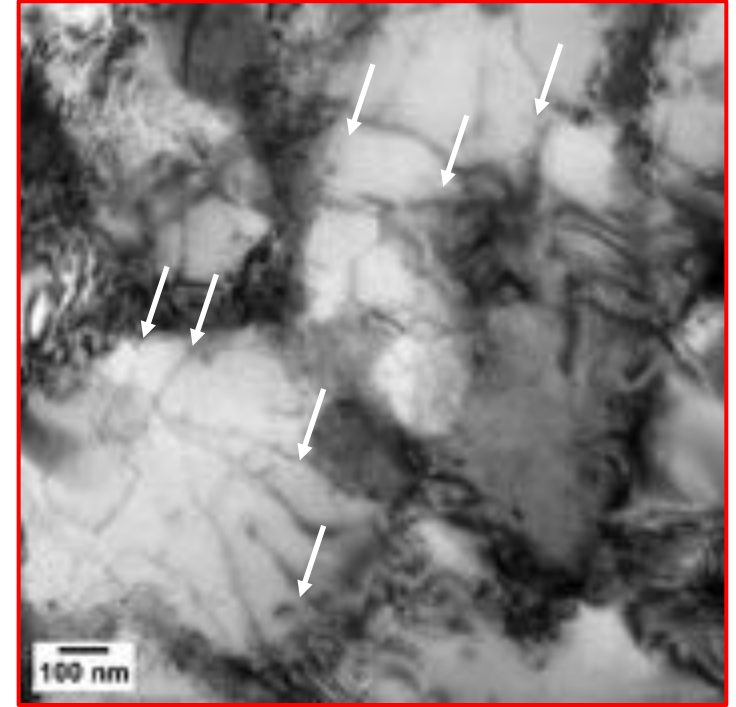
Observation des dislocations



Zone fine



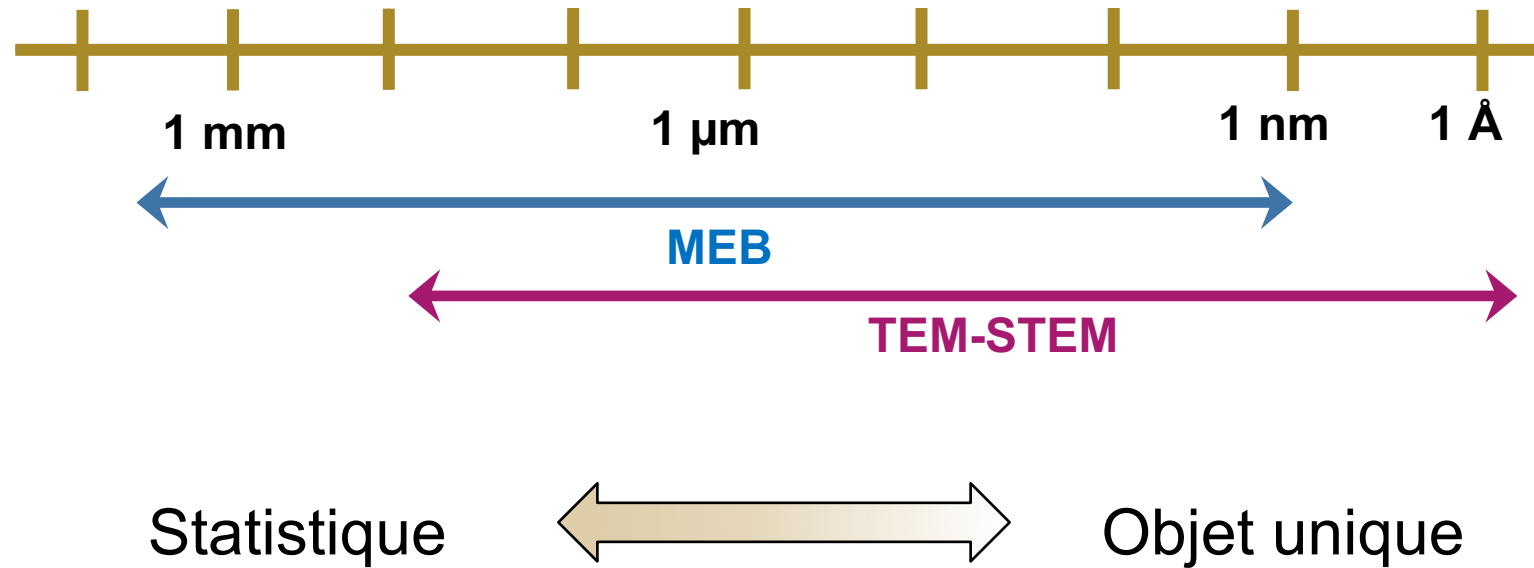
Zone grossière



- Dislocations épinglées sur les précipités et la sous-structure cellulaire dans les différentes zones
- ➔ Importance des précipités et de la sous-structure cellulaire dans le modèle de durcissement

En Bref

2 expériences distinctes → résultats et informations différentes
Complémentarité entre les 2 techniques :



Nécessité de croiser les techniques pour aller vers une caractérisation structurale et chimique multiéchelle
Aller au-delà et croiser avec SAT et XRD

Remerciements

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et vous pour votre attention !