

L'EBSD (Electron BackScatter Diffraction) est une technique d'acquisition et d'analyse automatisée basée sur la diffraction des électrons rétrodiffusés et implantée dans un microscope électronique à balayage (MEB), mais parfois aussi sur des microsondes Auger et historiquement de Castaing.

- Introduction à l'EBSD
- Caméras, vitesses d'acquisition
- Applications



## Technologie et comparaison des caméras utilisées en analyse EBSD

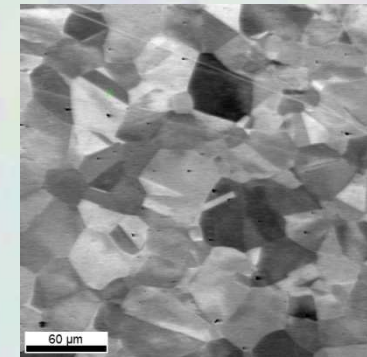
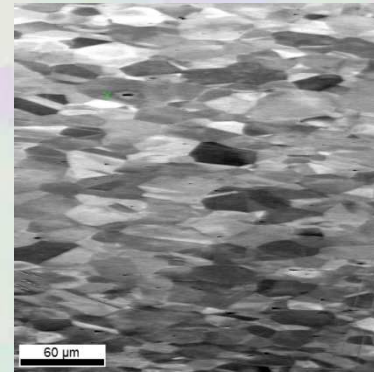
*D. Boivin / ONERA - F. Brisset / CNRS-ONERA*

- Les divers types de caméras - Principes généraux
- Utilisation de caméras CCD en analyse EBSD - Tests comparatifs
- Conclusion

GN-MEBA - Décembre 2002

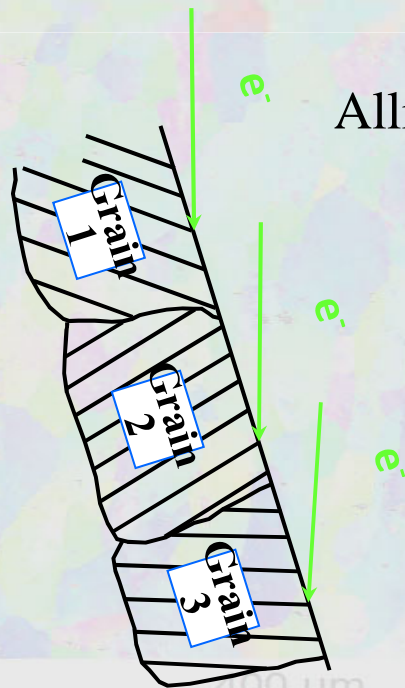
ONERA

# Electrons rétrodiffusés



SE et BSE  
Alliage de Ni (mêmes conditions d'acquisition - 70° d'inclinaison)

FSD incliné et corrigé



Incidence variable par rapport  
aux différents plans cristallins

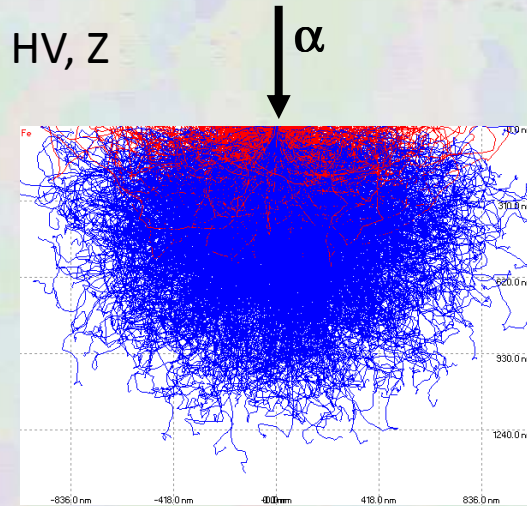


coefficients de rétrodiffusion  
différents

→ Information qualitative ...

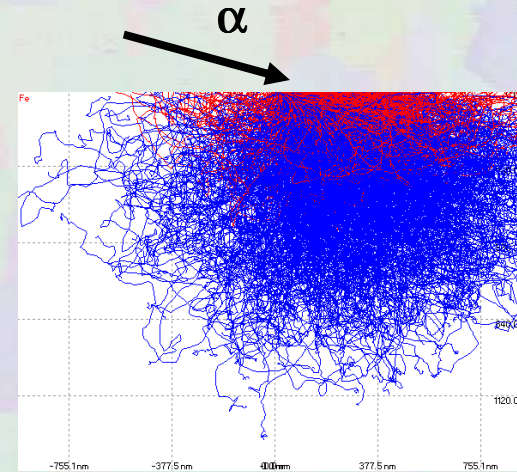
# Electrons rétrodiffusés

casino



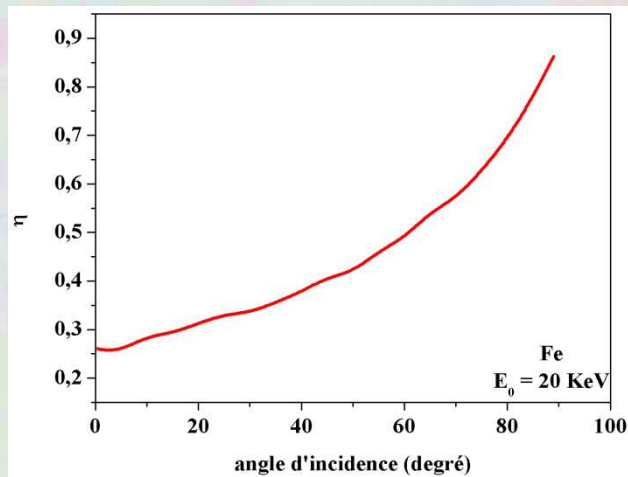
$0^\circ$   
0.29

$$\eta = \frac{n_{er}}{n}$$



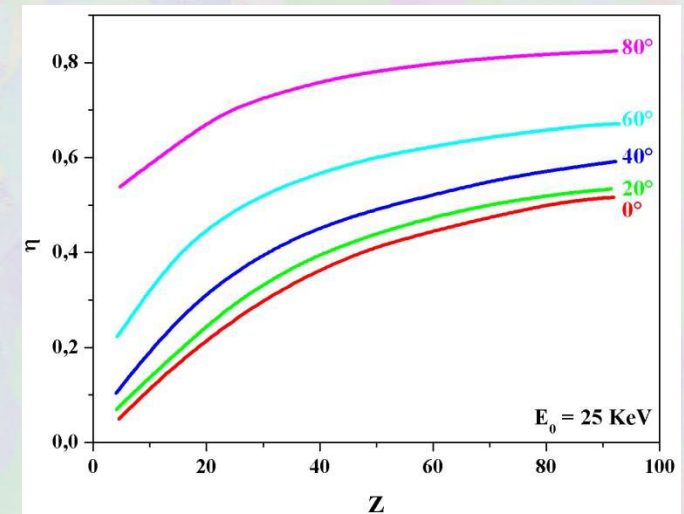
$70^\circ$   
0.58

Simulation des trajectoires électroniques (en rouge, électrons rétrodiffusés)



Si  $\alpha$  augmente le volume d'interaction se déplace vers la surface et  $\eta$  augmente

si  $\alpha$  augmente sa dépendance en Z diminue



400 um

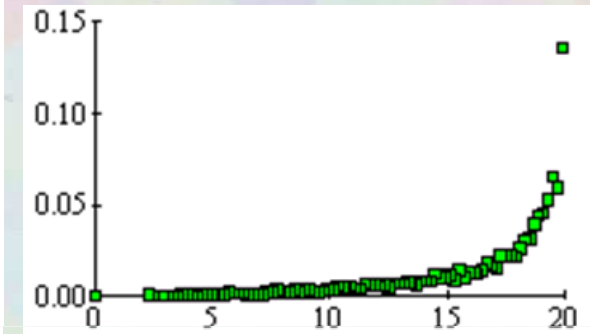
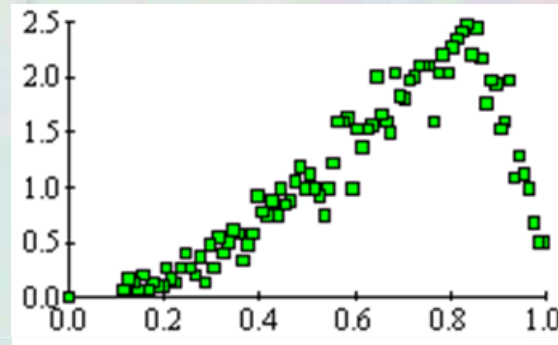
# Rappel électrons rétrodiffusés

tilt 0°  
 $\eta \sim 30\%$

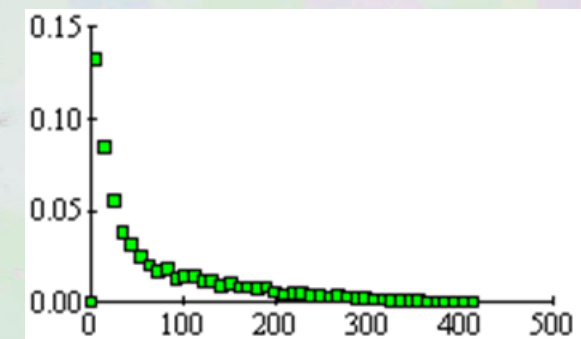
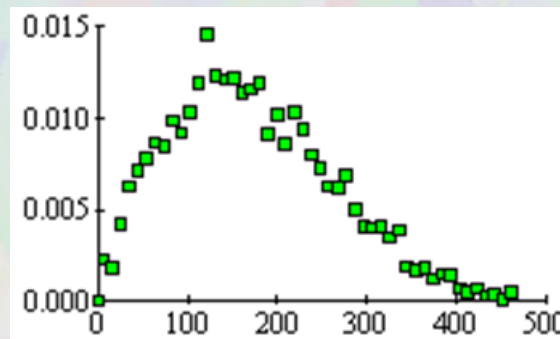
Fe - 20 keV

tilt 70°  
 $\eta \sim 60\%$

Distribution énergétique



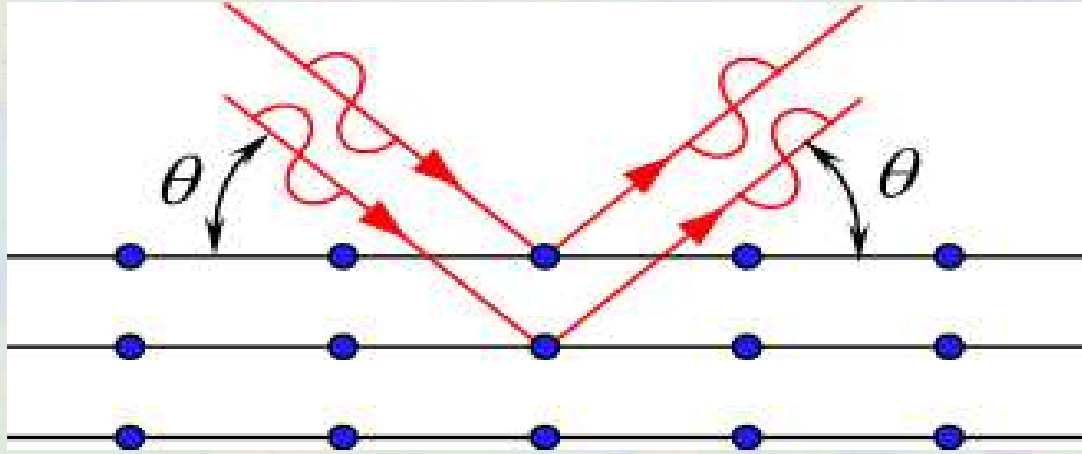
Profondeur d'échappement



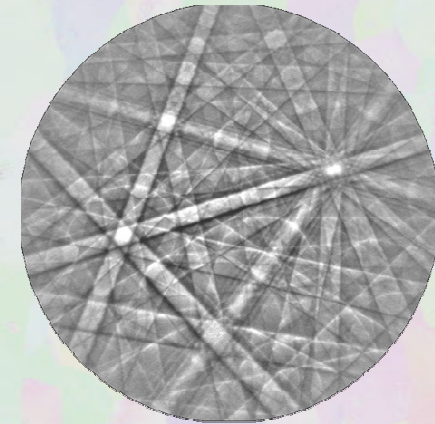
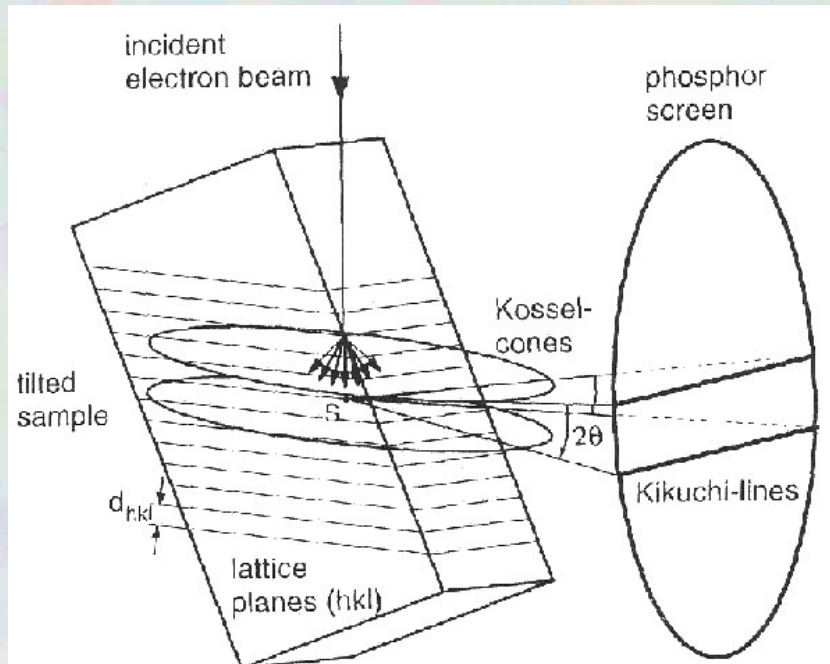
JLP-soft

400 um

# Loi de Bragg



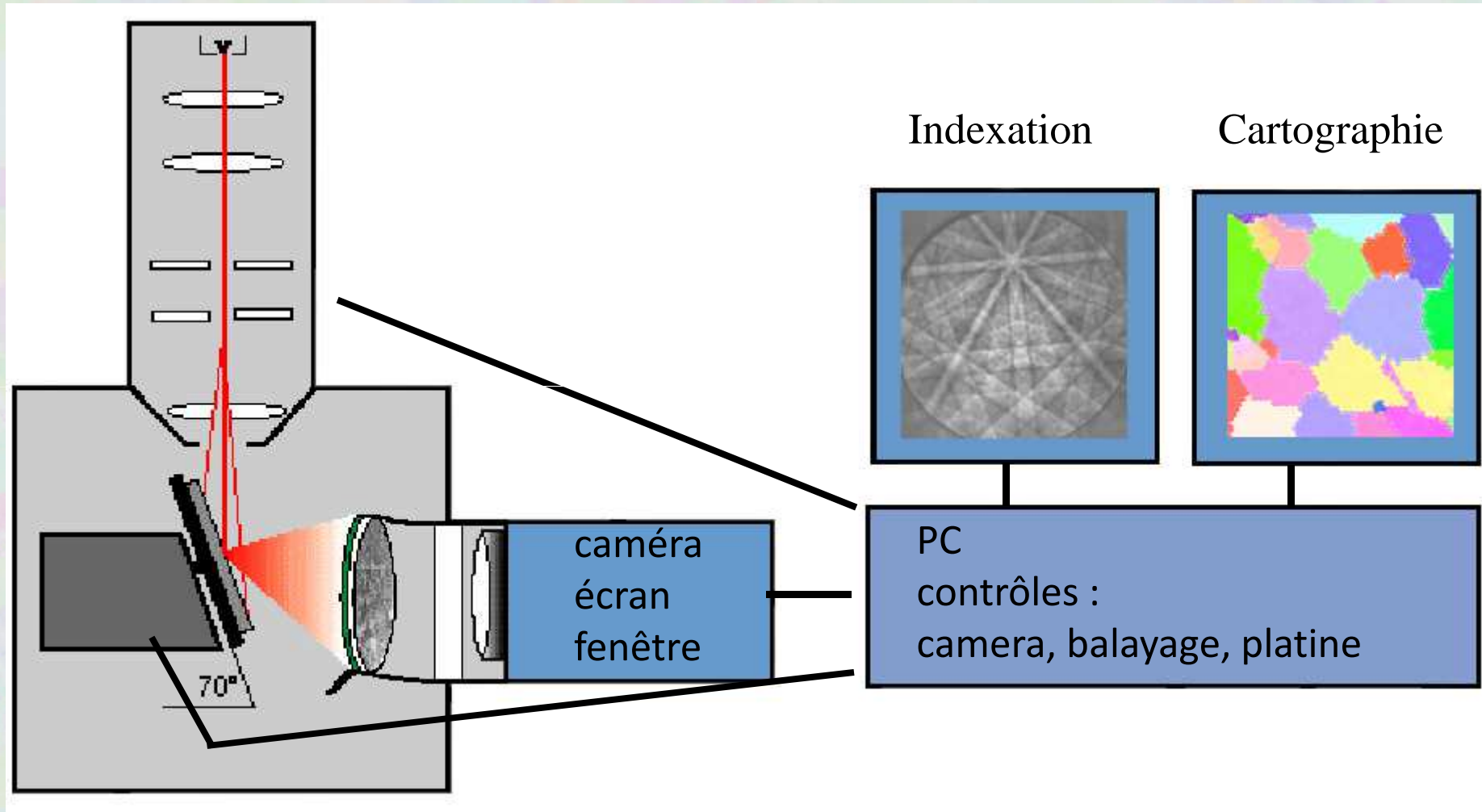
$$n\lambda = 2d_{hkl} \sin \theta$$



400 nm

# Le système EBSD

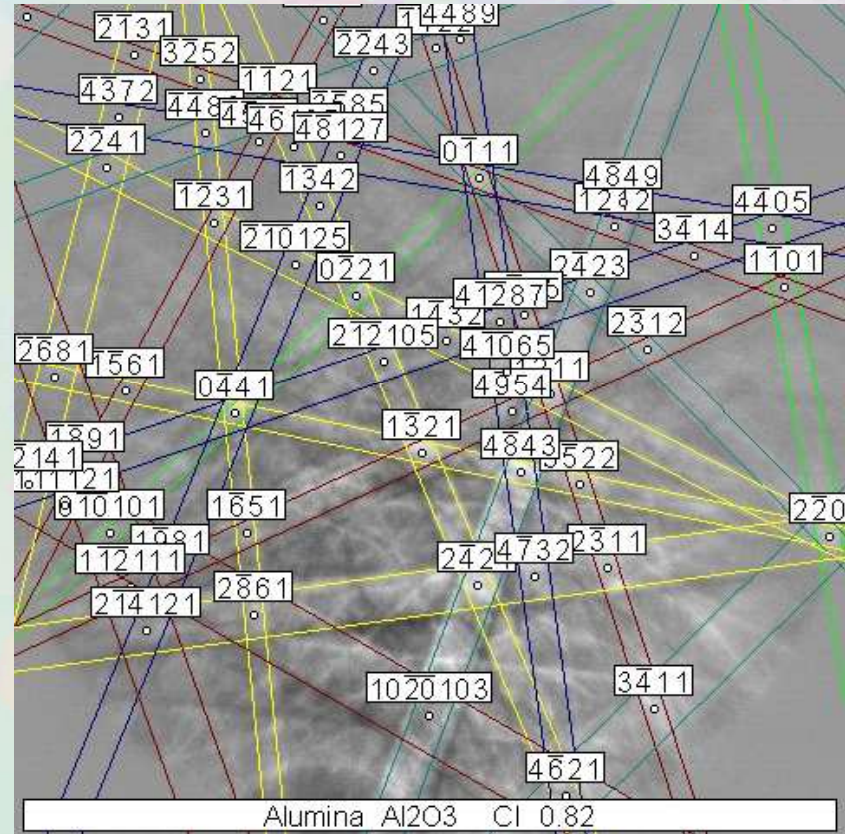
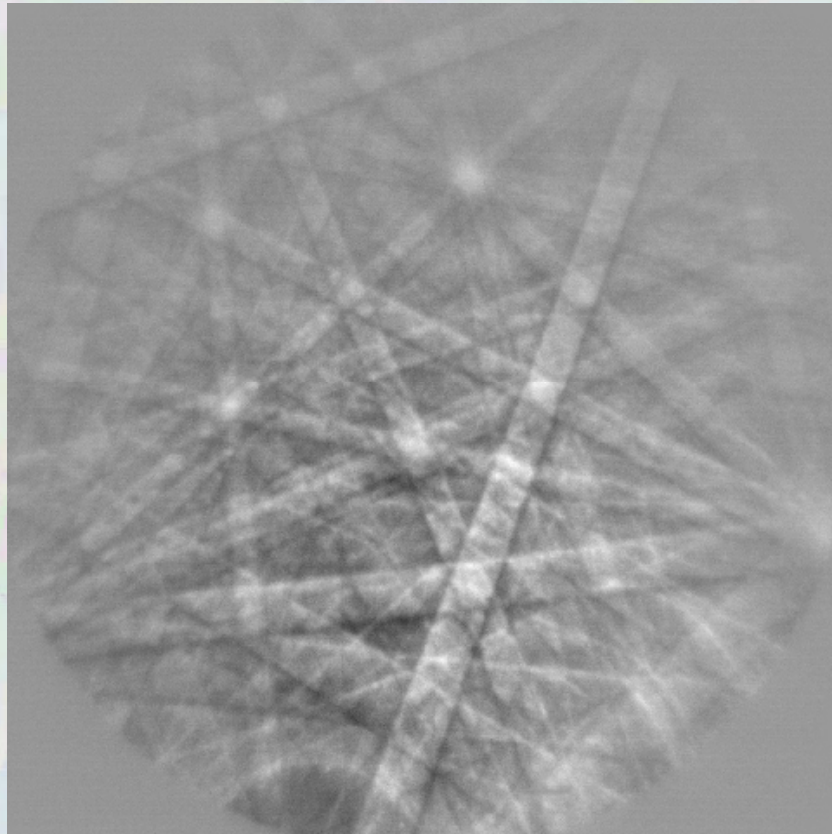
W, FEG cathode chaude (cold), (LaB6)



Détecteurs associés possibles : BSE ou FSD, AED et EDS

400 um

# Indexation d'un cliché EBSD – comment ?

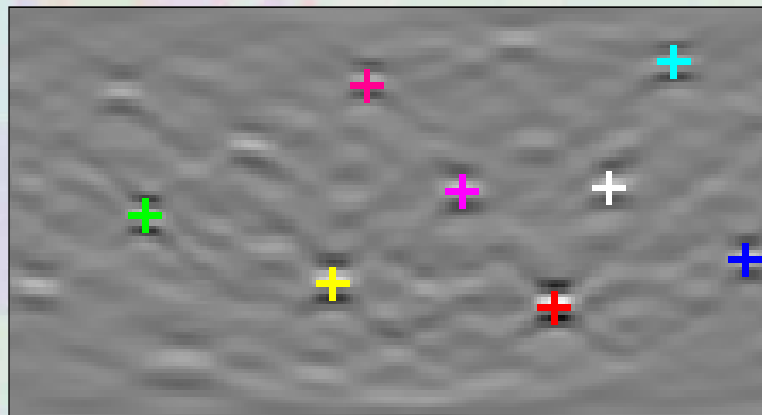
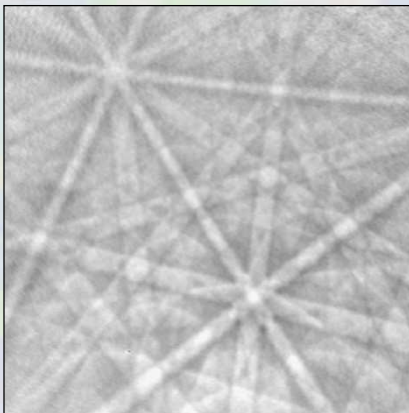


➡ Comment aller d'ici ..... à ..... ici.

400 um

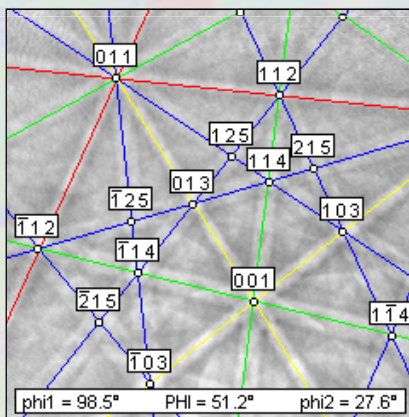
# Indexation d'un cliché

Acquisition d'un cliché EBSD

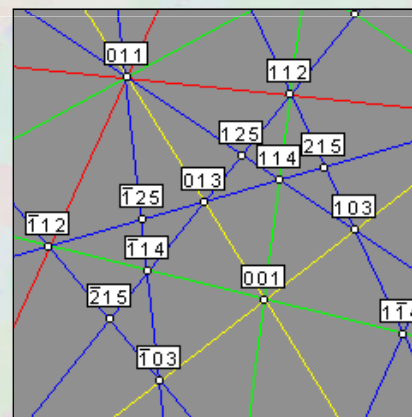


Calcul de la transformée de Hough

Détection des bandes (spot clairs)



Solution de l'orientation cristalline visible sur le cliché EBSD

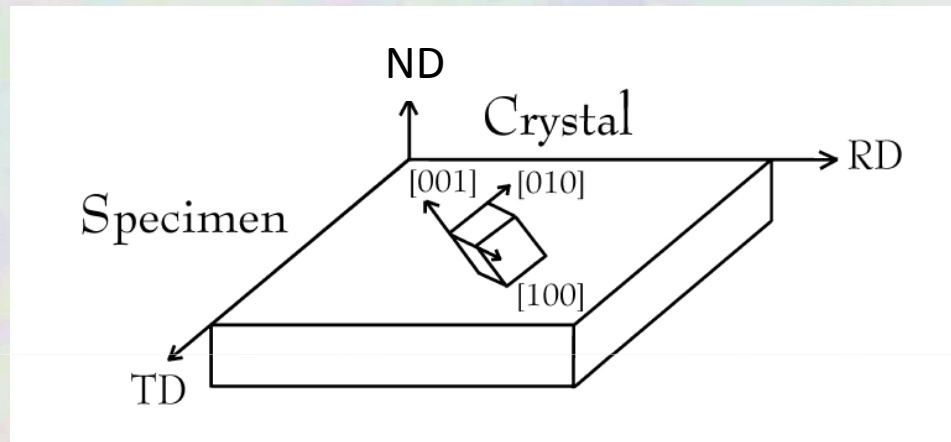


Indexation

400  $\mu\text{m}$

# Indexation d'un cliché : orientation

Rolling Dir, Transverse Dir, Normal Dir  
(représentation métallurgique)

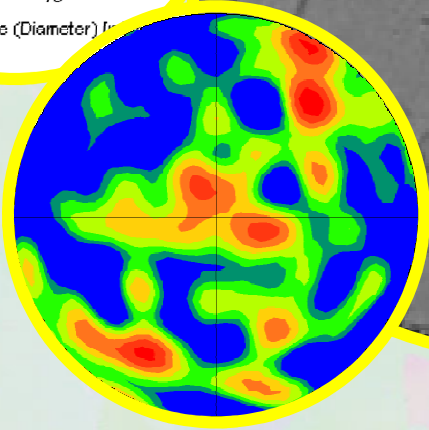
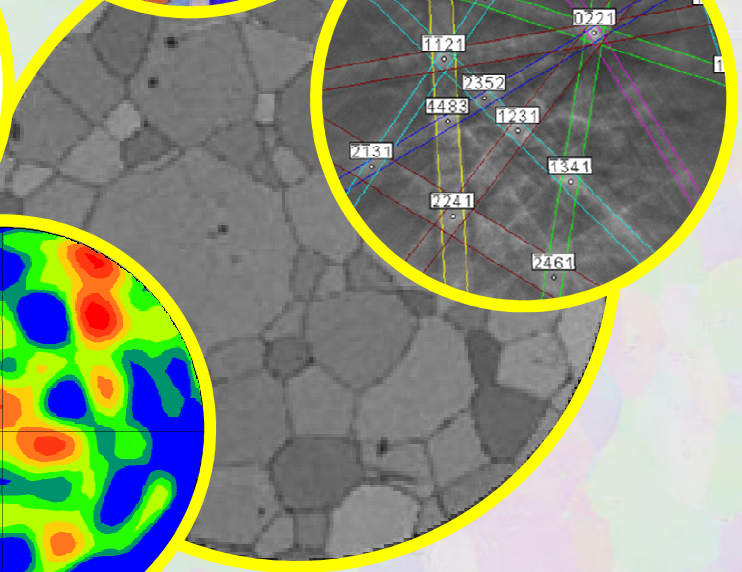
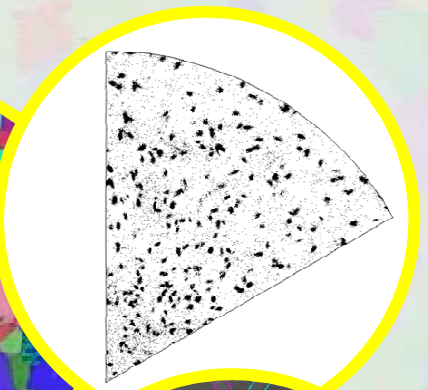
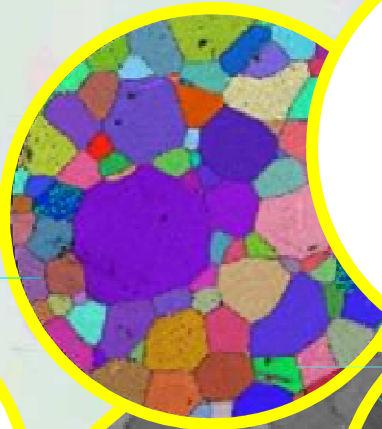
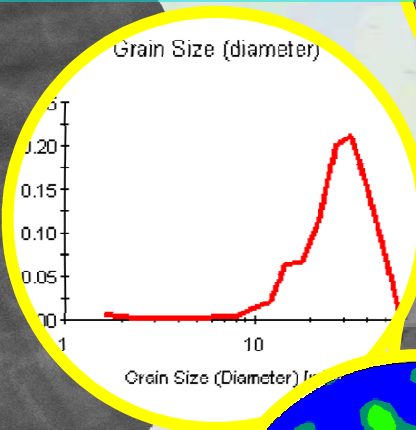
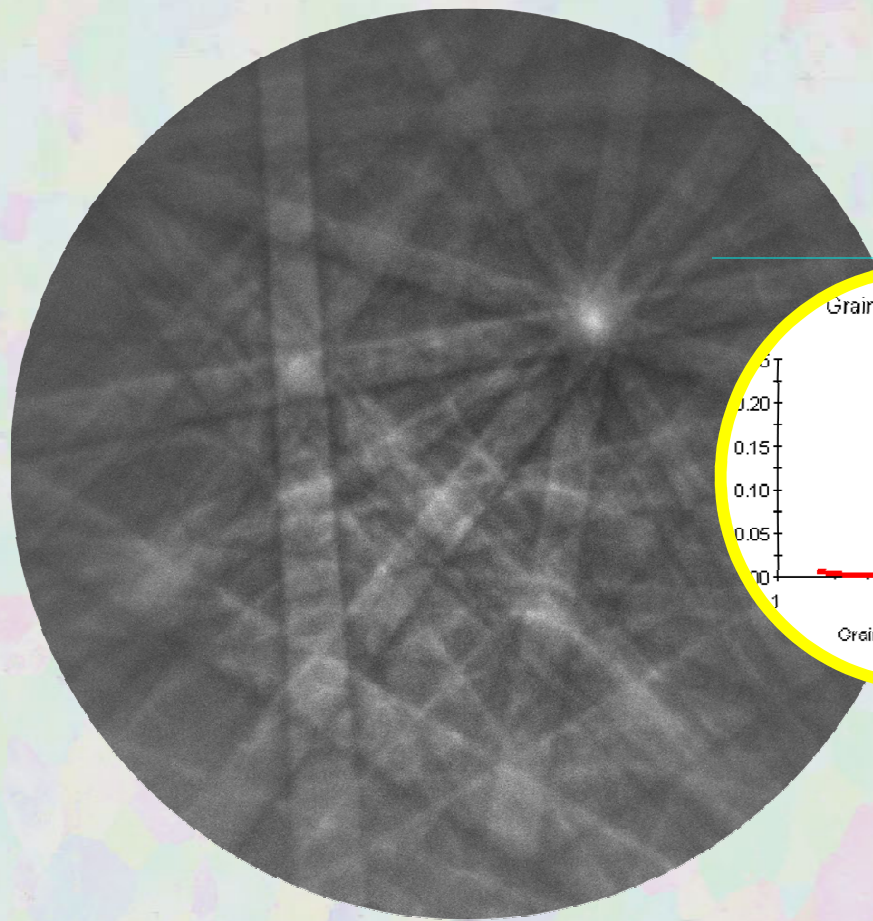


**3 angles d'Euler**  
 $\varphi_1, \Phi, \varphi_2$

3 angles d'Euler : décrivent les rotations nécessaires pour amener les axes du cristal en coïncidence avec les axes principaux de l'échantillon.

400  $\mu\text{m}$

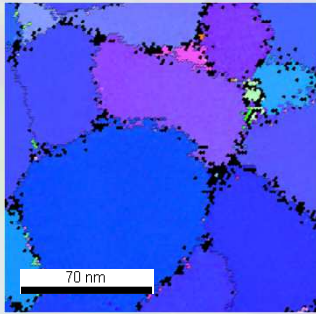
... toute sorte de représentation



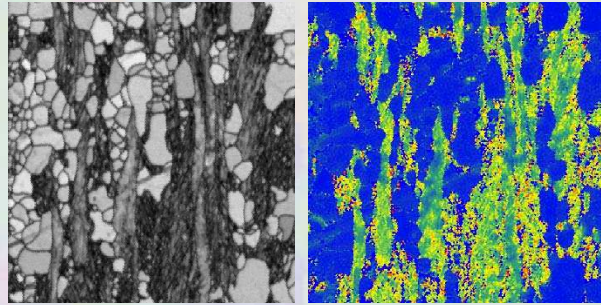
EBSD sur polycristal d' $\text{Al}_2\text{O}_3$

400  $\mu\text{m}$

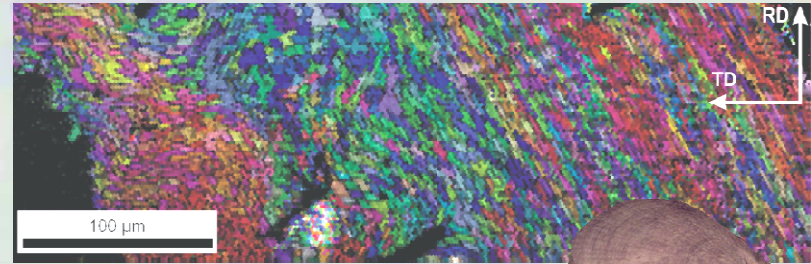
... toute sorte de matériau cristallisé



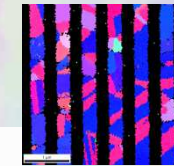
Dépôt PVD



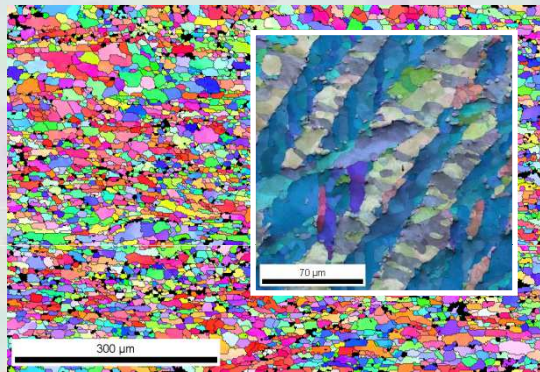
Matériaux partiellement recristallisés



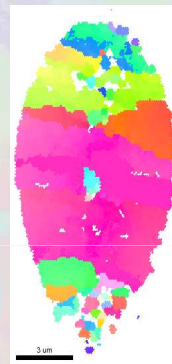
Coquilles



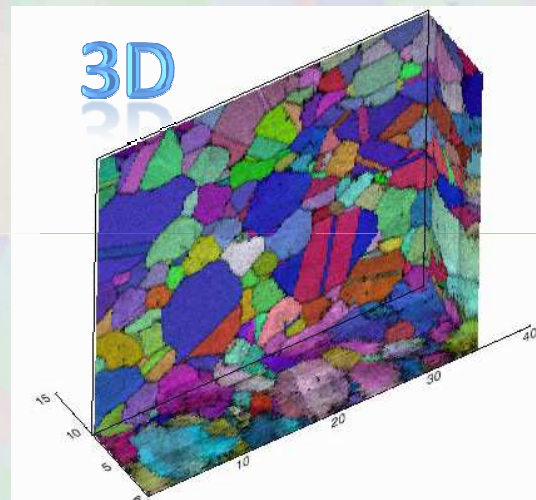
Semi-conducteurs



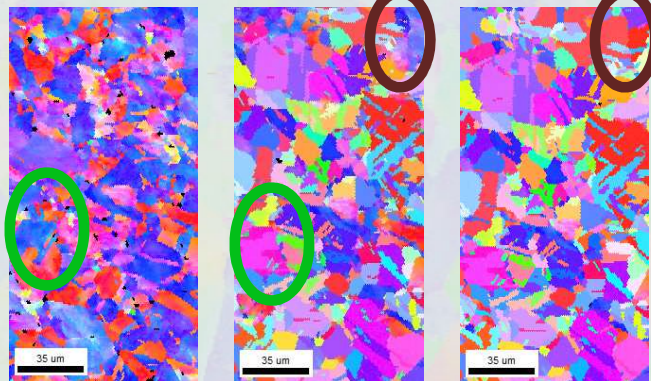
Roches et météorites



Verres

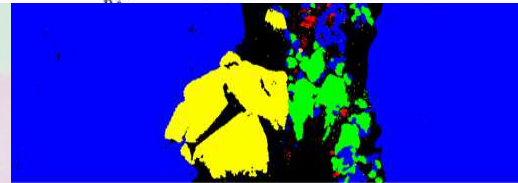


Matériaux multiphasés



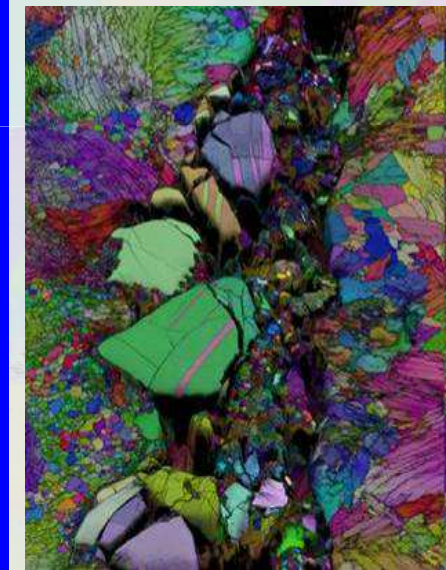
Essais in-situ

400 µm



100.0 µm = 100 steps Phase

- Alumina
- Erbium Oxide
- Monoclinic Zirconium Oxide
- Tetragonal Zirconium Oxide
- Titanium



# Evolution d'un modèle de caméra

## *CCD analogique, indexation on-line*

1990 : 10 sec/pattern semi-automatique

1995 : 3 sec/pattern automatique

1999 : 3 patterns/sec 166 MHz

2000 : 10 patterns/sec 300MHz

2001 : 20 patterns/sec 1GHz

## *CCD numérique, indexation on-line*

2002 : 40 patterns/sec caméra numérique + 2GHz

## *CCD numérique, indexation off-line*

2007 : 750 patterns/sec Dig.cameraGigE

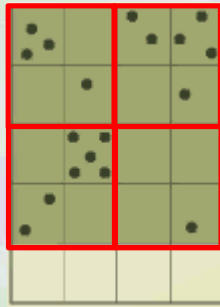
2009 : 1000 patterns/sec Dig.cameraGigE

2011 : 1170 patterns/sec Dig.cameraGigE

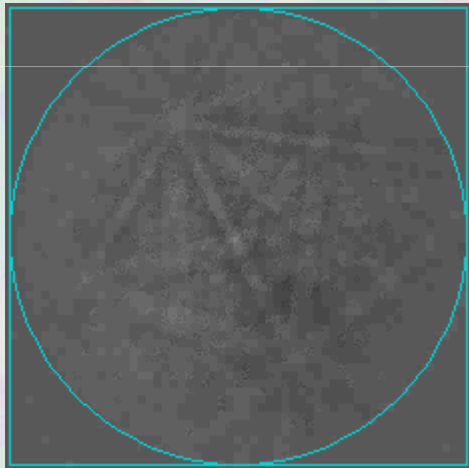
→ Courant de sonde, modifier des paramètres caméra, PC (rapide-RAM-DD), l'échantillon, ...

# Caméra binning

2 x 2



1 x 1 (1300 x 1030)

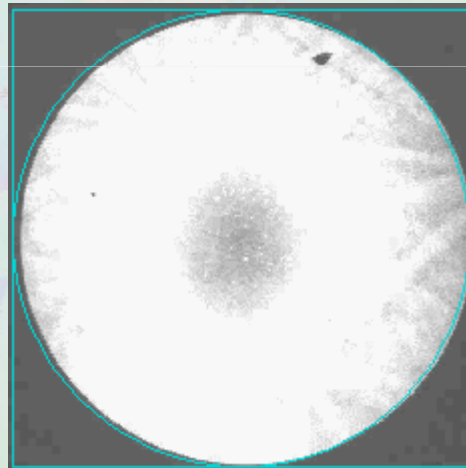


1 x 1

tps d'acquisition : T

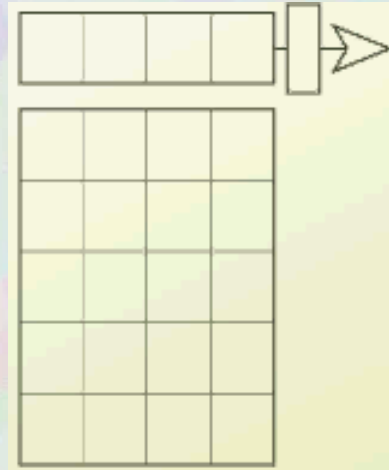


2 x 2 (650 x 510)

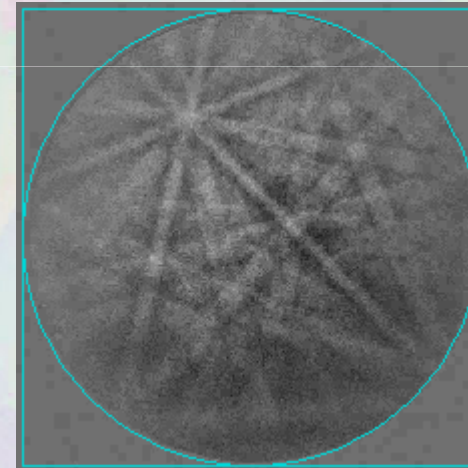


2 x 2

T



2 x 2 (650 x 510)



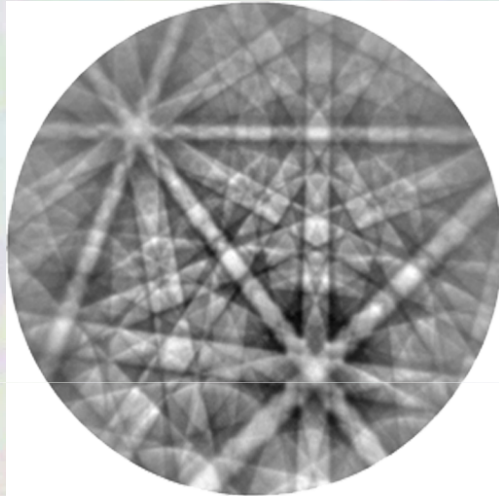
2 x 2

T/4

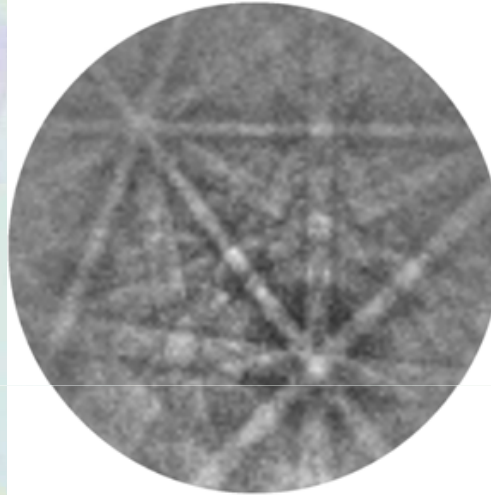
400 um

...autres possibilités diminuer HT, courant

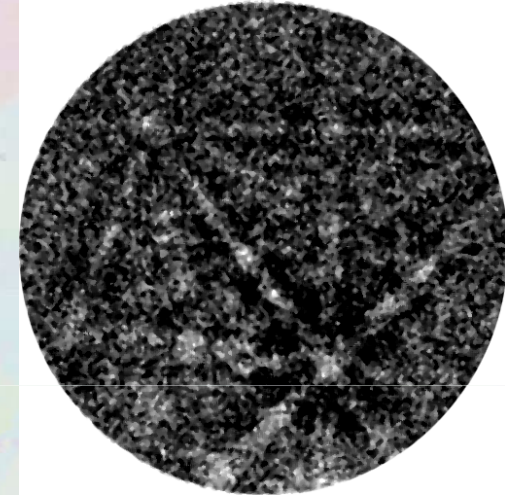
# Caméra binning / résolution



1 x 1



4 x 4



10 x 10

Avantages :

Augmenter l'intensité reçue sur le groupe de pixels

→ Aller plus vite pour lire les données

Inconvénients :

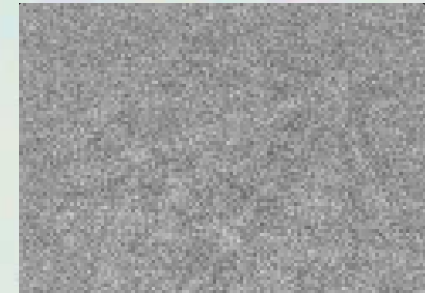
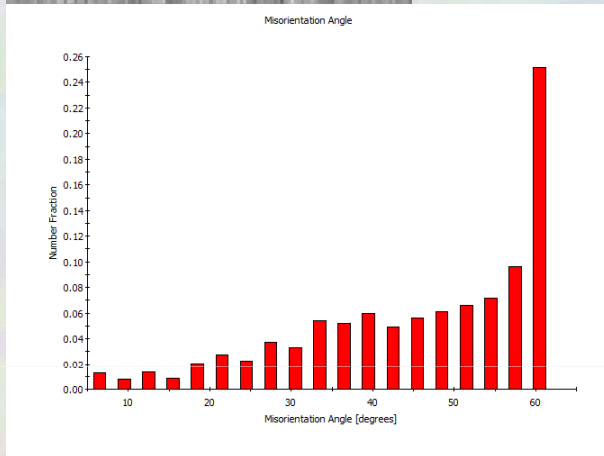
Cliché moins résolu

400 um

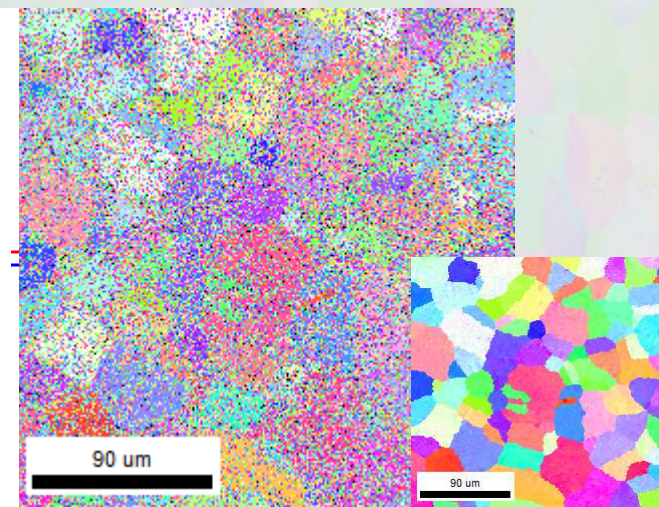
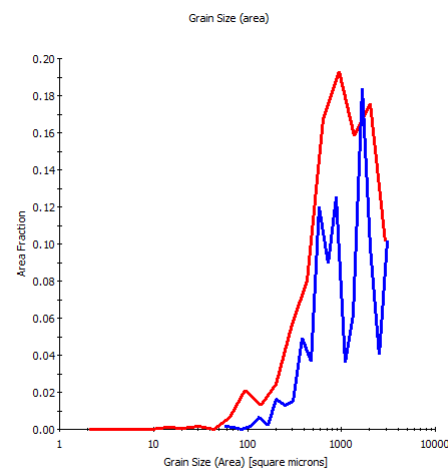
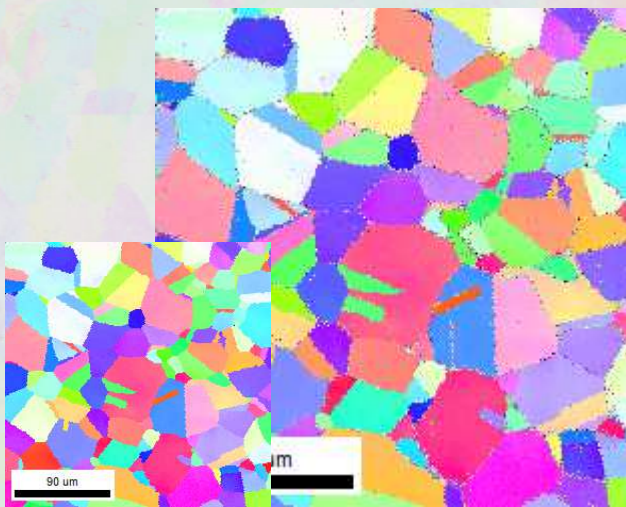
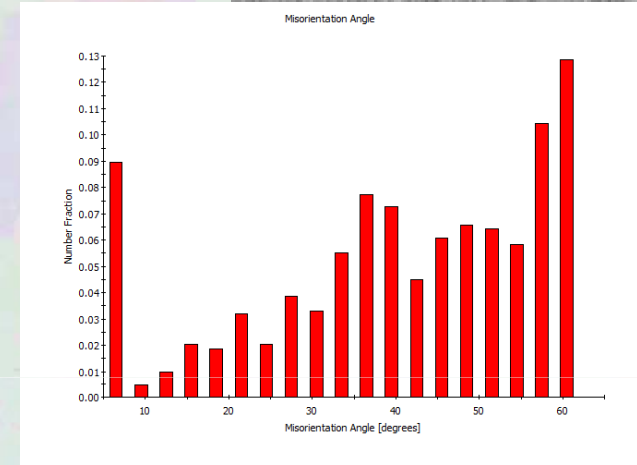
# binning 4x4, courant 0.5 nA



105

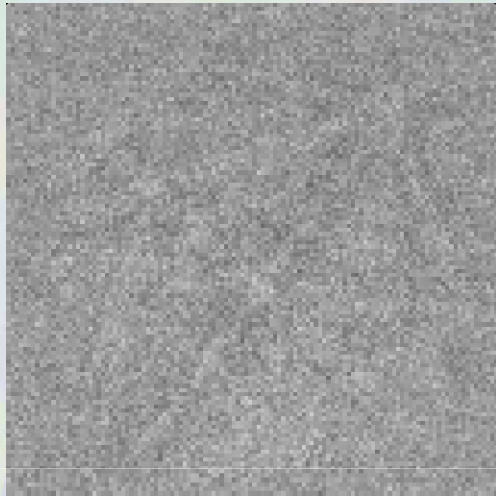


310

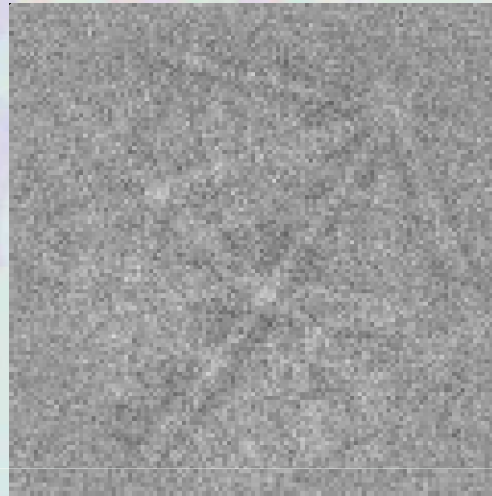


400 um

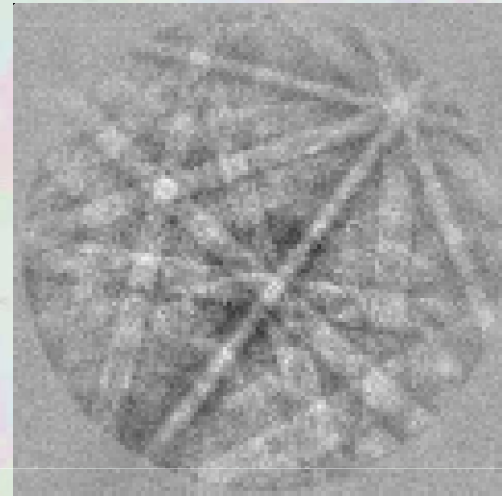
binning 4x4, 310 fr./sec.



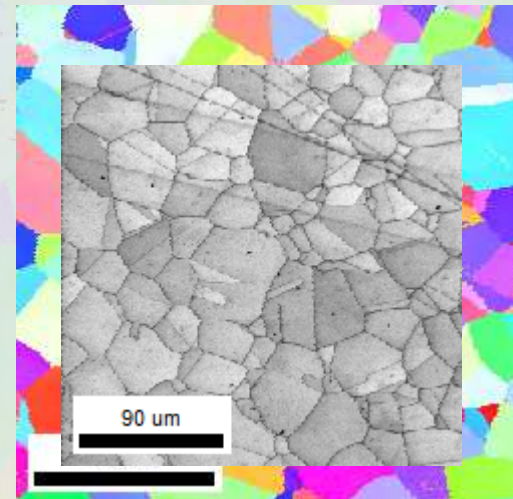
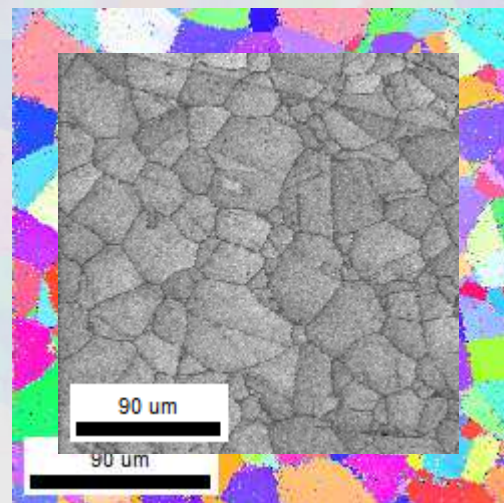
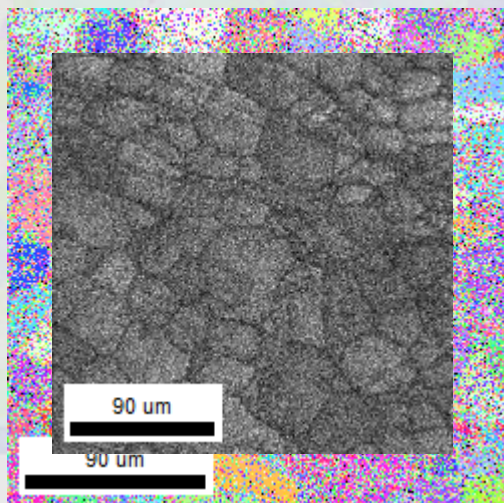
0.5 nA



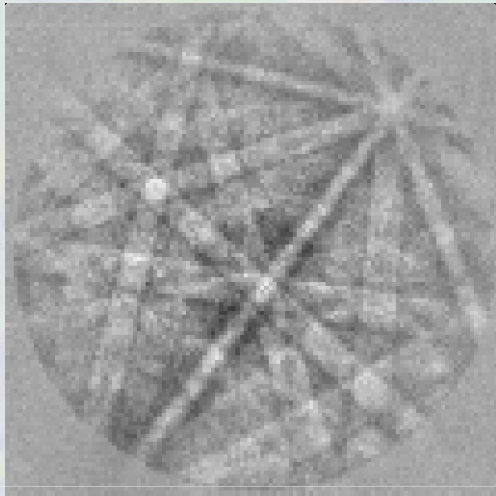
1.1 nA



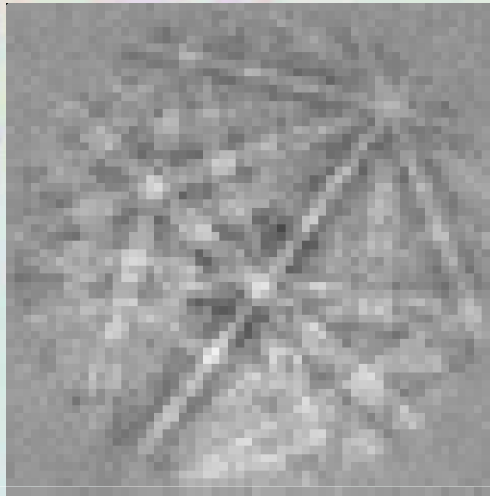
5.2 nA



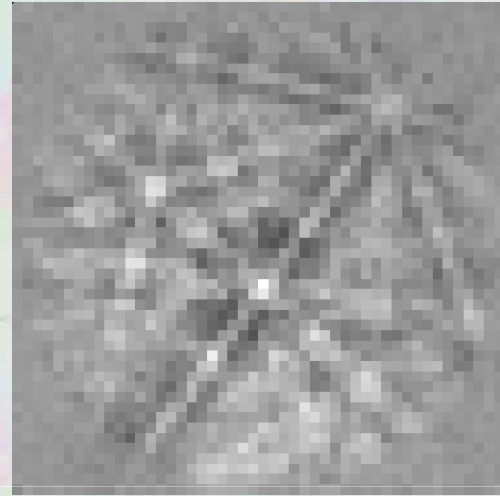
# Courant 5.2 nA – clichés intégrés



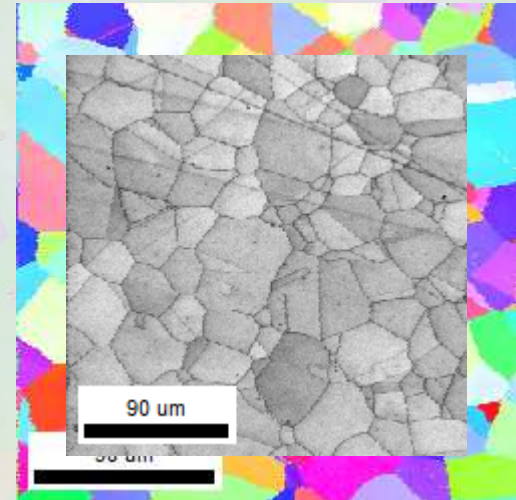
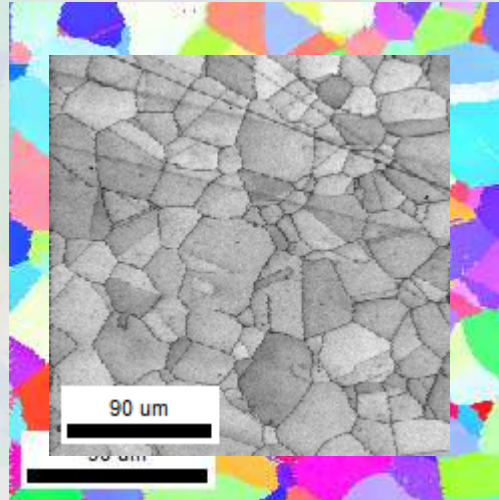
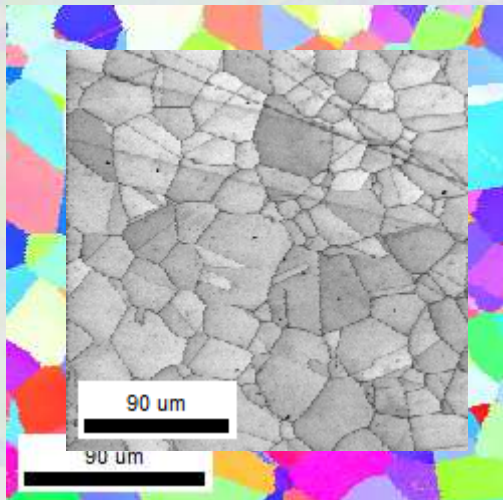
310 - 4x4



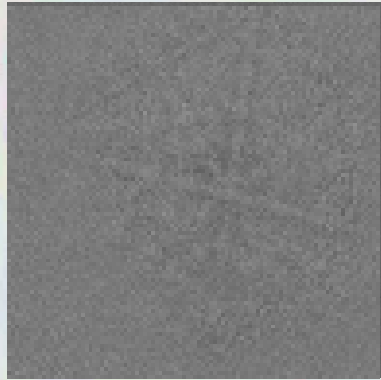
380 - 8x8



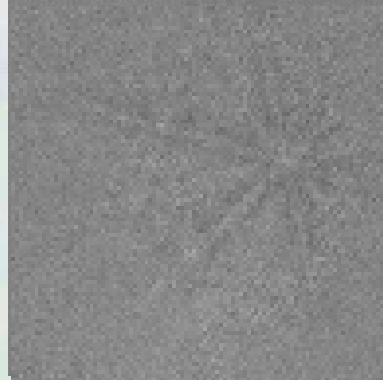
450 - 10x10



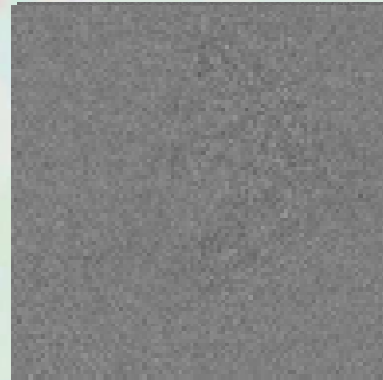
Courant 3.3 nA, binning 5 x 5



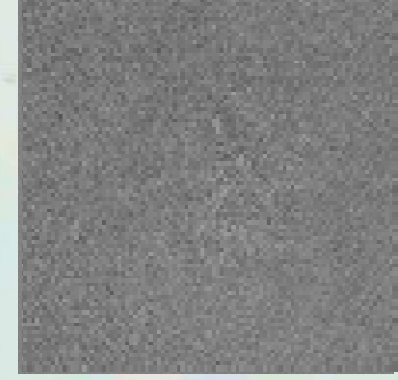
200



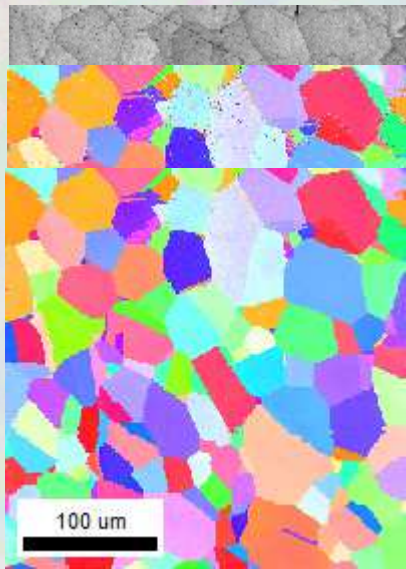
450



600



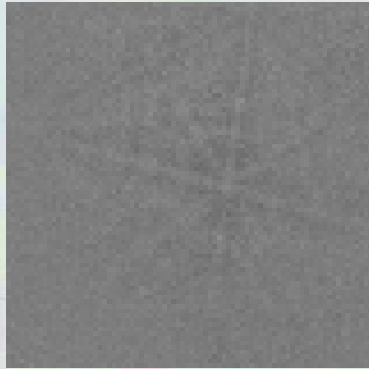
800



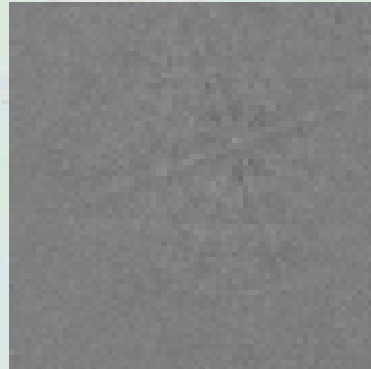
400 um



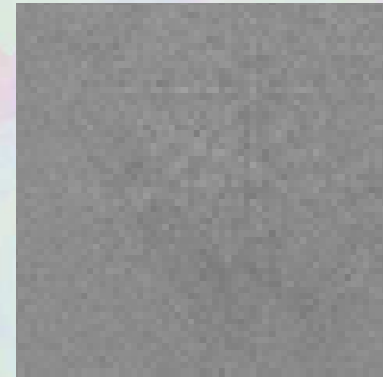
# Courant 8.8 nA



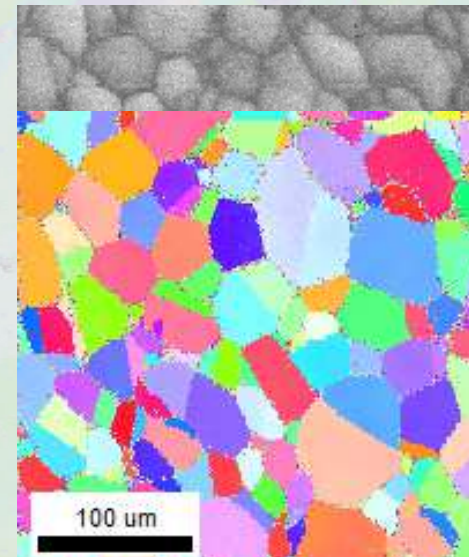
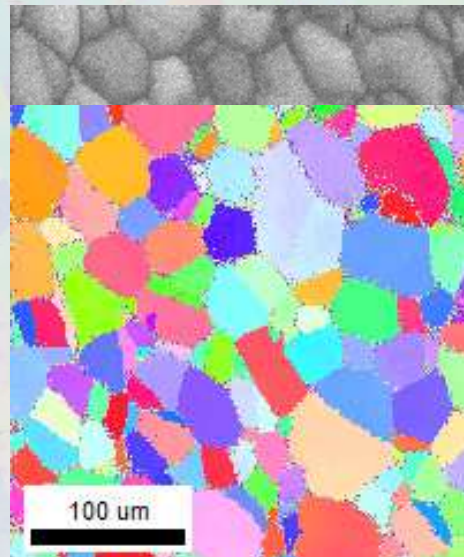
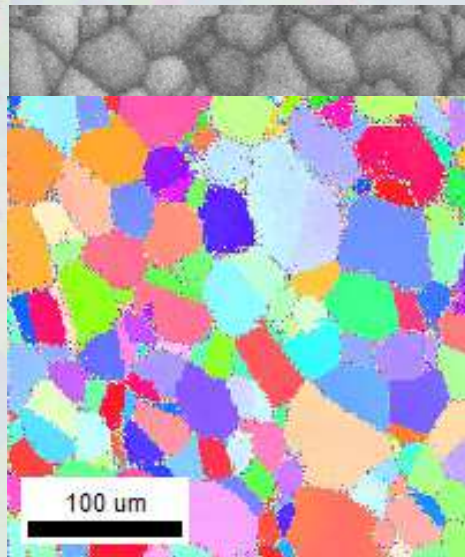
950



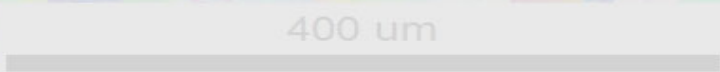
1050

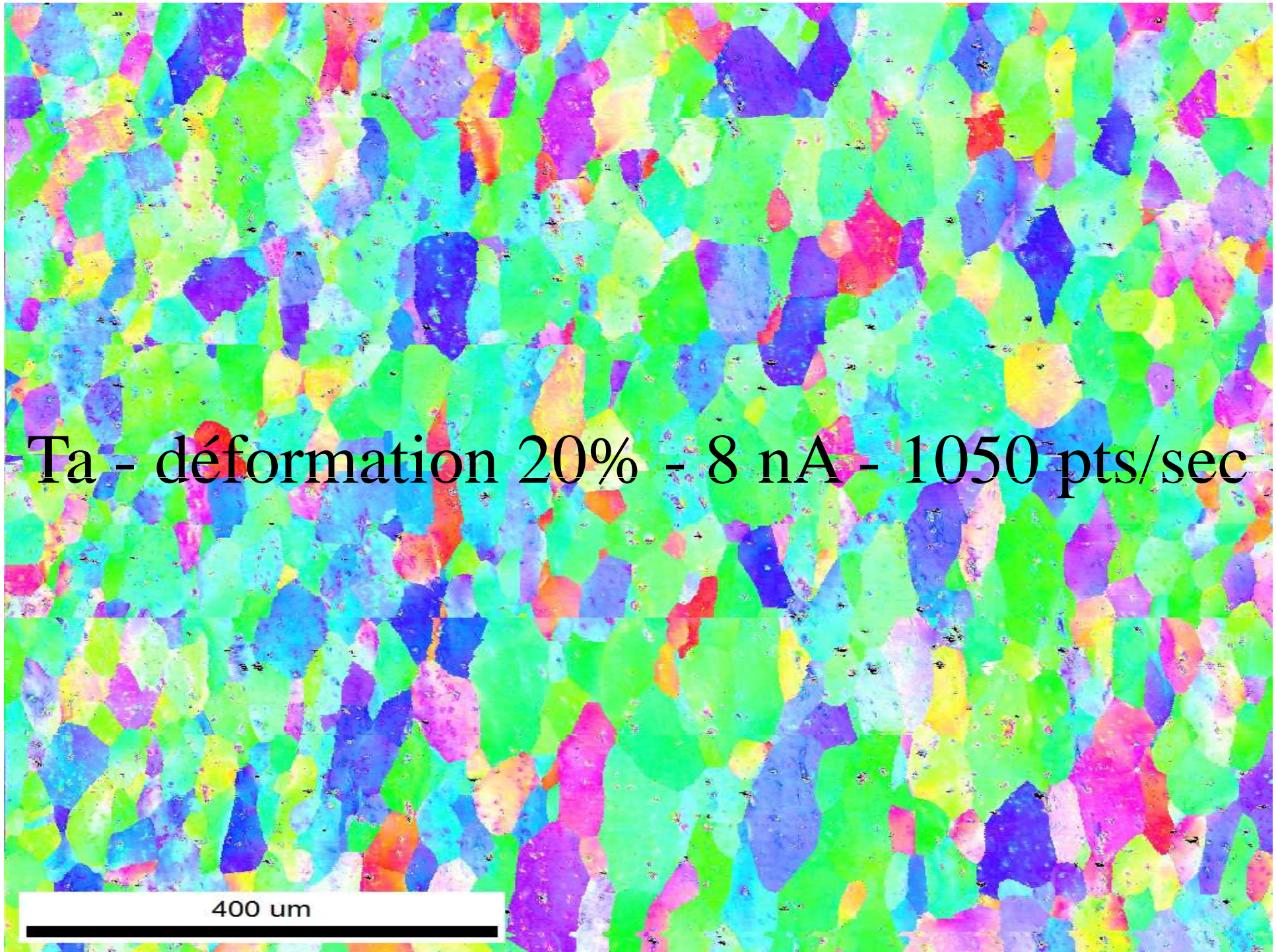


1170



400 μm

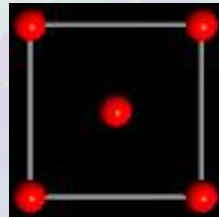
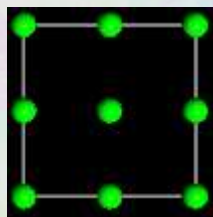
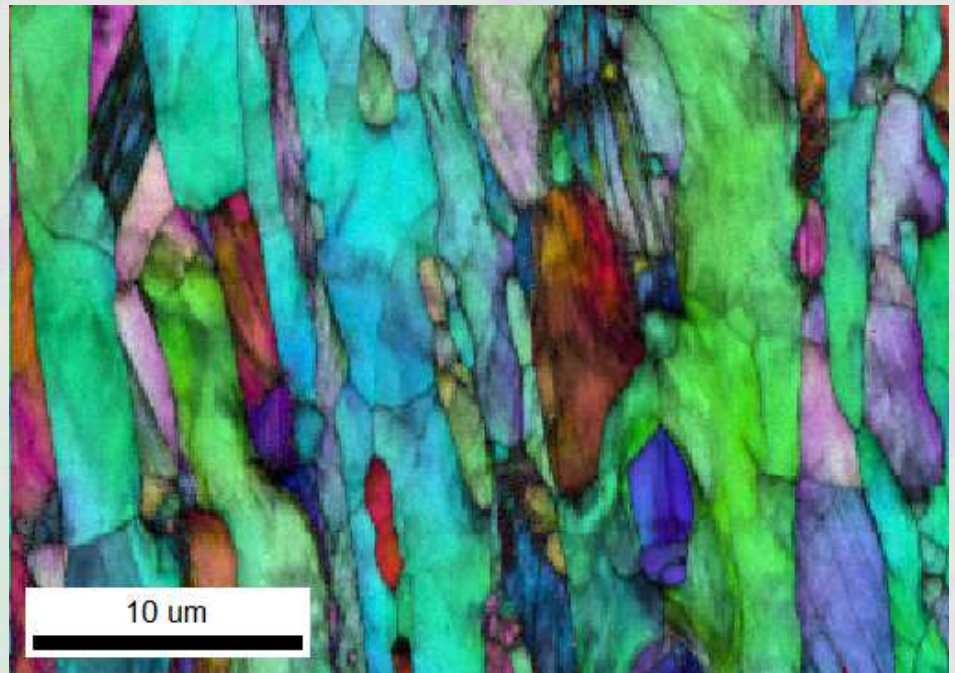
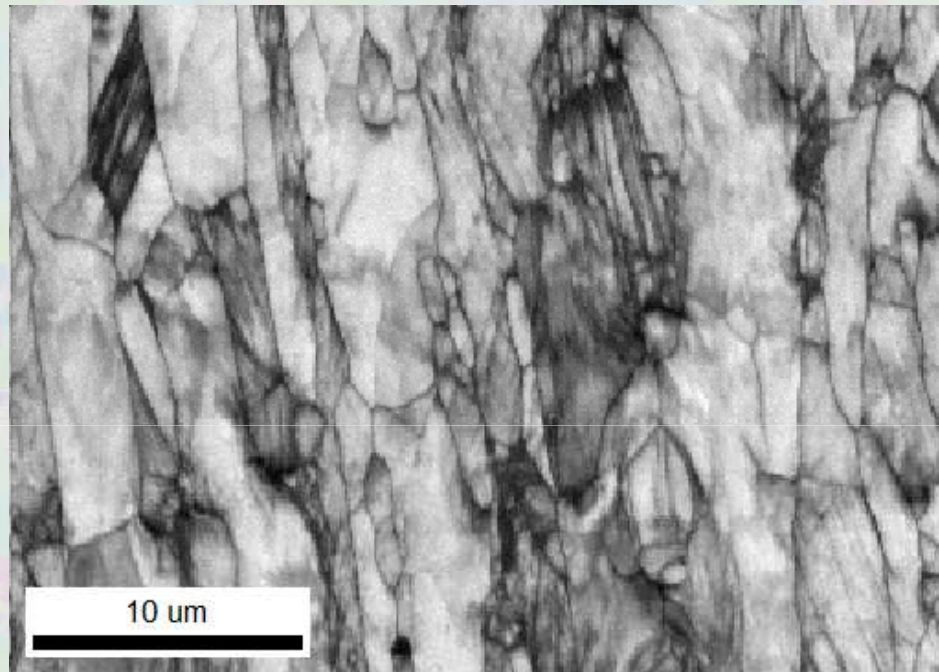




Ta - déformation 20% - 8 nA - 1050 pts/sec

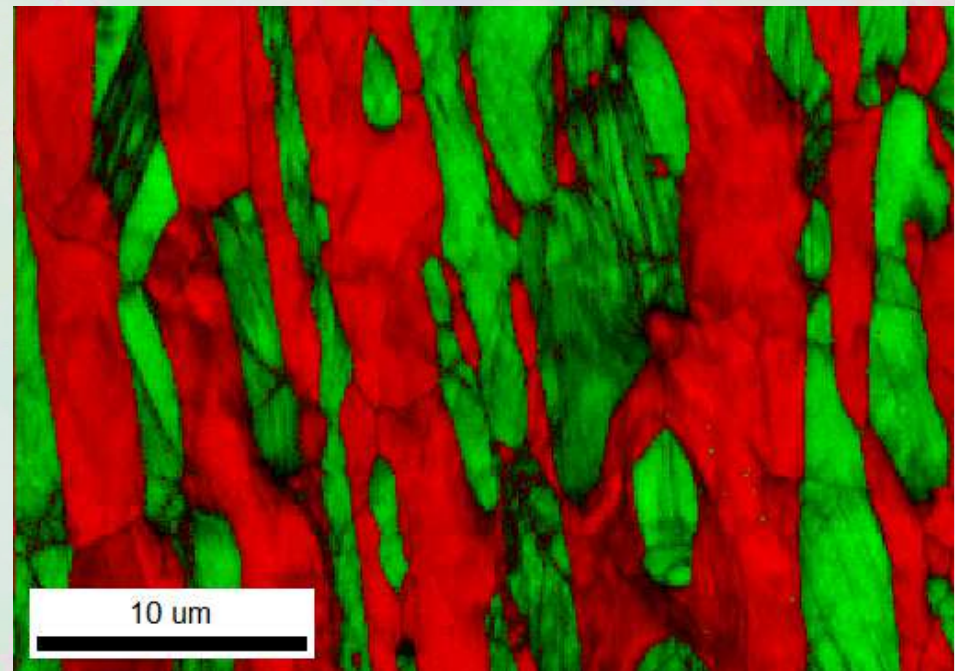
400 um

Courant 5 nA  
25 kV, binning 4x4  
pas 0.1 micron,

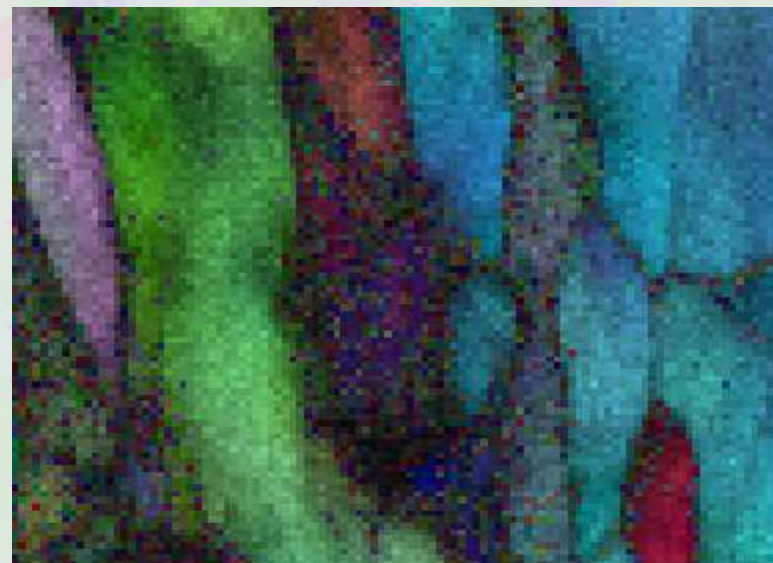
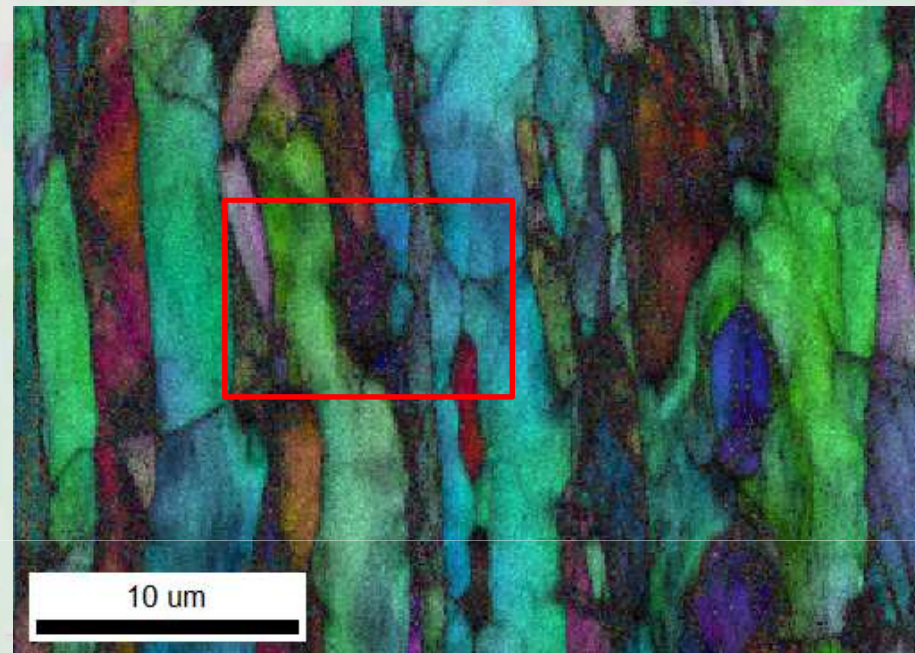
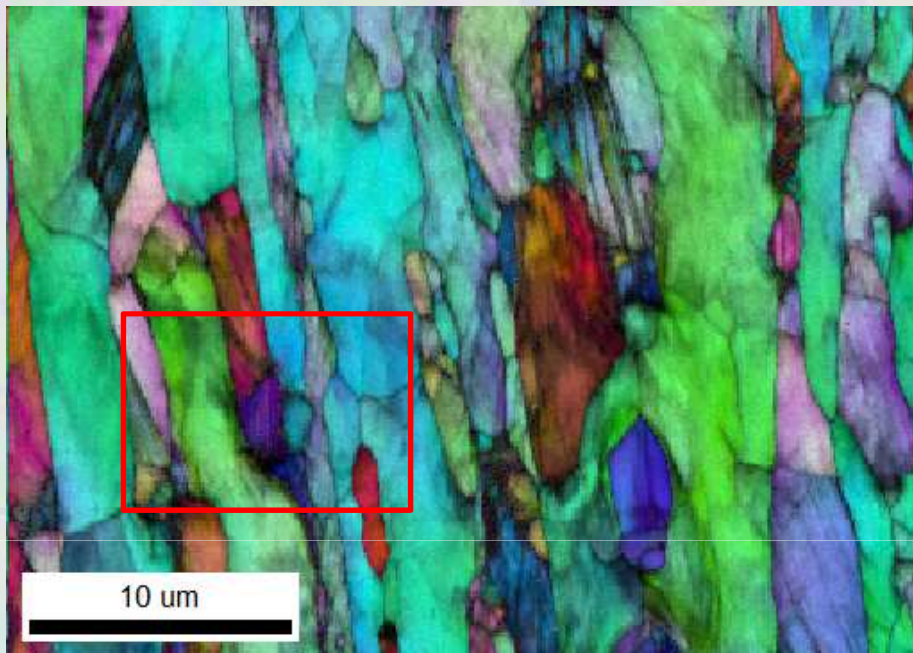


Acier duplex – 40% def.

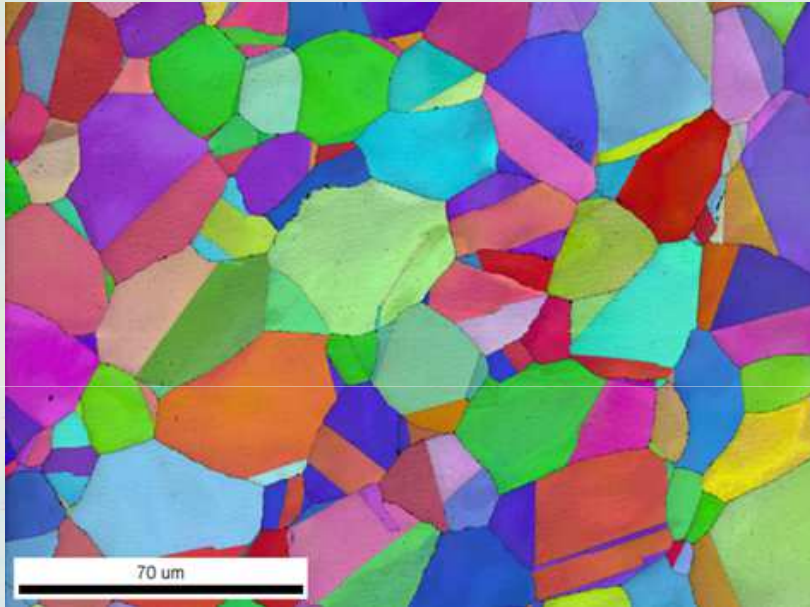
400 um



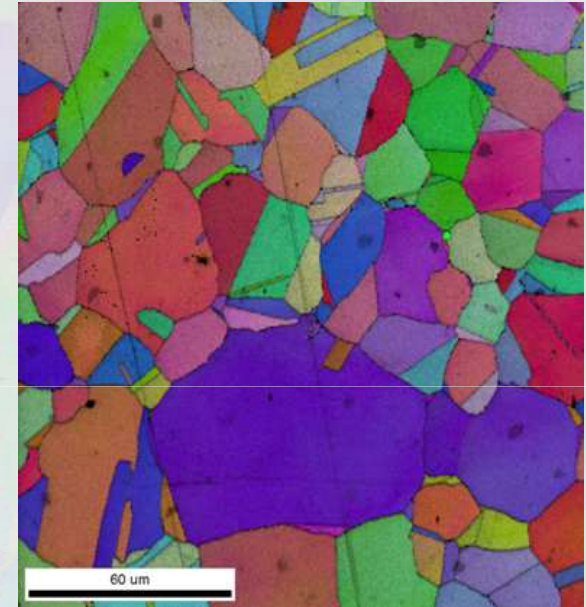
courant 5 nA et 20 nA  
25 kV, binning 4x4, pas 0.1 micron,



## 2 exemples : faible courant et faible tension



100 pA – 20 kV – 50 fr/sec



5 kV – 5 nA – 50 fr/sec

- Pourrait être utile pour des échantillons sensibles ou éventuellement non-conducteurs.
- Travailler à haute résolution – analyse de petits grains

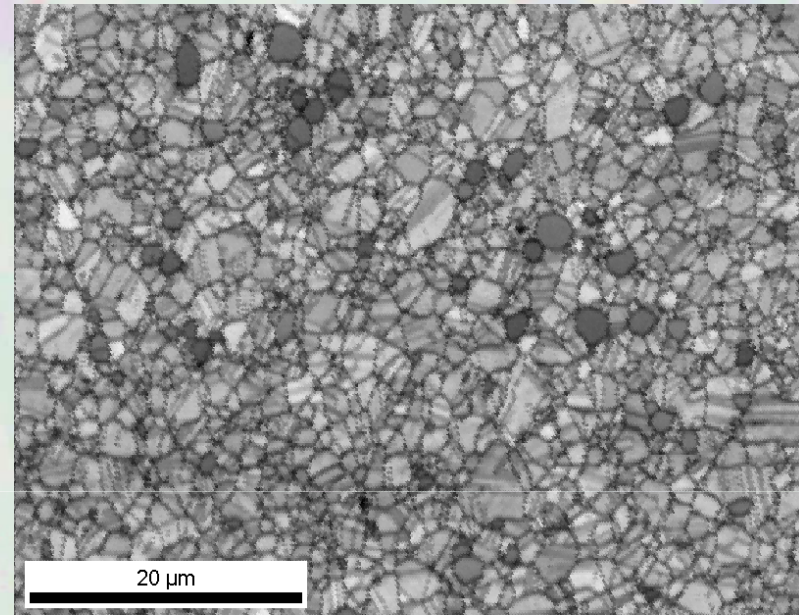
400 um

# EBSD - EDS

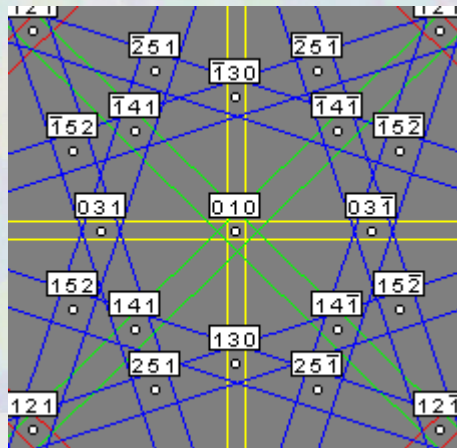
Echantillon à 3 phases

- 1) Cobalt
- 2) Carbure de chrome
- 3) Carbure de silicium

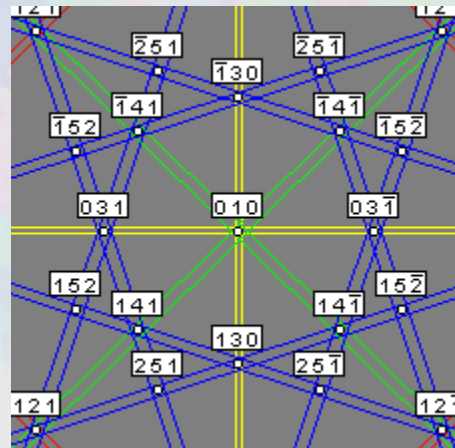
Les 3 phases sont cfc



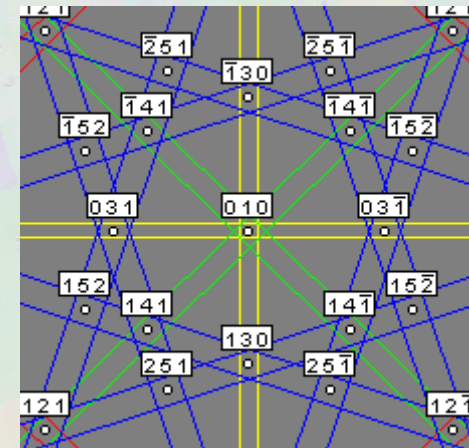
Cobalt



Carbure de chrome



Carbure de silicium



# EBSD - EDS



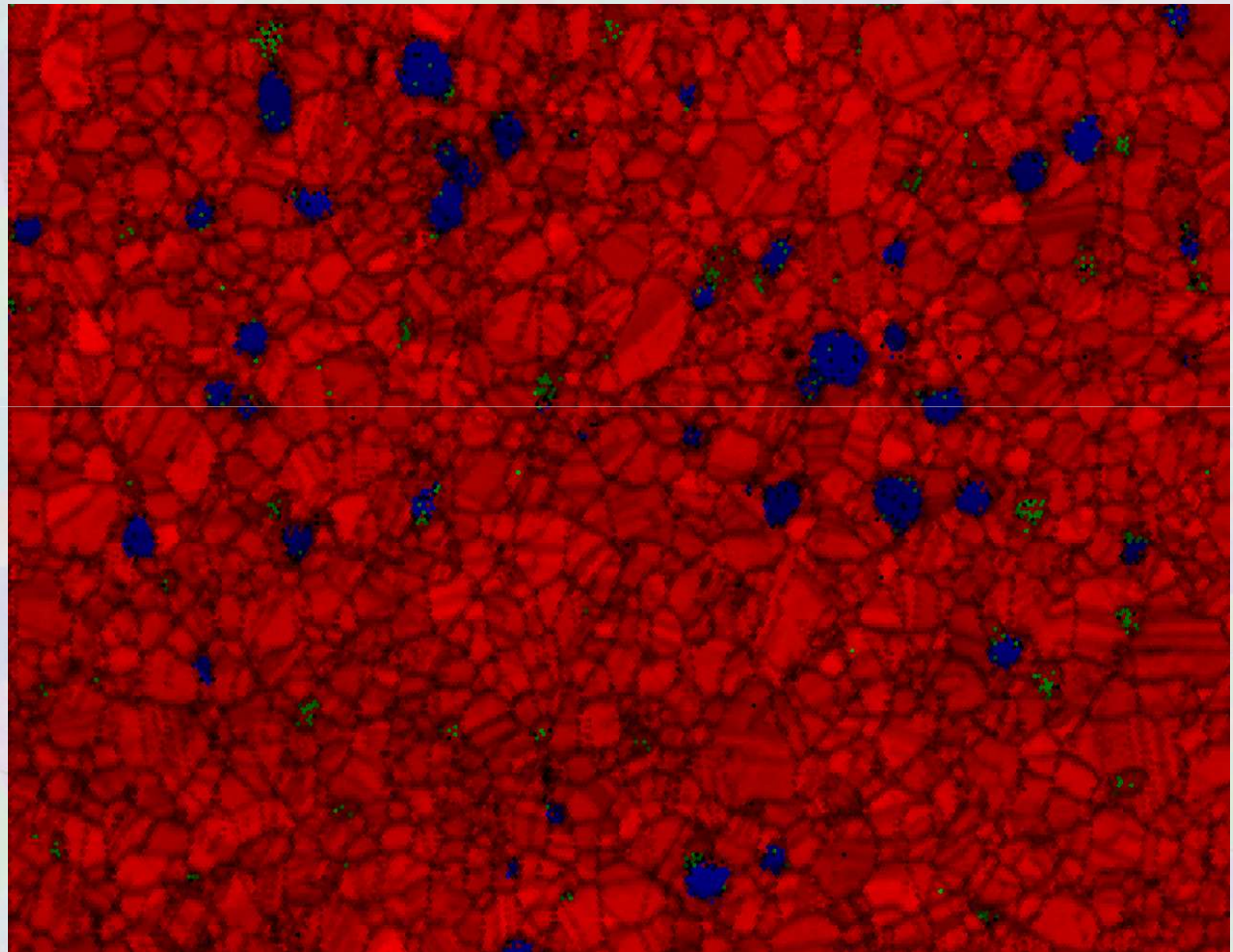
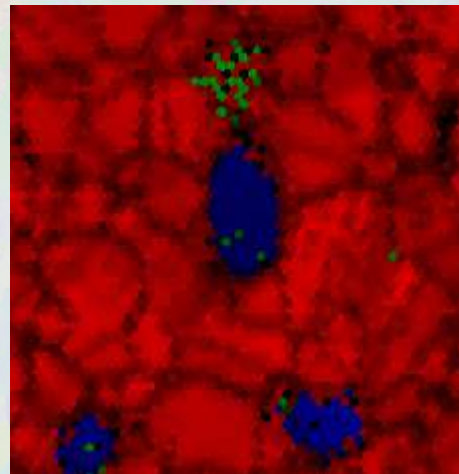
Cobalt



Carbure de chrome



Carbure de silicium

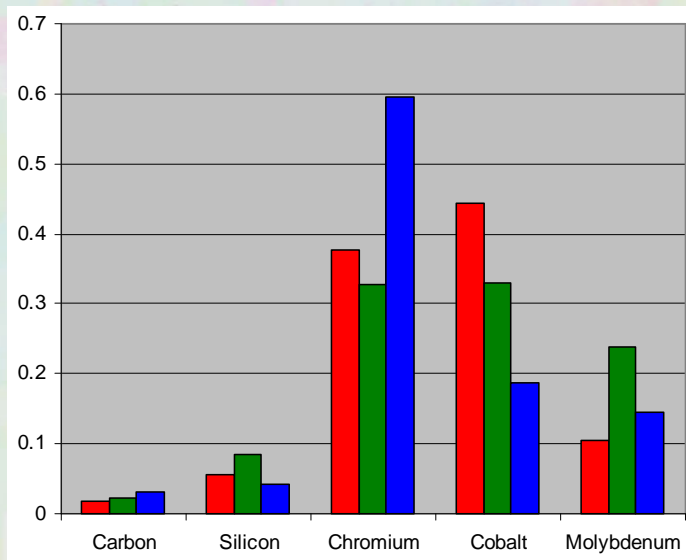
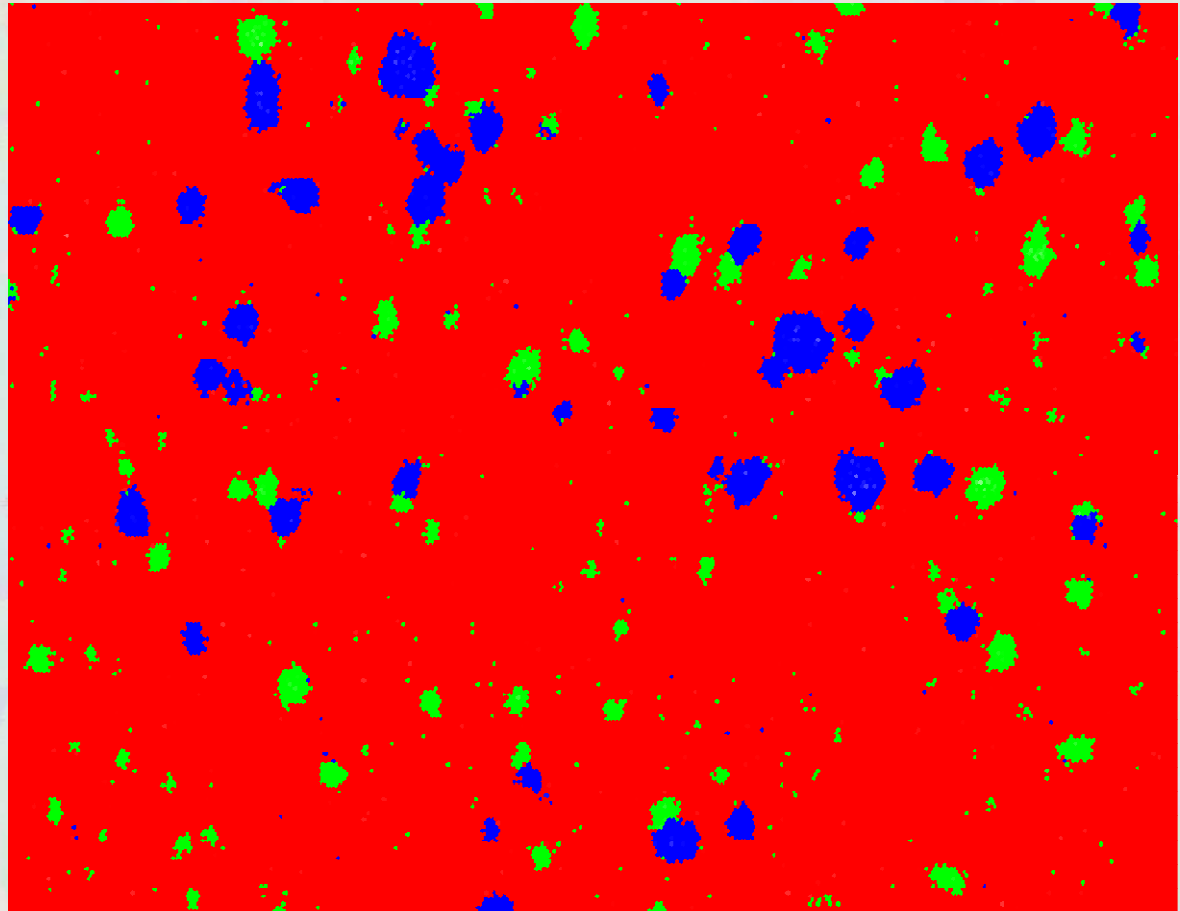


400  $\mu\text{m}$

20  $\mu\text{m}$

# EBSD - EDS

- Combiner les images
- Analyse des spectres et réaliser une analyse de phases
- Réindexer avec le filtre "EDS"



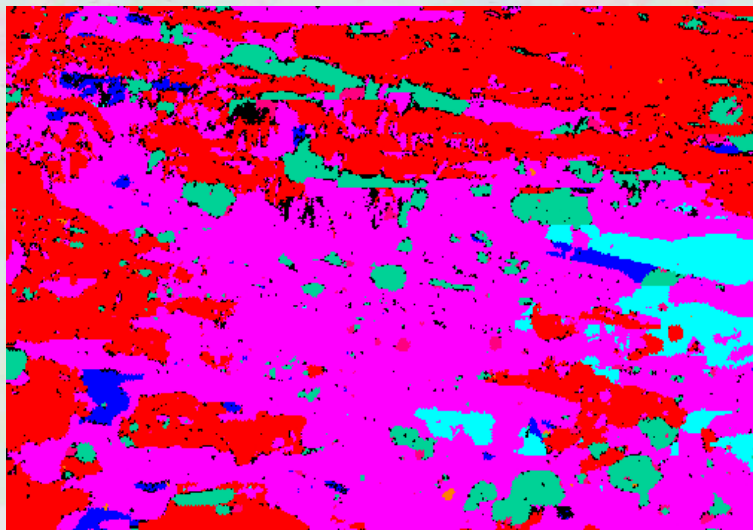
- Composé 1 > Cobalt
- Composé 2 > Carbure de chrome
- Composé 3 > Carbure de silicium

# EBSD – EDS – autre exemple

EBSD

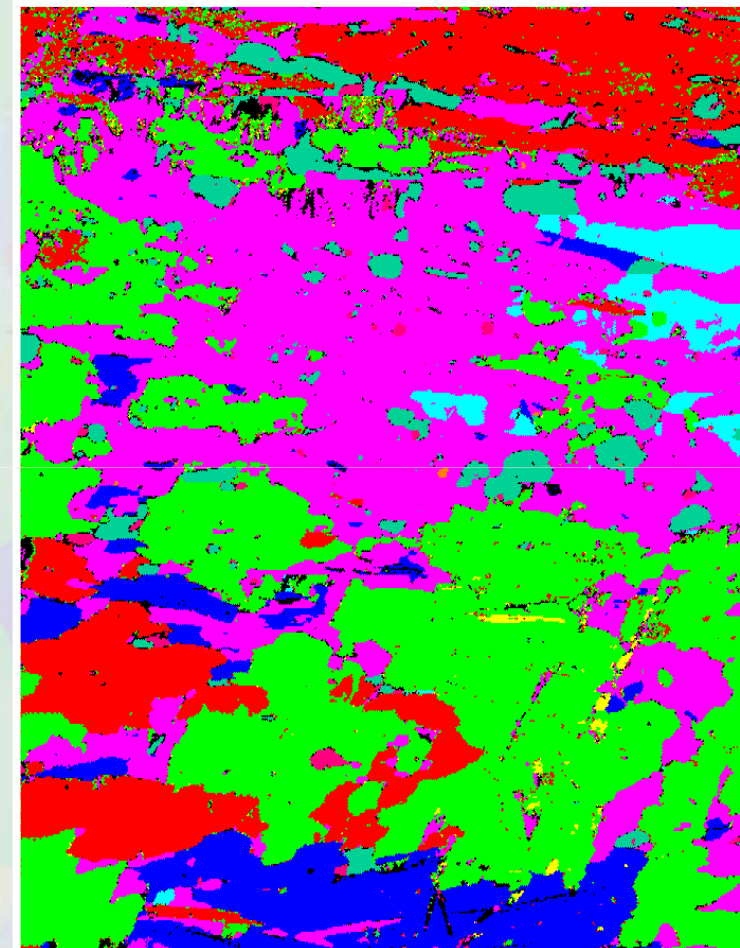


EDS



- Kyanite
- Andalusite
- Sillimanite
- Muscovite
- Quartz
- Chlorite
- Rutile
- Hematite
- Zircon

EBSD et EDS



- |   |   |  |
|---|---|--|
| <span style="display: inline-block; width: 15px; height: 15px; background-color: red; border: 1px solid black; margin-right: 5px;"></span> Kyanite        | <span style="display: inline-block; width: 15px; height: 15px; background-color: blue; border: 1px solid black; margin-right: 5px;"></span> Muscovite | <span style="display: inline-block; width: 15px; height: 15px; background-color: pink; border: 1px solid black; margin-right: 5px;"></span> Rutile   |
| <span style="display: inline-block; width: 15px; height: 15px; background-color: green; border: 1px solid black; margin-right: 5px;"></span> Andalusite   | <span style="display: inline-block; width: 15px; height: 15px; background-color: magenta; border: 1px solid black; margin-right: 5px;"></span> Quartz | <span style="display: inline-block; width: 15px; height: 15px; background-color: grey; border: 1px solid black; margin-right: 5px;"></span> Hematite |
| <span style="display: inline-block; width: 15px; height: 15px; background-color: yellow; border: 1px solid black; margin-right: 5px;"></span> Sillimanite | <span style="display: inline-block; width: 15px; height: 15px; background-color: cyan; border: 1px solid black; margin-right: 5px;"></span> Chlorite  | <span style="display: inline-block; width: 15px; height: 15px; background-color: orange; border: 1px solid black; margin-right: 5px;"></span> Zircon |

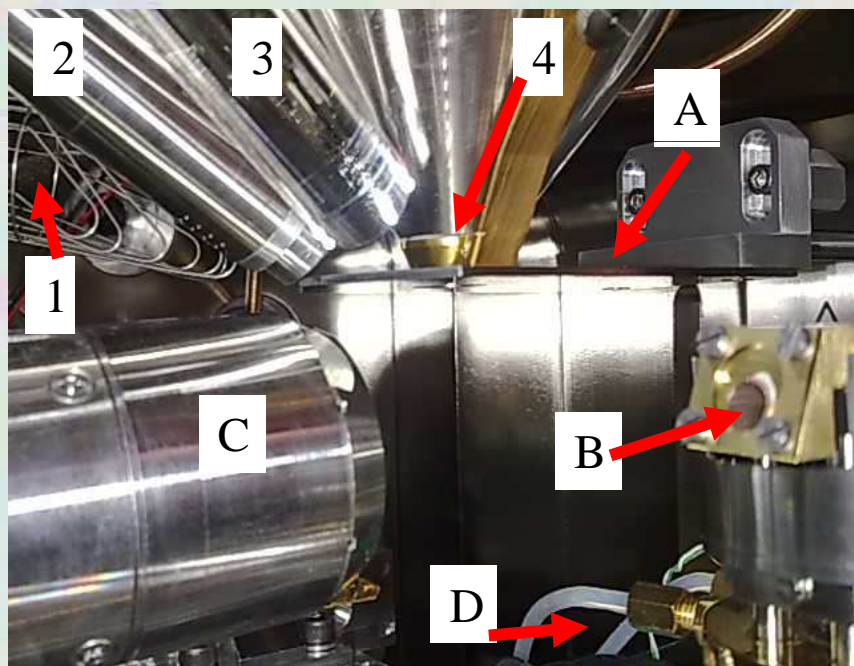
# In-situ hot stage



← Hot stage and copper sample

External assembly →

↓ Internal view

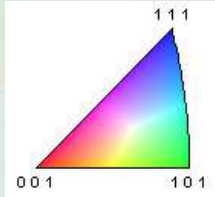


- A – Polar piece protection shutter.
- B – Cu sample fixed on the heating stage (not fully inserted).
- C – EBSD camera (fully inserted) and forscattered electron detector.
- D – Water cooling system of the base plate.
- 1 – SE detector
- 2 – EDS detector
- 3 – VPSE detector
- 4 – in-cup BSE detector

H1002D Gatan heating stage (up to 750°C)  
TSL Hikari OIM™ EBSD system

Zeiss Supra 55 VP

# Deformed state



EBSD maps of the cold drawn wire (copper fcc)

- Average grain size is about 15 μm

- Texture is mainly composed of:

$\langle 001 \rangle // \text{ND}$

and

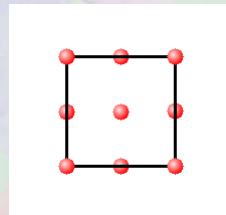
$\langle 111 \rangle // \text{ND}$  fibres

(ND the normal direction to the wire section)

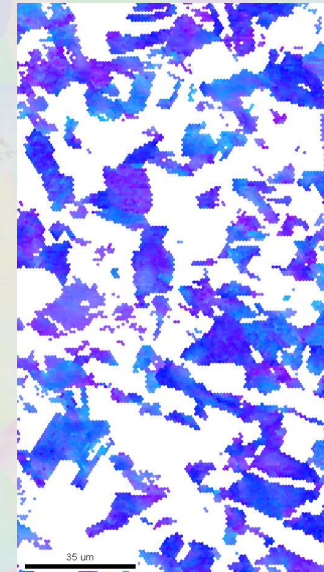
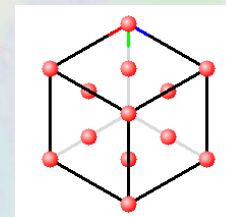


Distribution of {hkl} planes

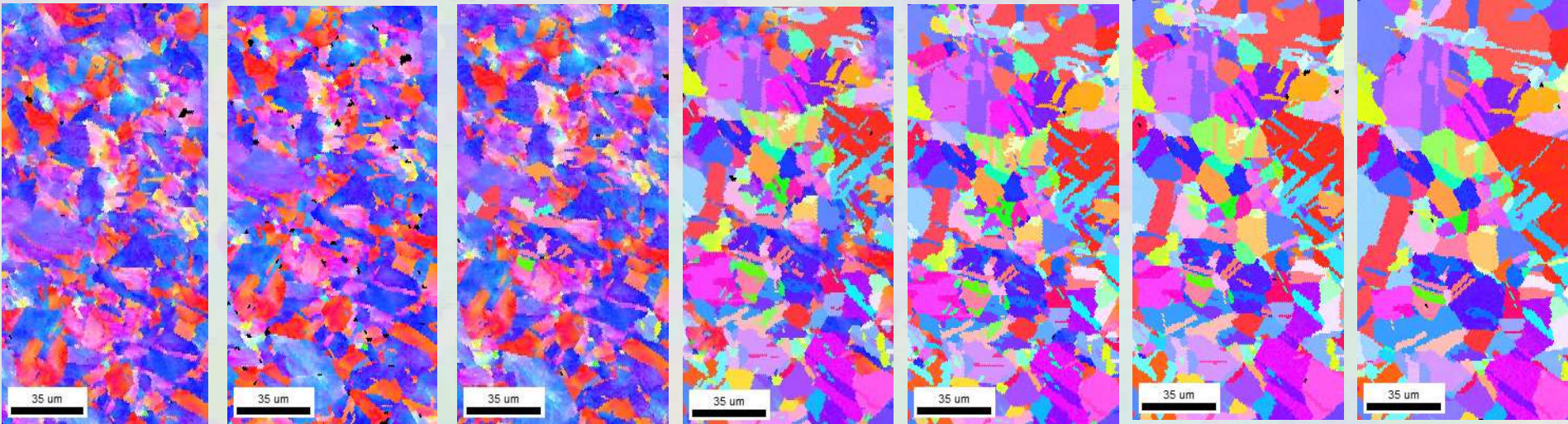
$\langle 001 \rangle // \text{ND}$  fibre



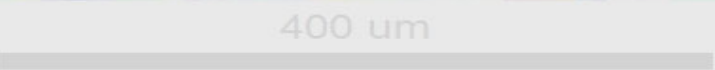
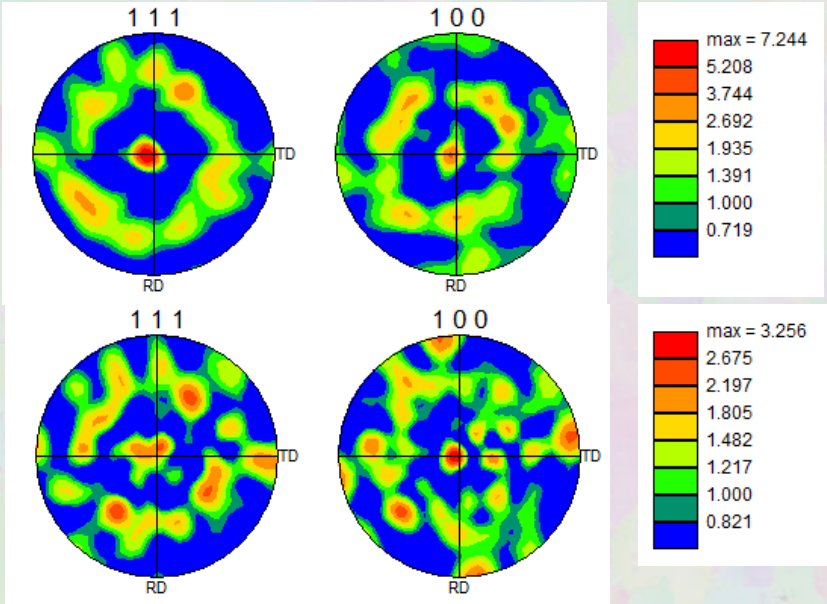
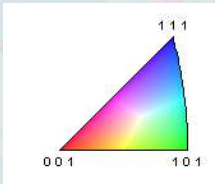
$\langle 111 \rangle // \text{ND}$  fibre



# In-situ sequence



Initial state    150°C    200°C    230°C – 2min.    230°C – 7min.    400°C    500°C



Top: initial state, Bottom: 230°C – 2 min.