

CHORD or how to get crystalline orientation maps using the electron or ion channelling contrast

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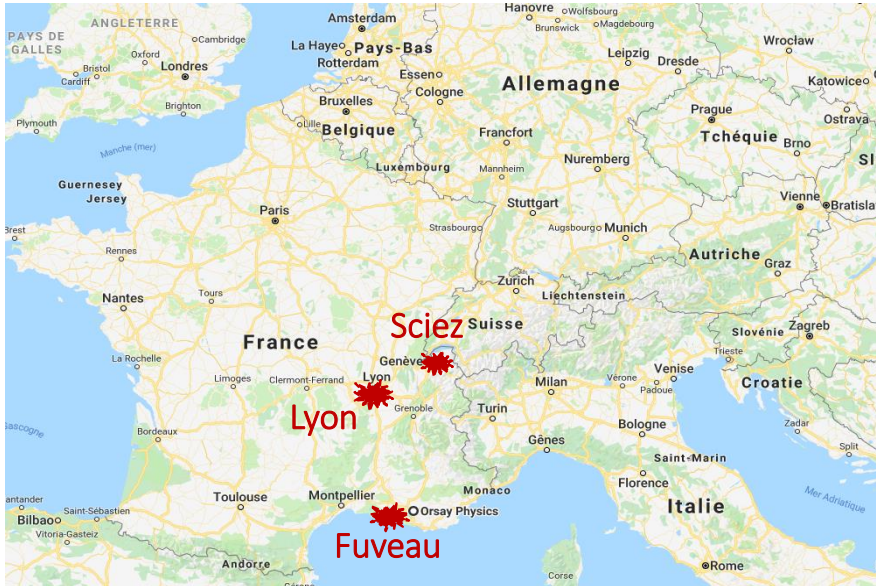
¹MATEIS laboratory – INSA Lyon, France ²Axon Square Ltd, Sciez, France ³Orsay Physics, Fuveau, France



- **CHORD presentation**
- **A few exemples**
- **Perspectives**
- **Live acquisition**



Locations and the team



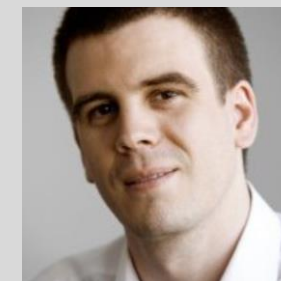
Jérémie Silvent
Application engineer



Cyril Langlois/ Thierry Douillard
Co-Inventors of the CHORD patent



Clément Lafond
Post-Doc



Sébastien Dubail
CEO, founder



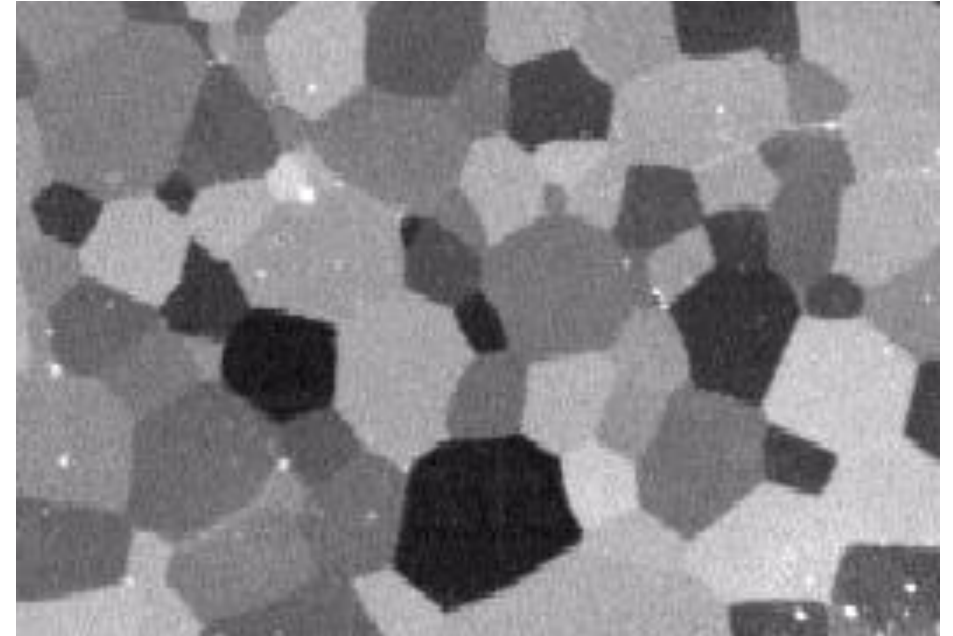
CHORD Principle

→ Goal : obtain an **orientation map**

CHORD : **CH**anneling **OR**ientation **D**etermination

electrons
↓
e-CHORD

ions
↓
i-CHORD

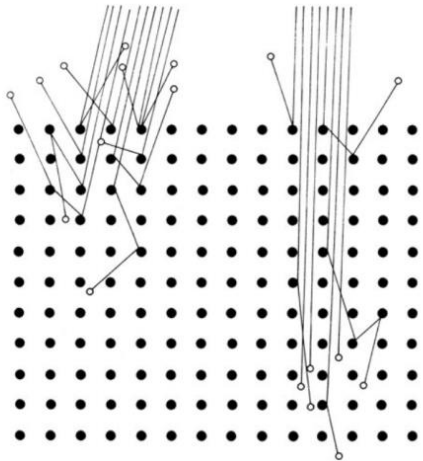


TiN – image Ion Ga⁺ 30kV 3nA



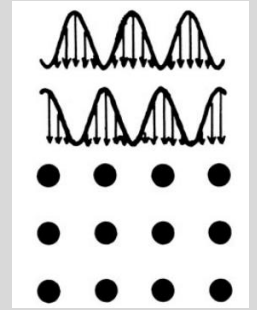
Channeling Contrast

The channeling of the incident beam by crystallographic planes is responsible for the grey level difference between differently oriented grains in a polycrystalline material. This channeling contrast may be used to obtain orientation maps.



If the ion beam is parallel to low index planes, the secondary electron are generated far under the surface. A low intensity is then detected.

With an electron beam, the intensity received by the detector is monitored by back scattered diffraction.



Intensity variation as a function of sample tilt

Crystallographic orientation contrast associated with Ga^+ ion channelling for Fe and Cu in focused ion beam method

Y. Yahiro, K. Kaneko, T. Fujita, W.-J. Moon and Z. Horita*

Journal of Electron Microscopy 53(5): 571-576 (2004)

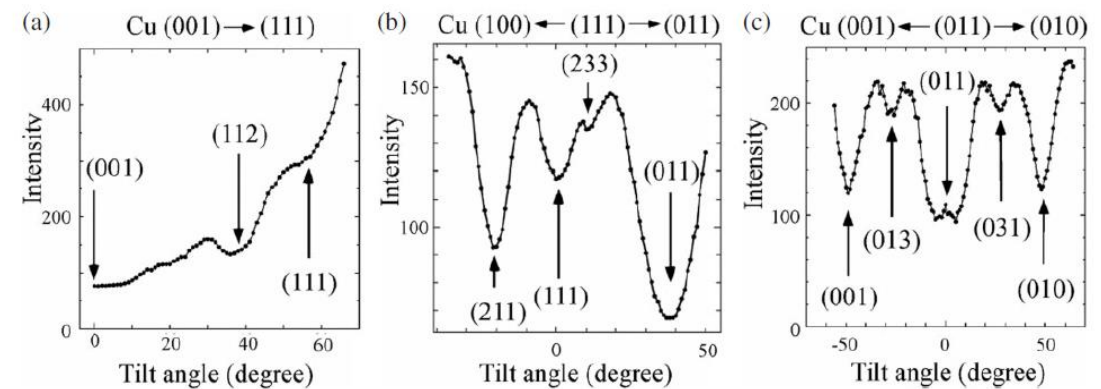
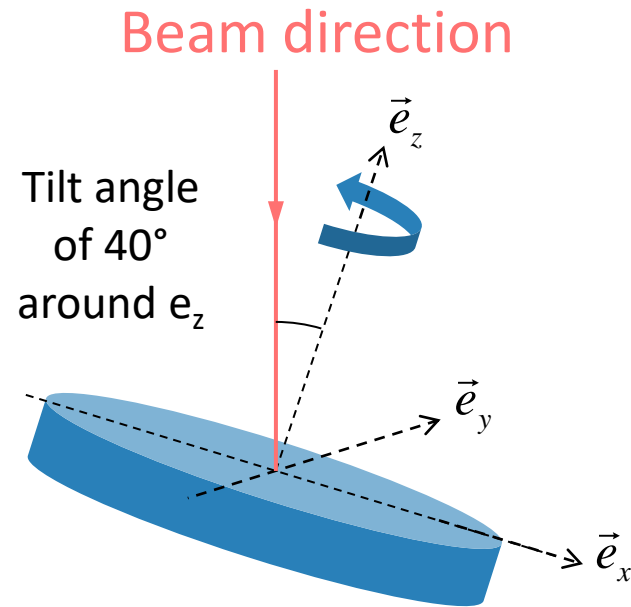
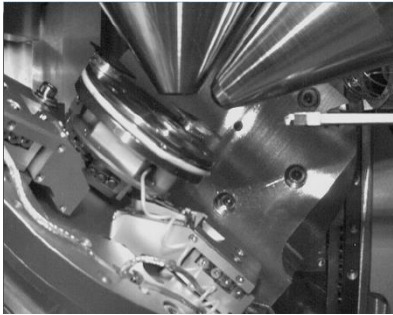


Fig. 2 Variation of the secondary electron intensity with respect to the tilt angle for Cu: (a) tilting direction from (001) to (111), (b) tilting direction from (111) to (011) and (100) and (c) tilting direction from (011) to (001) and (010).

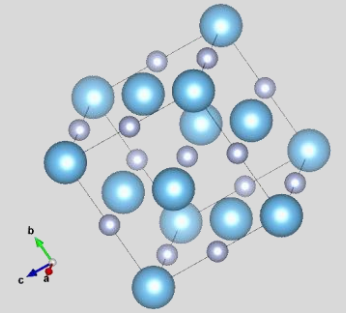
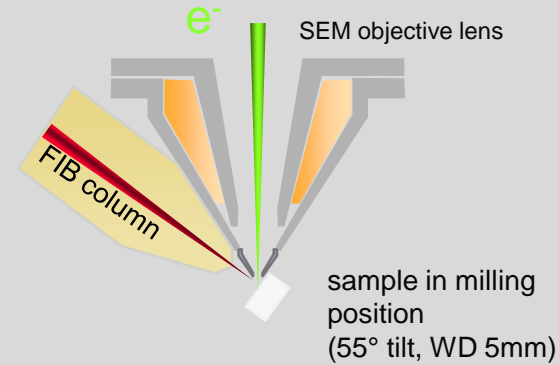


i-CHORD Acquisition

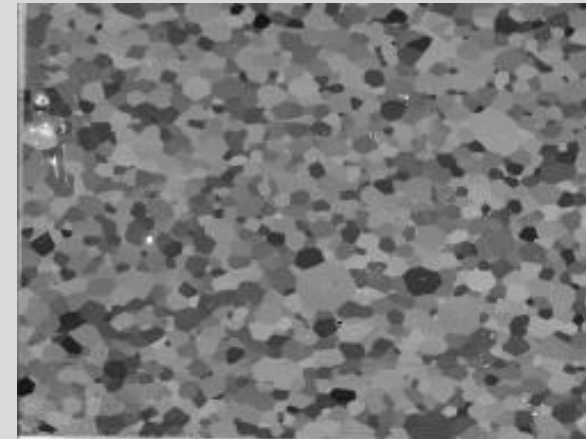
How to vary the orientation ?



- Starting point : ion beam perpendicular to the sample surface
- Then 40° sample tilt \rightarrow 40° between the beam and the sample normal direction
- Then rotation of the sample around the tilted normal \rightarrow one image acquisition every rotation step (automated)



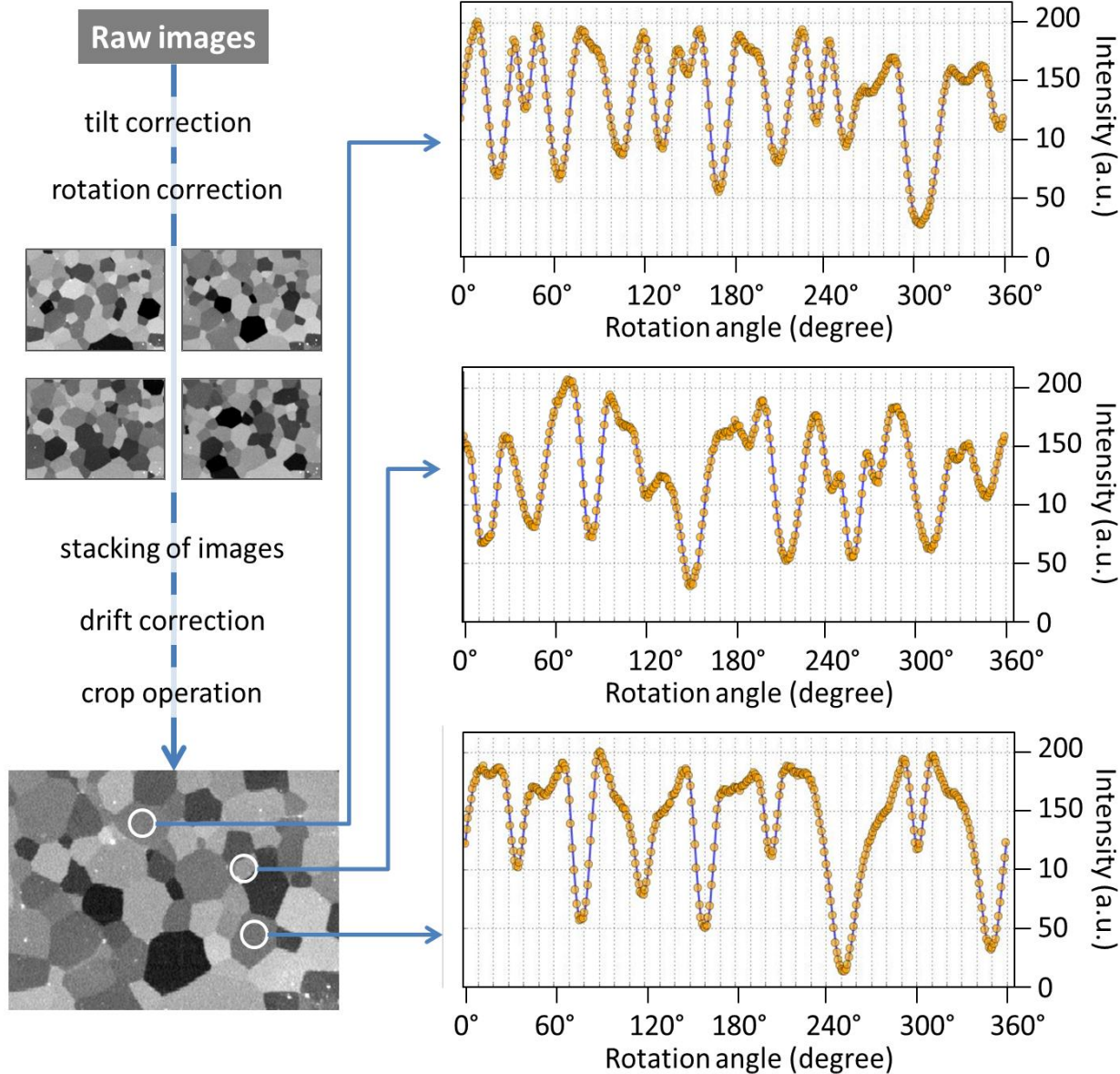
Sample : titanium nitride (TiN)
fcc structure



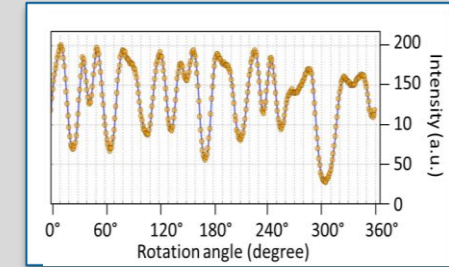
RAW IMAGE SERIES



From raw images to intensity profiles



From Intensity profiles to Euler angles



The idea :

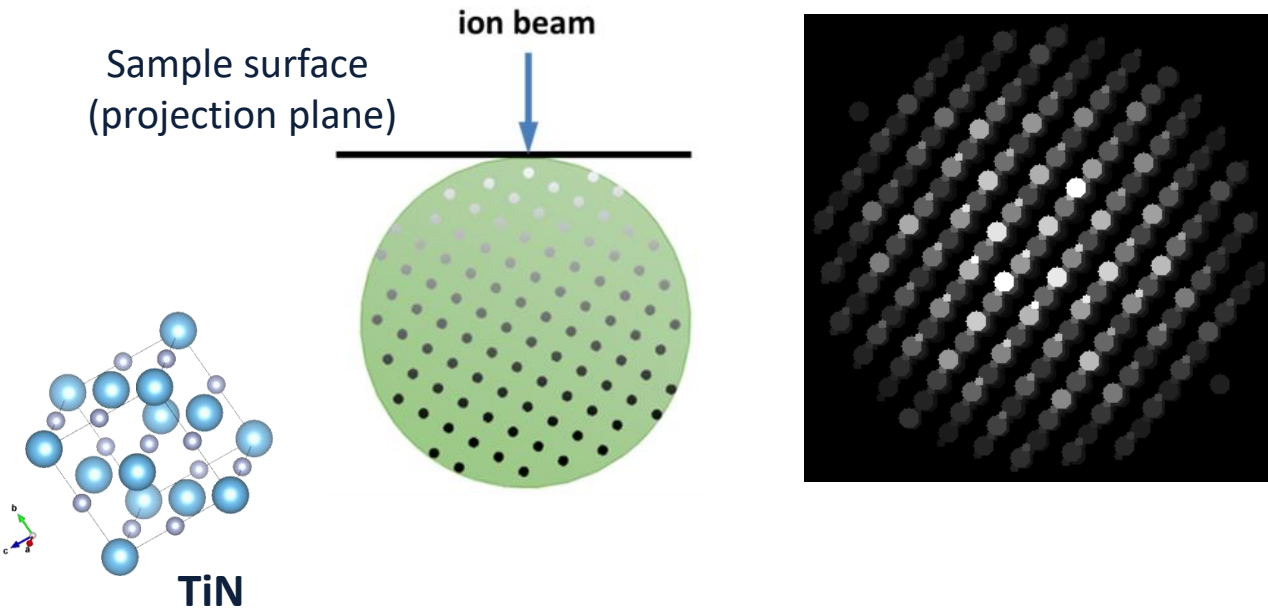
- constituting a profile database
- exploring the database to find the closest profile



Generation of theoretical profiles



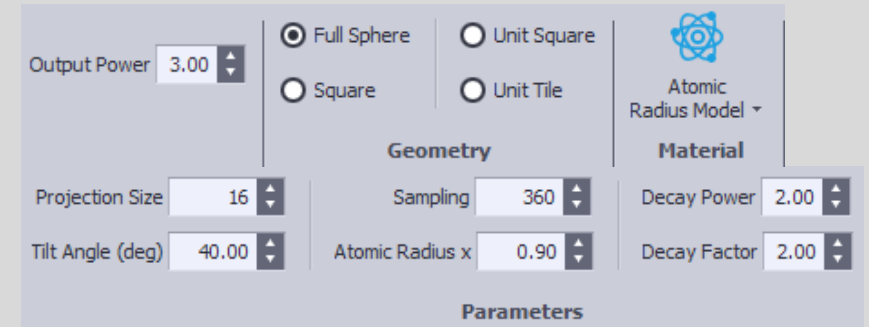
Theoretical profile construction



Relationship between the « shadow » of the structure and the intensity

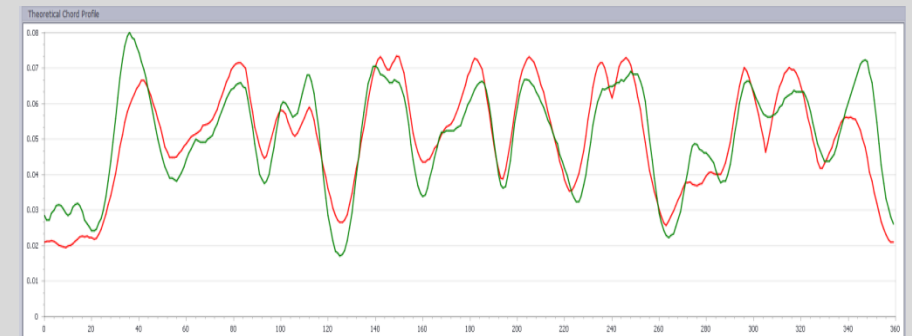
Intensity proportional to the sum of pixels grey levels in the projection

- **Spherical shape** to avoid edge effect when projecting the shadow
- **Ponderation as a function of depth**



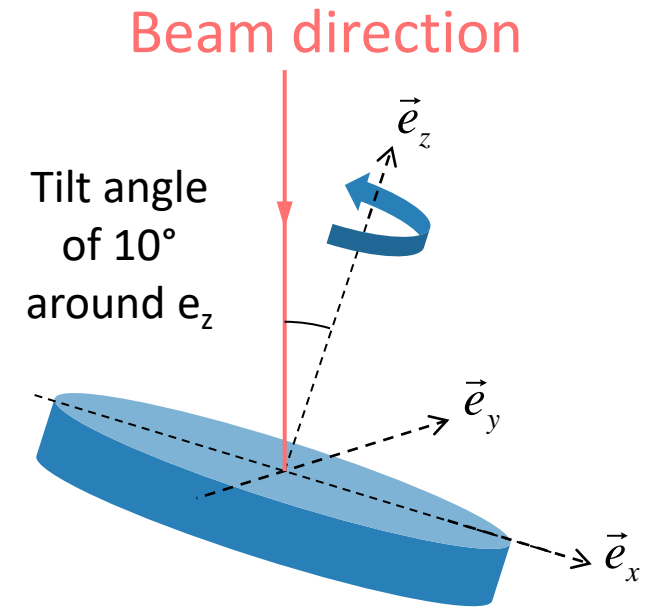
Parameters representative from ion – matter interactions

- **Normalised profiles**

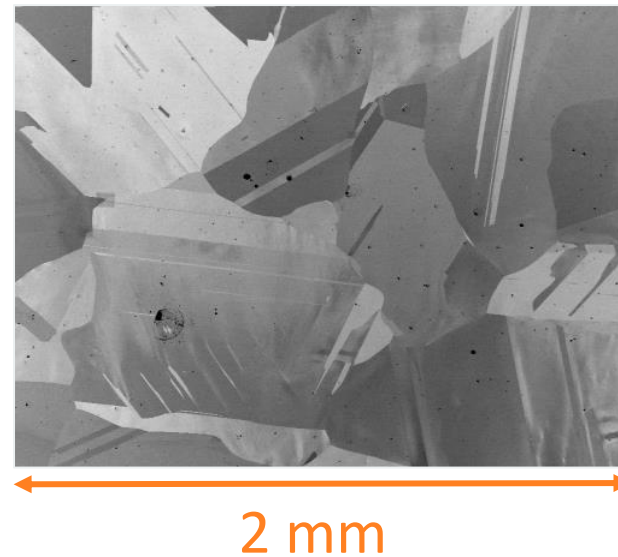


e-CHORD Acquisition

- Starting point : electron beam perpendicular to the sample surface
- Then tilting the sample: 10°
- Then rotation of the sample around the tilted normal
- One image acquisition every rotation step (automated)
- Automated correction of rotation

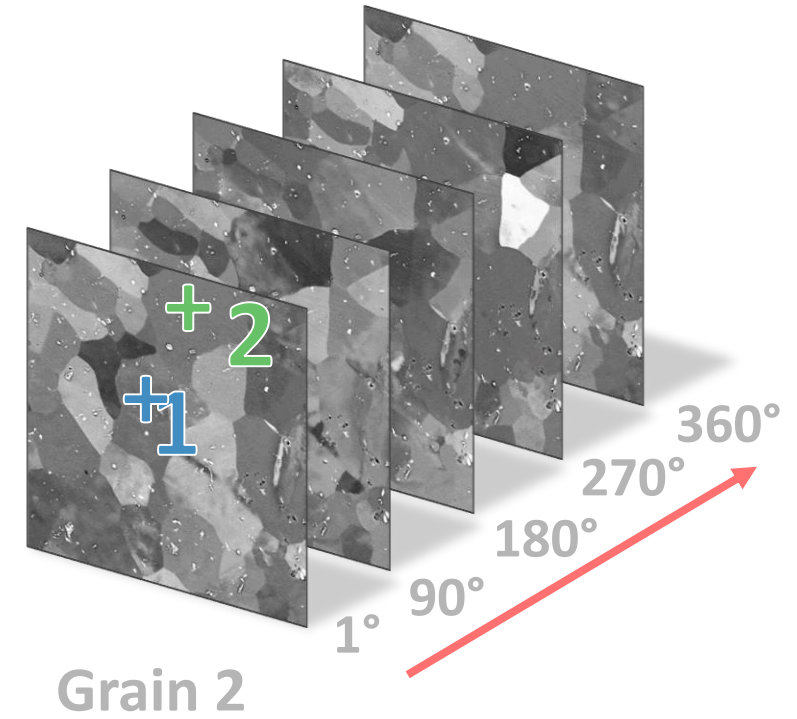


Copper – Raw Image Series
BSE Detector
5kV



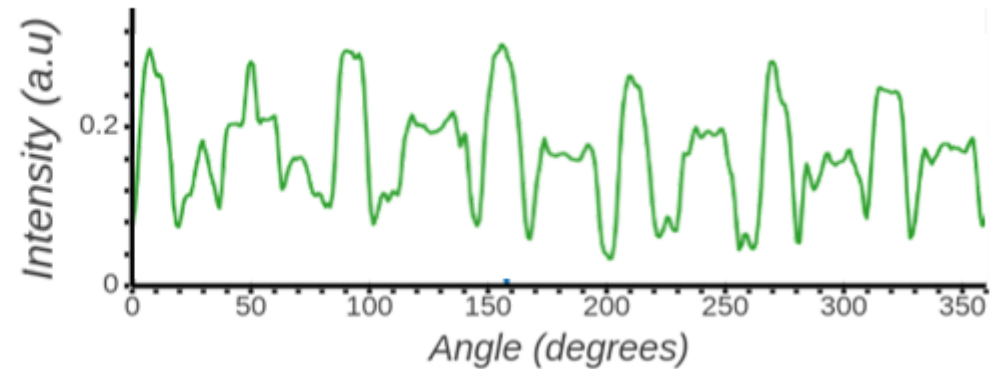
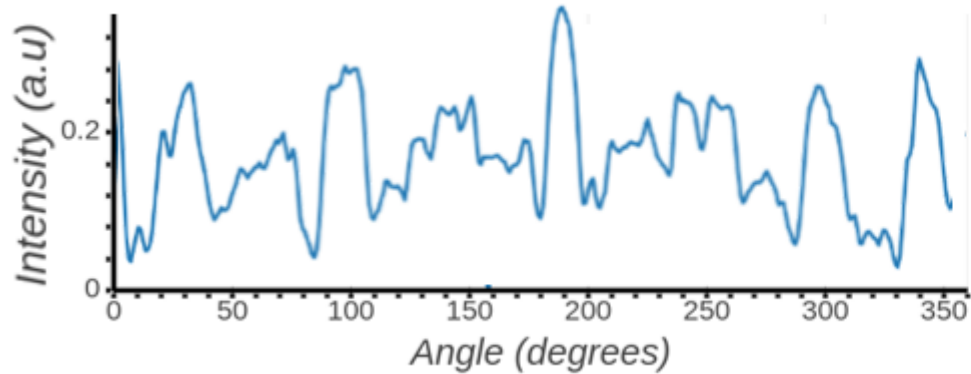
From raw image series to intensity profiles

- Registration
- Crop of the region of interest
- Denoising



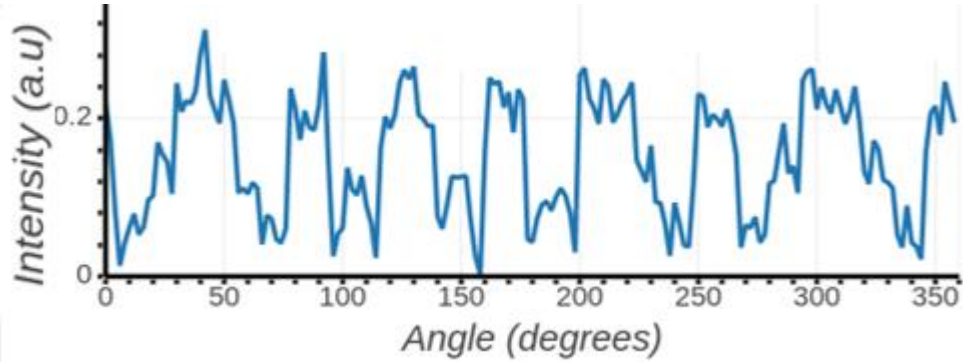
Grain 1

Grain 2

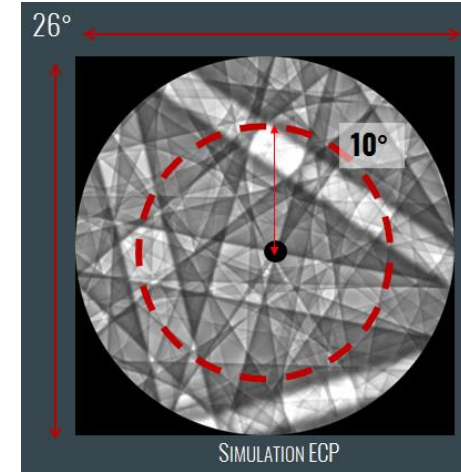


Recover the orientation

Experimental intensity profile

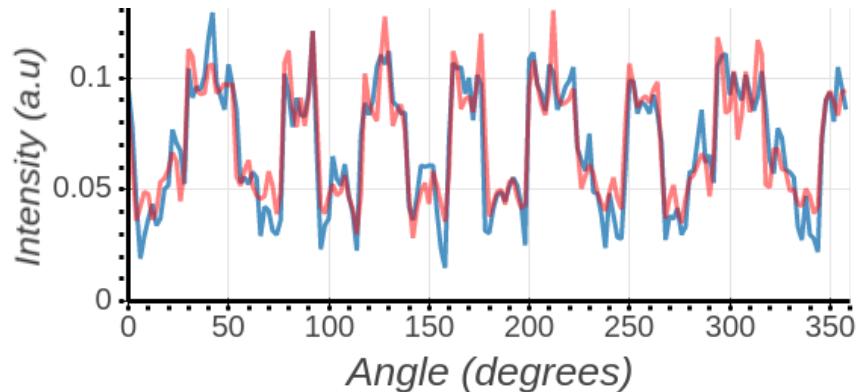


Theoretical profiles database



Singh, S., Ram, F., & De Graef, M. (2017) *EMsoft: Open source software for electron diffraction/image simulations*, *Microscopy and Microanalysis*, 23(S1), 212-213.

Indexation

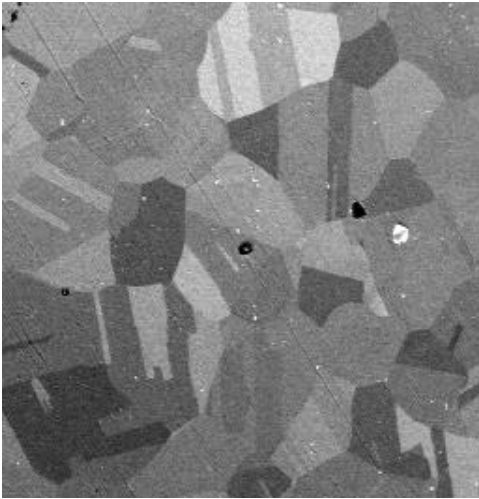


- Theoretical profile
- Experimental profile



Performances

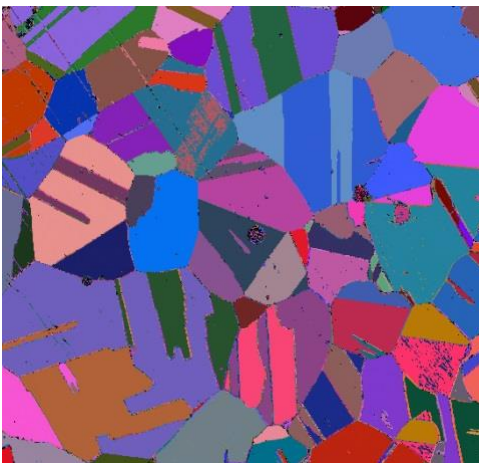
230 μm



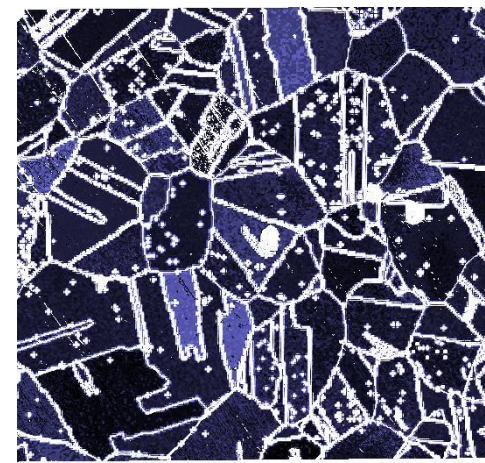
BSE image series



EBSD map



eCHORD map (raw)

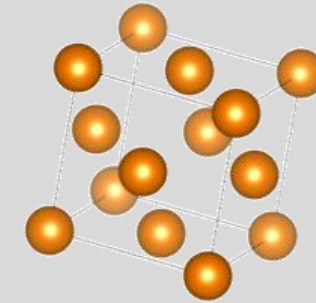


Disorientation map eCHORD / EBSD



Comparison with EBSD

Nickel

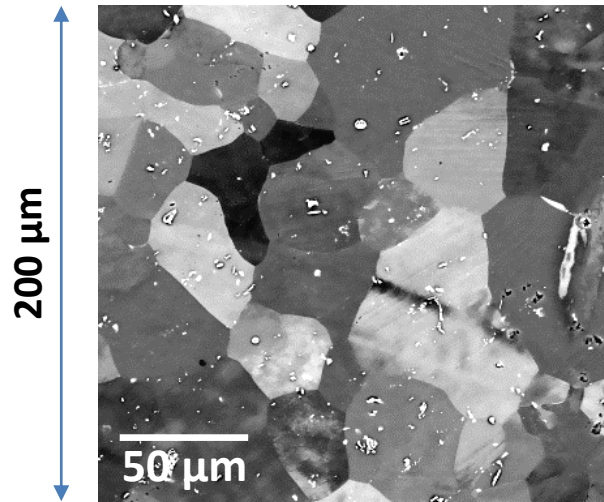


Profile database: 1 million theoretical profiles

Tension 15 kV / WD : 7 mm



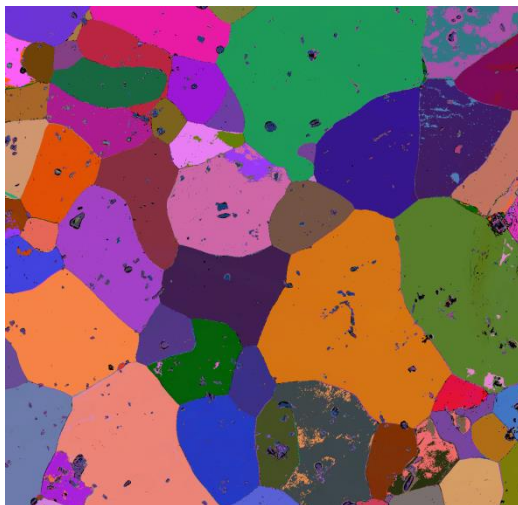
Comparison with EBSD



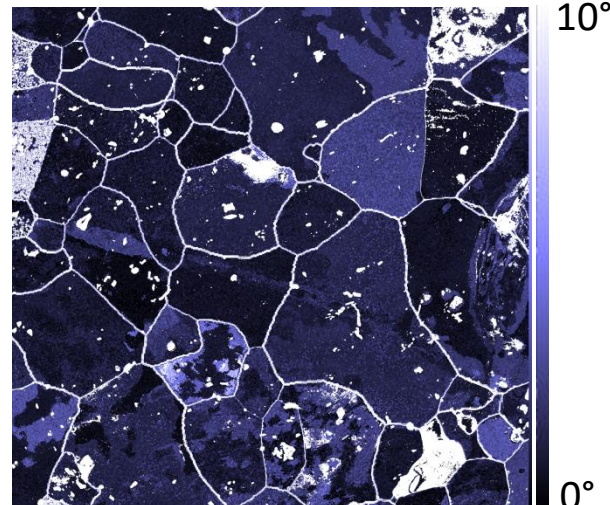
BSE image series



EBSD map (denoised)

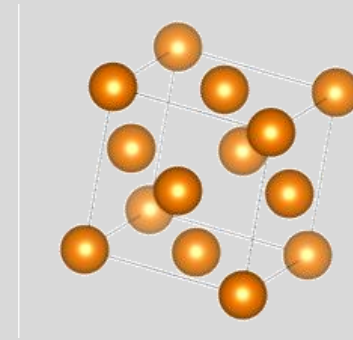


eCHORD map (raw)



Disorientation map eCHORD / EBSD

Aluminum

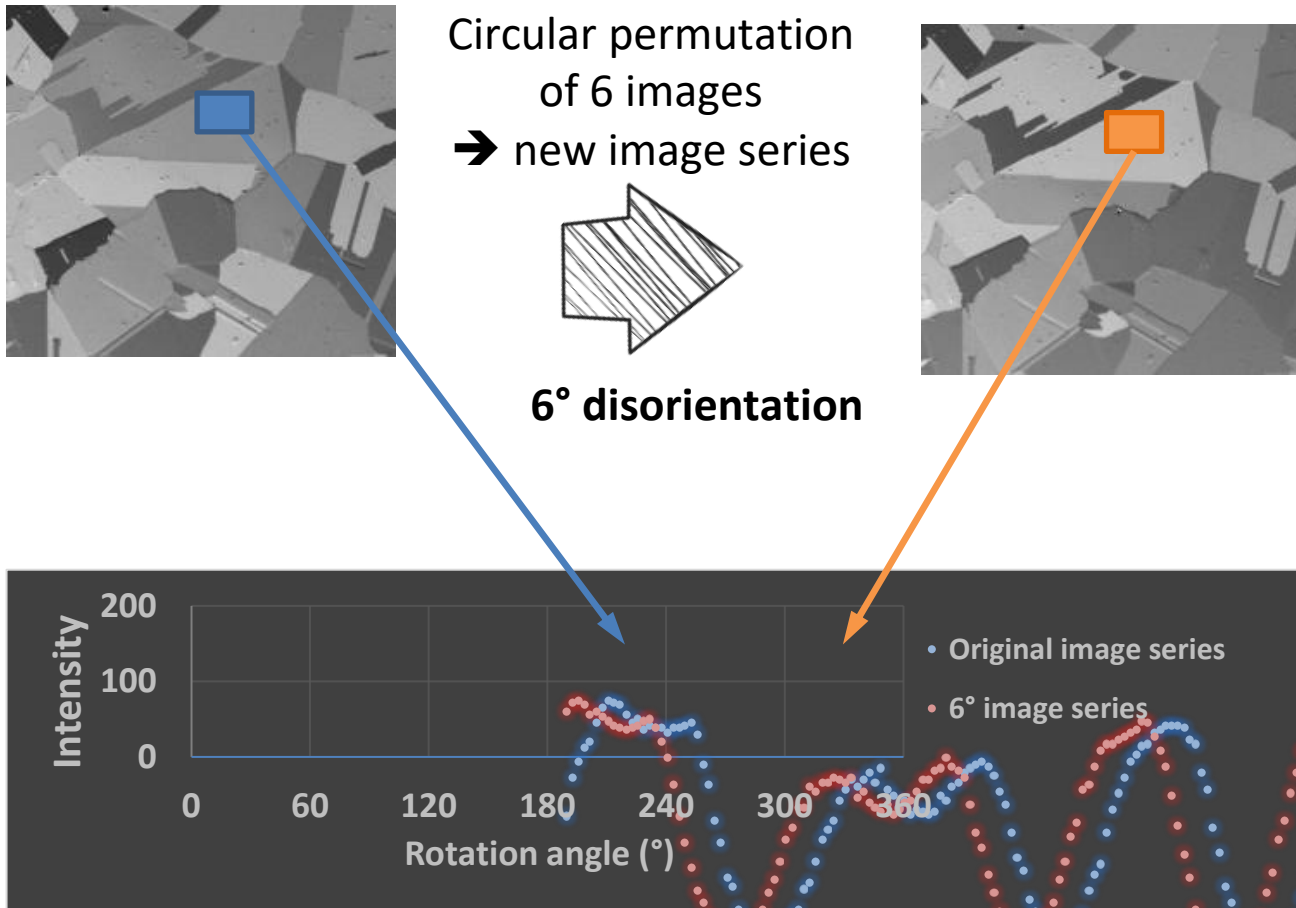


Profile database: 1 million
theoretical profiles

Tension 15 kV / WD : 7 mm



Angular resolution

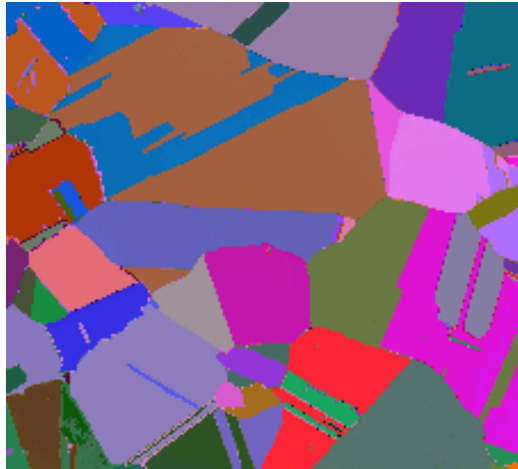
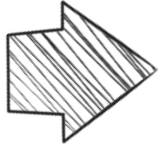


The idea: circular permutation of 6 frames in the image series

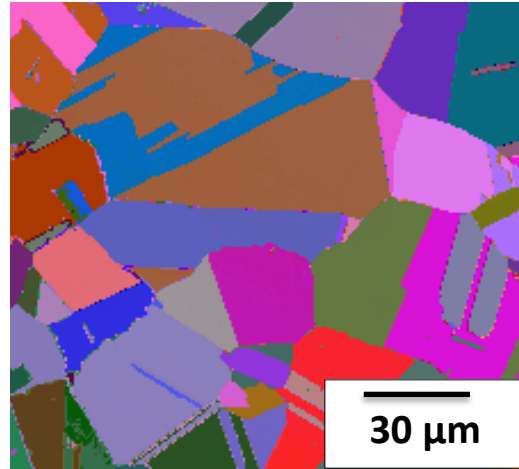
→ **Peak shift of 6° in the intensity profiles //**

Disorientation of 6° between the two maps

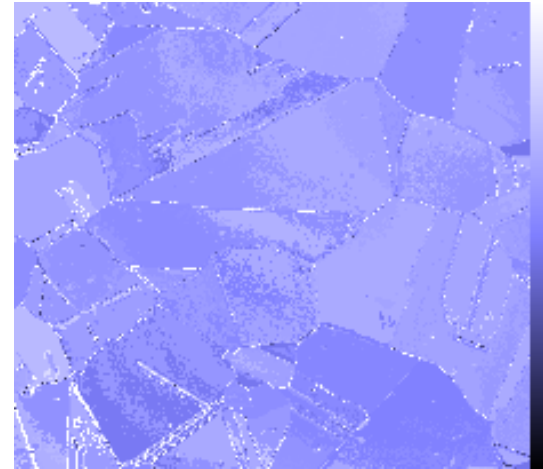
Angular resolution



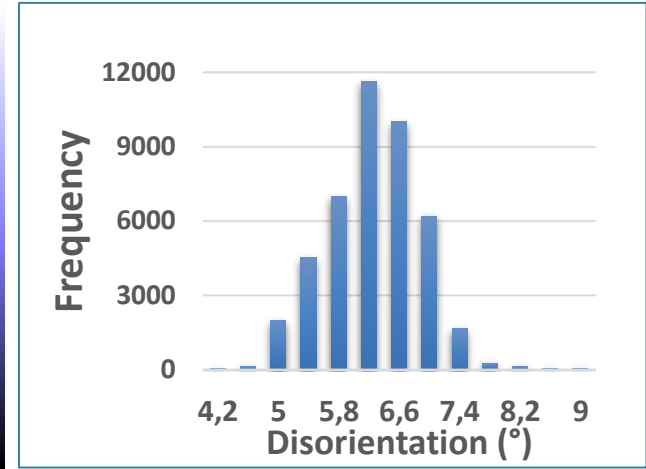
Original



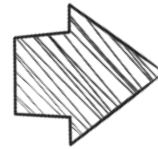
6° apart



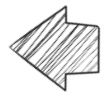
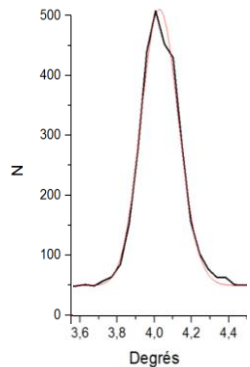
Disorientation map 0°



Disorientation distribution
 $6.5^\circ \pm 0.9^\circ$
with 500k profile database



**ICHORD ANGULAR RESOLUTION
ESTIMATED TO 0.9°**



**SAME EXPERIMENT WITH 4° DISORIENTATION FOR ELECTRONS
ECHORD ANGULAR RESOLUTION ESTIMATED TO 0.1°**



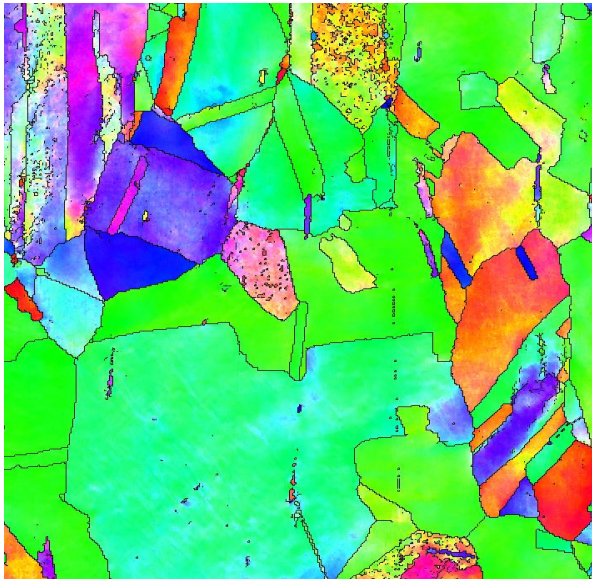
- **CHORD presentation**
- **A few exemples**
- **Perspectives**
- **Live acquisition**



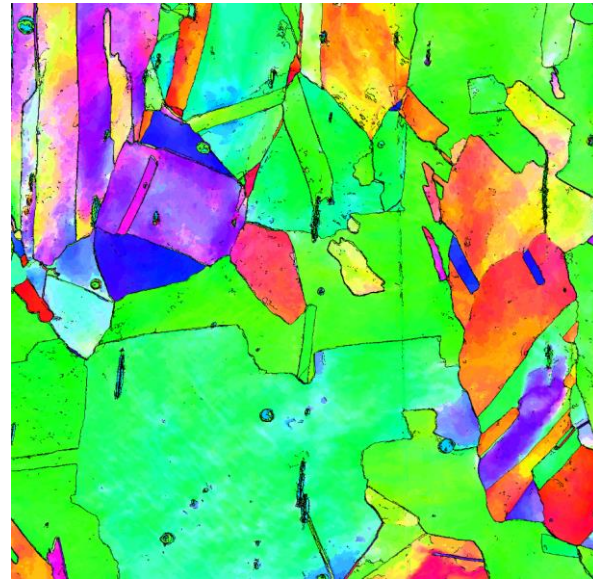
i-CHORD : Copper

Slightly Deformed Copper Sample

145 μm



**EBSD [001] IPF map
(noise corrected)**



iCHORD [001] IPF map

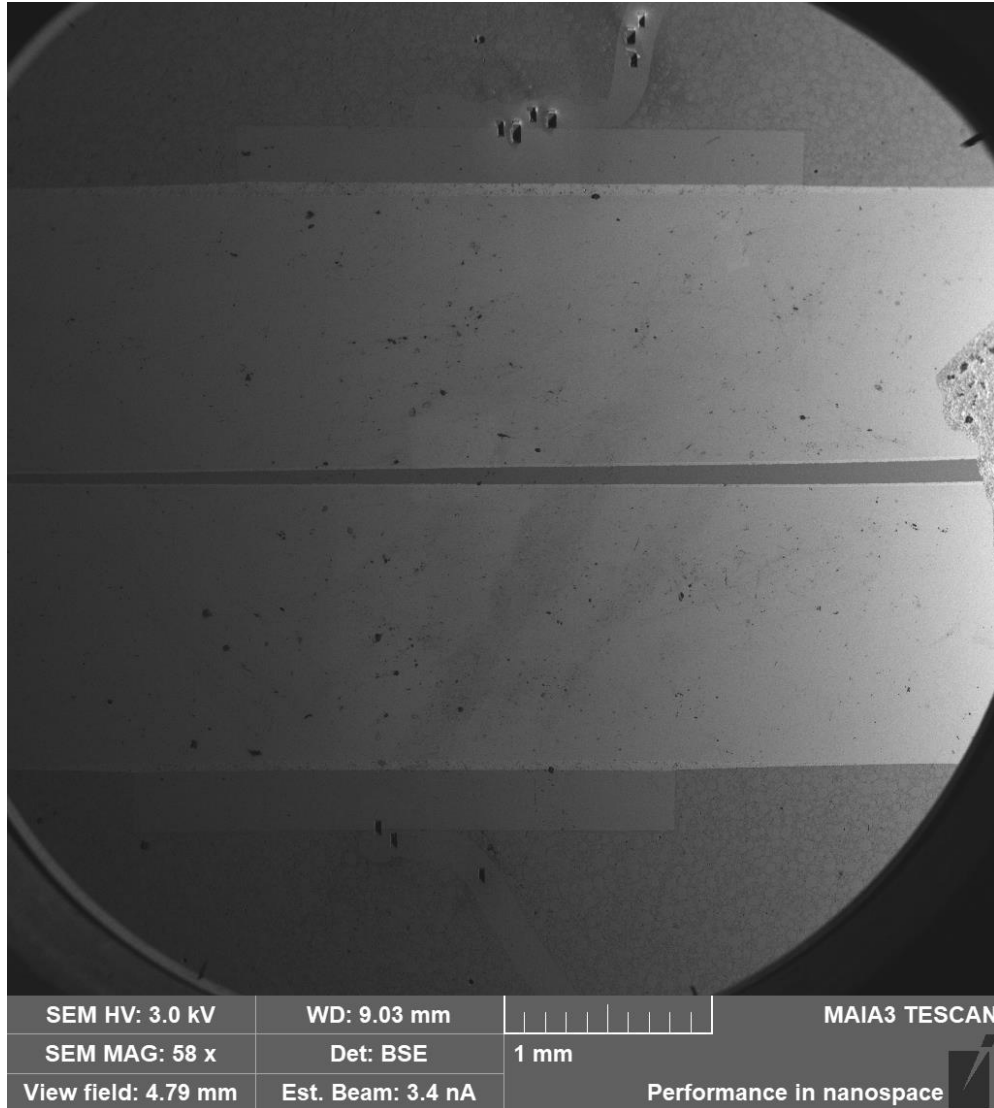
Color gradient represents an orientation gradient.
This orientation gradient is the signature of
deformation gradient.

This example shows that the angular resolution is
sufficient to trace this deformation gradient.

Acquisition condition :
30 keV 700 pA
40 min



e-CHORD : Al ribbon



2 parts on the sample : soft and hard

In each case : 3 regions of interest

- Wire
- Bottom wire
- Interface metal-wire

Goal : study the crystallographic orientation and grain size at different region of interest.

Courtesy of

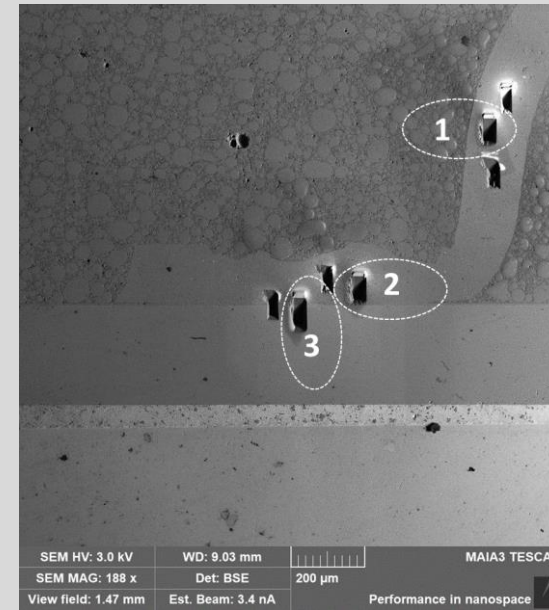




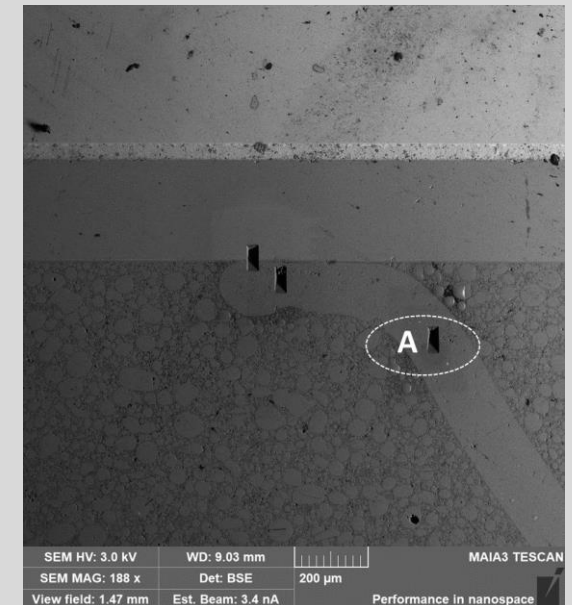
Cross sections realized with plasma FIB (Fera)

- for an excellent surface quality .
- Exactly at the ROI
- Large volume for image without any shadowing.

Experimental conditions : 20 min- rough milling current 30 nA
 Polishing current 1 nA



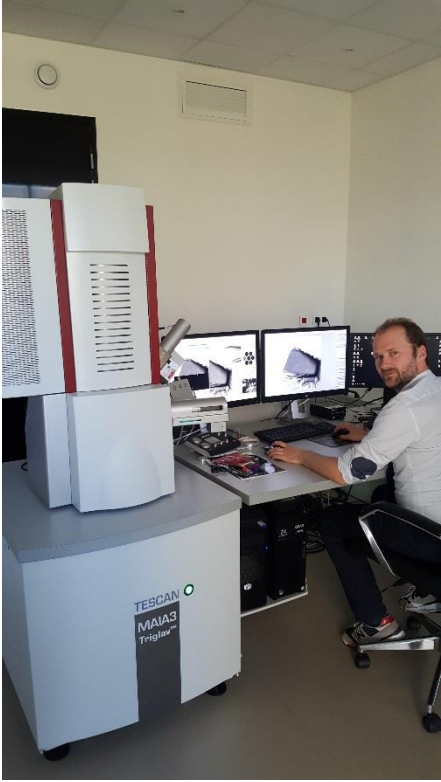
Hard part



Soft part

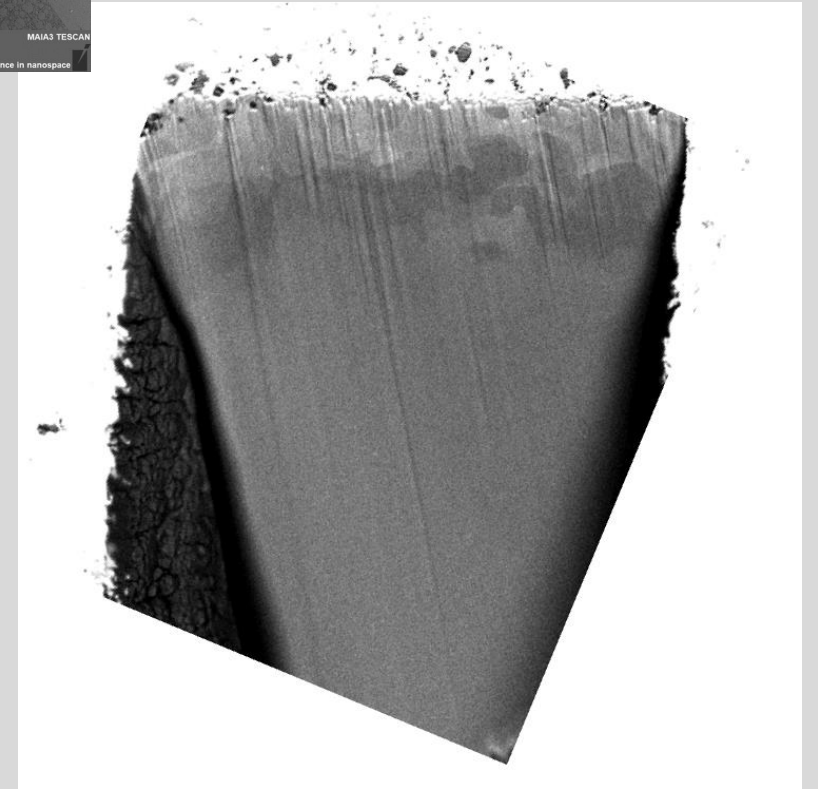
Courtesy of





- Series of images taken with SEM (Maia)
- Condition of acquisition : 3 keV WD = 9mm
- Time for the acquisition : 50 min

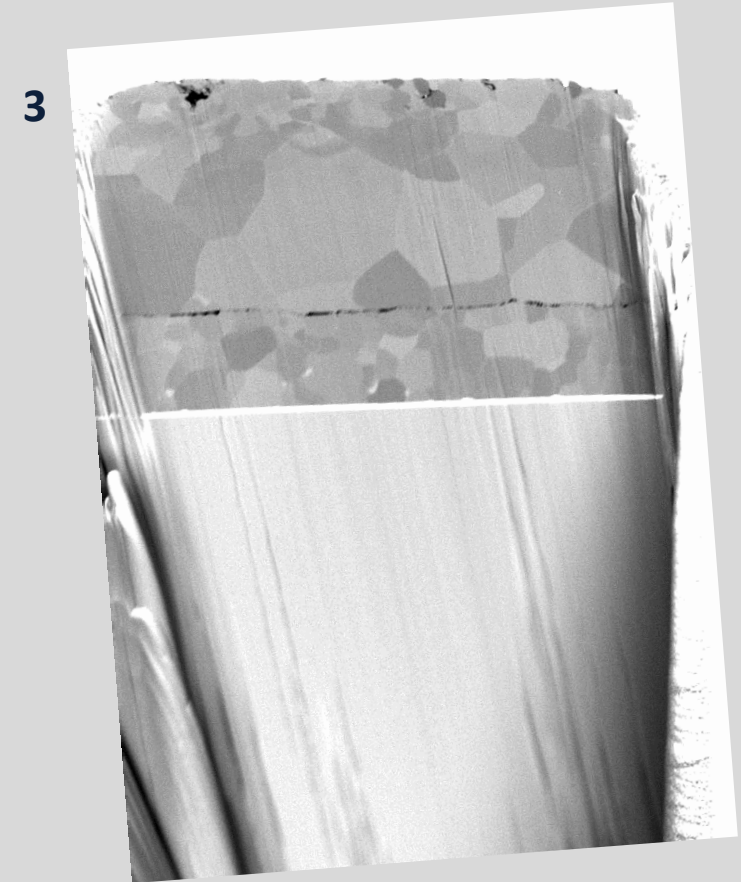
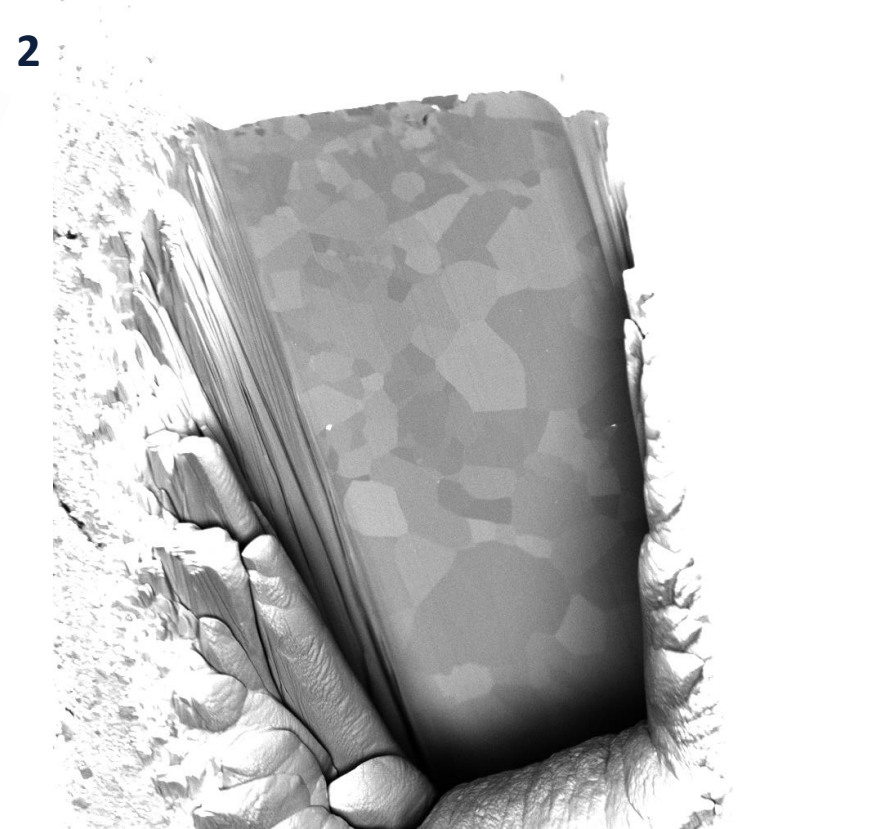
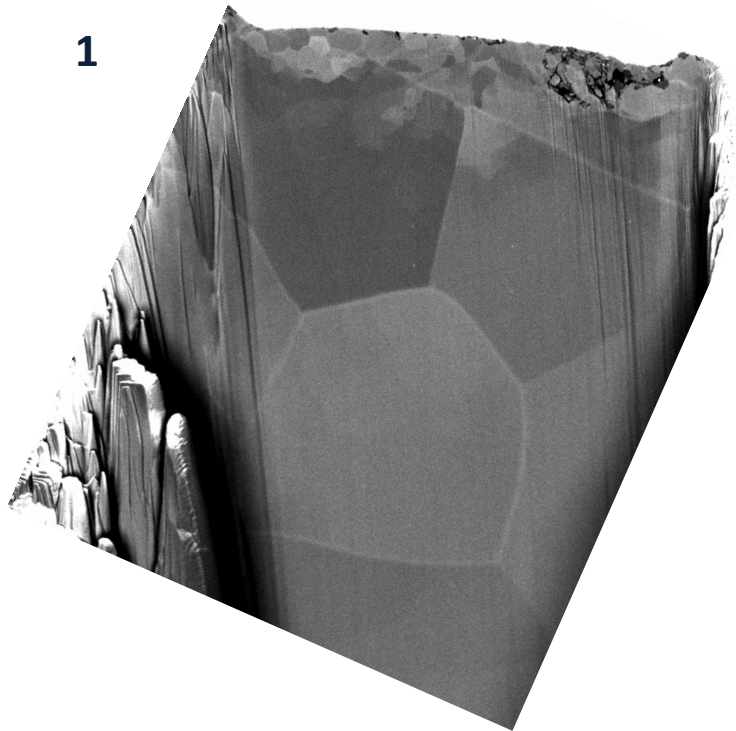
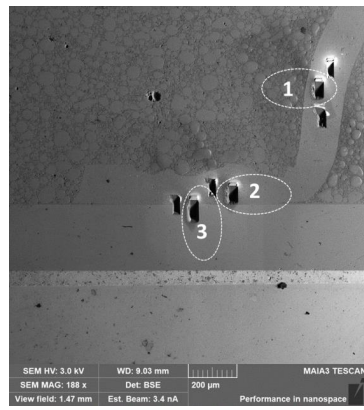
Al ribbon – Soft part



Courtesy of



Al ribbon – Hard part



e-CHORD : Aluminum Sputtering on Silicon wafer



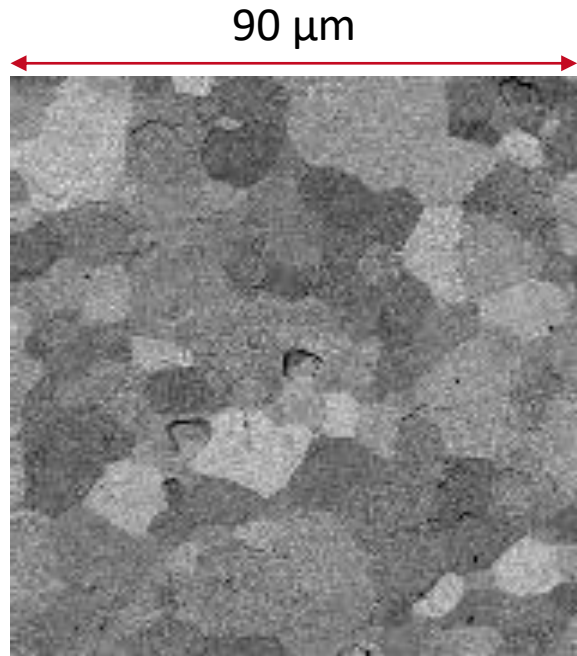
Conditions :

- Tilt 10°
- 3 keV accelerating voltage
- Microscope TESCAN Maia
- 120 images (3° rotation steps)
- No polishing

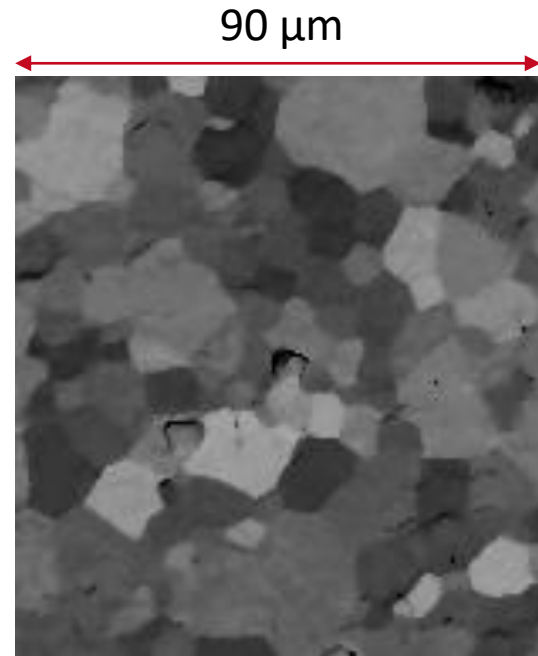


Image serie

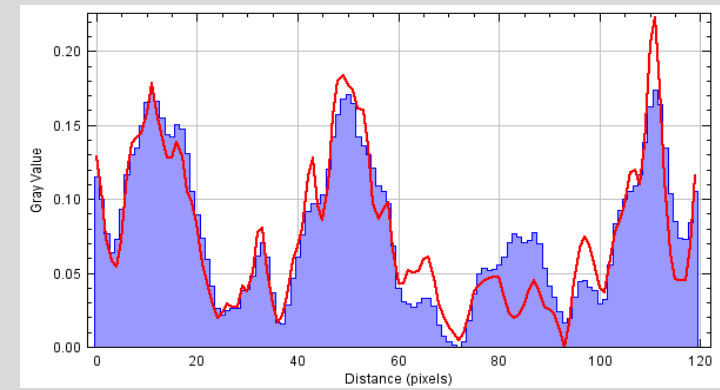
- Profil clustering
- Average profil by cluster
- Profil indexation



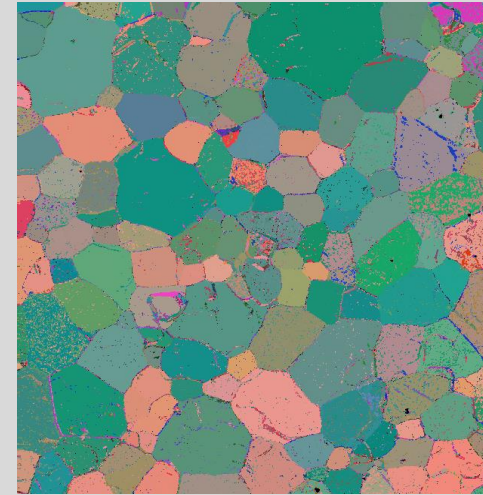
No denoising



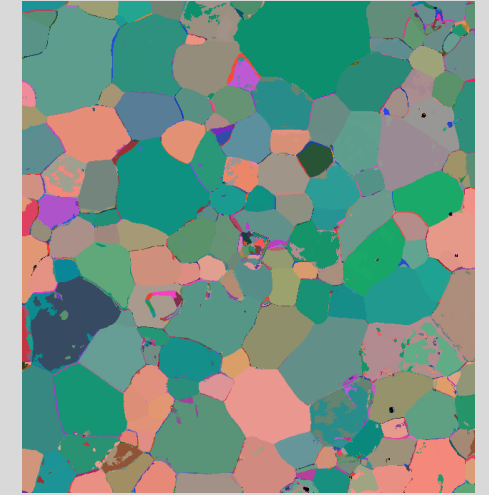
After denoising



Example of theoretical and experimental (3keV // 10°)



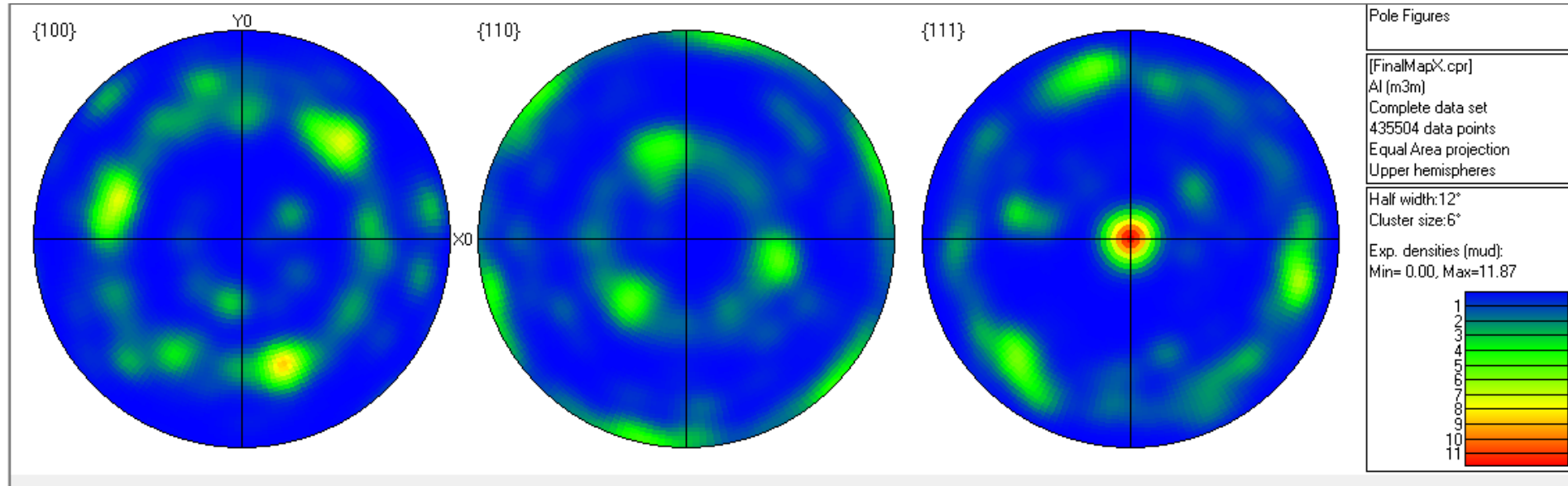
No denoising



After denoising



Pole Figures



[111] perpendicular to the surface
Preferential Crystallographic growth along [111] direction



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- **A few exemples**
- **Perspectives**
- **Live acquisition**

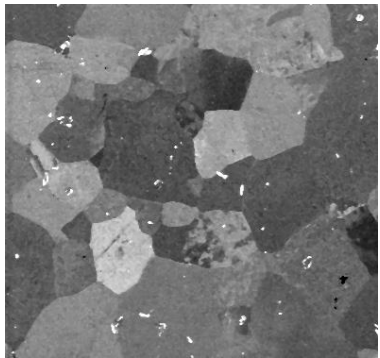


Perspectives and messages to take home

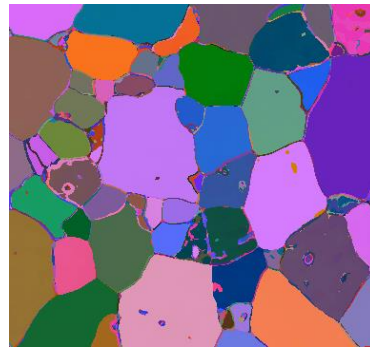
Fast CHORD

Aluminum. Tension 5 kV / WD: 7 mm

360 images → 45 images



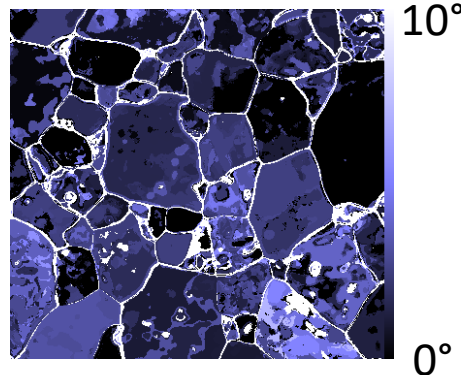
150 μm



eCHORD reference map



eCHORD map with 45 images

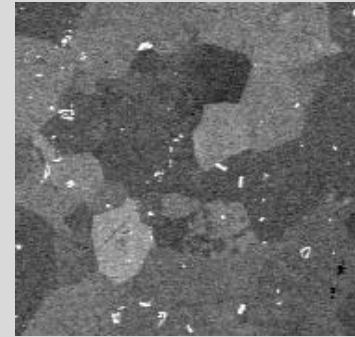


Point-to-point disorientation map

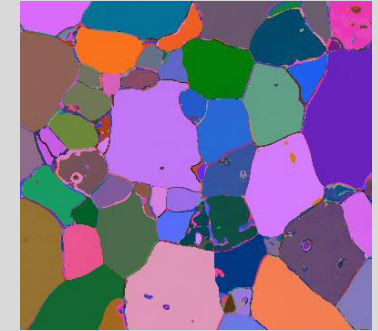
Strategies

1. Reducing number of images in the series
2. Reducing dwell time per pixel

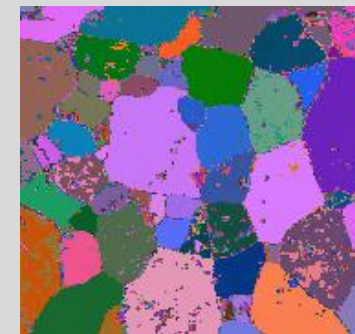
45 images of size 200 x 150 pixels
200 ms per image



150 μm



eCHORD reference map



eCHORD fast map
(45 images)

Total acquisition time

- 12 seconds for 45 images
- 28 seconds of latency between images
(to be optimized)



Simple method to define :

- Crystal orientation
- Grain Size

CHORD limitations

- Still necessary to polish the surface.
- For ion beam : sputtering and amorphization
- New technics : still need to be qualified

Interests compare to EBSD

Simplicity

- No specific detector requested, only a backscattered electron detector is required
- Possible on any microscope

Many possibilities

- Cross section preparation is possible using FIB
- Analyse anywhere on the surface
- Working at low tension (down to 3 kV)
 - Smaller interaction volume
 - Less depth averaging
 - Less charging

Small tilt angle

- Less image distortion
- Same probe size in X and Y
- More favorable geometry for coupling with EDS
- Scanning larger area without touching the pole piece



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Thank you for your attention

