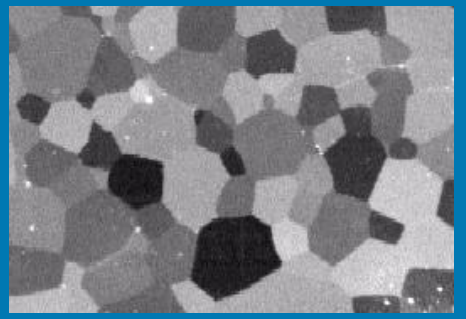
TESCAN
PERFORMANCE IN NANOSPACE



- CHORD –
When Ion or Electron Channeling meets Crystal Orientation Mapping.

C. Langlois[1], C. Lafond[1], T. Douillard[1], S. Cazottes[1], S. Dubail[2], Jérémie Silvent[3], Anne Delobbe[3].

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INSA INSTITUT NATIONAL DES SCIENCES APPLIQUÉES LYON

ORSAY PHYSICS
TESCAN ORSAY HOLDING

04.02.2019

Locations and the Team

Tescan Brno Facility

Mateis

Cyril Langlois/ Thierry Douillard

Co-Inventors of the CHORD patent

Orsay Physics

Jérémie Silvent

Application engineer

Clément Lafond

PhD

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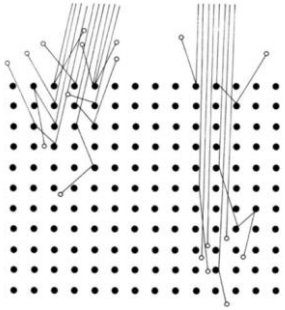
CHORD definition
Short reminder on EBSD technics

CHORD stands for : CHanneling ORientation Determination

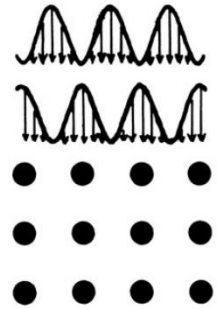
CHANNELING CONTRAST

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

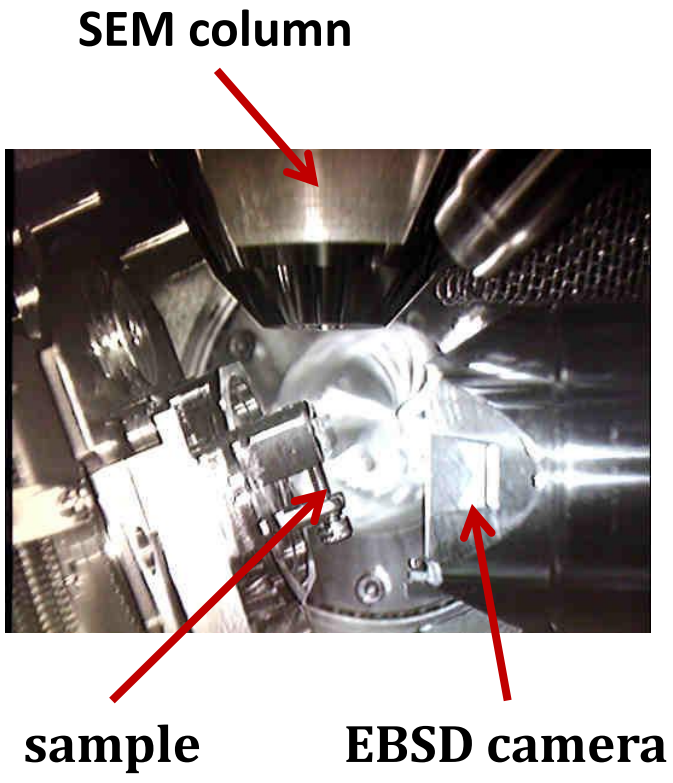
- if the ion beam is parallel to low index planes, the secondary electron are generated far under the surface. A low intensity is then detected.

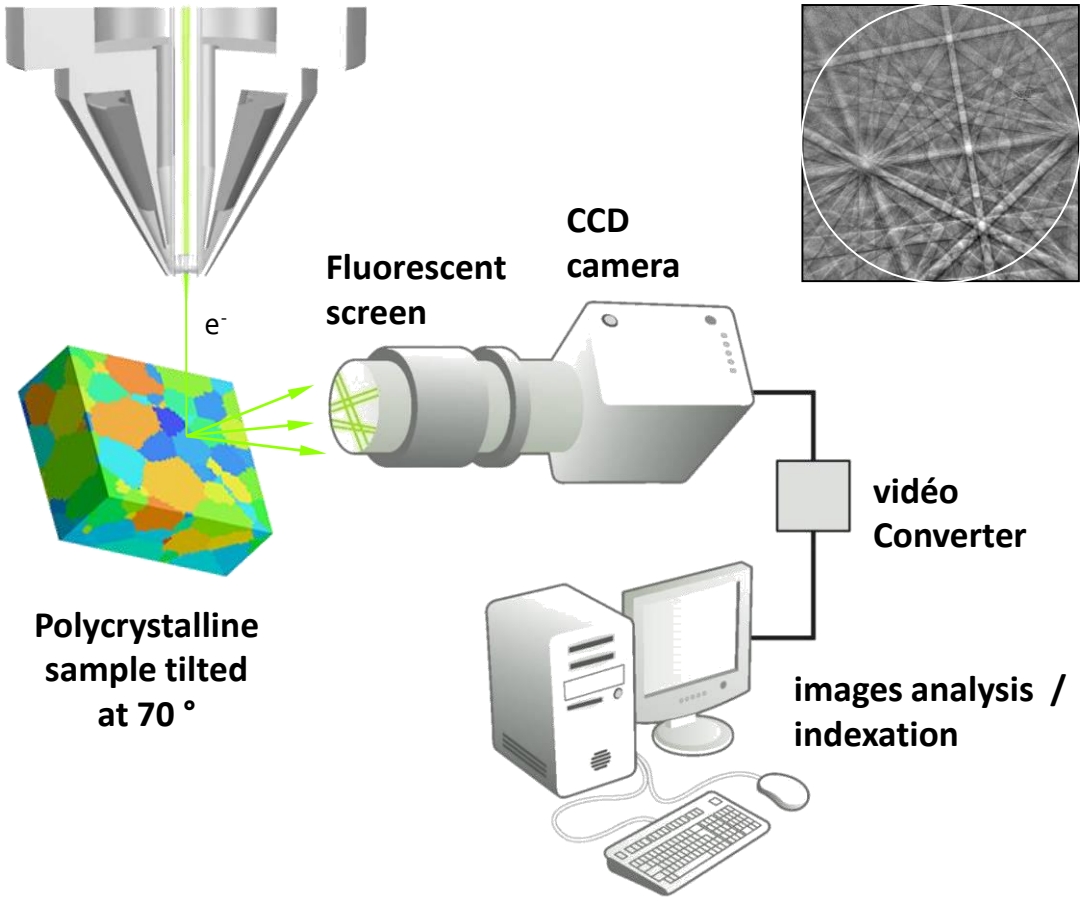


- with an electron beam, the intensity received by the detector is monitored by back scattered diffraction.

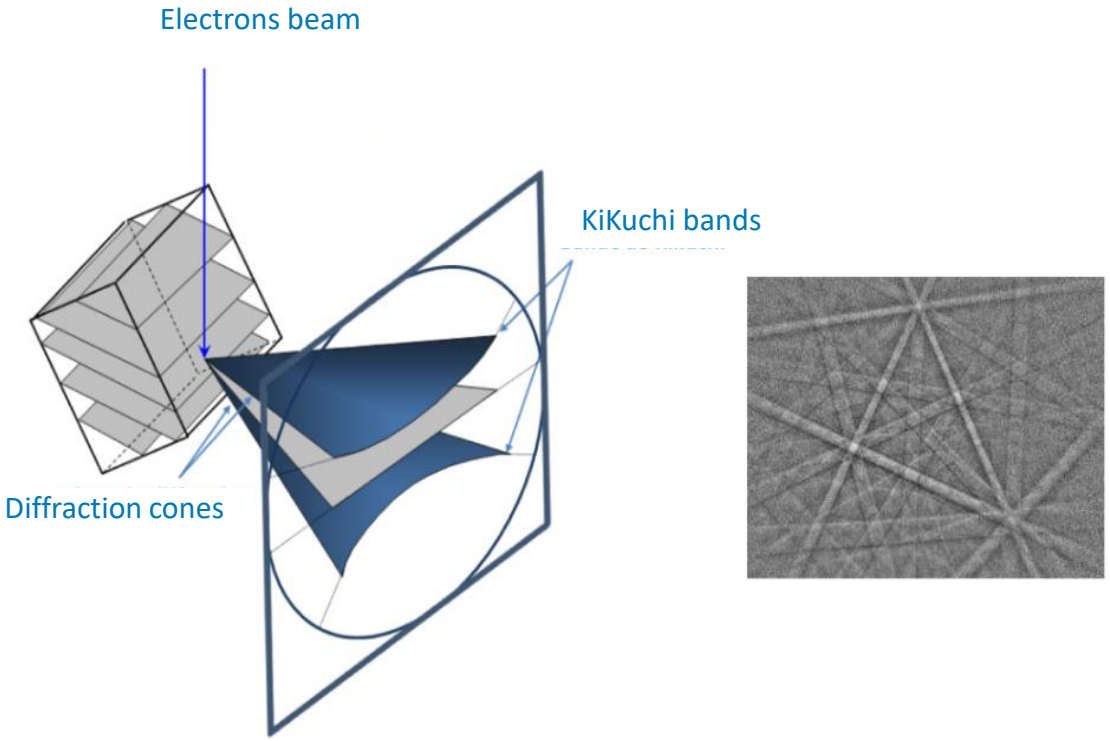


EBSD (Electron backscatter diffraction)

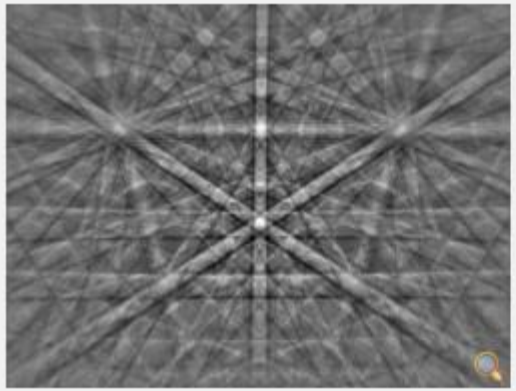




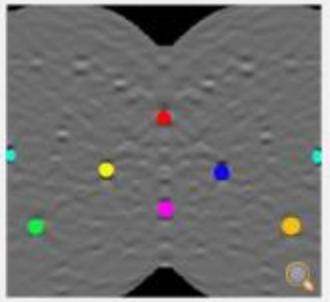
EBSD (Electron backscatter diffraction)



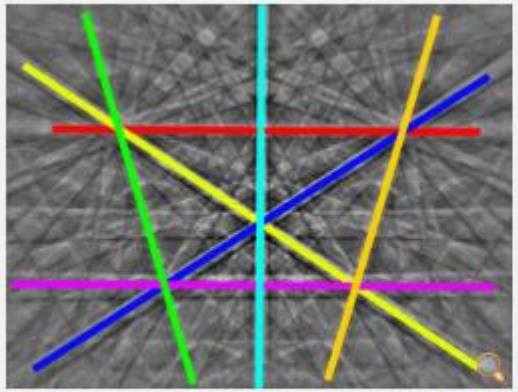
Pattern Formation



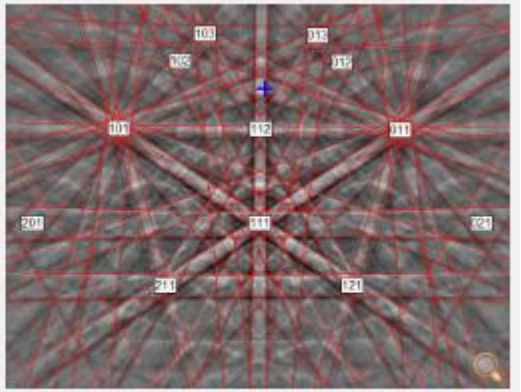
(a) Diffraction pattern collected from silicon at 20kV accelerating voltage;



(b) The peaks in the Hough transform identified and coloured;



(c) The bands in the original diffraction pattern corresponding to the peaks found in the Hough transform and coloured similarly;



(d) The indexed diffraction pattern with the blue cross indicating the position of the pattern centre.

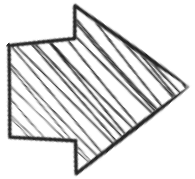
Interpreting the Diffraction Pattern

Web site by oxford instruments : www.ebsd.com

Crystal Orientation Mapping

Polycrystalline materials

strong relationship between orientation of grains and material properties



Microstructural information is very important

- grain size distribution
- morphologie of grains
- microtexture
- strain
- grain boundary

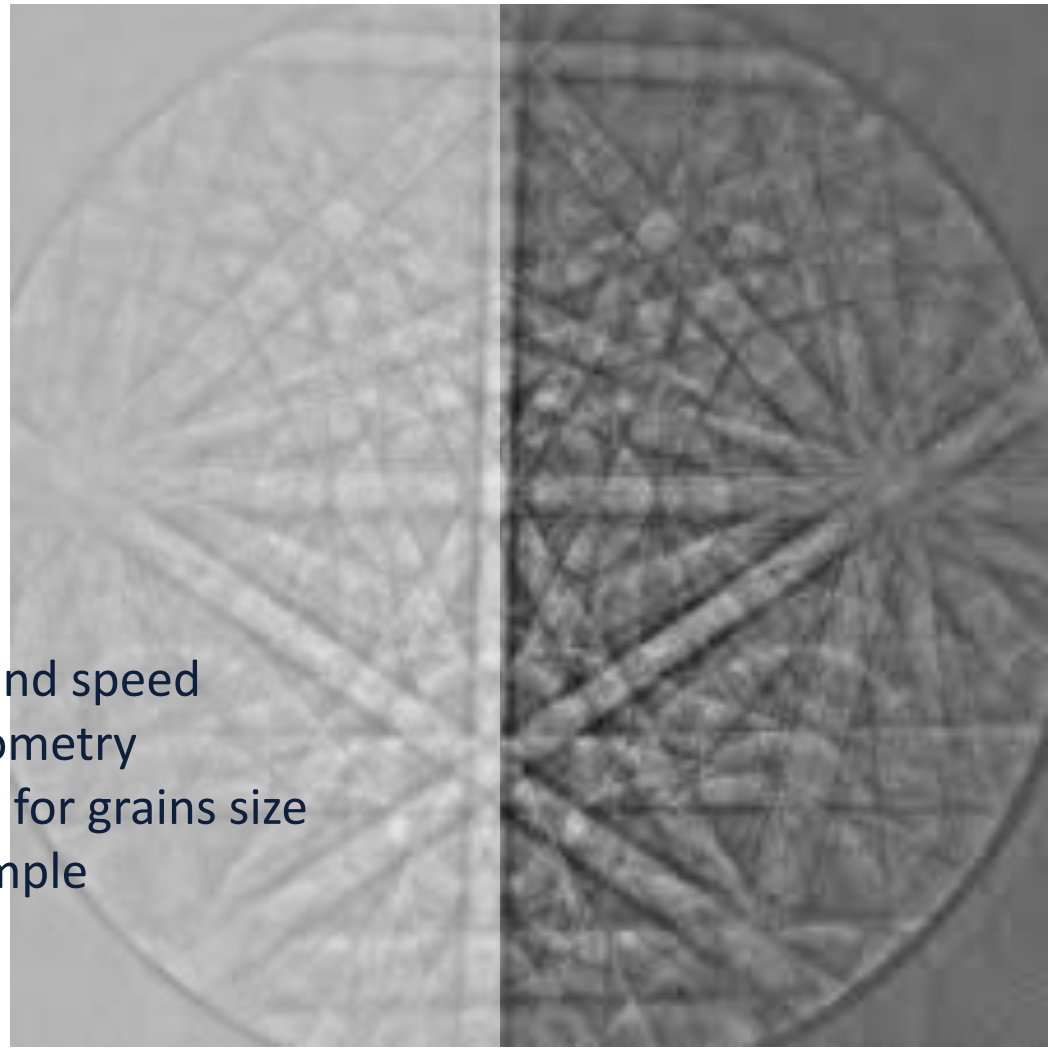
EBSD, advantages and drawbacks

Advantages

- reliability
- automated acquisition
- automated indexation
- widely used

Drawbacks

- supplementary camera
- choice between precision and speed
- constrained acquisition geometry
- Full indexation is necessary for grains size
- Difficult to handle large sample
- Complex 3D acquisition



Other ways to map the orientations ?

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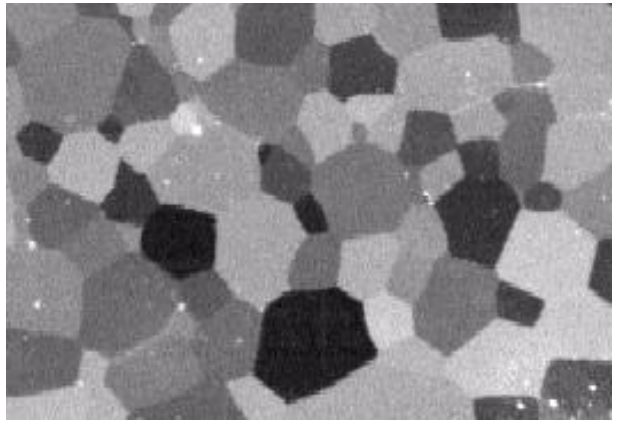
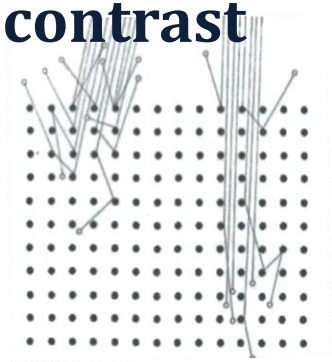
i-CHORD Principle



TiN – image Ion Ga^+ 30kV 3nA

Channeling contrast



… especially in a Focused Ion Beam Machine (FIB)

Comparison of Channeling Contrast between Ion and Electron Images

L.A. Giannuzzi[a1 c1] and J.R. Michael[a2]

Microscopy and Microanalysis

Microscopy and Microanalysis / Volume 19 / Issue 02 / April 2013, pp 344-349

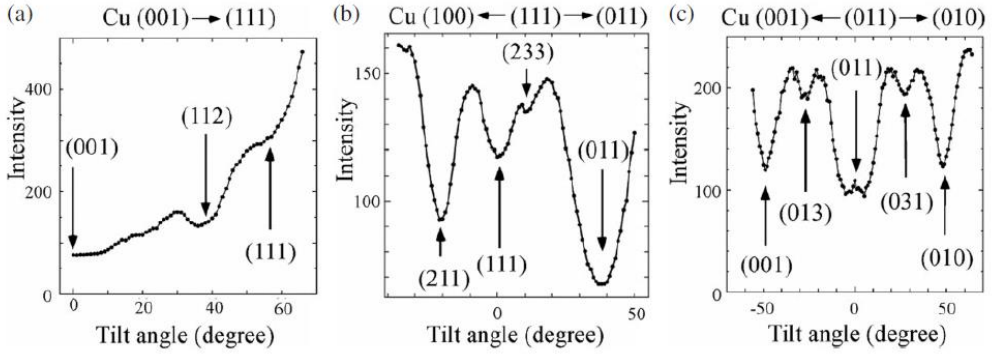


Fig. 2 Variation of the secondary electron intensity with respect to the tilt angle for Cu: (a) tilting direction from (001) to (111), (b) tilting direction from (111) to (011) and (100) and (c) tilting direction from (011) to (001) and (010).

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)

Intensity variation as a function of sample tilt

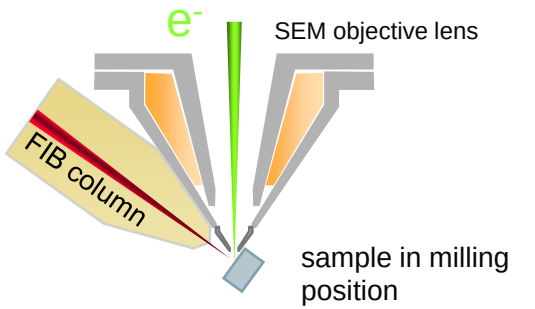
Vasilisa Veligura, Gregor Hlawacek*, Raoul van Gastel, Harold J. W. Zandvliet and Bene Poelsema

Beilstein J. Nanotechnol. **2012,** *3,* 501–506.

Open question

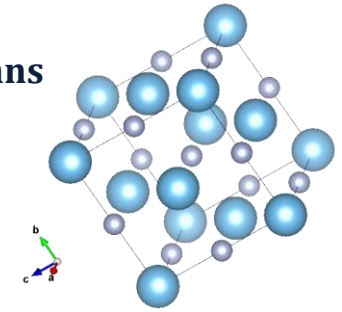
Is there a way to recover the crystallographic orientation from such intensity profiles ?

MICROSCOPE AND SAMPLE

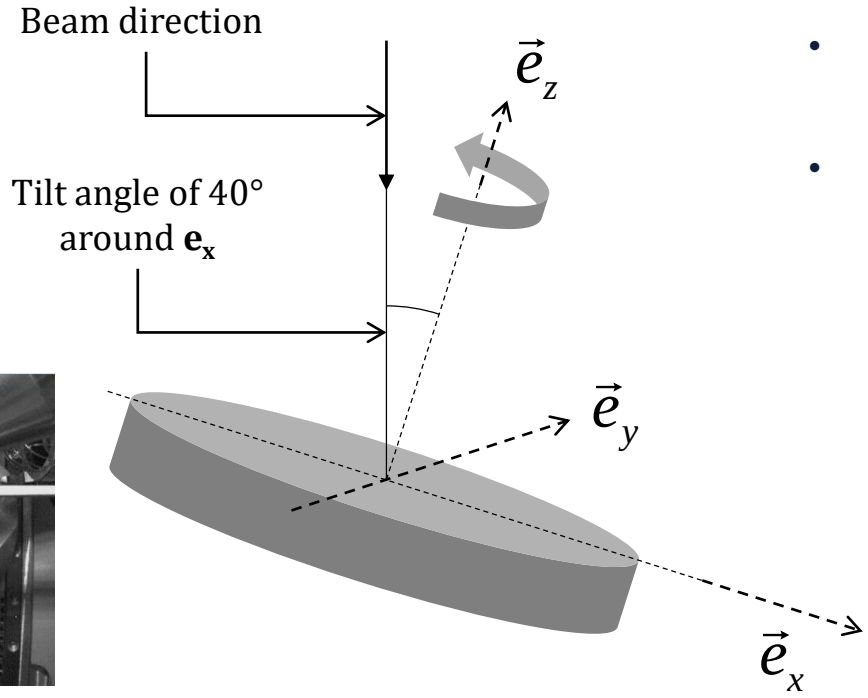


Scanning microscope with both ion and electron columns

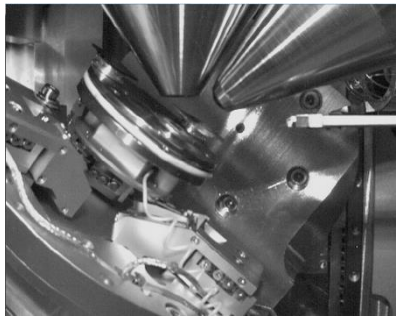
Sample : titanium nitride (TiN) – fcc structure

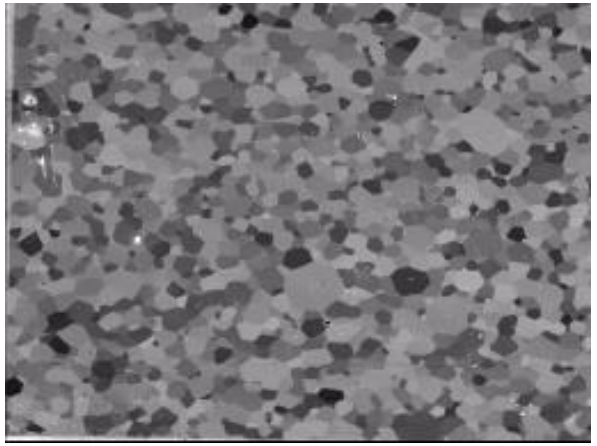


How to vary the orientation ?



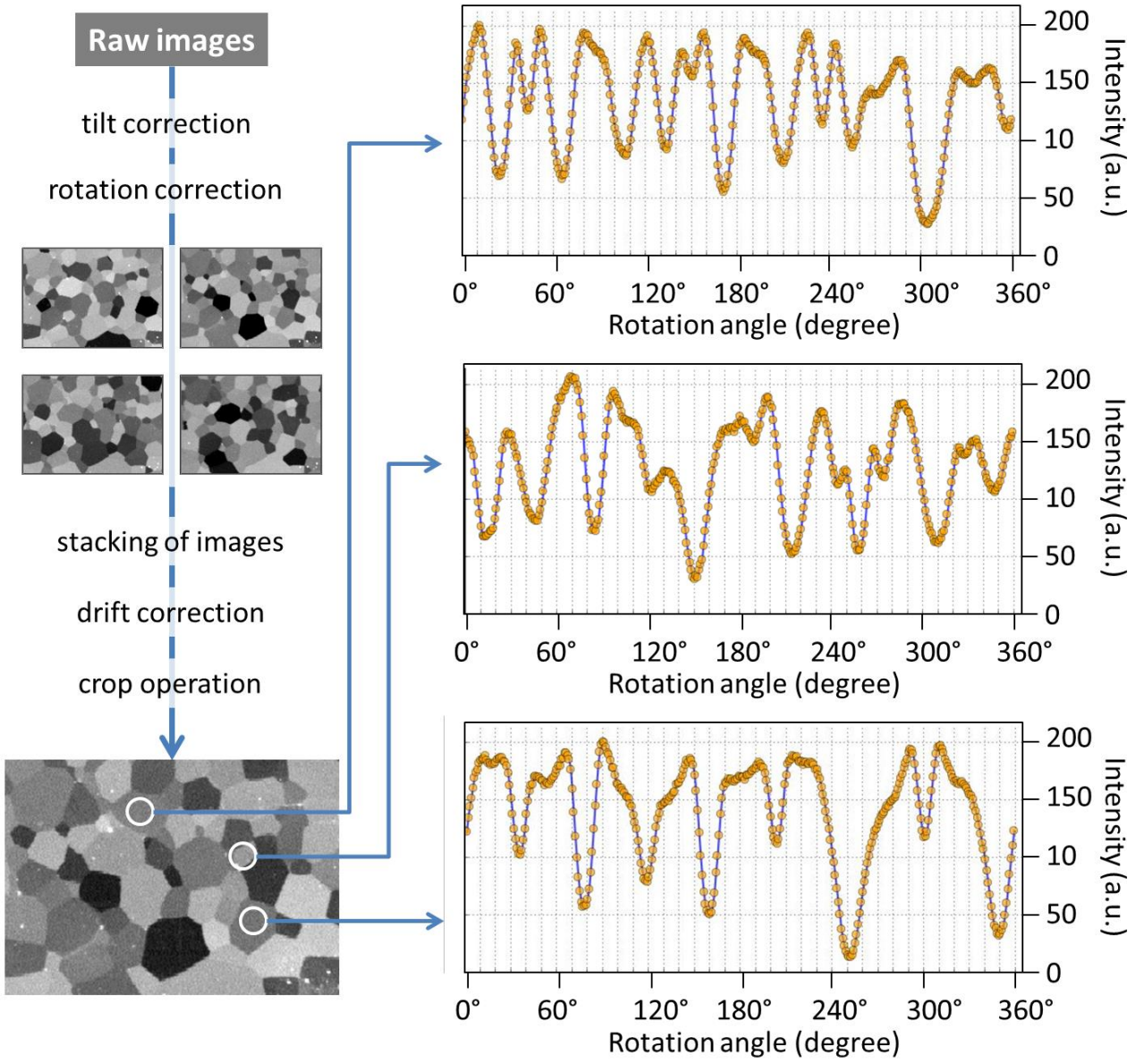
- Starting point : ion beam perpendicular to the sample surface
- then 40° sample tilt ➔ 40° between the beam and the sample normal direction
- then rotation of the sample around the tilted normal
  ➔ one image acquisition every rotation step (automated)



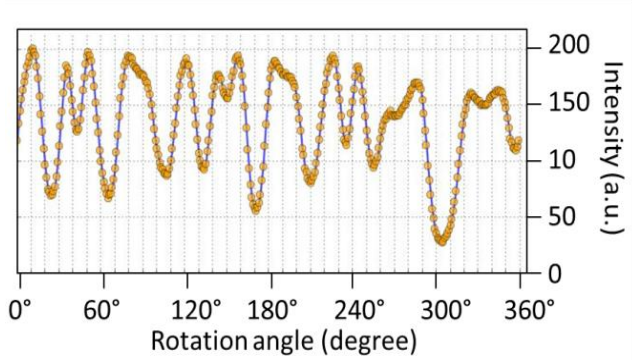


RAW IMAGE SERIES

FROM RAW IMAGES TO INTENSITY PROFILES



FROM INTENSITY PROFILES TO EULER ANGLES

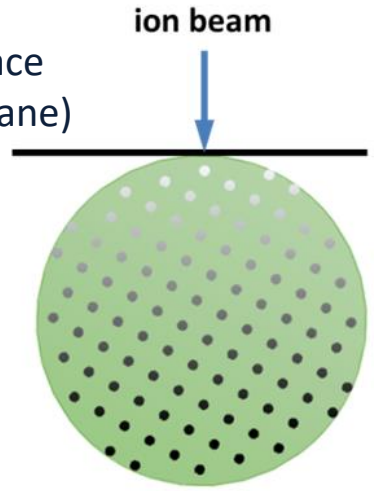


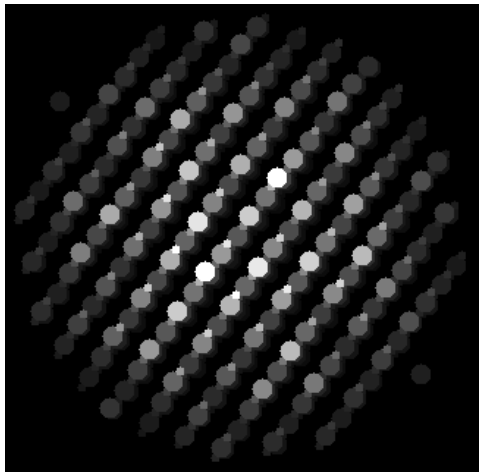
The idea :

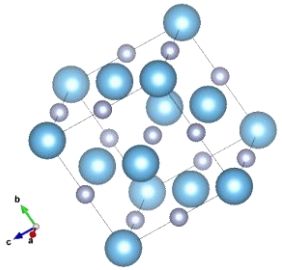
- constituting a profile database
- exploring the database to find the closest profile

Generation of theoretical profiles

THEORETICAL PROFILE CONSTRUCTION





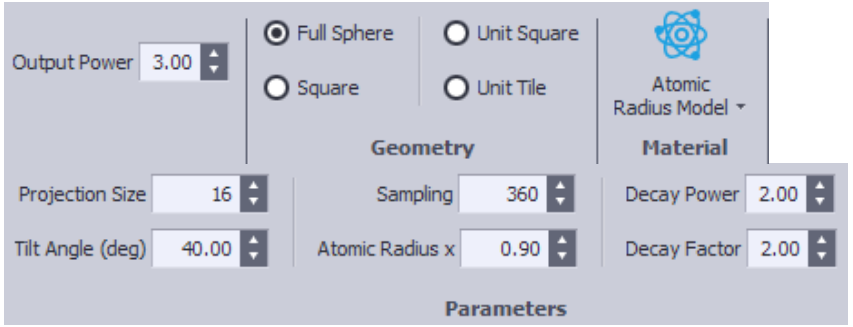


TiN

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

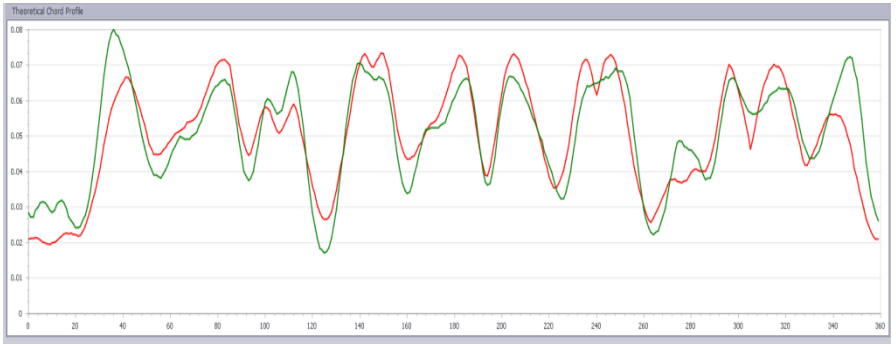
Intensity proportional to the sum of pixels grey levels in the projection

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



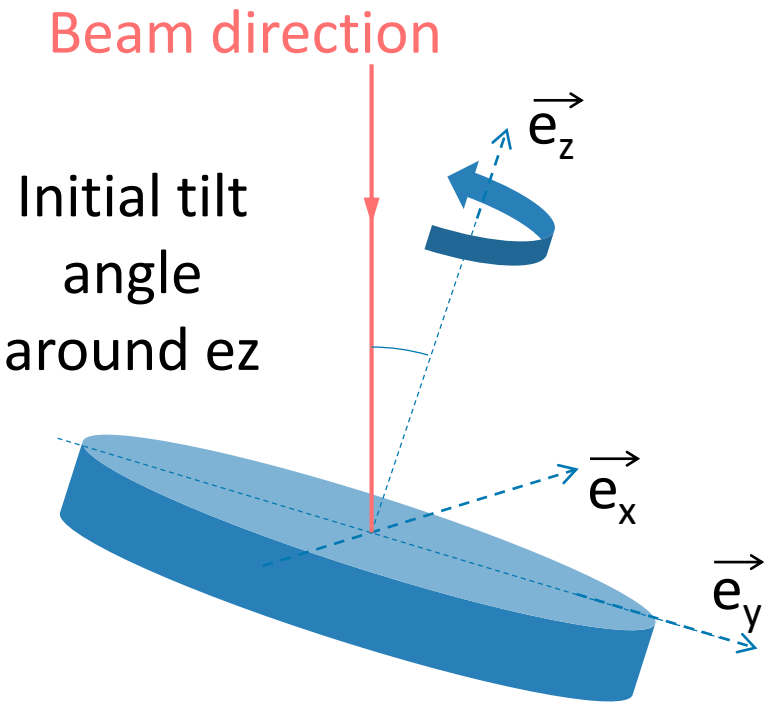
Parameters representative from ion – matter interactions

- **Normalised profiles**



e-CHORD Principle

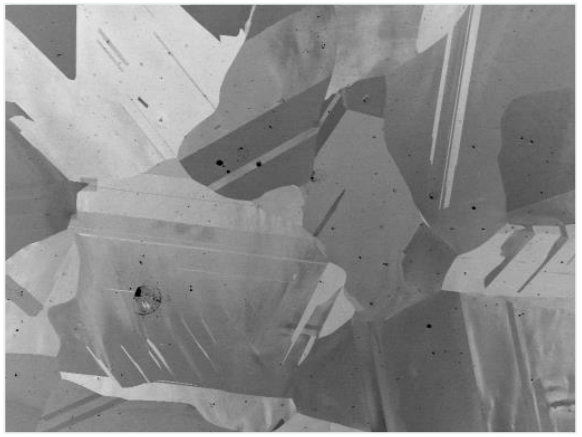
Acquisition



- Starting point : 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

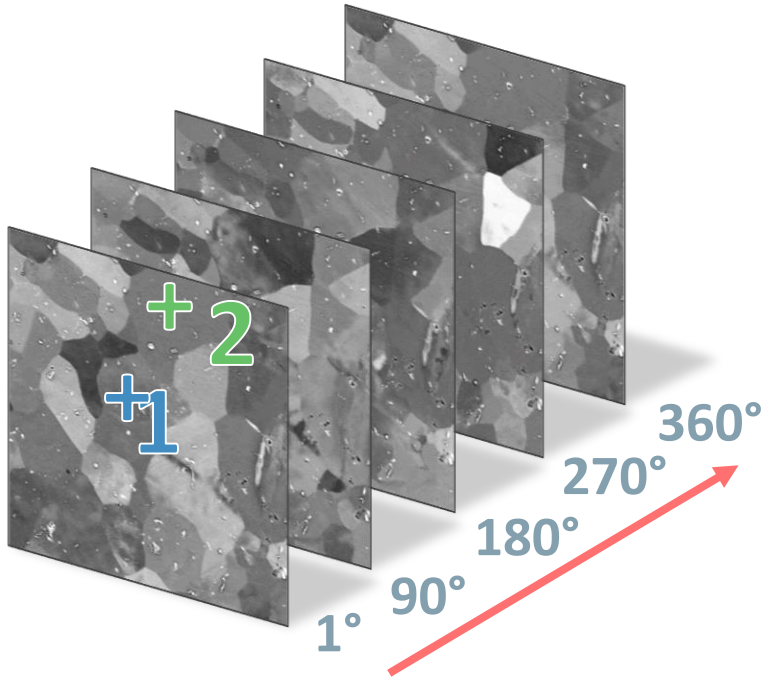
Exemple

Copper – Raw Image Series
BSE Detector
5kV



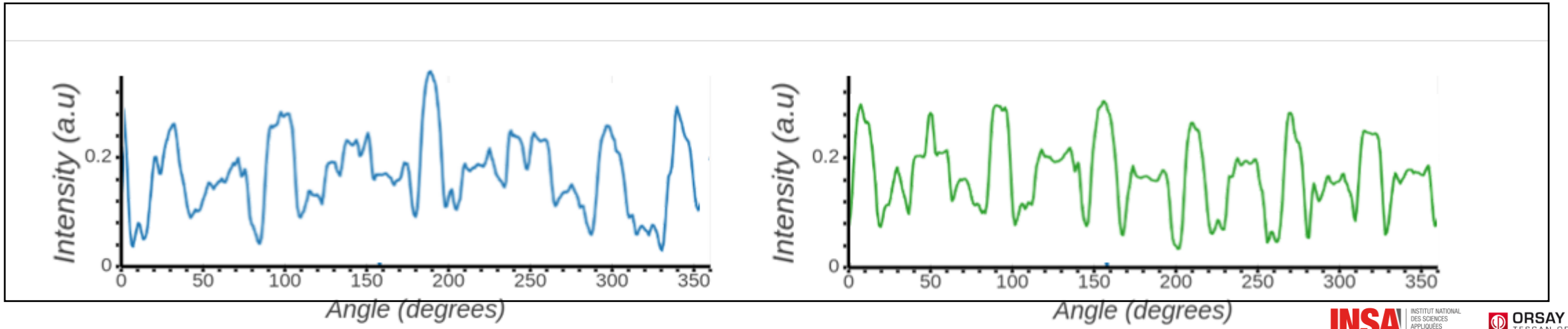
From raw image series to intensity profiles

- Registration
- Crop of the region of interest
- Denoising



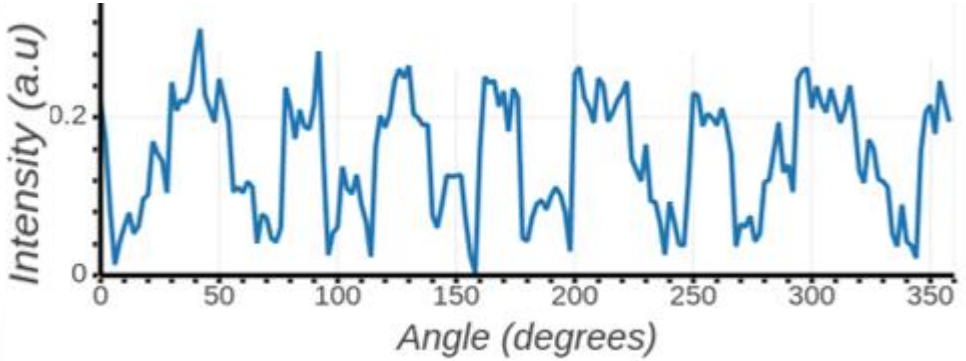
Grain 1

Grain 2

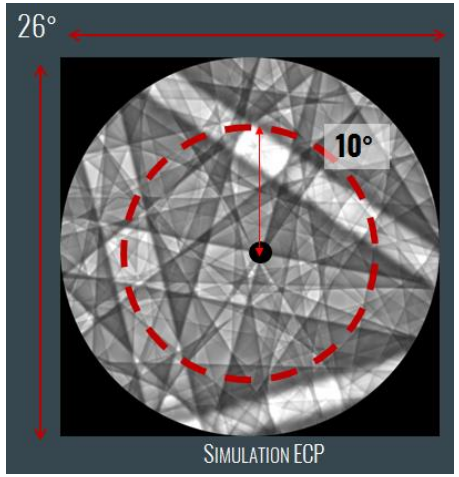


Recover the orientation

Experimental intensity profile

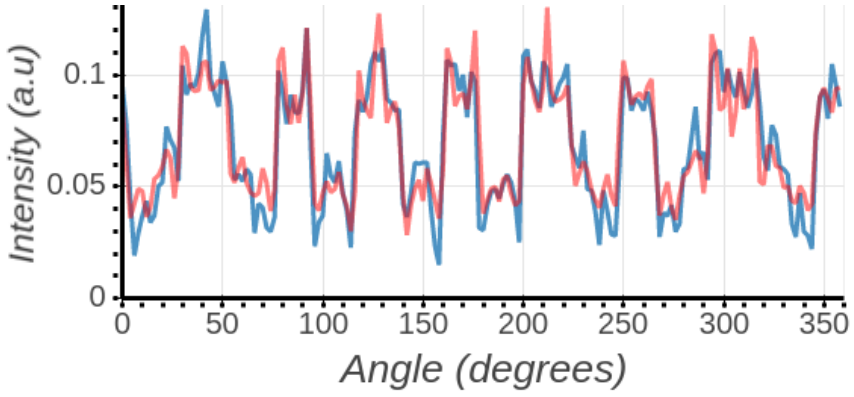


Theoretical profiles database

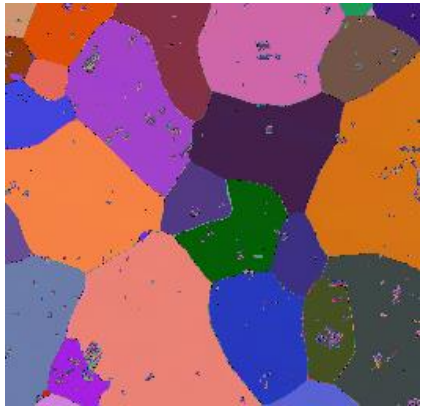


Singh, S., Ram, F., & De Graef, M. (2017) EMsoft: Open source software for electron diffraction/image simulations, *Microscopy and Microanalysis*, 23(S1), 212-213.

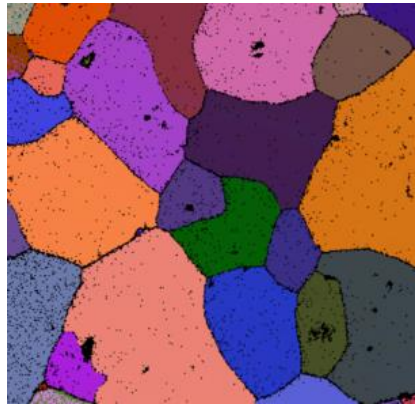
Indexation



Sample: Aluminum



eCHORD map



EBSD map

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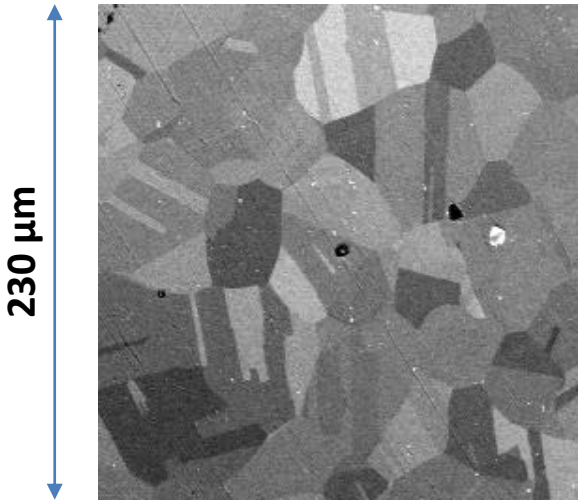
Performances

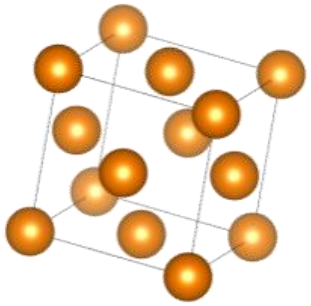
COMPARISON WITH EBSD

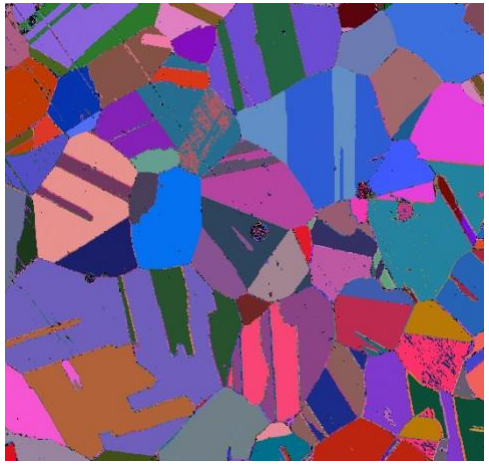


BSE image series

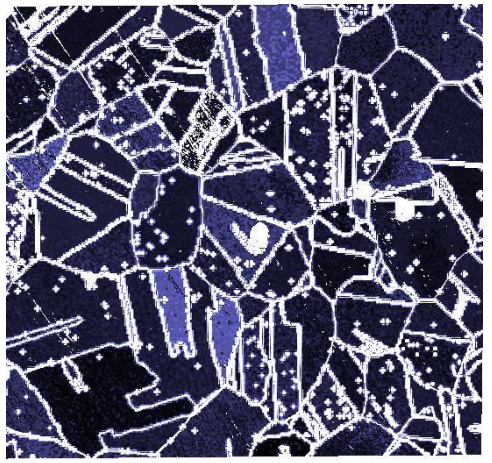
EBSD map

Nickel





eCHORD map (raw)

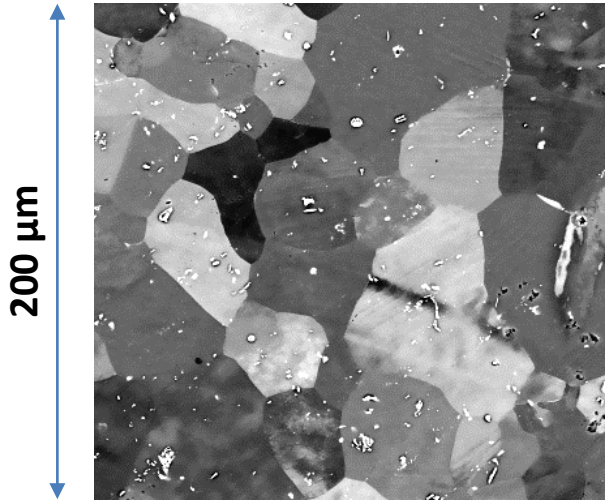


Disorientation map eCHORD / EBSD

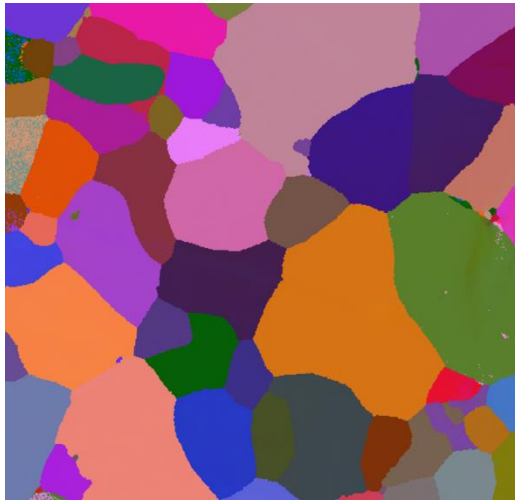
Profile database: 1 million theoretical profiles

Tension 15 kV / WD : 7 mm

COMPARISON WITH EBSD

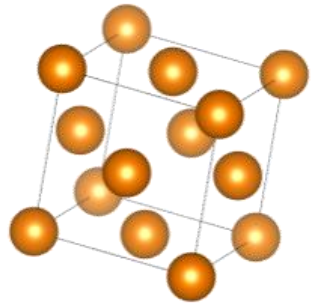


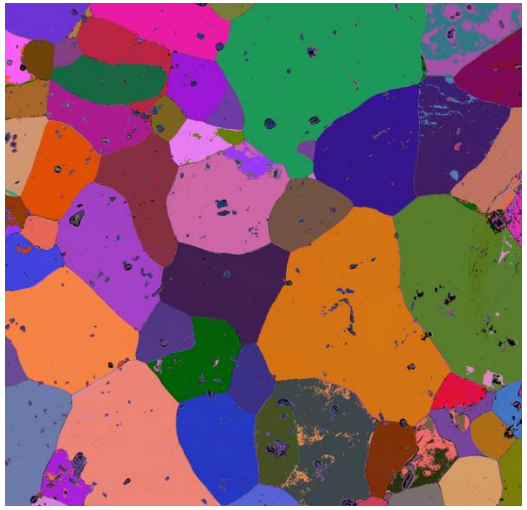
BSE image series



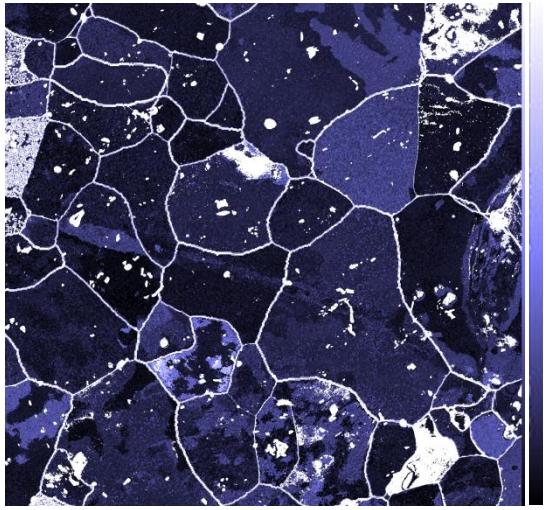
EBSD map (denoised)

Aluminum





eCHORD map (raw)



Disorientation map eCHORD / EBSD

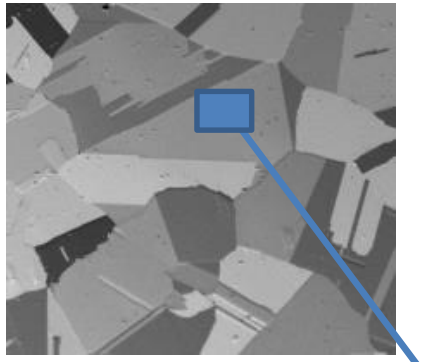
Profile database: 1 million theoretical profiles

Tension 15 kV / WD : 7 mm

ANGULAR RESOLUTION

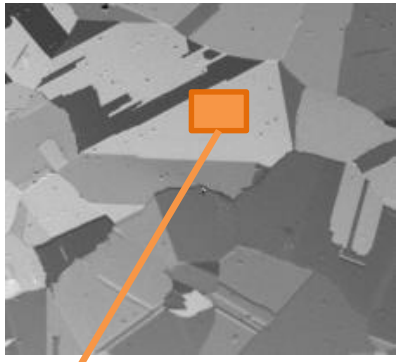
The idea: circular permutation of 6 frames in the image series

➔ **Peak shift of 6° in the intensity profiles // Disorientation of 6° between the two maps**

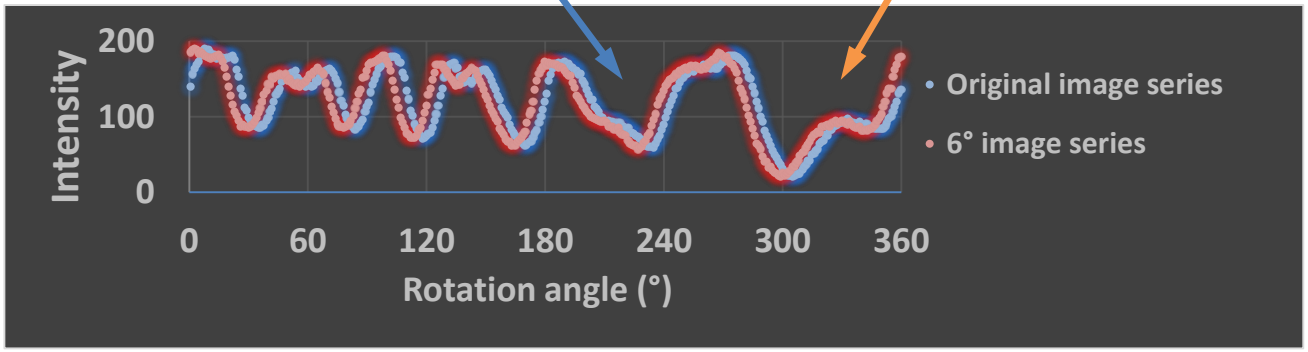


Circular permutation of 6 images

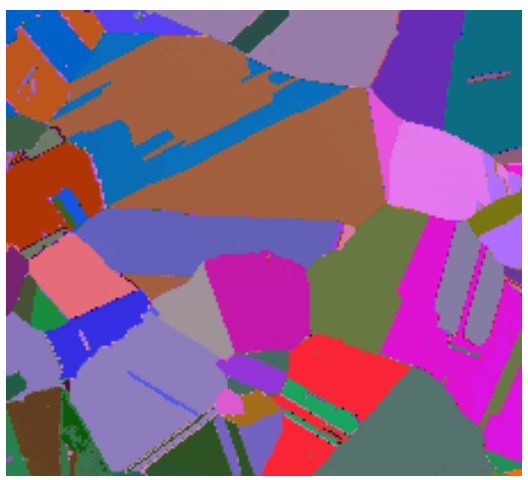
➔ new image series



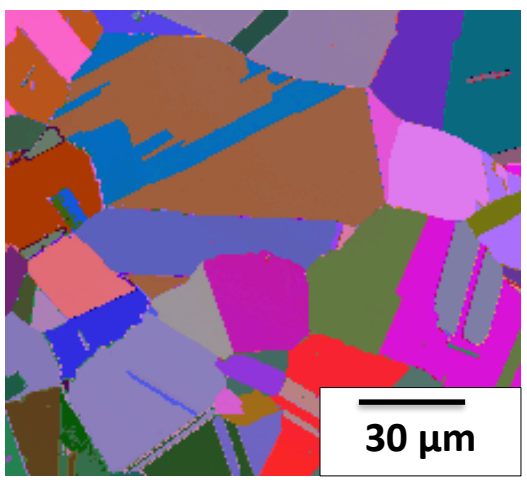
6° disorientation



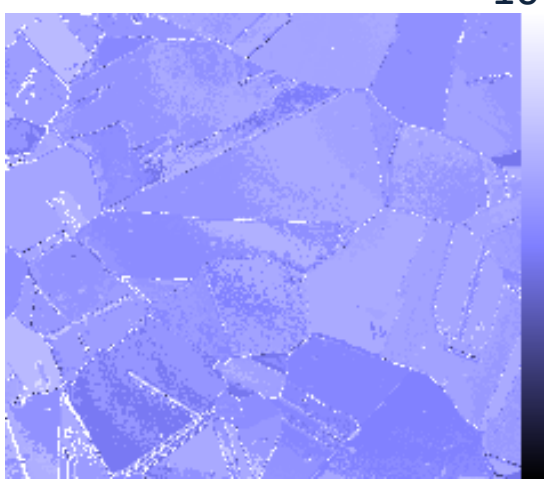
ANGULAR RESOLUTION



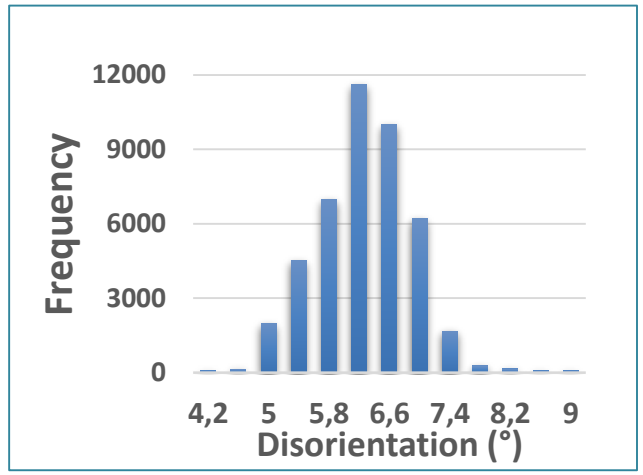
Original



6° apart



Disorientation map



Disorientation distribution

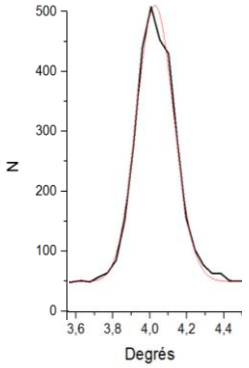
6.5° ± 0.9°

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°



TESCAN
PERFORMANCE IN NANOSPACE

Copper i-CHORD

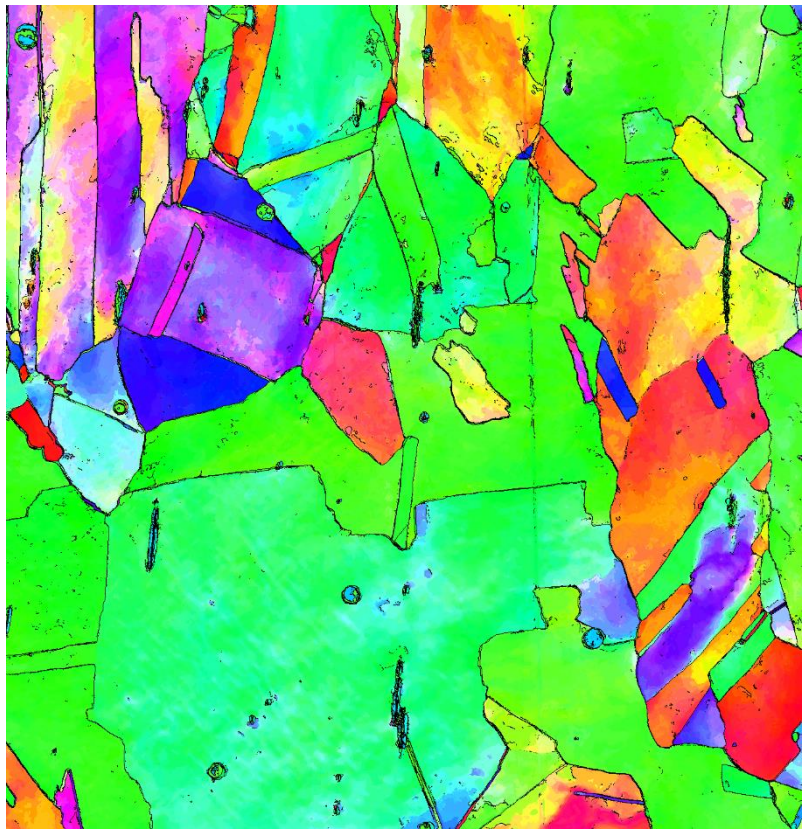
When Ion or Electron Channeling meets Crystal Orientation Mapping.

04.02.2019

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

TESCAN
PERFORMANCE IN NANOSPACE

SnCuAg Solder Bump e-CHORD

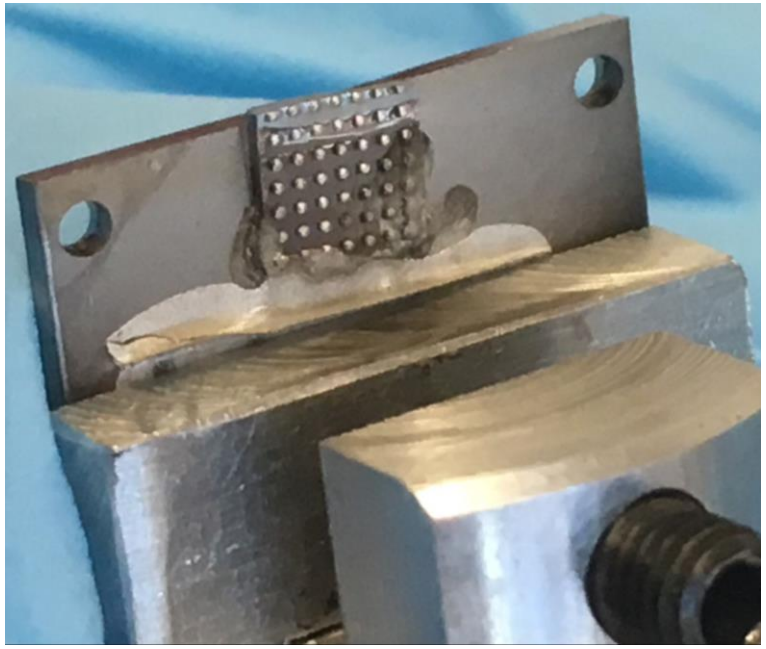
When Ion or Electron Channeling meets Crystal Orientation Mapping.

04.02.2019

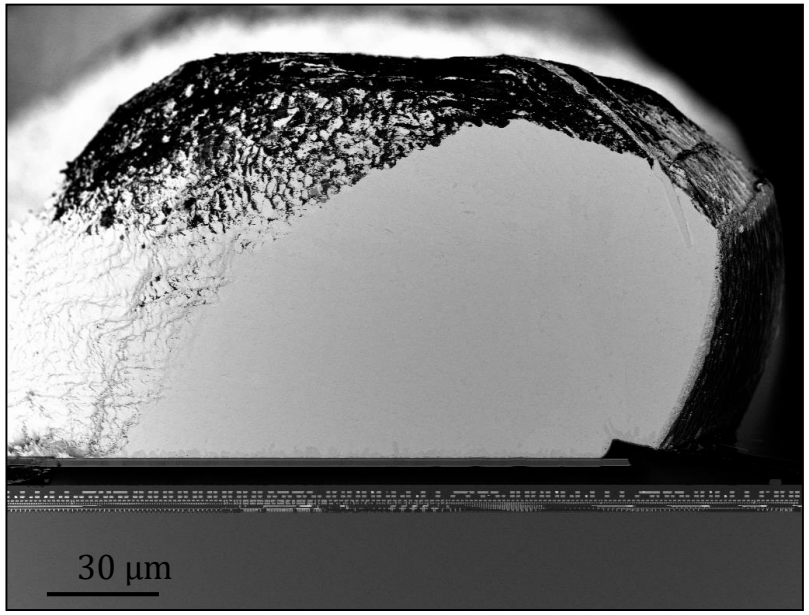
SnCuAg BUMP

In this case : no need to localized the default.

This sample was polished with an ion polisher => polishing was done on bumps which are on the side.

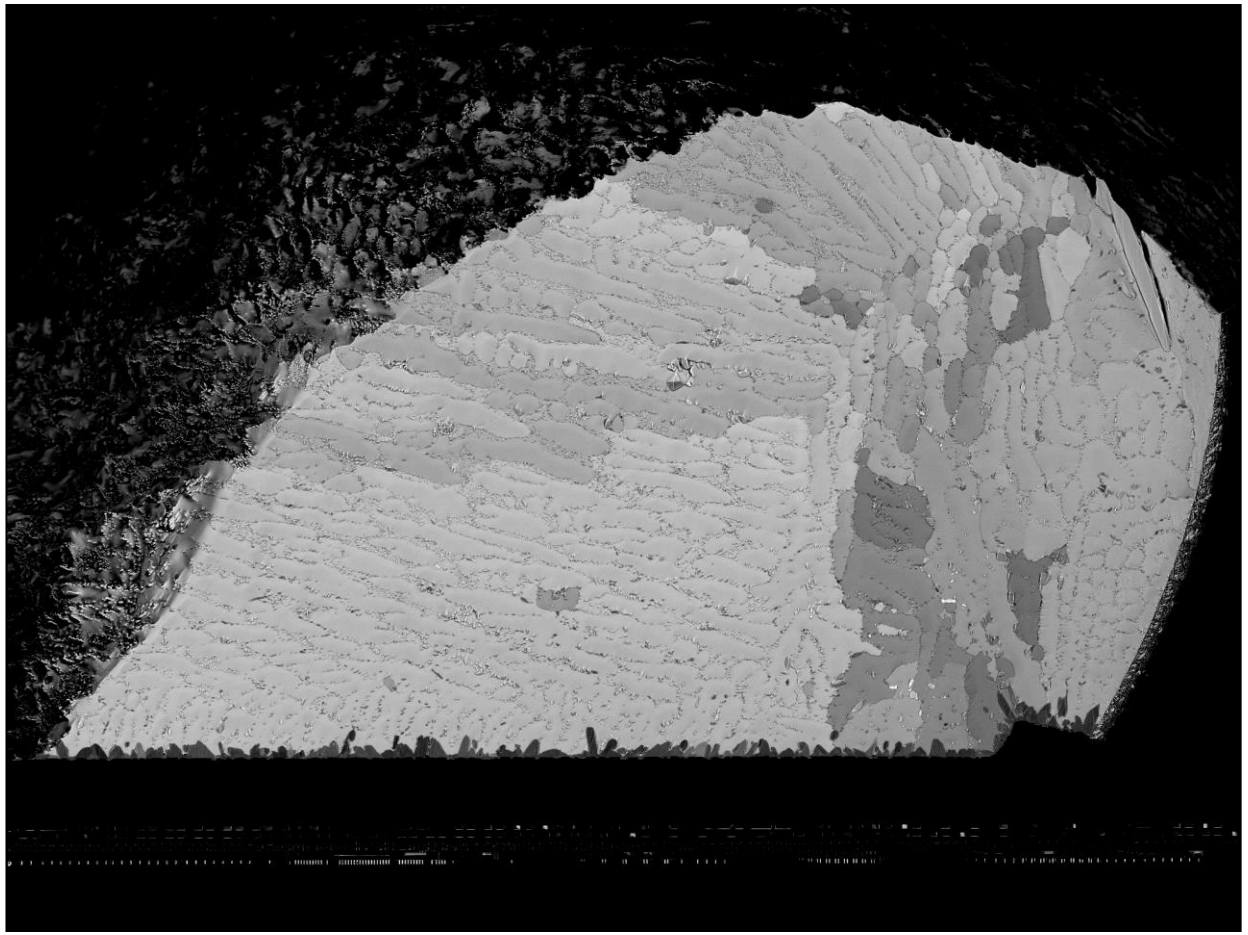


Courtesy of life.augmented



Sample preparation

BSE Image – 10kV

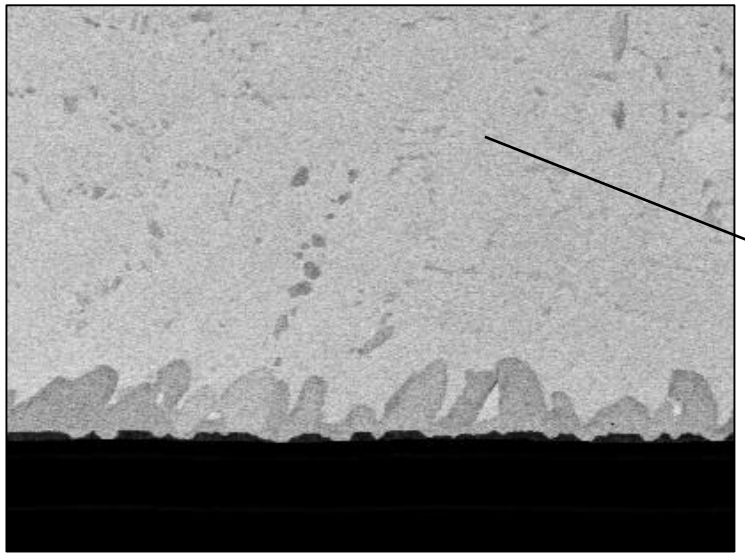


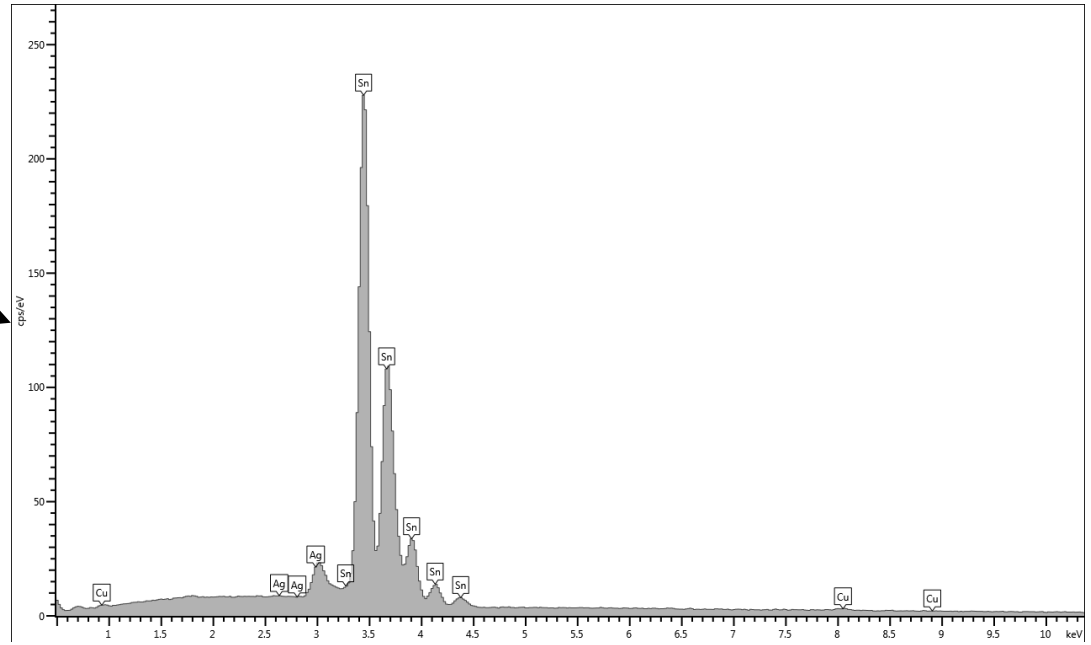
Courtesy of life.augmented

Sample preparation: Ar-BIB
X-Section
Sector milling angle : 90°
Rotation speed : 3rpm
Gun Tilt: 0°
Energy : 3kV, 30min + 0.5kV, 30min

SnCuAg : EDX spectrum on core of the solder bump

Accelerating Voltage: 20.00kV

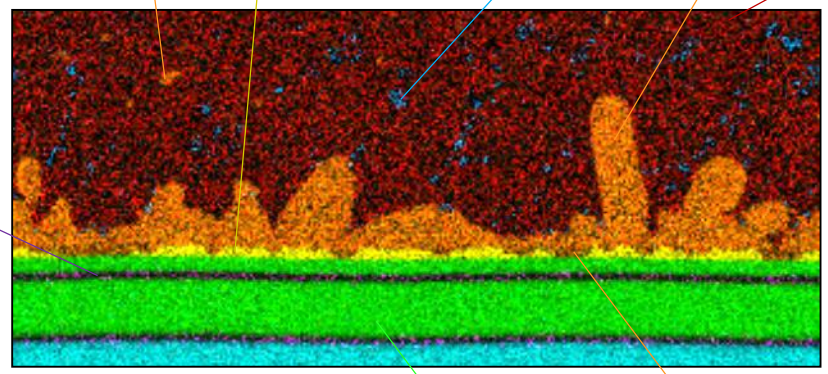




	Wt%	Wt% Sigma
Cu	0.57	0.11
Ag	3.79	0.13
Sn	95.65	0.17
Total	100.00	

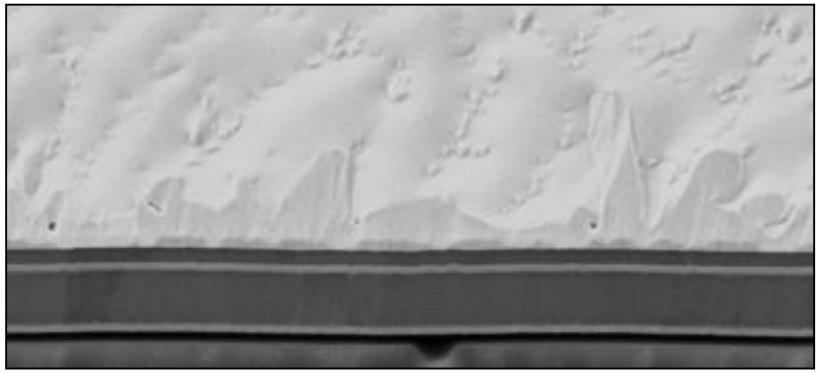
Courtesy of

SnCuAg : EDX Map



Sn | Ag | Cu | Ni, V | C, O | Al | Ti

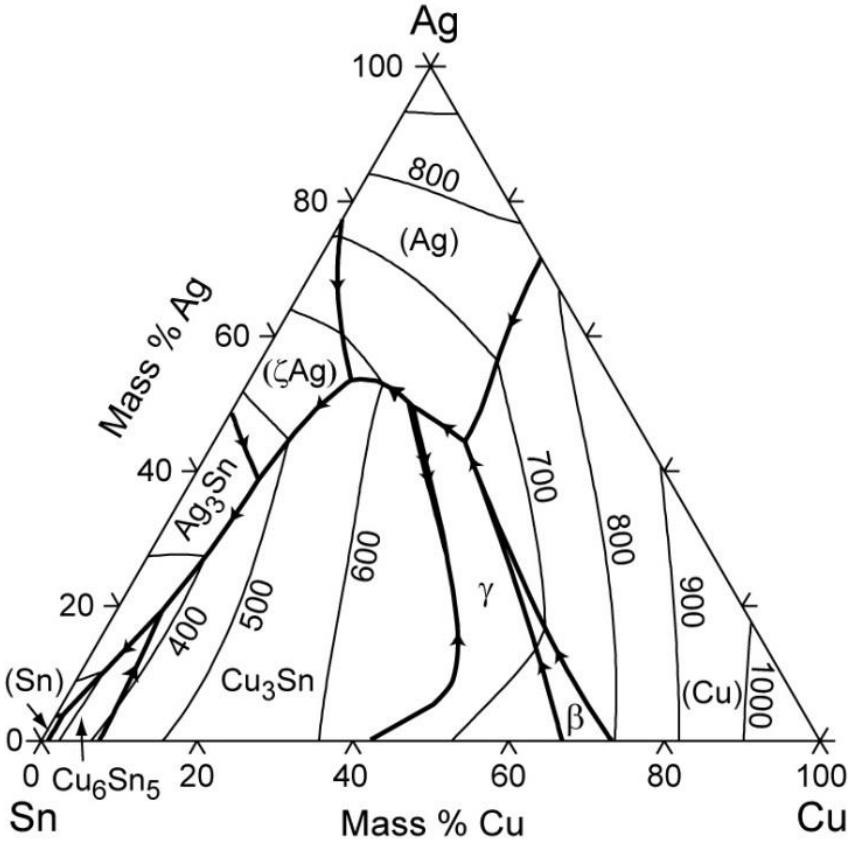
Lines:
- Sn L
- Ag L
- Cu L
- Ni L
- C K
- O K
- Al K
- Ti L

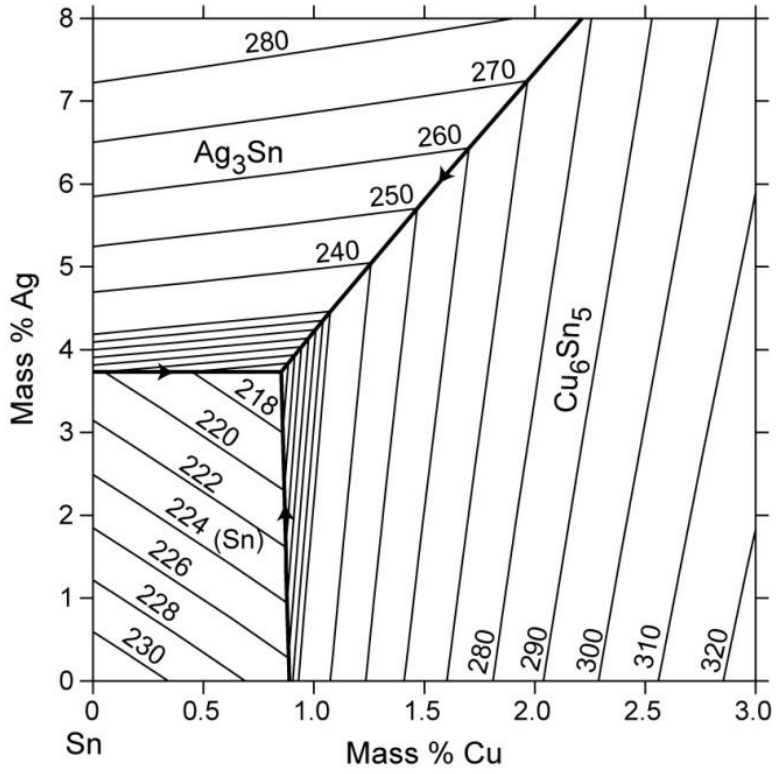


Accelerating Voltage: 5.0kV
Magnification: 3278x
Live Time: 166s
X-Max SDD 50mm² (Oxford Instruments)
320 pixels (22.1µm) x 144 pixels (9.92µm)

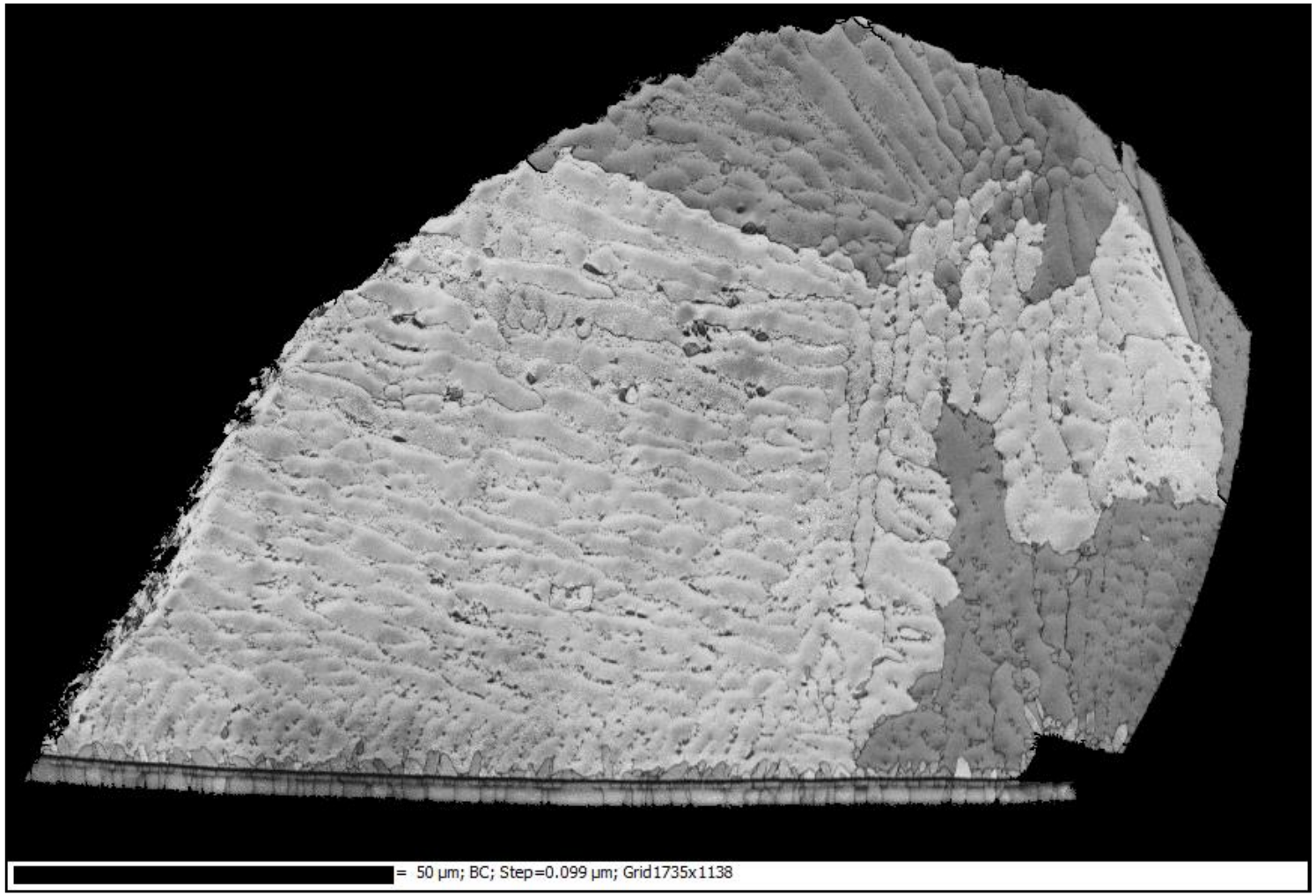
Courtesy of life.augmented

Ternary Phase Diagram





SnCuAg



Sn β Tetragonal

Ag_3Sn Orthorhombic

Cu_6Sn_5 Monoclinic

Cu_3Sn Orthorhombic

Cu_2NiSn Cubic

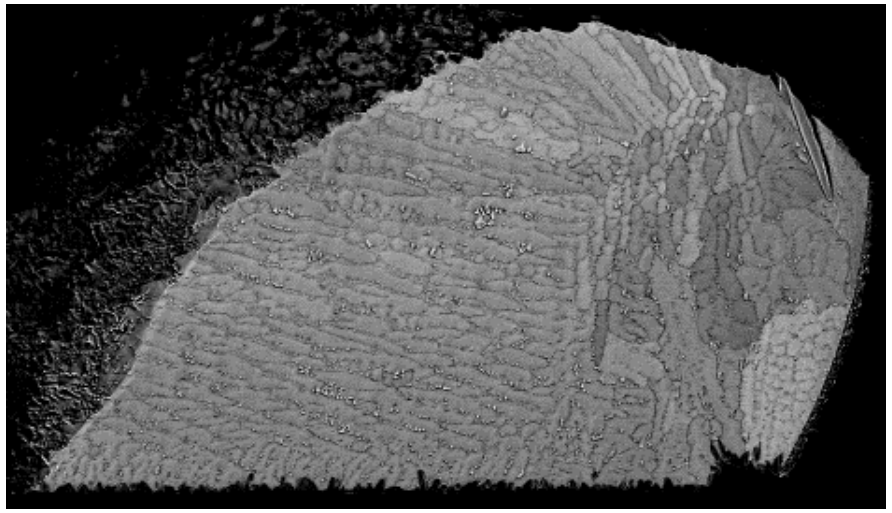
Ni_3V Tetragonal

Courtesy of

First Image Series at low magnification

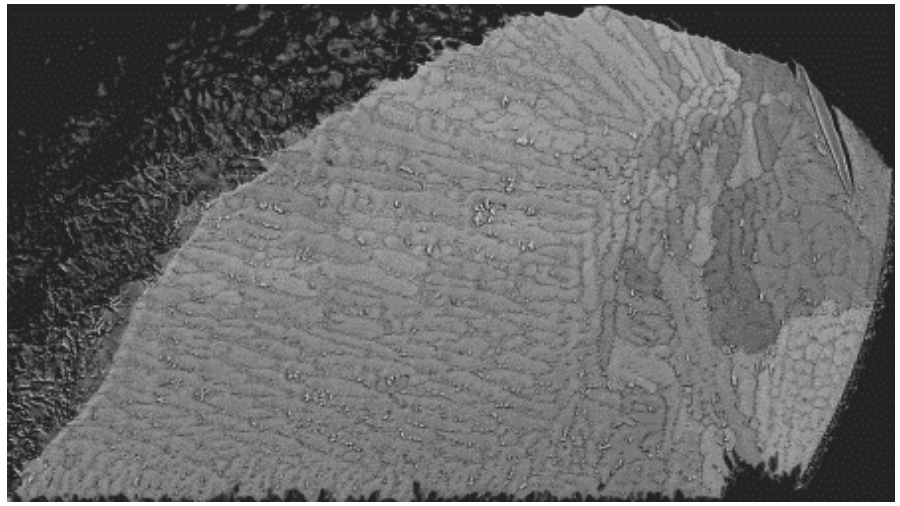
Acceleration Voltage	Current	Rotation	Acquisition Time
10kV	7 nA	360°/1° step	40 mn

Raw image series



160um

Treated image series



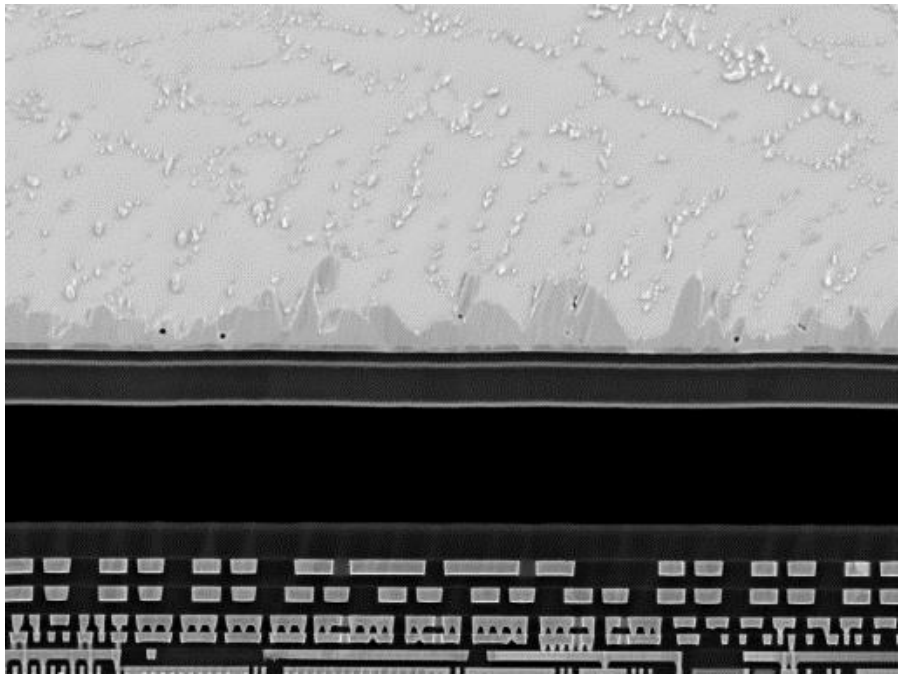
150um

Courtesy of

Second Image Series at high magnification

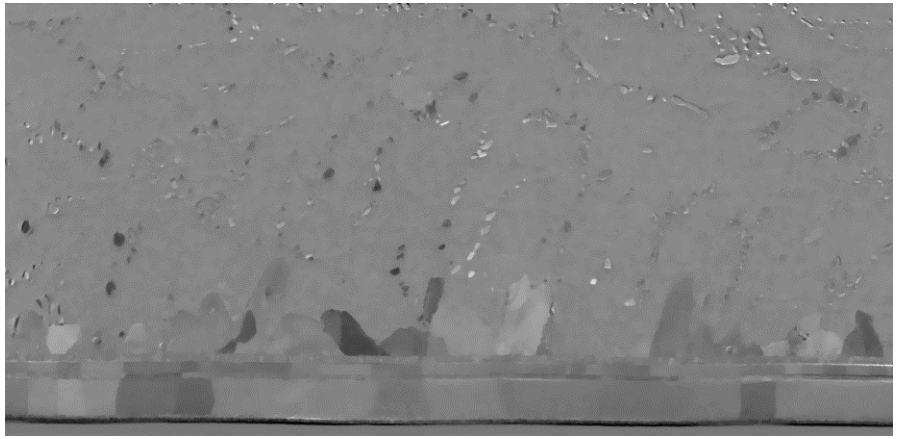
Acceleration Voltage	Current	Rotation	Acquisition Time
10kV	7 nA	360°/1° step	40 mn

Raw image series



30um

Treated image series



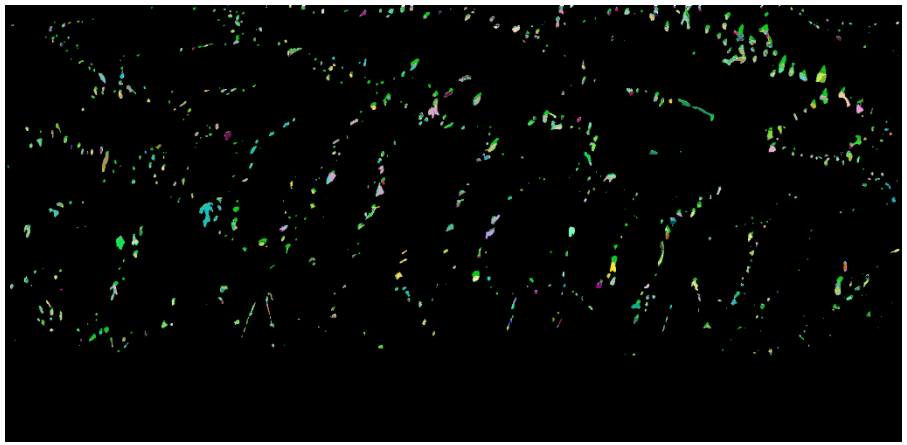
25um

Chemical and Topographic Contrast

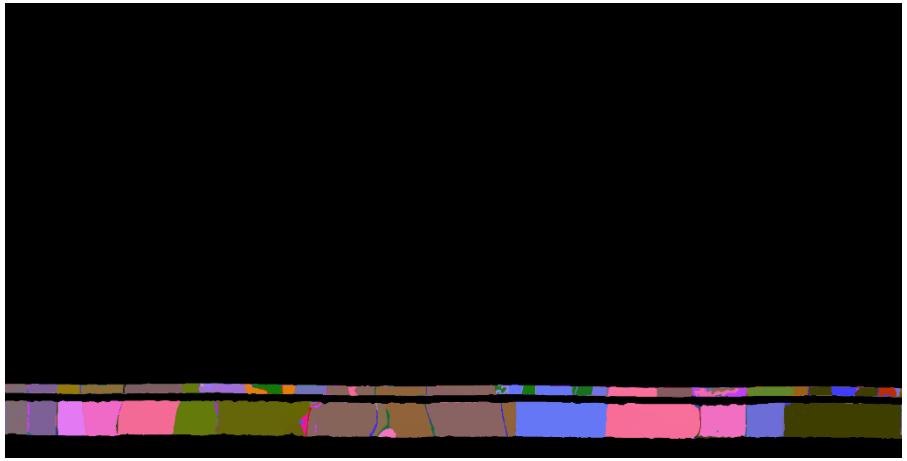
Courtesy of

SnCuAg: Exploiting the chemical contrast

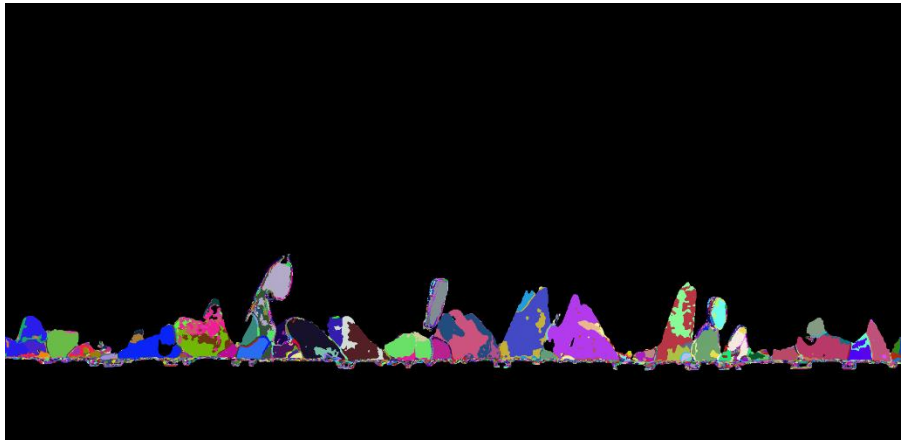
Ag3Sn



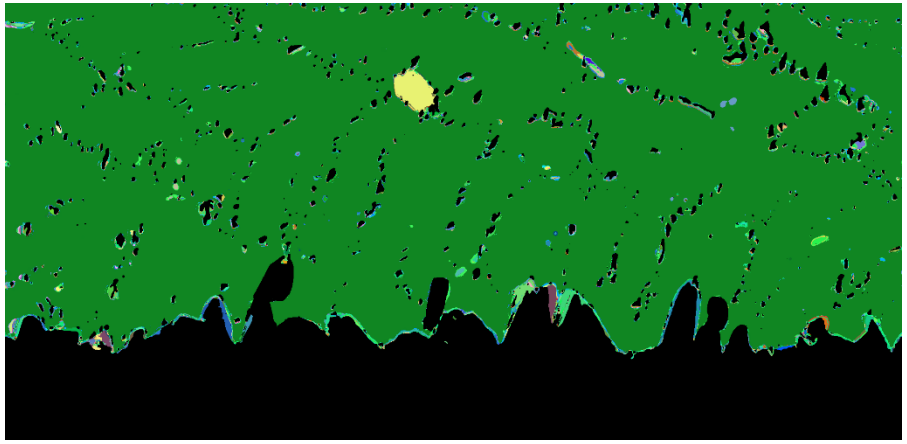
Al



Cu6Sn5



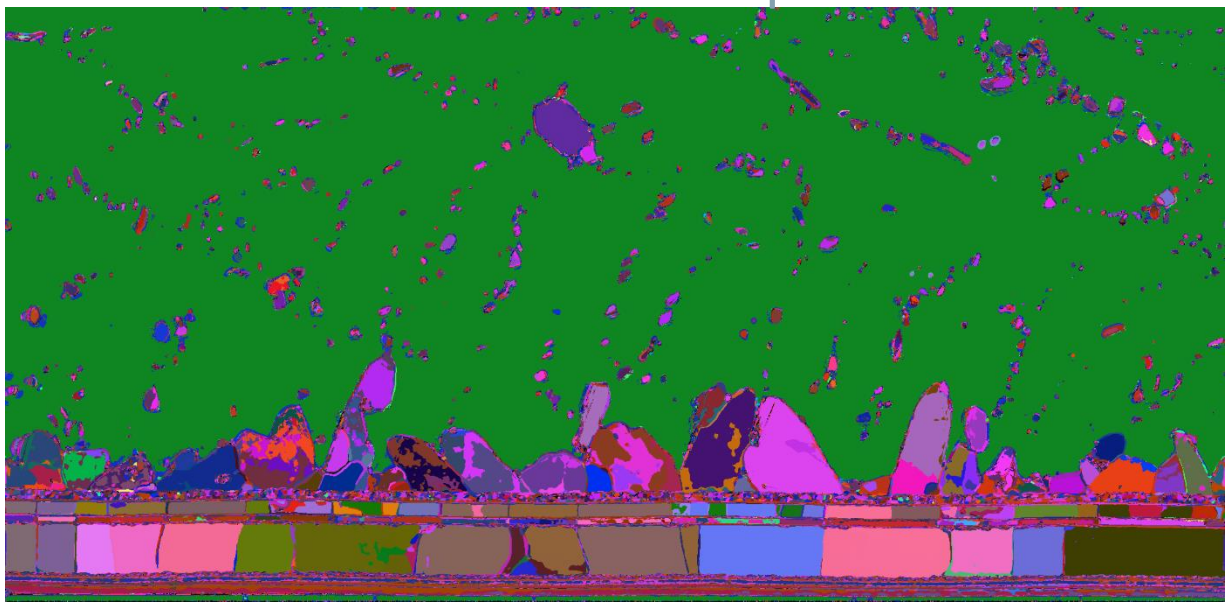
Sn



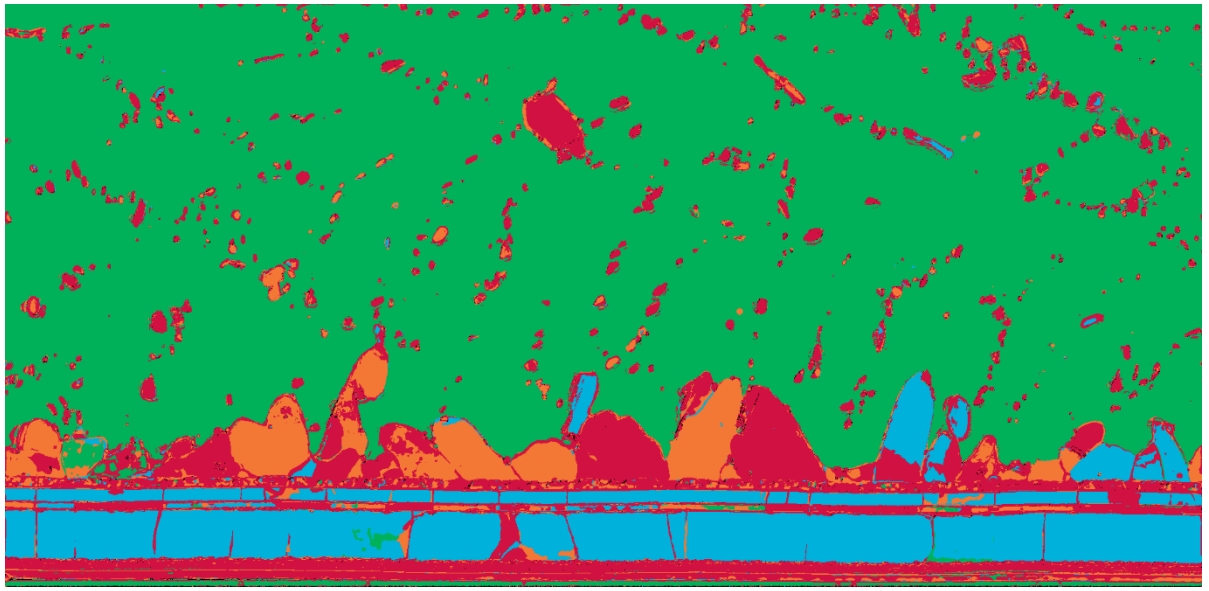
SnCuAg

Research on the complete database composed of 110M profiles from the 4 phases databases

Orientation Map



Phase Map



Al

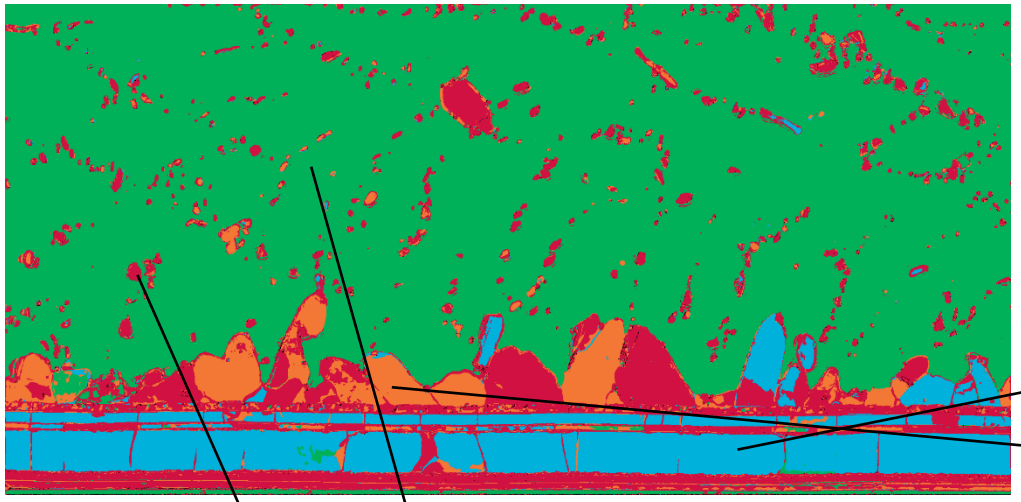
Sn

Ag_3Sn

Cu_6Sn_5

Courtesy of

SnCuAg

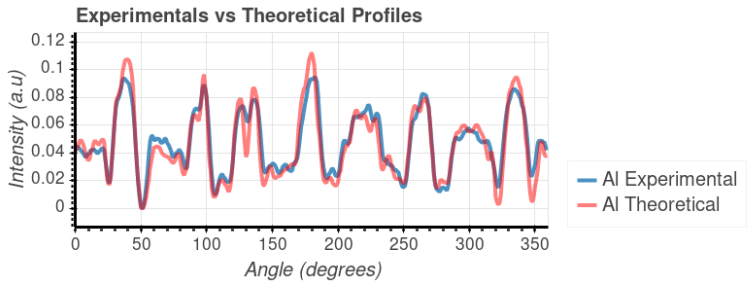


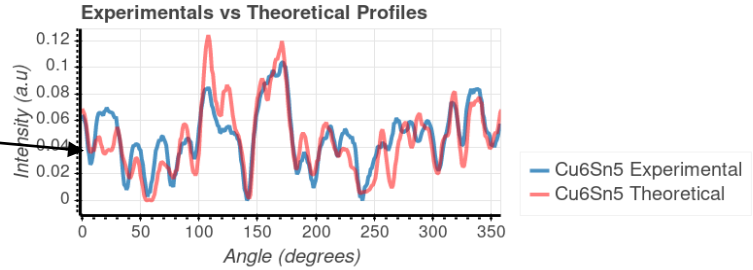
Al

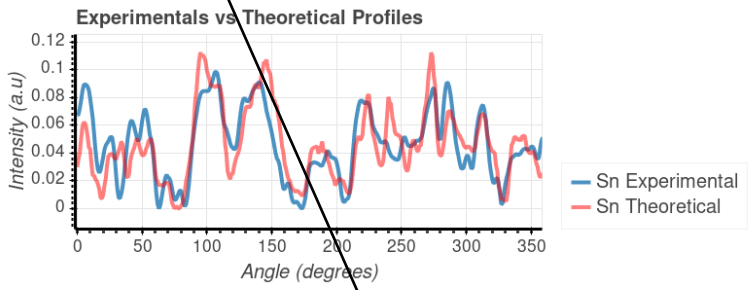
Sn

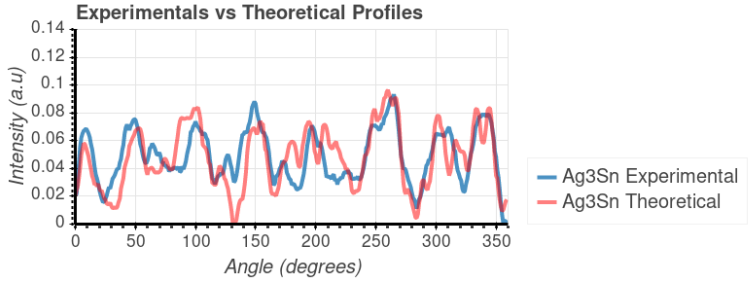
Ag_3Sn

Cu_6Sn_5





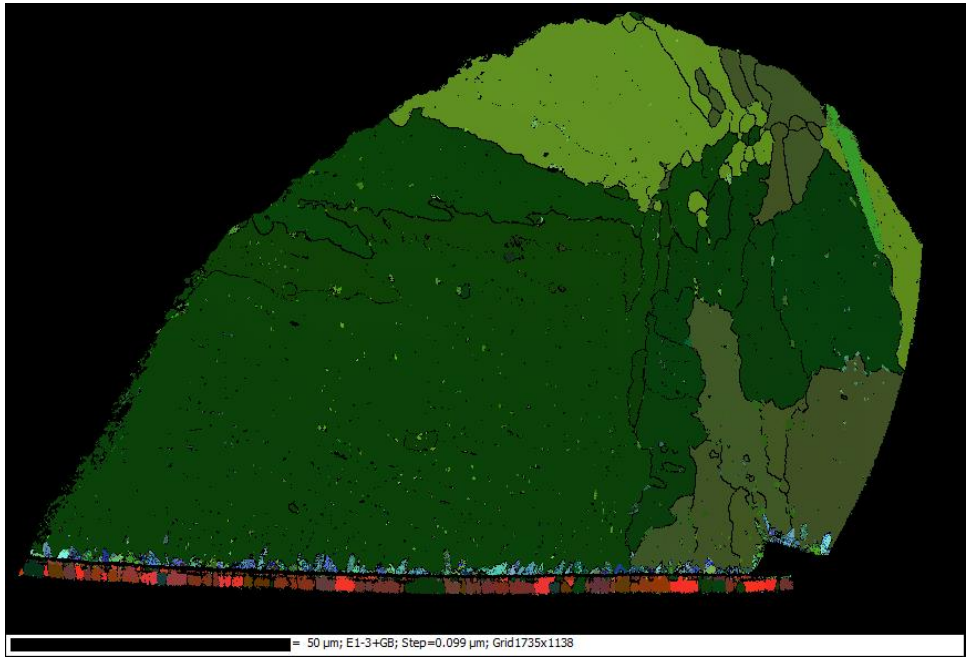




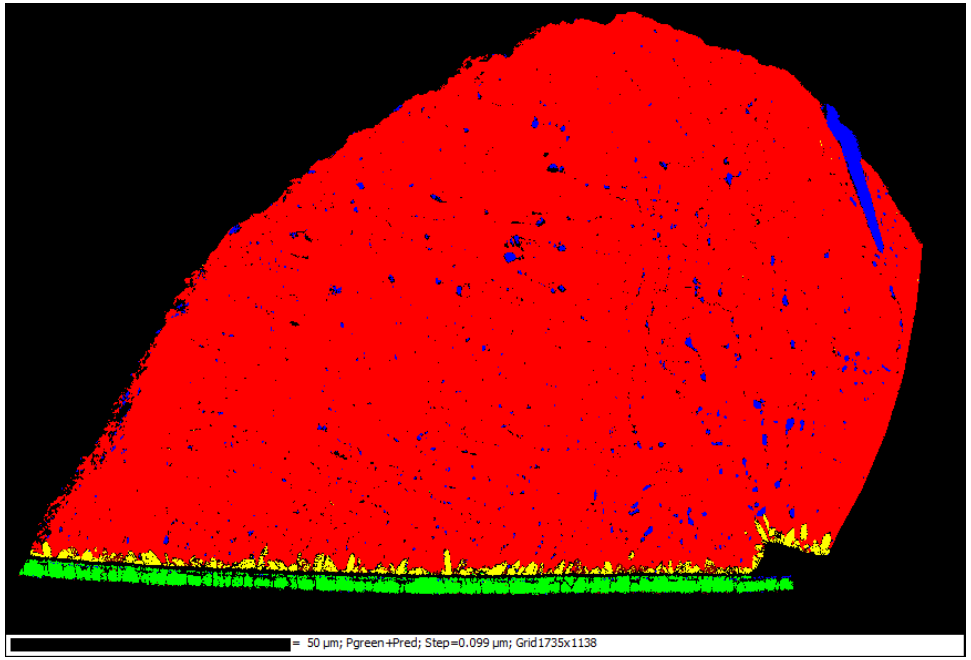
Good correspondence between theoretical profiles and experimental ones for each phase

Courtesy of

Comparison with EBSD

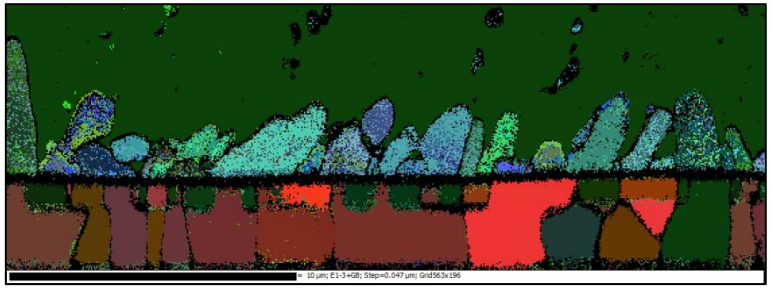


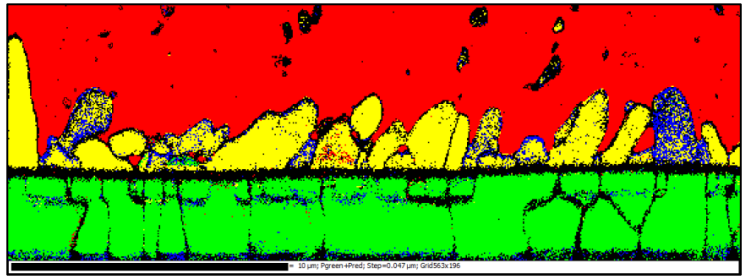
Orientation Map



Ag_3Sn Sn β Cu_6Sn_5 Al

Phase Map





Courtesy of

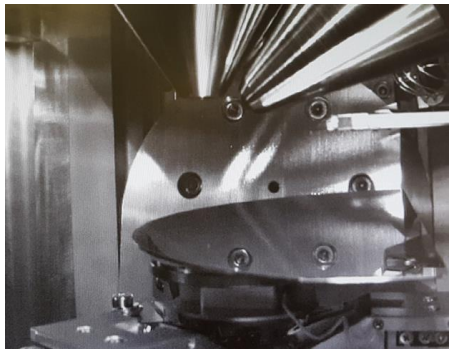
TESCAN
PERFORMANCE IN NANOSPACE

Al ribbon
e-CHORD

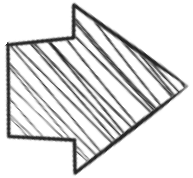
When Ion or Electron Channeling meets Crystal Orientation Mapping.

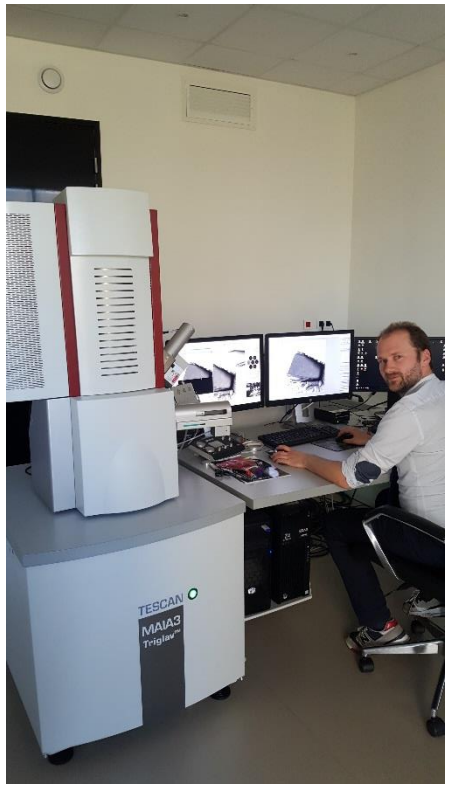
04.02.2019

Al Ribon



Quality of the polishing is good
Large enough area too avoid shadowing





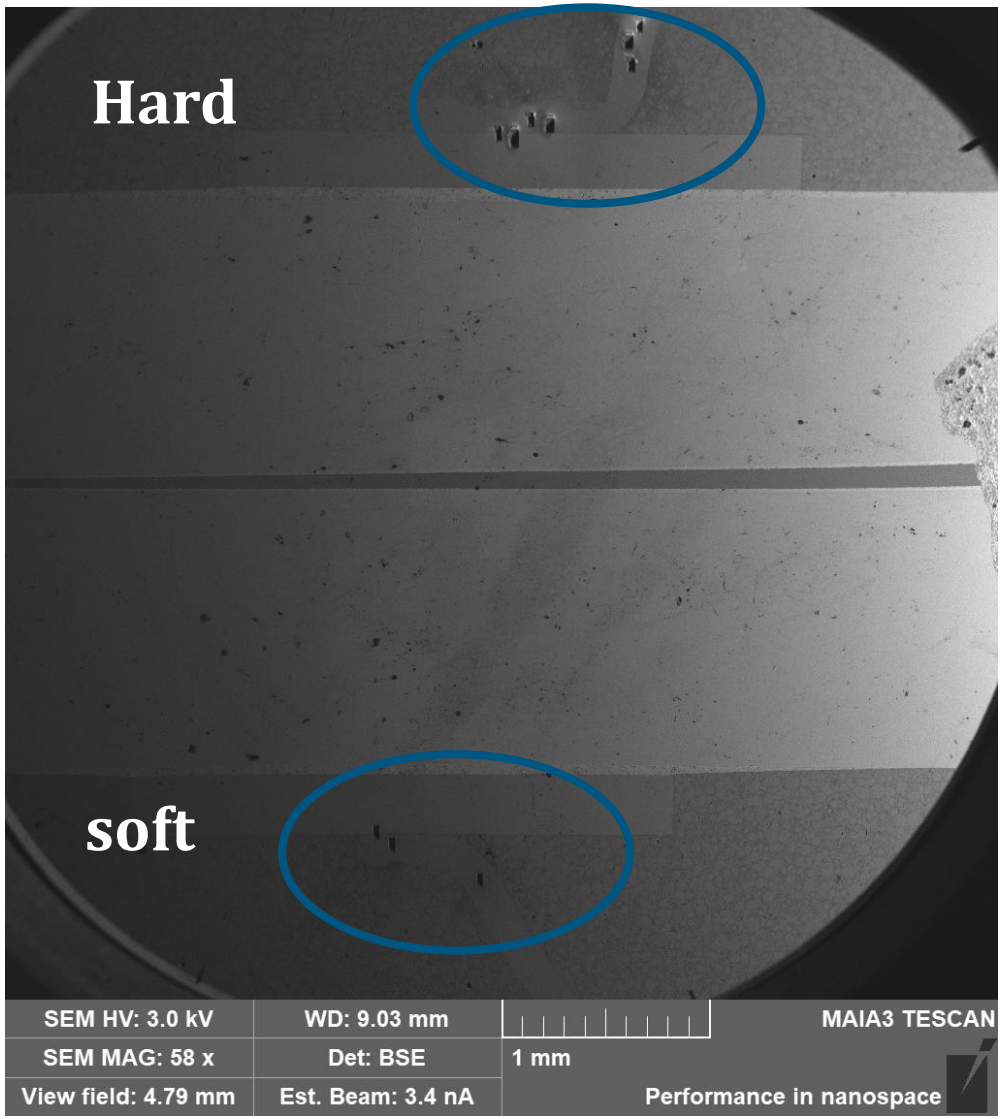
- Series of images taken with SEM (Maia)
- Condition of acquisition : 3 keV WD = 9mm
- Time for the acquisition : 50 min

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

Al ribbon



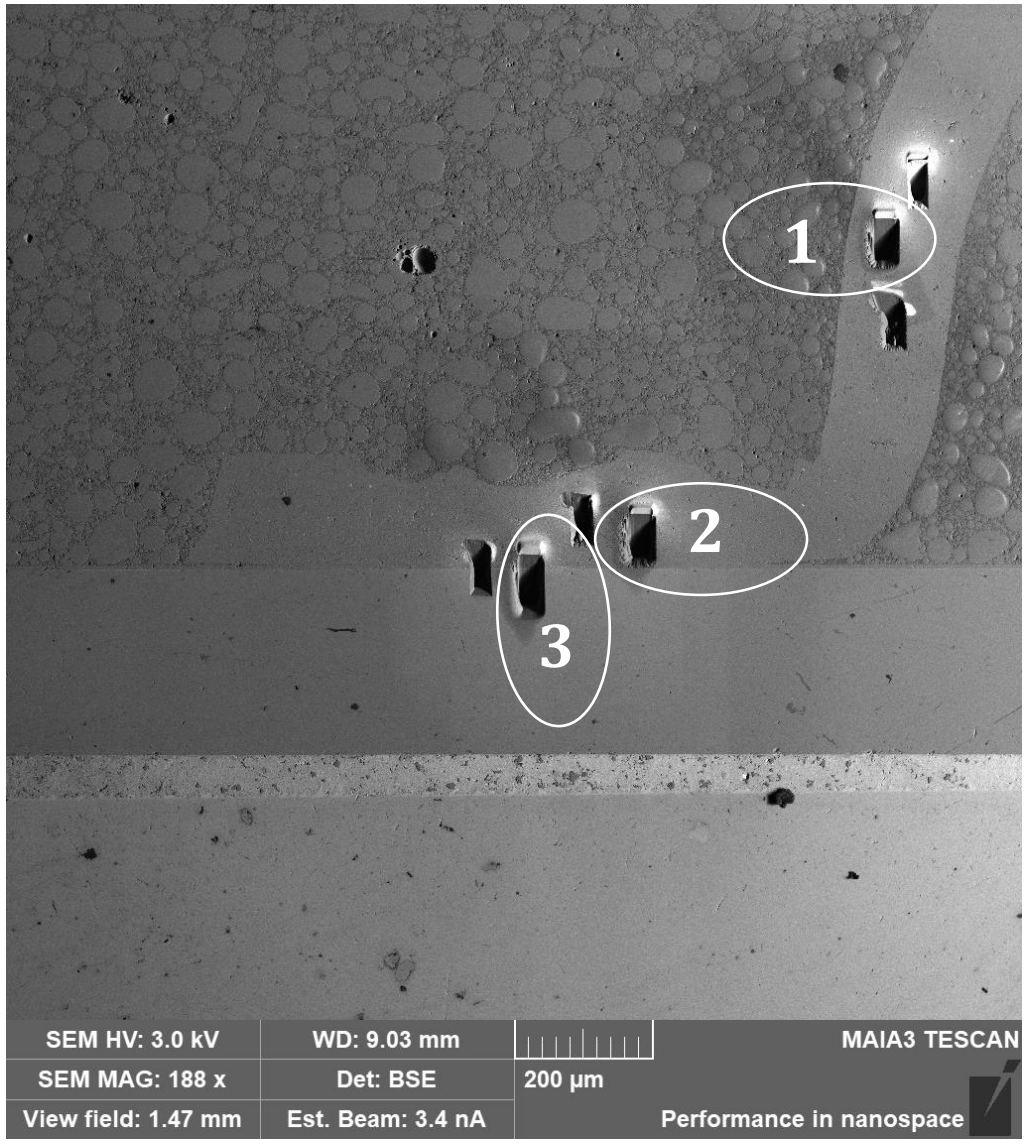
2 parts on the sample : soft and hard
In each case : 3 regions of interest

- Wire
- Bottom wire
- Interface metal-wire

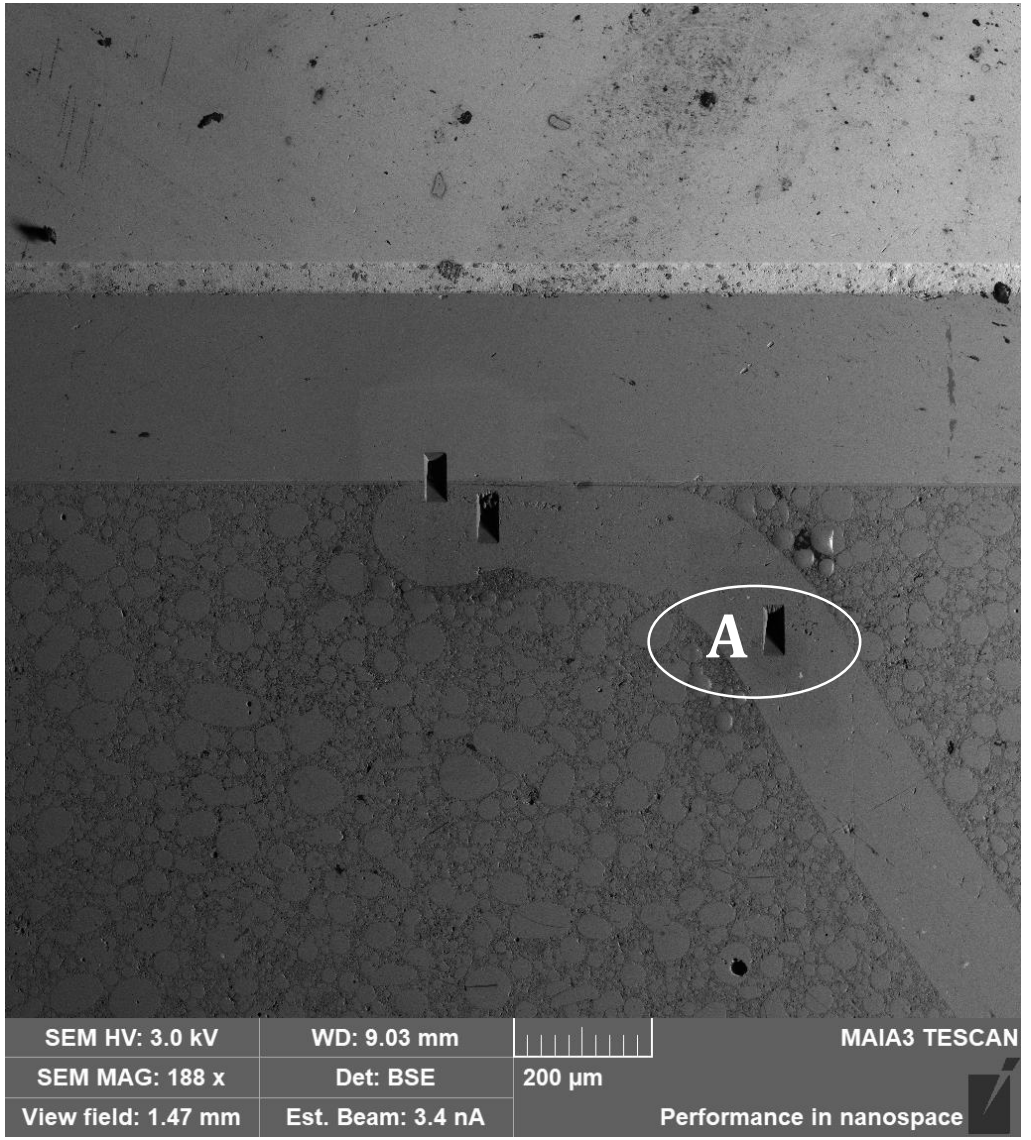
Goal : study the crystallographic orientation and grain size at different region of interest.

Courtesy of

Al ribbon- Hard part

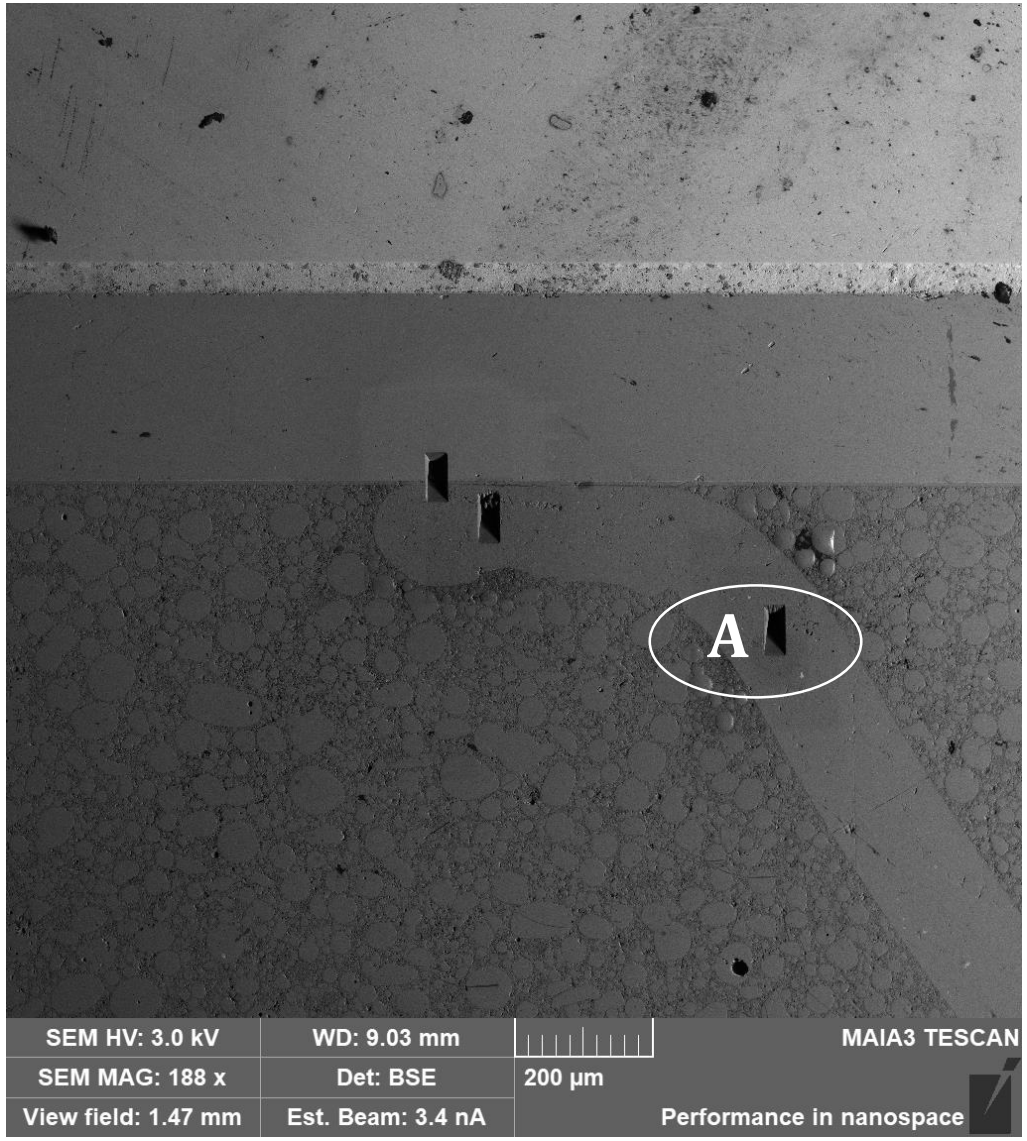


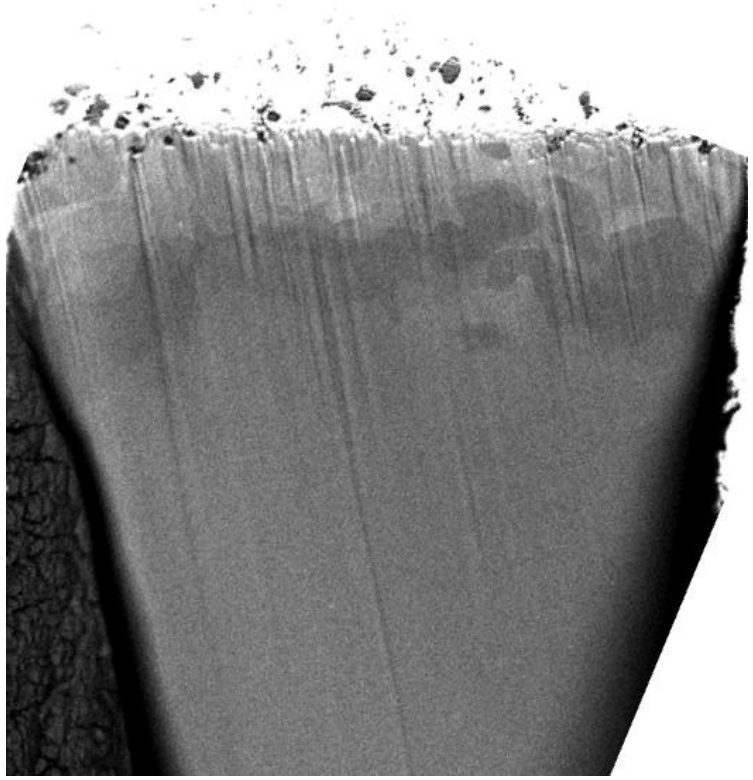
Al ribbon- Soft part



Courtesy of

Al ribbon- Soft part

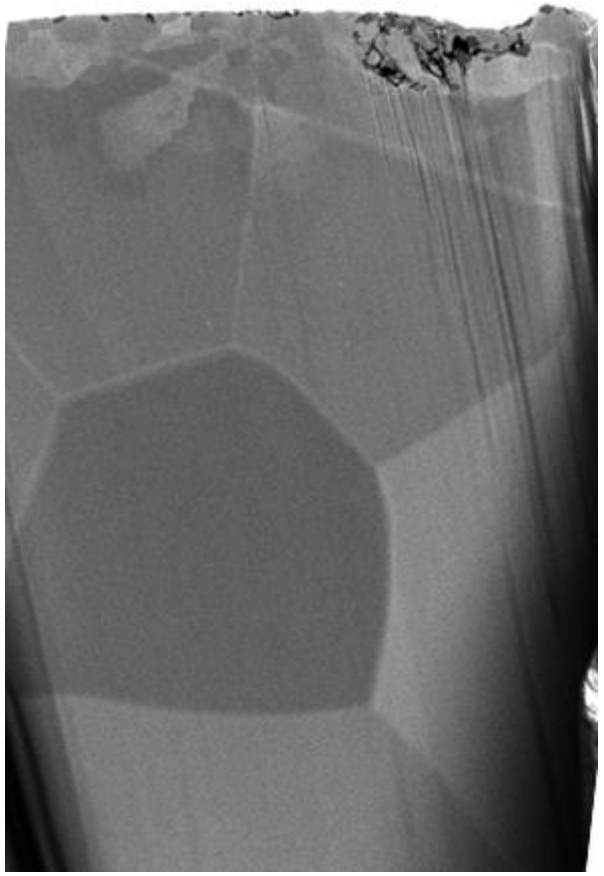




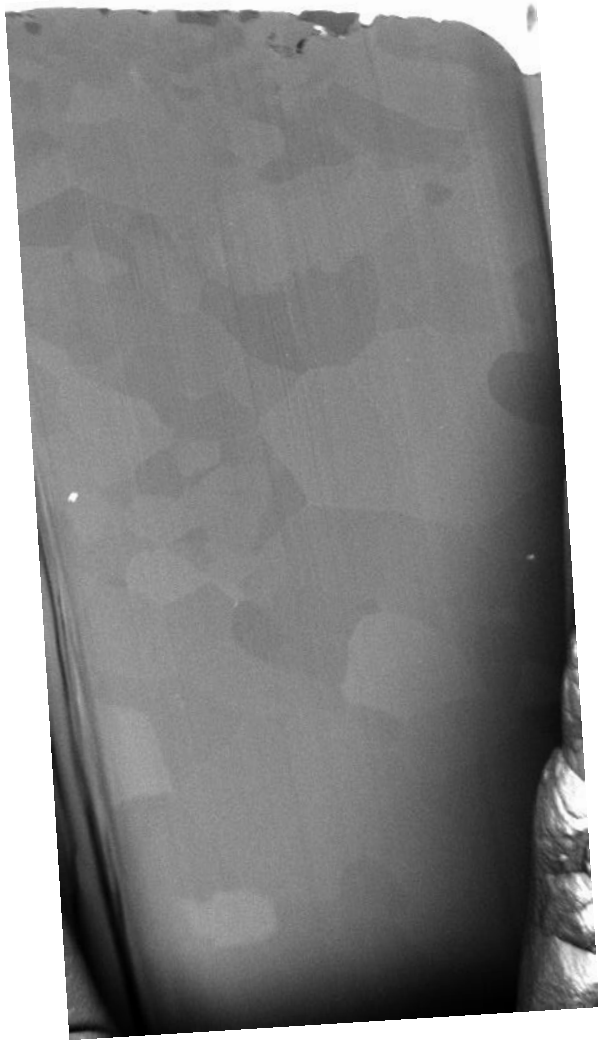
Courtesy of

Al ribbon: Hard part

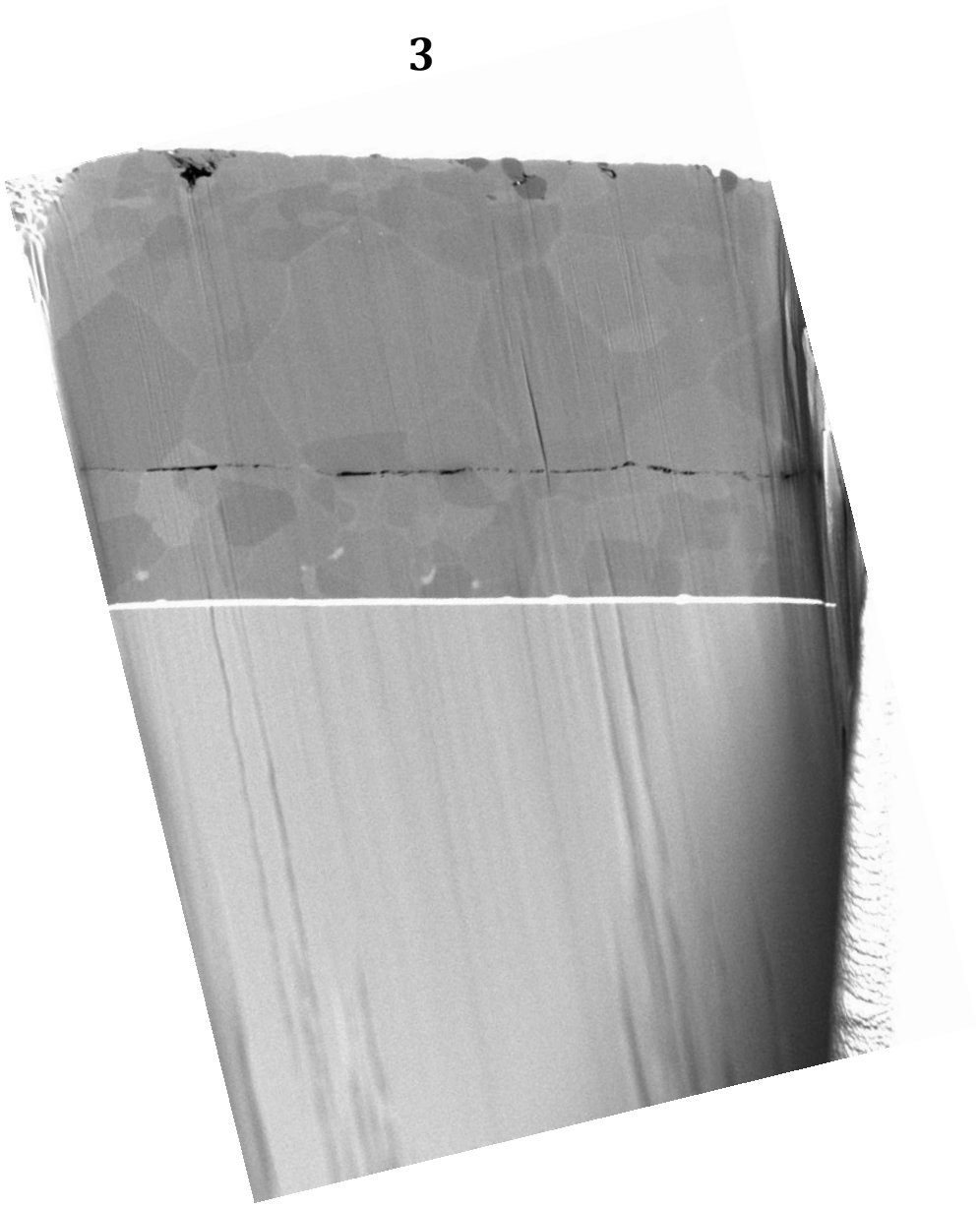
1



2



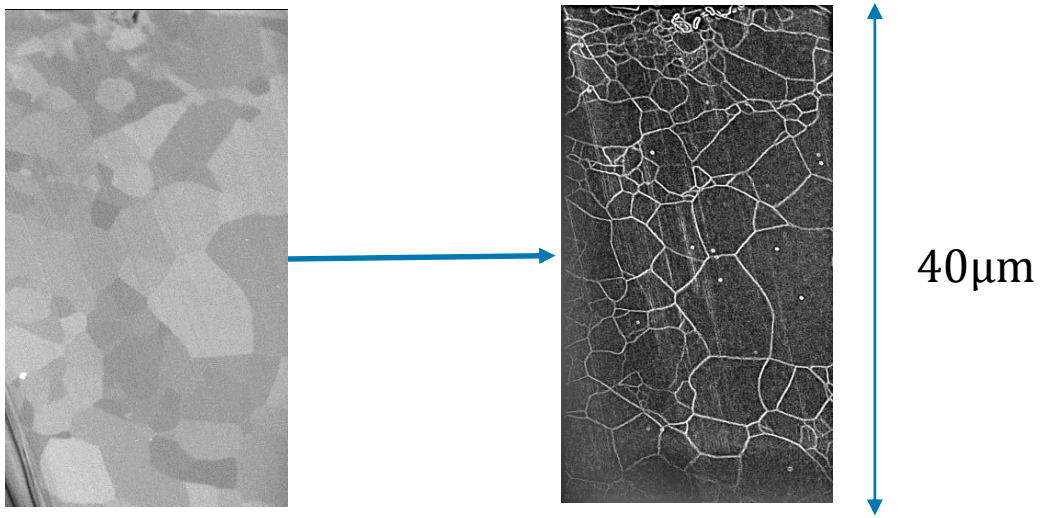
3



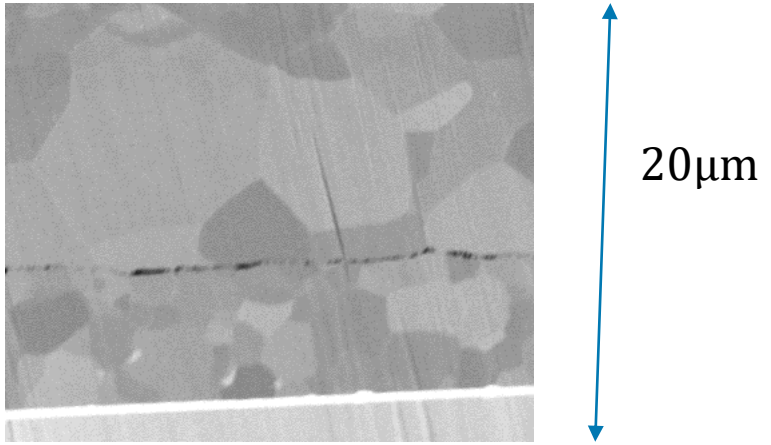
Courtesy of life.augmented

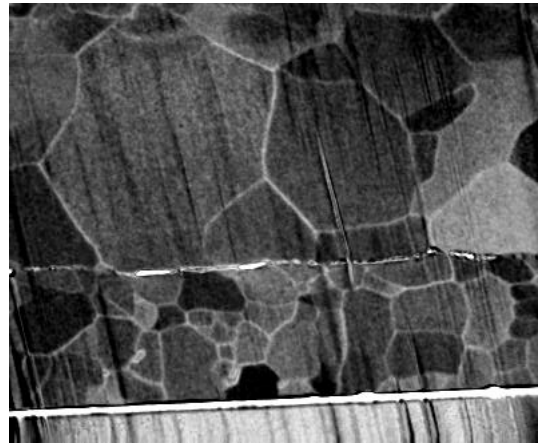
Al ribbon: Grain Size

2



3





Courtesy of

TESCAN
PERFORMANCE IN NANOSPACE

Aliminum Sputtering on Silicon wafer e-CHORD

When Ion or Electron Channeling meets Crystal Orientation Mapping.

04.02.2019

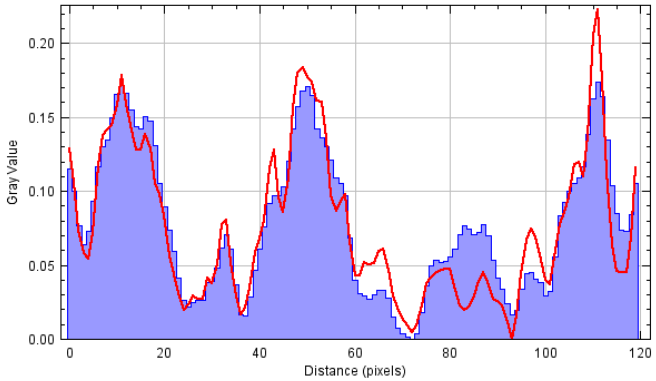
Sputtered Al

Conditions

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

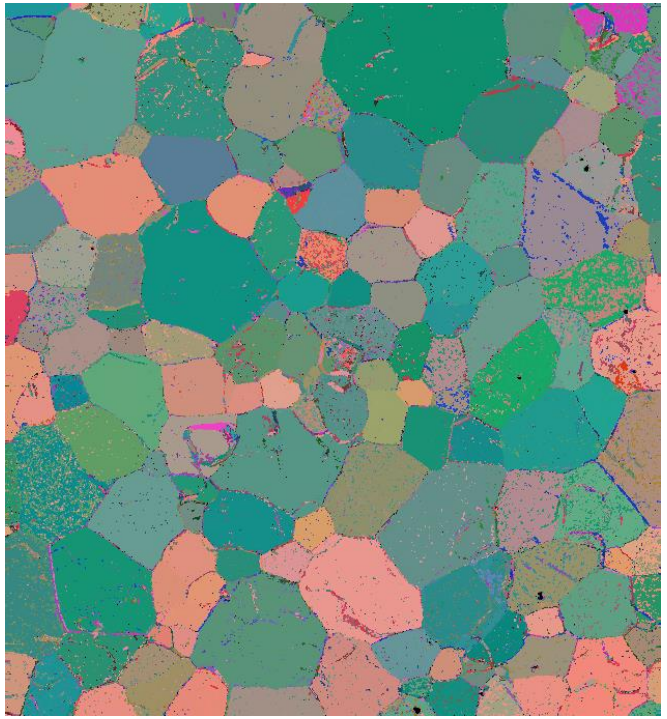
RAW (no denoising) IMAGE SERIE

- No denoising
- Profil clustering
- Average profil by cluster
- Profil indexation



90 µm

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

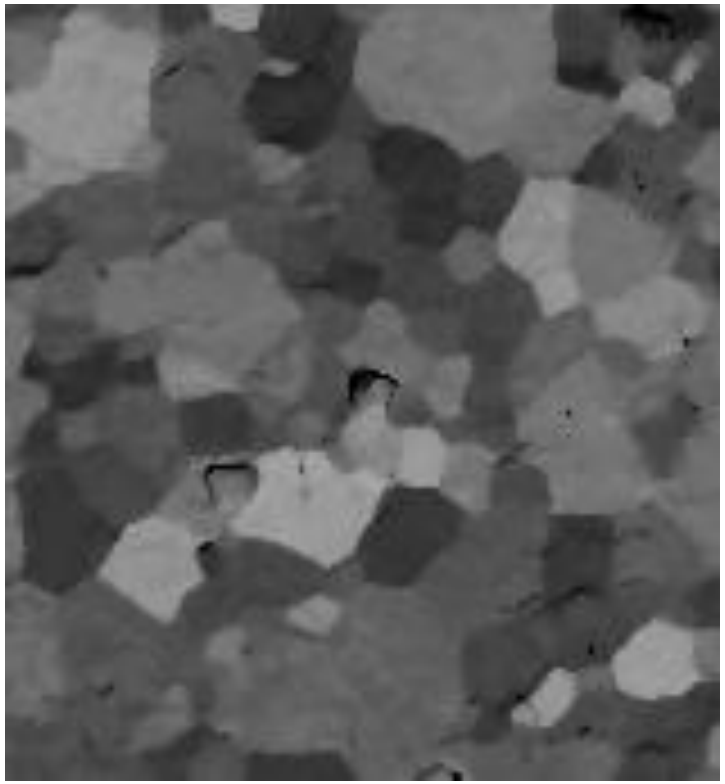


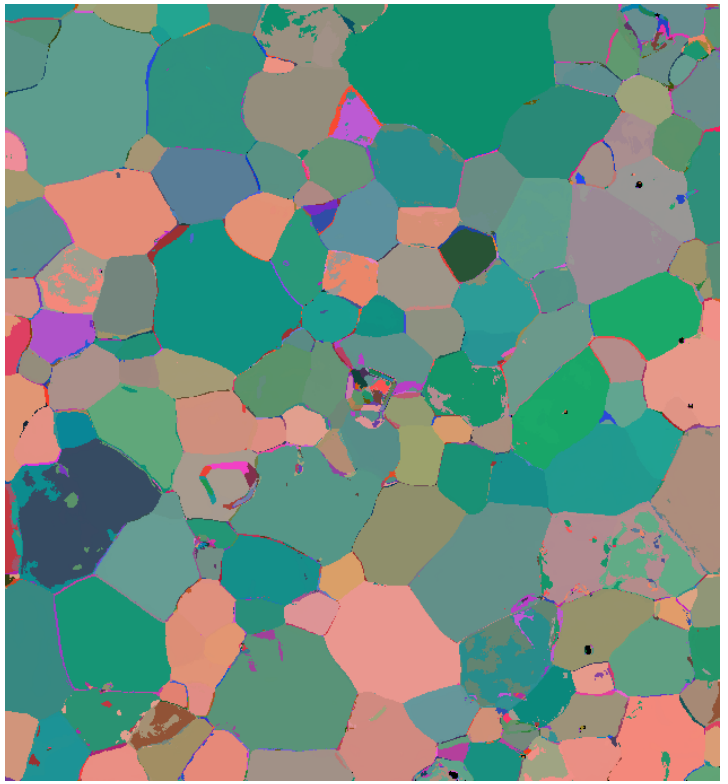
Courtesy of life.augmented

DENOISED IMAGE SERIE

- Denoising
- Profil clustering
- Average profil by cluster
- Profil indexation

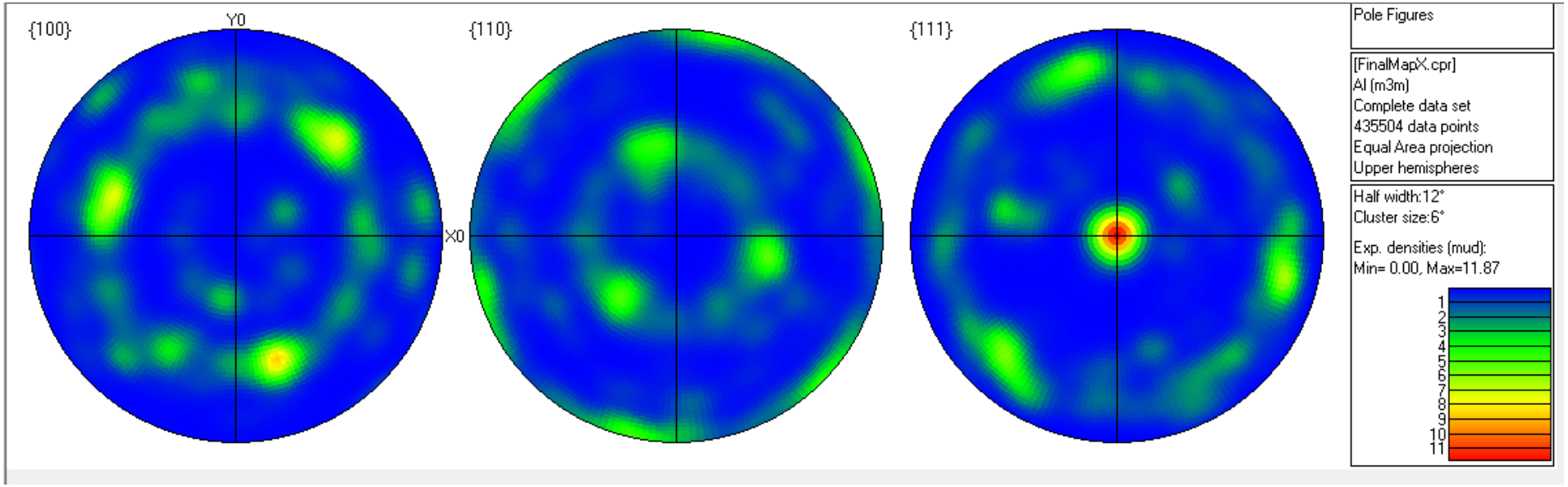
90 µm





Courtesy of

Film orientation



[111] perpendicular to the surface

Preferential Crystallographic growth along [111] direction

Courtesy of

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Perspectives and Messages to take home

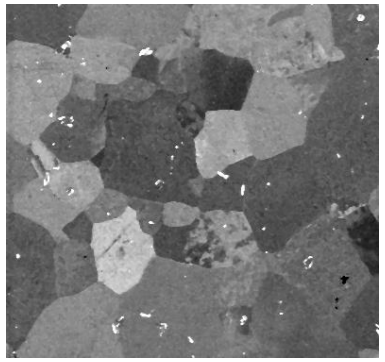
Fast CHORD

Strategies

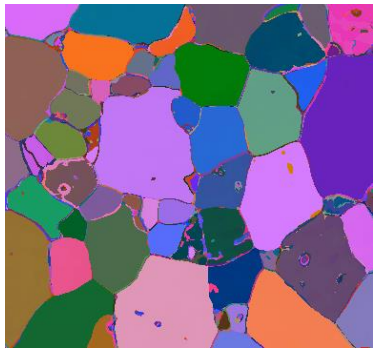
1. Reducing number of images in the series
2. Reducing dwell time per pixel

Aluminum. Tension 5 kV / WD: 7 mm

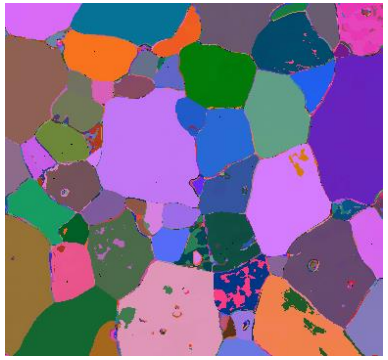
360 images ➔ 45 images



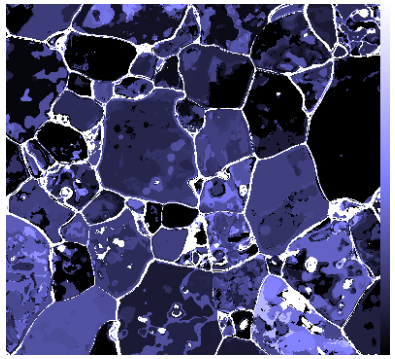
150 µm



eCHORD reference map

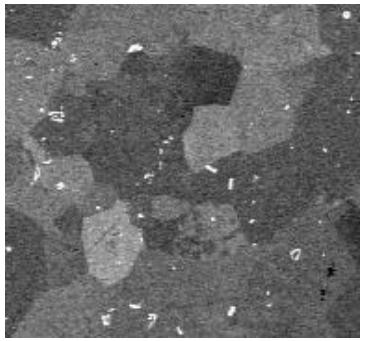


eCHORD map with 45 images

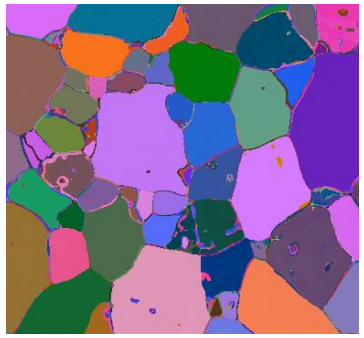


Point-to-point disorientation map

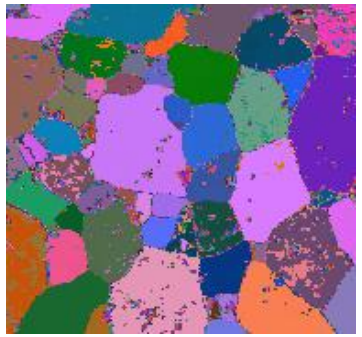
45 images of size 200 x 150 pixels
200 ms per image



150 µm



eCHORD reference map



eCHORD fast map (45 images)

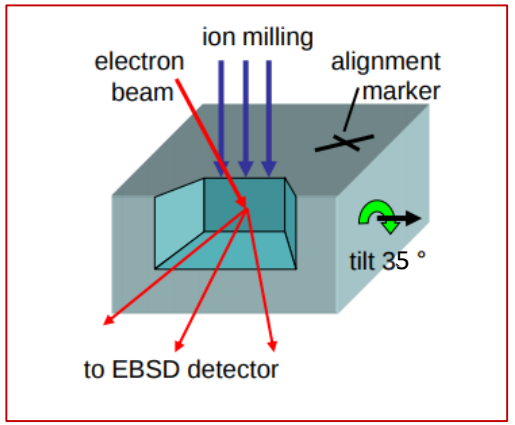
Total acquisition time

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

3D CHORD

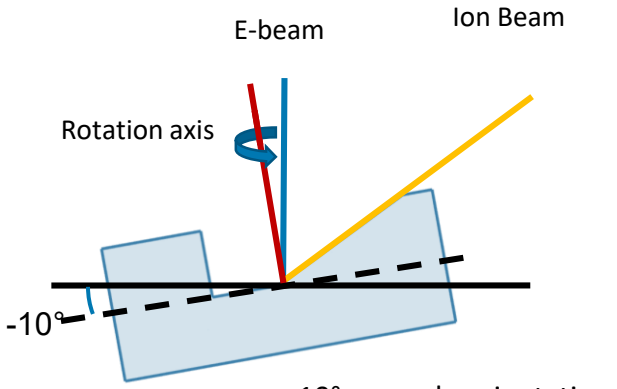
Current situation (FIB + EBSD)

- Complex acquisition
- Necessity to prepare a block to be extracted.

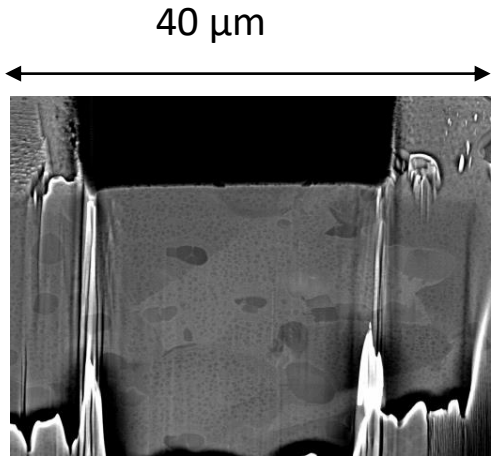


Explored way

coupling eCHORD + FIB ➔ 3D CHORD



-10° : sample orientation



Avantages

- Region of interest selected
- Fast cartography on each slide

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

Simple method to define :

- Crystal orientation
- Grain Size

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

Simplicity :

- Only a backscattered electron detector is required

Other advantages :

- Working at low tension (down to 1.5 kV)
  - Smaller interaction volume
  - Less depth averaging
  - Less charging

- Cross section preparation is possible
- anywhere on the surface

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Thank you for your attention !