EBSD en mode pression variable: un exemple d'application pour l'étude des biomatériaux

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Sommaire

→ Imagerie pression variable (VP) et nanoVP
→ Rappels EBSD: théorie et pratique
→ EBSD VP: influence des paramètres expérimentaux (mode VP, pression, binning, background, courant)
→ Traitement des données (Post-processing)
→ EBSD VP appliquée aux biomatériaux
→ Conclusion

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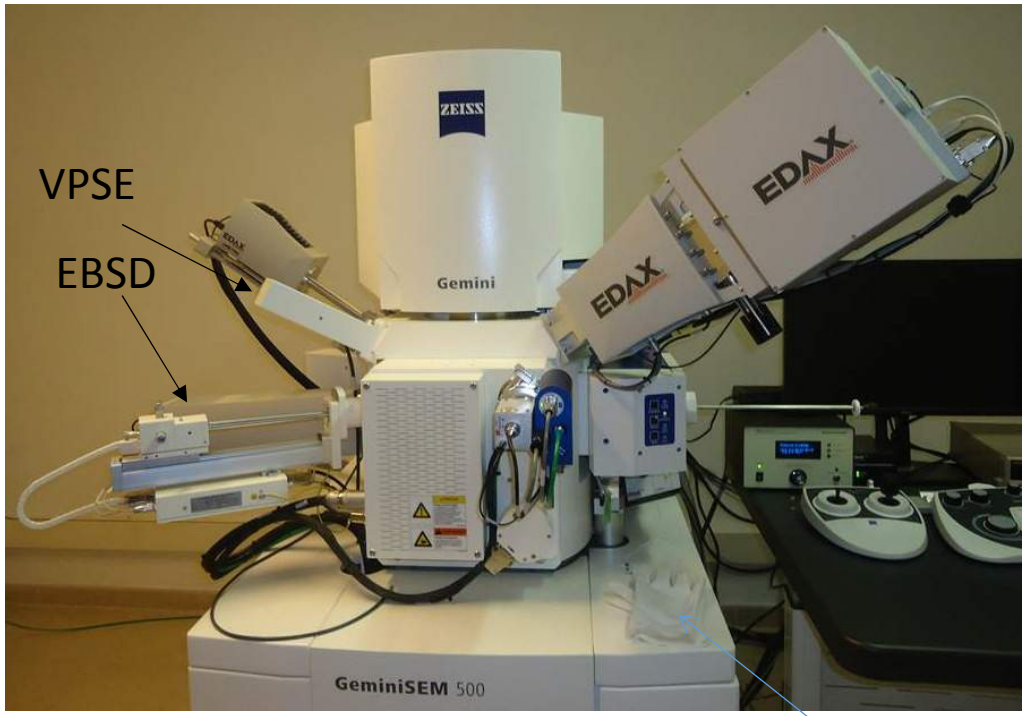
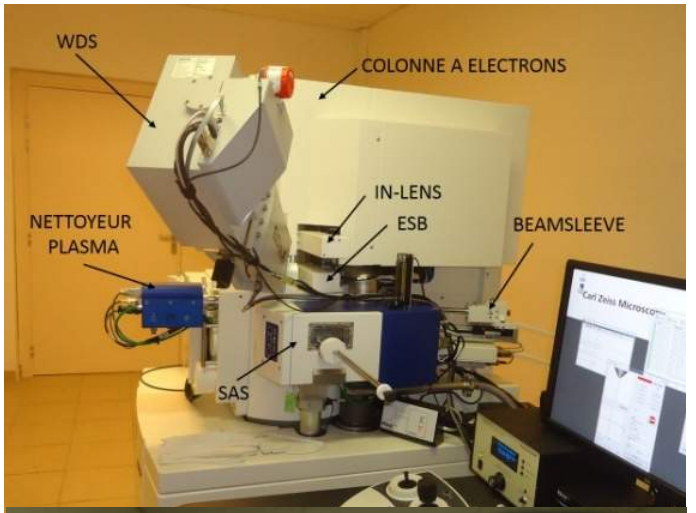
MEB ZEISS Gemini 500 20 nA





Resolution	1.2 nm (500 V) 1.1 nm (1 kV) 0.6 nm (15 kV)	1.8 nm (3kV, 50 Pa VP) 0.9 nm (1kV, Sample Bias)
Beam Current	3 pA (1 kV) - 20 nA (30kV), 2 Gun Modes, 14 Steps	
Variable Pressure	5 – 500 Pa high resolution configuration 5 – 150 Pa large field of view configuration	

1.8 nm à 30 Pa et 3 kV

Notre configuration

EBSD – EDAX HIKARI SUPER

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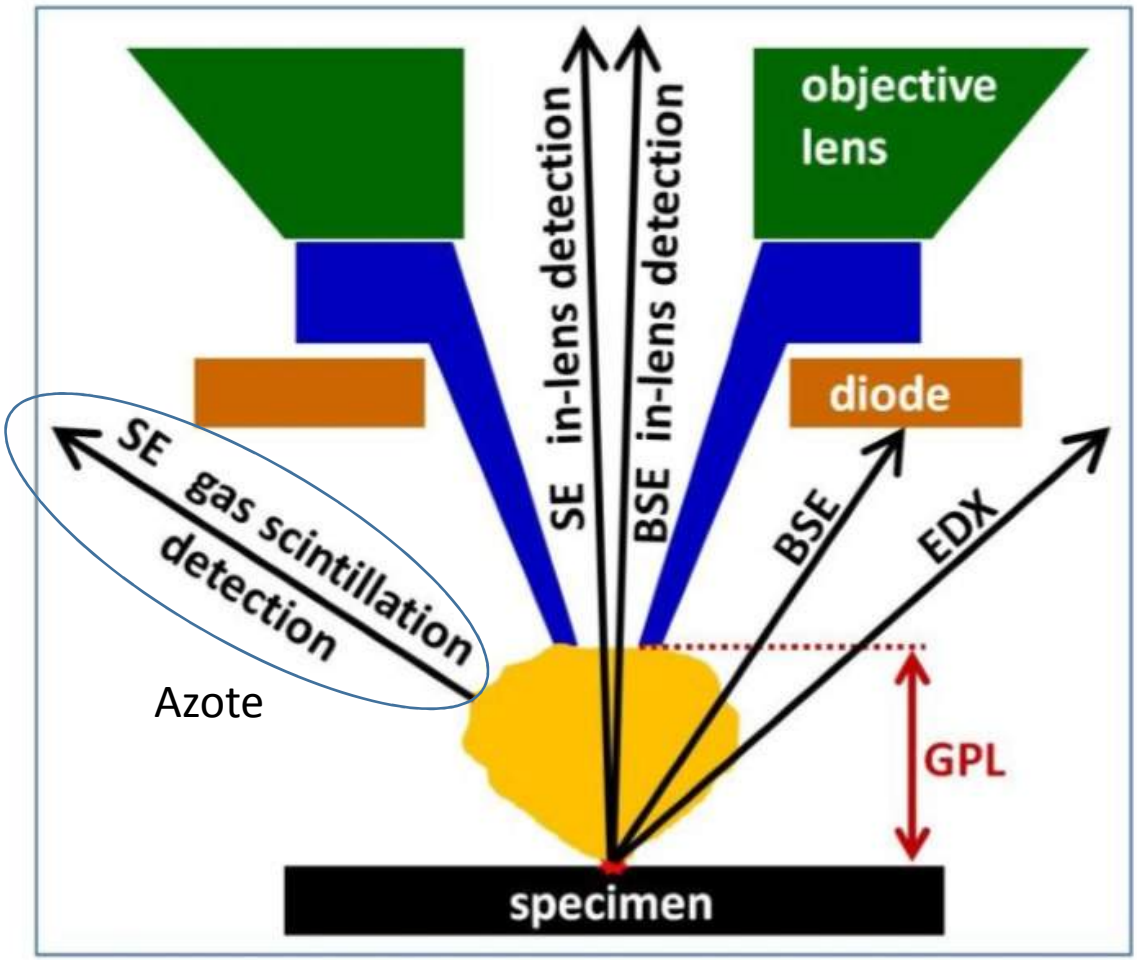
Pression Variable dans le SEM: Une façon simple d'éviter les effets de charge sur l'échantillon

L`ionisation dans le gaz produit un flux positif d'ions qui migrent vers la surface et la neutralisent

Un détecteur de scintillation du gaz peut être utilisé pour l'imagerie SE

DETECTEUR VPSE



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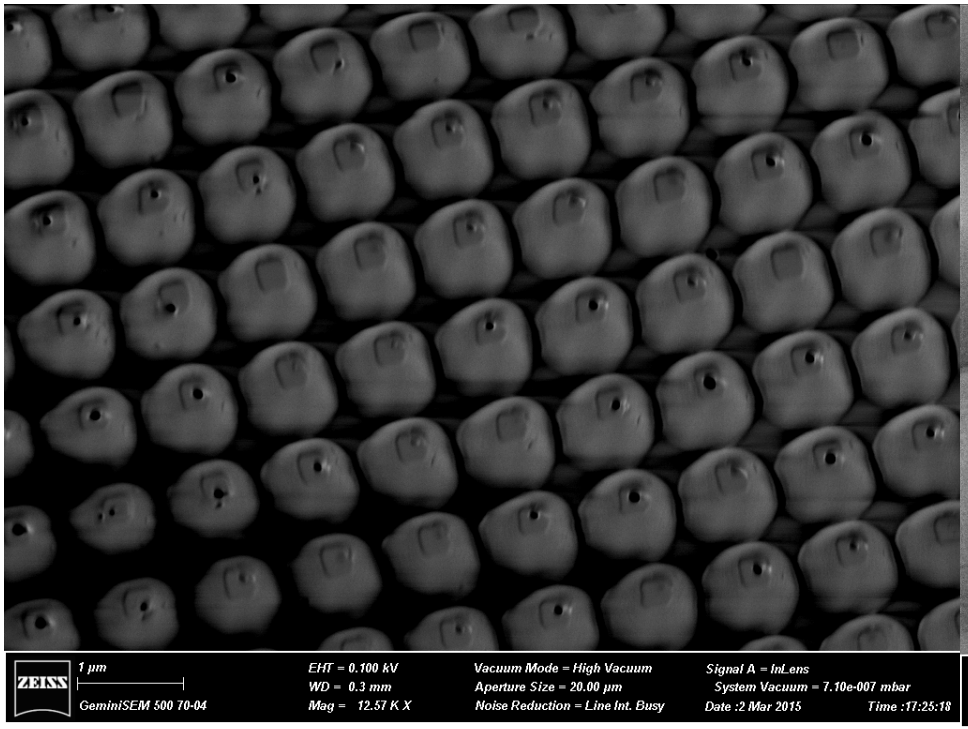
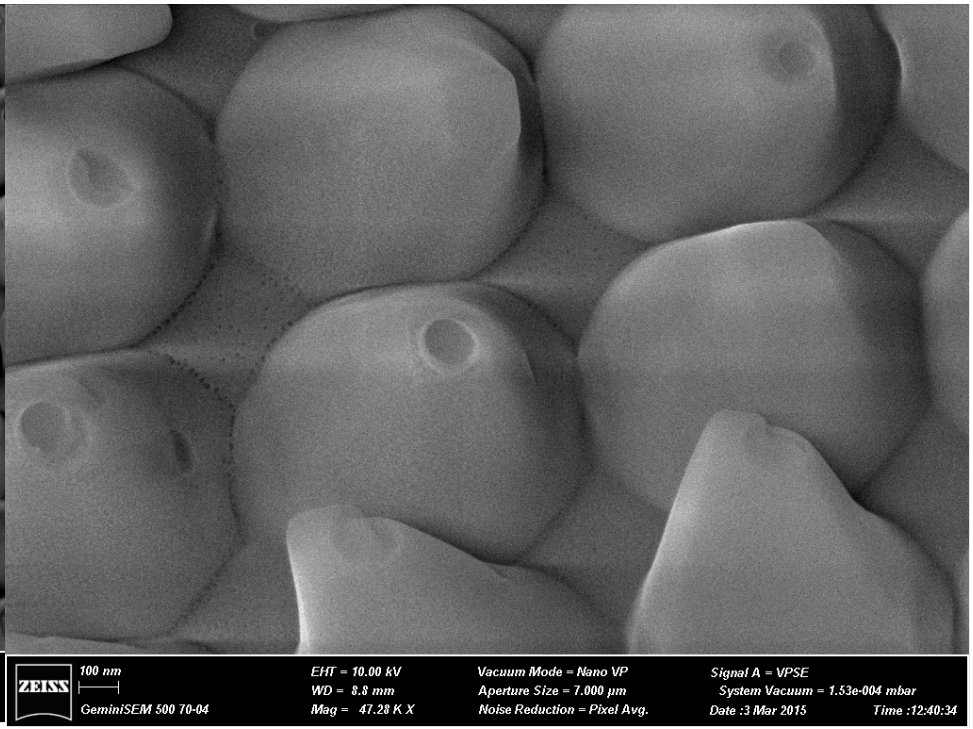
Pour éviter les phénomènes de charges, on peut utiliser la basse tension



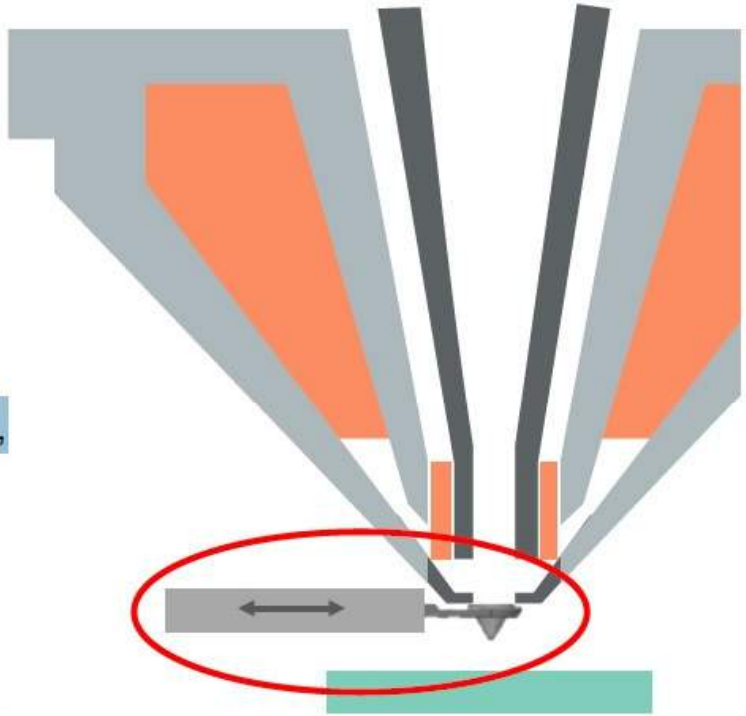


il y a des limites pour la basse tension

Et…pour l'EDS et l'EBSD on a besoin des tensions plus élevées

Different VP modes

- VP mode
  - Works the same as traditional VP mode
  - Pressure ragne 5 – 30 Pa
  - All but InLens/EsB detector
- NanoVP mode
  - EO is the same as high vaccum
  - Beamsleeve inserted
  - Pressure range 5 – 150 Pa for 360 µm aperture, 5 – 40 Pa for 800 µm aperture
  - All detectors
- XVP mode
  - EO is the same as VP
  - Beamsleeve inserted
  - Pressure range 5 – 500Pa for 360 µm aperture, 5 – 150 Pa for 800 µm aperture
  - All but InLens/EsB detector



Nano VP – smaller skirt effect – smaller spot size

Source: Fang, Zeiss

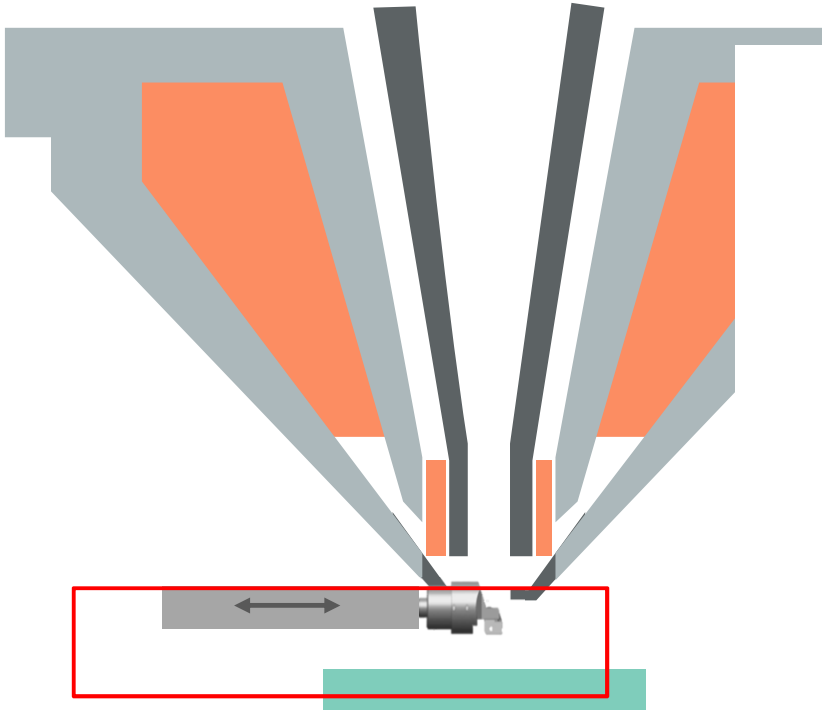
Option

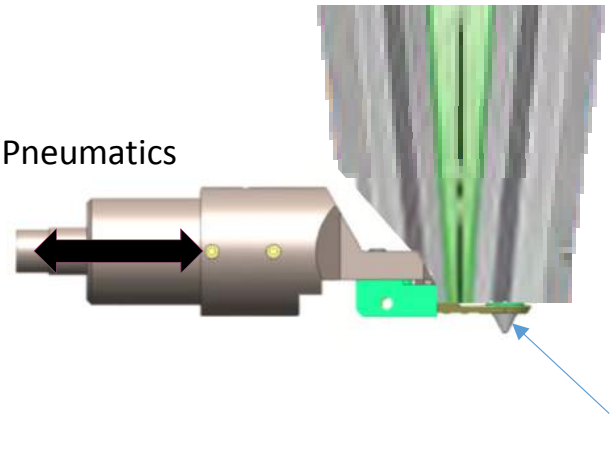
NanoVP – The Technology Behind

Source: Fang, ZEISS

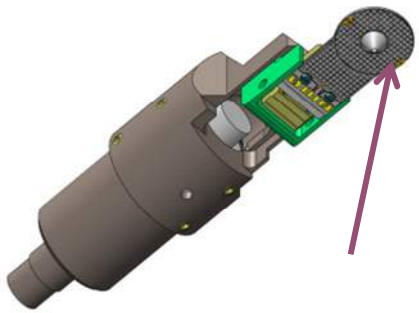
- In NanoVP mode a differential pumping aperture can be inserted below the objective lens.
- This greatly reduces the path length of the incident beam in the gas thus reducing beam broadening.
- Best imaging performance

  up to 150 Pa, full in lens detection, outstanding resolution (1.8 nm at 3kV, 30 Pa), reduced field of view (350µm)
- In order to enable chamber BSD detection in VP mode a one channel BSD detector, which is integrated in the beam sleeve pneumatics, is available.





Detecteurs VPSE + INLENS
+ VP BSD



BSD - 5 segments diode

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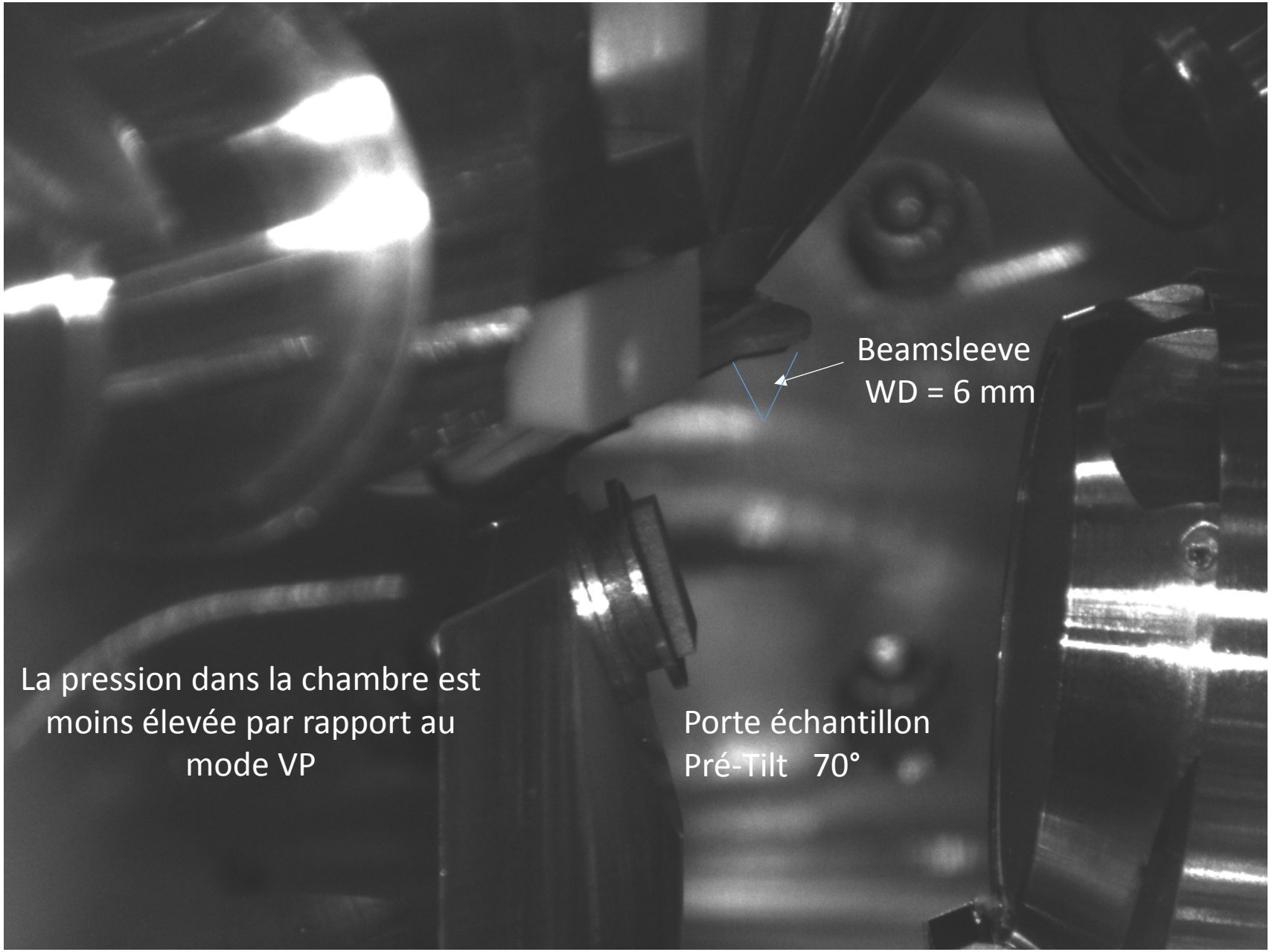
Mode NanoVP Pression entre 5 -150 Pa



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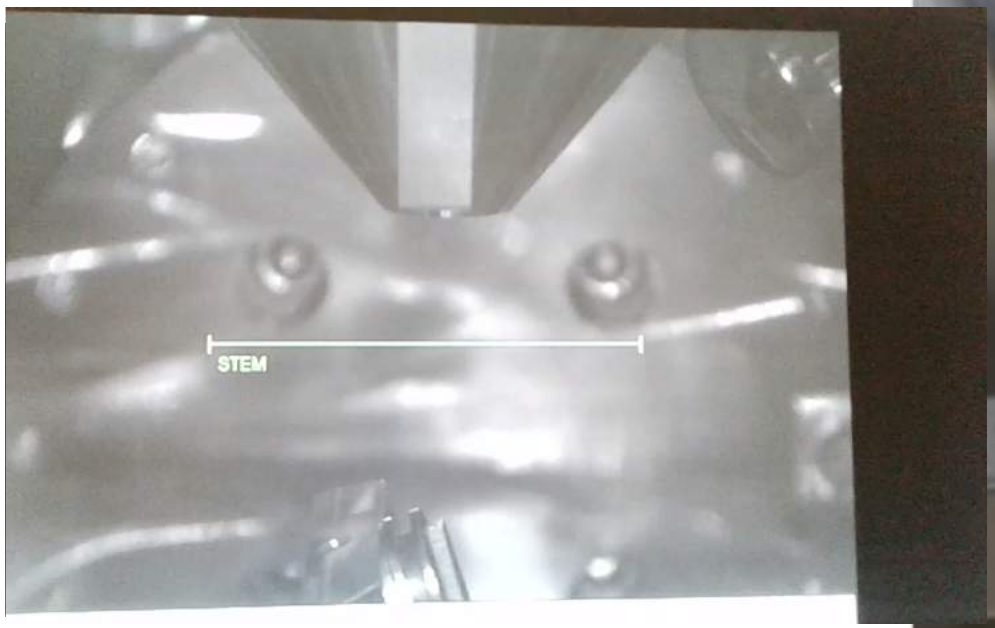

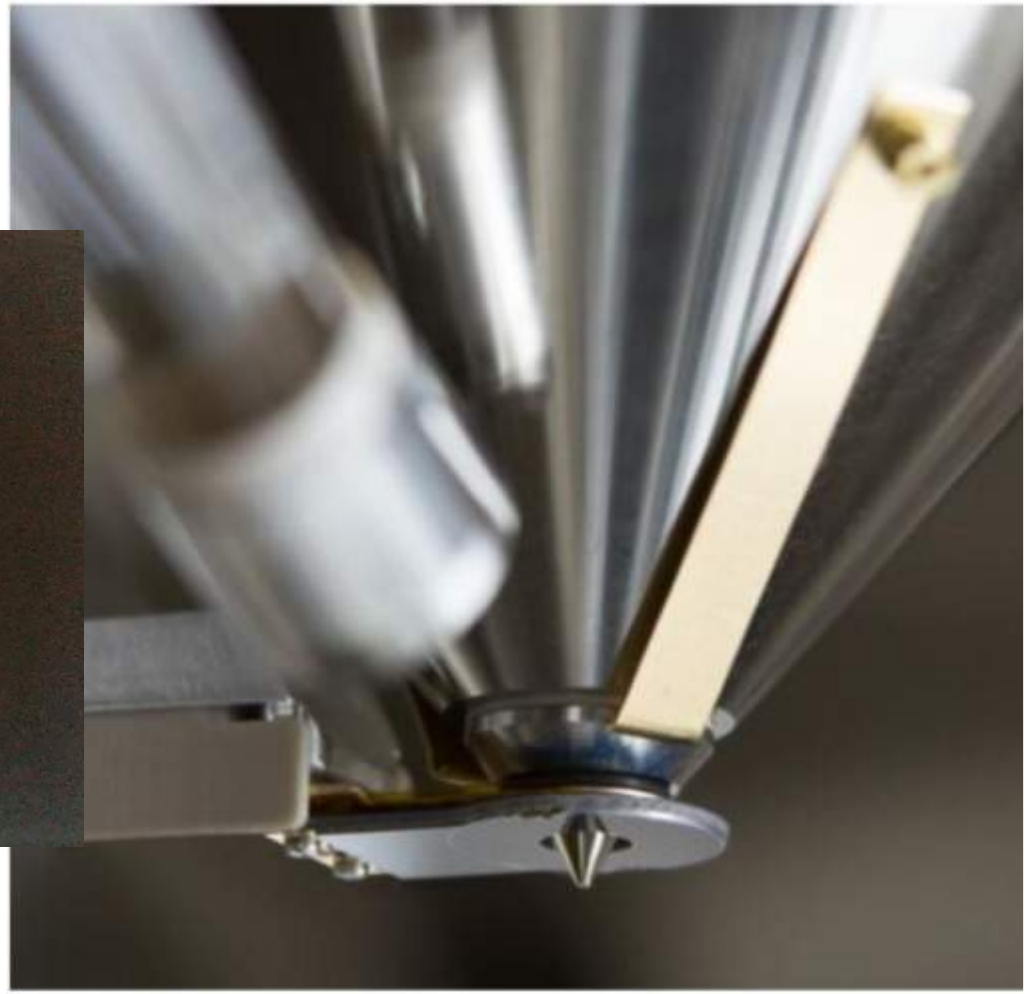

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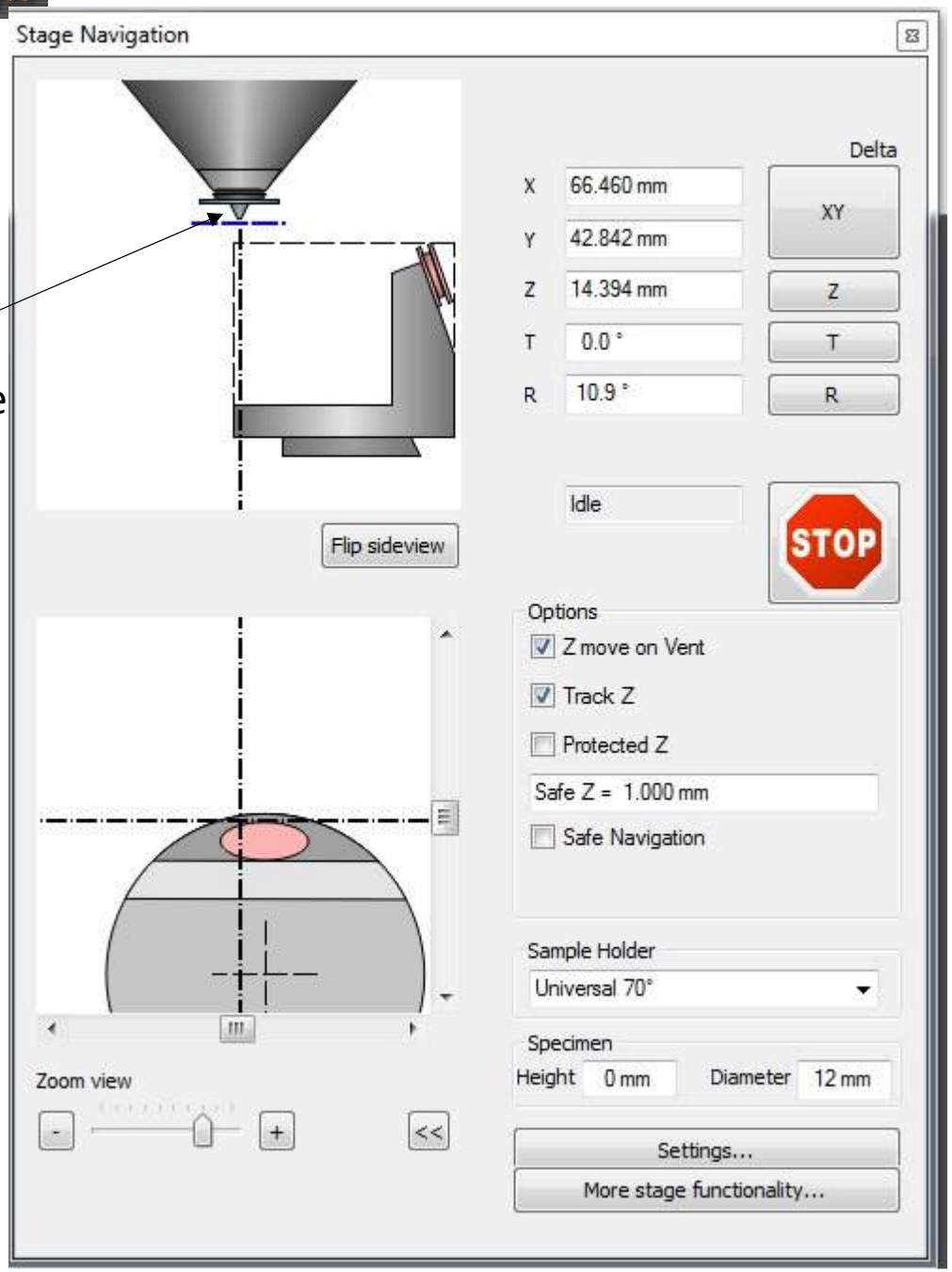
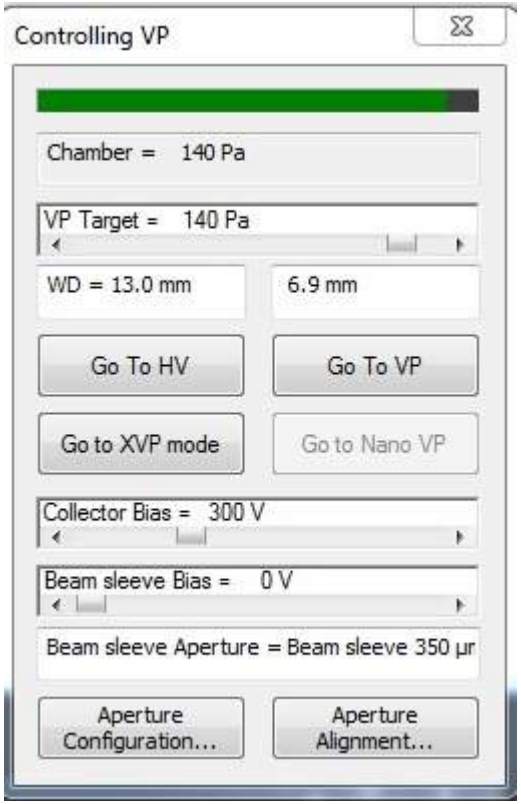
Beamsleeve





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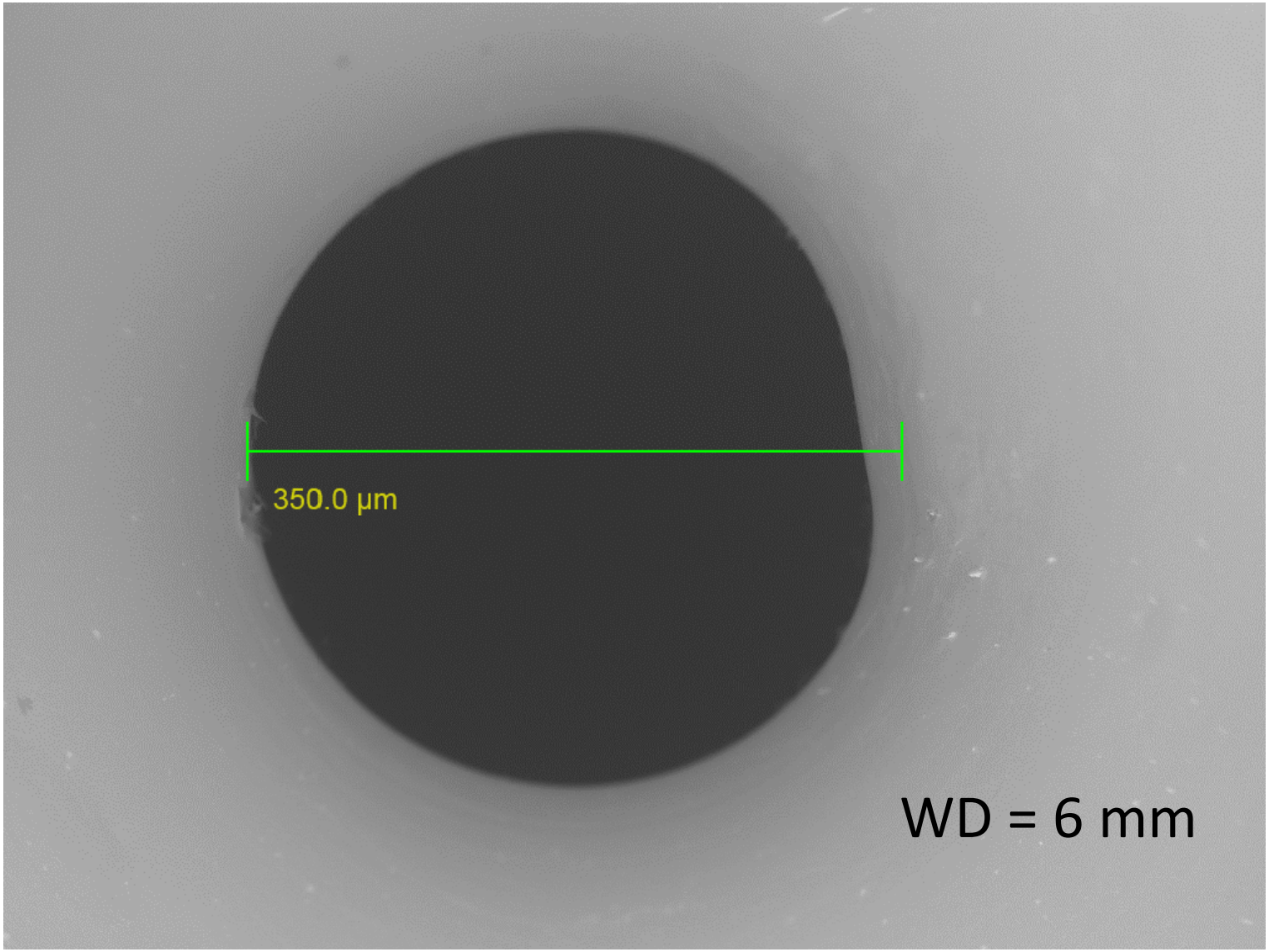
BEAMSLEEVE (CONE)



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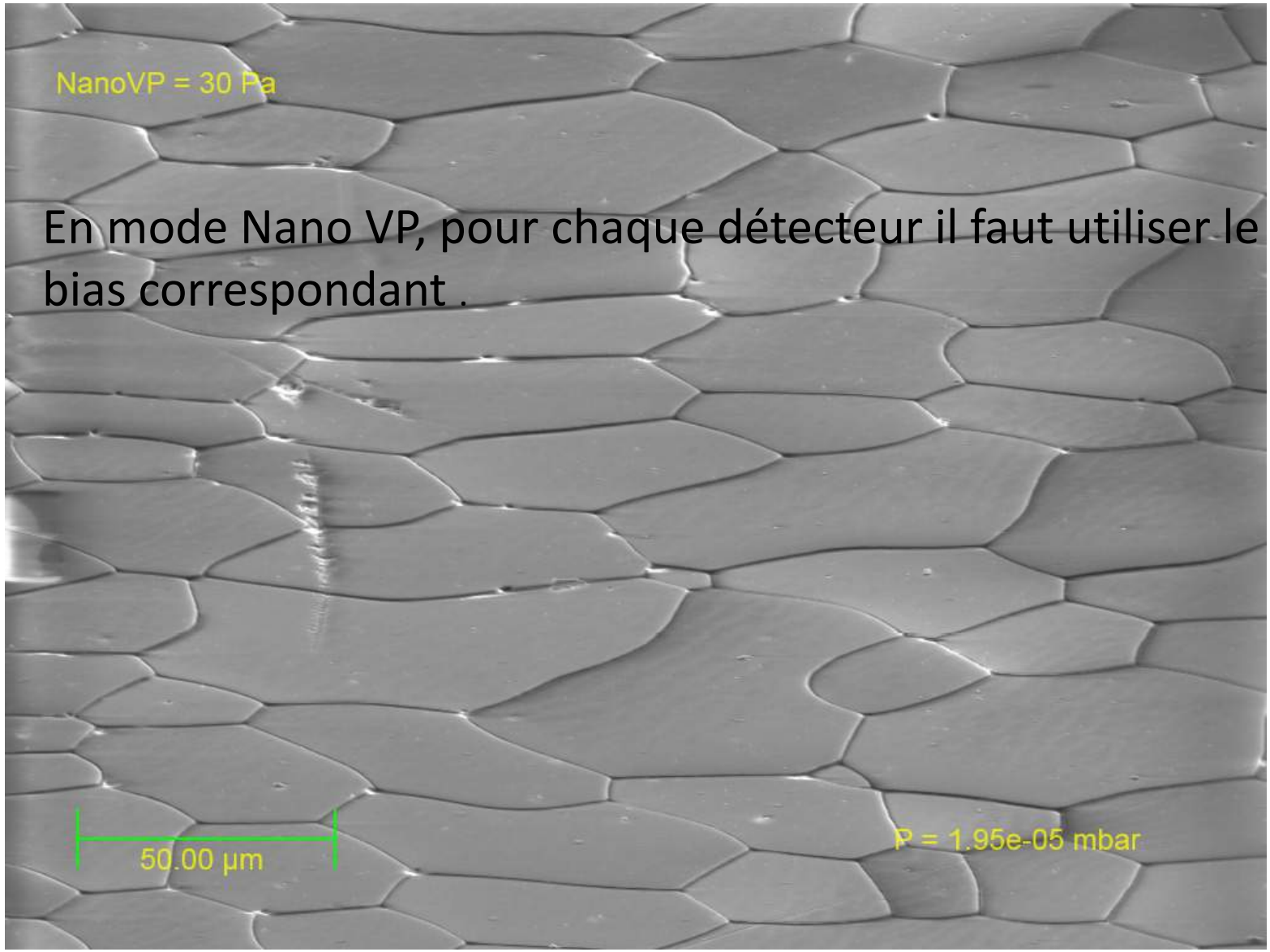
Images en Nano VP 20kV VPSE



En mode Nano VP, pour chaque détecteur il faut utiliser le bias correspondant .

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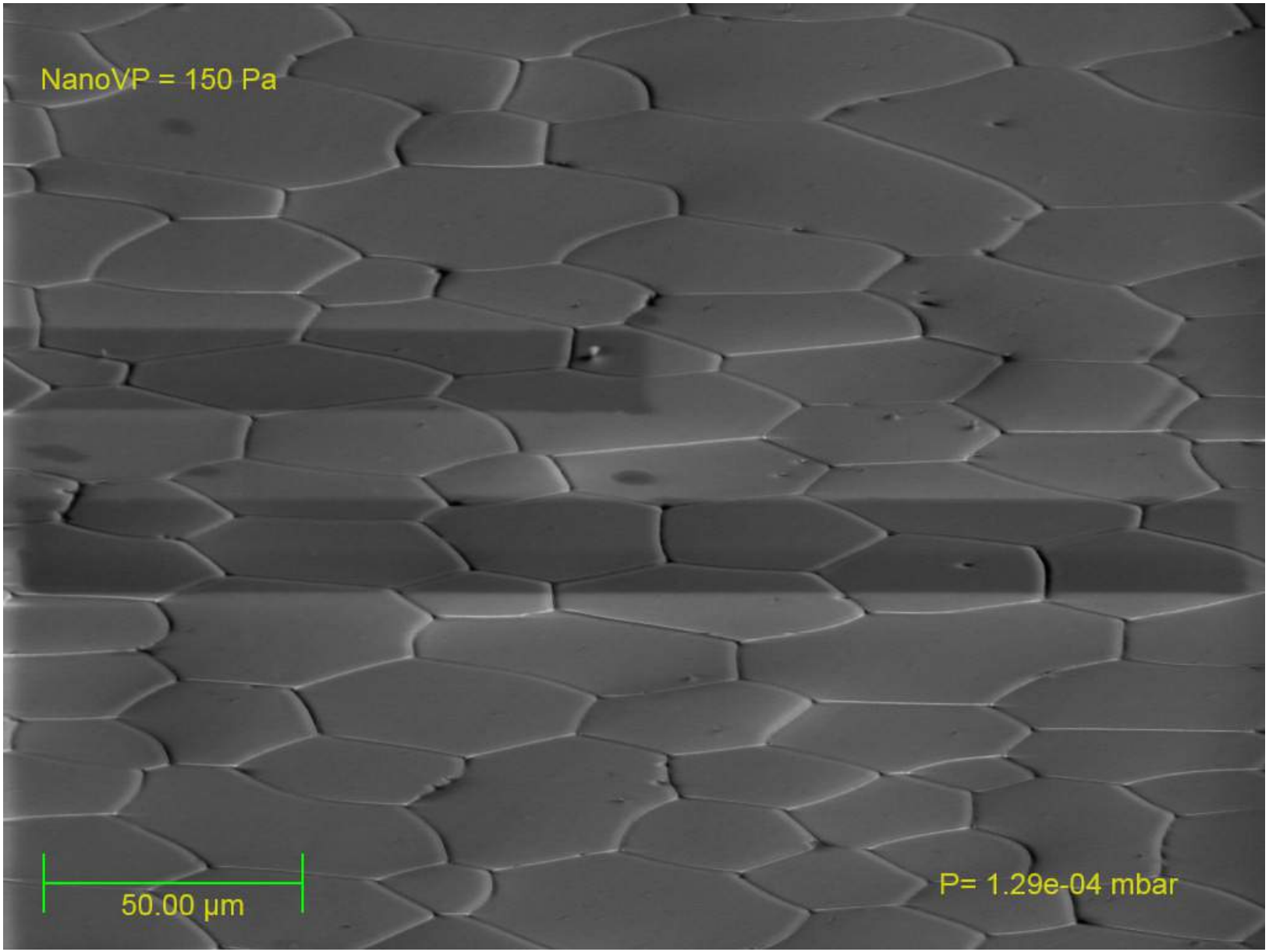
Images en mode Nano VP 20kV VPSE



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Mode VP Pression entre 5 – 30 Pa

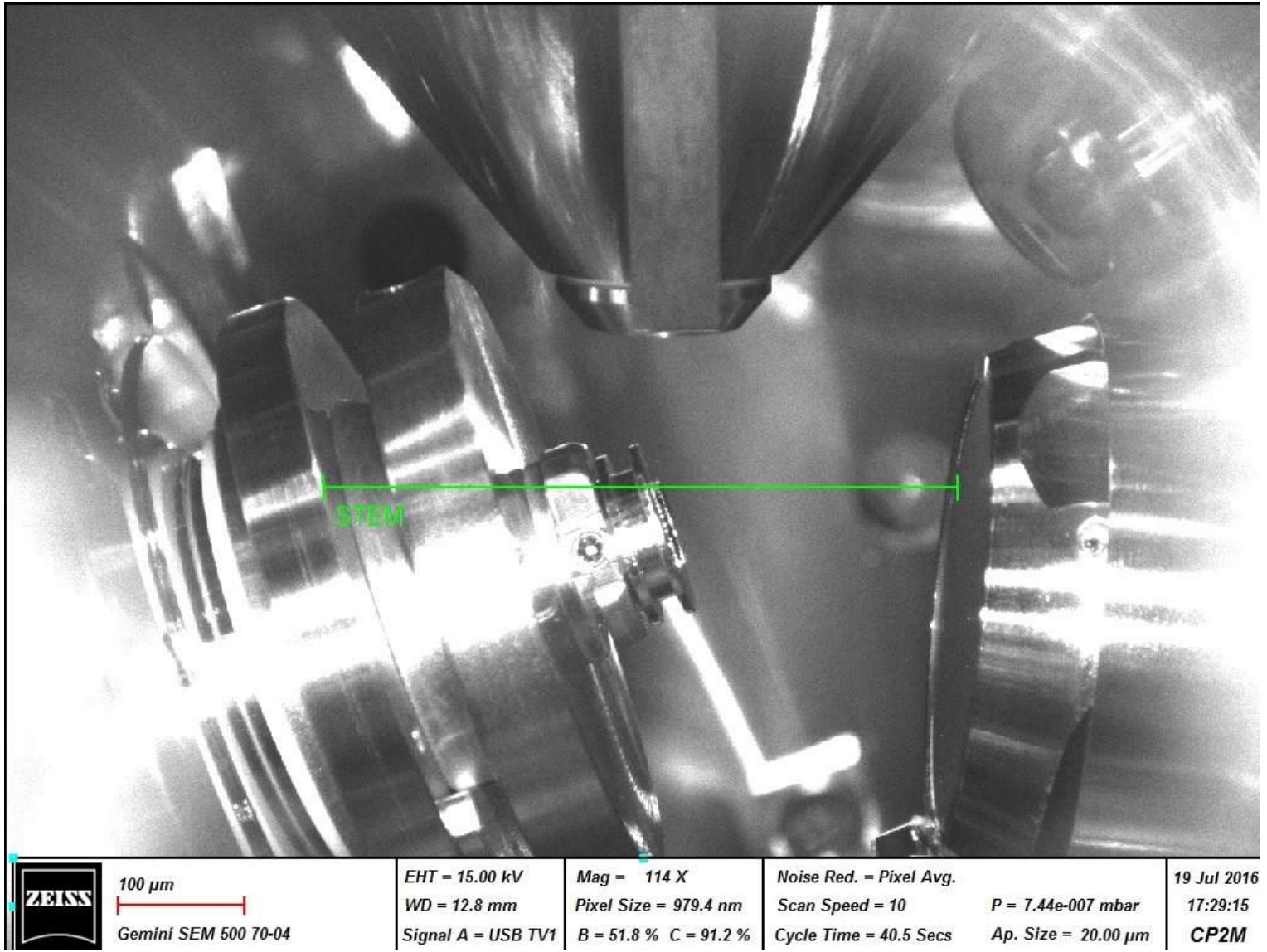

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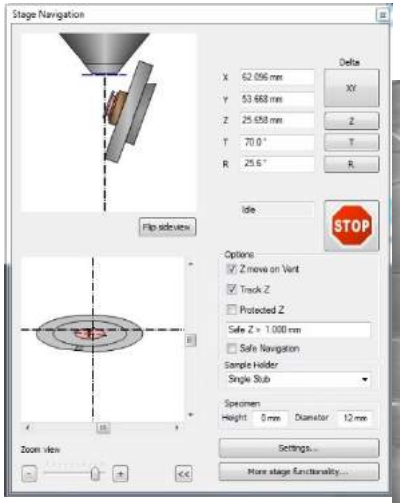
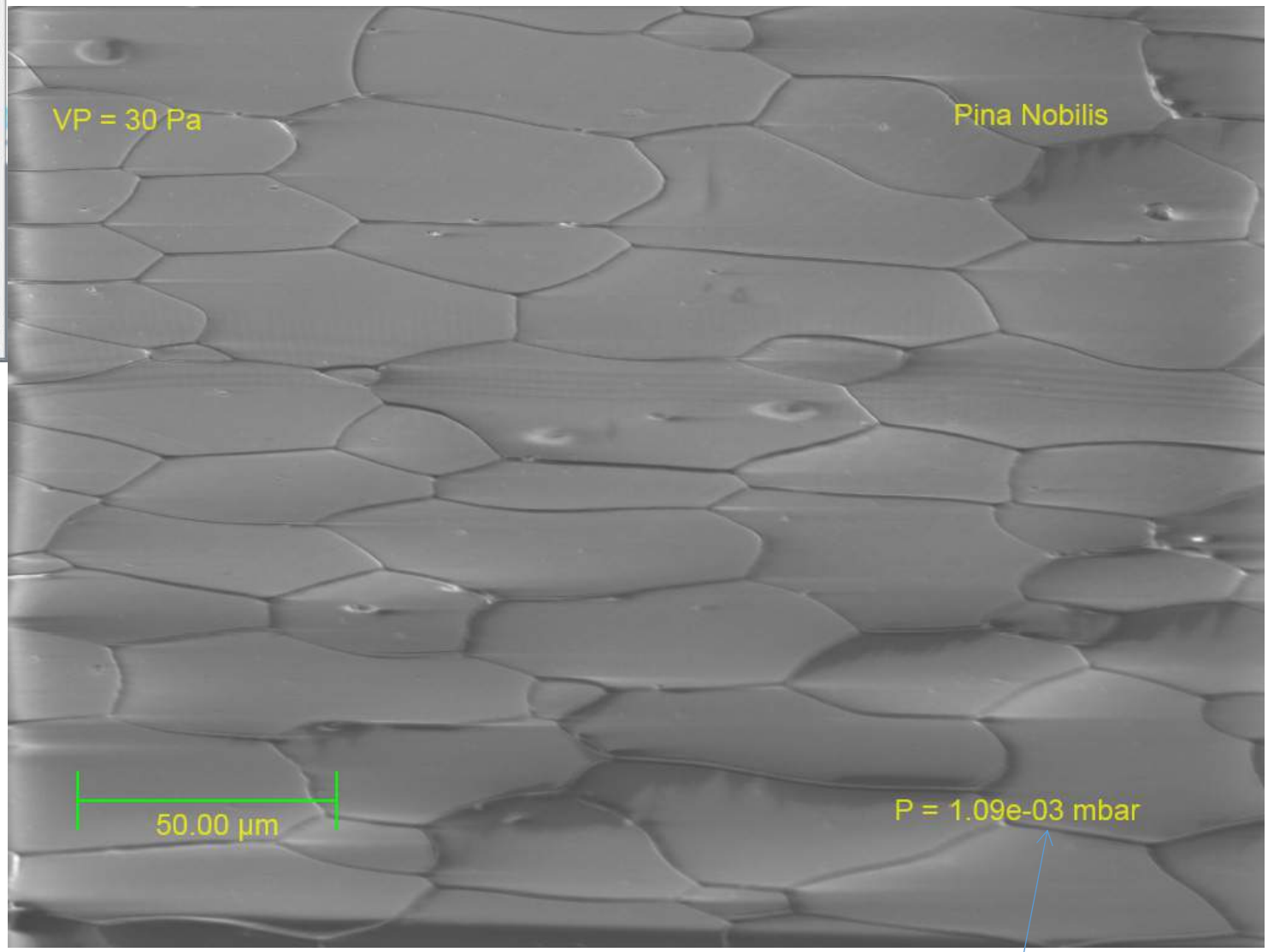
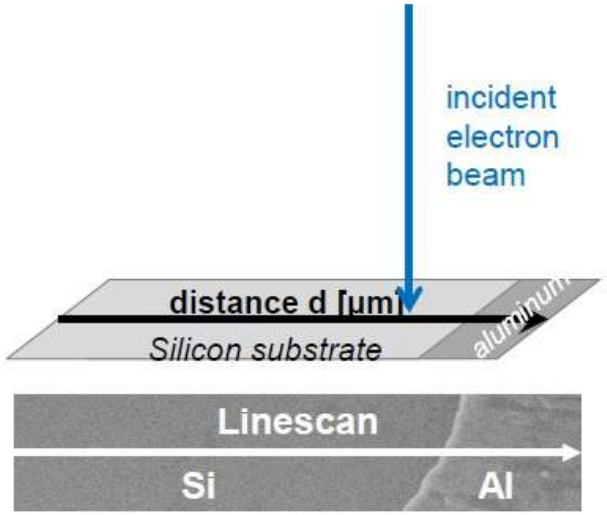
Image en mode VP 20 kV VPSE

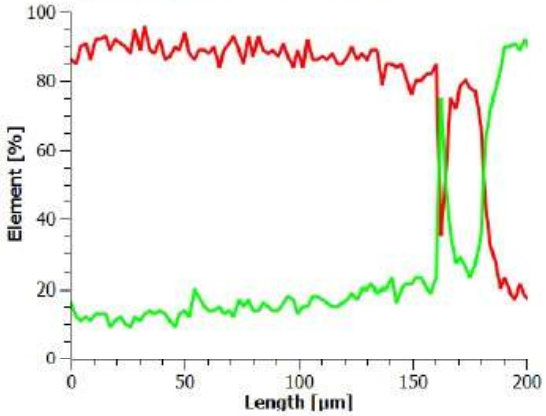




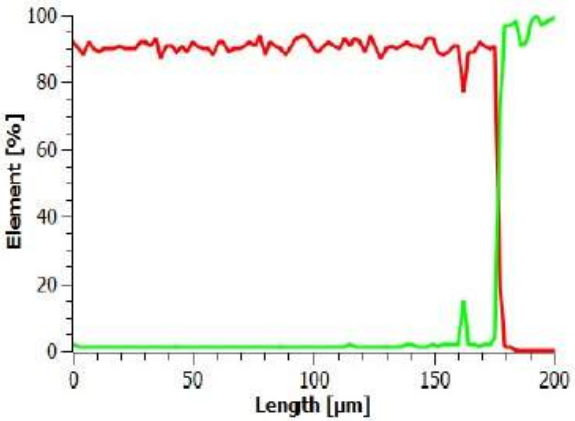
EDS analysis in VP mode



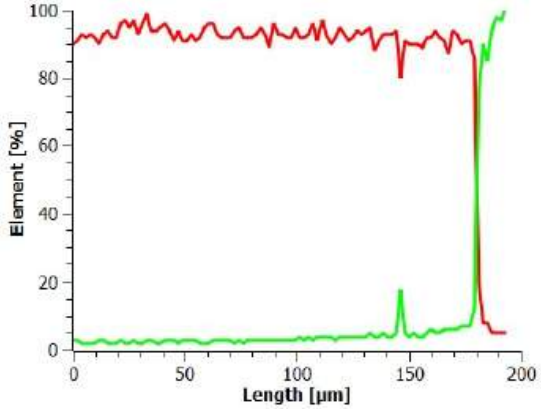
Normal VP 30 Pa



High Vaccum

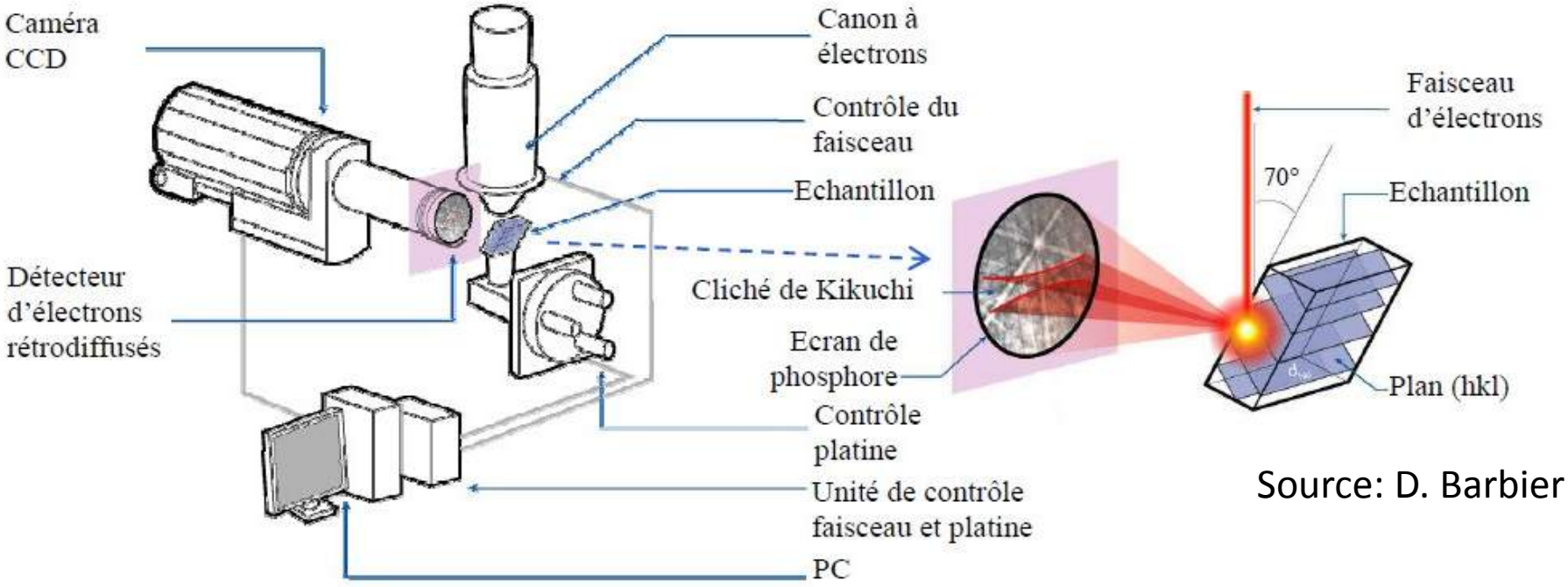


NanoVP 30 Pa



- Normal VP mode does not combine well with EDS analysis. The skirt effect creates a highly delocalized elemental signal.
- By controlling BGPL in NanoVP mode, spreading of EDS signal is largely reduced.
- High resolution EDS is possible on insulate samples with NanoVP.

EBSD: DISPOSITIF EXPERIMENTAL



Source: D. Barbier

L'échantillon cristallin – la surface est parfaitement polie – est incliné de sorte que l'angle formé entre sa surface et le faisceau d'électrons soit égal à 70°.

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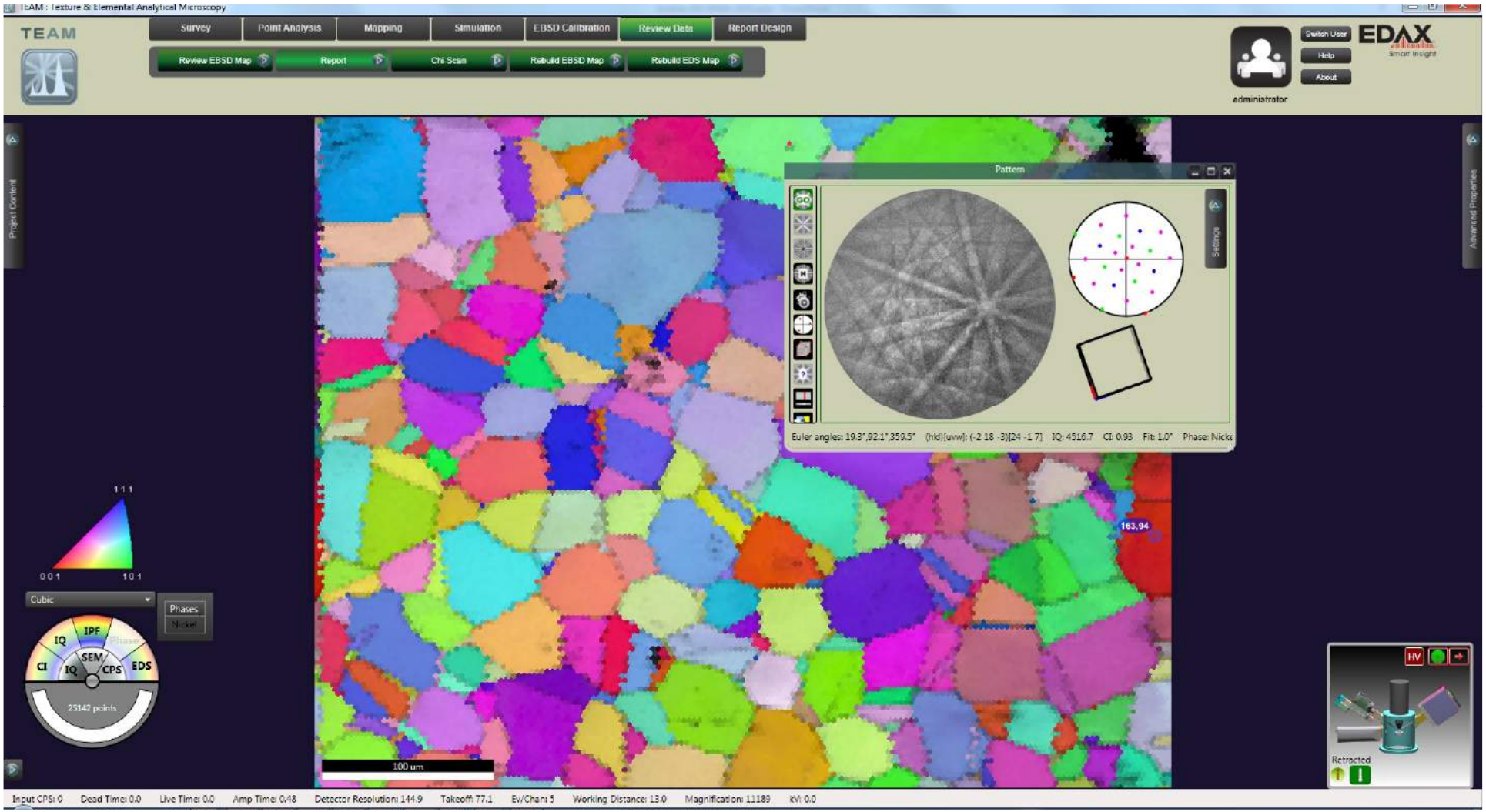
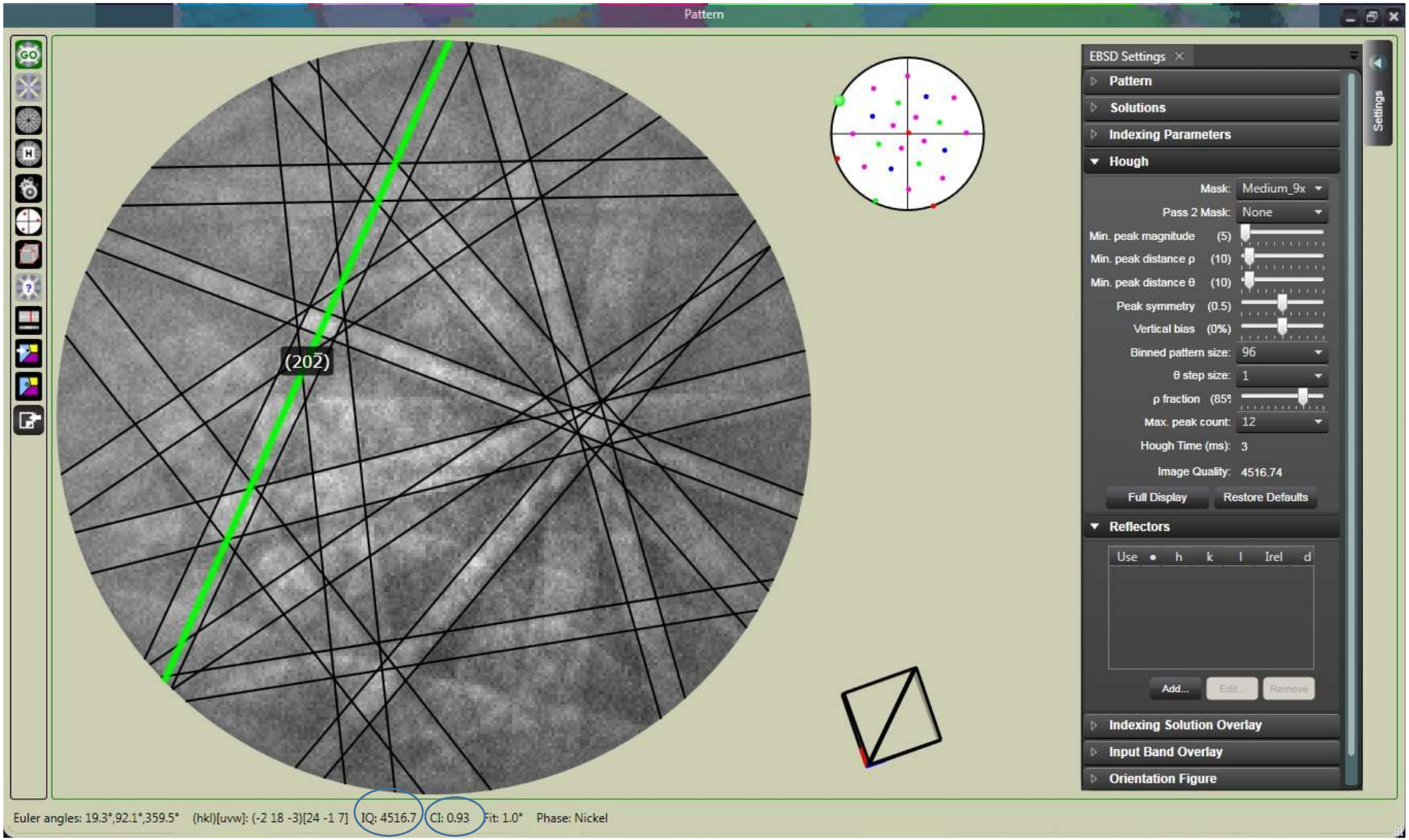
Mode **Cartographie**: Suivant une grille prédéfinie, acquisition et indexation du diagramme de Kikuchi pour chaque point

Analyse de la morphologie et de la taille des grains, caractérisation des joints de grain, analyse de la texture locale et globale





Microstructure: indice de qualité **(IQ)** du diagramme de diffraction
qualité de mesure : indice de confiance de l'indexation **(CI)**, écart entre les bandes détectées et simulées

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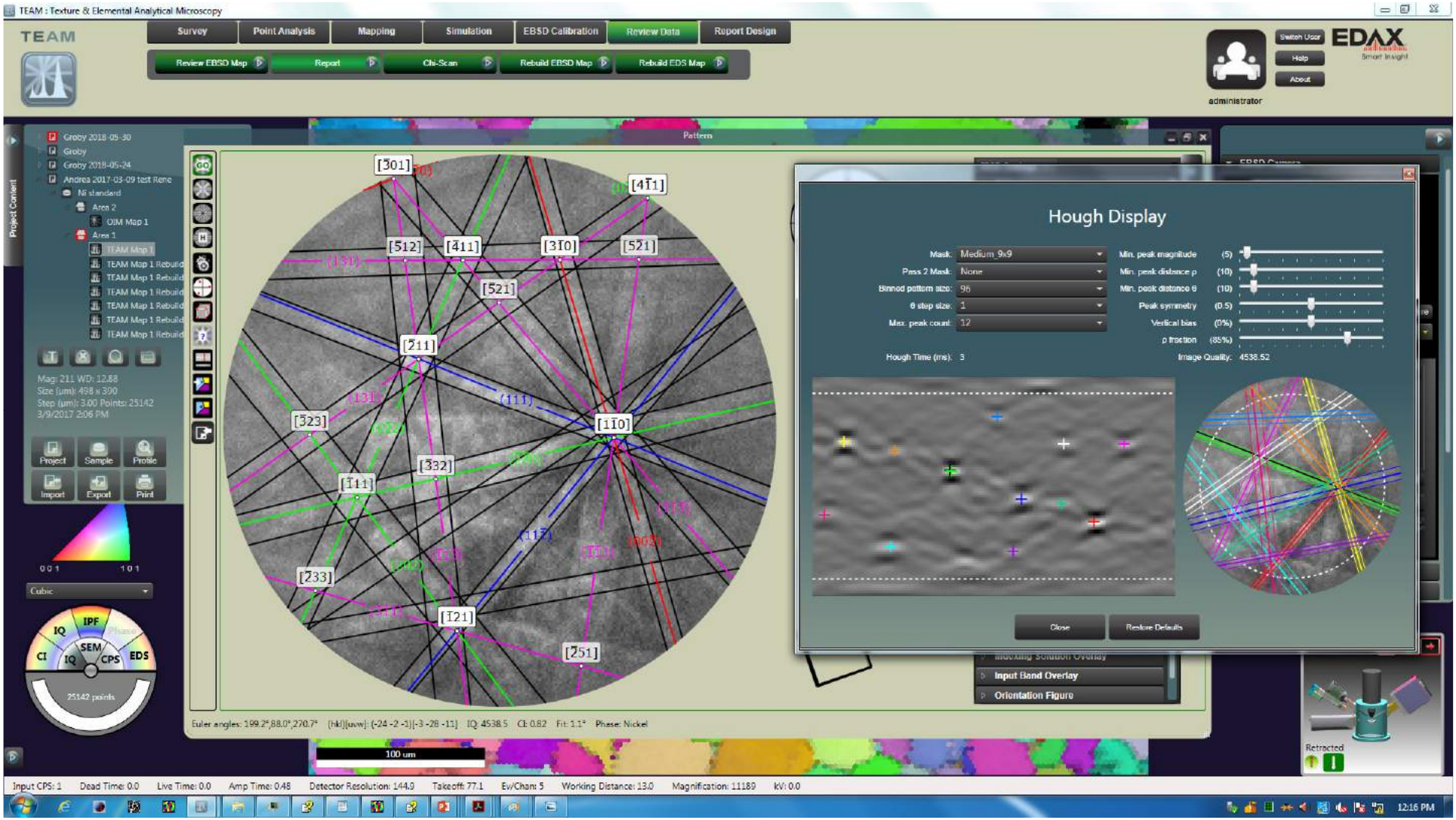
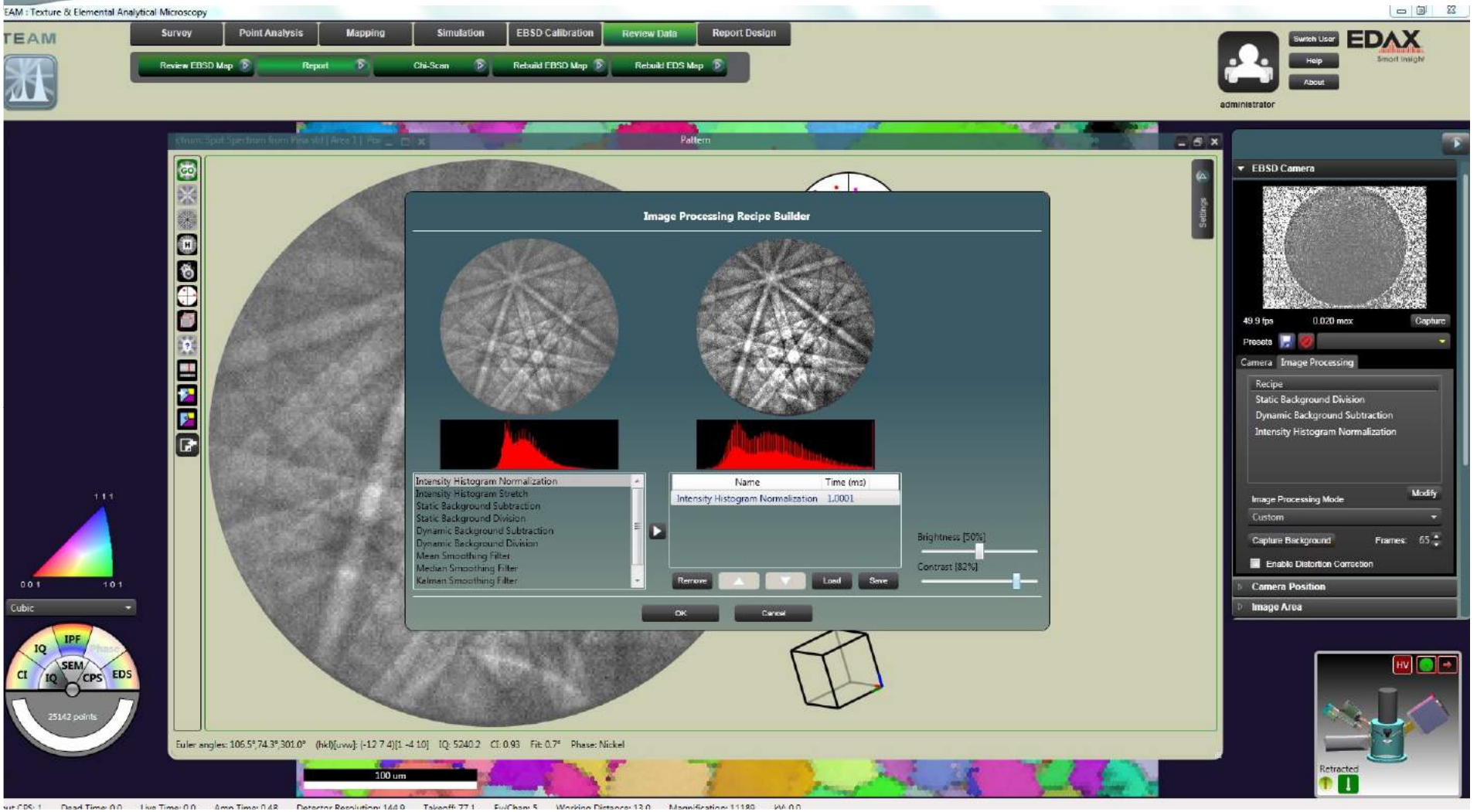


Importance du background



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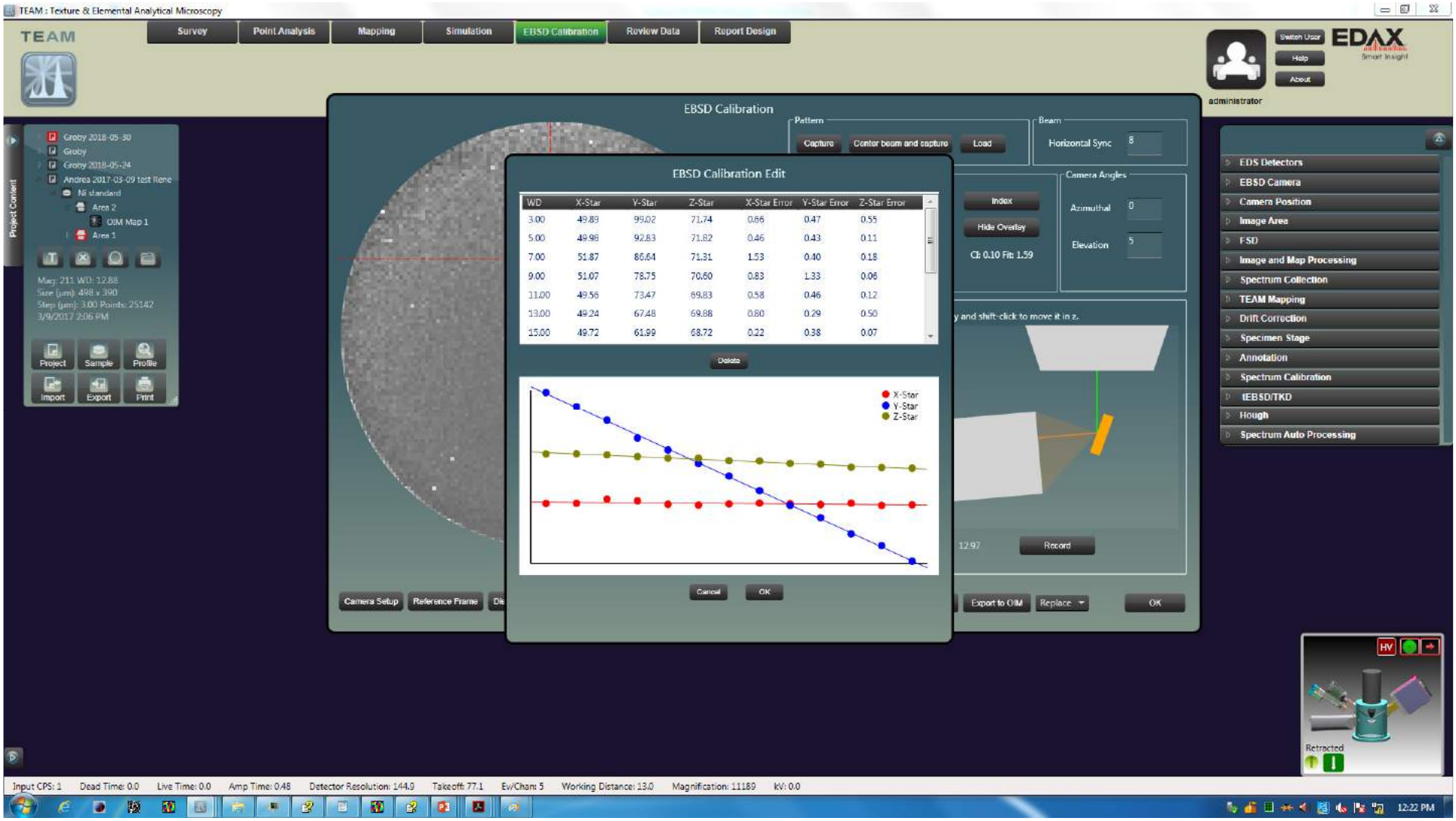
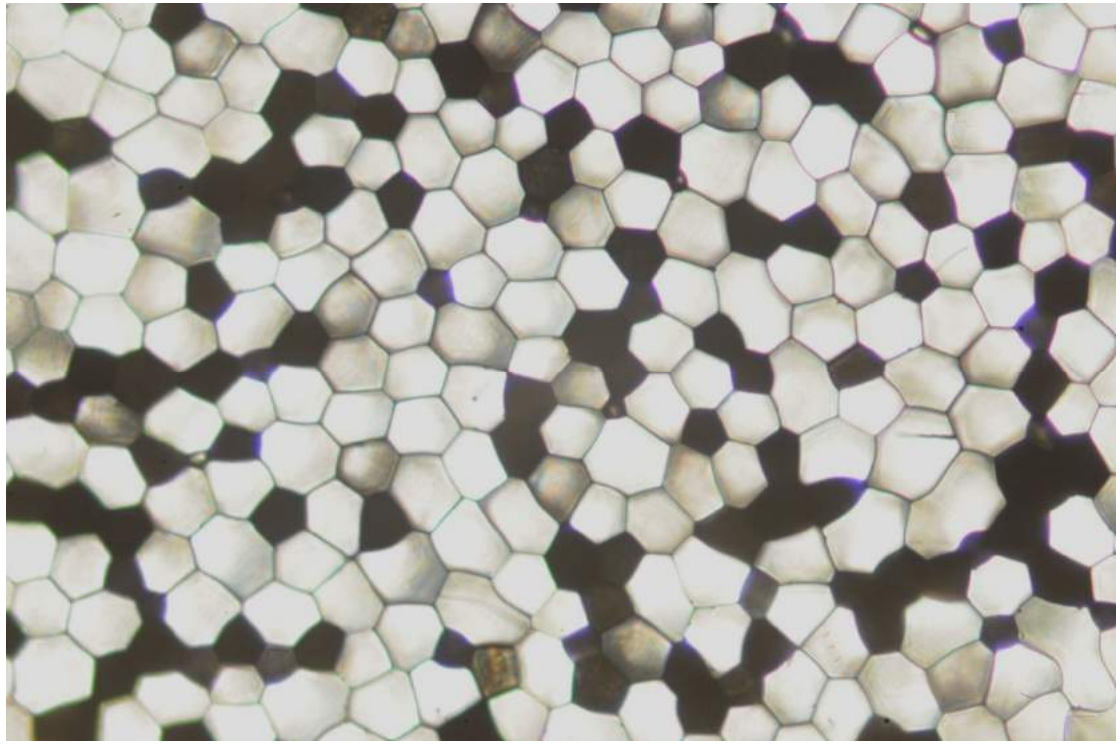
Calibration



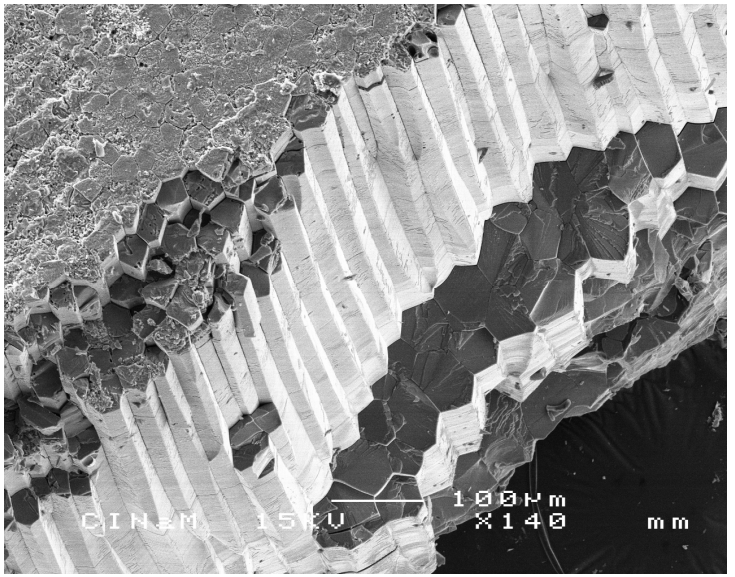
Echantillons – Biomatériaux



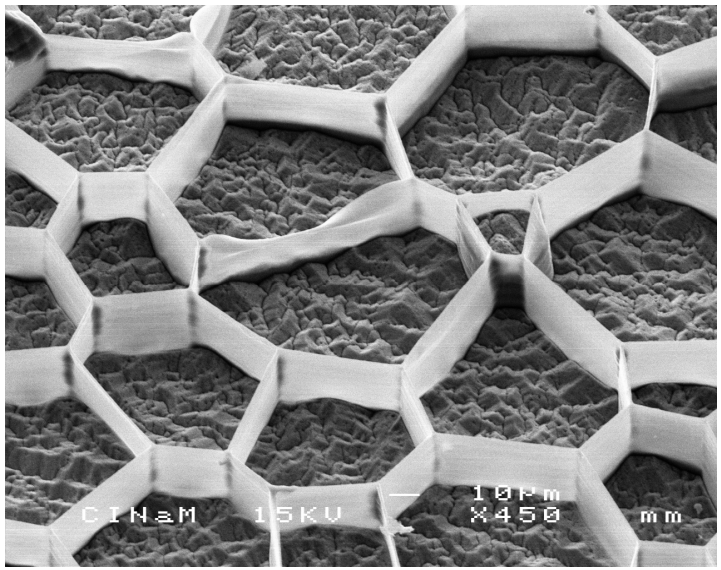
Echantillon 1 - Microscope polarisant

Calcite

Et pour les biomatériaux?



Application des modes VP



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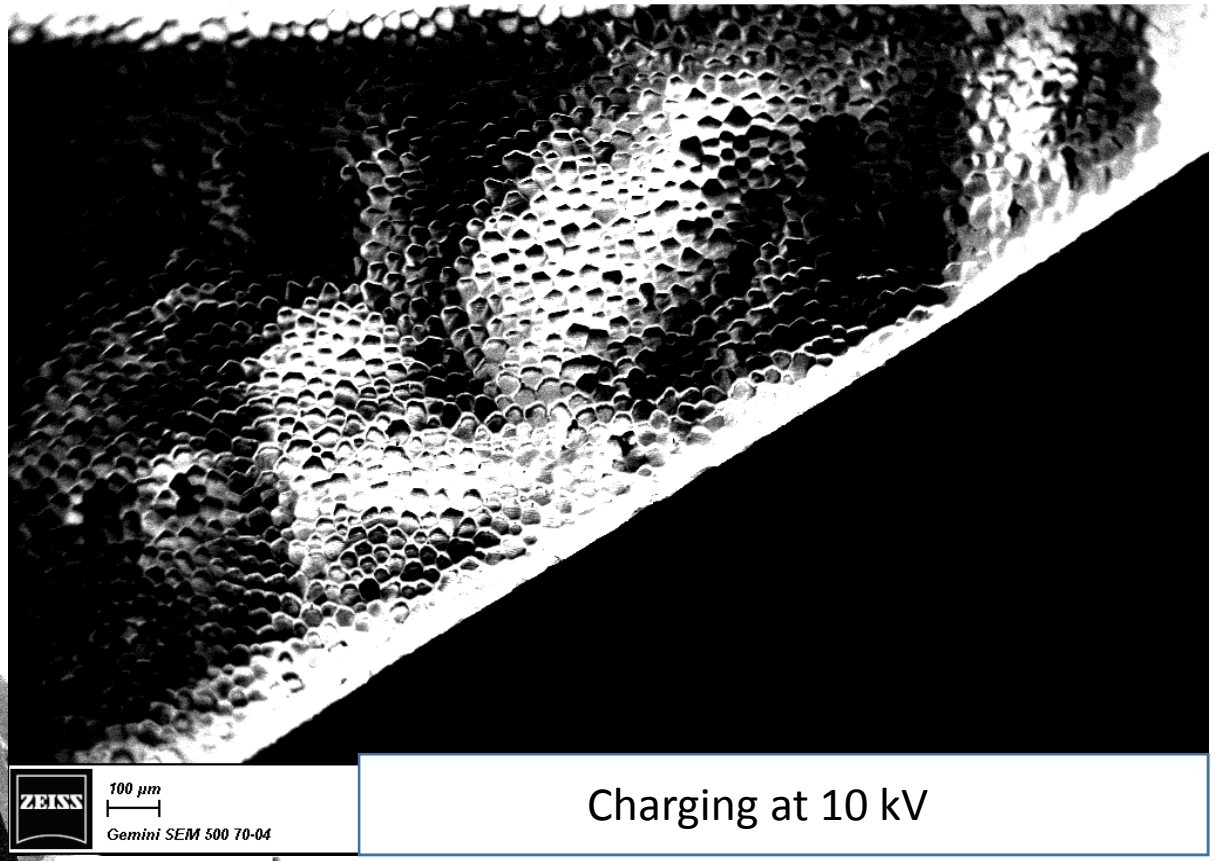
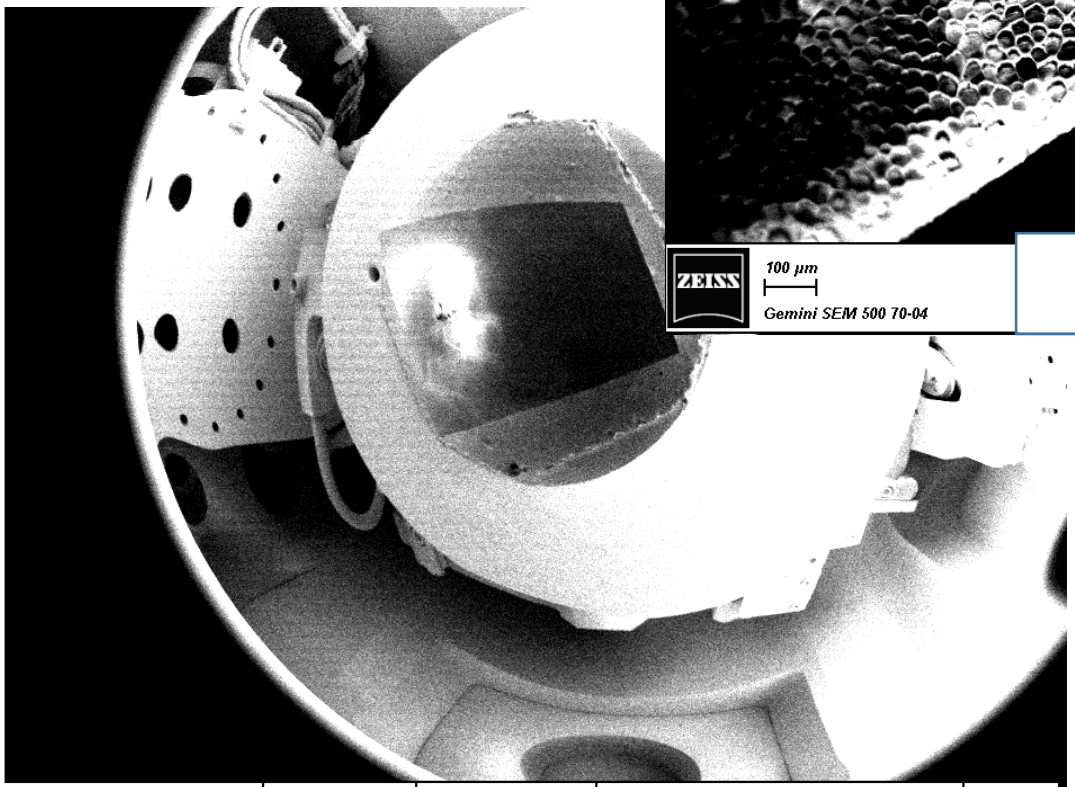


Charging at 10 kV



10 µm?	EHT = 0.100 kV	Mag = 671 X	Noise Red. = Pixel Avg.	10 Jun 2016
Gemini SEM 500 70-04	WD = 0.0 mm	Pixel Size = 166.5 nm	Scan Speed = 5 P = 1.52e-005 mbar	10:55:49
	Signal A = HE-SE2	B = 50.3 % C = 27.6 %	Cycle Time = 1.4 Secs Ap. Size = 20.00 µm	CP2M

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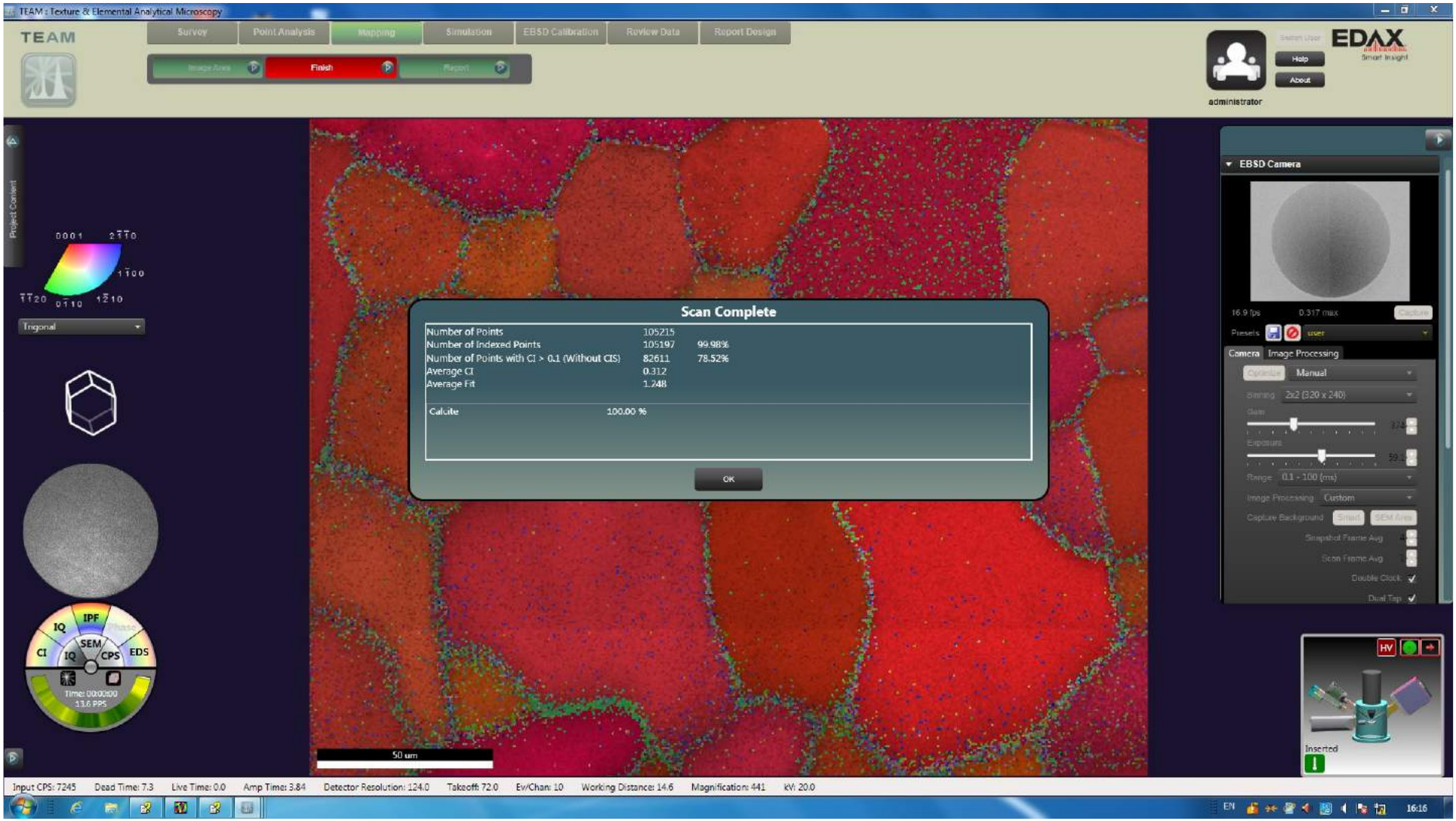
Mode VP – 1er essai sans beamsleeve (cône) 30 Pa



Background: Static Background Subtraction – Intensity Histogram Normalization

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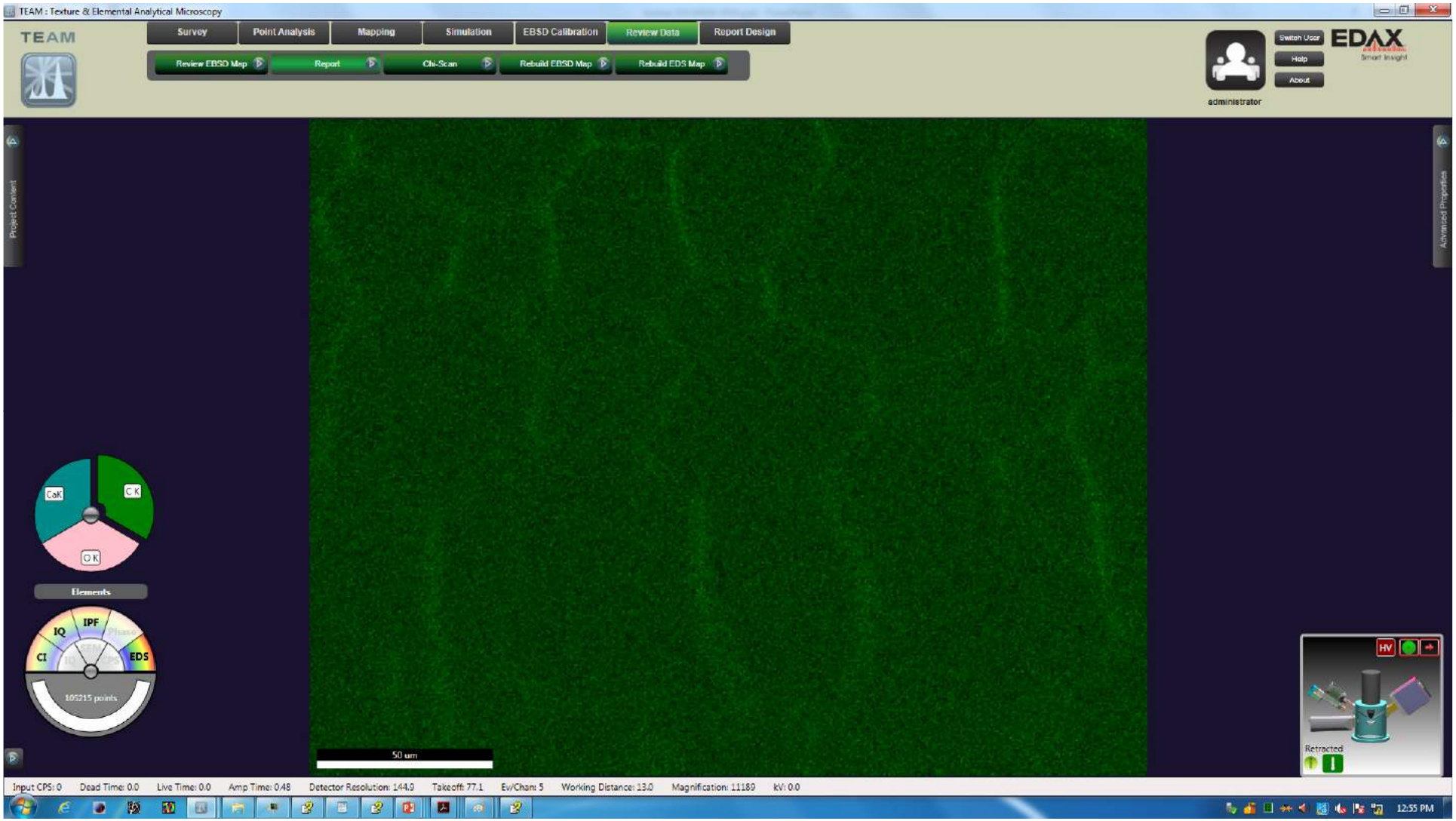
Mode VP – 1er essai sans beamsleeve (cône) 30 Pa



EDS pour localiser la matière organique

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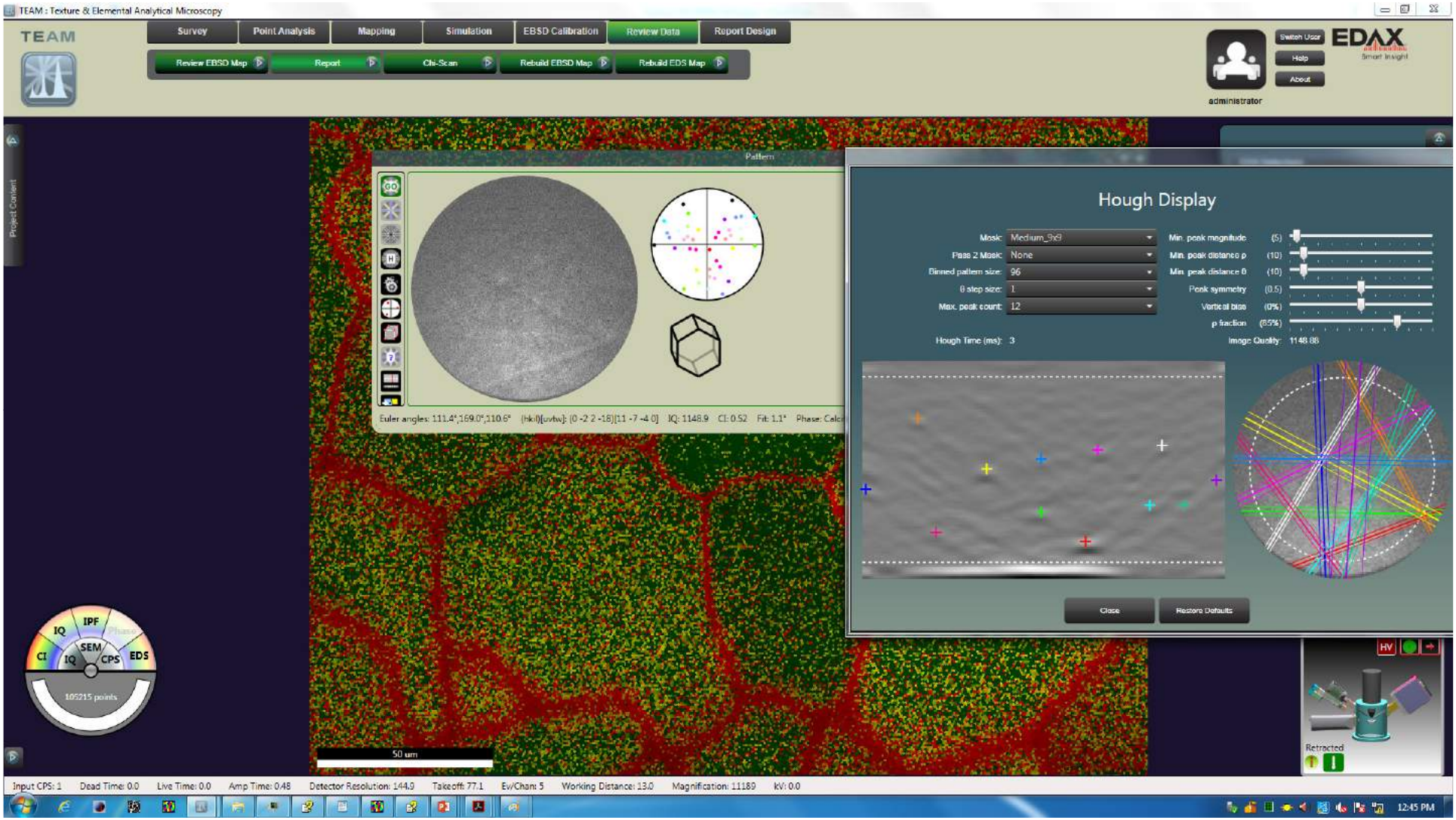
Mode VP – 1er essai sans beamsleeve (cône) 30 Pa



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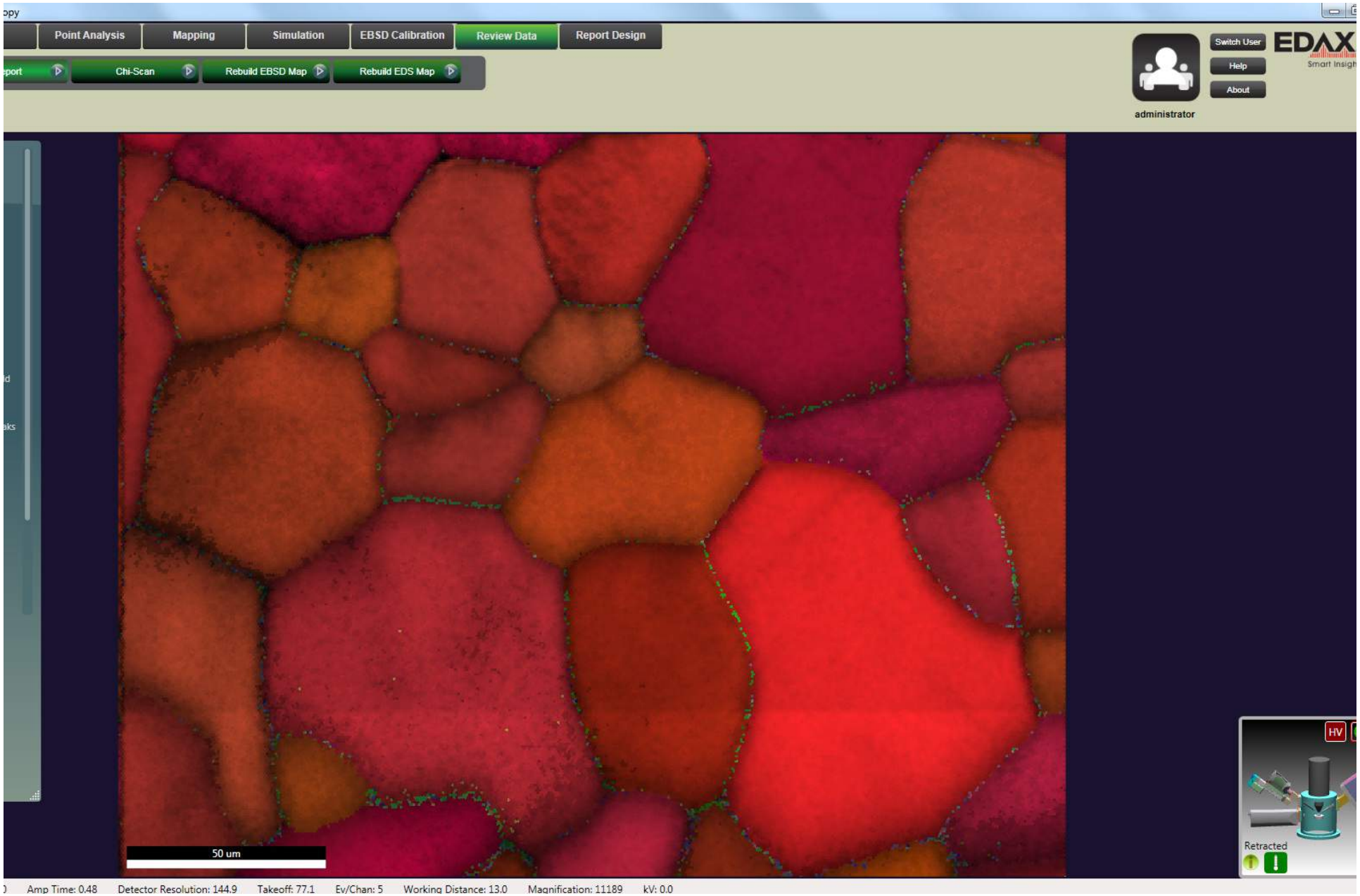
Mode VP – 1er essai sans beamsleeve (cône) 30 Pa

Application de NPAR - Neighbor Pattern Averaging and Re-indexing



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NPAR - Neighbor Pattern Averaging and Re-indexing

TEAM Help V4.5

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What's new in TEAM V4.3

Jackson[a]
[a]EDAX, 392 East 12300 South, Suite H, Draper UT 84020, USA
[b]EDAX, 91 McKee Drive, Mahwah NJ 07430, USA
[c]Carnegie Mellon University, Department of Material Science and Engineering, 5000 Forbes Avenue, Pittsburgh, PA 15213, USA
[d]BlueQuartz Software, 400 S. Pioneer Blvd, Springboro , OH 45066, USA

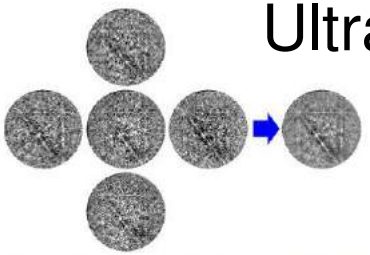
For each point in a scan, the corresponding pattern is averaged with the patterns of the four neighboring scan points as shown schematically in Fig. 1.

$$\overline{I_{ij}^{x,y}} = \frac{1}{5}\left(I_{ij}^{x,y} + I_{ij}^{x-\Delta x,y} + I_{ij}^{x+\Delta x,y} + I_{ij}^{x,y-\Delta y} + I_{ij}^{x,y+\Delta y}\right)$$

(Eq 1)

$I_{ij}^{x,y}$ is the intensity in the pattern at pixel coordinate i,j for the point in the scan with coordinates.

x, y . Δx and Δy denote the step sizes between neighboring points in the grid in the horizontal and vertical directions (they are equal in all the cases shown here).



Ultramicroscopy159(2015)81–94

Fig. 1. Schematic of the averaging of the pattern at a given point with the patterns of the neighboring points.

If the data is collected on a hexagonal grid then the averaging kernel contains the pattern of the point of interest and the patterns of the six nearest neighbors. The orientation maps shown in subsequent sections are sometimes referred to as Inverse Pole Figure or IPF maps. In these maps, the color designates the crystal direction parallel to the sample normal according to the color keys shown in Fig. 2.

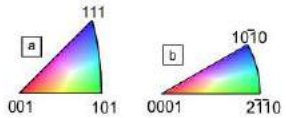


Fig. 2. Orientation map color keys for the (a) nickel alloy and steel samples and (b) the deformed magnesium sample.

Fig. 3 shows results for the duplex steel sample with artificial noise applied to the patterns after which the noisy patterns are re-scanned using both the conventional and NPAR approaches. The NPAR approach clearly provides a

5:17 PM

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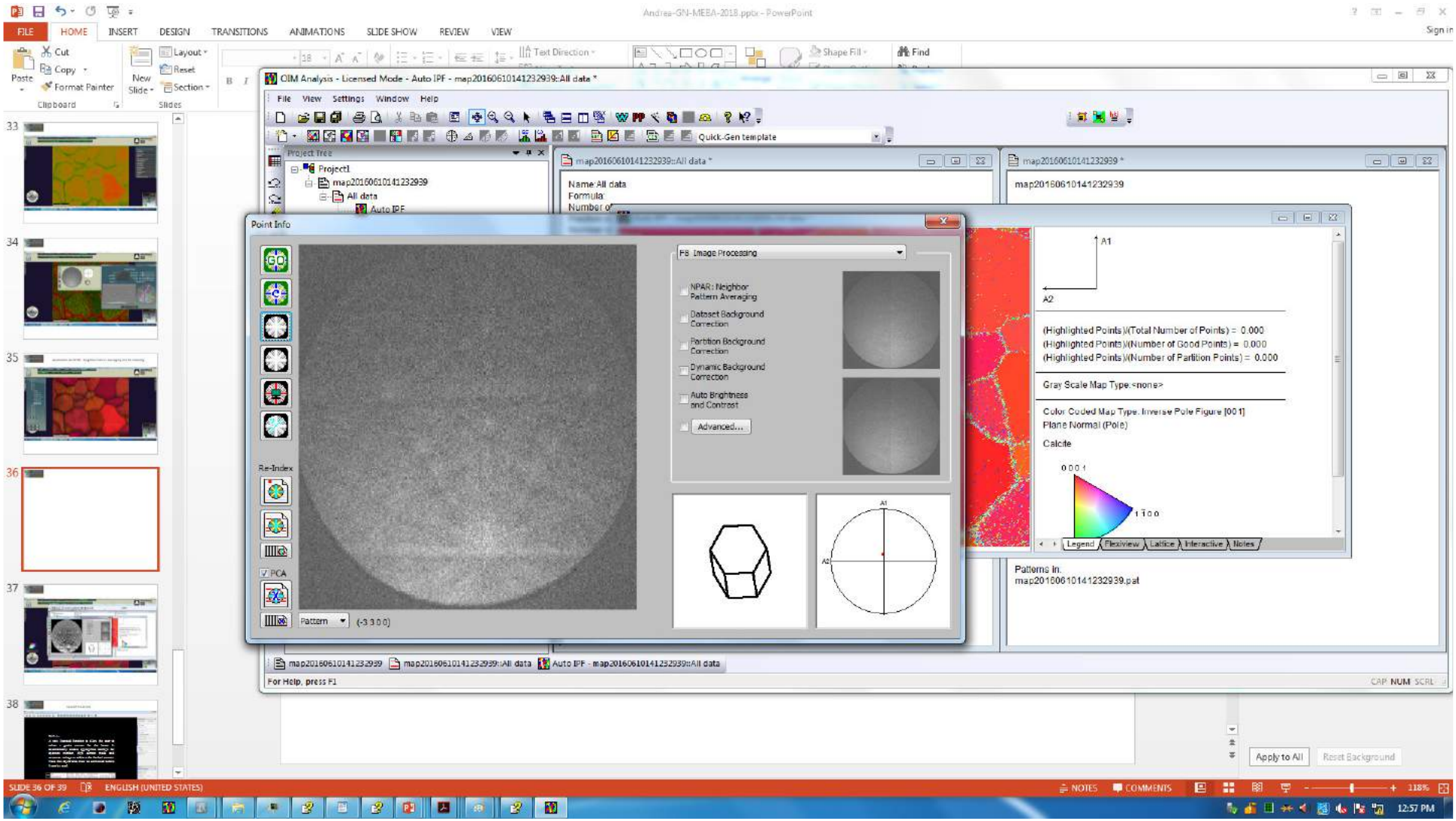
Utilisation du programme OIM



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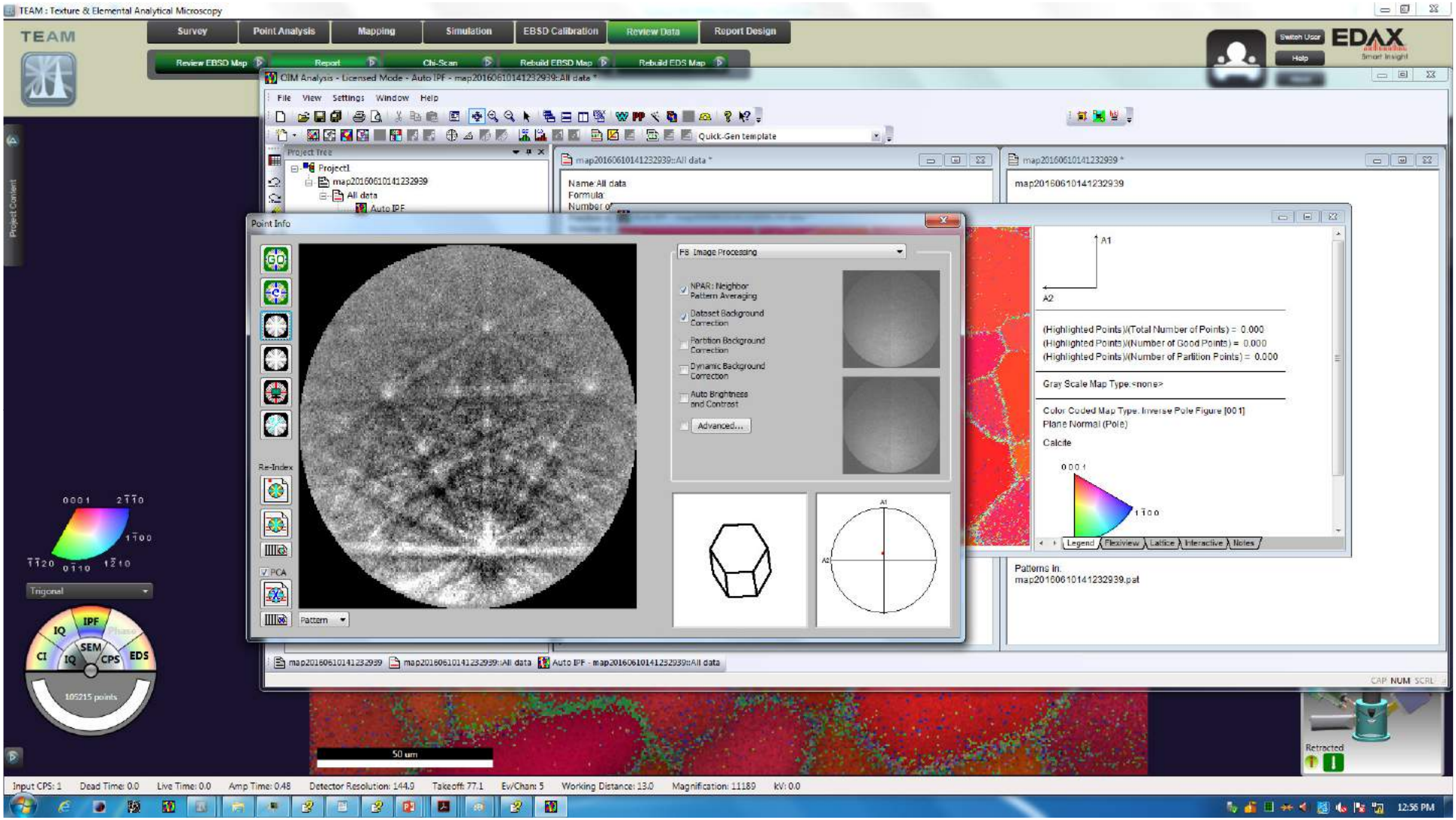
Utilisation du programme OIM



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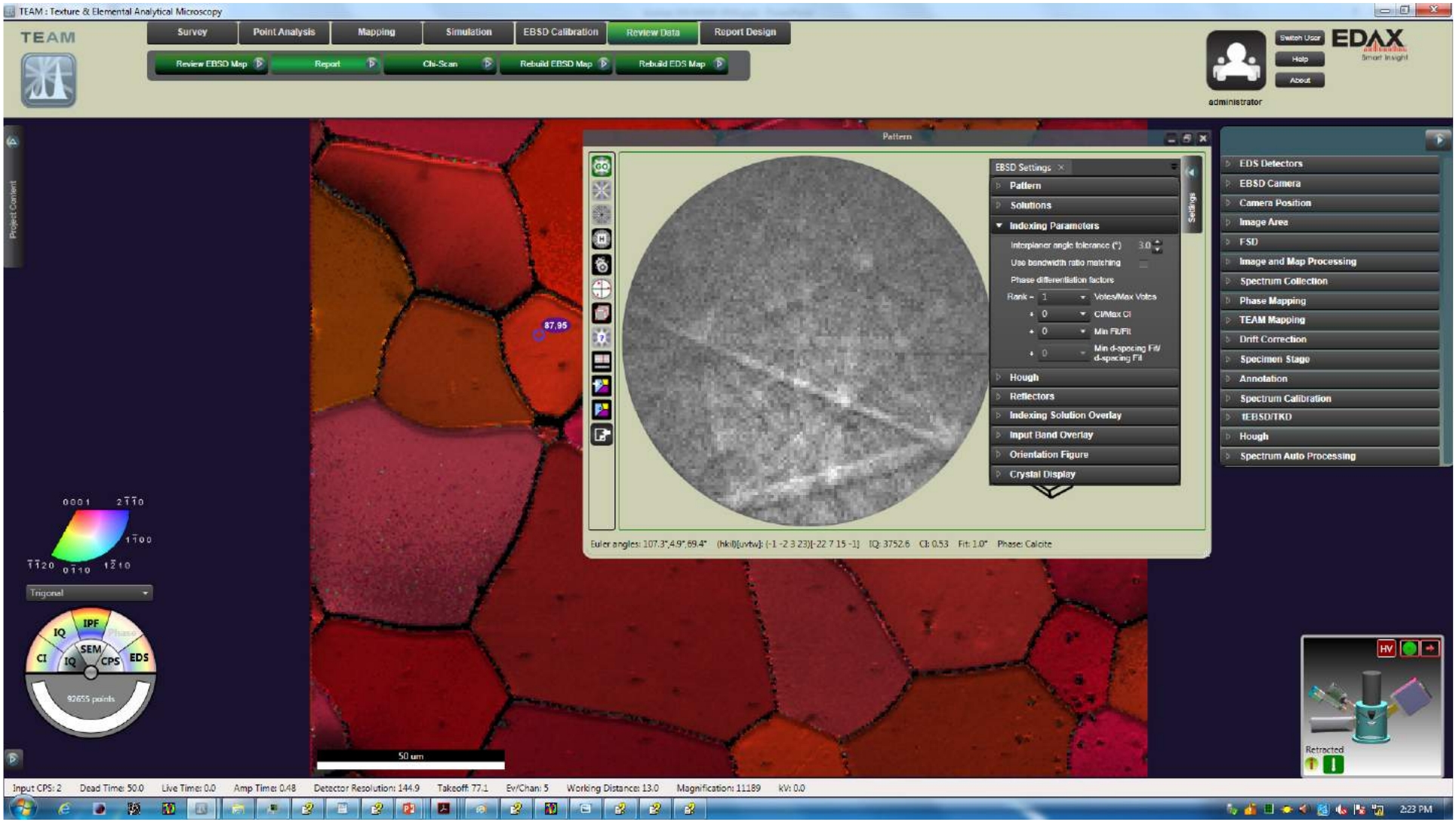
Mode nano VP (avec beamsleeve) - 30 Pa



CI et IQ

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Static Background Division
Dynamic Background Subtraction
Intensity Histogram Normalization

Mode Nano VP (avec beamsleeve) - 60 Pa

L'avantage du mode Nano VP est de pouvoir avoir des résultats similaires avec des pressions plus élevées

CI VERT

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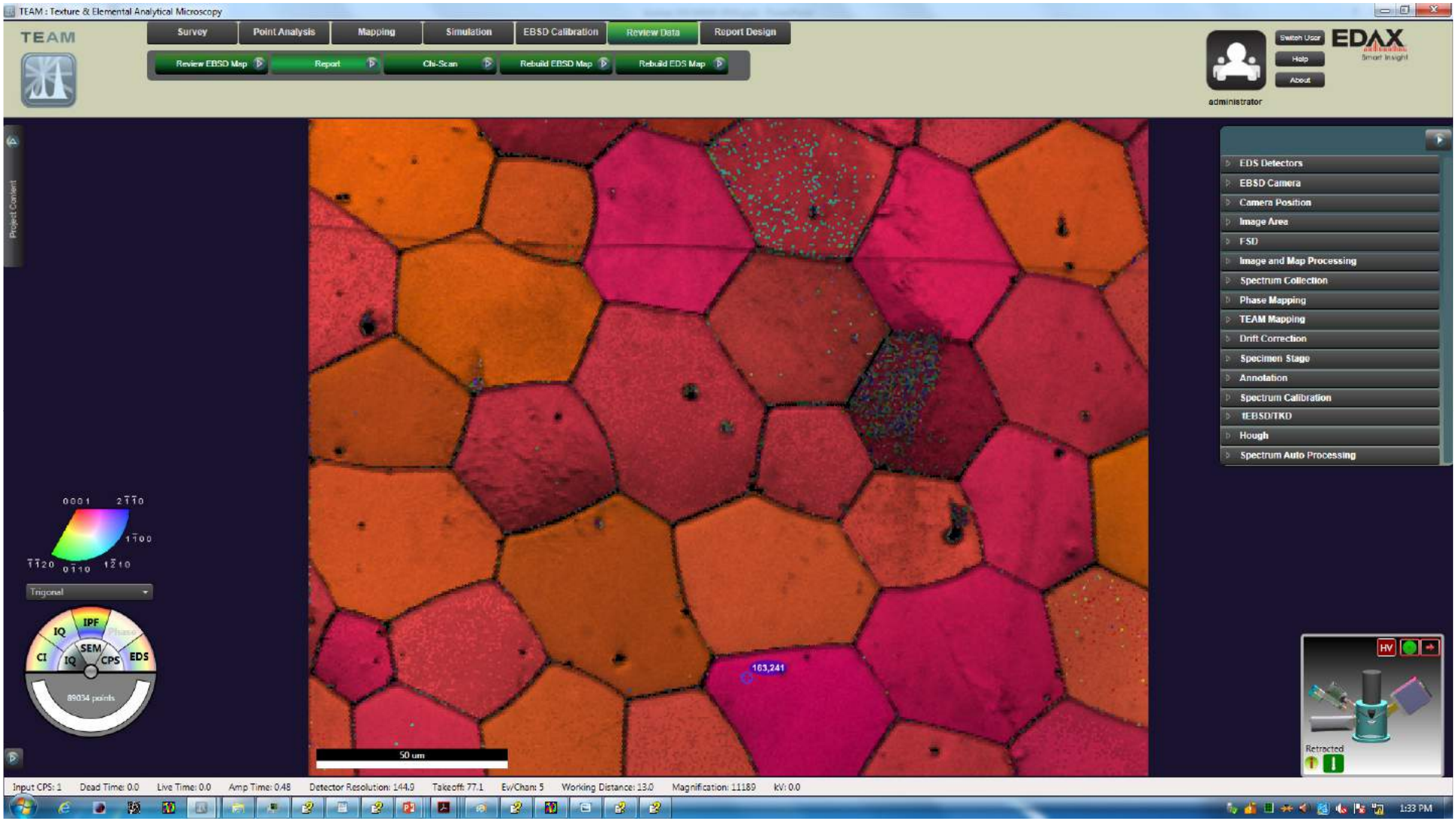
Mode Nano VP (avec beamsleeve) - 60 Pa



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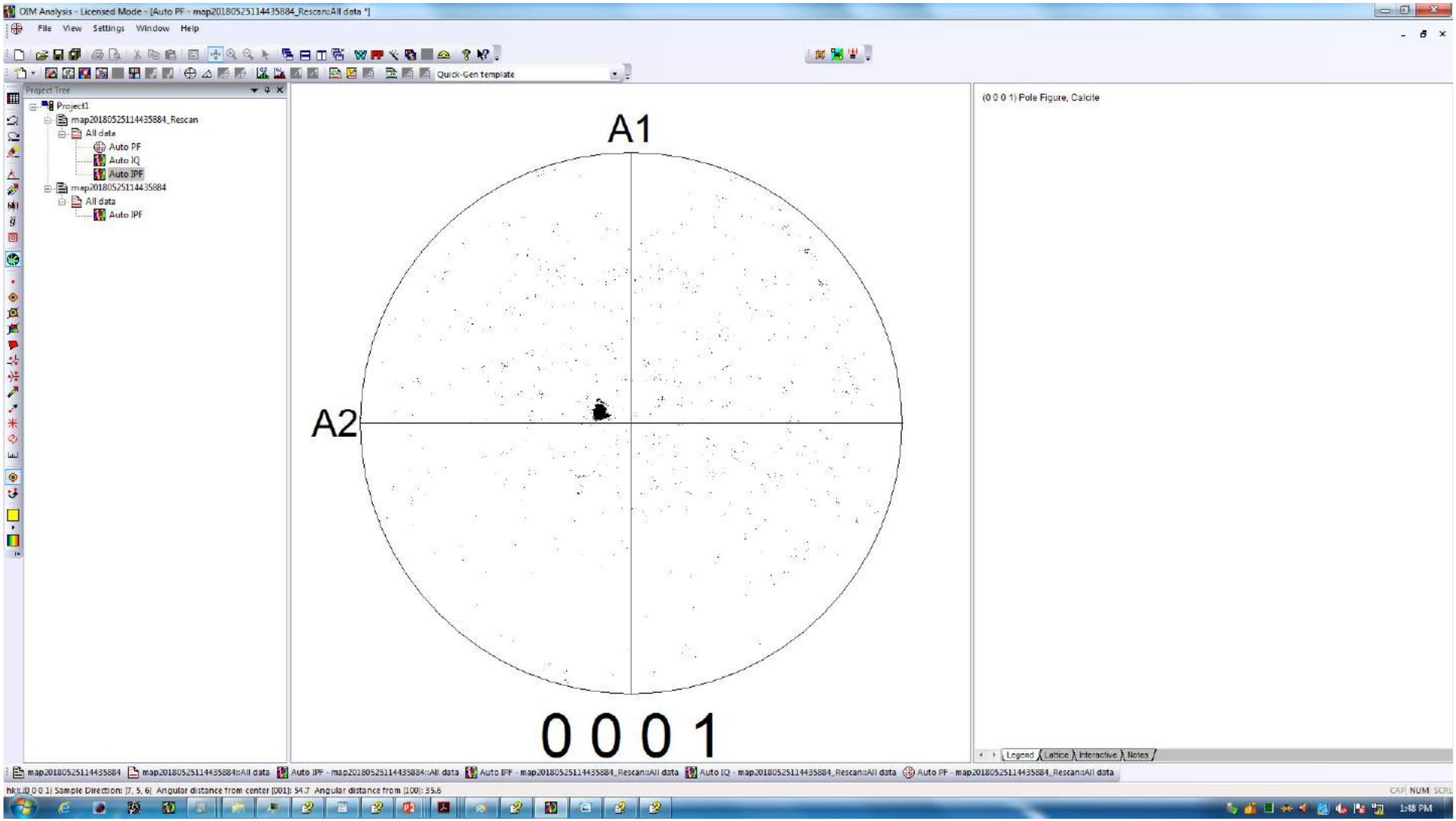
et de Microanalyse



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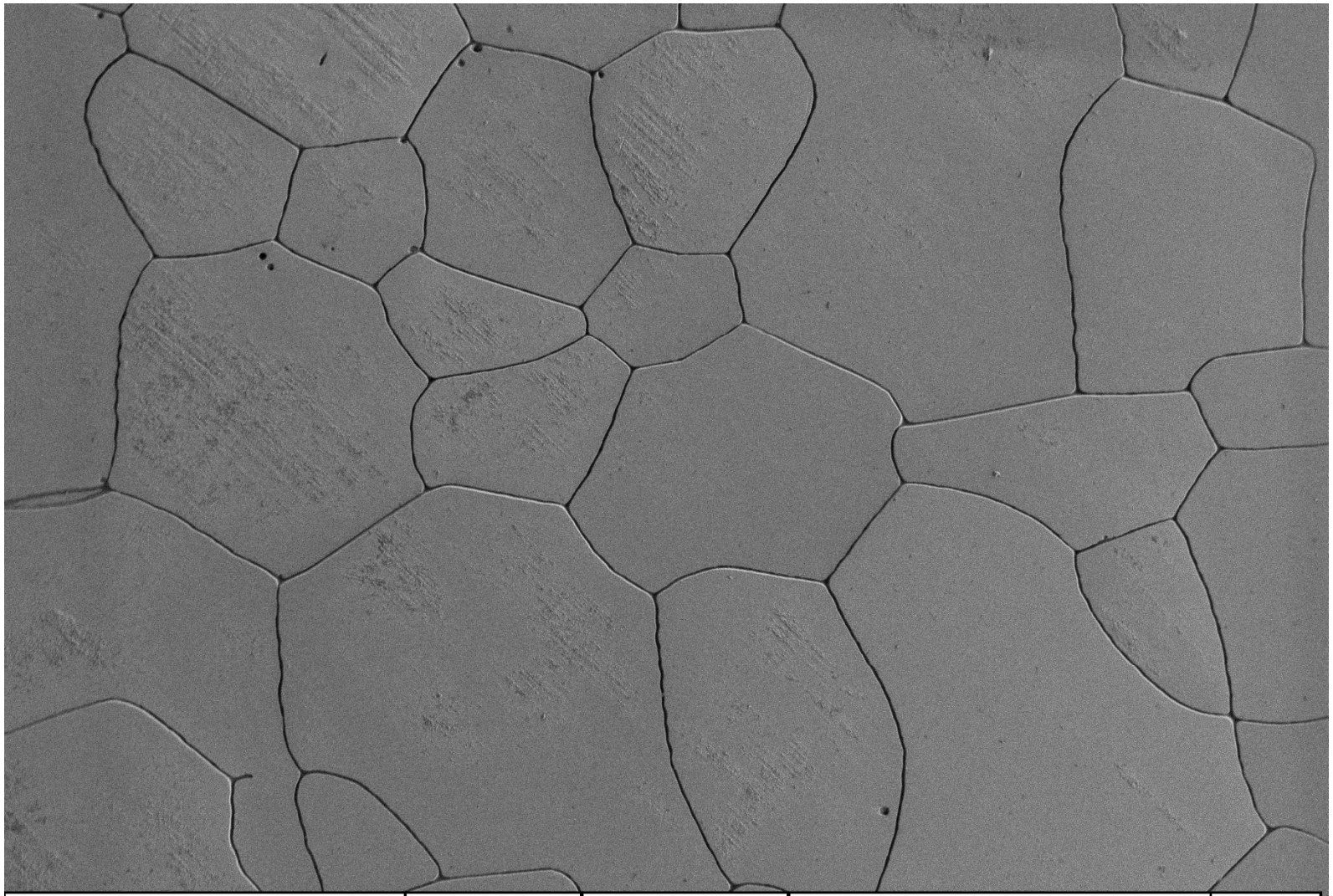

ZEISS 20 µm Gemini SEM 500 70-04	EHT = 1.00 kV WD = 6.4 mm Signal A = HE-SE2	Mag = 438 X Pixel Size = 63.74 nm B = 49.5 % C = 40.9 %	Noise Red. = Drift Comp. Frame Avg. Scan Speed = 3 P = 1.41e-005 mbar Cycle Time = 7.4 Mins Ap. Size = 20.00 µm	10 Jun 2016 17:11:51 CP2M

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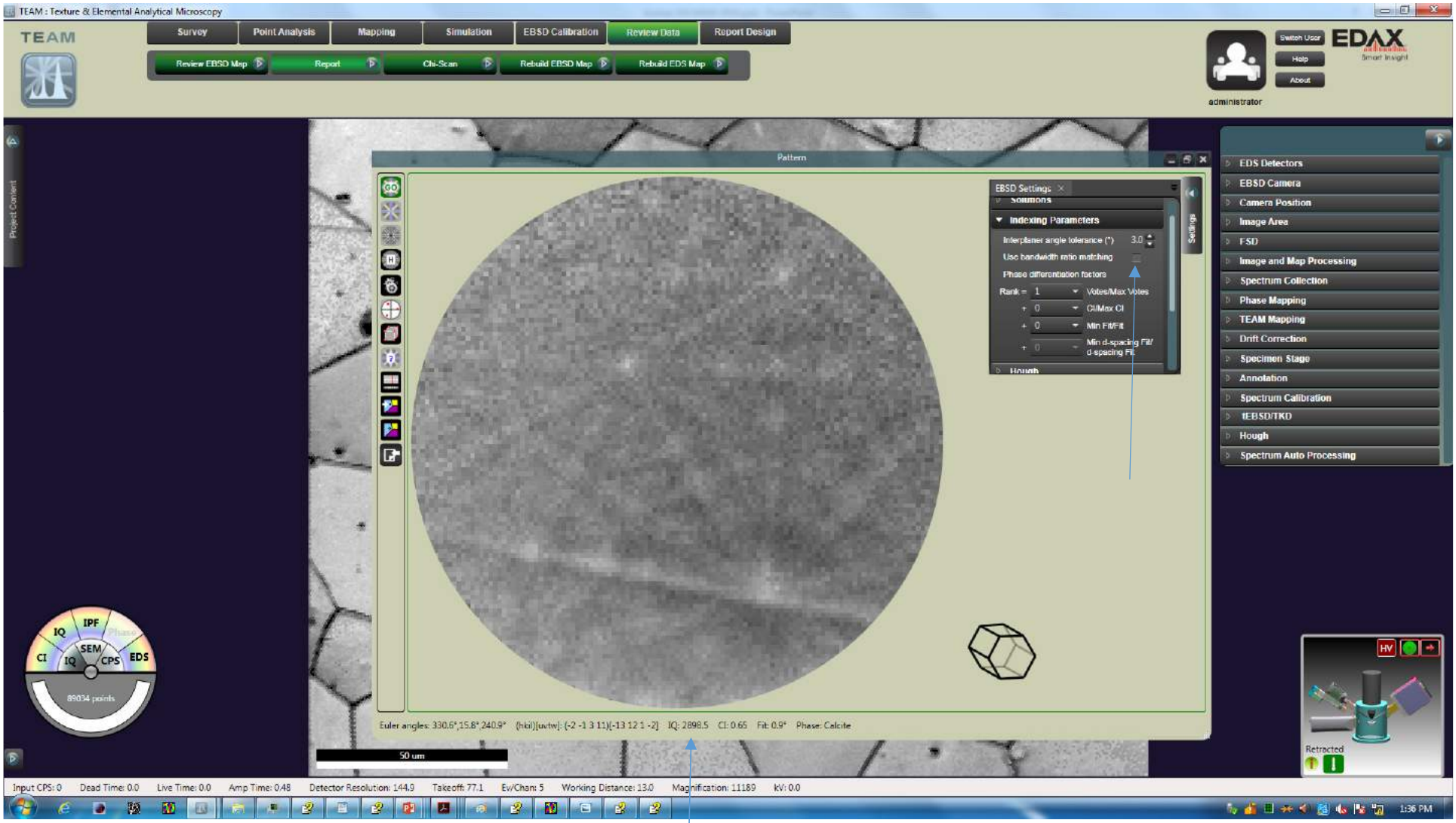
Mode Nano VP (avec beamsleeve) - 60 Pa



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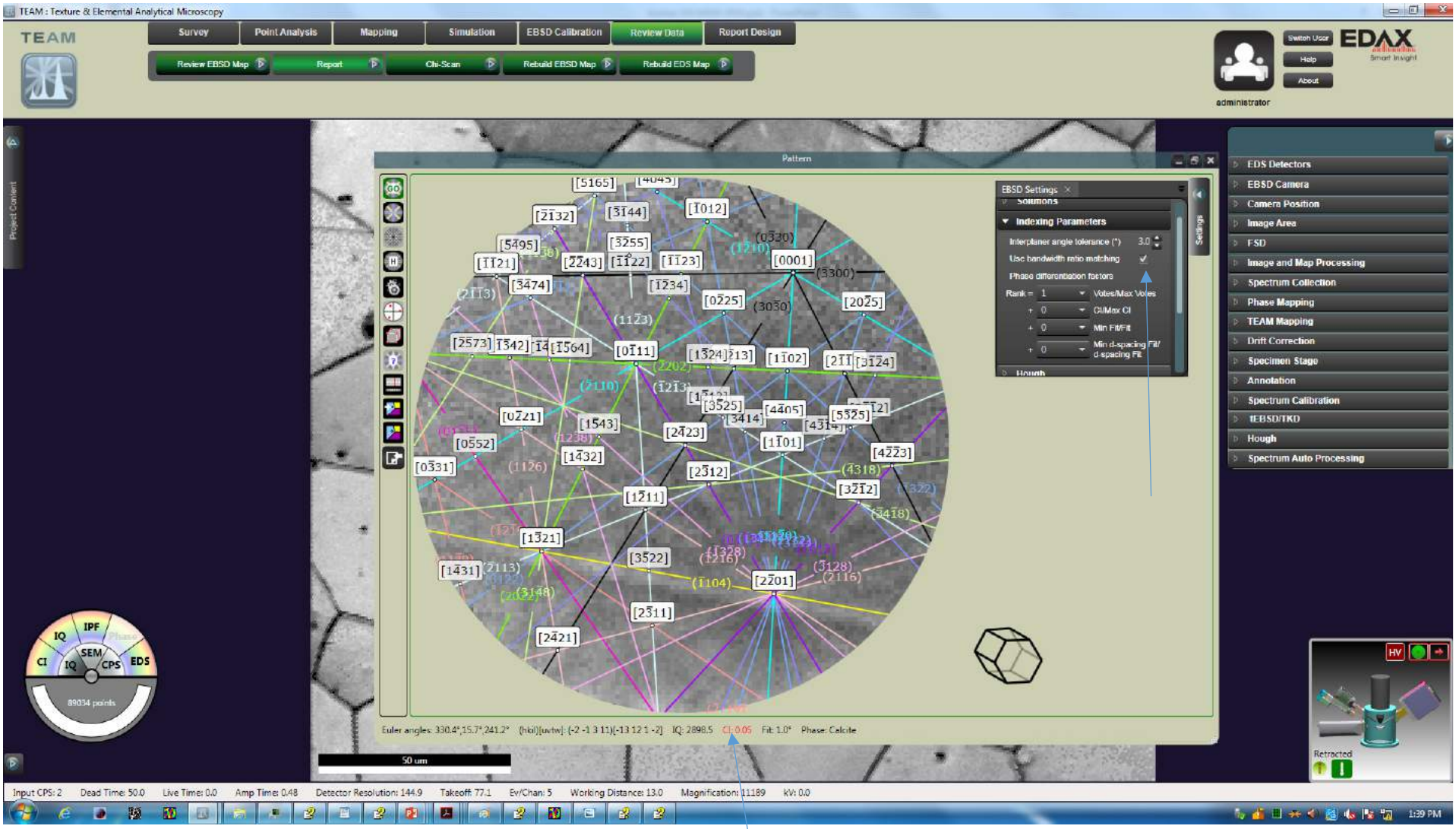
Mode Nano VP (avec beamsleeve) - 60 Pa



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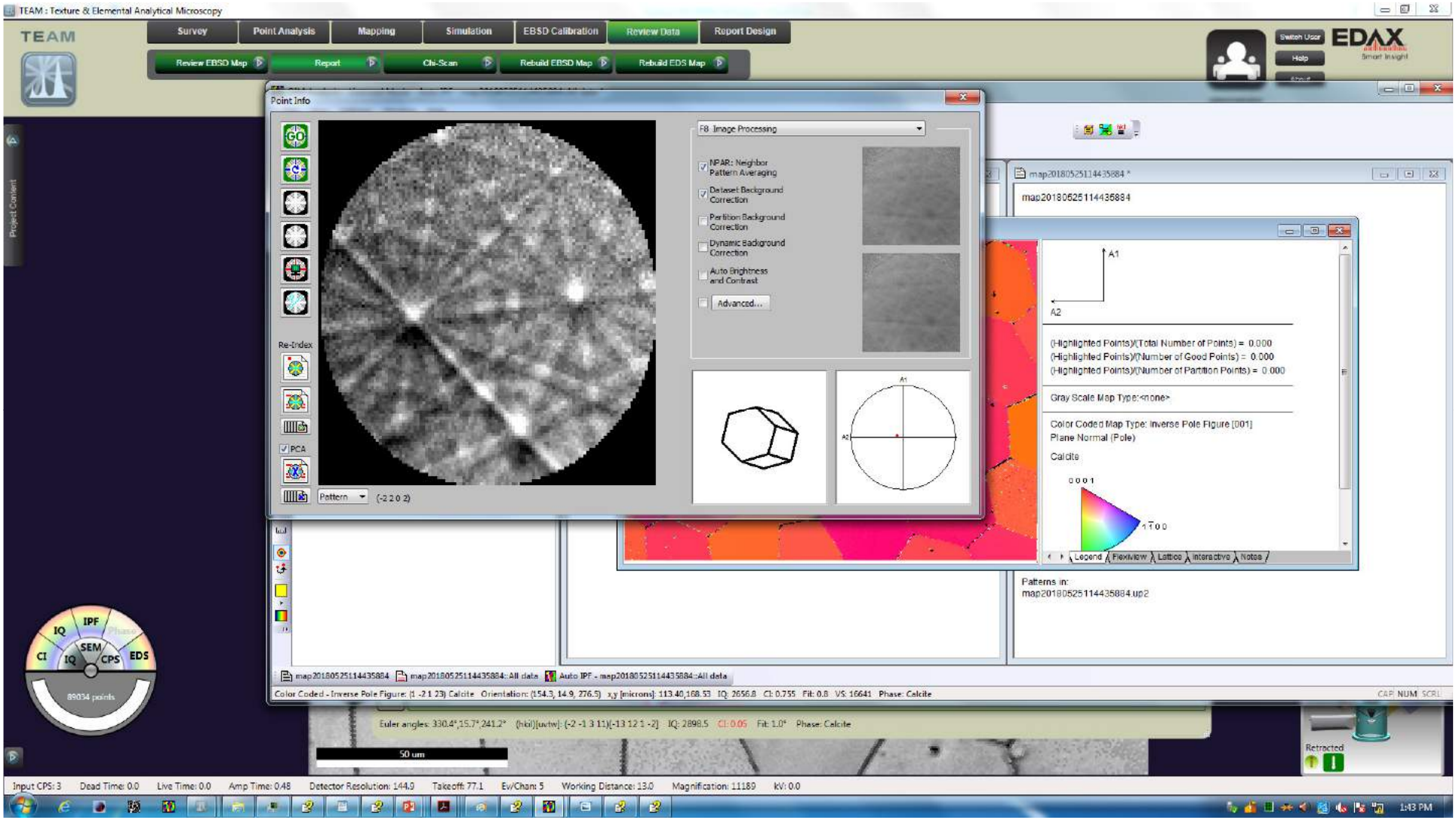
Mode Nano VP (avec beamsleeve) - 60 Pa - avec OIM



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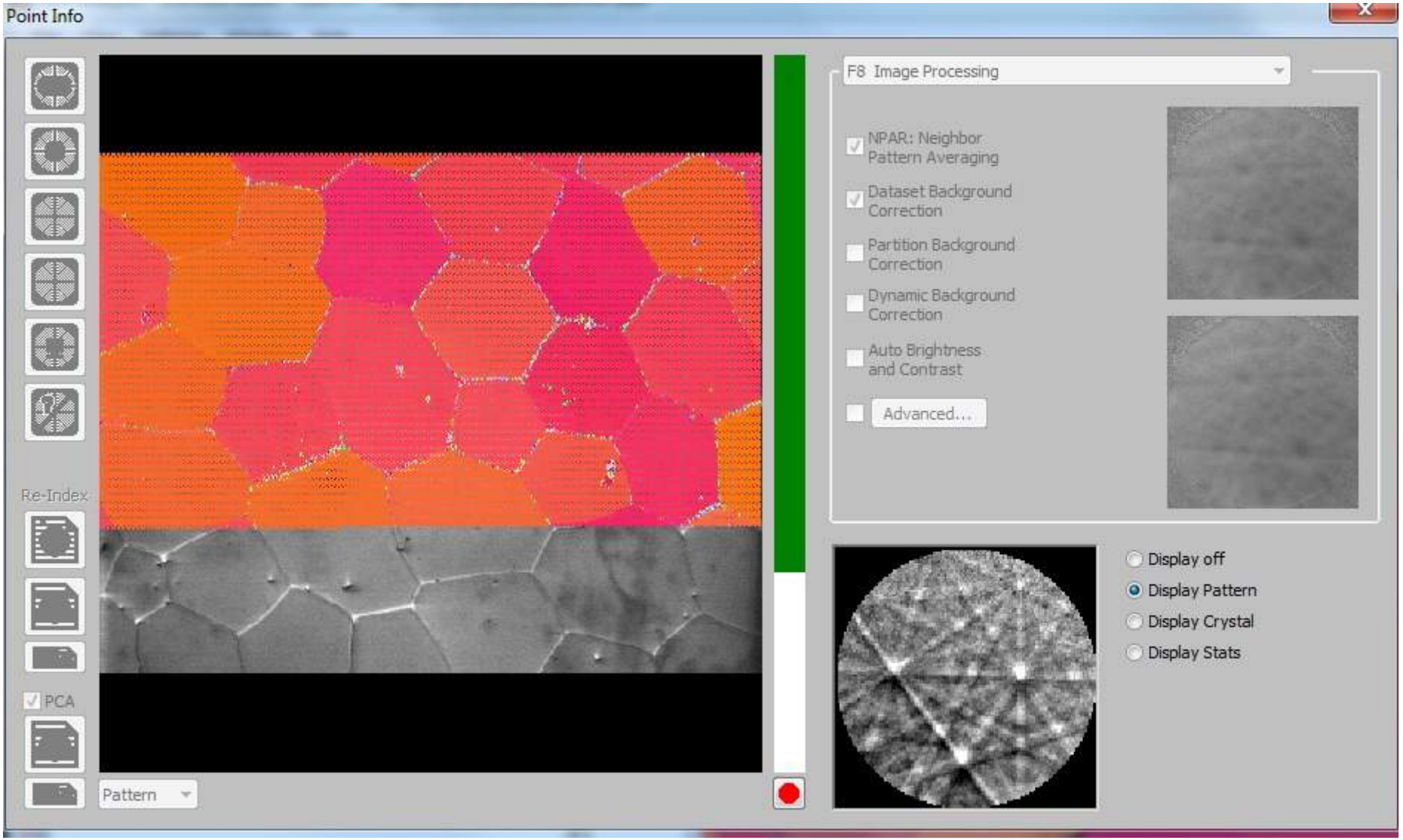
Post – processing OIM



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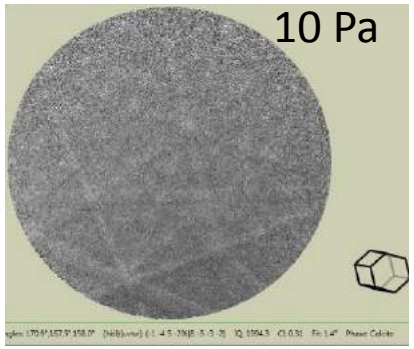
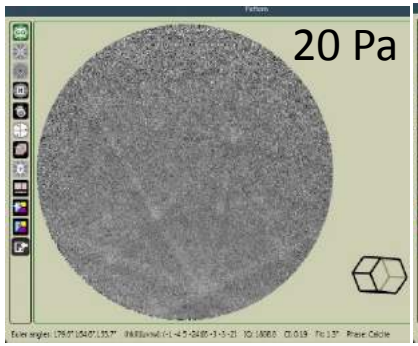
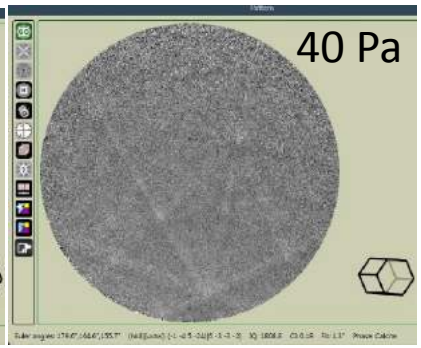
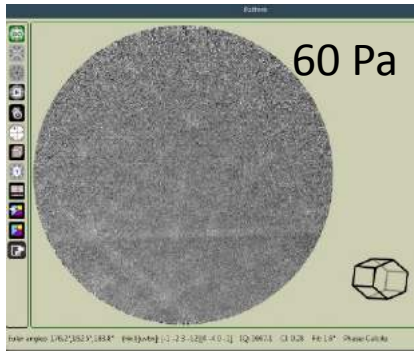
99 fps gain 0.8 max binning 2 x2 pas 0.7 µm
I Faraday 10 nA

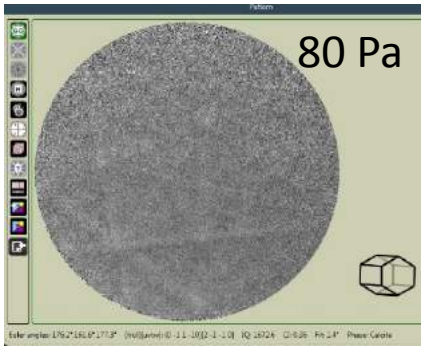
Static Background Division
Dynamic Background Subtraction
Intensity Histogram Normalization

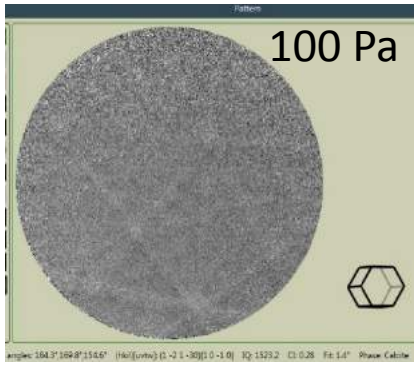


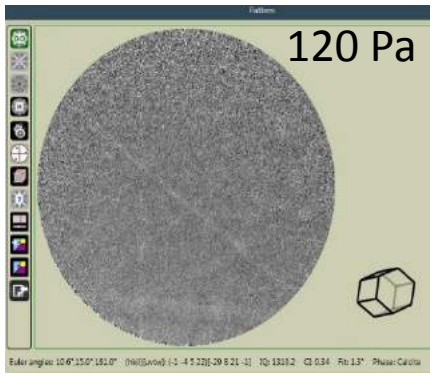


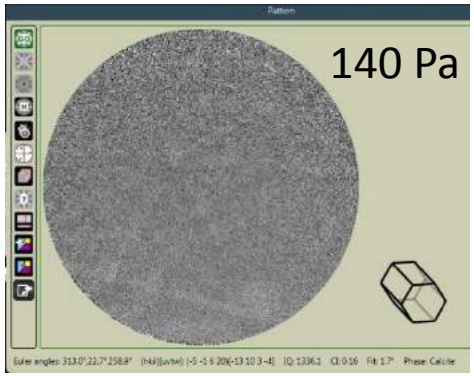


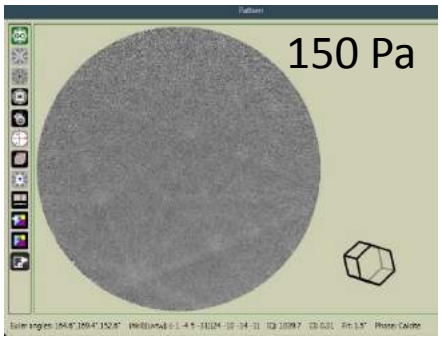


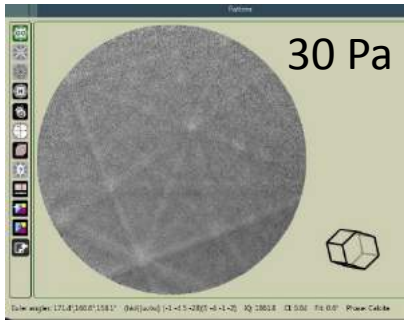












Mode Nano VP avec Beamsleeve (cône) 350 µm

IQ ↓ avec la pression mais il est possible indexer

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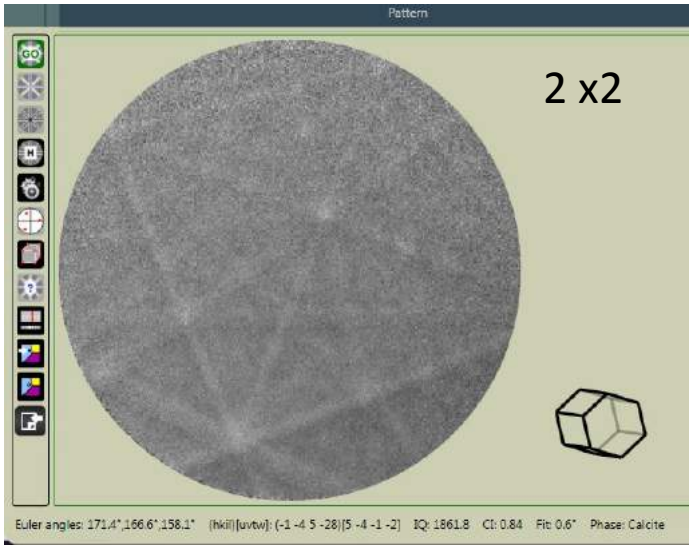
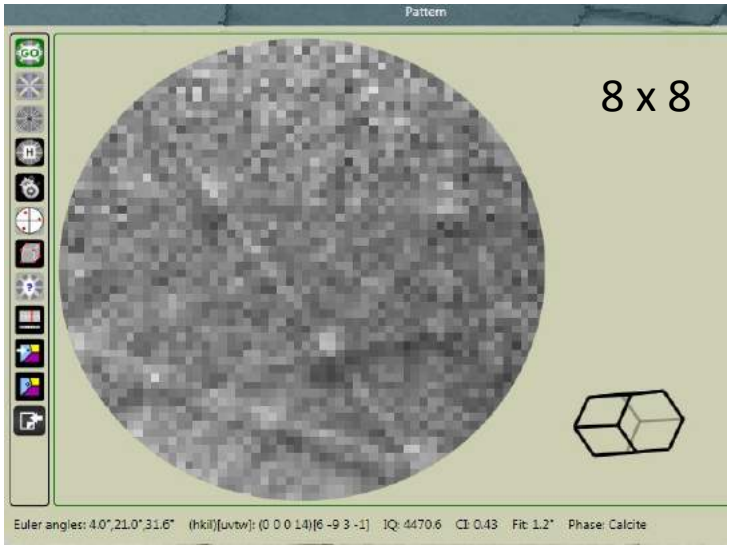
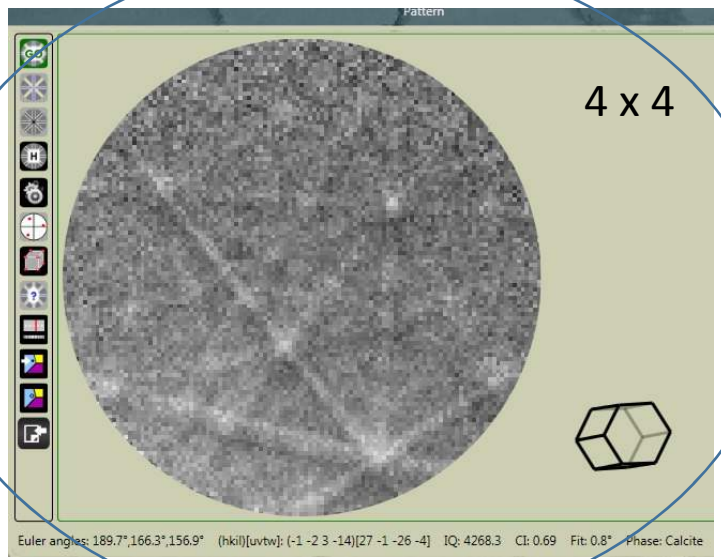
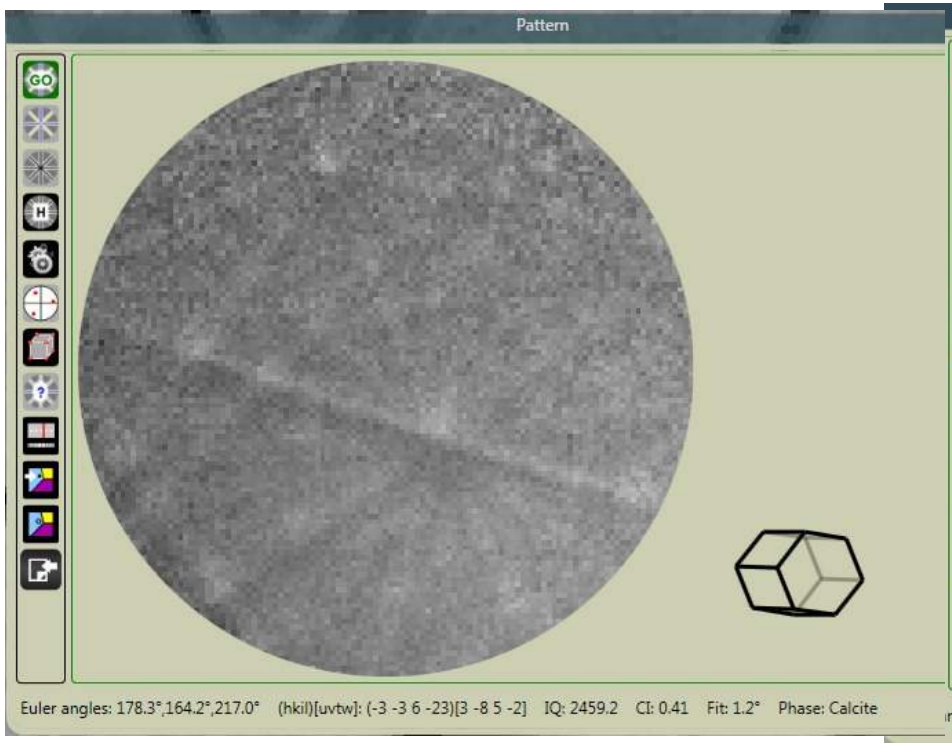
Binning 30 Pa

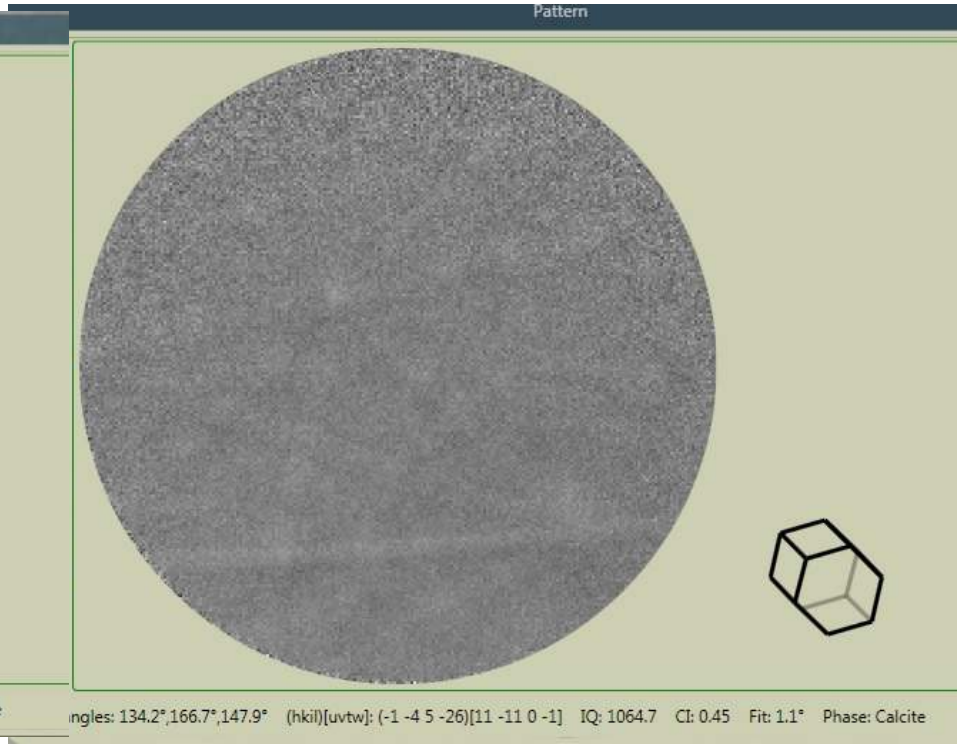






Courant - 30 Pa

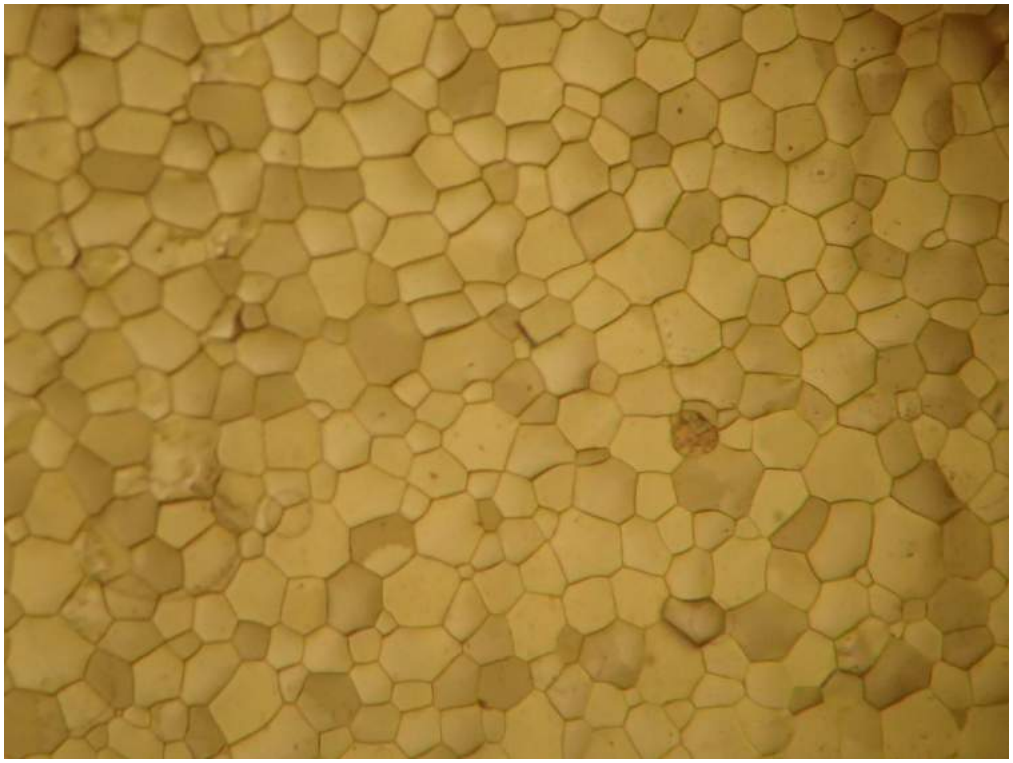




I Faraday = 10.57 nA
Zeiss mode Analytical

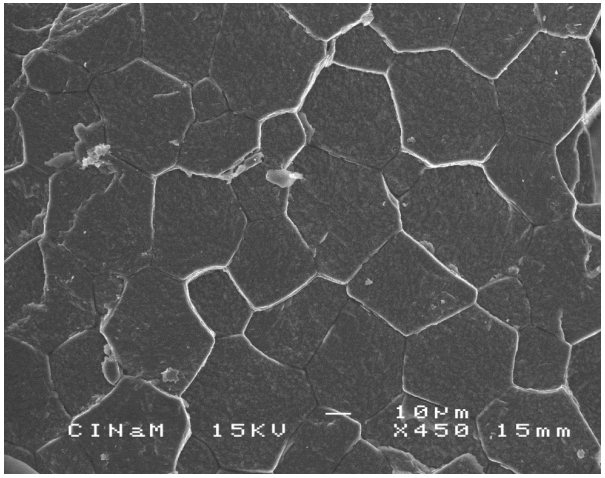
I Faraday = 5.44 nA
Zeiss mode Imaging

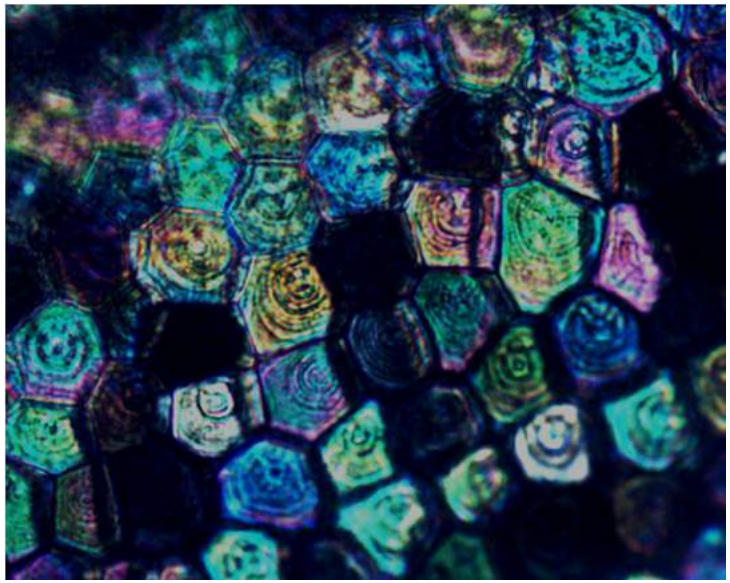
Echantillon 2



Microscope en réflexion

Calcite





Microscope polarisant

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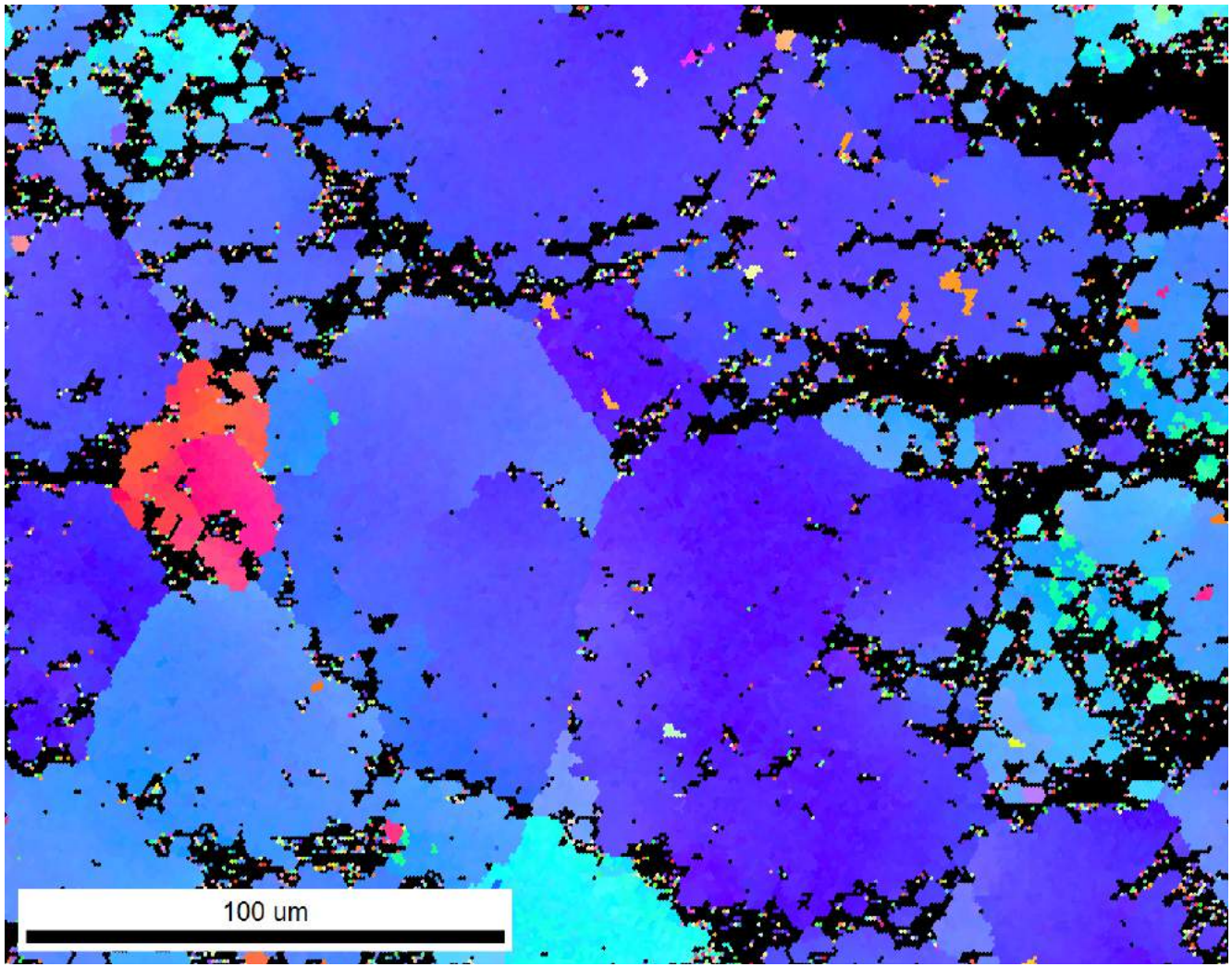
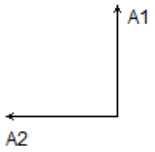
Echantillon 2





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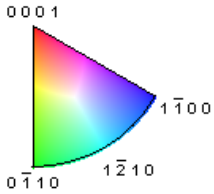
(Highlighted Points)/(Number of Good Points) = 0.000
(Highlighted Points)/(Number of Partition Points) = 0.000

Gray Scale Map Type:<none>

Color Coded Map Type: Inverse Pole Figure [001]

Calcite



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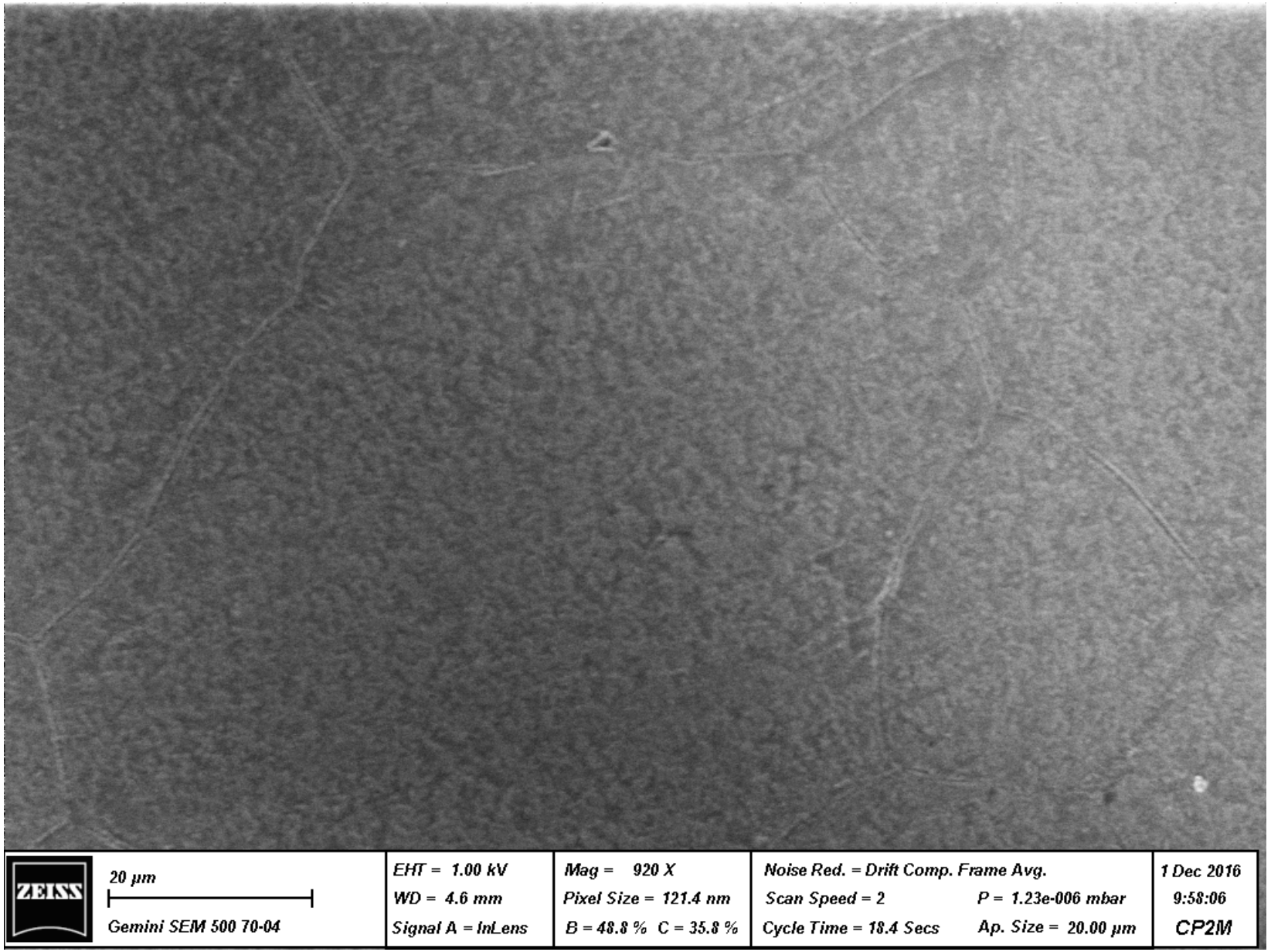
et de Microanalyse



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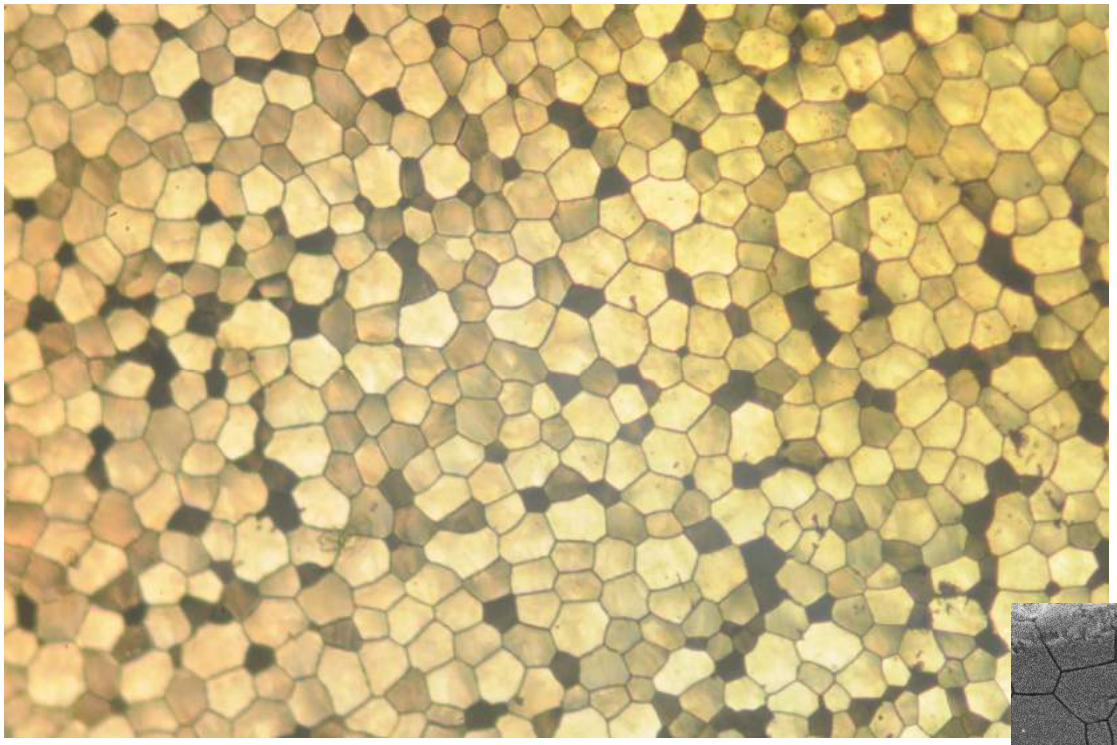
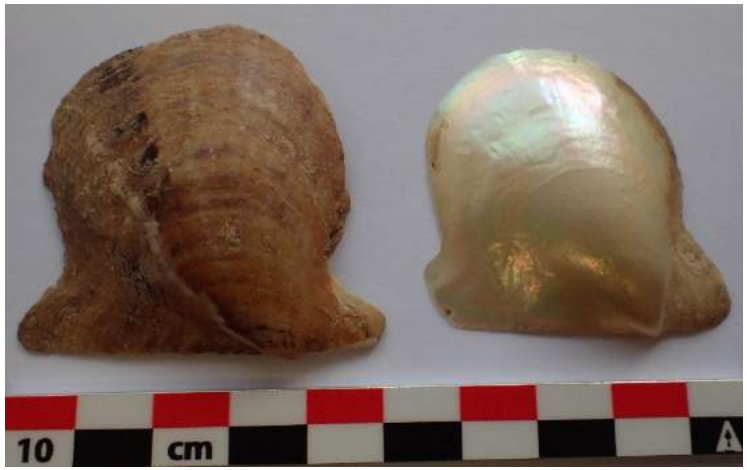
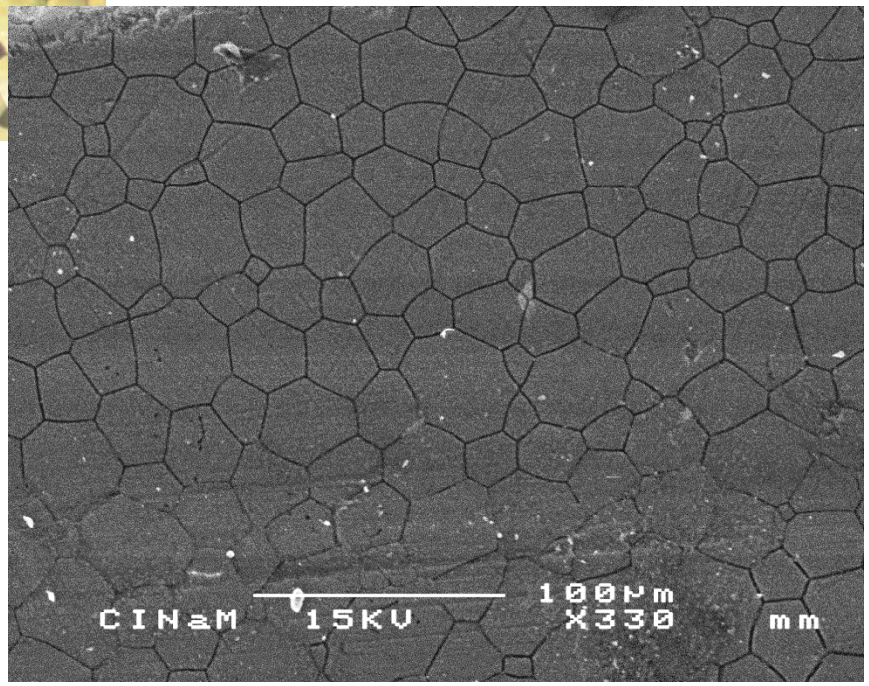


Microscope polarisant





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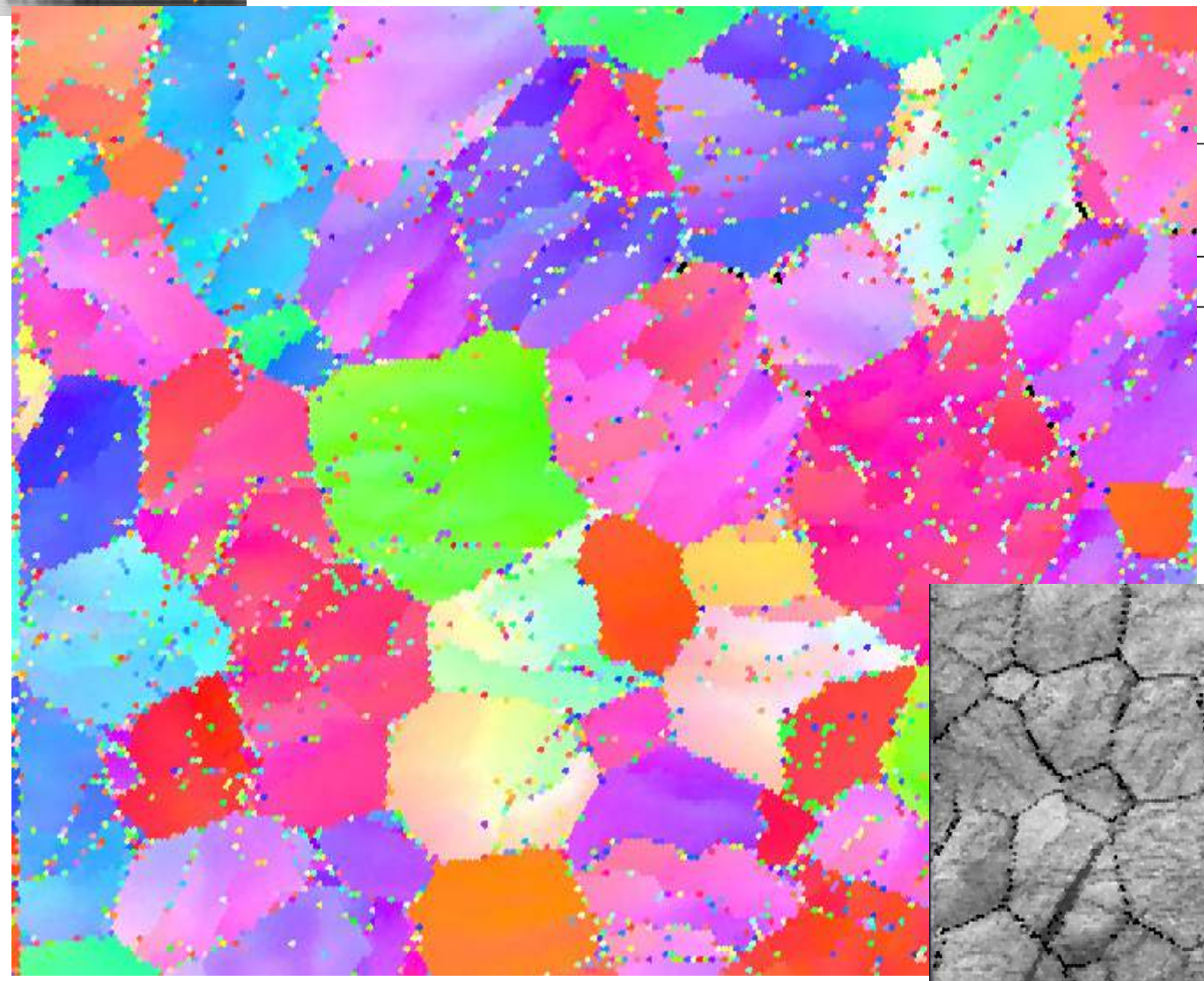
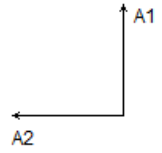
et de Microanalyse





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(Highlighted Points)/(Number of Good Points) = 0.000
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Gray Scale Map Type:<none>

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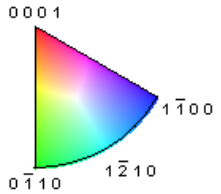
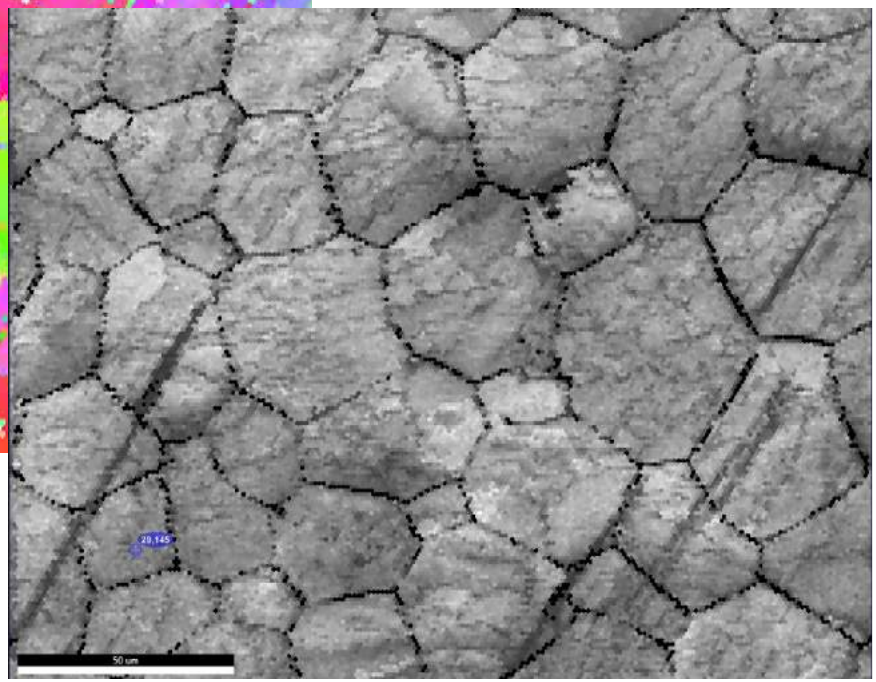
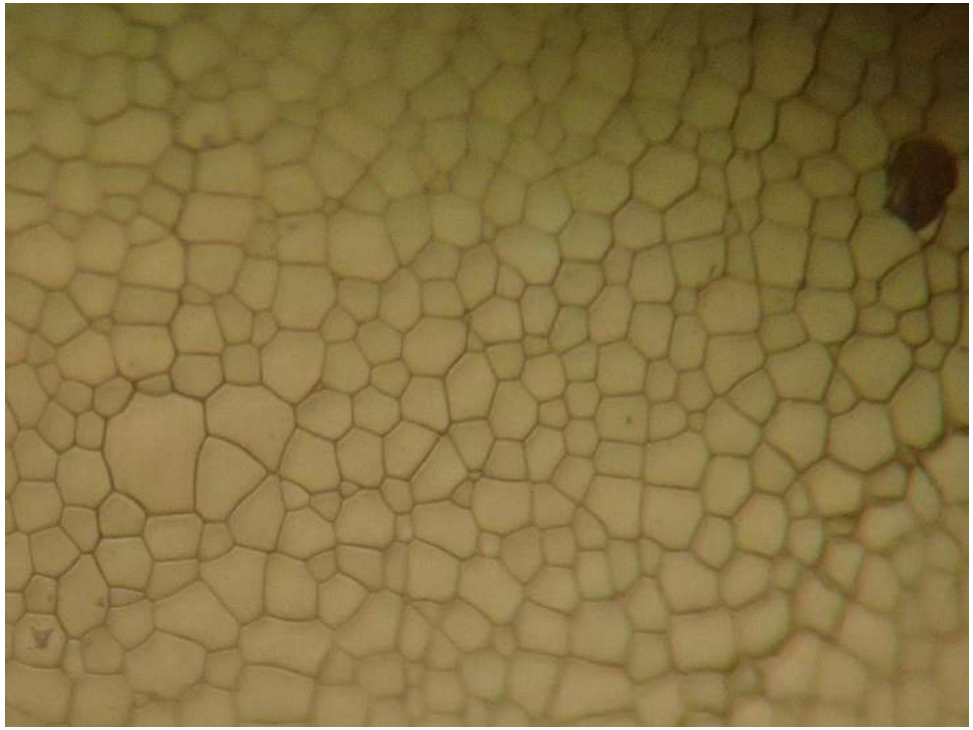


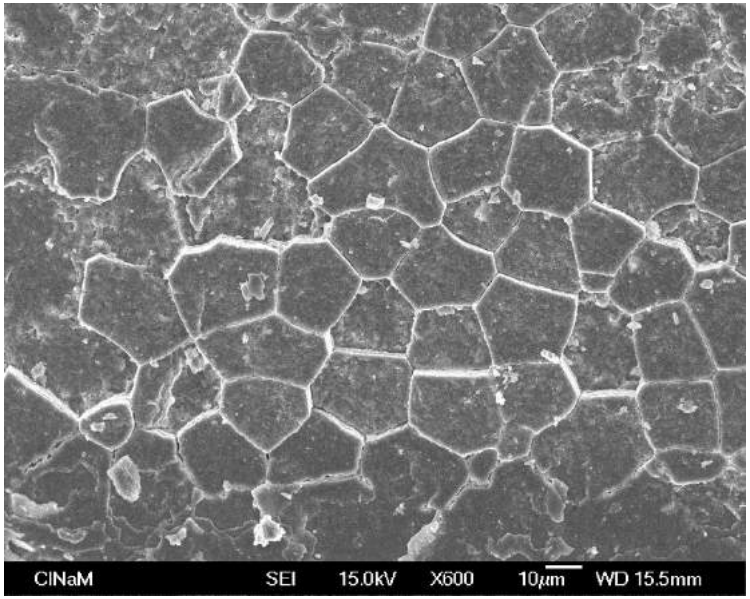


Echantillon 4



Microscope en réflexion

Aragonite



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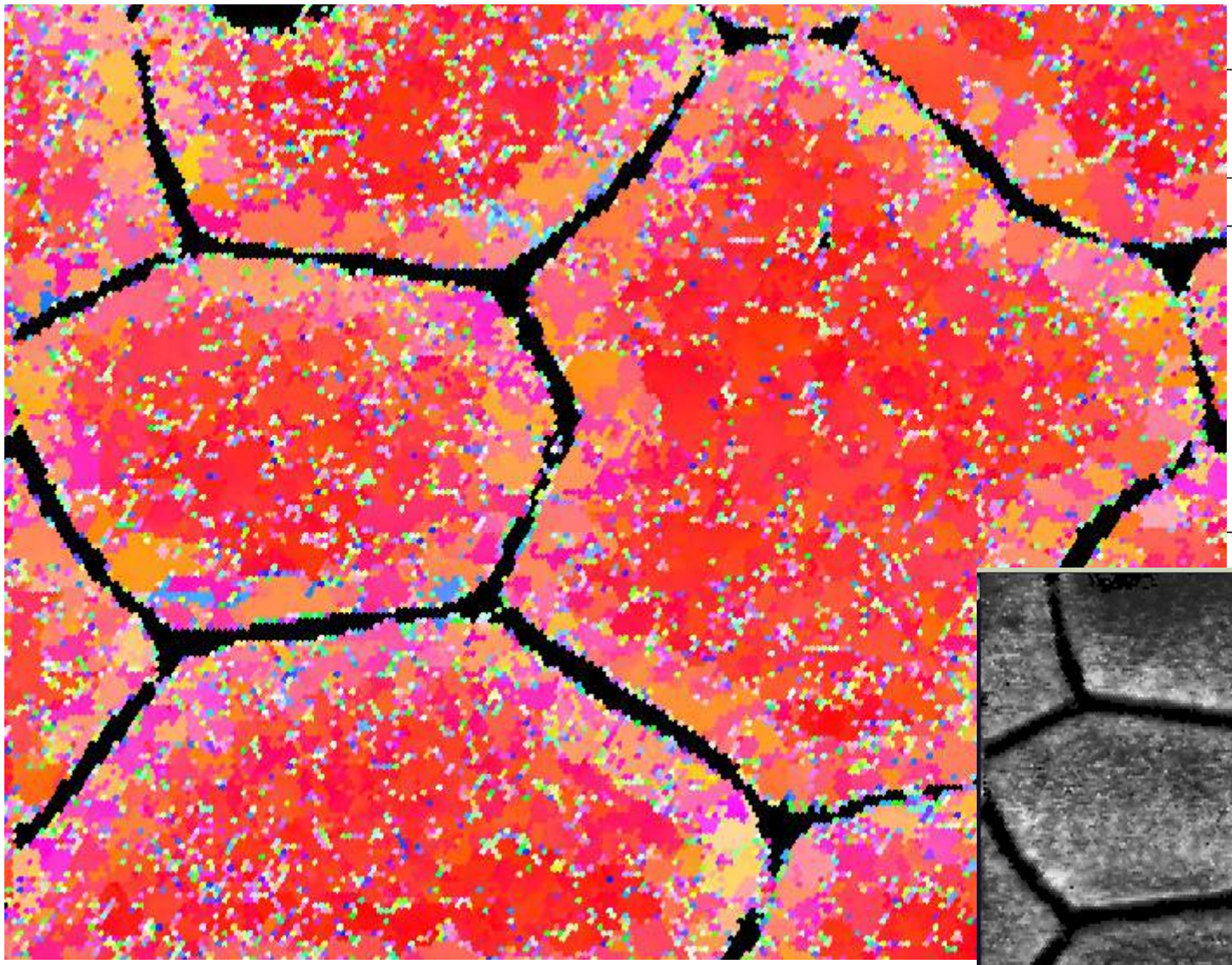

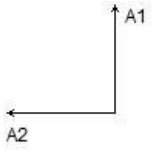

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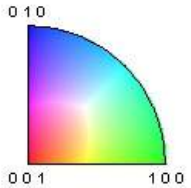
Gray Scale Map Type:<none>

Color Coded Map Type: Inverse Pole Figure [001]
Plane Normal (Pole)

Aragonite



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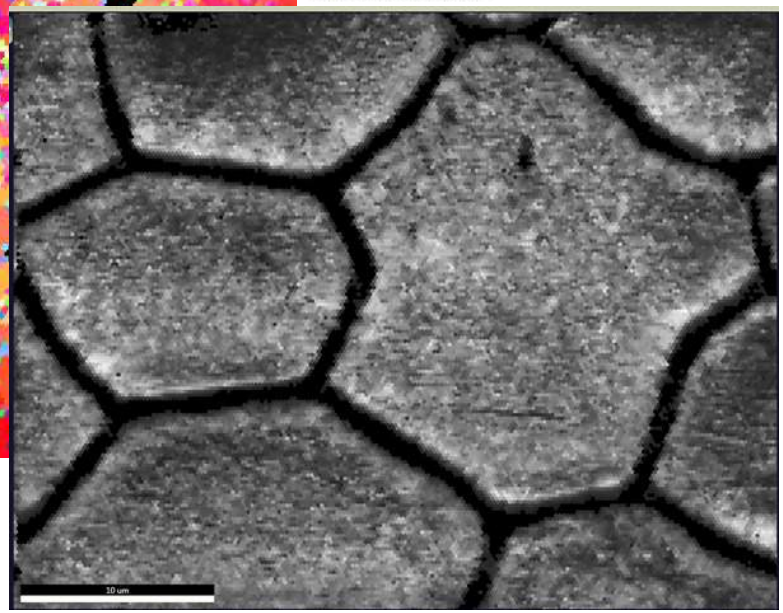

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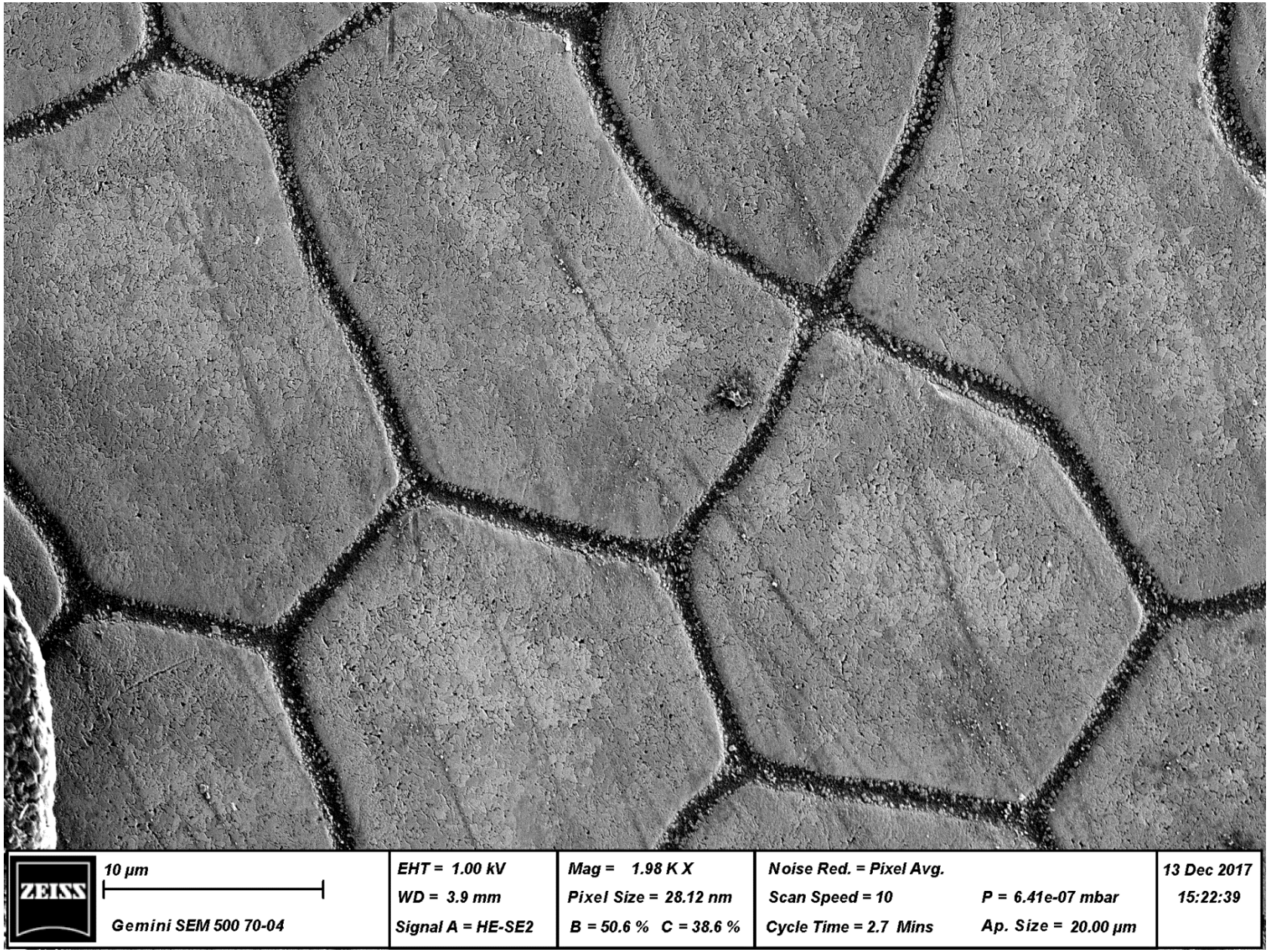
et de Microanalyse



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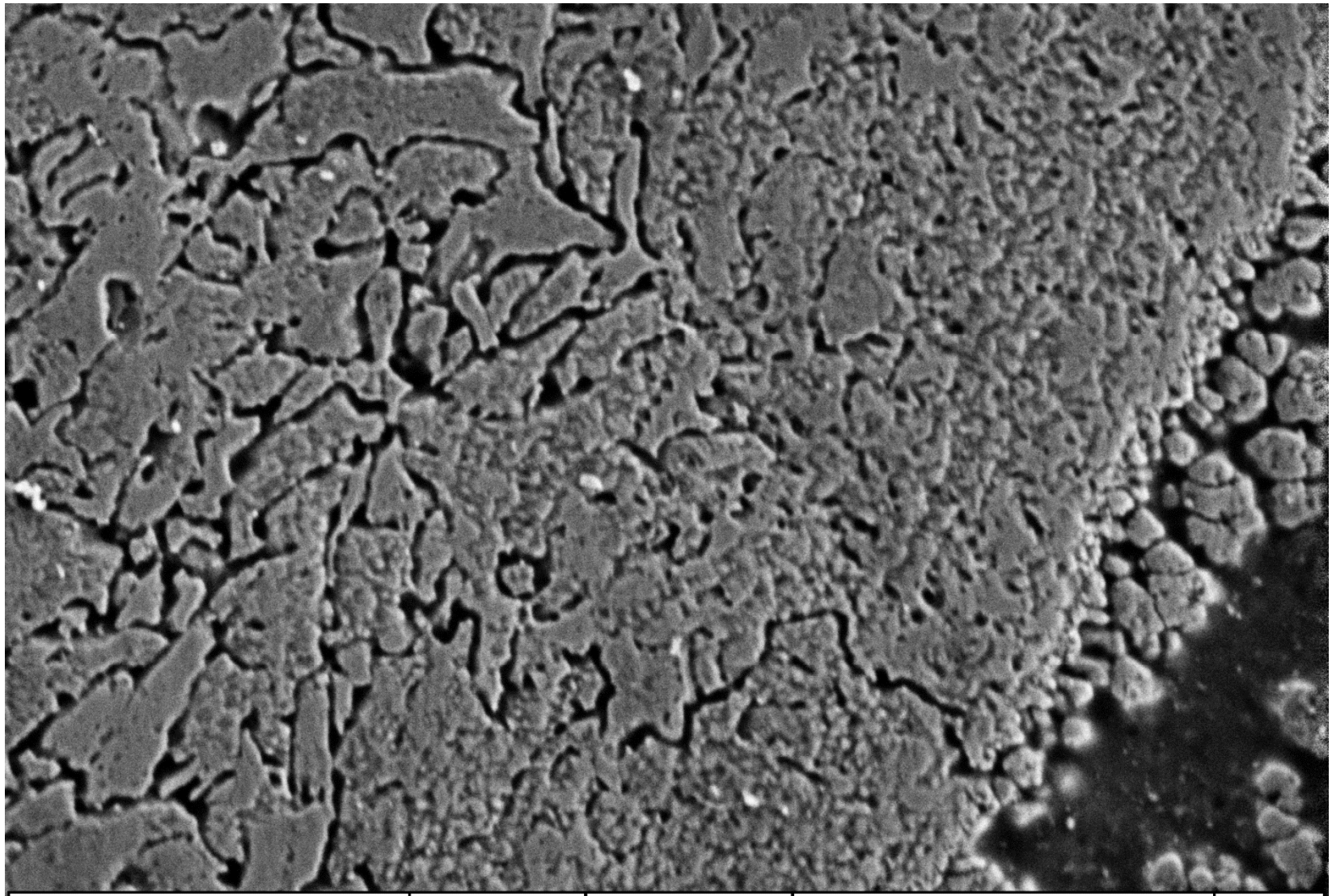
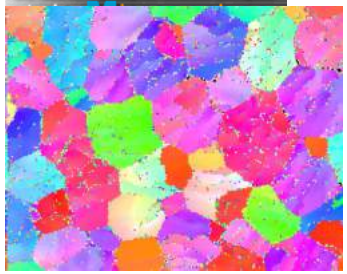
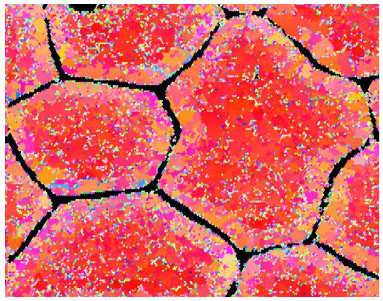


Centre Pluridisciplinaire de Microscopie électronique



CONCLUSION

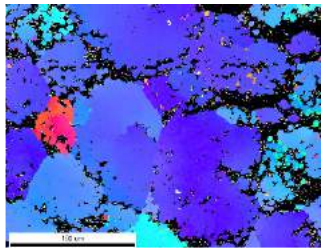


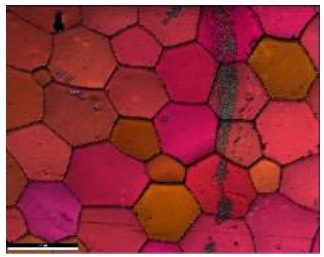
→ La carte d'orientations EBSD montre qu'il n'y a pas de contrôle de cristallisation

→ Possibilité de tracer la partie organique via EDS

→ Imagerie et EBSD dans une vaste gamme de pressions. Pour le cas du système Zeiss, travailler en VP à 30 Pa et en nano VP entre 60 et 80 Pa

→ Avec l'augmentation de la pression, diminution du IQ, mais amélioration avec le post-processing





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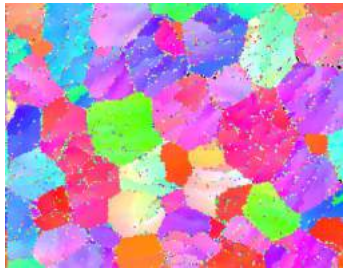
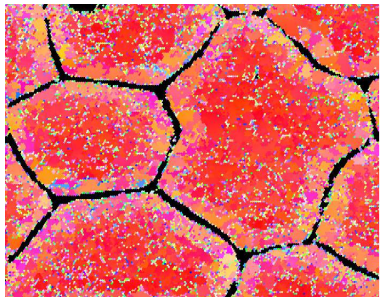


Remerciements



EDAX – Rene DE KLOE
ZEISS – Zhou FANG
CP2M – Martiane CABIE, Christian DOMINICI
François BRISSET
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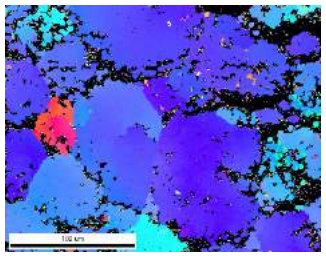

Merci pour votre attention!

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