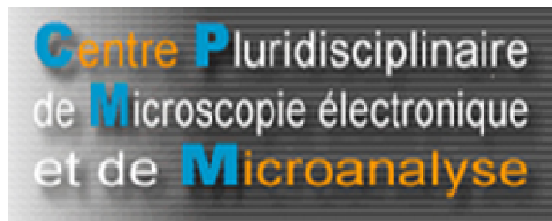


EBSD en mode pression variable: un exemple d'application pour l'étude des biomatériaux

A. Campos, O. Grauby
CP2M, CINaM

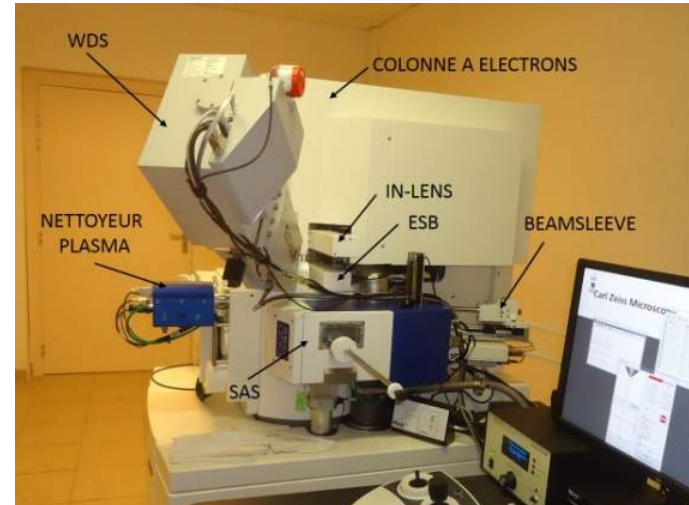


** Aix-Marseille University-CNRS*

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

MEB ZEISS Gemini 500 20 nA



Resolution	1.2 nm (500 V)	1.8 nm (3kV, 50 Pa VP)
	1.1 nm (1 kV)	
	0.6 nm (15 kV)	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	

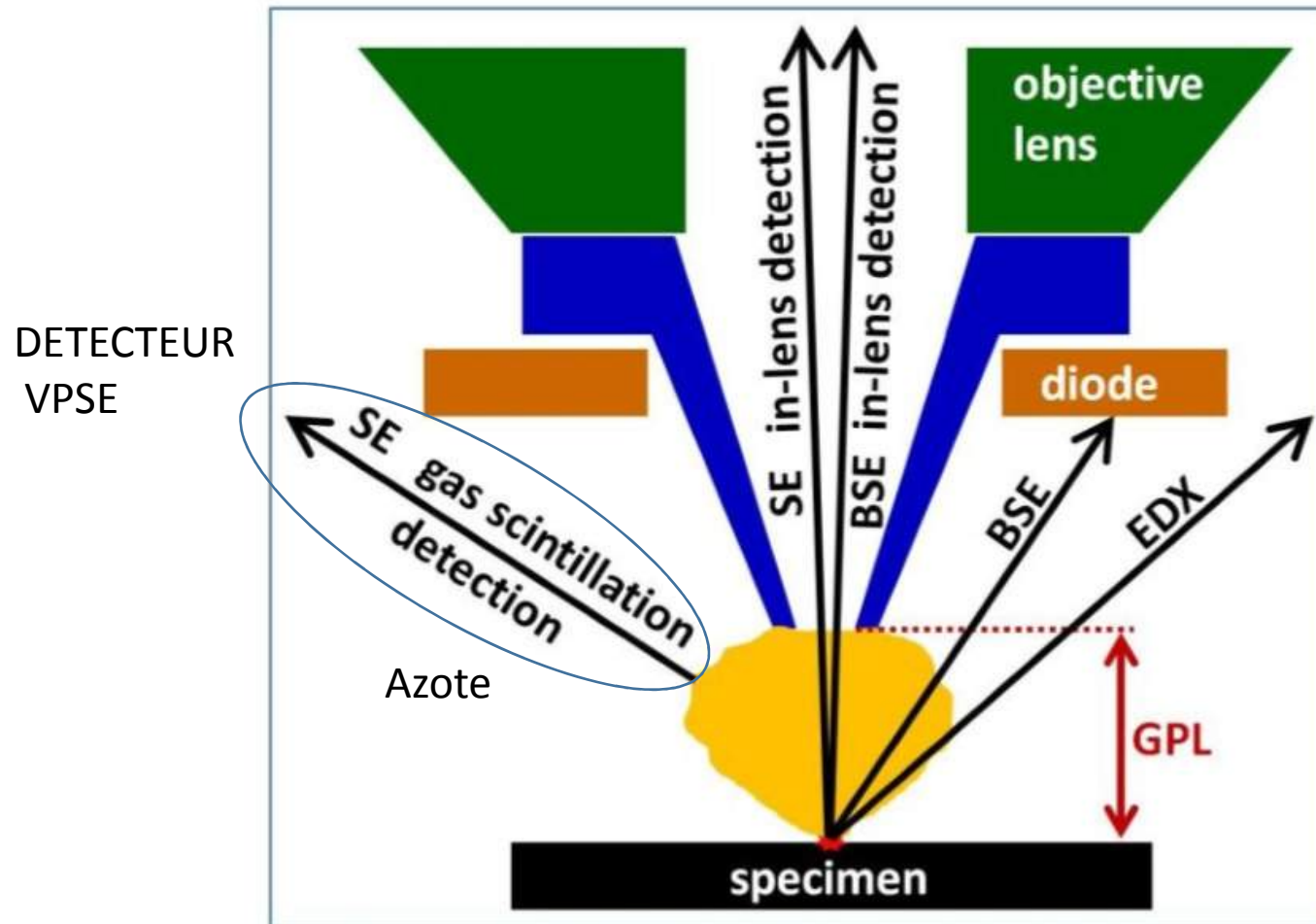
1.8 nm à 30 Pa et 3 kV

Notre configuration

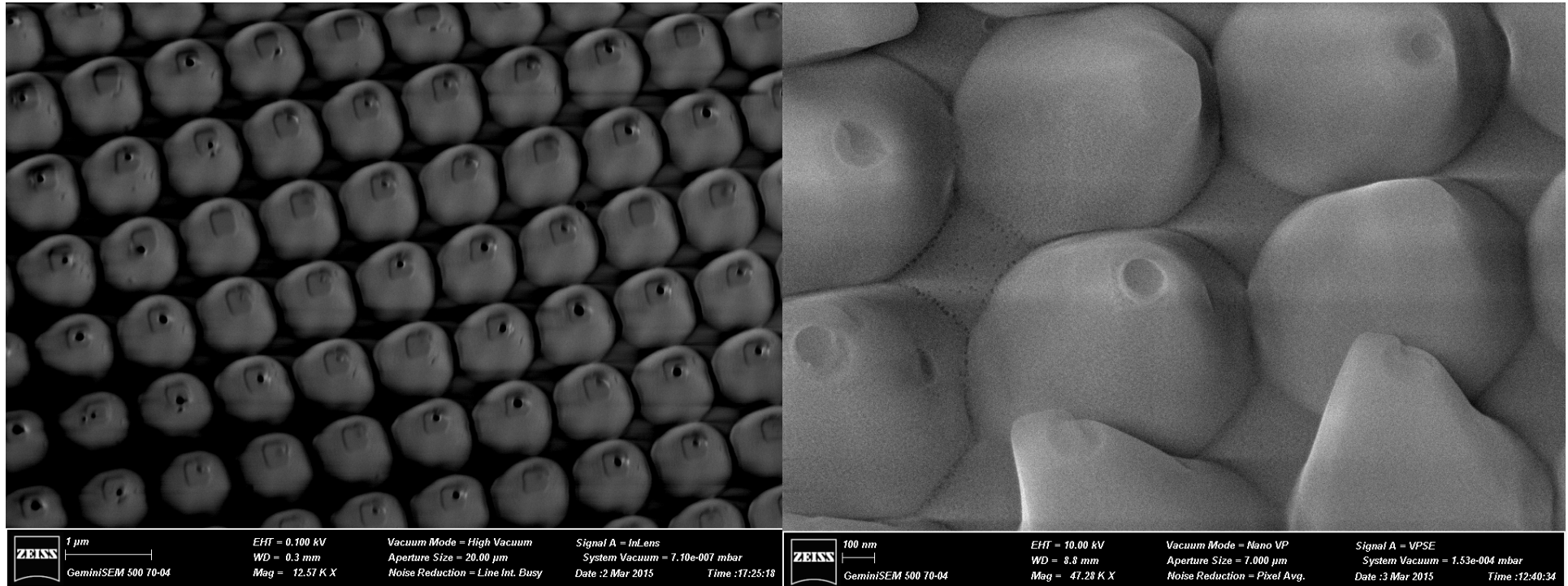
EBSD – EDAX HIKARI SUPER

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



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

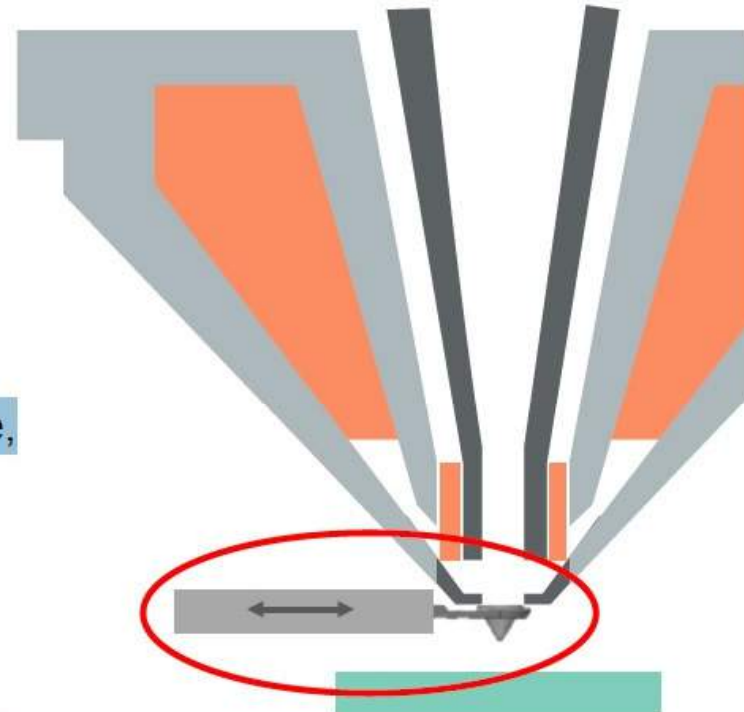


il y a des limites pour la basse tension
Et...pour l'EDS et l'EBSA on a besoin des tensions plus élevées

Different VP modes



- VP mode
 - Works the same as traditional VP mode
 - Pressure range 5 – 30 Pa
 - All but InLens/EsB detector
- NanoVP mode
 - EO is the same as high vacuum
 - 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 – 500 Pa 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

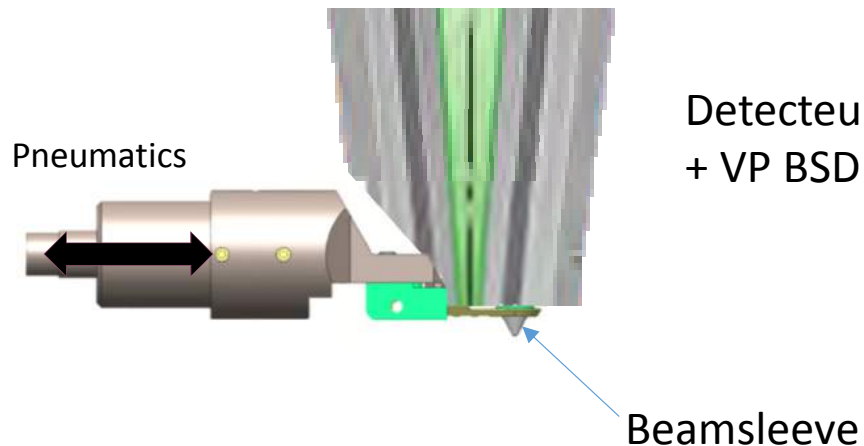
NanoVP – The Technology Behind

Source: Fang, ZEISS

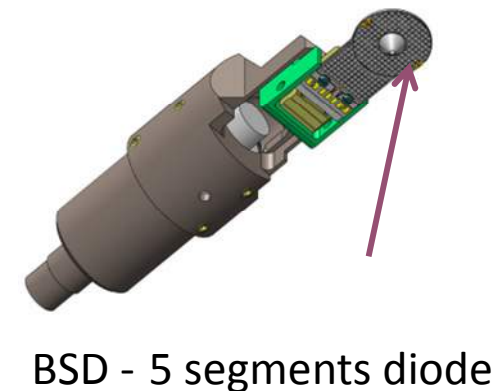
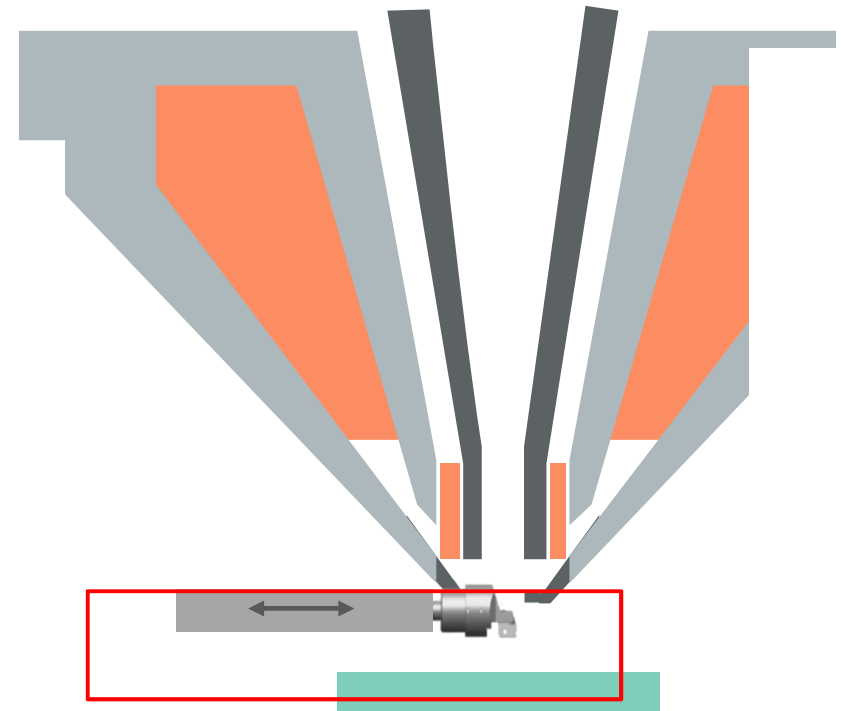
- In **NanoVP** mode a **differential pumping aperture** can be inserted **below the objective lens**.
- This greatly reduces the path length of the incident beam in the gas thus **reducing beam broadening**.
- **Best imaging performance**

up to 150 Pa, **full in lens detection**,
outstanding resolution (1.8 nm at 3kV, 30 Pa), reduced
field of view (350 μ m)

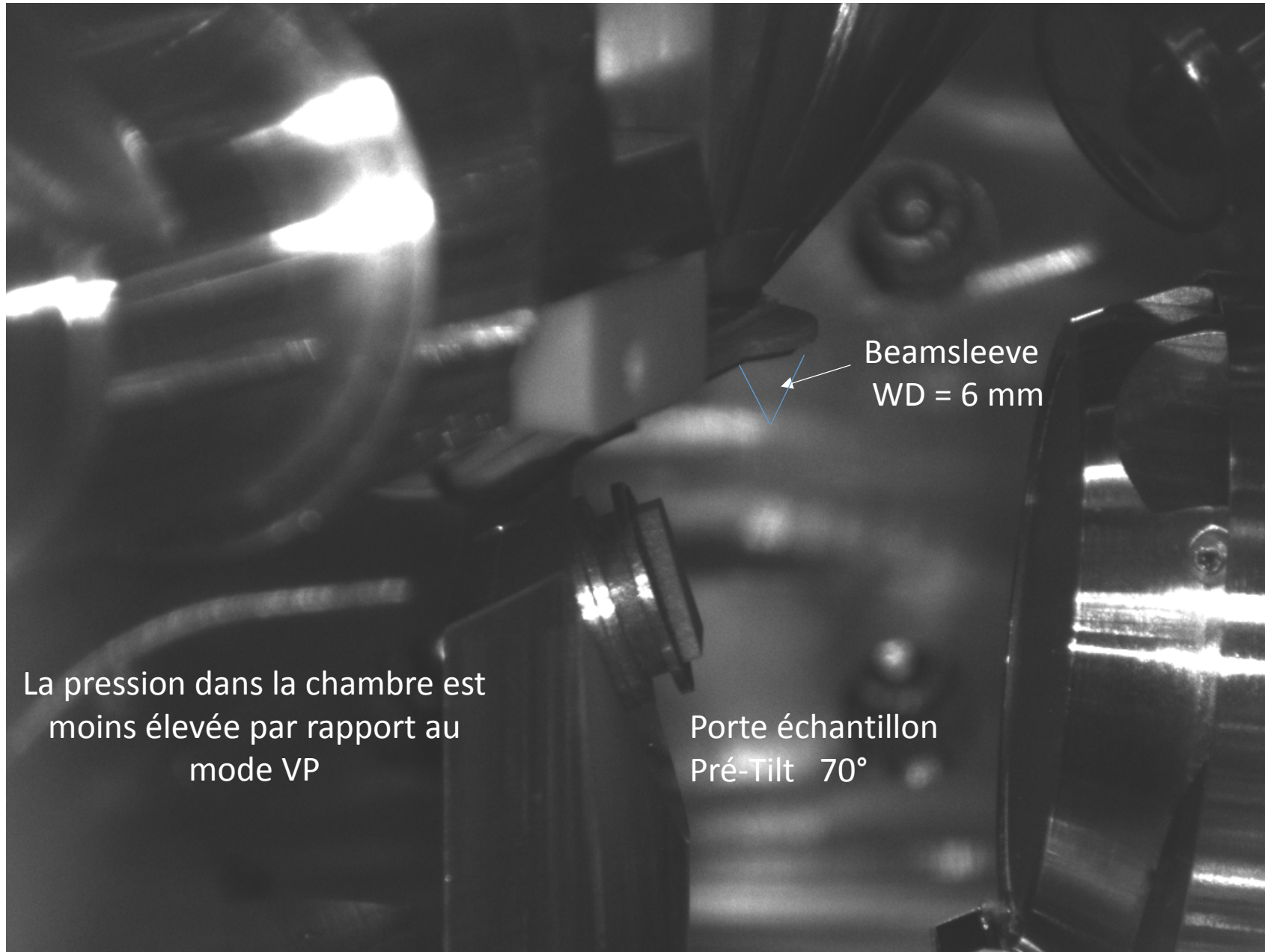
- In order to enable chamber **BSD detection in VP** mode a one channel BSD detector, which is integrated in the beam sleeve pneumatics, is available.

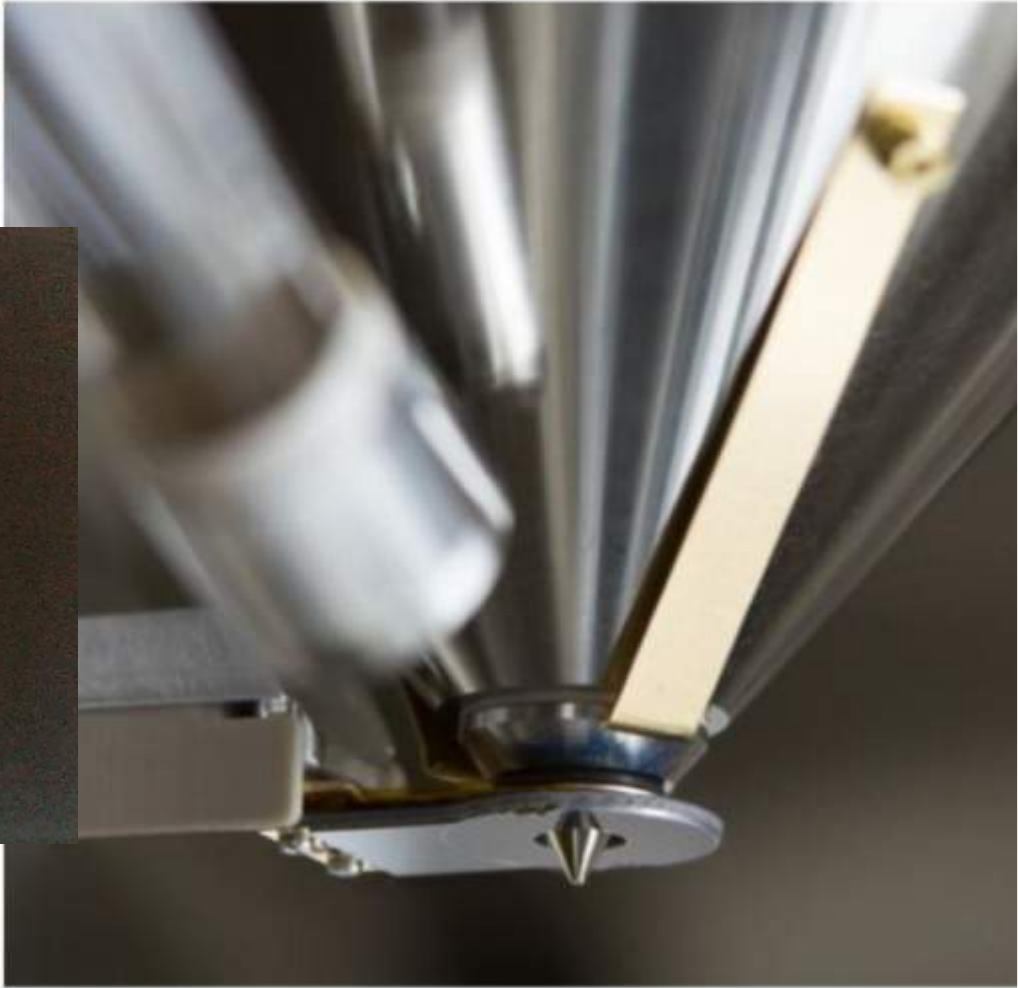


Detecteurs VPSE + INLENS
+ VP BSD



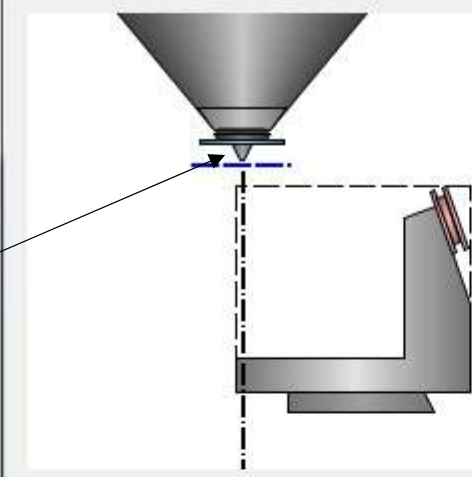
Mode NanoVP Pression entre 5 -150 Pa





Beamsleeve

Stage Navigation



Flip sideview

	Delta
X	66.460 mm
Y	42.842 mm
Z	14.394 mm
T	0.0 °
R	10.9 °

Idle

STOP

Options

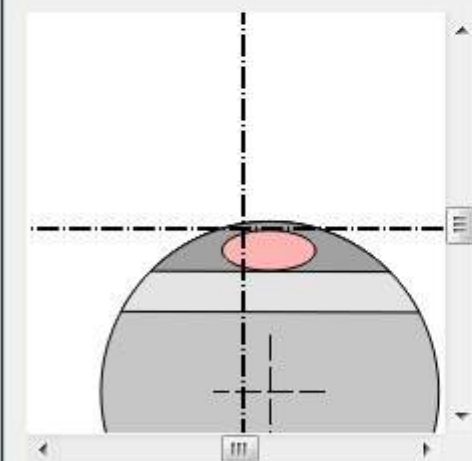
- Z move on Vent
- Track Z
- Protected Z
- Safe Z = 1.000 mm
- Safe Navigation

Sample Holder
Universal 70°


Specimen
Height 0 mm Diameter 12 mm

Settings...
More stage functionality...

Zoom view



Controlling VP



Chamber = 140 Pa

VP Target = 140 Pa

WD = 13.0 mm 6.9 mm

Go To HV Go To VP

Go to XVP mode Go to Nano VP

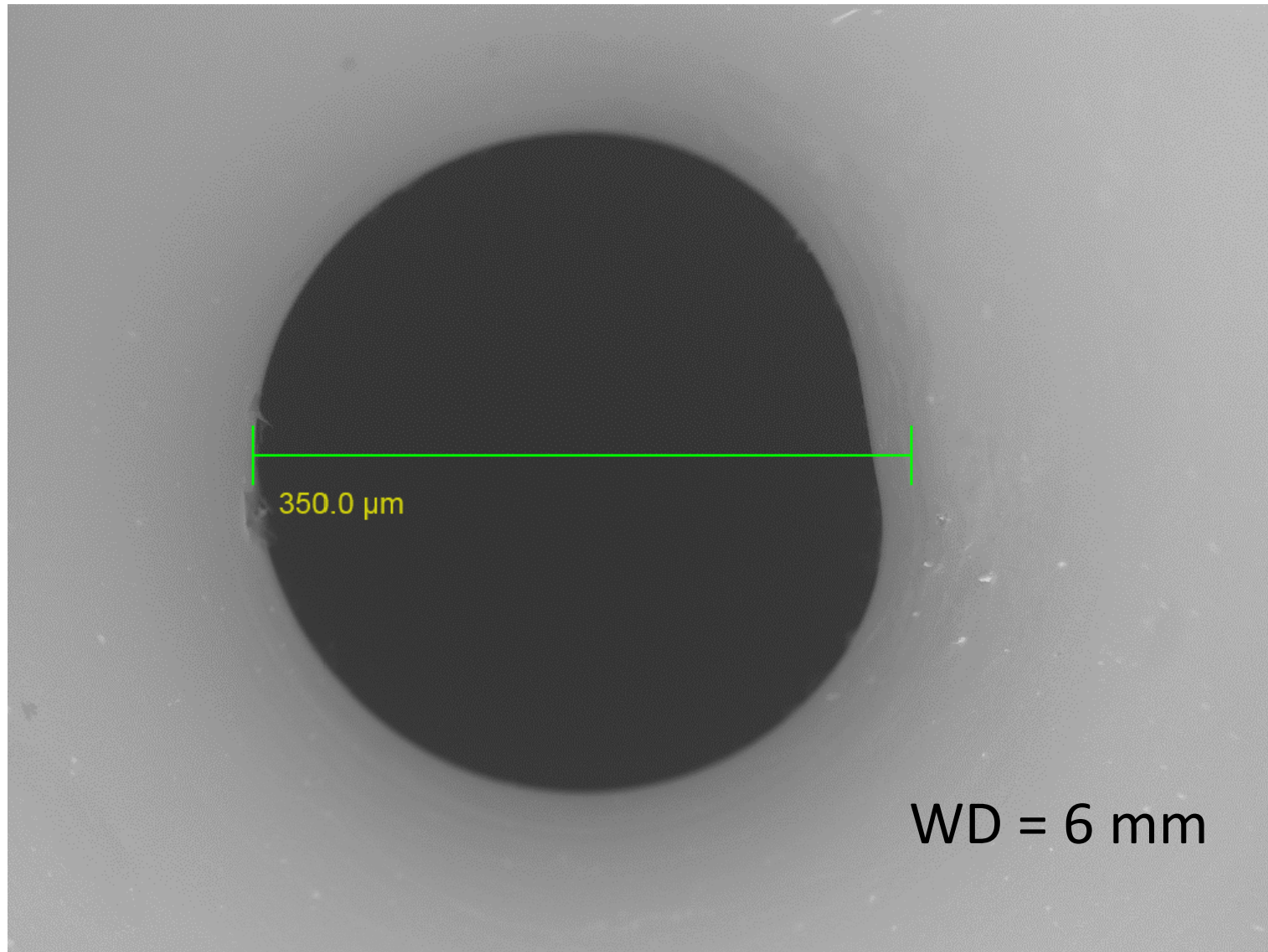
Collector Bias = 300 V

Beam sleeve Bias = 0 V

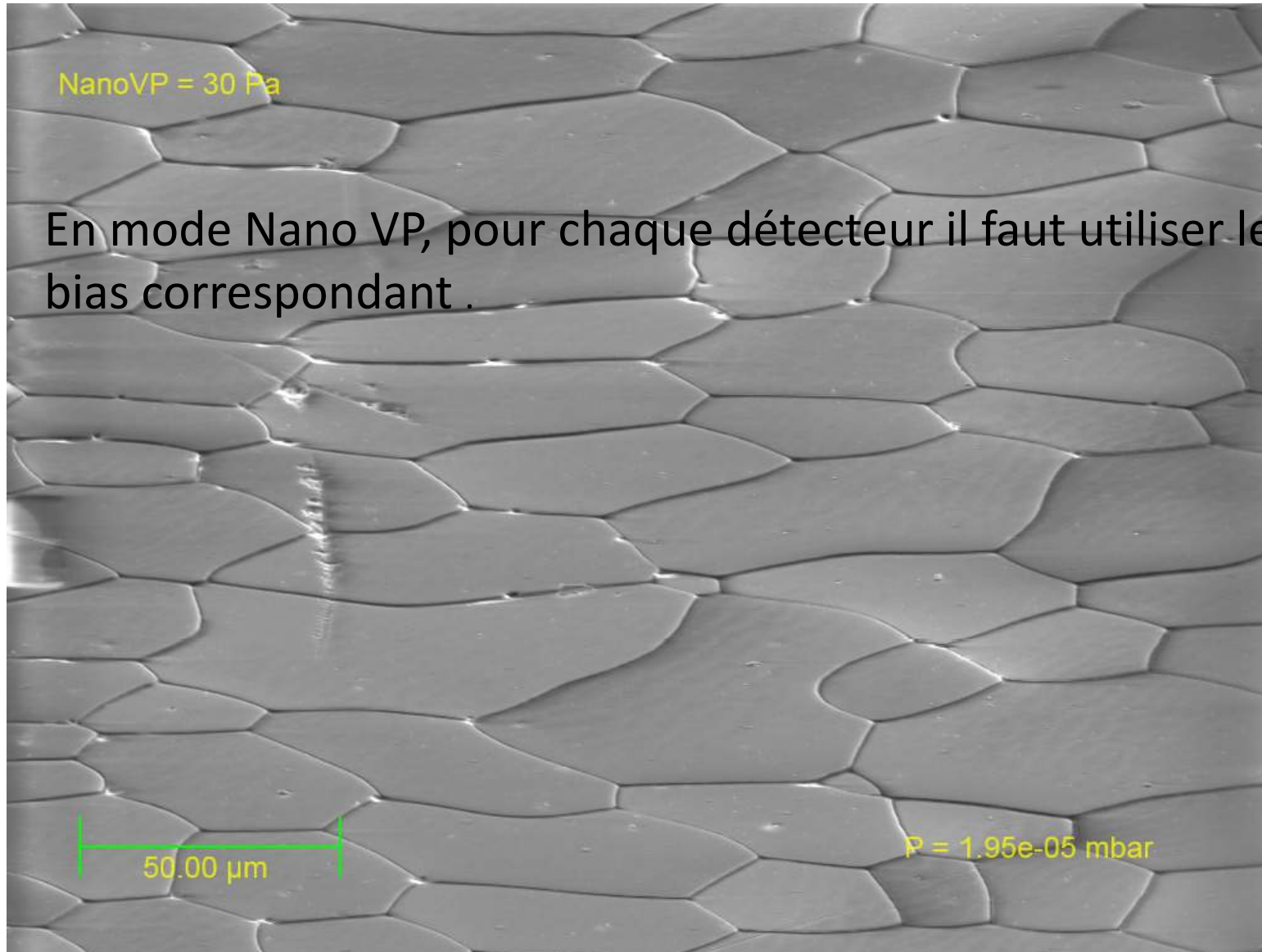
Beam sleeve Aperture = Beam sleeve 350 µm

Aperture Configuration... Aperture Alignment...

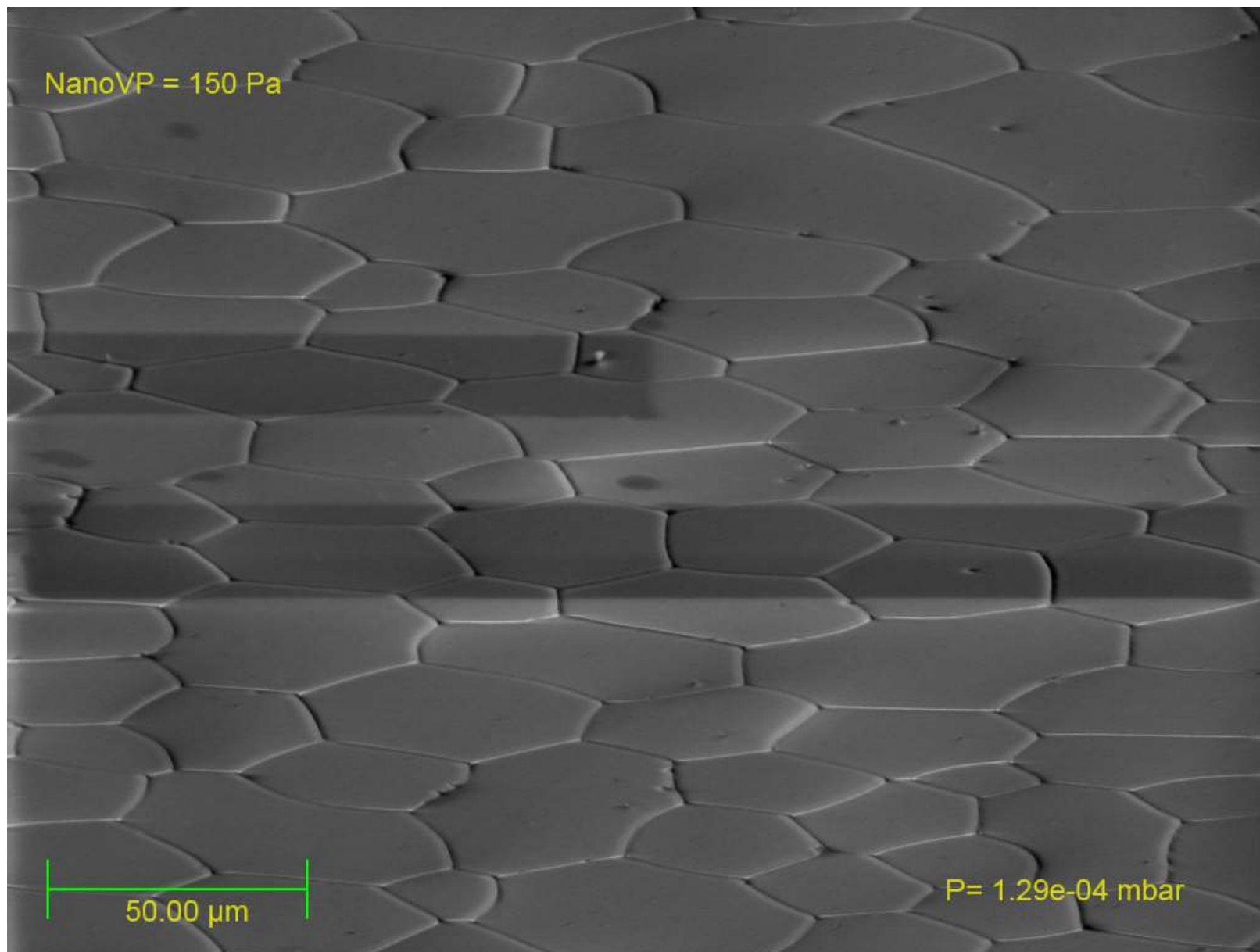
BEAMSLEEVE (CONE)



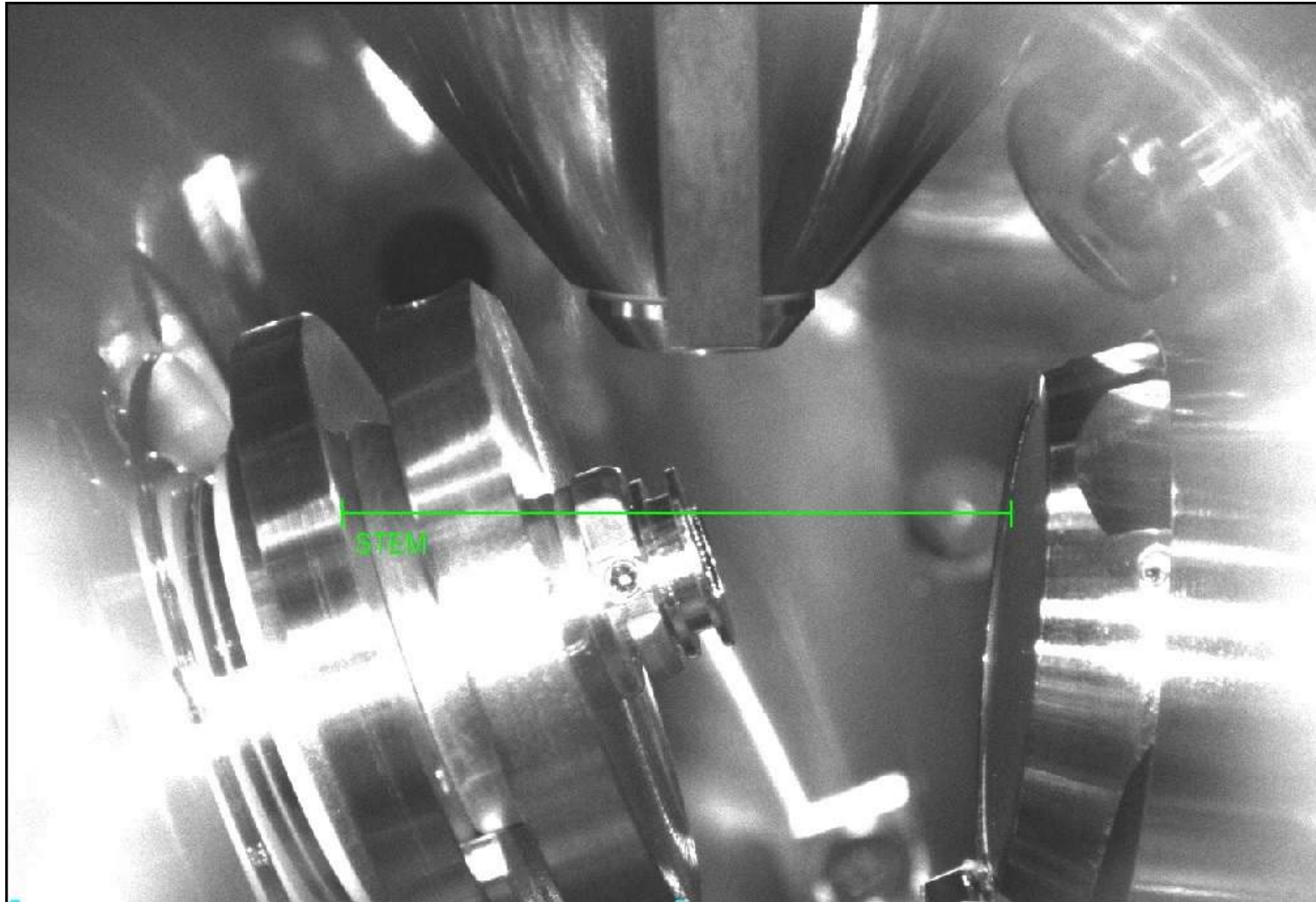
Images en Nano VP 20kV VPSE



Images en mode Nano VP 20kV VPSE



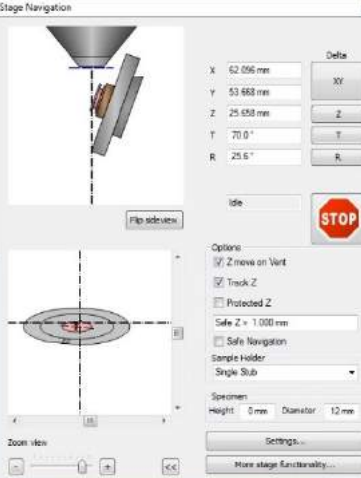
Mode VP Pression entre 5 – 30 Pa



	<p>100 μm</p>  <p>Gemini SEM 500 70-04</p>	<p>EHT = 15.00 kV WD = 12.8 mm Signal A = USB TV1</p>	<p>Mag = 114 X Pixel Size = 979.4 nm B = 51.8 % C = 91.2 %</p>	<p>Noise Red. = Pixel Avg. Scan Speed = 10 Cycle Time = 40.5 Secs</p>	<p>P = 7.44e-007 mbar Ap. Size = 20.00 μm</p>	<p>19 Jul 2016 17:29:15 CP2M</p>
---	--	---	--	---	--	--

Image en mode VP 20 kV VPSE

Stage Navigation



X: 62.056 mm
Y: 53.663 mm
Z: 25.458 mm
T: 70.0°
R: 25.6°

Delte
300
Z
T
R

idle

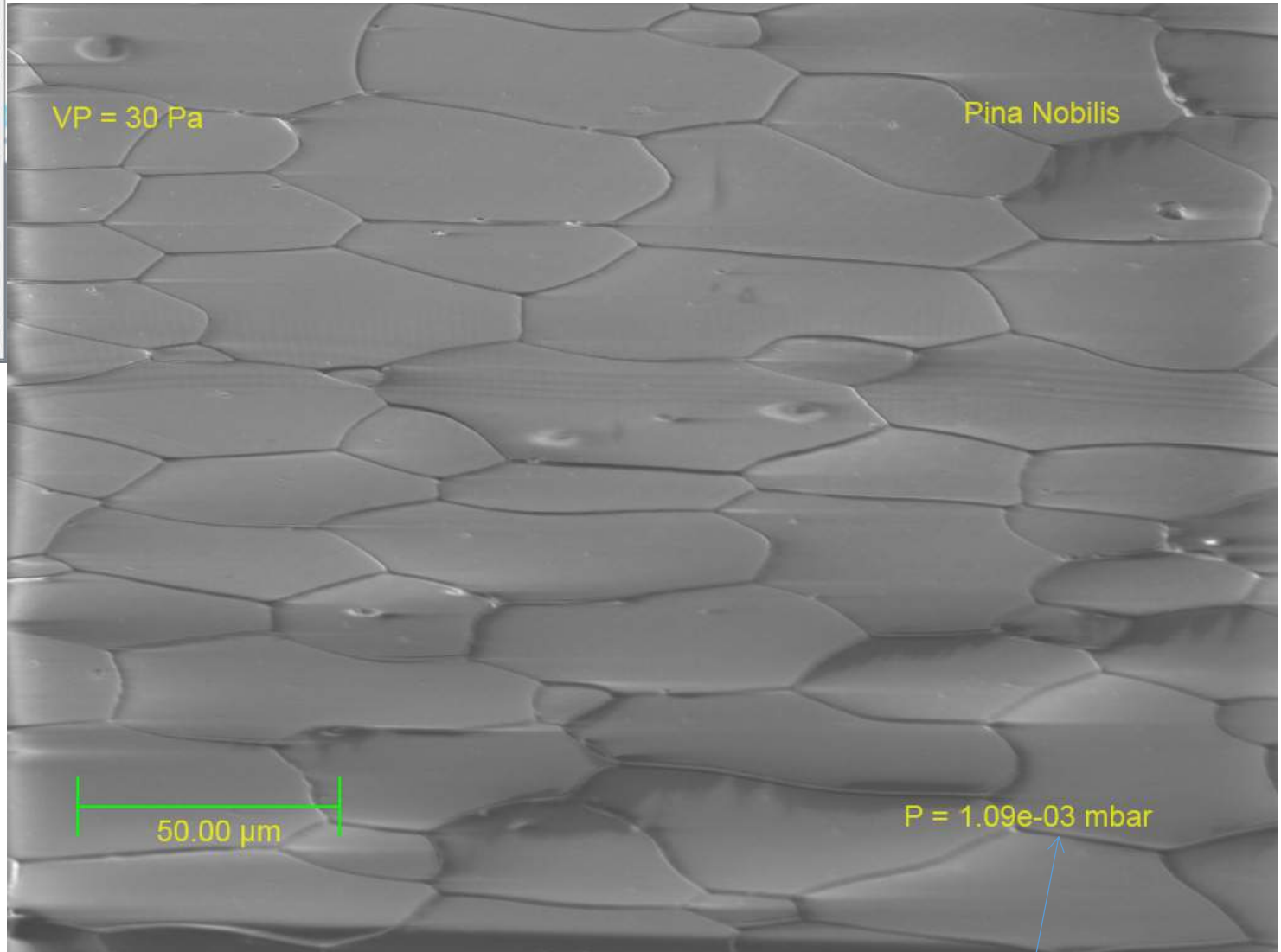
STOP

Options:
 Z move on Vert
 Track Z
 Protected Z
Safe Z = 1.000 mm
 Safe Navigation

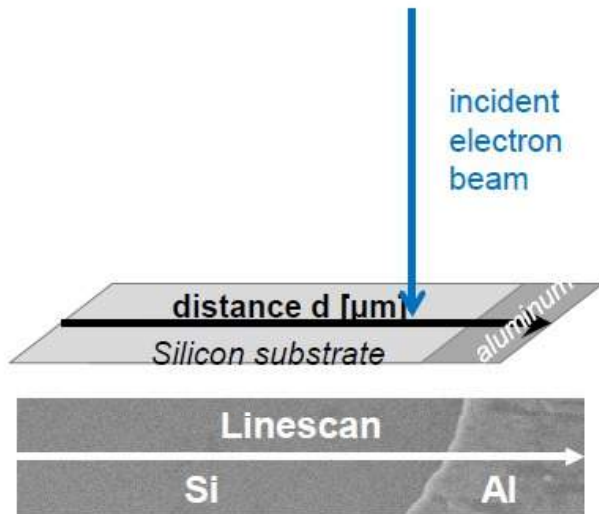
Sample Holder
Single Stub

Specimen
Height: 0 mm Diameter: 12 mm

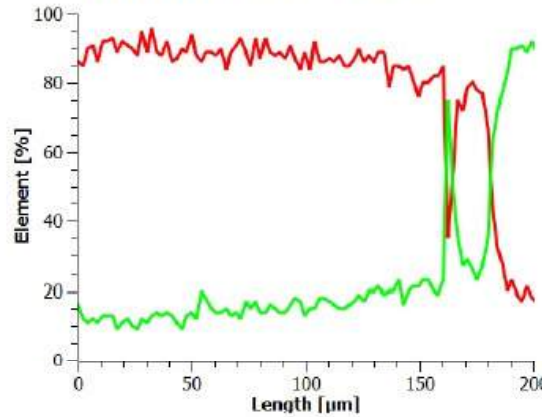
Settings...
More stage functionality...



EDS analysis in VP mode

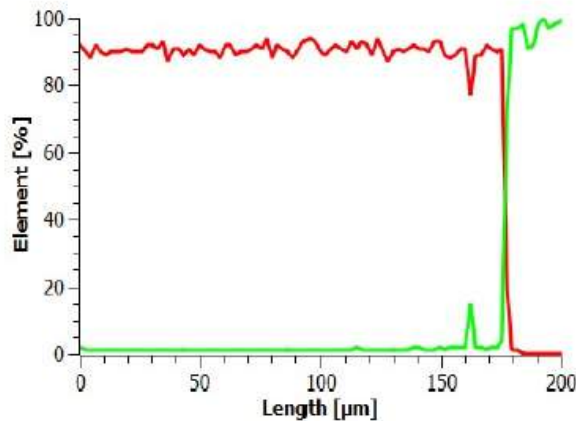


Normal VP 30 Pa

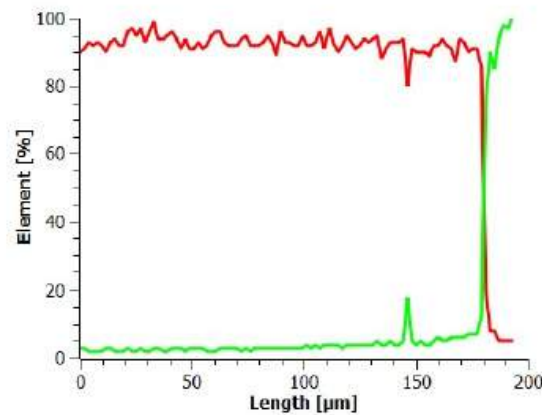


- Normal VP mode does not combine well with EDS analysis. The skirt effect creates a highly delocalized elemental signal.
- By controlling BGPL in NanoVP mode, spreading of EDS signal is largely reduced.
- High resolution EDS is possible on insulate samples with NanoVP.

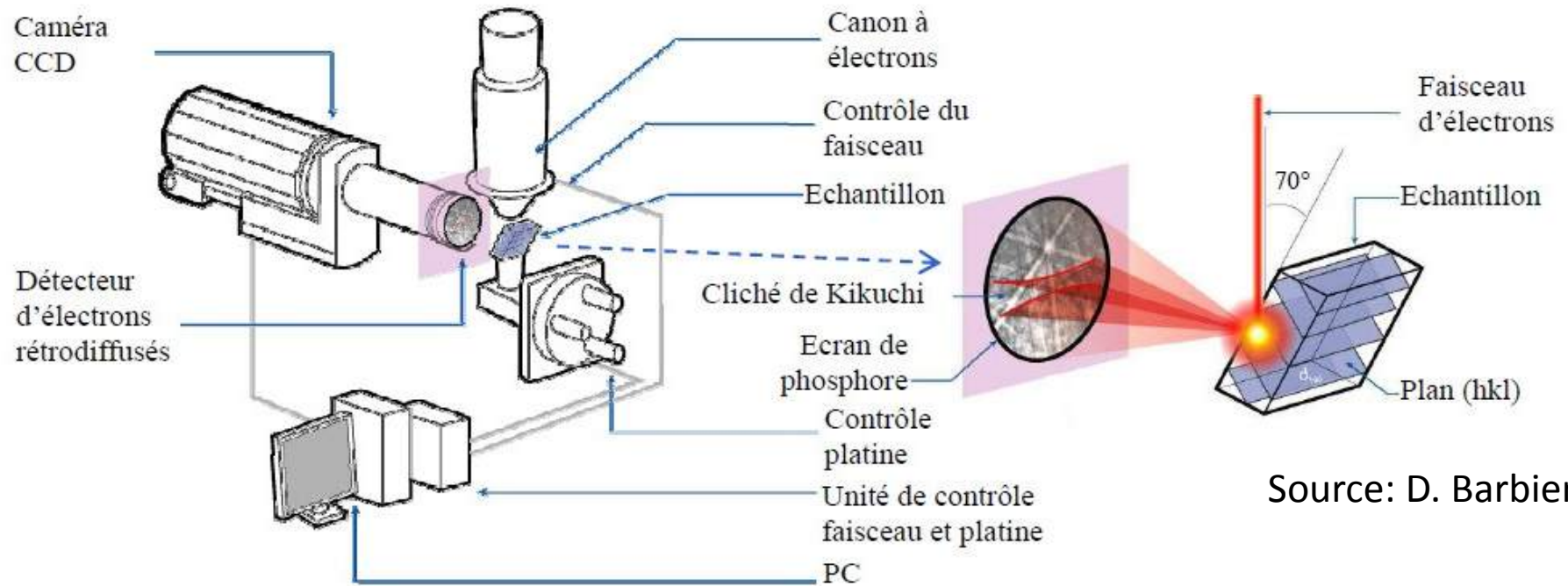
High Vacuum



NanoVP 30 Pa



EBSD: DISPOSITIF EXPERIMENTAL

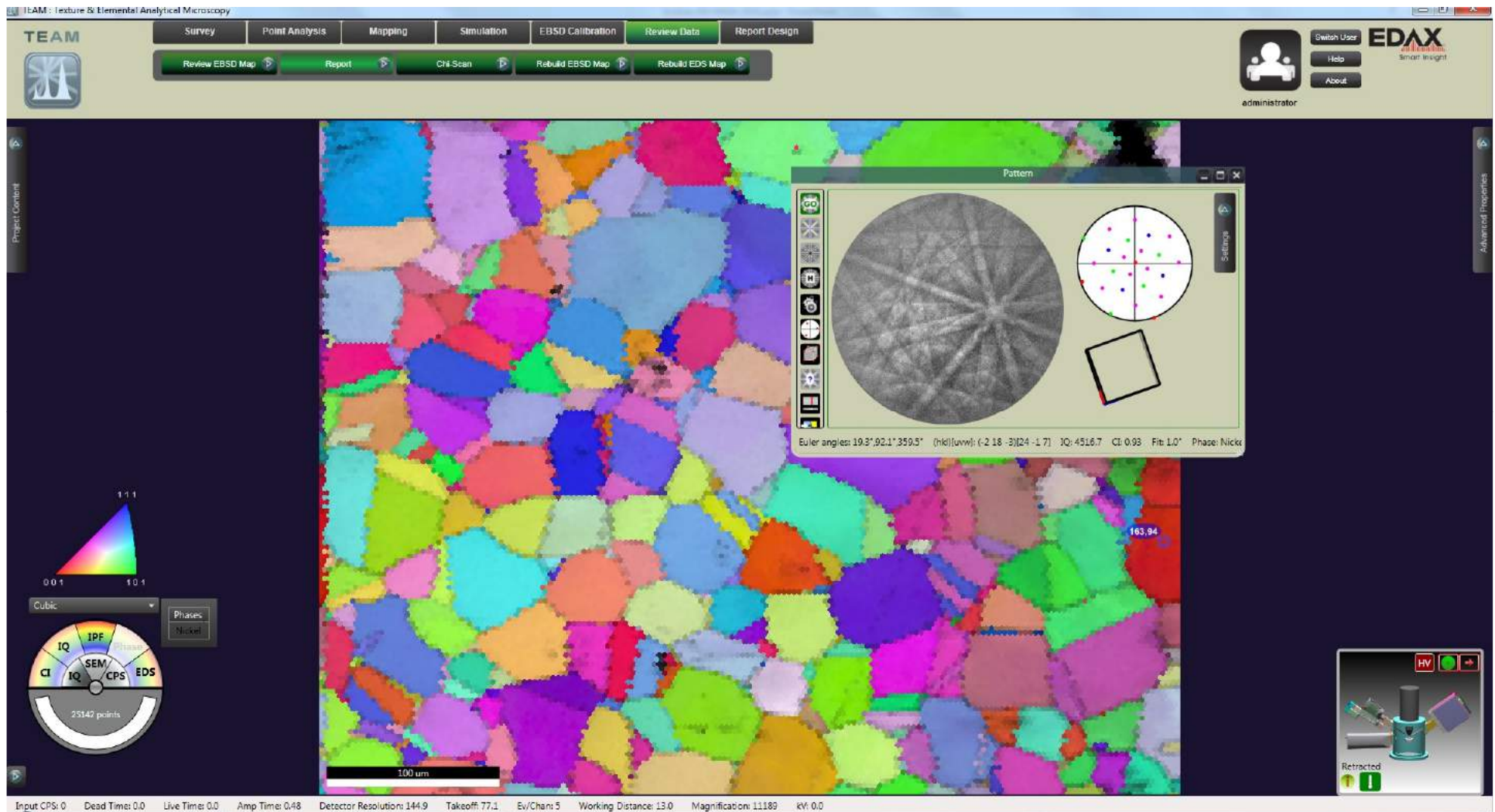


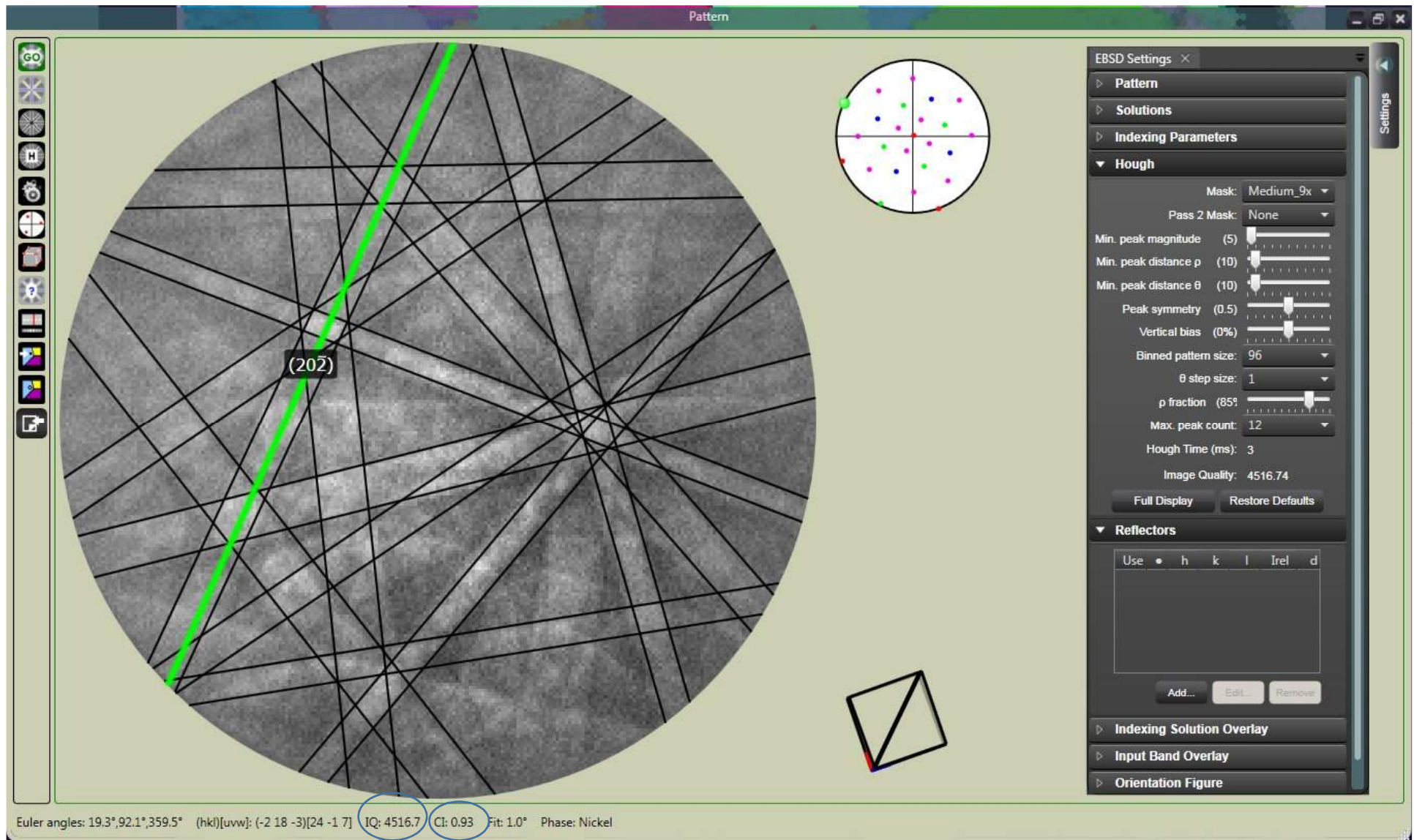
Source: D. Barbier

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° .

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

Importance du background



EDAX | Texture & Elemental Analytical Microscopy

TEAM | Survey | Point Analysis | Mapping | Simulation | EBSD Calibration | Review Data | Report Design

Review EBSD Map | Repeat | CH-Scan | Rebuild EBSD Map | Rebuild EDS Map

administrator | Switch User | Help | About | EDAX | Smart Insight

Image Processing Recipe Builder

Intensity Histogram Normalization

Name	Time (ms)
Intensity Histogram Normalization	1.0001

Remove | Load | Save

Brightness [50%] | Contrast [82%]

OK | Cancel

Euler angles: 106.5° 74.3° 301.0° (hkl)[uvw]: [-12 7 4][1 -4 10] IQ: 5240.2 CI: 0.93 Fit: 0.7° Phase: Nickel

100 um

ur EDS: 1 | Dead Time: 0.0 | Live Time: 0.0 | Amp Time: 0.42 | Detector Resolution: 164.0 | Takeoff: 77.1 | Fil/Chem: S | Working Distance: 13.0 | Magnification: 11180 | W: 0.0

EBSD Camera | 49.9 fps | 0.020 max | Capture | Presets | Camera | Image Processing | Recipe | Static Background Division | Dynamic Background Subtraction | Intensity Histogram Normalization | Image Processing Mode | Custom | Capture Background | Frames: 63 | Enable Distortion Correction | Camera Position | Image Area

Retracted

Calibration

TEAM : Texture & Elemental Analytical Microscopy

TEAM | Survey | Point Analysis | Mapping | Simulation | **EBSD Calibration** | Review Data | Report Design

Switch User | Help | About | administrator | EDAX | Smart Insight

Project Content

- Grobby 2018-05-30
- Grobby
- Grobby 2018-05-24
- Andrea 2017-03-09 test file
- NE standard
- Area 2
- OIM Map 1
- Area 1

Mag: 211 WD: 12.88
Size (µm): 498 x 390
Step (µm): 3.00 Points: 25142
3/9/2017 2:06 PM

Project | Sample | Profile | Import | Export | Print

EBSD Calibration

Pattern | Beam

Capture | Center beam and capture | Load | Horizontal Sync: 8

Camera Angles

Azimuthal: 0 | Elevation: 5

Index | Hide Overlay | Cb: 0.10 Fit: 1.59

Record

Export to OIM | Replace | OK

EBSD Calibration Edit

WD	X-Star	Y-Star	Z-Star	X-Star Error	Y-Star Error	Z-Star Error
3.00	49.88	99.02	71.74	0.66	0.47	0.55
5.00	49.98	92.83	71.82	0.46	0.43	0.11
7.00	51.87	86.64	71.31	1.53	0.40	0.18
9.00	51.07	78.75	70.60	0.83	1.33	0.06
11.00	49.56	73.47	69.83	0.58	0.46	0.12
13.00	49.24	67.48	69.88	0.80	0.29	0.50
15.00	49.72	61.99	68.72	0.22	0.38	0.07

Cancel | OK

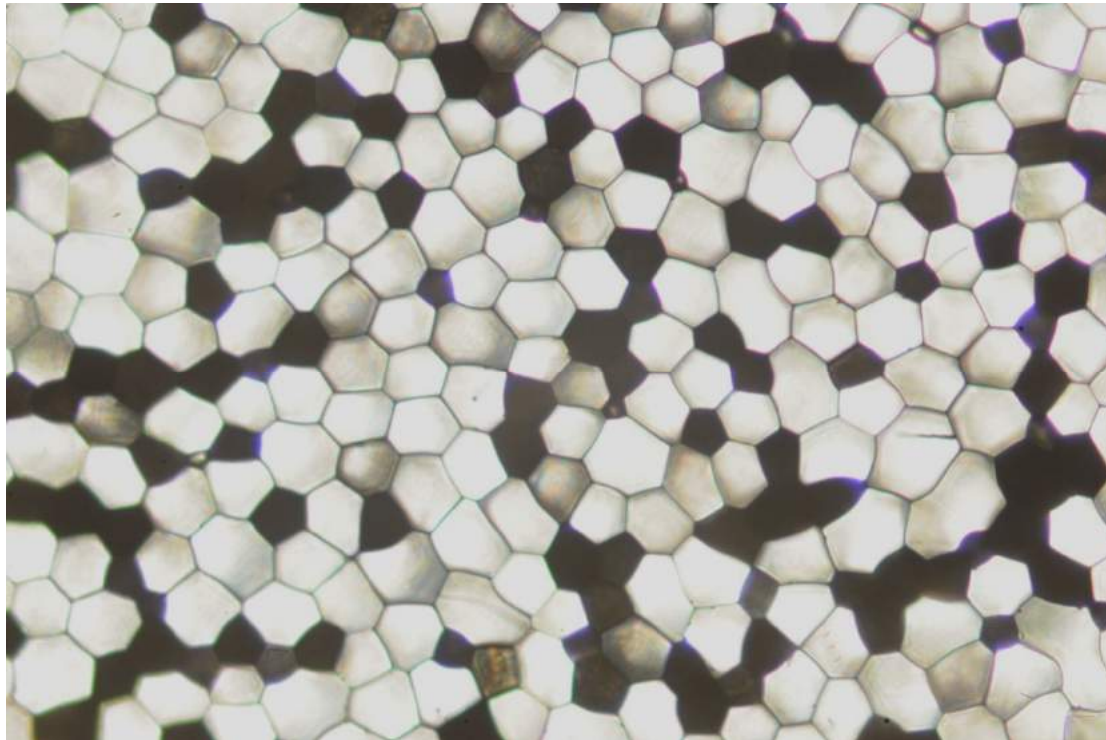
Camera Setup | Reference Frame | Dis

Input CPS: 1 | Dead Time: 0.0 | Live Time: 0.0 | Amp Time: 0.48 | Detector Resolution: 144.0 | Takeoff: 77.1 | Ev/Chan: 5 | Working Distance: 13.0 | Magnification: 11189 | Kv: 0.0

12:22 PM

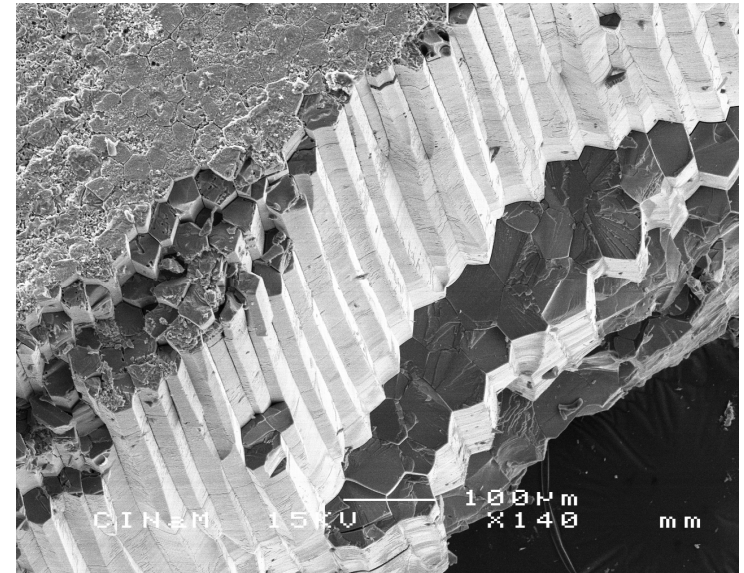
Echantillons – Biomatériaux



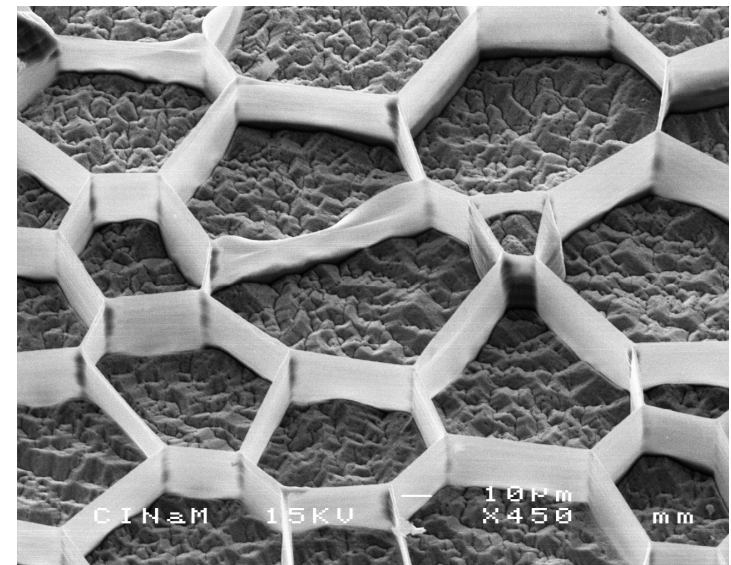


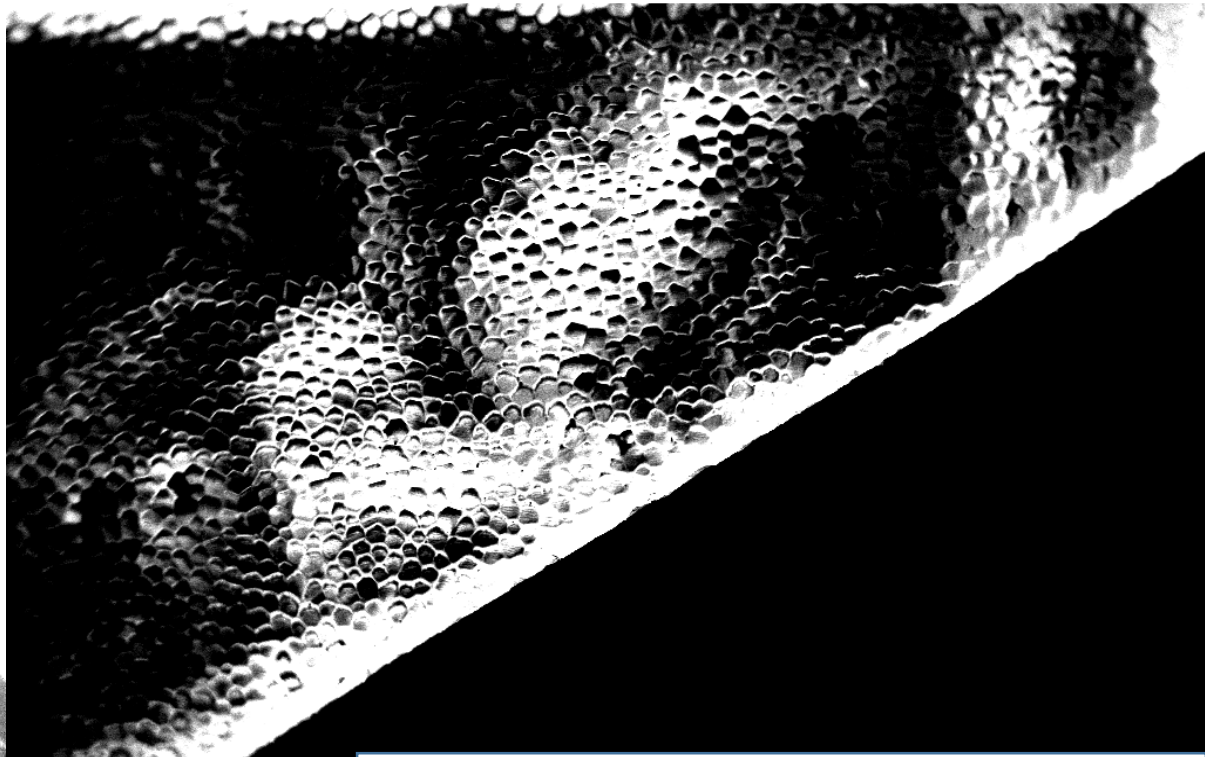
Echantillon 1 - Microscope polarisant
Calcite

Et pour les biomatériaux?

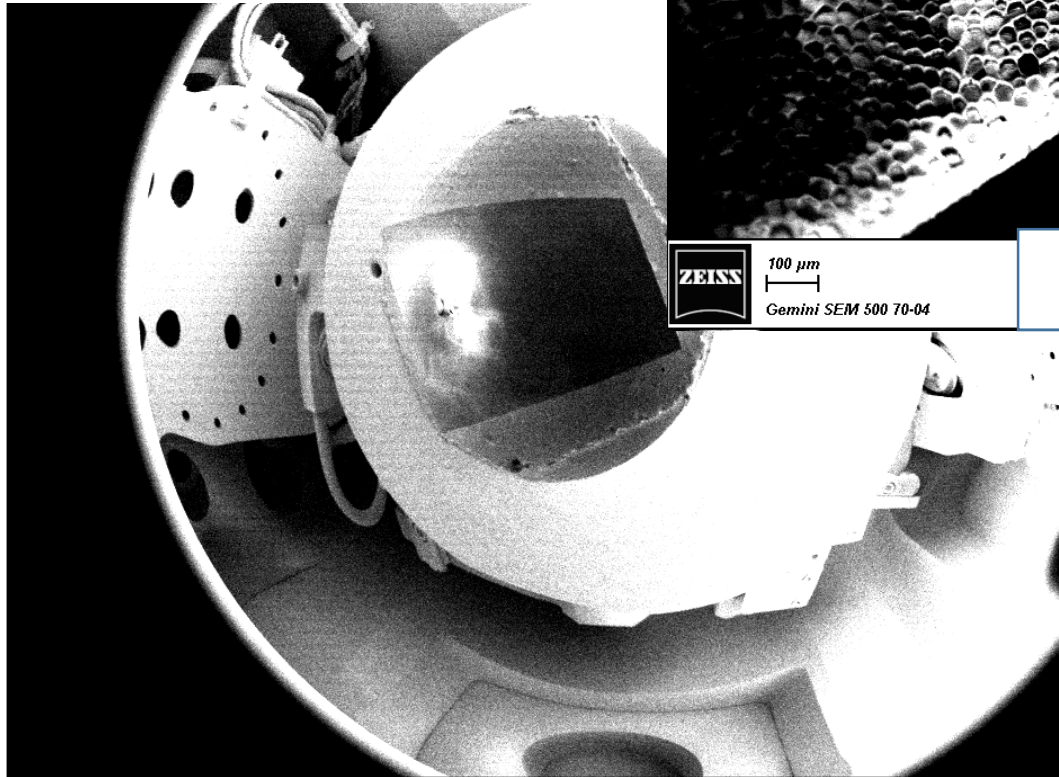


Application des modes VP

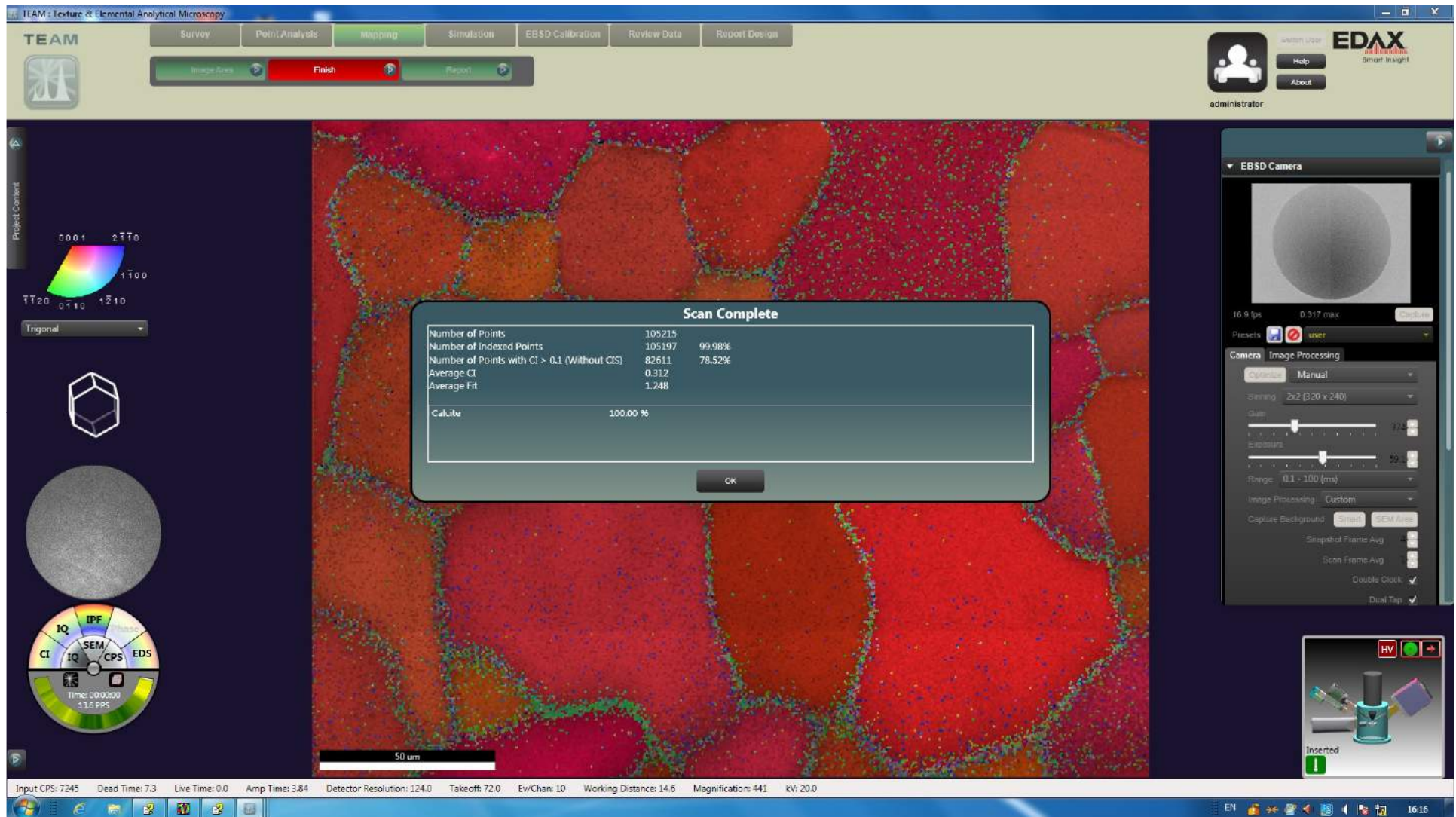




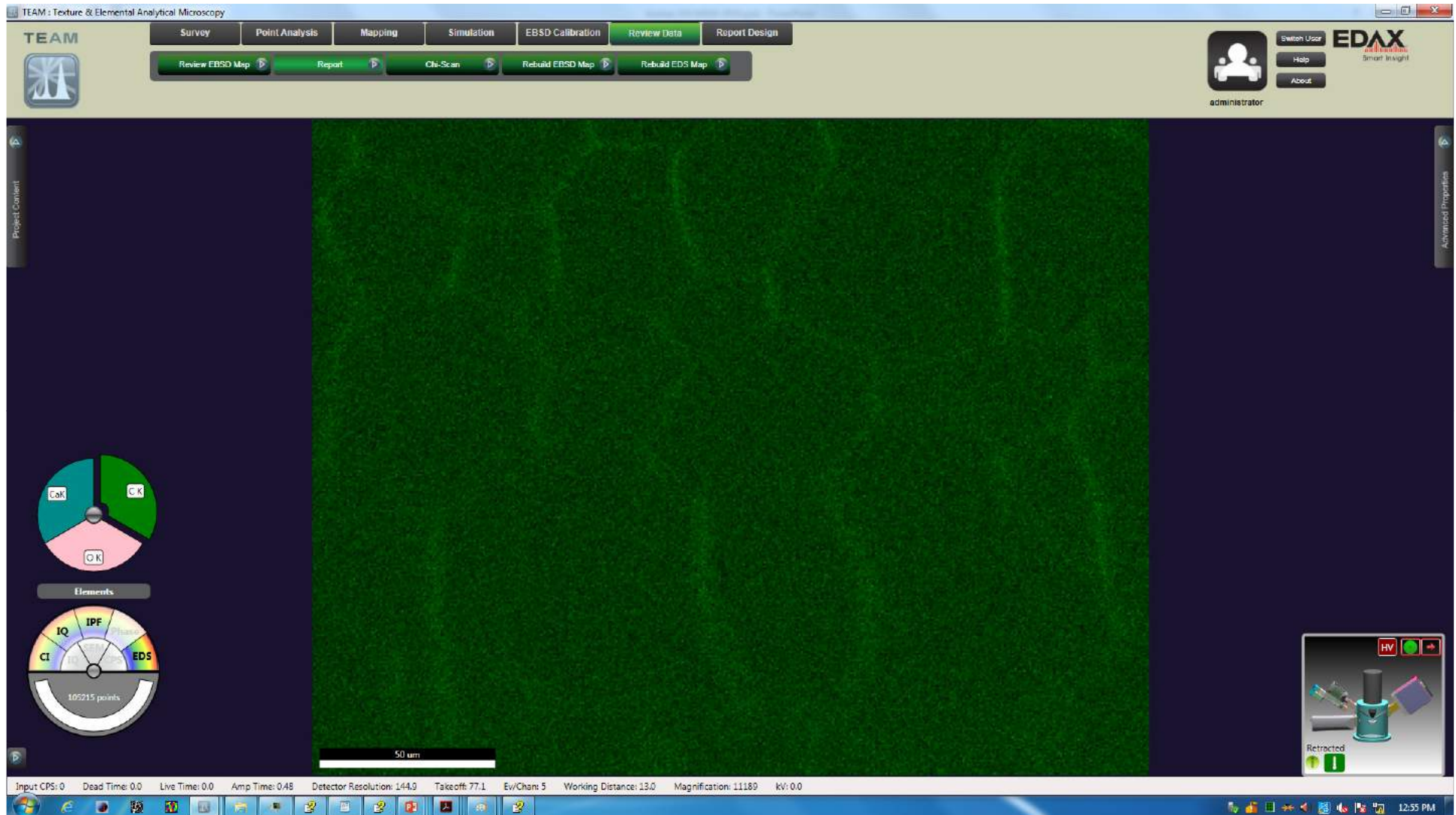
Charging at 10 kV



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



Background: Static Background Subtraction – Intensity Histogram Normalization



EDS pour localiser la matière organique

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

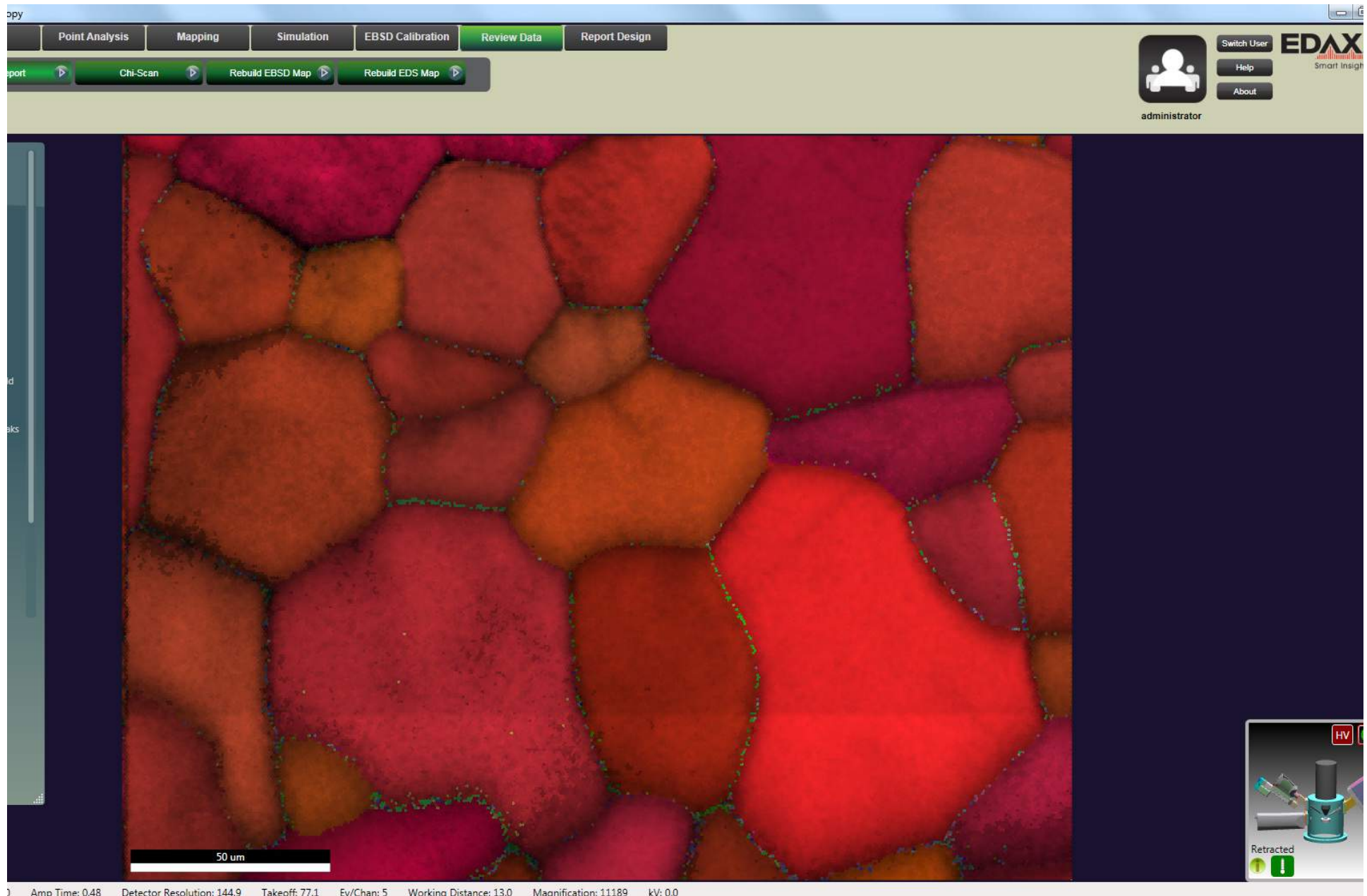
The screenshot displays the TEAM software interface for EBSD analysis. The main window shows a large EBSD map with a 50 μm scale bar. A 'Pattern' window is open, showing a selected EBSD pattern and its corresponding crystallographic data: Euler angles: 111.4°, 169.0°, 110.6°; (hkl)[uvw]: (0 -2 2 -16)[11 -7 -4 0]; IQ: 1148.9; CI: 0.52; Fit: 1.1°; Phase: Calc. A 'Hough Display' window is also open, showing a Hough transform plot with colored lines and a 'Close' button. The Hough Display window includes the following settings:

- Mask: Medium_Sx9
- Phase 2 Mask: None
- Binned pattern size: 96
- θ step size: 1
- Max. peak count: 12
- Hough Time (ms): 3
- Min. peak magnitude: (5)
- Min. peak distance p: (10)
- Min. peak distance B: (10)
- Peak asymmetry: (0.5)
- Vertical bias: (0%)
- p fraction: (0.5%)
- Image Quality: 1148.88

The bottom status bar provides technical parameters: Input CPS: 1, Dead Time: 0.0, Live Time: 0.0, Amp Time: 0.48, Detector Resolution: 144.0, Takeoff: 77.1, Ev/Chan: 5, Working Distance: 13.0, Magnification: 11189, Kv: 0.0. The system tray shows the time as 12:45 PM.

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

Application de NPAR - Neighbor Pattern Averaging and Re-indexing



NPAR - Neighbor Pattern Averaging and Re-indexing

TEAM Help V4.5

Hide Back Forward Print Options

Contents Index Search

Type in the word(s) to search for:

ncar

List Topics Display

Select topic: Found: 2

Title	Location	Rank
What's new in TEAM V4.3	TEAM Help V4.5	1
What's new in TEAM V4.4	TEAM Help V4.5	2

What's new in TEAM V4.3

Jackson^u
^aEDAX, 392 East 12300 South, Suite H, Draper UT 84020, USA
^bEDAX, 91 McKee Drive, Mahwah NJ 07430, USA
^cCarnegie Mellon University, Department of Material Science and Engineering, 5000 Forbes Avenue, Pittsburgh, PA 15213, USA
^dBlueQuartz 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.

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

(Fig 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).

Ultramicroscopy 159(2015)81–94

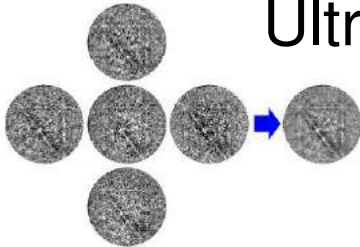


Fig. 1. Schematic of the averaging of the pattern at a given point with the patterns of the neighboring points.

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.

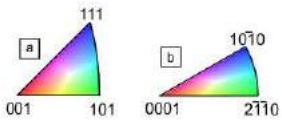


Fig. 2. Orientation map color keys for the (a) nickel alloy and steel samples and (b) the deformed magnesium sample.

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

Search previous results
 Match similar words
 Search titles only

5:17 PM

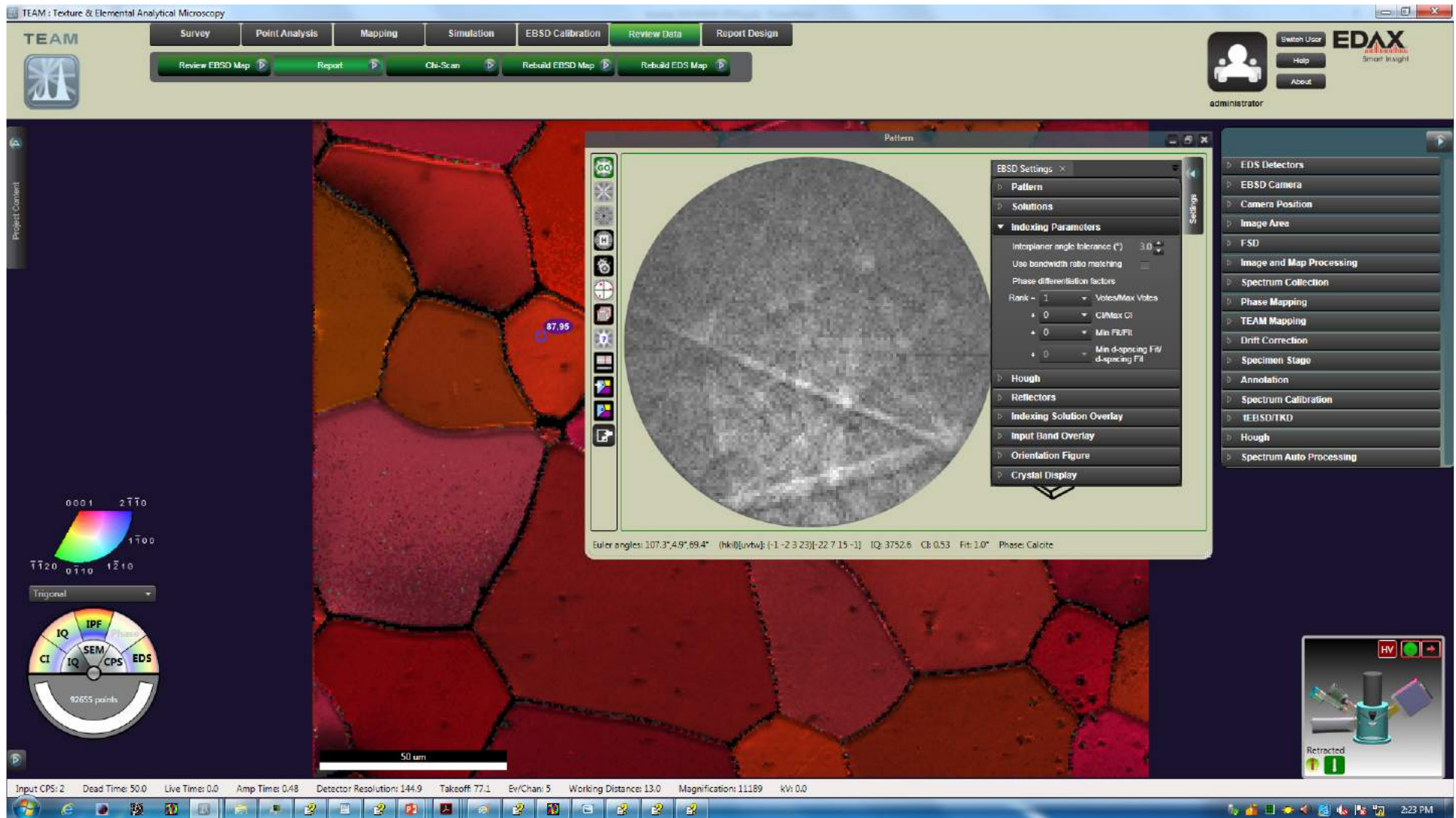
Utilisation du programme OIM

The screenshot displays the TEAM (Texture & Elemental Analytical Microscopy) software interface. The main window shows a large circular OIM image with a central bright spot. To the right, a 'Point Info' window is open, displaying a phase map and a legend. The legend includes the following information:

- (Highlighted Points)/(Total Number of Points) = 0.000
- (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]
- Plane Normal (Pole):
- Calculate
- Legend | Preview | Lattice | Interactive | Notes

The interface also features a top menu bar with options like Survey, Point Analysis, Mapping, Simulation, EBSD Calibration, Review Data, and Report Design. A bottom status bar provides technical details such as Input CPS: 1, Dead Time: 0.0, Live Time: 0.0, Amp Time: 0.48, Detector Resolution: 144.0, Takeoff: 77.1, Ev/Chan: 5, Working Distance: 13.0, Magnification: 11189, and Kv: 0.0. The system tray shows the time as 12:56 PM.

Mode nano VP (avec beamsleeve) - 30 Pa



CI et IQ ↑

Mode Nano VP (avec beamsleeve) - 60 Pa

Static Background Division
Dynamic Background Subtraction
Intensity Histogram Normalization

The screenshot displays the EDAX software interface. At the top, there is a navigation bar with tabs for Survey, Point Analysis, Mapping, Simulation, EBSD Calibration, Review Data, and Report Design. Below this, there are buttons for 'Snap Area', 'Finish', and 'Report'. The main window shows a large EBSD map of a polycrystalline material with a green overlay. A 'Scan Complete' dialog box is open in the center, displaying the following data:

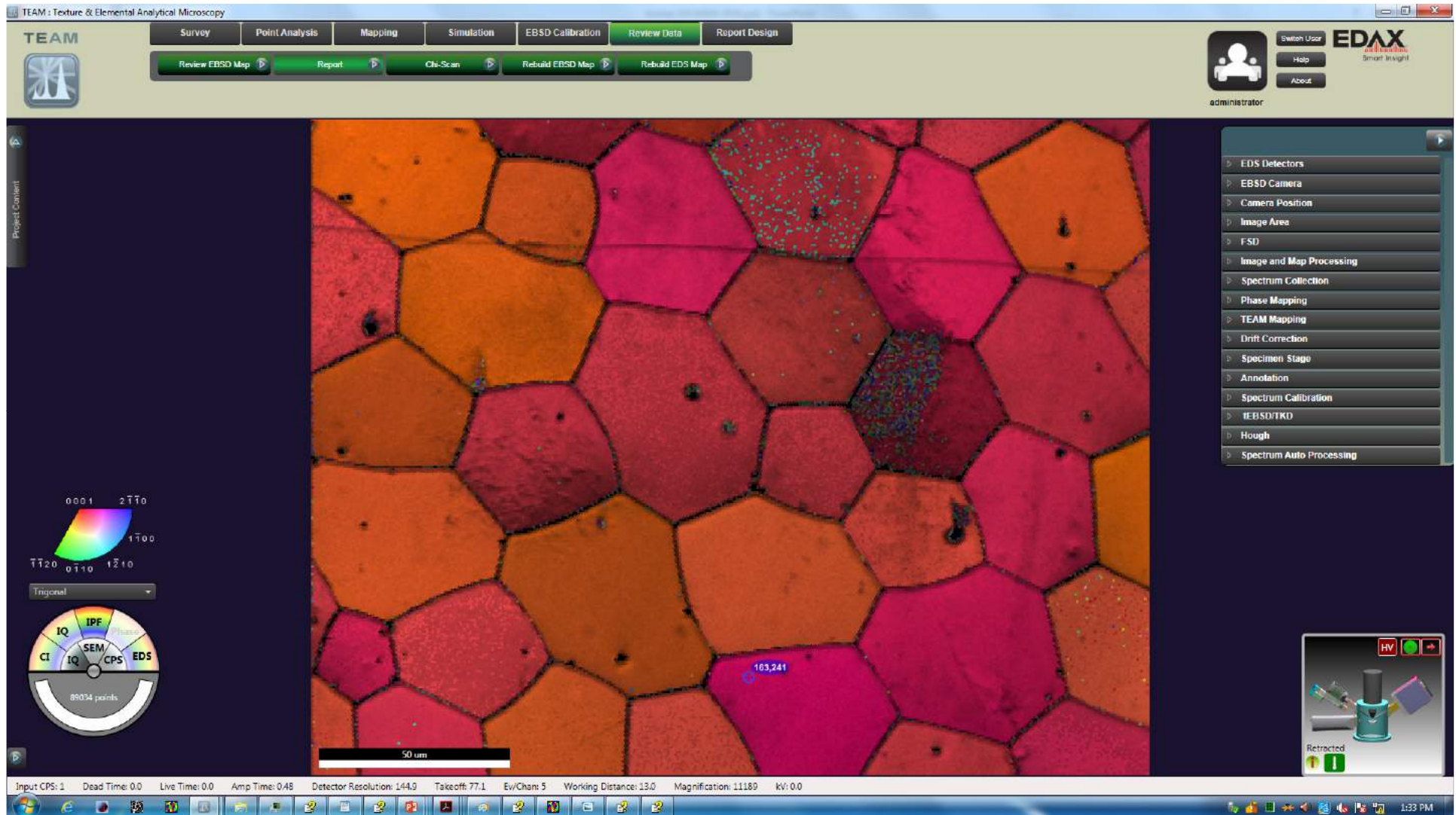
Scan Complete	
Number of Points	89034
Number of Indexed Points	88054 98.9%
Number of Points with CI > 0.1 (Without CIS)	84435 94.83%
Average CI	0.529
Average Fit	1.03
Calcite	100.00 %

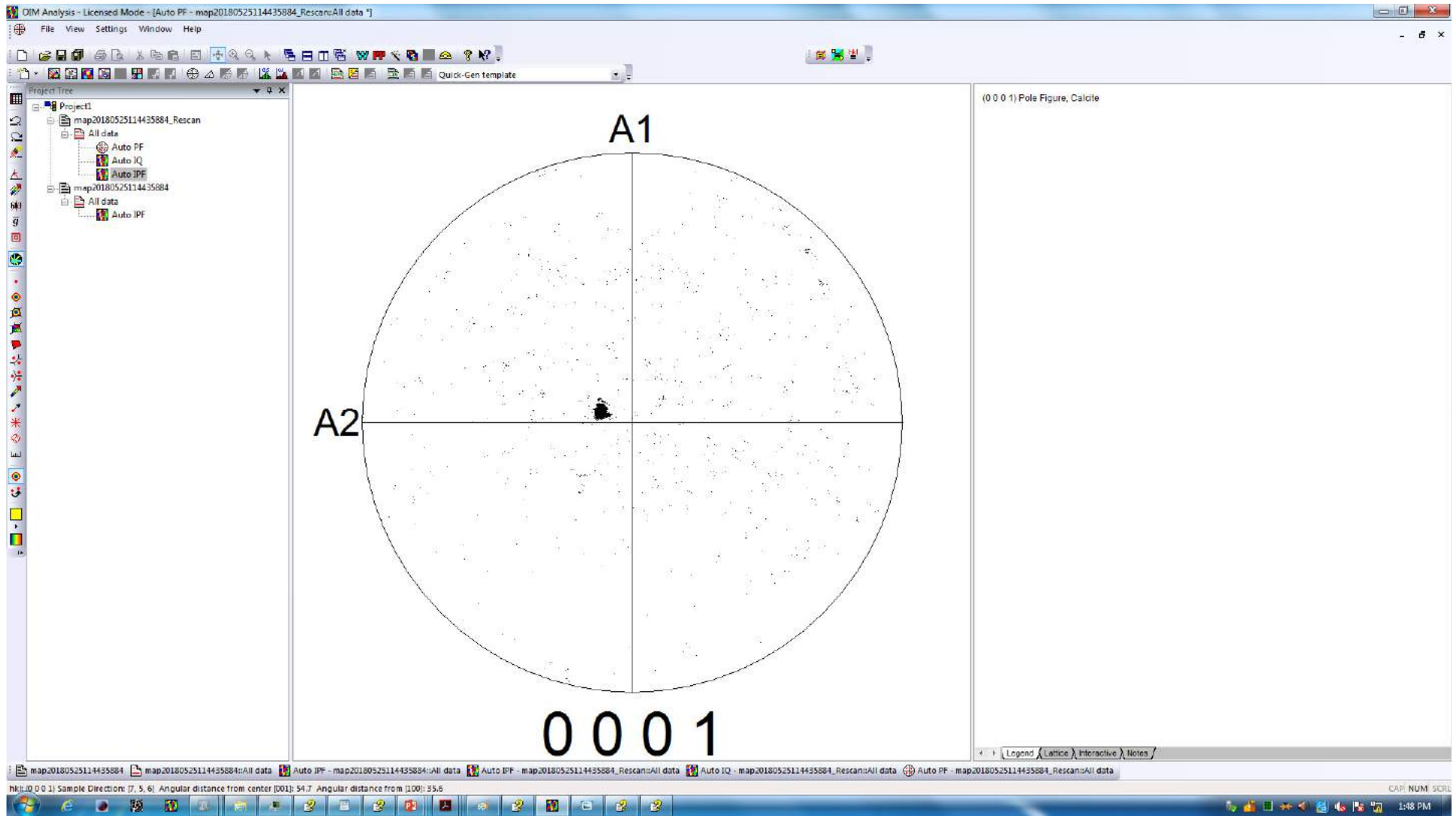
On the right side, the 'EBSO Camera' settings panel is visible, showing a camera icon, a 'Presets' dropdown, and 'Camera' and 'Image Processing' tabs. The 'Image Processing' tab is active, showing settings for 'Smoothing' (4x4 (160 x 120)), 'Gain', 'Exposure', 'Range' (0.1 - 100 (ms)), 'Image Processing' (Custom), 'Capture Background' (Off), 'Snapshot Frame Avg', 'Scan Frame Avg', 'Double Click', and 'Dual Tap'. A '50 um' scale bar is located at the bottom of the main image area.

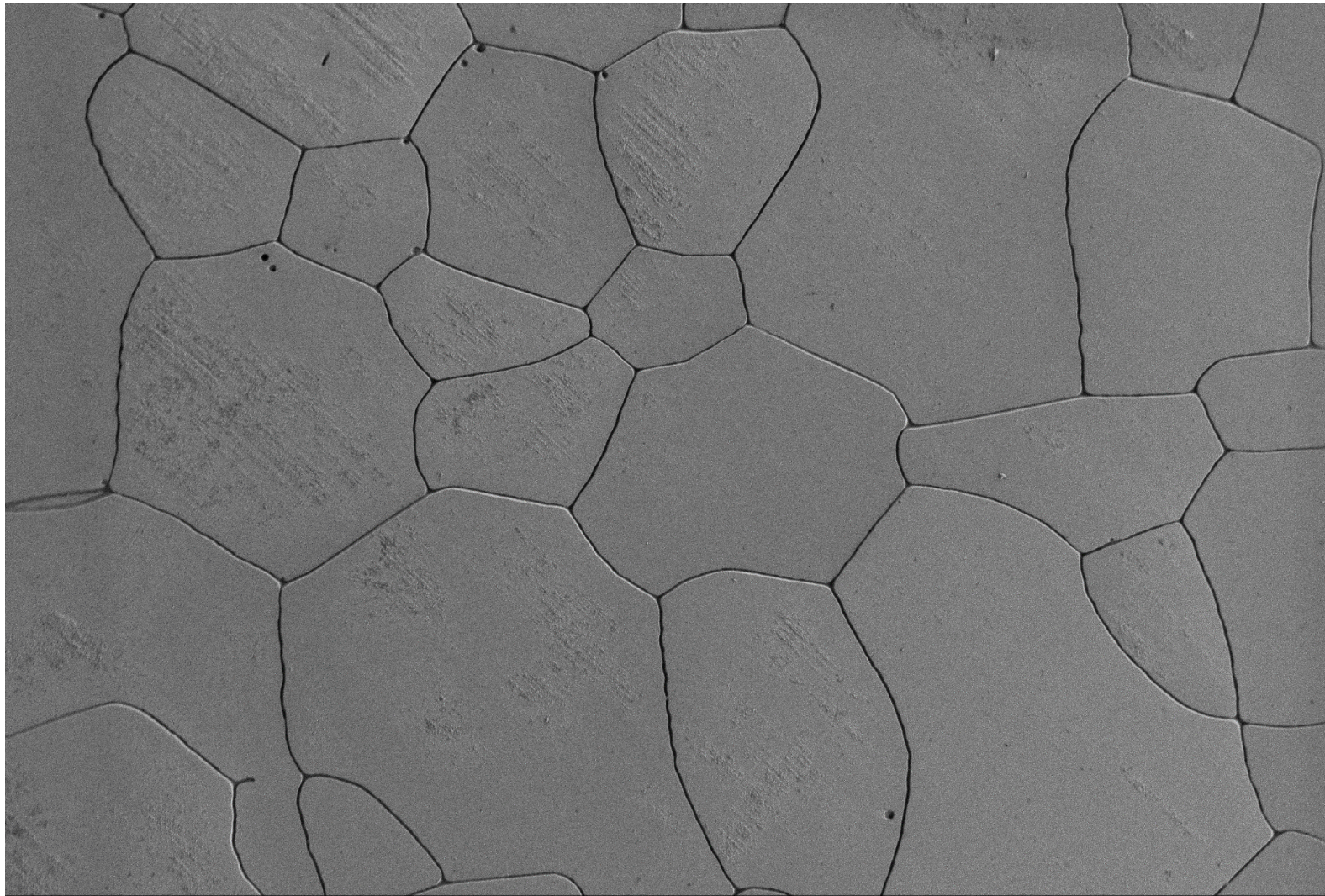
L'avantage du mode Nano VP est de pouvoir avoir des résultats similaires avec des pressions plus élevées


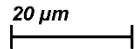
CI VERT

Mode Nano VP (avec beamsleeve) - 60 Pa







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

Mode Nano VP (avec beamsleeve) - 60 Pa

TEAM : Texture & Elemental Analytical Microscopy

Survey | Point Analysis | Mapping | Simulation | EBSD Calibration | Review Data | Report Design

Review EBSD Map | Report | Chi-Scan | Rebuild EBSD Map | Rebuild EDS Map

Switch User | Help | About | administrator

EDAX
ANALYTICAL
Smart In Sight

Project Content

EBSD Settings: **summons**

Indexing Parameters

- Interplanar angle tolerance (°) 3.0
- Use bandwidth ratio matching
- Phase differentiation factors
- Rank = 1 | Votes/Max Votes
- + 0 | ChiMax Cl
- + 0 | Min FitFit
- + 0 | Min d-spacing Fit/d-spacing Fit

Length

50 µm

Euler angles: 330.6°, 15.8°, 240.9° (hkl)[uvw]: (-2 -1 3 11) [-13 12 1 -2] - IQ: 2898.5 - CI: 0.65 - Fit: 0.9° - Phase: Calcite

Input CPS: 0 | Dead Time: 0.0 | Live Time: 0.0 | Amp Time: 0.48 | Detector Resolution: 144.0 | Takeoff: 77.1 | Ev/Chan: 5 | Working Distance: 13.0 | Magnification: 11189 | Kv: 0.0

1:36 PM

Mode Nano VP (avec beamsleeve) - 60 Pa

The screenshot displays the TEAM software interface for EBSD analysis. The main window shows a circular EBSD pattern with numerous indexed spots, each labeled with Miller indices such as [111], [200], [112], [210], [311], [400], [511], [600], [711], [800], [911], [1000], [1100], [1200], [1300], [1400], [1500], [1600], [1700], [1800], [1900], [2000], [2100], [2200], [2300], [2400], [2500], [2600], [2700], [2800], [2900], [3000], [3100], [3200], [3300], [3400], [3500], [3600], [3700], [3800], [3900], [4000], [4100], [4200], [4300], [4400], [4500], [4600], [4700], [4800], [4900], [5000], [5100], [5200], [5300], [5400], [5500], [5600], [5700], [5800], [5900], [6000], [6100], [6200], [6300], [6400], [6500], [6600], [6700], [6800], [6900], [7000], [7100], [7200], [7300], [7400], [7500], [7600], [7700], [7800], [7900], [8000], [8100], [8200], [8300], [8400], [8500], [8600], [8700], [8800], [8900], [9000], [9100], [9200], [9300], [9400], [9500], [9600], [9700], [9800], [9900], [10000].

On the right side, the 'EBSD Settings' panel is open, showing the 'Indexing Parameters' section. The 'Interplanar angle tolerance (°)' is set to 3.0. The 'Use bandwidth ratio matching' checkbox is checked. The 'Phase differentiation factor' is set to Rank = 1. The 'Voles/Max Voles' is set to 0. The 'Cl/Max Cl' is set to 0. The 'Min FWHM' is set to 0. The 'Min d-spacing FWHM/d-spacing FWHM' is set to 0.

At the bottom of the main window, the Euler angles are displayed: Euler angles: 330.4°, 15.7°, 241.2°. The phase is identified as Calcite. The fit quality is shown as Cl: 0.05, Fit: 1.0°.

The bottom status bar shows: Input: CPS: 2, Dead Time: 50.0, Live Time: 0.0, Amp Time: 0.48, Detector Resolution: 144.9, Takeoff: 77.1, Ev/Chan: 5, Working Distance: 13.0, Magnification: 11189, kV: 0.0.

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

The screenshot displays the TEAM software interface for EBSD analysis. The main window shows a circular EBSD map with a selected point. A 'Point Info' dialog is open, showing 'F8 Image Processing' options and a 3D crystal model. A 'Color Coded Map' window shows an inverse pole figure (IPF) map with a legend. The bottom status bar shows technical parameters like Euler angles and phase information.

Point Info Dialog:

- F8 Image Processing
 - NPAR: Neighbor Pattern Averaging
 - Dataset Background Correction
 - Partition Background Correction
 - Dynamic Background Correction
 - Auto Brightness and Contrast
 - Advanced...

Color Coded Map Legend:

- (Highlighted Points)/(Total Number of Points) = 0.000
- (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]
- Plane Normal (Pole)
- Calcite

Status Bar:

Euler angles: 330.4°, 15.7°, 241.2° (hkl)[uvw]: (-2 -1 3 11) [-13 12 1 -2] IQ: 2898.5 CI: 0.05 Fit: 1.0° Phase: Calcite

Post – processing OIM

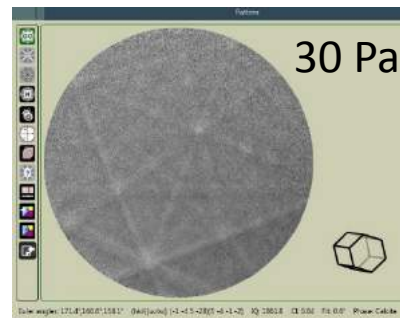
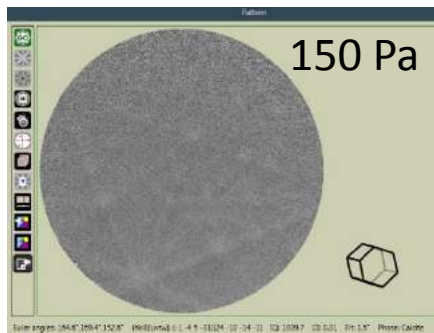
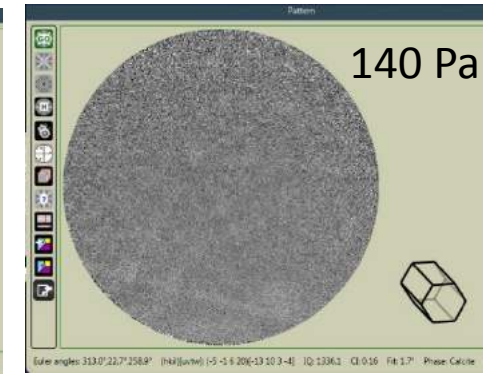
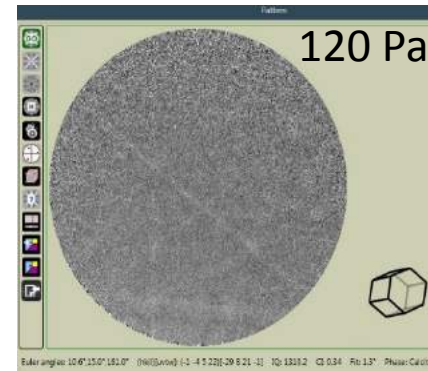
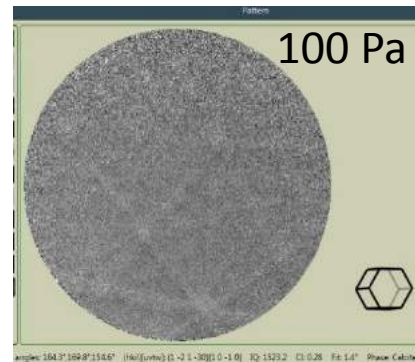
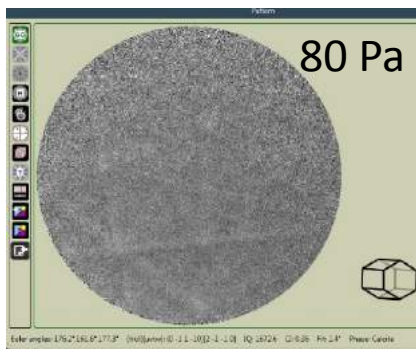
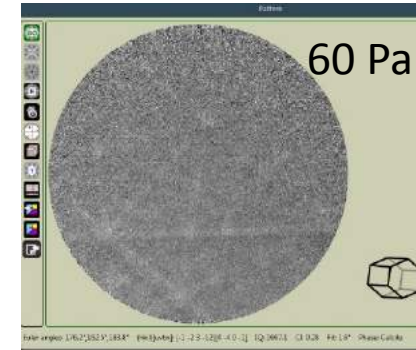
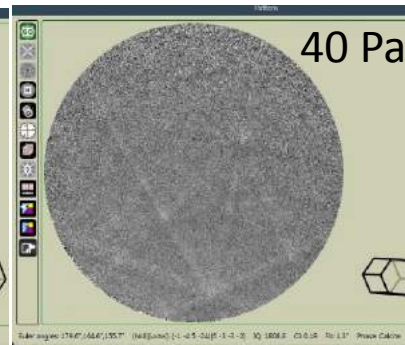
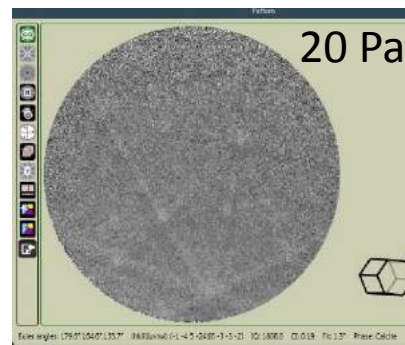
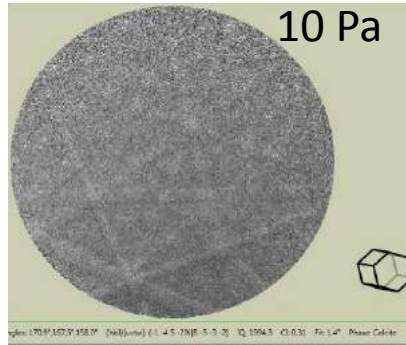
Point Info

The screenshot displays the 'Point Info' software interface. The main window is divided into several sections:

- Left Panel:** A vertical toolbar with icons for various processing steps. Below the icons is a 'Re-Index' section with three icons and a 'PCA' checkbox (checked). At the bottom is a 'Pattern' dropdown menu.
- Main View:** A large 2D OIM map showing a hexagonal pattern of grains. The top half is color-coded in shades of orange and red, while the bottom half is grayscale. A vertical color bar on the right side of the map transitions from green at the top to white at the bottom.
- Right Panel:** A control panel titled 'F8 Image Processing'. It contains several checkboxes:
 - NPAR: Neighbor Pattern Averaging
 - Dataset Background Correction
 - Partition Background Correction
 - Dynamic Background Correction
 - Auto Brightness and Contrast
 - Advanced...Two grayscale images are shown on the right side of this panel, likely representing the original and processed OIM data. Below these are radio buttons for display options:
 - Display off
 - Display Pattern
 - Display Crystal
 - Display Stats

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

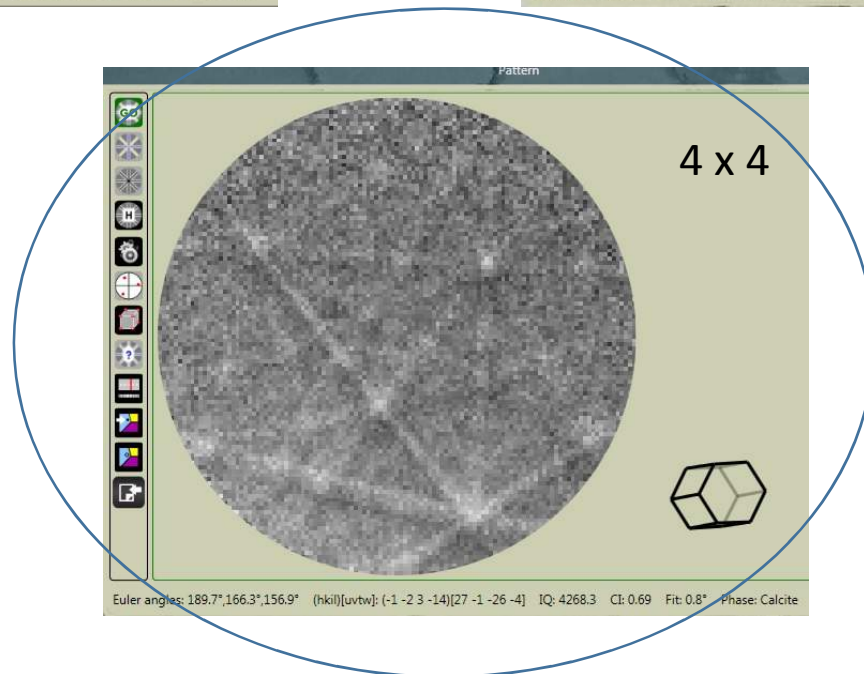
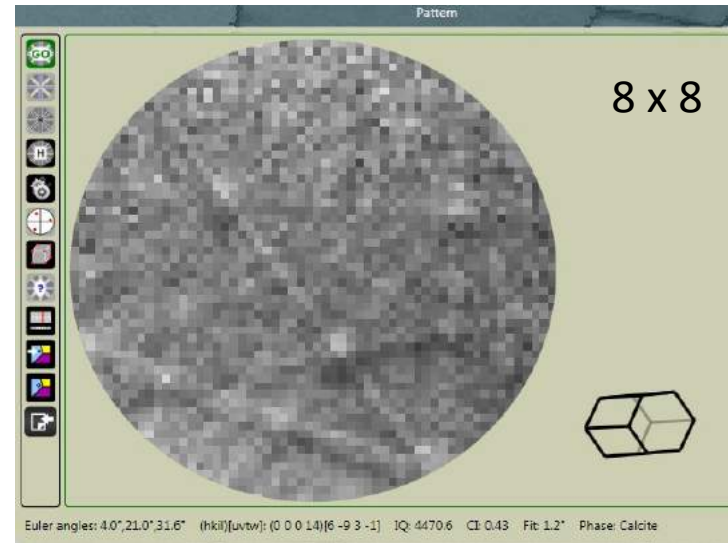
Static Background Division
Dynamic Background Subtraction
Intensity Histogram Normalization



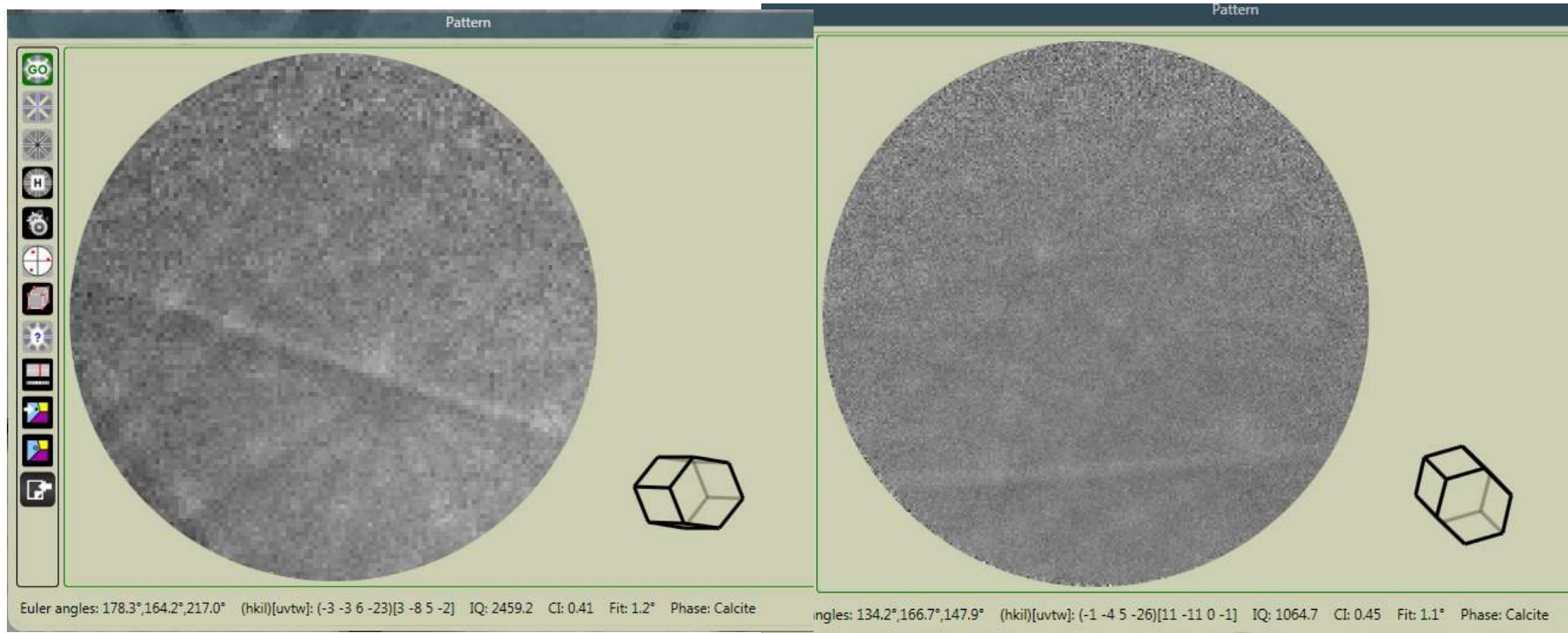
Mode Nano VP avec
Beamsleeve (cône) 350 μm

IQ \downarrow avec la pression mais il est possible
indexer

Binning 30 Pa



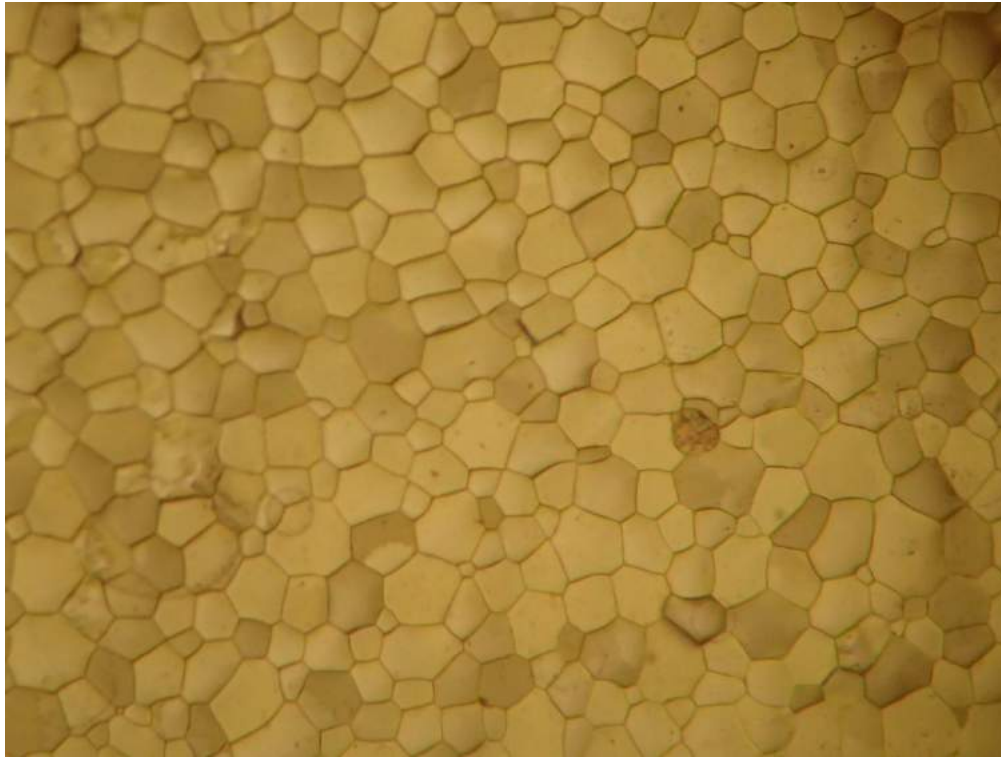
Courant - 30 Pa



I Faraday = 10.57 nA
Zeiss mode Analytical

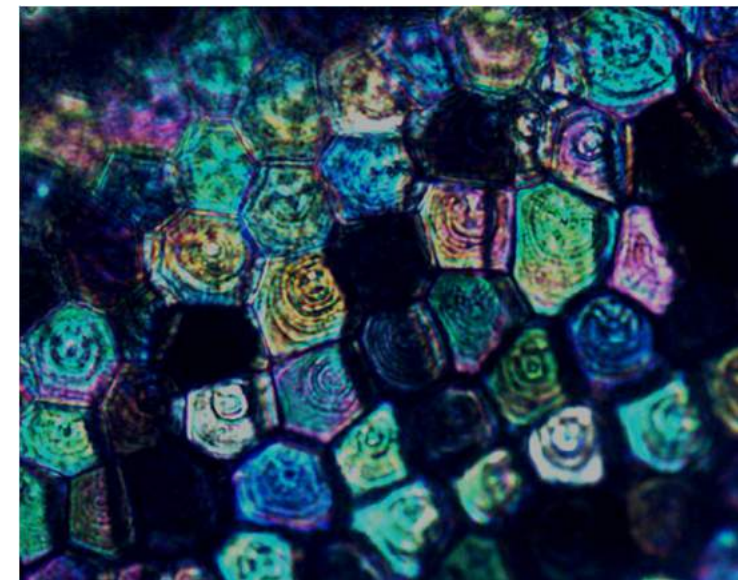
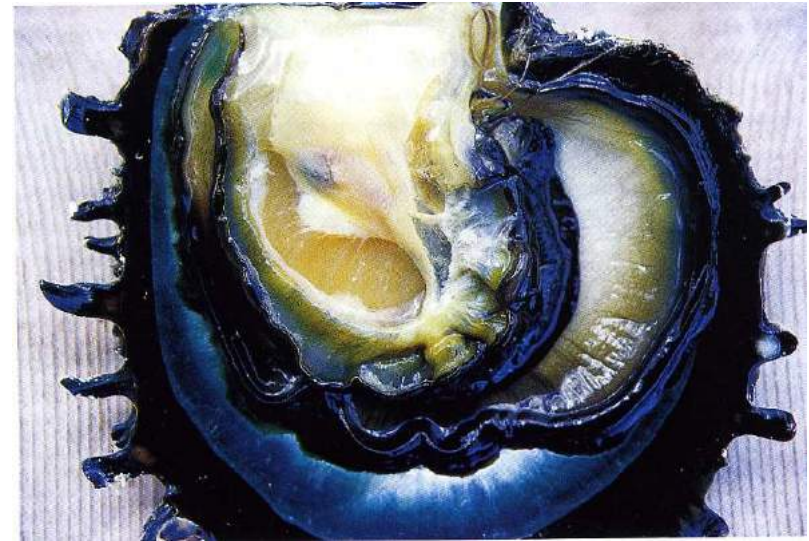
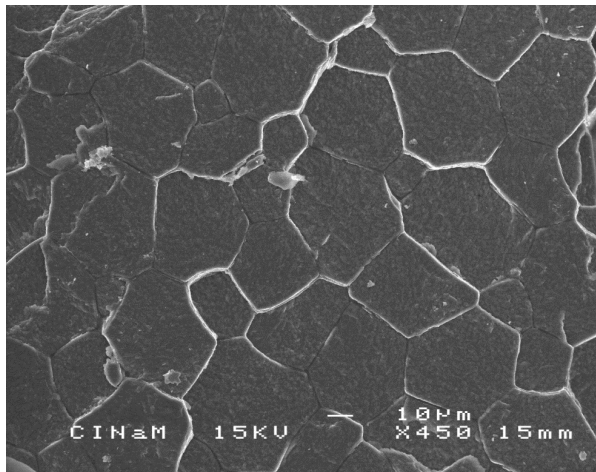
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Zeiss mode Imaging

Echantillon 2



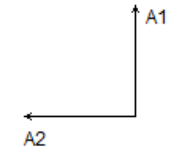
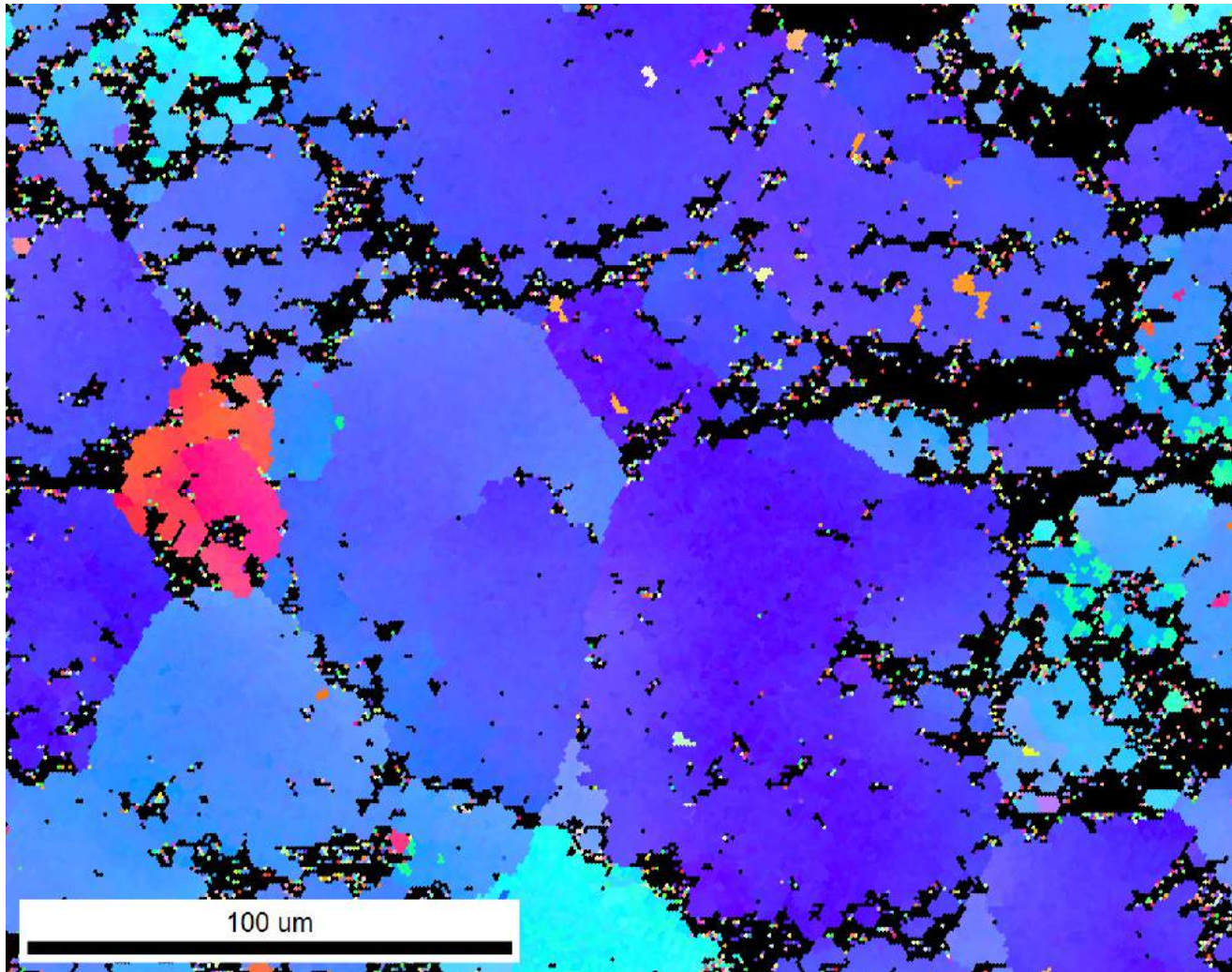
Microscope en réflexion

Calcite



Microscope polarisant

Echantillon 2

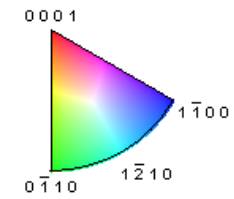


(Highlighted Points)/(Total Number of Points) = 0.000
(Highlighted Points)/(Number of Good Points) = 0.000
(Highlighted Points)/(Number of Partition Points) = 0.000

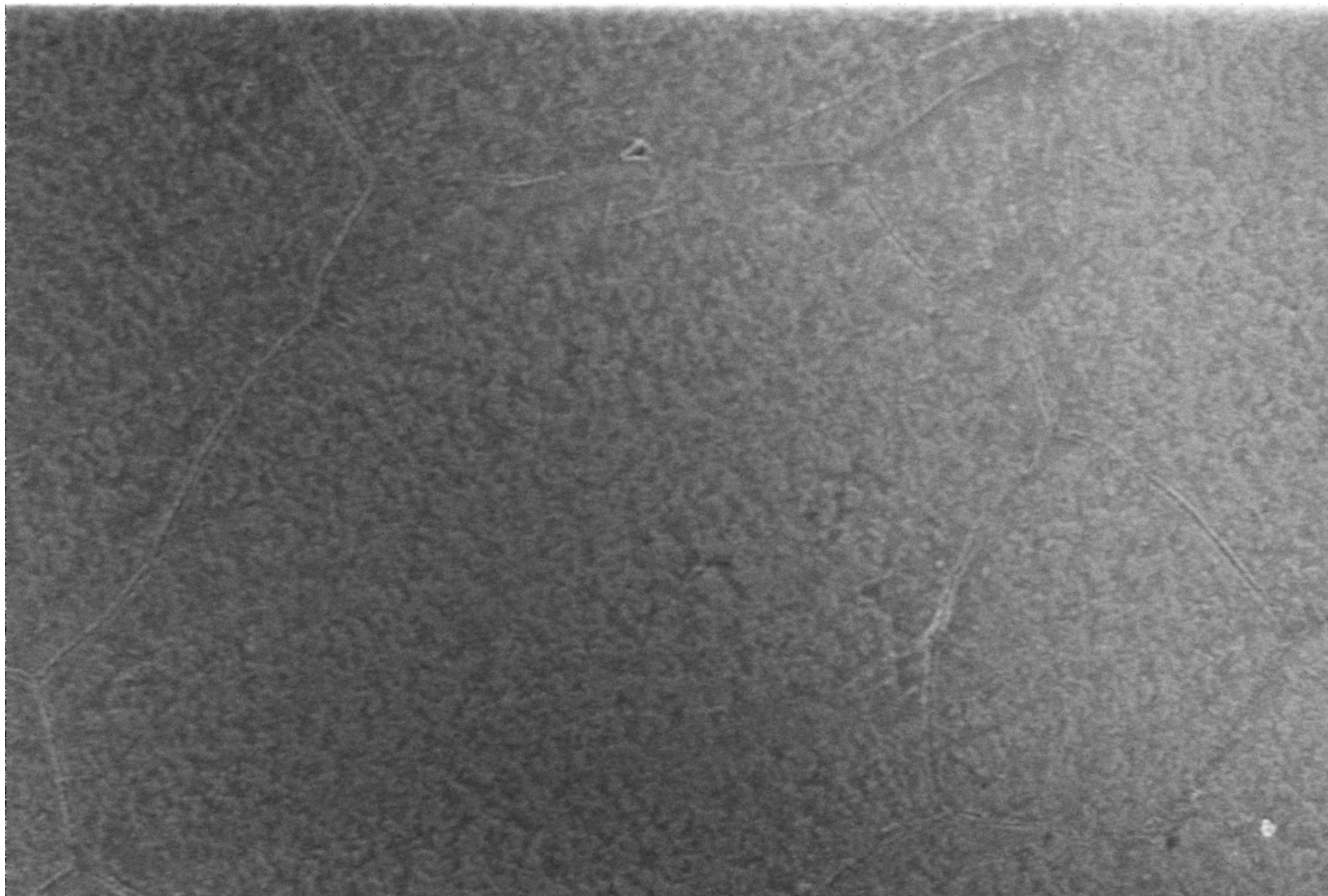
Gray Scale Map Type: <none>


Color Coded Map Type: Inverse Pole Figure [001]

Calcite

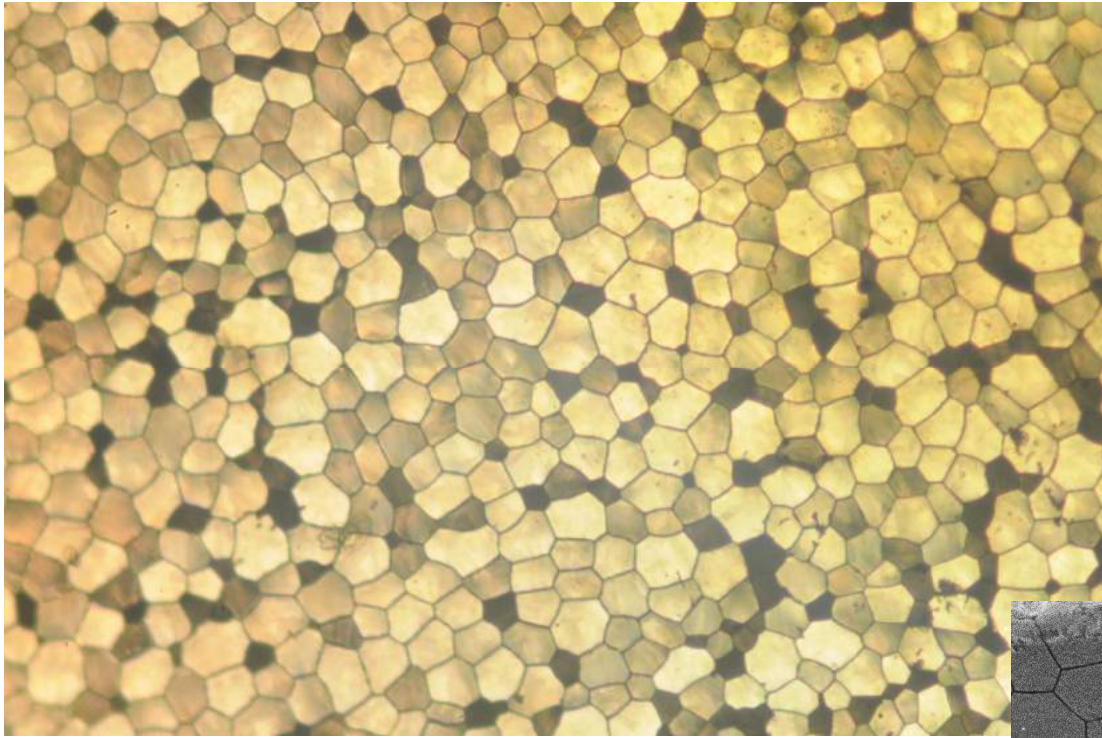


Boundaries: <none>



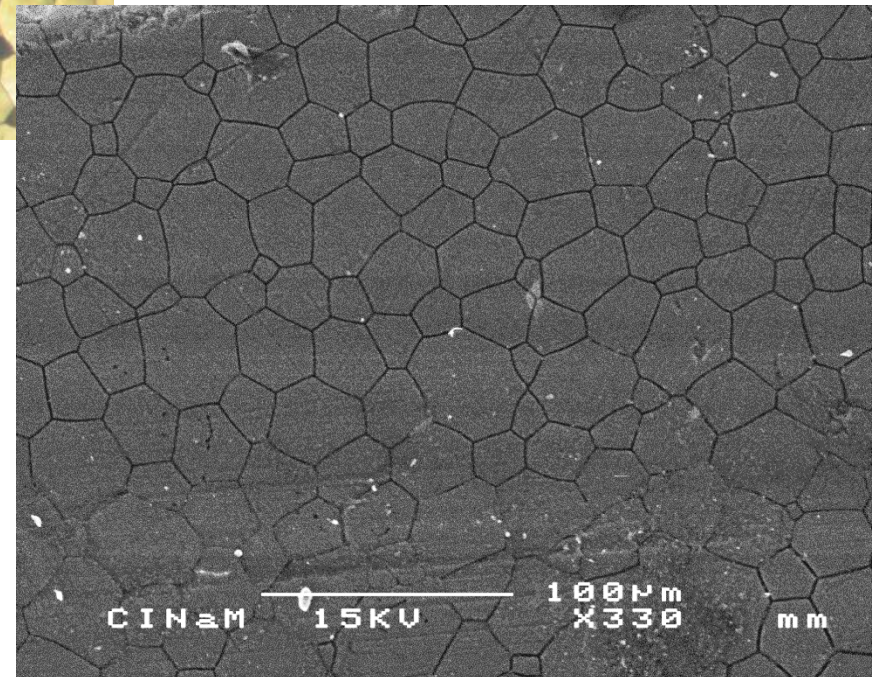
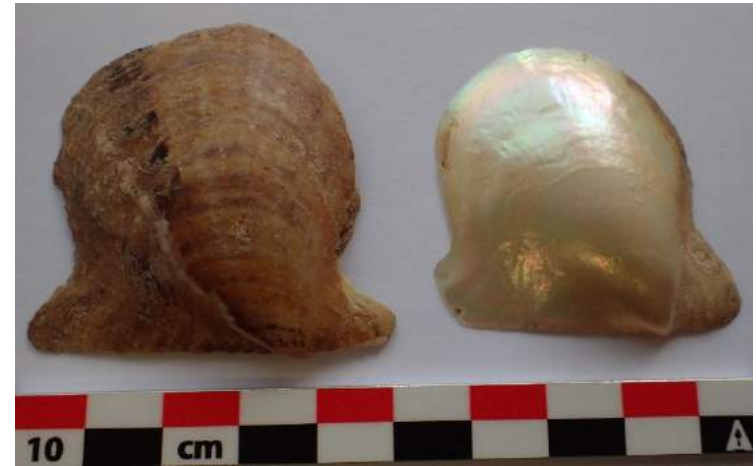
 <p>20 μm</p> <p>Gemini SEM 500 70-04</p>	<p>EHT = 1.00 kV WD = 4.6 mm Signal A = InLens</p>	<p>Mag = 920 X Pixel Size = 121.4 nm B = 48.8 % C = 35.8 %</p>	<p>Noise Red. = Drift Comp. Frame Avg. Scan Speed = 2 P = 1.23e-006 mbar Cycle Time = 18.4 Secs Ap. Size = 20.00 μm</p>	<p>1 Dec 2016 9:58:06 CP2M</p>
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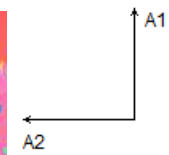
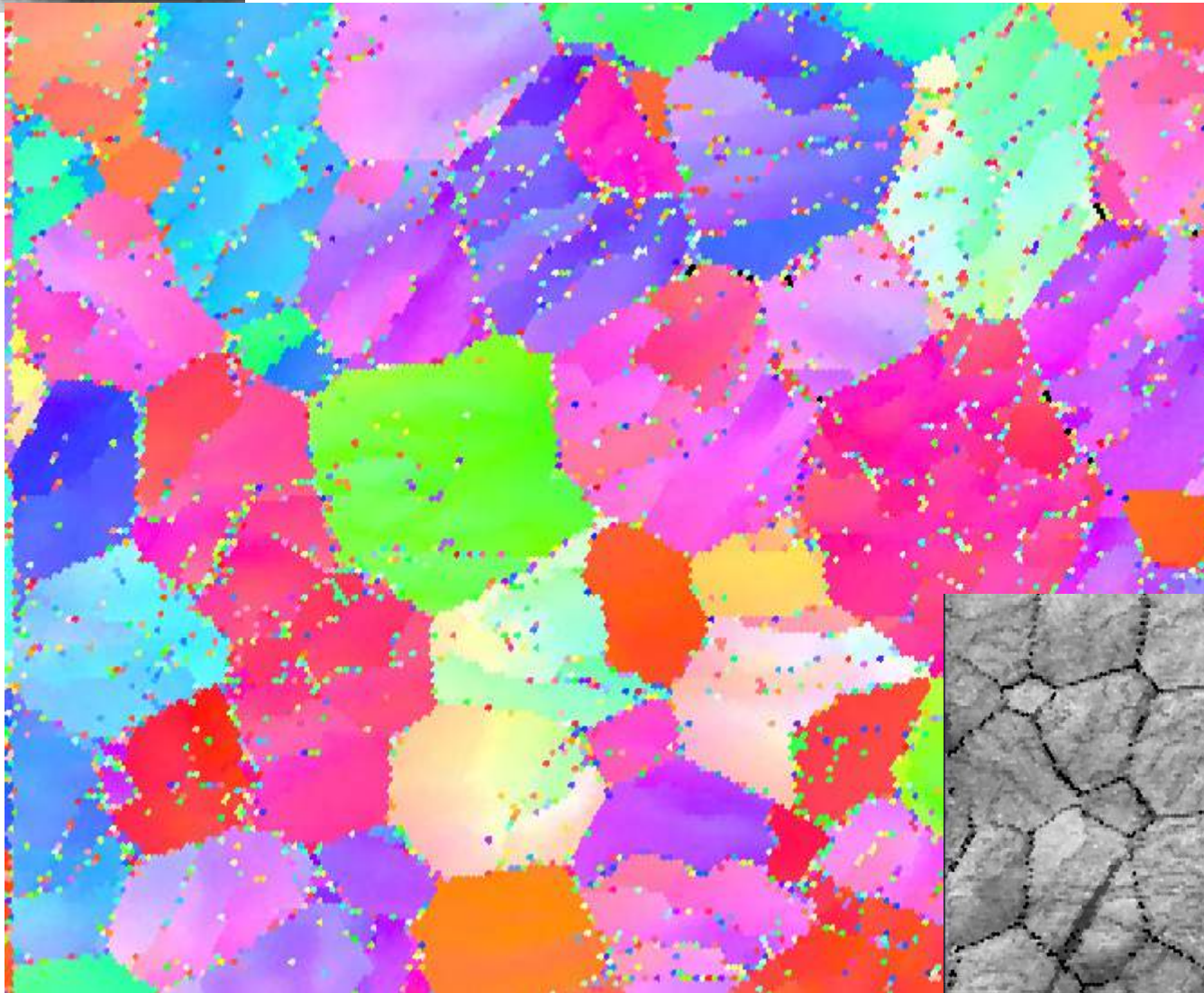
Echantillon 3



Microscope polarisant

Calcite



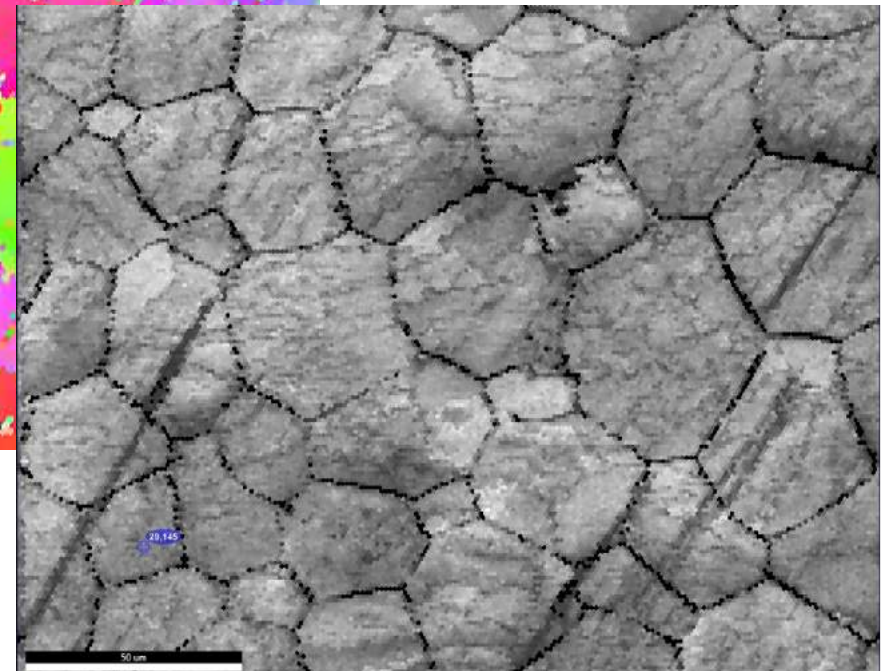
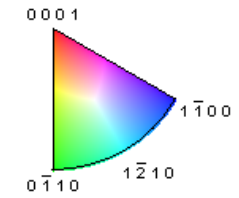


(Highlighted Points)/(Total Number of Points) = 0.000
(Highlighted Points)/(Number of Good Points) = 0.000
(Highlighted Points)/(Number of Partition Points) = 0.000

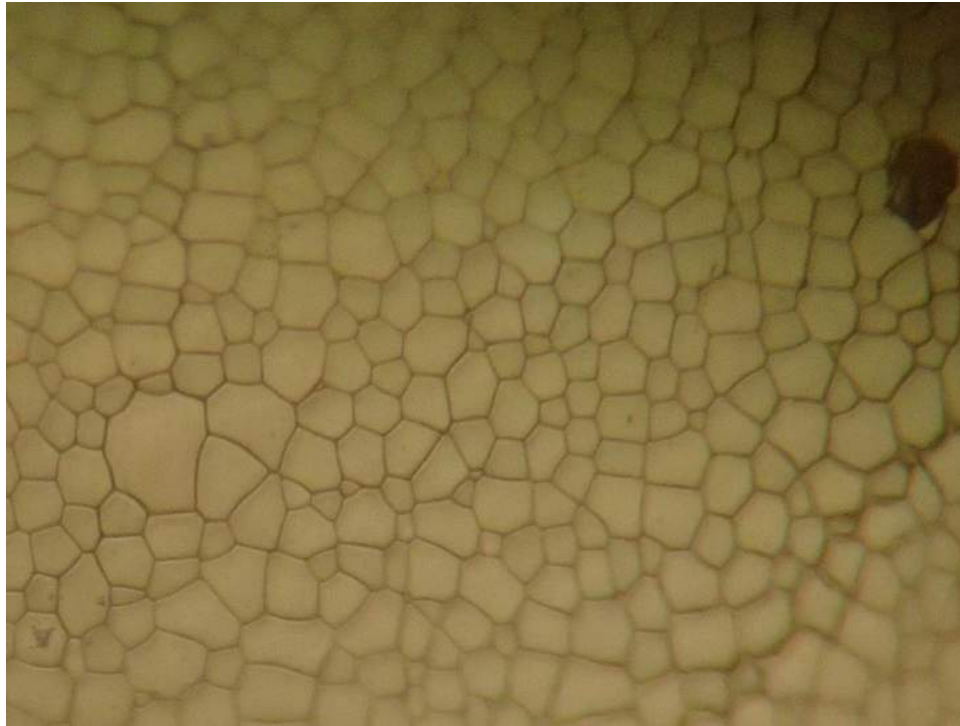
Gray Scale Map Type: <none>

Color Coded Map Type: Inverse Pole Figure [001]

Calcite



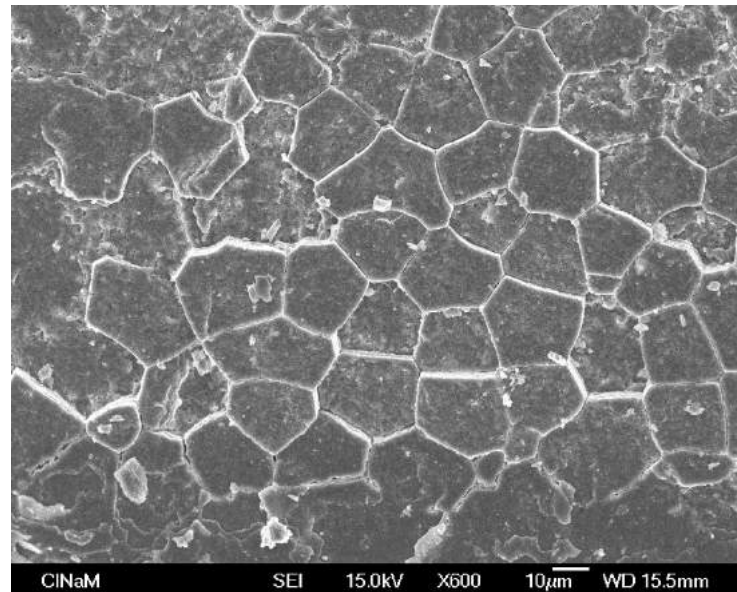
Echantillon 4

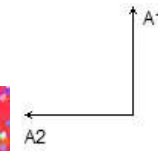
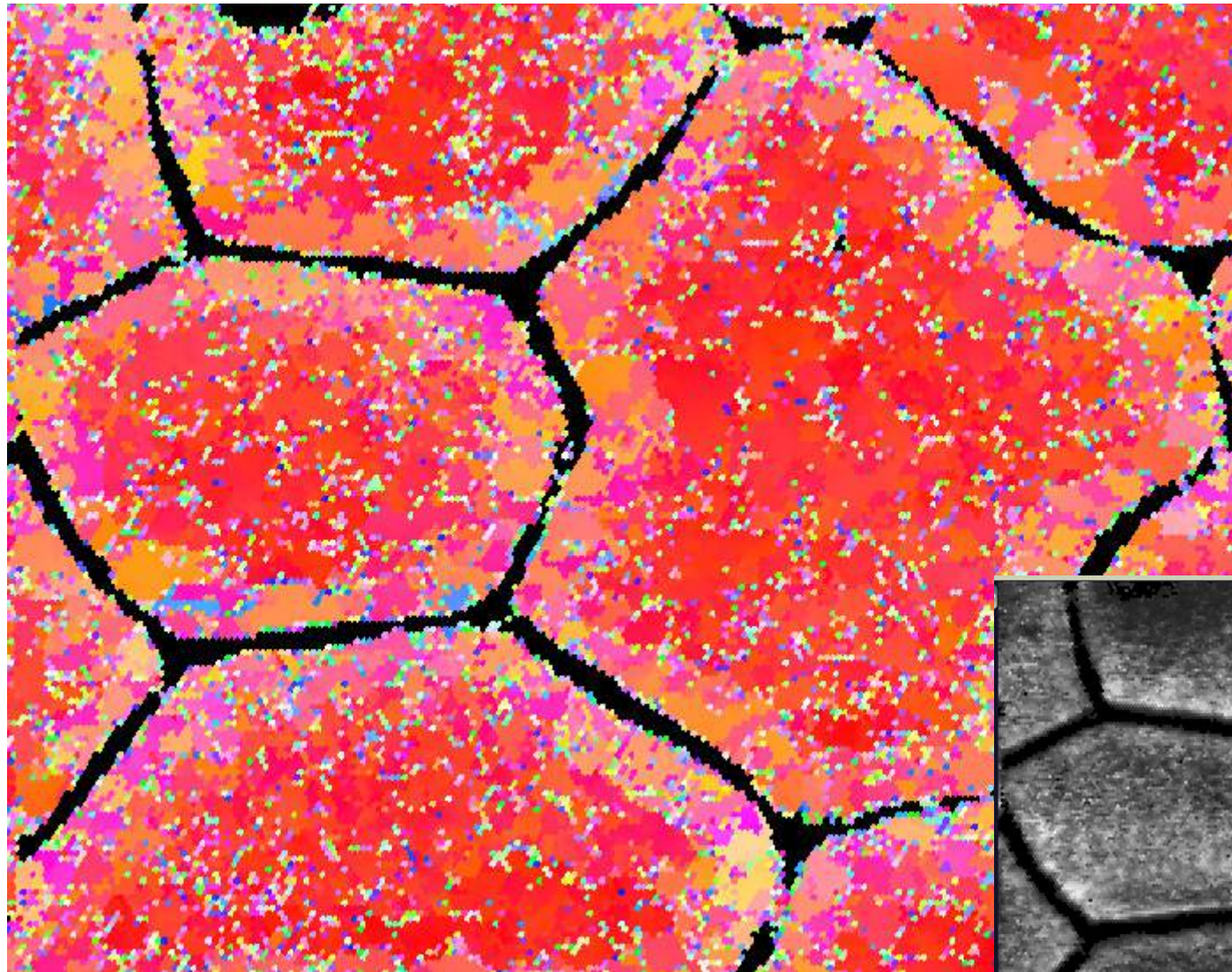


Microscope en réflexion



Aragonite



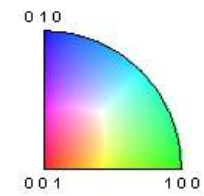


(Highlighted Points)/(Total Number of Points) = 0.000
(Highlighted Points)/(Number of Good Points) = 0.000
(Highlighted Points)/(Number of Partition Points) = 0.000

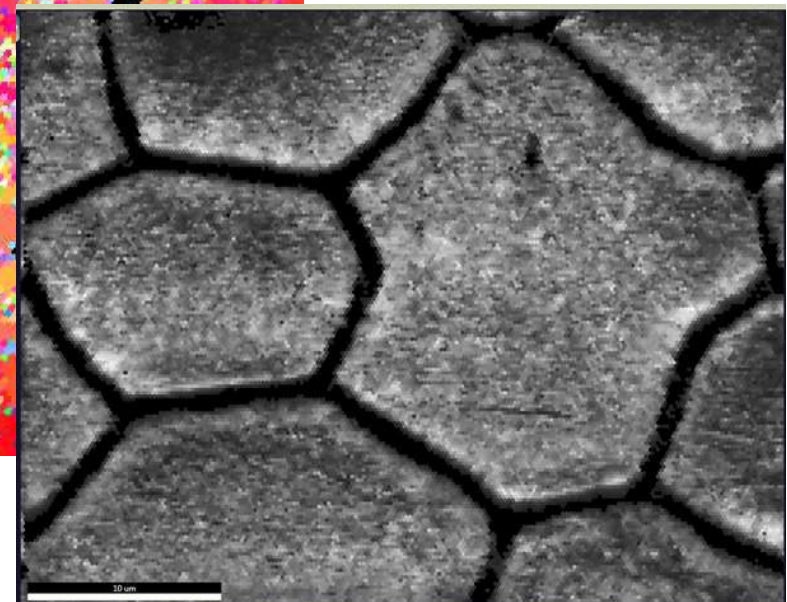
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Color Coded Map Type: Inverse Pole Figure [001]
Plane Normal (Pole)


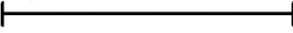
Aragonite

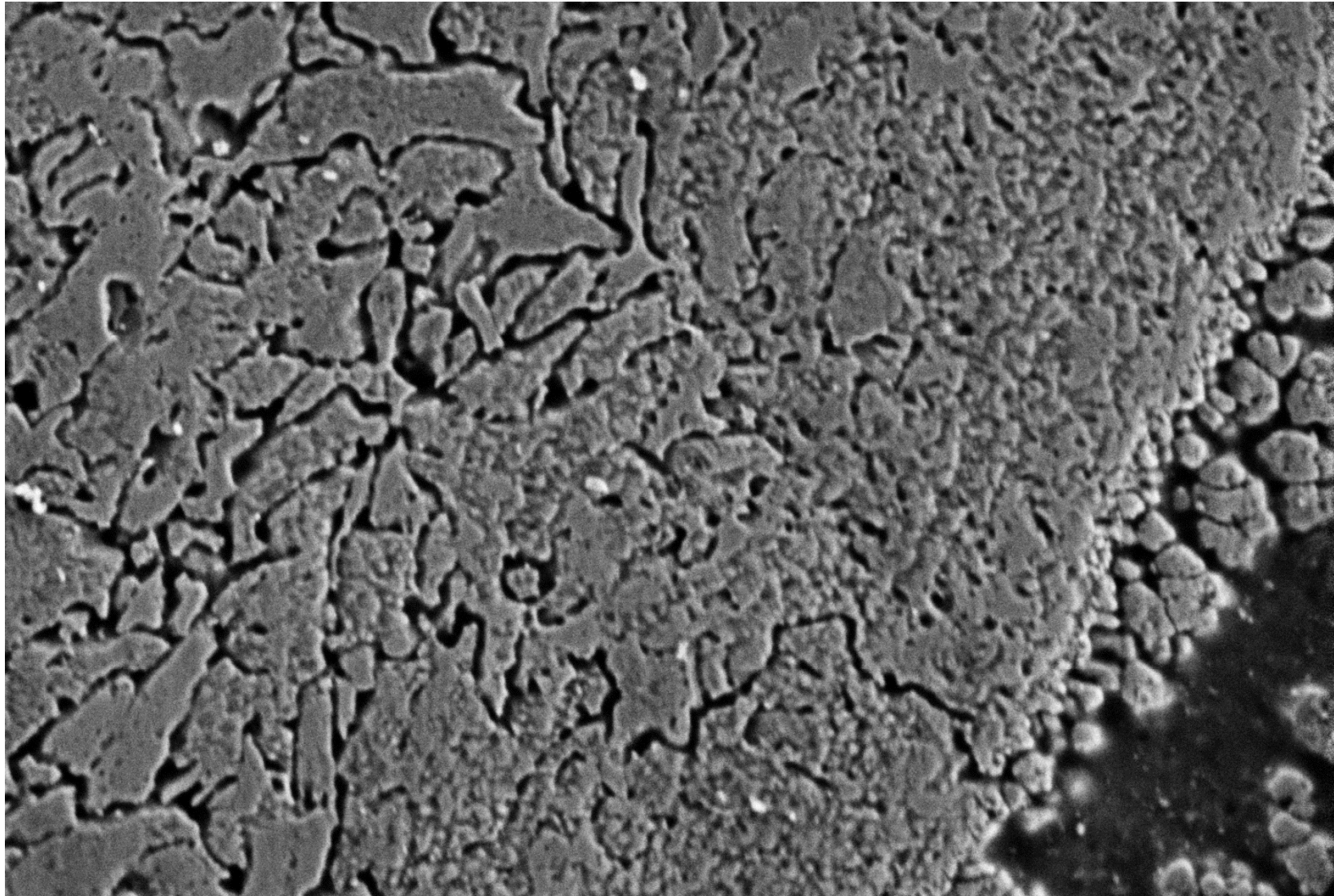




Boundaries: <none>

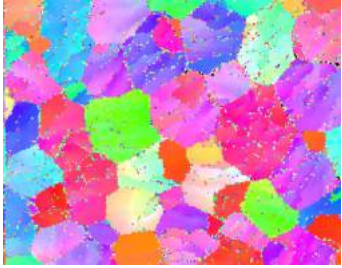




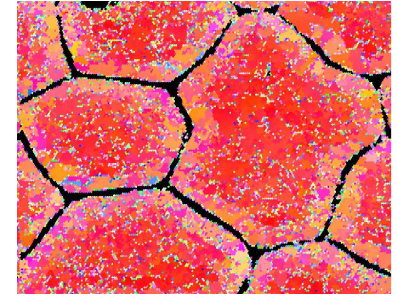
	10 μ m  Gemini SEM 500 70-04	EHT = 1.00 kV WD = 3.9 mm Signal A = HE-SE2	Mag = 1.98 KX Pixel Size = 28.12 nm B = 50.6 % C = 38.6 %	Noise Red. = Pixel Avg. Scan Speed = 10 Cycle Time = 2.7 Mins P = 6.41e-07 mbar Ap. Size = 20.00 μ m	13 Dec 2017 15:22:39
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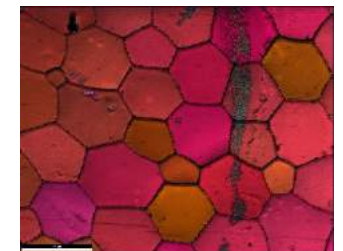
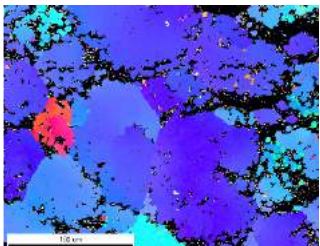
 Gemini SEM 500 70-04	200 nm 	<i>EHT</i> = 1.00 kV	<i>Mag</i> = 28.90 K X	<i>Noise Red.</i> = Drift Comp. Frame Avg.	13 Dec 2017
		<i>WD</i> = 3.9 mm	<i>Pixel Size</i> = 1.932 nm	<i>Scan Speed</i> = 3 <i>P</i> = 5.30e-07 mbar	15:37:17
		<i>Signal A</i> = HE-SE2	<i>B</i> = 46.1 % <i>C</i> = 40.8 %	<i>Cycle Time</i> = 1.3 Mins <i>Ap. Size</i> = 20.00 μm	

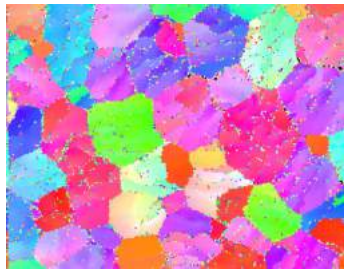


CONCLUSION

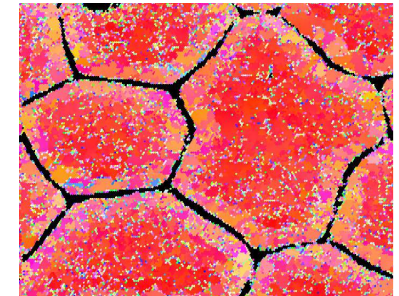


- La carte d'orientations EBSD montre qu'il n'y a pas de contrôle de cristallisation
- Possibilité de tracer la partie organique via EDS
- Imagerie et EBSD dans une vaste gamme de pressions. Pour le cas du système Zeiss, travailler en VP à 30 Pa et en nano VP entre 60 et 80 Pa
- Avec l'augmentation de la pression, diminution du IQ, mais amélioration avec le post-processing





Remerciements



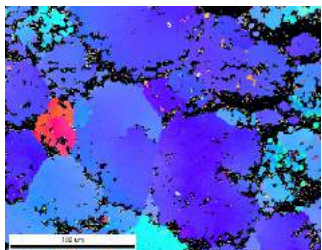
EDAX – Rene DE KLOE

ZEISS – Zhou FANG

CP2M – Martiane CABIE, Christian DOMINICI

François BRISSET

GN- MEBA



Merci pour votre attention!
andrea.campos@univ-amu.fr

