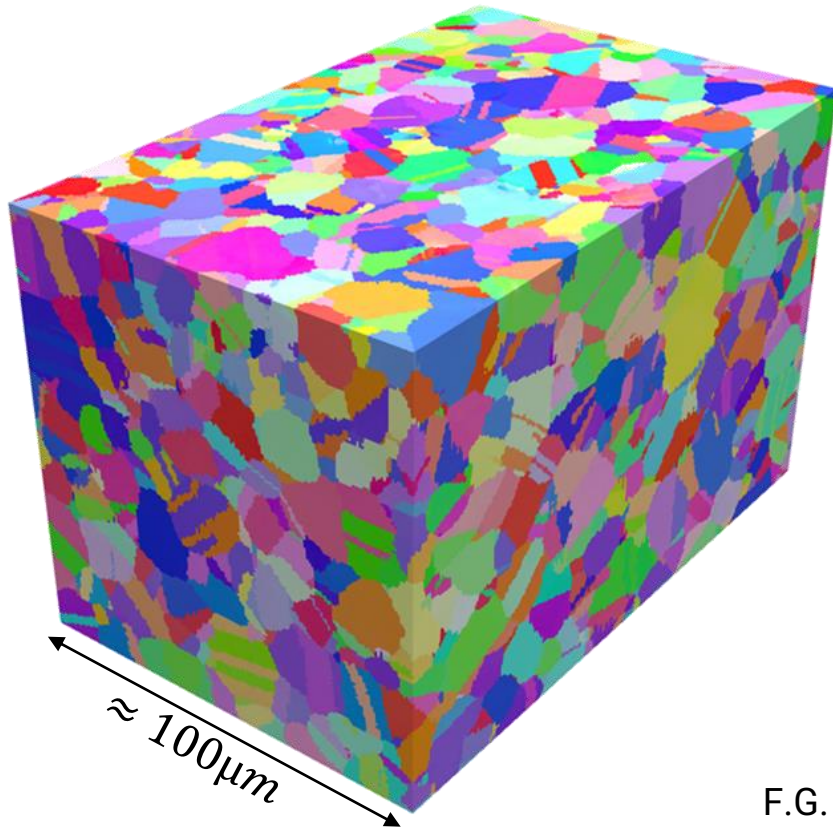




PSL

Cemef



# MEB FIB plasma pour l'analyse de « grands » volumes

Alexis NICOLAÏ – *ingénieur de recherche*

[alexis.nicolay@minesparis.psl.eu](mailto:alexis.nicolay@minesparis.psl.eu)

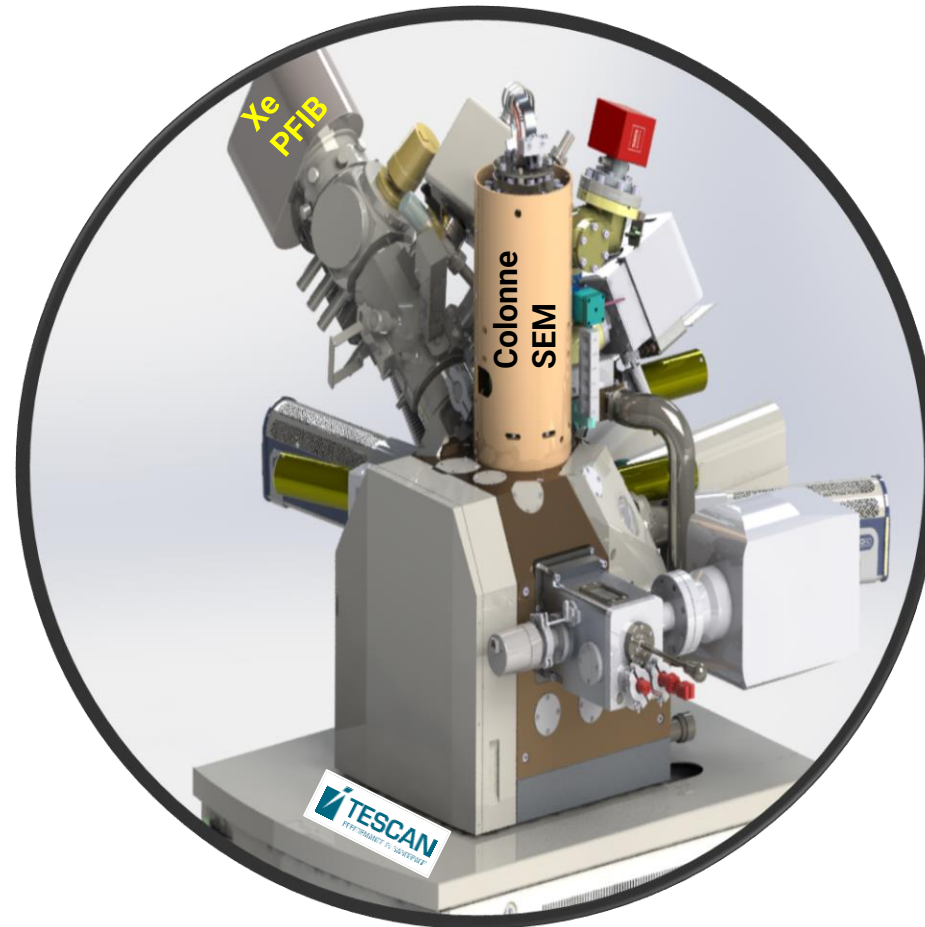
F.G. Jaime, N. Bozzolo, J. Silvent (*Orsay Physics*), G. Goupil (*Orsay Physics*), S. Desbief (*Tescan Analytics*)



# PLAN DE LA PRÉSENTATION

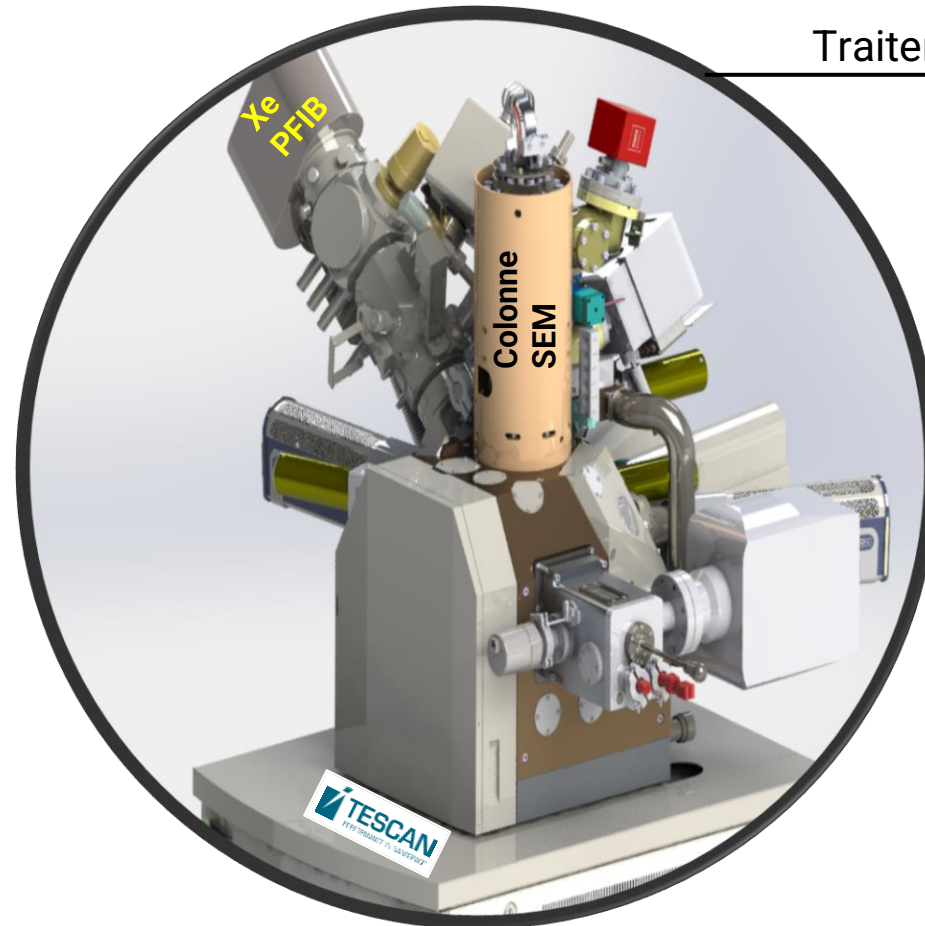
- Présentation rapide de l'équipement du CEMEF
- Principes généraux d'une analyse 3D (acquisition et post-traitement)
- Optimisation des conditions opératoires dans le cas de superalliages base nickel
- Exemples de volumes analysés
- Conclusions et perspectives

# PRÉSENTATION DE L'ÉQUIPEMENT DU CEMEF



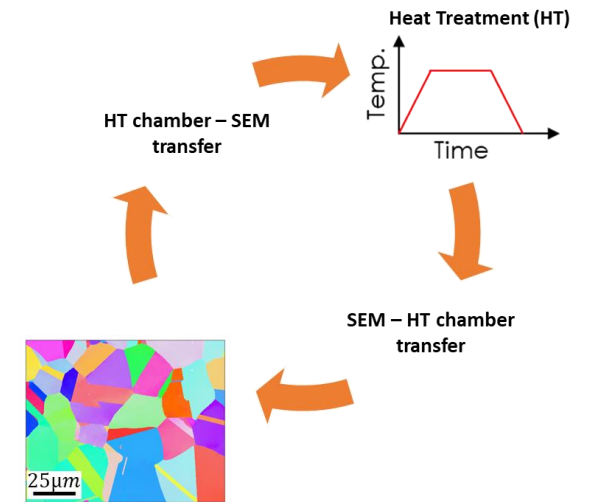
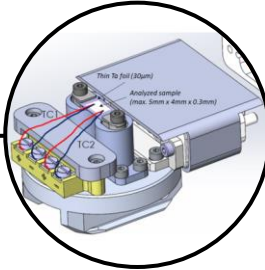
**TESCAN FERA 3**

# PRÉSENTATION DE L'ÉQUIPEMENT DU CEMEF

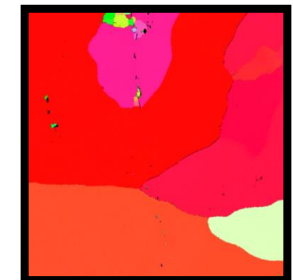


**TESCAN FERA 3**

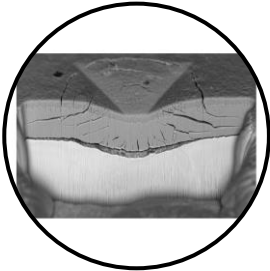
Traitements thermiques « in-situ »



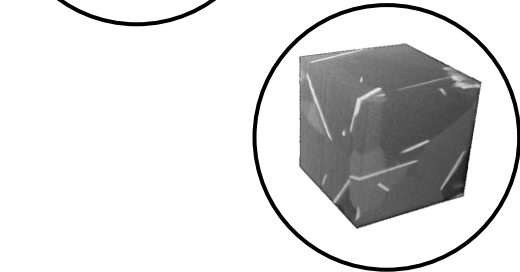
Exemple (alliage d'Aluminium)



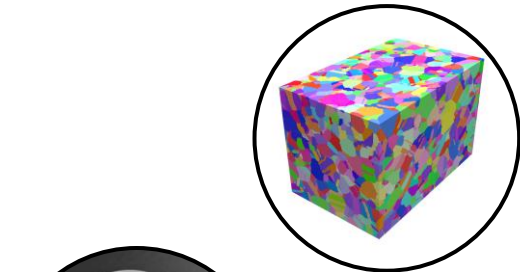
# PRÉSENTATION DE L'ÉQUIPEMENT DU CEMEF



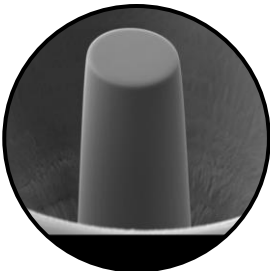
Cross-sections



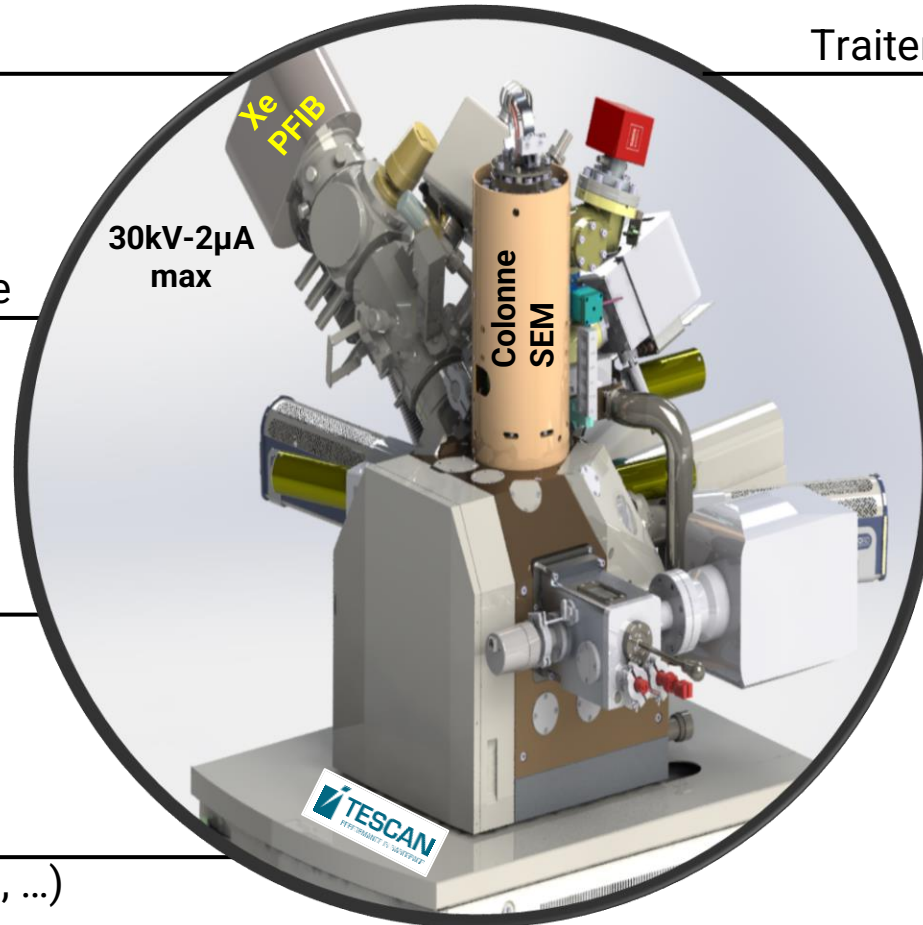
Tomographie



EBSD / EDS  
3D

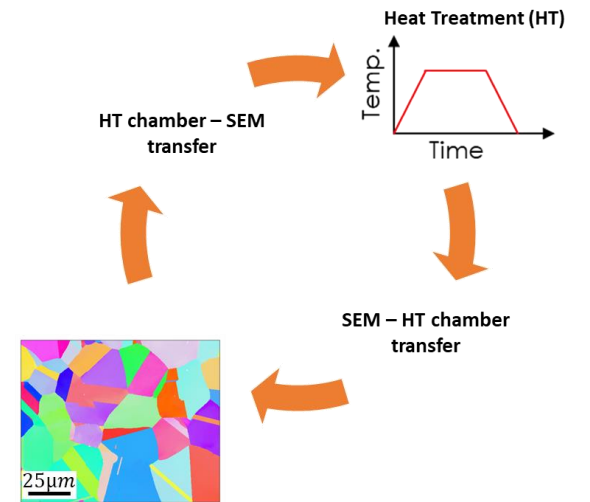
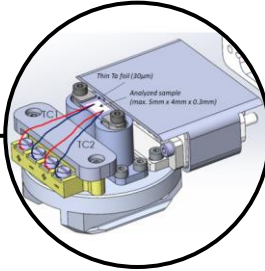


Autres préparations  
(Micro-piliers, Lames TEM, ...)

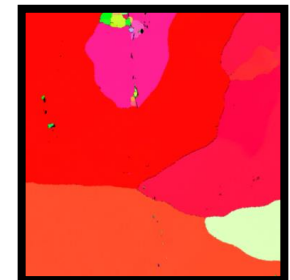


TESCAN FERA 3

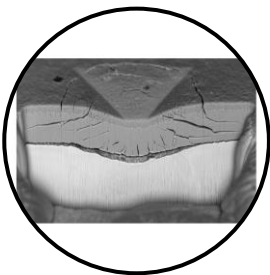
Traitements thermiques « in-situ »



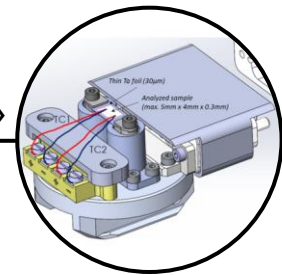
Exemple (alliage d'Aluminium)



# PRÉSENTATION DE L'ÉQUIPEMENT DU CEMEF

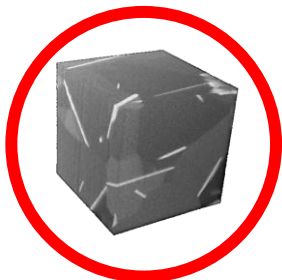


Cross-sections



Traitements thermiques « in-situ »

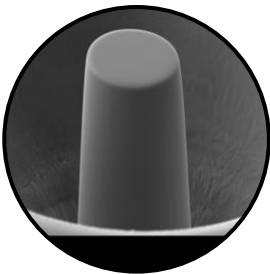
Scope de la présentation



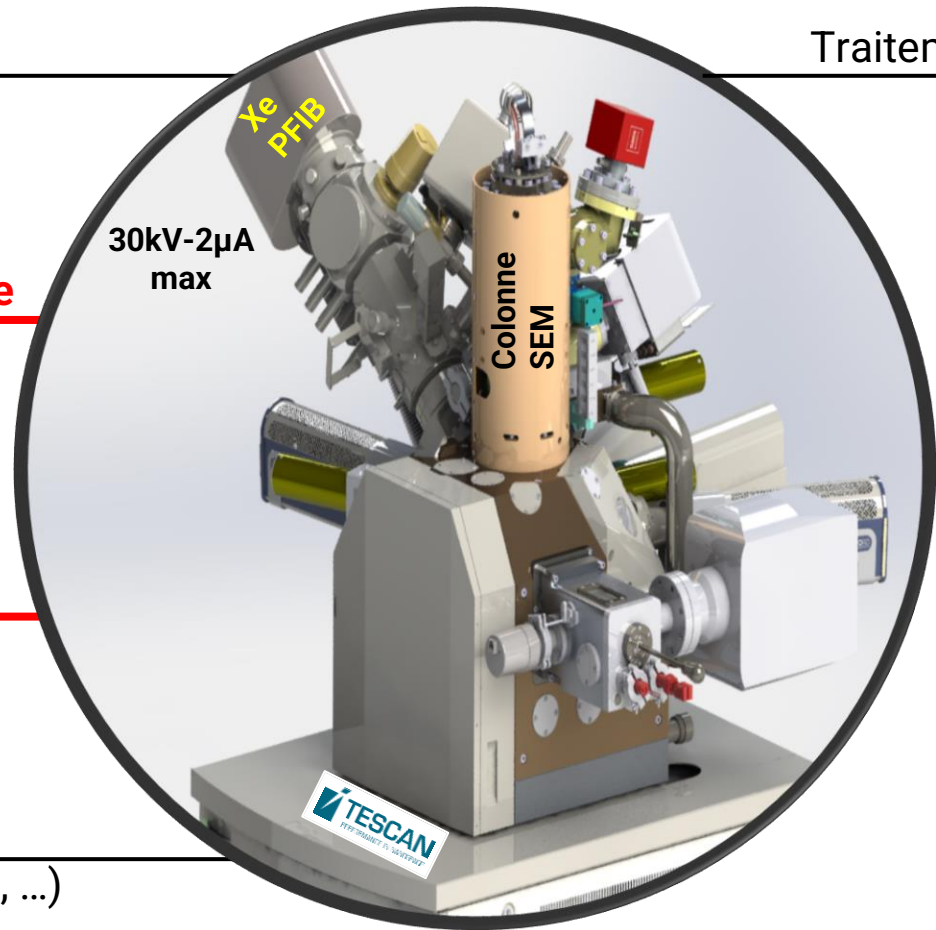
Tomographie



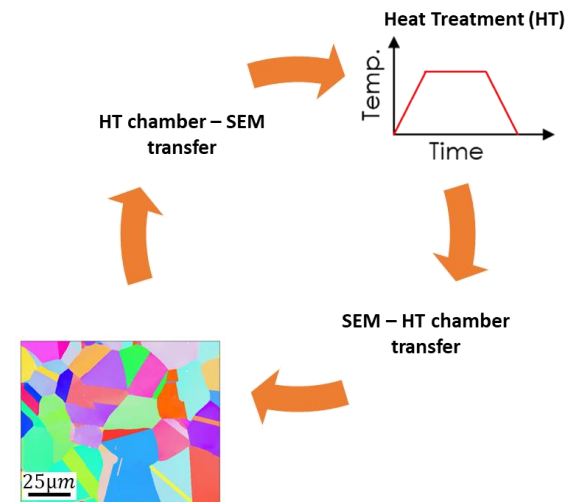
EBSD / EDS  
3D



Autres préparations  
(Micro piliers, Lames TEM, ...)



TESCAN FERA 3

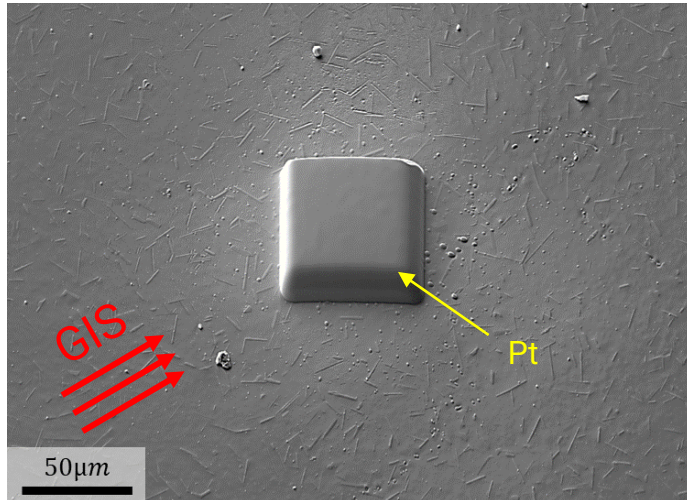


Exemple (alliage d'Aluminium)

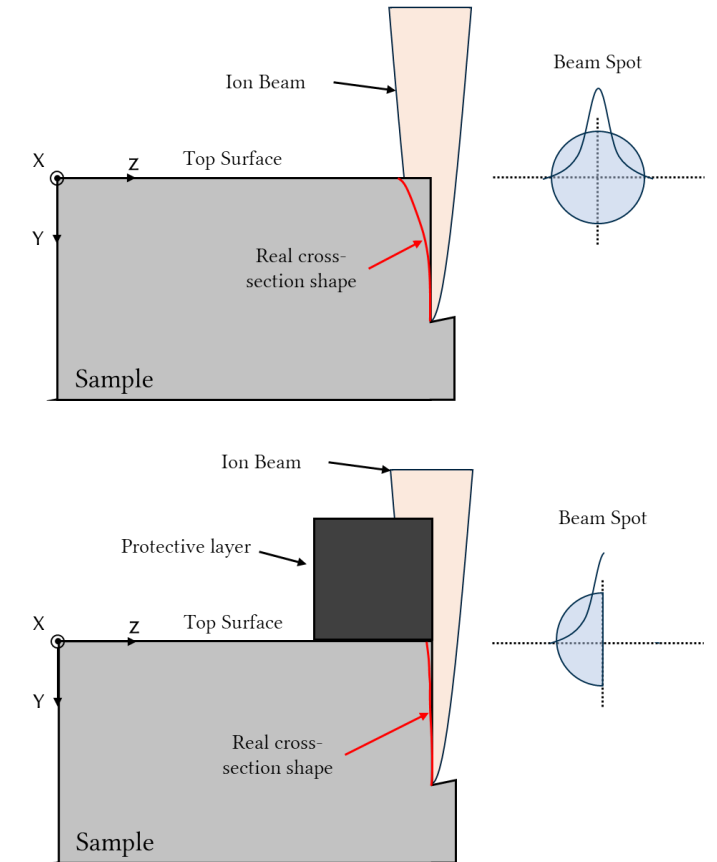
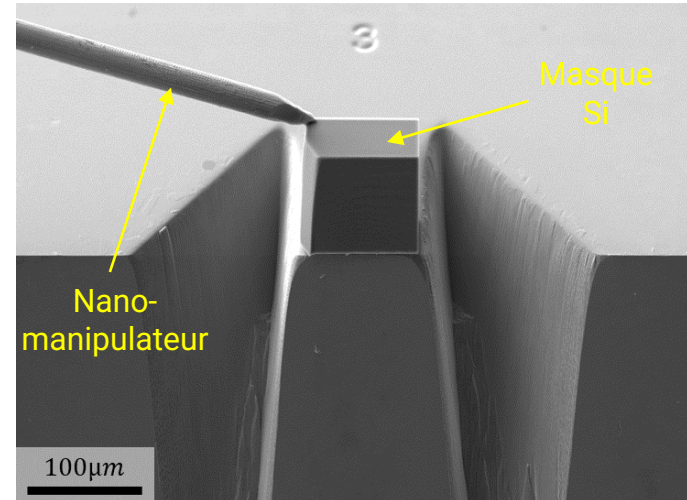


# PRINCIPES GÉNÉRAUX D'UNE ANALYSE 3D AU FIB

Dépôt de platine (Pt)



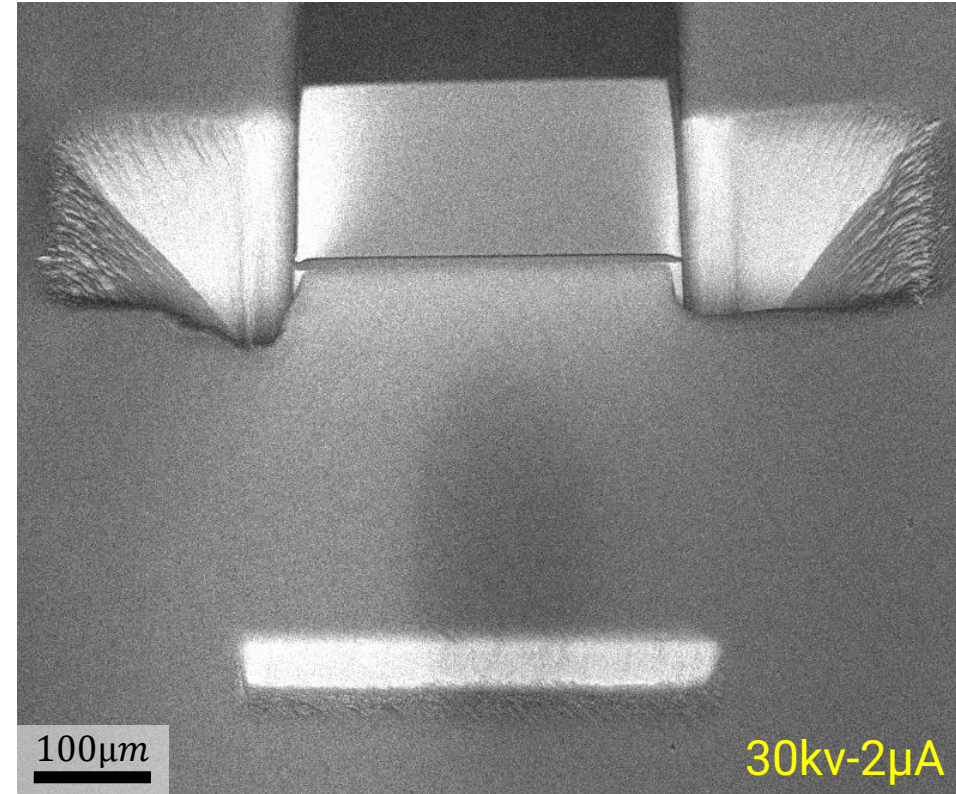
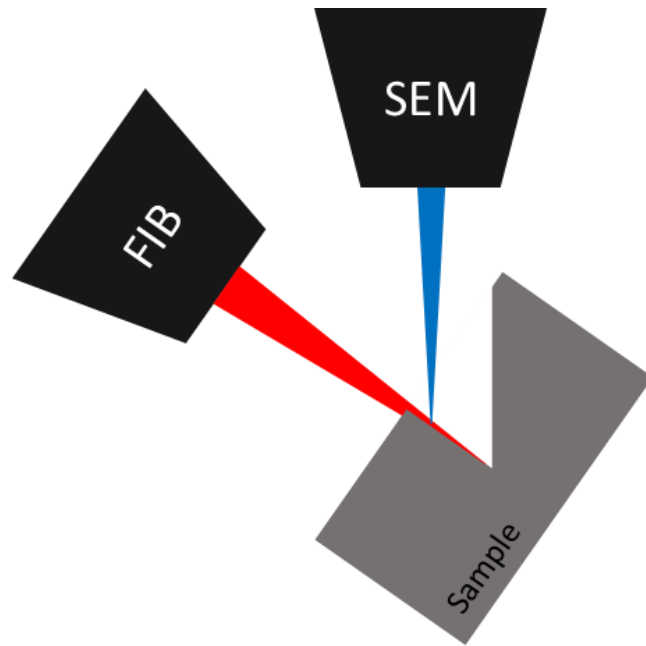
Dépôt de masque en Silicium (Si)



Protection ROI



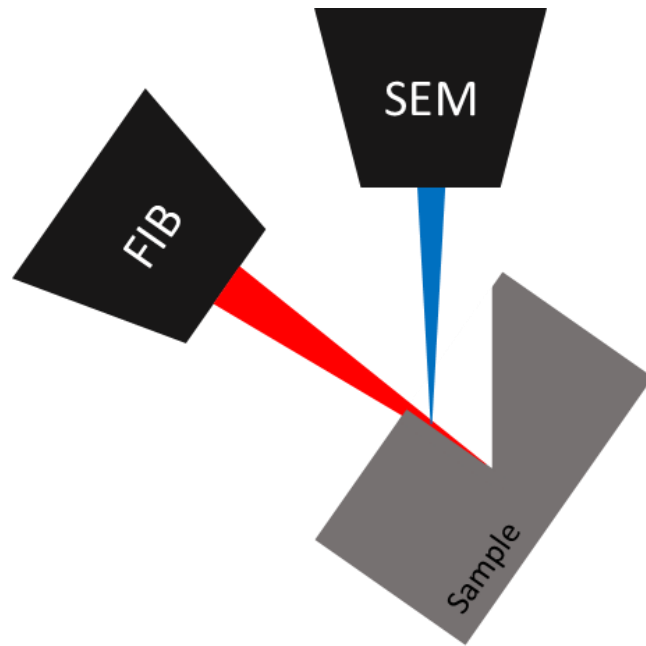
# PRINCIPES GÉNÉRAUX D'UNE ANALYSE 3D AU FIB



Protection ROI

Ouverture cross-section

# PRINCIPES GÉNÉRAUX D'UNE ANALYSE 3D AU FIB



Après abrasion à fort courant FIB  
*Curtaining*



Ion beam  
Spot quality

Material  
surface  
rugosity

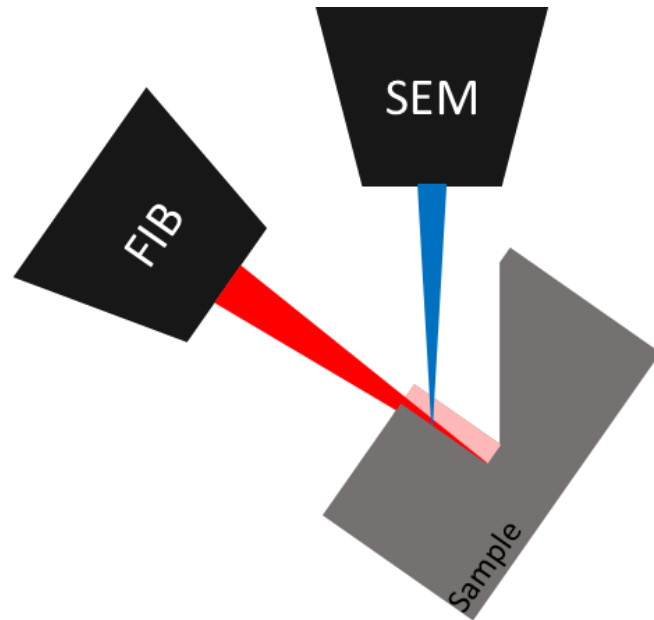
Ion beam  
intensity

Material  
chemical  
homogeneity

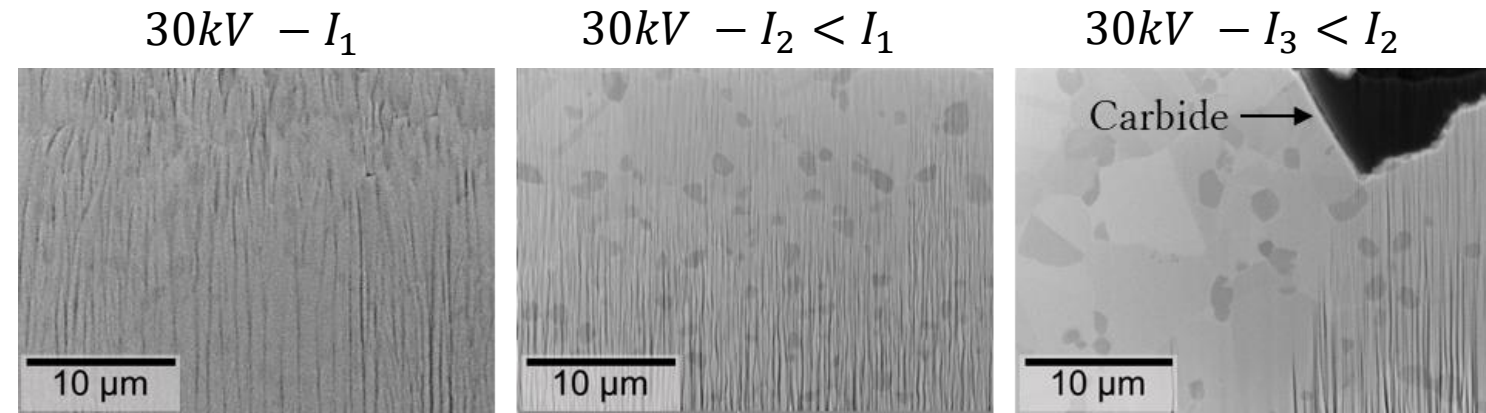
Protection ROI

Ouverture cross-  
section

# PRINCIPES GÉNÉRAUX D'UNE ANALYSE 3D AU FIB



**Polissages à des courant de plus en plus faibles**  
*AD730 (superalliage base Ni)*



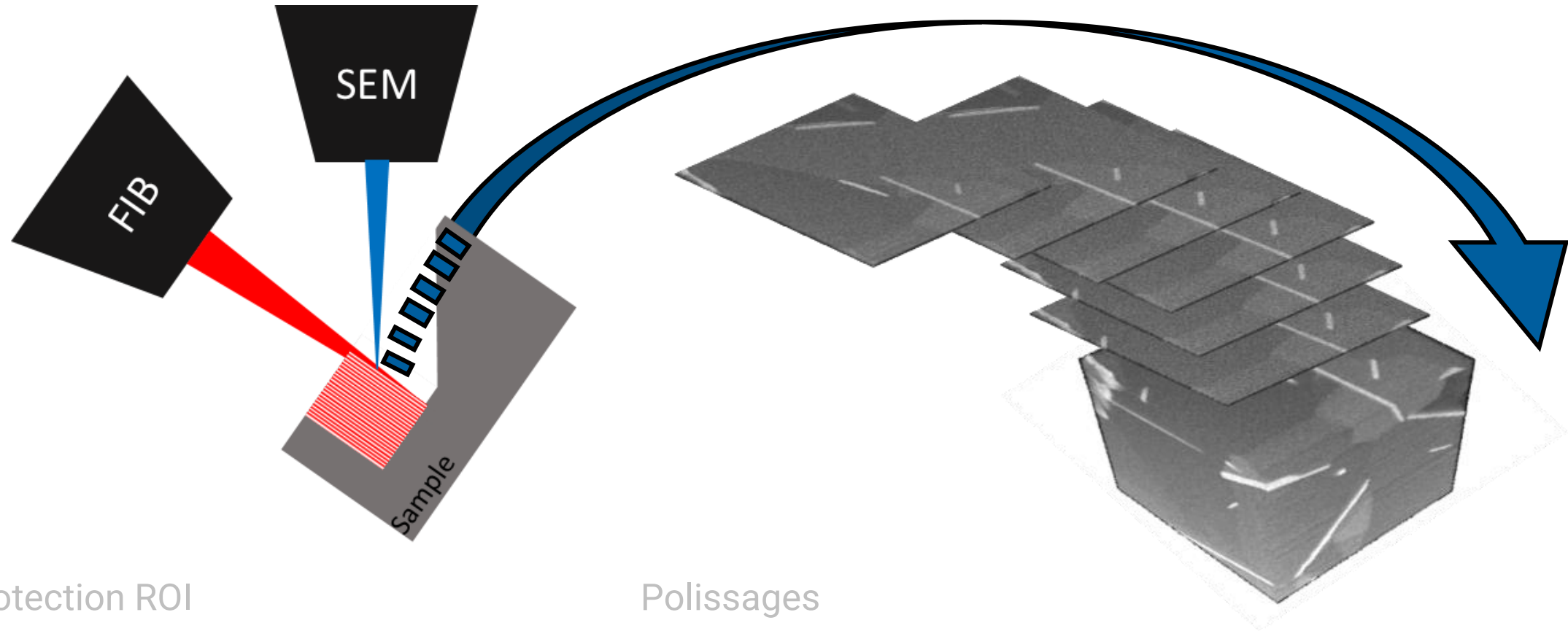
Amélioration de l'état de surface  
*Réduction du curtaining*

Protection ROI

Polissages

Ouverture cross-section

# PRINCIPES GÉNÉRAUX D'UNE ANALYSE 3D AU FIB



Protection ROI

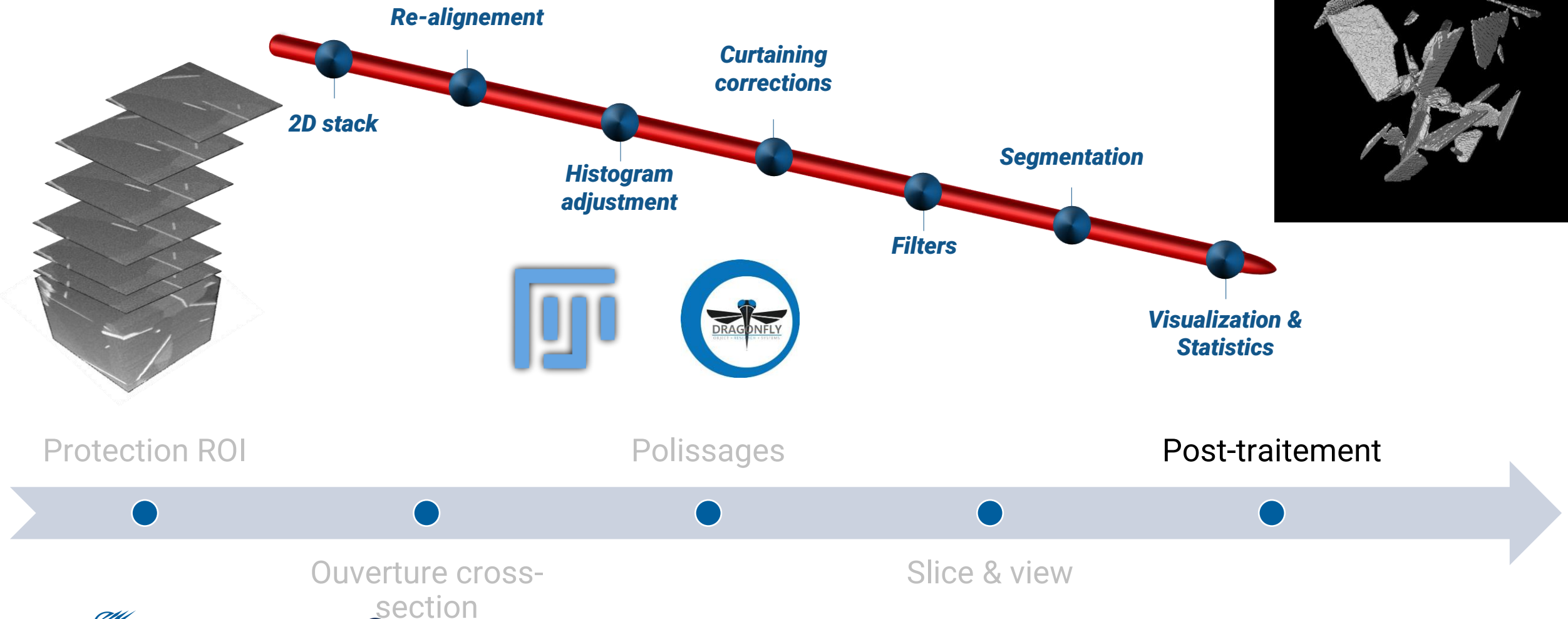
Polissages

Ouverture cross-section

Slice & view

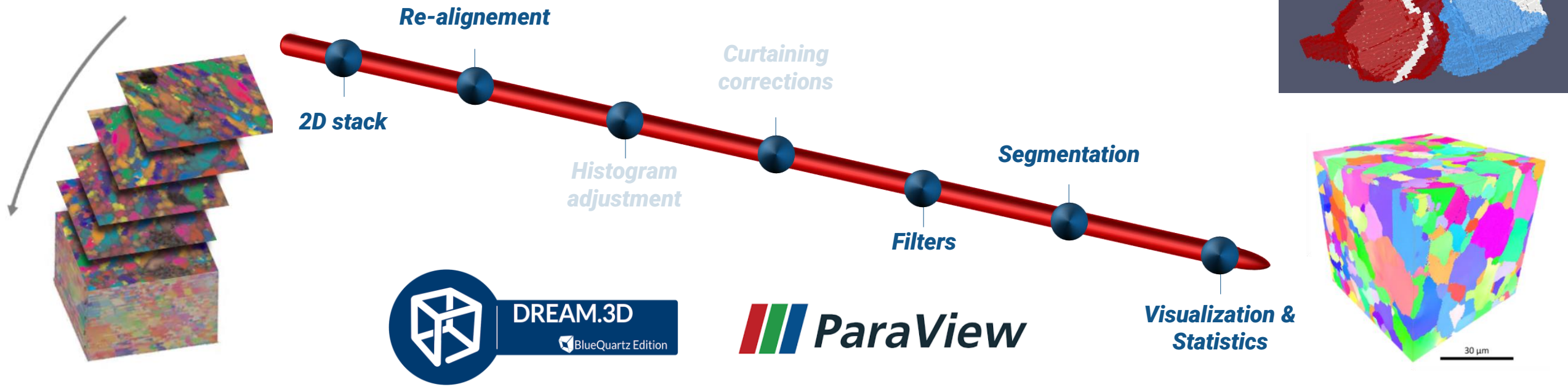
# PRINCIPES GÉNÉRAUX D'UNE ANALYSE 3D AU FIB

Séquence typique de post-traitement d'un stack d'images



# PRINCIPES GÉNÉRAUX D'UNE ANALYSE 3D AU FIB

## Séquence typique de post-traitement d'un stack EBSD



Protection ROI

Polissages

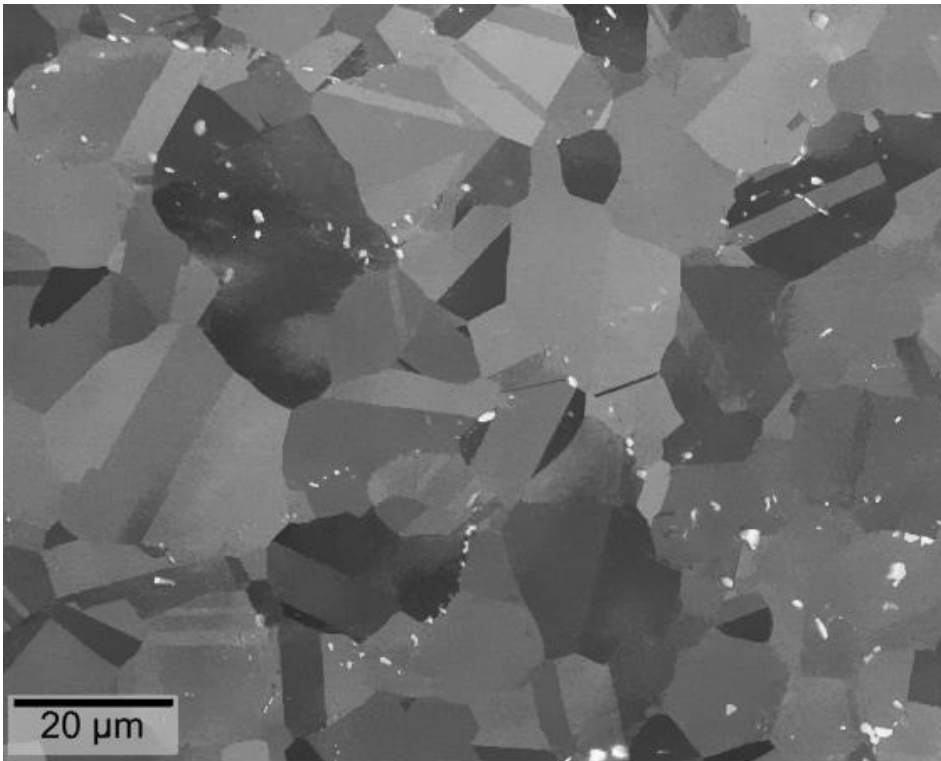
Post-traitement

Ouverture cross-section

Slice & view

# OPTIMISATION DES CONDITIONS OPÉRATOIRES

## CAS DES SUPERALLIAGES BASE NICKEL



### Inconel 718

$\gamma$  - matrice cfc

$\delta$  - précipités  $Ni_3Nb$

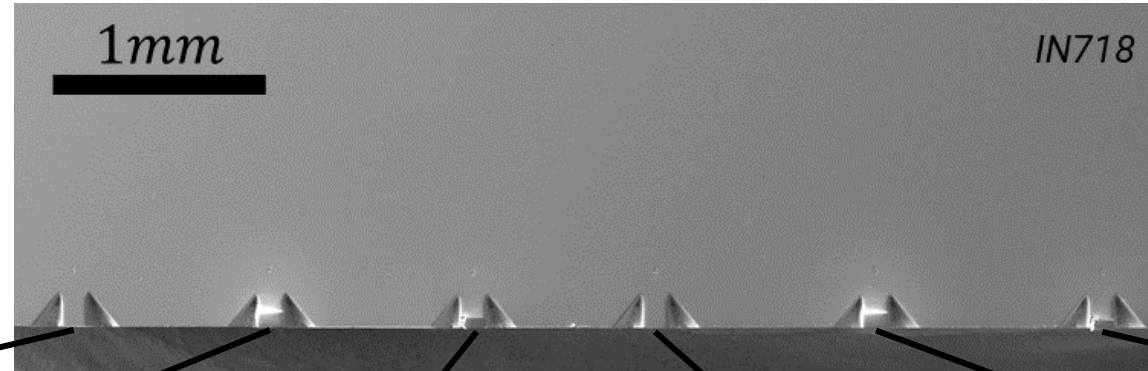
$$SR = 0.52 \mu m^3 / nA / s$$

### Superalliages base Nickel

- Taille de grains moyenne  $\sim 10\mu m$
- Nécessité d'analyser des volumes de tailles  $\sim 100 \times 100 \times 100 \mu m^3$  pour être représentatif de la microstructure moyenne de ces alliages ( $\rightarrow \sim 18h$  à  $30kV-30nA$ )
- Nécessité donc de travailler au courant le plus élevé possible avec niveau curtaining acceptable.
- $\rightarrow$  Maitriser du curtaining par un autre levier que la réduction du courant

# OPTIMISATION DES CONDITIONS OPÉRATOIRES

## CAS DES SUPERALLIAGES BASE NICKEL



Nothing

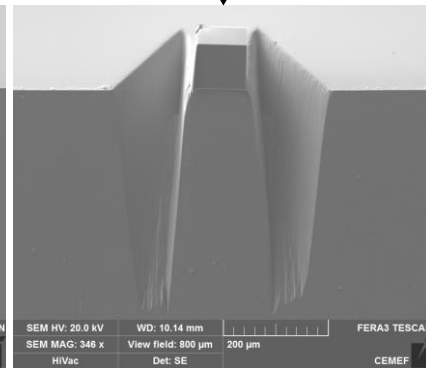
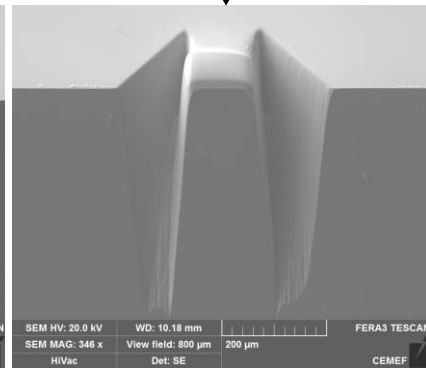
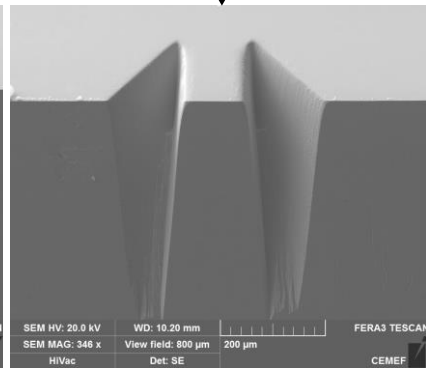
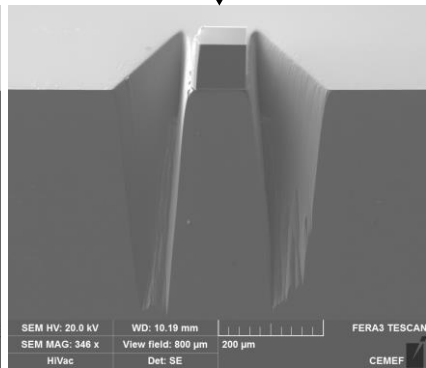
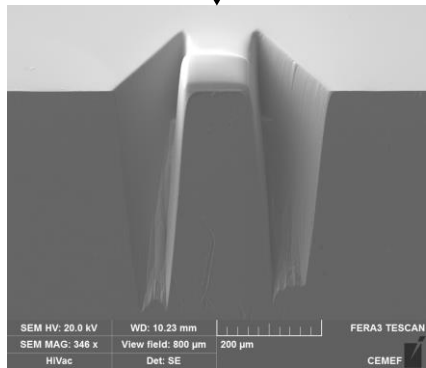
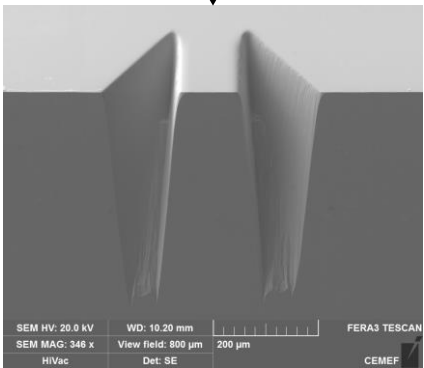
Pt dep.

Si mask

XeF<sub>2</sub>

Pt dep. + XeF<sub>2</sub>

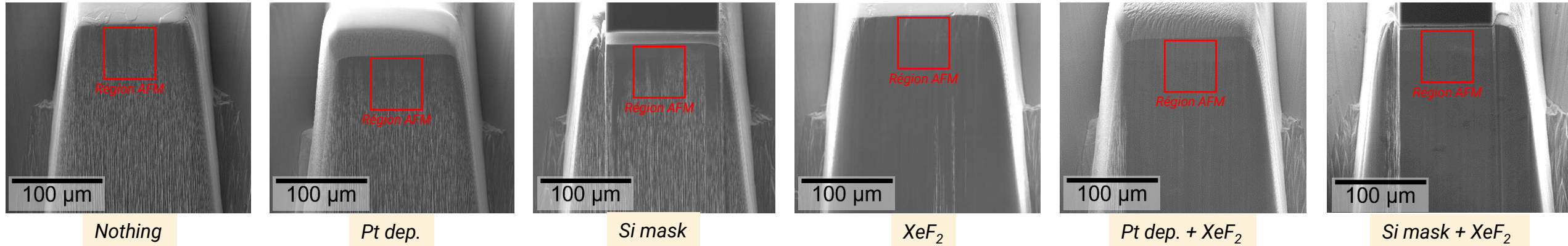
Si mask + XeF<sub>2</sub>



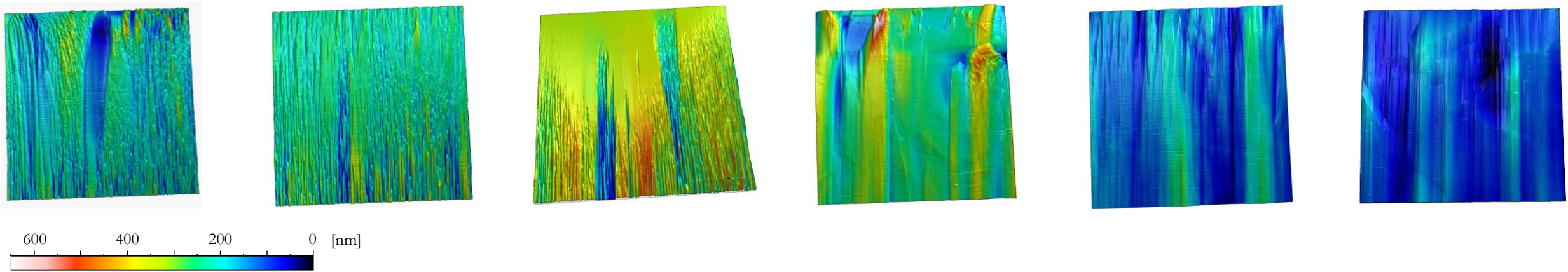
# OPTIMISATION DES CONDITIONS OPÉRATOIRES CAS DES SUPERALLIAGES BASE NICKEL

## Polissage FIB 30kV-1 $\mu$ A

Imagerie SE de la cross-section



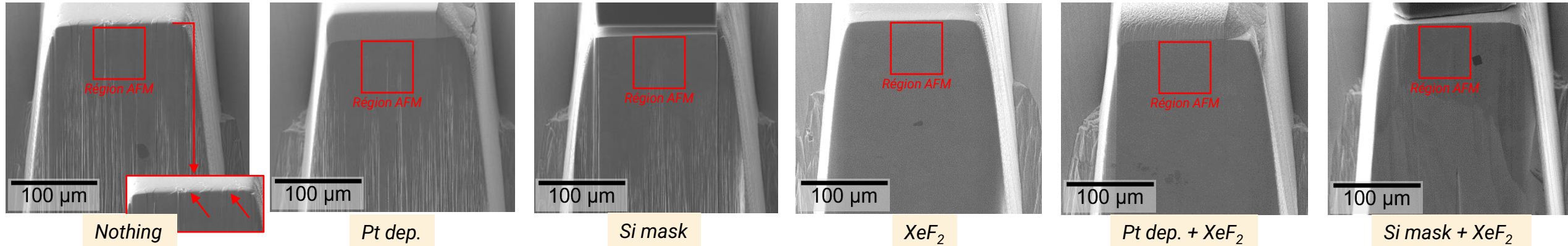
Mesures AFM



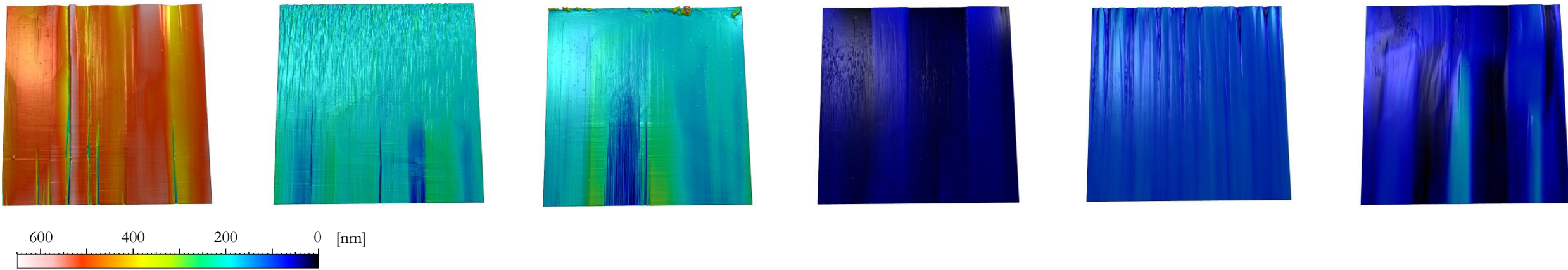
# OPTIMISATION DES CONDITIONS OPÉRATOIRES CAS DES SUPERALLIAGES BASE NICKEL

## Polissage FIB 30kV-100nA

Imagerie SE de la cross-section



Mesures AFM



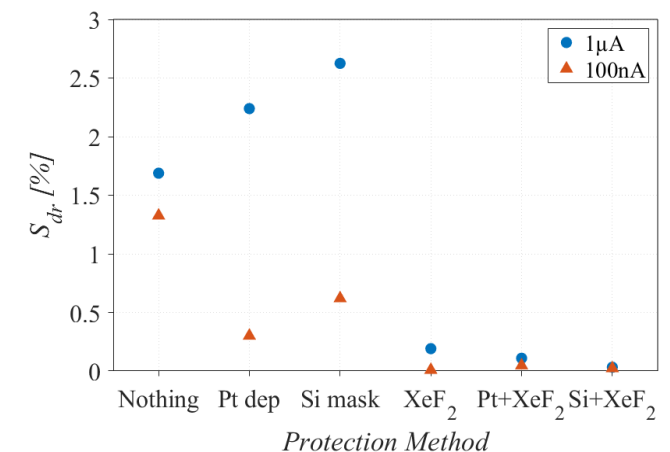
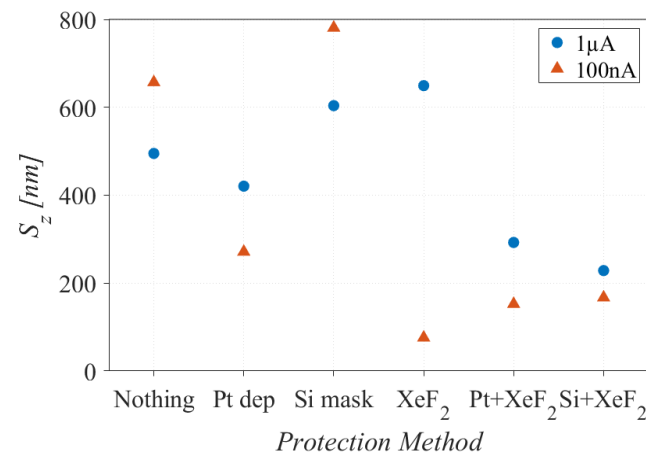
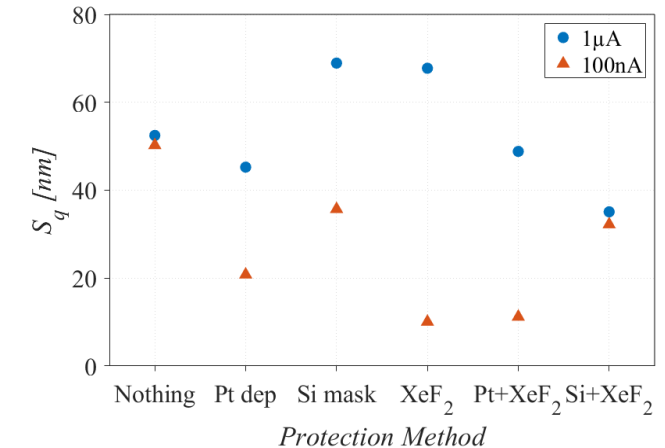
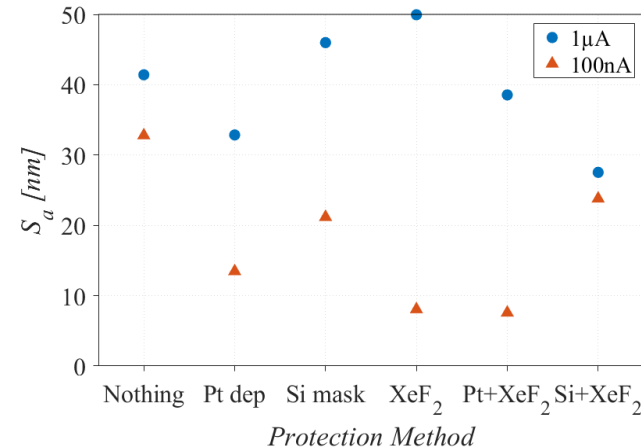
# OPTIMISATION DES CONDITIONS OPÉRATOIRES

## CAS DES SUPERALLIAGES BASE NICKEL

### Remarques

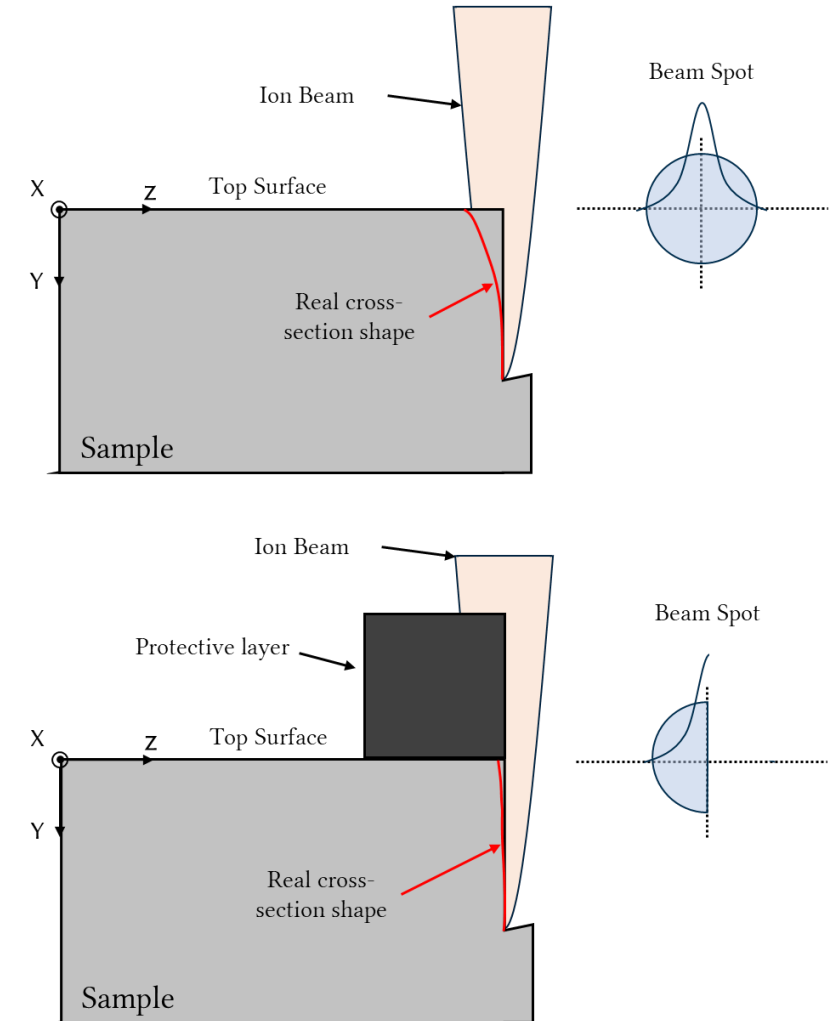
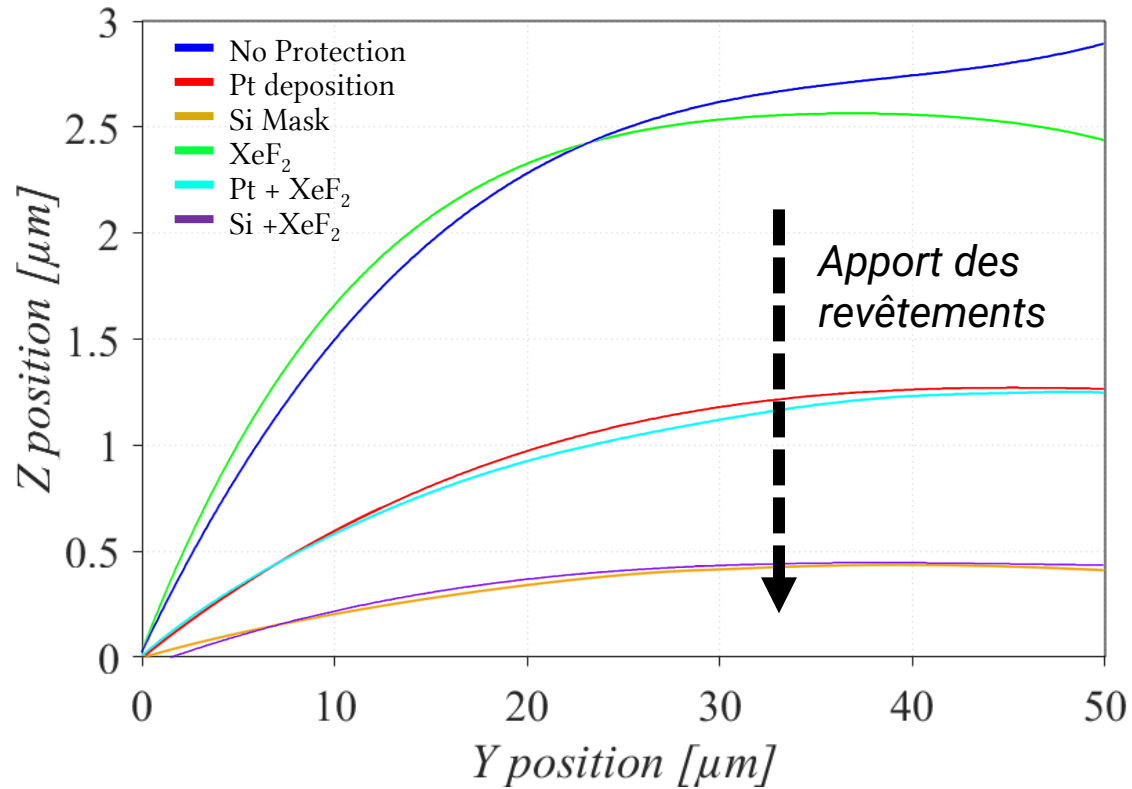
- Influence classique du courant observée : Niveau de curtaining diminue avec le courant
- XeF<sub>2</sub> surtout efficace à moyen courant (~100nA)
- Les meilleurs états de surface sont obtenus avec le fluor, plus petit gain supplémentaire en combinant avec un revêtement

### Paramètres classiques de rugosité surfacique



# OPTIMISATION DES CONDITIONS OPÉRATOIRES CAS DES SUPERALLIAGES BASE NICKEL

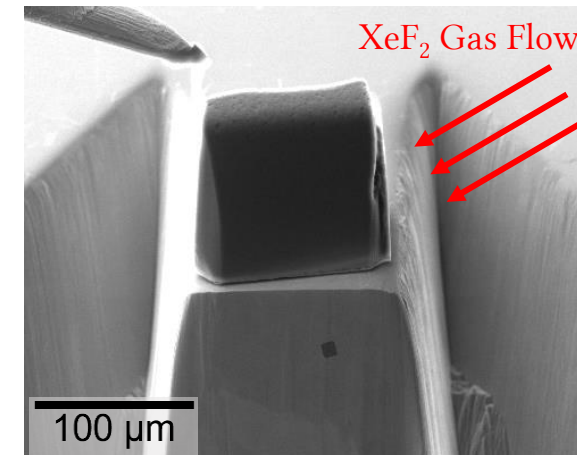
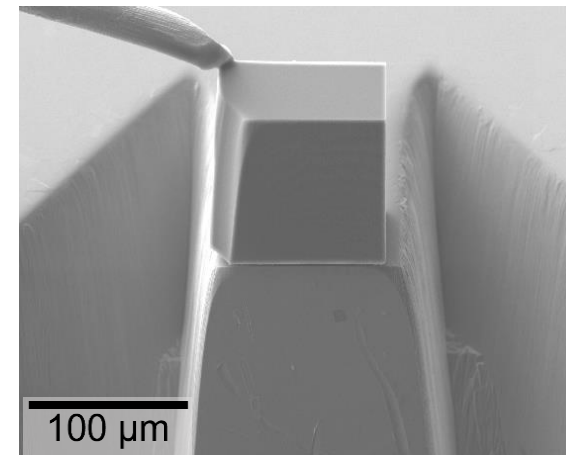
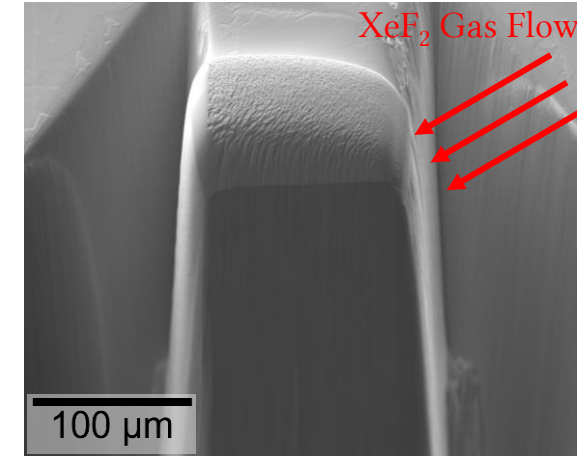
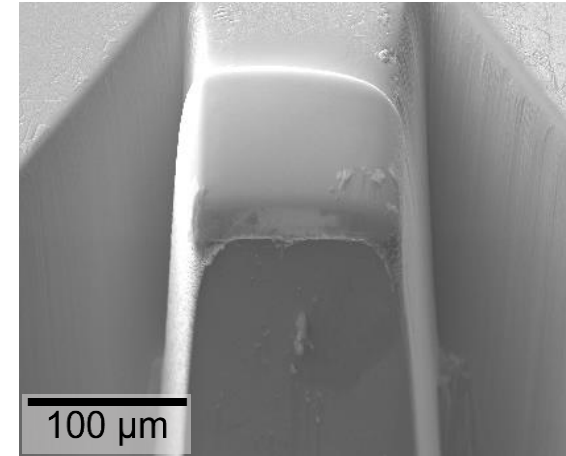
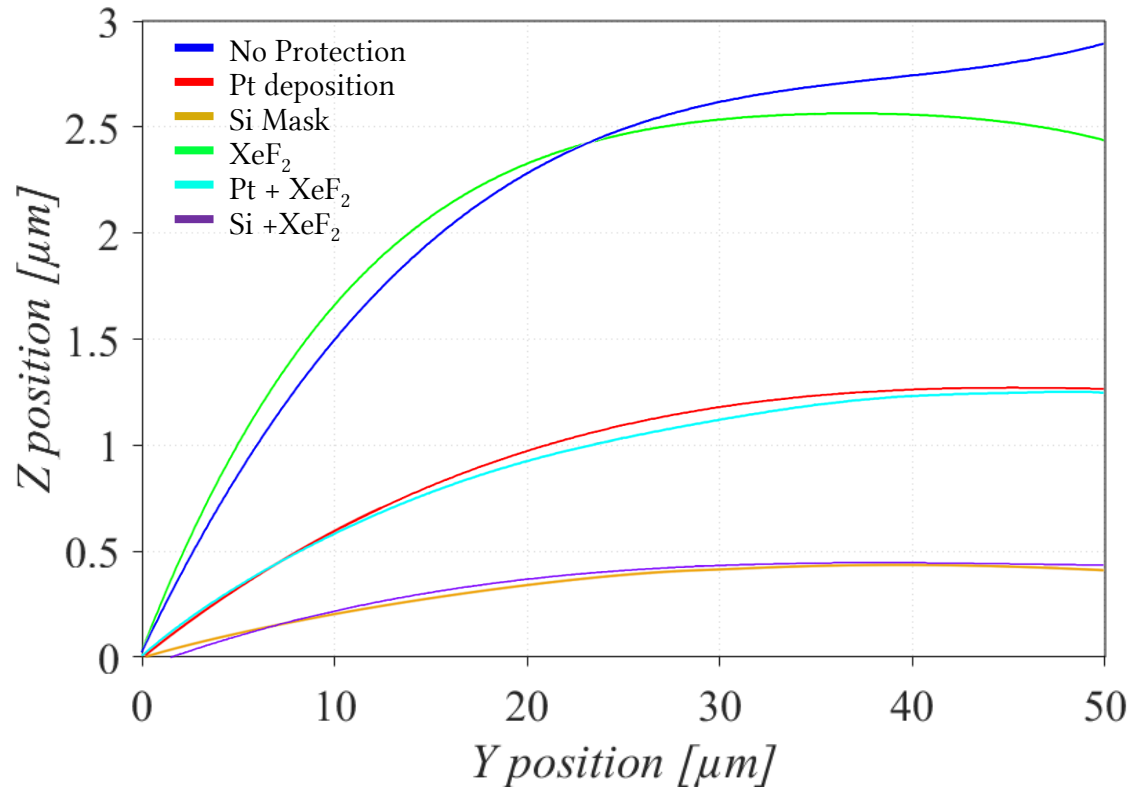
Profile de la cross-section après le polissage FIB 30kV-100nA



# OPTIMISATION DES CONDITIONS OPÉRATOIRES

## CAS DES SUPERALLIAGES BASE NICKEL

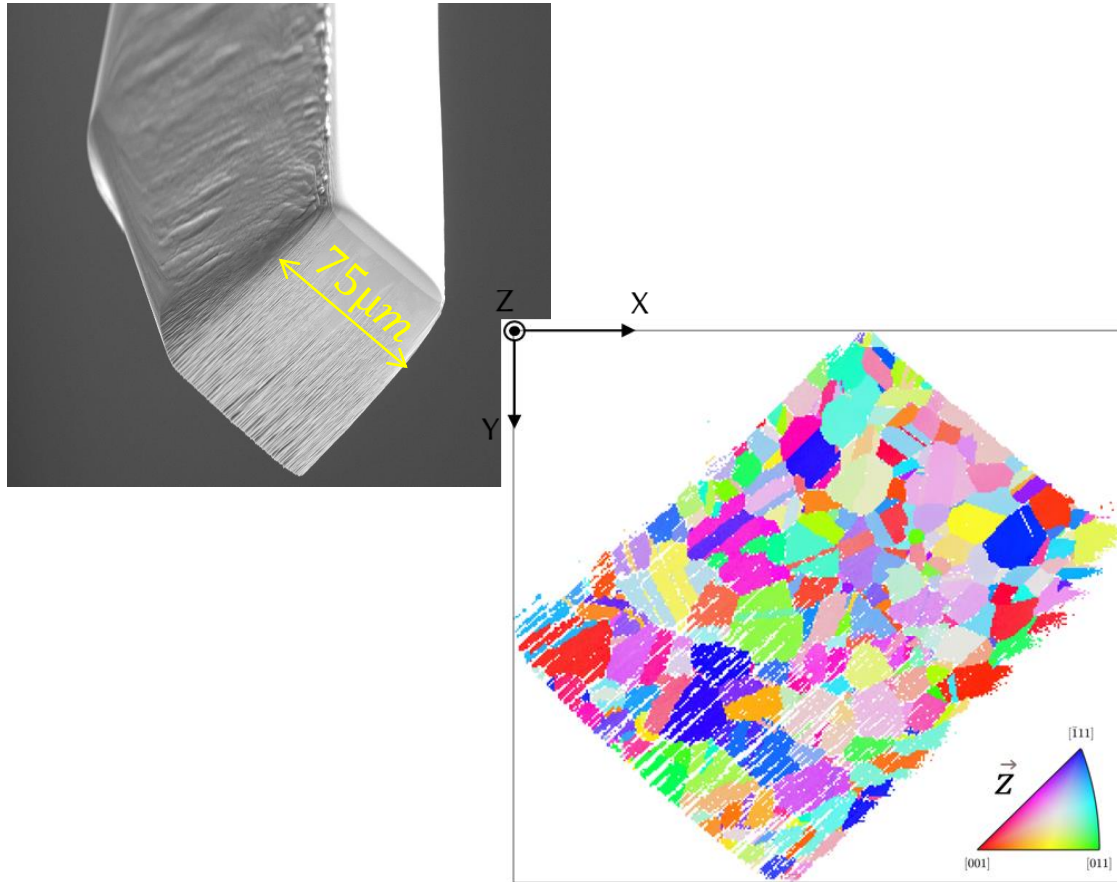
Profile de la cross-section après le polissage FIB 30kV-100nA



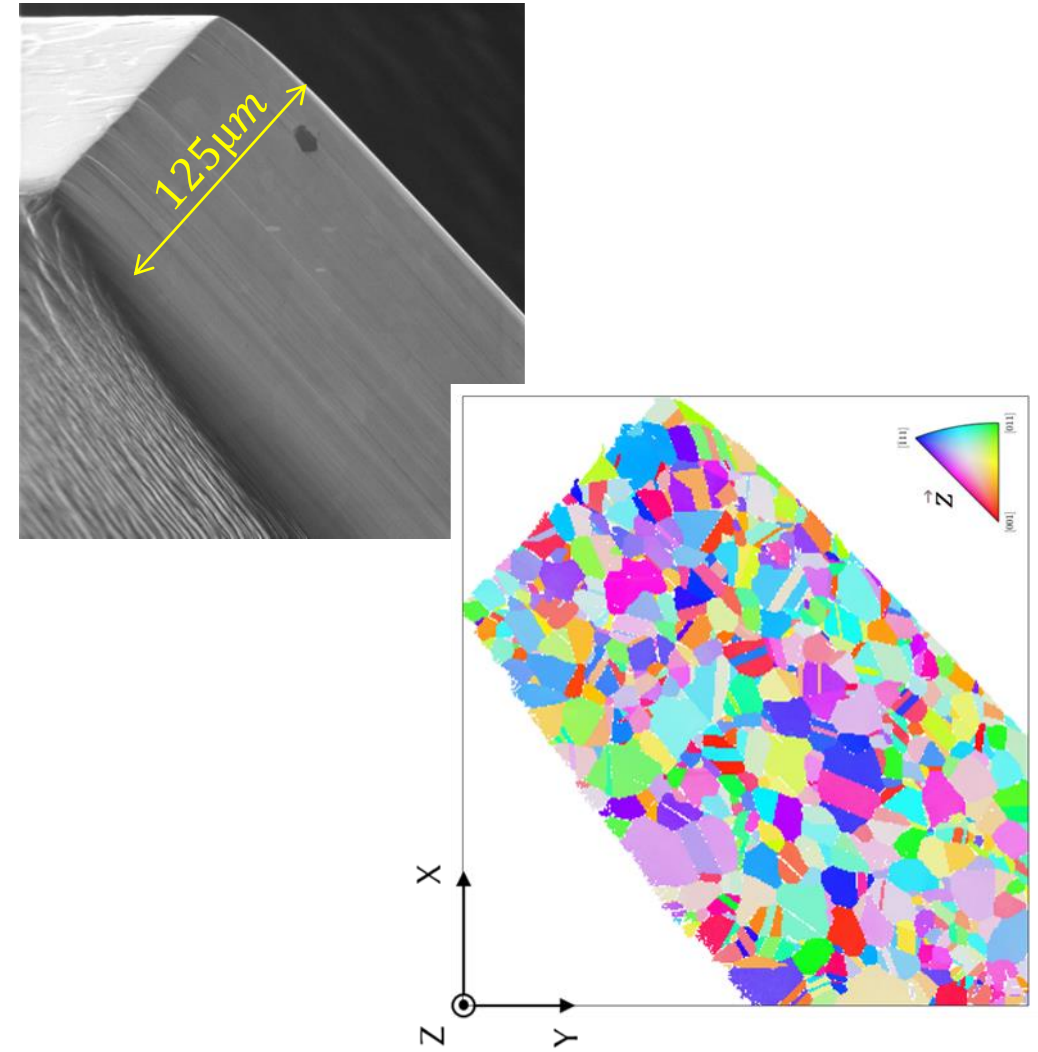
# OPTIMISATION DES CONDITIONS OPÉRATOIRES

## CAS DES SUPERALLIAGES BASE NICKEL

Slice EBSD pour un slicing FIB 30kV-30nA  
Dépôt Pt

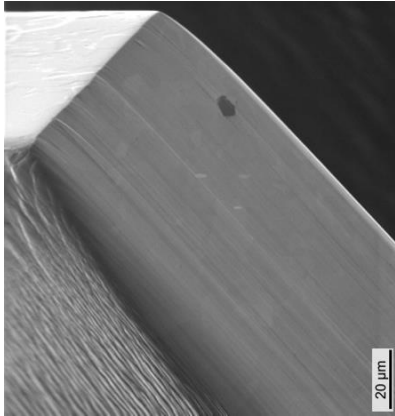


Slice EBSD pour un slicing FIB 30kV-300nA  
XeF<sub>2</sub>

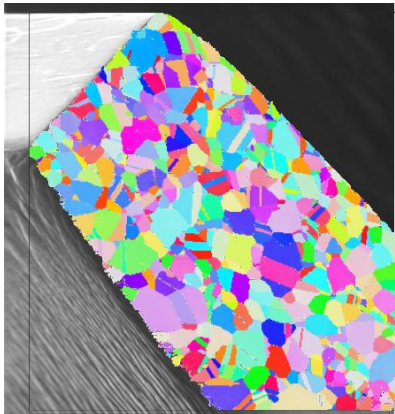


# EXEMPLES DE VOLUMES ANALYSÉS

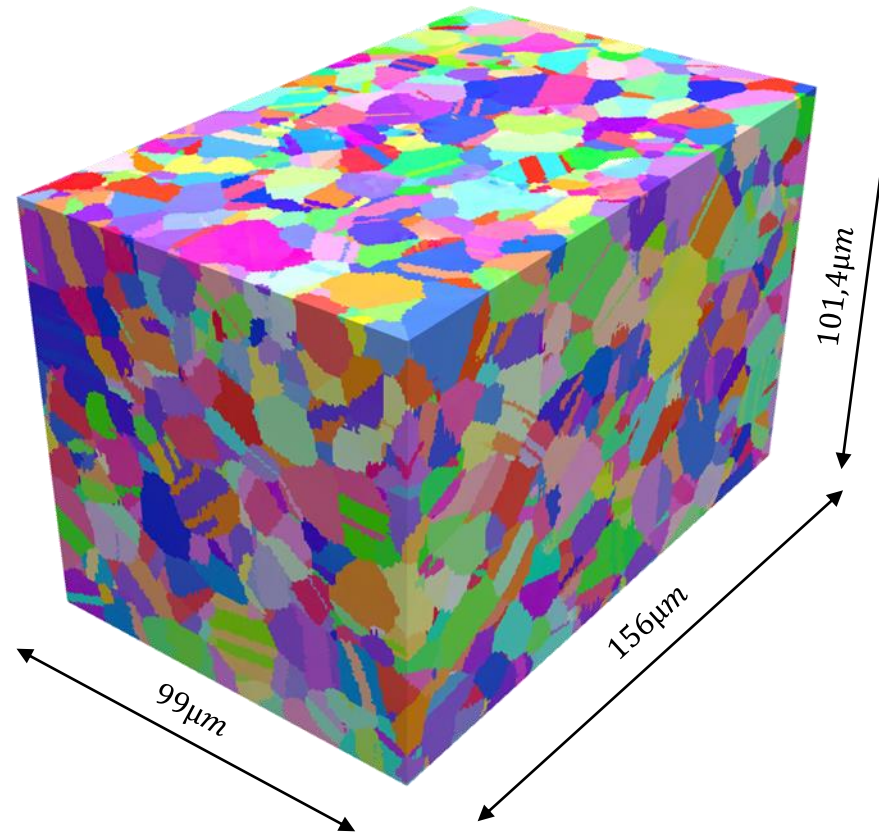
Slice SE



Slice EBSD



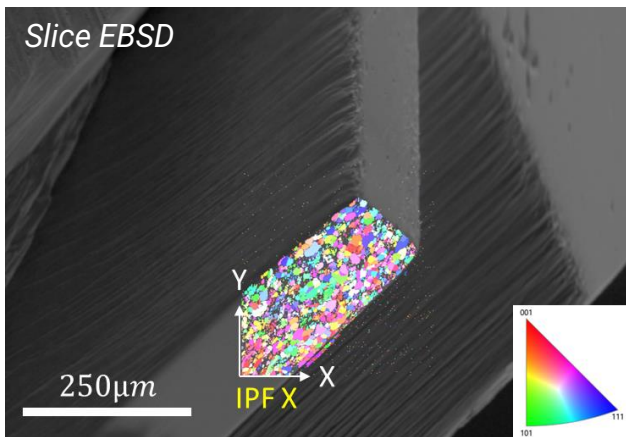
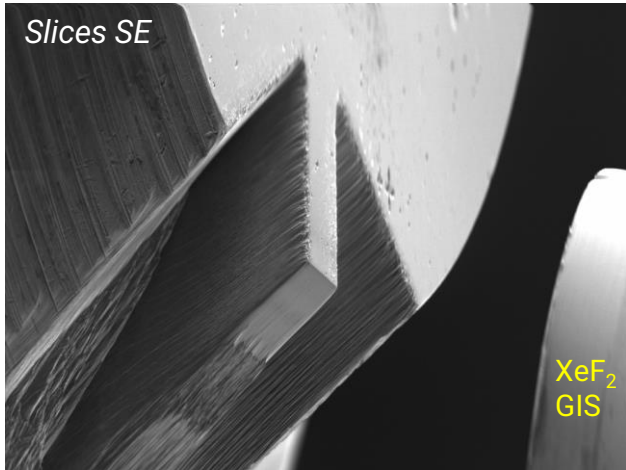
Inconel 718 (superalliage base Ni)  
Volume post-traité



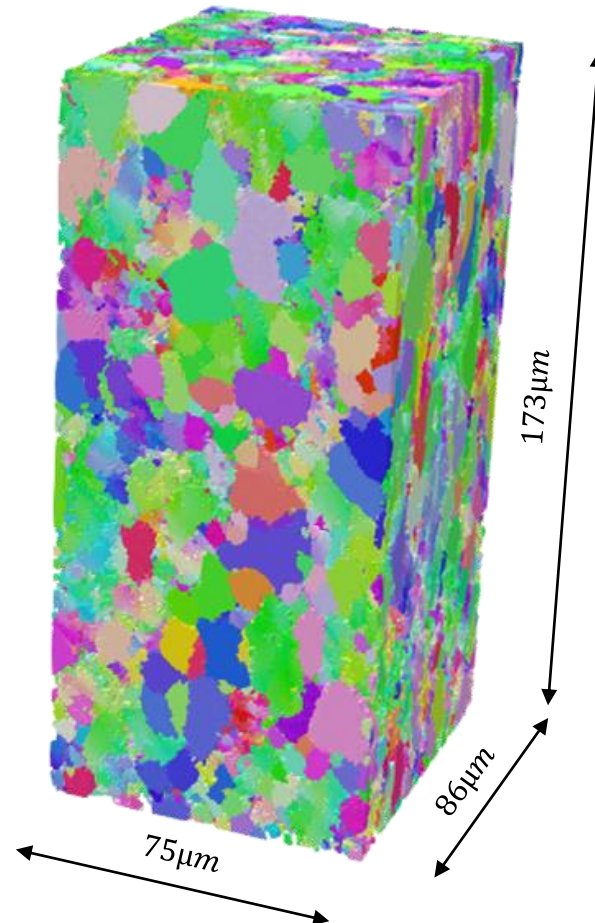
Conditions

Conditions FIB	$E_0 - I_0$	30kV – 276nA
	$z_{slice}$	0.6μm
	Sputt. rate	0.52μm <sup>3</sup> /nA / s
	Volume	99 × 156 × 101.4μm <sup>3</sup>
	$n_{slice}$	169
	Curtaining red.	XeF <sub>2</sub>
Conditions EBSD	$E_0 - I_0$	20kV – 5nA
	$\Delta x = \Delta y$	0.6μm
	Pattern reso.	156px × 128px
	Exposure time	7,4ms
	Averaging	1
	Indexing mode	standard Hough
	Indexing rate	99%
Temps	Total time	56h
	Slicing time	2'20/slice
	EBSD time	17'45/slice

# EXEMPLES DE VOLUMES ANALYSÉS



**MS-200 (acier de construction)**  
*Volume post-traité*



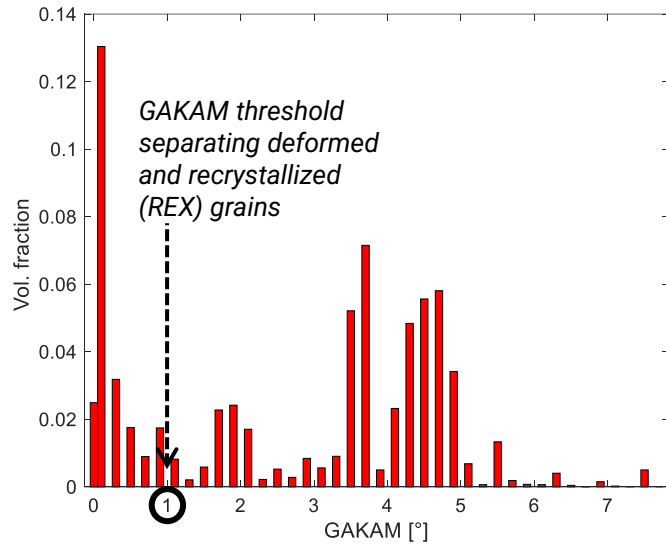
## Conditions

<b>Conditions FIB</b>	$E_0 - I_0$	30kV – 100nA
	$z_{slice}$	0.8μm
	<i>Sputt. rate</i>	0.23μm <sup>3</sup> /nA / s
	<i>Volume</i>	75 × 86 × 173μm <sup>3</sup>
	$n_{slice}$	108
	<i>Curtaining red.</i>	XeF <sub>2</sub>
<b>Conditions EBSD</b>	$E_0 - I_0$	20kV – 5nA
	$\Delta x = \Delta y$	0.8μm
	<i>Pattern reso.</i>	622px × 512px
	<i>Exposure time</i>	8,9ms
	<i>Averaging</i>	1
	<i>Indexing mode</i>	standard Hough
	<i>Indexing rate</i>	75%
<b>Temps</b>	<i>Total time</i>	≈ 55h
	<i>Slicing time</i>	11'30/slice
	<i>EBSD time</i>	21'40/slice

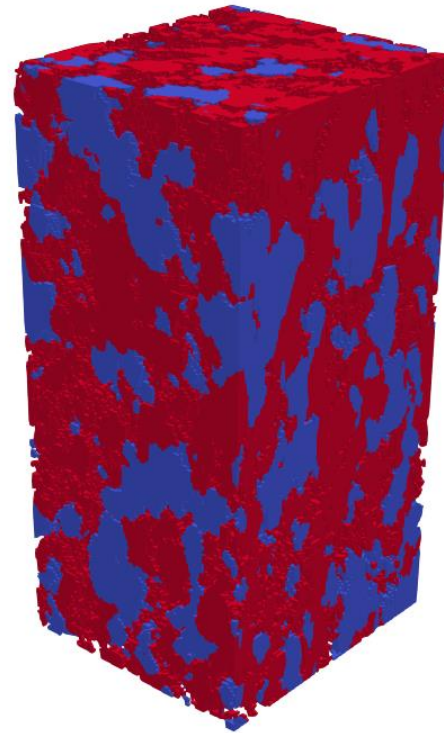
# EXEMPLES DE VOLUMES ANALYSÉS

## MS-200 (acier de construction) Volume post-traité

Identification des populations de grains déformés et recristallisés



GAKAM distribution



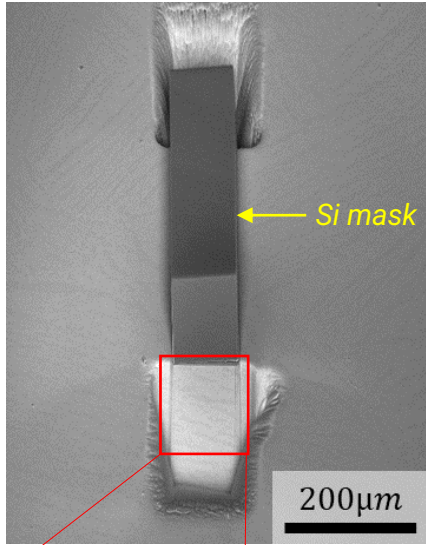
■ Deformed grains (Vol. Fract. ~52%\*)

■ REX grains (Vol. Fract. ~48%\*)

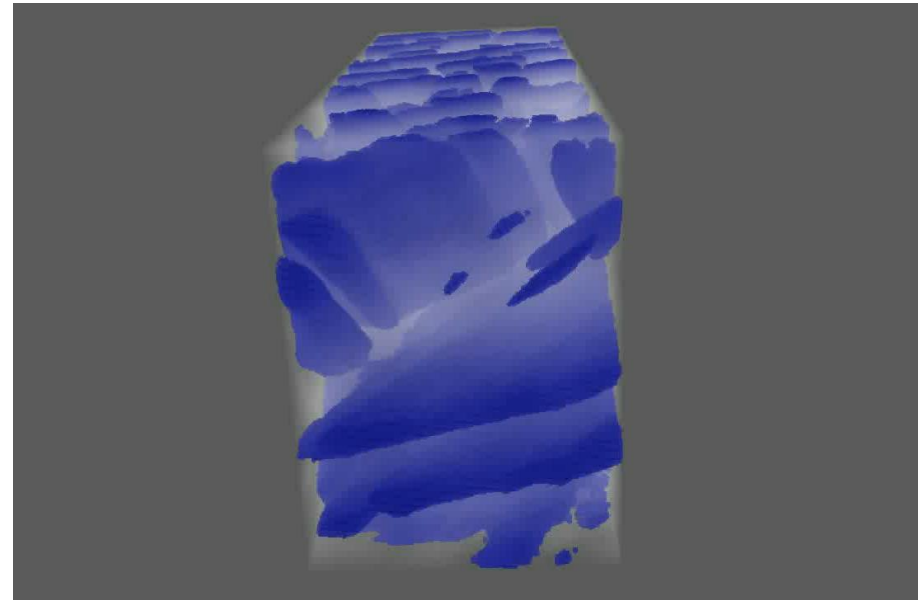
### Conditions

Conditions FIB	
$E_0 - I_0$	30kV – 100nA
$z_{slice}$	0.8 $\mu$ m
Sputt. rate	0.23 $\mu$ m <sup>3</sup> /nA / s
Volume	75 × 86 × 173 $\mu$ m <sup>3</sup>
$n_{slice}$	108
Curtaining red.	XeF <sub>2</sub>
Conditions EBSD	
$E_0 - I_0$	20kV – 5nA
$\Delta x = \Delta y$	0.8 $\mu$ m
Pattern reso.	622px × 512px
Exposure time	8,9ms
Averaging	1
Indexing mode	standard Hough
Indexing rate	75%
Temps	
Total time	≈ 55h
Slicing time	11'30/slice
EBSD time	21'40/slice

# EXEMPLES DE VOLUMES ANALYSÉS



TA6V (alliage de titane)  
Volume post-traité



■ Phase  $\alpha$  (lattes)  
■ Phase  $\beta$

Raw data

Processed data

Conditions

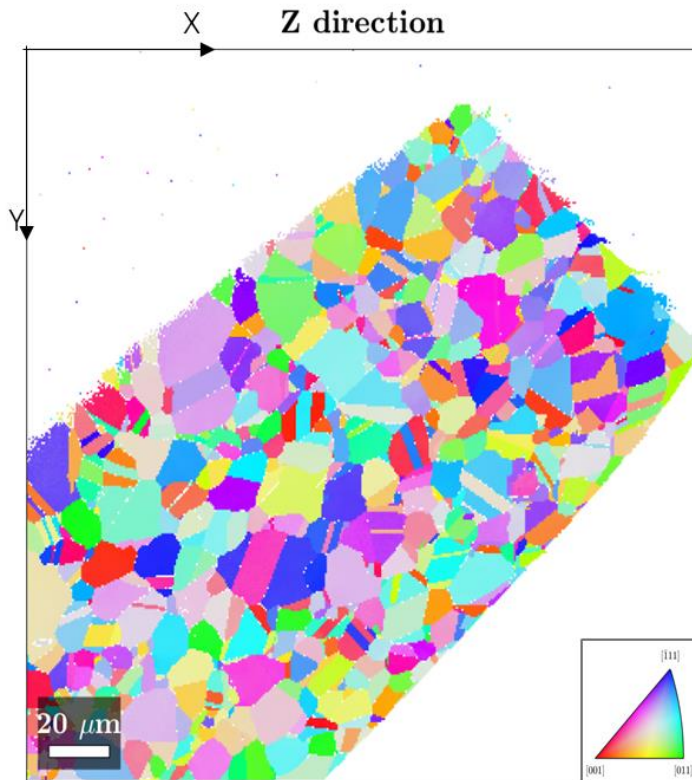
Conditions FIB	$E_0 - I_0$	30kV – 1 $\mu$ A
	$z_{slice}$	0.25 $\mu$ m
	Sputt. rate	0.41 $\mu$ m <sup>3</sup> /nA / s
	Volume	57 × 73 × 151 $\mu$ m <sup>3</sup>
	$n_{slice}$	658
	Curtaining red.	Si mask
Conditions Image	$E_0 - I_0$	20kV – 5nA
	$\Delta x = \Delta y$	0.25 $\mu$ m
	Image Résol.	316px × 509px
	Exposure time	32 $\mu$ s/px
	Imaging mode	BSE
Temps	Total time	≈ 12h
	Slicing time	0'12/slice
	Imaging time	0'51/slice

# CONCLUSIONS ET PERSPECTIVES

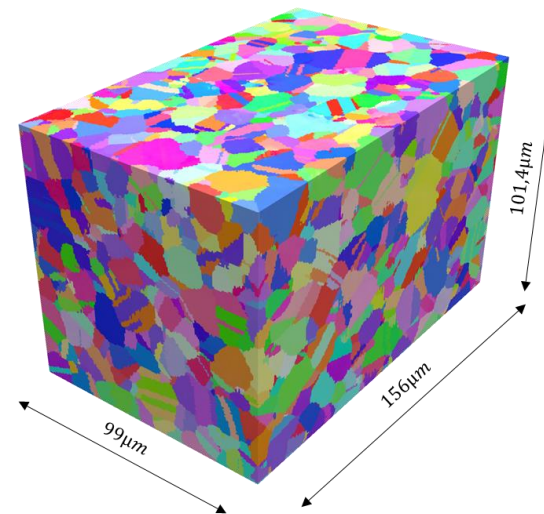
- Nécessité de travailler à courant FIB élevé (à partir de  $100nA$ ) pour l'analyse de « grands » volumes ( $\approx 100 \times 100 \times 100\mu m^3$ ) dans des temps raisonnables
- Le phénomène de curtaining peut être prononcé dans de telles conditions et même si les images peuvent être corrigées et/ou le système EBSD indexe dans ces zones il faut bien faire attention à la taille des objets d'intérêt par rapport à la profondeur du curtaining : on a tout intérêt à trouver des moyens de le réduire (dépôts, gazs, rocking stage,...)
- À courant de slicing élevé ( $\geq 100nA$ ) c'est le temps d'analyse EBSD qui devient limitant pour continuer de réduire la durée d'une analyse EBSD 3D

# CONCLUSIONS ET PERSPECTIVES

- Perte de temps pour chaque slice EBSD: 40% de la surface scannée est inutile (en dehors de la surface d'intérêt)

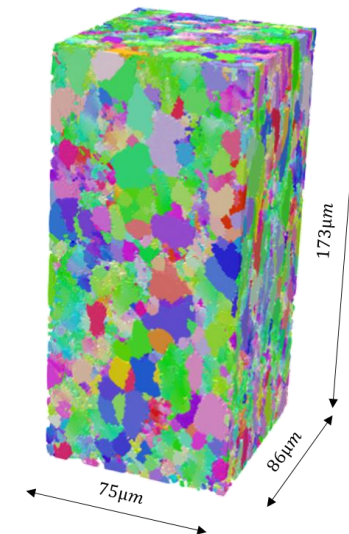


Inconel 718 (superaliage base Ni)  
Volume post-traité



*EBSD time* = ~~17'45~~ → 10'39  
**Total time = 56h → ≈ 36h**

MS-200 (acier de construction)  
Volume post-traité



*EBSD time* = ~~21'40~~ → 13'00  
**Total time = 55h → ≈ 44h**

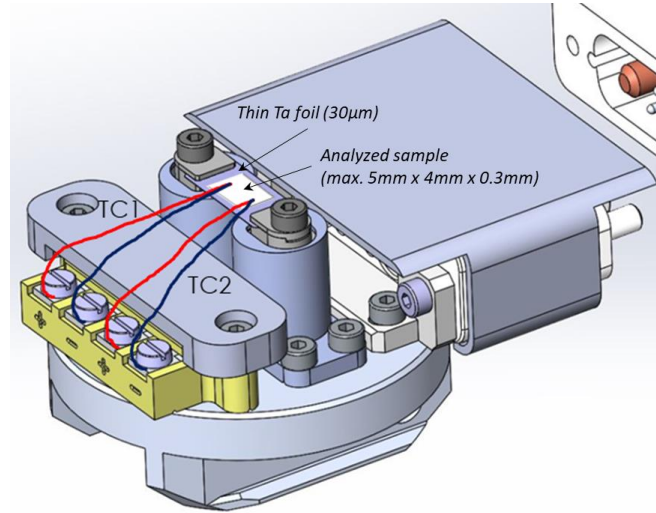
Merci

**QUESTIONS ?**

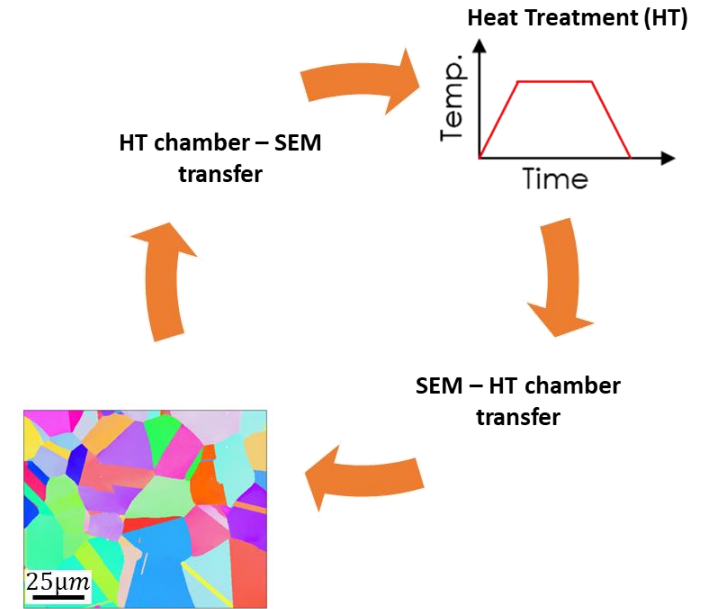
# HEAT TREATMENT CHAMBER FOR THE STUDY OF LOCAL PHENOMENA AT HIGH TEMPERATURE



## Heating stage



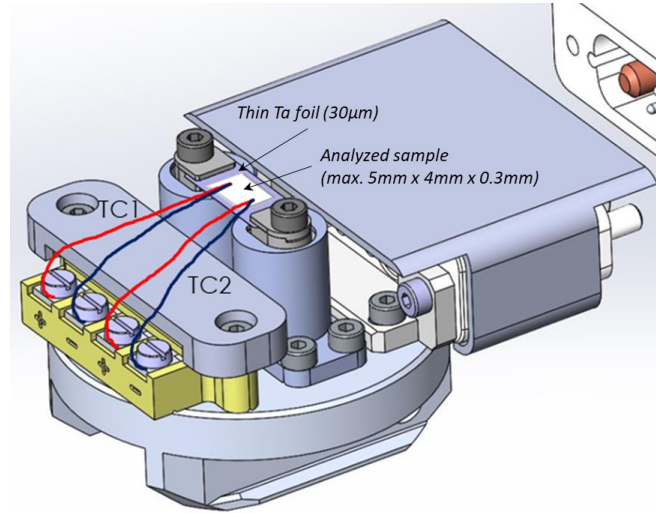
## Repeated sequence



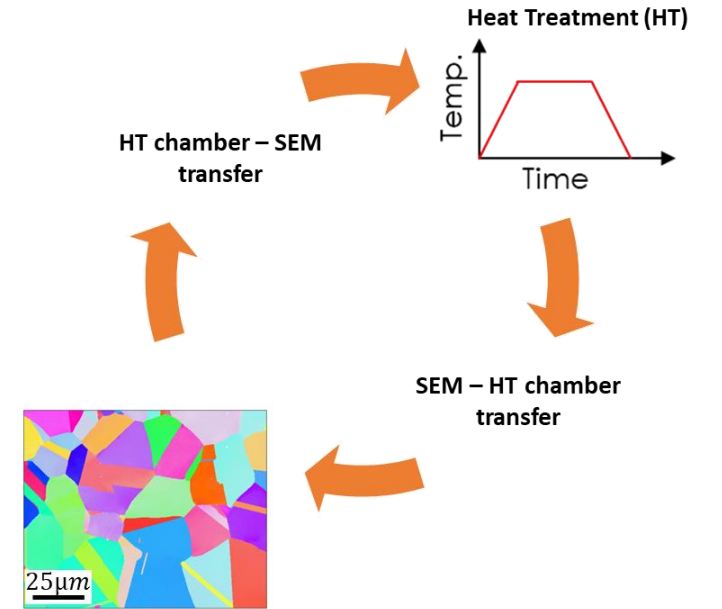
# HEAT TREATMENT CHAMBER FOR THE STUDY OF LOCAL PHENOMENA AT HIGH TEMPERATURE



## Heating stage

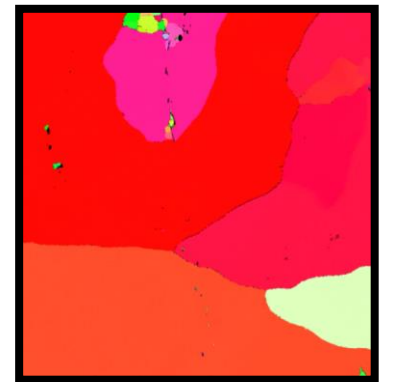
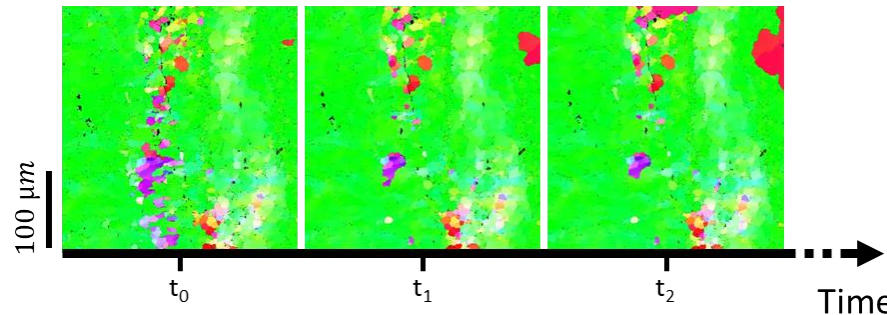


## Repeated sequence



## Example: REX in Al alloy

As rolled material

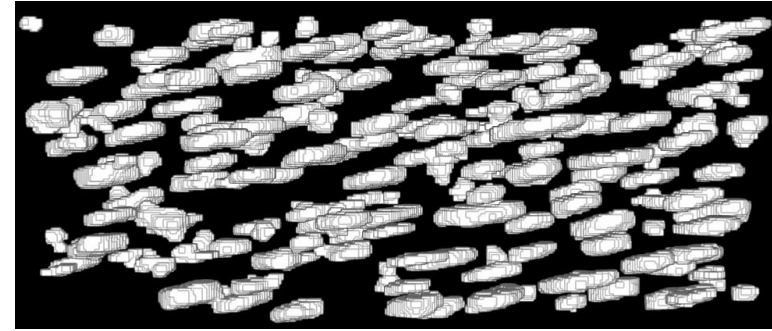
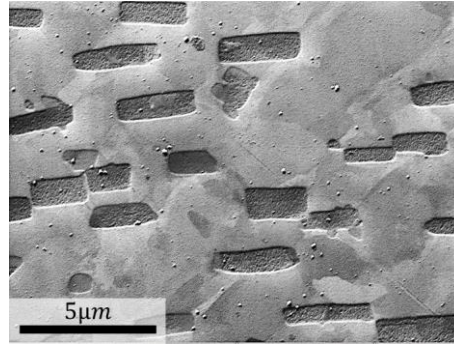


# PRECIPITATES 3D SHAPE ASSESSEMENT WITH P-FIB TOMOGRAPHY

**2D shape**

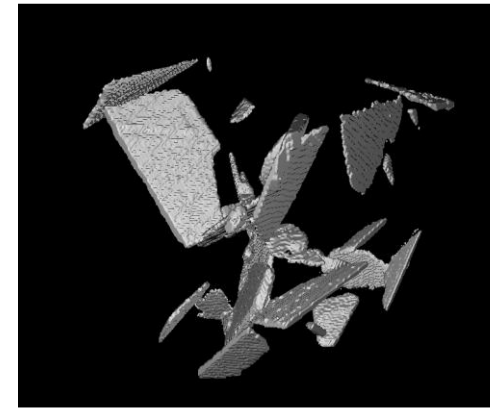
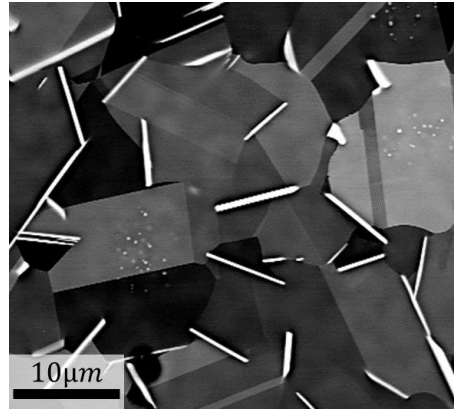
**3D shape (*processed and segmented data*)**

**"C- and T- type"  $\gamma'$  precipitates in AD730**



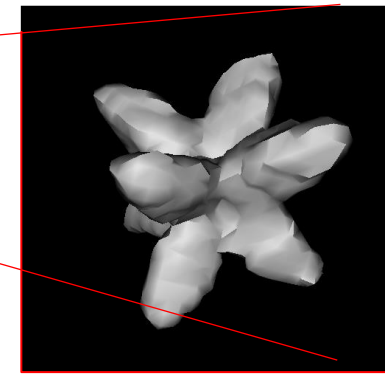
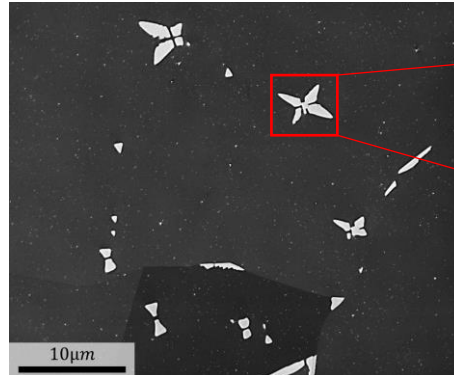
**Processed vol.  
~60 $\mu\text{m}$ x20 $\mu\text{m}$ x15 $\mu\text{m}$**

**$\eta/\delta$  precipitates in VDM780**



**Processed vol.  
~17.5 $\mu\text{m}$ x17.5 $\mu\text{m}$ x17.5  
 $\mu\text{m}$**

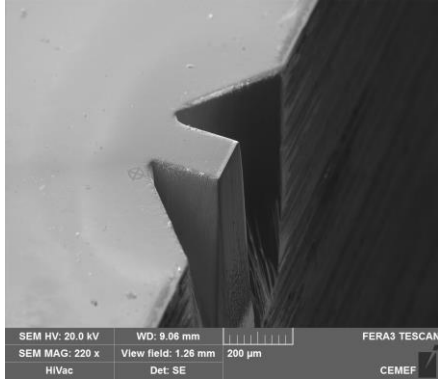
**$\gamma'$  precipitates in VDM780**



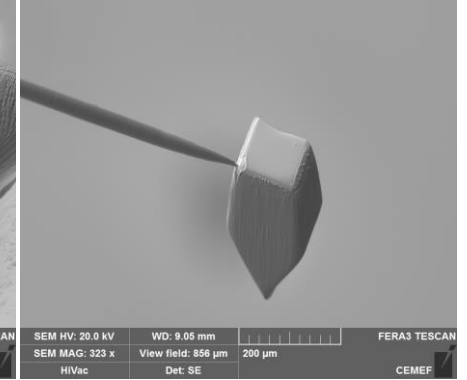
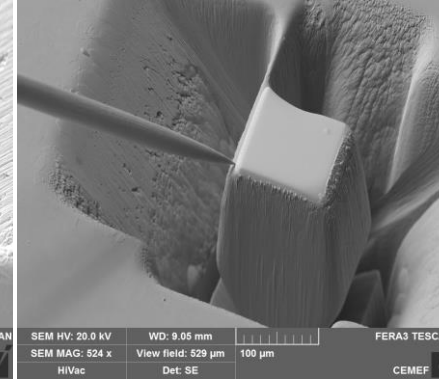
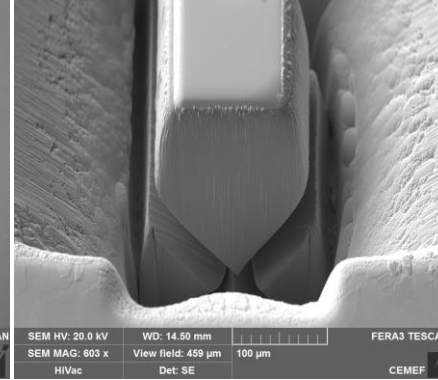
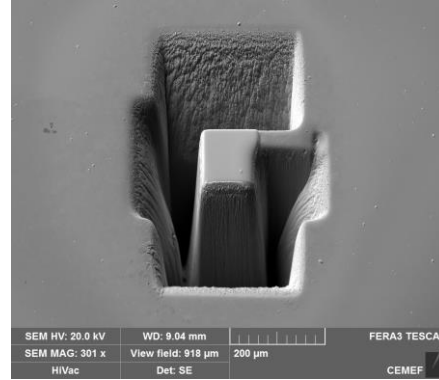
**Isolated particle  
~5 $\mu\text{m}$ x5 $\mu\text{m}$ x5 $\mu\text{m}$**

# 3D EBSD ANALYSES

## Sample edge



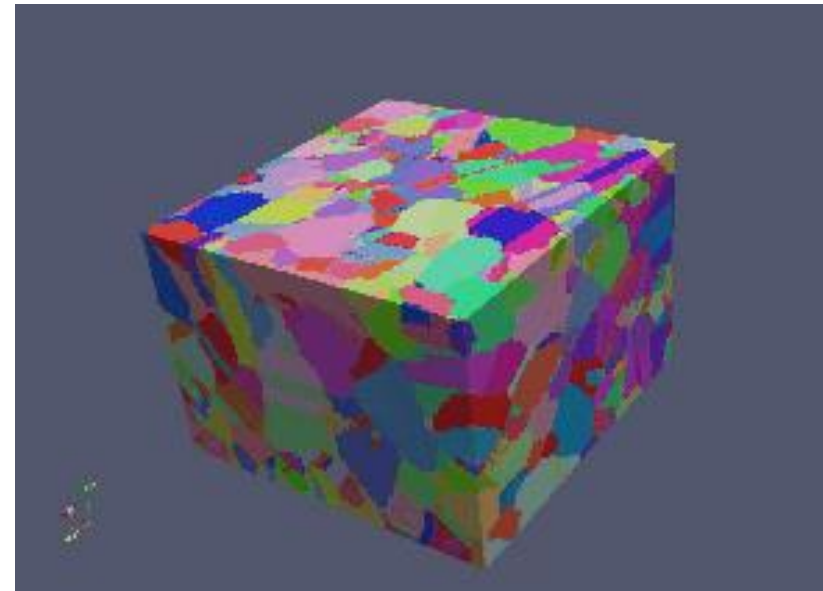
## Volume extraction



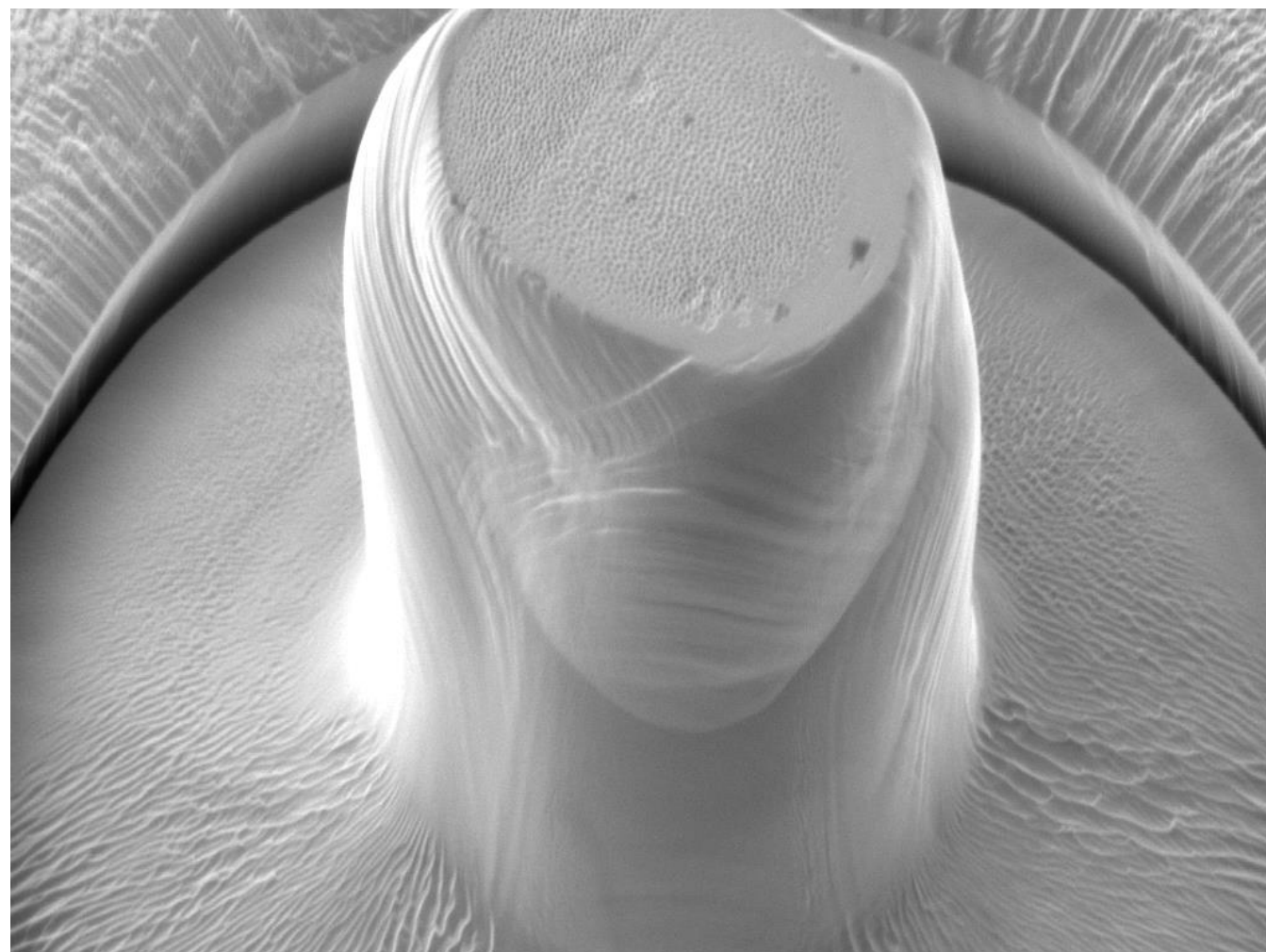
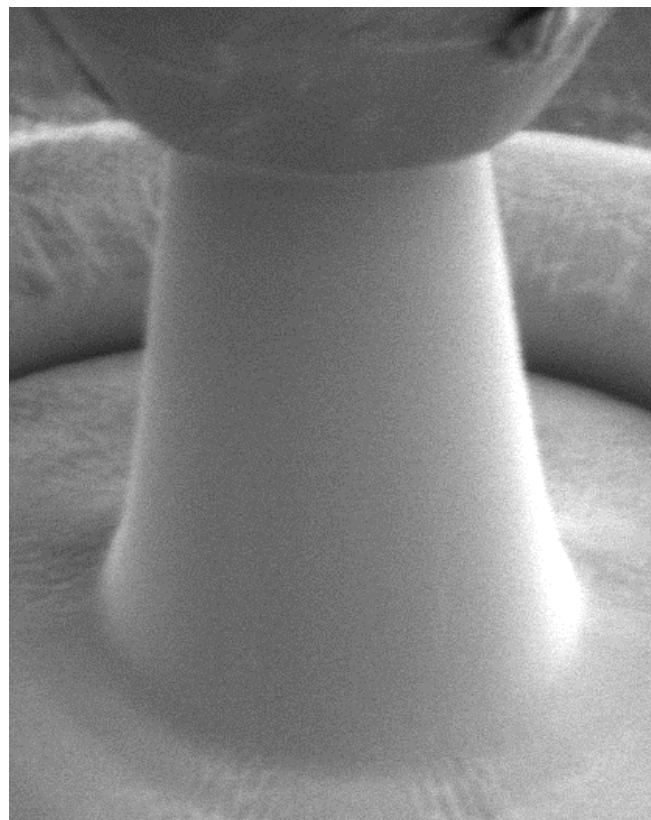
## After post-processing

**Example: Inconel 718**

**Processed vol.  
~75µm x 65µm x 55µm**



## Cuivre monocristallin (111)



SEM HV: 3.0 kV

WD: 29.76 mm

MAIA3 TESCAN

View field: 31.7 µm

Det: SE

10 µm

SEM MAG: 8.74 kx

Date(m/d/y): 08/24/21

CEMEF