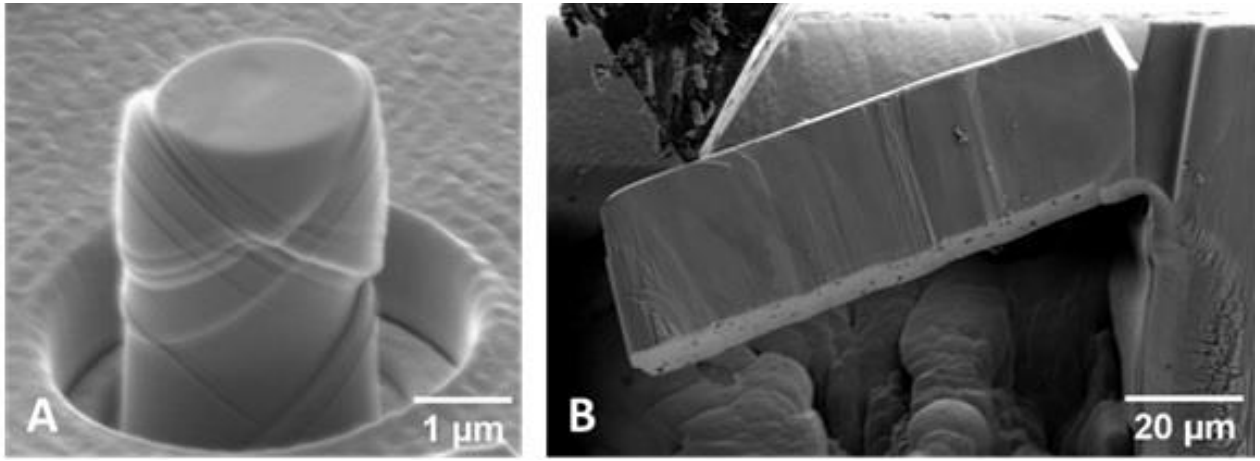
L’imagerie MEB au service de la (micro)-mécanique



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Présentation du GPM

Le laboratoire

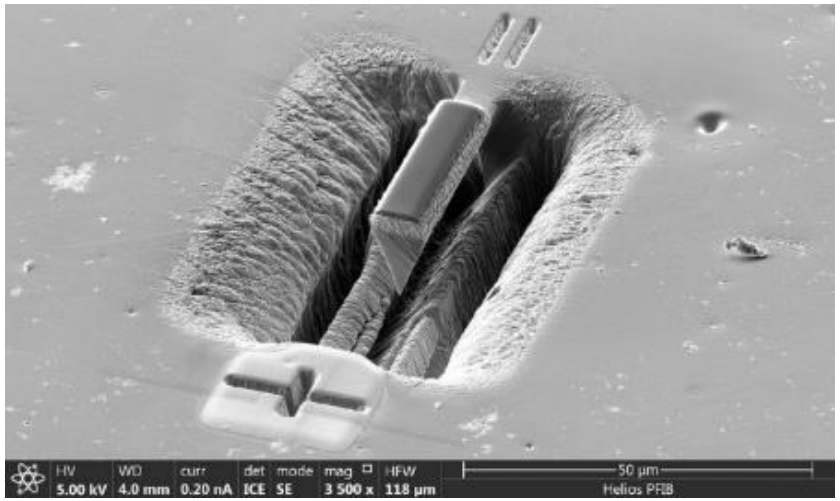
MEB FIB

Préparation d'échantillons

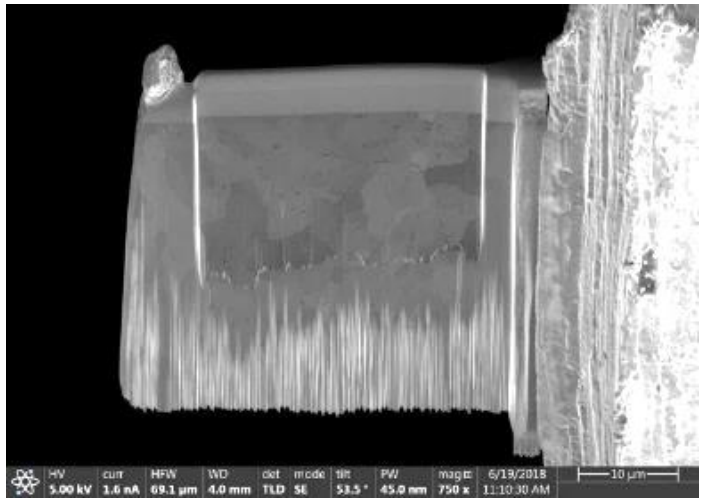
MET

SAT

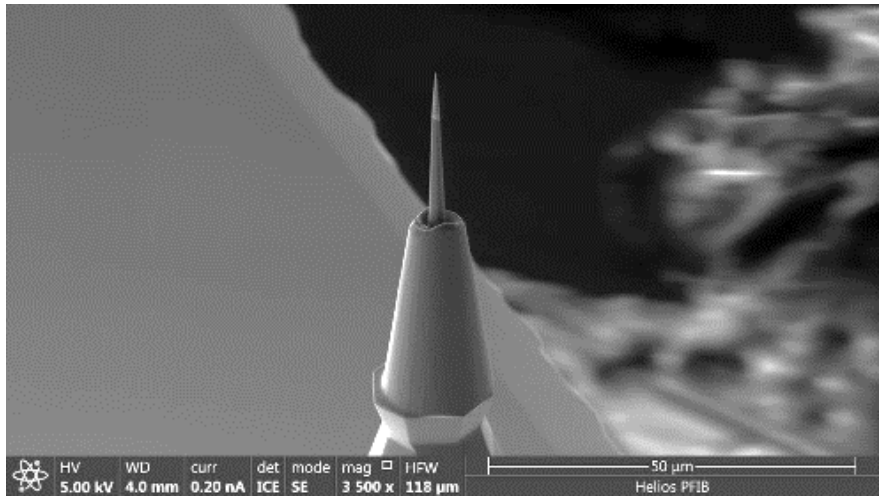
FIB – Usinage



FIB – Préparation de lame MET



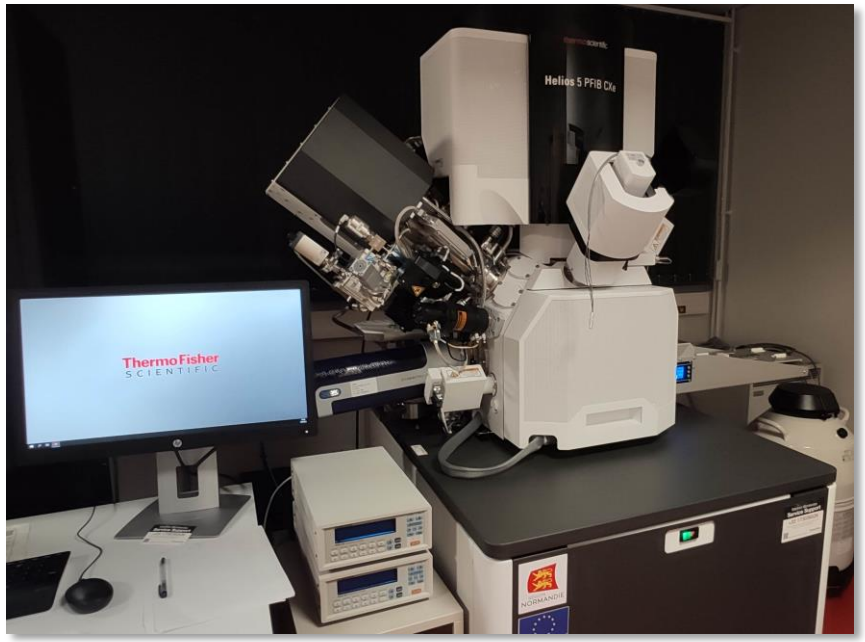
FIB – Préparation de pointe SAT



Présentation du GPM

MEB/FIB (Focused Ion Beam)

Gallium-FIB Helios G5 UX



Plasma-FIB Xenon Helios G5 CXe

MEB/FIB Gallium Zeiss XB540

EBSD, EDS…

Platine cryogénique + transfert UHV et/ou cryogénique vers SAT

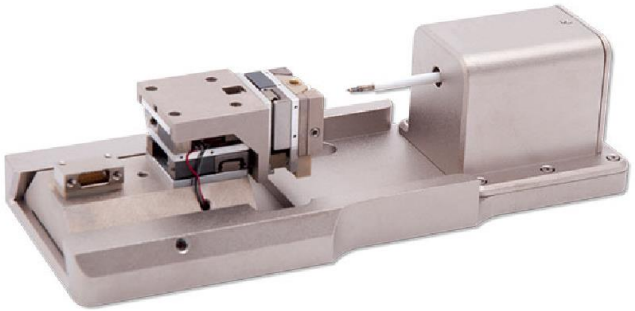
Nucléarisé

For Biological, "Air and/or Moisture Sensitive" Materials analysis :

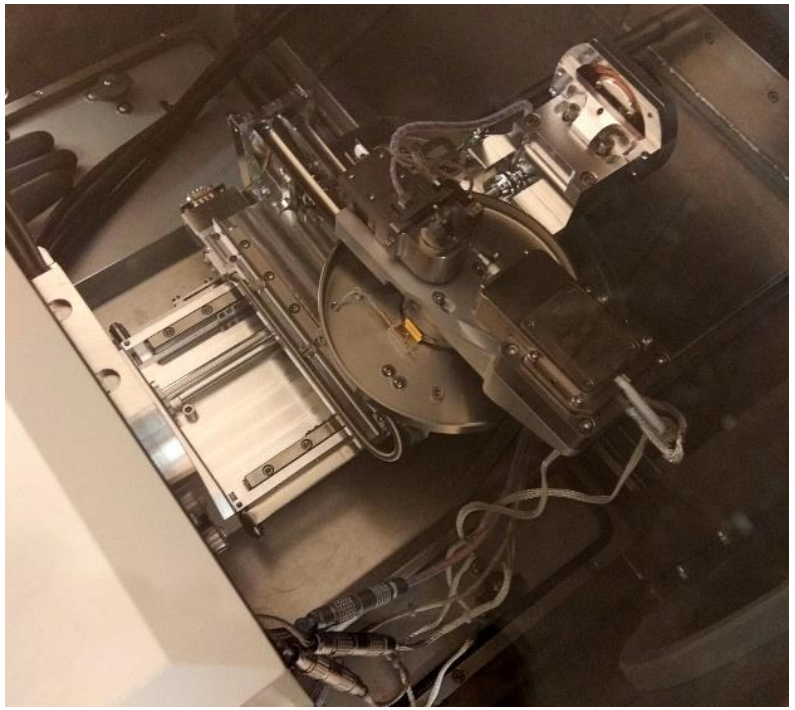
Battery and fuel cell Research, Catalytic materials, Surface reaction (oxydation, corrosion,…), Thin Films, PV (Selenium based), O(rganic)LED,….

Présentation du GPM

Essais micro-mécaniques

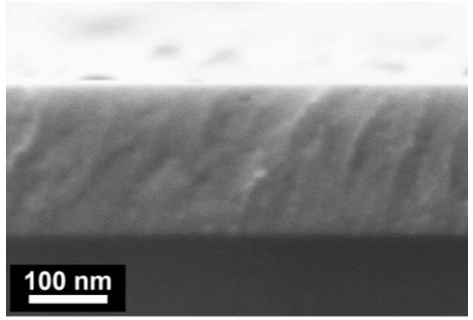


Bruker Hysitron PI85
SEM Pico Indenter

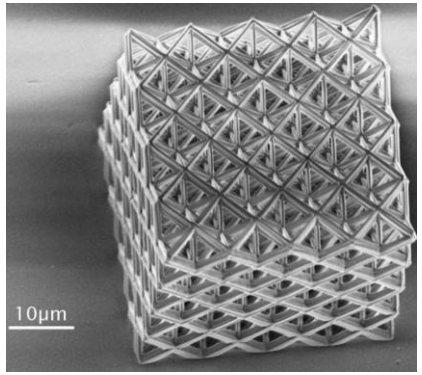


Placement sur la platine du MEB/FIB

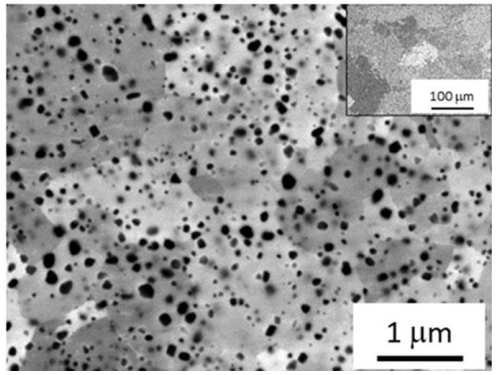
Introduction



Revêtements fins



Miniaturisation

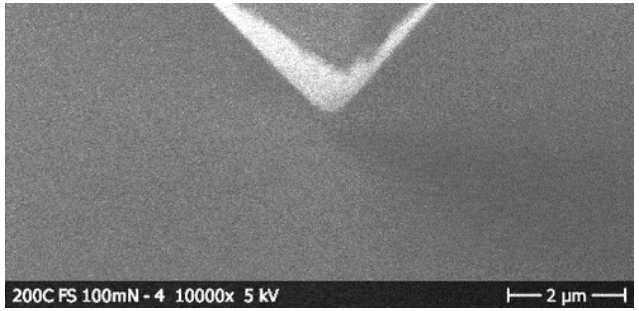


Microstructure

Introduction

Essais micromécaniques

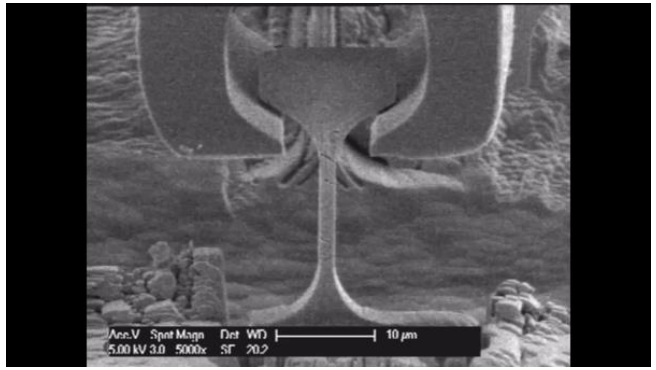
Nano-indentation



+ Préparation facile
+ Essais rapides

- Analyse complexe
- Quantitatif ?

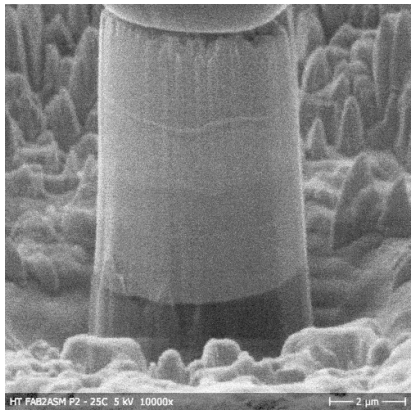
Micro-traction



++ Analyse

-- Préparation
-- Essais complexes

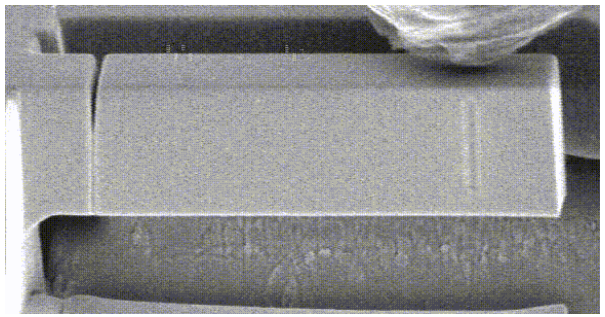
Micro-compression



+ Analyse

- Préparation

Micro-flexion



+ Analyse

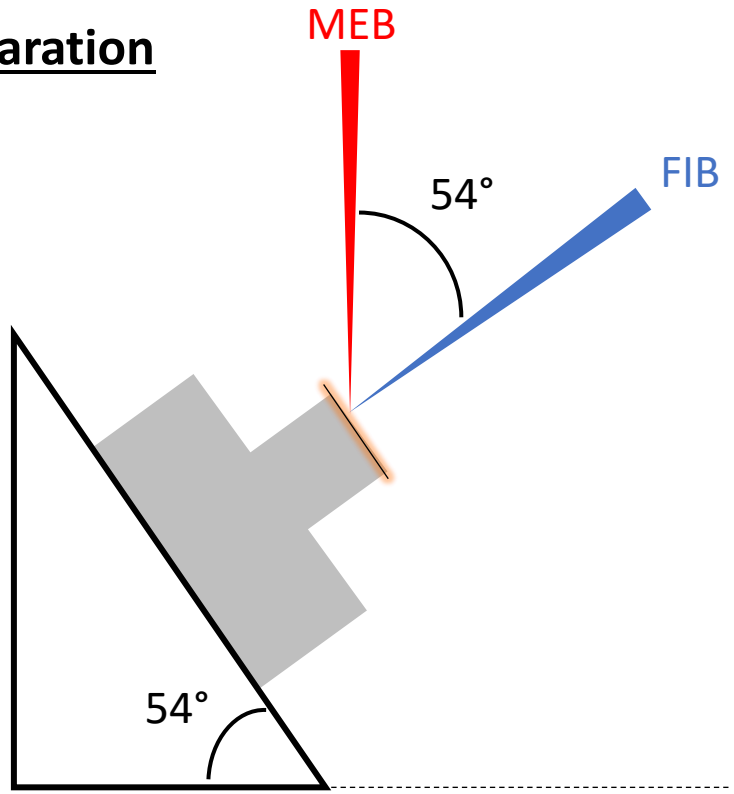
- Préparation longue
- Essais complexes

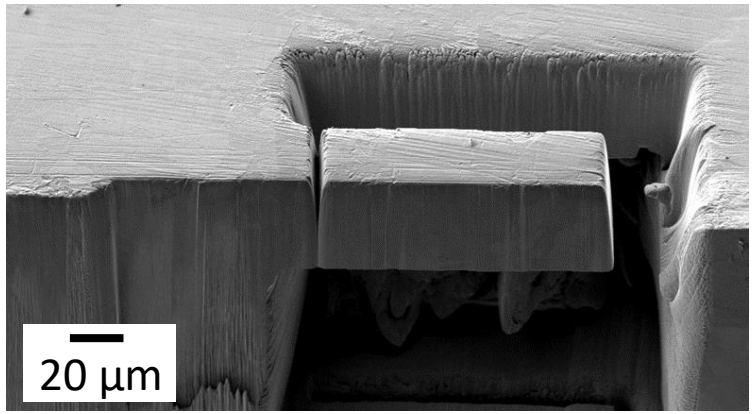
https://alemnis.com/

Essais de micro-flexion

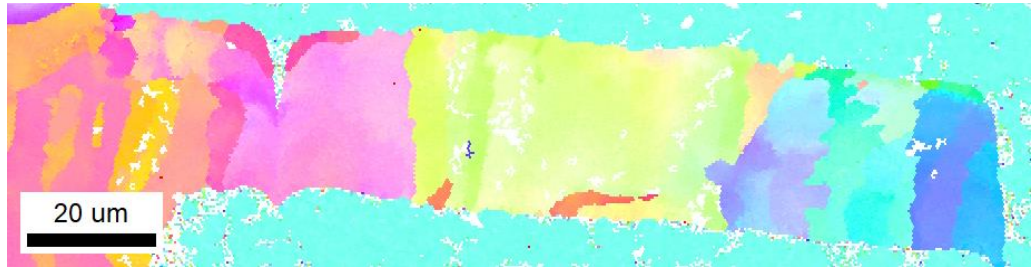
Essais de micro-flexion

Préparation





FIB milling

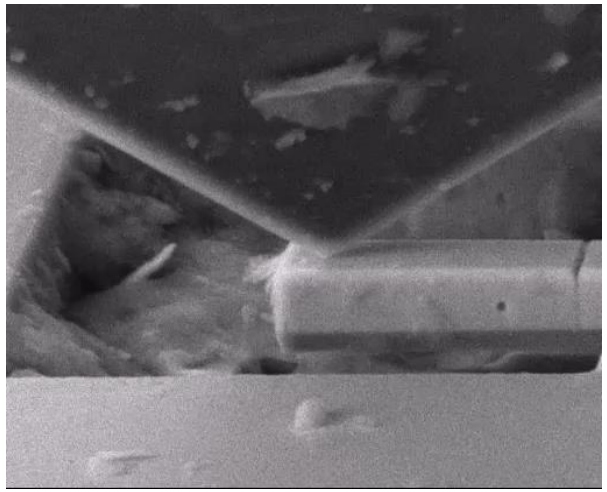


EBSD characterization

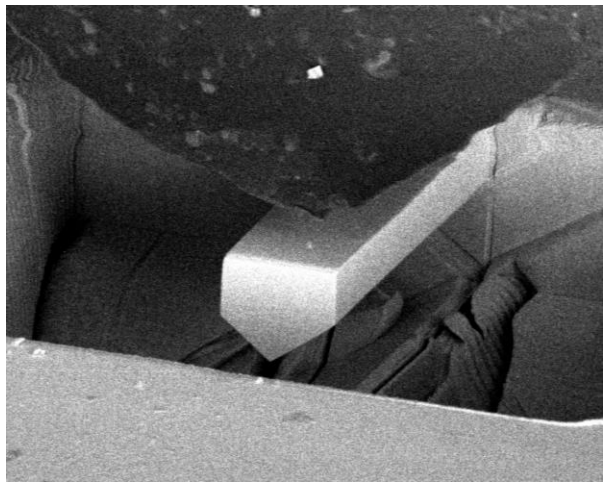
Essais de micro-flexion

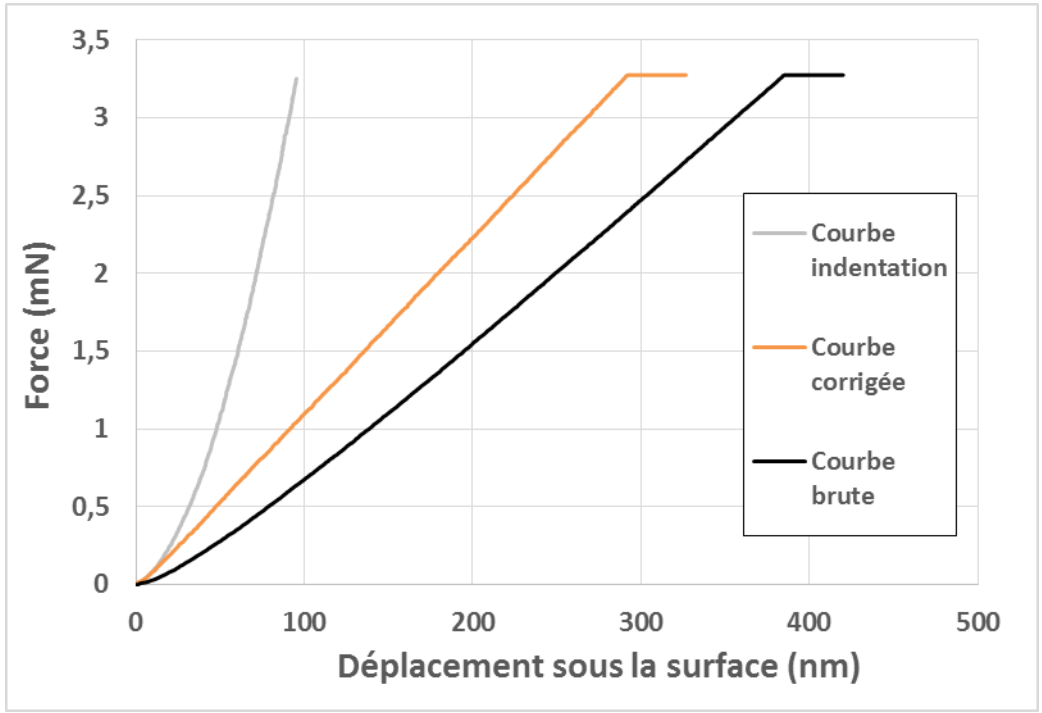
Sollicitation

Propagation <u>instable</u> de fissure : imagerie *in situ* peu intéressante



Videos (SEM) of micro-bending test on UO_2 fuels





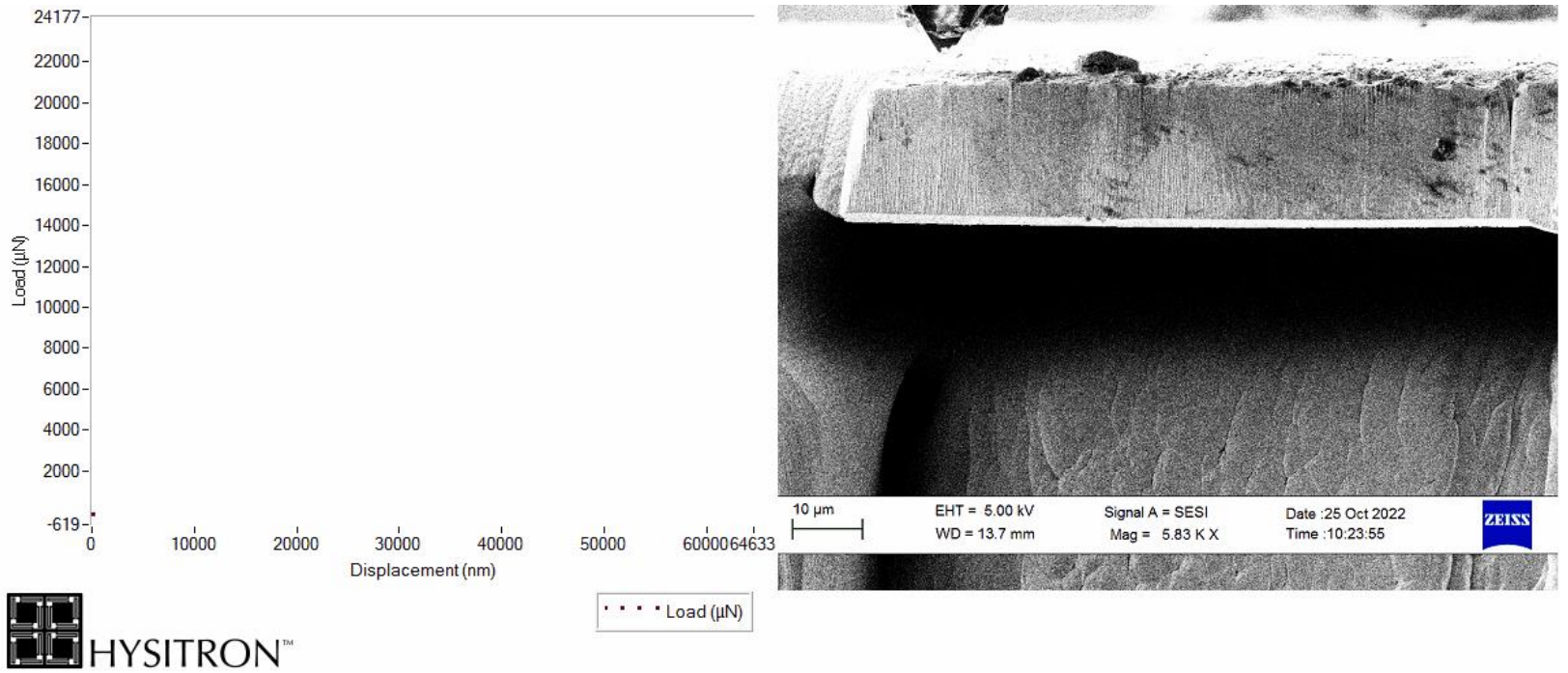
Force-displacement curves recorded in a micro-bending test

Essais de micro-flexion

Flexion *in situ :* film

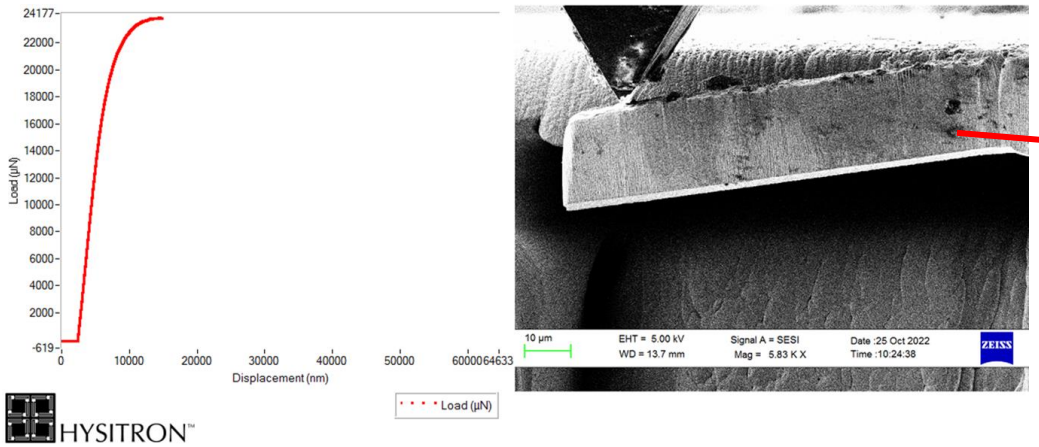
Propagation stable de fissure : imagerie *in situ* intéressante

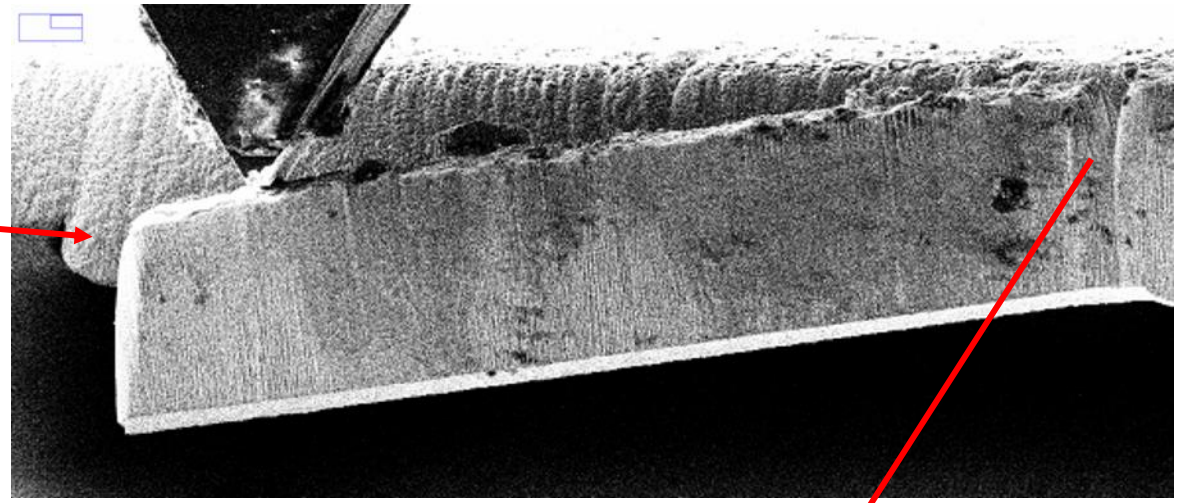
- Film : 1919 images et 0,10s/image



Essais de micro-flexion

Flexion *in situ :* film





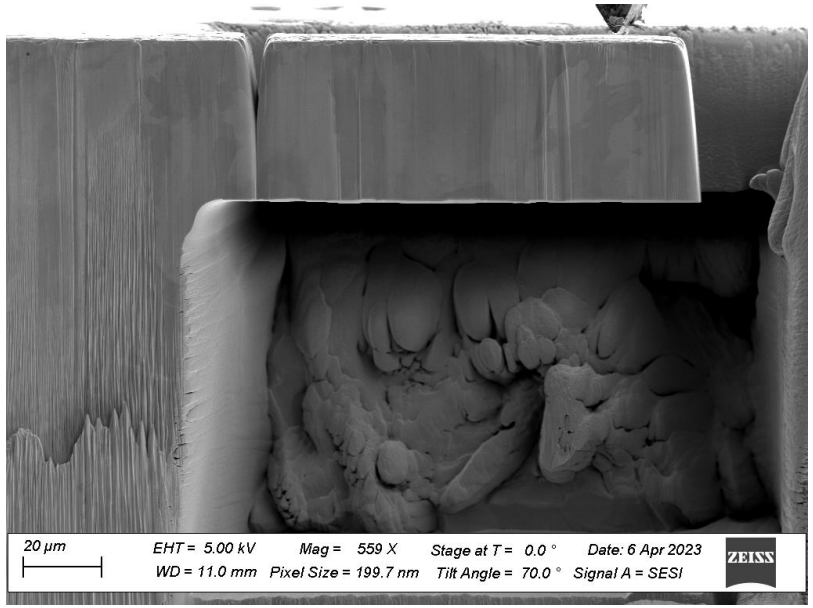
- Beaucoup d'images

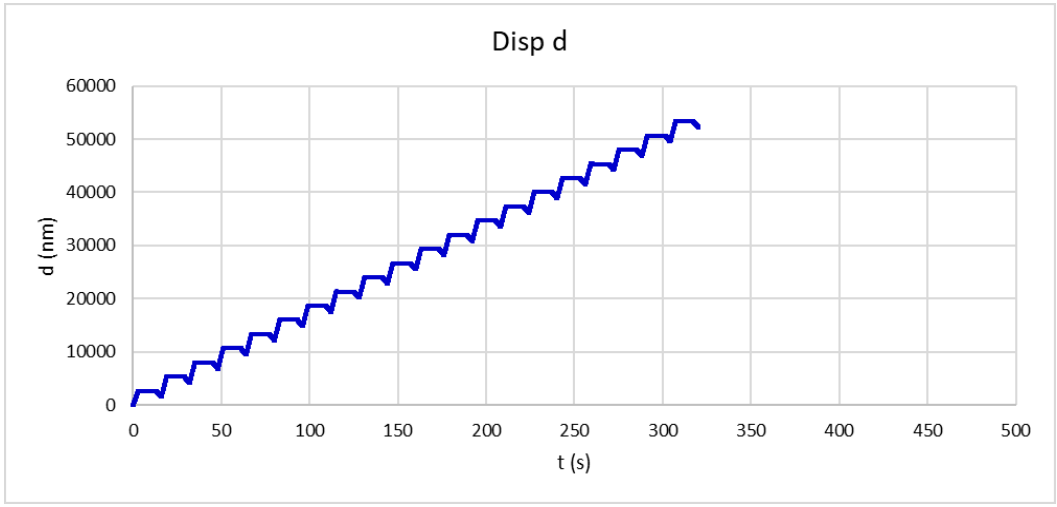
- Qualité d'image insuffisante pour suivi correct de fissure

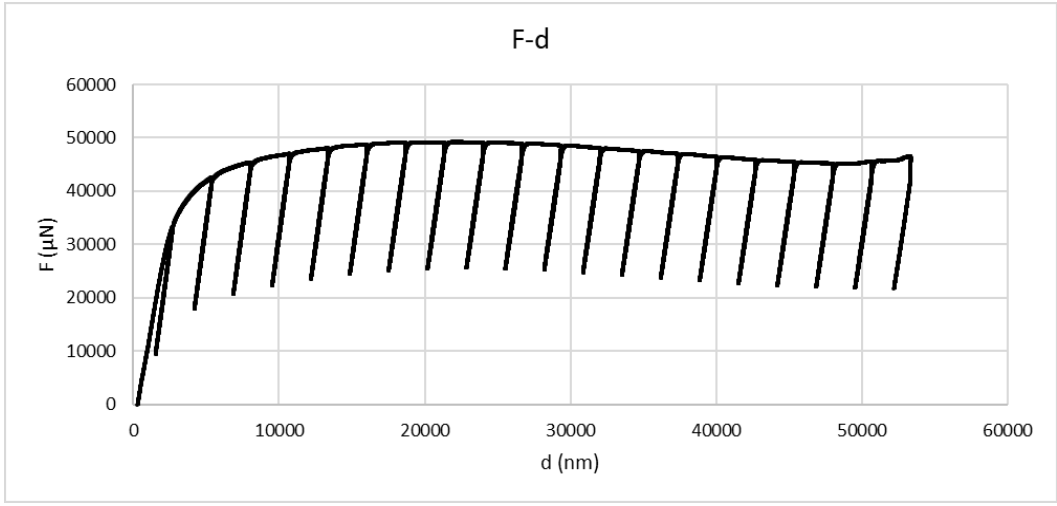
Essais de micro-flexion

Flexion *in situ :* pile d'images

- Pile d'images 50 images et 6s/image
- Sollicitation par paliers : une image/palier

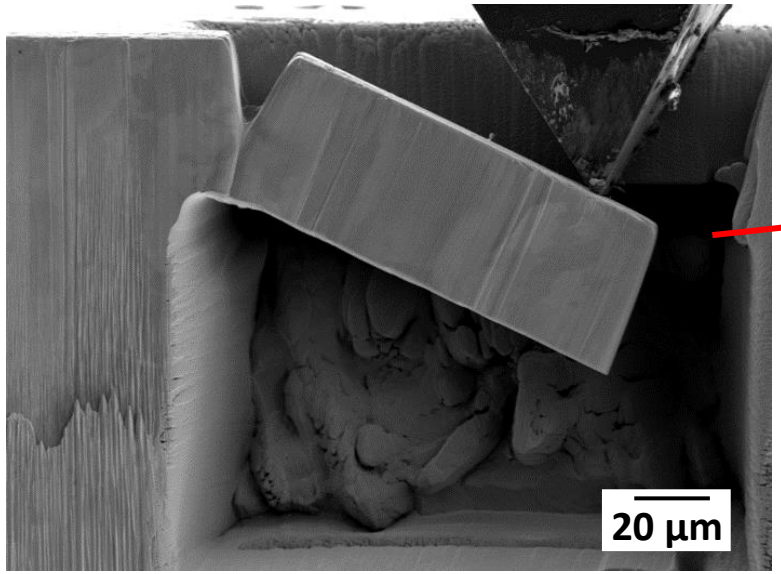


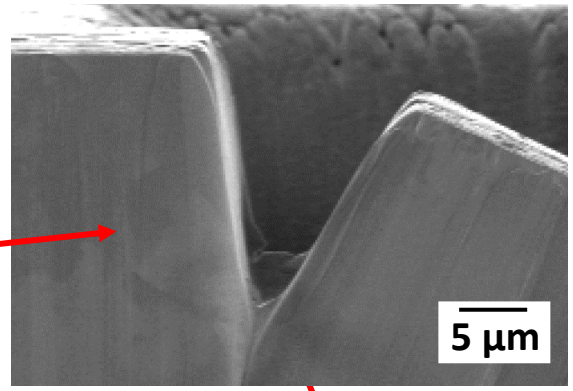


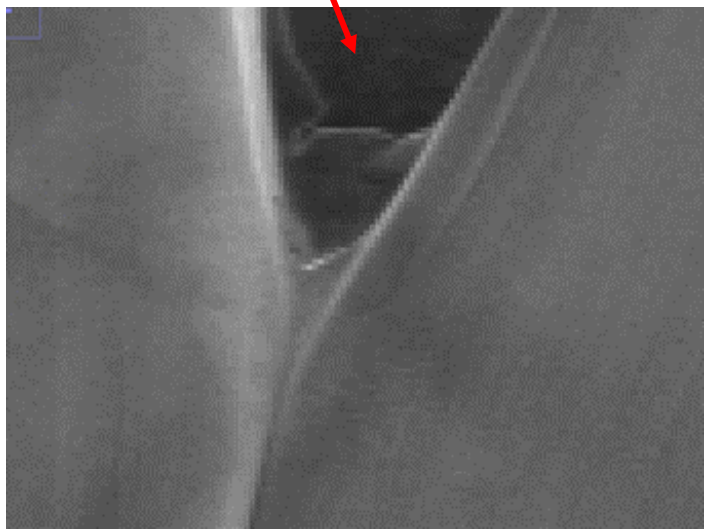


Essais de micro-flexion

Flexion *in situ :* pile d'images



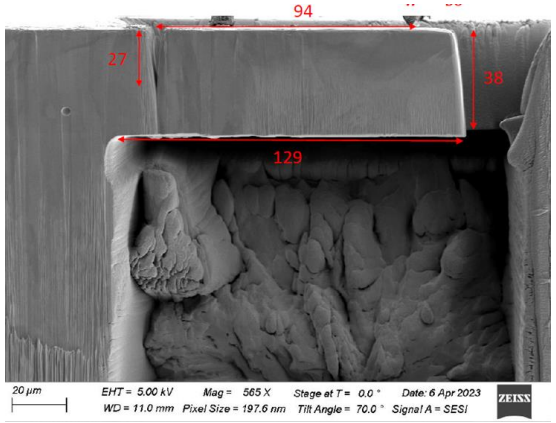




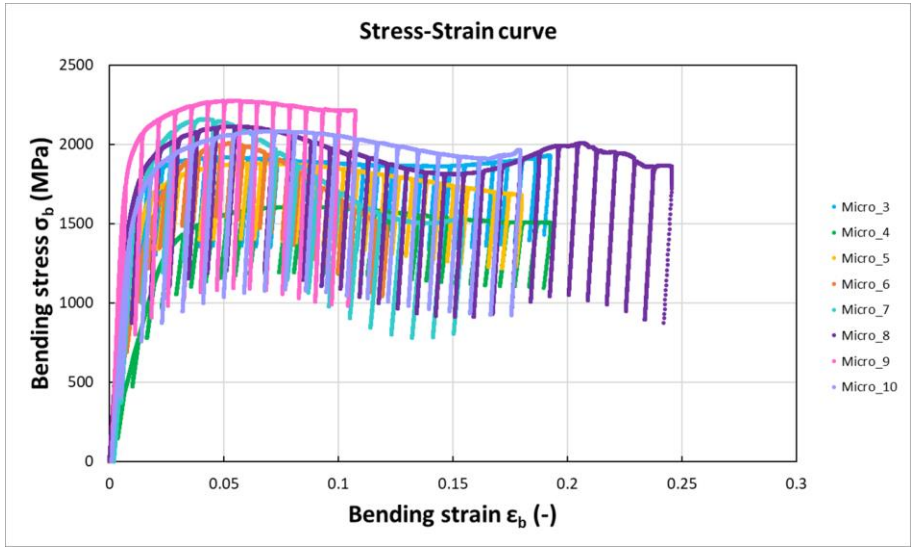
- Bonne qualité d'image pour suivi de fissure
- Peu d'images

Essais de micro-flexion

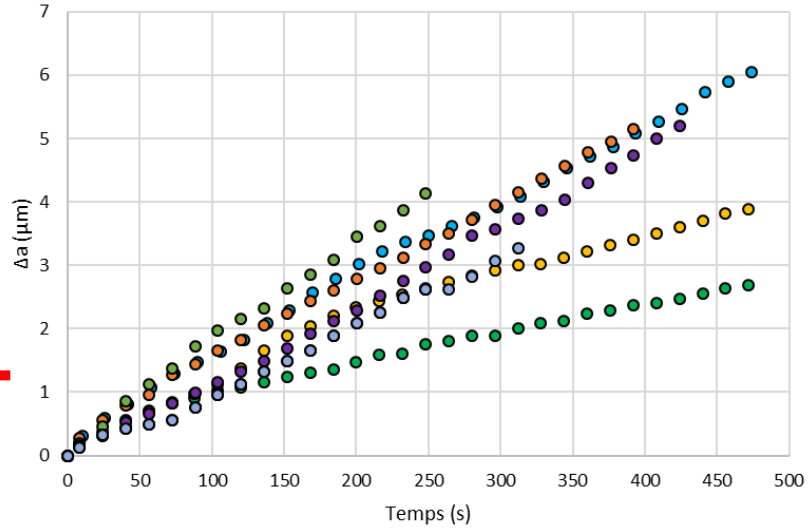
Flexion *in situ :* pile d'images



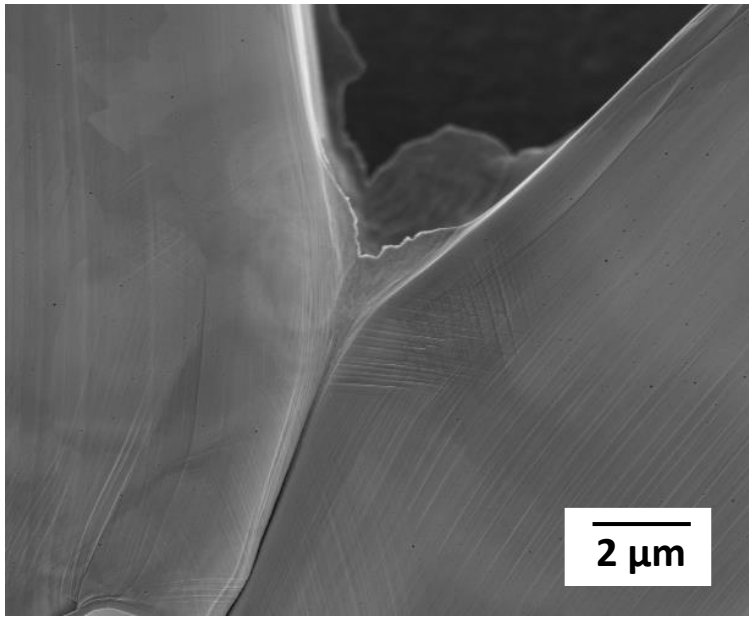
Dimensions des éprouvettes



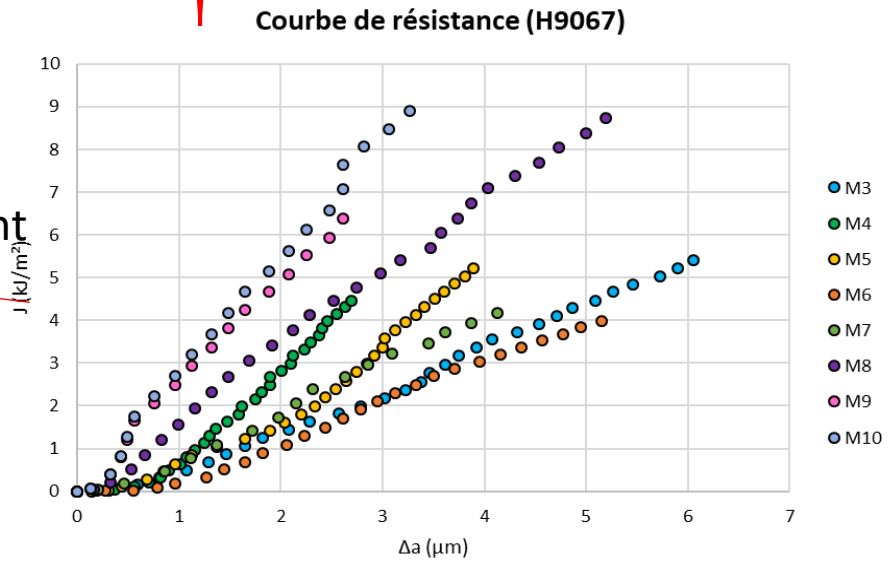
Courbe force-déplacement



Analyse d'image : longueur de fissure



Émoussement



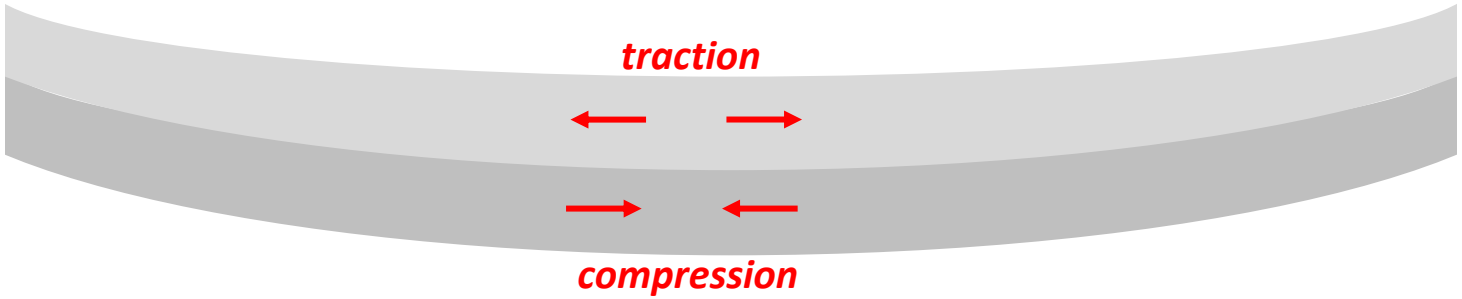
Mesure de contraintes résiduelles :

Mesure de champs de déformation par usinage FIB et Corrélation d'Images Numérique (CIN) d'images MEB

Voir poster Baptiste BOULET à la sfμ

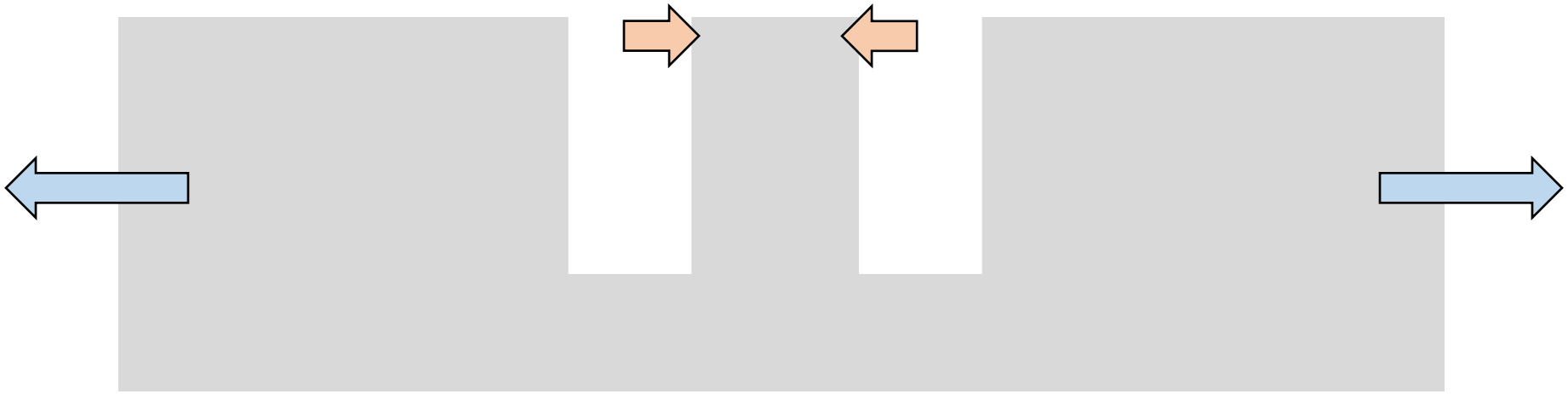
Contraintes résiduelles

Principe



Coefficients de dilatation thermique $\alpha_1 > \alpha_2$

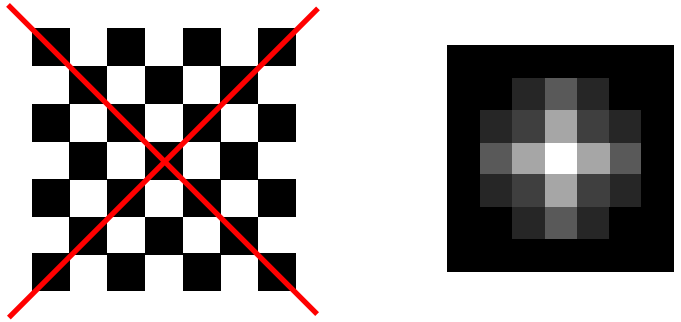
Mise en avant



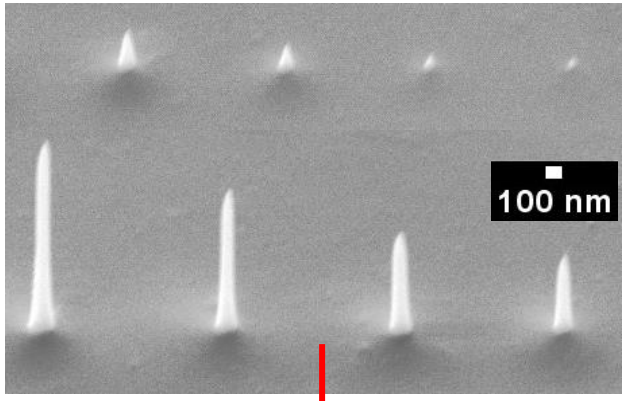
Corrélation d'images numériques

Mouchetis

Motif aléatoire, 3 à 5 pixels par particules

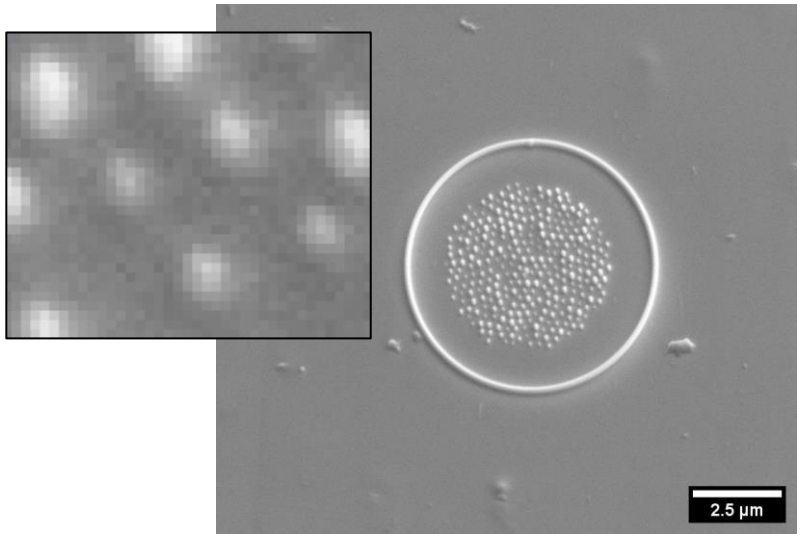


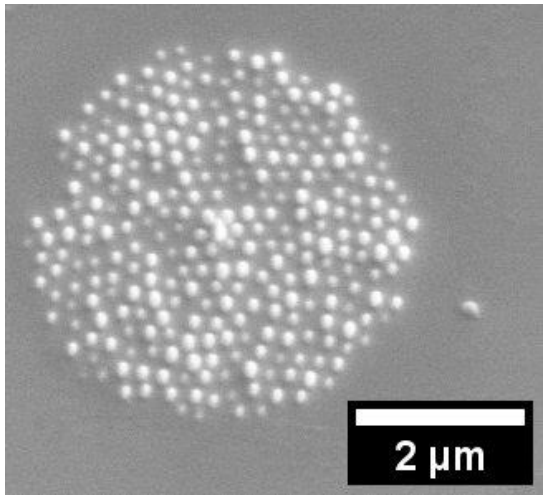
Dépôts de platine à 5kV et 200pA / 0,1 à 1s



Logiciel NPVE (FIBICS)

+ MATLAB ⇨





Motif aléatoire optimisé

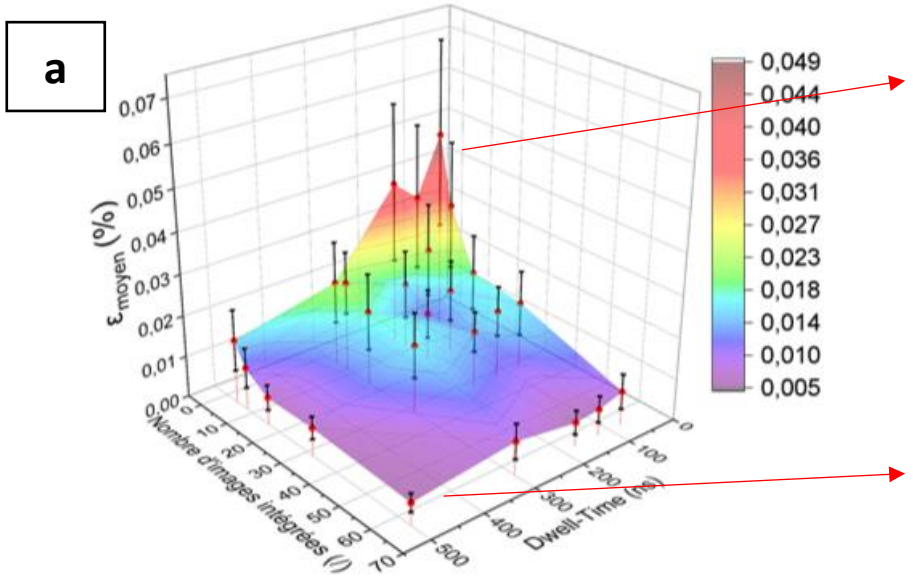
Paramètres dépendent du matériau

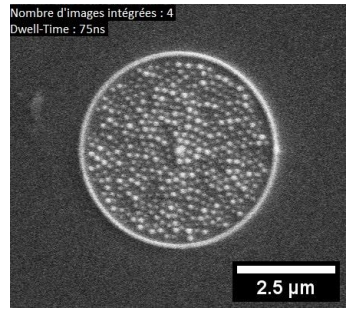
Imagerie MEB

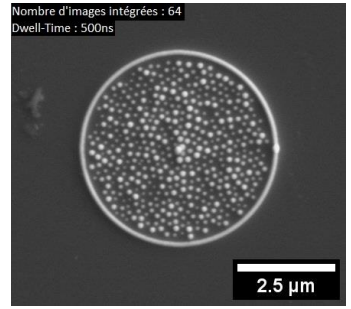
Étude paramétrique

- Mode de réduction du bruit : "frame integer"
- Nombre d'images pour la moyenne (a) : 40
- Dwell time (a) : 100 ns
- Courant (b) : 1 nA
- Tension (b) : 5 kV
- Distorsion (c) : bord de l'image

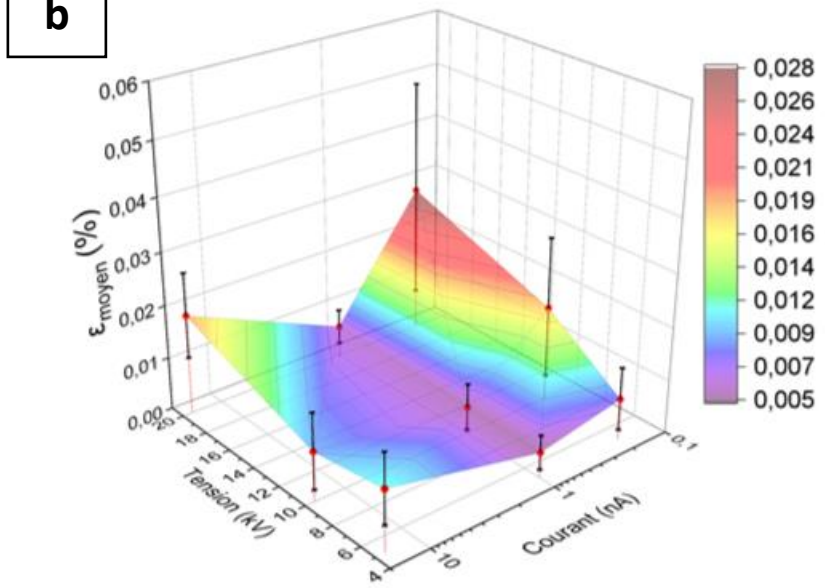
a



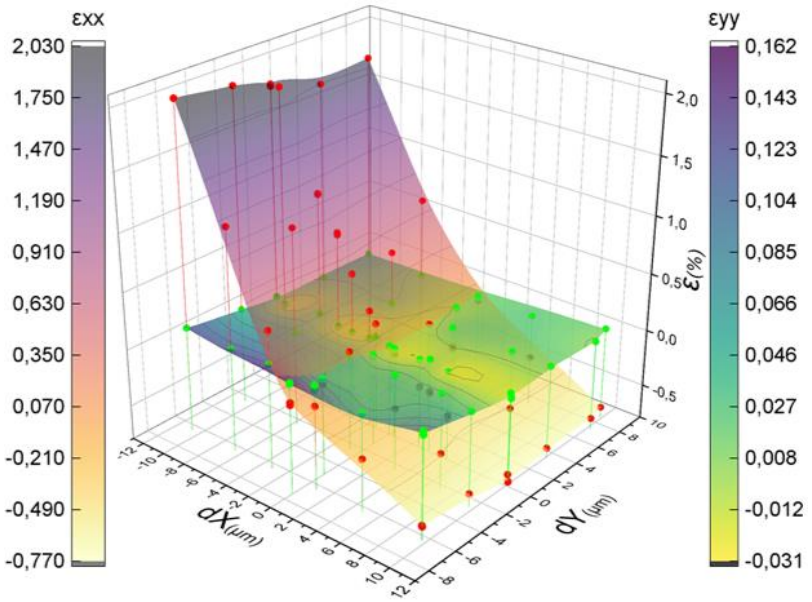




b

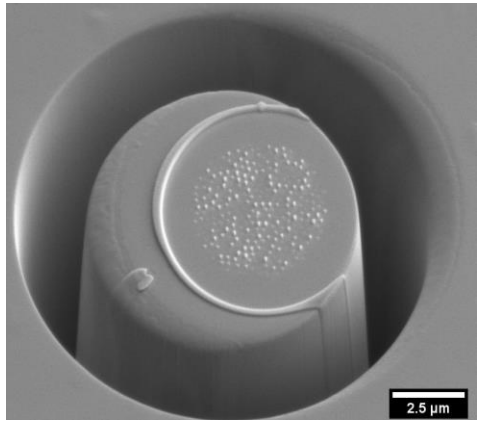


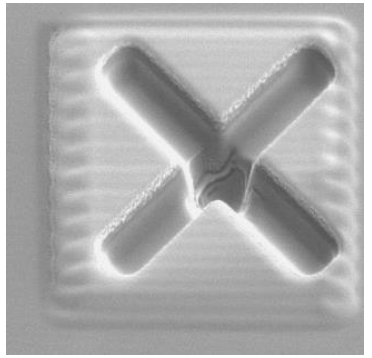
c



Autres paramètres

- Macro pour boucle (image MEB - usinage FIB)

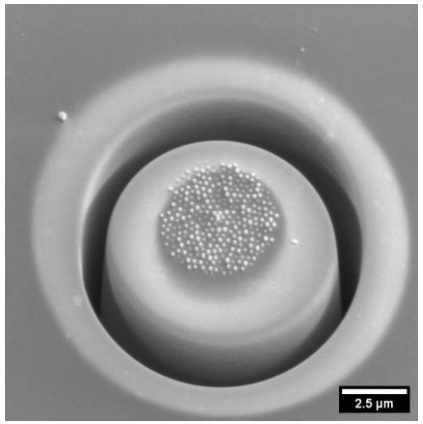


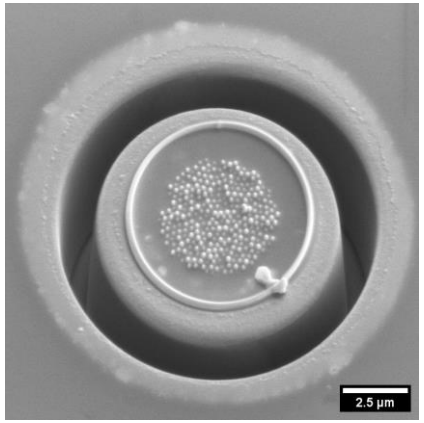


- Recaler le faisceau FIB : macro de suivi

- Recaler le faisceau MEB : imageJ en post-traitement

- Réduction de la re-déposition FIB
  - *Limiter courant FIB*
  - *Contour de protection*

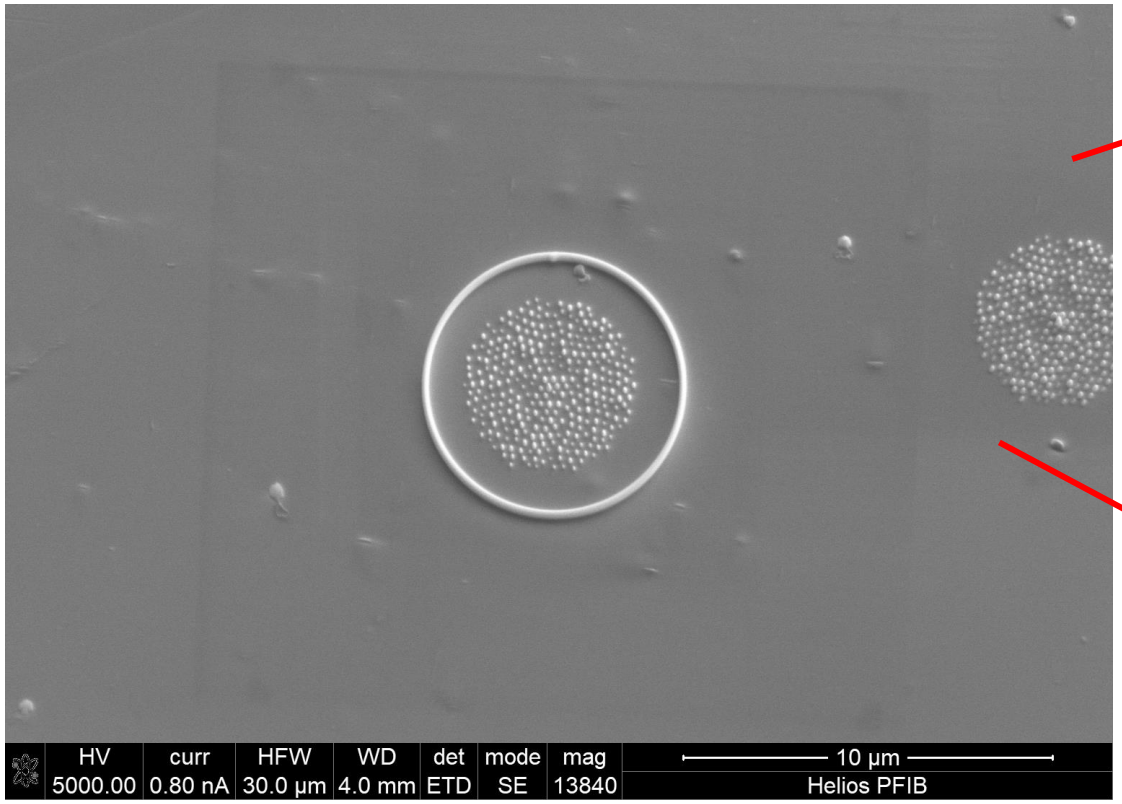


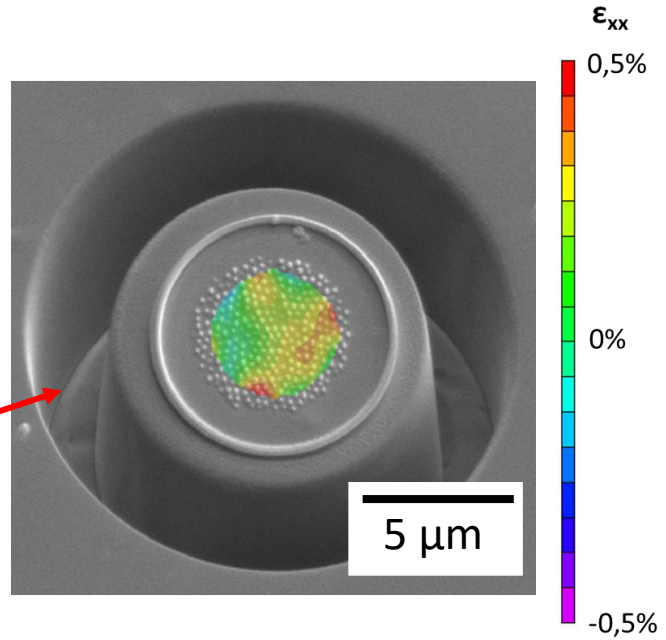


Images MEB du re-dépôt et de l'intérêt du mur de protection

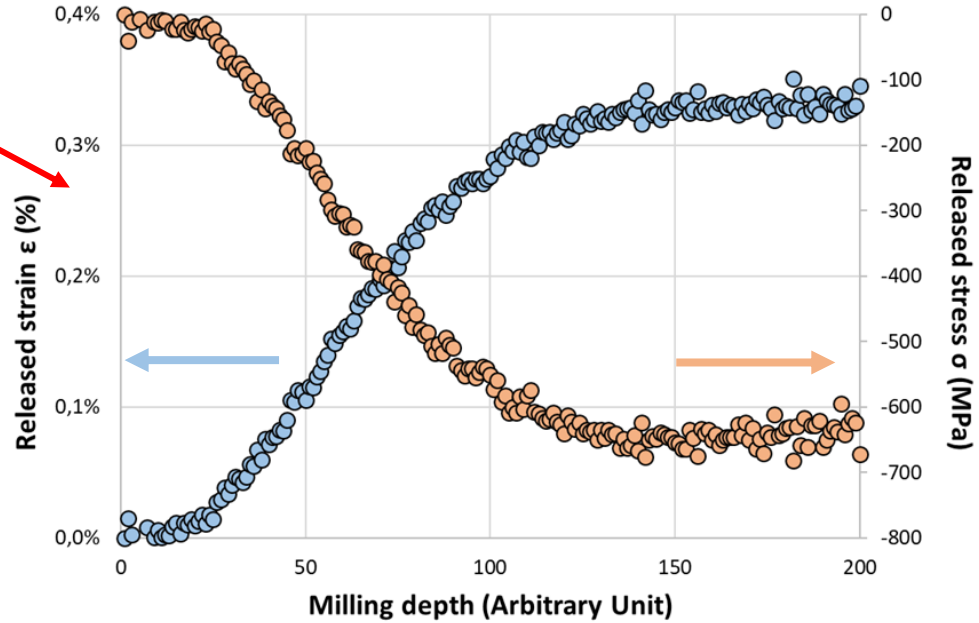
FIB-CIN

Résultats





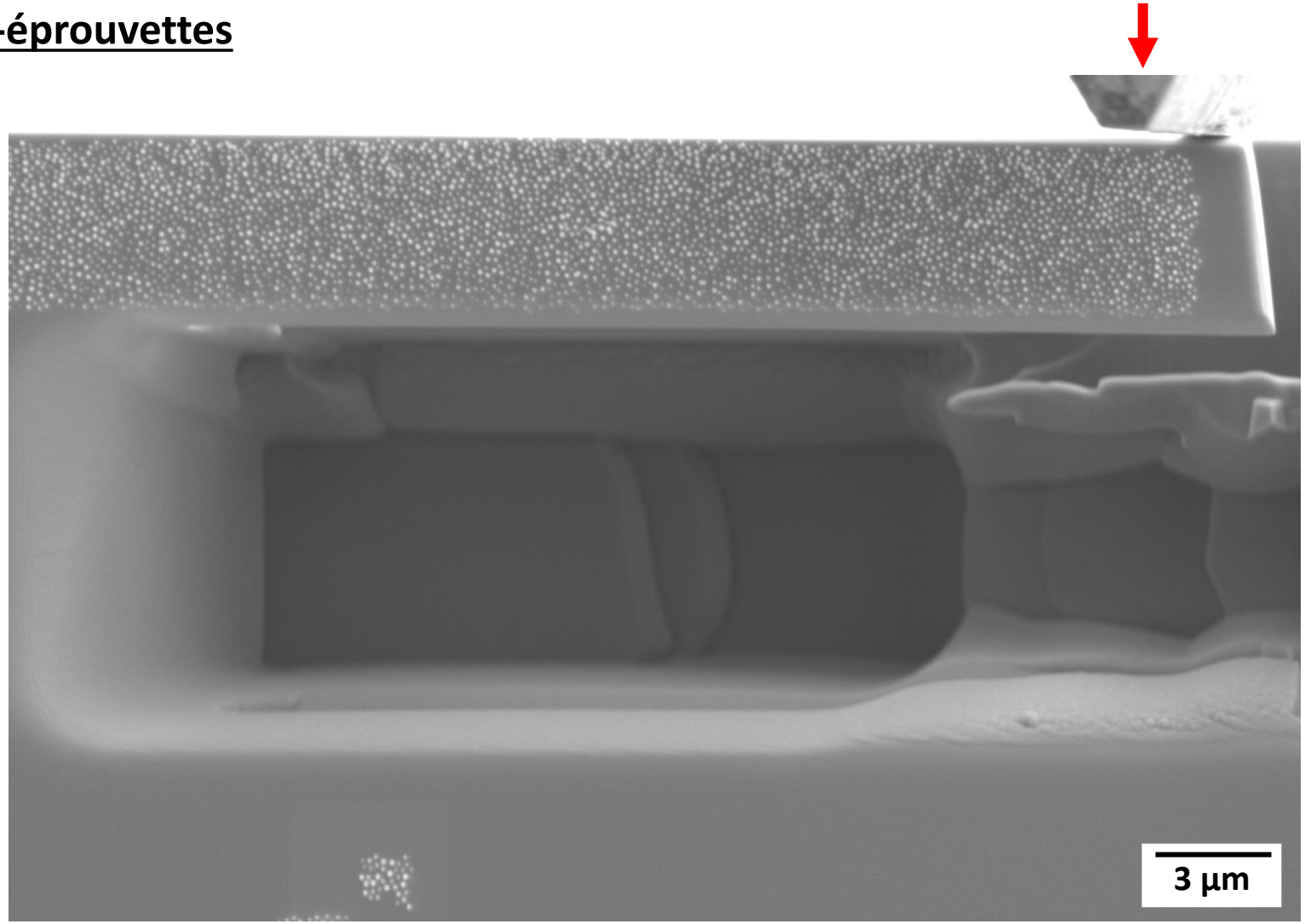
Mesures CIN du champs de déformation sur chaque image



Déformation et contraintes en fonction des étapes d'usinage

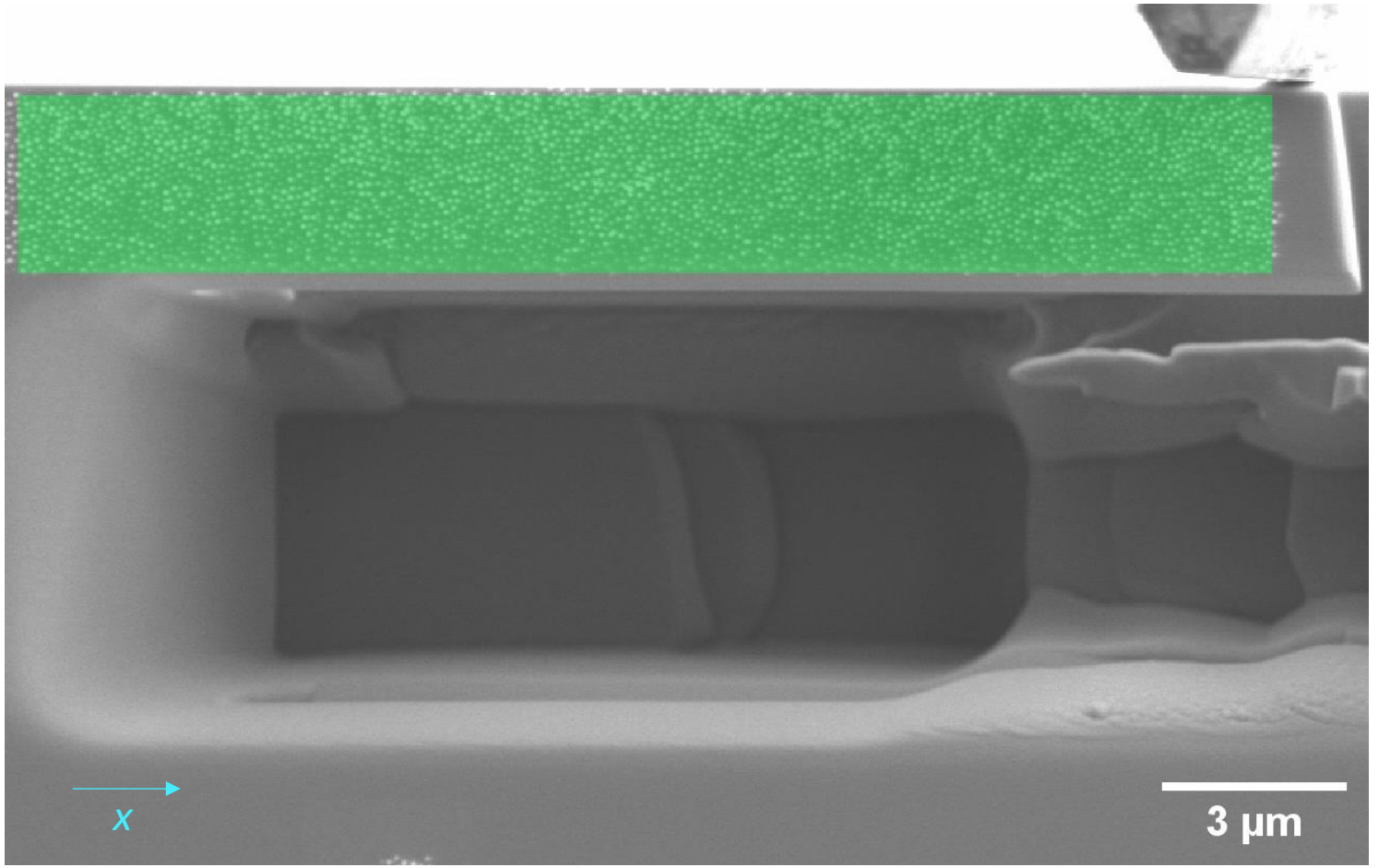
Couplage des techniques: flexion + CIN

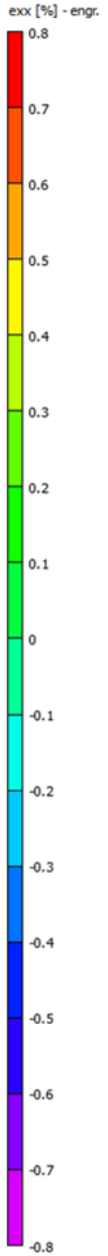
Mouchetis sur µ-éprouvettes



Couplage des techniques: flexion + CIN

CIN sur flexion *in situ*





Conclusions

MEB :
Microscope Électronique à Balayage

Essais in situ
- Acquisition des images : compromis entre temps et qualité

Corrélation d'images numériques :
- Réalisation du mouchetis
- Acquisition des images

Optimiser les paramètres

Merci pour votre attention