

Application du μ -usinage FIB à l'étude des propriétés mécaniques des revêtements DLC/acier

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St-Etienne, France*

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Bâtiment des Hautes Technologies
St-Etienne, France*

- Introduction (essais micro-mécaniques)
 - Revêtements DLC
 - Micro-poutres (σ résiduelle)
 - Micro-piliers ($\sigma(\varepsilon)$)
 - Conclusions & Perspectives

Introduction

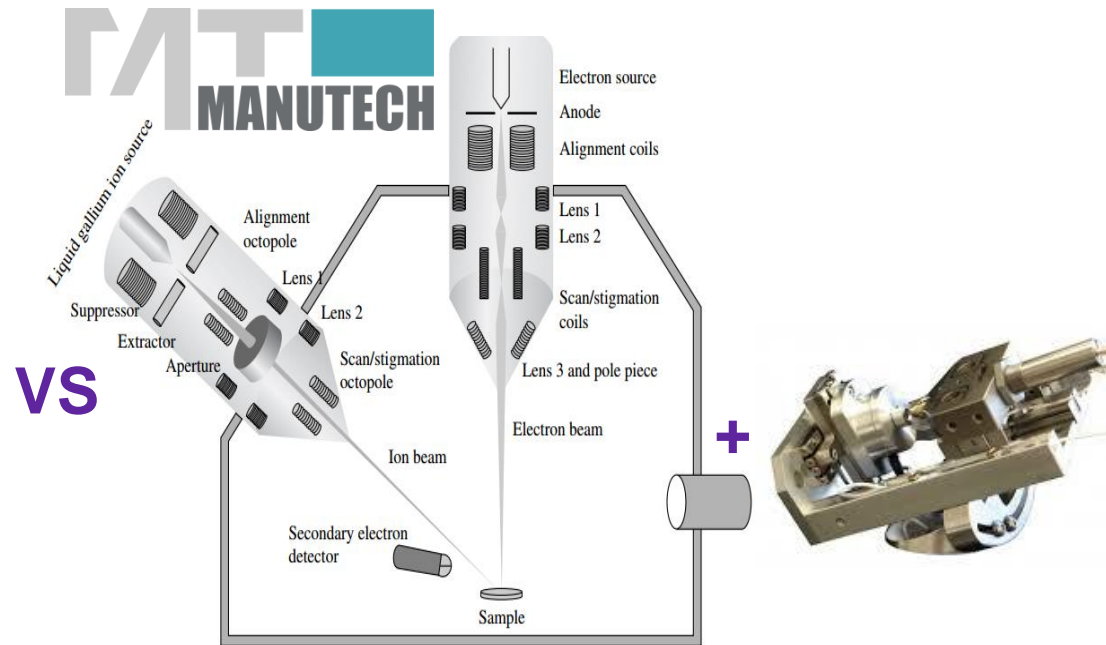
Pourquoi le μ -usinage FIB ?



Propriétés
mécaniques locales

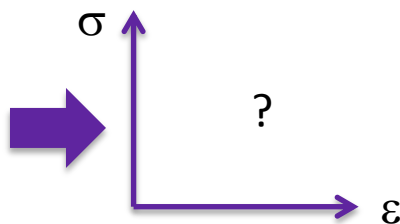
Matériaux polyphasés, Matériaux sensibles (UO₂), Couche mince...

Nano-indentation



VS

+

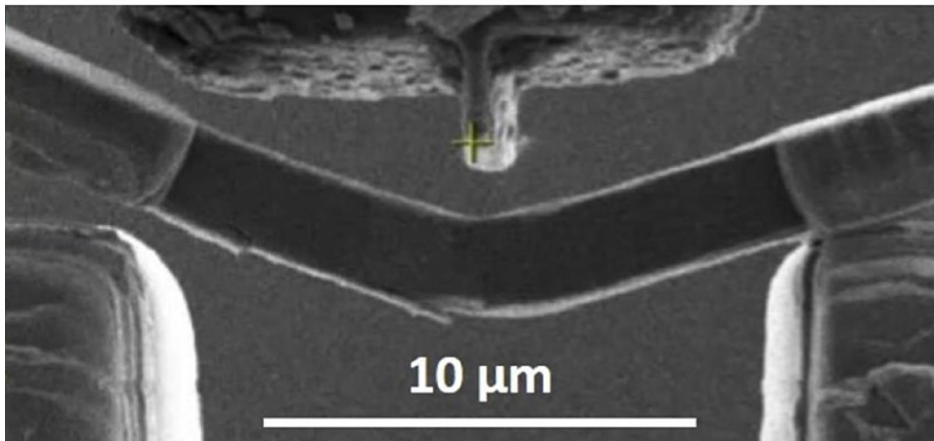
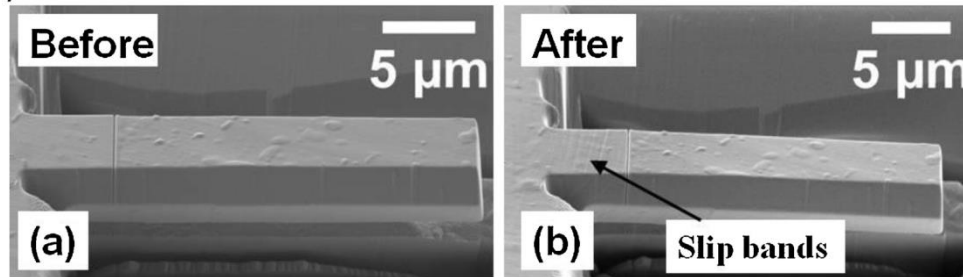


μ -pilier
 μ -poutre

Introduction

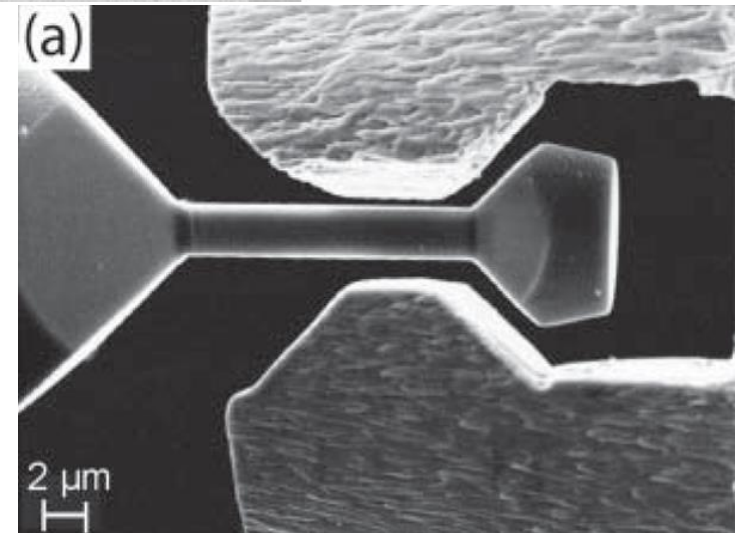
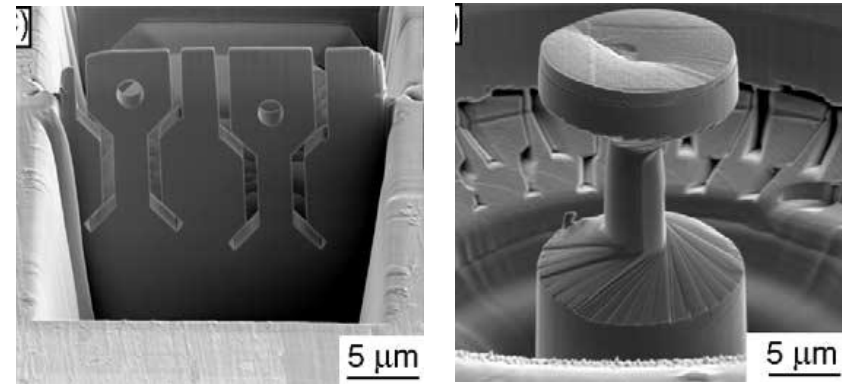
Essais de flexion

A. Stratulat and al., *Corrosion Science* 104 (2016)



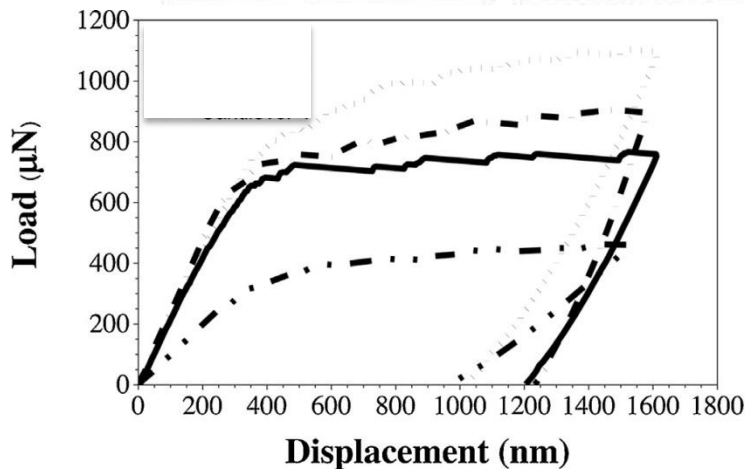
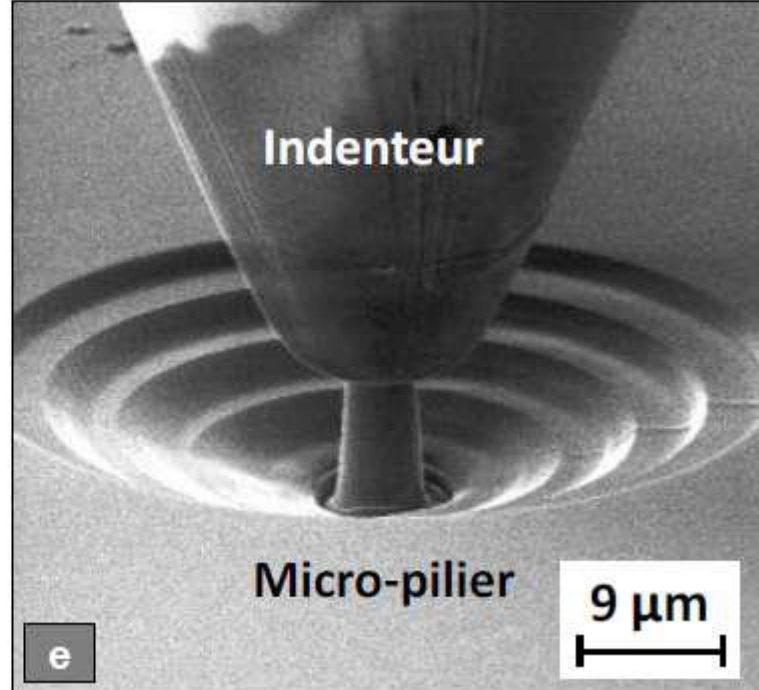
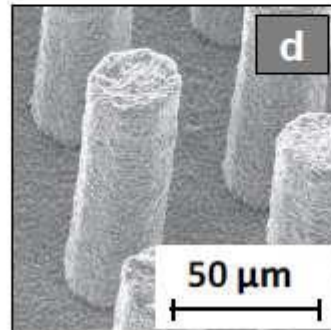
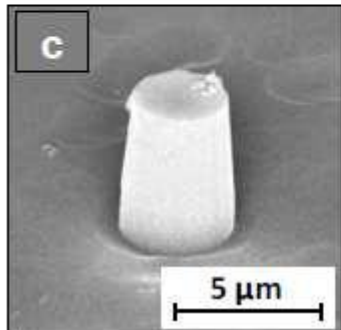
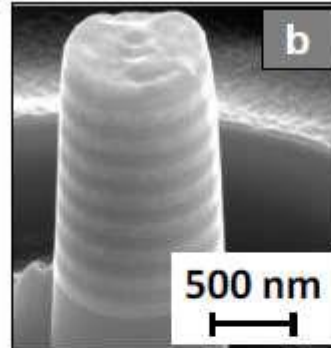
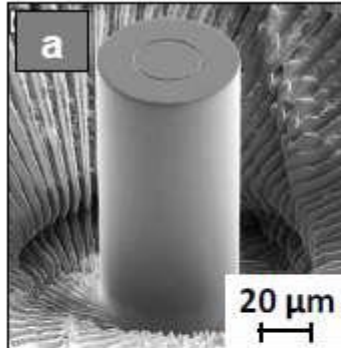
Essais de traction

D. Kiener and al., *Scripta Materialia*, 60, (2009)



Introduction

Essais de compression



➔ Limite d'élasticité, Module Young, Résistance à la rupture, Plasticité, Propagation de fissure, ...

Introduction

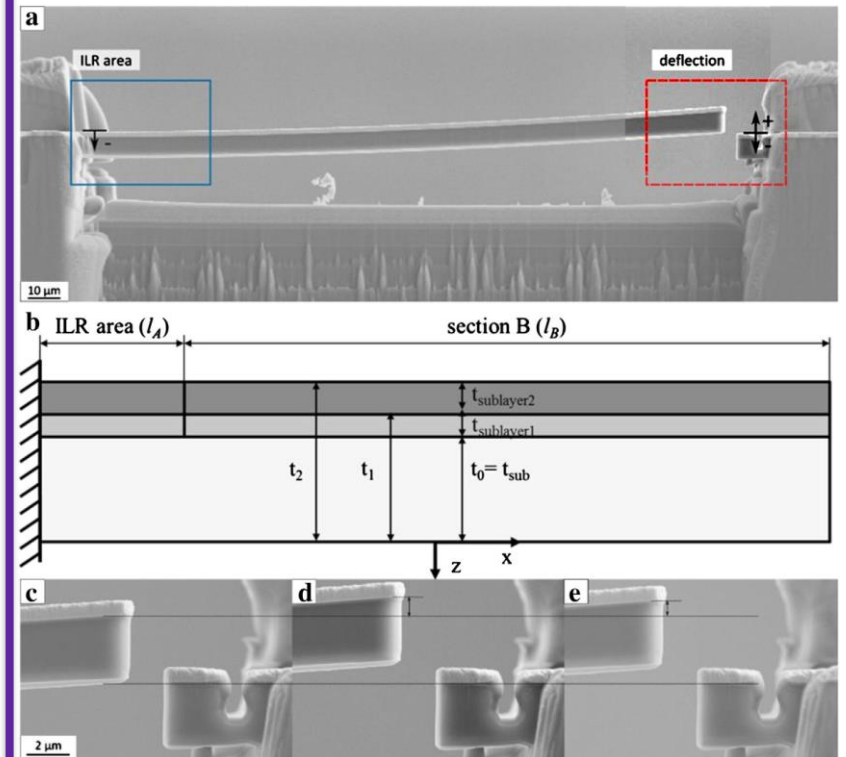
Mesure de contraintes résiduelles

FIB-DIC micro-ring core



M. Sebastiani and al. Materials Science and Engineering A (2011)

Ion beam layer removal method



R. Schönggrundner and al. Thin Solid Films (2014)

Revêtements Diamond Like Carbon DLC

Micro-electronics



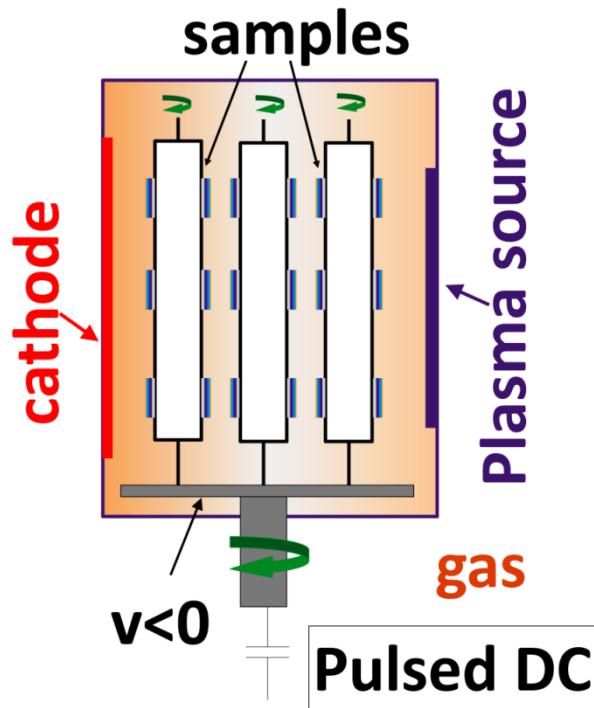
Bio-engineering



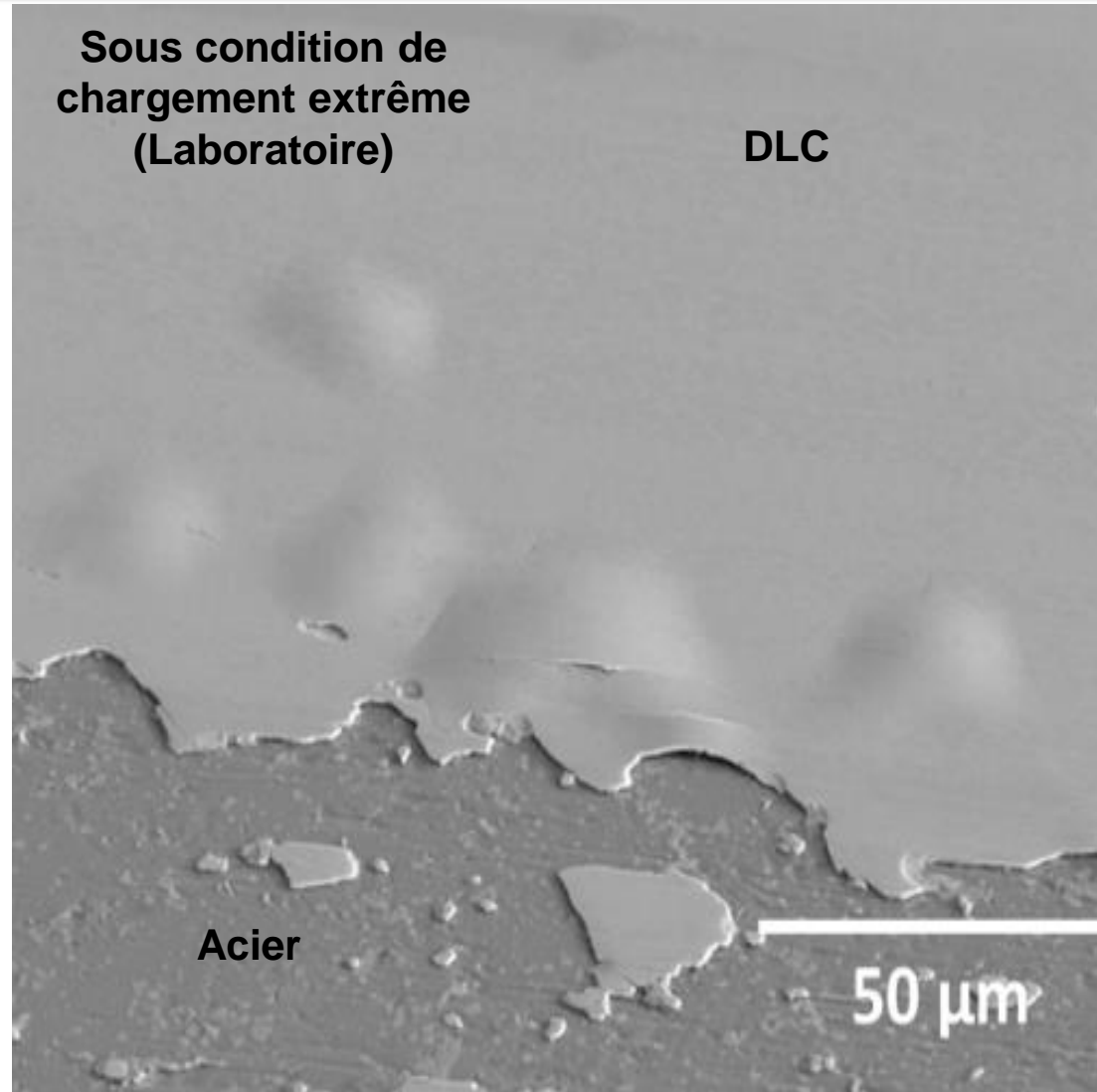
Automotive



PECVD and Samples

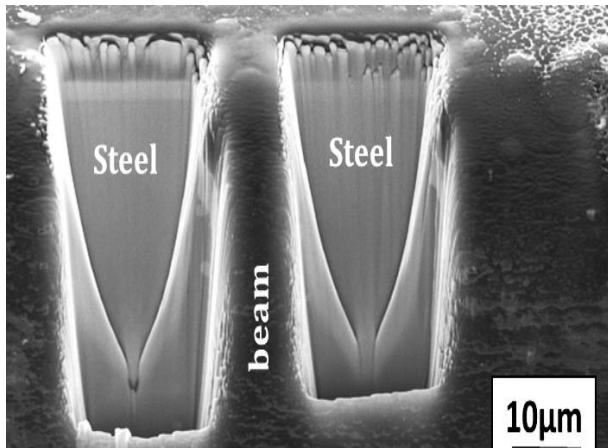


Deposition of thin films from vapor to a solid state on a substrate (PECVD)

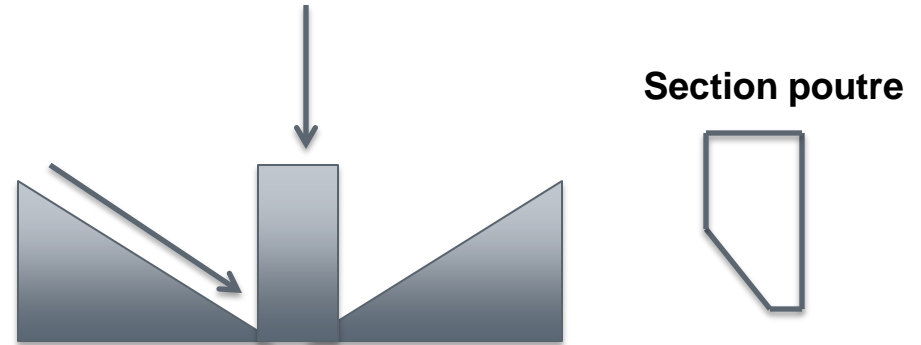


Optimisation des conditions d'usinage

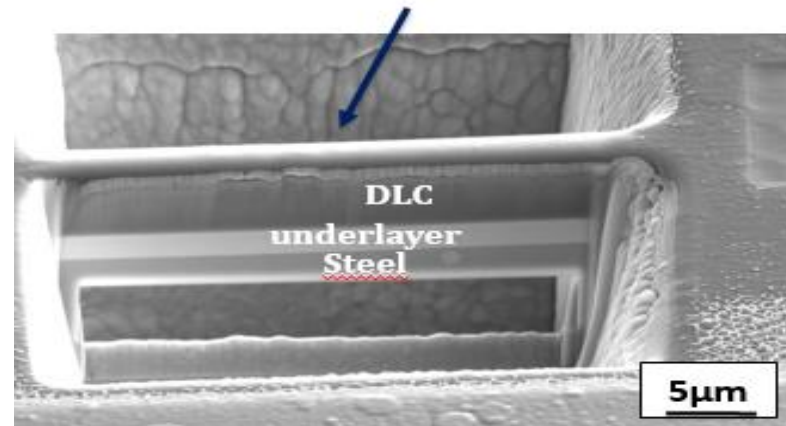
FEI Hellios Nanolab (Source Ga⁺)



Forme du faisceau ionique

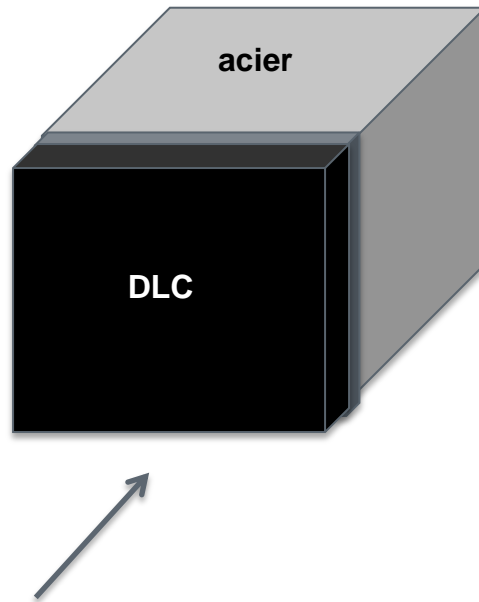


Tilted on 53°

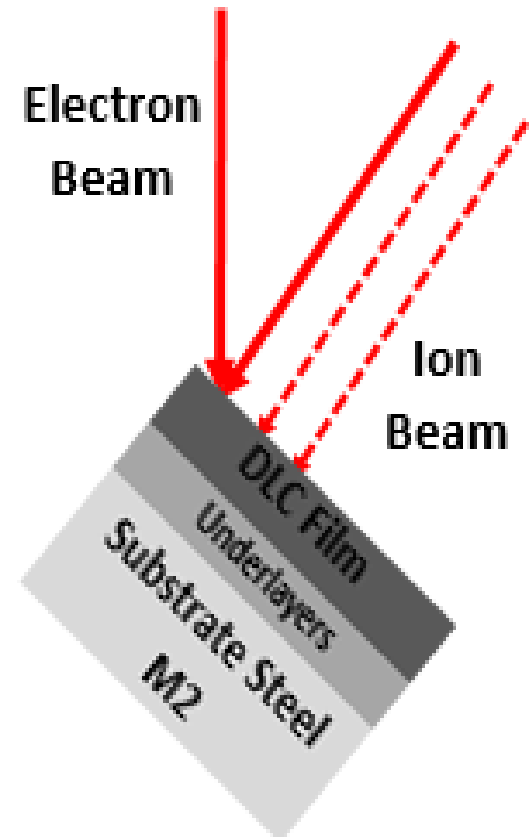


- Maitriser la géométrie de la poutre
- 3 étapes d'usinage

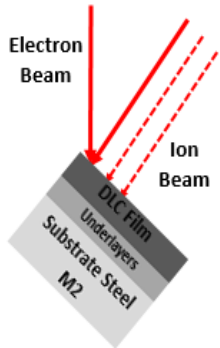
Étape 1: Préparation du micro-mur



Direction d'observation (faisceau FIB)

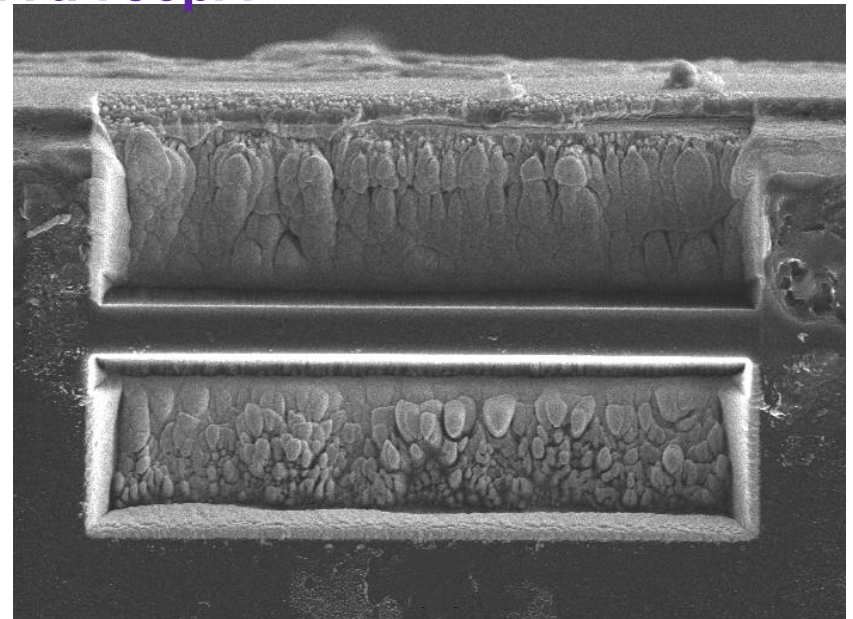
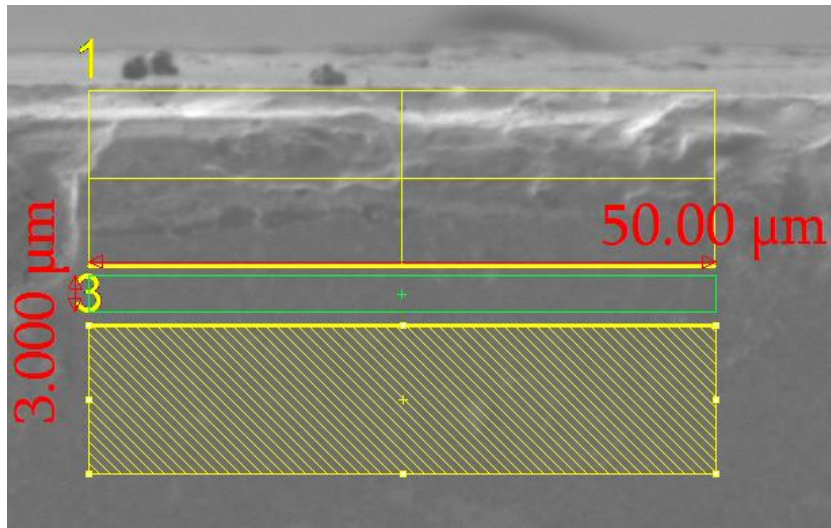


Étape 1: Préparation du micro-mur



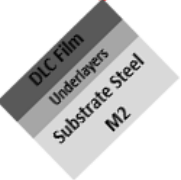
Usinage: 30kV, 2,5 à 9nA

Nettoyage: 30kV, 80pA à 790pA



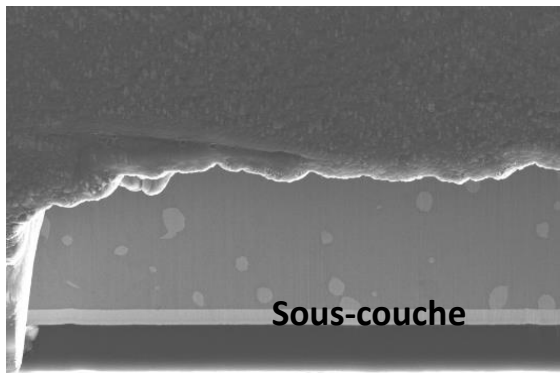
Étape 2: Préparation de la micro-poutre

Electron Beam
Ion Beam



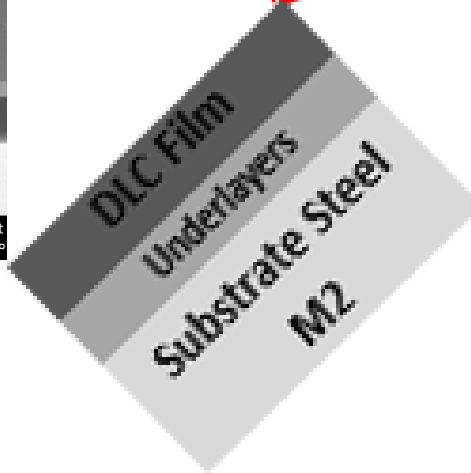
Electron
Beam

Ion
Beam

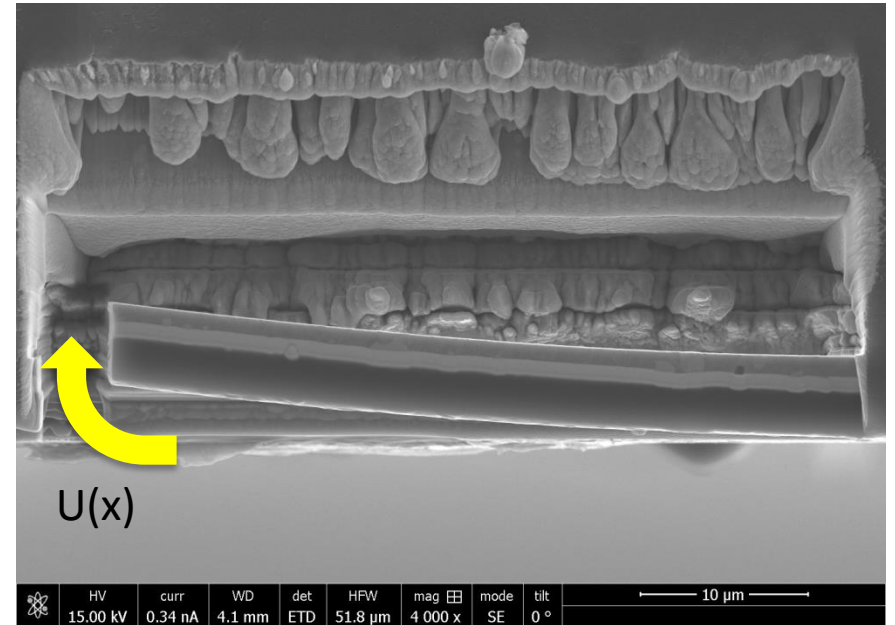
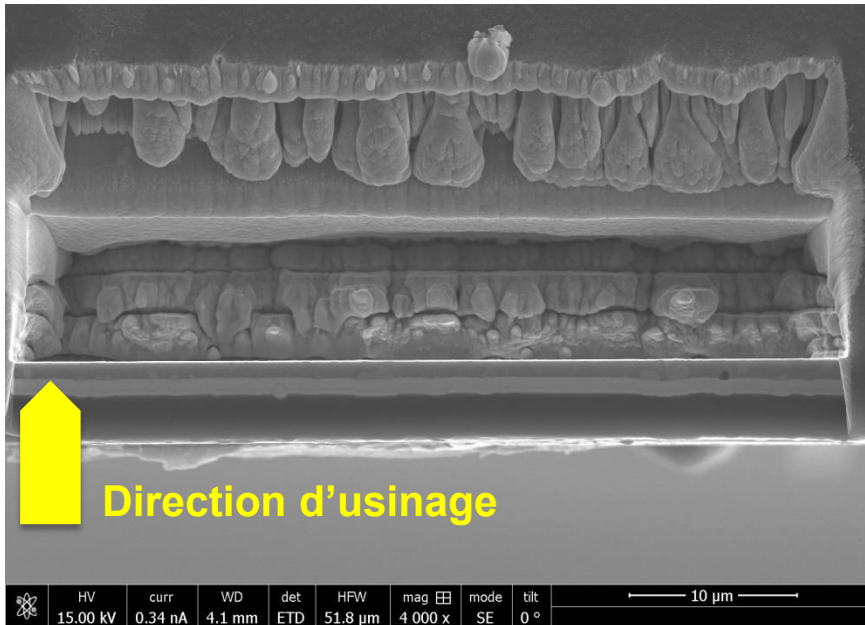


Sous-couche

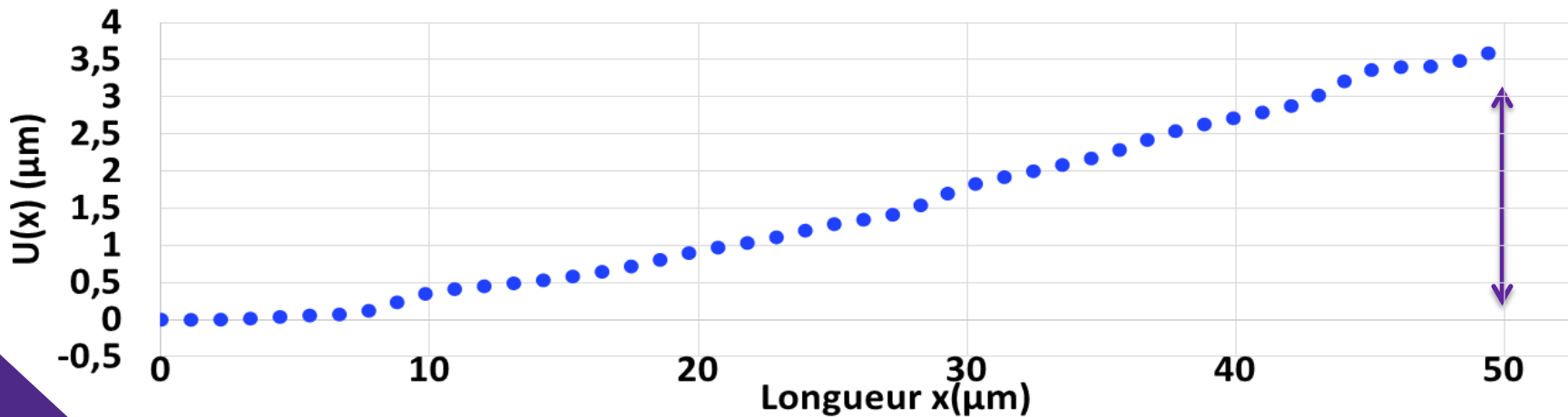
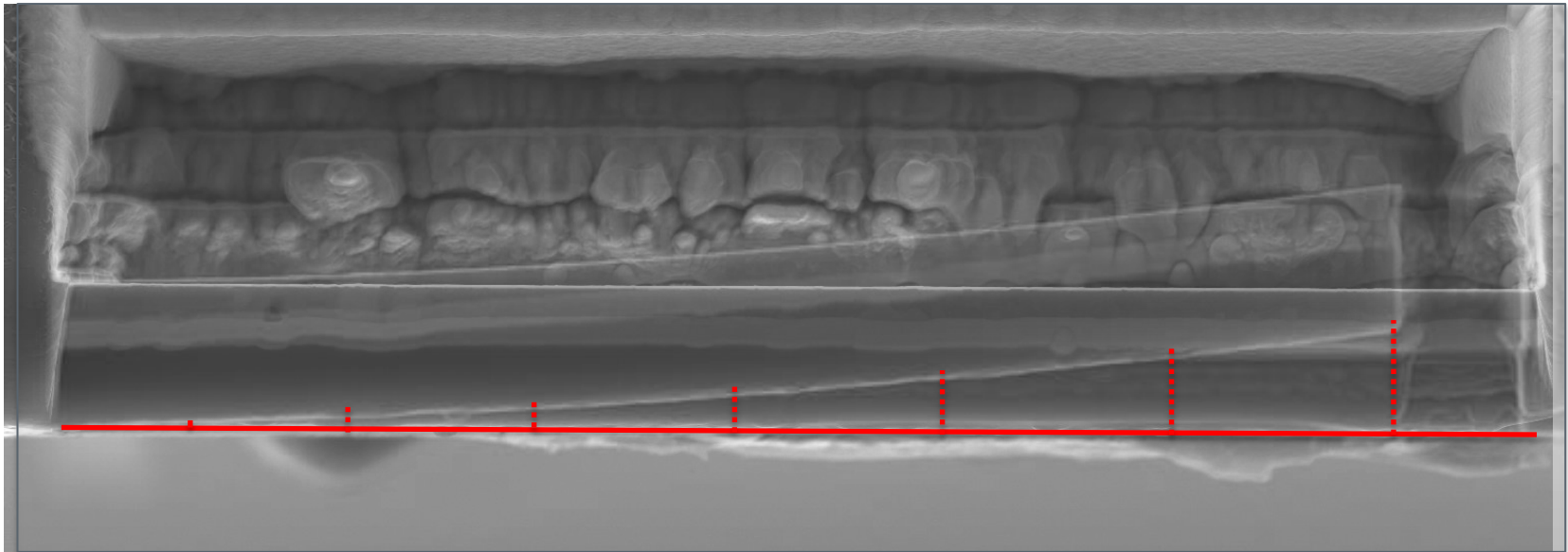
	HV	curr	WD	det	HPW	mag	mode	tilt
	10.00 kV	0.34 nA	4.1 mm	ETD	51.8 μm	4 000 x	SE	0 °



Étape 3

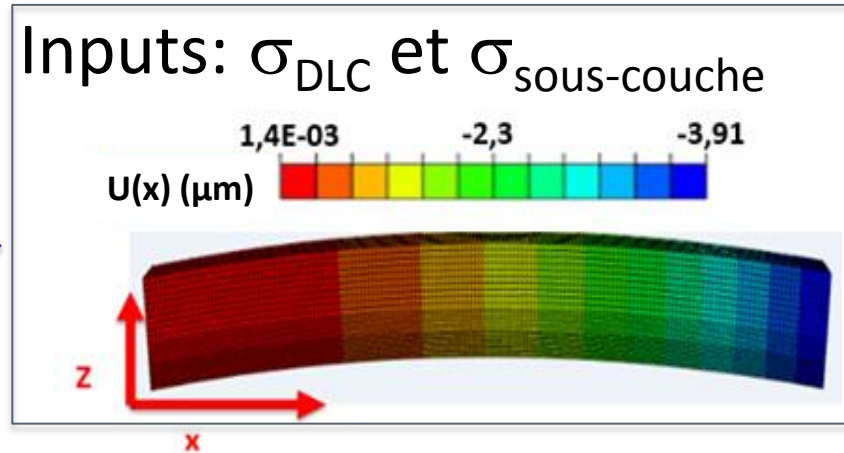


Mesure des déflexions



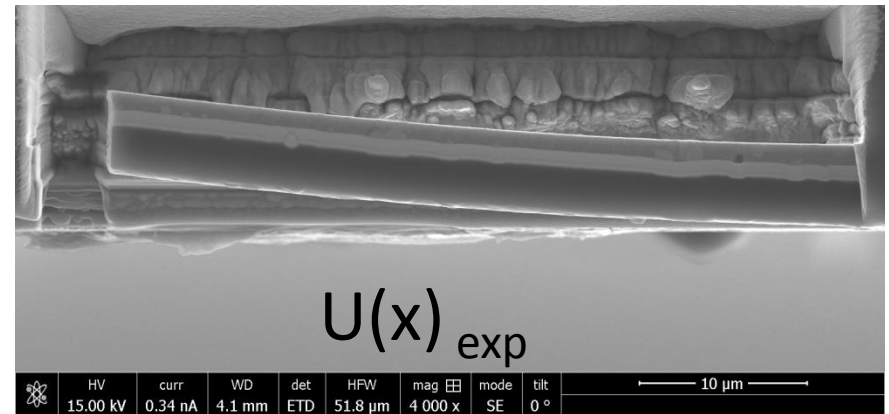
Modèle mécanique

Principe



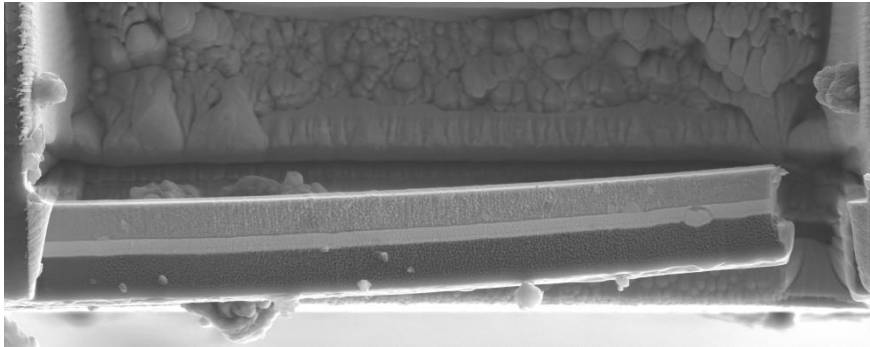
$U(x)_{\text{modèle}}$

$$U(x)_{\text{modèle}} = U(x)_{\text{exp}}$$

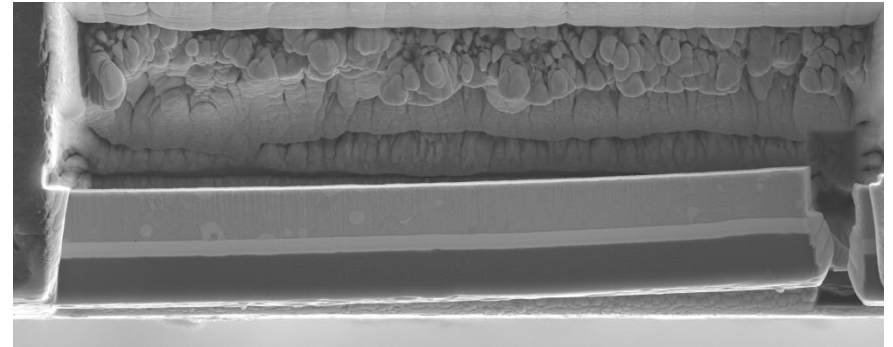


Poutres de géométrie optimale

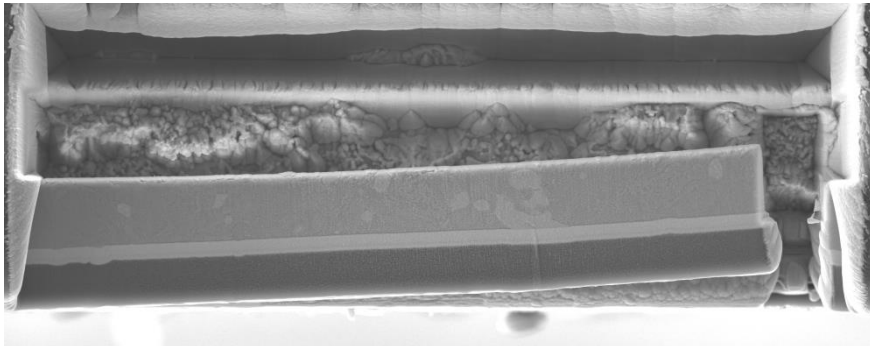
2 μ m M2



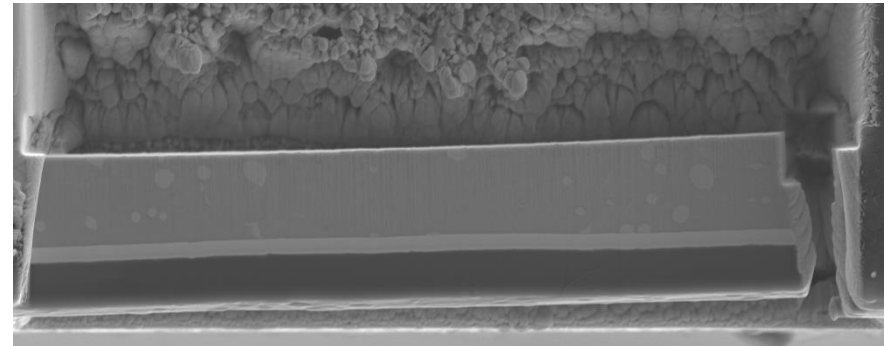
3 μ m M2



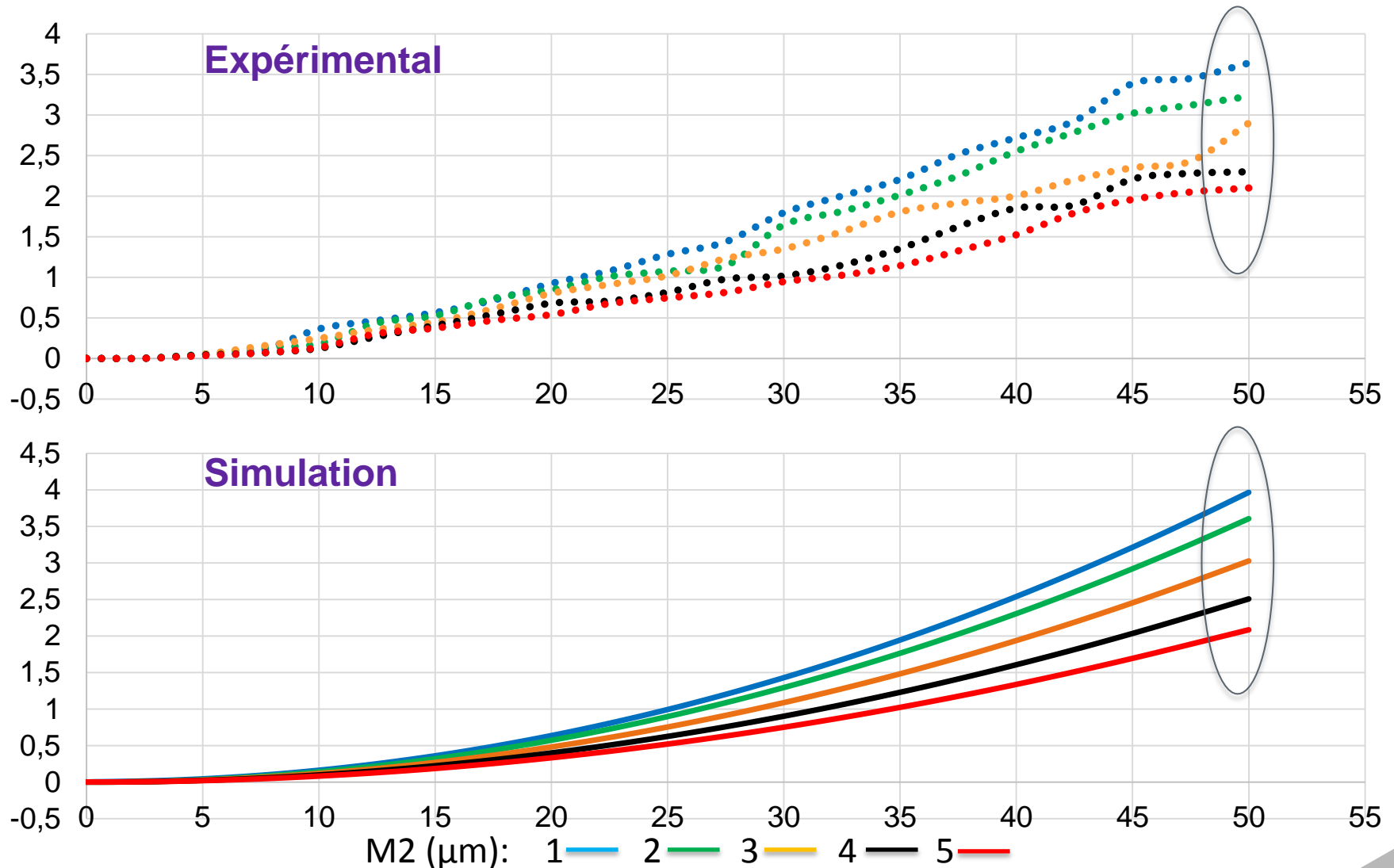
4 μ m M2



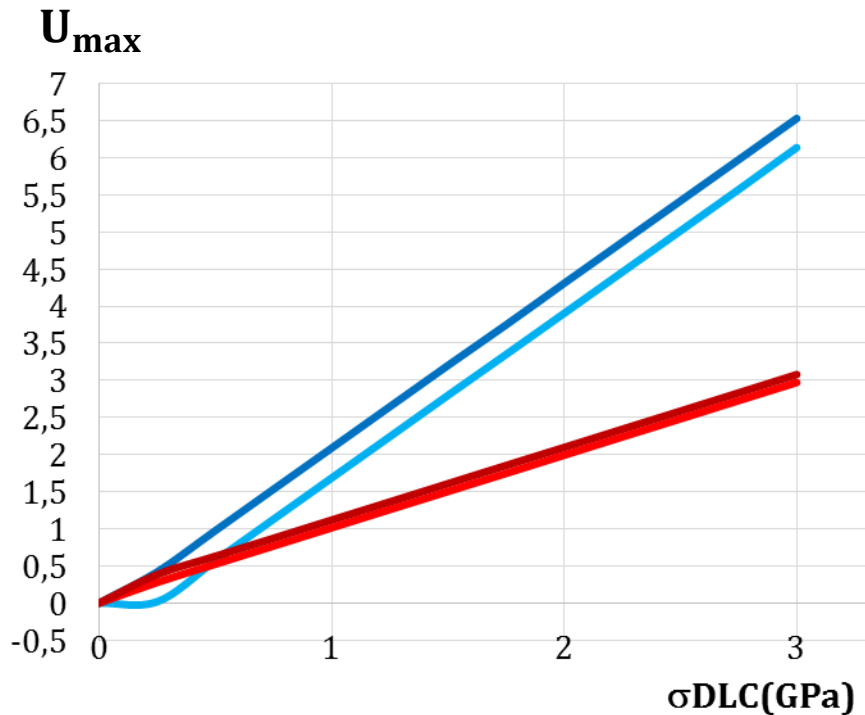
5 μ m M2



Comparaison déformées expérimentales et modélisées



Mesure de contraintes



$$\rightarrow U_{max} = 2,178\sigma_{DLC} - 0,535\sigma_{Scouche} + 0,07$$

$$\rightarrow U_{max} = 0,991\sigma_{DLC} + 0,1406\sigma_{Scouche} + 0,0094$$

Les inconnus ?

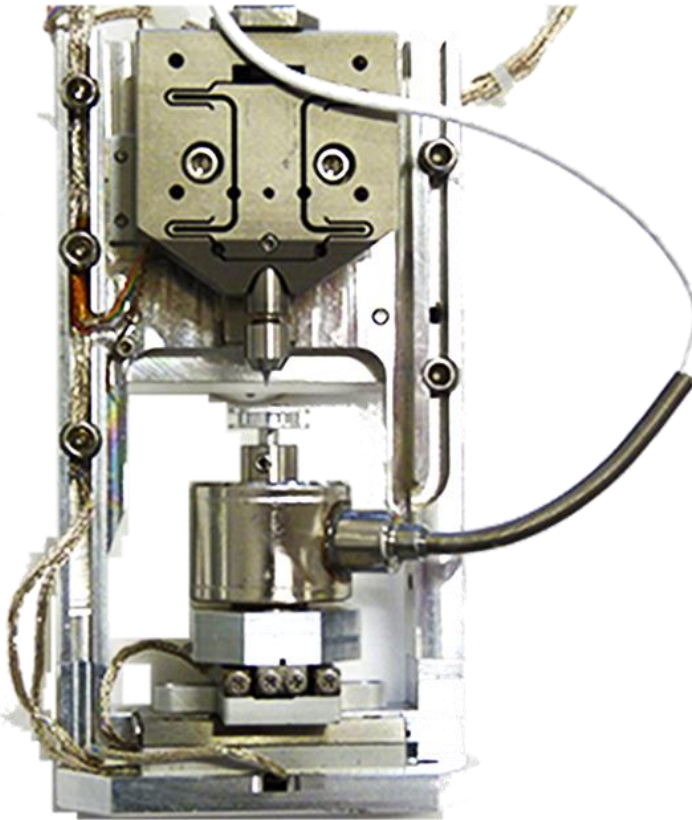
$$\vec{U}_{max} = \begin{pmatrix} 0,991 & 0,1406 \\ 2,178 & -0,535 \end{pmatrix} \begin{pmatrix} \sigma_{DLC} \\ \sigma_{Scouche} \end{pmatrix}$$

$$U_{max} (1\mu\text{m M2}) \approx 3,64 \mu\text{m}$$

$$U_{max} (5\mu\text{m M2}) \approx 1,9 \mu\text{m}$$

$$\rightarrow \begin{pmatrix} \sigma_{DLC} \\ \sigma_{Scouche} \end{pmatrix} = \begin{pmatrix} 1,82 \\ 0,63 \end{pmatrix} \text{ GPa}$$

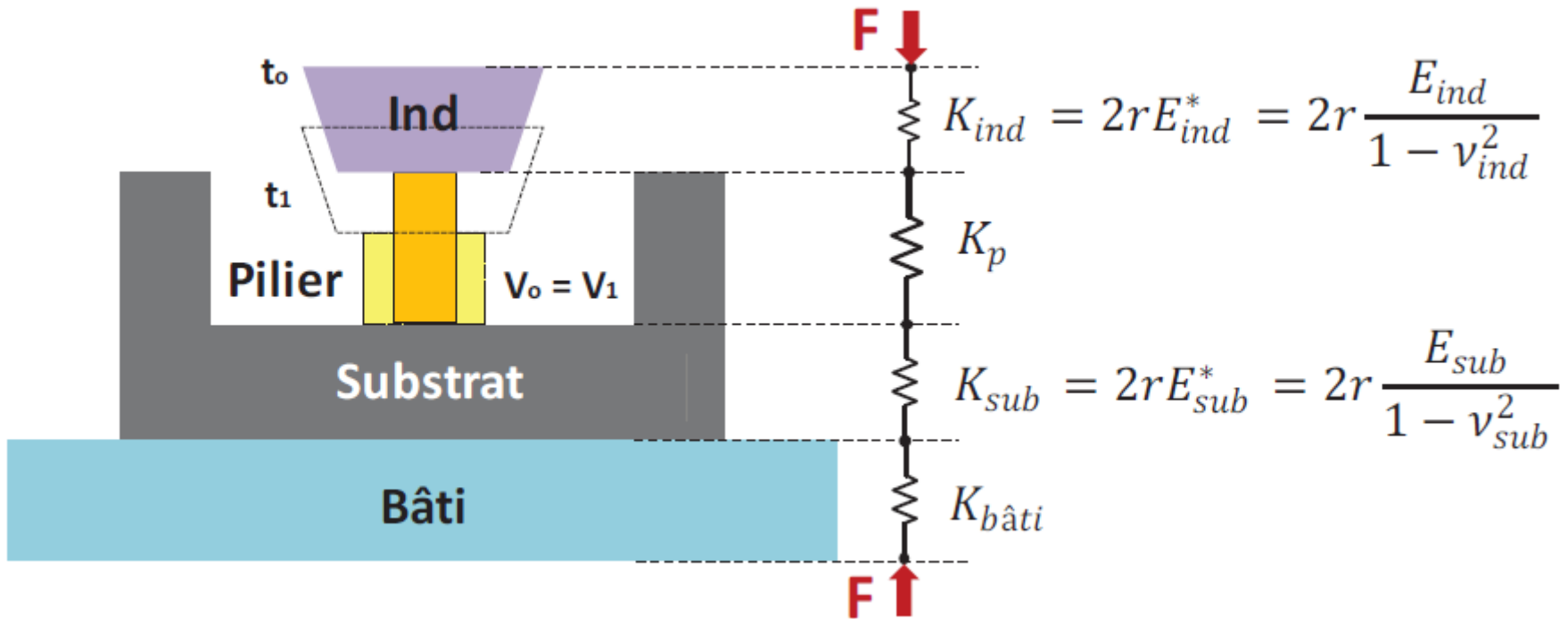
Nano-indenter insitu (Alemnis)



- Indentation range: 35 μm
- Displacement noise floor : <1nm
- Typical drift at room temperature : 5nm/min
- Load range: 500mN standard
- Load noise floor: 4 μN / 12 μN
- Tip to sample alignment range: 26 x 26 mm (x-y),
22 mm (indentation axe) 1.2nm resolution
- Indentation tips : Berkovich, Cube Corner, Flat top, Wedge, Spherical, etc. Easily exchangeable

Micro-piliers

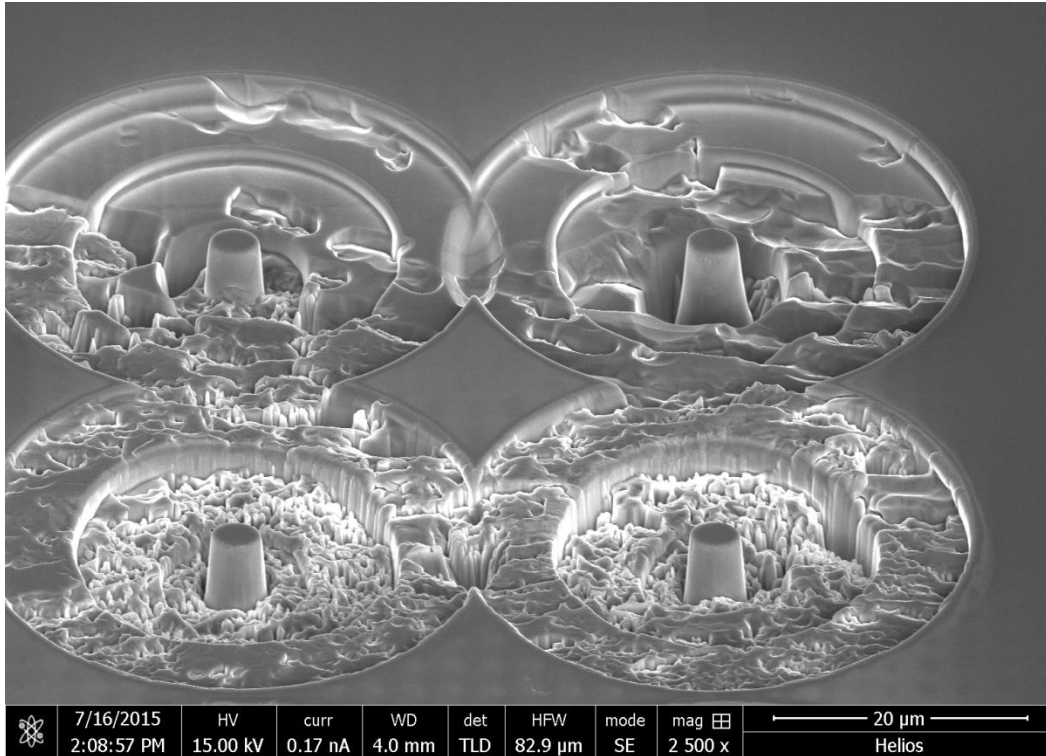
Calculs et mesures



$$u_p = u_m - u_{bâti} - u_{sub} - u_{ind} \quad \Rightarrow \quad u_p = u_m - u_{bâti} - \frac{F}{2rE_{sub}^*} - \frac{F}{2rE_{ind}^*}$$

$$V_o = \pi r^2 (h_o - u_p) \quad \Rightarrow \quad r = \sqrt{\frac{V_o}{\pi (h_o - u_p)}}$$

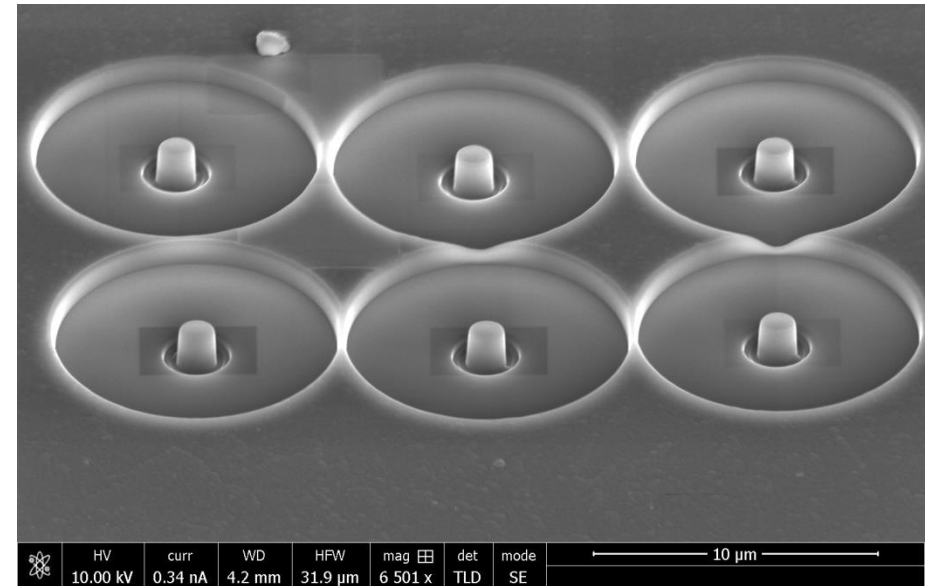
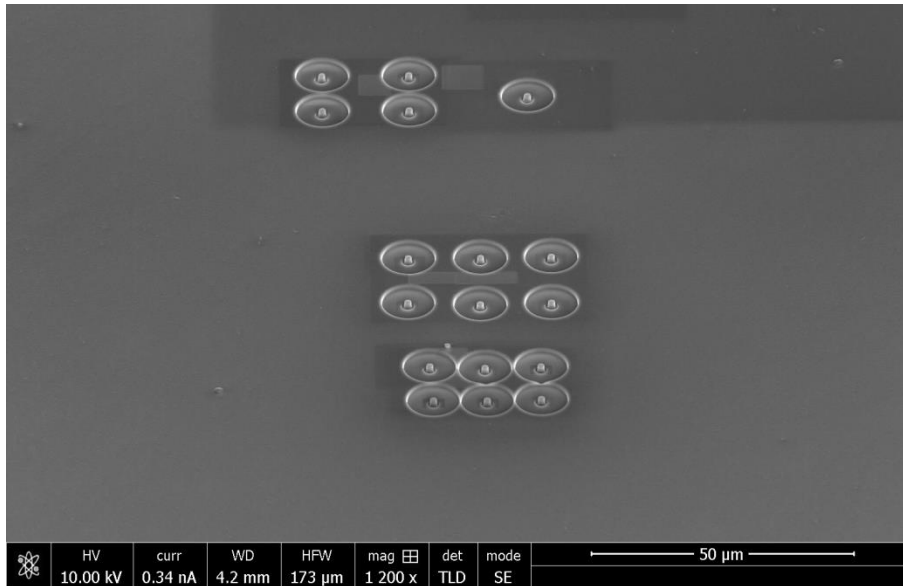
Difficultés d'usinage



- Hétérogénéité
- Control de la taille
- Control de la forme
- Visualisation du pilier
- Temps de fabrication

Fe pur , Thèse D. TUMBAJOY (2015)

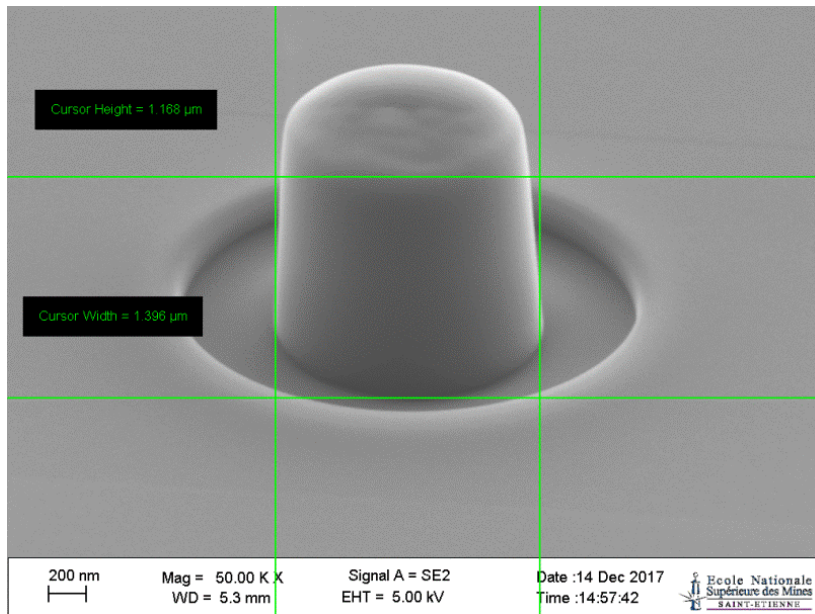
Réalisation de μ -piliers sur revêtements DLC



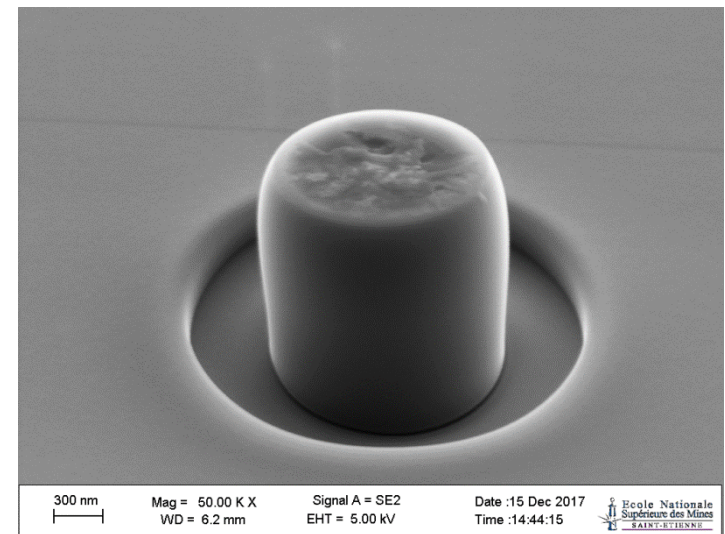
- Conditions Faisceau (30kV 790pA puis 40 pA)
- Usinage circulaire(de l'extérieur vers l'intérieure)
- Temps par pilier (30 à 40 min)

Micro-Compression de piliers.

Pilier avant compression (FIB)



Pilier après compression (FIB)



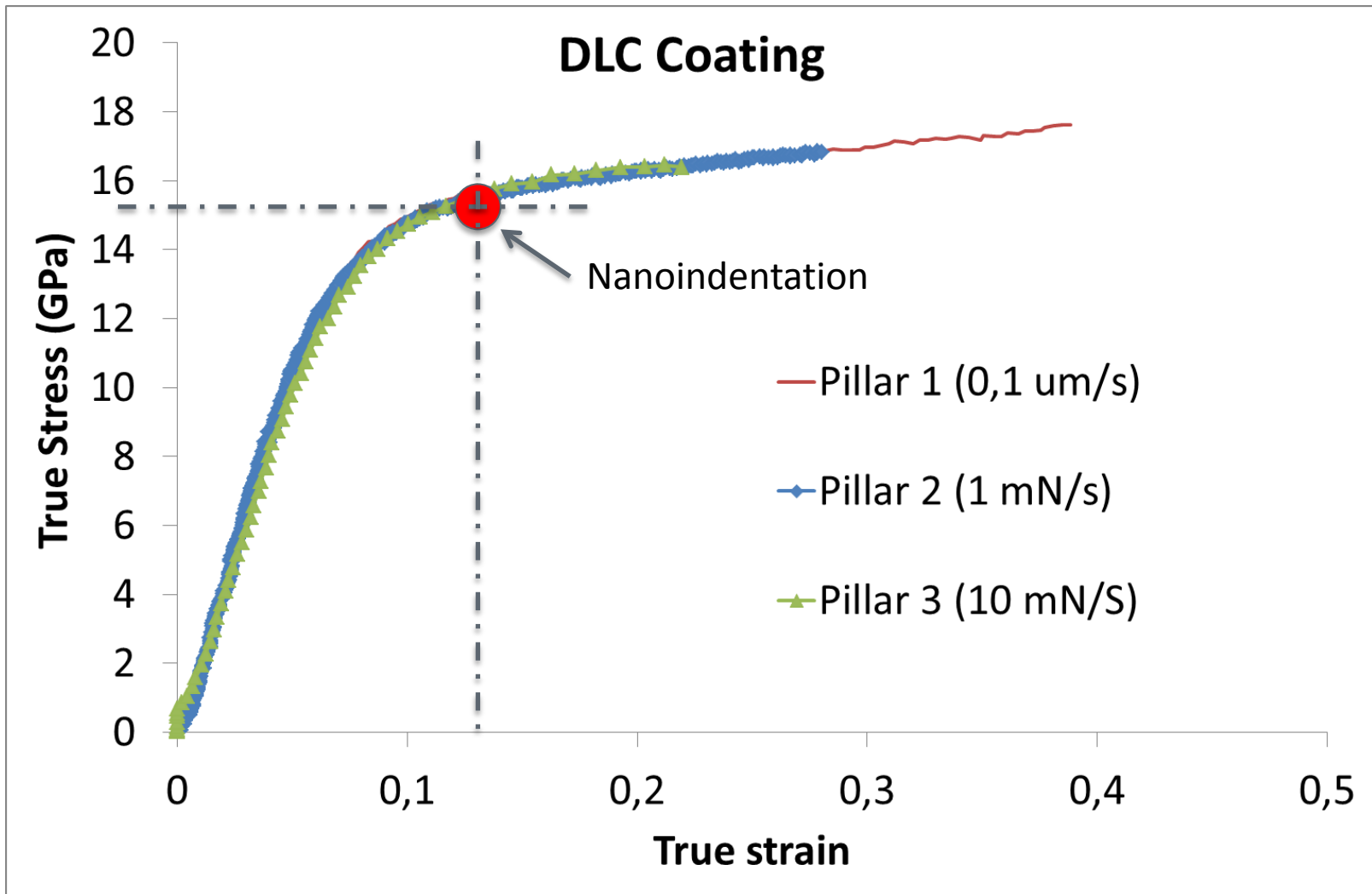
- Dimensions pilier:
 - hauteur: 1,5 µm
 - diamètre: 1µm

Conditions d'essai:

- Flat punch 10 µm
- Control en déplacement

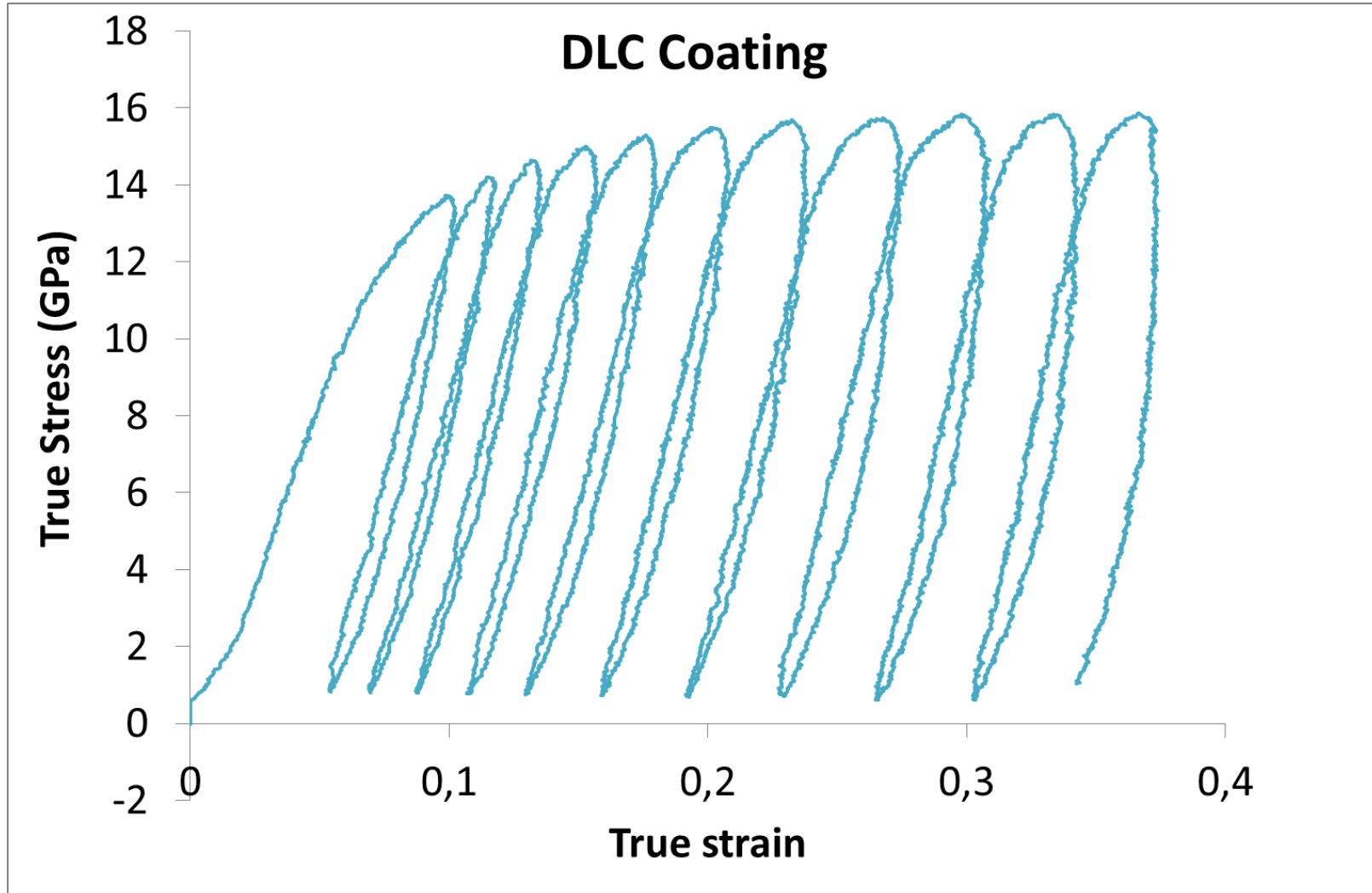
Micro-piliers

Micro-Compression de piliers.



Micro-piliers

Micro-Compression de piliers (cycles).



○ Micro-poutres

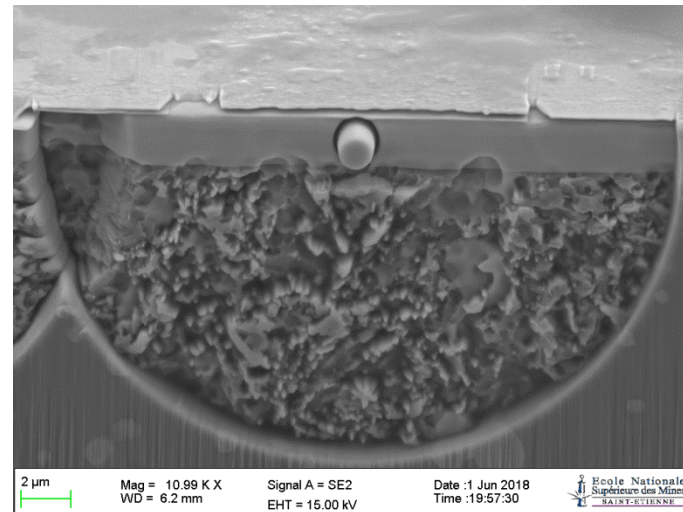
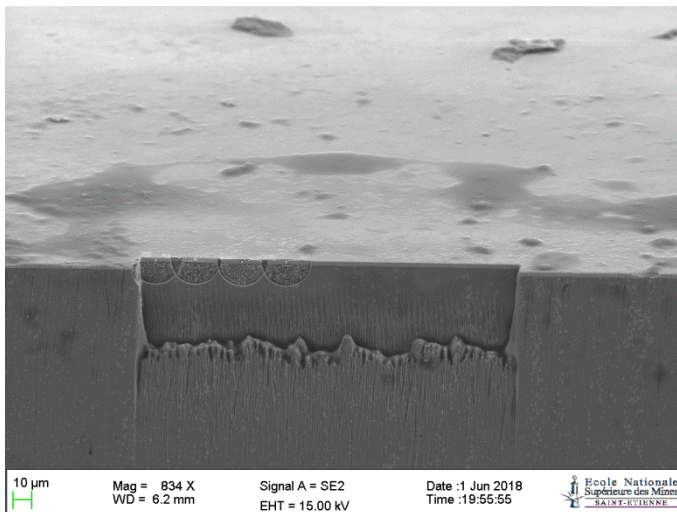
- Optimisation paramètres d'usinage FIB
- Réalisation de μ -poutres de différentes géométries
- Observation in situ du fléchissement des μ -poutres
- Méthode inverse de détermination des contraintes résiduelles

○ **Micro piliers**

- **Essai de Micro-compression (Simple et Cyclique)**
- **Accord avec les mesures de nano-indentation**
- **Première caractérisation du comportement élasto-plastique de revêtements DLC**
- **Le revêtement DLC a un comportement très proche du verre de silice (peu d'écrouissage)**

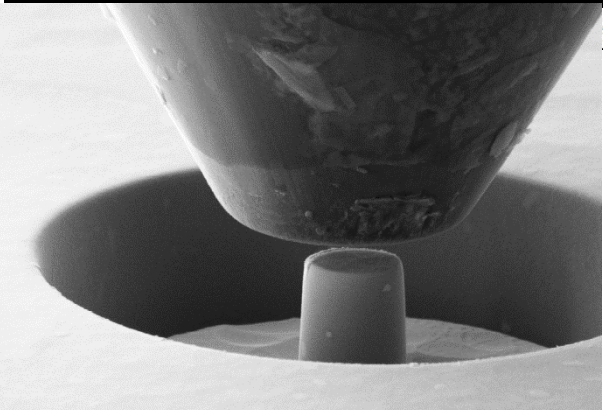
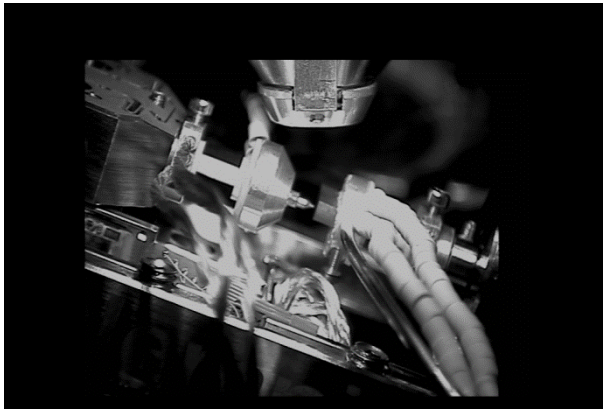
○ Dépôt DLC/Acier

- Influence de la sous-couche (indentation + micro-piliers)
- Essai de flexion (comportement interfaces)
- Micro compression en cross- section:



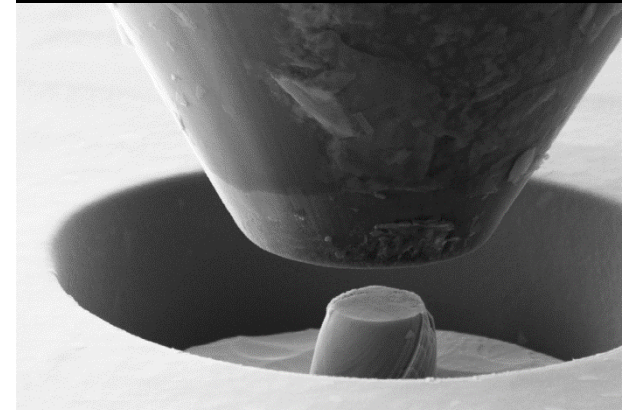
○ Dispositif indentation/compression in situ

- Module Haute Dynamique (3000 s⁻¹)
- Module Haute température :



2 μm Mag = 10.36 K X Signal A = SE2 Date :31 May 2018 Ecole Nationale Supérieure des Mines Saint-Étienne Time :12:46:16

Pilier alliage
HS25
Comprimé à 550 °C



2 μm Mag = 10.36 K X Signal A = SE2 Date :31 May 2018 Ecole Nationale Supérieure des Mines Saint-Étienne Time :13:07:14



Merci pour votre attention