



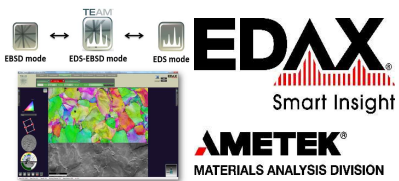
Upgrade MEB/MET



Rénovation de microscope  
EBIC/EBAC  
Topo 3D MEB



Equipement de micro-analyse



EDS / EBSD / WDS /  $\mu$ XRF

# EDX Detectors & Equipment for Nano



Ultra-microtomie  
Cryo-ultra-microtomie  
Cryo-préparation



Evaporateur carbone  
Métaliseur

Equipement ex-situ



Amincisseur ionique MEB/MET  
Porte échantillons (Tomo/Cryo)  
Polissage (Mécanique/Electrochimique)  
Plasma cleaner



Solutions de tests nano-mécaniques  
Indenteurs MEB 2,5N / HT 1000°C



Décontaminateur plasma



Solutions de nano-probing  
Nano-robots

Equipement in-situ



Platine en température in situ  
Platine de traction/compression in situ



Mesures environnementales in-situ MET



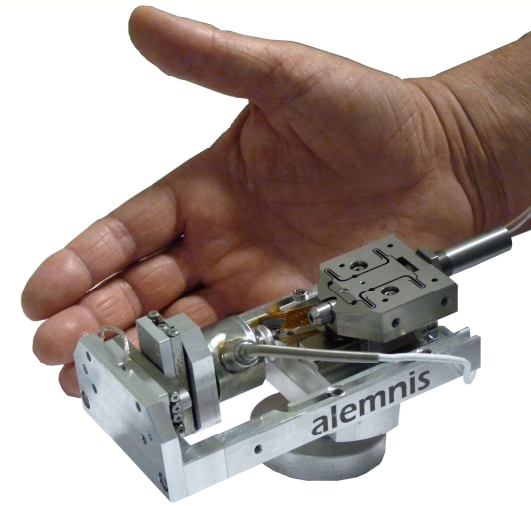
Cathodoluminescence  
Microscopie corrélative

3 décembre 2019

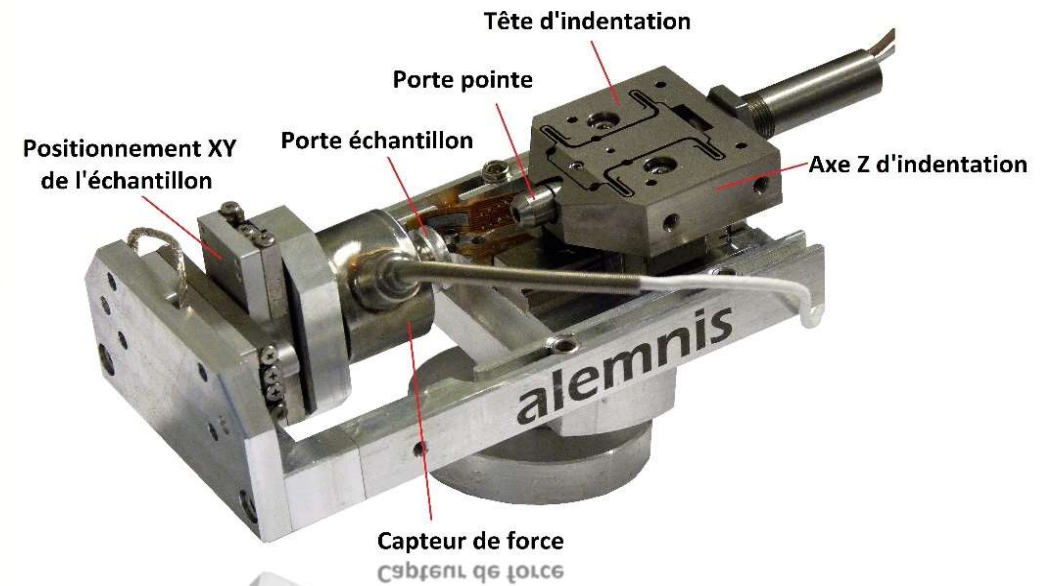
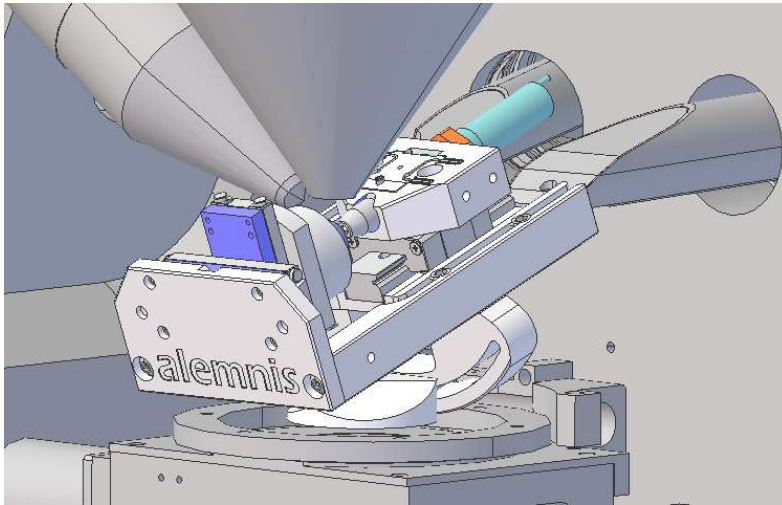
## TESTS NANOMECHANIQUES

- Alemnis est une spin-off de l'EMPA\*
- Alemnis est basée à Thun, à côté de Bern, au sein de l'institut EMPA
- Alemnis a été créée en **2008**

\*EMPA: Swiss Federal Laboratories of Materials Science & Technology

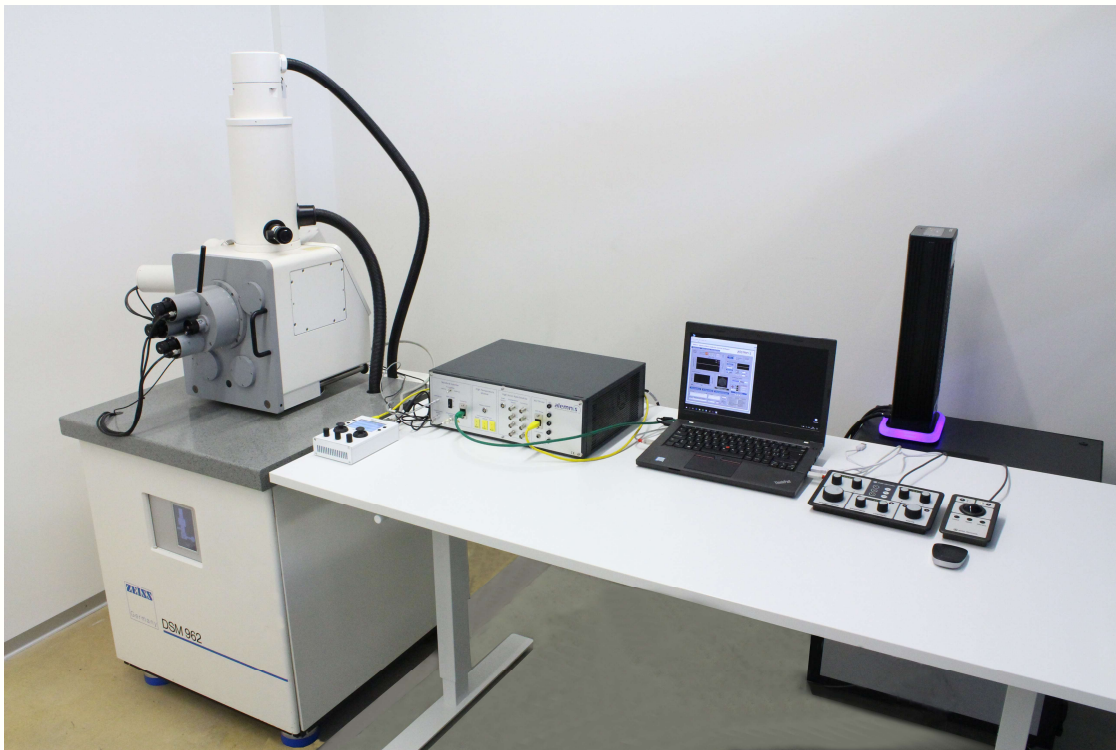


# Historique Alemnis



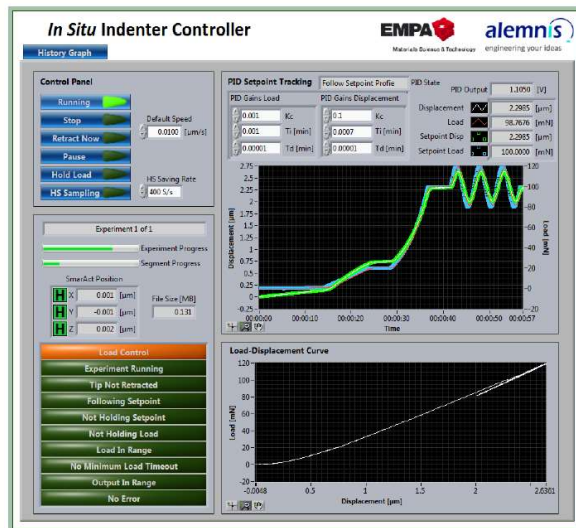
Le développement a commencé en 2001, premier prototype en 2004  
Alemnis a conçu le premier nano-indenteur compatible MEB sur le marché

# Intégration typique de l'ASA dans un MEB



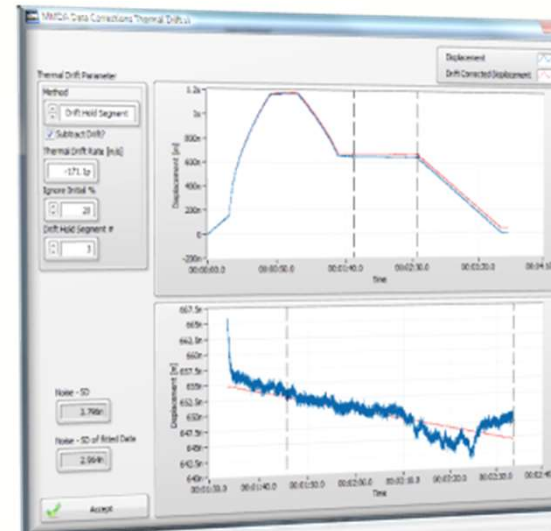
# 2 logiciels avancés flexibles (Labview)

## Indentation software (AMICS)



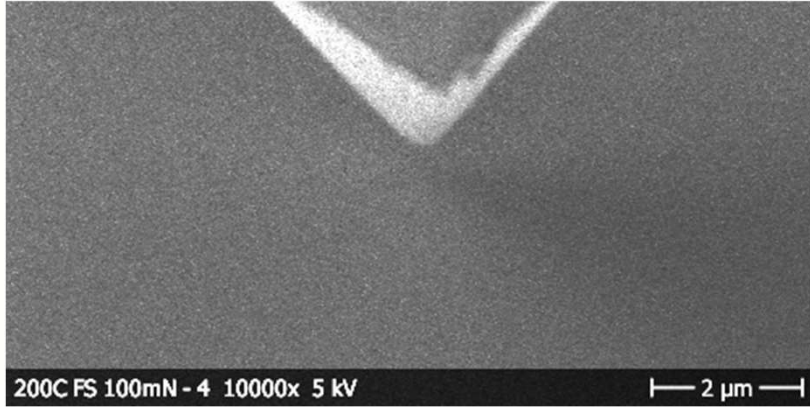
- Fully configurable
- Set-up, runs and records experiments
- Visualization of the main experimental parameters and measures
- Fully customizable (LabVIEW)

## Advanced Data Analysis (AMMDA)

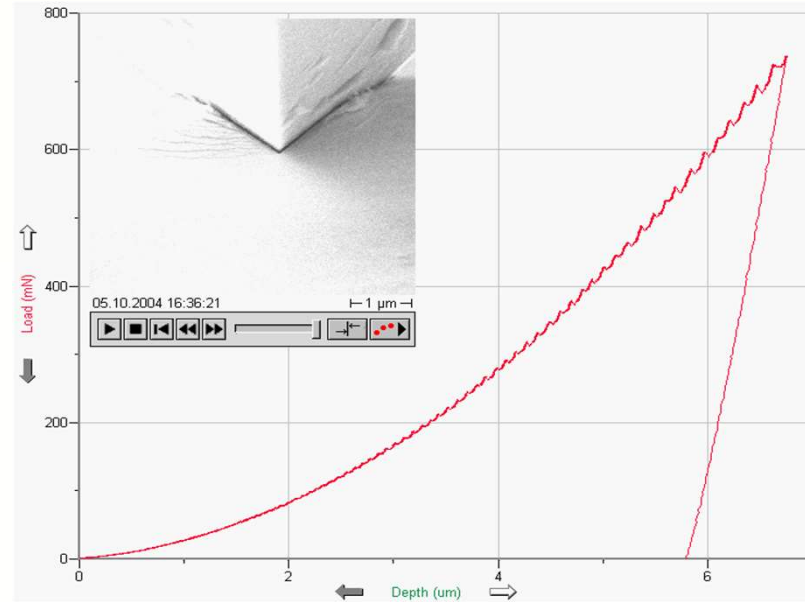


- Data analysis and data corrections
- Extract relevant parameters (hardness, modulus, stress/strain, indentation energies, elastic and plastic work, yield strength, creep, etc.)
- Indenter selection (flat punch, Berkovich, cube corner, etc.) and analysis

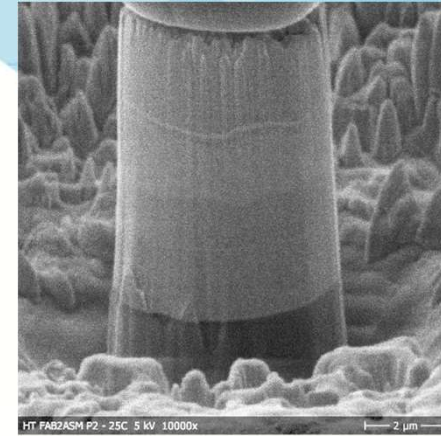
# Pourquoi le MEB?



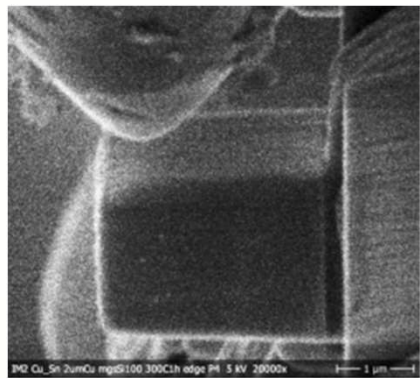
Indentation



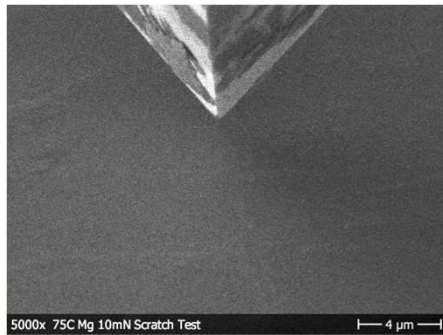
“Seeing is believing”  
Le cas des verres métalliques !



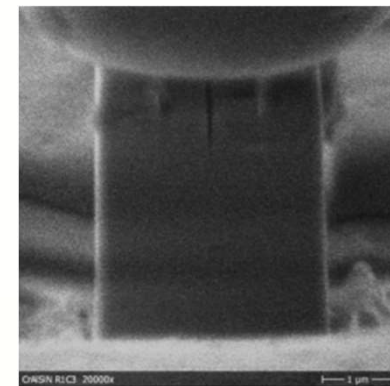
Compression



Flexion



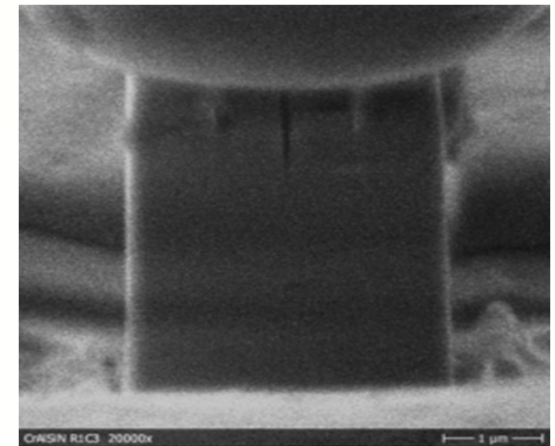
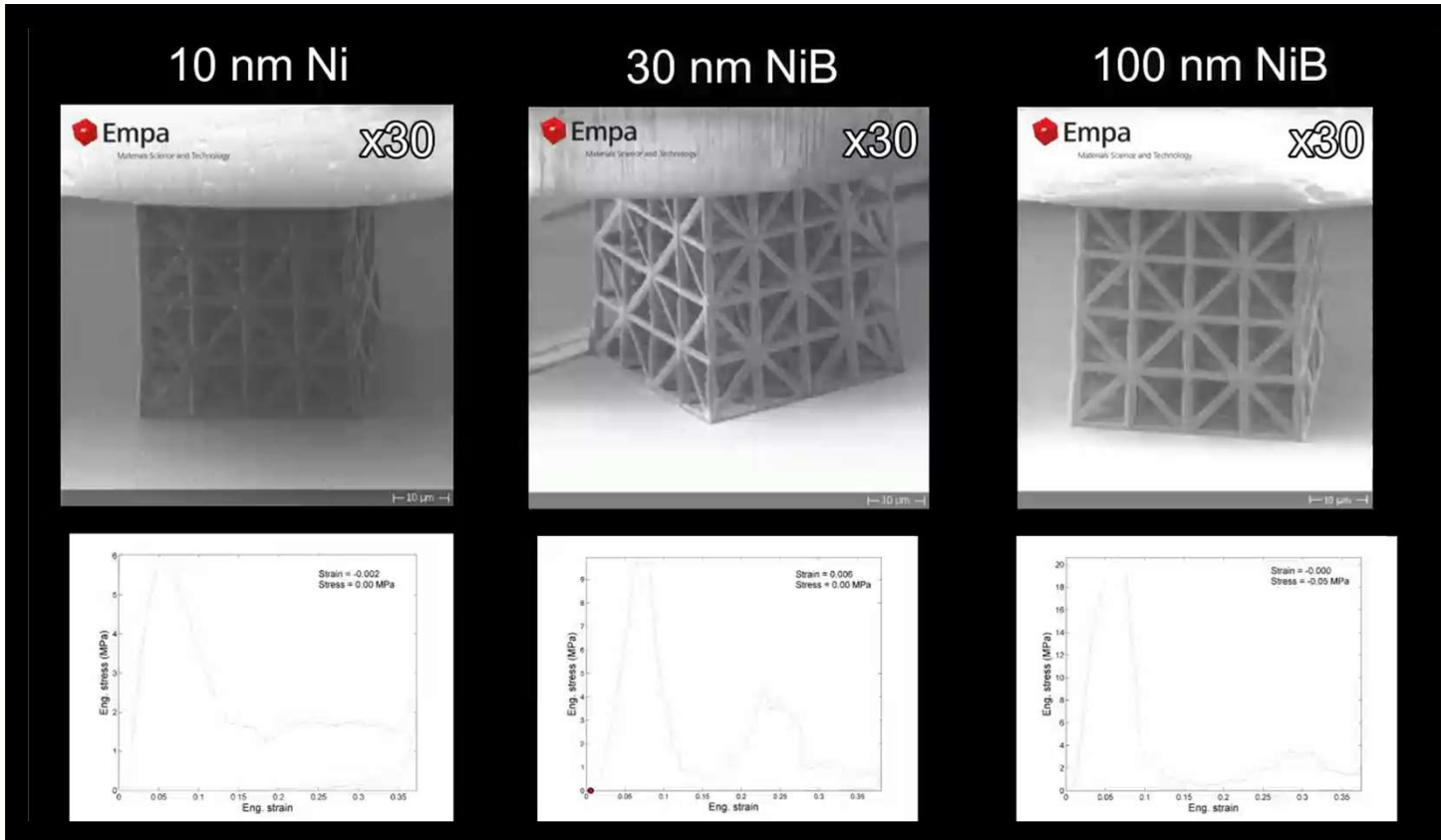
Scratch/Abrasion



Résistance à la fracture

**Le MEB est un des outils les plus utilisés en science des matériaux**

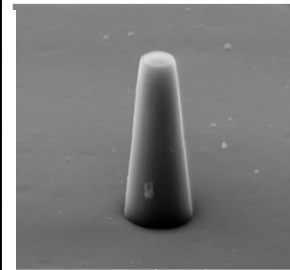
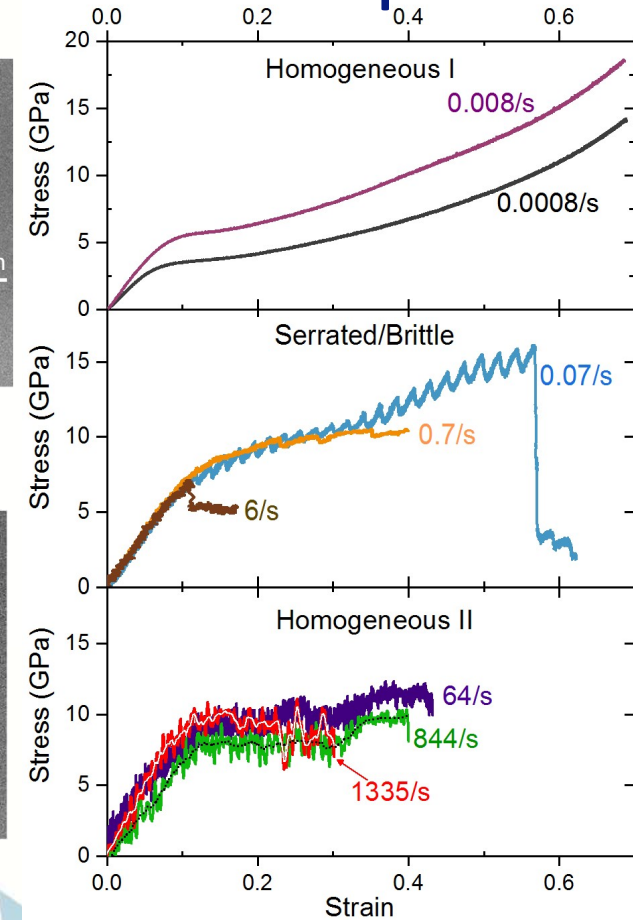
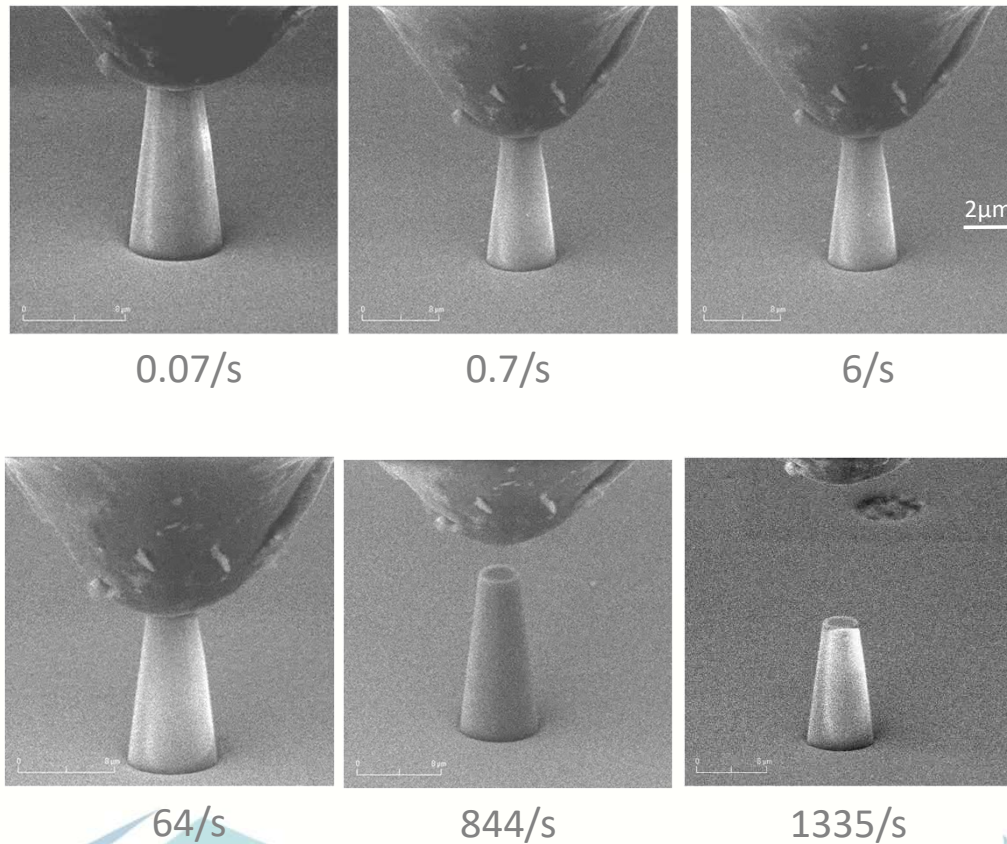
# True displacement mode: Analyse de défaillance



(Courtesy of Univ. of Cambridge, EMPA)

- Contrôle parfait de la déformation -> Mesures quantitatives précises de la force de réaction
- La structure de l'échantillon est préservée durant l'expérience (même après des glissements dans la résistance de la structure).

# Dépendance à la vitesse de déformation dans le cas de l'étude de micropilliers en silice amorphe

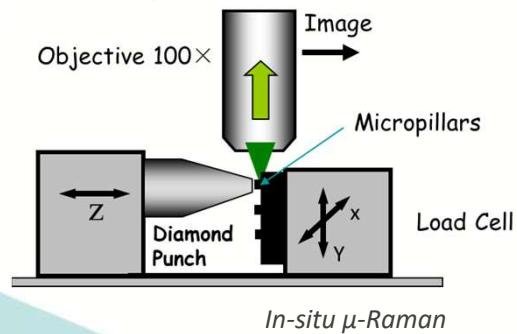
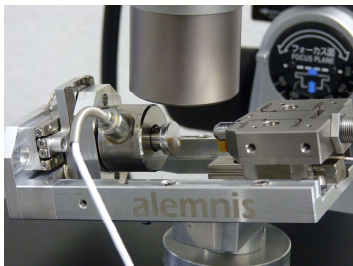


Transition ductile-friable-ductile pour un taux de déformation de 0.0008/s à 1335/s (8 ordres de grandeur)

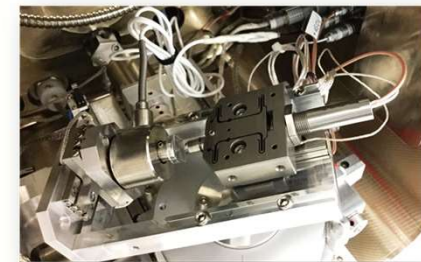
# Applications Alemnis

## En combinaison avec d'autres techniques

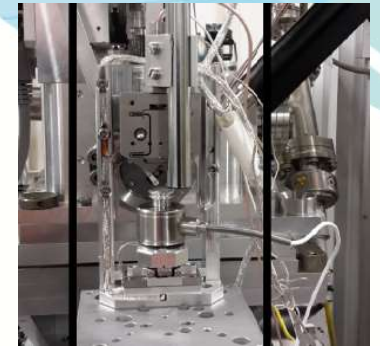
- Synchrotron, XRD, tomo 3D
- EBSD
- Corrélation d'images
- AFM
- $\mu$ -Raman
- Microscopie optique



NenoVision LiteScope SPM



In-situ SEM, EBSD, DIC



XRD, Synchrotron

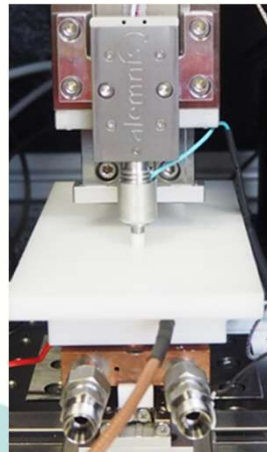
# Applications Alemnis

## Control environnemental

- Haute temperature 1000 °C
- Basse temperature -150 °C
- Nucléaire (labo chaud)
- Sous gaz (N<sub>2</sub>, Ar, H<sub>2</sub>, O<sub>2</sub>, etc..)
- Bio-indentation (RH & T)
- Cellule électro-chimique



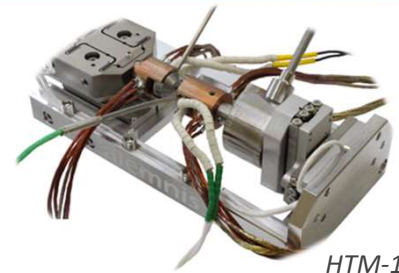
Electro-chemical cell



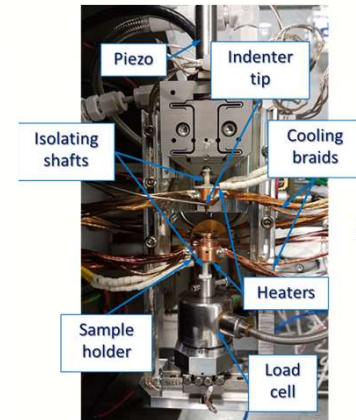
Bio-indenter (IBIO)



"Hot" chamber

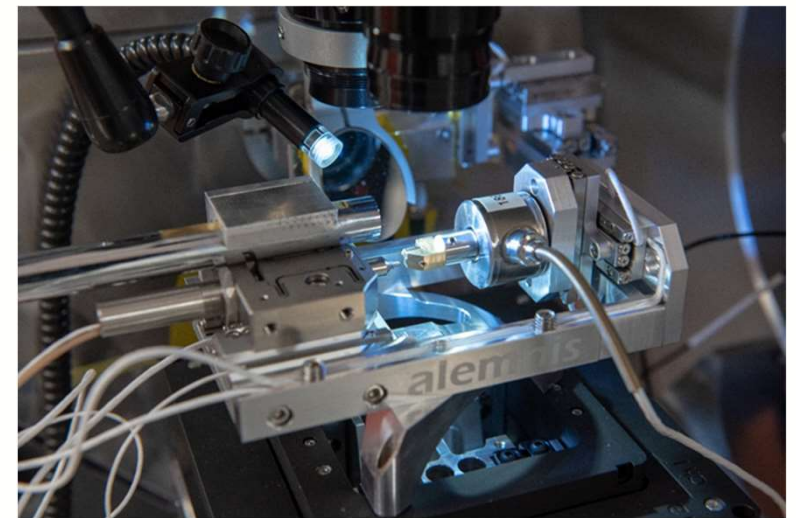
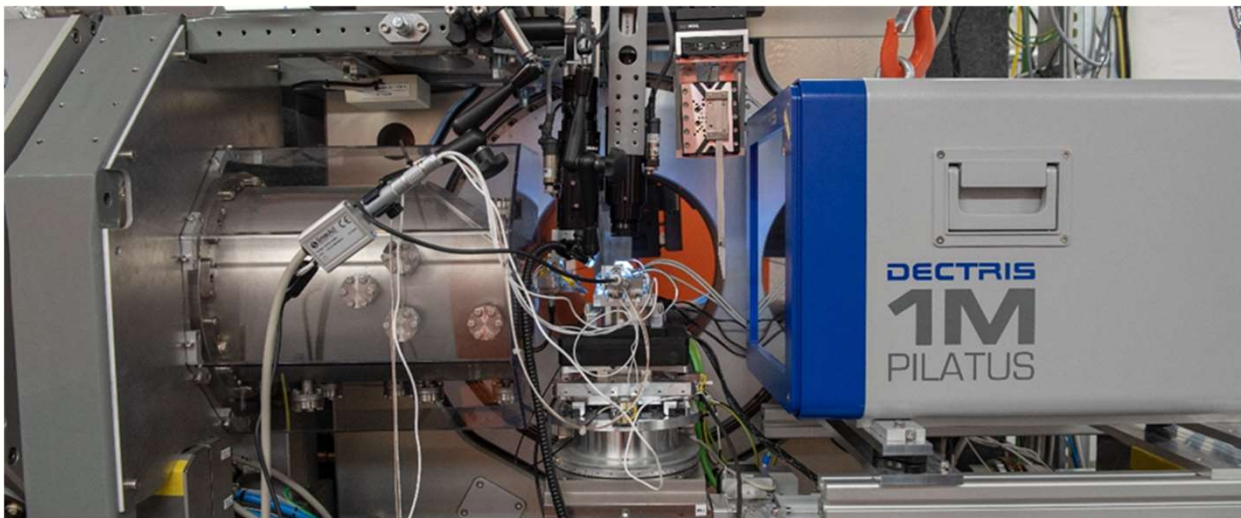


HTM-1000



LTM-CRYO

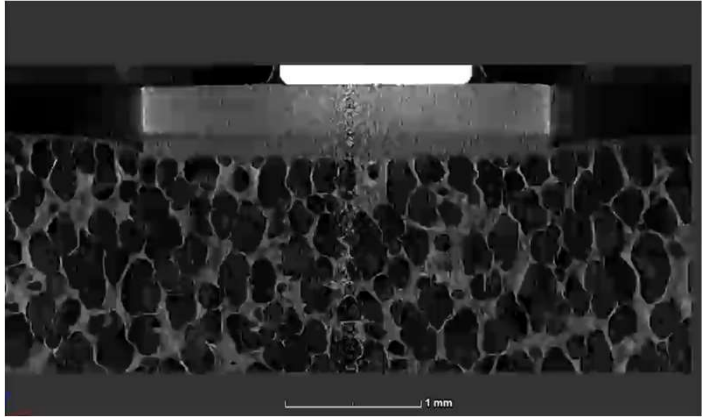
# ASA sous Synchrotron



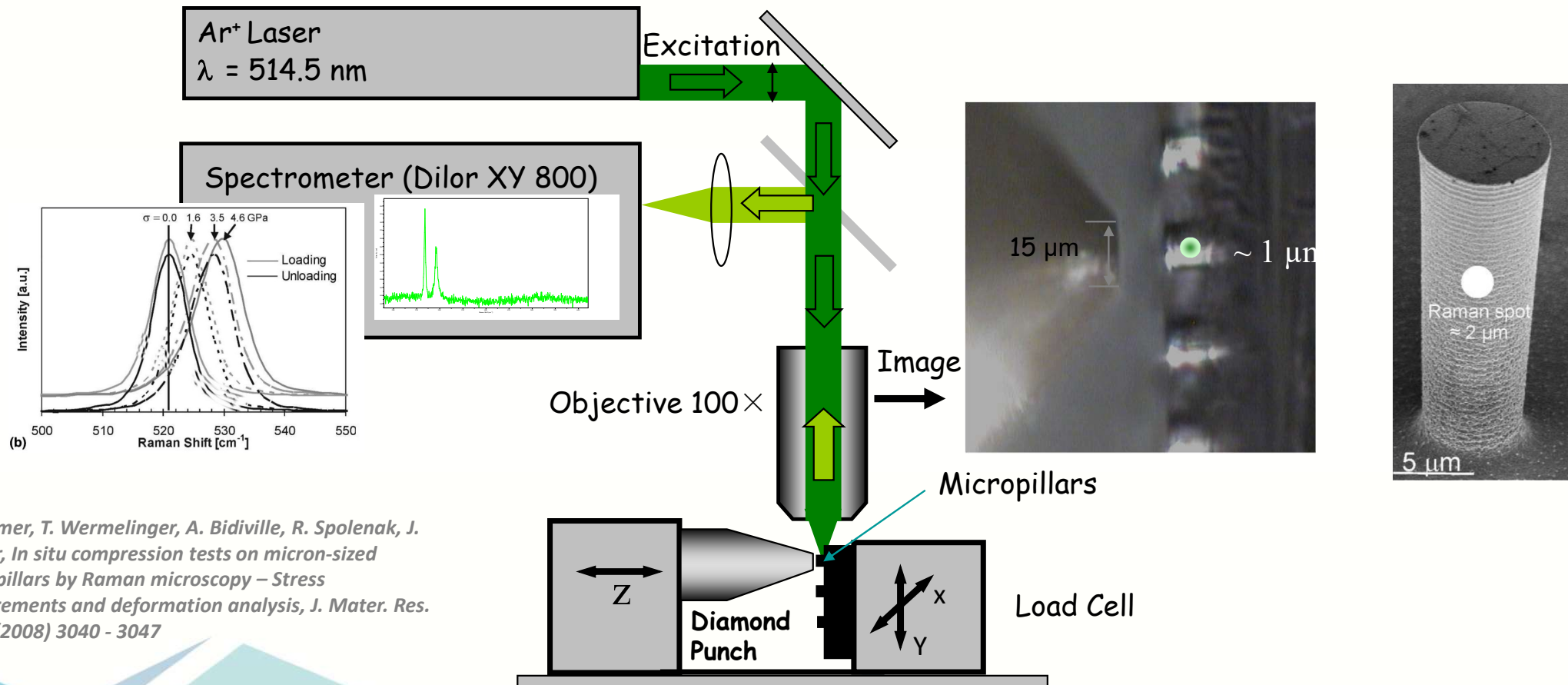
*(Courtesy of Sebastian Kalbfleisch, MAX IV Laboratory, Lund, Sweden)*

- J. Gamcová, G. Mohanty, Š. Michalik, J. Wehrs, J. Bednarčík, C. Krywka, J. M. Breguet, J. Michler, and H. Franz, Mapping strain fields induced in Zr-based bulk metallic glasses during in-situ nanoindentation by X-ray nanodiffraction, *Applied Physics Letters* 108 (2016) 031907
- A. Bhowmik, G Sernicola, J Lee, T. B. Britton, W. Liu, T-S Jun, M. Karimpour, D. S. Balint, F. Giuliani, Deformation behaviour of [001] oriented MgO using combined in-situ nano-indentation and micro-Laue diffraction, *Acta Materialia* 145 (2018) 516-531

# ASA en tomo X 3D

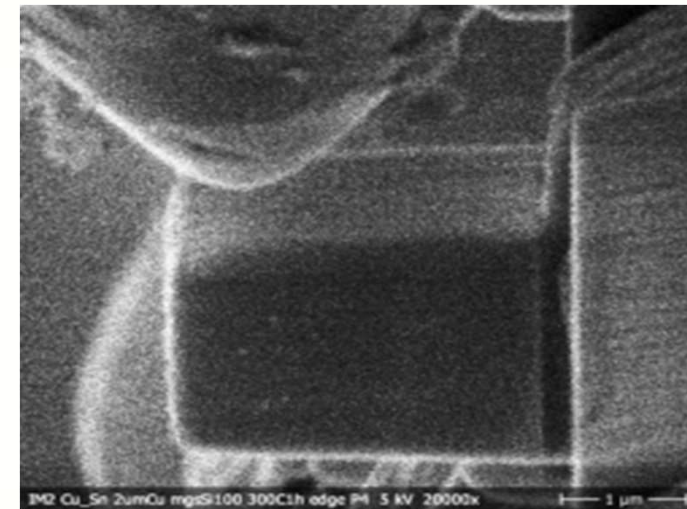
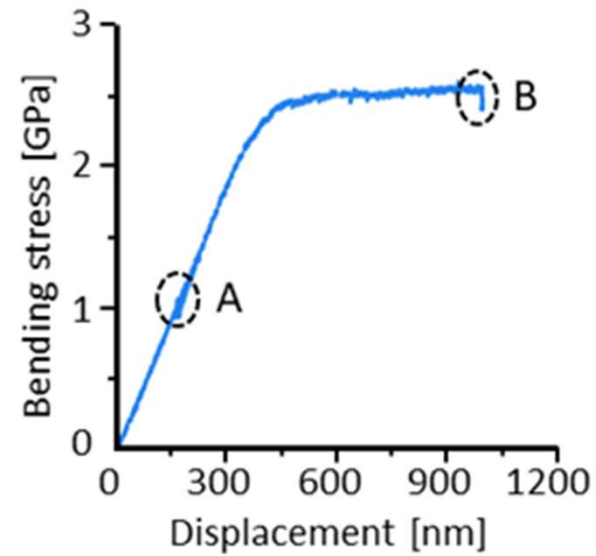
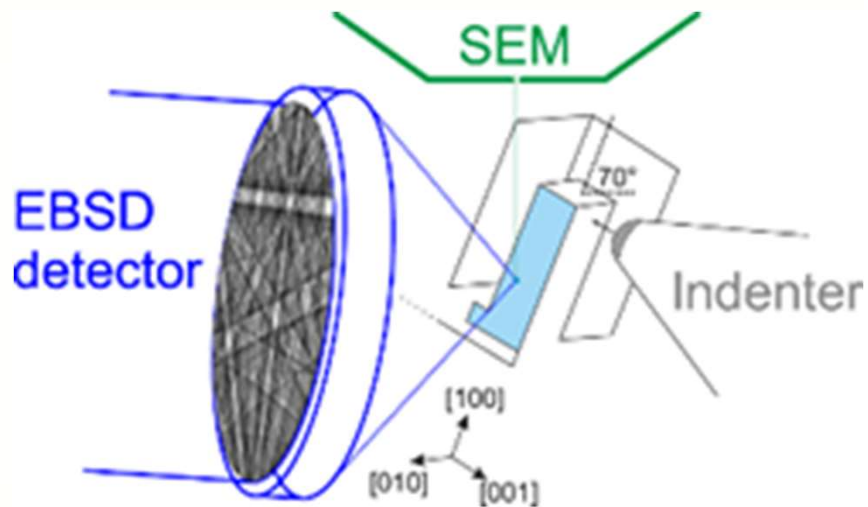


# Compression In-Situ Raman



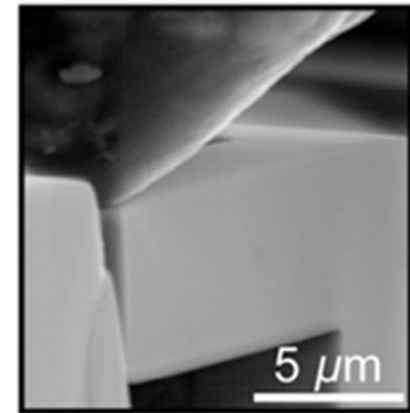
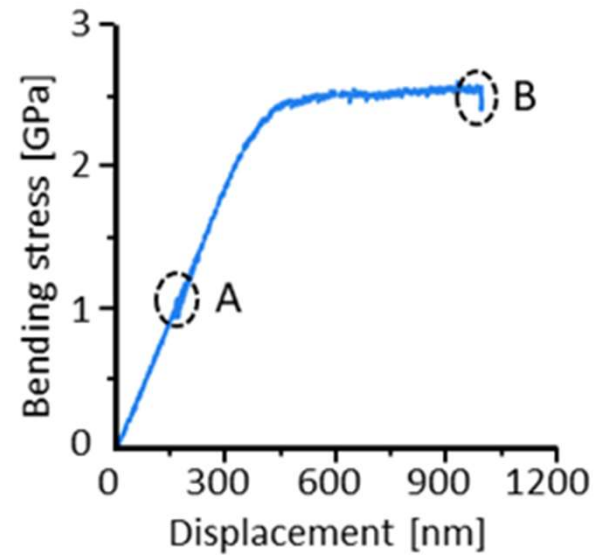
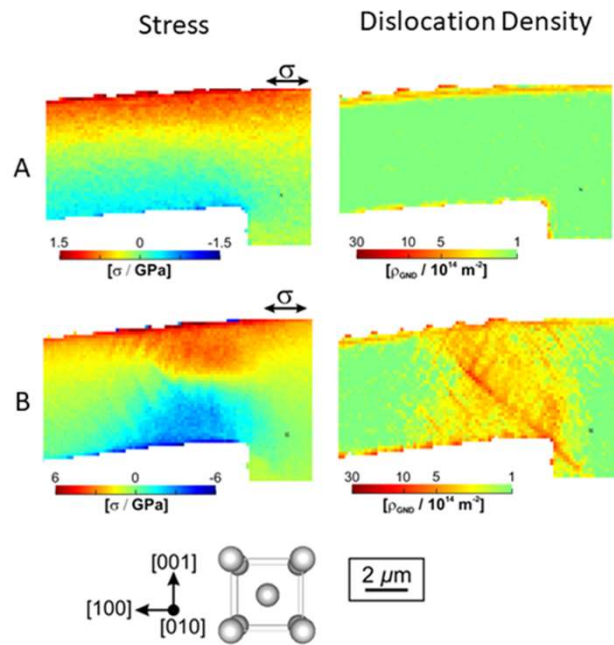
*K. Wasmer, T. Wermelinger, A. Bidiville, R. Spolenak, J. Michler, In situ compression tests on micron-sized silicon pillars by Raman microscopy – Stress measurements and deformation analysis, J. Mater. Res. 23, 11 (2008) 3040 - 3047*

# ASA avec EBSD



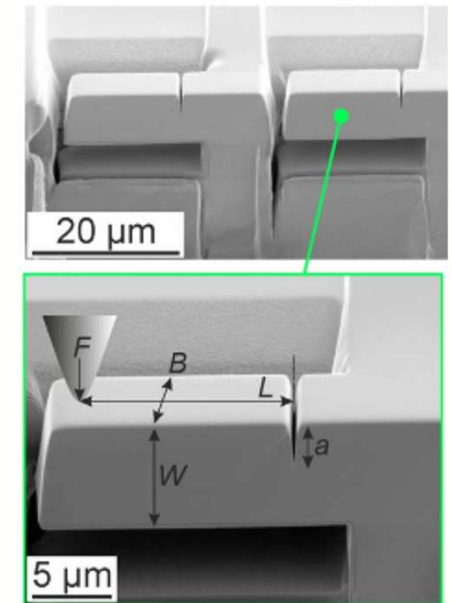
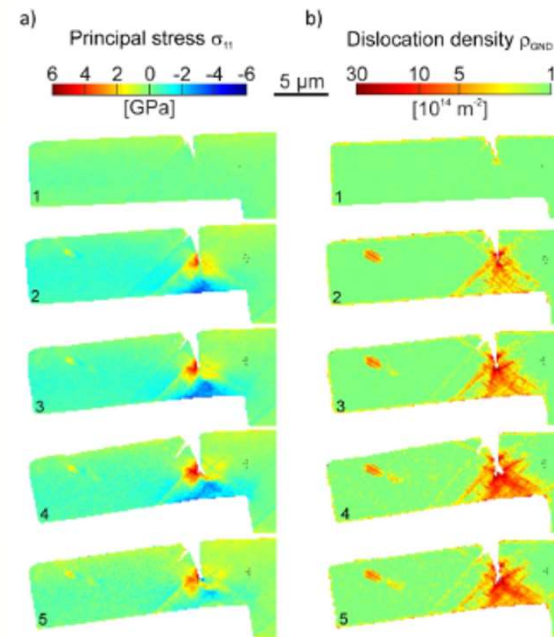
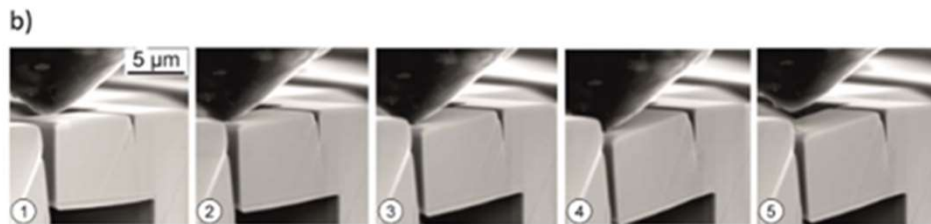
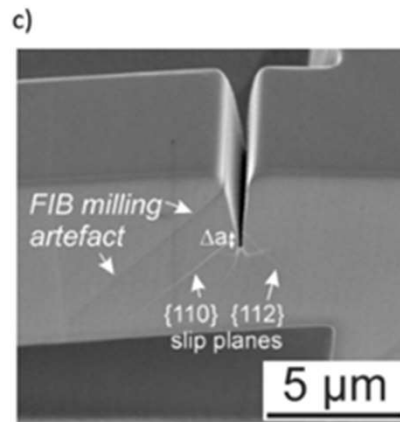
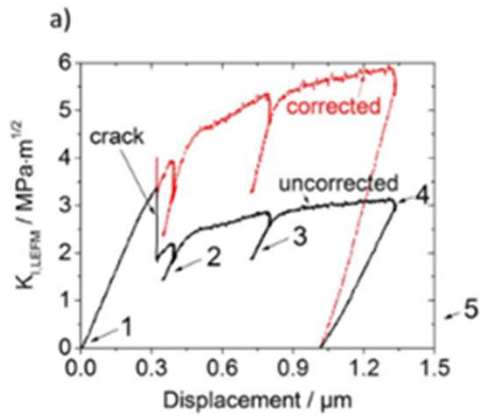
*J. Ast, G. Mohanty, Y. Guo, J. Michler, X. Maeder, In situ micromechanical testing of tungsten micro-cantilevers using HR-EBSD for the assessment of deformation evolution, Materials and Design 117 (2017) 265-266*

# ASA combiné avec l'EBS



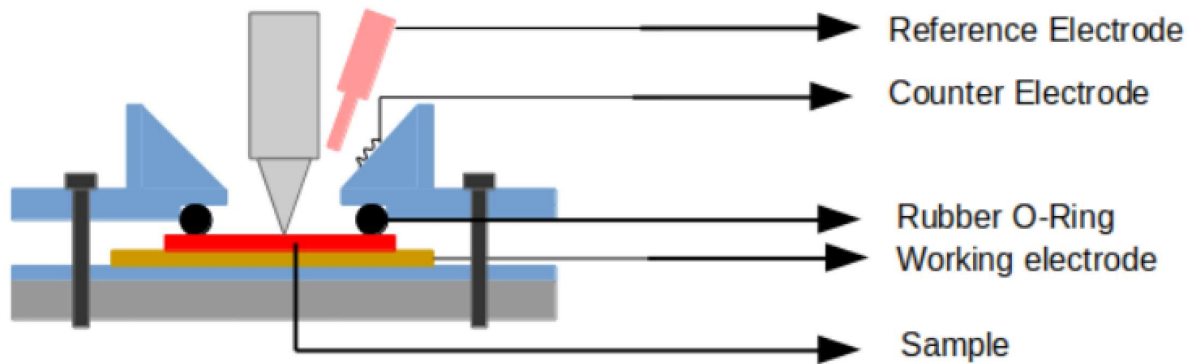
*J. Ast, G. Mohanty, Y. Guo, J. Michler, X. Maeder, In situ micromechanical testing of tungsten micro-cantilevers using HR-EBS for the assessment of deformation evolution, Materials and Design 117 (2017) 265-266*

# ASA combiné avec l'EBSD

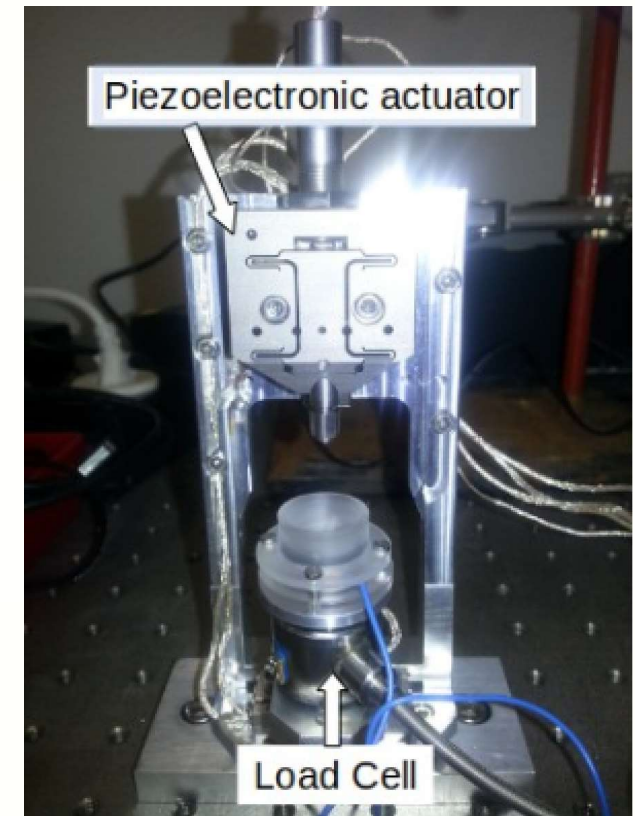


*J. Ast, M. N. Polyakov, G. Mohanty, J. Michler, X. Maeder, Mater. Sci. Eng. A 710 (2018) 400-412*

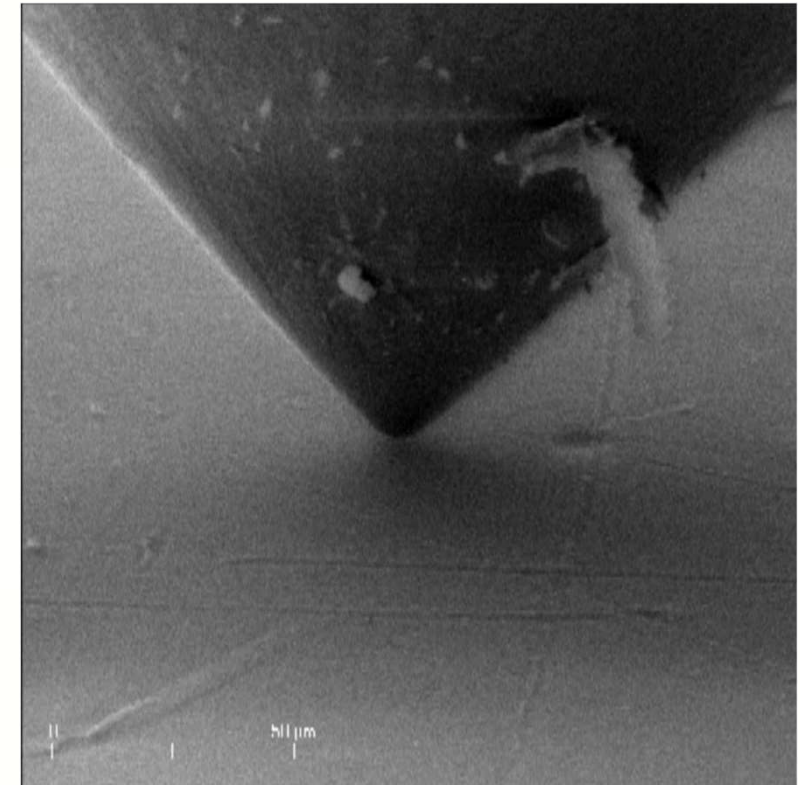
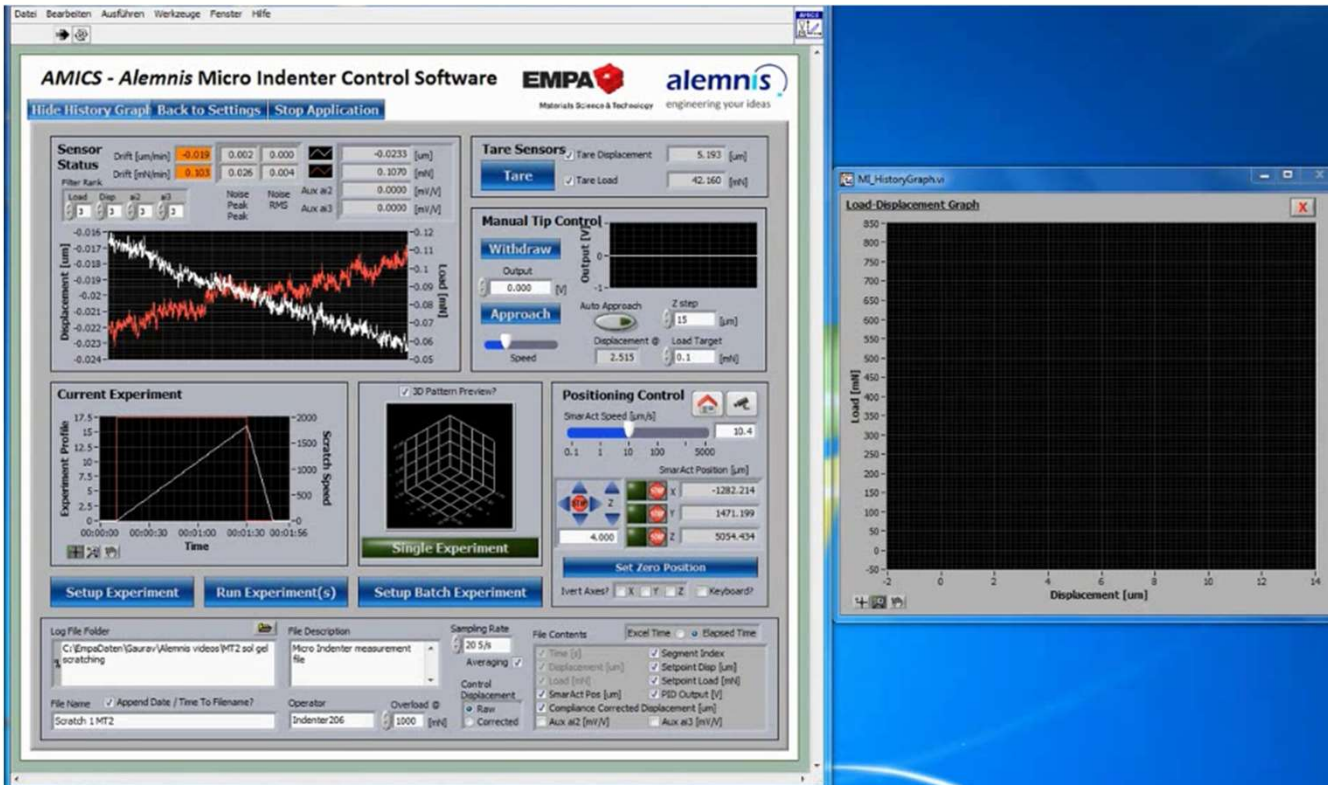
# Tests électrochimiques ex-situ



*MSc Thesis by Vance Nguyen, EPFL "Effect of Passive Oxide Layer on Plastic Deformation of Metals during Nanoindentation" (2016)*



# Tests de rayures

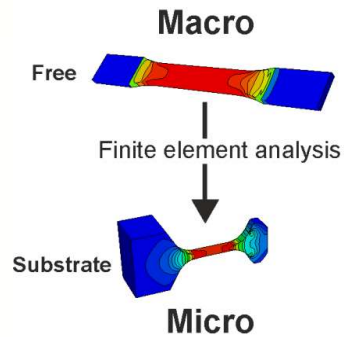


Des fissures périodiques coniques sur une silice solgel peuvent être observées en imagerie MEB

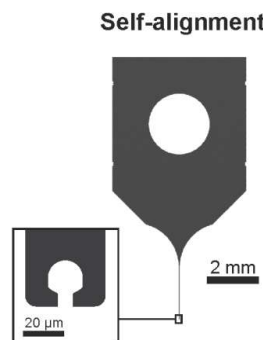
# Micro-traction

## A. Design

### 1. Sample design

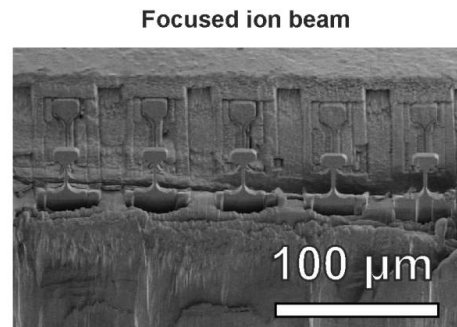


### 2. Gripper design



## B. Fabrication

### 3. Sample fabrication

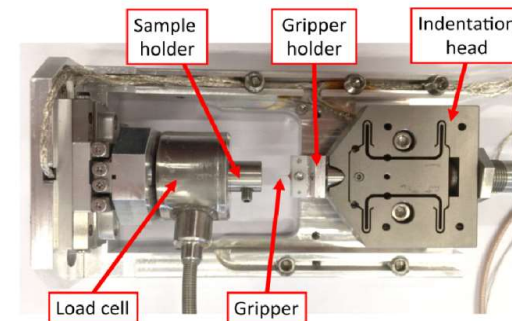


### 4. Gripper fabrication

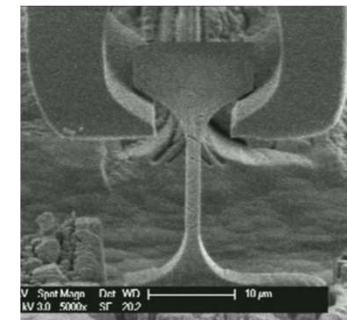


## C. Micromechanical testing

### 5. In situ microtensile setup

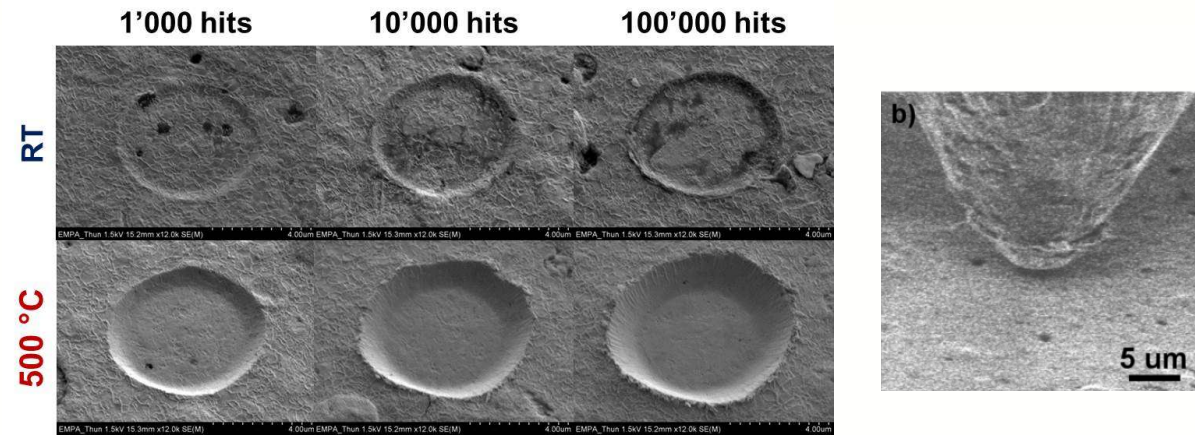
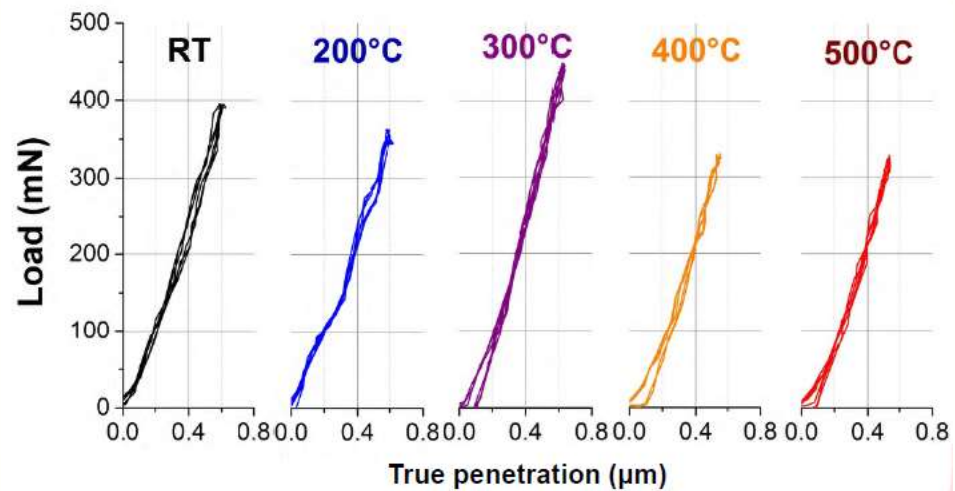


### 6. Validation self-alignment



# Tests d'impacts

Tests à haute température de nano-impacts sur des matériaux durs (CrN)  
Essais d'impacts à 1kHz



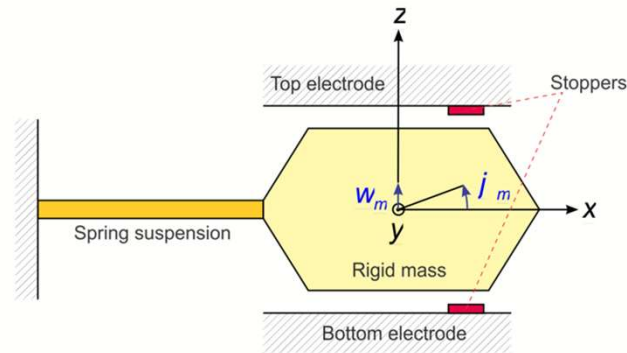
HR-SEM residual imprints from high-temperature nano-impact tests using a diamond flat punch onto CrN coated nitrided tool steel.

J. P. Best, G. Guillonéau, S. Grop, A. A. Taylor, D. Frey, Q. Longchamp, T. Schär, M. Morstein, J-M Breguet, J. Michler, Surf. Coat. Tech. 333 (2018) 178-186

# Tests de chocs

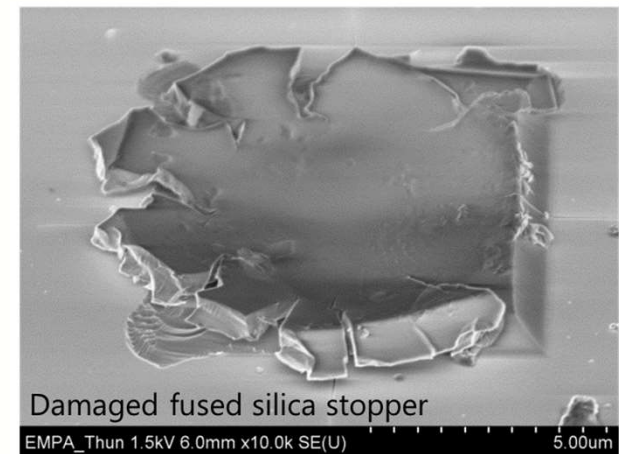
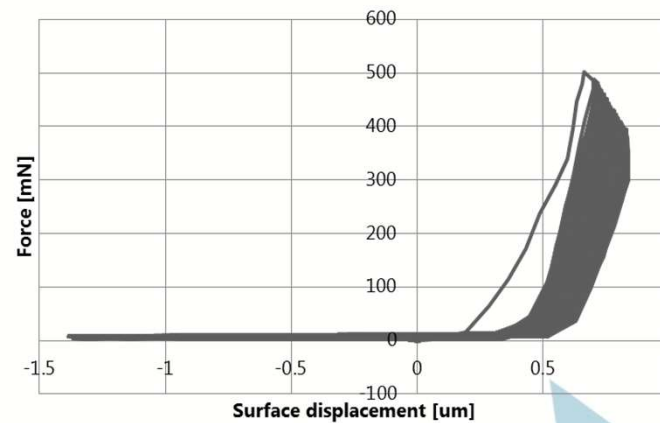
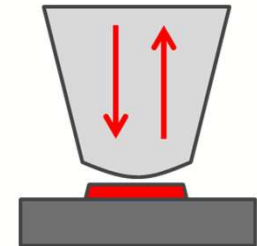
Tests répétitifs à fort taux de déformation : Fiabilité de MEMS dans le secteur de l'aérospatial

Simulation de dommage sur un accéléromètre capacitif, chocs sur les stoppers



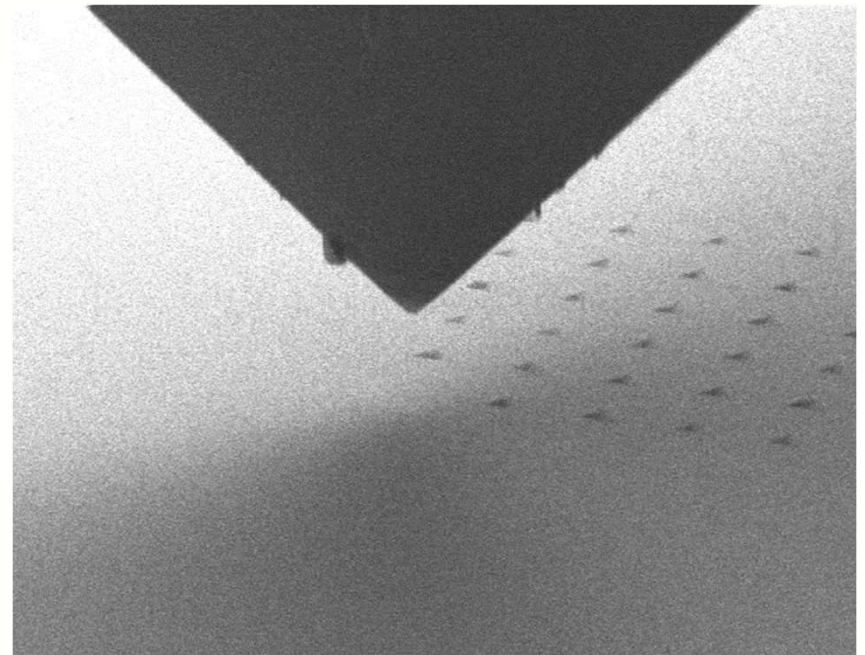
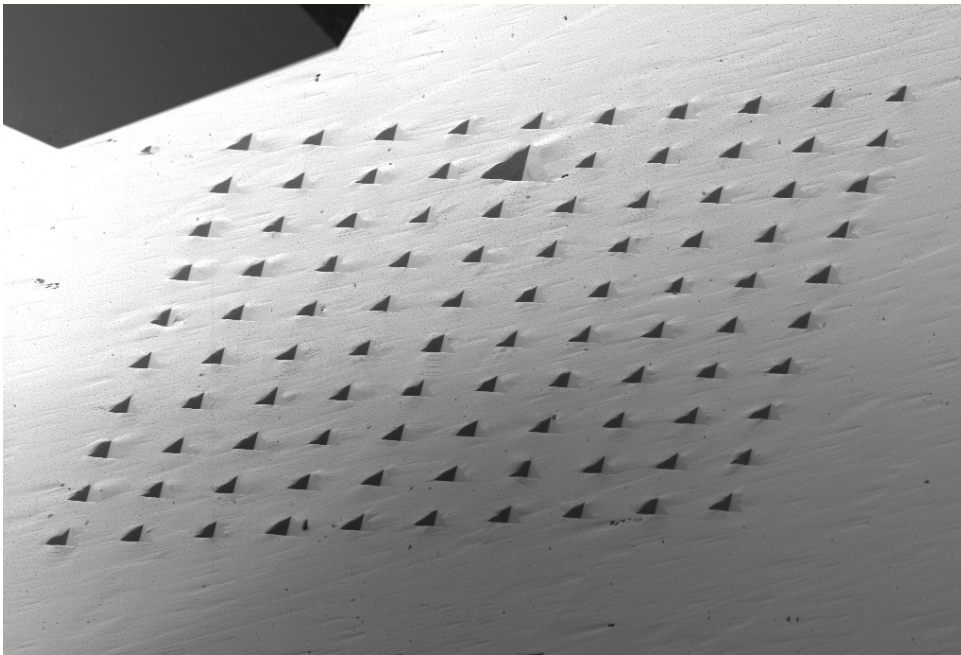
Tests at high frequencies to reproduce real shock conditions

100Hz, 0.4V, 2s

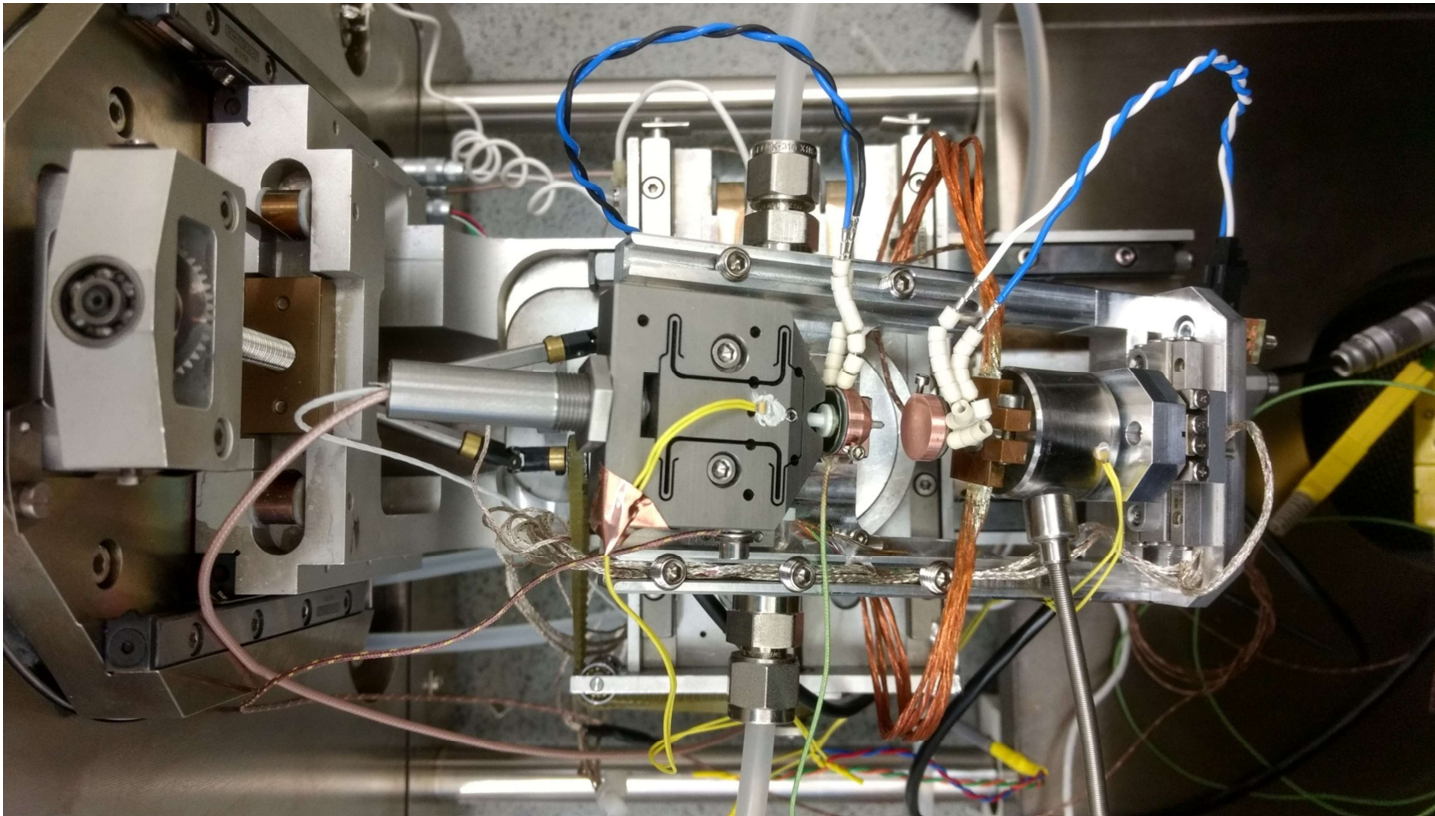


# Cartographie rapide

Grille d'essais (espacements de 15  $\mu\text{m}$ ) pour cartographier les propriétés de la surface avec une grande résolution spatiale. (Vitesse max : **5 indentations / seconde**)

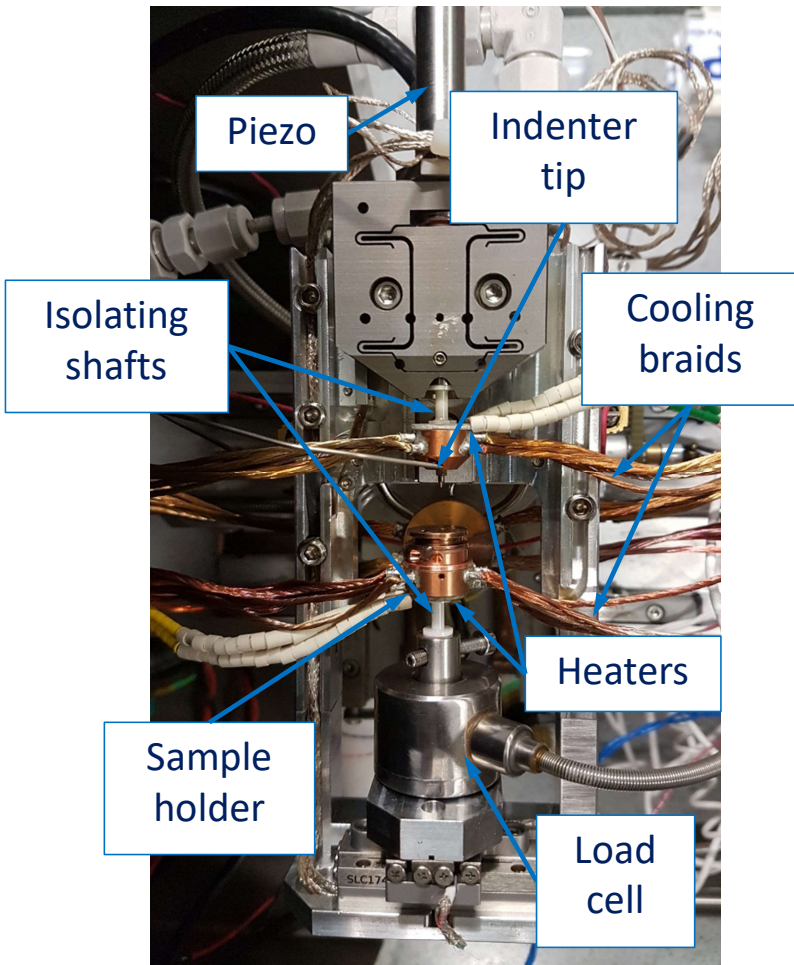


# Haute température In-Situ



- 400°C / 800°C / 1000°C
- Double chauffage combiné pointe et échantillon
- Des thermocouples sur l'échantillon et la pointe permettent une régulation de la température indépendante pour éviter la dérive thermique.
- Système de refroidissement de la structure avec water chiller.

# Basse température In-Situ

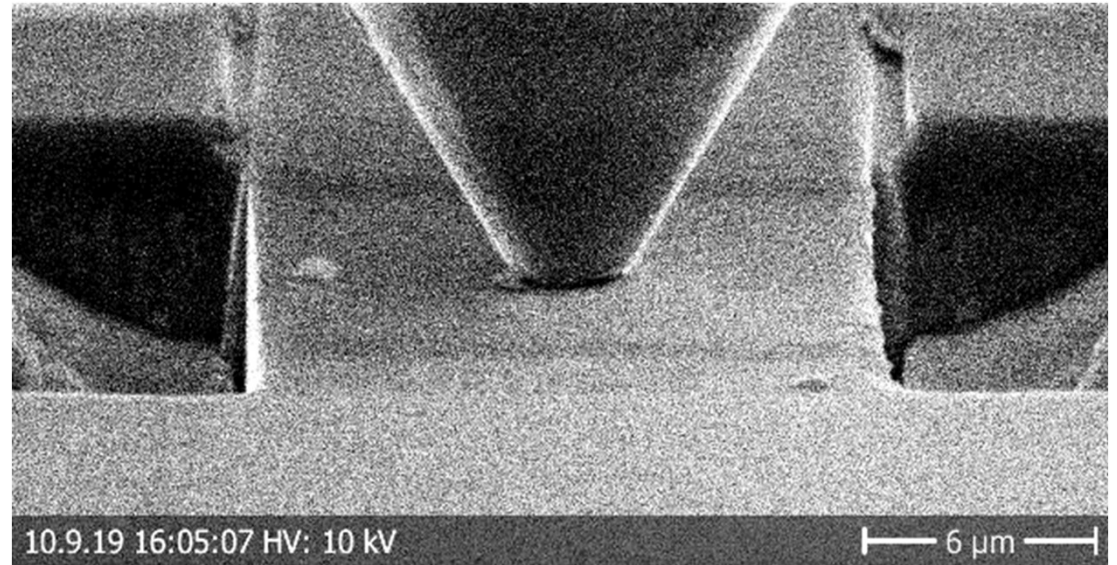
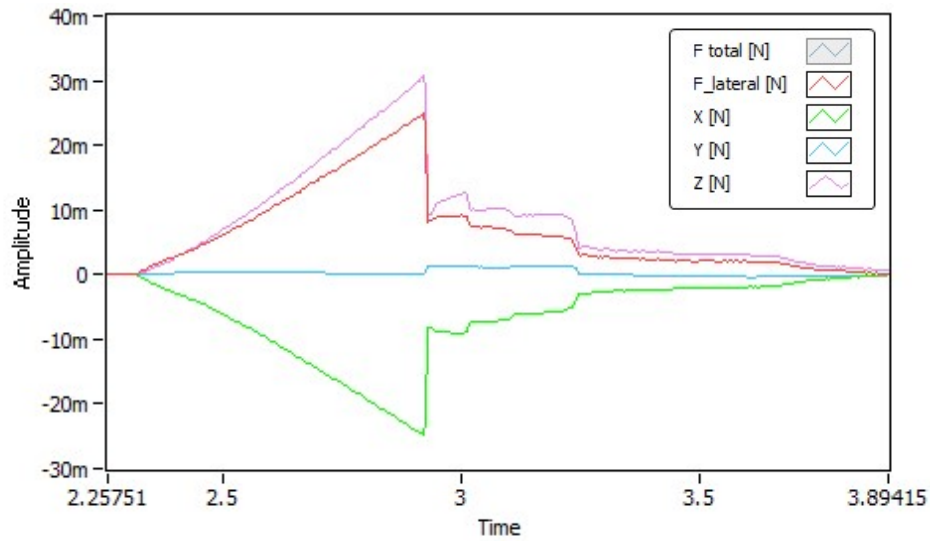


- 120K à 300K
- Taux de déformation:  $10^{-4}$  to  $10^{-1}$ s
- Gamme de force: 500mN
- Double refroidissement combiné pointe et échantillon
- Des thermocouples sur l'échantillon et la pointe ainsi que des résistances de chauffe permettent une régulation de la température indépendante.

# Tribologie: sur fibre

Résultats: Force de débonding latérale

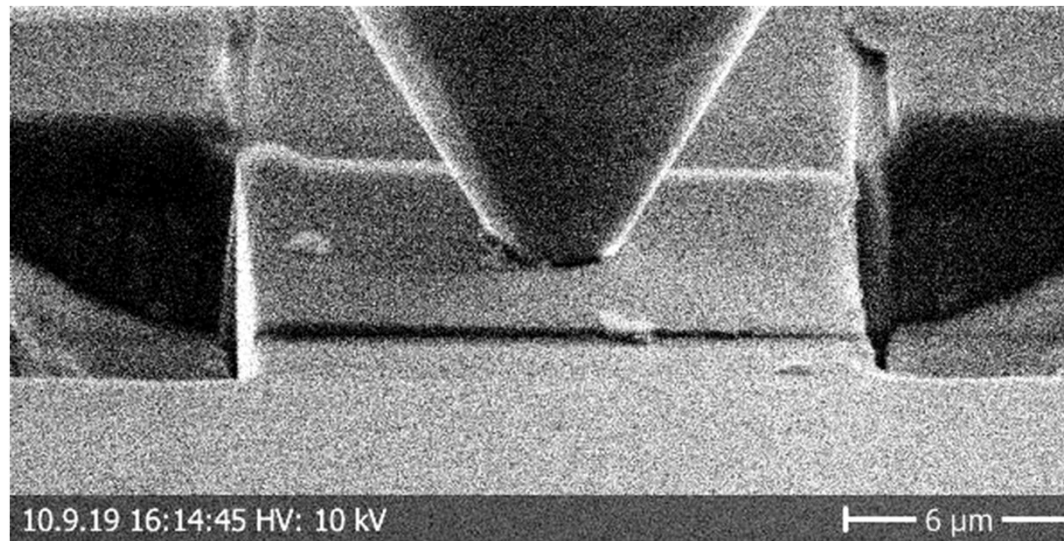
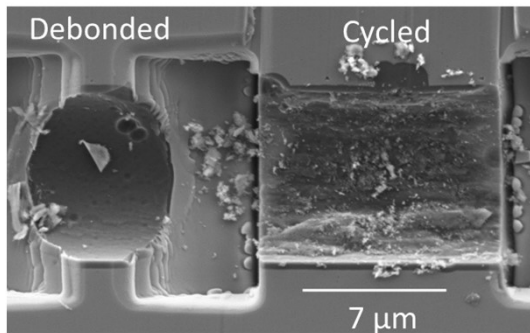
Contrainte constante de 130 MPa sur 3 échantillons



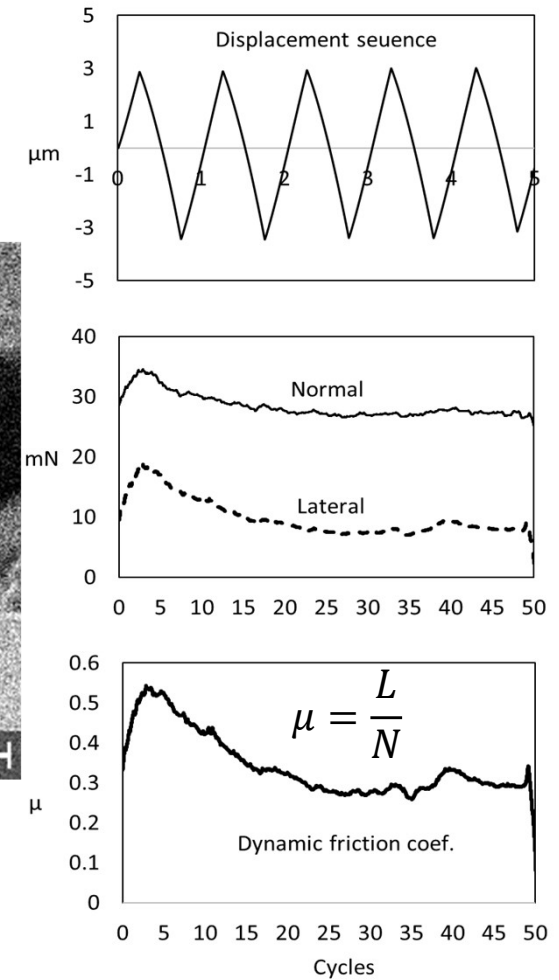
(Courtesy of J. Kabel, UC Berkeley, USA)

# Tribologie: sur fibre

Résultats: Friction cyclique latérale à la vitesse de 3  $\mu\text{m/s}$



(Courtesy of J. Kabel, UC Berkeley, USA)

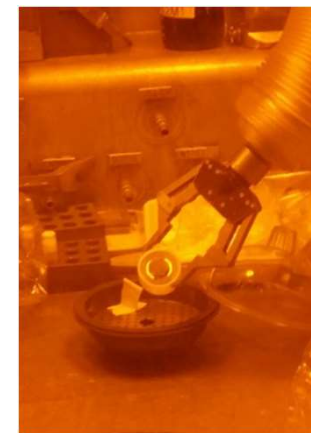


# Applications nucléaires : MEB en labo chaud

2 solutions en fonction des applications :  
Boite à gants



Chambre avec télémanipulateurs



# Tests uniques possibles avec le nano-indenteur In-Situ Alemnis

- Mode « True Displacement » (mode mixte possible avec le logiciel et le contrôleur FPGA Ultra-Rapide)
- Forces étendues jusqu'à 2.5 N, gamme de déplacement jusqu'à 100  $\mu\text{m}$
- Fort taux de déformation jusqu'à 10'000 par seconde
- Température jusqu'à 1'000°C (HTM) et à froid jusqu'à -150 °C (LTM-CRYO)
- Combinaison avec d'autres techniques In-Situ ou Ex-Situ et différents types d'environnements



*Short video showing an experimental overview of in-situ indentation  
in a scanning electron microscope (speed 5x).*

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**MERCI !**

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