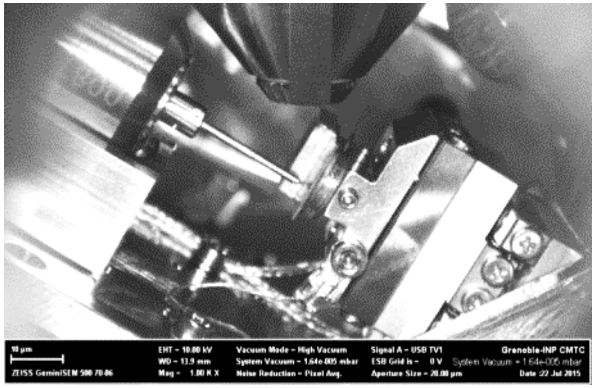
Nanoindentation in situ au MEB

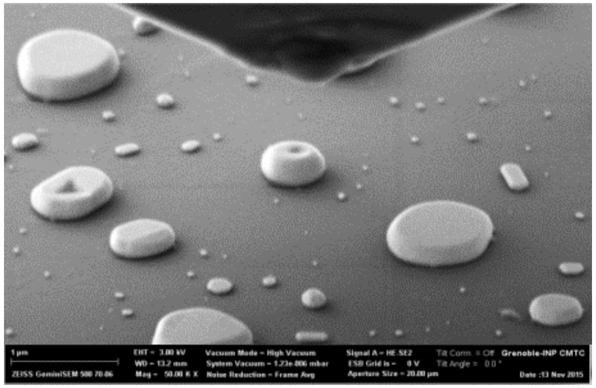
Solène Comby*[1], Fabien Volpi[1], Fréderic Charlot[2], Francine Roussel[2], Florence Robaut [2], Rachel Martin[2], Didier Pellerin [3], Marc Verdier[1]

*[1]*Univ. Grenoble Alpes, CNRS, SIMaP Lab., Grenoble, (France)*

[2]*CMTC, Grenoble Institute of Technology, Grenoble (France)*

[3]*CSI/ScienTec, les Ulis, (France)*





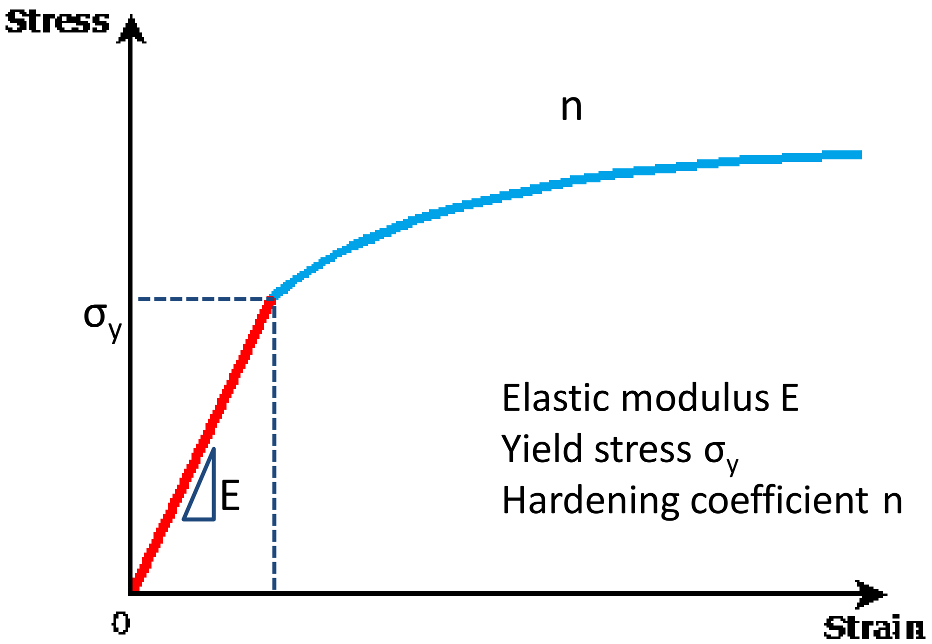
- Uniaxial tensile tests are used to obtain mechanical properties of materials.

1cm

Standard sample geometry with a constant section

1m

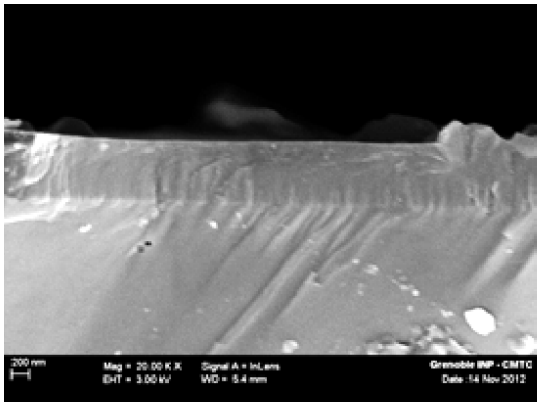
Tensile test machine



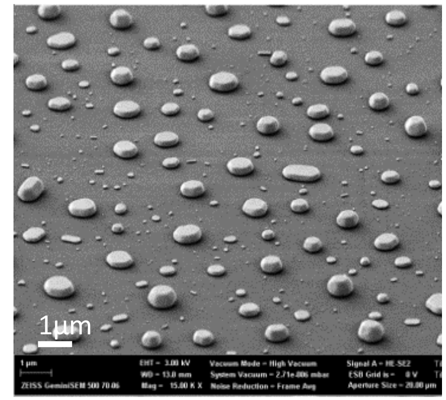
Tests quite easy, provided that tensile specimens can be prepared.

MOTIVATIONS:

❖ What happens when the sample cannot be easily designed as a tensile specimen?

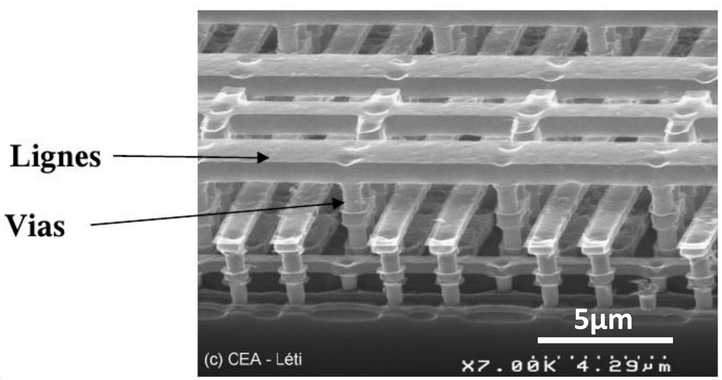


AlN thin film, F.Mercier, R. Boichot, N. Coudurier

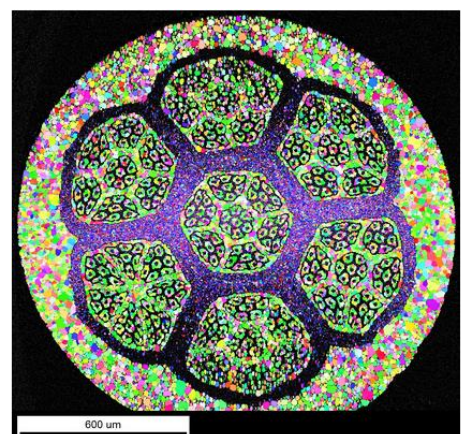


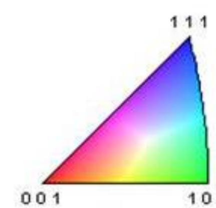
Solid phase dewetted Au island on Al_2O_3

❖ What happens when we need to measure local mechanical properties?



SEM image of a connector, LETI CEA





EBSD map of DSS alloy
Ph.D Hasan Naser

- To measure locally mechanical properties, micro-hardness tests can be used

10cm

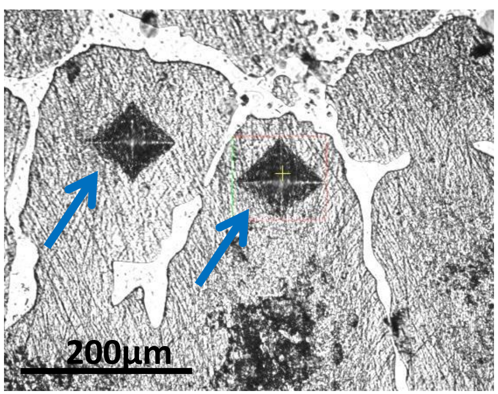
- Hardness:

$$H = \frac{F}{A}$$

F → Force applied on sample

A → Residual print size

- With mircohardness test, the imprint size >> 10µm (and the print depth >> 1µm)



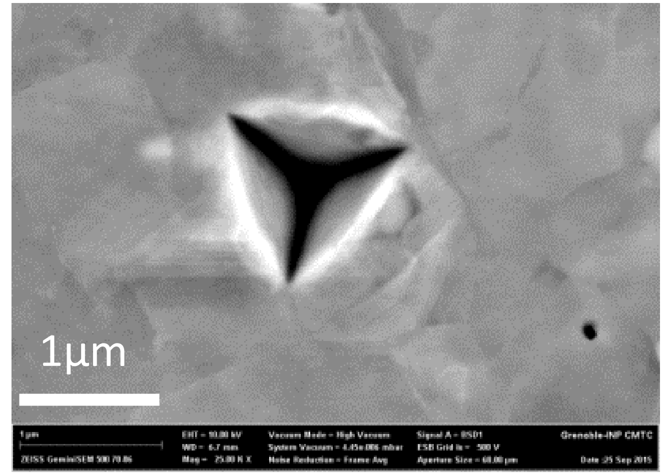
Vickers indents on AZ91 alloy, Ph.D Garrido Pacheco Mariano

PROBLEM:

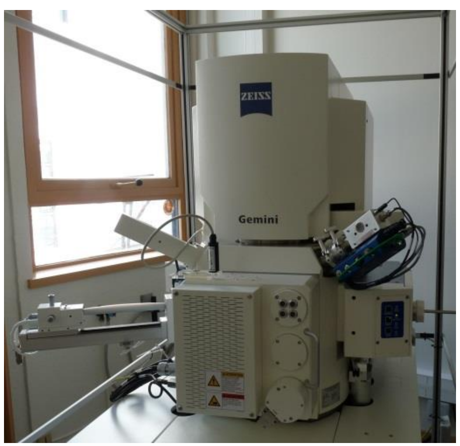
- Local properties small scale object <<1µm?
- Small scale hardness variations in systems <10µm?

SOLUTION: Nanoindentation, i. e. indentation at depths in the orders of nanometers.

- Nanoindentation, a good technique to measure locally mechanical properties of small scale object
- Depth of penetration ~ 100nm, typical print left ~1µm.



MOTIVATION FOR IN SITU SEM NANOINDENTATION

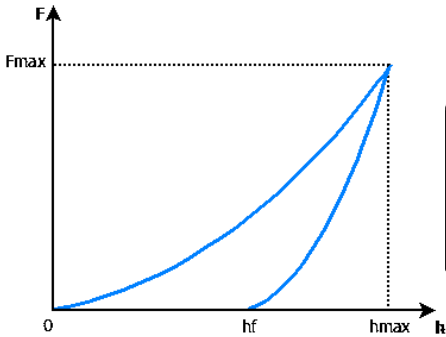


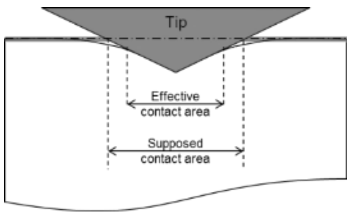
- Precise positioning of indents: investigation of small object
- Coupling indentation with chemical analysis (EDS), orientation map (EBSD),…
- Investigation of what happens during indentation

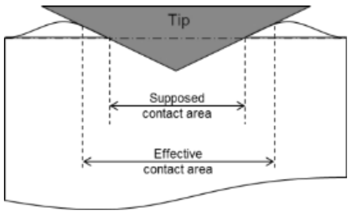
Outline

Introduction

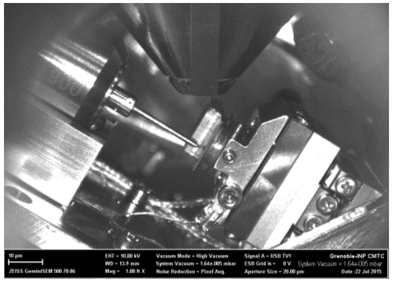
I) Basics on Nanoindentation



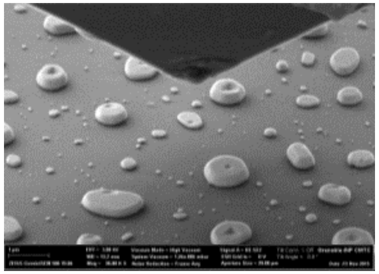


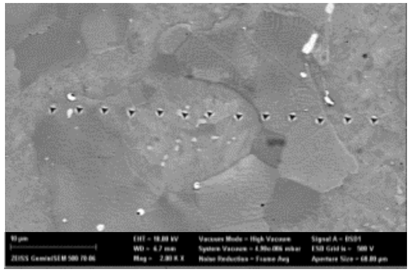


II) In Situ Setup



III) Examples of studies carried out with the setup



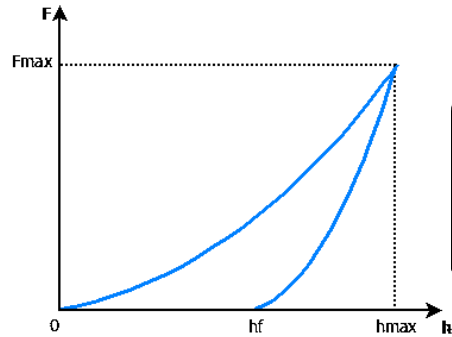


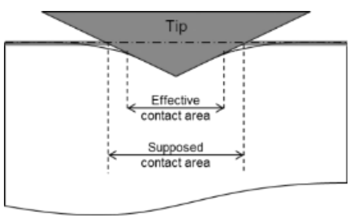
Conclusion and Perspectives

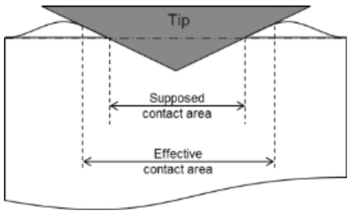
Outline

Introduction

I) Basics on Nanoindentation







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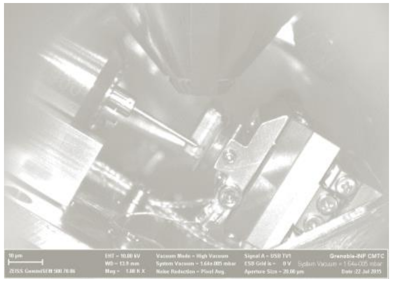

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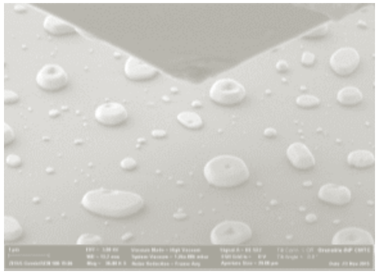
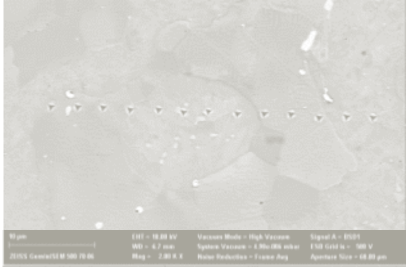




Conclusion and Perspectives

❖ What is nanoindentation?

- ❑ Nanoindentation developed around the 1980's [Pethica 1982, Loubet 1984] to investigate small volume of materials or thin films.

- ❑ Historically, nanoindentation = indentation at depths in the orders of nanometers.

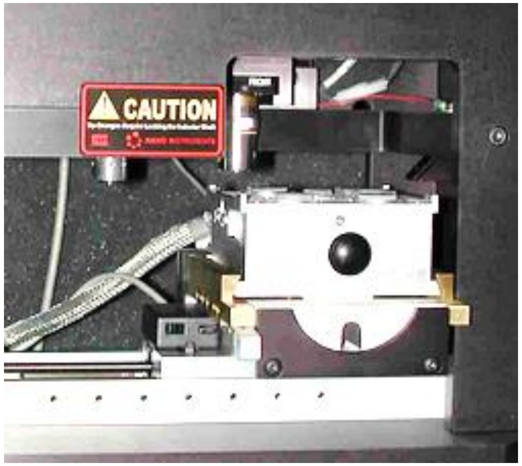
Was it so easy in the 1980's ?

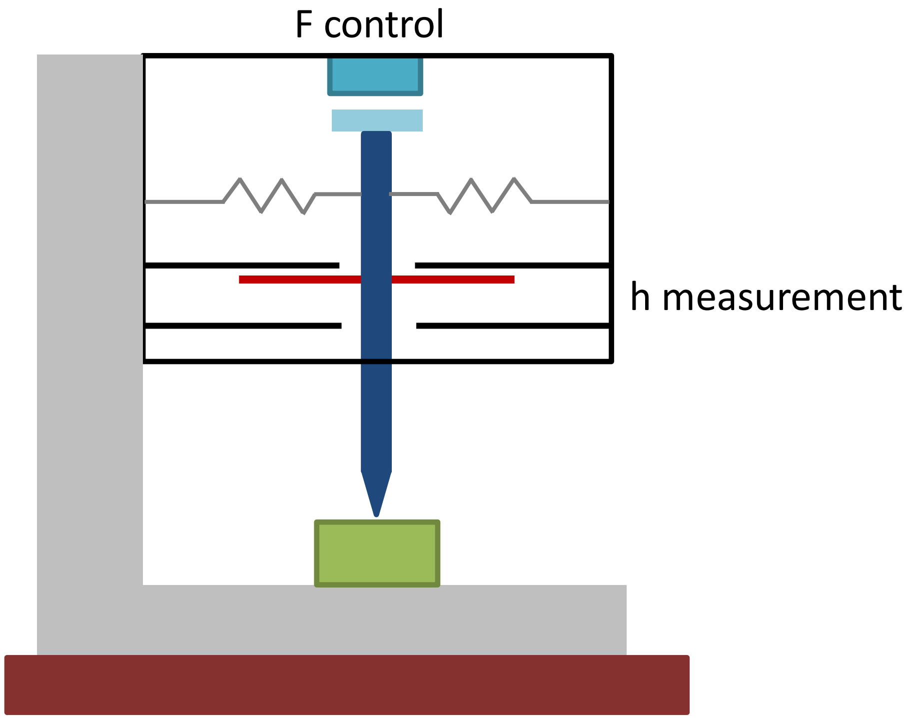
- ○ **Problem of indents imaging**! For indentation of several nm, imprint around the micrometer ➔ Hard to image indents in order to measure their size with usual optical microscope, and SEM not easily accessible!

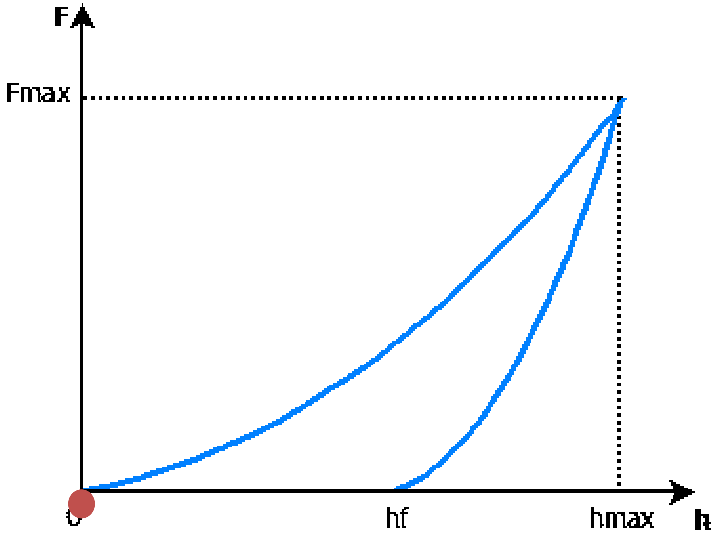
Nanoindentation developement without imaging indents

❖ <u>What is nanoindentation?</u>

➢ Load and penetration depth continuously and independently measured during the nanoindentation.







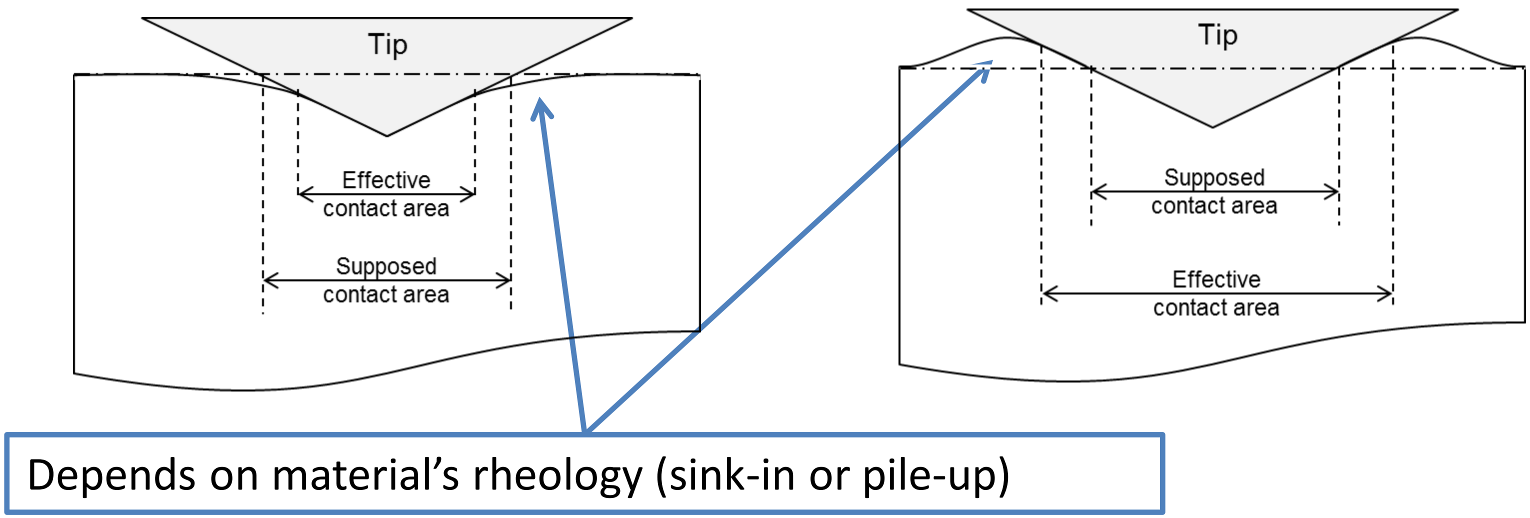
Load-displacement curves

Mechanical properties. And not only H!➔ E, n, σ_y

❖ How can we extract mechanical properties from indentation curves?

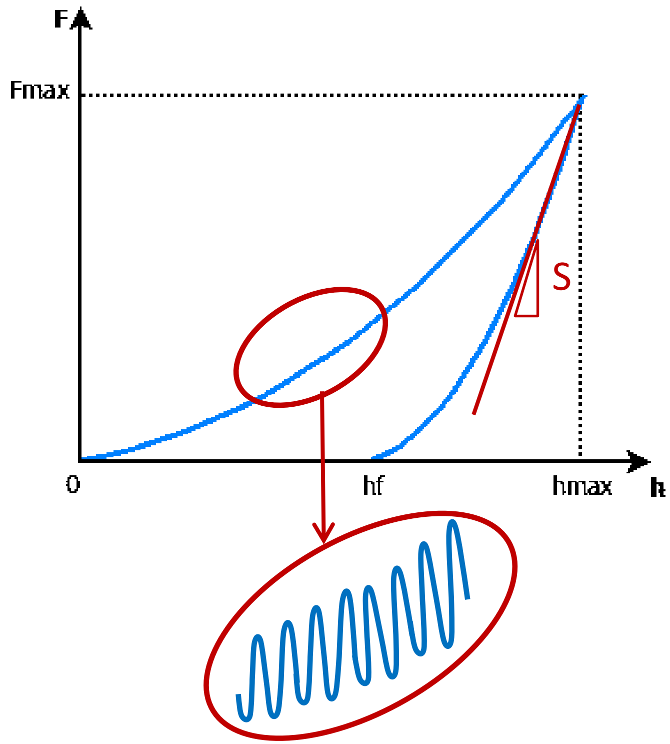
➢ Uniaxial tensile tests: transverse area where the load is applied is constant: Force-displacement curves ➔ stress-strain curves

➢ Nanoindentation: The contact area Ac is not constant during the test.



Main issue in nanoindentation: **how can we determine Ac?**

❖ How can we extract mechanical properties from indentation curves?



Continuous Stiffness Measurement (CSM) mode: Synchronous detection (micro cycles of loading-unloading)

Sneddon's equation [Sneddon 1965]

$$S = 2\,E^*\,r_c \quad \text{with } r_c = \sqrt{\frac{Ac}{\pi}}$$

Material's modulus known :

- Ac and so H is computed $H = \frac{Fmax}{Ac}$

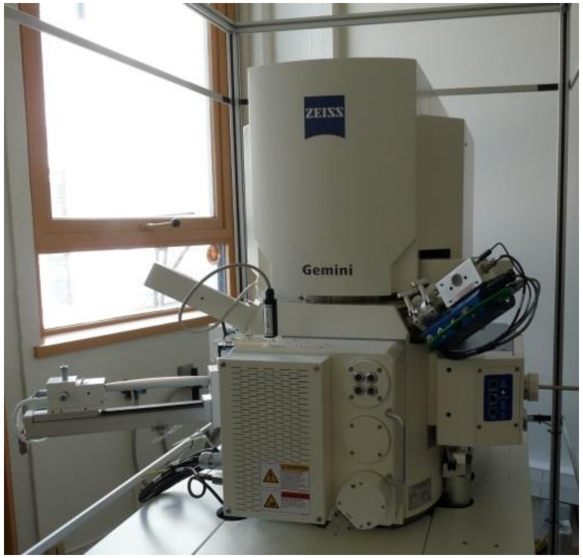
Material's modulus unknown :

- Ac determined with a reference material, assuming that it has the same rheology than the investigated material
- Both H and E can be computed

❖ To conclude:

Nanoindentation is a powerful technique which allows the complete mechanical characterization of materials.

❖ What is needed now?

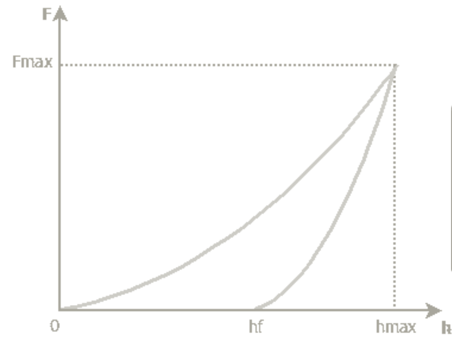


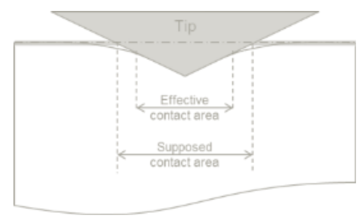
- Precise positioning of the device in order to investigate small scale systems or complex materials

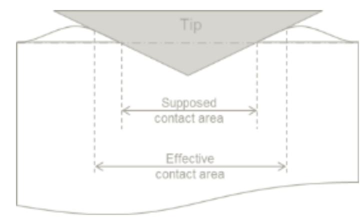
In Situ SEM nanoindentation

Outline

Introduction







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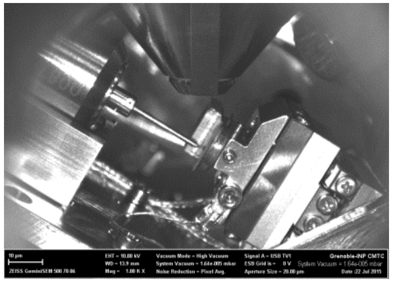

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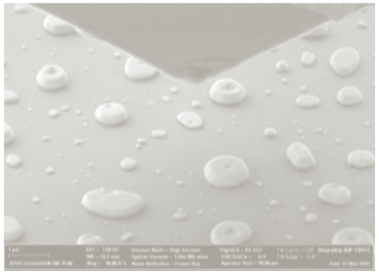
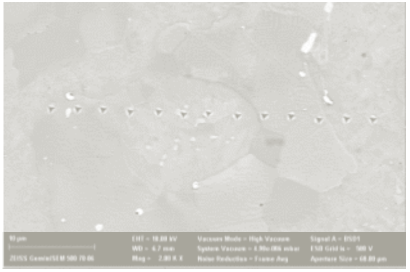
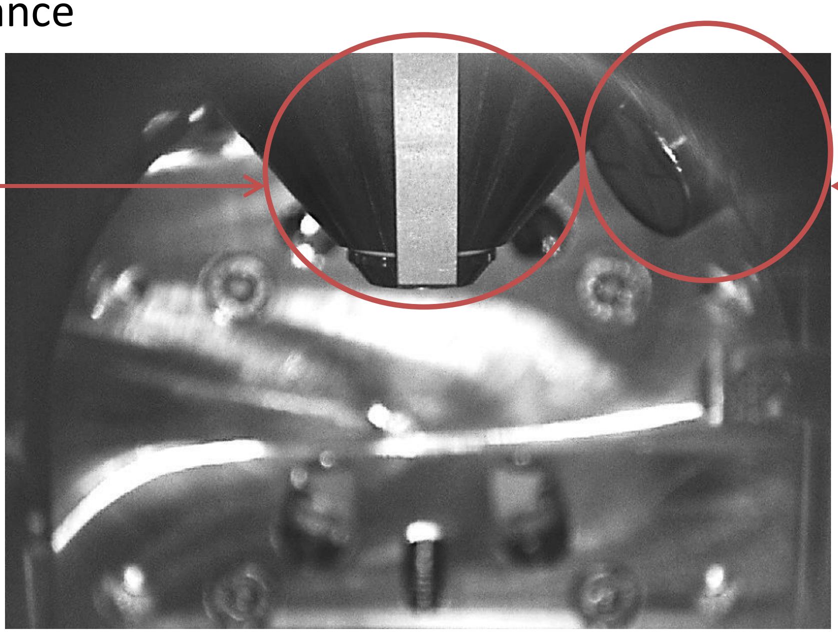




Conclusion and Perspectives

- For more flexibility, a home made setup was chosen instead of commercial ones (Hysitron, Alumni, Nanomechanics…).

- Specifications needed for the in situ setup
  - Nanoindenter's head optimized for high vacuum use
  - Enter into the SEM room, without any risk of collision, but with reasonable working distance



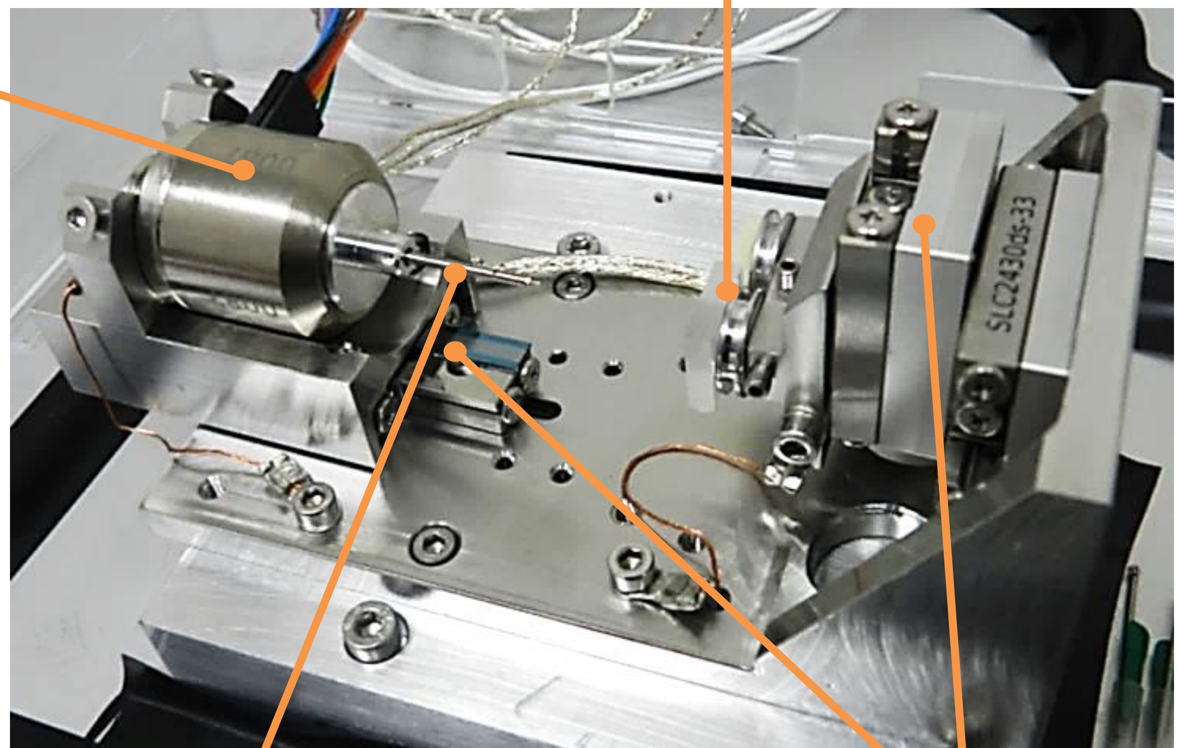
  - Possibility to image indent during indentation

❖ In Situ Setup description: The Nanoindenter

Nanoindenter's head : Inforce 50 from Nanomechanics Inc.

- Control in force with electromagnetic coils
- Displacement range = 50µm
- Displacement resolution = 0.02nm
- Maximum load = 50mN
- Load resolution = 1nN
- Mode CSM included and optimized for high vacuum conditions

sample



Extender + tip

SmarAct X-Y-Z macro piezo drivers

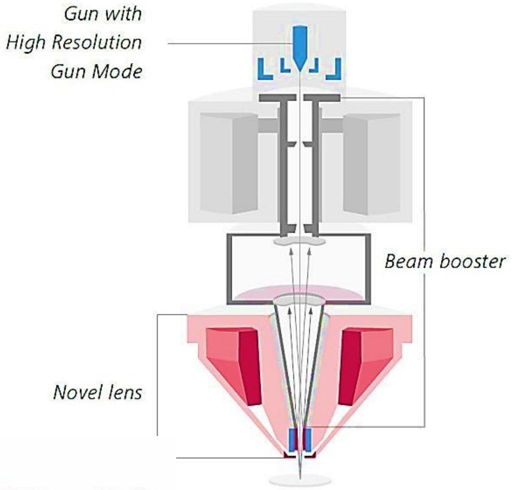
❖ In Situ setup description: The Scanning Electron Microscope. GeminiSEM 500 – ZEISS

Nouvelle génération

Principe de la colonne GEMINI

Avec des innovations pour limiter les aberrations:

- Du Canon FEG Schottky, un mode « haute résolution » pour diminuer la dispersion énergétique
- D'une nouvelle Lentille Objectif (Nano-Twin) couplant champ électrostatique et champ magnétique



Résolution en haut vide : 0.6 nm @ 15kV
1.1 nm @ 1kV

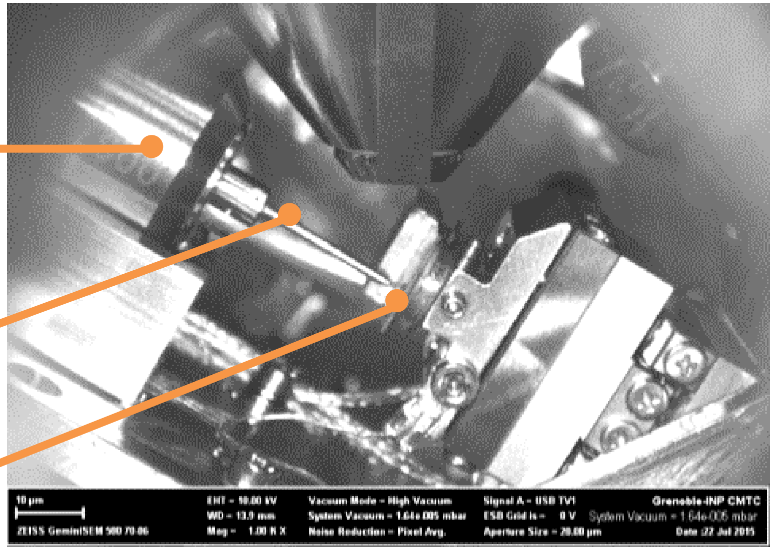
Ce microscope est à pression contrôlée sur une gamme allant de 5 Pa à 500Pa

❖ In Situ Setup: technical specifications

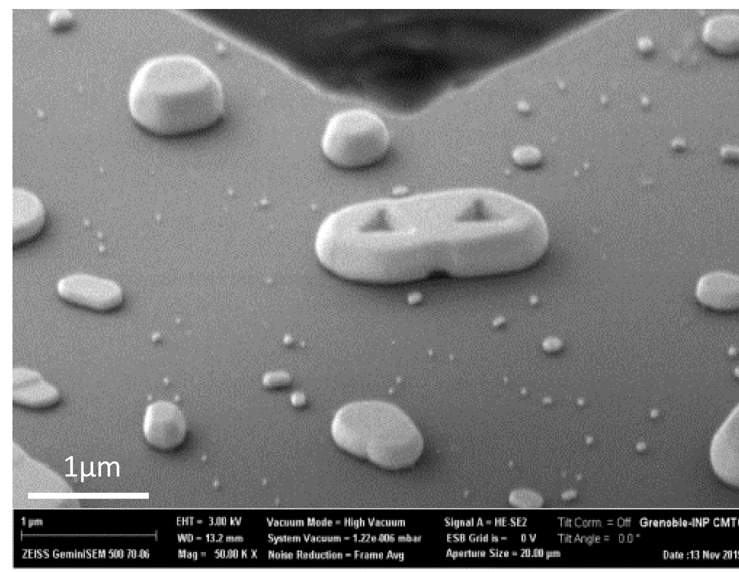
Nanoindenter's head

Tip + extender

Sample

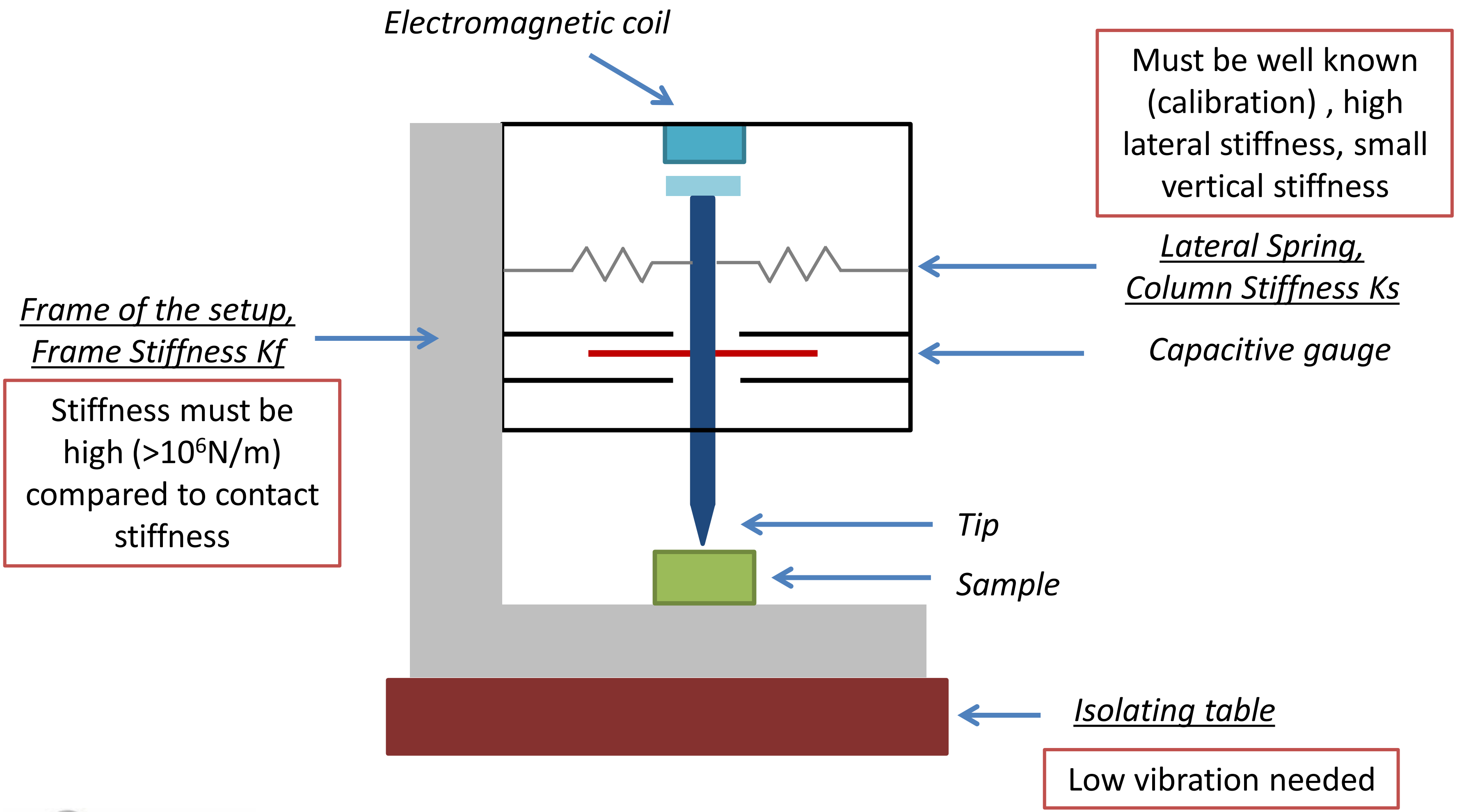


- Maximal tilt =30°
- Typical working distance used is 13-14 mm
- Observations with SE detectors (both SE2 and InLens). Set-up for BSE experiments under progress.

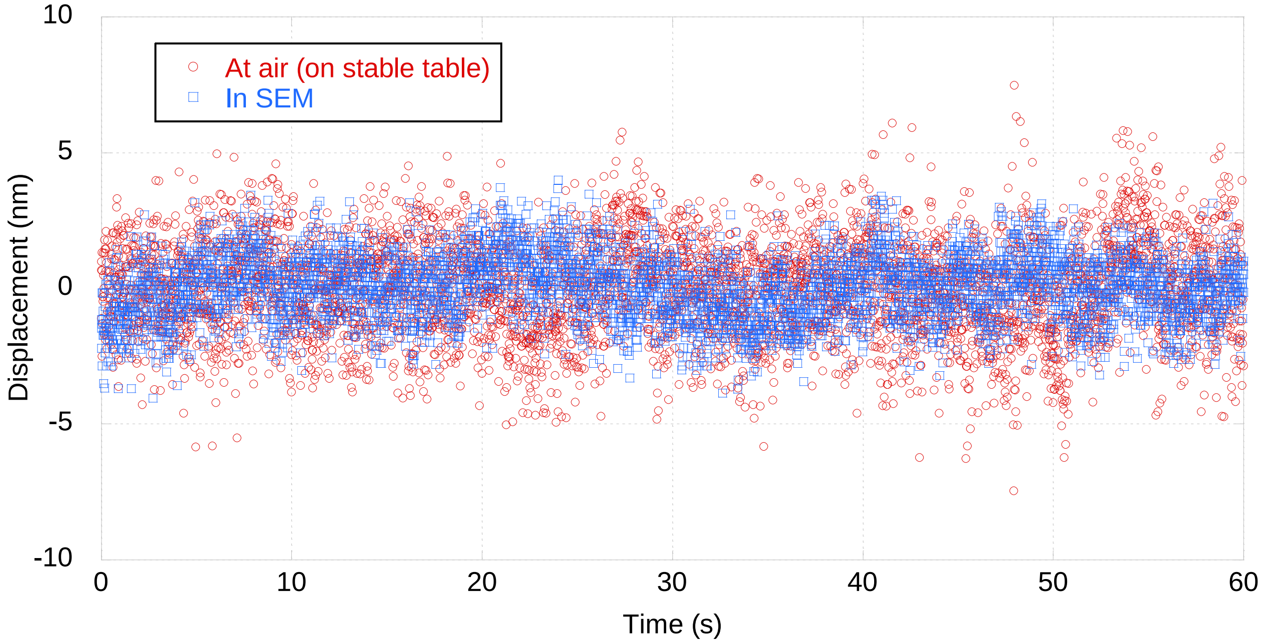


- High quality SEM pictures during indentation at magnification of X100k.
- Indents positioned with a precision of 100nm.

❖ In Situ Setup validation: what do we have to check?



❖ In Situ Setup validation : setup stability SEM



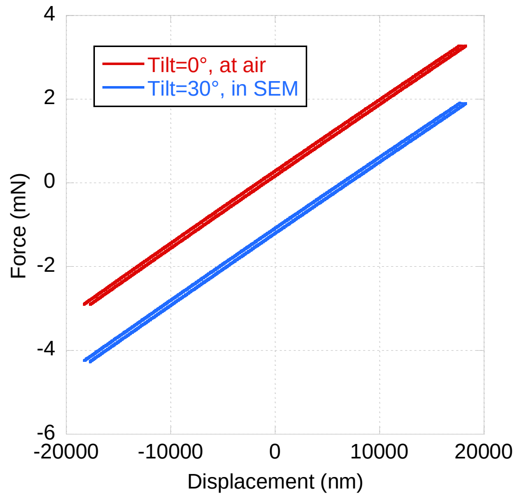
Std dev. at air = 1.8nm

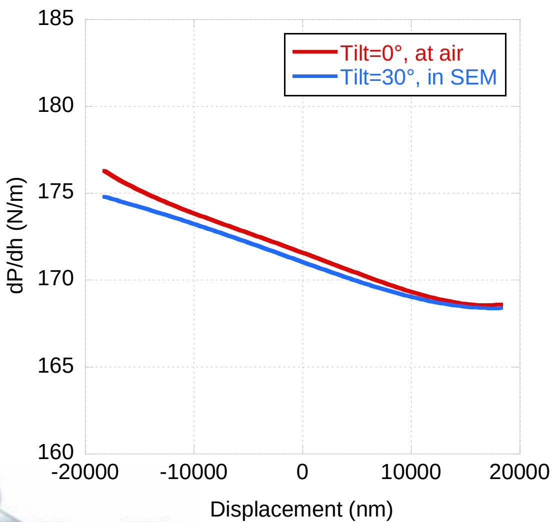
Std dev. In SEM= 1.2nm

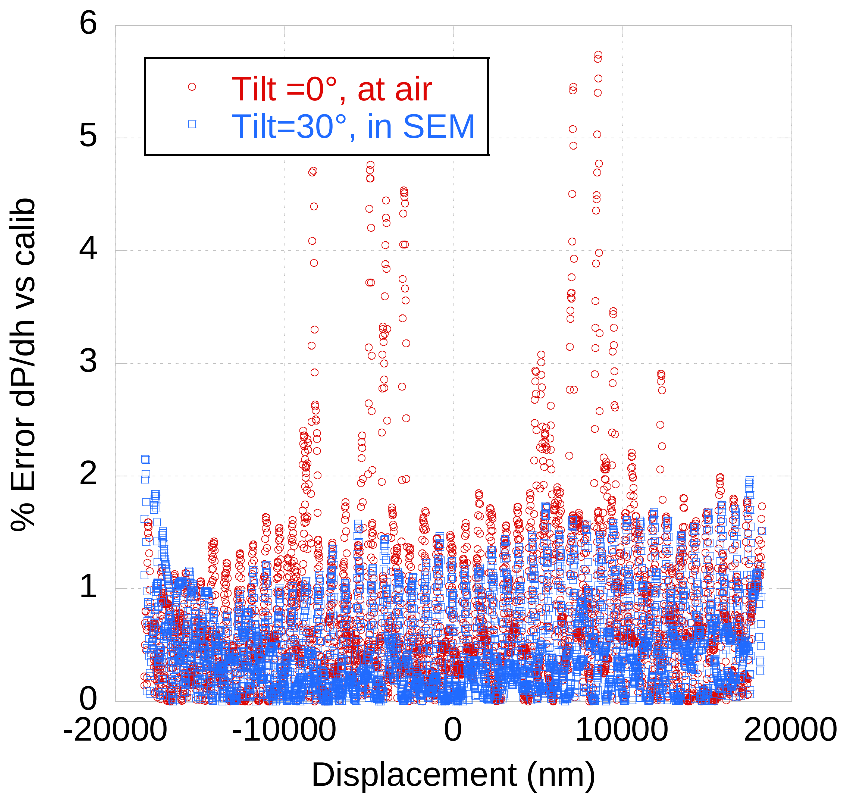
Noise acquisition at air and in SEM (displacement vs time at constant force)

- Even less noise in the in situ SEM setup
- Acquisition made with a sampling frequency = 300Hz

❖ In Situ Setup validation : column stiffness

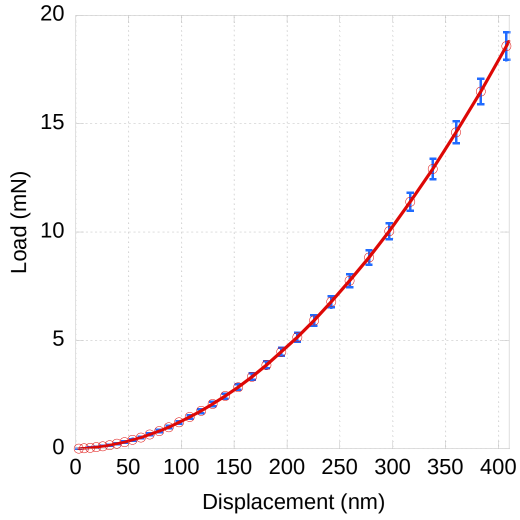


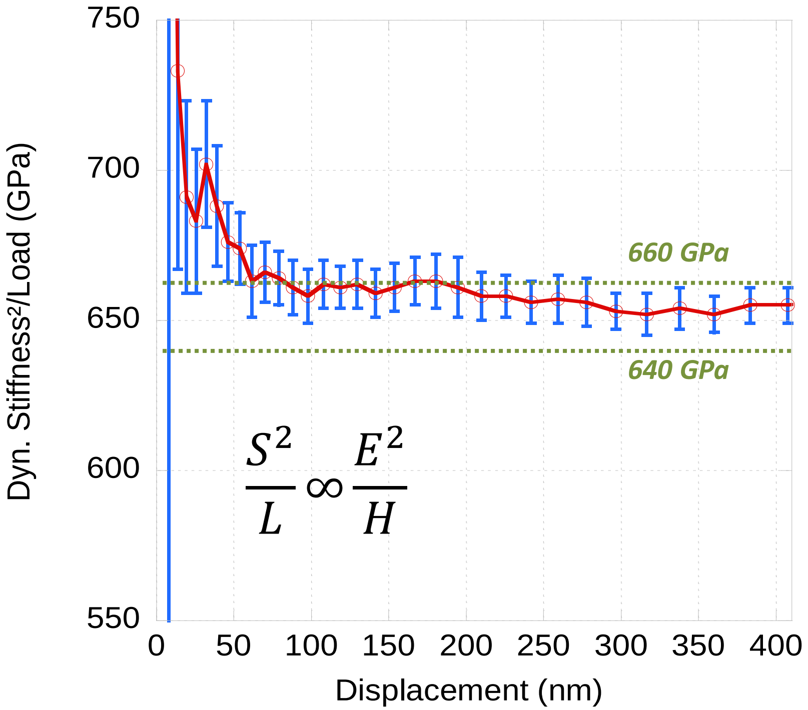




➢ Good column stiffness (The error relative to the calibration data is <2%), equal to the stiffness at air, with tilt=0°

❖ In Situ Setup validation : frame stiffness

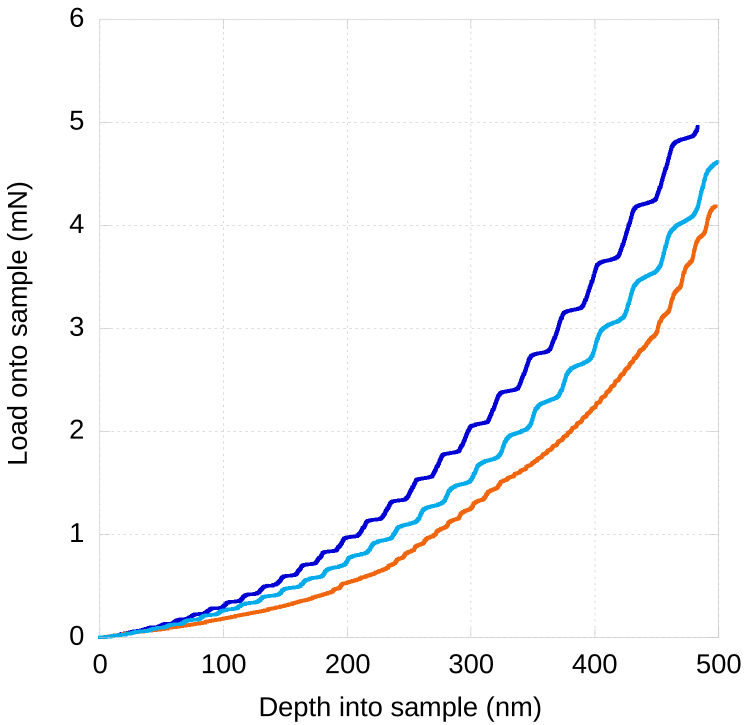




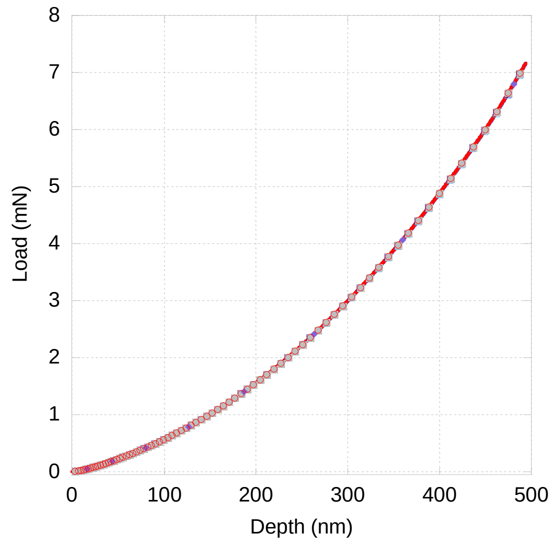
$$\frac{S^2}{L} \propto \frac{E^2}{H}$$

- Calibration on fused silica (reference material) of known elastic modulus .
- Hardness, Modulus and Dyn. Stiffness²/Load (~E²/H) stabilize at large depth.
- H=9.5+/-0.04 GPa (9.3-9.7 GPa expected for fused silica).
- Dyn. Stiffness²/Load = 655+/-2 GPa, which validates the value of the frame stiffness (order of magnitude 10^6 N/m)

❖ Problems experienced: beam scanning



In-situ SEM indentation of fused Si02, metallized with 5-7nm of carbon, Scanning beam active, EHT=10kV



Same sample, same indentation conditions, beam blanked.

- On insulating specimen, the effect of scanning beam can be seen on indentation curves (even if no charge effect in SEM image)!
- EHT value has to be carefully chosen (even on metallized samples)
  - ➔ 2-3kV for poor conductor

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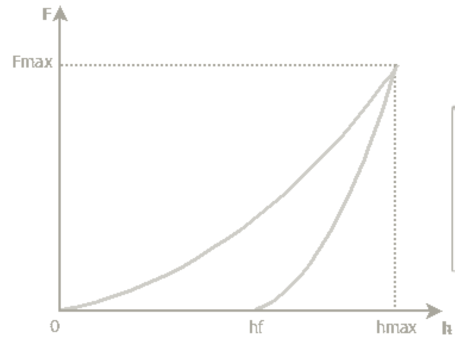
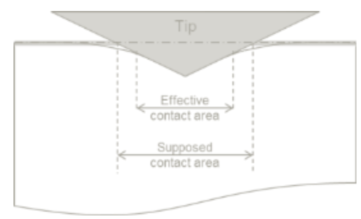
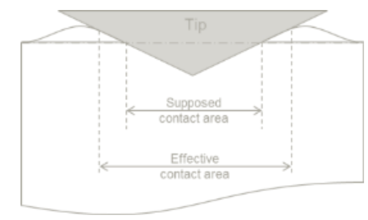
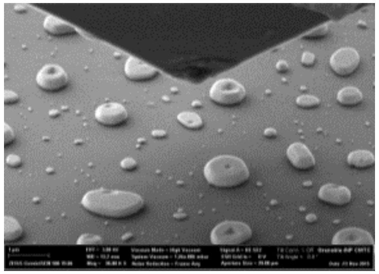


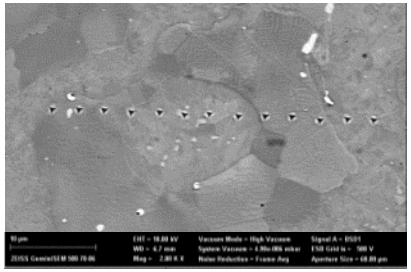




II) In Situ Setup

III) Examples of studies carried out with the setup

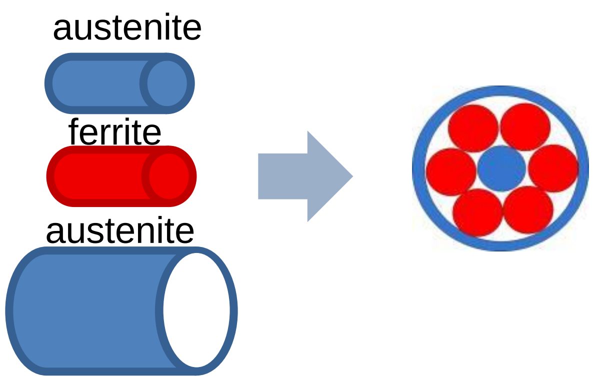


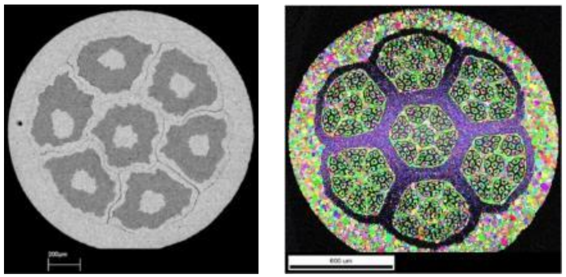


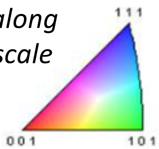
Conclusion and Perspectives

❖ Example of complex multiphased material: Co-deformed multiscale Duplex Stainless Steel

➢ Accumulative drawing and bundling of ferrite and austenite wires and tubes, keeping a 1D structure.

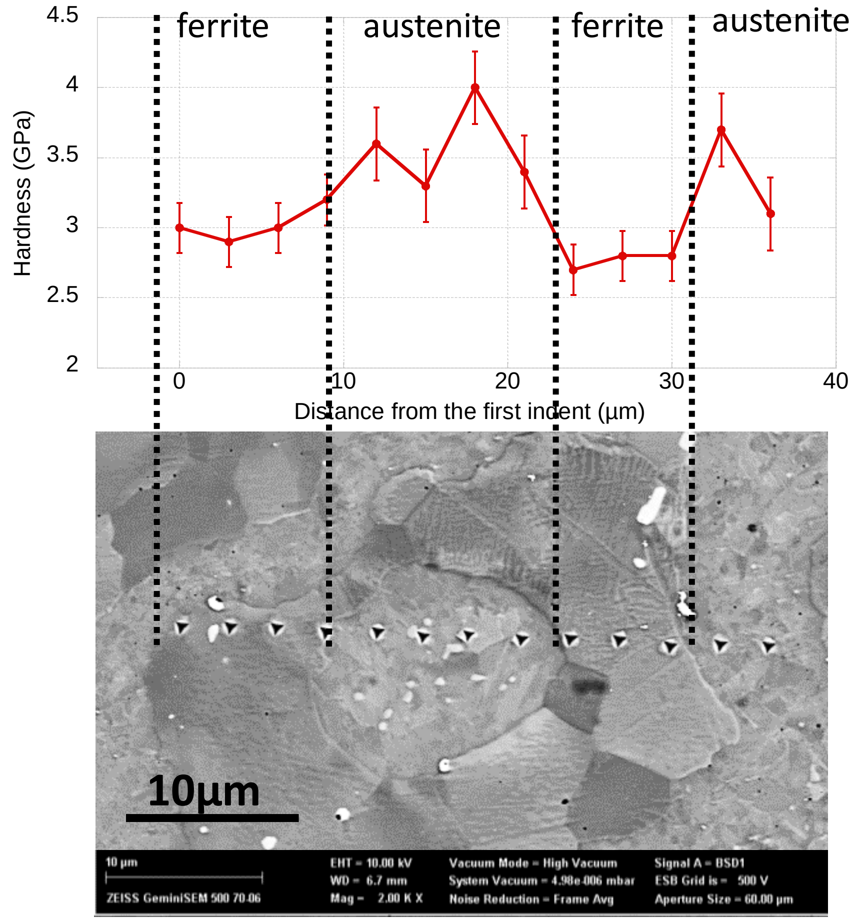




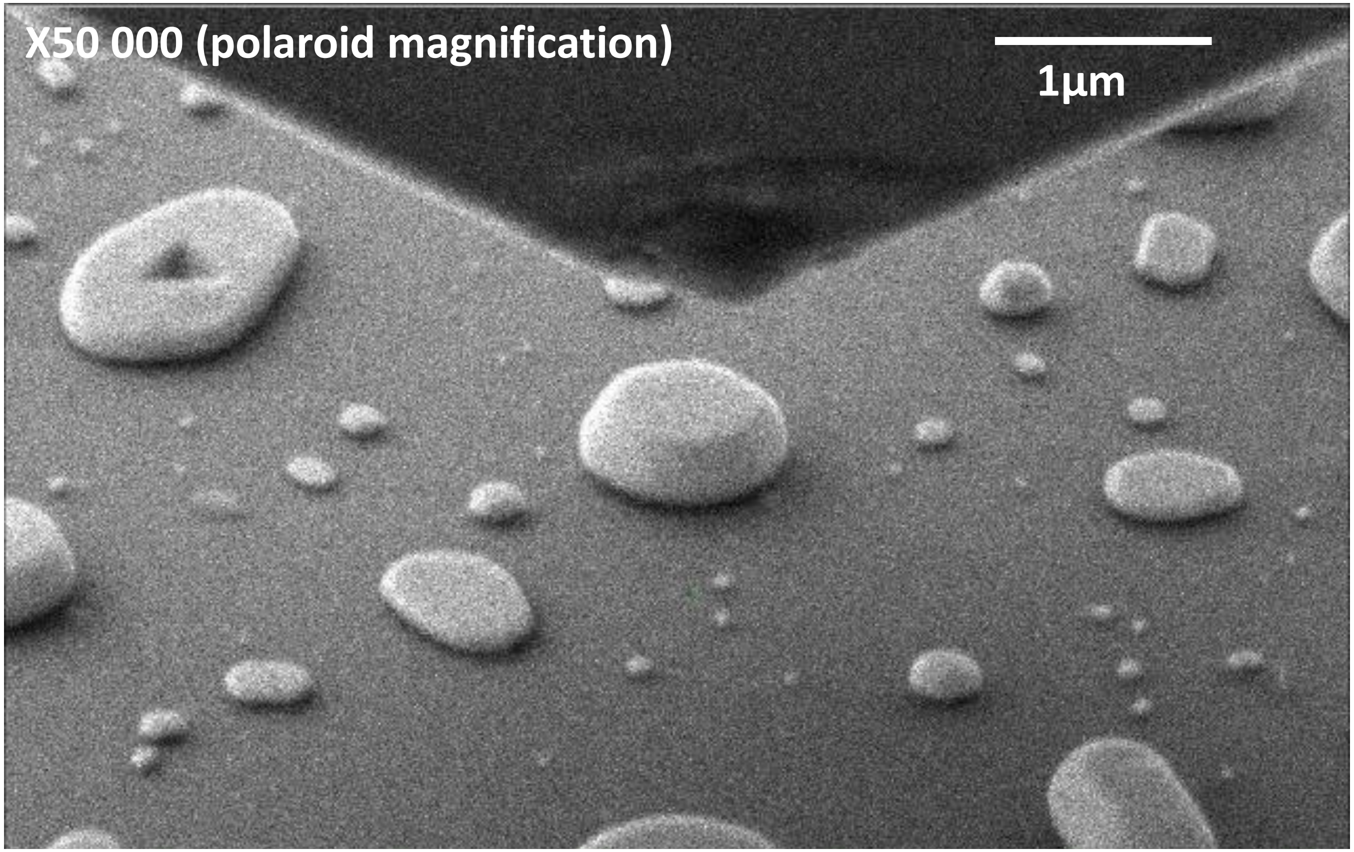


BSE pictures (left) and EBSD map along ND (right) of a co-deformed multiscale DSS alloy (from CEMAM, Ugitech).

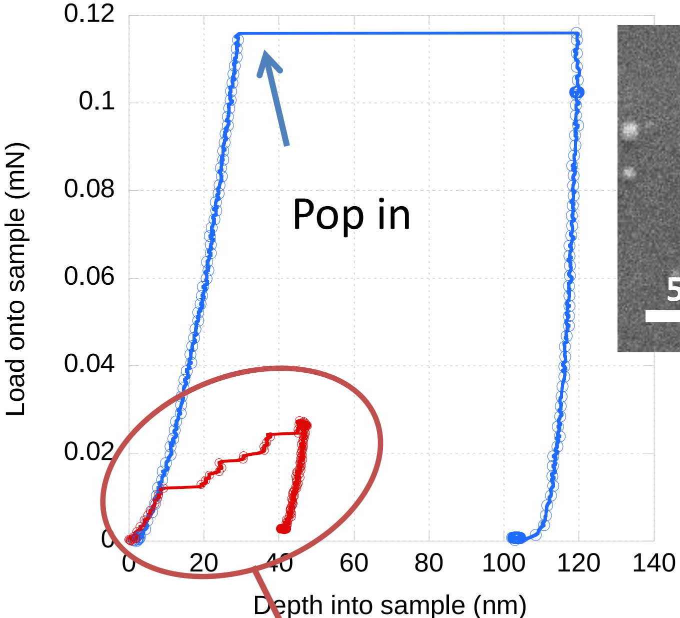
Ph.D Hasan Naser

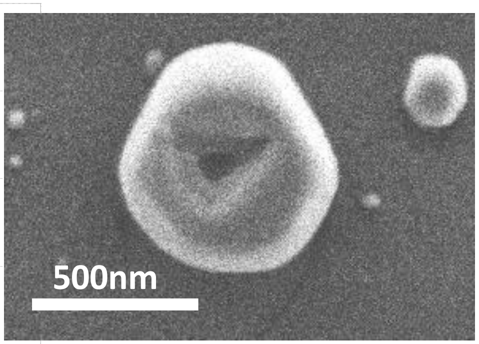


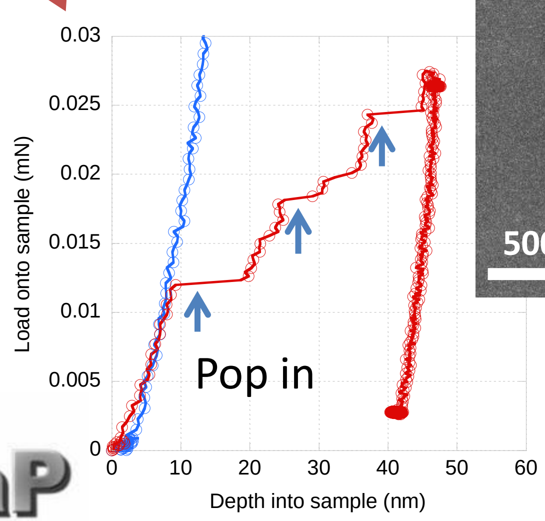
- Mechanical response of submicrometric object: Solid phase dewetted Au on Al_2O_3

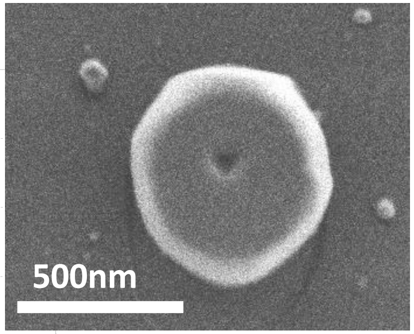


❖ Mechanical response of submicrometric object: Solid phase dewetted Au on Al_2O_3



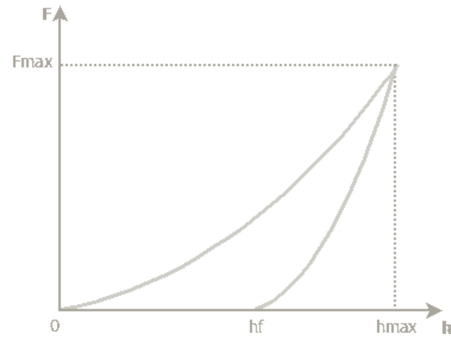


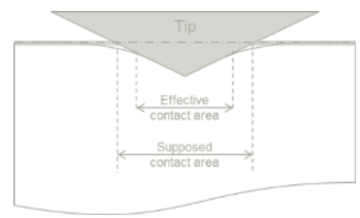


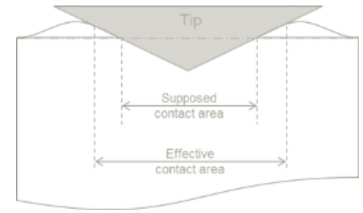


- Mechanical discontinuity (pop in): evidence of dislocations avalanche on Au islands
- Two different behaviors: large pop-in / multiple « small » pop in.
- Origin: shape ratio? Initial defects density?

Outline





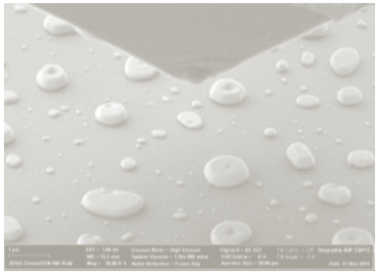


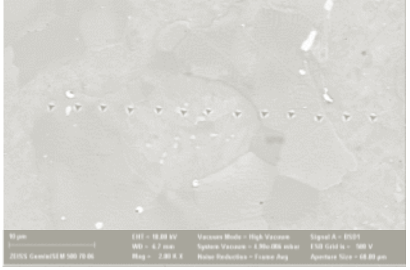
Introduction

I) Basics on Nanoindentation

II) In Situ Setup

III) Examples of studies carried out with the setup





Conclusion and Perspectives

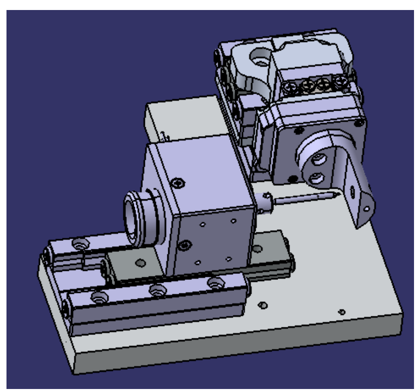
In situ SEM nanoindentation has been successfully carried out:

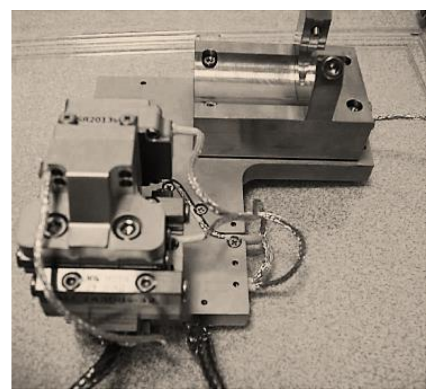
- Noise level small enough to perform nanoindentation in good stability conditions.
- Frame compliance high enough compared with contact stiffness values.
- Column stiffness not affected by the use in situ, with a 30° tilt.
- In situ device allows the positioning of indents with a precision better than 100nm

What's the next step?

- Use analytical facilities a SEM can offer:
  - EDS, to improve the identification of area to indent by identifying the different phases
  - EBSD, to investigate the change in lattice orientations during nanoindentation.

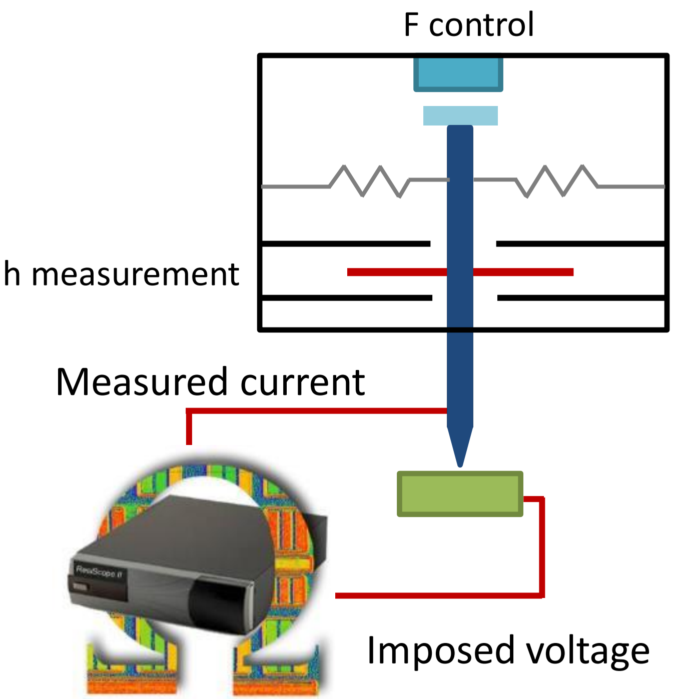
→ A new devoted system with extra degrees of freedom (one other translation and one rotation).





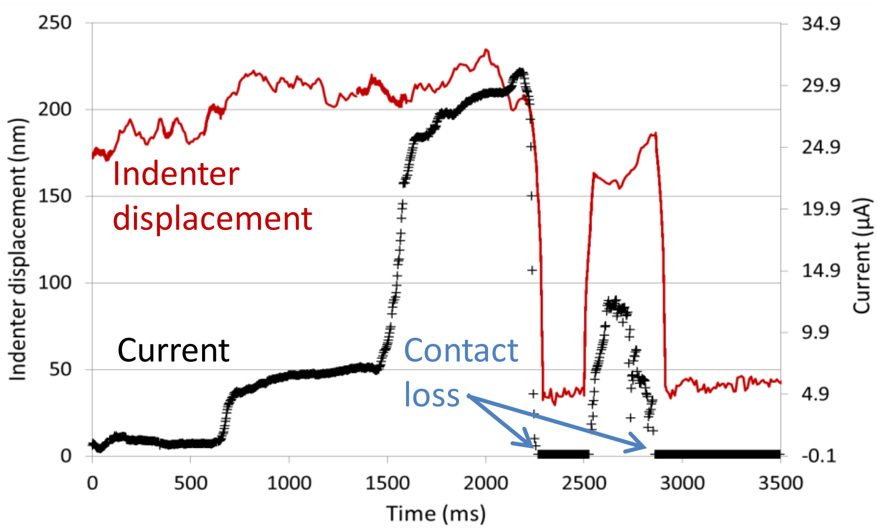
What's the next step?

- Fine electrical characterization during indentation.



- Investigation of change in electrical behavior of material during indentation

- Relation between electrical contact area and mechanical contact area



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THANKS FOR YOUR ATTENTION!