Mission pour les
Initiatives Transverses
et Interdisciplinaires

Ultra-thin Diamond Detectors for On-line Monitoring of Ion Microbeams

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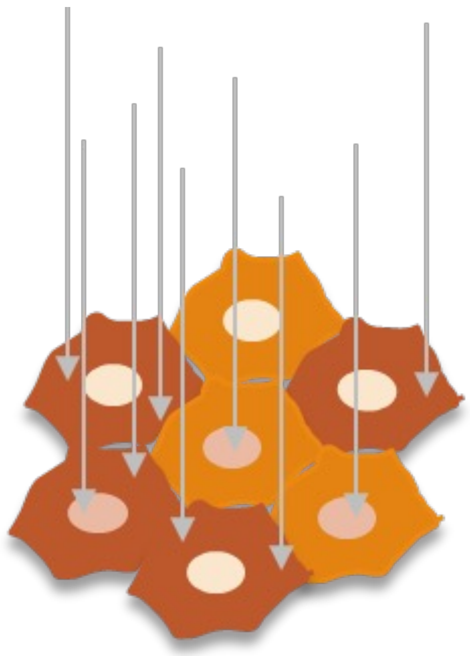
TABLE OF CONTENTS

01

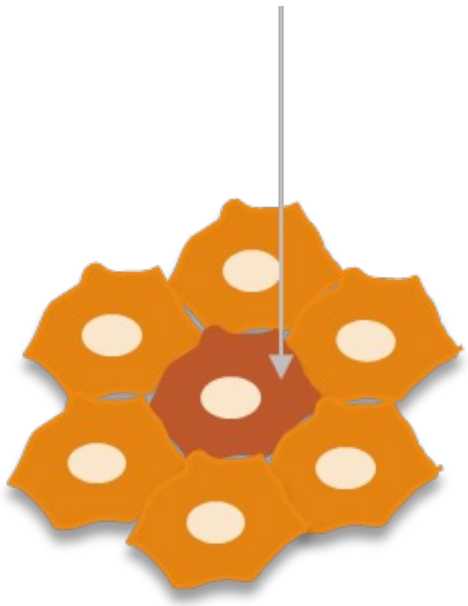
CONTEXTE:
Ion microbeam and monitoring

Microbeam's interest in radiobiology

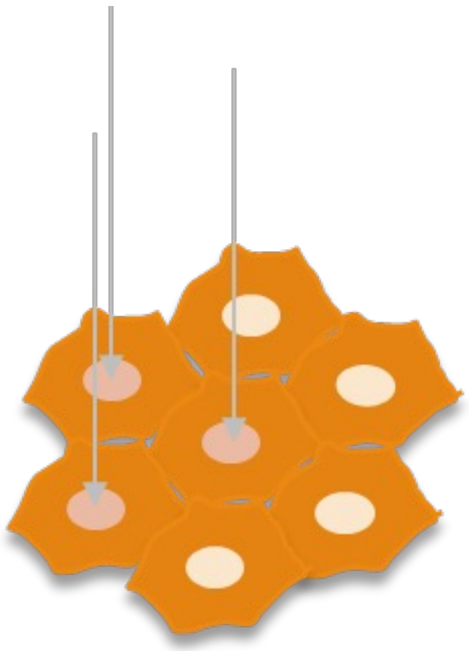
<u>Global irradiation</u>
Each cell receives a mean number of particle



<u>Irradiation by **microbeam**</u>
Targeted irradiation
-> select the irradiated cells
-> select the part of each cell



Irradiated cytoplasm



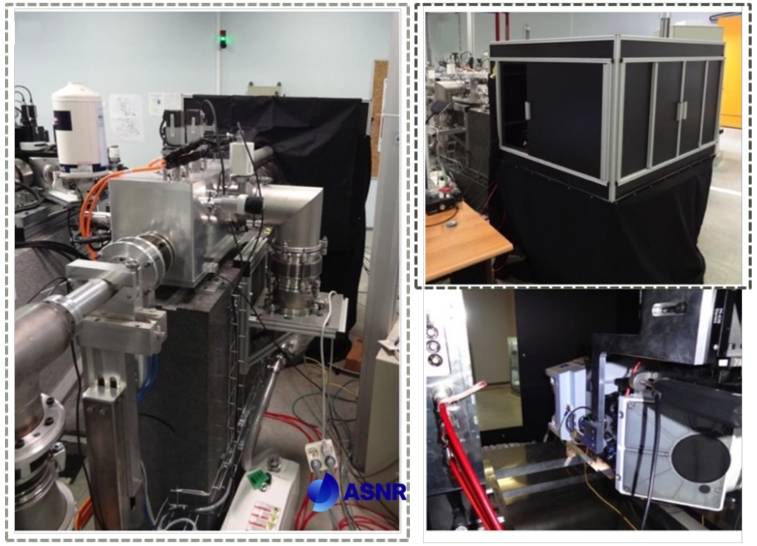
Irradiated nucleus

Two microbeam line with few differences

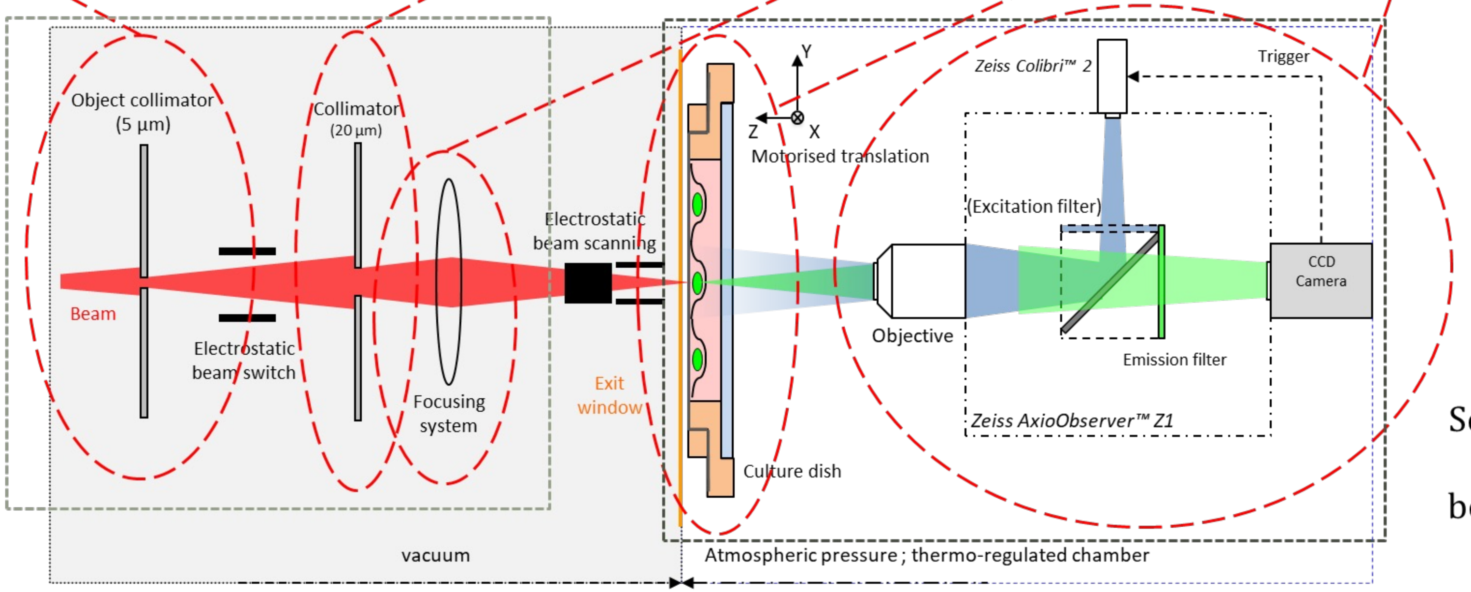
AIFIRA	MIRCOM
Gaseous ion sources	Solid and gaseous ion sources
Singeltron 3,5 MV accelerator	Tandetron™ 2 MV accelerator
3 MeV Proton 3 MeV Alpha	4 MeV Proton 6 MeV Alpha 8 MeV C^{3+}, O^{3+}

Presentation of microbeam lines

ASNR MIRCOM microbeam line

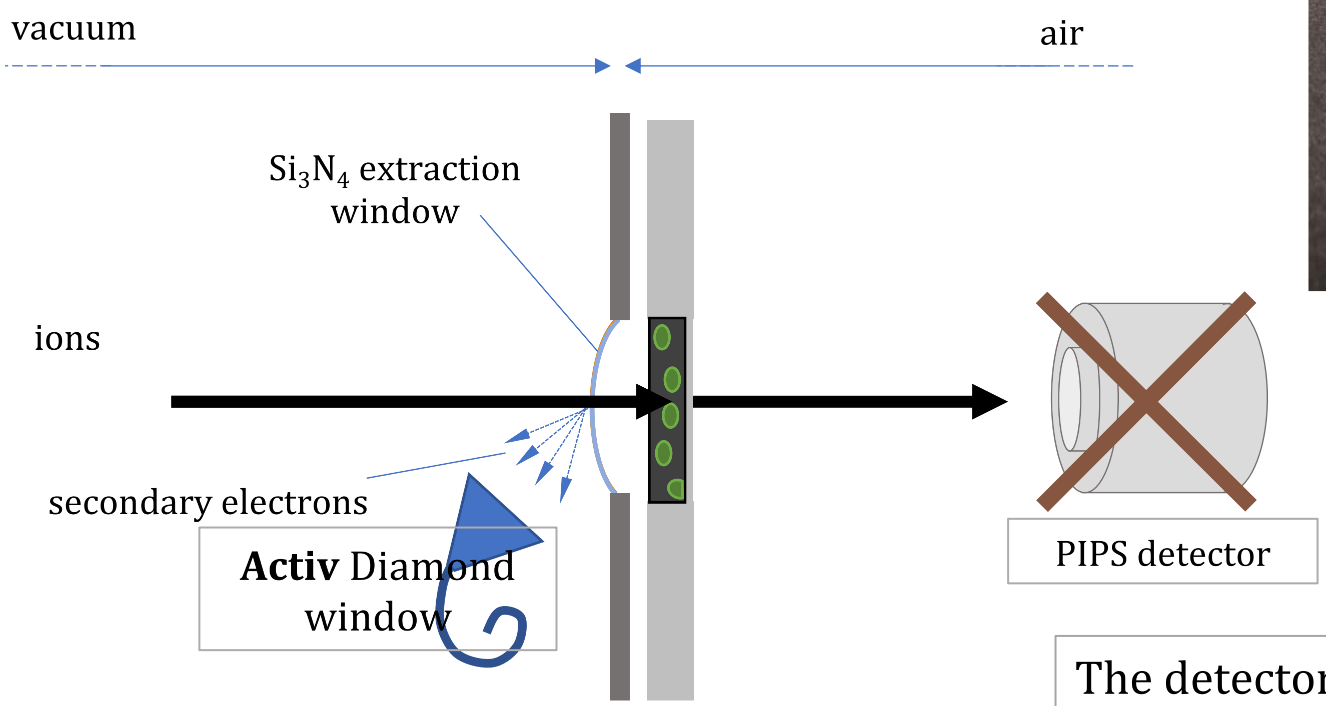


LP2i Bordeaux AIFIRA microbeam line



Schematic of both beamlines

Microbeam monitoring



Sample holder - Produced by the Detector, Instrumentation and Electronics Departments at LPSC

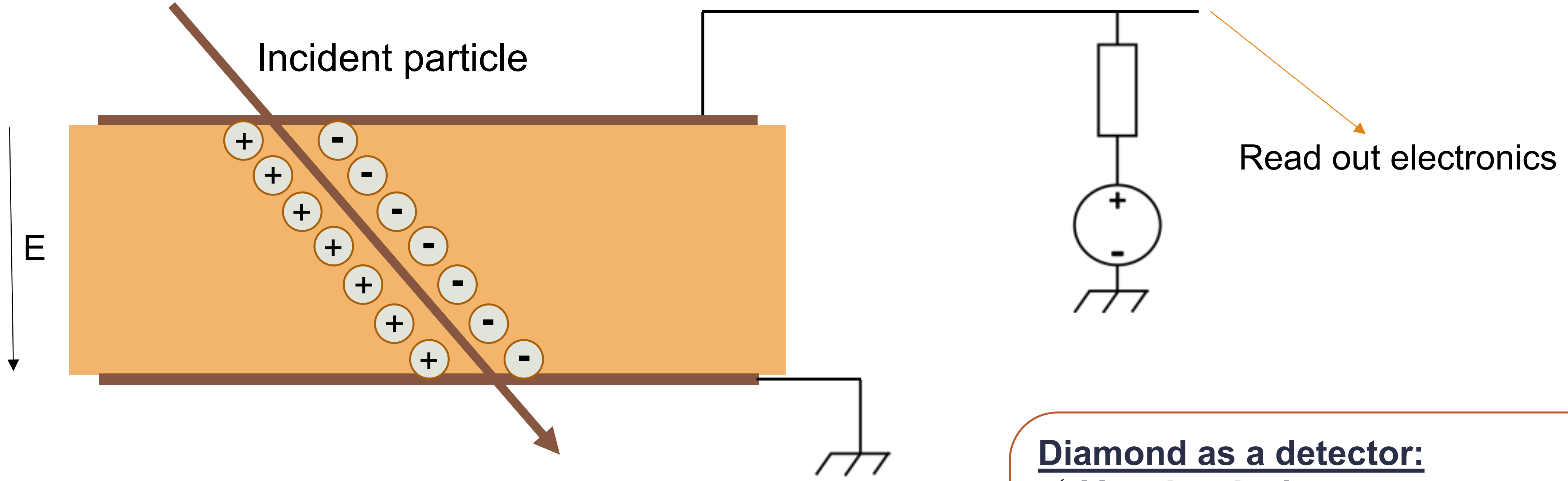
Chaneltron detector

- ✓ Efficient detection of carbon and oxygen ions
- ✓ Efficient detection of alpha particle but need to adapt the extraction window
- ✗ Not efficient for protons

(Lalanne, 2023, PhD thesis)

The detector must be able to:
- efficiently detect protons as well as heavier ions
- count ions at very low (single ion) and high beam intensities (10^4 particle.s^{-1})

The diamond as a solid-state ionization chamber



Diamond = Wide band-gap semiconductor

Diamond as a detector:

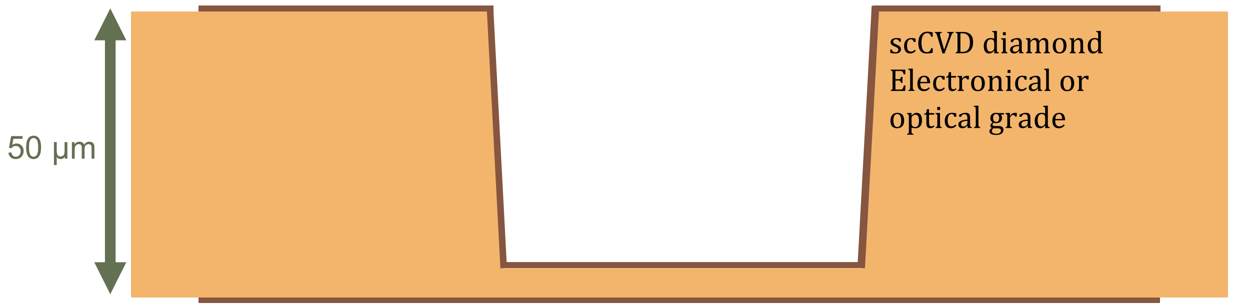
- ✓ **Very low leakage current**
- ✓ **Low noise**
- ✓ **Good radiation hardness**
- ✓ **Very fast**
- ✓ **Work at room temperature**

Ultra-thin diamonds and their problematics

Why ultra-thin?

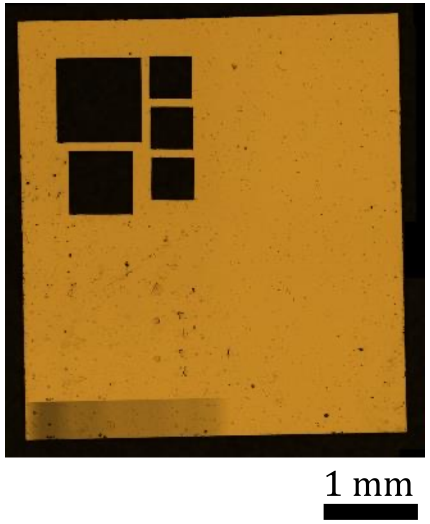
✓ **To have a minimum quantity of material before the biological environment,**

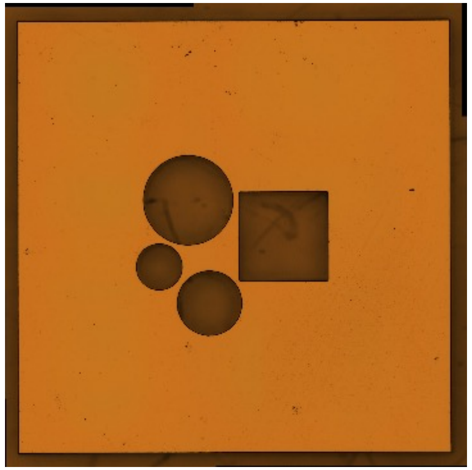
✓ **And to limit disturbances: loss of energy and spatial deviation.**



Window Ø 500 µm, few micrometers thin
Metallized electrodes for diamond biased and signal read-out

- How to obtain this kind of thinness ?
  - **Etching of diamond**
  - **Polycrystalline diamond mask**
  - <u>Objectives</u> **:**
    - good thickness homogeneity
    - sufficiently low surface roughness to limit leakage currents





Polycrystalline diamond masks with laser cut square and round patterns

02

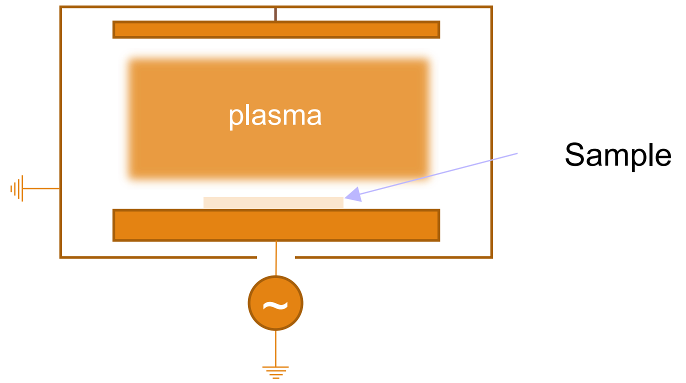
DIAMOND ETCHING

Plasma etching of diamond

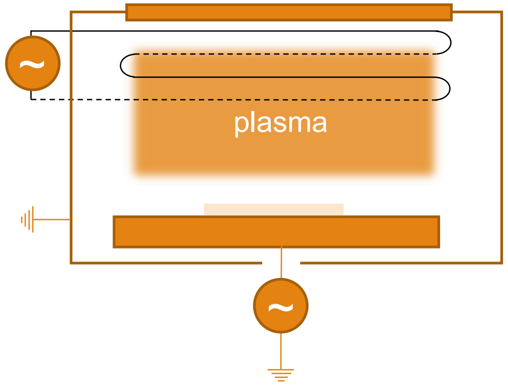
- **Reactive Ion Etching** : use of a mix of chemical reactant and chemically inert species ; physical and chemical process

- Can be of two types :

- **CCP** = Capacity Coupled Plasma : only one RF parameter
  Need a highly chemical specy (like O_2) for good kinetic.



- **ICP** = Inductively Coupled Plasma : two RF powers permitting to separate plasma **density** and its **acceleration**
  More suitable for deep etching because plasma can be denser.



Important characteristics to evaluate an etching

❖ **Etching rate** *(nm/min)* *measured with profilometers*

❖ Final **rugosity** of sample *(nm RMS)* *measured with profilometers*

→ to be compared with initial rugosity

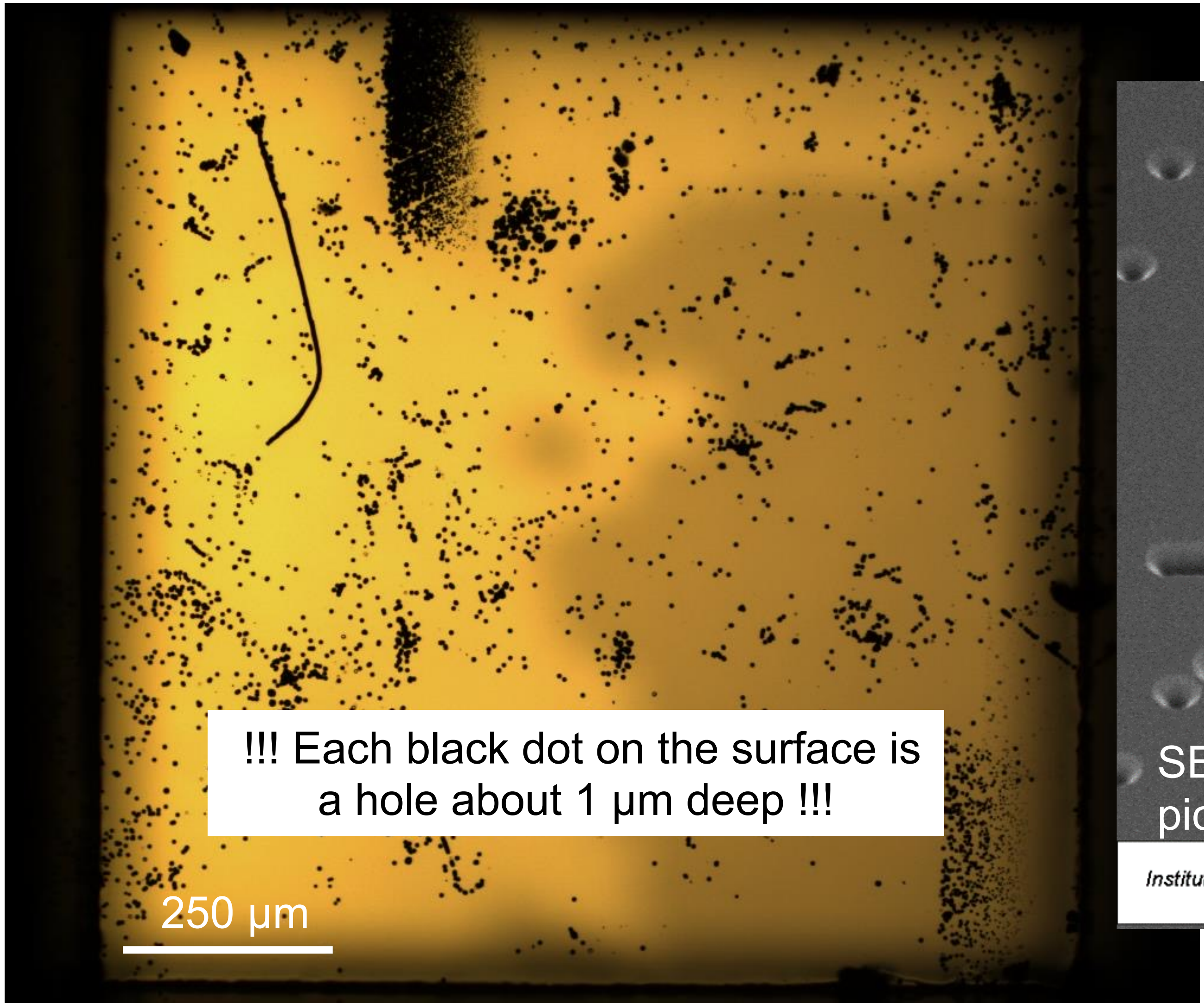
❖ Thickness **homogeneity** *(%)* *depends on initial planarity of sample*

❖ Etching defects : **etch pit** / **microtrenching**

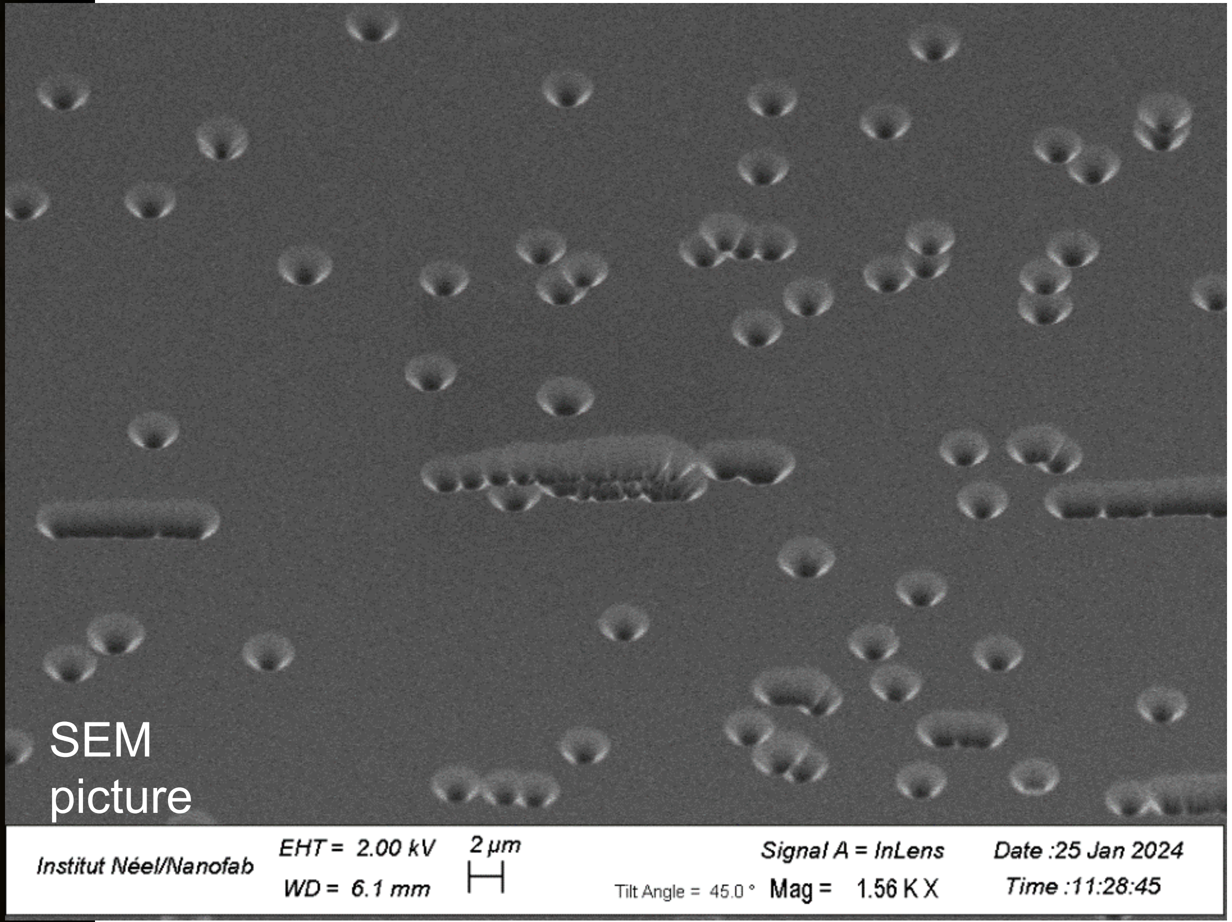
- Etch pits : preferential etching of dislocation by oxygen resulting in pits
- Microtrenching : overetching on the side of the pattern, due to the important thickness of both mask and deep etching

Etching defect : *etch pits*

RIE-CCP : 280 W Platen
CF_4 7 sccm + O_2 40 sccm
77 mTorr
19,2 µm etched (80 nm/min)



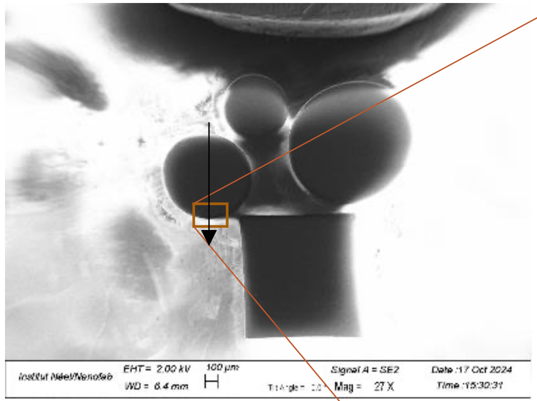
!!! Each black dot on the surface is a hole about 1 µm deep !!!

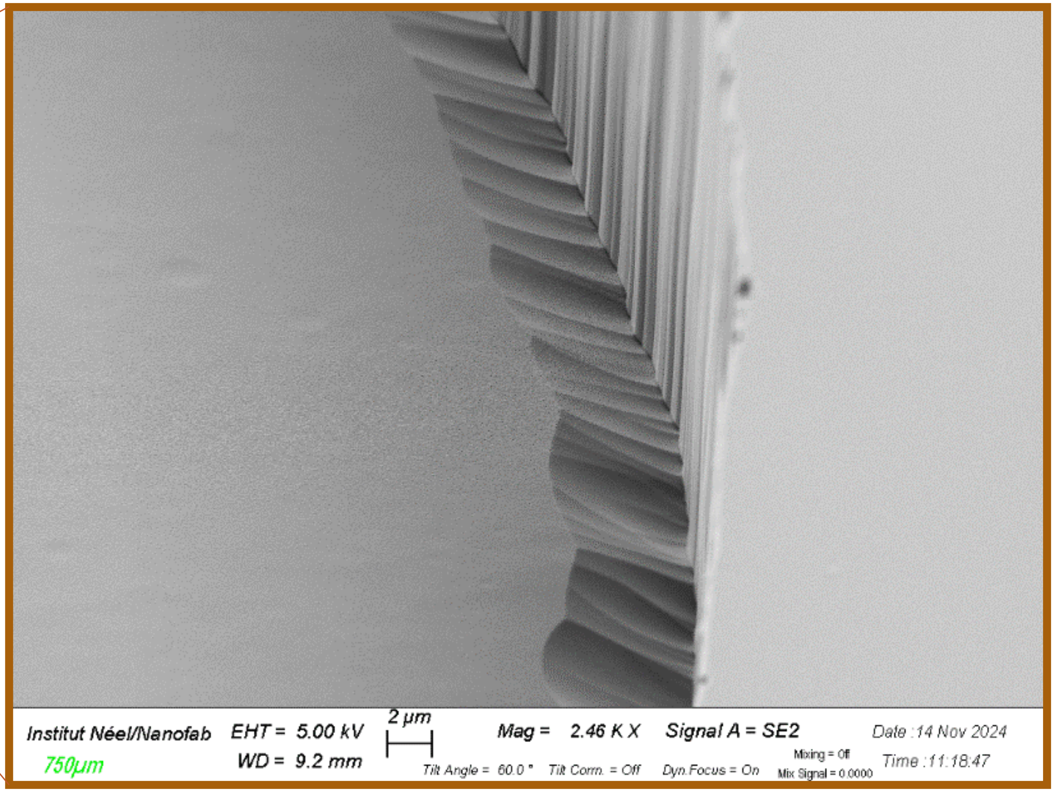


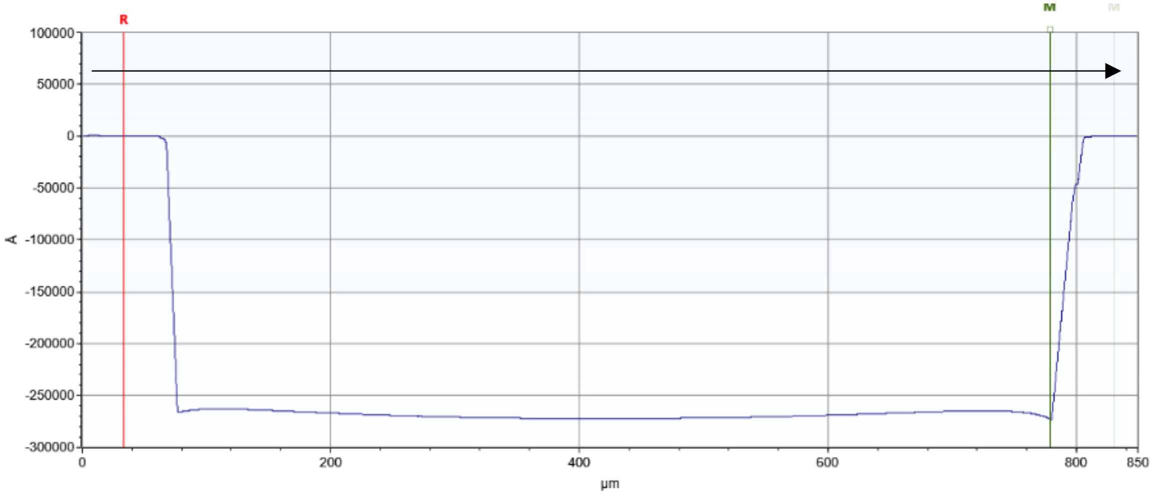
Resolved by eliminating O_2 in the process

Etching defect : *microtrenching*

RIE-ICP : 400 W Platen + 1600 W ICP
CF_4 25 sccm + Ar 25 sccm
10 mTorr
27,1 µm etched (113 nm/min)

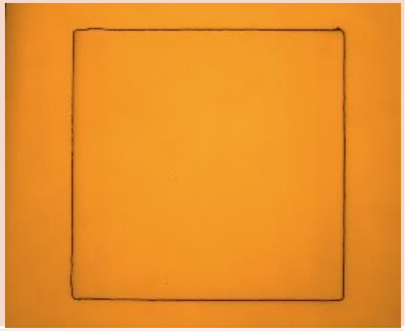
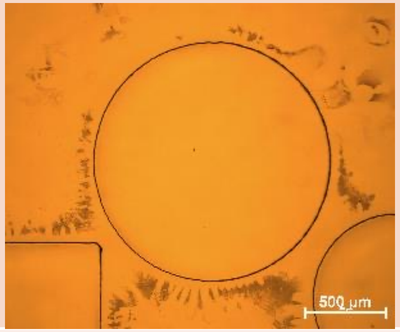
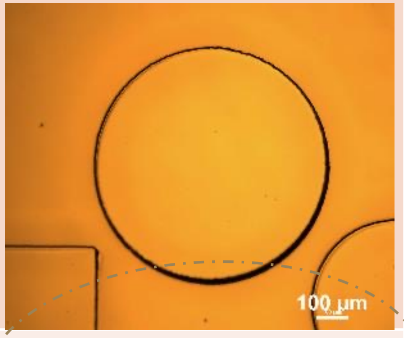






Microtrenching : 800 nm (3 % of etched depth)

Comparing etching recipes

Recipe	RIE-**CCP** : 280 W 50 sccm O_2 + 7 sccm CF_4 77 mTorr	RIE-**CCP** : 280 W Only CF_4 77 mTorr	RIE-**ICP** : 400 W + 1600 W Only CF_4 10 mTorr	RIE-ICP : 400 W + 1600 W 50 % CF_4 + 50 % Ar 10 mTorr	RIE-ICP : 400 W + 1600 W 40 % CF_4 + 60 % Ar 10 mTorr
Kinetic	80 nm/min	31 nm/min	133 nm/min	114 nm/min	83 nm/min
Optical characterization					
Profilometry (microtrenching)	No	No	10 % (~800 nm for 8,5 µm)	3 % (~800 nm for 27,1 µm)	1 % (~100 nm for 10 µm)

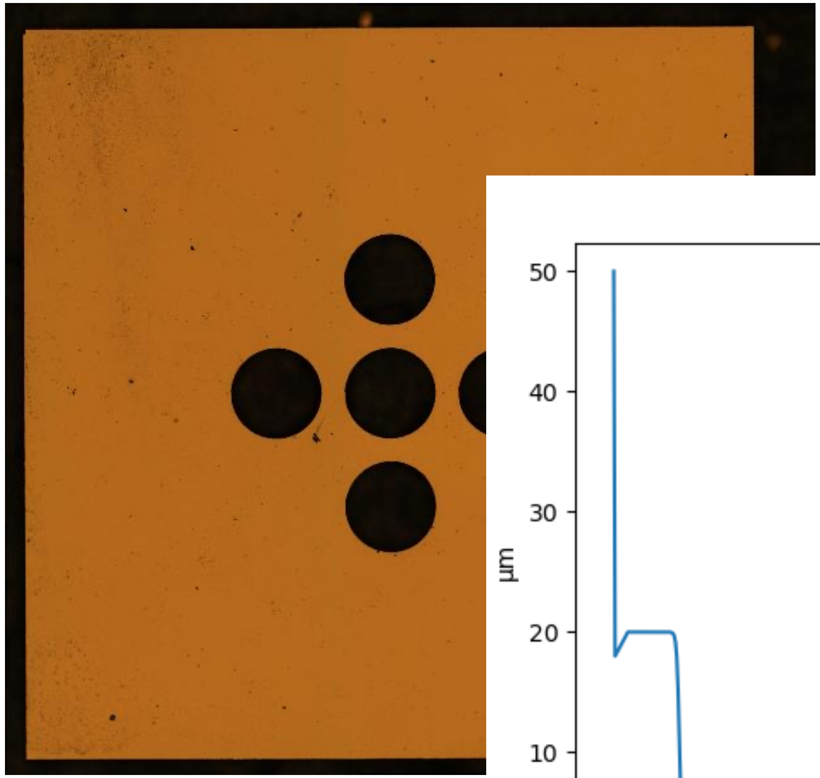
Promising but needed to be confirmed in longer etching

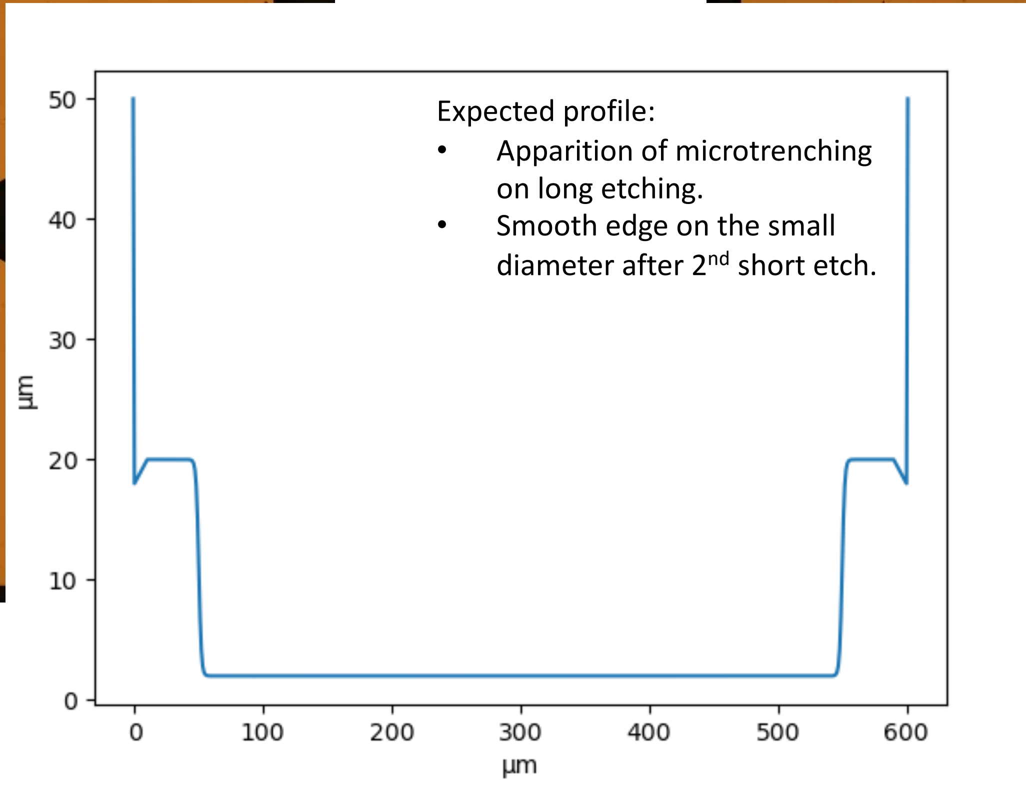
03

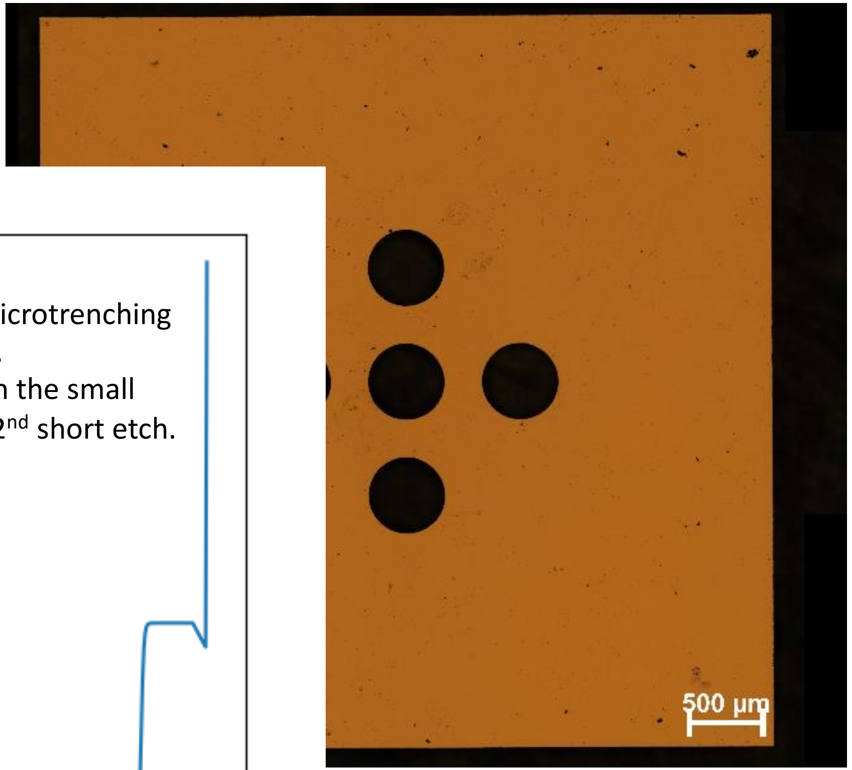
FABRICATION OF MEMBRANE DETECTOR

Etching process with two masks

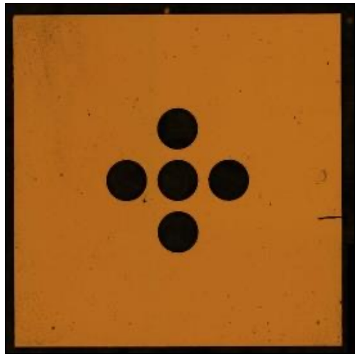
- ✓ Concentric patterns
- ✓ Diameters difference of 100 µm





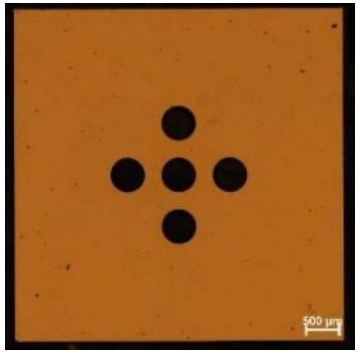


Etching process with two masks : optical and profilometer characterization



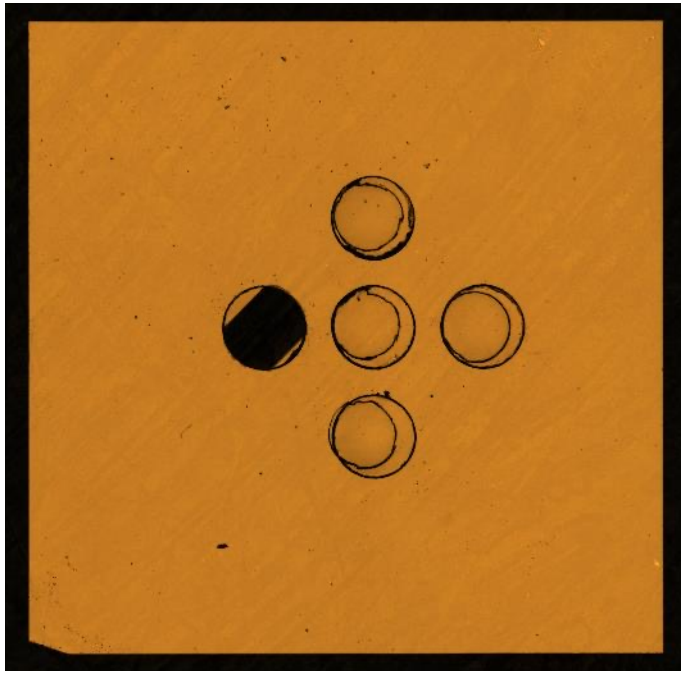
First deep etching

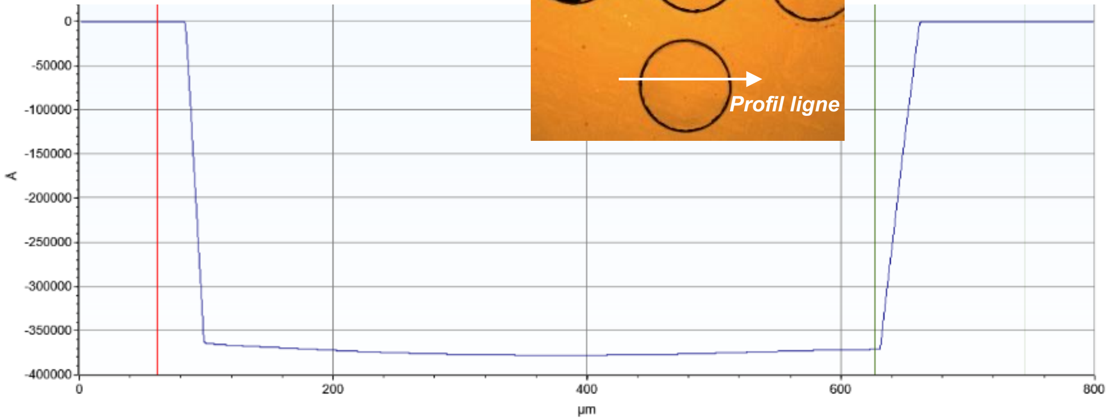
With Ø 600 µm mask



Second deep etching

With Ø 500 µm mask

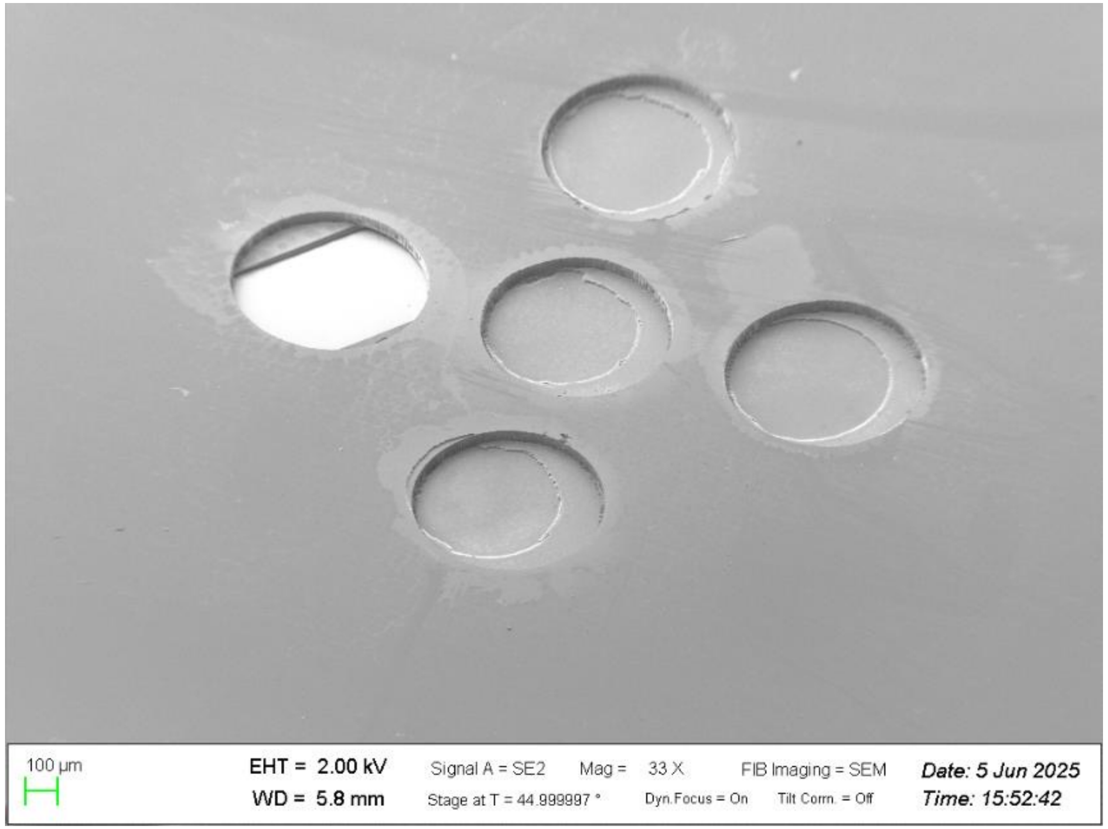


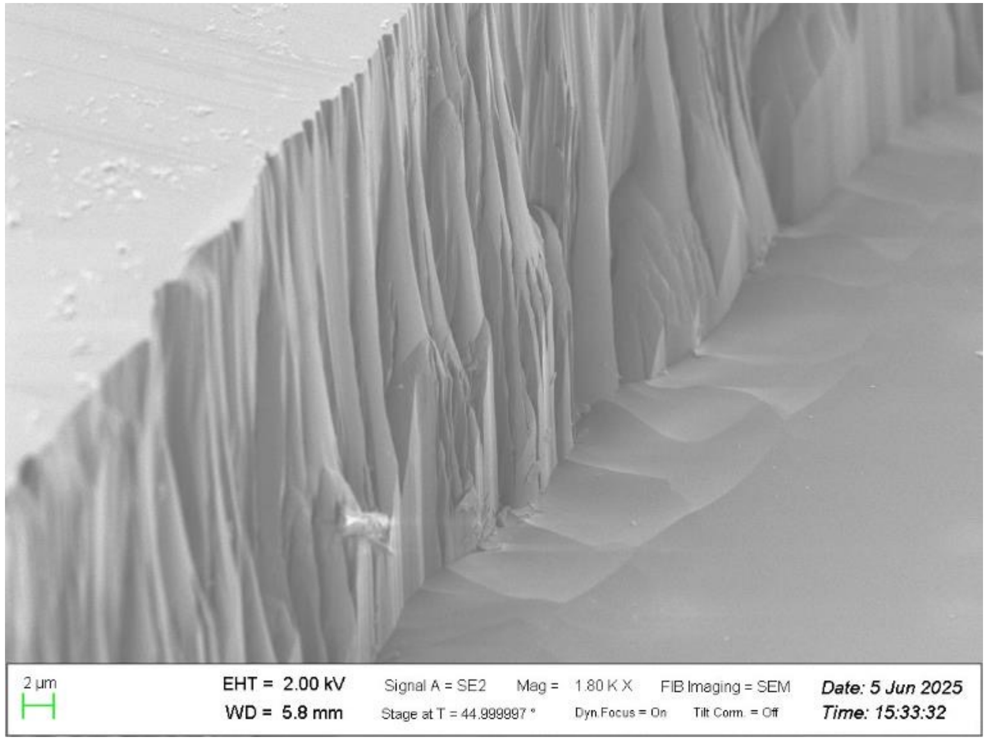


- 37,8 µm etched in 6h.
- One of the window broke during cleaning steps using ultrasound.
- No microtrenching measured by profilometer.

- Imperfect alignment of the two masks resulted in an offset in patterns.
- 42,4 µm etched in total, membrane is less than 8 µm thick.

Etching process with two masks : MEB characterization





- Microtrench spread out on ~10 µm but limited in depth.
- Etched surface is smooth and defect free.

CONCLUSION & PERSPECTIVES

Thinning of diamond for membrane detector is a real challenge:

- ❑ Etching defects are a problem for detection and could rupture the window. Oxygen in etching recipes is responsible for revealing etch pits. High kinetic recipe produce also microtrenching.
- ❑ Adjustments on etching recipe permit a sufficient kinetic with good surface quality for detector purpose but do not eliminate microtrenching problematic, only reduce it.
- ❑ A 8 µm-thick membrane was fabricated, with limited microtrenching and low surface defects concentration.

Test on detector capability are planned in July at AIFIRA.

Ultimate thinning of the membrane without any mask is considered.

LPSC
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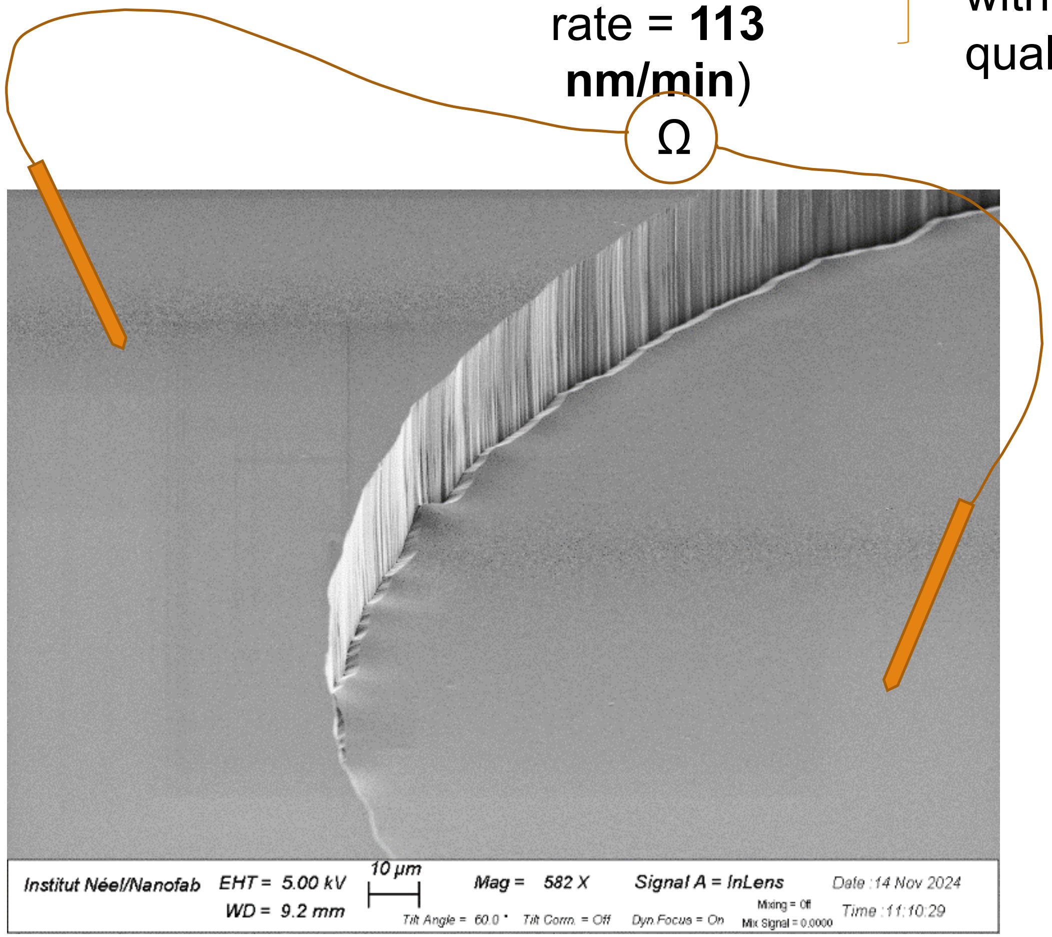
BACK-UP SLIDES

Contact deposition for bias and signal collection

27,1 µm on a test sample (etching rate = **113 nm/min**)

Deepest etching yet with this surface quality

400 W Platen + 1600 W ICP
CF_4 25 sccm + Ar 25 sccm
10 mTorr

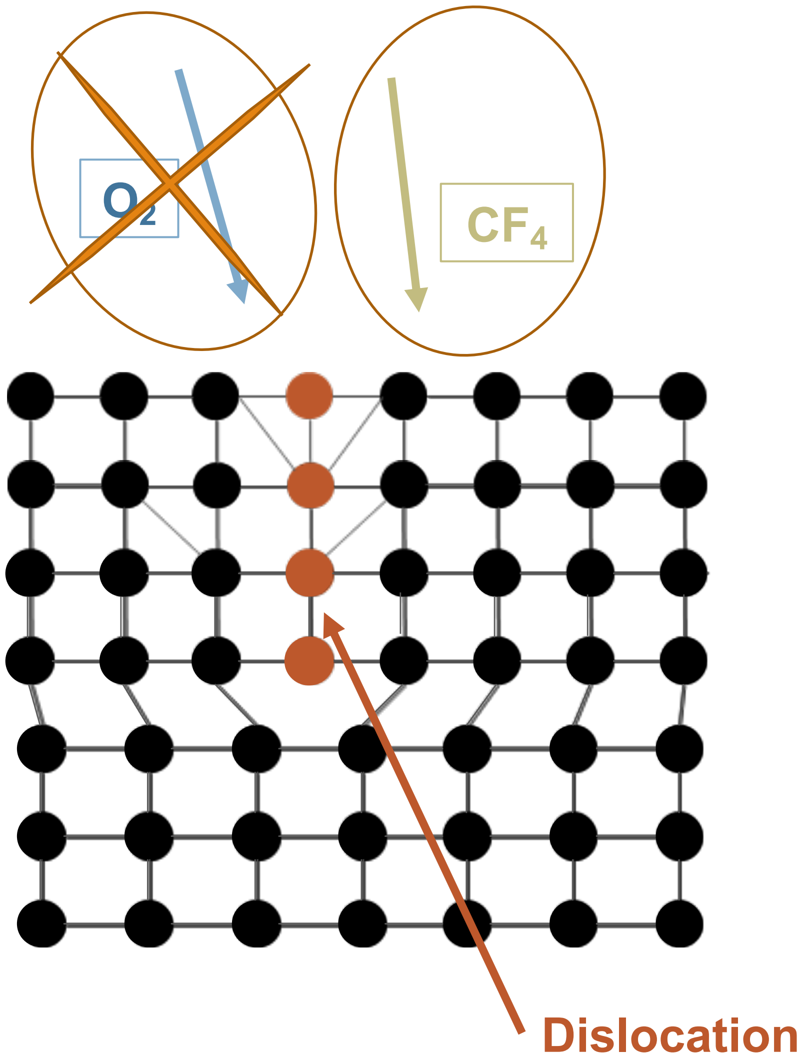


Next step = aluminum deposition
To ensure electric continuity between the bottom of the pit and the non-etch surface of the diamond

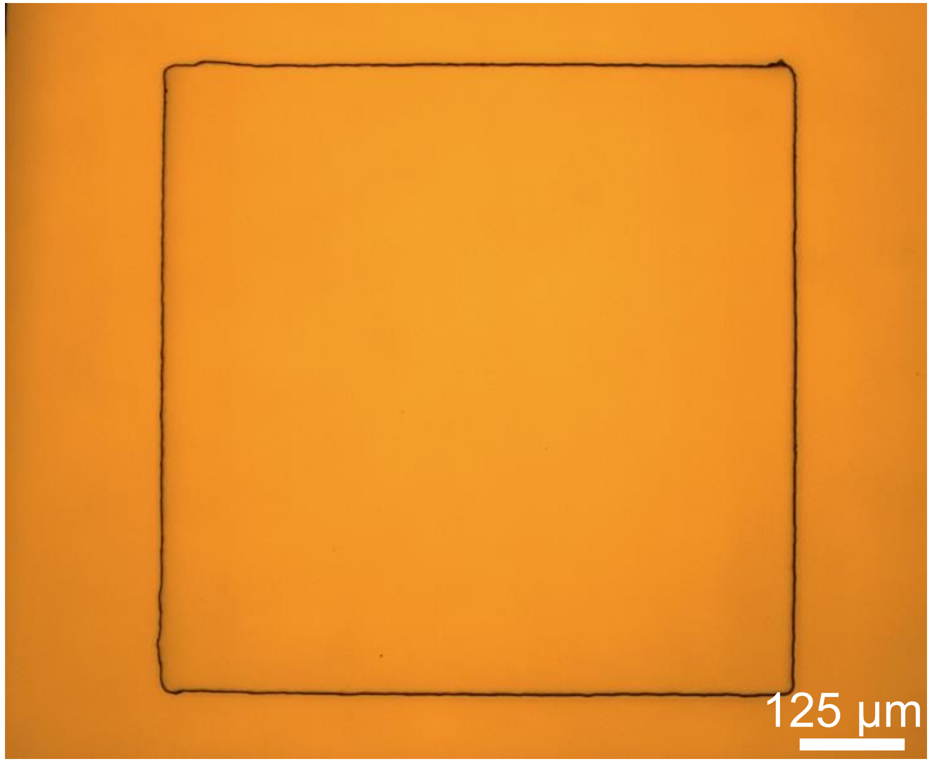
The technique of deposition is suitable for contact even with steep angles.

Measured resistance of a **few ohms** only

Evolution of the etching recipe: oxygen-free = etch pits-free



280 W
CF4 50 sccm
77 mTorr



1h20 etching
2,4 µm

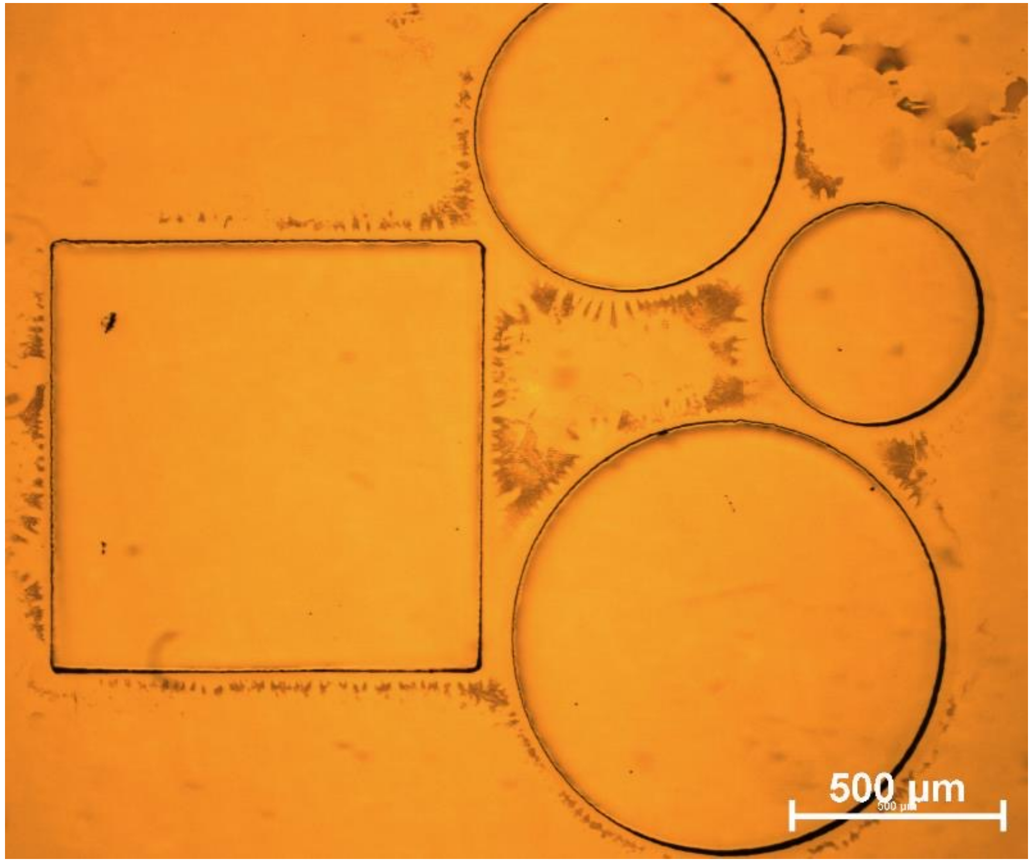
Etching rate = 31 nm/min

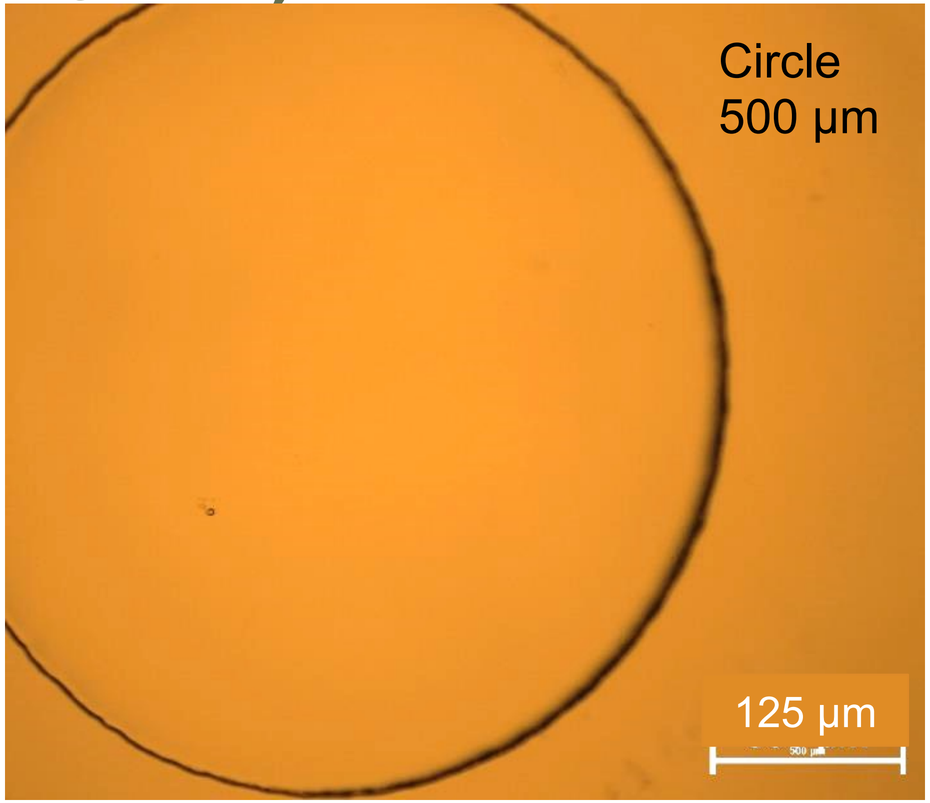
- Really slow, but without any etch pit.

Change of etching device: RIE ICP (oxford @ CEA)

ICP: **I**nductively **C**oupled **P**lasma,
Decoupling plasma density and acceleration with two distinct RF power

400 W Platen + 1600 W ICP
CF_4 50 sccm
10 mTorr





1h etching 8,5 µm ⇨ **Etching rate = 142 nm/min**

Rugosity: **14 nm RMS** for 9 nm initially

} Compatible with detection device manufacturing requirements