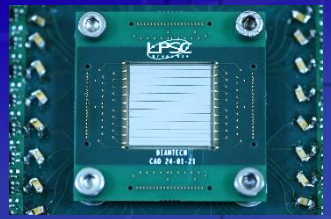
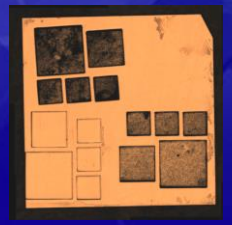
MICRO/NANO-FABRICATION EXAMPLES :

DIAMOND SENSOR MATRIX

ULTRATHIN DIAMOND MONITOR





MOTTE Jean-François, NanoFab clean room, Institut Néel CNRS

neel.cnrs.fr

MICRO/NANO-FABRICATION EXAMPLES

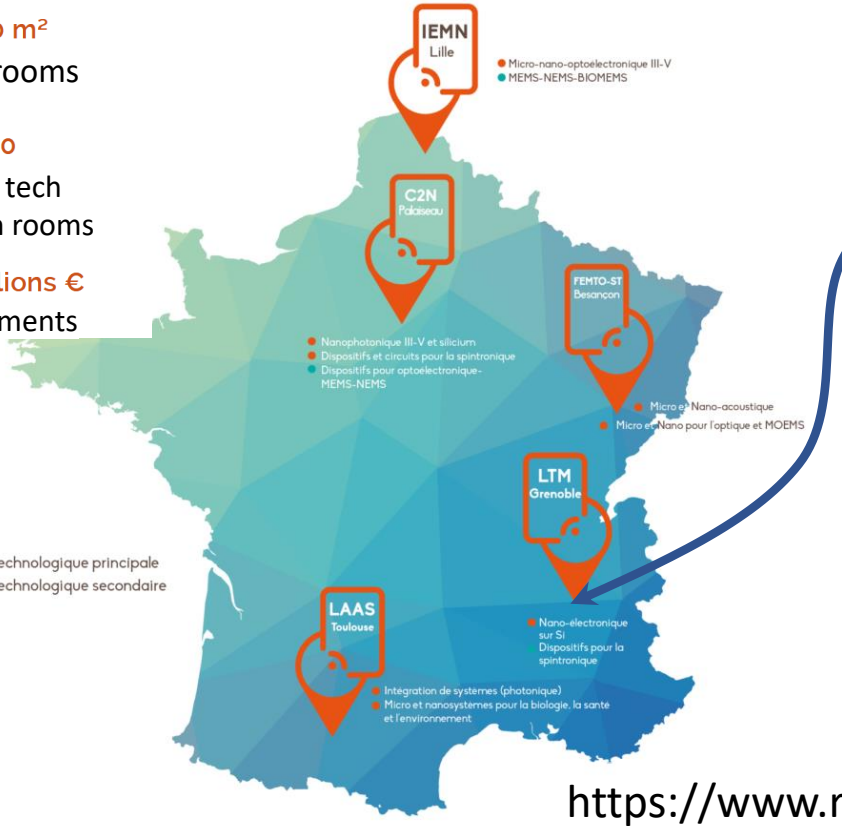
1. NanoFab a local clean room at Néel in RENATECH network
2. Context: diamond developments - multidisciplinary activities
3. Why go from Macro to Nano two ways
4. FISTR ex : Diamond sensor matrix beam monitoring in radiotherapy
5. Second ex : Ultra-fine diamonds and their problems
6. NanoFab Porfolio

NanoFab a Local clean room at Néel in

8300 m²
Clean rooms

150
Ing & tech
in clean rooms

130 millions €
equipements



RENATECH is the French academic network for cutting-edge equipment in the field of micro and nanotechnologies, led by the CNRS.

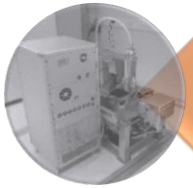
Our aim is to develop, maintain and supply a competitive infrastructure for research and R&D in micro- and nanofabrication in France.

We work with both academic and industrial customers.

https://www.renatech.org/

NanoFab a Local clean room at Néel

TECHNOLOGIES



LITHOGRAPHY

- Electron beam : 30/80kV
  (SEM LEO1530 & NB5)
- Optical UV
  (UVKUB-Smart Print-lithoLASER)

COATING DEPOSITION :

- Joule effect or electron gun
- Plasma spraying
- UHV

ETCHING :

- RIE (O_2, SF_6, CHF_3,CF_4, Ar)
- IBE (Ar)
- Chimique (XeF_2, KOH, …)
- FIB

SCIENTIFIC FIELD

- MECHANIC
- THERMIC
- BIOLOGY
  - Coponent/sensors
    - innovative radiotherapies is the objectives of radiobiology
- ELECTRONIC
  - Captors/components
    - Plasmonic (FIB)
    - Quantum engineering
    - Power electronics (diamond)
    - …

POSTER SESSION

CONTEXT: DIAMOND DEVELOPMENTS - MULTIDISCIPLINARY ACTIVITIES

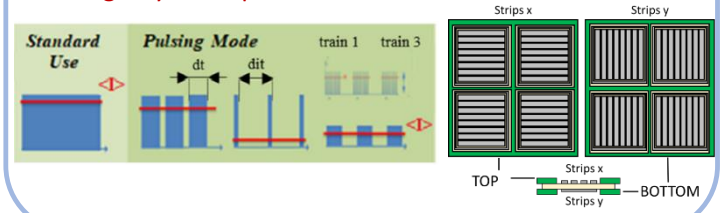
Medicals Physics

- **Beam monitoring in radiotherapy** (position/time labeling/counting)
- **on-line monitoring of ion microbeams**
  - Large area *(>1cm²)*
  - *High counting rate (100 MHz)*
  - *Good sensitivity and high dynamic range (detection of single particles in bunches up to 1010 particles in trains for Flash therapies)*
  - *Resistance to irradiation*

ANR DIAMMONI 2020-2024
LPSC / SUBATECH/ARRONAX

ANR-DIAMMONI ML Gallin-Martel et al.

High-dynamic pulsed-beam diamond monitor



on-line monitoring of ion microbeams
LPSC / LP2i / IRSN

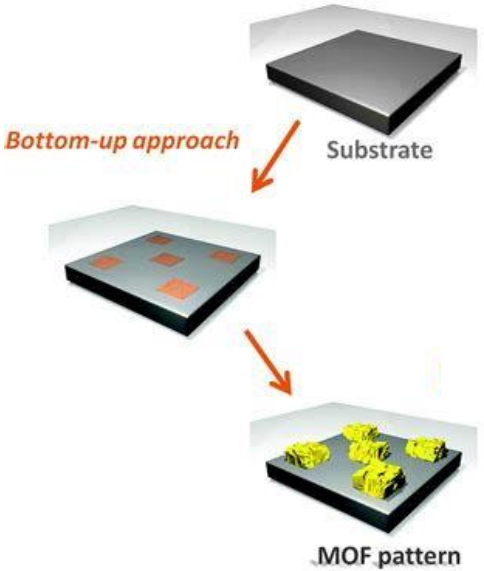
DEFI Diams Thèse de C. Léonhart

AIFIRA / MIRCOM

Diamond 4mm² x 50µm
window 500µm², 1 to 2µm thin
Metallized electrodes for diamond polarization and signal reading

Bottom-up

- Building nano-objects from individual building blocks (atoms, molecules)
- Self-organisation



Ex : Preparation of organometallic structures

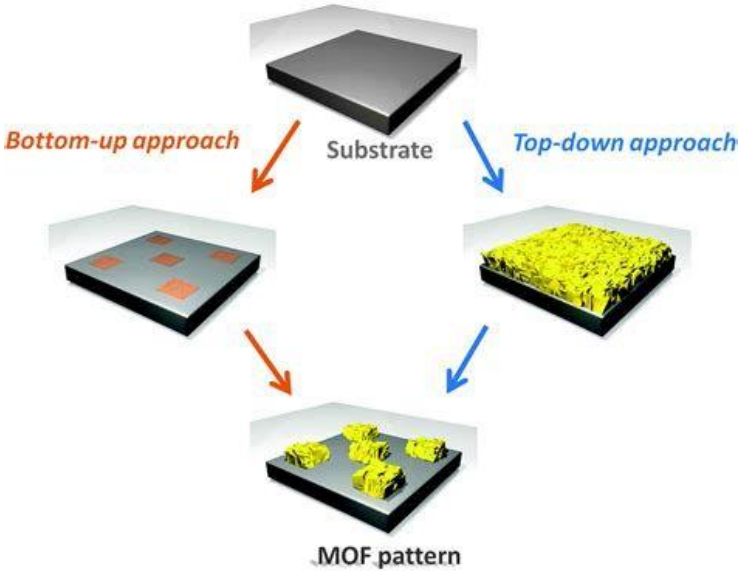
WHY GO FROM MACRO TO NANO TWO WAYS

Bottom-up

- Building nano-objects from individual building blocks (atoms, molecules)
- Self-organisation

Top-Down

- Shaping substrates
- Reducing the size of the "macroworld
- Lithography



Ex : Preparation of organometallic structures

CONTEXT: DIAMOND DEVELOPMENTS - MULTIDISCIPLINARY ACTIVITIES

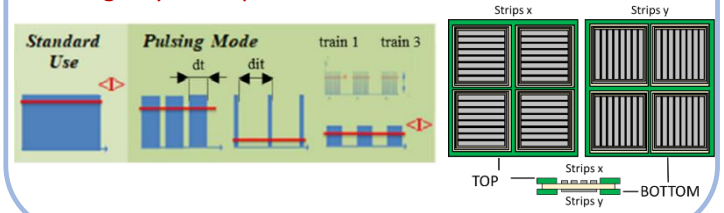
Medical Physics

- **Beam monitoring in radiotherapy** (position/time labeling/counting)
- **on-line monitoring of ion microbeams**
  - Large area *(>1cm²)*
  - *High counting rate (100 MHz)*
  - *Good sensitivity and high dynamic range (detection of single particles in bunches up to 1010 particles in trains for Flash therapies)*
  - *Resistance to irradiation*

ANR DIAMMONI 2020-2024
LPSC / SUBATECH/ARRONAX

ANR-DIAMMONI ML Gallin-Martel et al.

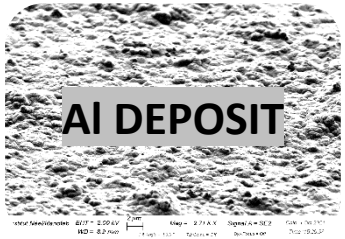
High-dynamic pulsed-beam diamond monitor



8. DIAMMONI : un détecteur pour le monitorage en ligne de très hauts flux pour le cyclotron d'ARRONAX
Robin Molle (LPSC - Grenoble)
26/06/2024 09:30
Semiconductors

DIAMOND SENSOR MATRIX
BEAM MONITORING IN RADIOTHERAPY

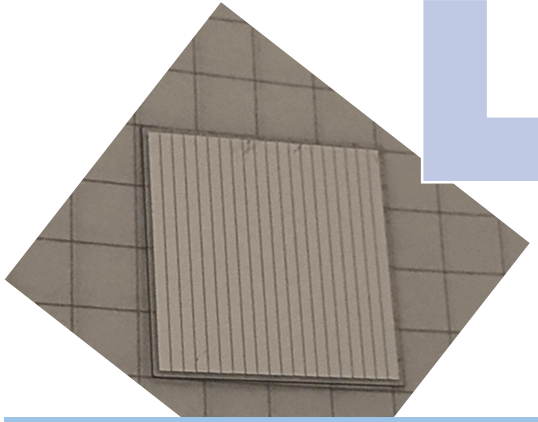
Latifa Abbassi (Institut Néel)

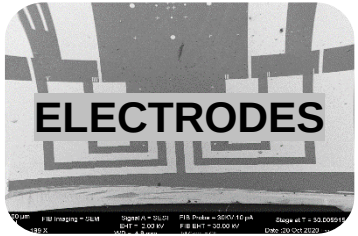


- Optical microscope observation, roughness, etc...
- Cleaning acid mixture
- Deposit quality control

state of the surface

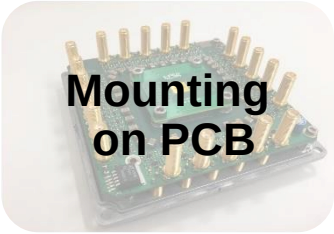
Melvyn Reynaud (LPSC)





- Resist spin cont^rol
- Insulating the resist with a laser
- Developpement
- Chemival etching

Write resolution
Sample size

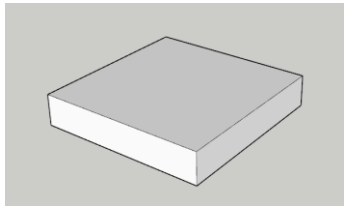


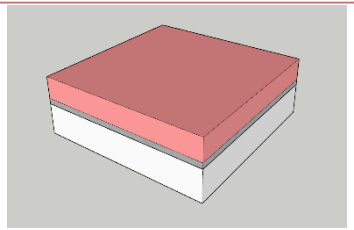
- Mechanical
- Assembly
- Bonding

Pixelated diamond sensors
- 19mm²
- 20 tracks 0,9mm spaced 0,05mm, accuracy 0,01mm

Diamond substrat

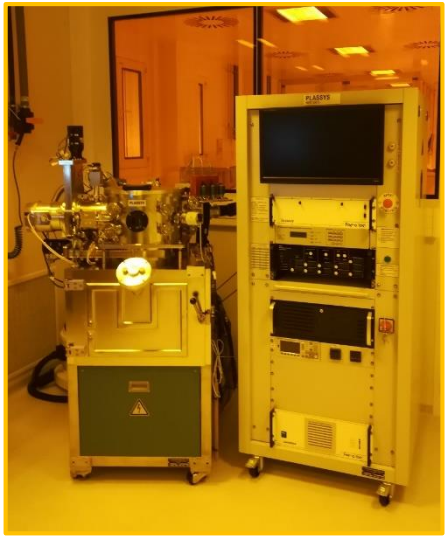
Coat 100nm Aluminium + ***ZPN resist 500/800nm***





Electron gun evaporator MEB550S PLASSYS

- Vacc (4.10-8mBar) Turbo + Cryo
- 8 available materials



Spin Coater

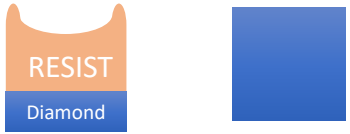
- Clean room and laminar flow (dust)
- Under hoods for toxic chemical

Spreading of resin by rotation (1000 to 10000 rpm)

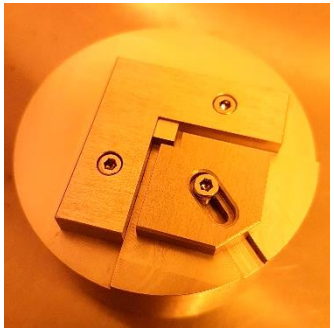
Annealing required to evaporate the solvent

- Major difficulties for diamonds :
  - Transparency
  - Shape and thickness: handling and even spreading of the resin

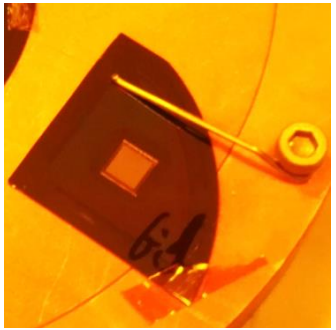
➢ Classical circular wafer 2” (50,8mm)



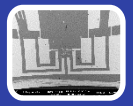
➢ 10mm² to 4 mm² square diamond



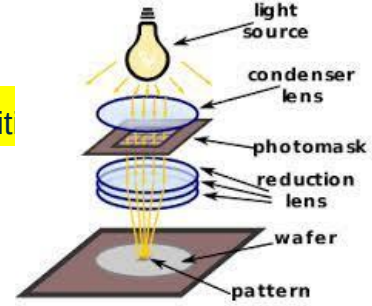
New sample holder
to limit edge effects



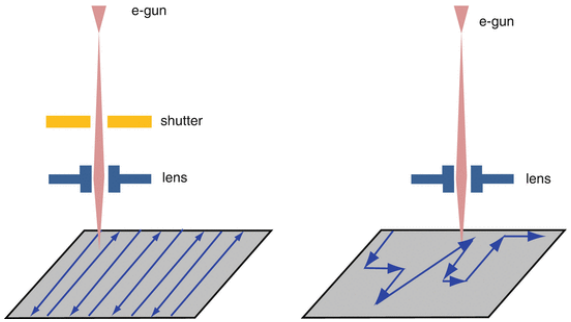
“gluing” to a support substrate
for diamonds < 200µm thick



– WITH RESIST (photon-électron sensit
  - Optical lithography
  - Electron lithography
  - Ion beam lithography

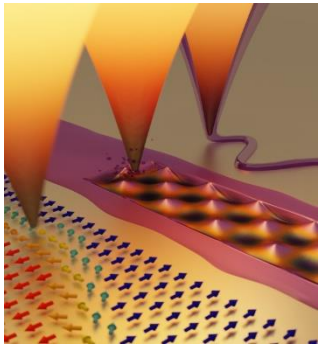


Projection photolithography through a mask



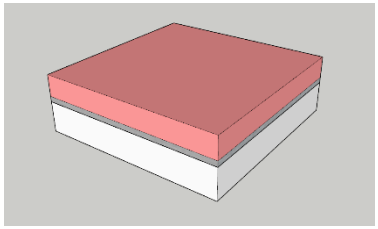
Direct-write lithography
LASER-eBeam

– WITHOUT RESIST
  - Ion beam (FIB)
  - Near field (AFM,STM)

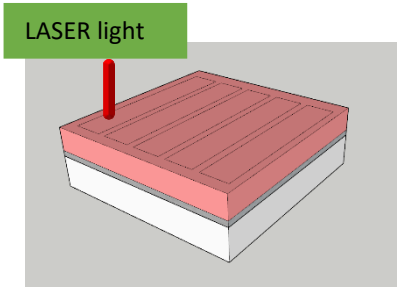


Direct-write lithography with local probe

Transfer a design



LASER lithography
direct writing

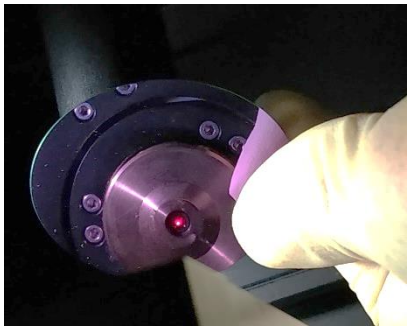


2 types of resists:

- ***Positive***
  light makes the resist soluble
- ***Negative***
  light makes the resist insoluble

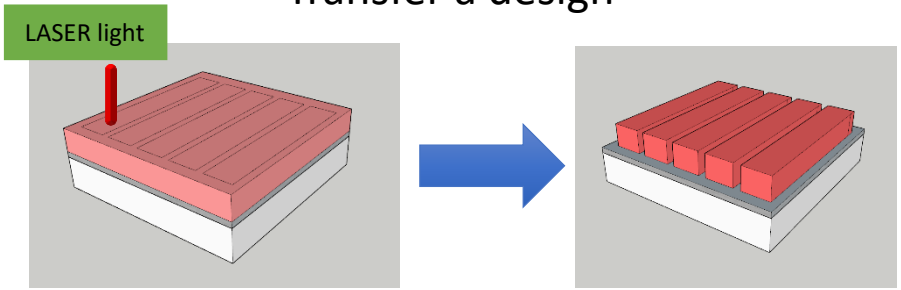
LASER lithography Heidelberg DWL 66S

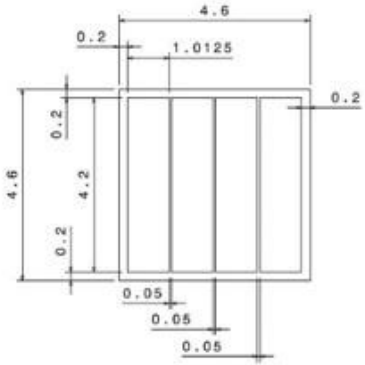
- Resolution 1µm
- 160mm max writefield
- direct writing = throughput

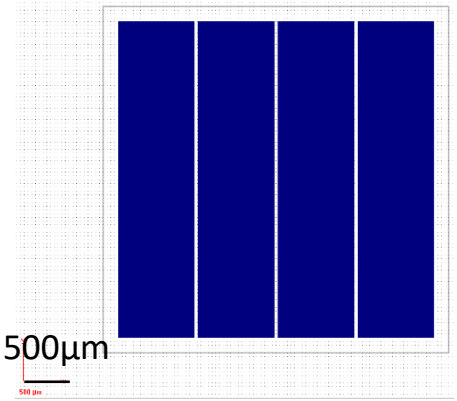


LASER spot coming out of the writing head lens

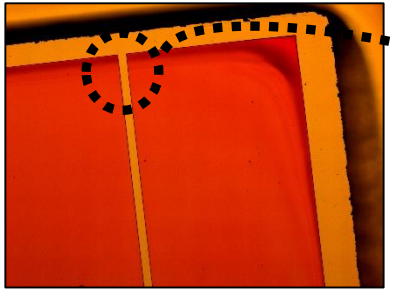
Transfer a design



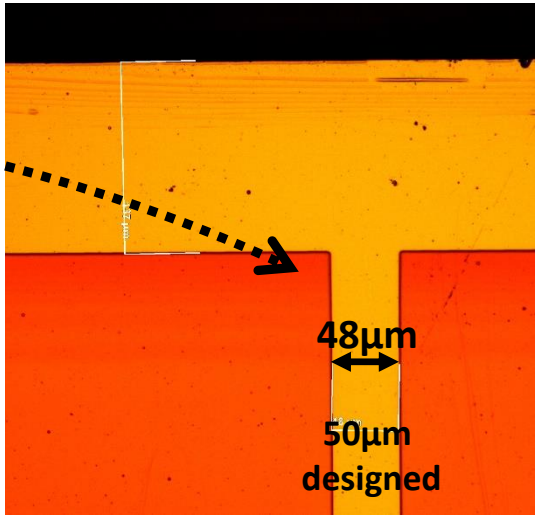




Conversion of CAD drawings into "GDS" lithography language

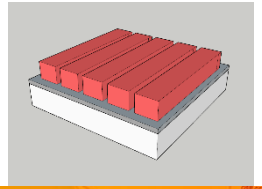


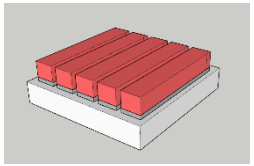
Drawing transferred in the resist onto the sample, after exposure and development

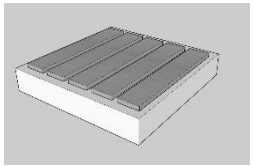


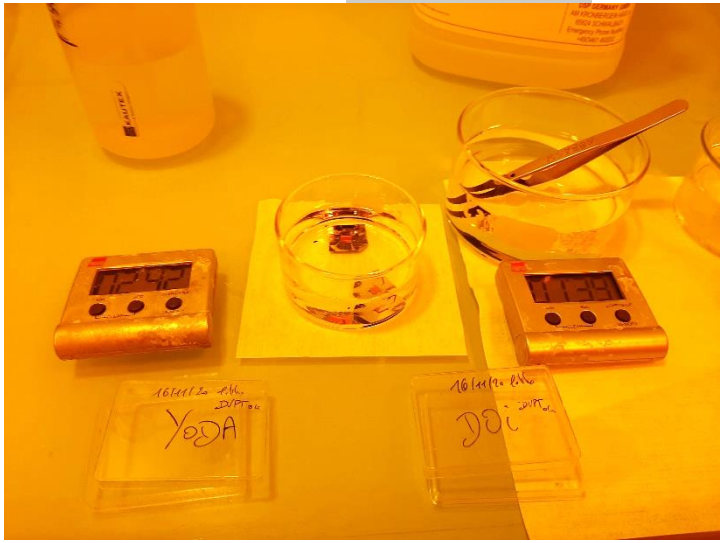
Aluminium etching (20nm/min)

Resist removed

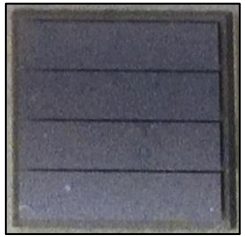


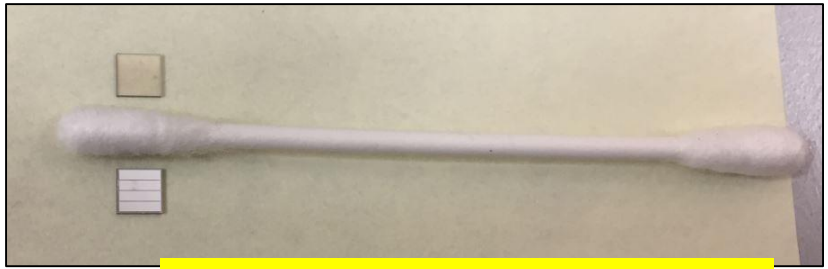






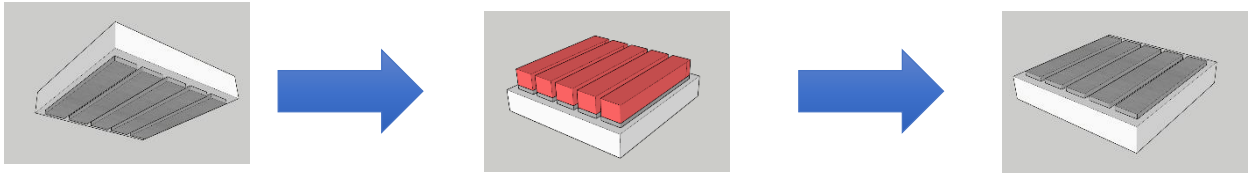
5 x 5 mm^2
1 x 4 traks
the trick is done
on one side





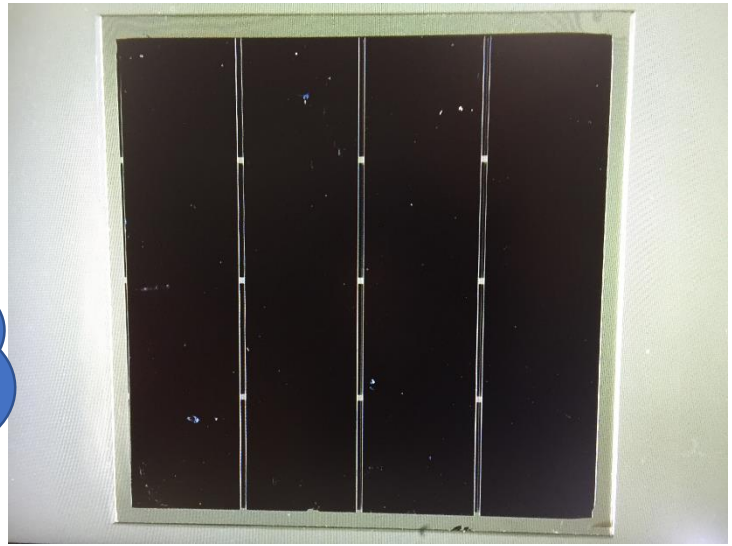
And now to do the second side

Bis repetita !!!!! Without damaging the rear face

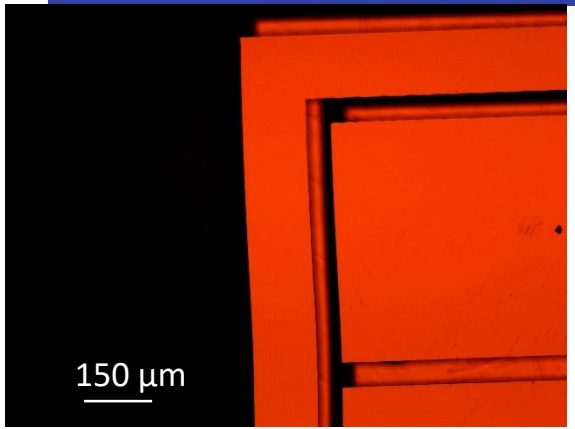


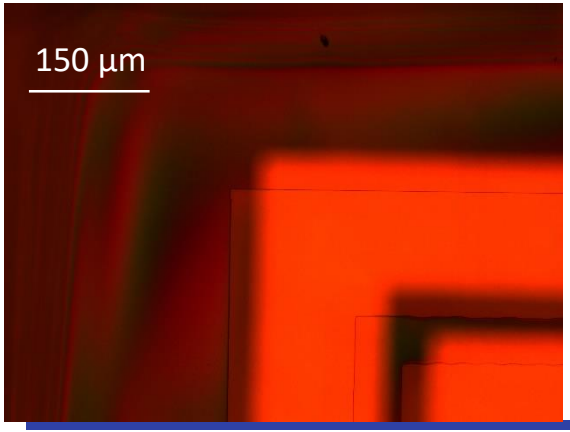
SCRATCH !!!
Protected and glued
on the back side

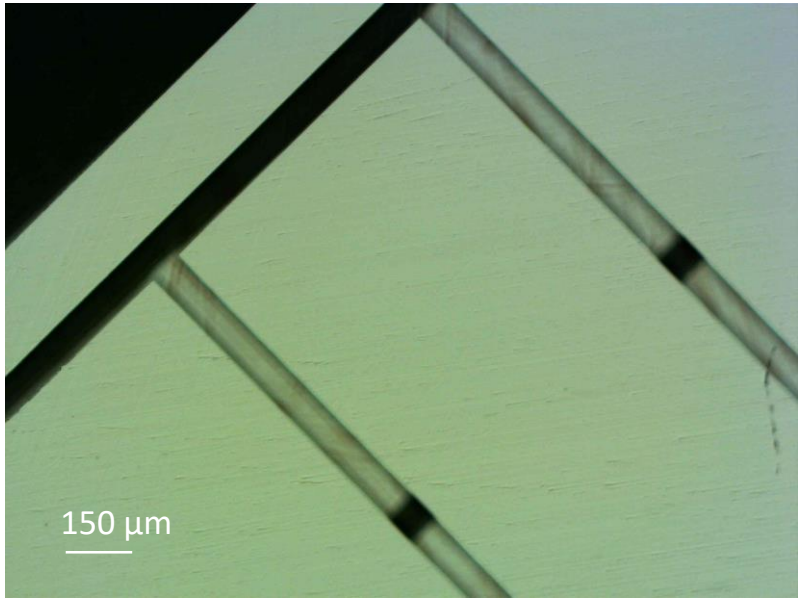
ROBUST PROCESS:
do not engrave
the back side

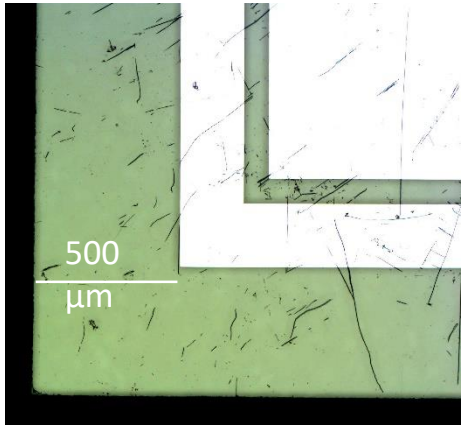


5 x 5 mm²
2 x 4 tracks at 90°
2 sides

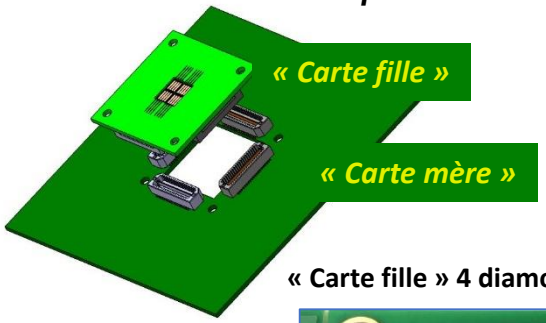

150 µm


150 µm

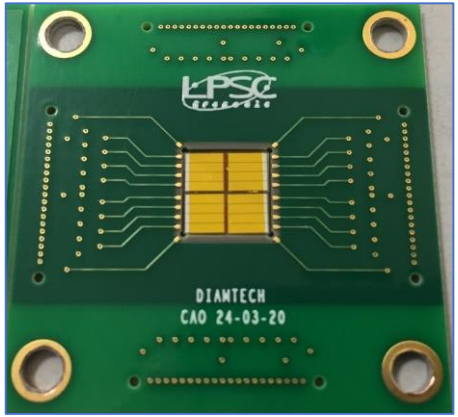

150 µm


500
µm

3D Conception



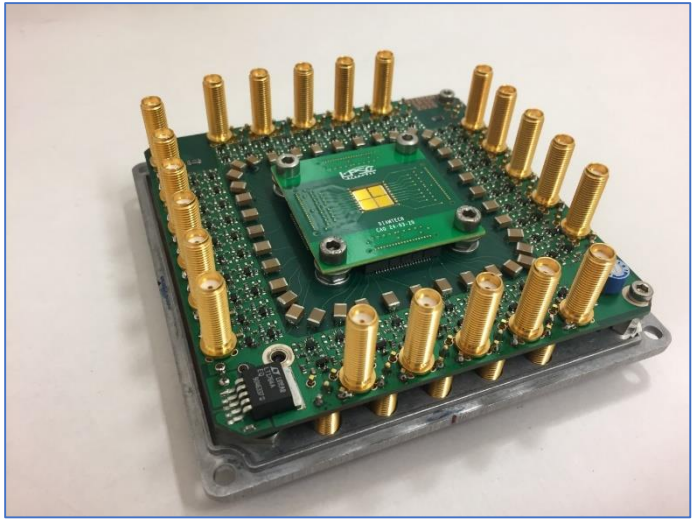
« Carte fille » 4 diamonds sc – 32 channels



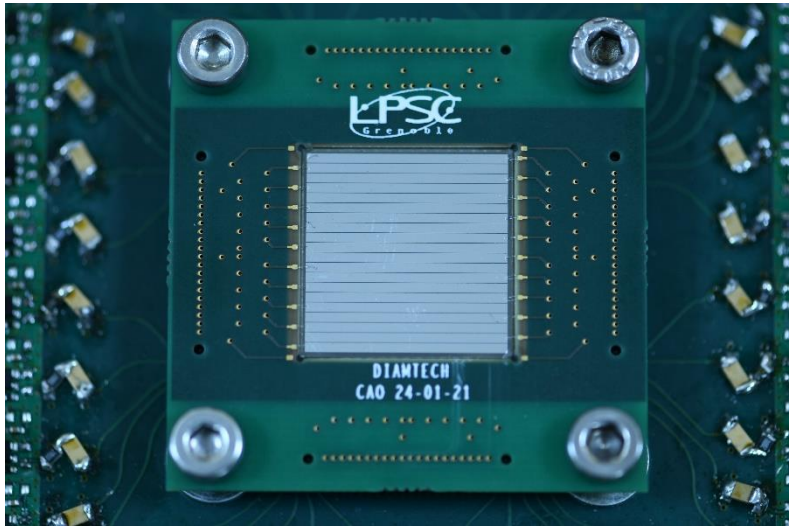
Jean-Luc Bouly, Germain Bosson, Marc Marton, J-F Muraz (LPSC)

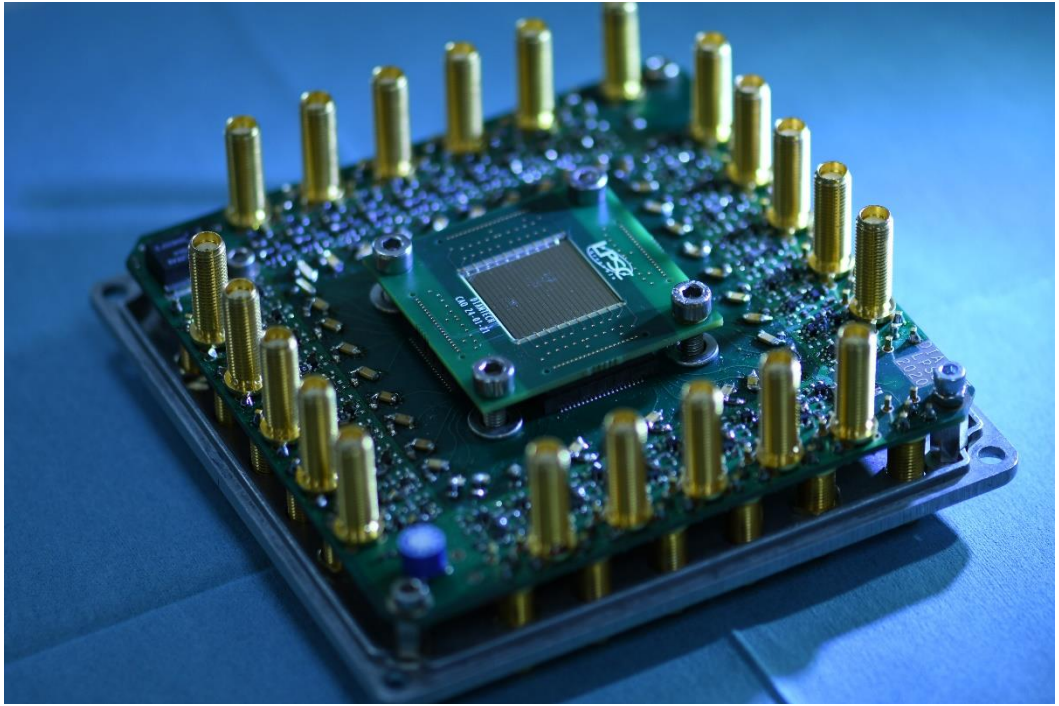
Advantages:

- 1 “carte fille” per diamond
- Assembly quick changeover (simply remove “carte fille”)
- Reuse of the same amplification channels

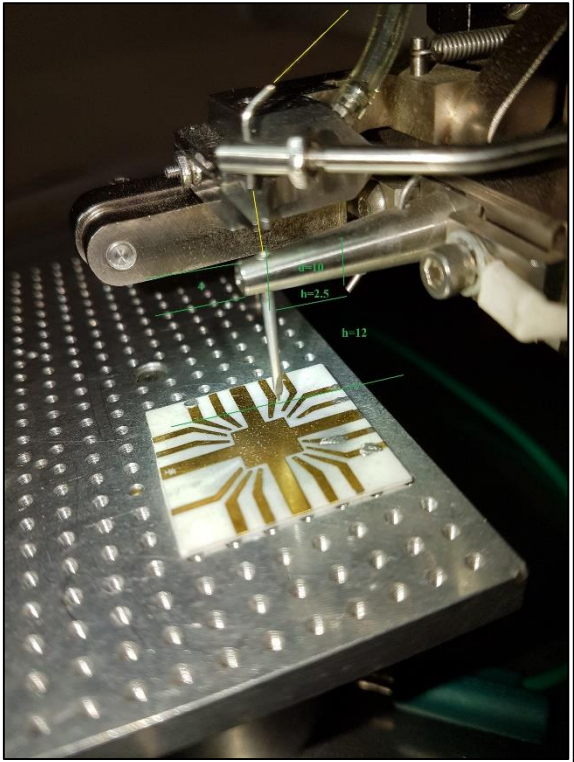


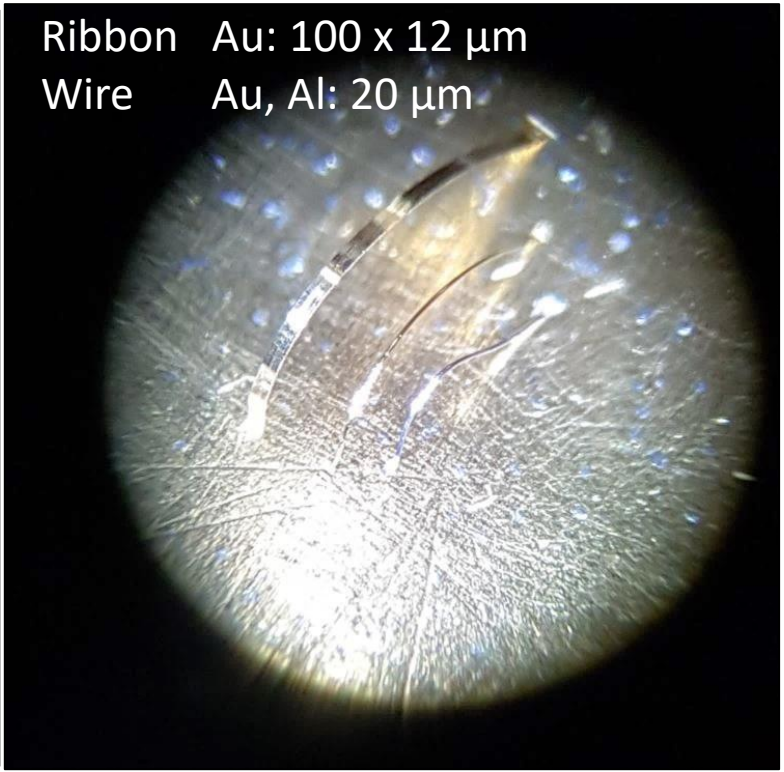
- Hodoscope 4 Diamonds (carte mère & fille 32 channels)
- sc 9.6 x 9.6 mm², 32 strips

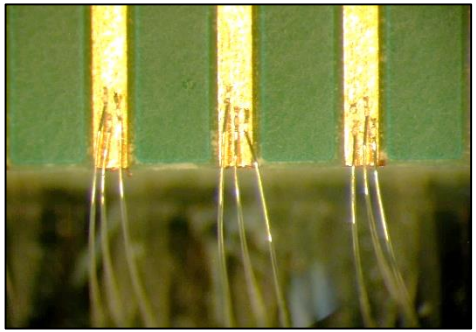




Hodoscope 1 Diamond
(carte mère & fille 40 channels)







Manual operation !!!
Under binoculars

Thierry CROZES (Institut Néel)

Medicals Physics

- **Beam monitoring in radiotherapy** (position/time labeling/counting)
- **on-line monitoring of ion microbeams**
  - Large area *(>1cm²)*
  - *High couting rate (100 MHz)*
  - *Good sensitivity and high dynamic range (detection of single particles in bunches up to 1010 particles in trains for Flash therapies)*
  - *Resistance to irradiation*

on-line monitoring of ion microbeams

LPSC / LP2i / IRSN

DEFI Diams Thèse de C. Léonhart

AIFIRA / MIRCOM

Diamond 4mm² x 50µm
window 500µm², 1 to 2µm thin
Metallized electrodes for diamond polarization and signal reading

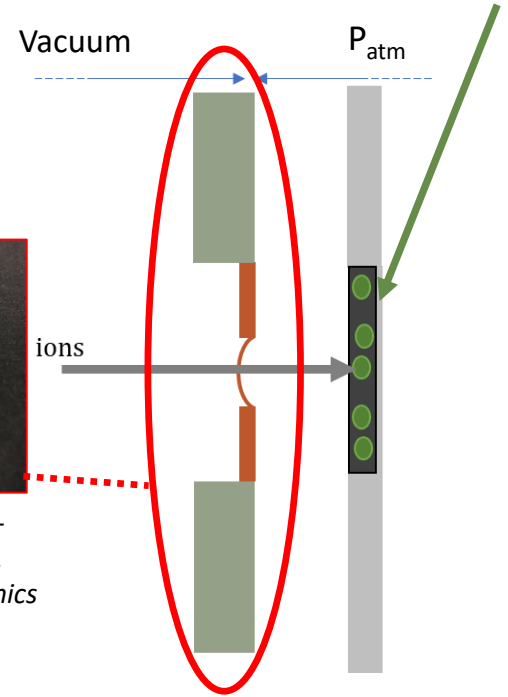
ULTRA-FINE DIAMONDS AND THEIR PROBLEMS

DEFI Diams Thèse de C. Léonhart

Why ultra-thin?

- ✓ To have a minimum quantity of material before the biological environment
- ✓ And to limit disturbances: loss of energy and spatial deviation

Diamond window placed on its sample holder upstream of **the biological medium**.



Diamond scCVD

Electronical or optical grade

Window 500µm², 1 to 2µm thin
Metallized electrodes for diamond polarization and signal reading

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

ULTRA-FINE DIAMONDS AND THEIR PROBLEMS

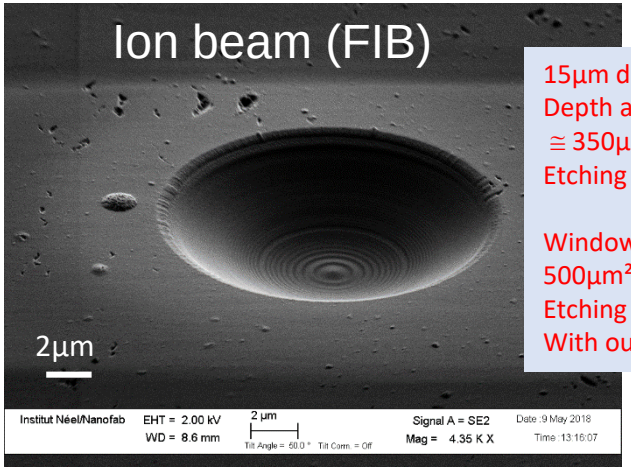
Diamond scCVD

Electronical or optical grade

Window 500µm², 1 to 2µm thin
Metallized electrodes for diamond polarization and signal reading

1. *MECHANICAL ETCHING :*

- Very High precision
- Too low Throughput

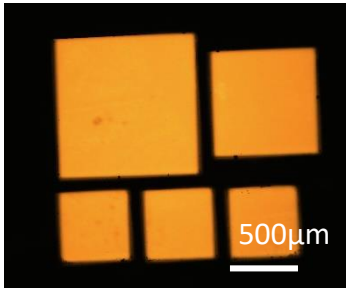


15µm diameter
Depth around 4µm
≅ 350µm³
Etching ≅ 15min

Windows
500µm²x49µm depth
Etching >8000H
With our FIB

2. *CHEMICAL REACTIVE ETCHING :*

- Very high Throughput
- Large surface (independent)
- Find a hard mask

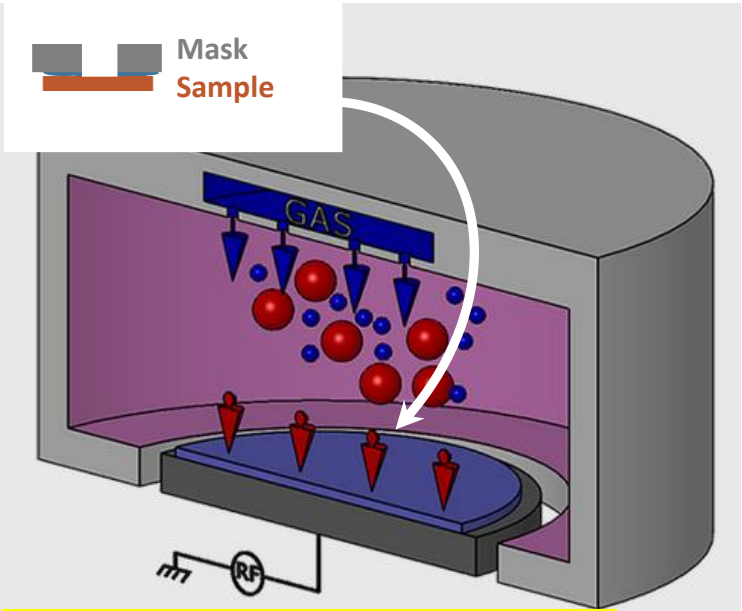


Diamond etching mask

Windows
1000/750/500µm²

Diamond etching : Principle and process

Principle of **R**eactive **I**on **E**tching

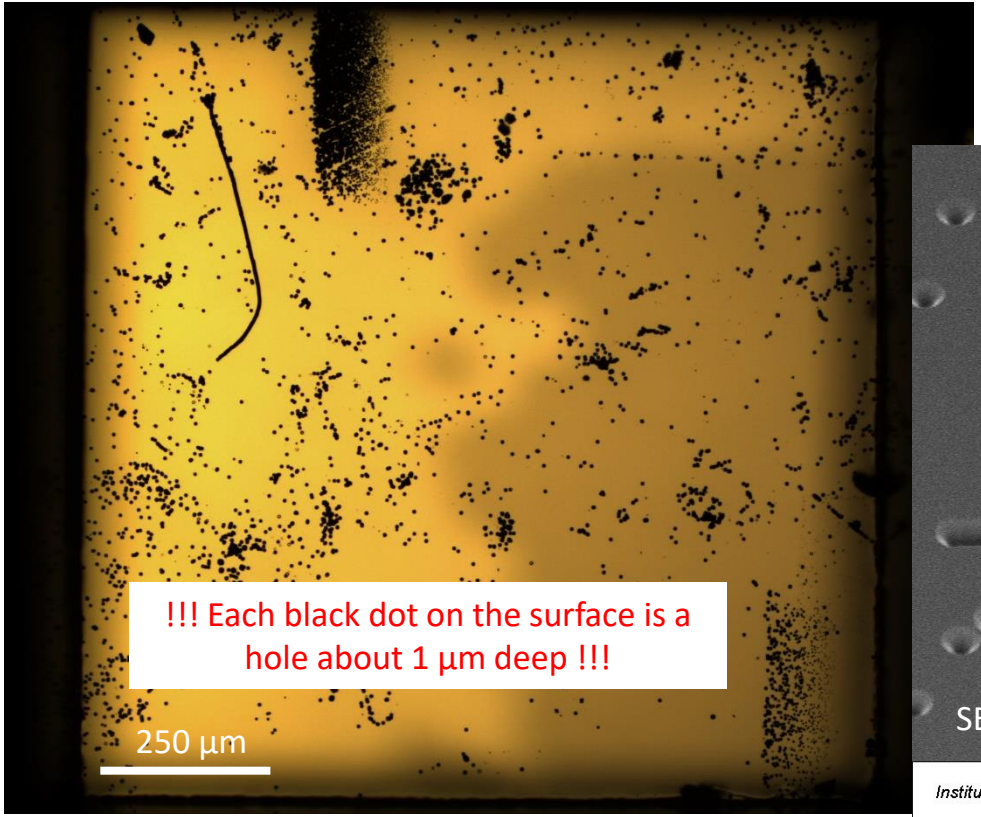


Recipe : CF_4 30sccm + O_2 40sccm / 150mT / 280W

https://corial.plasmatherm.com/en/technologies/reactive-ion-etching-rie

RIE machine on the Nanofab platform (PLASSYS)

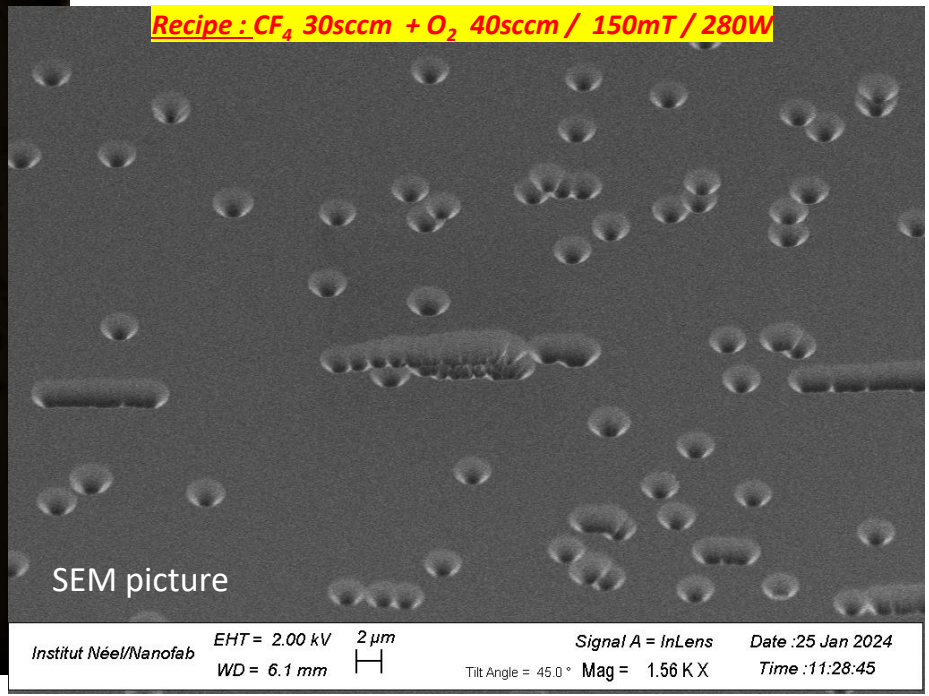
FIRST RESULTS: Surface state



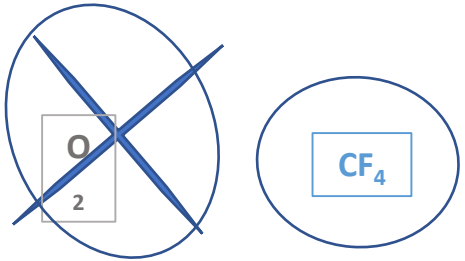
4h etching
19,2 µm depth

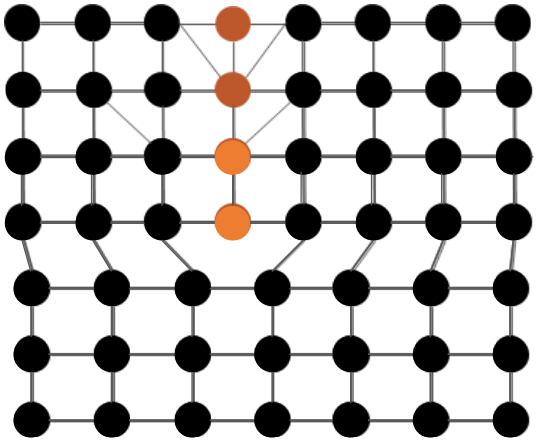
Less than half the target!

=> around 80 nm/min

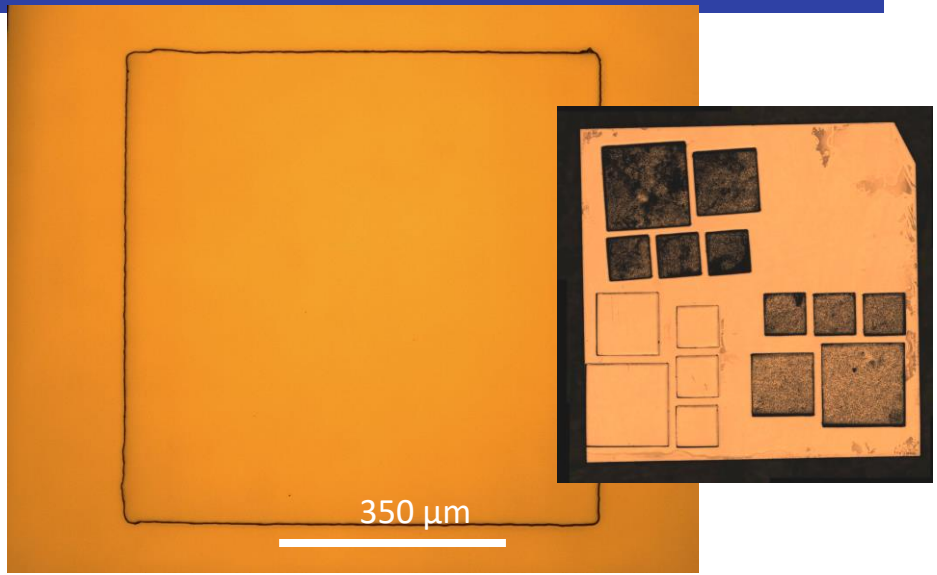


EVOLUTION OF THE ETCHING RECIPE : OXYGEN-FREE





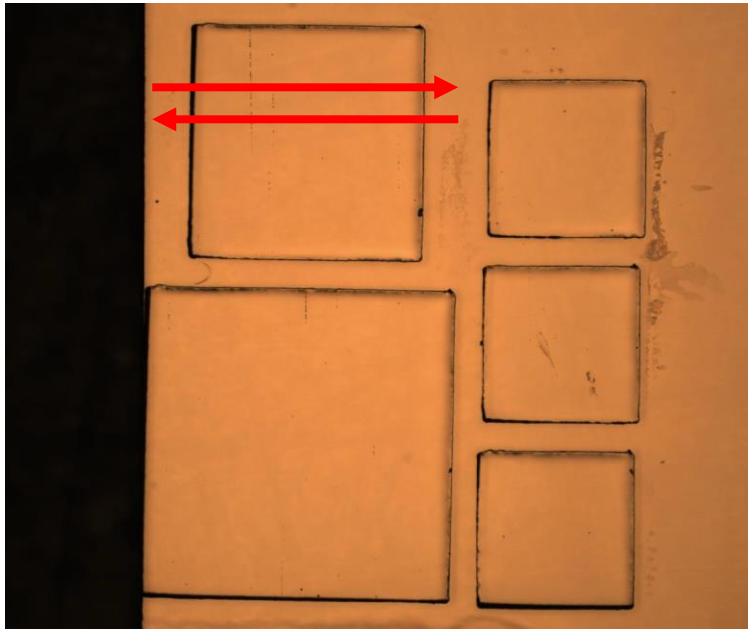
CF_4 50 sccm
10 mTorr
20°C (chiller)
400 W RF + 1600 W ICP
OXFORD via PTA



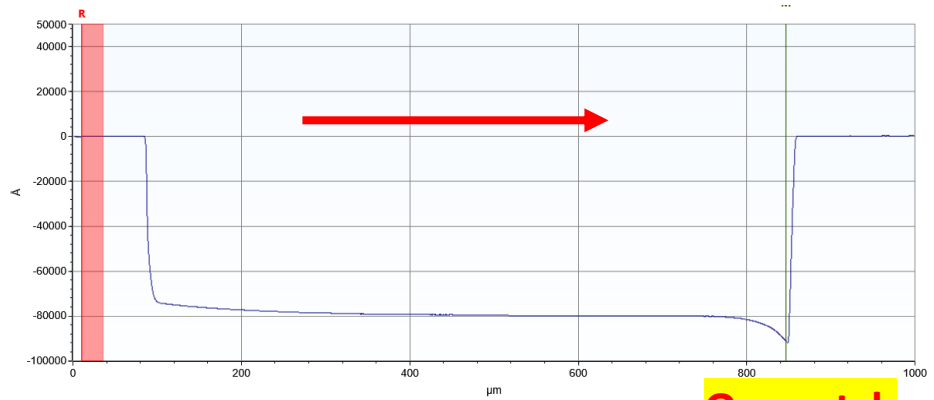
- Etch rate slower without O_2, but no etch pit formed.
- RiE-iCP more powerfull = increase the rate etch
- Recipe also validated for longer burn times.

Thomas Charvolin, PTA LTM

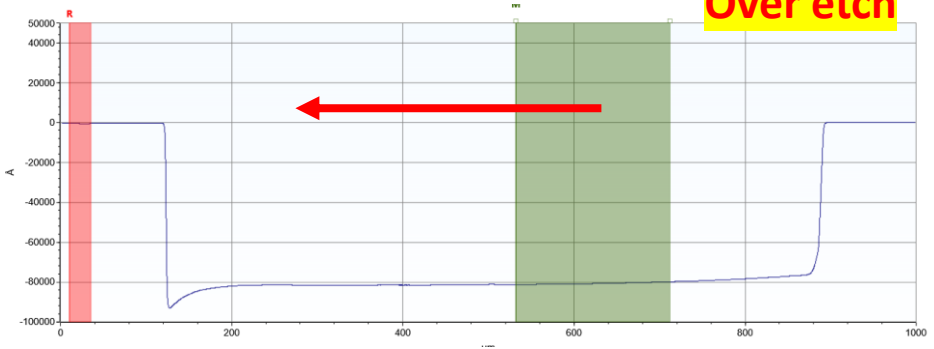
EVOLUTION OF THE ETCHING RECIPE : OXYGEN-FREE



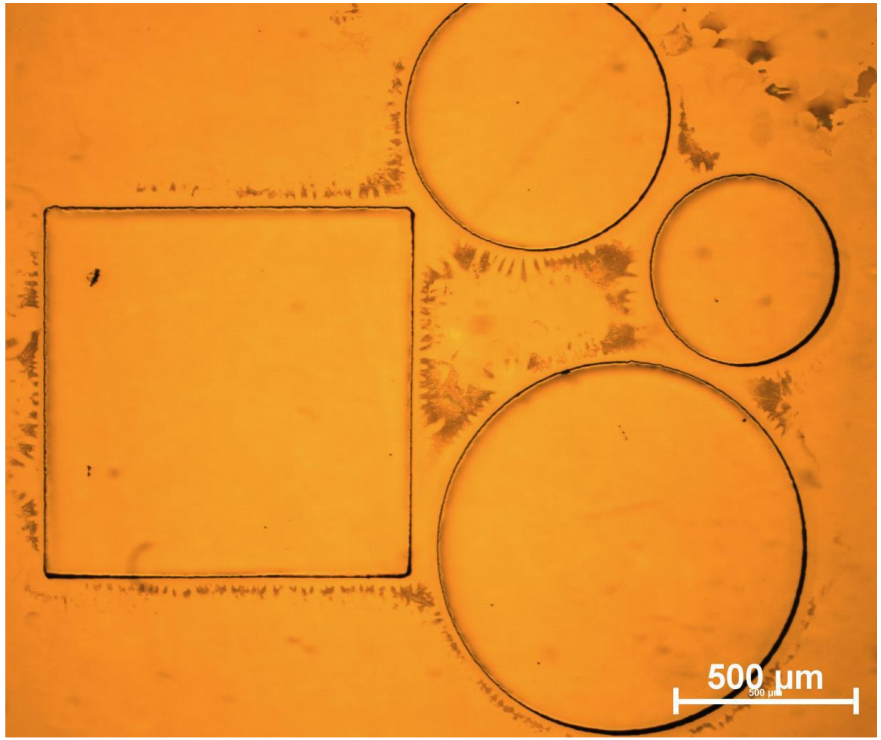
Step height: 8.0 µm Speed: 133 nm/min



Over etch



EVOLUTION OF THE ETCHING : MASK EFFECT

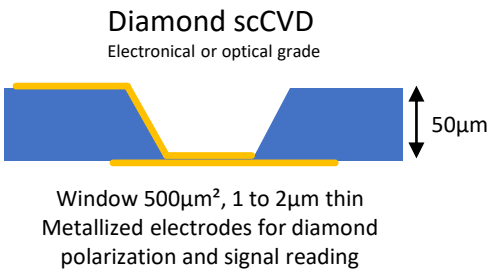


To be continued ….

CONCLUSION ULTRA-FINE DIAMONDS

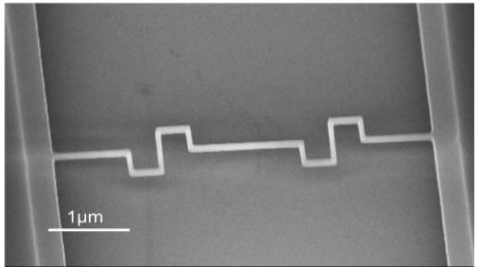
The thinning of the diamond is even less obvious than expected:

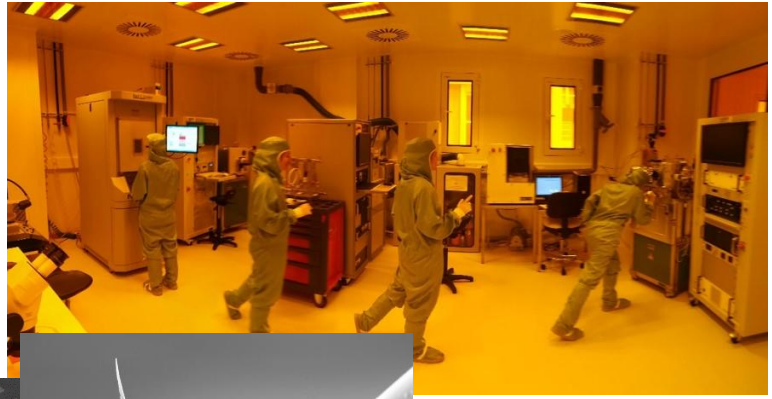
- Surface defects are disruptive to detection and could puncture the window !!!
- A better etching recipe, without oxygen, avoids these problems but requires a much longer etching time.
- The use of a polycrystalline diamond etching mask for the patterns has been validated.

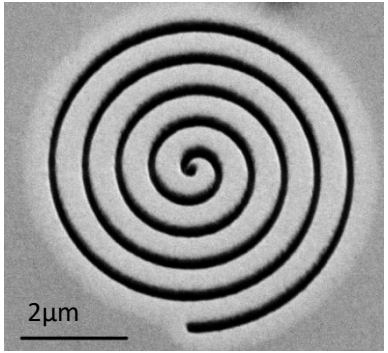


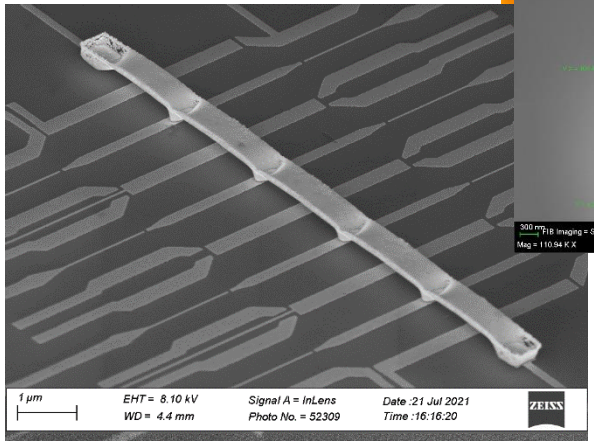
- Production of the first prototype expected in the next few months.
- This was followed by beam tests on the AIFIRA and MIRCOM lines to characterize the detector (thickness, response) and the impact on the beam.

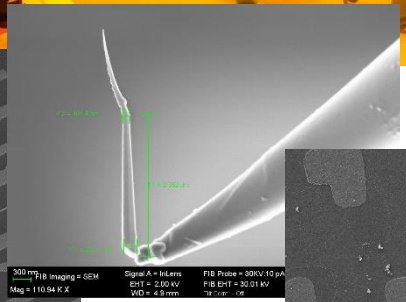
NANOFAB PORTFOLIO

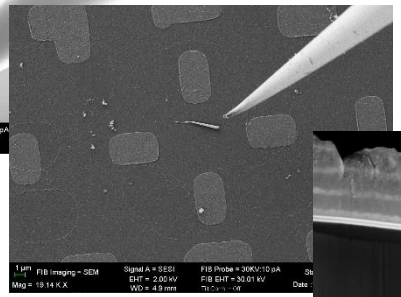


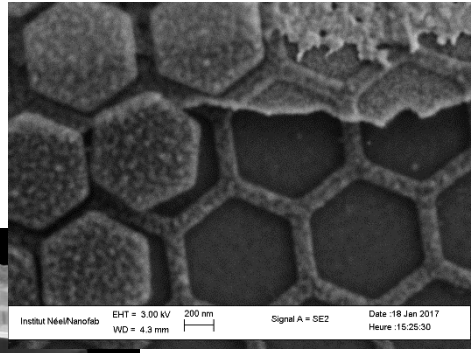


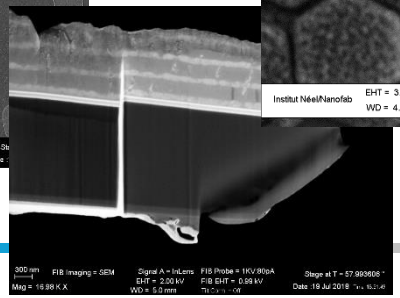












Diamond scCVD

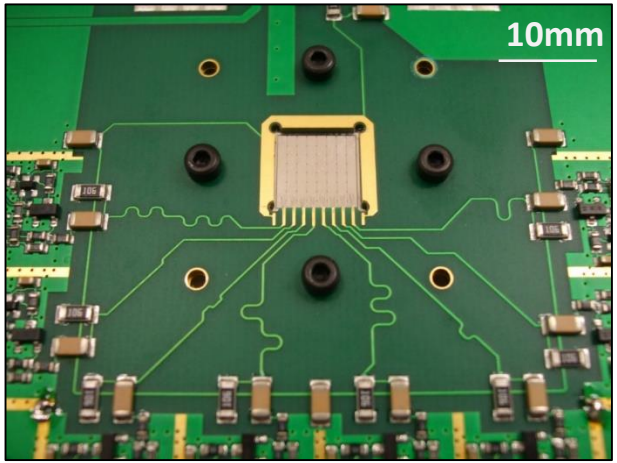
Electronical or optical grade

Window 500µm², 1 to 2µm thin

Metallized electrodes for diamond polarization and signal reading

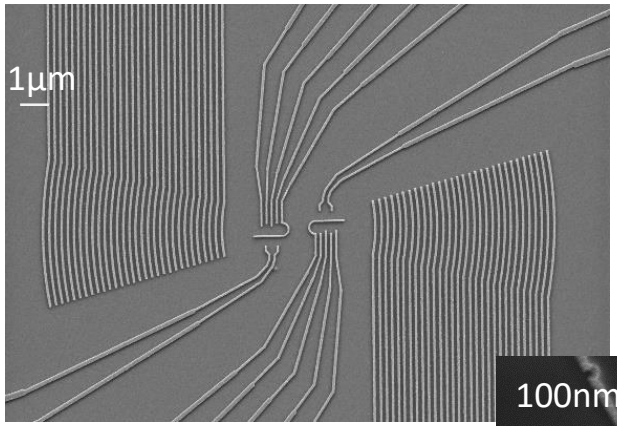
Pourquoi passer du monde Macro au Nano
ex TOP DOWN

Montage sur PCB, diamant 10x10 mm²

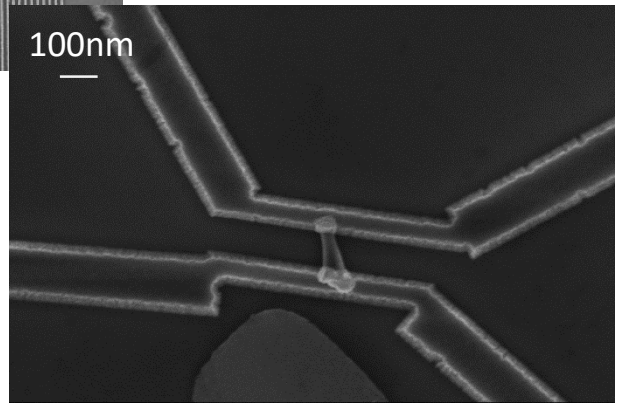


/ 10 000

-Ingénierie quantique : ligne sub µm haute densité /e-beam



- NanoPhysique :
Mesure électrique de nanofil SiC



- Réduire les dimensions du « macromonde »
- Densification des composants actifs

Contexte : développements diamants - activités multidisciplinaires

Physique Médicale

- **Monitorage faisceau en radiothérapies** (position/étiquetage temporel/comptage)
  - → *Grande surface (>1cm²)*
  - → *Fort taux de comptage (100 MHz)*
  - → *Bonne sensibilité et grande dynamique (détection de particules uniques dans des bunchs jusqu'à 10^{10} particules dans des trains pour les thérapies Flash)*

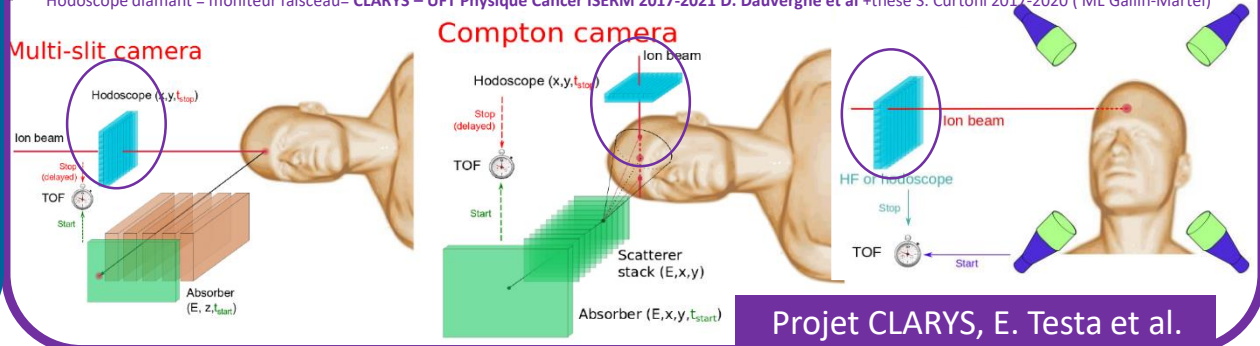
Prompt Gamma Imaging

Thèse O. Allegrini (E. Testa, JM Letang INSA CREATIS)

Prompt Gamma Peak Integral

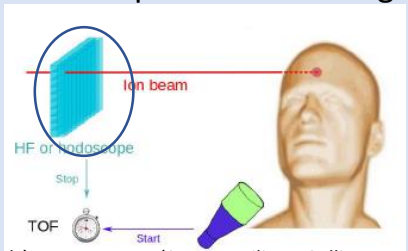
Thèse P. Everaere (ML Gallin-Martel E. Testa JM Letang)

Hodoscope diamant = moniteur faisceau= **CLARYS – UFT Physique Cancer ISERM 2017-2021 D. Dauvergne et al** *thèse S. Curtoni 2017-2020 (ML Gallin-Martel)



Projet CLARYS, E. Testa et al.

Prompt Gamma Timing



Thèse M. Jacquet (S. Marcatili ML Gallin-Martel)

Hodoscope diamant

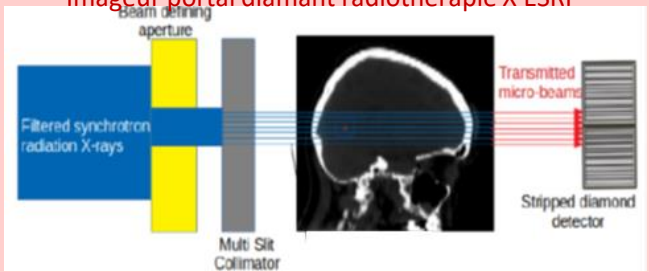
Projet TIARA physique cancer INSERM 2020-2023 S. Marcatili et al.

Projet R&T DIAMTECH ML Gallin Martel (LPSC) A. Guertin (SUBATECH) et al.

WP1 = IDSYNCHRO IN2P3 / UGA - INSERM

IDSYNCHRO J-F Adam et al

Imageur portal diamant radiothérapie X ESRF



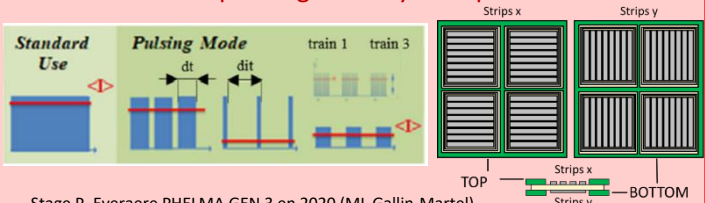
Thèse N. Rosuel 2018-2021 (D. Dauvergne JF Adam UGA-INSERM)

Apprentissage élève ingénieur PHELMA Grenoble INP L. Tribouilloy (L Gallin-Martel)

WP2 = ANR DIAMMONI 2020-2024 LPSC / SUBATECH/ARRONAX

ANR-DIAMMONI ML Gallin-Martel et al.

Moniteur faisceau pulsés grande dynamique en diamant



Stage P. Everaere PHELMA GEN 3 en 2020 (ML Gallin-Martel)

Stage R. Molle PHELMA GEN 3 en 2021 (ML Gallin-Martel)

Thèse X 2021-2024 (LPSC,SUBATECH)