

# ULTRA-THIN MEMBRANE AND 3D DIAMOND DETECTORS: FROM FABRICATION TO APPLICATIONS

Journée thématique: Fabrication de détecteurs semi-conducteurs, Paris 14/06/2017 | Pomorski Michal





## OUTLINE

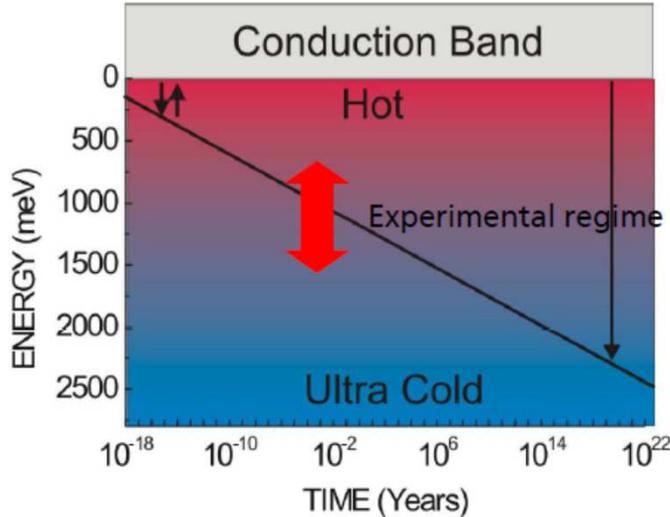
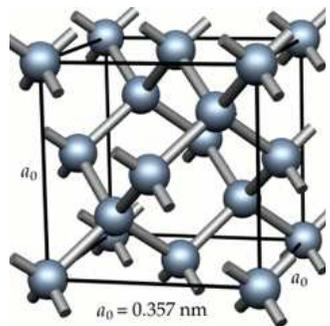
**Intro:** Diamond Physical Properties, Diamond Material

**Manufacturing:** 3D Diamond Detectors, Thin sc membrane detectors

**Applications:**

- x-ray beam monitoring at modern light sources
- external microbeams for radiobiology
- avalanche detectors and CCE recovery
- microdosimetry in hadrontherapy

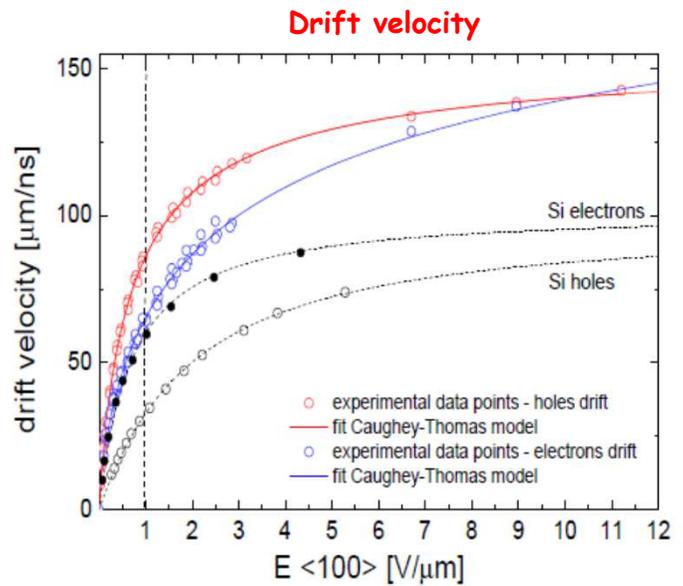
# DIAMOND PHYSICAL PROPERTIES



- low leakage I
- VIS blind
- high. temp
- large bandgap 5.5 eV
- radiation hard
- chemical inertness
- mechanically robust
- high charge drift/mobility
- low dielectric constant

## measured values a wish list

Property	Silicon	Diamond	applications [2, Effect in Diamond]
Band gap [eV]	1.12	5.45	Leakage current is very small and No cooling required
Electron mobility [cm <sup>2</sup> /V-sec]	1450	2200	Signal is very fast
Hole mobility [cm <sup>2</sup> /V-sec]	500	1800	
Dielectric constant	11.9	5.7	Low Capacitance and noise
Atomic displacement energy [eV]	21	43	Radiation hardness is high
Electron-hole pair creation energy [eV]	3.6	13.6	Smaller signal
Mean electron-hole pair by MIP (Minimum Ionizing Particle) in 1 μm	89	36	Smaller signal
Charge collection efficiency [%]	100 (Single crystal)	100% for Single Crystal & 50% for Polycrystalline	Smaller signal
Thermal conductivity [W/cm-K]	1.5	22	No cooling required



# DIAMOND MATERIAL



The largest in the world  
Blue HPHT lab-grown diamonds:  
(unveiled at JCK Show 2016)

Silicon < - > electronics industry  
diamond material ( just started ;) )  
→ high power electronics  
→ quantum applications  
→ jewellery

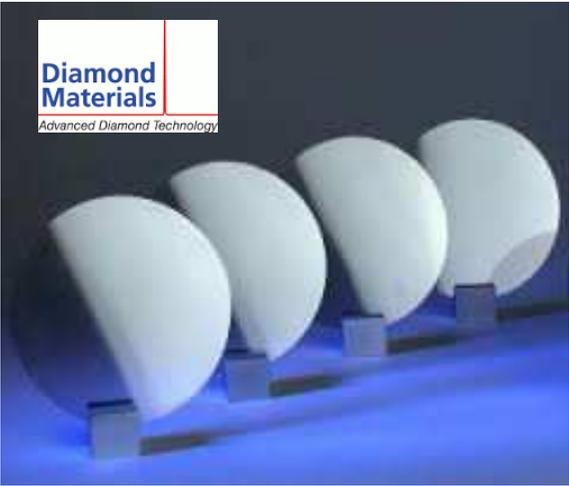
After 15 years still a reference:  
**electronic grade single crystal CVD**



[N] < 1 ppb and [B] < 5ppb

high purity, some dislocations  
CCD > few mm, spectroscopic

**polycrystalline CVD**



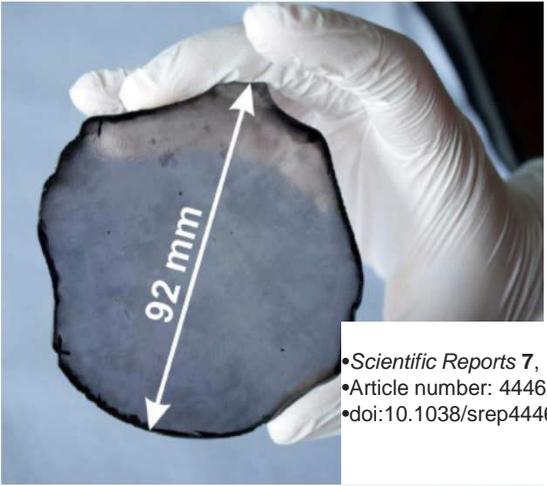
grain boundaries, columnar structure  
CCD~300µm, non-spectroscopic

**single crystal IIa HPHT**



high purity, approaching EG e6  
High CCD, spectroscopic, repro(?)

**single crystal Heteroepitaxy CVD**

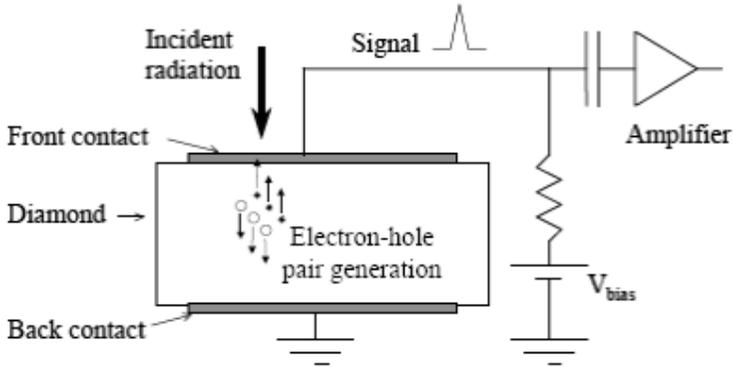


•Scientific Reports 7,  
•Article number: 44462 (2017)  
•doi:10.1038/srep44462

high purity, dislocations  
High CCD, almost spectroscopic, repro(?)

# DIAMOND DETECTORS

## solid state ionization chamber



$$\epsilon \nabla^2 \Psi = -\rho$$

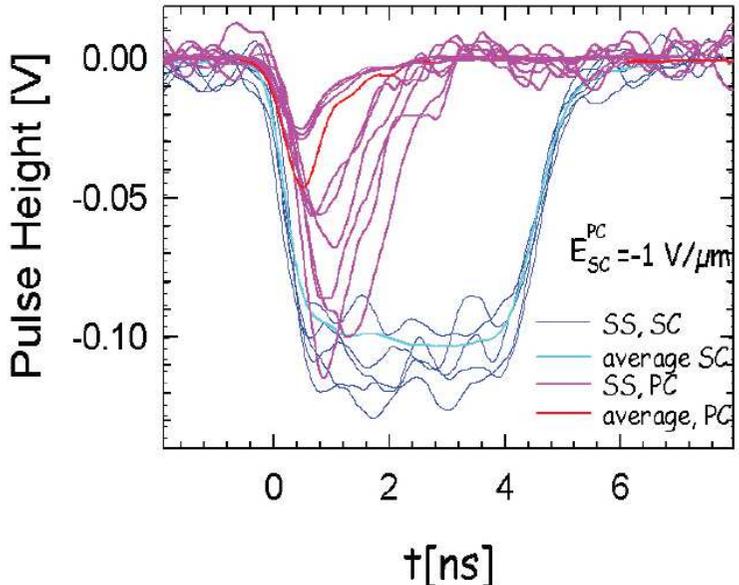
$$q \frac{\partial p}{\partial t} = -\nabla J_p - qR$$

$$q \frac{\partial n}{\partial t} = -\nabla J_n - qR$$



drift-diffusion

## induced current pulses

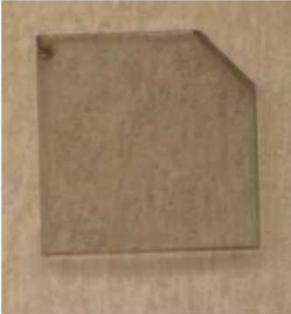


### Surfaces preparation:

- Cleaning+oxidation @ ~300 C
- H2SO4 + KNO3
- Rinsing DI water, IPA
- drying

**SIMPLISSIME**  
 LES RECETTES LES + FACILES DU MONDE  
**4 INGRÉDIENTS 4 GESTES**  
**C'EST PRÊT !**

### raw material



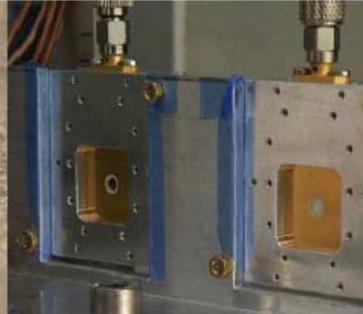
### plated diamond



### Applying contacts

- Sputtering, evaporation:**  
 Al, Cr+Au, Ti+Pt+Au, C,  
 ITO....  
 shadow masks, photolitho

### ready-to-use detector

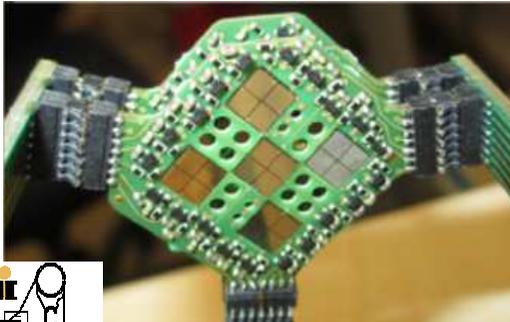


### packaging:

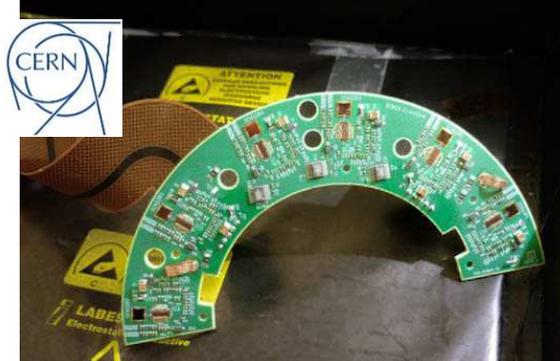
- pcbs
- micro bonding
- housings + connectors

# DIAMOND DETECTORS IN USE

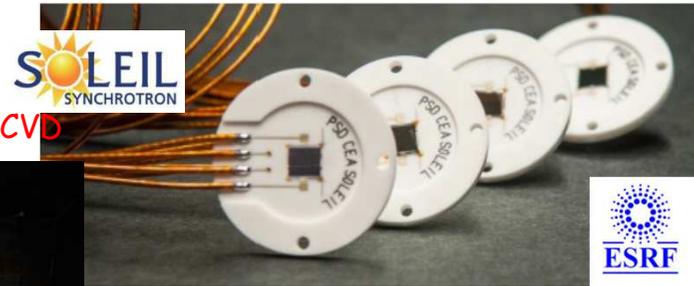
Start detectors scCVD



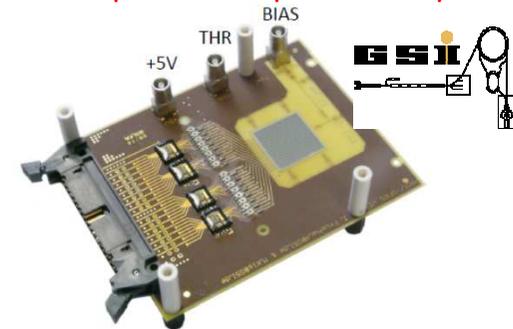
CMS beam condition monitors pcCVD



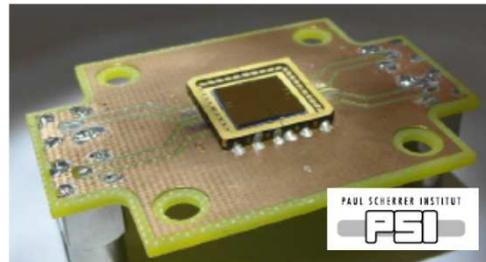
scCVD PSD for X-ray beam monitoring



2x2 cm pcCVD strip ToF heavy ions



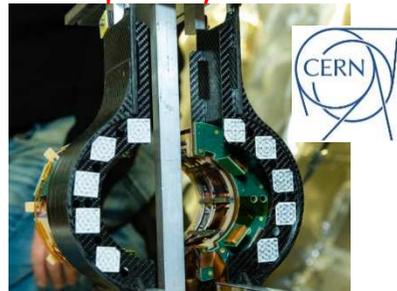
High temp  $\alpha$ -particles spectroscopy of SHE



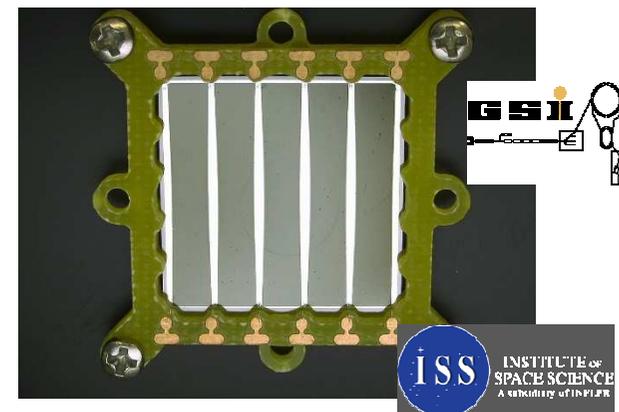
mosaic scCVD fast n detector



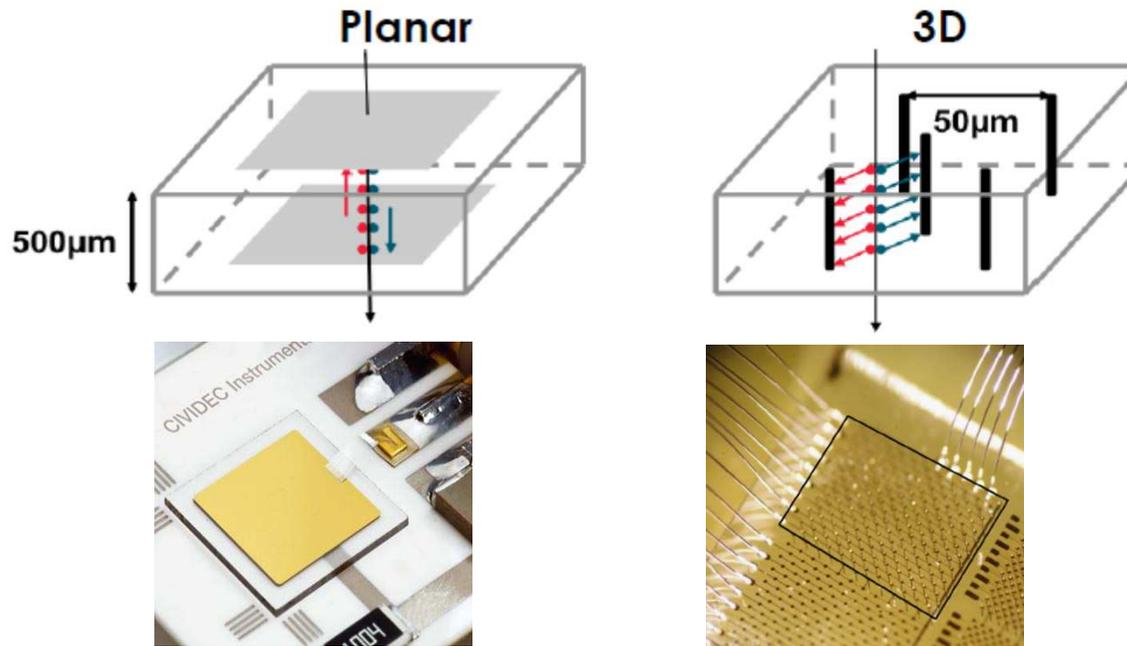
CMS PLT pixel system scCVD



2x2 cm pcCVD PSD particle beam monitoring



## 3D DIAMOND DETECTORS – THE CONCEPT



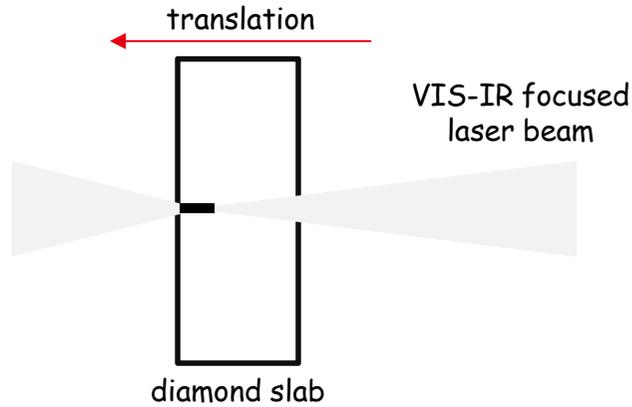
3D geometry  $\rightarrow$  same generated charge but much shorter drift path:

- more radiation hard (as detector)
  - fast signals
  - lower bias / higher E field
- better performance for lower quality materials

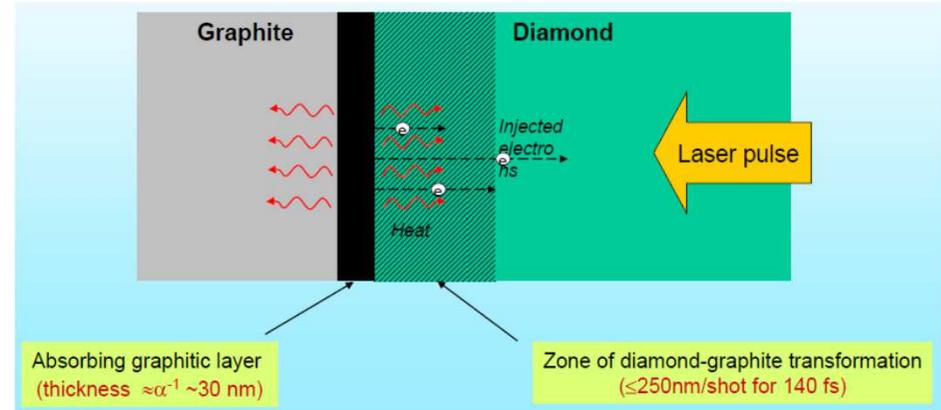
Good for: **HE physics, hadron therapy, fast neutron detection...**

# 3D DIAMOND DETECTORS – FABRICATION

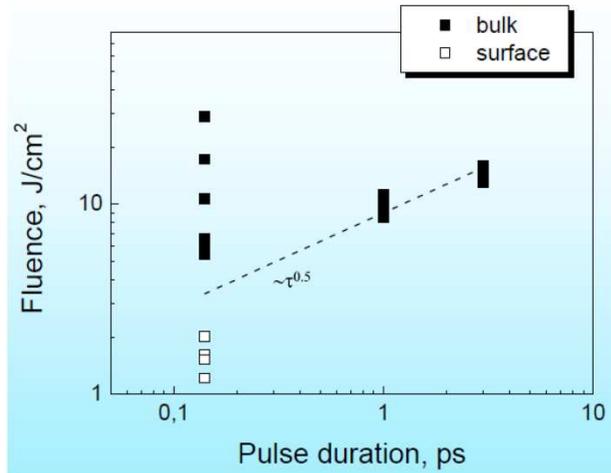
Laser writing of graphitic patterns



optical breakdown and graphitic front propagation



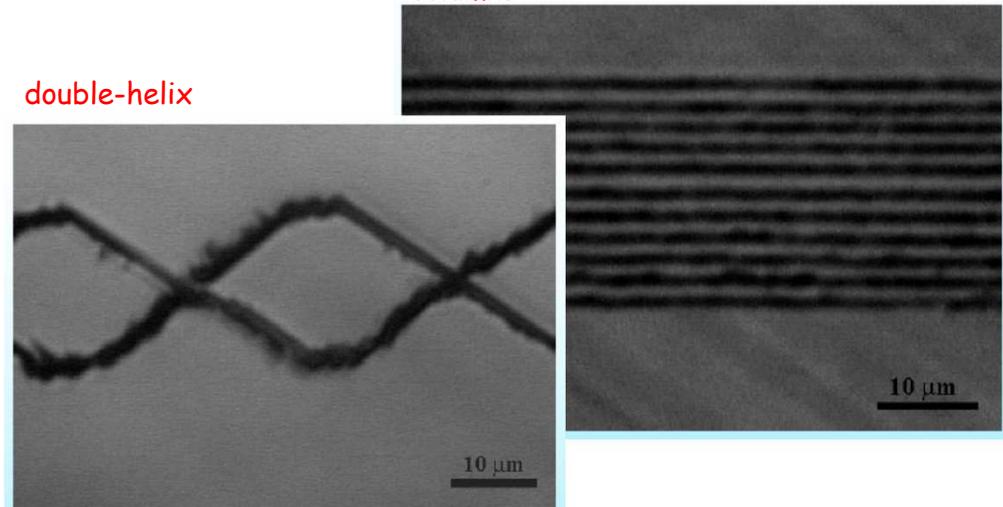
optical breakdown in diamond



typical energies 100-500 nJ/pulse  
(a fraction of available mJ)

columns

double-helix



All work here: T.V Kononenko et al

motivation: photonic applications

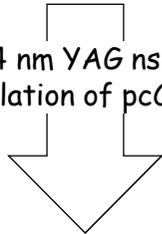
# 3D DIAMOND DETECTORS – SOME HISTORY

2010

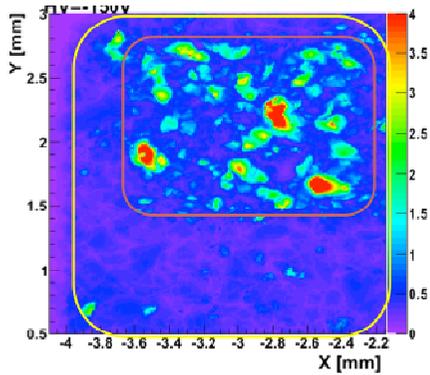
2014



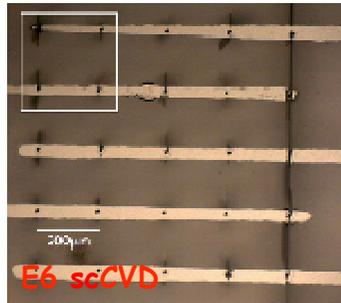
1064 nm YAG ns laser ablation of pcCVD



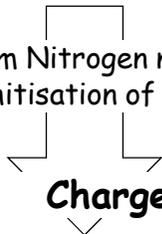
XBIC @ Diamond Source UK



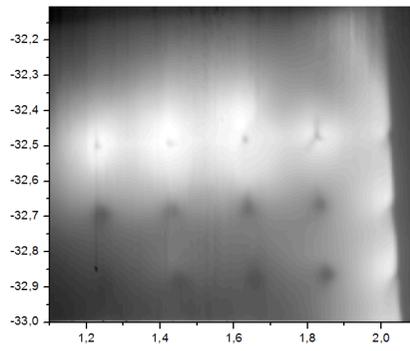
First evidence of enhanced CCE in pcCVD



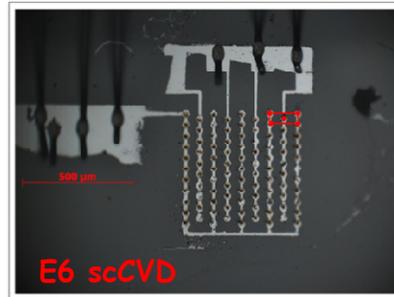
337 nm Nitrogen ns laser graphitisation of scCVD



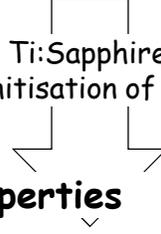
XBIC @ Diamond Source UK



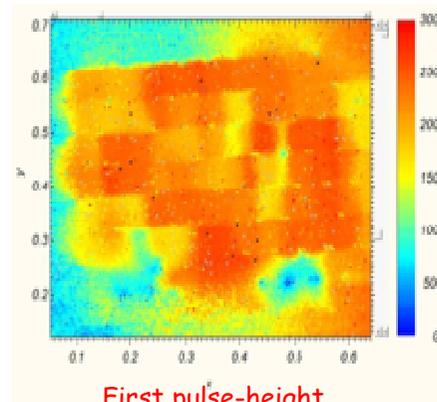
Alexander Oh, Benoit Caylar, Michal Pomorski, Thorsten Wengler, *Diam. And Relat. Mat* (2013)  
doi: 10.1016/j.diamond.2013.06.003



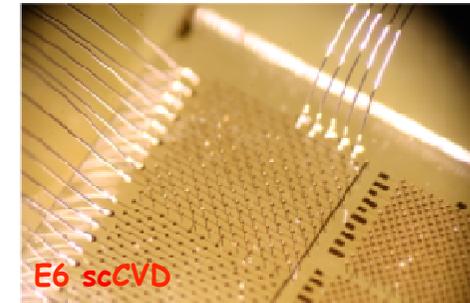
800 nm Ti:Sapphire fs laser graphitisation of scCVD



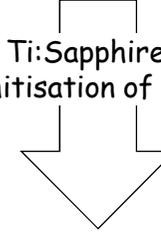
IBIC @ RBI, Zagreb



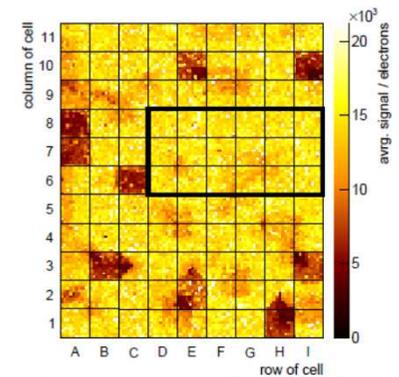
First pulse-height Mapping of 3D structure



800 nm Ti:Sapphire fs laser graphitisation of scCVD



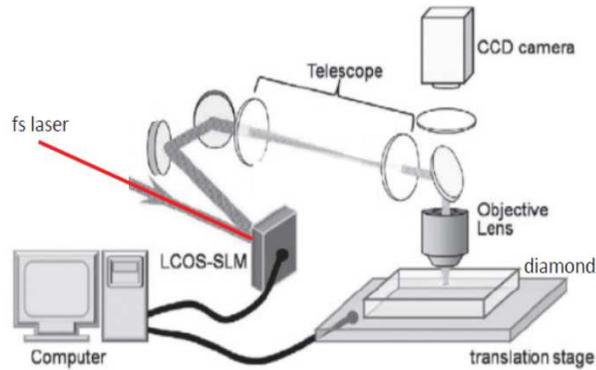
160 GeV p @ CERN



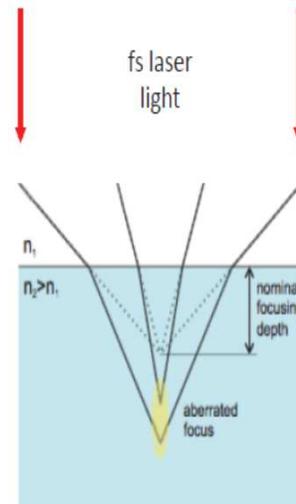
F. Bachmair et al., *NIM A* (2015)  
<http://dx.doi.org/10.1016/j.nima.2015.03.033>

# 3DDD FABRICATION – STATE OF THE ART

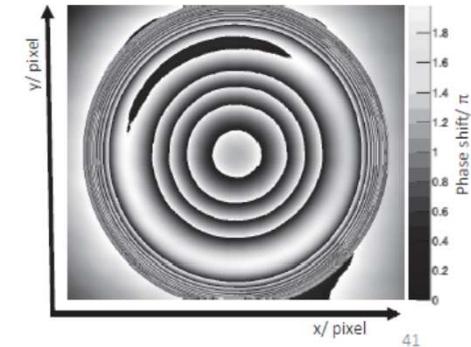
Current laser set-up at UManchester



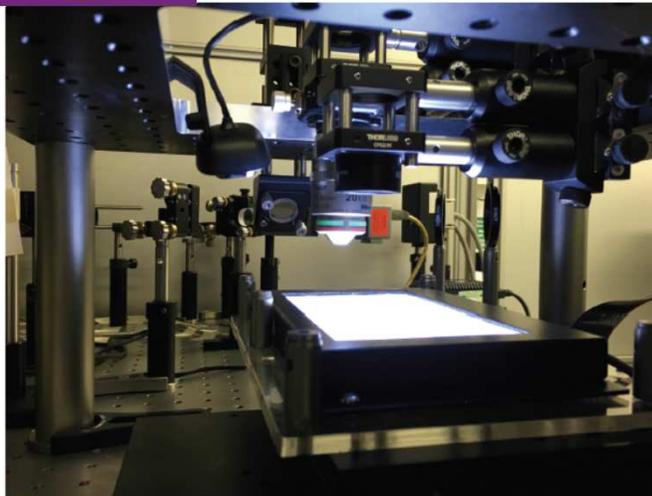
aberration correction



Spatial light modulator



Drilling efficiency (consistent)	~ 100 %
Resistivity	~ 0.1 Ωcm
Diameter	~ 1 μm



Still single column processing (~30s) but:

2D parallel fabrication

3D parallel fabrication

top view, lithium niobate

side view, fused silica

with SA corr

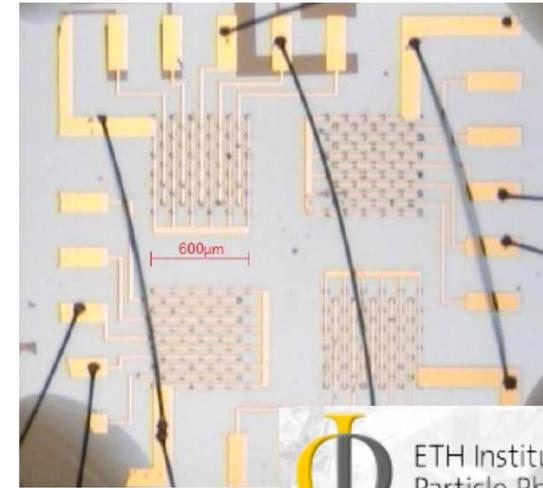
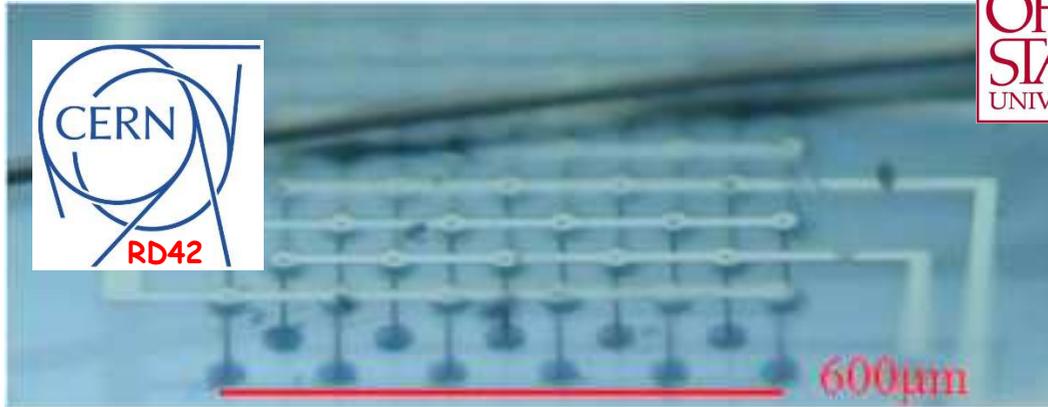
without SA corr

27 September 2010 / Vol. 18,  
No. 20 /  
OPTICS EXPRESS 21090

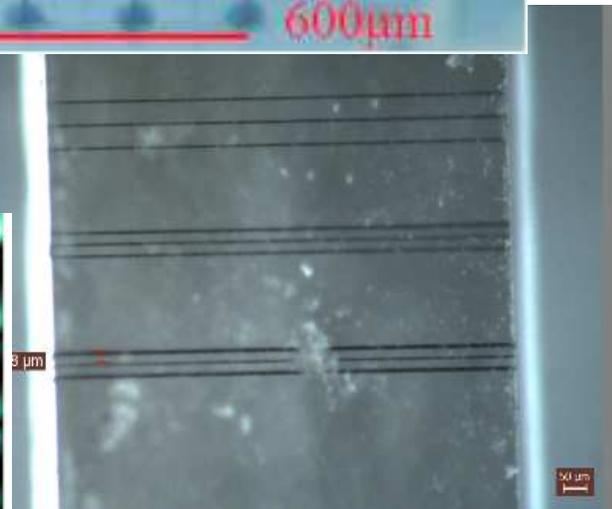
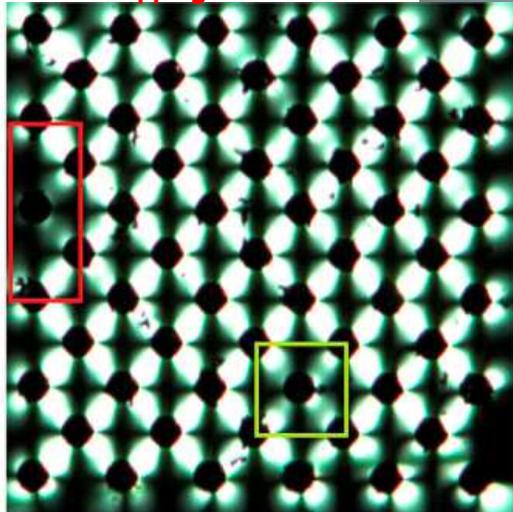
typical energies 100-500 nJ/pulse  
(a fraction of available mJ)

All content here:  
<http://www-adamas.gsi.de/ADAMAS05/talks/Houghton.pdf>

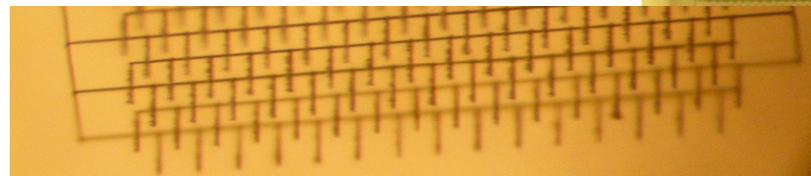
# 3DDD – STATE OF THE ART



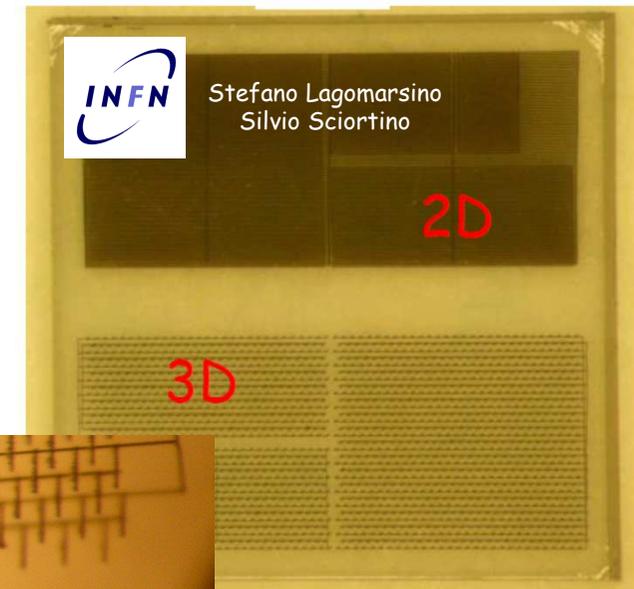
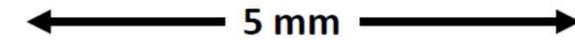
Strain mapping



All-carbon 3DDD



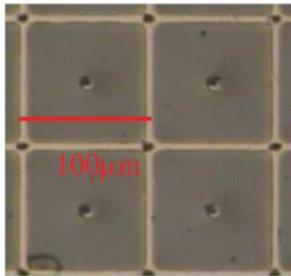
All-carbon 3DDD



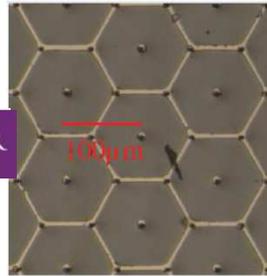
# 3DDD – PERFORMANCES – STATE OF THE ART



4.5 MeV proton microbeam CCE mapping  
@ IRB, Zagreb

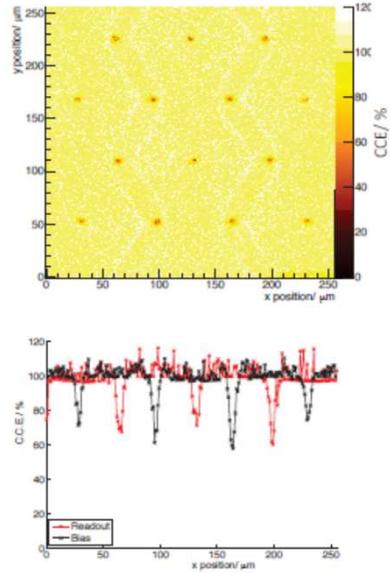
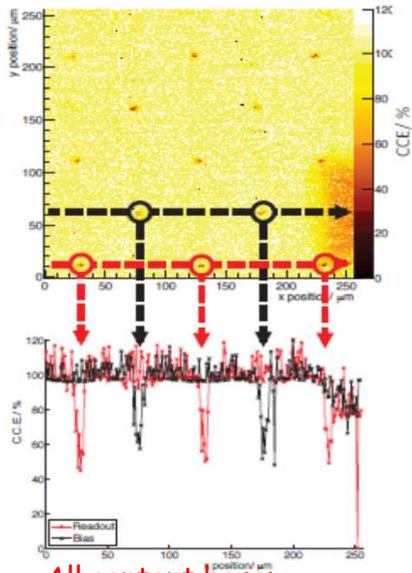


MANCHESTER  
1824



Square

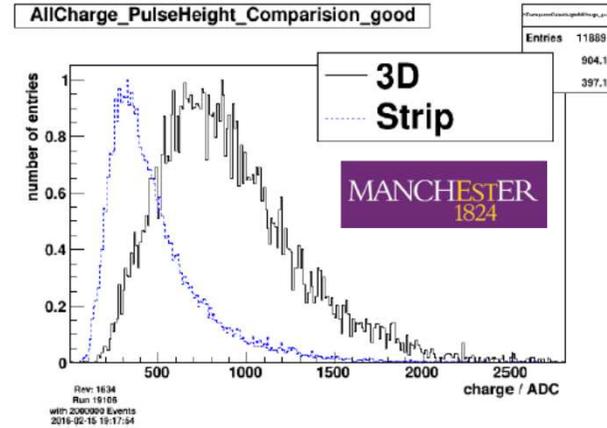
Hexagonal



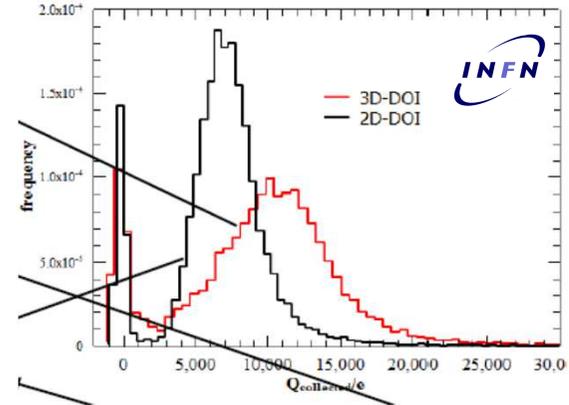
All content here:

<http://www-adamas.gsi.de/ADAMAS05/talks/Houghton.pdf>

pcCVD diamond - mip pulse-height

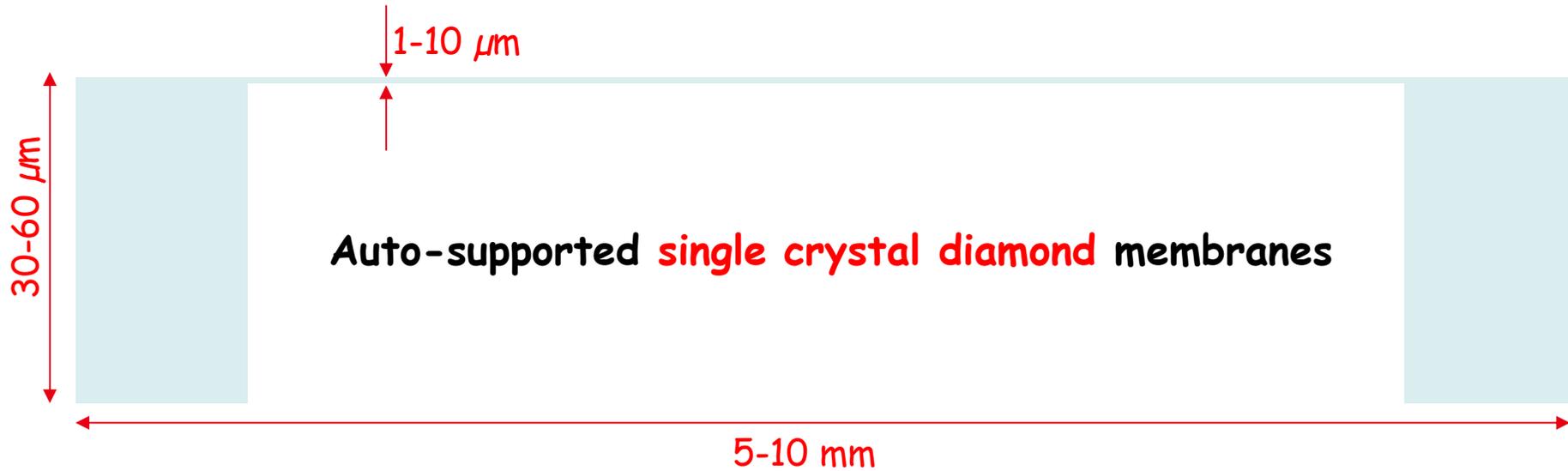


DOI diamond - mip pulse-height



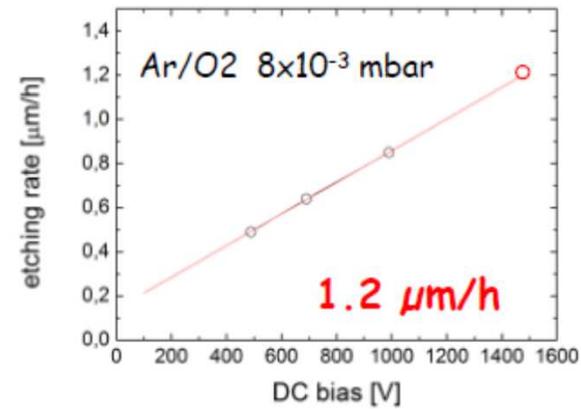
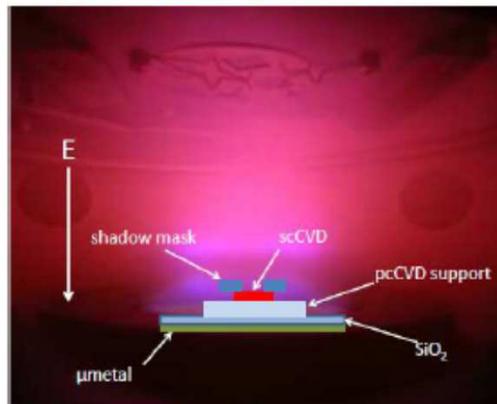
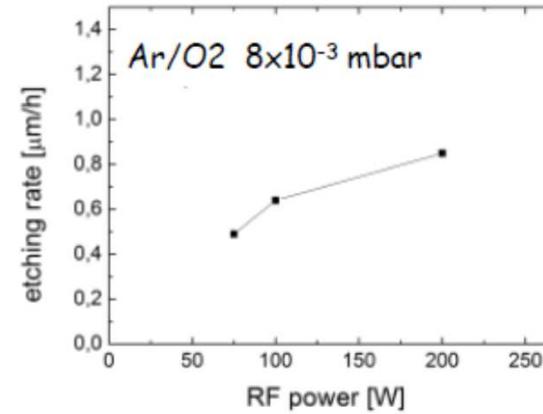
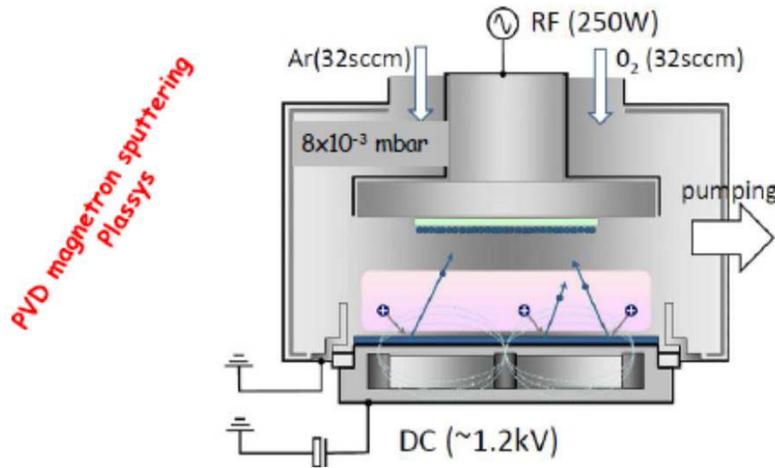
- + performance in HE particle beams
- + radiation hardness data
- + first 3D diamond pixel detector (CMS)
- + first trials as medical dosimeter

# ULTRA-THIN SCCVD MEMBRANE DETECTORS



# ULTRA-THIN SCCVD MEMBRANE DETECTORS

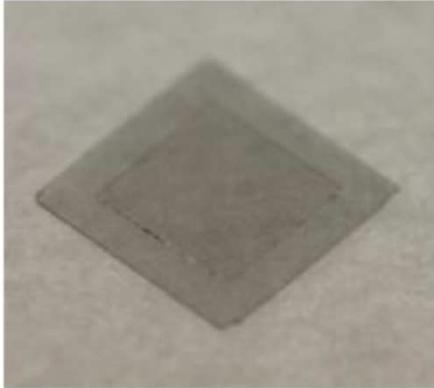
Ar/O plasma deep etching of diamond



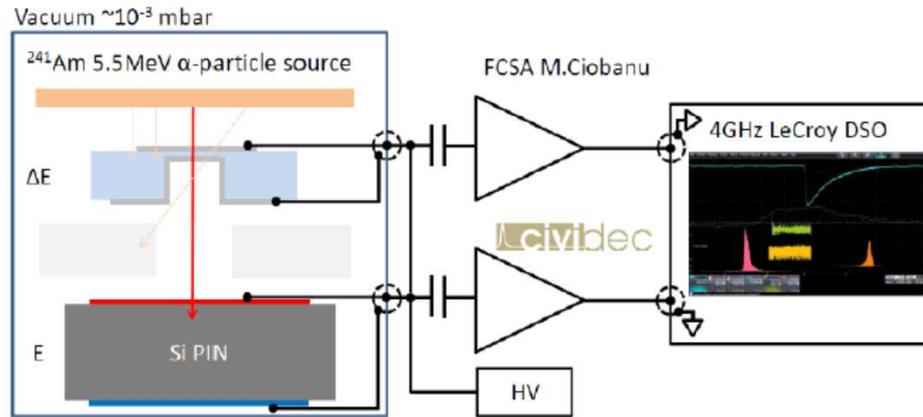
# ULTRA-THIN SCCVD MEMBRANE DETECTORS

characterization of charge transport properties

non-electronic grade e6 scCVD

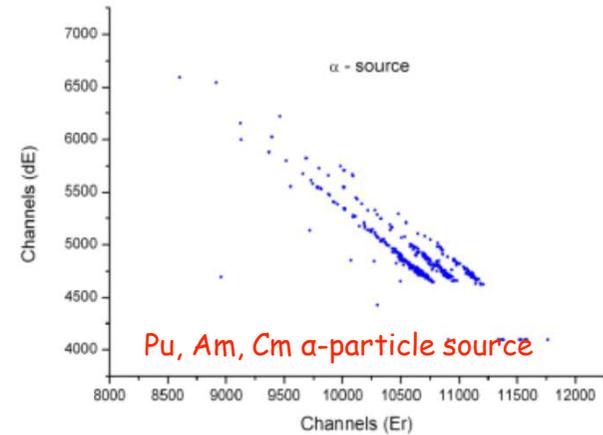
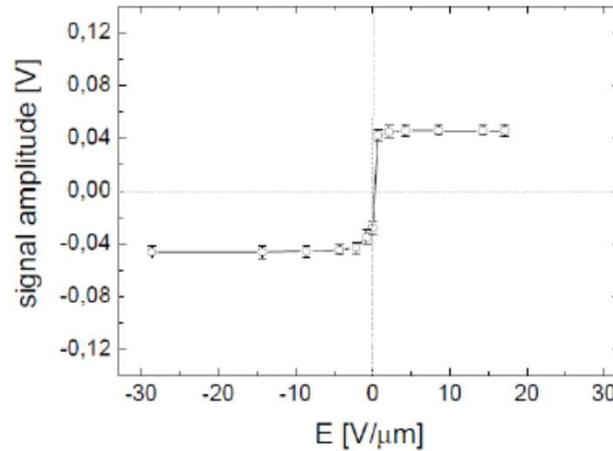
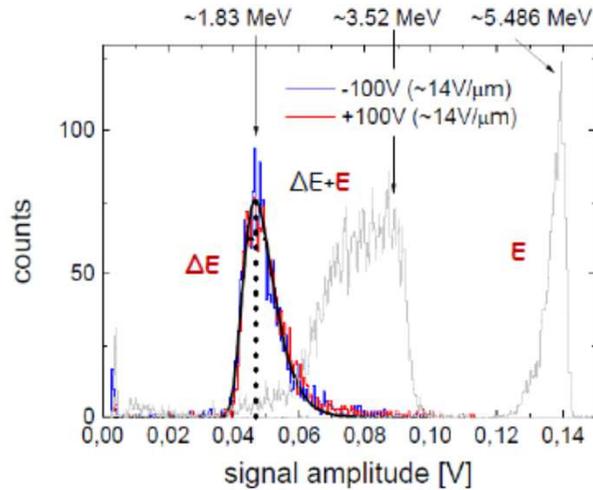


[N] ~ 1 ppm



full CCE

Spectroscopic  $\Delta E + E$

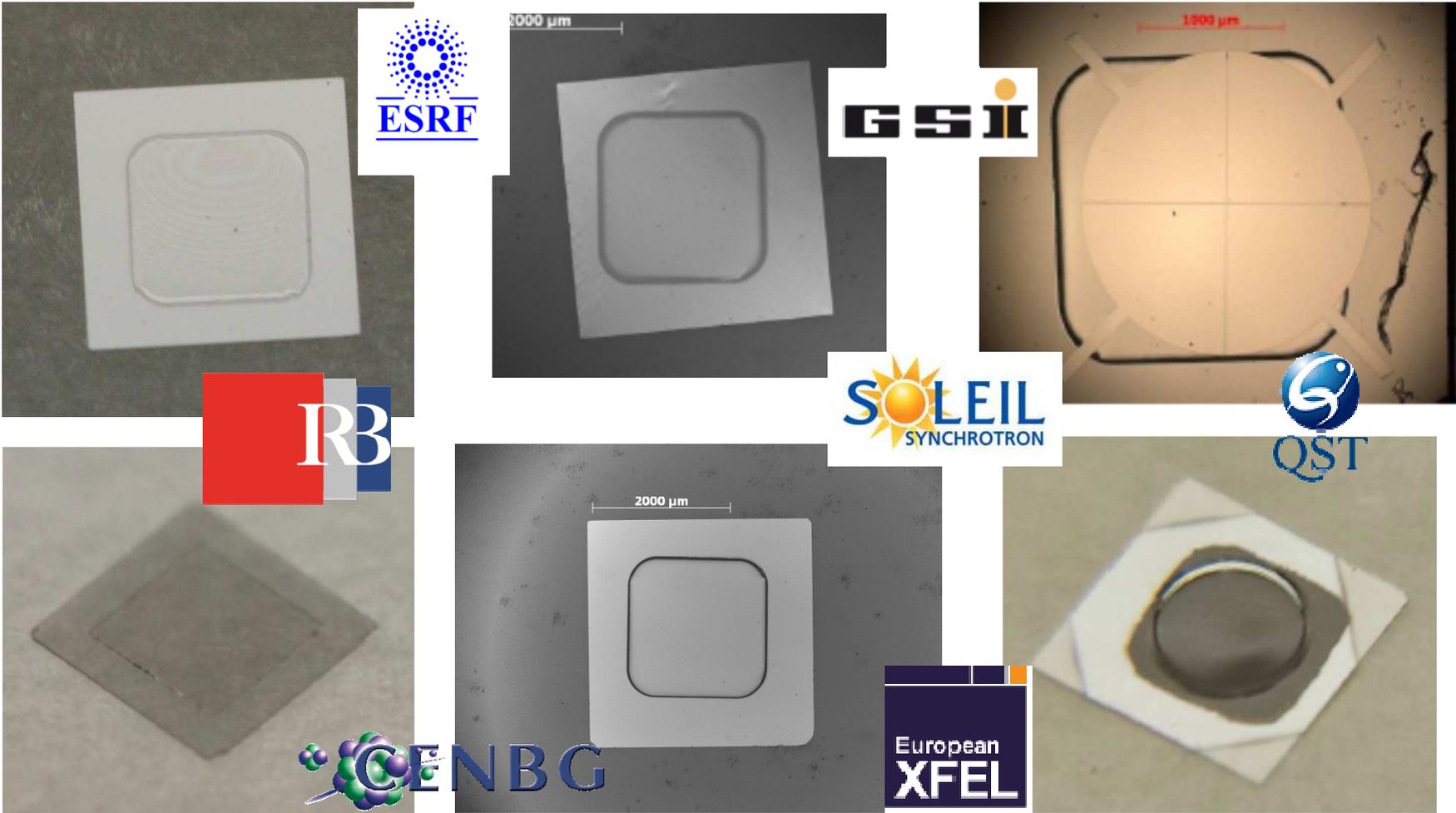


M. Pomorski, B. Caylar, P. Bergonzo, Appl. Phys. Lett 103, 112106 (2013)

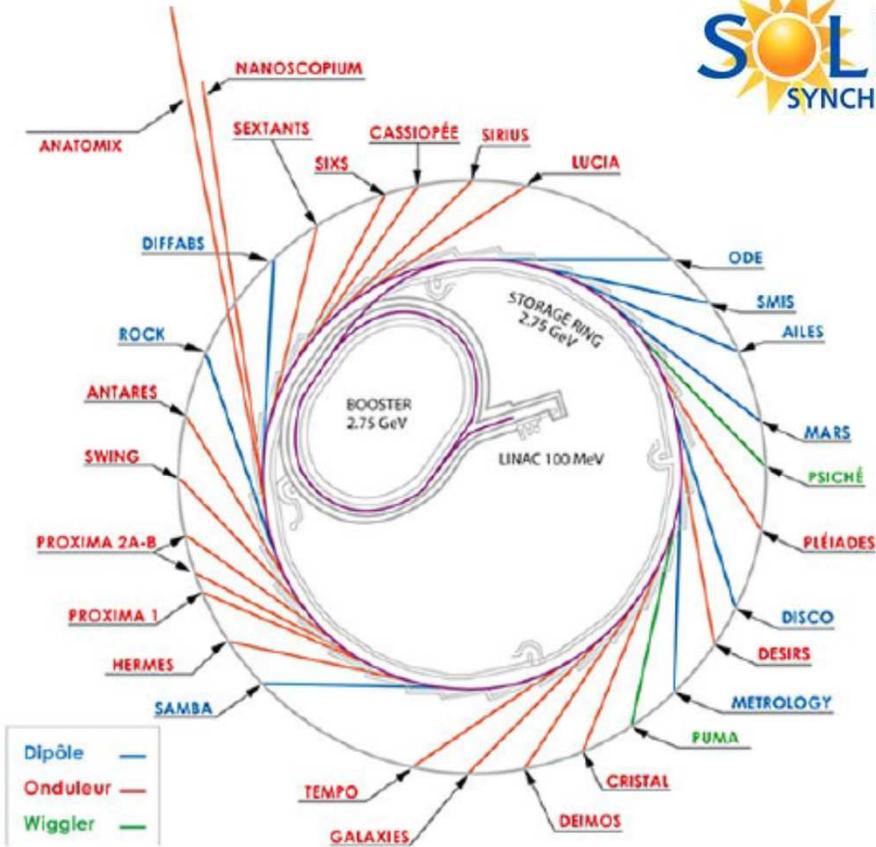
courtesy Beliuskina Olga, M. Traeger (BSI, Germany)

# ULTRA-THIN SCCVD MEMBRANE DETECTORS

some examples of etched scCVD membranes (3-8  $\mu\text{m}$  thick)



# X-RAY BEAM MONITORING AT MODERN LIGHT SOURCES



## X-ray beam monitoring requirements

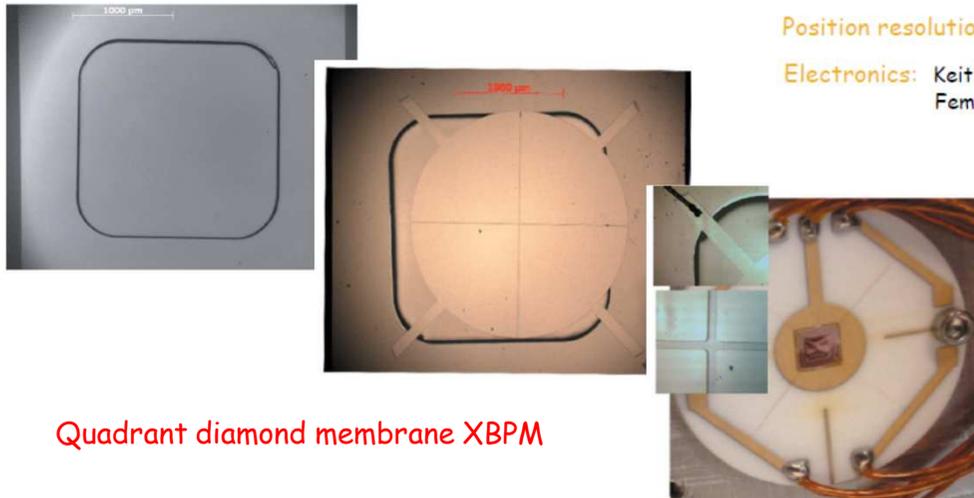
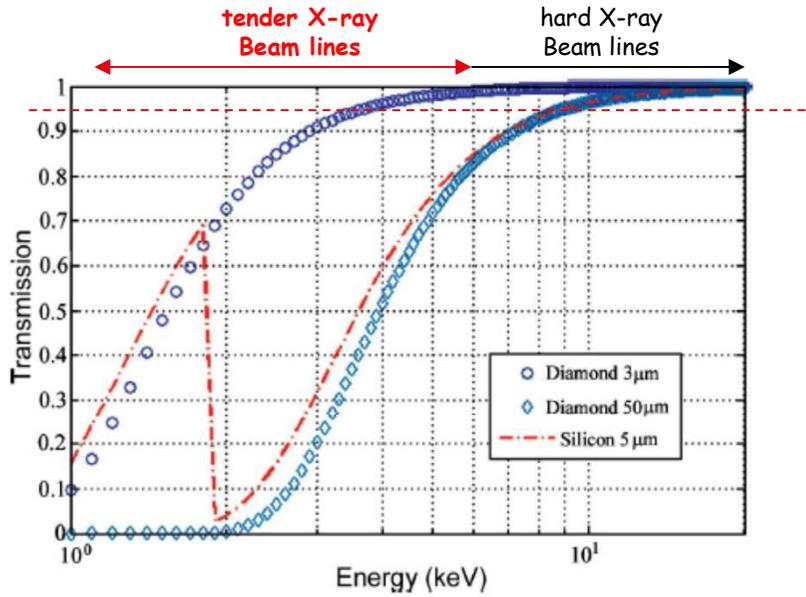
- **position:**  
Beam stability 10% of its size  $\sim 10\text{nm}-100\ \mu\text{m}$  @ kHz
- **intensity:** accuracy and linearity  $< 0.1\%$
- **device:** minimal beam interference, compact,
  - easy maintenance

## Diamond

- Z=6 low x-ray absorption, no leakage current, compact  
Highest thermal conductivity  $\rightarrow$  white beams, XFELs

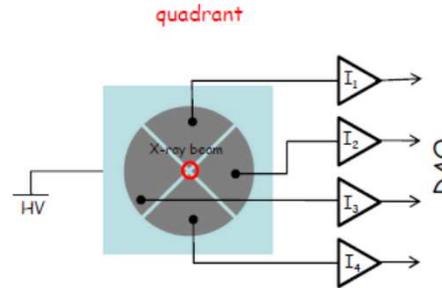
29 beamlines





Quadrant diamond membrane XBPM

## Transparent in-beam device



Beam intensity  $I_0$ :

$$I_0 = I_1 + I_2 + I_3 + I_4$$

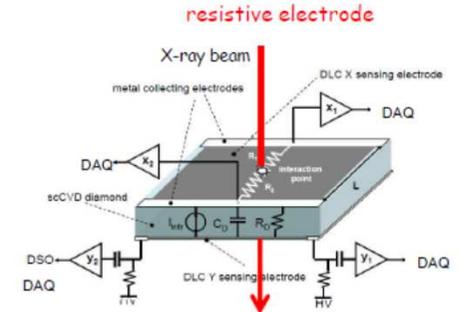
Beam position - 'center-of-mass' (difference/sum\*scale factor)

$$X = C * [(I_2 + I_3) - (I_1 + I_4)] / I_0$$

$$Y = C * [(I_3 + I_4) - (I_1 + I_2)] / I_0$$

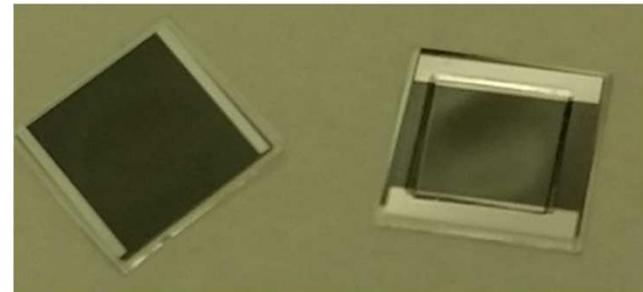
Position resolution:  $\sim I_0 \sim 1/\sigma_{ele(I)} \sim 1/L$

Electronics: Keithley electrometers R&D (100ms), existing XBPM electronics (LoCuM, Oxford, Libera ..), Femto fast amplifiers (pulse mode)..... diamond dedicated coming soon (?).....



$$I_0 = A(x_1) + A(x_2) \text{ or } A(y_1) + A(y_2)$$

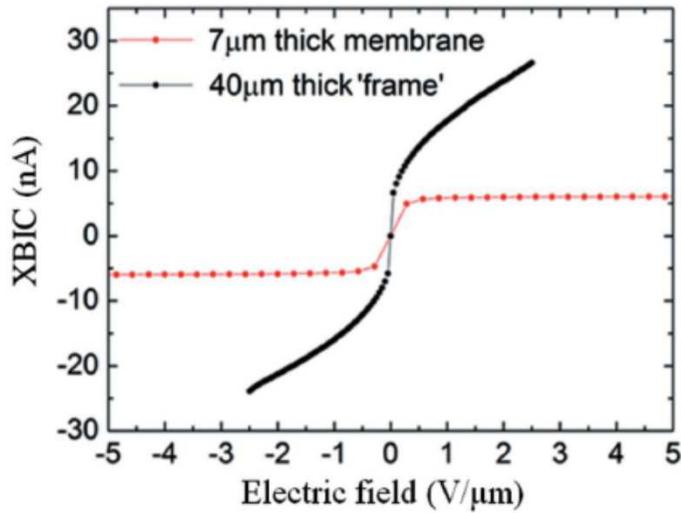
## Resistive electrodes diamond membrane PSD



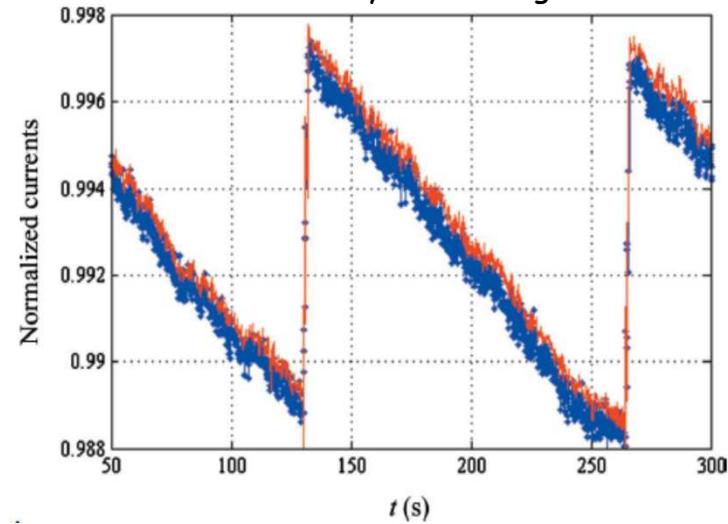
# X-RAY BEAM MONITORING – MEMBRANES PERFORMANCE

SIRIUS beam line, 4keV, flux  $2 \times 10^{12}$  photons/s,  $0.5 \times 0.4$  mm beamspot

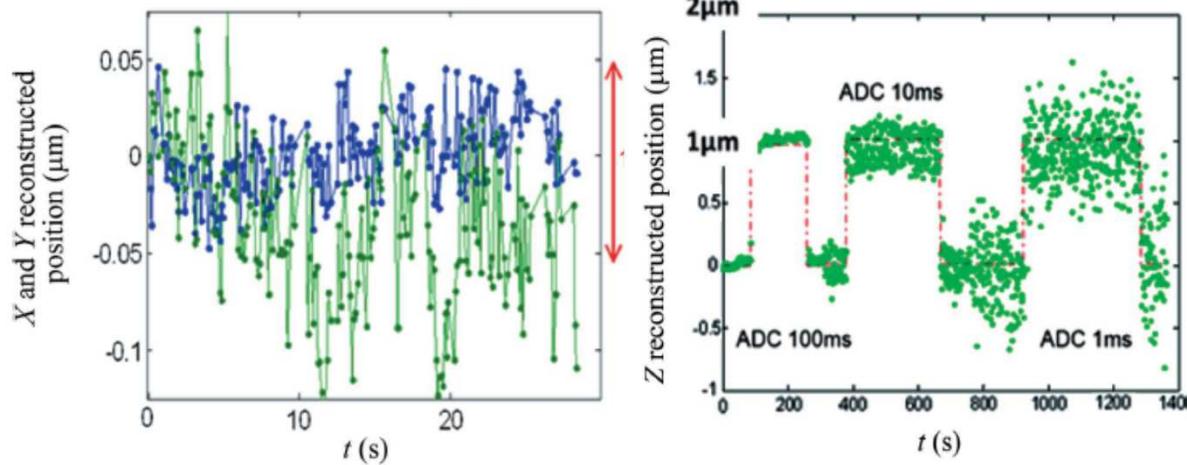
XBIC vs. detector bias



intensity monitoring



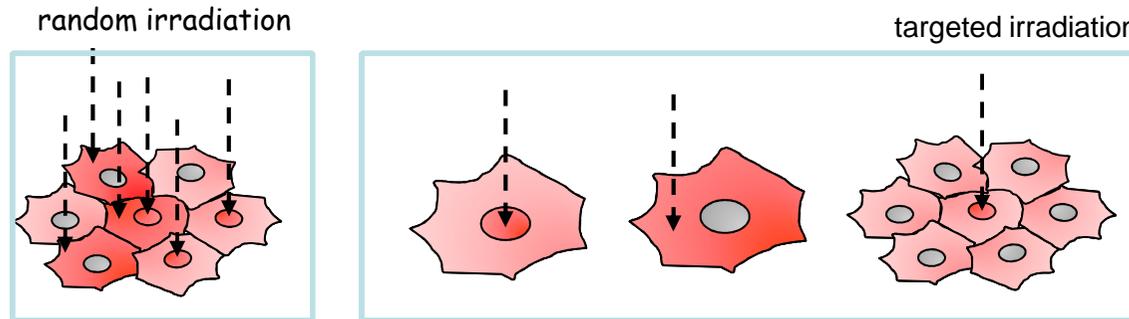
position resolution capability



Kewin Desjardins, M. Pomorski, J. Morse, J. Synchrotron Rad. (2014) 21

## RADIOBIOLOGY – EXTERNAL MICROBEAMS

Localized molecular damage in cells with focused / collimated radiation  
(lasers, UV microspots, X-rays, **charged-particles** - protons,  $\alpha$ , HI)



### Study of radiation effects at sub-cellular level:

- Visualization of the cellular/molecular response
- Dynamical study of the cellular response
- DNA damage and repair kinetics

### Information on:

oncogenic transformation, micronuclei formation, genetic instability  
low dose effects, micro-tracks models → hadron therapy

# RADIOBIOLOGY – EXTERNAL MICROBEAMS

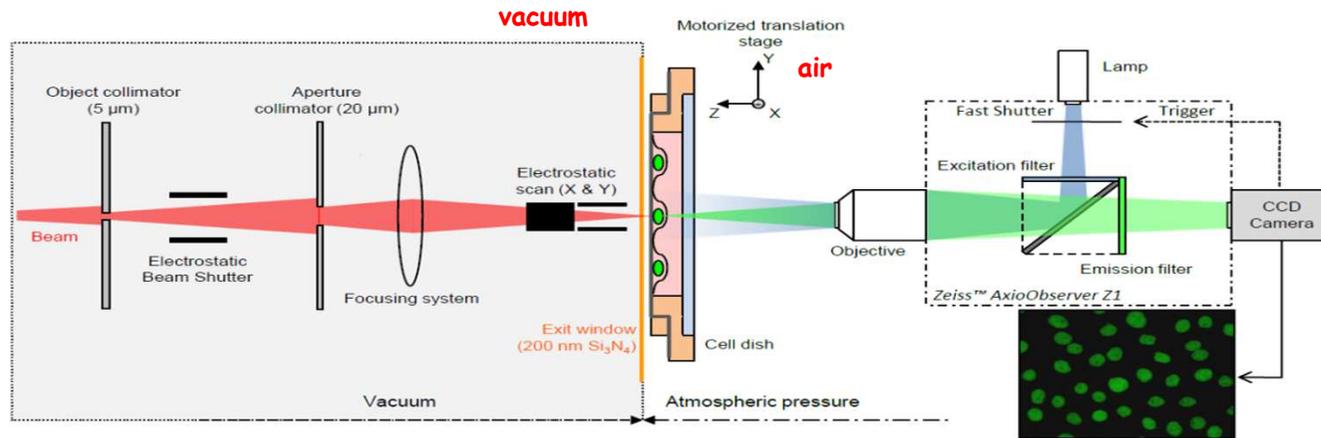
Radiobiology micro-beam line @ AIFIRA Facility, 3.5MV singletron ,CENBG, Bordeaux



## Micro-beam

Focusing <math>1\mu\text{m}</math> (fwhm)

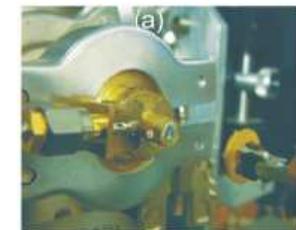
Electrostatic scanning:  
-high throughput (<math>100\mu\text{s}</math>)  
- few  $\text{mm}^2$  scan  
-geometrical patterns



... Precise information about the dose (or number of ions) is required ...



- statistics:  $N \rightarrow \text{error } \sqrt{N}$ , ok for 1000 ions
  - $\rightarrow$  impossible for single particle irradiation.
- thin transmission detectors  
(thin plastic scintillators, thin semi-conductors, gas detectors)  
*those are too thick for MeV  $\alpha$ -particles ...*

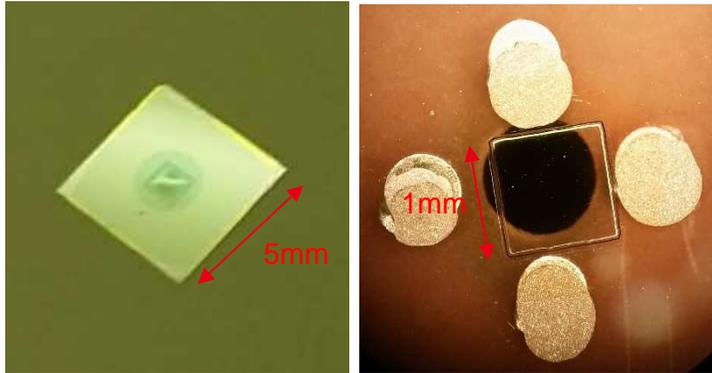


An ultra-thin diamond membrane as a transmission particle detector and vacuum window for external microbeams

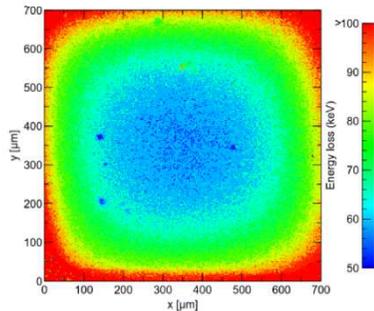
V. Grilj, N. Skukan, M. Pomorski, W. Kada, N. Iwamoto et al.

Appl. Phys. Lett. 103, 243106 (2013); doi: 10.1063/1.4833236

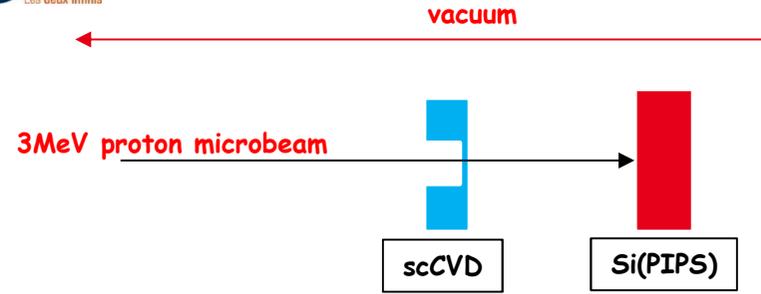
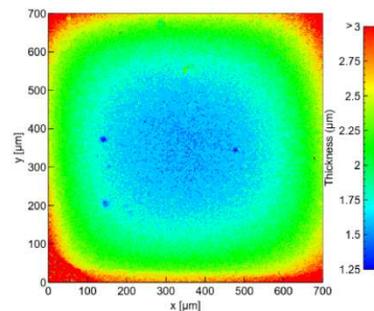
2.8  $\mu\text{m}$  scCVD membrane with ITO contacts as active VIS transparent vacuum window



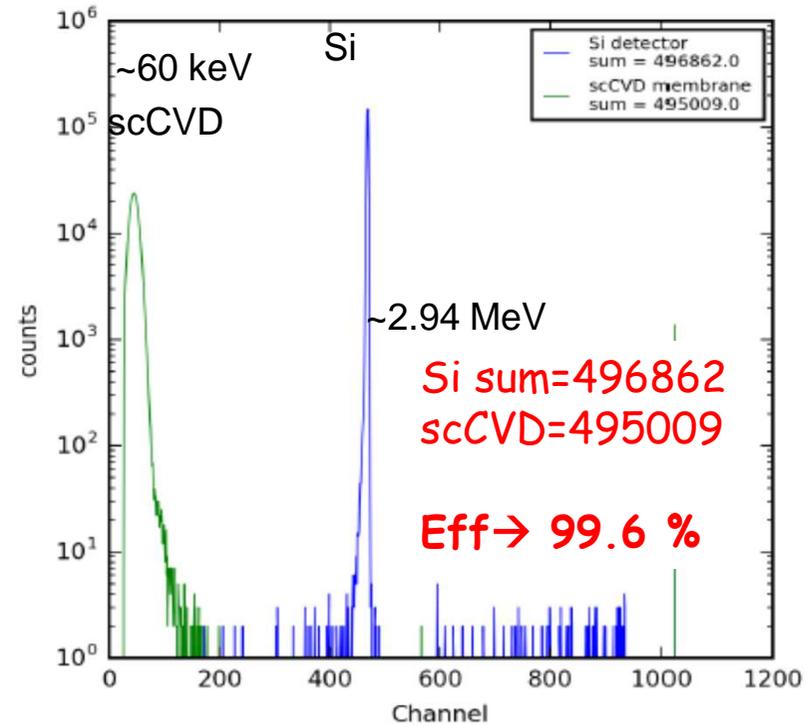
3MeV p pulse- height



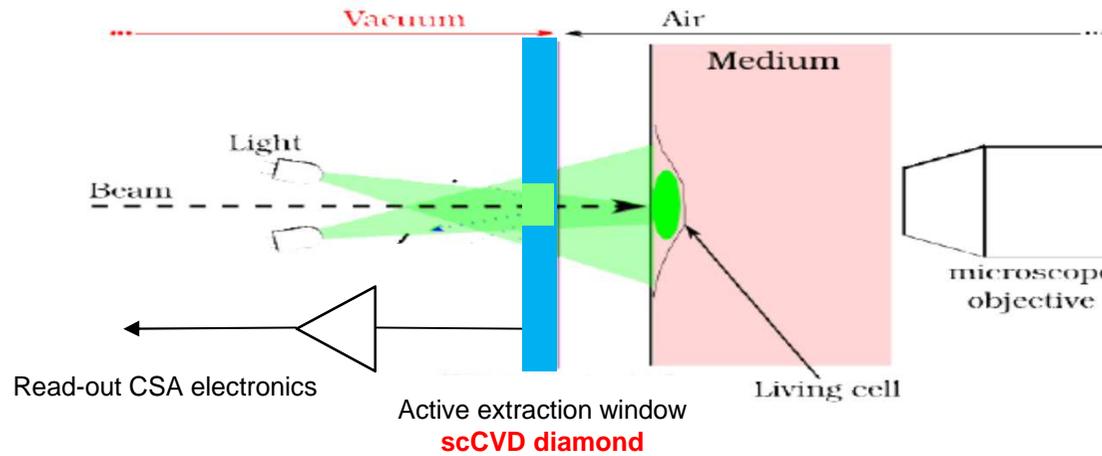
membrane thickness



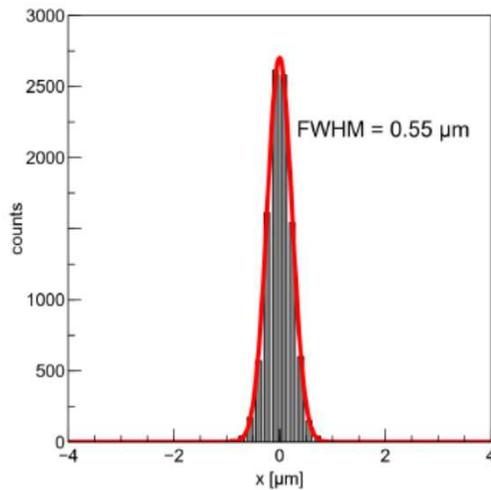
scCVD membrane efficiency vs Si detector



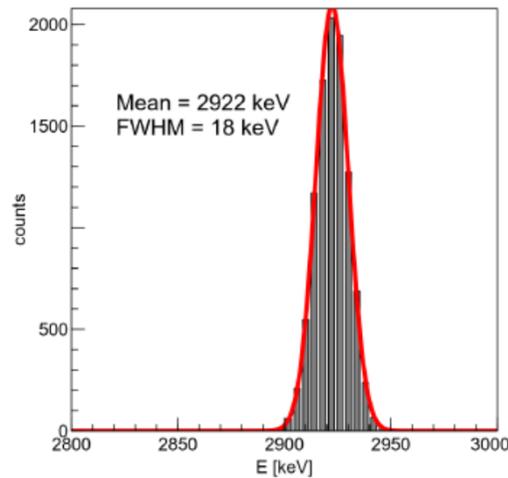
# RADIOBIOLOGY – FIRST IRRADIATIONS



External 3 MeV p beam properties after  
3 μm diamond active window @ 100 μm distance

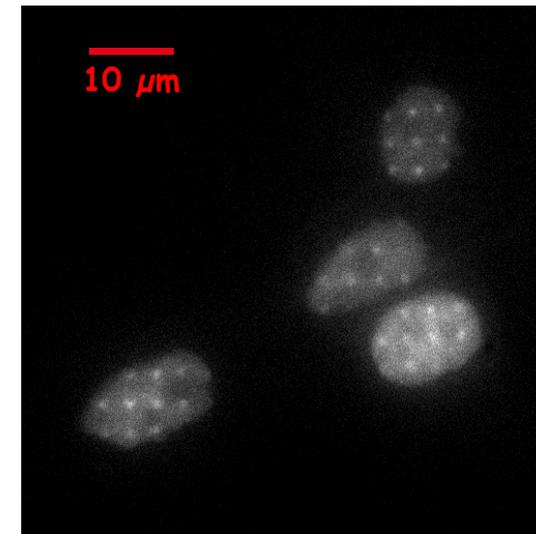


Position straggling



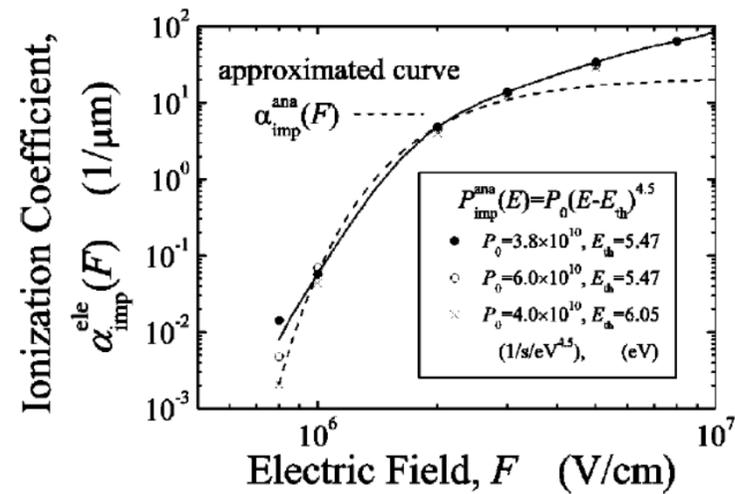
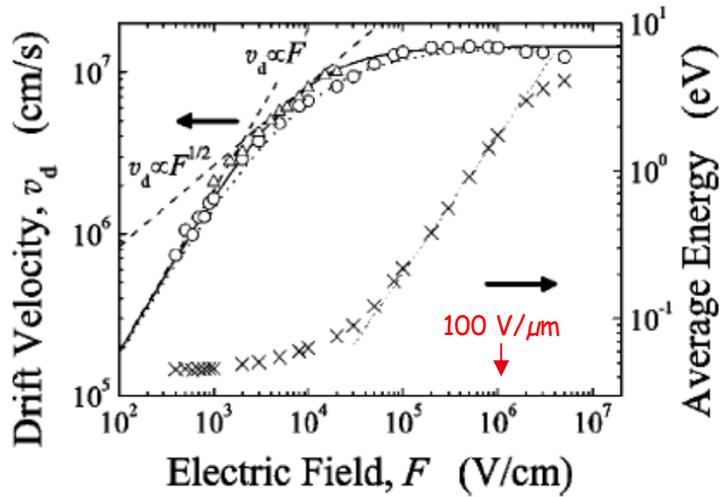
Energy straggling

scCVD controlled irradiation of living cells  
**U2OS XRCC1-GFP, 100 protons/point**



goal: single proton irradiation

Watanabe, T., Masatake, I., Teraji, T., Ito, T., Kamakura, Y., Taniguchi, K., Jpn. J. Appl. Phys. **40** (2001) L715-717.



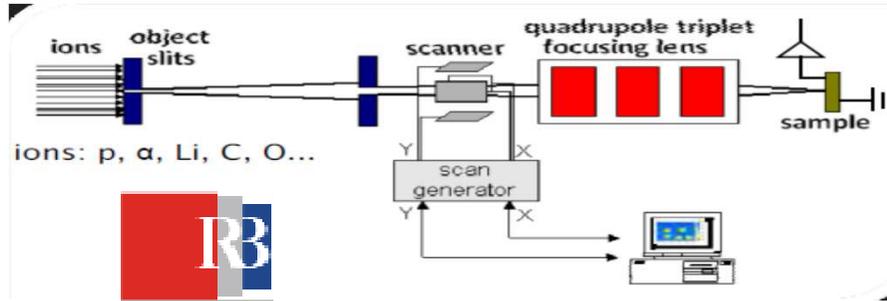
Threshold for impact ionization  $\sim E > 100 \text{ V/micron}$  (1MV/cm), commercial scCVD plates:

500  $\mu\text{m}$  - 50 kV bias (hmmm...),  
50  $\mu\text{m}$  - 5 kV bias (risky)

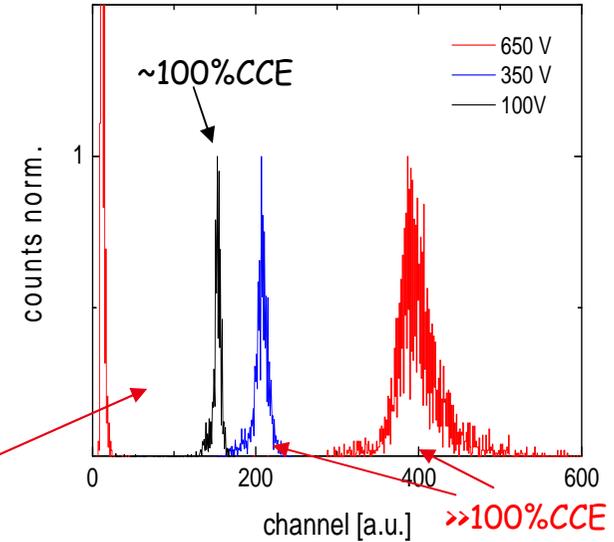
5  $\mu\text{m}$  membranes - 500 V bias (ok!)

→ a nice opportunity to try to study impact ionization

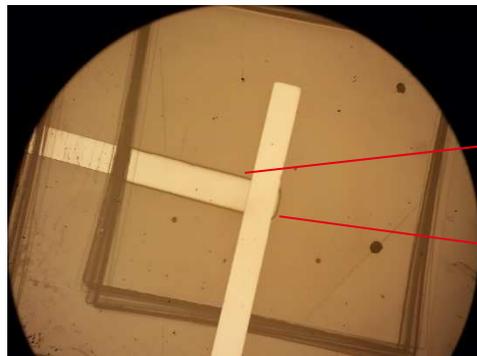
Ion microbeam line @ RBI, Zagreb, Croatia



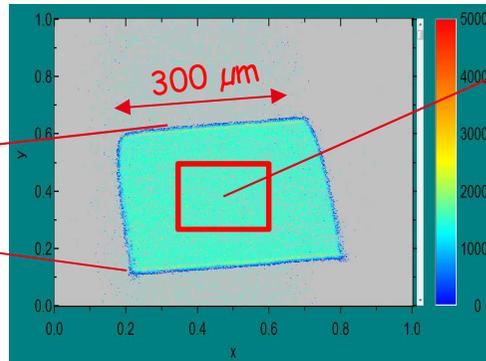
Corresponding PH spectra



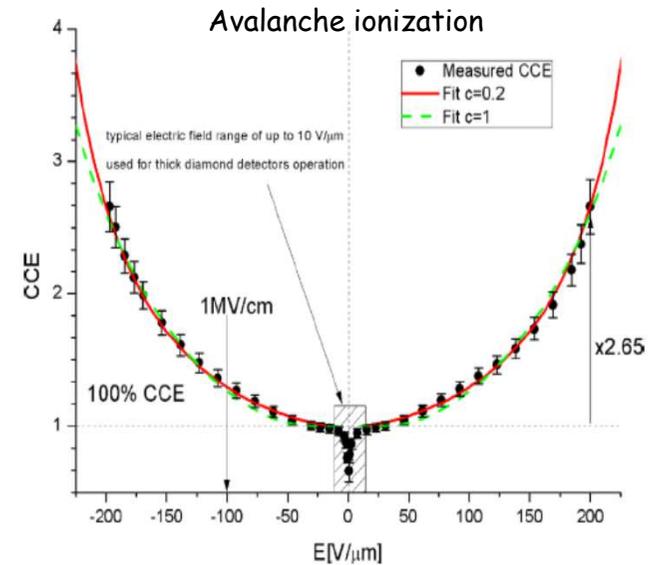
DUT with 18 MeV O ions



pulse-height map

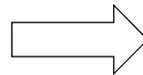
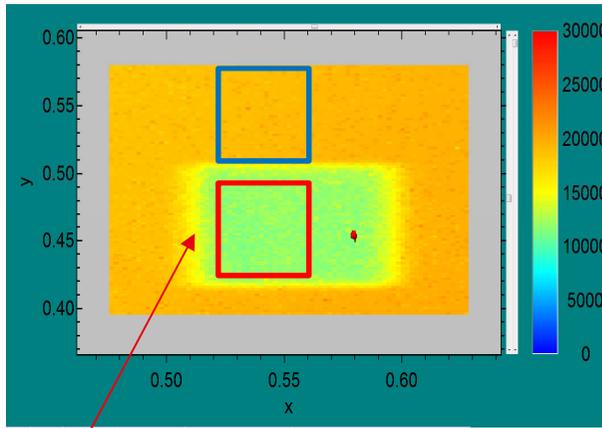


Not an easy task - examples of devices hard breakdowns

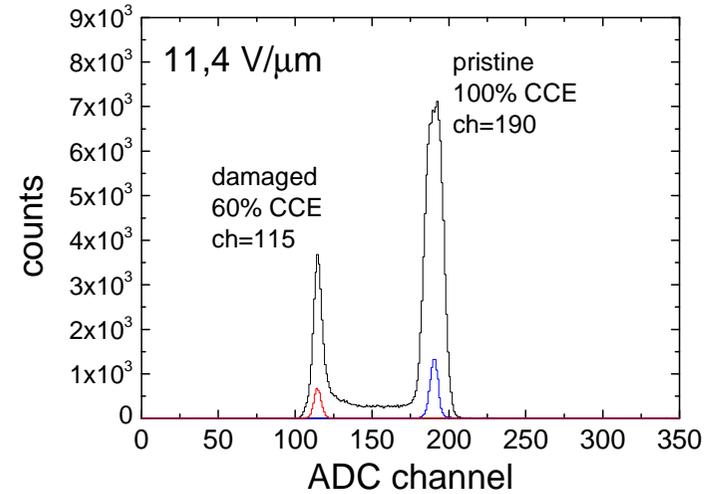


Appl. Phys. Lett. 109, 043502 (2016); doi: 10.1063/1.4959863

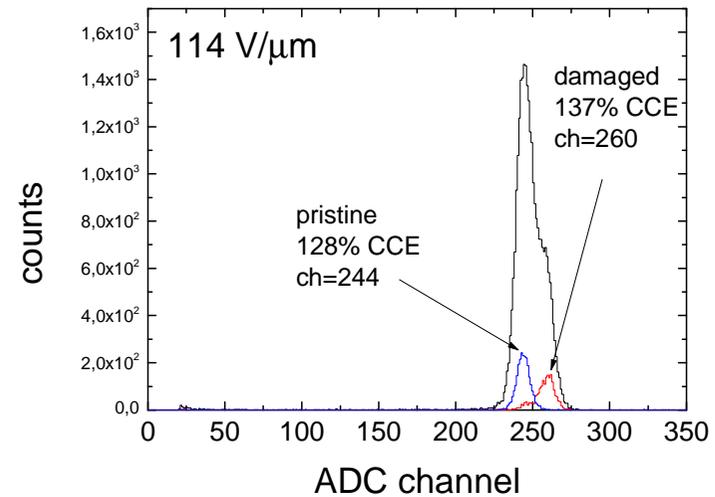
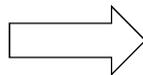
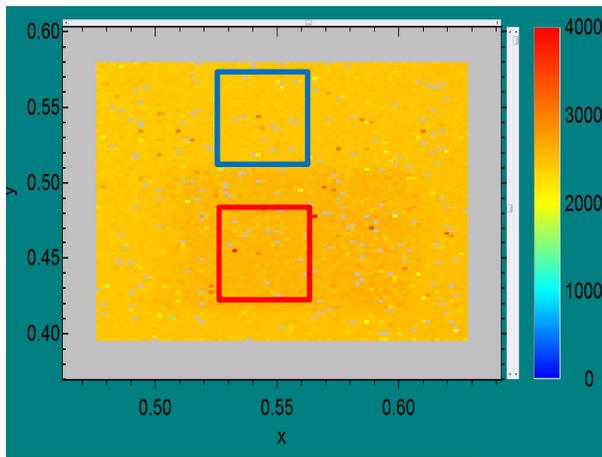
CCE map after radiation damage



corresponding pulse-height



region damaged with  $1 \times 10^{11}$  O (18MeV) ions/cm<sup>2</sup>

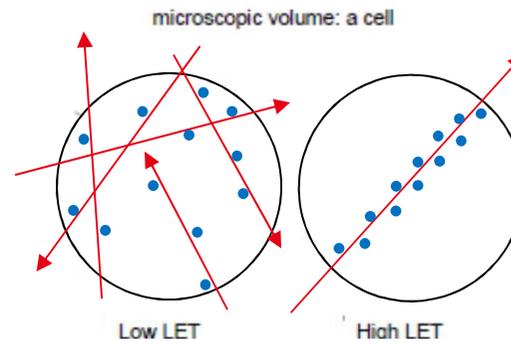
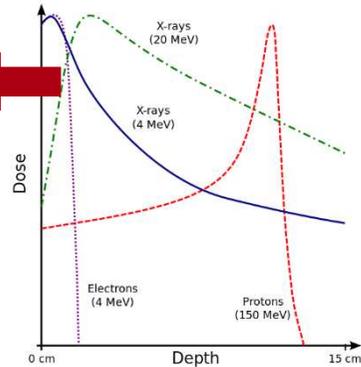
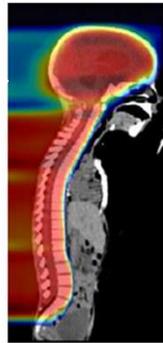


# MICRODOSIMETRY IN HADRON THERAPY

Photons distribution



Protons distribution



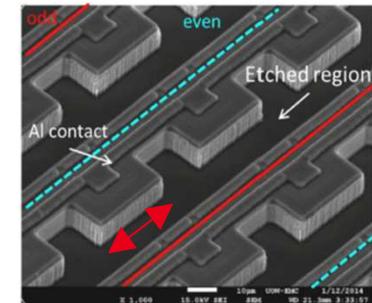
Same dose but different LET thus various biological effectiveness

A TEPC



silicon brige microdosimeter

- RBE (**Relative Biological Effectiveness**) of protons is uncertain : limits the efficiency of treatments;
- strong correlation between a microdosimetric quantity (i.e. spatial **distribution of energy deposition by single particle at cellular level**) and RBE : LET (linear energy transfer) and biological effects of charged particles in tissues are related;
- measurement of LET is difficult : today **no detector is available in clinical routine.**



B. Rosenfeld, NIM 2015

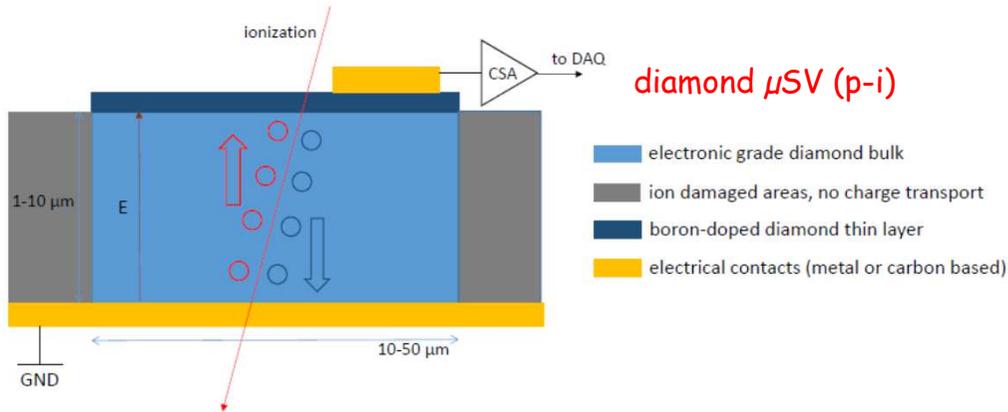
microdosimetry ≠ dosimetry at micron scale

single-particles (low charge), ns to μs integration time ( $10^9$  p/cm<sup>2</sup>), pulse-height spectra, SV of micro or nano size

~ms integration time, DC current or charge macroscopic (mm) SV size

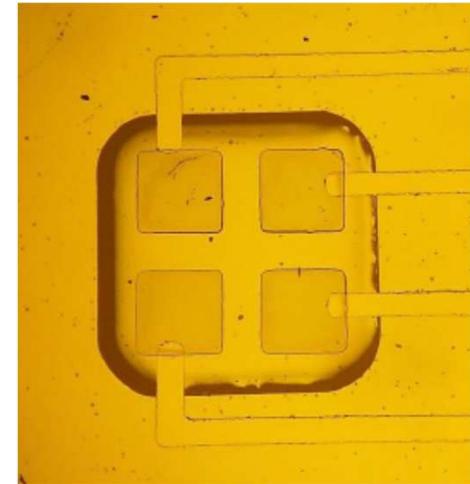
# DIAMOND MICRODOSIMETRY

Tissue-equivalent, radiation hard  
scCVD diamond membrane microdosimeter

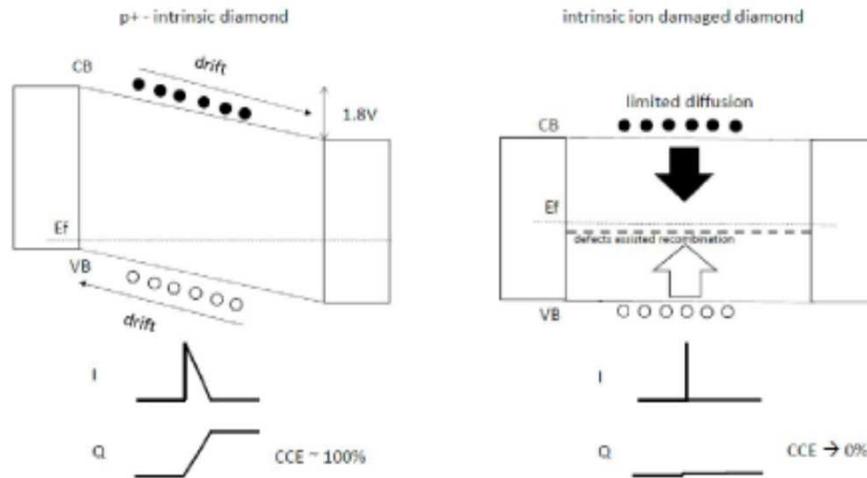
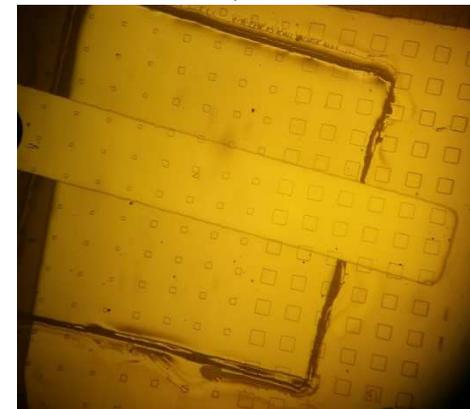


proof-of-principle  
scCVD diamond prototypes

4 x 300  $\mu$ m pixels (SV), 6  $\mu$ m thick



25, 50 and 100  $\mu$ m pixels (SV), 3  $\mu$ m thick

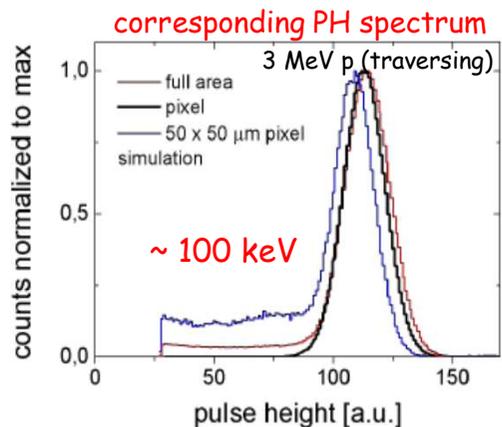
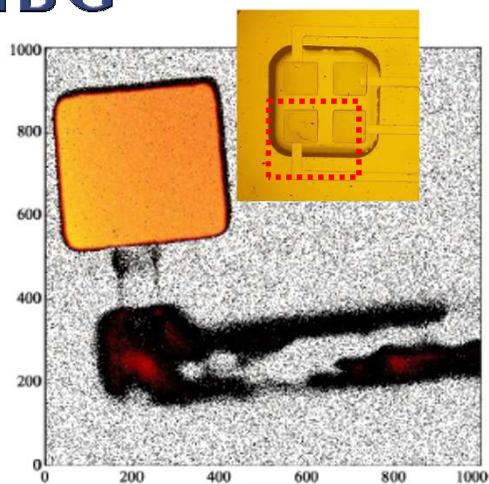


**p-i-m diamond junction**

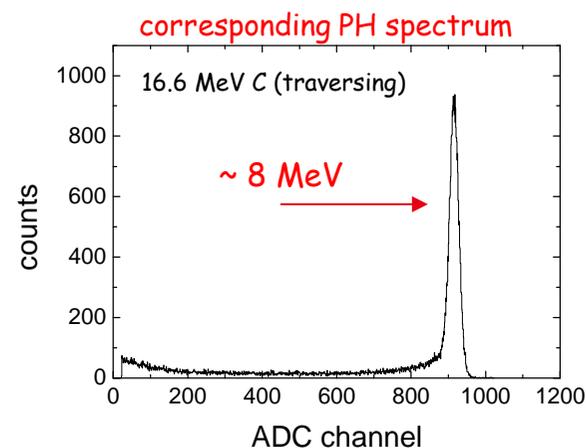
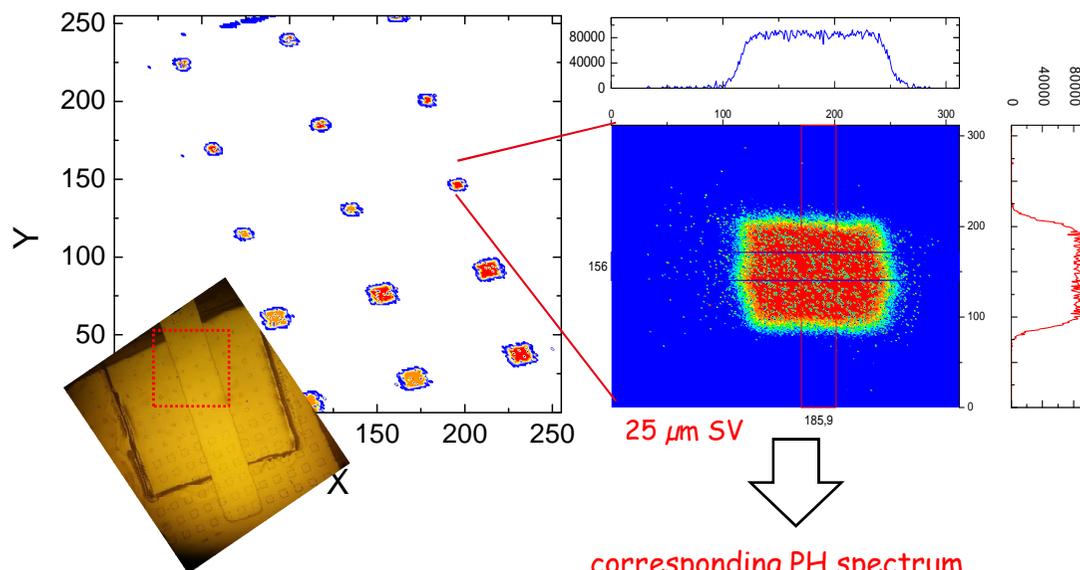
self-biased (1.8V), fully depleted

# DIAMOND MICRODOSIMETRY

3 MeV proton microbeam  
CCE mapping @ 0V



16.6 MeV carbon microbeam CCE mapping @ 0V



- works @ 0V
- complete CCE (p)
- well resolved energy-loss spectra
- well defined  $\mu\text{SV}$  (non-active m-i-m)

useful for LET measurements already at this stage

## SUMMARY

### Diamond material:

still large wafers of single crystal diamonds are missing  
...but some hope ahead..

### Diamonds detectors are here:

doing the job, commercialized, getting more popular

### R&D on new types of diamond detectors ongoing:

#### 3D Diamond Detectors:

from scratch to almost complete technology within 6 years

#### Ultra-thin scCVD membrane detectors:

transparent, robust devices, with excellent charge transport properties

numerous (but rather niche) applications including:

X-ray beam monitoring, radiobiology, avalanche counters, microdosimetry



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THANKS TO:



DiamFab for growing excellent quality p+ diamond homoepitaxial layers  
[www.diamfab.eu](http://www.diamfab.eu)

Diamond Microdosimetry



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Diamond Membranes for Radiobiology DIRAC



Instrumentation aux limites 2016

!!! Thank you very much for your kind attention !!!

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