Graphitic contacts in Silicon Carbide and Diamond for dosimetry and radiation detection

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Proton Therapy Workshop

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Centro Nacional de Microelectrónica IMB

CSIC Consejo Superior de Investigaciones Científicas

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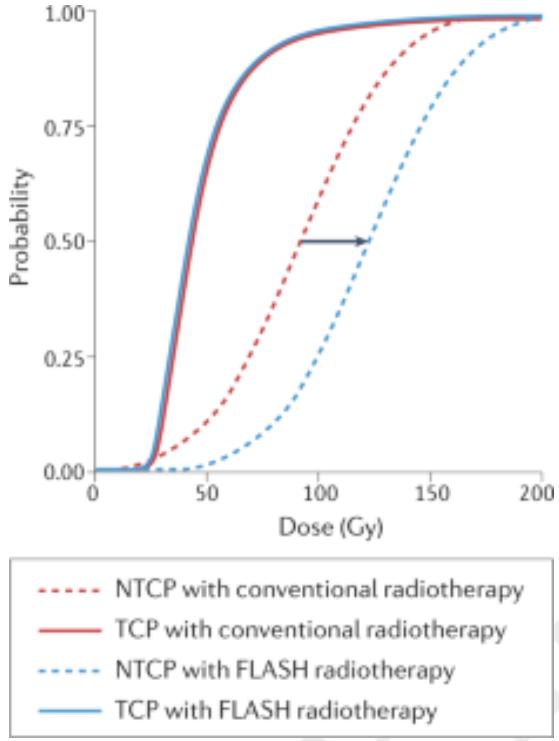
Instrumentation for FLASH

FLASH therapy reduces toxicity in cancer treatments

- 100 kGy/s to 10MGy/s in less than 200 ms
  - <0.03 Gy/s in conventional radiotherapy
- High radiation doses in short times

Radiation hardness need Fast response

Nature Reviews Clinical Oncology **vol 19**, 791–803 (2022)



Wide band-gap materials for dosimetry

Properties	Silicon	4H-SiC	CVD Diam.
Band-gap [eV]	1.12	3.23	5.5
e-h pair generation energy [eV]	3.6	7.8	13
Displacement energy threshold [eV]	13-20	22-35	40-50
Thermal conductivity [W/(m K)]	148	370	1000
Atomic number	14		6
Electron sat. velocity (1e17 cm/s)	0.86	3	2
Cost	O(100€)	O(1k€)	…

→ **Low signals, low background**

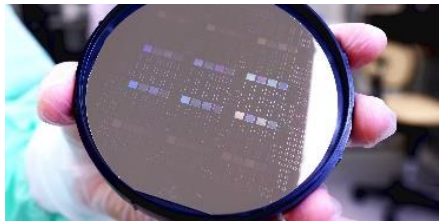
→ **Radiation hard**

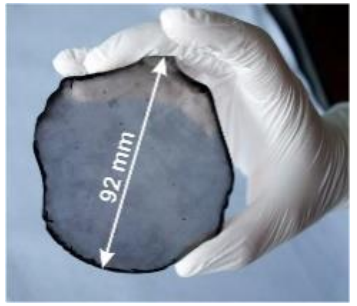
→ No need for cooling

→ **Tissue equivalent (Z_{eff} = 7.42)**

→ **Fast signals**

→ Expensive…





Sci Rep 7, 44462 (2017)

Graphitic contacts

Typical detectors have **metal** over the active region
→ contribution of secondary interactions
→ can stop low energy ions/light

Here, we try to replace the metal with **graphene/graphite,** a conductive carbon layer
→ less interaction with radiation
→ tissue equivalence

Wide band-gap detectors with carbon contacts

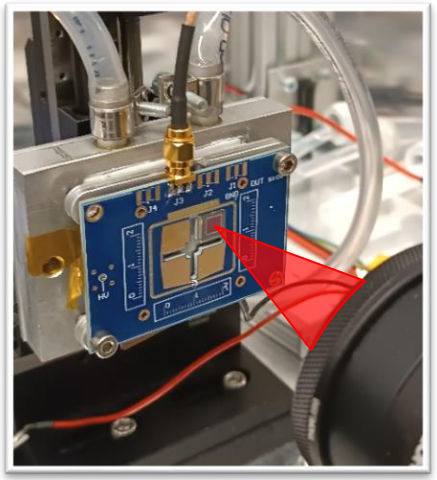
Technologies in this talk

SiC detectors with graphene:

- Study charge formation in 2D
- Dose rate linearity in pre-clinical LINAC
- Characterisaton in X-ray tube

Diamond with graphitic electrodes:

- First prototypes of radiation detectors at CNM



SiC with Graphene contacts

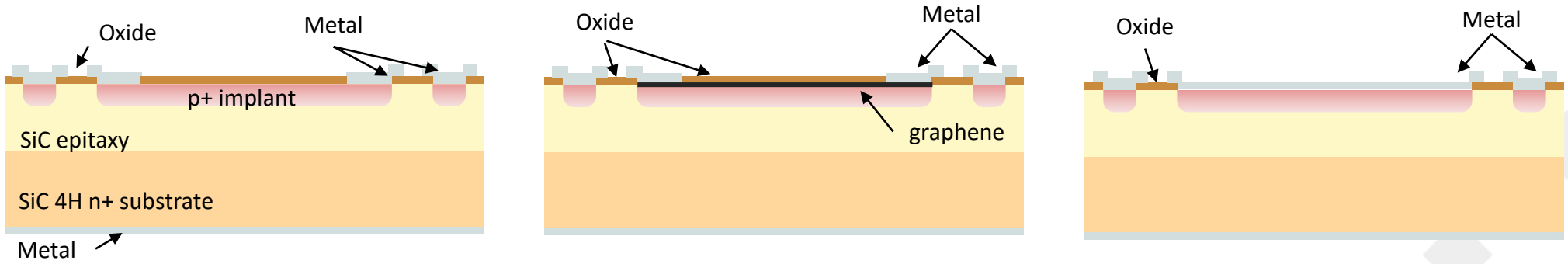
SiC detectors with Graphene contacts

SiC diodes (50um epitaxy) with three geometries fabricated at CNM

No Metal
Metal ring around the active region, nothing elsewhere

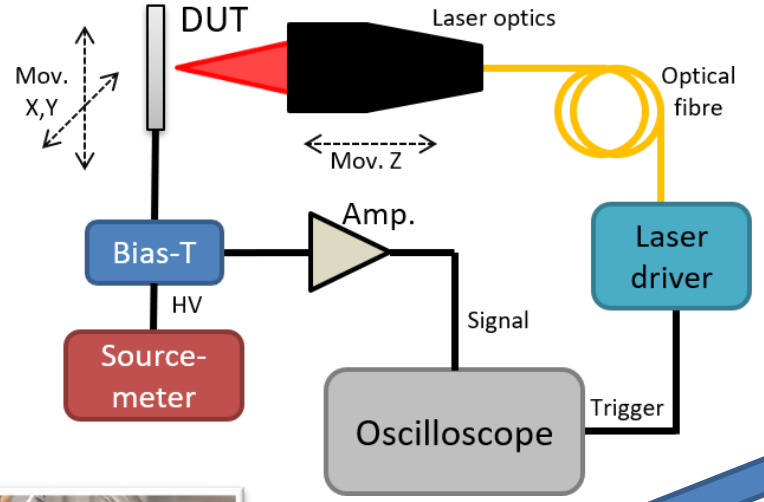
Graphene
Graphene layer contact over all active region, with metal ring for wirebonding

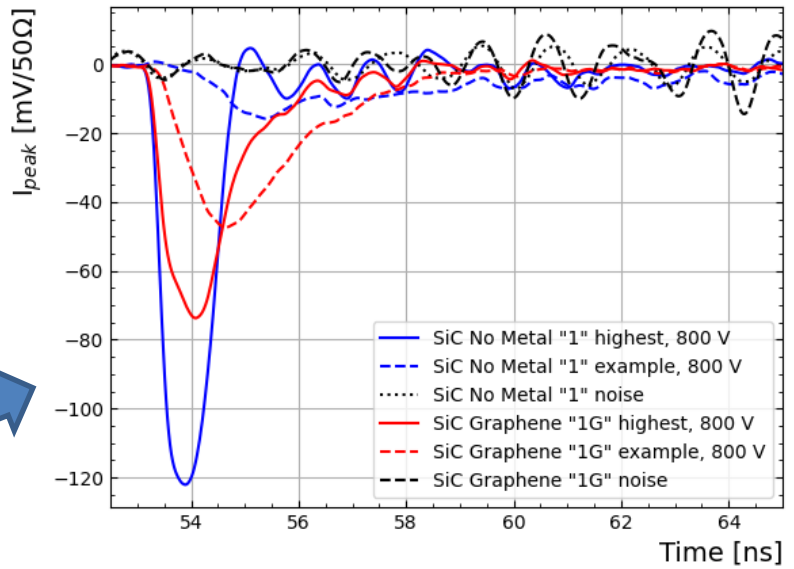
Metal
Metallic contact over all active region



Able to study the effect of Graphene in the detectors!

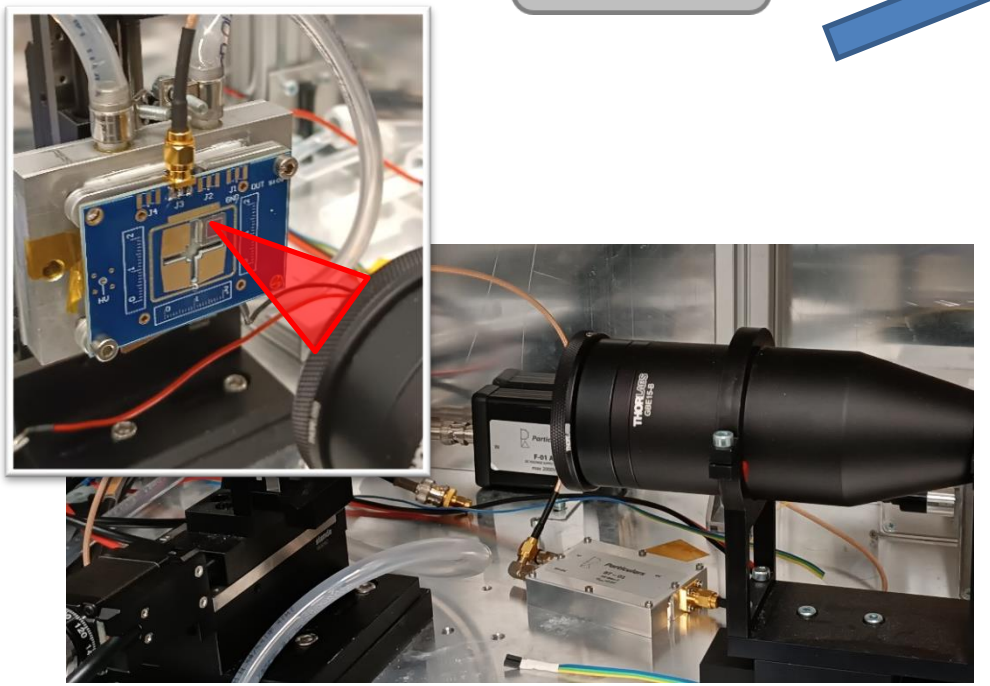
Effect of Graphene in charge collection





Comparison between signals produced by a "Graphene" and a "No Metal" sample

Note: "Metal" sample cannot be tested under this set-up



Transient Current Technique

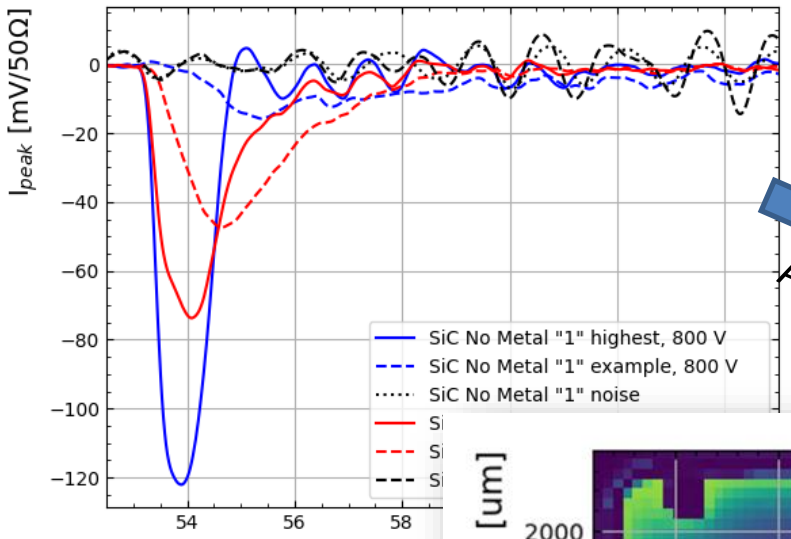
λ=369 nm (UV)

Pulse width: 100 ps

Freq.: 1 kHz rep. rate

Devices: SiC-Graphene
SiC-No Metal

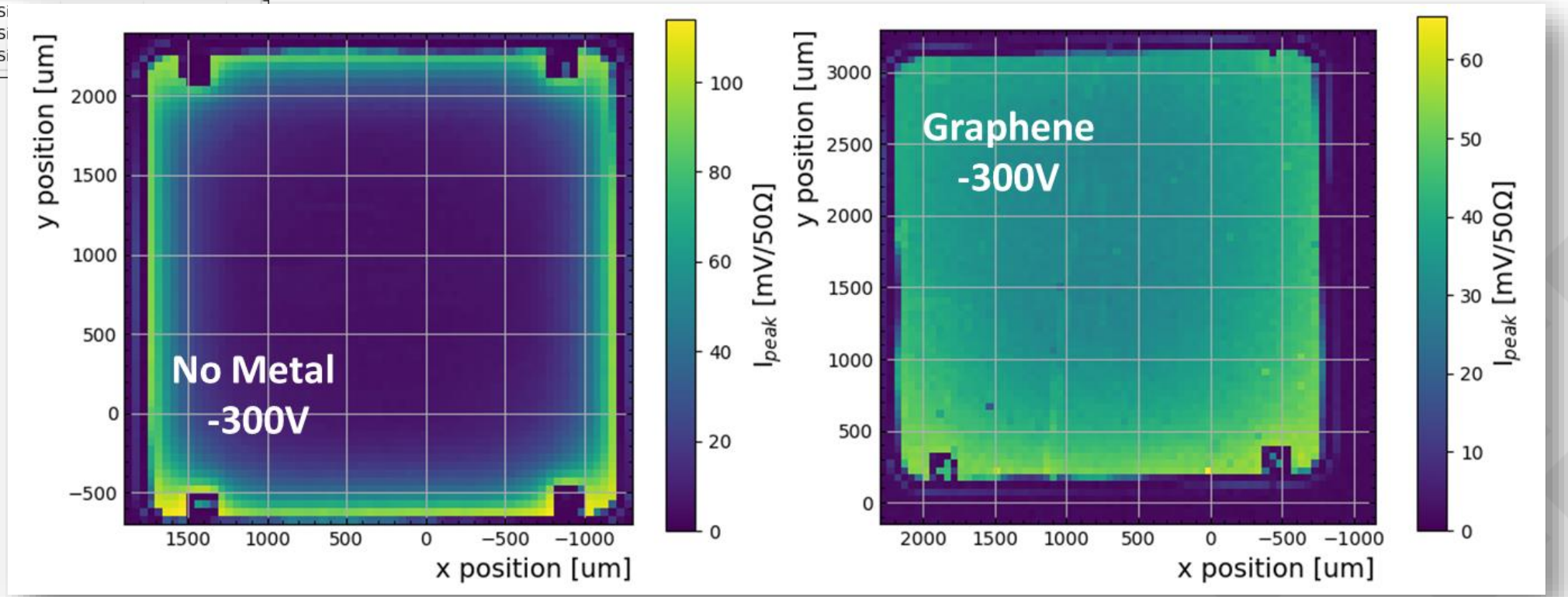
Effect of Graphene in charge collection



2D mapping

Amplitudes

Graphene lowers signal amplitude reduction



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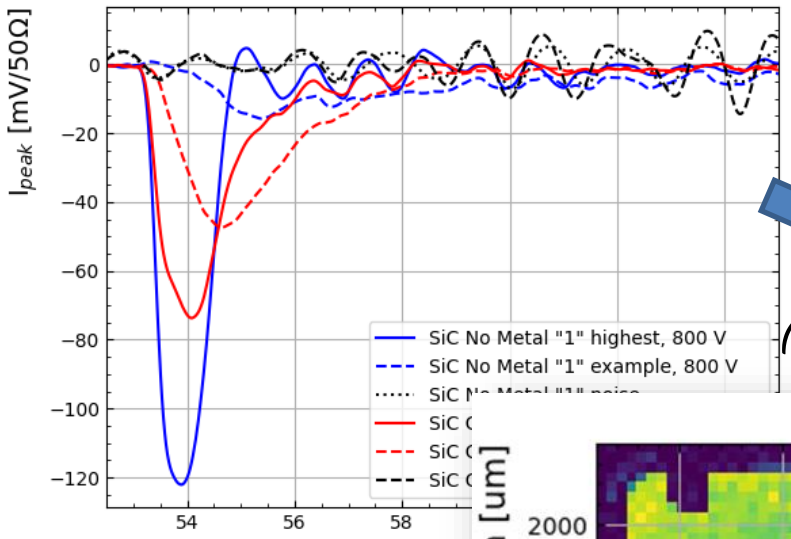
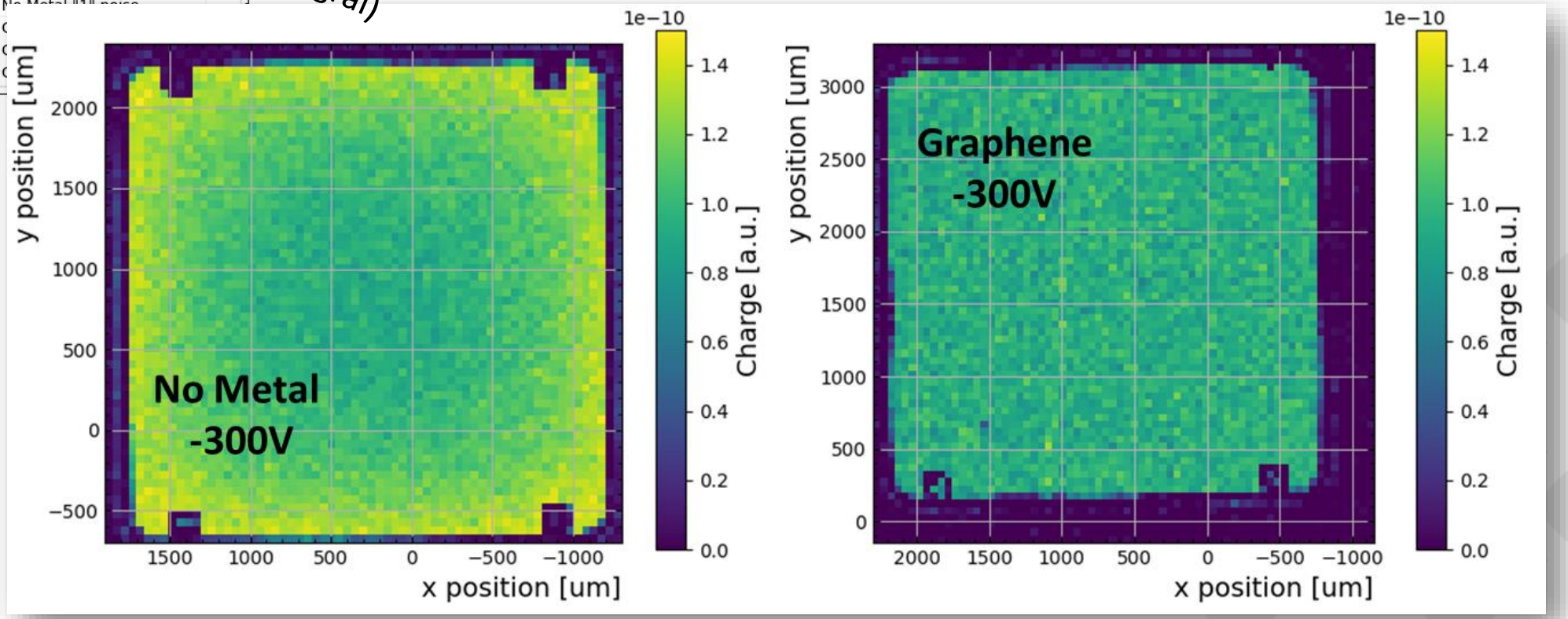


2D mapping

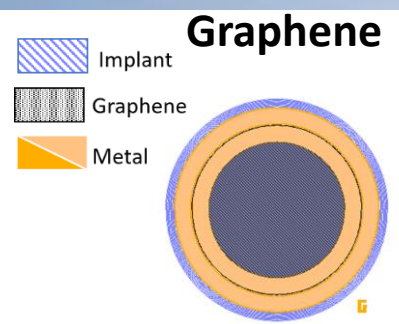
Charges (integral)

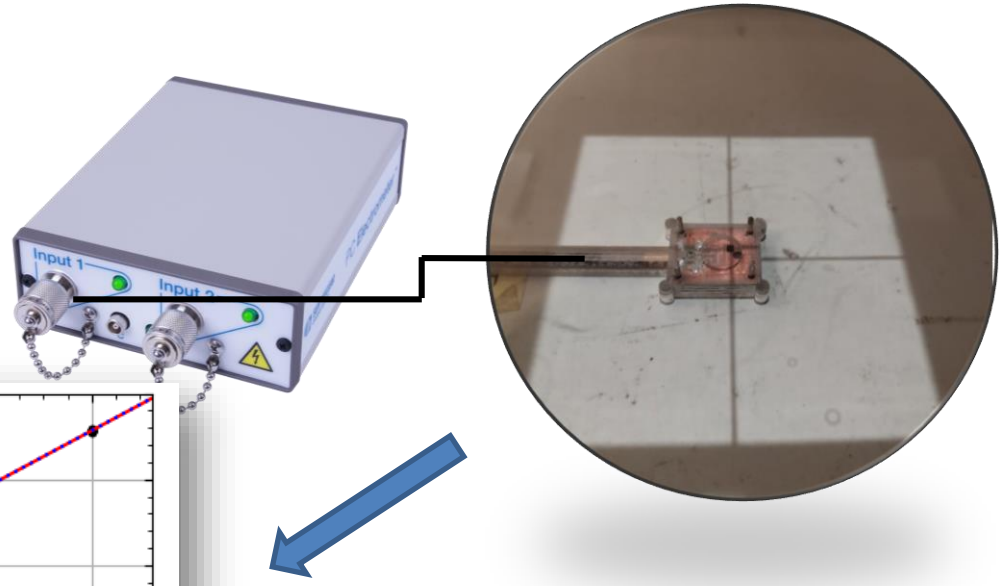
Total charge collection is uniform but less sensitive to noise

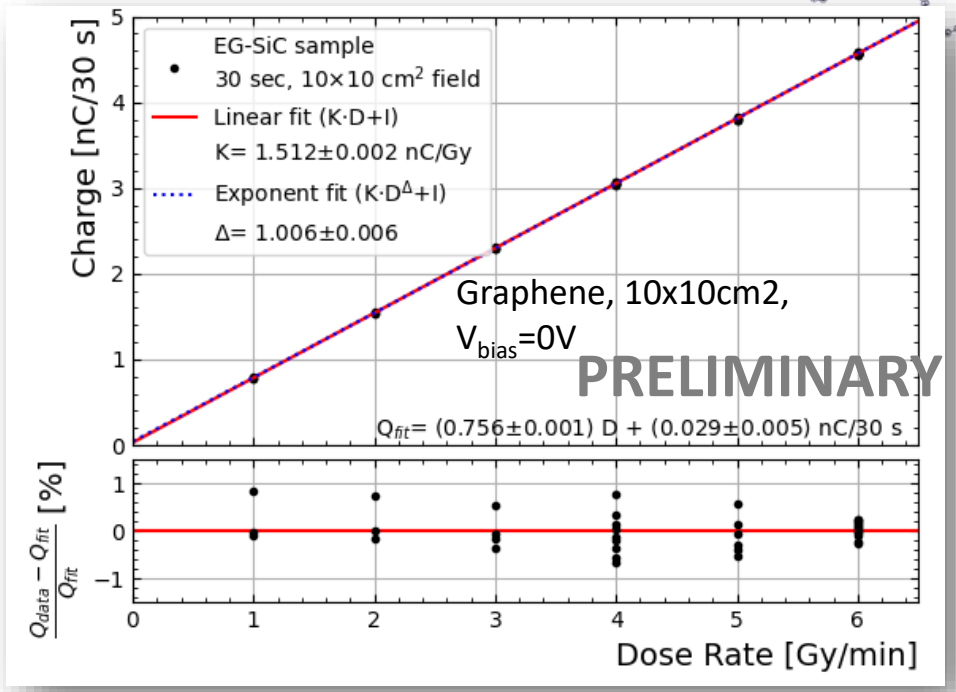


Dosimetry with SiC devices

LINAC
Op. Voltage: 60MV
Dose rate: 1 - 6 Gy/min
Devices: SiC -- graphene
Readout: Sun Nuclear e-meter







Linac at University of Santiago de Compostela, 8h beam time

Dosimetry with SiC devices

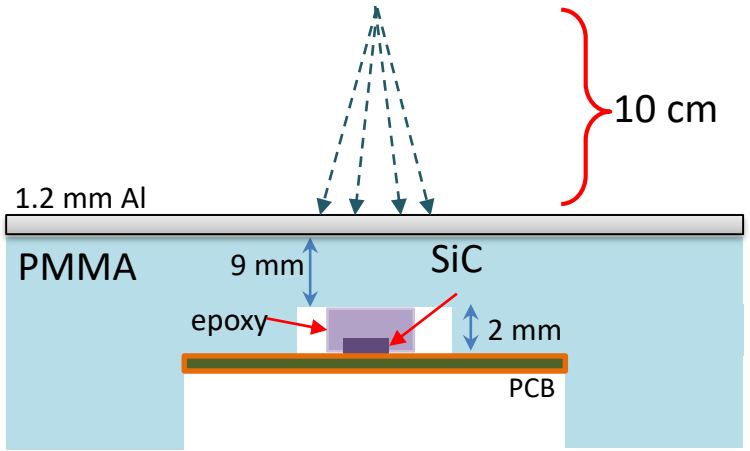
"No Metal" sample looks unstable

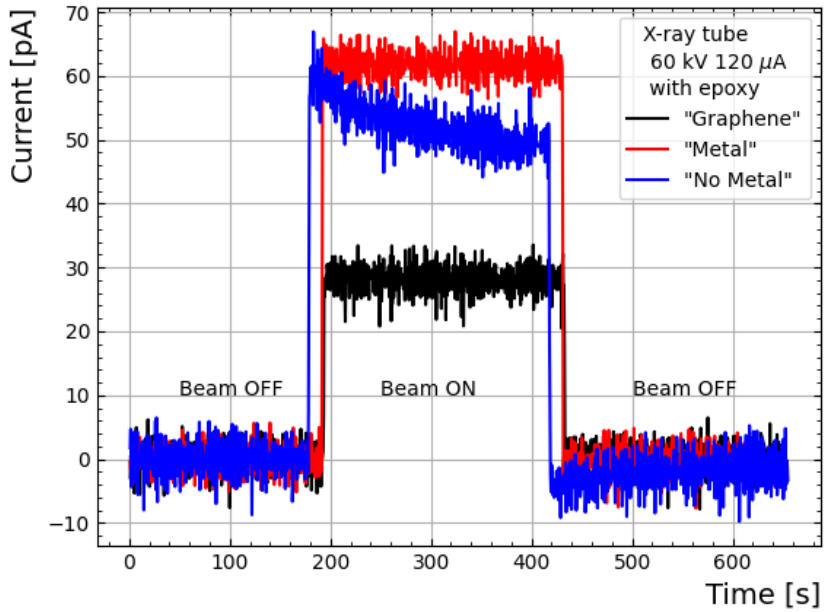
"Graphene" and "Metal" samples show a stable and fast behaviour

"Metal" sample is 2x more responsive than **"Graphene"**

- Multiple scattering due to heavy metals (Au and Ti) on active region

Tungsten anode X-ray tube
Op. Voltage: 60kV
Beam current: 60-120 uA
Devices: SiC-Graphene
SiC-Metal
SiC-No Metal
Readout: Sun Nuclear e-meter





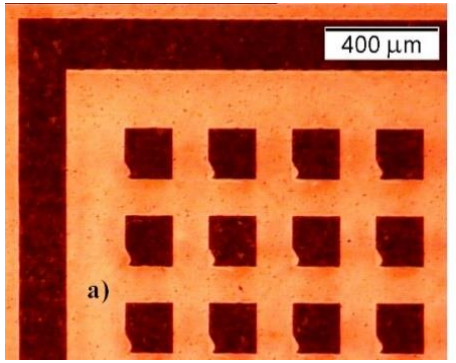
Diamond

All-carbon diamond detector approach

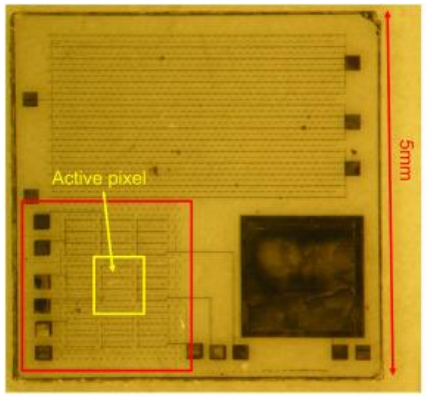
Typically fabricated with laser graphitisation, replacing metal by other groups

- Slow for large production! => O(10um/s) laser movement

NIMA 837 136142 (2016)



Diam and Rel Mat 133 109692 (2023)



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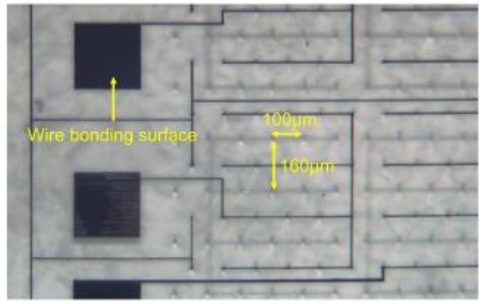


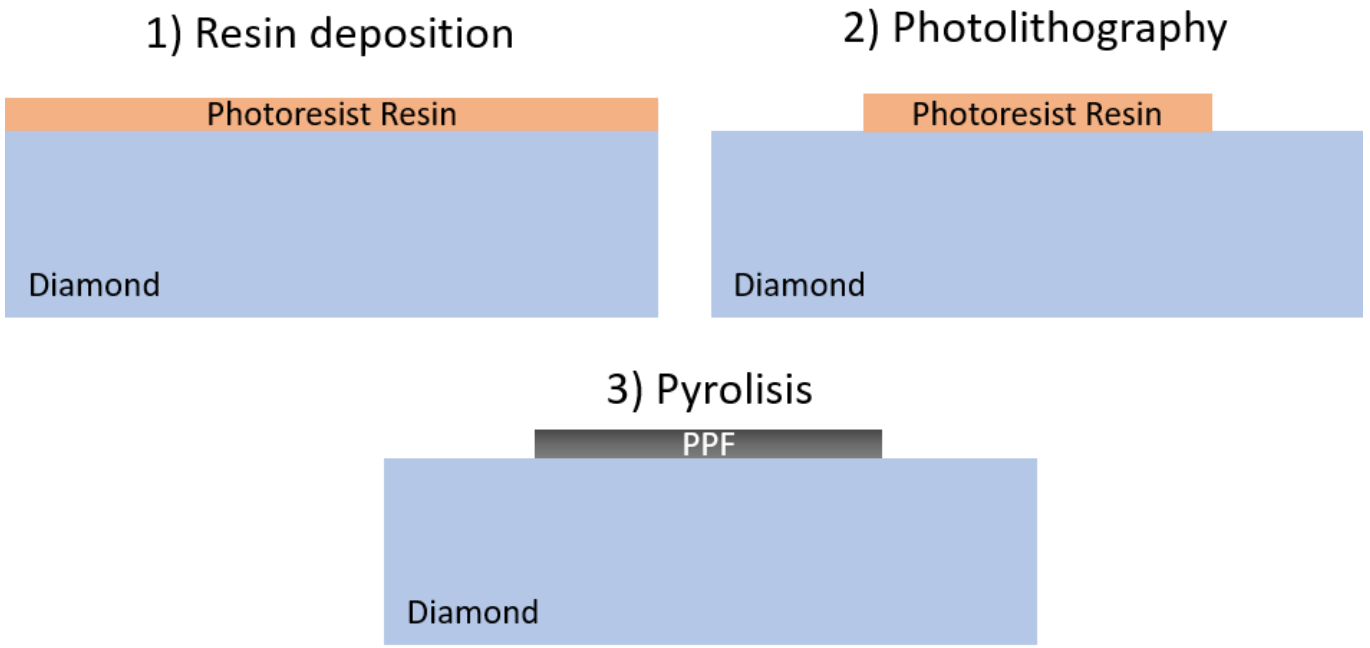
(b)

Next is an attempt to produce All-carbon devices with a different approach
(available in tne CNM clean room)

First proof-of-concept via PPF: Fabrication

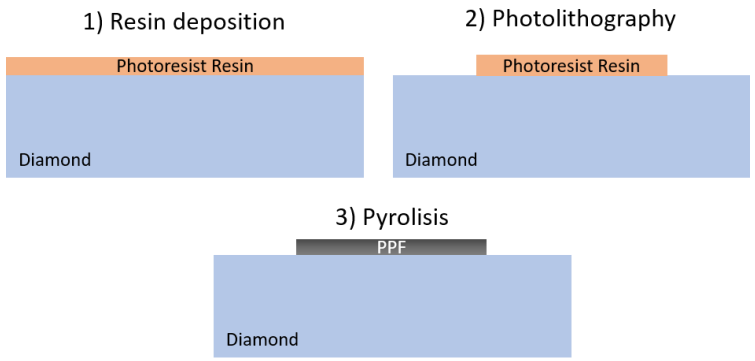
Pyrolitic Photoresist Film:

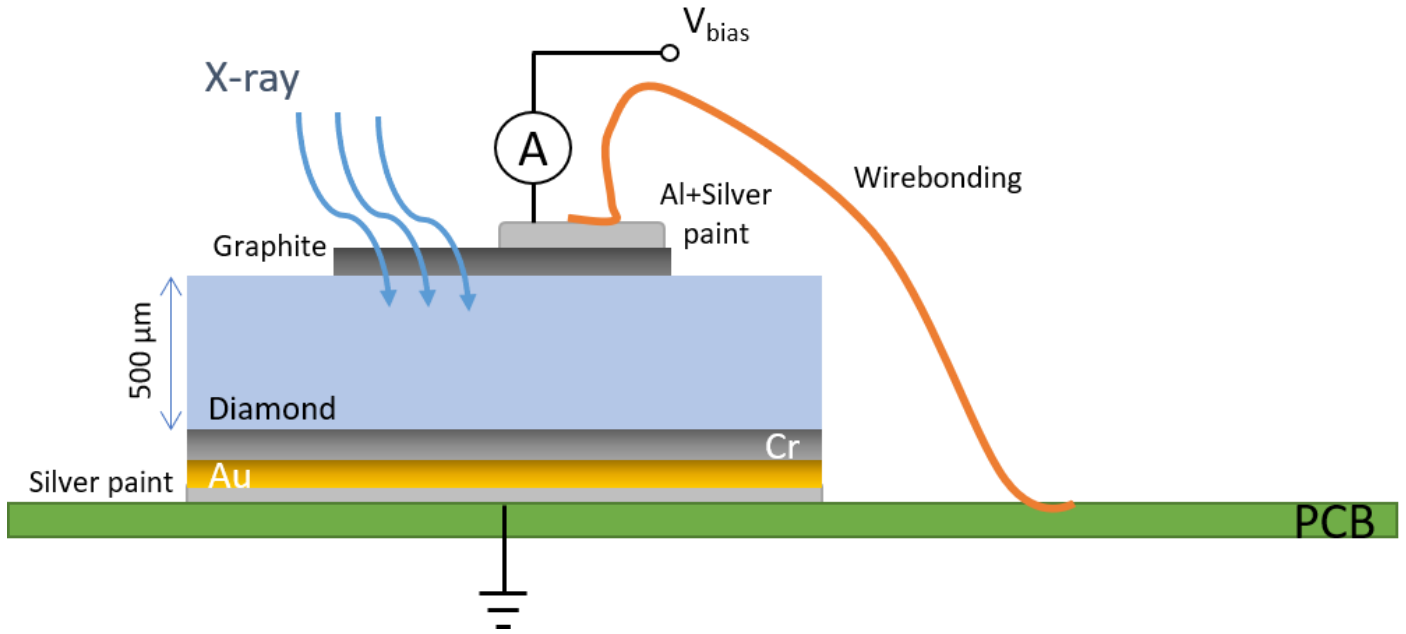
- Sublimate non-carbon components from resin with high temperature



Simple technological process, easy to scale for production!

First proof-of-concept via PPF: Fabrication



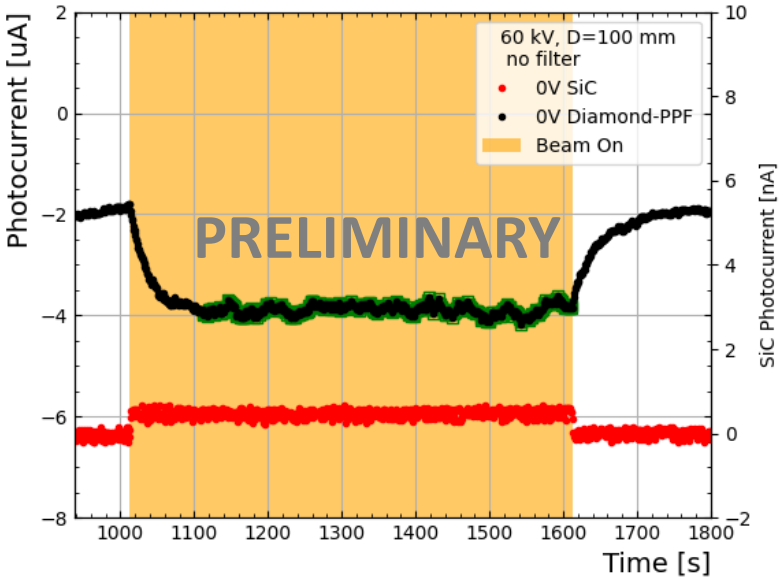


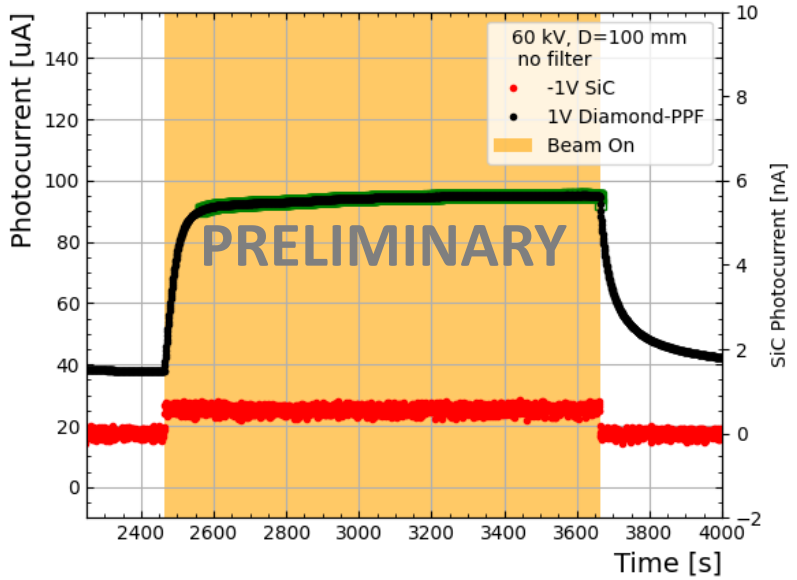
First proof-of-concept via PPF: Characterisation with X-ray tube

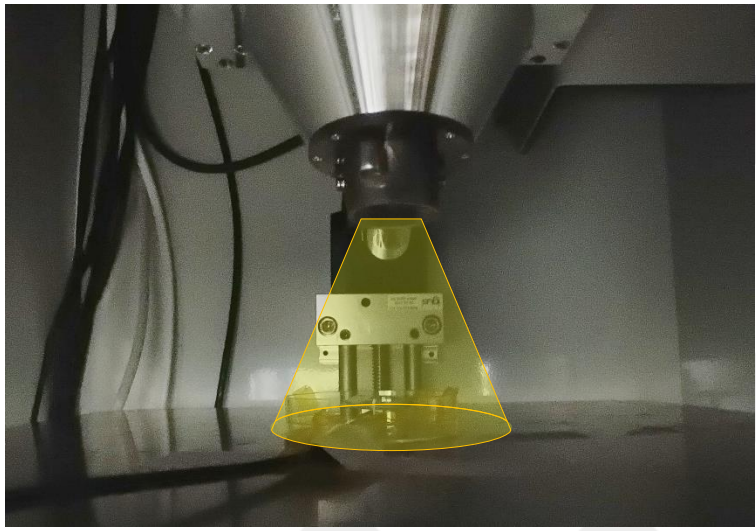
Tungsten anode X-ray tube
Op. Voltage: 60kV
Beam current: 60-120 uA
Devices: Diamond-PPF
SiC diode
Readout: Keithley 2410

Measure photocurrent, with beam on for 5 min at different beam currents:

- Prototype is **responsive even at 0V!**
- It's slow, needs about 100 sec to stabilise due to bulk material quality
- Needs pre-irradiation / "priming", usual case for diamond in general







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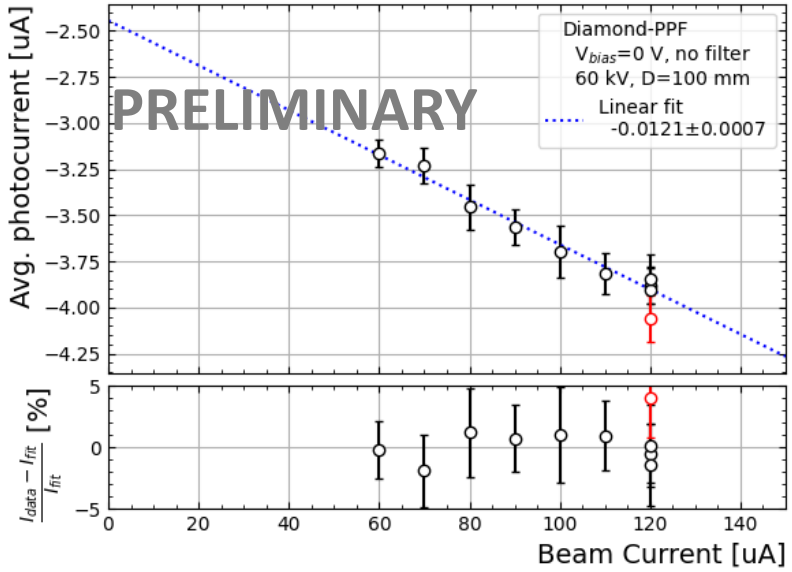

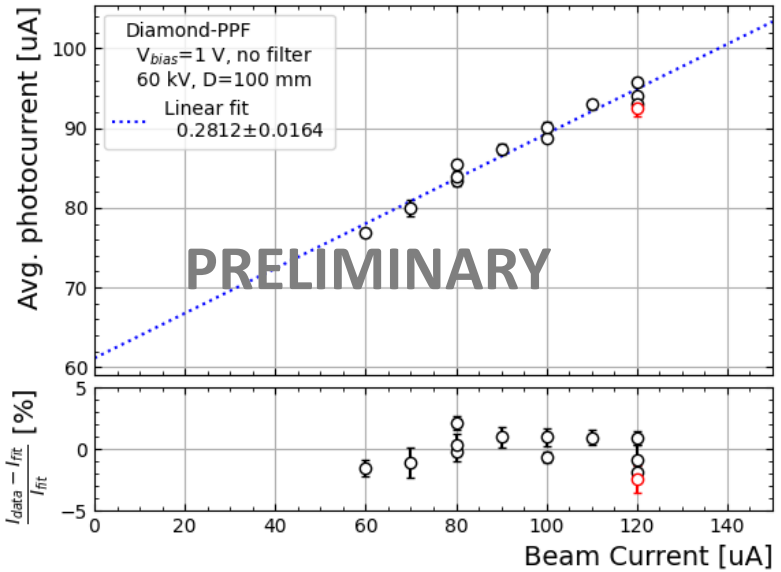

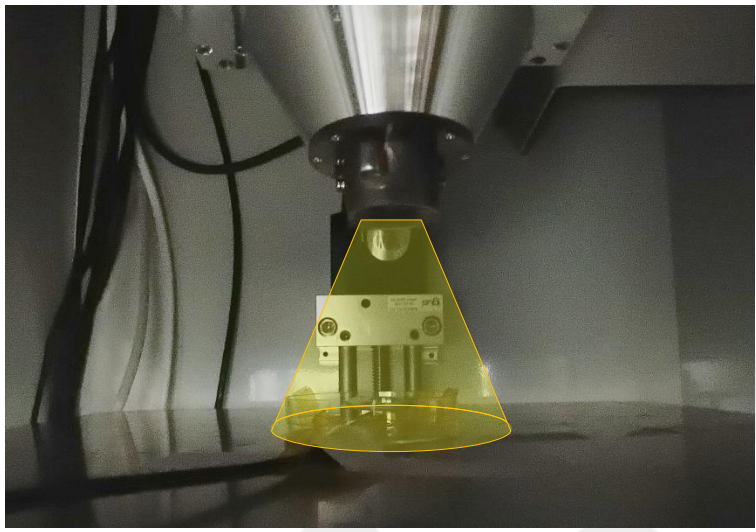

Conclusions

Wide band-gap detectors for dosimetry fabricated at IMB-CNM:

Silicon Carbide diodes with Graphene:

- Graphene improves charge collection on SiC diodes over not having metal
- Prototype works with good linearity in LINAC and laboratory

Diamond with PPF graphite:

- Proof-of-concept tested in laboratory
- Sensitive to lab X-rays

Next:

- Need to improve packaging
- Test under FLASH conditions
- Irradiations/radiation resistance

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Thanks for your attention

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Back-up slides

SiC detectors with Graphene contacts

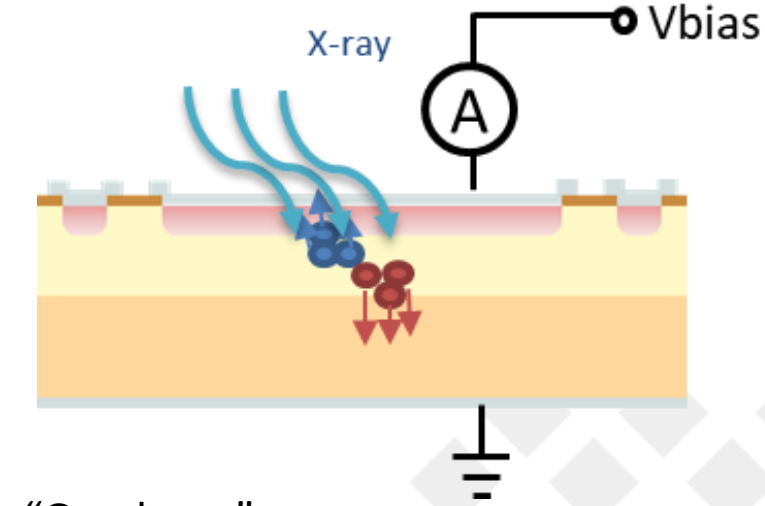
SiC diodes (50um epitaxy) with three geometries fabricated at CNM

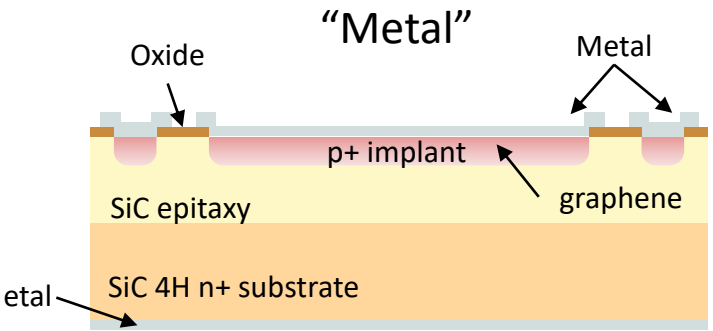
- **Metal:** Metallic contact over all active region
- **No Metal**: Metal ring around the active region, nothing elsewhere
- **Graphene**: Graphene layer contact over all active region, with metal ring for wirebonding

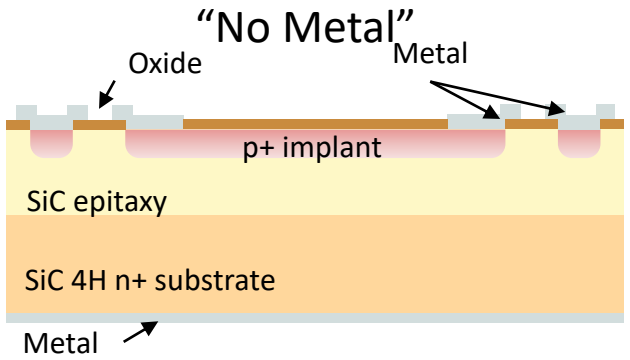
- Front (Ti 20nm/Pt 100nm/Au 100nm) and back (Ti 50 nm /Ni 150 nm /Au 50 nm) metals for contact

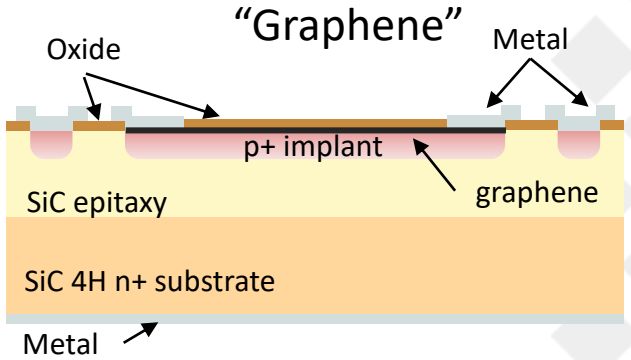
Able to study the effect of Graphene in the detectors!

Working principle:
- Reverse bias voltage applied across the diode
- Impinging radiation generates charge carriers
- These drift towards electrodes generating current





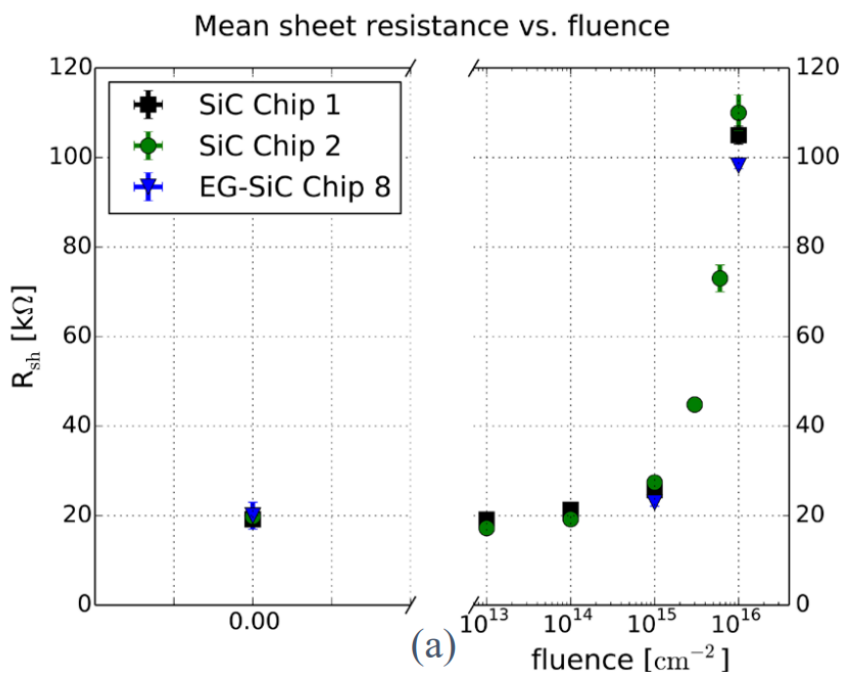


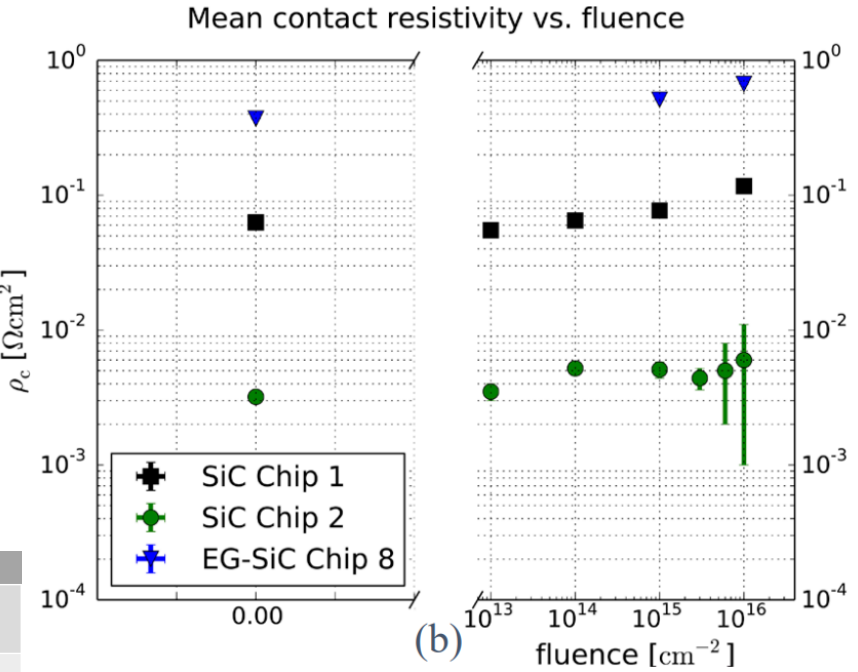


Contact resistance and contact resistivity

- Measured by CTLM in test structures
- Irradiation campaigns at the ATI Triga Mark II nuclear reactor
- R_{sh} : related to doping concentration
  - Increase as a function of fluence
    - Indication of acceptor removal
    - Consistent across EG-SiC and SiC samples
- ρ_c : related to metal-(graphene)-SiC contact
  - Increase as a function of fluence
  - EG-SiC sample much higher resistivity
    - Effect of graphene interface
  - Difference between SiC samples
    - Different metal stacks

Device	Metallisation
SiC Chip1	Ti/Al/Ti/W (15/90/30/100 nm - evaporation)
SiC Chip 2	Ti/Pt/Au (20/100/100 nm - sputtering)
EG-SiC Chip8	Ti/Pt/Au (20/100/100 nm - sputtering)

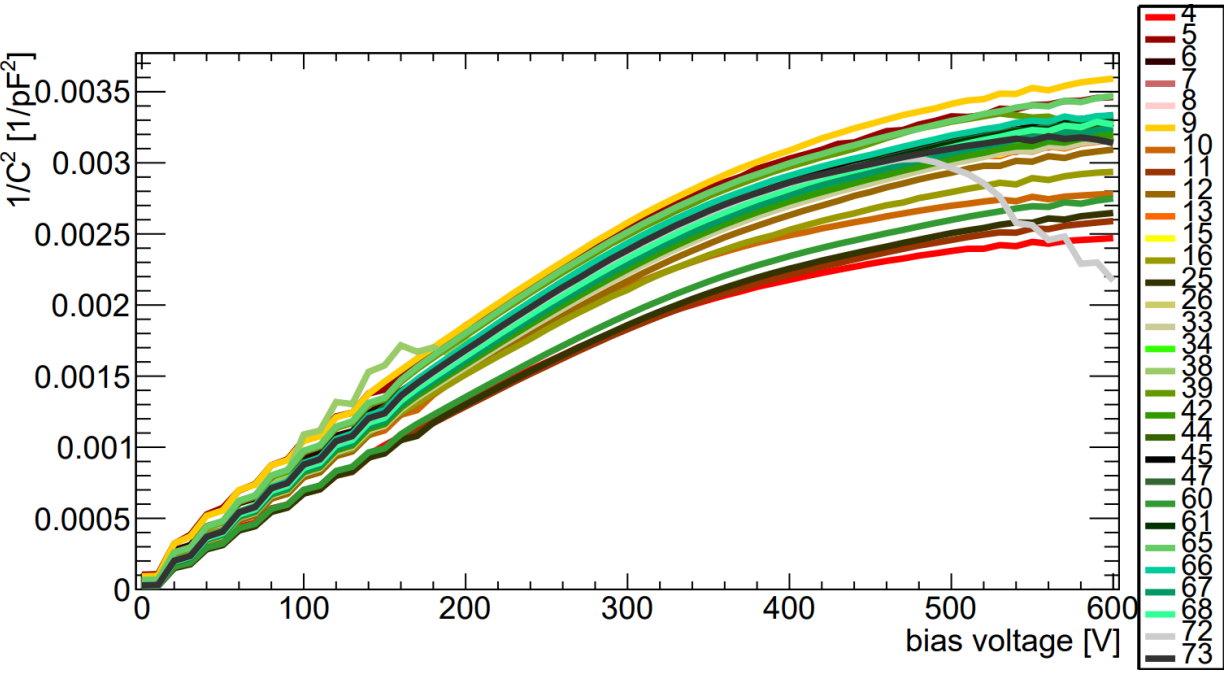




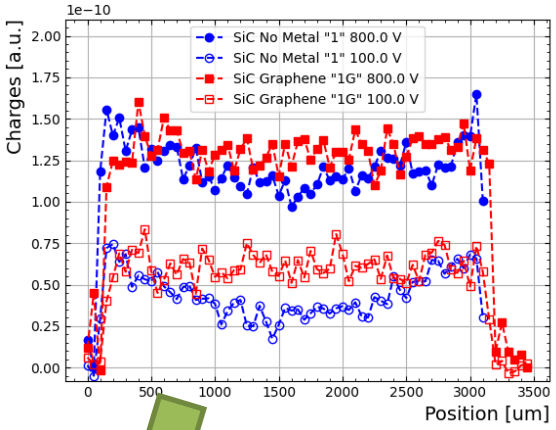
S. Otero Ugobono et al, Materials Science Forum. Trans Tech Publications, Ltd., May 31, 2022

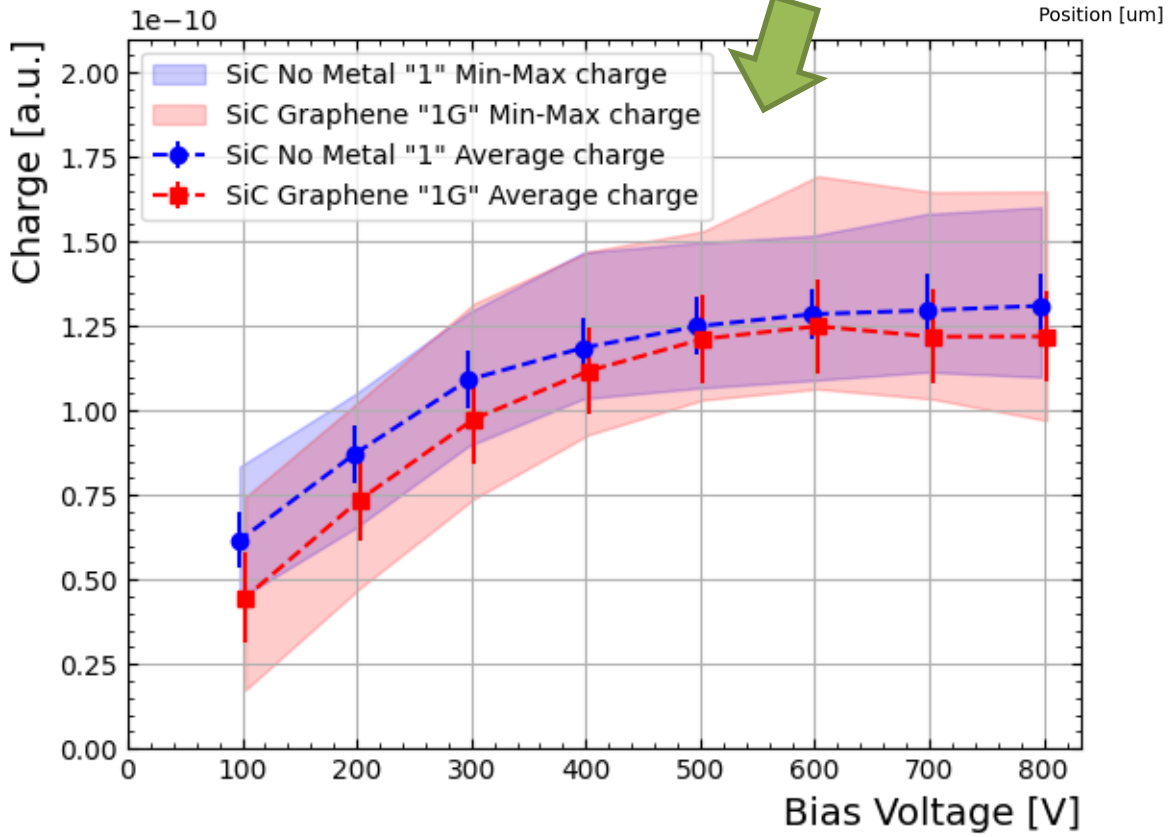
Charge collection vs Voltage

- Both samples seem to start to saturate at 400V
- Consistent with C-V measurements



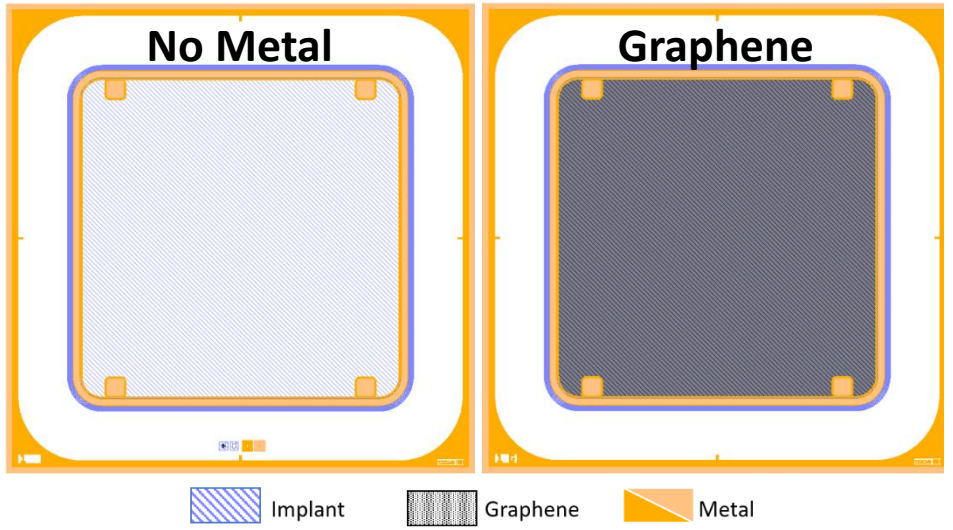
Presence of graphene does not affect saturation voltage

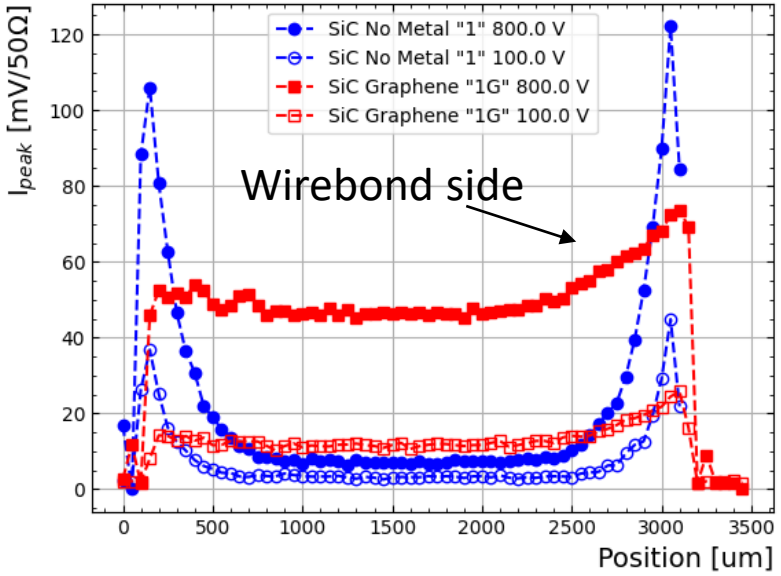


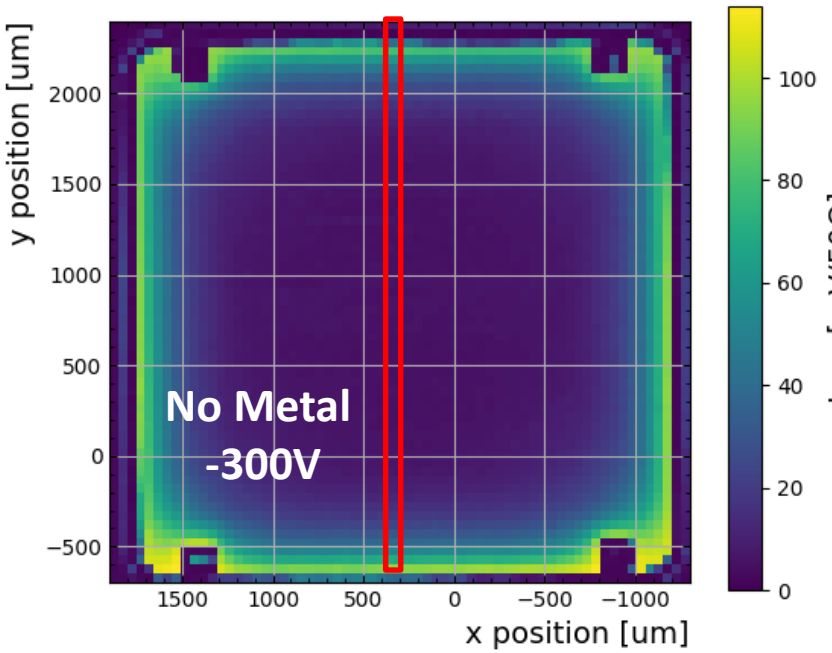


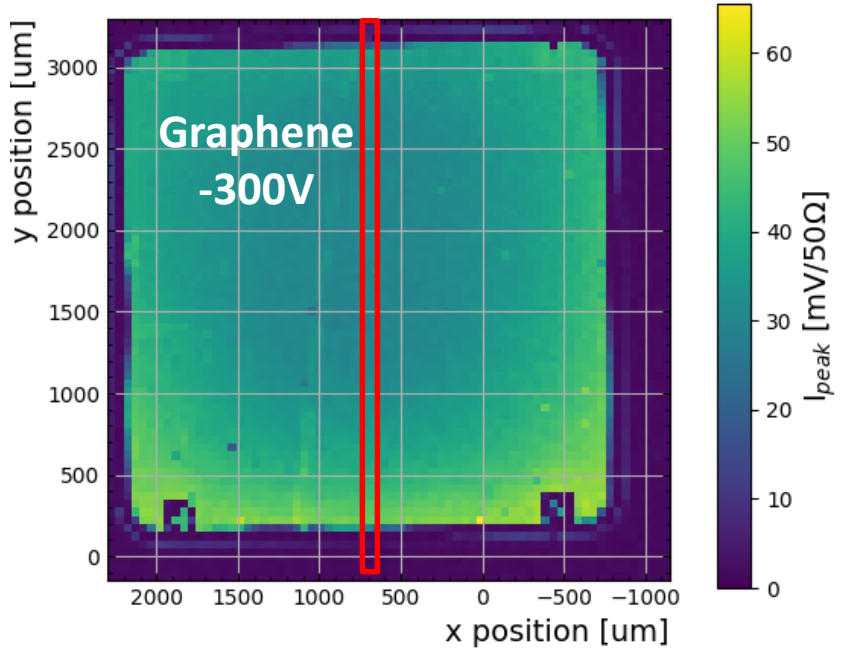
Effect of Graphene in charge collection

The **amplitude of the signals** are higher over most of the active region in the "Graphene" sample…





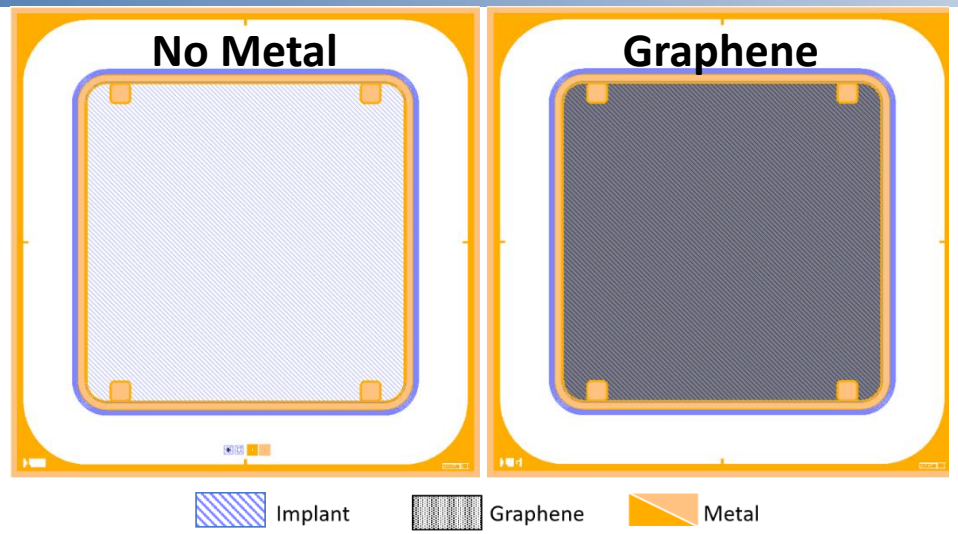




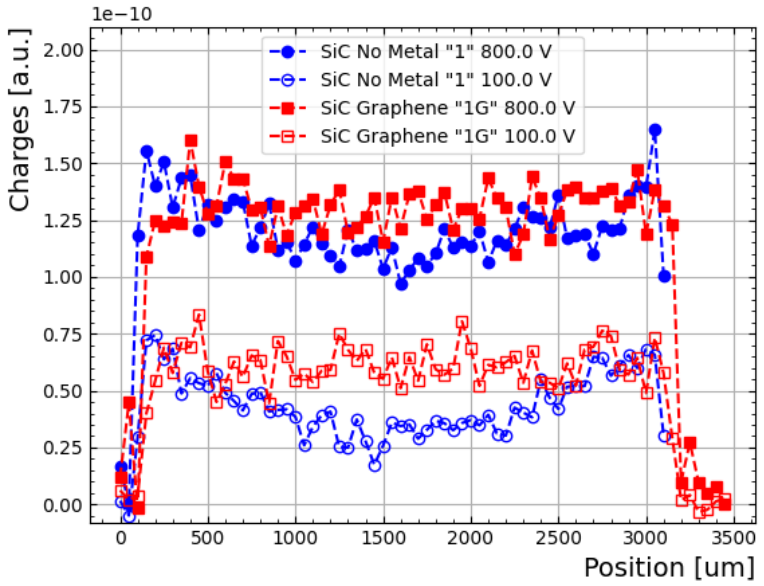
Effect of Graphene in charge collection

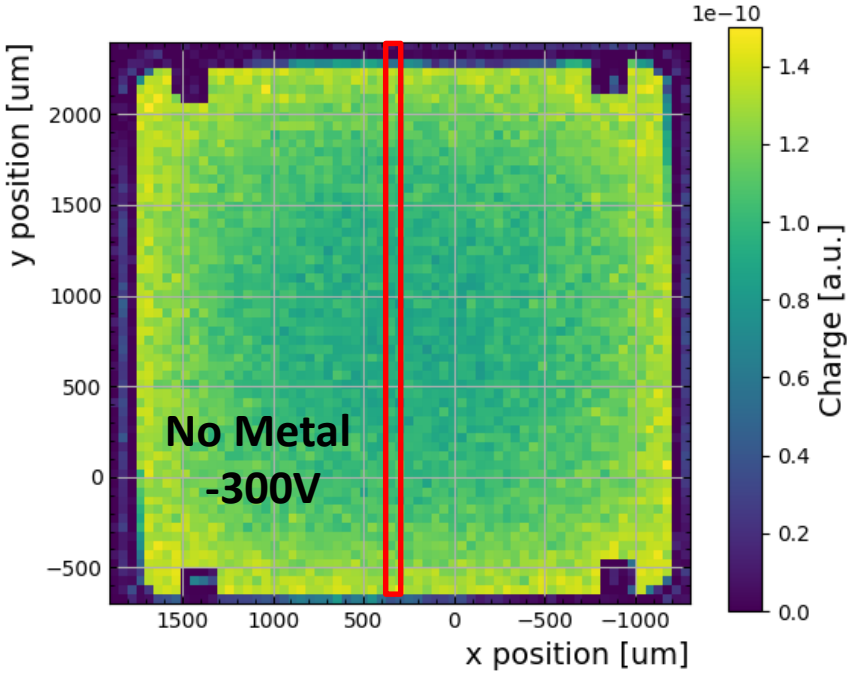
The **amplitude of the signals** are higher over most of the active region in the "Graphene" sample

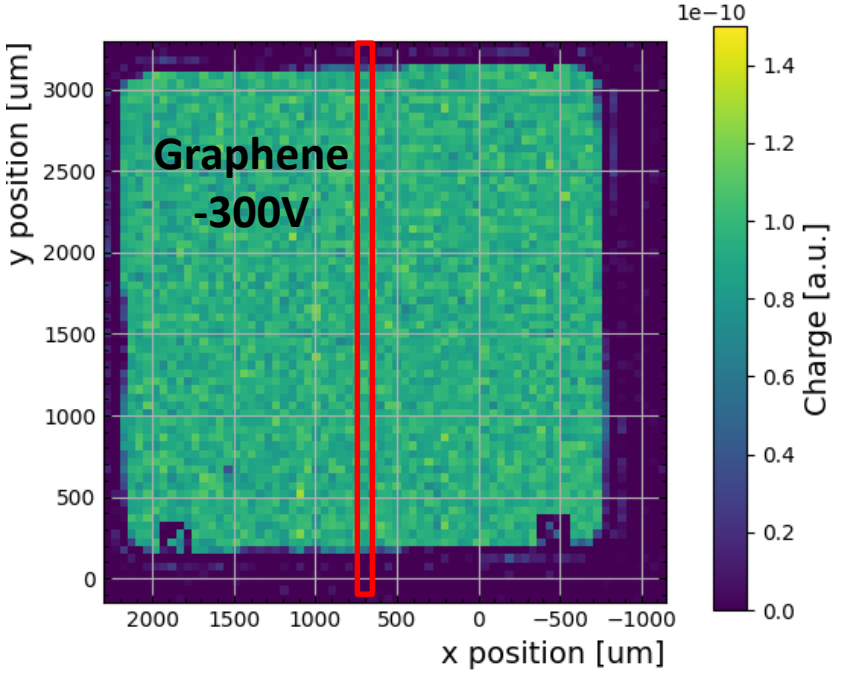
And uniform **charge collection** across samples provided large enough integration window in "No Metal" sample (more sensitive to noise).



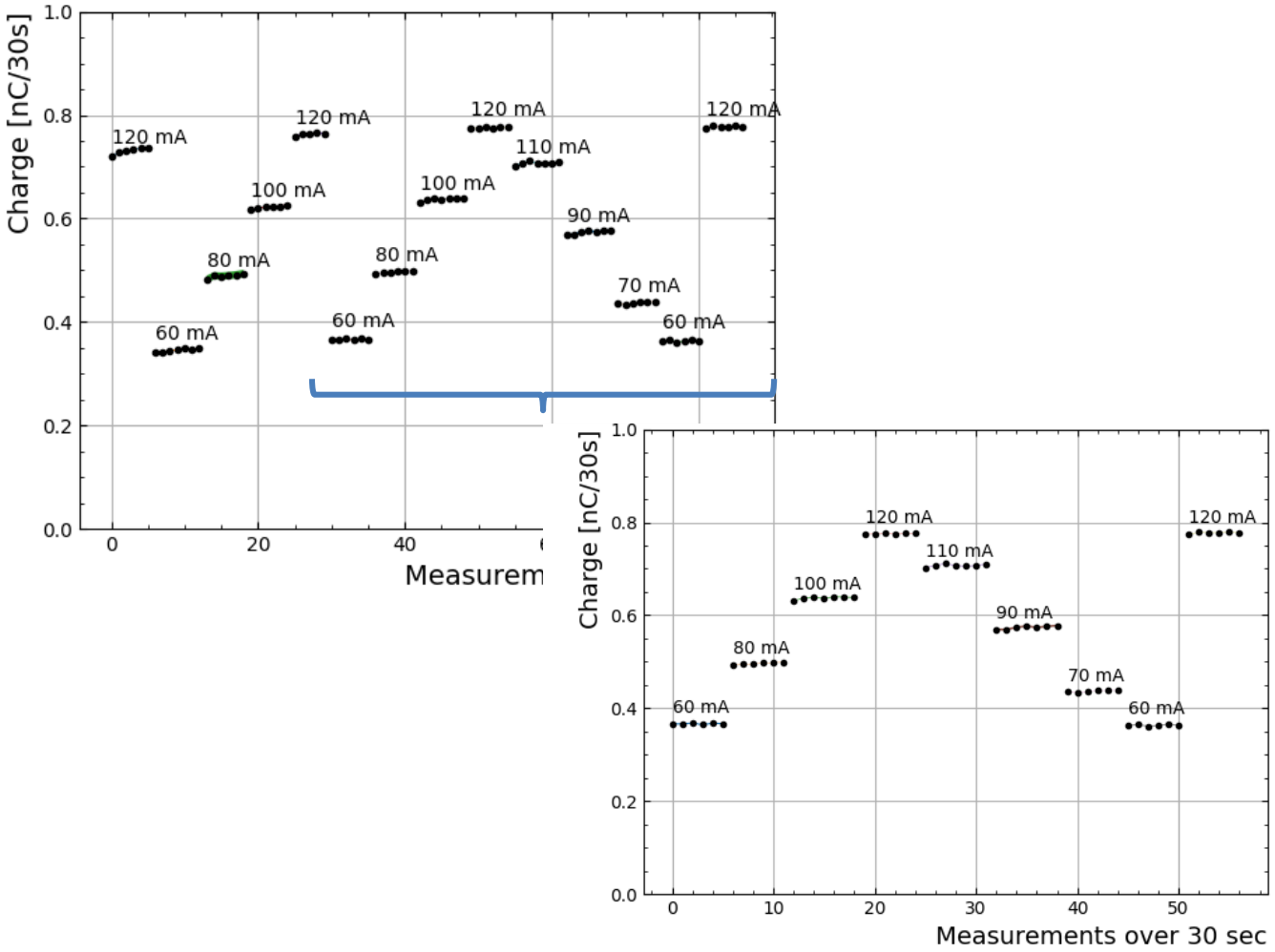
Graphene helps in making signals more uniform across the device over charge collection across implant (no metal)

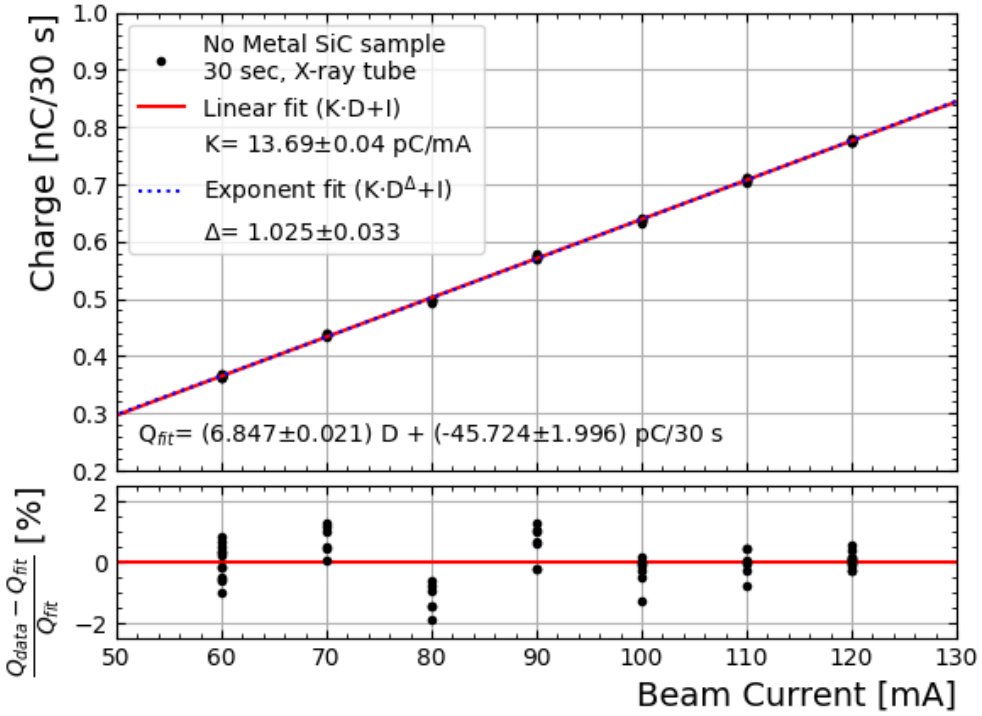






“No Metal” X-ray tube results



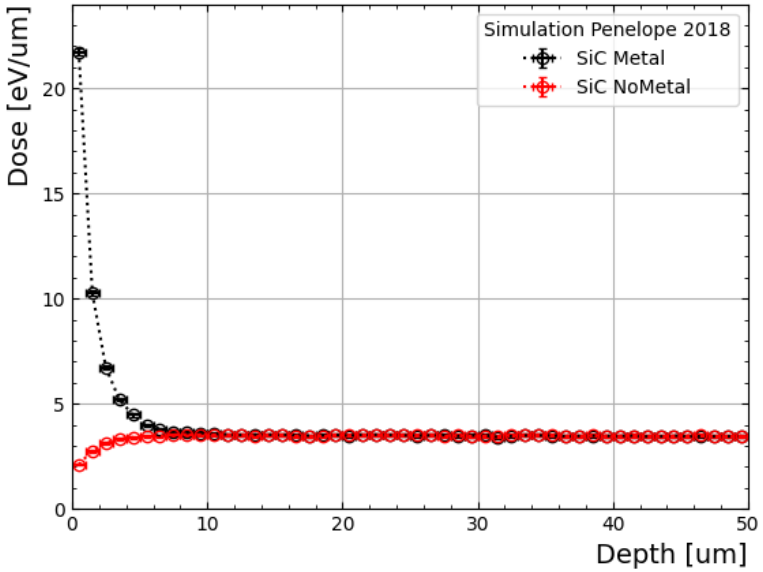


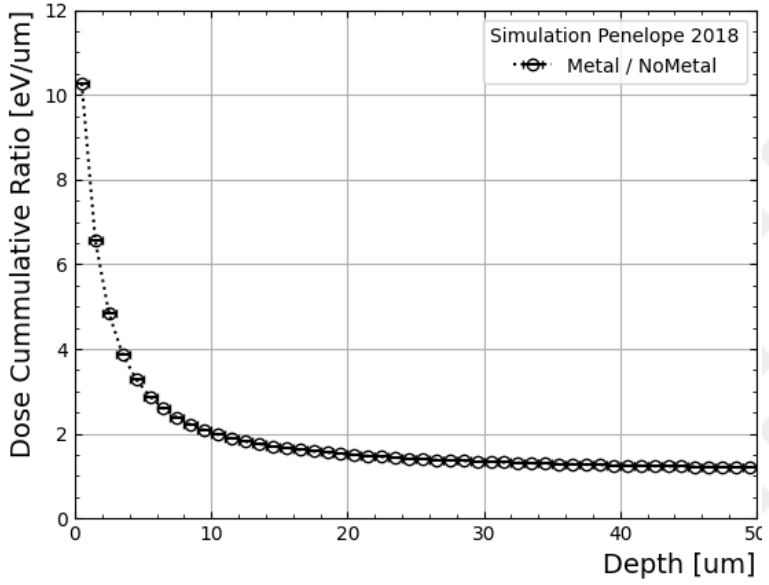
“No Metal” sample seems to struggle with linearity and stability

X-ray tube results

Simulation with Penelope 2018

- 1.2 mm Al, 10mm PMMA, Metal stack (when applicable), 50um+250 um SiC, Metal stack (Graphene is negligible)
- Results:
  - Dose in the first 5 um in the saple with metal contact is several times higher than that without metal, then othey are the same
  - Considering that 0V depletion distance is ~5-10 um (see TPA-TCT slides in RD50 Seville), the ratio of accumulated dose across that depth ranges between ~2.9 and ~ 2.1

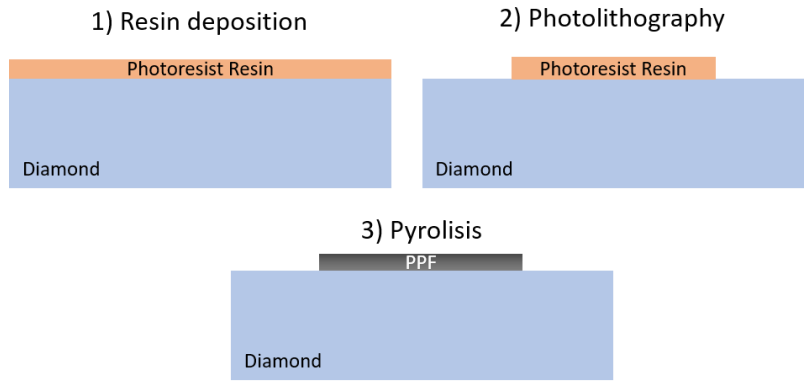




First proof-of-concept via PPF: Fabrication

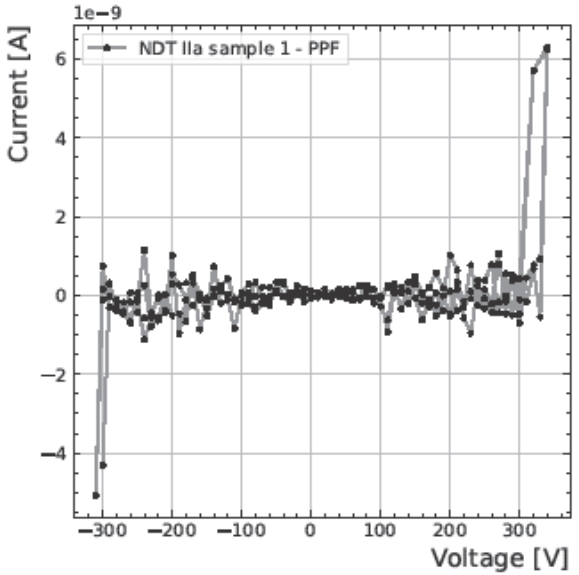
Pyrolitic Photoresist Film:

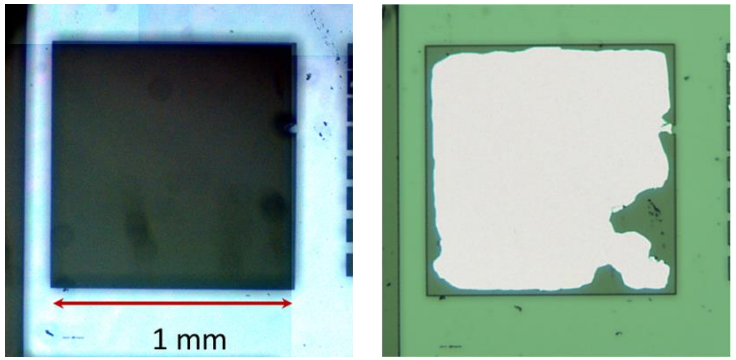
- Sublimate non-carbon components from resin with high temperatura



HPHT Type IIa diamond to produce proof-of-concept

- For wirebond connection add Al layer





Good electrical behaviour before packaging

First proof-of-concept via PPF: Fabrication

Pyrolitic Photoresist Film:

- Sublimate non-carbon components from resin with high temperature

HPHT Type IIa diamond to produce proof-of-concept

- For wirebond connection add Al layer

Silver paint to aid wirebond (low adhesión)

- … since then leakage current @ > 0 V very high, but works

First prototype fabricated!