RECENT DEVELOPMENTS IN THE FABRICATION OF PLANAR AND COAXIAL HPGE DETECTORS

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GAMMA RAYS DETECTION

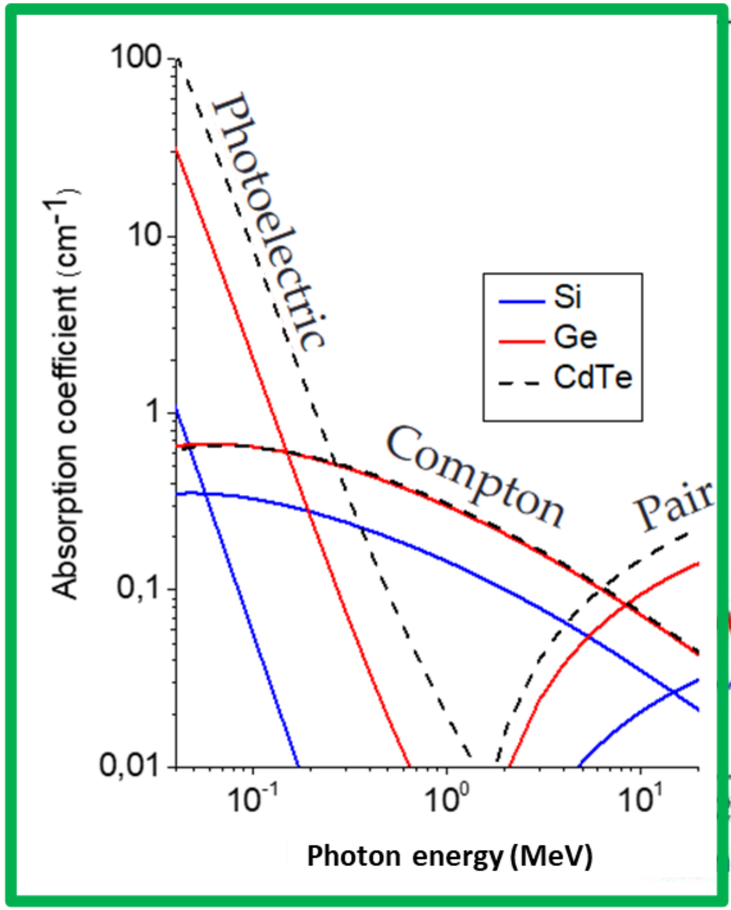
Why gamma-rays detection?

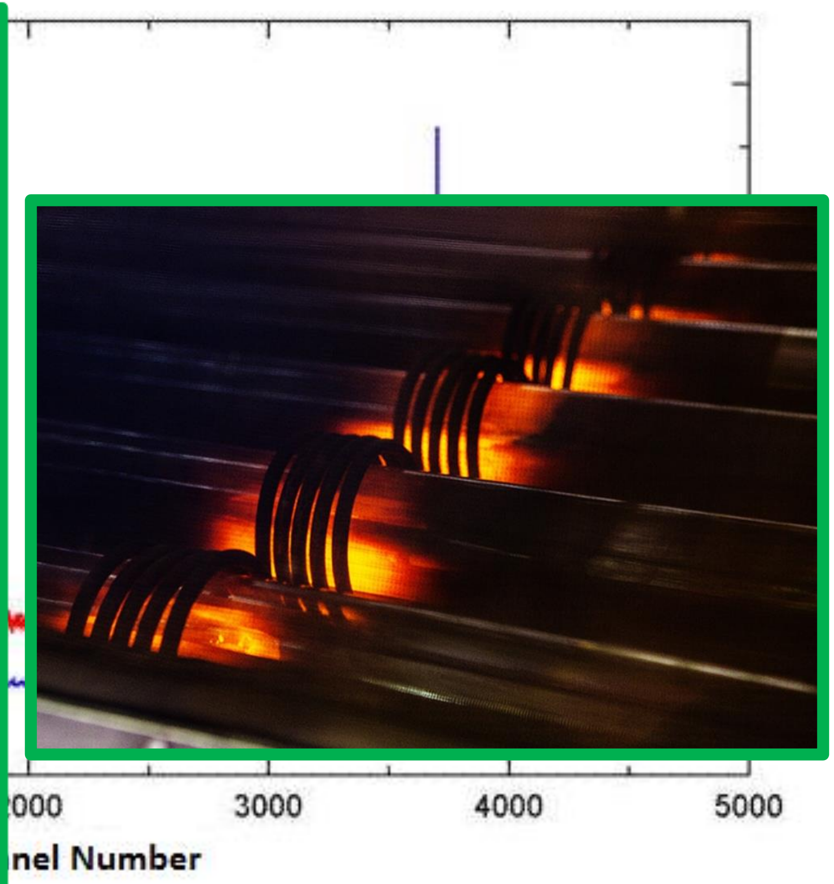
- Cosmic rays (satellites)
- Nuclear structure (large arrays)
- Medical (PET)
- Freight security (rad-portals)
- Radioactive sites (portable det.)
- Oil drilling
- Metal casting

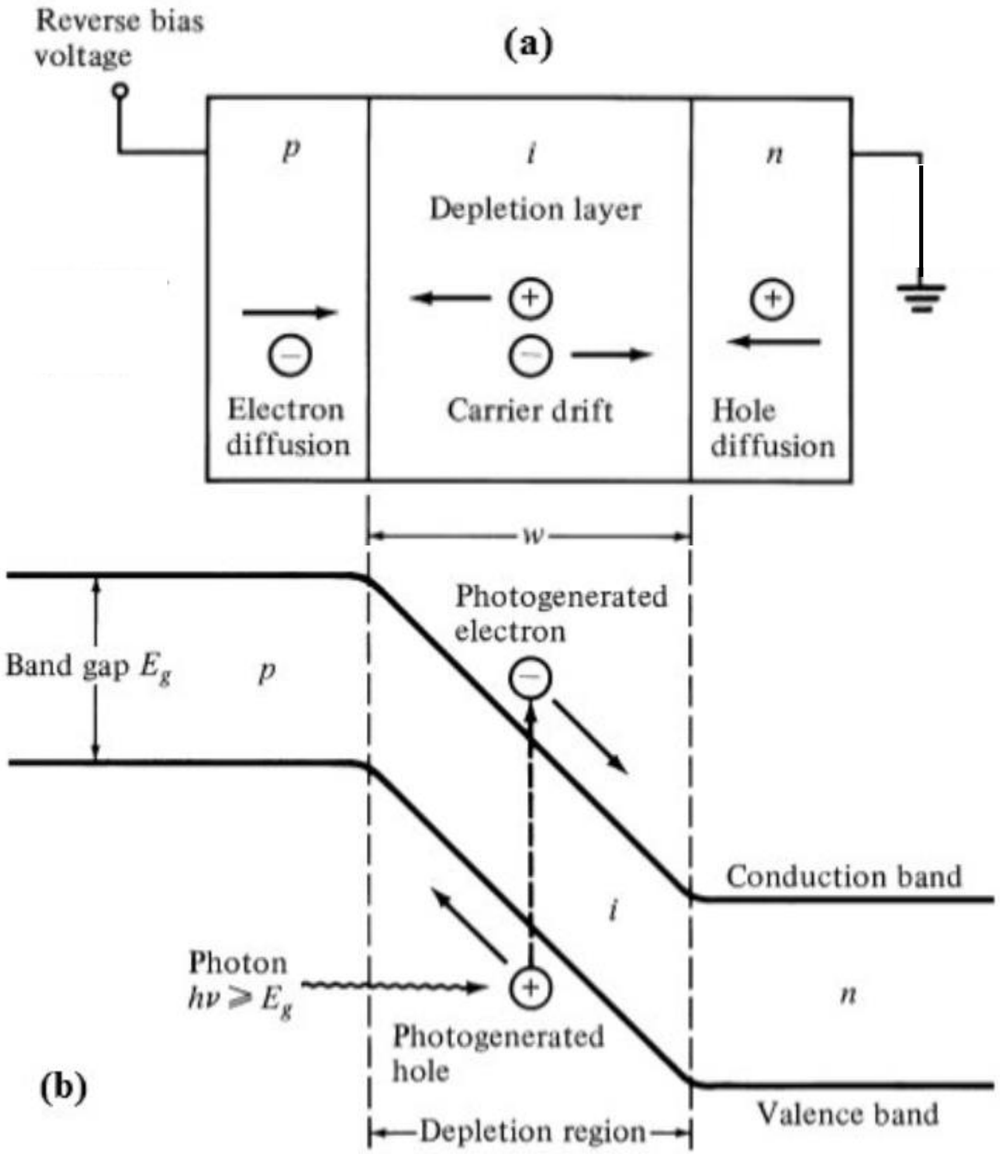
High Purity Germanium (HPGe) detectors

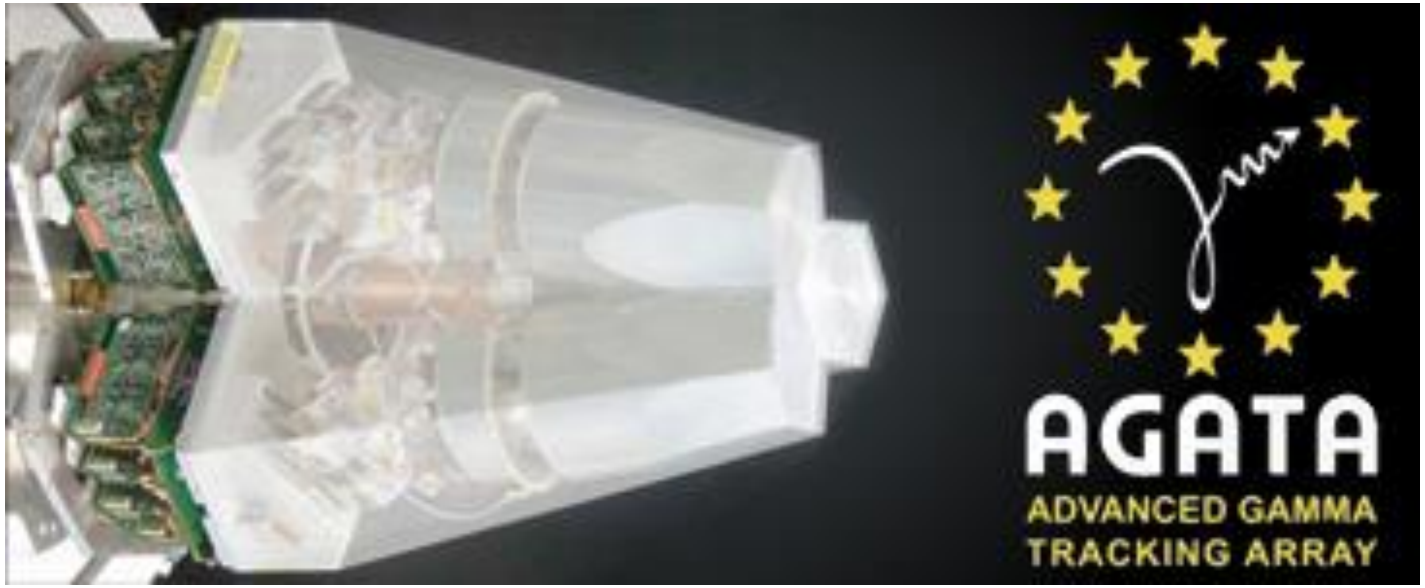
Why Germanium?

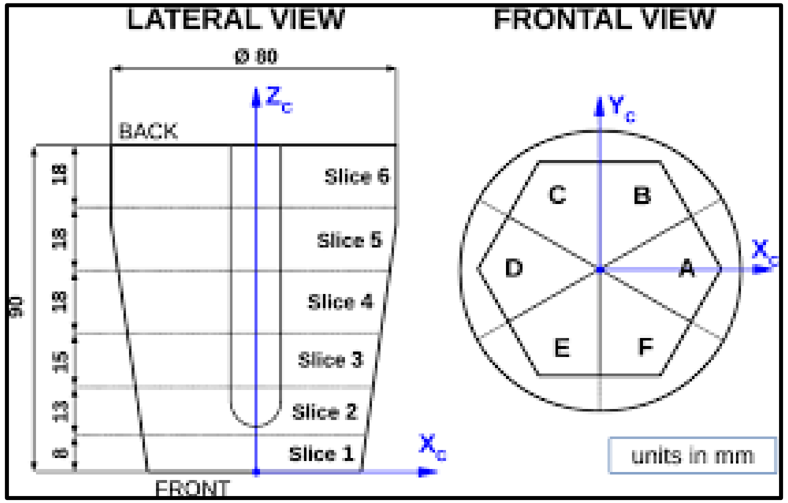
- Lowest Energy Gap: highest energy resolution among all detectors
- Moderate atomic number: high probability for gamma interaction
- Highest purification possible and larger active volumes

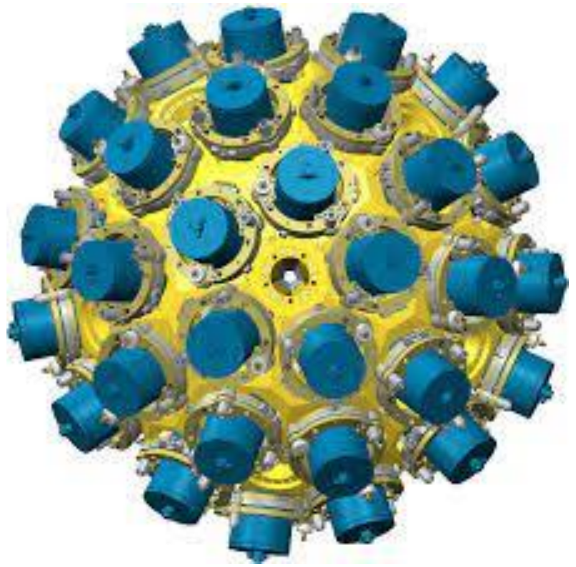




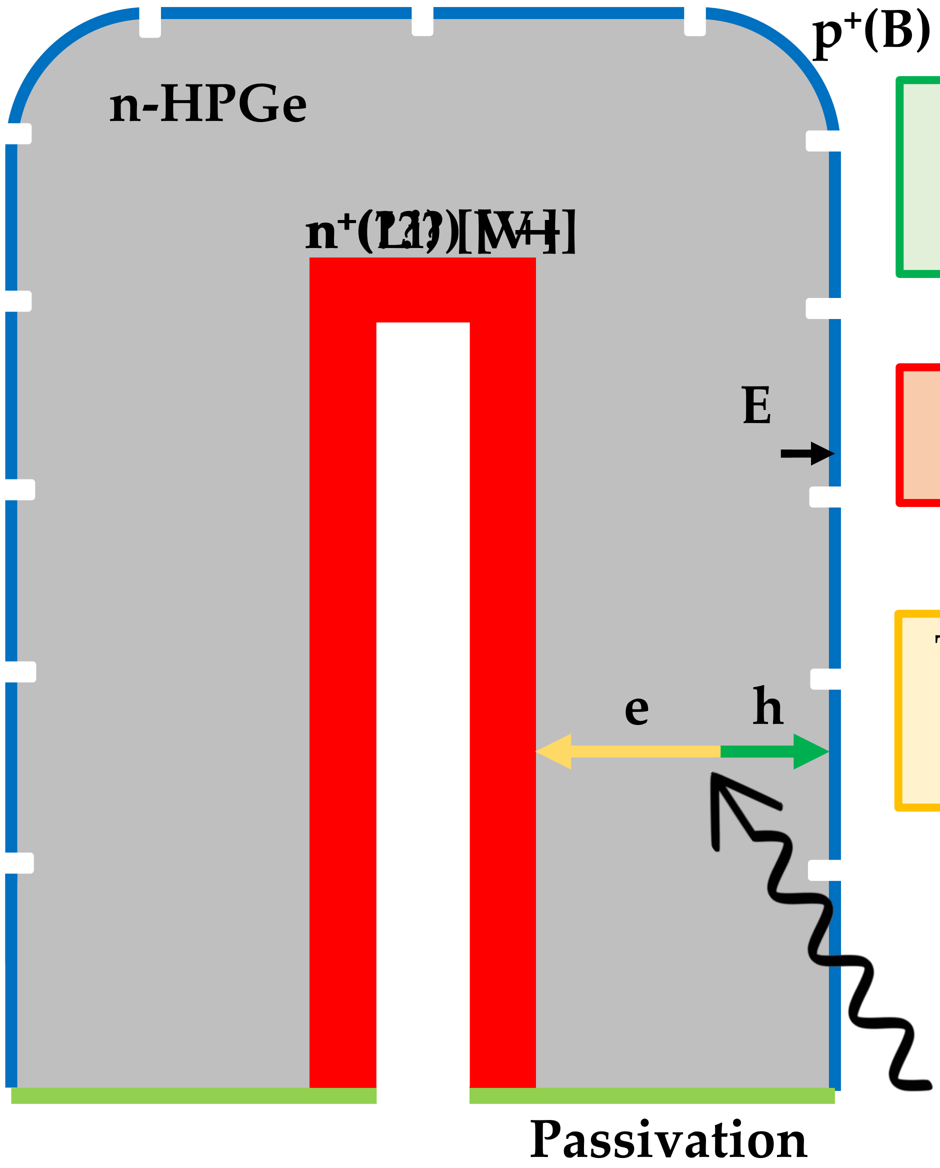

Reverse bias voltage
(a)
p
i
n
Depletion layer
Electron diffusion
Carrier drift
Hole diffusion
w
Photogenerated electron
Band gap E_g
p
Conduction band
i
Photon $h\nu \geq E_g$
n
Photogenerated hole
(b)
Depletion region
Valence band


AGATA
ADVANCED GAMMA TRACKING ARRAY


LATERAL VIEW
FRONTAL VIEW
Ø 80
BACK
Slice 6
Slice 5
Slice 4
Slice 3
Slice 2
Slice 1
FRONT
90
18
18
18
15
13
8
C
B
D
A
E
F
units in mm



State of the art



Boron junctions are thin, thermally stable and easily segmentable on n-HPGe.

↓

Lithium junctions are thick and thermally unstable.

↓

Thin and thermally stable n-type dopants (Pulsed Laser Melting).

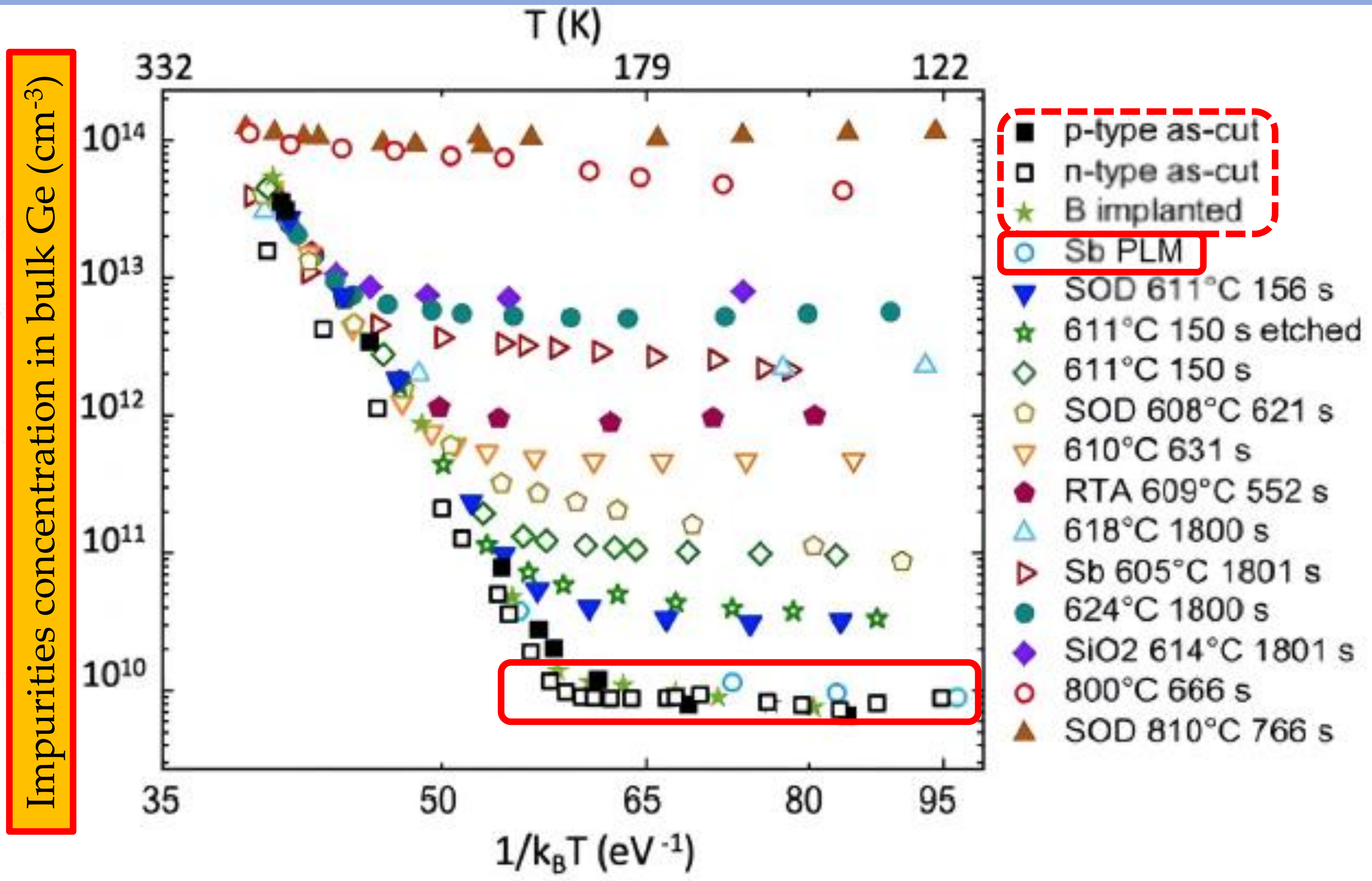
↓

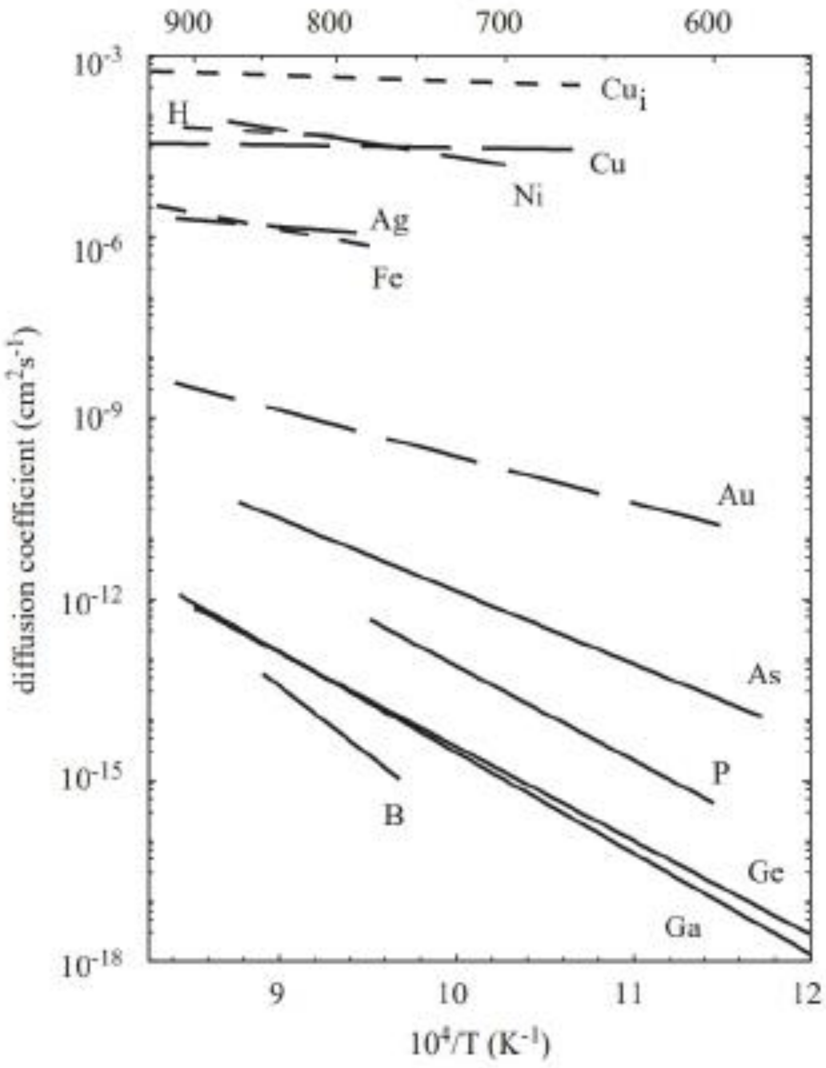
Expansion of these techniques to p+ dopants.

Strongly Out-Of-Equilibrium diffusion process

- Ultrafast (10^{-7} s)
- Hyperdoping ($>10^{20}$ at/cm^3)
- Dopant flexibility
- Thin junction (<1 μm)
- Surface-limited heating (<10 μm)

State of the art



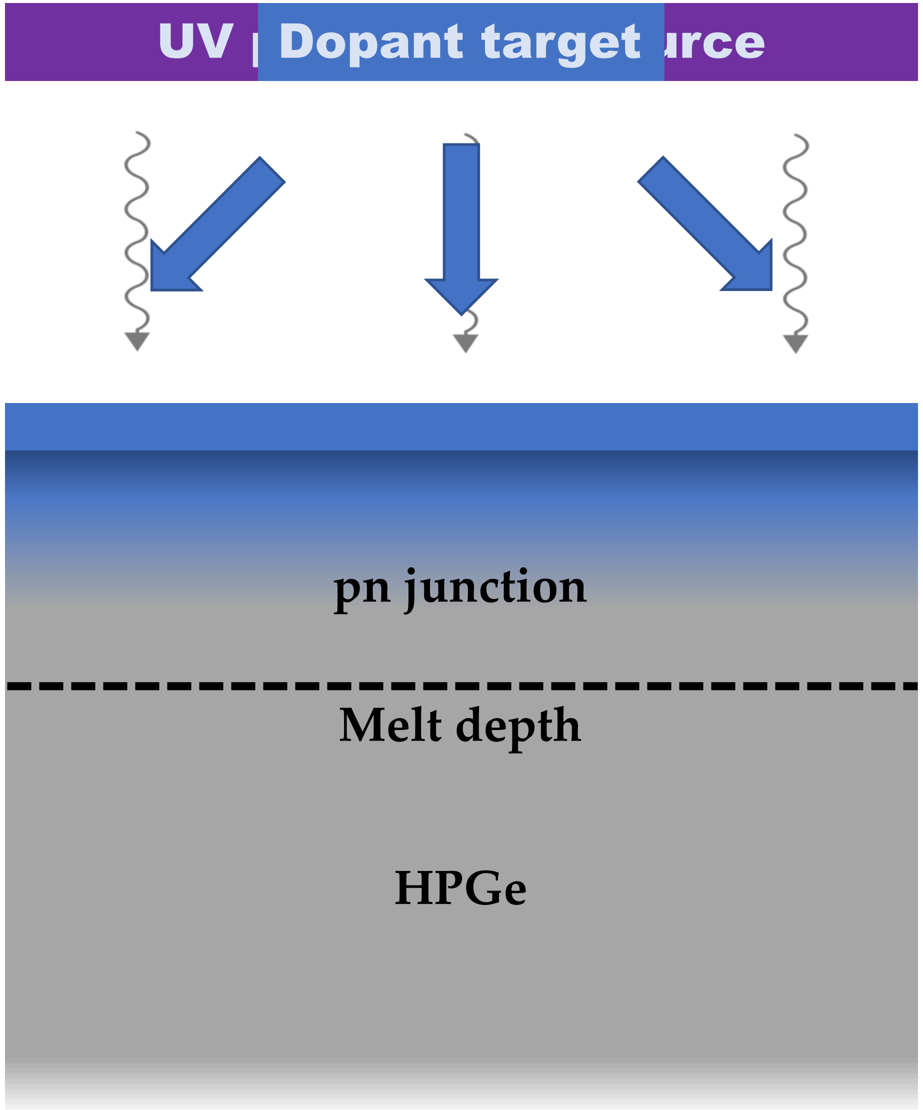


V.Boldrini et al. *Characterization and modeling of thermally-induced doping contaminants in high-purity Germanium* .J. Phys. D: Appl. Phys. 52(3), 035104 (2018)

- Introduction
- State of the art
- Process Overview
- Small thin samples
- Large thin samples
- Process Upgrades
- Thick planar detectors
- Coaxial detectors
- Conclusions

- Fabrication steps
- Characterization

Process overview



Polished HPGe surface

Sawing damage removal via alumina grinding and final polishing via wet etching

Dopant sputtering deposition

Few nm deposition of n-dopant or p-dopant in Ar plasma with ultrapure targets in vacuum (10^{-7} mbar)

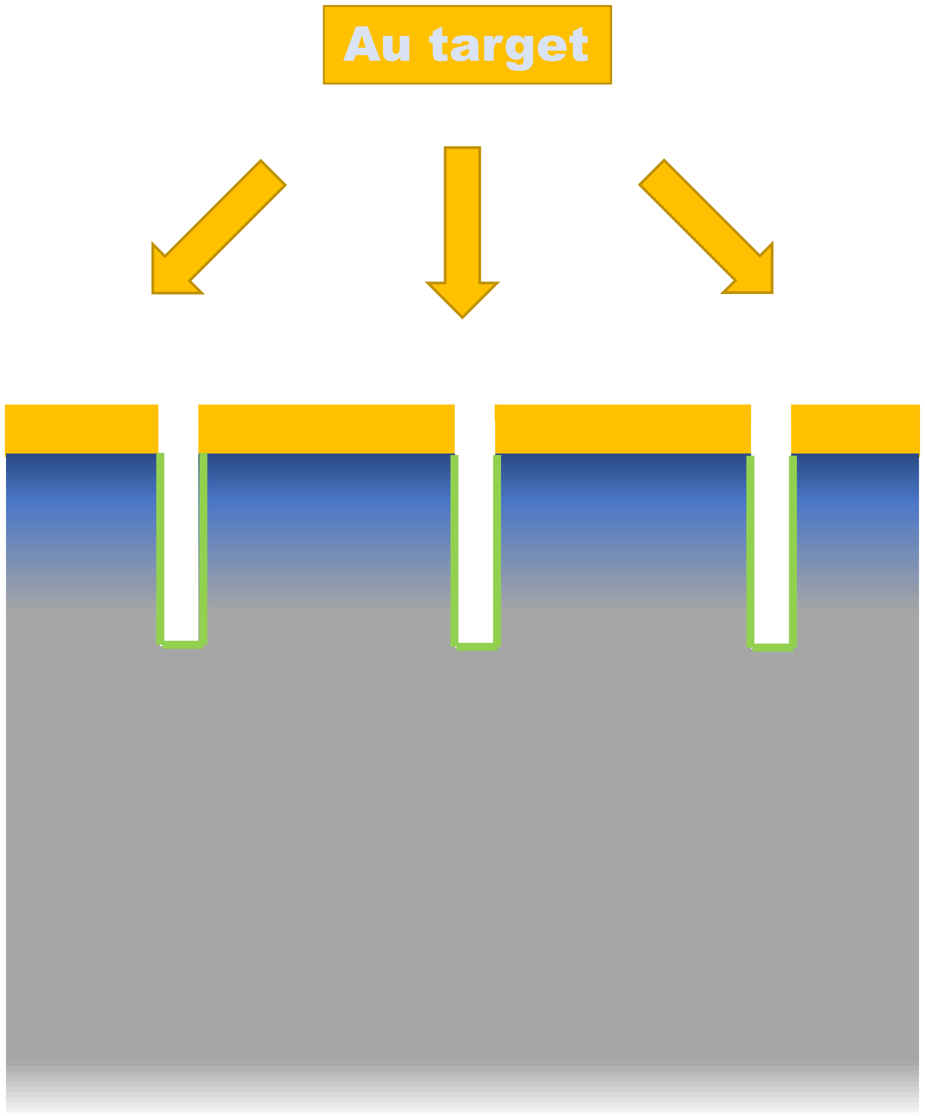
UV pulsed laser exposure

25 ns pulses at 248 nm wavelength to melt 150 nm of HPGe

Crystal regrowth

100 ns resolidification of germanium with dopant atoms in the crystalline structure

Process overview



Au deposition
100 nm deposition of Au in Ar plasma with ultrapure target in vacuum (10^{-7} mbar)

Photolithography
Photoresist deposition, baking, exposure and development, followed by Au stripping and resist removal.

Intercontact gaps passivation
3:1 HNO3 : HF etching followed by chemical quenching passivation.

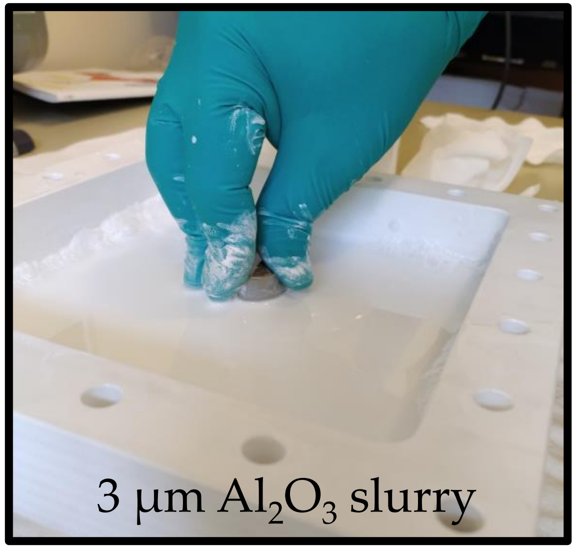
1^{st} step: crystal polishing

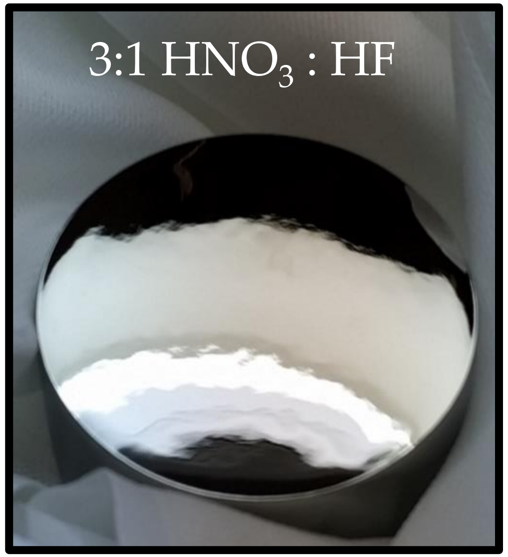
Grinding

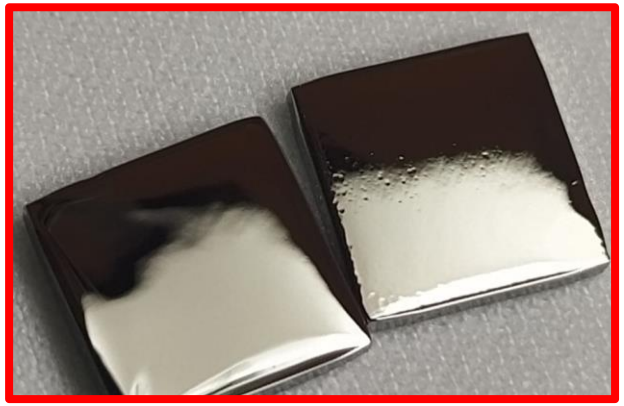
-Grinding with 3 μm Al_2O_3 slurry to remove sawing damages: the abraded thickness depends on last process.

Polishing

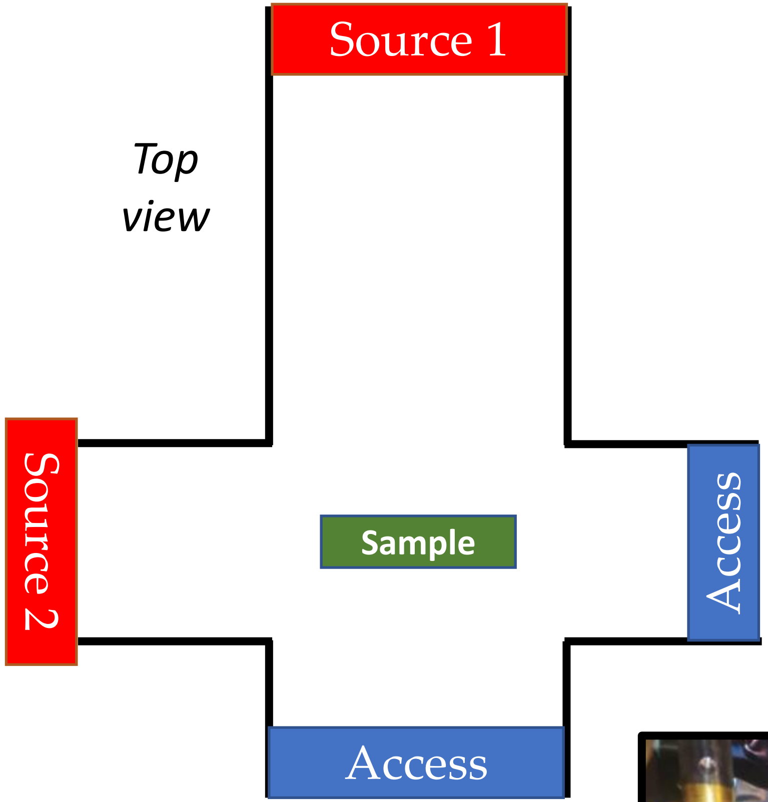
-Wet etching (3:1 HNO_3 : HF) for several minutes: 20 μm/min erosion rate.

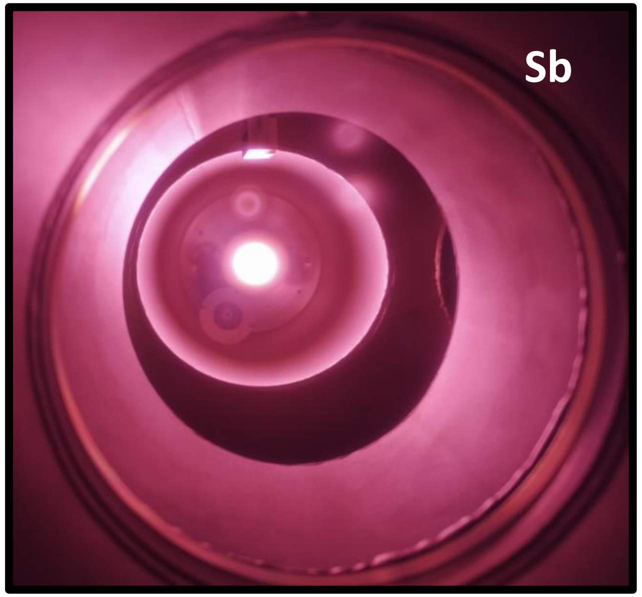






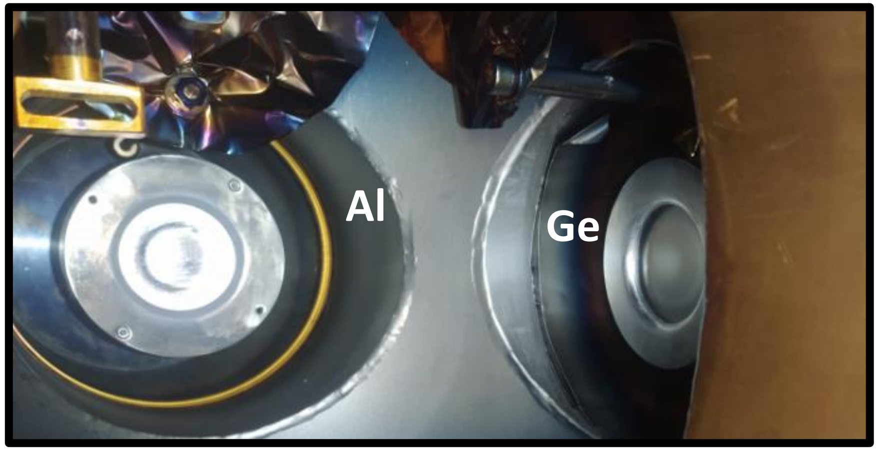
2^{nd} step: dopant deposition





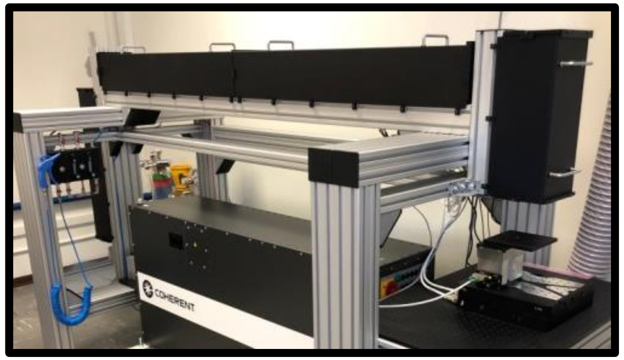
n^+	Sb	2 nm
p^+	Al (Ge cap)	4 nm (10 nm)

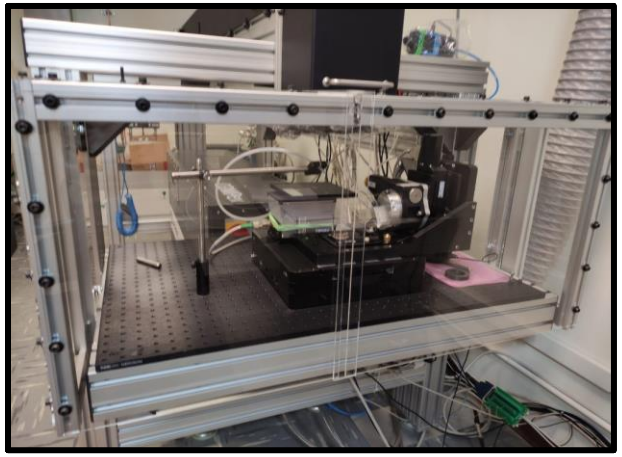
samples,two linear axes, 90° joint for sample tilting.



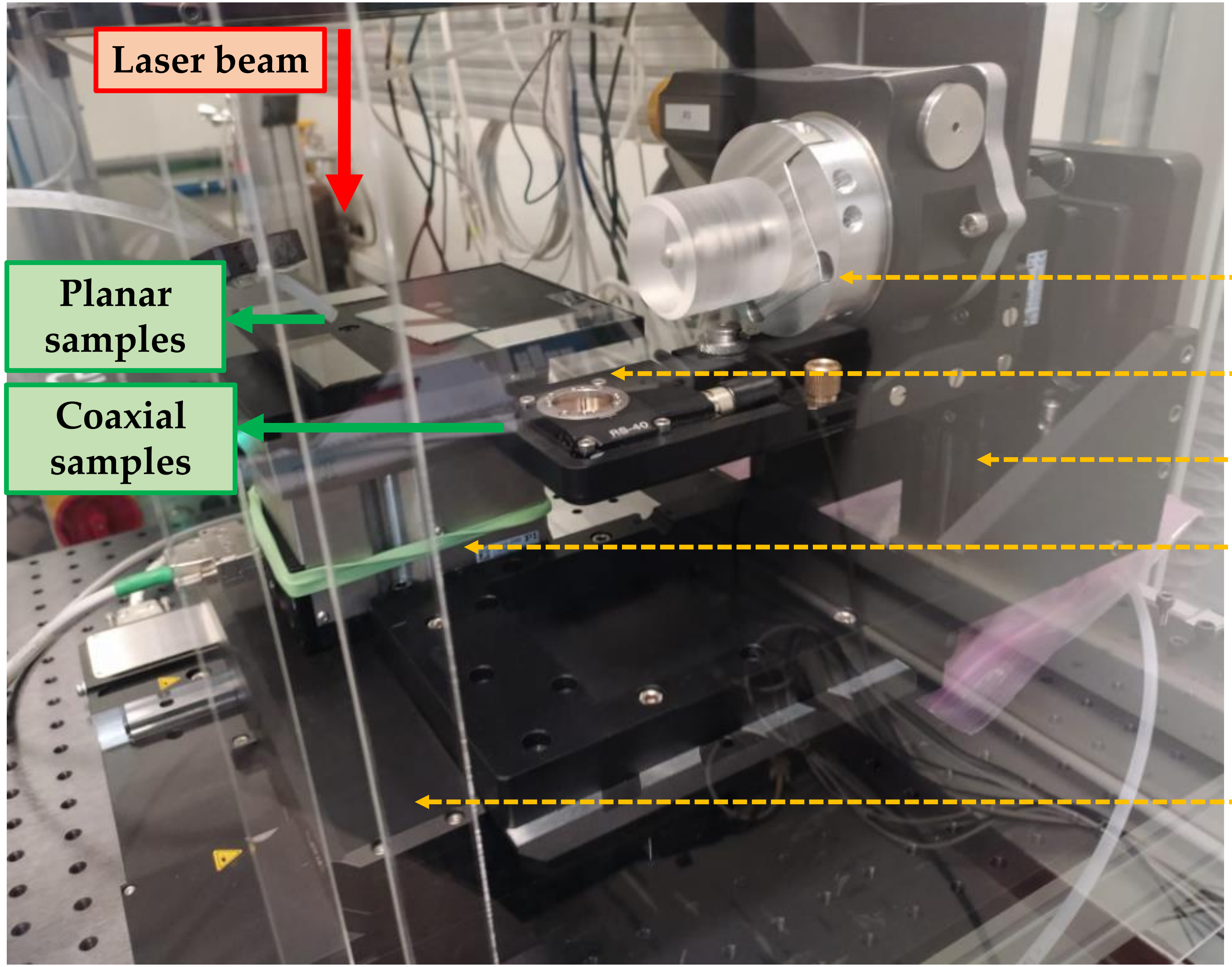
3rd step: Pulsed Laser Melting

248 nm, 25 ns pulse
KrF laser (Coherent)



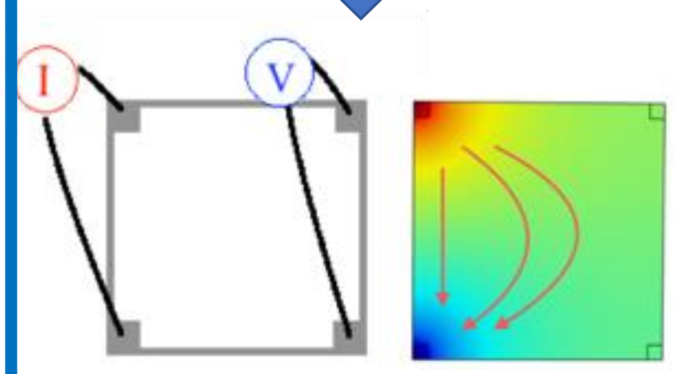


6 motorized DOF



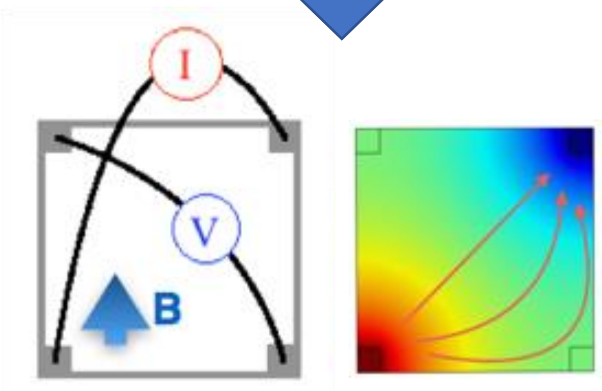
3rd step: Pulsed Laser Melting

Van Der Pauw



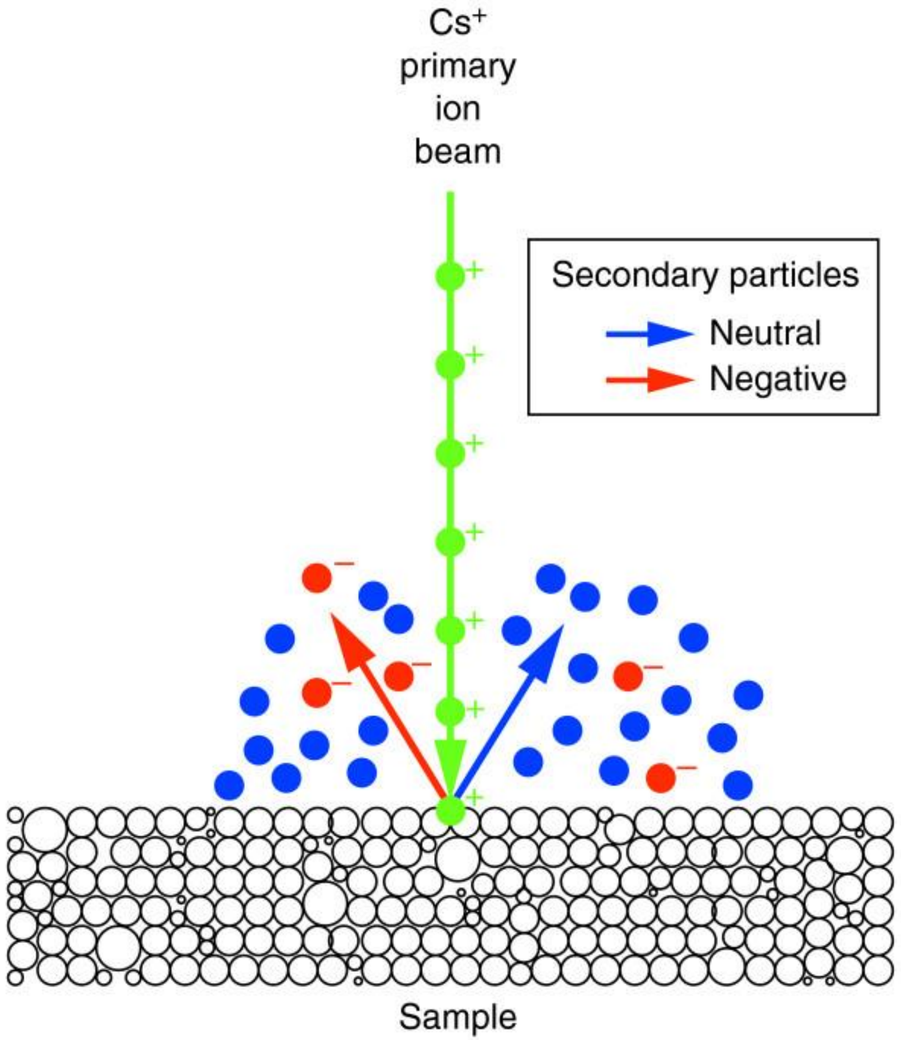
Sheet resistance

Hall

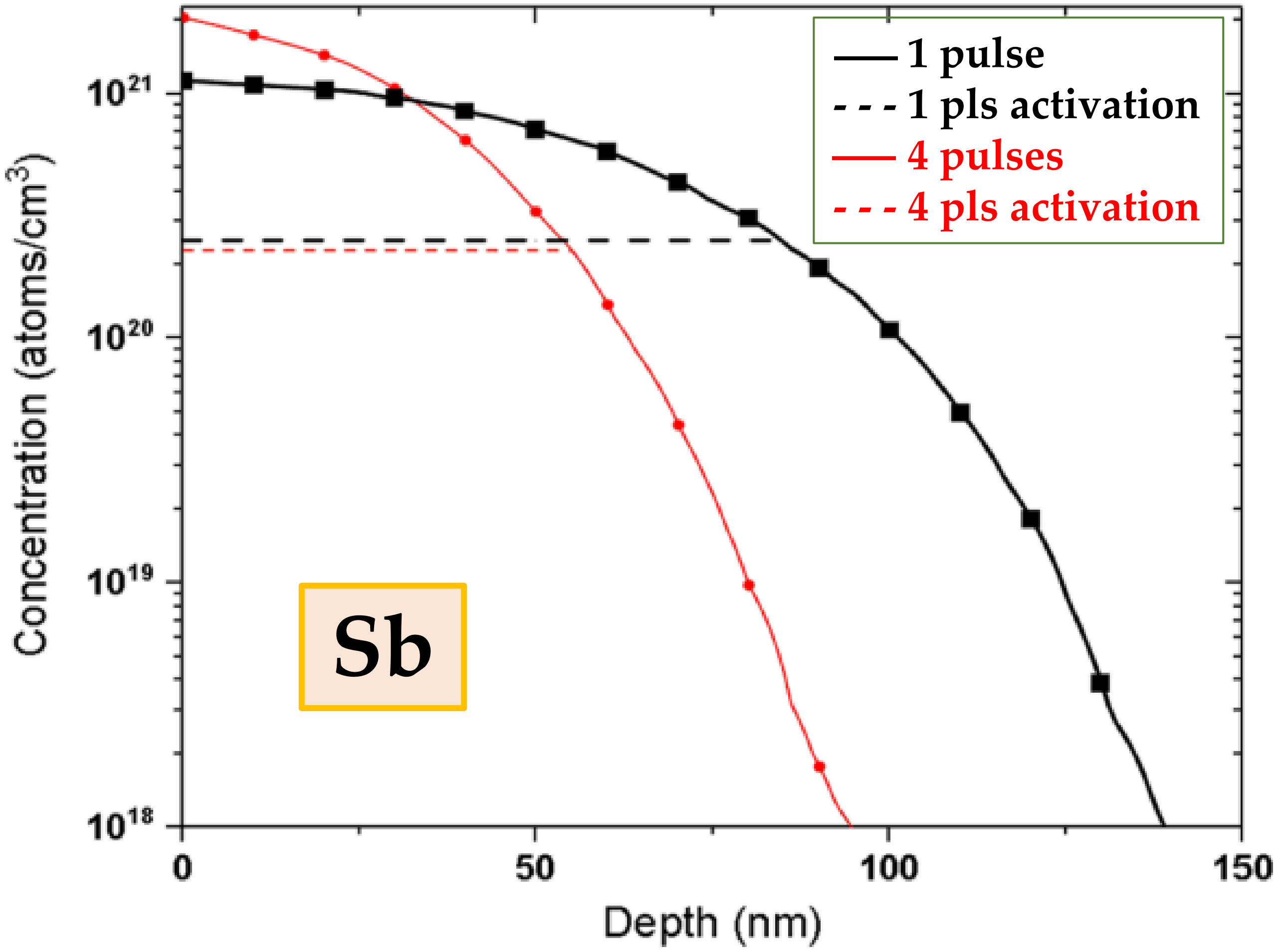


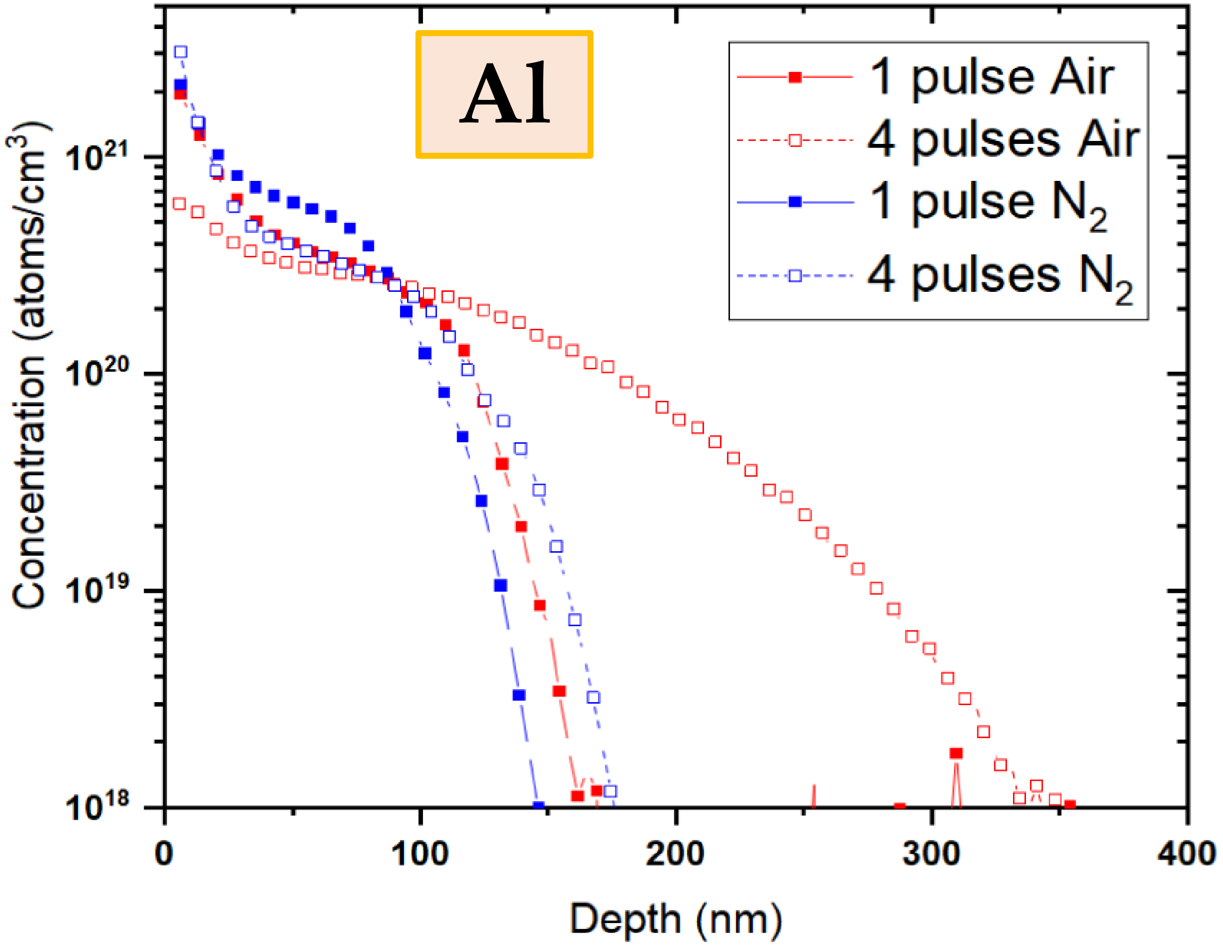
Charge carriers

Secondary Ions Mass Spectrometry



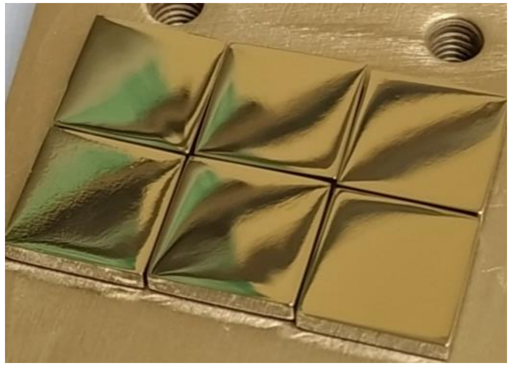
3^{rd} step: Pulsed Laser Melting





C. Carraro et al., "N-type heavy doping with ultralow resistivity in Ge by Sb deposition and pulsed laser melting", *Applied Surface Science*, ***509*** (2020)

Au sputtering

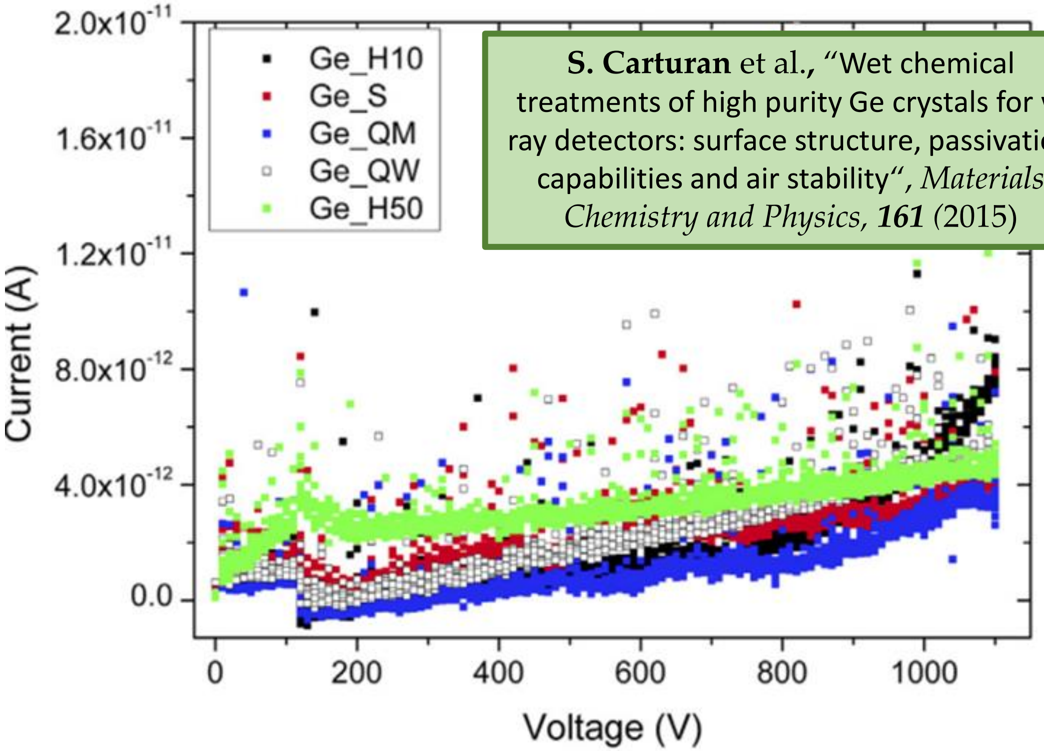


Photolithographic process:
1) Positive resist spin coating
2) Softbake
3) Exposure through acetate mask
4) Development
5) Gold stripping
6) Resist stripping
7) Trenching: strong acid attack (HNO_3 , HF)

5th step: passivation

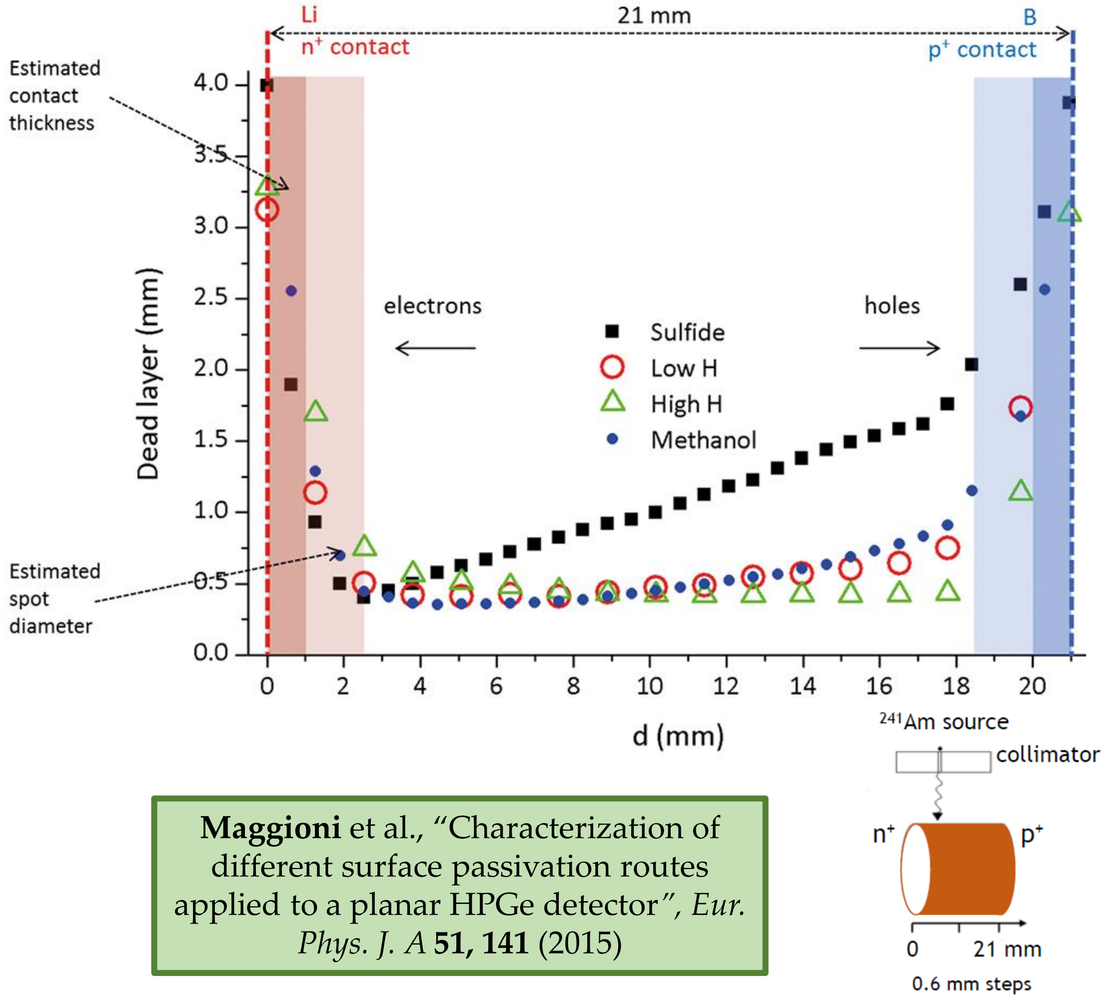
Trenching.

Quenching:
after the acid attack, rinse the sample in Methanol to saturate chemically active surfaces.
Methoxide-terminated Ge shows good surface insulation and stability



S. Carturan et al.**,** "Wet chemical treatments of high purity Ge crystals for γ-ray detectors: surface structure, passivation capabilities and air stability", *Materials Chemistry and Physics*, ***161*** (2015)

Dead layer estimation



Maggioni et al., "Characterization of different surface passivation routes applied to a planar HPGe detector", *Eur. Phys. J. A* **51, 141** (2015)

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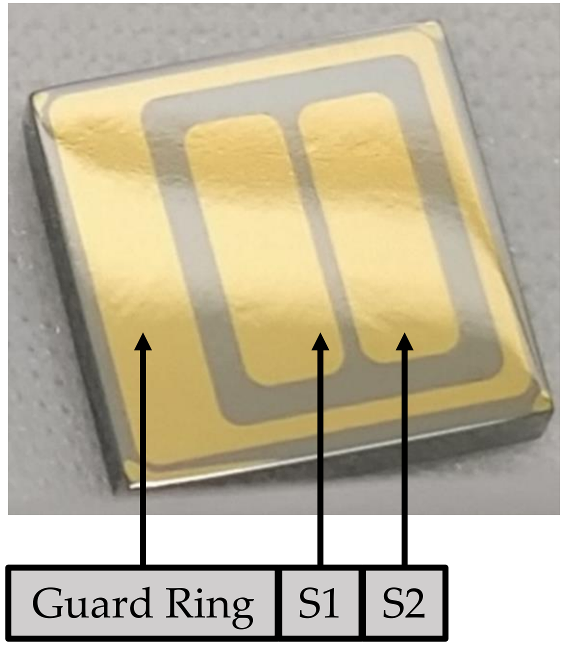
- Results
- Simulation

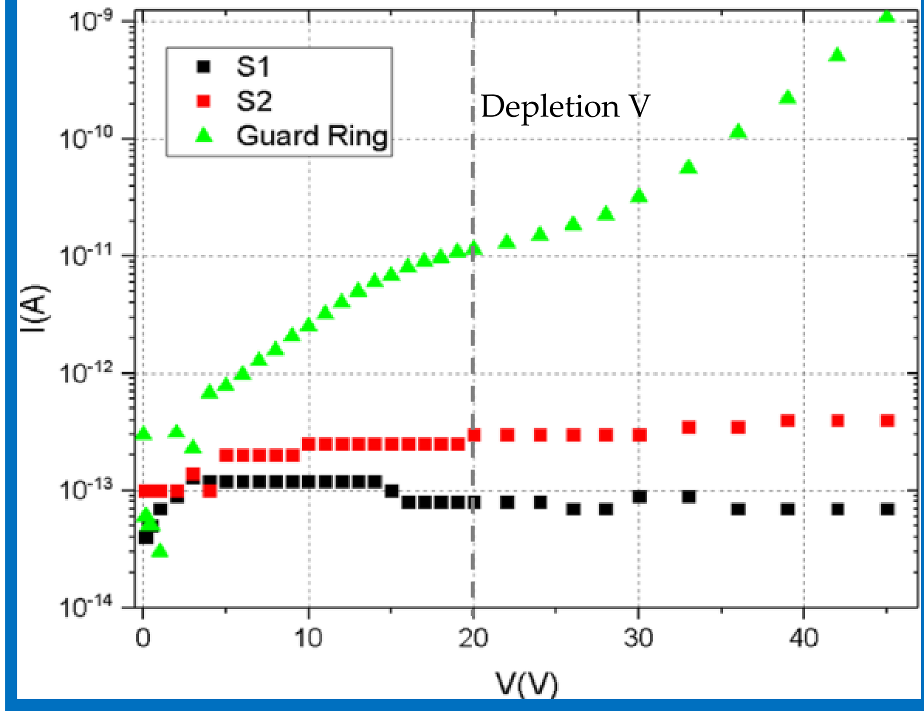
Small thin planar detectors

Sb junction, p-type, L=10mm, t=2mm

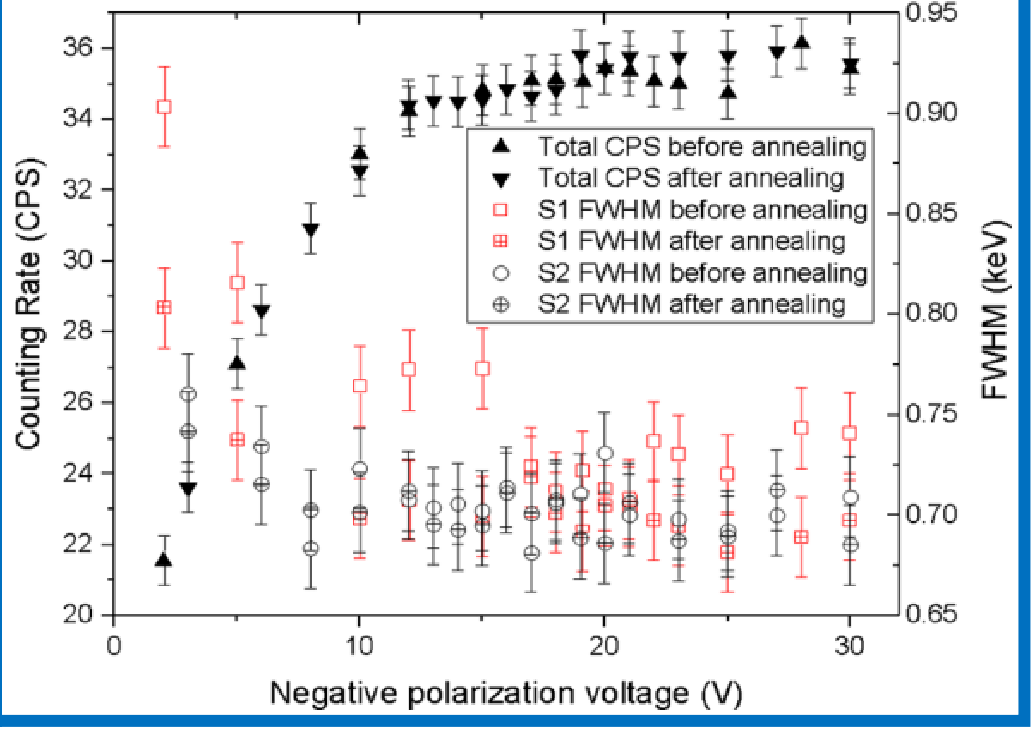


Measured before and after thermal annealing and re-passivation to test junction stability

Electrical test:
reverse I-V characteristics and resistance between contacts.



Spectroscopical test:
^{241}Am spectra acquisition.



S. Bertoldo et al. **“New method for the production of thin and stable, segmented n+ contacts in HPGe detectors”**, *Eur. Phys. J. A* (2021) 57:177

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Simulation

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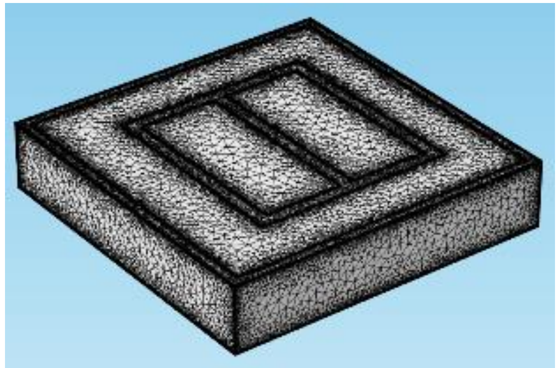
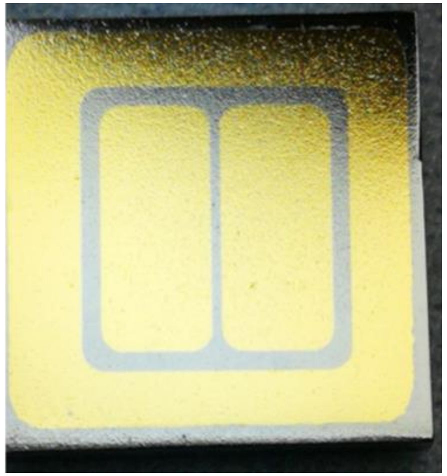




Geometry & mesh:

Electrostatic system

Electrodynamic system

Electrostatic system: weighting fields

Ordinary differential equations

Depletion process

Surface charges effects

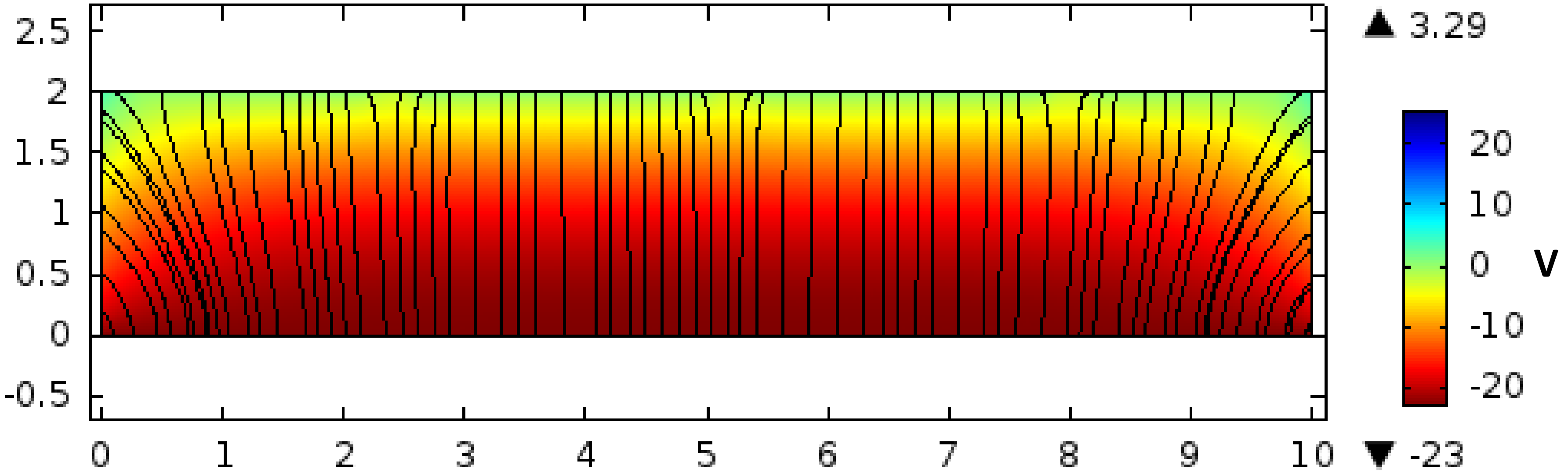
Capacitances

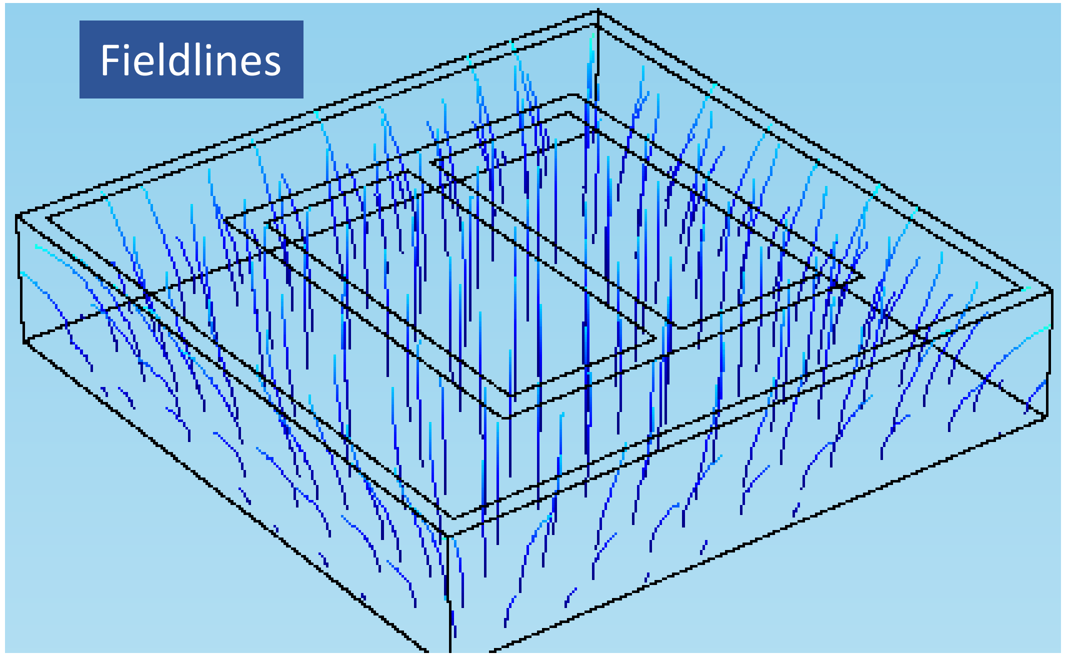
Leakage currents

Signals simulation

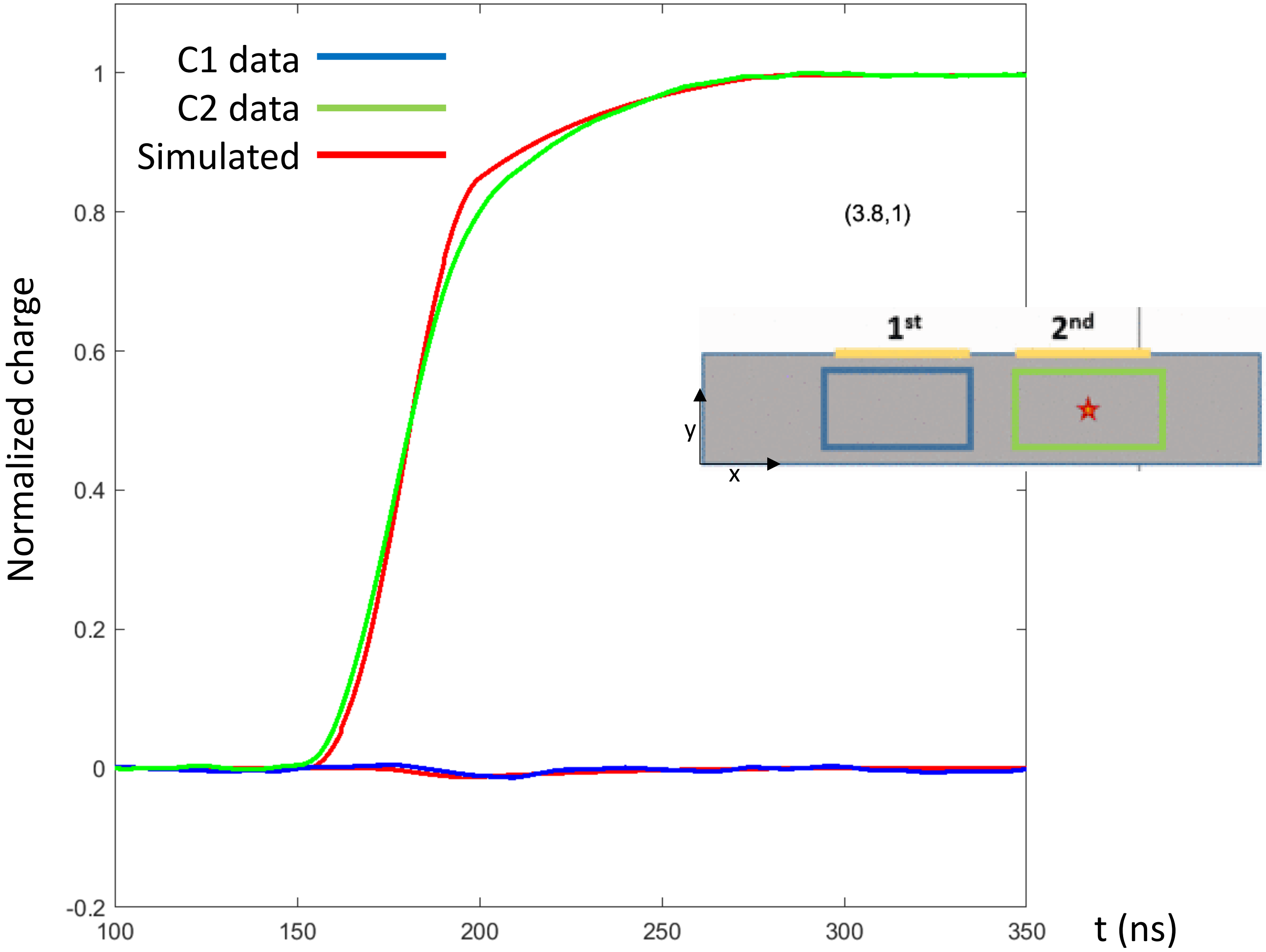
Simulation

$N_s = 10^9$ el/cm^2





Simulation



C1 data
C2 data
Simulated
Normalized charge
t (ns)
(3.8,1)
1st
2nd
y
x
1
0.8
0.6
0.4
0.2
0
-0.2
100
150
200
250
300
350

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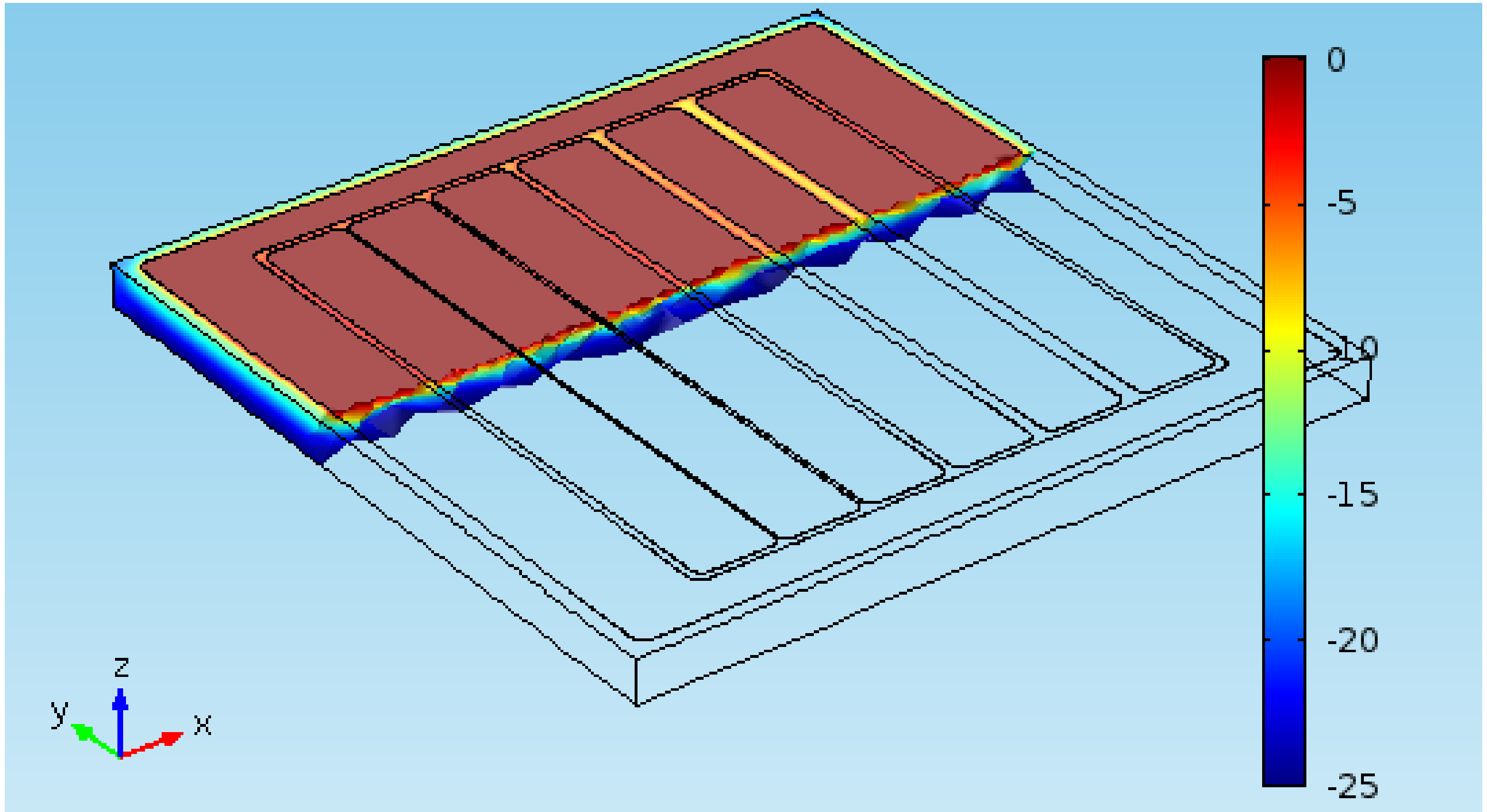
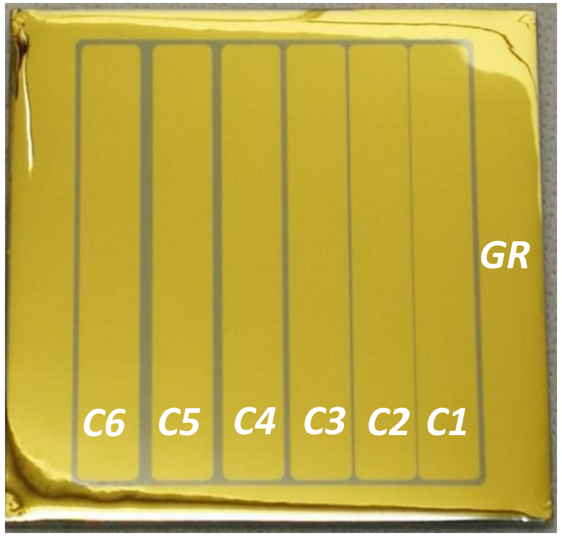
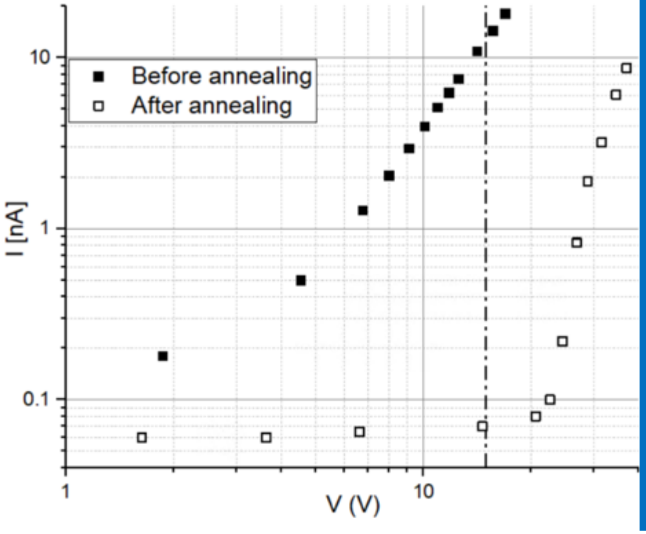
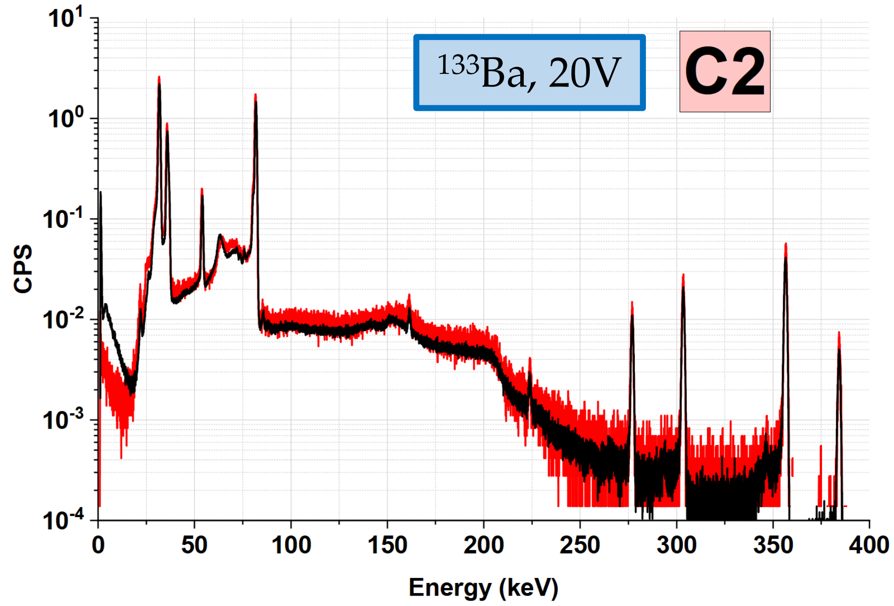


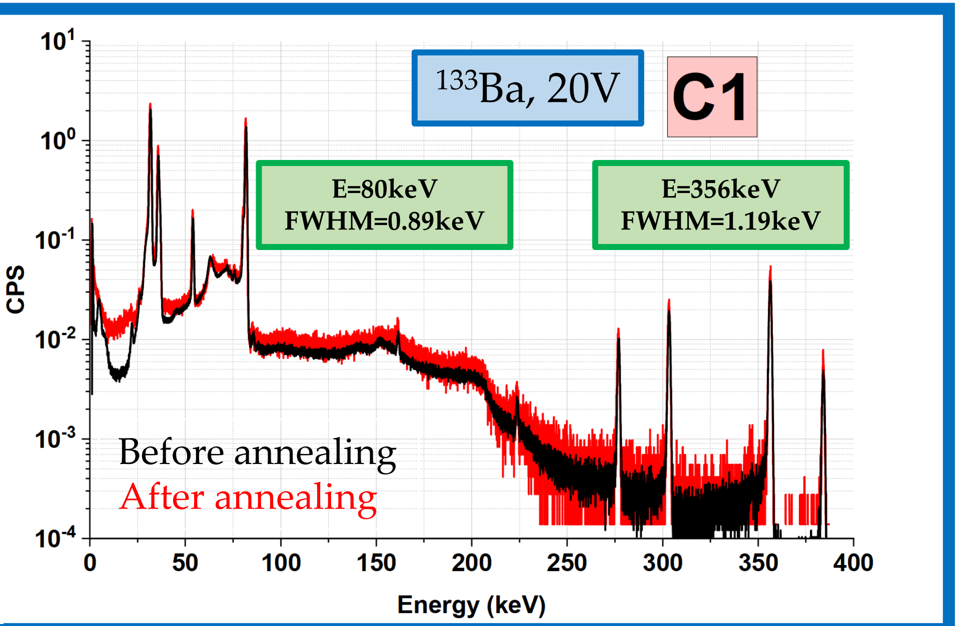
Large thin planar detectors

Sb/p-HPGE/Al, L=35mm, t=1.6mm

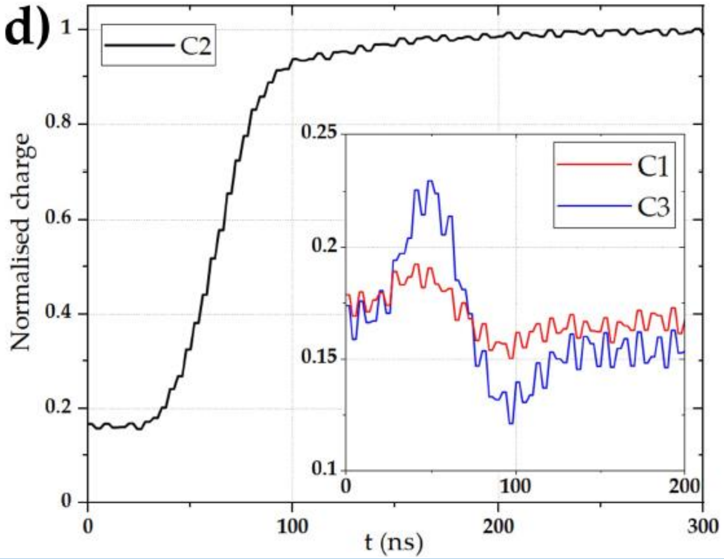








^{60}Co gamma rays hitting C2 and inducing transient signals on neighbors segments C1 and C3: from Pulse Shape Analysis it is possible to recover the exact location of the event.



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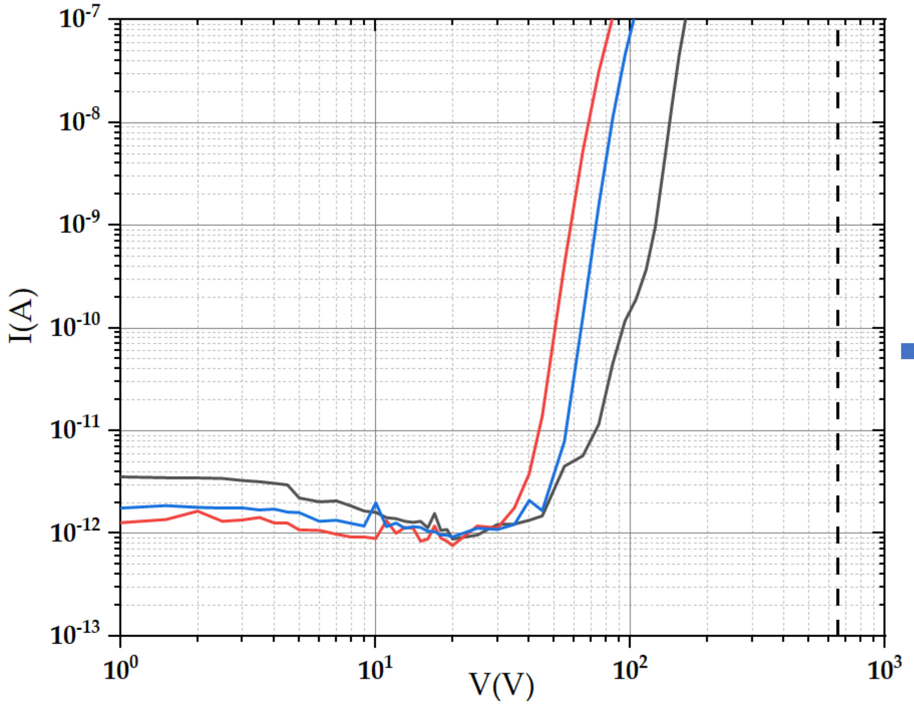


- ➢ Fabrication steps upgrades
- ➢ Characterization

Polishing: mirror surface for PLM phase

Dopant deposition: precursor influence

PLM recipes: thicker junction

Solvent cleaning: avoid surface defects

Plating-free lithography: buried junction

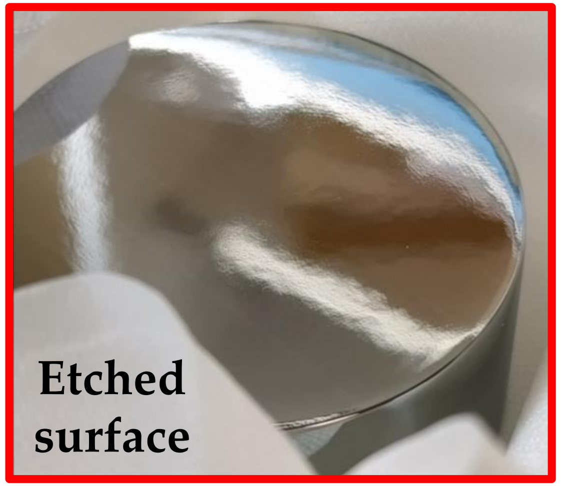
Process upgrade: crystal polishing

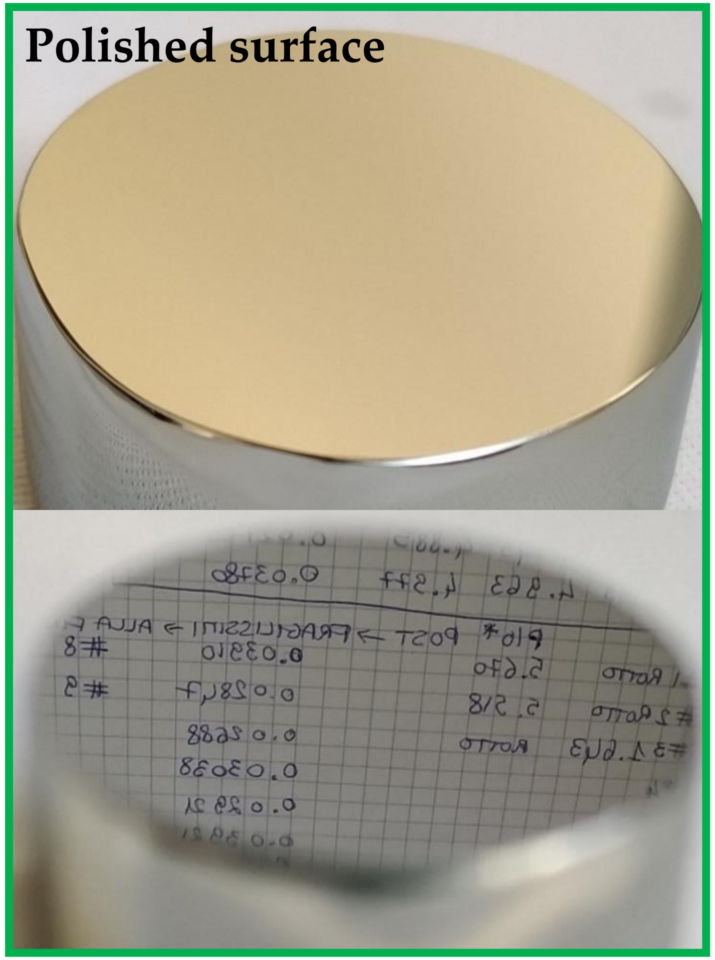
Grinding

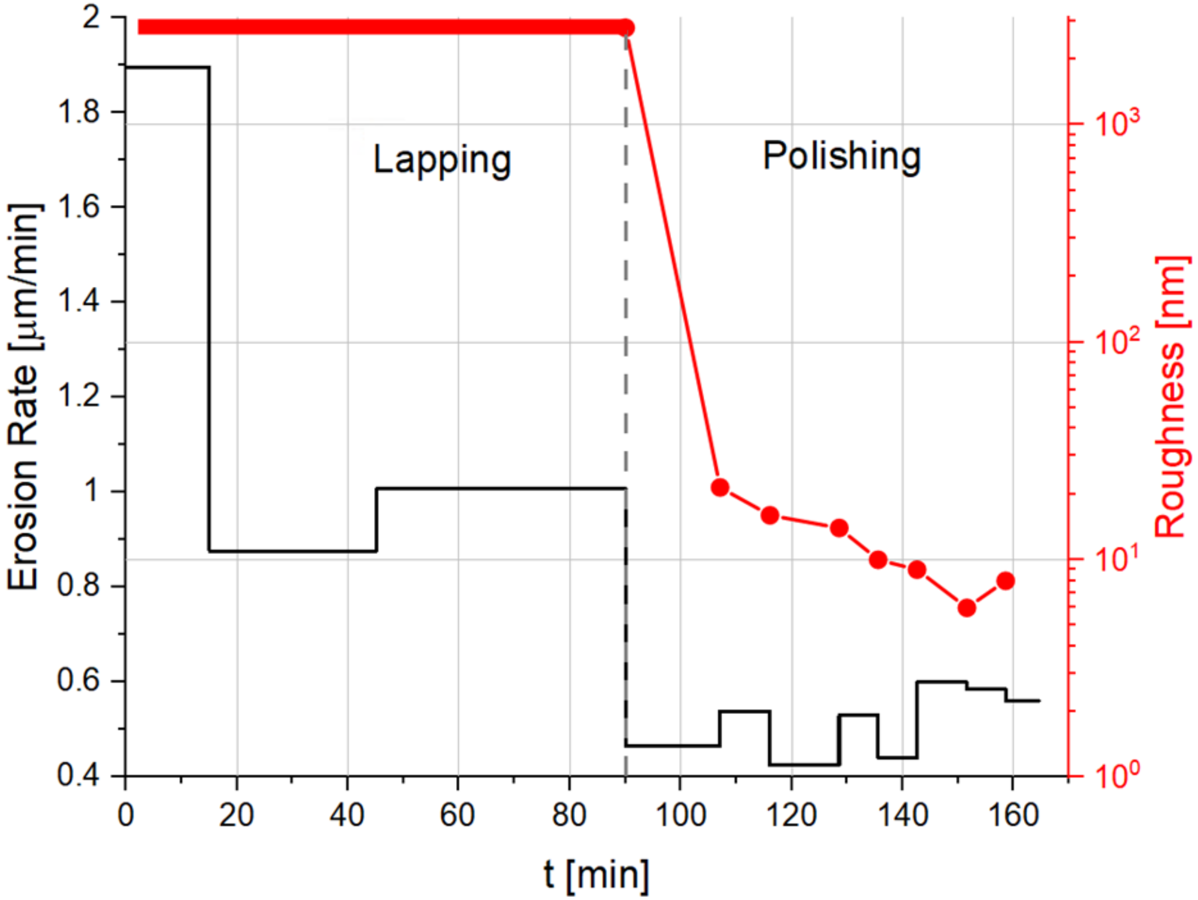
-Grinding with 3 μm Al_2O_3 slurry to remove sawing damages: the abraded thickness depends on last process.

Polishing

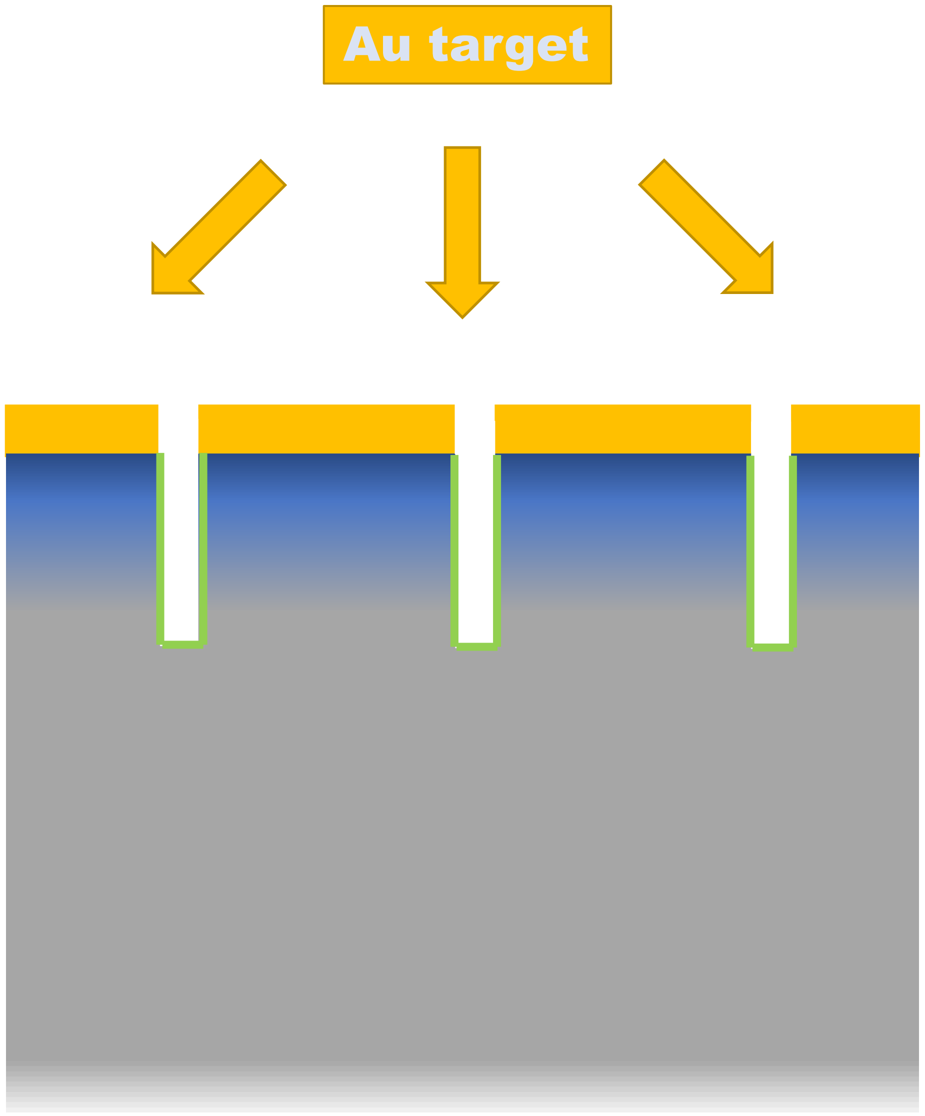
-Chemical-mechanical polishing with rotating disc rinsed with H_2O_2 1% (pH 12 with KOH)







Process upgrades

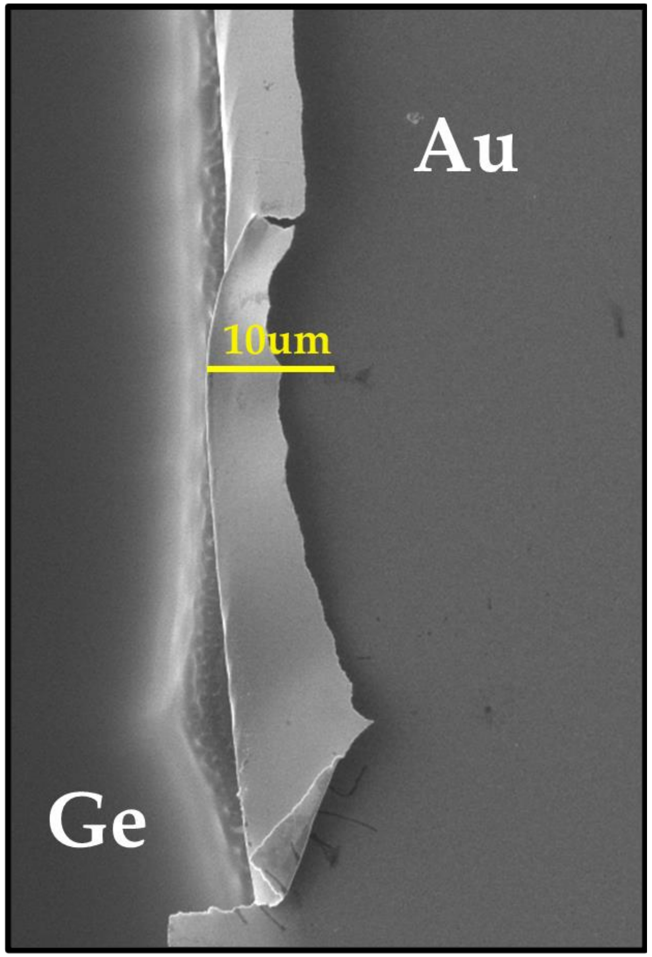


Full area ⇐ **PLM**

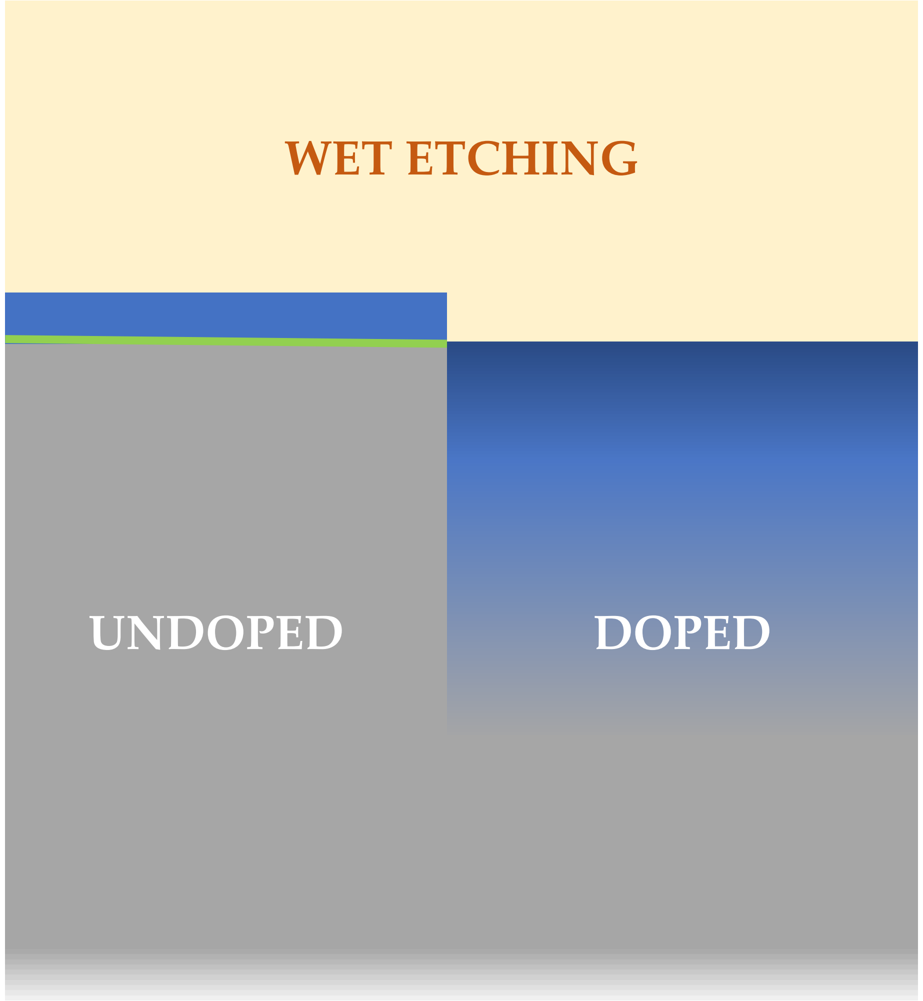
Au deposition
100 nm deposition of Au in Ar plasma with ultrapure target in vacuum (10^{-7} mbar)

Photolithography
Photoresist deposition, baking, exposure and development, followed by Au stripping and resist removal.

Intercontact gaps passivation
3:1 HNO3 : HF etching followed by chemical quenching passivation.



Process upgrades



Full area ⇦ PLM ⇨ Partial area

Au deposition
100 nm deposition of Au in Ar plasma with ultrapure target in vacuum (10^{-7} mbar)

Photolithography
Photoresist deposition, baking, exposure and development, followed by Au stripping and resist removal.

Intercontact gaps passivation
3:1 HNO3 : HF etching followed by chemical quenching passivation.

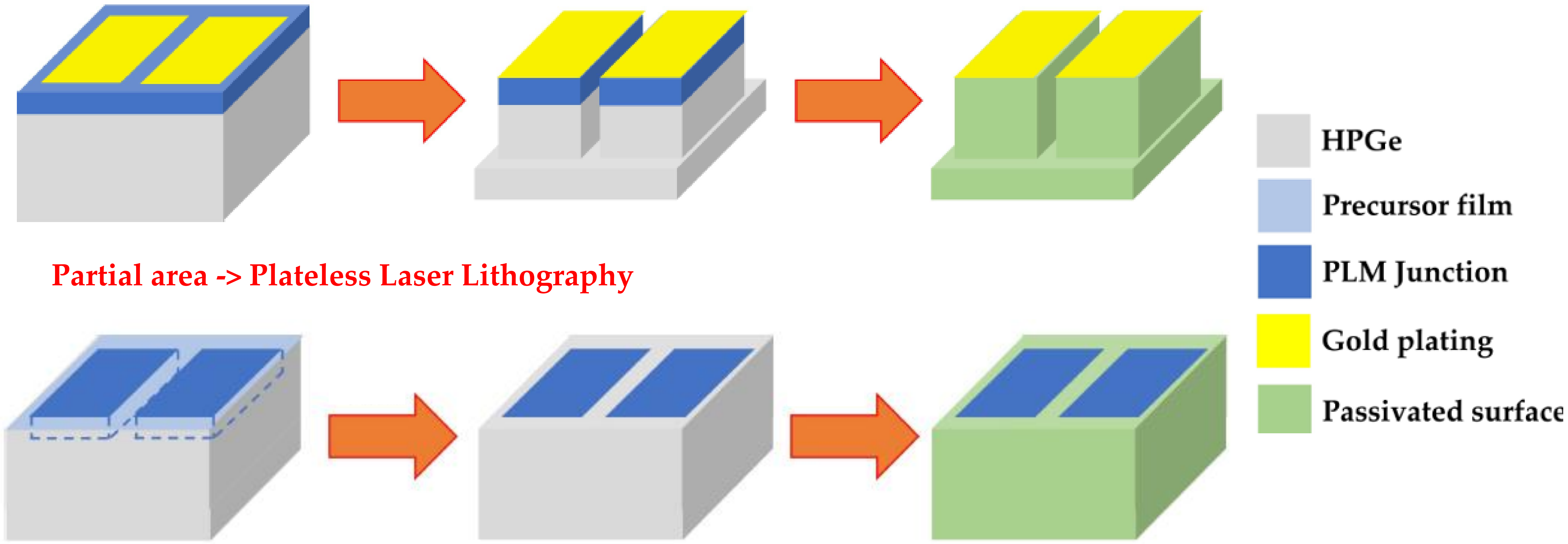
Selective etching
Removal of untreated dopant using selective etchants to protect the near junction.

Chemical passivation
Passivation of undoped surfaces with suitable solutions.

Process upgrades

Full area -> Plated Photolithography

Partial area -> Plateless Laser Lithography



Process upgrade: Pulsed Laser Melting

Full area

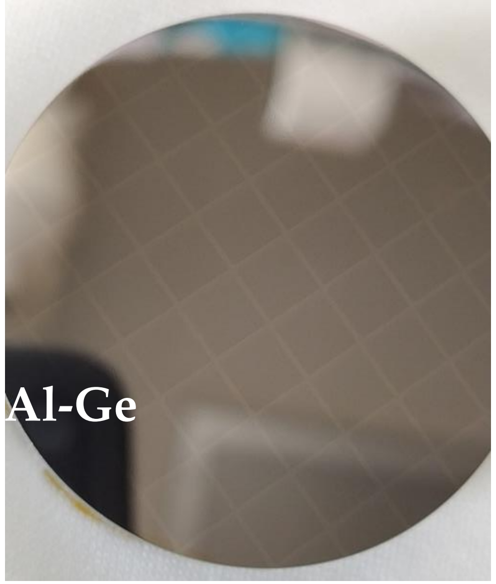
Same energy for every spot:

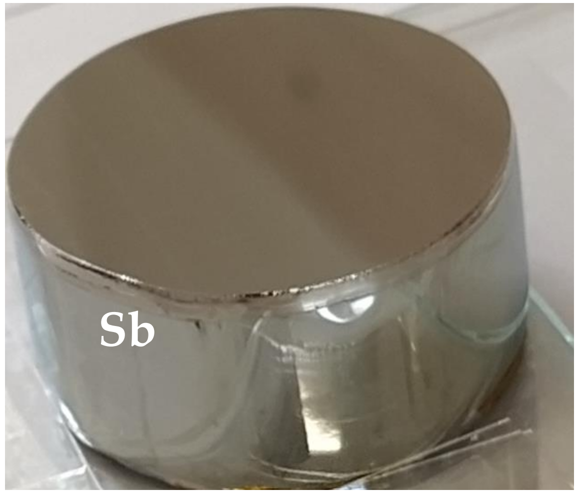
Al-Ge deposition:
100 mJ/cm² preannealing pulse + 500 mJ/cm² pulse

Patent WO 2021/214028 A1

Sb deposition:
no preannealing ,
500 mJ/cm² pulse

GeP deposition:
no preannealing ,
500 mJ/cm² 4 pulses

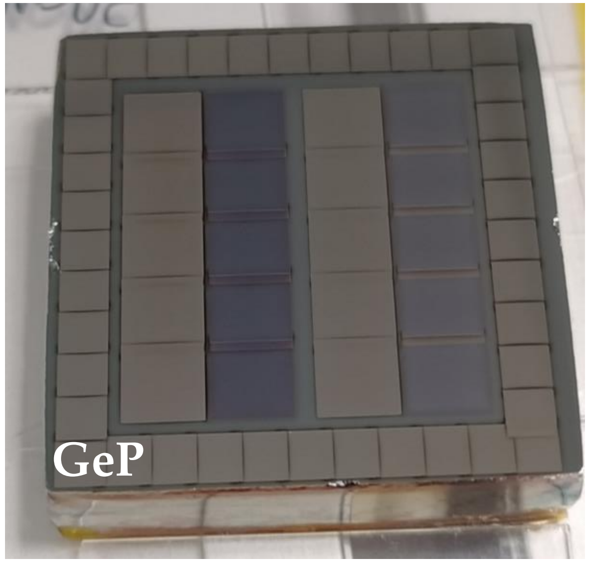


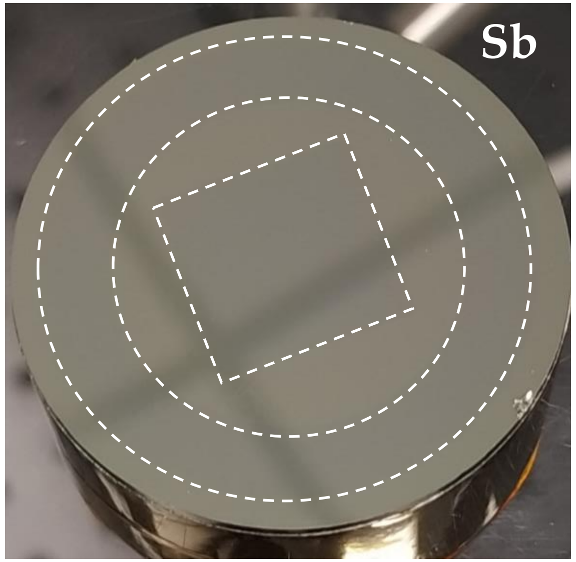


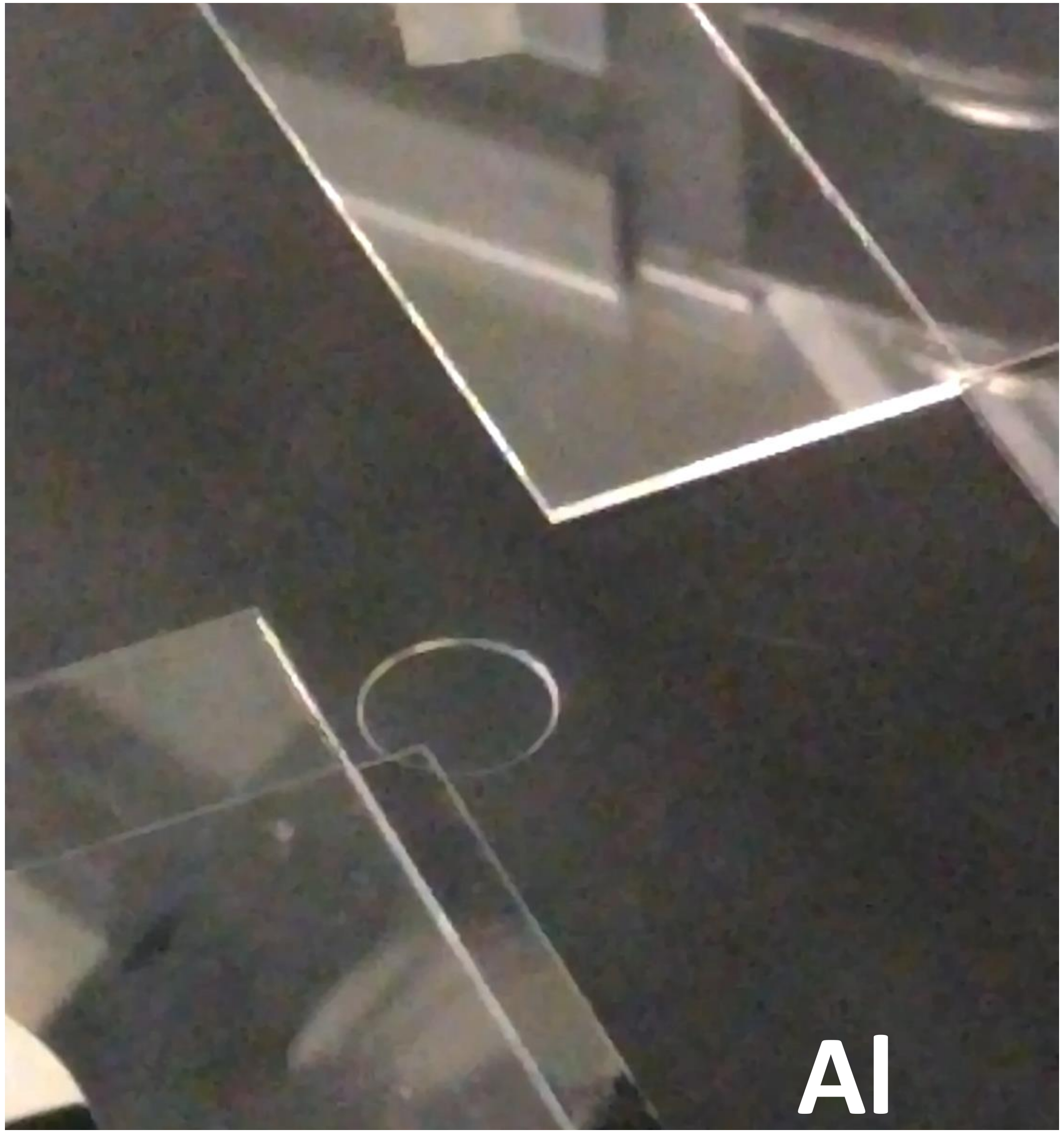
Partial area

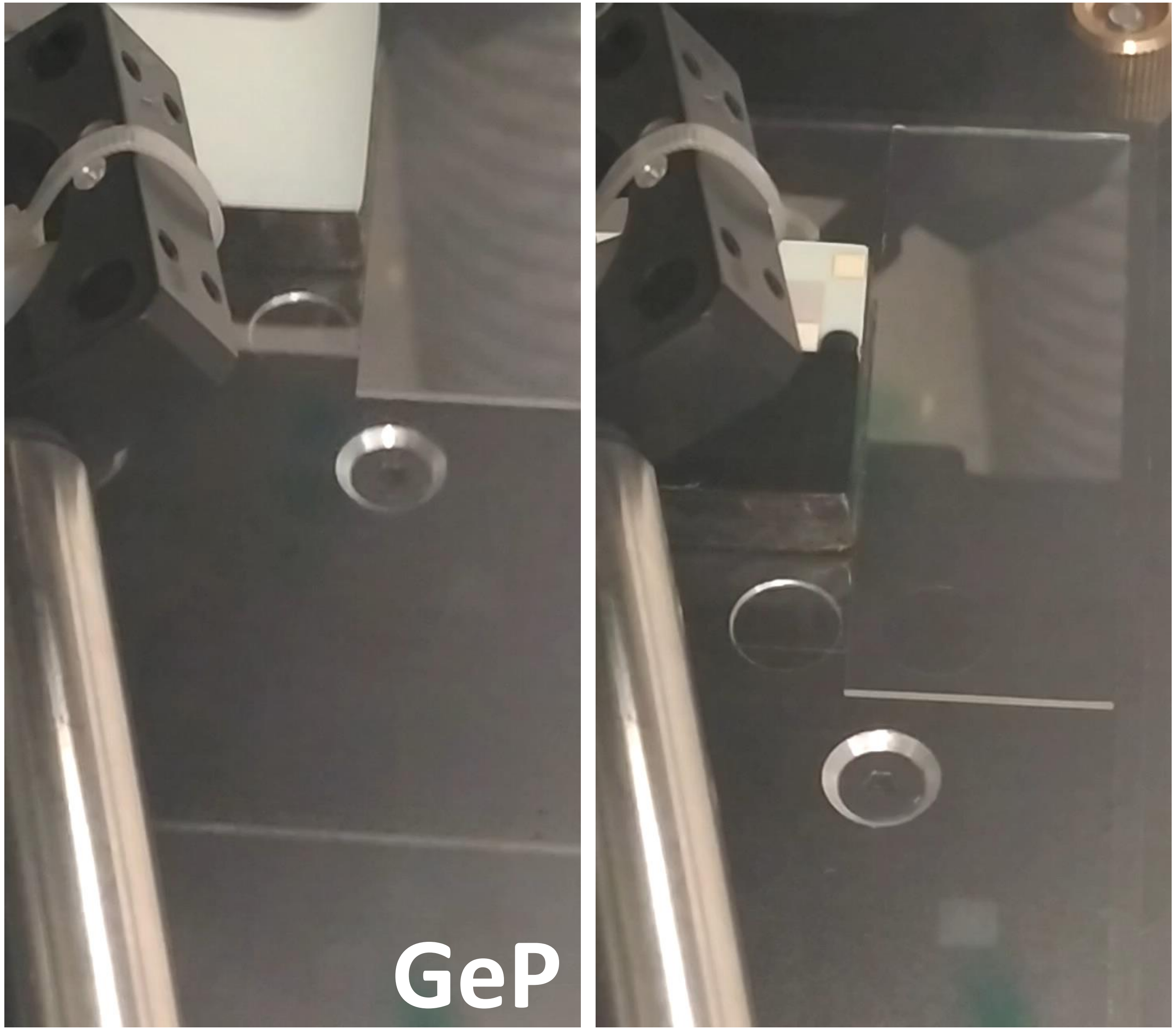
Different recipes:

All dopant types:
Different energy densities, different number of pulses, geometric flexibility, etc.






Al


GeP

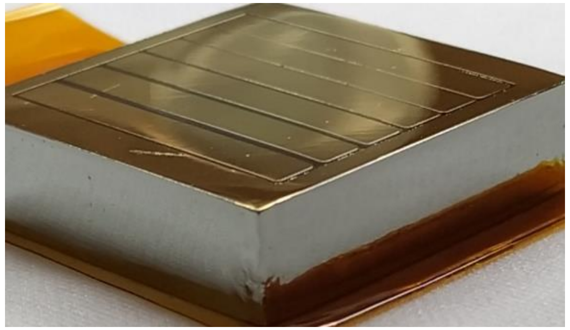
Process upgrade: lithography

Full area

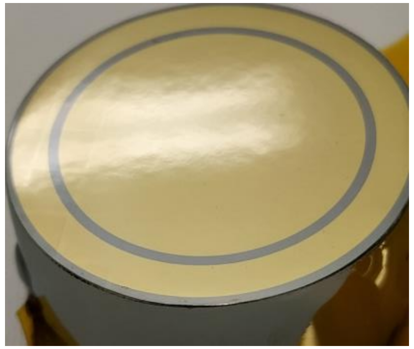
Gold sputtering deposition

Photolithographic process:

1) Positive resist spin coating
2) Softbake
3) Exposure through acetate mask
4) Development
5) Gold stripping
6) Resist stripping
7) Trenching: strong acid attack (HNO_3 , HF)



L=35mm, t=10mm



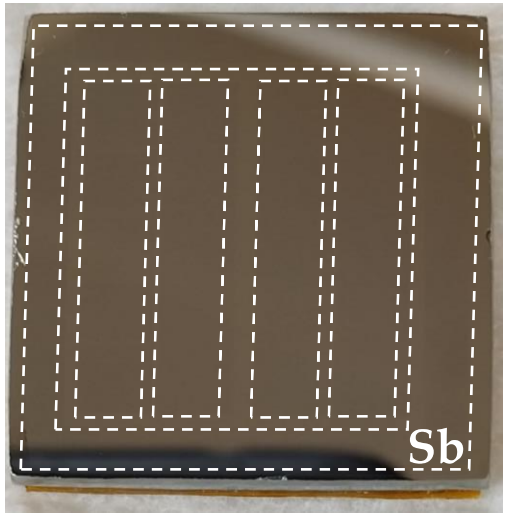
D=40mm, t=20mm

Partial area

Lithographic process using selective etching solutions:

- Hot pure H_2SO_4 for Sb deposition (preserve Sb junction)
- H_2O_2 for GeP deposition (slowly etches everywhere)
- Kern solutions (H_2O_2, H_3PO_4, Ethanol) for Al-Ge deposition (preserve Al junction)

L=35mm, t=10mm



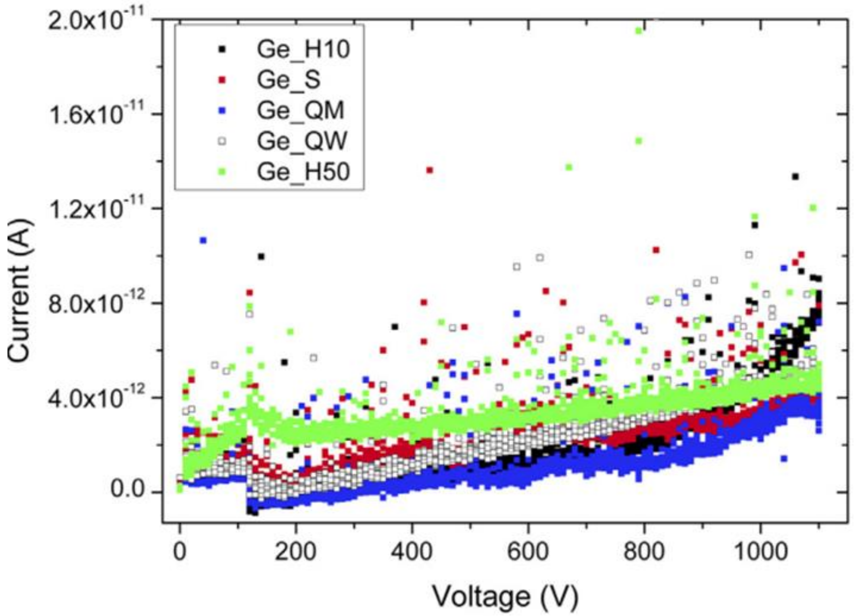
L=35mm, t=10mm

Process upgrade: passivation

Full area

Trenching.

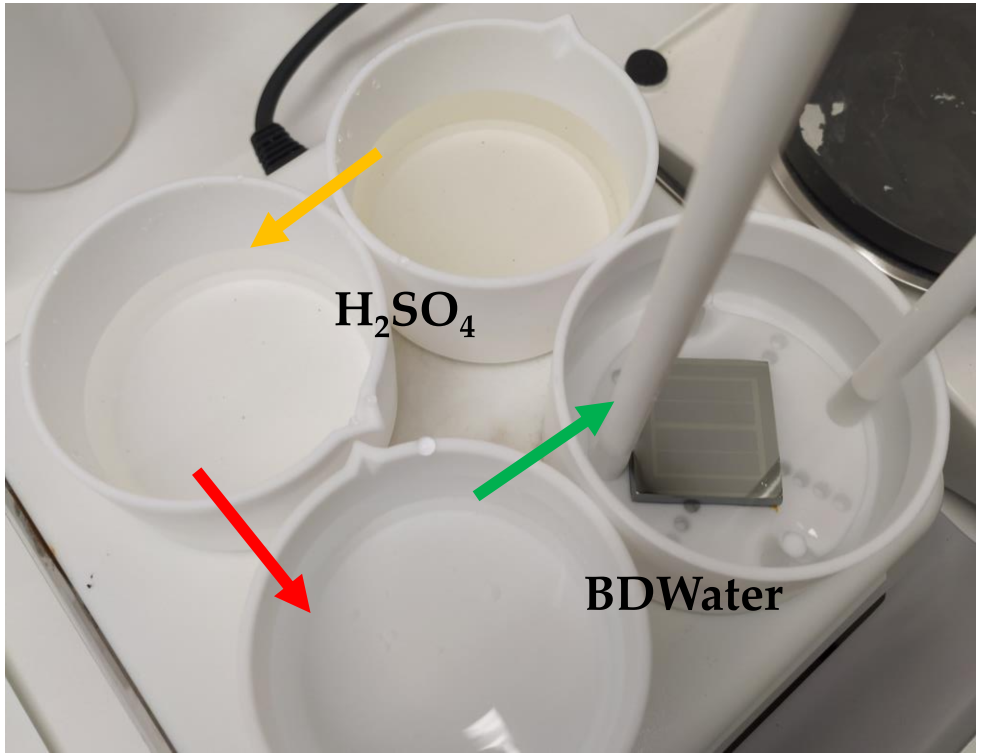
Quenching:
after the acid attack, rinse the sample in Methanol to saturate chemically active surfaces.
Methoxide-terminated Ge shows good surface insulation and stability



Partial area

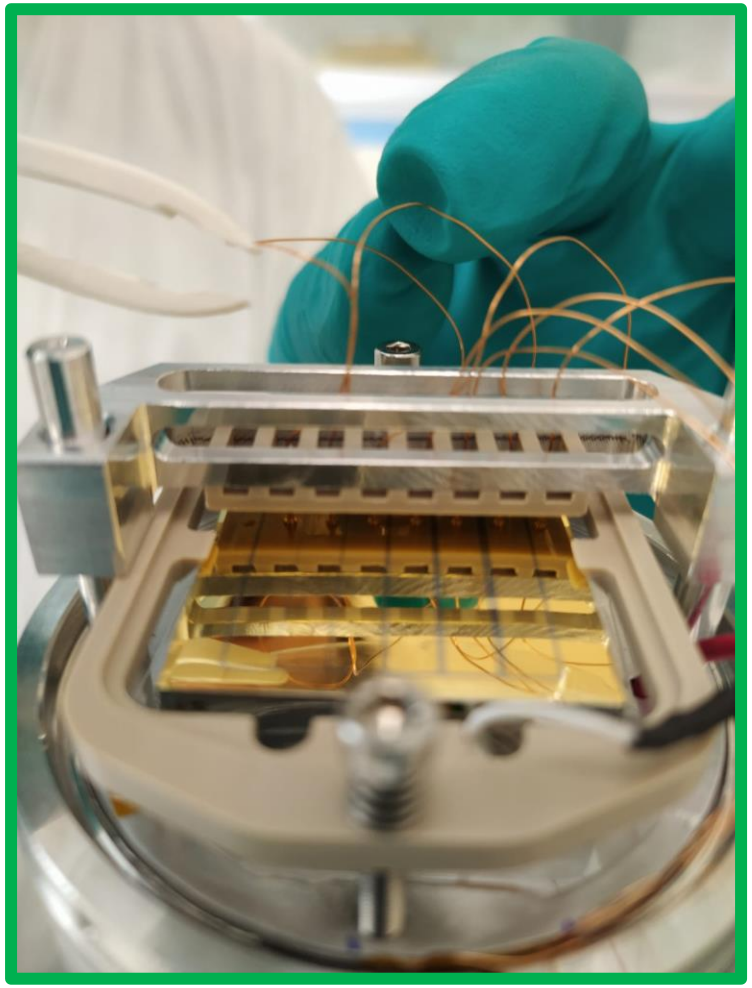
Quenching selective acids with suitable agents:

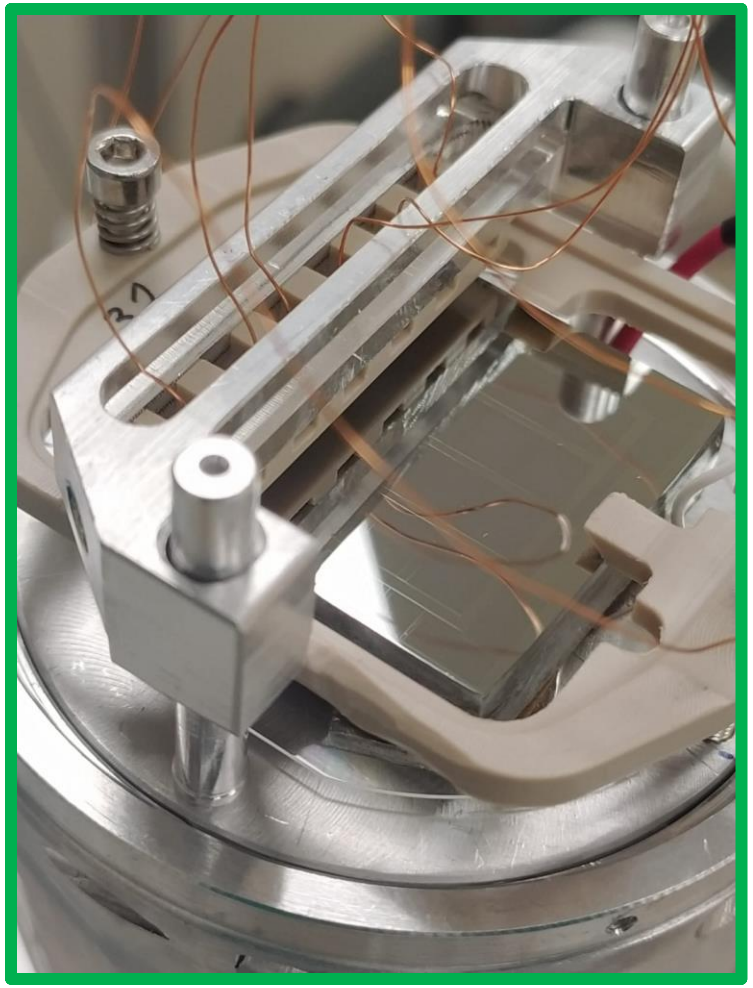
- H_2SO_4 (Sb) → Water (Safety)
- H_2O_2 (GeP) → Water (Already diluted)
- Kern (Al-Ge) → Methanol

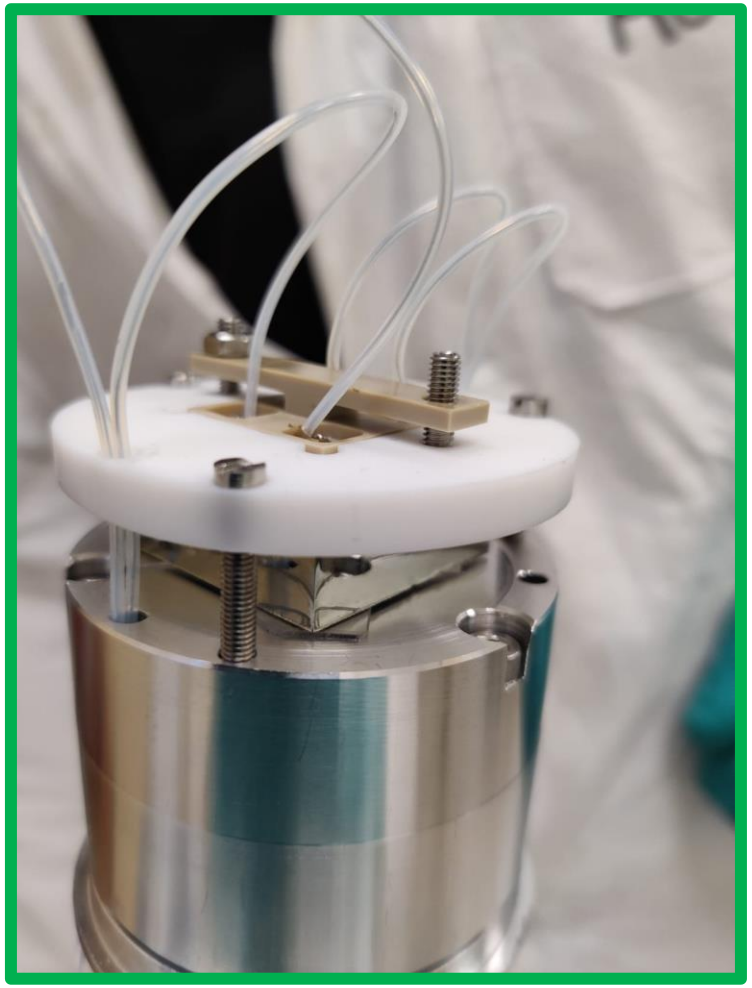


Process upgrade: passivation

Cooling:
samples hosted in prototyped cryostats and cooled down to LN2 temperature to reduce thermal carriers: delicate electrical contacts with springs on plated and non-plated segments.







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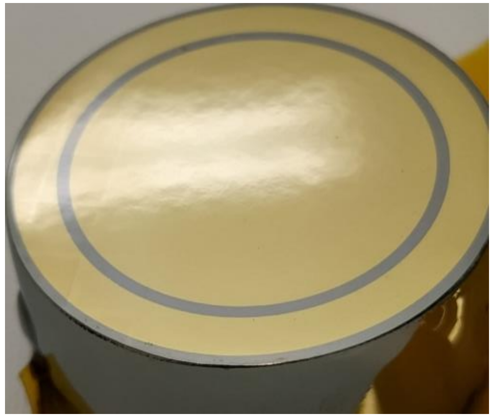
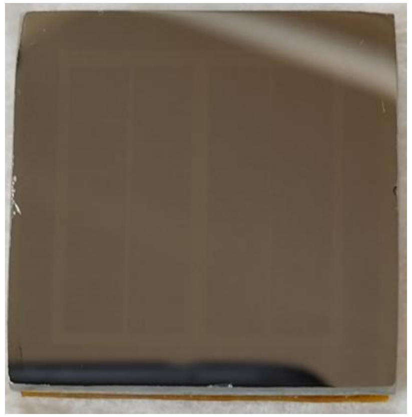
Thick planar detectors

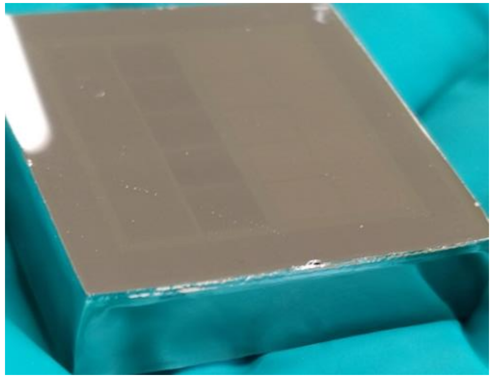
Al/n-HPGE/Sb,
D=40mm, t=20mm



Sb/p-HPGE/Al,
L=35mm, t=10mm

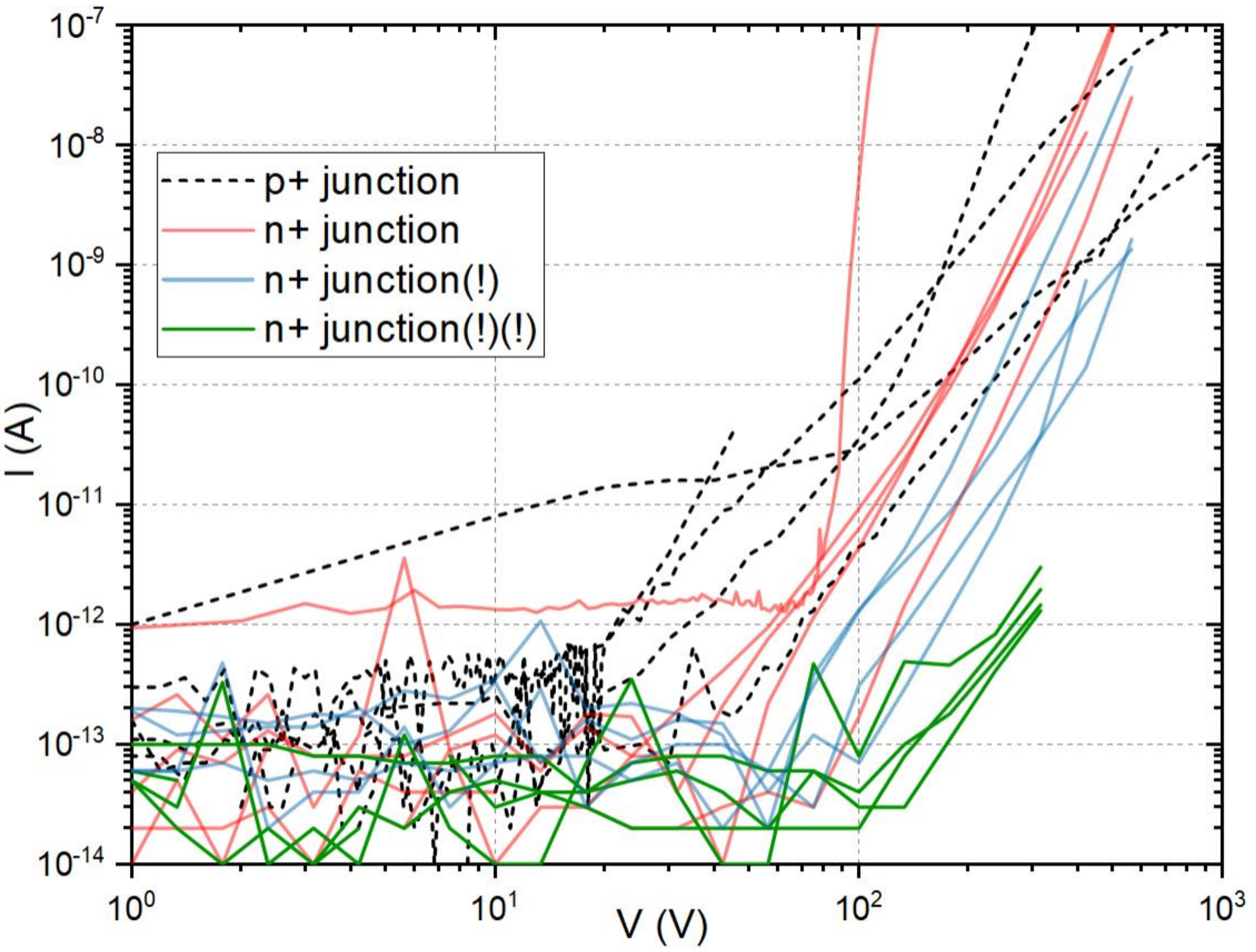


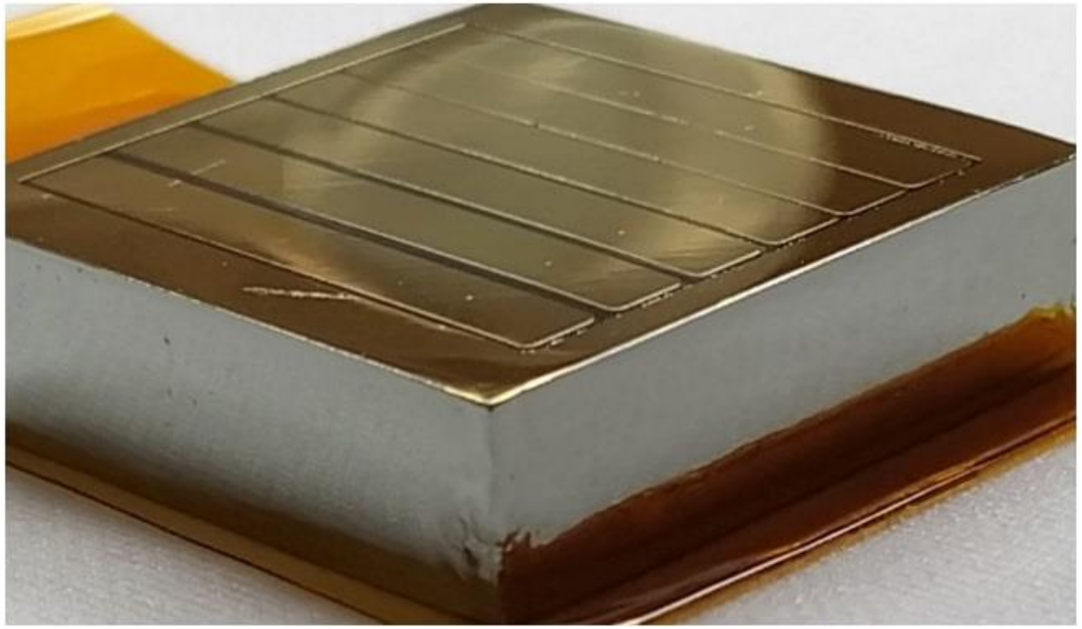
P/p-HPGE/Al,
L=35mm, t=10mm



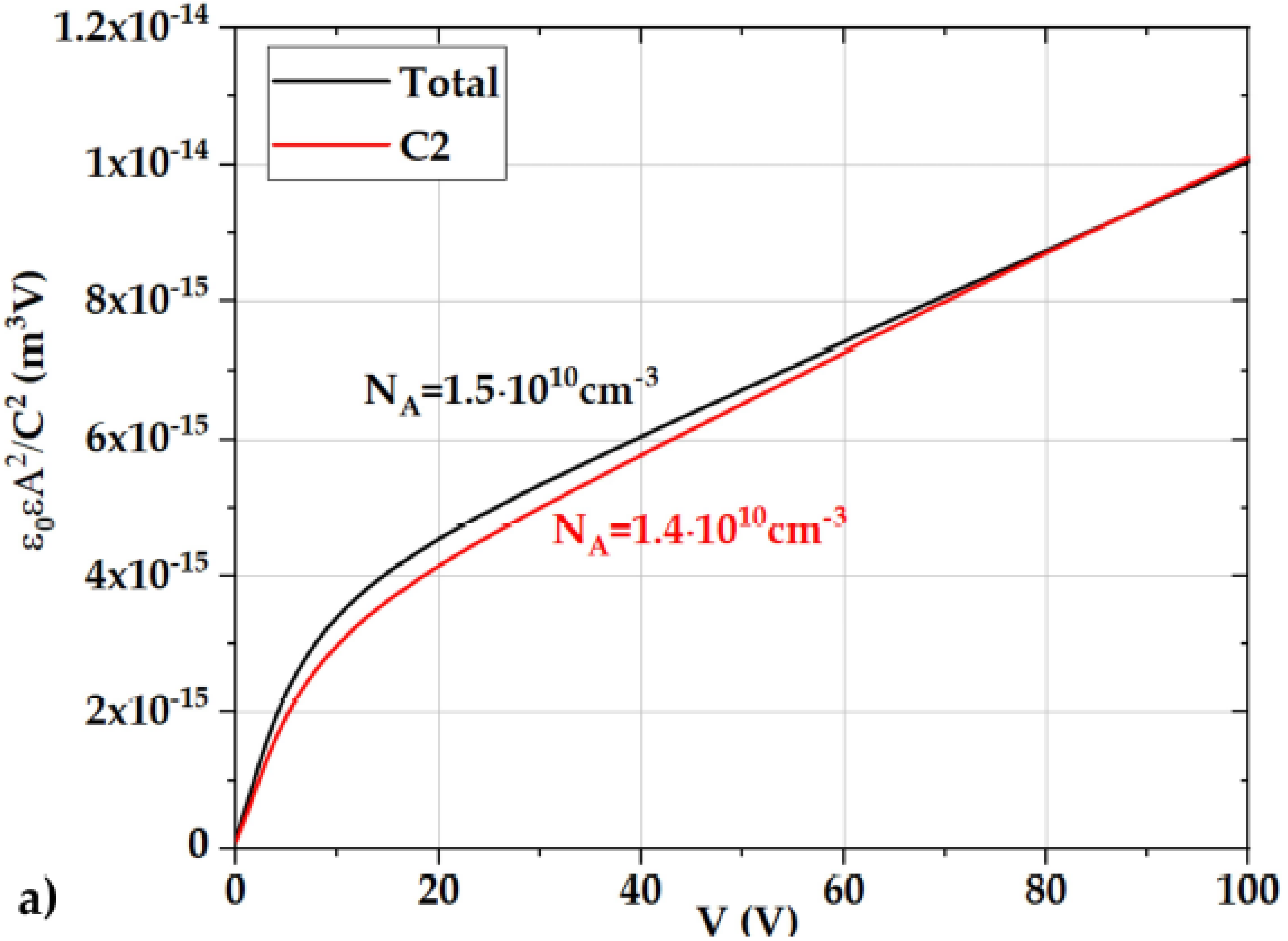
Difficult to reach depletion voltages (600V for 1cm, 2400V for 2 cm)

Probably due to junction mechanical weakness under manipulation or friction wearing.

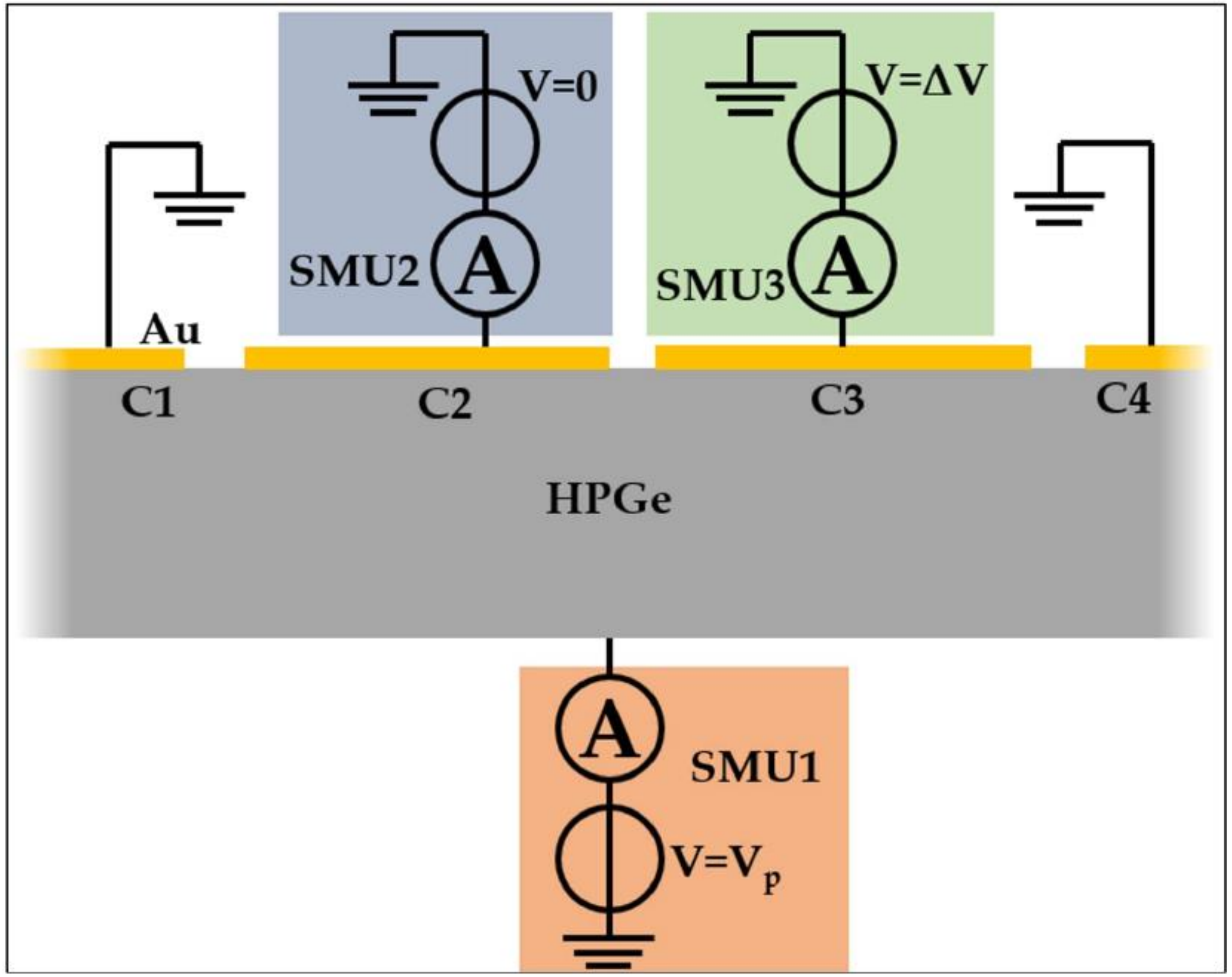


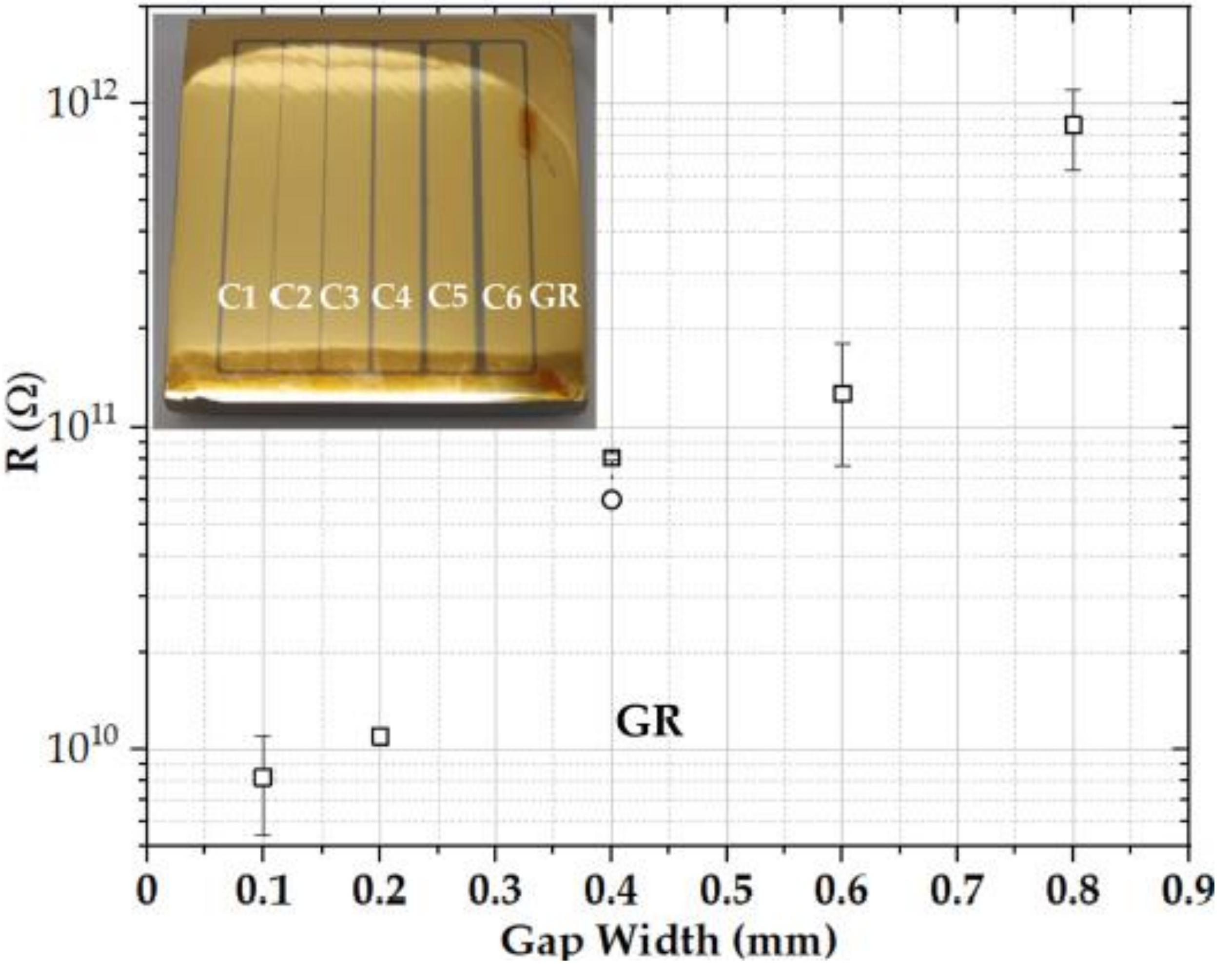


$$C \propto \frac{1}{\sqrt{V}}$$



Thick planar detectors

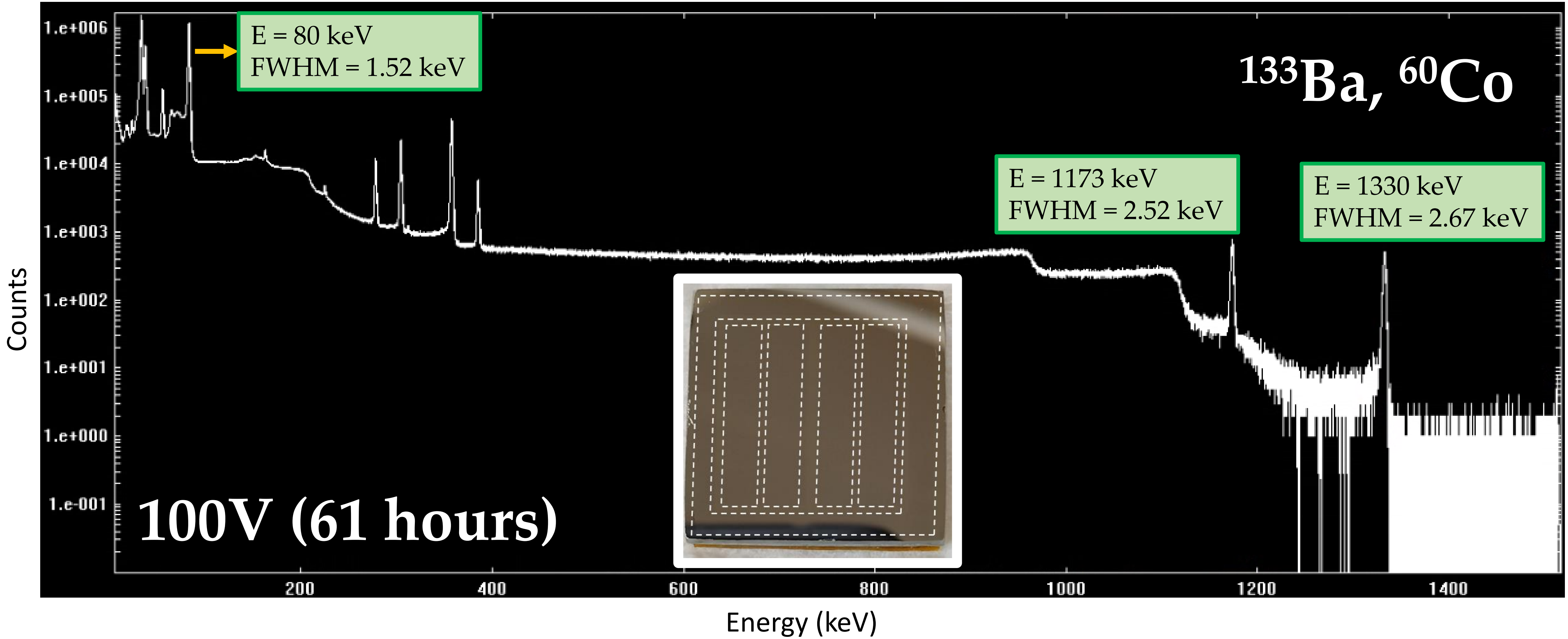




Poole-Frenkel hopping

Thick planar detectors

Partially depleted guarded segment of the Sb/p-HPGe/Al detector:



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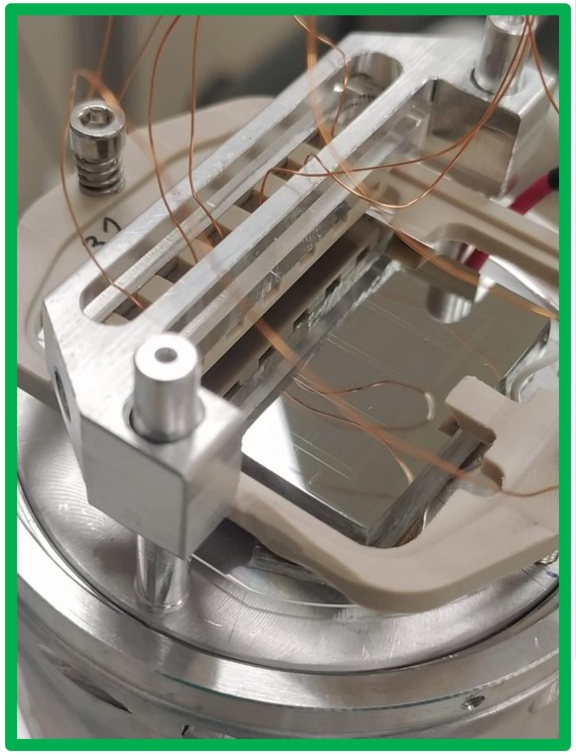
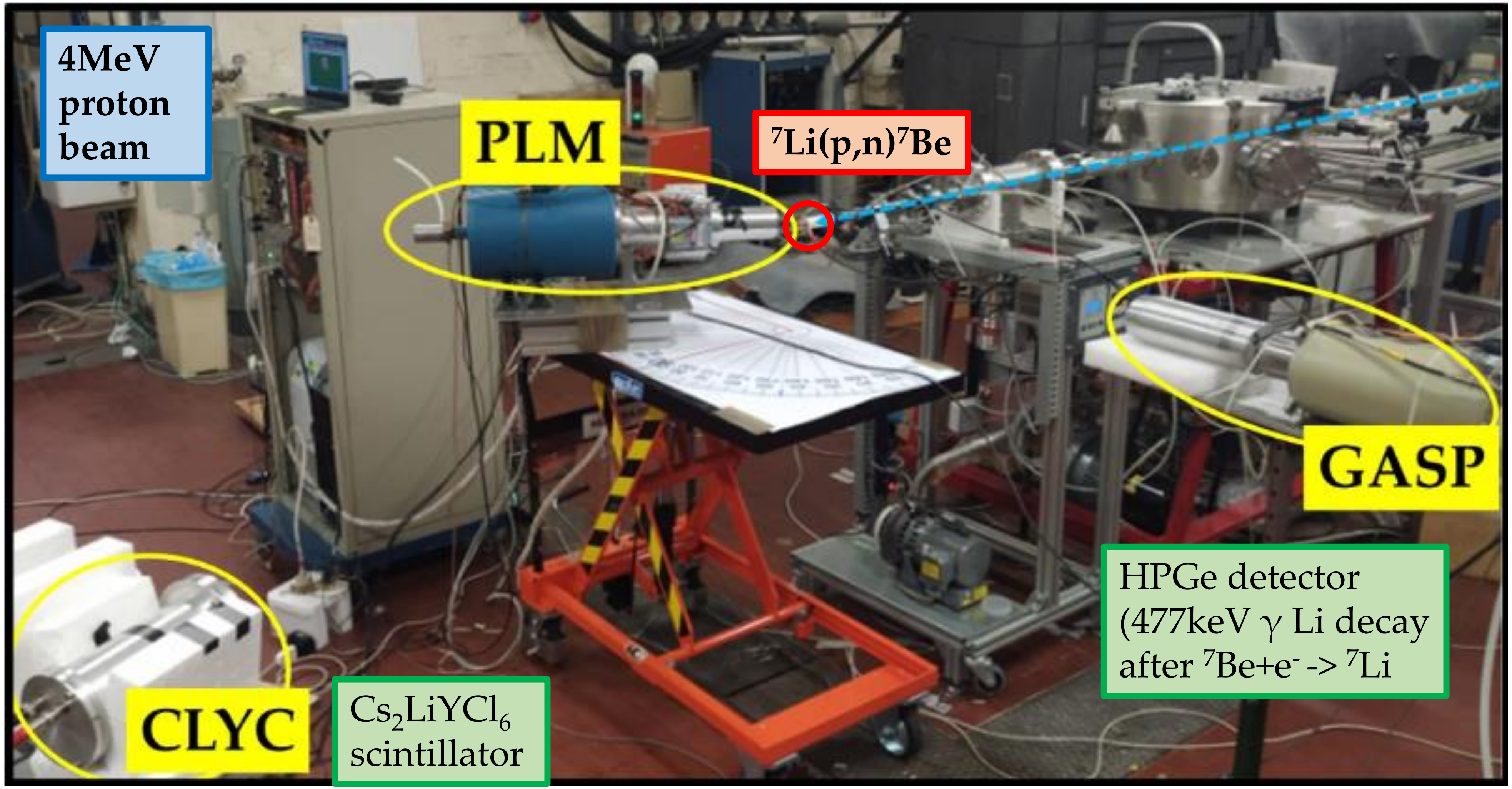
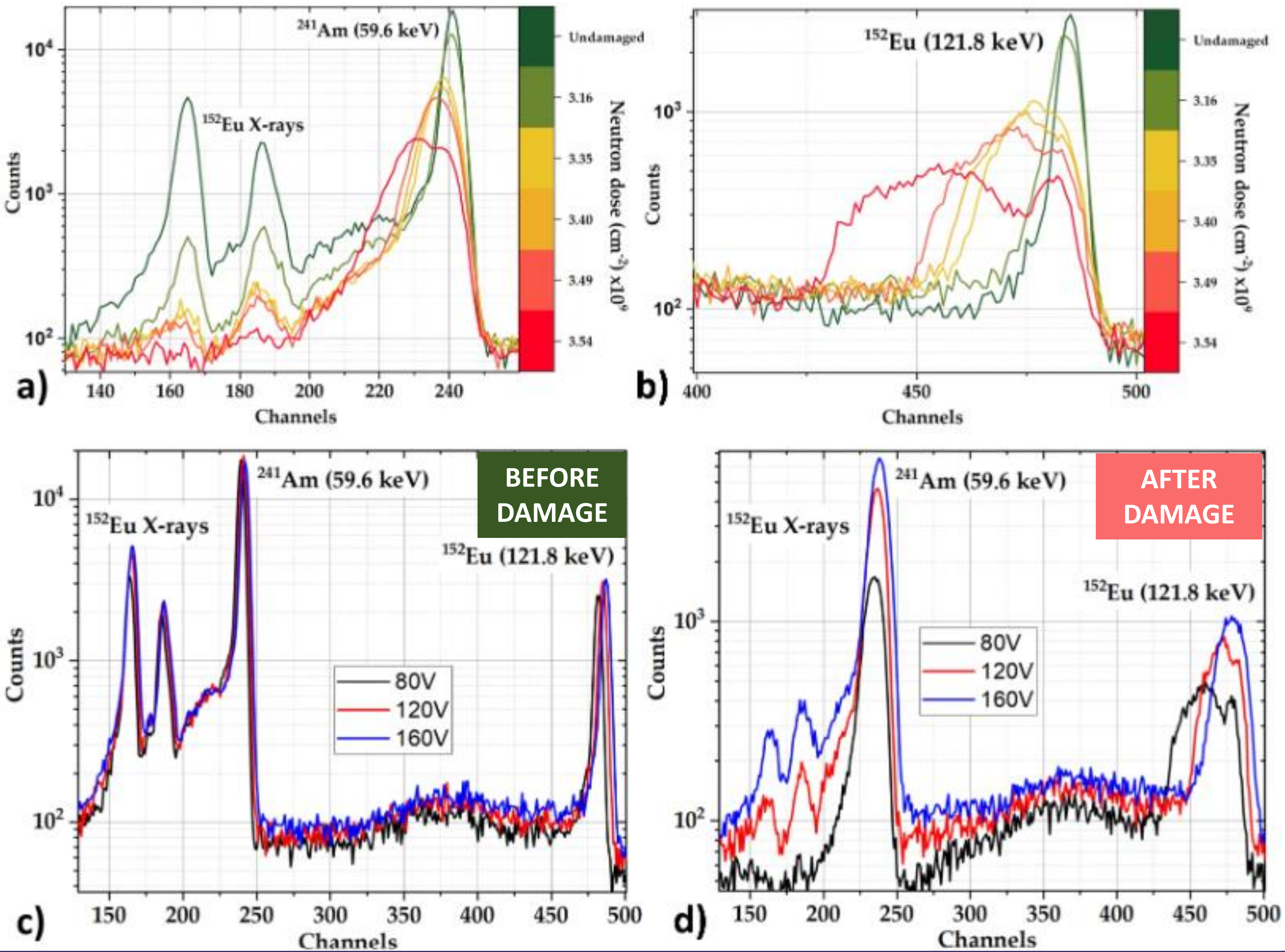
Test of recovery after n damage

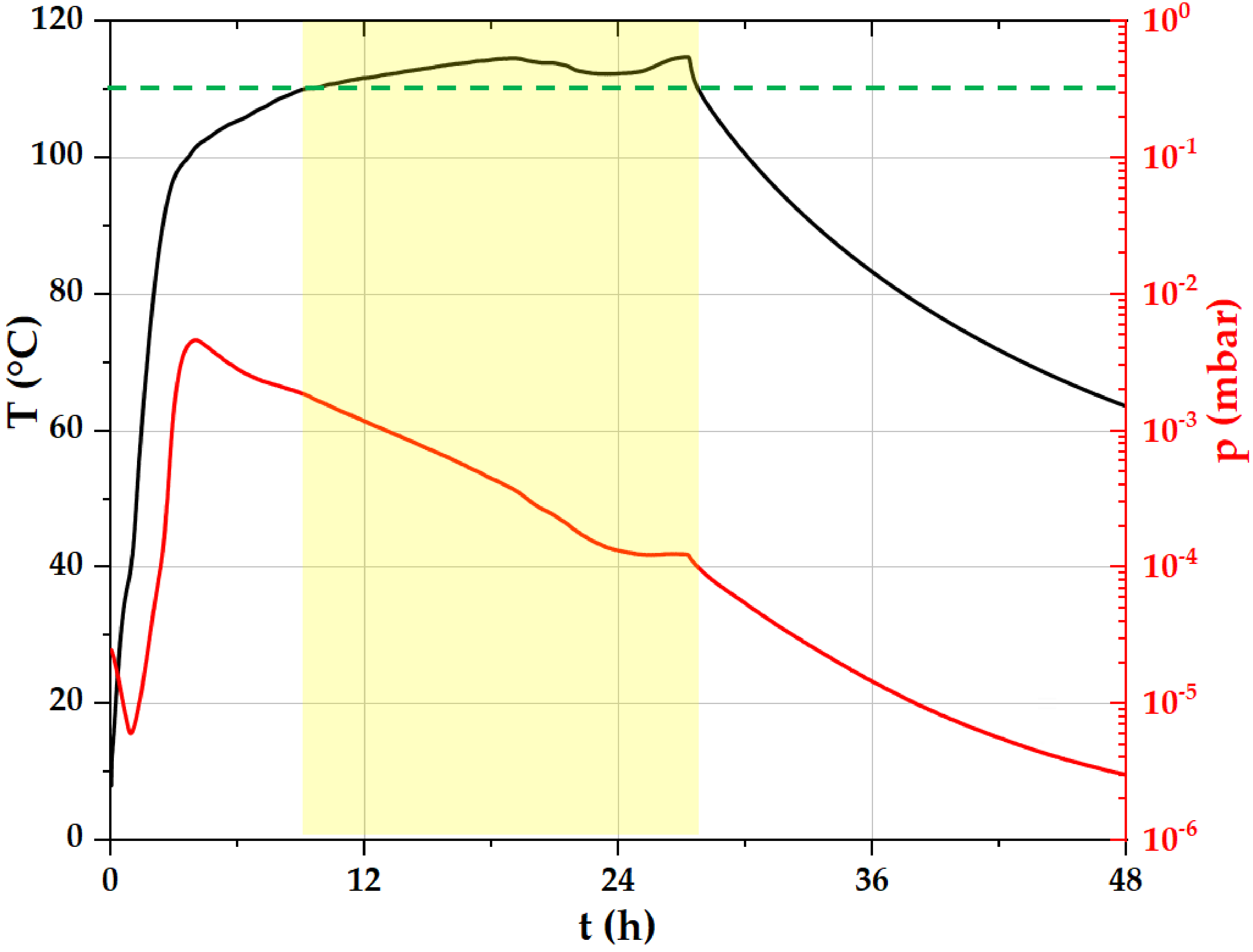


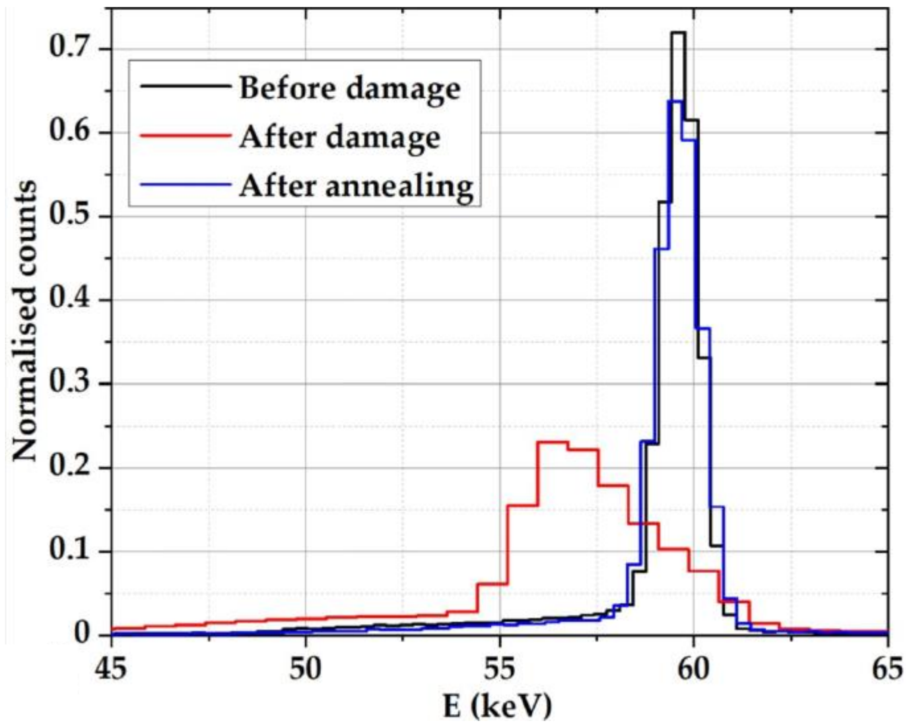


Test of recovery after n damage

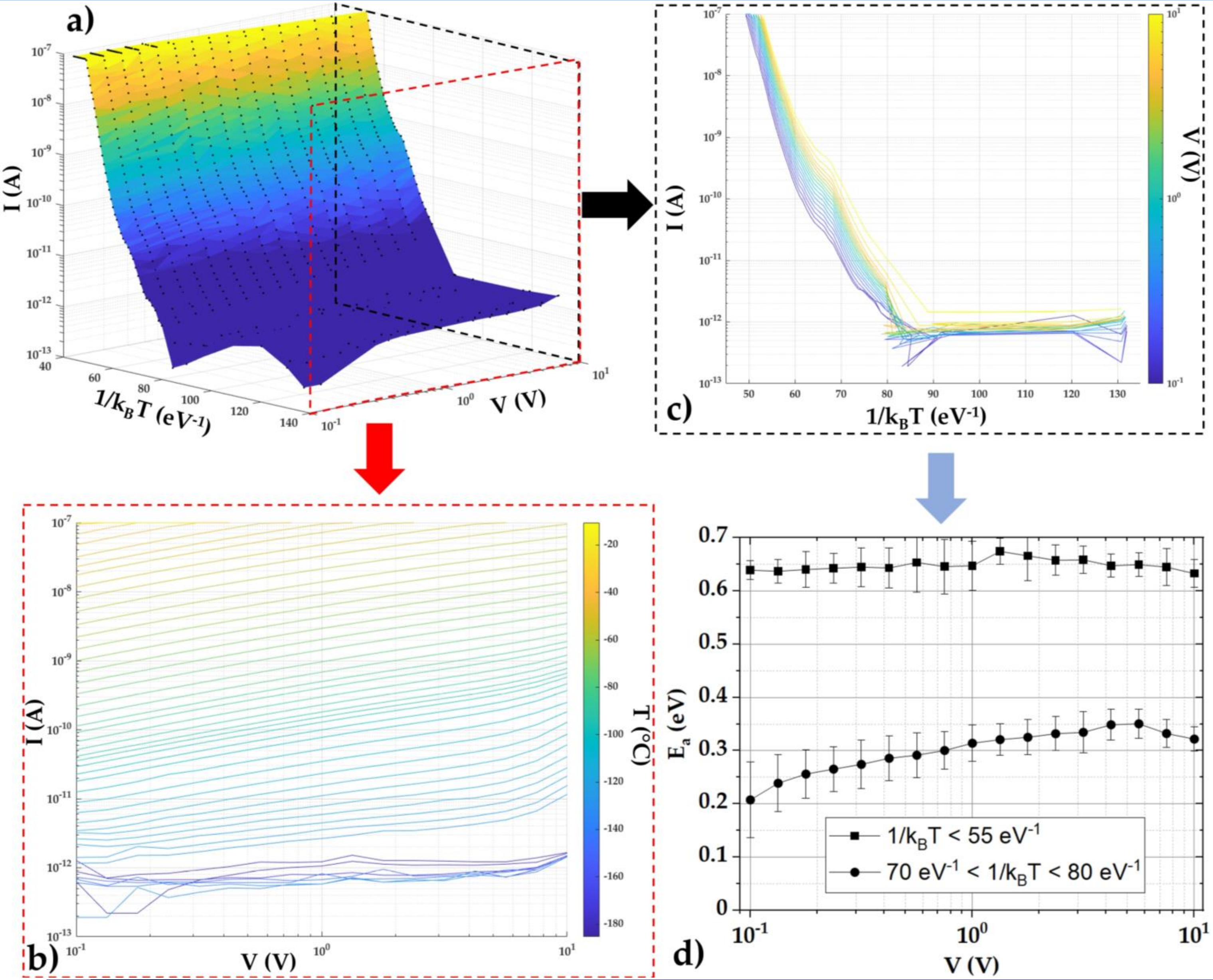


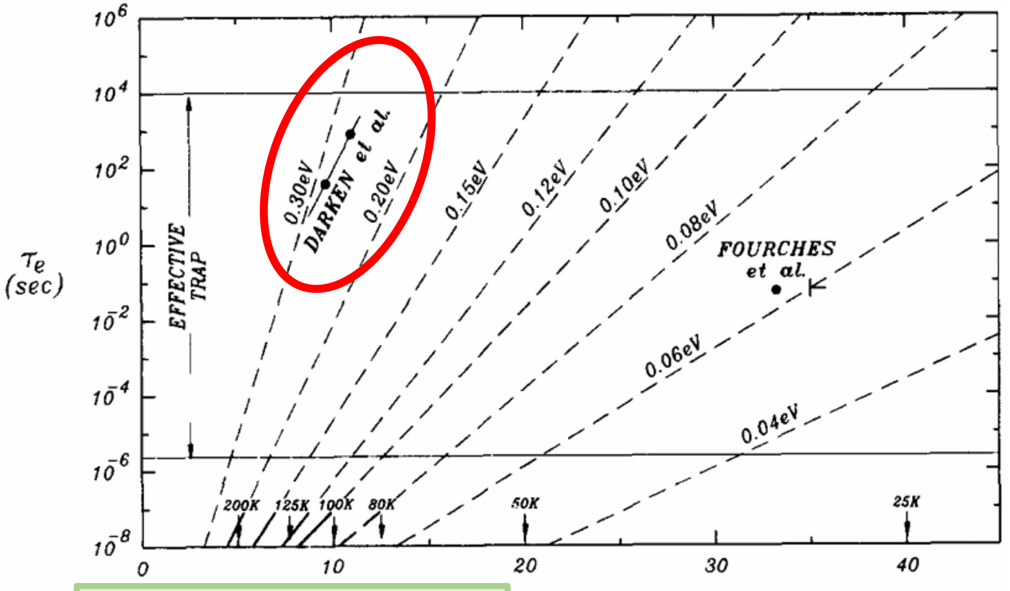
Test of recovery after n damage





Test of recovery after n damage





L.S. Darken (1993)

TABLE I

CONSTANTS OF THE RECOMBINATION PROCESS IN IRRADIATED GERMANIUM AND SILICON

	K (nvt-μsec)	$E_C - E_t$	σ_p(cm^2)	σ_n(cm^2)	$\partial N_t/\partial\phi$
nGe	$5.0\pm2.0\times10^{13}$	0.23 ev	1.0×10^{-15}	$\sim4\times10^{-15}$	3
pGe	$2.4\pm0.4\times10^{13}$	0.23 ev	1.0×10^{-15}	$\sim4\times10^{-15}$	3
nSi	$2.8\pm0.8\times10^{12}$	—	—	—	—
pSi	$3.2\pm1.1\times10^{12}$	—	—	—	—

G. Messenger and J. Spratt (1958)

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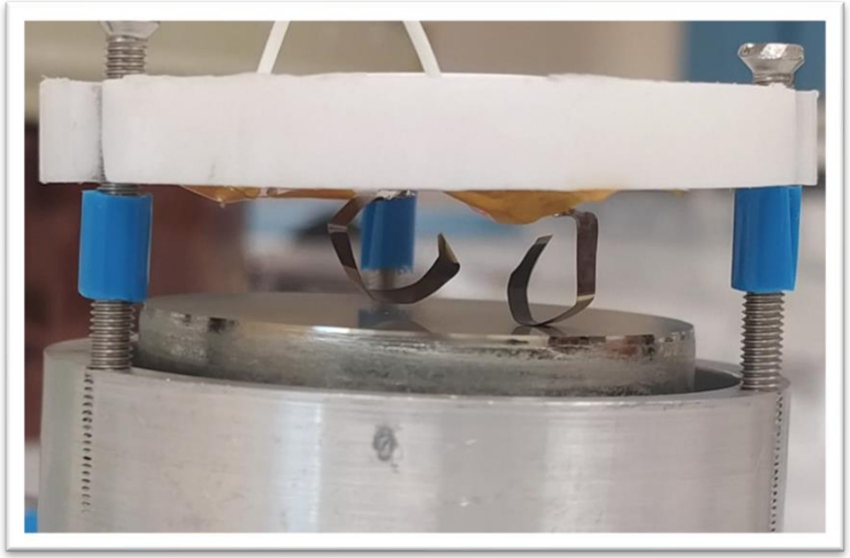
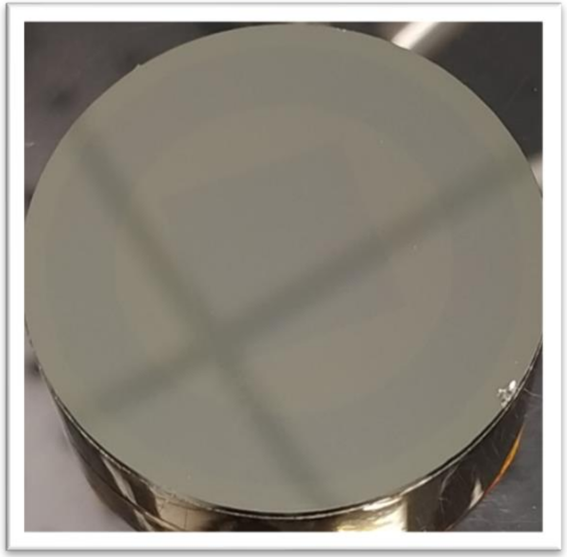
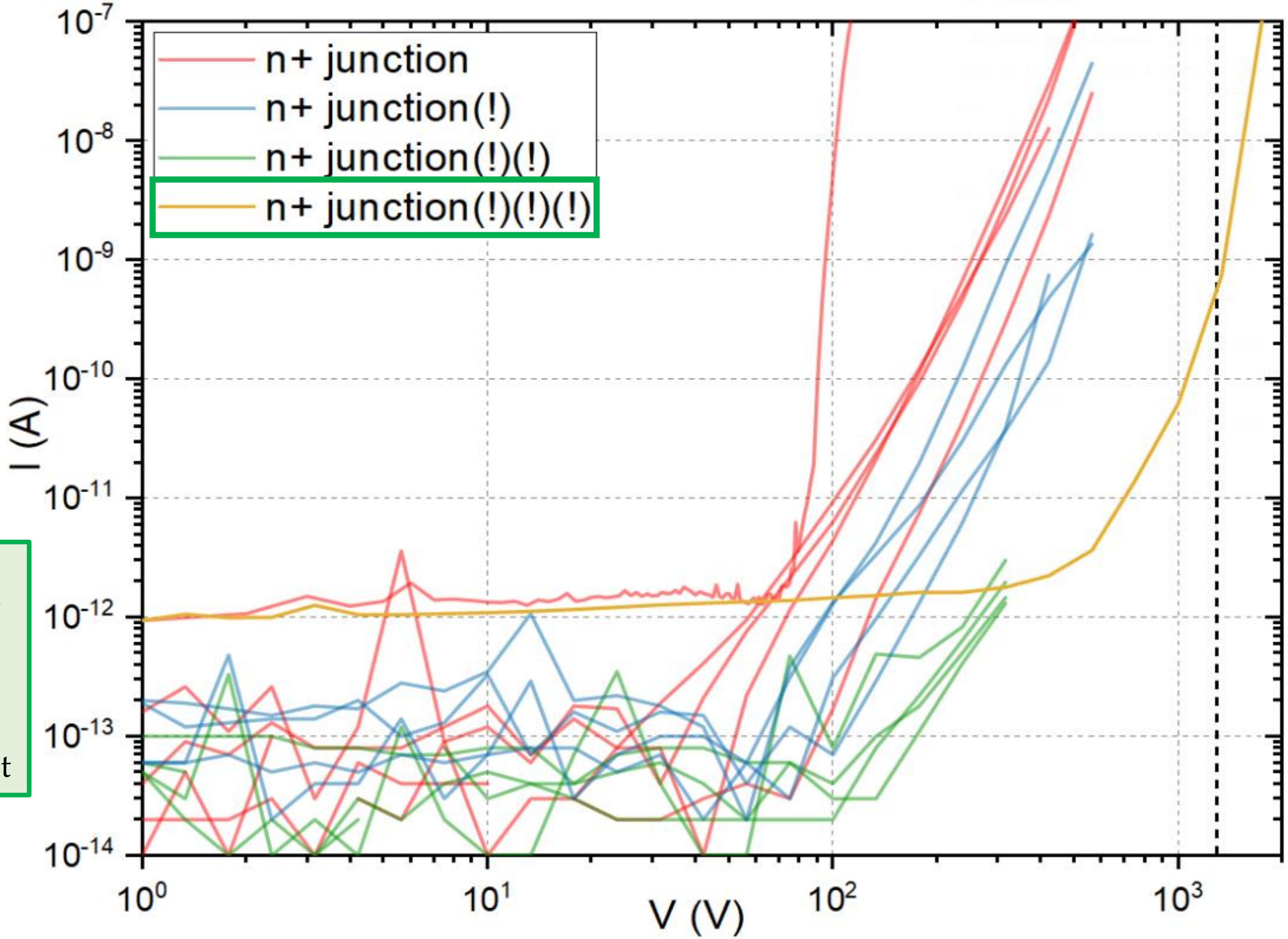
Plating upgrade





Sb/p-type/Al
N=6x10^9 cm^{-3}
d=40mm
t=19mm
Guard ring+
central contact



Plating upgrade

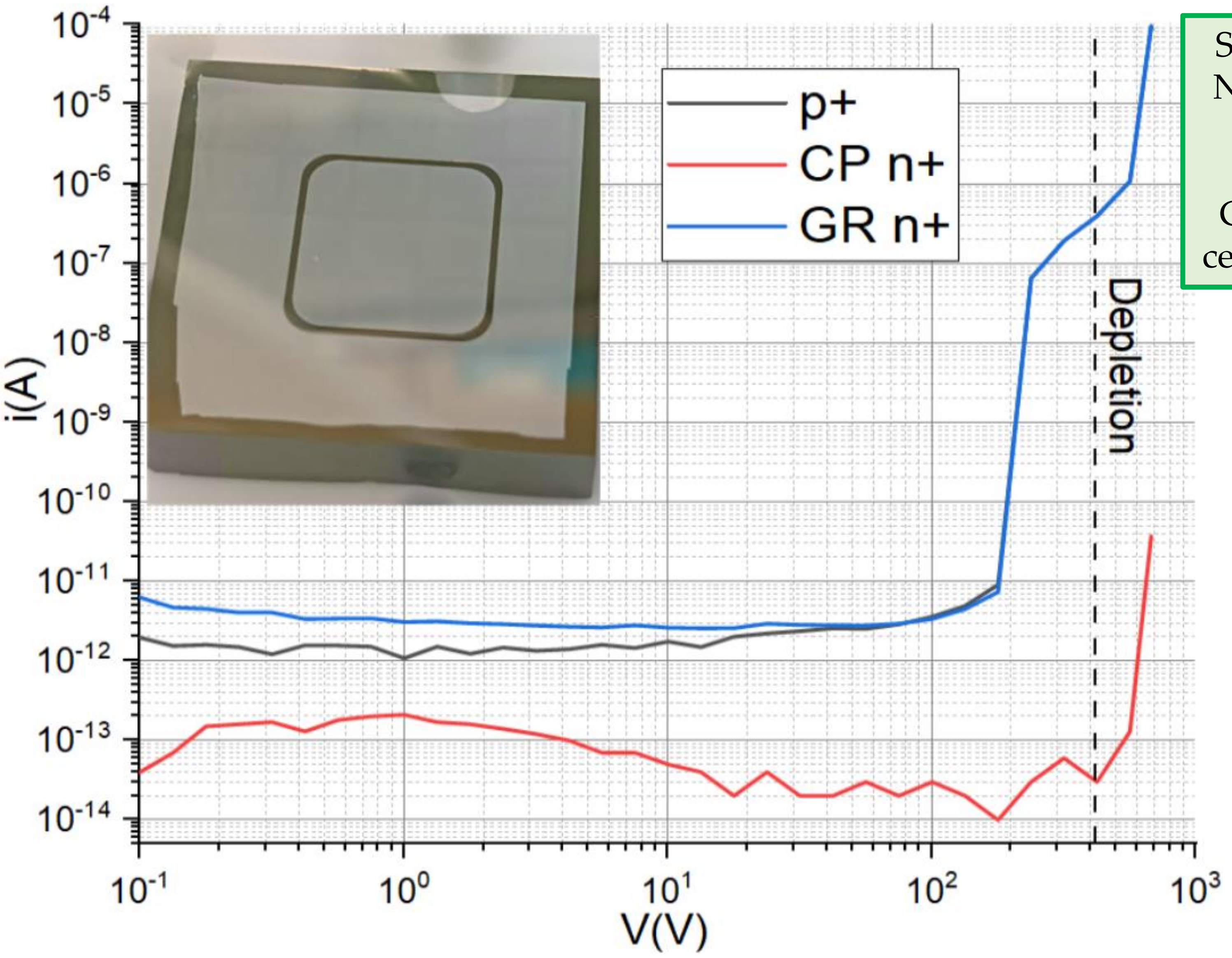
After thesis deposit

Thick Al sputtering deposition

Photolithographic process:

1) Positive resist spin coating
2) Softbake
3) Exposure through acetate mask
4) Development
5) Post-Exposure Bake
6) Al stripping and Ge trenching
7) Resist stripping

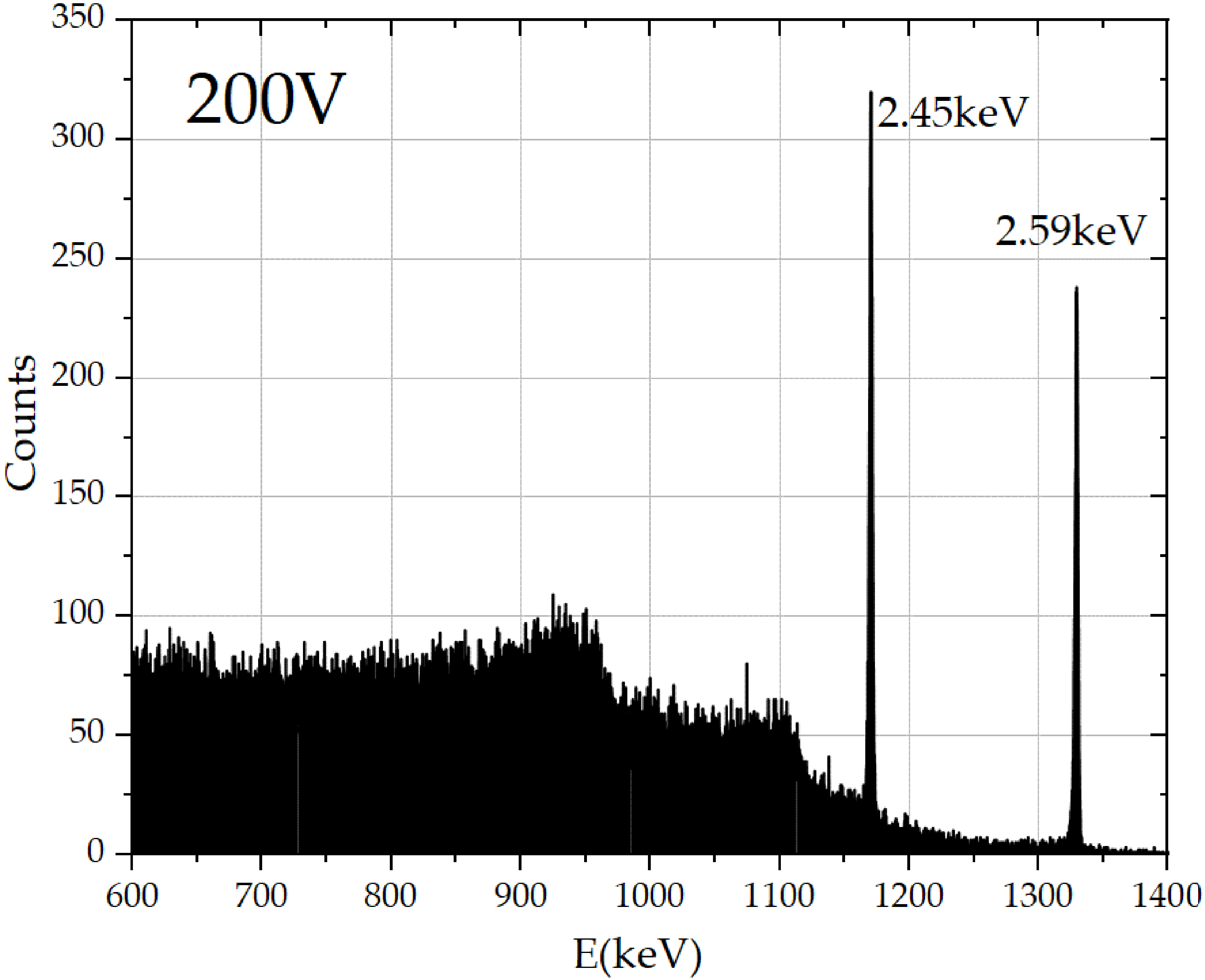
Passivation:
Diluted Kern solution (H_2O_2, H_3PO_4, Ethanol) fast bath
Quenching:
Methanol baths



Sb/p-type/Al
$N=6x10^9$ cm^{-3}
l=28mm
t=10mm
Guard ring+ central contact

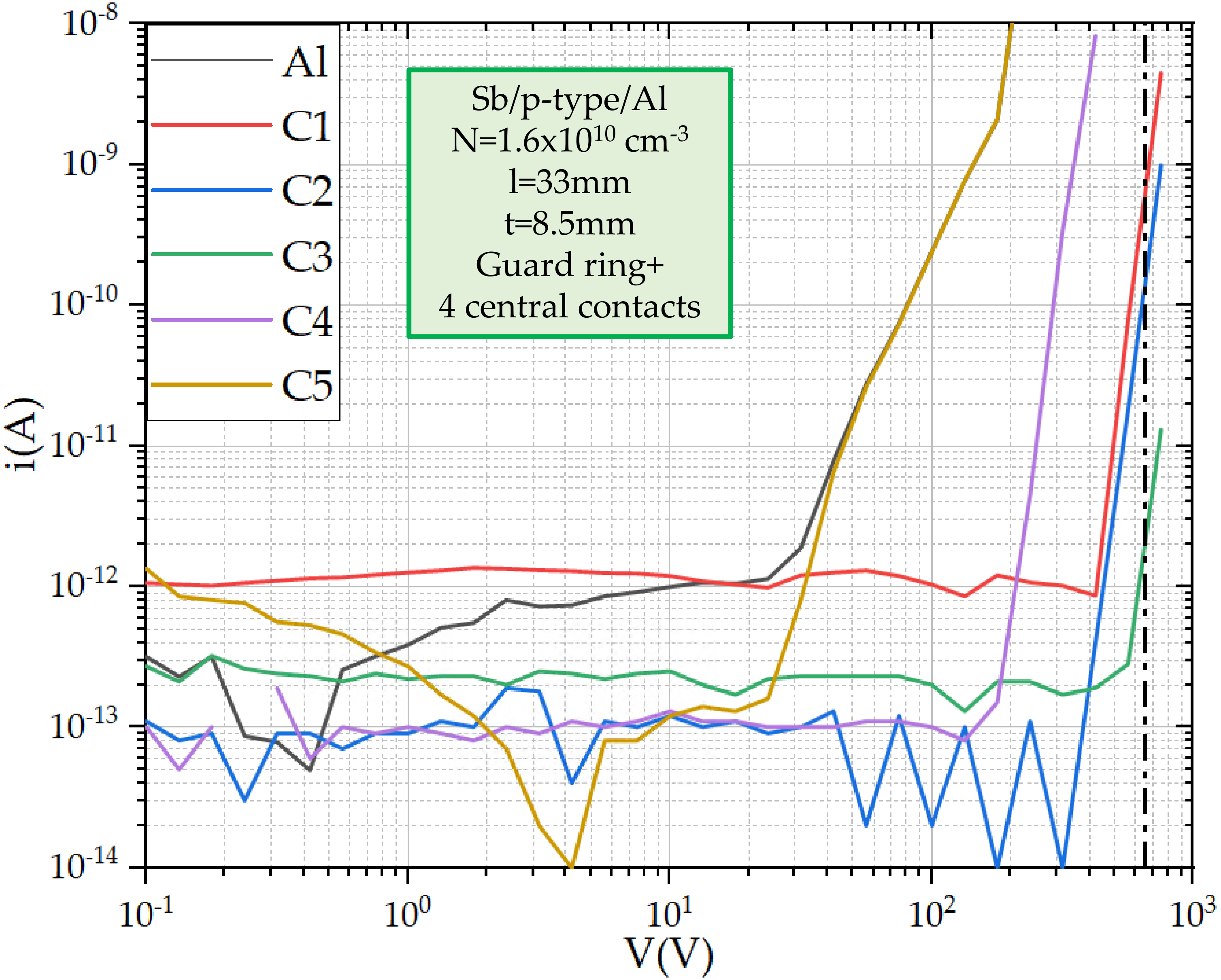
5/2024

Plating upgrade



6/2024

Plating upgrade



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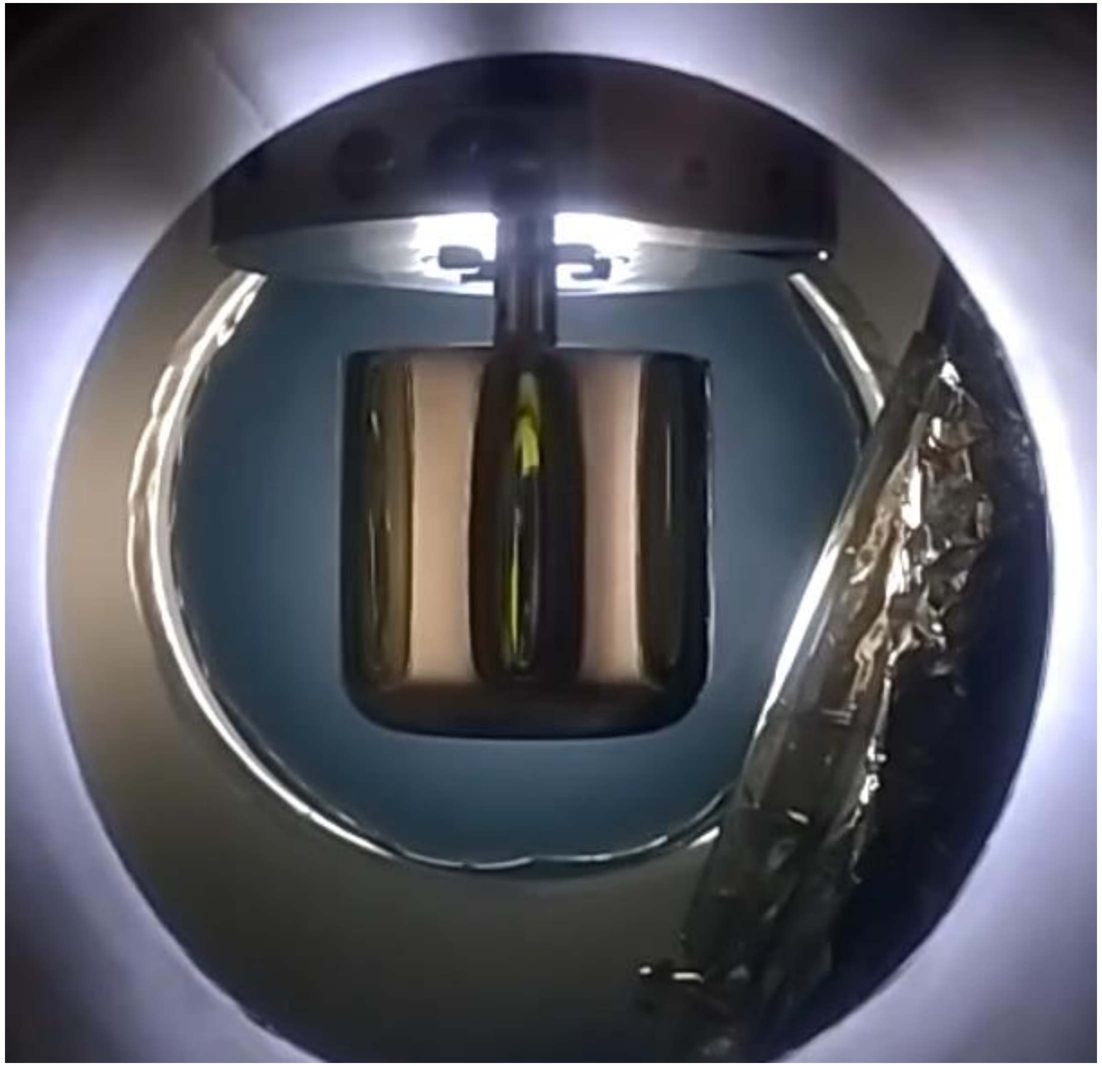
3D fabrication upgrade: crystal handling

Crystals must not be touched during and after the processes

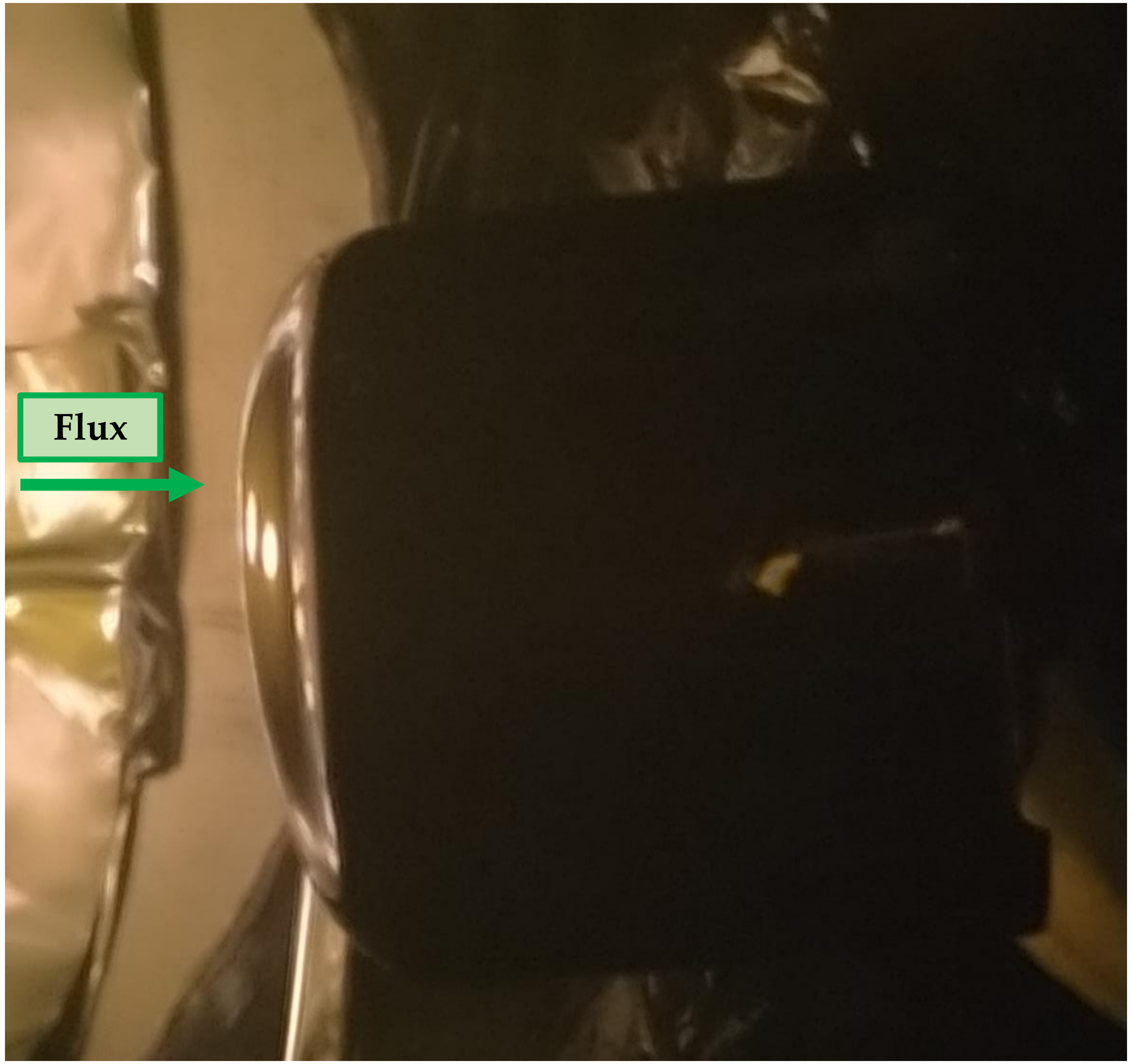
Handling system for deposition and PLM

3D fabrication upgrade: dopant deposition

Lateral surface rotating deposition

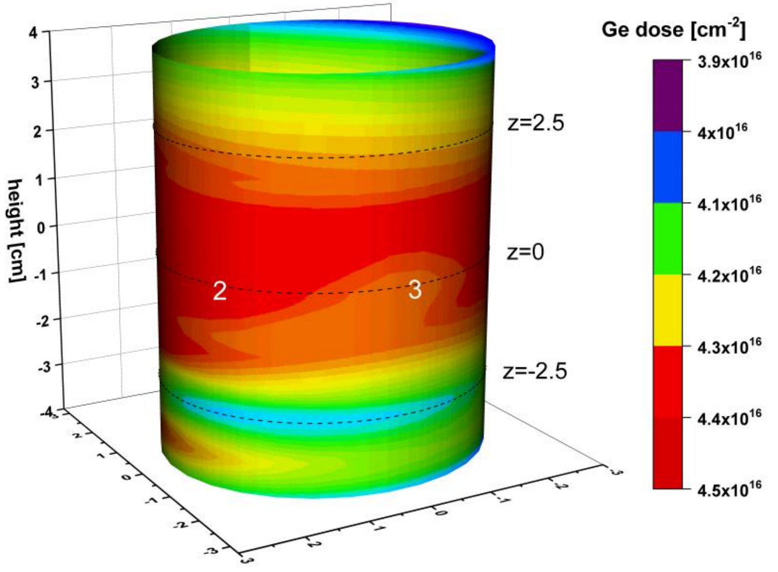


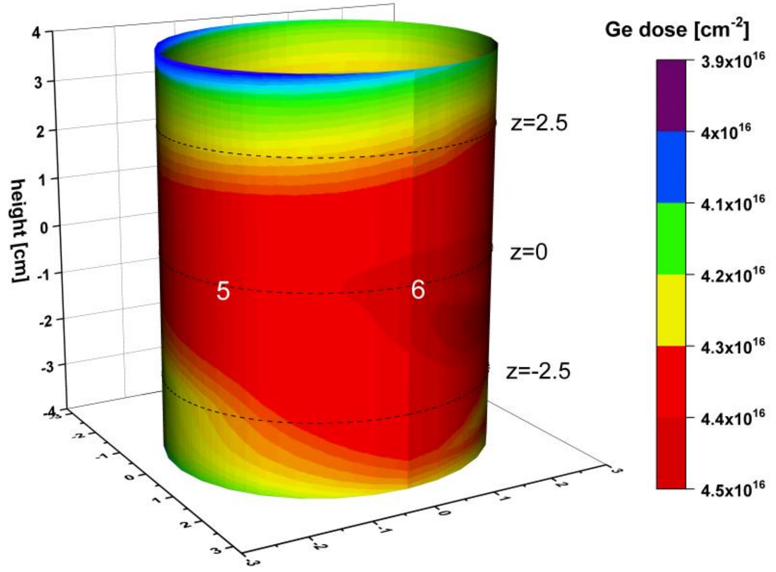
Frontal surface static deposition

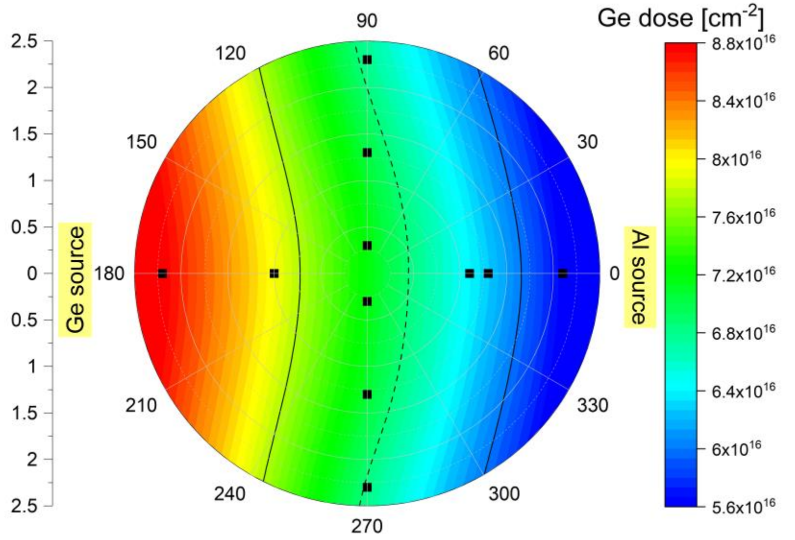


3D fabrication upgrade: dopant deposition

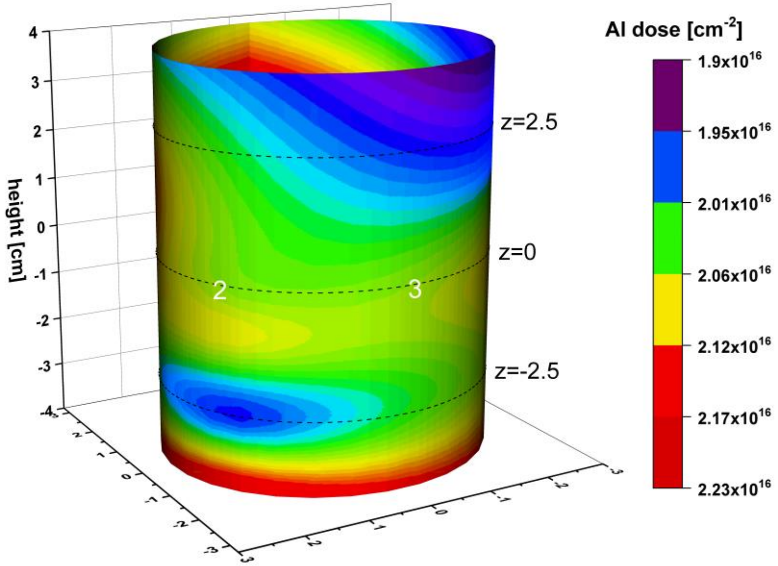
Germanium

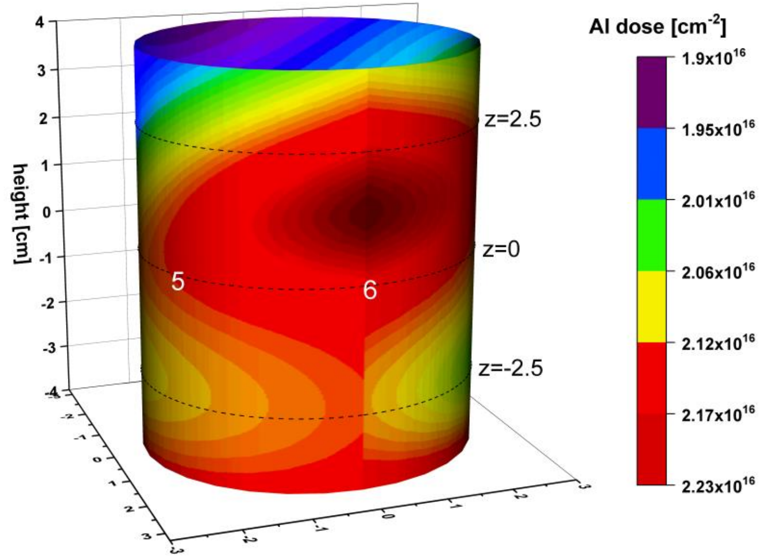


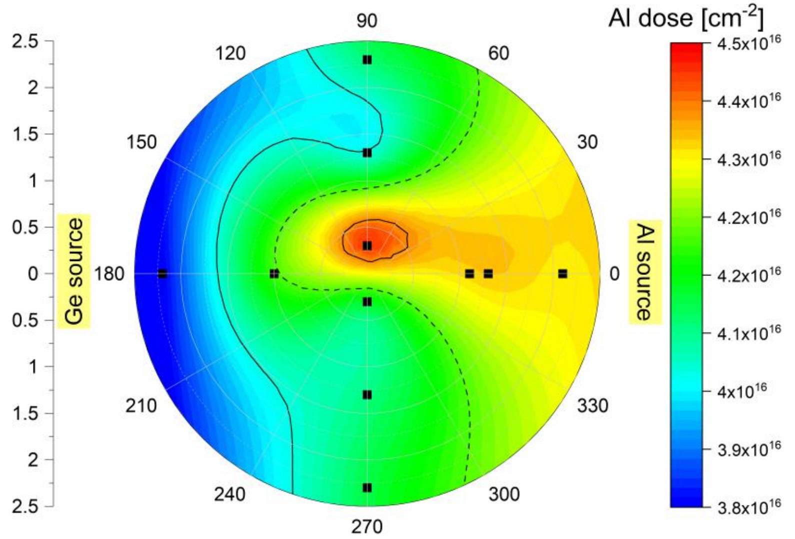




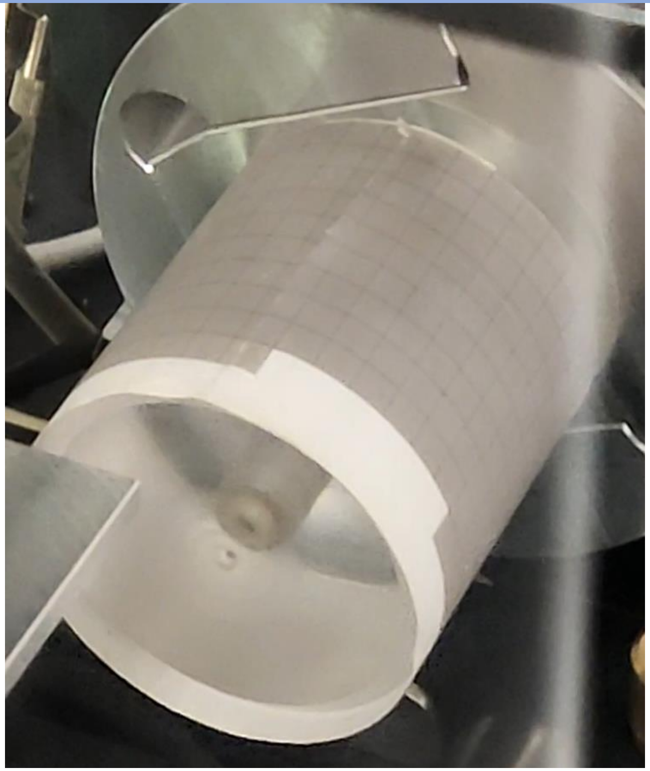
Aluminum

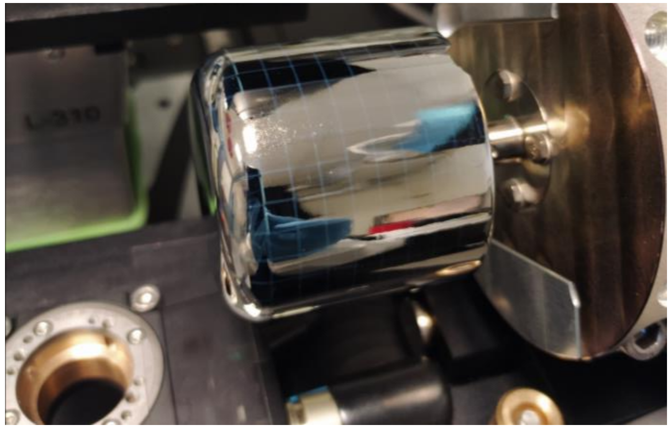






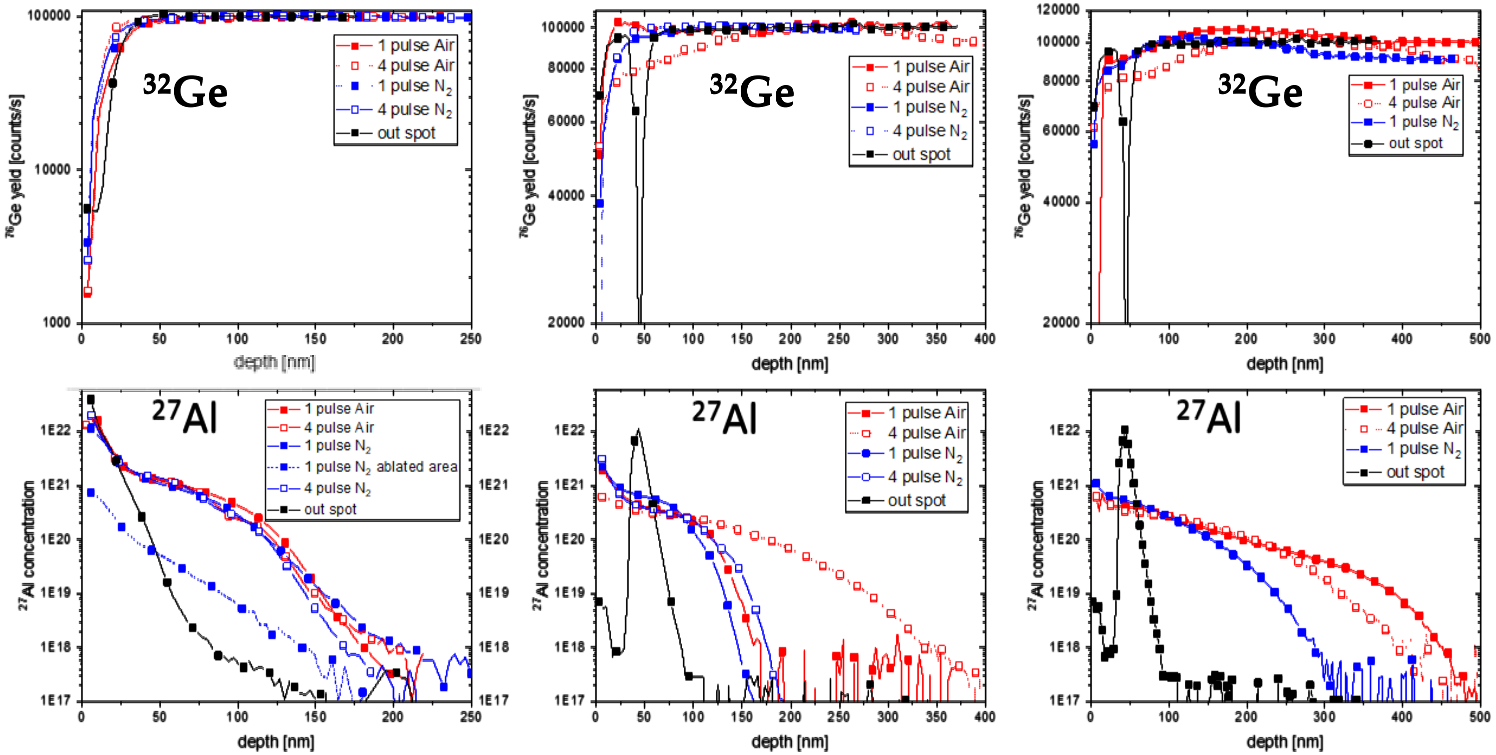
3D fabrication upgrade: Pulsed Laser Melting





AlGe deposition, 100 mJ/cm² pre-annealing to reduce morphological defects.

500 mJ/cm² only Al	500 mJ/cm² AlGe	700 mJ/cm² AlGe

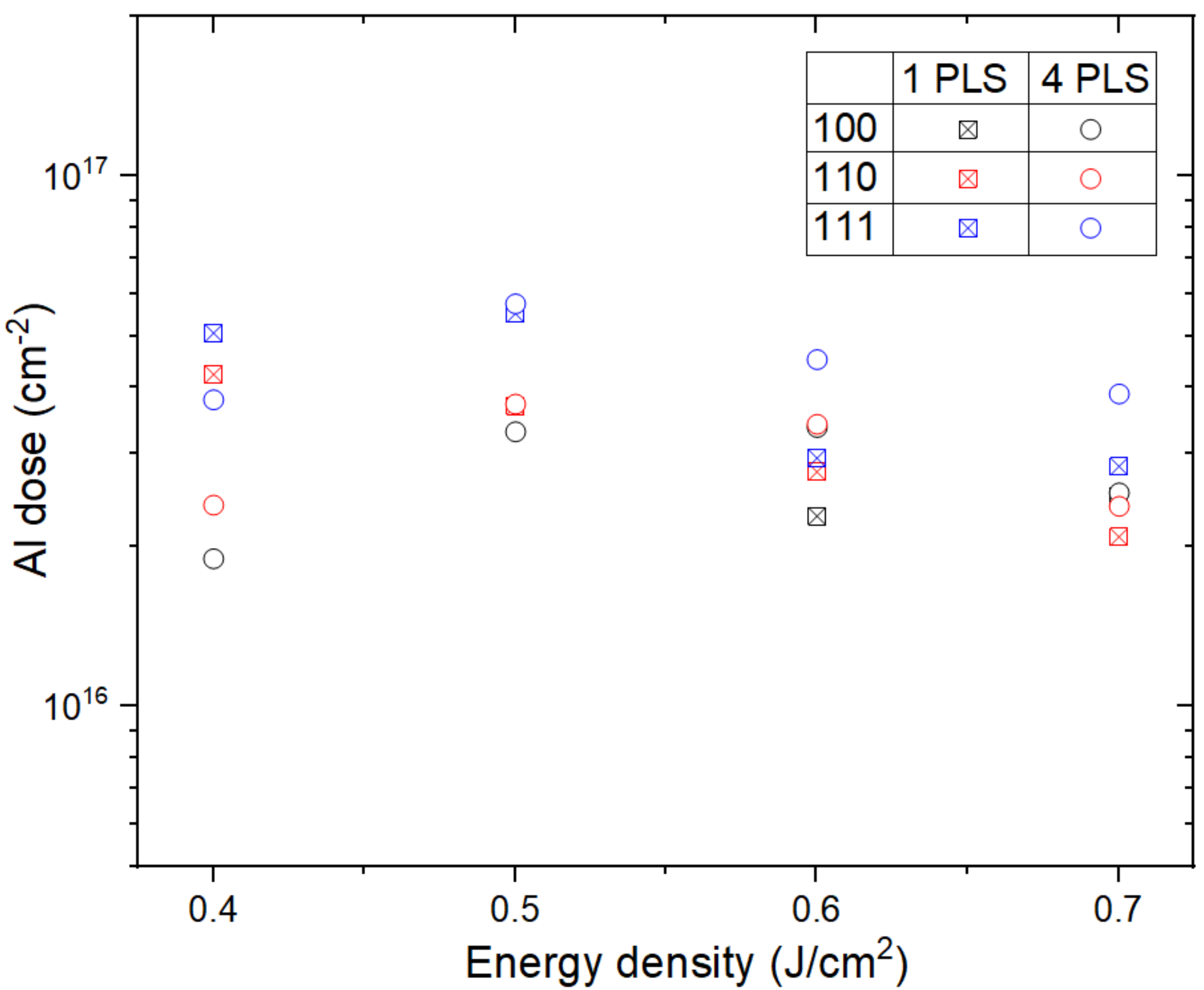


AlGe deposition, 100 mJ/cm² +700 mJ/cm².

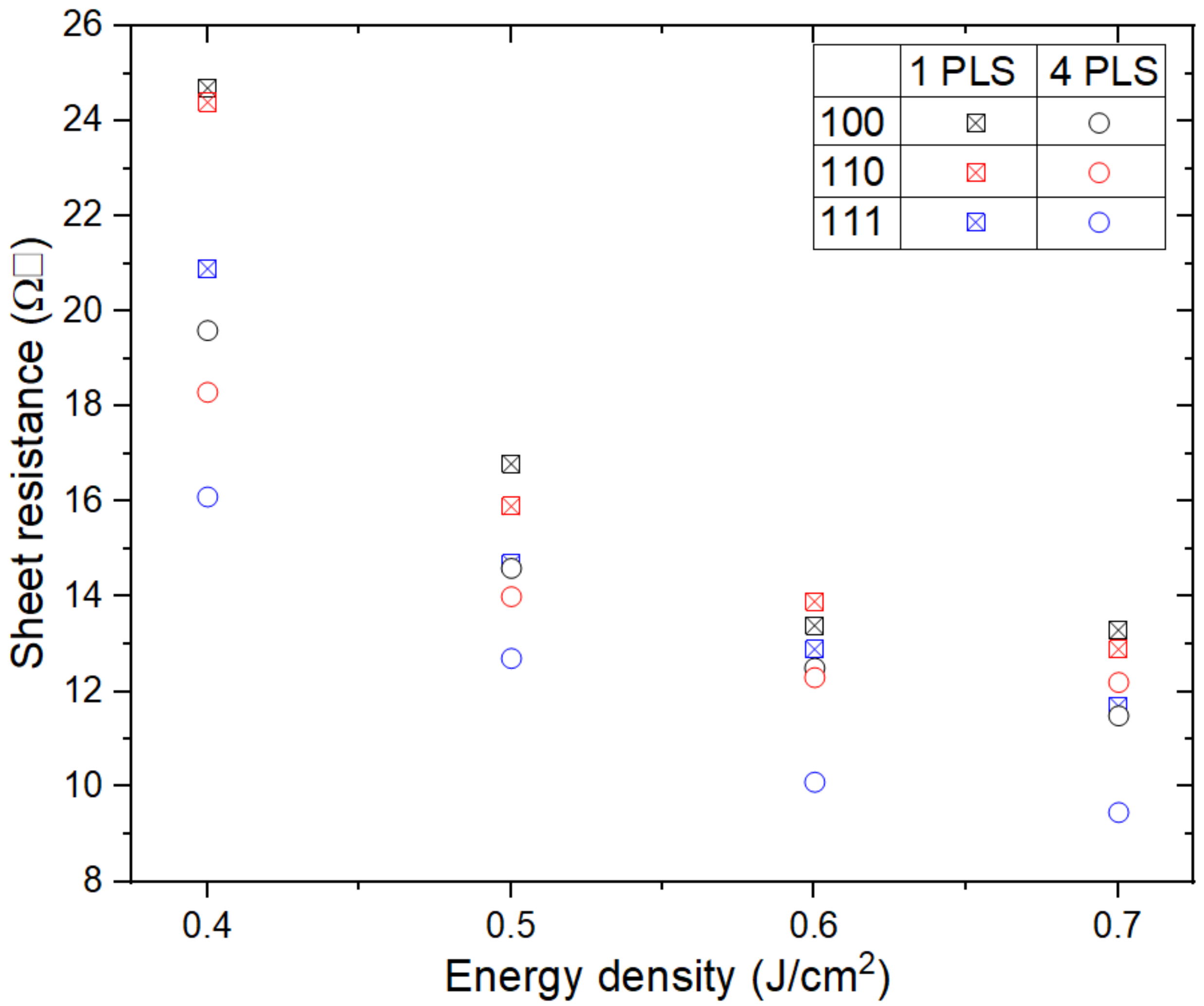
Patent WO 2021/214028 A1

3D fabrication upgrade: Pulsed Laser Melting

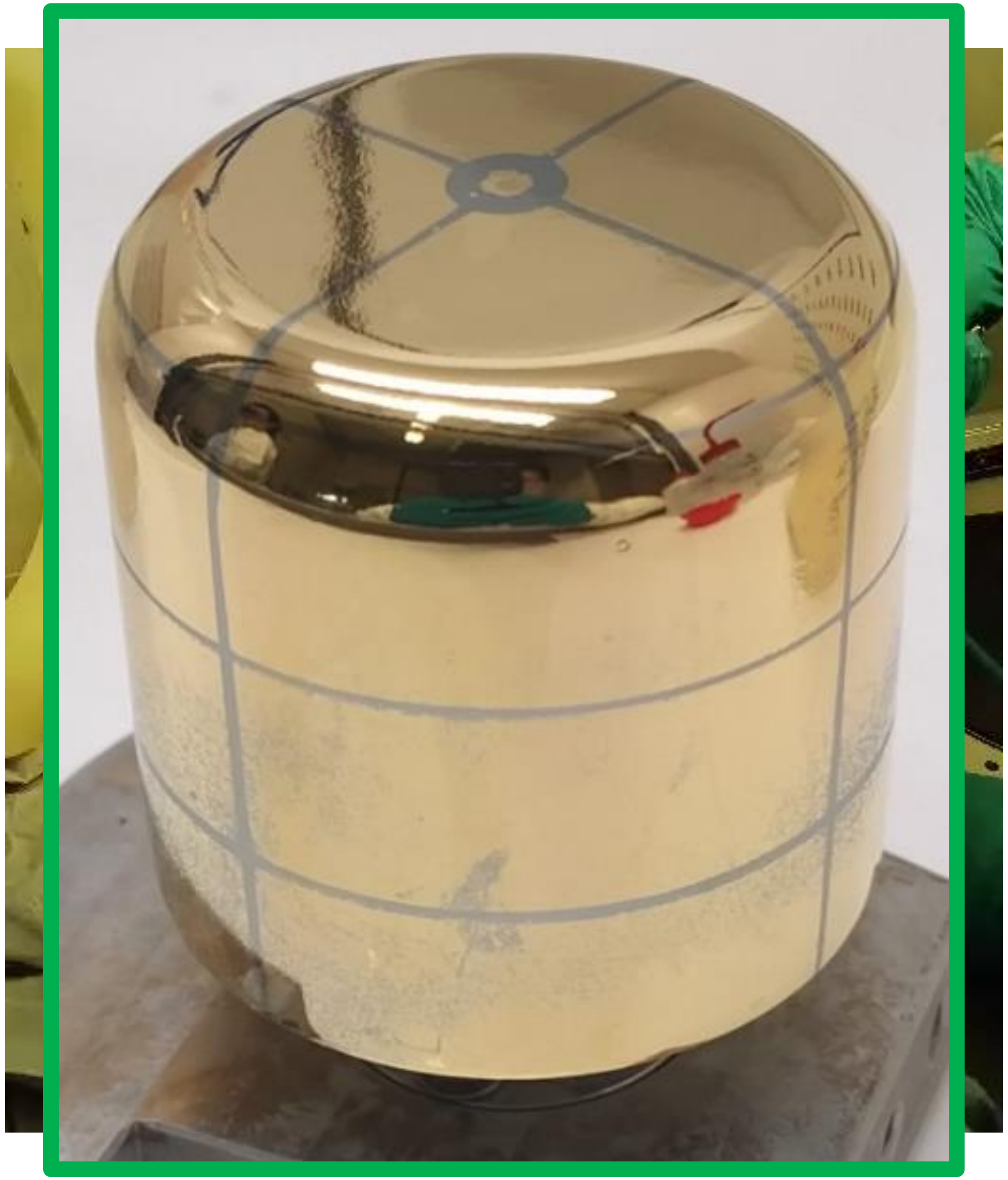
AlGe (0,1 J/cm² pre-annealing) Hall dose

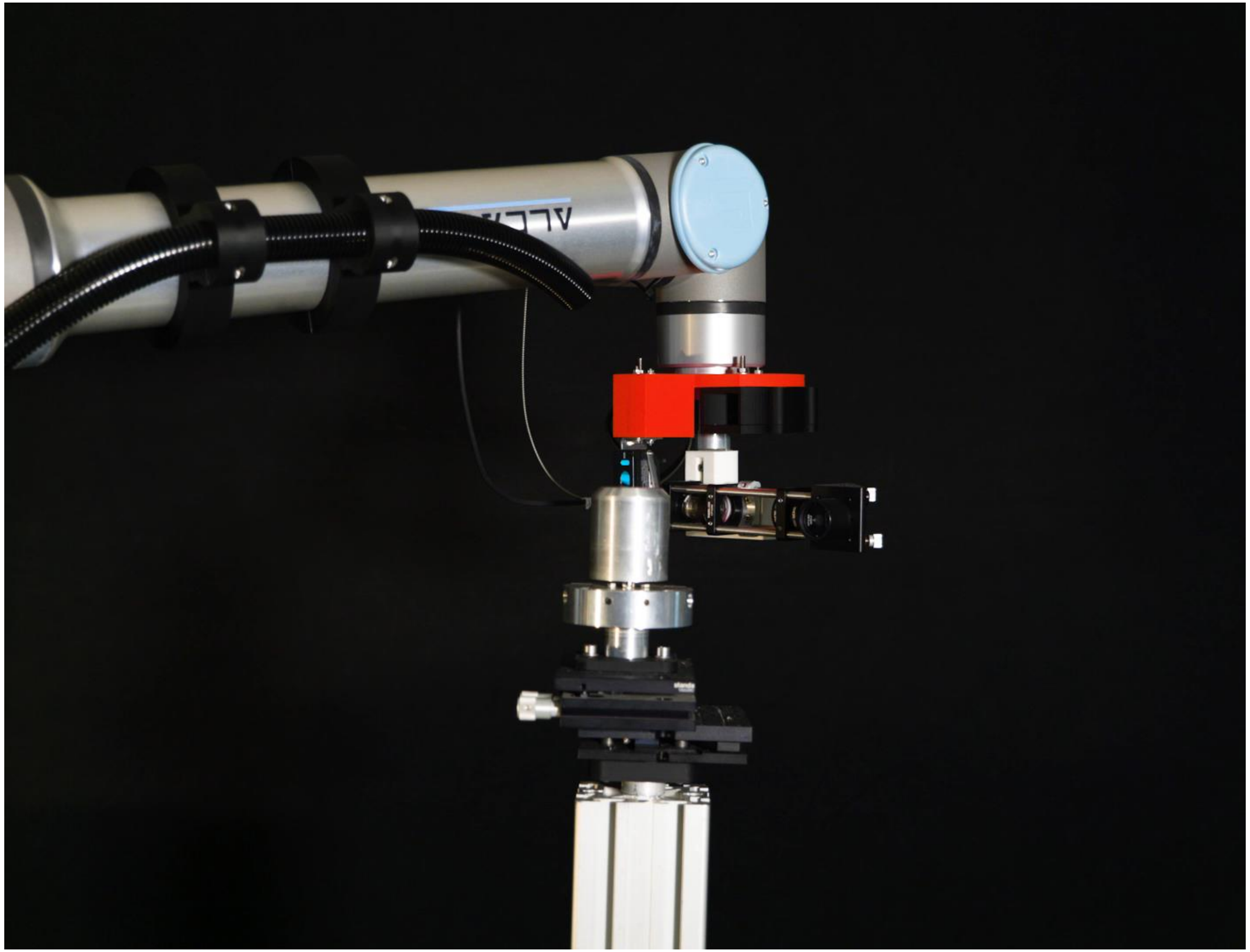


AlGe (0,1 J/cm² pre-annealing) Sheet resistance



3D fabrication upgrade: photolithography

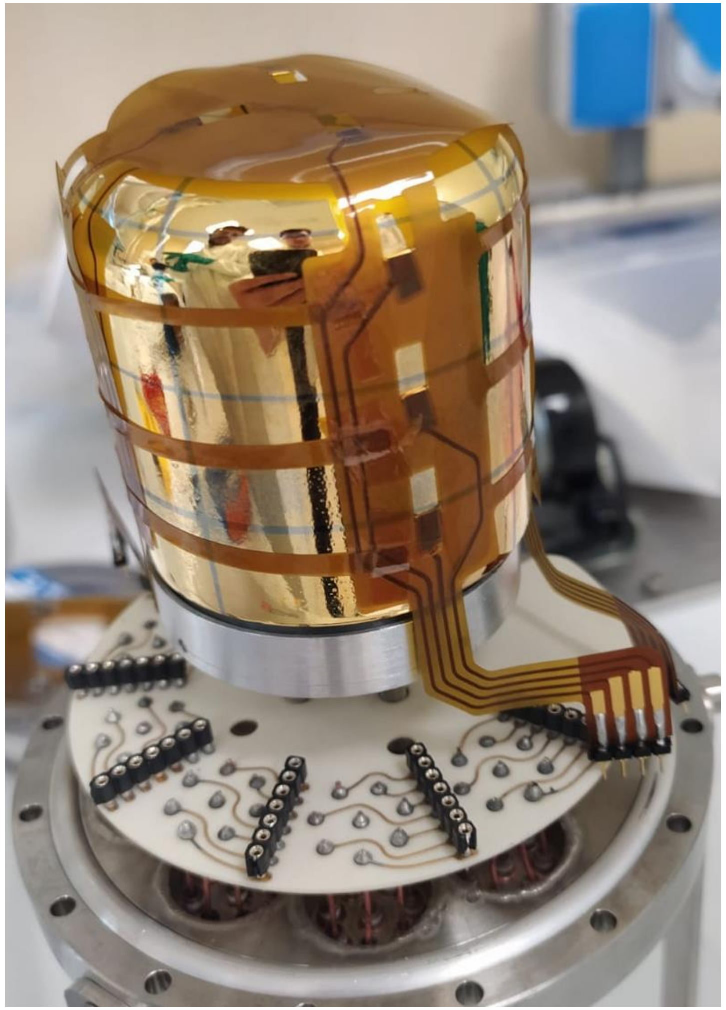


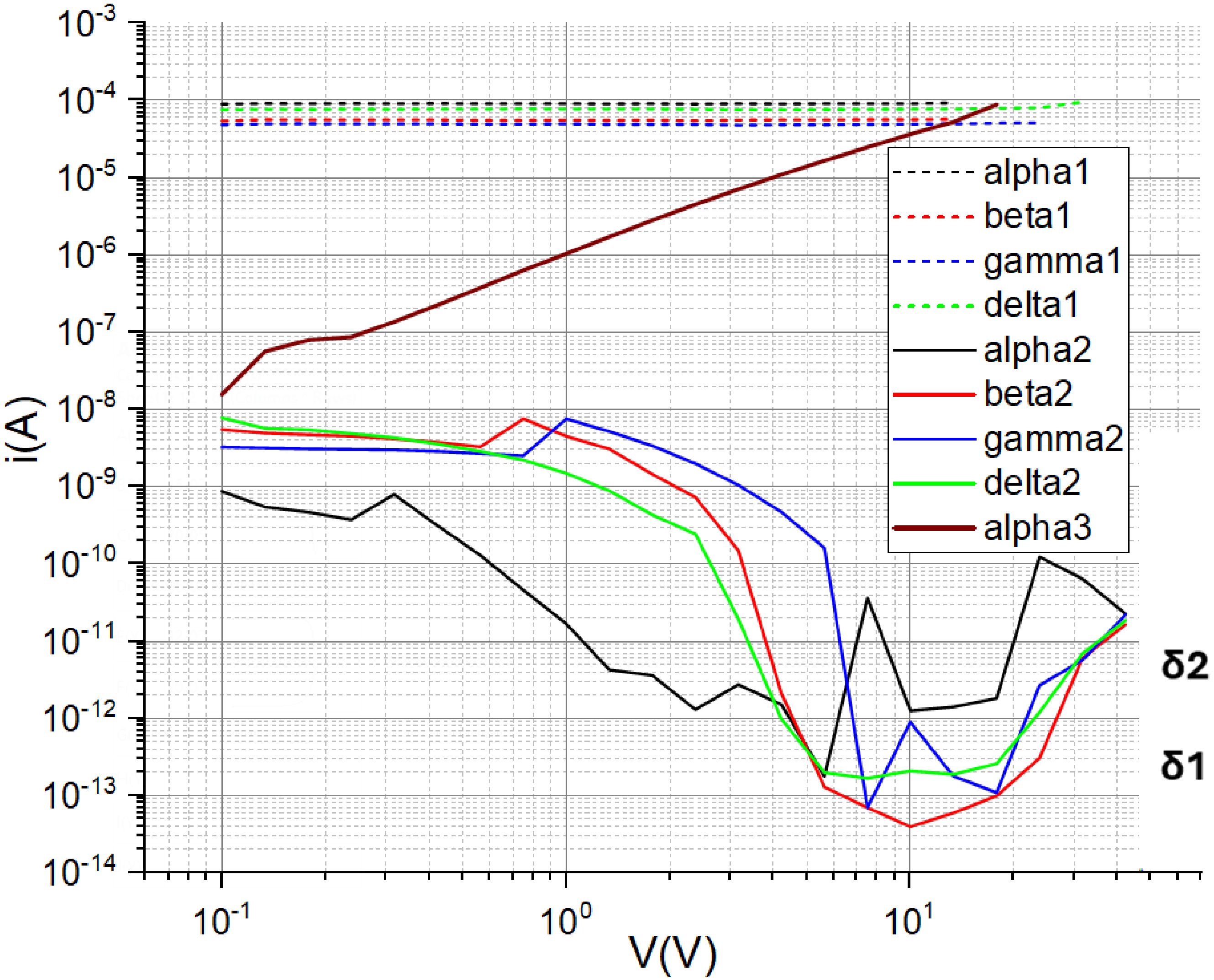


Coaxial detectors

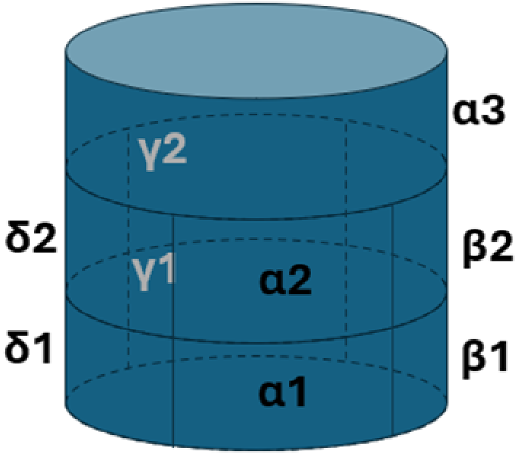
After thesis deposit

50mm d, 50mm h, n-type crystal, AlGe PLM junction, Li core



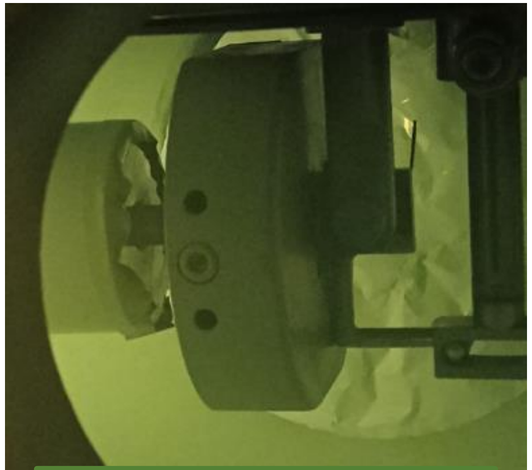


R(row2)
$\alpha\beta$ = 14 TΩ
$\beta\gamma$ = 3 TΩ
$\gamma\delta$ = 8 TΩ
$\delta\alpha$ = 3 TΩ

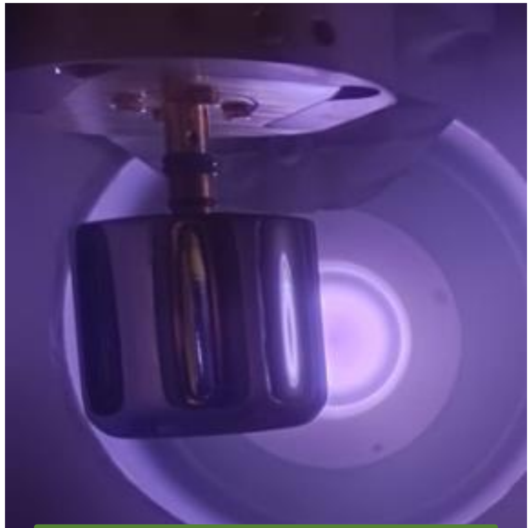


Coaxial detectors

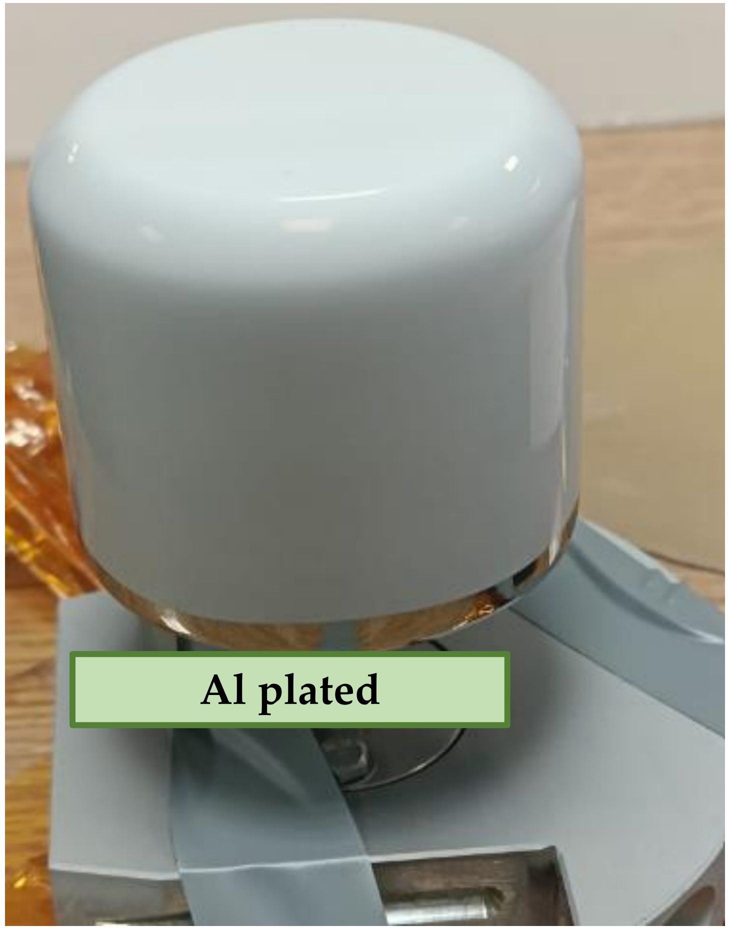
50mm d, 50mm h,
n-type crystal,
AlGe PLM junction, Li core



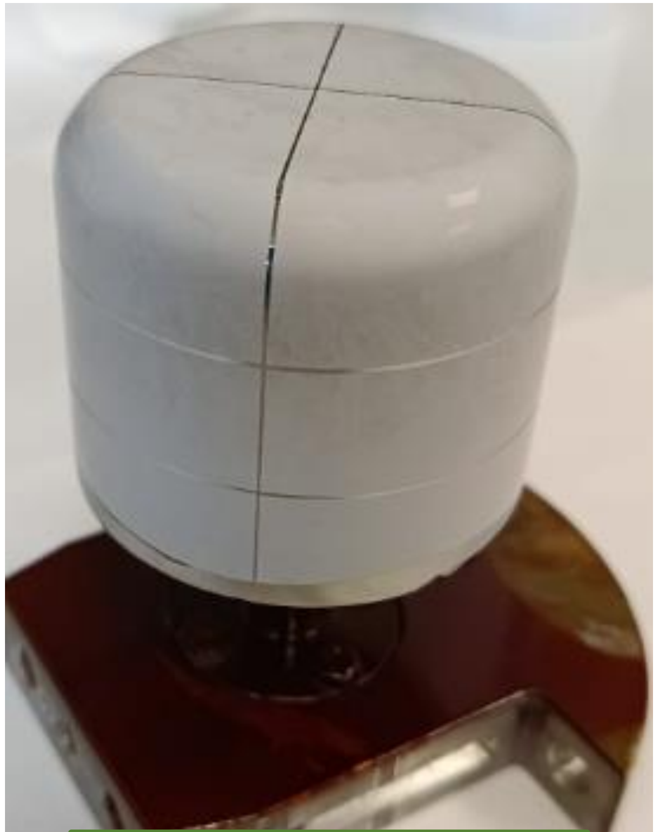
Frontal Al plating



Lateral Al plating



Al plated



After photolithography

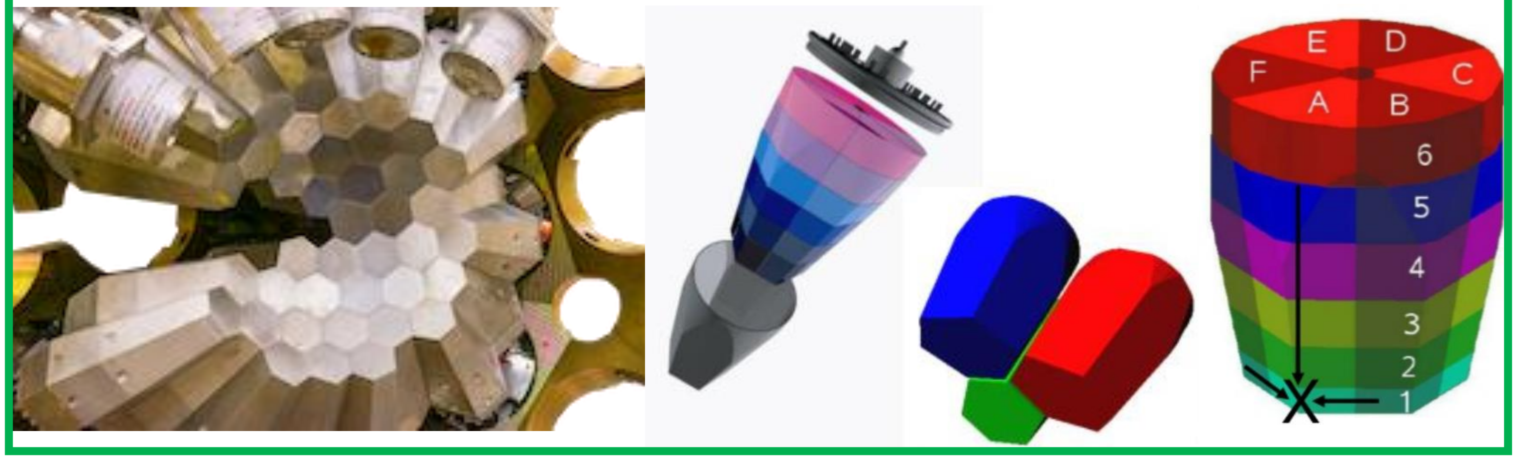
Conclusions

- We developed thin and thermally stable hyperdoped n-junction with Sb using magnetron sputtering and Pulsed Laser Melting technique, which preserve bulk purity.
- We tested different lithographic procedures on different planar geometries and started testing on coaxial detectors.

Future plans

- We need to investigate pressure effects and plating techniques (Au on Al bi-plate) on the thin junction to increase maximum reverse voltage on thick samples and extend it from planar to coaxial geometries.
- Industrialization (MIRION Technology research collaboration agreement) from this work.

AGATA (Advanced Gamma Tracking Array)



Collaboration Agreement

MIRION

INFN
LNL

THANK YOU FOR YOUR ATTENTION