

# 4th AGATA-GRETINA/GRETA Tracking Arrays Collaboration Meeting



## HPGe crystal research at LNL-INFN

**Walter Raniero**

INFN – LNL

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# OUTLINE

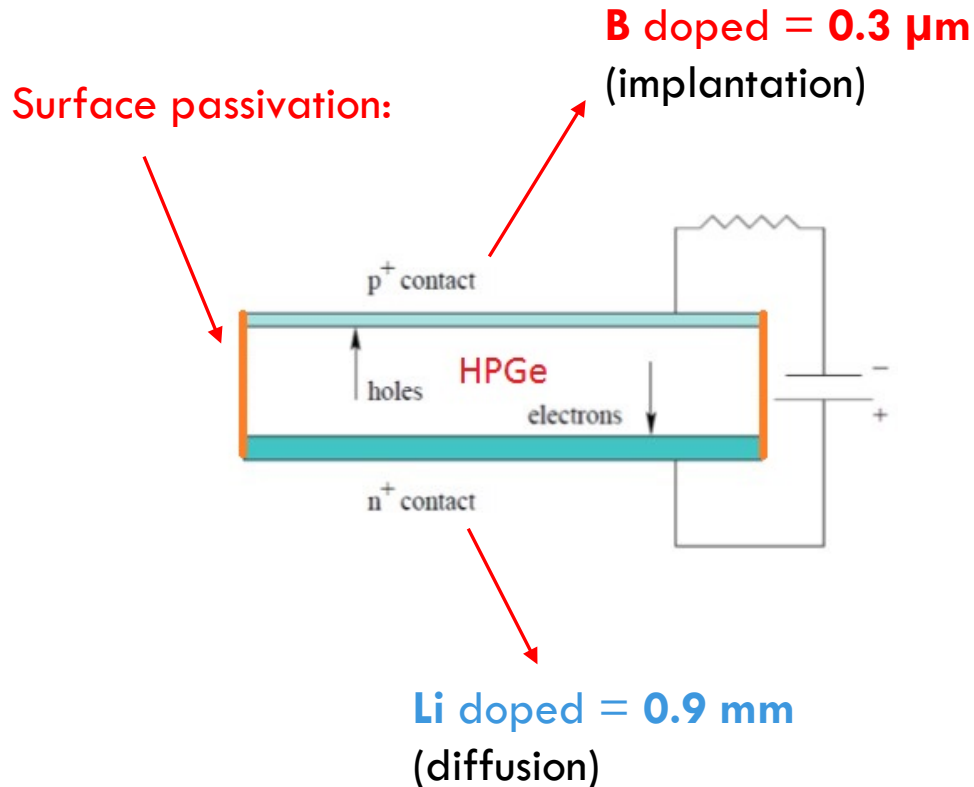
- Introduction: HPGe gamma detectors
- Gamma detector state of the art
- *PLM (Pulse Laser Melting)*: Next generation of segmented contact/junction on HPGe detectors
- PLM Planar gamma segmented detectors
- PLM Coaxial gamma segmented detectors
- Neutron damage in PLM planar segmented detectors

# Schematic *HPGe* planar gamma detector

**HPGe**

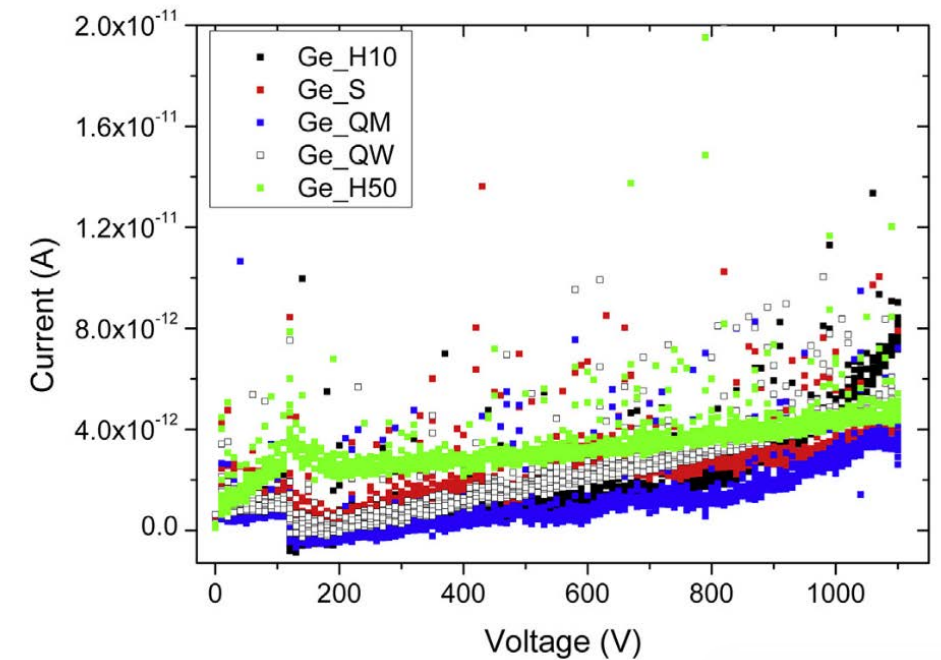
Charge carrier density:  $10^{10} \text{ cm}^{-3}$

**Chemical passivation:**



$\text{HNO}_3/\text{HF}$  (3:1) etching  
and quenching bath:

- Water ( $\text{Ge}_W$ )
- Methanol ( $\text{Ge}_{QM}$ )
- Sulfide termination ( $\text{Ge}_S$ )
- Hydride termination ( $\text{Ge}_{H10}$ )



S. Carturan et al., *Mater. Chem. Phys.* 2015

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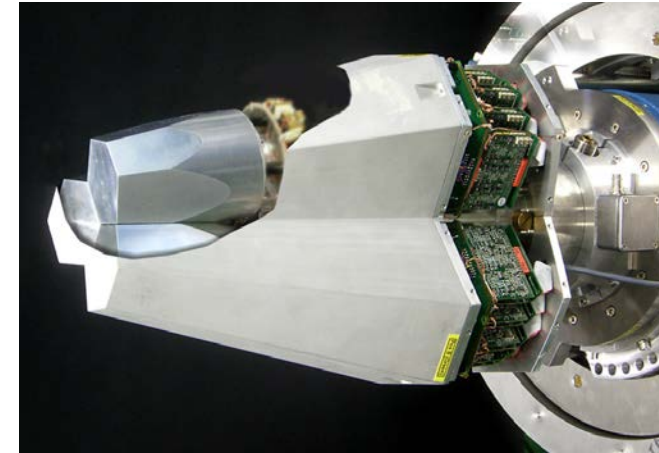
# Gamma detector state of the art (**AGATA** and **GRETA**)

Encapsulated coaxial HPGe n-type detectors

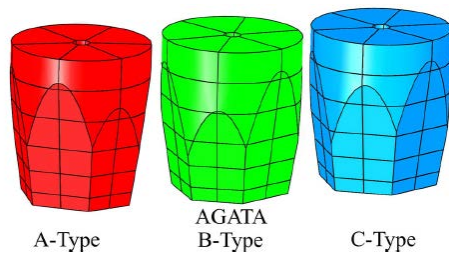


- 2Kg weight
- 80 mm diameter
- 90mm long
- 1 core inner contact
- 36 segments

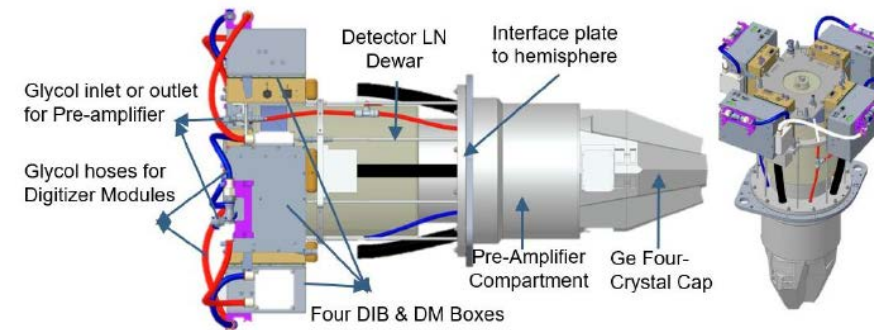
**ATC** (AGATA triple cluster detector)



3 asymmetrical HPGe detector (AGATA)

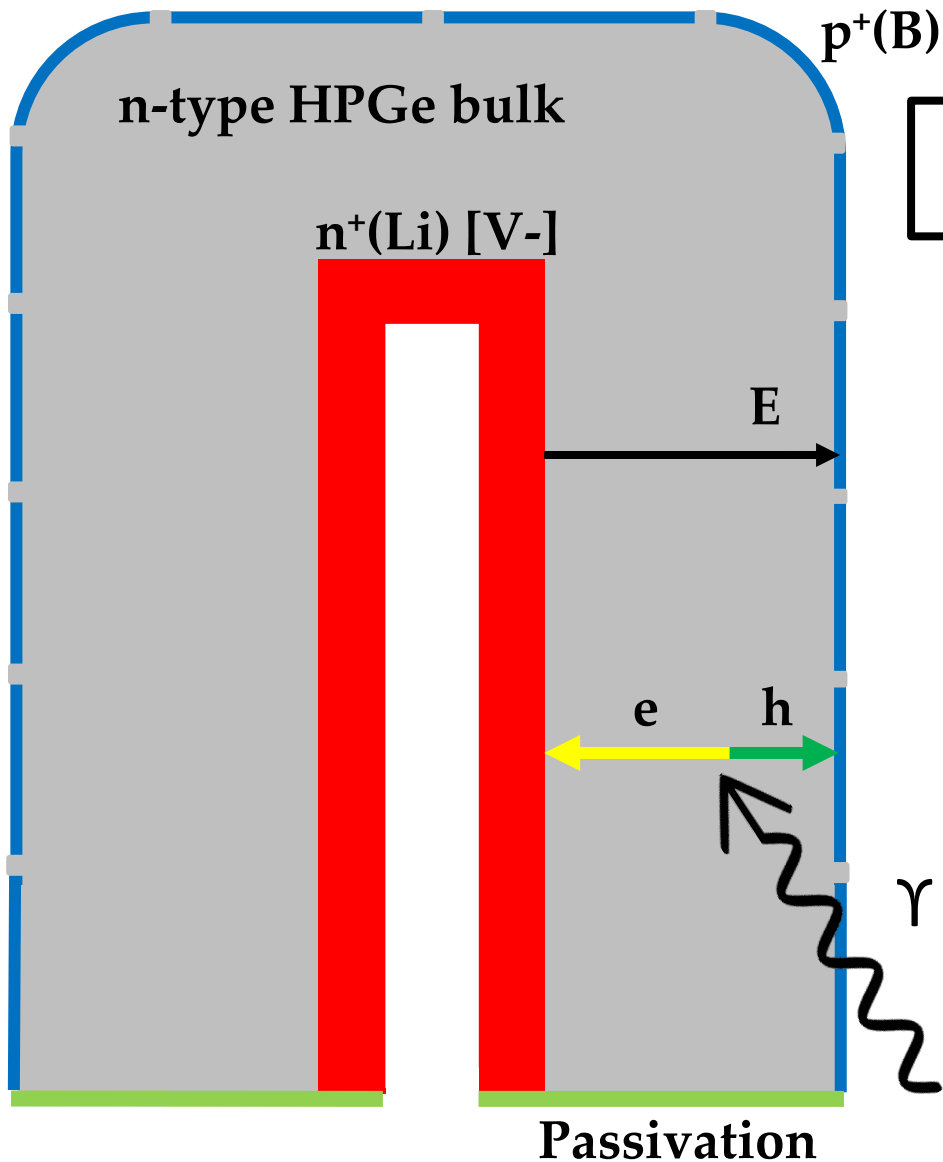


**GRETA** (Quad Detector Module)



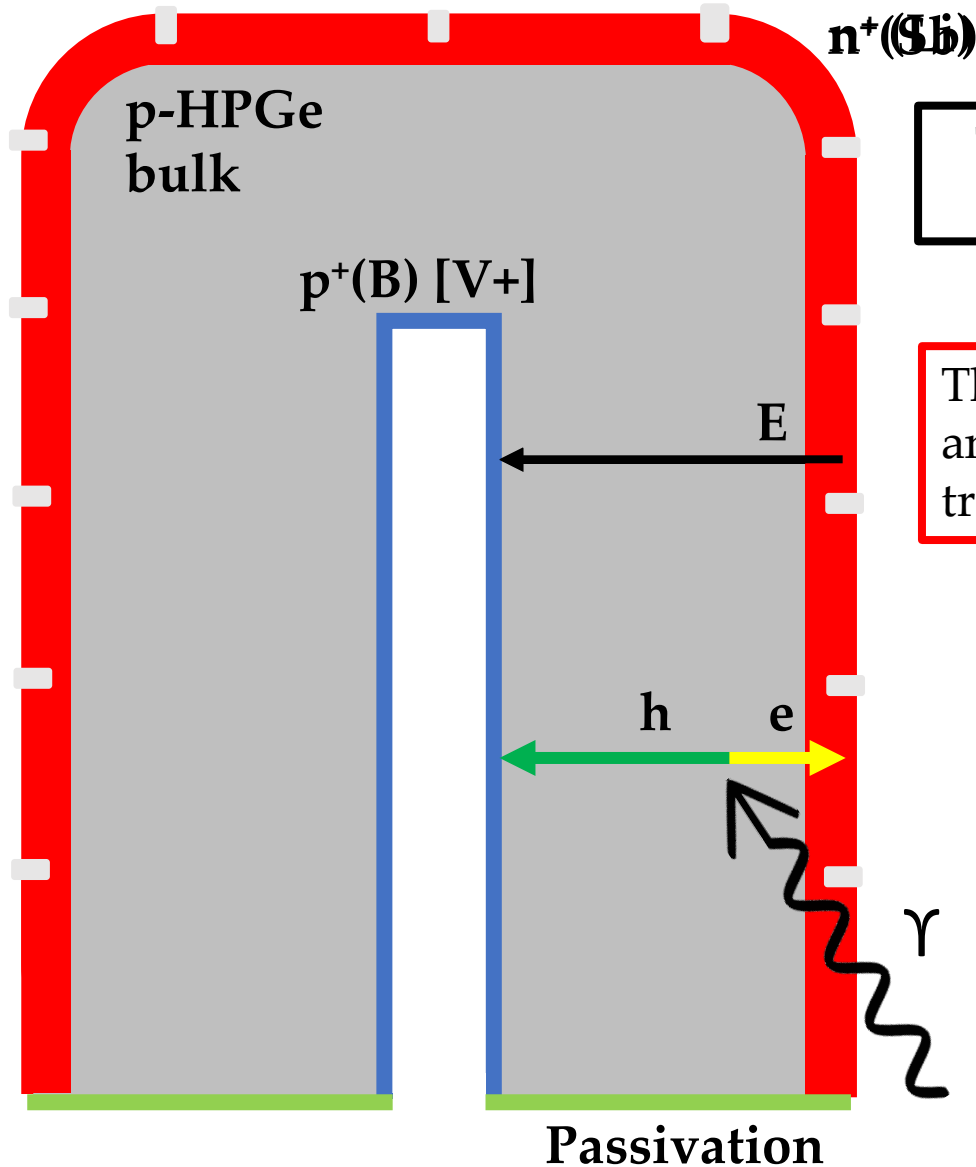
J. Eberth et al. **Eur. Phys. J. A** (2023) 59: 179

# Schematic coaxial segmented *n-type* detector



The boron junction is thin,  
and easily segmentable

# Schematic coaxial segmented *p-type* detector



The boron junction is thin, and easily segmentable

The lithium junction is thick and not stable under annealing treatments

Polarity inversion demanded to test higher damage resistivity (hole trapping by neutron damage)

J. Eberth et al. *Eur. Phys. J. A* (2023) 59: 179

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

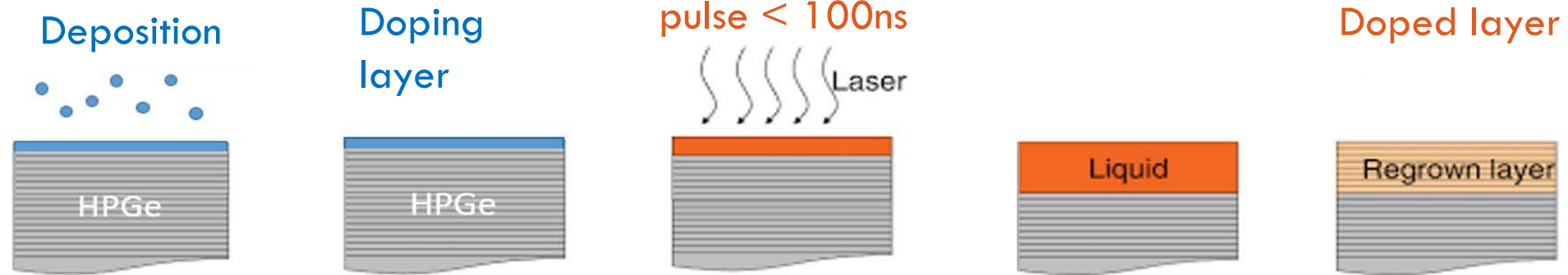
p-HPGe with segmentable  $n^+$  junction collecting electrons.

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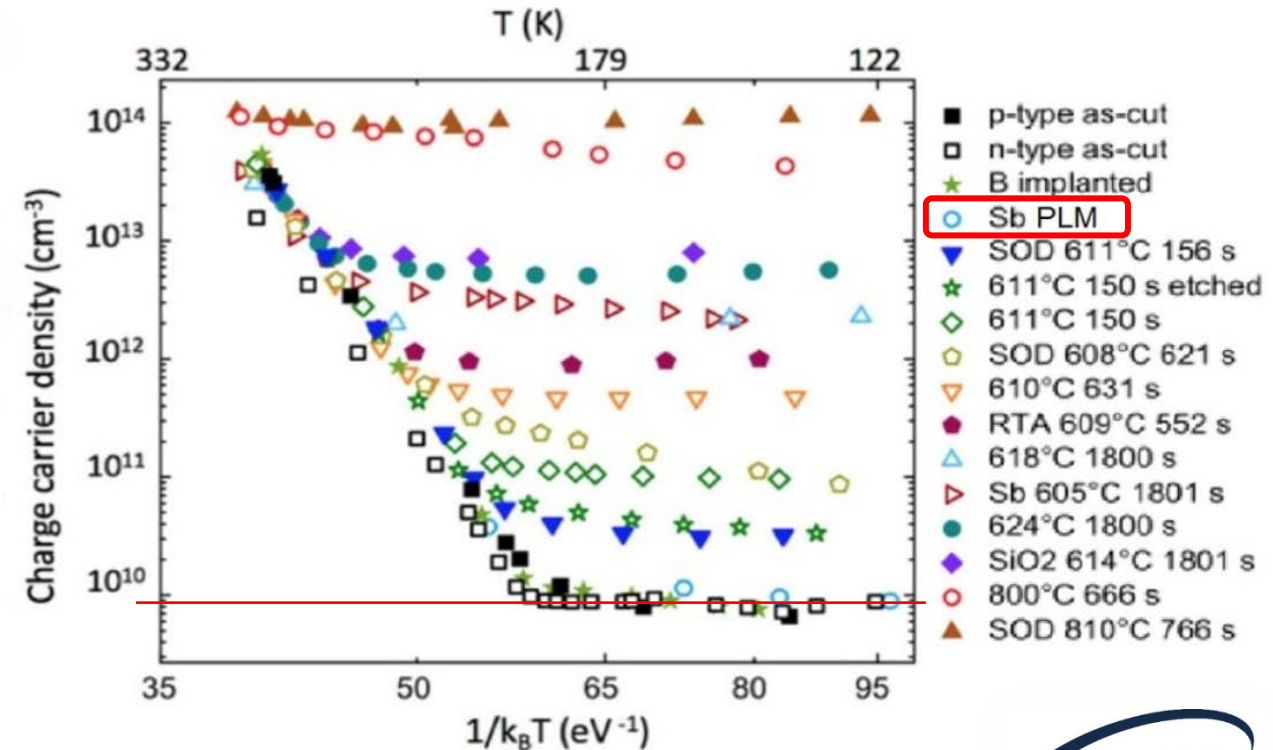
# New contact/junction on HPGe: PLM (Pulse Laser Melting)



## Advantages:

- Melting temperature is reached - short time (<100 ns)
- Only the surface (< 200 nm) is melted, the bulk is at room temperature
- High dopant concentrations with very sharp dopant profile
- Doping with heavy elements without crystal damage
- Very clean process suitable for preserving the Ge hyperpurity
- Suitable for complex contact geometries (**segmentation**)

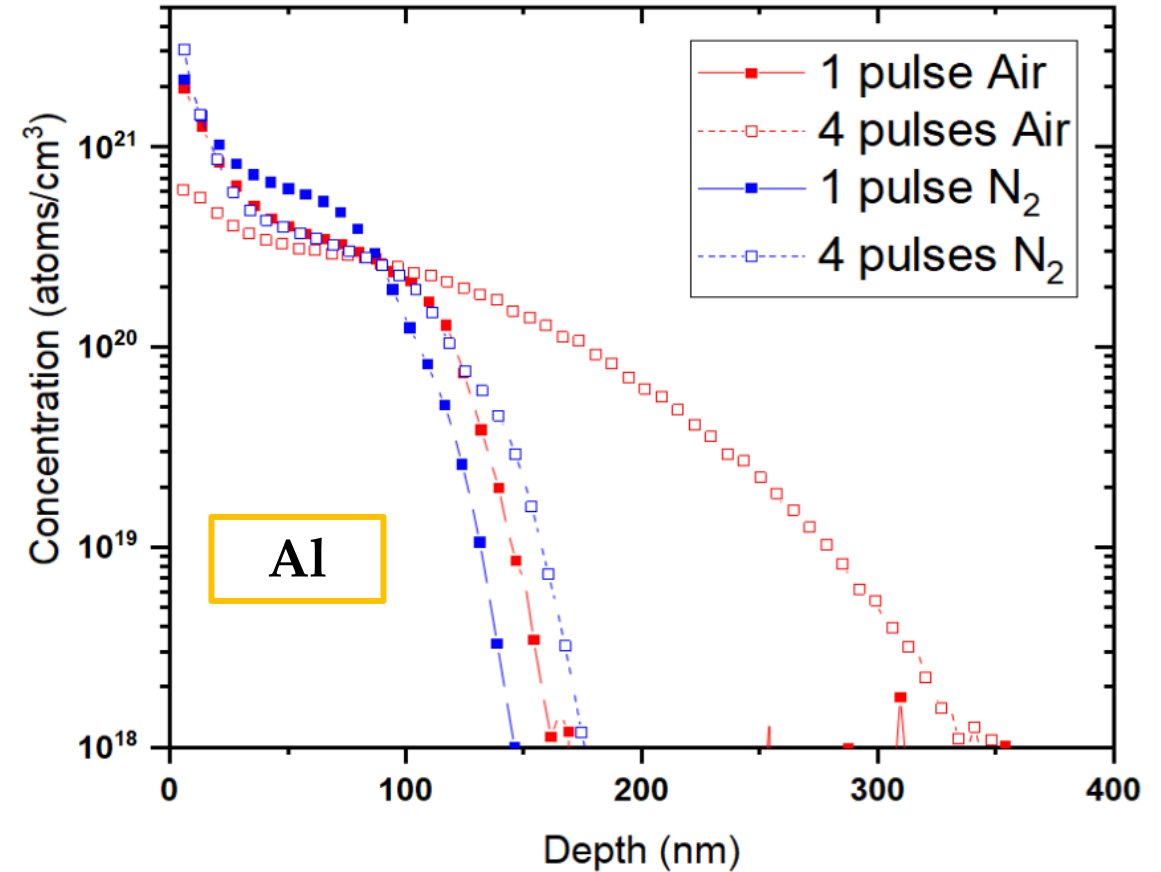
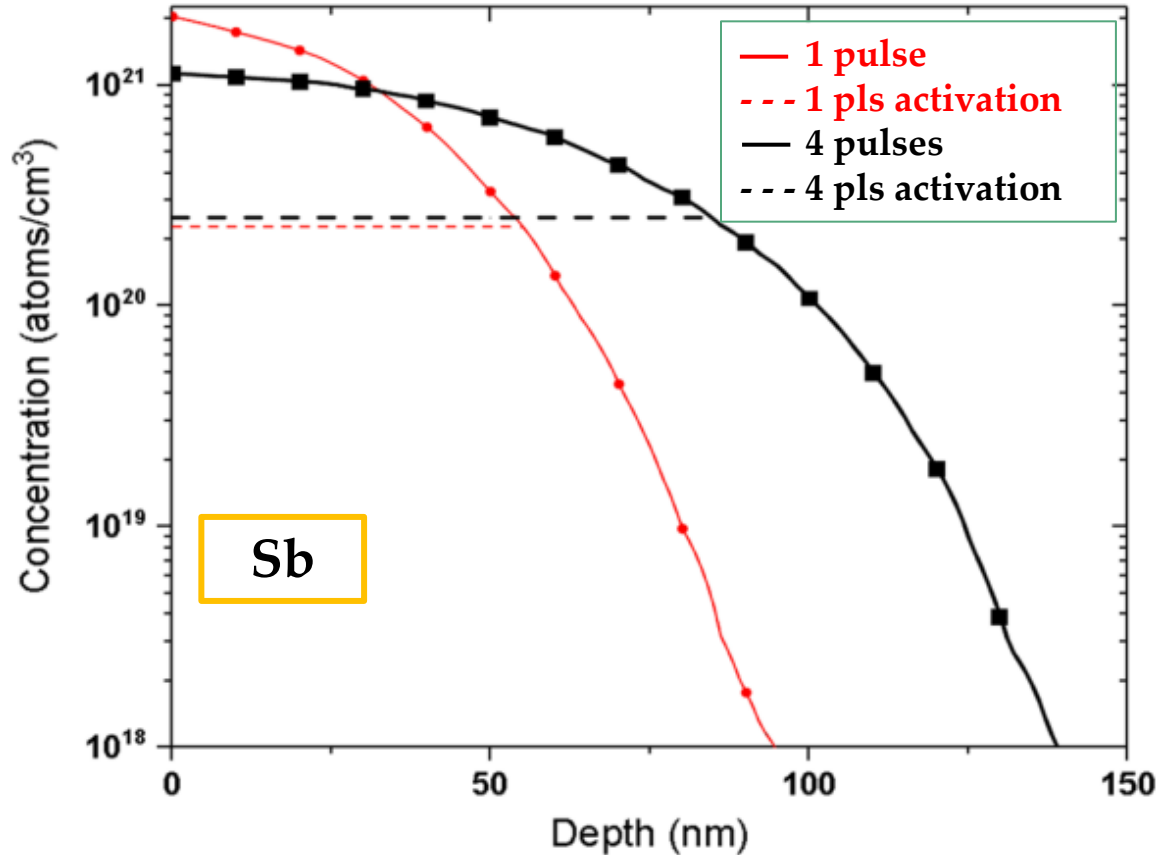
Impurities concentration in Ge bulk



V. Boldrini et al., *Journal of Physics D: Applied Physics* (2018) volume 52, 3

# New contact/junction on HPGe: Chemical concentration profile

SIMS (Secondary Ions Mass Spectrometry)

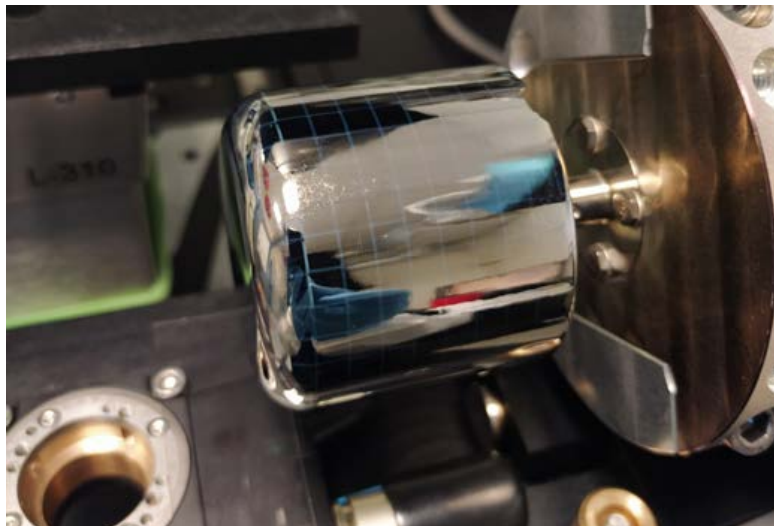
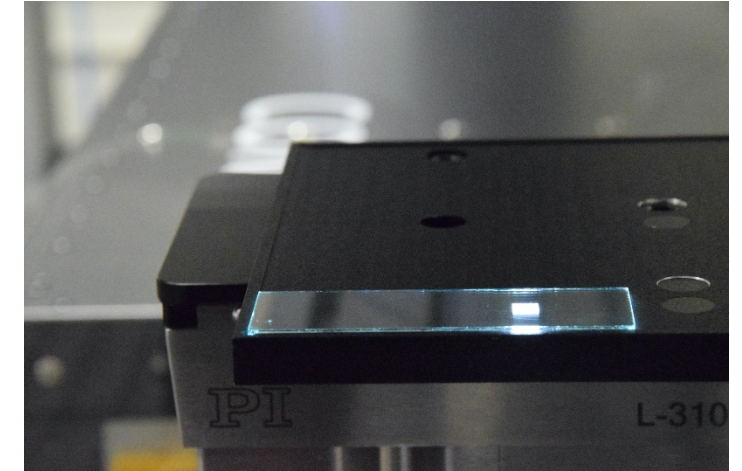
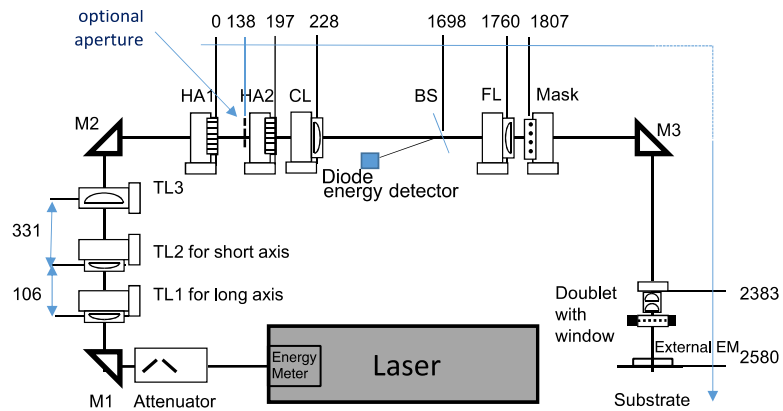


S. Bertoldo et al., *Eur. Phys. J. A* (2021) 57:177

J. Eberth et al. *Eur. Phys. J. A* (2023) 59: 179

# New contact/junction on HPGe: PLM Laser technology

## Excimer KrF laser



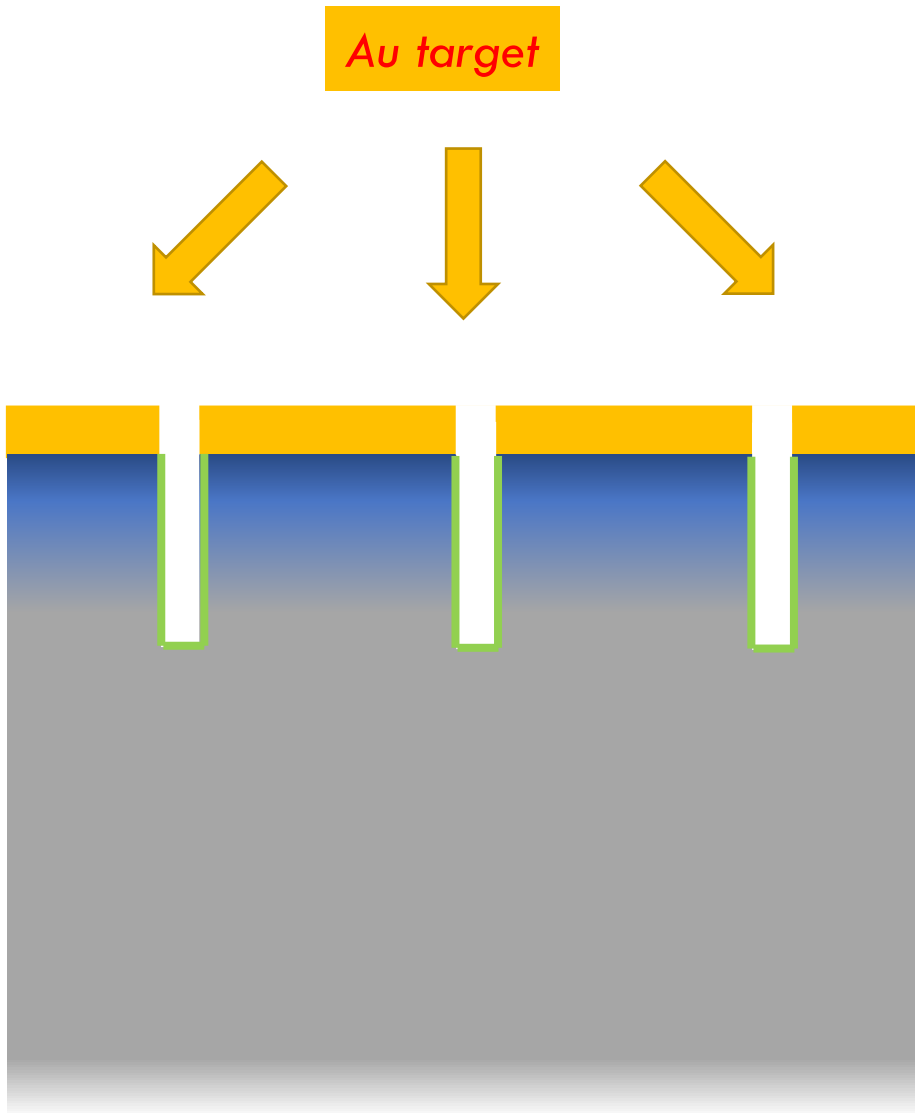
- $\lambda=248$  nm, 22 ns
- Frequency: 1-10 Hz
- ED= 50-1300 mJ/cm<sup>2</sup>
- Square 5x5mm<sup>2</sup> spot
- Homogeneity: < 2%
- lateral resolution <30  $\mu$ m
- Motorized XYZ stage



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# PLM contact/junction: 1° type Segmentation



Full area ← PLM



## Au deposition

100 nm PVD deposition of Au in Ar plasma with ultrapure target in vacuum ( $10^{-6}$  mbar)

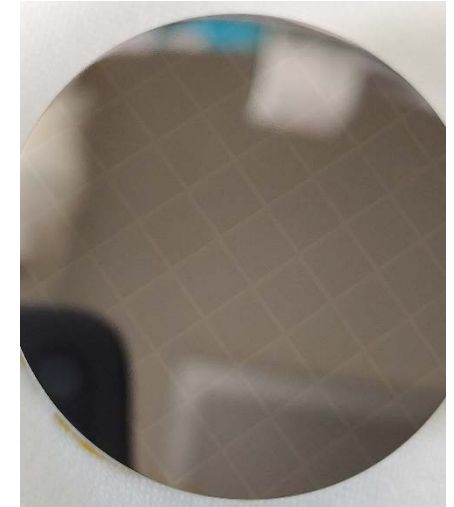


## Photolithography

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

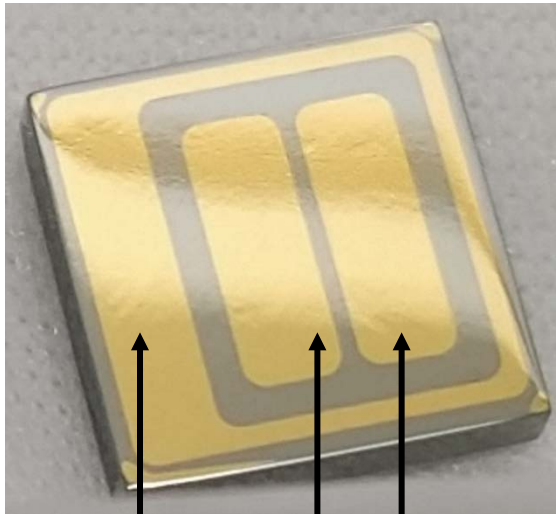


**Intercontact gaps passivation**  
(3:1)  $\text{HNO}_3$  : HF etching followed by chemical quenching passivation.



# Thin planar HPGe detectors

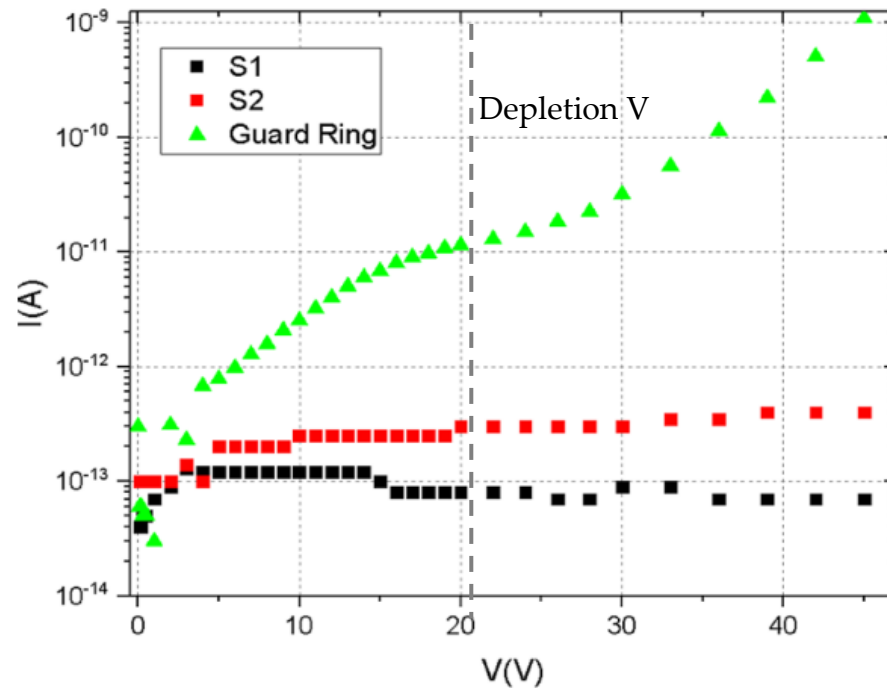
Sb  $n^+$  junction, p-type HPGe,  
B  $p^+$  L=10mm, t=2mm



Guard Ring S1 S2

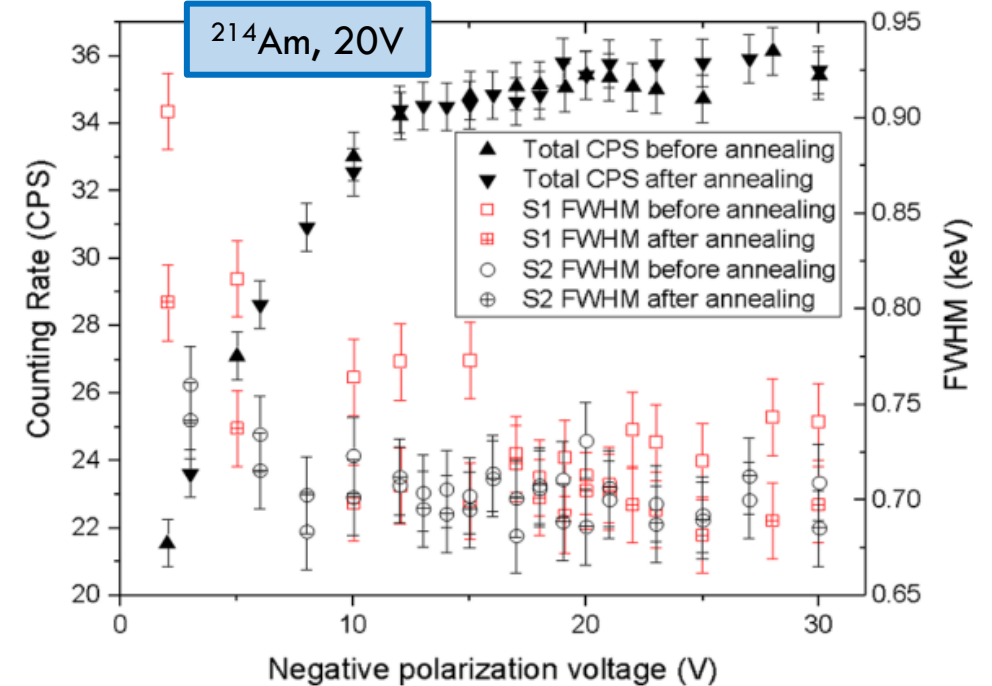
Measured before and after recovery annealing and re-passivation to test junction stability

Electrical test:  
reverse I-V characteristics



Gamma ray test:  
241Am spectra acquisition

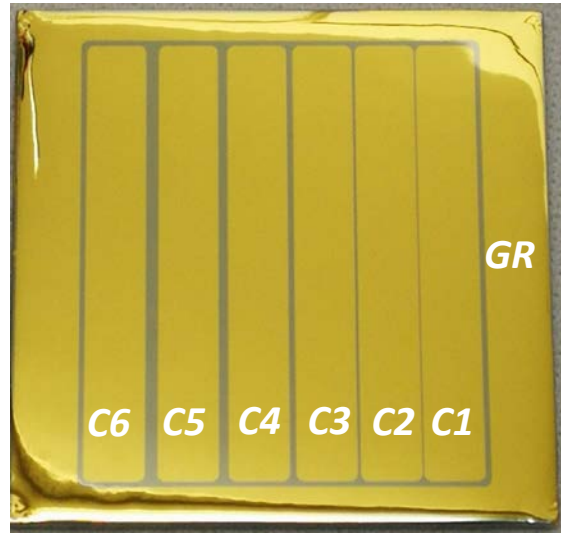
Annealing at 105°C



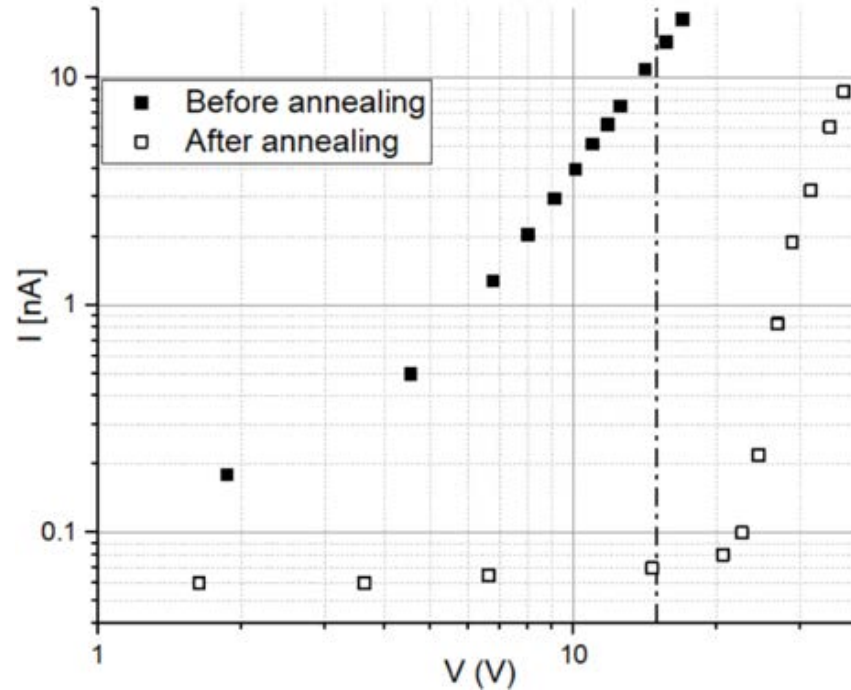
S. Bertoldo et al., *Eur. Phys. J. A* (2021) 57:177

# Thin planar HPGe detectors

Sb/p-HPGE/Al, L=35mm,  
t=2mm



Minimum gap tested 0.1mm



$^{133}\text{Ba}$ , 20V

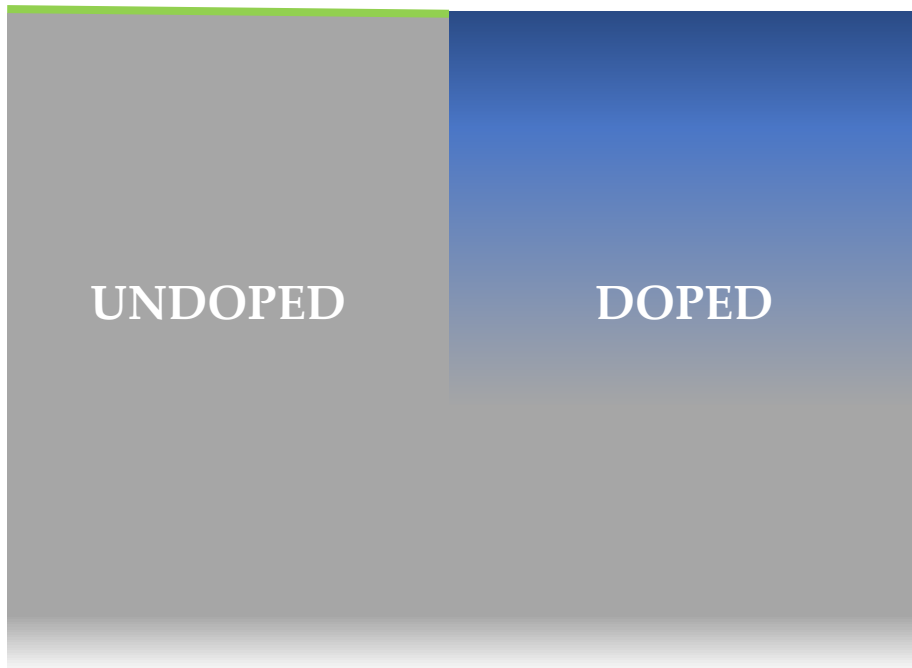
E=80keV  
FWHM=0.89keV

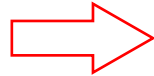
E=356keV  
FWHM=1.19keV

Before annealing  
After annealing

W. Raniero et al., *II NUOVO CIMENTO* 44 C (2021) 154

# PLM contact/junction: 2° type Segmentation



PLM  Partial area

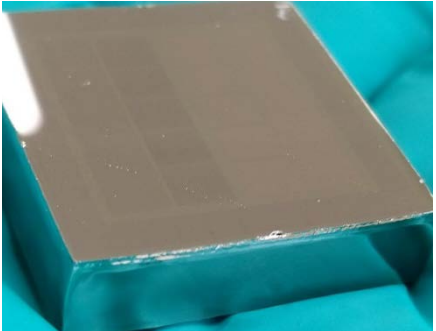
- Lithographic process using selective etching solutions:**
- Hot pure  $H_2SO_4$  for Sb deposition (preserve Sb junction)
  - $H_2O_2$  for GeP deposition (slowly etches everywhere)
  - Kern solutions ( $H_2O_2$ ,  $H_3PO_4$ , Ethanol) for Al-Ge deposition (preserve Al junction)



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



**Chemical passivation**  
Passivation of undoped surfaces with suitable solutions.





# Thick planar HPGe detectors

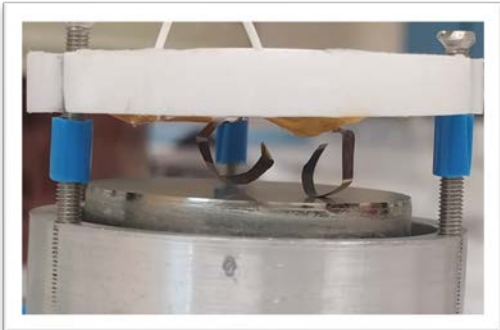


Sb/p-HPGE/Al,  
L=35mm, t=10mm

n+ junction  
(*spring contact*)



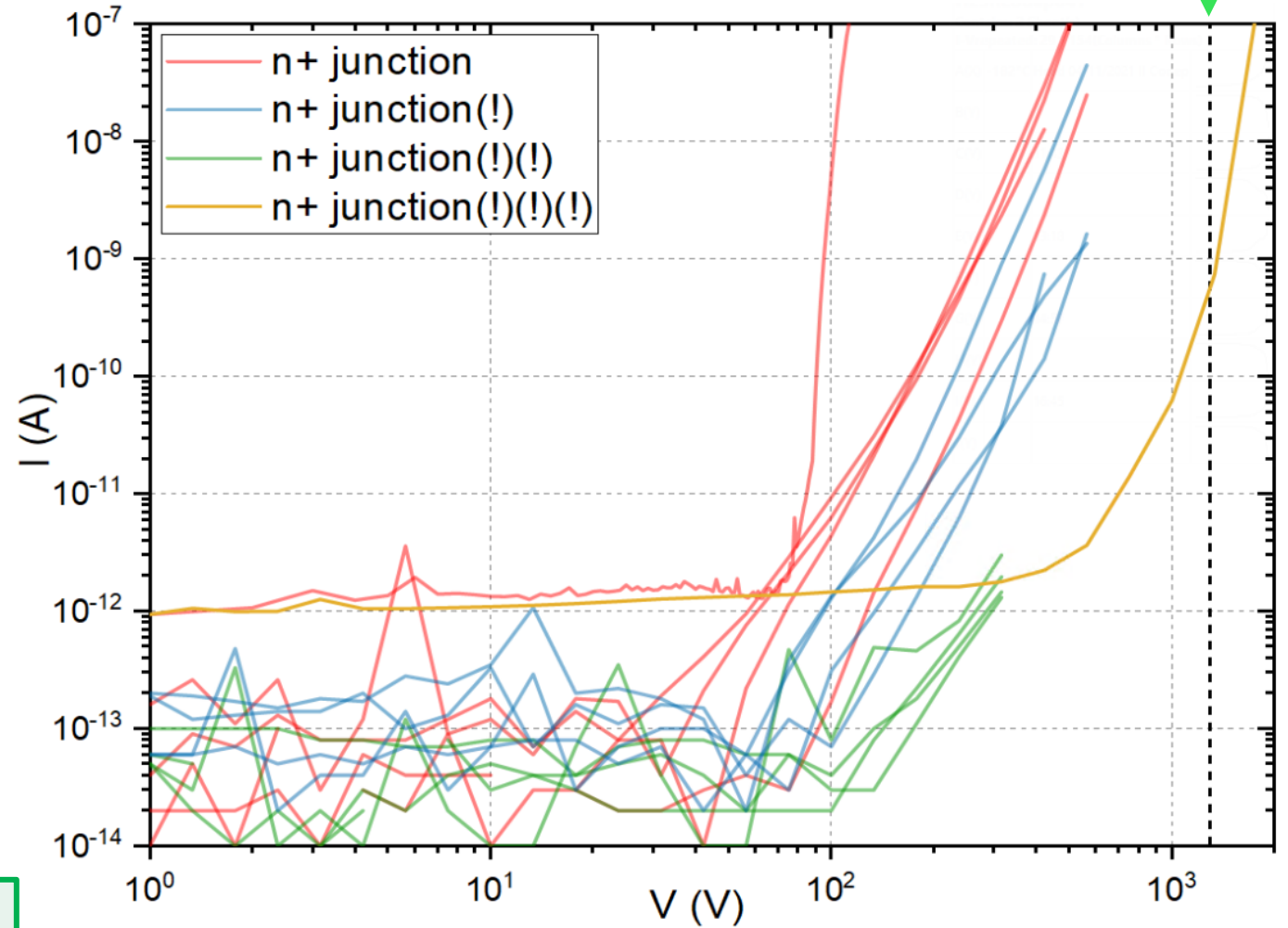
n+ junction (!) / (!)(!  
(*indium pad*)



n+ junction (!)(!)(!  
(*elastic tabs*)

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

$V_{\text{depl.}} : 1300\text{V}$



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# Coaxial Photolithography: Robot 3D

The laser micrometer measures the surface after a rotation of the coaxial detector while keeping the robot in the same position

## Coaxial Dummy



Misalignment of the segmentation lines at the top of the sample

The error is non-reproducible and is caused by the gripping system of the coaxial detector and the hole in the crystal itself

3D mapping of the coaxial detector and obtaining its coordinates relative to the robot's coordinate system with an accuracy of less than 0.1 mm

PEEK



VITON o-ring

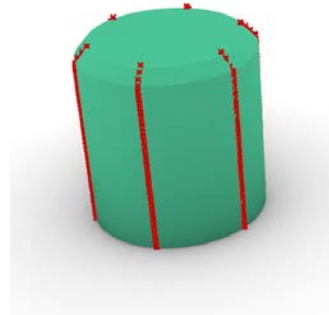


# Coaxial Photolithography: Robot 3D

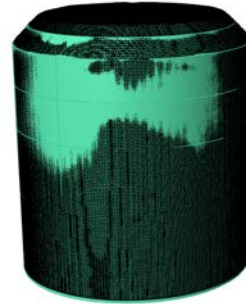
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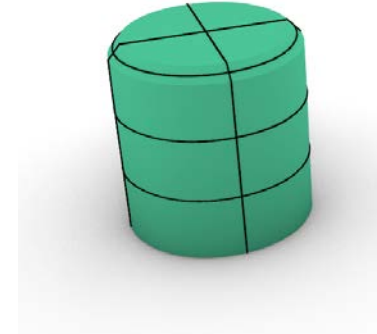
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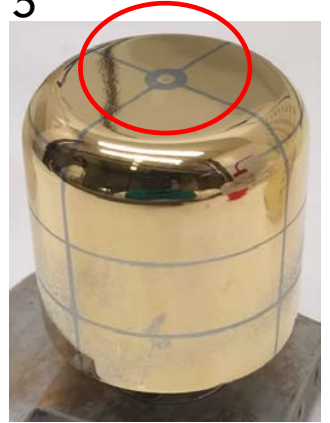
3



4



5



1. Mapping of the cylinder through vertical lines: line formed by a series of points  
Each point is determined by the robot's position + laser micrometer measures
2. 3D reconstruction via lofting technique.
3. Comparison with a professional 3D scanner , Accuracy  $<0.1$  mm.
4. Construction of the pattern to be lithographed in the robot's coordinate system
5. Photolithography carried out by the robot

UV photolithography robot

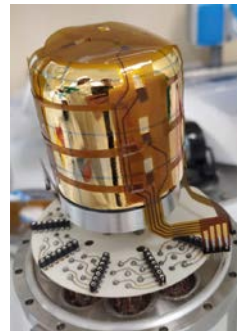
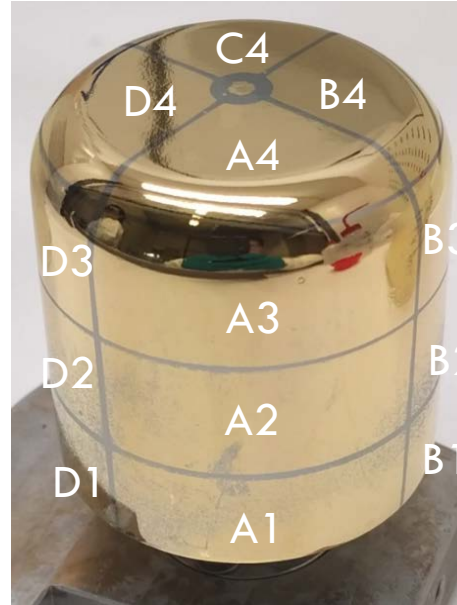


# Segmentation Test of Coaxial detector

T= 25°C

$\Omega$	A1		B1		C1		D1
A2	17.8	B2	16.6	C2	16.4	D2	22.1
A3	23.6	B3	21.3	C3	21.5	D3	27.3
A4	26.8	B4	23.8	C4	23.6	D4	30.5

$\Omega$	A1		A2		A3		A4
B1	22.5	B2	18.0	B3	17.7	B4	18.0
C1	27.0	C2	19.8	C3	19.4	C4	20.1
D1	21.7	D2	16.3	D3	18.1	D4	17.4



T= 80°K

$G\Omega$	C1	C3	D2	D4
Up	0.4	20	1.4	/
Down	/	0.6	7.5	3.0
Right	6.0	7.7	5.3	31.3
Left	4.3	25.0	0.1	12.8

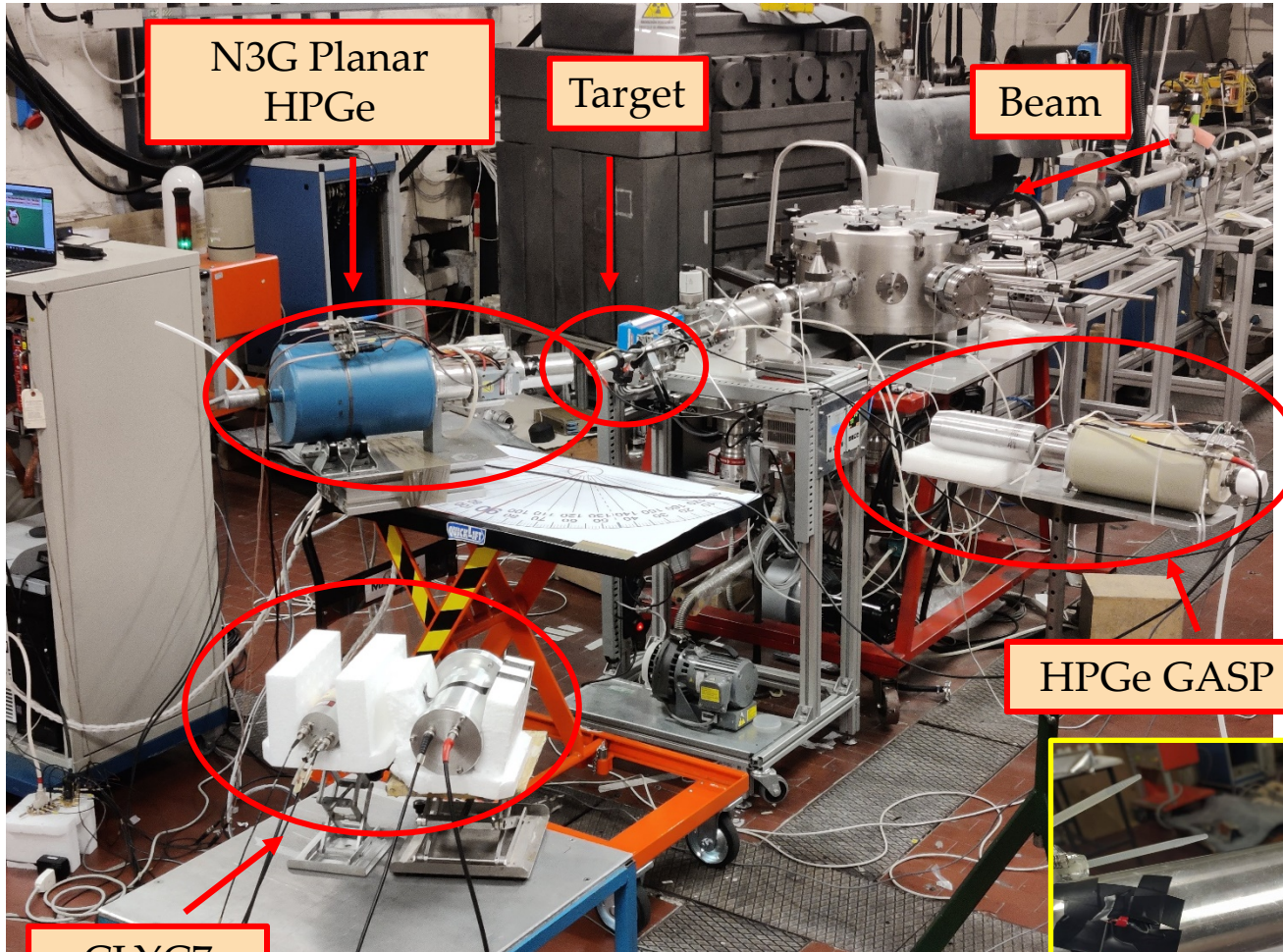
$G\Omega$	A1	B2	B4
Up	8.9	21.7	/
Down	/	3.1	27.8
Right	62.5	0.2	5.0
Left	62.5	11.4	12.5

High resistance between the segments is measured, exceeding **100 M $\Omega$**

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# Neutrons damage on planar PLM segmented detector



380nA 4MeV proton beam  
 $^7\text{Li}$  target, 100 $\mu\text{m}$

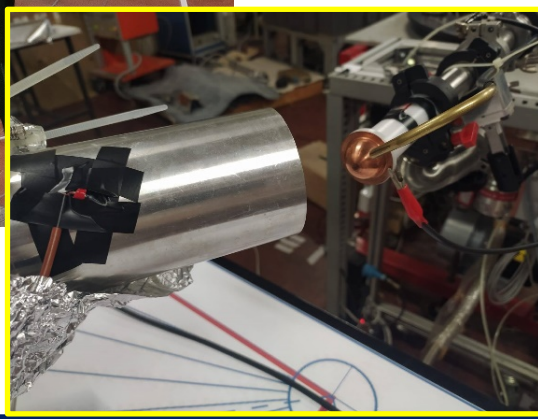
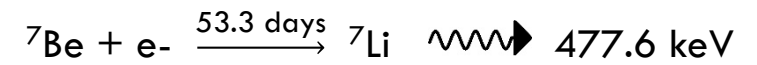
Reaction:  $^7\text{Li} (p,n) ^7\text{Be}$

Prototype detector is located at 30° 9.5 cm

Neutrons are directly measured with

- CLYC7 scintillators, 30° 2 m

- GASP HPGe  $\gamma$  detector, 90° 1 m



R. Escudeiro et al. "Neutron radiation damage on a planar segmented germanium detector" proceeding Presented at the XXXVII Mazurian Lakes Conference on Physics, Piaski, Poland, September 3-9, 2023

# Neutrons damage on planar PLM segmented detector: before run

## Detector prototype:

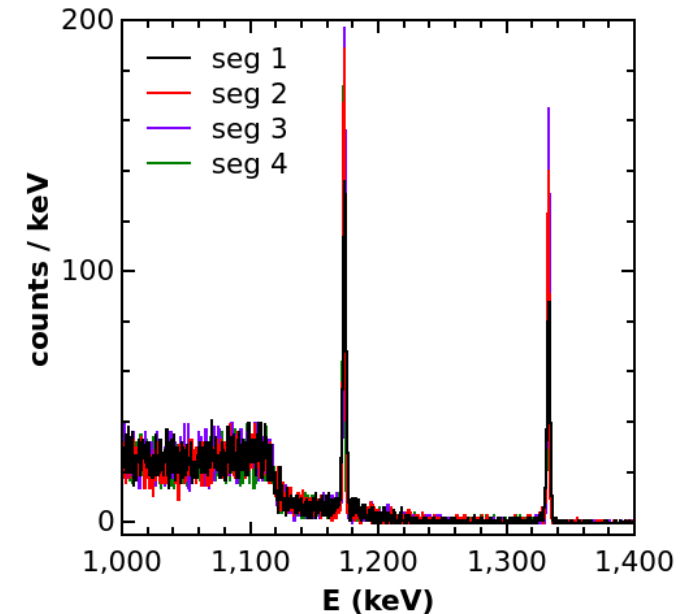
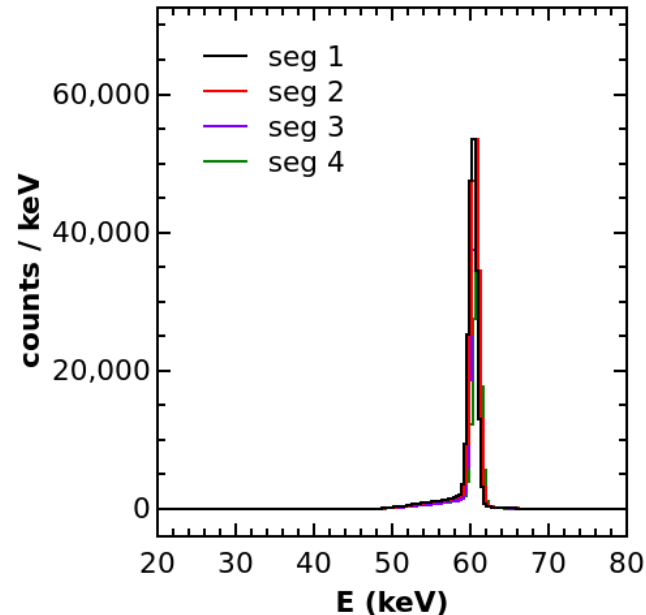
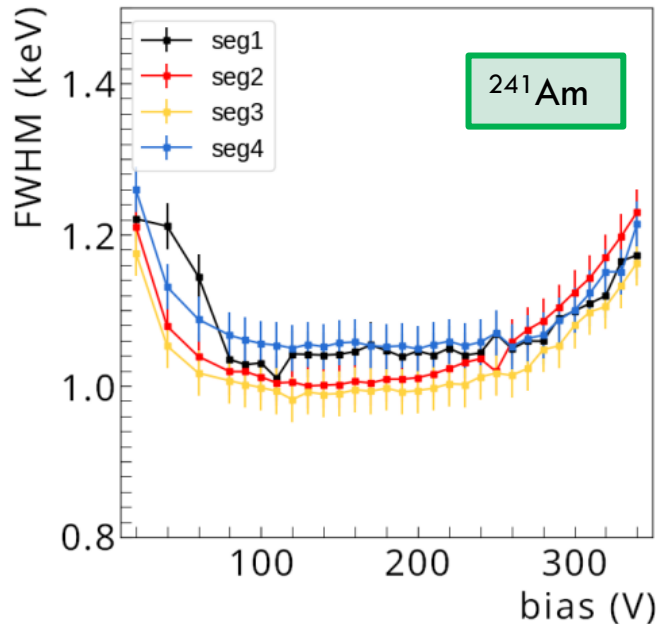
Sb/p-HPGE/Al, L= 32mm, t= 8.6mm  
4 contacts + guard ring

**Starting resolution** at 80V operational bias  
before the neutron damage run

## Energy resolution in the lab

$^{241}\text{Am}$   
E = 59,5 keV  
FWHM < 1,1 keV

$^{60}\text{Co}$   
E = 1332 keV  
FWHM < 1,8 keV



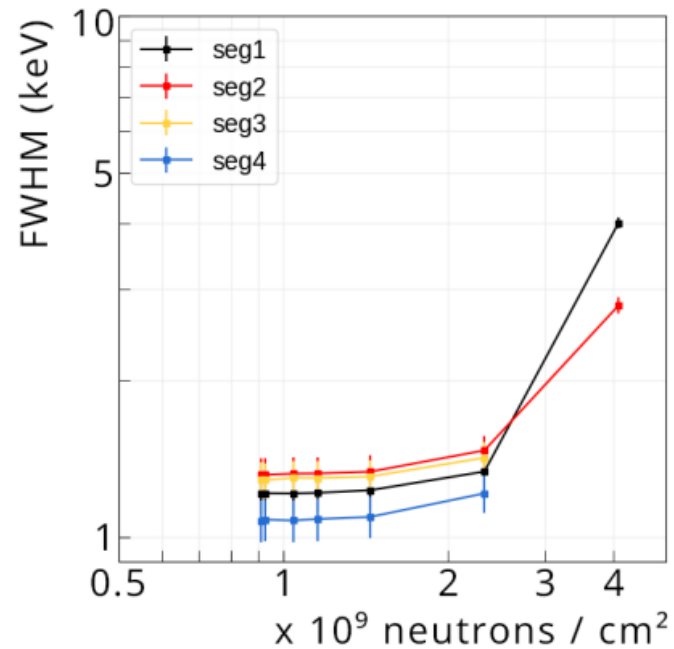


# Neutrons damage on planar PLM segmented detector: after 1° run

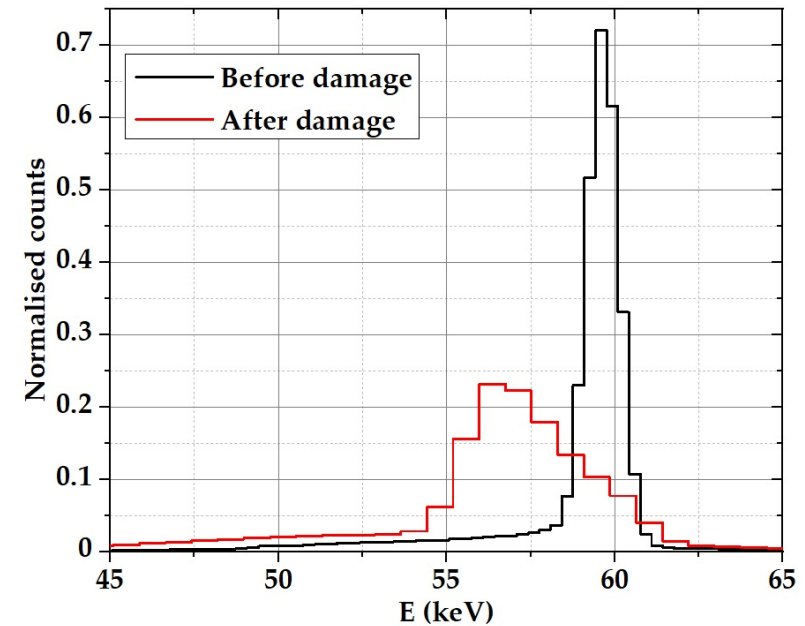
Operational Voltage 80V

Neutron irradiation for increasing time intervals alternated to 5 min runs with  $^{241}\text{Am}$  and  $^{60}\text{Co}$  leads to increasing resolution worsening

After 4 hours of irradiation time,  $\approx 4 \cdot 10^9$  neutrons/cm<sup>2</sup>, detector is no longer operable



$^{241}\text{Am}$   
E = 59,5 keV  
FWHM = 3,2 – 4,2 keV

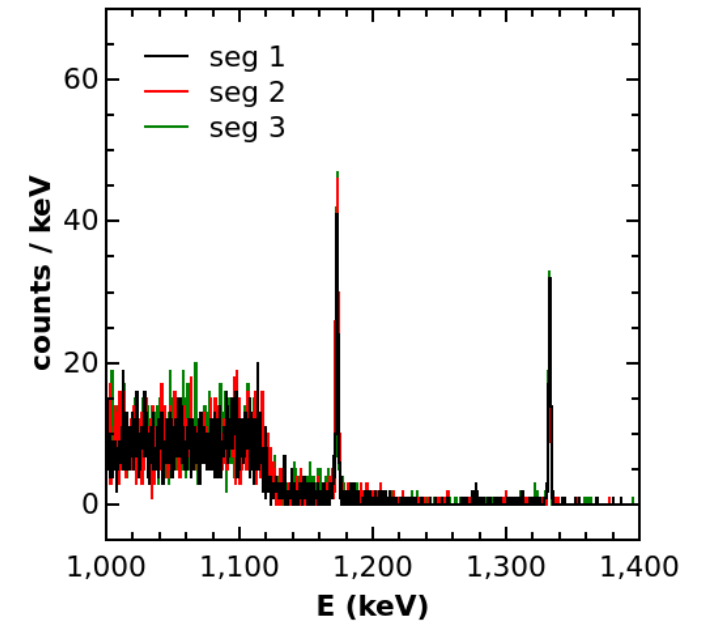
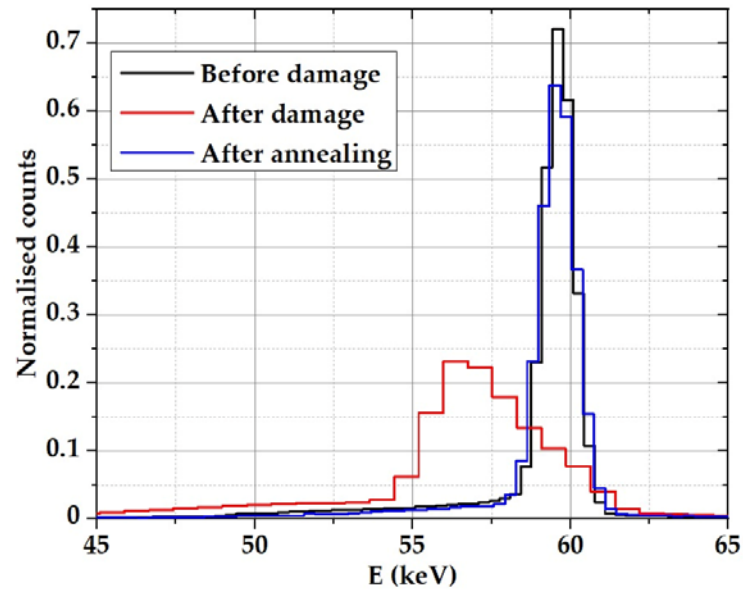
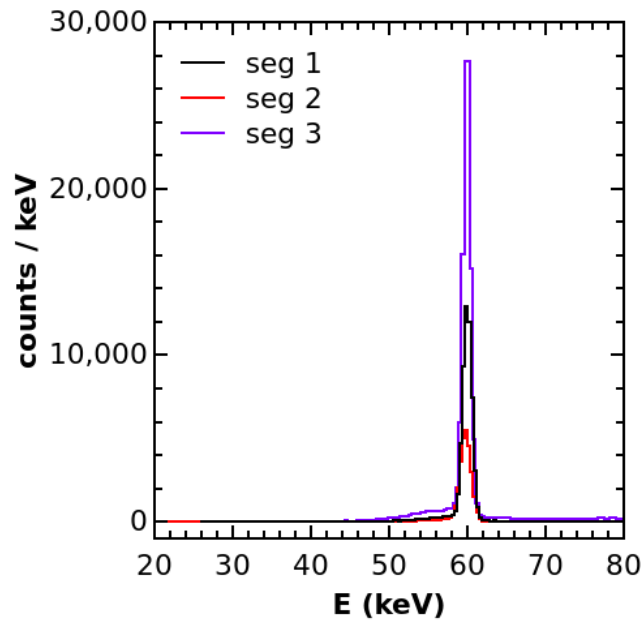


# Neutrons damage on planar PLM segmented detector: After Recovery

Annealing procedure: 7 days at 105°C continuously pumped inside the cryostat

$^{241}\text{Am}$   
E = 59,5 keV  
FWHM < 1,6 keV

$^{60}\text{Co}$   
E = 1332 keV  
FWHM < 2 keV



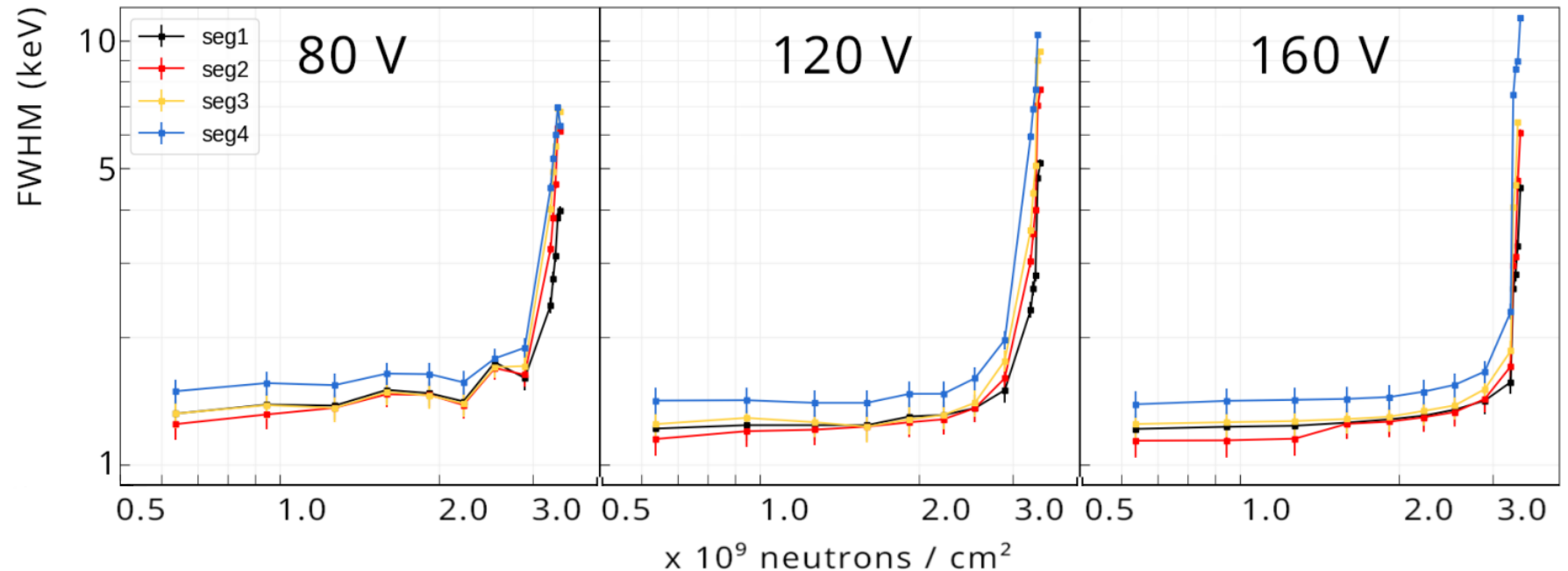
# Neutrons damage on planar PLM segmented detector: After 2° run

Operational Voltage 80-120-160V

Neutron irradiation for 20 and 2 min to 5 min runs with  $^{241}\text{Am}$  to better characterize resolution worsening

Drastic drop in resolution after  $\approx 3 \cdot 10^9$  neutrons/cm<sup>2</sup> irradiation fluence

$^{241}\text{Am}$   
E = 59,5 keV  
FWHM = < 2 keV until threshold



S. Bertoldo PhD thesis: Developments on new detector technologies for high resolution gamma spectroscopy

# SUMMARY

- PLM technology can be apply to HPGe crystal (**hyperpurity preserve**)
- PLM junction is thin, segmentable and termally stable (**annealing**)
- PLM and segmentation technology can be applied to both planar and coxial detectors  
(**2D to 3D PLM shape LNL-INFN has a collaboration agreement with MIRION**)
- PLM segmented detector recovers after Neutron damage with a very good energy resolution  
(**annealing recovery**)

# R&D Gamma ray detector Team

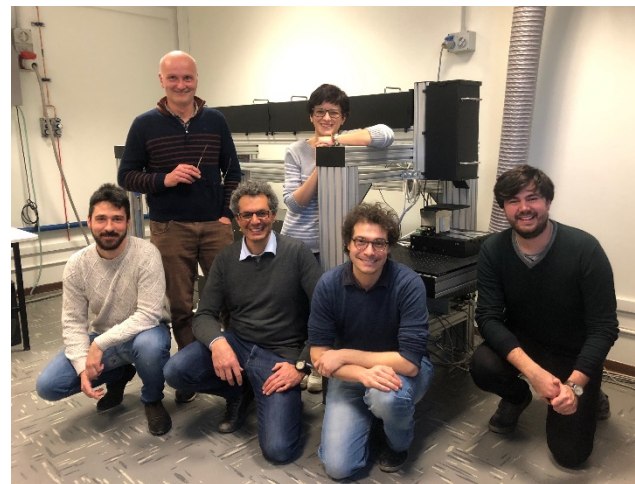
Davide De Salvador  
Stefano Bertoldo  
Enrico Napolitani  
Francesco Sgarbossa

Sara Carturan  
Gianluigi Maggioni  
Francesco Recchia  
Dino Bazzacco

Walter Raniero  
Daniel Napoli  
Chiara Carraro

Stefano Capra  
Giacomo Secci  
Alberto Pullia  
Bénédicte Million  
Luciano Manara

Andrea Mazzolari  
Lorenzo Malagutti  
Andres Gadea



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## HPGe crystal research at LNL-INFN

**Walter Raniero**

INFN – LNL

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