Passivation and segmentation developments at LNL

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PSeGe

ENSAR2
Work Package 10 - Joint Research Activity 2
Research & Development on
Position-Sensitive Germanium Detectors
CSIC – CNRS – GSI – IKP – INFN – Univ. of Liverpool

ENSAR2

HORIZON 2020 projects

Work Package n. 10 - JRA2 PSeGe

R&D on Position-Sensitive Germanium Detectors for Nuclear Structure and Applications

Task 1: **New technologies on passivation and segmentation of HPGe**
(Coordination: INFN)

Task 2: **R&D on novel Ge-detector geometries for ultimate position resolution and efficiency**
(Coordination: GSI)

Task 3: **R&D on segmented p-type coaxial detectors**
(Coordination: CSIC)

Task 4: **Network activity: Demonstration of imaging applications and associated detector technologies**
(Coordination: Univ. of Liverpool)

Coordinators: A.Gadea (CSIC,**JRA2 leader**), D.R.Napoli (INFN, **deputy**), P.Reiter (IKP, **deputy**), J.Gerl (GSI), A.Boston (U.of Liverpool)

Task 1: The aim is to find new passivation methods for solving the instabilities of the intrinsic Ge surfaces in between contacts and segments, which lead to an increase of the leakage current and a reduction of both energy and position resolution.
The interdisciplinary group of the Materials Laboratory of the LNL is studying chemical and physical methods of passivation in HPGe.

Task 3: In position-sensitive detectors for tracking arrays, the most important signals are the ones from the segments. Thus, a development focused on the possibility to use p-type HPGe detectors will greatly benefit present and future detector arrays.
This task proposes a R&D on alternative materials for n- and p-type contacts that could be segmented.
The interdisciplinary group of INFN-LNL is also participating to this task.

Main topics of our current activity at LNL

- To study the effects of the passivation of the intrinsic surface on the HPGe detector characteristics (depletion voltage, resolution, efficiency, dead layer, …)
- To develop new passivation/protection techniques
- To develop new thinner contacts, which could replace the present ones (especially Li)
- To improve the method of construction and the robustness of the surfaces for costs reduction in both fabrication and maintenance (needed also for applications).

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Multidisciplinary Team

INFN-LNL	D.R. Napoli, W. Raniero
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INFN-PG and University of Perugia	S. Riccetto
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IKP Cologne	J. Eberth
CNR-IMM Bologna	R. Nipoti, F. Mancarella, M. Bellettato

High Purity Ge detectos production

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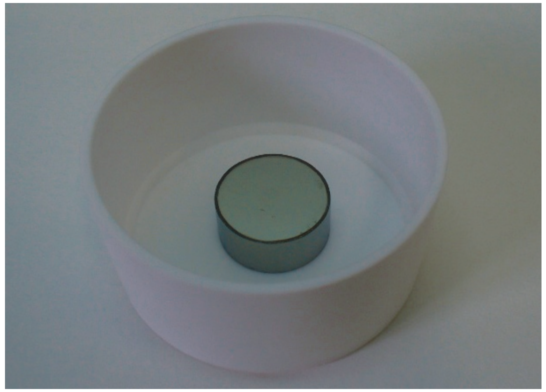
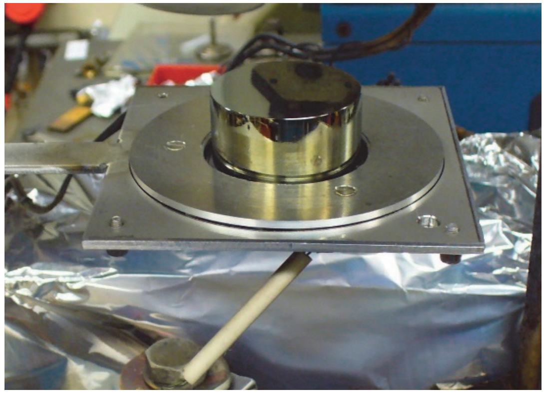
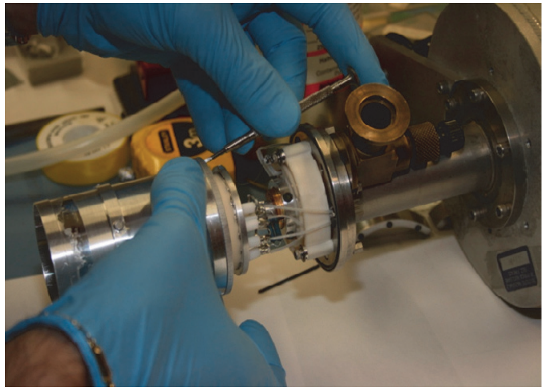
R&D on HPGe production technologies

First step:
we acquired the basic know-how for the **in-house** production of commercial-like planar HPGe detectors.



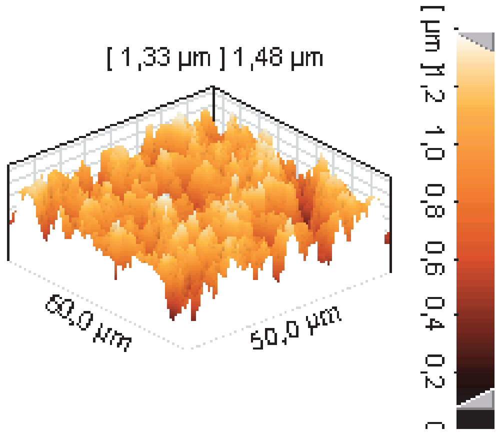


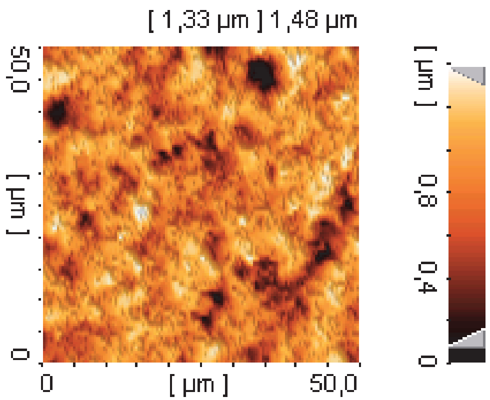


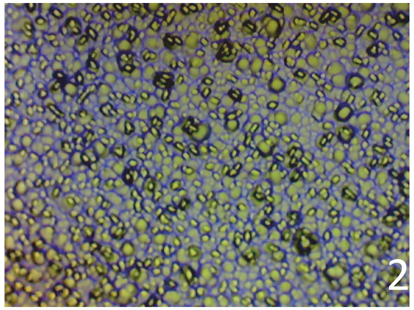
- Mechanical treatment and chemical preparation of the surfaces
- n^+ -contact made with Li diffusion doped method (600 - 900 µm thick)
- p^+ -contact with B implantation (about 0.3 µ thick) + Au film deposition...
- Surface passivation & mounting

Mechanical treatment of the HPGe surfaces (lapping)

AFM



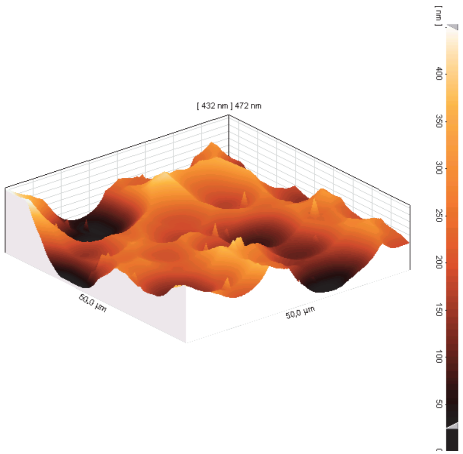


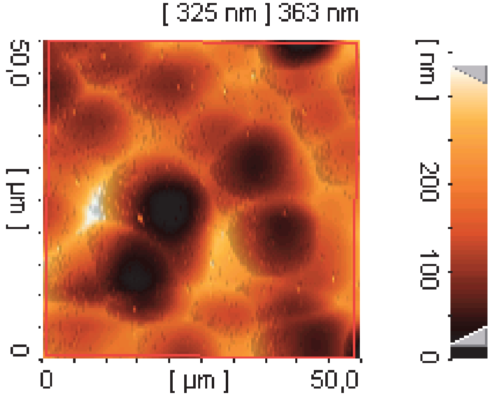


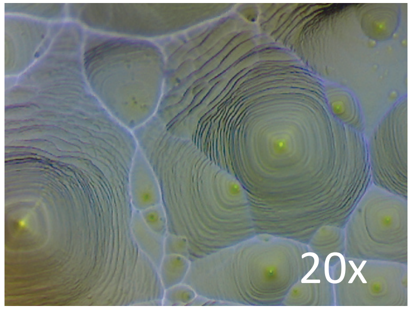
Count of the crystal dislocations

Roughness after lapping: 150 - 250 nm

Chemical preparation of the HPGe surfaces (etching)

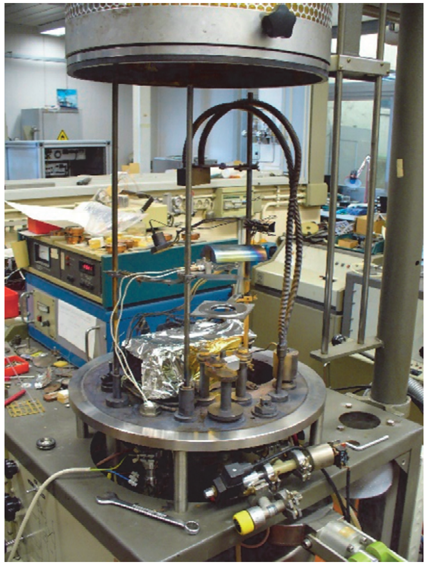




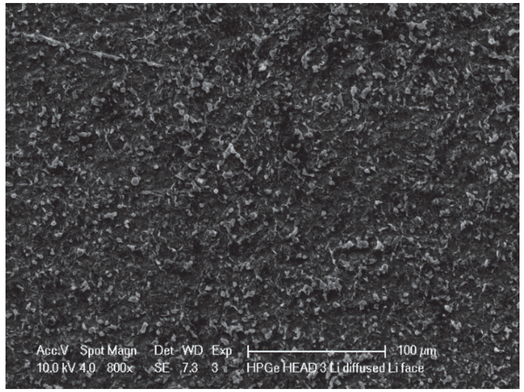


Roughness after etching: 50 - 100 nm

n+ -contact made with Li diffusion method



Evaporation apparatus

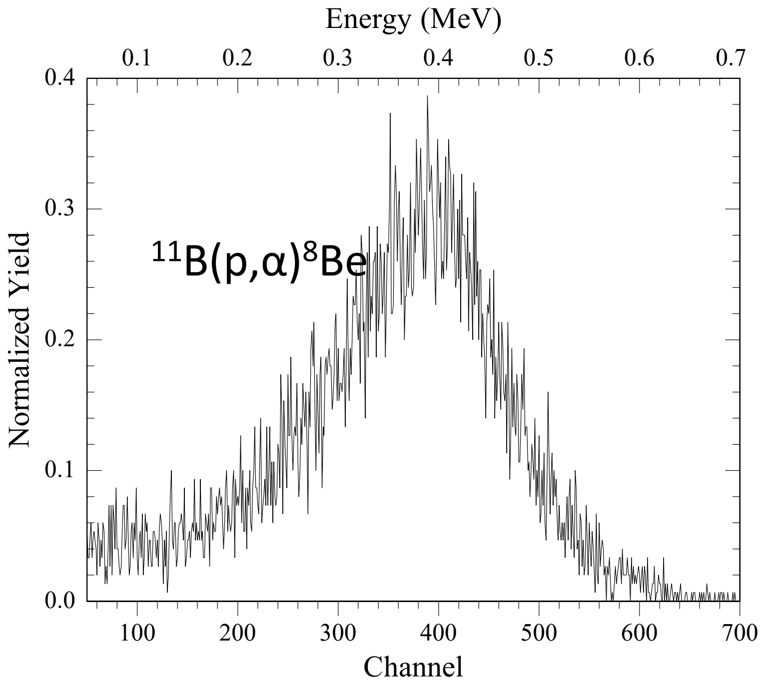


Li-coated surface

p+ -contact made with B implantation

Ion implantation apparatus

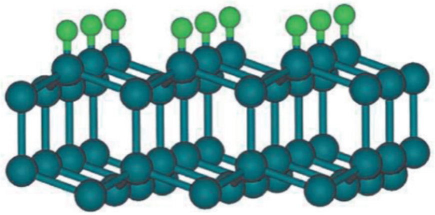
NRA technique

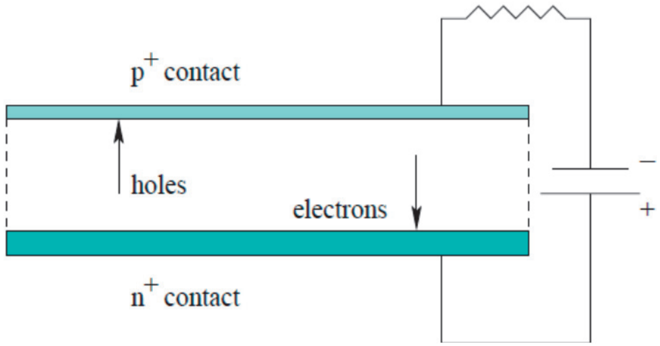


PASSIVATION

Passivation of the intrinsic surface

Ge





Ideal Passivation

<u>electrical</u>: low leakage current

<u>chemical</u>: prevents the surface from reacting with the atmosphere

Ge is not like Si: natural Ge oxides are not stable

Some known passivations: SiO_2 or amorphous Ge(H)

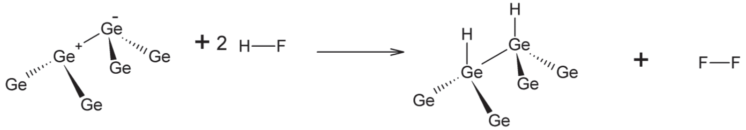
Passivation techniques

- **Hydride termination - three types:**

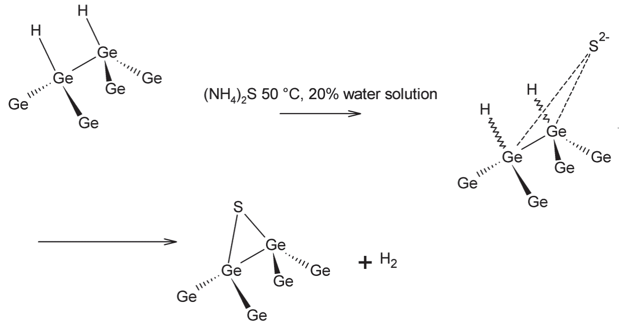
Low hydride (low concentration HF)

High hydride (high concentration HF)

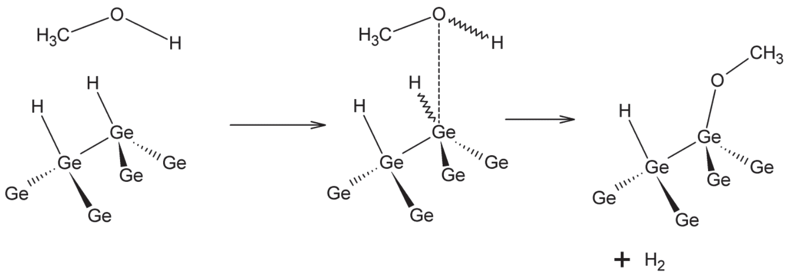
Hyper hydride (same like High hydride but with after a different surface preparation)



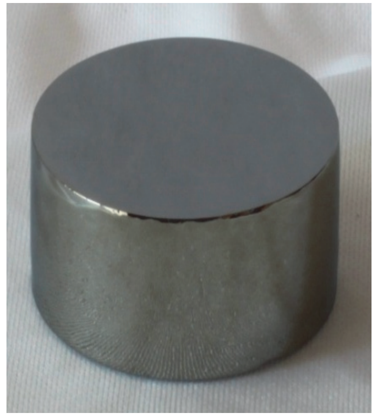
- **Sulfide termination** ($(NH_4)_2S$)



- **Methanol Passivation** (3:1 etching followed by methanol quenching)



O-H bond dissociation

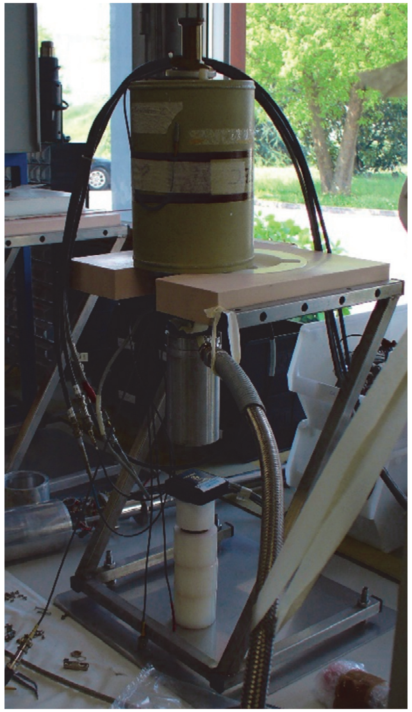


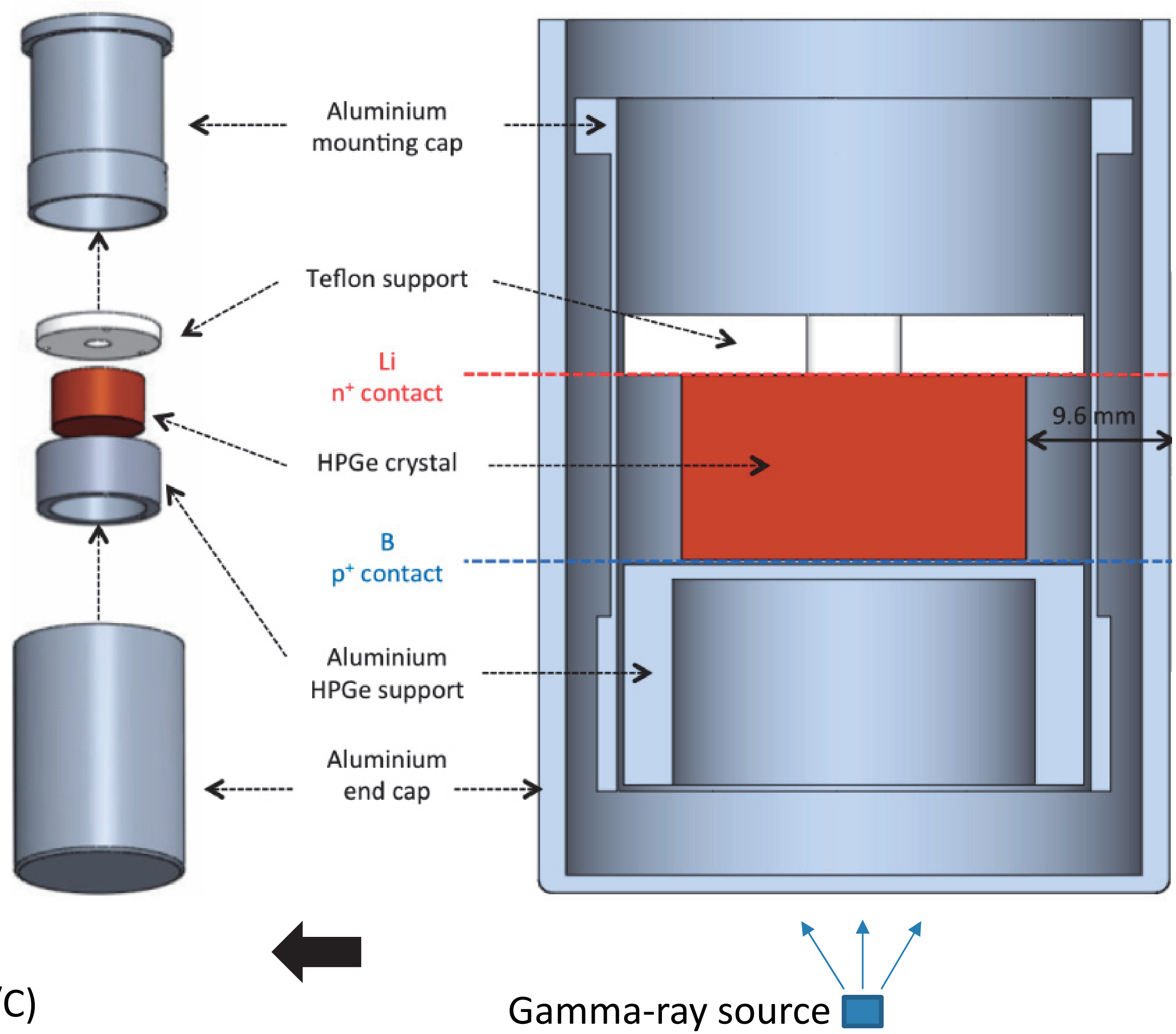
n-type HPGe crystal planar detector with B and Li contacts NO Guard Ring

ϕ 40 mm, h 20 mm

The **same detector** was used for **all** the different passivation treatments to avoid bulk effects of the crystal and highlight surface effects.

Measurement setup for determining the detector bulk properties



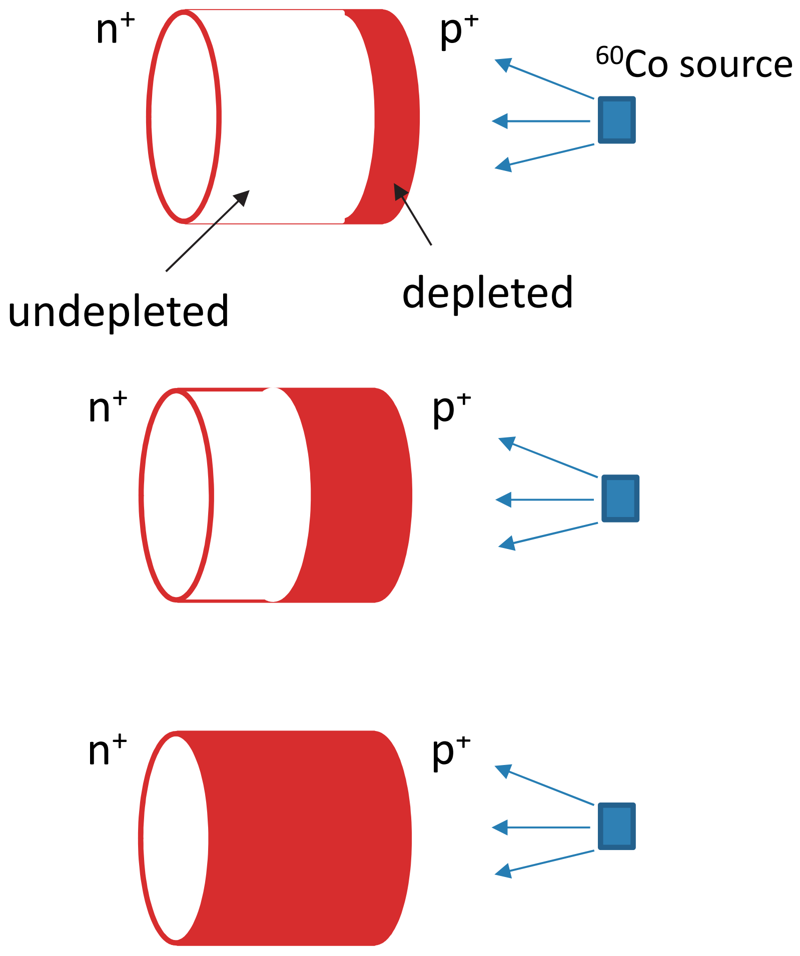


"Bulk" properties:

- Depletion voltage
- Efficiency
- Resolution
- Peak-to-Compton ratio (P/C)

Depletion voltage

How can we measure it?

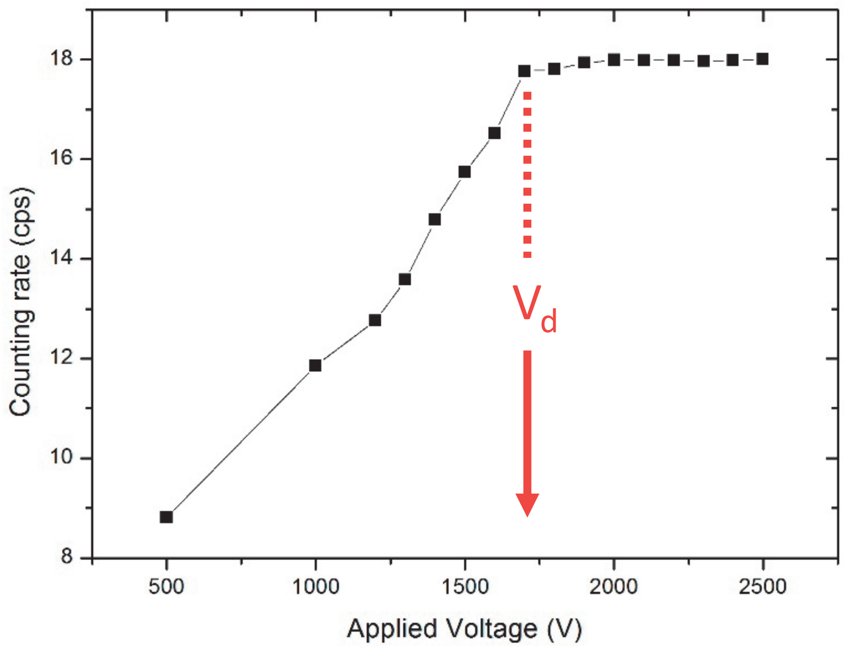


Depletion voltage: the voltage at which the depletion depth is equal to the detector thickness

If surface effects can be neglected

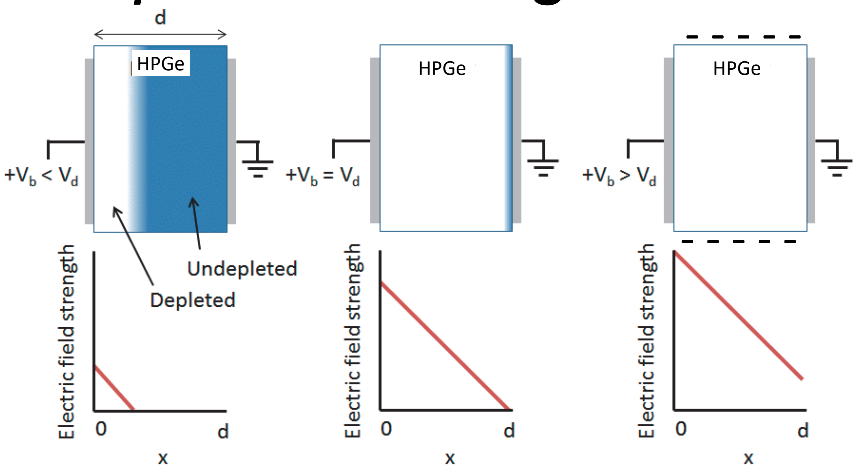
$$V_d = N\, q\, d^2 / 2\, \varepsilon$$

N = impurity density in the bulk
q = electronic charge
d = detector thickness
ε = dielectric constant of germanium



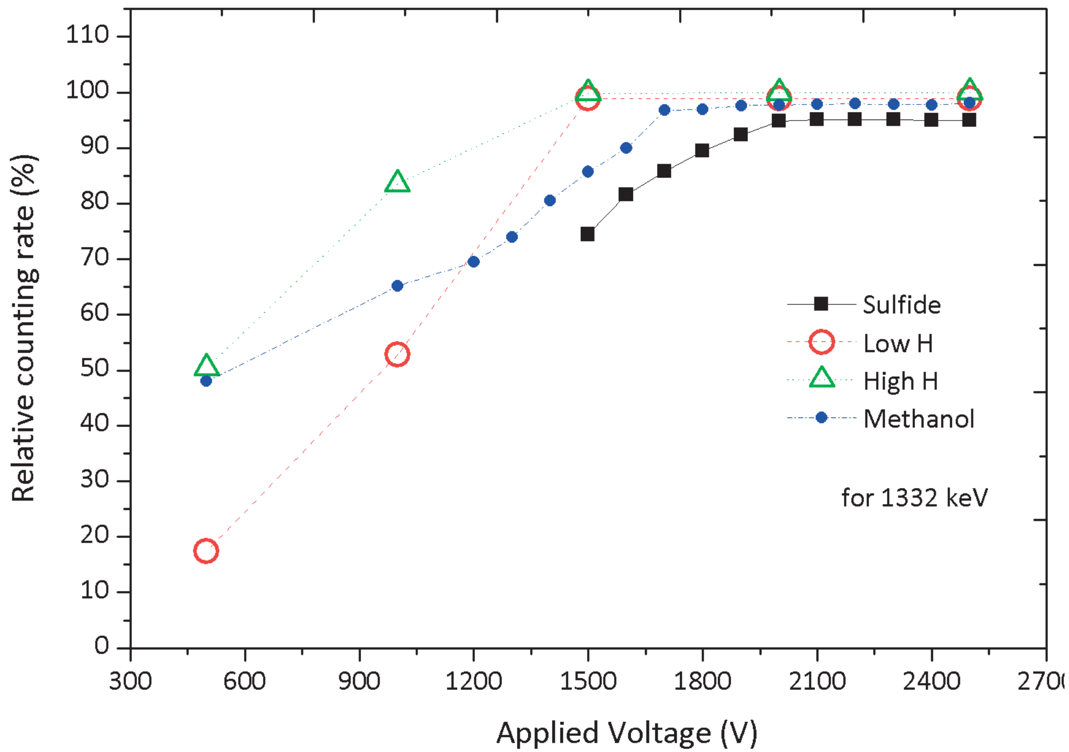
Increasing ΔV → Increases the counting rate till a plateau

Depletion voltage



Experimental results

(from G. Maggioni et al., EPJA 2015)



	Depletion Voltage (V)
Low H	≤ 1500
High H	≤ 1500
Methanol	1700
Sulfide	2000

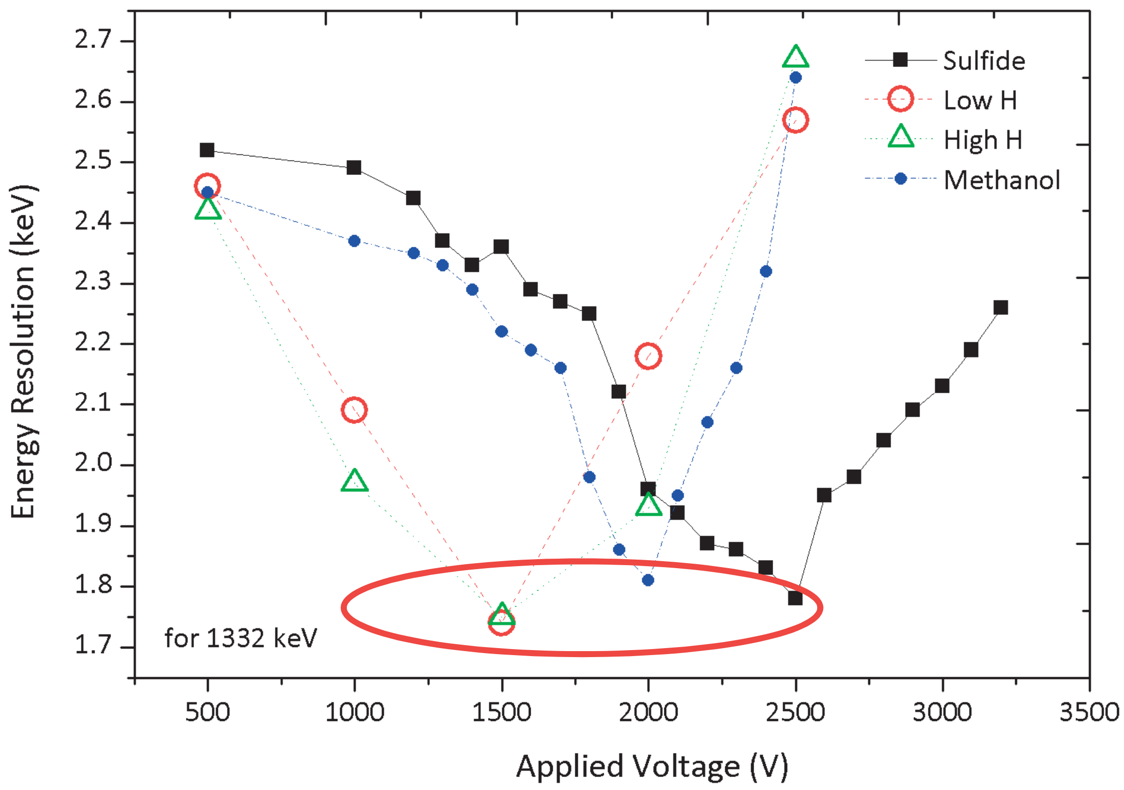
Surface effects are important → Passivation can give rise to a charge accumulation, which distorts the local field

Plateau value of counting rate for sulfide is lower → lower active volume (90-95%)

Detector resolution (FWHM) vs bias voltage

Detector exposed to a ^{60}Co source

Resolution ranges from 1.7 to 2.7 keV



	Voltage for best resolution (V)
Low H	1500
High H	1500
Methanol	2000
Sulfide	2500

The best resolution is comparable for all the passivations

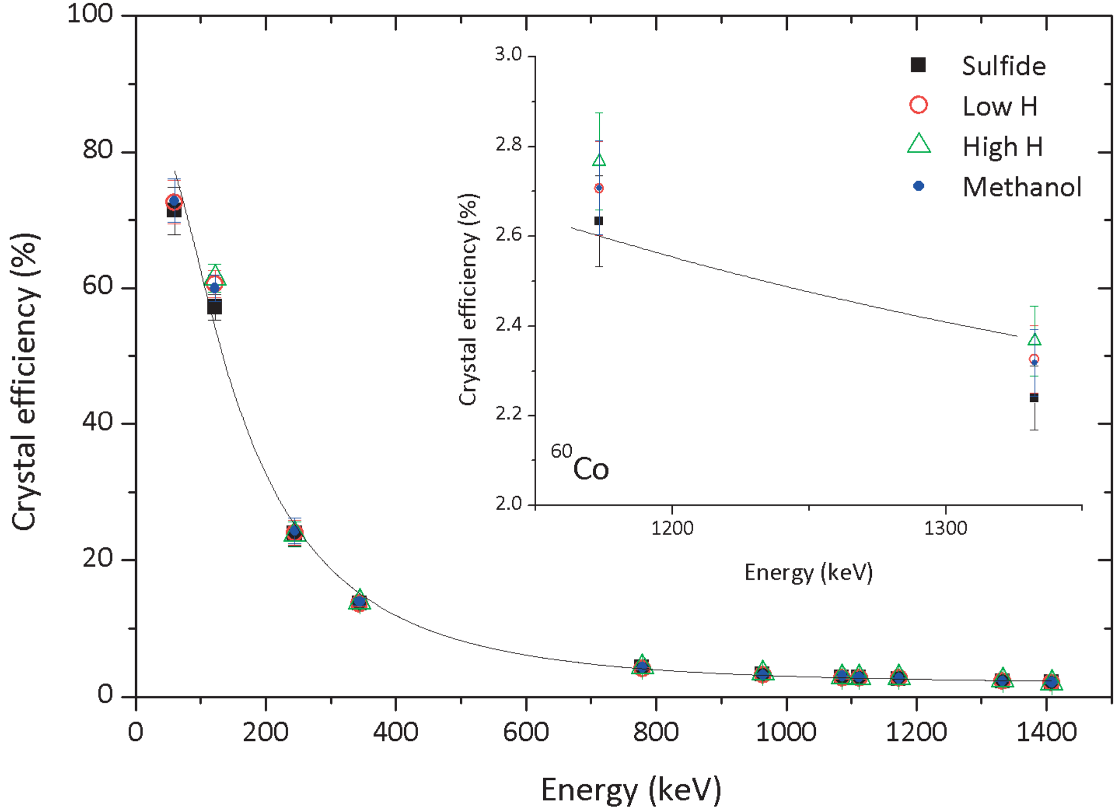
Detector efficiency

$$\epsilon_{exp} = \frac{R}{A \times I \times \exp(\sum_{i=1}^{3} -(\mu_i \times d_i) \times (\Omega/4\pi)}$$

R = counting rate
A = source activity
I = emission probability of the line of interest
d_i and μ_i = thickness and total absorption coefficient of the absorbing layers, respectively
Ω = solid angle subtended by the detector

(from A. Elanique et al., Appl. Rad. Isot. 2012)

^{60}Co, ^{152}Eu and ^{241}Am

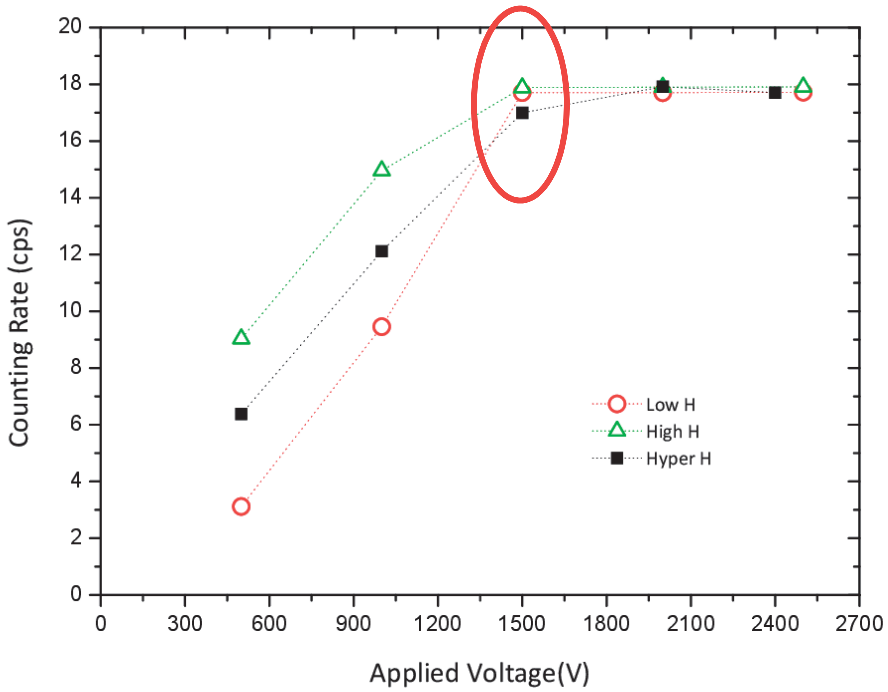


Surface passivation slightly affects the detector efficiency

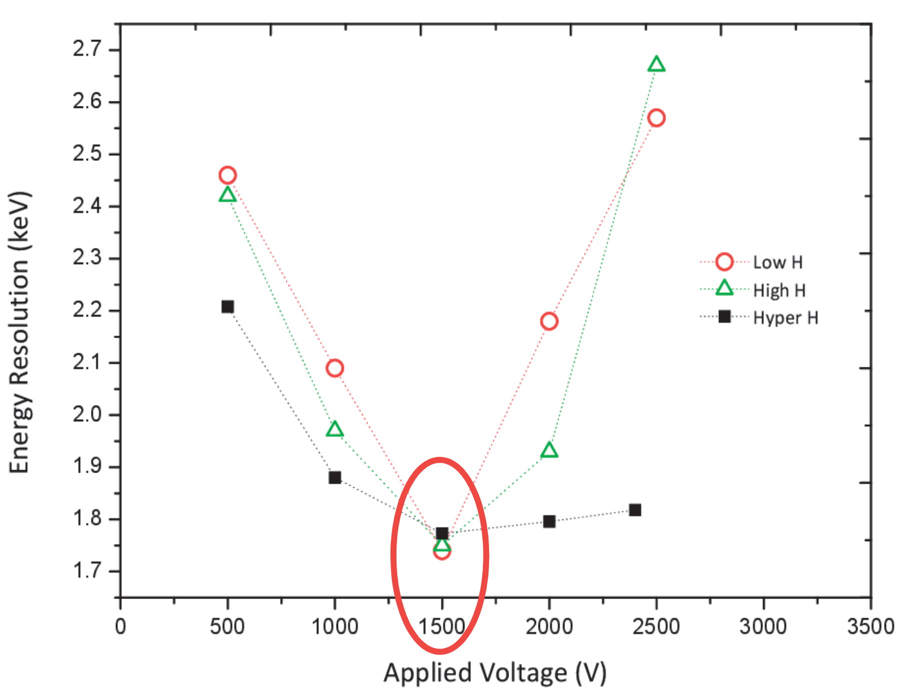
Detector properties and surface passivation: current results

Passivations: Low H, High H and Hyper H

Depletion Voltage



Energy Resolution



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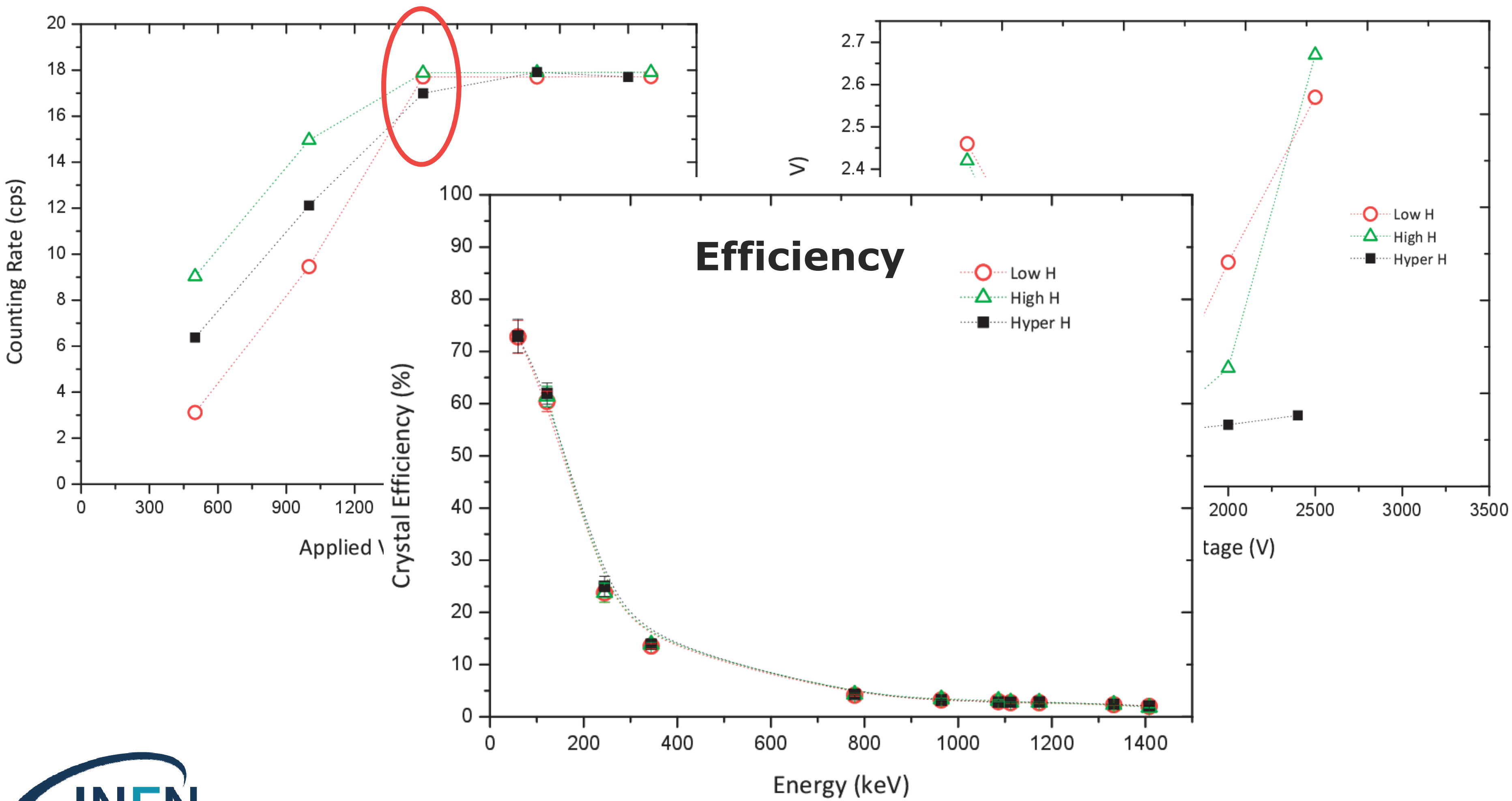
(from S. Riccetto et al., to be submitted to **EPJA**)

Passivations: Low H, High H and Hyper H

Depletion Voltage **Energy Resolution**



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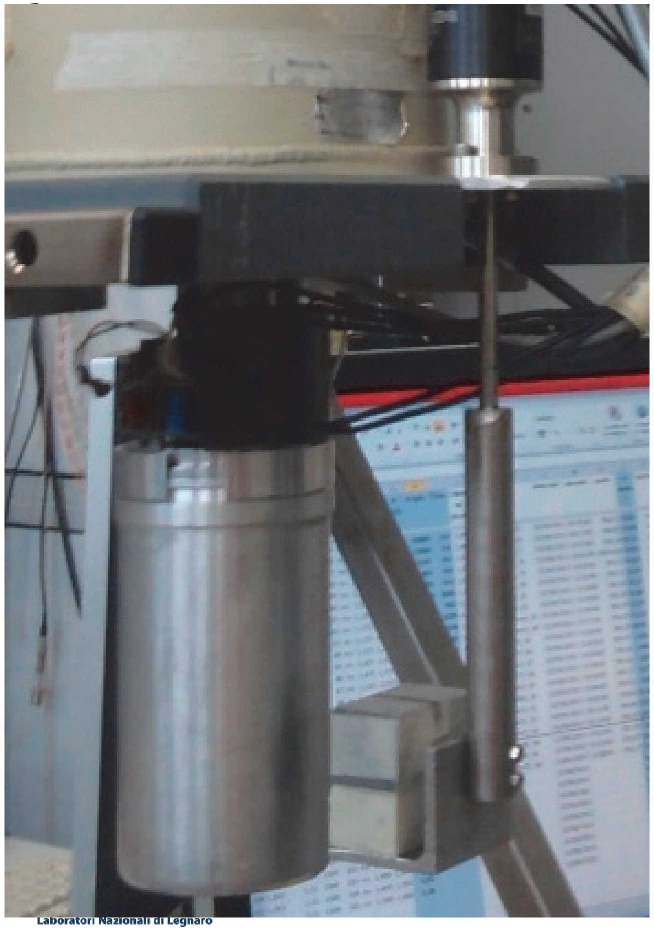
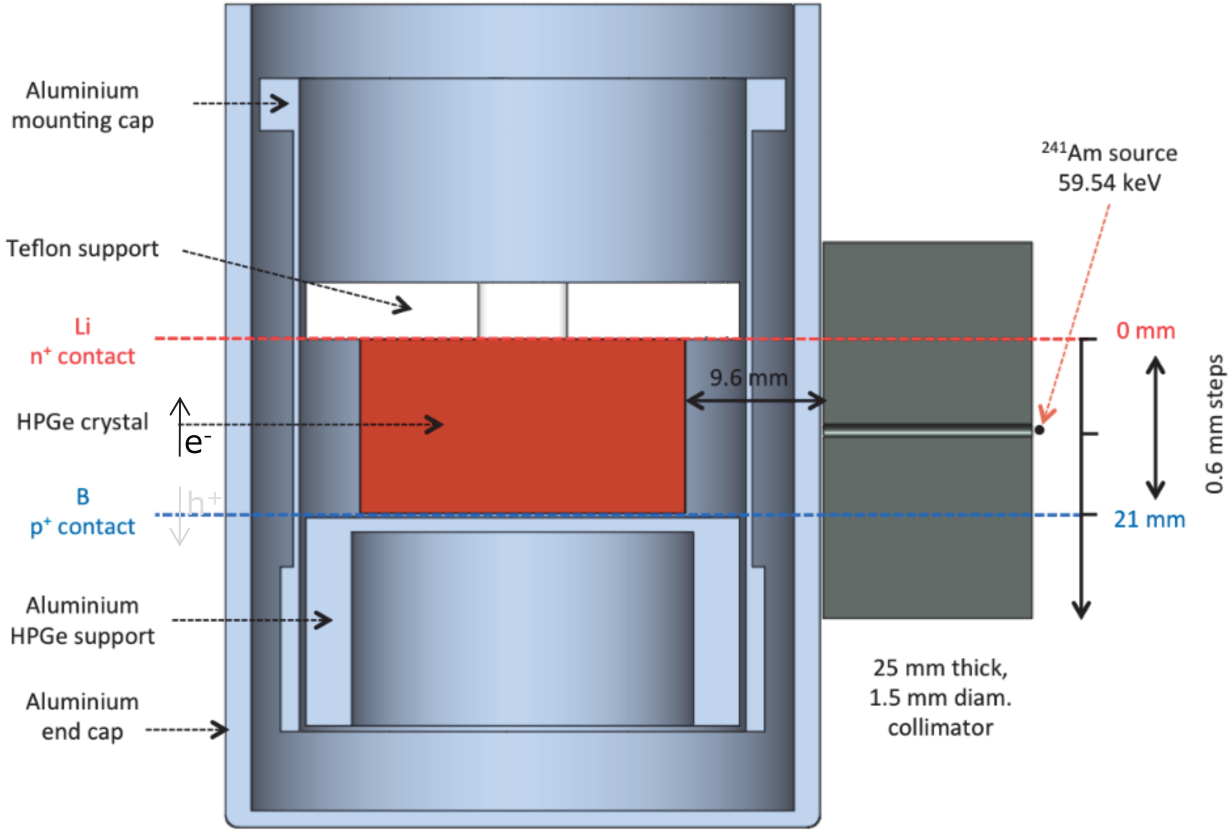
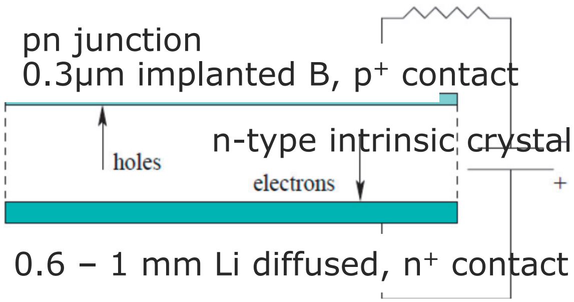
(from S. Riccetto et al., to be submitted to **EPJA**)

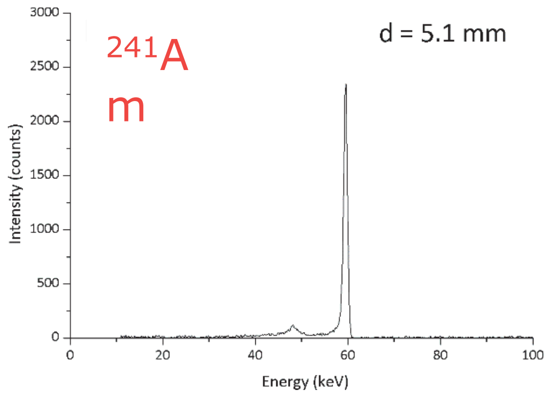
Lateral scan with 60eV ϒ-rays

Lateral scans with a Collimated ^{241}Am Source to study the passivated surface.

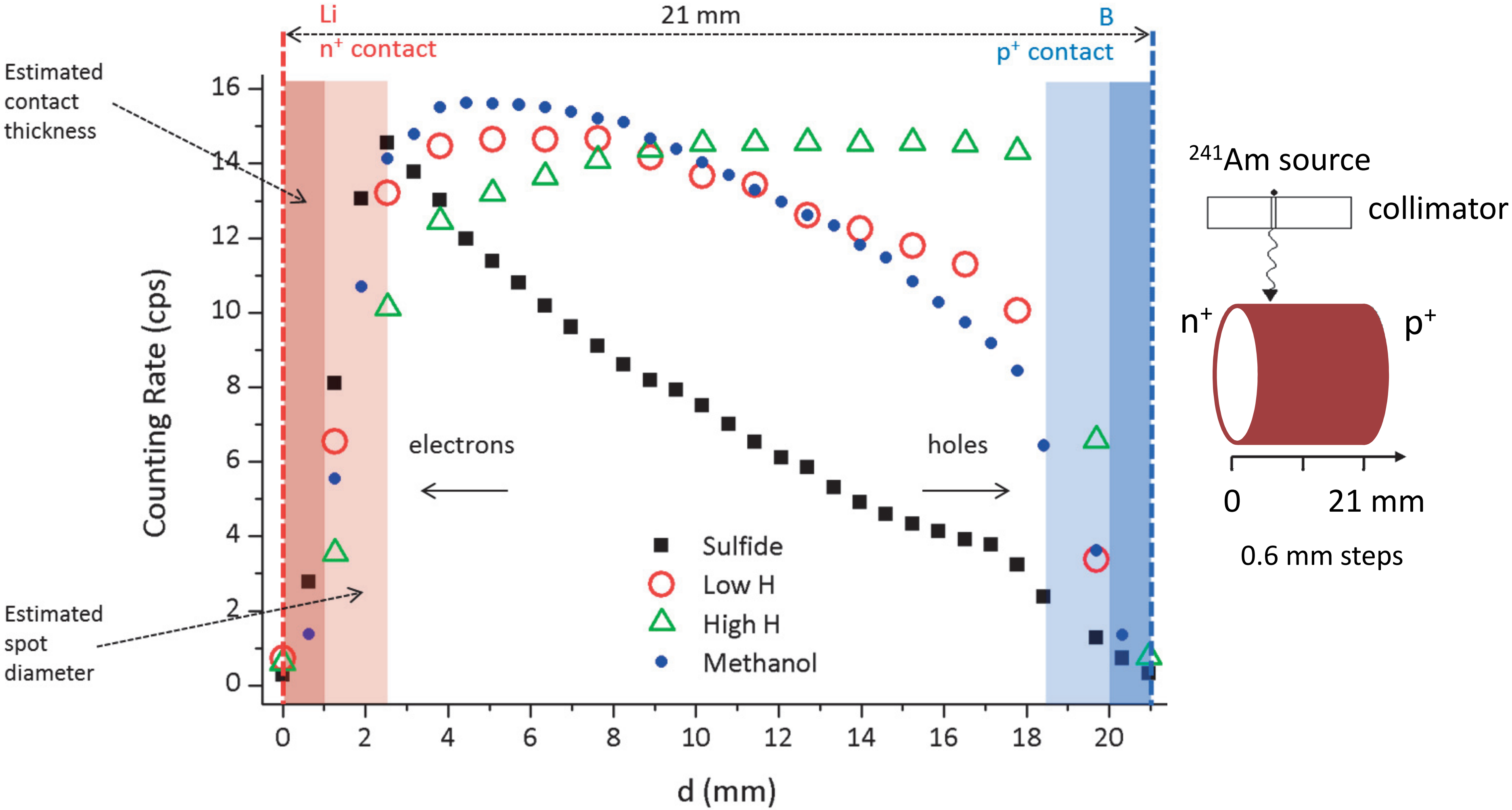






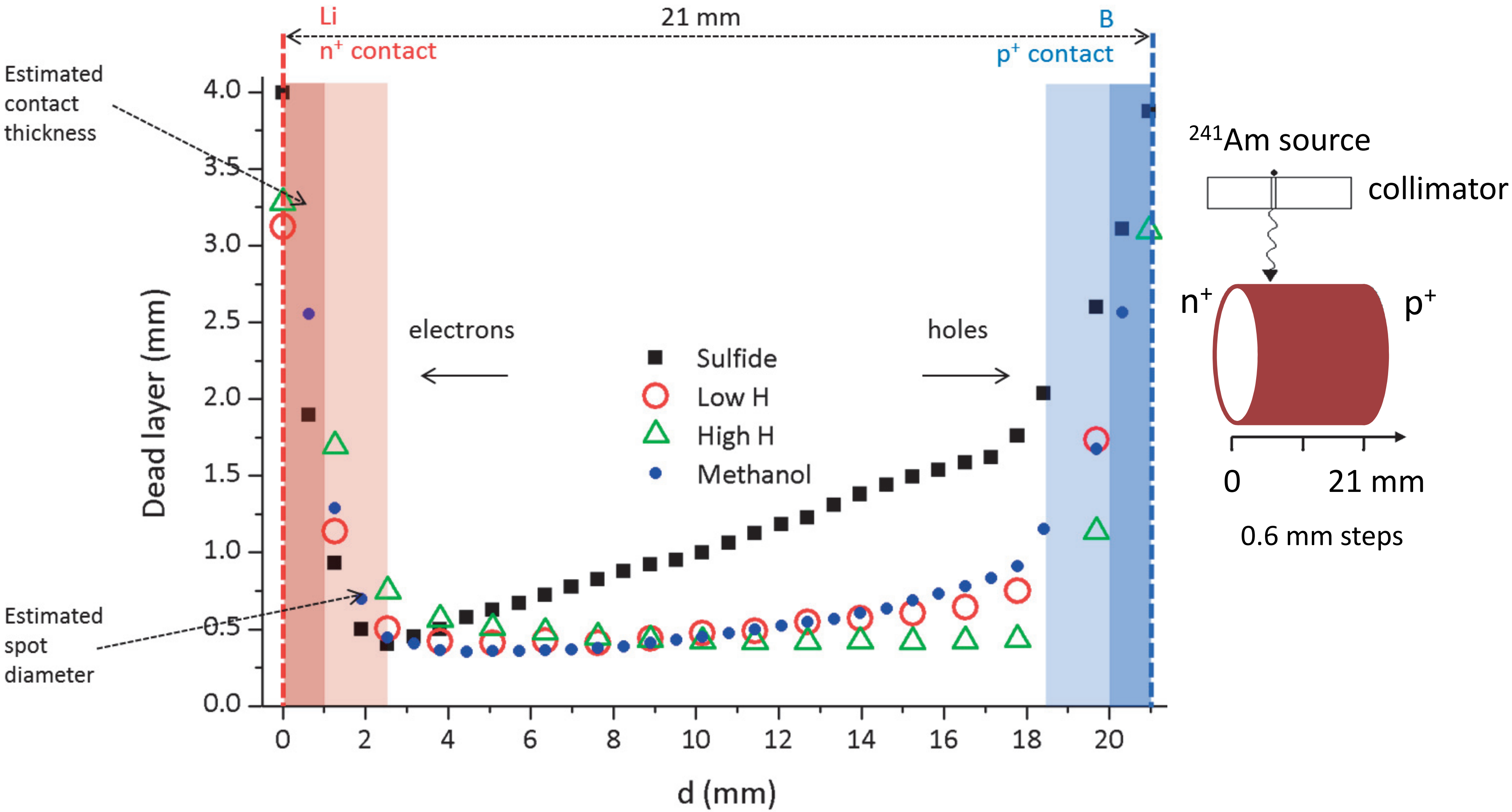


Scan of the passivated surface: counting rate vs distance



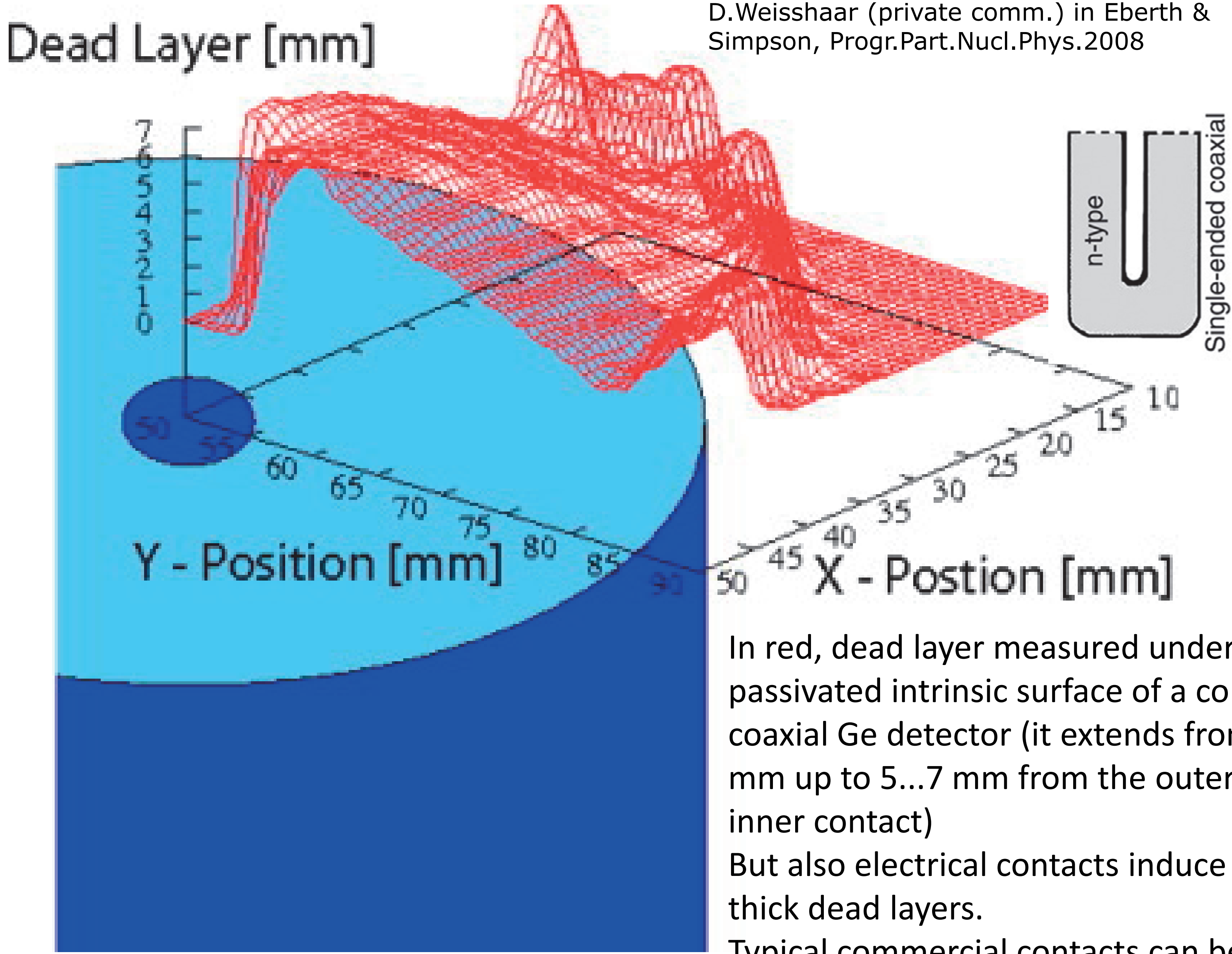
- Strong decrease of counting rate close to the electrodes
- S-terminated detector: n-type surface behavior, which can be related to a negative charge accumulation
- For the other passivations the n-type/p-type behavior is less pronounced

Scan of the passivated surface: dead layer vs passivation



- The average dead layer is below 1 mm, except for S-terminated detector
- The difference in dead layer thickness involves a difference in the active volume, which can reach 10% in the case of the S-terminated detector

D.Weisshaar (private comm.) in Eberth & Simpson, Progr.Part.Nucl.Phys.2008



We need to reduce dead volumes inside the detector!

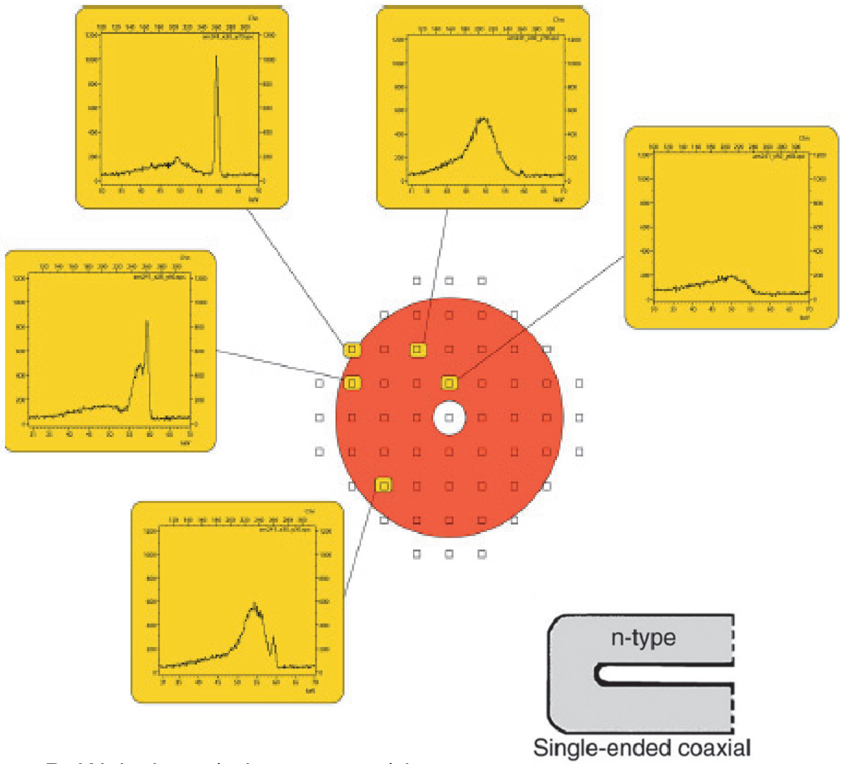
In red, dead layer measured under the passivated intrinsic surface of a commercial coaxial Ge detector (it extends from 1...2 mm up to 5...7 mm from the outer to the inner contact)

But also electrical contacts induce relatevily thick dead layers.

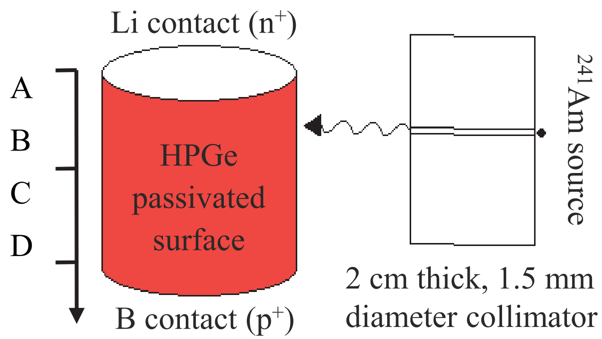
Typical commercial contacts can be: Li, B, a-Ge(H), a-Si(H), Y, etc.

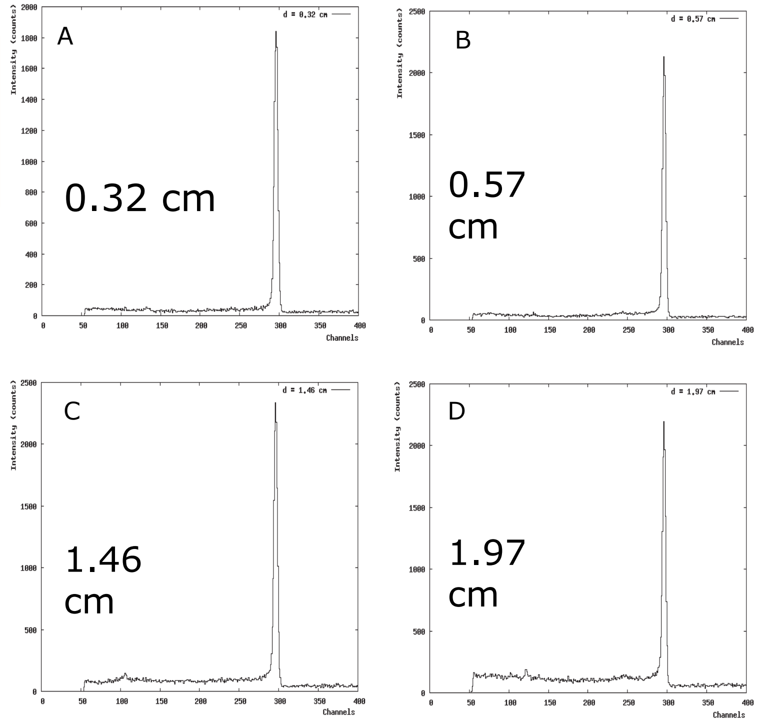
Incomplete charge collection near the passivated surface

Comparison between an LNL prototype and a commercial detector



from D. Weisshaar (private comm.) in
J. Eberth and J. Simpson, Progress in
Particle and Nuclear Physics 60(2008)283





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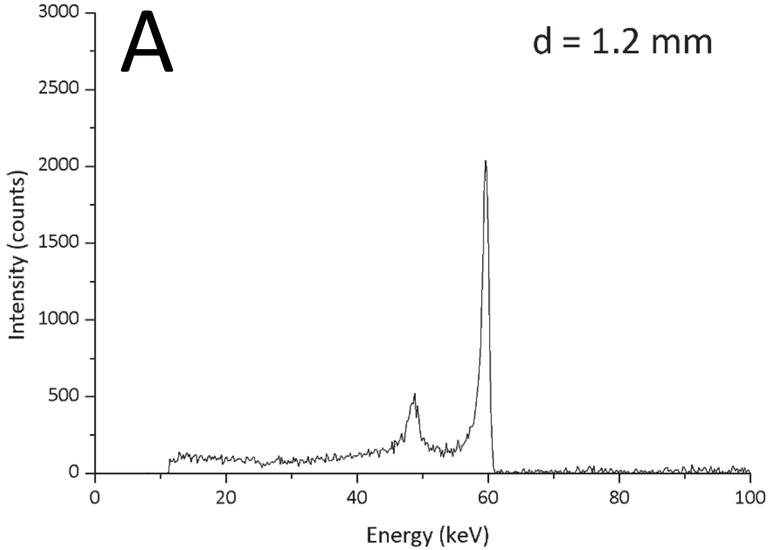
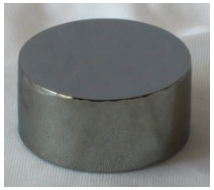
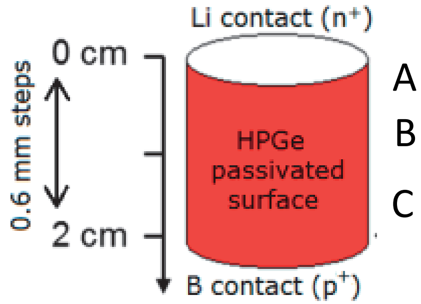
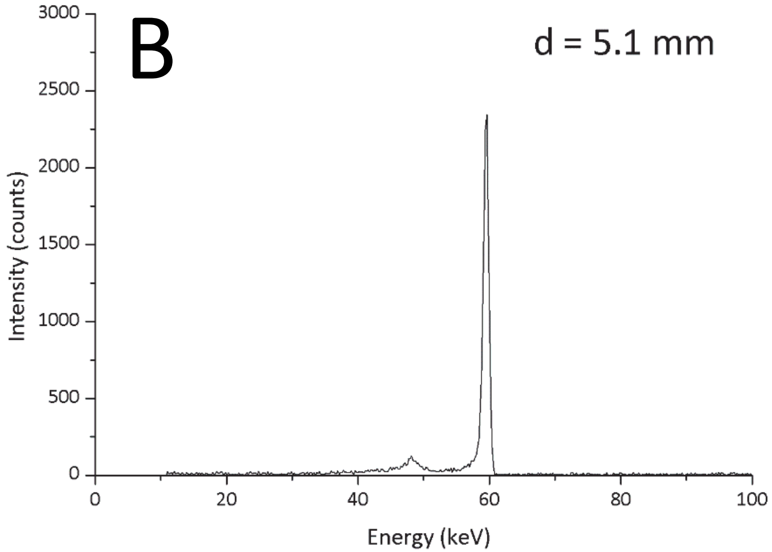
High H-passivated detector: ^{241}Am spectra

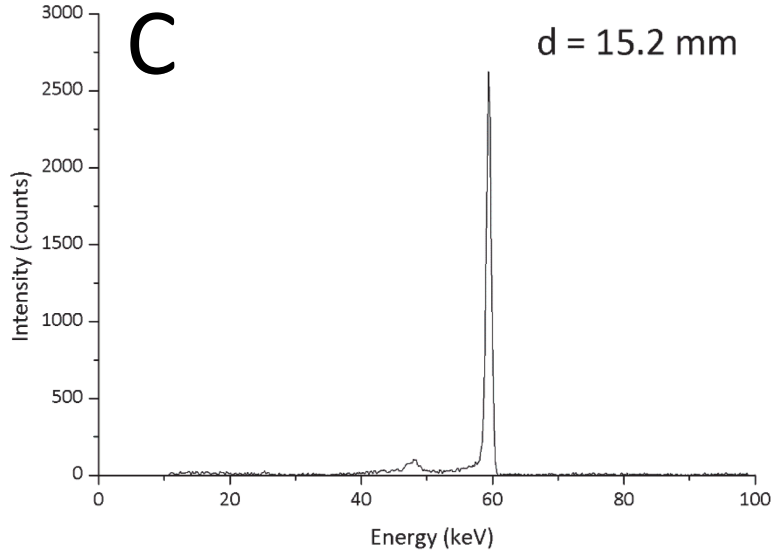




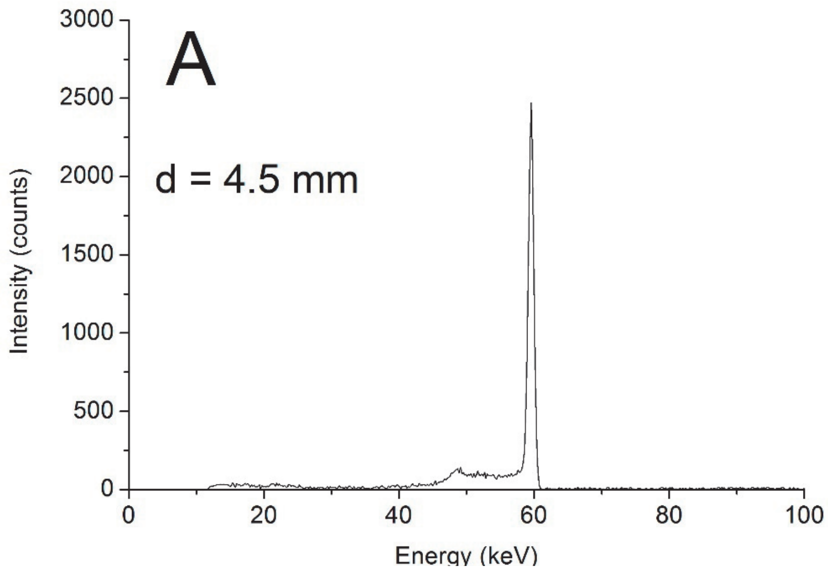


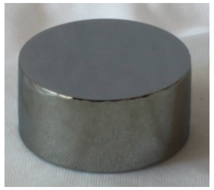
Constant photopeak area normalization

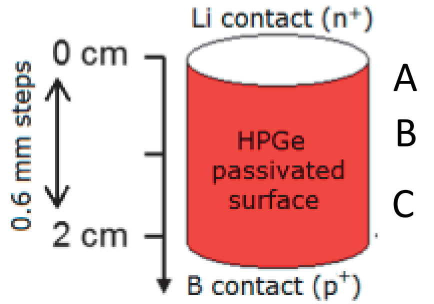




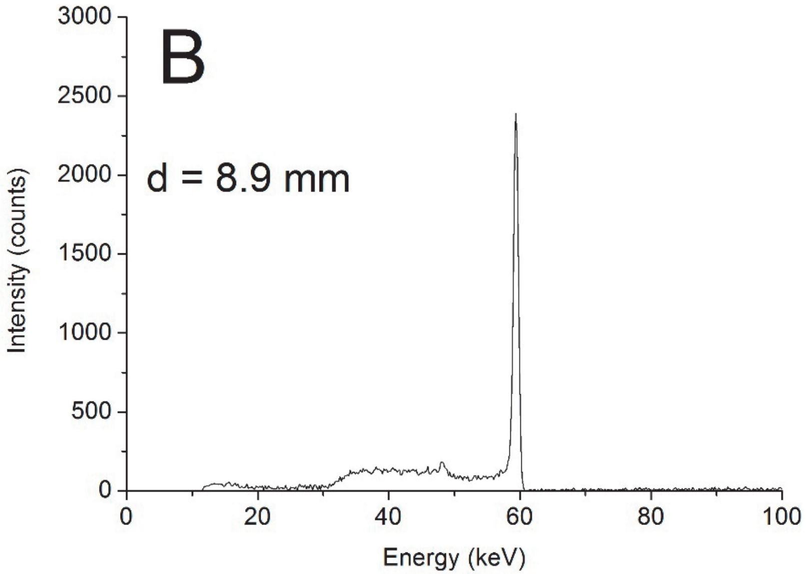
Sulfide passivated detector: ^{241}Am spectra

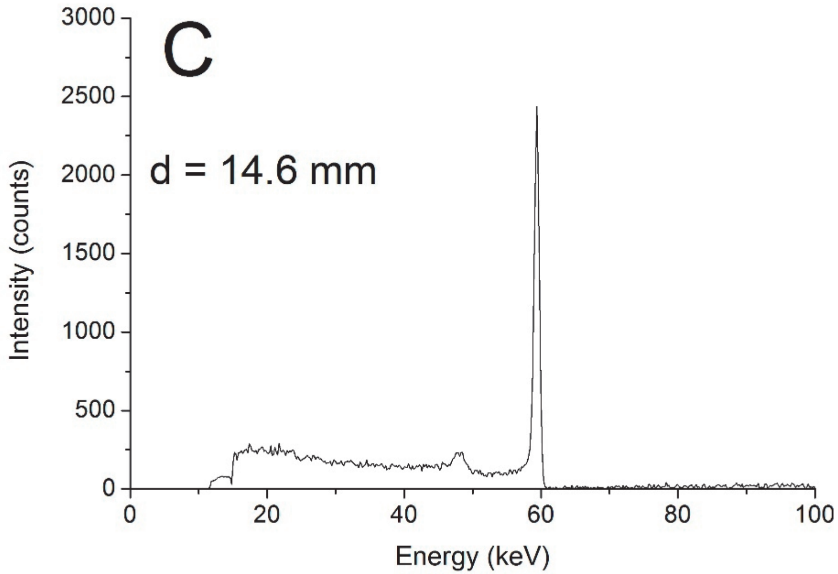




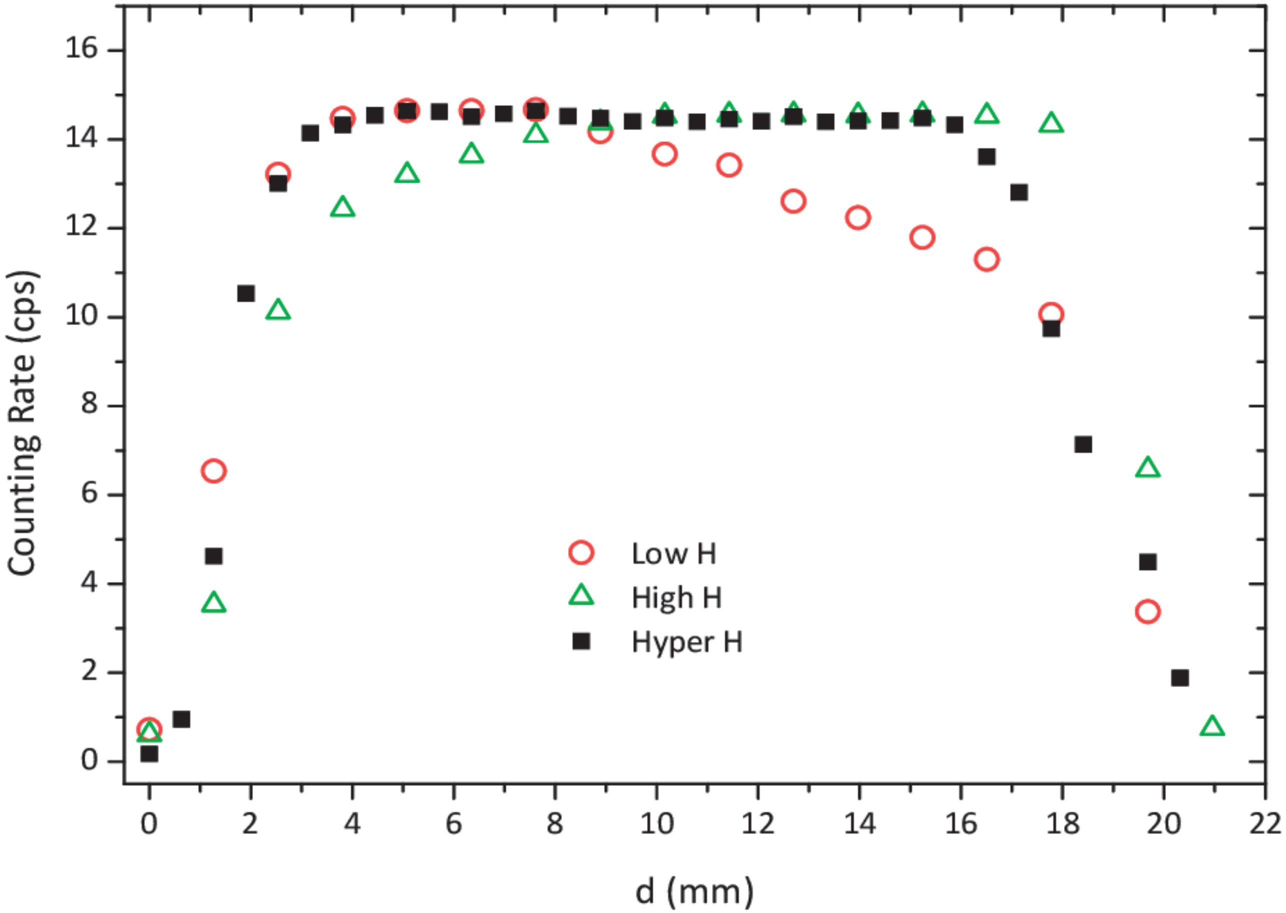


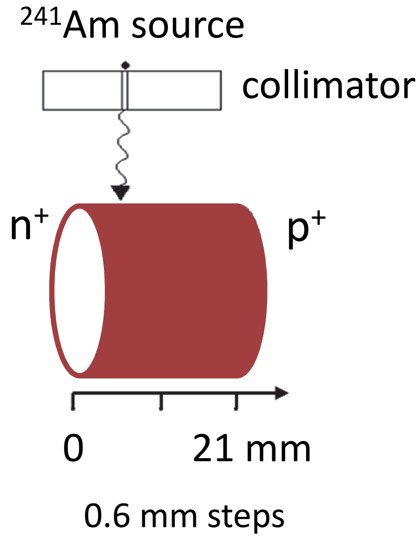
Constant photopeak area normalization





Scan of the passivated surface: counting rate

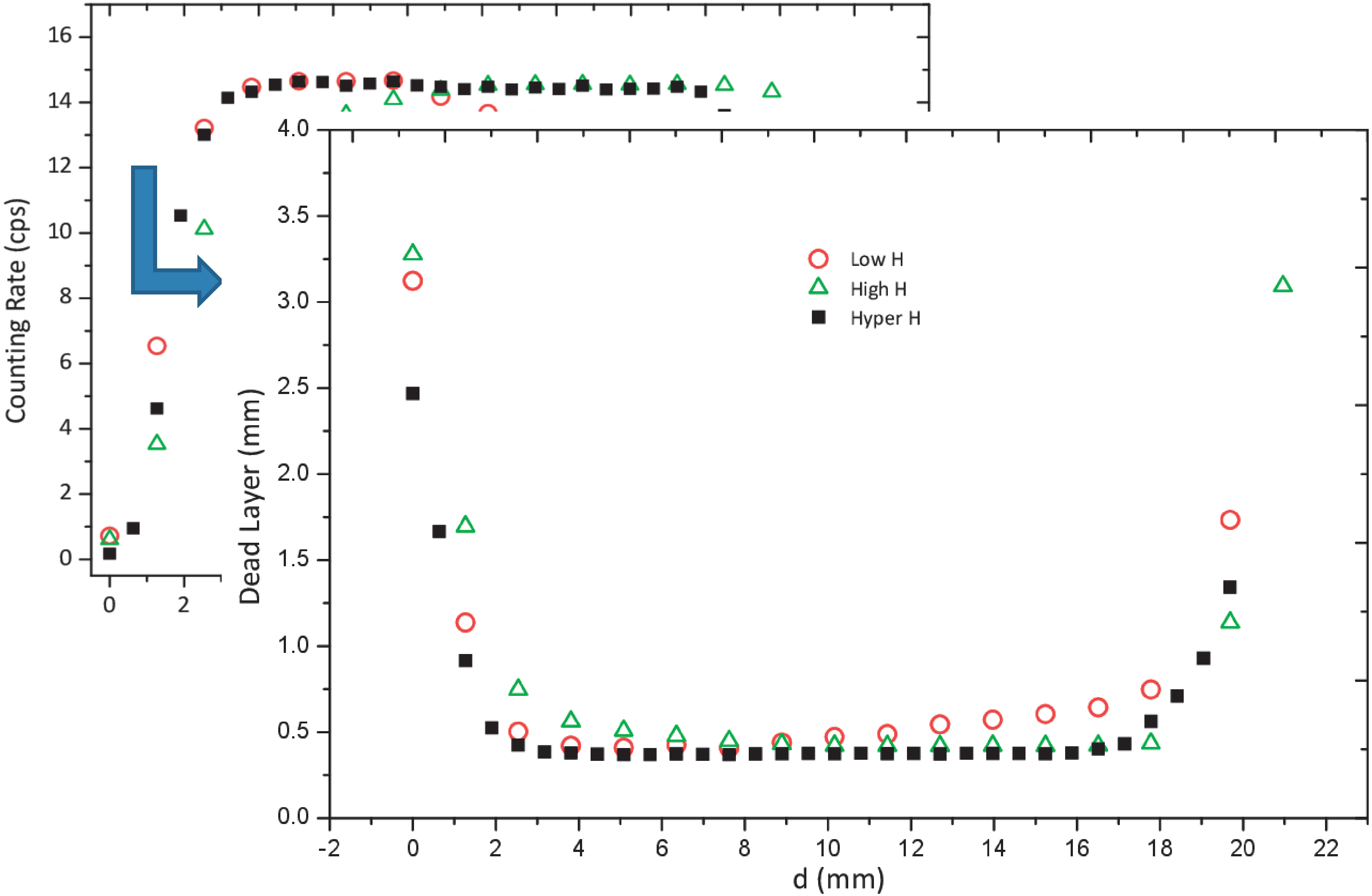




- Low H: slightly n-type surface
- High H: slightly p-type surface
- Hyper H: essentially “neutral”

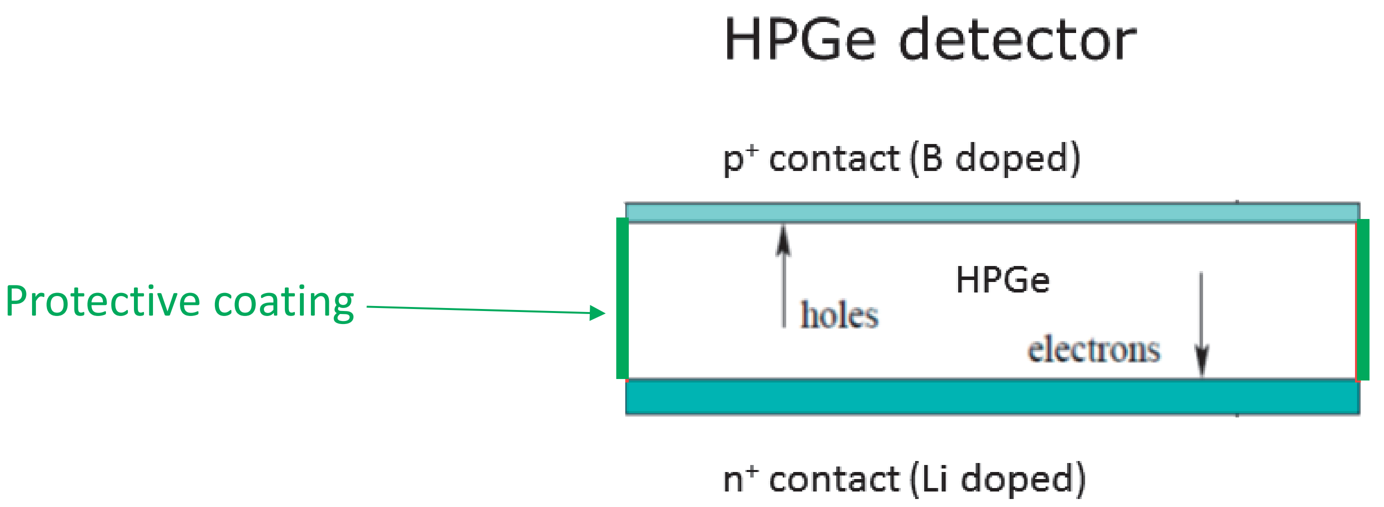
(from S. Riccetto et al., to be submitted to **EPJA**)

Hyper H: the smallest dead volume



Problem: all these passivations are not stable

Solution: New protective coating

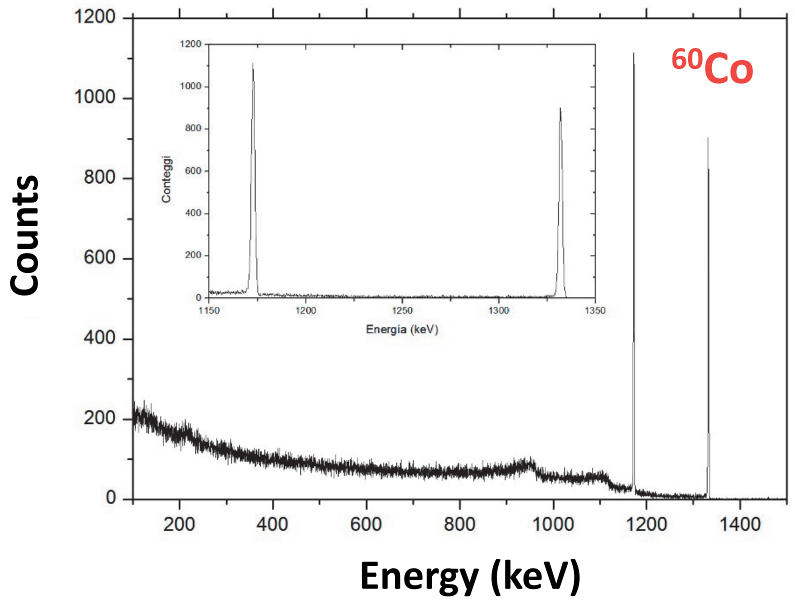


Preliminary tests of coated detectors

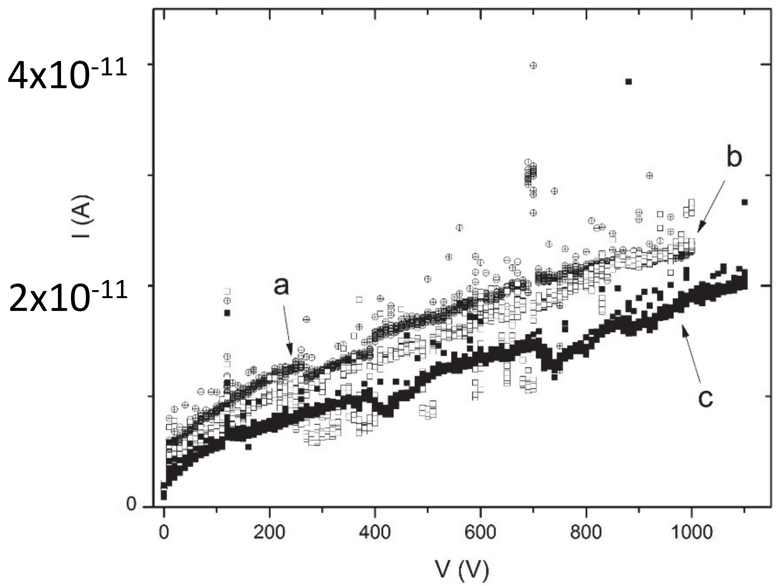
✓ Methanol passivation followed by the protective coating application

- 19 months at room temperature under static vacuum
- 10 minutes at room temperature on the desk
- 40 hours at 103°C under vacuum

The properties of the coated detector do not change after these tests



Leakage current



NOVEL CONTACTS

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Laboratori Nazionali di Legnaro

Development of novel contacts

Thermally-induced dopant diffusion

↓

Thermally-induced contamination

(Impurity diffusion inside semiconductors is a thermally activated process)

Which one of them is predominating?

''Thermal process window''?

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List of studied processes

- ➢ P diffusion by Spin-On-Doping **[V. Boldrini et al., Appl. Surf. Sci. 392 (2017)]**
- ✶ Sb diffusion from a remote sputtered source **[G. Maggioni et al., MSSP (2018)]**
- ✶ High-T annealing treatments on as cut samples

➢ Temperature range: from 600 to 810°C
➢ Time range: from 2 to 30 minutes

- ➢ Reference samples:
  - n-type HPGe } Supplied by Umicore
  - p-type HPGe } Supplied by Umicore
  - B ion implanted (23 keV, 1e15 cm^{-2})

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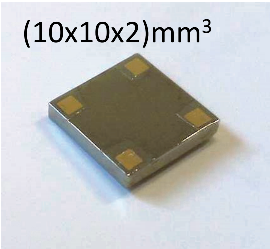
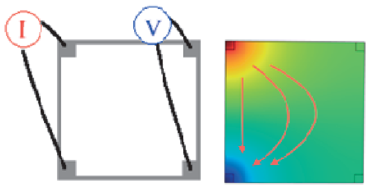
Contamination of HPGe – LTA technology

Four-wire resistance and Hall measurement



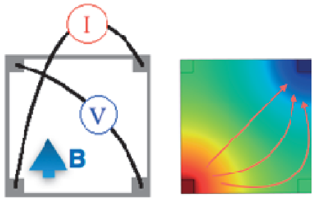
Van der Pauw method

R_{sheet}



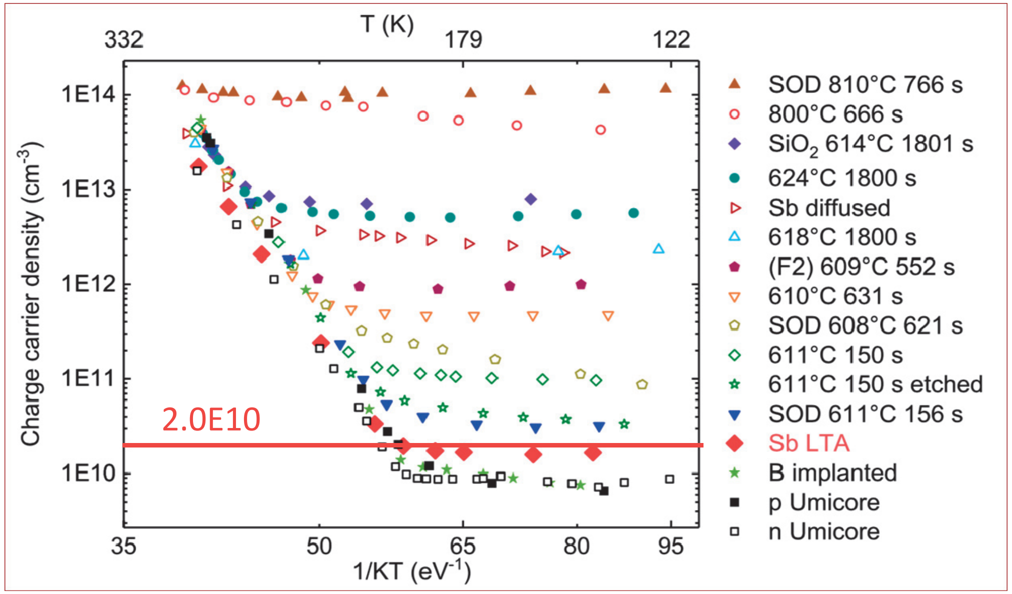
Hall-effect method

Carrier type



[From V. Boldrini et al., **Mater. Sci. Semicond. Proc.** (2018), *submitted*]

Contamination after laser treatment



Carrier density within the specifications (0.4-2.0E10 cm^{-3})

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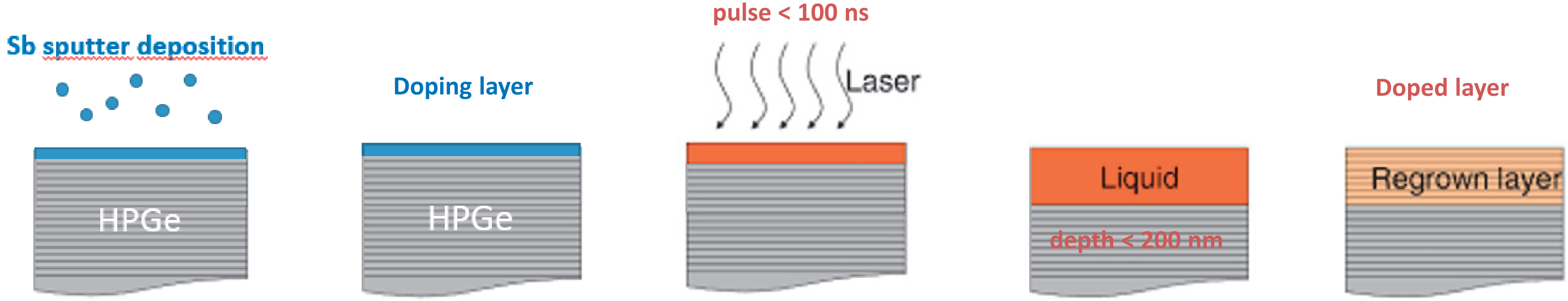
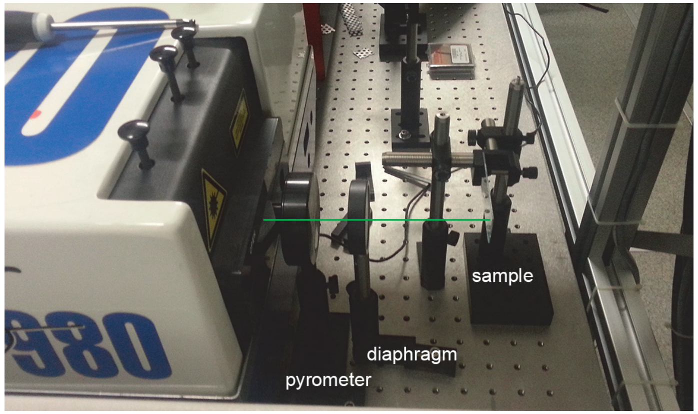
n^+ contact on HPGe – LTA technology



Advantages:

- Melting temperature is reached and maintained for a short time (<100 ns)
- Only the surface (< 200 nm) is melted, the bulk is at room temperature
- High dopant concentrations →
  → near-ideal p-n- junctions
- Doping with heavy elements without crystal damage
- Very clean process suitable for preserving the Ge hyperpurity
- Suitable for complex contact geometries (segmentation)



Pulsed Laser @ LNL-INFN

<u>Nd:YAG</u>

λ=355 nm (third harmonic generator)

τ=7 ns; Ø=6,5 mm; Rate = 10Hz

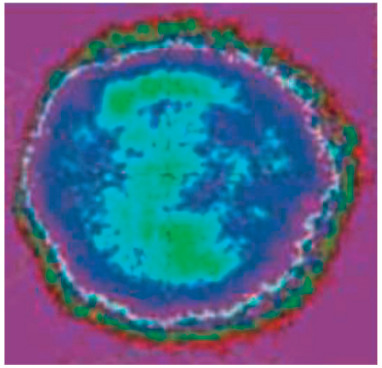
Radiant power ~1500 mW

Fluence ~300-400 mJ/cm^2

The small detector prototype

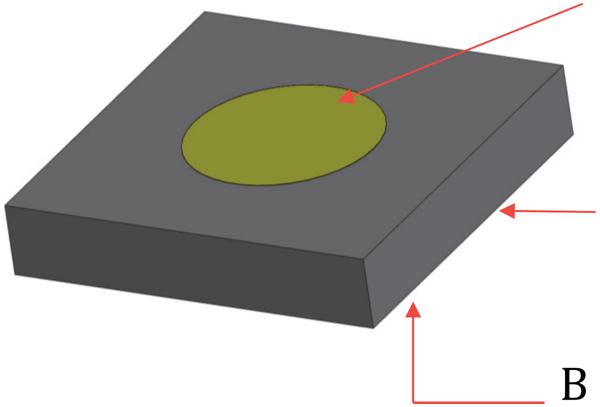
Laser geometry was favoured at the expense of detector performance

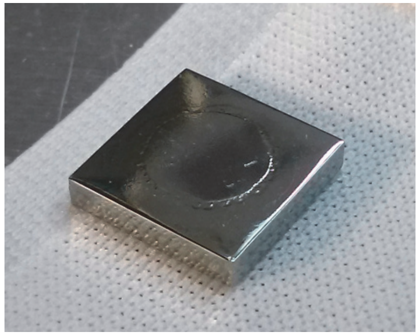
Laser spot



Ø = 7 mm

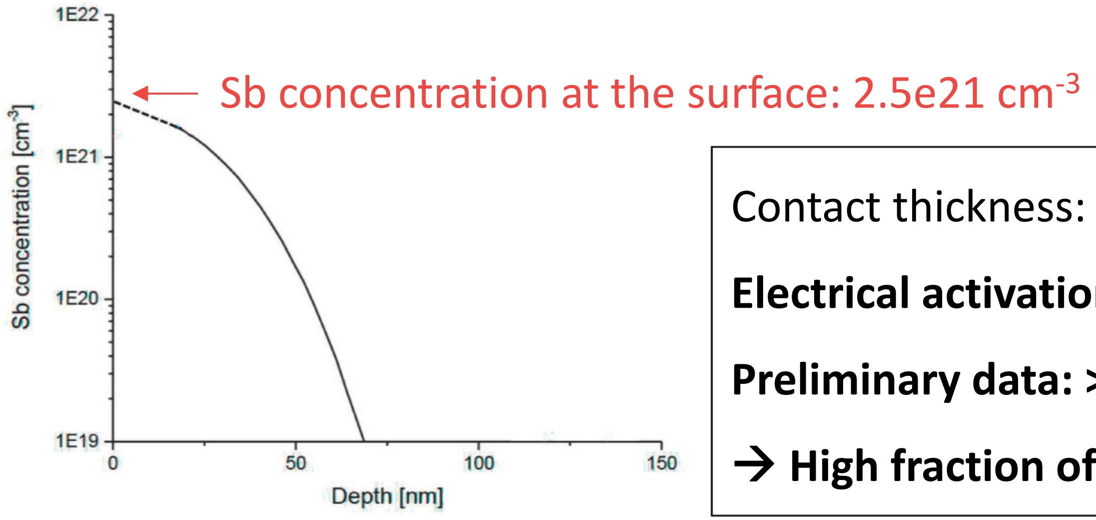
Detector sample
(10 x 10 x 1.9 mm^3)





No guard ring

Sb diffusion profile (SIMS)



Contact thickness: ≤ 100 nm

Electrical activation of dopant

Preliminary data: > 50%

→ **High fraction of substitutional Sb**

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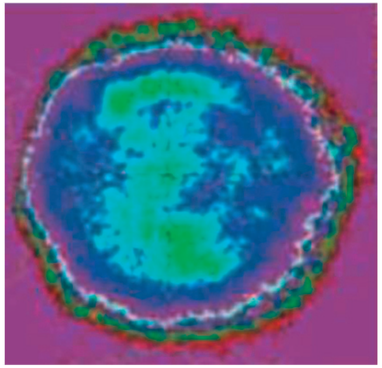
The small detector prototype

3^{rd} PSeGe Workshop – IPHC, Strasbourg 2018
Daniel R. Napoli

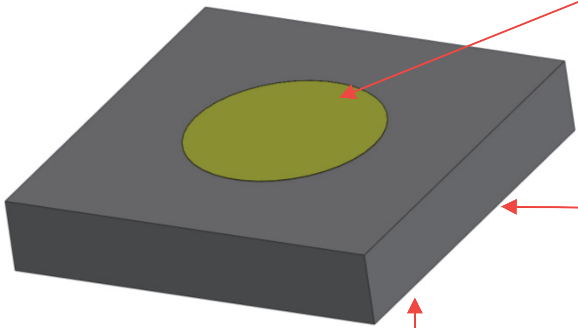
Laser geometry was favoured at the expense of detector performance

Laser spot



Ø = 7 mm

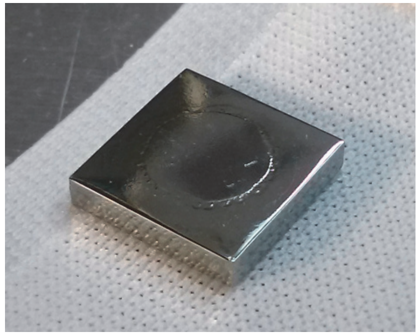
Detector sample
(10 x 10 x 1.9 mm^3)



Sb contact
Ø = 5 mm

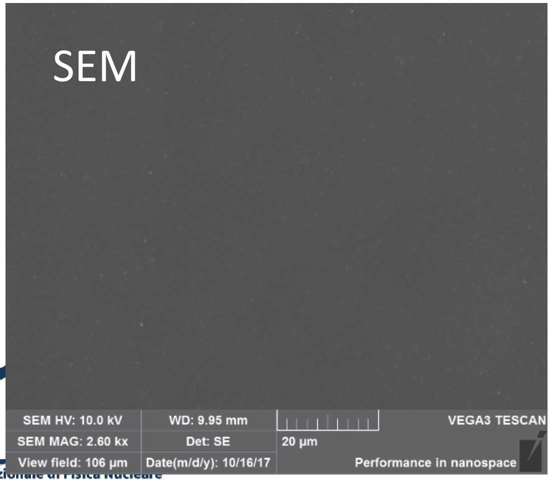
P-type HPGe

B contact
10 x 10 mm^2

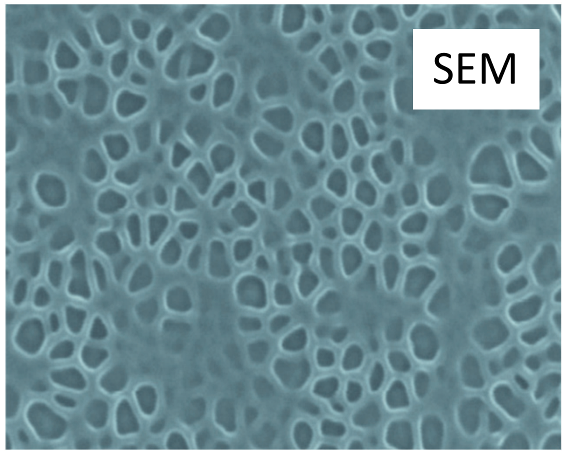


No guard ring

Surface morphology:
No laser-induced defects



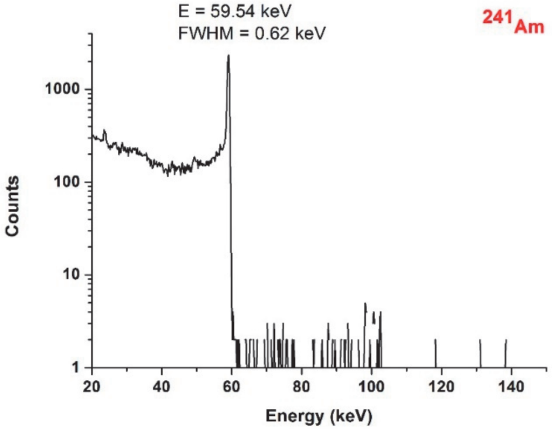
Sb
Ion Implantation
(6×10^{15} Sb cm^{-2} , 50 KeV, at LN_2)

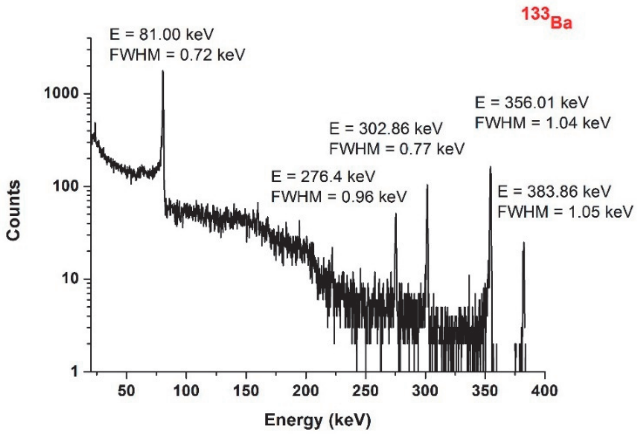


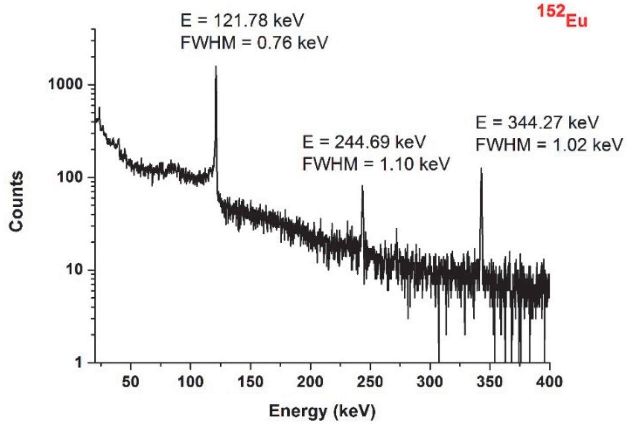
(from E. Bruno et al., **JAP** (2010))

Test of small HPGe prototype: detector configuration

Sources: ^{241}Am, ^{152}Eu and ^{133}Ba

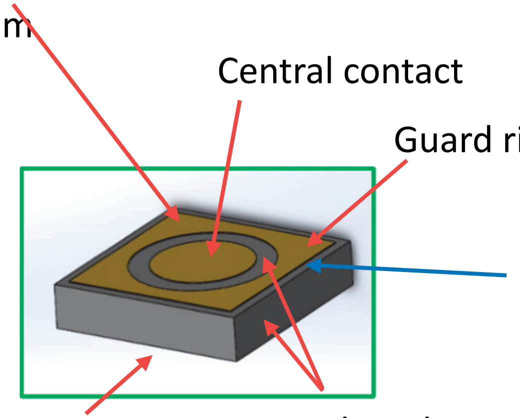






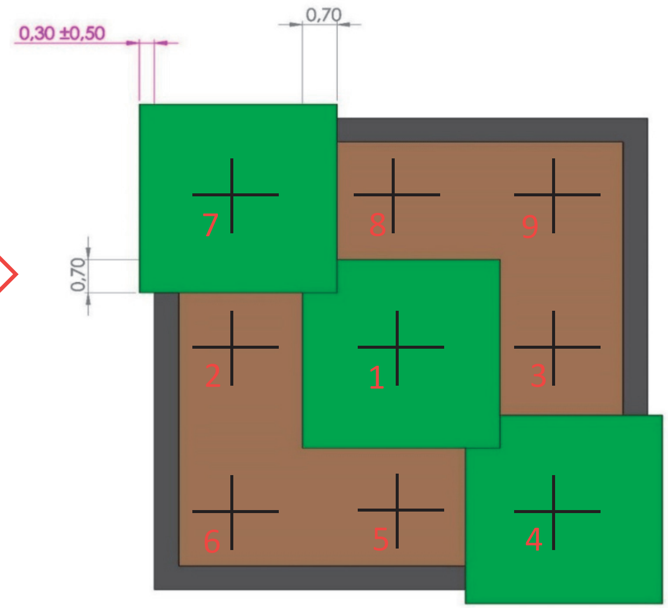
Good energy resolution all the energy range up to 400KeV

Prototype HPGe by LTA technology @LNL-INFN

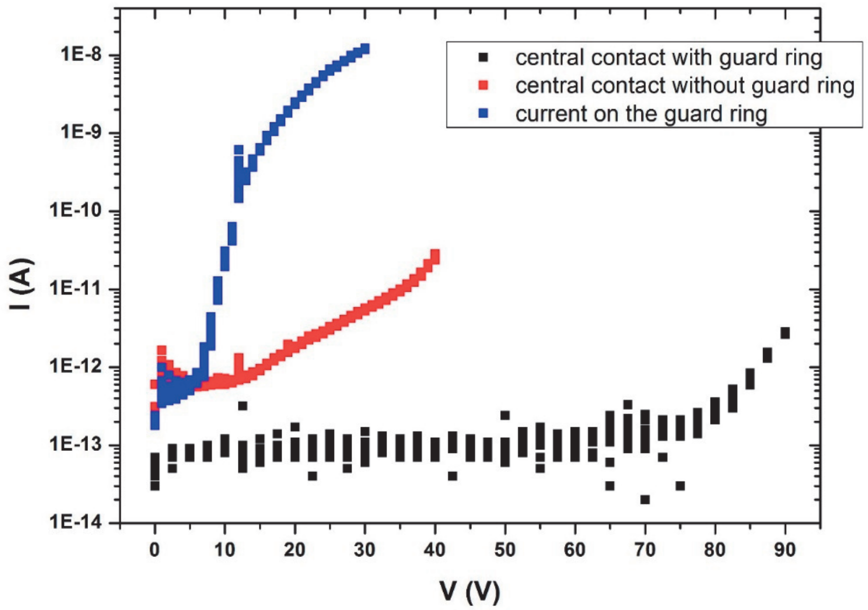


Laser thermal annealing

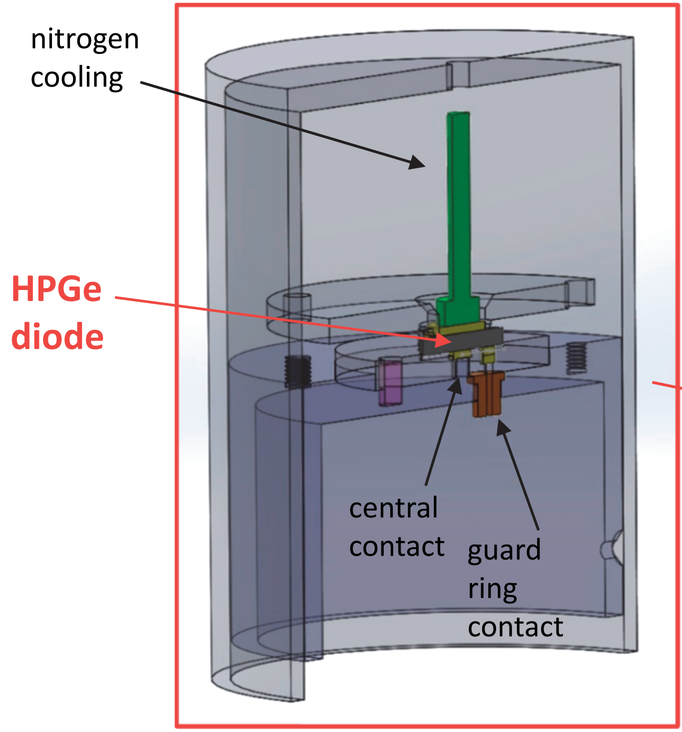
with 9 laser spots (4x4)mm^2 all the Sb surface (10x10)mm^2 was annealed



Test of small HPGe prototype: I-V diode configuration



3D cryostat design

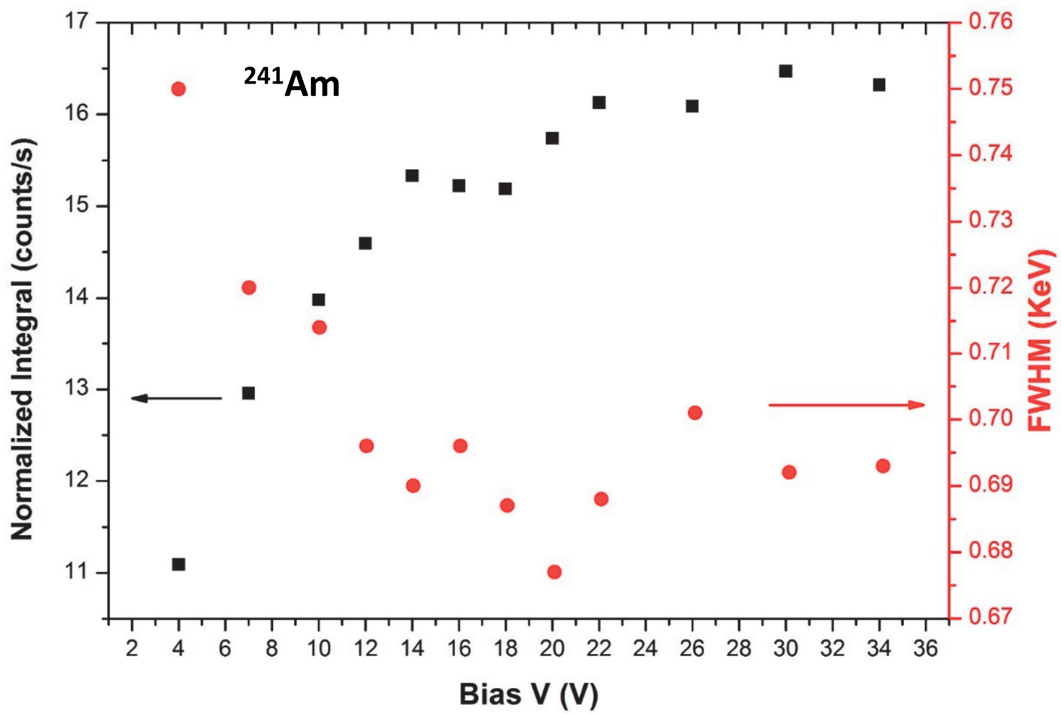


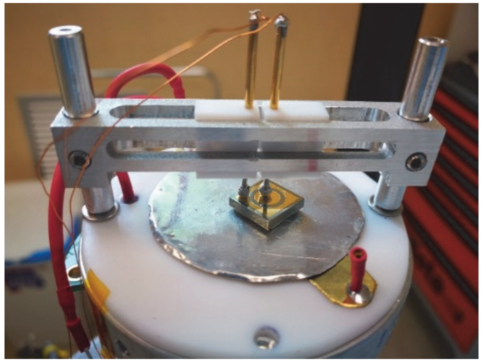
Central contact low level of leakeage current, 1pA @ 80V

Direct bias V=0.5 V I=0.8 μA

Test of small HPGe prototype: detector configuration

Depletion Voltage - Energy resolution



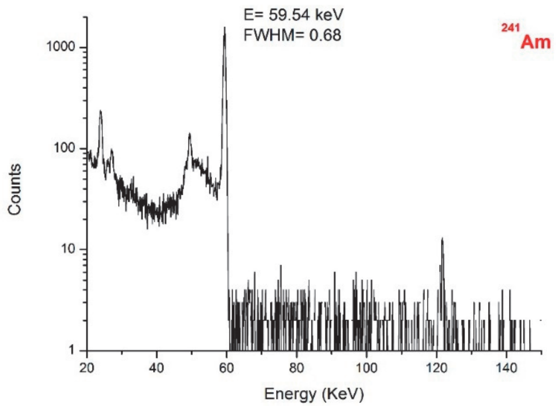


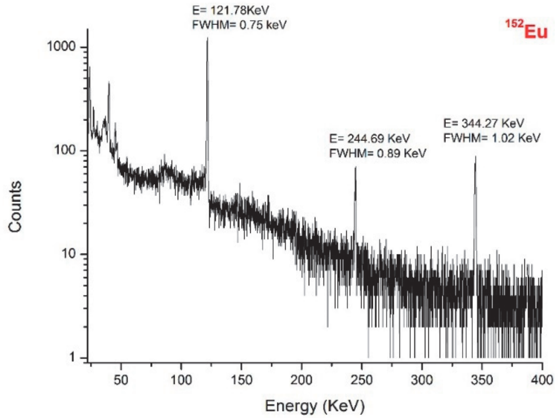
central contact
0.68 keV @ 59.54 keV (^{241}Am)

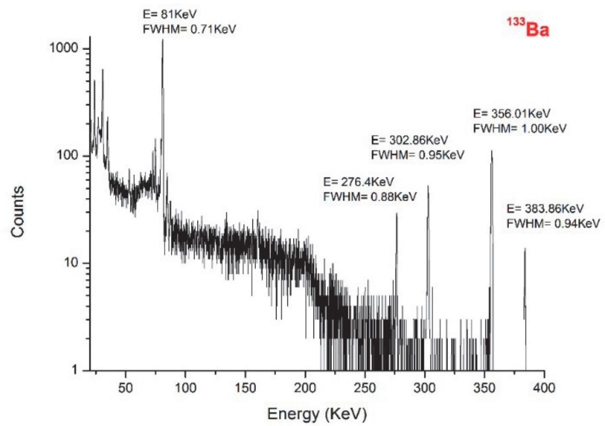
- detector fully depleted (plateau of normalize integral)
- very good energy resolution

Test of small HPGe prototype: detector configuration

Sources: ^{241}Am, ^{152}Eu and ^{133}Ba







Good energy resolution all the energy range up to 400KeV

SUMMARY

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Summary

- Several passivation routes have been applied to a planar HPGe detector and their effects on the detector characteristics have been investigated.
- Lateral scan measurements highlighted the electrical nature of the passivated surface (either n-type or p-type) and its influence with bulk propertis of Ge dets.
- Hydride-based passivations are the bests to reduce dead layers in HPGe dets.
- A detector coating has been developed to substitute the mechanical incapsulation for protecting and stabilizing the passivated surfaces (also in between segments!)
- A new method to produce contacts based in laser annealing techniques is very promising. A novel Sb contact has been developed.

Work in progress

- Development of planar detectors with laser-annealed Sb contacts (scaling up)
- Development of detector samples with novel contacts using different deposition methods (vacuum evaporation, spin coating, etc.) and different doping elements (P, As for n contacts and B, Ga for p contacts).