Passivations and Dead Layers in HPGe Detectors: Recent R&D Activity at LNL

Gianluigi Maggioni

INFN – Legnaro National Laboratories

Location
- 40 km (25 miles) from Venice
- 8 km (5 miles) from Padua

INFN – Legnaro National Laboratories

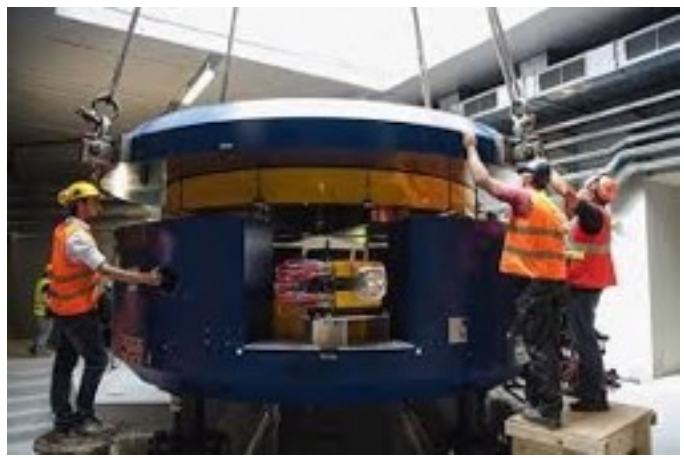
Facilities

+

ALPI & PIAVE

INFN
Istituto Nazionale di Fisica Nucleare
Laboratori Nazionali di Legnaro

SPES Project



5500h/y

4500h/y

46% ENEA, ISS & Universities for Interdisciplinary activities and applications

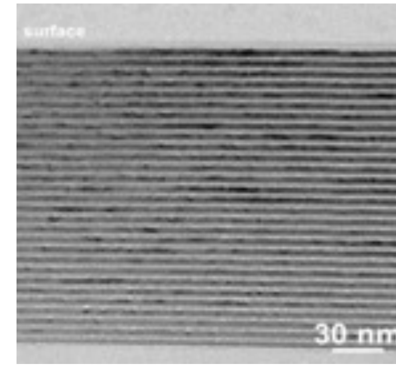
10.000 hours of beam on target/year

p → Au, from keV/A → few MeV/A, from few pnA → few pμA

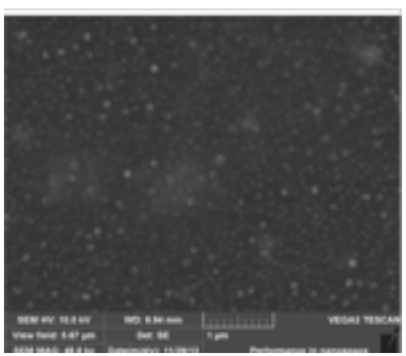
Materials & Detectors Laboratory

- R&D on novel thin film materials and surface treatments
- Characterization of the properties of thin films and surfaces

PVD facilities



EUV mirrors (multilayer)



High-k nanocomposite Parylene materials

Luminescent solar concentrators

Organic scintillators for neutron detection

HPGe Chemistry Laboratory

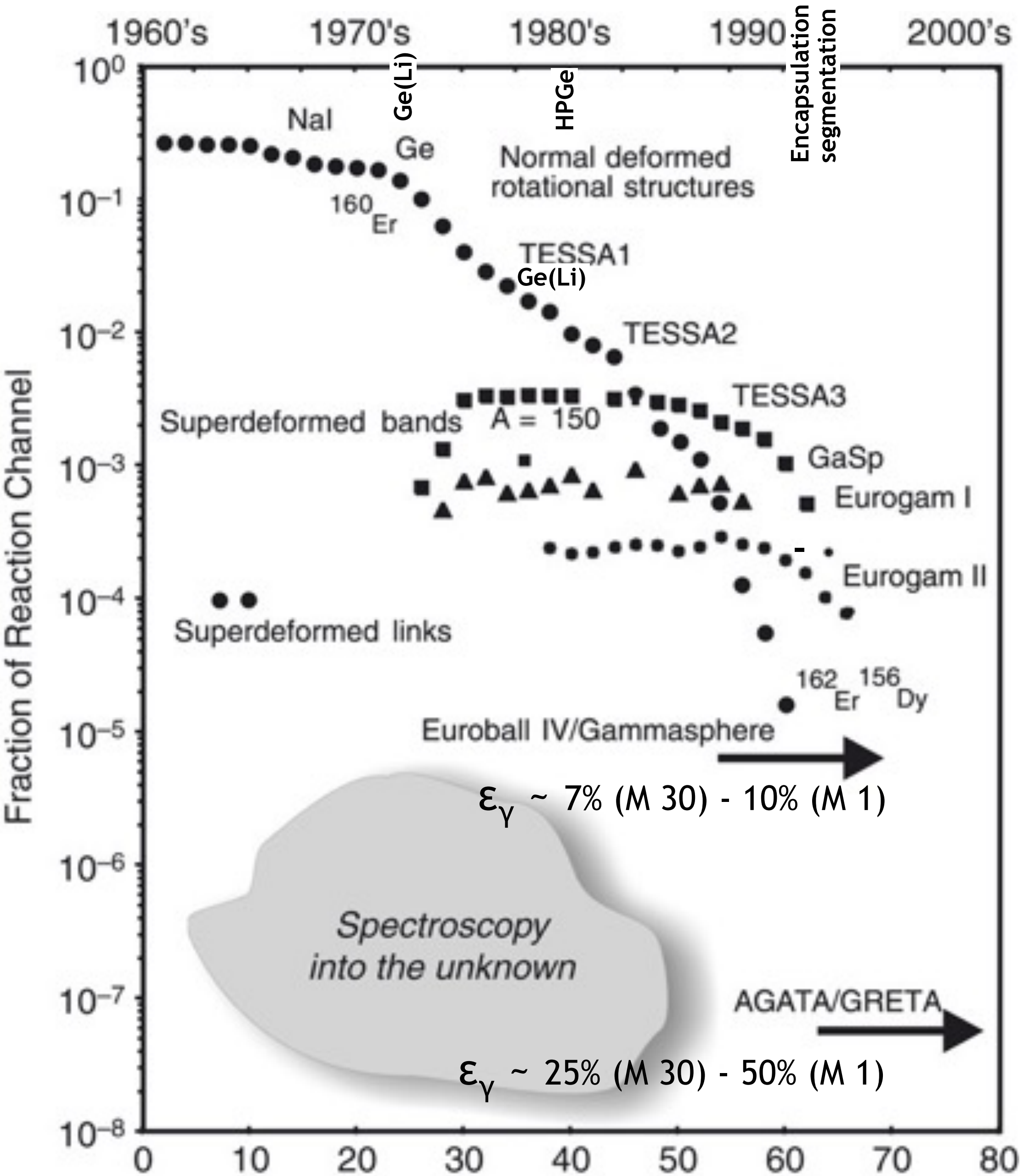
- R&D on HPGe detectors: passivations and new contacts
- Chemical and thermal treatments of semiconductor materials

Dedicated PVD

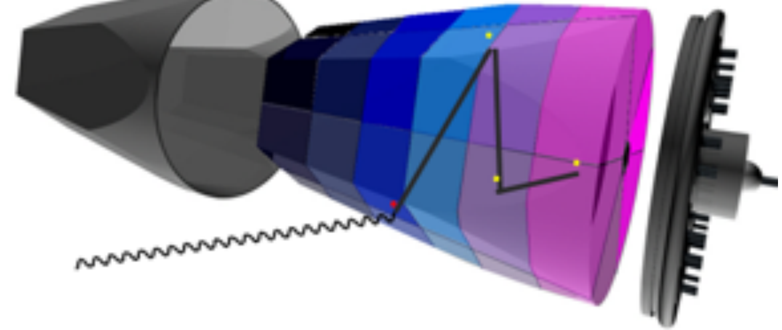
- **Temperature control (±0.1˚C)**
- **Relative humidity control (RH < 50%)**
- **Particulate control (ISO 7)**
- **Access control**

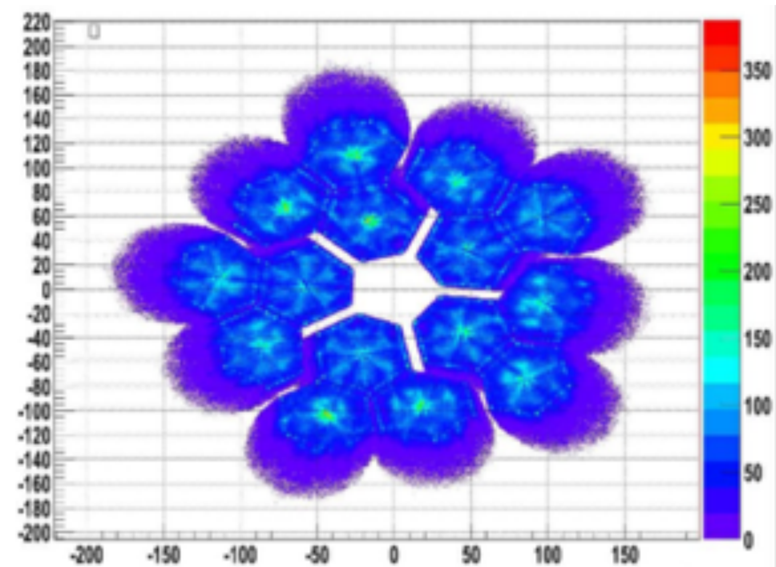
Thermal treatments in controlled atmosphere

HPGe detectors



Thanks to J. Simpson



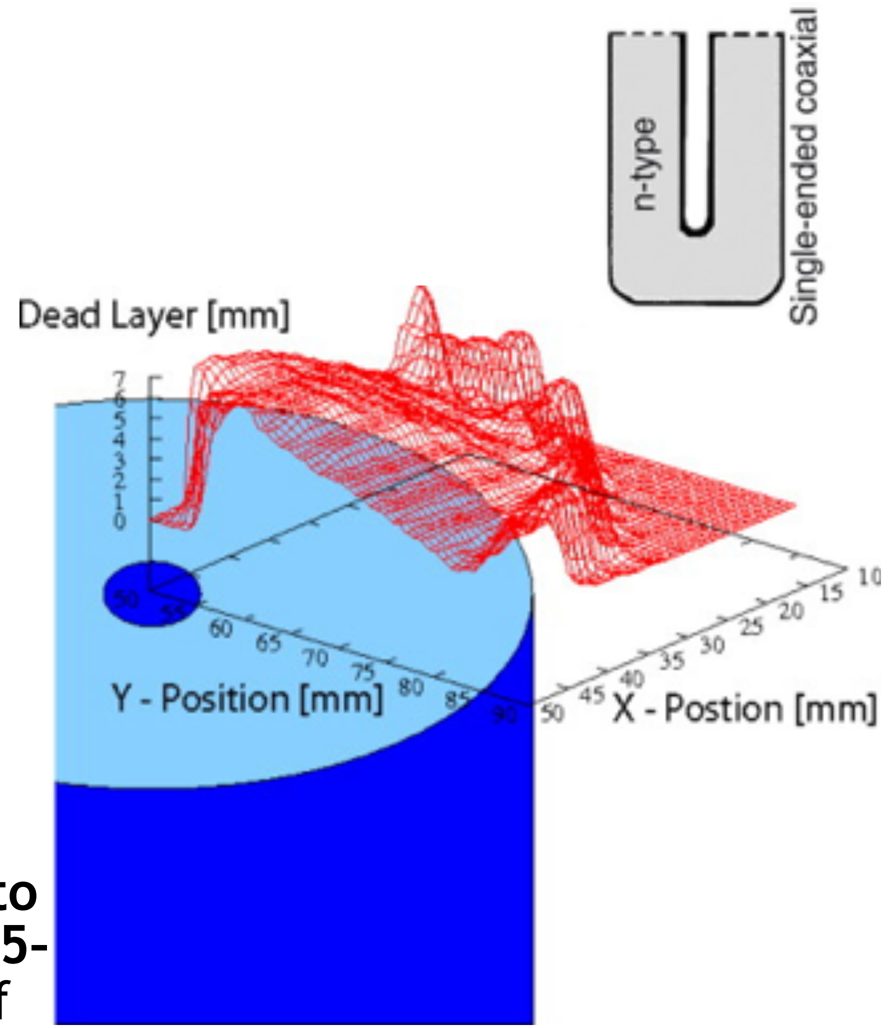


State of the art

- Highly segmented HPGe detectors
- Encapsulated HPGe detectors

HOT TOPIC: dead layers inside the detector volume

- The electrical contacts (n^+ and p^+) can induce a thick dead layer, especially Li contact which is several hundreds of micron thick
- The passivation of intrinsic surfaces can produce dead layers



- The dead layers at the intrinsic surface of a **coaxial Ge detector** have been measured to extend **from 1 to 2 mm** at the outer radius **to 5-7 mm** at the inner contact of the detector.

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

Aims 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 techniques

- To develop new thinner contacts, which could replace the present ones (especially Li)

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Virginia Boldrini's talk (tomorrow morning)

Two alternatives:

- Commercial detectors

The easier way

No complete control

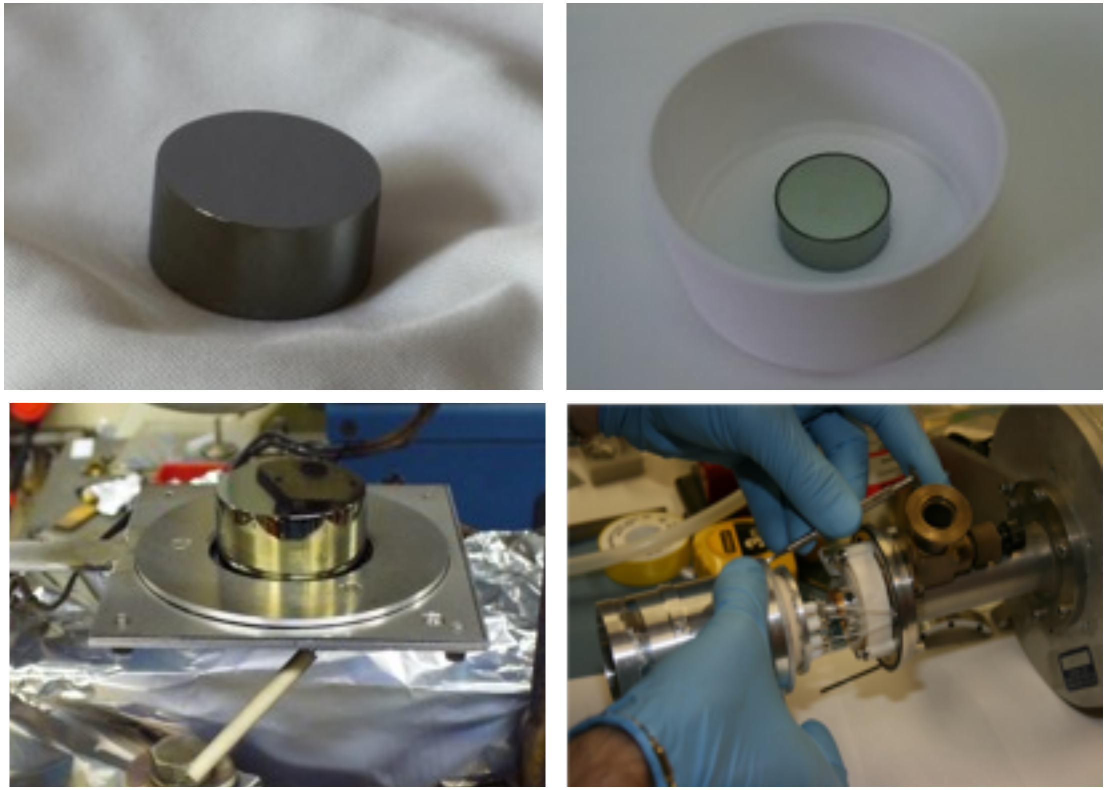
- Home-made detectors

The harder way

Complete control

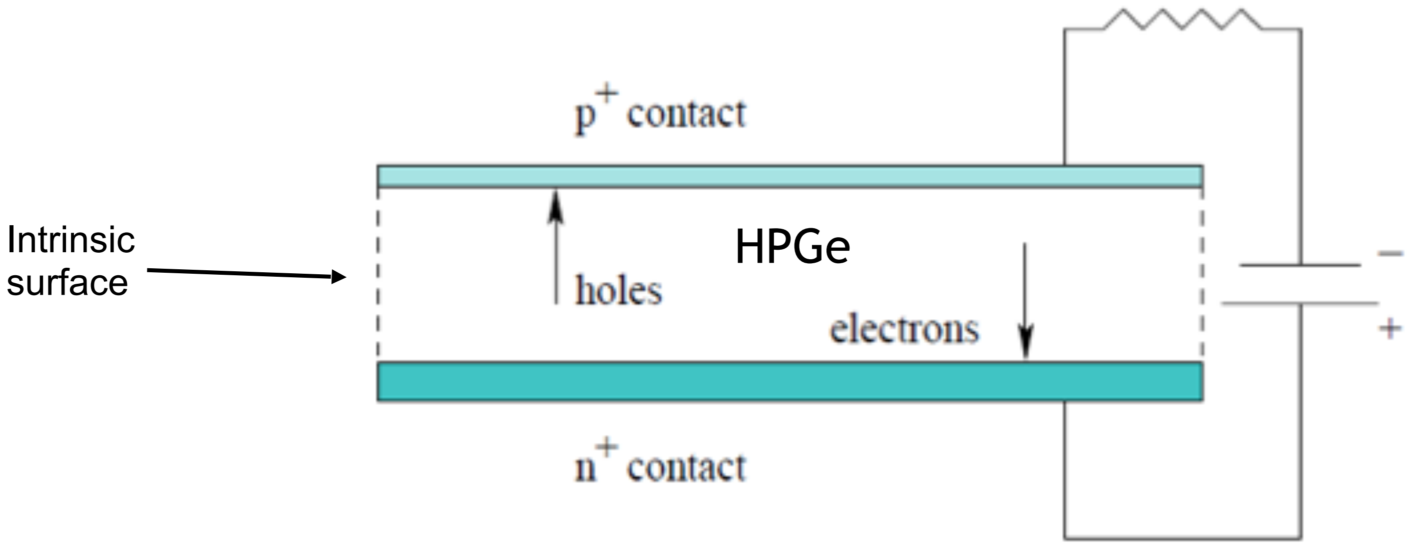
R&D on HPGe production technologies *(Planar HPGe detectors)*

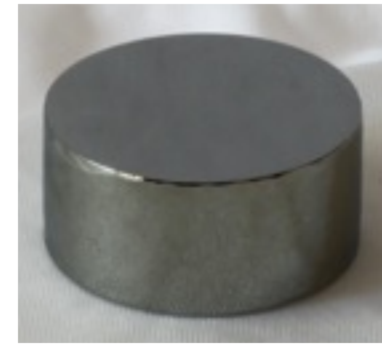
Basic know-how for the **in-house** production of HPGe detectors



- 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 and In protection
- Surface passivation & mounting

HPGe detectors produced for our studies



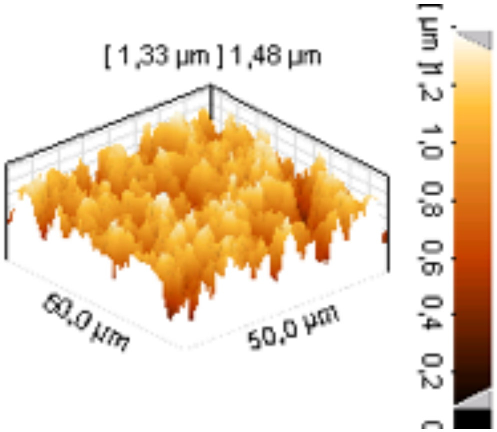


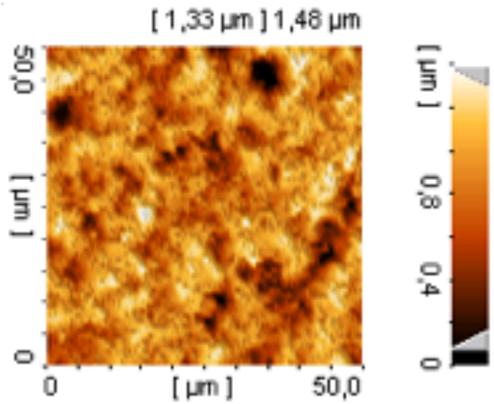
Diameter: 40 mm
Height: 20 mm

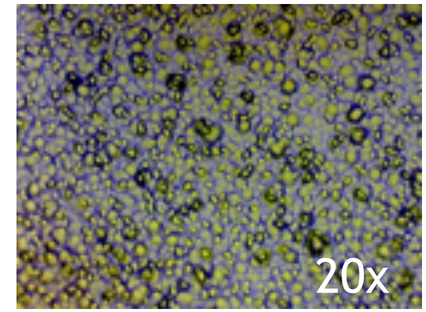
- An HPGe detector is a diode produced from an extremely pure p-type or n-type Ge single crystal with a maximum net impurity concentration of 10^{10} atoms cm^{-3}.
- The contacts of the diode are usually made by boron implantation (about 0.3 μ thick) on one side and by Lithium diffusion (about 600-μm thick) on the other side.
- In p-type bulk material, the Li-diffused contact forms the pn-junction and the boron-implanted contact limits the possible depletion layer, while for n-type material the contacts act in the opposite way.
- The intrinsic surface in between the contacts has to be electrically passivated in order to avoid surface leakage currents when the high voltage (about 1kV/cm) is applied. The ideal passivation needs to have flat band conditions, to introduce no additional noise and has to be stable against thermal cycles.

Mechanical treatment of the HPGe surfaces (lapping)

AFM



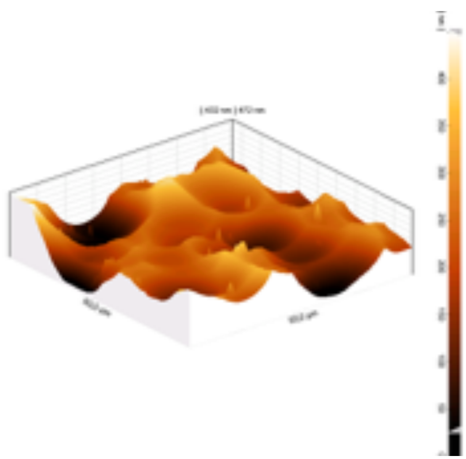


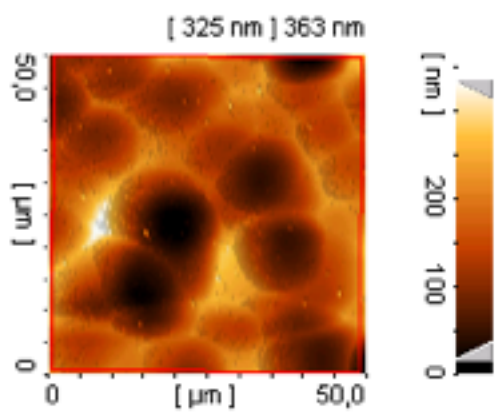


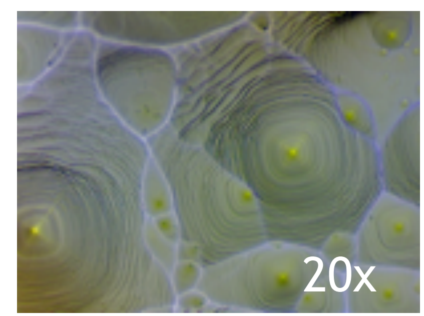
Count of the crystal dislocations

Roughness after lapping: 150 - 250 nm

Chemical preparation of the HPGe surfaces (etching)



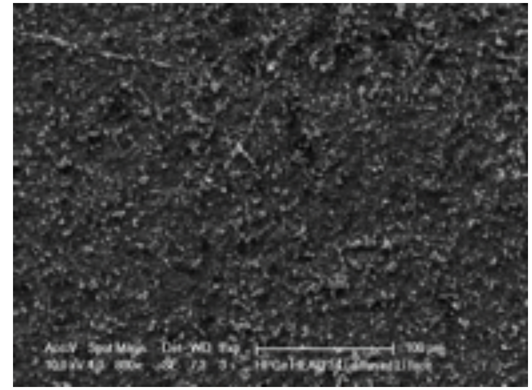




Roughness after etching: 50 - 100 nm

n+ -contact made with Li diffusion method

Evaporation apparatus

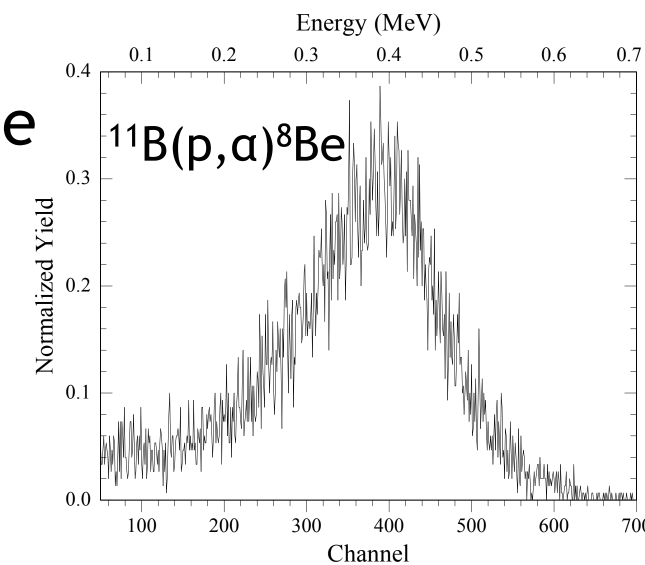


Li-coated surface

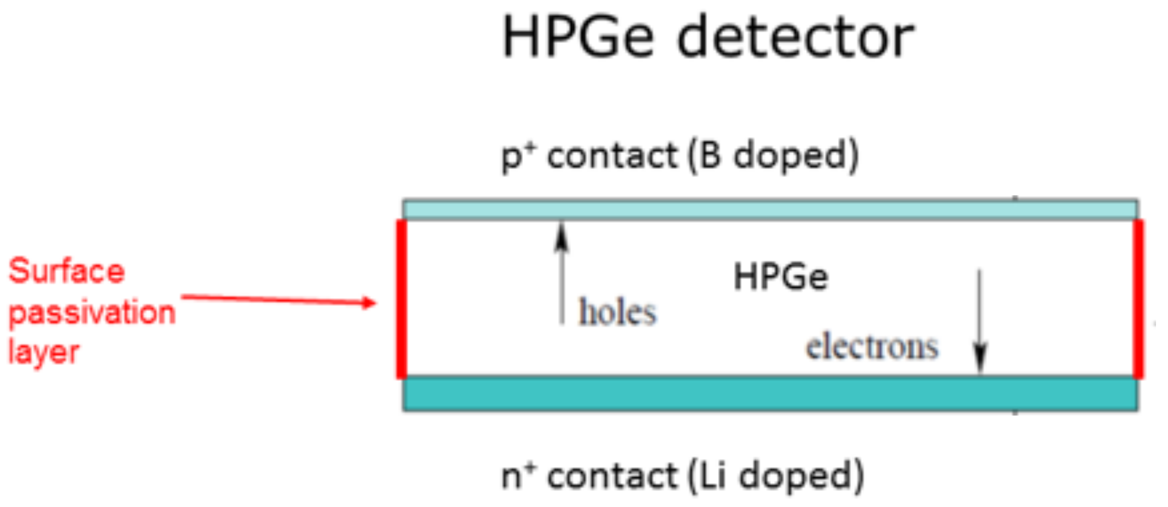
p+ -contact made with B implantation

Ion implantation apparatus

NRA technique

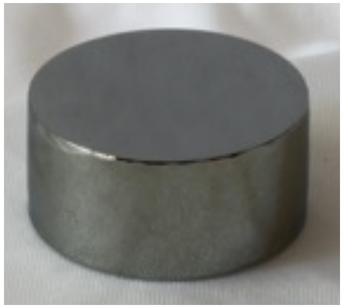


Passivation of the intrinsic surface



The **same detector** (n type planar detector) was used for **all** the different passivation treatments.

Bulk effects due to different crystals are avoided



Diameter: 40 mm
Height: 20 mm

What kind of passivation?

Ideal Passivation → electrical: low leakage current

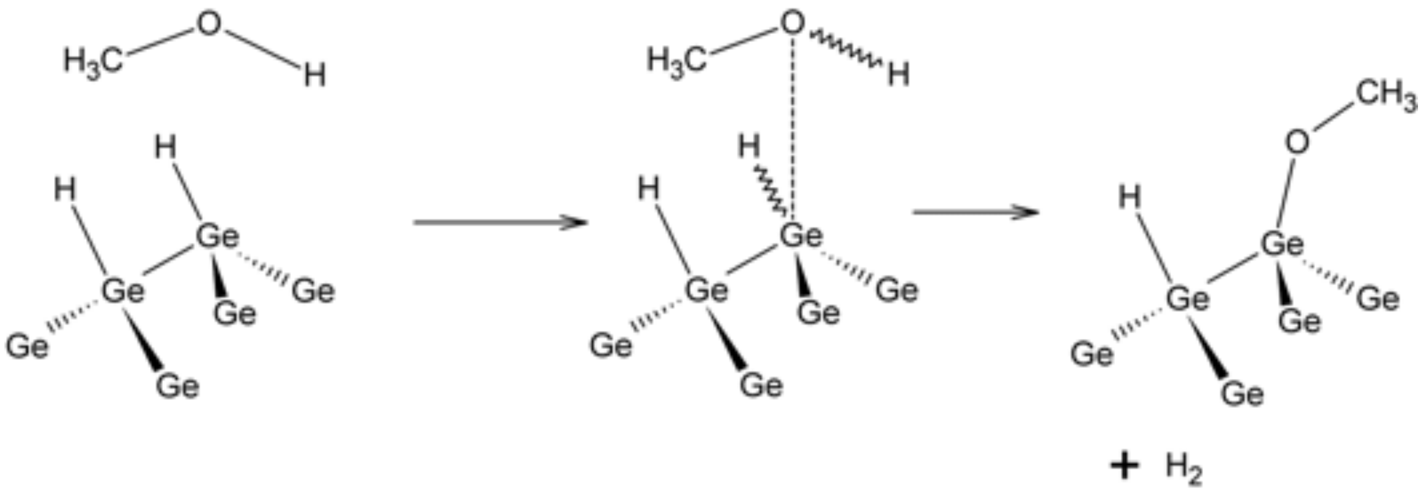
Ideal Passivation → chemical: prevents the surface from reacting with the atmosphere

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

Some used passivations: SiO_2 or amorphous Ge(H)

Passivation techniques

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



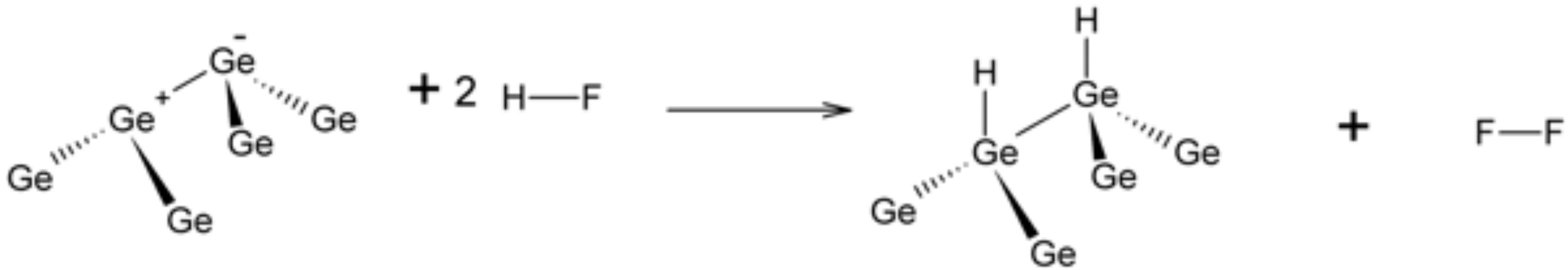
O-H bond dissociation

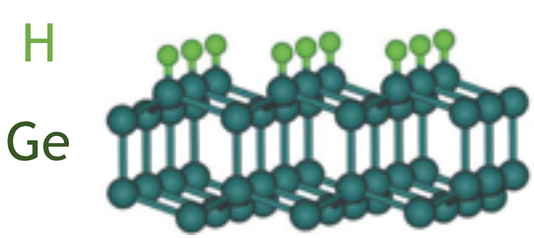
Dissociative adsorption of methanol on the Ge surface

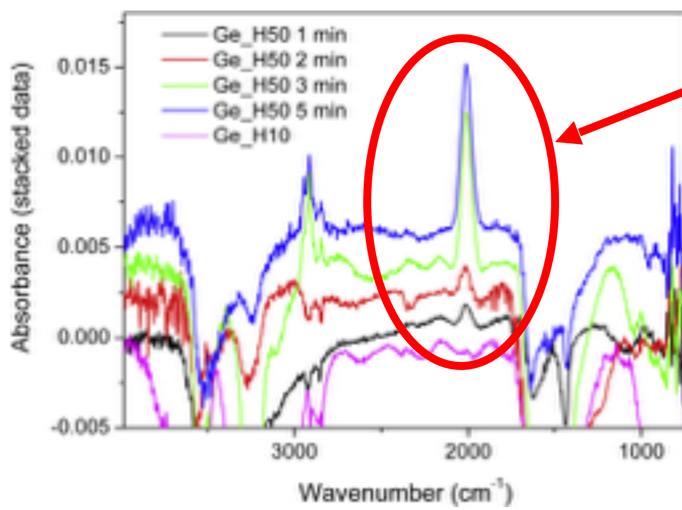
HNO_3/HF etching bath

Passivation techniques

- **Hydride termination - two types:**
  - **Low hydride** (10% HF, 2 min, 5 cycles)
  - **High hydride** (50% HF, 5 min, 1 cycle)







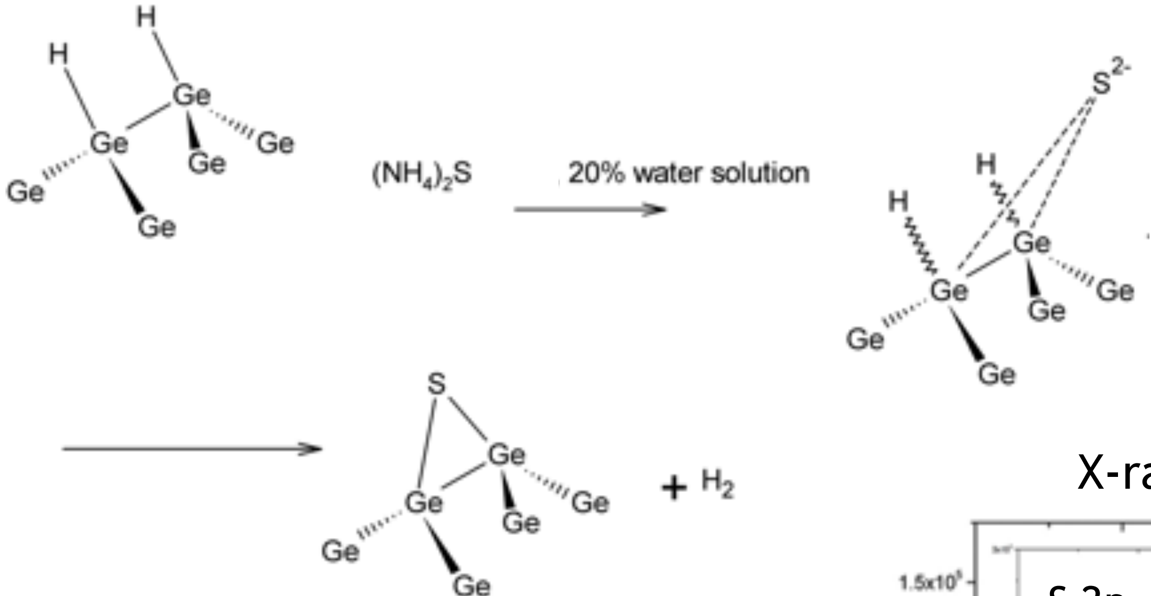
Ge-H peak

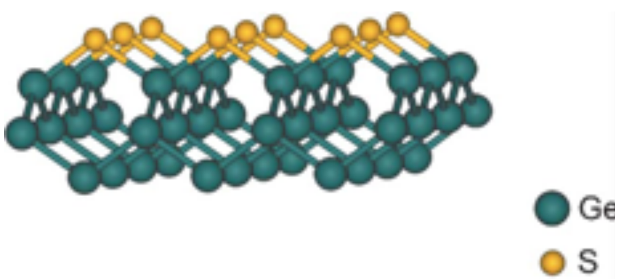
HATR-IR spectra

(from S. Carturan et al., **Mater. Chem. Phys.** 2015)

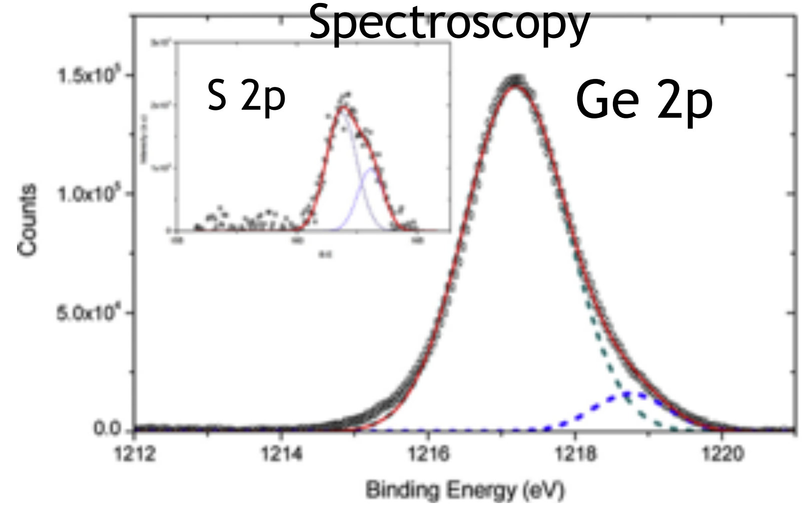
Passivation techniques

- **Sulfide termination** ($(NH_4)_2S$, 20 min, 1 cycle)



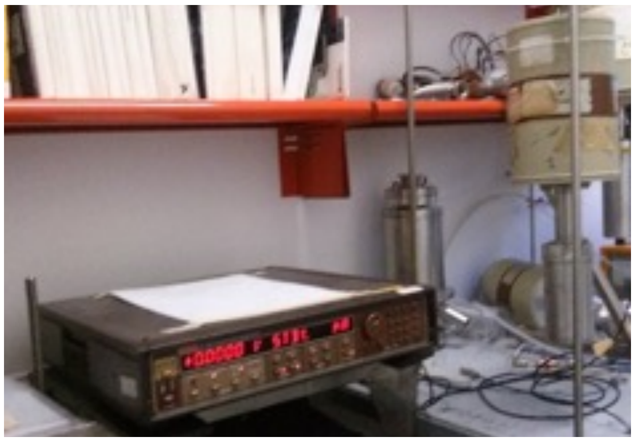


X-ray Photoelectron Spectroscopy

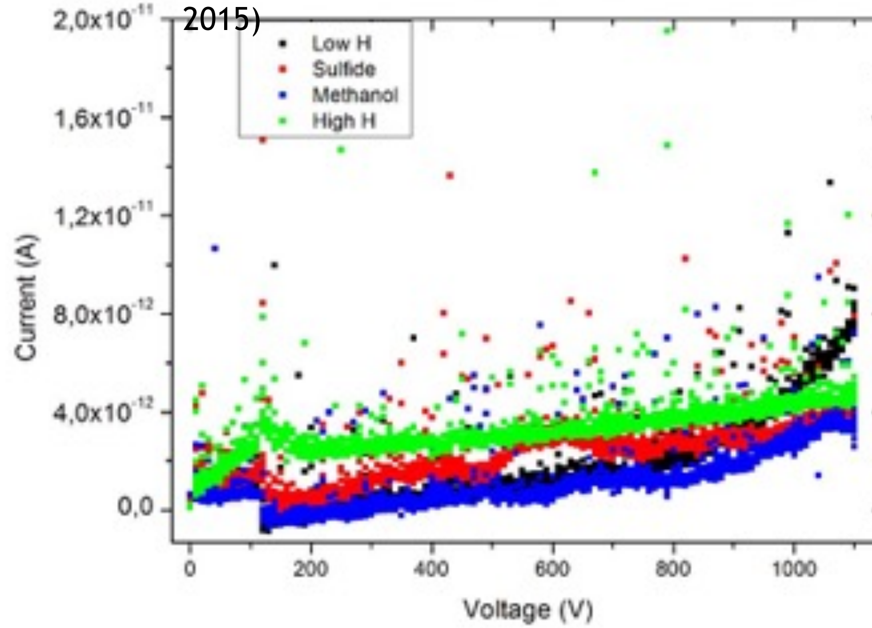


(from S. Carturan et al., **Mater. Chem. Phys.** 2015)

1st test: leakage current of the passivated diode



(from S. Carturan et al., **Mater. Chem. Phys. 2015**)



The reverse leakage current keeps very low in the whole measurement range, even at the highest applied voltage

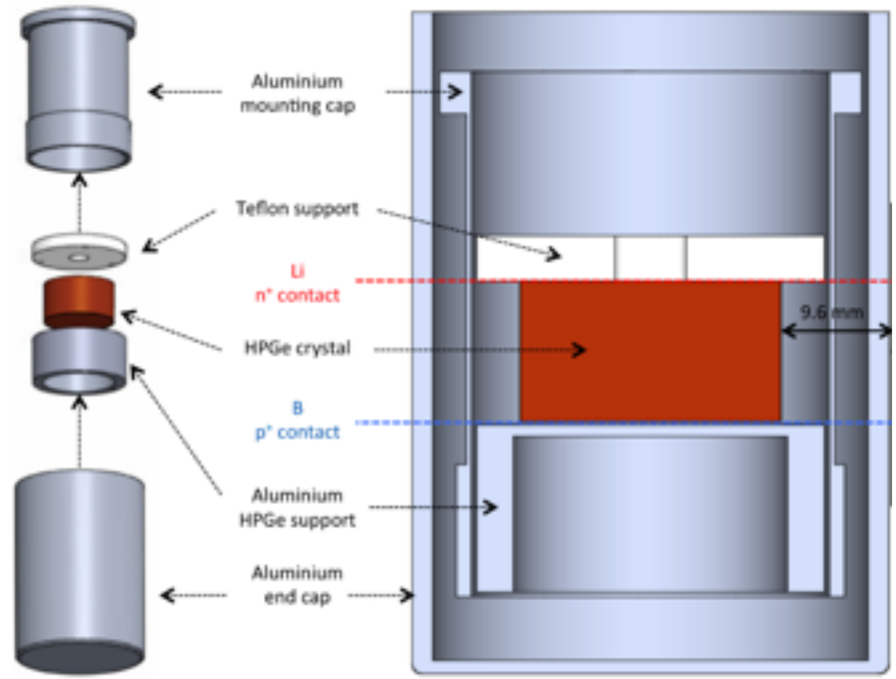
The breakdown voltage of the HPGe diode is well above 1100 V for all the tested passivations

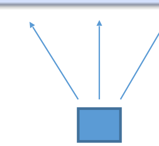
Measurement setup

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

"Bulk" properties:

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





Gamma-ray source

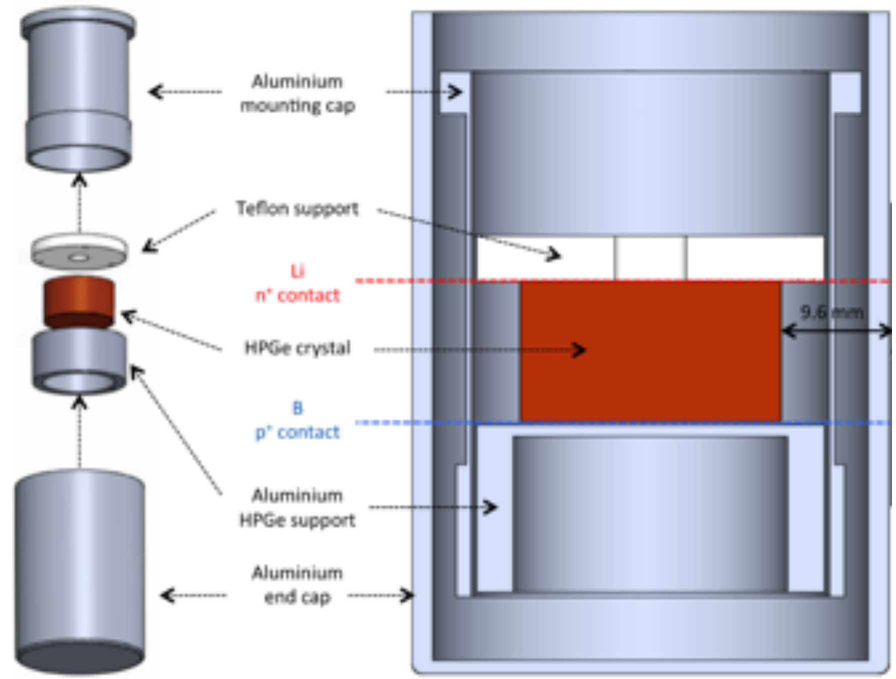
A standard electronic NIM was used with: in front-end a standard Ortec **preamplifier** (model n° 137), in backend and data acquisition a Silena **amplifier** (model n° 7611/L) with 3 μs shaping time, a Silena HV **power supply** (model n° 7716/LN) and standard Ortec Easy-**MCA**.

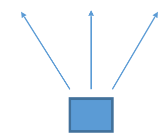
Measurement setup

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

"Bulk" properties:

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





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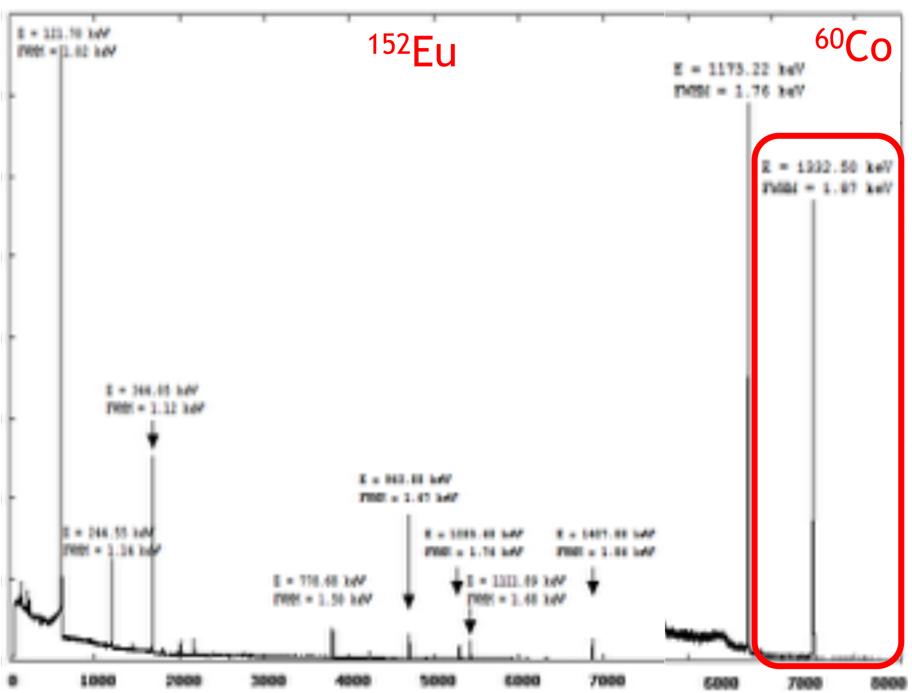
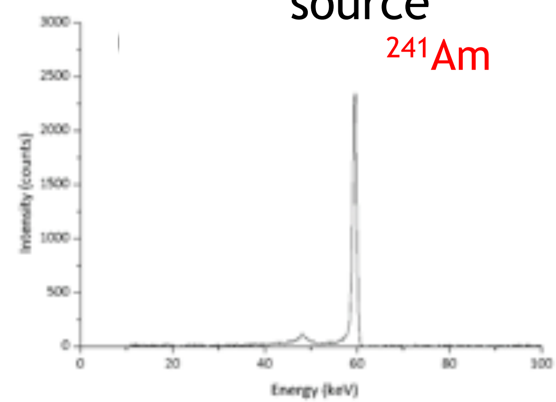
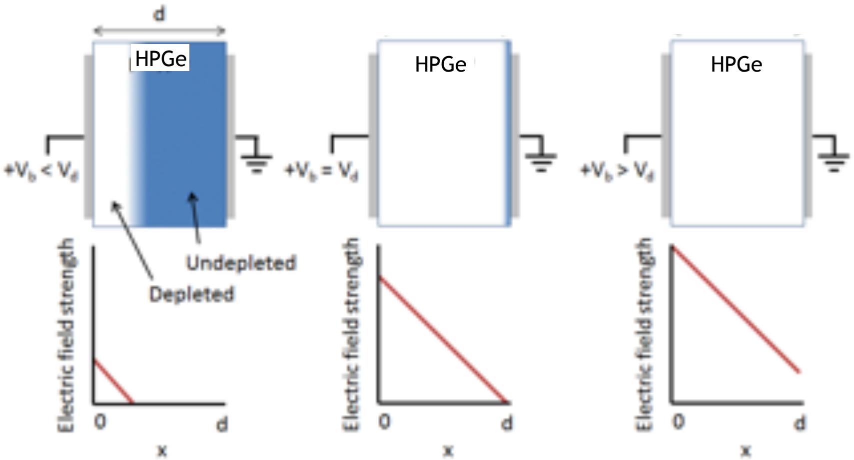




Depletion voltage



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

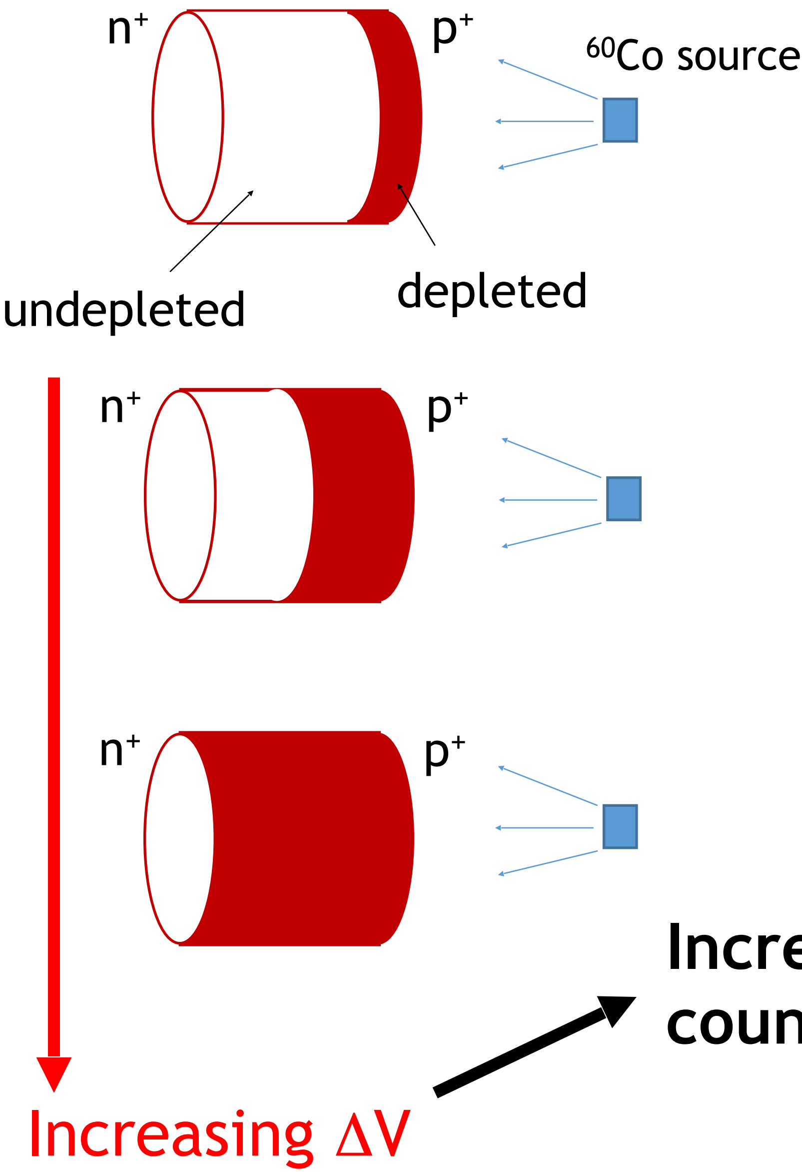
If surface effects can be neglected

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

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

Depletion voltage

How did we measure it?



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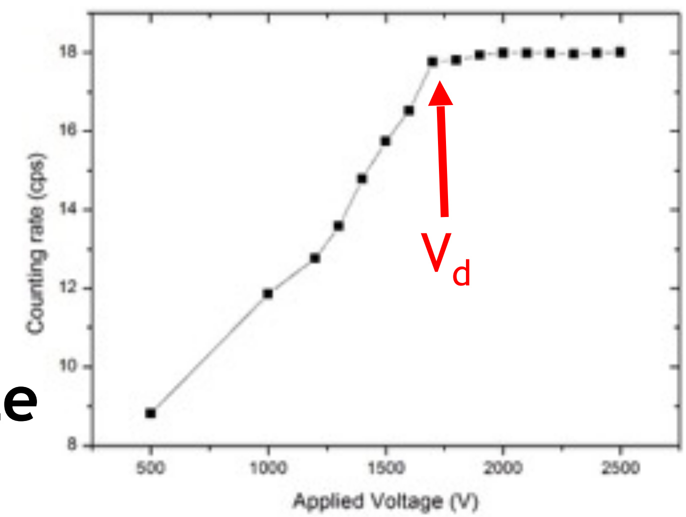
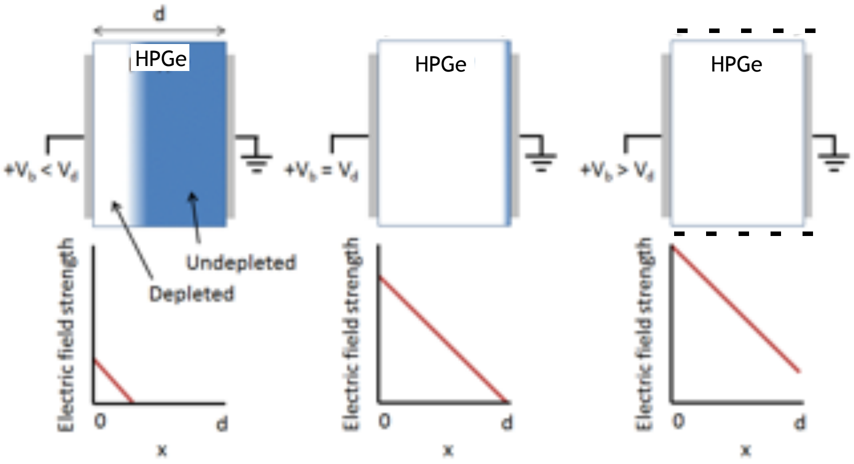
Increase of counting rate



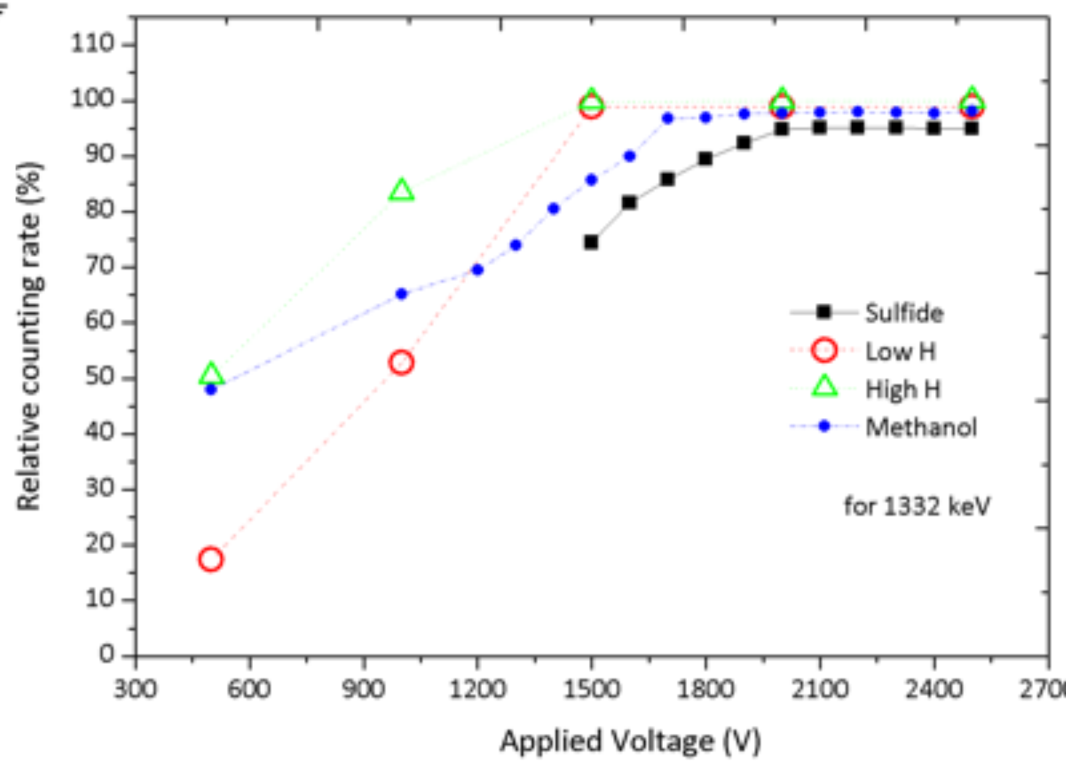
Depletion voltage



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

Experimental results

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



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%)

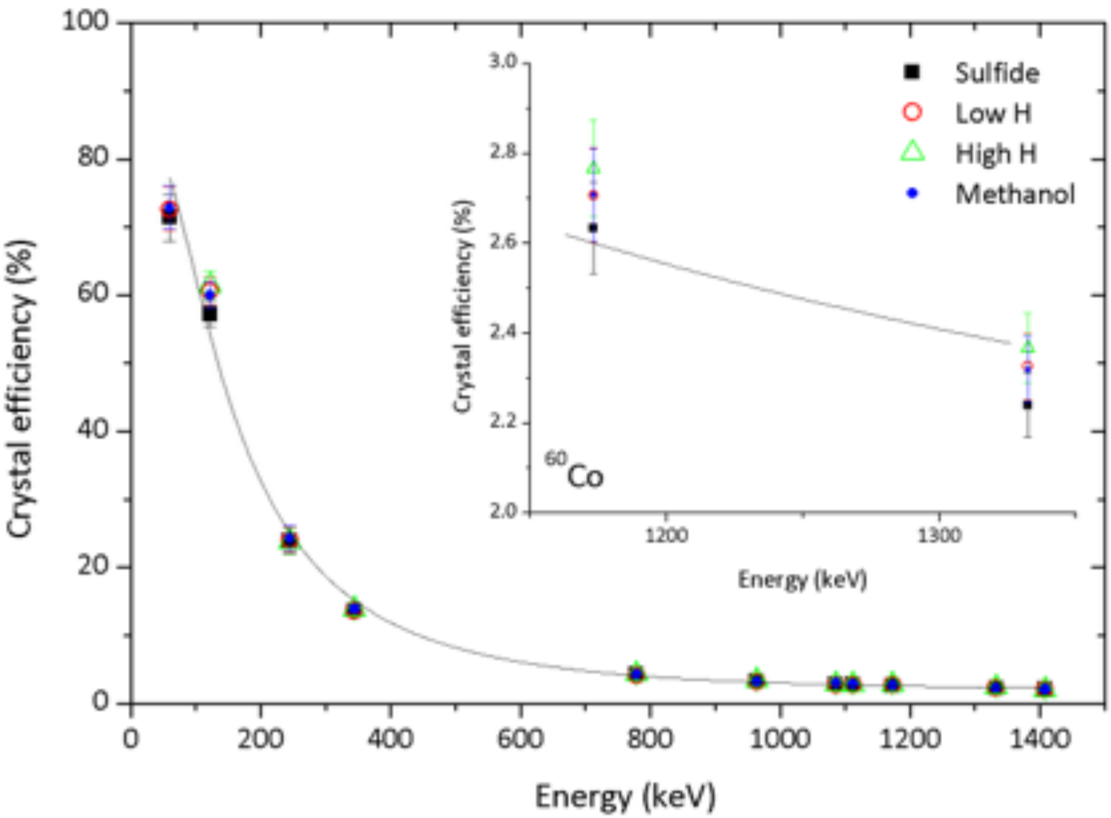
Detector efficiency

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

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

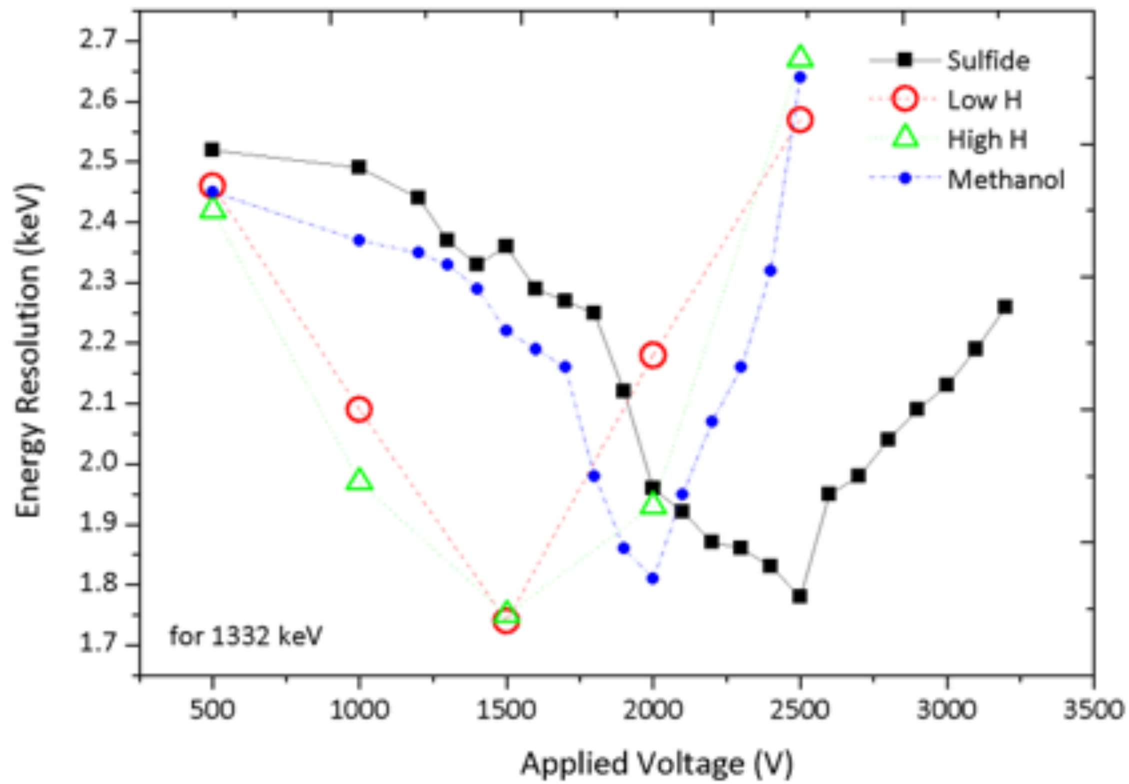
Detector resolution (FWHM) vs bias voltage

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

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



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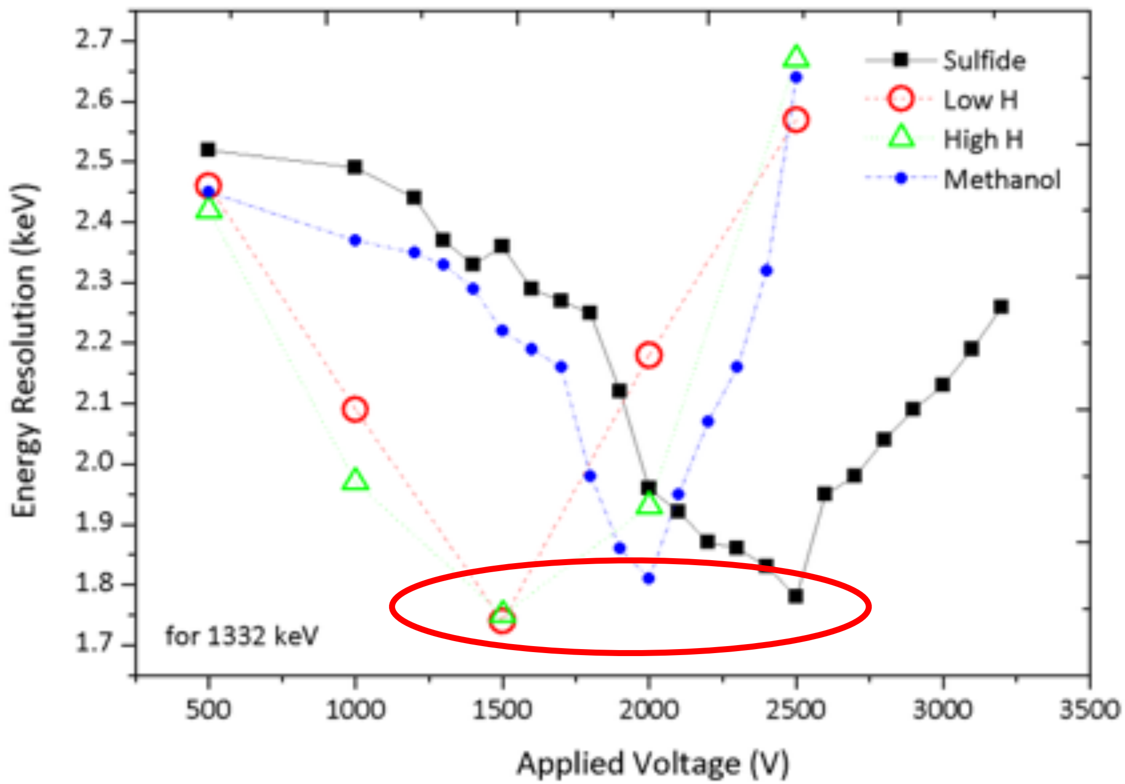


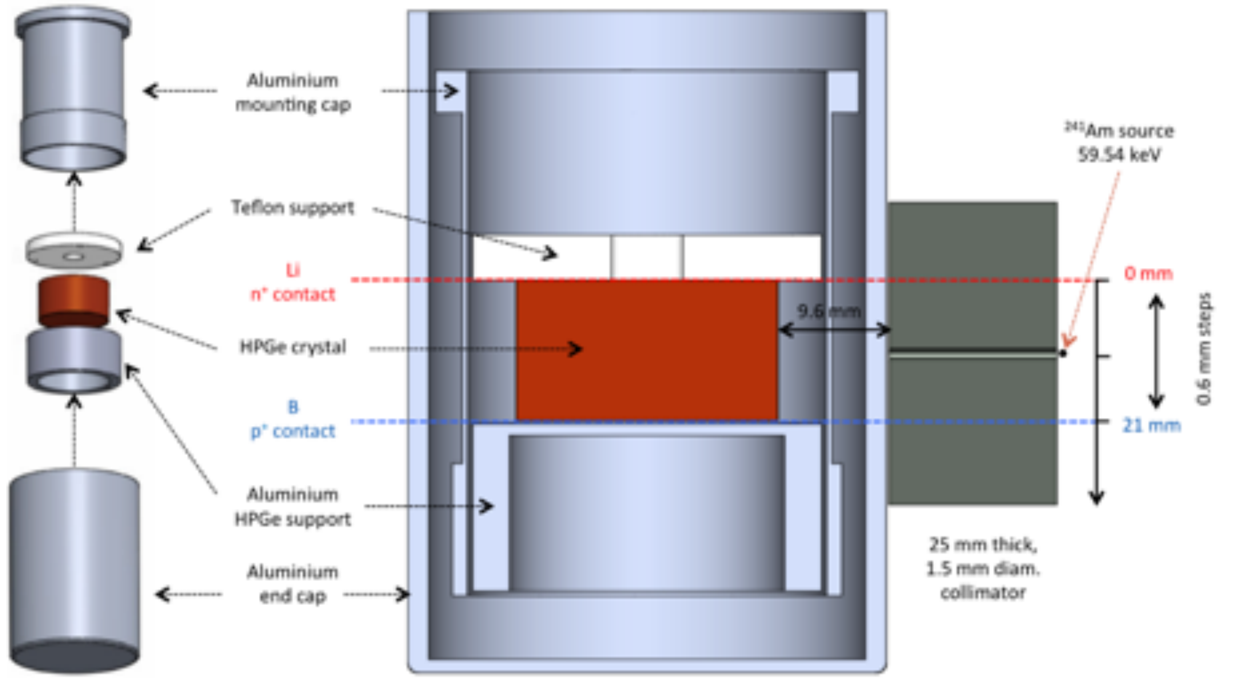
	Voltage for best resolution (V)
Low H	1500
High H	1500
Methanol	2000
Sulfide	2500

The best resolution is comparable for all the passivations

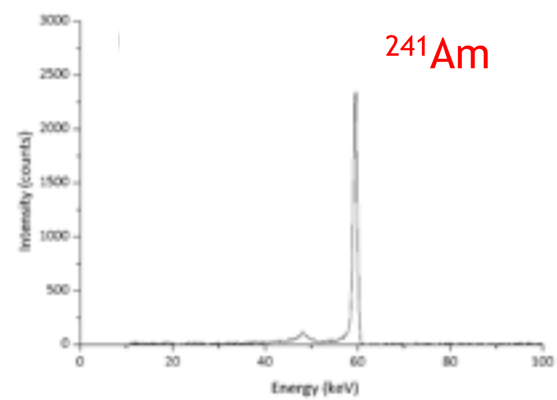
Lateral scan: setup configuration

Collimated Source: ^{241}Am

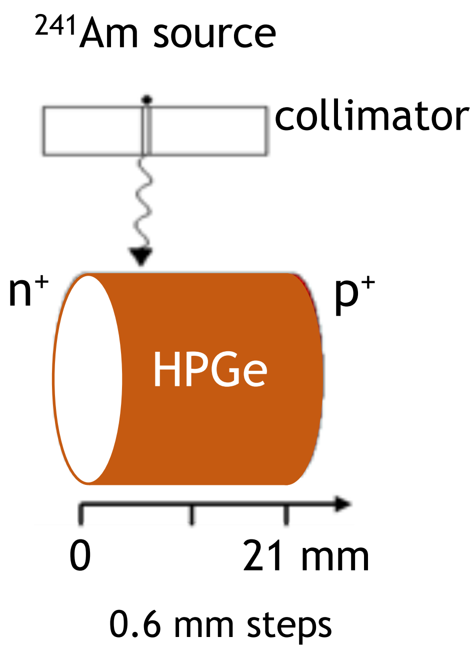
Lateral scans (passivated surface)



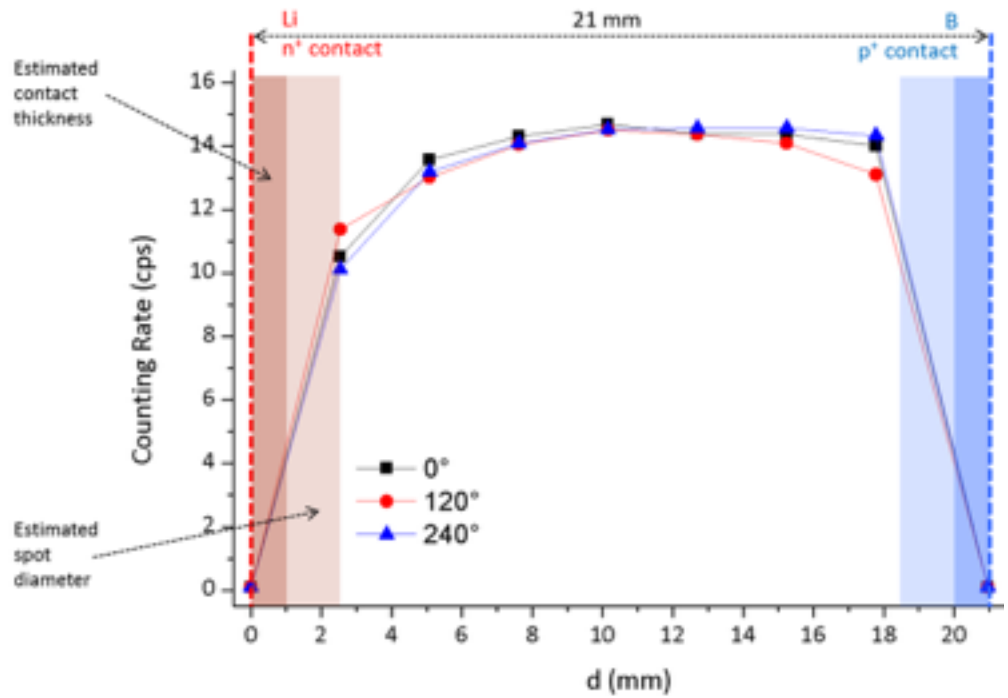
Collimated ^{241}Am Source (23.9 Bq)



Scan of the passivated surface: angular position



High Hydride passivated



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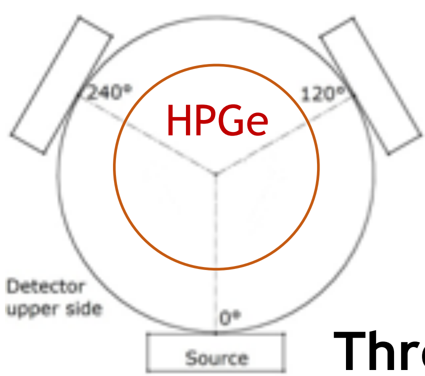
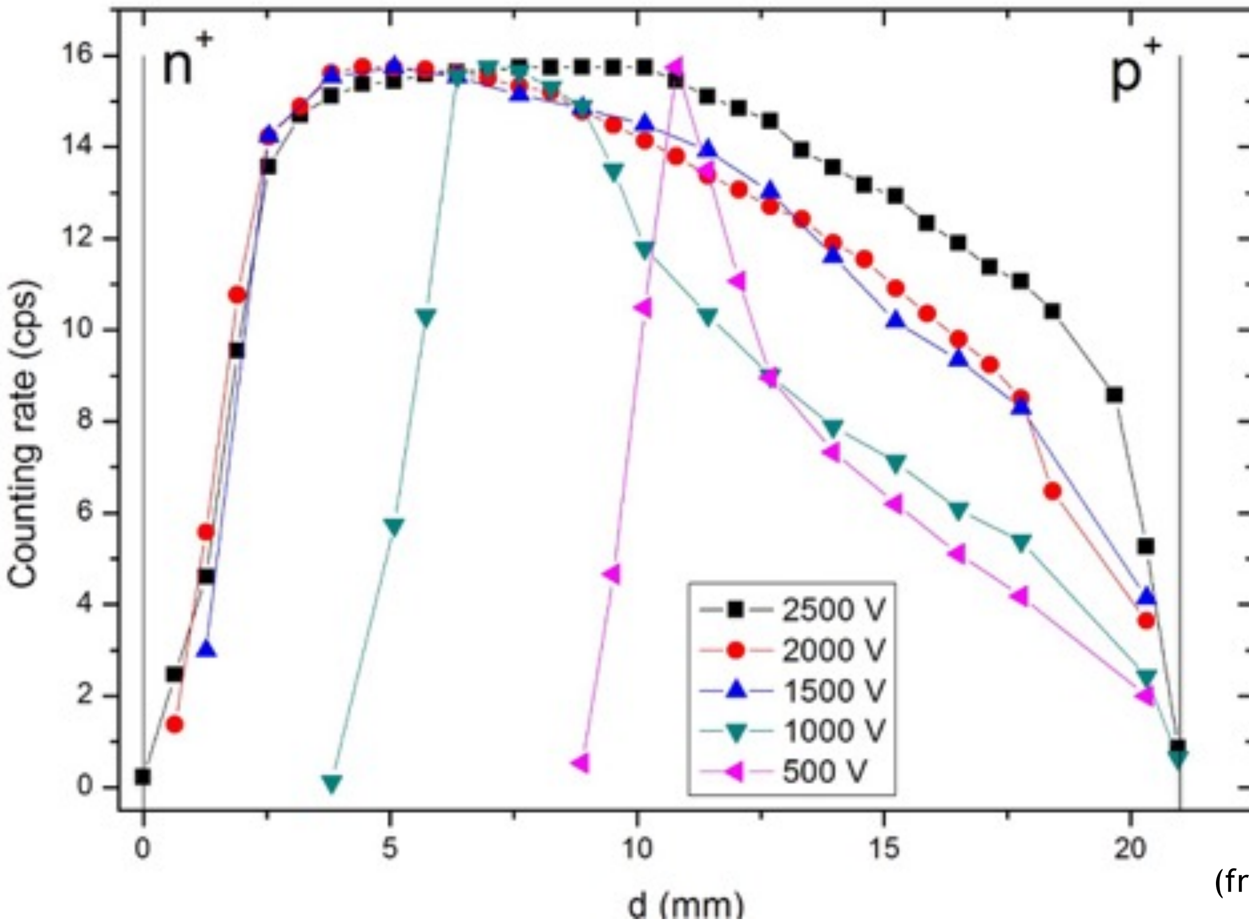


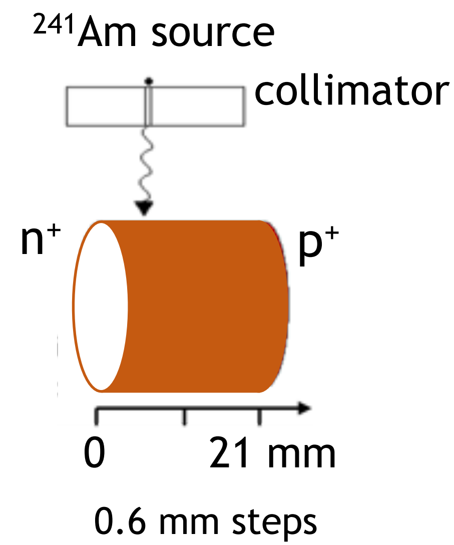
Three different angular positions

Good lateral homogeneity of the passivation treatment

Scan of the passivated surface: counting rate vs bias voltage

Methanol passivated detector

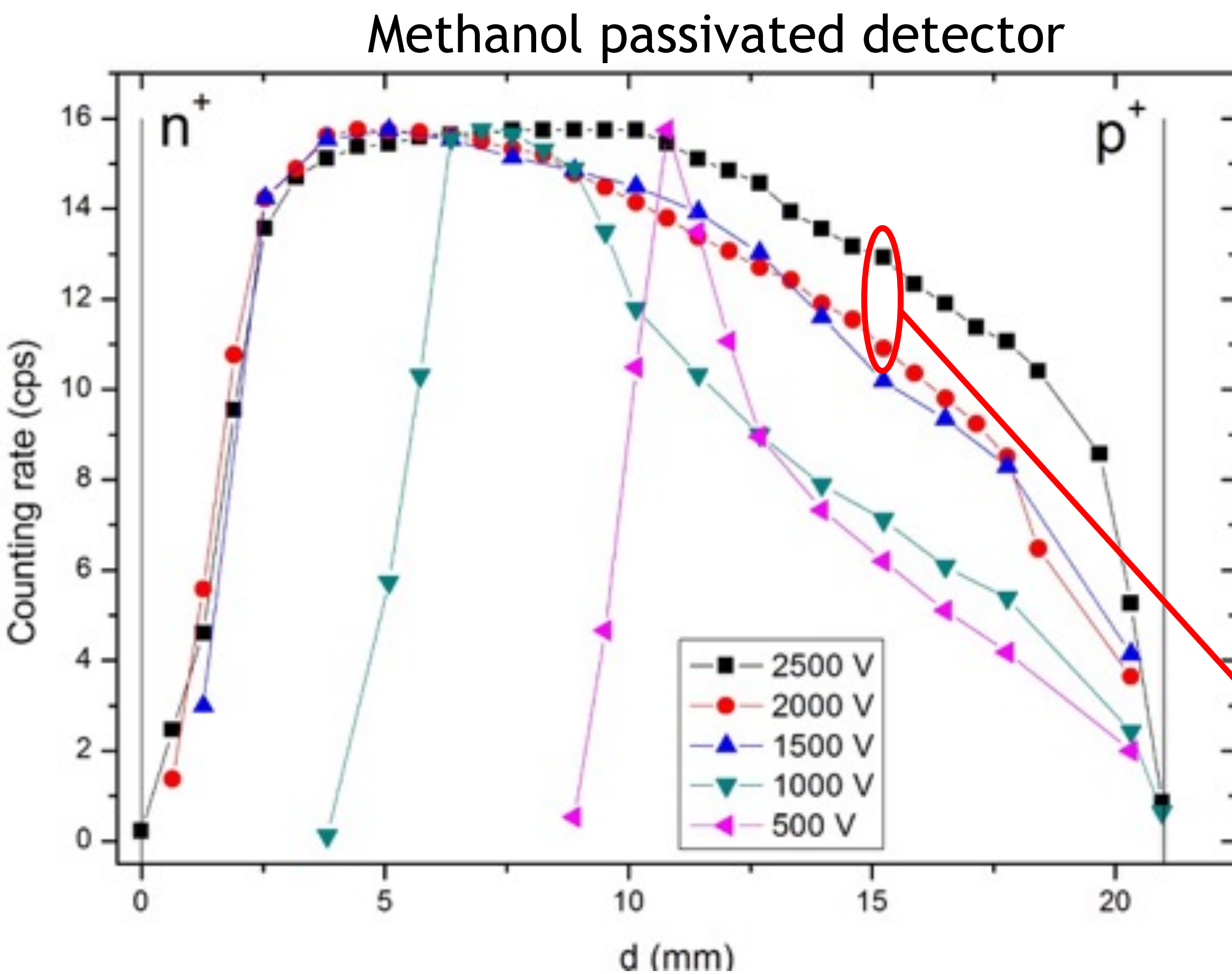


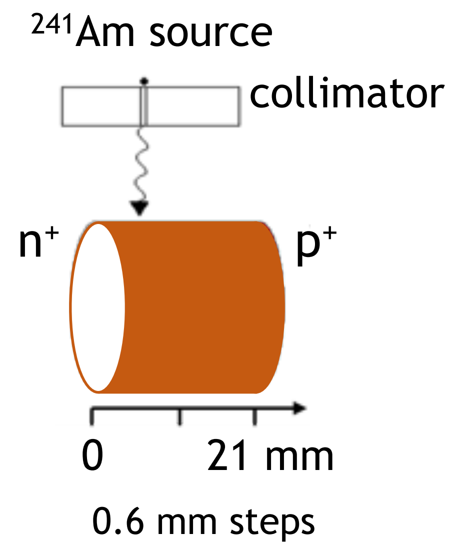


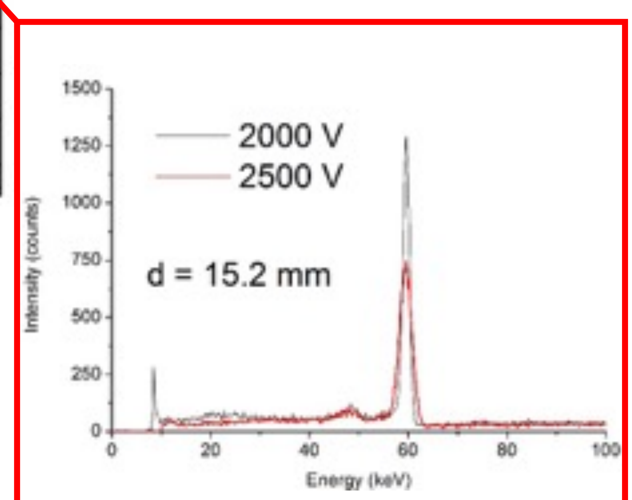
(from G. Maggioni et al., **EPJA** 2015)

- Counting rate depends on the bias voltage
- In the region close to the p+ contact, the counting rate increases but to the detriment of energy resolution

Scan of the passivated surface: counting rate vs bias voltage

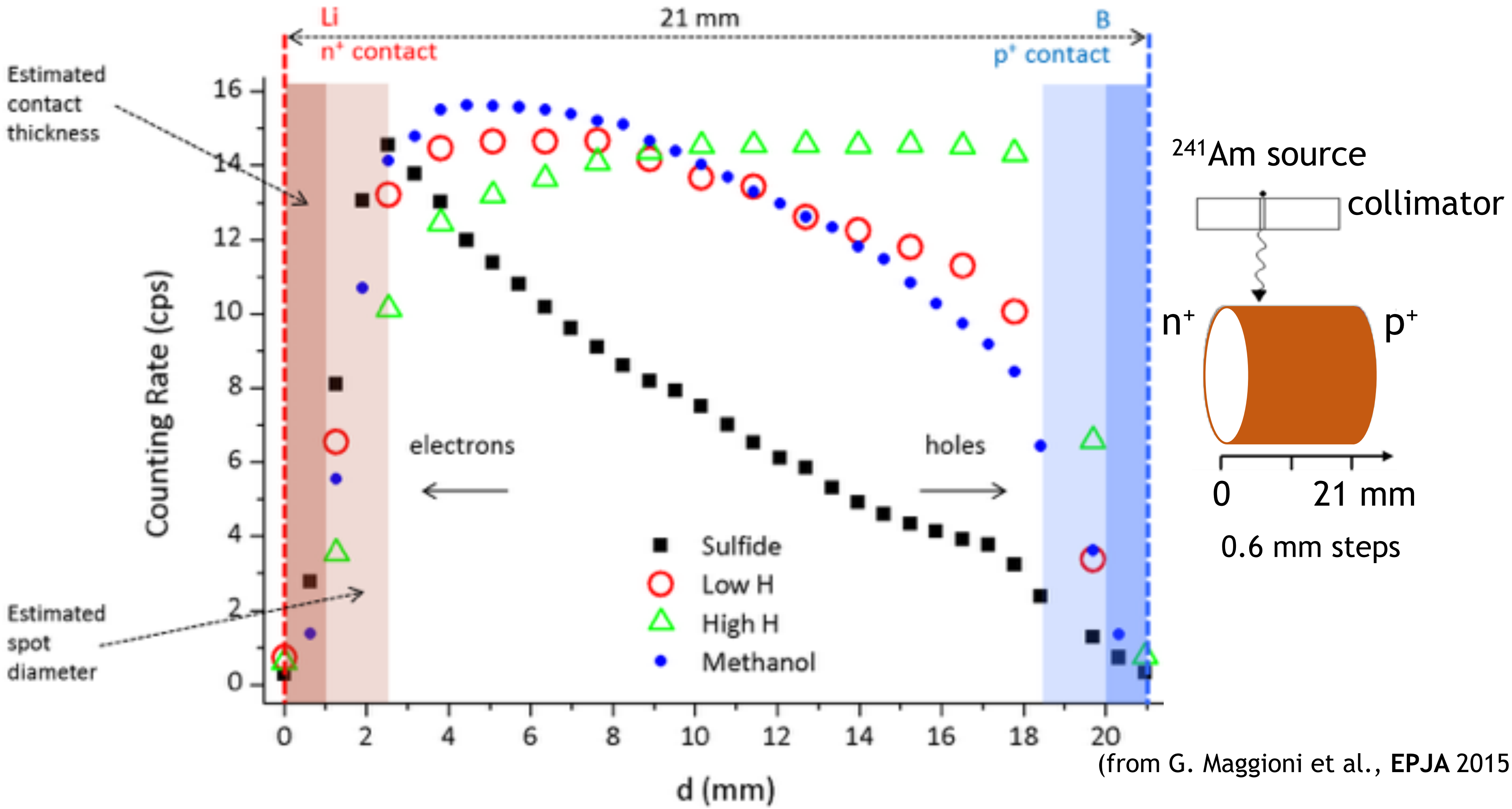






- Counting rate depends on the bias voltage
- In the region close to the p+ contact, the counting rate increases but to the detriment of energy resolution

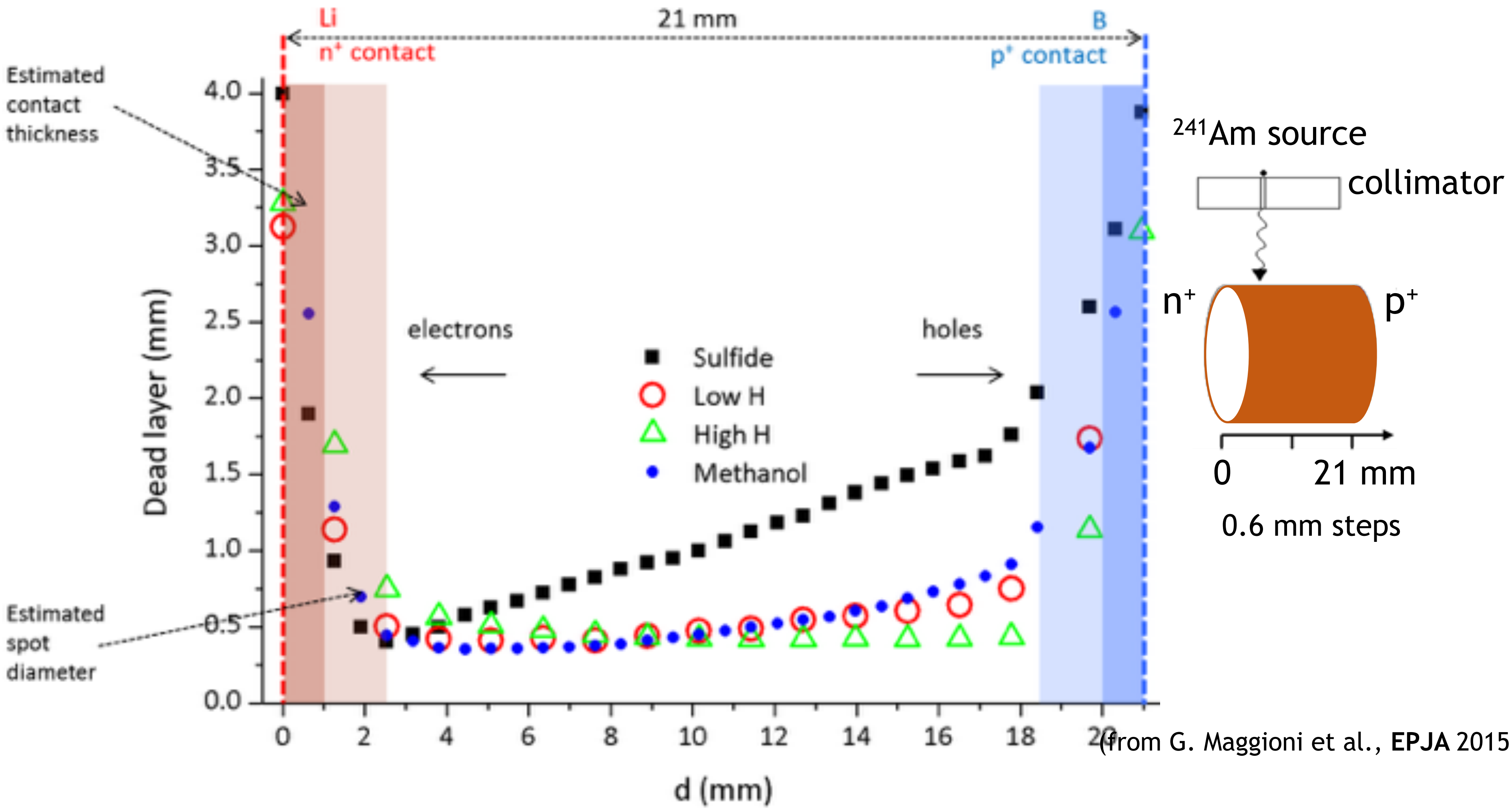
Scan of the passivated surface: counting rate vs passivation



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

- 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

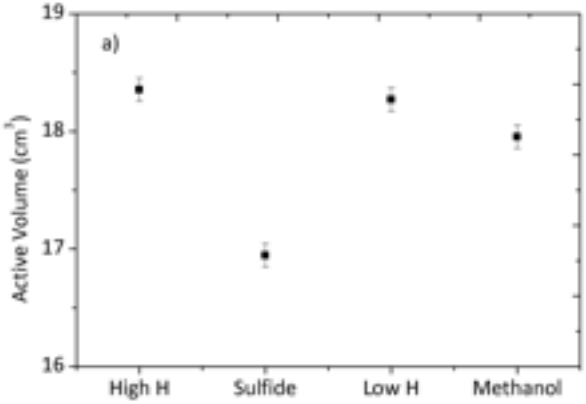


(from G. Maggioni et al., **EPJA** 2015)

- 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 is close to 10% in the case of the S-terminated detector

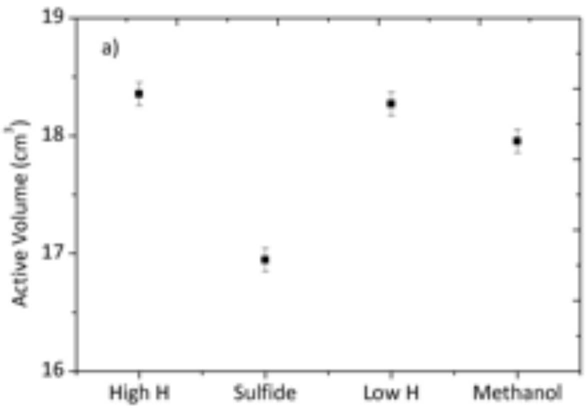
Correlation between active volume, counting rate plateau and efficiency

Active volume

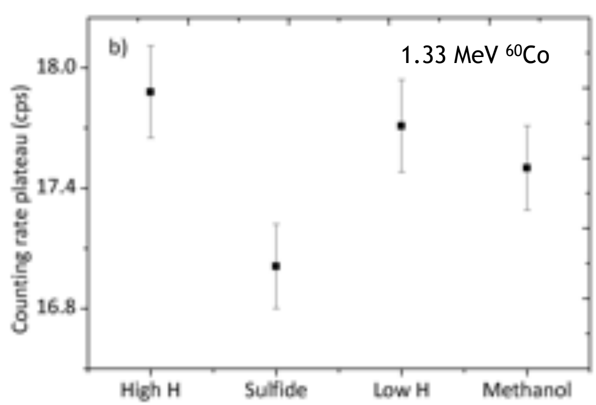


Correlation between active volume, counting rate plateau and efficiency

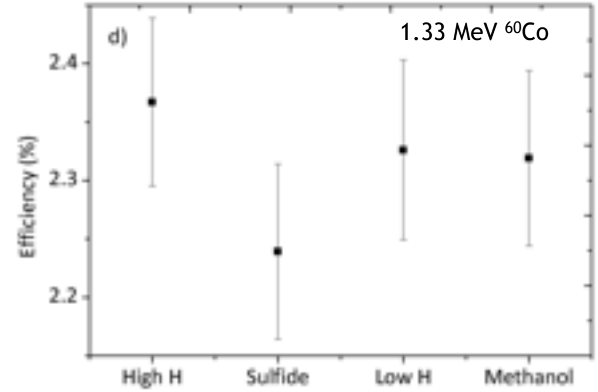
Active volume



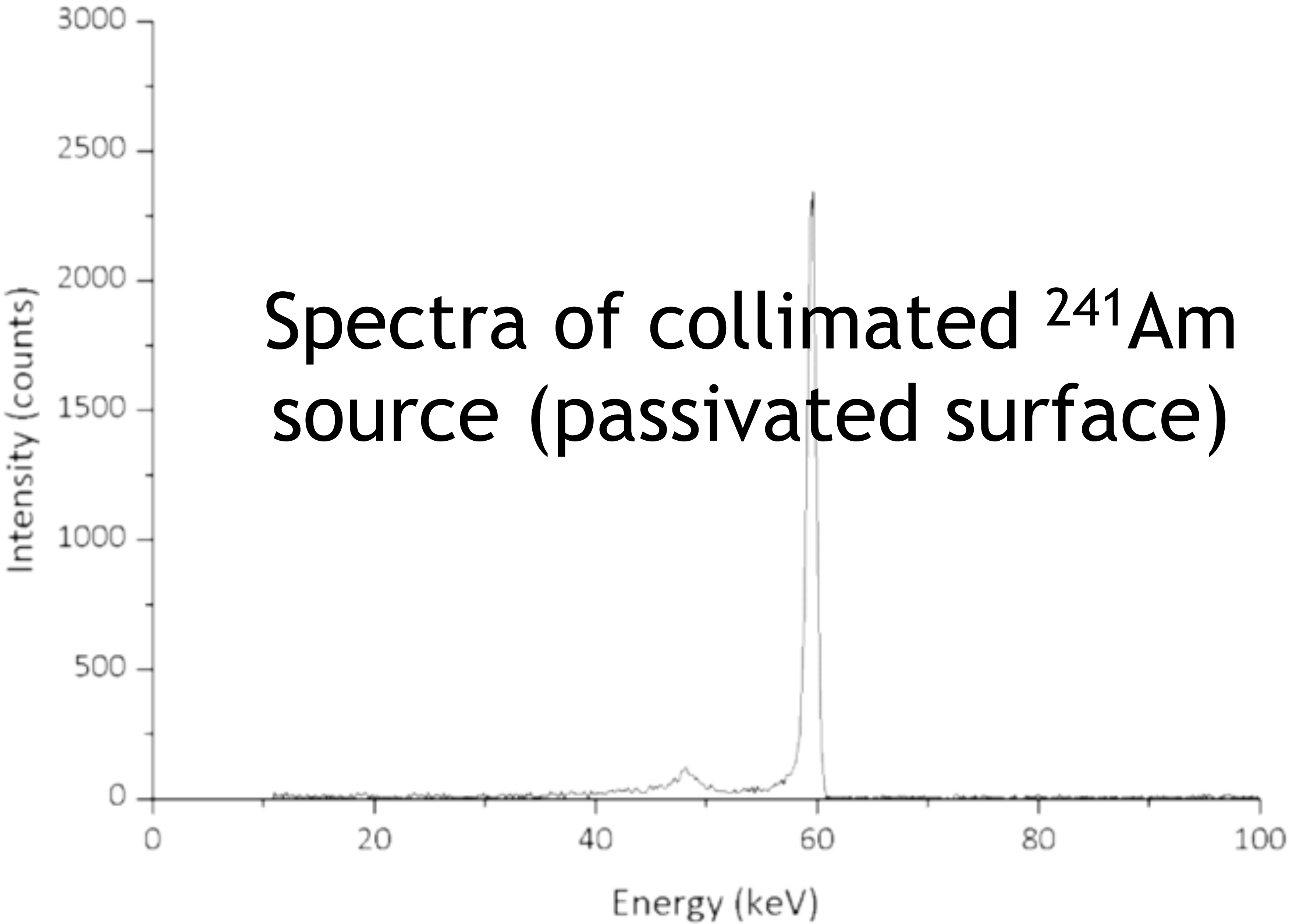
Counting rate plateau



Efficiency

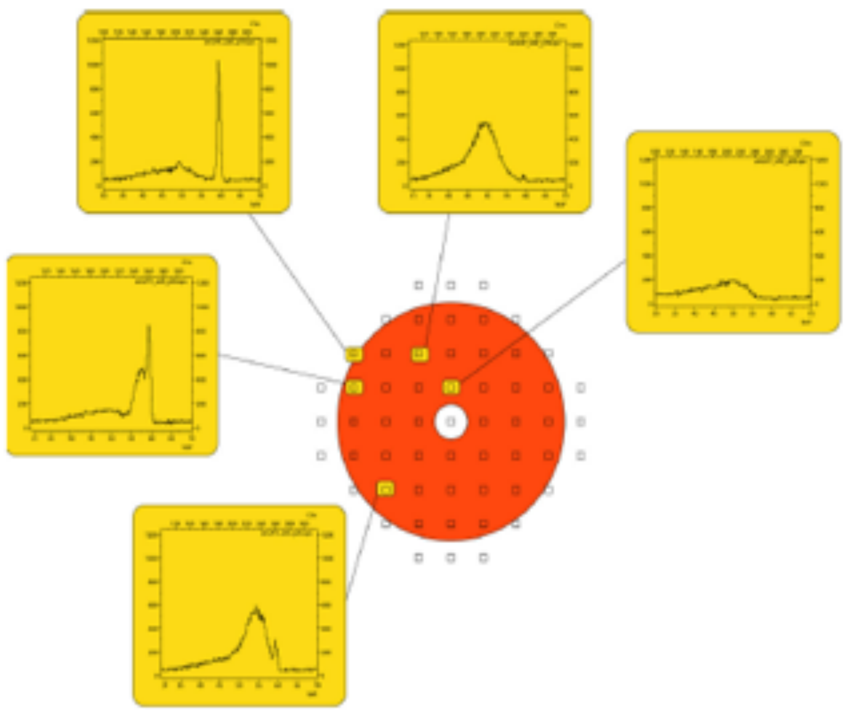


Clear correlation

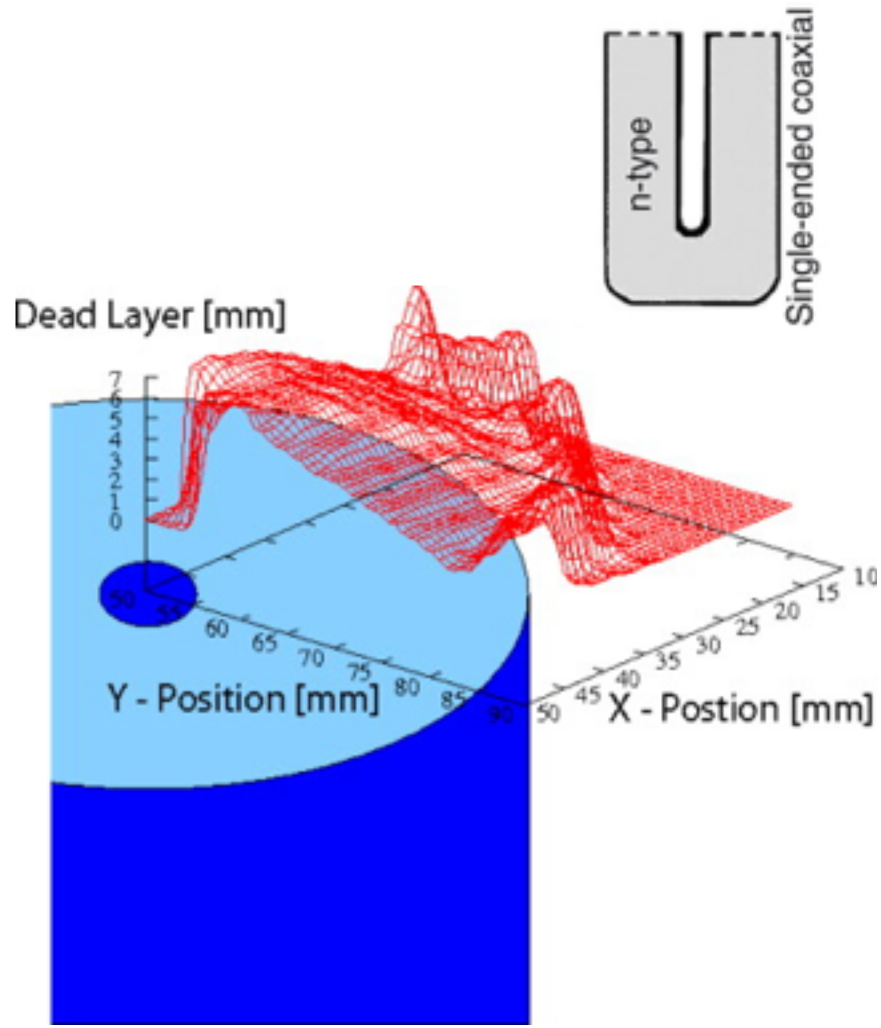


Spectra of collimated ^{241}Am source (passivated surface)

241 Am spectra for a commercial detector close to the passivated surface

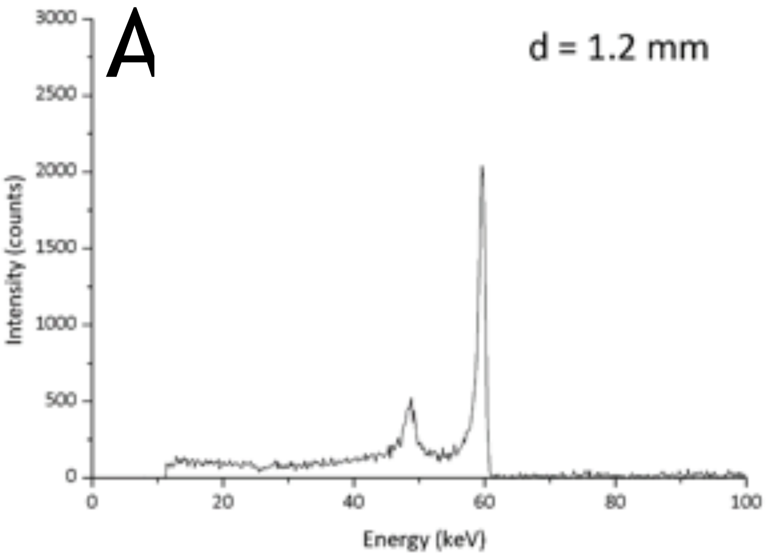


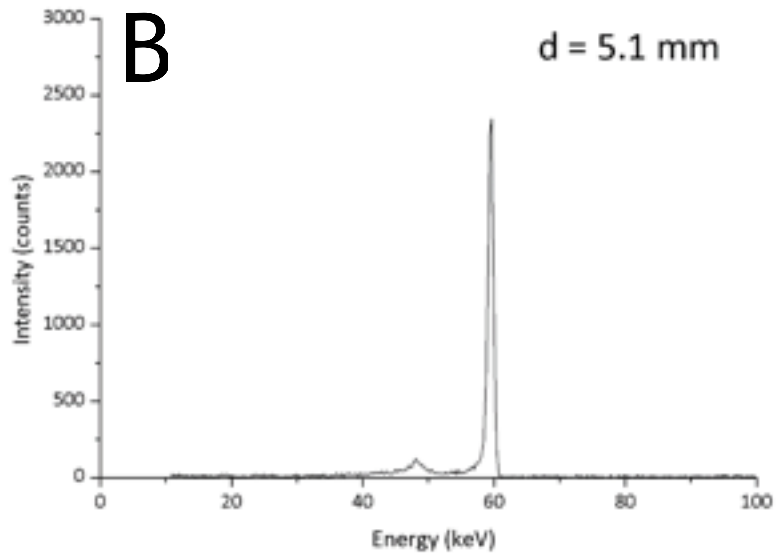
Distortion of ^{241}Am spectra

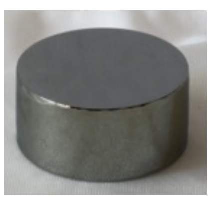


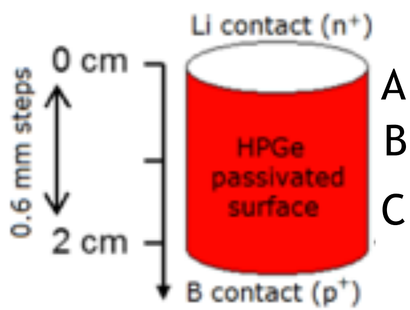
D.Weisshaar (private comm.) in Eberth & Simpson, **Progr.Part.Nucl.Phys.**2008

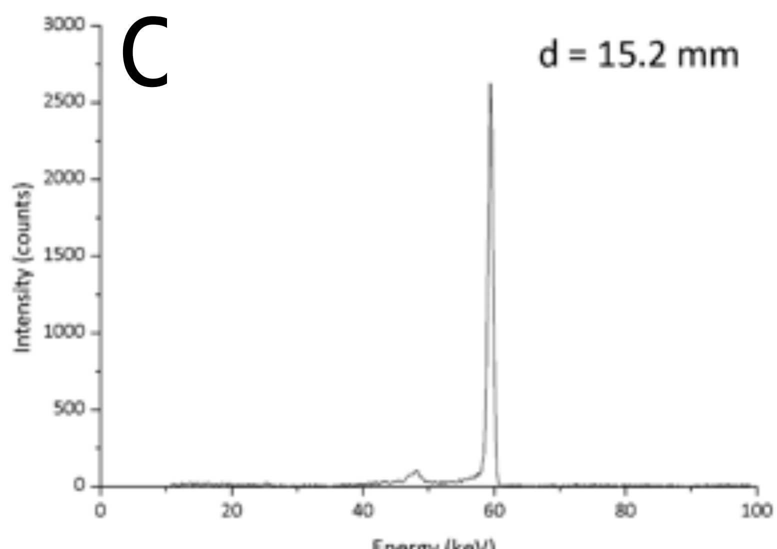
High H-passivated detector: ^{241}Am spectra





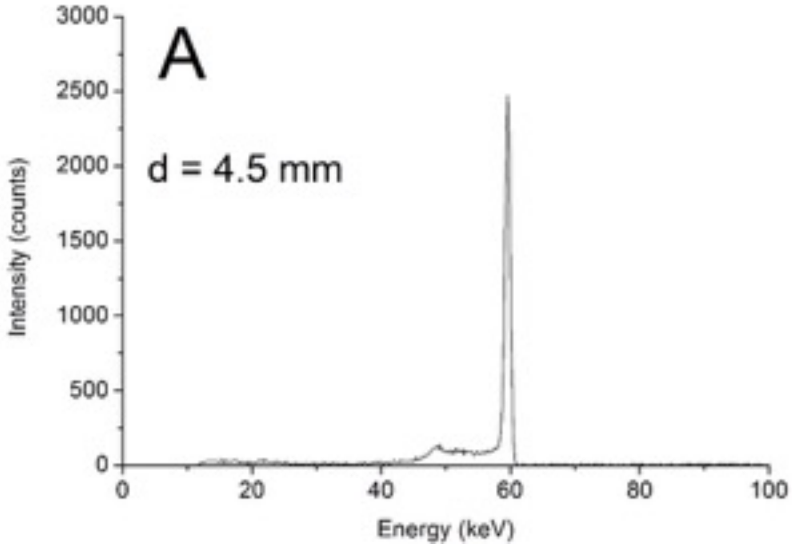


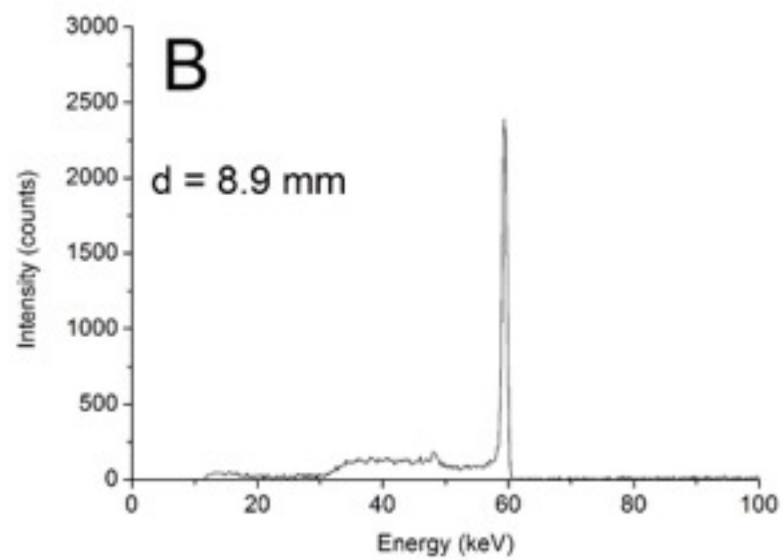


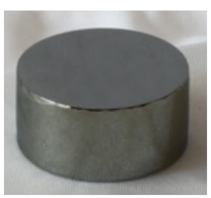


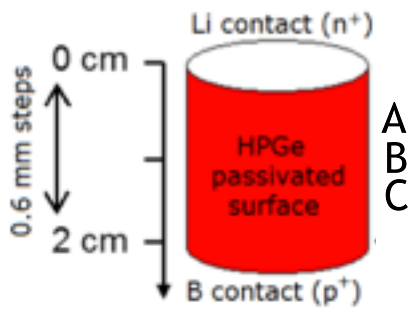
Constant area of the photopeak

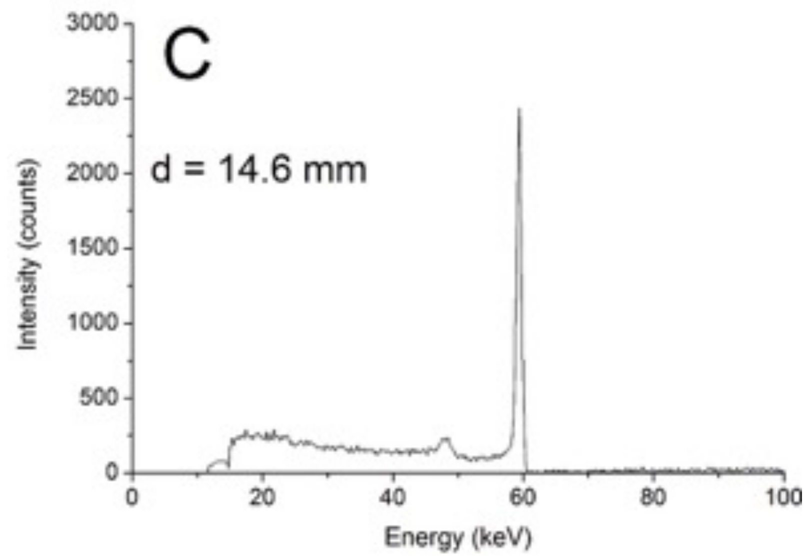
Sulfide passivated detector: ^{241}Am spectra











Constant area of the photopeak

Development of new passivation techniques: Surface passivation by GeN_x and GeO_xN_y films

GeN_x and GeO_xN_y films are studied as passivation layers for microelectronic applications

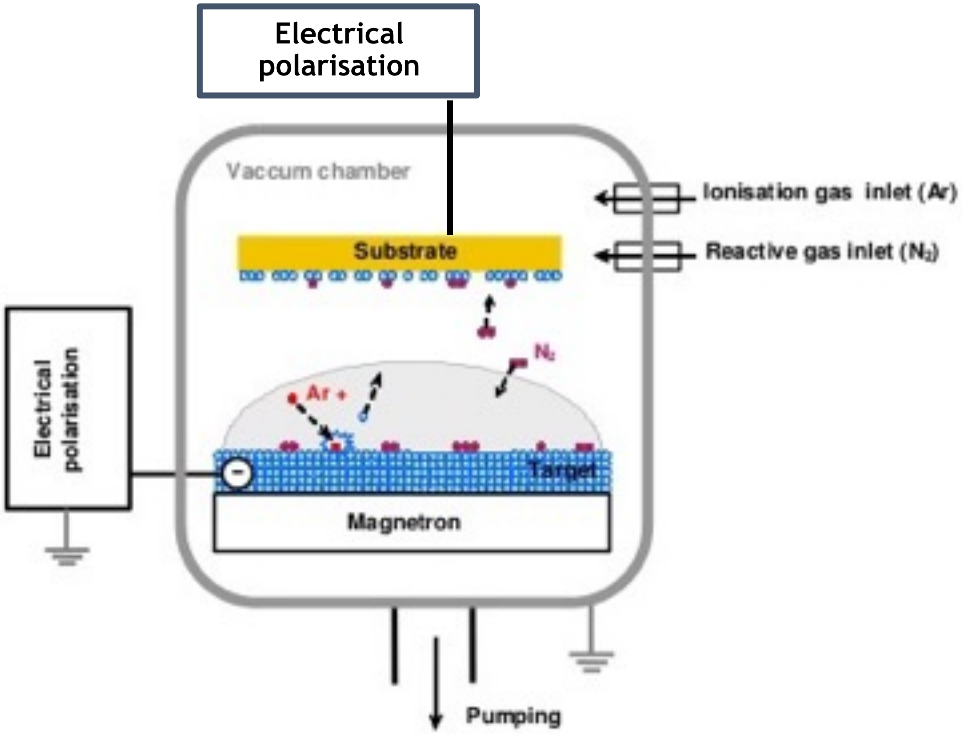
IEEE TRANSACTIONS ON ELECTRON DEVICES, VOL. 53, NO. 7, JULY 2006

Nanoscale Germanium MOS Dielectrics—Part I: Germanium Oxynitrides

Chi On Chui, *Member, IEEE*, Fumitoshi Ito, *Member, IEEE*, and Krishna C. Saraswat, *Fellow, IEEE*

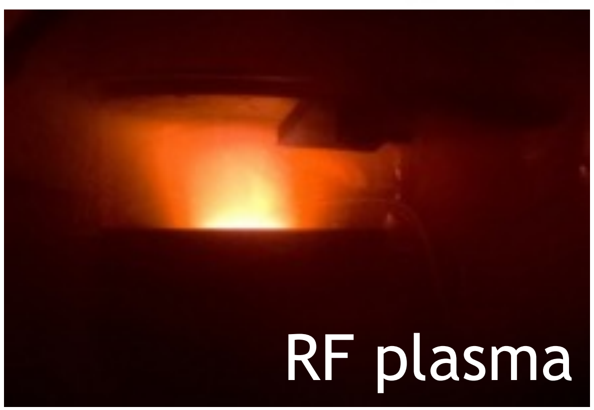
- Ge_3N_4 is a high-k material, is water-insoluble and the thermal decomposition temperature is higher than GeO_2
- Ge oxynitrides (GeO_xN_y) are also very interesting because N incorporation in Ge oxides improves chemical and thermal stability
- Several techniques can be used for the deposition of these materials, but these usually require heating the substrates at high temperatures, which are not compatible with the passivation of HPGe crystals for gamma detectors
- We explored a room temperature deposition technique: RF reactive

Sputtering deposition setup

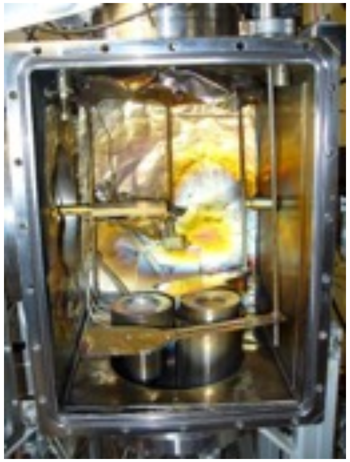


Experimental parameters

- RF reactive magnetron sputtering
- Ge target
- N_2 and N_2/Ar atmospheres
- Low RF power: 40 to 60 W
- **Room temperature deposition**
- Substrate bias: 0 to -100 V

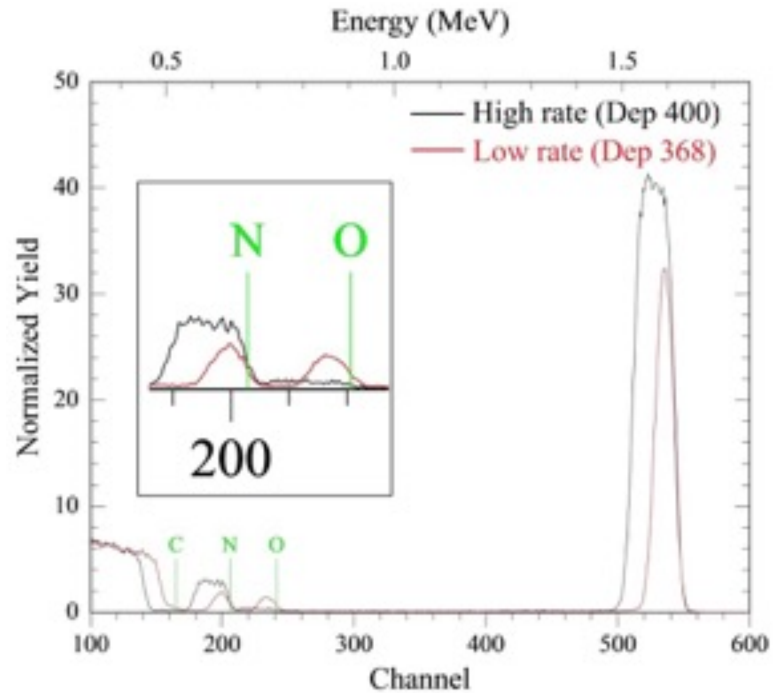


You can change the film properties by changing the deposition parameters



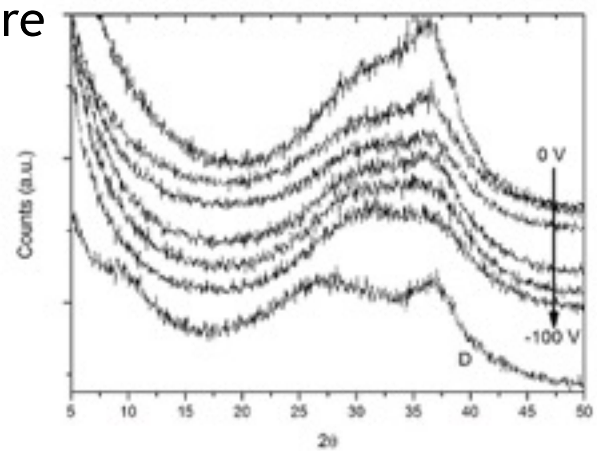
Film composition and structure: RBS and XRD results

Film composition is strongly affected by:
- Deposition rate
- Atmosphere composition
- Ion bombardment of the growing film



RBS

Effects of ion bombardment of the growing film on:
- Deposition rate
- Film microstructure



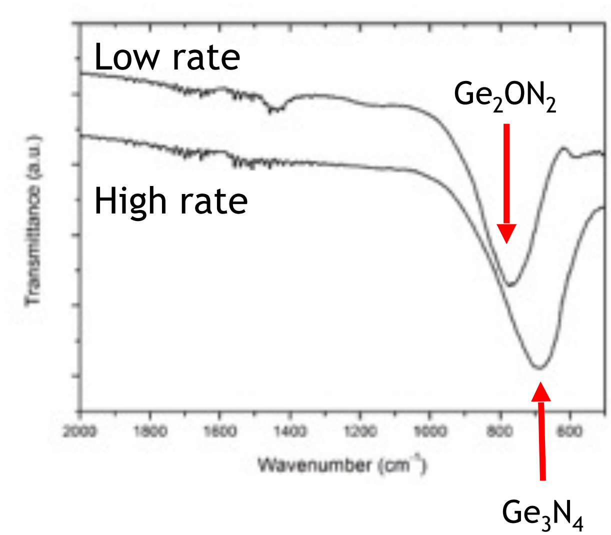
XRD

Very poor crystallization

(from G. Maggioni et al., **Appl. Surf. Sci.** 2016, *under review)*

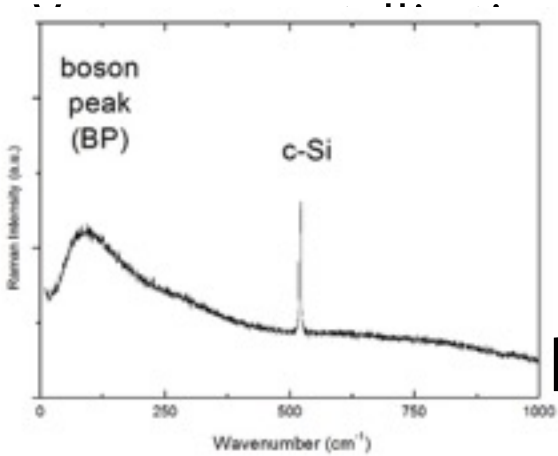
Film composition, structure and morphology: FT-IR, Raman and AFM results

Film composition:
- Germanium nitride (Ge_3N_4) → High rate
- Germanium oxynitride (Ge_2ON_2) → Low rate



FT-IR

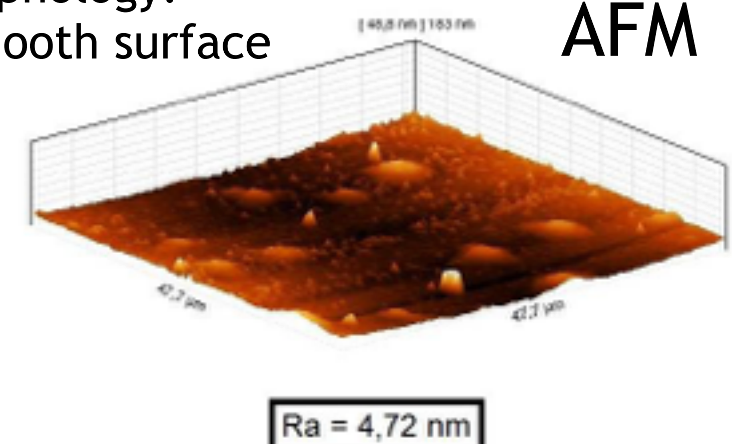
Film structure:
-



Raman

Film morphology:
- Very smooth surface

AFM



(from G. Maggioni et al., **Appl. Surf. Sci.** 2016, *under review)*

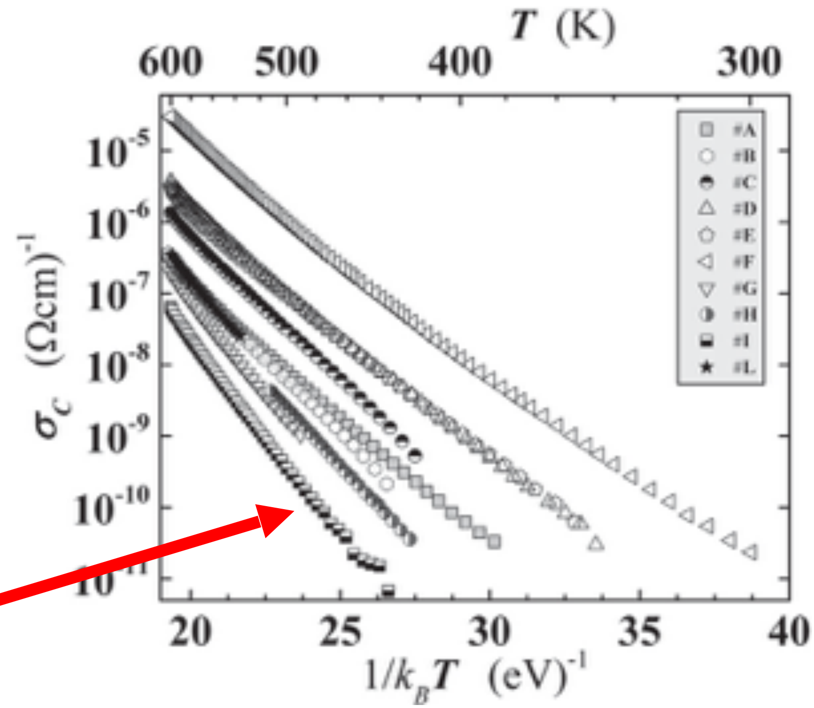
Electrical conductivity of the films

The electrical conductivity of the films was measured as a function of temperature.

All the films are highly insulating at room temperature and the conductivity is related to the deposition parameters

The electrical properties of the films can be controlled by changing the deposition parameters

The conductivity of Ge oxynitride film is the lowest one



(from N. Pinto et al., **J. Non-Cryst. Solids** 2016)

The conduction mechanism is thermally activated: activation energy ranges from 0.6 to 1.4 eV

à Charge carrier transitions involving localized states inside the energy-gap

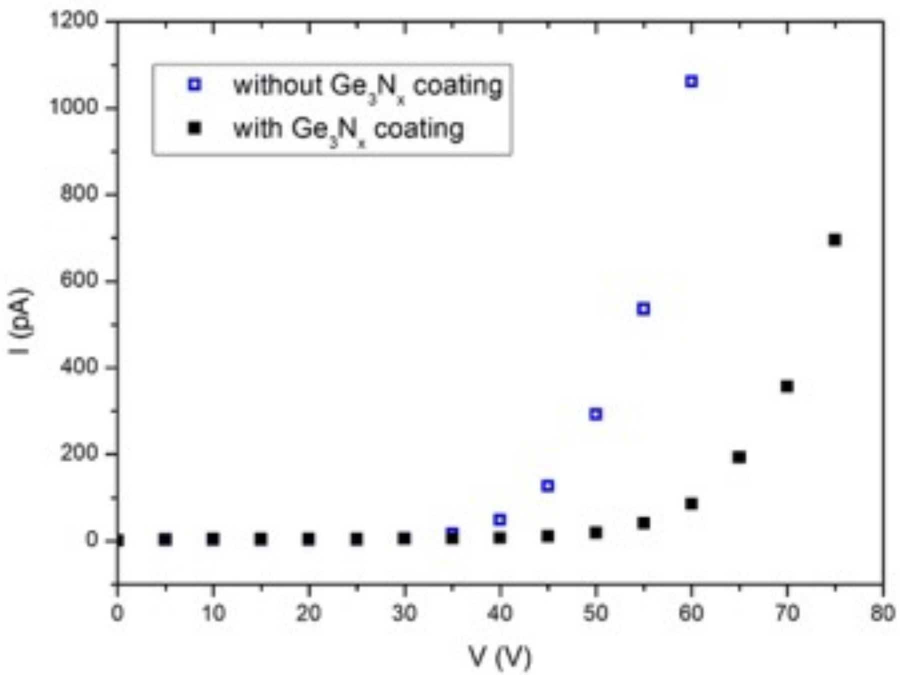
$$\sigma(T) = \sigma_0 \exp(-E_a/k_BT) \qquad \sigma_0 = \sigma_{00} \exp(E_a/E_{MN})$$

Meyer-Neldel rule

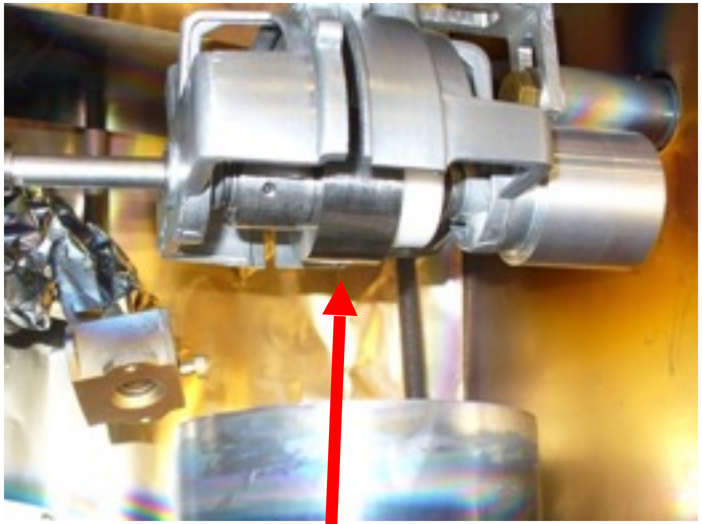
Very preliminary test: coating on a HPGe diode

A Ge_3N_4 film was deposited on the intrinsic surface of a defective HPGe diode

I-V characteristic of the diode was measured



(from G. Maggioni et al., **Appl. Surf. Sci.** 2016, *under review)*



HPGe diode

Further work is needed!

Main results (so far)

- Several passivation routes have been applied to a planar HPGe detector and their effects on the detector properties have been investigated
- As compared to a commercial passivation, the dead layers produced by the tested passivations are thinner and their thickness is more homogeneous
- The lateral scan measurements highlighted the electrical nature of the passivated surface (either n-type or p-type)
- Studies of new passivation methods started and are proceeding

Outlook

Task 1: New technologies on passivation and segmentation (INFN)

- Improvement of the present technologies for passivation and segmentation in HPGe detectors
- It is then necessary to find passivation methods that could be applied on the segments boundaries or on the whole surface of the detector without affecting the performance of the contacts in the segments

  → passivation and segmentation techniques need to be developed together
- Careful 2D scans of the full Ge volume in order to determine the charge collection and deduce the electric field in the prototype crystals

  → the benefits of the different surface-treatment techniques tested will be quantified

Goals:

- R&D of segmented contacts in HPGe detectors and of the passivation of the boundary

- regions between contacts

Multidisciplinary team

- INFN LNL: D.R. Napoli
- INFN and Padova University: G. Maggioni, S. Carturan, D. De Salvador, E. Napolitani, V. Boldrini, C. Scian, U. Pieri
- INFN and Trento University: G. Della Mea
- INFN and Verona University: G. Mariotto
- INFN and Camerino University: N. Pinto, S. Riccetto
- Cologne: J. Eberth

Thank you for your attention!!