2nd Position Sensitive Germanium Detectors (PSeGe)

On the way towards a coaxial segmented Ge detector at LNL-INFN

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INFN – Laboratori Nazionali di Legnaro

Ensar2 H2020-Infraia 2014/2015
n° 654002 WP10-Jra2 PSeGe

Multidisciplinary Team

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On the way towards a coaxial segmented Ge detector at LNL-INFN

OUTLINE

- HPGe coaxial:
  - lapping surface preparation (shaping)
  - (p+, n+) contact
  - passivation on intrinsic HPGe surface

Addendum

Alternative contact technology: SOD (spin on doping)

HPGe Surface preparation

Germanium surface polishing

Manual polishing

ceramic slurry:
deionized (DW) $H_{2}0$ + Al_2O_3 (9 - 6 - 3) μm

Automated polishing

Diamond polishing (15 - 9 - 6 - 3 - 1 - 0.25) μm

Accurate Cleaning :
- DW
- warm IPA

Chemical etching

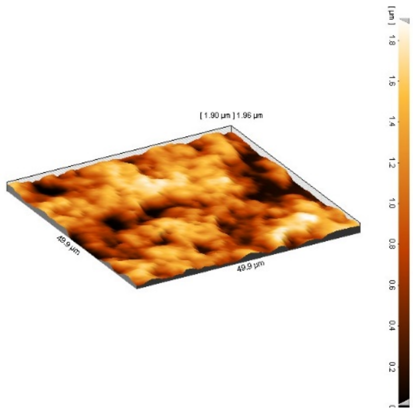
HNO_3:HF , 3:1
volume ratio

“Germanium strips for channeling: Study of the crystal quality after slicing and chemical etching”
S. Carturan et. al
Materials Chemistry and Physics 132 (2012) 641– 651

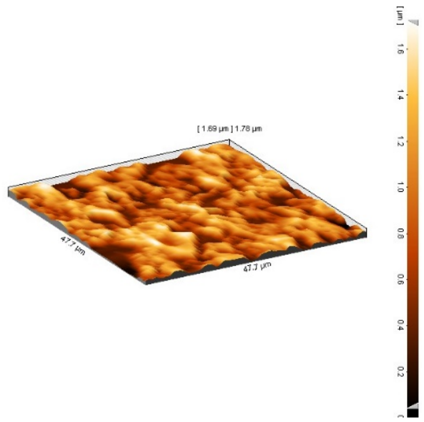
HPGe wafer surface roughness

AFM (atomic force microscope) morfology characterization

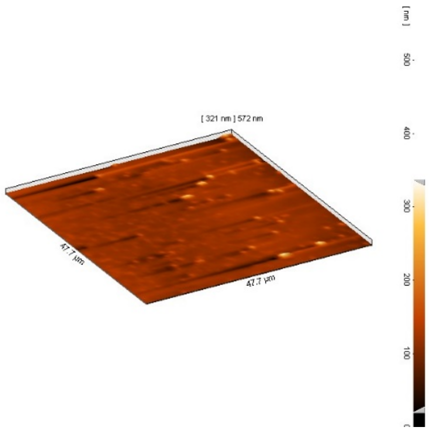
HPGe p-type as received (Umicore)



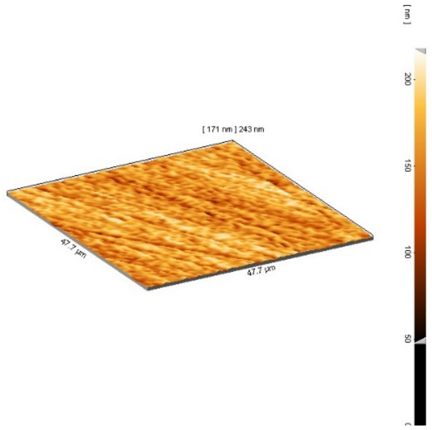
Manual polishing



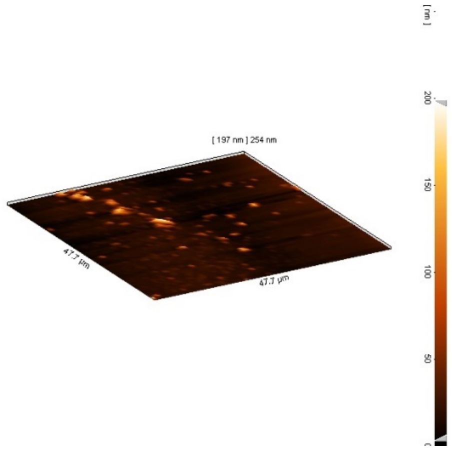
Manual polishing/etched



Automated polishing



Automated polishing/etched



Sample	R quadratic (nm)
HPGe as received	330 ± 85
Manual polishing	350 ± 40
Manual polishing/etched	37 ± 7
Automated polishing	22 ± 4
Automated polishing/etched	18 ± 4

→ down to 3µm

→ down to 0.25µm

HPGe surface dislocations
(EPD: etch pits density)

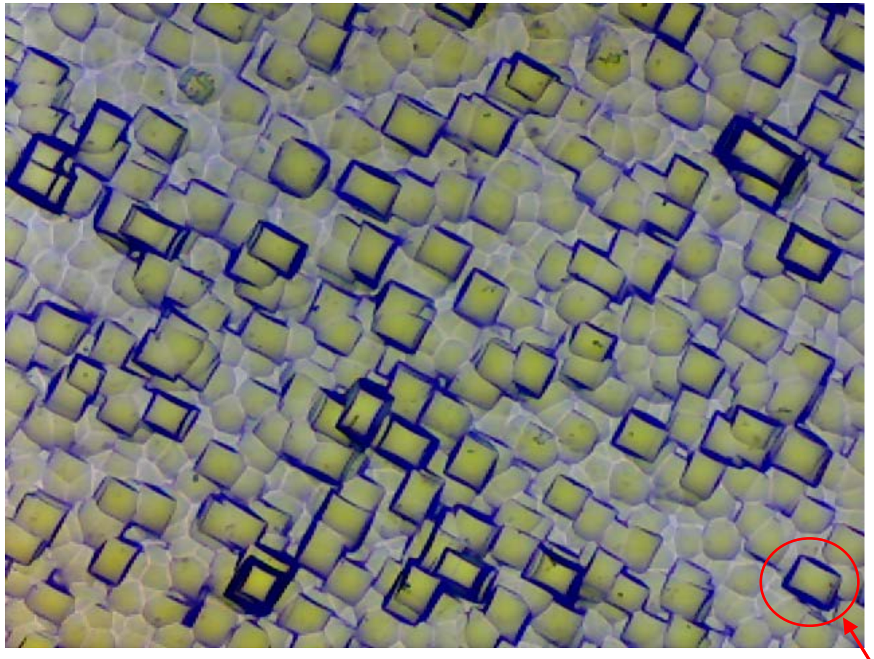
Umicore HPGe (100) as received
(50.8mm diameter, 2mm thick)
bulk dislocation: about 10^3 /cm^2

↓

SUPEROXOL (1 min)
chemical treatment
H_2O_2:HF:H_2O, 1:1:4

↓

EPD (etch pits density)
(n° dislocation / cm^2)



Magnification: 200 X
Area: 1,2x10^{-3} cm^2
Dislocation: >10^6 /cm^2

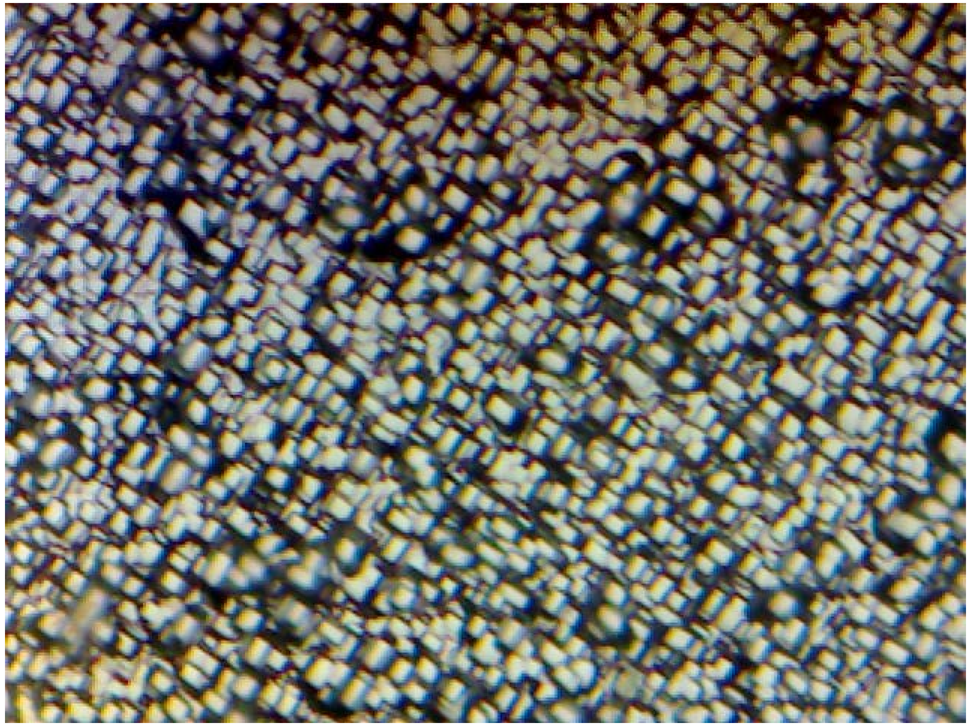
Dislocation

High dislocation number on the surface

Manual Polishing - EPD (etch pits density)

- polishing Al_2O_3 (grain size 3µm) + H_2O (DW)
  + Superoxol 1 min

- Etching 3:1 (HNO_3:HF) rate 20µm/min (etch=3min)
  + Superoxol 1 min



Magnification: 500 X
Area: 1,92x10^{-4} cm^2
Dislocation: >10^6 /cm^2

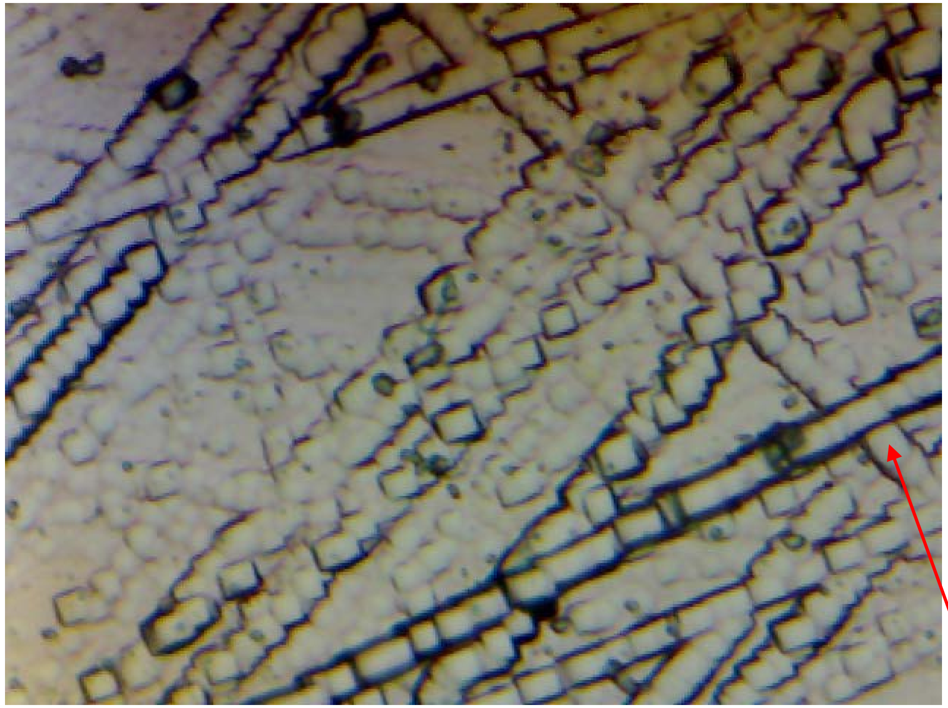
+ etching

Magnification: 200 X
Area: 1,23x10^{-3} cm^2
Dislocation: 4x10^3 /cm^2

Automated Polishing - EPD (etch pits density)

- Diamond polishing (down to 3μm)
  + *Superoxol* 1 min

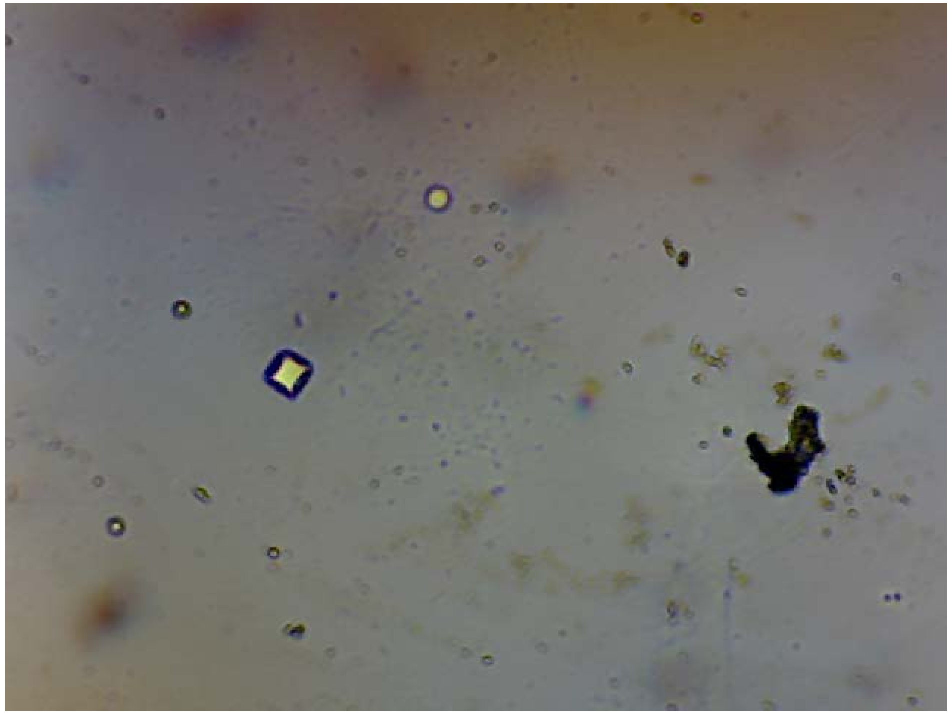
- Etching 3:1 (HNO_3:HF) (etch=3min)
  + *Superoxol* 3 min



Magnification: 500 X
Area: 1,92x10^{-4} cm^2
Dislocation: >10^6 /cm^2

strips

+ etching

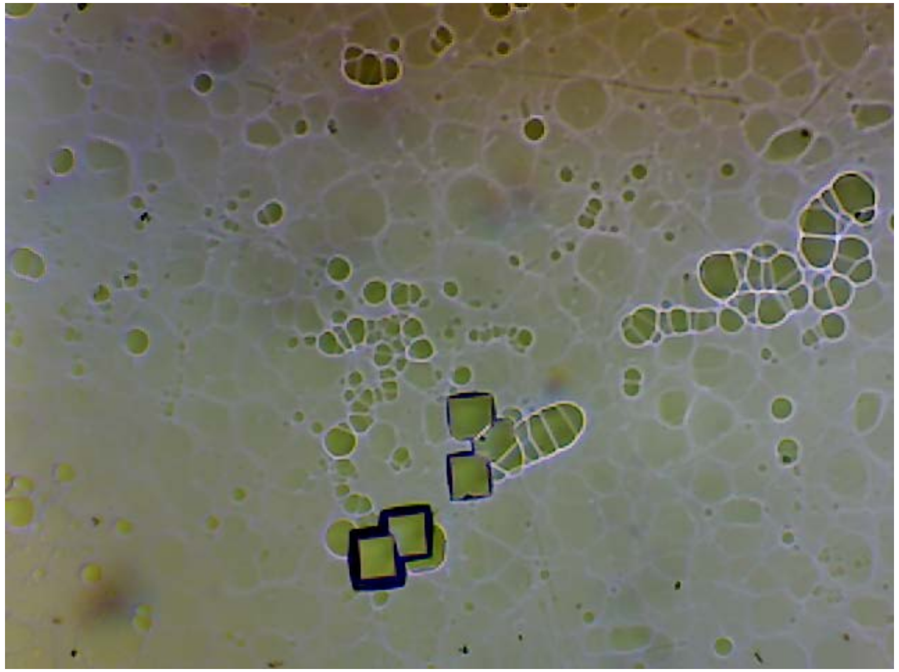


Magnification: 200 X
Area: 1,23x10^{-3} cm^2
Dislocation: 3x10^3 /cm^2

EPD (etch pits density)

Manual polishing

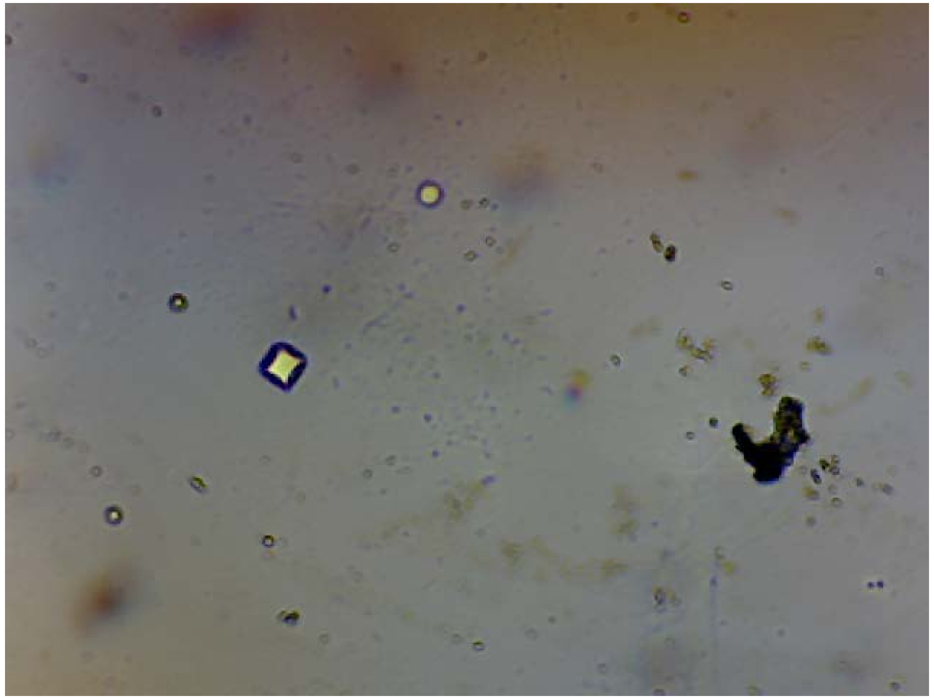
Magnification: 200 X
Area: 1,23x10^{-3} cm^2
Dislocation: 4x10^3 /cm^2



- High purity Al_2O_3 and DW water, no contamination of HPGe crystal
- Better control on the lapping process (based on the operator’s sensitivity)

Automated polishing

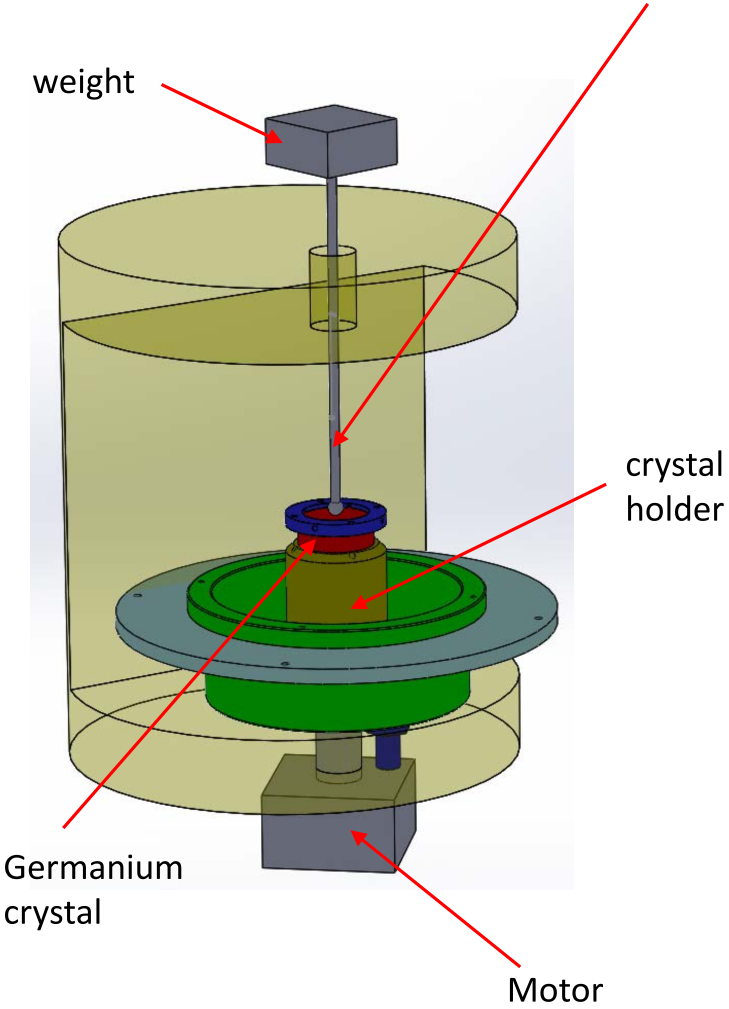
Magnification: 200 X
Area: 1,23x10^{-3} cm^2
Dislocation: 3x10^3 /cm^2



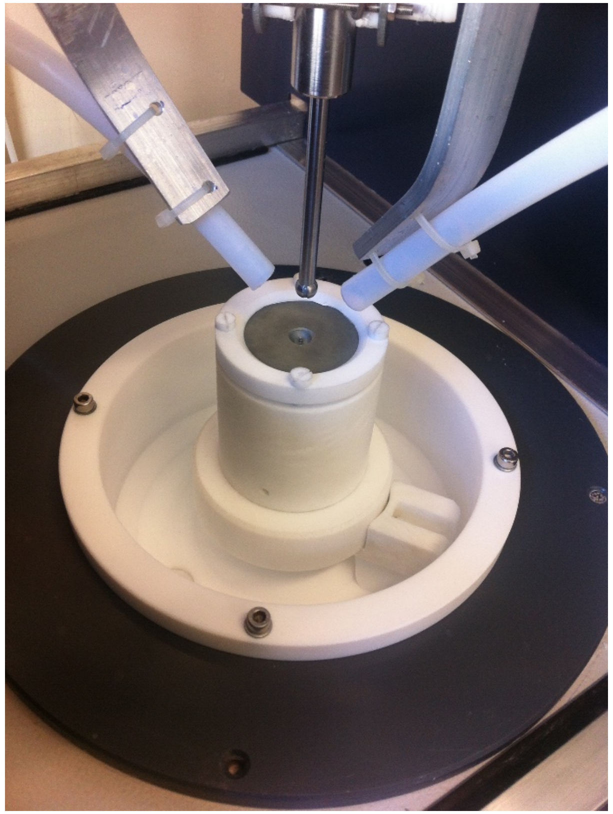
- No control on the chemical impurities in the used materials
- Worse control on lapping process (strip defects)
- Good technology for first crystal shaping

Drilling system for coaxial detector

3D design



IKP tip is fed with ceramic slurry [H_2O (DW) + Al_2O_3 ($30\mu m$)]



depth of hole ≈ 30mm
Hole diameter ≈ 9mm

Ge crystal

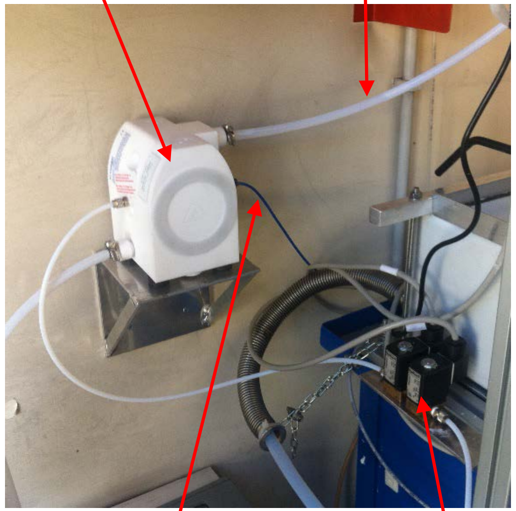
$\varnothing_{ext} = 50$mm
Height = 43mm

Drilling system for coaxial detector

PTFE (Polytetrafluoroethylene) circuit to feed the tip with ceramic slurry

PTFE pump

PTFE pipes

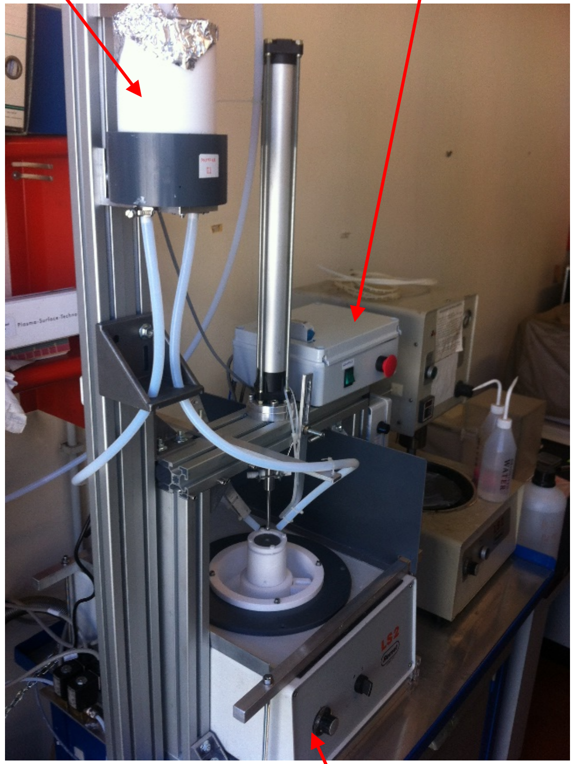


Pump break sensor

Power pump: nitrogen gas

PTFE tank

switching amplifier



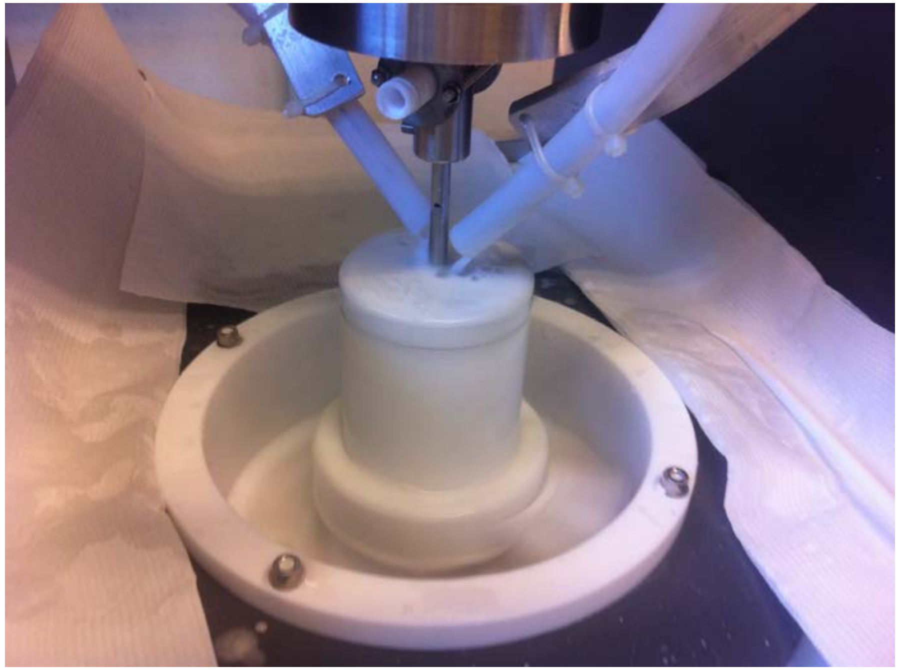
Rotation speed controller

Drilling rate < 0.3 mm/h

Slurry ≈ 70% (H$_2$0) – 30% (Al$_2$O$_3$, 30μm) weight %

Weight tip ≈ 2Kg

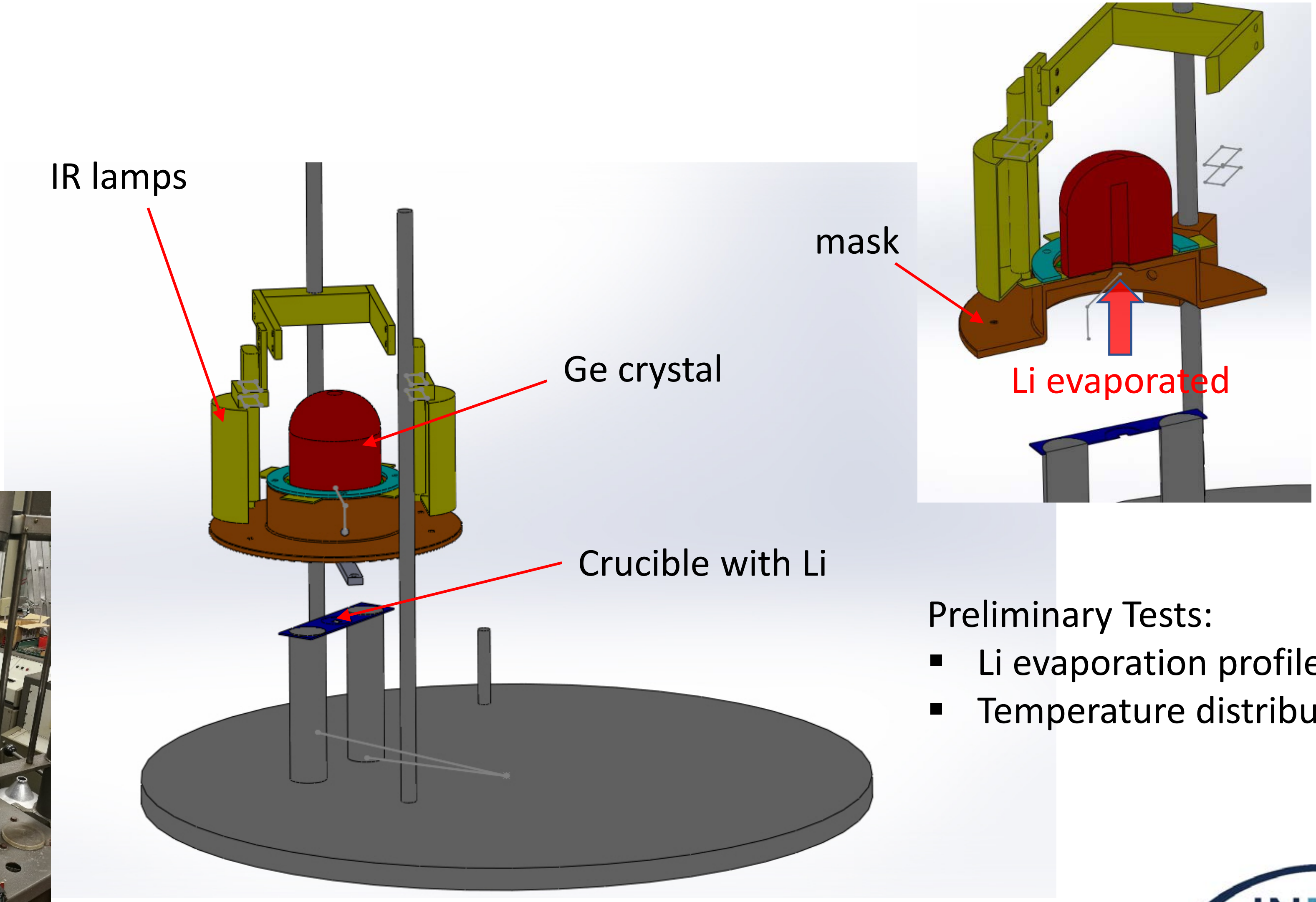
Rotation speed < 70rpm



n+ contact Lithium diffusion at LNL-INFN

Coaxial crystal handler at LNL-INFN to perform the lithium diffusion in **n-type HPGe** crystal

Thermal Evaporator



Preliminary Tests:

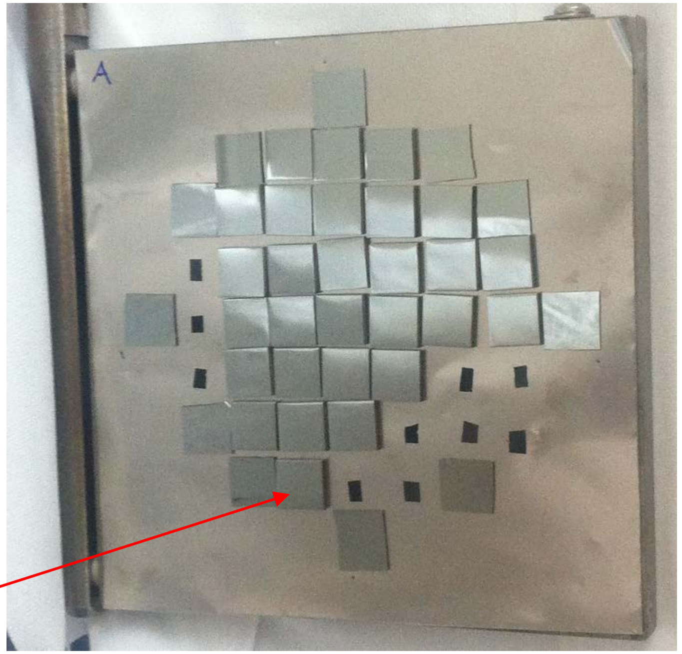
- Li evaporation profile
- Temperature distribution

p^+ contact on HPGe

^{11}B Ionic Implantation IMM (Institute for Microelectronics and Microsystem) - Bologna

HPGe wafer cut and cleaning (isopropanol 80°C and DW 80°C)

Pressure= 3.8×10^{-7} torr
Energy= 23KeV
Dose= 1×10^{15} atoms/cm^2

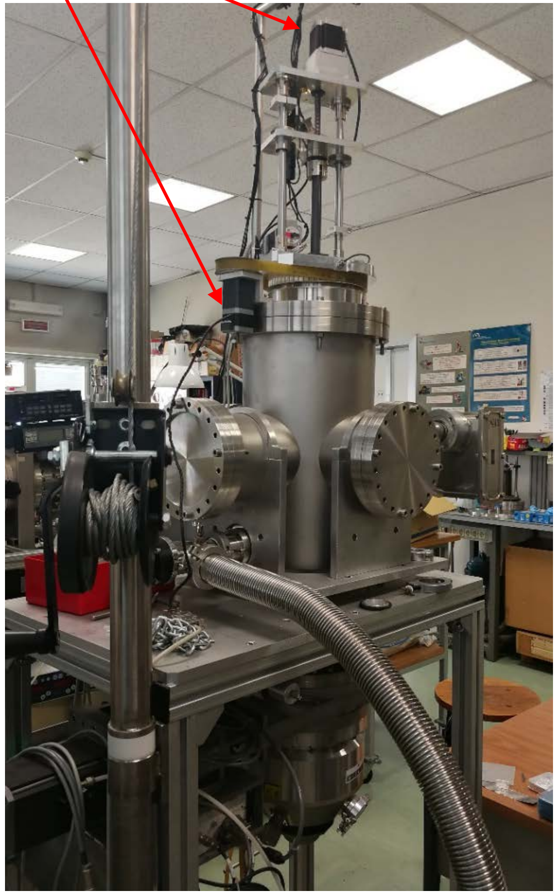


HPGe
(10x10x2) mm^3

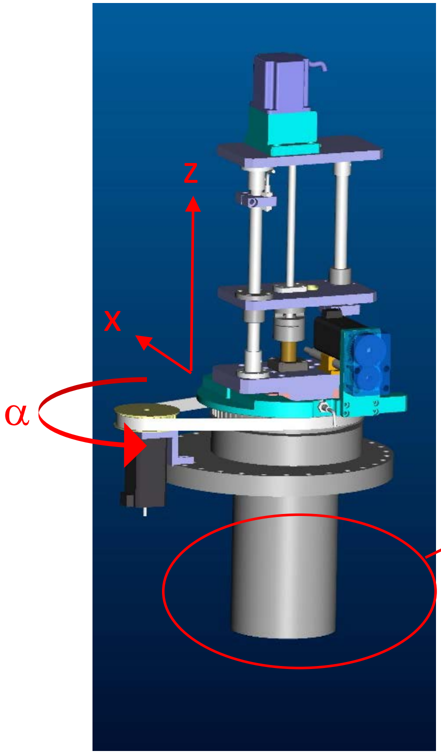
Coaxial crystal handler for Boron implantation

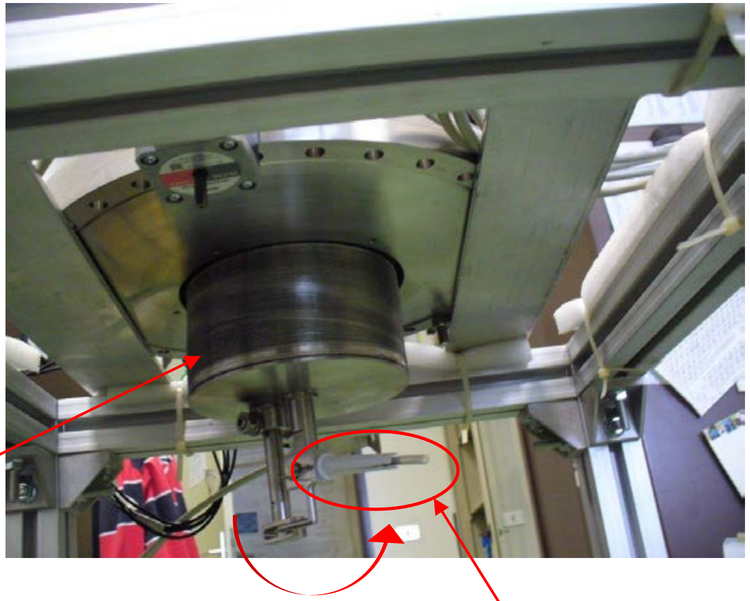
External Motors

will be installed at IMM (Institute for Microelectronics and Microsystem) - CNR Bologna



3D design



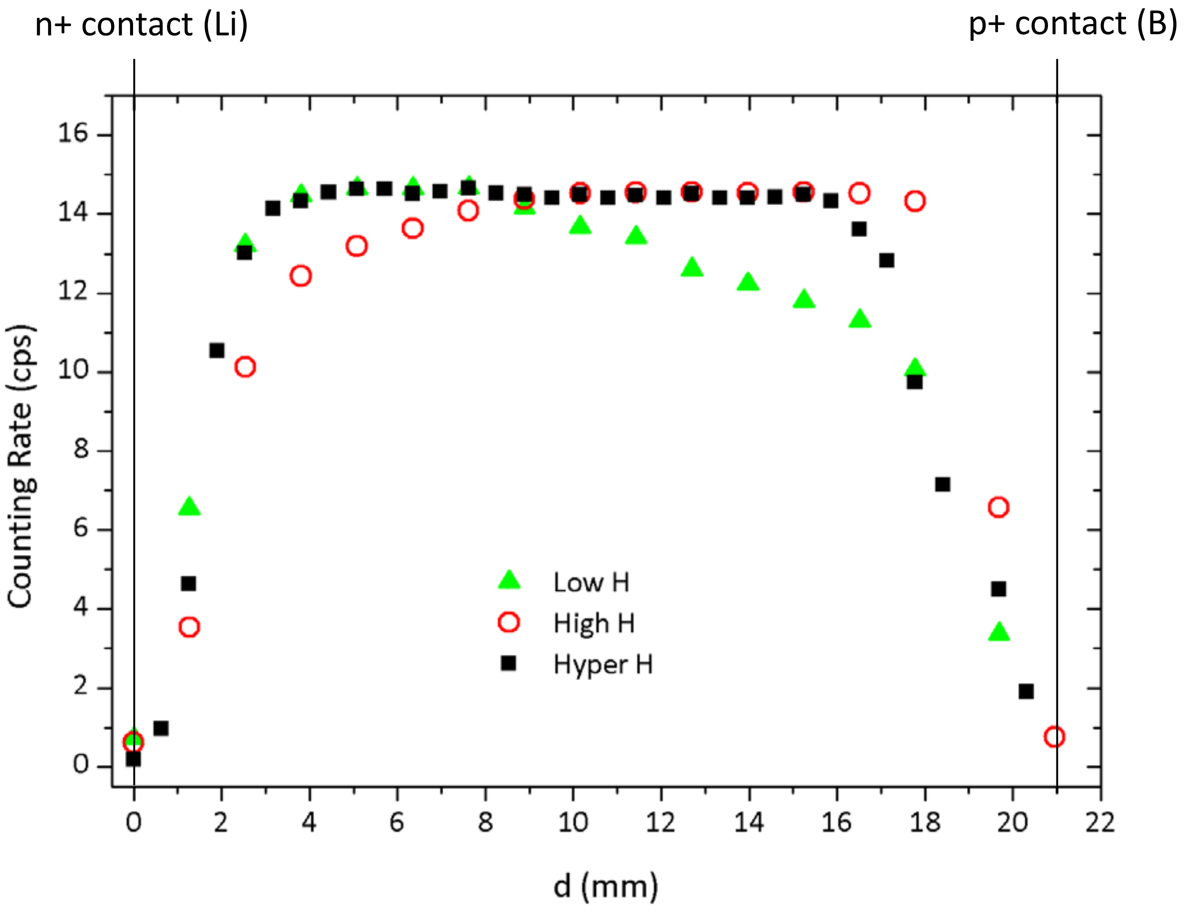


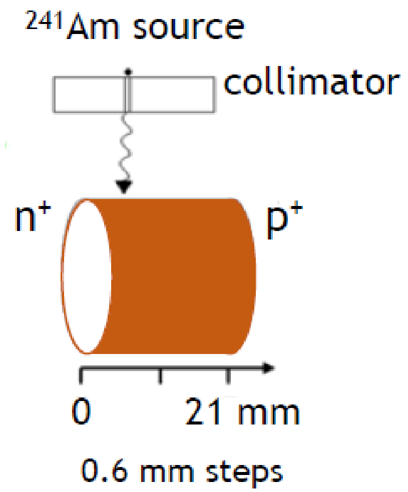
90°

For single ended

Coaxial crystal holder

HPGe Passivation: lateral scan ^{241}Am on passivated surface





G. Maggioni et al. Eur. Phys. J. A (2015) 51: 141
S. Riccetto et al. to be submitted (see next presentation)

- Strong decrease of counting rate close to the electrodes
- Hyper-H flat counting rate

On the way towards a coaxial segmented Ge detector at LNL-INFN

summary

- Manual lapping allows better process control, and the agents in contact with the hyperpure germanium is high purity. The dislocation number 10$^{\wedge 3}$ cm^{-2} after etching process confirm the bulk HPGe dislocation density

- In Automated lapping small splinters that detach during the process and scratch the surface germanium crystal. The agents used (diamond sprays) do not have a known composition and can contaminate the HPGe crystal.

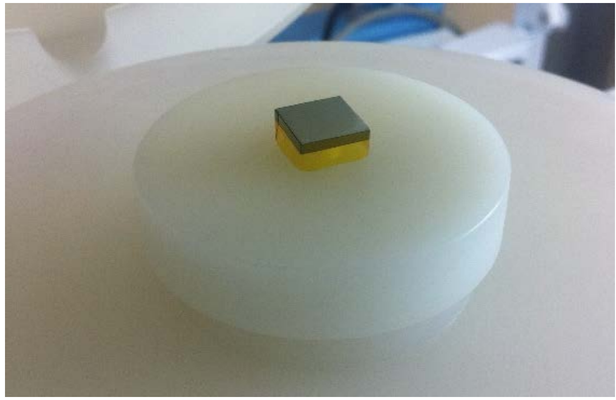
- The drilling system allows to obtain blind hole on germanium crystals, and therefore allows for good control over the presence of contaminants since the entire circuit is in PTFE in contact with the known ceramic slurry

Addendum

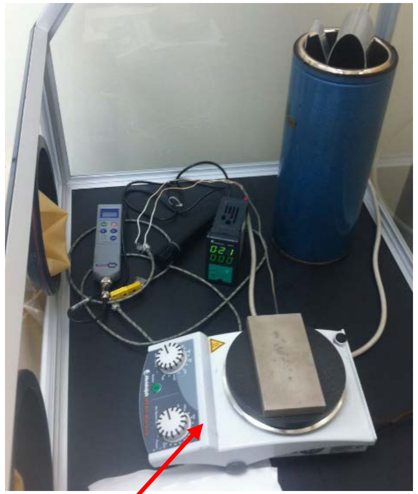
HPGe diode: n$^+$ contact on p-type HPGe

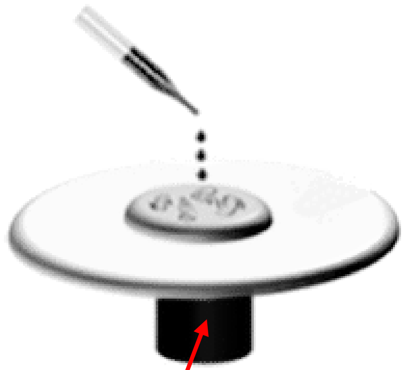
Precursor
P 507 (Phosphorus)
Filmtronics

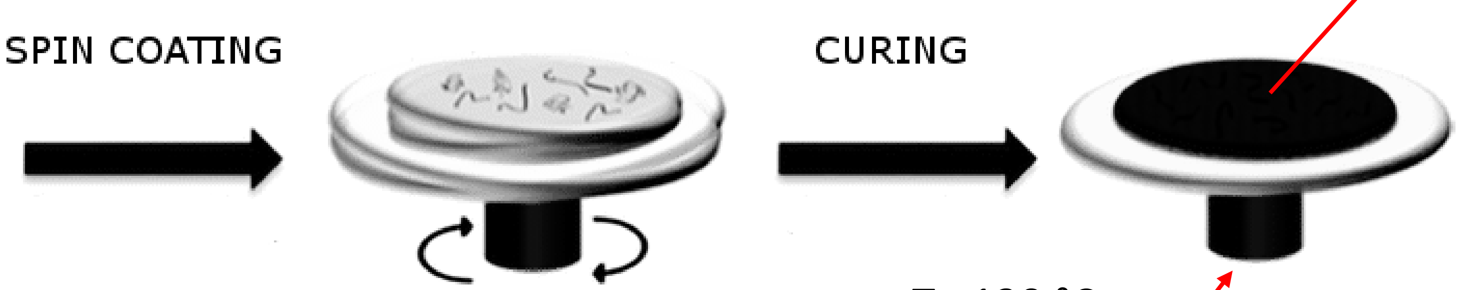
SOD (Spin on Doping)



Lateral Kapton on p-type HPGe







HPGe p-type
size (10x10x2)mm^3

4500 rpm

T= 130 °C
t= 30min
RH < 20%

Spike annealing
610°C – 12minutes

Etching HF 10% to
remove the glassy coating

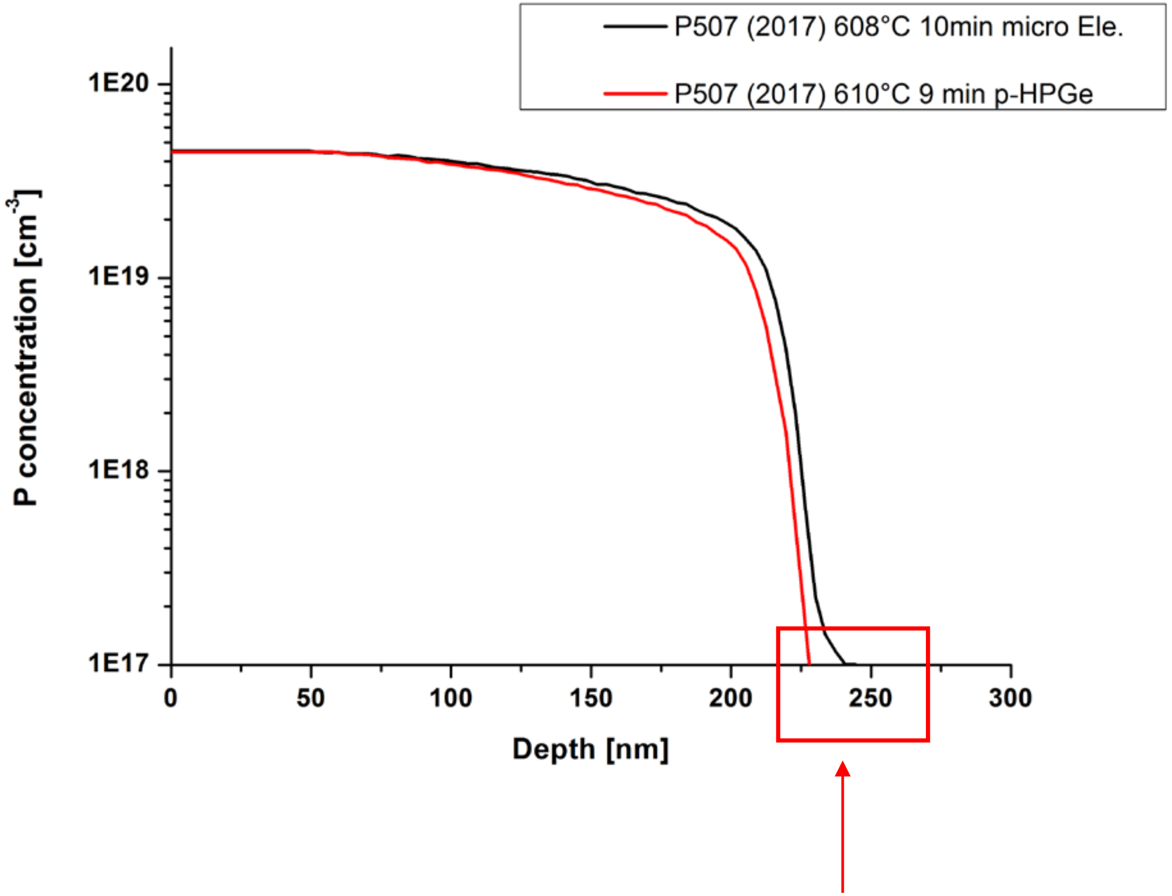
“Optimal process parameters for phosphorus spin-on-doping of germanium”
V. Boldrini et al., Applied Surface Science 392 (2017) 1173–1180

SIMS profiles of Phosphorus diffusion on HPGe

The **SOD** process is applied to HPGe p-type crystal. The optimization of **temperature**, **time** and **humidity** are fundamental to obtain reproducible SOD contact.

HPGe SIMS profile is similar to micro electronic Ge SIMS profile.

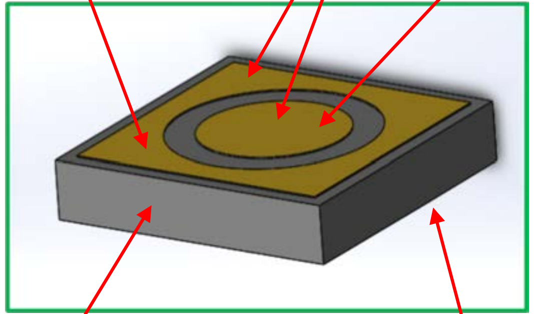
SOD technique can be applied to HPGe crystal !!



Thin contact layer < 250nm !!!

On the way towards a γ– detector (prototype)

HPGe diode



guard ring contact

Metal thin film layer (Au) on boron implanted contact

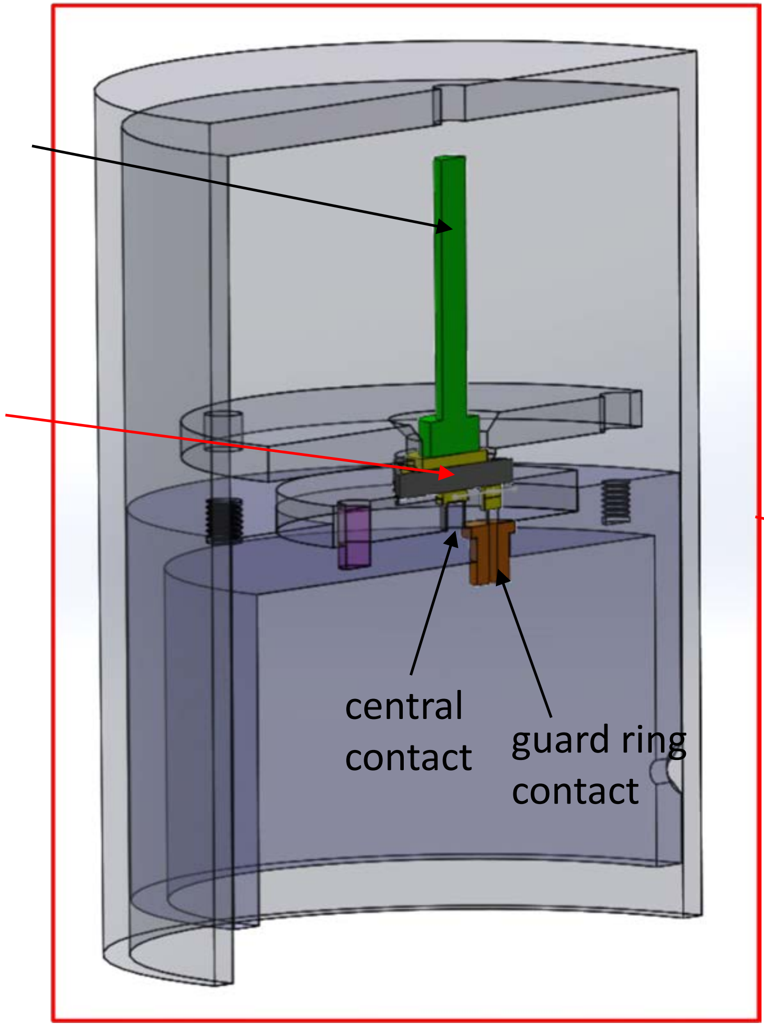
central contact

Diode size (10x10x2) mm^3

Lateral surface with methanol passivation

Indium foil on phosphorous (SOD) contact

3D cryostat design



nitrogen cooling

HPGe diode

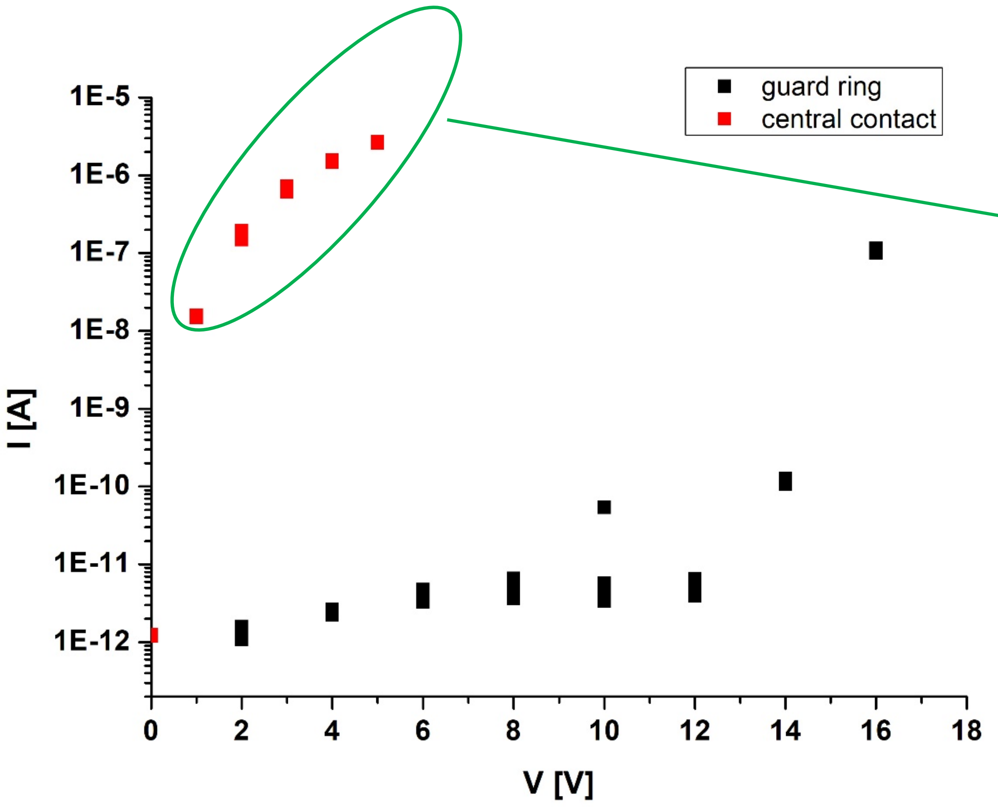
central contact

guard ring contact

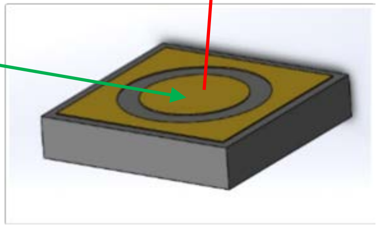
I-V Diode characterization

HPGe p-type
boron – phosphorus contact
methanol (CH_3OH) lateral surface passivation

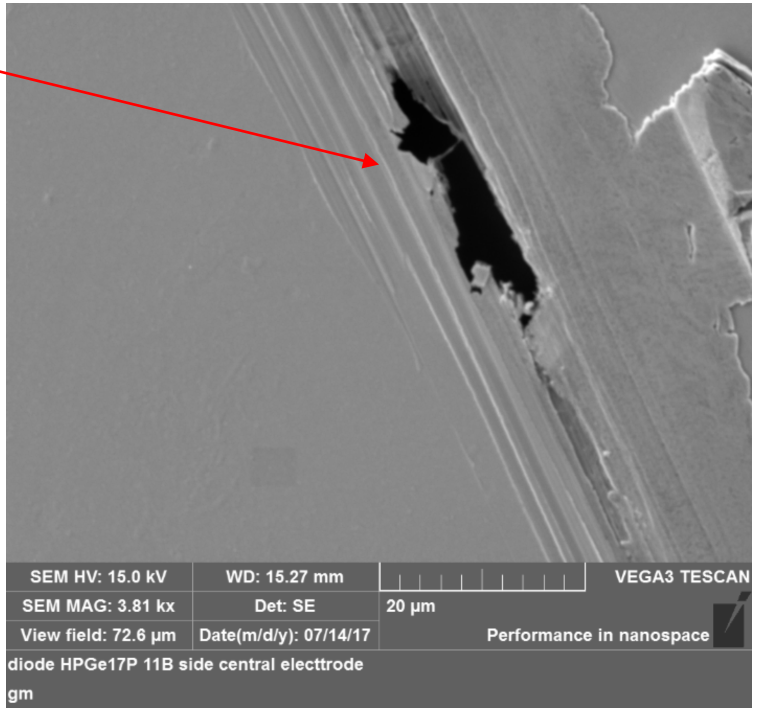
high current on central contact



Defects on central contact

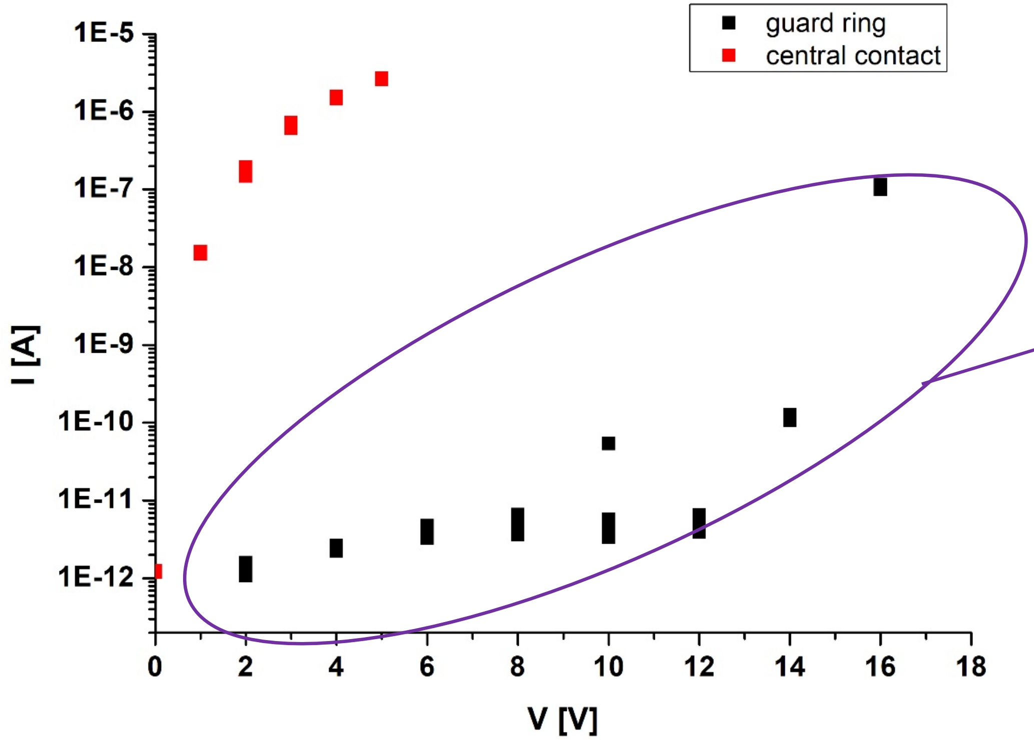


Diode size (10x10x2) mm^3

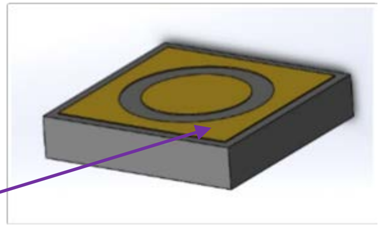


I-V Diode characterization

HPGe p-type
boron – phosphorus contact
methanol (CH_3OH) lateral surface passivation



Diode size
(10x10x2) mm^3



SOD diffusion thermal treatment change the net impurity concentration of HPGe
2 x 10^^{11} atoms/cm^3 (see Virgina Boldrini talk)

summary

- We have measured a p-n junction made by SOD technology but the HPGe detector is not fully depleted

outlook

- We are still working with alternative contacts (see F. Sgarbossa and V. Boldrini talks)

Thanks for the attention !!

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2nd Position Sensitive Germanium Detectors (PSeGe)

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(Ensar2 agreement)

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