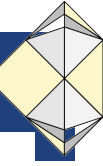


Diamond Pixel Fabrication

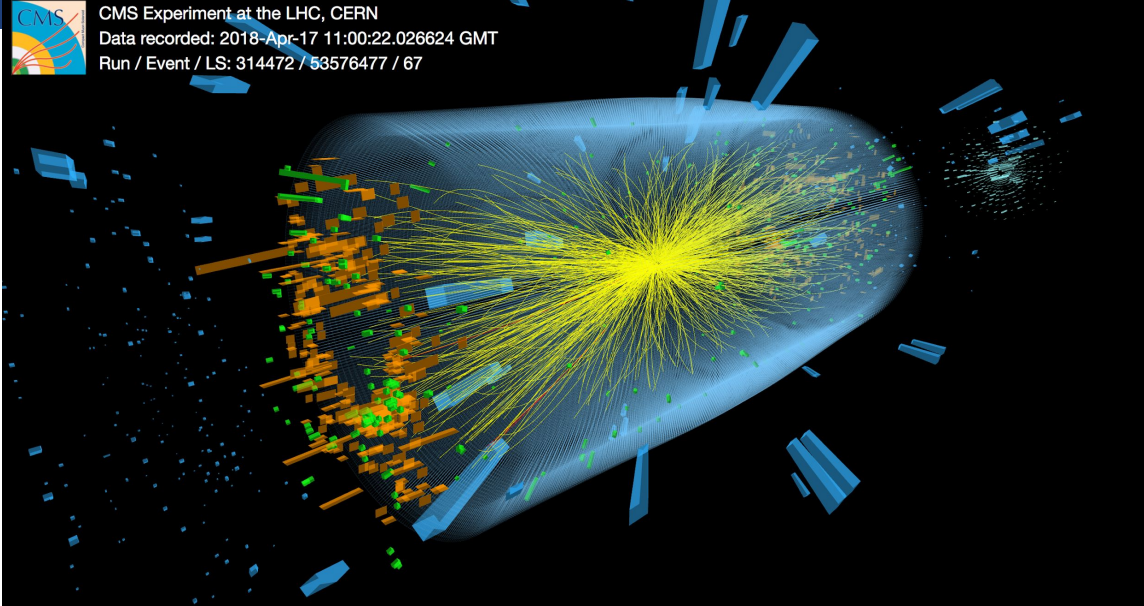
PhD Seminar 2019

Diego Alejandro Sanz Becerra

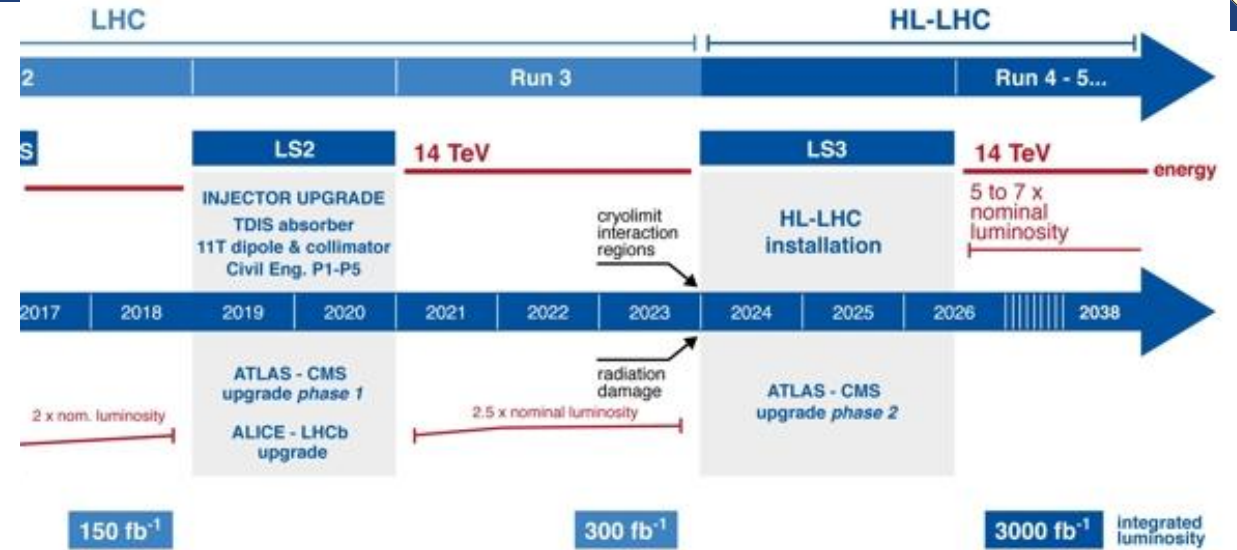




- Why Diamonds?
- Planar Hybrid Pixelated Detectors
- Microfabrication Techniques
- Samples Preparation
- Fabrication Process
- Preliminary Results (Testbeam September 2019)



CMS Experiment at the LHC, CERN
Data recorded: 2018-Apr-17 11:00:22.026624 GMT
Run / Event / LS: 314472 / 53576477 / 67

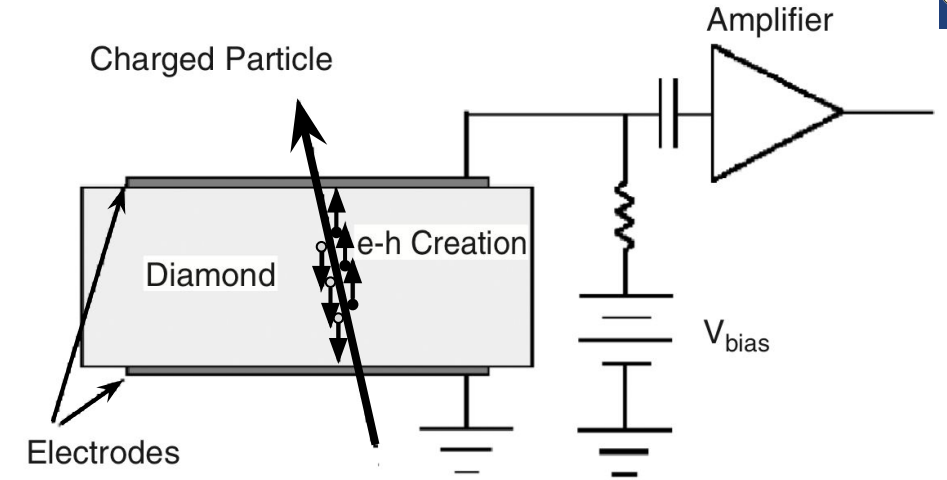


- The innermost detectors in HL-LHC experiments will be exposed to a total fluence of 2×10^{16} hadrons/cm² (~ 1 GHz/cm²; ~ 500 MRad)
- Diamond is the best radiation tolerant sensor material in locations with no cooling

Make pixel detectors for future HEP experiments at ETH



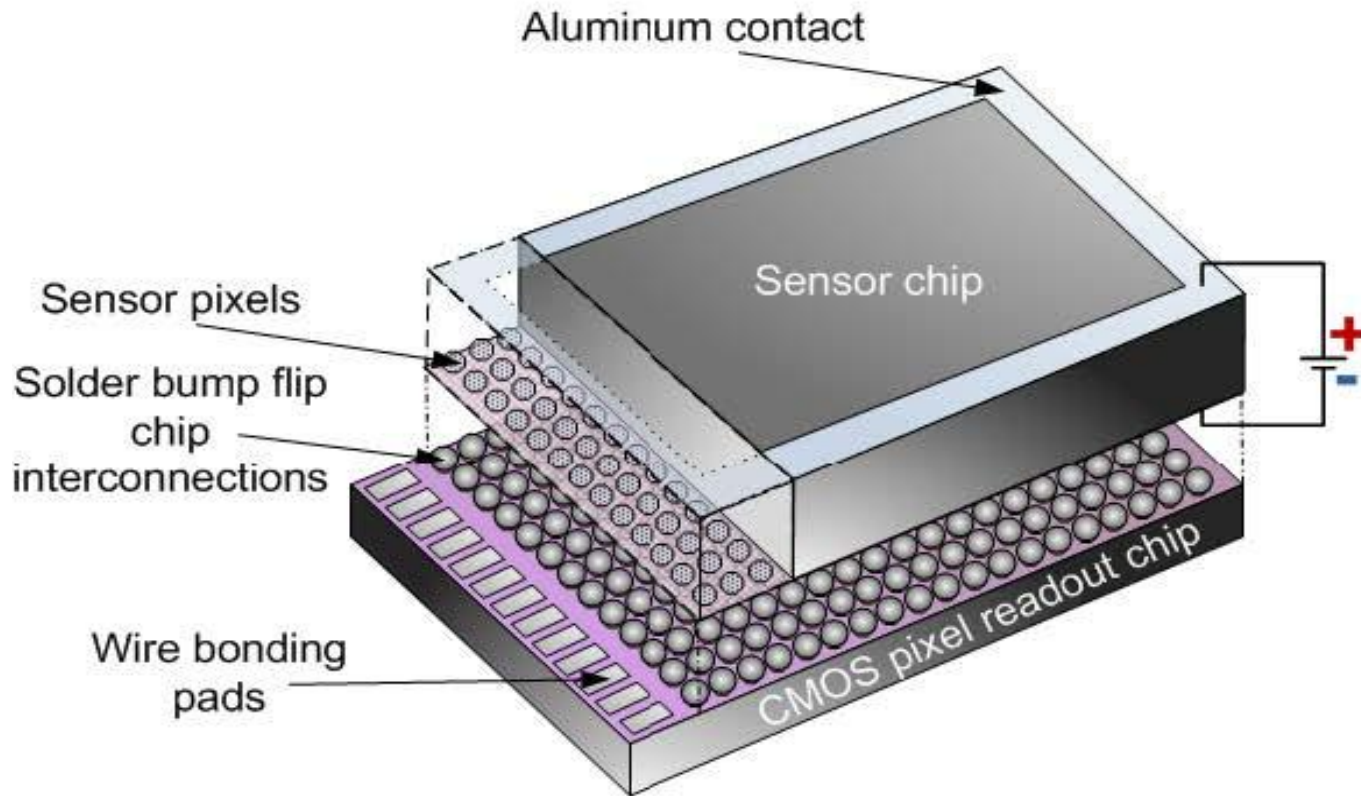
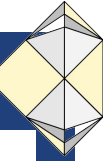
Characteristic	Silicon	Diamond	Comment
Band Gap [eV]	1.12	5.45	Low Leakage Current
Electron Mobility [cm ² /Vs]	1450	1714	Fast Signals
Hole Mobility [cm ² /Vs]	500	2064	Fast Signals
Saturation Velocity [cm/s]	0.8 x 10 ⁷	~ 1 x 10 ⁷	Fast Signals
Breakdown Field [V/m]	3 x 10 ⁵	2.2 x 10 ⁷	Withstand High Fields
Resistivity [Ω m]	3200	> 10 ⁴⁰	Low Leakage Current
Dielectric Constant	11.9	5.7	Low Input Capacitance
Displacement Energy [eV]	13-20	43	Radiation Hardness
Thermal Conductivity [W/cmK]	1.5	22	Efficient Heat Spreading
e-h Creation Energy [eV]	3.6	13	Small Signal
Average e-h Pairs per MIP per μm	89	36	Small Signal
Charge Collection Distance / thickness	100%	50% polycrystalline - 100% single crystal	Small Signal



Artificially grown diamonds by chemical vapor deposition:

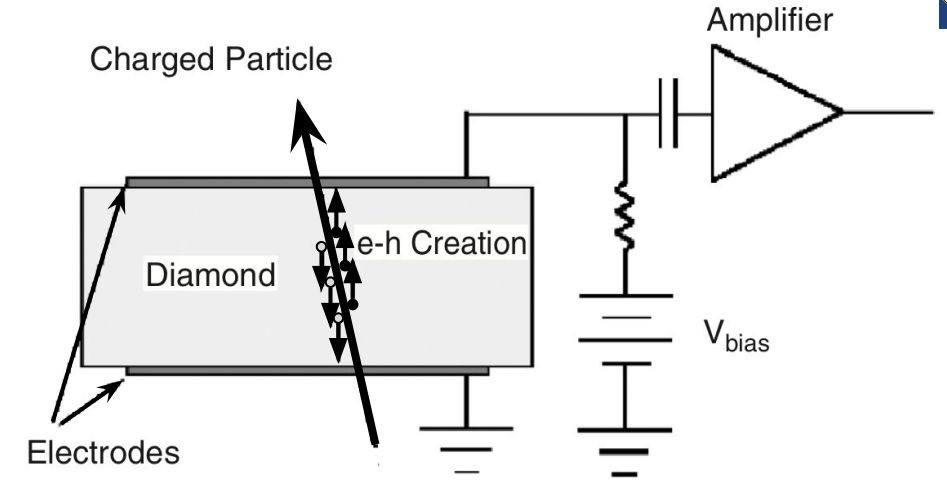
- Single crystal (large signals but up to $\sim 8 \times 8 \text{ mm}^2$; more expensive)
- Polycrystalline (small signals but up to $\varnothing 6 \text{ in}$ wafers; cheaper)

Diamond detectors are operated as ionisation chambers. Different from silicon sensor detectors Planar devices are the first step before attempting more advanced geometries (i.e. 3D detectors)

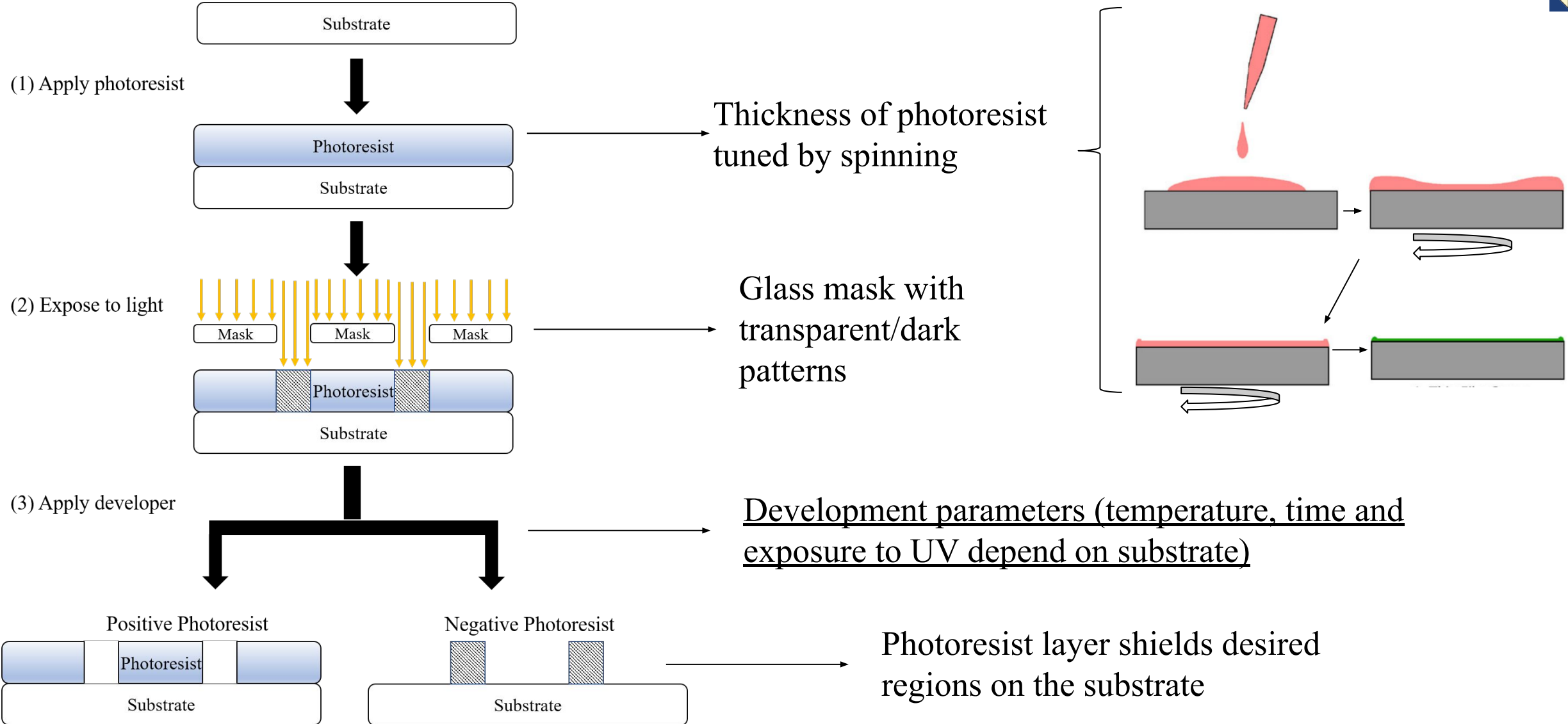


Generic pixel detector

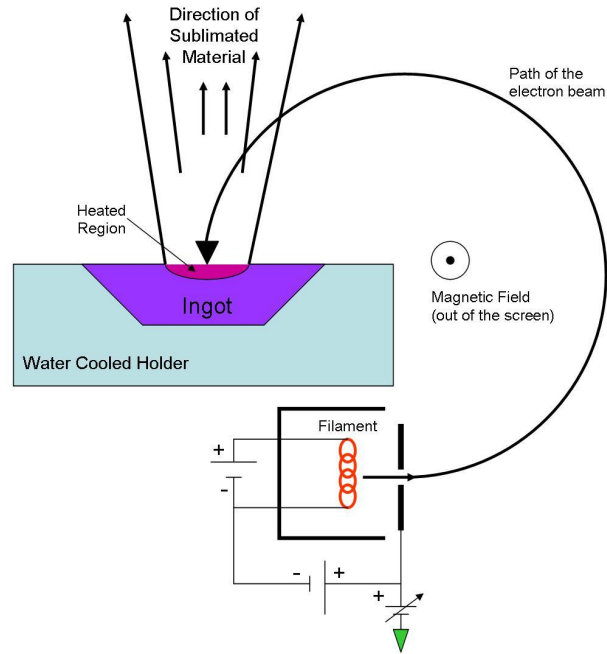
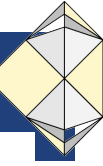
Quality Issues in Current and Future Silicon Detectors workshop 3-November-2011



- Sensors are typically 500 μm thick polycrystalline diamond of 1 cm x 1 cm
- Readout chip used is the psi46digV2.1 respin
 - Used for CMS layer 2 - 4
- **Develop microfabrication techniques to couple the diamond sensor with the silicon readout chip**
 - **Standard silicon microfabrication recipes don't work with diamond**



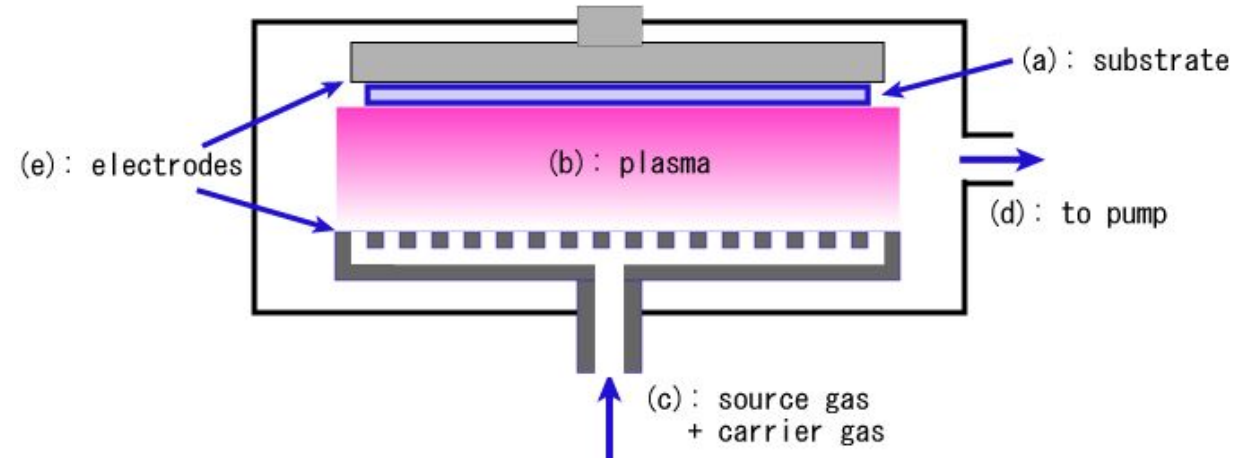
https://en.wikipedia.org/wiki/File:Photoresist_of_Photolithography.png



https://en.wikipedia.org/wiki/File:Electron_Beam_Deposition_001.jpg

Evaporation

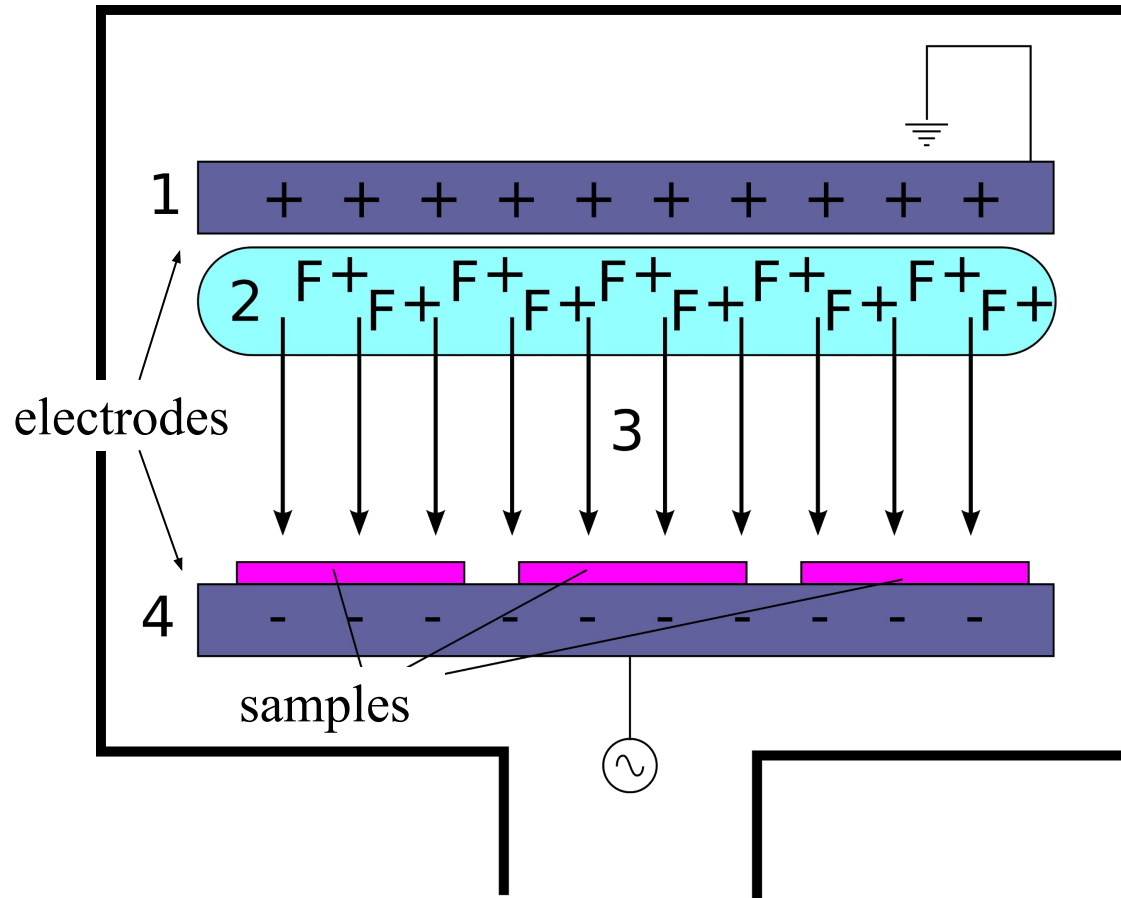
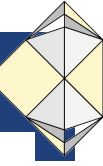
- Metals are heated in a vacuum chamber
- Metal vapors rise and coats surface on top
- Suitable metallizations that form good contacts with diamond were tested



<https://commons.wikimedia.org/wiki/File:PlasmaCVD.PNG>

Chemical Vapor Deposition

- Layers of a material are grown on a substrate under specific chemical reactions
- The mixture of gases, and chamber conditions determine the properties of the deposited material and the deposition rate
- A compatible recipe with the process was developed to grow a passivation/protective layer

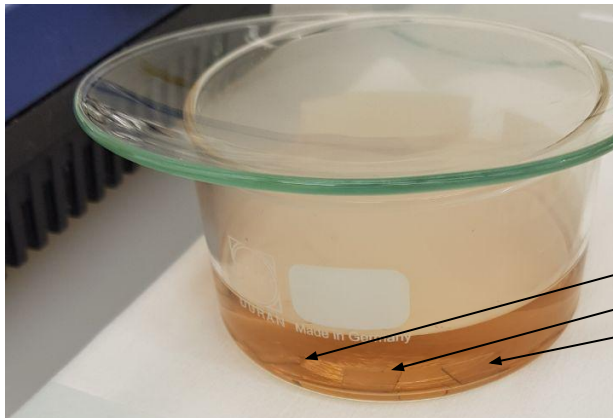


<https://commons.wikimedia.org/wiki/File:Rieoperation.svg>

Reactive Ion Etching

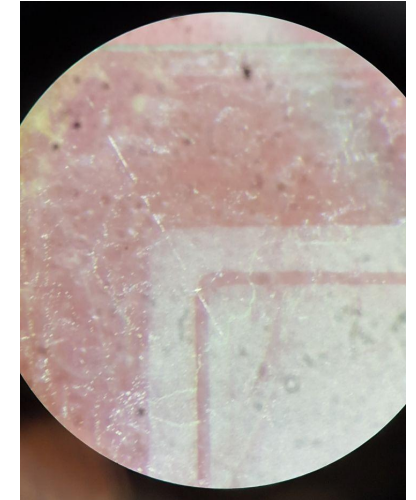
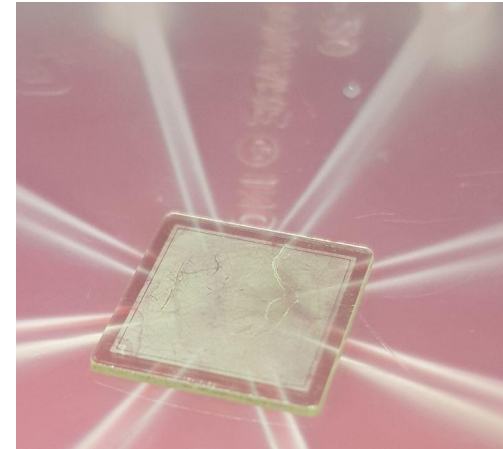
- A plasma of a gas mixture is used to remove desired material
- A specific recipe to etch through the passivation layer was developed and characterized

- Clean with different acids at boiling temperatures to remove any surface contamination
 - From the diamond growth
 - From previous detectors (we reuse diamonds)
- Etch with Reactive Ion Etching tuned with Ar/Cl₂ and O₂ plasmas to remove ~2μm of diamond from each side
- Clean with solvents in ultrasound bath

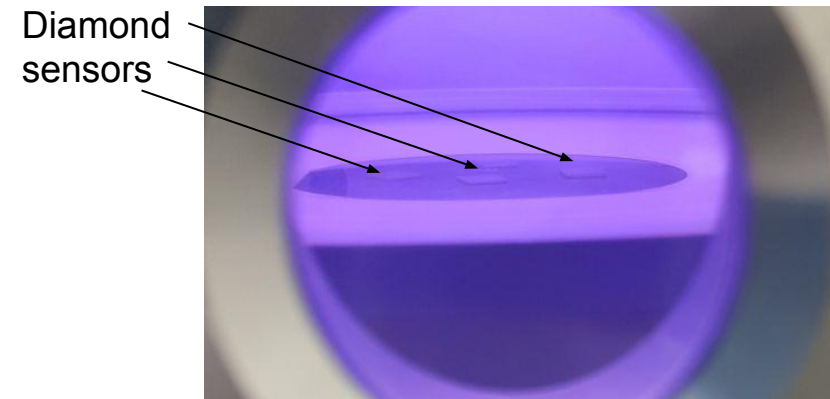


Diamond sensors

Sensors cooling down after Aqua-regia cleaning



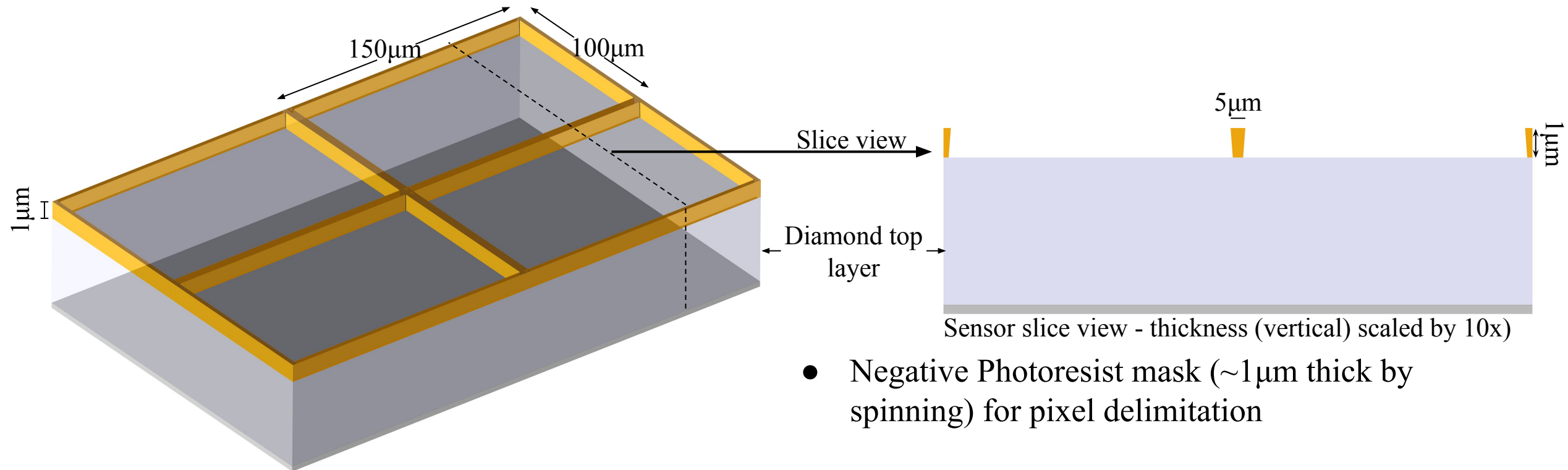
Pictures taken in between boiling acids cleaning. It is evident the residue from old-fabrication in the sample



Plasma etch with Ar/Cl₂



1. Pixels metallization - Photoresist mask

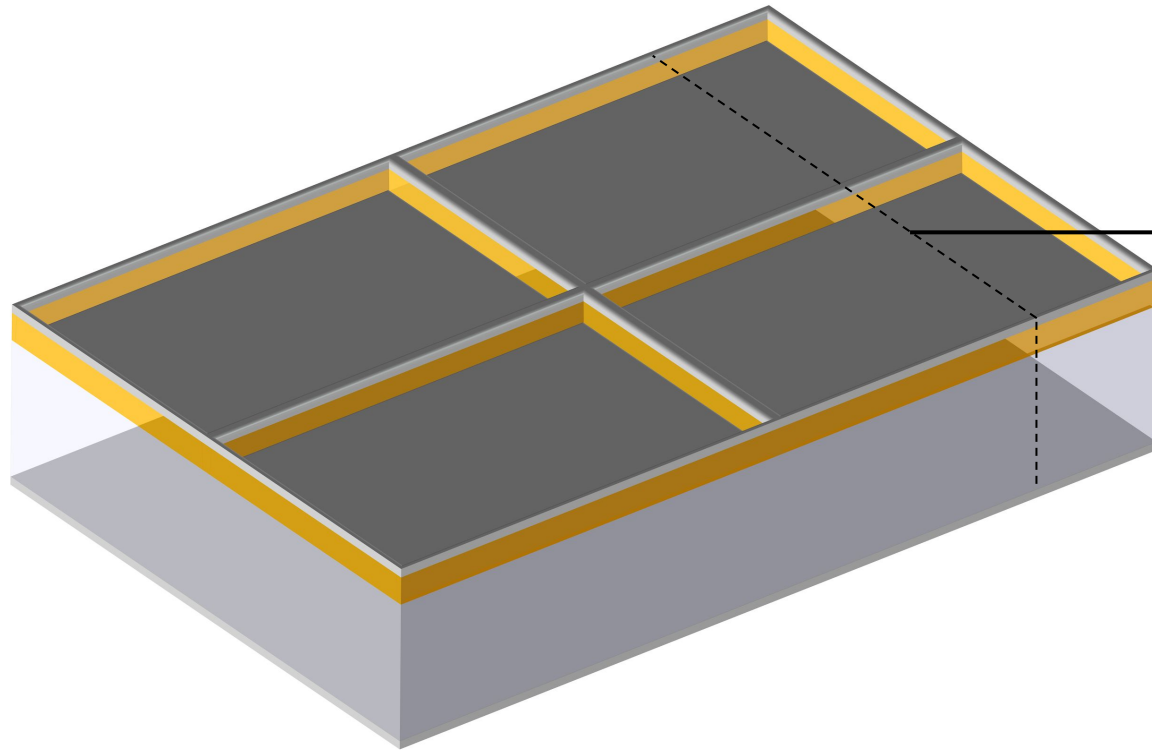


- Negative Photoresist mask ($\sim 1\mu\text{m}$ thick by spinning) for pixel delimitation

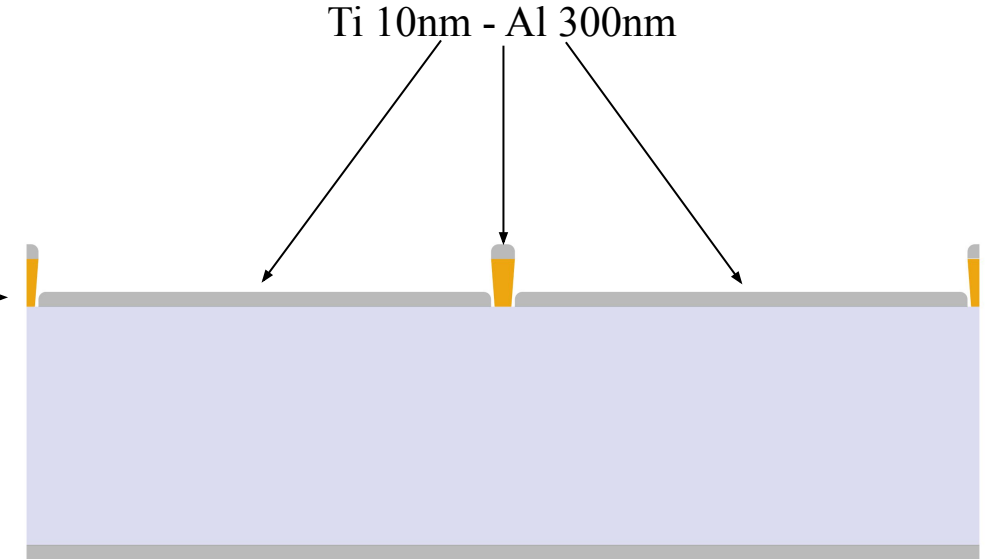
Sensor top surface view - thickness scaled by 10x



1. Pixels metallization - Metal deposition (electron beam evaporation)



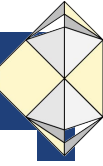
Slice view →



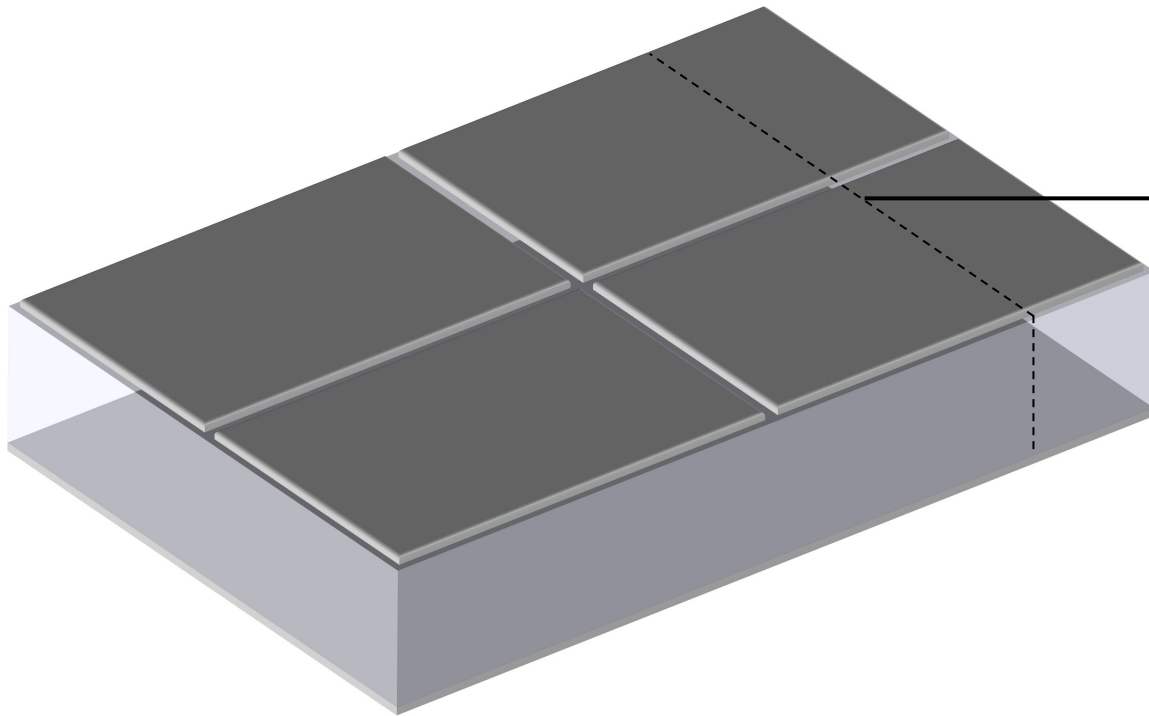
Sensor slice view - thickness (vertical) scaled by 10x)

- Negative Photoresist mask ($\sim 1\mu\text{m}$ thick by spinning) for pixel delimitation
- Deposition of 10nm of Ti and 300nm of Al

Sensor top surface view - thickness scaled by 10x



1. Pixels metallization - Lift-off and annealing



Sensor top surface view - thickness scaled by 10x

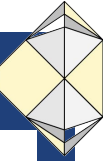


Slice view

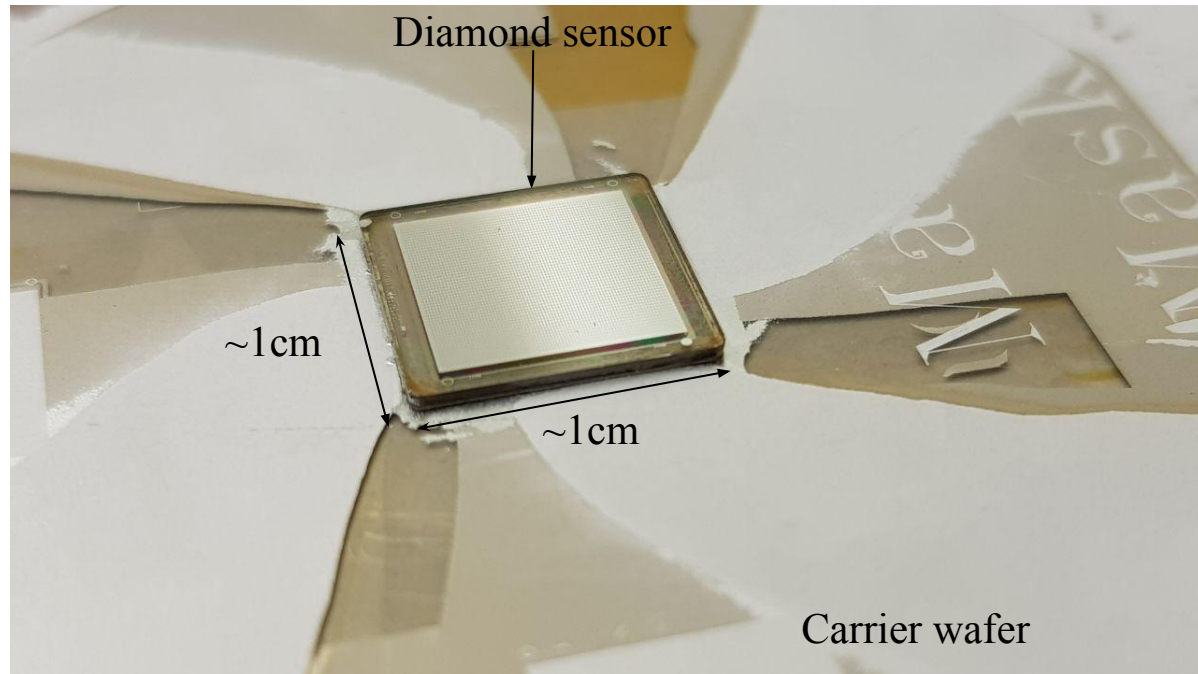


Sensor slice view - thickness (vertical) scaled by 10x)

- Negative Photoresist mask ($\sim 1\mu\text{m}$ thick by spinning) for pixel delimitation
- Deposition of 10nm of Ti and 300nm of Al
- Photoresist and excess metal lift-off using solvents
- 400°C annealing with Ar \rightarrow carbide binding (C-Ti) for ohmic contact formation on the diamond



1. Pixels metallization - Lift-off and annealing



Picture of diamond sensor after first evaporation and lift-off

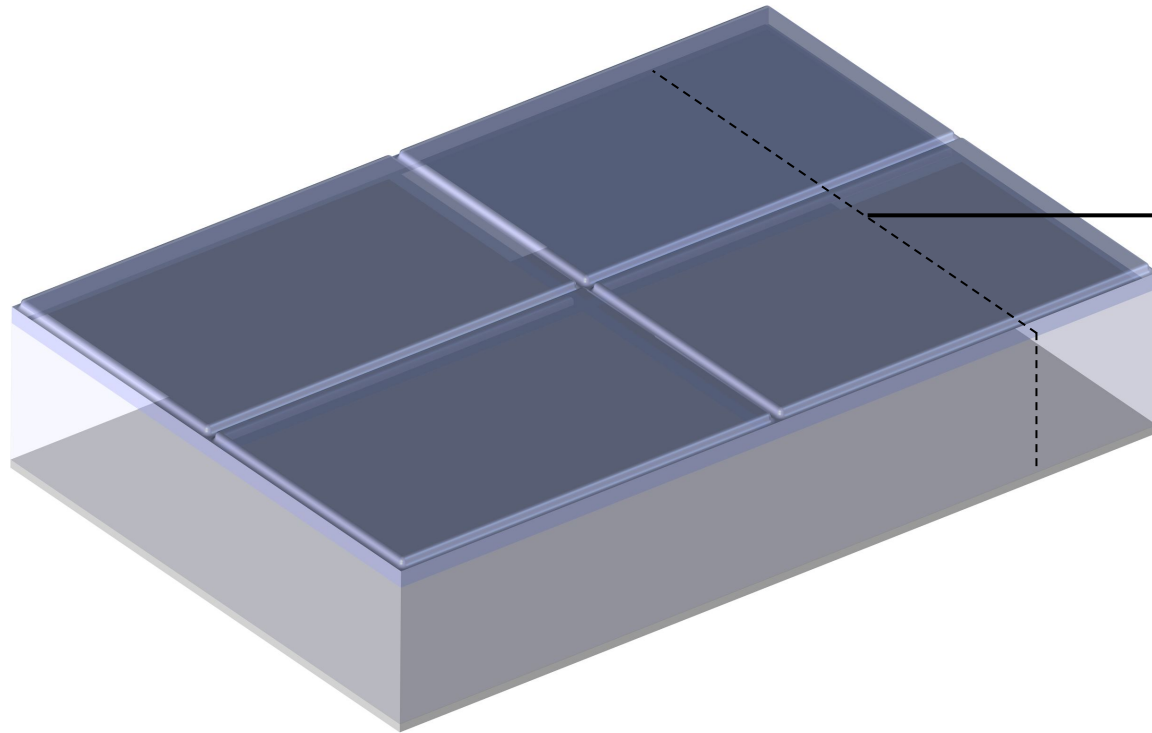


Sensor slice view - thickness (vertical) scaled by 10x)

- Negative Photoresist mask ($\sim 1\mu\text{m}$ thick by spinning) for pixel delimitation
- Deposition of 10nm of Ti and 300nm of Al
- Photoresist and excess metal lift-off using solvents
- 400°C annealing with Ar \rightarrow carbide binding (C-Ti) for ohmic contact formation on the diamond



2. Passivation layer - Plasma enhanced chemical vapor deposition (peCVD) of SiO_xN_y



Slice view



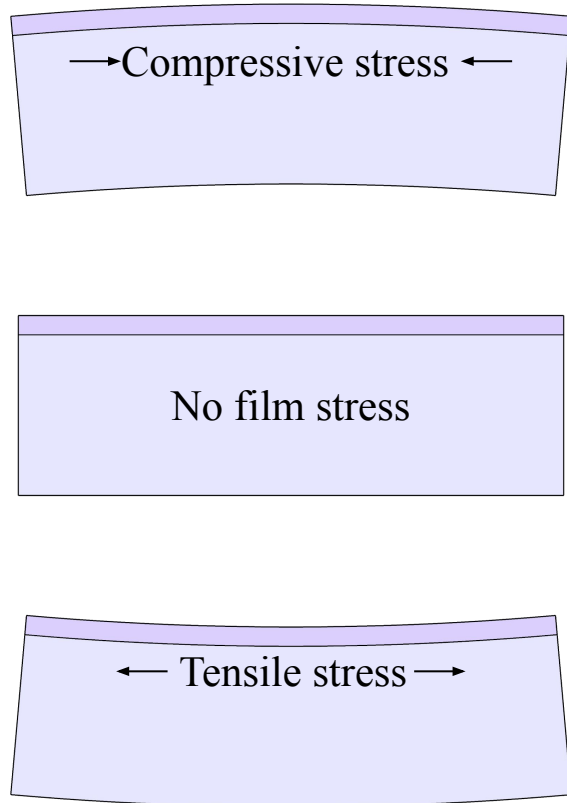
Sensor slice view - thickness (vertical) scaled by 10x

- 600nm low-stress film deposition of SiO_xN_y covering all the sensor's surface

Sensor top surface view - thickness scaled by 10x



2. Passivation layer - Plasma enhanced chemical vapor deposition (peCVD) of SiO_xN_y

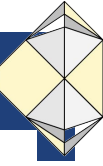


Remanent stress can crack the deposited SiON film and also the metallic layers below → Low stress is desired for our detector

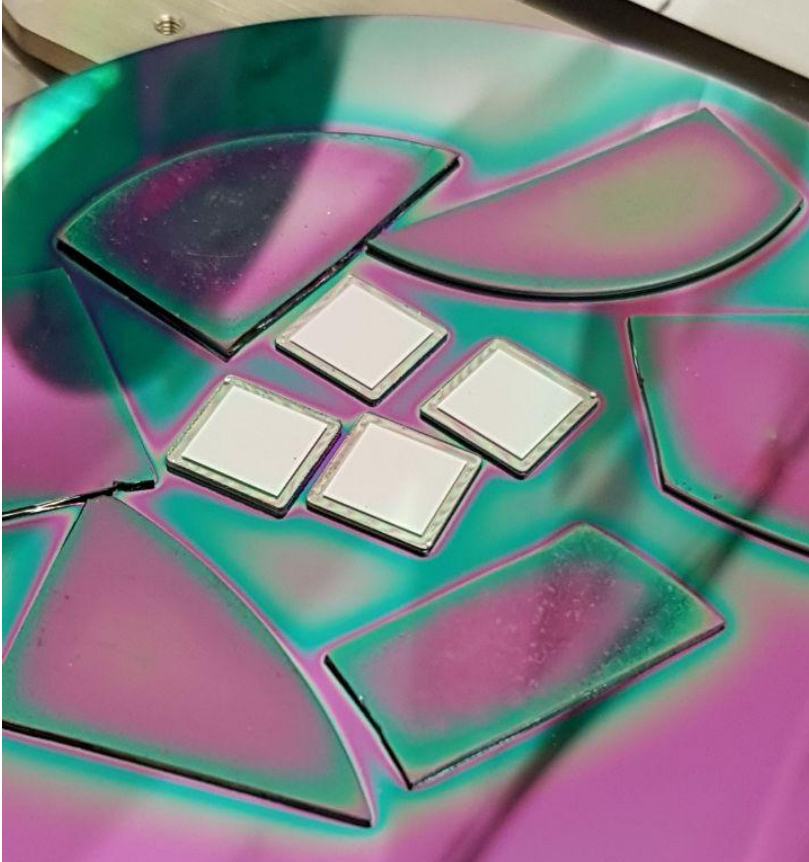


Sensor slice view - thickness (vertical) scaled by 10x)

- 600nm low-stress film deposition of SiO_xN_y covering all the sensor's surface



2. Passivation layer - Plasma enhanced chemical vapor deposition (peCVD) of SiO_xN_y

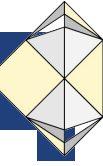


Picture of 4 diamond samples and silicon carriers after SiON deposition

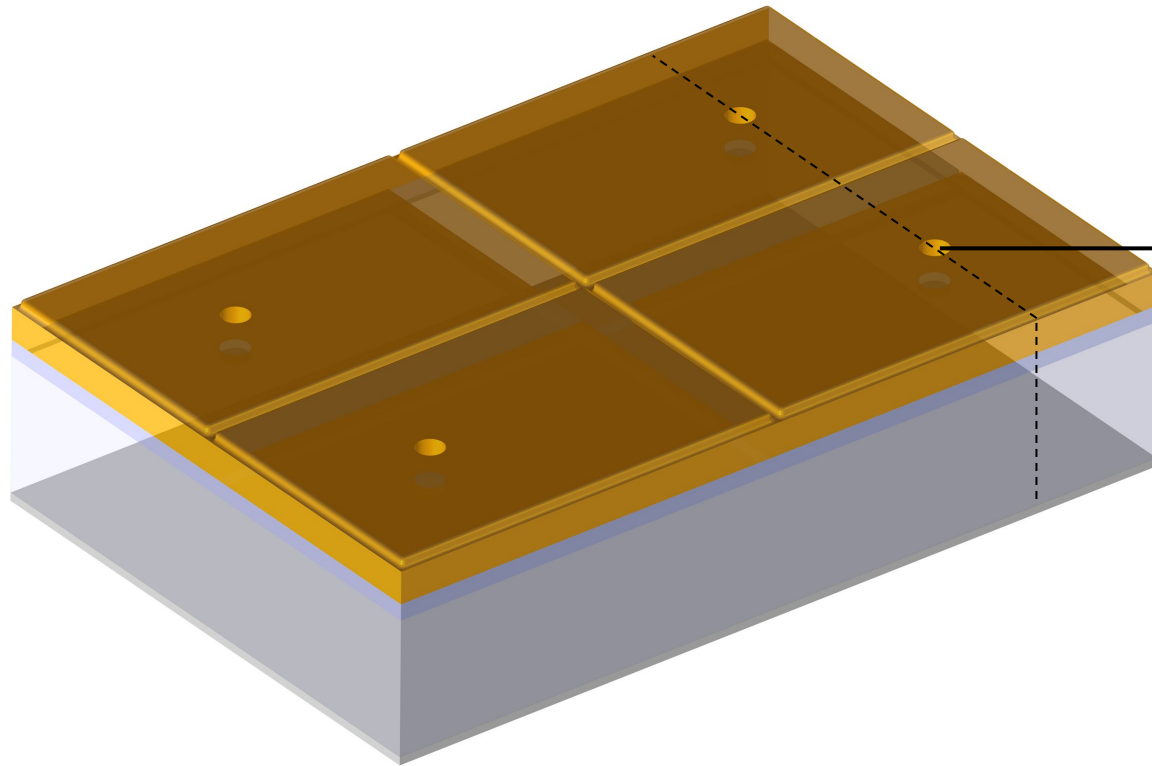


Sensor slice view - thickness (vertical) scaled by 10x)

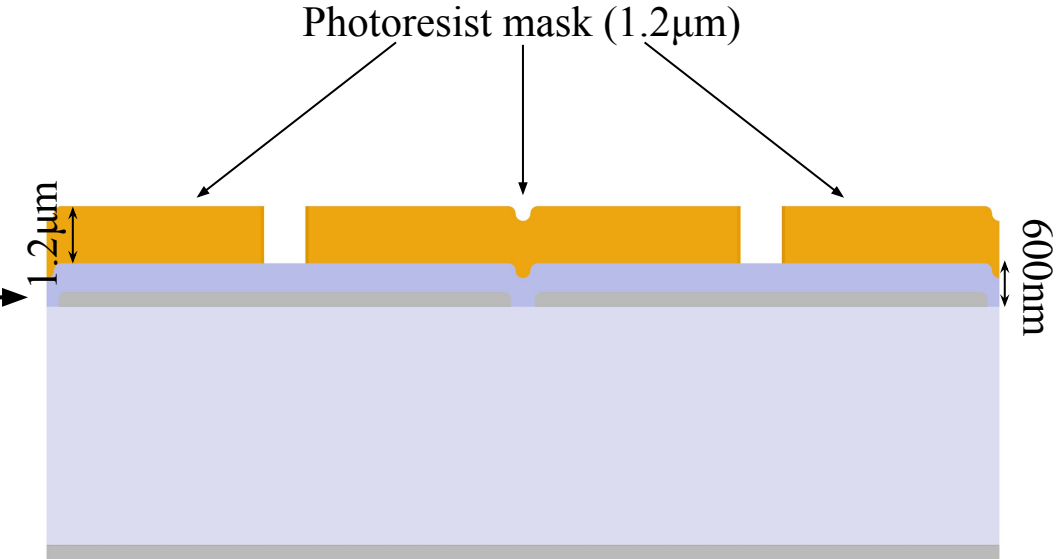
- 600nm low-stress film deposition of SiO_xN_y covering all the sensor's surface



2. Passivation layer - Reactive ion etching (RIE) through passivation layer



Slice view



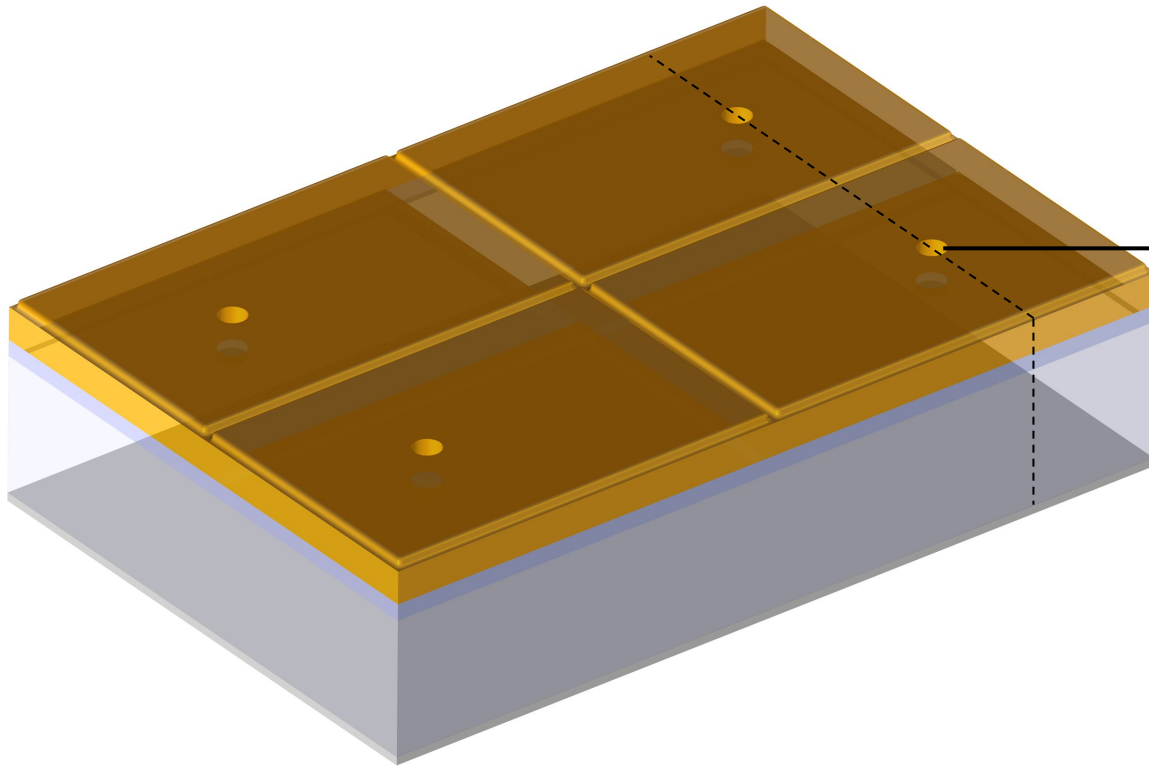
Sensor slice view - thickness (vertical) scaled by 10x)

- 600nm low-stress film deposition of SiO_xN_y covering all the sensor's surface
- Dry etching with a reactive plasma of CHF_3 until the metallization is reached

Sensor top surface view - thickness scaled by 10x



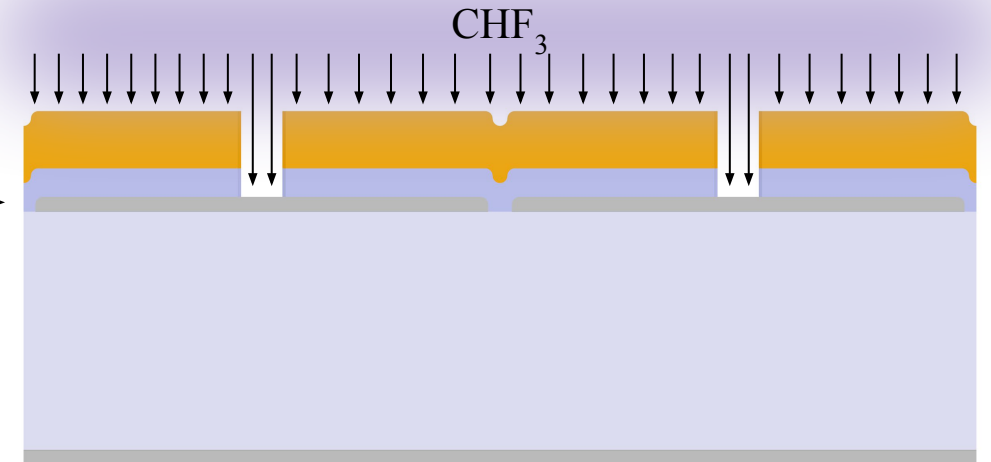
2. Passivation layer - Reactive ion etching (RIE) through passivation layer



Sensor top surface view - thickness scaled by 10x



Slice view →

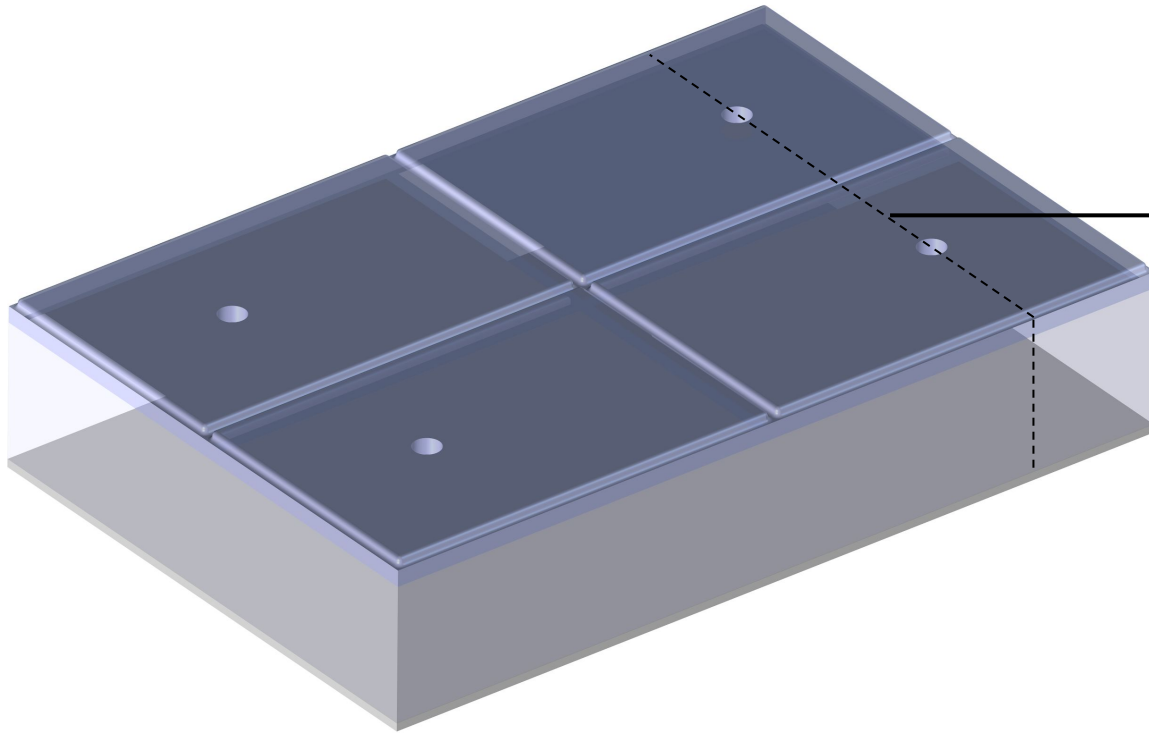


Sensor slice view - thickness (vertical) scaled by 10x)

- 600nm low-stress film deposition of SiO_xN_y covering all the sensor's surface
- Dry etching with a reactive plasma of CHF_3 until the metallization is reached



2. Passivation layer - Photoresist removal



Sensor top surface view - thickness scaled by 10x

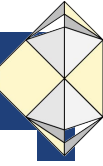


Slice view →



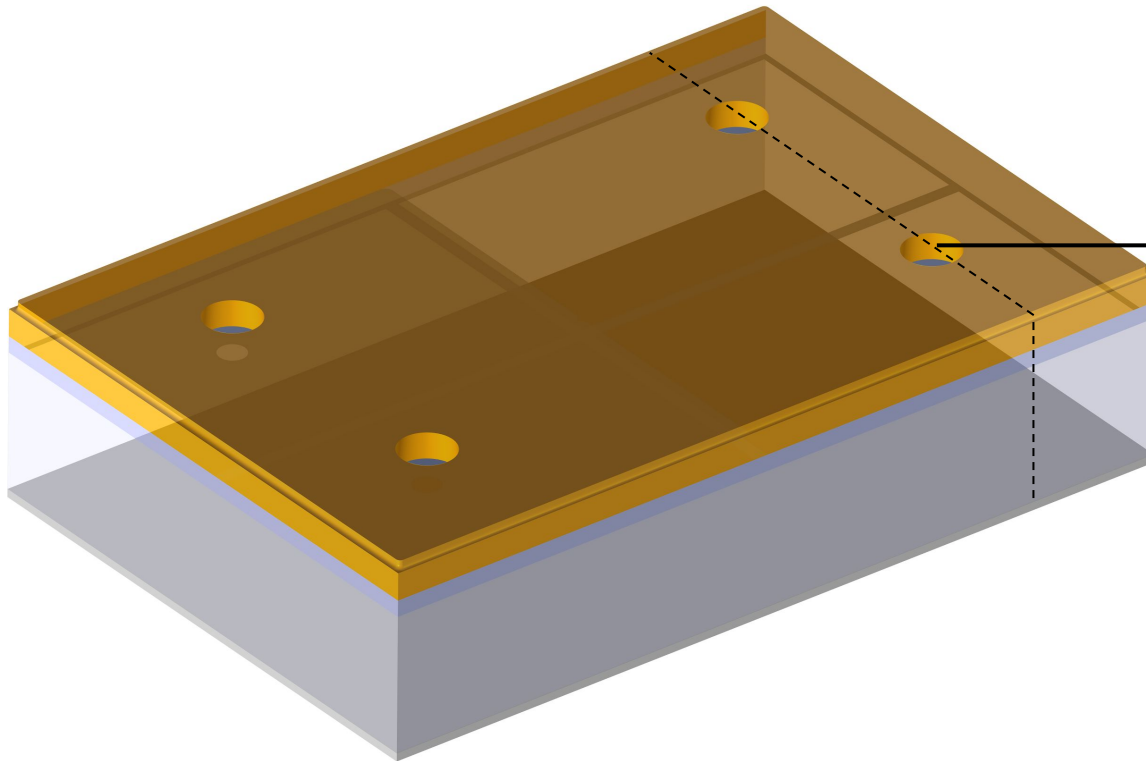
Sensor slice view - thickness (vertical) scaled by 10x)

- 600nm low-stress film deposition of SiO_xN_y covering all the sensor's surface
- Dry etching with a reactive plasma of CHF_3 until the metallization is reached
- Photoresist stripping with solvents

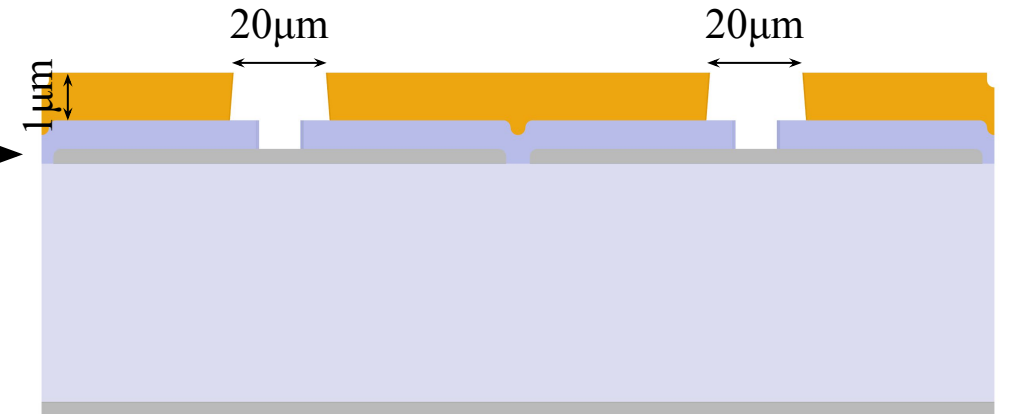


3. Under bump metallization (UBM) - Photoresist mask

→ Metallization required for bumps to stick in the correct position



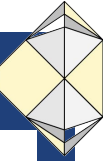
Slice view →



Sensor slice view - thickness (vertical) scaled by 10x)

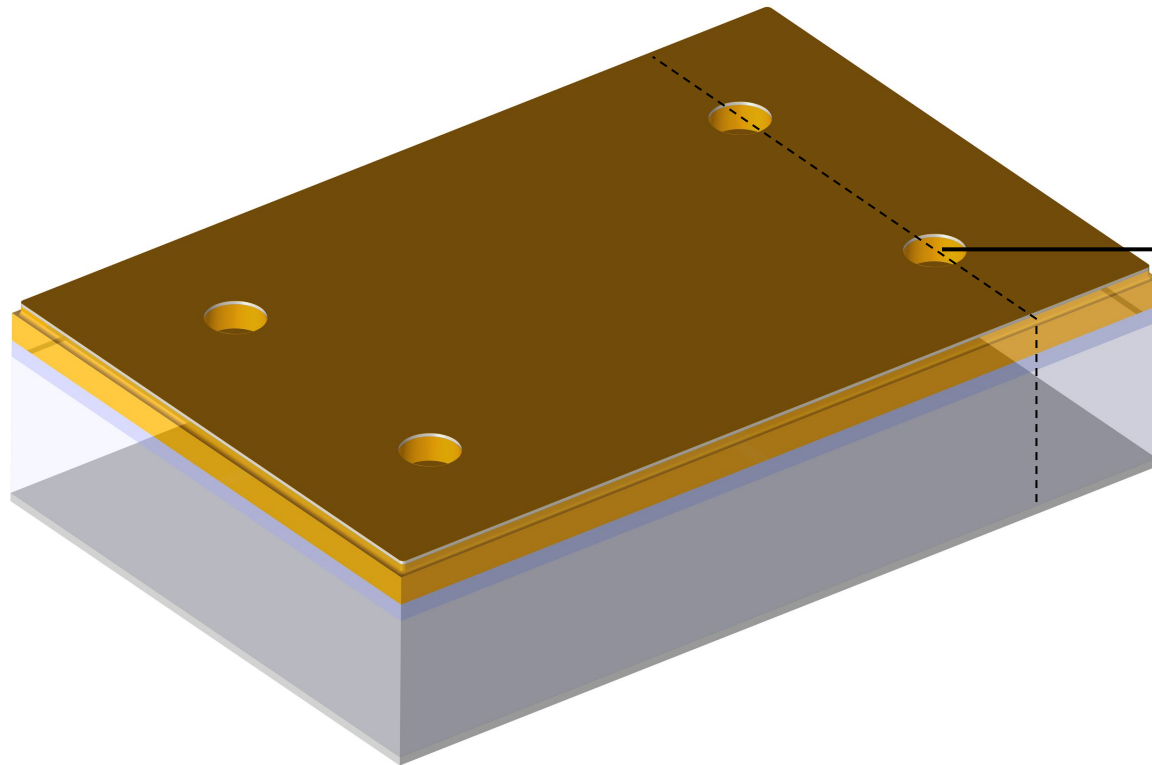
- Negative Photoresist mask ($\sim 1\mu\text{m}$ thick by spinning) for UBM delimitation

Sensor top surface view - thickness scaled by 10x

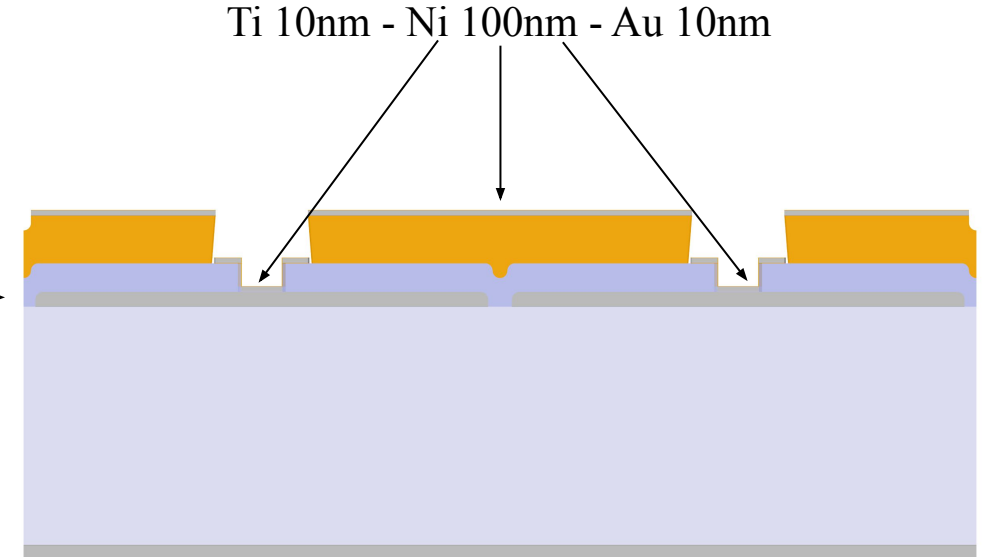


3. Under bump metallization (UBM) - Metal deposition (electron beam evaporation)

Metallization required for bumps to stick in the correct position



Slice view



Sensor slice view - thickness (vertical) scaled by 10x

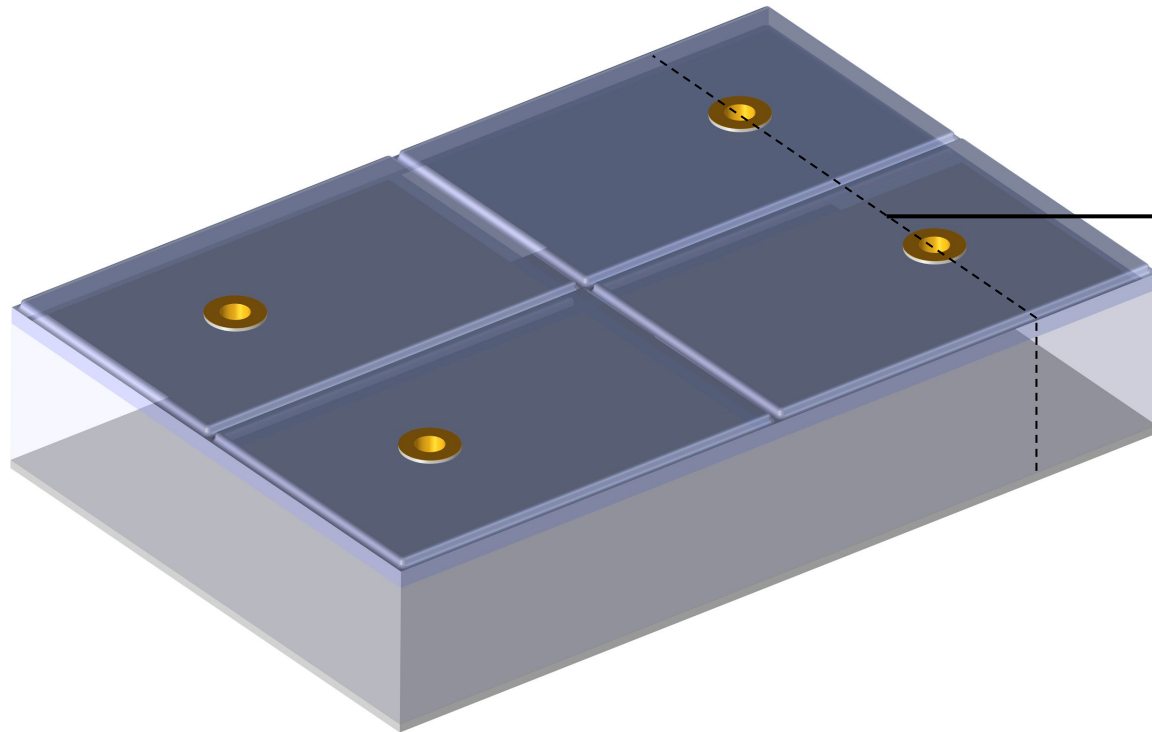
- Negative Photoresist mask ($\sim 1\mu\text{m}$ thick by spinning) for UBM delimitation
- Deposition of 10nm of Ti, 100nm of Ni and 10nm of Au

Sensor top surface view - thickness scaled by 10x



3. Under bump metallization (UBM) - Lift-off

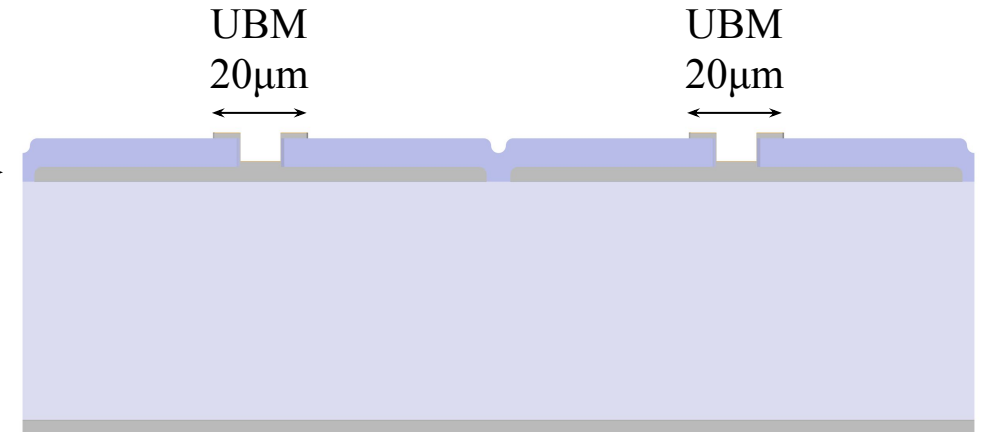
Metallization required for bumps to stick in the correct position



Sensor top surface view - thickness scaled by 10x



Slice view

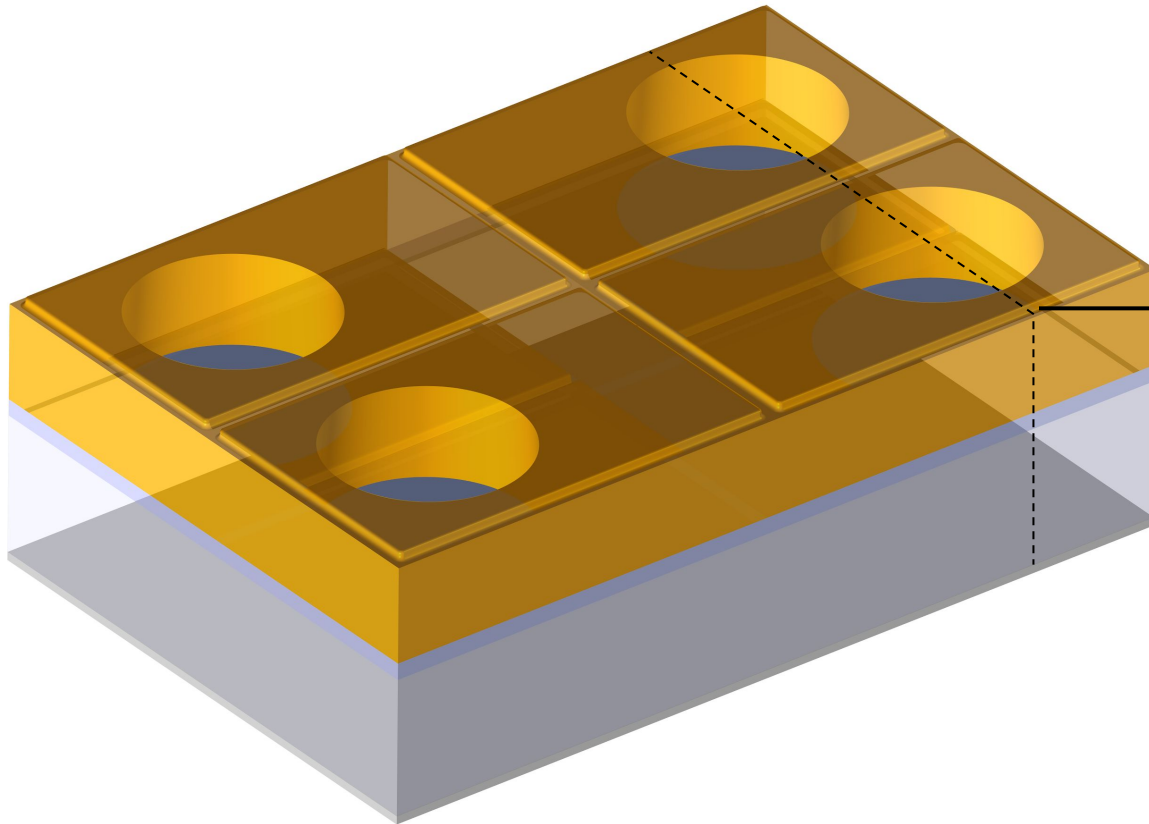


Sensor slice view - thickness (vertical) scaled by 10x)

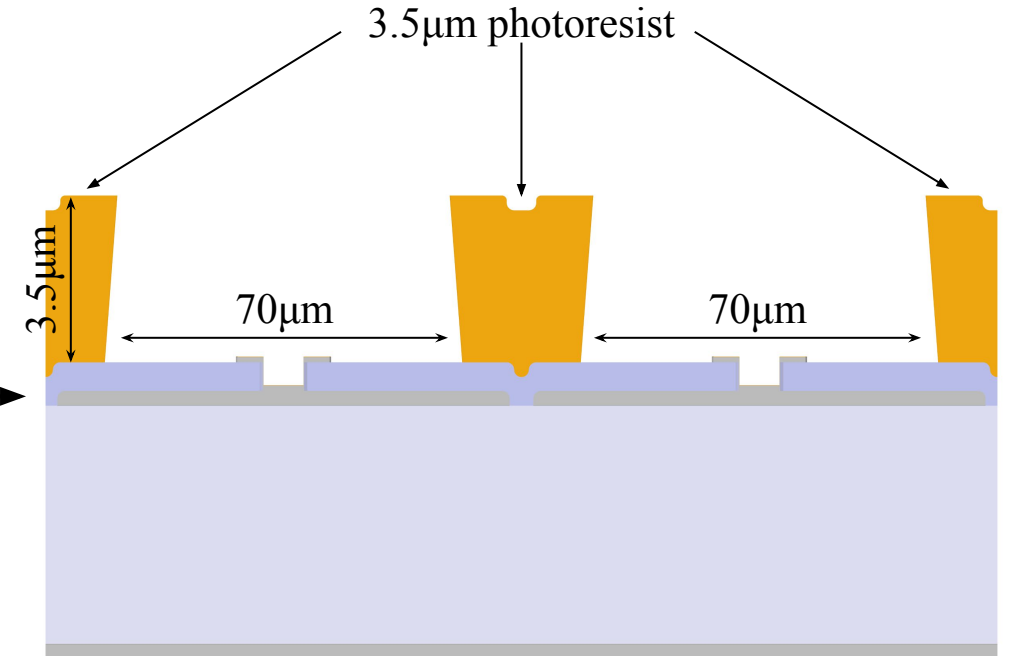
- Negative Photoresist mask ($\sim 1\mu\text{m}$ thick by spinning) for UBM delimitation
- Deposition of 10nm of Ti, 100nm of Ni and 10nm of Au
- Photoresist and excess metal lift-off using solvents
- UBM is required for correct bump formation



4. Indium bumps - Photoresist mask



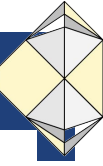
Slice view



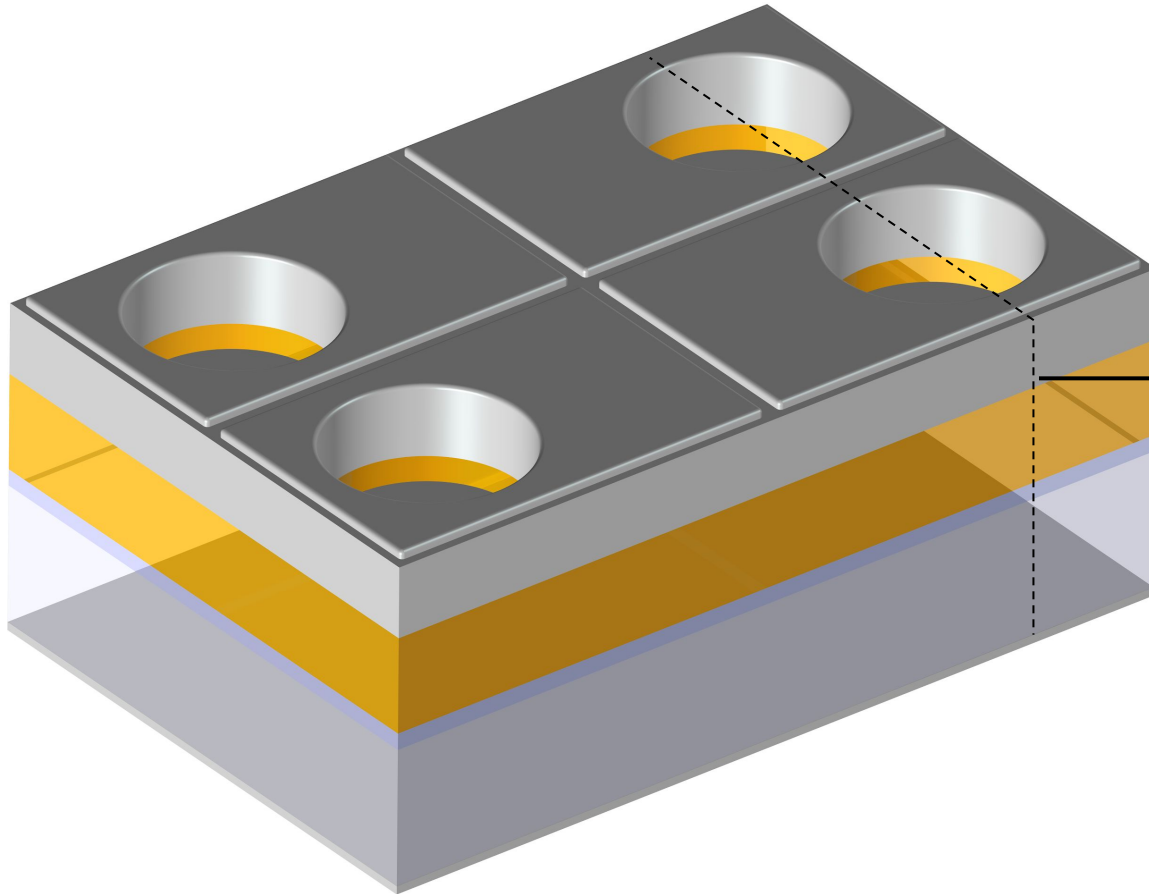
Sensor slice view - thickness (vertical) scaled by 10x

- Negative Photoresist mask ($\sim 3.5\mu\text{m}$ thick by spinning) for In delimitation

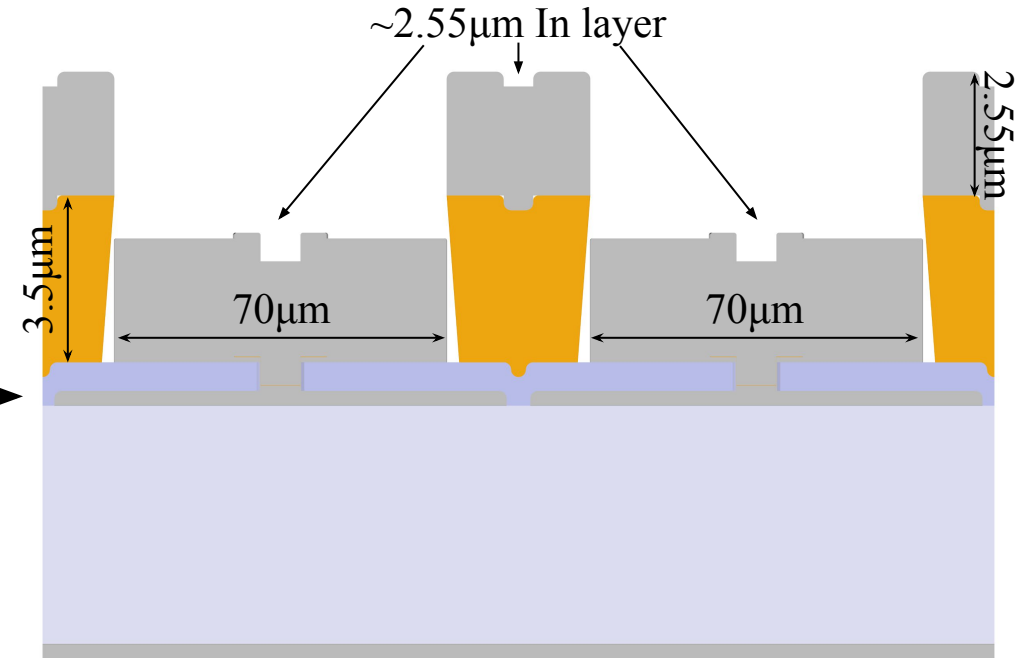
Sensor top surface view - thickness scaled by 10x



4. Indium bumps - Indium evaporation



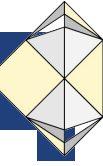
Slice view



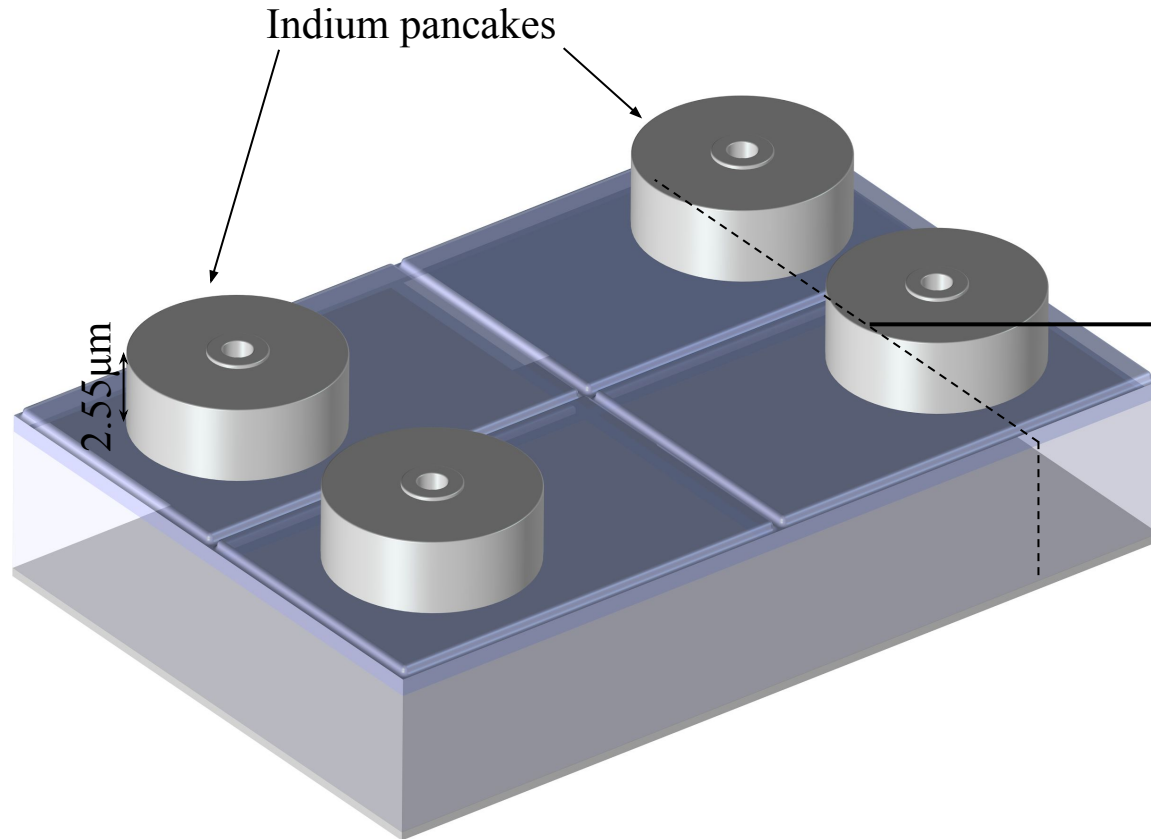
Sensor slice view - thickness (vertical) scaled by 10x

- Negative Photoresist mask ($\sim 3.5\mu\text{m}$ thick by spinning) for In delimitation
- Deposition of $2.55\mu\text{m}$ of In (3g)

Sensor top surface view - thickness scaled by 10x



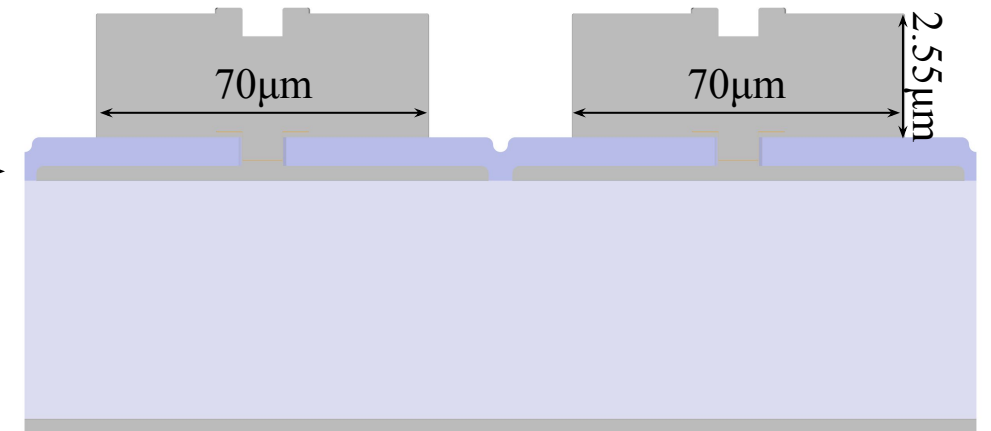
4. Indium bumps - Lift-off; Indium pancakes formation



Sensor top surface view - thickness scaled by 10x



Slice view →

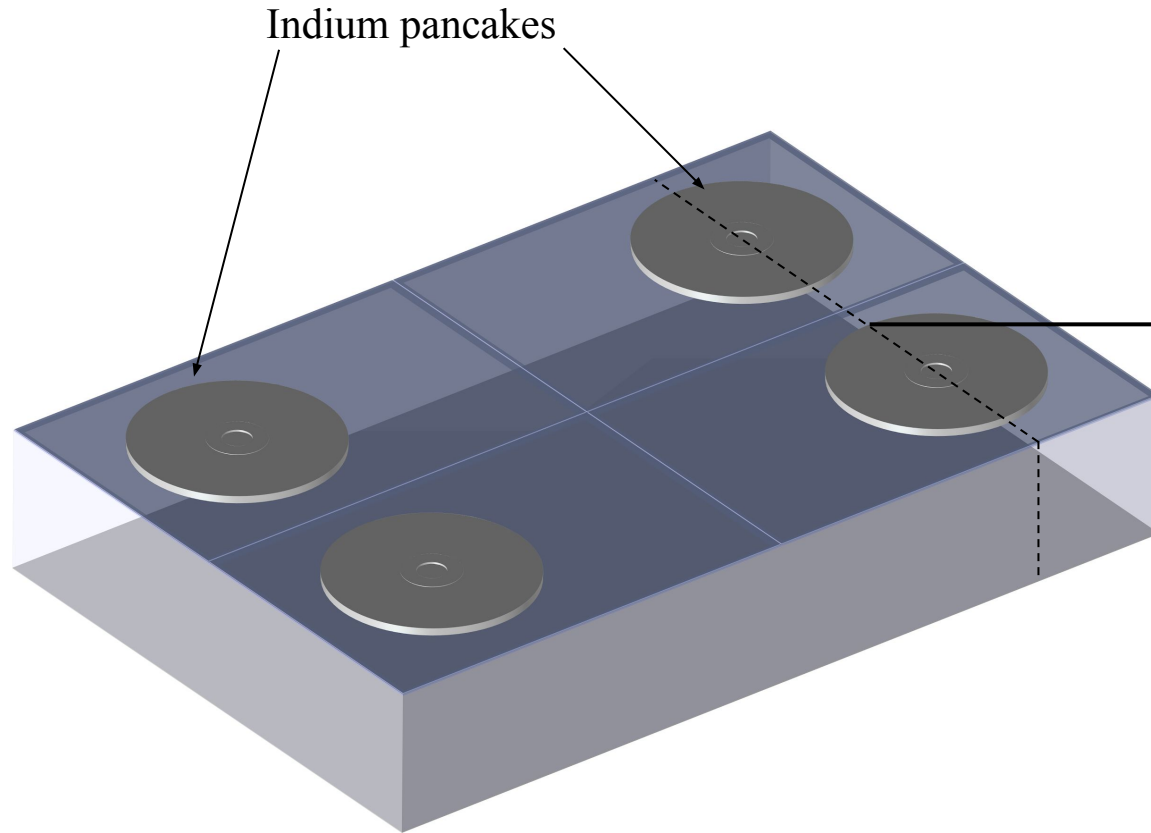


Sensor slice view - thickness (vertical) scaled by 10x

- Negative Photoresist mask ($\sim 3.5\mu\text{m}$ thick by spinning) for In delimitation
- Deposition of $2.55\mu\text{m}$ of In (3g)
- Photoresist and excess metal lift-off using solvents



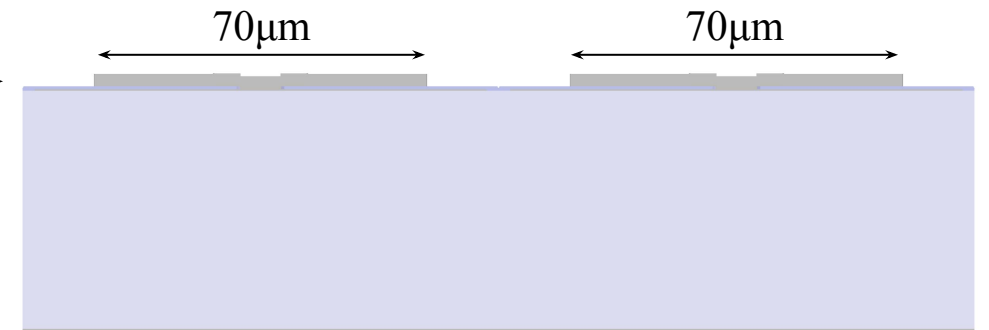
4. Indium bumps - Lift-off; Indium pancakes formation



Sensor top surface view (actual thickness)



Slice view

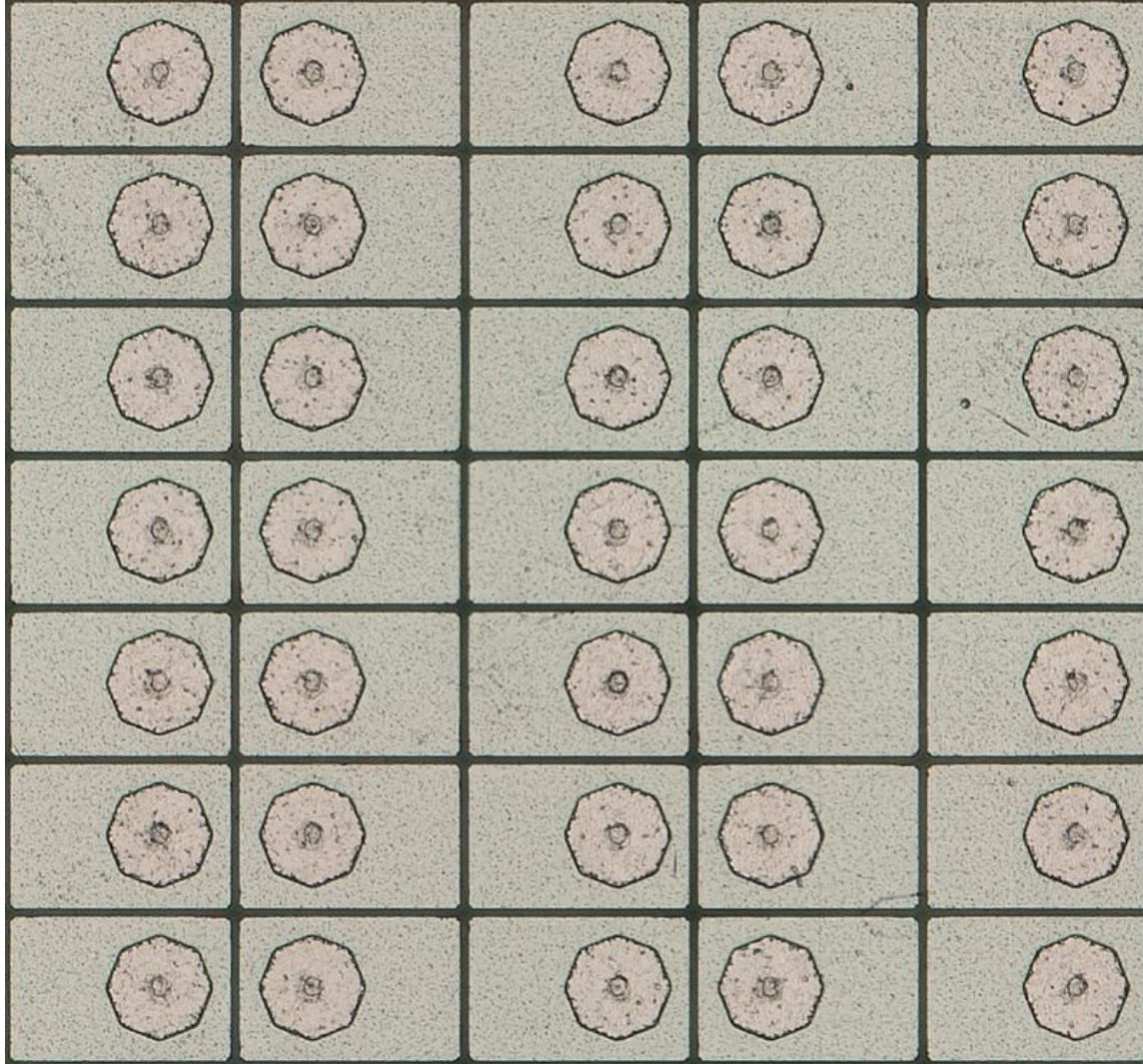


Sensor slice view (actual thickness)

- Negative Photoresist mask ($\sim 3.5\mu\text{m}$ thick by spinning) for In delimitation
- Deposition of $2.55\mu\text{m}$ of In (3gr)
- Photoresist and excess metal lift-off using solvents
- Indium pancakes formation before reflow



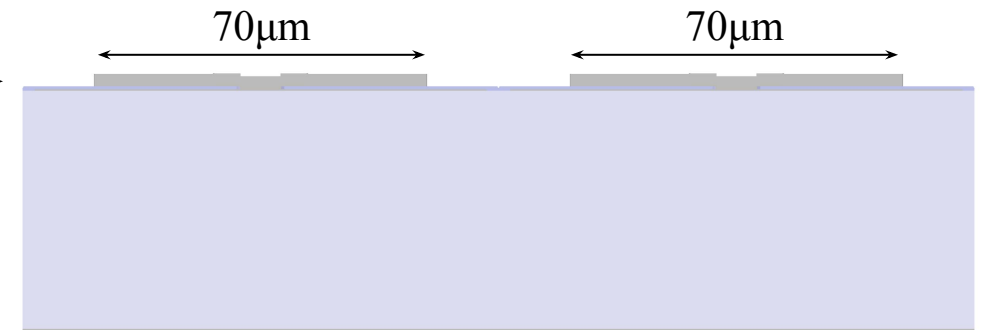
4. Indium bumps - Lift-off; Indium pancakes formation



Microscope picture before first reflow

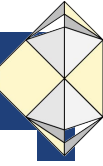


Slice view →



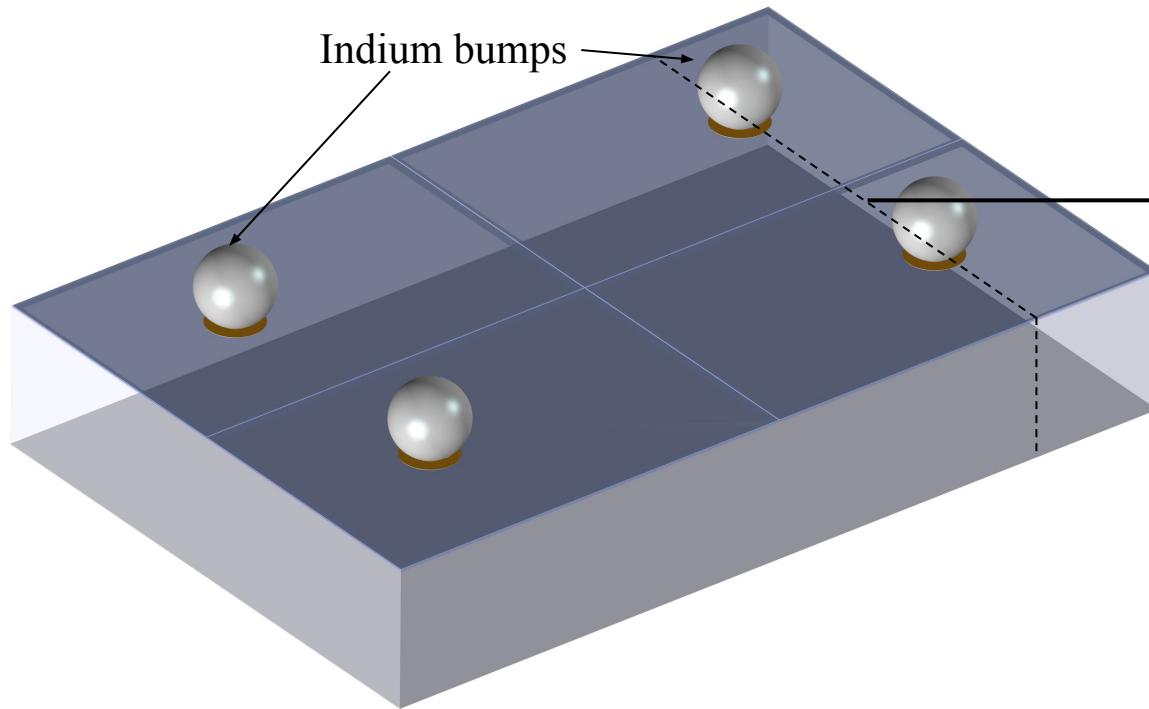
Sensor slice view (actual thickness)

- Negative Photoresist mask ($\sim 3.5\mu\text{m}$ thick by spinning) for In delimitation
- Deposition of $2.55\mu\text{m}$ of In (3g)
- Photoresist and excess metal lift-off using solvents
- Indium pancakes formation before reflow

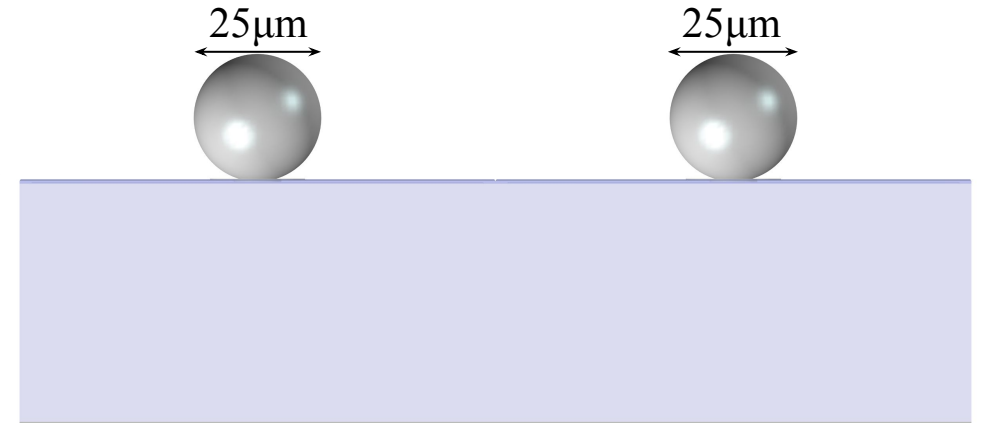


5. Bump bonding - First reflow

Reflow is a process used to form bumps using temperature in a controlled atmosphere



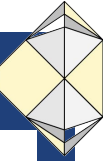
Slice view



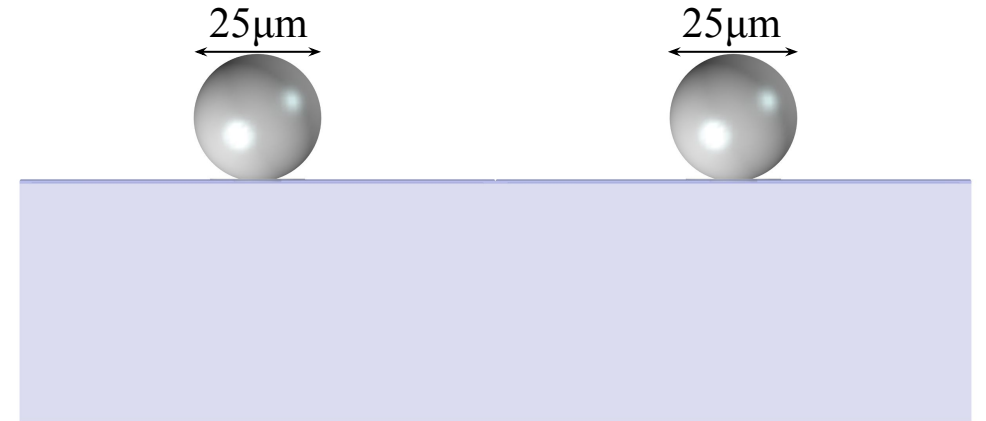
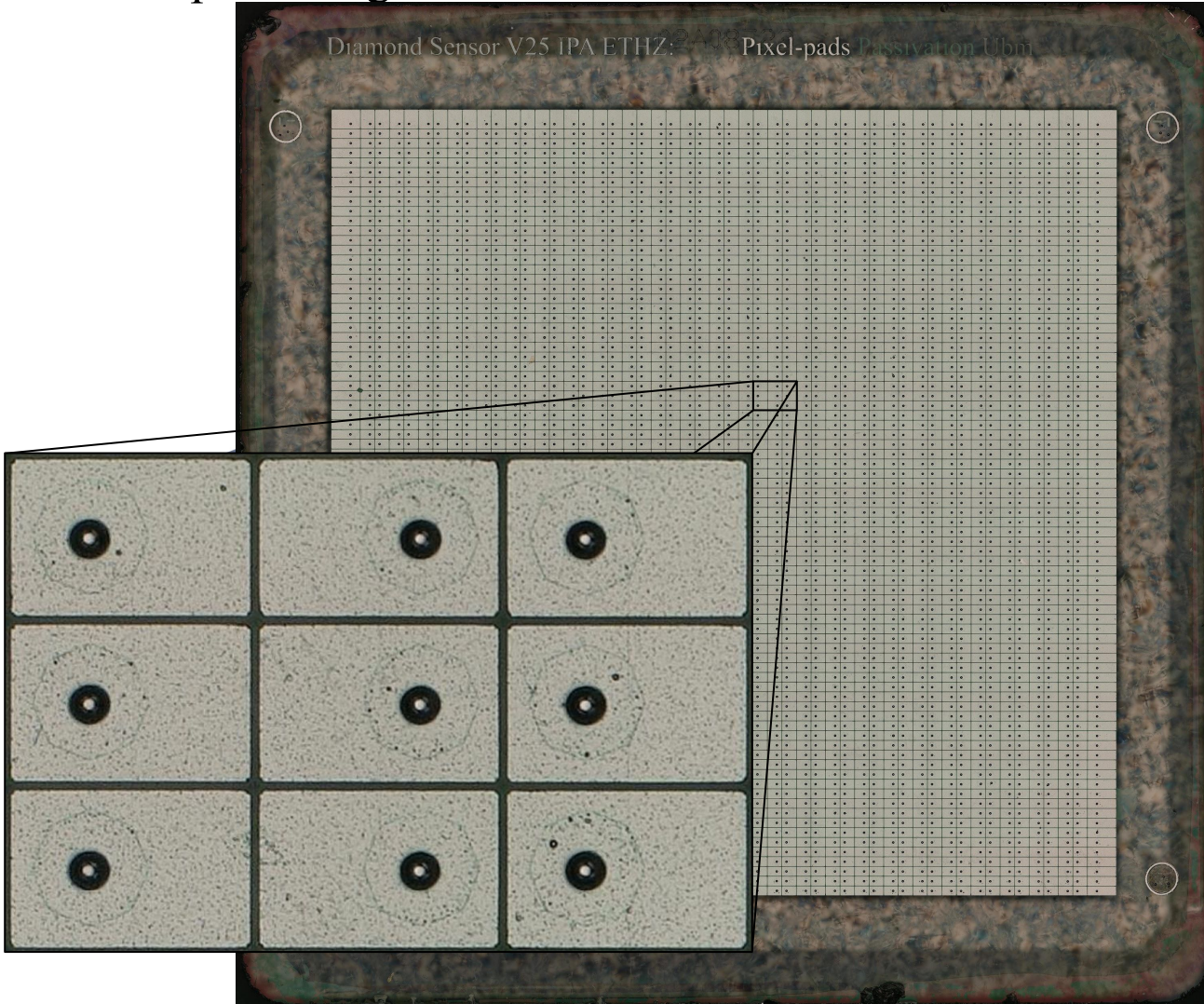
Sensor slice view (actual thickness)

- $\varnothing \sim 25\mu\text{m}$ Indium bump formation through two-step reflow process

Sensor top surface view (actual thickness)



5. Bump bonding - First reflow



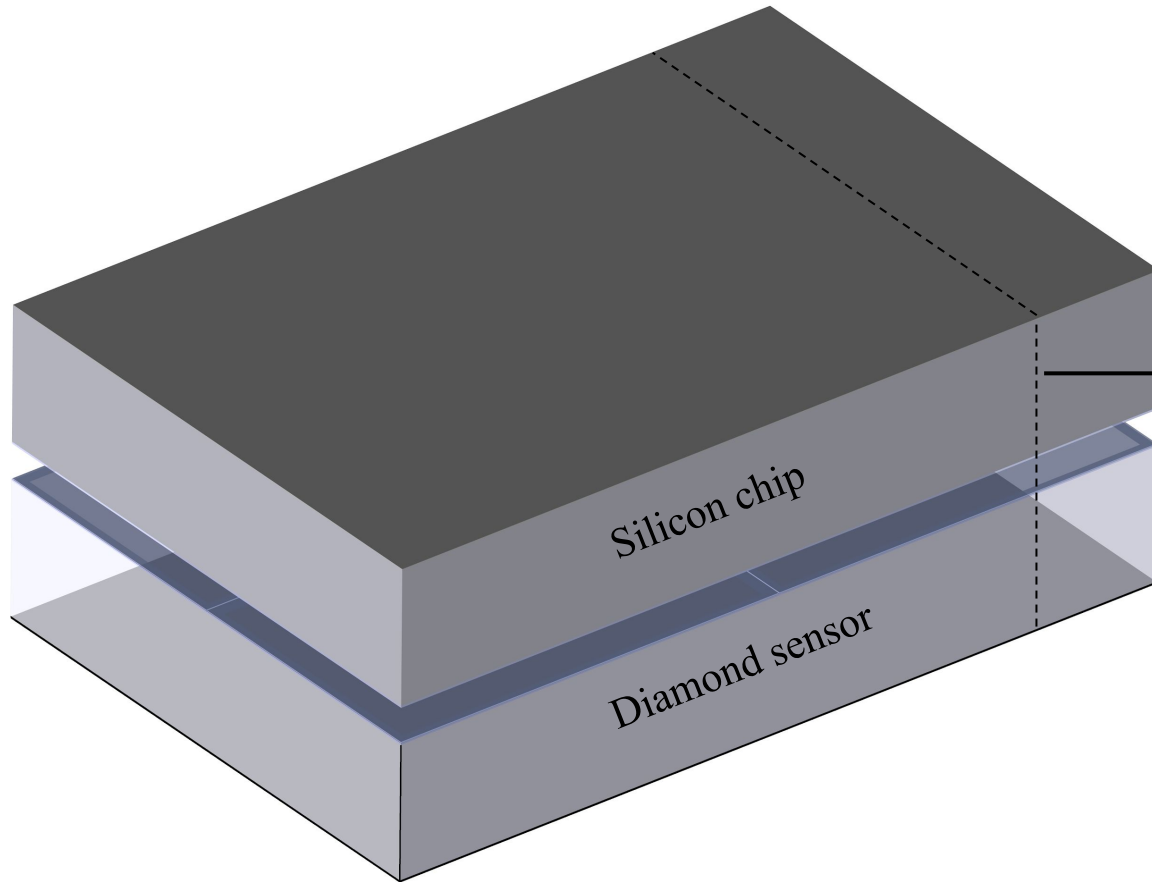
Sensor slice view (actual thickness)

$\varnothing \sim 25\mu\text{m}$ Indium bump formation through two-step reflow process

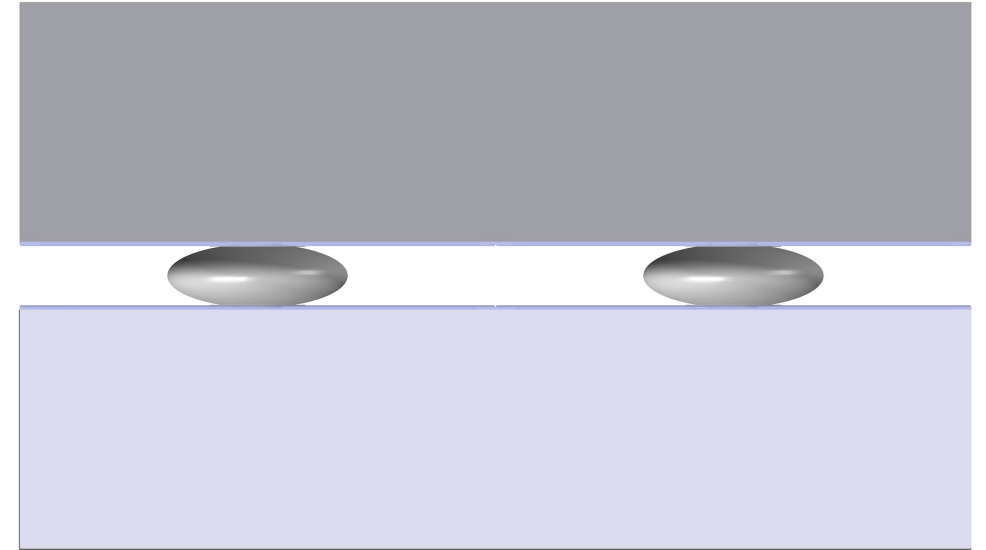
Microscope picture after first reflow: $99.9 \pm 0.1\%$ correct bump formation (31 visually imperfect bumps on 4 detectors)



5. Bump bonding - Flip-chip pixels connection



Slice view



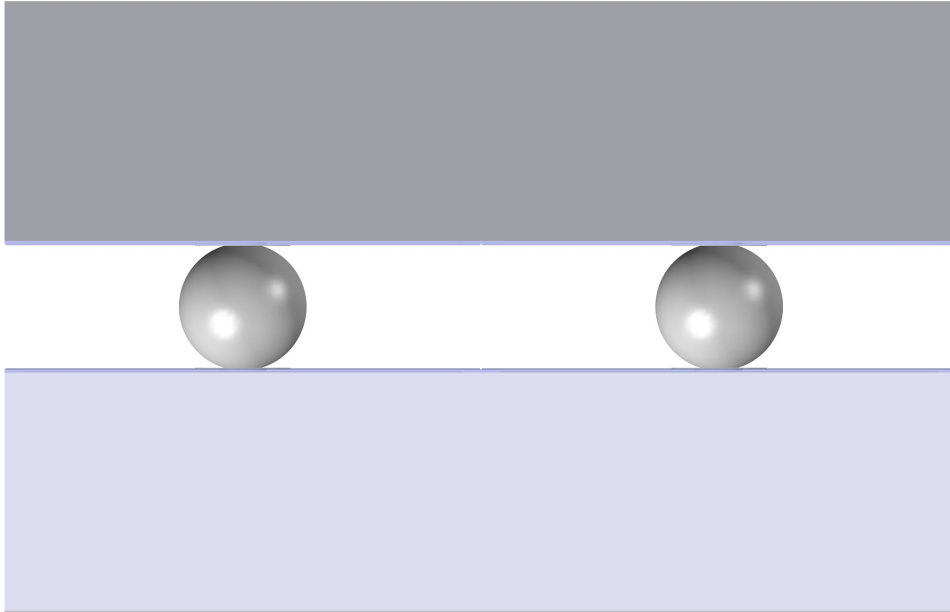
Sensor slice view (actual thickness)

- $\varnothing \sim 25\mu\text{m}$ Indium bump formation through two-step reflow process
- Silicon ROC (psi46digV2.1 respin) pressed with $\sim 4\text{kg}$ (9mN per bump) on the sensor

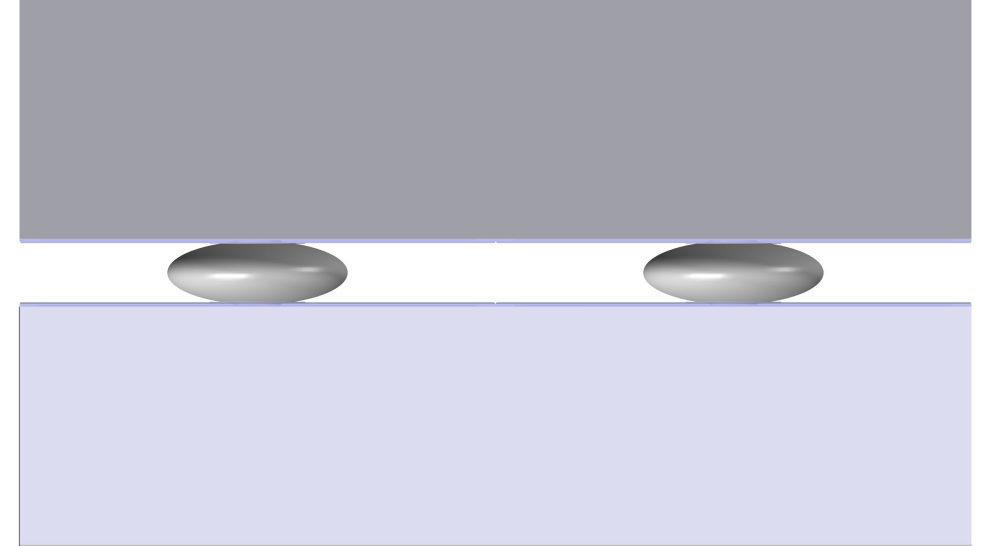
Sensor top surface view (actual thickness)



5. Bump bonding - Second reflow

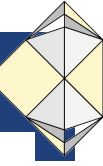


Sensor slice view (actual thickness)

← 2nd Reflow

Sensor slice view (actual thickness)

- $\varnothing \sim 25\mu\text{m}$ Indium bump formation through two-step reflow process
- Silicon ROC (psi46digV2.1 respin) pressed with $\sim 4\text{kg}$ (9mN per bump) on the sensor
- 2nd reflow homogenizes the bump bonding and corrects misalignments



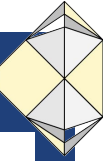
5. Bump bonding - Second reflow



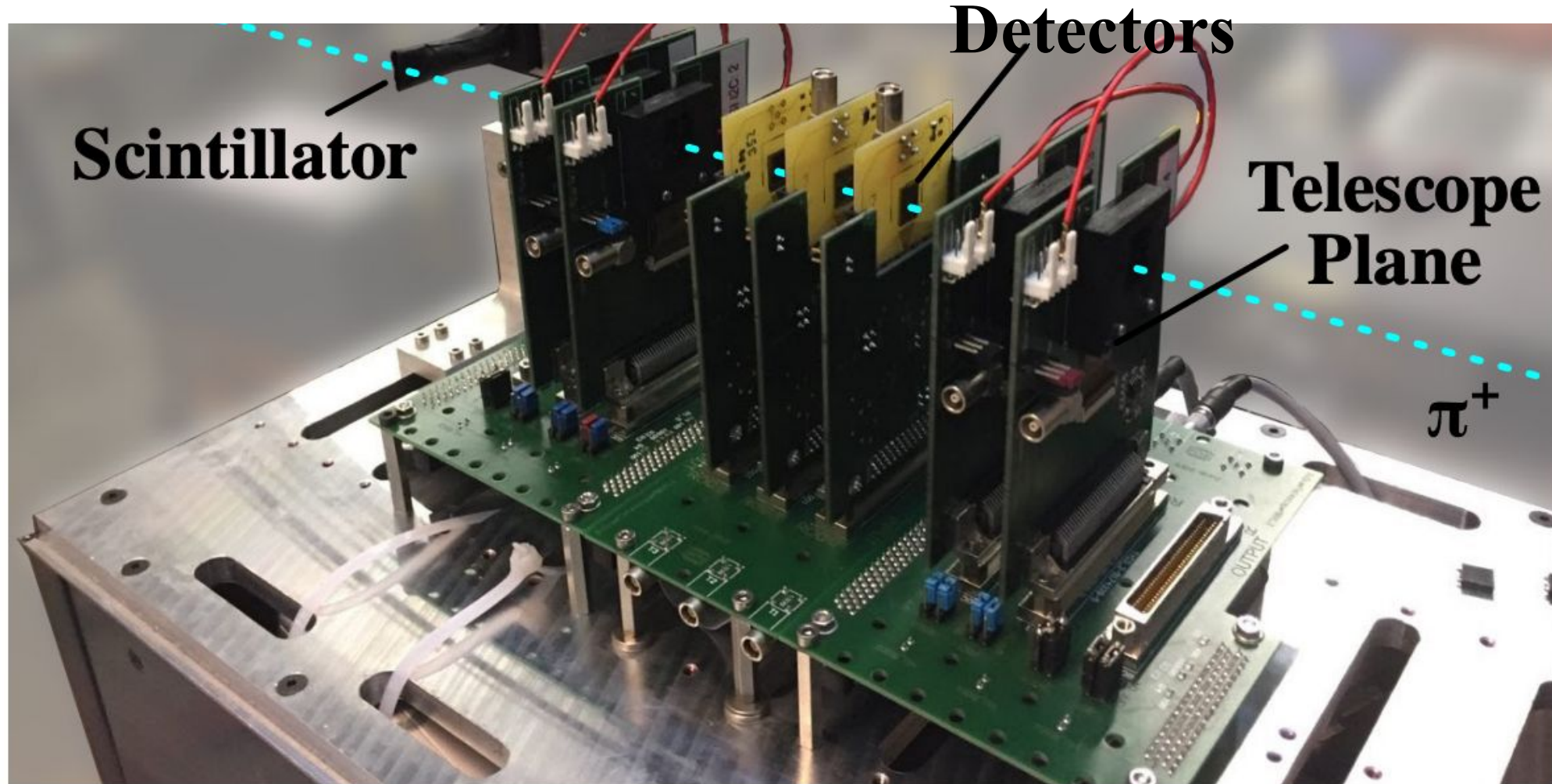
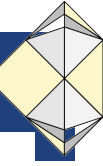
- Glue detector to adaptor board
- Wirebond Readout Chip (ROC) pads to adaptor board
- Wirebond sensor's back-plane to High Voltage line



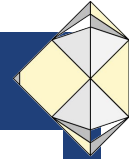
Prototypes 2019



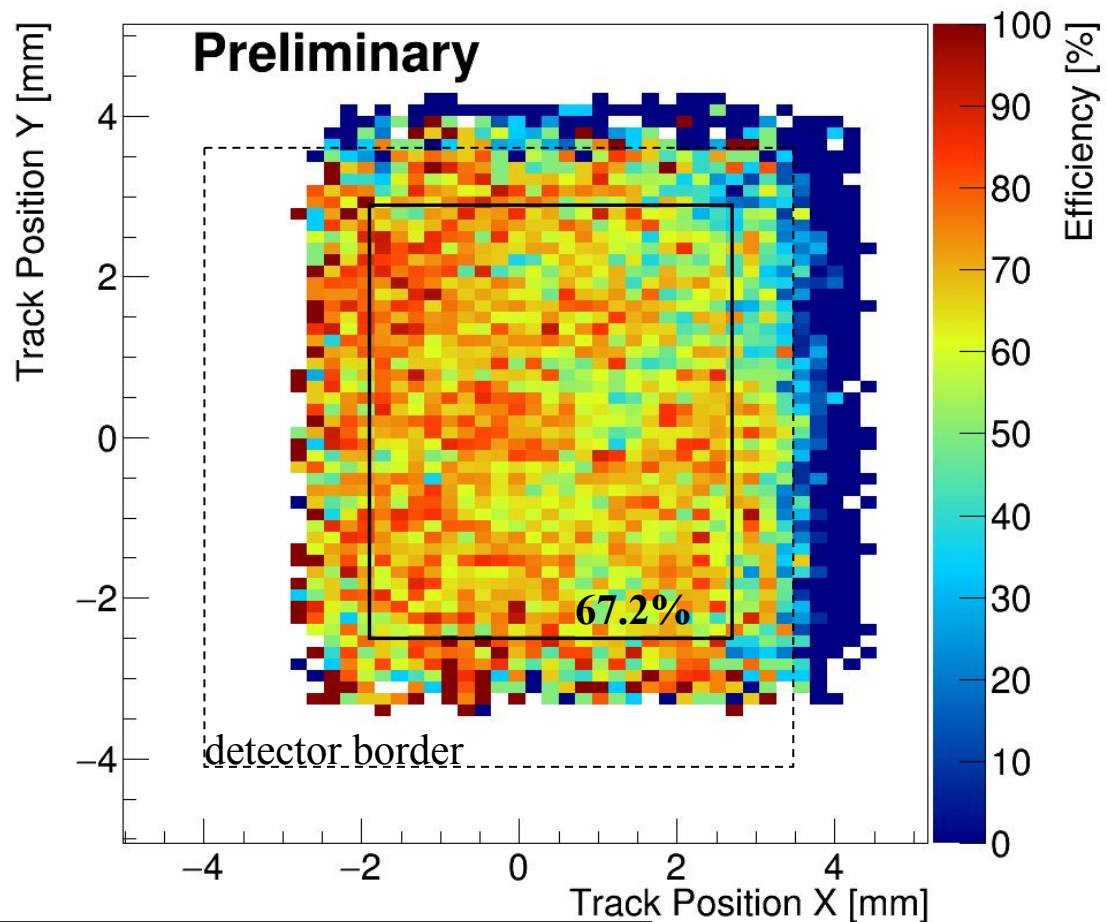
Preliminary results



- ETH Telescope: Consists on 4 CMS analog pixel planes for tracking, a Scintillator for ns timing and up to 3 DUT (any digital detector)
- It has been used over the past 5 years at PSI Pi-M1 beam line (250MeV/c pions)



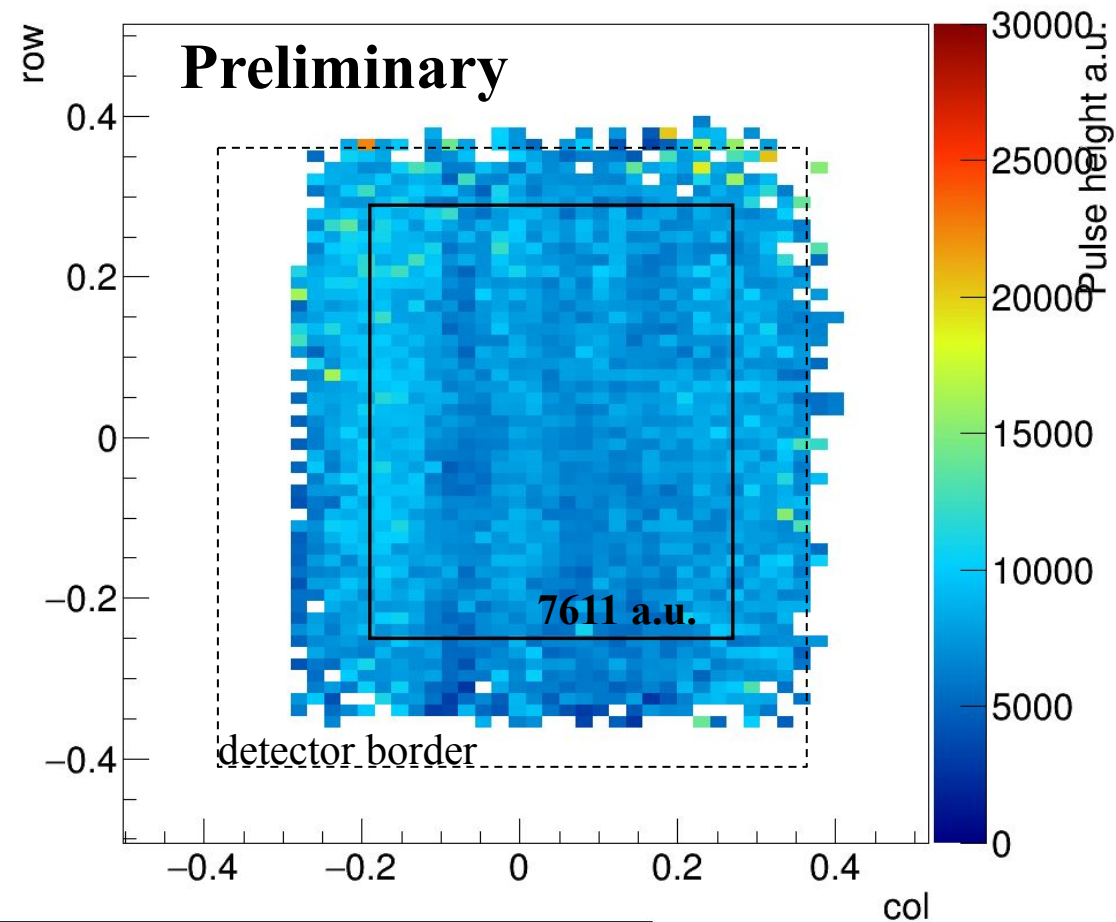
Efficiency Map CMS01



Run 9: 337 kHz/cm², 18159 days, 1:08:57 (451392 evts)
 Detector: CMS01 (September 2019)
 Info: -396V, nonirradiated, Att:

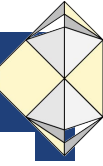
git hash: 9737767

Pulse Height Map Il6-93

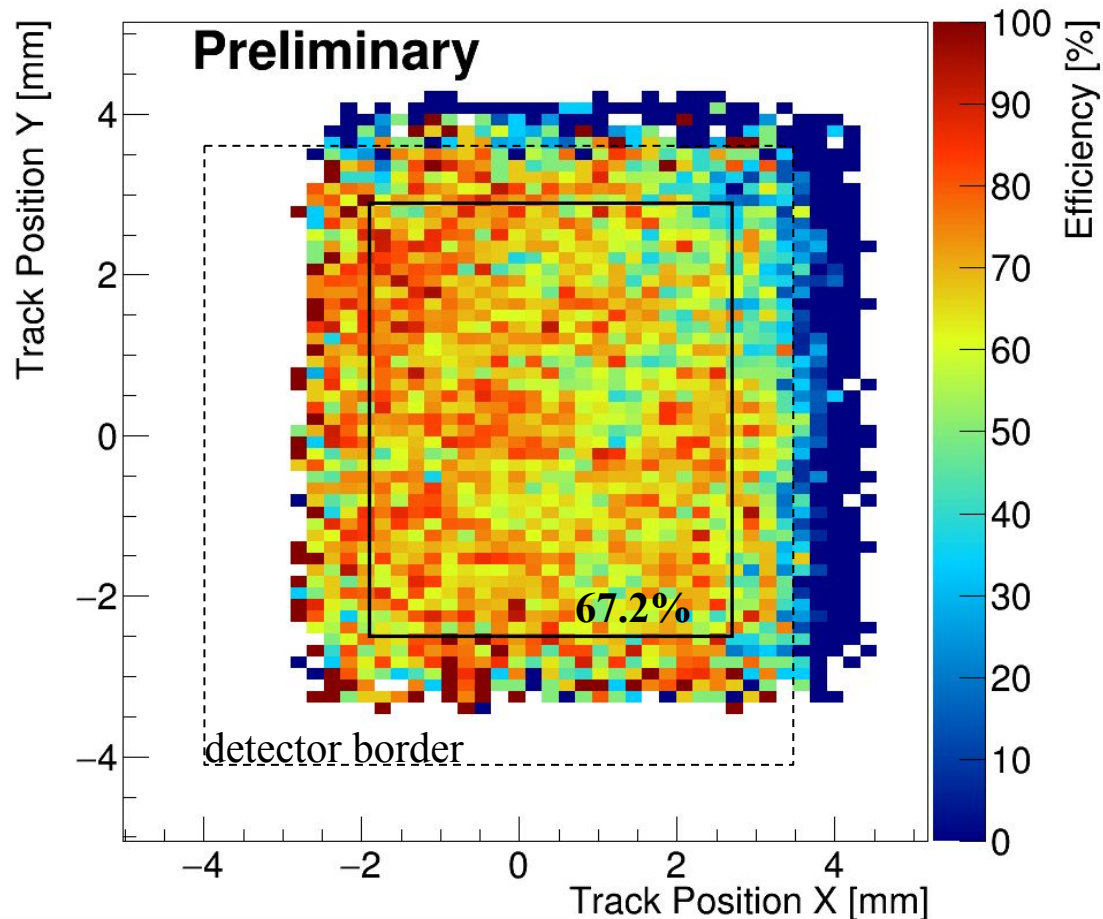


Run 9: 337 kHz/cm², 18159 days, 1:08:57 (451392 evts)
 Detector: Il6-93 (September 2019)
 Info: -700V, nonirradiated, Att:

git hash: 9737767



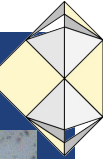
Efficiency Map CMS01



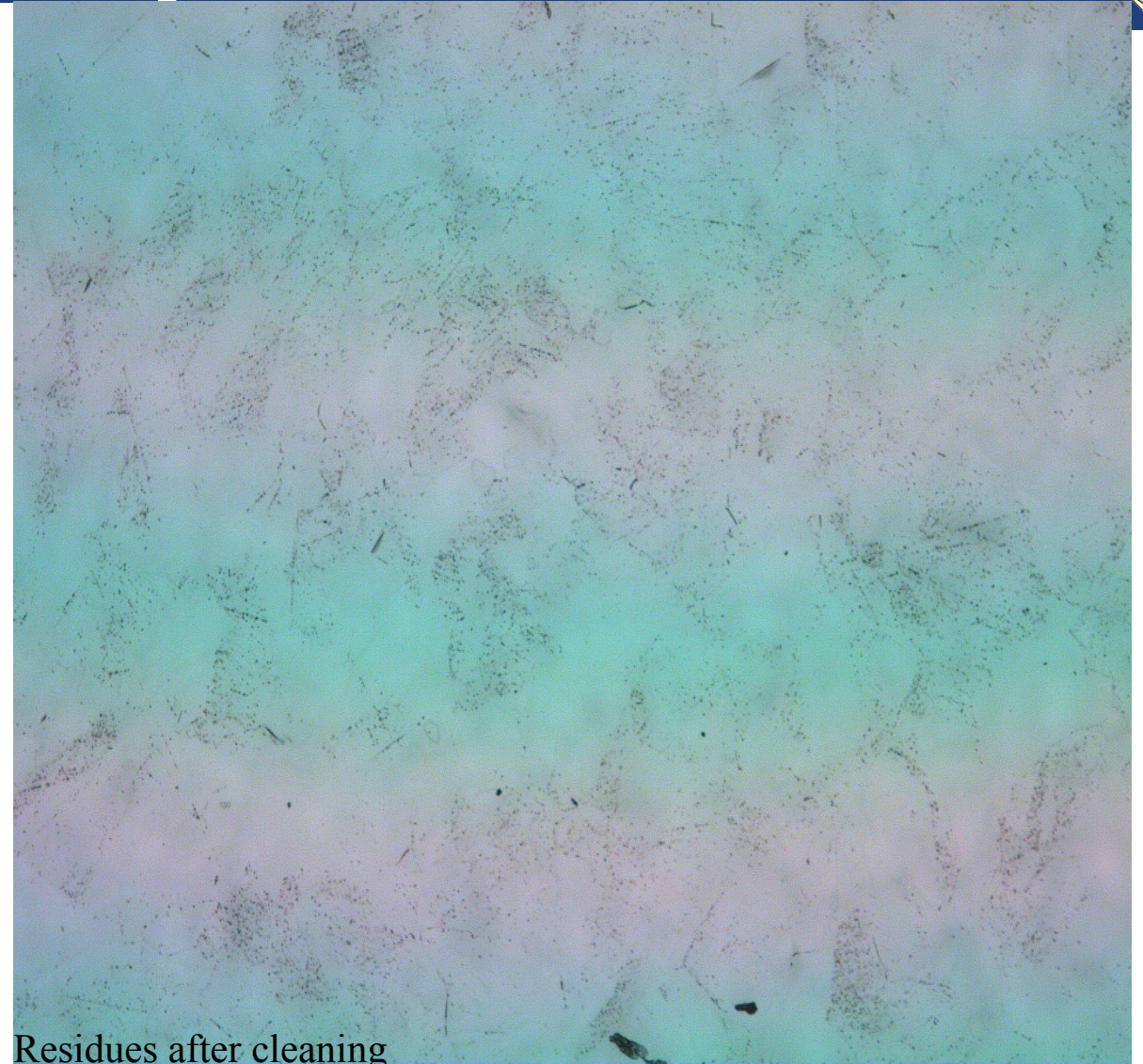
Run 9: 337 kHz/cm², 18159 days, 1:08:57 (451392 evts)
 Detector: CMS01 (September 2019)
 Info: -396V, nonirradiated, Att:

git hash: 9737767

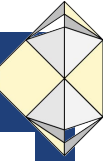
- As it is a polycrystalline diamond, the charge collection is at least 50% compared to a single crystal diamond
- Grain boundaries and lattice defects trap charges in different location in the diamond
- Different efficiency regions could be due to:
 - Polycrystalline features
 - Problems during the fabrication process
 - Metallization sometimes lifts from the diamond - Bad contact
 - Embedded features that remain after cleaning procedure in some samples - Improve cleaning
 - Diamond quality
 - Analysis problems
- This questions must be resolved before scaling the fabrication process to a full module (1 sensors with 16 readout chips)



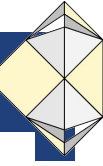
Metallization peeling



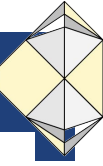
Residues after cleaning



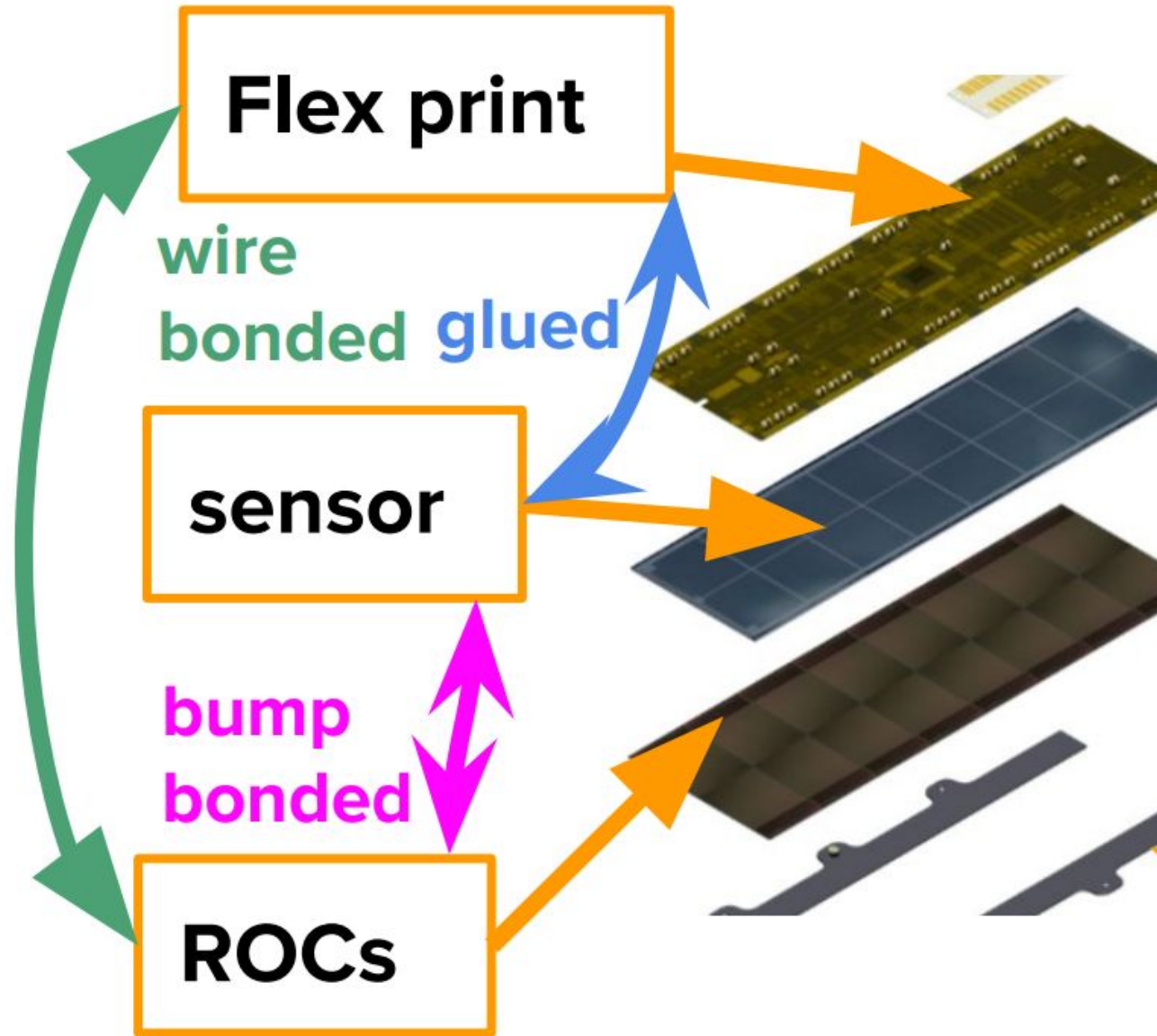
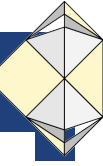
- A fabrication procedure to make diamond planar pixel detectors has been developed
- Five working devices have been fabricated and tested in Testbeams in the past year
- Preliminary results show that there are still issues that have to be solved before moving to the next step
- If successful, the next milestone is a full module fabrication (scaling up the process)

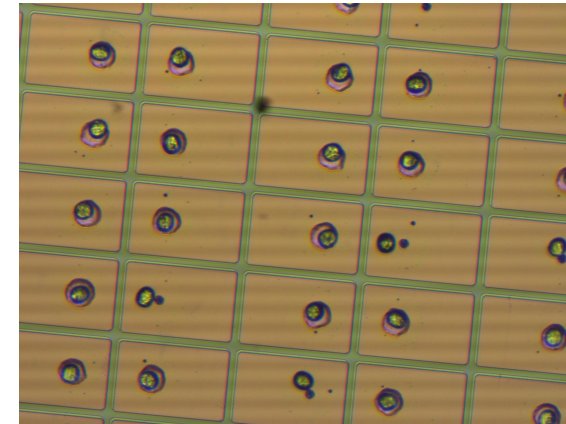
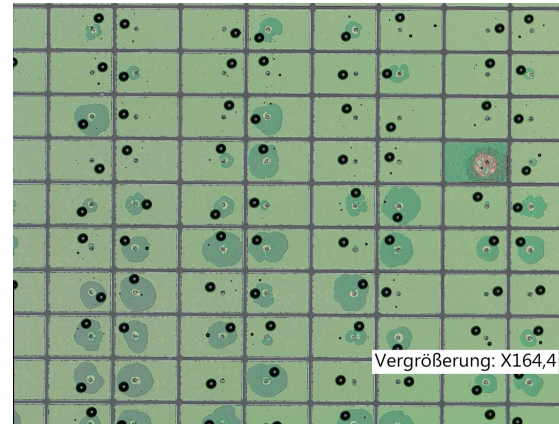
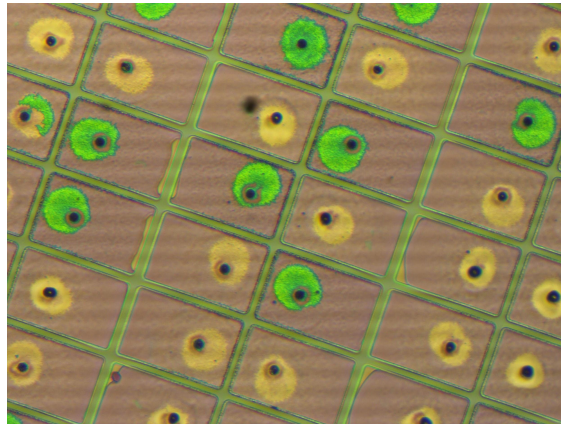
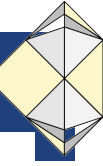


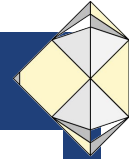
Thank you



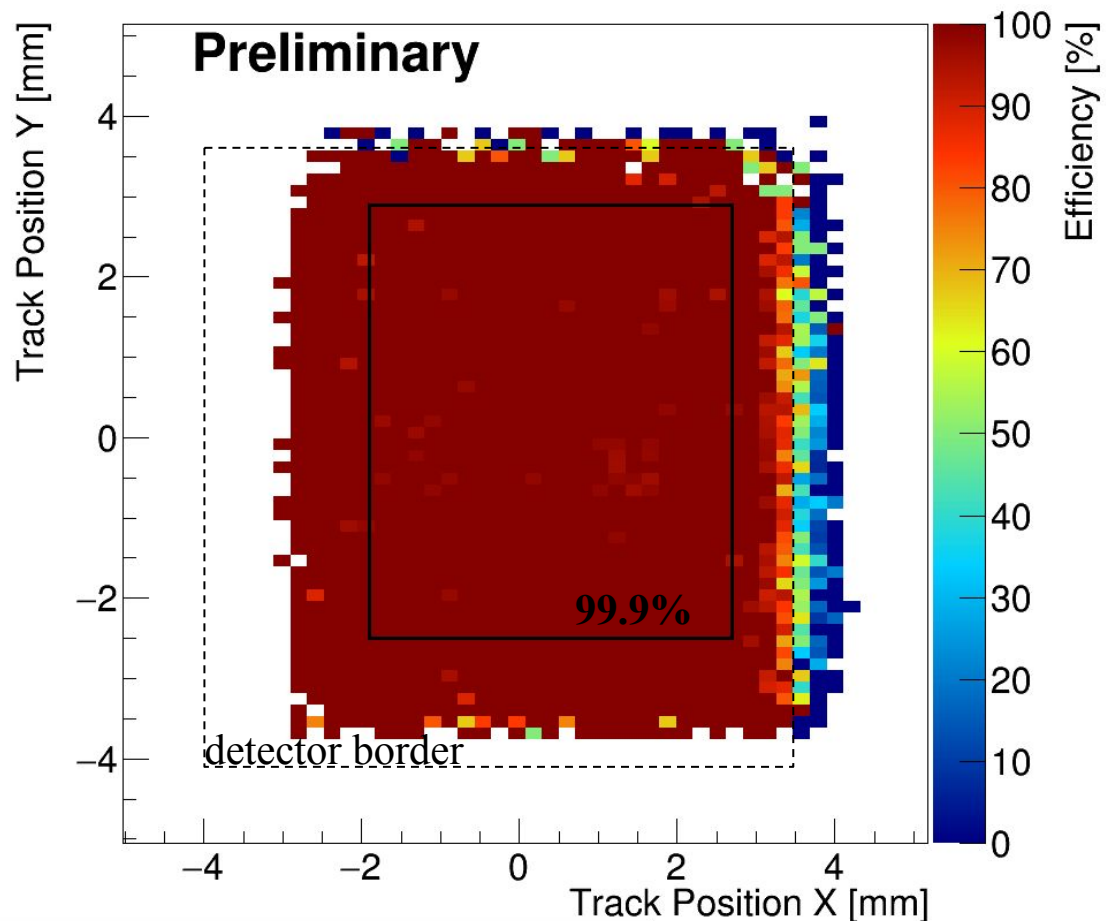
Backup







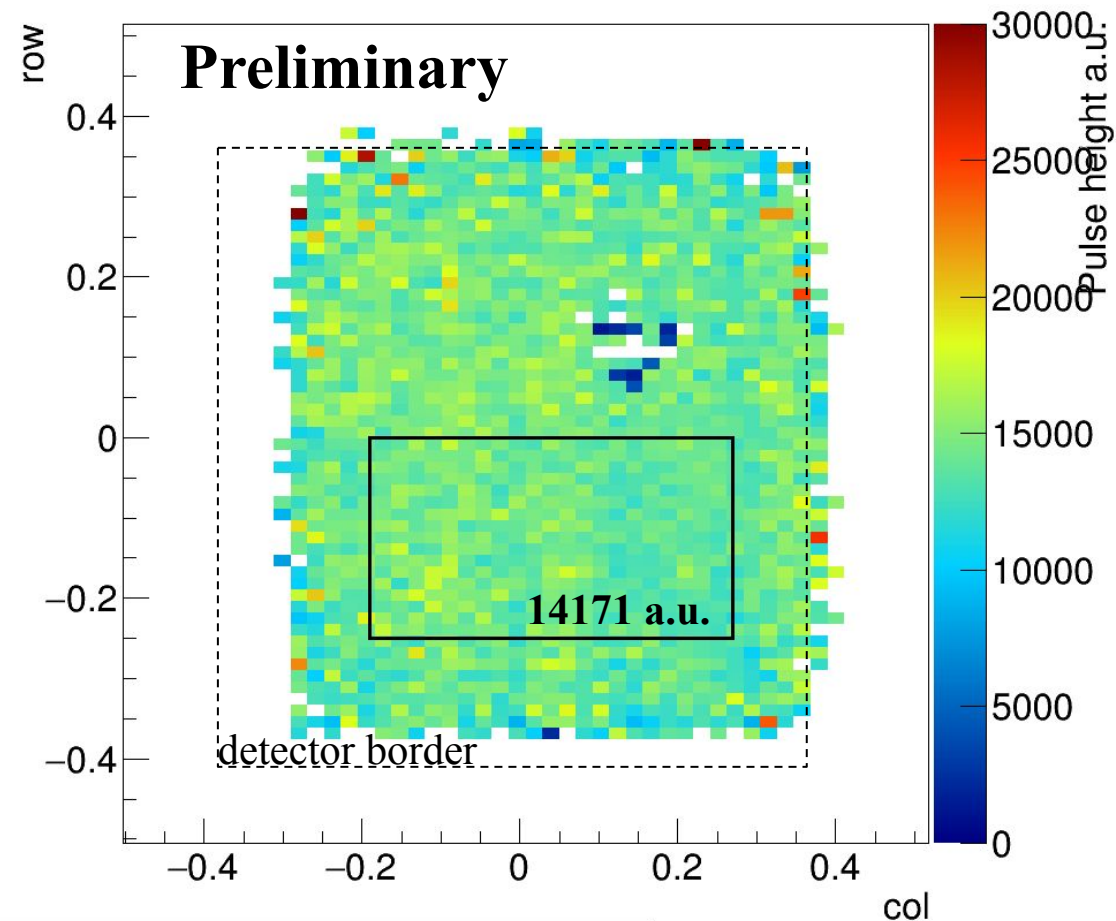
Efficiency Map D2



Run 9: 337 kHz/cm², 18159 days, 1:08:57 (451392 evts)
 Detector: D2 (September 2019)
 Info: -200V, nonirradiated, Att:

git hash: 9737767

Pulse Height Map SiD2



Run 9: 337 kHz/cm², 18159 days, 1:08:57 (451392 evts)
 Detector: D2 (September 2019)
 Info: -200V, nonirradiated, Att:

git hash: 9737767