Integration of Graphene-based nanostructures for novel gaseous detectors

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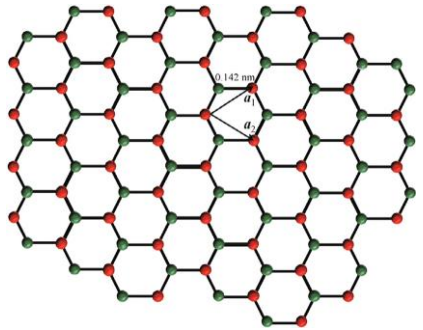
Graphene transfer on top of GEM foils

Properties of 2D materials such as graphene could offer new perspectives for novel gaseous radiation detectors.

Graphene:

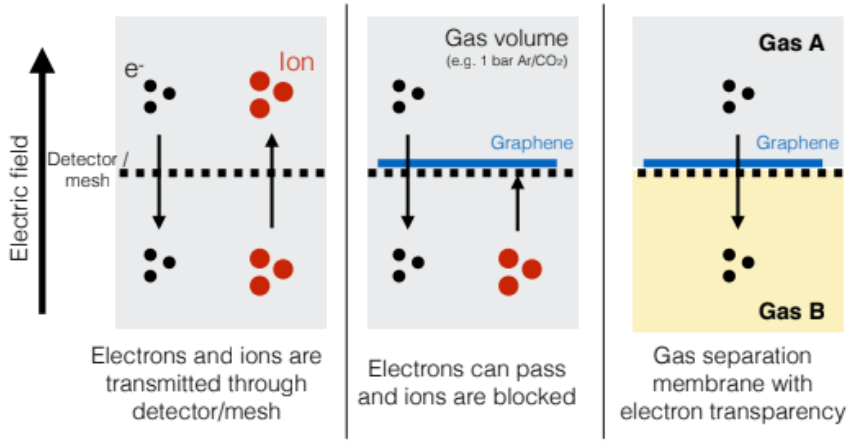
- **Single/few layers** of graphite
- Each layer is 1 atom thick
- **C atoms** arranged in a hexagonal lattice with sp^2 hybridised atoms(planar, 120°)



- Promising material:
- It is easy to handle → without the necessity of clean room facilities

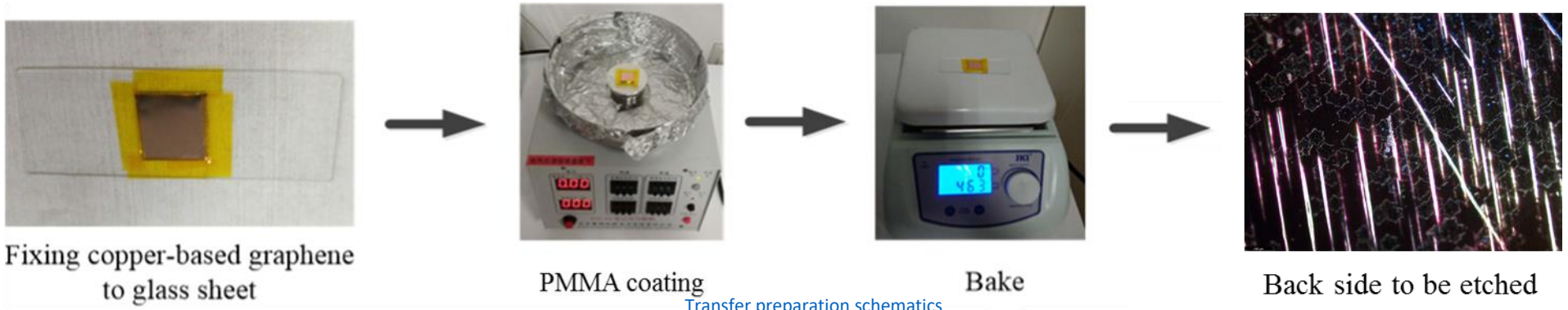
Mono/few layers of Graphene are transferred on top of GEMs foils:

- **Ions back flow** suppression → Graphene should be transparent to the electrons but not to ions.
- **Gas separation** Graphene should physically separate the drift and amplification regions.

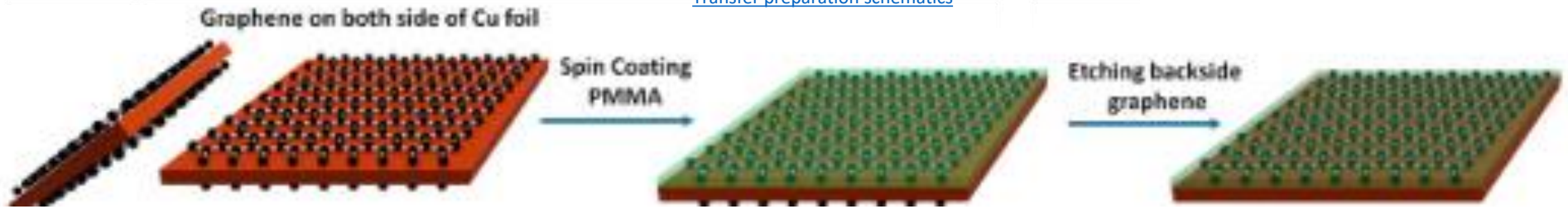


Graphene wet transfer procedure

- **Graphene foil preparation**:
- **PMMA coating**: the PMMA was spin-coated on the CVD graphene on copper foil; Then, baked @90°C for 2 min.
- **Backside Etching**: Because during CVD process grew graphene layers on both sides of the copper foil, graphene on other side (no PMMA) of the foil is etched via an **oxygen plasma** treatment.



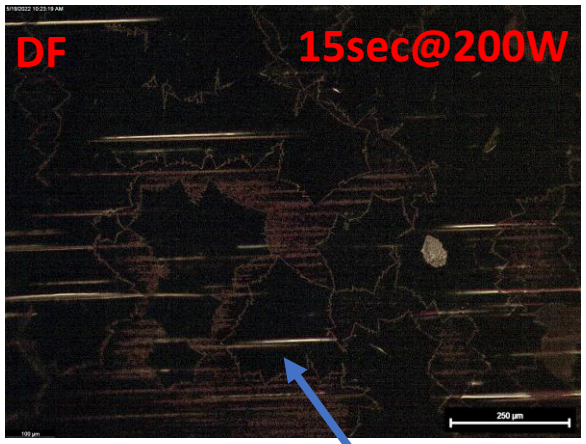
Transfer preparation schematics



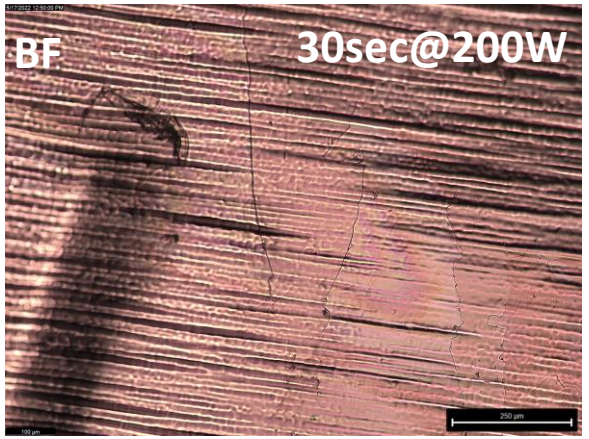
Shirin Afyouni Akbari et al., Scientific Report 10, 6426(2020)

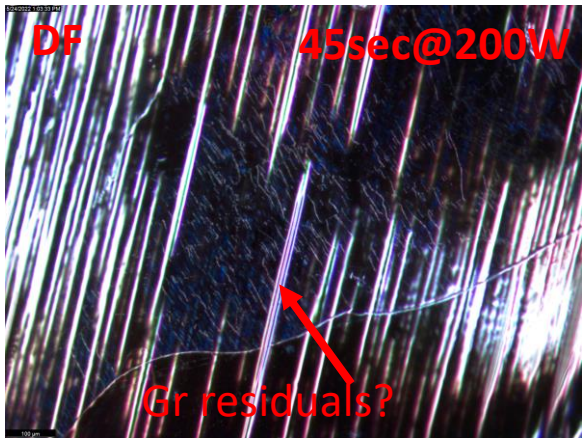
Oxygen plasma etching

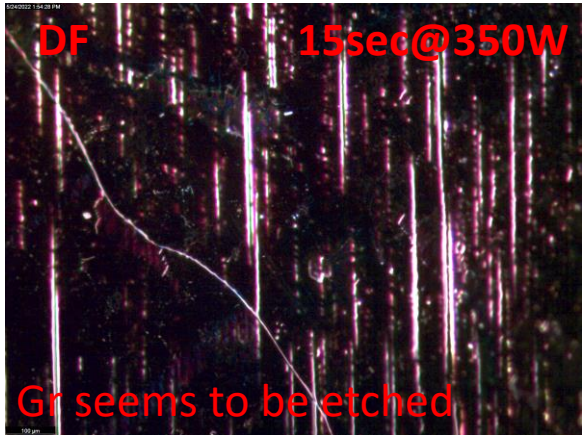
Optical Microscope images showing the back side of the foils after the dry etching treatment

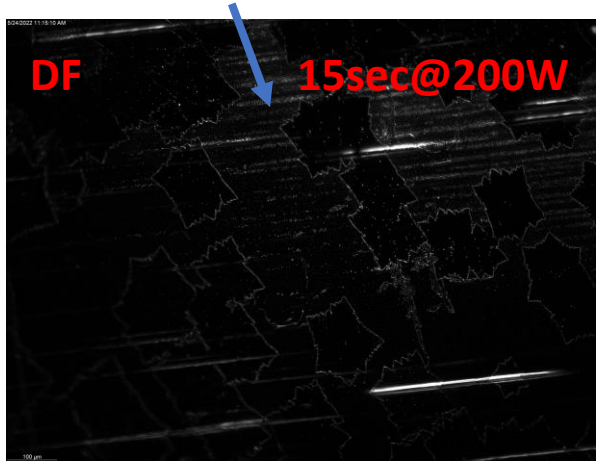


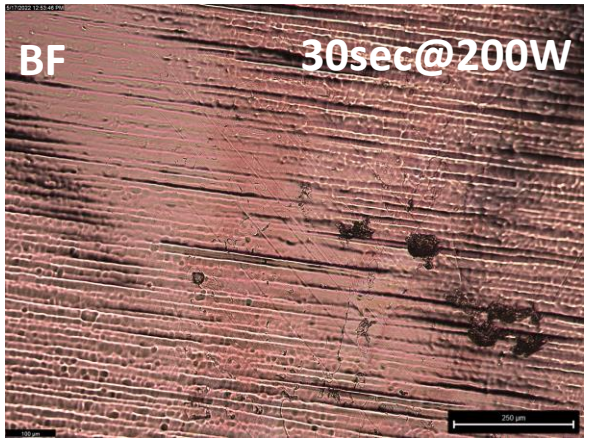
Gr residuals?

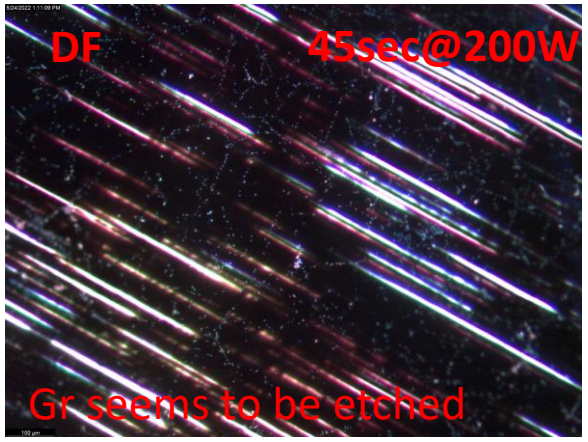








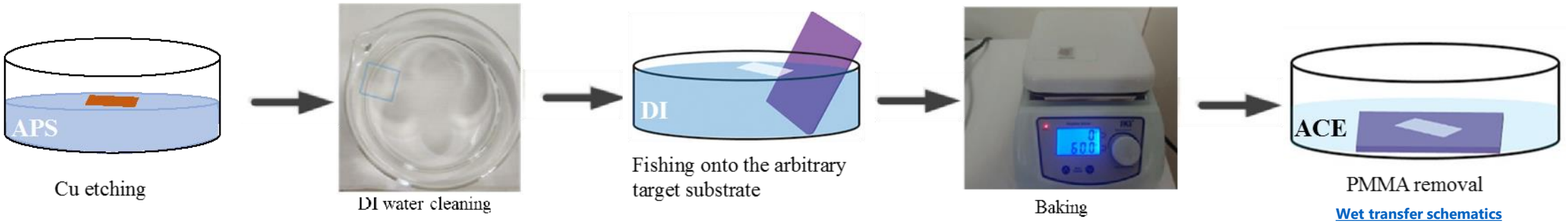




Graphene WET transfer procedure

- **Transfer procedure:**

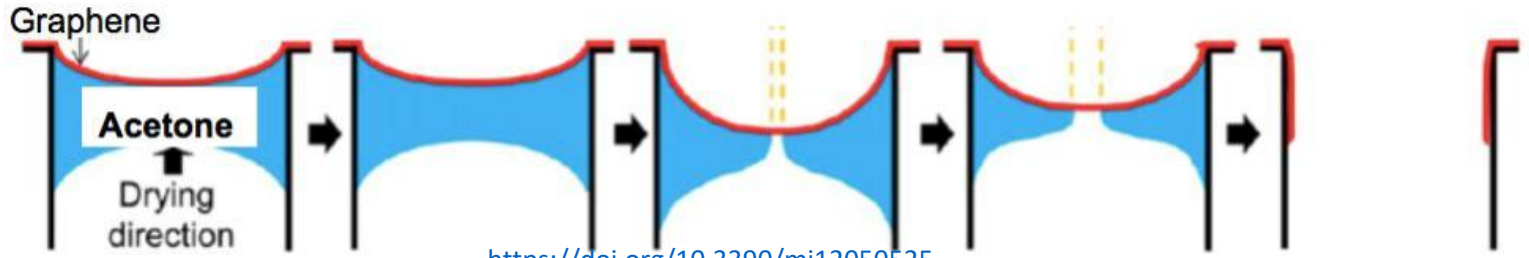
- **Wet etching treatment**: The **copper foil is etched** in an Ammonium persulfate solution (APS) for ˜ 1.5/2 h.
- After copper etching, PMMA/Graphene is **rinsed** with fresh deionized water.
- After the DI water rinsing, the PMMA/graphene stack is **left on the DI water for 12 h** to remove impurities and residue.
- **PMMA/graphene stack is fished** onto the target substrate.
- Substrate is backed @90°C for 20min.
- **PMMA removal**: PMMA is removed soaking the sample in **Acetone**



Wet transfer schematics

Free standing graphene transfer

- Critical steps of transferring a single/few layers of graphene on holes:
  - **PMMA removal and drying procedure:**
- **Adhesion issues of Graphene** during the **PMMA removal:** some areas of the layer detach from the substrate.
  Not optimized substrates:
  - Surface roughness
  - Coating
- **Drying procedure:** If the sample is dipped in ACE, during the drying process solvent surface tension acting on the graphene membrane will completely break the membrane.



https://doi.org/10.3390/mi12050525

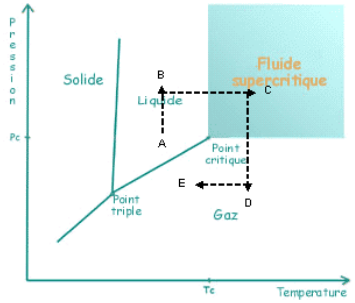
To solve the issues during the drying procedure, two approaches have been explored:

Inverted floating method (IFM)

- **PMMA** is dissolved in **ACE** via an **IFM**

Critical point dryer (CPD)

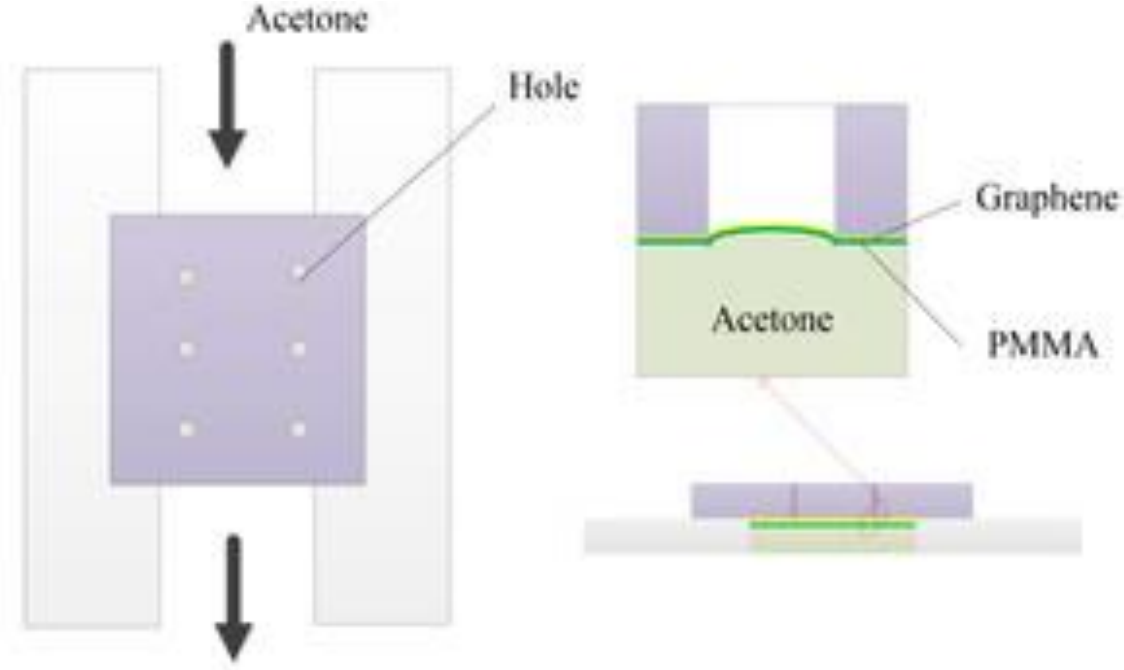
- **PMMA** dissolved by dipping the sample in **ACE**
- **Dried CPD**

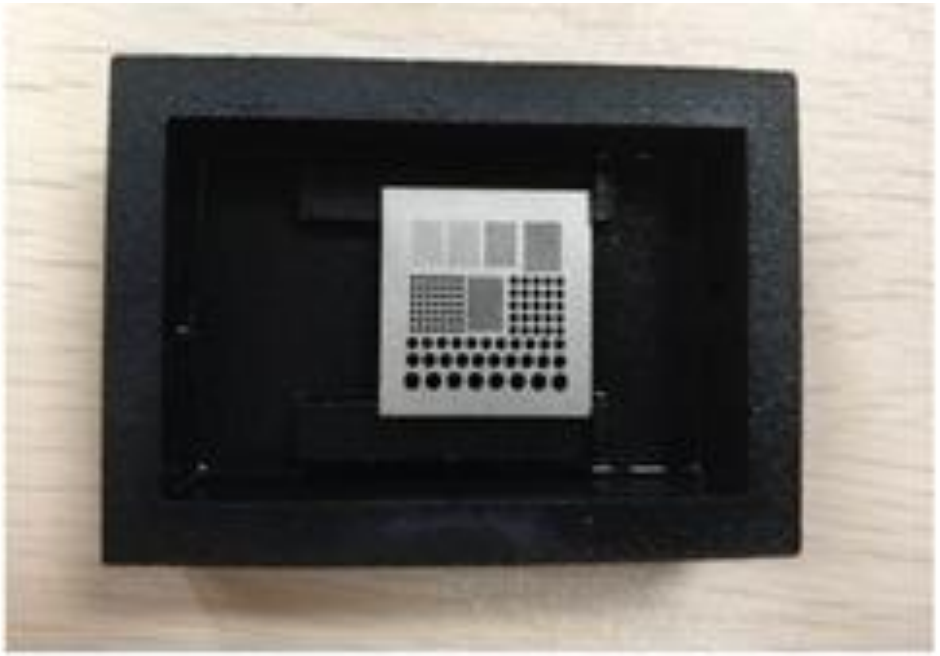


CMi critical point dryer

Inverted floated method

- PMMA removal → The sample is placed upside down in order to keep only the PMMA/Gr side in contact with the solvent.
- Few minutes in contact with the ACE and dried
- To have more gentle drying procedure, ACE could be exchanged with a solvent with a lower surface tension





Shirin Afyouni Akbari et al., Scientific Report 10, 6426(2020); https://doi.org/10.3390/mi12050525

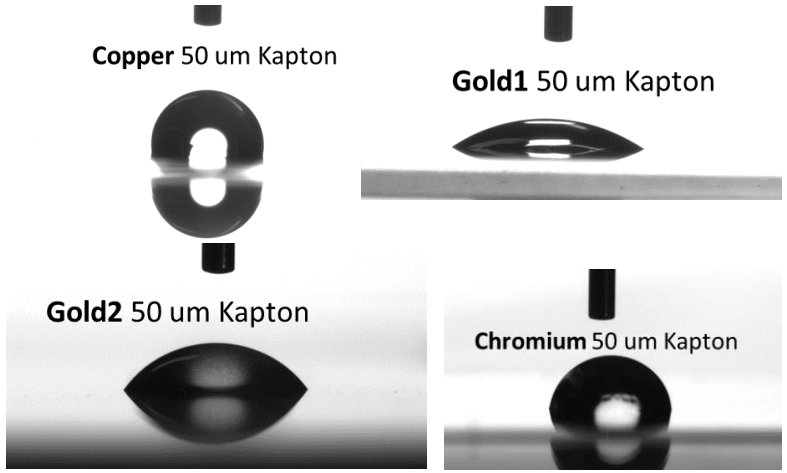
Graphene transfer on foils

- **Wet Graphene transfers** have been explored on foils with different coatings available for GEMs fabrication:

Kapton thickness	Coatings		
50 um	Cu	Au1	Cr
25 um	Cu	\	Cr

- **Contact angle measurements** on different available foils:

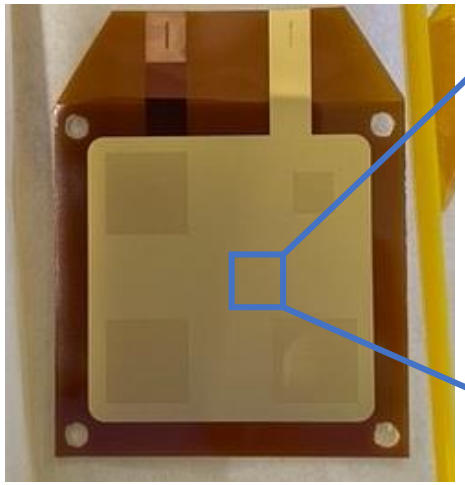
50 um Kapton thickness	
Coating type	Contact angle measurement [deg]
Copper	90.42
Gold1	39.72
Gold2	55.13
Chromium	84.32

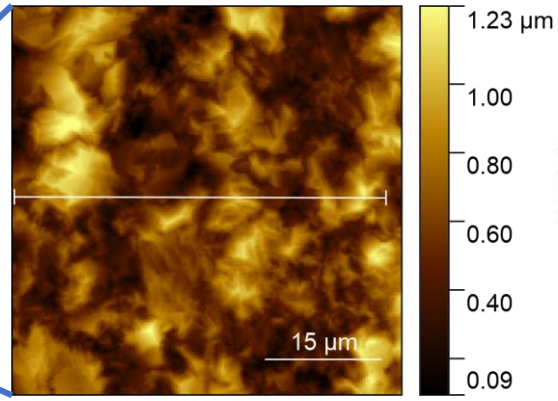


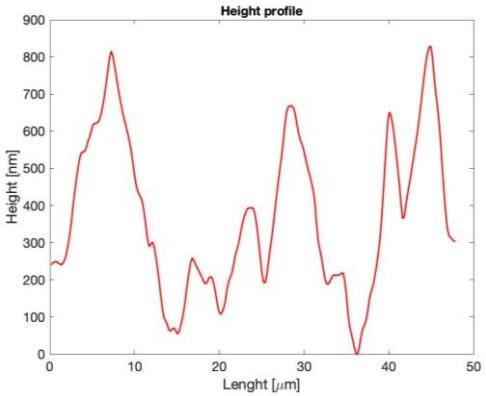
- **Surface roughness** → In the order of few 100 nm for all the foils except for Chromium which is few nm

Graphene transfer on GEMs

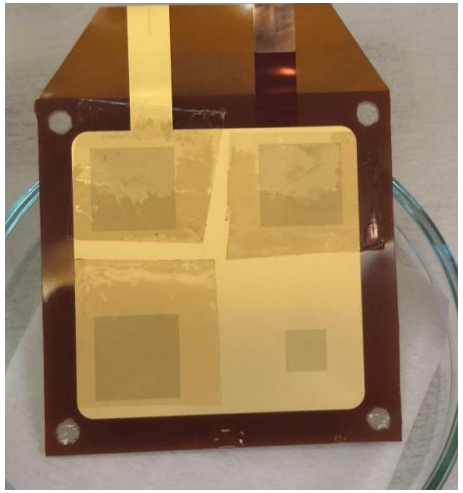
- **Substrate details:**
- Au coated GEM
- Holes diameter: 50 µm
- Kapton Thickness: 50 µm





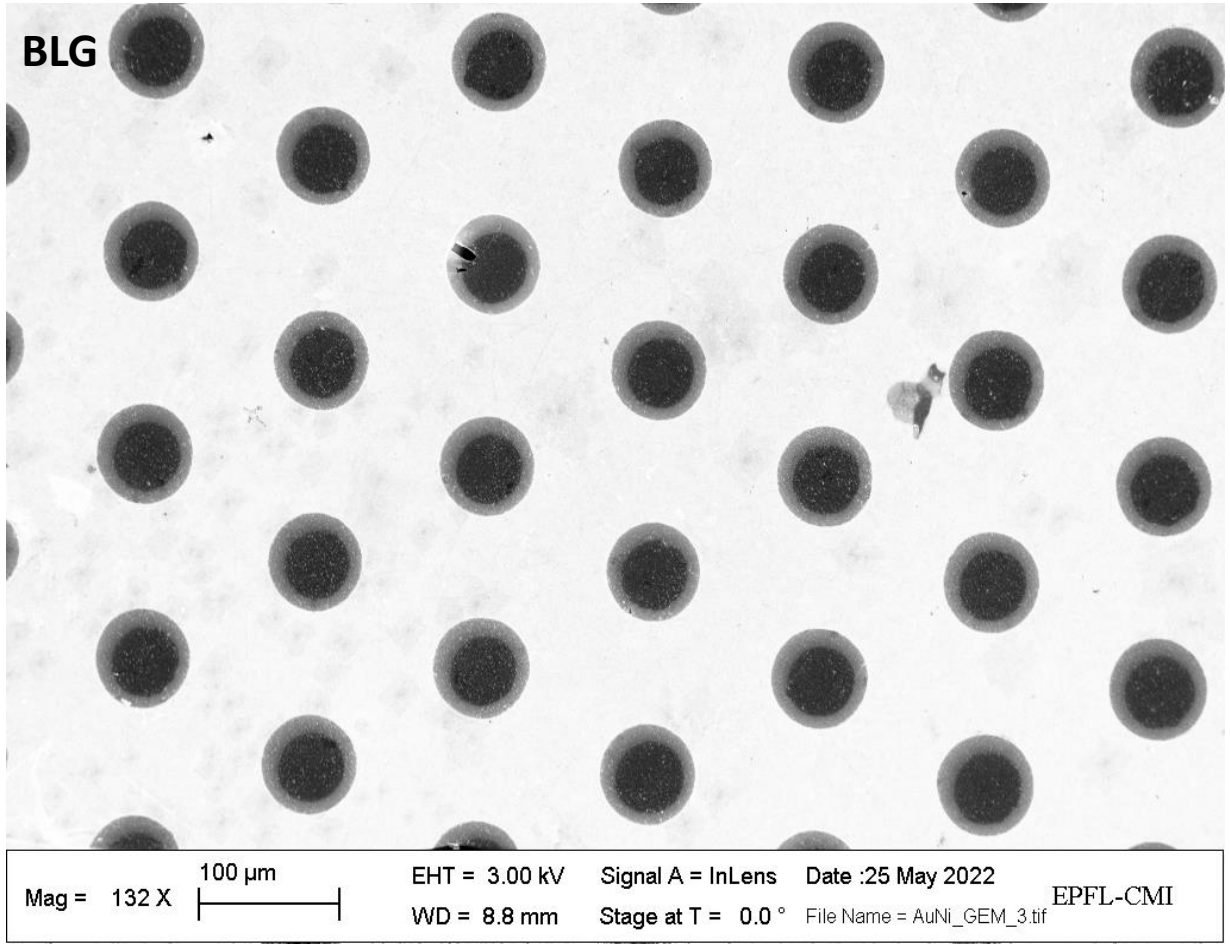


- **Transfer details:**
- Graphene is protected w/ PMMA+PPC coating
- Cu etching in APS (3mg in 100 ml DI)
- DI rinses x3
- Transfer on the target substrate → The sample is kept vertically, until water is dried from the holes
- Hot plate @90°C ~30 min
- PMMA removal
  - IFM
  - Sample is soaked in ACE, Then dried with CPD
- Samples are characterized with SEM

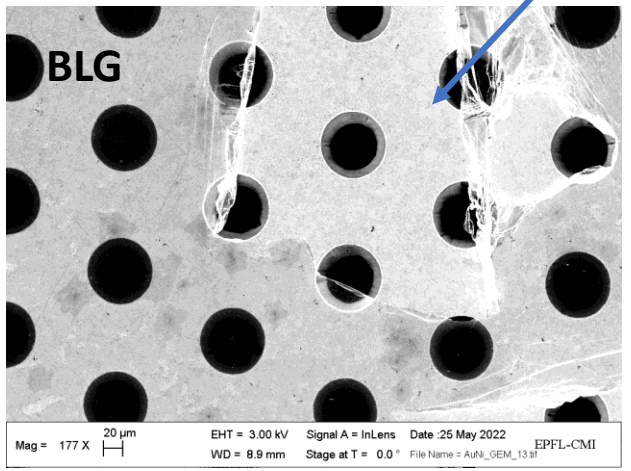


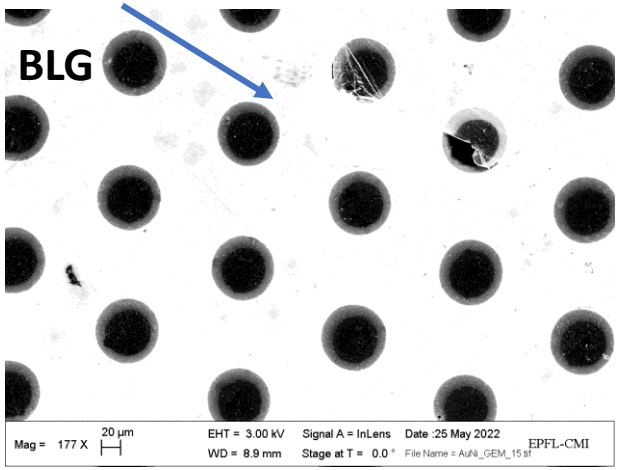
CPD Transfer: **BLG** on Au GEM 0.5x0.5 cm^2

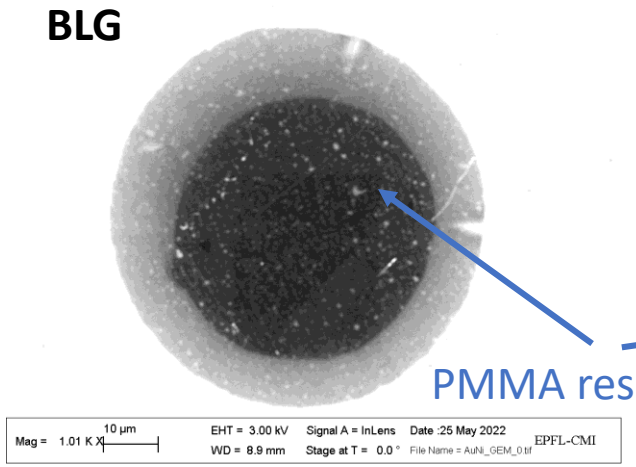
- **BLG** partially detached and rolled up
- **Good coverage**; most of the holes are covered
- Some of them are completely or partially broken

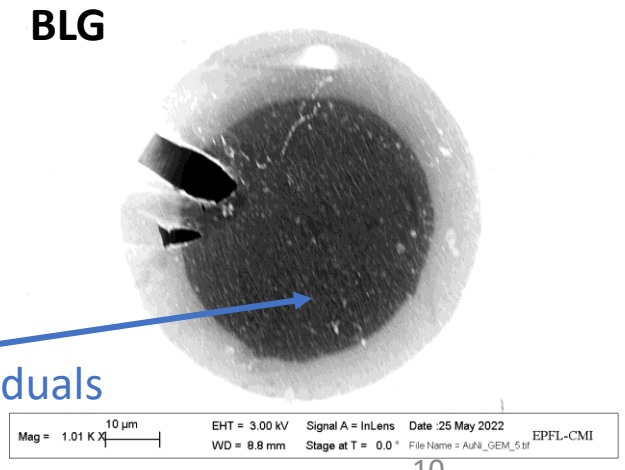


Membrane detachment during the PMMA removal





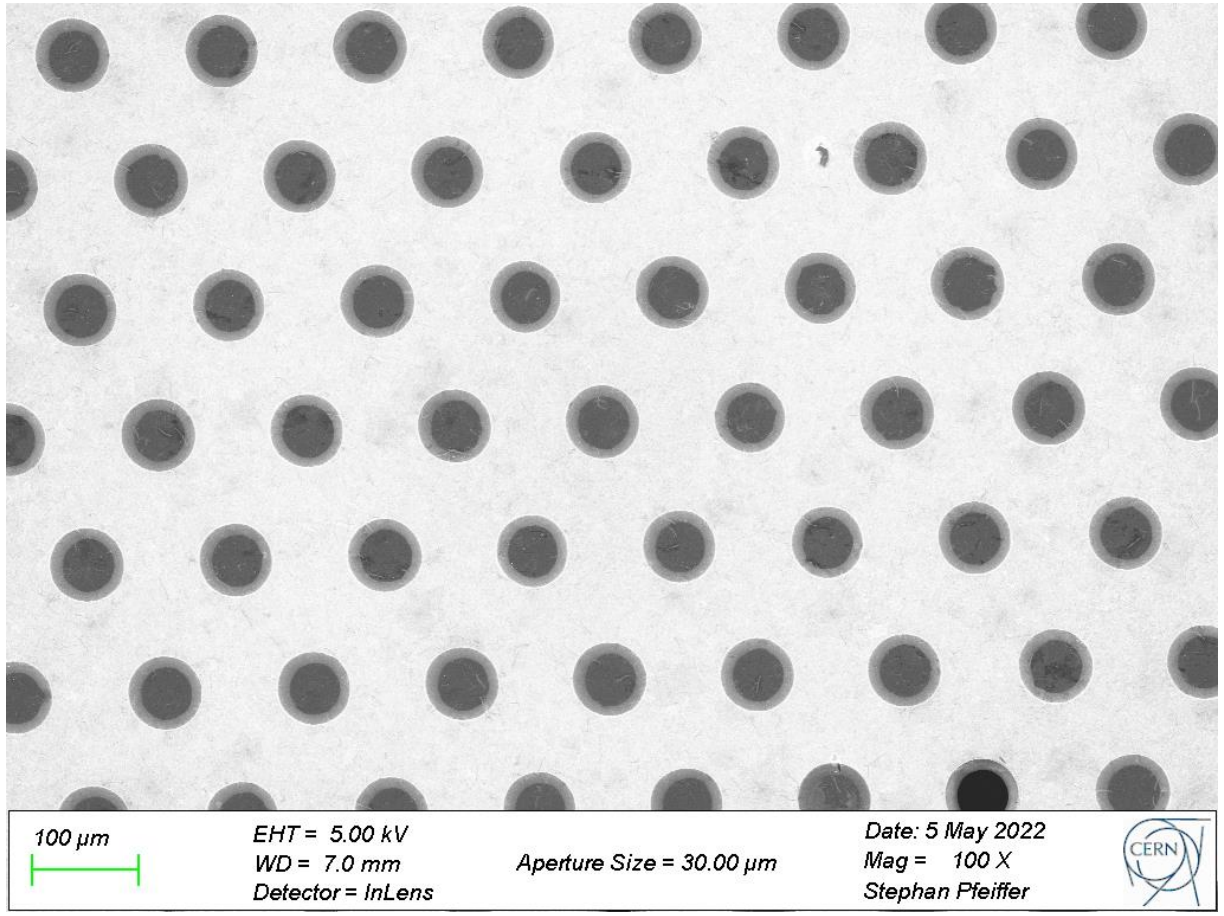


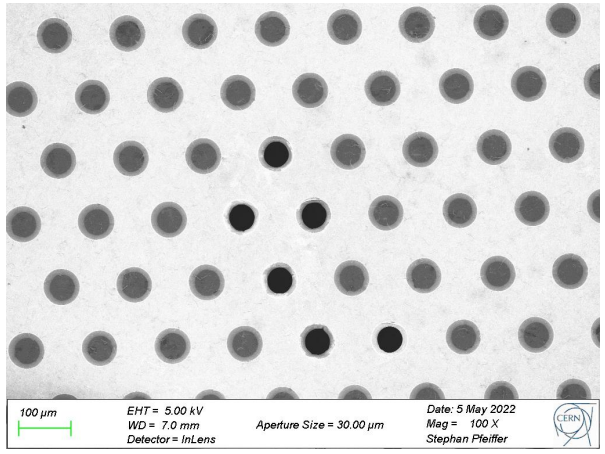


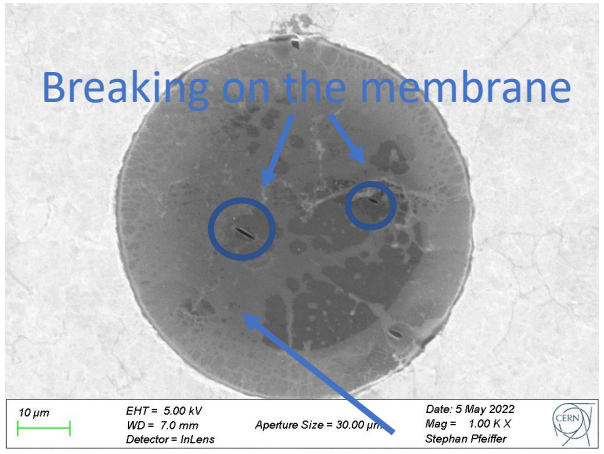
PMMA residuals

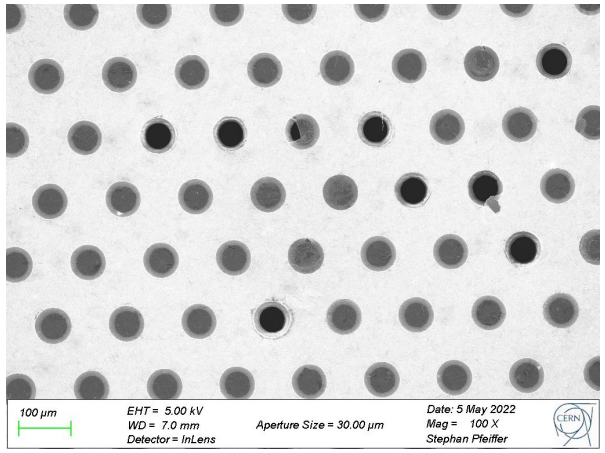
IFM Transfer: **BLG** on Au GEM 0.5x0.5 cm^2

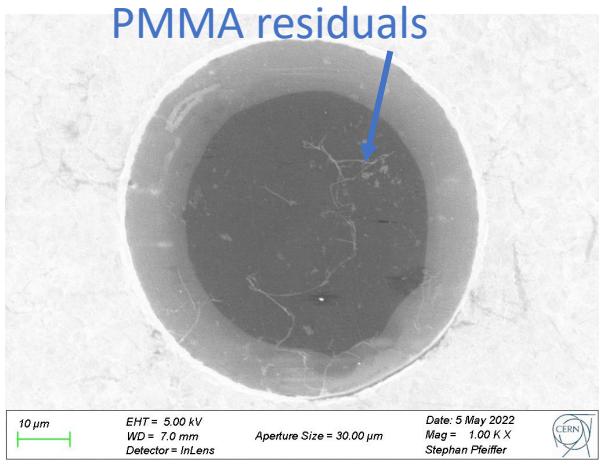
- **BLG** adhesion is okay
- **Good coverage**; most of the holes are covered
- Some of them are completely or partially broken





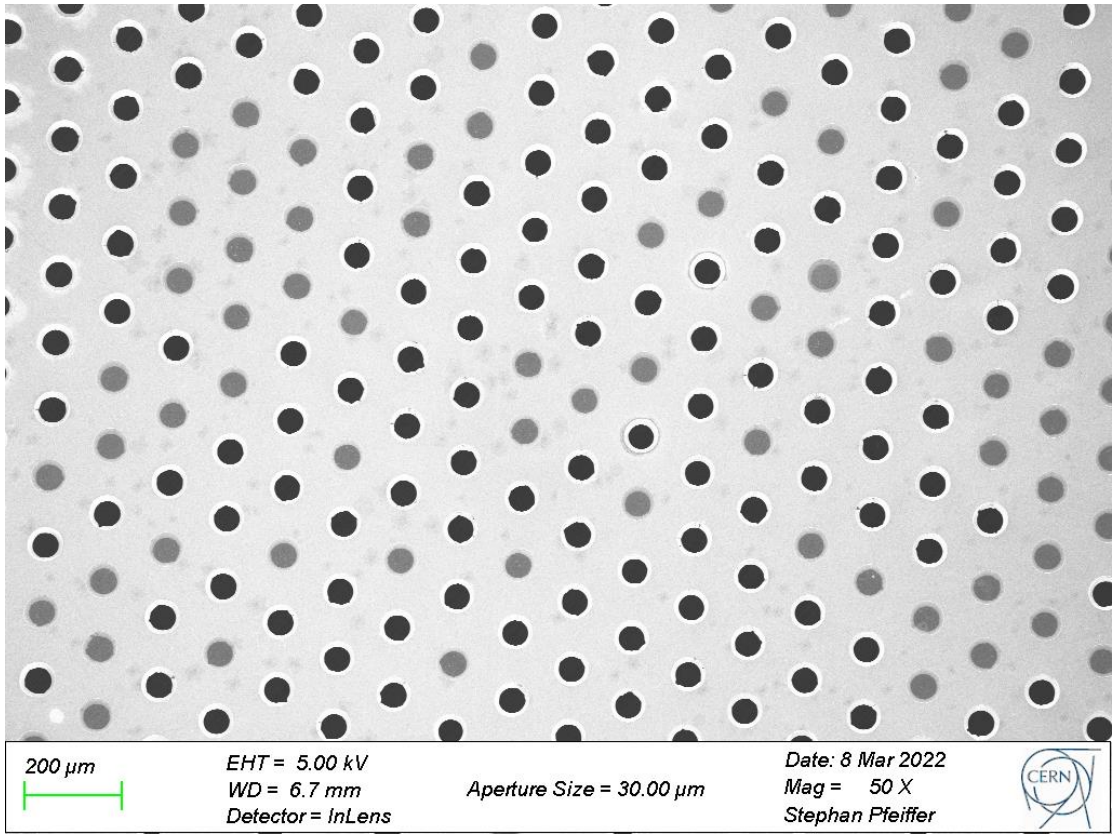


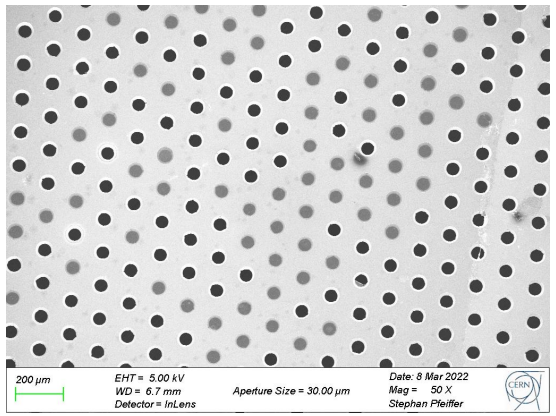


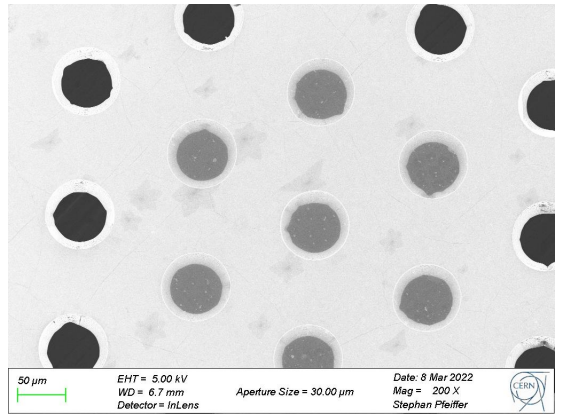


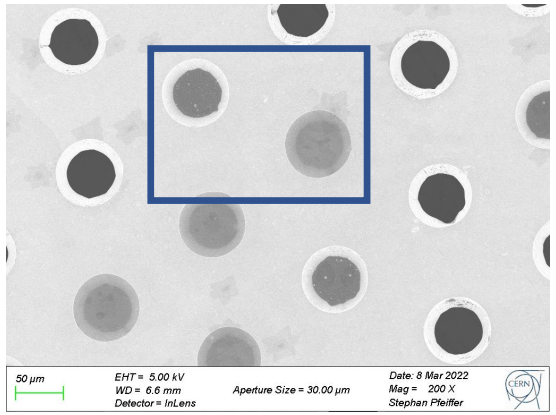
IFM Transfer: **MLG** on a Cu GEM piece

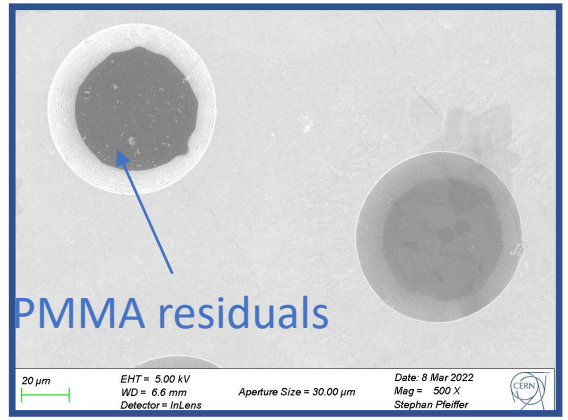
- **MLG** adhesion is okay
- **Poor coverage**; most of the holes are uncovered or partially covered
- Some of them are covered











Future Perspective

- The transfer procedure have to be optimized in order to:
- Reach a **full coverage**
- Membrane with **no breakings inside the holes**

- **Lowering PMMA residuals** as much as possible → **PMMA removal** via ACE is not enough:
  - Annealing step would be needed

- Electron transparency measurements in the range of 5-20 eV.
- Gas separation test.

Thank you for your attention!