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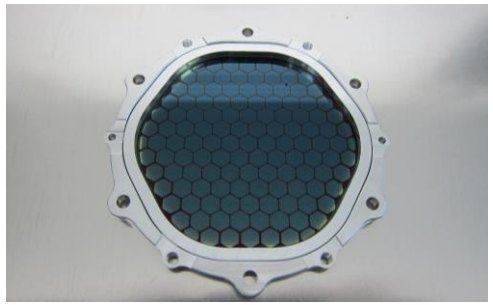
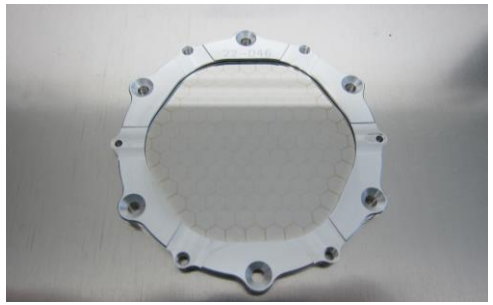
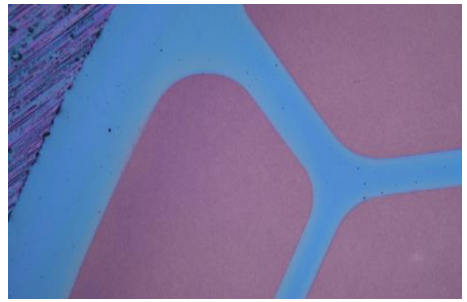
# Final meeting presentation

Fully carbon-based filters for X-ray astronomy
ESA Contract No. 4000139215/22/NL/GLC/rk







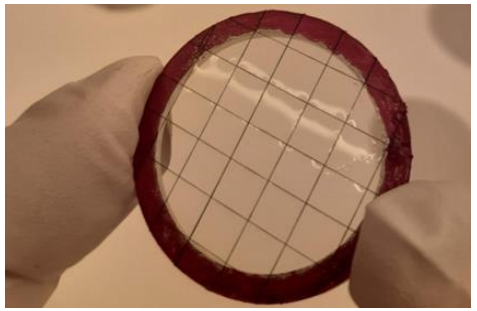
# Starting situation of the activity

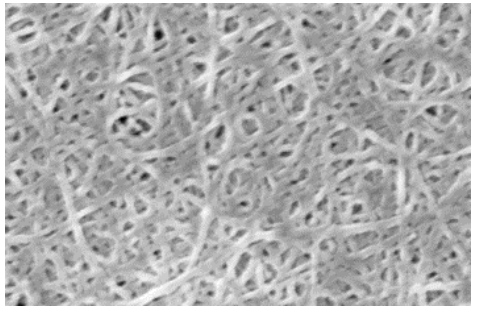
Very promising results demonstrated in the previous LAOF CCN activity

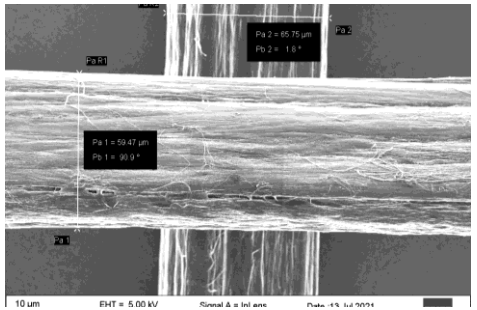
- Impressive scalability and manufacturing robustness of the structures also with large areas
- Compatibility of the CNT membranes with metal meshes studied and demonstrated in the previous activity
- The first feasibility test with carbon yarn meshes
- Development of the CNT membrane control
  - Orientation of the mesh, Density of the mesh, Pyrolytic filling of the membrane

Drawbacks

- CNT membrane adhesion on the metal mesh or carbon yarn
- Fragile membranes on the carbon yarn
- Wrinkles on the Al coated membranes
- Non-uniformity of the UV/VIS/IR attenuation







# Key things so far from this activity

**Key Improvements**

Elegant carbon nanotube (CNT) mesh invented

- Mesh and membrane densified together leading to excellent adhesion
- Oriented CNT mesh

Wrinkles on the Al coated membranes basically eliminated

Dual layer SWCNT membrane to eliminate pin holes and improve CNT uniformity

Developed understanding of the local variations of the Al coating

**Key observations**

Filter structures very robust and reliable to manufacture

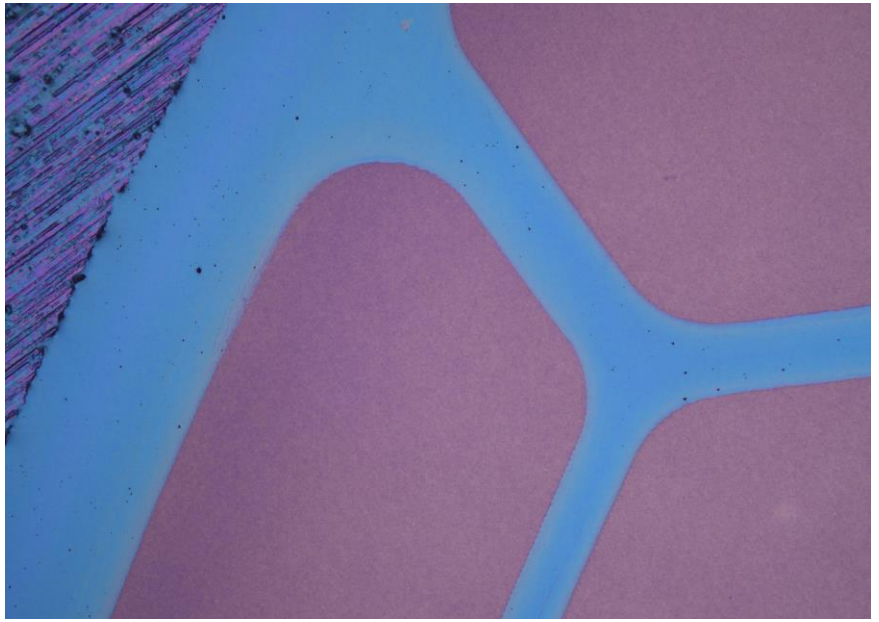
Mess patterning creates flakes CNT flakes

- Total elimination challenging so far
- Not necessarily crucial (bind on the membrane, extremely light)

Patterning process

- Width control of the mesh bars
- Linewidth capabilities of the process

Al coating depended on the background material and density of the mesh

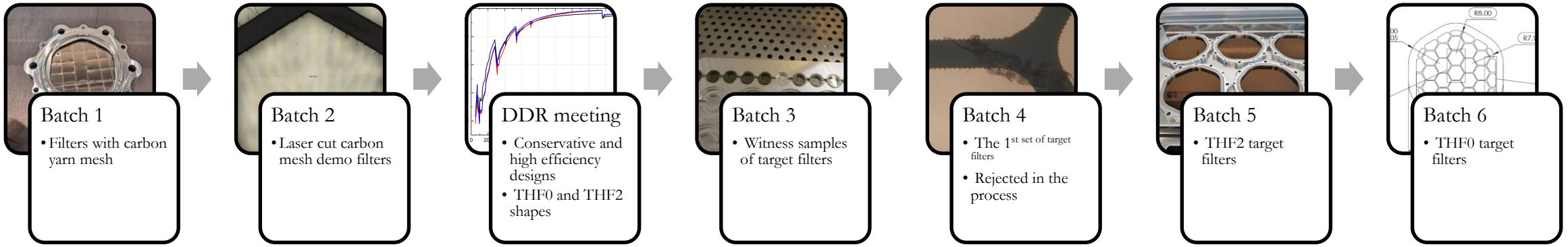


**Key question**

How strong is the CNT mesh and the whole structure in reality?

What is the quality of the Al coating?

# Fabricated batches in the activity



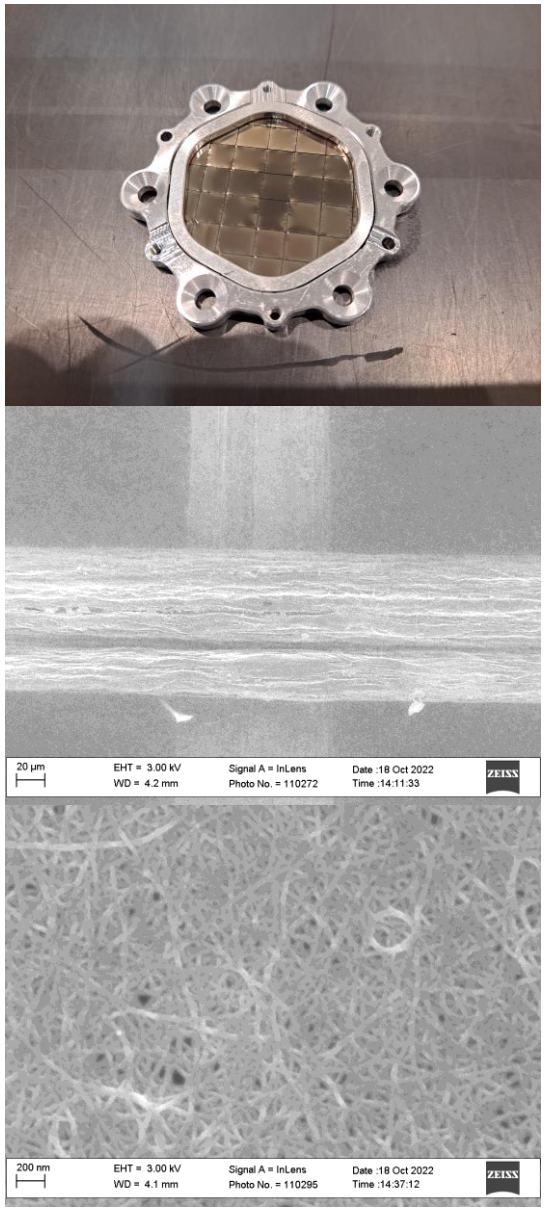
# Batch 1 carbon yarn meshes

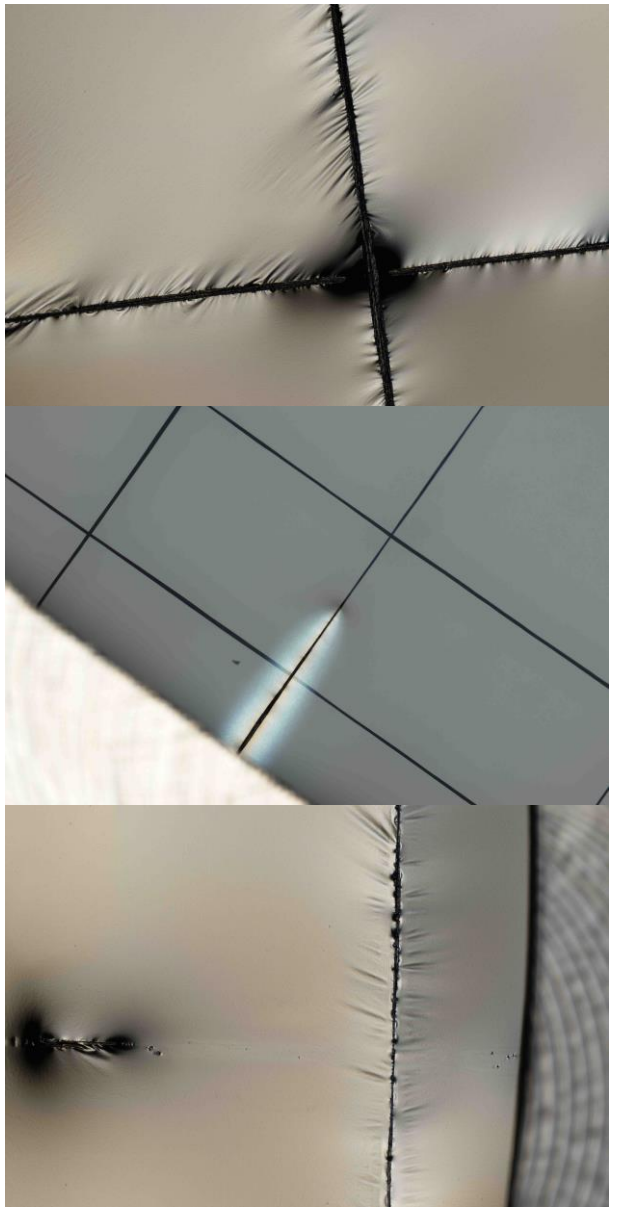
**Samples:**

- Meshes based on carbon yarns cross weaved over each others
- Totally 14 samples
- Two different yarn diameters of 21 µm and 50 µm
- Membrane: CNT T 85 % @550 nm, Al 30 nm
- Mesh pitch 2.8 mm and 5 mm
- Frames THF0 and THF2

**Observations:**

- Strong and robust mesh material
- Quality of the mesh
  - Yarns not fully smooth
  - Crossing yarns can move against each others
  - Pitch width variation
- Quality of the membrane
  - Membrane not fully attached on the yarn
  - Yarn crossings especially challenging
  - Al causes a lot of wrinkles
- Robustness questionable



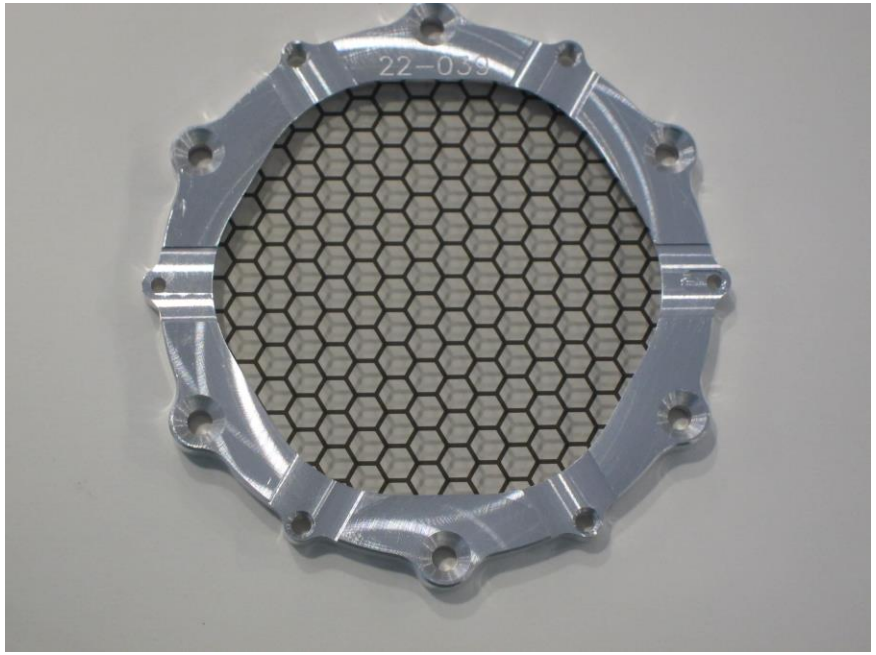
# Batch 2 – Patterned CNT meshes

**Background:**

- Mesh is same but just thicker material than the CNT membrane.
- Patterned hexagonal mesh
- CNT Membrane and mesh bonds together.

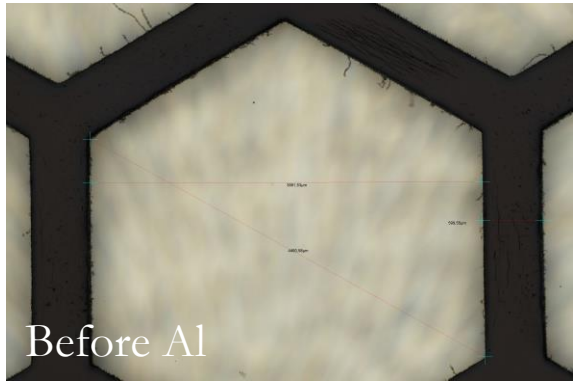
**Samples:**

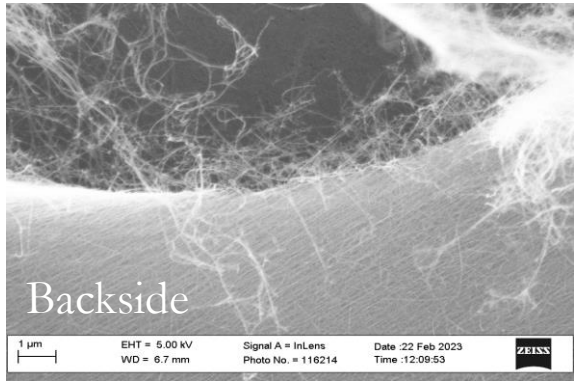
- Meshes based on thick CNT membranes
- Totally 20 samples
- Three different mesh thicknesses of 0.2 %, 15 % and 40 % @550nm, that roughly refer to graphite thicknesses of 1.67 µm, 0.51 µm and 0.25 µm, respectively.
- Mesh open area appr. 80 %.
- Membrane: CNT T 85 % @550 nm, Al 30 nm
- Mesh pitch 3.9/4.5 mm (hexagon), mesh bar width 0.6 mm
- Frames THF0 and THF2



# Batch 2 - mesh quality

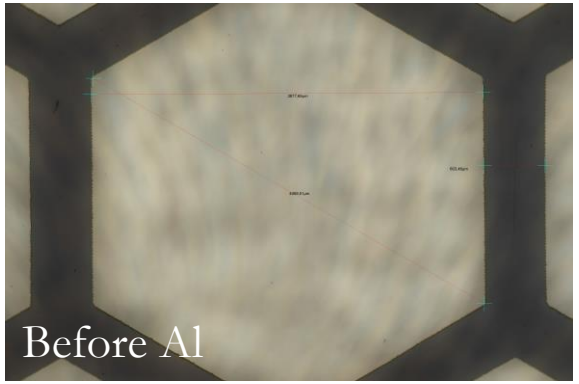
**0.2 % @550 nm**

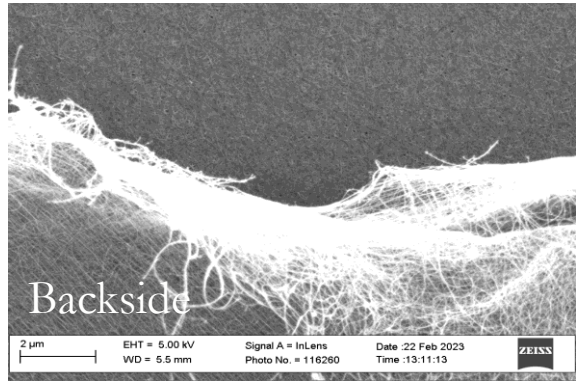


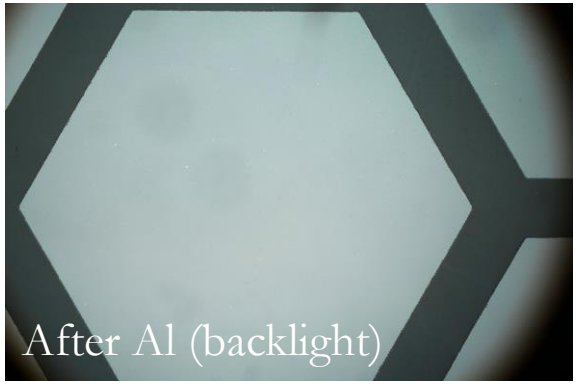





**40 % @550 nm**

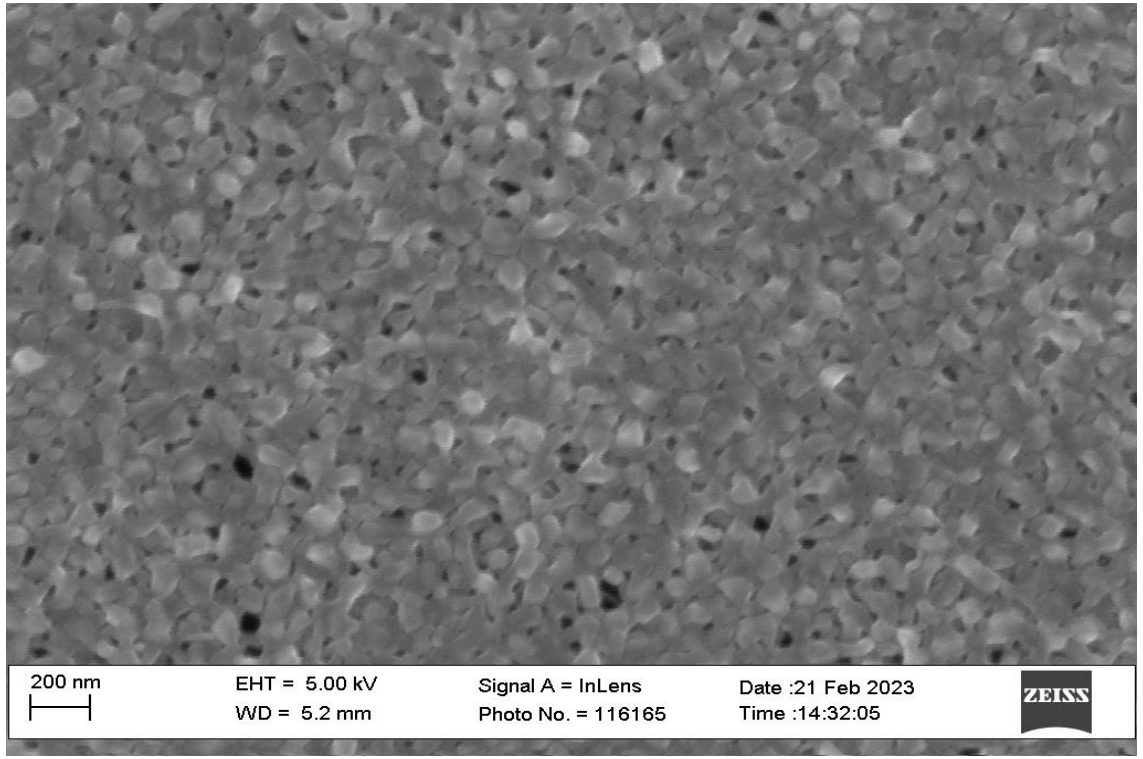


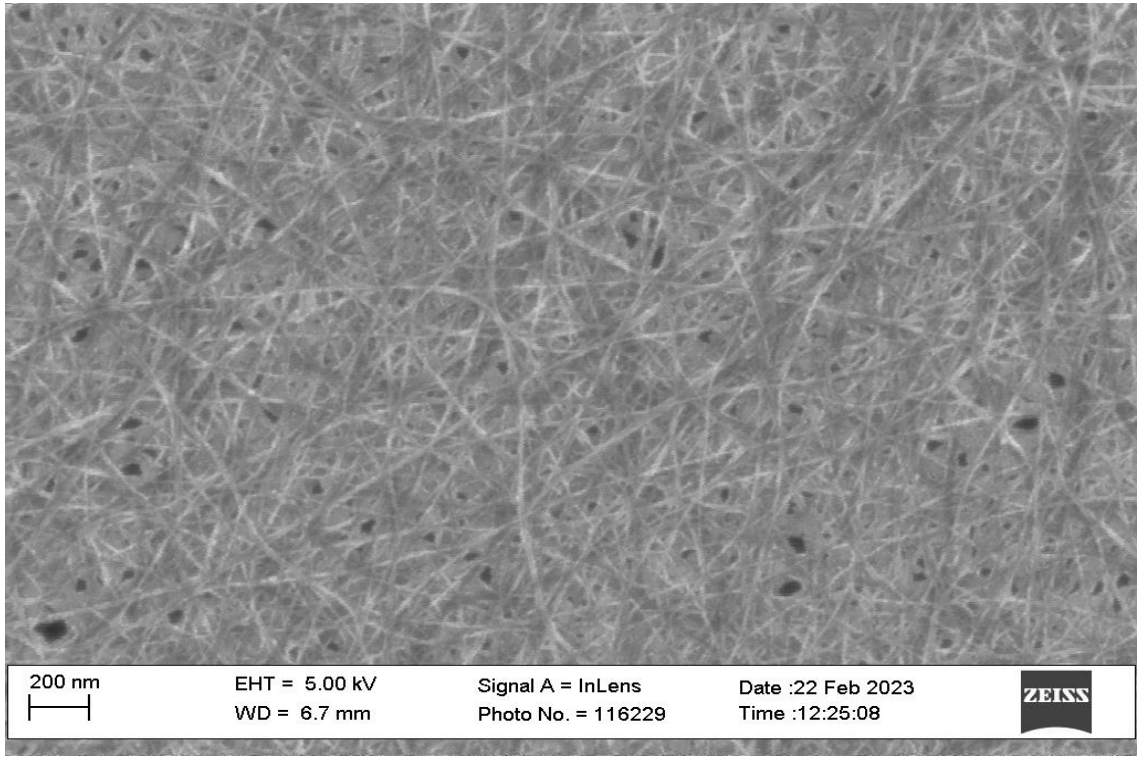



# Batch 2 - membrane after 30 nm Al coating

## Deposition side




## Back side



# Target filters selected for the fabrication

| Filter type | | Volume | Mesh | | | | Membrane | | | |
|---|---|---|---|---|---|---|---|---|---|---|
| **Frame** | **Model** | **pcs** | **Type** | **Pitch** | **Bar width** | **T[1]** | **CNT** | **CNT T** | **Al** | **AlN** |
| THF2 | conservative | 5 | LC[2] oriented CNT | 5mm | 250µm | 15% | SWCNT[3] dual layer[4] | 44% | 30nm single side | 2-3nm |
| THF2 | high efficiency | 5 | LC oriented CNT | 5mm | 250µm | 44% | SWCNT dual layer | 55% | 30nm single side | 2-3nm |
| THF0 | conservative | 5 | LC oriented CNT | 5mm | 250µm | 15% | SWCNT dual layer | 44% | 30nm single side | 2-3nm |
| THF0 | high efficiency | 5 | LC oriented CNT | 5mm | 250µm | 44% | SWCNT dual layer | 55% | 30nm single side | 2-3nm |
| TF110 | conservative mesh material | 5 | oriented CNT[5] | - | - | 15% | - | - | - | - |
| TF110 | high efficiency mesh material | 5 | oriented CNT | - | - | 44% | - | - | - | - |
| TF110 | conservative pellicle | 5 | - | - | - | - | SWCNT dual layer | 44% | - | - |
| TF110 | high efficiency pellicle | 5 | - | - | - | - | SWCNT dual layer | 55% | - | - |
| TF110 | conservative pellicle & Al | 5 | - | - | - | - | SWCNT dual layer | 44% | 30nm single side | 2-3nm |
| TF110 | high efficiency pellicle & Al | 5 | - | - | - | - | SWCNT dual layer | 55% | 30nm single side | 2-3nm |

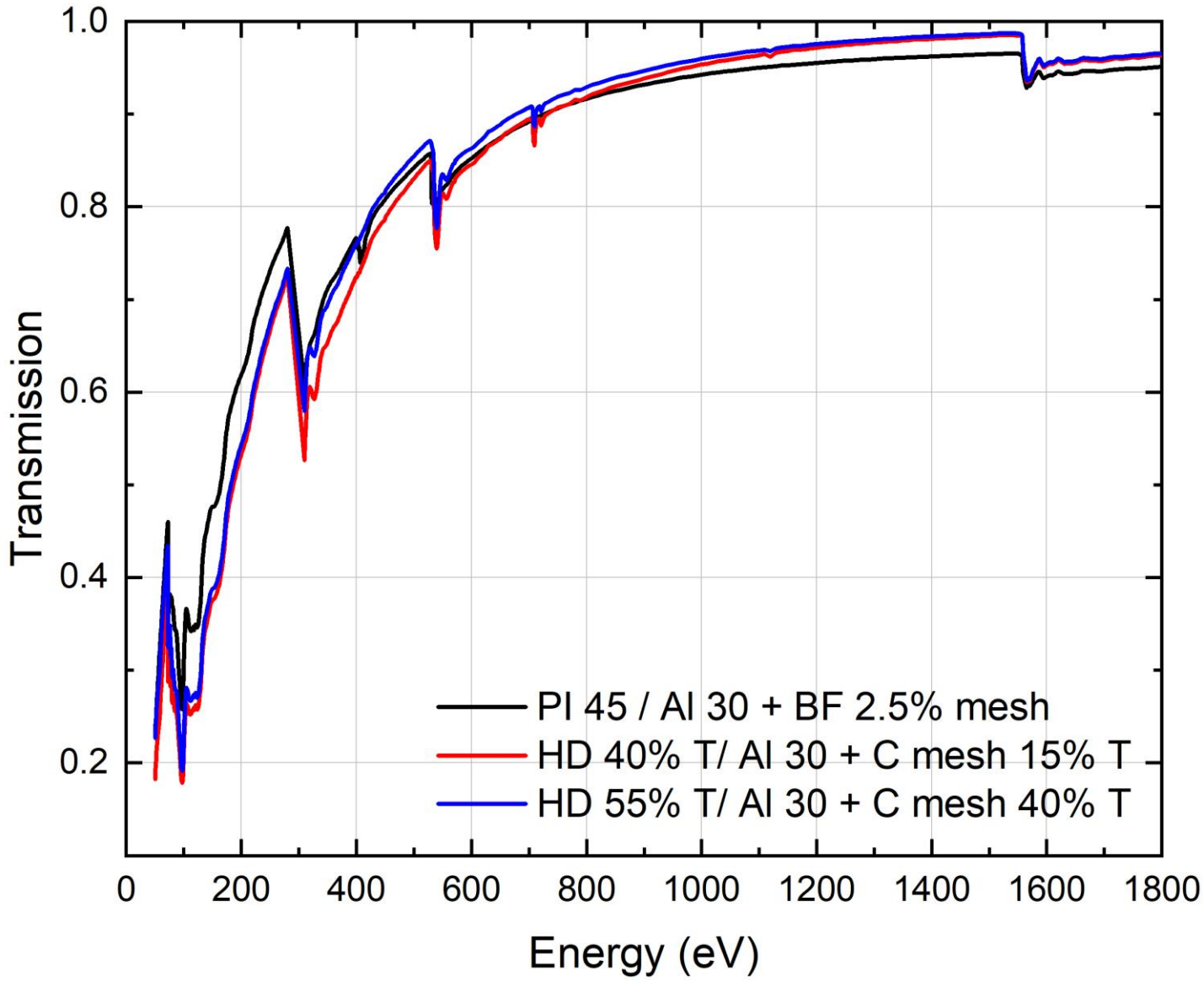
## Simulated target filter transmission

The figure shows the performance comparison of these two filters with respect to a PI45nm/Al30 + metal mesh filter.

Only at very low energies CNT is less competitive

- a non negligible reduction and in a scientifically significant region

The baseline PI 45nm/Al 30nm is also probably too thin especially for large filters.



# Batch 3 – small witness samples

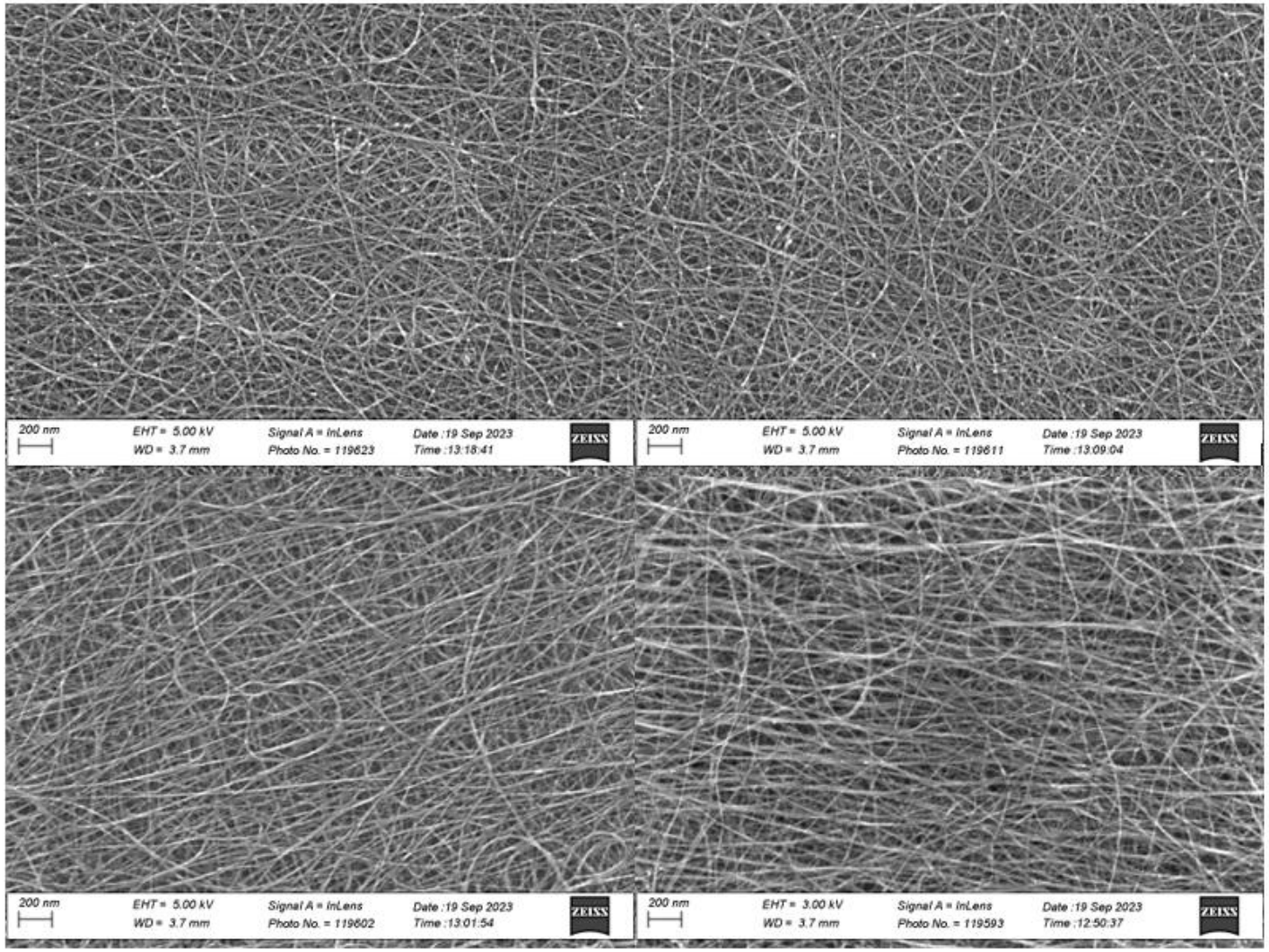
**(top left)** FC-B3-F01 represents high efficiency membrane material

**(top right)** FC-B3-F06 represents conservative membrane material

**(bottom left)** FC-B3-F11 represents high efficiency mesh material.

**(bottom right)** FC-B3-F16 represents conservative mesh material.

The main difference between the images are that MWCNTs **(bottom images)** used in the mesh are thicker and more oriented compared to SWCNTs **(top images)**.

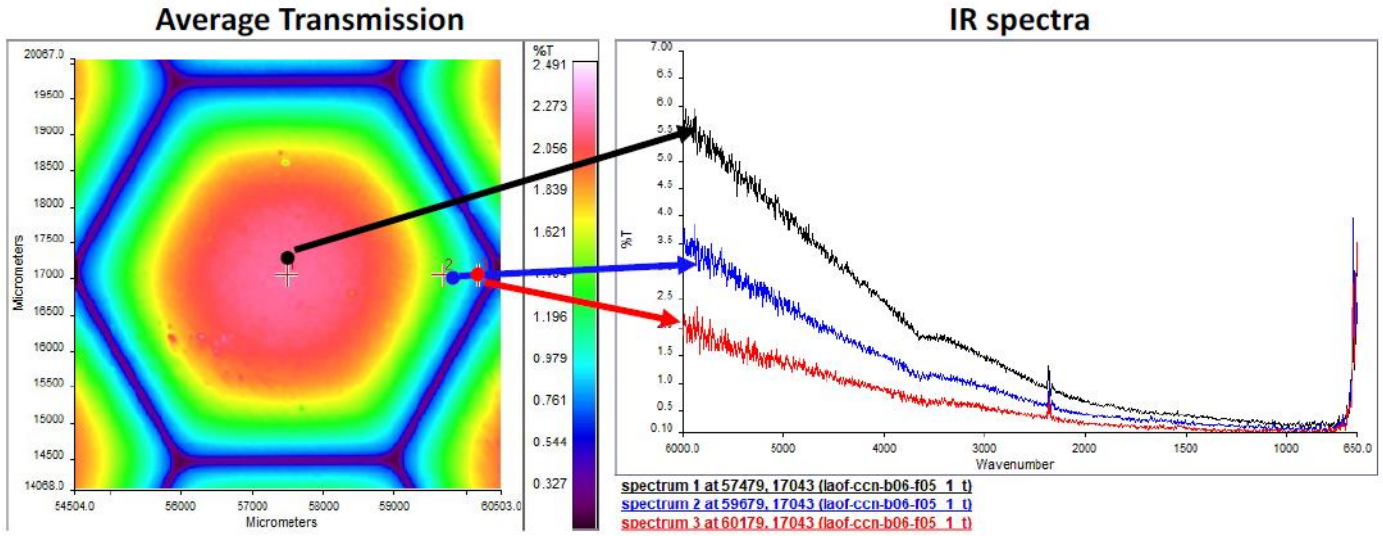


# IR attenuation on the metal mesh - reminder

In the LAOF CCN activity we observed that IR attenuation of the Al coated CNT membrane variates locally.

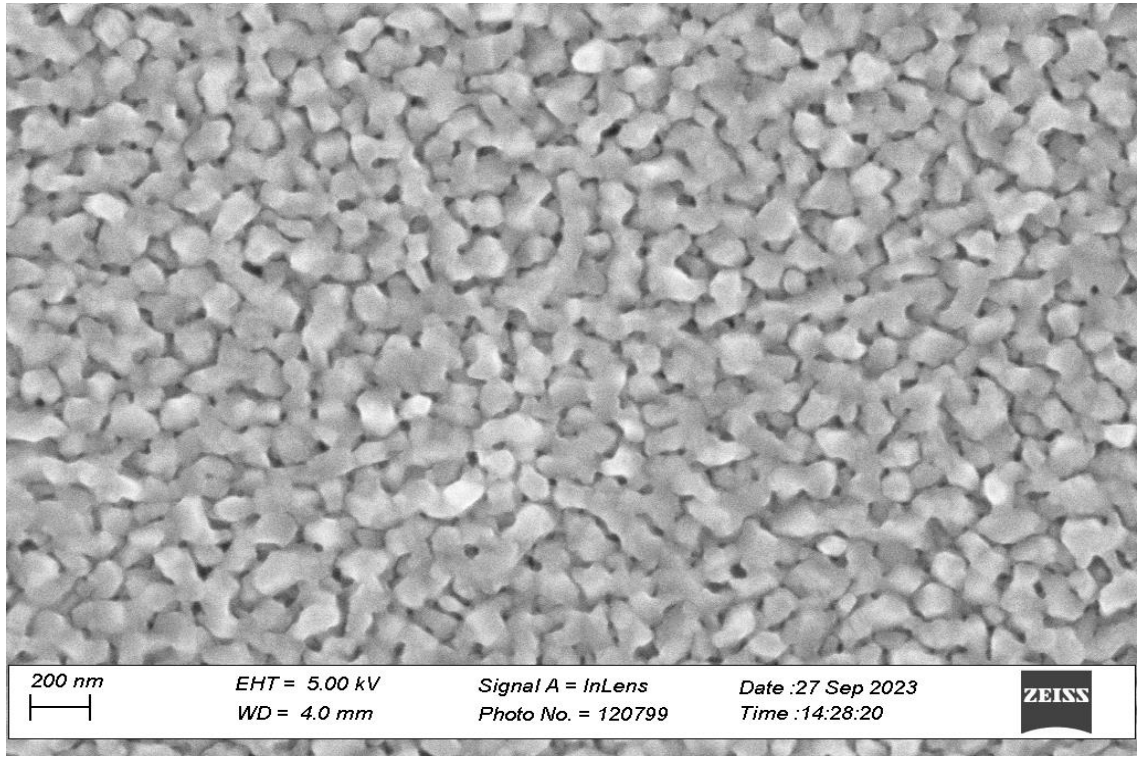
Variation has symmetry related to the metal mesh.

**LAOF-CCN-B06-F05:** THF2 CNT HD30 with BeCu mesh plated with Au by LUXEL

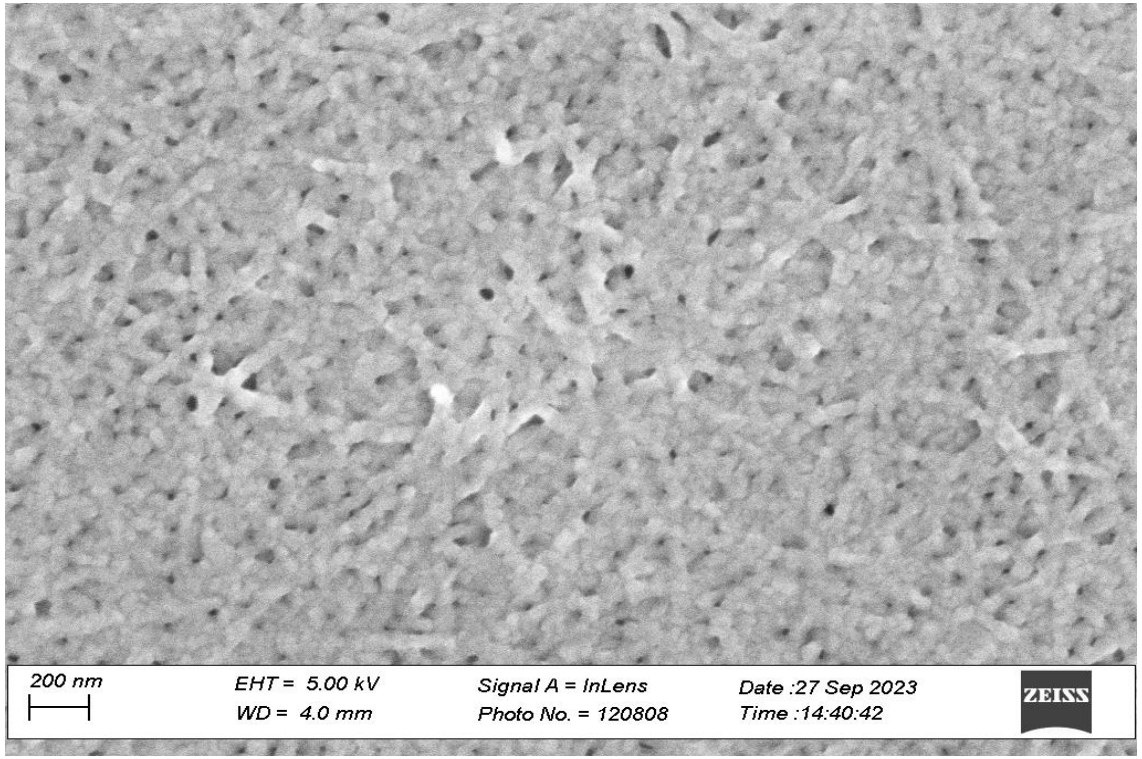


# Batch 3 - membrane after 30 nm Al coating

## Coated membrane – few mm far from frame




## Coated membrane – near the metal frame



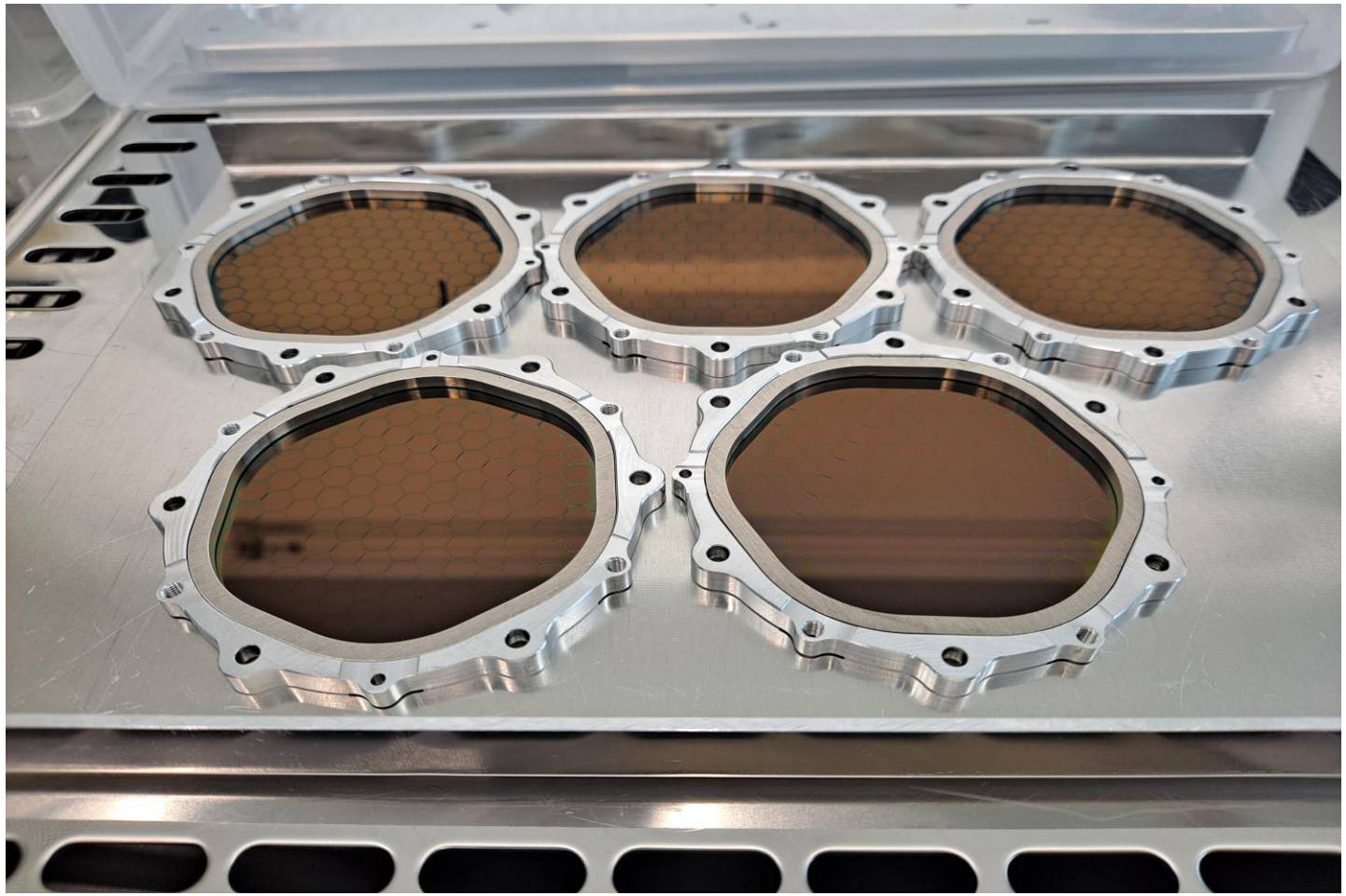
# Batch 5 – THF2 target filters

Generally, the quality of the Batch 5 containing new set of target THF2 samples was good, and the filters are on different level compared to the starting level and compared to the level of the Batch 2

These filters were robust to fabricate with high yield

The visual outlook is good although some non-uniformities can be visually observed.

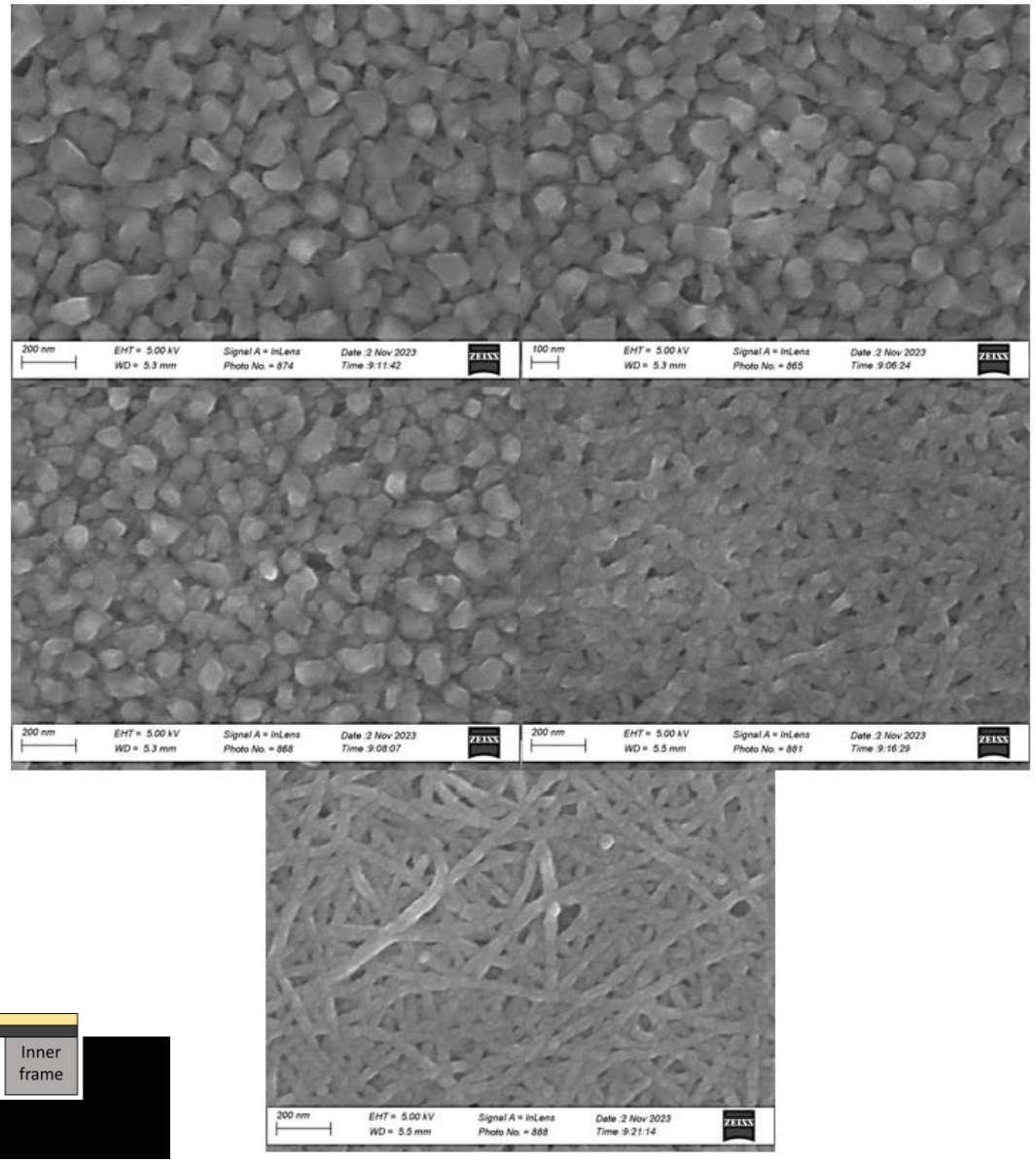
Even the quality has increased we were able to spot few defect types during the inline inspection before shipping the samples to extensive characterization to UniPa.



# Batch 5 - Al uniformity

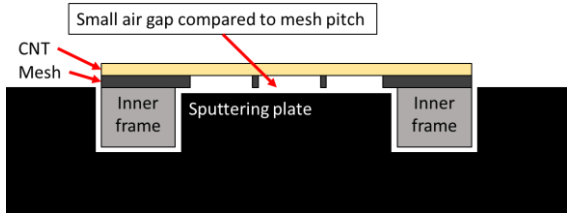
SEM images from sample FC-B3-F2 from different locations including:

- **(top left)** middle of the hexagonal cell in the middle of the filter,
- **(top right)** near the mesh in the middle of the filter,
- **(middle left)** on top of the mesh in the middle of the filter,
- **(middle right)** near the outer metal frame,
- **(bottom)** on top of the outer metal frame.



We tested a new sputtering plate decreasing the gap between the carrier plate and membrane to 0,5 mm but it did not have clear influence.

We tested also carrier plate in contact with the CNT structure, but it did not have clear influence.



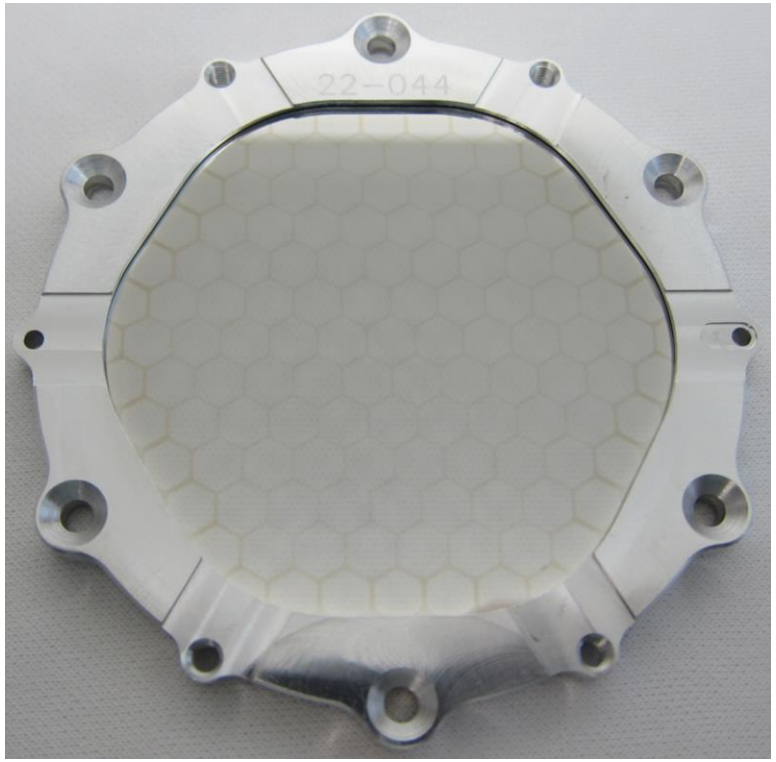

Al coatings with other methods pending

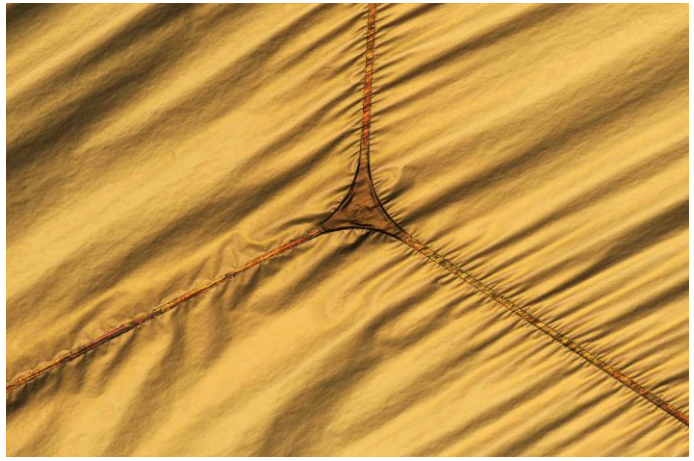
## Batch 5 - Al uniformity

Uniformity of the Al coating is the best so far even we do not know that is it out of the best quality.

Al coating has less difference between the area on the top of the mesh and the area outside the CNT mesh than with the filters using metal frames.

Wrinkles observed with the metal meshes are basically eliminated.

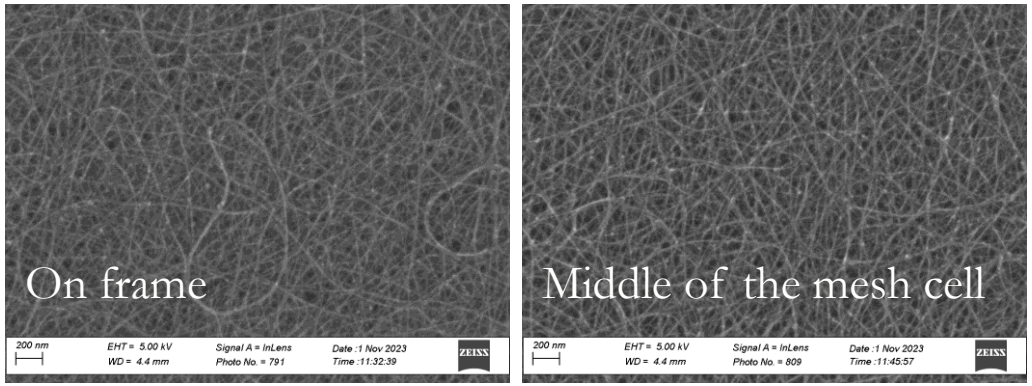


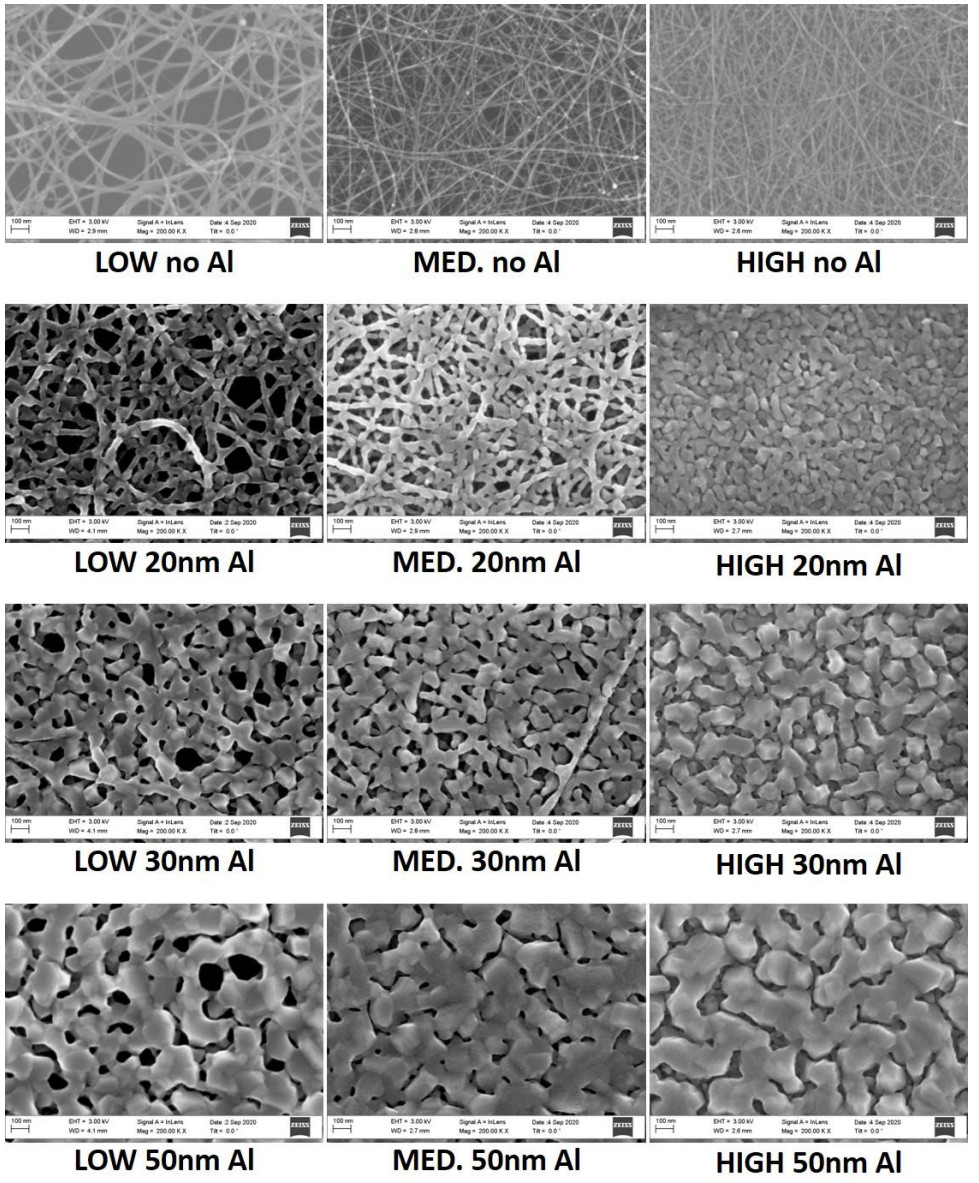



# Al uniformity – LAOF CCN reminder

Looking back the LAOF CCN SEM images one can also see that the CNT density has effect how the Al coating happens.

It can be that form of the CNT membrane is different although such a thing is not observed from the SEM images



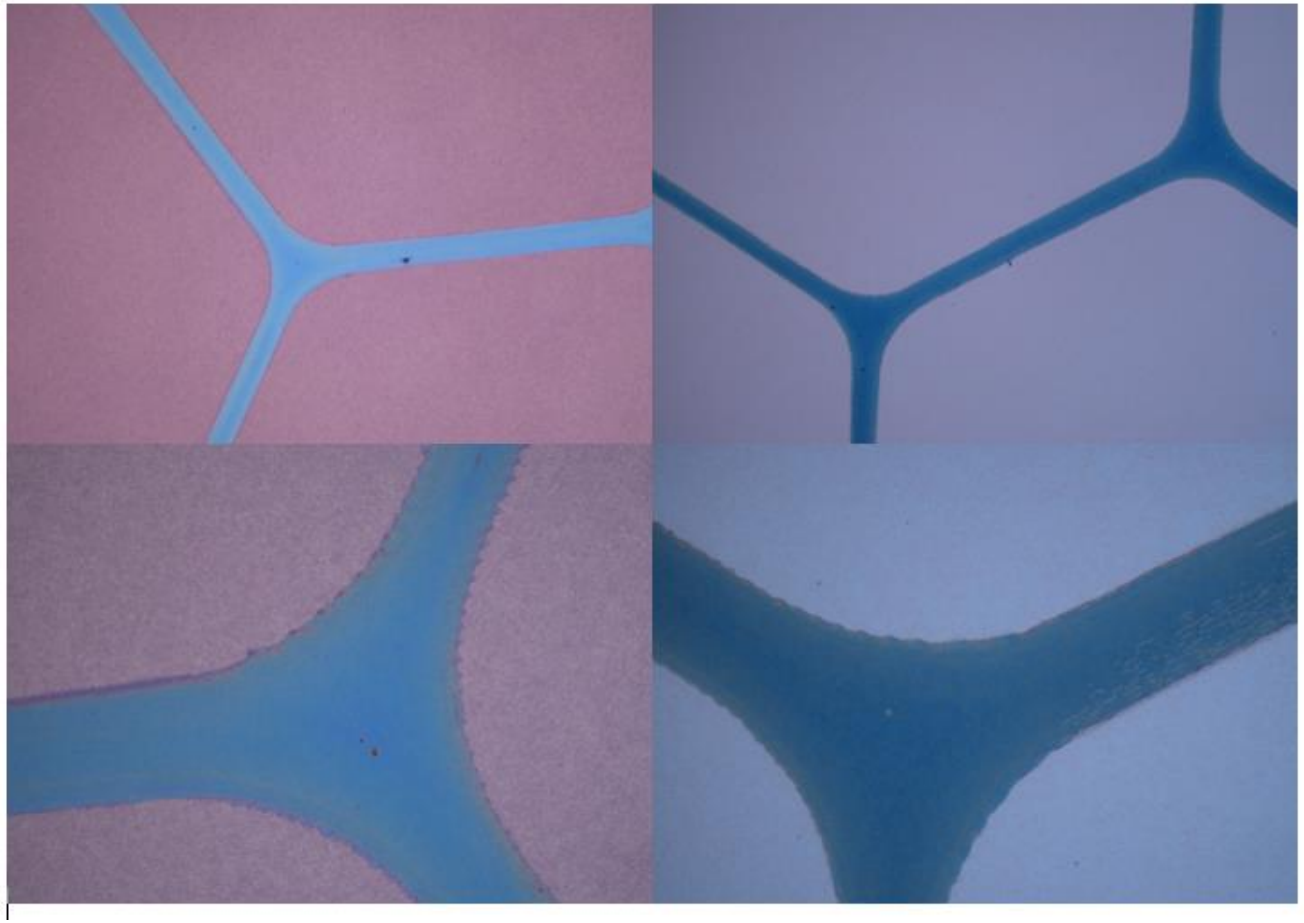
# Typical quality in Batch 5 filters

**(top left)** FC-B3-F1 conservative sample typical good quality

**(top right)** FC-B3-F5 high efficiency sample typical good quality

**(bottom left)** FC-B3-F1 high efficiency sample typical quality of the laser cut on the corners

**(bottom right)** FC-B3-F6 conservative sample typical quality of the laser cut on the corners.



# Observed defects in Batch 5 filters

**(top left)** partial bonding between the mesh and membrane

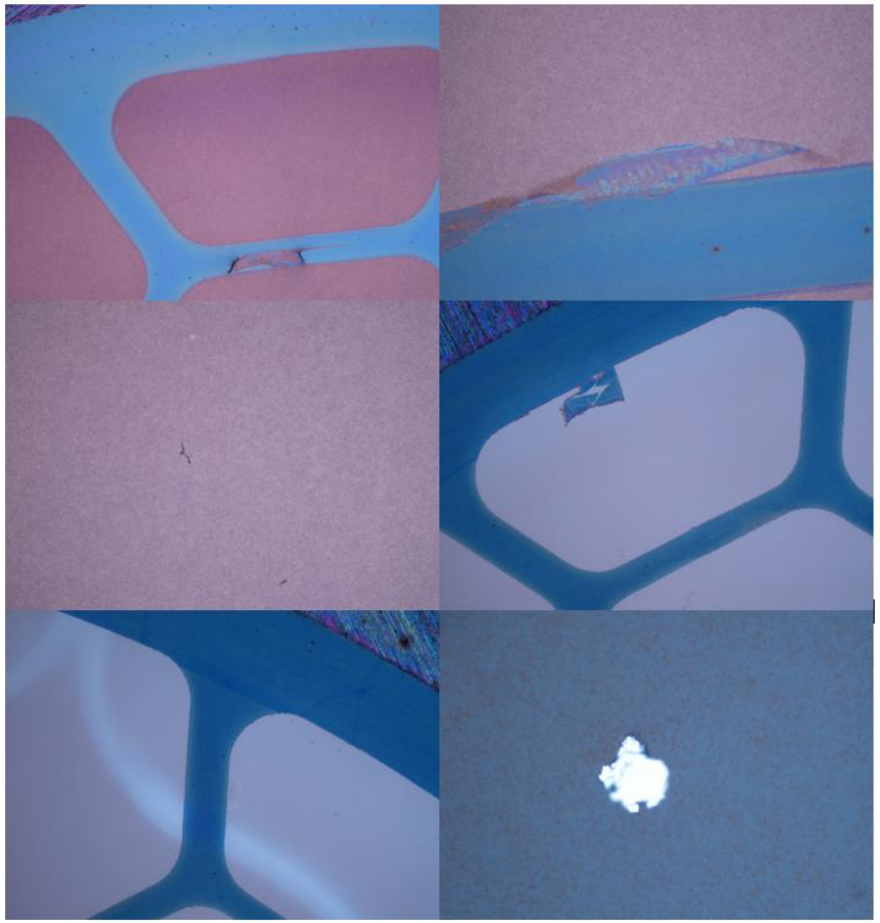
**(top right)** a "hair" like CNT hanging from the mesh

**(middle left)** minor dust like particles

**(middle right)** non proper patterning

**(bottom left)** Drying mark from fabrication process

**(bottom right)** a pin hole



# Conclusions (1/2) of the filter fabrication

- Several batches of the fully carbon-based CNT filter structures have been manufactured in the activity.
- The key ideas
  - Meshes from oriented MWCNT membranes. Mesh becomes integral part of the total filter structure when it is joined together with the CNT based membrane structure. There is strong bonding between the mesh CNTs and membrane CNTs.
  - Uniformity of the SWCNT membranes was improved by utilizing dual layers where two membrane layers are stacked on top of the mesh and each other's. Using of dual layer approach decreases the chance for pin holes and increases the uniformity of membrane.
- Mesh bar width quite high and thickness low.
  - What is the provided strength?
  - Need for further optimization?
- In the inline characterization target filters represented a decent quality. Also, some defects were observed as expected remembering the novelty of the utilized filter technology.

# Conclusions (2/2) of the filter fabrication

- Al coating was found to be more uniform but not necessarily better on fully carbon-based samples compared to metal mesh supported CNT samples.
- The great thing with the fabricated filters was that they are basically free of wrinkles that are typical defect with the metal mesh supported CNT filters coated with Al. This improvement is probably caused by the good bonding between the MWCNT mesh and SWCNT membrane.
- There is a clear need to investigate more the Al coating.
- Patterning process
  - Uniformity of the mesh pattern
  - Flake creation
  - Thicker structures
  - Smaller linewidth

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# Thank you