

TXRF Reference Materials for Cascade Impactor Air Quality Monitoring Systems



Politecnico
di Torino

LAB

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IMA 2021

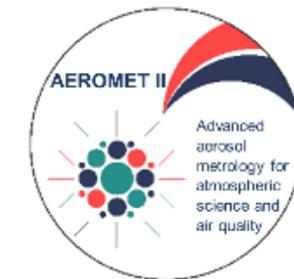
INRiM
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DI RICERCA METROLOGICA



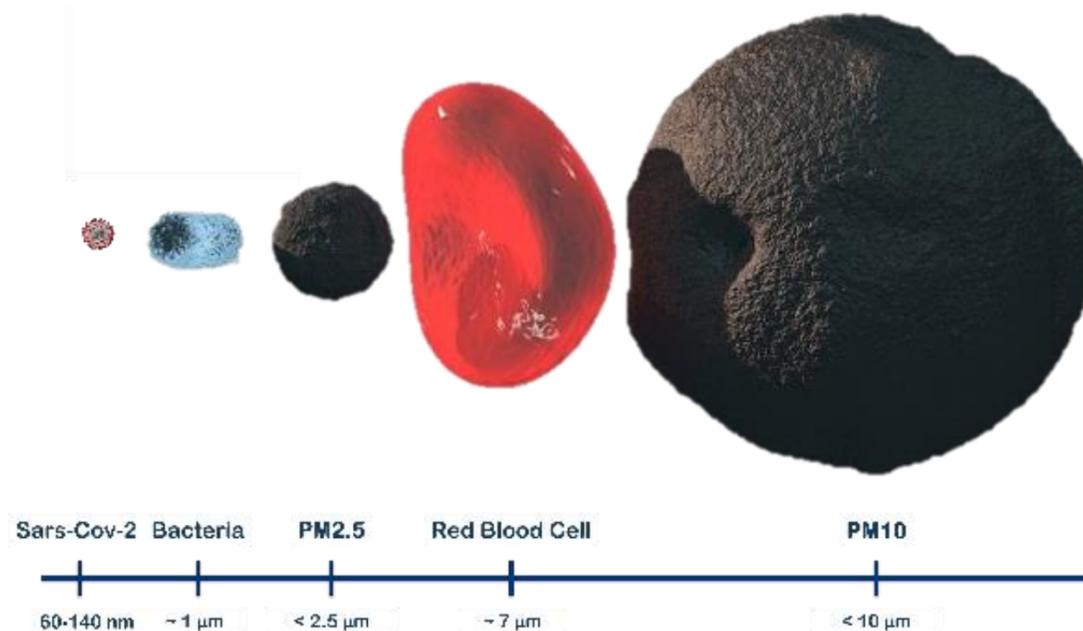
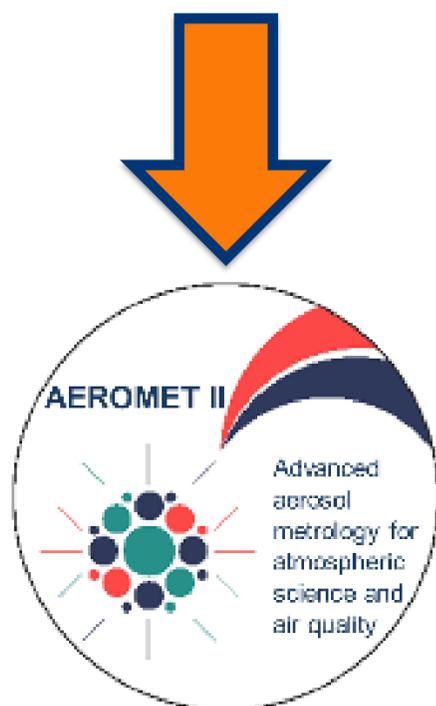
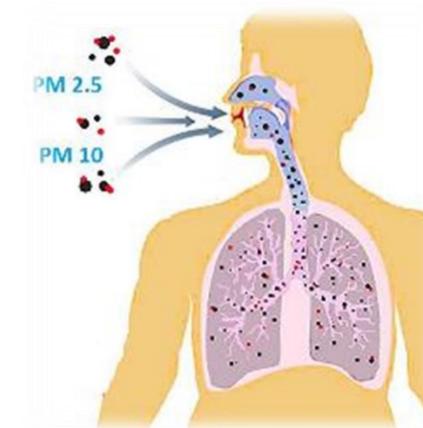
BAM

Sept. 20th - 23rd, 2021

The Problem



- Air pollution is the leading cause of environmentally related health effects and climate change issues.
- Inhalable aerosol particles are the largest contributors and are very harmful.
- EU has developed some air quality regulations that needs to be met.
- Quantitative measurements of aerosol particles are of a vital importance.
- Lack of traceable standards and harmonized calibration procedures for measuring PM.
- The most important metric to monitor particulate air pollution is mass concentration.

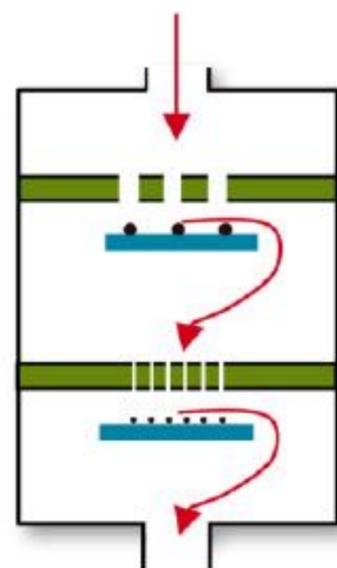


The challenge for metrology

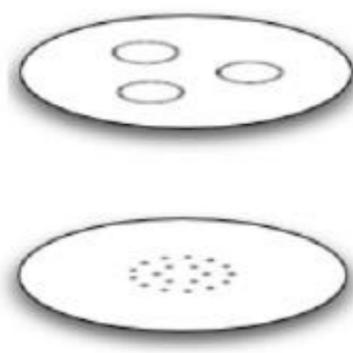
Cascade impactor sampling

Mobile TXRF

Ambient aerosol

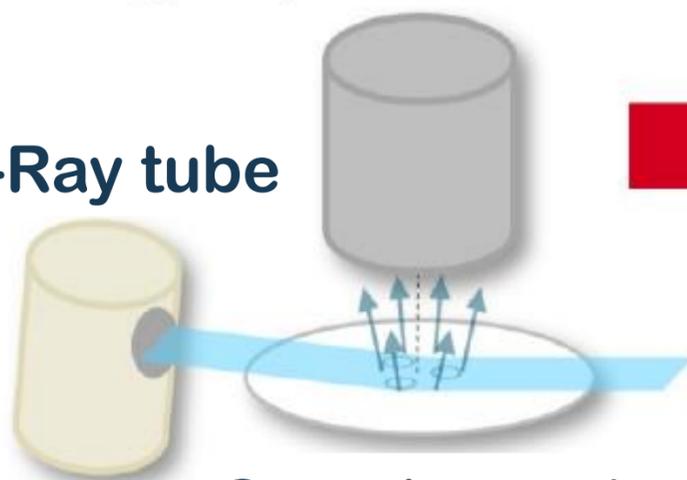


Carriers with aerosol samples

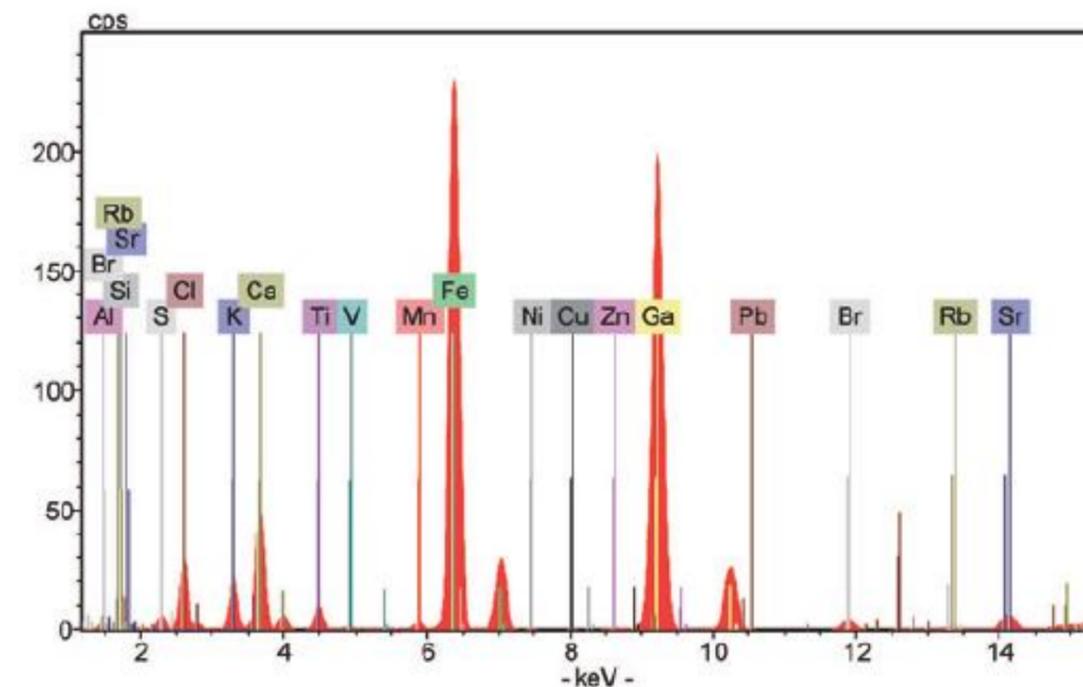


Energy-dispersive detector

X-Ray tube

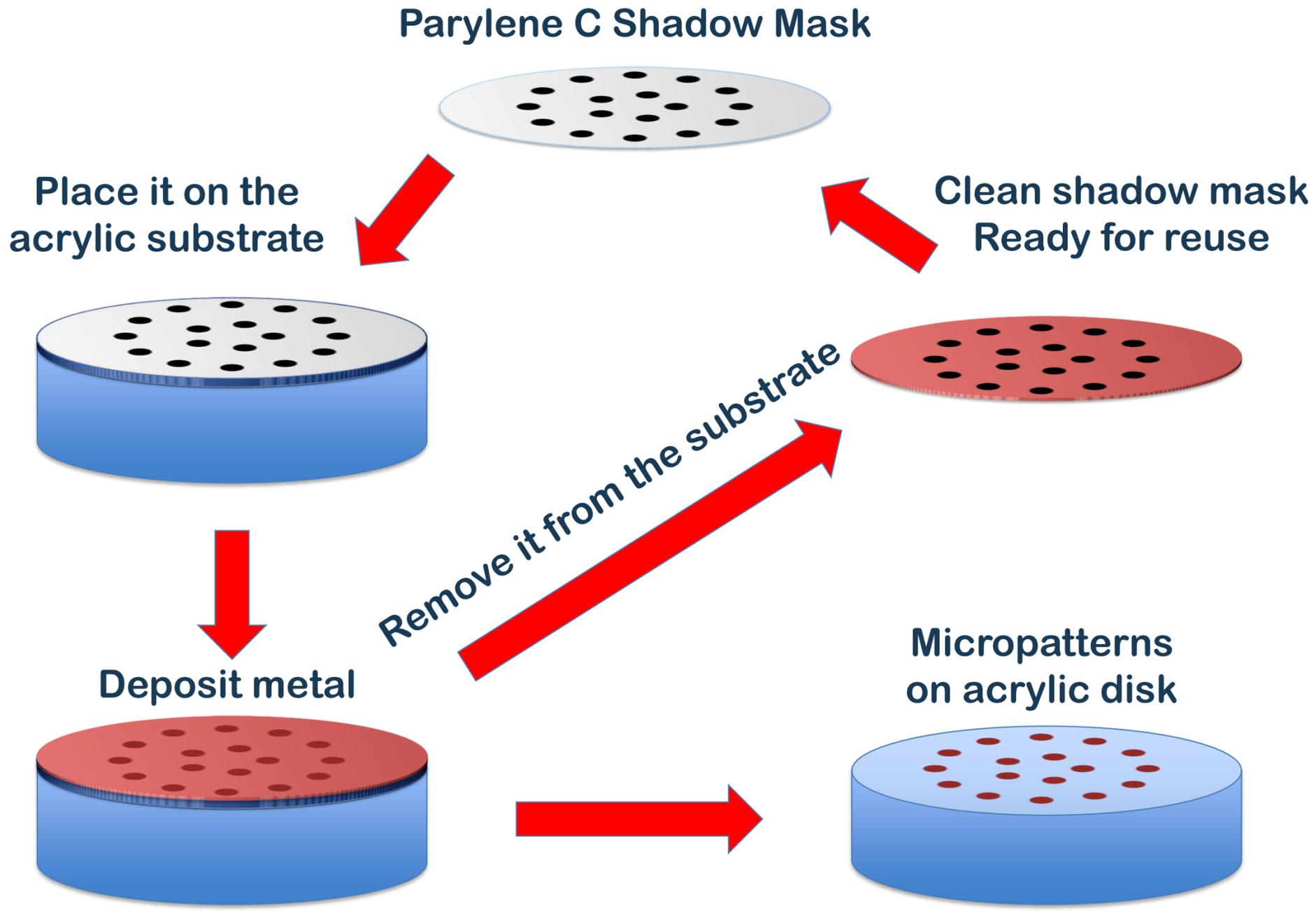


Sample carrier



The goal is to develop **certified reference substrates** and **traceable measurement techniques** for the quantification of regulated and unregulated elements in ambient air with cascade impactor sampling and XRF.

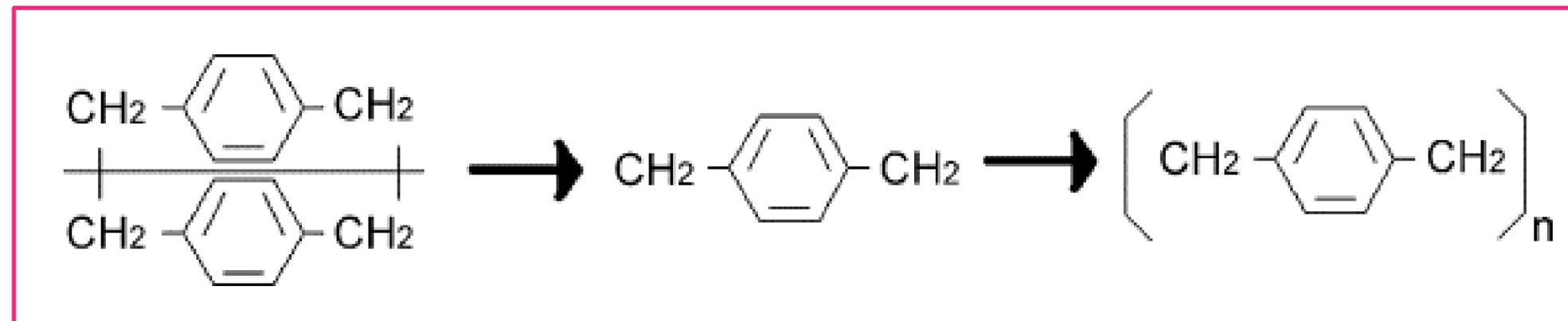
The idea: micropatterning using reusable flexible parylene shadow mask



Parylene C Deposition



Deposition of 7 μm Parylene C layer on a 4" silicon wafer by means of a CVD process performed at room temperature



Vaporization process:
a solid parylene dimer is vaporized at 175 °C

Pyrolysis process:
the parylene gas is decomposed into the parylene monomer at 690 °C

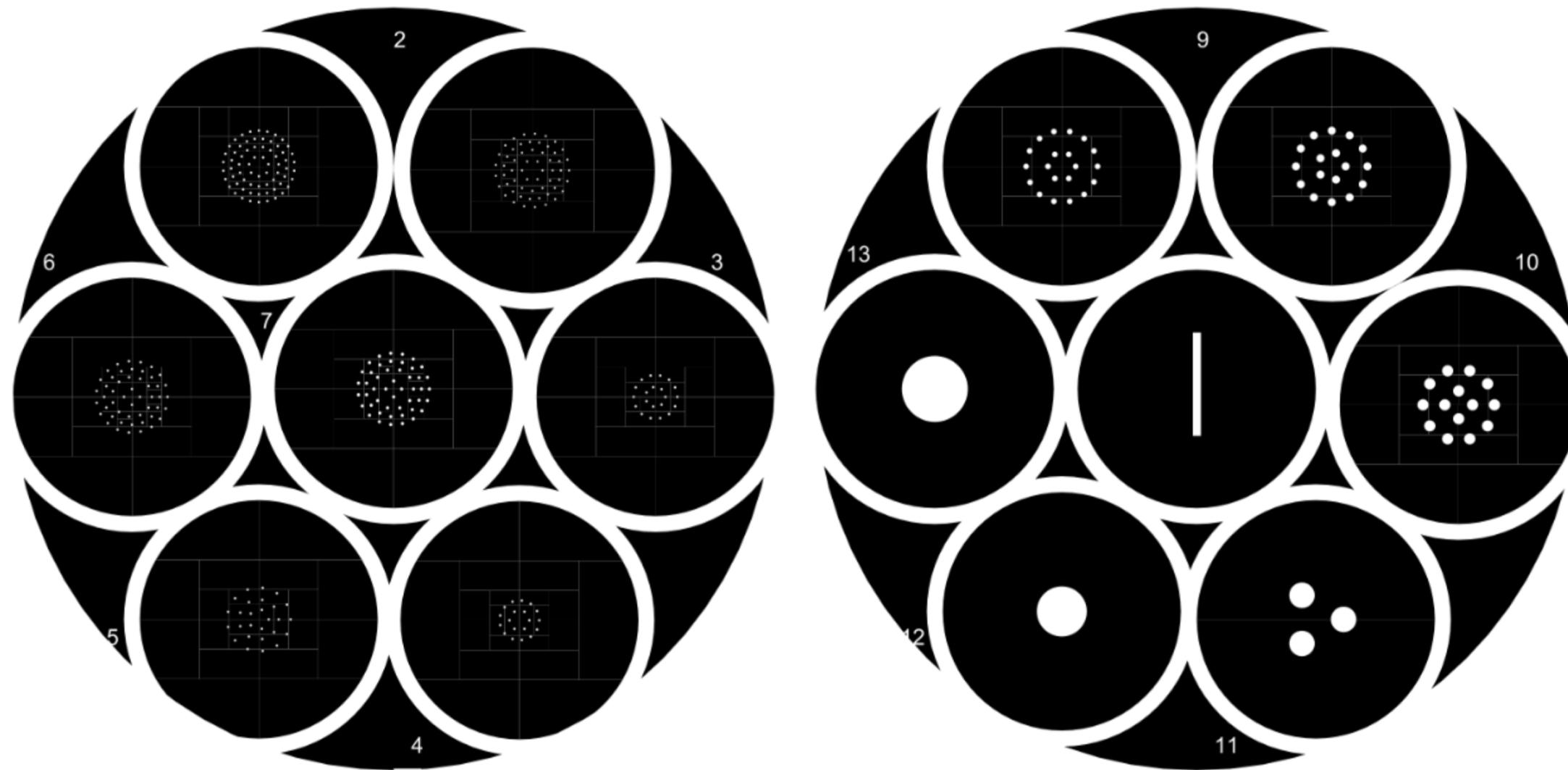
Deposition process:
the parylene monomers get adsorbed on the substrate surface and polymerize at RT

Al Deposition



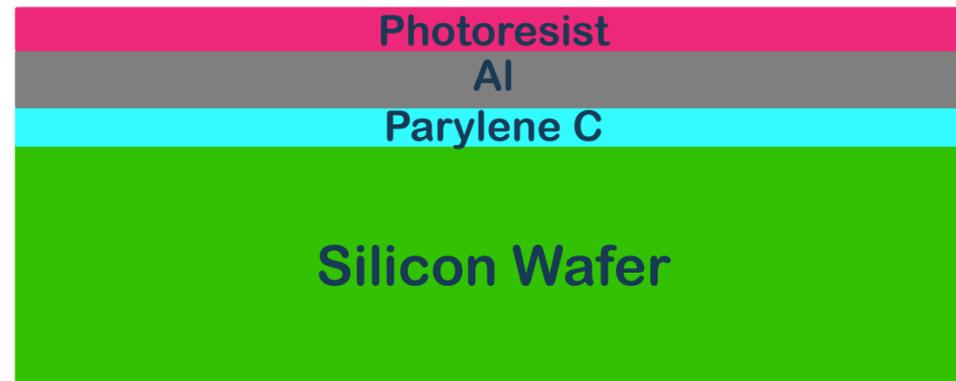
Deposition of 100 nm of Al
used as sacrificial layer
through an e-beam evaporator

Mylar Masks

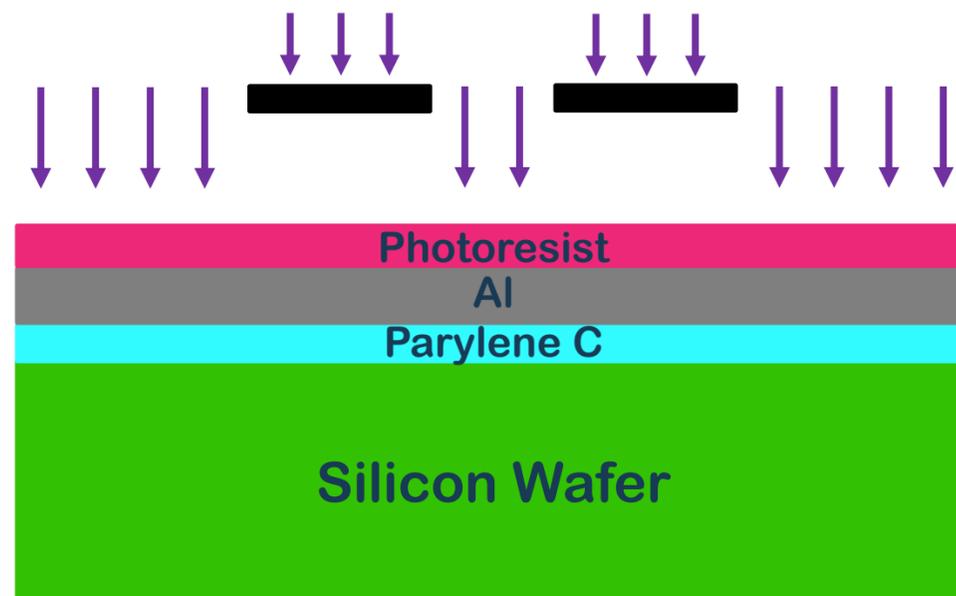


Realization of 2 Mylar masks in order to pattern the 14 stages of the reference samples

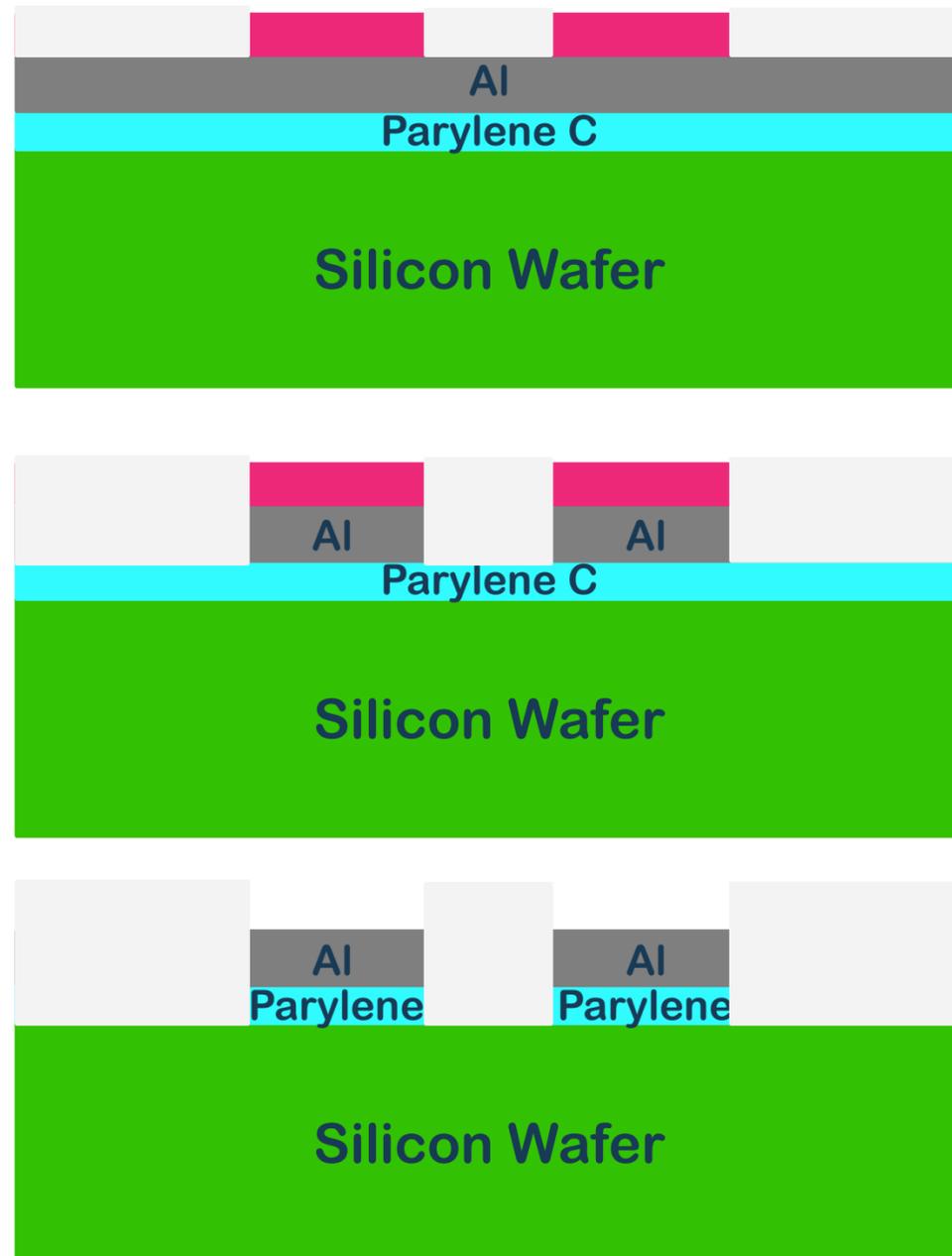
Photolithography Step



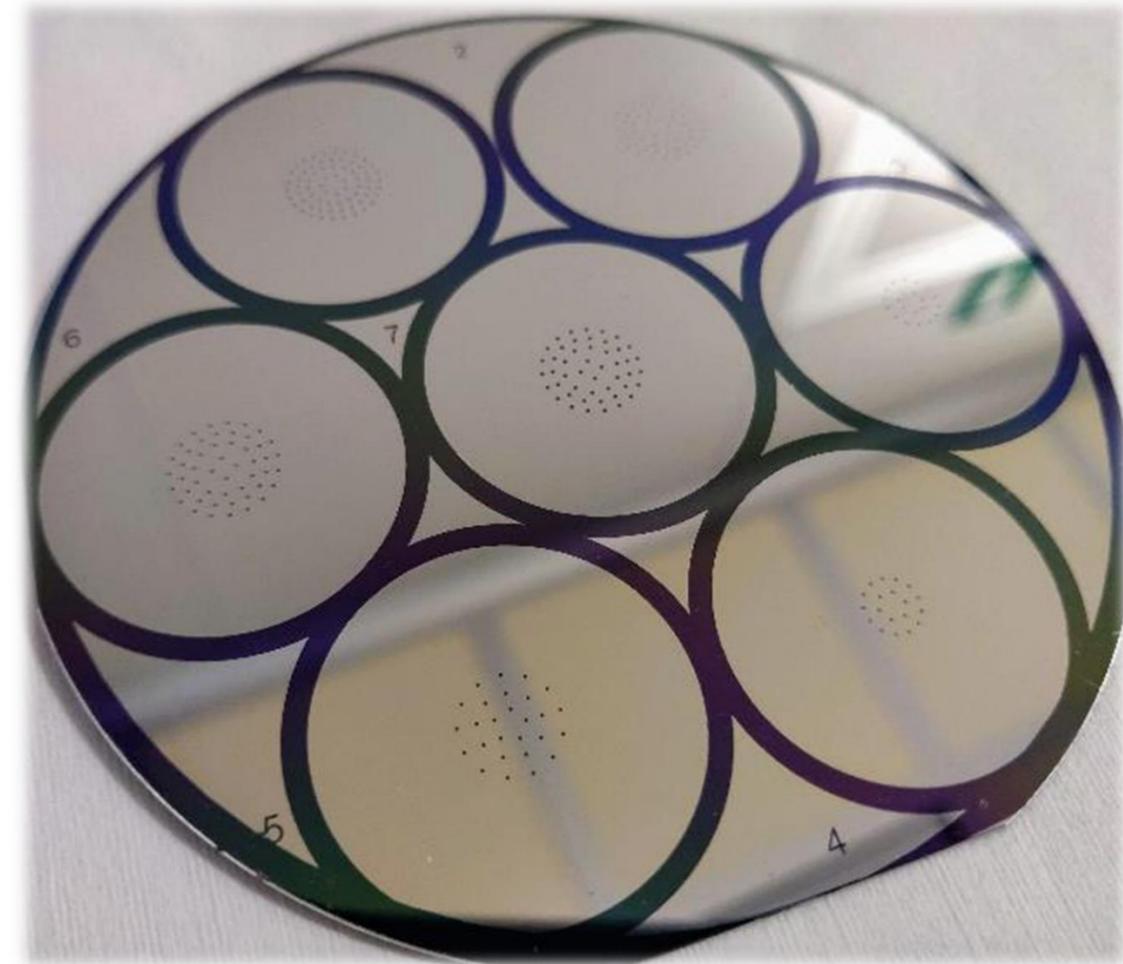
- ❖ Positive photoresist AZ1518 deposition through a spin coater
- ❖ UV exposure for 7" at 10 mW/cm²
- ❖ Photoresist development



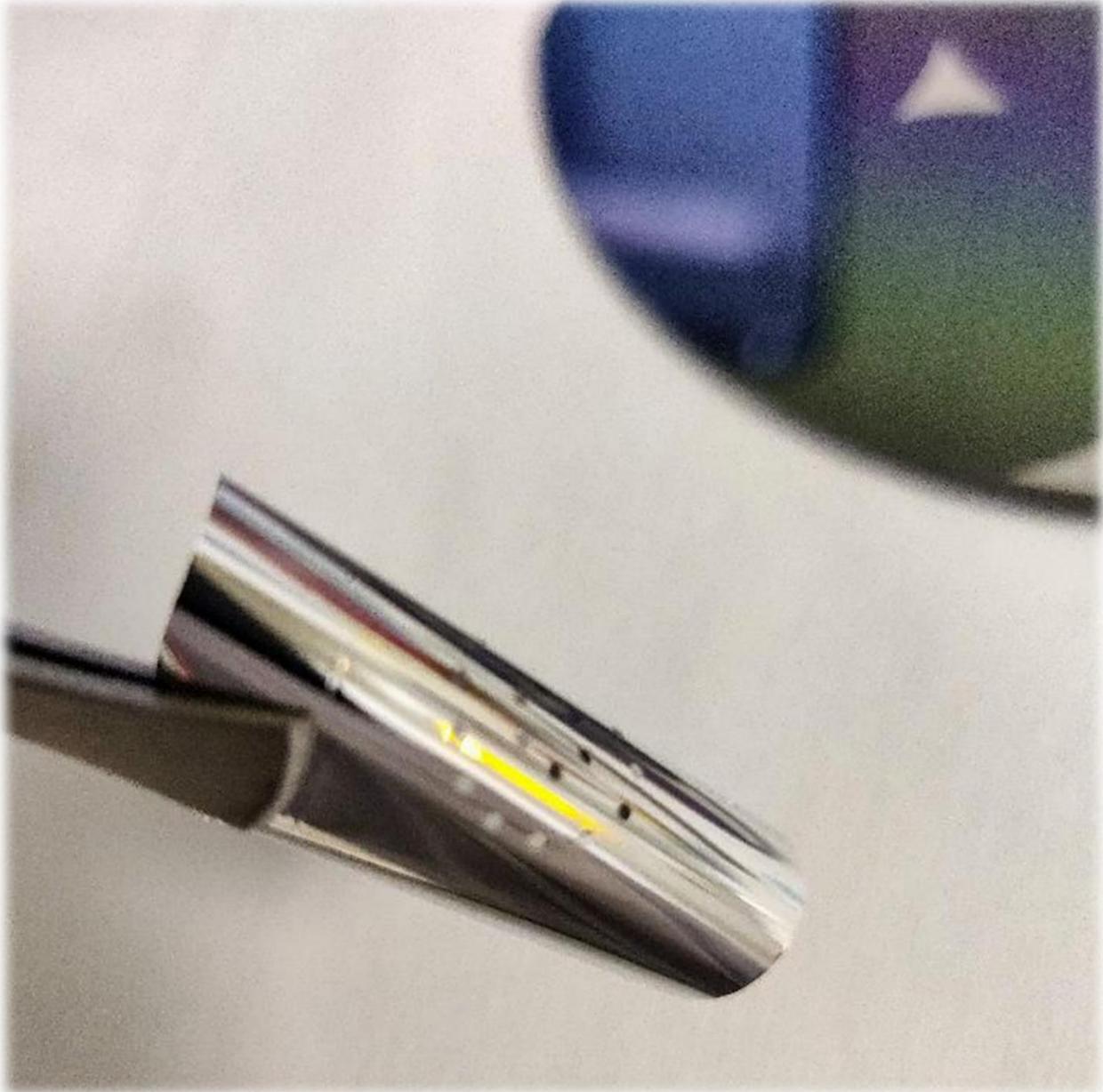
Etching



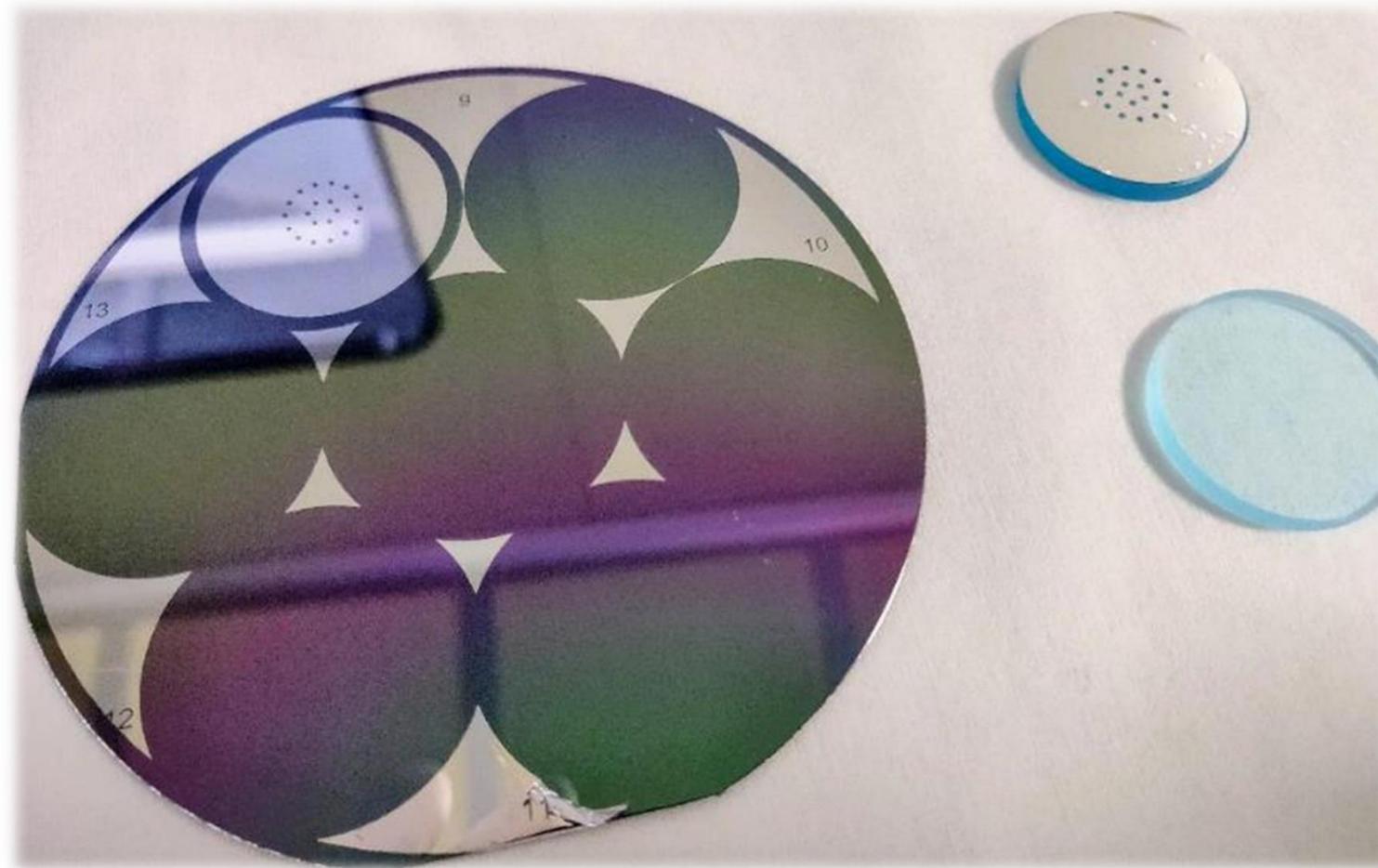
- ❖ Al wet etching
- ❖ RIE dry etching



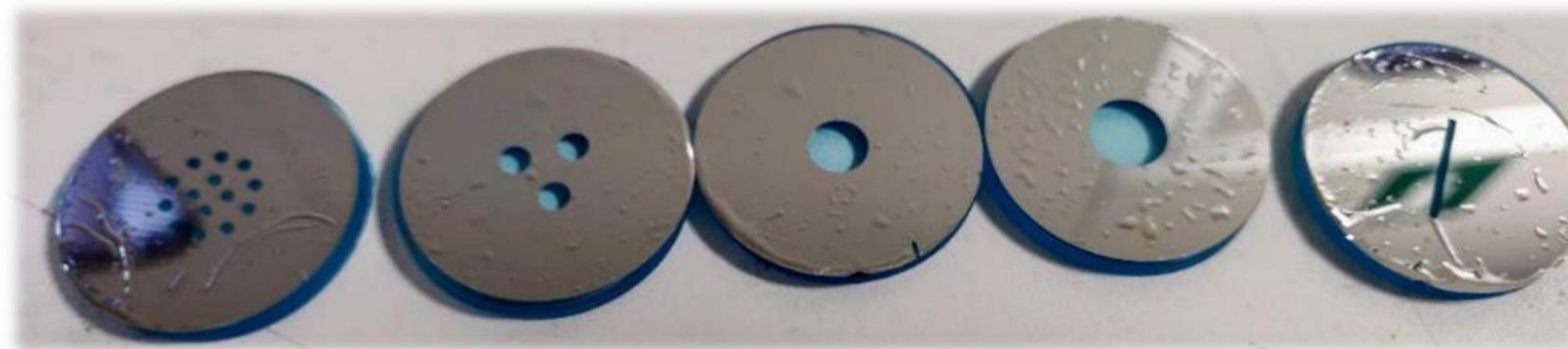
Peel-Off



Transfer Step



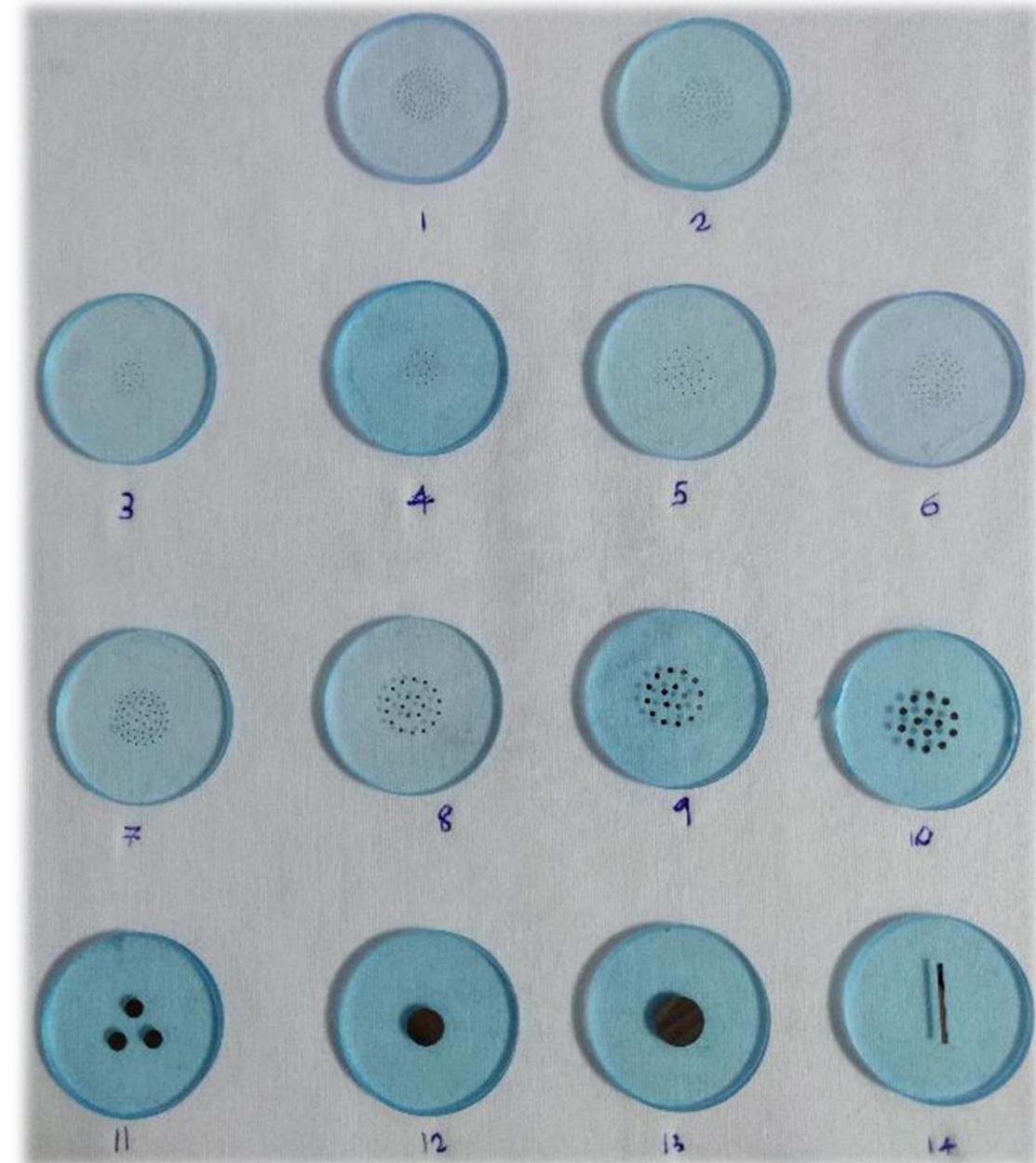
After fabrication, single parylene C masks have been peeled off and transferred on the PMMA disks



Metal Evaporation



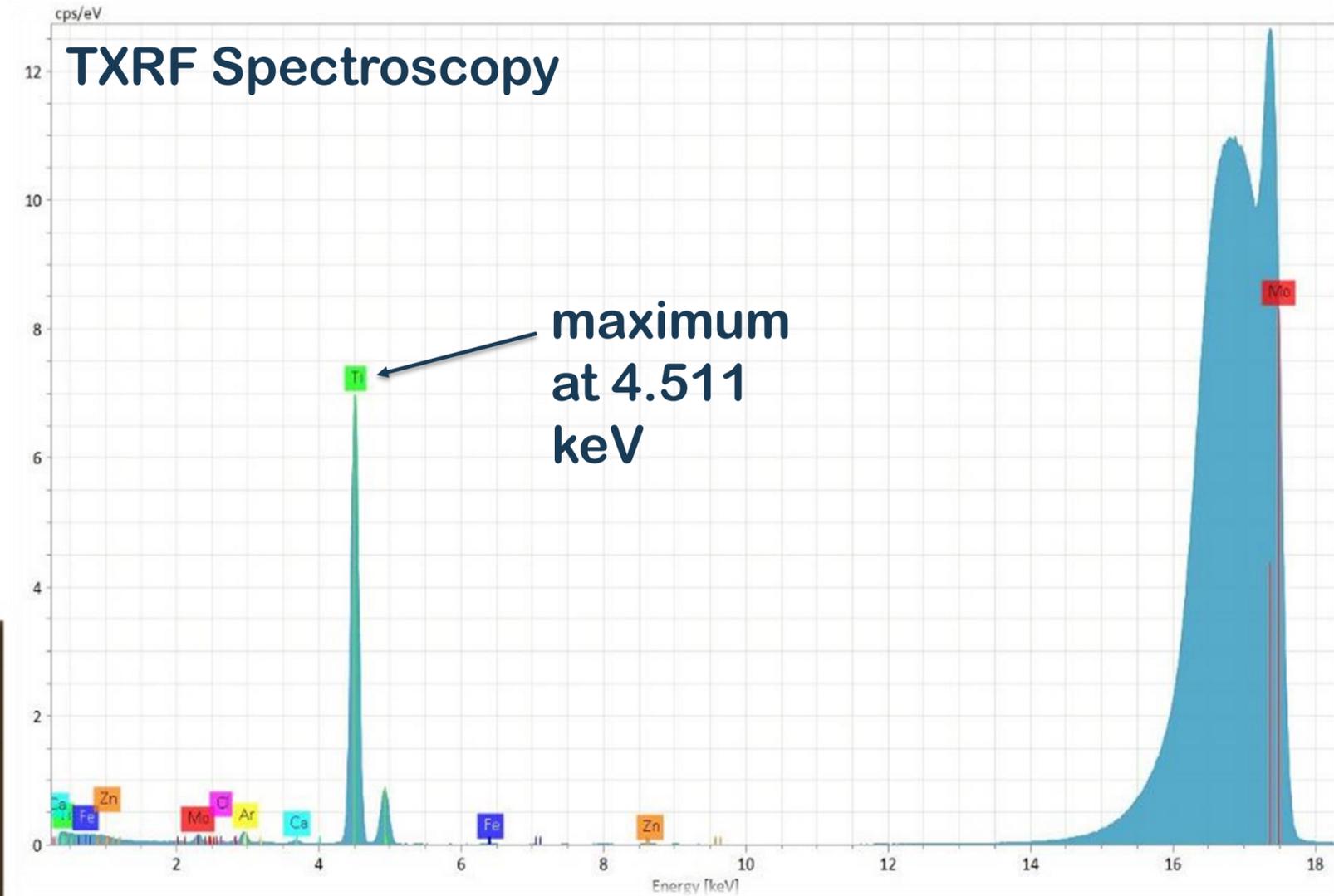
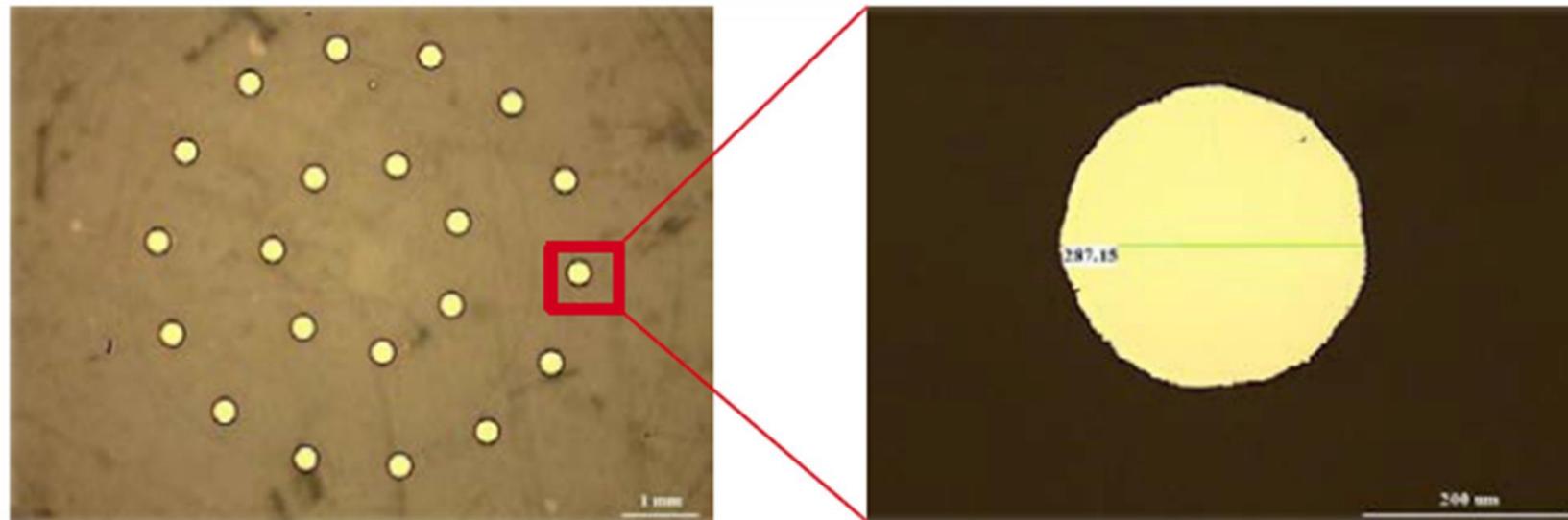
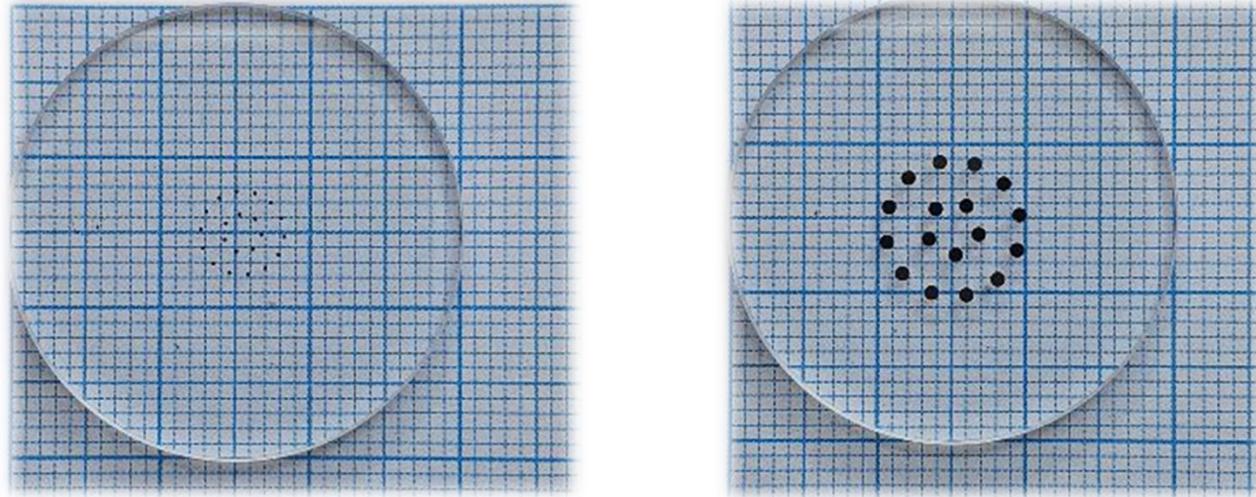
Deposition of 80-90 nm
of Ti through an
e-beam evaporator



Available metals:

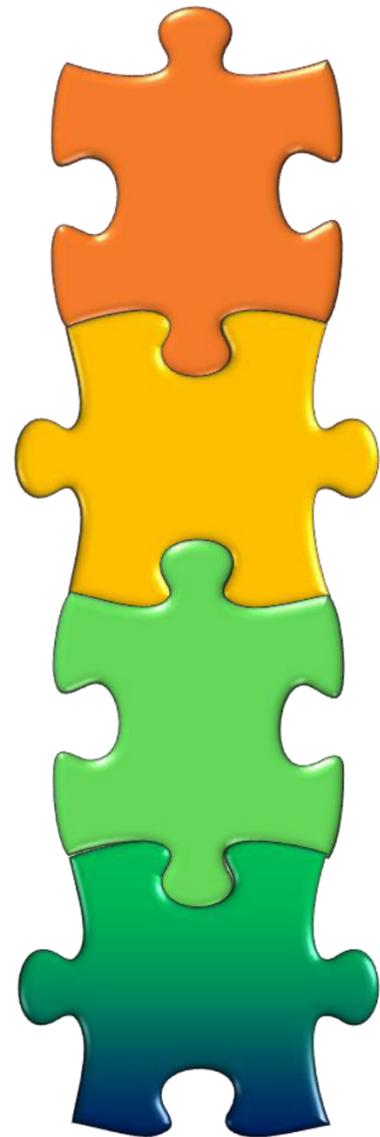
- ❖ Al
- ❖ Ag
- ❖ Mo
- ❖ Au
- ❖ Pt
- ❖ Ni
- ❖ Cu
- ❖ Sn

Reference samples characterization



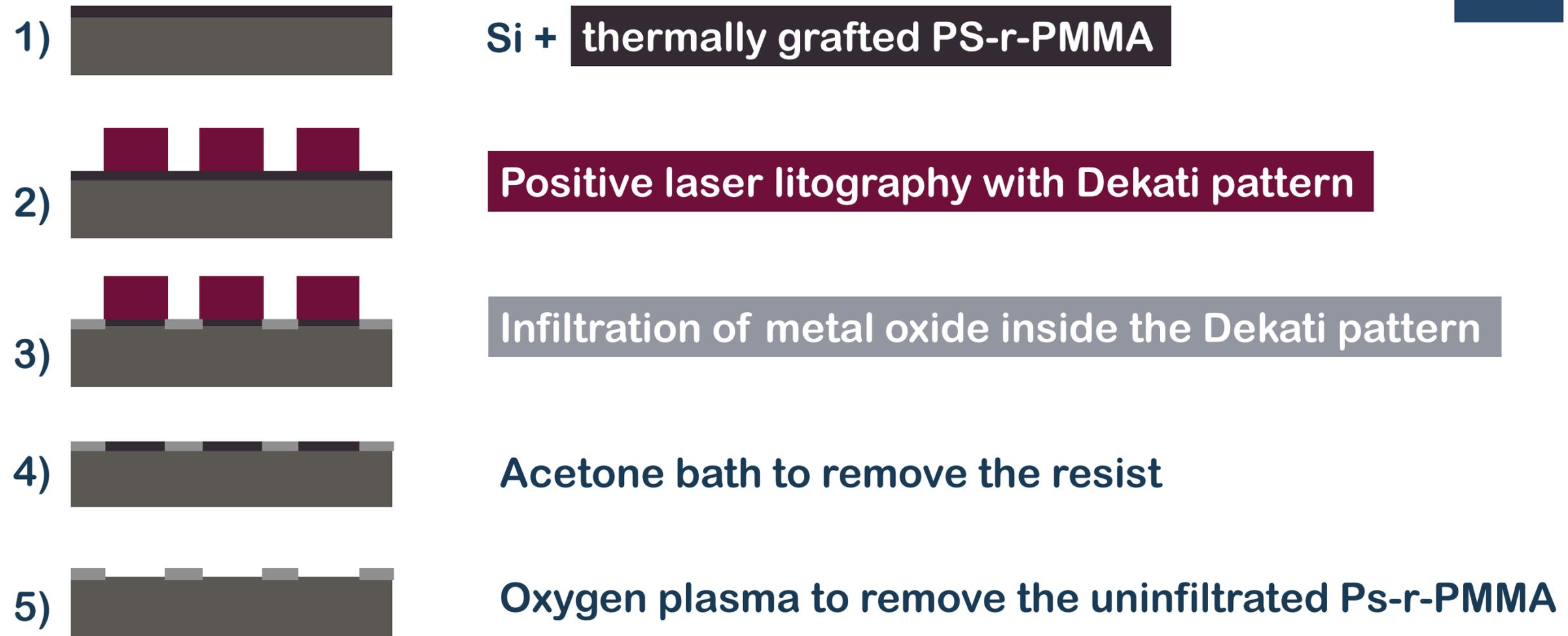
Conclusion

Lasciare o eliminare slide??



- Reference samples with different cascade impactor stages patterns were successfully realized with this technique.
- Highly flexible direct patterning of acrylic substrates otherwise impossible with conventional photolithography procedures.
- Parylene C micro-stencils were successfully reused multiple times without any damage or contamination.
- Good accuracy achieved on features down to 250 μm with an error less than 4%.

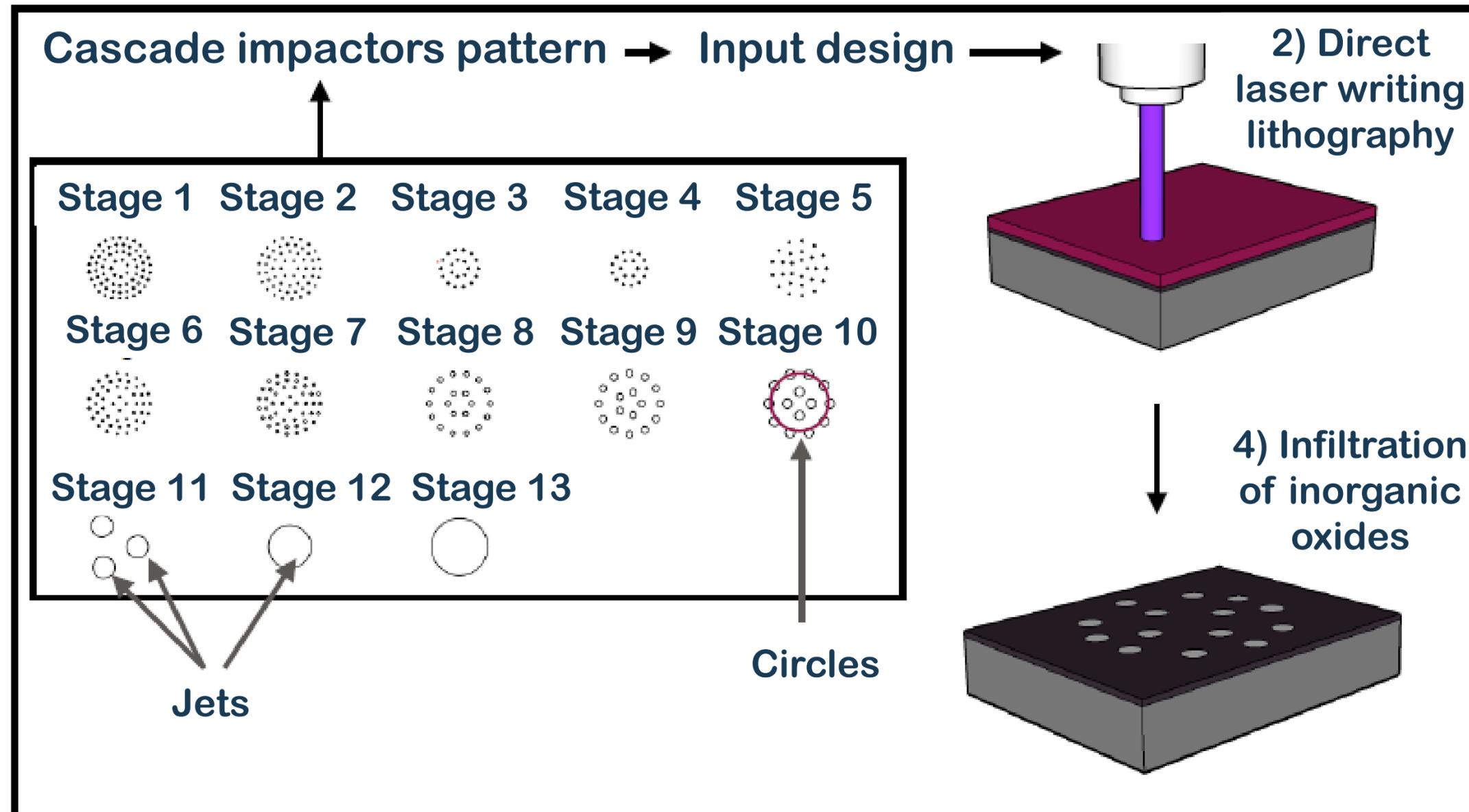
Artificial reference samples by using SIS method inside diblock copolymers



Available metallic oxides for ALD-based infiltration are TiO_2 , Al_2O_3 , ZnO

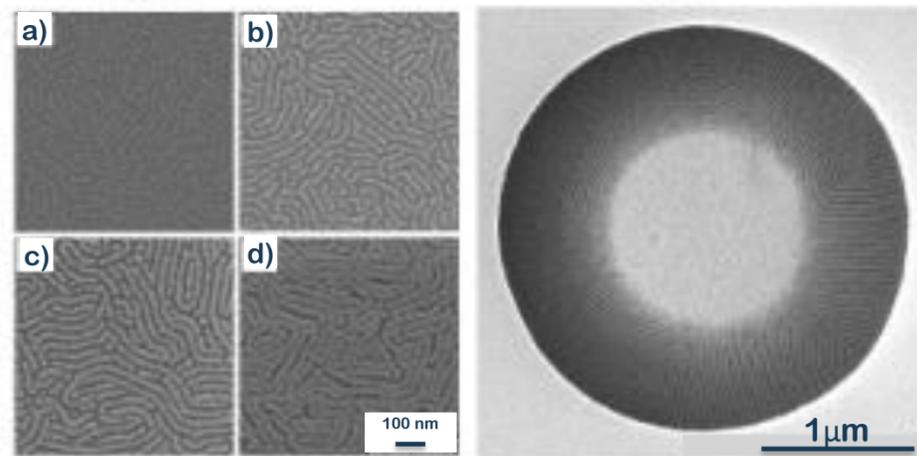
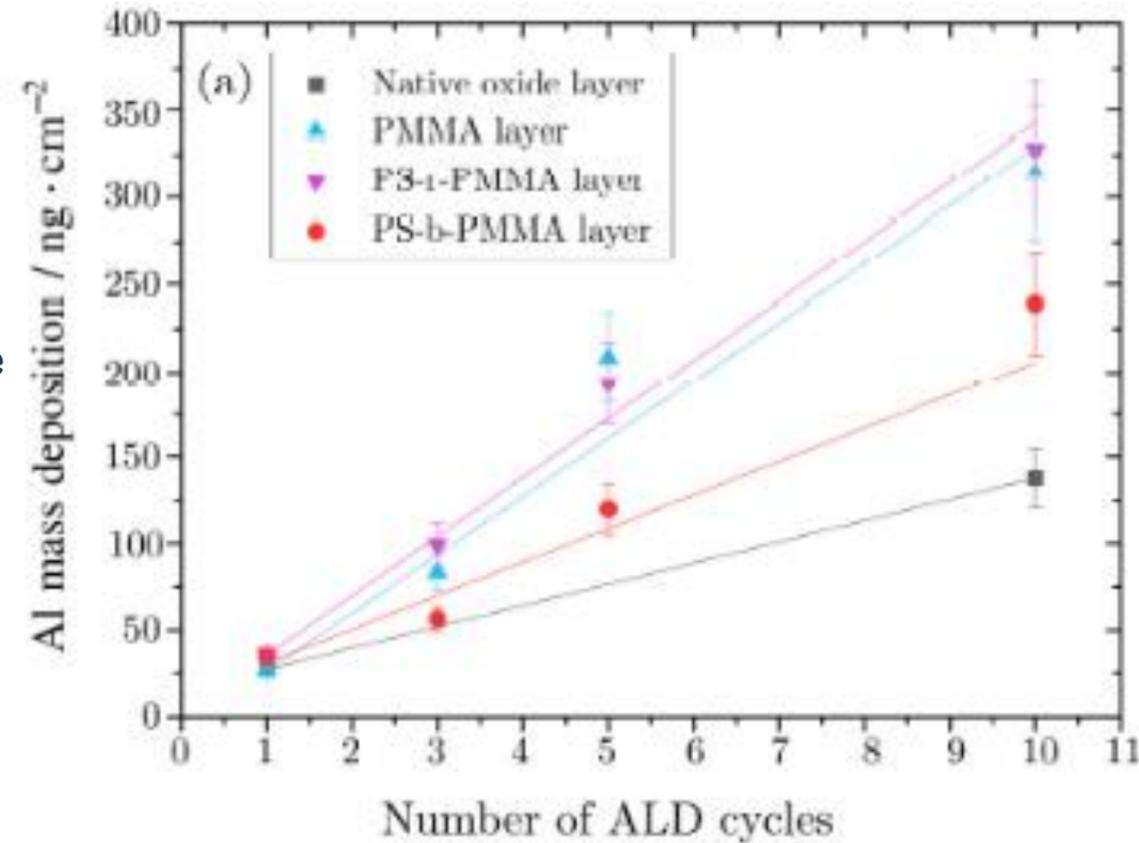
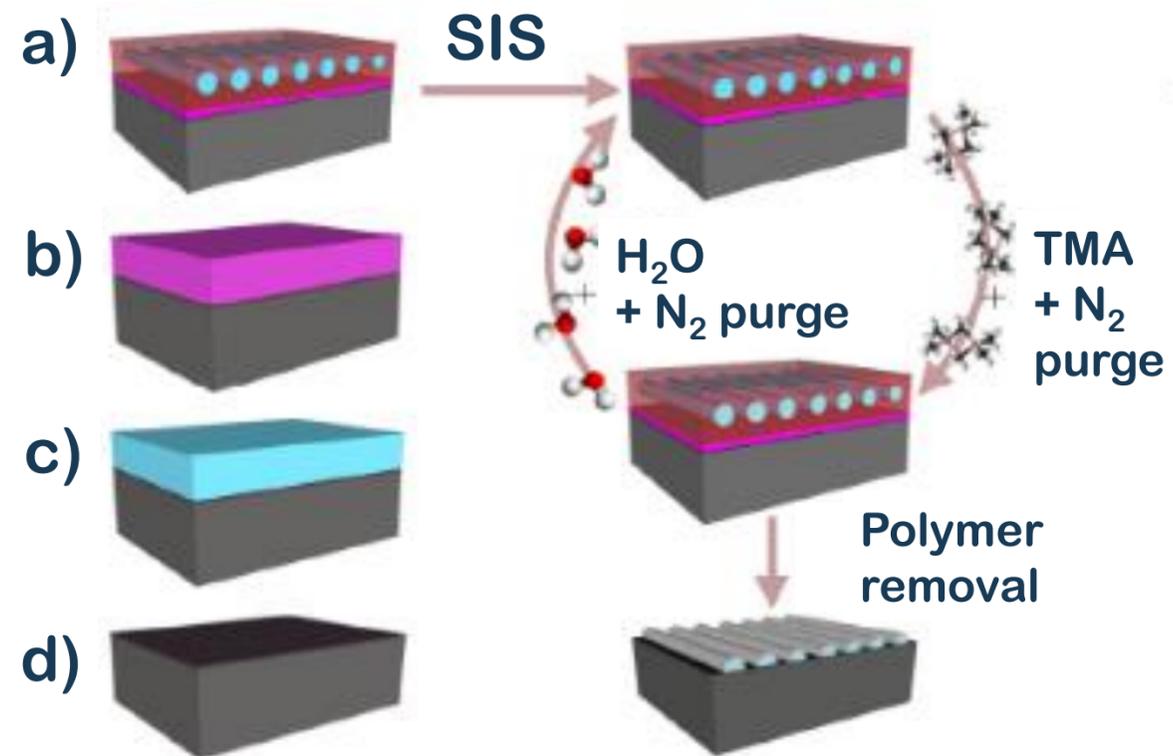
Chemical affinity between the precursor and the polymer functional group is required for the infiltration.

Artificial reference samples by using SIS method inside diblock copolymers



Artificial reference samples by using SIS method inside diblock copolymers

10 cycles of ALD on PS-r-PMMA yields 300 ng/cm² of Al



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**Thank you
for your
kind
attention!**