

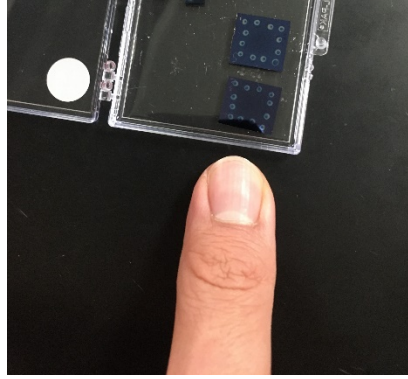


Dry box in Allen B103

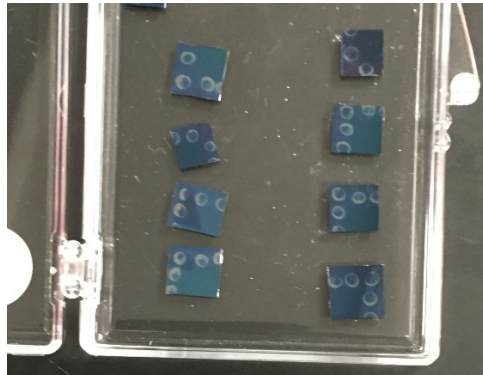
2. SOP

Seed Layer Deposition on CVD Grown monolayer MoS₂



1. Get MoS₂ samples (CVD Grown) from Pop group
 - a. Check the quality from optical microscope / AFM / SEM
 - b. Quality varies from exfoliation / transfer / CVD-grown, so it should be checked before the experiment. Also, CVD-grown samples have variations as can be seen in the table below, so it is important to check them with microscopes in advance.
 - c. Substrate roughness should also be checked since monolayer 2D materials will be deposited which has thickness of $\sim 0.65\text{nm}$. Roughness of the sample should be on the order of the sample substrate.
 - d. Other CVD-grown 2D materials are also available in Pop group, so for any collaboration, contact Prof. Eric Pop. The chip size is $\sim 1\text{inch}$ by 1inch . For more experiments to be done, it can be cleaved into pieces exactly as is done with Si wafers. The circles at the edges are Perylene-3,4,9,10-tetracarboxylic acid tetrapotassium acid salt (PTAS) which helps grow MoS₂ towards the middle. These parts are not considered important after the MoS₂ CVD process, so this may be a good position to place sample holders. PTAS contains Potassium, and some K is found at the surface of the samples (verified with Auger electron spectroscopy).

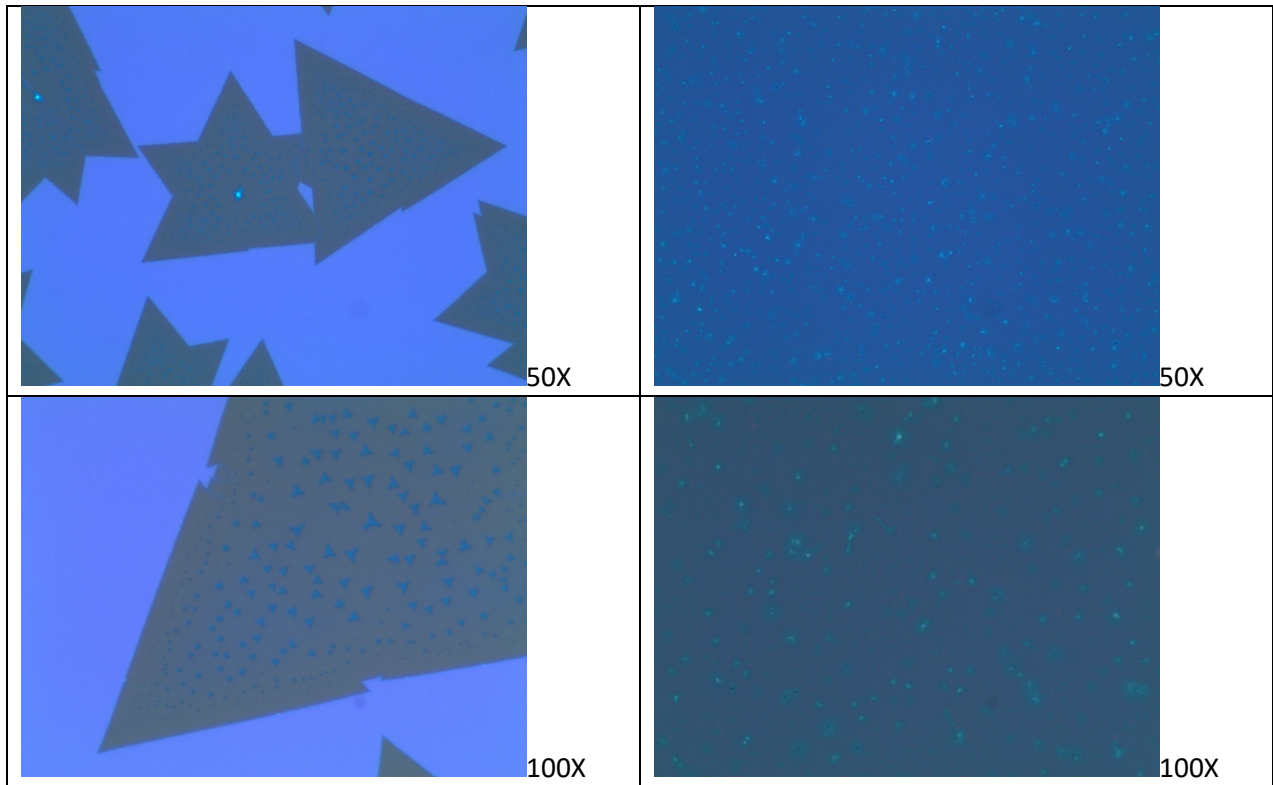


Two MoS2 CVD grown sample chips and chip size



MoS2 sample chips cleaved into 4 pieces

Sample #1 (sample with less continuous growth)	Sample #2 (sample with very continuous growth)
 <p>5X</p>	 <p>5X</p>



MoS₂ samples under optical microscope to show sample variation

2. AFM / SEM on bare MoS₂ samples

- a. Since monolayer MoS₂ is being grown, the expected roughness should be approximately that of the underlying SiO₂/Si substrate. Small amounts of additional roughness are due to residues from MoO₃ involved in the growth process. This value may vary based on the substrates being used, and for our samples, the roughness before the deposition is measured to be ~200pm.
- b. Bilayer and grain boundaries can be seen, but no devices are made at the grain boundaries or bilayer regions, so measurement should be done on areas where it seems to be clean monolayer MoS₂.
- c. If the chip size is large, and we want to perform parallel multiple experiments, samples with 2D materials can be cleaved in the same way as Si wafers. Before any deposition on the samples, it is always a good practice to blow the surface of the samples with an N₂ gun to reduce the likelihood of dust causing problems.

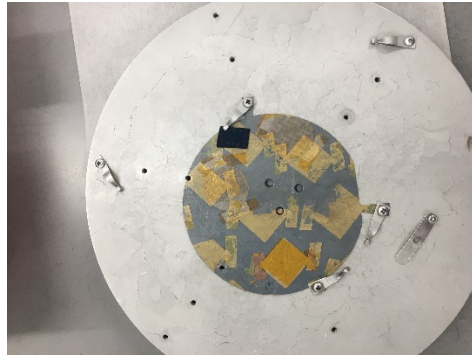
3. E-beam evaporate metal on MoS₂ surface with AJA

- a. Blow the surface of the samples with N₂ gun which is at the side of AJA



N2 gun placed at the side of AJA

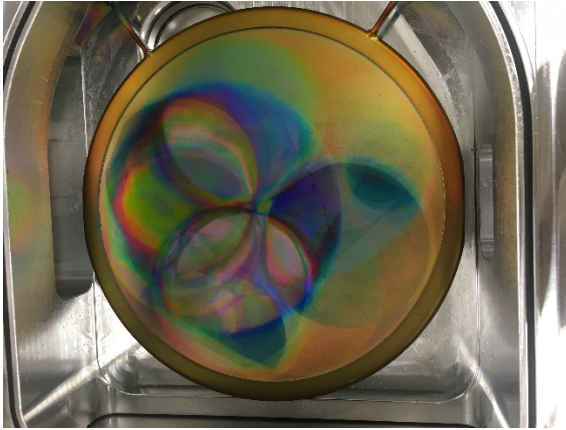
- b. Place the samples on the sample holder plate (multiple samples can be held)



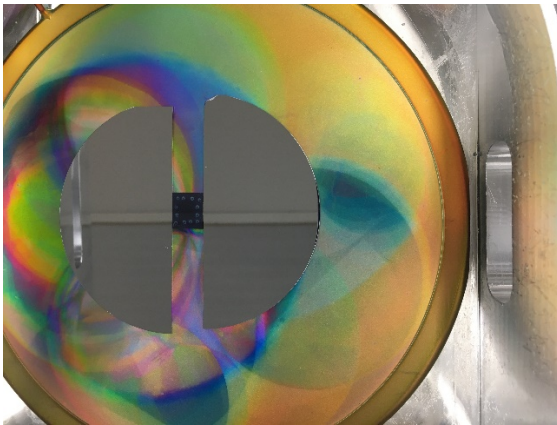
Samples placement: make sure it is attached firmly since the plate will be hanging upside down

- c. In case of aluminum, use the Al_0.2A/s deposition recipe.
 - i. The deposition rate should be watched closely since this may be unstable from time to time.
 - ii. The Al_0.2A/s is developed for thin Al film deposition, so use this recipe for seed layer deposition.
- d. Deposit metal at the target seed layer thickness
4. Remove the samples from AJA. In the case of alumina, the seed layer will naturally oxidize; it has been verified from other group members that the full depth of the seed layer is oxidized for up to a 2nm seed layer using XPS.
5. AFM / SEM on the samples after e-beam evaporation
 - a. Since 2D materials have not been well characterized with ellipsometry tools, thickness can be measured from step height from AFM. For our case, we determined the step height using equivalent experiments on Si substrate and extracting the thickness from that

6. ALD using standard recipe on 2D samples with Fiji 2



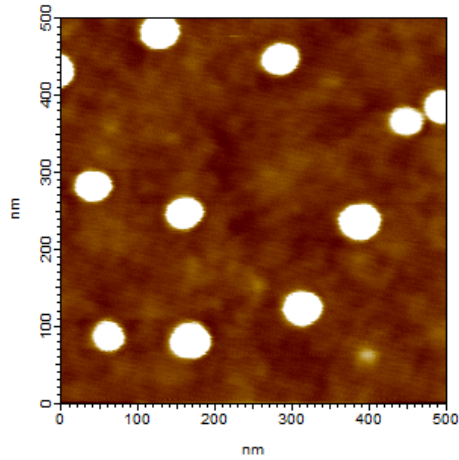
Inside Fiji 2 loadlock



Sample placement when using small pieces

- a. If samples are pieces, they can be put in load lock using pocket wafer (process for making one is in Fiji 1/2 wiki page). Another option which may be simpler is to put test wafers around the sample so that it does not move when loading and deposition process.
 - b. When retracting the arm to drop the plate back at the chamber, it must be done carefully so that the sample does not move significantly. Otherwise, the samples may move a lot, or in the worst case, flip over.
8. AFM / SEM on the samples
- a. Measurement should be done at a small scan area, since using a larger scan window tends to average out roughness. Recommended window for small area scan is 500nmX500nm.
 - b. Large scan area is helpful for understanding the surface topology, and helps identify and measure grain boundaries, bilayer regions, residues, pinholes, etc.
 - c. During measurement, some areas seem to include multiple island-shaped dot regions. This is likely due to either AFM tip artifacts or real island-shaped residues. Whether they are artifacts or real islands can be verified by measuring the same scan area after rotating the samples. It can also be simply verified from line scan of the areas whether

the islands have a distinct height, and also from phase scan information. From our measurement, the height of the island showed $\sim 1\text{nm}$ and phase information showed that it is a different material from the background material. As a result, we believe the islands observed are real residues. Image below shows some residues which are in color white, and these areas will be more easily found in grain boundaries. The measured height of the white islands in the image were $\sim 1\text{nm}$.



AFM image (500nmX500nm) with MoO₃ residues on the flakes (white dots)