

Exercise 1: MoS₂ transfer

Location: LANES laboratory, BM 2133

Contact: Zhenyu Wang, zhenyu.wang@epfl.ch

1. Summary

In this exercise you will carry out the transfer of MoS₂, grown on sapphire using the MOCVD process. The chips produced in this way will then serve as the basis for subsequent steps of device fabrication and later, characterization. The exercise will be carried out in the LANES laboratory (Prof. Kis).

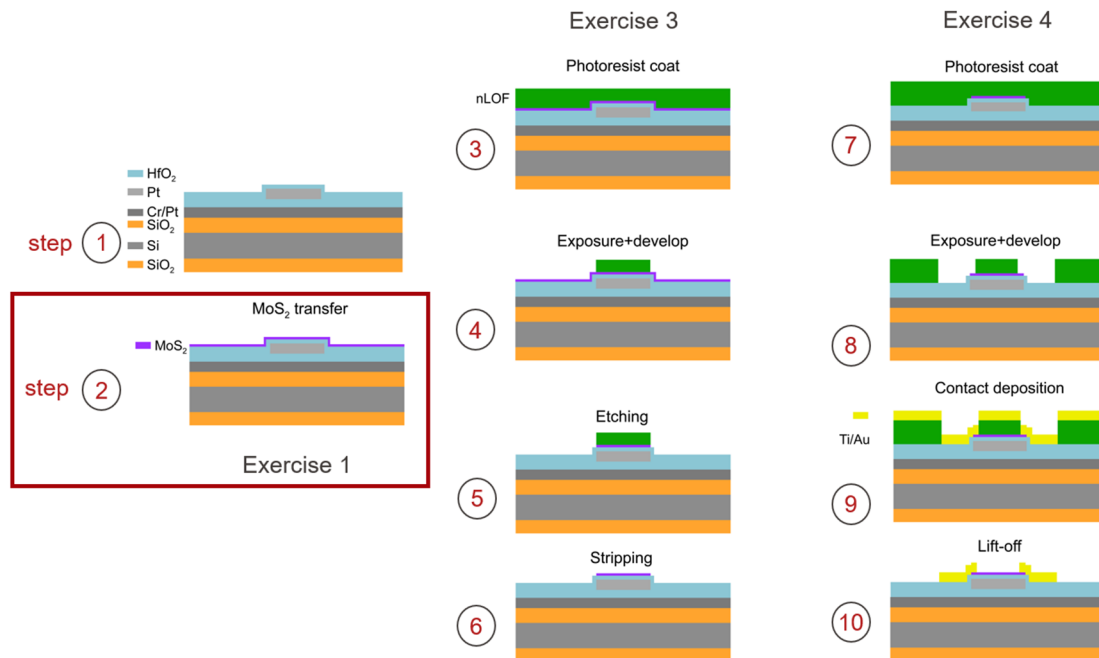


Figure 1. Overview of the complete process flow for the device fabrication in this course. The current exercise deals with step 2, the transfer of MoS₂ from the growth substrate to the device substrate with the pre-patterned gate stack.

2. Background

There are several ways of preparing 2D materials for devices, each with its advantage and disadvantage. The historically first approach was based on the so-called micromechanical exfoliation or “scotch-tape technique”, referring to the preparation of thin films by attaching a piece of adhesive tape to a bulk crystal of for example graphite or MoS₂. The tape is then slowly and carefully peeled off, placed on top of a target substrate (for example silicon) and removed slowly. With some luck and skill, it is then possible to have several small pieces of 2D crystals on the substrate. While easy to implement, this technique is not really very useful for fabrication of more complicated devices and circuits and therefore only has its use for

laboratory research. You will encounter such exfoliated samples in the next exercise, where you will do basic optical inspection and characterization of the 2D film.

3. Detailed description of experiments and tasks

Following is the overview of the tasks and operations to be carried out in this exercise. The main goals are to produce two samples. For the first one, a single MoS₂ layer will be transferred from the growth substrate onto the chip with the gate stack. Work with two chips in parallel. In the second task, you will take one of these chips and repeat the transfer process, resulting in a second chip with two MoS₂ layers on top of each other. In this way, you will be able to compare both chips in the subsequent exercises.

3.1. Single transfer of MoS₂

The procedure for the transfer is outlined in the following recipe:

1. Clean the target substrates using oxygen plasma

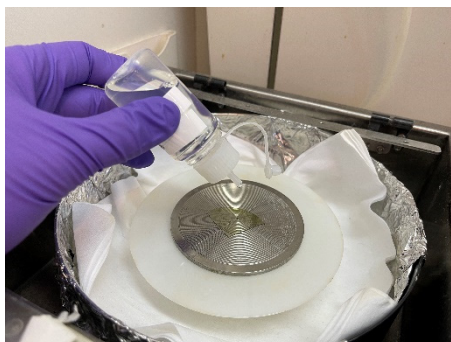
Together with the assistant, clean the target substrates in the LANES' group oxygen plasma cleaner in order to remove carbon contamination from the surface. This will ensure stronger adhesion between the 2D film and the substrate.



2. Coating PMMA A2 on MoS₂ wafer

Spin-Coating: 1500 rpm/60s with 10s acceleration and deacceleration time;

Baking: 180°C/ 5min

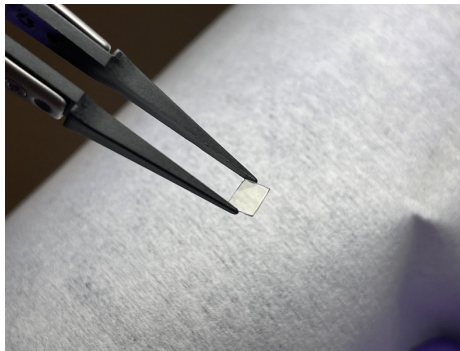


Coat the surface of the sapphire chip with a PMMA A2 resist (polymethyl methacrylate dissolved in anisole, 2% weight concentration). PMMA is chemically identical to Plexiglas and is commonly used as a resist for electron beam lithography. We use it here because it

can be coated into a thin film, because it is relatively robust and can be easily removed when no longer needed. The purpose of this step is to add a relatively thicker film on top of the 2D material to protect it so that it can more easily withstand the following steps. At the end of the transfer, we will remove the film.

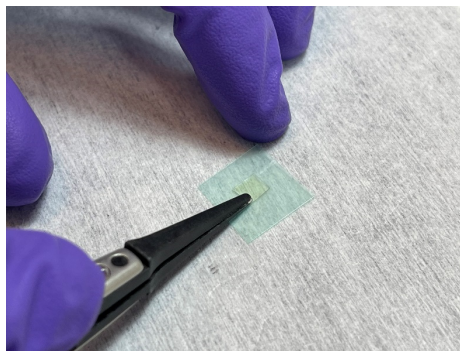
Coating is carried out using a spin-coater, a machine that spins the substrate to relatively high rpm (revolutions per minute) number. The interplay of resist viscosity (controlled by the molecular mass of the polymer and concentration) and the spin speed allows the thickness to be very precisely and reproducibly controlled. Once the spin-coating is complete, the substrate is baked so that the organic solvent is removed and the polymer undergoes glass transition (melts), resulting in a smooth and robust film.

3. Dice the wafer

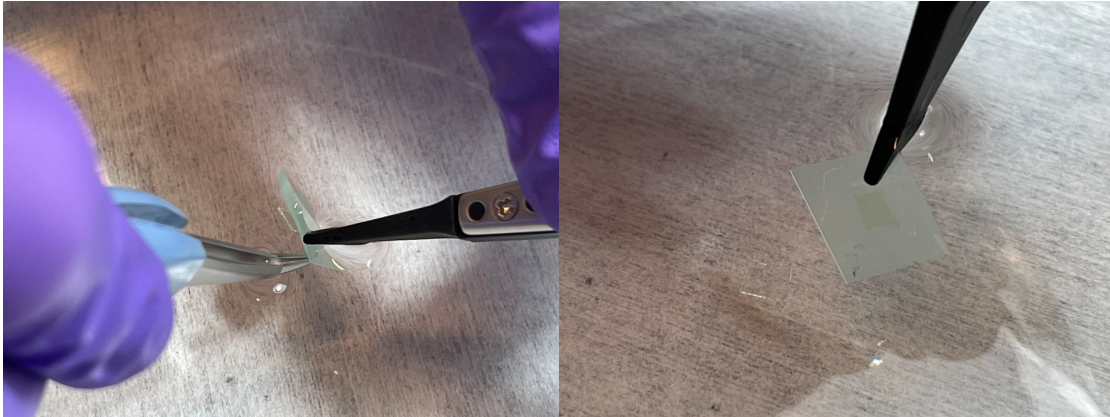


Dice the wafer to small chips using a diamond pen. The chip size should be close to the target substrate, for a more efficient transfer. Scratch the edges of the film by a sharp tweezer, which helps the film to be detached from sapphire substrate by water tension in the subsequent step. Rinse the sample with IPA and dry with N₂ gun.

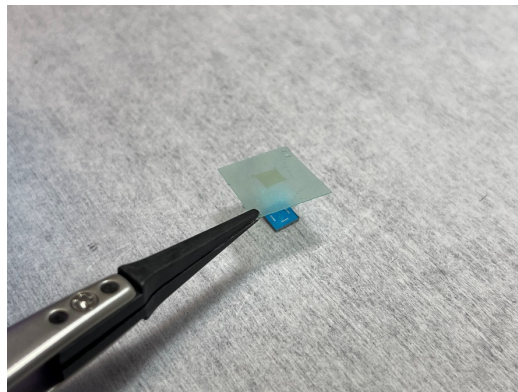
4. Cover the chip with thermo-release tape



Remove the cover of thermo-release tape, and put the tape on top of MoS₂, making a sandwich structure: tape/PMMA/MoS₂/sapphire substrate. For a better adhesion, use a flat tweezer to push out the bubbles.

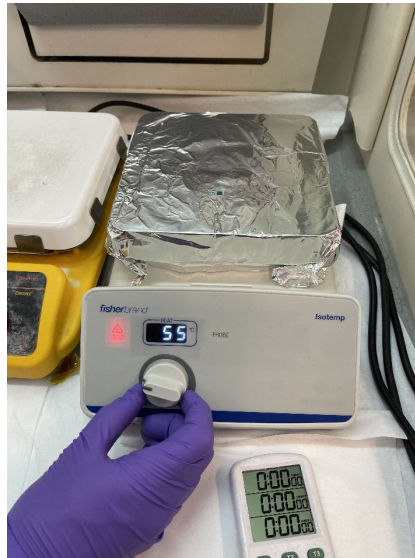
5. Detach the film from sapphire

Leave the laminated sample under water for 5 min, then let water go into the gap between the tape and sapphire substrate. Peel-off the tape gently and detach it from the sapphire substrate. Because MoS₂ has a stronger interaction with the PMMA layer than with the sapphire substrate, it should now be detached from sapphire and remain on the PMMA film with the tape. Take out the sample and dry with N₂ gun.

6. Put the film on the target substrate

Put the peeled-off sample (MoS₂/PMMA/tape) on top of the target substrate. Make sure that the centre of the substrate where the gate stack locates are covered. Then use a flat tweezer to remove the bubbles and strengthen the adhesion. Cut off extra part of the tape by a knife.

7. Heat the transferred sample



Put the sample (tape/PMMA/MoS₂/target substrate) on the hotplate at 55°C for 1h to get a better adhesion. (If the baking time is not enough, bubbles will remain underneath MoS₂, which causes a bad transfer. A minimum baking time of 1h is necessary. If there are still bubbles, increase baking time to 1h15min.) Then increase the baking temperature to 135°C, the thermo-release tape will be removed.

8. Sample cleaning

Put the sample into Acetone overnight for cleaning. Then take it out and rinse with IPA, dry with N₂ gun.

3.2. Double transfer of MoS₂

Here, you will repeat the transfer steps and use as a substrate the chip that already contains one transferred MoS₂ layer. As a result, you will have a second chip with two layers of MoS₂ on top of each other.

3.3. Sample inspection

Using the optical microscope in the LANES group, inspect the surface of the chip and record images of several typical areas around your sample, using both low and high-magnification objectives. Pay special attention to areas in the center, especially around the local gates, since this is where the devices will be realized in the next exercises. Summary of results and observations

4. Summary of experiments and tasks

1. Prepare a sample with a single transferred MoS₂ layer
2. Prepare a second sample with two MoS₂ layers transferred on top of each other
3. Acquire optical images of the produced samples.

5. Questions for the report

In the report, please show the following:

1. Show optical images of the transferred films, for both samples (single and double transfer).
2. Comment on the general appearance of the film, does it contain cracks, does it appear to be clean/dirty?
3. If the appearance of the film is not satisfactory, what could be the reason, how could the process be improved next time?

6. References

- [1] K. Kang, S. Xie, L. Huang, Y. Han, P. Y. Huang, K. F. Mak, C.-J. Kim, D. Muller, and J. Park, *High-Mobility Three-Atom-Thick Semiconducting Films with Wafer-Scale Homogeneity*, *Nature* **520**, 656 (2015).
- [2] H. Kim, D. Ovchinnikov, D. Deiana, D. Unuchek, and A. Kis, *Suppressing Nucleation in Metal–Organic Chemical Vapor Deposition of MoS₂ Monolayers by Alkali Metal Halides*, *Nano Lett.* **17**, 5056 (2017).
- [3] D. Dumcenco, D. Ovchinnikov, K. Marinov, P. Lazić, M. Gibertini, N. Marzari, O. L. Sanchez, Y.-C. Kung, D. Krasnozhon, M.-W. Chen, S. Bertolazzi, P. Gillet, A. Fontcuberta i Morral, A. Radenovic, and A. Kis, *Large-Area Epitaxial Monolayer MoS₂*, *ACS Nano* **9**, 4611 (2015).