Characterization of TMDs and Contact schemes for Photovoltaic Applications

Standard Operating Procedures

ENGR 241 Project
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1. SOP Objective

The standard operating procedures for 1. Optimal TMD exfoliation on SiO_2 substrate, 2. Etching of TMD flakes, 3. Transfer from TMD flakes from SiO_2 substrate to a metallic substrate are explained in details.

All these SOPs are extremely useful for the growing group of SNF members who work on TMDs.

2. SOP Description

2.1. Optimal TMD exfoliation on SiO_2 substrate

The SNF instruction manual for Drytek2 and RTA explains all the procedures on how to use these tools. Hot plate is also very straightforward to use. Here, we will explain how to do the exfoliation using the blue tape or the gel film.

2.1.1. Exfoliation using blue tape (Figure 2)

1. Place the TMD crystal on a blue tape to exfoliate layers of TMD on top of the blue tape.
2. Place the SiO_2 substrate (face down) on top of a blue tape which has the TMD flakes on it.
3. Place a clean blue tape on top of the stack to fix the substrate in place. Your substrate is now fixed between two blue tapes.
4. Flip over the stack and gently press down on the flakes on the front face of the substrate.
5. In case you are doing hot plate anneal, place the stack on top of the hot plate (90°C) and let is stay for 2 minutes.
6. Very gently peel off the front-face blue tape. You will see some flakes being left on the substrate surface.
7. Cap the blue tape, which still has the flakes, with another similar-sized tape for use in the future.

Fig 1. A blue exfoliation tape containing WSe_2 crystals, prepared from a bulk WSe_2 crystal

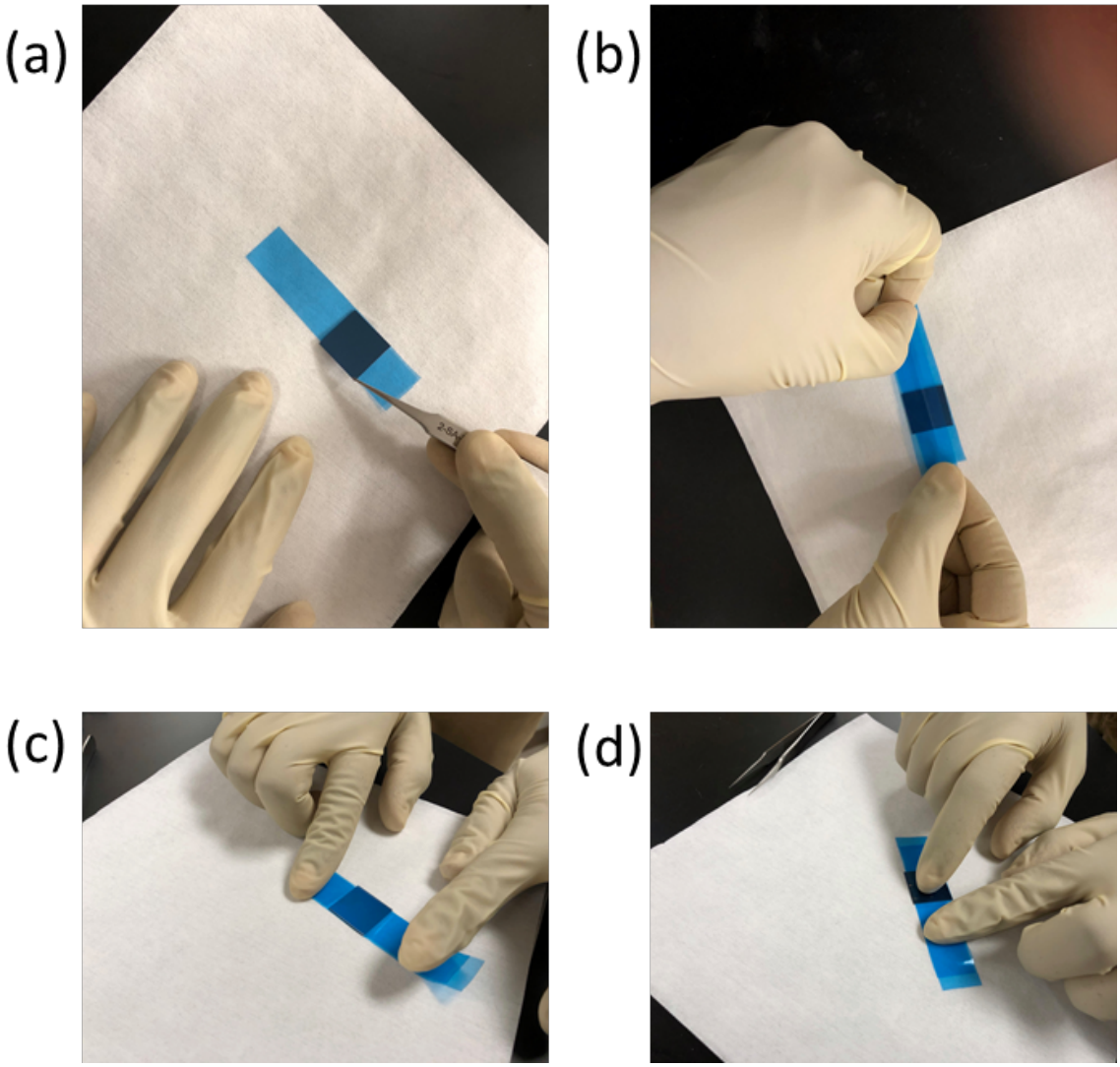

Fig 2. Step-by-step procedure for exfoliating using blue tape (a) Placing the substrate over the flakes on blue tape, (b) Sticking another strip of blue tape on the back side of the substrate, (c) Gently press using fingers around the edges of the substrate, (d) Flip over the substrate and very gently press on the substrate.

2.1.2. Exfoliation using gel film (Figure 3)

1. Peel off the plastic cover from the gel-film.

2. Place the blue tape containing TMD crystals onto the gel-film and gently press.

3. Remove the blue tape from the gel film – some crystals will be transferred onto the gel-film

4. Gently drop the gel-film onto the substrate and gently poke at one corner with tweezers – the gel-film sticks onto the substrate through gravity and adhesion.

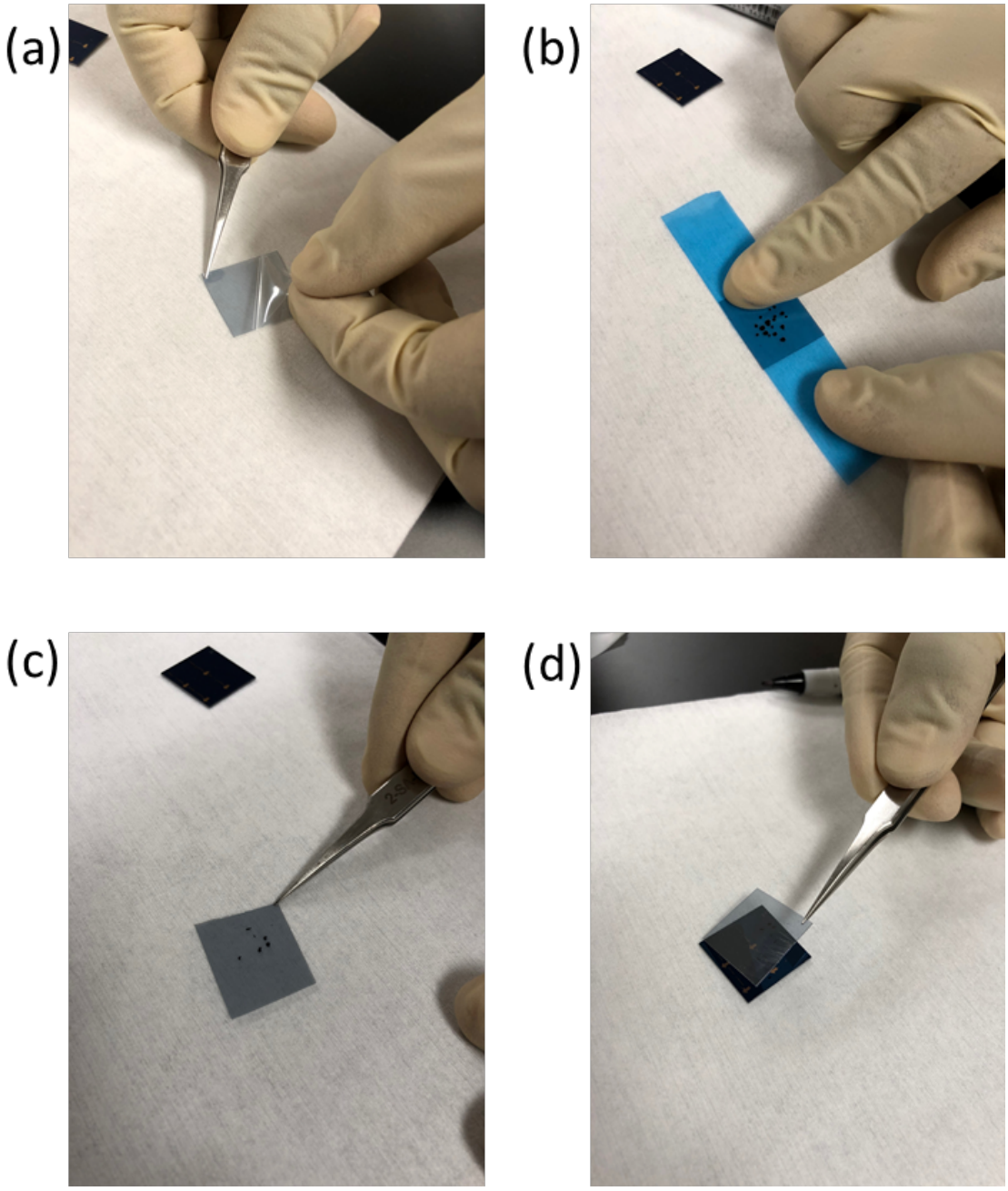

Fig 3. Step-by-step procedure for exfoliating using gel film (a) Peeling off the plastic cover from the gel-film, (b) Placing the blue tape containing TMD flakes onto the gel-film and gently pressing, (c) Picking up the gel-film using tweezers after removing the blue tape (d) Gently dropping the gel-film onto the acceptor substrate and gently poking at one corner with tweezers – the gel-film sticks onto the acceptor substrate through gravity and adhesion.

2.2. Etching of TMD flakes

Etching process consist of a lithography (using Heidelberg) to define the etch patterns followed by an etch done at the pt-mtl.

For the lithography we followed the following steps:

- Resist Coating:
  - LOL 2000 (3000 rpm, 60 seconds on Headway)
  - 5 minute bake at 200ºC
  - 3612 (5500 rpm, 30 seconds on Headway, resulting in a 1-um thick resist)
  - 1 minute bake at 90ºC
- Exposure
  - Heidelberg (Dose: 70 J/cm^2, defocus: 0)
- Post-exposure bake
  - 1 minute bake at 115ºC
- Develop
  - 1 minute in MF 26A solution
  - At least 1 minute in deionized water

The SNF instruction manual for pt-mtl explains all the procedures on how to use the pt-mtl. The recipe details can be found on table 6. For more information, please check out Koosha_SF6 recipe at pt-mtl.

Note that we are dealing with pieces rather than 4 inch wafers. In order to use pt-mtl, we need to mount the pieces on top of a carrier wafer using Kapton tape (figure 4) or diffusion pump oil. The use of Kapton tape is to physically and thermally attached the piece to the carrier wafer.

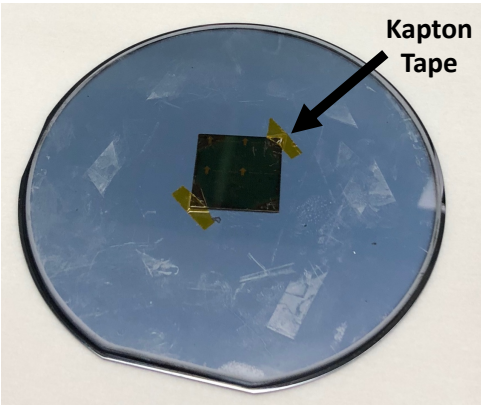

Fig 4. Using a 4-inch carrier wafer in order to etch a piece using pt-mtl

2.3. Transfer from TMD flakes from SiO_2 substrate to a metallic substrate

This part is mainly adopted from the previous work done by Victoria Chen and Connor Bailey on SiO_2 substrate to SiO_2 substrate transfer of TMDs, with modifications tailored to our specific process.

1. loading the sample into the loadlock (LL)

1. a. Refill the load lock (LL) with N_2 (takes 5 seconds) and load samples.

1. b. Close the LL and evacuate, ensuring the decrease of the LL pressure. (takes 5-10 seconds)

1. c. Refill again (without opening) and evacuate again.

1. d. Repeat this process 10x to ensure that as much oxygen is purged as possible. Never open both the evacuation and refill valve at the same time. In other words, cycle through: Open evac. valve→ wait to pump down→ close evac. valve→ open refill valve→ wait for pressure to rise→ close refill valve→ open evac. valve. (Repeat).

Fig 5. Glovebox components mentioned in the instructions. Numbers corresponds to steps in the SOP

2. With the LL refilled and both valves closed, open the LL from the inside of the glovebox and take samples in. Close the LL and keep it evacuated.

3. Mount the glass slide/PDMS/PPC material onto the micromanipulator with the desired material facing down (towards the stage).

4. Place the first substrate onto the stage under the microscope.

5. Turn on the microscope light. The view will be displayed on the monitor on the table to the right of the glovebox.

6. Adjust the stage and micromanipulator in the x-y plane until the material is over the target substrate in the desired place – target flake.

7. Carefully lower the micromanipulator arm until the stamp is in contact with the target substrate.

8. Turn on heat if desired. Heater controls are located underneath the glovebox, and will heat the microscope stage if turned on. A typical value may be 55°C, and will depend on your target substrate and PPC.

9. After a few seconds, pull up the slide/PDMS/PPC stack. The target flake is expected to be attached to this stack.

10. Place the second substrate onto the stage under the microscope.

11. Adjust the stage and micromanipulator in the x-y plane until the material is over the target substrate in the desired place.

12. Carefully lower the micromanipulator arm until the stamp is in contact with the target substrate.

13. Turn on heat if desired. Heater controls are located underneath the glovebox, and will heat the microscope stage if turned on. A typical value may be 90°C, and will depend on your target substrate and PPC. A typical time to heat is 5 minutes, but will vary on your conditions.

14. After heating, the PPC should easily release and the stamp can be raised from the substrate, leaving behind the desired material onto the target substrate.

15. Unload the sample though the LL, making sure that the inside of the glovebox is never exposed to ambient. Always leave the LL evacuated.

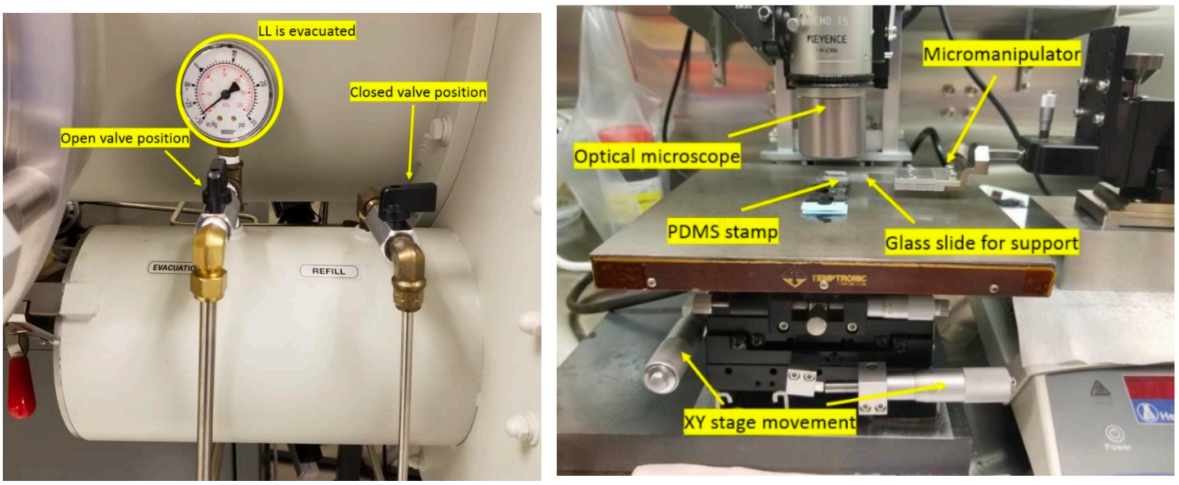

Fig 6. Various components of the glovebox

Additional notes:

- Retracting your arms from the gloves while the stamp is down will cause a pressure differential which will lead to vibrations in the glovebox that can hurt the transfer.
- Keep an eye on the oxygen and trace moisture levels in the glovebox. They are typically measured in ppm but at very high levels will be measured in %.
- The glovebox can be purged by opening the N_2 valve located on top.
- If they remain at high levels for too long, samples inside the glovebox may degrade and a regeneration may be necessary. Regeneration refers to restoring the power of oxygen- and moisture-absorbing materials used in the purifier columns of the gas management system.
- Solvents must be loaded in the solvent transfer container (with a pinhole on the lid).
- If solvents are used in the glovebox, make sure to close the inlet and outlet valves in the back of the glovebox to prevent catalyst damage.
- Always clean up when you are done using the glovebox, and do not heat materials on the stage that could melt.