Li Lab SOP - Amal Bumbia

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Material Exfoliation and Transfer

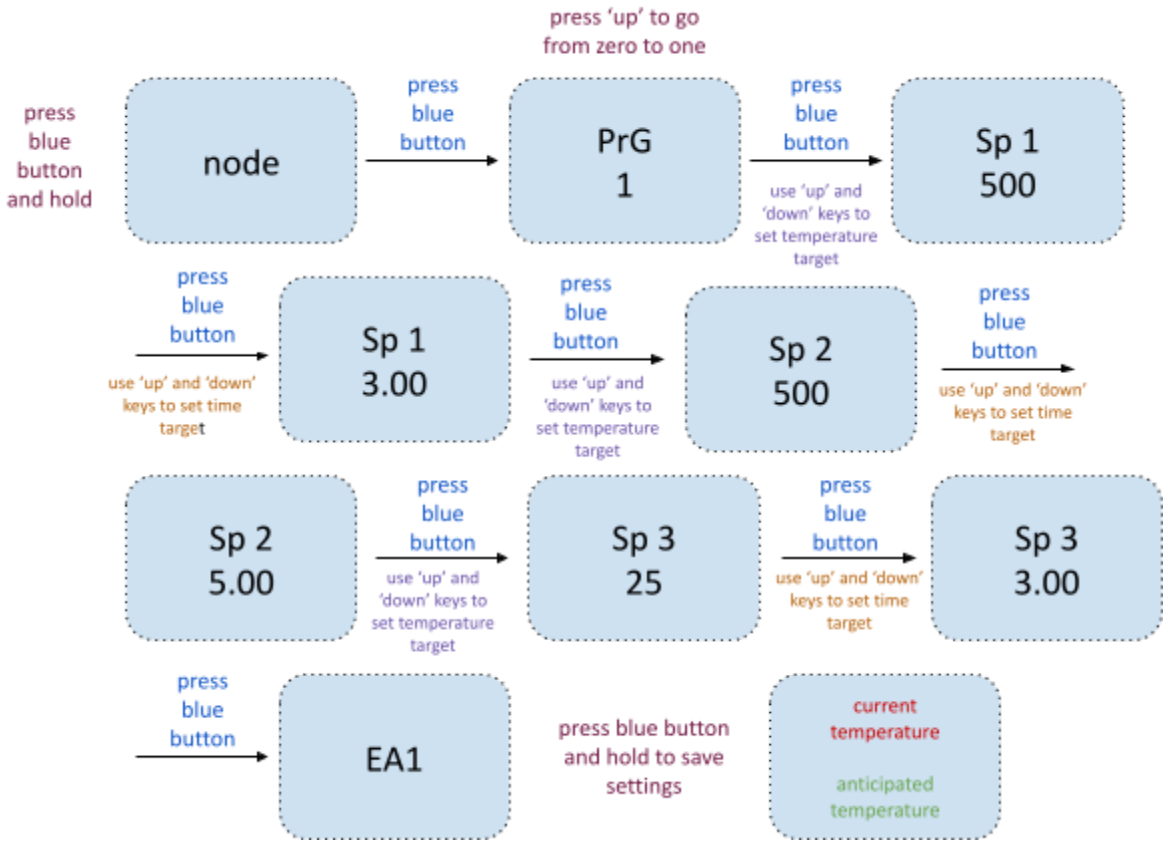
Silicon Substrate Preparation

- [ ] Place large kimwipe on surface, wear gloves, obtain splicing tool, scoring pen, and ruler.
- [ ] Handle silicon with tweezers only.
- [ ] Remove a silicon wafer from the box and cut into quarters if not already cut via splicing tool.
- [ ] Take one or two quarters to use, keep the rest in the box and put them away.
- [ ] Place the chosen silicon shiny side down on the kimwipe. Use ruler and scoring pen to mark a grid on the opaque side such that the squares are about a centimeter by centimer in area.
- [ ] Pick up silicon by the edge and use splicing tool to cut the individual squares. Ensure everything remains on the kimwipe and that you do not touch the silicon even with gloves on.
- [ ] Place each individual silicon piece into a petri dish ensuring the shiny side is facing up. **DO NOT TOUCH OR SCORE THE SHINY SIDE.**
- [ ] Take this dish to the fume hood. In the containers labeled for SI cleaning acetone and IPA use, place half a centimeter deep amount of acetone and IPA respectively.
- [ ] Place another large kimwipe near these dishes.
- [ ] Using the tweezers, pick up each silicon piece by the edges and place them in the acetone first.
- [ ] Take a silicon piece out of the acetone. As you do this, hold it shiny side up against the kimwipe and dry it with a pulse or two of the nitrogen gun. I use this method to ensure my tweezers do not touch the surface of the silicon and so that the nitrogen does not blow the silicon away.
- [ ] Now place the silicon in the IPA. Take it out of the IPA back onto the kimwipe, shiny side up, and dry it with the nitrogen gun again.
- [ ] Place the silicon back in the petri dish shiny side up.
- [ ] Repeat the previous three steps for each silicon piece individually. Throughout the process, ensure the silicon remains shiny side up as we NEVER want it to be face down on any surface if we can avoid it.
- [ ] Check some of the cleaned silicon under the microscope to ensure there's no large blemishes or amounts of residue. In that case, recleaning is necessary. Silicon must also be recleaned if it has not been used for exfoliation within a few weeks.

Annealing

The annealing machine is located in Lai Lab on the 3rd floor. Before transferring HBN or TMD, you need to anneal the samples. It is not necessary to anneal graphene samples. Depending on the material, you need to apply different settings to the machine before running it. First, place the samples in a boat, preferably one with a handle. Unscrew the side of the tube from the holder and place the boat inside the tube. Use the wire to push it into the center. Screw the tube to the base and close the lid of the chamber. Alter and save the settings. Turn the vacuum pump on and let the pressure go down to the order of 1E-5. Start the machine by pressing the 'down' key and holding it until the red light on the front of the console turns on. When unloading samples, the chamber can be opened at around 70 degrees and things can be taken out around 50 degrees. Vent the vacuum pump via a knob on the back and then unscrew the tube base. Take the boat with the samples out using the wire.

HBN Settings



Graphene Exfoliation

I find that the more haphazard the exfoliation process is, the better samples I get. Being too careful is honestly detrimental and results in samples that are too small or have too much residue or are not properly isolated from the bulk or have layers of varying thicknesses.

- [ ] Take a piece of scotch tape and fold the ends inward a little so that you can easily hold the tape.
- [ ] Using tweezers, place some bulk graphite on various regions of the tape and ensure they are flattened.
- [ ] Holding the ends of the tape, start folding in various orientations so that the bulk spreads across the tape to where there is a non-uniform dispersion of pieces densely populating the tape. Ensure that you are not pressing the tape. This is the mother tape.
- [ ] Take a second piece of tape and fold the ends similarly. Place it over the mother tape so that the tapes are touching and stuck together, but do not press them together. Slowly peel the second tape away from the mother tape.
- [ ] Repeat the above process with a third tape against the second tape.
- [ ] Take a silicon substrate and place it shiny side up on a slide preferably lined with thin double sided black tape so that the substrate does not move. Place the third tape over the shiny side of the substrate such that a diverse region of graphite pieces are aligned with the silicon.

- [ ] Get a cotton stick and swipe across to press the tape into the substrate using even strokes in the same direction. Press with marginal pressure.
- [ ] Peel the tape back from the substrate slowly with an even velocity, ideally perpendicular to the direction of swiping. As you peel, keep the tape perpendicular to the surface as well.
- [ ] Check for viable samples under the microscope.

HBN Exfoliation

UV Machine - Before exfoliating HBN, it is necessary to clean the already cleaned silicon substrates you will use with a UV lamp. The UV machine is in the 13th floor lab space. Place your substrates on the plate of the machine, ensuring it is off as you do so. Do not place anything but the substrates on the plate. Close the lid and turn the machine on. Set the timer to 15 minutes at 100 degrees C and start the machine. Once complete, turn the machine off and wait 15-20 minutes. Then take out your silicon pieces.

- [ ] Take a single granule of HBN and place it on a piece of opaque blue tape with the ends folded inward. Fold over the HBN until it is dispersed across the tape.
- [ ] Take a translucent blue thermal release tape (TRT) and fold the ends inward. Place it over the opaque tape, press very lightly if needed.
- [ ] Slowly peel back the TRT. Take one or more silicon substrates and place them shiny side up on a surface. Place the TRT over the substrate(s) and press with a cotton tip just enough so that the tape is attached to the substrates. Cut off any excess tape.
- [ ] Heat the hot plate to around 80 celsius and place the substrate-tape configuration on it such that the tape is at the top. Remove the configuration once the tape gradually begins to shrink and peel back from the substrates. To test whether the hot plate is too hot or not hot enough, test it with the excess tape.
- [ ] Manually peel the tape away from the substrates. Check samples under the microscope.

TMD Exfoliation

UV Machine - Like before, 100 degrees C for 15 minutes, let the substrates rest for 15-20 minutes while the machine is off before taking them out. These silicon pieces should look very shiny.

- [ ] The TMDs are in the vacuum chamber. Check with someone to see which containers or tapes you are allowed to use for exfoliation. There may be some TMDs preserved between opaque blue tape layers — these are mother tapes that you can use directly.
- [ ] Close and depressurize the vacuum chamber once relevant TMD is removed to be used for exfoliation.
- [ ] Obtain opaque blue tape and a PDMS box. Wear gloves.
- [ ] If you procured a mother tape from the vacuum chamber to use for exfoliation, ensure that the area of the tape covered by the TMD is roughly the same area as the silicon substrate you will use. You do not want to use silicon substrates that are too small.
- [ ] Take a mother tape and cut off the excess tape where there is no TMD so ensure no extra tape residue gets onto the PDMS box. Leave enough excess tape to hold onto when exfoliating onto the PDMS box.
- [ ] Place the TMD onto the PDMS holding it via the excess tape. Use a cotton tip to press down if needed. Quickly peel the tape back. You want to see visible TMD pieces on the PDMS. There is an acceptable threshold of density.

- [ ] Repeat this process of exfoliating the TMD onto the PDMS box until you exhaust the tape. Try not to go over the same area of PDMS multiple times.
- [ ] Have UV cleaned substrates prepared or prepare them shortly after since you want to transfer the TMD on the PDMS to silicon the same day.
- [ ] Place a silicon piece shiny side down onto the PDMS where there is a good density of TMD flakes. Press down gently so that it picks up the TMD.
- [ ] Check under microscope for usable pieces. Transfer the same day.

PDMS Slide - Take microscope slide and clean it with IPA. Use blade to cut out PDMS piece with the circle in the center. Use tweezers to place it face up on the slide. Take a piece of scotch tape and use it to press down on the PDMS so it sticks to the slide. Scotch tape can also be used to clean the PDMS. You can trim excess PDMS with a blade once it is on the slide. Keep the circle intact.

PPC Slide

- [ ] Take a PDMS slide and PPC from the fridge.
- [ ] Use the wooden end of a cotton tip to pick up a drop of PPC and put it on the center of the PDMS. Place that into the spin-coater such that the square is directly where the vacuum suction is.
- [ ] Take the slide out of the spin-coater after its cycle is complete and put it on a hot-plate for 2 minutes at 150 degrees celsius.
- [ ] You can clean the slide with scotch tape to remove the PPC when/if needed.

Spin-Coater Settings - Turn on vacuum using button after settings are in place, vacuum light should be green. Close lid of spin-coater after slide is in and press I/O to run it. Also press I/O to save each setting as you change it. While running, I/O will be lit green. That light will turn off when the spin-coater has stopped.

PC Slide

- [ ] Take a prepared PDMS slide and about 3-5 regular microscope slides. Two of these slides should have a piece of scotch tape on one side so that it is easier to distinguish which side of the slide is facing up at any given point. The other slides will be used to make tape windows — you can just have one slide designated for this, but I like using three for efficiency.
- [ ] The initial two slides are marked with tape because they will be where we put the PC solution. First, clean the face-up side of these slides with IPA. Let the IPA evaporate. Then, put a few drops of PC solution on one of the slides and flip the second slide over so that the cleaned side covers the PC solution. Swipe that

second slide to spread the PC solution a little bit. Place both slides down so that the PC solution is facing up and let it evaporate a little.

- ☐ For your tape window slides, place 2-3 pieces of scotch tape perpendicularly to the length of the slide on each slide. You want there to be enough tape going outside the slide to where you can peel it off and hold the tapes on either end, but not too much excess. Take a blade and cut out a square from the centers of the tapes on the slides that is large enough to cover the PDMS square (this is why we trim the excess PDMS).
- ☐ When your PC has rested for a moment on the other two slides you prepared, examine the PC to make sure there's a large enough section that is not reflecting rainbows or has too many blemishes on it. You want region with an even layer that is not too thin. Use the blade and a small piece of scotch tape to cut out any larger excess PC on the slides so that they do not get onto the final tape window.
- ☐ Take a tape window and lay it on the PC region you want to pick up. Pick it up and check the window to make sure that the PC covering it looks good. Lay that over the PDMS slide, obviously so that the sticky side of the tape window sticks onto the slide.
- ☐ Place this slide face up onto a hot plate at 100 degrees celsius for at least 20 minutes.

Making PC Solution - Obtain a small, dark glass bottle with a lid. Also obtain a digital scale and small funnel. Place the bottle and funnel onto the scale and tare it. Take the container of hard PC pieces and gently pour them into the bottle via the funnel. **This should be 1 GRAM worth of PC pieces.** Pour a small amount of chloroform into the designated beaker under the fume hood. Pour that through the funnel and into the bottle quickly if that setup is outside of the fume hood. **This should be 10 GRAMS of chloroform.** Take care to not pour in too much chloroform by monitoring the scale. You can add more as needed. Once complete, screw the lid of the bottle on. Label the bottle with your name, the type of solution you made, and the date. Leave it in the containers under the fume hood. It is usable once the PC pieces have dissolved properly into the chloroform. This can take a few days. Make sure to store the bottle vertically.

Transfer

PC Transfer

*If transferring with PC, we do NOT anneal the samples prior.

Initial Pickup

- ☐ Place the substrate on the stage.
- ☐ Turn on the illuminator and locate the sample on the computer, focusing with the attached microscope. Use the knobs on the base of the station to move the stage that the substrate is on.
- ☐ Once the sample is located, turn the vacuum on and twist the lever connected to the vacuum pump if needed. Make sure the slide holder has a vacuum suction.
- ☐ Place the PC slide in the slide holder so that the PC is facing down towards the substrate.
- ☐ Use the higher knobs to move the slide so that the PDMS circle is over the sample. You should be able to see the shadow of the circle on the computer screen.
- ☐ Focus the screen to where you can see both the circle and the sample you want to pick up. Move the slide so that the sample is off-center of the circle.
- ☐ Use the top knob to lower the slide closer to the sample and adjust as needed. Lower the slide until it is almost touching the substrate, but not quite.

- [ ] Turn the power supply on and provide about 4-5 volts of power. Turn the voltmeter on so that you can monitor the temperature.
- [ ] At about 80-90 degrees, lower the slide down to the sample so that the center of the circle touches the substrate near the sample. The color on the screen will change at this point.
- [ ] Let the temperature increase to 110-130 degrees as the PC melds towards the sample via the heat and covers it. At this point, you can pick up the sample by raising the slide up. Some people do it slowly and others do it quickly.
- [ ] Check to make sure the sample is no longer on the substrate. Take the slide out.
- [ ] Turn everything off and check the slide under the microscope to see the sample quality.

Transfer to New Substrate

- [ ]

PPC Transfer

- [ ] This is the same as the PC transfer except your samples need to be annealed and you want to lower the slide around 30 degrees and pick up at 40-43 degrees. Use 1-2.5 volts.

PC Post-Transfer Cleaning - Hold sample in chloroform for 2 minutes. Then hold in acetone for 2 minutes. Then hold the IPA for 2 minutes. Place face up on kimwipe and dry with a nitrogen gun. Repeat if the PC is still on the substrate.

Glovebox Transfer

Cleanroom Apparatuses

Mask Aligner + Spincoater

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PVD75 e-beam