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AFM SOP (Contact mode):

Warnings:

- Before moving AFM head, the laser beam must be off
- AFM head is delicate, do not damage when moving, make sure it is properly grounded

Start:

1. Review Warnings
2. Turn on Computer
3. Turn on Park XE AFM controller
4. Check software:
    a. Image pixel size (less pixels means faster scanning, lower resolution image)
    b. Scan rate (too high can make strange shapes in topographical image)
    c. Z Servo Gains, P Gain, I Gain = 1
    d. Check which axis the tip moves along, X or Y axis
    e. Adjust setpoint if needed
    f. Ensure in correct imaging mode (contact/non contact/tapping)
5. Mount your sample (on a slide then on the sample stage). Use the clip

AFM tip/cantilever

6. Use silver paste to mount AFM tip onto tip holder.
7. put the tip holder into Z stage/AFM head gently until it clicks into place (magnet?). Align the pins to the cantilevers slots (don't let anything touch the tip)
8. Slide AFM head into instrument until it can't be moved anymore
9. Lock by moving the black paddles out.
10. Plug in Laser cable (left side of head)
11. Turn on head on AFM head/Z stage and on the computer (top right)

Engaging Substrate

12. Turn on light (top of AFM) (to see tip and substrate on the TV)
13. Focus camera on the cantilever
14. Move the photodiode beam using dials on top of Z stage until A+B~=2.5. Needs to be on end of cantilever.
15. Adjust the two knobs on side so that:
    a. $|A-B| < 0.1$
    b. $|C-D| < 0.1$
16. Lower the tip to sample
17. Focus the lens on the substrate. Adjust position of substrate until tip is above the sample. May need to do this more when you lower closed
18. Raise the focus to half
19. Then approach until you see substrate again
20. Do this until you are 200-400nm away
21. Adjust position of sample again

22. If needed, adjust A-B and C-D values again
23. Ensure that Contact mode is on
24. Press SCAN ON and adjust the scanning area size as desired. Adjust the setpoint if needed.
25. SCAN OFF
26. Close and lock the doors
27. Press “Approach” (Z Stage section of screen).
28. After the tip finishes approaching the sample, press SCAN ON and Start (under Image)
29. As the topographical image and graph forms, determine what part of the sample the tip is over, and adjust the location of the scanning area accordingly (to make sure the AFM scans the correct area)

Lifting to another part of sample:

30. Define distance tip should be lifted then press Lift. lift in intervals of 100nm
31. Use dials on sample stage to move tip to another part of sample.
32. Close door again and approach

Finishing:

33. SCAN OFF/ Image off
34. Press Lift Z.
35. Open the doors
36. Remove sample.
37. Turn off photodiode.
38. Make sure all LEDs are off, then disconnect laser cable.
39. Move the paddles in
40. Slide z stage off to left (avoid objective)
41. Place z stage in the stand.

Other Considerations:

- Do not use too hard setpoint force for a hard tip, may damage tip and/or sample
- Harder setpoint force for soft tip can help remove residue from the sample, because the tip will push residue to the sides.
- Cleaning AFM tip reduces residue on sample
- If there is a nonzero slope detected on sample, pressing AUTO will flatten the topographical graph
- AUTO shows proper scale for graph profile
- Higher error signal occurs on boundary of sample

Exfoliation SOPs:

Graphite:

1. Gather: Tweezers, gloves, scotch tape, graphite, silicon substrate, sticky slide
2. Prepare Mother tape: take out tape and fold at the ends.

3. Use tweezers to place a cluster flakes of graphene on mother tape.
4. Spread the flakes out and flatten them. Make sure to leave no empty spaces in your cluster
5. Fold mother tape in half (can press only with mother tape). Unstick it, and continue folding and unsticking to spread the graphite around. Don't do it too much, still want there to be big flakes left
6. Now, prepare daughter tape.
7. Put mother and daughter tape together and then pull apart. KEEP MOTHER TAPE
8. Prepare granddaughter tape.
9. Put daughter and granddaughter tape together and then pull apart. KEEP DAUGHTER TAPE
10. Put silicon on sticky slide with tweezers. Make sure it is actually sticking!!!
11. Find an area of graphite on granddaughter tape that doesn't have too thick flakes, but also isn't too empty. Place that area of tape on the silicon, keeping the tape parallel to the silicon.
12. Press HARD with Q tip and slide in one direction. Hard enough to make trails and to leave an outline of the silicon on the tape.
13. SLOWLY peel off tape, keep tape perpendicular to silicon. KEEP GRANDDAUGHTER TAPE
14. Now, look at your substrate through microscope for any samples.
15. Save pictures of any samples, and record where they are on which substrate to keep track of them.
16. If you get usable samples, reuse the mother tape. When finished with the tapes, you can store them for reference (mostly useful for mother tape, dont need to store all the descendants if not needed)

hBN:

1. Obtain gloves, tweezers, thermal release tape, hotplate, and hBN flakes in a mother tape (blue tape).
2. Peel apart mother tapes.
3. On each mother tape, attach thermal expansion tape (do not press) and peel off.
4. To thin out: put more thermal expansion tape on the thermal expansion tape
5. Once thin enough, cut out irrelevant parts of tape and leave the relevant part and an area to hold.
6. place tape on silicon.
7. Turn on hot plate and test heat by putting pieces of thermal expansion tape on it. Want the tape to start wrinkling when on hot plate
8. When you find the correct temperature, place the silicon with tape and then use two tweezers to pull on the thermal expansion tape when it wrinkles
9. Take silicon off hot plate and turn off hot plate

Make mother tape:

1. Take blue tape and fold on both sides
2. Use tweezers to take out a flake of hBN and put on mother tape
3. Make another blue tape and put it on the first.
4. Peel on and off (no pressing) to spread it around and thin it out
5. Done

TMDs:

1. Prepare mother tape: take flat and thin bulk and put on sticky blue tape. Put another sticky blue tape on it
2. Open mother tape
3. Use more blue tape to thin it out (if needed). Can also use tweezers to pick up flakes to help thin out

4. Once thin enough after however many tapes, cut tape to a strip with the width of a silicon substrate
5. Can also thin out by pressing mother tapes together in a way that they don't overlap and then take apart again. Try to make a region of TMD that is the size of your silicate.
6. Put tape on pdms box (press with Q tip experiment with force). Then take tape off quickly.
7. Repeat on other parts of box (if too little coming off try another tape)
8. Then put silicon face down on a good section of TMD and press with tweezer so that there are no bubbles (can look at bottom of box for bubbles)
9. After some time (?) take off silicon and check

Considerations:

- Thickness changes in a sample causes bubbles to try to keep thickness uniform when thinning out your flakes
- Can clean pdms box of flakes by using tape. Can you reuse?? Perhaps you can reuse for sample storage but not for more exfoliation bc of the tape residue

Transfer SOPs:

PPC:

1. Turn on vacuum pump. Ensure the yellow knob points along the tube coming out of the pump.
2. Place sample on stage. Turn on light
3. Locate sample using bottom two knobs. Focus using microscope knobs at top and the pin. If needed, rotate sample using the knob at the back.
4. Add your pcc slide. Focus on your pcc slide to either locate the sample on the pcc slide or just the bubble. Position the bubble such that the sample to be picked up is within the bubble (not in the middle) or that the two samples are on top of each other in the correct orientation (when doing monobilayer make sure edges are at the angle you want, edge prob corresponds to lattice basis vector.
5. Turn on power supply to about 2 V, increase if you need temperature increase faster. Turn on thermometer
6. Keep power supply on to increase temperature to 30*C.
7. Meanwhile, slowly lower pcc slide onto sample using top vertical knob. Stop once ripples form/ there is a bubble of different color forming. Have the sample covered when the temperature increases to 40-43
8. Pick up quickly by Anything the bubble picks up is green.
9. Separate the pcc slide and the substrate with upper vertical knob.
10. Remove pcc slide.
11. Focus on substrate to see that your sample is gone.

PC:

Procedure is the same as PPC, except heat your pc slide up to 100*C at 4-5 V. Start heating up while finding sample, because it takes a while to heat up. Touch down at like 80*C and cover your sample when temperature is 100*C-120*C. When sample covered, increase hight of slide so that the dot is right over the end of sample, then reduce the temp

General considerations for building device:

- When putting down layers, heat ppc to 130*C. Leads to residue but hopefully we can clean with afm

- Clean your device with chloroform 10 min acetone 2 min IPA 2 min

Slide Making SOPs:

PC:

1. Put on gloves. Obtain 4 clean slides from the red box with pdms slides (clean with scotch tape), bottle of pc, knife, scotch tape, Q tip.
2. Hold two clean slides together at an angle
3. Use wrong side of Q tip to obtain drop of pc, then put on the end of one slide, then slide the slides together to thin it out. Do this quickly, pc dries quickly
4. Check the thickness. If too thick or thin, peel off the pc with scotch tape. If ok thickness, put down the pc coated slides after that.
5. Then, with some new clean slides, put folded scotch tape on them, and cut a window with your knife. Window should be a little bigger than the pdms squares. Do this on each side of each slide.
6. Inspect your pc coated slides, and make cuts with knife according to how big the pdms on each completed slide should be.
7. Take off your scotch tape with window and put on pc section of coated slide. Fold the tape over if needed to not pick up adjacent pc section.
8. Press around the window with Q tip so it sticks. Then peel off tape with now attached pc in the window.
9. Put the fully completed window on a pdms slide.
10. Heat for 30 minutes at 100*Cs
11. Do this process for every window and every pc thing.

Some considerations:

- pc that is too thin has rainbow pattern
- Too thin pc increases risk of pc wrinkling
- Too thick pdms increases risk of pc wrinkling
- As you use a pc slide for transfer, it wrinkles more

PPC:

1. Obtain pdms slide (clean with scotch tape), newish ppc from fridge, Q tip
2. Use wrong end of Q tip to place a drop of ppc in middle of your pdms slide
3. Once correct

Annealing:

Starting:

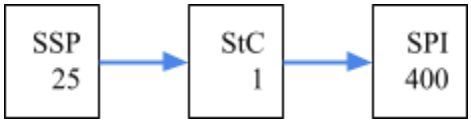
1. Open annealing chamber (make sure at a good temperature before opening (?))
2. Unscrew the base from the annealing tube.
3. Get wire with hook on end and put through tube (not supposed to go beyond a certain point?) to latch on to any boats still in annealing chamber and pull them out.
4. Take the convenient boat (has hole at end) and load it with your samples.

5. Put loaded boat in tube and push it to middle of annealing chamber with the wire
6. Take wire out, screw the base back on, and close and lock annealing chamber

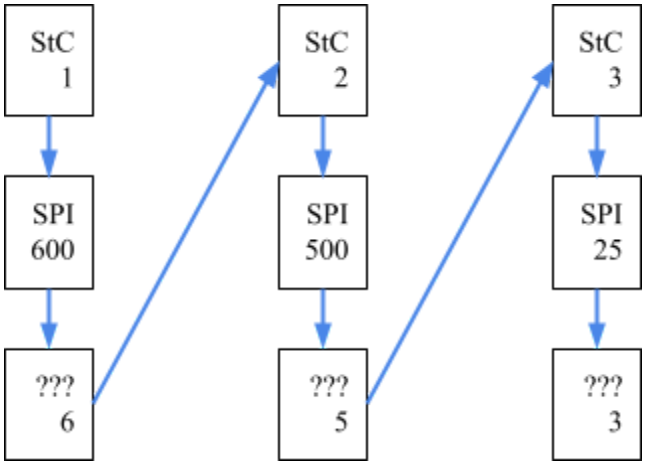
Settings:

- The red number on top is the current temperature, the green number at the bottom is the anticipated temperature.

7. Press blue button until nodE / nES.
8. Then press again and it should say PrG. press up arrow so it says 1
9. Press blue until the following (blue arrows are presses of blue button):



10. Now use up arrow to change the 400 to 500 (temperature for hBN). The following diagram explains to how to set up the full settings (the last row is where you set the time settings):



11. Continuing to press the blue button will just cycle you through the settings again. There's a section 4 but ignore it (?).
12. Hold down the blue button to save your settings (goes back to the temp/goal temp screen)

Vacuum Pump:

13. Make sure the screw thingy at the bottom is loose as per the sign on the vacuum pump
14. Turn on 
15. Use < and > arrows to switch between parameters
16. Good parameters: 1.6E -4 hPa and 1000 Hz - two pumps the second one sound different (quieter)
17. ???
18. Don't know how to adjust

Back to annealing chamber:

19. Hold the down arrow button to run the annealing. Makes a clicking sound
20. Come back after like 14 hours, can only take samples out when temp is <=50 (?)

Taking sample out: vent vacuum pump:

1. Turn it off
2. Once 100ish Hz, turn small black knob at the back. The pressure reading will increase and there will be a hissing sound

3. Stop turning for few seconds then start again (do this multiple times)
4. Do this until no more hissing

Finishing:

5. Unscrew the base and use the wire to take your boat out
6. Unload the boat and take your samples
7. Screw the base back on

UV Cleaning: Need to UV clean silicon before exfoliating hBN or TMDs

1. Open the chamber. Double check if sample already inside
2. Never put petri dish in UV machine it will melt. Instead take out your silicon and place them facing upwards on the plate
3. Close the chamber
4. On switch at back of machine
5. Set it to 100*C for 15 minutes. Press start
6. After the 15 minutes, let the machine cool down for a while until you open and take the silicon (15 min to 20 min)
7. Turn machine off

Silicon slicing and cleaning:

Slicing:

1. Get the 3323 silicon box and use tweezers to remove a silicon quartercircle. If no quartercircles, you need a quarter.
2. How to cut: Take the pen-knife from the chemical storage cabinet and a protractor/any straight edge. Use the pen and straight edge to draw any lines you want to cut. Too much force and you may break it, too little and it wont cut along that line.
3. After making all the cuts

Silicon gate cutting and cleaning:

Cutting:

1.

Cleaning:

1. Clean silicon in regular way with Acetone and IPA (to get rid of silicon dust/particles) - don't need this step actually
2. sonicate the beaker of leftover acetone for 5 minutes - no
3. Put gates back in acetone and sonicate 5 minutes
4. Put gates back in leftover IPA container and sonicate 5 minutes
5. Then UV clean for 40 minutes - do we have to use it soon after UV cleaning like for regular silicon?

Glovebox: