Fabrication of electronic devices on 2D materials and heterostructures

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518 (CINT)/1026; Teams

https://cint.lanl.gov/news/events1.shtml

CINT Nanofabrication Workshop Series (CNWS)

SAND2023-04357PE

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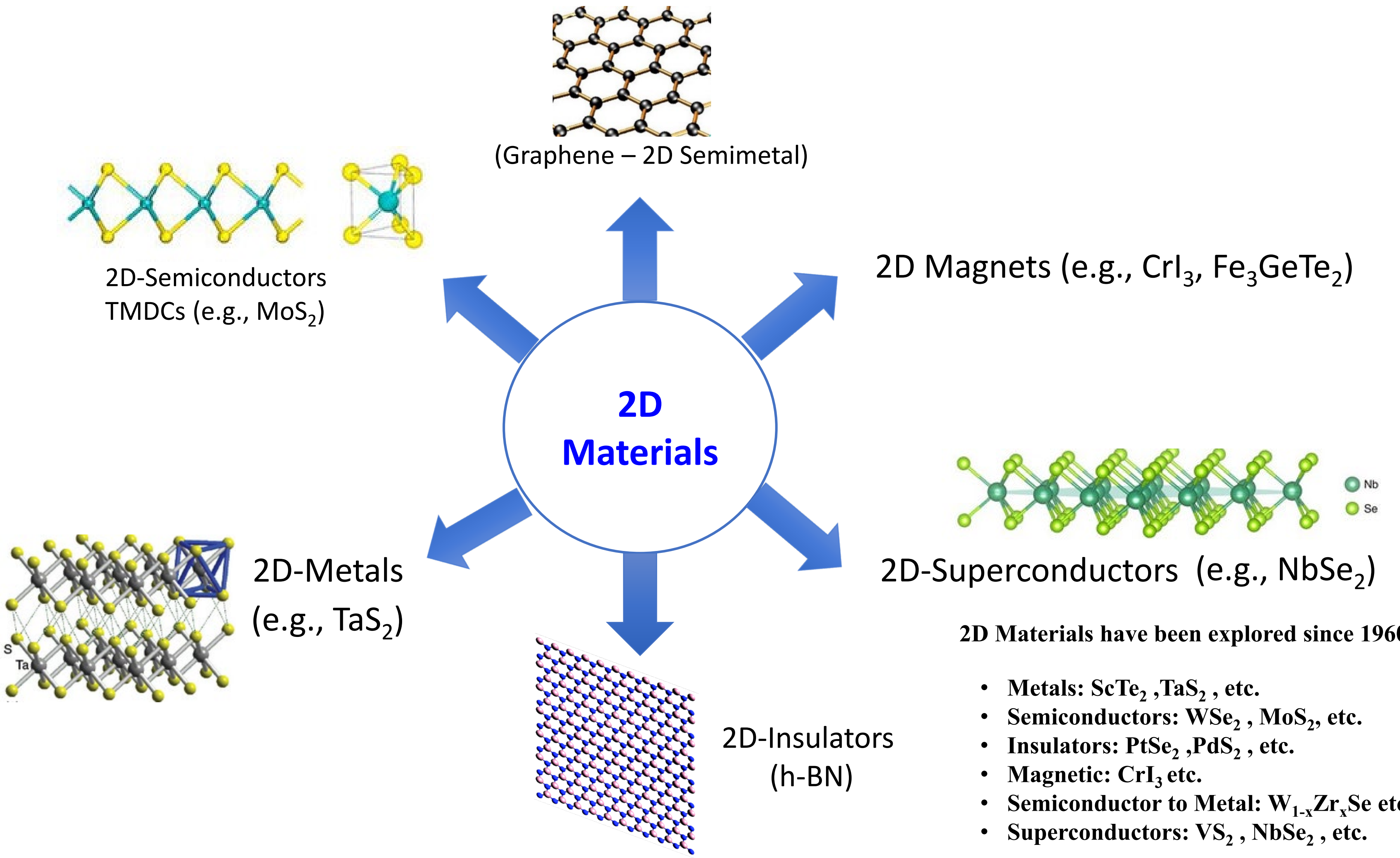
Outline for fabrication of 2D material based electronic devices

- Methods of obtaining 2D materials
- Correct substrate selection, cleaning, and prefabrication preparation
- Locating 2D materials for design
- Efficient contact pattern design for randomly oriented 2D structures
- Global alignment using NPGS for accurate navigation.
- E-beam patterning using NPGS
- Fabricated device and electrical measurements

Fabrication Golden Rules

- Do not start from scratch
  - Literature survey
  - Ask for an established/similar recipe, help is always available in the building
  - Check each fabrication step with a verification step
- Critically think about each fabrication step
  - Always think about all possible outcomes from each fabrication step (good or bad)
  - Keep tool, chemical, and material tolerances in mind while designing a recipe
- Detail everything and organize steps
  - Always maintain detailed lab notes
  - Create Process sheets for your fab process to follow every time
  - Organize cleanroom activity before performing/running any fab
- Treat everything with care and respect
  - Respect the tool, IL staff and fellow users
  - Follow all the rules for the IL and specific tools.
  - Ask for help if you are not sure about a step

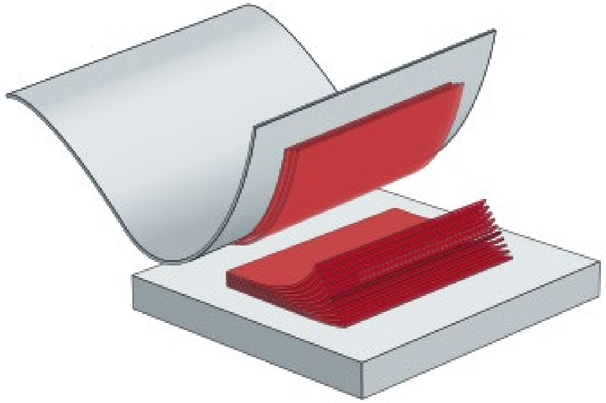
Partly taken from Troy's April presentation



2D Materials have been explored since 1960s

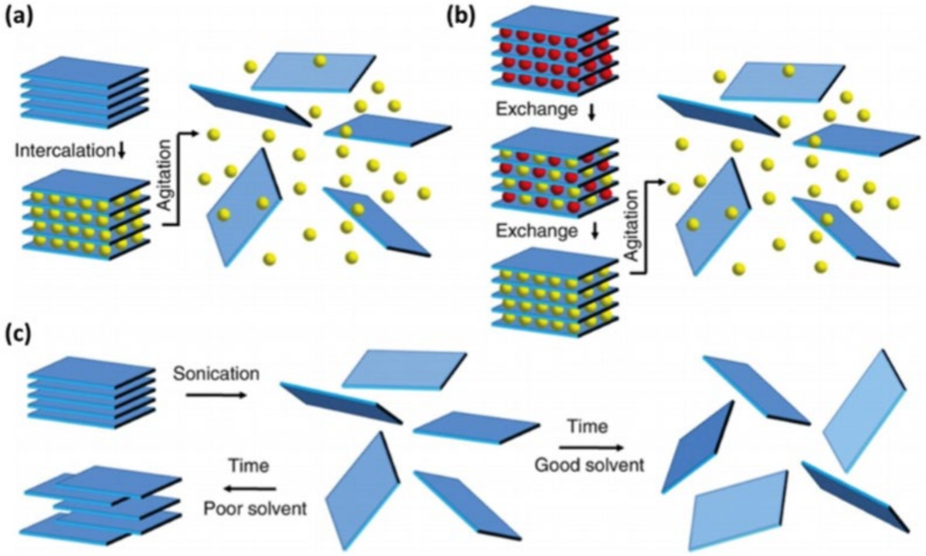
- **Metals: $ScTe_2$,TaS_2 , etc.**
- **Semiconductors: WSe_2 , MoS_2, etc.**
- **Insulators: $PtSe_2$,PdS_2 , etc.**
- **Magnetic: CrI_3 etc.**
- **Semiconductor to Metal: $W_{1-x}Zr_xSe$ etc.**
- **Superconductors: VS_2 , $NbSe_2$, etc.**

Top-Down Approach



- Less chance of adding impurities
- Small scale and time consuming

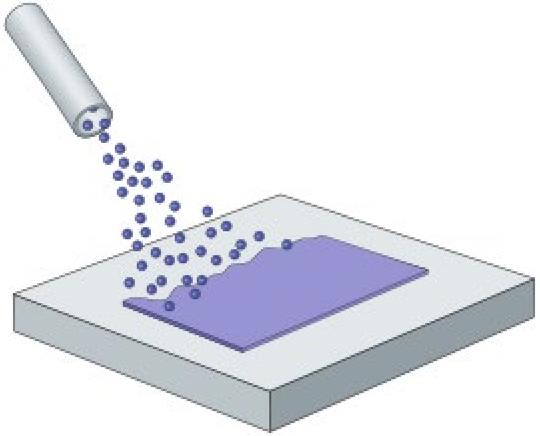
Micro-mechanical exfoliation



- Sample yield
- More impurities from solvents

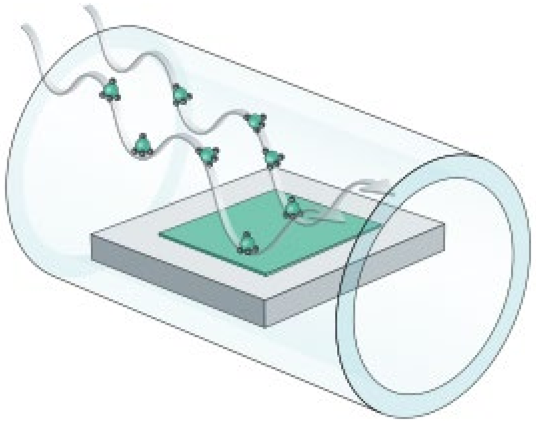
Liquid phase exfoliation

Bottom-up



- Large Area samples
- Expensive

Physical vapor deposition



- Large Area samples
- Grain Boundaries and defects

Chemical vapor deposition

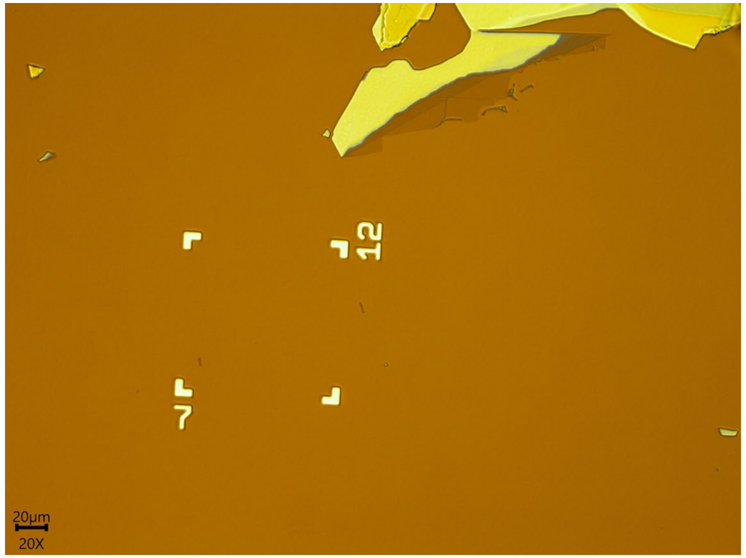
Mannix, Andrew J., et al. "Synthesis and chemistry of elemental 2D materials." *Nature Reviews Chemistry* 1.2 (2017): 0014.

Rana, Sohel, and Raul Fangueiro, eds. *Advanced composite materials for aerospace engineering: processing, properties and applications*. Woodhead Publishing, 2016.

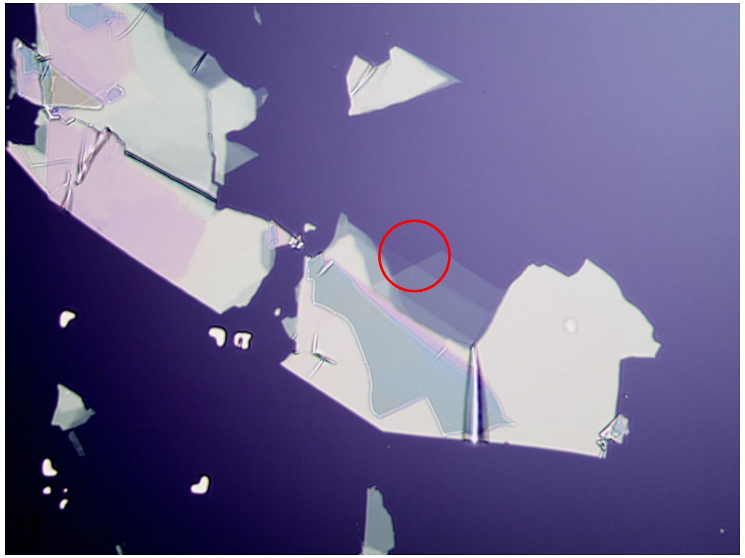
List of Tools typically needed for 2D material fabrication – available at CINT

1. Contact/ contactless Photolithography
    - MLA 150
2. Dicing saw (preferred)
3. O_2 Plasma for substrate cleaning
    - Anatech Barrel Asher (200W)
    - LOLA (2000W)
4. Optical microscope
5. SEM with NPGS capability
    - SEM-1
    - SEM-2
6. Reactive Ion Etching
    - Oxford instruments RIE-1
7. Electron beam metal evaporators
    - EG-1
    - EG-2
    - EG-3

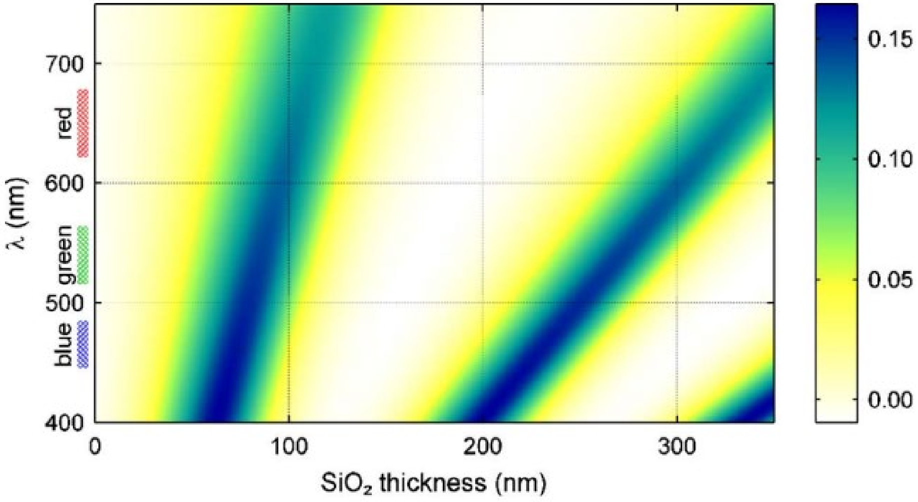
Choosing the correct substrate



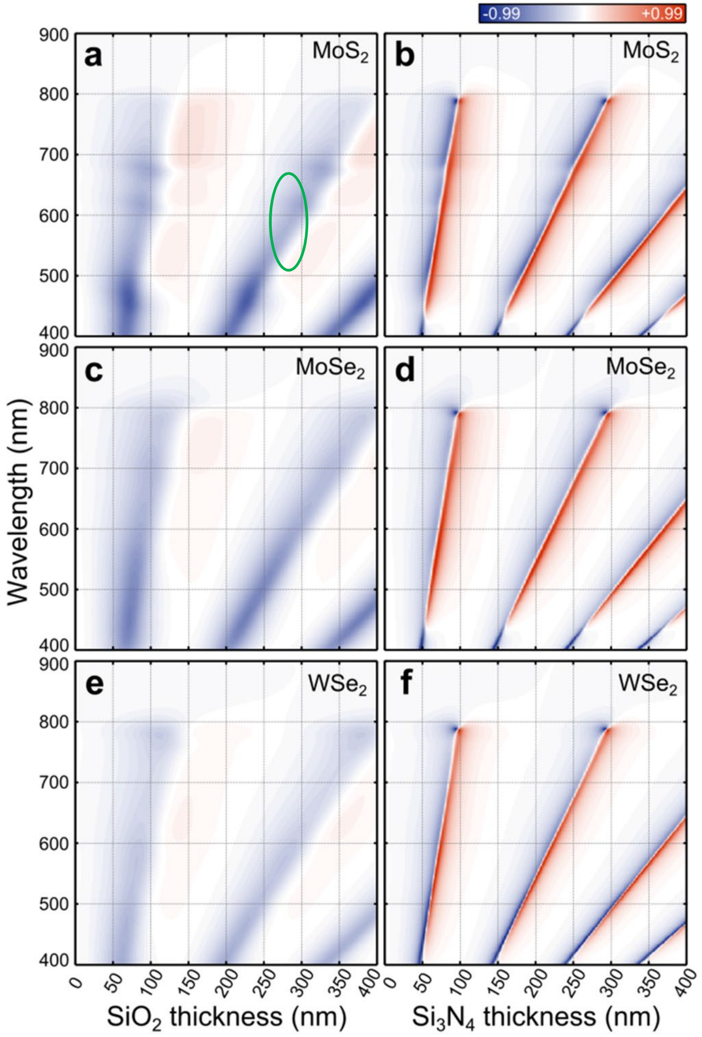
Good contrast



Bad contrast



Contrast for graphene on Si/SiO_2



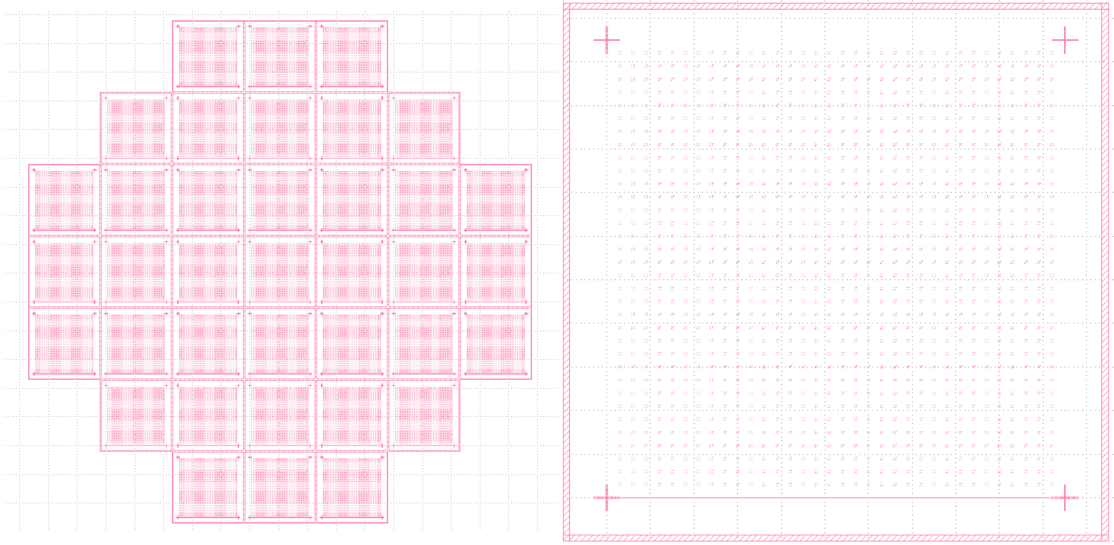
Good underlying dielectric thickness for good contrast

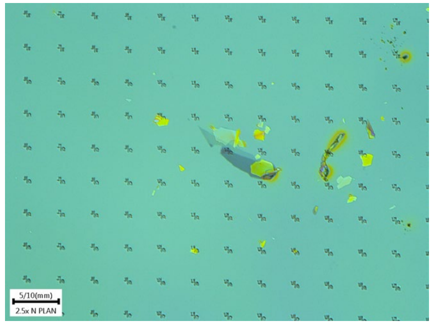
Rubio-Bollinger, Gabino, et al. "Enhanced visibility of MoS2, MoSe2, WSe2 and black-phosphorus: making optical identification of 2D semiconductors easier." *Electronics* 4.4 (2015): 847-856.

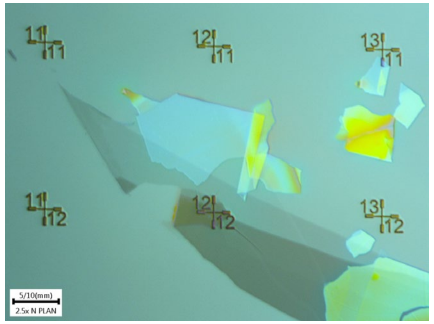
Blake, P., et al. "Making graphene visible." *Applied physics letters* 91.6 (2007): 063124.

Substrate cleaning and preparation

- Alignment marks
  - Before exfoliation
    - Use a 4” wafer to create an inventory of parts with standardized alignment marks to work
  - After exfoliation/growth
    - Use EBL/MLA to expose an array of alignment marks around the target material.
    - This needs an extra fabrication step.

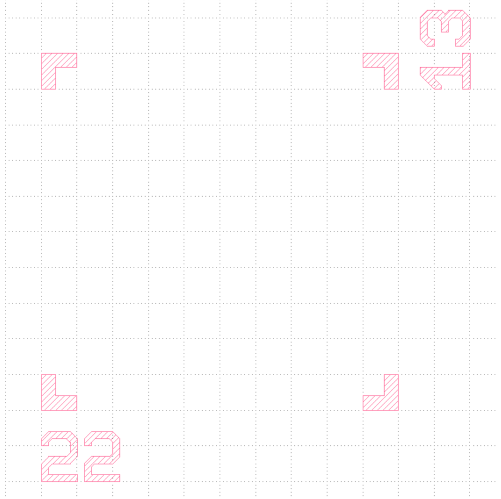






The diced parts must be cleaned properly before performing any fabrication

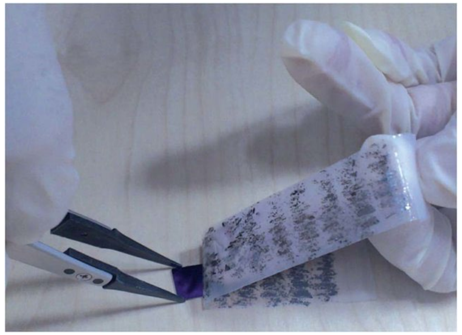
- **Acetone brush in the wet bench**
- **O_2 plasma clean for ~10 minutes using LOLA**



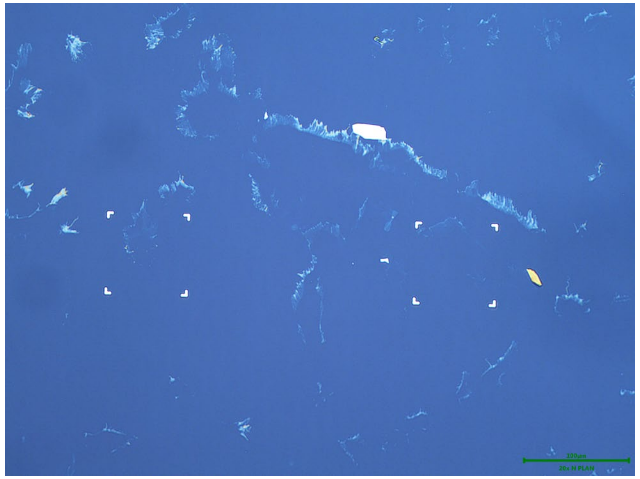
Good and bad mechanical exfoliation

Mechanical exfoliation is an adhesive or polymer-based exfoliation technique.

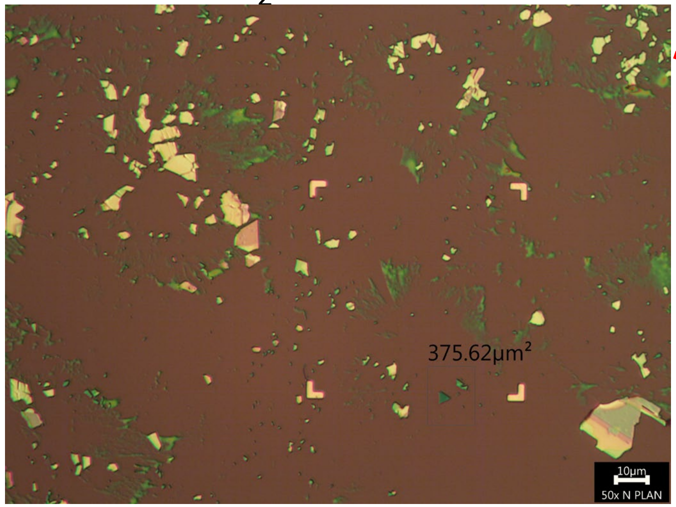
Scotch tape



Exfoliation needs to be performed on a hotplate at 100 C for MoS_2.



SPV-224 Nitto tape
"Lesser glue residue"

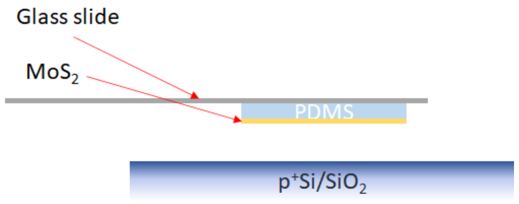


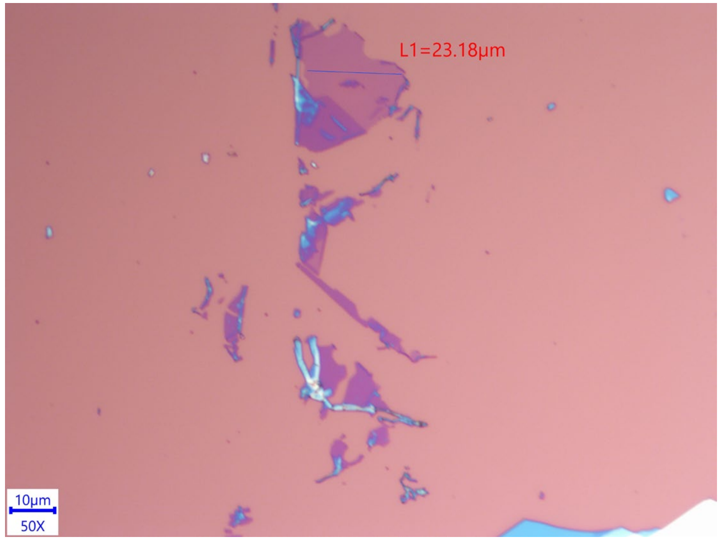
Observation:

1. Small and thick flakes
2. A lot of glue residue
3. Too many unwanted flakes

Optimized exfoliation process

Tape exfoliated samples stamped onto PDMS

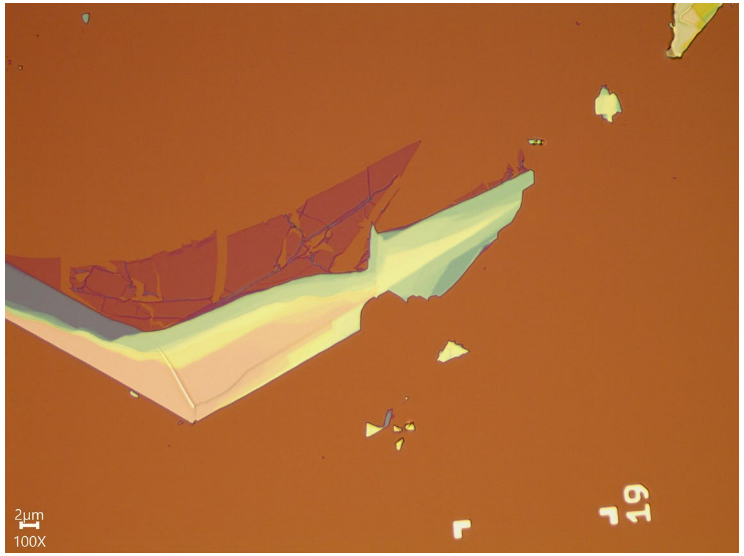




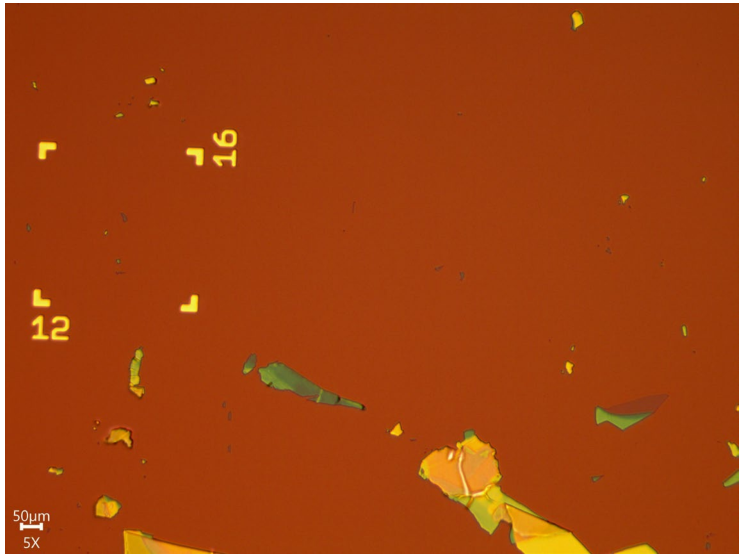
Wei, Jiacheng, Thuc Vo, and Fawad Inam. "Epoxy/graphene nanocomposites–processing and properties: a review." *Rsc Advances* 5.90 (2015): 73510-73524.

Correct way of locating target 2D flakes

- Optical images of flakes of interest must be taken along with alignment marks in the same image.
- A standard contrast to have an idea of the thickness of the target material.
- Images taken at multiple magnifications.
- Include Debris/unwanted material in the picture if present close to target material.



Good for locating but not good for EBL.

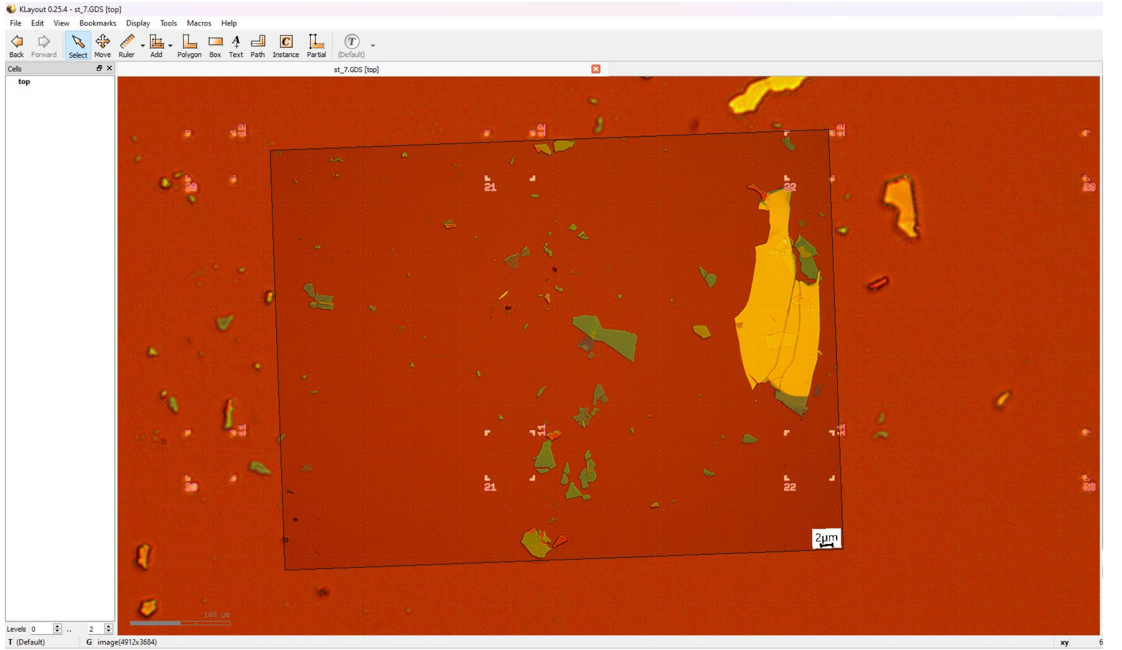


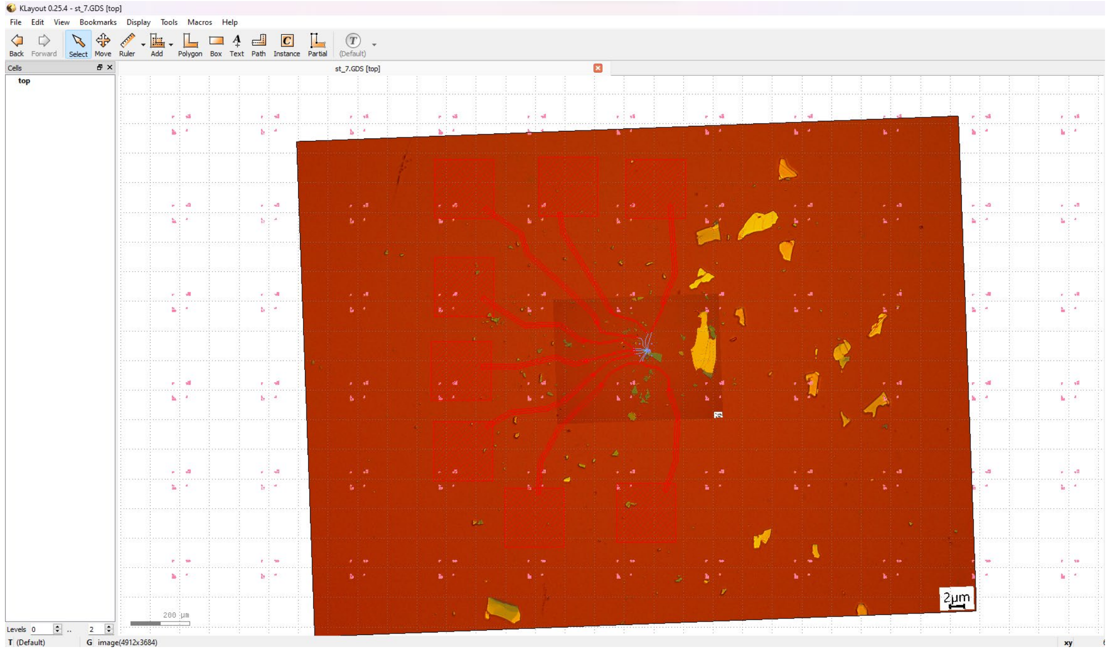
50x image

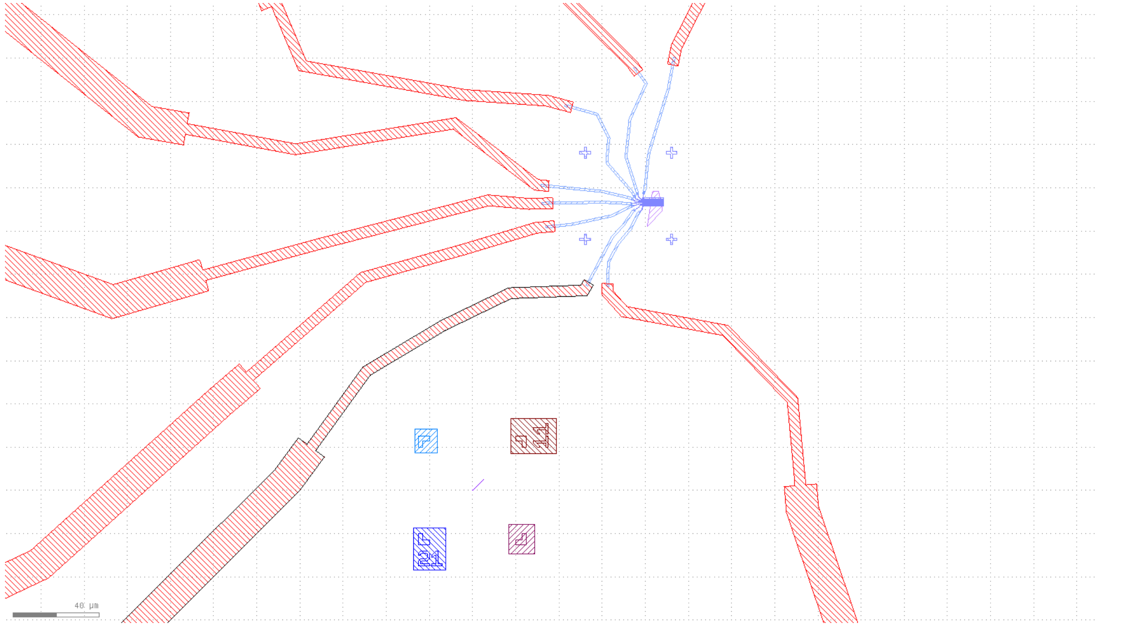
20x image

Patterning electrical leads on randomly oriented flakes

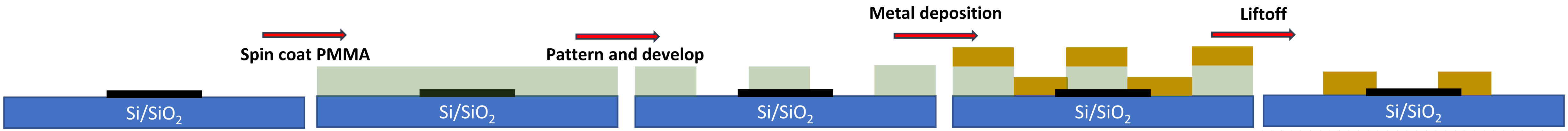
- Due to randomly oriented flakes one needs to create an image overlay on the alignment marks of the diced part
- Things to keep in mind while designing:
  - Making sure the leads and the lead extensions to contact pads do not touch any other Flakes (multiple magnification images come in handy).
  - If EBL has multiple steps it is recommended to make alignment marks close to target flake in 1st step for better alignment.
  - Alignment marks used for aligning the sample will get exposed and will develop out, hence tolerances need to be kept in mind
- If one needs to etch the material before patterning, Etch mask need to be patterned along with the electrical leads.

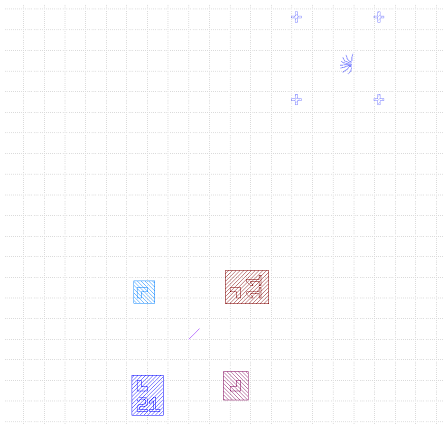


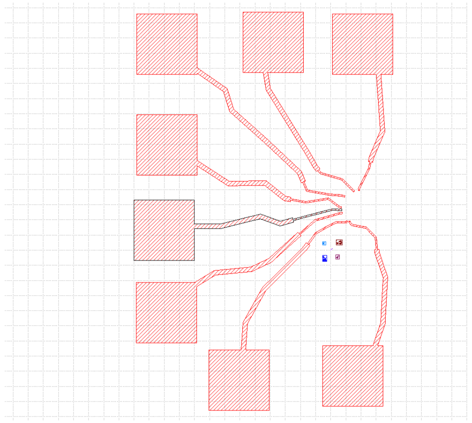




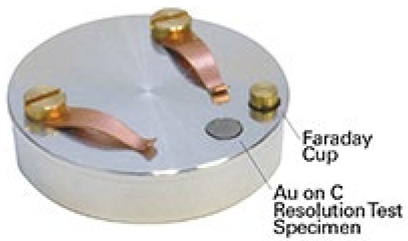
Things to keep in mind while running NPGS using an SEM tool



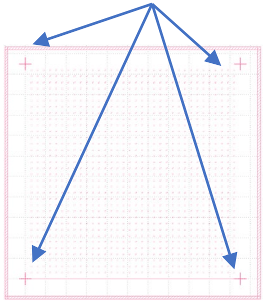

CAD file for small features

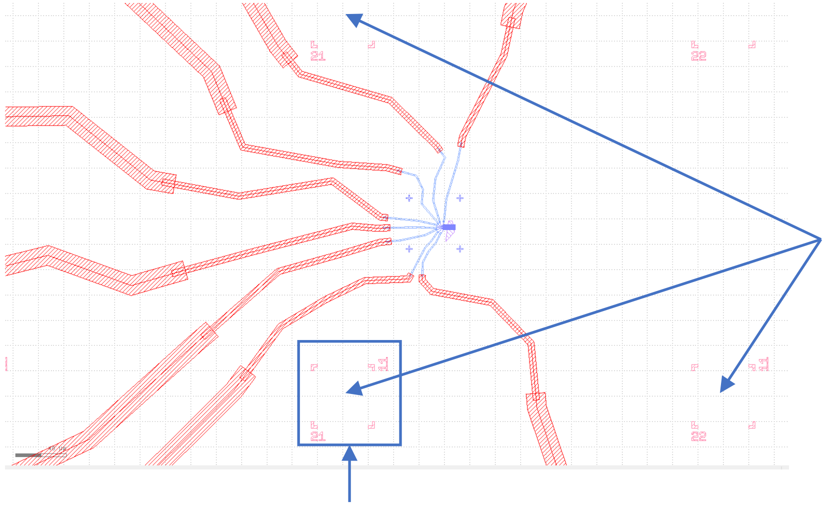

CAD file for larger features

- Field of View based writing:
  - The whole CAD pattern needs to be in field of view of SEM.
  - Break the pattern into multiple files
- Correct focus near the write field:



Global alignment marks





Local alignment marks

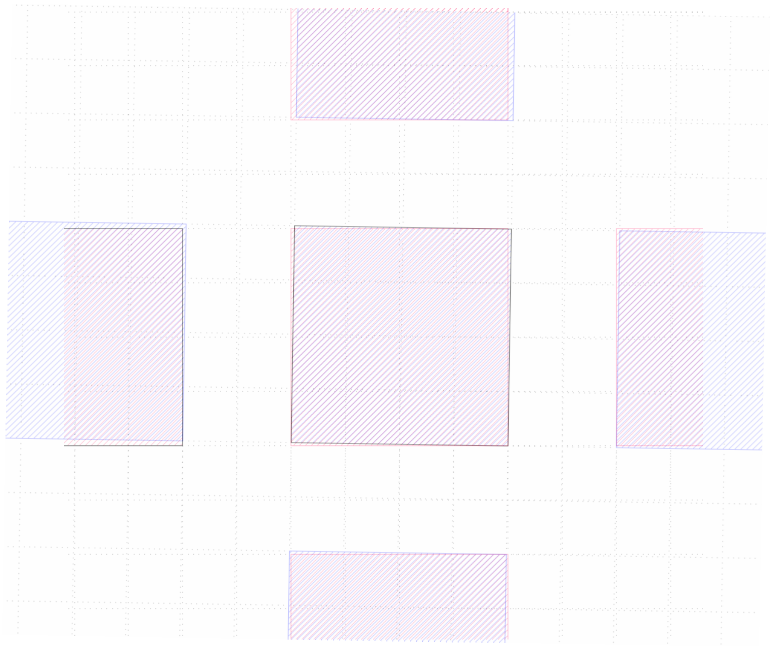
Places that can be used for better focus near write field

In order to focus the point, one needs to navigate to the correct place accurately

Global alignment and navigation using NPGS

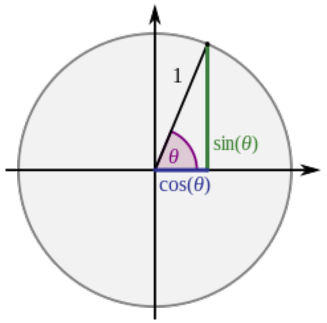
Why use NPGS and not SEM's software

- When aligning global marks, one normally uses XT-Align feature on SEM (visual alignment)



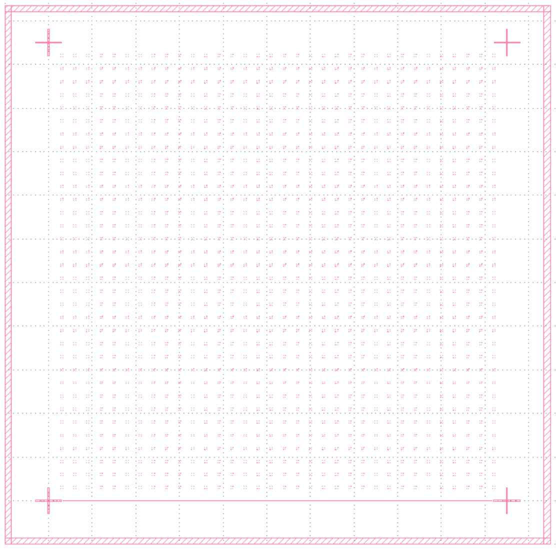
1 degree offset global alignment mark

Let's say we want to move 1 cm in x-axis,
The relative y-movement for 1^0 is $y = 1cm \times \sin(1^0)$
~170 μm



Using Global rotation matrix and Direct stage control on NPGS

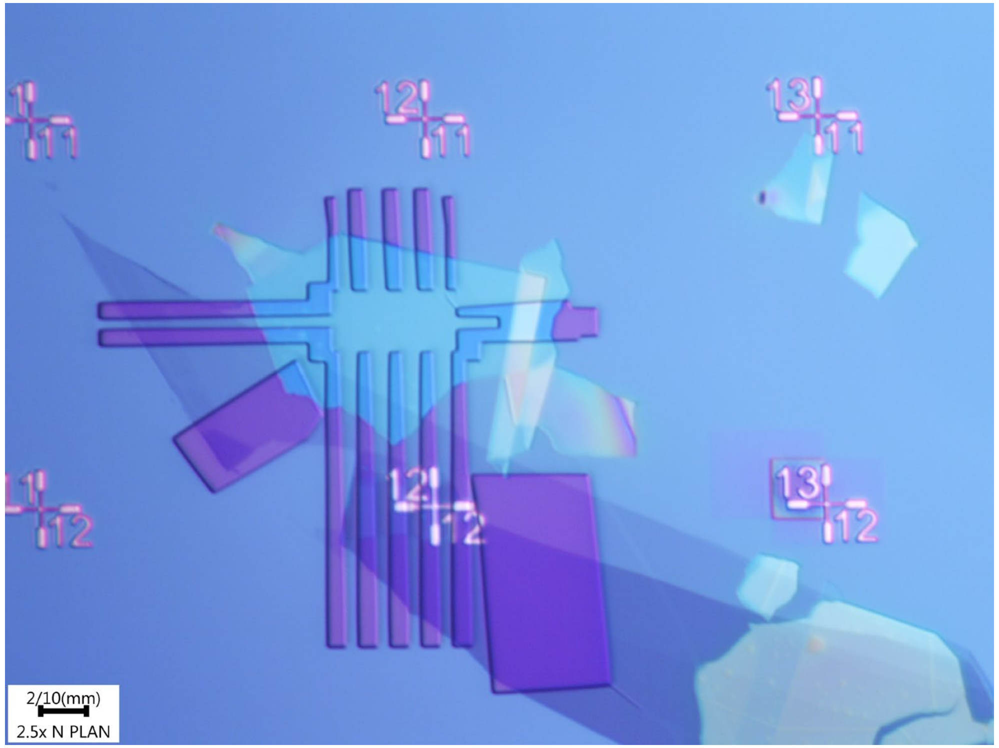
- Using global rotation matrix takes care of the rotation as it is Stage position based and not vision based.
- Calculation of matrix needs 3 points on Sample. They must be $(0,0)$, (x_1, y_1), (x_2, y_2).
- Using "Direct stage control" in NPGS menu rectifies the small angle error by performing relative stage movement.



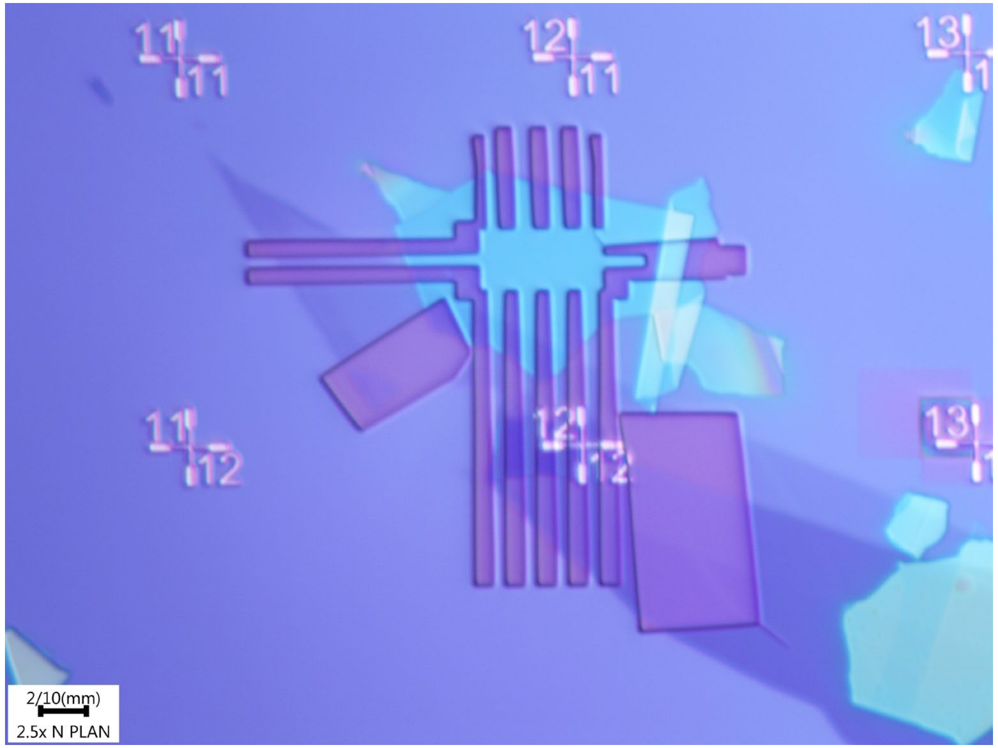
Etching

- One can fabricate 2D heterostructures by hBN encapsulation for higher electrical mobility in the channel.
- One needs to etch material to make electrical contact with the semiconducting/semi metallic channel.
- RIE can be used to etch out materials by opening windows using EBL
  - Must be designed properly by keeping the electrical path in mind

- Oxygen plasma etches graphene
- A mixture of Ar and SF_6 plasma etches hBN and MoS_2.

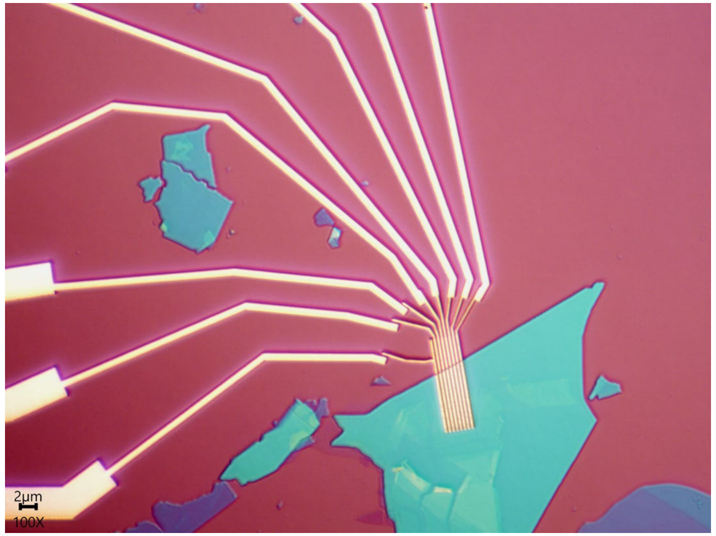


Before Etch

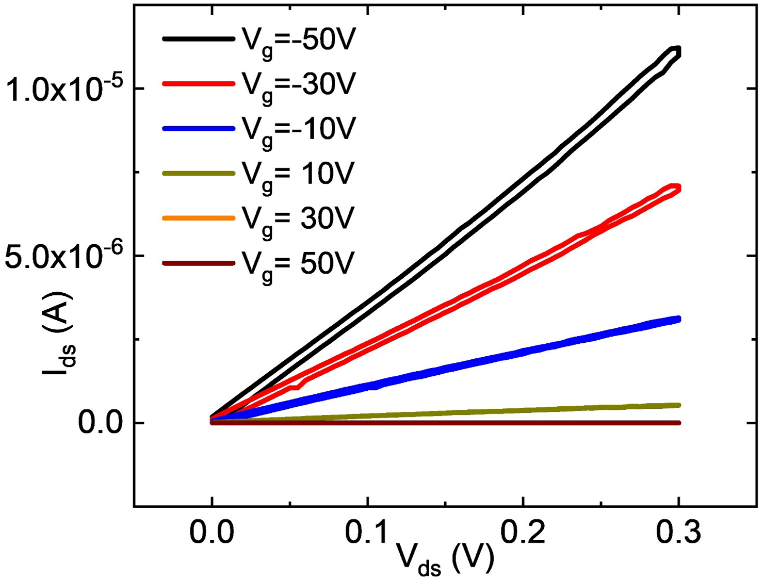


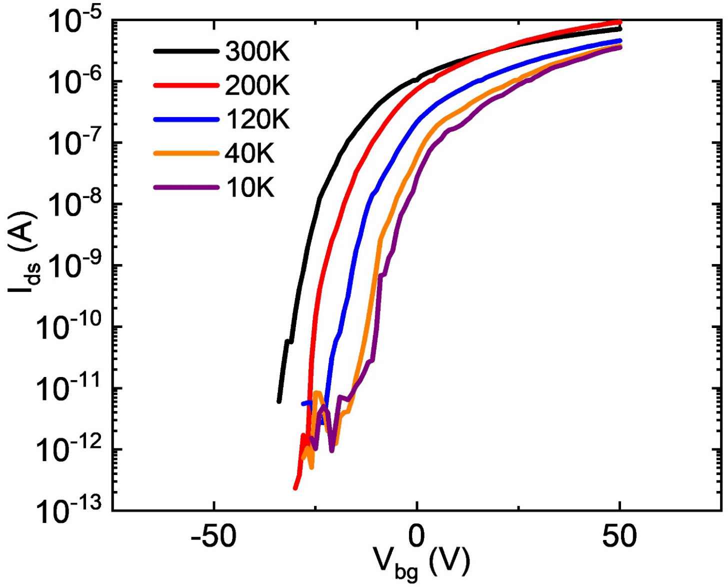
After Etch

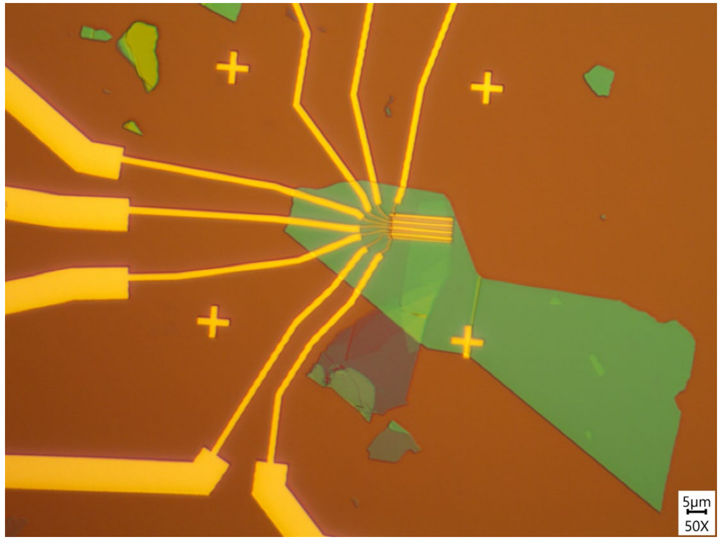
Example devices and Measurements



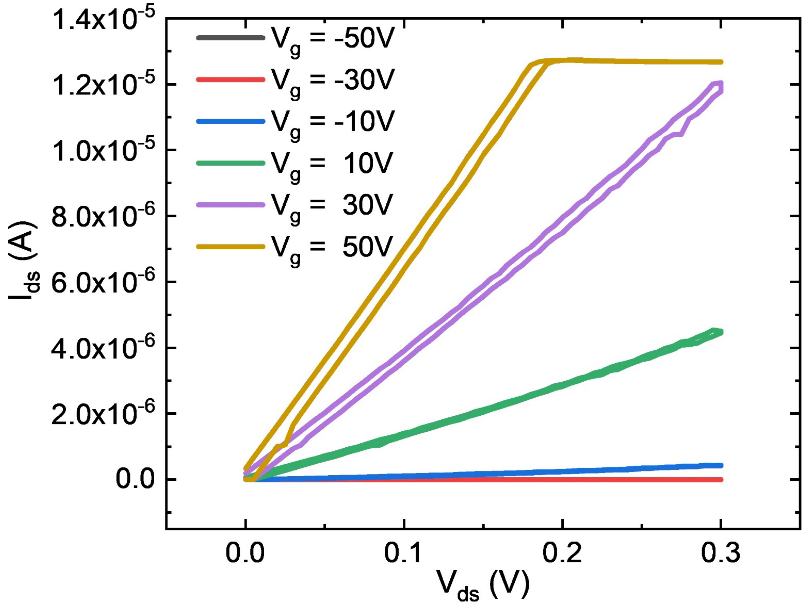
Leads not aligned
Test all the available leads.

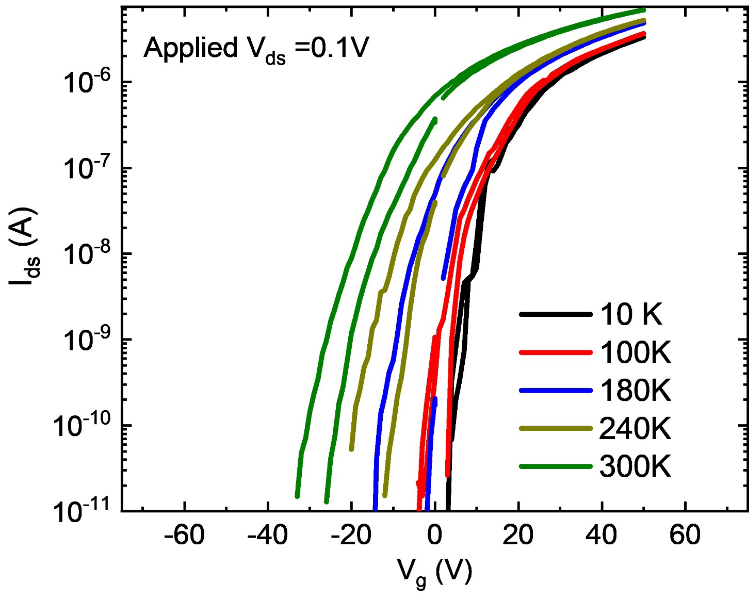






Aligned Leads





Questions?