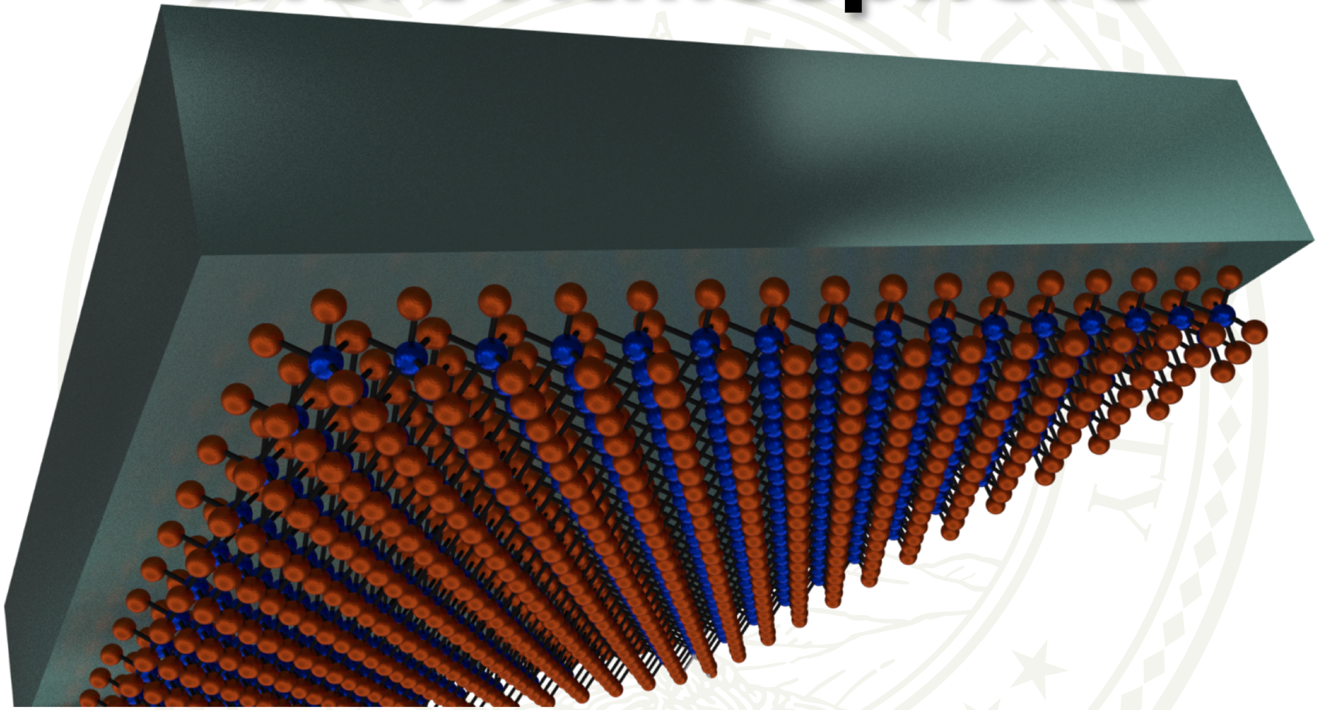
2D Materials Transfer in an Inert Atmosphere



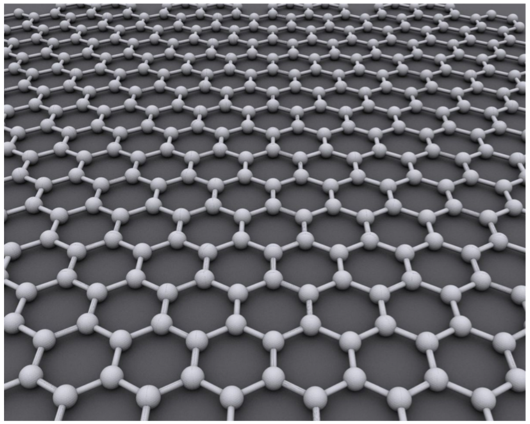
Team: Victoria Chen and Connor Bailey

Mentors: Michelle Rincon and Hye Ryoung Lee

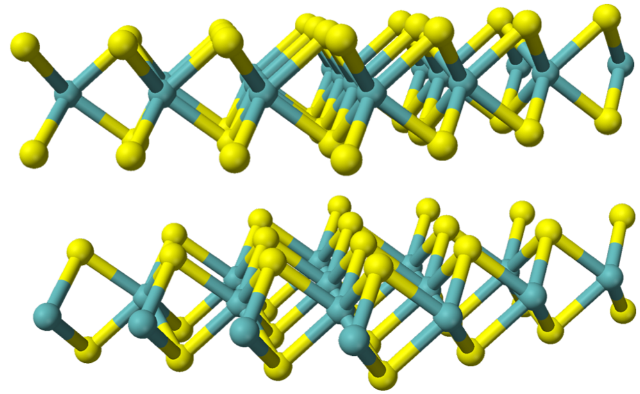
ENGR 241 – June 7th, 2018

2D Materials

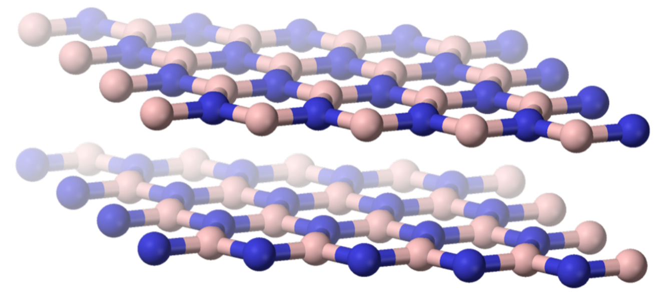
Band Gap → Increasing



Graphene
(Semimetal)



Transition Metal Dichalcogenides (MoS_2, WSe_2, etc.)
(Semiconductors)



Hexagonal Boron Nitride (hBN)
(Insulator)

- Naturally layered crystal structures (no dangling bonds)
- Applications in electronics, catalysis, optoelectronics, and more
- Thinness yields unique mechanical, thermal, optical properties
- Two methods to obtain thin samples:
  - Mechanical exfoliation from bulk crystal ("Scotch-Tape Method")
  - Direct CVD growth

→ Transfers are necessary to get material on arbitrary substrates

Image Source: Wikipedia

Material Air-Stability

	$-S_2$	$-Se_2$	$-Te_2$
Mo			
W			
Nb, Sn			
Hf, Zr Ta, Ti			

WSe_2 selected for this project:

- Ambipolar semiconductor
- Relatively air stable (~weeks-months)
- CVD capabilities at Stanford

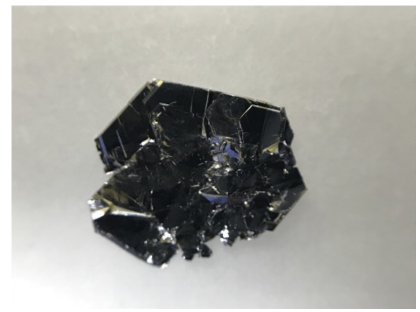
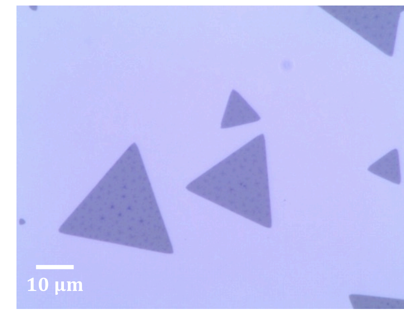
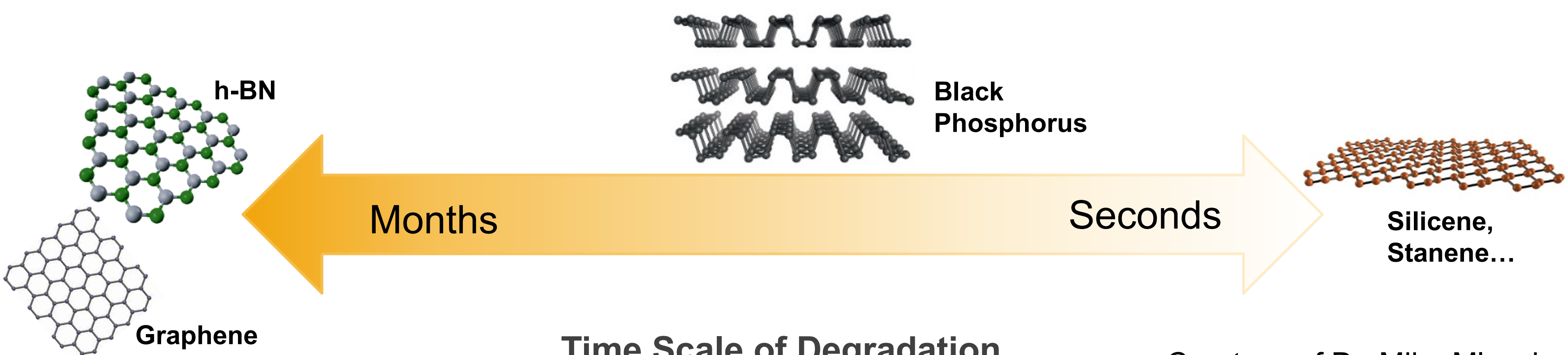


Image Source: 2D Semiconductors

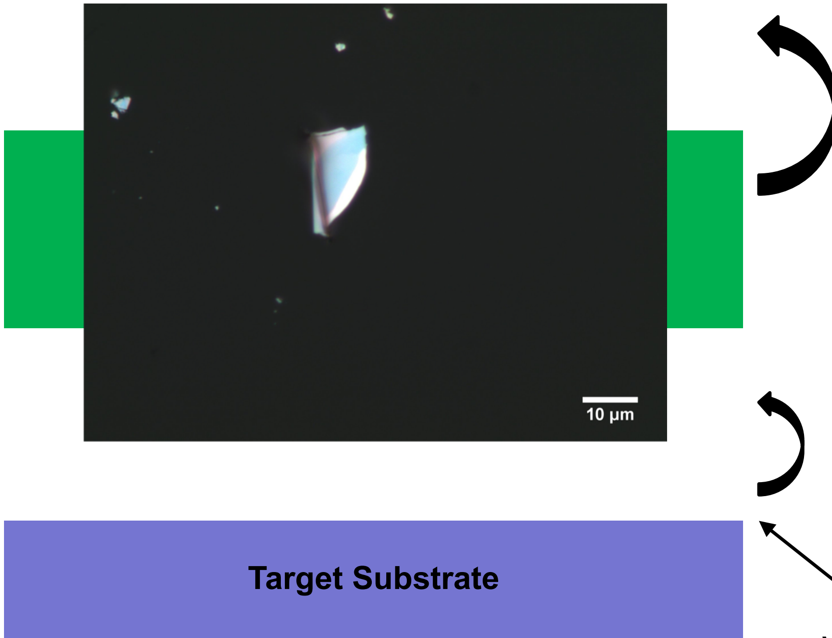


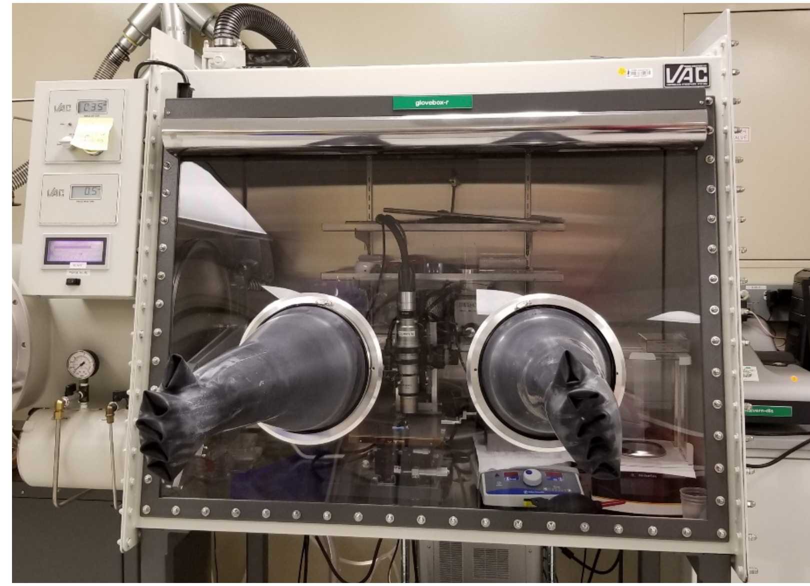


Time Scale of Degradation

Courtesy of Dr. Mike Mleczko

Transfer Process in ExFab Glovebox





WSe_2 transferred to arbitrary substrate

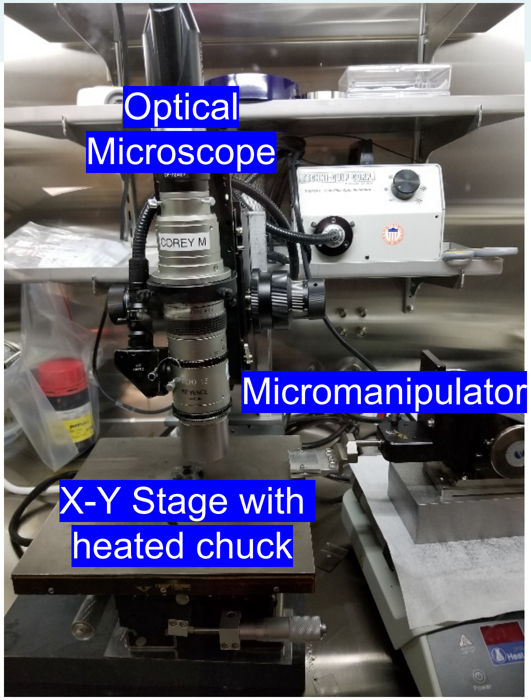
- PDMS used as a “handle” to stick to and exfoliate WSe_2
- Heat helps release the PDMS from the target substrate
- Solvents used to reduce polymer residue from PDMS and tape
- Flakes with lateral dimensions of 10s-100s of µm

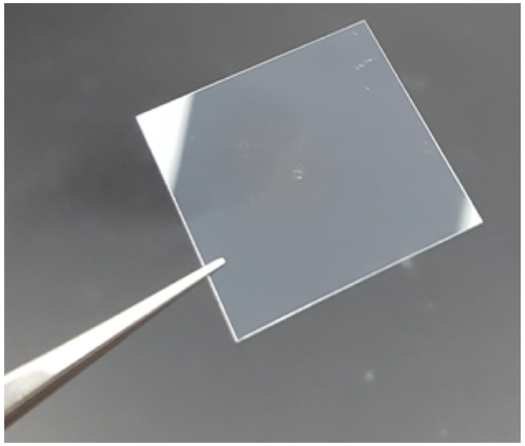
Design of Experiment

- Goals: **optimize** the transfer process conditions and **explore** any variation induced by different target substrates

PDMS Base:Curing Agent Ratio	PDMS Release Temperature	Target Substrate
5:1	130°C	SiO_2/Si
7.5:1	150°C	PEN
9:1		

Curing ratio affects consistency and stickiness of PDMS

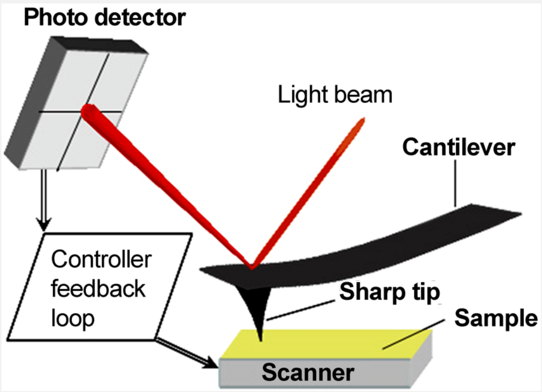




PEN – Polyethylene naphthalate (Flexible Substrate)

Characterization Techniques

Atomic Force Microscopy (AFM)

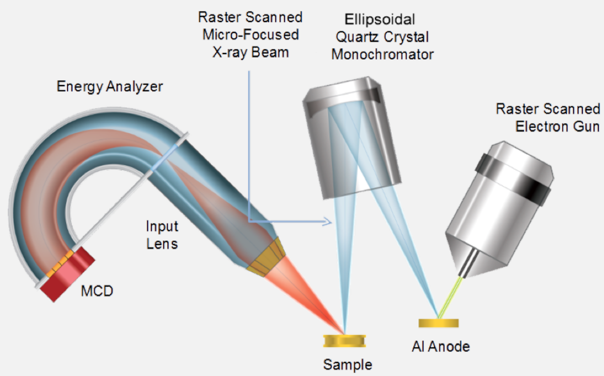


Nat. Hazards Earth Syst. Sci. **17**, 31-44 (2017)

- Sharp tip rasters across a sample
- Deflection correlated to topography

✓ Measured surface roughness of flakes after transfer

X-Ray Photoelectron Spectroscopy (XPS)

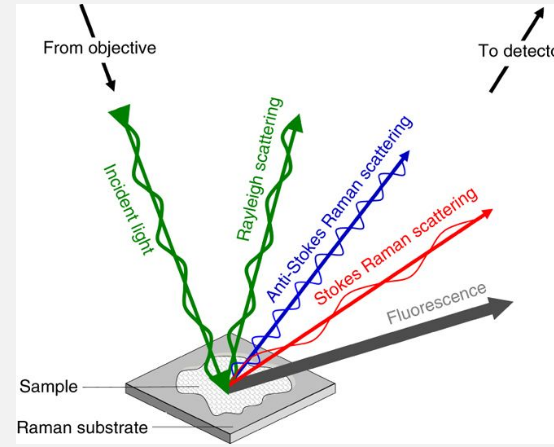


PHI Company

- X-rays stimulate the release of photoelectrons
- Measures bonding energies

✓ Used peak ratios to assess residue left behind by our transfer process

Raman Spectroscopy

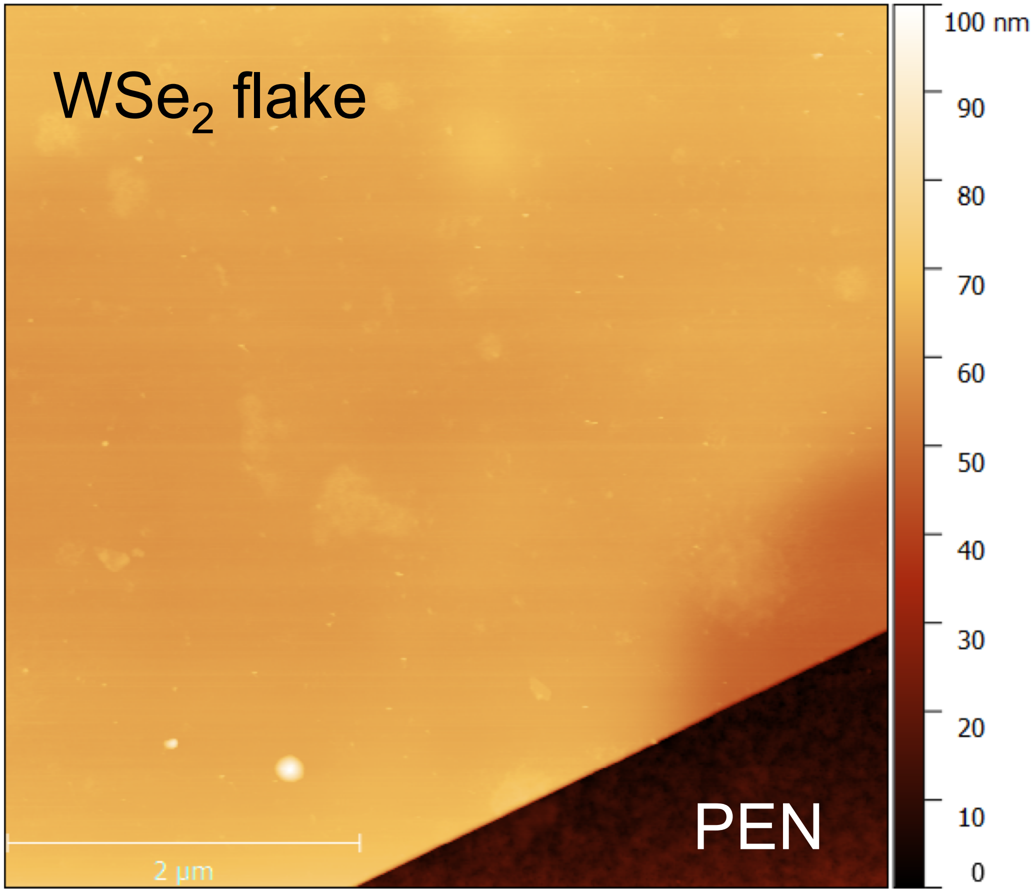


Nature Protocols **11**, 664-687 (2016)

- Relates vibrational modes to light scattering
- Spectrum acts as a material "fingerprint"

✓ Used the width and position of major peaks to quantify material quality

AFM: Surface Roughness Analysis



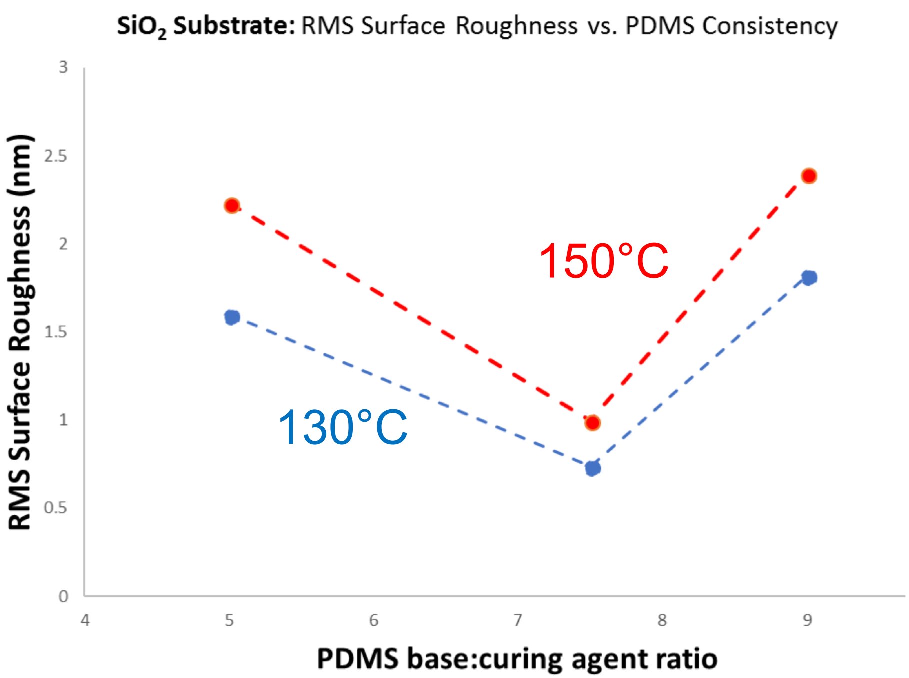
AFM image of WSe_2 flake transferred to PEN

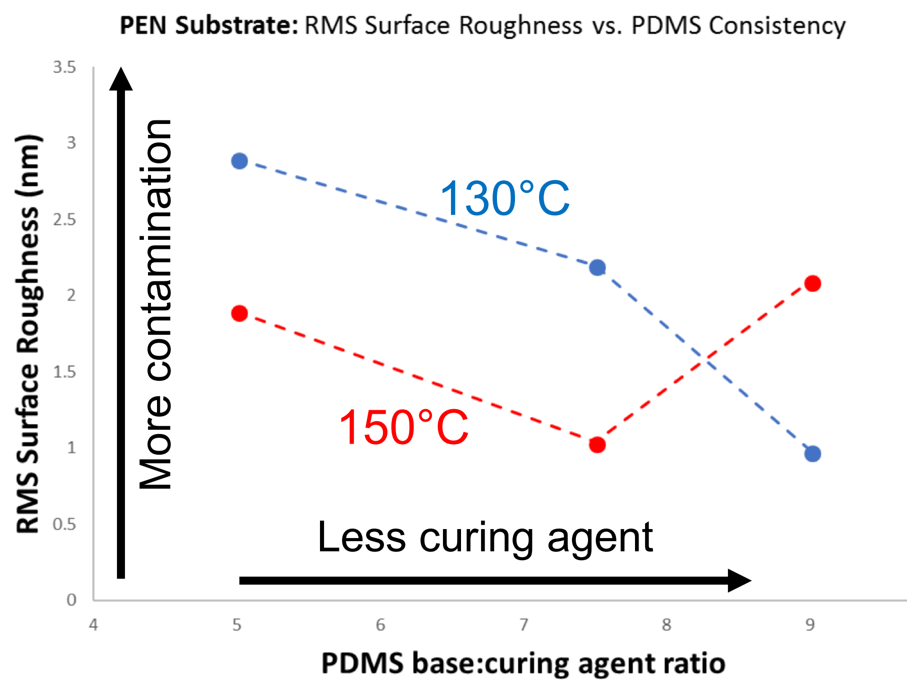
Surface topography:

Increased levels of residue from transfer should increase surface roughness of transferred material

Goal is to detect residue levels that may not be visible optically

AFM: Compiled Surface Roughness Data





SiO_2 substrate: initial surface roughness is 2.09 nm

PEN substrate: initial surface roughness is 2.00 nm

Surface roughness is on the order of the substrate, but flakes are relatively thick

Tentative conclusions:

- 7.5 (base) to 1 (curing) may leave the least residue
- Lower release temperature is better for Si substrate, higher for PEN

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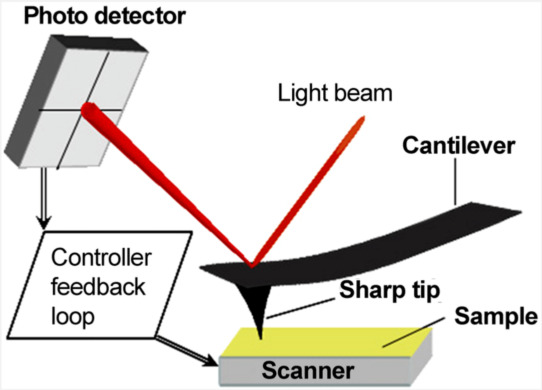

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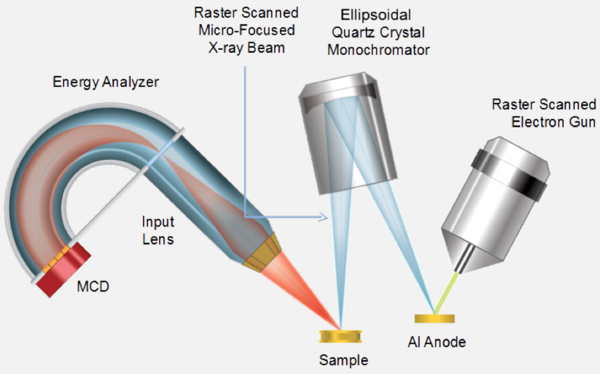

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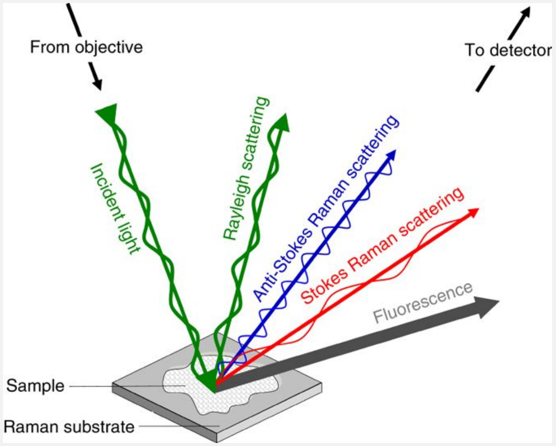

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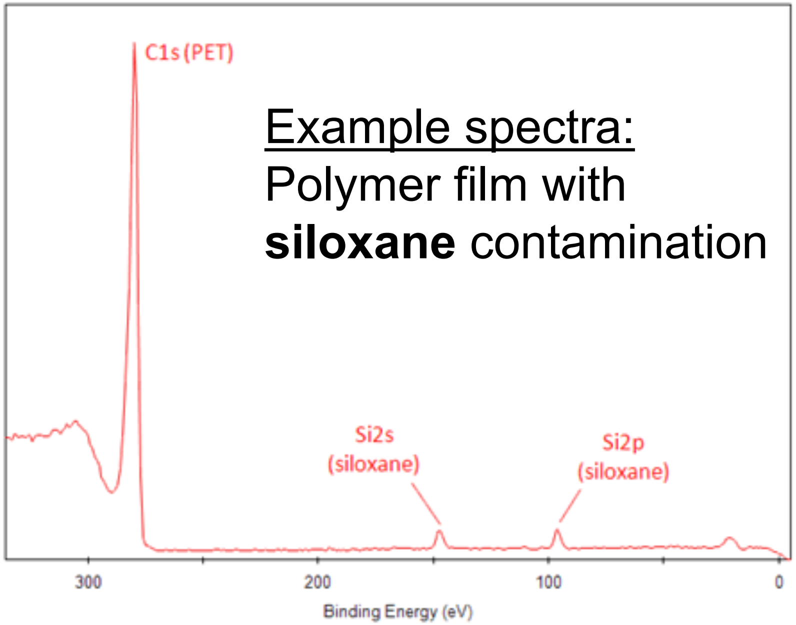
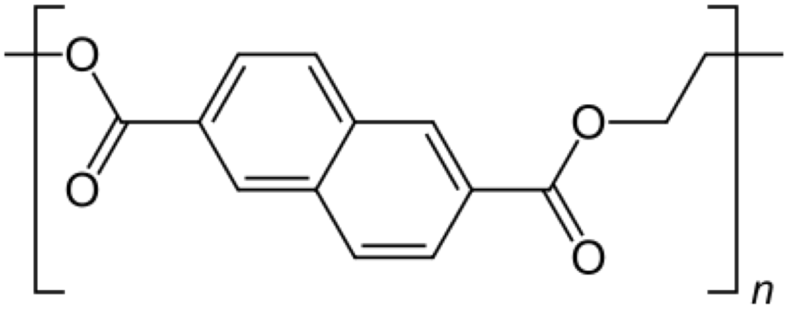
XPS: Qualitative Assessment of Residue

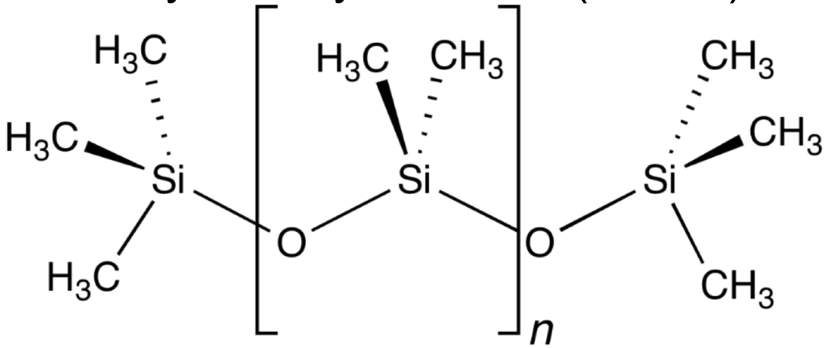
- PEN target substrate should not inherently have Si peaks
- Si peaks would indicate contamination
  - Likely induced by environment or transfer process
- SiO_2 target substrate should not inherently have C peaks
- C peaks would indicate contamination



Polyethylene naphthalate (PEN)



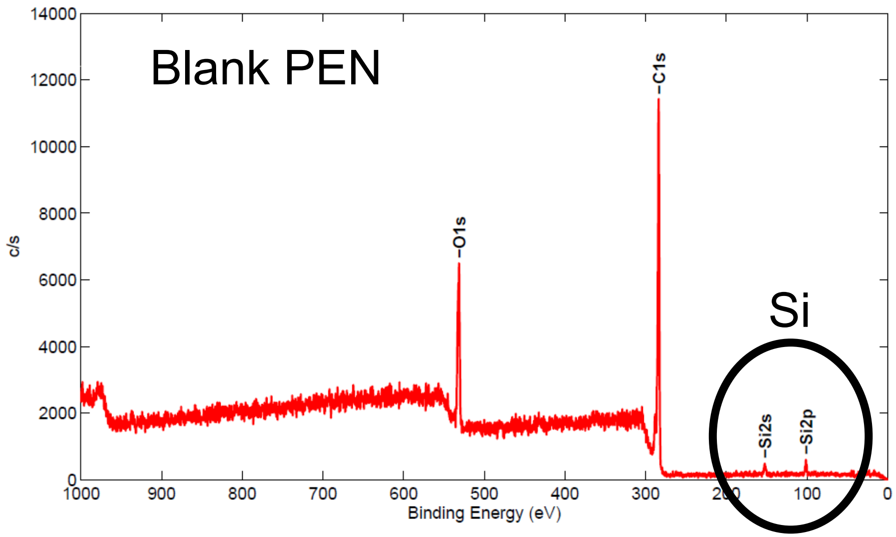
Polydimethyl**siloxane** (PDMS)

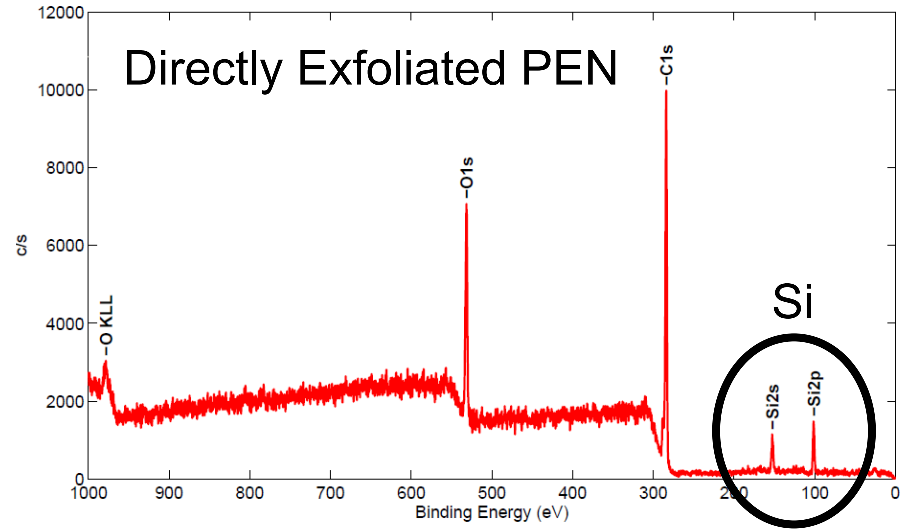


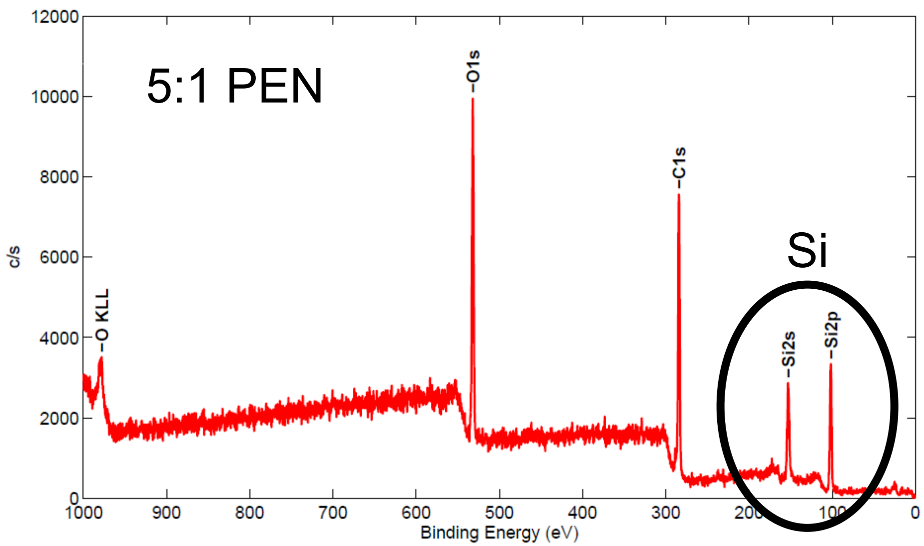
https://en.wikipedia.org/wiki/Polyethylene_naphthalate

https://en.wikipedia.org/wiki/Polydimethylsiloxane

XPS: PEN Substrate with 150°C Release Temp.







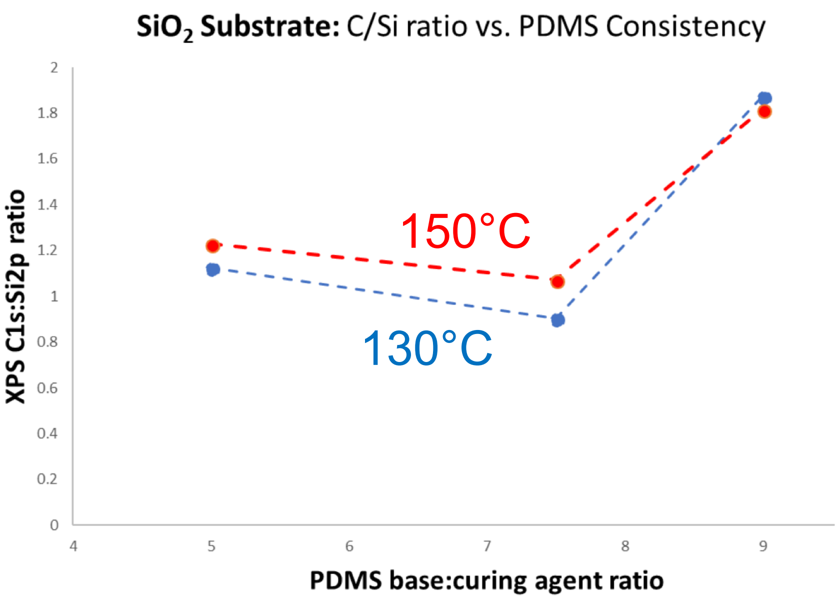
Increasing relative Si peaks

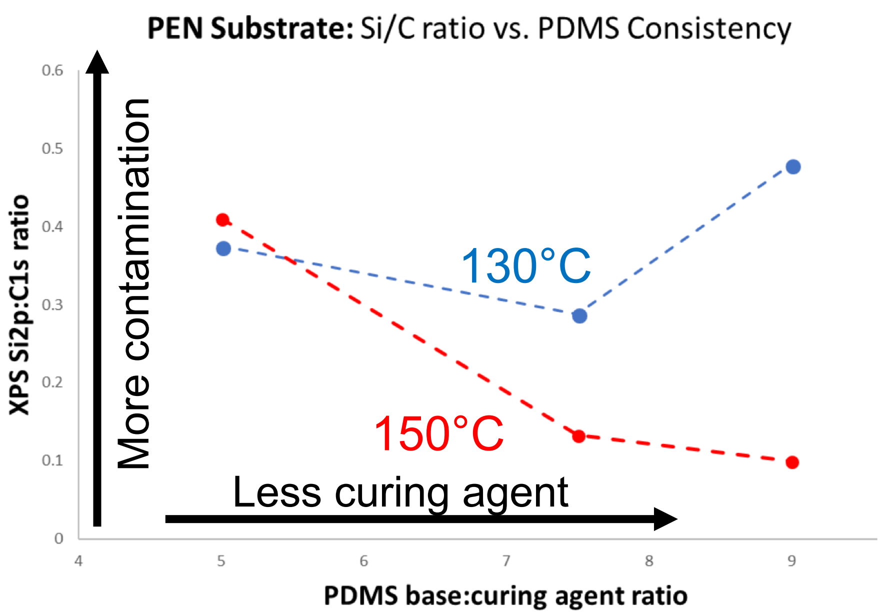
Blank PEN: handled with other samples but no direct contact with PDMS

Directly Exfoliated PEN: contact with blue exfoliation tape, but not PDMS

5:1 PEN: contact with PDMS

XPS: Compiled Data





SiO_2 substrate: assume C is contamination

PEN substrate: assume Si is contamination

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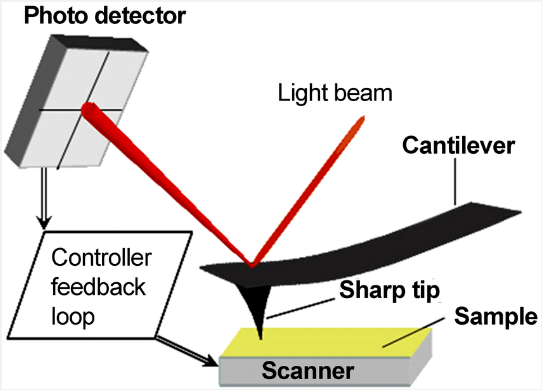

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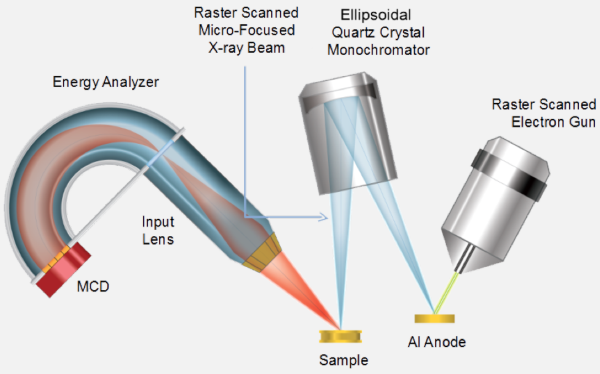

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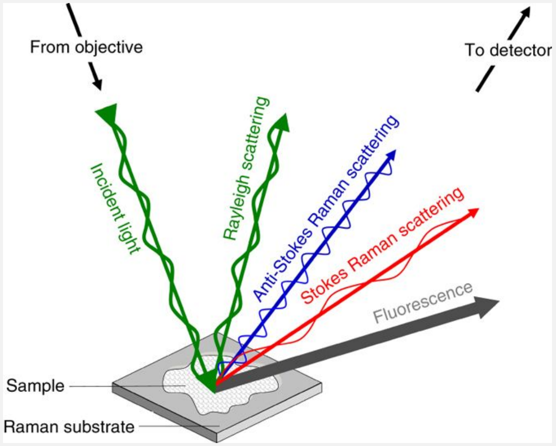

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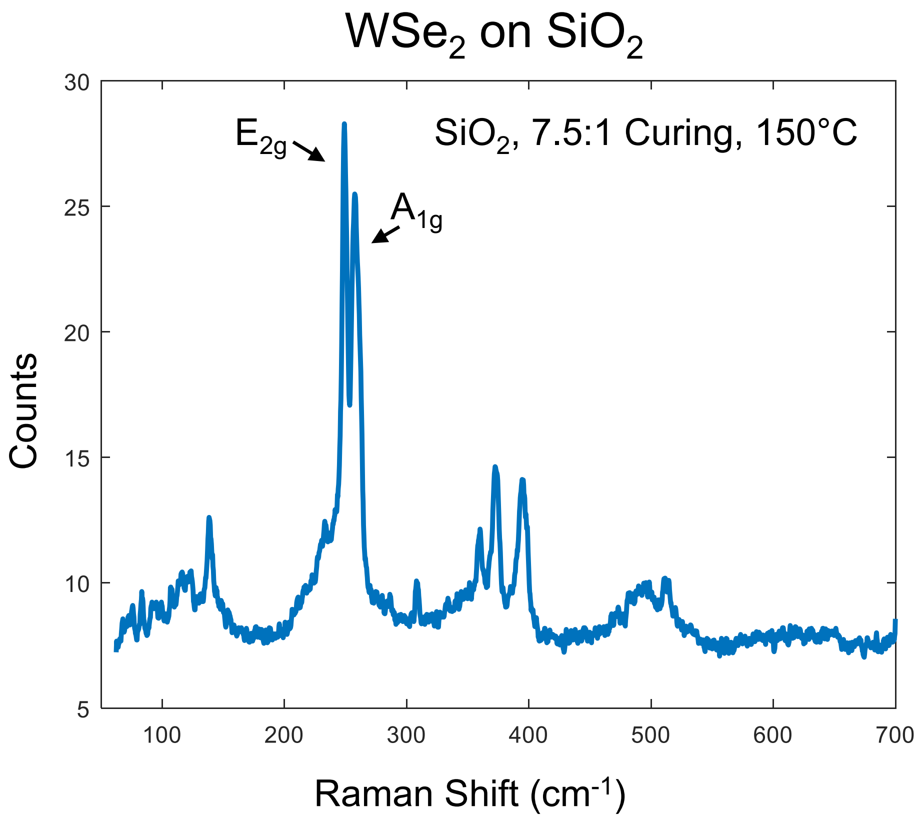
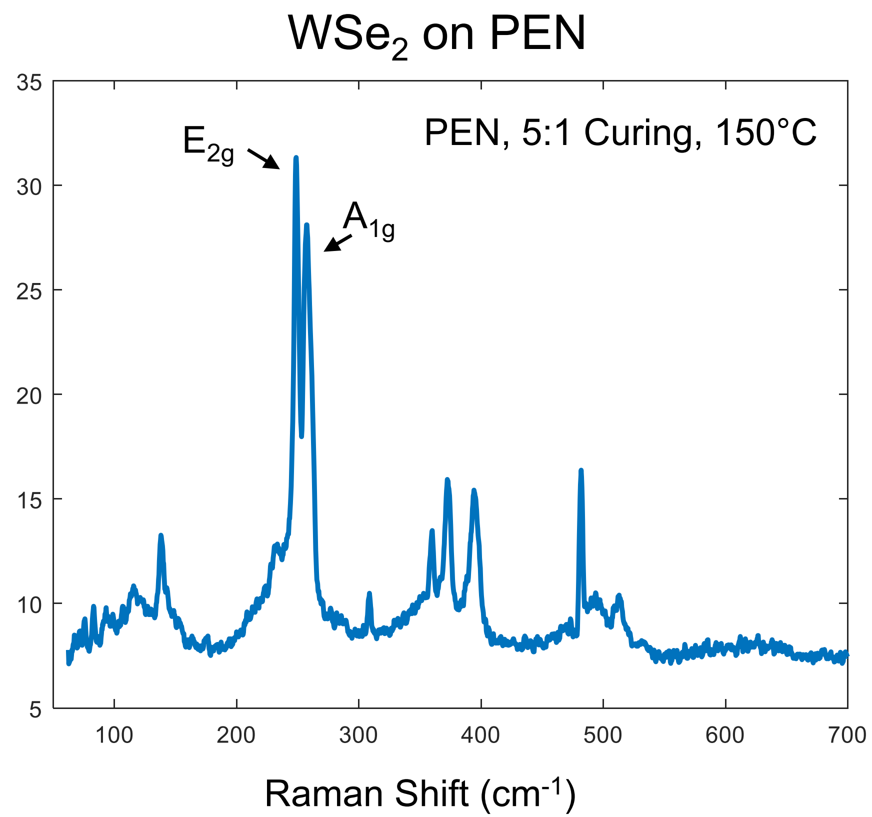
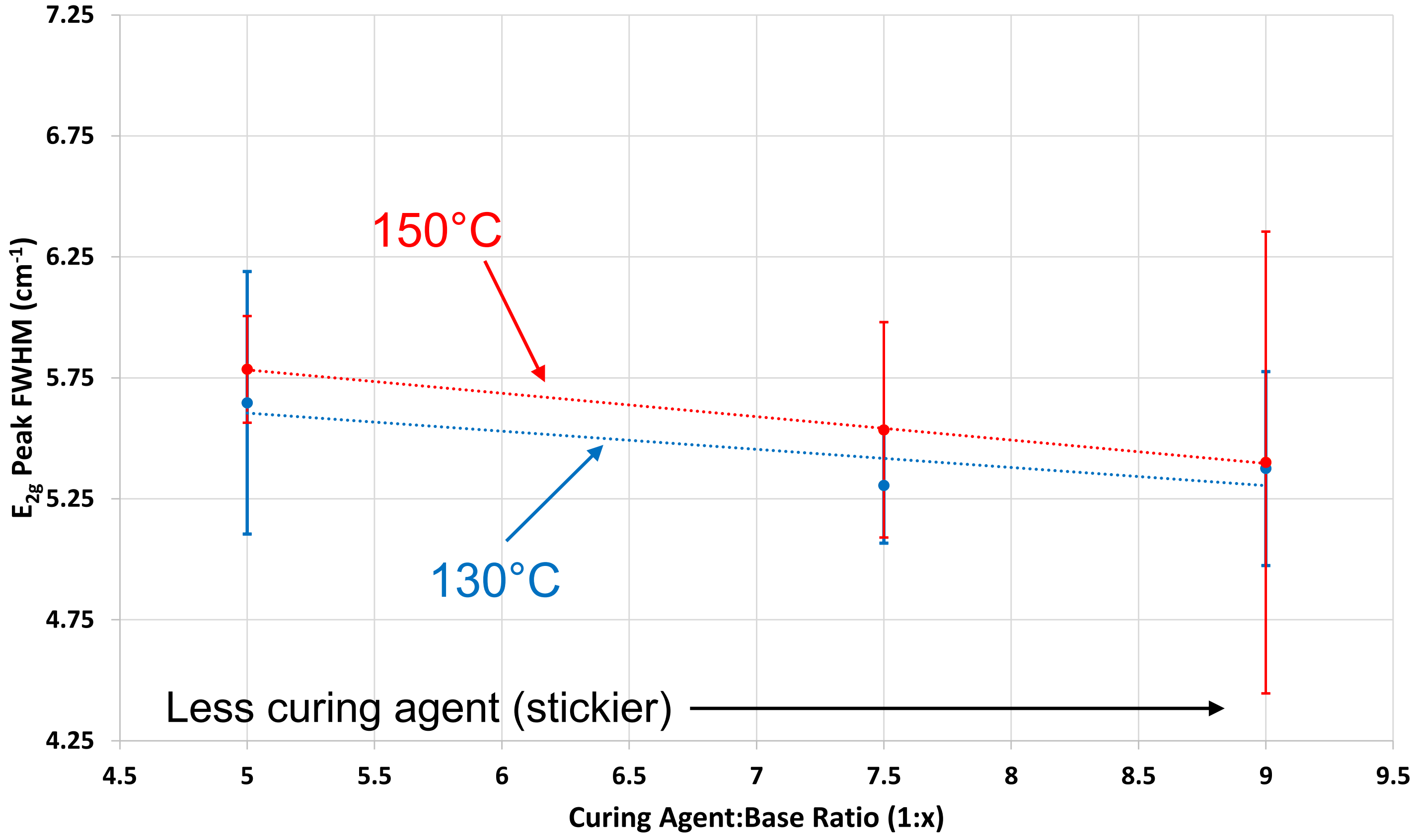
Raman Spectroscopy – Typical WSe_2 Spectra





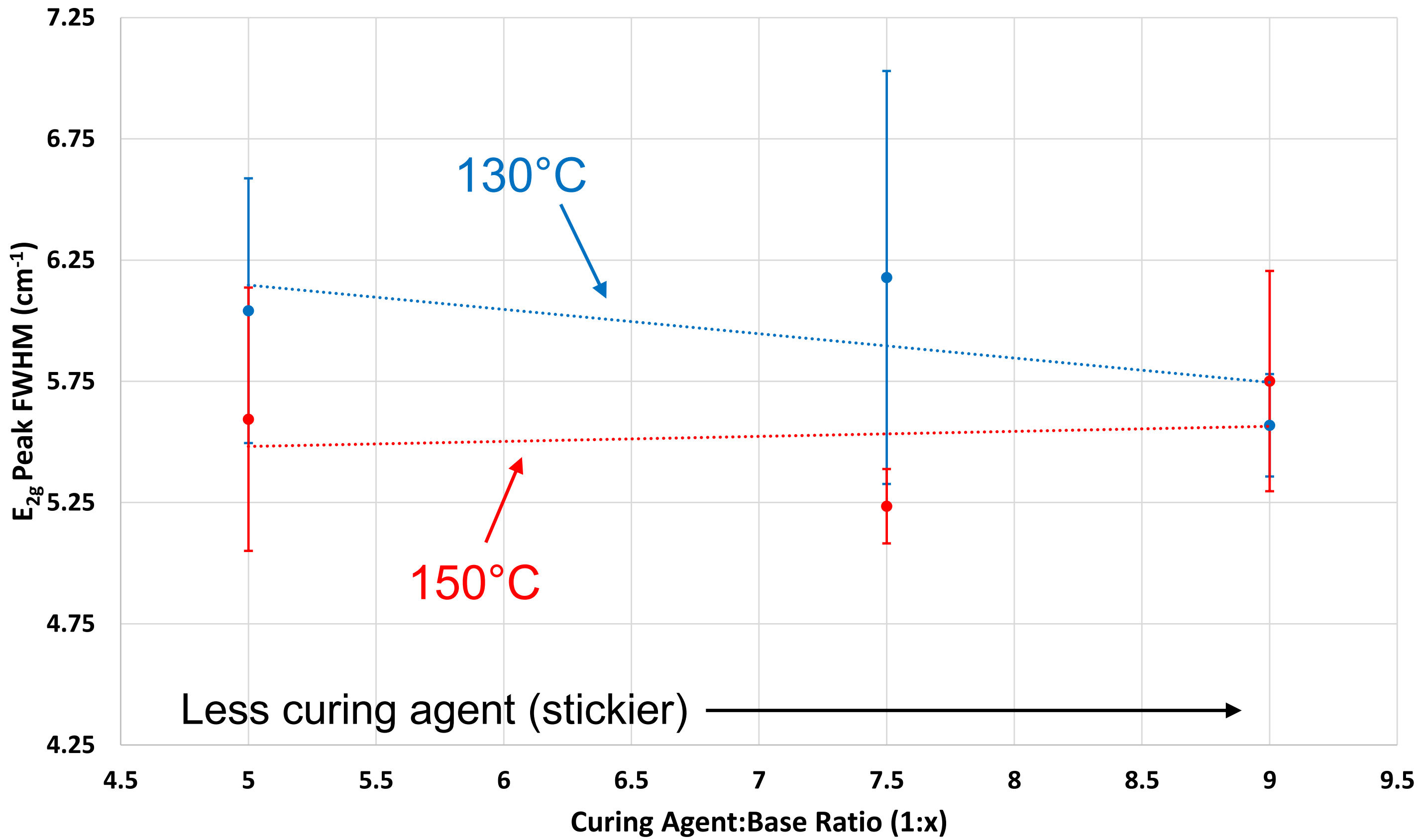
- E_{2g} peak is associated with in plane modes, A_{1g} out of plane
- E_{2g} peak height and width used to quantify quality

Raman Peak Width Trends – SiO_2/Si



- Lower release temperature → narrower Raman (better)
- Less curing agent → narrower Raman
- Baseline ~ 5 → Transfer induces some damage

Raman Peak Width Trends – PEN



- Opposite temperature trend → 150°C is better (?)
- Sample size of 3 flakes → more needed to see accurate trends
- Baseline ~5.5 → 150°C barely induces damage

Tentative Conclusions and Future Work

- Transfer conditions for lower residue, less material damage:
  - PDMS consistency: 7.5 (base) to 1 (curing agent)
  - Lower release temperature for SiO_2 target substrate
  - Higher release temperature for PEN target substrate

- Need more data points for further optimization

- Our contribution: a generalized transfer procedure that can be applied to a variety of material systems for different applications