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Few-Layer Transition Metal Dichalcogenide Exfoliation, Characterization, and Modeling for Future Electronics

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Goal

- Exfoliate monolayer flakes of transition metal dichalcogenides (TMDs) for characterization and devices, simulation of electrical and mechanical device behavior

Motivation

- Thin TMD flakes exhibit unique electrical properties (high mobility, small band gap[1]) and mechanical properties (high flexibility[2])
- Attractive for construction of low-power, nanoscale devices and memory, leading to demand for reliable source of flakes for prototyping

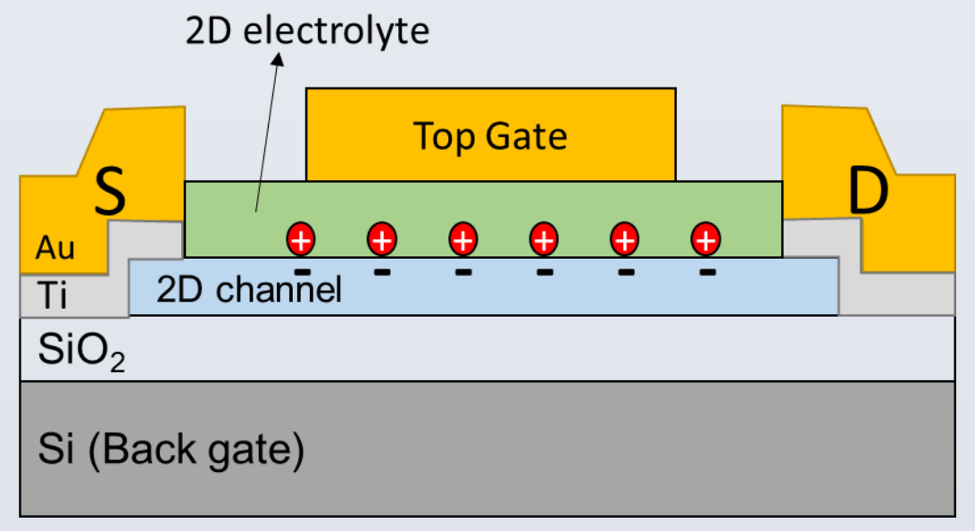

Fig. 1 Schematic of proposed device, using exfoliated MoS_2 and graphene as the 2D channel and top gate, respectively.

Exfoliation

- Mechanical exfoliation of thin flakes using the standard method, Figure 2.

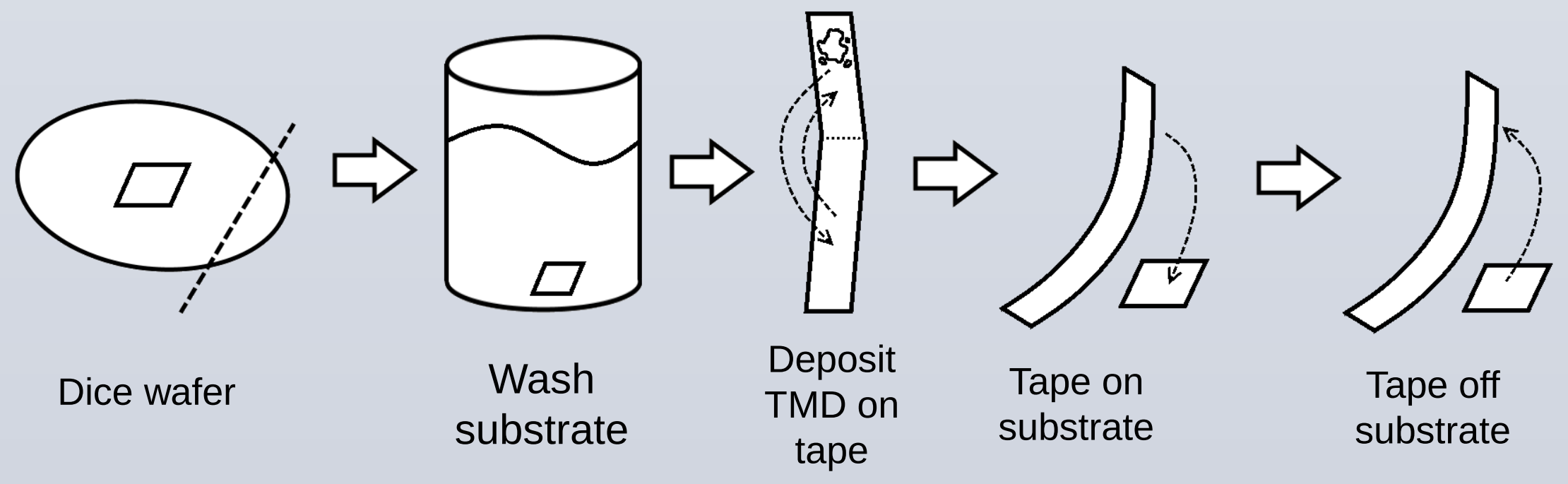

Fig. 2 Standard method for exfoliating thin flakes.

- A candidate flake for a device is longer than 15μm and thinner than 10nm.
- Standard method for MoS_2 yields poor results (2-3μm lateral size, 10-15nm thick)
- Experimenting with modifications to exfoliation process:
  - Thermal Annealing[3]
    - Anneal substrate for 5 minutes at 100° C prior to exfoliation
  - Reactive Ion Etching (RIE)[3]
    - Clean substrate with O_2 plasma prior to exfoliation
  - Varying levels of adhesive to perform exfoliation
    - Low (name), medium (name), high (Scotch)

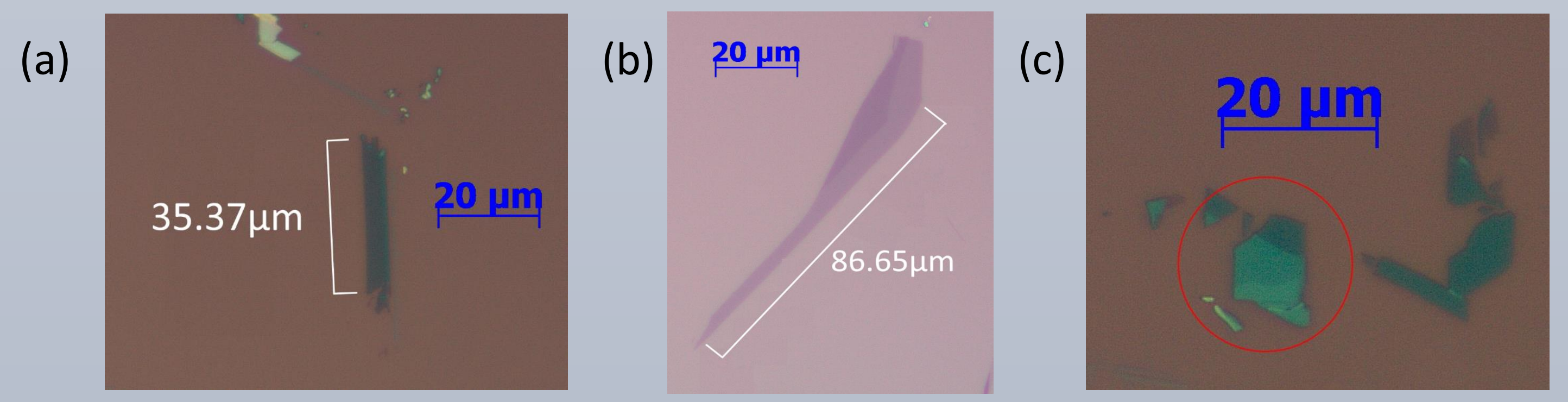

Fig. 3 Optical images of few-layer flakes. (a) Candidate MoS_2 flake exfoliated using thermal annealing . (b) Candidate graphene flake exfoliated using standard method. (c) An unusable flake (too thick).

Alternate MoS_2 Exfoliation Method Results

- Annealing with the medium adhesive tape (Fig. 4b) and annealing + RIE with low adhesive tape (Fig 4a) gave the best results with 1/2 depositions
  - RIE is an expensive and time-consuming process
  - Annealing with 1/2 depositions using medium tape best option

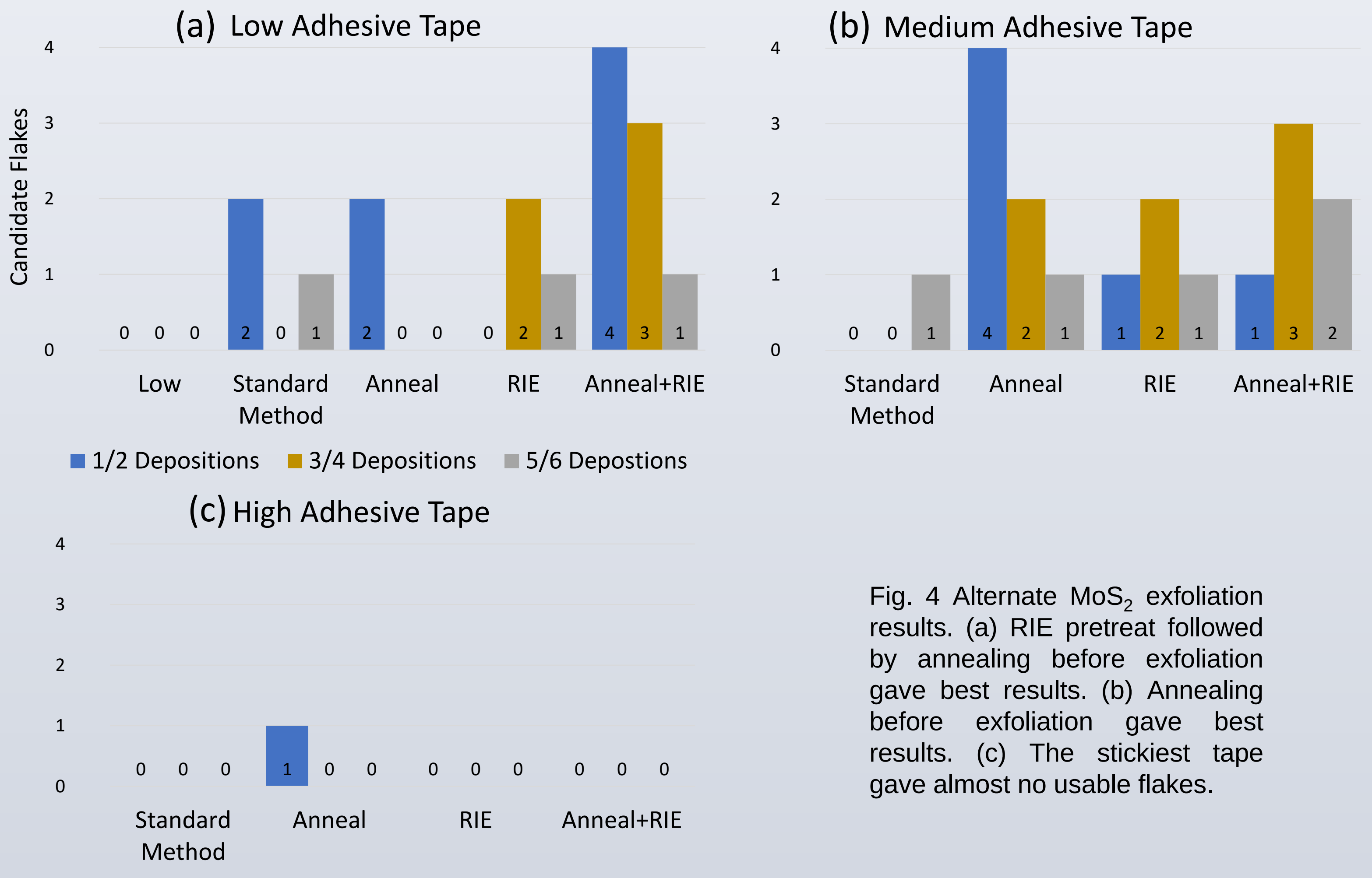

Fig. 4 Alternate MoS_2 exfoliation results. (a) RIE pretreat followed by annealing before exfoliation gave best results. (b) Annealing before exfoliation gave best results. (c) The stickiest tape gave almost no usable flakes.

Modeling

- When few-layer TMDs are bent, they undergo a phase transition from semiconductor to metal[4]
  - This phase transition happens at 3% strain in $MoTe_2$
  - This can be reached at 3V with a channel length of 2.5μm.
- To bend the flake, use an ionic polymer metal composite (IPMC)
  - In an IPMC, the anions are held in place, but the cations are free to move under bias. This causes a mechanical deformation of the material due to electrostatic pressure[5]
  - The strain ε on the plane can be found using the following equations[6], where α is the magnitude of steric strain, β is the electrostatic pressure, and γ is the crowding factor.

$$\varepsilon = \frac{S-L}{L}, \quad S = \frac{\theta}{K}, \quad \theta = cos^{-1}\left(\frac{2R^2-L^2}{2R^2}\right),$$

$$\mathrm{K}(V) = \alpha\sqrt{1-\gamma}\,\frac{V}{\sqrt{1+\frac{V}{8}}} + \frac{2}{3}\beta(1-2\gamma)\sqrt{2(1-\gamma)}\,V^{\frac{3}{2}}$$

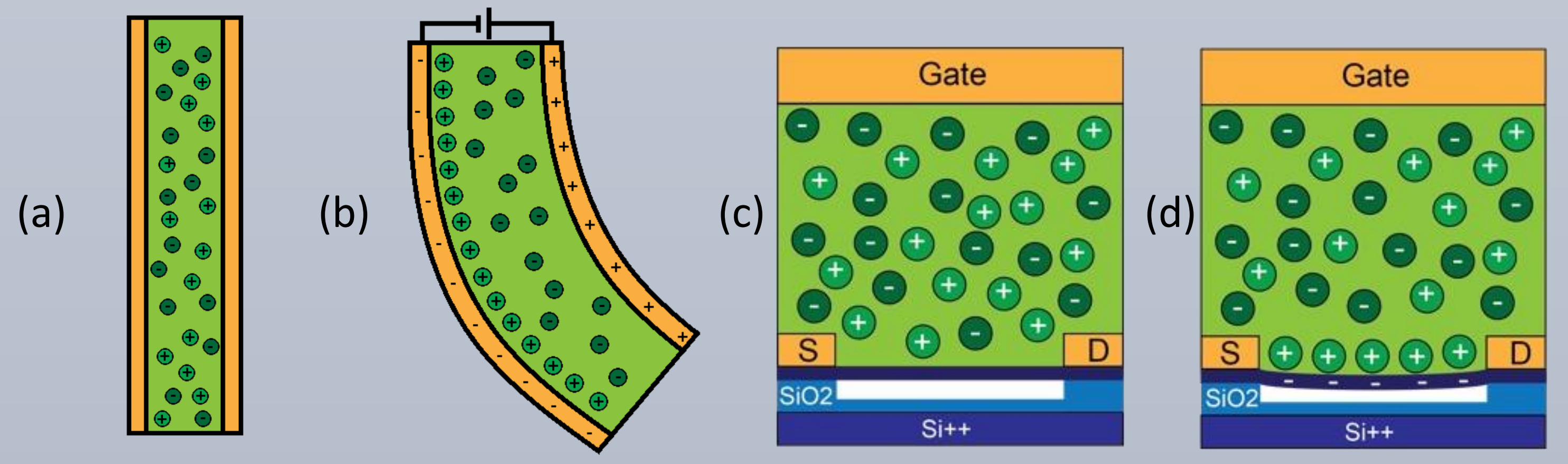

Fig. 5: Proposed mechanics of strain-based device. (a) Schematic of IPMC device with no bias between the contacts. (b) Schematic of the IPMC device with voltage applied. (c) Device with no bias. (d) Device with $V_G > 0$, causing strain on $MoTe_2$

Characterization

- Using AFM to quantify:
  - Lateral size: small flakes (<10um) cannot be used in devices
  - Thickness: New exfoliation methods lead to flake thickness approaching monolayer MoS_2 (0.3nm)
  - Roughness: If surface too rough ($R_Q < 1.5nm$) monolayer electrolyte (CoCRPc) will not lay flat

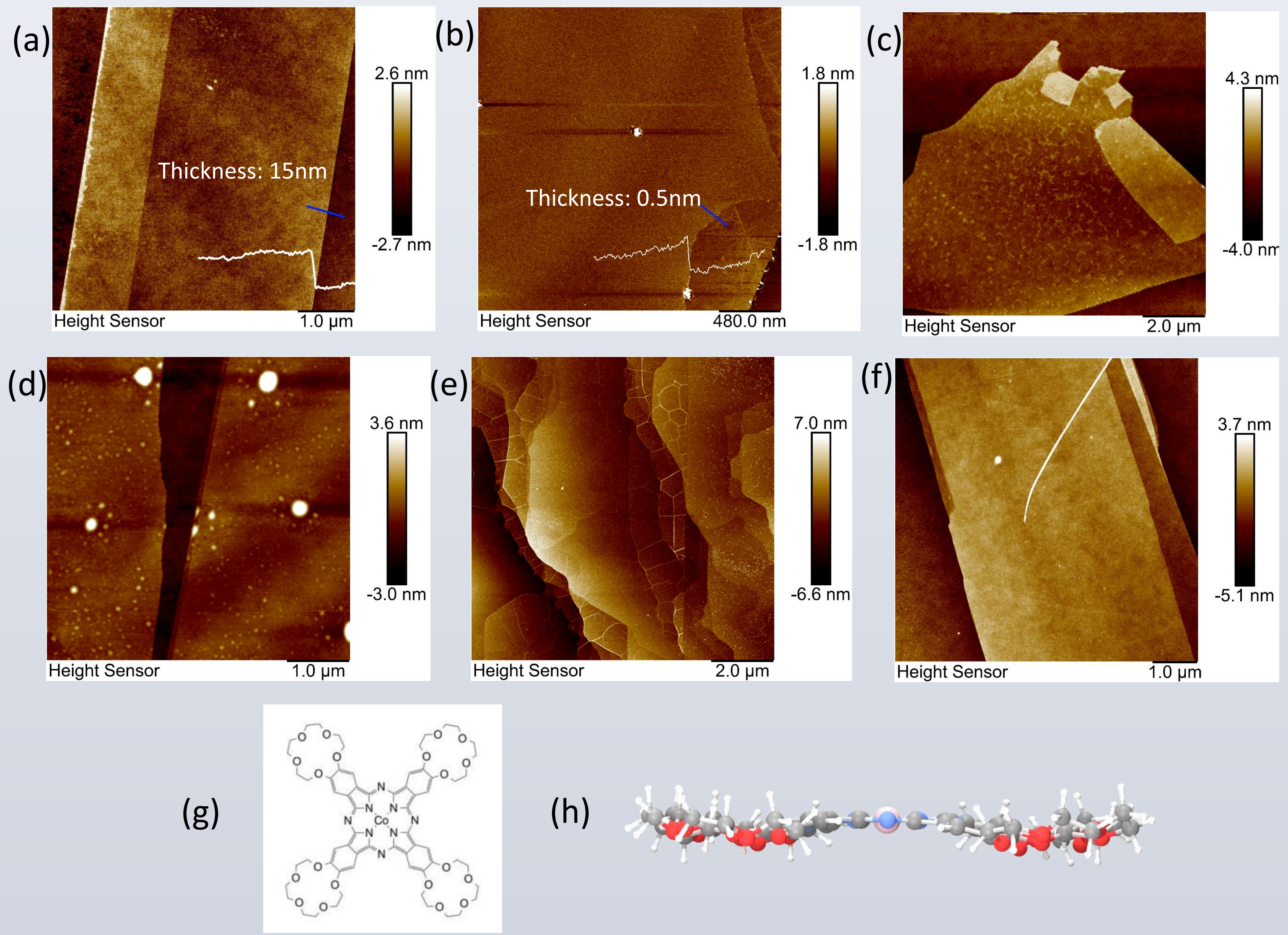

Fig. 4 AFM images of few-layer flakes. (a) Step edge of uncoated MoS_2 flake. (b) Step edge on homogeneous CoCrPc monolayer on exfoliated graphene. (c) Bare boron nitride (BN) flake, used as an insulator. (d) A close-up of a crack in a MoS_2 flake. (e) Epitaxially grown graphene after photoresist removal. (f) Wrinkled MoS_2 flake. (g) Monolayer electrolyte cobalt crown ether phthalocyanine (CoCRPc) top view. (h) Monolayer electrolyte (CoCRPc) side view with Li^+ ions solvated in crown ethers.

Conclusions and Future Work

1. Adding the thermal annealing step during exfoliation allows for more consistent flake thickness and lateral size in MoS_2
2. The difference in exfoliation results between graphene and MoS_2 suggests that each material may need a different process or substrate.
3. Annealing does not leave any residue from the tape on the flakes.

Future work:

- Explore other TMD materials such as tungsten diselenide (WSe_2), and exfoliation methods
- Use C-AFM for measuring electrical properties of flakes/coatings
- Use COMSOL to determine electrical and mechanical properties of devices that undergo strain

References
[1] Radisavljevic, B., et al. *Nature nanotechnology* 6.3 (2011): 147-150.
[2] Fan, Z., et al. *Chin. Phys. B* 24.9 (2015): 097103-1-5
[3] Huang, Y., et al. *ACS nano* 9.11 (2015): 10613-10620
[4] Yun, W., et al. *Physical Review B* 85 (2012): 033305-2-4
[5] Lee, A., et al. *J. Phys.: Condens. Matter* 25 (2013): 082203-1-5
[6] Lee, A., et al. *Soft Matter* 9 (2013): 3762-3773

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