Low Temperature Spin Coating of Two-Dimensional Molybdenum Disulfide

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MOTIVATION OF WORK

- There is an ever-increasing demand for 2D materials such as molybdenum disulfide (MoS_2) for new thin film semiconductor devices.
- Different deposition methods are being investigated to determine the best way to synthesize these materials.
- Spin coating is an effective and economical way to deposit semiconductor thin films onto substrates [1].
- **Research goal:** to utilize spin coating to create thin films of MoS_2 at low temperatures (200°C) for microelectronics.
- **Optimization parameters:** precursor solution, substrate type and pre-treatment, rotation speed, and temperature.

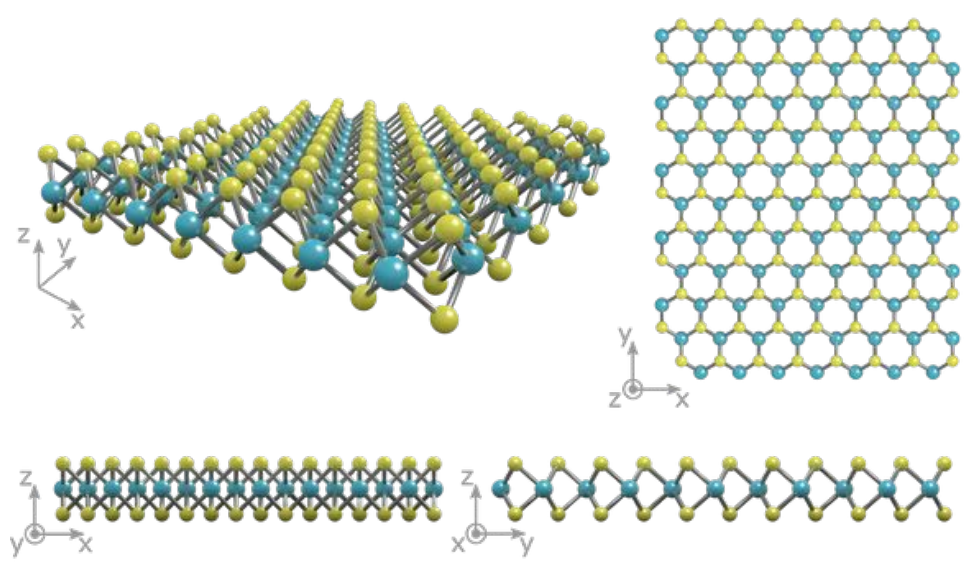


Figure 1: Crystal structure of MoS_2 [2]

BACKGROUND

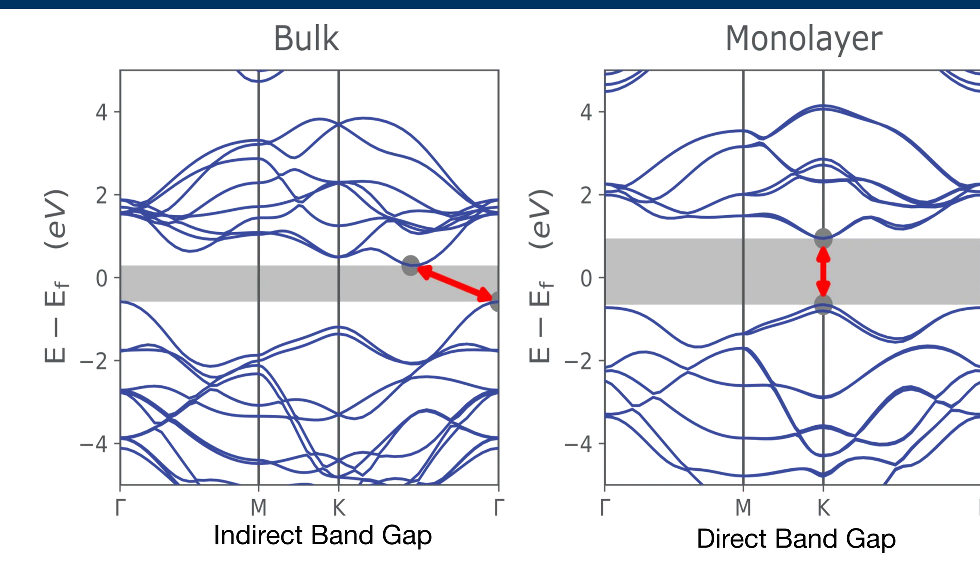


Figure 2: MoS_2 band structure [2]

- 2D materials allow for smaller devices to continue Moore's Law scaling.
- Monolayer 2D MoS_2 has a direct band gap, while multilayer MoS_2 has an indirect band gap [2].
- MoS_2 is made of a layer of molybdenum atoms sandwiched between two layers of sulfur atoms, with weak interlayer van der Waals forces connecting the trilayers [2].

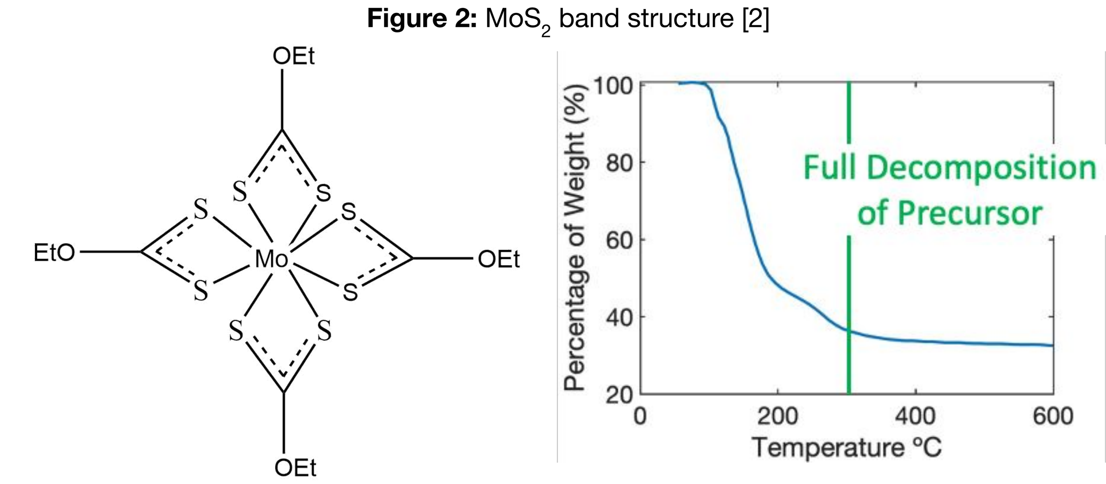


Figure 3: $Mo(S_2COEt)_4$ structure and TGA [3]

- Chemical vapor deposition (CVD) requires high temperatures. Mechanical exfoliation leads to inconsistent films.
- Spin coating involves dropping a precursor solution on the substrate which is then spun at high speeds.
- Previous studies show $\delta \sim \omega^{-1/2}$ [1], where δ is film thickness and ω is rotation speed.
- Xanthate precursors have a low decomposition temperature near our target.

EXPERIMENTAL METHODS

Initial Spin Coating Conditions

- Substrates: Si/SiO_2, sapphire (Al_2O_3), and mica
- Concentrations: 20.7 mM, 13.8 mM, and 10.4 mM in DMF
- Rotation speeds: 500 rpm, 1000 rpm, and 2000 rpm
- Spin coating program for the first 27 samples:
  - 30 seconds acceleration to target speed
  - 60 seconds spin at target speed
  - 60 seconds spin w/ substrate ramp to 200°C
  - 60 seconds spin w/ heating at constant 200°C

Optimized Spin Coating Conditions

- Substrate: Si/SiO_2
- Concentrations: 0.125 M and 0.250 M in DMF
- Rotation speeds: 500 rpm, 1000 rpm, and 1500 rpm
- Spin coating program:
  - 5 seconds ramp to target speed
  - 30 seconds at target speed, heat to 60 °C
  - 2 minutes annealing at 200 °C, no spinning

Characterization

- Optical microscopy, optical profilometry, atomic force microscopy (AFM) , and Raman spectroscopy

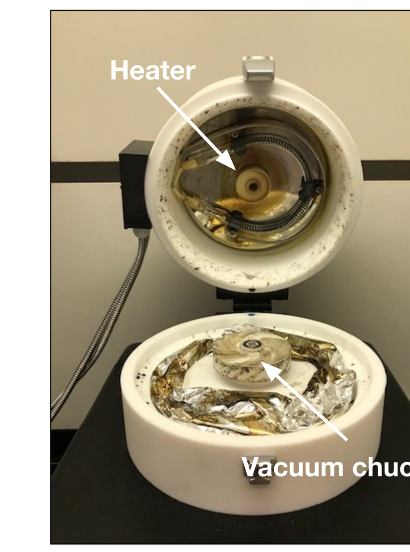


Figure 4: MTI Spin Coater

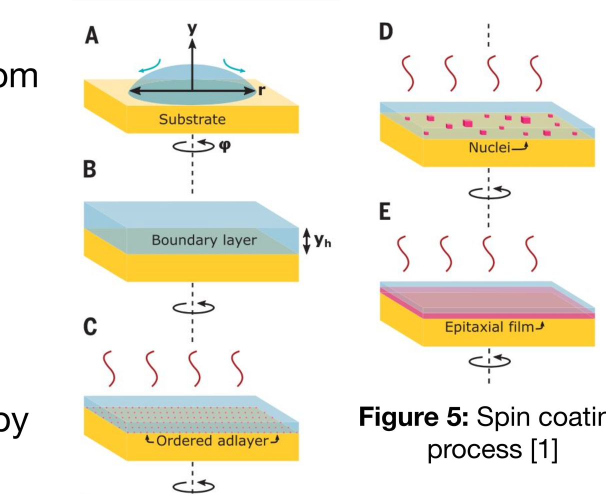


Figure 5: Spin coating process [1]

RESULTS: FILM THICKNESS

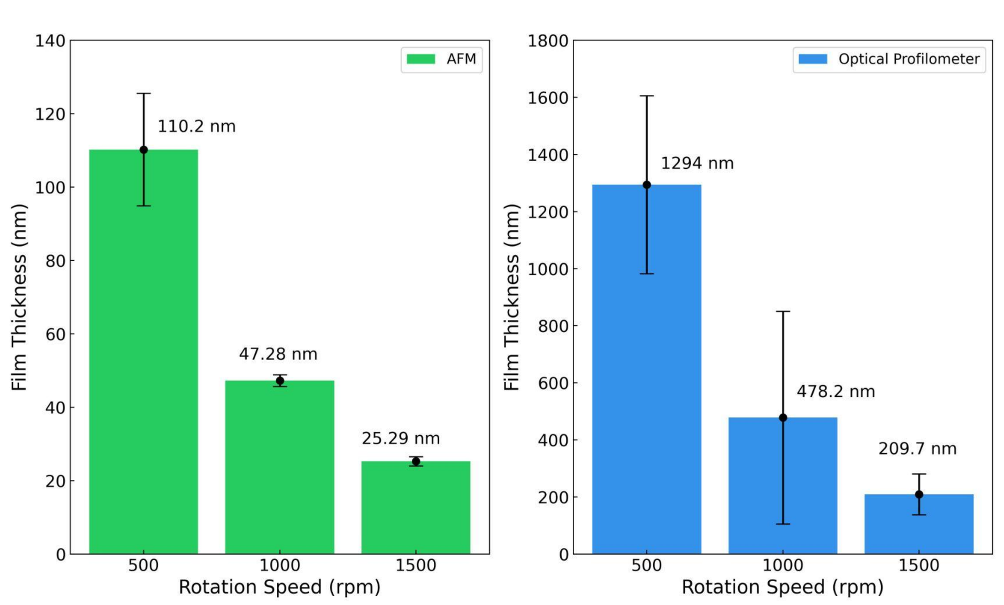


Figure 6: Rotation speed vs. scratch thickness graph

- Centrifugal force from rotation causes the precursor solution to spread out and evenly cover the substrate.
- We observed the film becoming thinner as the rotation speed increased, agreeing with prior work.

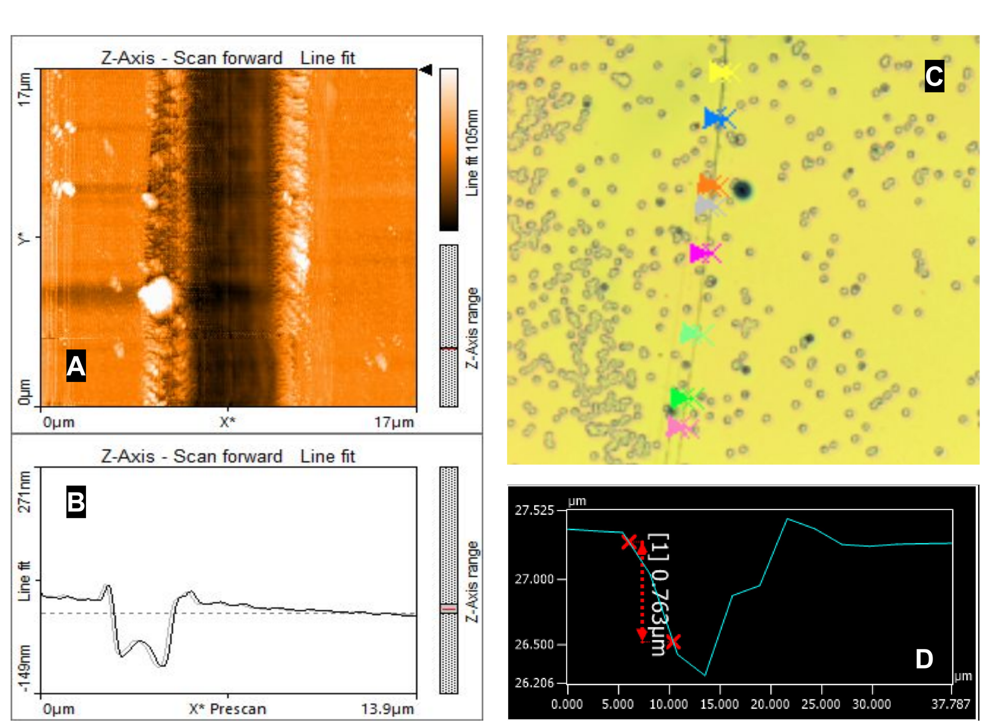
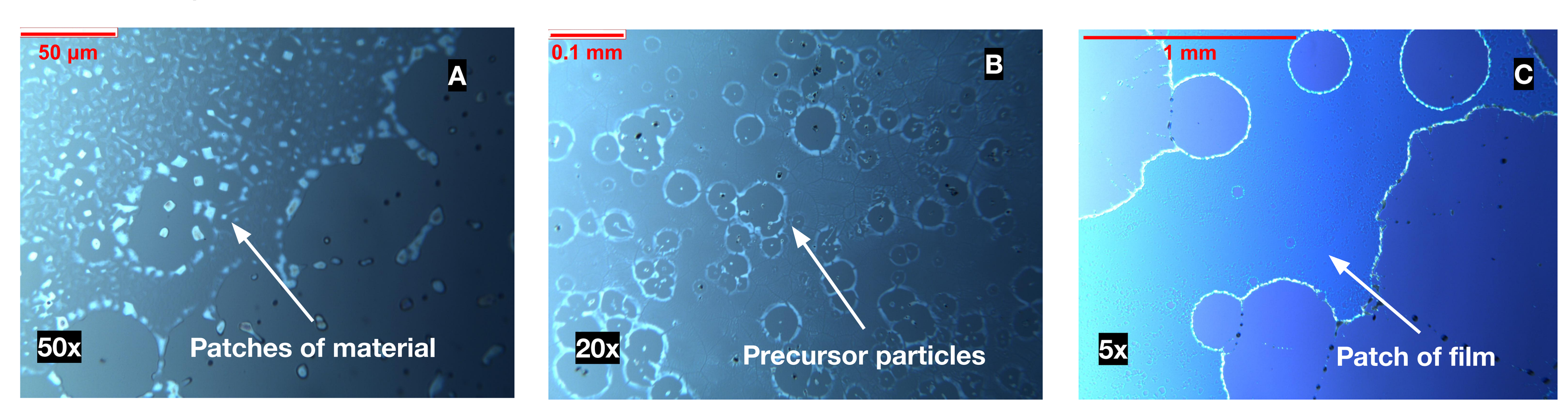


Figure 7A: AFM image during profile measurement
Figure 7B: AFM profile measurement
Figure 7C: Optical profilometer image during profile measurement
Figure 7D: Optical profilometer measurement

RESULTS: MORPHOLOGY CONTROL AND CONVERSION OF FILM TO MoS_2

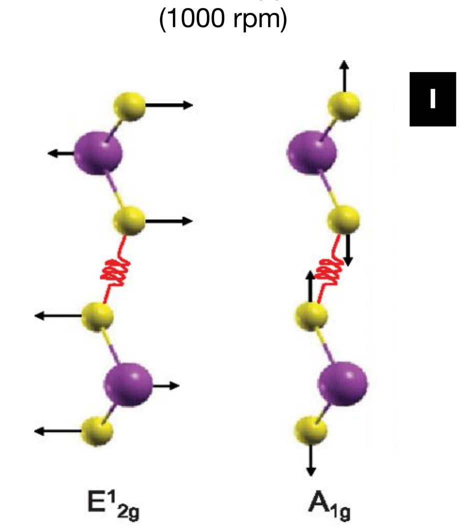
Initial Samples

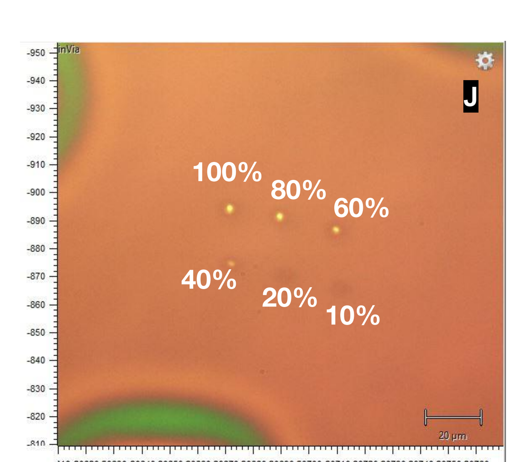


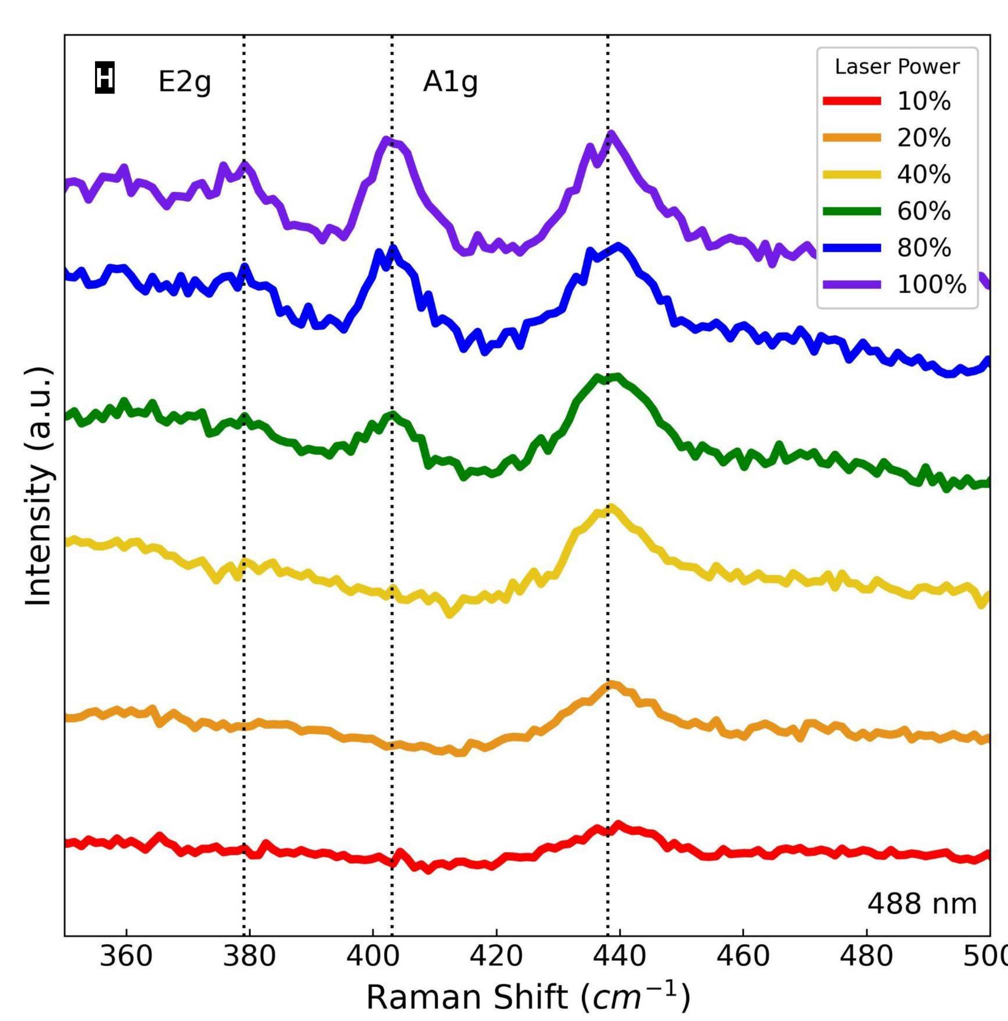
20.7 mM Sapphire (1000 rpm)

20.7 mM Mica (1000 rpm)

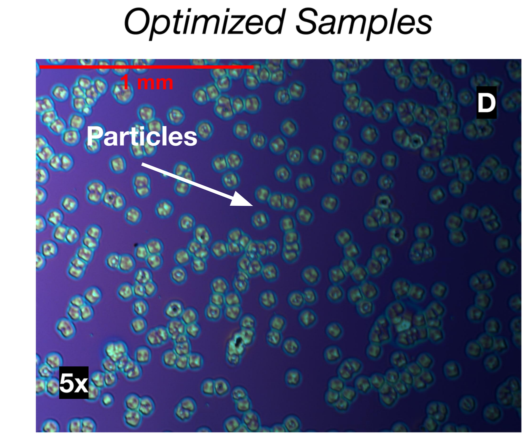
20.7 mM Si/SiO_2 (1000 rpm)



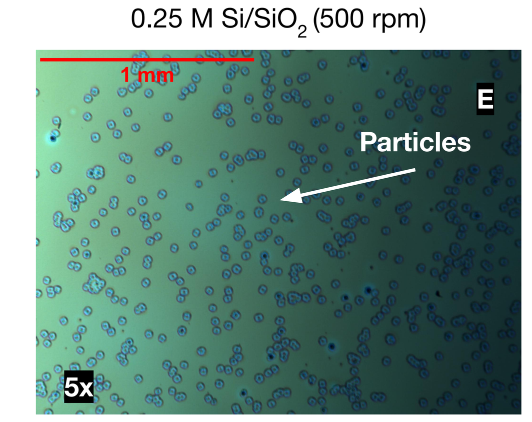




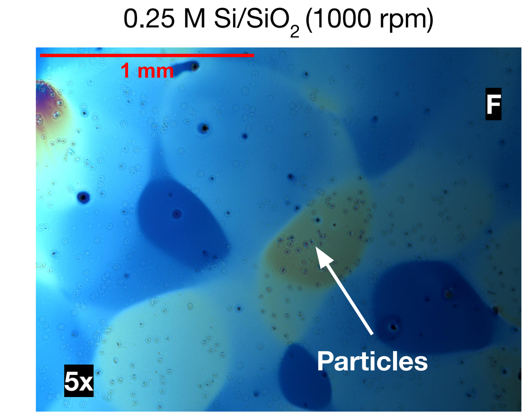
Optimized Samples



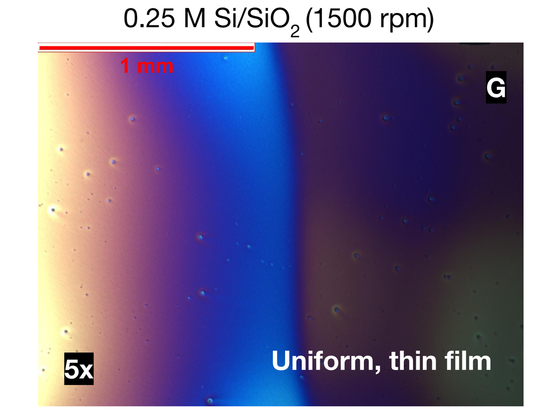
0.25 M Si/SiO_2 (500 rpm)



0.25 M Si/SiO_2 (1000 rpm)



0.25 M Si/SiO_2 (1500 rpm)



Ultrasonicated 0.25 M Si/SiO_2 (500 rpm)

Figures 8A-8G: Optical microscope images of initial and optimized samples
Figure 8H: Raman data showing conversion to MoS_2 starting at 60% power
Figure 8I: MoS_2 vibration modes [4]
Figure 8J: Raman laser conversion and etching

SUMMARY

- Developed initial samples with three different substrates, rotation speeds, and precursor solution concentrations.
- Observed that a higher concentration precursor solution was needed, that the most promising substrate was Si/SiO_2, and that lower rotation speeds mean thicker films.
- Optimized samples had uniform, thin films of what is most likely MoS_3, an amorphous semiconductor that is related to MoS_2.
- Conversion to MoS_2 is observed during heating from the Raman laser at 488 nm.
- Future steps: measure electrical and structural properties of samples, and observe the effectiveness of UV light conversion to MoS_2.

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