# Synthesis of 2D Transition Metal Oxides

Robert Michael Loh, Material Science and Engineering
Mentor: Prof. Qing Hua Wang , Suneet Kale
School for Engineering of Matter, Transport and Energy

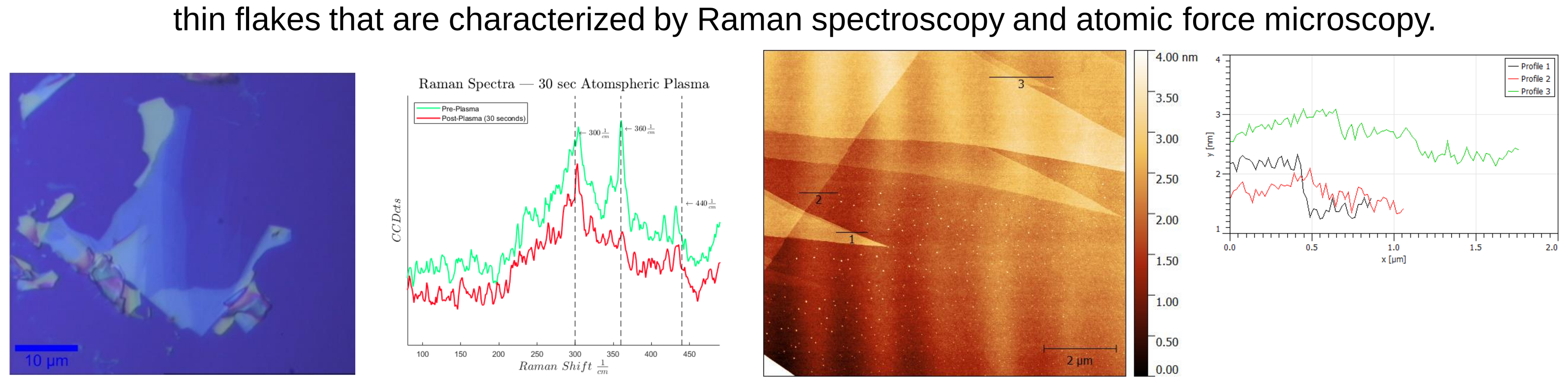
## Abstract


- The goal of this FURI project is to synthesize thin film transition metal oxides. Metal oxides have known bulk properties but some have not been synthesized in a thin film forms. Materials take on vastly different properties when they are on the nano-scale.
- Nanomaterials have been utilized in many ways such as batteries, insulation, medicine, transportation, and optics[1].
- The hypothesis of this project is that thin film transition metal oxides of germanium dioxide ($GeO_2$), hafnium dioxide ($HfO_2$), and palladium dioxide ($PdO_2$) can be synthesized based on previous research by the Wang research group[2]. Their results show that molybdenum trioxide ($MoO_3$) and tungsten trioxide ($WO_3$) can be synthesized from their respective sulfides $MoS_2$ and $WS_2$ by atmospheric plasma treatment in the form of thin-flake nanomaterials.


## First Semester Results

### Synthesis of $GeS_2$ Flakes

- Referencing an article on material synthesis of thin flakes[3] mechanical exfoliation of $GeS_2$ was found to be most favorable by plasma treating substrates to be hydrophilic and exposing samples to 100 ºC heat for one minute before cleaving. This produced thin flakes that are characterized by Raman spectroscopy and atomic force microscopy.

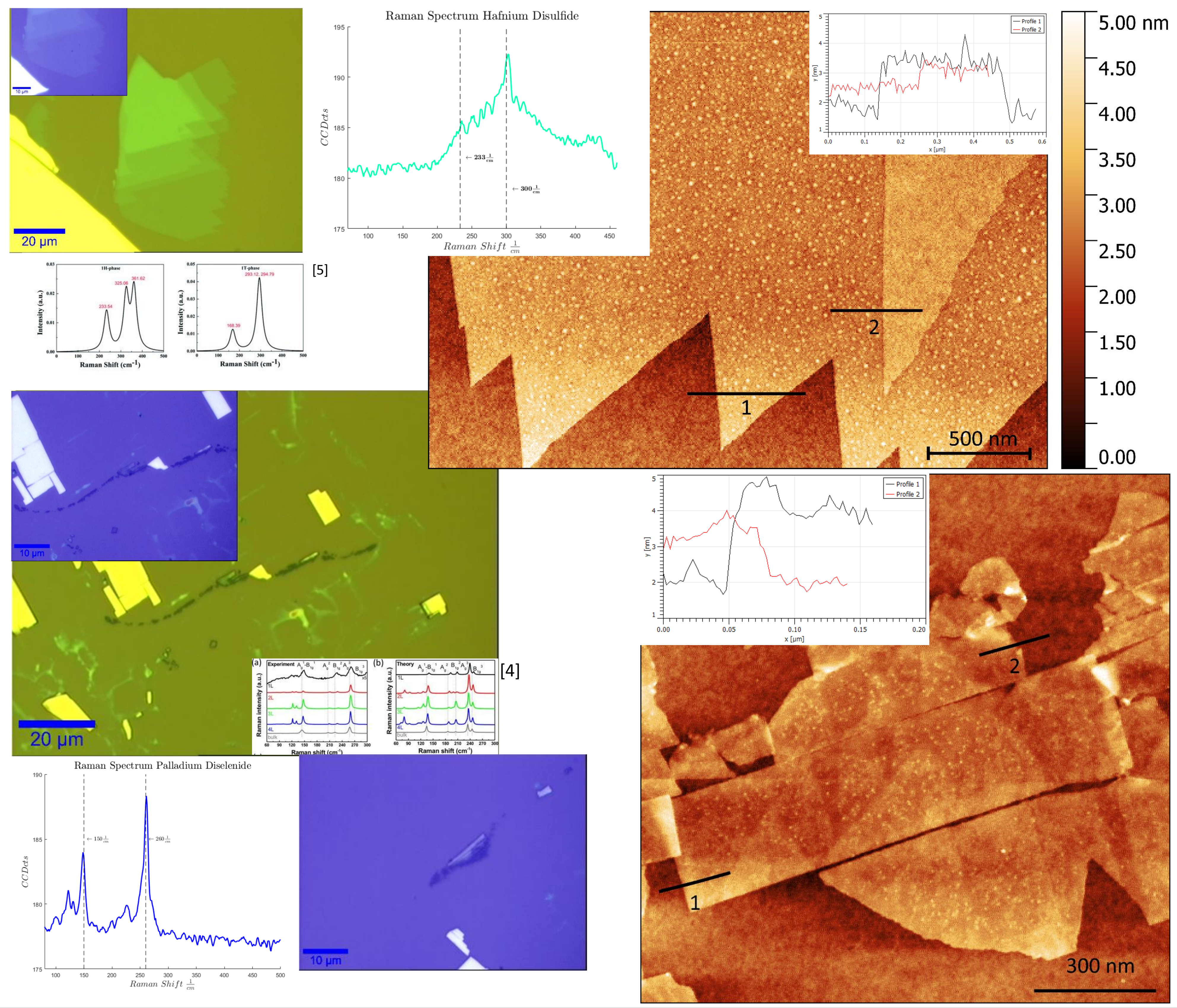



## Current Research

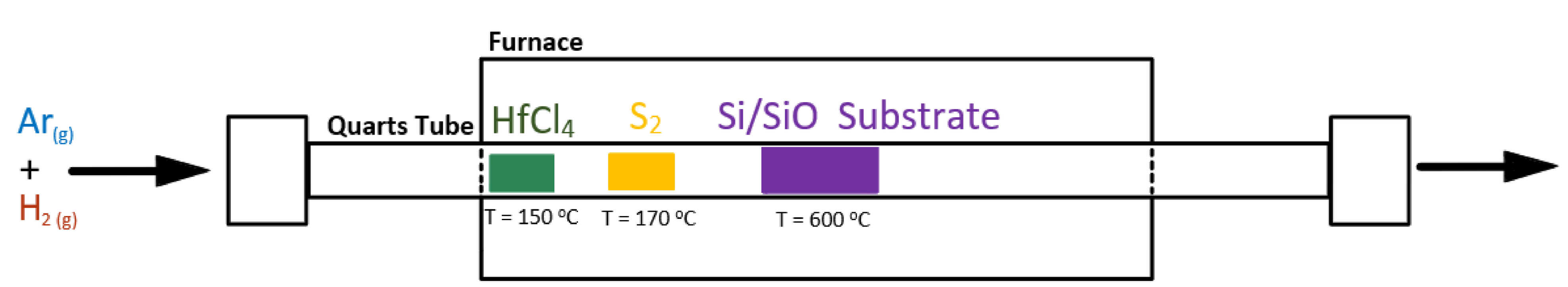
### Synthesis of $HfS_2$ and $PdSe_2$ Flakes

- Using the same synthesis techniques for mechanical exfoliation of $HfS_2$ and $PdSe_2$ was found to yield the largest area and highest-quality flakes by exposing pre-exfoliated samples to 100 ºC hot plate for one minute and cleaving while the substrates were hot.
- Thin flakes are characterized by Raman spectroscopy and atomic force microscopy.
- Complications arose with both $HfS_2$ and $PdSe_2$
- $HfS_2$ is very unstable in air as a thin film. Within one-hour, thin film samples found and characterized with Raman were disintegrated by the time atomic force microscopy (AFM) was performed.
- Chemical vapor deposition (CVD) could lead to less reactive $HfS_2$ 2D flakes.
- Initial $HfS_2$ CVD results indicate possible vertical morphology that is being investigated.
- $PdSe_2$ has yielded small single or double layer 1-micron by 1-micron flakes.
- High quality exfoliated $PdSe_2$ flakes are too small to characterize with Raman spectroscopy but have been imaged using AFM.

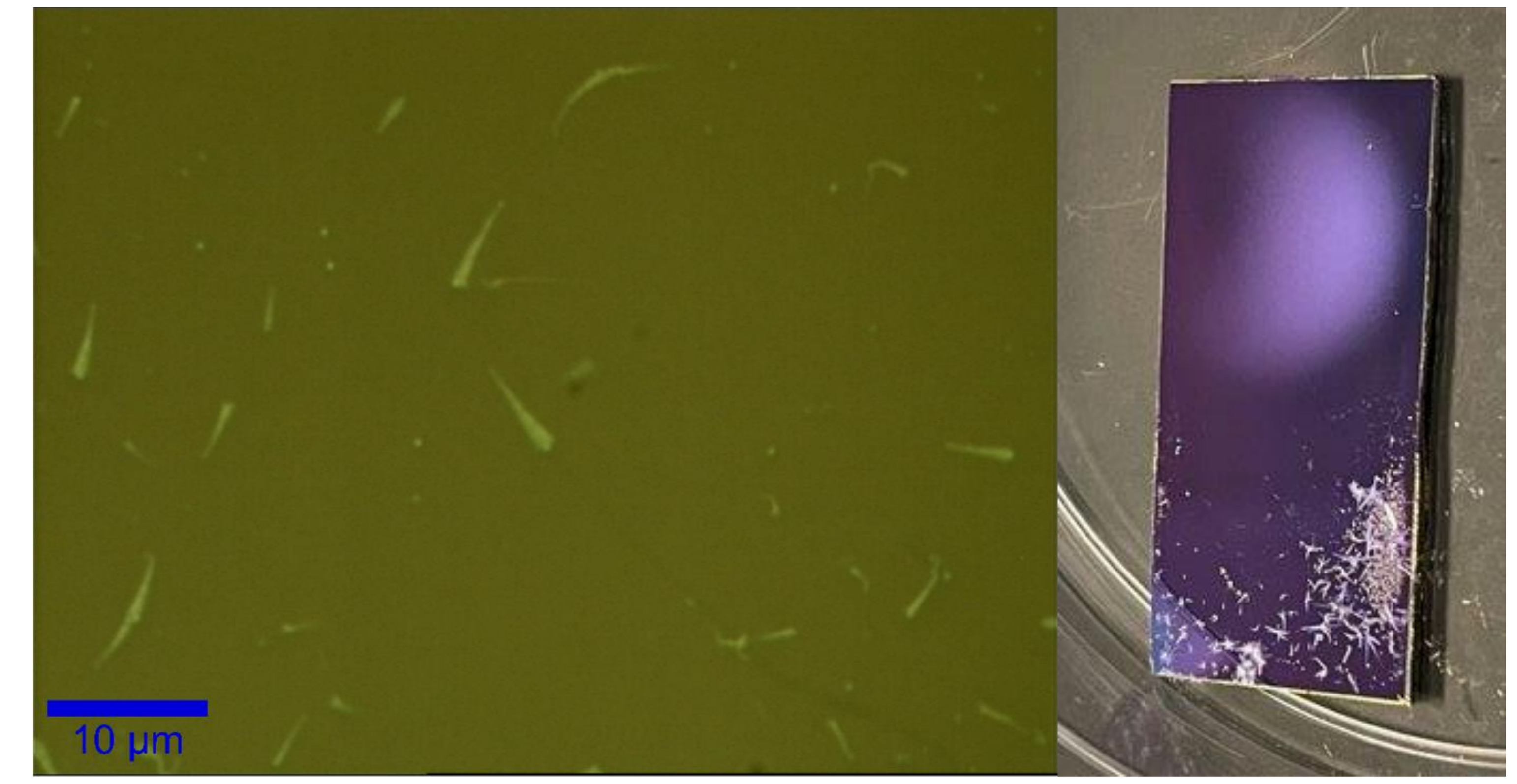
## Exfoliation of $HfS_2$ and $PdSe_2$




## Chemical Vapor Deposition (CVD) $HfS_2$




## CVD Synthesis Steps $HfS_2$




### Synthesis of $HfS_2$ via Chemical Vapor Deposition

- Hafnium disulfide was also made using a method called chemical vapor deposition. A mixture of argon ( 50 sccm) and hydrogen (15 sccm) gas are passed into a tube furnace made of quarts at ambient pressure. They carry vaporized hafnium chloride (24 mg) and sulfur (196.9 mg) into the center of the chamber where the substrate is heated to 600 ºC. A chemical reaction takes place that results in deposited hafnium sulfide on the surface. The outcome was possibly vertically growing thin flakes but more experimentation is required to achieve usable thin flakes. This growth process is being refined to produce thin flakes suitable for air plasma treatment.

## Future Work

### Plasma Treatment of 2D Transition Metal Dichalcogenides

- Thin flakes of $HfS_2$ and $PdSe_2$ are nearing a level of quality and size to be treated with air plasma to test the hypothesized method for synthesizing transition metal oxides. Once these ideal flakes are achieved the methods used from the first semester on $GeS_2$ will be used on $HfS_2$ and $PdSe_2$.
- Air plasma treatments to 2D transition metal dichalcogenides are hypothesized to create 2D transition metal oxides.

## References


[1] Nano Technology Initiative 2000, accessed September 26th, 2020, <https://www.nano.gov/you/nanotechnology-benefits>
[2] Chu, X. S., Li, D. O., Green, A. A., Wang, Q. H. (2017). Formation of $MoO_3$ and $WO_3$ nanoscrolls from $MoS_2$ and $WS_2$ with atmospheric air plasma. *Journal of Materials Chemistry C., Vol.(5)* pg 11301 - 11309. DOI: 10.1039/c7tc02867a
[3] Yuan, Huang, et al, (2015). Reliable Exfoliation of Lare-Area High-Quality Flakes of Graphene and Other Two-Dimensional Materials. Journal of ACS NANO, Vol. (9), NO. 11, 10612–10620.
[4] Oyedele, Akinola D., et al. *Journal of the American Chemical Society* (2017)
[5] Singh, Deobrat, et al. "2D-HfS 2 as an efficient photocatalyst for water splitting." *Catalysis Science & Technology* 6.17 (2016): 6605-6614.