Introduction

2D materials, only a few atoms thick, have gained scientific attention in the past few years for their excellent electronic, mechanical, and optical properties. One such material is tungsten diselenide (WSe₂), a transition metal dichalcogenide that takes on a hexagonal crystalline shape. 2D WSe₂ has a direct band gap, which gives it an advantage over traditional semiconductors like silicon. It is important to gain a deeper understanding of the properties of WSe₂ due to the material's potential applications in flexible electronics and optoelectronics [1]. Prior research shows that WSe₂ grows under a certain range of conditions, but those conditions vary per study [2]. In order to further understand the properties of WSe₂, we established our own parameters that aid in the process of optimizing both the size and quality of CVD grown grains.

Methods

We used chemical vapor deposition (CVD) to grow WSe₂. In our setup, we placed a glass tube inside a furnace, which was connected to smaller plastic tubes on both ends. The tubes on the left side were connected to hydrogen and argon gas, which blew Se particles towards WO₃ and carried away gaseous by-products. The tubes on the right side were connected to a container filled with mineral oil to prevent gaseous backflow, which in turn was connected to another tube through which the gases were removed. A mixture of WO₃ powder and sodium chloride was placed in a alumina boat in the center of the furnace. A boat of selenium sits slightly upstream and at a lower temperature. A SiO₂ wafer, cleaned by chemicals and ozone, was placed on top of the boat filled with WO₃. After the furnace reached the preset temperature at a predetermined rate, we waited a few minutes before cooling the furnace down. After we removed the silicon wafer from the furnace, we observed it under an optical microscope. If there was substantial growth, we observed the wafer using Raman spectroscopy to confirm the identity and thickness of the WSe₂. To further confirm our results, we consulted the graph obtained from photosimulcience (PL) spectroscopy. Finally, we took detailed notes on the quality and size of WSe₂ growth under each experimental condition.

Furnace Diagram

![Furnace Diagram](image)

Figure 2: The placement of the wafer and both alumina boats inside the glass tube, along with the direction of gas flow

Optimization of Monolayer WSe₂ Growth

Using Chemical Vapor Deposition

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Results

We ran experiments that tested the parameters of WSe₂ growth, including gas flow rate, rate at which the temperature increased, amount of starting material, and the amount of time the furnace is held at the desired temperature. Through multiple trials, we found that WSe₂ grows best within a temperature range of 700-800 °C. The optimal amount of starting material should be less than 1 grams, with the weight of the WO₃/NaCl mixture around 0.1 g. We found that the amount of selenium does not have a noticeable impact on the quality of the growth, but that too much will result in it accumulating inside the boat. The flow rates of the gases were best at a hydrogen flow rate of 20 sccm and an argon flow rate of 80 sccm. The temperature in the furnace was increased at a rate of 50 °C/min, and kept at the desired temperature for 5 minutes. We observe that white areas on the grains are consistently present. These results produced hexagonal WSe₂ grains up to 40 micrometers in length. The identity of the growth was confirmed by Raman and PL spectroscopy.

Images of WSe₂ Under an Optical Microscope

![Images of WSe₂ Under an Optical Microscope](image)

Figure 3: (a) Monolayer WSe₂ with lighter sections in the center and edges. The magnification of this, along with the other images, are 6x. The triangular growths are multilayer WSe₂. (b) Larger hexagonal growth with thicker flakes towards the center of the flakes; used a 0.1 ratio of WO₃ to NaCl. (c) Smaller WSe₂ growth that is a couple of layers thick; used a 0.3 ratio of WO₃ to NaCl.

Raman and Photoluminescence Spectrum of Monolayer WSe₂

![Raman and Photoluminescence Spectrum of Monolayer WSe₂](image)

Figure 4: (a) Image of a WSe₂ flake under the Raman microscope. The sides of the hexagonal growth are slightly longer than 20 micrometers. (b) Raman spectrum of WSe₂ that is 0.2-layer thick. (c) Lattice vibrations that cause the peak at 250 cm⁻¹. (d) PL spectrum of WSe₂. The graph matches what is expected of monolayer WSe₂, with Judd-Ofelt 1. (e) Judd-Ofelt 2. (f) Judd-Ofelt 3.

We conducted other experiments on, such as increasing the amount of starting material, reaching the preset temperature at a predetermined rate, the time that the sample is held at a predetermined temperature, rate at which the temperature increased, amount of starting material, and the amount of time the furnace is held at the desired temperature. Through multiple trials, we found that WSe₂ grows best within a temperature range of 700-800 °C. The optimal amount of starting material should be less than 1 grams, with the weight of the WO₃/NaCl mixture around 0.1 g. We found that the amount of selenium does not have a noticeable impact on the quality of the growth, but that too much will result in it accumulating inside the boat. The flow rates of the gases were best at a hydrogen flow rate of 20 sccm and an argon flow rate of 80 sccm. The temperature in the furnace was increased at a rate of 50 °C/min, and kept at the desired temperature for 5 minutes. We observe that white areas on the grains are consistently present. These results produced hexagonal WSe₂ grains up to 40 micrometers in length. The identity of the growth was confirmed by Raman and PL spectroscopy.

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Discussion

Our data shows that multiple factors have an impact on the size and quality of CVD grown WSe₂. A change in one parameter affects the quality of WSe₂ growth in different ways. For instance, the amount of starting precursors (WO₃ and Se) affect the thickness of the growth. When there is not too much starting material, there is often a thick layer of WSe₂ on the wafer, which is reflected in its Raman graph. To gain monolayer growth, we either reused the powder, or used less powder to begin with. Without the salt, which lowers the melting point of WO₃, there was either little or no growth using the temperatures we tested (550 - 850 °C). Using temperatures outside our optimal range, the WSe₂ growth looked incomplete. We did not run enough trials to ascertain how the gas flow rate, the amount of starting material, and the amount of time the furnace is held at the desired temperature affects the quality of WSe₂ growth.

Conclusions

Over the course of six weeks, we sought to grow large, thin, and pure grains of WSe₂. Through experimenting with the many parameters that affect WSe₂ growth, we created grains that we could conduct other experiments on, such as straining the monolayers and observing changes in its mechanical properties. Although we were able to achieve quality WSe₂ growth using our conditions, more experimentation needs to be done to fine tune the process and make the results more consistent. Our research can lead to improved transistors, solar cells, and flexible electronics. [8, 9]

References


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