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Growth and Characterization of Tungsten Disulfide Monolayer Using Solution Precursor-Assisted Chemical Vapor Deposition Method

Maral Boomipour[1]*, Ali Khatibi[2], Babak Shokri[3]

[1] Department of Physics, Faculty of Science, Shahid Beheshti University, Tehran, Iran
[2] Laser and Plasma Research Institute, Shahid Beheshti University, Tehran, Iran
[3] Department of Physics, Faculty of Science, Shahid Beheshti University, Tehran, Iran.

***Corresponding Author:**

Boomipour.m@gmail.com

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ABSTRACT

Transition metal dichalcogenides (TMDs) have attracted significant attention due to their unique optical and electronic properties, positioning them as promising candidates for various electronic and optoelectronic devices such as sensors, transistors, and batteries. However, the synthesis of these materials has always posed challenges, particularly in terms of achieving uniformity and controlling the precursor vapor during the synthesis procedure. In this study, we addressed these challenges by utilizing a soluble precursor. This approach enabled us to grow high-quality, monolayer tungsten disulfide flakes on SiO2/Si substrates using the chemical vapor deposition (CVD) method. By examining and adjusting various growth parameters such as growth temperature, solution precursor concentration, and substrate position, we successfully optimized the growth conditions. To characterize the monolayer flakes, we employed Raman and photoluminescence (PL) spectroscopies.

Keywords: transition metal dichalcogenides, chemical vapor deposition, tungsten disulfide

Introduction

Attracting attention to two-dimensional layered materials with unique physical and chemical properties began with the discovery of exfoliated graphene in 2004 [1]. Graphene, a semimetal with a zero bandgap, consists of a single layer of carbon atoms. However, the lack of an energy band gap limits the use of graphene in semiconductor applications. Therefore, there is a need to investigate two-dimensional materials with semiconducting properties [2]. Two-dimensional layered Transition metal dichalcogenides (TMDs) exhibit various properties including semimetal, insulator, semiconducting, and even superconductor properties, depending on their crystalline structure. Semiconducting TMDs are particularly promising candidates for future applications in energy storage, sensing, optoelectronic, and electronic devices [3]. One exciting feature of these materials is the ability to change the indirect energy band gap, which is present in few-layer structures, to a direct band gap in the monolayer form.[4]

The practical application of these materials in various fields requires their homogenous and high-quality growth. Several techniques are available for synthesizing these materials, and one of the most popular and promising methods is chemical vapor deposition (CVD).

CVD method enables the epitaxial growth of high-quality single layers of these materials. In this approach, the precursors, typically in solid powder form, are heated and vaporized in different temperature zones of a quartz tube. Subsequently, the vapor of these precursors is transported to the surface of the substrate by the carrier gas. During this epitaxial process, solid precursor (transition metals like Tungsten) and gas precursor (chalcogenides like Sulfur) diffuse and react on the substrate surface and form crystal structures. the solid metal precursor utilized in CVD encounters the challenge of the accumulation and uneven distribution

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of this precursor on the substrate's surface, resulting in the growth of multiple layers of these materials.

In this study, we utilized a solution precursor to avoid the aggregation of tungsten precursor and enhance the enlargement of the grown flakes. The liquid precursor exhibits greater mobility on the substrate surface, resulting in a uniform distribution of the precursor across the surface.

furthermore, we demonstrated a systematic control of monolayer WS_2 growth under different growth conditions. By optimizing growth temperature, solution precursor concentration, and the distance between the sulfur precursor and the growth substrate, we successfully achieved high-quality WS_2 monolayers confirmed by optical microscopy as well as Raman and PL spectroscopies.

Result and Discussion

Synthesis of monolayer WS_2

Firstly, SiO_2/Si substrates were ultrasonically cleaned in Acetone, Isopropyl alcohol (IPA), and Distilled water for 5 minutes to remove all adsorbed contaminants. Subsequently, we treated the clean substrates with O_2 plasma for 2 minutes to enhance hydrophilicity. Then, we spin-coated the SiO_2/Si substrate with the Na_2WO_4 aqueous solution. For the sulfur precursor, we used 150 mg of sulfur powder.

Afterward, the spin-coated SiO_2/Si substrates and sulfur precursor were inserted into the center of a 2-inch CVD furnace. The growth process by this method is greatly influenced by the distance between the sulfur powder and the substrates. This is because when the sulfur precursor powder is closer to the furnace's heating zone, its evaporation rate increases, leading to a decrease in the growth time. Conversely, increasing the distance between the sulfur powder and the furnace's heating zone decreases the evaporation rate and extends the growth time. Both of these factors are crucial in the growth of WS_2 flakes.

In this study, to ensure the stability of these variables and investigate additional influential factors in the growth process, the amount of sulfur powder remains consistently at 150 mg and is positioned 13 cm away from the substrates and the temperature zone of the furnace. The synthesis was carried out under ambient pressure, with argon gas flowing at a rate of 150 SCCM as a carrier gas to deliver the sulfur source to the spin-coated substrates. Prior to heating the setup, the quartz tube was purged with Argon gas for 20 minutes. The CVD furnace configuration used in this study is illustrated in Figure 1. a.

The heating procedure shown in Figure 1. b demonstrates that the temperature zone was initially raised to 200 degrees in 10 minutes. It was then maintained at this temperature for 5 minutes to allow the water vapor and pollution on the substrate's surface and inside the growth environment to evaporate, leading to a better-quality growth process. Subsequently, the temperature gradually increased to 850 degrees in 24 minutes, which is the optimal temperature for WS_2 growth. The tube was then maintained at 850 degrees until all the sulfur powder evaporated, a process that typically lasts for approximately 16 to 20 minutes, referred to as the growth time. Finally, the tube was cooled back down to room temperature.

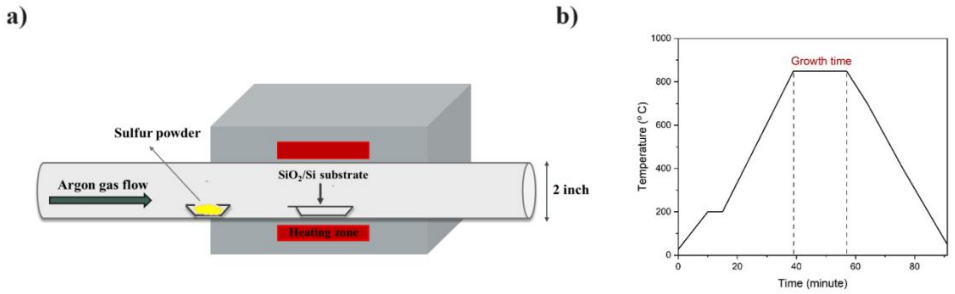


Figure 1. a. *Schematic illustration of the CVD setup,* **b.** *heating procedure for the growth of monolayer WS_2 flakes.*

Solution precursor concentration

In this section, we will investigate the impact of solution precursor concentration on the growth process. We created four different solutions with varying concentrations to observe how they affect the samples being grown. Specifically, we dissolved 16.5, 66, 132, and 198 mg of sodium tungstate ($Na_2WO_4.2H_2O$) in 10 mL of distilled water to obtain Na_2WO_4 aqueous solutions with concentrations of 5 mM, 20 mM, 40 mM, and 60 mM respectively. Next, we subjected all the solutions to ultrasonic sonication for 15 minutes, followed by magnetic stirring for an additional 15 minutes. Finally, we performed another round of ultrasonic sonication for 15 minutes to ensure complete mixing and achieve a completely uniform and homogeneous solution. As for the precursor solutions with different concentrations, we spin-coated them onto the SiO_2/Si growth substrate at a speed of 2500 rpm for a duration of 2 minutes.

Figure 2 displays the optical images of WS_2, which were prepared using various Na_2WO_4 precursor

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concentrations, ranging from 5 to 60 Mm. The growth temperature was set at 850 degrees. In terms of location, the substrate was positioned as the second one on the alumina boat. In the case of a 5 mM concentration, no flake was observed on the substrate. However, with a 20 mM concentration, a higher nucleation density was achieved, resulting in the formation of monolayer WS_2 flakes with large domains.

As the concentration of Na_2WO_4 increases to 40 and 60 mM, we can observe the formation of multilayer structures and asymmetric flakes. One crucial factor that influences the growth and thickness of the resulting WS_2 structure is the ratio between the two precursors, tungsten and sulfur. In the case of a 5mM solution, the lack of tungsten precursor hinders the nucleation process and prevents the successful synthesis. On the other hand, a concentration of 20 mM is considered the optimal concentration for the tungsten precursor, leading to the formation of uniform monolayer WS_2 flakes. *Additionally, the formation of multilayers occurs when the tungsten precursor becomes saturated at concentrations of 40 and 60 mM.*

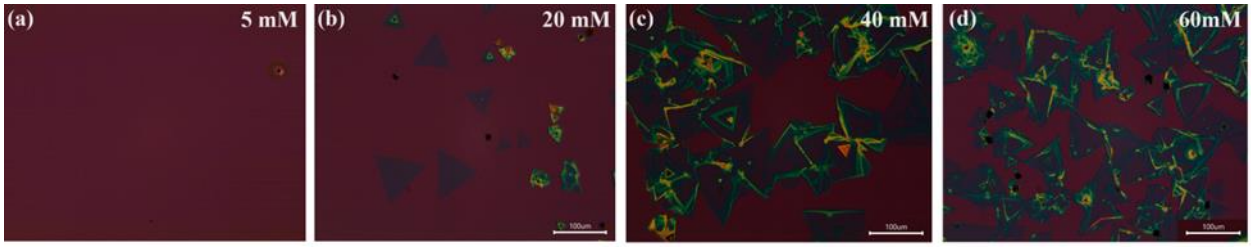


Figure 2. *Optical Microscopy images of WS_2 samples grown on SiO_2/Si by* **a.** *5 mM,* **b.** *20 mM,* **c.** *40 mM and* **d.** *60 mM Na_2WO_4 precursor concentrations.*

Growth Temperature

In order to investigate the impact of growth temperature on the grown samples, We performed the same experiment at three different growth temperatures. The concentration of the Na_2WO_4 precursor was set at 20 mM, and the substrate was positioned as the second one on the alumina boat. The appropriate temperature for growth ensures the even distribution of the soluble precursor across the substrate's surface and influences the structures that form on the substrate. Furthermore, in our approach, the temperature that is applied to the sulfur powder also depends on the growth temperature that is applied to the furnace. This implies that as the temperature inside the furnace increases, the temperature applied to the sulfur powder also increases, leading to an increase in its evaporation rate, which directly influences the growth process and the resulting sample.

Figure 3 displays the WS_2 samples obtained at varying growth temperatures of 700, 850, and 1000 degrees. By utilizing a growth temperature of 700 degrees, we will observe the formation of circular structures exclusively on the surface of the substrate. These circular structures represent tungsten precursors that have formed on the substrate. However, due to the low growth temperature, they were unable to disperse evenly and efficiently across the surface, thus hindering the growth process. While observing the sample that was grown at a temperature of 850 degrees, we observed WS_2 flakes with the desired size and quality. This observation suggests that the optimal temperature for growth is indeed 850 degrees. By analyzing the sample grown at a temperature of 1000 degrees, it appears that the growth temperature of 1000 degrees for this material is greater than the ideal condition. This is because, based on the morphology, it appears that WS_2 flakes are created at lower temperatures. Furthermore, as the temperature continues to rise, these formations are dismantled.



Figure 3. *Optical Microscopy images of WS_2 samples grown on SiO_2/Si substrate at* **a.** *700 °C;* **b.** *850 °C and* **c.** *1000 °C growth temperature.*

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Substrate position

A crucial parameter in both the growth process and the resulting material structures is the distance between the substrate and the source of the sulfur precursor. In previous experiments, the position of the substrate on the alumina boat was considered fixed while other parameters were altered. However, in this particular investigation, we have placed four substrates in different locations on the alumina boat, as shown in Figure 4. a. As a result of changing the substrate positions, their distance from the sulfur precursor also varies.

Analyzing images of the samples grown in the four specified positions (Figure 4 b-e) reveals that the distance between the substrate and the sulfur source has an impact on both the quantity and size of the flakes. The samples obtained from the substrate located closest to the sulfur precursor indicate the existence of multiple small-size WS_2 flakes. By relocating, specifically in positions 2 and 3, we will observe the development of WS_2 flakes with a larger size. However, by increasing the distance and placing the substrate in position 4 on the boat, we will observe a decrease in both the number and size of WS_2 flakes. This decrease can be attributed to the absence of the sulfur precursor. As a result, the optimal position for placing the substrate to achieve the desired number and size of WS_2 flakes is either position 2 or position 3 on the alumina boat.

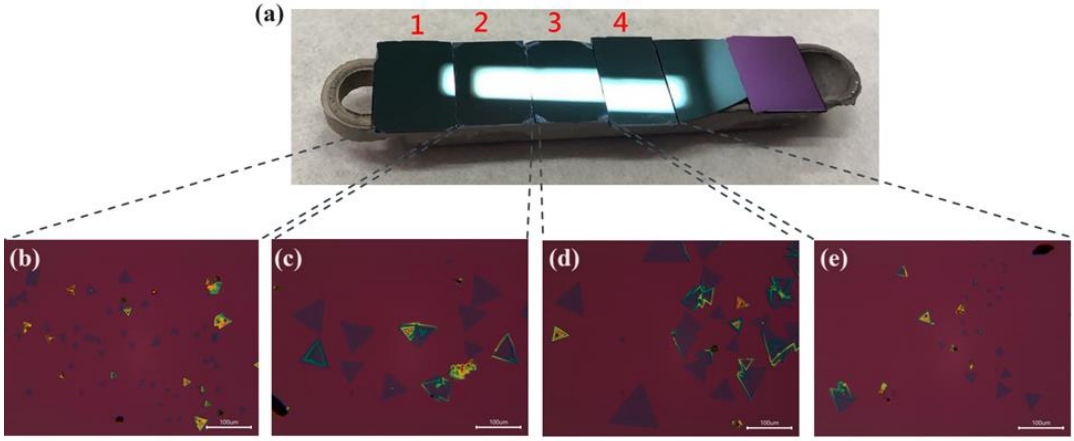


Figure 4. a. *Various positions of substrates placed on the alumina boat; Optical Microscopy images of WS_2 samples grown on SiO_2/Si in* **b.** *position 1,* **c.** *position 2,* **d.** *position 3, and* **e.** *position 4 on the alumina boat.*

Sample characterization

Raman and PL analyses prove to be valuable methods in assessing both the thickness and crystal quality of grown WS_2 flakes. The WS_2 flake obtained under the optimal conditions, as explored and achieved, is depicted in Figure 5. WS_2 Raman spectrum displays two distinctive peaks, known as E^1_{2g} and A_{1g}, which signify the in-plane and out-of-plane vibrations of the lattice. The frequency difference between these two vibrational modes can be utilized to determine the number of layers in the obtained WS_2.

As shown in Figure 6. a, the two characteristic Raman active modes, E^1_{2g} and A_{1g}, are respectively located at 349 cm^{-1} and 415 cm^{-1}, and the frequency difference between these two vibration peaks is approximately 66 cm^{-1}, which aligns well with the values previously reported for monolayer WS_2.

Furthermore, the PL spectrum displayed in Figure 6. b exhibits a distinct peak at 1.95 eV. This peak possesses a relatively high intensity and a narrow full width at half-maximum of 0.07 eV, which clarifies the presence of a direct energy band gap. In addition, this peak provides evidence that the WS_2 flake is single layer. It also suggests that the product possesses excellent crystallinity and overall high quality.

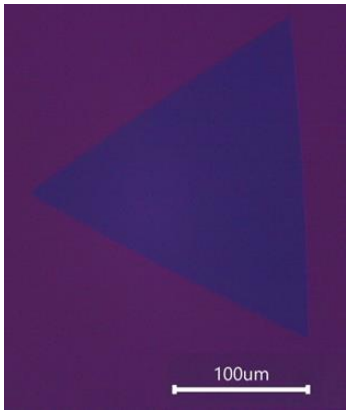


Figure 5. *Optical Microscopy image of the as-grown WS_2 flake on SiO_2/Si substrate using solution precursor under optimal achieved condition.*

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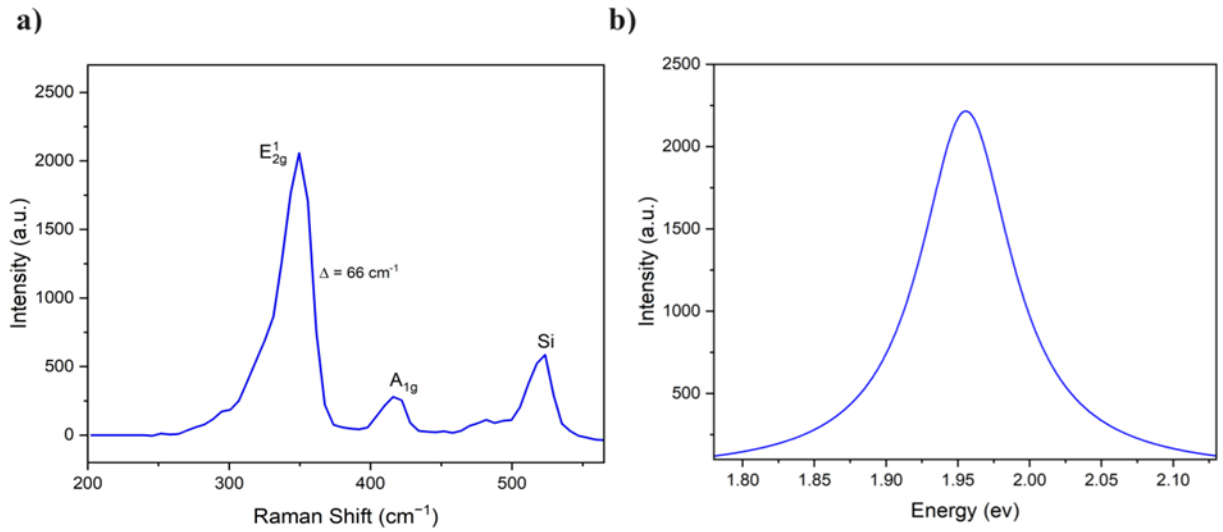


Figure 6. a. *Raman spectrum and* **b.** *PL spectrum of the as-grown WS_2 flake.*

Conclusion

In this study, we successfully synthesized monolayer WS_2 flakes on a SiO_2/Si substrate using the CVD method. We were able to overcome the challenge of tungsten precursor aggregation on the substrate, which is commonly encountered in powder-based CVD methods, by utilizing a solution-based precursor. This approach allowed the uniform diffusion of the precursor on the substrate, resulting in monolayer WS_2 flakes. Furthermore, through the variation and investigation of growth parameters such as growth temperature, solution precursor concentration, and substrate position, we achieved optimal growth conditions for monolayer WS_2 flakes. The high quality of the grown samples was confirmed by Raman and PL spectroscopies. We also believe that this approach can also be applied to the synthesis of other TMDs with different metal precursors. Furthermore, the large size and high quality of the grown samples make them suitable for the fabrication of electronic and optoelectronic devices as well as future practical applications.

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