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Controllable synthesis and optoelectronic applications of wafer-scale MoS$_2$ films

Youngchan Kim
School of Mechanical and Automotive Engineering, KyungSung University, 309, Suyeong-ro, Nam-gu, Busan, Republic of Korea
E-mail: ykim2020@ks.ac.kr
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Abstract

The chemical vapor deposition (CVD) method is widely used for synthesizing two-dimensional (2D) materials such as molybdenum disulfide (MoS$_2$) because of the process' simplicity, relatively low cost, compatibility with other processes, and tendency to result in high-quality crystalline materials. However, the growth of films with a uniform large area of several square centimeters with control of the number of layers remains challenging. Here, a MoS$_2$ synthesis technique that enables thickness and size control of wafer-scale films with high uniformity and continuity is proposed. This CVD technique is a powerful and simple method to control the layer number and size of MoS$_2$ films without using additive chemicals or a complex process. The thickness of the MoS$_2$ films can be controlled from one to four layers by adjusting the concentration of MoO$_3$, MoS$_2$ films with dimensions greater than 10 cm can be grown by manipulating the Ar/H$_2$S ratio. In addition, a photodetector based on CVD-grown MoS$_2$ is shown to exhibit a high current on–off ratio of 10$^5$ and gate-tunability. It also shows a high responsibility of 1.2 A W$^{-1}$, external quantum efficiency of 345%, and a specific detectivity of 1.2 × 10$^{11}$ Jones. The proposed CVD technique can provide a facile direction for the controllable synthesis of wafer-scale MoS$_2$ films with diverse applications in future optoelectronic devices.

1. Introduction

Two-dimensional (2D) transition–metal dichalcogenides (TMDs) are representative layered materials that consist of a transition–metal layer composed of, for example, Mo or W, and two chalcogen layers composed of S, Se, or Te. Two-dimensional TMDs have attracted extensive attention because of their outstanding mechanical, optical, and electrical properties and strong potential for use in applications involving nanoelectronics and optoelectronics [1–5]. In particular, MoS$_2$ is among the most studied 2D TMD materials because of its unusual physical properties, which include high flexibility, optically controllable valley polarization, and unusual bandgap transition [6–8]. MoS$_2$ has been used in various optoelectronics devices, including photodetectors, light-emitting diodes, and solar cells, because of its high electrical mobility (∼200 cm$^2$ V$^{-1}$ s$^{-1}$) and excellent quantum luminescence efficiency [9–11]. Because of the strong potential and wide applicability of 2D TMDs, wafer-scale synthesis methods of these materials have been developed to enable their practical device applications. Synthesis techniques such as atomic layer deposition (ALD), metal–organic chemical vapor deposition (MOCVD), and CVD can fulfill this requirement. ALD is a suitable method for growing large-area 2D TMDs with excellent thickness control because of its self-limiting mechanism [12, 13]. However, the crystal quality of the resultant nanosheets is relatively low. MOCVD has been used to produce wafer-scale films of 2D TMDs with high crystal quality because, unlike general CVD methods, it uses all-gas-phase precursors [14–16]. However, MOCVD has critical shortcomings, including long processing times (>20 h), high operating costs, complex equipment, and a limited number of compatible metal–organic precursors. By contrast, CVD involves a simple process and relatively low-cost setup and provides a high degree of freedom for manipulating the structure of 2D TMDs [17–19]. Because of these advantages, the CVD method is the most widely used method.
for growing 2D TMDs. However, the CVD process also has limitations, including lower crystal quality compared with that of exfoliation samples and low reproducibility of the growth. In particular, CVD suffers from poor control of the thickness and size of the grown films because the use of a solid-state precursor makes controlling the vapor pressure difficult. Although some strategies have been developed for growing multilayer 2D TMDs, the resultant films exhibited a limited size and area, on the scale of micrometers. The synthesis of uniform wafer-scale films of 2D TMDs with controlled thickness remains a challenge.

A CVD method for the wafer-scale synthesis of a monolayer MoS2 film using MoO3 and H2S as precursors has been reported [20]. The CVD-grown monolayer MoS2 showed high uniformity, good continuity, and high electrical mobility (~1 cm² V⁻¹ s⁻¹). However, control of the layer number and size of MoS2 films synthesized using precursors of MoO3 and H2S and their optoelectronic devices applications is also challenging. In the present work, a controllable synthesis method for wafer-scale, homogeneous MoS2 films is proposed. The relation between the precursor concentration and the size of the grown MoS2, and that between the precursor concentration and the MoS2 layer thickness were systematically investigated. The size of the obtained MoS2 film can be controlled by manipulating the ratio between Ar and H2S gases. For instance, the size of the MoS2 film can be expanded to 10 cm when a Ar/H2S ratio of 500 is used in the growth process. The growth length is linearly proportional to the Ar/H2S ratio. In addition, the thickness of the obtained MoS2 film can be controlled from one layer (1L) to four layers (4L) by increasing the concentration of MoO3 from 5 to 20 mg. The relation between number of layers and the concentration of MoO3 exhibits a nearly linear proportion. The 1 × 1 cm² MoS2 films (1L–4L) exhibit high uniformity and continuity in area. Their optical properties and thickness were characterized by Raman spectroscopy and photoluminescence (PL) spectroscopy. Moreover, a photodetector array based on a CVD-grown MoS2 film with high uniformity was fabricated. It showed a high photoresponsivity of 1.2 A W⁻¹, an external quantum efficiency (EQE) of 35%, and a specific detectivity (D*) of 1.2 × 10¹¹ Jones, which are comparable to, or even greater than, those of other devices based on CVD-grown MoS2. The photodetector also exhibits highly gate-tunable optoelectronic properties and a high electrical current on–off ratio of 10⁷. These results confirm that wafer-scale CVD-grown MoS2 has strong potential for use in next-generation optoelectronic devices and systems.

2. Experimental details

Synthesis of MoS2 films: Before the synthesis, a 300 nm SiO2/Si wafer was cleaned for 10 min in acetone and isopropyl alcohol (IPA), respectively, and then rinsed in deionized water (DIW) several times. MoO3 powder (99.5%, Sigma-Aldrich) was used as a precursor for the growth of MoS2. As shown in figure S1, MoS2 films were prepared in a custom-made quartz tube furnace under a low pressure of 10⁻³ Torr. The MoO3 powder in a quartz boat was placed at the center of furnace and a Si/SiO2 substrate was placed downstream from MoO3 powder. The furnace was heated to 600 °C at a rate of 20 °C min⁻¹. After H2S gas was injected into the quartz tube at 600 °C, a MoS2 film was directly grown for 30 min by sulfurization of MoO3. The sample was rapidly cooled down to room temperature after growth. In whole process, Ar gas used as a transporting material and for control of size with H2S gas. The density and supply duration of H2S and Ar gas were exactly adjusted by mass flow controller and CVD system.

Characterization of CVD-grown MoS2: Raman spectroscopy and PL spectroscopy (NTMDT AFM-Raman spectroscope) were carried out using a 532 nm wavelength laser to obtain PL and Raman spectra. To avoid thermal damage to the samples, the laser intensity was set at a value less than 1 mW. Before measurements, the instrument was calibrated using the Si peak at 520 cm⁻¹.

Fabrication of electrical devices based on as-grown MoS2: Electrical devices were fabricated using conventional photolithography. Metal electrodes of Cr/Au(5/50 nm) were deposited onto as-grown MoS2 by a lift-off process using an e-beam evaporation system. Isolation of channel area of MoS2 was performed by additional photolithography and plasma etching. The electrical properties of devices were evaluated using a semiconductor parameter analyzer (Agilent 4155C). The photoelectrical properties of the devices were investigated under illumination of a solid-state laser with a broad wavelength range from 400 to 700 nm.

3. Results

Figure 1(a) shows CVD-grown 1L MoS2 on a SiO2/Si(300 nm/500 μm) substrate, where the MoS2 was grown under different Ar/H2S gas ratios at a fixed temperature of 600 °C and with a fixed MoO3 concentration of 5 mg. The process was performed in a homemade CVD apparatus at a pressure of 10⁻³ Torr. As the ratio of Ar/H2S was increased from 50 to 500, the growth length of MoS2 increased from 2.4 to 10.3 cm. The growth length as a function of the Ar/H2S ratio is plotted in figure 1(b), where the sublinear Ar/H2S ratio dependency of the growth length shows a slope of ~0.017. In the present work, the maximum Ar/H2S ratio in the CVD apparatus...
was limited to 500. If the experiments could be conducted at Ar/H2S ratios greater than 500, a MoS2 film larger than 10 cm could likely be grown on SiO2. This simple CVD method does not require any additional complex processes. The results reveal that the film size of MoS2 can be controlled by manipulating the Ar/H2S ratio.

Figure 1 (c) is an optical microscope image of the CVD-grown MoS2 on SiO2/Si. Part of the MoS2 was scratched for better identification of the MoS2 film. Figure 1 (c) shows a highly uniform and continuous MoS2 film grown on the SiO2/Si. The Raman spectrum was obtained for qualitative analysis of the MoS2. The laser intensity was sustained at 1 mW to avoid thermal damage to the sample. Figure 1 (d) shows a typical Raman spectrum of CVD-grown MoS2 with a peak distance of 20.5 cm$^{-1}$ between the A1g mode at 405.3 cm$^{-1}$ and the E1$^2$g mode at 384.8 cm$^{-1}$, indicating that it is a perfect monolayer [21, 22]. The PL spectrum of MoS2 also indicates a typical monolayer, showing peaks at 1.89 and 2.03 eV, which correspond to the A1 and B1 direct excitonic modes, respectively [23]. The Raman mapping across an area 50 $\times$ 50 $\mu$m was performed and a related statistical histogram was plotted (Figure S2). The histogram proves that the average peak distance between the A1g mode and the E1$^2$g mode of 20.5 cm$^{-1}$ is the same result as single Raman data. The Raman mapping image and histogram show high uniformity and homogeneity of the MoS2 sample.

Figure 2 (a) demonstrates that MoS2 can be synthesized with a thickness from 1L [24–26] to 4L over an area of 2 $\times$ 2 cm$^2$. Each of the CVD-grown MoS2 films is highly uniform and continuous. The number of layers of MoS2 was controlled by adjusting the concentration of MoO3 while the other growth conditions were fixed (temperature: 600 °C, Ar/H2S = 200). Compared with bare SiO2, the sample contrast increases with increasing MoS2 thickness because of light interference between the sample and the SiO2 substrate. To identify the number of layers of these samples, Raman spectroscopy was conducted (figure 2(b)). According to a previous report, Raman spectroscopy is a powerful tool to confirm the MoS2 thickness from 1L to 4L. The distances between the E2g and A1g peaks were calculated from the spectra of the 1L to 4L samples (figure 2(b)) (1L: 19.5 cm$^{-1}$, 2L: 21.6 cm$^{-1}$, 3L: 23.6 cm$^{-1}$, 4L: 24.3 cm$^{-1}$). These results are in good agreement with those of a previous report [21, 22]. To confirm thickness of these samples more precisely, AFM measurements were conducted. The height profile revealed that the thicknesses of the MoS2 films were about 0.8, 1.5, 2.5, 3.5 nm (Figure S3). The thickness of monolayer MoS2 is about 0.7–0.8 nm. Considering a 0.2 nm roughness value of amorphous SiO2 substrate, the number of layers of each sample was estimated to be 1, 2, 3, and 4 respectively. Also, AFM topography of MoS2 films with various thickness shows high continuity and uniformity. As the concentration of MoO3 was increased from 5 to 20 mg, the number of layers of MoS2 increased from 1L to 4L (figure 2(c)). The relation between the thickness of MoS2 and the concentration of MoO3 shows linear proportionality, demonstrating that the number of layers of MoS2 can be perfectly controlled by adjusting the concentration of MoO3. Thus, the proposed CVD technique is a simple and powerful method to control the thickness of large-scale and highly uniform MoS2 films without the assistance of complex processes or chemical additives.

Figure 1. Control of the size of MoS2. (a) Image of as-grown MoS2 films on SiO2 (length: from 2 to 10 cm, width: 1 cm); (b) growth length as a function of the Ar/H2S ratio; (c) OM image of as-grown MoS2 with a scratched area; (d) Raman spectrum of MoS2; and (e) PL spectrum of MoS2.
conductance band, which results in the formation of a smaller barrier between the conductance band of MoS2. Contrary, when a positive voltage is applied, the Fermi level moves from the valence band toward the large barrier, and the density of the photocurrent is therefore lower than that under the zero gate voltage. On the other hand, the Fermi level of Au metal. The photocurrent increases because of the smaller barrier.

The photocurrent at $V_G = 80$ V. The photocurrent at $V_G = 80$ V was greater than that under dark conditions ($I_{ph}$). Moreover, the generated photocurrent ($I_{ph} = I_{illumination} - I_{Dark}$) could be enhanced by controlling the gate voltage. That is, the MoS2-based photoresistor showed highly gate-tunable optoelectronic properties, consistent with previous reports of devices based on exfoliated MoS2 or CVD-grown MoS2.

To precisely evaluate the enhancement of the photocurrent generation efficiency by the gate voltage, photoresponsivity ($R$), which is one of a critical parameters used to characterize the performance of a photodetector, was estimated. The photoresponsivity is defined as the ratio between the output photocurrent ($I_{ph}$) and the input optical power ($P_{in}$) in the active area of a device: $R = I_{ph}/P_{in}$. In figure 3(d), the photoresponsivity is $\sim 1$ mA W$^{-1}$ under a zero gate voltage. As the gate voltage increases to 80 V, the photoresponsivity increases dramatically. The maximum value of the photoresponsivity is $\sim 0.5$ A W$^{-1}$ at $V_G = 80$ V. The photocurrent at $V_G = 80$ V is 500 times greater than that at $V_G = 0$ V (1 mA W$^{-1}$). As mentioned above, a phototransistor composed of a mechanically exfoliated MoS2 monolayer exhibits a maximum responsivity of 0.42 mA W$^{-1}$ at $V_G = 0$ V and 7.5 mA W$^{-1}$ at $V_G = 40$ V. This result is comparable to those previously reported for CVD-grown MoS2 films [24–26]. Conversely, the photoresponsivity decreases when a negative gate voltage is applied (figure 3(d)). According to previous studies, the $V_G$-dependent photoresponsivity of a MoS2 phototransistor is a consequence of $n$-type doping of MoS2 [27, 28]. After deposition of the Au metal contact, MoS2 exhibits a quasi-equilibrium Fermi level, which can be moved by controlling the gate voltage. When a negative voltage is applied, the Fermi level shifts from the conductance band of MoS2 toward its valence band, which indicates the formation of a larger barrier between the conductance band of MoS2 and the Fermi level of Au metal. The photogenerated carriers are resistant to drift because of the large barrier, and the density of the photocurrent is therefore lower than that under the zero gate voltage. On the contrary, when a positive voltage is applied, the Fermi level moves from the valence band toward the conductance band, which results in the formation of a smaller barrier between the conductance band of MoS2 and the Fermi level of Au metal. The photocurrent increases because of the smaller barrier.

In general, the photoresponse mechanism of graphene is related to photothermoelectric (PTE) and bolometric effects. However, unlike graphene-base devices, optoelectronic devices of TMDs such as MoS2 are

![Figure 2. Control of the number of layers of MoS2. (a) Image of as-grown MoS2 (from 1L to 4L) over an area of 2 $\times$ 2 cm$^2$; (b) Raman spectra of as-grown MoS2 (from 1L to 4L); (c) peak difference between the A$_{1g}$ mode and the E$_{2g}$ mode as a function of the concentration of MoO$_3$ powder.](Image)
more dependent on photoconductive (PC) and photogating (PG) effects than on photothermal and bolometric effects [29, 30]. The PC effect results in photocurrents by creating electron–hole pairs when photons with an energy greater than the bandgap energy of monolayer MoS2 (≈1.89 eV) are absorbed in MoS2 devices. The PG effect is a special case of the photocurrent effect. The localized energy states at defects and impurities can trap the charge carriers generated under illumination. These trapped carriers can form local gates that severely affect carrier conductance. The photocurrent generated by the PG effect is proportional to $g_m \Delta V_{th}$ ($g_m = \frac{dI_d}{dV_G}$, which is the transconductance; $\Delta V_{th}$ is the change in the threshold voltage) [31]. That is, the PG effect can be described as the difference in $V_{th}$ between dark and illumination conditions. In figure 4(a), a clear change of $\approx 9$ V is observed for the threshold voltage under illumination ($V_{th,dark}$: 42.2 V, $V_{th,illumination}$: 51.2 V), which indicates that a phototransistor based on 1L-MoS2 exhibits strong dependency on the PG effect. The results demonstrate that phototransistors based on exfoliated MoS2 exhibit a strong photovoltaic effect because of defects and impurities. Moreover, the structural defects and impurities in CVD-grown MoS2 are assumed to be more abundant than those in intrinsic exfoliated MoS2 crystals because of the relatively lower crystallinity and greater number of impurities generated by the complex synthesis process. For these reasons, the threshold voltage shifts under incident light until the localized trap states are fully occupied (figure 3(c)).

To verify the optical power dependency of the photocurrent, the photocurrent is plotted as a function of the optical power in figure 4(a). The experiments were carried out using a laser with a 550 nm excitation wavelength and a fixed $V_G$ of 50 V. To avoid thermal damage, the optical power should be less than 1 mW. When the maximum optical power in the MoS2 channel area (2000 $\mu m^2$) was increased from 0.78 to 114.59 nW, the photocurrent increased gradually (figure 4(a)). The photocurrent under a fixed $V_D$ of 1 V was extracted and plotted in figure 4(b). The sublinear relationship between photocurrents and optical powers can be described as $I_{ph} \propto P^{\alpha}$, where $I_{ph}$ is the photocurrent, $P$ is the optical power, and $\alpha$ represents a constant. An $\alpha$ value of 0.78 was calculated through the power-function fitting method. According to previous reports, the sublinear power law $I_{ph} \propto P^{0.78}$ can be caused by the defects in MoS2 and by absorbed molecules and impurities at the interface of MoS2 and the SiO2 substrate. Figure 4(c) shows the optical spectral response of a MoS2 phototransistor at different excitation wavelengths in the range of visible light (from 400 to 700 nm) under $V_G = 80$ V and $V_D = 1$ V. As previously mentioned, the photoresponsivity was calculated by the equation $R = I_{ph}/P_{in}$. 

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**Figure 3.** Optoelectronic properties of as-grown MoS2. (a) Schematic of an electrical device based on as-grown MoS2; (b) image and OM image of an electrical device based on as-grown MoS2; (c) transfer curves of a MoS2 photodetector under illumination; and (d) responsivity of a MoS2 photodetector as a function of the gate voltage.
The EQE is a critical parameter to evaluate the performance of optoelectronic devices. The EQE is the ratio between the number of electron–hole pairs that participate in the photocurrent and the number of incident photons. It can be expressed as \( \text{EQE} = \frac{I_{\text{ph}}/e}{P_{\text{in}} \lambda / (h \cdot c)} \), where \( e \) is the elementary charge \((1.6 \times 10^{-19} \text{ C})\), \( h \) is Planck’s constant, \( \lambda \) is the excitation wavelength, and \( c \) is the speed of incident light. Both the photoresponsivity and EQE tend to decrease with increasing illumination wavelength, consistent with the previously discussed results. In particular, the responsivity and EQE increase to 1.2 A W\(^{-1}\) and 354% at 400 nm, respectively. The bandgap of 1L-MoS\(_2\) is 1.89 eV, which corresponds to a wavelength of \( \sim 660 \text{ nm} \). An incident photon with an energy greater than the bandgap energy of MoS\(_2\) can theoretically be absorbed into MoS\(_2\) phototransistors. According to a previous study, the absorbance of a MoS\(_2\) monolayer decreases dramatically as the illumination wavelength increases from 300 to 1200 nm \([32]\). The remarkable increases of \( R \) and EQE at short wavelengths are attributed to a greater absorbance of photons. In addition, photons with a higher energy (shorter wavelength) can create more photocurrent because of a greater probability of overcoming the localized trapping by defects. Here, we note that most of the EQE values are greater than 100%, except that at 650 nm. The high EQE values are caused by the generation of multiple electron–hole pairs from a single absorbed photon. The carriers in the MoS\(_2\) device can recirculate numerous times before recombination, resulting in greater efficiency.

The other important characteristic of a photodetector is detectivity \((D^*)\). The detectivity as a function of the illumination wavelength is plotted for the same optical power and gate and drain voltages in figure 4(b). If the shot noise from dark current dominates the total noise of a photodetector, then the detectivity can be defined as \( D^* = RA/(2eI_{\text{dark}})^{1/2} \), where \( R \) is the photoresponsivity, \( A \) is the effective area, \( e \) is the elementary charge, and \( I_{\text{dark}} \) is the dark current. Figure 4(d) does not show remarkable changes in the detectivity with increasing illumination wavelength. The maximum value of the detectivity is \( \sim 1.22 \times 10^{11} \) Jones at a wavelength of 400 nm, which is comparable to the detectivity of photodetectors of TMDs such as ReS\(_2\), WSe\(_2\), and MoTe\(_2\), as well as to the detectivity of phototransistors based on MoS\(_2\) \([33–35]\).

Time-resolved experiments were conducted to investigate the photoswitching characteristics and reproducibility of 1L-MoS\(_2\) phototransistors. As shown in figure 5(a), the drain current as a function of time was measured under repeated on–off photoswitching at a fixed \( V_D \) of 1 V and a constant optical power at a wavelength of 550 nm. Figure 5(a) shows a highly sustainable and stable photoresponse during on–off
photoswitching tests. In Figure 5(b), the photocurrent was recorded in a higher temporal resolution to calculate the response rate. The rise time of the MoS$_2$ phototransistor is $\sim$0.7 s, and the decay time is $\sim$2 s. This rise time is longer than that in an exfoliated MoS$_2$ phototransistor (50 ms). According to previous reports, the response time of the photodetector is mainly affected by defect states and adsorbents [36–40]. TMDs such as MoS$_2$ and WSe$_2$ synthesized at high temperature show high density of defect states because of chalcogen vacancies. The defect states of vacancies near the Fermi level become additional trapping centers of photogenerated carriers, and yield long response time with low photocurrent [39, 40]. Also, the absorbent such as environmental oxygen and water molecules can give critical effects on response time of 2D materials due to high surface to volume ratio. The absorbed oxygen molecules on the surface defects act another trapping center, and result in larger response time. Especially, the oxygen captures the photogenerated hole under illumination, and it hinder recombination of trapped holes and photogenerated electrons [38, 41, 42]. Because of the environmental absorbents, relatively many photodetectors based on TMDs shows tendency of larger decay time compared to rise time [43]. However, the responsivity and EQE were 70 times greater than those of the exfoliated MoS$_2$, as discussed previously. In general, the more abundant localized trap states generated by defects and impurities because of the relatively low crystallinity of CVD-grown MoS$_2$ can enhance the photoresponsivity and the photoefficiency by the photogating effect but also result in a slower response. Consequently, a MoS$_2$ phototransistor exhibits a relatively high responsivity and a low response rate.

4. Conclusion

In the present work, the controllable synthesis of wafer-scale MoS$_2$ films was achieved via the CVD method using precursors of MoO$_3$ and H$_2$S. The number of layers and the size of MoS$_2$ films were demonstrated to be controlled through manipulation of the concentration of MoO$_3$ and the Ar/H$_2$S ratio, respectively. The CVD-grown MoS$_2$ film exhibited high uniformity and continuity. These results show that the proposed CVD technique is a simple and powerful method to control the size and thickness of not only 2D MoS$_2$ films but also films of other wafer-scale 2D materials. In addition, an optoelectronic device based on the as-grown MoS$_2$ exhibited gate tunability, a high responsivity of 1.2 A W$^{-1}$, an EQE of 345%, and a specific detectivity of $1.2 \times 10^{11}$ Jones. The CVD method described here can help expand the range of feasible applications of 2D materials, including MoS$_2$, in practical nanoelectronic and optoelectronic devices.

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Data availability statement

The data generated and/or analysed during the current study are not publicly available for legal/ethical reasons but are available from the corresponding author on reasonable request.

ORCID iDs

Youngchan Kim @ https://orcid.org/0000-0001-7637-7515

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