Growth of a Large, Single-Crystalline WS$_2$ Monolayer for High-Performance Photodetectors by Chemical Vapor Deposition

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Abstract: 2D WS$_2$ is a promising candidate for the next generation nanoelectronics, spintronics, valleytronics, and optoelectronics. However, the uncontrollably large-area growth of WS$_2$ nanosheets and their unsatisfactory performance of the photodetectors based on WS$_2$ hinder its applications. Here, we proposed a CVD method using tungstic acid as the precursors to grow WS$_2$ flakes. After being characterized by AFM, Raman, PL, and TEM, we found the as-grown WS$_2$ flakes were high-quality structures. Then the photodetectors based on the as-grown WS$_2$ were fabricated, which exhibited high responsivity (7.3 A W$^{-1}$), a fast response rate (a response time of 5 ms and a recovery time of 7 ms), prefect external quantum efficiency (EQE) (1814%), and remarkable detectivity ($D^*$) (3.4 x 10$^{12}$ Jones). Our works provided a new CVD method to grow some high-quality WS$_2$ nanosheets.

Keywords: WS$_2$ flakes; tungstic acid; chemical vapor deposition; photodetectors

1. Introduction

Atomically thin tungsten disulfide (WS$_2$), a 2D crystal with some interesting and important properties, is a promising candidate for the next generation of nanoelectronics, spintronics, valleytronics, and optoelectronics [1–7]. For example, WS$_2$ has a direct bandgap in the visible range and high absorption relative to its thickness [5,6]. WS$_2$ exhibits ambipolar field-modulation behavior [9]. The theoretical calculations predict that it has a reduced effective mass, allowing higher carrier mobility [10]. Monolayer WS$_2$ has strong PL emission efficiency, stronger than other TMDCs [11,12]. WS$_2$ exhibits strong spin-orbit coupling and band splitting due to spin enabling spintronics/valleytronics [13–16]. WS$_2$ also has high nonlinear susceptibility, suggesting its use for nonlinear optical devices [17,18]. Nevertheless, most researchers on WS$_2$ are largely limited because of the relatively small lateral size of exfoliated flakes, not to mention the randomness of their shape, thickness, and crystal quality [19,20]. Liquid exfoliation has an advantage for the mass production of WS$_2$, but it is difficult to control defects, hindering its application to electronic devices [21–23]. CVD is a hopeful approach to grow large-area WS$_2$ flakes for extensive device applications. There are two common methods to synthesize WS$_2$ by supplying tungsten sources on inert substrates before sulfurization in CVD. One is thin tungsten films or thin tungsten oxide films deposited by various methods including e-beam evaporation, magnetron sputtering, and atomic layer deposition [11,24–29]. The other is that of tungsten oxides solid precursors vaporized with solid sulfur sources simultaneously during the CVD process [30–34]. As it is difficult to control the tungsten sources location, distribution, and uniformity precisely, and the poor adhesion of the sources to inert substrates, the controlled CVD growth of large-area uniform monolayer WS$_2$ remains a challenge. Some groups used metal substrates (Au) instead of the inert substrates to synthesize a large-area monolayer WS$_2$ film; they also tested the electrical properties of WS$_2$-based field-effect transistors.
after bubbling transfer from Au substrates to SiO$_2$/Si substrates [35–37]. Here we report a new method, spin-coating tungsten acid as a tungsten source on SiO$_2$/Si substrates directly before sulfurization, to grow single crystalline WS$_2$ monolayers with a size of up to hundreds of micrometers. We also found that photodetectors based on single crystalline WS$_2$ flakes have a high responsivity of 7.3 A W$^{-1}$ with a fast response rate of 5 ms, an external quantum efficiency (EQE) of 1814%, and a detectivity ($D^*$) of 3.4 × 10$^{12}$ Jones.

2. Materials and Methods

The WS$_2$ flakes were synthesized by using tungstic acid on SiO$_2$/Si substrates as the precursors and sulfurizing via a chemical vapor deposition (CVD) method. First, 2.8 g of tungstic acid (99.9%, Alfa) was dispersed in the oxalic acid solution (0.15 M/L) by ultrasound. Then the tungstic acid colloidal solution was dispersed on a clean SiO$_2$/Si substrate by spin-coating. After that, the whole substrate was annealed in air at 100 °C for 1h. Finally, the SiO$_2$/Si substrates with tungstic acid were sulfurized by CVD. For CVD growth, the Si/SiO$_2$ substrates with the tungstic acid colloidal solution were placed at the center of the quartz tube, and the 0.1 g S powders (99.5%, Alfa) were placed on the upstream side of the Si/SiO$_2$ substrates. The quartz tube was flushed with Ar (5N) gas several times and purged to 0.1 Pa with a mechanical pump. The furnace temperature was raised to 900 °C in 30 min and kept for 30 min with a flow rate of 50 sccm Ar gas. After growth, the furnace was naturally cooled to room temperature. The synthesized WS$_2$ samples were characterized by an atomic force microscope (AFM, SPM9700, Shimadzu), Raman spectroscopy, and a transmission electron microscope (TEM, Tecnai G$^2$ F30 S-TWIN, FEI). The Raman spectra were recorded in the backscattering geometry at a 532 nm line with an argon ion laser Raman spectrometer (LabRAM HR800, Horiba JobinYvon). The Raman mappings were collected by alpha300 R, WITec GmbH, Ulm, Germany, the laser wavelength was 532 nm, and the scanning step interval was 300 nm.

We fabricated the photodetectors based on the WS$_2$ flakes by a standard photolithography procedure (MDA-400M, Midas). The 10/50 nm Ti/Au electrode patterns were deposited by an E-beam deposition system (Nexdep, Angstrom Engineering). The photodetector measurement devices contained a broadband laser-driver light source (LDLS, EQ-1500, Energetiq) calibrated by a UV-enhanced silicon photodiode in an ambient atmosphere, a semiconductor characterization system (4200-SCS, Keithley), and an oscilloscope (DSO-X 3052A, Keysight). The oscilloscope light pulse chopped was 500 nm, and the frequency was 3 Hz.

3. Results and Discussion

Figure 1a shows the optical image of the as-grown triangle WS$_2$ flakes on SiO$_2$/Si substrate; the majority of the flakes were more than 220 µm. The thickness of these triangle crystals was measured by atomic force microscopy (AFM) (Figure 1b). The AFM step height of the WS$_2$ flake was typically measured at 0.7 nm, which corresponds to a one-layer structure.

Figure 1. (a) Optical microscope images of the monolayer WS$_2$. (b) AFM images of the monolayer WS$_2$. 
We have further investigated the layer number of the resulting atomically thin WS$_2$ triangles by Raman spectroscopy. As shown in Figure 2a, the strongest peak of the WS$_2$ triangle flake at about 350 cm$^{-1}$ includes three subpeaks, which are resolved by a multipeak Lorentzian fitting. According to the calculated phonon dispersion [38] and experimental studies [13,39–41] of 1L-WS$_2$, the in-plane vibrational $E_{1g}^1$(M) mode peak is at 343 cm$^{-1}$, the second-order mode of longitudinal acoustic phonon $2LA$(M) peak is at 350 cm$^{-1}$, and the in-plane vibrational $E_{1g}^2$(Γ) mode peak is at 355 cm$^{-1}$, respectively. The other peaks at 418, 295, and 322 cm$^{-1}$ are attributed to the out-of-plane $A_{1g}$(Γ) mode, the combination modes of $2LA$(M)$-2E_{2g}^2$(Γ), and the combination modes of $2LA$(M)$-E_{2g}^2$(Γ), respectively. The spectral fingerprint of a monolayer WS$_2$ is the frequency separation of 62 cm$^{-1}$ between the $E_{1g}^1$(Γ) mode and the $A_{1g}$(Γ) mode [11]. Raman images (Figure 2b,c) plotted by extracting the intensity were acquired to demonstrate the uniformity of our WS$_2$ samples. The $2LA$(M) phonon mode was much more intense than the $A_{1g}$(Γ) mode. The as-grown WS$_2$ triangle flake was uniform.

![Figure 2](image)

**Figure 2.** (a) Raman characterization of the monolayer WS$_2$ triangle flake with 532 nm excitation wavelengths. (b) $A_{1g}$(Γ) intensity mapping for the WS$_2$ triangle flake. (c) Intensity ratio mapping of $2LA$(M) over $A_{1g}$(Γ) for the WS$_2$ triangle flake.

The optical properties of the monolayer WS$_2$ triangle flakes were further investigated by microphotoluminescence (PL). The monolayer WS$_2$ flake has a PL peak at about 642 nm, and its full width half maximum (FWHM) value is 22 nm (Figure 3a). The corresponding PL peak-integrated intensity, position, and width mappings of the monolayer WS$_2$ triangle flake are shown in Figure 3b–d, respectively. The results of the PL indicated the as-grown WS$_2$ was defect free and uniform.

The structures of the monolayer WS$_2$ triangle flakes were characterized by TEM. Figure 4a shows the low magnitude bright-field TEM image of a WS$_2$ triangular crystal. The high-resolution TEM (HRTEM) image (Figure 4b) shows the hexagonal lattice fringes, which indicates a perfect atomic structure with a lattice spacing of 0.27 nm, corresponding to the (100) planes. The corresponding selected area electron diffraction (SAED) pattern (Figure 4c) revealed the defect free nature of the WS$_2$ flake [36]. Then the elemental compositions of the WS$_2$ triangle flakes were acquired by EDX mapping and EDX spectrum. EDX mapping in Figure 4d,e indicates the W and S elements distribute evenly. EDX spectrum in Figure 4f demonstrates the atomic ratio of W and S is around 1.2, which is consistent with the original stoichiometry of WS$_2$. The TEM results further prove that as-grown WS$_2$ flakes are defect free.
To research the optoelectronic properties of the WS\(_2\) flakes, photodetectors based on them were fabricated. The spectral response curve reaches a minimum at the wavelength of \(\approx 645\) nm in Figure 5a. Hence, the bandgap is about 1.92 eV by calculation, which is consistent with the PL results. Fitting the plot of photocurrent \(I_{\text{ph}}\) on light intensity \(P\) for the WS\(_2\) flake as \(I_{\text{ph}} \approx P^\theta\) obtains the value of \(\theta \approx 0.96\) (Figure 5b), hinting the as-grown WS\(_2\) has very few defects or traps to photo-induced electron/hole pairs in the test power density range [42]. We further tested the cyclability of the photodetector under 500 nm incident light with the light on/off time interval of 30 s under a bias of 1 V (Figure 5c).
The performances of the photodetector are stable. The response and recovery rates were 5 ms and 7 ms tested by an oscilloscope, respectively (Figure 5d). The photoresponsivity was 7.3 A W\(^{-1}\), according to the \(R_\lambda = I_{\text{ph}}/P_S\), where \(I_{\text{ph}}\) is the photoexcited current, \(P\) is the light power intensity, and \(S\) is the effective area of the photodetector. The external quantum efficiency (EQE) was 18.14%, according to the \(\text{EQE} = \frac{hcR_\lambda}{e\lambda}\), where \(h\) is Plank’s constant, \(c\) is the light velocity, \(R_\lambda\) is the photoresponsivity, \(e\) is the elementary electronic charge, and \(\lambda\) is the exciting wavelength. The specific detectivity \((D^*)\) was \(3.4 \times 10^{12}\) Jones, according to the \(D^* = \frac{R_\lambda S^{1/2}}{(2eI_{\text{dark}})^{1/2}}\), where \(I_{\text{dark}}\) is the dark current. The performance of our photodetector is more inspiring than the most reported WS\(_2\) based photodetectors shown in Table 1.

![Figure 5. Optoelectronic properties of the WS\(_2\) triangle flake. (a) The spectral response curve of the WS\(_2\) triangle flake. Inset: the fitting curve of obtaining the bandgap. (b) Photocurrent as a function of illumination intensity at \(V_{\text{bias}} = 1\) V. Inset: the optical image of the WS\(_2\) photodetector; the scale bar is 10 \(\mu\)m. (c) Time-resolved photoresponse of the WS\(_2\) photodetector under a bias voltage of 1 V and illumination power of 3.68 mW cm\(^{-2}\). (d) Response and recovery curves.](image)

**Table 1.** Comparison of the key parameters of our photodetector.

| Photodetectors | Fabrication Method | \(R_\lambda\) (A/W) | EQE (%) | Response Time (ms) | Reference |
|---------------|-------------------|--------------------|---------|-------------------|-----------|
| Multilayer WS\(_2\) | CVD | \(92 \times 10^{-6}\) | - | 5.3 | [4] |
| Multilayer WS\(_2\) | Exfoliated | 5.7 | 1118 | <20 | [43] |
| Monolayer WS\(_2\) | CVD | \(18.8 \times 10^{-3}\) | - | <4.5 | [44] |
| Multilayer WS\(_2\) | PLD | 0.51 | 137 | \(4.1 \times 10^{-3}\) | [45] |
| Monolayer WS\(_2\) | CVD | 3.07 | 763 | 370 | [46] |
| Multilayer WS\(_2\) | Drop casting | \(145 \times 10^{-3}\) | - | 153.78 | [47] |
| Monolayer WS\(_2\) | CVD | 7.3 | 1814 | 5 | This work |

4. Conclusions

In summary, we grew the monolayer WS\(_2\) triangle flakes via a CVD method with the tungstic acid, and researched the performance of the photodetectors based on them. The utilization of tungstic acid colloidal solution could improve the uniformity of the tungsten sources on the substrates. The as-grown monolayer WS\(_2\) flakes have a size of about 220 \(\mu\)m, a bandgap of about 1.92 eV, and no defects. The photodetectors based on them showed
excellent performance, such as high responsivity of 7.3 A W⁻¹, large EQE of 1814%, and a fast response rate of 5 ms.

Funding: This research was funded by the Natural Science Foundation of China, grant number 21701041; the Open Foundation of Hubei Collaborative Innovation Center for High-efficiency Utilization of Solar Energy, grant number HBSSKQN2017001; and the Talents of high-level scientific research foundation of the Hubei University of Technology, grant number BSQD2017010.

Institutional Review Board Statement: Not applicable.

Informed Consent Statement: Not applicable.

Data Availability Statement: No new data were created or analyzed in this study. Data sharing is not applicable to this article.

Conflicts of Interest: The authors declare no conflict of interest.

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