

## Large Area Growth and Electrical Properties of p-Type WSe<sub>2</sub> Atomic Layers

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### Supporting Information

**ABSTRACT:** Transition metal dichalcogenides represent a unique class of two-dimensional layered materials that can be exfoliated into single or few atomic layers. Tungsten diselenide (WSe<sub>2</sub>) is one typical example with p-type semiconductor characteristics. Bulk WSe<sub>2</sub> has an indirect band gap (~1.2 eV), which transits into a direct band gap (~1.65 eV) in monolayers. Monolayer WSe<sub>2</sub>, therefore, is of considerable interest as a new electronic material for functional electronics and optoelectronics. However, the controllable synthesis of large-area WSe<sub>2</sub> atomic layers remains a challenge. The studies on WSe<sub>2</sub> are largely limited by relatively small lateral size of exfoliated flakes and poor yield, which has significantly restricted the large-scale applications of the WSe<sub>2</sub> atomic layers. Here, we report a systematic study of chemical vapor deposition approach for large area growth of atomically thin WSe<sub>2</sub> film with the lateral dimensions up to ~1 cm<sup>2</sup>. Microphotoluminescence mapping indicates distinct layer dependent efficiency. The monolayer area exhibits much stronger light emission than bilayer or multilayers, consistent with the expected transition to direct band gap in the monolayer limit. The transmission electron microscopy studies demonstrate excellent crystalline quality of the atomically thin WSe<sub>2</sub>. Electrical transport studies further show that the p-type WSe<sub>2</sub> field-effect transistors exhibit excellent electronic characteristics with effective hole carrier mobility up to 100 cm<sup>2</sup> V<sup>-1</sup> s<sup>-1</sup> for monolayer and up to 350 cm<sup>2</sup> V<sup>-1</sup> s<sup>-1</sup> for few-layer materials at room temperature, comparable or well above that of previously reported mobility values for the synthetic WSe<sub>2</sub> and comparable to the best exfoliated materials.



**KEYWORDS:** layered materials, semiconductor, tungsten diselenide, field effect transistor, large area growth

Graphene has attracted considerable interest for applications in diverse electronic and optoelectronic devices due to its unique electronic properties and atomically thin geometry.<sup>1–12</sup> However, the gapless band structure limits the potential of graphene for digital electronic devices.<sup>2,4</sup> It has been shown the energy band structure of the transition metal dichalcogenides (TMD) materials exhibit a unique indirect-to-direct band gap transition as their layer number is reduced to one.<sup>13–18</sup> For example, bulk WSe<sub>2</sub> is a p-type semiconductor with an indirect band gap of ~1.2 eV, whereas its monolayer exhibits a direct band gap of ~1.65 eV.<sup>17,19</sup> The direct band gap of atomically thin TMDs can offer exciting opportunities for potential applications in both digital electronic and optoelectronic devices.<sup>20–24</sup> For example, it has been recently reported that exfoliated monolayer WSe<sub>2</sub> can be used to create a high performance p-type field-effect transistor (FET).<sup>25</sup> However, the size of the monolayer materials obtained from mechanical exfoliation method is limited in a few to a few tens of micrometers. It has also been recently reported that a chemical vapor deposition approach can be used to grow WSe<sub>2</sub> atomic flakes, but only in separated domains with the domain size on the order of 10 μm.<sup>26–28</sup> Therefore, the preparation of large-

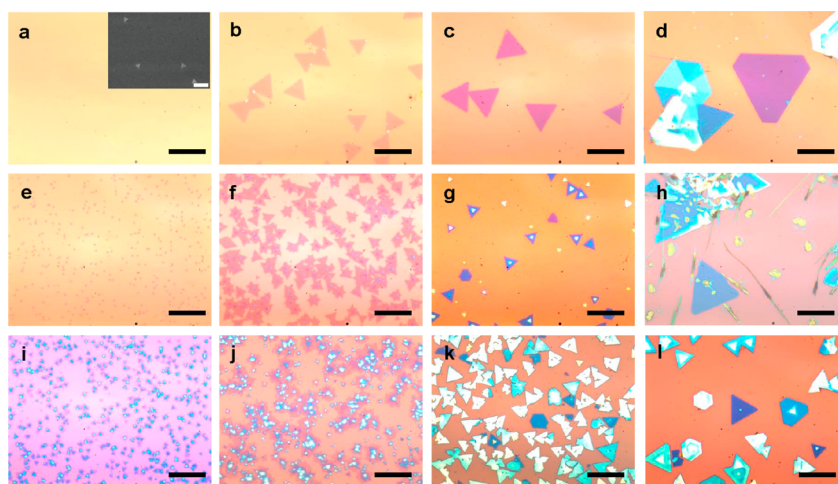
area monolayer WSe<sub>2</sub> film is essential for practical applications yet remains a significant challenge.

Here, we report a systematic study of chemical vapor deposition approach for the preparation of large area atomically thin WSe<sub>2</sub> films directly on SiO<sub>2</sub>/Si substrates with areal size of monolayer WSe<sub>2</sub> film up to 1 cm<sup>2</sup>. Microphotoluminescence mapping demonstrates distinct layer-number dependent photoluminescence, with the monolayer area exhibit much stronger emission than bilayer or multilayers. The transmission electron microscopy (TEM) studies reveal excellent crystalline quality of the atomically thin WSe<sub>2</sub> and electrical transport studies further demonstrate that the p-type WSe<sub>2</sub> field-effect transistors exhibit excellent electronic characteristics with hole carrier mobility up to 100 cm<sup>2</sup> V<sup>-1</sup> s<sup>-1</sup> for monolayer and up to 350 cm<sup>2</sup> V<sup>-1</sup> s<sup>-1</sup> for few-layer materials, comparable or well above that of previously reported mobility values or the synthetic WSe<sub>2</sub> and comparable to the best exfoliated materials.<sup>26,29–31</sup>

The growth processes are performed in a homemade tube furnace with detailed growth procedures described in the

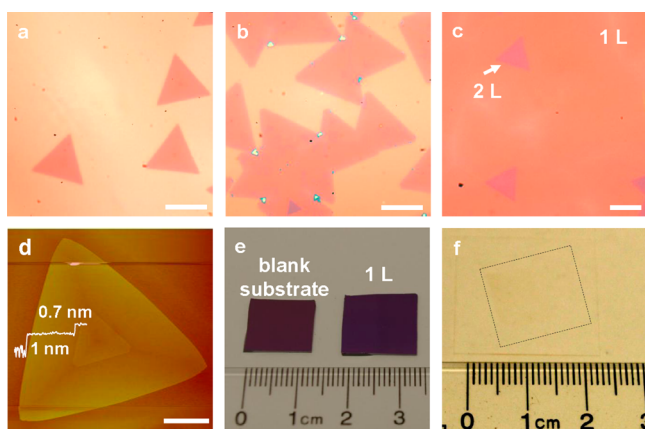
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**Figure 1.** Reactor-conditions-dependent WSe<sub>2</sub> growth with varying temperature and flow rate of argon. Optical microscope images of WSe<sub>2</sub> samples grown at different temperature under designed flow rate for 20 min: (a) 750 °C, 100 sccm (inset: SEM image of same size sample, indicating high density but small nucleations (~300 to 500 nm); scale bar, 5 μm); (b) 765 °C, 100 sccm; (c) 780 °C, 100 sccm; (d) 795 °C, 100 sccm; (e) 750 °C, 150 sccm; (f) 765 °C, 150 sccm; (g) 780 °C, 150 sccm; (h) 795 °C, 150 sccm; (i) 750 °C, 200 sccm; (j) 765 °C, 200 sccm; (k) 780 °C, 200 sccm; (l) 795 °C, 200 sccm. All of the scale bars are 20 μm.

Experimental section in Supporting Information. Briefly, the WSe<sub>2</sub> powders are placed in an alumina boat at the center of a quartz tube inside a one-inch tube furnace, and the clean SiO<sub>2</sub>/Si substrates are used as the growth substrate for the deposition of WSe<sub>2</sub> atomic layers at the downstream end with variable substrate temperature. Argon is continuously supplied through the reactor with designed flow rate as the carrier gas. We have first conducted systematic studies to investigate the effect of substrate temperature and flow rate of carrier gas with a constant source temperature at 1060 °C (Figure 1). Most of the WSe<sub>2</sub> domains exhibit a triangular shape as shown by the optical microscope images in Figure 1b, c and Figure 2a–c, and some of them show a hexagonal shape as shown in Figure 1d, g,



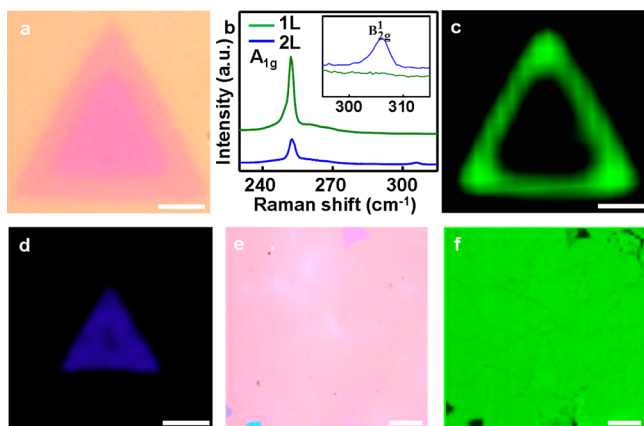
**Figure 2.** Time dependent growth of large area monolayer WSe<sub>2</sub> single crystal domains. (a) Typical optical microscope images of the monolayer WSe<sub>2</sub> domains taken after 20 min of growth, (b) 30 min of growth, and (c) 40 min of growth. (d) Atomic force microscope image of a WSe<sub>2</sub> monolayer and bilayer domains and their line scan profile. (e) Photograph of the ~1 cm sized monolayer WSe<sub>2</sub> films obtained with 40 min growth time. Left: blank 300 nm SiO<sub>2</sub>/Si substrate. Right: fully covered WSe<sub>2</sub> monolayer on blank 300 nm SiO<sub>2</sub>/Si substrate. (f) Photograph of the ~1 cm sized monolayer WSe<sub>2</sub> films transferred onto a glass template by chemical etching of SiO<sub>2</sub> layers. Scale bars of panels a–d are all 5 μm.

and l. In general, with a fixed source temperature, the lower substrate temperature typically results in a higher nucleation density, and a higher flow rate of the carrier gas produces a similar effect due to higher supersaturation of the precursors over the substrate surface. On the other hand, the higher substrate temperature promotes the nucleation of the extra atomic layers to produce multilayers.

At lower substrate temperature of 750 °C with low flow rate of 100 sccm, there are no visible WSe<sub>2</sub> domains observed on the SiO<sub>2</sub>/Si substrates under optical microscope. However, after analyzing the same sample by using scanning electron microscopy (SEM), very small nucleations (~300 nm of average edge size) were observed with nucleation density of ~4200/mm<sup>2</sup>. By increasing the substrate temperature to 765 °C, the monolayer WSe<sub>2</sub> domains (~10 μm of average edge size) start to appear with a nucleation density of ~1060/mm<sup>2</sup> (Figure 1b). When the growth temperature reaches 780 °C, the bilayer WSe<sub>2</sub> domains (~20 μm of average edge size) start to appear with an overall lower domain density reduced to ~350/mm<sup>2</sup> (Figure 1c). As the growth temperature is further increased to 795 °C, even thicker and larger flakes (~50 μm of average edge size) appear with even lower domain density of ~280/mm<sup>2</sup> (Figure 1d). Overall, higher growth temperature yields a lower density of thicker WSe<sub>2</sub> domains with a larger domain size. By increasing the flow rate of the carrier gas, both the layer number and the domain density of the WSe<sub>2</sub> are increased. For instance, at growth temperature of 765 °C, the nucleation density is ~1060/mm<sup>2</sup> under 100 sccm flow rate of carrier gas, which increases to ~11 000/mm<sup>2</sup> under 150 sccm, and ~23 000/mm<sup>2</sup> under 200 sccm. All of our observations are consistent with the nucleation model of the vapor phase deposition developed by W. K. Burton and N. Cabrera, where they predict that the nucleation probability is proportional to the supersaturation and inversely proportional to the substrate temperature.<sup>32</sup>

Based on the above studies, we have identified an optimized condition for the large area growth of WSe<sub>2</sub> monolayers at a substrate temperature of 765 °C and 100 sccm of carrier gas. Figure 2a shows the OM images of typical triangular WSe<sub>2</sub> monolayers after 20 min growth with an average edge length of

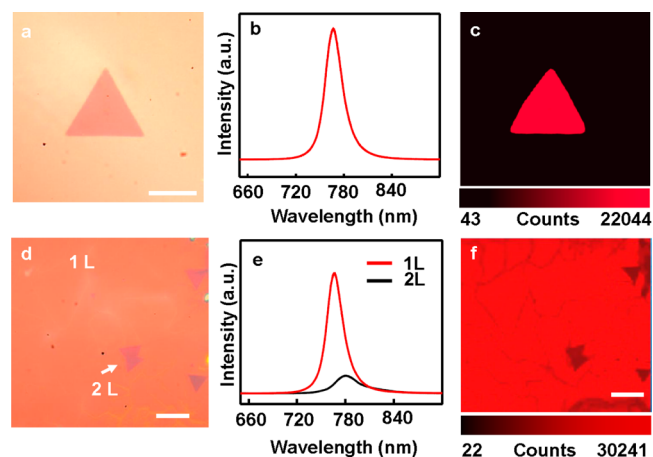
approximately 5  $\mu\text{m}$ . With the continued growth, the monolayer domains starts to merge together at 30 min, with bilayer and few-layer domains occasionally appeared on their first layer (Figure 2b). After 40 min growth, the WSe<sub>2</sub> monolayer domains are completely merged together to form a continuous monolayer, with a few second layer triangular domains seen with different optical contrast (Figure 2c). In this case, the SiO<sub>2</sub>/Si substrates are completely covered by the monolayer WSe<sub>2</sub> domains with less than 5% areal coverage of the bilayer. The number of WSe<sub>2</sub> atomic layers is also determined by AFM measurements (Figure 2d). The AFM step height of WSe<sub>2</sub> monolayer on the SiO<sub>2</sub> substrate is typically measured between 0.7–1.0 nm, and the step heights of the second layer on monolayer is around  $\sim 0.7$  nm (line scan in Figure 2d), which is consistent with the published reports of exfoliated WSe<sub>2</sub>.<sup>14,15</sup> The larger step height observed in the first layer is commonly seen in graphene or other layered materials such as MoSe<sub>2</sub>.<sup>16</sup> The lateral size of the resulted continuous monolayer WSe<sub>2</sub> film is as large as 1 cm<sup>2</sup> and is only limited by the size of the furnace (Figure 2e). The large area monolayer WSe<sub>2</sub> film can be readily transferred onto another substrate such as the glass (Figure 2f). We have further investigated the layer number dependent morphological and optical properties of the resulting atomically thin WSe<sub>2</sub> films. To this end, we have employed the  $\mu$ -Raman to study the monolayer/bilayer WSe<sub>2</sub> domains. The bilayer WSe<sub>2</sub> domain shows a triangular shape, which prefers to crystallize from the center of the first layer. The layer number of the WSe<sub>2</sub> domains is further confirmed using  $\mu$ -Raman studies. The Raman spectrum of monolayer region shows a single peak at 252 cm<sup>-1</sup> (green line in Figure 3b), corresponding to the A<sub>1g</sub> resonance mode of WSe<sub>2</sub>; and that of bilayer area shows an additional small peak at 307 cm<sup>-1</sup> (red line in Figure 3b), corresponding to the B<sub>2g</sub> resonance mode of WSe<sub>2</sub>. In general, the B<sub>2g</sub> signature mode is only active on the bilayer or few-layer region, which could reflect the presence of the additional interlayer interaction.<sup>14,15</sup>



**Figure 3.** Micro-Raman investigation of the monodomain WSe<sub>2</sub>. (a) Optical microscope image of a typical monolayer WSe<sub>2</sub> with bilayer domain on the center. (b) The Raman spectra of the monolayer and bilayer WSe<sub>2</sub>. (c) and (d) Typical Raman map of the single domain monolayer (c, center wavenumber:  $\sim 252$  cm<sup>-1</sup>) with bilayer (d, center wavenumber:  $\sim 307$  cm<sup>-1</sup>) WSe<sub>2</sub> domain. (e) Optical microscope image of large scale monolayer WSe<sub>2</sub> with several bilayer or few-layer domain. (f) Raman map of the large scale monolayer (center wavenumber:  $\sim 250$  cm<sup>-1</sup>) WSe<sub>2</sub> with several bilayer and few-layer domain. Scale bars of panels a, c, and d are all 5  $\mu\text{m}$ ; Scale bars of panels e and f are 10  $\mu\text{m}$ .

In contrast, the Raman A<sub>1g</sub> mode of WSe<sub>2</sub> is less sensitive to layer thickness, only with the intensity increasing with reducing atomic layers. The corresponding Raman map recorded at A<sub>1g</sub> mode can be further used to determine the layer number of the WSe<sub>2</sub> (Figure 3c–f). The darker triangular region on the Raman map of A<sub>1g</sub> mode corresponds to the bilayer or few-layer WSe<sub>2</sub> domain. The Raman mapping shows nearly the same color contrast throughout the monolayer and bilayer or few-layer area, indicating a uniform crystal quality.

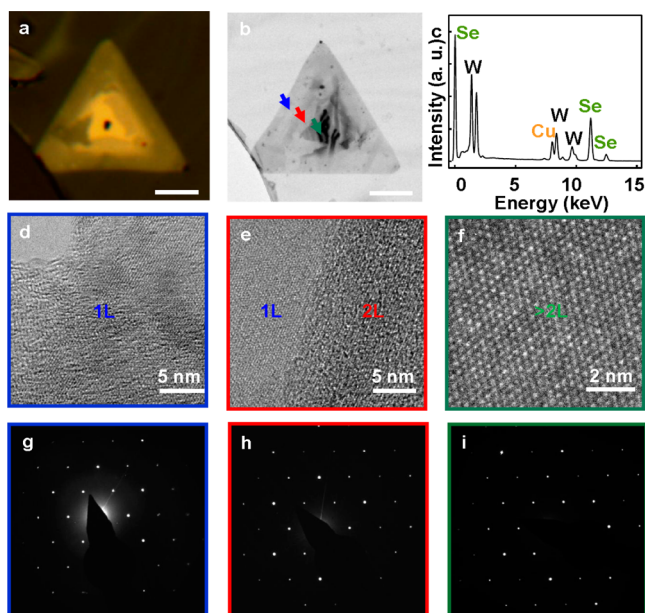
The optical properties of the monolayer WSe<sub>2</sub> domains and the continuous WSe<sub>2</sub> sheets were further investigated using microphotoluminescence ( $\mu$ -PL). The WSe<sub>2</sub> monodomain with single layer shows the PL peak located at approximately 767 nm, with full-width-half-maximum (FWHM) values of 25 nm (Figure 4b), which is comparable to the published data for



**Figure 4.** Optical properties of the single domain and fully covered WSe<sub>2</sub> monolayers. (a) Optical microscope image of a typical monolayer WSe<sub>2</sub>. Scale bar is 5  $\mu\text{m}$ . (b) PL spectra of the monolayer WSe<sub>2</sub> and PL map (c) of WSe<sub>2</sub> single domain showed in panel a. (d) Optical microscope image of the full covered monolayer WSe<sub>2</sub> with some bilayer domains. Scale bar is 10  $\mu\text{m}$ . (e) PL spectra of the monolayer and bilayer WSe<sub>2</sub>. (f) Corresponding PL map of the same region taken at panel d. Scale bar is 10  $\mu\text{m}$ .

exfoliated WSe<sub>2</sub>.<sup>14,15</sup> The corresponding  $\mu$ -PL map of the monolayer WSe<sub>2</sub> single crystal domain shows a very uniform contrast (Figure 4c), indicating the high crystalline quality and uniformity of the as-grown WSe<sub>2</sub> atomic layers. We have also conducted  $\mu$ -PL studies on the continuous WSe<sub>2</sub> film that are merged together (Figure 4d). The  $\mu$ -PL spectra taken of the monolayer region show the characteristic peak around 766 nm (red curve in Figure 4e). The strong light emission from monolayer indicates the high quality of the continuous WSe<sub>2</sub> films. The  $\mu$ -PL spectra taken at bilayer WSe<sub>2</sub> region (the darker triangular area on the OM image in Figure 4d) shows a wider peak at  $\sim 790$  nm with significantly lower intensity (black curve in Figure 4e). We have further conducted  $\mu$ -PL mapping studies over a relatively large area ( $\sim 60 \times 60$   $\mu\text{m}$ ) to evaluate the overall quality of the material (Figure 4f). Importantly, the PL mapping shows rather uniform emission across the entire film, indicating that the film is largely consisted of monolayer crystals. There are a few darker triangles in the PL mapping image, corresponding to the bilayer region. Additionally, it is also noted that there are some slightly darker lines, which can be attributed to the grain boundaries between merged monolayer domains.

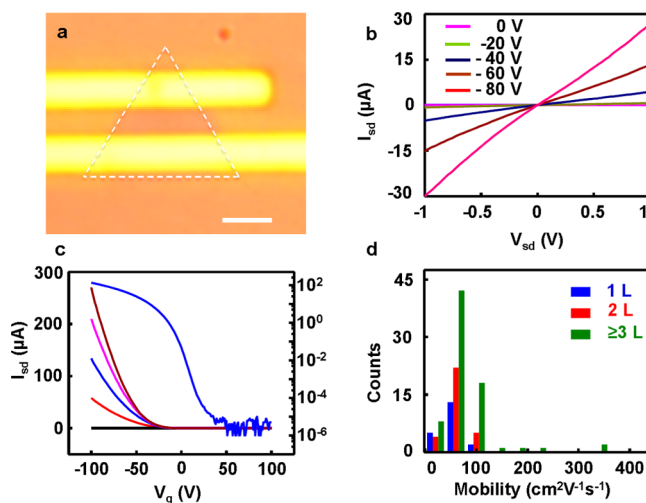
To further evaluate the atomic structure of the WSe<sub>2</sub> atomic layers, we have performed high resolution transmission electron microscopy (HRTEM) and electron diffraction (ED) studies. To this end, the as-grown WSe<sub>2</sub> crystals were transferred onto a carbon-coated TEM grid. Figure 5a is the optical microscopy



**Figure 5.** Crystalline structure characterization. (a) Optical microscope image of a typical monolayer WSe<sub>2</sub> with domain of few layers on the center. Scale bar is 10  $\mu\text{m}$ . (b) Low magnification bright field TEM image of a typical transferred WSe<sub>2</sub> triangular crystal. (c) Energy dispersive X-ray spectra showing the ratio between W and Se obtained from the integrated peak area is approximately 1:2. (d–f) HRTEM images of the atomic structure of the WSe<sub>2</sub> monolayer (d), bilayer (e), and multilayers. These images were taken on the regions indicated on panel b by the arrows with blue, red, and dark cyan colors, respectively. Scale bars are 5 nm (d,e) and 2 nm (f). (g–i) SAED pattern of the monolayer (g), bilayer (h) and few-layer (i) WSe<sub>2</sub> with the zone axis of [0001], the 6-fold symmetry in the position of the diffraction spots demonstrates that the triangular monolayer is predominantly single crystal with hexagonal structures.

image. Figure 5b shows a low magnification TEM image of a typical transferred WSe<sub>2</sub> triangular crystal, which shows increasing contrast from the edge to the center, with three distinct regions corresponding to the monolayer, bilayer, and few-layer area. The energy dispersive X-ray studies demonstrate that the atomic ratio between W and Se is approximately 1:2 (Figure 5c), consistent with the expected stoichiometry. We have further conducted HRTEM on different regions of the same WSe<sub>2</sub> flake. Figure 5d–f show the HRTEM images of the atomic structure of the WSe<sub>2</sub> monolayer, bilayer, and multilayers, respectively. These images were taken on the regions indicated on Figure 5b by the arrows with blue, red, and dark cyan colors, respectively. The hexagonal lattice is clearly visible from each atomic resolution image, confirming the excellent crystalline quality of the atomic layered material. The selected-area electron diffraction (SAED) is used to characterize the crystal structure of the film. Figure 5g–i show the SAED patterns of the monolayer, bilayer, and few-layer WSe<sub>2</sub> with the zone axis of [0001]. The single set of diffraction spots with 6-fold symmetry demonstrates that the triangular monolayer is single crystals with hexagonal structures.<sup>33,34</sup>

We have further evaluated the electronic properties of the WSe<sub>2</sub> atomic layers. To this end, we have fabricated back-gated field-effect transistors (FETs) from the synthetic WSe<sub>2</sub> on the 300 nm SiO<sub>2</sub>/Si substrates (Figure 6a). The source and drain



**Figure 6.** Electronic properties of WSe<sub>2</sub> atomic layers. (a) Optical microscope image of a monolayer WSe<sub>2</sub> transistor; scale bar is 2  $\mu\text{m}$ . (b)  $I_{\text{sd}}-V_{\text{sd}}$  output characteristics of the WSe<sub>2</sub> transistor shown in panel a. (c)  $I_{\text{sd}}-V_{\text{g}}$  transfer characteristics of the device shown in (a) at  $V_{\text{sd}} = 0, 1, 2, 3,$  and  $4$  V. (d) Summary of the mobility values obtained in WSe<sub>2</sub> field effect transistors with different number of atomic layers, demonstrating a mobility range of  $10-350$   $\text{cm}^2 \text{V}^{-1} \text{s}^{-1}$  and maximum on/off ratio over  $10^8$ .

electrodes (100 nm Au) defined by the electron beam lithography (EBL) and deposited using thermal evaporation. More than 100 transistors were fabricated on WSe<sub>2</sub> monolayer, bilayer, and few-layer monodomains. The standard transistor measurements were conducted under ambient conditions to obtain the on-currents, carrier mobilities, and on/off ratios for all devices. Figure 6b shows the  $I_{\text{ds}}-V_{\text{ds}}$  output characteristic of a monolayer WSe<sub>2</sub> FETs at various gate voltages. The linear and symmetric curves suggested that ohmic contacts were formed at the source and drain electrodes. The  $I_{\text{ds}}-V_{\text{bg}}$  curve of the same device was measured at different drain bias from 0 to 4 V, with the back-gate voltage sweeping from  $-100$  to  $100$  V (Figure 6c). Additionally, the  $I_{\text{ds}}-V_{\text{bg}}$  curve of the typical WSe<sub>2</sub> monolayer, bilayer and few-layer FETs are shown in Supporting Information Figure S2. All these IV curves show typical p-type semiconductor characteristic, consistent with mechanically exfoliated WSe<sub>2</sub> materials. The highest mobility of the monolayer WSe<sub>2</sub> FETs is  $100$   $\text{cm}^2 \text{V}^{-1} \text{s}^{-1}$ . The measured on/off ratio of this device reaches a maximum value above  $1 \times 10^8$  for gate voltages swept in the range from  $-100$  to  $100$  V with a source-drain bias of 2 V, which is comparable to the best reported values for exfoliated samples, and greatly higher than synthetic samples reported previously. After analysis of all the fabricated WSe<sub>2</sub> FETs, we have plotted a histogram to show the mobility distribution for all devices (Figure 6d). Importantly, the highest mobility of the few-layer WSe<sub>2</sub> FETs can reach up to  $350$   $\text{cm}^2 \text{V}^{-1} \text{s}^{-1}$ , which represents the highest value observed in these atomically thin TMDs at room temperature,<sup>26,35–40</sup> further highlighting the high crystalline quality of these as-grown materials.

In summary, we have reported a systematic investigation on the chemical vapor deposition growth of large area p-type WSe<sub>2</sub>

atomic layers. By systematically tuning the synthetic parameters, we show that high quality monolayer WSe<sub>2</sub> thin film can be prepared over large area with the lateral dimensions up to ~1 cm<sup>2</sup>. Microphotoluminescence studies demonstrate uniform PL emission from the monolayer materials and the transmission electron microscopy studies confirm the excellent crystalline quality. Electrical transport studies further demonstrate the p-type WSe<sub>2</sub> back-gated field-effect transistors exhibit excellent electronic characteristics with hole mobility over 350 cm<sup>2</sup> V<sup>-1</sup> s<sup>-1</sup> and on/off ratio above 10<sup>8</sup>. The availability of high quality large area p-type atomically thin materials offers an important building block for the design of future van de Waals heterostructures for electronic and photonic devices.

## ■ ASSOCIATED CONTENT

### Supporting Information

Experimental details; Figures S1–S2. This material is available free of charge via the Internet at <http://pubs.acs.org>.

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### Notes

The authors declare no competing financial interest.

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