



Field effect properties of single-layer $\text{MoS}_{2(1-x)}\text{Se}_{2x}$ nanosheets produced by a one-step CVD process

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ABSTRACT

In recent years, interfacial doping with other atoms, molecules, and nanoparticles in molybdenum disulfide (MoS_2) has been proven as a new route to explore the potential application of 2D materials in microelectronic devices. In this paper, we utilized a one-step chemical vapor deposition approach to synthesize monolayer $\text{MoS}_{2(1-x)}\text{Se}_{2x}$ nanosheets in atmospheric pressure using MoO_3 , S, and Se powders as precursors. AFM and visible-light microscopy showed that the as-grown nanosheets were single layers, their surface was atomic flat, and the maximum grain size was over 100 μm . XPS characterization demonstrated that the concentration of selenium in $\text{MoS}_{2(1-x)}\text{Se}_{2x}$ nanosheets was affected by the amount of selenium powder in the doping process. The back-gate FETs were fabricated to investigate the electrical properties of monolayer $\text{MoS}_{2(1-x)}\text{Se}_{2x}$ nanosheets with different Se contents. The field effect properties of $\text{MoS}_{2(1-x)}\text{Se}_{2x}$ ($x = 0.65$) transistors indicated that a moderate mobility was achieved, and ohmic contact was obtained at the interface of the $\text{MoS}_{2(1-x)}\text{Se}_{2x}$ channel and metal electrodes. Characterization using high-resolution transmission electron microscopy showed that the microstructure of as-grown $\text{MoS}_{2(1-x)}\text{Se}_{2x}$ ($x = 0.65$) had a regular hexagonal lattice structure, which revealed that it was a single-crystalline two-dimensional material.

Introduction

As a member of two-dimensional transition metal dichalcogenides (TMDs) [1, 2], molybdenum disulfide (MoS_2) has been widely studied due to its unique

material properties [3–6]. Unlike graphene, which has no bandgap, molybdenum disulfide is a semiconductor and its bandgaps are around 1.2–1.8 eV depending on the number of layers. When MoS_2 is decreased from few layers to a single layer (around 0.65 nm), MoS_2 will change from an indirect semiconductor to a direct

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semiconductor [7, 8]. Due to its outstanding physical and chemical properties, MoS₂ has great prospects in the fields of microelectronics [9, 10], optoelectronics [11, 12], and energy storage [13–15]. In order to achieve the desired physical and chemical properties of MoS₂, some progress has been made in experimental and theoretical calculation of molybdenum disulfide doping [16–19]. Dolui et al. investigated the impact of substituted elements on the characteristics of MoS₂ by first-principles calculations [19]. Other studies have reported that the bandgap was clearly modulated when substituted elements such as Se or W were introduced during the preparations of MoS₂ [20–23]. However, there are still many major challenges in preparing the MoS_{2(1-x)}Se_{2x} alloy. For example, it is still hard to synthesize large-area and layer-controlled MoS_{2(1-x)}Se_{2x} nanosheets. The effect of the Se/S source ratio on the concentration and uniformity of substituted selenium in the samples has not been fully investigated. In addition, the performance of electrical devices fabricated by MoS_{2(1-x)}Se_{2x} is still not optimal.

Here, we reported the synthesis of large-area MoS_{2(1-x)}Se_{2x} monolayers by one-step atmospheric pressure chemical vapor deposition (APCVD). During the preparation process, the ratio of selenium and sulfur source not only determined the elemental composition of the samples, but also affected the grain size of the MoS_{2(1-x)}Se_{2x} monolayers. The content and distribution of selenium in the triangular MoS_{2(1-x)}Se_{2x} domains were characterized by XPS and EDX spectra. The central positions and intensities of Raman peaks corresponding to MoS_{2(1-x)}Se_{2x} nanosheets with different Se contents clearly demonstrated variation of vibration modes in the alloy. High-resolution transmission electron microscopy (HRTEM) images demonstrated that as-grown samples were single crystals. To measure the electrical properties, synthesized MoS_{2(1-x)}Se_{2x} nanosheets were used as channels in field effect transistors. With different Se content, these exquisite devices showed promising electrical performances. Our research provides the possibility for the application of MoS_{2(1-x)}Se_{2x} in the micro–nano–electronic field.

Results and discussion

MoS_{2(1-x)}Se_{2x} nanosheets were grown on SiO₂/Si substrates, using a three-temperature-zone tube furnace (Fig. 1a). Due to the different melting points,

selenium powders and sulfur powders were placed separately in different upstream regions. During the experiment, sulfur and selenium vapor reacted with MoO₃ after being transported by the carrier gas in downstream zone III to where the substrates were located. Here, the carrier gas was a mixture gas of hydrogen and argon (8% H₂). As a reduction agent, the introduced H₂ could convert MoO₃ to MoO_{3-x}, which facilitated the further reaction (see Supporting Information (SI): SI-1).

The images from the visible-light microscope in Fig. 1b–f showed the isolated triangular domains of MoS_{2(1-x)}Se_{2x} nanosheets, which ranged in size from a few microns to more than 100 μm. The grain size of the samples was closely related to the ratio of selenium and sulfur powders. The total amount of selenium and sulfur powders used in the experiments was 20 mg. As the ratio of selenium powder increased, the size of MoS_{2(1-x)}Se_{2x} triangular domains clearly decreased (Fig. 1b–f). The major reason for this phenomenon is that the chemical activity of sulfur is stronger than selenium, and thus crystallization of the MoS_{2(1-x)}Se_{2x} nanosheets is more difficult at the Se-rich reaction process compared to the S-rich atmosphere [24–26]. Topography of MoS_{2(1-x)}Se_{2x} nanosheets was characterized by an atomic force microscope (AFM). The triangular domain in Fig. 1g had a homogenous contrast and no obvious particles, which revealed that the surface of the prepared sample was clean and atomic flat. The white curve in Fig. 1g was the cross-sectional scan of the sample (marked by a red line), and its height was ~ 0.8 nm, indicating that the MoS_{2(1-x)}Se_{2x} nanosheet was a single layer [27]. In the process of MoS_{2(1-x)}Se_{2x} growth, the amount of hydrogen flow was the main factor to obtain these monolayer samples (see SI: Fig. S1 and Fig. S2).

The chemical states and Se contents in MoS_{2(1-x)}Se_{2x} nanosheets were confirmed by X-ray photoelectron spectroscopy (XPS). Figure 2a showed that the S 2p had two peaks at ~ 162.8 and ~ 164.0 eV, which were attributed to S 2p^{3/2} and S 2p^{1/2}, respectively. The binding energy of Se 3p^{3/2} was observed at ~ 161.2 eV. According to the above-mentioned results, *x* in MoS_{2(1-x)}Se_{2x} could be calculated by the following formula:

$$S/Se = (I_S \times F_{Se}) / (I_{Se} \times F_S) \quad (1)$$

where *I_S* and *I_{Se}* were the areas under the peaks of S 2p^{3/2} and Se 3p^{3/2}, respectively; and *F_S* and *F_{Se}*

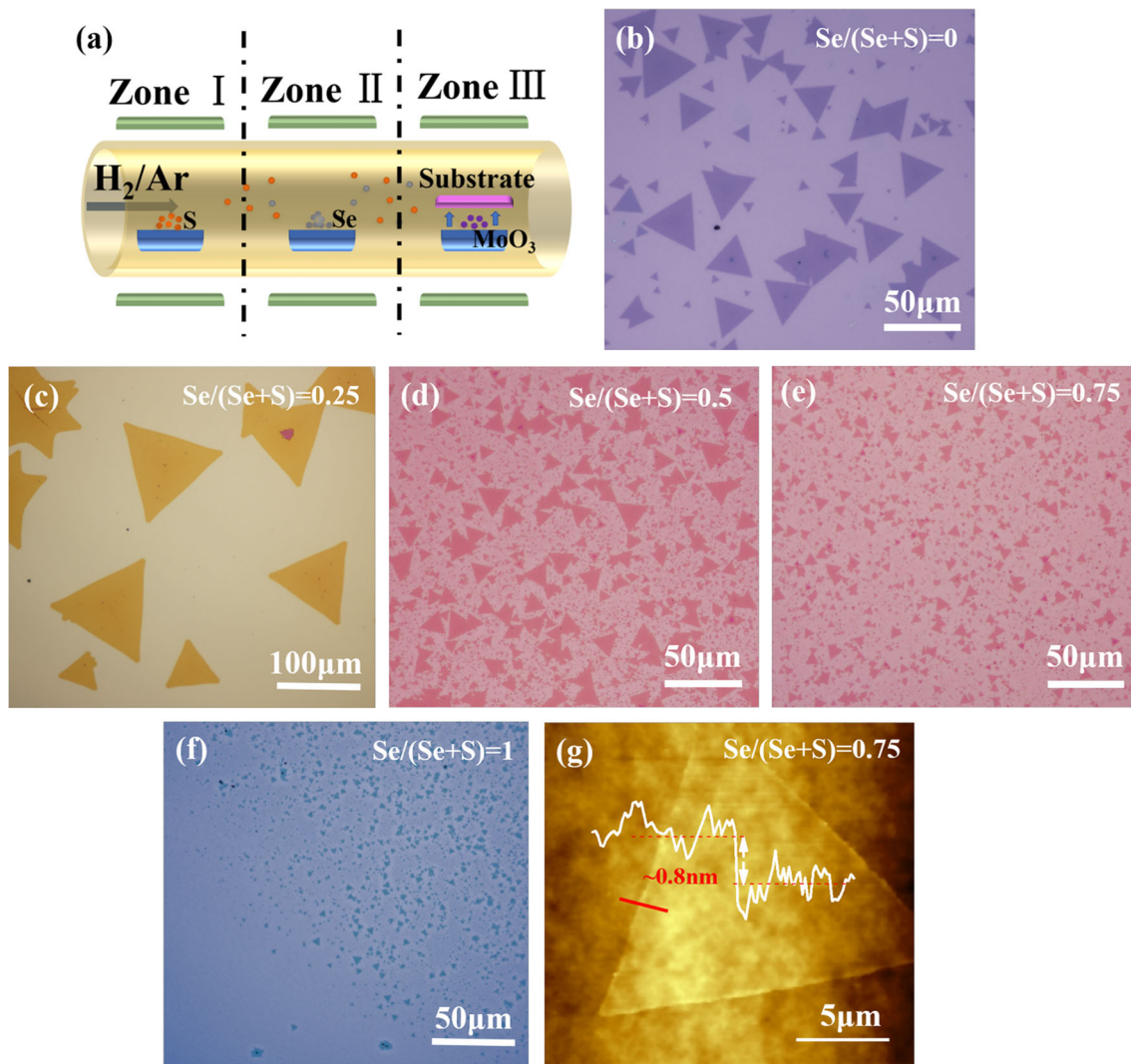


Figure 1 **a** Schematic illustration for the growth of monolayer $\text{MoS}_{2(1-x)}\text{Se}_{2x}$ nanosheets. **b–f** Visible-light microscope images of as-grown $\text{MoS}_{2(1-x)}\text{Se}_{2x}$ nanosheets with different ratios of

selenium and sulfur powders. **g** AFM images of monolayer triangular $\text{MoS}_{2(1-x)}\text{Se}_{2x}$ nanosheets.

represented the relative symmetric factors for $\text{S } 2p^{3/2}$ (0.4453) and $\text{Se } 3p^{3/2}$ (0.8493), respectively [28]. It was notable that x in $\text{MoS}_{2(1-x)}\text{Se}_{2x}$ went up as the ratio of selenium powders rose during the experiments (Fig. 2b). With increase in the Se content in $\text{MoS}_{2(1-x)}\text{Se}_{2x}$ nanosheets, the peak of $\text{Se } 3p^{3/2}$ was obvious, while the intensities of $\text{S } 2p^{3/2}$ and $2p^{1/2}$ became weak and begin to disappear. The peaks at ~ 229.7 and ~ 232.8 eV corresponded to $\text{Mo}^{4+} 3d^{5/2}$ and $\text{Mo}^{4+} 3d^{3/2}$, respectively (Fig. 2c), in agreement with previously reported values [7]. There was no obvious peak of $\text{Mo}^{6+} 3d^{5/2}$ at ~ 236 eV, indicating that MoO_3 was completely reduced to $\text{MoS}_{2(1-x)}\text{Se}_{2x}$. The doublet peaks of $\text{Se } 3d^{5/2}$ and $\text{Se } 3d^{3/2}$ were evident at ~ 55.2 and ~ 56.1 eV, as long

as selenium was contained in the nanosheets (Fig. 2d) [29].

Raman spectroscopy was an effective method to analyze the phonon vibration mode properties of the TMDs. While $\text{MoS}_{2(1-x)}\text{Se}_{2x}$ was produced from APCVD, the two sets of composition-dependent peaks were obtained in the Raman spectra [30], corresponding to an MoSe_2 -related feature at low frequency ($200\text{--}300\text{ cm}^{-1}$) and an MoS_2 typical feature at high frequency ($350\text{--}410\text{ cm}^{-1}$) (Fig. 3a). There were two typical peaks of MoS_2 centered at $\sim 401.1\text{ cm}^{-1}$ (E_{2g}^1) and $\sim 380.5\text{ cm}^{-1}$ (A_{1g}), which were attributed to in-plane vibration of S atoms and out-of-plane vibration of S and Mo atoms, respectively. The inset in Fig. 3b showed the schematic of

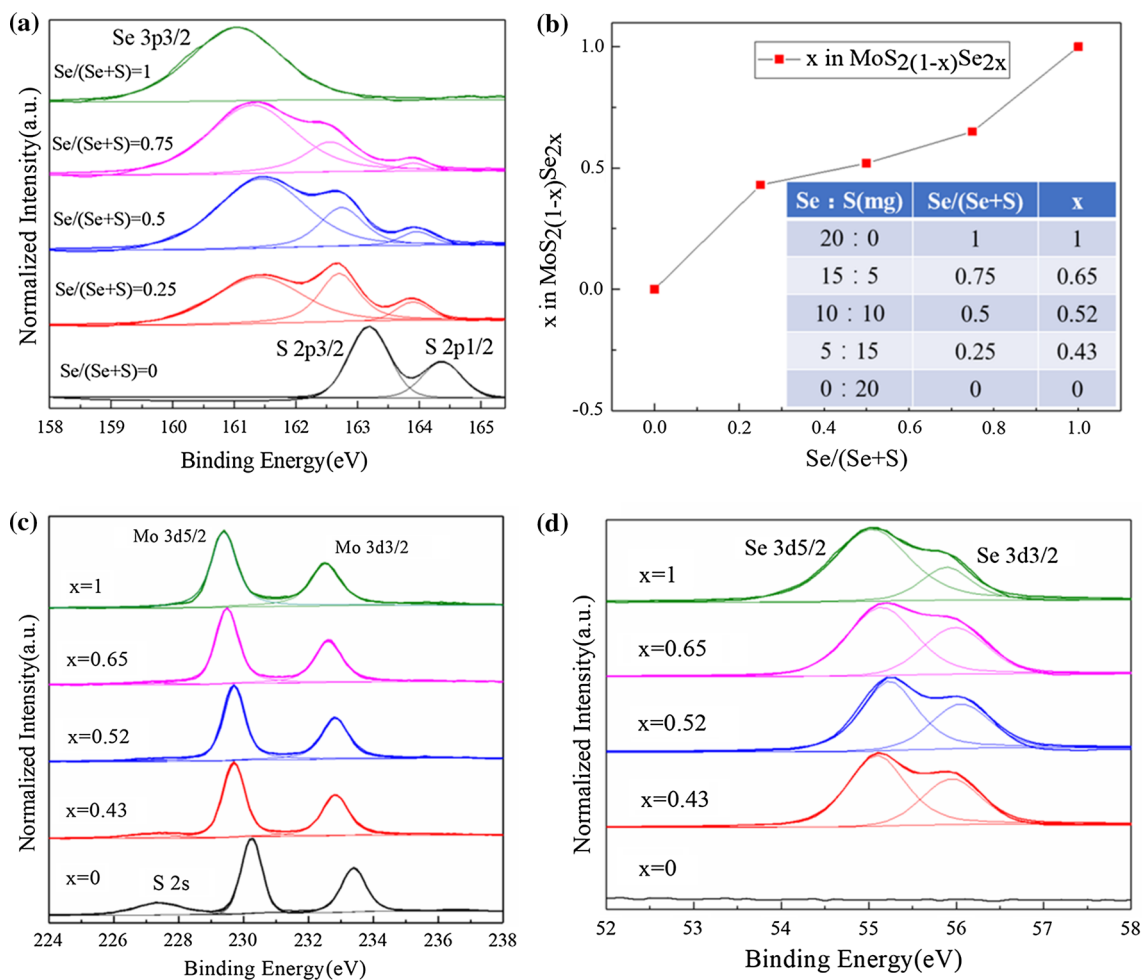
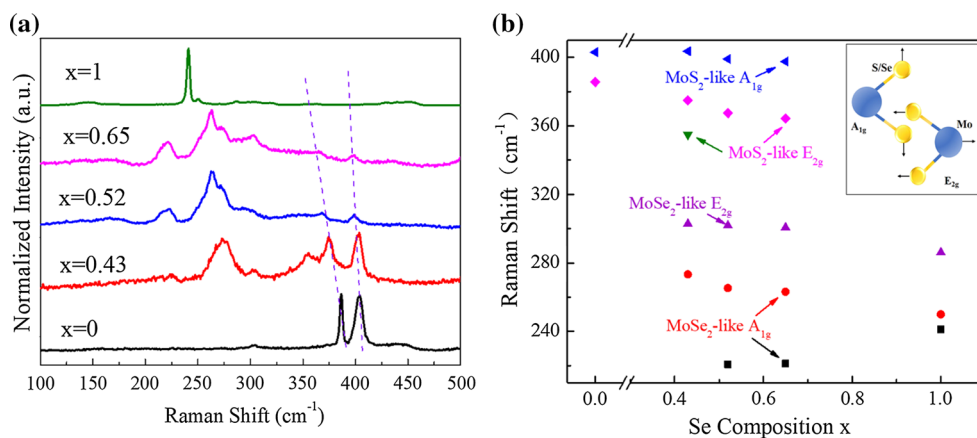


Figure 2 XPS spectra of $\text{MoS}_2(1-x)\text{Se}_{2x}$ monolayers. **a** 158–165 eV binding energy stage. **b** The curve of the x value in $\text{MoS}_2(1-x)\text{Se}_{2x}$ with the amount of selenium sources. **c** 224–238 eV binding energy stage. **d** 52–58 eV binding energy stage.

two vibration modes. These MoS_2 -like modes shifted to low frequency by increasing the Se content in $\text{MoS}_2(1-x)\text{Se}_{2x}$. This result suggested that the introduction of Se atoms distorted the Mo-S bonds and

softened the vibrations of E_{2g}^1 and A_{1g} modes of MoS_2 . As the Se atoms occupied the parts of sulfur sites, MoSe_2 -like peaks of monolayer $\text{MoS}_2(1-x)\text{Se}_{2x}$ appeared and became noticeable between 200 and

Figure 3 **a** Raman spectra of $\text{MoS}_2(1-x)\text{Se}_{2x}$ monolayers. **b** Plots of Raman shifts along with Se composition x in $\text{MoS}_2(1-x)\text{Se}_{2x}$ monolayer (inset is the schematic of A_{1g} and E_{2g}^1 modes).



300 cm⁻¹. When selenium was completely substituted for sulfur, the out-of-plane A_{1g} mode of MoSe₂ was identified at 241.1 cm⁻¹.

The field effect transistors (FETs) were fabricated by electron beam lithography (EBL) to demonstrate the electrical properties of the synthesized MoS_{2(1-x)}Se_{2x} monolayers with different Se contents ($x = 0, 0.43, 0.65$). 10 nm Ti and 100 nm Au were deposited as the electrodes of FETs by electron beam evaporation and magnetron sputtering, respectively. The $I_{ds}-V_{gs}$ transfer characteristic curves (Fig. 4a, c, e) suggested that all the MoS_{2(1-x)}Se_{2x} devices exhibited n-type transport behavior. The high I_{on}/I_{off} ratios of $\sim 10^7$ were extracted from semilogarithmic coordinates in the inset shown in Fig. 4a, c, e. Carrier mobility μ was calculated by the following formula: [3, 31]

$$\mu = [dI_{ds}/dV_{gs}] \times [L/(WC_{ox}V_{ds})] \quad (2)$$

where L and W were channel length of 2 μm and width of 1 μm , respectively; and C_{ox} was the SiO₂ gate capacitance, which was estimated to be approximately $1.16 \times 10^{-8} \text{ Fcm}^{-2}$. As Se contents rose from $x = 0$ to $x = 0.65$, carrier mobility of devices

gradually improved from ~ 0.12 to $\sim 3.72 \text{ cm}^2 \text{ V}^{-1} \text{ s}^{-1}$. The much lower carrier mobility of $x = 0$ and $x = 0.43$ was possibly attributed to the following reasons: first, poor contact existed between MoS_{2(1-x)}Se_{2x} channels and the Au electrodes, causing serious contact resistance. Second, introduced Se caused inevitable lattice distortion or defects in the MoS_{2(1-x)}Se_{2x} samples reducing the efficiency of carrier transport. Third, organic or inorganic impurity derived from the process of fabricating and testing FETs might have resulted in the degradation of device performance. The contact between the channel and electrodes could be characterized by $I_{ds}-V_{ds}$ curves (Fig. 4b, d, f). The contact of $x = 0$ and $x = 0.43$ belonged to the Schottky contact (Fig. 4b, d), but when $x = 0.65$, the linear curves in Fig. 4f indicated that ohmic contact was formed between the MoS_{2(1-x)}Se_{2x} channel and the Au electrodes. The ohmic contact improved the transport of carrier mobility in monolayer MoS_{2(1-x)}Se_{2x} ($x = 0.65$) FETs. Another reason for obtaining the best electrical properties in $x = 0.65$ samples was that the bandgap of MoS_{2(1-x)}Se_{2x} decreased with the rise in selenium contents [32], which made it easier for carriers to be

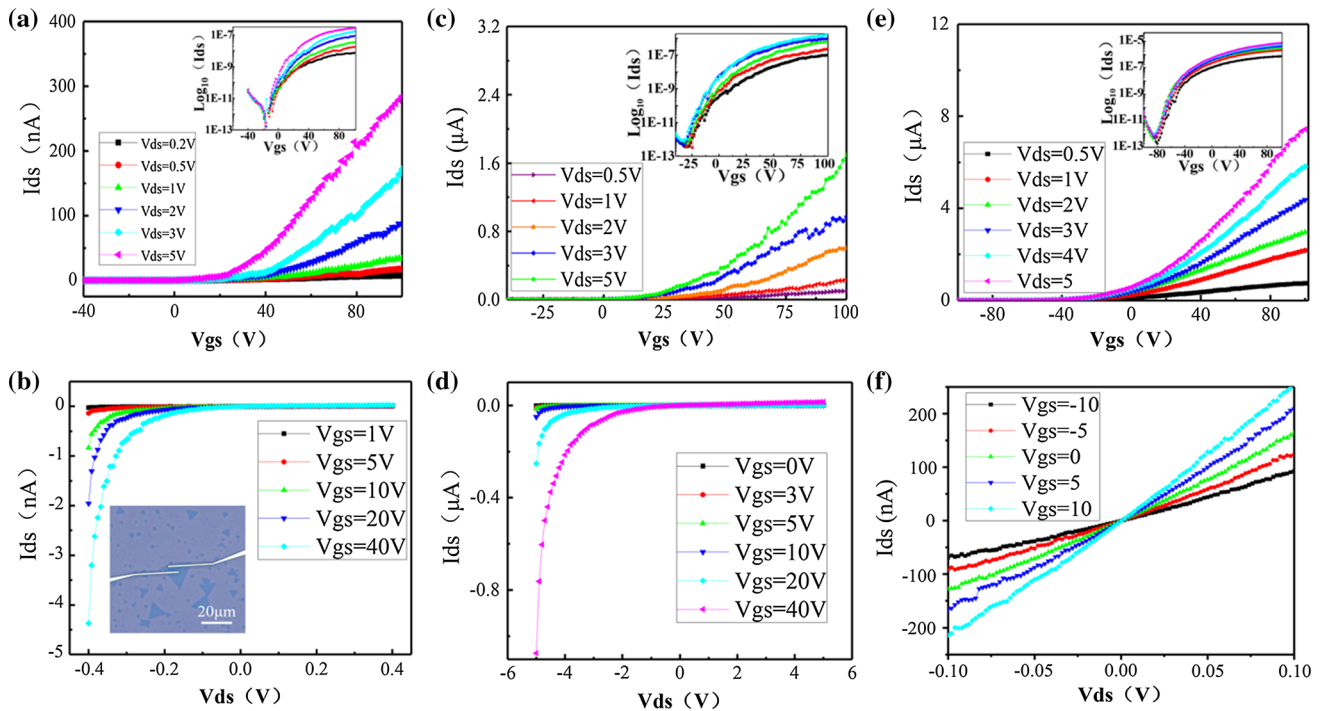


Figure 4 a, c, e The source-drain current to the gate voltage ($I_{ds}-V_{gs}$) and b, d, f the source-drain current to the source-drain voltage ($I_{ds}-V_{ds}$) for FETs in which the MoS_{2(1-x)}Se_{2x} nanosheets were used as channels. a, b for $x = 0$; c, d for $x = 0.43$; e, f for

$x = 0.65$. The insets of a, c, e were semilogarithmic coordinates of $I_{ds}-V_{gs}$ curves and the inset of b was the visible-light microscope image of the FETs.

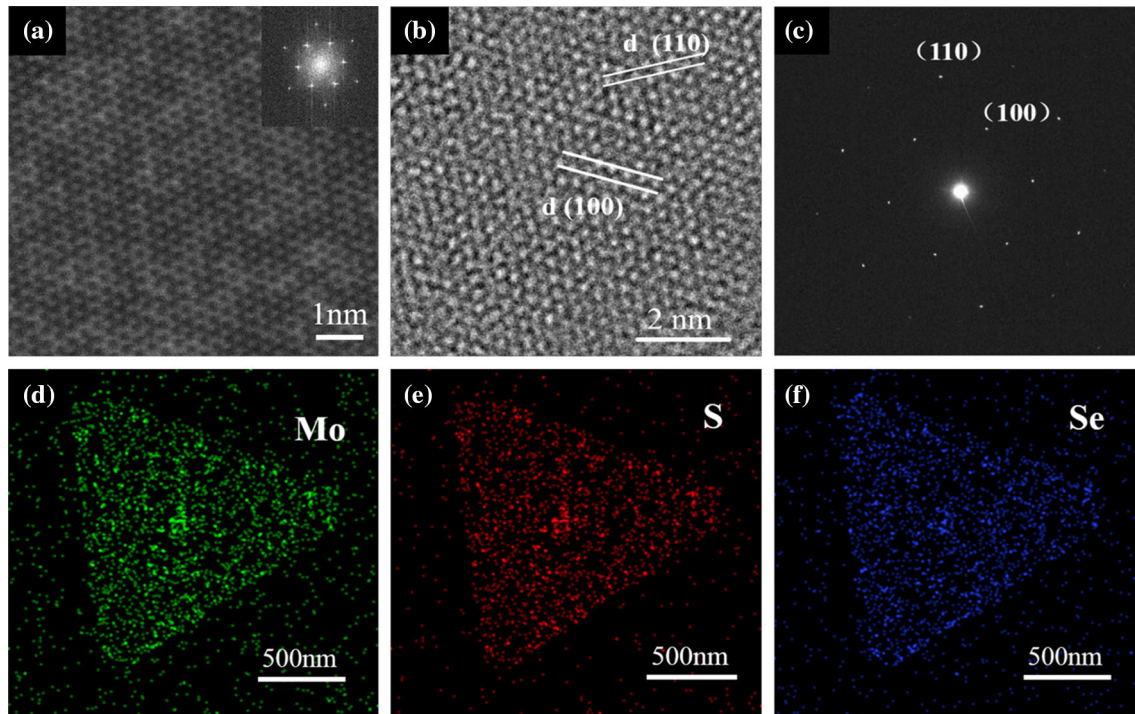


Figure 5 **a** HAADF-STEM image of the $\text{MoS}_{2(1-x)}\text{Se}_{2x}$ nanosheet ($x = 0.65$). **b** High-resolution TEM images of the $\text{MoS}_{2(1-x)}\text{Se}_{2x}$ nanosheet ($x = 0.65$). **c** The bright spots in selected

area electron diffraction (SAED) pattern reveal that the sample is a single crystal. EDX mappings of **d** Mo, **e** S and **f** Se in the $\text{MoS}_{2(1-x)}\text{Se}_{2x}$ nanosheet.

exciting from the top of the valence band to the conduction band, thus improving the electrical properties. To further promote the performance of monolayer $\text{MoS}_{2(1-x)}\text{Se}_{2x}$ FETs, elaborating the high- k top-gating [33] and tuning the contact between channels and electrodes [34] should be considered in future research.

In order to further explore the microstructure of the monolayer $\text{MoS}_{2(1-x)}\text{Se}_{2x}$ nanosheets ($x = 0.65$) with optimal electrical properties, transmission electron microscopy (TEM) combined with energy-dispersive X-ray spectroscopy (EDX) was conducted to investigate its lattice structure and element distribution. In the image of atomic-resolution high-angle annular dark-field (HAADF) scanning transmission electron microscopy (STEM) (Fig. 5a), different atoms could be identified by Z contrast ($Z = \text{atomic number}$) [20]. The Mo sites were brighter than most bi-chalcogen (X_2 , $X = \text{S, Se}$) sites because the atomic number of Mo (42) was larger than those of Se (34) and S (16). Figure 5a revealed that Se existed in the lattice of MoS_2 in the form of substitution doping. Two predominant lattice spacings measured from the high-resolution TEM image (Fig. 5b) were ~ 0.28 nm and ~ 0.16 nm, representing (100) and (110) planes, respectively

[35, 36]. This result was consistent with selected area electron diffraction (SAED) patterns (Fig. 5c) which confirmed that the sample was the single crystal of a hexagonal structure [37]. EDX mapping was used to characterize the distribution of the elements in the samples (and EDX spectrum of $\text{MoS}_{2(1-x)}\text{Se}_{2x}$ could be seen in SI: Fig. S3). The identical and similar triangles of Fig. 5d–f indicated the ideal element uniformity in the as-grown $\text{MoS}_{2(1-x)}\text{Se}_{2x}$ monolayer. Combined with images of high-resolution TEM and EDX mapping, $x = 0.65$ of samples had no fatal defects in the lattice arrangement, and the distribution of elements in single samples was homogeneous. Single-crystalline microstructures were important reasons that $\text{MoS}_{2(1-x)}\text{Se}_{2x}$ ($x = 0.65$) could obtain distinctive electrical properties.

Conclusion

To summarize, we have synthesized the triangular and uniform domains of $\text{MoS}_{2(1-x)}\text{Se}_{2x}$ monolayer nanosheets by a simple APCVD approach. The impact of the ratio of selenium and sulfur powder on the size and Se contents of $\text{MoS}_{2(1-x)}\text{Se}_{2x}$ was

investigated in detail. XPS confirmed that the Se contents in $\text{MoS}_{2(1-x)}\text{Se}_{2x}$ nanosheets increased with increasing selenium powders. The electrical properties of the samples with different selenium contents were also discussed in this paper. The results showed that optimal electrical properties were obtained when the Se concentration of the nanosheets reached the maximum ($x = 0.65$). According to the microstructure of $\text{MoS}_{2(1-x)}\text{Se}_{2x}$ ($x = 0.65$), HRTEM combined with EDX showed that the as-grown nanosheets were single crystals and the distribution of Mo, S and Se was uniform in the triangular domain. This work provides a feasible and convenient method to synthesize high-quality and large-area $\text{MoS}_{2(2-x)}\text{Se}_{2x}$ monolayers with controllable stoichiometry.

Experimental details

Growth of monolayer $\text{MoS}_{2(1-x)}\text{Se}_{2x}$ nanosheets

$\text{MoS}_{2(1-x)}\text{Se}_{2x}$ nanosheets were grown on SiO_2/Si substrates, utilizing a three-temperature-zone tube furnace. Sulfur and selenium powders were placed in upstream zone I (150 °C) and zone II (300 °C), respectively, due to their different melting points; the total amount of selenium and sulfur powders was 20 mg. Silicon oxide substrates were immersed in acetone and isopropanol for ultrasonic cleaning for 10 min as pretreatment. SiO_2 substrates dried by nitrogen were placed face down on ceramic boats with 10 mg MoO_3 powder in downstream zone III. The growth temperature of $\text{MoS}_{2(1-x)}\text{Se}_{2x}$ in zone III was about 700 °C to 800 °C. The carrier gas was a mixture of argon–hydrogen (8% H_2) gas with 65 sccm during growth.

Characterizations of monolayer $\text{MoS}_{2(1-x)}\text{Se}_{2x}$ nanosheets

The topography and thickness of as-grown $\text{MoS}_{2(1-x)}\text{Se}_{2x}$ nanosheets were characterized by visible-light microscopy (Olympus) and atomic force microscopy (AFM, 5600LS). X-ray photoelectron spectroscopy (XPS, Thermo Scientific Escalab 250Xi) was performed with a monochromatic Al $K\alpha$ X-ray source to investigate the elemental composition of the $\text{MoS}_{2(1-x)}\text{Se}_{2x}$ nanosheets. The phonon vibration mode properties of $\text{MoS}_{2(1-x)}\text{Se}_{2x}$ were characterized

by Raman spectrum (ThermoFisher DXR) at 532 nm excitation wavelength. HAADF-STEM (Titan Cubed Themis G2 300) was used to identify the atomic phase of $\text{MoS}_{2(1-x)}\text{Se}_{2x}$ nanosheets at 100 keV. High-resolution transmission electron microscopy combined with EDX spectrum (Talos F200X) was applied at 200 keV to characterize the crystalline structure of the $\text{MoS}_{2(1-x)}\text{Se}_{2x}$ nanosheets.

Fabrication and characterization of $\text{MoS}_{2(1-x)}\text{Se}_{2x}$ FETs

The source and drain electrodes of the monolayer $\text{MoS}_{2(1-x)}\text{Se}_{2x}$ FETs were fabricated by electron beam lithography. The electrode materials were 10 nm Ti and 100 nm Au deposited by electron beam evaporation and magnetron sputtering deposition, respectively. The back-gate electrodes were fabricated by etching p^{++} SiO_2 substrates with BOE solution. The electrical properties of the FETs were measured using a semiconductor parameter analyzer (Agilent B1500A).

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Compliance with ethical standards

Conflict of interest The authors declare that they have no conflict of interest.

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