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Selective Growth of WSe₂ with Graphene Contacts

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Abstract

Nanoelectronics of two-dimensional (2D) materials and related applications are hindered with critical contact issues with the semiconducting monolayers. To solve these issues, a fundamental challenge is selective and controllable fabrication of p-type or ambipolar transistors with a low Schottky barrier. Most p-type transistors are demonstrated with tungsten selenides (WSe₂) but a high growth temperature is required. Here, we utilize seeding promoter and low pressure CVD process to enhance sequential WSe₂ growth with a reduced growth temperature of 800 °C for reduced compositional fluctuations and high hetero-interface quality. Growth behavior of the sequential WSe₂ growth at the edge of patterned graphene is discussed. With optimized growth conditions, high-quality interface of the laterally stitched WSe₂-graphene is achieved and characterized with transmission electron microscopy (TEM). Device fabrication and electronic performances of the laterally stitched WSe₂-graphene are presented.

Keywords: Contacts, WSe₂, Electronics, Heterostructures, Interfaces

Introduction

Monolayer van der Waals materials, such as graphene and transition metal dichalcogenide (TMD), exhibit excellent electronic performances and atomically thick body without dangling bonds on the surface, which offers potential solutions for fundamental limit of channel materials in Moore's law, such as short channel effects and various challenges in the scaling [1, 2]. In the past decade, nanoelectronics of two-dimensional (2D) materials and related applications are highly hindered by critical contact issues with the semiconducting TMD monolayers due to significant Fermi level pinning effect from the defects involved in the synthetic, fabrication, and integration processes [3-6]. Considerable efforts, including phase engineering of the channel materials (from semiconducting 1H phase to conductive 1T phase) [7], geometry of contacts [8-11], and interface engineering with graphene buffer layer [12, 13], are carried out for essential electronic performances with improved contact properties.

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Recently, integration of conductive graphene and semiconducting TMD for improved contacts and novel properties is realized by direct growth of TMD using chemical vapor deposition at the edge of artificially patterned graphene [14–21]. Heterojunctions among different 2D materials enable essential multifunctionality of the monolayer channels for broader capacity and integration [22-27]. Weak tunneling barrier is achieved at the heterojunction of the laterally stitched MoS2-graphene, enabling inverter and negative-AND (NAND) gates for a complete set of logic circuits based on 2D materials [16, 17]. The next essential goal is to realize basic electronic units of complementary metal-oxide semiconductor (CMOS) inverters and other logic circuits with scalable 2D materials. Towards this goal, however, it remains a long-lasting challenge on selective and controllable fabrication of p-type or ambipolar transistors with a low Schottky barrier [28]. Most p-type transistors are demonstrated with tungsten selenides (WSe₂) but a high temperature is required for the WSe₂ growth because of a higher evaporation temperature of the WO_3 precursor [29-31]. A low temperature synthesis of the sequential monolayer growth at the pre-patterned 2D materials is mainly achieved with Mo-based TMD.



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Here, we utilize seeding promoter and low pressure CVD process to enhance sequential WSe_2 growth with a reduced growth temperature for reduced compositional fluctuations and high hetero-interface quality [32, 33]. Growth behavior of the sequential WSe_2 growth at the edge of patterned graphene is discussed. With optimized growth conditions, high-quality interface of the laterally stitched WSe_2 -graphene is achieved and studied with TEM. Device fabrication and electronic performances of the laterally stitched WSe_2 -graphene are presented.

Method/Experimental

Synthesis of WSe₂ and Graphene

Large-area WSe₂ films were synthesized on sapphire and SiO₂/Si substrates in the furnace. Before growth process, the substrates were cleaned with acetone, isopropanol, and then water for 10 min, respectively. Perylene-3,4,9, 10-tetracarboxylic acid tetrapotassium salt (PTAS) was uniformly coated on the substrate surface as seeding promoters to enhance the activity and growth rate of the monolayers. High purity solid precursors of WO₃ (Alfa Aesar, 99.9995% CAS#1313-27-5) and Se (Sigma-Aldrich, 99.5% CAS#7704-34-9) were placed in two ceramic

crucibles, and the substrates were placed face up and next to the WO₃ powder. The WSe₂ samples were synthesized during 800~900 °C for 10 min with a heating rate of 30 °C min⁻¹ and under a mixture of N₂/H₂ flow at 1.2 Torr. Graphene is synthesized on Cu foil at 1000 °C for 10 min with a heating rate of 30 °C min⁻¹ and under a mixture of CH₄/H₂ flow at 4 Torr. The pattern graphene is carried out by e-beam lithography and oxygen plasma etching.

Device Fabrication

The graphene-WSe₂ devices were fabricated without sample transfer. E-beam lithography process was performed to define the electrodes on the patterned graphene layer. A thin metal layer of Pd (40 nm) was deposited using e-beam evaporation and a following lift-off process was carried out in acetone. Encapsulation layer and gate dielectric of the device are fabricated by using atomic layer deposition (ALD) of thin Al₂O₃ films (50 nm). A thin metal of Pd (40 nm) was deposited on the dielectric layer to use as the gate electrodes. To improve electronic performances, the devices are annealed at ~ 120 °C for ~ 12 h in a vacuum environment of ~ 10⁻⁵ Torr.



Fig. 1 Controlled growth of the WSe₂ at patterned graphene. **a** Schematic of the laterally stitched WSe₂-graphene synthesis. **b** Raman mapping for the G' band of the graphene and **c** AFM image of the patterned growth of the WSe₂-graphene. **d** Raman spectra of the E_{2g} mode (WSe₂—blue) and the G' band (graphene—green) in **c**. Raman mapping of **e** the E_{2g} mode of the WSe₂ and **f** the G' band of the graphene in the monolayer heterojunction

Characterizations

Raman spectra and photoluminescence (PL) were obtained by commercial confocal Raman spectroscopy (Micro Raman/PL/TR-PL Spectrometer, Ramaker, Protrustech). Wavelength and spot size of the laser are 532 nm and $1-2 \mu m$, respectively. Typical gratings were used with 300 g/mm for PL (low resolution) to get broadband spectrum and (high resolution) 1800 g/mm for Raman signals to get detail information of material. The TEM samples were prepared by using standard PMMA transfer technique to place the graphene-WSe₂ nanosheets onto the holey-carbon Cu grid. The TEM images were performed at an accelerate voltage of 80 kV (Cs-corrected STEM, JEOL, JEM-ARM200F). The electrical measurements were measured using an Agilent B1500a Semiconductor Device Analyzer.

Results and Discussion

To control the synthesis of the lateral heterojunction of graphene and WSe₂, sequential growth of the monolayer TMD at the graphene edges is demonstrated in Fig. 1a. Monolayer graphene is first grown on a copper foil and later transferred onto a fresh sapphire substrate by using standard PMMA-assisted transfer method. Conventional e-beam lithography and O_2 plasma etching processes are

conducted to define the region for sequential growth of the monolayer WSe₂. Direct synthesis of monolayer WSe₂ at the edges of patterned graphene on sapphire substrate is achieved by low pressure CVD with PTAS as seeding promoters. More detailed information on the synthesis is described in the "Method/Experimental" section. In Fig. 1b, Raman mapping of the G' band in the laterally stitched graphene-WSe₂ displays a uniform contrast, which confirms a reduced damage of the prepatterned graphene after the sequential CVD synthesis of the WSe₂ growth. In Fig. 1c, AFM image of the patterned growth of the graphene-WSe2 indicates a smooth surface morphology of the monolayer heterojunction. Figure 1d presents the Raman spectra of the E_{2g} mode (WSe₂-blue) and the G' band (graphene-green) as the labels in Fig. 1c, which are consistent with the reported studies [34]. To illustrate uniformity of the as-grown heterojunction, Raman mapping of the patterned graphene- WSe_2 is shown in Fig. 1 e and f, respectively. A uniform contrast of the Raman intensity in the mapping images is clearly observed, suggesting controllable synthesis on heterogeneous growth of high-quality monolayer WSe₂ at the edges of the pre-patterned graphene.

To clarify growth behavior of the stitched graphene-TMD, the WSe_2 synthesis at the patterned graphene is



Fig. 2 Temperature-dependent WSe₂ growth with seeding promoter: Optical images, Raman mapping images of the A_{1g} mode (WSe₂) and the G' band (graphene) of the samples that are synthesized at different temperatures **a**, **b** without and **c**, **d** with PTAS as seeding promoter

carried out with and without promoters. Figure 2 a and b suggest the WSe₂ growth at different temperatures without PTAS as seeding promoter. Above 850 °C, the sequential growth of the WSe₂ appears at the graphene edges. A high growth temperature for WSe2 growth is required due to reduced gaseous reactants for the solid precursor of the WO₃, as elaborated in previous papers [29–31]. A macroscopically smooth boundary of the asgrown WSe₂ implies random distributed and small size of grains. In contrast, the sequential WSe₂ growth at different temperatures with PTAS as seeding promoter is presented in Fig. 2 c and d. The PTAS promoters significantly reduce growth temperature for perfect sequential WSe₂ growth at the graphene edges with larger domain sizes, which is similar to the growth behavior in the TMD-TMD heterojunctions [22]. After the sequential WSe₂ growth at 800 °C, observation of a uniform contrast and higher intensity in Raman mapping of the G' band (graphene) indicates a reduced damage of the graphene because of the low temperature growth. With increased temperature, a continuous WSe₂ film fills in the patterned regions with ideal contact to the edges of the patterned graphene (Fig. 2d). Note that a larger domain size with a clear triangular shape of the monolayer WSe₂ stitched to the edges of the graphene (Fig. 2c), suggesting a better quality of the sequential WSe₂ growth. With optimized growth conditions on seeding promoters and temperature, scalable and high-quality monolayer WSe₂ is realized by the LPCVD system as presented in the supporting information (Additional file 1: Figure S1). It is noteworthy that the sequential TMD synthesis at the edges of patterned graphene is universally observed in other heterojunctions of different TMD and graphene as shown in the supporting information (Additional file 1: Figure S2).

To further investigate the heterojunction of the WSe₂-graphene, high-resolution transmission electron microscopy (HRTEM) measurement is performed. In Fig. 3a, selected area TEM image indicates that the overlap region between black (graphene end) and green (TMD end) dashed lines is composed of the prepatterned graphene and the sequential grown WSe₂





monolayer. Width of the overlapping region is about 500 nm. An amorphous-like TEM image for the graphene lattice is observed as expected because of unavoidable distortions of graphene with the energetic electron beam. Figure 3 c and d present the calculated and experimental observation on the HRTEM image for better understanding on the sequential TMD growth at the heterojunction. Observation of hexagonal lattices and unit cell of graphene (~ 2.5 Å) and WSe₂ (~ 3.3 Å) is consistent with the parameters in bulk lattices of graphene (2.46 Å) and WSe₂ (3.28 Å). The TEM characterizations indicate that the sequential WSe₂ growth initiates at the edges of the pre-patterned graphene because higher defect density at the graphene edge enhances the vertical island growth with more nucleation sites. A large lattice mismatch more than 20% between the lattice of graphene and TMD might be responsible for a disorder interface with higher defect density and for combined vertical and lateral TMD growth at the heterojunction. Moreover, the insets in Fig. 3d show the corresponding diffractograms by fast Fourier transform (FFT) of real space atomic images in the overlap region and graphene region. Only one set of diffraction pattern is observed at the graphene region (left), while two sets of diffraction patterns rotated with a twisting angle of 0.35° are observed at the overlapped region (right). A highly reduced twisting angle between graphene and WSe₂ lattices implies that the sequential growth of the WSe_2 favors coherent stacking at the graphene edges.

To demonstrate the field-effect properties of the asgrown WSe₂ stitched at the edges of patterned graphene hetero-device, the device is fabricated without sample transfer. Customized fabrication process based on surface functionality for e-beam lithography on an insulator is developed. Electronic transport performance of the stitched graphene-WSe₂ device is studied by connecting metal electrodes (Pd 40 nm) with the patterned graphene and depositing Al₂O₃ (50 nm) as gate dielectric. Figure 4 a and b show the schematic illustration of the top-gated heterojunction device and the optical image of the as-fabricated device, respectively. Two-terminal electronic transport measurements are carried out using commercial probe-station (Lake Shore Cryotronics PS-100 with Agilent B1500a) under vacuum at room temperature. The transfer curve of the device exhibits a p-type transport behavior with an on/off ratio (~ 10^4) and high on-current of approximately a few 100 nA (Fig. 4c). The field-effect mobility of the device in the linear region is around $0.07 \text{ cm}^2/\text{Vs}$ at $V_{\rm d}$ = 2 V, which is evaluated using the following equation:

$$\mu = \frac{1}{C_{\rm ox}} \frac{L}{W} \frac{\partial I_{\rm D}}{\partial V_{\rm G}} \frac{1}{V_{\rm D}}$$
(1)



where $C_{\text{ox}} = \varepsilon_0 \varepsilon_r / d$ is the oxide capacitance and L (9 µm) and W (24 µm) are the channel length and channel width, respectively. Moreover, the output curves of the device at various gate voltages are shown in Fig. 4d. The linear *I-V* curves confirm a good contact between graphene layer and WSe₂ layer. An enhanced electronic performance of the stitched TMD-graphene monolayer heterojunctions is achieved because of improved contact properties, suggesting the synthesis for sequential TMD growth at the edges of artificially patterned graphene moves a significant step towards 2D nanoelectronics.

Conclusions

Sequential WSe₂ growth at the edges of the patterned graphene is achieved on sapphire by using promoterassisted LPCVD. The PTAS promoters significantly reduce growth temperature for ideal sequential WSe₂ growth at the graphene edges with larger domain sizes.

The TEM characterizations indicate that the sequential WSe₂ growth initiates at the edges of the pre-patterned graphene. A highly reduced twisting angle between graphene and WSe₂ lattices implies that the sequential WSe₂ growth favors coherent stacking at the graphene edges. An enhanced electronic performance of the stitched TMD-graphene monolayer heterojunctions is achieved because of improved contact properties.

Supplementary information

Supplementary information accompanies this paper at https://doi.org/10. 1186/s11671-020-3261-y.

Additional file 1: Figure S1. Scalable synthesis of monolayer WSe₂ on sapphire: influence on (a) concentration of the seeding promoters and (b) temperature for the WSe₂ growth. Figure S2. Synthesis of the laterally stitched graphene-WS₂: (a) optical and (b)AFM image of the graphene-WS₂ and (c) PL spectrum of the WS₂. Raman mapping of (d) the E_{2g} mode of WS₂ and (e) the G' mode of graphene. (f) PL mapping of the Graphene-WS₂. (Scale bar: 4 μ m).

Abbreviations

2D: Two-dimensional; AES: Auger electron spectroscopy; EIS: Electrochemical impedance spectroscopy; SEM: Scanning electron microscopy; TEM: Transmission electron microscopy; TMD: Transition metal dichalcogenides; XPS: X-ray photoelectron spectroscopy; XRD: X-ray diffraction

Authors' Contributions

YHL designed and supervised the project. YHL, XQZ, YTL, and YFC co-wrote the paper. XQZ and YTL contributed equally to this work. YTL and PHC performed device fabrication and measurements. XQZ, ECL, and JGR performed material synthesis, monolayer transfer, and material characterizations with the assistanceon TEM from C.C.C. and C.O. All authors discussed the results and commented on the manuscript at all stages. All authors read and approved the final manuscript.

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Availability of Data and Materials

All data generated or analyzed during this study are included in this published article and its supplementary information files.

Competing Interests

The authors declare that they have no competing interests.

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