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Probing Photoresponse of Aligned Single-Walled Carbon Nanotube Doped Ultrathin MoS₂

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Abstract

We report a facile method to produce ultrathin molybdenum disulfide (MoS₂) hybrids with polarized near-infrared (NIR) photoresponses, in which horizontally-aligned single-walled carbon nanotubes (SWNTs) are integrated with single- and few-layer MoS₂ through a two-step chemical vapor deposition (CVD) process. The photocurrent generation mechanisms in SWNT-MoS₂ hybrids are systematically investigated through wavelength- and polarization-dependence scanning photocurrent measurements. When the incident photon energy is above the direct bandgap of MoS₂, isotropic photocurrent signals are observed, which can be primarily attributed to the direct band gap transition in MoS₂. In contrast, if the incident photon energy in the NIR region is below the direct bandgap of MoS₂, the maximum photocurrent response occurs when the incident light is polarized in the direction along the SWNTs, indicating that photocurrent signals mainly result from anisotropic SWNTs absorption. More importantly, these two-dimensional (2D) hybrid structures inherit the electrical transport properties from MoS₂, displaying n-type characteristics at a zero gate voltage. These fundamental studies provide a new way to produce ultrathin MoS₂ hybrids with inherited electrical properties and polarized NIR photoresponses, opening doors for engineering various 2D hybrid materials for future broadband optoelectronic applications.

Keywords

MoS₂; SWNT; NIR; photoresponse; CVD

1. Introduction

Two-dimensional (2D) materials with a layered structure such as transition metal dichalcogenides (TMDCs) have attracted a large amount of interest owing to their unique properties^{1–4} and advancement in synthesis techniques.^{5–6} As a member of the TMDC family, molybdenum disulfide (MoS₂) shows promising transistor performance with a room-temperature current on/off ratio of 10⁸.⁷ The intrinsic direct bandgap for monolayer MoS₂ is 1.9 eV, whereas bulk MoS₂ is an indirect-bandgap semiconductor with a bandgap of 1.2 eV,

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Conflicts of interest

There are no conflicts to declare.

2, 7–8 which is suitable for optoelectronic applications in the visible spectral region.⁹ For example, metal-MoS₂-metal devices have exhibited a high photoresponsivity of 880 A/W at a wavelength of 561 nm, where strong photocurrent signals are generated at the metal-MoS₂ junctions.⁹ However, the sizeable bandgap (> 1 eV) of MoS₂ restricts its optoelectronic applications in the near-infrared (NIR) spectral region. The photoresponse of MoS₂ photodetectors would decrease drastically under the illumination of 785 nm (1.6 eV) and 1550 nm (0.8 eV) lasers, where the phonon energies are not enough to excite electrons from the valence band to the conduction band of MoS₂.¹⁰ Various hybrid structures have been developed to extend the optoelectronic applications of MoS₂ in the NIR fields.^{11–13} Atomic p-n heterojunctions between MoS₂ and black phosphorus (BP) have been fabricated as photodiodes, where BP, a novel semiconducting material with a thickness-dependent direct bandgap from 0.3 eV to 2 eV,^{14–16} has been utilized to enhance the photoresponses of photodiodes in the NIR spectral region.^{11–13} However, the mechanical exfoliation as well as complicated transfer processes involved in the fabrication of these devices limit their potential applications for mass production. It is, therefore, desirable to develop a method of producing 2D hybrid materials that inherit the electrical performance from TMDCs and exhibit enhanced photoresponse in the NIR spectral region.

Single-walled carbon nanotubes (SWNTs), one-dimensional (1D) carbon-based nanomaterials, have gained much attention due to their fascinating features.^{17–20} Recently, highly-efficient generation of electron-hole pairs (EHPs) have been reported in SWNT p-n junctions, where a single photon can induce multiple EHPs.²¹ Moreover, carbon nanotubes can absorb light with wavelengths ranging from 200 nm to 200 μ m due to a wide distribution of nanotube diameters that leads to various bandgaps for individual nanotubes.²² Many techniques have been developed to produce carbon nanotubes with specific alignment, density, and even chirality in a large scale.^{23–25} For instance, slow vacuum filtration has been used to obtain wafer-scale monodomain films of highly-packed aligned SWNTs with a density of 10⁶ nanotubes in a cross-sectional area of 1 μ m².²³ These thickness-controllable and chirality-enriched SWNT films can be made as semiconductor-enriched devices with polarized electronic and optoelectronic properties.²³ Another approach with novel catalysts called Trojan or Trojan-Mo has recently been developed to synthesize high-ratio (> 91%) and high-density (>100 nanotubes per μ m) semiconducting-SWNT arrays through chemical vapour deposition (CVD).²⁴ Thus, carbon nanotubes are promising add-on components for future broadband optoelectronics²⁶.

Here we report a two-step CVD method to synthesize ultrathin SWNT-MoS₂ hybrids with enhanced NIR photoresponses. This hybrid material can easily be visualized under an optical microscope, making the subsequent optical and electrical characterization much easier. Interestingly, enhanced Raman intensities of MoS₂ modes are observed in the areas where aligned SWNTs are located, indicating MoS₂ aggregation or graphitic substrate may influence the Raman spectra of MoS₂. We also find that the hybrid material possesses a similar electrical property as MoS₂ and inherits the optoelectronic properties from both MoS₂ and SWNTs. Under 650 nm illumination, the hybrid displays isotropic photoresponses, suggesting the photocurrent signals are mainly attributed to MoS₂ absorption. However, the photocurrent response is polarized along the SWNT direction when the wavelength of the incident light is 850 nm, a typical behaviour for SWNT

absorption. This indicates SWNT arrays can help enhance the photoresponse of MoS₂ in the NIR spectral region. Combining unique properties from two materials, these SWNT-MoS₂ hybrids may provide new avenues to the development of 2D hybrid materials toward future broadband optoelectronic applications.

2. Materials and methods

We adopted a standard method using copper as the catalyst and ethanol as the carbon source to synthesize high-density aligned SWNTs on stable temperature (ST)-cut quartz substrates (Hoffman Material Inc.).^{27–28} The CuCl₂/polyvinylpyrrolidone (PVP) alcohol solution, as catalyst precursors, was glued onto the pre-patterned tape mask on the quartz surface. Line-shaped catalyst precursors were left on the substrate after removing the tape mask (Fig. 1a). Here, the thickness of the catalyst particles is a few nanometers, which can affect the diameter of SWNTs.²⁸ The spacing between catalyst lines is a few millimeters to avoid the contact between metal electrodes and catalyst lines during the following electrical transport measurements. The single-crystal substrates were subsequently annealed in air inside a one-inch quartz tube at 750 °C for 30 min to remove the polymers and to oxidize the composites as Cu_xO_y. After the samples cooled down to room temperature, an ambient growth was conducted with a flow of 200 sccm H₂ at 750 °C for 30 min, and an additional flow of 50 sccm Ar through an alcohol bubbler at 900 °C for 10 min. The samples were then allowed to cool down to room temperature. Fig. 1b shows an atomic force microscopy (AFM, Bruker Inc) image of horizontally-aligned SWNTs on a quartz substrate and the diameters of these SWNTs range from 0.8 nm to 2.2 nm (Fig. 1c), which are typical for as-grown SWNTs.²⁸

Subsequently, MoS₂ was synthesized atop horizontally-aligned SWNTs to form hybrid structures. Substrates with pre-grown aligned SWNTs were loaded into a one-inch quartz tube and placed face-down above a quartz boat containing 10 mg of MoO₃ (99.95% Alfa Aesar #11837) with another boat containing 300 mg sulfur (99.5% Alfa Aesar #43766) located in the upstream area. The distance between sulfur and MoO₃ was 16–18 cm, which was optimized for monolayer MoS₂ growth on ST-cut quartz. The furnace was first pumped down to 0.8 Torr, refilled with ultrapure Ar until 550 Torr, and repeated once. Later, the furnace was heated up to 650 °C at 30 °C per min with a 10 sccm Ar flow. The sulfur was about to sublime at ~ 200 °C, while the temperature of the middle part of the furnace ramped up to 650 °C (Fig. 1d) for 5 min and then cooled down to 500 °C for 10 min. Finally, we opened the furnace lid and added a 500 sccm Ar flow for rapid cooling. SWNT-MoS₂ hybrids synthesized through a two-step chemical vapor deposition process can provide clean interfaces between SWNTs and MoS₂, since there is no polymer routinely used for transferring processes involved. More importantly, SWNTs, graphene-like molecules, are expected to promote the layer growth of MoS₂ to improve their electrical properties.²⁹ The electrical properties of the hybrids will be discussed in the following section.

3. Results and discussion

At each stage of the synthesis, the samples were visually characterized under an optical microscope (Olympus BX51WI). On nearly transparent quartz substrates, SWNTs cannot be observed after the first growth. However, they can be clearly imaged as white lines under an

optical microscope after the subsequent growth of MoS₂ (Fig. 2a). Similar results have been reported previously, where nanotubes can be observed under optical microscopes with the help of either TiO₂ nanoparticles³⁰ or water vapor.³¹ Scanning electrical microscopy (SEM) was also used to investigate these SWNT-MoS₂ hybrids for detailed information. As shown in Fig. 2b, aggregated MoS₂ flakes were observed along each SWNT, leading to the ‘line-shape’ contrast and allowing us to observe them under an optical microscope. Recently, researchers have obtained large-area MoS₂ films by utilizing different graphene-like molecules as seeding promoters.^{29, 32} Here SWNTs may act as ‘seeds’ for initial nucleation of MoS₂ films, likely facilitating MoS₂ growth along the nanotubes.

Moreover, the visually-recognizable SWNT-MoS₂ hybrids make the subsequent Raman characterization much easier. Raman spectra were collected at room temperature through a Thermo Scientific DXR Raman spectrometer from 100 to 1800 cm⁻¹. A 532 nm laser beam was focused into a diffraction-limited spot (~ 1 μm) by a 100X Olympus objective. Fig. 2c displays that the intensities of MoS₂ mode (E_{1/2g} and A_{1g}) are higher at the locations where G peaks of SWNTs are observed than those of other spots. Similar results are also presented in the Raman mapping of the hybrid, where strong E_{1/2g} and A_{1g} modes are presented in the ‘line-shape’ areas (Figs. 2e and 2d), suggesting that the Raman enhancement is likely due to the aggregated MoS₂ flakes along SWNTs. The SWNT substrate may also facilitate the charge transfer between MoS₂ and SWNTs and influence the Raman spectra of MoS₂. Previous Raman studies of MoS₂ films on graphene plate have demonstrated that the photoelectrons could easily transfer between two layered materials and then affect the Raman signals.^{33–34} Another possible reason for enhanced Raman signals is that SWNT substrates could help enhance the Raman signals of MoS₂ films by reducing the substrate interference.³⁵ Also, layered MoS₂ is a hexagonal arrangement of Mo and S atoms in a sandwich structure, which is similar to the honeycomb structure of graphene and SWNTs. The structural similarity between SWNTs and MoS₂ may add to the vibrational coupling at such interfaces and lead to the Raman enhancement.³⁶ Interestingly, the G band of SWNTs is observed at 1605 cm⁻¹ on quartz substrates, an “up-shift” band compared with 1590 cm⁻¹ on SiO₂/Si substrates. This “up-shift” behaviour may result from the strong interaction between SWNTs and quartz substrates, which can deform the C-C bonds in SWNTs and thus affect the atomic vibration during Raman spectroscopy.³⁷ After the hybrids were transferred from quartz to SiO₂/Si substrates, the structural deformation disappeared at interfaces and thus G band returned to 1590 cm⁻¹ (Fig. 2c), a typical frequency for SWNTs.

Next, we investigated the electrical transfer performance of SWNT-MoS₂ hybrids in two different directions. As shown in Fig. 3a, when the gate voltage swept from -20 V to 50 V, the conductance of the hybrid in the direction perpendicular to SWNT arrays was recorded under a source-drain bias of 100 mV. The device exhibits n-type electrical characteristics at a zero gate voltage, a typical electrical transport property of CVD-grown MoS (Fig. 3b).^{38–39} Similar electrical behaviour was also observed for the hybrid in the direction parallel to SWNTs (black curve, Fig. 3a), indicating that the electrical transport property of the hybrid is dominated by MoS₂ films. For comparison, we performed the electrical transport measurements for a horizontally-aligned SWNT array transistor. As shown in Fig. 3c, when the gate voltage swept from -3 V to 4 V, the conductance of horizontally-aligned SWNTs was collected at a source-drain bias of 50 mV. It displays p-type characteristics at zero gate

bias, which is consistent with other reports,²⁷ but different from our SWNT-MoS₂ hybrids. With the second growth of an ultrathin MoS₂ layer on top of SWNTs, the electrical transfer characteristics changes from p-type to n-type and becomes dominated by MoS₂ films, likely due to the relatively low density of 1D SWNTs (Fig. 1b) in comparison with 2D MoS₂ films. It is obvious that these SWNT-MoS₂ hybrids inherit the electrical transport properties from MoS₂. More importantly, the electrical conductivity of the hybrid along the direction perpendicular to the SWNTs is similar to that along the direction parallel to the SWNTs, which is three times larger than that of CVD-grown MoS₂. This indicates that the high conductivity of SWNTs has limited effect on the electrical transport behavior of the hybrid. Therefore, the conductivity improvement may mainly result from the better quality of MoS₂ films grown on SWNT substrates, as reported in previous literatures that graphene-like molecules can promote the growth of MoS₂.^{29, 32} Here, the interaction between SWNTs and MoS₂ is likely non-covalent stacking since covalent bonds may reduce the conductivity of MoS₂ due to the carrier scattering.

To study the optoelectronic properties of SWNT-MoS₂ hybrids, we performed spatially resolved scanning photocurrent measurements with an Olympus BXWI51 microscope (Fig. 4a). A diffraction-limited laser spot ($\sim 1 \mu\text{m}$) scanned over a SWNT-MoS₂ hybrid device by two-axis scanning mirrors with a nanometer spatial resolution and photocurrent signals were recorded as a function of position, resulting in a spatially-resolved photocurrent map of the device. The reflection of the incident laser beam was simultaneously collected by a Si photodetector to locate the position of photocurrent signals on the device (Fig. 4b). Under 850 nm illumination (1.46 eV), strong photocurrent signals were observed in the areas where aligned SWNTs were located (Figs. 4c and S1), which is likely owing to the absorption of SWNTs since the photon energy is not enough to excite the electron from the valance band to the conduction band of MoS₂. To further explore the relative contributions of different materials to the overall photocurrent response, we performed polarization-dependent photocurrent measurements. As shown in the Fig. 4e, the measured polarization-dependence of the photoresponse in the hybrid is similar to that of a typical SWNT,⁴⁰ where the maximum signal intensity observed along the SWNT direction ($\theta = 0^\circ$). The polarization effect arises from the 1D geometry of the SWNT, where a smaller part of the incident light can be coupled into the SWNT for the cross-polarized light. The optical selection rules are also important for the polarizability of a nanotube. This suggests that the photocurrent signals mainly result from nanotubes. As illustrated in Fig. 4g, when SWNTs contact MoS₂, both the conduction and valence bands of SWNTs are higher than those of MoS₂ due to the alignment of their work functions, leading to band offsets at the interfaces between SWNTs and MoS₂. Under 850 nm illumination, photons are absorbed in SWNTs to generate photo-excited EHPs. Owing to the band offset at the conduction bands between SWNTs and MoS₂, photo-excited electrons in SWNTs can be injected into MoS₂,⁴¹ which can locally dope MoS₂ and thus bend its band structure, resulting in a local built-in electric field along the lateral MoS₂ channel. Therefore, the photocurrent signals are likely induced by the photovoltaic effect. Here, we can effectively enhance the NIR photoresponse of MoS₂ films by doping them with SWNTs. More importantly, the NIR photorsponse of SWNT-MoS₂ hybrids can be further improved by increasing the density of SWNTs. On the other hand, the electrons in the valence bands of both MoS₂ and SWNTs can be excited to their conduction

bands upon illumination of 650 nm laser (Fig. 4h). The polarization-dependent photocurrent measurements have shown that photocurrent signals at the SWNT-MoS₂ interface are isotropic (Figs. 4d and 4f), suggesting that the photocurrent responses mainly result from MoS₂ absorption and the hybrids inherit the optical properties from MoS₂ in the visible spectral region.

4. Conclusion

In summary, we develop a simple method to synthesize ultrathin SWNT-MoS₂ hybrids, in which a thin layer of MoS₂ helps visualize horizontally-aligned SWNT arrays under optical microscopes. Moreover, with the introduction of MoS₂ during the second growth, the electrical transfer characteristics changes from p-type for SWNTs to n-type for SWNT-MoS₂ hybrids, indicating that the hybrid materials inherit electrical transport properties from MoS₂ films. SWNT-MoS₂ hybrids also obtain unique optoelectronic properties from SWNTs in the NIR spectral region. This fundamental study may offer a new way to develop novel hybridized materials, which could provide promising applications for future broadband optoelectronics.

Supplementary Material

Refer to Web version on PubMed Central for supplementary material.

Acknowledgments

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Fig. 1. (a) Schematic illustrations of the synthesis of horizontally-aligned SWNTs. (b) An AFM image of SWNTs on a quartz substrate and (c) a height profile of SWNTs along the red line in (b). (d) Illustration of the furnace setup for SWNT-MoS₂ hybrid synthesis.

Fig. 2.

(a) Optical and (b) SEM images of SWNT-MoS₂ hybrids on quartz substrates. (c) Raman spectra of SWNT-MoS₂ hybrids on different substrates. Raman mapping images of (d) E_{2g}¹ and (e) A_{1g} modes of SWNT-MoS₂ hybrids on quartz substrates, respectively.

Fig. 3.

Gate-dependent measurements of (a) a SWNT-MoS₂ hybrid transistor along the directions of perpendicular (black) and parallel to (red) SWNTs, respectively; (b) a MoS₂ transistor; and (c) a horizontally-aligned SWNT array transistor. Insets: optical images of (a) a SWNT-MoS₂ hybrid transistor and (b) a MoS₂ transistor; and (c) SEM image of a SWNT array transistor.

Fig. 4.

(a) Schematic diagram of a scanning photocurrent measurement setup. (b) Reflection and scanning photocurrent images of a SWNT-MoS₂ transistor illuminated by (c) 850 nm and (d) 650 nm laser, respectively. Note: Electrodes and SWNTs are marked by golden solid lines and green dotted-lines from the reflection image, respectively. Normalized photocurrent intensities at a SWNT-MoS₂ interface as a function of (e) 850 nm and (f) 650 nm incident light polarization angle. Gate voltage and source-drain bias were 0 V during the measurements. Schematic illustrations of different mechanisms under (g) 850 nm and (h) 650 nm laser, respectively.