A Facile Way to Fabricate High-Performance Solution-Processed n-MoS$_2$/p-MoS$_2$ Bilayer Photodetectors

Jian Ye$^1$, Xueliang Li$^2$, Jianjun Zhao$^1$, Xuelan Mei$^1$ and Qian Li$^1$

Abstract

Two-dimensional (2D) material has many advantages including high carrier mobilities and conductivity, high optical transparency, excellent mechanical flexibility, and chemical stability, which made 2D material an ideal material for various optoelectronic devices. Here, we developed a facile method of preparing MoS$_2$ nanosheets followed by a facile liquid exfoliation method via ethyl cellulose-assisted doping and utilizing a plasma-induced p-doping approach to generate $t$ effectively the partially oxidized MoS$_2$ (p-MoS$_2$) nanosheets from the pristine n-type nanosheets. Moreover, an n-p junction type MoS$_2$ photodetector device with the built-in potentials to separate the photogenerated charges is able to significantly improved visible light response. We have fabricated photodetector devices consisting of a vertically stacked indium tin oxide (ITO)/pristine n-type MoS$_2$ nanosheets/p-MoS$_2$/Ag structure, which exhibit reasonably good performance illumination, as well as high current values in the range of visible wavelength from 350 to 600 nm. We believe that this work provides important scientific insights for photoelectric response properties of emerging atomically layered 2D materials for photovoltaic and other optoelectronic applications.

Keywords: Liquid exfoliation method, Partially oxidized MoS$_2$, Photodetectors

Background

Over the last decade, two-dimensional (2D) nanomaterials have drawn great attention because of their unique structures, large natural abundance, and distinctive properties compared to their bulk forms, and a broad range of applications in catalysis, electronics, energy-storage devices, optoelectronics, and so on [1–11]. In particular, the semiconducting layered transition metal dichalcogenides (LTMDs, e.g., WSe$_2$, WS$_2$, and MoS$_2$) have gained significant interest on optoelectronics due to their direct bandgaps, possessing intriguing optical properties suitable for optoelectronic applications in light-emitting diodes and photovoltaics [12–14]. Usually, LTMDs have a unique 2D X–M–X structure in which the transition metal atom layer is sandwiched between two close-packed chalcogen atom layers [1, 2, 15–17].

As a prototypical compound of LTMDs, MoS$_2$ has been extensively studied. Bulk MoS$_2$ is a typical semiconductor with an indirect bandgap. Expectedly, monolayer MoS$_2$ transistors have been demonstrated with on/off ratios of $10^6$ and ultralow standby power dissipation [17–19]. However, to realize the highly efficient optoelectronic devices based on MoS$_2$, it is also important to develop a strategy to prepare ultrathin MoS$_2$ nanosheets and tune the bandgaps with facile process. Several methods, such as mechanical exfoliation (the so-called Scotch tape method), liquid exfoliation, colloidal synthesis, chemical vapor deposition, chemical exfoliation, and electrochemical exfoliation have been developed to prepare ultrathin MoS$_2$ nanosheets [2, 20–30]. Among these methods, liquid exfoliation not only produces novel materials with the same composition yet dramatically changed electrical properties but also provides a facile way to prepare thin-layer nanosheets, which offers novel opportunities in the optoelectronics applications [17, 31–34].

In this work, we report that a novel liquid exfoliation method via ethyl cellulose-assisted doping can prepare an
excellent thin MoS$_2$ nanosheets and very effective method to generate the partially oxidized MoS$_2$ (p-MoS$_2$) nanosheets from the pristine n-type nanosheets. Moreover, an n-p junction type MoS$_2$ photodetector device with the built-in potentials to separate the photogenerated charges can result in significantly improved visible light response. We have fabricated photodetector devices consisting of a vertically stacked indium tin oxide (ITO)/pristine n-type MoS$_2$ nanosheets/p-MoS$_2$/Ag structure, which exhibit reasonably good performance illumination, as well as high current values in the range of visible wavelength from 350 to 600 nm. This work provides important scientific insights for leveraging unique optoelectronic properties of 2D materials for photodetector applications.

**Methods**

**Material Synthesis**

Molybdenum disulfide (MoS$_2$) nanosheets were synthesized by liquid ultrasound exfoliation as reported in the literature [35, 36]. Typically, MoS$_2$ powder (0.25 g, Aladdin) was dispersed in ethyl cellulose (EC) isopropanol solution (1 % w/v dispersion, 100 ml) in a SEBC bottle. The dispersion was sonicated for 24 h at 60 W in water bath. The resulting dispersion was centrifuged (Desktop High-speed Refrigerated Centrifuge Model TGL-16) at 5000 rpm for 15 min, and then the supernatant liquid was directly collected. Deionized water was mixed with the supernatant liquid (3:4 weight ratio) and subsequently centrifuged at 7500 rpm for 10 min. Whereafter, the lower precipitation was collected and dried. Deionized water was mixed with the supernatant liquid (3:4 weight ratio) and subsequently centrifuged at 7500 rpm for 8 min, discarding the supernatant. To debride any residual salt, the resulting MoS$_2$ precipitation was washed with deionized water and collected by vacuum filtration (0.45 μm filter paper). Finally, the MoS$_2$ nanosheet product was dried as a fine black powder. The final MoS$_2$ nanosheets were defined as n-MoS$_2$. For the preparation of p-MoS$_2$ nanosheets, the n-MoS$_2$ powder was taken a UV-ozone plasma treatment for 40 min to completely change to p-MoS$_2$ nanosheets.

**Characterizations**

TEM images were taken by a FEI TECNAI G2 F20-TWIN TEM. Raman spectra were recorded on inVia Raman microscope. XPS and UPS measurements were conducted using an ESCALAB 250Xi (Thermo) system. X-ray diffraction (XRD) patterns of the MoS$_2$ was carried out on a Bruker D8 Focus X-ray diffractometer operating at 30 kV and 20 mA with a copper target (λ=1.54 Å) and at a scanning rate of 1°/min.

**Photodetector Device Fabrication**

All devices were fabricated on pre-treatment ITO glass substrates [37] (sheet resistance <10 Ωsq$^{-1}$, ShenZhen NanBo Display Technology Co., Ltd.); cleaned sequentially using sonication in acetone, detergent, deionized water, and isopropanol; and then dried under a nitrogen stream, followed by ultraviolet light irradiation. Then, the n-MoS$_2$ nanosheets (10 mg/ml, in isopropanol) spin coated with 2000 rpm and thermally annealed at 150 °C for 15 min receive a thickness of 80 nm. Thereafter, the p-MoS$_2$ nanosheets (15 mg/ml, in isopropanol) was spin coated on n-MoS$_2$ nanosheets layer, followed by thermal annealing at 150 °C for 10 min in atmospheric environment. Eventually, Argentum Ag (150 nm) was deposited over the p-MoS$_2$ nanosheets layer by thermal.
evaporation under a vacuum of $6 \times 10^{-6}$ Torr to accomplish the device fabrication. The effective area of one cell was $\sim 1 \text{ cm}^2$. The photocurrent-voltage curves and I-T curves were measured with a Keithley 2400 source meter and a 150-W Xe lamp light source. The dark current-voltage curves were measured by Keithley 2400 source meter under dark. All the measurements were performed under ambient atmosphere at room temperature. The incident photo-to-electron conversion efficiency spectrum (IPCE) were detected under monochromatic illumination (Oriel Cornerstone 260 1/4 m monochromator equipped with Oriel 70613NS QTH lamp), and the calibration of the incident light was performed with a monocrystalline silicon diode.

**Results and Discussion**

The equal concentration of pristine MoS$_2$ and MoS$_2$ nanosheets after the liquid ultrasound exfoliation solution

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![Fig. 2](image1.png)

**Fig. 2** The transmission electron microscopy (TEM) images of (a) pristine MoS$_2$ and (b) MoS$_2$ nanosheets films on glass substrate, and the inset is selected area electron diffraction (SAED) pattern of the MoS$_2$ nanosheets. The scanning electron microscopy (SEM) images of (c) pristine MoS$_2$ and (d) MoS$_2$ nanosheets films on glass substrate.

![Fig. 3](image2.png)

**Fig. 3** (a) XRD patterns of the MoS$_2$ films on glass substrate. (b) Raman spectrum of MoS$_2$ films on glass substrate.
(10 mg/ml) was treated with ultrasound in ethanol for 30 min, respectively. The detailed process is demonstrated in experimental section. The photographs of pristine MoS$_2$ and MoS$_2$ nanosheets isopropanol dispersion solutions after ultrasound treatment are shown in Fig. 1. After storing for 48 h, humorous aggregation can be observed in pristine MoS$_2$ solution (Fig. 1a) and evident MoS$_2$ particles adhere to the sidewall. In contrast, the MoS$_2$ nanosheets after the liquid ultrasound exfoliation solution show a highly uniform and homogeneous suspension solution (Fig. 1b), indicating the successful preparation of MoS$_2$ nanosheets with the good dispensability.

In order to verify the degree of dispersion of exfoliated MoS$_2$ nanosheets by ethyl cellulose ethanol solution via liquid ultrasound exfoliation, transmission electron microscopy (TEM) and scanning electron microscopy (SEM) were performed (Fig. 2). For comparison, the morphologies of the pristine MoS$_2$ nanosheets prepared by 150 °C thermal annealing for 10 min were also determined. All of samples were spin-coated on ITO and tested in the same testing conditions. Figure 2a shows a rough morphology of the pristine MoS$_2$ and clearly stacked MoS$_2$ can be seen. However, Fig. 2b displays an individual MoS$_2$ sheet with six spot pattern in the selected-area electron diffraction (SAED) of MoS$_2$, suggesting that MoS$_2$ is scattered as individual MoS$_2$ nanosheet [38, 39]. Also, the severe aggregation of the pristine MoS$_2$ can be observed in SEM images (Fig. 2c), intriguingly, after being treated by ethyl cellulose ethanol solution via liquid ultrasound exfoliation, MoS$_2$ nanosheets can fully cover and tightly attach on the ITO substrate with a quite smooth surface morphology (Fig. 2d).

To further verify morphology results, the XRD patterns of pristine and exfoliated MoS$_2$ nanosheets (Fig. 3a)
only the peaks of (103) and (002) plane remain after li-
quid exfoliation which confirms that the MoS₂ nano-
sheets were successfully striped [40, 41]. Moreover, the
disappearance of other peaks could prove that ultrathin
MoS₂ nanosheets are tightly deposited on the ITO glass
with preferred ductility. The Raman spectrum can once
again prove the exfoliation of MoS₂ nanosheets. The two
peaks (1 and 2 g) between 360 and 430 cm⁻¹ are the
main peak of MoS₂ [42–44]. After liquid exfoliation, the
obvious decrease of the intensity of the two peaks was
observed.

It is well known that the MoS₂ nanosheets are n-type
semiconductor materials and several researches have
been reported that MoS₂ could be changed as a p-type
semiconductor material with a relative high work func-
tion after UV-ozone plasma treatment. Thus, the prop-
ties of MoS₂ nanosheets with or without the UV-ozone
plasma treatment were also investigated. Figure 4a is
the X-ray photoelectron spectroscopy (XPS) profile of
n-MoS₂ nanosheets (without plasma treatment) and p-
MoS₂ nanosheets (with plasma treatment). The Mo 3D
spectra of pristine MoS₂ nanosheets demonstrate out-
standing Mo⁴⁺3d⁵/₂ and Mo⁴⁺3d³/₂ bands at 228.7 and
231.5 eV, in agreement with the other works for n-MoS₂
nanosheets. However, the two strong peaks have a notable
shift to 235.3 and 232.5 eV, respectively, which is simi-
lar with the spectra of MoO₃ [45, 46]. Therefore, it
proved that n-MoS₂ nanosheets can be successfully ox-
idized to p-type materials after UV-ozone plasma
treatment. Since the MoS₂ layer is very thin via the
spin-coating method, it is important to analyze the bi-
layer junction existing at the interface of n-MoS₂/p-
MoS₂. To gain insight into the electronic structures of
the n-MoS₂/p-MoS₂ bilayer junction, we have per-
formed the UPS analysis. The work function was cal-
culated through the difference between the cutoff of
the highest binding energy and the photon energy of
the exciting radiation. The valence band (VB) can be
calculated from the cutoff from the lowest binding en-
ergy. As shown in Fig. 4b, after UV-ozone plasma
treatment, the work function of the MoS₂ nanosheets
has increased from 4.3 to 5.2 eV. The energy

![Fig. 6](image)

**Fig. 6** Current-voltage curves of the device **a** under a 150-W Xe lamp light source illumination and **b** in dark

![Fig. 7](image)

**Fig. 7** **a** The output signal of photocurrent under alternating light on and light off, where the entire device was illuminated by a 150-W Xe lamp
irradiation. **b** Photoresponse of MoS₂-based photodetector at a 0-V DC bias voltage. **b** Photoresponse of MoS₂-based photodetector at 1-V DC bias
voltage. **c** The spectral photoresponse vs. wavelength, showing a broad photosresponse range from 350 to 650 nm, which is, the absorption
spectrum of the nanohybrid covers the whole energy range of visible light.
difference between the Fermi level and valence band maximum is decreased from 1.4 to 0.4 eV, demonstrating the n-type MoS$_2$ nanosheets change to p-type MoS$_2$ nanosheets [47].

On the basis of the above results, we have constructed an energy diagram showing the band bending behavior at the n-MoS$_2$/p-MoS$_2$ bilayer junction interface, as shown in Fig. 5a. The n-MoS$_2$/p-MoS$_2$ bilayer junction with a built-in potential promises an excellent photodetector performance with a ITO/n-MoS$_2$/p-MoS$_2$/Ag device structure (Fig. 5b) which will be discussed later. The photocurrent-voltage curves and the photocurrent-voltage were measured with the Keithley 2400 source meter. As shown in Fig. 6a, b, the device shows the photovoltaic response under a 150-W Xe lamp light source illumination. The result shows the device has a p-n junction inside. In order to understand the photodetector properties in more detail and detect potential application in photoelectronic fields, we have performed further experiments of photodetector at a 1-V DC bias as shown in Fig. 7a, b. As seen from Fig. 7a, b, the photocurrent increases at an applied dc bias voltage of 0 and 1 V. Moreover, the photoreponse is steady, prompt, and reproducible during repeated on/off cycles of visible light illumination. More importantly, the n-MoS$_2$/p-MoS$_2$ bilayer junction-based device shows a very broad photoelectric response range from 350 to 600 nm, as shown in Fig. 7c, and therefore, the n-MoS$_2$/p-MoS$_2$ bilayer junction can harvest nearly the whole energy range of visible light.

Conclusions
We have demonstrated a high-quality n-MoS$_2$/p-MoS$_2$ bilayer junction-based device to achieve the high-performance photoreponse which can harvest nearly the whole energy range of visible light. Excellent, thin exfoliated MoS$_2$ nanosheets are realized by a facile liquid exfoliation, changing the n-type MoS$_2$ nanosheets to p-type MoS$_2$ nanosheets via a simple plasma treatment. This work shows that thin MoS$_2$ nanosheets can be fully integrated into the photodetector manufacturing process, which holds promise for realizing 2D materials in a variety of optical electronic and optical devices.

Competing Interests
The authors declare that they have no competing interests.

Authors’ Contributions
JY carried out the experiments. JY, XL, JZ, and QL participated in the design of the study. JY and XL conceived of the study, participated in its design and coordination, and helped draft the manuscript. All authors read and approved the final manuscript.

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Author details
1 Department of Chemistry and Environmental Engineering, Bengbu College, Bengbu, Anhui 233030, China. 2 School of Chemistry and Chemical Engineering, Hefei University of Technology, Hefei, Anhui 230009, China.

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