

Fabrication of Highly Photosensitive MoS₂ Photodetector Films Using Rapid Electrohydrodynamic-Jet Printing Process

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Molybdenum disulfide (MoS₂) is a representative transition metal dichalcogenide (TMD) that has been extensively studied as a potential semiconducting material for next generation optoelectronic applications due to its exceptional optical, electrical, and mechanical properties. However, the lack of an efficient and scalable technique for MoS₂ film synthesis is a major drawback. Therefore, in this study, electrohydrodynamic (EHD) jet printed MoS₂ (EJP MoS₂) photodetectors are reported. The optoelectronic characteristics of the EJP MoS₂ photodetector are investigated using two modes: two-terminal and three-terminal configuration under light illumination. For the two-terminal mode of the EJP MoS₂ photodetector, a photosensitivity of 3444% and a photoresponsivity of 1.68 A W⁻¹ are achieved, respectively. Concurrently, the three-terminal mode of the EJP MoS₂ photodetector exhibits an improved photosensitivity (4495%) and photoresponsivity (3.78 A W⁻¹) under gate voltage. The proposed EHD jet printing method and the EJP MoS₂ based photodetector will provide a feasible solution for high performance TMDs based photonics.

sensitive photodetectors due to its thickness-dependent bandgap (1.3–1.9 eV), high absorption coefficient, and superior carrier mobility (>100 cm² V⁻¹ s⁻¹)^[9–12] With these attractive properties, MoS₂ films were obtained by mechanical or chemical exfoliation as well as chemical vapor deposition (CVD) and physical deposition such as radio frequency sputtering and pulsed laser deposition (PLD) for photodetectors.^[13–18] However, the development of efficient and scalable synthesis method for MoS₂ film is still insufficient for practical applications. The inkjet printing technique is a promising method for the fabrication of MoS₂ films due to its scalability and speed. Additionally, it is an economical process, and it requires simple equipment.^[19–21]

In this study, an electrohydrodynamic (EHD) jet printed MoS₂ (EJP MoS₂) photodetector was proposed.

The process of deposition involved EHD-jet printing of a (NH₄)₂MoS₄-based solution in the Taylor cone-jet mode, which is economical, rapid, scalable, and results in minimal wastage of printing solution. Atomic force microscopy (AFM), scanning electron microscopy (SEM), Raman spectroscopy, photoluminescence (PL), X-ray photoelectron spectroscopy (XPS) measurements were performed to characterize the EJP MoS₂ film. The photoresponsive characteristics of the EJP MoS₂ photodetector were investigated using two operating modes: two-terminal EJP MoS₂ photodetector and three-terminal EJP MoS₂ photodetector under light illumination. High photosensitivity (≈3444%) and photoresponsivity (≈1.68 A W⁻¹) were achieved under light illumination (excitation wavelength: 405 nm, light intensity: 1.0 mW cm⁻²) at two-terminal mode. Concurrently, the photosensitivity and photoresponsivity of the EJP MoS₂ photodetector were significantly improved up to ≈4495% and ≈3.78 A W⁻¹, respectively, by applying gate voltage. The proposed EHD jet printing technique and the EJP MoS₂ based photodetector provide a feasible solution for high-performance TMD-based photonics.

1. Introduction

2D materials have been shown great potential in electronic and optoelectronic of their applications.^[1–3] Transition metal dichalcogenides (TMDs), one of the attractive 2D materials, have recently emerged as next-generation semiconducting materials due to their excellent electrical, optical, and mechanical properties.^[4–8] Particularly, molybdenum disulfide (MoS₂) exhibits attractive optoelectrical characteristics for highly

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2. Result and Discussion

2.1. EHD-Jet Printing Process and EJP MoS₂

Figure 1a shows a photograph of the EHD jet printer used for the synthesis of the MoS₂ film. Initially, an ammonium tetrathiomolybdate ((NH₄)₂MoS₄) precursor solution was dissolved in

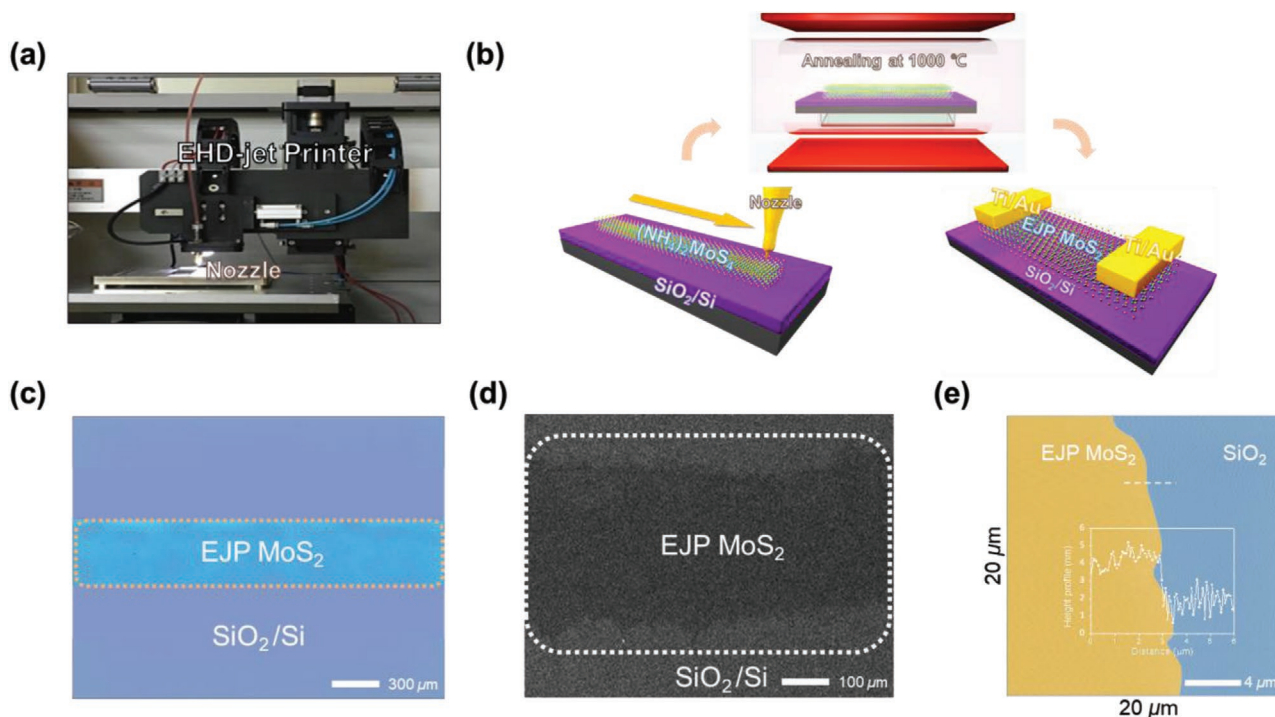


Figure 1. Schematic diagrams and surface characterizations of EJP MoS₂. a) The EHD-jet printer setup used for printing the MoS₂ film with Taylor cone-jetting mode. b) The fabrication process of EJP MoS₂ photodetector via EHD-jet printing. c) Optical microscopy and d) SEM image of the EJP MoS₂ film. e) AFM image of EJP MoS₂ on SiO₂/Si with a height profile of between MoS₂ and SiO₂ surfaces measured along with the white dashed line.

a mixture of ethanolamine, *n*-butylamine, and *n,n*-dimethylformamide (solvent wt.% = 2:2:1). To increase the element ratio of sulfur, carbon disulfide (CS₂) was added into the (NH₄)₂MoS₄ mixture. Subsequently, the (NH₄)₂MoS₄ mixture was ejected from a nozzle of the EHD-jet printer on a SiO₂/Si substrate. The ejection of the (NH₄)₂MoS₄ mixture was controlled by the electrostatic field (1.80–1.85 kV) between the nozzle and the SiO₂/Si substrate. The tip height, jetting speed, stage speed, and stage temperature were fixed at 2 mm, 0.0032 μL s⁻¹, 2000–8000 μm s⁻¹, and 50–60 °C, respectively. To convert (NH₄)₂MoS₄ into MoS₂, pre-annealing and annealing were performed after the EHD-jet printing process at 150 °C for 10 min and 1000 °C for 1 h, respectively. More detail EHD-jet printing process is described in our previous work.^[21] Ti/Au (20/100 nm) was deposited as the source and drain (S/D) electrodes and patterned on EJP MoS₂ using electron-beam evaporation and conventional lift-off photolithography techniques. Figure 1b shows the 3D schematic illustration of the aforementioned fabrication process of the EJP MoS₂ photodetector. An optical microscope image of the EJP MoS₂ film is shown in Figure 1c. The horizontal pattern of the EJP MoS₂ film was uniformly deposited on a SiO₂/Si substrate. The EJP MoS₂ film with a vivid light blue color contrasted with the SiO₂/Si substrate. To enable a practical evaluation of the EJP MoS₂ film quality, the surface of the EJP MoS₂ film was analyzed by scanning electron microscopy (SEM) as shown in Figure 1d. Figure 1e exhibits the thickness of the EJP MoS₂ film, which was measured using contact-mode atomic force microscopy (AFM). The height profile revealed that the average thickness of the EJP MoS₂ was ≈ 3 nm as shown in the inset of Figure 1e. Since the theoretical

thickness of the MoS₂ monolayer is ≈ 0.65 nm thickness,^[22] it can be inferred that the EJP MoS₂ film fabricated in this study was approximately four to five layers thick.

2.2. Surface Analysis of the EJP MoS₂

Raman spectroscopy, photoluminescence (PL) spectroscopy, and X-ray photoelectron spectroscopy (XPS) were performed to analyze the EJP MoS₂ film. Figure 2a shows the Raman spectrum of the EJP MoS₂ film presenting two distinct Raman modes: E_{2g}¹ (381.10 cm⁻¹) and A_{1g} (404.51 cm⁻¹) using an excitation laser (wavelength: 532 nm). The frequency difference (Δk = 23.4 cm⁻¹) between the two peaks (E_{2g}¹ and A_{1g}) indicated that the EJP MoS₂ film consisted of a few-layer structure.^[23] Then, multi-point Raman and PL spectroscopy of the EJP MoS₂ were performed as described in Figure S1 (Supporting Information). The multi-point Raman exhibited 22.2–23.4 cm⁻¹ gap between the two peaks that the whole area of the EJP MoS₂ seems to be few-layer, as shown in Figure S2 (Supporting Information).^[23] Figure 2b shows the PL spectrum of the EJP MoS₂ film and two main peaks were observed at 1.82 eV (A₁) and 1.95 eV (B₁) using an excitation laser (wavelength: 532 nm), indicating that the EJP MoS₂ was few-layer.^[24] Moreover, multi-point PL spectroscopy of the EJP MoS₂ were observed at 1.82–1.83 eV (A₁) and 1.97 eV (B₁) on the whole area of the EJP MoS₂ that well-matched with Raman spectroscopy of the EJP MoS₂, as described in Figure S3 (Supporting Information).^[24] To investigate the elemental composition and chemical bonding states of the EJP MoS₂ film, the bonding states of the Mo and S atoms were measured using

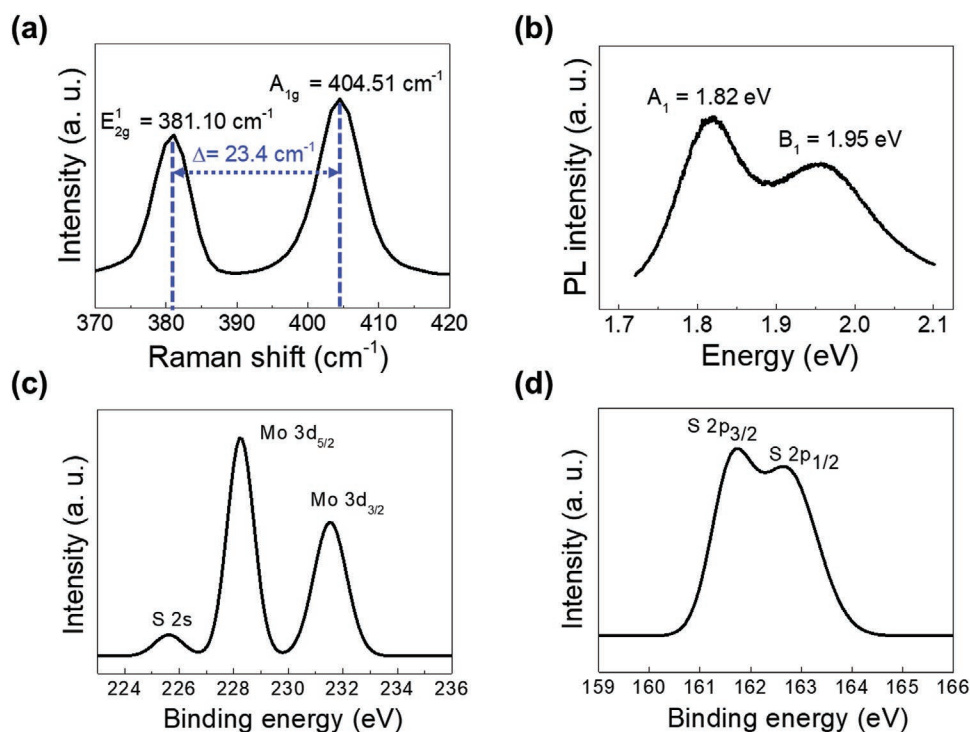


Figure 2. Surface analysis of the EJP MoS₂. a) Raman spectroscopy and b) PL spectrum of EJP MoS₂. XPS spectra of c) Mo 3d and d) S 2p core levels in EJP MoS₂.

X-ray photoelectron spectroscopy (XPS). Figure 2c shows the Mo 3d core levels of the EJP MoS₂ film. The two strong peaks visible at 228.96 and 232.15 eV were ascribed to Mo 3d_{5/2} and Mo 3d_{3/2}, with a third peak representing S 2s at 226.40 eV.^[25] Meanwhile, the S 2p peaks were observed at 163.05 and 162.00 eV, corresponding to S 2p_{3/2} and S 2p_{1/2}, respectively, as shown in Figure 2d.^[25] The results of XPS analysis revealed that the MoS₂ film was successfully deposited using the EHD-jet printing method.

2.3. Photoresponsive Characteristics of Two-Terminal EJP MoS₂ Photodetector

To compare the optoelectrical performance of the EJP photodetector with and without gate voltage, the EJP MoS₂ photodetector was investigated with two-terminal configuration (i.e., source and drain without gate). Figure 3a schematically depicts the two-terminal EJP MoS₂ photodetector under light illumination (excitation wavelength: 405 nm). The channel length and width of the EJP MoS₂ photodetector was 10 and 195 μm , respectively. The optoelectrical properties of the EJP MoS₂ photodetector with two-terminal configuration were measured under light illumination with five different light intensity variations from 0.1 to 1.0 mW cm^{-2} as plotted in Figure 3b (linear scale) and 3c (logarithmic scale), respectively. The drain current (I_{ds}) gradually increased with an increase in the light intensity and drain voltage (V_{ds}). A higher light intensity generated a greater number of photogenerated electron-hole pairs in the channel. This light intensity-dependent photocurrent of the EJP MoS₂ photodetector was attributed to the photoconductive

(PC) effect.^[26] Figure 3d shows the photosensitivity (S_{ph}) and the photoresponsivity (R_{ph}) calculated to obtain optical properties of the two-terminal EJP MoS₂ photodetector. The S_{ph} of the two-terminal EJP MoS₂ photodetector had a maximum value of 3588% at a light intensity of 1.0 mW cm^{-2} . S_{ph} is defined by $S_{\text{ph}} = \frac{I_{\text{total}} - I_{\text{dark}}}{I_{\text{dark}}}$, where I_{total} and I_{dark} are the total current and dark current, respectively. As shown in Figure 3d, the R_{ph} of the two-terminal EJP MoS₂ photodetector (maximum value: 1723 A W^{-1} at $V_{\text{ds}} = 10 \text{ V}$ under light illumination with a light intensity of 0.1 mW cm^{-2}) were extracted from $I_{\text{ds}}-V_{\text{ds}}$ characterization of the two-terminal EJP MoS₂ photodetector (Figure 3b). The R_{ph} was calculated using the following equation: $R_{\text{ph}} = \frac{I_{\text{photo}}}{P_{\text{opt}} \cdot A}$ (A W^{-1}), where I_{photo} , P_{opt} , and A is the photocurrent, light intensity, and effective area, respectively. It was observed that the two-terminal EJP MoS₂ photodetector exhibited decent optoelectronic performance.

2.4. Photoresponsive Characteristics of EJP MoS₂ Photodetector

To achieve an improved optoelectronic performance of the EJP MoS₂ photodetector, the EJP MoS₂ photodetector with three-terminal configuration (i.e., source, drain, and gate) was evaluated under the gate voltage (V_{gs}) and light illumination at $V_{\text{ds}} = 1 \text{ V}$. Without light illumination, the EJP MoS₂ photodetector shows a typical n-type electrical behavior, the threshold voltage (-18.39 V) and subthreshold swing (33.79 V dec^{-1}) was extracted. And the field-effect mobility (μ_{eff}) was calculated as 0.001 $\text{cm}^2 \text{ V}^{-1} \text{ s}^{-1}$ using the following equation: $\mu_{\text{eff}} = g_{\text{m}}(W_{\text{ch}}/L_{\text{ch}})(C_i V_{\text{ds}})$, where g_{m} is the transconductance, W_{ch} and

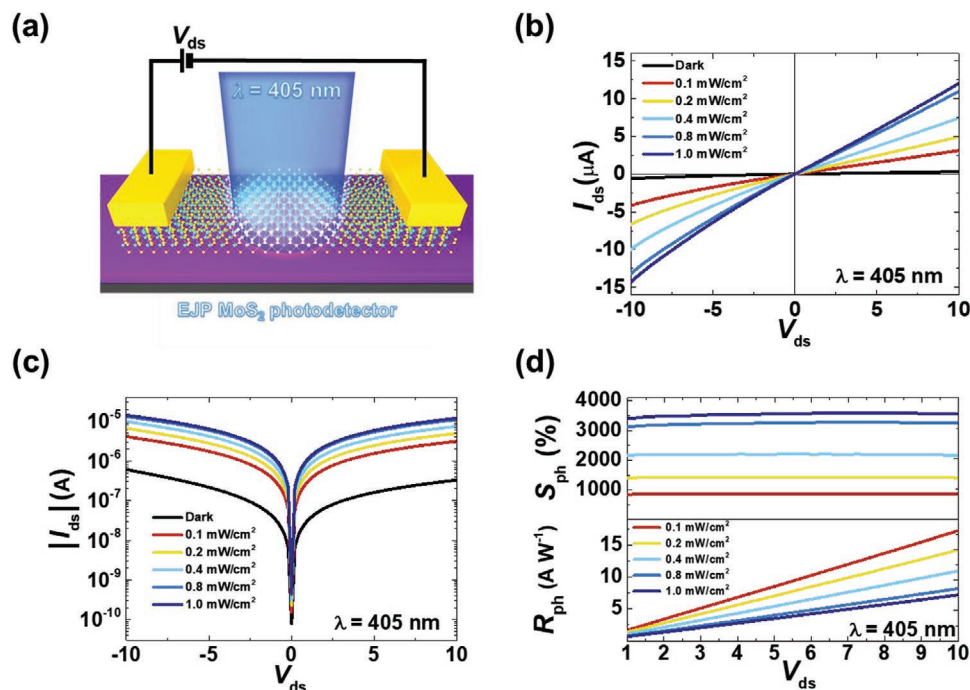


Figure 3. Photoresponsive characteristics of the two-terminal EJP MoS₂ photodetector. a) Illustration of the EJP MoS₂ photodetector under light illumination. I_{ds} - V_{ds} characteristics of the EJP MoS₂ photodetector under light illumination with b) linear and c) logarithmic scale. d) S_{ph} and R_{ph} of the two-terminal EJP MoS₂ photodetector.

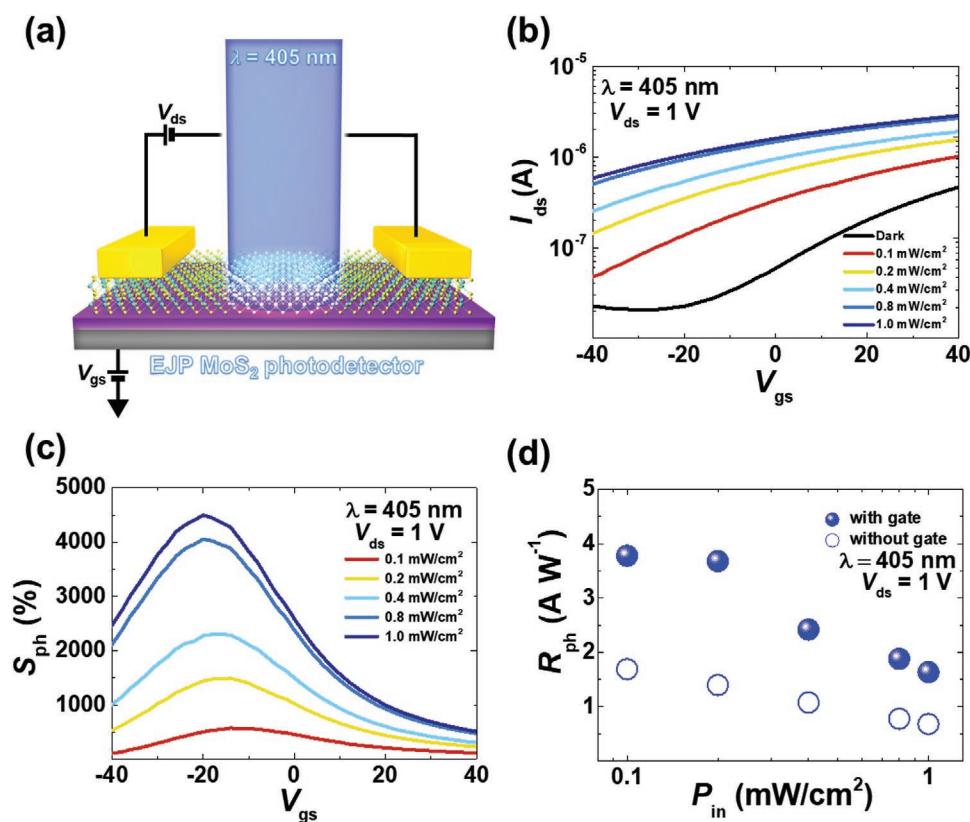


Figure 4. Photoresponsive characteristics of the three-terminal EJP MoS₂ photodetector. a) Illustration of the three-terminal EJP MoS₂ photodetector under light illumination. b) Transfer curves of the three-terminal EJP MoS₂ photodetector. c) S_{ph} of the three-terminal EJP MoS₂ photodetector. d) Comparison with R_{ph} of two-terminal and three-terminal EJP MoS₂ photodetector.

L_{ch} are the width and length of the channel, C_i is the capacitance of the gate insulator per unit area, and V_{ds} is the drain voltage. Then, optoelectrical characteristic of the EJP MoS₂ photodetector with three-terminal configuration was performed under light illumination as shown in Figure 4a. Figure 4b shows the transfer curves of the three-terminal EJP MoS₂ photodetector under light illumination with light intensity from 0.1 to 1.0 mW cm⁻². The photocurrent of the EJP MoS₂ photodetector significantly increased under the entire gate region. To evaluate the performance of the three-terminal EJP MoS₂ photodetector, S_{ph} and R_{ph} was extracted from the transfer curves of the device in Figure 4b. Figure 4c shows the S_{ph} calculated for the three-terminal EJP MoS₂ photodetector as a function of gate voltage. The maximum S_{ph} of the three-terminal EJP MoS₂ photodetector was 4495% at $V_{ds} = 1$ V, light intensity of 1.0 mW cm⁻², and $V_{gs} = -20$ V, which was enhanced to 30.25% in comparison with that of the two-terminal EJP MoS₂ ($S_{ph_two-terminal} = 3444\%$ under $V_{ds} = 1$ V, and light intensity of 1.0 mW cm⁻²). A comparison between the extracted R_{ph} of the three-terminal EJP MoS₂ photodetector and the two-terminal EJP MoS₂ photodetector indicated a significant improvement by applying gate voltage in EJP MoS₂ photodetector as shown in Figure 4d. The maximum R_{ph} of the EJP MoS₂ photodetector was 3.78 A W⁻¹ under a light intensity of 0.1 mW cm⁻² at $V_{ds} = 1$ V, and $V_{gs} = +40$ V, which was 123.7% greater than that of the two-terminal EJP MoS₂ photodetector ($R_{ph_two-terminal} = 1.68$ A W⁻¹ under a light intensity of 0.1 mW cm⁻² at $V_{ds} = 1$ V). Therefore, the EJP MoS₂ photodetector can achieve a higher S_{ph} and improved R_{ph} by applying gate voltage. This improvement is probably due to photogating effect which was reported by previous reports.^[27,28] Photoswitching characteristics of our device are shown in Figures S4 and S5 (Supporting Information). The rising time (τ_{rise} , 0.41 s) and falling time (τ_{fall} , 0.36 s) were defined as the times taken for the current increase from 20% to 80% and decrease from 80% to 20% of the maximum current, respectively.^[29] For comparison of our device and previous devices, optical properties of the EJP MoS₂ photodetector and MoS₂-based photodetectors are presented in Table S1 (Supporting Information). The proposed (EHD) jet printing method and the three-terminal EJP MoS₂ photodetector will provide a feasible solution for high performance TMDs based photonics.

3. Conclusion

In summary, an EJP MoS₂ photodetector was fabricated using EHD-jet printing method. SEM, AFM, Raman, PL, and XPS analyses confirmed that the EJP MoS₂ film was successfully deposited on the silicon substrate. The optoelectronic properties of the EJP MoS₂ photodetector were investigated using two modes (two-terminal and three-terminal) under light illumination. In three-terminal mode, the photosensitivity and photoresponsivity of the EJP MoS₂ photodetector were significantly improved in comparison with those of two-terminal EJP MoS₂ photodetector. Therefore, the proposed EHD-jet printing method and the EJP MoS₂ photodetector can provide a simple and feasible process for high-performance MoS₂ based optoelectronic devices.

4. Experimental Section

Preparation of Precursor Solution: An ammonium tetrathiomolybdate ((NH₄)₂MoS₄) precursor solution with a concentration of 0.025 M was prepared for printing MoS₂ patterns. For this purpose, (NH₄)₂MoS₄ (99.97%) was dissolved in a mixture of ethanolamine and *n*-butylamine, followed by the addition of *n,n*-dimethylformamide (solvent wt.% = 2:2:1). A sulfur solution containing carbon disulfide (CS₂) was added dropwise into the above mixture to obtain an S-rich solution, instead of placing sulfur powder in a tube furnace, which was conventionally performed during the chemical vapor deposition method. The method used in this study is based on previous works.^[21,30,31]

Fabrication of EJP MoS₂ Lines: An EHD-jet printer (Tera Leader Inc.) was used to create MoS₂ linear pattern lines using the Taylor cone-jet mode. The solution was injected into a syringe pump and then ejected through the 100-nm inner-diameter of a tip capillary onto the target substrate while the flow rate and applied voltage were adjusted. The solution could be positively charged, while the substrate was negatively charged by controlling the voltage. The resulting electrostatic field in the space between these two opposite poles, in combination with the force of gravity, pulled the solution downward toward the substrate. This is the operating principle of the EHD-jet printing technique. While the substrate was clamped on a movable stage in a horizontal plane to change the pattern dimensions in the X, Y directions, the tip could also be moved vertically (Z direction) to vary the stand-off height for several purposes. Additional printing parameters such as stage or substrate temperature and stage speed were also crucial to obtain patterns with clean surfaces and expected line sizes. In particular, the tip height, jetting speed, and stage temperature were fixed at 2 mm, 0.0032 μ L s⁻¹, and 50–60 °C, respectively. The stage speed was varied in the range of 2000–8000 μ m s⁻¹, and the voltage between the substrate and nozzle tip was 1.80–1.85 kV during line patterning. To observe the printing process, a digital microscope and high-speed video camera were used. Once the (NH₄)₂MoS₄ lines were EHD-jet printed, they underwent annealing steps for conversion to MoS₂ patterns, which were performed by pre-annealing at 150 °C for 10 min in ambient air and annealing at 1000 °C for 1 h in a tube furnace. Subsequently, the furnace was cooled to 25 °C at a rate of 15 °C min⁻¹.

Fabrication of EJP MoS₂ Photodetectors: From the bottom up, the fabricated bottom-gate top-contact TFTs contained a p-doped Si gate, 300-nm-thick SiO₂ dielectric, EJP MoS₂ semiconductor, and source and drain electrodes (Ti/Au). Specifically, UV/O₃-treated SiO₂/Si substrates and the 0.025 M (NH₄)₂MoS₄ precursor solution were prepared before EHD-jet printing. After printing, the linear patterns were dried and annealed for conversion to MoS₂. The source (20-nm-thick Ti) and drain (100-nm-thick Au) electrodes were deposited on the MoS₂ film using electron beam deposition and conventional lithography techniques.

Materials and TFT Characterization: The surface morphologies of the patterns were observed using an optical microscope (Olympus-BX51M) and an atomic force microscope (AFM Nano Xpert II). The Raman and PL spectra of the synthesized MoS₂ patterns were obtained using a 532-nm laser and 1800 grooves mm⁻¹ grating (Alpha 300 S, WITec). XPS (Nexsa, Thermo Scientific) was used to investigate the chemical elements in the EJP MoS₂, with a pass energy of 50 eV. The photodetector measurements were conducted using a semiconductor parameter analyzer (Keithley 4200-SCS) and multi-channel fiber coupled laser source (MCLSI, Thorlabs) in a dark box at room temperature.

Supporting Information

Supporting Information is available from the Wiley Online Library or from the author.

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Conflict of Interest

The authors declare no conflict of interest.

Data Availability Statement

Research data are not shared.

Keywords

electrohydrodynamic inkjet, molybdenum disulfide, photodetector, transition metal dichalcogenides

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