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## Facile fabrication of gas sensors based on molybdenum disulfide nanosheets and carbon nanotubes by self-assembly†

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The rising importance of gas detection has prompted rigorous research on flexible and transparent high-performance gas sensors. We demonstrated a sensor for NO<sub>2</sub> detection at room temperature, in which our device was fabricated *via* screen printing on a flexible substrate, and MoS<sub>2</sub> and single-walled carbon nanotube (SWCNT) were coated on a specific area by the self-assembly method. This fabrication process is rapid, facile, and cost-effective. The proposed sensor enables precise and stable NO<sub>2</sub> gas sensing from 50 ppb to 100 ppm. This method should also be applicable to the selective detection of other gases.

## 1 Introduction

Two-dimensional transition metal dichalcogenides (2D TMDs) have attracted immense research interest in recent years owing to their physical flexibility, unique 2D geometry, and high surface-to-volume ratio.<sup>1–4</sup> In particular, 2D TMDs have shown great promise for use as chemical sensors because of their enhanced sensitivity and room-temperature gas-sensing ability in relation to conventional metal oxides, which generally require high operating temperatures.<sup>5–8</sup> Among the 2D TMDs, MoS<sub>2</sub> has been widely used in thin electronics because MoS<sub>2</sub> nanosheets have unique physical and chemical properties such as optical transparency, flexibility, and tunable band gaps depending on their structure. MoS<sub>2</sub> is a typical 2D TMD used for gas detection owing to its high surface-to-volume ratio, tunable band gap, and high adsorption coefficient.

For application in electrical devices, MoS<sub>2</sub> nanosheets have been synthesized *via* the exfoliation of bulky MoS<sub>2</sub> or chemical vapor deposition through the sulfurization of molybdenum precursors.<sup>9–12</sup> Recently, composites of polymers and 2D semiconducting nanostructures, such as those of MoS<sub>2</sub>, have been synthesized to overcome the sensitivity limitations of 2D TMD-based sensors.<sup>13–16</sup> Because 2D carbon-based materials are highly sensitive to gases owing to their intrinsic electrical properties, hybrid layers based on 2D TMDs and graphene with

high electron transfer rates and surface-to-volume ratios were utilized to enhance electrochemical and sensing behaviors.<sup>17–19</sup>

One-dimensional carbon nanostructures such as carbon nanotubes (CNTs), graphene oxide (GO), and reduced graphene oxide (rGO) were explored for application as gas sensors. Carbon-based materials have large surface areas for adsorbing gas molecules and high strength and stability.<sup>20–22</sup> In addition, carbon-based materials such as single-walled carbon nanotubes (SWCNTs) were extensively studied as semiconductors for fabricating flexible and sensitive gas sensors, in which SWCNT-based networks are functionalized with polymers, oxide nanoparticles, and other compounds.<sup>23–25</sup>

NO<sub>2</sub>, which is emitted by fossil fuel consumption and diesel vehicles, is harmful to both humans and the environment, necessitating its efficient and cost-effective detection.<sup>26–29</sup>

In this study, NO<sub>2</sub> gas sensors were fabricated *via* the selective assembly of nanostructures on the thermoplastic elastomers styrene-ethylene-butylene-styrene (SEBS) and polyurethane (PU), which are stretchable and intrinsically flexible.<sup>30–32</sup> PU has a more hydrophilic surface than SEBS, which causes the MoS<sub>2</sub> nanosheets and SWCNTs to selectively assemble on its surface. Therefore, PU patterns with a hydrophilic surface were utilized to assemble millions of MoS<sub>2</sub>/SWCNT nanosheets over large surface areas, while hydrophobic SEBS patterns were used to prevent any unwanted adsorption on nanostructures. Additionally, the MoS<sub>2</sub>/SWCNT nanosheets were placed uniformly over the PU surface. A metallic silver electrode was layered on the substrate using screen printing as well. Owing to the wide band gap and flexibility of the MoS<sub>2</sub> nanosheets, the flexibility and gas detection performance of the gas sensors were improved through hybridization of the nanosheets with SWCNTs. The MoS<sub>2</sub>/SWCNT-based sensors exhibited NO<sub>2</sub> gas sensitivities of up to 50 ppb at room temperature. As we only used screen printing and selective self-assembly for

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the fabrication of flexible gas sensors, this approach may lead to the facile industrial-level production of 2D TMD-based devices for practical applications.

## 2 Experimental

### 2.1 Synthesis and preparation of $\text{MoS}_2$ nanosheets and SWCNTs

$\text{MoS}_2$  was synthesized using the hydrothermal method. For the synthesis of  $\text{MoS}_2$  nanosheets, 1 g of sodium molybdate dihydrate (4.13 mmol) and 1 g of thioacetamide (13 mmol) were dissolved in deionized water (30 mL) and stirred for 30 min. The mixed solution was transferred to a Teflon-lined stainless-steel autoclave, where it underwent hydrothermal reaction at 180 °C for 2 h, after which it was cooled to 25 °C. The resulting suspension was centrifuged with deionized water and ethanol several times to remove the supernatant. The resulting black  $\text{MoS}_2$  nanosheets were dried overnight in a vacuum oven at 80 °C. Subsequently, 10 mg of  $\text{MoS}_2$  nanosheets, 4 mg of SWCNTs, and 20 mg of sodium dodecylbenzenesulfonate as the suspension agent were suspended in a solution of deionized water (10 mL).

### 2.2 Fabrication of flexible $\text{MoS}_2$ /SWCNT-based gas sensor

SEBS was mixed with toluene in the mass ratio of 2:5 and stirred for 24 h at room temperature. The solution was used to fabricate a 120  $\mu\text{m}$ -thick SEBS layer on a polyethylene terephthalate (PET) substrate *via* bar coating using a 4-side applicator. The applicator can be used to adjust the thickness of SEBS. Further, PU was then dissolved in *N,N*-dimethylformamide (DMF) in the mass ratio of 1:4. PU patterns were fabricated on top of the SEBS layer by screen printing using shadow mask and squeeze in Fig. 1(b) and (c).  $\text{MoS}_2$  and SWCNTs were then spin-coated onto the PU and SEBS layers at 2000 rpm for 30 s in Fig. 1(d). Finally, as indicated in Fig. 1(f), the silver electrode was screen-printed on the PU with a channel of 1000  $\mu\text{m}$  and a length of 100  $\mu\text{m}$  shadow mask and squeeze.

### 2.3 Material characterization and gas-sensing measurements using $\text{MoS}_2$ /SWCNT-based gas sensors

The morphologies of the synthesized  $\text{MoS}_2$  nanosheets were investigated through transmission electron microscopy (TEM, Titan Cube G2 60-300, FEI company). X-ray diffraction (XRD, Rigaku) was conducted to identify the crystal phase of the synthesized  $\text{MoS}_2$  nanosheets with  $\text{Cu-K}\alpha$  radiation ( $\lambda = 1.5418 \text{ \AA}$ ) at a current of 40 mA and voltage of 40 kV. For performing chemical-information Raman spectroscopy (inVia Raman spectrometer, Renishaw), X-ray photoelectron spectroscopy (XPS, K-alpha, Thermo Scientific) with an  $\text{Al-K}\alpha$  radiation ( $h\nu = 1486.6 \text{ eV}$ ) was used to examine the compositions of the  $\text{MoS}_2$  nanosheets. The morphological structures of the  $\text{MoS}_2$ /SWCNT-based gas sensor samples were characterized by field-emission scanning electron microscopy (FE-SEM, Hitachi, S-4700) and energy-dispersive X-ray spectroscopy (EDS).

The electrical properties of the sensors were measured using a semiconductor parameter analyzer (Keithley-4200, Keithley Instruments, USA).  $\text{NO}_2$ ,  $\text{CO}$ ,  $\text{H}_2\text{S}$ ,  $\text{NH}_3$ , acetone, and ethanol gases were individually injected into the sensing chamber to analyze the resistance of the sensors toward them; the sensors were placed 2 cm from the gas inlet, and gas-sensing measurements were carried out at room temperature and under 25% relative humidity.

## 3 Results and discussion

The screen-printing technique was used to fabricate a  $\text{MoS}_2$ /SWCNT-based gas sensor, as illustrated in Fig. 1. In the first instance, a 120  $\mu\text{m}$ -thick SEBS layer was fabricated on a PET substrate using the 4-side applicator (Fig. 1(a)). Next, the PU patterns were printed on top of the SEBS layer. When we spin-coated the mixture of  $\text{MoS}_2$  and SWCNTs onto the PU and SEBS patterns, we only observed the assembling of nanostructures on the PU pattern, which has a hydrophilic surface. The hydrophobic SEBS layer prevented adsorption on the

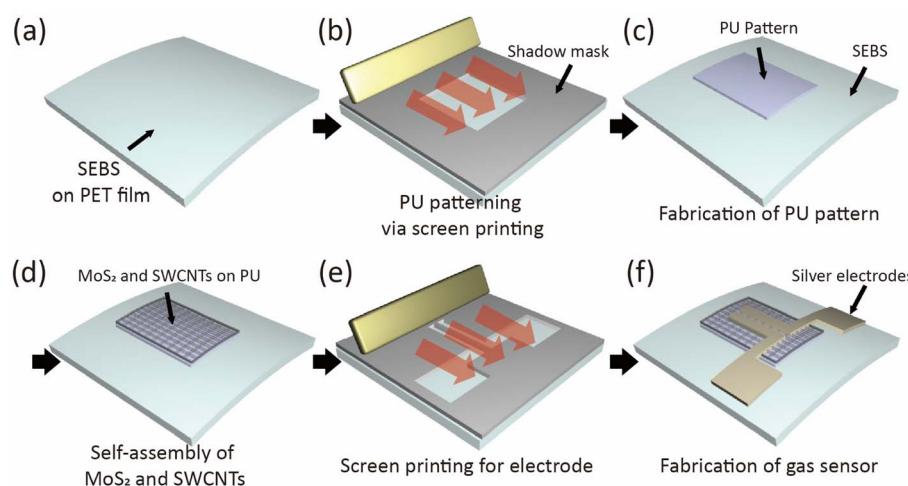


Fig. 1 Schematic of the fabrication process of  $\text{MoS}_2$ /SWCNT-based gas sensor. (a) Screen printing styrene-ethylene/butylene-styrene (SEBS) on a polyethylene terephthalate (PET) film. (b) Screen printing polyurethane (PU) through shadow mask on SEBS. (c) Fabrication of PU pattern. (d)  $\text{MoS}_2$ /SWCNT on PU by self-assembly. (e) Screen printing for silver electrode. (f) Fabrication of  $\text{MoS}_2$ /SWCNT gas sensor.



SWCNTs. Then, Fig. 2(a)–(d) displays the results of the TEM, XRD, Raman spectroscopy and XPS analyses for the synthesized MoS<sub>2</sub>. As shown in Fig. 2(a(i)) the interlayer distance of the MoS<sub>2</sub> nanosheets was approximately 0.65 nm, which corresponds to the (002) plane of bulk MoS<sub>2</sub>. The lattice *d*-spacing was estimated to be 0.25 nm, which corresponds to the (100) lattice plane of the MoS<sub>2</sub> phase; this concurs with the XRD results. Fig. 2(a(ii)) MoS<sub>2</sub> selected area electron diffraction (SAED) patterns indicating the hexagonal lattice structure of MoS<sub>2</sub> nanosheets. Fig. 2(a(iii) and (iv)) displays crumpled MoS<sub>2</sub> nanosheets with a size of hundreds of nanometers.

Fig. 2(b) illustrates the XRD spectra of the MoS<sub>2</sub> nanosheets; the characteristic peaks of the MoS<sub>2</sub> nanosheets were observed at 11.52°, 33.90°, and 56.62°, corresponding to the (002), (100), and (110) planes, respectively. Fig. 2(c) represents the Raman spectra ( $\lambda_{\text{exc}} = 532$  nm) displaying the signals for the in-plane vibrational mode (E<sub>2g</sub><sup>1</sup>) and out-of-plane vibrational mode (A<sub>1g</sub>) of MoS<sub>2</sub> at 383.02 and 408.14 cm<sup>-1</sup>, respectively. Fig. 2(d)

illustrates the XPS results for Mo 3d and S 2p. For Mo 3d, two strong peaks are located at 229.6 and 232.8 eV, which can be ascribed to Mo 3d<sub>5/2</sub> and Mo 3d<sub>3/2</sub>. The small peak at 226.8 eV was assigned to the S 2s of MoS<sub>2</sub>. Other observed peaks at 162.4 and 163.6 eV were assigned to S in 2H-MoS<sub>2</sub>. This confirms the successful synthesis of MoS<sub>2</sub>.

Fig. 3(a) depicts the SEM images of PU and SEBS after the assembly of MoS<sub>2</sub> and the SWCNTs. These results indicate that, upon spin coating, both MoS<sub>2</sub> nanosheets and SWCNTs assembled selectively on the PU region, and SWCNTs could not be observed on the hydrophobic SEBS surface. Here, the density of MoS<sub>2</sub>/SWCNT adhering to the PU surface can be controlled by adjusting the concentration of the MoS<sub>2</sub>/SWCNT solution. The hydrophilic surface of PU adsorbed MoS<sub>2</sub> and SWCNTs, and the hydrophobic SEBS layer repelled SWCNTs. The EDS measurements also confirmed the uniform assembly of MoS<sub>2</sub>/SWCNT network patterns on the PU substrate (Fig. 3(b)). The MoS<sub>2</sub>/SWCNT network on PU was composed of O, S, Mo, and C. Finally, a silver layer with a channel of 1000 μm and length of 100 μm was screen-printed on the PU surface. It acts as the conductive metallic layer required to complete the fabrication of the MoS<sub>2</sub>/SWCNT-based gas sensors. As indicated in the SEM images and EDS analyses of PU and SEBS boundary after the assembly of MoS<sub>2</sub> and the SWCNTs, MoS<sub>2</sub> nanosheets were assembled on both PU and SEBS, whereas the SWCNTs were observed only on PU (Fig. S1 in ESI†). Additionally, the MoS<sub>2</sub>/SWCNT assembled on PU was analyzed by using XPS in order to confirm the chemical state and bonding of MoS<sub>2</sub>/SWCNT in Fig. S2 in ESI†.

Fig. 4(a) shows the response of the sensor to 100 ppm NO<sub>2</sub> observed during the two testing cycles at room temperature; the cyclic nature of the response demonstrates the sensor's repeatable gas response and recovery in Fig. S3 in ESI†. It is well-known that, in the presence of MoS<sub>2</sub> and SWCNTs, NO<sub>2</sub> acts as an electron acceptor while NH<sub>3</sub> acts as an electron donor. These results clearly demonstrated the enhanced response of the MoS<sub>2</sub>/SWCNT system to both NO<sub>2</sub> and NH<sub>3</sub>, in which the resistance of the device decreased with NO<sub>2</sub> adsorption and increased with NH<sub>3</sub> adsorption. In previous reports, p-type carbon materials, such as graphene or SWCNTs functionalized with n-type MoS<sub>2</sub> nanosheets were found to behave as p-type materials. Similarly, the analyses led us to conclude that the proposed MoS<sub>2</sub>/SWCNT hybrid structure was a p-type semiconductor material. The sensing mechanism of the NO<sub>2</sub> gas-sensor MoS<sub>2</sub>/SWCNT was explained as in previous references. As NO<sub>2</sub> gathers a free electron from the MoS<sub>2</sub> and SWCNTs, MoS<sub>2</sub>/SWCNT exhibits p-type semiconductor properties. The two components cause the generation of more holes when hybridized. Hence, when the sensor was exposed to NO<sub>2</sub>, its resistance decreased and sensitivity increased. We performed a cycling test to evaluate the stability of the gas sensors in Fig. 4(a). The curves exhibited reproducible responses when air and NO<sub>2</sub> were injected alternately. The sensor exhibited a sensitivity of 88% to 100 ppm of NO<sub>2</sub> at room temperature. The gas sensitivity was calculated as  $\Delta R/R_a = (R_g - R_a)/R_a$ , where  $R_a$  and  $R_g$  are the resistances of the sensor to synthetic air and the target gas, respectively.<sup>14,33–35</sup>

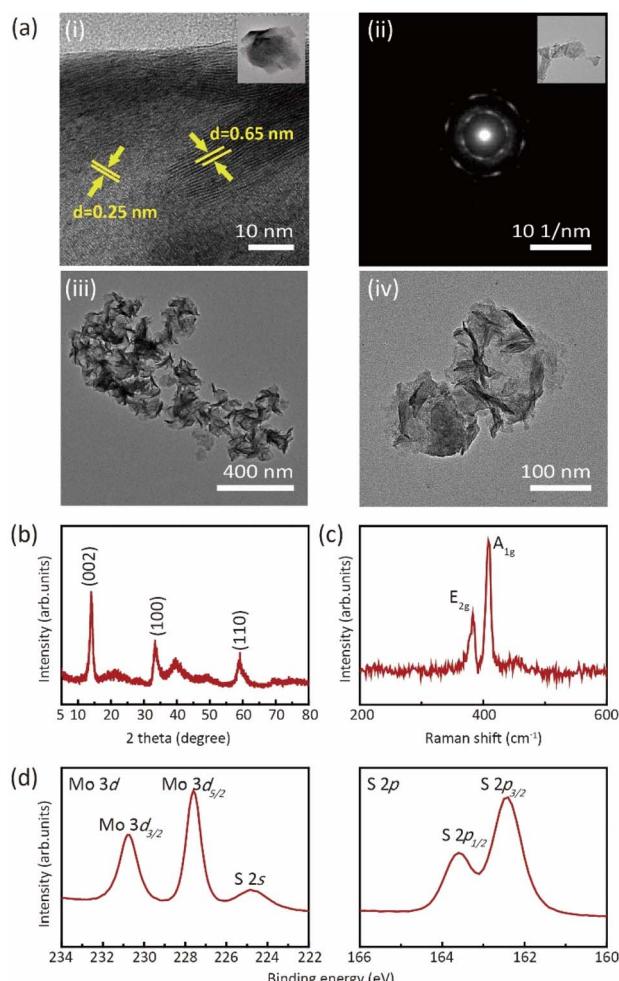


Fig. 2 (a) Transmission electron microscopy (TEM) images of MoS<sub>2</sub> nanosheets: (i) TEM images of MoS<sub>2</sub> show the lattice distance. (ii) Selected area electron diffraction (SAED) pattern. (iii and iv) TEM images of MoS<sub>2</sub> nanosheets. (b) X-ray diffraction (XRD) patterns of MoS<sub>2</sub> nanosheets (c) Raman spectra of MoS<sub>2</sub>. X-ray photoelectron spectroscopy (XPS) spectra of (d) Mo 3d and S 2p.



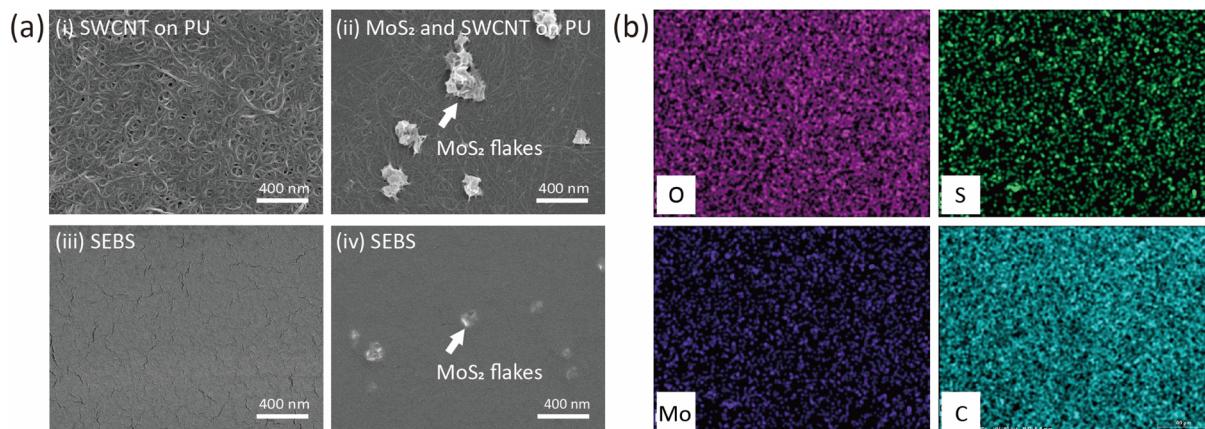
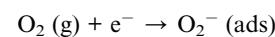


Fig. 3 (a) Scanning electron microscopy (SEM) images of MoS<sub>2</sub>/SWCNT on PU and SEBS: (i) SWCNTs self-assembly on PU (ii) MoS<sub>2</sub>/SWCNT self-assembly on PU (iii) SWCNTs on SEBS (iv) MoS<sub>2</sub>/SWCNT on SEBS (b) energy dispersive spectroscopy (EDS) elemental mapping of MoS<sub>2</sub>/SWCNT O, S, Mo, and C elements on PU substrate.

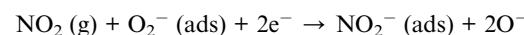
At various NO<sub>2</sub> concentrations, the detection limit for NO<sub>2</sub> gas ranged from 50 ppb to 1 ppm (Fig. 4(b)). In Fig. 4(b), detection of 50 ppb of NO<sub>2</sub> can be regarded as the minimum detection limit of the MoS<sub>2</sub>/SWCNT gas sensor, with a response value of 4.5%. These results indicate good performance compared to other studies for NO<sub>2</sub> gas sensors using MoS<sub>2</sub> and graphene hybrid materials in Table S1 in ESI.† When the concentration was increased from 50 ppb to 1 ppm, the response increased proportionally (Fig. 4(c)). As the relationship between concentration and sensitivity was linear, we concluded that the structure of the nanomaterial did not affect the sensing performance; therefore, the proposed fabrication method can be used with any nanoscale material, including nanowires, nanotubes, and nanoparticles. The response of the MoS<sub>2</sub>/SWCNT-based gas sensor to various other gases (CO, H<sub>2</sub>S, NH<sub>3</sub>, acetone vapor, and ethanol vapor) at room temperature was also investigated (Fig. 5). We tested 100 ppm of a specific gas at room temperature in two cycles. The responses to CO, H<sub>2</sub>S, and NH<sub>3</sub> were 2.6%, 7.8%, and 14.8%, respectively, with no response to acetone or ethanol gases in Fig. 5(a)–(e). In contrast to the resistance change caused by NO<sub>2</sub>, the resistance of the MoS<sub>2</sub>/SWCNT-based gas sensor to NH<sub>3</sub> and H<sub>2</sub>S gases increased with the adsorption of their molecules. This

is because NO<sub>2</sub> accepts electrons, whereas NH<sub>3</sub> and H<sub>2</sub>S act as electron donors. The response value of 100 ppm NO<sub>2</sub> was 11.3 times that of 100 ppm H<sub>2</sub>S and 5.9 times that of 100 ppm NH<sub>3</sub> (Fig. 5(f)). This substantiates the gas sensor's excellent selectivity to NO<sub>2</sub> at room temperature.

As illustrated in Fig. 4, the response of the sensor decreases for NO<sub>2</sub> gas. First, the material is exposed to air, and O<sub>2</sub> molecules are adsorbed and form O<sub>2</sub><sup>−</sup> ions on the material surface.



Upon exposure to the material, NO<sub>2</sub> gas becomes NO<sub>2</sub><sup>−</sup> by accepting electrons from the O<sub>2</sub><sup>−</sup> ions.



This confirms that the gas-sensing material increased the concentration of holes on the surface of the material. As shown

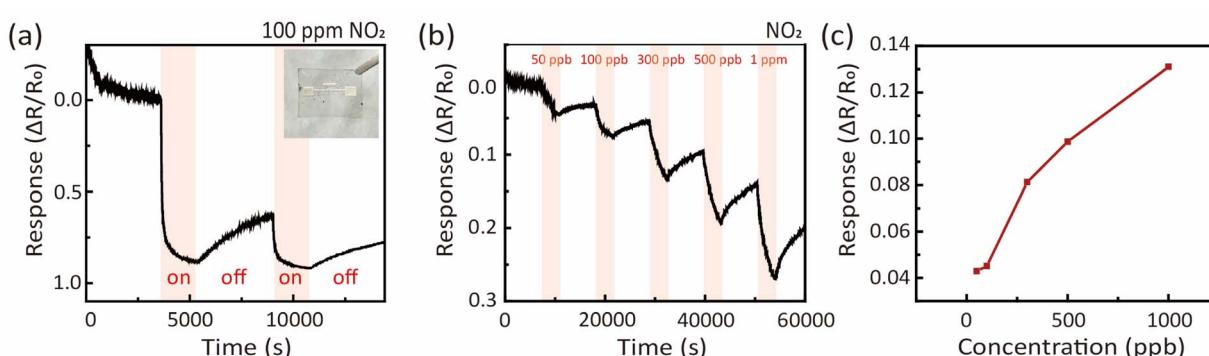
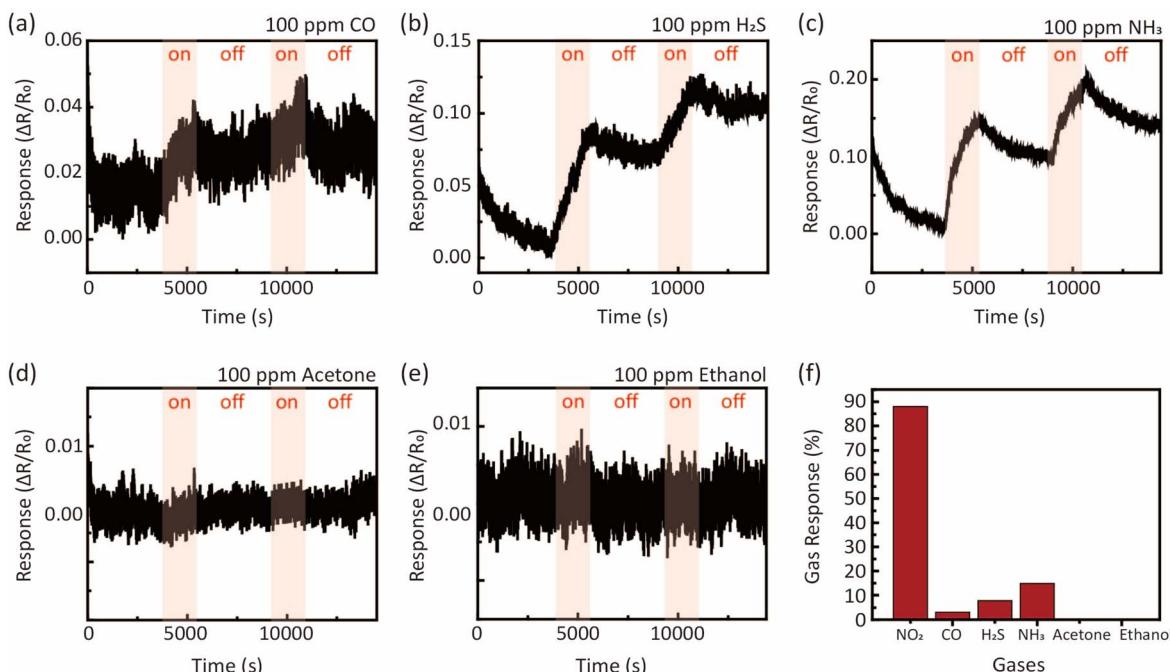


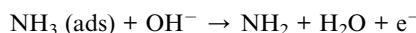
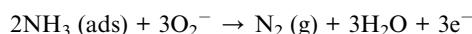
Fig. 4 The response of NO<sub>2</sub> gas at room temperature. (a) Two successive sensing responses at NO<sub>2</sub> 100 ppm, (b) dynamic responses and recovery curves of MoS<sub>2</sub>/SWCNT gas sensor towards NO<sub>2</sub> gas at room temperature (c) fitting curve of response versus concentration.





**Fig. 5** Gas response performance of MoS<sub>2</sub>/SWCNT sensor at room temperature 100 ppm. (a) CO gas response, (b) H<sub>2</sub>S gas response, (c) NH<sub>3</sub> gas response, (d) acetone gas response, (e) ethanol gas response, and (f) gas selectivity summary of the sensor to various gases at room temperature.

in Fig. 5(c), the transient resistance responses of the sensor to NO<sub>2</sub> and NH<sub>3</sub> were compared. In contrast to the resistance change caused by NO<sub>2</sub>, the resistance increased when exposed to NH<sub>3</sub> because the MoS<sub>2</sub>/SWCNT sensor is a p-type semiconductor.



When the MoS<sub>2</sub>/SWCNT-based sensor was exposed to NH<sub>3</sub>, following this mechanism, the O species reacted with NH<sub>3</sub>. Then the electrons were released back to MoS<sub>2</sub>/SWCNT, causing an increase in the electron concentration in the conduction band of MoS<sub>2</sub>. The transferred electrons recombined with the carriers, thus decreasing the charge carrier concentration and consequently increasing the electrical resistance.<sup>34–36</sup> Therefore, owing to the synergistic actions of MoS<sub>2</sub> and SWCNTs, the MoS<sub>2</sub>/SWCNT-based gas sensor exhibited high sensitivity to NO<sub>2</sub> gas.

## 4 Conclusions

We demonstrated a facile method of fabricating high-performance MoS<sub>2</sub>/SWCNT-based gas sensors *via* screen printing, making it a rapid, simple, and cost-effective technology. While MoS<sub>2</sub> assembled itself on both SEBS and PU, the SWCNTs attached only to the hydrophilic PU; therefore, MoS<sub>2</sub>/SWCNT hybrid nanostructures were successfully assembled on

PU substrates. The MoS<sub>2</sub>/SWCNT-based gas sensor exhibited high selectivity for NO<sub>2</sub> at room temperature, as well as high performance, reliable response, and recovery. In particular, the response to 100 ppm NO<sub>2</sub> at room temperature was 11.3 and 5.9 times higher than that to 100 ppm H<sub>2</sub>S and NH<sub>3</sub>, respectively. Moreover, the hybrid gas sensor was fabricated on a flexible substrate; therefore, it should be applicable in sensitive wearable electronics.

## Conflicts of interest

There are no conflicts to declare.

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