



## Article

# SnO<sub>2</sub> Nanowire/MoS<sub>2</sub> Nanosheet Composite Gas Sensor in Self-Heating Mode for Selective and ppb-Level Detection of NO<sub>2</sub> Gas

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**Abstract:** The development of low-cost and low-power gas sensors for reliable NO<sub>2</sub> gas detection is important due to the highly toxic nature of NO<sub>2</sub> gas. Herein, initially, SnO<sub>2</sub> nanowires (NWs) were synthesized through a simple vapor–liquid–solid growth mechanism. Subsequently, different amounts of SnO<sub>2</sub> NWs were composited with MoS<sub>2</sub> nanosheets (NSs) to fabricate SnO<sub>2</sub> NWs/MoS<sub>2</sub> NS nanocomposite gas sensors for NO<sub>2</sub> gas sensing. The operation of the sensors in self-heating mode at 1–3.5 V showed that the sensor with 20 wt.% SnO<sub>2</sub> (SM-20 nanocomposite) had the highest response of 13 to 1000 ppb NO<sub>2</sub> under 3.2 V applied voltage. Furthermore, the SM-20 nanocomposite gas sensor exhibited high selectivity and excellent long-term stability. The enhanced NO<sub>2</sub> gas response was ascribed to the formation of n-n heterojunctions between SnO<sub>2</sub> NWs and MoS<sub>2</sub>, high surface area, and the presence of some voids in the SM-20 composite gas sensor due to having different morphologies of SnO<sub>2</sub> NWs and MoS<sub>2</sub> NSs. It is believed that the present strategy combining MoS<sub>2</sub> and SnO<sub>2</sub> with different morphologies and different sensing properties is a good approach to realize high-performance NO<sub>2</sub> gas sensors with merits such as simple synthesis and fabrication procedures, low cost, and low power consumption, which are currently in demand in the gas sensor market.

**Keywords:** MoS<sub>2</sub> nanosheet; SnO<sub>2</sub> NWs; self-heating; NO<sub>2</sub> gas; sensing mechanism



**Citation:** Kim, J.-Y.; Mirzaei, A.; Kim, J.-H. SnO<sub>2</sub> Nanowire/MoS<sub>2</sub> Nanosheet Composite Gas Sensor in Self-Heating Mode for Selective and ppb-Level Detection of NO<sub>2</sub> Gas. *Chemosensors* **2024**, *12*, 107. <https://doi.org/10.3390/chemosensors12060107>

Academic Editor: Boris Lakard

Received: 10 May 2024

Revised: 7 June 2024

Accepted: 7 June 2024

Published: 9 June 2024



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## 1. Introduction

NO<sub>2</sub> is a highly dangerous gas emitted from industrial activities, fuel combustion, biomass burning, and electricity generation [1]. NO<sub>2</sub> can affect global air quality and human health [2]. Long-term exposure to parts per million (ppm) levels of NO<sub>2</sub> can cause infections in the respiratory tract and lungs. Asthma, tissue hypoxia, pulmonary edema, and cardiovascular disease are affected by the presence of NO<sub>2</sub> gas [3–5]. Additionally, NO<sub>2</sub> contributes to the formation of acid rain and reduces the visibility of atmospheric photochemical smog [6,7]. Therefore, the threshold limit for NO<sub>2</sub> gas is set at 3 ppm [8]. In addition to its toxic effects, NO<sub>2</sub> gas is also considered a biomarker of lung infections [9]. Thus, the detection of NO<sub>2</sub> gas is highly important from safety and health perspectives.

Semiconducting metal oxides are often used for the detection of toxic gases. However, they often need a high temperature to show their best sensing performance [10,11]. Transition metal dichalcogenides (TMDs) with a two-dimensional (2D) nanosheet (NS) nature have the general formula MX<sub>2</sub> (M = Mo or W; X = S, Se, or Te), in which the metal layers are sandwiched between two chalcogen layers [12,13]. They can be used in different applications as a gas adsorbent [14], microwave adsorbent [15], and gas sensor.

WS<sub>2</sub> [16], WSe<sub>2</sub> [17], MoS<sub>2</sub> [18], and MoSe<sub>2</sub> [19] are the most important TMDs for gas sensing studies. In particular, MoS<sub>2</sub> has features such as fast charge transfer, adjustable band gap, high carrier mobility, and large surface area owing to its 2D nature, making it a favorable TMD for gas sensing applications, particularly low- or room-temperature gas

sensing [20]. However, its sensing properties in pristine form are generally not adequate for the high standards of today's life. Accordingly, it can be decorated [21], doped [22], or composited with other materials [23,24] to enhance its sensing properties. In particular, composite fabrication leads to the formation of heterojunctions, which can provide an additional source of resistance modulation, ultimately leading to significant resistance modulation [25].

Semiconducting *n*-type SnO<sub>2</sub> ( $E_g = 3.37$  eV) [26] has high electron mobility, high availability, ease of synthesis, high stability, and excellent gas sensing properties [27,28]. Accordingly, different morphologies of SnO<sub>2</sub>-like nanoparticles (NPs) [29], nanorods [30], nanobelts [31], nanotubes [32], nanofibers [33], and nanowires (NWs) [34] have been used for the detection of various gases. Even though some room temperature SnO<sub>2</sub> gas sensors have been reported in the literature [35], SnO<sub>2</sub> gas sensors often require high temperatures to achieve their best performance.

Thus, SnO<sub>2</sub>-MoS<sub>2</sub> nanocomposites are a good choice for gas sensing [36–39], combining the relatively good sensitivity of MoS<sub>2</sub> at room temperature (RT) with the high sensitivity of SnO<sub>2</sub> at higher temperatures, resulting in the realization of a room-temperature or relatively low-temperature gas sensor with good performance. For example, Bai et al. [40] reported the growth of vertically aligned MoS<sub>2</sub> on SnO<sub>2</sub> nanotubes for the room-temperature detection of NO<sub>2</sub> gas with a response of approximately 35 to 100 ppm NO<sub>2</sub> gas. In addition, polyaniline-MoS<sub>2</sub>-SnO<sub>2</sub> nanotubes were reported as room-temperature ammonia gas sensors [41]. Wang et al. [42] used SnO<sub>2</sub> NPs-MoS<sub>2</sub> NSs for ammonia sensing at RT. Viet et al. [43] decorated MoS<sub>2</sub> NSs on SnO<sub>2</sub> NWs to detect and discriminate between CO, NH<sub>3</sub>, and H<sub>2</sub> gases. Xu et al. [44] used MoS<sub>2</sub> NSs/SnO<sub>2</sub> nanotubes for the detection of trimethylamine at 200 °C. Han et al. reported a MoS<sub>2</sub> NSs-SnO<sub>2</sub> NPs composite gas sensor with an 18.7–5 ppm response of NO<sub>2</sub> gas at RT [45]. Anyway, less attention has been paid to the composites of SnO<sub>2</sub> NWs with MoS<sub>2</sub> NSs. SnO<sub>2</sub> NWs with strong intrinsic gas sensing features, high surface area, and one-dimensional (1D) nature, in combination with MoS<sub>2</sub> NSs, can generate numerous heterojunctions, which offer new opportunities for the detection of NO<sub>2</sub> gas. Additionally, the operation of gas sensors in self-heating conditions is a promising approach to not only significantly decrease the sensing temperature but also remarkably lower power consumption. Hence, self-heated sensors offer opportunities for application in places with limited energy access.

In this study, SnO<sub>2</sub> NWs were initially produced using a vapor–liquid–solid (VLS) growth mechanism, which is a simple, low-cost, and highly efficient method for the synthesis of metal oxide NWs [46]. Afterward, SnO<sub>2</sub> NWs (10, 20, and 30 wt.%) were composited with MoS<sub>2</sub> NSs. Overall, the synthesis procedure is highly cost-effective, and even large-scale synthesis is feasible for possible industrial applications. After different advanced characterizations, gas sensors were fabricated, and the sensor with 20 wt.% SnO<sub>2</sub> NWs revealed the highest response to NO<sub>2</sub> gas under 3.2 V in self-heating mode with parts per billion (ppb)-level detection ability, high selectivity, and long-term stability. The enhanced NO<sub>2</sub> gas sensing performance was mainly related to the formation of SnO<sub>2</sub>-MoS<sub>2</sub> n-n heterojunctions and the high surface area of the nanocomposite. We believe that the optimal sensor developed in this study, with low power consumption, low synthesis cost, and high sensing performance, can be regarded as a potential choice for industrial and practical applications.

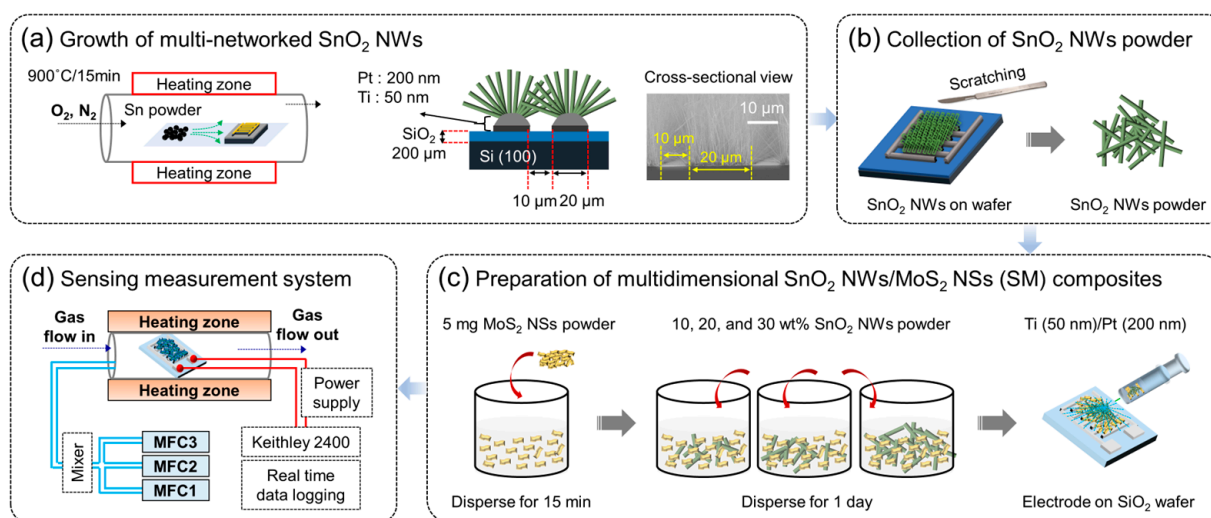
## 2. Materials and Methods

### 2.1. Starting Materials

Metallic Sn powders with high purity of 99.5% (Merck, <150 μm size, Darmstadt, Germany) were used for growth of SnO<sub>2</sub> NWs. Also, commercial MoS<sub>2</sub> NSs (ACS Material, 100–200 nm, Pasadena, CA, USA) were used to prepare the SnO<sub>2</sub>/MoS<sub>2</sub> nanocomposites.

## 2.2. Synthesis of SnO<sub>2</sub> NWs and SnO<sub>2</sub> NWs/MoS<sub>2</sub> NS Composite

Networked SnO<sub>2</sub> NWs were synthesized via a VLS growth mechanism similar to that reported in a previous study [47]. First, high-purity metallic Sn powder was put in a crucible inside a tubular furnace. A SiO<sub>2</sub>-grown (200 μm) Si substrate equipped Ti (50 nm)/Pt (200 nm) bi-electrode was placed within a short distance. Then, the temperature was gradually increased in the presence of flowing O<sub>2</sub> (10 sccm) and N<sub>2</sub> (300 sccm) gases, and SnO<sub>2</sub> NWs were grown at 900 °C for 15 min on the substrate (Figure 1a). The SnO<sub>2</sub> NWs were then scratched from the substrate (Figure 1b) to form a composite with MoS<sub>2</sub> NSs. To prepare the composite, 5 mg MoS<sub>2</sub> NSs were mixed with 10, 20, and 30 wt.% SnO<sub>2</sub> NWs (denoted as SM-10, SM-20, and SM-30, respectively) under magnetic stirring for 24 h (Figure 1c).



**Figure 1.** (a) Schematic of SnO<sub>2</sub> NWs grown via VLS mechanism on the surface of substrate equipped with electrodes. (b) Scratching of SnO<sub>2</sub> NWs for characterizations. (c) Preparation of SnO<sub>2</sub>/MoS<sub>2</sub> composites. (d) Gas sensing measuring system.

## 2.3. Characterizations

Field-emission scanning electron microscopy (FE-SEM; Hitachi S-4200, Tokyo, Japan) and transmission electron microscopy (TEM; JEM2100F/JEOL, Tokyo, Japan) were used for morphological analysis. In FE-SEM, cold type was used as field emission gun with 15 kV power. X-ray photoelectron spectroscopy (XPS; K-Alpha/Thermo scientific, Seoul, Republic of Korea) was used for compositional analysis of the surface elements. Monochromated Al K $\alpha$  was used as the X-ray source. The surface area was evaluated using the Brunauer–Emmett–Teller method (BET, MICROMERITICS Tristar, Norcross, GA, USA) from the N<sub>2</sub> adsorption–desorption isotherms. Induced temperature due to the Joule effect during the operation of the sensor in self-heating mode was monitored using a thermometer (IT-480S, Horiba, Kyoto, Japan). Ultraviolet photoemission spectroscopy (UPS, Thermo Fisher Scientific Co. Theta probe, Seoul, Republic of Korea) was used to estimate the work function values. He I (21.22 eV) was used as the light source.

## 2.4. Gas Sensing Tests

First, 5 mg of the sensing material was mixed with ethanol, and 0.075 μL of the solution (in three drops) was drop-coated onto the SiO<sub>2</sub> substrate equipped with Ti (50 nm)/Pt (200 nm) bi-layer electrodes (Figure 1c). Also, digital images of fabricated sensor are provided in Figure S1. A lab-made gas-sensing apparatus was used for the experiments (Figure 1d). The chamber was placed inside a tubular quartz furnace connected to a Keithley 2400 source meter. The gas was mixed with dry air and injected into the gas chamber using MFCs at a total flow rate of 100 sccm. The resistances in air ( $R_a$ ) and in the presence of

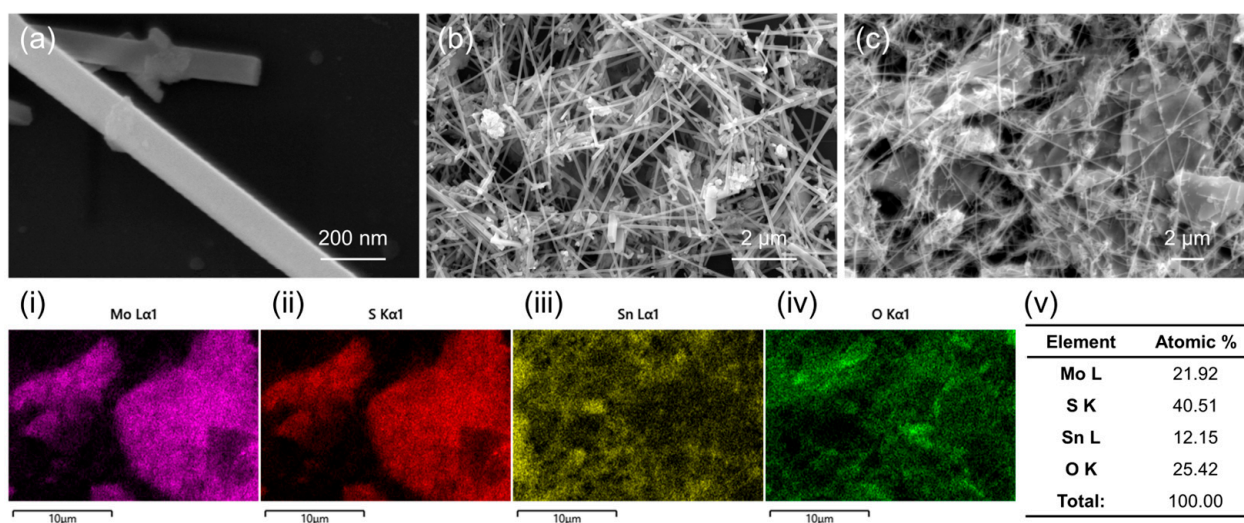
the target gas ( $R_g$ ) were measured constantly, and the sensor response was calculated as  $R = R_g/R_a$  for  $\text{NO}_2$  gas and vice versa for reducing gases. Additionally, the response time ( $\tau_{\text{res}}$ ) and recovery time ( $\tau_{\text{rec}}$ ) were defined as the times required for the resistance to reach its 90% final value after injection and stoppage of  $\text{NO}_2$  gas, respectively [48]. During the gas sensing tests, the relative humidity (RH) in the chamber was 30% at RT. However, to evaluate the effect of higher humidity on the gas response, 80% RH was introduced into the gas chamber and measured at RT.

### 3. Results and Discussion

#### 3.1. Characterization Studies

Figure S2a–c present low-magnification SEM images of pristine  $\text{SnO}_2$ , pristine  $\text{MoS}_2$ , and SM-20 nanocomposite samples, respectively.  $\text{SnO}_2$  NWs are densely packed, while  $\text{MoS}_2$  are loosely packed. Also, the SM-20 nanocomposite is comprised of both  $\text{SnO}_2$  NWs and  $\text{MoS}_2$  NSs with some voids among different components.

Figure 2a–c display SEM images of the SM-20 composite. In the high-magnification image (Figure 2a), the diameter of  $\text{SnO}_2$  NWs is approximately 60–100 nm. Furthermore, in the lower-magnification images (Figure 2b,c), the formation of a nanocomposite comprising NSs and NWs is demonstrated. Due to the 1D morphology of  $\text{SnO}_2$  NWs and 2D morphology of  $\text{MoS}_2$  NSs, there are some voids among them, which are advantageous for the diffusion of gases. SEM-TEM EDS mapping analysis of different elements, namely Mo (panel i), S (panel ii), Sn (panel iii), and O (panel iv), shows that the composition of NSs is  $\text{MoS}_2$  and the composition of NWs is  $\text{SnO}_2$ . SEM-EDS compositional analysis is presented in panel v of Figure 2. The amounts of Mo, S, Sn, and O elements were 21.92, 40.51, 12.15, and 25.42 at.%, respectively.

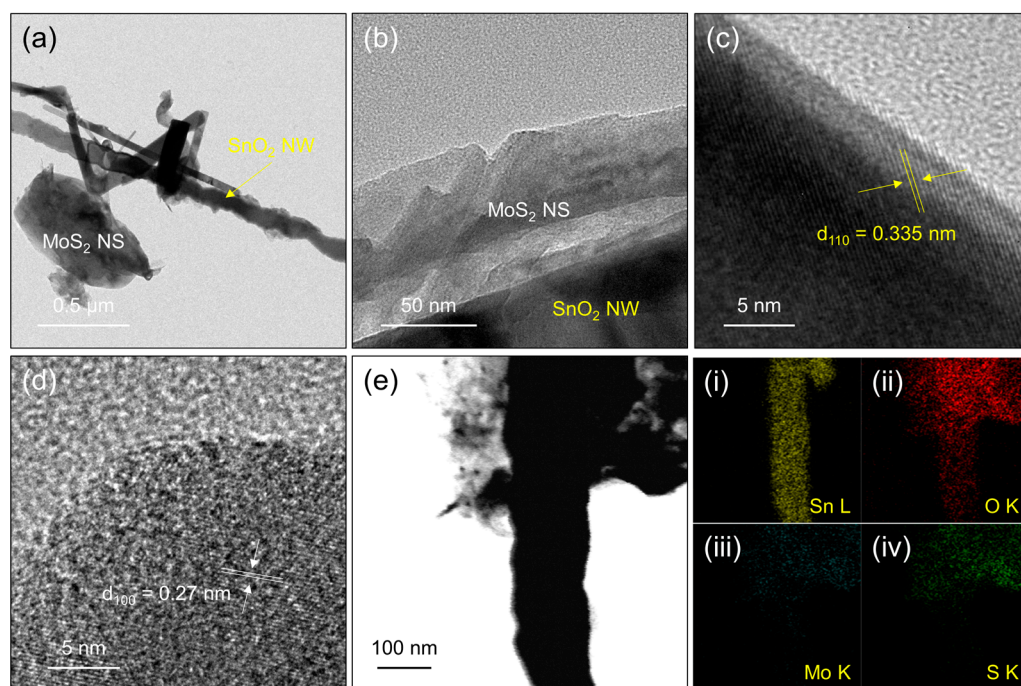


**Figure 2.** (a–c) SEM images of SM-20 composite. EDS mapping analysis of SM-20 composite: (i) Mo, (ii) S, (iii) Sn, and (iv) O mapping. (v) Compositional analysis of SM-20 composite.

Figure 3a,b show TEM views of the SM-20 nanocomposite at two different magnifications. Both  $\text{MoS}_2$  NSs and  $\text{SnO}_2$  NWs co-exist in the composite. High-resolution TEM (HRTEM) images of the SM-20 nanocomposite are shown in Figure 3c,d. The spacings between the parallel fringes are 0.335 and 0.27 nm, which correspond to (110) and (100) crystalline planes of  $\text{SnO}_2$  and  $\text{MoS}_2$ , respectively [49,50]. TEM-EDS elemental mapping is presented in Figure 3e, panel i–iv. Based on the distribution of Sn, O, Mo, and S in panel i–iv of Figure 3, the NW morphology is mainly composed of Sn and O and, therefore, has a composition of  $\text{SnO}_2$ , whereas NSs have an  $\text{MoS}_2$  composition. Figure S3a shows the XPS survey of the SM-20 composite. It shows the signals related to C (ambient carbon), Mo, S, Sn, and O, which demonstrates a high purity of the synthesized SM-20 composite.



Figure S3b displays the Mo 3d core-level region of the SM-20 composite, with two main peaks related to Mo 3d<sub>3/2</sub> and Mo 3d<sub>5/2</sub> at 233.1 and 229.9 eV, respectively, which can be attributed to Mo<sup>6+</sup> ions in MoS<sub>2</sub> [51]. Additionally, a peak related to S 2s is observed near the Mo 3d peaks. Figure S3c manifests the S 2p core-level region of the SM-20 composite. It is comprised of S 2p<sub>1/2</sub> and S 2p<sub>3/2</sub> peaks, corresponding to S<sup>2-</sup> ions in MoS<sub>2</sub> [52]. Figure S3d presents the Sn 3d core-level region of the SM-20 composite, where two high-intensity peaks at 495.5 and 487.1 eV with a separation of 8.4 eV belong to Sn 3d<sub>3/2</sub> and Sn 3d<sub>5/2</sub>, respectively, in SnO<sub>2</sub> [53]. The O 1s core-level region is also presented in Figure S3e.



**Figure 3.** Analysis of SM-20 nanocomposite (a,b) TEM images at two different magnifications. (c,d) HRTEM images. (e) TEM-EDS elemental mapping analysis displaying the distribution of (i) Sn, (ii) O, (iii) Mo, and (iv) S elements.

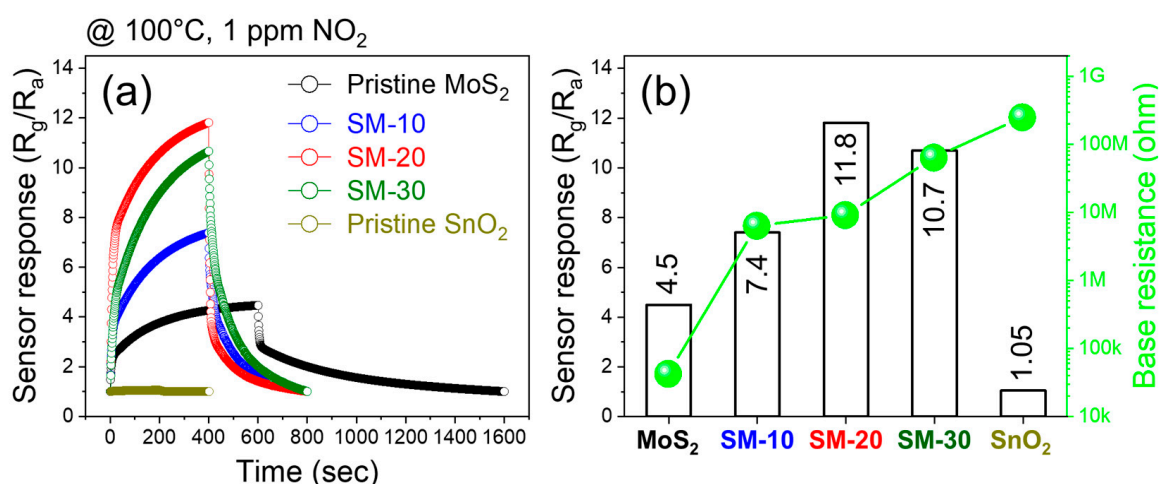
Figure S4a–e display the N<sub>2</sub> adsorption–desorption curves of different samples. Based on these curves, the surface areas of the pristine MoS<sub>2</sub> and pristine SnO<sub>2</sub>, SM-10, SM20, and SM-30 nanocomposites were samples were 0.65, 1.69, 2.14, 2.32, and 2.67 m<sup>2</sup>/g, respectively. Therefore, after composite formation, the surface area increased by approximately four times relative to the MoS<sub>2</sub> NSs. In addition, it was approximately 1.4 times higher relative to the SnO<sub>2</sub> NWs. Thus, the composite sensors are expected to provide more adsorption sites for NO<sub>2</sub> gas and a higher response. Also, even though the SM-30 sample has a higher surface area relative to the SM-20 sample, it is expected to show a lower response due to the fact that in the SM-30 sample, more SnO<sub>2</sub> NWs are present, which have poorer sensing properties relative to MoS<sub>2</sub> at low temperatures.

### 3.2. Gas Sensing Studies

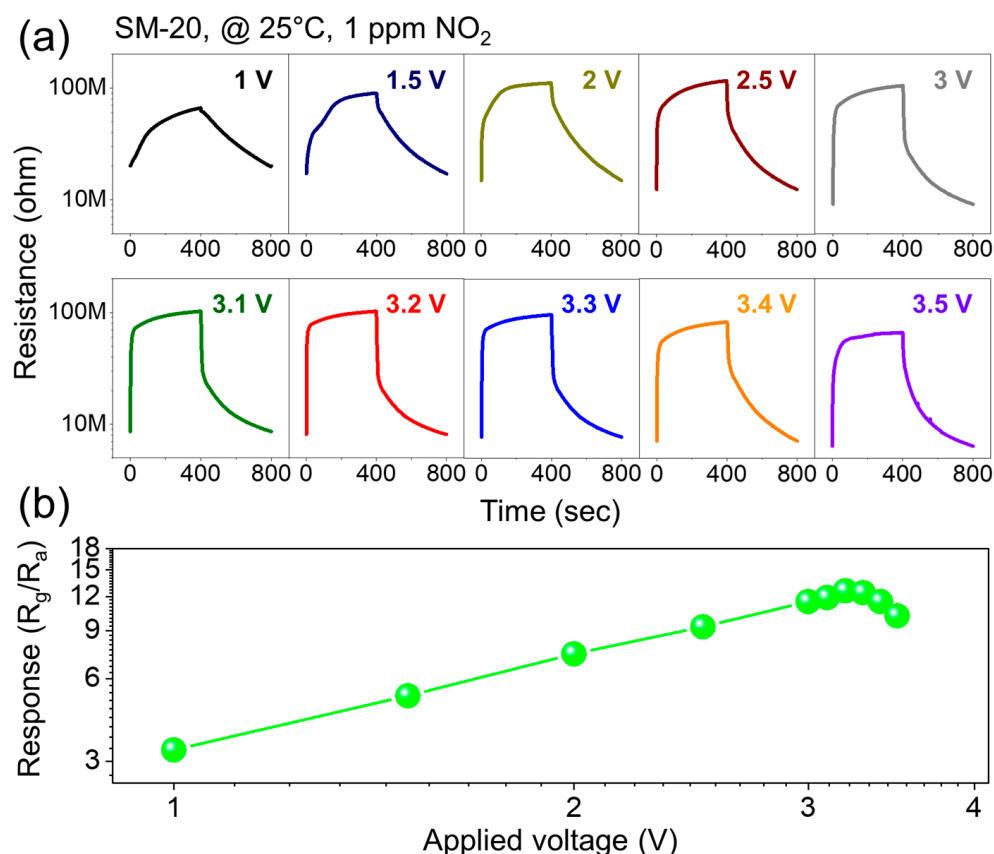
Figure S5a,b exhibit the dynamic resistance and dynamic response plots of the pristine MoS<sub>2</sub> NSs gas sensor to 1 ppm NO<sub>2</sub> at 25 °C (RH 30%) and higher temperatures (50–150 °C) under 1 V applied voltage, respectively. The resistance increased upon injection of the NO<sub>2</sub> gas, revealing the *n*-type nature of MoS<sub>2</sub>. For better insight, the corresponding NO<sub>2</sub> gas response and baseline resistance versus operating temperature are depicted in Figure S5c. The baseline resistance gradually decreased with increasing temperature due to the jumping of electrons to the conduction band under the influence of temperature. This behavior demonstrates the semiconducting nature of the MoS<sub>2</sub> gas sensor. Furthermore, the tracking of response versus temperature shows that the highest response occurs at 100 °C, with a

response of 4.5–1 ppm NO<sub>2</sub> gas. At lower temperatures, there is insufficient energy for NO<sub>2</sub> gas to be sufficiently adsorbed on the sensor surface, and at higher temperatures, the desorption rate surpassed the adsorption rate. At 100 °C, maximum net adsorption occurs, resulting in enhanced gas response. Figure S5d,e show dynamic resistance and response plots of pristine SnO<sub>2</sub> NW gas sensors to 1 ppm NO<sub>2</sub> at 25 °C (RH 30%) and higher temperatures (50–350 °C) under 1 V applied voltage, respectively. Additionally, the corresponding NO<sub>2</sub> gas response and baseline resistance versus operating temperature are plotted in Figure S5f. Similar to the MoS<sub>2</sub> NSs gas sensor, the SnO<sub>2</sub> NWs gas sensor displays an *n*-type semiconducting behavior. However, its optimal sensing temperature was at 300 °C, with a high response of 38–1 ppm NO<sub>2</sub> gas. Therefore, although the optimal sensing temperature (100 °C) of the MoS<sub>2</sub> NSs sensor was lower relative to that of the SnO<sub>2</sub> NWs gas sensor (300 °C), the response of the SnO<sub>2</sub> NWs sensor ( $R_g/R_a = 38$ ) was almost eight times that of the MoS<sub>2</sub> NSs gas sensor ( $R_g/R_a = 4.5$ ). Conversely, at 100 °C, which is considered a low temperature for sensors, the response of the MoS<sub>2</sub> NSs sensor ( $R_g/R_a = 4.5$ ) was more than four times higher than that of the SnO<sub>2</sub> NWs gas sensor ( $R_g/R_a = 1.05$ ). This implies that to achieve high-performance gas sensors at low temperatures, the presence of only SnO<sub>2</sub> NWs is insufficient, and they should be used in combination with other materials, such as MoS<sub>2</sub>, which have better sensing properties at low temperatures.

In the next step, we explored the NO<sub>2</sub> gas-sensing features of all fabricated gas sensors at 100 °C. Figure 4a displays the dynamic response curves of different gas sensors to 1 ppm NO<sub>2</sub> gas at 100 °C, and Figure 4b compares the response and baseline values of different gas sensors. The SnO<sub>2</sub> NWs gas sensor showed the lowest response of 1.05, whereas the response of the MoS<sub>2</sub> NSs gas sensor was 4.5. The responses of the SM-10, SM-20, and SM-30 sensors to 1 ppm NO<sub>2</sub> gas were 7.4, 11.8, and 10.7, respectively. Thus, all composite gas sensors exhibited a higher response than both the SnO<sub>2</sub> and MoS<sub>2</sub> gas sensors alone. In addition, among all gas sensors, the SM-20 composite exhibited the highest response; hence, it was selected for further study. In addition, the resistance of the gas sensors increased with increasing SnO<sub>2</sub> content, and pure MoS<sub>2</sub> and SnO<sub>2</sub> sensors exhibited the lowest and highest baseline resistances, respectively. Next, we exposed the SM-20 composite gas sensor to 1 ppm NO<sub>2</sub> gas at different applied voltages (1–3.5 V), as shown in Figure 5a. Figure 5b plots the response as a function of voltage. The response gradually increased with the applied voltage, and the maximum response occurred at 3.2 V. Thus, the optimal applied voltage was 3.2 V, and subsequent experiments were performed under this applied voltage.



**Figure 4.** (a) Sensing curves of different gas sensors to 1 ppm NO<sub>2</sub> gas at 100 °C. (b) Comparison of response to 1 ppm NO<sub>2</sub> gas at 100 °C and baseline resistance of different gas sensors.

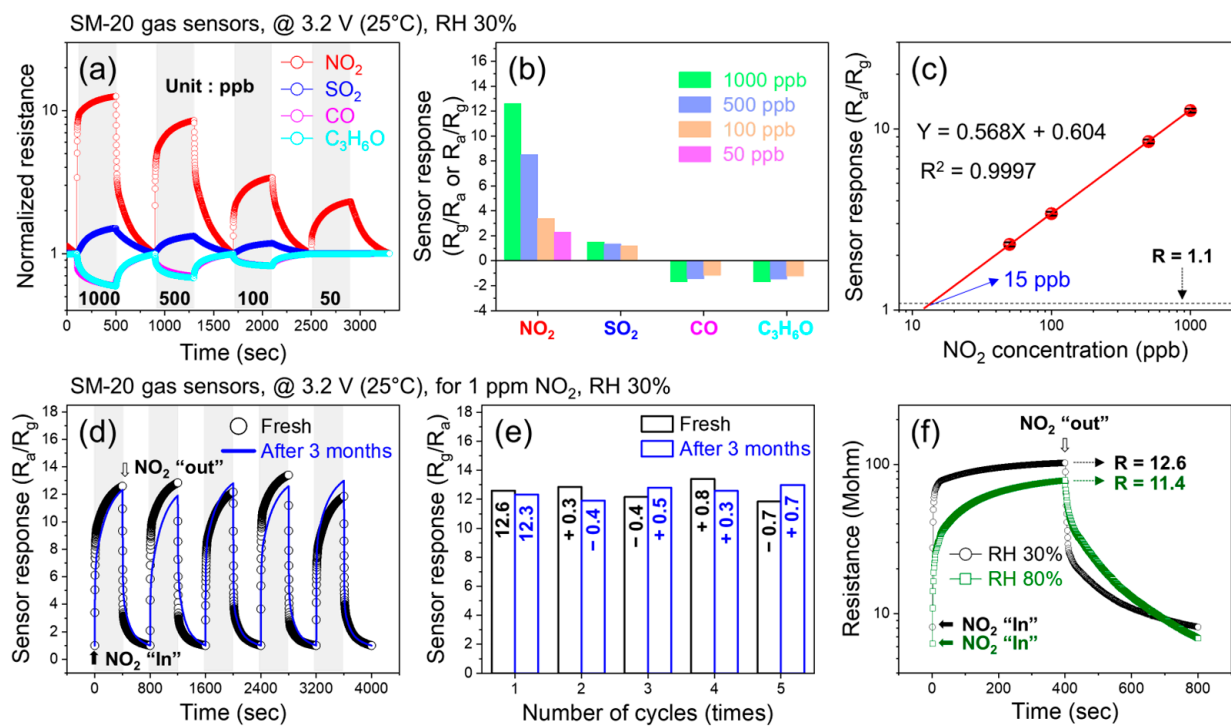


**Figure 5.** (a) Sensing curves of SM-20 gas sensor to 1 ppm NO<sub>2</sub> gas at 25 °C under various applied voltages. (b) Corresponding response of 1 ppm NO<sub>2</sub> gas at 25 °C versus applied voltage.

Figure 6a shows the dynamic normalized resistance curves of the SM-20 sensor at low concentrations of various gases at a fixed 3.2 V. The corresponding selectivity histogram is presented in Figure 6b. The responses to 1000 ppb NO<sub>2</sub>, SO<sub>2</sub>, CO, and C<sub>3</sub>H<sub>6</sub>O were 13, 2, 1.9, and 2, respectively. Thus, the sensor exhibited a much higher response to NO<sub>2</sub> gas than to other gases, demonstrating its high selectivity towards NO<sub>2</sub> gas. To check the reproducibility of the optimal sensor, we prepared three gas sensors under the same experimental procedures and checked their selectivity behavior, as shown in Figure S6a–c. All fabricated sensors revealed almost the same sensing response towards different gases, as shown in Figure S6d. Thus, the reproducibility of the sensor was demonstrated. In addition, the sensor exhibited a linear calibration curve for the detection of ppb levels of NO<sub>2</sub> gas (Figure 6c). Based on extrapolation to the y-axis, the experimental detection limit was 15 ppb, which was close to the theoretically calculated (Text S1 in Supporting Information) LOD (17.9). Figure 6d exhibits the sensing graphs (five cycles) of the SM-20 composite sensor in the fresh state and after three months of exposure to 1 ppm NO<sub>2</sub> gas at 3.2 V, and Figure 6e compares the responses in the fresh state and after three months. Overall, negligible differences were observed in the responses, even after three months. To be more quantitative, the average response and standard deviation of the sensor in the fresh state were 12.6 and 0.525, respectively, and those parameters for the sensor after three months were 12.52 and 0.386, respectively. If we define the stability factor as the average response after three months to the average response in the fresh state, it is 12.52/12.6 = 0.99. This demonstrates the good long-term stability of the sensor.

Finally, we explored the response of the SM-20 composite sensor at 80% RH (Figure 6f). The response to 1 ppm NO<sub>2</sub> gas at 3.2 V under dry conditions was 12.6, which decreased to 11.4 under humid (80% RH) conditions. Thus, although the response decreased in a humid environment, the sensor still exhibited a high response. In humid conditions, H<sub>2</sub>O molecules are adsorbed on the sensor surface, limiting the number of available adsorption

sites. Therefore, a smaller amount of NO<sub>2</sub> gas can be adsorbed onto the sensor surface, bringing about a decrease in the sensor response in humid atmospheres [54].



**Figure 6.** Sensing performance of SM-20 gas sensor. (a) Sensing curves at low concentrations of various gases at fixed 3.2 V. (b) Corresponding selectivity histogram. (c) Calibration curve for low concentrations of NO<sub>2</sub> gas. (d) Dynamic resistance curves (five cycles) in fresh state and after three months of 1 ppm NO<sub>2</sub> gas at 3.2 V. (e) Comparison of the responses in fresh state and after three months. (f) Dynamic resistance curves for 1 ppm NO<sub>2</sub> gas at 3.2 V under dry and humid (80% RH) conditions.

Table 1 compares the NO<sub>2</sub> gas-sensing properties of present work with those obtained in other studies, which demonstrates good performance of present sensor.

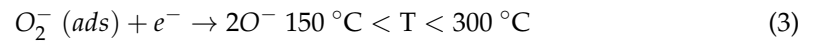
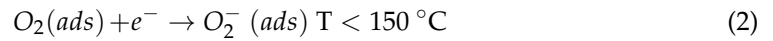
**Table 1.** Comparison of the NO<sub>2</sub> gas-sensing responses obtained in this study with those reported in other papers. The optimal sensor in this study has a higher performance in terms of high response, fast response, and recovery time relative to most of the listed sensors.

Sensing Material	T (°C)	Conc. (ppb)	Response ( $R_g/R_a$ ) or ( $R_a/R_g$ )	$\tau_{Res}/\tau_{Rec}$	Ref.
SnO <sub>2</sub> NWs (20 wt%)-MoS <sub>2</sub> NSs composite gas sensor	RT, (3.2 V)	1000	12.6	268/63 s	This work
WS <sub>2</sub> /Graphene heterostructure	RT	50	6.0	110/168 s	[55]
Nb-MoSe <sub>2</sub>	150	3000	8.0	30/30 min	[56]
WSe <sub>2</sub> nanosheets	RT	1000	8.21	50/1050 s	[57]
MoS <sub>2</sub> -rGO heterojunction	160	3000	1.24	8/20 s	[58]
Trilayer WSe <sub>2</sub> film	RT	10,000	2.8	960/600 s	[59]
3D crumpled reduced graphene oxide nanosheets	RT	1000	1.5	500/3000 s	[60]
SnO <sub>2</sub> -rGO nanocomposites	50	500	1.5	400/300 s	[61]
SnO <sub>2</sub> /Ti <sub>3</sub> C <sub>2</sub> T <sub>x</sub> nanocomposite	RT	300	78.2% [ $\Delta R/R_a$ (%)]	54/400 s	[62]
ZnO/Ti <sub>3</sub> C <sub>2</sub> T <sub>x</sub> nanocomposite	160	8	3.6	254/~380	[63]
MoS <sub>2</sub> /MXene nanocomposite	RT	100	65.6% [ $\Delta R/R_a$ (%)]	~700/~900	[64]
MoS <sub>2</sub> /Ti <sub>3</sub> C <sub>2</sub> T <sub>x</sub> nanocomposite	RT	20	65.6% [ $\Delta R/R_a$ (%)]	525/155	[65]
Ti <sub>3</sub> C <sub>2</sub> T <sub>x</sub> /CuO nanocomposite	RT	100	59% [ $\Delta R/R_a$ (%)]	~100/~100	[66]

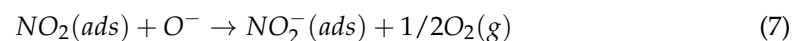


### 3.3. Gas-Sensing Mechanism

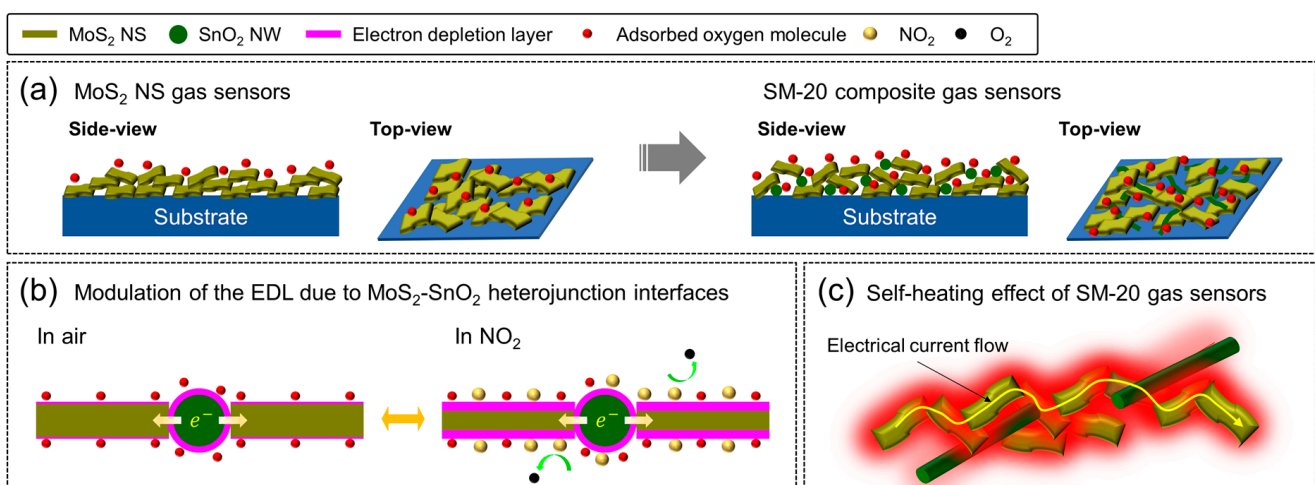
Initially, when the fresh sensors are in the air, oxygen gas is adsorbed on the sensor surface; because of the high electron affinity of oxygen, it takes electrons from the conduction band of the sensing material as follows [67].



Hence, at room temperature and at 300 °C, dominant oxygen species are  $O_2^-$  and  $O^-$ , respectively. The depletion of electrons from the exposed surfaces of the sensing layer with *n*-type semiconducting nature leads to the appearance of an electron depletion layer (EDL), where the concentration of electrons is lower than that in the core regions, resulting in the high resistance of *n*-type sensors in air. Upon exposure to  $NO_2$  gas, which is an oxidizing gas, more electrons are abstracted from the sensing layer as follows [8].



Consequently, the EDL width increases in the presence of  $NO_2$ , which brings about the higher resistance of the sensor in the presence of  $NO_2$  gas. However, both pristine  $SnO_2$  and  $MoS_2$  sensors revealed a low response at 100 °C due to limited sources of resistance modulation. All composite sensors exhibited a higher response to  $NO_2$  than the pristine sensors, which could be related to the presence of *n-n* heterojunctions in the composite sensors. Figure 7a shows side views of  $MoS_2$  NSs and the SM-20 composite on the substrate. In the composite sensor, it is expected that  $SnO_2$  NWs bridge among  $MoS_2$  NSs owing to the lower amount of  $SnO_2$  NWs relative to  $MoS_2$  NSs; hence, numerous *n-n* heterojunctions were created. Figure S7a presents the UPS spectra of the  $MoS_2$  NSs and  $SnO_2$  NWs. Based on energy-cut-off values and procedure reported in [68], the work functions of  $MoS_2$  and  $SnO_2$  were calculated to be 4.82 and 4.37 eV, respectively.



**Figure 7.** (a) Side views of  $MoS_2$  NSs and SM-20 composite on the substrate. (b) Schematic of  $NO_2$  gas sensing mechanism of SM-20 composite gas sensor. (c) Self-heating effect of SM-20 gas sensor.

Accordingly, we constructed their energy band levels, as shown in Figure S7b. Owing to the difference between the work functions of SnO<sub>2</sub> and MoS<sub>2</sub>, upon intimate contact, the electrons were moved from SnO<sub>2</sub> to MoS<sub>2</sub> to equate the Fermi levels on both sides of the contact. This led to band bending and the formation of *n-n* heterojunction barriers in the air. Furthermore, due to the flow of electrons to MoS<sub>2</sub>, which acts as the main sensing material, the thickness of the EDL on MoS<sub>2</sub> was smaller than that of the pristine MoS<sub>2</sub> sensor (Figure 7b). Accordingly, more electrons are available for extraction by NO<sub>2</sub> gas; hence, higher resistance modulation is expected. In addition, when the composite sensors were exposed to NO<sub>2</sub>, more electrons were abstracted from the sensor surface, and the height of the heterojunction barriers further increased, which eventually led to an increase in resistance in the presence of NO<sub>2</sub> gas, contributing to the sensing signal. Thus, the presence of numerous heterojunctions in composite gas sensors is beneficial for NO<sub>2</sub> gas sensing. Therefore, the SM-20 composite exhibited a higher response than the SM-10 composite sensor.

However, a further increase in SnO<sub>2</sub> content decreased the sensor response, which could be related to the agglomeration of SnO<sub>2</sub> NWs, a decrease in the number of *n*-MoS<sub>2</sub>/*n*-SnO<sub>2</sub> heterojunctions, and a simultaneous increase in the number of SnO<sub>2</sub>-SnO<sub>2</sub> homojunctions. Additionally, as the amount of SnO<sub>2</sub> is increased, the amount of MoS<sub>2</sub> NSs that are better sensing materials at 100 °C is simultaneously decreased. In other words, the contribution of SnO<sub>2</sub> NWs with interferer sensing response at 100 °C may be significant in the CM-30 nanocomposite sensor, resulting in a decrease in the overall performance. In addition to the formation of heterojunctions, the higher surface area of the composite gas sensors and the presence of voids between the SnO<sub>2</sub> NWs and MoS<sub>2</sub> NSs were beneficial for the diffusion and migration of NO<sub>2</sub> gas molecules. Owing to the combination of 1D SnO<sub>2</sub> NWs with 2D MoS<sub>2</sub> NSs, some voids were created among them, which acted as channels for the high diffusion of NO<sub>2</sub> gas into deeper parts of the sensor.

High selectivity to NO<sub>2</sub> gas can be related to (i) the high electron affinity of NO<sub>2</sub> gas (2.28 eV) compared to oxygen (0.43 eV), which can directly abstract electrons on the sensor surface, whereas other gases must react with adsorbed oxygen species to generate a sensing signal [69], (ii) the presence of N in NO<sub>2</sub> gas with an unpaired electron, which can bond with the sensor surface [70], and (iii) the relatively low bond energy of O–NO (305.0 kJ/mol) in NO<sub>2</sub>, which improves the response to NO<sub>2</sub> [71].

During the operation of the gas sensors in self-heating mode, electrons accelerate owing to the application of voltage, and on their pathways, they lose their high kinetic energies as heat after collision with other electrons, ions, and atoms in a process known as the Joule heating effect. Figure S7a manifests the induced temperatures of the MoS<sub>2</sub> NSs, SnO<sub>2</sub> NWs, and SM-20 composite gas sensor versus applied voltage. Among them, the temperature of the SM-20 composite sensor was higher at a fixed applied voltage, demonstrating the presence of more sources of heat generation inside the sensor owing to the contact areas between the MoS<sub>2</sub> NSs and SnO<sub>2</sub> NWs, which acted as powerful sources of Joule heating (Figure 7c). Figure S7b shows the induced temperature of the SM-20 composite as a function of applied voltage in the range of 1–3.5 V. Under 1, 1.5, 2, 2.5, 3, 3.1, 3.2, 3.3, 3.4, and 3.5 V applied voltage, the induced temperature values were 38, 55, 71, 88, 106, 110, 114, 118, 122, and 126 °C, respectively. Therefore, under the optimal applied voltage of 3.2 V, a sufficiently high temperature was induced inside the sensor, which was sufficient to activate the adsorption and reaction of NO<sub>2</sub> gas on the sensor surface. Under optimal sensing temperature and voltage, the power consumption ( $V^2/R$ ) of MoS<sub>2</sub> NSs (100 °C, 1 V), SnO<sub>2</sub> NWs (300 °C, 1 V), and SM-20 nanocomposite (RT, 3.2 V) sensors were calculated to be = 23.6, 0.2, and 1.3 μW, respectively. Despite the low power consumption of the SnO<sub>2</sub> NW gas sensor (0.2 μW), an increase in the temperature to 300 °C using external heating will result in significant power consumption, as an external heater is required to maintain this high temperature. For example, if an external heater uses 5 V to increase the sensor temperature to 300 °C, the power consumption will be 5.2 μW. Therefore, the SM-20 nanocomposite gas sensor showed the lowest power consumption in this study.

#### 4. Conclusions

Briefly, we introduced self-heated NO<sub>2</sub> gas sensors based on SnO<sub>2</sub> NWs/MoS<sub>2</sub> NSs composites. SnO<sub>2</sub> NWs were synthesized via a VLS mechanism, and then 10, 20, and 30 wt.% SnO<sub>2</sub> NWs were composited with MoS<sub>2</sub> NSs. Different characterization techniques, such as SEM/TEM and EDS, demonstrated the formation of nanocomposites with desired compositions. Also, some voids were presented among NWs and NSs, which were beneficial for efficient gas diffusion. Different voltages were applied on the sensor electrodes in self-heating mode, and the SM-20 composite, with 20 wt% SnO<sub>2</sub> NWs, showed the highest response of 13 to 0.1 ppm NO<sub>2</sub> gas at 3.2 V applied voltage. Furthermore, the optimal sensor revealed selectivity, long-term stability, reproducibility, and repeatability. The improved sensing performance was attributed to the generation of *n*-SnO<sub>2</sub>/*n*-MoS<sub>2</sub> heterojunctions, which acted as sources of resistance modulation, high surface area due to the NW and NS nature of SnO<sub>2</sub> and MoS<sub>2</sub> materials, respectively, along with the presence of voids in the SM-20 composite sensor. The present strategy, which combines the gas sensing properties of SnO<sub>2</sub> and MoS<sub>2</sub> with 1D and 2D morphologies, is a promising approach to boost the sensing features of the resultant gas sensor.

**Supplementary Materials:** The following supporting information can be downloaded at: <https://www.mdpi.com/article/10.3390/chemosensors12060107/s1>, Text S1: Calculation of limit of detection; Figure S1: (a,b) Digital images of fabricated sensor; Figure S2: SEM images of (a) SnO<sub>2</sub> NW, (b) MoS<sub>2</sub> NSs, and (c) SM-20 nanocomposite; Figure S3: (a) XPS survey of SM-20 composite. XPS core-level regions of (b) Mo 3d, (c) S 2p, (d) Sn 3d, and (e) O 1s; Figure S4: N<sub>2</sub> adsorption–desorption isotherms of (a) MoS<sub>2</sub> NSs, (b) SnO<sub>2</sub> NWs, (c) SM-10, (d) SM-20, and (e) SM-30 nanocomposite; Figure S5: Dynamic resistance and dynamic response plots of pristine MoS<sub>2</sub> NS gas sensor to 1 ppm NO<sub>2</sub> at (a) 25 °C and (b) different temperature (50–150 °C) under 1 V applied voltage. (c) Corresponding NO<sub>2</sub> gas response and baseline resistance versus operating temperature. Dynamic resistance plots of pristine SnO<sub>2</sub> NW gas sensors to 1 ppm NO<sub>2</sub> at (d) 25 °C and (e) different temperatures (50–350 °C) under 1 V applied voltage. (f) Corresponding NO<sub>2</sub> gas response and baseline resistance versus operating temperature; Figure S6. Reproducibility tests of three SM-20 gas sensors prepared under the same conditions. Sensing performance of SM-20 gas sensor (a) number 1, (b) number 2, and (c) number 3 (a) to low concentrations of various gases at fixed 3.2 V. (d) Corresponding selectivity histograms of three gas sensors; Figure S7: (a) UPS spectra and energy cut-off values of MoS<sub>2</sub> NSs and SnO<sub>2</sub> NWs. (b) Energy band levels of MoS<sub>2</sub> NSs and SnO<sub>2</sub> NWs before and after intimate contact; Figure S8: (a) Sensor temperature versus applied voltage for different gas sensors. (b) Temperature of SM-20 gas sensor versus applied voltage in the range of 1 to 3.5 V.

**Author Contributions:** Investigation, data curation, writing—original draft preparation, writing—review and editing, J.-Y.K.; writing—original draft preparation, writing—review and editing, supervision, A.M.; conceptualization, validation, formal analysis, investigation, writing—original draft preparation, writing—review and editing, visualization, supervision, funding acquisition, J.-H.K. All authors have read and agreed to the published version of the manuscript.

**Funding:** This work was supported by INHA UNIVERSITY Research Grant.

**Institutional Review Board Statement:** Not applicable.

**Informed Consent Statement:** Not applicable.

**Data Availability Statement:** Data will be made available upon request.

**Conflicts of Interest:** The authors declare no conflicts of interest.

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