

## Article

# High-Performance Sulfur Dioxide Gas Sensor Based on Graphite-Phase Carbon-Nitride-Functionalized Tin Diselenide Nanorods Composite

Hao Zhang<sup>1,2</sup>, Qiannan Pan<sup>3</sup>, Yating Zhang<sup>3</sup>, Yanting Zhang<sup>1</sup> and Dongzhi Zhang<sup>3,\*</sup> 

<sup>1</sup> College of Mechanical and Electrical Engineering, China University of Petroleum (East China), Qingdao 266580, China

<sup>2</sup> Technology Inspection Center, Shengli Oilfield, Dongying 257000, China

<sup>3</sup> College of Control Science and Engineering, China University of Petroleum (East China), Qingdao 266580, China

\* Correspondence: dzzhang@upc.edu.cn

**Abstract:** In this paper, a composite of tin diselenide (SnSe<sub>2</sub>) functionalized by graphite-phase carbon nitride (g-C<sub>3</sub>N<sub>4</sub>) was successfully prepared by a hydrothermal method, and was characterized by X-ray diffraction (XRD), scanning electron microscope (SEM) and X-ray photoelectron spectroscopy (XPS). These microstructure characterization results verified the successful synthesis of a multilayer g-C<sub>3</sub>N<sub>4</sub>/rod-shaped SnSe<sub>2</sub> composite. The gas sensitivity results showed that when the g-C<sub>3</sub>N<sub>4</sub> ratio was 30%, the g-C<sub>3</sub>N<sub>4</sub>/SnSe<sub>2</sub> composite sensor had the highest response (28.9%) at 200 °C to 20 ppm sulfur dioxide (SO<sub>2</sub>) gas, which was much higher than those of pristine g-C<sub>3</sub>N<sub>4</sub> and SnSe<sub>2</sub> sensors at the optimum temperature. A series of comparative experiments proved that the g-C<sub>3</sub>N<sub>4</sub>/SnSe<sub>2</sub> composite sensor demonstrated an excellent response, strong reversibility and good selectivity for ppm-level SO<sub>2</sub> gas detection. The possible SO<sub>2</sub> sensing mechanism was ascribed to the heterostructure between the n-type SnSe<sub>2</sub> and n-type g-C<sub>3</sub>N<sub>4</sub> nanomaterials. Furthermore, we also proposed the influence of the special structure of the g-C<sub>3</sub>N<sub>4</sub> functionalized SnSe<sub>2</sub> composite on the gas-sensing characteristics.

**Keywords:** tin diselenide; carbon nitride; hydrothermal; heterostructure; SO<sub>2</sub> sensors



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## 1. Introduction

As an important indicator of air pollution, sulfur dioxide (SO<sub>2</sub>) is an irritating, highly toxic and colorless gas, which mainly comes from factory exhaust and automobile exhaust emissions [1,2]. When the emitted concentration of sulfur dioxide in air is too high, it is oxidized to sulfur trioxide and combined with water to form acid rain, which can destroy buildings, pollute the environment and reduce soil fertility [3,4]. In addition, human health is seriously threatened by some harmful gas species including sulfur dioxide gas. Respiratory diseases such as bronchitis and asthma can result from the excessive inhalation of sulfur dioxide gas [5]. There are reports that the human-permissible exposure limit for SO<sub>2</sub> gas is 5 ppm and the long-term exposure limit is 2 ppm [6]. Therefore, the development of portable, efficient and reliable gas sensors can be used to monitor the composition and concentration of environmental gas pollutants in the atmosphere, which can help people to deal with dangerous gases in time, and further protect social safety and human health.

Recently, two-dimensional (2D) layered inorganic materials (such as graphene and transition metal materials) have attracted attention due to their unique crystal structures and characteristics. Among them, two-dimensional transition metal dichalcogenides (TMDs) are ideal materials for preparing field effect transistors [7], photodetectors [8] and electronic devices [9] because of their unique electronic, magnetic, optical and mechanical properties. Due to their planar crystal structure, high specific surface area and

physical affinity, they also have unique advantages in sensing applications [10]. According to the reports from a few studies, two-dimensional transition metal compounds such as molybdenum disulfide ( $\text{MoS}_2$ ) [11,12], tungsten disulfide ( $\text{WS}_2$ ) [13,14] and tin diselenide ( $\text{SnSe}_2$ ) [15,16] offer good gas sensing characteristics for detecting dangerous gases.  $\text{SnSe}_2$  is an n-type two-dimensional transition chalcogenide, and its gas-sensitive properties have been widely reported [15–17]. The results of a study reported by Moreira et al. on the ammonia ( $\text{NH}_3$ ) and nitrogen dioxide ( $\text{NO}_2$ ) gas sensitivity of tin-diselenide-based sensors revealed that these sensors had steady repeatability and long-term stability under UV radiation [17]. Zhang et al. fabricated coral-like tin diselenide/metal-organic frameworks (MOFs)-derived nanoflower-like tin dioxide ( $\text{SnO}_2$ ) heteronanostructures via a hydrothermal method. The  $\text{SnSe}_2/\text{SnO}_2$  nanocomposite sensor exhibited excellent  $\text{NO}_2$ -sensing performance at room temperature, which was significantly improved under UV illumination. The enhanced  $\text{NO}_2$  sensing performance was attributed to the formation of an n-n heterostructure and light-excited electrons [18]. By using the template-sacrificial approach, Wang et al. created rod-shaped  $\text{SnSe}_2$  and polyhedral zinc oxide ( $\text{ZnO}$ ) composite nanostructures, and the  $\text{ZnO}/\text{SnSe}_2$  heterostructures exhibited an enhancement of carbon monoxide ( $\text{CO}$ )-sensing properties at room temperature [19]. Pan et al. reported a coral-like Au-modified  $\text{SnSe}_2$  Schottky-junction-based ammonia gas sensor and demonstrated good gas sensitivity to ammonia gas detection. In addition, the effect of Au modification on ammonia gas molecules adsorption was also investigated using a first-principles density functional theory (DFT). Because of these studies, it can be concluded that  $\text{SnSe}_2$  is a feasible material as building block for constructing high-performance gas sensors [20]. However, the use of a single  $\text{SnSe}_2$  material for gas sensitivity research has certain limitations, and the test results may show poor selectivity and low sensitivity. According to previous studies, the sensing characteristics of gas sensors can be improved by forming a heterojunction [21,22]. Graphite-phase carbon nitride ( $\text{g-C}_3\text{N}_4$ ), as a common two-dimensional material, has a structure like graphene and provides more active sites for gas adsorption. The material has excellent chemical stability, good electron mobility and low cost. In addition,  $\text{g-C}_3\text{N}_4$  can also promote the uniform dispersion of active ingredients, so it can be used as a stable catalyst carrier [23,24].

In this paper, a composite of  $\text{SnSe}_2$  functionalized by  $\text{g-C}_3\text{N}_4$  was prepared via a hydrothermal method and served as the sensitive nanomaterial for  $\text{SO}_2$  gas sensing. The nanostructure of the  $\text{g-C}_3\text{N}_4/\text{SnSe}_2$  composite was characterized by X-ray diffraction (XRD), scanning electron microscope (SEM) and X-ray photoelectron spectroscopy (XPS). The characterization results verified the successful synthesis of a multilayer  $\text{g-C}_3\text{N}_4$ /rod-shaped  $\text{SnSe}_2$  composite. The as-prepared  $\text{g-C}_3\text{N}_4/\text{SnSe}_2$  composite sensor showed an excellent gas response and rapid adsorption/desorption ability towards  $\text{SO}_2$  under the optimal temperature of  $200\text{ }^\circ\text{C}$ , which was much higher than those of pristine  $\text{g-C}_3\text{N}_4$  and  $\text{SnSe}_2$  sensors. A series of comparative experiments proved that the  $\text{g-C}_3\text{N}_4/\text{SnSe}_2$  composite sensor demonstrated an excellent response, strong reversibility and good selectivity for ppm-level  $\text{SO}_2$  gas detection. In this paper, two innovative two-dimensional materials,  $\text{g-C}_3\text{N}_4$  and  $\text{SnSe}_2$ , were used to discuss in detail the mechanisms that may improve the  $\text{SO}_2$  sensing performance, such as the synergistic interaction between  $\text{g-C}_3\text{N}_4$  and  $\text{SnSe}_2$ , and the effective structural features.

## 2. Experimental Section

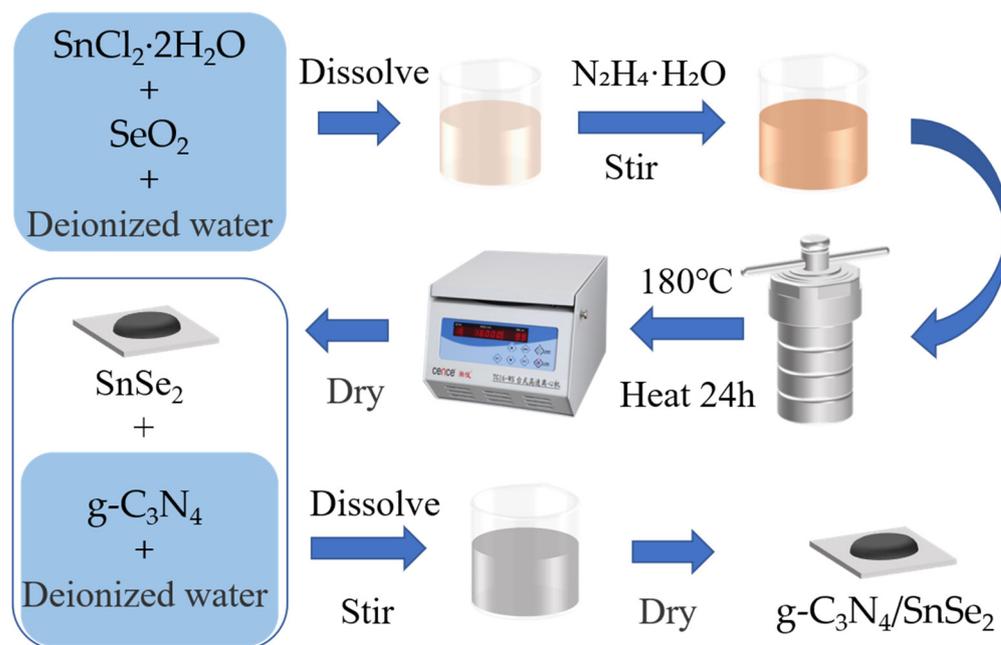
### 2.1. Materials

Tin chloride dihydrate ( $\text{SnCl}_2 \cdot 2\text{H}_2\text{O}$ ), hydrazine hydrate ( $\text{N}_2\text{H}_4 \cdot \text{H}_2\text{O}$ ), selenium dioxide ( $\text{SeO}_2$ ), ethanol ( $\text{CH}_3\text{CH}_2\text{OH}$ ) and graphitic carbon nitride ( $\text{g-C}_3\text{N}_4$ ) were all supplied by Sinopharm Chemical Reagent Co. Ltd. (Shanghai, China).

### 2.2. Material Synthesis and Sensor Fabrication

The preparation process of the materials is shown in Figure 1. The  $\text{SnSe}_2$  was prepared by a hydrothermal reduction method. A total of 0.01 mol  $\text{SnCl}_2 \cdot 2\text{H}_2\text{O}$  and 0.02 mol  $\text{SeO}_2$

were added into 70 mL deionized water and stirred for 30 min. A total of 10 mL of hydrazine hydrate was added into the above mixture solution, and then transferred to a 100 mL Teflon-lined stainless-steel autoclave, and hydrothermally treated at 180 °C for 24 h. The resulting precipitate was filtered, washed and dried at 60 °C for 8 h to obtain a black tin diselenide product.



**Figure 1.** Synthesis process of  $g\text{-C}_3\text{N}_4/\text{SnSe}_2$  nanomaterials.

A total of 280 mg of  $\text{SnSe}_2$  powder and 120 mg of  $g\text{-C}_3\text{N}_4$  powder were dissolved in 20 mL of DI water. After vigorously stirring for 1 h, the  $g\text{-C}_3\text{N}_4$  was effectively anchored on the surface of  $\text{SnSe}_2$ . The resulting product was dried at 60 °C for overnight. Finally, the  $g\text{-C}_3\text{N}_4/\text{SnSe}_2$  composite was obtained. The composite materials with different ratios (0, 20, 30, and 50%) of  $g\text{-C}_3\text{N}_4$  and  $\text{SnSe}_2$  were prepared by adjusting the quality ratio of  $g\text{-C}_3\text{N}_4$  and  $\text{SnSe}_2$ . In addition, the two materials were dispersed in deionized water in a certain proportion, and after a period of ultrasonic treatment, they were effectively combined through strong physical effects and interactions between charges.

### 2.3. Gas Sensor Fabrication

The structure illustration of the ceramic tube based  $\text{SO}_2$  gas sensor is shown in Figure 2. It consisted of an  $\text{Al}_2\text{O}_3$  ceramic tube and a base. The ceramic tube was 4 mm long and 1.2 mm in diameter, and its surface was equipped with gold electrodes and two pairs of platinum wires for electrical signals. The heating resistor of the Ni–Cr alloy coil passed through a hollow ceramic tube for heating. The sensing layer materials were coated on the surface of the ceramic tube, and the electrodes were led out to complete the preparation of the sensor. After preparing the sensing film, the sensor was dried at 60 °C for 6 h and then aged at 200 °C for 24 h before the test to obtain good resistance stability. The  $\text{SO}_2$ -sensing measurement was performed in a home-made gas sensing detection system [25] as shown in Figure 3. The sensor was placed in a home-made chamber, and the  $\text{SO}_2$  gas with different concentrations of 1–200 ppm was obtained by diluting 1000 ppm  $\text{SO}_2$  standard gas with high-purity air. The sensor resistance was measured with an Agilent 34970A digital multimeter and connected with a computer through RS-232 for data acquisition. The operation temperature of the sensor was controlled by an applied voltage to the Ni–Cr heating resistor. A steady power supply of GPD-4303S was employed for applying voltage for heating. The response of the sensor is defined as  $S = (R_A - R_G)/R_A \times 100\%$  ( $R_A$ : resistance in air;  $R_G$ : resistance in  $\text{SO}_2$  gas).

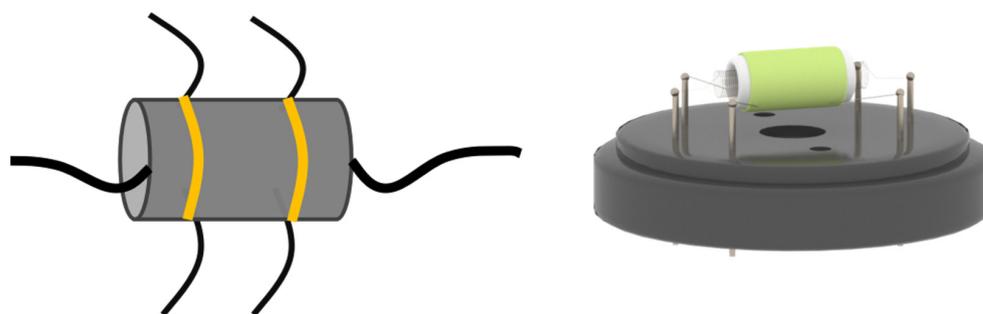


Figure 2. Structure illustration of the ceramic-tube-based SO<sub>2</sub> gas sensor.

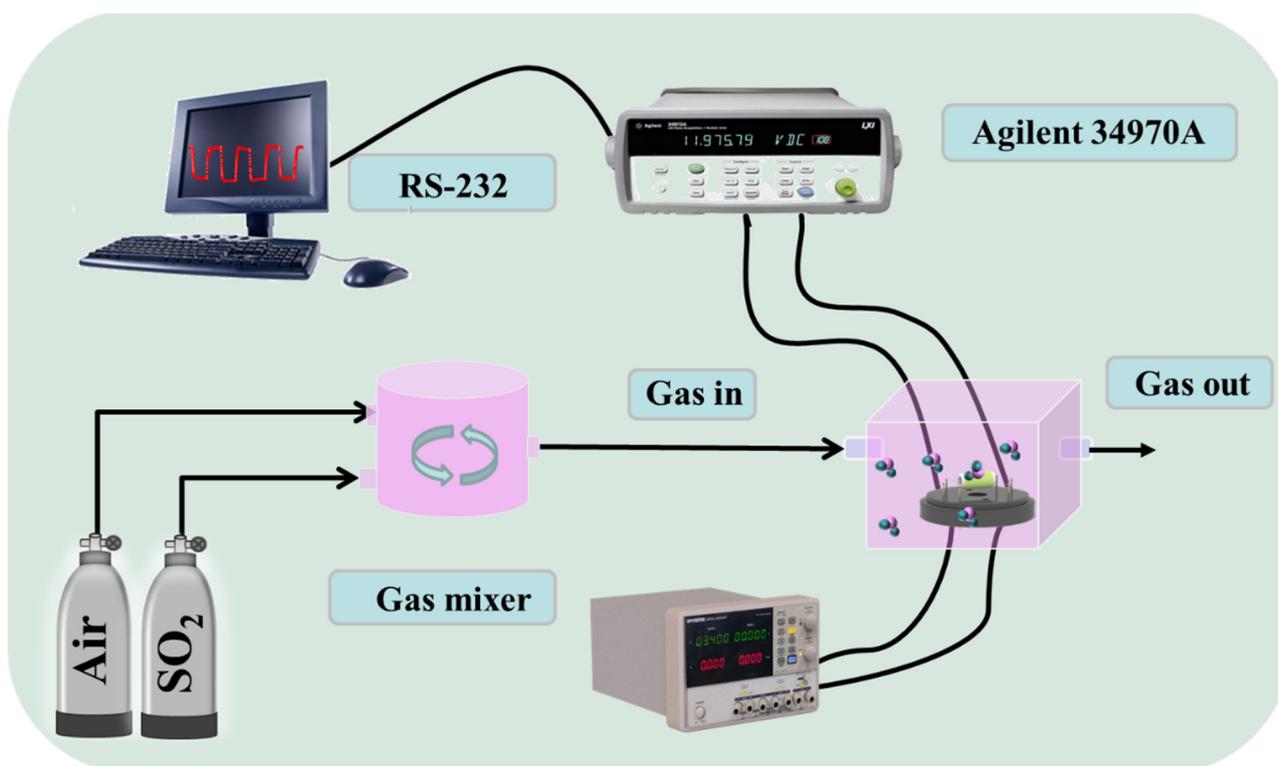


Figure 3. Schematic illustration of sensor performance test platform.

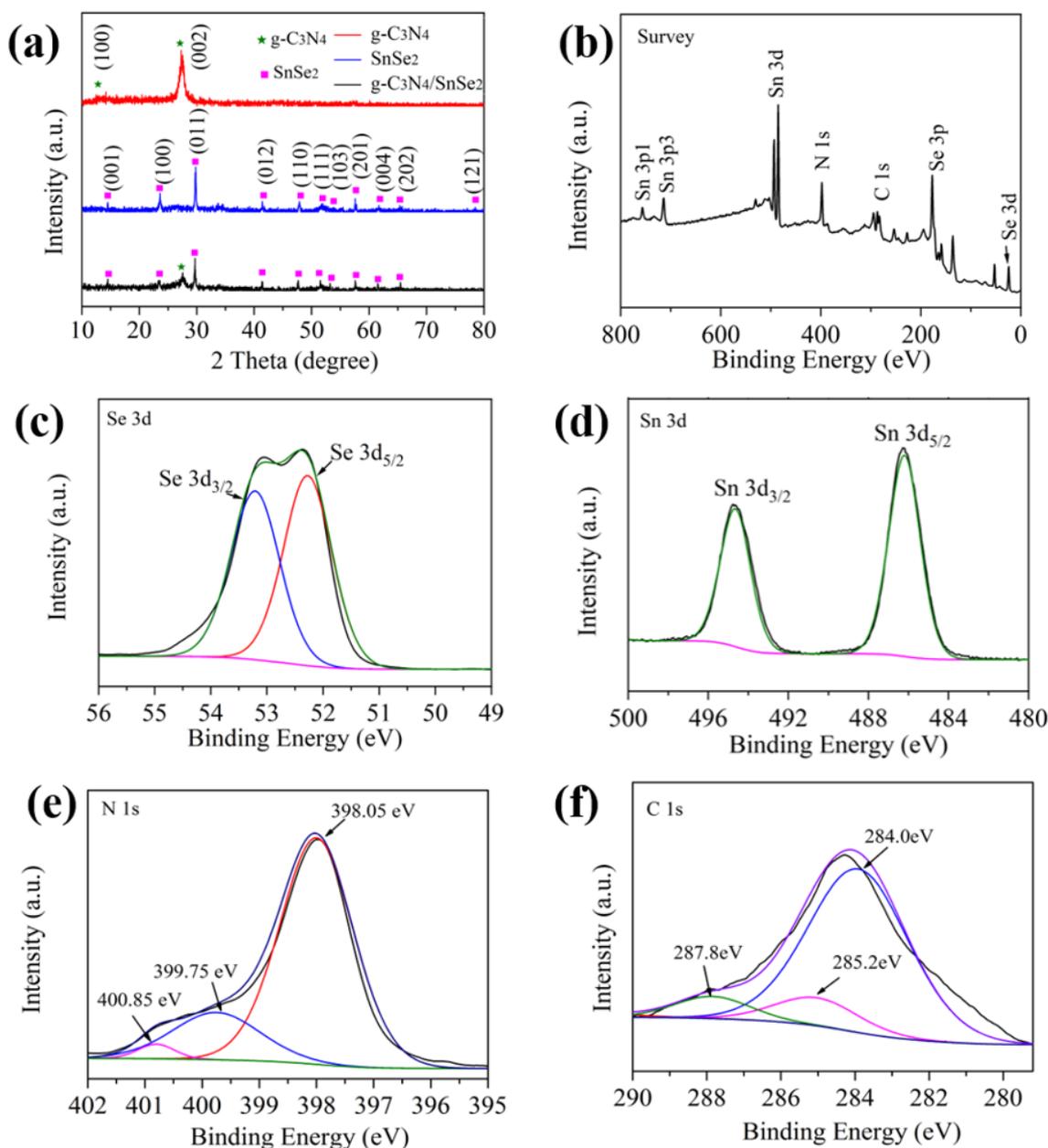
### 3. Results and Discussion

#### 3.1. Structure Characterization

The crystal structures of the SnSe<sub>2</sub>, g-C<sub>3</sub>N<sub>4</sub> and g-C<sub>3</sub>N<sub>4</sub>/SnSe<sub>2</sub> were characterized by X-ray powder diffraction (XRD, Rigaku D/Max-2550, Rigaku, Japan) with Cu K $\alpha$  radiation ( $\lambda = 0.15418$  nm). X-ray photoelectron spectroscopy (XPS, Thermo Scientific K-Alpha XPS spectrometer, Thermo Scientific, Waltham, MA, USA) was used to detect the chemical composition of the samples, and the morphology of pristine SnSe<sub>2</sub>, g-C<sub>3</sub>N<sub>4</sub> and g-C<sub>3</sub>N<sub>4</sub>/SnSe<sub>2</sub> nanocomposites were observed by a scanning electron microscope (SEM, Hitachi S-4800, Hitachi, Japan). The g-C<sub>3</sub>N<sub>4</sub>/SnSe<sub>2</sub> sample with a ratio of 30% g-C<sub>3</sub>N<sub>4</sub> was used for characterization.

The crystal phases of the as-prepared materials were identified by XRD analysis. As shown in Figure 4a, the peaks of SnSe<sub>2</sub> (JCPDS card number 23-0602) located at 14.38°, 26.98°, 30.88°, 40.17°, 47.77°, 50.19°, 52.58°, 57.92°, 60.34°, 63.96° and 78.22° corresponded to the (001), (100), (011), (012), (110), (111), (103), (201), (004), (202) and (121) diffraction planes of the orthorhombic-phase SnSe<sub>2</sub> structures, respectively [26]. The XRD pattern of g-C<sub>3</sub>N<sub>4</sub> was well in accordance with the hexagonal crystal g-C<sub>3</sub>N<sub>4</sub> (JCPDS Card no. 36-1451) [27]. The formation of the two diffraction peaks of (100) and (002) can be attributed

to the in-plane structure stacking of the tri-s-triazine part and the in-plane stacking of the conjugated aromatic hydrocarbon system, respectively. There were corresponding characteristic peaks of g-C<sub>3</sub>N<sub>4</sub> and SnSe<sub>2</sub> in the XRD pattern of the g-C<sub>3</sub>N<sub>4</sub>/SnSe<sub>2</sub> nanocomposite, and there were no other characteristic peaks, indicating the successful preparation of the SnSe<sub>2</sub>/g-C<sub>3</sub>N<sub>4</sub> composite.

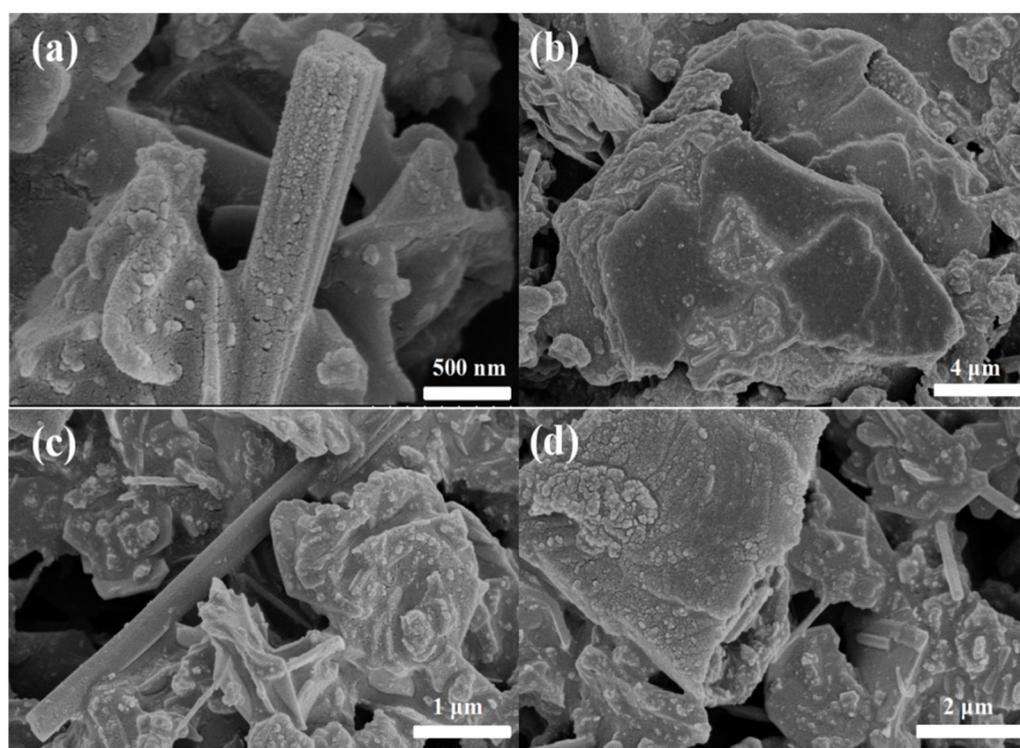


**Figure 4.** (a) XRD patterns of g-C<sub>3</sub>N<sub>4</sub>, SnSe<sub>2</sub> and g-C<sub>3</sub>N<sub>4</sub>/SnSe<sub>2</sub>. XPS spectra of g-C<sub>3</sub>N<sub>4</sub>/SnSe<sub>2</sub> product: (b) full spectrum, (c) Se 3d, (d) Sn 3d, (e) N 1s and (f) C 1s.

XPS is a measurement technique for detecting the elemental composition and chemical valence of materials. Figure 4b is the total spectrum of the g-C<sub>3</sub>N<sub>4</sub>/SnSe<sub>2</sub> composite, and the existence of the four elements Se, Sn, C and N was confirmed from the peak positions in the figure. The Se 3D spectrum shown in Figure 4c is represented by two peaks with binding energies of 53.4 and 52.2 eV, corresponding to Se 3d<sub>5/2</sub> and Se 3d<sub>3/2</sub>, respectively [28–30]. The Sn 3d spectrum shown in Figure 4d shows two distinctive peaks at energies of 486.3 and 495.2 eV, which correspond to Sn 3d<sub>5/2</sub> and 3d<sub>3/2</sub>, respectively.

Since there was an energy difference of 8.4 eV between the two peaks, the  $\text{Sn}^{4+}$  state can be confirmed [31]. Figure 4e is the characteristic peak of N 1s. Three asymmetric peaks at 398.3, 400.0 and 401.0 eV were attributed to C=N-C, N-(C)<sub>3</sub> and C-N-H, respectively. Figure 4f shows the XPS spectrum of C 1s, where the characteristic peak at 284.6 eV was due to the formation of indefinite carbon absorption on the surface of the material, and the diffraction peak at 287.8 eV may be formed by N=C-N coordination [32].

SEM is a detection technology used to observe the micro-morphology of nanomaterials. Figure 5 shows the SEM characterization of the g-C<sub>3</sub>N<sub>4</sub>/SnSe<sub>2</sub>-sensitive material. Figure 5a shows the SEM characterization of the intrinsic SnSe<sub>2</sub>. The SnSe<sub>2</sub> nanorods were composed of numerous nanoparticles, the cross-sectional radius and the length of which were about 300–500 nm and 3–5  $\mu\text{m}$ , respectively. Figure 5b shows the SEM characterization image of the graphite-like-phase carbon nitride. The exfoliated g-C<sub>3</sub>N<sub>4</sub> nanosheets had a 2D layered morphology, which was manifested as multiple thin layers stacking together. Figure 5c,d show the surface morphology of the g-C<sub>3</sub>N<sub>4</sub>/SnSe<sub>2</sub> composite. It can be clearly observed that the nanorod-shaped SnSe<sub>2</sub> and multilayer g-C<sub>3</sub>N<sub>4</sub> grew and aggregated together well, which shows that the g-C<sub>3</sub>N<sub>4</sub>/SnSe<sub>2</sub> composite was successfully synthesized.

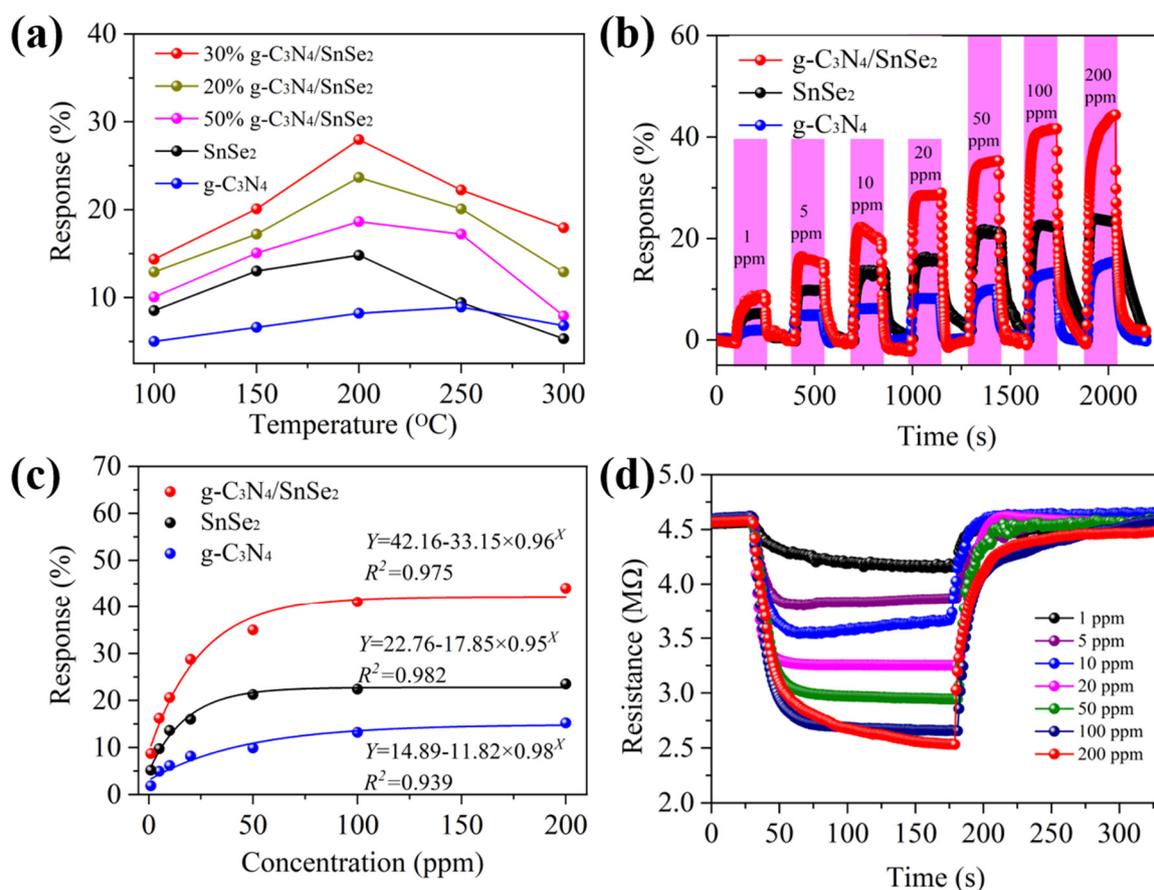


**Figure 5.** SEM images of (a) SnSe<sub>2</sub>, (b) g-C<sub>3</sub>N<sub>4</sub>, and (c,d) g-C<sub>3</sub>N<sub>4</sub>/SnSe<sub>2</sub>.

### 3.2. SO<sub>2</sub>-Sensing Properties

The working temperature is an important key variable related to the sensing characteristics of the g-C<sub>3</sub>N<sub>4</sub>/SnSe<sub>2</sub> sensor [33–35]. It affects the chemical adsorption and surface reaction of gas molecules. Therefore, to determine the optimal operating temperature as well as the optimum ratio of the two materials, we studied the responses of pristine g-C<sub>3</sub>N<sub>4</sub>, pristine SnSe<sub>2</sub> and the x% g-C<sub>3</sub>N<sub>4</sub>/SnSe<sub>2</sub> (x = 0, 20, 30, 50) film sensors to 20 ppm SO<sub>2</sub> gas at different operating temperatures. As shown in Figure 6a, the operating temperatures and responses of the sensors presented a “triangular” shape. The optimal temperatures of g-C<sub>3</sub>N<sub>4</sub>, SnSe<sub>2</sub> and g-C<sub>3</sub>N<sub>4</sub>/SnSe<sub>2</sub> were 250 °C, 200 °C and 200 °C, respectively. When the g-C<sub>3</sub>N<sub>4</sub> ratio was 30%, the response (28.9%) of g-C<sub>3</sub>N<sub>4</sub>/SnSe<sub>2</sub> at 200 °C was the highest, which was much higher than those of the pristine g-C<sub>3</sub>N<sub>4</sub> and SnSe<sub>2</sub>. The gas-sensing properties of the SnSe<sub>2</sub>, g-C<sub>3</sub>N<sub>4</sub> and 30% g-C<sub>3</sub>N<sub>4</sub>/SnSe<sub>2</sub> sensors

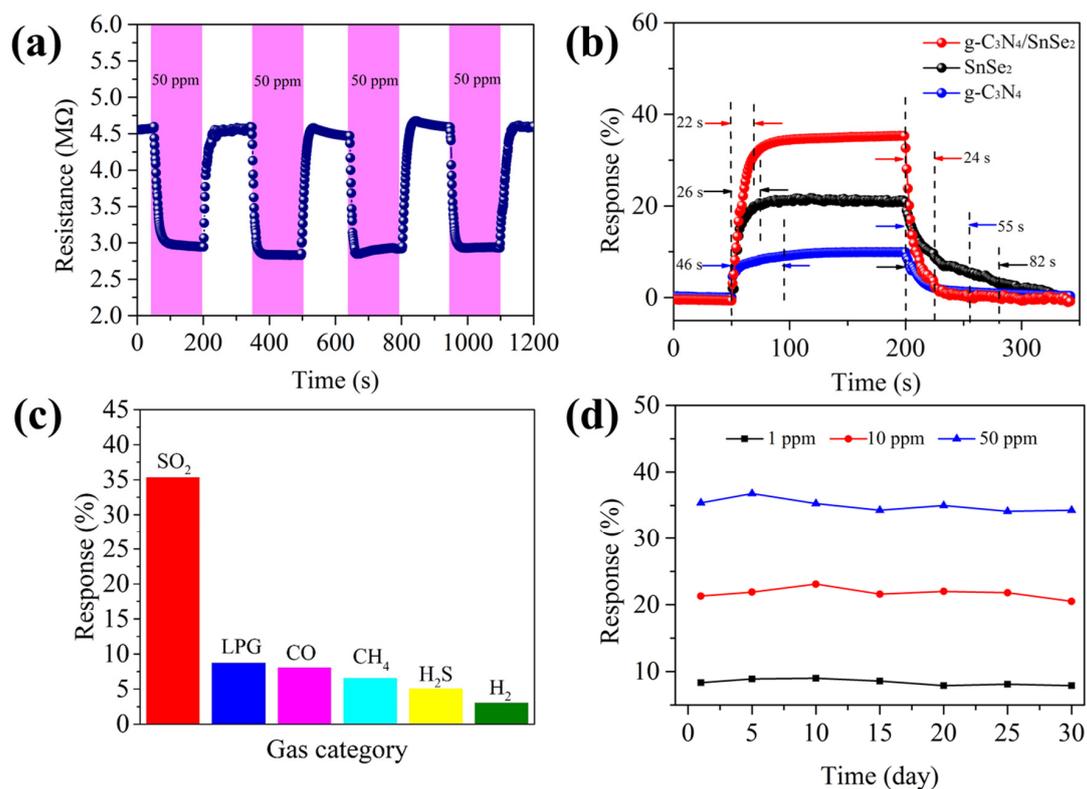
were investigated by recording the changes in resistance when they were exposed to different concentrations (1–200 ppm) of SO<sub>2</sub> gas at 200 °C, as shown in Figure 6b. When switched to air, the resistances of the three sensors returned to the base value in air. The response values of 30% g-C<sub>3</sub>N<sub>4</sub>/SnSe<sub>2</sub> towards 1, 5, 10, 20, 50, 100, 200 ppm SO<sub>2</sub> gas were, respectively, 8.82%, 15.59%, 20.22%, 28.52%, 35.09%, 41.54% and 44.34%, higher than those of the pristine SnSe<sub>2</sub> and g-C<sub>3</sub>N<sub>4</sub>. Figure 6c shows the fitting curves of the three sensors between sensitivity (Y) and concentration of SO<sub>2</sub> (X). The fitting functions of g-C<sub>3</sub>N<sub>4</sub>, SnSe<sub>2</sub> and g-C<sub>3</sub>N<sub>4</sub>/SnSe<sub>2</sub> sensors were  $Y = 14.89 - 11.82 \times 0.98^X$ ,  $Y = 22.76 - 17.85 \times 0.95^X$  and  $Y = 42.16 - 33.15 \times 0.96^X$ , respectively. The R<sup>2</sup> of the fitting curve of the g-C<sub>3</sub>N<sub>4</sub>/SnSe<sub>2</sub> sensor was 0.975. Figure 6d shows the response/recovery curves of the g-C<sub>3</sub>N<sub>4</sub>/SnSe<sub>2</sub> sensor toward the desired concentrations of SO<sub>2</sub> gas. The sensor exhibited stable response and recovery behaviors.



**Figure 6.** (a) Responses of g-C<sub>3</sub>N<sub>4</sub>/SnSe<sub>2</sub> composites with different quality ratios to 20 ppm SO<sub>2</sub> gas at various temperatures. (b) Sensing properties of pristine g-C<sub>3</sub>N<sub>4</sub>, SnSe<sub>2</sub> and g-C<sub>3</sub>N<sub>4</sub>/SnSe<sub>2</sub> composite sensors at different concentrations of SO<sub>2</sub> gas at 200 °C. (c) Response fitting curves of three sensors versus SO<sub>2</sub> concentration at 200 °C. (d) Typical response–recovery curves for various concentrations of SO<sub>2</sub> gas at 200 °C.

The repeatability of the g-C<sub>3</sub>N<sub>4</sub>/SnSe<sub>2</sub> composite sensor against a gas with a concentration of 50 ppm SO<sub>2</sub> at 200 °C is examined in Figure 7a. For each run, the resistance could fully recover its initial state and changes from 4.5 MΩ to 2.9 MΩ, showing a good reproducibility. Figure 7b shows the response/recovery curves of the g-C<sub>3</sub>N<sub>4</sub>, SnSe<sub>2</sub>, and g-C<sub>3</sub>N<sub>4</sub>/SnSe<sub>2</sub> composite sensors exposed to 50 ppm SO<sub>2</sub> gas. The response/recovery time of the g-C<sub>3</sub>N<sub>4</sub>/SnSe<sub>2</sub> sensor was 22/24 s, while the values of the pristine g-C<sub>3</sub>N<sub>4</sub> and SnSe<sub>2</sub> sensors were 46/55 s and 26/82 s, respectively, suggesting that the g-C<sub>3</sub>N<sub>4</sub>/SnSe<sub>2</sub> sensor had a faster detection rate towards SO<sub>2</sub> gas compared with the pristine g-C<sub>3</sub>N<sub>4</sub> and SnSe<sub>2</sub> sensors at 200 °C. Excellent selectivity is also an important factor for nanomaterial-based

gas sensors [36,37]. Therefore, we further studied the SO<sub>2</sub> selectivity of the g-C<sub>3</sub>N<sub>4</sub>/SnSe<sub>2</sub> composite sensor at 200 °C. The sensor was exposed to 50 ppm of various interfering gases, such as LPG, CO, CH<sub>4</sub>, H<sub>2</sub>S and H<sub>2</sub>. As shown in Figure 7c, the g-C<sub>3</sub>N<sub>4</sub>/SnSe<sub>2</sub> composite sensor had the highest response to SO<sub>2</sub> gas, indicating that the selectivity to SO<sub>2</sub> gas was excellent. Additionally, the long-term stability of the g-C<sub>3</sub>N<sub>4</sub>/SnSe<sub>2</sub> composite sensor for various SO<sub>2</sub> gas concentrations (1, 10 and 50 ppm) at a working temperature of 200 °C was examined. The sensor's response had no obvious changes in a period under the same experimental conditions and exhibited a good stability, as shown in Figure 7d.



**Figure 7.** At the optimal temperature of 200 °C (a) repeatability of g-C<sub>3</sub>N<sub>4</sub>/SnSe<sub>2</sub> composite sensor. (b) Response/recovery curves of g-C<sub>3</sub>N<sub>4</sub>, SnSe<sub>2</sub> and g-C<sub>3</sub>N<sub>4</sub>/SnSe<sub>2</sub> composite sensors exposed to 50 ppm SO<sub>2</sub> gas. (c) Selectivity and (d) long-term stability of g-C<sub>3</sub>N<sub>4</sub>/SnSe<sub>2</sub> composite sensor.

Numerous studies have described the detection of SO<sub>2</sub> gases utilizing a variety of sensitive materials up to this point. As far as we know, there have been no studies using g-C<sub>3</sub>N<sub>4</sub>/SnSe<sub>2</sub> for SO<sub>2</sub> gas detection. Table 1 lists the comparison of this work with previously reported works. The different SO<sub>2</sub> gas sensors are compared in terms of sensing environment, response value and gas concentration. The comparison results showed that the as-prepared g-C<sub>3</sub>N<sub>4</sub>/SnSe<sub>2</sub> sensor featured a higher response value and a lower operating temperature.

**Table 1.** Comparison of the SO<sub>2</sub>-sensing performance between the present work and previous reported studies.

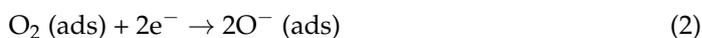
Sensing Material	Sensing Environment	Response	Concentration	Ref.
AlGaIn/ZnO/rGO	RT	2.5%	120 ppb	[38]
SnO <sub>2</sub> /rGO	RT/UV	1.7%	5 ppm	[39]
Pt/rGO	120 °C	5%	100 ppm	[40]
g-C <sub>3</sub> N <sub>4</sub> /rGO	RT	3.2%	100 ppm	[41]
SnO <sub>2</sub> /MWCNT	60 °C	6	500 ppm	[42]

Table 1. Cont.

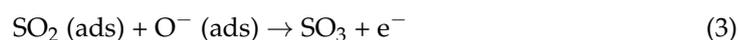
Sensing Material	Sensing Environment	Response	Concentration	Ref.
V <sub>2</sub> O <sub>5</sub> /WO <sub>3</sub> /TiO <sub>2</sub>	400 °C	5%	20 ppm	[43]
WO <sub>3</sub>	350 °C	5%	1 ppm	[44]
Cu-SnO <sub>2</sub>	400 °C	1.1%	20 ppm	[45]
SnO <sub>2</sub> -PANI	RT	3.1%	4 ppm	[46]
TiO <sub>2</sub> /rGO	RT	11.14%	5 ppm	[47]
NiO-SnO <sub>2</sub>	180 °C	8.3%	50 ppm	[48]
TiO <sub>2</sub>	200 °C	11%	10 ppm	[49]
ZnO	RT	0.2%	100 ppm	[50]
WO <sub>3</sub> -PANI	RT	4.3%	5 ppm	[51]
Ni-MoS <sub>2</sub>	RT	7.4%	5 ppm	[52]
V <sub>2</sub> O <sub>5</sub> /SnO <sub>2</sub>	350 °C	45%	5 ppm	[53]
V-doped TiO <sub>2</sub>	400 °C	10%	10 ppm	[54]
GO	RT	6%	5 ppm	[55]
PANI	RT	4.2%	10 ppm	[56]
g-C <sub>3</sub> N <sub>4</sub> /SnSe <sub>2</sub>	200 °C	28.9%	20 ppm	This work

### 3.3. SO<sub>2</sub> Gas-Sensing Mechanism

The sensing mechanism has been widely explained using the surface charge caused by adsorbed oxygen. For the pristine SnSe<sub>2</sub> gas sensor, when exposed to air, oxygen molecules in air adsorb on the surface of the sensing material to form adsorbed oxygen molecules. The adsorbed oxygen molecules extract electrons from the conduction band of SnSe<sub>2</sub> to form chemically adsorbed oxygen O<sup>-</sup> at 200 °C. On the surface of the g-C<sub>3</sub>N<sub>4</sub>/SnSe<sub>2</sub> sensing material, an electron depletion layer consequently forms. The following is a description of the reactions [57,58]:

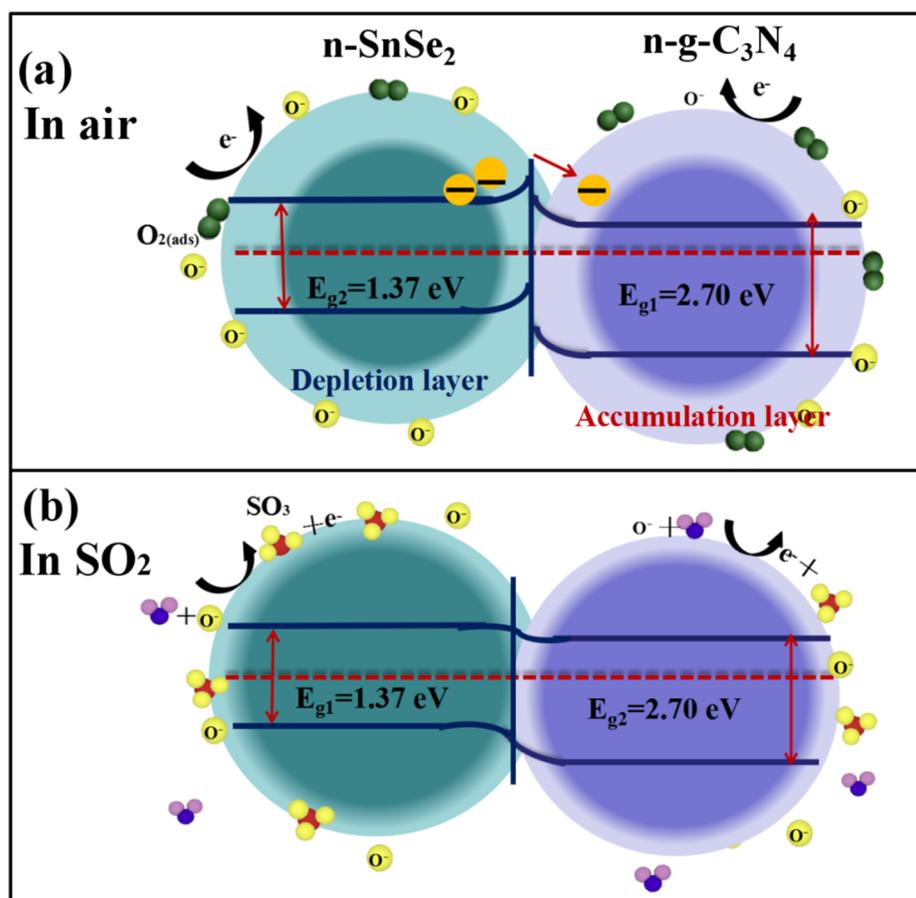


When the reducing gas SO<sub>2</sub> is introduced, the SO<sub>2</sub> molecules adsorbed on the surface of the sensing material will further react with O<sup>-</sup> ions and release electrons to the SnSe<sub>2</sub> conduction band to form SO<sub>3</sub>, thereby increasing the number of charge carriers and reducing the resistance of the sensor. The specific reaction process is illustrated as follows [59]:



In our experiment, it is worth noting that the g-C<sub>3</sub>N<sub>4</sub>/SnSe<sub>2</sub> sensor exhibited an improved response to SO<sub>2</sub> gas than the pristine C<sub>3</sub>N<sub>4</sub> and SnSe<sub>2</sub> sensors. This phenomenon could be explained by two positive factors of g-C<sub>3</sub>N<sub>4</sub> decoration. Firstly, the morphology, specific surface area and electrical properties of the material are important factors affecting its gas-sensing performance [60]. The addition of the layered two-dimensional structure of g-C<sub>3</sub>N<sub>4</sub> increases the specific surface area of the composite material and provides more active sites, increasing the production of adsorbed oxygen. Another factor influencing the effectiveness of the gas sensing system is the n-n heterojunction created between g-C<sub>3</sub>N<sub>4</sub> and SnSe<sub>2</sub> [61–63]. Figure 8a shows the energy band structure of the heterojunction formed by the n-type SnSe<sub>2</sub> nanorods and n-type g-C<sub>3</sub>N<sub>4</sub> layered nanosheets in air, where the band gaps of SnSe<sub>2</sub> and g-C<sub>3</sub>N<sub>4</sub> were 1.37 and 2.7 eV, respectively. Their Fermi levels were different, and the work function (4.3 eV) of SnSe<sub>2</sub> was lower than that (4.67 eV) of g-C<sub>3</sub>N<sub>4</sub> [64]. When the two materials contact with different work functions, electrons will transfer from SnSe<sub>2</sub> to g-C<sub>3</sub>N<sub>4</sub> to reach a Fermi energy balance. Therefore, the n-n heterojunction is formed at the interface between SnSe<sub>2</sub> and g-C<sub>3</sub>N<sub>4</sub> in air [65]. When exposed to SO<sub>2</sub> gas, SO<sub>2</sub> molecules absorbed on the surface of the composite material react with O<sup>-</sup> to generate electrons [66]. Therefore, the carrier concentration in the heterojunction increases by receiving electrons, resulting in a narrowing of the depletion layer at the interface of the two materials [61,67,68], which reduces the resistance of the sensor (Figure 8b). The built-

in electric field generated by the nano-scale heterojunction can accelerate the separation process of electrons and facilitate electron transfer.



**Figure 8.** Schematic diagram of microscopic sensing mechanism of  $g\text{-C}_3\text{N}_4/\text{SnSe}_2$  composite sensor.

#### 4. Conclusions

In this paper, a  $g\text{-C}_3\text{N}_4$  functionalized  $\text{SnSe}_2$  composite thin film sensor was successfully prepared. XRD, XPS and SEM techniques were used to characterize the elements and structure of the  $g\text{-C}_3\text{N}_4/\text{SnSe}_2$  composite material. The optimal temperature ( $200\text{ }^\circ\text{C}$ ) of the  $g\text{-C}_3\text{N}_4/\text{SnSe}_2$  composite sensor was determined through gas-sensing experiments under different working temperatures. The  $\text{SO}_2$  gas-sensing performance for the  $g\text{-C}_3\text{N}_4/\text{SnSe}_2$  composite sensor was investigated at the optimal temperature. The experimental results showed that under the optimal temperature, the 30%  $g\text{-C}_3\text{N}_4/\text{SnSe}_2$  composite sensor gained the highest sensitivity to  $\text{SO}_2$  gas. The two-dimensional layered structure of the  $g\text{-C}_3\text{N}_4$ -modified n-type  $\text{SnSe}_2$  nanorods not only increased the specific surface area and the gas adsorption site of the  $g\text{-C}_3\text{N}_4/\text{SnSe}_2$  sensor, but also formed an n-n heterojunction between the  $g\text{-C}_3\text{N}_4$  nanosheets and  $\text{SnSe}_2$  nanorods, which improved the sensing performance of the  $g\text{-C}_3\text{N}_4/\text{SnSe}_2$  sensor toward  $\text{SO}_2$  gas.

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