**Article**

**Indium Oxide Decorated WS$_2$ Microflakes for Selective Ammonia Sensors at Room Temperature**

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**Abstract:** Tungsten sulfide decorated with indium oxide nanoparticles (In$_2$O$_3$/WS$_2$) was studied for a chemiresistive-type NH$_3$ sensor at room temperature. It was found that the responses of the developed In$_2$O$_3$/WS$_2$ heterostructure nanocomposite-based sensors are significantly improved to 3.81 from 1.45 for WS$_2$. The response and recovery time of the heterostructure-based sensor was found to significantly decrease to 88 s/116 s (10 ppm) from 112 s/192 s for the WS$_2$-based one. The sensor also exhibits excellent selectivity and signal reproducibility. In comparison to WS$_2$ decorated with both ZnO and SnO$_2$, in similar ways, the In$_2$O$_3$-decorated WS$_2$ has overall better sensing performance in terms of sensitivity, selectivity and response/recovery speeds for NH$_3$ from 1 ppm to 10 ppm at room temperature. The improved sensing properties of WS$_2$ incorporating In$_2$O$_3$ could be attributed to the joint enhancement mechanisms of the “electronic and catalytic” sensitizations.

**Keywords:** WS$_2$; WS$_2$-In$_2$O$_3$; heterostructure nanocomposites; ammonia; gas sensor

1. **Introduction**

Ammonia is a natural, colorless and corrosive gas that is potentially toxic to human health and the environment [1]. It comes from the decomposition of feces and dead animals and plants and exists in the atmosphere at low concentrations [2]. Ammonia is mainly used as a fertilizer to increase crop yields. It is also widely used in refrigeration, fuel, textile production and other industrial fields [3]. As a carbon-free hydrogen carrier, ammonia contains 17.6 wt.% hydrogen. It can be used as a green fuel for internal combustion engines and gas turbines which play an indispensable role in addressing the chemical fuel depletion crisis [4,5]. However, a high level of ammonia exposure can cause damage to human health; therefore, the upper exposure concentration of ammonia within 8 h is limited to 25 ppm [6]. Excessive ammonia emissions can subsequently lead to environmental acidification which needs to be monitored in real time [7,8].

To fulfill the demand for monitoring ammonia, sensors working at room temperature with low cost, low power consumption, high sensitivity, fast response, and good selectivity need to be developed [9]. Metal oxides such as SnO$_2$, ZnO, TiO$_2$, WO$_3$, and In$_2$O$_3$ are widely explored as the sensing materials for detecting ammonia. However, they are usually operated at high temperatures between 100 °C and 550 °C, which requires high power consumption [10–16]. To fabricate low-temperature chemiresistive-type gas sensors, graphene with a two-dimensional (2D) network of carbon has become a promising candidate material due to its advantages of high specific surface area and excellent electrical conductivity [17]. However, its application is limited by the zero band gap feature, and graphene is not very responsive to ammonia at room temperature. Therefore, graphene was usually functionalized with metal nanoparticles, metal oxides, organic molecules and conductive polymers to enhance its functionality [18].
As analogs to graphene, metal dichalcogenides such as MoS₂, SnS₂ and WSe₂ have also become potential candidate materials for fabricating nanoelectronic devices [19]. Singh et al. synthesized a p-MnS₂/n-WO₃ heterostructure for a chemical ammonia sensor and found it displayed an improved response to ammonia at 200 °C compared to the pure p-MnS₂-based one [20]. Xia et al. reported a WS₂/SnO₂-based sensor with 2D/0D heterostructure and noticed the response to NO₂ could be further improved under UV at room temperature [21]. Wang et al. found that the (2D/2D) rGO/WS₂ heterostructure-based ammonia sensor showed improved response/recovery at room temperature. The reasons are attributed to the extra active sites introduced by the secondary 2D phase [22]. Kim et al. fabricated a Au-decorated WS₂-SnO₂ core–shell nanosheet-based gas sensor and demonstrated that it has enhanced sensing properties for CO at 3.4 V [23].

In previous studies, WS₂ has been reported to show promise in detecting harmful gases at room temperature [24]. Metal oxides such as SnO₂ and ZnO and noble metals such as Pt and Au have been strategically used to improve the sensing properties in terms of sensitivity, selectivity and response/recovery speeds. In₂O₃ is also a good semiconducting material for gas sensing because it has a lower resistance and a good response to HCHO [25], NO₂ [26] and CO [27]. Therefore, in this work, the 2D WS₂ heterostructures by indium oxide nanoparticles was examined. Indium oxide was selected as the secondary phase to improve the sensing properties of a WS₂-based chemiresistive-type sensor at room temperature for the following two reasons: We found that the synthesized sensors all displayed higher responses and faster recovery to ammonia than the pure WS₂ micro-flake-based one at room temperature. Comparing the response rates of the developed sensors to the pure sensor, In₂O₃/WS₂ heterostructure nanocomposite-based sensors exhibited a much faster response, The improved performance of the In₂O₃/WS₂-based sensors could be mainly attributed to the electronic sensitization between In₂O₃ and WS₂. Sensors based on WS₂: heterostructured with ZnO/SnO₂ were also characterized and compared.

2. Experimental Method

2.1. Synthesis of In₂O₃/WS₂ Nanocomposites

The WS₂ powders used in this study were purchased from Aladdin Co., Shanghai, China. In₂O₃ powders were purchased from Macklin Biochemical Co., Ltd., Shanghai, China. ZnO and SnO₂ powders were obtained from Aladdin Co., Shanghai, China. All chemicals were of analytical grade and used as received. First, 50.0 mg WS₂ powders and 55.5 mg In₂O₃ powders were added to 15 mL and 20 mL deionized (DI) water, respectively, followed by the sonication in an ultrasound water bath for 15 min. Different amounts of In₂O₃ solutions (0.1 mL, 0.2 mL, 0.6 mL and 1 mL) were added drop-wise to the WS₂ dispersion solution with vigorous stirring for half an hour to obtain the WS₂ suspensions with different concentrations of In₂O₃ (0.5%, 1%, 3% and 5%). WS₂ suspensions with different ZnO contents and SnO₂ contents (0.5%, 1%, 3% and 5%) for comparisons were obtained by the same method. Then the obtained mixtures were washed with absolute alcohol and DI water three times in sequence and dried at 60 °C overnight in a drying oven.

2.2. Characterization

X-ray diffraction (XRD, D8 Advance, Bruker Corporation, Billerica, MA, USA) was used to investigate the chemical information and crystal structure of indium oxide decorated tungsten sulfide (IO/WS₂) heterostructure nanocomposite-based samples. A field emission scanning electron microscope (FESEM, Gemini 300, ZEISS Corporation, Jena, Germany) and a transmission electron microscope (TEM, Tecnai G2 F20, FEI Corporation, Hillsboro, OR, USA) were used to study the surface morphologies of the prepared IO/WS₂ samples. X-ray photoelectron spectroscopy (XPS, ESCALAB 250Xi, Thermo Fisher Scientific Corporation, Waltham, MA, USA) was performed to explore the elemental compositions and chemical oxidation states of the IO/WS₂ samples.
2.3. Fabrication and Measurement of the Gas Sensors

A printable paste was formed by grinding a small amount of the obtained IO/WS₂ materials and DW in an agate mortar. Then the paste was spread on the gold interdigital electrodes pre-printed on the ceramic substrate, which were dried at 60 °C for 12 h in a drying oven to develop the IO/WS₂-based chemiresistive-type sensor. To measure the sensing properties of the fabricated sensor, the sensor was placed in a testing chamber with a volume of 50 L, and the resistance of the sensor was recorded using a digital multimeter (Keysight 34465A). A corresponding amount of liquid solutions including ammonia, formaldehyde, toluene, methanol, ethanol and acetone, which were purchased from Kermel Co., Tianjin, China, was vaporized to achieve different gas vapors by heating after being injected into the small crucible at the corner of the testing chamber. The gas vapor concentrations were calculated by the following formula [28]:

$$C = \frac{22.4 \times q \times d \times V_1}{(M \times V_2)}$$  \hspace{1cm} (1)

where C (ppm) represents the targeted gas concentration, q (g/mL) represents the liquid density, d represents the liquid purity, M (g/mol) represents the liquid molecular weight, V₁ (L) represents the liquid volume and V₂ (L) represents the volume of the testing chamber (50 L). The sensor response (S) was defined by the resistance ratio of $R_g/R_a$, where $R_a$ is the resistance of the sensor in target ammonia and $R_g$ is the resistance of the sensor in air. The response time and recovery time were measured by the time course of 90% of the maximum response amplitude.

3. Results and Discussion

3.1. Microstructure Characterizations

Figure 1 shows the XRD patterns of the pure WS₂ and IO/WS₂ with 1% In₂O₃ (IO/WS₂-1) heterostructure nanocomposite-based samples. The main peaks in the XRD pattern of WS₂ (red) correspond to 2H-WS₂ (PDF # 87-2417). The peaks in the XRD pattern of the IO/WS₂-1 heterostructure nanocomposite-based sample contain the diffraction peaks of both WS₂ and In₂O₃ (PDF # 71-2194) without other impurities. The size of In₂O₃ particles can be calculated by the Scherrer equation as follows [29]:

$$D = K\lambda/(\beta \cos\theta)$$  \hspace{1cm} (2)

where D is the size of the particle, λ is the X-ray wavelength (1.54178 Å), β is full width at half maximum (FWHM) of the diffraction peak, θ is the angle of diffraction and K is Scherrer’s constant (K = 0.943). The size of the In₂O₃ particles estimated from XRD by the Scherrer equation is 61 nm. However, the size of WS₂ microflakes cannot be calculated by the Scherrer equation because it does not meet the required condition (D < 100 nm).
As shown in Figure 2a,b, the morphology of WS2 microflakes did not exhibit much change after being incorporated with In2O3. The WS2 microflakes are 0.4–4.3 μm in diameter. The In2O3 nanoparticles show a cube shape with particle size around 30 nm distributed on WS2 microflakes as shown in Figure 2c. Compared to the size calculated by the Scherrer equation, the size of In2O3 particles observed in the TEM was smaller. This may be caused by the strained lattice in the crystals of In2O3. The In2O3 particles and WS2 microflakes were physically mixed without further heating; as a result, the size and morphology of In2O3 particles remained unchanged. Figure 2d shows the high-resolution TEM (HRTEM) image of the IO/WS2-1 heterostructure nanocomposite-based sensor. The lattice spacing of 0.273 nm corresponds to the (100) plane of WS2, and the lattice spacing of 0.292 nm corresponds to the (222) plane of In2O3 nanoparticles, which confirmed the heterostructure formed between In2O3 and WS2. The SEM elemental mapping images of IO/WS2-1 heterostructure nanocomposites are shown in Figure S1 in the Supplementary Materials. The synthesized sample contains four elements: W, S, In and O. The relative concentrations of each element are summarized in Table S1.
Figure 2. SEM images of (a) WS₂ and (b) IO/WS₂-1; (c) TEM image of IO/WS₂-1; (d) HRTEM image of IO/WS₂-1.

Figure 3a shows the survey of the XPS spectra of WS₂ microflakes, In₂O₃ nanoparticles and IO/WS₂-1 heterostructure nanocomposites. As shown in Figure 3b, the peaks centered at 531.3 eV and 532.4 eV correspond to the adsorbed oxygen and SO₄²⁻ of WS₂, respectively [30]. The peaks at 531.3 eV, 532.4 eV, 530.1 eV and 533.32 eV shown in Figure 3c correspond to the adsorbed oxygen, SO₄²⁻, lattice oxygen and hydroxyl in the IO/WS₂-1 heterostructure nanocomposites, respectively [28,31]. The concentrations of the adsorbed oxygen contents estimated from the integrated areas under each peak for the adsorbed O₂ on both WS₂ and IO/WS₂-1 samples were found to increase from 25.57 at.% in the WS₂ sample to 54.65 at.% in the IO/WS₂-1 sample. However, the amount of SO₄²⁻ in the IO/WS₂-1 sample was reduced, which may be due to the reduction reaction with the hydroxyl groups. The refined core spectra of W 4f and S 2p of both WS₂ and IO/WS₂-1 samples are shown in Figure S2. The binding energies of both W and S shift to the lower binding energy direction by 0.05 eV after the incorporation of In₂O₃ nanoparticles. This could be attributed to the electronic equilibrium effect between WS₂ and In₂O₃ in which WS₂ obtained the electrons transferred from In₂O₃. In a comparison of Figure S3 (shown in the Supplementary Materials) and Figure 3d, the refined core spectra of In 3d of In₂O₃ and IO/WS₂-1 samples demonstrate that the binding energy of In 3d shifts to a higher energy by 1.1 eV after the incorporation with WS₂ microflakes, indicating the loss of electrons.
3.2. Electrical and Sensing Performance

Figure 4 shows the I–V curves with applied voltages between −5 V and +5 V of the WS₂ microflake-based sensor and the IO/WS₂ heterostructure nanocomposite-based sensors. It is found that the resistances of the fabricated IO/WS₂ heterostructure nanocomposite-based sensors increased with the increase in the In₂O₃ nanoparticle content. The resistance values of WS₂, IO/WS₂-0.5, IO/WS₂-1, IO/WS₂-3 and IO/WS₂-5 sensors estimated from Figure 4 are 2.7 M, 3.6 M, 6.6 M, 10.4 M and 31.3 M, respectively. The I–V curves are linear in an ohmic connection between the sensing materials and gold electrodes.
The responses of sensors based on WS$_2$ and IO/WS$_2$ heterostructure nanocomposites with different In$_2$O$_3$ concentrations to 10 ppm ammonia at room temperature are shown in Figure 5a. The IO/WS$_2$: heterostructure nanocomposite-based sensor exhibited higher responses than the pure WS$_2$: microflake-based one. With an increase in the In$_2$O$_3$: content, the response of IO/WS$_2$: heterostructure nanocomposite-based sensors firstly increases as the In$_2$O$_3$: percentage increases from 0.5% to 1% and then decreases above 3% In$_2$O$_3$. It achieves the maximum response of 3.81 with 1% In$_2$O$_3$: content. However, as shown in Figure 5b, the response and recovery time of IO/WS$_2$: heterostructure nanocomposite-based sensors continue to decrease as the concentration of In$_2$O$_3$: increases from 0.5% to 5% with In$_2$O$_3$: introduction.

**Figure 5.** (a) Responses of WS$_2$: and IO/WS$_2$: heterostructure nanocomposite-based sensors to 10 ppm ammonia at room temperature; (b) corresponding response time and recovery time.

The gas-sensing mechanism of the IO/WS$_2$: heterostructure nanocomposite-based sensor is illustrated in Figure 6. As shown in Figure 6a, as the WS$_2$: is in contact with In$_2$O$_3$: the electrons will move from the conducting band of In$_2$O$_3$: to the conducting band of WS$_2$: while the holes will move from the valence band of WS$_2$: to the valence band of In$_2$O$_3$: due to the work function of In$_2$O$_3$: (4.28 ev) being lower than that of WS$_2$: (4.9 ev). The transfer of the electrons and holes creates a hole depletion layer (HDL) on the WS$_2$: side and a hole accumulation layer (HAL) on the In$_2$O$_3$: side, which leads to an increase in the resistance on the WS$_2$: and a decrease in the resistance on the In$_2$O$_3$: side. As a p-type semiconductor, the total resistance of the fabricated IO/WS$_2$: heterostructure nanocomposite-based sensor increased for the WS$_2$: determination. When the Fermi levels reach equilibrium, the IO/WS$_2$: heterostructure is formed, as shown in Figure 6b. Figure 6c explains the reason for the improved gas-sensing performance of the IO/WS$_2$: heterostructure nanocomposite-based sensor. As the IO/WS$_2$: heterostructure formed, WS$_2$: obtained the electrons transferring from In$_2$O$_3$: leading to more oxygen molecules adsorbed on WS$_2$: (reaction 4) with more oxygen ions produced. The result is consistent with the XPS result. Reaction 5 is thus promoted. When exposed to ammonia, the generated oxygen ions would react with the ammonia to produce more nitric oxide, water and electrons [32].
The released electrons would return to WS₂, leading to the improved sensing response of the IO/WS₂ heterostructure nanocomposite-based sensor. However, beyond the optimum concentrations of 1%, the concentration of IO/WS₂ heterostructures decreased because the formed IO/WS₂ heterostructures were fully covered by the introduced In₂O₃ particles, leading to a lower sensor response [33]. The involved chemical reaction equations are as follows [30]:

\[
O_2 \text{ (air)} \rightarrow O_2 \text{ (ads)} \quad \text{(3)}
\]

\[
O_2 \text{ (ads)} + e^- \rightarrow O_2^- \text{ (ads) (below 100 °C)} \quad \text{(4)}
\]

\[
4\text{NH}_3 + 5O_2^- \rightarrow 4\text{NO} + 6\text{H}_2\text{O} + 5e^- \quad \text{(5)}
\]

The sensing properties of the WS₂ microflake-based sensor and the IO/WS₂ heterostructure nanocomposite-based sensors in response to different concentrations of ammonia from 1 ppm to 10 ppm at room temperature are displayed in Figure 7a. It can be seen that all the IO/WS₂ heterostructure nanocomposite-based sensors have much higher responses than the pure WS₂ microflake-based sensor. The IO/WS₂-1 sample with 1% In₂O₃ content exhibited the maximum response to different concentrations of ammonia. The calibration plots of the sensors are shown in Figure 7b. The response of the sensors to NH₃ from 1 ppm to 10 ppm indicates a typical tendency as reported in most studies. Figure 7c demonstrates that the fabricated IO/WS₂-1 sensor exhibits good repeatability at room temperature. The good long-term stability of the IO/WS₂-1 sample is evidenced in Figure 7d. This sample has small baseline fluctuation under the same conditions.
Figure 7. (a, b) Response curves and values of WS₂ microflake-based sensor and IO/WS₂ heterostructure nanocomposite-based sensors upon exposure to different concentrations of ammonia at room temperature; (c) repeatability of IO/WS₂-1 heterostructure nanocomposite-based sensor at room temperature; (d) long-term stability and baseline of IO/WS₂-1 heterostructure nanocomposite-based sensor.

Figure 8a exhibits the selectivity of the WS₂ microflake-based sensor and the IO/WS₂-1 heterostructure nanocomposite-based sensor for ammonia, formaldehyde, toluene, methanol, ethanol and acetone at room temperature. It indicates that the synthesized IO/WS₂-1 heterostructure nanocomposite-based sensor has an obvious response to ammonia. However, this sensor has no obvious response to other test gases, showing good selectivity at room temperature. Based on a previous report for WS₂, the WS₂ matrix phase has good response to ammonia [24]. However, it does not have responses to other gases such as formaldehyde, toluene, methanol, ethanol and acetone at room temperature. With the introduction of In₂O₃, the formed IO/WS₂ heterostructure improved the performance of WS₂ in response to ammonia while not changing the non-responsive function of WS₂ for other gases.

Figure 8b shows the influence of the relative humidity on the response/baseline of the IO/WS₂-1 sensor for 10 ppm NH₃ at room temperature. The IO/WS₂-1 sensor attained its maximum response when the humidity was about 50%. When the humidity was lower or higher than 50%, the response of the sensor decreased. The corresponding baseline resistance also had the minimum value at 50% RH, which became larger when the humidity was above or below 50% RH.
In comparison, the ZnO/WS₂ and SnO₂/WS₂ heterostructure nanocomposites were also examined in this work. The detailed sensing characterizations are provided in Figures S4–S8 in the Supplementary Materials. The preparation and testing methods for the two materials are basically the same as those for the In₂O₃/WS₂ heterostructure nanocomposites. The major merits of each type of WS₂-based sensor are summarized in Table 1 together with the comparisons to more results reported in the literature. The In₂O₃-incorporated WS₂ nanocomposites show more advantages in detecting NH₃ at room temperature in terms of the sensitivity and response/recovery speeds. This indicates that the special “chemical catalytic” properties of the In₂O₃ in response to ammonia may play a role in this difference in performance. It is reported that In₂O₃ nanoparticles could provide more hydroxide species, resulting in the promotion of ammonia electro-oxidation [34].

**Table 1. A comparison of the performance of NH₃ sensors based on WS₂ materials.**

| Sensitive Material | NH₃ Concentration | Temperature (°C) | Response (%) | Response/Recovery (s/s) | Ref. |
|--------------------|------------------|-----------------|--------------|------------------------|------|
| WS₂:nanoflakes     | 5 ppm            | RT              | 217 a        | 120/150                | [24] |
| rGO/WS₂            | 10 ppm           | RT              | 71           | 240/600                | [35] |
| 2:1 CuO/WS₂        | 60 ppm           | RT (30 °C)      | 59.5         | 35/213                 | [36] |
| nanohybrids        |                  |                 |              |                        |      |
| WS₂/VO₂            | 10 ppm           | 150 °C          | 400          | ~150/~100              | [37] |
| Au/WS₂             | 1 ppm            | RT              | 191.54       | 968/788                | [30] |
| IO/WS₂             | 1 ppm            | RT              | 129.84       | 160/44                 | This work |
| ZnO/WS₂            | 1 ppm            | RT              | 133.63       | 288/44                 | This work |
| SnO₂/WS₂           | 1 ppm            | RT              | 117          | 224/24                 | This work |

*a*: data obtained by conversion of data in the literature.

**4. Conclusions**

Compared to the pure WS₂ microflake-based sensor, the sensors based on In₂O₃-incorporated WS₂ heterostructure nanocomposites exhibited a significantly improved response to ammonia at room temperature. In comparison to the ZnO/WS₂ and SnO₂/WS₂ nanocomposites, In₂O₃-decorated WS₂ has better sensing features overall, including a better response with a faster response speed. The WS₂ with 1 wt.% In₂O₃ nanocomposites achieve the highest response and the fastest response/recovery speeds. The sensor exhibited excellent signal repeatability and good long-term stability. It also possesses a good
selectivity for formaldehyde, toluene, methanol, ethanol and acetone and is thus a promising candidate for detecting low-concentration NH3 at room temperature.

**Supplementary Materials:** The following supporting information can be downloaded at: https://www.mdpi.com/article/10.3390/chemosensors10100402/s1, Figure S1. Elemental distribution of IO/WS2-1 heterostructure nanocomposites based sensor; Figure S2. (a,b) W 4f and S 2p refined core spectra of WS2; (c,d) W 4f and S 2p refined core spectra of IO/WS2-1 heterostructures nanocomposites based sensor; Figure S3. In 3d refined core spectra of In2O3; Figure S4. Elemental distribution of SnO2/WS2 heterostructure nanocomposites based sensor; Figure S5. (a, b) Response curves and response values of WS: and ZnO/WS: heterostructure nanocomposites based sensors to different concentrations of ammonia at room temperature, (c) response/recovery time of WS: and ZnO/WS: heterostructure nanocomposites based sensors to 10 ppm ammonia at room temperature, (d) relationship between ZnO/WS2-1 heterostructure nanocomposites based sensor’s response/baseline resistance and humidity; Figure S6. (a). The response curve of the prepared ZnO/WS: heterostructure nanocomposites based sensors to 0.47 ppm ammonia at room temperature, (b) the selectivity of the WS: and ZnO/WS: heterostructure nanocomposites based sensors to different gases at room temperature; Figure S7. (a,b) Response curves and values of WS: microflakes based sensor and SnO2/WS: heterostructure nanocomposites based sensors to different concentrations of ammonia at room temperature, (c) response/recovery time of the SnO2/WS: heterostructure nanocomposites based sensors to 10 ppm ammonia at room temperature, (d) repeatability of SnO2/WS2-1 heterostructure nanocomposites based sensor; Figure S8. (a) The relationship between the response/baseline resistance and humidity of SnO2/WS2-1 heterostructure nanocomposites based sensor, (b) selectivity of the WS: microflakes based sensor and SnO2/WS: heterostructure nanocomposites based sensors to different gases; Table S1. The relative concentrations of each element in IO/WS2-1 heterostructure nanocomposites based sensor.

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