



Original Article

Preparation of In_2O_3 Nanosheets: Effect of Experimental Condition on Morphology and Structure

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Abstract: In this study, In_2O_3 nanostructures were synthesized by the hydrothermal method through a two-step process, including hydrothermal treatment followed by heat treatment. The hydrothermal conditions, namely temperature and time, were varied to obtain different morphological characteristics of the In_2O_3 nanostructures. The hydrothermal time and temperature were investigated at three different temperature points and three different time points to determine the optimal conditions at which the In_2O_3 nanosheets exhibit the most well-defined morphology for gas sensing applications. The hydrothermal condition of 180 °C for 12 h was selected for further investigation of the structural and electrical properties. The morphological and structural characteristics were analyzed using techniques such as field-emission scanning electron microscopy (FESEM) and X-ray diffraction (XRD). The results reveal that the nanostructures exhibit diverse morphologies, depending on the hydrothermal conditions, particularly temperature and time. In addition, the gas sensing capabilities of the In_2O_3 nanosheets toward ammonia were also investigated.

Keywords: In_2O_3 , nanosheets, hydrothermal, gas sensor.

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1. Introduction

Currently, metal oxide semiconductors (MOS) have been extensively studied and widely utilized in various fields such as gas sensing, solar cells, supercapacitors, and optoelectronics [1]. Metal oxide semiconductors are generally classified into two types: n-type semiconductors, where electrons are the majority charge carriers, and p-type semiconductors, where holes are the majority charge carriers. Typical n-type metal oxides include In_2O_3 [2], V_2O_5 [3], WO_3 [4], ZnO [5], while representative p-type metal oxides include CuO [6], NiO [7], Co_3O_4 [8],... Among them, In_2O_3 has attracted significant research interest due to its diverse morphologies and structures, excellent thermal stability, and outstanding electrical, optical, as well as physicochemical properties [9]. In_2O_3 is a typical n-type semiconductor metal oxide with a wide band gap in the range of 2.3–3.75 eV [10]. Over the past decades, extensive efforts have been devoted to the synthesis and fabrication of In_2O_3 nanostructures with various morphologies. In_2O_3 nanomaterials have been reported in diverse forms such as nanosheets [11], nanowires [12], nanorods [13], nanotubes [14], hollow polyhedrons [15],...

Among these morphologies, two-dimensional (2D) In_2O_3 nanostructures exhibit significant potential due to their large specific surface area and high porosity [16]. Two-dimensional (2D) materials possess superior properties across various scientific fields, making them promising candidates for chemical sensing applications. Their nanosheet-like structure provides a large specific surface area along with distinctive electrical properties. Gas adsorption on the surface of these nanosheets significantly affects their electrical conductivity, enabling the effective adsorption and trapping of individual gas molecules, thereby resulting in excellent gas sensing performance. Gas sensors based on nanosheet-like nanostructures offer several advantages, including ease of fabrication, high sensitivity, and fast response.

Numerous studies worldwide have focused on the fabrication of In_2O_3 nanostructures using various synthesis methods such as hydrothermal [17], sol-gel [18], electrospinning [19] and chemical vapor deposition [20],... Each method has its own advantages and limitations. For instance, chemical vapor deposition can produce various morphologies by tuning parameters such as temperature and pressure; however, it typically requires high deposition temperatures, long processing times, and high operational costs. In contrast, electrospinning is cost-effective and simple but offers limited control over the resulting morphology. Among these approaches, the hydrothermal method is one of the most widely used techniques for synthesizing nanosheets, as it allows precise control over synthesis parameters such as temperature, reaction time, and precursor concentration. This method offers several advantages, including low cost, simple experimental procedures, high purity of the obtained products, and uniform morphology.

In this study, In_2O_3 nanosheets were successfully synthesized via a hydrothermal method without the use of surfactants. The effects of hydrothermal reaction time and temperature on the morphology of the obtained In_2O_3 nanostructures were systematically investigated.

2. Experimental

The precursors used in this study included indium(III) nitrate ($\text{In}(\text{NO}_3)_3 \cdot 4.5\text{H}_2\text{O}$, $\geq 99\%$), ethylene glycol ($(\text{CH}_2\text{OH})_2$), ethanol ($\text{C}_2\text{H}_5\text{OH}$), and deionized water (H_2O). Indium(III) nitrate ($\text{In}(\text{NO}_3)_3 \cdot 4.5\text{H}_2\text{O}$, $\geq 99\%$) was purchased from Sigma-Aldrich (Germany) and used as received without further purification.

In_2O_3 nanosheets were synthesized via a simple hydrothermal method. The precursor solution was prepared by dissolving 0.172 g of indium(III) nitrate ($\text{In}(\text{NO}_3)_3 \cdot 4.5\text{H}_2\text{O}$) in a mixed solvent containing 15 mL of ethylene glycol ($(\text{CH}_2\text{OH})_2$), 10 mL of ethanol ($\text{C}_2\text{H}_5\text{OH}$), and 5 mL of deionized water (H_2O). The solution was magnetically stirred for 30 min at room temperature to obtain a homogeneous white solution. The resulting solution was then transferred into a 100 mL Teflon-lined stainless-steel

autoclave, sealed, and heated at different temperatures (110, 180, and 220 °C) for different reaction durations (6, 12, and 24 h).

After completion of the hydrothermal process, the precipitated products were collected by filtration, thoroughly washed with deionized water, and dried at 80 °C to obtain powder samples. The dried precursors were then placed in an alumina boat and calcined in a tubular furnace at 500 °C for 2 h. Through the thermal decomposition of the precursor, which mainly consisted of indium oxyhydroxide (InOOH) nanostructures, polycrystalline In₂O₃ nanostructures were formed after calcination (Fig. 1).

The as-prepared precursors and the calcined In₂O₃ nanostructures were systematically characterized using X-ray diffraction (XRD) and field emission scanning electron microscopy (FESEM). XRD measurements were carried out on a Bruker D5005 diffractometer employing Cu K α_1 radiation ($\lambda = 1.5406$ Å) operated at 40 kV and 40 mA. SEM images were obtained using a Hitachi S-4800 field-emission scanning electron microscope at an accelerating voltage of 5 kV.

The as-synthesized In₂O₃ nanomaterial was mixed with an N-vinylpyrrolidone (NVP) solution. The resulting suspension was then drop-cast onto Si/SiO₂ substrates with interdigitated Pt electrodes, forming a uniform and mechanically robust sensing film. The electrode assemblies were subsequently annealed in ambient air at 500 °C for 2 h to improve film stability and interfacial adhesion.

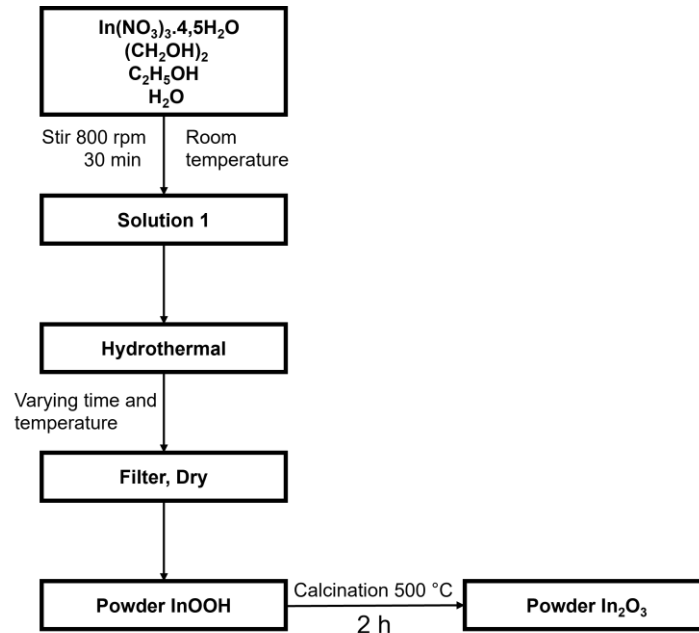


Figure 1. Preparation procedure of In₂O₃ nanostructures at different times and temperatures.

Electrical resistance measurements were performed using a Keithley Instruments 2602 source meter while the sensor was exposed cyclically to NH₃ in dry air. Gas concentrations (50–1000 ppm) were precisely controlled using MKS Instruments GV50 mass flow controllers, with a constant total flow rate of 400 sccm. All gas-sensing measurements were carried out at 50 °C in a custom-built measurement chamber. The sensor response was defined as $S = R_a/R_g$, where R_a and R_g are the steady-state resistances in air and in the presence of the target gas, respectively [21].

3. Results and Discussion

3.1. Effect of Synthesis Time and Temperature

The hydrothermal process was employed to regulate the morphology of the as-synthesized indium oxyhydroxide nanostructures, in which reaction time and temperature served as the controlling parameters. FESEM images of the indium oxyhydroxide sample synthesized at 110 °C for 6 h, acquired at different magnifications, are presented in Figure 2. The FESEM observations reveal that the synthesized InOOH exhibits uniformly distributed spindle-like structures with tapered ends and lengths in the range of 300–400 nm. Therefore, under this hydrothermal condition, the obtained morphology is not in the desired nanosheet form.

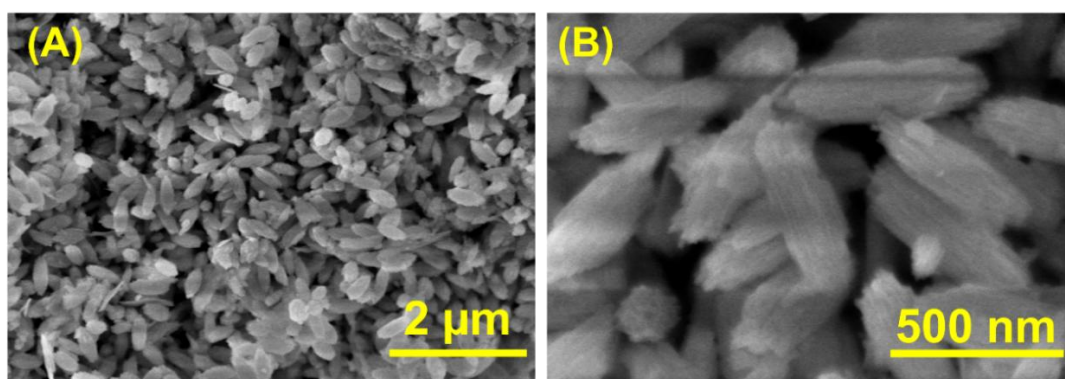


Figure 2. FESEM images of as-prepared indium oxyhydroxide nanostructure at 110 °C for 6 h at different magnifications: A) low magnification, B) high magnification.

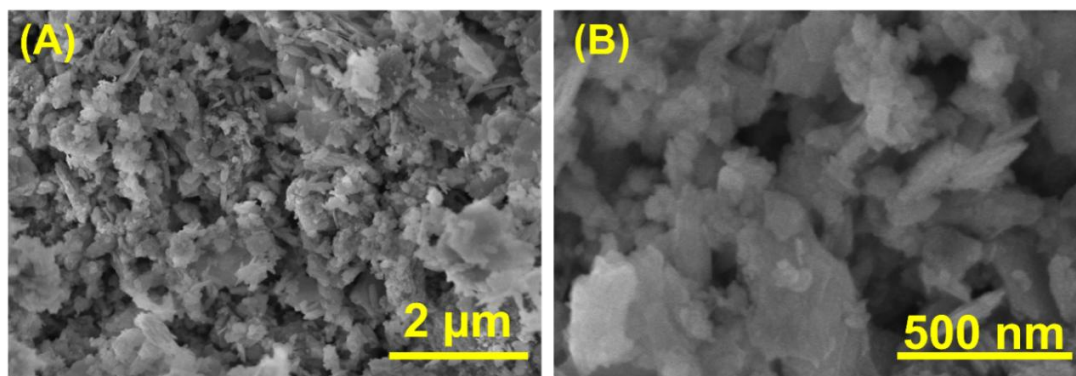


Figure 3. FESEM images of as-prepared indium oxyhydroxide nanostructure at 110 °C for 24 h at different magnifications: A) low magnification, B) high magnification.

Following the initial hydrothermal synthesis at 110 °C for 6 h, the temperature was maintained while the reaction time was extended to 24 h. The obtained product was subsequently characterized and is presented in Figure 3, which includes FESEM images of the indium oxyhydroxide nanosheets recorded at different magnifications. The morphology consists of small, fragmented nanosheets that are tightly aggregated and stacked into large, disordered clusters, with individual sheet sizes ranging from 100 to 300 nm. From these two hydrothermal conditions, it can be observed that increasing the hydrothermal time promotes the formation of the desired nanosheet morphology; however, the nanosheets remain relatively small and densely packed.

To investigate the effect of hydrothermal temperature on the morphology of indium oxyhydroxide nanosheet structures, the reaction time was fixed at 6 h while the temperature was increased to 220 °C. The FESEM images of the indium oxyhydroxide nanostructures synthesized at 220 °C for 6 h are presented in Figure 4. The obtained nanosheets exhibit lateral sizes in the range of 200–700 nm.

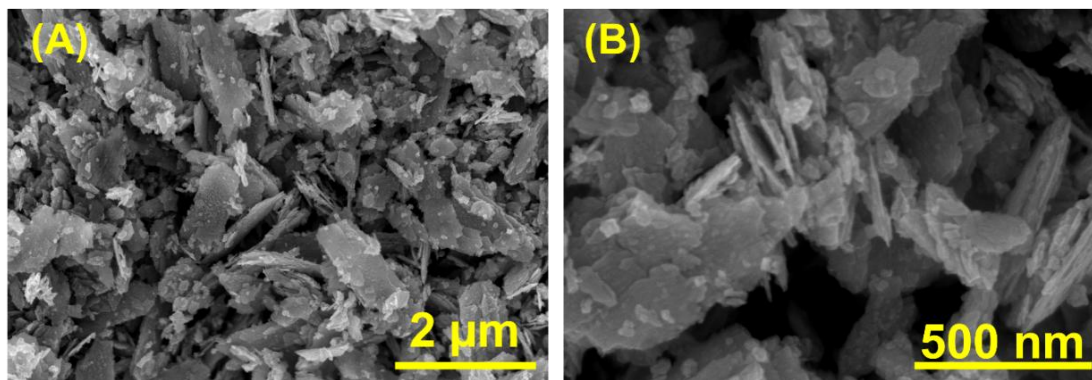


Figure 4. FESEM images of as-prepared indium oxyhydroxide nanostructure at 220 °C for 6 h at different magnifications: A) low magnification, B) high magnification.

Subsequently, the reaction temperature was maintained at 220 °C while the hydrothermal time was extended to 24 h. The FESEM images of the indium oxyhydroxide nanostructures synthesized at 220 °C for 24 h are presented in Figure 5. The nanosheet morphology is well developed, exhibiting longer and more uniform sheets, which are less densely stacked compared to the sample synthesized at 110 °C for 24 h (Figure 3). The nanosheets possess lengths in the range of 200–600 nm. These observations indicate that increasing the hydrothermal temperature leads to an increase in the nanosheet size and surface area. In comparison with the sample synthesized at 220 °C for 6 h, the nanosheet dimensions are relatively similar. However, under the condition of 220 °C for 6 h, the nanosheets tend to aggregate into dense clusters, whereas at 220 °C for 24 h, the nanosheets are more loosely distributed. Therefore, the variation in hydrothermal time also plays a significant role in determining the morphology of the material.

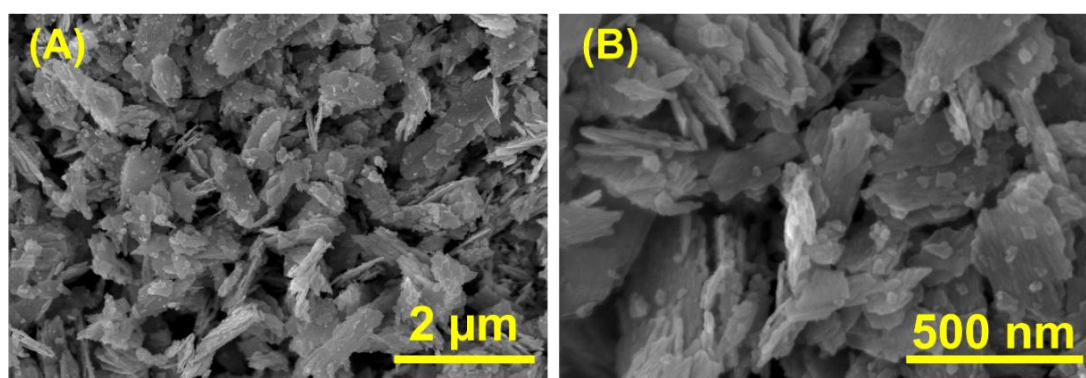


Figure 5. FESEM images of as-prepared indium oxyhydroxide nanostructure at 220 °C for 24 h at different magnifications: A) low magnification, B) high magnification.

In the search for an ideal equilibrium point, the hydrothermal time was set at 12 h while the temperature was maintained at 180 °C which have been shown to act as a "bridge" connecting the

conditions investigated above. Under this synthesis condition, the intermediate phase of the material exists entirely in the form of nanosheets and achieves extremely ideal morphological uniformity, as shown in Figure 6.

The FESEM images reveal the presence of thin, elongated nanosheets with lateral sizes ranging from 300 to 700 nm, thickness of 5–15 nm. Among the investigated samples, the material synthesized under hydrothermal conditions at 180 °C for 12 h exhibits the most uniform nanosheet morphology and the best-defined structure. Therefore, this synthesis condition was selected for subsequent characterization in this study.

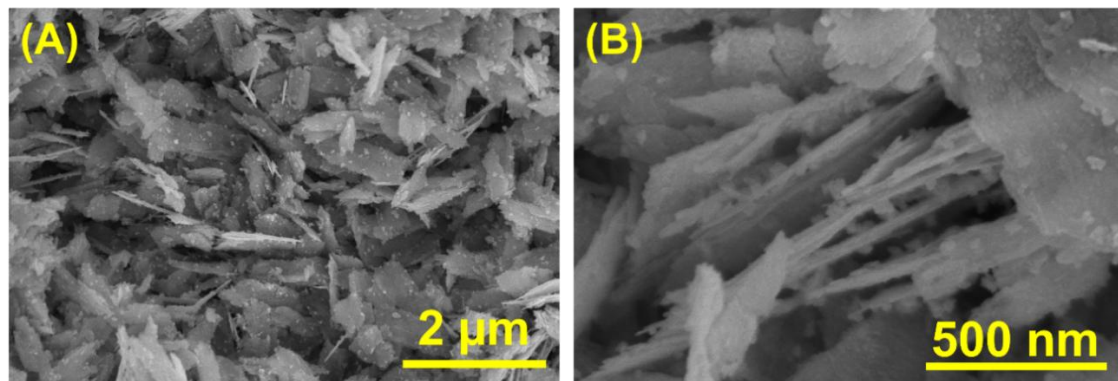


Figure 6. FESEM images of as-prepared indium oxyhydroxide nanosheets at 180 °C for 12 h at different magnifications: A) low magnification, B) high magnification.

3.2. Structural Properties

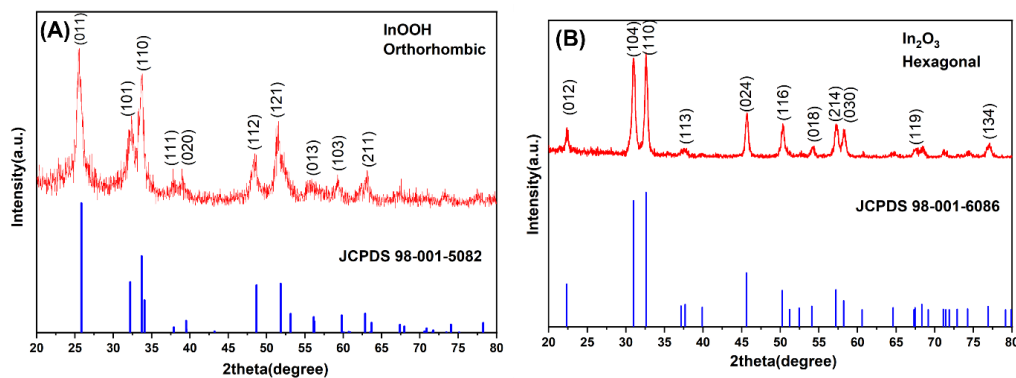


Figure 7. A) XRD of as-prepared indium oxyhydroxide nanosheets at 180 °C/12 h and B) Indium oxide nanosheets annealed at 500 °C.

Figure 7 presents the X-ray diffraction (XRD) patterns of the precursor nanosheets obtained after hydrothermal treatment at 180 °C for 12 h and the corresponding indium oxide nanosheets after subsequent calcination at 500 °C for 2 h. The XRD pattern of the precursor, as shown in Fig. 7(A), reveals the characteristic features of indium oxyhydroxide InOOH formed after the hydrothermal process. The XRD pattern of InOOH exhibits major diffraction peaks at 2θ values of 25.47°, 32.28°, 33.66°, 37.97°, 39.21°, 48.40°, 51.48°, 55.68°, 59.43°, 63.06° and 67.49°, which can be indexed to the (110), (101), (011), (111), (210), (211), (121), (002), (301), (112) and (202) crystallographic planes,

respectively. These peaks are in good agreement with the standard JCPDS card No. 98-001-5082 corresponding to orthorhombic InOOH. The most intense diffraction peak corresponds to the (011) plane. No additional impurity-related peaks are observed, confirming the successful formation of phase-pure InOOH precursor.

The XRD pattern of the sample after thermal treatment at 500 °C is shown in Fig. 7(B). The diffraction peaks are observed at 2θ values of 22.40°, 31.04°, 32.51°, 37.63°, 45.80°, 50.46°, 54.19°, 57.39°, 58.41° and 68.51°, which are indexed to the (012), (104), (110), (113), (024), (116), (018), (214), (300) and (220) planes, respectively. These results are consistent with the standard JCPDS card No. 98-001-6086 of hexagonal In₂O₃. No impurity peaks are detected, indicating the complete transformation of the precursor into phase-pure In₂O₃ upon calcination, thus confirming the high purity of the obtained material. Notably, the (104) and (110) reflections exhibit the highest intensities, suggesting preferential crystal growth along these crystallographic directions.

3.3. Gas Sensing Property

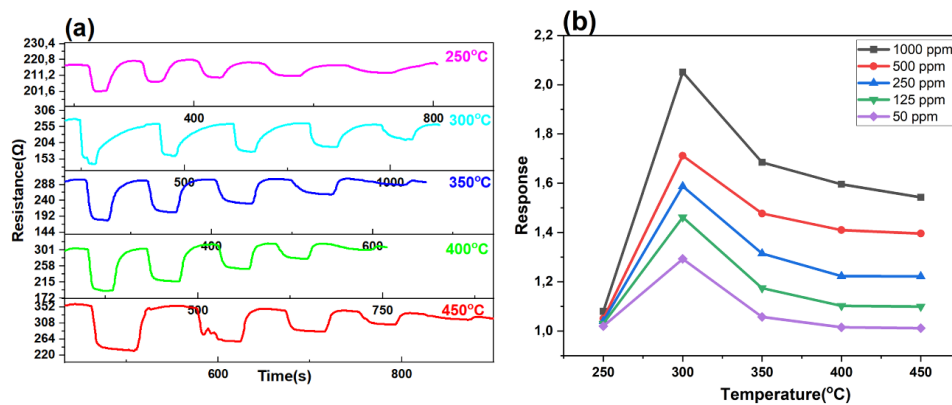
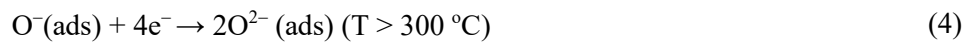
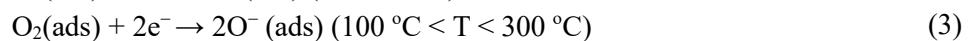


Figure 9. Response to NH₃ of the In₂O₃ sensor prepared at 180 °C/12 h: (a) Transient resistance verse response time to 50–1000 ppm at various working temperatures 250 °C – 450 °C, (b) Response to diverse concentrations 50–1000 ppm at different temperatures 250 °C – 450 °C.

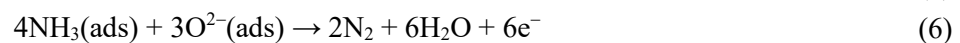
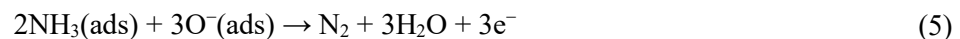
The In₂O₃ nanosheets synthesized under hydrothermal conditions at 180 °C for 12 h and subsequently annealed at 500 °C for 2 h were selected for the investigation of ammonia sensing performance. Fig. 8a and 8b present the dynamic resistance response curves and the corresponding sensor response of the In₂O₃-based sensor at different operating temperatures (250, 300, 350, 400, and 450 °C) toward NH₃ concentrations ranging from 50 to 1000 ppm. Upon exposure to NH₃ gas, the sensor resistance decreases rapidly and then recovers to its baseline value after removal of the target gas, confirming the typical n-type semiconducting behavior of In₂O₃.

At an operating temperature of 250 °C, the In₂O₃ sensor exhibits responses of 1.08, 1.05, 1.04, 1.03, and 1.02 toward NH₃ concentrations of 1000, 500, 250, 125, and 50 ppm, respectively. These results clearly indicate that the sensor response increases with increasing NH₃ concentration. The highest response ($S = 2.05$) is achieved at an operating temperature of 300 °C for 1000 ppm NH₃. At higher operating temperatures of 350, 400, and 450 °C, the corresponding responses toward 1000 ppm NH₃ are 1.68, 1.59, and 1.54, respectively. The response–temperature relationship shows an increasing trend as the temperature rises from 250 °C to 300 °C, followed by a decreasing trend as the temperature further increases from 300 °C to 450 °C. Based on these results, it can be concluded that the In₂O₃ nanosheet sensor exhibits optimal sensing performance at 300 °C toward NH₃ gas.

The gas sensing mechanism of the In_2O_3 sensor is based on the resistance variation induced by the adsorption and desorption processes of oxygen molecules on the surface of In_2O_3 . During the oxygen adsorption process, when the sensor is exposed to air (or an oxygen-containing atmosphere), oxygen molecules are adsorbed onto the surface of the In_2O_3 nanomaterial. Since In_2O_3 is an n-type semiconductor, free electrons in the conduction band are captured by the adsorbed oxygen molecules, forming ionized oxygen species such as O_2^- , O^- , and O^{2-} . This process reduces the electron concentration in the material, leading to the formation of an electron depletion layer and consequently increasing the electrical resistance. Depending on the operating temperature, the adsorbed oxygen species are formed on the material surface according to the following reactions:



When the sensor is exposed to the reducing gas NH_3 , ammonia molecules react with the previously adsorbed oxygen species on the surface and release electrons back into the conduction band, thereby lowering the potential barrier height and reducing the width of the electron depletion layer [22]. As a result, the electrical resistance of In_2O_3 decreases. The reaction can be expressed by equation (5), (6) as follows:



4. Conclusion

This study reports the hydrothermal synthesis of In_2O_3 nanosheet nanostructures and systematically investigates the effects of reaction time and temperature on their structural evolution. Among the investigated conditions, hydrothermal treatment at 180°C for 12 h yielded well-defined In_2O_3 nanosheets with optimized morphology, and this condition was selected for subsequent studies. The gas sensing performance of the synthesized In_2O_3 nanosheets toward NH_3 was then evaluated. The sensor based on In_2O_3 nanosheets exhibited its highest sensing response toward NH_3 at an optimal operating temperature of 300°C , with a response value of 2.05 toward 1000 ppm NH_3 , highlighting the strong potential of In_2O_3 nanosheets for ammonia gas sensing applications.

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