Electrospun ZnSnO$_3$/ZnO composite nanofibers and its air-sensitive properties

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Abstract

In this work, a novel heterojunction based on ZnSnO$_3$/ZnO nanofibers was prepared using electrospinning method. The crystal, structural and surface compositional properties of sample based on ZnSnO$_3$ and ZnSnO$_3$/ZnO composite nanofibers were investigated by X-ray diffractometer (XRD), Scanning electron microscope (SEM), X-ray photoelectron spectrometer (XPS) and Brunauer-Emmett-Teller (BET). Compared to pure ZnSnO$_3$ nanofibers, the ZnSnO$_3$/ZnO heterostructure nanofibers display high sensitivity and selectivity response with fast response towards ethanol gas at low operational temperature. The sensitivity response of sensor based on ZnSnO$_3$/ZnO composite nanofibers were 19.6 towards 50 ppm ethanol gas at 225°C, which was about 1.5 times superior than that of pure ZnSnO$_3$ nanofibers, which can be owed mainly to the presence of oxygen vacancies and the synergistic effect between ZnSnO$_3$ and ZnO.

Keywords: Electrospinning; ZnSnO$_3$/ZnO nanofibers; Sensing performance; n-n heterojunction

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1. Introduction

In recent years, with the increasing demand for the detection of toxic gases, volatile organic compounds and clinical gas analysis, gas sensor research has become more and more extensive. High sensitivity, fast response, excellent selectivity and long-term stability are the basic parameters of a highly sensitive gas sensor [1]. Semiconductor metal oxide gas sensors have attracted much attention because of their advantages such as fast response, low cost, simple structure and good compatibility. Semiconductor metal oxide materials (e.g. NiO [2], SnO₂ [3,4], ZnO [5], CuO [6], etc.) are widely used for their high performance, cost effectiveness and excellent gas sensing properties, but these binary oxide materials operate at high temperatures, so the search for a sensing material with low operating temperature and high sensitivity has become the focus of current research.

At present, ternary transition metal oxides as new gas-sensitive materials have received widespread attention due to their excellent gas-sensitive properties, structural stability, especially their unique energy band structure and unique physical and chemical properties. However, due to insufficient exposure area and low electron transfer, many ternary metal oxides tend to show lower gas sensitivity [7]. Among the ternary tin-based metal oxides, ZnSnO₃ is a typical perovskite structure oxide material with Zn at the A-site and Sn at the B-site. Compared to ZnO and SnO₂, ZnSnO₃ has been widely used in gas sensors for its high chemical sensitivity and excellent electrical properties [8]. The spatial arrangement of the atoms in the ZnSnO₃ system is an octahedron. The special crystal structure of ZnSnO₃ can have a lot of oxygen vacancies, provide more oxygen adsorption sites, can promote the reaction with reducing gases, thereby improving the gas sensitivity [8]. Many studies have focused on single component ZnSnO₃ as a gas sensing material. Wang et al.[9] reported that the sensitivity response to 100 ppm H₂S of hollow, cubic-structured ZnSnO₃ sample was up to 1418 at an optimum operating temperature of 335°C. ZnSnO₃ nanomicromospheres was prepared by hydrothermal method, which had high selectivity for n-butanol at 200°C [10]. Wang and his team found that ZnSnO₃ samples prepared by co-precipitation and heat treatment had a high sensitivity response to ethanol, reaching 147 [11]. Researchers have also prepared heterogeneous structures to improve gas sensing performance, such as ZnSnO₃/TiO₂, SnO₂/WO₃/SnO₂ and ZnSnO₃/ZnO [12-15]. To further improve the gas-sensitive performance of ZnSnO₃, Yu et al. compounded ZnSnO₃ with CuO and found a sensitivity response to ethanol of up to 131 [16]. Zhang et al.[17] and Cheng et al.[18] reported that ZnSnO₃ was compounded with SnO₂, respectively, due to the excellent physical and chemical properties of both SnO₂ and ZnSnO₃, the gas-sensitive properties of the SnO₂/ZnSnO₃ composite were significantly improved compared to both SnO₂ and ZnSnO₃ materials. The cubic ZnSnO₃/ZnO heterostructure showed a much improved response to 50 ppm triethylamine compared to pure ZnSnO₃ (R/R₀=21) [15]. These results indicate that heterostructures composed of MOₓ and ZnSnO₃ have great potential for gas sensing applications.

Currently, researchers can prepare ZnSnO₃ nanofibers with different morphologies (e.g. spherical, polyhedral, etc.) [19,20], but there are few reports on nanofiber samples with ZnSnO₃-based heterojunctions. The electrospinning method allows to obtain nanofibrous materials with high specific surface area and different morphologies. Based on the above considerations, ZnSnO₃ nanofibers and ZnSnO₃/ZnO composite nanofibers were synthesised by electrospinning in this paper. The results of gas-sensitive performance of ZnSnO₃ nanofibers and ZnSnO₃/ZnO nanofibers showed that the ZnSnO₃/ZnO nanofibers had a lower operating temperature (225°C), a sensitivity response of 19.6 at 50 ppm in ethanol atmosphere and better stability than ZnSnO₃. The possible enhanced gas sensing mechanism was proposed.

2 Experimental section

2.1 Material preparation

A combination of electrospinning and calcination was used to prepare ZnSnO₃/ZnO
nanofibers. The experimental procedure was as follows: First, appropriate amounts of stannous chloride (SnCl₂·2H₂O) and zinc chloride (ZnCl₂) were dissolved in a mixed solution of 8 g N, N-dimethylformamide (DMF) and 12 g anhydrous ethanol under constant stirring so that the molar ratio of Sn⁺ to Zn²⁺ was 1:2. Next, 2 g of PVP (average molecular weight of 130000 g mol⁻¹) was added and stirred thoroughly to finally obtain the precursor solution required for the electrospinning method. The electrospinning precursor solution was then loaded into a plastic syringe with a 22-gauge needle and the advance rate of the syringe pump was set at 0.4 mL h⁻¹. A voltage of 15 kV is applied between the needle and the collector, and a layer of aluminium foil is wrapped around the rotating receiver 20 cm from the needle to collect the fibres. The collected precursor fibres were calcined in air at 450 °C for 2 h to obtain ZnSnO₃/ZnO nanofibers.

2.2 Characterization

The physical phases of the calcined samples were analysed by X-ray powder diffractometer (XRD, Bruker D8 Advance) using a copper target Kα line with a scanning range of 20 - 60° and a scanning speed of 6°/min. The field emission scanning electron microscopy (Merlin Compact, Carl Zeiss, Germany) was used to characterize morphology of the samples. Thermogravimetric tests were carried out by a thermal analysis system (Diamond TG/DTA, Perkin Elmer S. A.) to determine the calcination temperature of the precursors, using samples weighing 4 - 6 mg and heated to 600 °C at a heating rate of 15 °C/min. The elemental composition and valence states in the material were characterized using an X-ray photoelectron spectrometer (Thermo Scientific K-Alpha, XPS). Specific surface area of the prepared nanofibers by N₂ adsorption/desorption test (BET, Micromeritics ASAP 2020).

2.3 Gas-sensitive performance measurement

A 200 mg sample of ZnSnO₃/ZnO nanofibers was mixed well with 0.2 mL of deionised water in an agate mortar and ground to a paste. The ground paste sample is then uniformly coated on the surface of the Ag-Pd forked finger electrode on an Al₂O₃ substrate and dried for a certain period of time to obtain the gas sensitive element required for the test. Before testing, the dried gas-sensitive elements are aged in air for 10 h. The sensitivity response, operating temperature and stability of the preparations were tested on the CGS-ITP Intelligent Gas Sensitive Analysis System.

3 Results and Discussion

3.1 Microstructure and morphology

In order to determine the calcination temperature of the ZnSnO₃ precursor obtained by electrospinning methods, TG tests were carried out on the ZnSnO₃ precursor in an air atmosphere in the temperature range of 30 - 600 °C with a heating rate of 15 °C/min. The results are shown in Figure 1(a). The graph shows an 8 wt% drop in sample mass at 300 °C. This is due to the evaporation of organic solvents and the evaporation of gases and moisture adsorbed on the surface of the sample. The sharp decrease in sample mass around 350 °C can be attributed to the decomposition of the precursor, while the significant drop in the curve around 400°C can be attributed to the decomposition of PVP. In addition, no significant weight changes were observed above 450 °C, which proves that all organic matter in the precursor decomposes at 450 °C and that calcination of the precursor at temperatures higher than 450 °C is sufficient. Figure 1(b) shows the XRD results of the ZnSnO₃/ZnO precursor after 2 h of calcination at 450 °C. A comparison with the standard card (PDF No. 28-1486) shows that the XRD diffraction peaks at 2θ of 26.5°, 33.7°, 37.7°, 51.6° and 54.6° correspond to the (012), (110), (015), (116), (018) and (214) crystal planes of ZnSnO₃ respectively. The remaining XRD diffraction peaks were compared with the standard card (PDF No. 65-3411) and were found to correspond to (100), (101), (102) and (110) crystal planes at 2θ of 31.7°, 36.3°, 47.5° and 56.6°, respectively. That is, the XRD diffraction peak corresponding to the standard card (PDF No. 65-3411) is the diffraction peak of ZnO. The results show that the ZnSnO₃/ZnO
precursors were successfully prepared after calcination in air at 450 °C for 2 h.

![Graph](image)

Figure 1 (a) TG test results of ZnSnO$_3$ precursors; (b) XRD of ZnSnO$_3$ and ZnSnO$_3$/ZnO calcined at 450 °C

Figure 2(a) and (b) shows the SEM results of ZnSnO$_3$ samples prepared after calcination at 450 °C for 2 h in an air atmosphere. As can be seen in Figure 2(a), the ZnSnO$_3$ sample after calcination at 450 °C for 2 h in an air atmosphere is coarse and uniformly fibrous, with no breakage of the continuous fibers. The average diameter of the ZnSnO$_3$ sample is about 300 - 400 nm, as shown in Figure 2 (b). Figure 2(c) and (d) shows the SEM results of ZnSnO$_3$/ZnO samples prepared after calcination at 450 °C for 2 h in air atmosphere. As can be seen in Figure 2(c), the ZnSnO$_3$/ZnO sample exhibits uniformly coarse and fine fibers with no fracture in the fiber continuity. The average diameter of the ZnSnO$_3$/ZnO nanofibers is even smaller, around 200 nm, as shown in Figure 2(d). Due to the low calcination temperature of 450 °C, the rate of gas production from the decomposition of pharmaceuticals is slow and the pressure difference between the interior and exterior of the fibers is not large, so no hollow structure is formed [21]. The above experiments show that the diameter of the ZnSnO$_3$/ZnO nanofibers obtained by compounding ZnSnO$_3$ with ZnO is significantly reduced compared to the ZnSnO$_3$ nanofibre samples. The BET analysis shows that the specific surface area of the ZnSnO$_3$/ZnO nanofibers (31.24 m$^2$ g$^{-1}$) was substantially larger than that of pristine ZnSnO$_3$ nanofibers (20.15 m$^2$ g$^{-1}$).

![SEM images](image)

Figure 2 SEM images of ZnSnO$_3$ and ZnSnO$_3$/ZnO: (a) and (b) ZnSnO$_3$; (c) and (d) ZnSnO$_3$/ZnO

XPS are carried out to study the elemental composition and valence states in
ZnSnO$_3$/ZnO, and the results are shown in Figure 3. Figure 3 (a) shows the full XPS spectrum of the ZnSnO$_3$/ZnO nanofibers, from which it can be observed that the obtained ZnSnO$_3$/ZnO nanofibers contain the elements Zn, Sn, C and O. The peak at 284.8 eV corresponds to the spin-orbit peak of C 1s. The broad XPS spectrums of the Zn-2p range, Sn-3d and O-1s range are shown in Fig.3(b) and (d). In case of Zn-2p spectrum, the Zn-2p peak can be distributed into two signals as shown in Fig. 3(b). Zn-2p$_{3/2}$ and Zn-2p$_{1/2}$ signals are focused at 1021.1 eV and 1044.1 eV, respectively, which indicates that the chemical valence of Zn in the system is $+2$ [22]. As shown in Fig. 3(c), the two peaks at 486.8 and 495.1 eV correspond to the Sn 3d$_{5/2}$ and Sn 3d$_{3/2}$ spin-orbit peaks, respectively. The bimodal spin orbit shows a split value of approximately 8.3 eV, indicating the presence of Sn$^{2+}$ cations [23]. The O$_{1s}$ spectrum of ZnSnO$_3$ and ZnSnO$_3$/ZnO is illustrated in Fig. 3(d) and (e), which is deconvoluted into three characteristic peaks by Gaussian fitting[16,24]. The three fitting peaks are attributed to three important oxygen species [25], denoted as O$_L$, O$_V$, O$_C$, respectively, corresponding to O$^-$ species in the crystal lattice, VOs and chemically adsorbed or dissociated oxygen species, respectively [26]. The three characteristic peaks of O$_{1s}$ in ZnSnO$_3$ nanofibers are located at 530.06 eV, 531.65 eV and 533.13 eV, while the characteristic peaks of O$_{1s}$ in ZnSnO$_3$/ZnO nanofibers are located at 529.99 eV, 531.33 eV, and 532.27 eV, which can be corresponded to the O$_L$, O$_V$, O$_C$ species, respectively[17,27]. It must be pointed out that the gas sensitivity characteristics may be highly dependent on the type of VO present on the semiconductor surface [28]. Fig. 4(f) shows that the relative percentage of each oxygen species in ZnSnO$_3$ and ZnSnO$_3$/ZnO nanofibers. The proportion of oxygen species in obtained ZnSnO$_3$ and ZnSnO$_3$/ZnO nanofibers are shown in the table 1. The results of Fig.4(f) and table 1 are clear that compared with the percentage of O$_V$ of ZnSnO$_3$ nanofibers, that of ZnSnO$_3$/ZnO nanofibers substantially increases.

![XPS spectra of ZnSnO$_3$/ZnO nanofibers](image)

**Figure 3** XPS spectra of ZnSnO$_3$/ZnO nanofibers: (a) a survey spectrum, high resolution spectra for (b) Zn 2p, (c) Sn 3d, (d) O$_{1s}$ (of ZnSnO$_3$), (e) O$_{1s}$ (of ZnSnO$_3$/ZnO), (f) fitting results of O 1s XPS spectra of ZnSnO$_3$ and ZnO/ZnSnO$_3$.

**Table 1** The proportion of oxygen species in obtained ZnSnO$_3$ and ZnO/ZnSnO$_3$ nanofibers.

| Sample            | O$^-$ (%) | O$^+$ (%) | O$_V$ (%) |
|-------------------|-----------|-----------|-----------|
| ZnSnO$_3$         | 26.2%     | 32.4%     | 41.4%     |
| ZnO/ZnSnO$_3$     | 10%       | 44.3%     | 45.7%     |
3.2 Gas-sensitive properties of ZnSnO$_3$/ZnO nanofibers

It is well known that changes in operating temperature can highly influence the gas sensing characteristics of sensing materials. To verify this, the response of ZnSnO$_3$ samples and ZnSnO$_3$/ZnO composite nanofibers toward 50 ppm ethanol gas are tested at different temperatures and the results are shown in Figure 4. Figure 4(a) show that the response of the ZnSnO$_3$ sample increases with increasing temperature until the temperature rises to 300 °C, when the response is maximum at 13.4. Then the temperature continues to increase and the response decreases instead. Figure 4(b) shows the sensitivity response of ZnSnO$_3$/ZnO samples towards 50 ppm ethanol gas at different temperatures. From the graph it can be concluded that the optimum working temperature for the ZnSnO$_3$/ZnO sample is 225°C and the sensitivity response for the ZnSnO$_3$/ZnO sample is 19.6. The optimum operating temperature of the ZnSnO$_3$/ZnO sample is significantly lower than that of the ZnSnO$_3$ sample. The sensitivity response has also been improved, with a 46.3 % increase in sensitivity response.

![Fig 4](image)

Fig.4 Sensitivity of ZnSnO$_3$ and ZnSnO$_3$/ZnO towards 50 ppm ethanol at different temperatures: (a) ZnSnO$_3$; (b) ZnSnO$_3$/ZnO

To further investigate the gas-sensitive performance of the ZnSnO$_3$/ZnO samples, the response of ZnSnO$_3$ and ZnSnO$_3$/ZnO samples to different concentrations of ethanol and the stability of the response to 50 ppm ethanol were tested at the optimum operating temperature, respectively, and the results are shown in Figure 5. As seen in Fig. 5(a), the response of ZnSnO$_3$/ZnO increases with increasing ethanol concentration due to the fact that with increasing ethanol concentration, more ethanol molecules react with the oxygen ions adsorbed on the surface of the material, resulting in a higher carrier concentration and thus a subsequent increase in the response of the material. Figure 5(b) show that the response of the ZnSnO$_3$/ZnO composite samples increased with increasing ethanol concentration, while the response of the ZnSnO$_3$/ZnO samples was higher than that of the ZnSnO$_3$ samples at the same concentration of ethanol. Figure 5 (c) and (d) show the stability of the ZnSnO$_3$ sample and the ZnSnO$_3$/ZnO sample under ethanol atmosphere at 50 ppm, respectively. As can be seen from the graphs, the ZnSnO$_3$ and ZnSnO$_3$/ZnO samples showed no significant fluctuations in response after six repeated exposures to ethanol at 50 ppm, indicating that both the ZnSnO$_3$ and ZnSnO$_3$/ZnO samples have good stability. The above experimental results show that the ZnSnO$_3$/ZnO sample has a lower working temperature, higher response and better stability for ethanol atmosphere than the ZnSnO$_3$ sample.
Fig. 5 Sensitivity of ZnSnO$_3$ and ZnSnO$_3$/ZnO to different concentrations of ethanol at the optimal working temperature: (a) ZnSnO$_3$; (b) ZnSnO$_3$/ZnO; the best Stability of sensitivity to 50 ppm ethanol at working temperature: (c) ZnSnO$_3$; (d) ZnSnO$_3$/ZnO

3.3 Gas-sensing mechanism

The sensing mechanism based on metal oxide semiconductor gas sensor is the changed resistance caused by the adsorption and desorption of the target gas molecules on the surface of sensitive materials, which depends mainly on the concentration and mobility of the charge. Therefore, the gas-sensitive mechanism of ZnSnO$_3$ and ZnSnO$_3$/ZnO composite nanofibers can be understood simply as a surface-controlled mechanism. The change in resistance depends on the type and amount of oxygen chemisorbed on the surface. In ambient air, the resistance of ZnSnO$_3$ and ZnSnO$_3$/ZnO composite nanofibers is mainly controlled by the concentration of adsorbed oxygen ($O^\cdot$ and $O^{2-}$). The variation in response for ZnSnO$_3$ nanofibers is mainly related to the adsorption-desorption reaction of ethanol gas on the surface of ZnSnO$_3$ samples. ZnSnO$_3$ is a typical N-type semiconductor material, when it is in the air oxygen molecules adsorbed on the surface of ZnSnO$_3$ and gain electrons and thus convert into oxygen ions. When a certain amount of oxygen ions accumulated on the material surface reaches equilibrium, an electron depletion layer is formed on the material surface, which reduces the carrier concentration of the material system and in turn leads to an increase in the material resistance. However, when ZnSnO$_3$ is placed in an ethanol atmosphere, the ethanol molecules will react with oxygen ions, releasing a large number of electrons and causing the resistance of the material to drop, the equation for the above process reaction is as follows[29]:

$$O_2(gas) \leftrightarrow O_2(ads) \quad O_2 + e^- \rightarrow O^-, O^-, O^{2-} \quad (1)$$

$$C_2H_5OH + 6O^{2-}(ads) \leftrightarrow 2CO_2 + 3H_2O + 12e^- \quad (2)$$

According to ref [15], since both ZnSnO$_3$ and ZnO are N-type semiconductor materials, when ZnO is successfully compounded with ZnSnO$_3$ to form a homotypic heterojunction (i.e. N-N junction), this N-N junction is a key factor in improving the ethanol gas sensitivity.
between ZnSnO₃ and ZnO. Differences in the Fermi energy levels of two connected semiconductor compounds. The transfer of electrons from higher energy states to lower energy states until equilibrium is reached at the Fermi energy level, resulting in the formation of a depletion layer in the interface region. In contrast to ZnSnO₃, the electrons on the ZnSnO₃ conduction band in the ZnSnO₃/ZnO sample are transferred to the ZnO conduction band to be trapped by oxygen molecules, resulting in the formation of a large number of oxygen anions. At the same time, the electron depletion layer appears near the surface of ZnSnO₃/ZnO, which is an important component of the gas sensing process. In the air environment, the increased height of the potential barrier of the ZnSnO₃/ZnO heterostructured composite makes electron transfer more difficult, which also leads to a significant increase in sensor resistance (Rₛ). In an ethanol environment, the reaction of ethanol molecules with oxygen ions releases electrons back into the ZnSnO₃/ZnO heterojunction, resulting in an increase in electron concentration. This significantly reduces the potential barrier height of ZnSnO₃ and ZnO, significantly reducing the sensor resistance (Rₛ). The analysis of the above gas-sensitive properties revealed that the heterogeneous structure of the ZnSnO₃/ZnO composites enhanced electron transport and enabled effective separation of electrons and holes compared to the ZnSnO₃ nanofibers material.

Compared to ZnSnO₃ nanofibers, ZnSnO₃/ZnO composite nanofibers have a larger VOs of 44.3%, almost 1.5 times that of ZnSnO₃ nanofibers, which is conducive to improve the sensitivity characteristics of gas-sensitive materials[30]. It is also suggested that in the "chemical sensitization" mechanism established by the spillover effect, ZnO plays an important role in activating the dissociation rate of molecular oxygen[31].

4 Conclusion

In conclusion, ZnSnO₃ nanofibers and ZnSnO₃/ZnO nanofibers were successfully prepared using a simple electrospinning method. The average diameter of the ZnSnO₃/ZnO composite nanofibers is approximately 200 nm. Compared to ZnSnO₃ nanofibers, ZnSnO₃/ZnO nanofibers have a lower operating temperature (225 °C), higher sensitivity (19.6) and better stability in an ethanol atmosphere, which can be owed mainly to the presence of oxygen vacancies and the synergistic effect between ZnSnO₃ and ZnO. The results confirmed that ZnSnO₃/ZnO nanofibers could have an outstanding potential candidate for ethanol detection.

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